



Check for  
updates

**NIST Internal Report  
NIST IR 8519**

**Cannabis Laboratory  
Quality Assurance Program:  
Exercise 2 Cannabinoid Final Report**

Andrea J. Yarberry  
Melissa M. Phillips  
Walter B. Wilson

This publication is available free of charge from:  
<https://doi.org/10.6028/NIST.IR.8519>

**NIST Internal Report  
NIST IR 8519**

**Cannabis Laboratory  
Quality Assurance Program:  
Exercise 2 Cannabinoid Final Report**

Andrea J. Yarberry

Melissa M. Phillips

Walter B. Wilson

*Chemical Sciences Division  
Material Measurement Laboratory*

This publication is available free of charge from:

<https://doi.org/10.6028/NIST.IR.8519>

June 2024



U.S. Department of Commerce  
*Gina M. Raimondo, Secretary*

National Institute of Standards and Technology

*Laurie E. Locascio, NIST Director and Under Secretary of Commerce for Standards and Technology*

Certain equipment, instruments, software, or materials, commercial or non-commercial, are identified in this paper in order to specify the experimental procedure adequately. Such identification does not imply recommendation or endorsement of any product or service by NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

**NIST Technical Series Policies**

[Copyright, Use, and Licensing Statements](#)

[NIST Technical Series Publication Identifier Syntax](#)

**Publication History**

Approved by the NIST Editorial Review Board on 2024-04-02

**How to Cite this NIST Technical Series Publication**

Yarberry AJ, Phillips MM, Wilson WB (2024) Cannabis Laboratory Quality Assurance Program: Exercise 2 Cannabinoid Final Report. (National Institute of Standards and Technology, Gaithersburg, MD), NIST Internal Report (IR) NIST IR 8519. <https://doi.org/10.6028/NIST.IR.8519>

**Author ORCID iDs**

Andrea J. Yarberry: 0000-0001-6899-3000

Melissa M. Phillips: 0000-0003-0477-7637

Walter B. Wilson: 0000-0003-1763-781X

**Contact Information**

[cannabis@nist.gov](mailto:cannabis@nist.gov)

## **Abstract**

NIST launched a Cannabis Laboratory Quality Assurance Program (CannaQAP) in 2020 to assist laboratories in demonstrating and improving cannabis (hemp and marijuana) measurement comparability and competence. CannaQAP provides tools that allow analysts and laboratories to assess how their methods perform relative to the community and to an accepted value. Exercise 2 of CannaQAP focused on the determination of cannabinoids ( $\Delta^9$ -THC, THCA, total THC, CBD, CBDA, total CBD, and up to 13 additional cannabinoids), moisture, and toxic elements (As, Cd, Co, Cr, Mo, Ni, Pb, Se, U, Mn, Be, V, and Hg) in cannabis plant material.

## **Keywords**

Cannabinoids; cannabis; CannaQAP; hemp; marijuana; moisture.

## Table of Contents

<b>Executive Summary.....</b>	<b>1</b>
<b>1. Introduction.....</b>	<b>3</b>
<b>1.1. Overview.....</b>	<b>3</b>
<b>1.1.1. Statistics.....</b>	<b>4</b>
<b>1.1.2. Individualized Data Table.....</b>	<b>4</b>
<b>1.1.3. Summary Data Table .....</b>	<b>6</b>
<b>1.1.4. Figures .....</b>	<b>6</b>
<b>1.1.4.1. Data Summary View (Method Comparison Data Summary View).....</b>	<b>6</b>
<b>1.1.4.2. Data Summary View (Method Comparison Data Summary View).....</b>	<b>8</b>
<b>2. Study Material Preparation and Characterization.....</b>	<b>10</b>
<b>2.1. Study Material Acquisition and Preparation .....</b>	<b>10</b>
<b>2.1.1. NRC HEMP-1 (Plant Sample 1).....</b>	<b>10</b>
<b>2.1.2. Plant Samples 2 through 6 .....</b>	<b>10</b>
<b>2.2. NIST Methods for Material Preparation and Characterization .....</b>	<b>11</b>
<b>2.2.1. Sample Preparation and Cannabinoid Extraction .....</b>	<b>11</b>
<b>2.2.2. Analytical Methods.....</b>	<b>11</b>
<b>2.2.2.1. LC-PDA Analysis.....</b>	<b>11</b>
<b>2.2.2.2. LC-MS/MS Analysis.....</b>	<b>13</b>
<b>2.2.3. Quality Metrics/ID and QM.....</b>	<b>16</b>
<b>2.3. Characterization of Cannabinoids in Study Samples .....</b>	<b>18</b>
<b>2.3.1. LC-PDA Characterization .....</b>	<b>18</b>
<b>2.3.2. LC-MS/MS Characterization .....</b>	<b>20</b>
<b>2.4. Assignment of Target Values for CannaQAP Exercise 2 Study Samples.....</b>	<b>21</b>
<b>3. Δ<sup>9</sup>-THC, THCA, And Total Δ<sup>9</sup>-THC .....</b>	<b>23</b>
<b>3.1. Study Overview .....</b>	<b>23</b>
<b>3.2. Sample Information .....</b>	<b>23</b>
<b>3.3. Reporting Statistics.....</b>	<b>24</b>
<b>3.4. Study Results and Discussion.....</b>	<b>27</b>
<b>3.4.1. Δ<sup>9</sup>-THC.....</b>	<b>29</b>
<b>3.4.1.1. Within- and Between-Laboratory Precision .....</b>	<b>29</b>
<b>3.4.1.2. Accuracy .....</b>	<b>34</b>
<b>3.4.1.3. Candidate Analytical Methods .....</b>	<b>44</b>
<b>3.4.1.4. Examples and Recommendations.....</b>	<b>45</b>
<b>3.4.2. THCA.....</b>	<b>49</b>

<b>3.4.2.1. Within- and Between Laboratory Precision .....</b>	<b>49</b>
<b>3.4.2.2. Accuracy .....</b>	<b>53</b>
<b>3.4.2.3. Candidate Analytical Methods .....</b>	<b>60</b>
<b>3.4.3. Total Δ<sup>9</sup>-THC .....</b>	<b>61</b>
<b>3.4.3.1. Within- and Between Laboratory Precision .....</b>	<b>61</b>
<b>3.4.3.2. Accuracy .....</b>	<b>63</b>
<b>3.4.3.3. Candidate Analytical Methods .....</b>	<b>74</b>
<b>3.4.3.4. Examples and Recommendations.....</b>	<b>75</b>
<b>3.5. Conclusions .....</b>	<b>76</b>
<b>4. CBD, CBDA, and Total CBD .....</b>	<b>77</b>
<b>4.1. Study Overview .....</b>	<b>77</b>
<b>4.2. Sample Information .....</b>	<b>77</b>
<b>4.3. Reporting Statistics .....</b>	<b>78</b>
<b>4.4. Study Results and Discussion.....</b>	<b>79</b>
<b>4.4.1. CBD .....</b>	<b>81</b>
<b>4.4.1.1. Within- and Between-Laboratory Precision .....</b>	<b>81</b>
<b>4.4.1.2. Accuracy .....</b>	<b>85</b>
<b>4.4.1.3. Candidate Analytical Methods .....</b>	<b>92</b>
<b>4.4.2. CBDA.....</b>	<b>93</b>
<b>4.4.2.1. Within- and Between-Laboratory Precision .....</b>	<b>93</b>
<b>4.4.2.2. Accuracy .....</b>	<b>97</b>
<b>4.4.2.3. Candidate Analytical Methods .....</b>	<b>104</b>
<b>4.4.3. Total CBD .....</b>	<b>105</b>
<b>4.4.3.1. Within- and Between-Laboratory Precision .....</b>	<b>105</b>
<b>4.4.3.2. Accuracy .....</b>	<b>106</b>
<b>4.5. Conclusions .....</b>	<b>114</b>
<b>5. Minor Cannabinoids .....</b>	<b>115</b>
<b>5.1. Study Overview .....</b>	<b>115</b>
<b>5.2. Sample Information .....</b>	<b>116</b>
<b>5.3. Reporting Statistics.....</b>	<b>119</b>
<b>5.4. Study Results and Discussion.....</b>	<b>121</b>
<b>5.4.1. Within- and Between Laboratory Precision.....</b>	<b>122</b>
<b>5.4.2. Accuracy.....</b>	<b>125</b>
<b>5.4.2.1. CBC and CBCA .....</b>	<b>125</b>
<b>5.4.2.2. CBDV and CBDVA .....</b>	<b>136</b>

<b>5.4.2.3. CBG and CBGA.....</b>	<b>139</b>
<b>5.4.2.4. CBL and CBLA .....</b>	<b>144</b>
<b>5.4.2.5. CBN and CBNA .....</b>	<b>147</b>
<b>5.4.2.6. THCV and THCVA.....</b>	<b>152</b>
<b>5.4.2.7. Δ<sup>8</sup>-THC .....</b>	<b>156</b>
<b>5.4.3. Candidate Analytical Methods .....</b>	<b>156</b>
<b>5.5. Conclusions .....</b>	<b>158</b>
<b>References.....</b>	<b>160</b>
<b>Appendix A. List of Symbols, Abbreviations, and Acronyms.....</b>	<b>164</b>
<b>Appendix B. Tables of participant data .....</b>	<b>168</b>
<b>Appendix C. Method questionnaire .....</b>	<b>258</b>
<b>C.1. General Laboratory Questions.....</b>	<b>259</b>
<b>C.1.1. Participant sectors.....</b>	<b>259</b>
<b>C.1.2. Accreditation.....</b>	<b>260</b>
<b>C.1.3. Level of Experience.....</b>	<b>261</b>
<b>C.2. Sample Preparation Questions .....</b>	<b>262</b>
<b>C.2.1. Homogenization .....</b>	<b>262</b>
<b>C.2.2. Homogenization method .....</b>	<b>263</b>
<b>C.2.3. Sample Size .....</b>	<b>264</b>
<b>C.3. Extraction Procedure .....</b>	<b>265</b>
<b>C.3.1. Extraction Solvent .....</b>	<b>265</b>
<b>C.3.2. Extraction Equipment .....</b>	<b>266</b>
<b>C.3.3. Extraction Volume .....</b>	<b>267</b>
<b>C.3.4. Extraction Time .....</b>	<b>268</b>
<b>C.3.5. Filtration .....</b>	<b>269</b>
<b>C.3.6. Dilution .....</b>	<b>270</b>
<b>C.4. Analytical Methods Questions.....</b>	<b>271</b>
<b>C.4.1. Instrumental Techniques .....</b>	<b>271</b>
<b>C.4.2. Injection Volumes.....</b>	<b>272</b>
<b>C.4.3. Needle Wash Solvent.....</b>	<b>273</b>
<b>C.4.4. Analytical Columns .....</b>	<b>274</b>
<b>C.4.5. Mobile Phase Programs .....</b>	<b>275</b>
<b>C.4.6. Isocratic Separation Conditions.....</b>	<b>276</b>
<b>C.4.6.1. Organic Modifiers.....</b>	<b>276</b>
<b>C.4.6.2. Column Temperatures (°C).....</b>	<b>277</b>

<b>C.4.7. Gradient Separation Conditions .....</b>	<b>278</b>
<b>C.4.7.1. Mobile Phases.....</b>	<b>278</b>
<b>C.4.7.2. Organic Modifiers.....</b>	<b>279</b>
<b>C.4.7.3. Column Temperature (°C) .....</b>	<b>280</b>
<b>C.4.8. Detector Wavelength.....</b>	<b>281</b>
<b>C.5. Calibration Questions .....</b>	<b>282</b>
<b>C.5.1. Calibration Methods.....</b>	<b>282</b>
<b>C.5.2. Internal Standards .....</b>	<b>283</b>
<b>C.5.3. Source of Calibrants.....</b>	<b>284</b>
<b>C.6. Sample Classification Questions .....</b>	<b>285</b>
<b>C.6.1. Cutoff/Threshold Points for Distinguishing Hemp and Marijuana .....</b>	<b>285</b>
<b>C.6.2. Laboratories Classification of Hemp Samples.....</b>	<b>286</b>
<b>C.6.2.1. NRC HEMP-1 (Plant Sample 1).....</b>	<b>286</b>
<b>C.6.2.2. Plant Sample 4 .....</b>	<b>287</b>
<b>C.6.2.3. Plant Sample 6 .....</b>	<b>288</b>
<b>C.6.3. Laboratories Classification of Marijuana Samples .....</b>	<b>289</b>
<b>C.6.3.4. Plant Sample 2 .....</b>	<b>289</b>
<b>C.6.3.5. Plant Sample 3 .....</b>	<b>289</b>
<b>C.6.3.6. Plant Sample 5 .....</b>	<b>290</b>

## List of Tables

<b>Table 1-1. Example of an individualized data table.....</b>	<b>4</b>
<b>Table 1-2. Example data summary table. ....</b>	<b>6</b>
<b>Table 2-1. Preparation of Plant Samples 2 through 6 used in CannaQAP Exercise 2. ....</b>	<b>10</b>
<b>Table 2-2. Mobile phase program for the LC-PDA cannabinoid method.....</b>	<b>12</b>
<b>Table 2-3. Mobile phase program for the LC-MS/MS cannabinoid method.....</b>	<b>14</b>
<b>Table 2-4. MS/MS parameters for the LC-MS/MS cannabinoid method. ....</b>	<b>14</b>
<b>Table 2-5. Quality metrics used for LC-PDA and LC-MS/MS measurements to ensure accuracy.....</b>	<b>16</b>
<b>Table 2-6. Cannabinoid mass fractions in HM20NOV-1 control material from UK-PT program determined using LC-MS/MS.....</b>	<b>17</b>
<b>Table 2-7. Summary information for cannabinoids determined in Exercise 2 study samples using LC-PDA.....</b>	<b>19</b>
<b>Table 2-8. Summary information for Exercise 2 study samples using LC-MS/MS.....</b>	<b>20</b>
<b>Table 2-9. NIST Consensus Builder model inputs and target value outputs using LC-PDA and LC-MS/MS. ....</b>	<b>21</b>
<b>Table 2-10. Target values and expanded uncertainties for CannaQAP Exercise 2 study samples.....</b>	<b>22</b>

<b>Table 3-1. Example individualized data summary table for <math>\Delta^9</math>-THC, THCA, and total <math>\Delta^9</math>-THC.....</b>	<b>24</b>
<b>Table 3-2. Enrollment and reporting statistics for <math>\Delta^9</math>-THC, THCA, and total <math>\Delta^9</math>-THC. ....</b>	<b>25</b>
<b>Table 3-3. Number of laboratories reporting use of qualitative and quantitative analysis for <math>\Delta^9</math>-THC. .</b>	<b>25</b>
<b>Table 3-4. Number of laboratories reporting use of qualitative and quantitative analysis for THCA. ....</b>	<b>26</b>
<b>Table 3-5. Number of laboratories reporting use of qualitative and quantitative analysis for total <math>\Delta^9</math>-THC. ....</b>	<b>26</b>
<b>Table 3-6. Within-laboratory and between-laboratory variabilities for the determination of <math>\Delta^9</math>-THC, THCA, and total <math>\Delta^9</math>-THC. ....</b>	<b>28</b>
<b>Table 3-7. Summary of sample preparation and analytical methods used by participants reporting results for <math>\Delta^9</math>-THC, THCA, and total <math>\Delta^9</math>-THC.....</b>	<b>29</b>
<b>Table 3-8. Within- and between-laboratory variabilities for <math>\Delta^9</math>-THC measurements using candidate standardized analytical methods. ....</b>	<b>44</b>
<b>Table 3-9. Within- and between-laboratory variabilities for THCA measurements using candidate standardized analytical methods. ....</b>	<b>60</b>
<b>Table 3-10. Reporting for total <math>\Delta^9</math>-THC based on qualitative assessments. ....</b>	<b>74</b>
<b>Table 3-11. Within- and between-laboratory variabilities for total <math>\Delta^9</math>-THC measurements using candidate standardized analytical methods. ....</b>	<b>75</b>
<b>Table 4-1. Example individualized data summary table for CBD, CBDA, and total CBD.....</b>	<b>78</b>
<b>Table 4-2. Reporting statistics for the enrollment to measure CBD, CBDA, and total CBD. ....</b>	<b>79</b>
<b>Table 4-3. Number of laboratories reporting quantitative mass fractions for CBD, CBDA, and total CBD. ....</b>	<b>79</b>
<b>Table 4-4. Within-laboratory and between-laboratory variabilities for the determination of CBD, CBDA, and total CBD. ....</b>	<b>81</b>
<b>Table 4-5. Percent of laboratories reporting specific sample preparation and analytical methods for the determination of CBD, CBDA, and total CBD. ....</b>	<b>81</b>
<b>Table 4-6. Within- and between-laboratory variabilities for CBD measurements using candidate standardized analytical methods. ....</b>	<b>92</b>
<b>Table 4-7. Within- and between-laboratory variabilities for CBDA measurements using candidate standardized analytical methods. ....</b>	<b>104</b>
<b>Table 5-1. Example individualized data summary table for CBC, CBCA, CBDV, and CBDVA. ....</b>	<b>116</b>
<b>Table 5-2. Example individualized data summary table for CBG, CBGA, CBL, and CBLA. ....</b>	<b>117</b>
<b>Table 5-3. Example individualized data summary table for CBN, CBNA, THCV, THCVA, and <math>\Delta^8</math>-THC. ..</b>	<b>118</b>
<b>Table 5-4. Reporting statistics for the enrollment to measure minor cannabinoids. ....</b>	<b>119</b>
<b>Table 5-5. Percent of reporting laboratories reporting quantitative mass fractions for minor cannabinoids. ....</b>	<b>120</b>
<b>Table 5-6. Percent of laboratories reporting specific sample preparation and analytical methods for the determination of minor cannabinoids.....</b>	<b>121</b>
<b>Table 5-7. Within-laboratory and between-laboratory variabilities for the determination of minor cannabinoids. ....</b>	<b>124</b>

**Table 5-8. Within- and between-laboratory variabilities for minor cannabinoid measurements using candidate standardized analytical methods. ....157**

**List of Figures**

<b>Fig. 1-1. Example data sample summary view. ....</b>	<b>8</b>
<b>Fig. 1-2. Example sample/sample comparison view.....</b>	<b>9</b>
<b>Fig. 2-1. Chromatogram of a calibrant analyzed using the LC-PDA method.....</b>	<b>12</b>
<b>Fig. 2-2. Total ion chromatogram of a calibrant analyzed using the LC-MS/MS method. ....</b>	<b>15</b>
<b>Fig. 2-3. Example UV spectral comparison between reference and study sample spectra. ....</b>	<b>17</b>
<b>Fig. 3-1. Percent difference between the consensus mean and target value for <math>\Delta^9</math>-THC, THCA, and total <math>\Delta^9</math>-THC. ....</b>	<b>27</b>
<b>Fig. 3-2. Laboratory means for <math>\Delta^9</math>-THC in NRC HEMP-1 and Plant Sample 6 (sample/sample comparison view). ....</b>	<b>31</b>
<b>Fig. 3-3. Laboratory means for <math>\Delta^9</math>-THC in NRC HEMP-1 and Plant Sample 4 (sample/sample comparison view). ....</b>	<b>32</b>
<b>Fig. 3-4. Laboratory means for <math>\Delta^9</math>-THC in Plant Sample 4 and Plant Sample 6 (sample/sample comparison view). ....</b>	<b>33</b>
<b>Fig. 3-5. <math>\Delta^9</math>-THC in NRC HEMP-1 (data summary view – analytical method). ....</b>	<b>35</b>
<b>Fig. 3-6. <math>\Delta^9</math>-THC in Plant Sample 4 (data summary view – analytical method). ....</b>	<b>36</b>
<b>Fig. 3-7. <math>\Delta^9</math>-THC in Plant Sample 6 (data summary view – analytical method). ....</b>	<b>37</b>
<b>Fig. 3-8. <math>\Delta^9</math>-THC in Plant Sample 2 (data summary view – analytical method). ....</b>	<b>38</b>
<b>Fig. 3-9. <math>\Delta^9</math>-THC in Plant Sample 3 (data summary view – analytical method). ....</b>	<b>39</b>
<b>Fig. 3-10. <math>\Delta^9</math>-THC in Plant Sample 5 (data summary view – analytical method). ....</b>	<b>40</b>
<b>Fig. 3-11. Laboratory Z' scores for <math>\Delta^9</math>-THC for laboratories B001 to B114. ....</b>	<b>42</b>
<b>Fig. 3-12. Laboratory Z' scores for <math>\Delta^9</math>-THC for laboratories B115 to B236. ....</b>	<b>43</b>
<b>Fig. 3-13. Absorbance spectra rejects initial identification of coeluting <math>\Delta^9</math>-THC peak.....</b>	<b>46</b>
<b>Fig. 3-14. Absorbance spectra confirms identification of coeluting <math>\Delta^9</math>-THC peak. ....</b>	<b>47</b>
<b>Fig. 3-15. LC-UV chromatogram at 220 nm for a mixture of <math>\Delta^9</math>-THC, <math>\Delta^8</math>-THC, and CBNA calibration solutions.....</b>	<b>47</b>
<b>Fig. 3-16. The impact of calibration curve design. ....</b>	<b>49</b>
<b>Fig. 3-17. Laboratory means for THCA in Plant Sample 6 and NRC HEMP-1 (sample/sample comparison view). ....</b>	<b>51</b>
<b>Fig. 3-18. Laboratory means for THCA in Plant Sample 3 and Plant Sample 5 (sample/sample comparison view). ....</b>	<b>52</b>
<b>Fig. 3-19. THCA in NRC HEMP-1 (data summary view – analytical method).....</b>	<b>54</b>
<b>Fig. 3-20. THCA in Plant Sample 4 (data summary view – analytical method). ....</b>	<b>55</b>
<b>Fig. 3-21. THCA in Plant Sample 6 (data summary view – analytical method). ....</b>	<b>56</b>

<b>Fig. 3-22. THCA in Plant Sample 2 (data summary view – analytical method).</b> .....	57
<b>Fig. 3-23. THCA in Plant Sample 3 (data summary view – analytical method).</b> .....	58
<b>Fig. 3-24. THCA in Plant Sample 5 (data summary view – analytical method).</b> .....	59
<b>Fig. 3-25. Laboratory means for total <math>\Delta^9</math>-THC in NRC HEMP-1 and Plant Sample 6 (sample/sample comparison view).</b> .....	64
<b>Fig. 3-26. Laboratory means for total <math>\Delta^9</math>-THC in Plant Sample 3 and Plant Sample 5 (sample/sample comparison view).</b> .....	65
<b>Fig. 3-27. Total <math>\Delta^9</math>-THC in NRC HEMP-1 (data summary view – analytical method).</b> .....	66
<b>Fig. 3-28. Total <math>\Delta^9</math>-THC in Plant Sample 4 (data summary view – analytical method).</b> .....	67
<b>Fig. 3-29. Total <math>\Delta^9</math>-THC in Plant Sample 4 (data summary view – GC methods).</b> .....	68
<b>Fig. 3-30. Total <math>\Delta^9</math>-THC in Plant Sample 6 (data summary view – analytical method).</b> .....	69
<b>Fig. 3-31. Total <math>\Delta^9</math>-THC in Plant Sample 2 (data summary view – analytical method).</b> .....	70
<b>Fig. 3-32. Total <math>\Delta^9</math>-THC in Plant Sample 3 (data summary view – analytical method).</b> .....	71
<b>Fig. 3-33. Total <math>\Delta^9</math>-THC in Plant Sample 3 (data summary view – GC methods).</b> .....	72
<b>Fig. 3-34. Total <math>\Delta^9</math>-THC in Plant Sample 5 (data summary view – analytical method).</b> .....	73
<b>Fig. 4-1. Percent difference between the consensus mean and target value for CBD, CBDA, and total CBD.</b> .....	80
<b>Fig. 4-2. Laboratory means for CBD in NRC HEMP-1 and Plant Sample 4 (sample/sample comparison view).</b> .....	83
<b>Fig. 4-3. Laboratory means for CBD in Plant Sample 2 and Plant Sample 5 (sample/sample comparison view).</b> .....	84
<b>Fig. 4-4. CBD in NRC HEMP-1 (data summary view – analytical method).</b> .....	86
<b>Fig. 4-5. CBD in Plant Sample 4 (data summary view – analytical method).</b> .....	87
<b>Fig. 4-6. CBD in Plant Sample 6 (data summary view – analytical method).</b> .....	88
<b>Fig. 4-7. CBD in Plant Sample 2 (data summary view – analytical method).</b> .....	89
<b>Fig. 4-8. CBD in Plant Sample 3 (data summary view – analytical method).</b> .....	90
<b>Fig. 4-9. CBD in Plant Sample 5 (data summary view – analytical method).</b> .....	91
<b>Fig. 4-10. Laboratory means for CBDA in NRC HEMP-1 and Plant Sample 6 (sample/sample comparison view).</b> .....	95
<b>Fig. 4-11. Laboratory means for CBDA in Plant Sample 3 and Plant Sample 5 (sample/sample comparison view).</b> .....	96
<b>Fig. 4-12. CBDA in NRC HEMP-1 (data summary view – analytical method).</b> .....	98
<b>Fig. 4-13. CBDA in Plant Sample 4 (data summary view – analytical method).</b> .....	99
<b>Fig. 4-14. CBDA in Plant Sample 6 (data summary view – analytical method).</b> .....	100
<b>Fig. 4-15. CBDA in Plant Sample 2 (data summary view – analytical method).</b> .....	101
<b>Fig. 4-16. CBDA in Plant Sample 3 (data summary view – analytical method).</b> .....	102
<b>Fig. 4-17. CBDA in Plant Sample 5 (data summary view – analytical method).</b> .....	103

<b>Fig. 4-18. Total CBD in NRC HEMP-1 (data summary view – analytical method).</b> .....	<b>107</b>
<b>Fig. 4-19. Total CBD in Plant Sample 4 (data summary view – analytical method).</b> .....	<b>108</b>
<b>Fig. 4-20. Total CBD in Plant Sample 4 (data summary view – GC methods).</b> .....	<b>109</b>
<b>Fig. 4-21. Total CBD in Plant Sample 6 (data summary view – analytical method).</b> .....	<b>110</b>
<b>Fig. 4-22. Total CBD in Plant Sample 2 (data summary view – analytical method).</b> .....	<b>111</b>
<b>Fig. 4-23. Total CBD in Plant Sample 3 (data summary view – analytical method).</b> .....	<b>112</b>
<b>Fig. 4-24. Total CBD in Plant Sample 5 (data summary view – analytical method).</b> .....	<b>113</b>
<b>Fig. 5-1. Minor cannabinoid mass fraction (%) comparison between consensus and target values.</b> .....	<b>122</b>
<b>Fig. 5-2. CBC in NRC HEMP-1 (data summary view – analytical method).</b> .....	<b>126</b>
<b>Fig. 5-3. CBC in Plant Sample 4 (data summary view – analytical method).</b> .....	<b>127</b>
<b>Fig. 5-4. CBC in Plant Sample 6 (data summary view – analytical method).</b> .....	<b>128</b>
<b>Fig. 5-5. CBC in Plant Sample 2 (data summary view – analytical method).</b> .....	<b>129</b>
<b>Fig. 5-6. CBC in Plant Sample 3 (data summary view – analytical method).</b> .....	<b>130</b>
<b>Fig. 5-7. CBC in Plant Sample 5 (data summary view – analytical method).</b> .....	<b>131</b>
<b>Fig. 5-8. CBCA in NRC HEMP-1 (data summary view – analytical method).</b> .....	<b>132</b>
<b>Fig. 5-9. Laboratory means for CBC in NRC HEMP-1 and Plant Sample 4 (sample/sample comparison view).</b> .....	<b>134</b>
<b>Fig. 5-10. Chromatogram and absorbance spectra for tentative CBC peak.</b> .....	<b>135</b>
<b>Fig. 5-11. LC-UV chromatograms at 220 nm for CBC and THCA reference standards under different conditions.</b> .....	<b>135</b>
<b>Fig. 5-12. CBDV in NRC HEMP-1 (data summary view – analytical method).</b> .....	<b>137</b>
<b>Fig. 5-13. CBDVA in NRC HEMP-1 (data summary view – analytical method).</b> .....	<b>138</b>
<b>Fig. 5-14. Absorbance spectra for chromatographic peaks identified as CBDV and CBDVA in Plant Sample 4.</b> .....	<b>139</b>
<b>Fig. 5-15. CBG in NRC HEMP-1 (data summary view – analytical method).</b> .....	<b>141</b>
<b>Fig. 5-16. CBG in Plant Sample 5 (data summary view – analytical method).</b> .....	<b>142</b>
<b>Fig. 5-17. CBGA in NRC HEMP-1 (data summary view – analytical method).</b> .....	<b>143</b>
<b>Fig. 5-18. Absorbance spectra for chromatographic peaks identified as CBG and CBGA in Plant Sample 4.</b> .....	<b>144</b>
<b>Fig. 5-19. CBL in NRC HEMP-1 (data summary view – analytical method).</b> .....	<b>145</b>
<b>Fig. 5-20. CBLA in NRC HEMP-1 (data summary view – analytical method).</b> .....	<b>146</b>
<b>Fig. 5-21. Absorbance spectra for chromatographic peak identified as CBL in Plant Sample 4.</b> .....	<b>147</b>
<b>Fig. 5-22. CBN in NRC HEMP-1 (data summary view – analytical method).</b> .....	<b>148</b>
<b>Fig. 5-23. CBN in Plant Sample 3 (data summary view – analytical method).</b> .....	<b>149</b>
<b>Fig. 5-24. CBN in Plant Sample 5 (data summary view – analytical method).</b> .....	<b>150</b>
<b>Fig. 5-25. CBNA in NRC HEMP-1 (data summary view – analytical method).</b> .....	<b>151</b>

<b>Fig. 5-26. Chromatogram and absorbance spectra for tentatively identified CBN peak in Plant Sample 3.....</b>	<b>152</b>
<b>Fig. 5-27. THCV in NRC HEMP-1 (data summary view – analytical method).....</b>	<b>154</b>
<b>Fig. 5-28. THCVA in NRC HEMP-1 (data summary view – analytical method). .....</b>	<b>155</b>
<b>Fig. 5-29. Absorbance spectra for chromatographic peak identified as THCV in Plant Sample 3.....</b>	<b>156</b>

## Acknowledgments

Maryam Abdur-Rahman, Bruce A. Benner, Carolyn Q. Burdette, Jacolin A. Murray, Jerome J. Mulloor, and Aaron A. Urbas are acknowledged for their work on the preparation and packaging of plant materials.

Shaun P. Kotoski is acknowledged for his work on the preparation of data tables and figures for preliminary results.

Catherine A. Rimmer, David L. Duewer, and Edward Sisco of the NIST Material Measurement Laboratory are acknowledged for their careful review of this report.

## Executive Summary

Two hundred and twenty-six laboratories signed up to participate in Exercise 2 of the Cannabis Laboratory Quality Assurance Program (CannaQAP) to measure cannabinoids, toxic elements, and/or moisture in six cannabis plant samples. Approximately 90 % and 22 % of laboratories registered to submit results for cannabinoids in three hemp and marijuana plant samples, respectively. Plant Sample 1, Plant Sample 4, and Plant Sample 6 were materials prepared with total  $\Delta^9$ -THC mass fractions  $\leq 0.3\%$ . Plant Sample 1 was a Certified Reference Material prepared by the National Research Council of Canada (NRC HEMP-1). Plant Sample 4 and Plant Sample 6 were grown in the United States and prepared at NIST following normal reference material procedures [1]. Plant Sample 2, Plant Sample 3, and Plant Sample 5 were marijuana materials prepared at NIST to have  $\Delta^9$ -THC mass fractions at approximately 0.5 %, 1 %, and 2 %, respectively. Target mass fraction values were provided for Plant Sample 2 through Plant Sample 6 by NIST and determined using liquid chromatography (LC) combined with photodiode array detection (PDA) and/or tandem mass spectrometry (MS/MS). Target mass fractions were provided for two sets of major cannabinoids ( $\Delta^9$ -THC, THCA, total  $\Delta^9$ -THC and CBD, CBDA, total CBD) and 13 minor cannabinoids (CBC, CBCA, CBDV, CBDVA, CBG, CBGA, CBL, CBLA, CBN, CBNA, and  $\Delta^8$ -THC).

Laboratories generally provided more accurate cannabinoid results when the mass fractions were above their method limits of quantitation but not high enough to require sample dilutions. Difficulties with accurate quantitation of lower mass fraction samples could be the result of calibration bias at the lower end of the calibration curve, increased impact from chromatographic interferences, or cannabinoid misidentification. To prevent calibration bias, laboratories should use calibration standards that meet ISO standards, ensure all purity information is reviewed, independently prepare calibrants routinely, and are traceable to the International System of Units (SI), if possible. Difficulty measuring cannabinoids with high mass fraction levels ( $\geq 1\%$ ) was observed for THCA and CBDA, which may be a result of calibration bias at the high end of the calibration curve or poor sample dilution procedures. While dilutions are typically made volumetrically, gravimetric dilution is the recommended method due to the increased accuracy.

To reduce the impact of interfering matrix components on the quantitative measurements of cannabinoids, chromatographic methods should be periodically evaluated to ensure baseline separation of known cannabinoids with similar retention times. For example, the consensus value for  $\Delta^9$ -THC in NRC HEMP-1 was bias high, which was likely a result of a coeluting CBNA peak falsely increasing the  $\Delta^9$ -THC peak response. The consensus value was also higher than the target value for 8 out of the 12 minor cannabinoids in the NRC HEMP-1 sample. Certified mass fractions for cannabinoids in NRC HEMP-1 were assigned by LC-MS/MS, but approximately 80 % of laboratories used LC with absorbance detection, which is less selective. In most instances, laboratories are relying solely on retention times for selectivity and cannabinoid identification. LC absorbance measurements performed at NIST with a PDA detector provided examples of how four of the minor cannabinoids could be misidentified when relying solely on retention time for cannabinoid confirmation.

The majority of within-laboratory (repeatability, %RSD<sub>r</sub>) variabilities did meet the acceptable repeatability criteria outlined by AOAC International Standard Method Performance Requirements (SMPR) for hemp plant samples (e.g., %RSD<sub>r</sub> ≤ 5% for THCA). However, almost none of the between-laboratory (reproducibility, %RSD<sub>R</sub>) variabilities met the AOAC requirements (e.g., %RSD<sub>R</sub> ≤ 10 % for THCA). The criteria for between-laboratory precision are meant to be applied to variabilities from multiple laboratories using a single analytical method, not variabilities from multiple laboratories using multiple analytical methods, as was the case for this study. As part of this exercise, NIST provided a list of nine candidate analytical methods from AOAC and ASTM international standard organizations for participants to use if an in-house analytical method was not available. Laboratories using these methods generally provided repeatability results that meet AOAC criteria but did not provide reproducible results for most samples.

## 1. Introduction

The Cannabis Laboratory Quality Assurance Program (CannaQAP) was formed in 2020 to assist laboratories in demonstrating and improving measurement comparability and competence in cannabis plant and cannabis-derived matrices. CannaQAP offers the opportunity for participating laboratories to assess their in-house measurements of cannabinoids, moisture content, and toxic elements, with areas of expansion to include contaminants (pesticides and mycotoxins) and terpenes. Reports and certificates of participation are provided to laboratories that may be used to validate their analytical scheme, demonstrate compliance with cGMPs, and fulfill proficiency requirements established by related accreditation bodies when PTs are not available. In the future, results from CannaQAP exercises may be used by NIST to identify problematic matrices and analytes for which consensus-based methods of analysis would benefit the stakeholders in numerous cannabis communities.

CannaQAP provides matrices and analytes to the community that are used to assess existing sample preparation and analytical methods within the measurement community. Each exercise is designed to identify biases among the different sample extraction techniques, analytical methods, and/or calibration approaches by incorporating emerging and challenging measurements in Cannabis and Cannabis-derived matrices. Participating laboratories can use the results to demonstrate the accuracy and comparability of their in-house methods with respect to the measurement community. In areas where few quality control materials have been developed, CannaQAP offers a tool for assessment of the quality of measurements and provides feedback about performance that can assist participants in improving laboratory operations.

In the second exercise of CannaQAP, seven plant samples were shipped to participants for analysis of cannabinoids, moisture, and toxic elements. This report summarizes the results of the cannabinoid measurements, the moisture [2] and toxic element [3] reports were published separately. Two hundred and twenty-six laboratories responded to the call for participants in January 2021. Samples were shipped to participants in April 2021 and results were returned to NIST by May 2021. Participants received a summary of the preliminary data in May 2021 and had the opportunity to correct any data entry errors before the preliminary certificates were sent out in July 2021. This report contains the final cannabinoid results and detailed discussions for potential biases.

### 1.1. Overview

In addition to this report, individualized data tables and certificates are provided to the participants who submitted data. Community tables and figures are provided to participants using randomized laboratory codes, with identities known only to NIST and individual laboratories. Examples of these data tables are included in each section of this report with the individual laboratory results section omitted. The statistical approaches are outlined below for each type of data representation.

### 1.1.1. Statistics

Data tables and figures throughout this report contain information about the performance of each laboratory relative to that of the other participants in this study and, if available, relative to a target around the expected result. All calculations are performed in PROLab Plus (QuoData GmbH, Dresden, Germany). The consensus means and standard deviations are calculated according to the robust Q/Hampel method outlined in ISO 13528:2022, Annex C [4].

### 1.1.2. Individualized Data Table

The individualized data table contains data that is specific to each participating laboratory. The purpose of the table is to allow participants to directly compare their data to the summary statistics (consensus or community data as well as NIST-certified, non-certified, or estimated values, when available). Participating laboratories received uniquely coded individualized data tables in a separate distribution that included a randomized laboratory code, located in the upper left section of the data table. Example individualized data tables are included in this report with the section allocated for individual laboratory data (Section 1. Your Results) shaded as illustrated in **Table 1-1**.

**Table 1-1. Example of an individualized data table.**

Lab Code: (Code)		Section 1. Your Results				Section 2. Community Results			Section 3. Target		
	Sample <sup>a</sup>	Units <sup>b</sup>	$x_i$	$s_i$	$Z'_{\text{comm}}$	$Z_{\text{NIST}}$	$N$	$x^*$	$s^*$	$x_{\text{NIST}}$	$u$
C <sub>1</sub> <sup>c</sup>	a <sub>1</sub>	b <sub>1</sub>	<i>Individual laboratory results will appear in this section; laboratory-specific results were provided to each participant separately from this report.</i>				$N_1$	$x^*_1$	$s^*_1$	$x_{\text{NIST}1}$	$u_1$
...	...	...					...	...	...	...	...
...	...	...					...	...	...	...	...
C <sub>n</sub>	a <sub>n</sub>	b <sub>n</sub>					$N_n$	$x^*_n$	$s^*_n$	$x_{\text{NIST}n}$	$u_n$

$x_i$  Mean of reported values  
 $s_i$  Standard deviation of reported values  
 $Z'_{\text{comm}}$  Z'-score with respect to community consensus  
 $Z_{\text{NIST}}$  Z-score with respect to NIST value

$N$  Number of quantitative values reported  
 $x^*$  Robust mean of reported values  
 $s^*$  Robust standard deviation

<sup>a</sup> Samples used in the study.

<sup>b</sup> Units used to describe the measured values.

<sup>c</sup> Analytes measured in the study.

Section 1 of the data table (Your Results) contains the laboratory results as reported, including the mean and standard deviation when multiple values were reported. A blank indicates that NIST does not have data on file for that laboratory for the corresponding analyte. An empty box for standard deviation indicates that the participant reported a single value or a limit of quantitation (LOQ).

Also included in Section 1 are two Z-scores. The first Z-score,  $Z'_{\text{comm}}$ , is calculated with respect to the community consensus value, taking into consideration bias that may result from the

uncertainty in the assigned consensus value, using the consensus mean ( $x^*$ ), robust estimate of the standard deviation of the reported values ( $s^*$ ), and standard deviation for proficiency assessment ( $\sigma_{PT}$ ) determined from the Q/Hampel estimator Eq. (1).

$$Z'_{\text{comm}} = \frac{x_i - x^*}{\sqrt{\sigma_{PT}^2 + s^{*2}}} \quad (1)$$

The second Z-score,  $Z_{NIST}$ , is calculated with respect to the target value ( $x_{NIST}$ , NIST certified, non-certified, or estimated value, when available), and either  $U_{95}$ , where  $U_{95}$  is the expanded uncertainty on an assigned value), Eq. (2), or  $U_{NIST}$ (where  $U_{NIST}$ is an estimated expanded uncertainty of NIST and/or other measurements), Eq (3).

$$Z_{NIST} = \frac{x_i - x_{NIST}}{2 * U_{95}} \quad (2)$$

$$Z_{NIST} = \frac{x_i - x_{NIST}}{2 * U_{NIST}} \quad (3)$$

The significance of the Z-score and  $Z'$ -score is as follows [4]:

- $|Z| < 2$  indicates that the laboratory result is considered to be within the community consensus range (for  $Z'_{\text{comm}}$ ) or NIST target range (for  $Z_{NIST}$ ).
- $2 < |Z| < 3$  indicates that the laboratory result is considered to be marginally different from the community consensus value (for  $Z'_{\text{comm}}$ ) or NIST target value (for  $Z_{NIST}$ ).
- $|Z| > 3$  indicates that the laboratory result is considered to be significantly different from the community consensus value (for  $Z'_{\text{comm}}$ ) or NIST target value (for  $Z_{NIST}$ ).

Section 2 of the data table (Community Results) contains the consensus results, including the number of laboratories reporting quantitative values for each analyte ( $n$ ), the mean value determined for each analyte ( $x^*$ ), and a robust estimate of the standard deviation of the reported values ( $s^*$ ) [4]. Additional information on calculation of the consensus mean and standard deviation can be found in Section 1.1.1.

Section 3 of the data table (Target) contains the NIST target values for each analyte ( $x_{NIST}$ ), when available. When possible, the target value is a certified value, a non-certified value, or a value determined at NIST. A NIST certified value is a value for which NIST has the highest confidence in its accuracy and that all known or suspected sources of bias and variability have been considered [5]. For samples in which a NIST certified or non-certified value is not available, a target value may be determined at NIST using an established method, or data from a collaborating laboratory. The target value represents the mean of at least three replicates. For materials acquired from and/or evaluated as a part of another interlaboratory study or proficiency testing program, the consensus value and uncertainty from the completed round is used as the target range.

In this study, target values for the plant samples were determined at NIST using liquid chromatography with photodiode array (LC-PDA) and liquid chromatography with tandem mass spectrometry (LC-MS/MS) methods summarized in Section 2.3. The target values represent the mean of a minimum of three independent measurements, which permitted NIST to provide an expanded uncertainty ( $U_{NIST}$ ) to encompass variability due to inhomogeneity between packaged

units. A unique feature of NIST QAPs is the accuracy-based component provided by comparison of participant results to a NIST-measured value.

### 1.1.3. Summary Data Table

This data table includes a summary of all reported data for a particular analyte in a particular study. Participants can compare the raw data from their laboratory to data reported by the other participating laboratories and to the consensus data. A blank indicates that the laboratory signed up and received samples for that analyte and matrix, but NIST does not have data on file for that laboratory. The standard deviation (SD) for the target value in this table is the uncertainty ( $U_{NIST}$  or  $U_{95}$ ) around the target value. Data highlighted in red have been flagged as a data entry of zero or results that include text (e.g., “< LOQ” or “present”). Data highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to yield  $|Z'_{comm}| > 2$ . The summary data tables are presented in the format shown in **Table 1-2**.

**Table 1-2. Example data summary table.**

		Analyte									
		Sample 1 (units)				Sample 2 (units)					
		A	B	C	Avg <sup>a</sup>	SD <sup>b</sup>	A	B	C	Avg	SD
Individual Results	Target				c <sub>1</sub>	d <sub>1</sub>				c <sub>2</sub>	d <sub>2</sub>
	e <sub>1</sub>	X <sub>A1-1</sub>	X <sub>B1-1</sub>	X <sub>C1-1</sub>	$\bar{X}_{1-1}$	$S_{1-1}$	X <sub>A2-1</sub>	X <sub>B2-1</sub>	X <sub>C2-1</sub>	$\bar{X}_{1-2}$	$S_{1-2}$
	...	...	...	...	...	...	...	...	...	...	...
	e <sub>n</sub>	X <sub>A1-n</sub>	X <sub>B1-n</sub>	X <sub>C1-n</sub>	$\bar{X}_{n-1}$	$S_{n-1}$	X <sub>A2-n</sub>	X <sub>B2-n</sub>	X <sub>C2-n</sub>	$\bar{X}_{n-2}$	$S_{n-2}$
Community Results		Consensus Mean			f <sub>1</sub>		Consensus Mean			f <sub>2</sub>	
		Consensus Standard Deviation			g <sub>1</sub>		Consensus Standard Deviation			g <sub>2</sub>	
		Maximum			h <sub>1</sub>		Maximum			h <sub>2</sub>	
		Minimum			i <sub>1</sub>		Minimum			i <sub>2</sub>	
	N				j <sub>1</sub>		N			j <sub>2</sub>	

a Arithmetic average of sample replicates.

b Standard deviation of sample replicates.

c Target value for the sample.

d Standard deviation of the target value for the sample.

e Laboratory identifier for the participant.

f Robust mean of reported results.

g Robust standard deviation of reported results.

h Maximum of reported average results.

i Minimum of reported average results.

j Number of quantitative values reported.

### 1.1.4. Figures

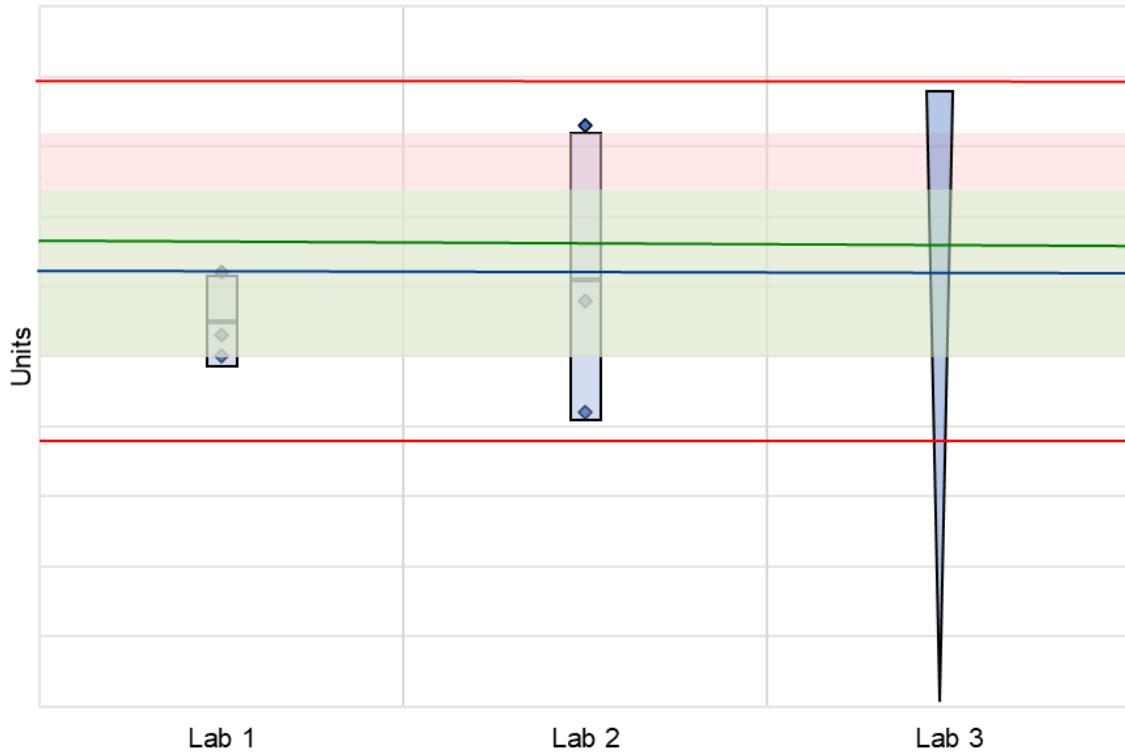
#### 1.1.4.1. Data Summary View (Method Comparison Data Summary View)

In this view (**Fig. 1-1**), individual laboratory data (diamonds) and the individual laboratory SD (rectangle) are plotted. Laboratories reporting values below the LOQ are shown in this view as downward triangles beginning at the LOQ, reported as Quantification Limit (QL) on the figures. Laboratories reporting values below LOQ can still be successful in the study if the target value is also below the laboratory LOQ. The solid blue line represents the consensus mean and the green

shaded area represents the 95 % confidence interval for the consensus mean, which is based on the standard uncertainty of the consensus mean ( $u_{\text{mean}}$ ). The uncertainty in the consensus mean is calculated using the repeatability standard deviation ( $s_r$ ), the reproducibility standard deviation ( $s_R$ ), the number of participants reporting data ( $n_{\text{participants}}$ ), and the average number of replicates reported by each participant ( $n_{\text{Average Number of Replicates per Participant}}$ ) (Eq. 4). The uncertainty about the consensus mean is independent of the range of tolerance (solid red lines). Where appropriate, two consensus means may be calculated for the same sample if bimodality is identified in the data. In this case, two consensus means and ranges will be displayed in the data summary view.

$$u_{\text{mean}} = \sqrt{\frac{s_R^2 - s_r^2}{n_{\text{participants}}} + \frac{s_R^2}{n_{\text{participants}} \times n_{\text{Average Number of Replicates per Participant}}}} \quad (4)$$

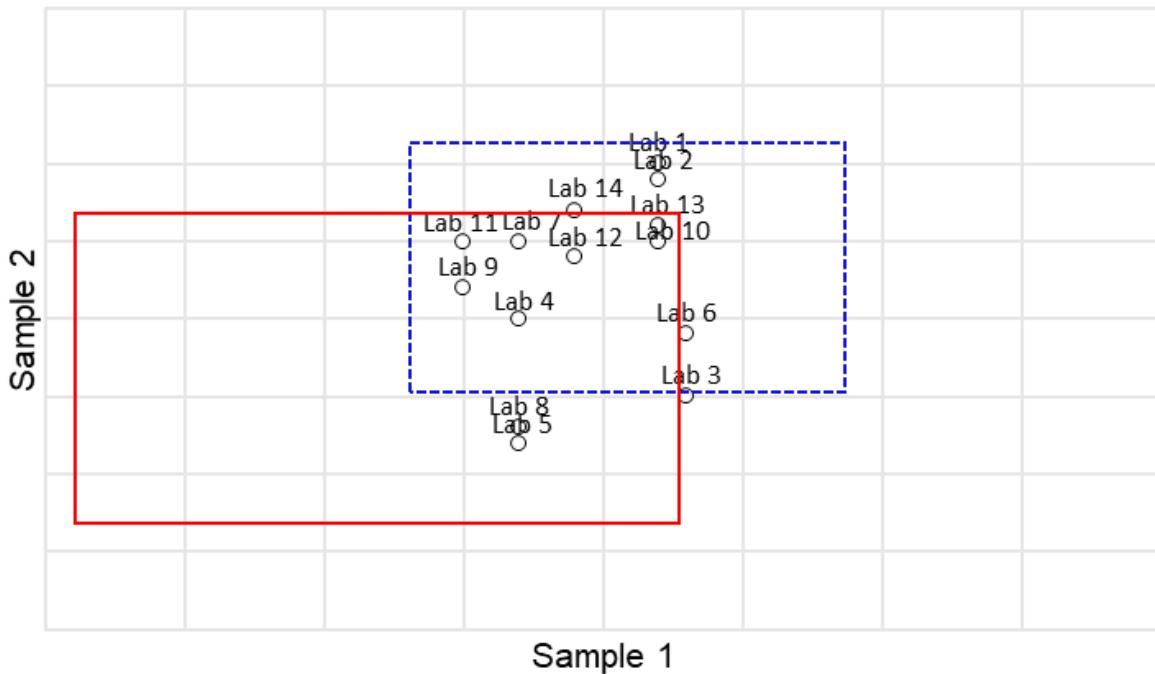
The red shaded region in the data summary figure represents the target range for “acceptable” performance, which encompasses the NIST target value (solid green line) bounded by twice its uncertainty ( $U_{95}$  or  $U_{\text{NIST}}$ ). The solid red lines represent the range of tolerance (values that result in an acceptable Z'-score,  $|Z'| \leq 2$ ). If the lower limit is below zero, the lower limit is set to zero. In the data summary view, the relative locations of individual laboratory data and consensus ranges with respect to the target range can be compared easily. In most cases, the target range, and the consensus range overlap, which is the expected result. Major program goals include both reducing the size of the consensus range and centering the consensus range about the target value. Analysis of an appropriate reference material as part of a quality control scheme can help to identify sources of bias for laboratories reporting results that are significantly different from the target range. In the case in which a method comparison is relevant, different colored data points may be used to identify laboratories that used a specific approach for sample preparation, analysis, or quantitation.



**Fig. 1-1. Example data sample summary view.**

#### **1.1.4.2. Data Summary View (Method Comparison Data Summary View)**

In this view (**Fig. 1-2**), the individual laboratory results for one sample (e.g., NIST Standard Reference Material<sup>®</sup> (SRM<sup>®</sup>) or Reference Material (RM) with a certified, non-certified, or NIST-determined value; a less challenging matrix) are compared to the results for another sample (e.g., NIST SRM with a more challenging matrix; a commercial sample). The solid red box represents the target range for the first sample (x-axis) and the second sample (y-axis), if available. The dotted blue box represents the consensus range for the first sample (x-axis) and the second sample (y-axis). The axes of this graph are centered about the consensus mean values for each sample or control, to a limit of twice the range of tolerance. Depending on the variability in the data, the axes may be scaled proportionally to better display the individual data points for each laboratory. In some cases, when the consensus and target ranges have limited overlap, the solid red box may only appear partially on the graph. If the variability in the data is high (greater than 100 % relative standard deviation (RSD)), the dotted blue box may also only appear partially on the graph. These views emphasize trends in the data that may indicate potential calibration issues or method biases. Primary program goals are to identify such calibration or method biases and assist participants in improving analytical measurement capabilities. In some cases, when two equally challenging materials are provided, the same view (sample/sample comparison) can be helpful in identifying commonalities or differences in the analysis of the two materials.



**Fig. 1-2. Example sample/sample comparison view.**

## 2. Study Material Preparation and Characterization

### 2.1. Study Material Acquisition and Preparation

#### 2.1.1. NRC HEMP-1 (Plant Sample 1)

NRC HEMP-1, a dried, ground hemp Certified Reference Material (CRM) developed by the National Research Council of Canada (NRC) [6] was used for Plant Sample 1. NIST received 108 amber bottles of NRC HEMP-1, each containing at least 10 g of ground hemp that was stored at -20 °C prior to shipment. After arrival at NIST, these bottles were combined into a single container, blended for 15 min, and then stored at -80 °C until being packaged. Approximately 3 g of NRC HEMP-1 was weighed and sealed into plastic bags, then each plastic bag was sealed into an aluminized mylar bag along with a desiccant silica pouch. NRC HEMP-1 was manually blended throughout the packaging process to ensure homogeneity across the packaged samples. NRC HEMP-1 was stored at -20 °C until shipment to participating laboratories. Approximately 400 bags of NRC HEMP-1 were packaged.

#### 2.1.2. Plant Samples 2 through 6

Three independent bulk batches of cannabis and an additional batch of hemp stems, freshly harvested in the United States, were used to prepare the plant samples provided for this study. Bulk batches of cannabis were delivered to NIST containing buds, leaves, and stems. An additional package of stems was delivered to NIST to use as a Δ<sup>9</sup>-THC dilution for the hemp samples. The bulk material for each sample was ground and sieved, then the combinations of cannabis for each sample were mixed for 10 min to 30 min, then stored at -80 °C prior to being packaged and shipped to participants. The cannabis mixture and sieve size were based on the desired Δ<sup>9</sup>-THC mass fraction of the sample. **Table 2-1** details the composition and particle size of Plant Sample 2 through Plant Sample 6.

**Table 2-1. Preparation of Plant Samples 2 through 6 used in CannaQAP Exercise 2.**

<b>Sample</b>	<b>Bulk Cannabis Used</b>	<b>Sieve Size (μm)</b>
Sample 2	Cannabis 1: buds and leaves	< 250
Sample 3	Cannabis 1 and 2: buds and leaves	< 250
Sample 4	Cannabis 1: buds, leaves, and stems plus Stems 2	250 to 710
Sample 5	Cannabis 1 and 3: buds and leaves	< 250
Sample 6	Cannabis 1: small stems and Stems 2	< 250

After the materials were prepared, approximately 3 g of each material was weighed and sealed into plastic bags. Plant materials were manually blended throughout the packaging process to ensure homogeneity across the packaged samples. Each plastic bag was then sealed into an aluminized mylar bag along with a desiccant silica pouch and stored at -20 °C until shipment to participating laboratories. Approximately 400 bags of Plant Sample 4 and Plant Sample 6 were packaged. Approximately 100 aliquots of Plant Sample 2, Plant Sample 3, and Plant Sample 5 were packaged.

## 2.2. NIST Methods for Material Preparation and Characterization

### 2.2.1. Sample Preparation and Cannabinoid Extraction

Samples were prepared following the approach of Vaclavik *et al.*[7], modified to use methanol (MeOH) instead of ethanol (EtOH) for cannabinoid extraction. The samples were removed from the -20 °C freezer, equilibrated at room temperature for 1 h, and mixed thoroughly by hand to ensure homogeneity. Subsamples ( $\approx 0.50 \text{ g} \pm 0.05 \text{ g}$ ) of each plant sample were weighed into 50 mL polypropylene centrifuge tubes. MeOH (20 mL) was added to each sample and the sample was vortexed for 10 s to ensure initial suspension. Samples were then mechanically shaken for 30 min at room temperature and centrifuged for 5 min at 209.4 rad/s (2,000 rpm). The MeOH extract was decanted into a new 50 mL centrifuge tube and a second 20 mL aliquot of MeOH was added to the remaining solid material. After shaking and centrifugation as described above, the two extracts were combined. Small portions ( $\approx 2 \text{ mL}$ ) of the extracts were filtered through a 0.45  $\mu\text{m}$  polytetrafluoroethylene (PTFE) membrane filter (Phenomenex, AF0-1102-52) and 10-fold and 100-fold MeOH dilutions were prepared for analysis.

### 2.2.2. Analytical Methods

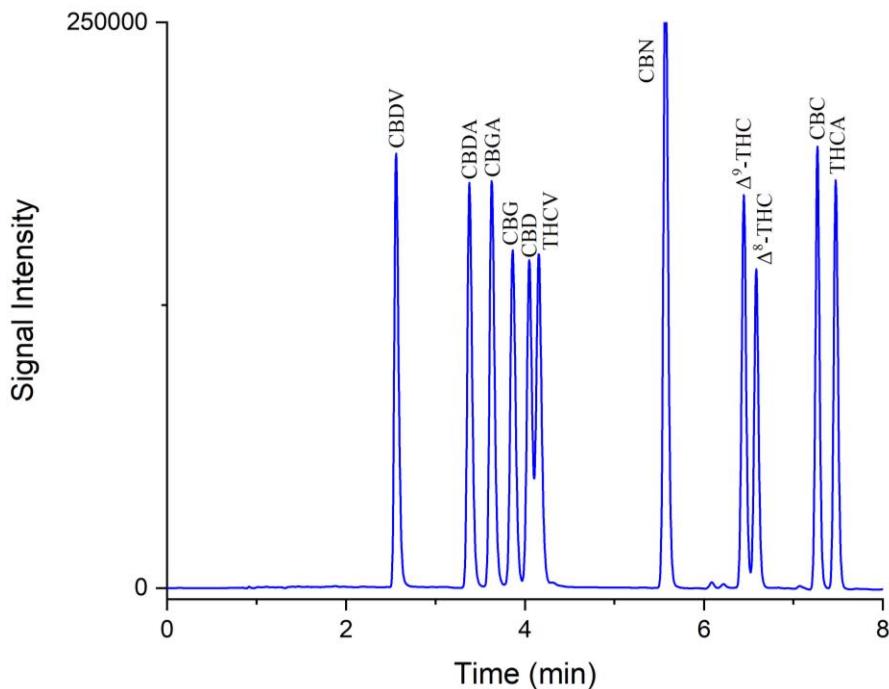
Extracts and dilutions from independent extractions were analyzed using two independent methods at NIST: LC-PDA and LC-MS/MS.

#### 2.2.2.1. LC-PDA Analysis

All study materials were characterized using a previously published LC-PDA method [8, 9, 10]. The method was validated for eleven cannabinoids: cannabidivarin (CBDV), cannabidiolic acid (CBDA), cannabidiolic acid (CBGA), cannabigerol (CBG), cannabidiol (CBD), tetrahydrocannabivarin (THCV), cannabinol (CBN),  $\Delta^9$ -tetrahydrocannabinol ( $\Delta^9$ -THC),  $\Delta^8$ -tetrahydrocannabinol ( $\Delta^8$ -THC), cannabichromene (CBC), and  $\Delta^9$ -tetrahydrocannabinolic acid (THCA). Cannabinoid separations were carried out on a NexLeaf CBX for Potency C<sub>18</sub> column with 15.0 cm length, 4.6 mm inner diameter, and 2.7  $\mu\text{m}$  average particle diameter, protected by installation of a NexLeaf CBX guard column (Shimadzu Scientific Instruments). Premixed mobile phase solvents [water (H<sub>2</sub>O) and acetonitrile (ACN) containing 0.085 % phosphoric acid (PA) (volume ratios)] were obtained from Shimadzu Scientific Instruments. The injection volume, column temperature, and flow rate were 5  $\mu\text{L}$ , 40 °C, and 1.6 mL/min, respectively. The gradient mobile phase program is summarized in **Table 2-2**. The wavelength range for the PDA detector was 190 nm to 700 nm and quantitative measurements were conducted at 220 nm for all cannabinoids unless otherwise indicated. An example chromatogram from a calibration solution containing the 11 cannabinoids in the LC-PDA method is shown in **Fig. 2-1**. The accuracy and precision of the LC-PDA method have previously been demonstrated using four University of Kentucky Proficiency Testing Program (UK-PT) reference samples [11].

**Table 2-2. Mobile phase program for the LC-PDA cannabinoid method.**

Time (min)	0.085 % PA in H <sub>2</sub> O	0.085 % PA in ACN
0.0	30	70
3.0	30	70
7.0	15	85
7.1	5	95
8.0	5	95
8.1	30	70
10.0	30	70



**Fig. 2-1. Chromatogram of a calibrant analyzed using the LC-PDA method.**

Quantitation was performed using the external standard approach with a calibration curve gravimetrically prepared from a solution mixture containing 11 cannabinoids (CBDV, CBDA, CBGA, CBG, CBD, THCV, CBN,  $\Delta^9$ -THC,  $\Delta^8$ -THC, CBC, and THCA) in ACN obtained from Shimadzu with individual concentrations of  $\approx 250$  mg/L ( $\pm 1.9$  mg/L to  $2.7$  mg/L). The final mass concentrations of each cannabinoid in the calibration solutions were approximately 2.50 mg/L, 10.0 mg/L, 25.0 mg/L, and 50 mg/L. Peak areas were plotted for each compound against the corresponding mass concentration to construct an external calibration curve. The concentration of each cannabinoid was converted from mass concentration in the extract to mass fraction in plant sample using Eq. (5),

$$w_{\text{analyte}} = \frac{\rho_{\text{analyte}} \times V_{\text{MeOH}}}{m_{\text{sample}}} \quad (5)$$

where  $\rho_{\text{analyte}}$  is the mass concentration of the cannabinoid in milligram per liter (mg/L),  $V_{\text{MeOH}}$  is the volume of methanol in liters (L),  $m_{\text{sample}}$  is the mass of plant material sampled in grams (g), and  $w_{\text{analyte}}$  is the mass fraction of the cannabinoid in milligrams per gram (mg/g).

The density of MeOH at 22.2 °C (70 °F), 0.789 g/L, was used to convert the mass of MeOH to volume of MeOH [12]. For the diluted samples, a dilution factor was calculated using Eq. (6) and the mass fraction of the diluted sample was multiplied by the dilution factor,

$$F_{\text{dilution}} = \frac{m_{\text{MeOH}} \times m_{\text{extract}}}{m_{\text{extract}}} \quad (6)$$

where  $m_{\text{MeOH}}$  is the mass of methanol in grams (g),  $m_{\text{extract}}$  is the mass of undiluted extract in grams (g), and  $F_{\text{dilution}}$  is the unitless dilution factor.

The total CBD and total  $\Delta^9$ -THC mass fractions were calculated using Eq. (7) and Eq. (8), respectively

$$w_{\text{Total CBD}} = w_{\text{CBD}} + 0.877 \times w_{\text{CBDA}} \quad (7)$$

$$w_{\text{Total THC}} = w_{\text{THC}} + 0.877 \times w_{\text{THCA}} \quad (8)$$

where the mass fractions are all expressed as percentages.

### 2.2.2.2. LC-MS/MS Analysis

Study materials were also characterized using a LC-MS/MS method developed at NIST. Chromatographic separation was performed on an Agilent Technologies 1290 Infinity II ultra-high pressure liquid chromatograph (UHPLC) (Santa Clara, CA) equipped with a quaternary pump. The column was a Restek Raptor ARC-18 (Bellefonte, PA) with 5.0 cm length, 2.1 mm inner diameter, and 2.7  $\mu\text{m}$  average particle diameter. The flow rate was 0.4 mL/min and column temperature was held at 15 °C. A binary solvent gradient method was used for separation with 0.1 % formic acid (FA) in H<sub>2</sub>O as mobile phase A and 0.1 % FA in 50:50 ACN:MeOH (by volume) as mobile phase B (**Table 2-3**). MeOH was purchased from J. T. Baker (Phillipsburg, NJ), H<sub>2</sub>O and ACN premixed with 0.1 % FA were obtained from Honeywell (Charlotte, NC), and reagent-grade FA (> 95 %) was purchased from Sigma Aldrich (St. Louis, MO). The autosampler temperature and injection volume were 10 °C and 3  $\mu\text{L}$ , respectively. Detection was performed using an Agilent 6410B triple quadrupole mass spectrometer (Santa Clara, CA). The MS was operated in positive and negative electrospray ionization (ESI) switching mode and detection was performed in scheduled multiple reaction monitoring mode (MRM) with two transitions monitored for each cannabinoid (**Table 2-4**). Neutral cannabinoids were analyzed in positive electrospray mode and acidic cannabinoids were analyzed in negative electrospray mode. The gas temperature, gas flow, nebulizer pressure, and capillary voltage were set to 350 °C, 11 L/min, 345 kPa (50 psi), and 4000 V, respectively. An example chromatogram from a calibration solution containing the 17 cannabinoids and 5 internal standards (ISs) detected in the LC-MS/MS method is shown in **Fig. 2-2**.

**Table 2-3. Mobile phase program for the LC-MS/MS cannabinoid method.**

<u>Time (min)</u>	<u>Mobile Phase A</u>	<u>Mobile Phase B</u>
<b>0.00</b>	40	60
<b>0.50</b>	40	60
<b>10.00</b>	32	68
<b>16.75</b>	32	68
<b>17.10</b>	25	75
<b>25.75</b>	21	79
<b>25.76</b>	5	95
<b>27.50</b>	5	95
<b>27.60</b>	40	60
<b>30.00</b>	40	60

**Table 2-4. MS/MS parameters for the LC-MS/MS cannabinoid method.**

<u>Cannabinoid</u>	<u>Precursor Ion (m/z)</u>	<u>Quantitative Ion (m/z)</u>	<u>Qualitative Ion (m/z)</u>	<u>Fragmentation (V)</u>	<u>Collision Energy (V)</u>
$\Delta^9\text{-THC}$	315.2	123.0	259.0	110	40/20
$\Delta^9\text{-THC-}d_3$	318.2	196.2	NA	90	24
THCA	357.2	245.0	191.0	140	36/36
THCA- $d_3$	360.2	248.2	NA	170	36
CBD	315.2	193.0	123.0	80	20/40
CBD- $d_3$	318.2	196.0	NA	80	16
CBDA	357.3	245.1	226.9	125	28/36
CBC	315.2	193.0	123.0	80	16/32
CBC- $d_3$	318.2	236.2	NA	50	8
CBCA	357.2	191.0	179.1	140	32/24
CBDV	287.2	165.0	123.0	125	20/36
CBDVA	329.2	217.0	151.1	125	32/24
CBG	317.2	193.1	123.1	90	12/32
CBG- $d_3$	320.3	122.9	NA	50	32
CBGA	359.2	297.0	191.1	85	24/20
CBL	315.2	235.2	81.1	100	12/36
CBLA	357.2	191.0	245.0	140	32/36
CBN	311.2	223.1	208.0	145	20/32
CBNA	353.2	279.1	171.1	145	40/40
THCV	287.2	165.1	91.1	90	24/56
THCVA	329.2	163.1	217.0	125	32/28
$\Delta^8\text{-THC-}d_3$	315.2	123.0	NA	140	40/24

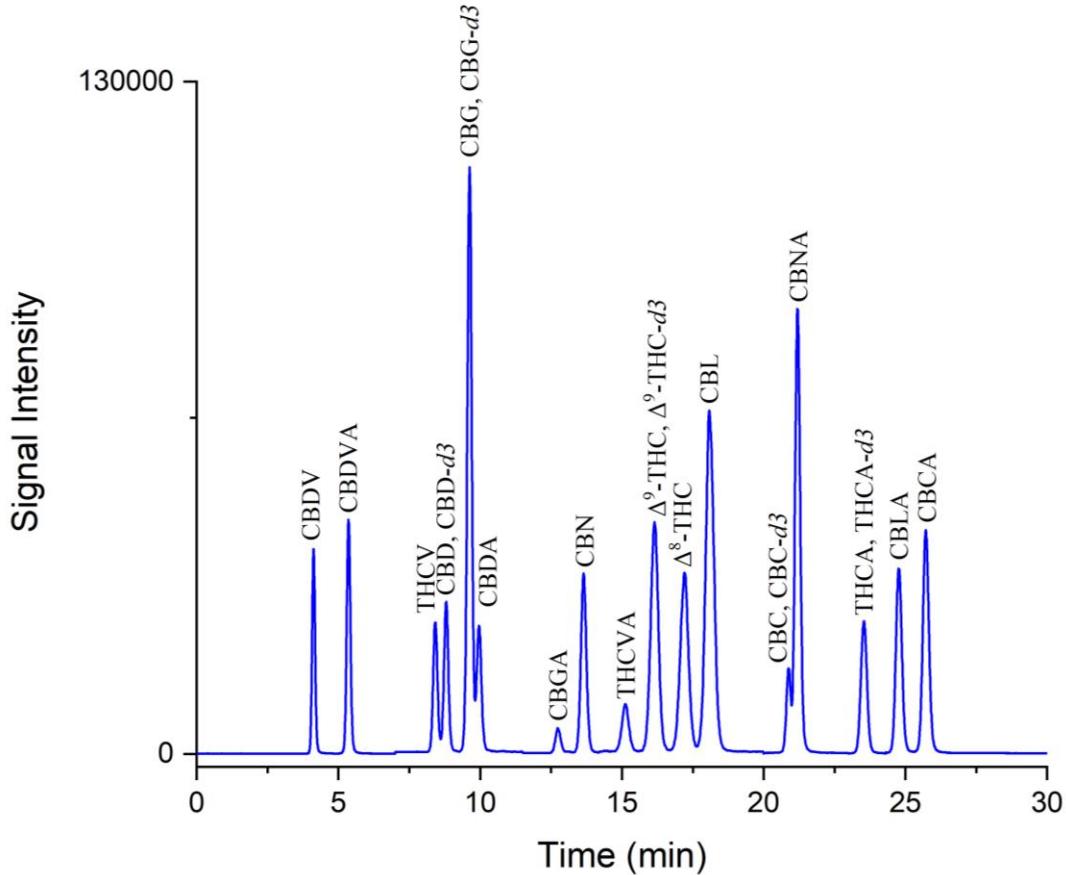


Fig. 2-2. Total ion chromatogram of a calibrant analyzed using the LC-MS/MS method.

Quantitation was performed using the IS approach for 17 cannabinoids using a linear regression model. The calibrants were prepared in MeOH from two working solutions that consisted of an acidic cannabinoid solution containing cannabidivarinic acid (CBDVA), CBDA, CBGA, tetrahydrocannabivarinic acid (THCVA), THCA, and cannabichromenic acid (CBCA) (Cerilliant), a neutral cannabinoid solution containing CBDV, THCV, CBD, CBG, CBN, Δ<sup>9</sup>-THC, Δ<sup>8</sup>-THC, and CBC (Cerilliant), and single stock solutions of cannabicyclol (CBL, Cayman), cannabicyclolic acid (CBLA, Cerilliant), and cannabinolic acid (CBNA, Cerilliant). An IS mix that contained CBD-d<sub>3</sub>, CBG-d<sub>3</sub>, Δ<sup>9</sup>-THC-d<sub>3</sub>, CBC-d<sub>3</sub>, and THCA-d<sub>3</sub>, all obtained from Cerilliant, was then added to the calibrants, sample extracts, and sample dilutions. For cannabinoids without an available isotopically labeled analog, ISs were selected from available labeled ISs with a similar retention time and signal response. Three-point calibration curves were constructed using the mass concentration ratios (x-axis) and peak area ratios (y-axis) of analyte to IS for quantitation. The precursor to quantitative ion transition was used for quantitation and the precursor to qualitative ion transition was used for confirmation. The mass concentrations (mg/L) for the sample extracts were converted to mass fractions in plant samples using Eq. (5). Density of MeOH (0.789 g/L at 22.2 °C) was used to convert the mass of MeOH to volume of MeOH [12]. For the diluted samples, the final mass fractions were calculated by multiplying the mass fractions of the diluted sample by the dilution factor, calculated using Eq. (6). The total CBD and total Δ<sup>9</sup>-THC mass fractions were calculated using Eq. (7) and Eq. (8), respectively.

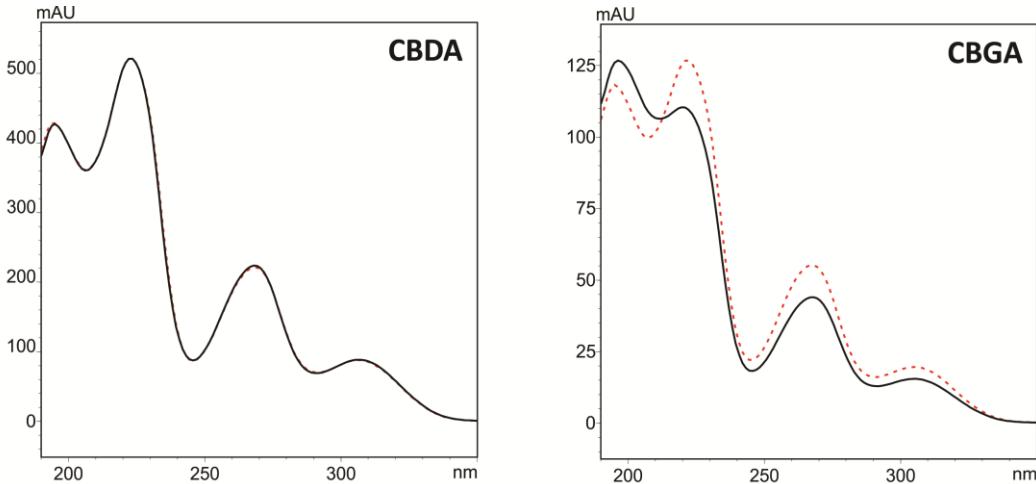
### 2.2.3. Quality Metrics/ID and QM

Quality metrics for the LC-PDA and LC-MS/MS methods (**Table 2-5**) were used to improve the confidence in the cannabinoid measurements for each sample. The quality metrics were applied after the peak was detected at the given retention time within the parameters set in each method. For the LC-PDA method, the normalized absorbance spectrum of each analyte was compared to a reference absorbance spectrum collected from a calibrant chromatogram. In cases where the two spectra did not overlap completely, the potential for bias was considered (**Fig. 2-3**). In addition to absorbance spectral comparison, the peak purity was also assessed to determine if the composition of the eluted peak was homogeneous. While spectral homogeneity across a peak is not strictly indicative of the absence of any impurities, when used in tandem with absorbance spectral comparison between the peak in a study sample and a reference analyte, confidence in value accuracy is improved. To improve confidence in the LC-MS/MS analysis, only analytes with a deuterated analog that were in control were considered for target value assignment. Statistical comparison between the NIST value and the UK-PT COA value [11] was performed using the guidance provided by the European Reference Materials [13]. Additionally, if the method control value for an analyte was outside the concentration range for the analyte in the study sample, that analyte was not considered for target value assignment (**Table 2-6**). When the value of a cannabinoid in a study sample was not able to be verified using the outlined quality metrics, no target value was assigned. After applying the quality metrics to the results from the LC-PDA method, CBDA, CBG, CBD, CBN,  $\Delta^9$ -THC, and THCA were used for target value assignment, when present. CBD,  $\Delta^9$ -THC, CBC, and THCA were quantified by LC-MS/MS when present and within the mass fraction range of the HMNOV20-1 reference material.

**Table 2-5. Quality metrics used for LC-PDA and LC-MS/MS measurements to ensure accuracy.**

<b>Quality Metric</b>	<b>Description</b>
<b>LC-PDA</b>	
<b>Absorbance spectrum</b>	Analyte spectrum in study sample corresponds to the spectrum of the analyte in the calibrant after background compensation
<b>Purity assessment</b>	Analyte chromatogram is > 0.90 pure when using the Shimadzu purity assessment tool.
<b>LC-MS/MS</b>	
<b>Internal Standard</b>	A deuterated analog of the analyte was used.
<b>Control Material</b>	A UK-PT program material was used as a method control. When appropriate <sup>a</sup> , analytes in the study samples were assigned a value if the measured value of the corresponding analyte in the UK-PT sample was consistent with the assigned value.

<sup>a</sup> If the analyte mass fraction in the UK-PT control sample was outside of the mass fraction of that analyte in the study sample, that analyte was not considered for target value assignment.



**Fig. 2-3. Example UV spectral comparison between reference and study sample spectra.**

The left panel displays the CBDA reference spectrum (red dotted line) and the study sample spectrum (black solid line) for Plant Sample 2; the right panel displays the CBGA spectra. The CBDA spectra are well correlated, which is indicative of peak purity. In the case of CBGA, the two spectra are not well correlated, indicating the presence of a coeluting analyte.

**Table 2-6. Cannabinoid mass fractions in HM20NOV-1 control material from UK-PT program determined using LC-MS/MS.**

Cannabinoid	UK-PT Assigned Value (%)	NIST LC-MS/MS (%)	Statistically Different
	Mean ± SD	Mean ± SD	
Δ <sup>9</sup> -THC	0.1476 ± 0.0031	0.1404 ± 0.0037	No
THCA	0.0476 ± 0.0018	0.0461 ± 0.0007	No
CBD	2.678 ± 0.040	2.60 ± 0.15	No
CBDA	3.632 ± 0.062	3.42 ± 0.15	No
CBC	0.2224 ± 0.0035	0.2150 ± 0.0089	No
CBCA	NA <sup>a</sup>	0.1726 ± 0.0067	
CBDV	0.0209 ± 0.0034	0.0160 ± 0.0006	No
CBDVA	0.0387 ± 0.0045	0.0284 ± 0.0016	Yes
CBG	0.0743 ± 0.0060	0.0615 ± 0.0008	Yes
CBGA	0.0951 ± 0.0056	0.0732 ± 0.0005	Yes
CBL	NA <sup>a</sup>	0.0053 ± 0.0003	
CBLA	NA <sup>a</sup>	0.0086 ± 0.0005	
CBN	0.0193 ± 0.0012	0.0139 ± 0.0005	Yes
CBNA	NA <sup>a</sup>	0.0044 ± 0.0002	
THCV	NA <sup>a</sup>	DNQ <sup>b</sup>	
THCVA	NA <sup>a</sup>	ND <sup>c</sup>	
Δ <sup>8</sup> -THC	NA <sup>a</sup>	ND <sup>c</sup>	

<sup>a</sup> NA = Not Available

<sup>b</sup> DNQ = detected but not quantified

<sup>c</sup> ND = Not Detected

## 2.3. Characterization of Cannabinoids in Study Samples

### 2.3.1. LC-PDA Characterization

**Table 2-7** provides summary information for the cannabinoids detected in Plant Samples 2 through Plant Sample 6 using LC-PDA. The information includes: the mean and standard deviation of the reported mass fractions (MF) expressed as a percentage, the relative standard deviation (RSD expressed as a percentage,  $100 \times \text{SD}/\text{Mean}$ ), the dilution factor used, the number of measurements ( $n$ ), and the assessed purity of the peak at its maximum (Purity).

Cannabinoids with minimal spectral differences between the study sample spectrum and the reference spectrum are included in the LC-PDA tables; however, if the peak purity value indicated the presence of an impurity, or coeluting analyte, the cannabinoid mass fraction was not included in the target value assignment. The RSDs of the LC-PDA measurements made between packets for the cannabinoids in Plant Sample 2 to Plant Sample 5 were below 6.03 %, indicating the homogeneity of these samples was fit-for-purpose as study samples for CannaQAP Exercise 2. Plant Sample 6 had marginally higher RSDs (5.13 % to 9.49 %) than the other materials provided to participants, which may be related to the composition of the sample material. Plant Sample 6 was composed of only small stems, while the other study samples were prepared with buds, which may have resulted in less consistent cannabinoid extraction. Despite the higher RSDs, Plant Sample 6 was used as a study sample in CannaQAP Exercise 2 as the between-packet variability was less than the 10 % parameter established by NIST.

**Table 2-7. Summary information for cannabinoids determined in Exercise 2 study samples using LC-PDA.**

Cannabinoid	Mean MF (%) $\pm$ SD (%)	RSD (%)	Dilution	n	Purity <sup>a</sup>
<b>Sample 2</b>					
$\Delta^9$ -THC	0.1267 $\pm$ 0.0075	5.96	No Dilution	12	1.00
THCA	0.424 $\pm$ 0.026	6.03	10-Fold	12	1.00
Total $\Delta^9$ -THC	0.498 $\pm$ 0.015	3.10	NA <sup>c</sup>	12	
CBD	1.051 $\pm$ 0.025	2.40	10-Fold	12	1.00
CBDA	15.13 $\pm$ 0.17	1.15	100-Fold	12	1.00
Total CBD	14.32 $\pm$ 0.14	1.00	NA <sup>c</sup>	12	
CBG	0.0546 $\pm$ 0.0022	4.01	No Dilution	12	0.08
CBN	ND <sup>b</sup>				
<b>Sample 3</b>					
$\Delta^9$ -THC	0.2920 $\pm$ 0.0068	2.32	10-Fold	6	0.99
THCA	1.289 $\pm$ 0.024	1.87	10-Fold	6	1.00
Total $\Delta^9$ -THC	1.422 $\pm$ 0.028	1.94	NA <sup>c</sup>	6	
CBD	1.134 $\pm$ 0.018	1.63	10-Fold	6	1.00
CBDA	13.82 $\pm$ 0.16	1.12	100-Fold	6	100
Total CBD	13.25 $\pm$ 0.14	1.09	NA <sup>c</sup>	6	
CBG	0.0661 $\pm$ 0.0035	5.23	No Dilution	6	0.48
CBN	0.0299 $\pm$ 0.0010	3.22	No Dilution	6	1.00
<b>Sample 4</b>					
$\Delta^9$ -THC	0.0675 $\pm$ 0.0016	2.31	No Dilution	12	0.99
THCA	0.2403 $\pm$ 0.0048	2.00	10-Fold	12	0.99
Total $\Delta^9$ -THC	0.2788 $\pm$ 0.0058	2.06	NA <sup>c</sup>	12	
CBD	0.587 $\pm$ 0.011	1.83	10-Fold	12	0.99
CBDA	8.11 $\pm$ 0.21	2.59	100-Fold	12	0.99
Total CBD	7.72 $\pm$ 0.18	2.38	NA <sup>c</sup>	12	
CBG	0.0299 $\pm$ 0.0009	2.86	No Dilution	12	0.41
CBN	ND <sup>b</sup>				
<b>Sample 5</b>					
$\Delta^9$ -THC	0.442 $\pm$ 0.016	3.65	10-Fold	6	0.99
THCA	1.996 $\pm$ 0.077	3.84	10-Fold	6	1.00
Total $\Delta^9$ -THC	2.193 $\pm$ 0.083	3.78	NA <sup>c</sup>	6	
CBD	1.392 $\pm$ 0.048	3.46	10-Fold	6	1.00
CBDA	12.99 $\pm$ 0.11	0.84	100-Fold	6	1.00
Total CBD	12.79 $\pm$ 0.10	0.80	NA <sup>c</sup>	6	
CBG	0.0856 $\pm$ 0.0005	0.57	No Dilution	6	0.93
CBN	0.0647 $\pm$ 0.0010	1.53	No Dilution	6	1.00
<b>Sample 6</b>					
$\Delta^9$ -THC	0.0280 $\pm$ 0.0026	9.35	No Dilution	6	1.00
THCA	0.136 $\pm$ 0.011	7.92	No Dilution	6	1.00
Total $\Delta^9$ -THC	0.147 $\pm$ 0.012	8.18	NA <sup>c</sup>	6	
CBD	0.241 $\pm$ 0.023	9.49	No Dilution	6	1.00
CBDA	4.21 $\pm$ 0.23	5.47	100-Fold	6	0.99
Total CBD	3.93 $\pm$ 0.20	5.13	NA <sup>c</sup>	6	
CBG	ND <sup>b</sup>				
CBN	ND <sup>b</sup>				

<sup>a</sup> Purity assessments are from a single sample analysis

<sup>b</sup> ND = not detected in sample extract

<sup>c</sup> NA = not applicable, analyte was calculated

### 2.3.2. LC-MS/MS Characterization

**Table 2-8** provides summary information for the cannabinoids detected in Plant Samples 2 through Plant Sample 6 using LC-MS/MS. The information includes: the mean and standard deviation of the reported mass fractions (MF) expressed as a percentage, the relative standard deviation (RSD expressed as a percentage,  $100 \times SD / Mean$ ), the dilution factor used, the number of measurements ( $n$ ), and whether the analyte concentration was similar to that of the control.

Cannabinoids with a deuterated analog as the IS are included in the LC-MS/MS tables; however, if the analyte was not in agreement with the UK-PT material or the mass fraction of the analyte in the sample was outside of the mass fraction range in the UK-PT material the cannabinoid mass fraction was not included in the target value assignment. When possible, the analytes quantitated using LC-MS/MS were combined with the values measured using LC-PDA to assign the target values in the study samples.

**Table 2-8. Summary information for Exercise 2 study samples using LC-MS/MS.**

<u>Cannabinoid</u>	<u>Mean MF (%) <math>\pm</math> SD (%)</u>	<u>RSD (%)</u>	<u>Dilution</u>	<u>n</u>	<u>In Control<sup>a</sup></u>
<b>Sample 2</b>					
$\Delta^9$ -THC	0.1467 $\pm$ 0.0043	2.95	10-Fold	3	Yes
THCA	0.3577 $\pm$ 0.0036	1.01	10-Fold	3	No
Total $\Delta^9$ -THC	0.4604 $\pm$ 0.0036	0.78	NA <sup>b</sup>	3	No
CBD	1.311 $\pm$ 0.080	6.12	100-Fold	2	Yes
CBC	0.1181 $\pm$ 0.0036	3.07	10-Fold	3	Yes
<b>Sample 3</b>					
$\Delta^9$ -THC	0.2954 $\pm$ 0.0056	1.90	10-Fold	3	No
THCA	1.040 $\pm$ 0.025	2.37	10-Fold	3	No
Total $\Delta^9$ -THC	0.2954 $\pm$ 0.0056	1.88	NA <sup>b</sup>	3	No
CBD	1.289 $\pm$ 0.030	2.32	100-Fold	3	Yes
CBC	0.1207 $\pm$ 0.0017	1.37	10-Fold	3	Yes
<b>Sample 4</b>					
$\Delta^9$ -THC	0.0642 $\pm$ 0.0026	4.04	10-Fold	3	Yes
THCA	0.195 $\pm$ 0.013	6.50	10-Fold	3	No
Total $\Delta^9$ -THC	0.235 $\pm$ 0.013	5.46	NA <sup>b</sup>	3	No
CBD	0.595 $\pm$ 0.025	4.16	100-Fold	3	Yes
CBC	0.0522 $\pm$ 0.0021	4.02	10-Fold	3	Yes
CBG	0.0229 $\pm$ 0.0013	5.85	10-Fold	3	No
<b>Sample 5</b>					
$\Delta^9$ -THC	0.380 $\pm$ 0.060	15.7	10-Fold	3	No
THCA	1.717 $\pm$ 0.065	3.77	10-Fold	3	No
Total $\Delta^9$ -THC	1.885 $\pm$ 0.099	5.26	NA <sup>b</sup>	3	No
CBD	1.41 $\pm$ 0.14	9.86	100-Fold	3	Yes
CBC	0.121 $\pm$ 0.023	18.9	10-Fold	3	Yes
<b>Sample 6</b>					
$\Delta^9$ -THC	0.0303 $\pm$ 0.0014	4.51	No Dilution	3	Yes
THCA	0.1198 $\pm$ 0.0043	3.6	No Dilution	3	Yes
Total $\Delta^9$ -THC	0.1353 $\pm$ 0.0049	3.66	NA <sup>b</sup>	3	No
CBD	0.2846 $\pm$ 0.010	3.54	100-Fold	3	No
CBC	0.0223 $\pm$ 0.0014	6.18	No Dilution	3	Yes

<sup>a</sup> “In Control” here indicates that the analyte concentration of the study sample was within that of the control

<sup>b</sup> NA = not applicable, analyte was calculated

## 2.4. Assignment of Target Values for CannaQAP Exercise 2 Study Samples

Target values for cannabinoids in each of the study samples were assigned in all cases where the quality metrics were confirmed using one or both of the analytical methods. In the case where one analytical method was used to determine the target value, the associated uncertainty was the SD of those measurements. When both methods were used to assign a target value, the target value and uncertainty were determined using hierarchical Bayes (Gaussian) model in the NIST Consensus Builder [14]. The default Bayesian fit parameters were used for model predictions. The mass fraction, SDs, and degrees of freedom used as the Consensus Builder inputs as well as the mass fraction and standard uncertainty outputted by the Consensus Builder are included in **Table 2-9**. The assigned target mass fractions and uncertainties for CannaQAP Exercise 2 Plant Sample 1 through Plant Sample 6 are summarized in **Table 2-9** and **Table 2-10**. Mass fractions reported here are traceable to the mass concentration values of the respective components of the solution mixtures obtained from the manufacturer used for calibration and to the mass and volume values of calibrants and test samples provided within this report. The calibrants used were not traceable to the SI.

**Table 2-9. NIST Consensus Builder model inputs and target value outputs using LC-PDA and LC-MS/MS.**

Cannabinoid	LC-PDA Values (%)		LC-MS/MS Values (%)		Degrees of Freedom (LC-PDA, LC-MS/MS)	Consensus Builder Output (%)	
	MF ± SD	MF ± SD	MF ± SD	MF ± SD		MF ± SD	MF ± SD
<b>Sample 2</b>							
Δ <sup>9</sup> -THC	0.1267 ± 0.0075		0.1467 ± 0.0043		11, 2	0.138 ± 0.023	
CBD	1.051 ± 0.025		1.311 ± 0.080		11, 1	1.16 ± 0.34	
<b>Sample 3</b>							
CBD	1.134 ± 0.018		1.289 ± 0.030		5, 2	1.20 ± 0.19	
<b>Sample 4</b>							
Δ <sup>9</sup> -THC	0.0675 ± 0.0016		0.0642 ± 0.0026		11, 2	0.0663 ± 0.0040	
CBD	0.587 ± 0.011		0.595 ± 0.025		11, 2	0.589 ± 0.019	
<b>Sample 5</b>							
CBD	1.392 ± 0.048		1.41 ± 0.14		5, 2	1.400 ± 0.063	
<b>Sample 6</b>							
Δ <sup>9</sup> -THC	0.0280 ± 0.0026		0.0303 ± 0.0014		5, 2	0.0295 ± 0.0029	
THCA	0.136 ± 0.011		0.1198 ± 0.0043		5, 2	0.126 ± 0.022	
Total THC	0.147 ± 0.012		0.1353 ± 0.0049		5, 2	0.139 ± 0.013	

**Table 2-10. Target values and expanded uncertainties for CannaQAP Exercise 2 study samples.**

<u>Cannabinoid</u>	<u>NRC HEMP-1</u>		
	(Plant Sample 1)	Plant Sample 2	Plant Sample 3
$\Delta^9$ -THC	0.0318 $\pm$ 0.0086	0.138 $\pm$ 0.023	0.292 $\pm$ 0.014
THCA	0.0979 $\pm$ 0.0084	0.424 $\pm$ 0.052	1.289 $\pm$ 0.048
Total $\Delta^9$ -THC	0.118 $\pm$ 0.014	0.498 $\pm$ 0.030	1.422 $\pm$ 0.056
CBD	0.541 $\pm$ 0.070	1.16 $\pm$ 0.34	1.20 $\pm$ 0.19
CBDA	1.46 $\pm$ 0.080	15.13 $\pm$ 0.34	13.82 $\pm$ 0.32
Total CBD	1.82 $\pm$ 0.12	14.32 $\pm$ 0.28	13.25 $\pm$ 0.28
CBC	0.0325 $\pm$ 0.0084	0.1181 $\pm$ 0.0072	0.1207 $\pm$ 0.0034
CBCA	0.045 $\pm$ 0.011	NA <sup>a</sup>	NA <sup>a</sup>
CBDV	0.0188 $\pm$ 0.0032	NA <sup>a</sup>	NA <sup>a</sup>
CBDVA	0.0719 $\pm$ 0.0054	NA <sup>a</sup>	NA <sup>a</sup>
CBG	0.00478 $\pm$ 0.00094	NA <sup>a</sup>	NA <sup>a</sup>
CBGA	0.0117 $\pm$ 0.0012	NA <sup>a</sup>	NA <sup>a</sup>
CBL	0.0074 $\pm$ 0.0014	NA <sup>a</sup>	NA <sup>a</sup>
CBLA	0.0187 $\pm$ 0.0018	NA <sup>a</sup>	NA <sup>a</sup>
CBN	0.0490 $\pm$ 0.0070	NA <sup>a</sup>	0.0299 $\pm$ 0.0020
CBNA	0.0350 $\pm$ 0.0036	NA <sup>a</sup>	NA <sup>a</sup>
THCV	0.00143 $\pm$ 0.00020	NA <sup>a</sup>	NA <sup>a</sup>
THCVA	0.00728 $\pm$ 0.00064	NA <sup>a</sup>	NA <sup>a</sup>
$\Delta^8$ -THC	NA <sup>a</sup>	NA <sup>a</sup>	NA <sup>a</sup>
<u>Cannabinoid</u>	<u>Plant Sample 4</u>		
	Plant Sample 4	Plant Sample 5	Plant Sample 6
$\Delta^9$ -THC	0.0663 $\pm$ 0.0040	0.442 $\pm$ 0.032	0.0295 $\pm$ 0.0029
THCA	0.2403 $\pm$ 0.0096	2.00 $\pm$ 0.15	0.126 $\pm$ 0.022
Total $\Delta^9$ -THC	0.279 $\pm$ 0.012	2.19 $\pm$ 0.17	0.139 $\pm$ 0.013
CBD	0.589 $\pm$ 0.019	1.400 $\pm$ 0.063	0.241 $\pm$ 0.046
CBDA	8.11 $\pm$ 0.42	12.99 $\pm$ 0.22	4.21 $\pm$ 0.46
Total CBD	7.72 $\pm$ 0.36	12.79 $\pm$ 0.20	3.93 $\pm$ 0.40
CBC	0.0522 $\pm$ 0.0042	0.121 $\pm$ 0.046	0.0223 $\pm$ 0.0028
CBCA	NA <sup>a</sup>	NA <sup>a</sup>	NA <sup>a</sup>
CBDV	NA <sup>a</sup>	NA <sup>a</sup>	NA <sup>a</sup>
CBDVA	NA <sup>a</sup>	NA <sup>a</sup>	NA <sup>a</sup>
CBG	NA <sup>a</sup>	0.0856 $\pm$ 0.001	NA <sup>a</sup>
CBGA	NA <sup>a</sup>	NA <sup>a</sup>	NA <sup>a</sup>
CBL	NA <sup>a</sup>	NA <sup>a</sup>	NA <sup>a</sup>
CBLA	NA <sup>a</sup>	NA <sup>a</sup>	NA <sup>a</sup>
CBN	NA <sup>a</sup>	0.0647 $\pm$ 0.002	NA <sup>a</sup>
CBNA	NA <sup>a</sup>	NA <sup>a</sup>	NA <sup>a</sup>
THCV	NA <sup>a</sup>	NA <sup>a</sup>	NA <sup>a</sup>
THCVA	NA <sup>a</sup>	NA <sup>a</sup>	NA <sup>a</sup>
$\Delta^8$ -THC	NA <sup>a</sup>	NA <sup>a</sup>	NA <sup>a</sup>

<sup>a</sup> NA = analyte was not assigned a target value

### 3. $\Delta^9$ -THC, THCA, And Total $\Delta^9$ -THC

#### 3.1. Study Overview

The medicinal and recreational use of cannabis and cannabis-derived finished products continues to increase across the United States. While recreational use of marijuana is legal in a number of states, there is still a federal requirement to differentiate between hemp and marijuana. Depending on the jurisdiction, hemp is defined as having either a  $\Delta^9$ -THC or a total  $\Delta^9$ -THC mass fraction by dry weight of less than 0.3 %, which requires the accurate quantitation of  $\Delta^9$ -THC, THCA, and/or total  $\Delta^9$ -THC. The primary psychoactive cannabinoid in cannabis products is  $\Delta^9$ -THC; however, the precursor to  $\Delta^9$ -THC, THCA, is the cannabinoid formed by the plant. THCA is synthesized in the glandular trichomes of the cannabis plant and forms  $\Delta^9$ -THC via decarboxylation due to prolonged storage and/or exposure to heat or light [15]. In this study, participants were asked to use in-house analytical methods to determine the as-received mass fractions (%) of  $\Delta^9$ -THC, THCA, and total  $\Delta^9$ -THC in six cannabis plant samples. Through participation in this study, laboratories can better understand the performance of their in-house methods relative to those being used by others in the community and gage the accuracy of their measurements via comparison to target mass fractions assigned by NIST.

#### 3.2. Sample Information

The target values and uncertainties for  $\Delta^9$ -THC, THCA, and total  $\Delta^9$ -THC in the hemp and marijuana plant samples are provided in **Table 3-1** on an as-received basis. The target values and uncertainties for NRC HEMP-1 were determined by NRC Canada and taken from the HEMP-1 COA [6]. The target values and uncertainties in the remaining five cannabis plant samples were determined at NIST using LC-PDA and/or LC-MS/MS measurements as described in Section 2.2.2. Participants receiving the three hemp samples (NRC HEMP-1, Plant Sample 4, and Plant Sample 6) were informed prior to the study that the mass fraction levels for  $\Delta^9$ -THC, THCA, and total  $\Delta^9$ -THC were  $\leq 0.3$  %. Participants receiving the three marijuana samples also knew those samples had a total  $\Delta^9$ -THC content  $> 0.3$  %. Laboratories were required to provide documentation approving them to receive marijuana samples. No other information regarding the mass fractions of the cannabinoids in the samples was provided to participants.

**Table 3-1. Example individualized data summary table for  $\Delta^9$ -THC, THCA, and total  $\Delta^9$ -THC.**

Laboratory-specific results and Z-scores were provided to each participant separately from this report to protect laboratory identities.

(*Lab Name*)

**Exercise 2 – Cannabinoids in Cannabis Plant Samples**

Lab Code:	(Code)	1. Your Results				2. Community Results			3. Target			
		Sample	Units	$x_i$	$s_i$	$Z^i_{\text{comm}}$	$Z_{\text{NIST}}$	N	$x^*$	$s^*$	$x_{\text{NIST}}$	$u$
$\Delta^9$ -THC	NRC HEMP-1	%						108	0.037	0.014	0.0318	0.0086
$\Delta^9$ -THC	Plant Sample 2	%						26	0.151	0.031	0.138	0.023
$\Delta^9$ -THC	Plant Sample 3	%						26	0.293	0.046	0.292	0.014
$\Delta^9$ -THC	Plant Sample 4	%						132	0.068	0.014	0.0663	0.0040
$\Delta^9$ -THC	Plant Sample 5	%						26	0.420	0.075	0.442	0.032
$\Delta^9$ -THC	Plant Sample 6	%						110	0.0310	0.0088	0.0295	0.0029
THCA	NRC HEMP-1	%		<i>Individual laboratory results will appear in this section; laboratory-specific results were provided to each participant separately from this report.</i>				138	0.091	0.020	0.0979	0.0084
THCA	Plant Sample 2	%						24	0.381	0.049	0.424	0.052
THCA	Plant Sample 3	%						23	1.09	0.11	1.289	0.048
THCA	Plant Sample 4	%						142	0.215	0.030	0.2403	0.0096
THCA	Plant Sample 5	%						21	1.80	0.17	2.00	0.15
THCA	Plant Sample 6	%						140	0.137	0.021	0.126	0.022
Total $\Delta^9$ -THC	NRC HEMP-1	%						150	0.111	0.034	0.118	0.014
Total $\Delta^9$ -THC	Plant Sample 2	%						33	0.481	0.062	0.498	0.030
Total $\Delta^9$ -THC	Plant Sample 3	%						30	1.24	0.13	1.422	0.056
Total $\Delta^9$ -THC	Plant Sample 4	%						152	0.249	0.042	0.279	0.012
Total $\Delta^9$ -THC	Plant Sample 5	%						29	1.94	0.22	2.19	0.17
Total $\Delta^9$ -THC	Plant Sample 6	%						151	0.145	0.030	0.139	0.013

$x_i$  Mean of reported values  
 $s_i$  Standard deviation of reported values  
 $Z^i_{\text{comm}}$  Z-score with respect to community consensus  
 $Z_{\text{NIST}}$  Z-score with respect to NIST value

$N$  Number of quantitative values reported  
 $x^*$  Robust mean of reported values  
 $s^*$  Robust standard deviation

$x_{\text{NIST}}$  NIST-assessed value

$u$  standard uncertainty about the NIST-assessed value and assigned values by NRC Canada for HEMP-1

### 3.3. Reporting Statistics

The enrollment and reporting statistics for  $\Delta^9$ -THC, THCA, and total  $\Delta^9$ -THC are described in **Table 3-2** for all plant samples. A total of 226 laboratories registered to participate in Exercise 2 of CannaQAP with approximately 84 %, 81 %, and 90 % of participants signing up to report values for  $\Delta^9$ -THC, THCA, and total  $\Delta^9$ -THC in the three hemp plant samples (NRC HEMP-1, Plant Sample 4, and Plant Sample 6), respectively. Approximately 22 % of the registered laboratories provided NIST with the appropriate DEA paperwork and signed up to measure  $\Delta^9$ -THC, THCA, and total  $\Delta^9$ -THC in the three marijuana samples (Plant Sample 2, Plant Sample 3, and Plant Sample 5). The percentage of laboratories that requested samples and returned results for  $\Delta^9$ -THC, THCA, and

total  $\Delta^9$ -THC was between 75 % and 81 % for the hemp samples and between 48 % and 80 % for the marijuana samples.

**Table 3-2. Enrollment and reporting statistics for  $\Delta^9$ -THC, THCA, and total  $\Delta^9$ -THC.**

Cannabinoid	Number of Participants	Percent Reporting Results (%) for Hemp Samples		
		NRC HEMP-1	Plant Sample 4	Plant Sample 6
$\Delta^9$ -THC	190	75	76	76
THCA	182	79	79	79
Total $\Delta^9$ -THC	204	80	81	80

Cannabinoid	Number of Participants	Percent Reporting Results (%) for Marijuana Samples		
		Plant Sample 2	Plant Sample 3	Plant Sample 5
$\Delta^9$ -THC	50	56	56	56
THCA	50	48	48	48
Total $\Delta^9$ -THC	50	80	80	80

Participants were asked to either submit quantitative results or qualitative results based on LOQs or threshold values for  $\Delta^9$ -THC (**Table 3-3**), THCA, (**Table 3-4**) and total  $\Delta^9$ -THC (**Table 3-5**). Laboratories reporting values below LOQ were considered successful in the study if the target value was also below the laboratory LOQ.

#### $\Delta^9$ -THC

Approximately 75 % of laboratories reported quantitative results for  $\Delta^9$ -THC in NRC HEMP-1 and Plant Sample 6 and 91 % of laboratories reported quantitative results for  $\Delta^9$ -THC in Plant Sample 4. A larger percentage of laboratories that measured  $\Delta^9$ -THC in NRC HEMP-1 and Plant Sample 6 reported values as below LOQ than the other samples, with LOQs ranging from 0.003 % to 1.5 %. For NRC HEMP-1, Plant Sample 4, and Plant Sample 6, 64 %, 56 %, and 69 % of laboratories reported LOQs above the target range, respectively, which was considered a successful analysis. Approximately 93 % of laboratories measuring  $\Delta^9$ -THC in the marijuana samples reported quantitative results. The laboratories that reported qualitative results for the marijuana samples reported values as both greater than a set threshold value and as LOQs, all of which were above the target range. Laboratories that reported greater than a set threshold value that was above the target range were not considered to have a successful analysis.

**Table 3-3. Number of laboratories reporting use of qualitative and quantitative analysis for  $\Delta^9$ -THC.**

Samples	Total Number of Laboratories Reporting Results	Number of Laboratories	Number of Laboratories
		Reporting Qualitative Results	Reporting Quantitative Results
NRC HEMP-1	143	35	108
Plant Sample 4	145	13	132
Plant Sample 6	144	34	110
Plant Sample 2	28	2	26
Plant Sample 3	28	2	26
Plant Sample 5	28	2	26

## THCA

With the exception of Plant Sample 5 (88 %), at least 96 % of laboratories reported quantitative results for THCA in the cannabis plant samples. Fewer laboratories reported qualitative results for THCA than  $\Delta^9$ -THC. Between 1 % and 3 % of laboratories reporting THCA results for the hemp samples reported qualitative results as LOQs. Approximately, 50 % to 100 % of laboratories reported LOQs above the target range for the hemp samples, which was considered a successful analysis. For participants reporting qualitative results for the marijuana samples, 100 % reported greater than or threshold values, which was not considered a successful analysis.

**Table 3-4. Number of laboratories reporting use of qualitative and quantitative analysis for THCA.**

<u>Samples</u>	<u>Total Number of Laboratory Reporting Results</u>	<u>Number of Laboratories Reporting Qualitative Results</u>	<u>Number of Laboratories Reporting Quantitative Results</u>
NRC HEMP-1	143	5	138
Plant Sample 4	144	2	142
Plant Sample 6	144	3	140
Plant Sample 2	24	0	24
Plant Sample 3	24	1	23
Plant Sample 5	24	3	21

## Total $\Delta^9$ -THC

Approximately 92 % of laboratories reported quantitative results for total  $\Delta^9$ -THC in the three hemp samples. Of laboratories reporting LOQs for the hemp samples, 85 % to 93 % reported LOQs higher than the target range, which was considered a successful analysis. Fewer laboratories reported quantitative results (73 % to 83 %) for total  $\Delta^9$ -THC in the marijuana samples than in the hemp samples. For Plant Sample 2, all laboratories that reported qualitative results reported LOQs above the target range, which was considered a successful analysis. For Plant Sample 3 and Plant Sample 5, with the exception of one entry that appeared to be an error, 100 % of participants that reported qualitative results reported threshold values with approximately 90 % of the threshold values below the target range, which was considered a successful analysis.

**Table 3-5. Number of laboratories reporting use of qualitative and quantitative analysis for total  $\Delta^9$ -THC.**

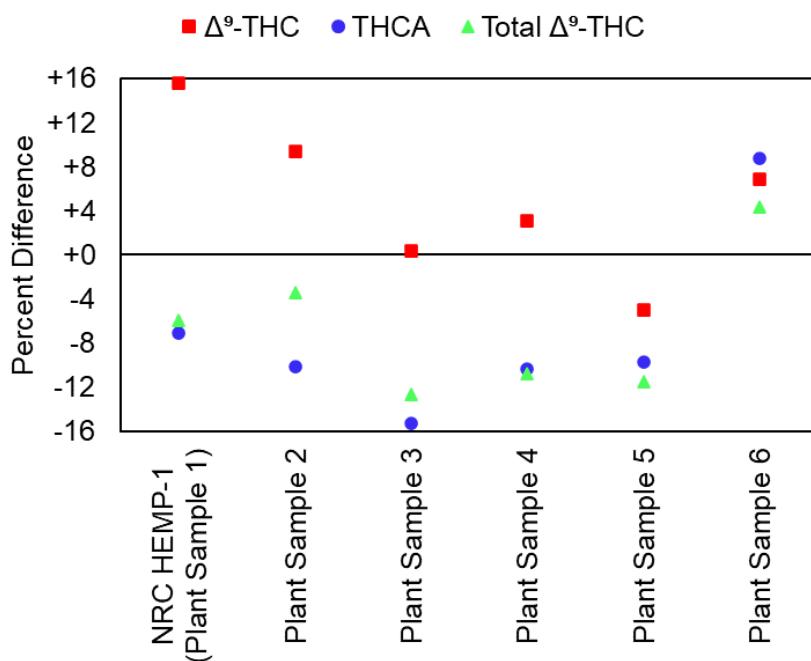
<u>Samples</u>	<u>Total Number of Laboratory Reporting Results</u>	<u>Number of Laboratories Reporting Qualitative Results</u>	<u>Number of Laboratories Reporting Quantitative Results</u>
NRC HEMP-1	164	14	150
Plant Sample 4	165	13	152
Plant Sample 6	164	13	151
Plant Sample 2	40	7	33
Plant Sample 3	40	10	30
Plant Sample 5	40	11	29

Of the laboratories that reported LOQs for  $\Delta^9$ -THC, THCA, and total  $\Delta^9$ -THC, 40 %, 75 % to 80 %, and 100% of laboratories reported LOQs higher than the published standard method performance requirements of  $\leq 0.05$  %, respectively [16]. A review of the qualitative results

indicates a combination of laboratories that have determined a method LOQ based on limits of their analytical processes and others that are predetermined based on legal reporting requirements (e.g., 1 % to 2 %). Laboratories should be clear in describing the method by which an LOQ was determined. Many appropriate methods for determining LOQs have been published, including empirical and hypothetical approaches (e.g., linear range and spiking studies) as well as those based on practical use of the method (e.g., decision point thresholds).

### 3.4. Study Results and Discussion

The participant results are summarized in the community results section of **Table 3-1** and were based on the numerical results reported by participating laboratories summarized in **Appendix B**. The percent difference between the consensus and target values for  $\Delta^9$ -THC, THCA, and total  $\Delta^9$ -THC in the cannabis plant samples is illustrated in **Fig. 3-1**. With the exception of Plant Sample 5, the consensus values for  $\Delta^9$ -THC were biased high with respect to the target values, with the consensus value for Plant Sample 3 falling within 0.34 % of the target value. Conversely, the consensus values for THCA in all samples except Plant Sample 6 were biased low with respect to the target values. For total  $\Delta^9$ -THC, the bias closely followed the THCA bias because THCA was a larger contributor to the total  $\Delta^9$ -THC values than  $\Delta^9$ -THC.



**Fig. 3-1. Percent difference between the consensus mean and target value for  $\Delta^9$ -THC, THCA, and total  $\Delta^9$ -THC.**

The 0 % line represents no difference between the consensus mean and the target value.

No obvious trends were observed relating cannabinoid mass fractions to bias, suggesting that the differences between the target and consensus values were not due to cannabinoid extraction issues. Additionally, the observed biases do not support error due to the decarboxylation of THCA into  $\Delta^9$ -THC over the duration of the study. Based on the findings of Meija et al. [17], the total  $\Delta^9$ -THC equivalent, on average, decayed at 2 % per month at room temperature, but the

participants in this study were advised to store the plant samples *below* room temperature and analyze them within a month of sample receipt. The percent bias of total Δ<sup>9</sup>-THC in the study samples should have been 2 % or less if the increase in Δ<sup>9</sup>-THC and decrease in THCA were solely due to decarboxylation. The lack of these common trends indicates that the analytical differences between the consensus and target values were more likely based on errors in calibration. Further discussion of specific analyte bias is included for Δ<sup>9</sup>-THC (Section 3.4.1), THCA (Section 3.4.2), and total Δ<sup>9</sup>-THC (Section 3.4.3) below.

**Table 3-6** details the within-laboratory (repeatability, %RSD<sub>r</sub>) and between-laboratory (reproducibility, %RSD<sub>R</sub>) variabilities for Δ<sup>9</sup>-THC, THCA, and total Δ<sup>9</sup>-THC in the six cannabis plant samples. While the majority of within-laboratory variabilities met the acceptable repeatability criteria outlined by AOAC for low THC hemp varieties (%RSD<sub>r</sub> ≤ 5 %), all but one analyte/plant sample combination were outside the AOAC reproducibility performance criteria (%RSD<sub>R</sub> ≤ 10 %) [16]. Other than use of multiple analytical methods for quantitation of these cannabinoids, one contributing factor to the high between-laboratory variabilities for Δ<sup>9</sup>-THC, THCA, and total Δ<sup>9</sup>-THC could be the inconsistency in storage conditions among laboratories. Although most laboratories indicated that samples would be stored below room temperature as described previously, 4 % of laboratories intended to store samples at room temperature. Neutral and acidic cannabinoids have been shown to degrade at room temperature [17], with Reason et al. [18] observing more than 10 % degradation in THCA mass fractions at room temperature over 60 days.

**Table 3-6. Within-laboratory and between-laboratory variabilities for the determination of Δ<sup>9</sup>-THC, THCA, and total Δ<sup>9</sup>-THC.**

Sample	Δ <sup>9</sup> -THC		THCA		Total Δ <sup>9</sup> -THC	
	%RSD <sub>r</sub>	%RSD <sub>R</sub>	%RSD <sub>r</sub>	%RSD <sub>R</sub>	%RSD <sub>r</sub>	%RSD <sub>R</sub>
NRC HEMP-1	4.3	38.2	3.8	21.6	4.5	30.9
Plant Sample 4	5.6	21.2	4.4	13.8	4.0	16.8
Plant Sample 6	4.4	28.3	3.5	15.2	4.5	20.4
Plant Sample 2	2.3	20.4	2.6	12.9	2.1	13.0
Plant Sample 3	2.9	15.5	3.1	10.4	2.3	10.3
Plant Sample 5	2.1	17.8	1.8	9.6	2.0	11.2

Most laboratories used a combination of solvent extraction for sample preparation and LC with either absorbance (ABS) or PDA for analysis (**Table 3-7**). Approximately 94 % of laboratories used solvent extraction and because the samples were plant material, the ≈ 6 % of laboratories reporting dilution also likely used solvent extraction prior to dilution. Due to the lack of data on actual solvents used for extraction as well as lack of detailed extraction procedures, no trends based on sample preparation were able to be assessed unless a candidate method was used. The results and discussion presented here will focus on comparability of different instrumental methods, chemical interferences, and calibration approaches. Additional information for sample preparation and LC instrumental methods are provided in **Appendix C** based on responses to a method questionnaire filled out by 93 participants.

**Table 3-7. Summary of sample preparation and analytical methods used by participants reporting results for  $\Delta^9$ -THC, THCA, and total  $\Delta^9$ -THC.**

<u>Preparation Method</u>	<u><math>\Delta^9</math>-THC (%)</u>	<u>THCA (%)</u>	<u>Total <math>\Delta^9</math>-THC (%)</u>
Solvent Extraction	94.0	94.2	91.8
Dilution	5.4	5.2	5.2
Other/No Response	0.6	0.6	2.9
<u>Analytical Method</u>	<u><math>\Delta^9</math>-THC (%)</u>	<u>THCA (%)</u>	<u>Total <math>\Delta^9</math>-THC (%)</u>
LC-PDA	49.2	50.5	40.8
LC-ABS	35.1	36.2	29.5
LC-MS	1.2	1.0	0.7
LC-MS/MS	8.1	8.2	6.4
GC-FID	1.7	1.8	6.5
GC-MS	2.3	0.0	13.2
Other/No Response	2.3	2.4	2.9

### 3.4.1. $\Delta^9$ -THC

#### 3.4.1.1. Within- and Between-Laboratory Precision

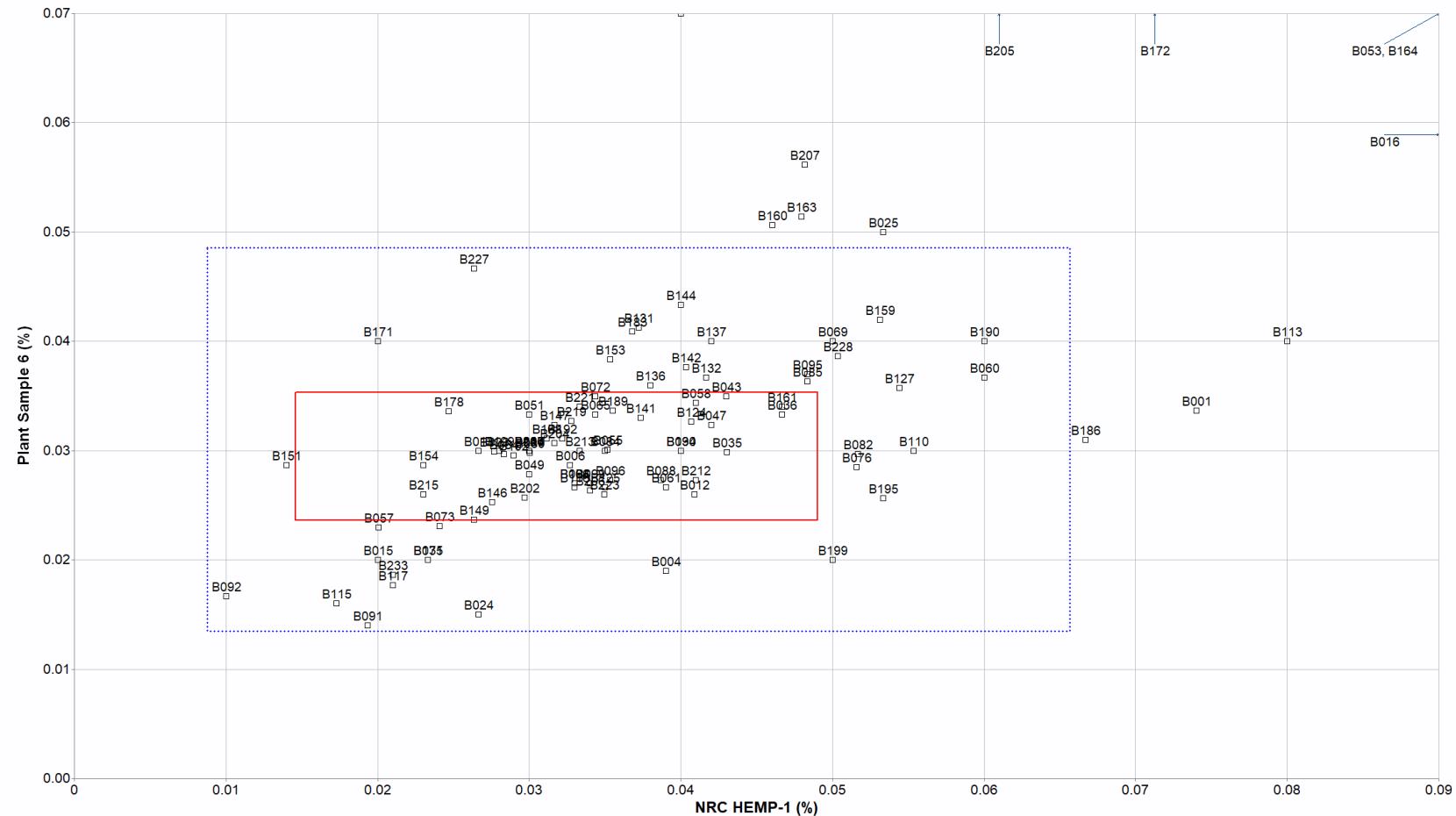
Laboratories reporting quantitative results for  $\Delta^9$ -THC in all plant samples demonstrated within-laboratory variability (repeatability, %RSD<sub>r</sub>) less than or equal to 5.6 % with higher variability observed for the hemp samples than for the marijuana samples (Table 3-6). The published method performance criterion state that samples with  $\Delta^9$ -THC levels between 0.05 % and 0.5 % (by dry weight) should be determined with %RSD<sub>r</sub> ≤ 5 % [16, 21]. NRC HEMP-1 and Plant Sample 6 both contained less than 0.05 %  $\Delta^9$ -THC, which generally indicates that a higher within-laboratory variability would be acceptable. However, a specific requirement has not been published for plant samples containing levels of  $\Delta^9$ -THC below 0.05 %. The repeatability for the hemp samples was ≤ 5 % for 58 % (NRC HEMP-1), 58 % (Plant Sample 4), and 68 % (Plant Sample 6) of laboratories measuring  $\Delta^9$ -THC. A higher percentage of laboratories reported within-laboratory variabilities at or below the published expectations for Plant Sample 2 (88 %), Plant Sample 3 (88 %), and Plant Sample 5 (100 %). As expected, as the mass fraction of  $\Delta^9$ -THC in the cannabis samples decreased, the within-laboratory variability increased. As cannabinoid mass fractions approach the method LOQs, potential for random errors increased during sample preparation and inadequate instrumental response influenced the measurement variability.

A similar trend was observed for the between-laboratory variabilities for  $\Delta^9$ -THC measurements, with the variability being higher in the hemp samples than the marijuana samples. The between-laboratory variabilities for  $\Delta^9$ -THC in all samples ranged from 15.5 % to 38.2 %, which were all above the published recommendations. However, the AOAC criteria for between-laboratory precision are meant to be applied to variabilities from multiple laboratories using a single analytical method, not variabilities from multiple laboratories using multiple analytical methods as was the case for this study. The between-laboratory variabilities for  $\Delta^9$ -THC in the marijuana plant samples and Plant Sample 4 for this exercise are comparable to the UK-PT program overall analyte relative standard deviations for  $\Delta^9$ -THC in hemp reported over the course of 15 studies (8.87 % to 21 %) [19]. The high overall between-laboratory variability is possibly due to all

participants not storing samples under the recommended conditions and the differences in sample preparation methods (**Appendix C**).

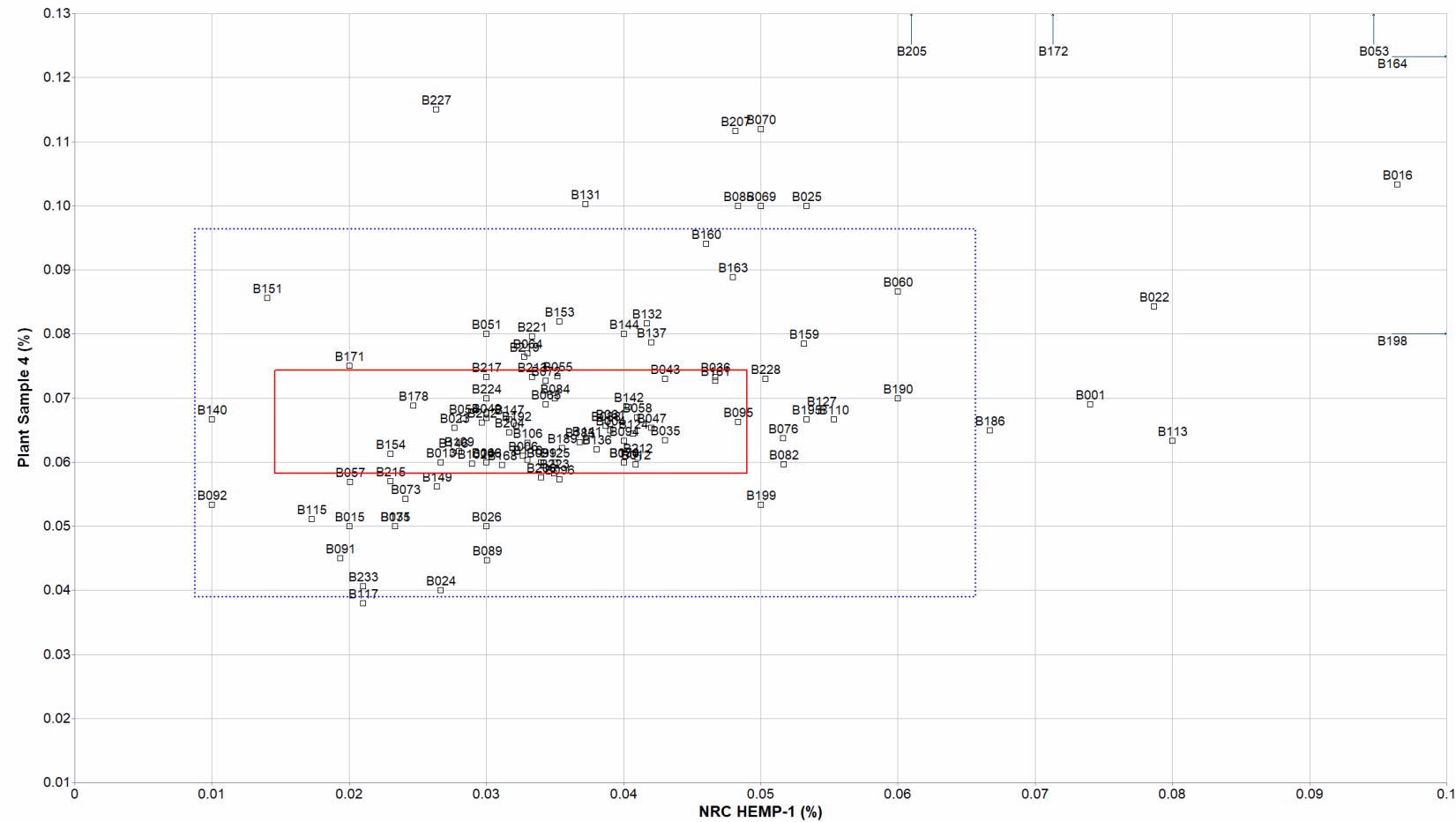
To further examine the potential causes of between-laboratory variability, laboratory performance was compared on two separate samples with similar analyte mass fractions. NRC HEMP-1 and Plant Sample 6 both contained low levels of  $\Delta^9$ -THC, at or near the LOQs for participating laboratories, and between-laboratory variabilities for measurements of both were outside the published performance criteria and the UK-PT program published range. The trend observed when comparing NRC HEMP-1 to Plant Sample 6 (**Fig. 3-2**) and Plant Sample 4 (**Fig. 3-3**) is slightly linear, which indicates that systematic between-laboratory variabilities were more pronounced than random variabilities. Possible systematic errors that would present as high between-laboratory variability include calibration bias and global methodological issues, such as poor chromatographic separation of coeluting cannabinoids. A larger percent of laboratories reported high for NRC HEMP-1 than the other two hemp samples, possibly due to the higher concentrations of coeluting compounds in NRC HEMP-1 than in the other two hemp samples. When the  $\Delta^9$ -THC data from Plant Sample 4 was compared to Plant Sample 6, the majority of laboratories that reported either high or low relative to the target and consensus ranges did so consistently between samples, which is more indicative of calibration bias. To prevent calibration bias laboratories should use calibration standards that meet ISO standards, and ensure all purity information is reviewed, independently prepared, and traceable to the SI, if possible.

It is also possible that random errors were at play for laboratories such as B186 and B001, which had  $\Delta^9$ -THC values within the target range for Plant Sample 4 and Plant Sample 6, but outside the target range for NRC HEMP-1. The reported  $\Delta^9$ -THC values by B186 and B001 were two times higher than the reference value for NRC HEMP-1, but within the target range for THCA, indicating that there was a calculation error for  $\Delta^9$ -THC. It is important for laboratories to double check that their calculations are accounting for all dilution factors when determining cannabinoid values.



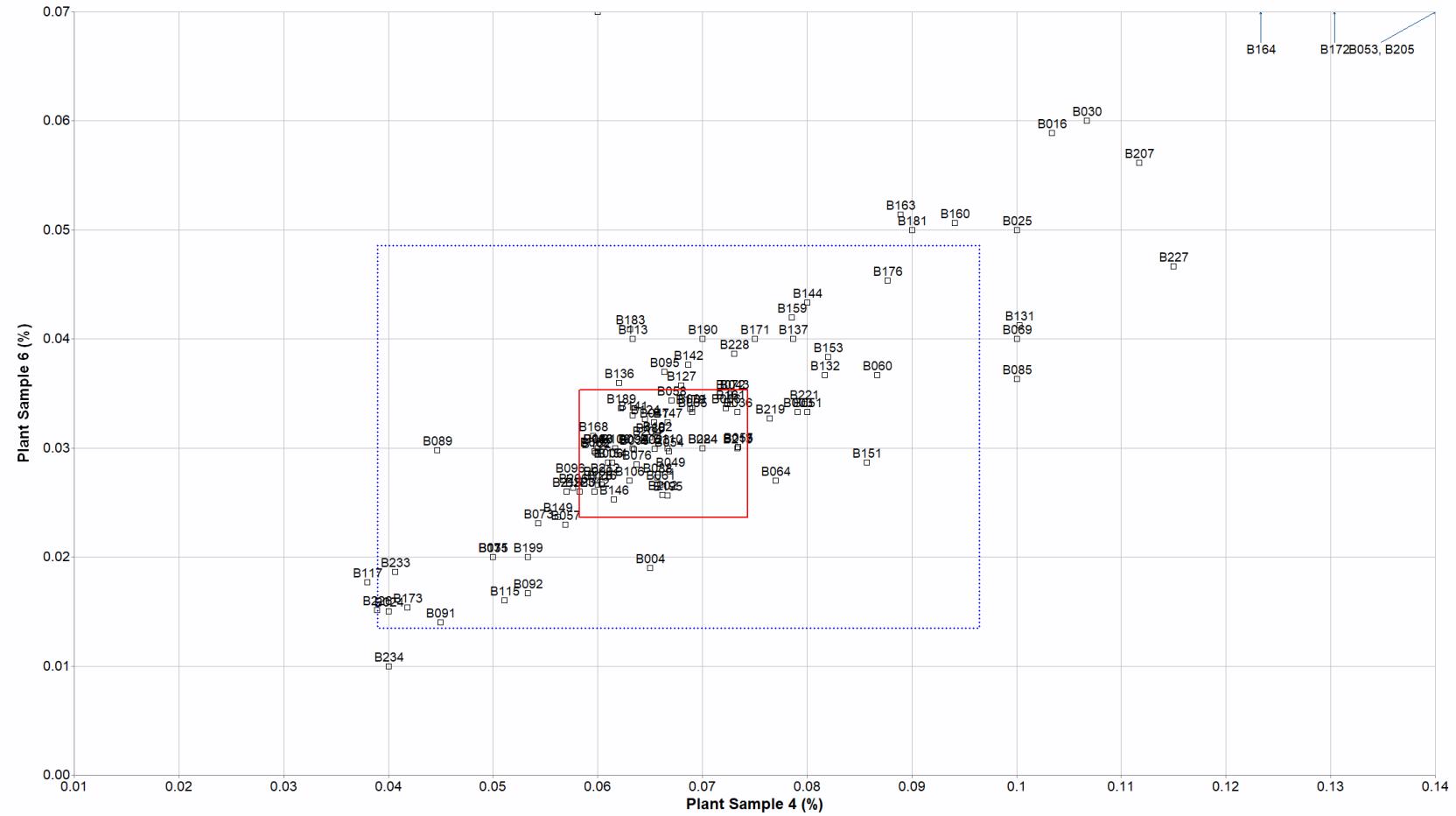
**Fig. 3-2. Laboratory means for  $\Delta^9\text{-THC}$  in NRC HEMP-1 and Plant Sample 6 (sample/sample comparison view).**

In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (Plant Sample 6). The solid red box represents the target range of tolerance for the two samples, NRC HEMP-1 (x-axis) and Plant Sample 6 (y-axis), which encompasses the target values bounded by their uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The dotted blue box represents the consensus range of tolerance for NRC HEMP-1 (x-axis) and Plant Sample 6 (y-axis), calculated as the values above and below the consensus means that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ .



**Fig. 3-3. Laboratory means for  $\Delta^9$ -THC in NRC HEMP-1 and Plant Sample 4 (sample/sample comparison view).**

In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (Plant Sample 4). The solid red box represents the target range of tolerance for the two samples, NRC HEMP-1 (x-axis) and Plant Sample 4 (y-axis), which encompasses the target values bounded by their uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The dotted blue box represents the consensus range of tolerance for NRC HEMP-1 (x-axis) and Plant Sample 4 (y-axis), calculated as the values above and below the consensus means that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ .

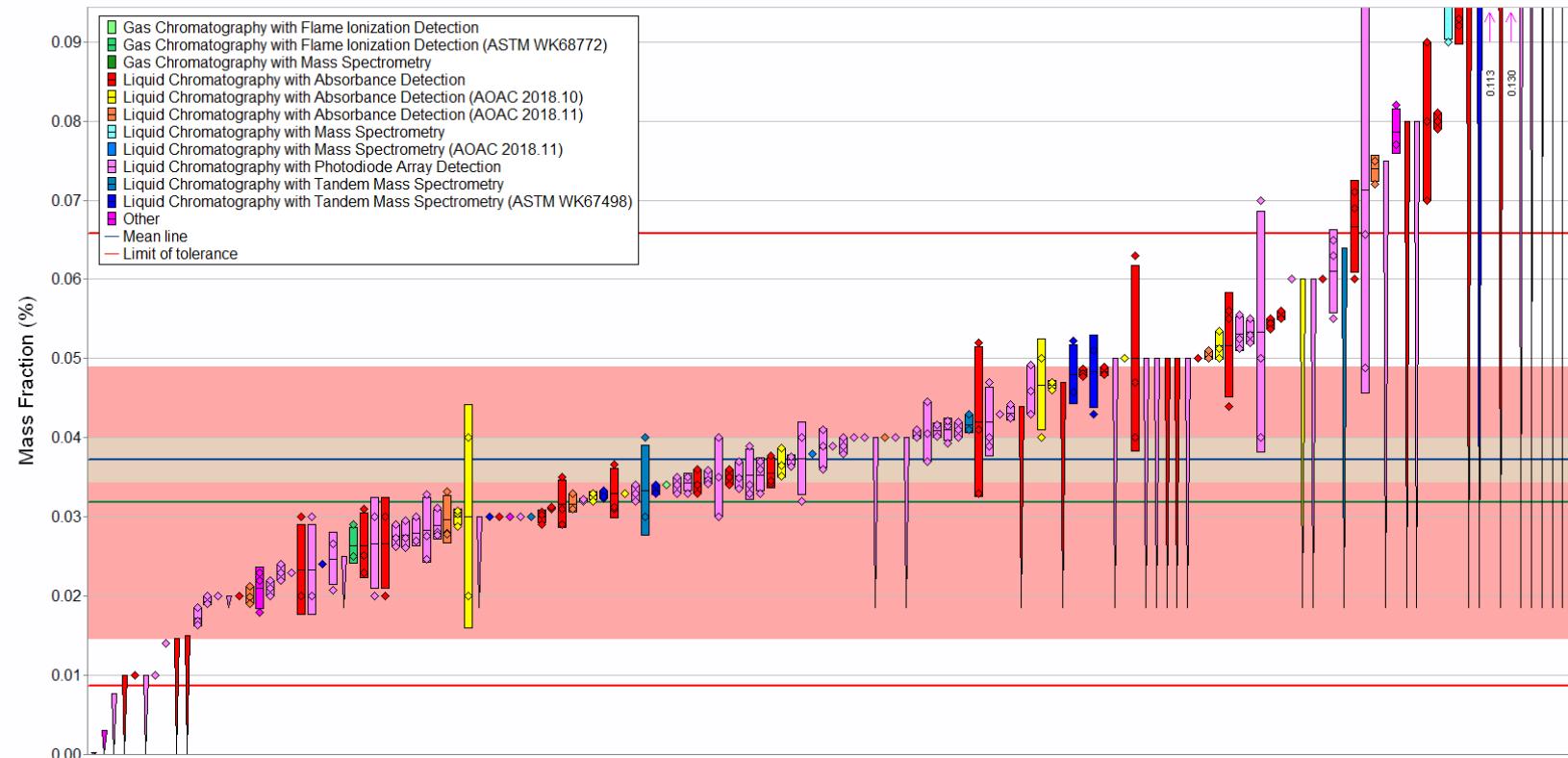


**Fig. 3-4. Laboratory means for  $\Delta^9\text{-THC}$  in Plant Sample 4 and Plant Sample 6 (sample/sample comparison view).**

In this view, the individual laboratory mean for one sample (Plant Sample 4) is compared to the individual laboratory mean for a second sample (Plant Sample 6). The solid red box represents the target range of tolerance for the two samples, Plant Sample 4 (x-axis) and Plant Sample 6 (y-axis), which encompasses the target values bounded by their uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The dotted blue box represents the consensus range of tolerance for Plant Sample 4 (x-axis) and Plant Sample 6 (y-axis), calculated as the values above and below the consensus means that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ .

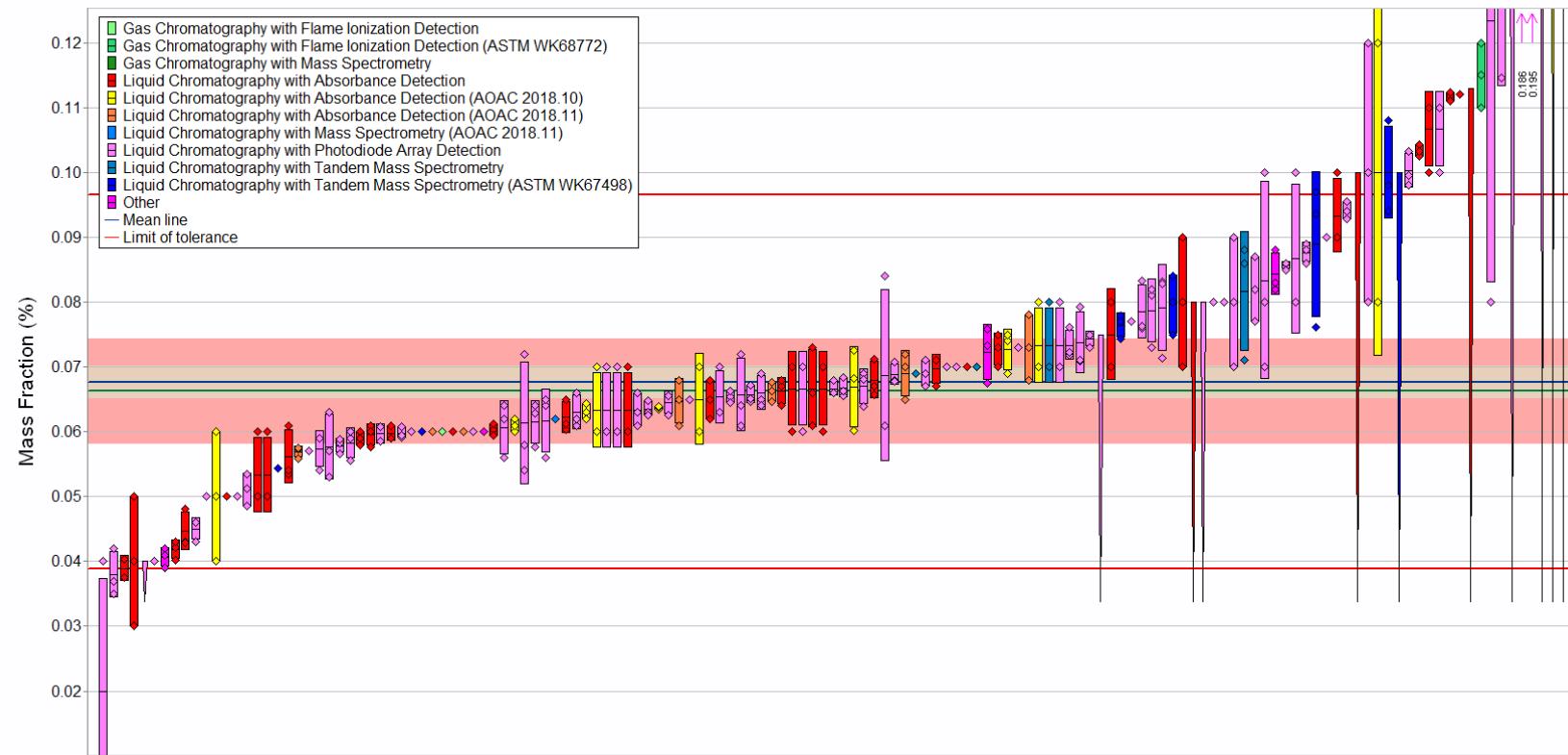
### 3.4.1.2. Accuracy

The individual participant, consensus, and target mass fraction results are presented in a tabular form in **Appendix B** and graphically in **Fig. 3-5** through **Fig. 3-10**. The consensus ranges for  $\Delta^9\text{-THC}$  in all cannabis samples were completely within the target ranges. Between 73 % and 86 % of participants used either LC-ABS or LC-PDA as their analytical method across all samples and no trend was observed to suggest bias due to use of one method over another. Between 25 % and 44 % of laboratories reporting quantitative results reported a mean result outside the target range for the three hemp samples and 15 % to 42 % of laboratories reported outside the target range for the three marijuana samples. For the marijuana samples, an increased percentage of laboratories reported outside the target range as the  $\Delta^9\text{-THC}$  value in the sample approached 0.3 %. The consensus (0.293 %  $\Delta^9\text{-THC}$ ) and target values (0.292 %  $\Delta^9\text{-THC}$ ) for Plant Sample 3 were only 0.34 % different; yet 42 % of participants reported  $\Delta^9\text{-THC}$  values outside both the target and consensus ranges for that sample (**Fig. 3-9**). It is possible that the upper limit of the calibration curves being used by these laboratories is close to 0.3 % for  $\Delta^9\text{-THC}$ . Some laboratories are not quantitating  $\Delta^9\text{-THC}$  above a threshold value that deems the material marijuana, which would be in the range of 0.3 % to 1 % for most jurisdictions. The accuracy for  $\Delta^9\text{-THC}$  measurements will decrease as the mass fractions near the limits of the calibration range, which may be the case for laboratories reporting outside the target and consensus ranges for Plant Sample 3 and Plant Sample 5.



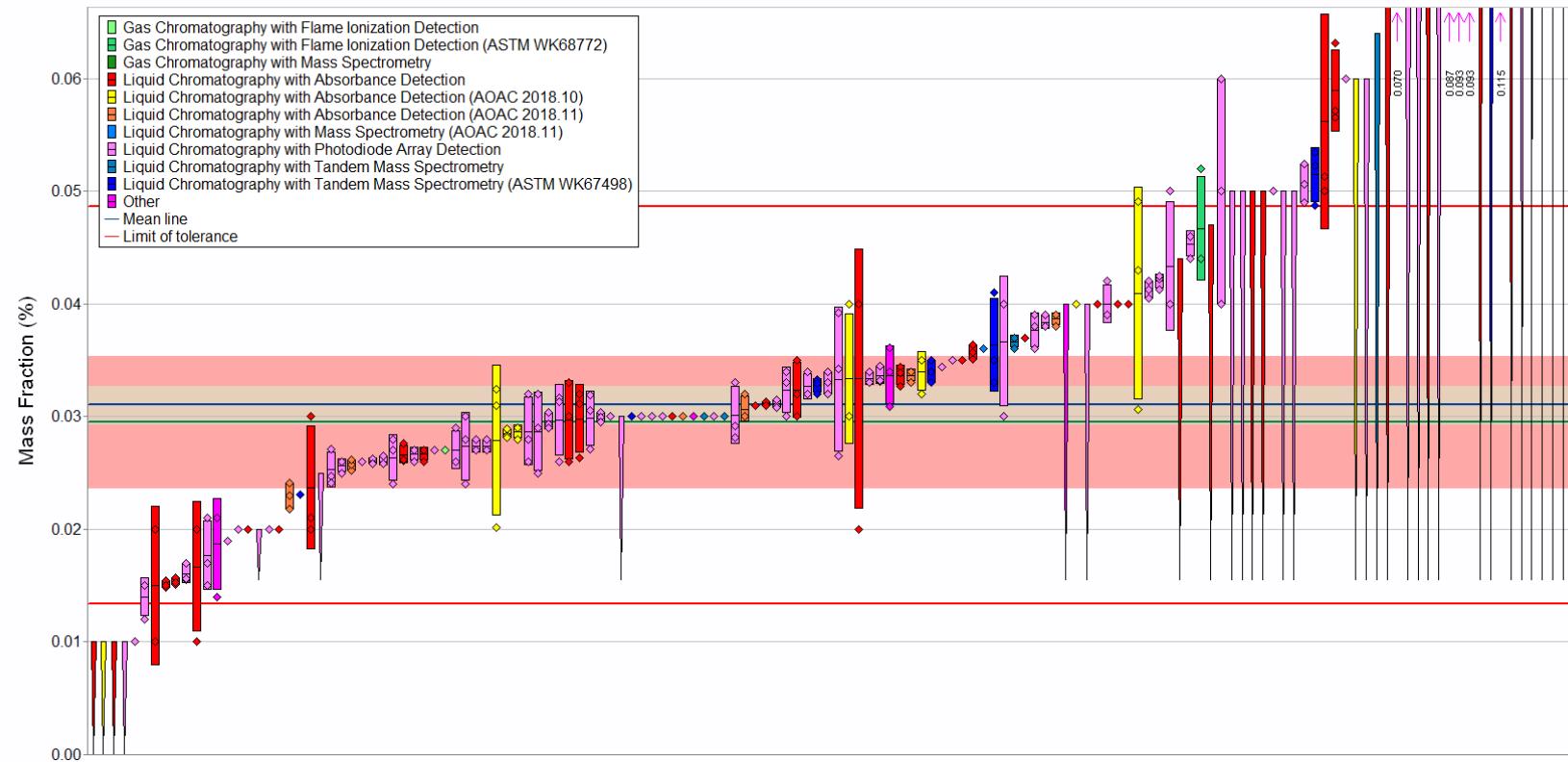
**Fig. 3-5.  $\Delta^9\text{-THC}$  in NRC HEMP-1 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its  $u$  uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



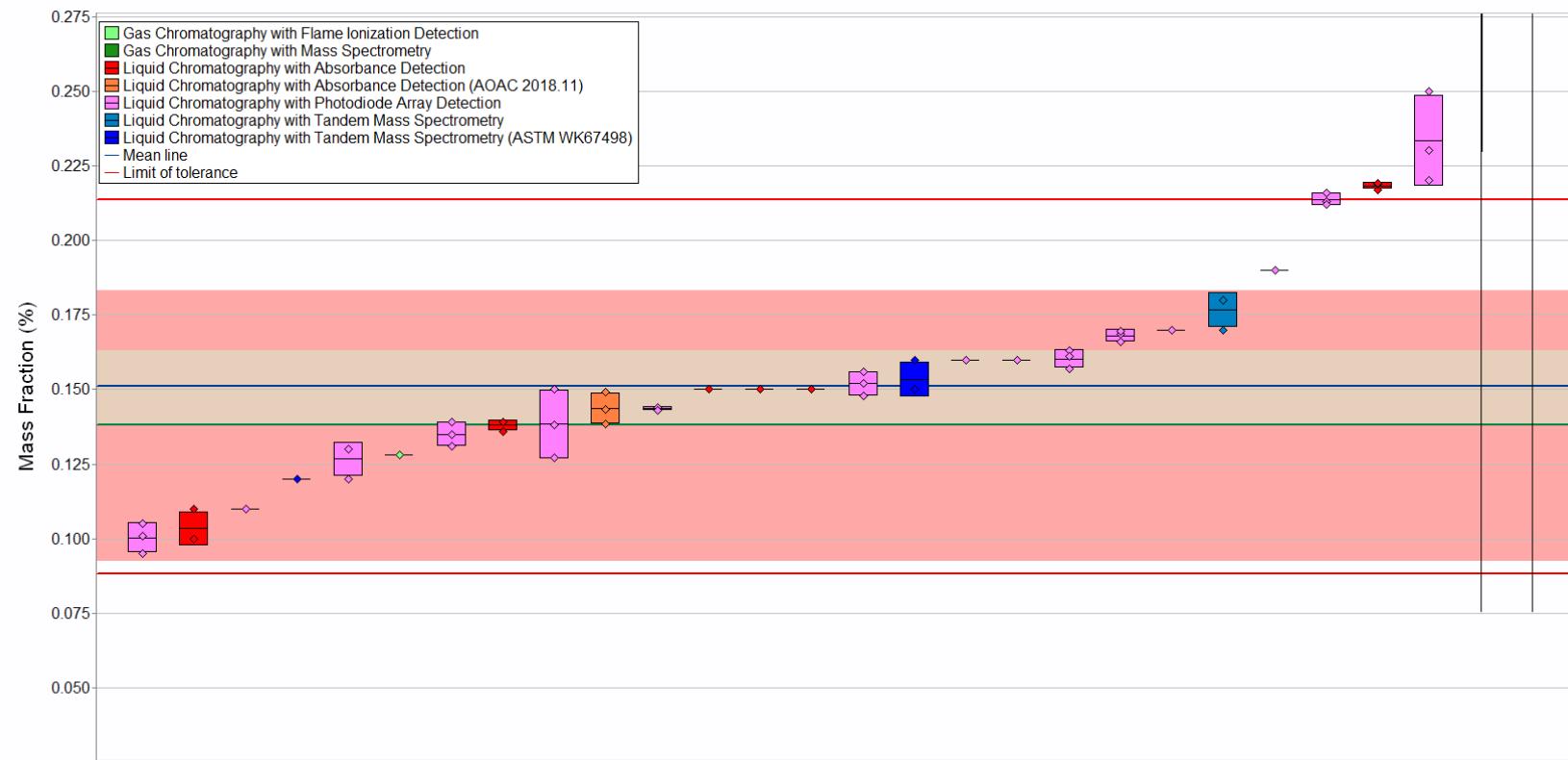
**Fig. 3-6.  $\Delta^9\text{-THC}$  in Plant Sample 4 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



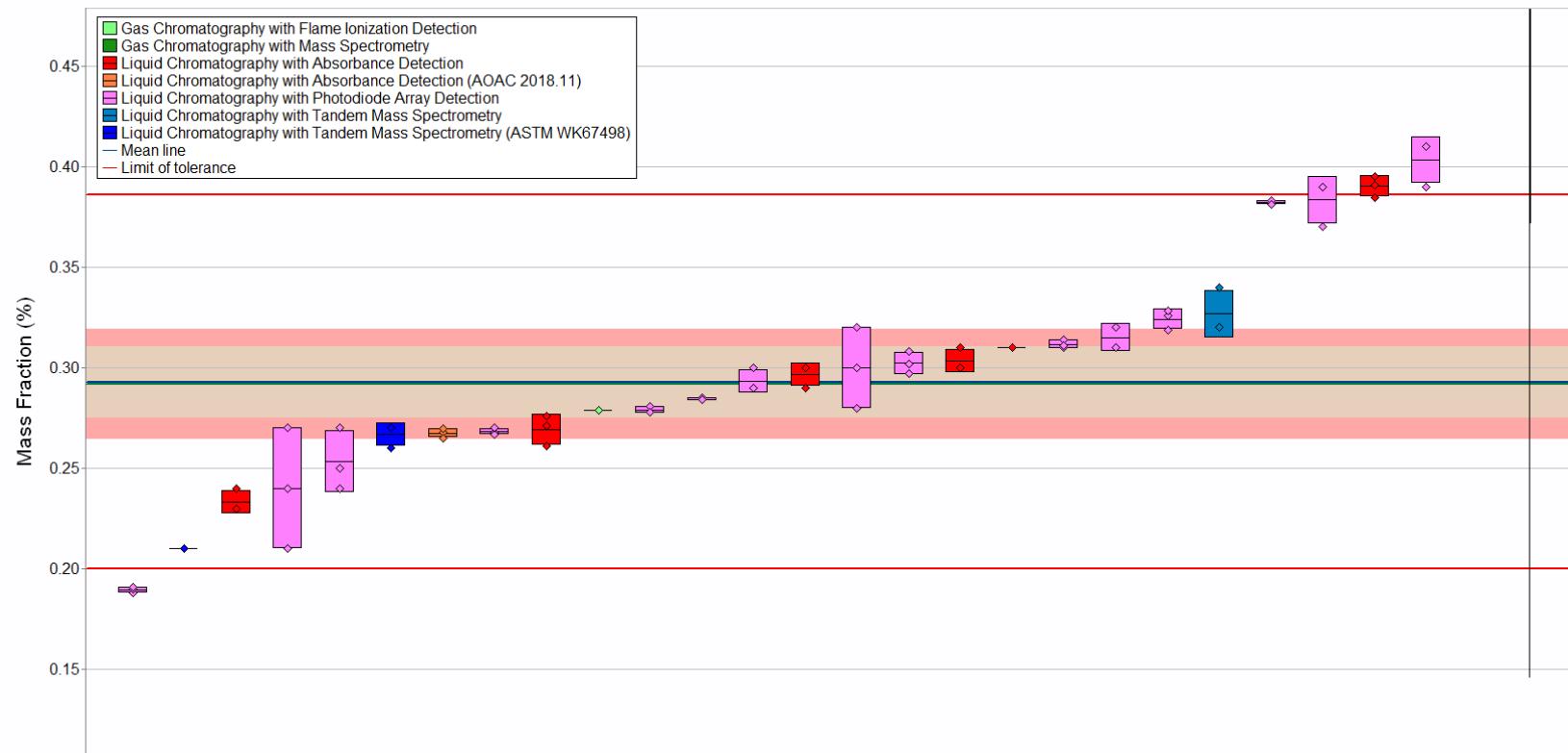
**Fig. 3-7.  $\Delta^9\text{-THC}$  in Plant Sample 6 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its  $u$  uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



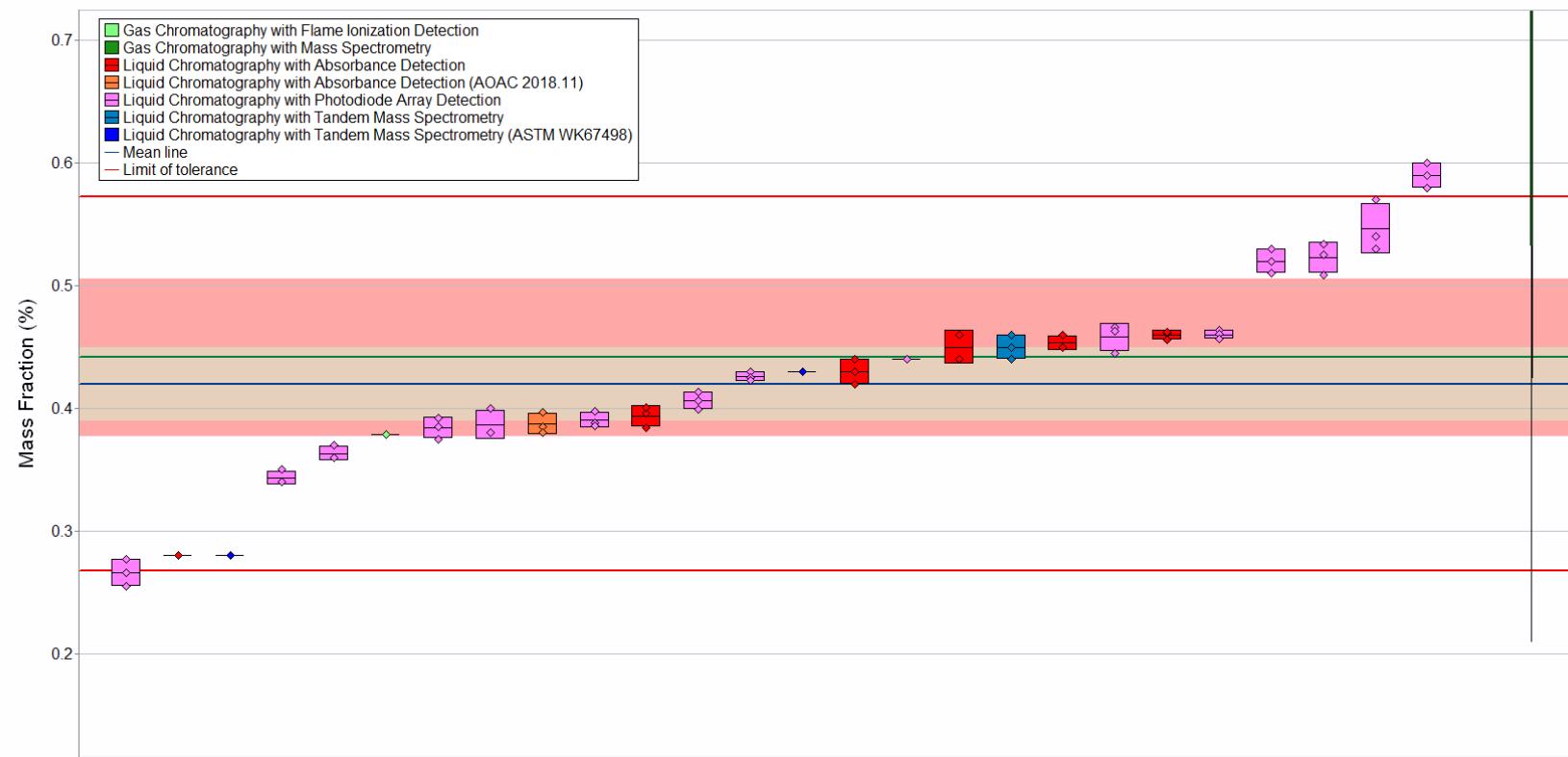
**Fig. 3-8.  $\Delta^9\text{-THC}$  in Plant Sample 2 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 3-9.  $\Delta^9\text{-THC}$  in Plant Sample 3 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 3-10.  $\Delta^9$ -THC in Plant Sample 5 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

With the exception of Plant Sample 5, the consensus values for  $\Delta^9$ -THC were 0.34 % to 16 % higher than the target values in the study samples. No obvious trends were observed correlating results to the analytical methods being used. The high bias in consensus values relative to the target values may be due to calibration issues, interfering cannabinoids, and/or decarboxylation of THCA into  $\Delta^9$ -THC. As was discussed in Section 3.4.1.1, the measurement errors appear to be more systematic than random. Calibration bias would present as a single laboratory consistently reporting  $\Delta^9$ -THC biased high or biased low for samples with  $\Delta^9$ -THC falling in the same region of the calibration curve. **Fig. 3-11** and **Fig. 3-12** show the  $Z'_{comm}$  scores for each laboratory across all six plant samples. In this figure, flags pointing to the right indicate values that were biased high relative to the consensus mean, and flags pointing to the left indicate values that were biased low relative to the consensus mean. Laboratories either consistently reported high or low for  $\Delta^9$ -THC across the hemp samples and the same was true for the majority of laboratories reporting values for the marijuana samples. In Section 3.4.1.4, an example shows how calibration curves can affect bias. To prevent calibration bias laboratories should use calibration standards that meet ISO standards, and ensure all purity information is reviewed, independently prepared routinely, and traceable to the SI, if possible. Accuracy will be improved if the calibration range being used during analysis does not extend over multiple orders of magnitude and sample dilutions are implemented during sample preparation.

While a similar percent of participants reported values over the target range for NRC HEMP-1 (22 %) and Plant Sample 4 (17 %), the consensus range for NRC HEMP-1 was above the target value and the consensus range for Plant Sample 4 contained the target value. The increased bias for  $\Delta^9$ -THC measurements in NRC HEMP-1 could be due to co-elution of interfering cannabinoids, namely CBNA, in addition to calibration bias, as discussed in Section 3.4.1.4. Coelution issues are resolvable by improving the baseline separation between analytes. Baseline separation is increasingly important for laboratories using UV absorbance detection because the absorbance spectra for cannabinoids are similar and absorbance detectors lack the specificity of mass spectrometers [20].

A reason for the  $\Delta^9$ -THC consensus values being generally higher than the target values is that the THCA in the study samples could have decarboxylated into  $\Delta^9$ -THC; however, the likelihood of this occurring is low for participants that properly stored the samples. A previous study observed that a decay in THCA resulted in a simultaneous increase in  $\Delta^9$ -THC for up to 450 days at room temperature, after which time  $\Delta^9$ -THC began degrading. However, when samples were refrigerated, the degradation rate of THCA was an order of magnitude lower and over the course of 700 days, the change in  $\Delta^9$ -THC concentrations were marginal [18]. Because the storage conditions for the samples in this study were recommended to be at -20 °C or below, the increase in  $\Delta^9$ -THC concentrations between the target and consensus values is not likely to be due to THCA decarboxylating into  $\Delta^9$ -THC.

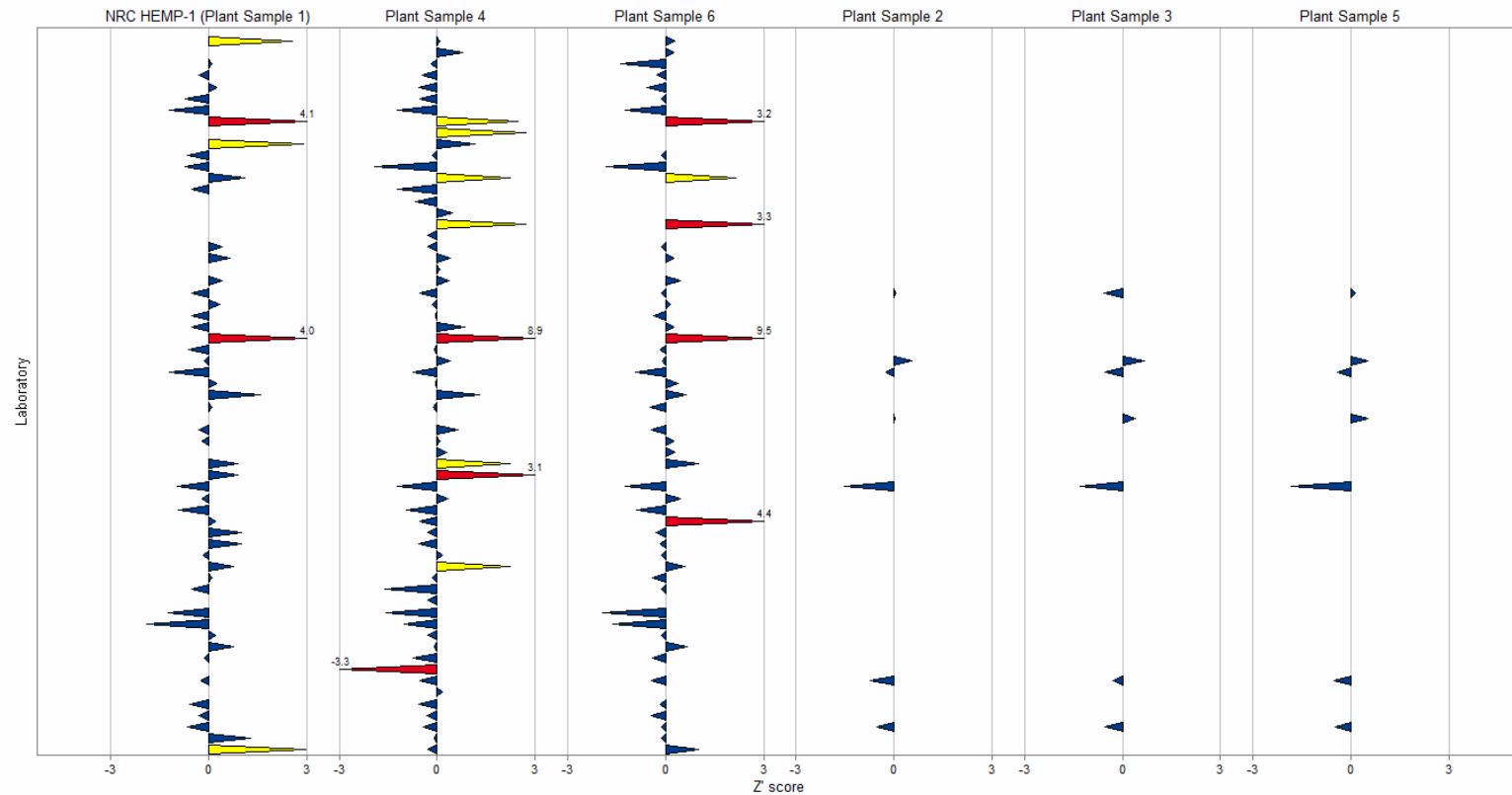
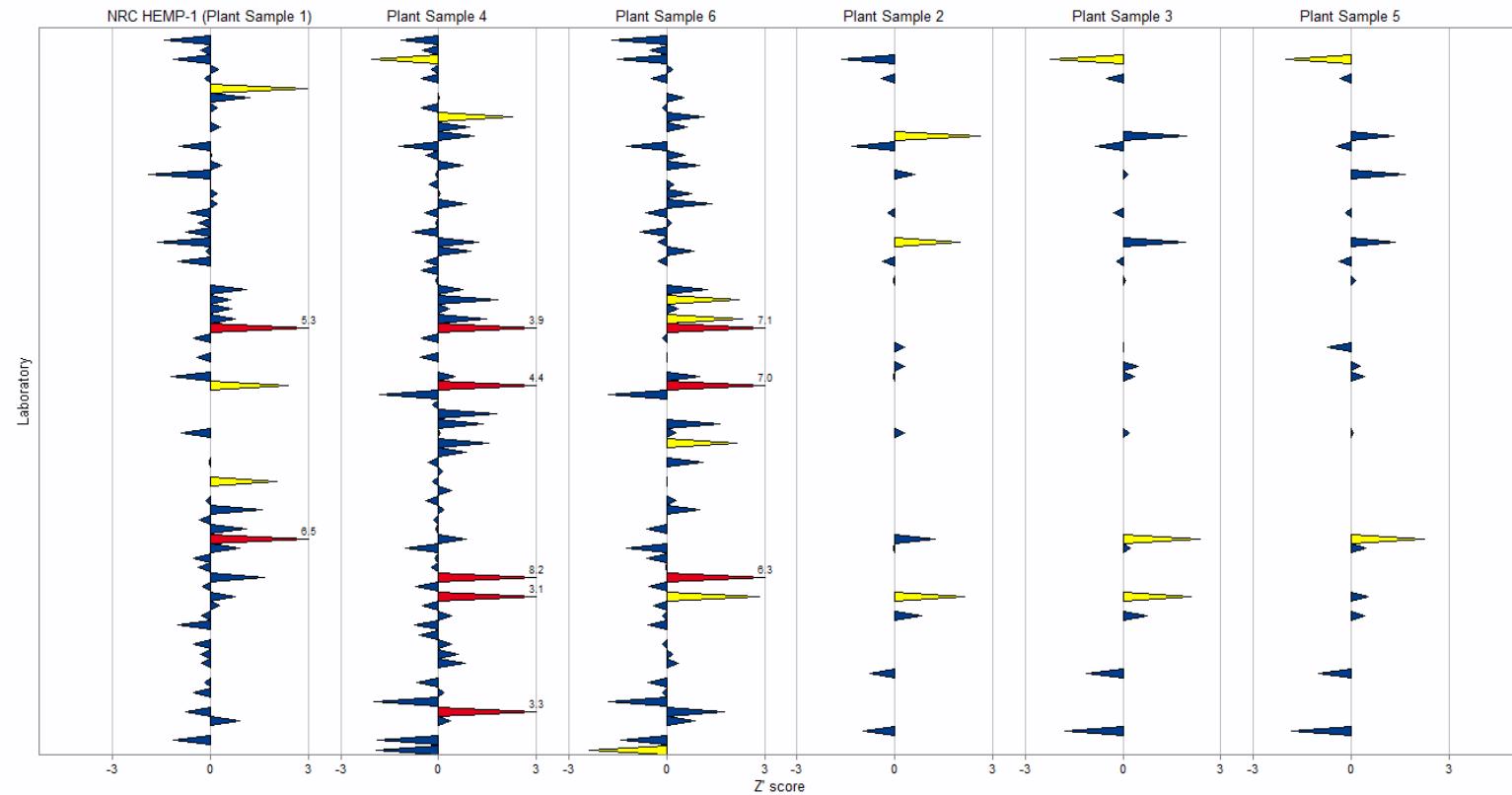


Fig. 3-11. Laboratory Z' scores for  $\Delta^9\text{-THC}$  for laboratories B001 to B114.

The blue triangles represent a Z' score within an acceptable  $Z'_\text{score}$  range,  $|Z'_\text{score}| \leq 2$ . The yellow triangles represent a Z' score outside the acceptable  $|Z'_\text{score}|$  range between 2 and 3. The red triangles represent a Z' score outside the acceptable  $|Z'_\text{score}|$  range,  $|Z'_\text{score}| > 3$ .



**Fig. 3-12. Laboratory Z' scores for  $\Delta^9$ -THC for laboratories B115 to B236.**

The blue triangles represent a Z' score within an acceptable  $Z'_\text{score}$  range,  $|Z'_\text{score}| \leq 2$ . The yellow triangles represent a Z' score outside the acceptable  $|Z'_\text{score}|$  range between 2 and 3. The red triangles represent a Z' score outside the acceptable  $|Z'_\text{score}|$  range,  $|Z'_\text{score}| > 3$ .

### 3.4.1.3. Candidate Analytical Methods

NIST provided a list of nine candidate standard methods for participants to use from AOAC International and ASTM International if an in-house analytical method was not available. Mass fractions were submitted by participating laboratories for  $\Delta^9$ -THC using an ASTM LC-MS/MS method and two AOAC methods. The within- (repeatability, %RSD<sub>r</sub>) and between-laboratory (reproducibility, %RSD<sub>R</sub>) variabilities are summarized for candidate method/sample pairs for which participants reported at least two independent measurements for a sample and at least five laboratories reported data (**Table 3-8**). Both AOAC methods were previously approved by an expert review panel using criteria established in SMPR 2017.002 for the quantitation of cannabinoids in dried cannabis plant samples [21], which requires %RSD<sub>r</sub> and %RSD<sub>R</sub> to be  $\leq 5\%$  and  $\leq 7\%$ , respectively, for samples containing 0.1 % to 1 %  $\Delta^9$ -THC. AOAC has since published SMPR 2019.003 for quantitation of cannabinoids in hemp plant samples [16], which accounts for lower mass fraction  $\Delta^9$ -THC materials. AOAC SMPR 2019.003 requires %RSD<sub>r</sub> and %RSD<sub>R</sub> to be  $\leq 5\%$  and  $\leq 10\%$ , respectively, for samples containing 0.05 % to 0.5 %  $\Delta^9$ -THC. The requirements established in SMPR 2019.003 were used in this study for the AOAC and ASTM methods.

**Table 3-8. Within- and between-laboratory variabilities for  $\Delta^9$ -THC measurements using candidate standardized analytical methods.**

	NRC HEMP-1			Plant Sample 4			Plant Sample 6		
	<u>n<sup>a</sup></u>	%RSD <sub>r</sub>	%RSD <sub>R</sub>	<u>n<sup>a</sup></u>	%RSD <sub>r</sub>	%RSD <sub>R</sub>	<u>n<sup>a</sup></u>	%RSD <sub>r</sub>	%RSD <sub>R</sub>
LC-Absorbance									
AOAC 2018.10	8	4.0	28.0	10	12.4	13.0	7	9.3	17.6
AOAC 2018.11	6	3.6	56.3	7	4.0	11.7	7	2.2	31.4
<u>LC-MS/MS</u>									
ASTM WK67498	5	4.2	17.5	5	4.8	35.6	5	3.6	17.0

<sup>a</sup> n = number of laboratories

#### AOAC 2018.10

The %RSD<sub>r</sub> published for AOAC 2018.10 was 3.56 % for dried flowers containing 0.03 %  $\Delta^9$ -THC ( $n = 4$ , [22]), which was within the published requirement of  $\leq 5\%$  [16]. The average within-laboratory variability observed for the participants reporting use of AOAC 2018.10 was greater than both the %RSD<sub>r</sub> published in the method and the SMPR for Plant Sample 4 and Plant Sample 6. The  $\Delta^9$ -THC level in Plant Sample 6 (0.0295 %), however, was below the LOQ in the published method (0.03 %), so slightly higher within-laboratory variability might be expected in this sample. The  $\Delta^9$ -THC level in Plant Sample 4 (0.0663 %) was above the stated method LOQ and laboratories should have achieved %RSD<sub>r</sub>  $\leq 5\%$ . The  $\Delta^9$ -THC values reported by laboratories using AOAC 2018.10 were higher than the target value for NRC HEMP-1 (28 %) and Plant Sample 6 (14 %) and lower than the target value for Plant Sample 4 (1.5 %). The between-laboratory variabilities observed for  $\Delta^9$ -THC measured by AOAC 2018.10 in the hemp samples were above the 10 % requirement for %RSD<sub>R</sub> [16]; observed reproducibility of AOAC 2018.10 had not been published at the time of this report.

### AOAC 2018.11

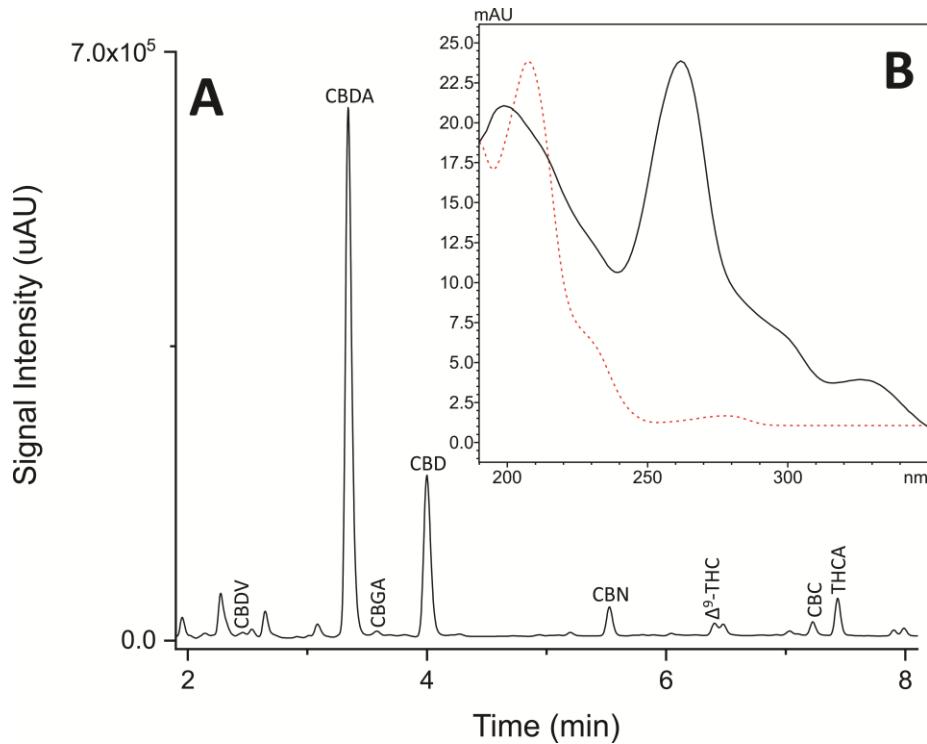
The %RSD<sub>r</sub> published for AOAC 2018.11 was reported from two separate analysts [7]. The combined %RSD<sub>r</sub> was 2.9 % for dried plant material samples containing 0.04 % Δ<sup>9</sup>-THC ( $n = 10$ ), which was within the published requirement of ≤ 5 % [16]. The average within-laboratory variability observed for the participants reporting use of AOAC 2018.11 was consistent with the %RSD<sub>r</sub> published in the method and the SMPR [16]. The Δ<sup>9</sup>-THC levels in all samples were above the published LOQ for AOAC 2018.11 (0.010 %). Laboratories using the AOAC 2018.11 method reported Δ<sup>9</sup>-THC values that were higher than the target value for NRC HEMP-1 (28 %) and Plant Sample 6 (7 %) and lower than the target value for Plant Sample 4 (3 %). The between-laboratory variabilities observed for Δ<sup>9</sup>-THC measured using AOAC 2018.11 in the hemp samples were above the 10 % requirement for %RSD<sub>R</sub> [16]; observed reproducibility of AOAC 2018.11 had not been published at the time of this report.

### ASTM WK67498

The ASTM WK67498 method was developed following the AOAC [23] and ASTM [24] guidelines. The %RSD<sub>r</sub> published for ASTM WK67498 was tested on 5 lots of the same hemp sample with %RSD<sub>r</sub> between 2 % and 5 % for hemp samples containing between 0.0365 % and 0.0481 % Δ<sup>9</sup>-THC ( $n = 3$ , [25]), which were all within the published requirement of ≤ 5 % [16,21]. The average within-laboratory variability observed for the participants reporting use of ASTM WK67498 was consistent with the %RSD<sub>r</sub> published in the method and the SMPR [16]. Laboratories using the ASTM WK67498 method reported Δ<sup>9</sup>-THC values that were higher than the target value for NRC HEMP-1 (19 %), Plant Sample 4 (23 %), and Plant Sample 6 (24 %). The between-laboratory variabilities observed for Δ<sup>9</sup>-THC measured using ASTM WK67498 in the hemp samples were above the 10 % requirement [16]; observed reproducibility of ASTM WK67498 had not been published at the time of this report.

#### 3.4.1.4. Examples and Recommendations

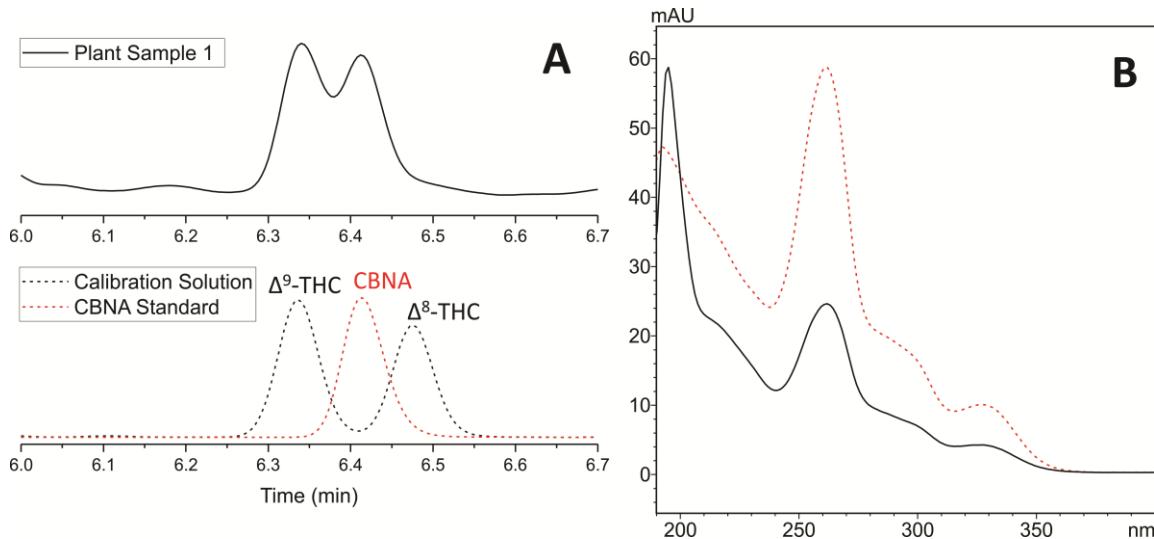
For the laboratories that are reporting Δ<sup>9</sup>-THC above the target and consensus ranges using absorbance as the detection method, the likelihood of coeluting cannabinoids is high if the chromatographic method has not been thoroughly evaluated. As shown in **Table 3-7**, most laboratories reported results from LC-UV and LC-PDA measurements using a single absorbance wavelength and may have relied solely on retention time for Δ<sup>9</sup>-THC identification without evaluating the purity of the chromatographic peak. The potential for coelution is highlighted for Δ<sup>9</sup>-THC in NRC HEMP-1 (**Fig. 3-13 A**) using the LC-PDA method summarized in Section 2.2.2.1. Δ<sup>9</sup>-THC was identified in the chromatogram based on the retention time and confirmed using the UV absorbance spectrum. The co-eluting peak at the tail of the Δ<sup>9</sup>-THC peak was tentatively identified by the instrumental software as Δ<sup>8</sup>-THC based on the retention time of 6.42 min for Δ<sup>9</sup>-THC versus 6.53 min for Δ<sup>8</sup>-THC. However, the absorbance spectra in **Fig. 3-13 B** confirmed that the suspected peak in NRC HEMP-1 was not consistent with the Δ<sup>8</sup>-THC absorbance spectrum in the calibration standard.



**Fig. 3-13. Absorbance spectra rejects initial identification of coeluting  $\Delta^9$ -THC peak.**

Panel A displays a LC-UV chromatogram at 220 nm for NRC HEMP-1 that has a peak that overlaps that of  $\Delta^9$ -THC. The thin black curve in the panel B in Inset displays the absorbance spectra of the peak that coelutes at the tail of the  $\Delta^9$ -THC peak; the dotted red curve displays the spectrum of a  $\Delta^8$ -THC calibration standard.

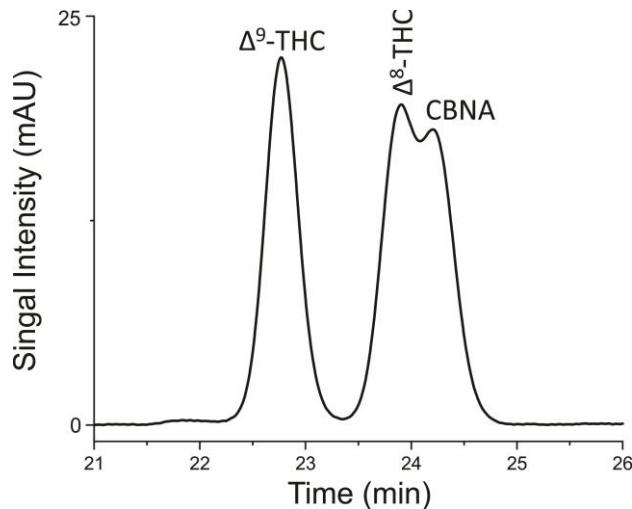
Upon further investigation, the coeluting peak was tentatively identified as CBNA by comparing the retention times and absorbance spectra from the extract to a CBNA reference standard (**Fig. 3-14**). The influence of CBNA could not be removed from the chromatographic peak of  $\Delta^9$ -THC by adjusting the detection wavelength. For quantitative measurements at NIST, coeluting peaks were integrated as one peak and then split using the drop down tools in the instrumental software, resulting in a  $\Delta^9$ -THC mean mass fraction and SD of  $0.0328\% \pm 0.0006\%$ , which was slightly higher (but not significantly different) than the NRC Canada certified value of  $0.0318\% \pm 0.0086\%$  [13]. The influence from CBNA in the NIST LC-PDA method did not significantly impact the  $\Delta^9$ -THC quantitation because the CBNA and  $\Delta^9$ -THC peaks had similar chromatographic signal responses and mass fractions [6]. However, if  $\Delta^9$ -THC and CBNA were less separated in the chromatographic method, the final mass fraction of  $\Delta^9$ -THC would have been more significantly biased, which may be an issue for laboratories using LC-absorbance and reporting above the target and consensus ranges. This example demonstrates the importance of evaluating the absorbance spectra across a chromatographic peak as was summarized in Section 2.2.3.



**Fig. 3-14. Absorbance spectra confirms identification of coeluting  $\Delta^9$ -THC peak.**

The upper segment of Panel A displays at high resolution the  $\Delta^9$ -THC segment of the LC-UV chromatogram for NRC HEMP-1. The lower segment of Panel A displays the same chromatographic segment for the calibrant 4 solution which contains  $\Delta^9$ -THC and  $\Delta^8$ -THC (dotted black line) along with the same segment for the CBNA standard (dotted red line). The inset panel B displays the absorbance spectra collected at the maximum of the suspected unknown peak in NRC HEMP-1 (solid black curve) and CBNA reference standard (dotted red curve).

Laboratories that are consistently reporting  $\Delta^9$ -THC values outside the target and consensus ranges should explore potential biases in the chromatographic method. Previous LC studies have been conducted at NIST investigating the different retention characteristics of 15 cannabinoids including  $\Delta^9$ -THC,  $\Delta^8$ -THC, and CBNA as shown in Fig. 3-15. The LC chromatogram was obtained at 220 nm using a C<sub>18</sub> column with the dimensions of 150 mm x 4.6 mm and 3  $\mu$ m average particle diameter. Cannabinoids were separated with a 1.0 mL/min flow rate, 15 °C column temperature, and isocratic elution at 75 % ACN and 25 % H<sub>2</sub>O mobile phase with 0.1 % FA.



**Fig. 3-15. LC-UV chromatogram at 220 nm for a mixture of  $\Delta^9$ -THC,  $\Delta^8$ -THC, and CBNA calibration solutions.**

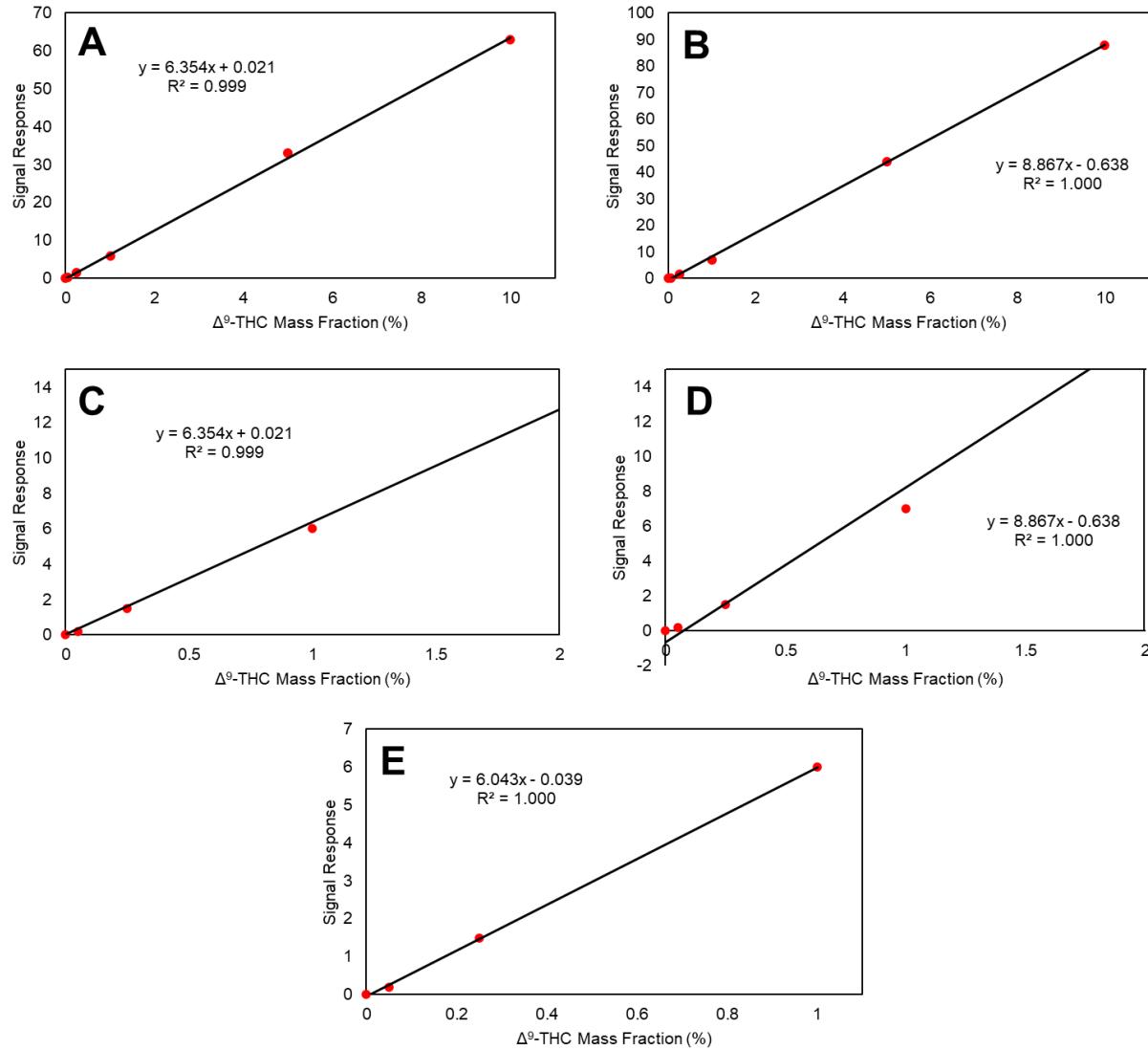
Chromatogram used an isocratic mobile phase of 75 % ACN and 25 % H<sub>2</sub>O (0.1 % FA) on a C18 stationary phase.

A significant retention difference was observed for CBNA using this LC method (**Fig. 3-15**), compared to the separation using the LC method described in Section 2.2.2.1 (**Fig. 3-14**). In the LC method with gradient elution and a column temperature of 40 °C,  $\Delta^9$ -THC coelutes with CBNA and  $\Delta^8$ -THC; whereas in the LC method with isocratic separation at 75 % ACN and a column temperature of 15 °C,  $\Delta^9$ -THC is baseline separated from both CBNA and  $\Delta^8$ -THC. Similar observations were found with 79 % MeOH and 21 % H<sub>2</sub>O mobile phase with 0.1 % FA (data not shown). Analytical methods must be modified to improve baseline separation of  $\Delta^9$ -THC from other cannabinoids and should be routinely reevaluated for potential chromatographic cannabinoid interferences as commercial standards become available, even after methods are validated and implemented.

The high bias for  $\Delta^9$ -THC in the hemp samples could also be caused using a wide calibration range targeted for measuring  $\Delta^9$ -THC in both hemp and marijuana samples. Even though a calibration curve with an analytical range of (0.0 % to 10.0 %) will obtain a correlation coefficient ( $r^2$ )  $\approx$  0.999, the best fit line ( $y = mx + b$ ) for the calibration curve will be influenced more by the higher calibration points than the lower points resulting in two scenarios of possible calibration bias (Scenario 1, **Fig. 3-16 A** and Scenario 2, **Fig. 3-16 B**).

The calibration bias represented in Scenario 1 (**Fig. 3-16 A** and **C**) results in a signal response for the 5.0 % and 10.0 % calibration points of 33 and 63, respectively. The best fit line in this scenario falls above the lower calibration points, as depicted in **Fig. 3-16 C**, resulting a high bias for an unknown sample with a mass fraction in this lower range. However, in Scenario 2 (**Fig. 3-16 B** and **Fig. 3-16 D**), the 5.0 % and 10.0 % calibration points had a signal response of 44 and 88, respectively. The best fit line in Scenario 2 falls below the two lowest calibration points, as depicted in **Fig. 3-16 D**, resulting in a low bias for an unknown sample with a mass fraction in this lower range. Extending a calibration range across multiple orders of magnitude is useful to demonstrate versatility of a method, but ultimately can result in biases in either direction when used to quantify cannabinoid concentrations over a small range.

As shown in **Fig. 3-16 E**, removal of the 5.0 % and 10.0 % calibration points results in an improved best fit line for the lower calibration range, which would more accurately represent the  $\Delta^9$ -THC in the lower mass fraction samples. This example illustrates the importance of ensuring that the calibration curve for quantitation is representative of the cannabinoid concentrations in the sample. If the cannabinoid concentration falls in the lower section of the calibration curve, consider truncating the curve to determine the mass fraction with improved accuracy.



**Fig. 3-16. The impact of calibration curve design.**

Panels A through D depict a cannabinoid with an analytical range of 0.0 % to 10.0 %. Calibration curves A and B illustrate the same calibration range, but A has a lower signal response for the 5.0 % and 10.0 % calibrants compared to B. Calibration curves C and D are zoomed in versions of A and B, respectively to illustrate the vertical distance of each point from the trendline. Calibration curve E has an analytical range of 0.0 % to 1.0 %.

### 3.4.2. THCA

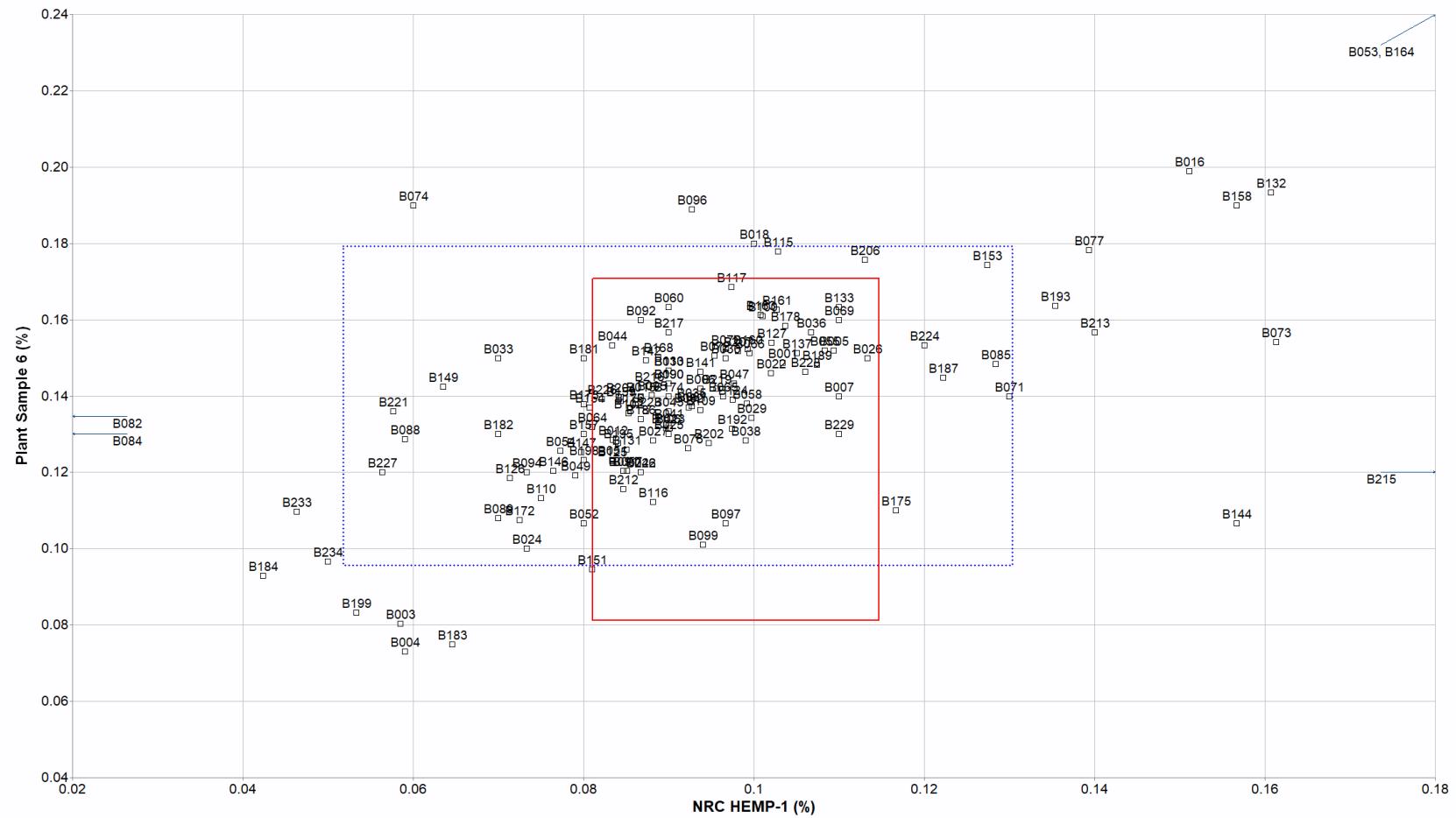
#### 3.4.2.1. Within- and Between Laboratory Precision

Laboratories reporting quantitative results for THCA in all plant samples demonstrated within-laboratory precision ( $\leq 4.4\%$ ) at or below the published expectation of  $\leq 5\%$  for cannabinoids in hemp [16], with higher variability reported for the hemp samples than the marijuana samples (**Table 3-6**). A majority of laboratories measuring THCA demonstrated repeatability at or below 5 % for NRC HEMP-1 (59 %), Plant Sample 4 (60 %), and Plant Sample 6 (69 %). Higher precision ( $\leq 3\%$ ) is recommended for plant samples with mass fractions of THCA between 0.5 % and 5 %

[16], which is applicable for Plant Sample 3 and Plant Sample 5. In general, more laboratories reported within-laboratory variabilities for THCA measurements at or below the published expectations in Plant Sample 2 (75 %), Plant Sample 3 (65 %), and Plant Sample 5 (81 %) than in the hemp samples. Greater than desired within-laboratory variability in all samples may be due to the use of smaller sample size in comparison to NIST homogeneity measurements (0.5 g) or additional sample grinding (**Appendix C**).

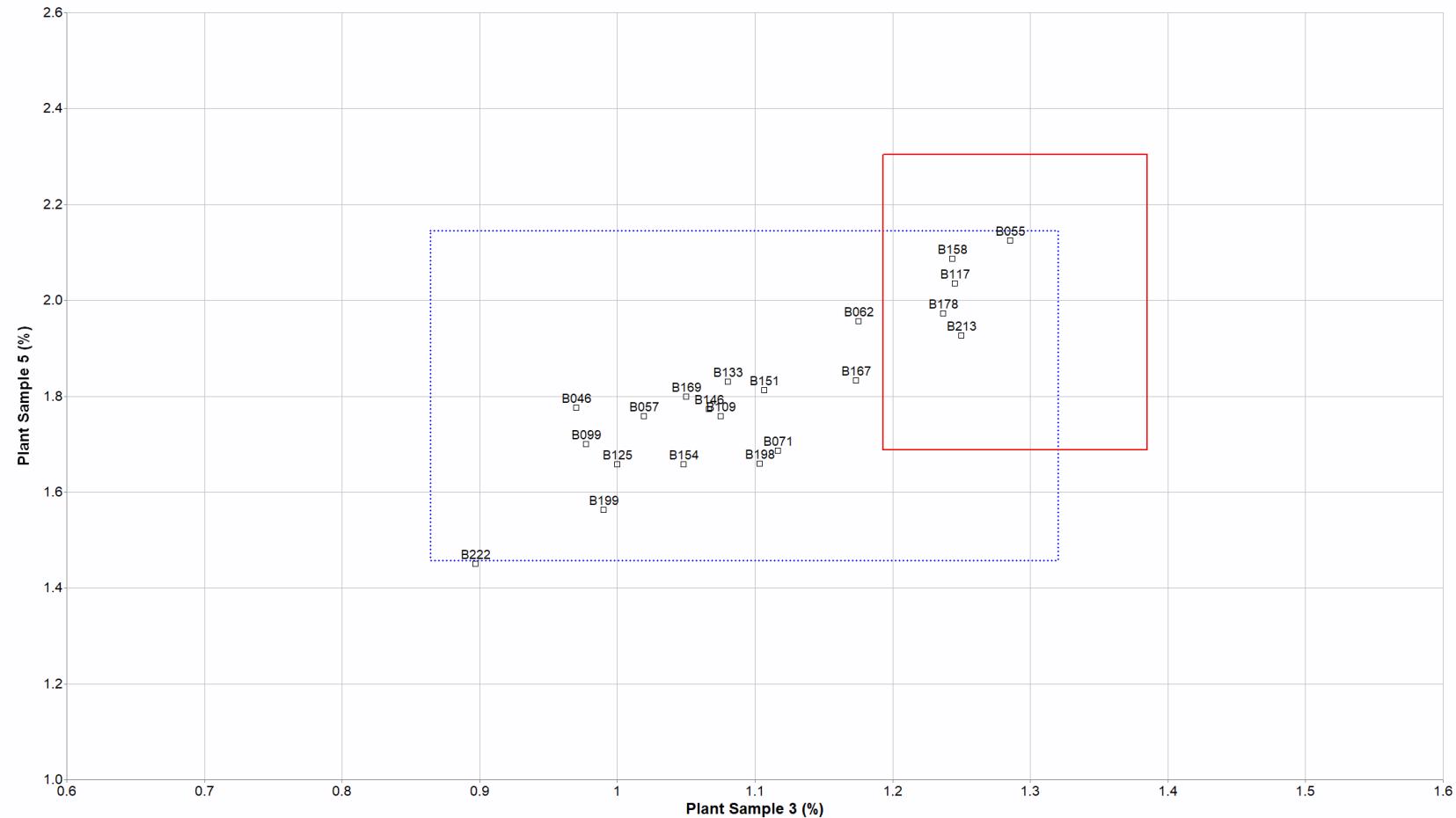
As observed for  $\Delta^9$ -THC measurements, the between-laboratory variabilities for THCA measurements were higher in the hemp samples than the marijuana samples. The between-laboratory variabilities for THCA measurements ranged from 9.6 % (Plant Sample 5) to 21.6 % (NRC HEMP-1), which were all outside the published requirements of  $\leq 10$  % and  $\leq 8$  % based on THCA mass fractions [16]. However, the AOAC guidelines for between-laboratory precision are meant to be applied to data from multiple laboratories using a single analytical method not data from multiple laboratories using multiple analytical methods, as was the case for this study. The between-laboratory variabilities for THCA in the study samples for this exercise are comparable to the UK-PT program overall analyte RSDs (13.4 % to 25.4 %) for samples having as received THCA mass fractions similar to that in NRC HEMP-1 [19].

To further examine the potential causes of between-laboratory variability, laboratory performance on two separate samples with similar analyte mass concentrations was compared. NRC HEMP-1 and Plant Sample 6 contained the lowest levels of THCA in the study samples. The THCA data from these samples exhibited a slightly linear trend, indicating the presence of both systematic and random errors (**Fig. 3-17**). The low mass fractions of THCA in NRC HEMP-1 and Plant Sample 6 may have led to increased likelihood of systematic errors such as calibration bias and potential for coeluting compounds. Differences in sample storage conditions, balance and pipetting procedures, and as well as data entry errors may cause random variability in the results. Some laboratories reported THCA values within the target and consensus ranges for Plant Sample 6 and outside the ranges for NRC HEMP-1 (B215, B082, and B084). For two of these laboratories, the reported values were an order of magnitude outside the target value, and for one, the value entered was zero. These results lead to false outliers even though participants were asked to double check values after receipt of the preliminary certificate. If an analyte is not detected, a laboratory should report the LOQ value rather than zero. The majority of laboratories consistently reported either high or low relative to the target and consensus ranges for marijuana samples with the highest THCA mass fractions, which is indicative of systematic error (**Fig. 3-18**). All laboratories that reported low THCA mass fractions did so in both Plant Sample 3 and Plant Sample 5, which could indicate poor sample storage, inadequate sample preparation procedures, or calibration issues.



**Fig. 3-17. Laboratory means for THCA in Plant Sample 6 and NRC HEMP-1 (sample/sample comparison view).**

In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (Plant Sample 6). The solid red box represents the NIST range of tolerance for the two samples, NRC HEMP-1 (x-axis) and Plant Sample 6 (y-axis), which encompasses the target values bounded by their uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The dotted blue box represents the consensus range of tolerance for NRC HEMP-1 (x-axis) and Plant Sample 6 (y-axis), calculated as the values above and below the consensus means that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ .



**Fig. 3-18. Laboratory means for THCA in Plant Sample 3 and Plant Sample 5 (sample/sample comparison view).**

In this view, the individual laboratory mean for one sample (Plant Sample 3) is compared to the individual laboratory mean for a second sample (Plant Sample 5). The solid red box represents the NIST range of tolerance for the two samples, Plant Sample 3 (x-axis) and Plant Sample 5 (y-axis), which encompasses the target values bounded by their uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The dotted blue box represents the consensus range of tolerance for Plant Sample 3 (x-axis) and Plant Sample 5 (y-axis), calculated as the values above and below the consensus means that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ .

### 3.4.2.2. Accuracy

The individual participant, consensus, and target mass fraction results are presented in tabular form in **Appendix B** with examples shown in **Fig. 3-19** through **Fig. 3-24**. Between 83 % and 87 % of participants used either LC-ABS or LC-PDA as their analytical method across all samples and no trend was observed to suggest bias of one method over another. The consensus values for THCA were between 7 % (NRC HEMP-1) and 15 % (Plant Sample 3) lower than the target values for all but Plant Sample 6, which had a consensus value approximately 9 % higher than the target value. Potential reasons for decreased THCA concentrations include decarboxylation of THCA due to improper sample storage conditions, incomplete extraction, and calibration errors as discussed in Section 3.4.1.4.

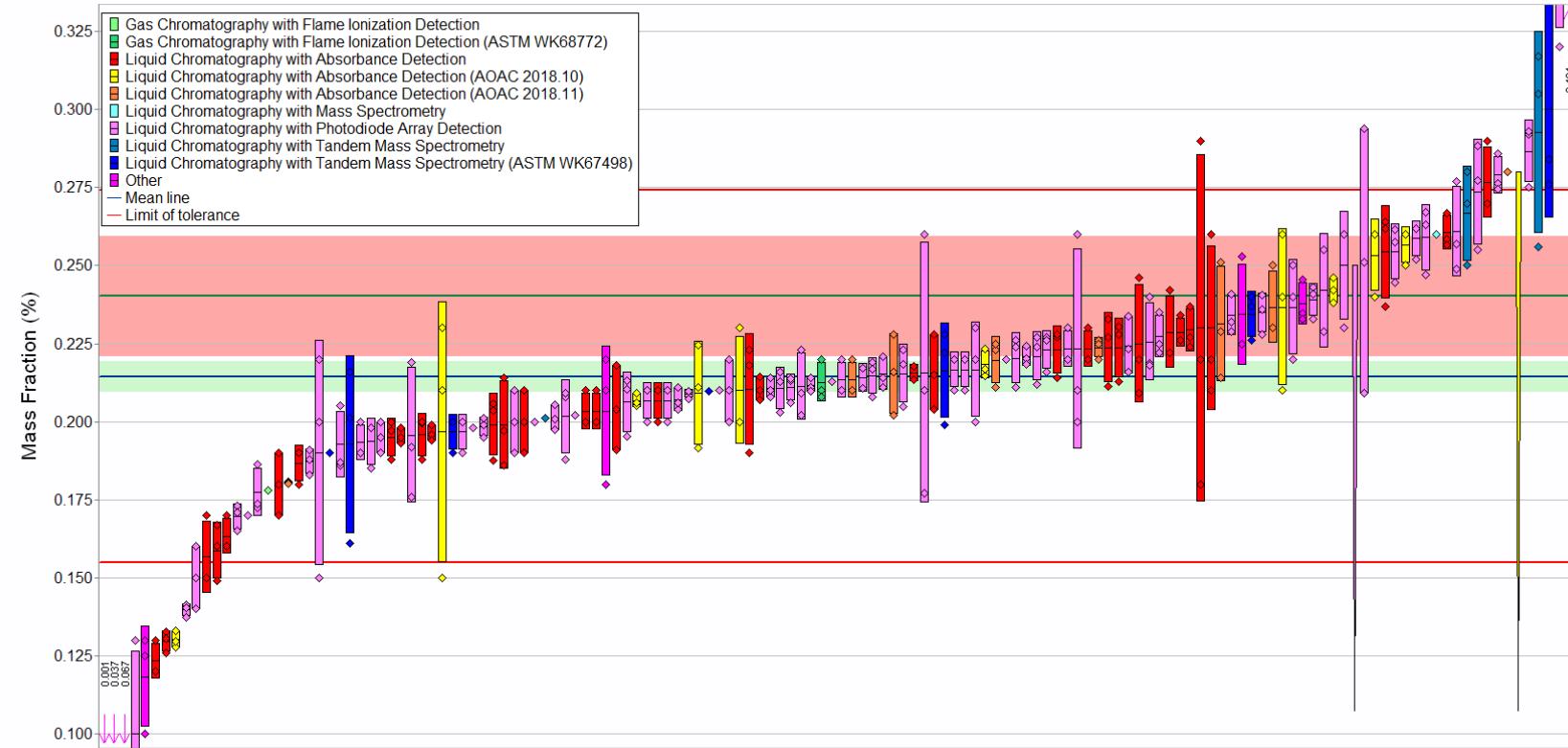
As mentioned in Section 3.4.2.1, Plant Sample 6 and NRC HEMP-1 contained similar amounts of THCA. If all of the error in estimating THCA was due to calibration bias, the consensus value for THCA in both the NRC HEMP-1 and in Plant Sample 6 would be expected to have similar bias. However, the consensus value for THCA in NRC HEMP-1 was below the target value and for Plant Sample 6, the consensus value for THCA was above the target value. NRC HEMP-1 and Plant Sample 6 are independently prepared materials, and the extractability of THCA may have been higher in one sample than the other. Plant Sample 6 contained all stems with a particle size of < 250 µm. Even though the particle size was small, the THCA was likely more easily extracted from the less dense particles. The consensus value for THCA may have been higher than the target value in Plant Sample 6 due to a majority of laboratories not thoroughly mixing the material prior to sampling, leaving behind the more dense, less extractable particles.

With the exception of Plant Sample 3 (marijuana, **Fig. 3-23**) and Plant Sample 4 (hemp, **Fig. 3-20**), the consensus ranges for THCA were within the target ranges. A higher percentage of laboratories reported quantitative results outside of the target range for Plant Sample 3 (78 %) and Plant Sample 4 (74 %) than for the other samples (between 1.4 % and 38 %). In comparison, NIST measurements for Plant Sample 3 and Plant Sample 4 had smaller variability than for the other samples. Even though the consensus bias for Plant Sample 3 and Plant Sample 4 was similar to the other samples, the consensus ranges fell outside the target ranges. Some participants did report sampling sizes < 0.5 g, which may have resulted in sample size dependent inhomogeneity. The NIST sample preparation, and thus the assigned values, were based on 0.5 g of sample and 40 mL of MeOH over a two-step extraction process (Section 2.2.1). The increased number of laboratories reporting outside the target range could be because they did not mix the sample adequately enough prior to sampling, which would have been especially important for Plant Sample 4, which had a broader range of particle sizes (250 µm to 710 µm) than the other NIST samples and a more complex material composition (buds, leaves, and stems).



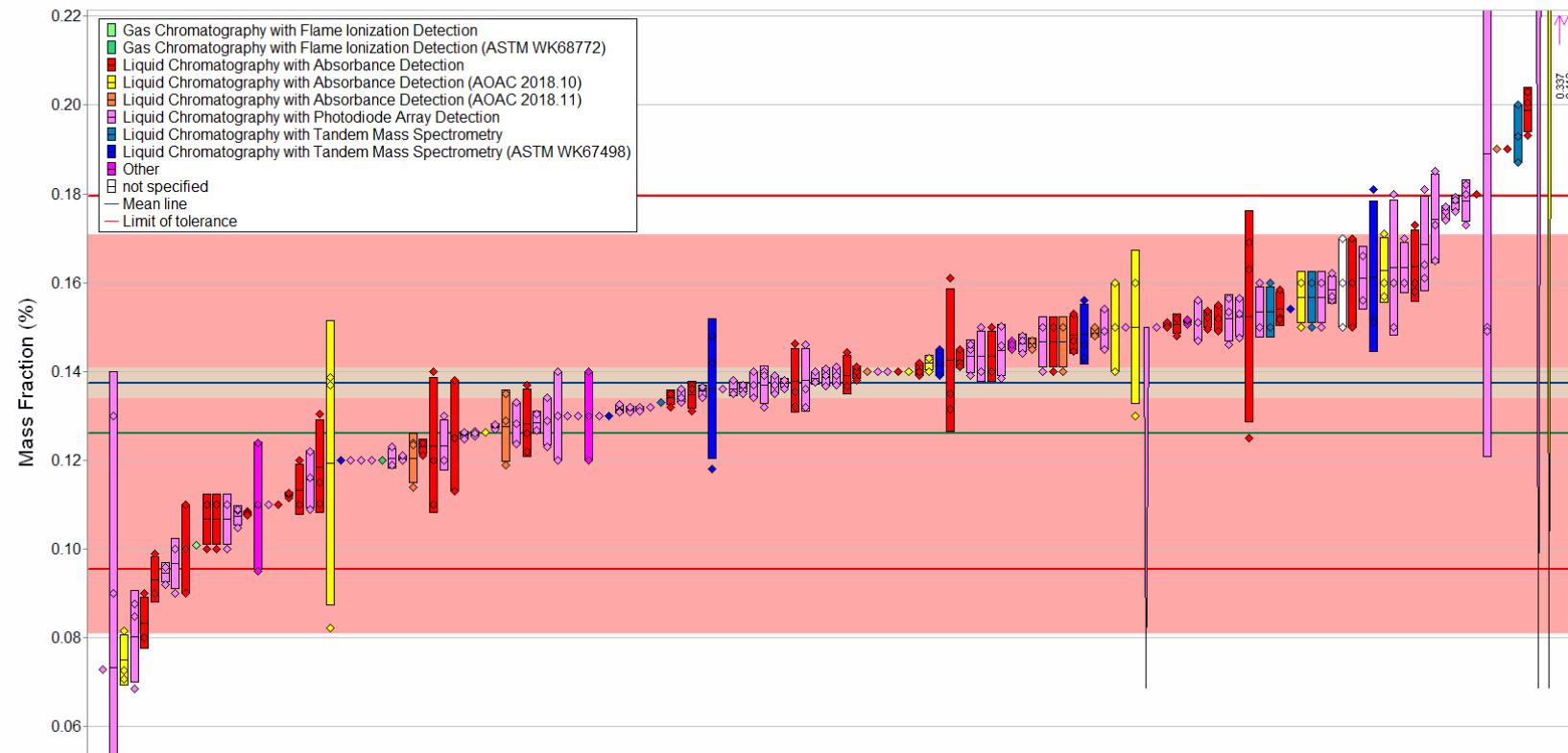
**Fig. 3-19. THCA in NRC HEMP-1 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



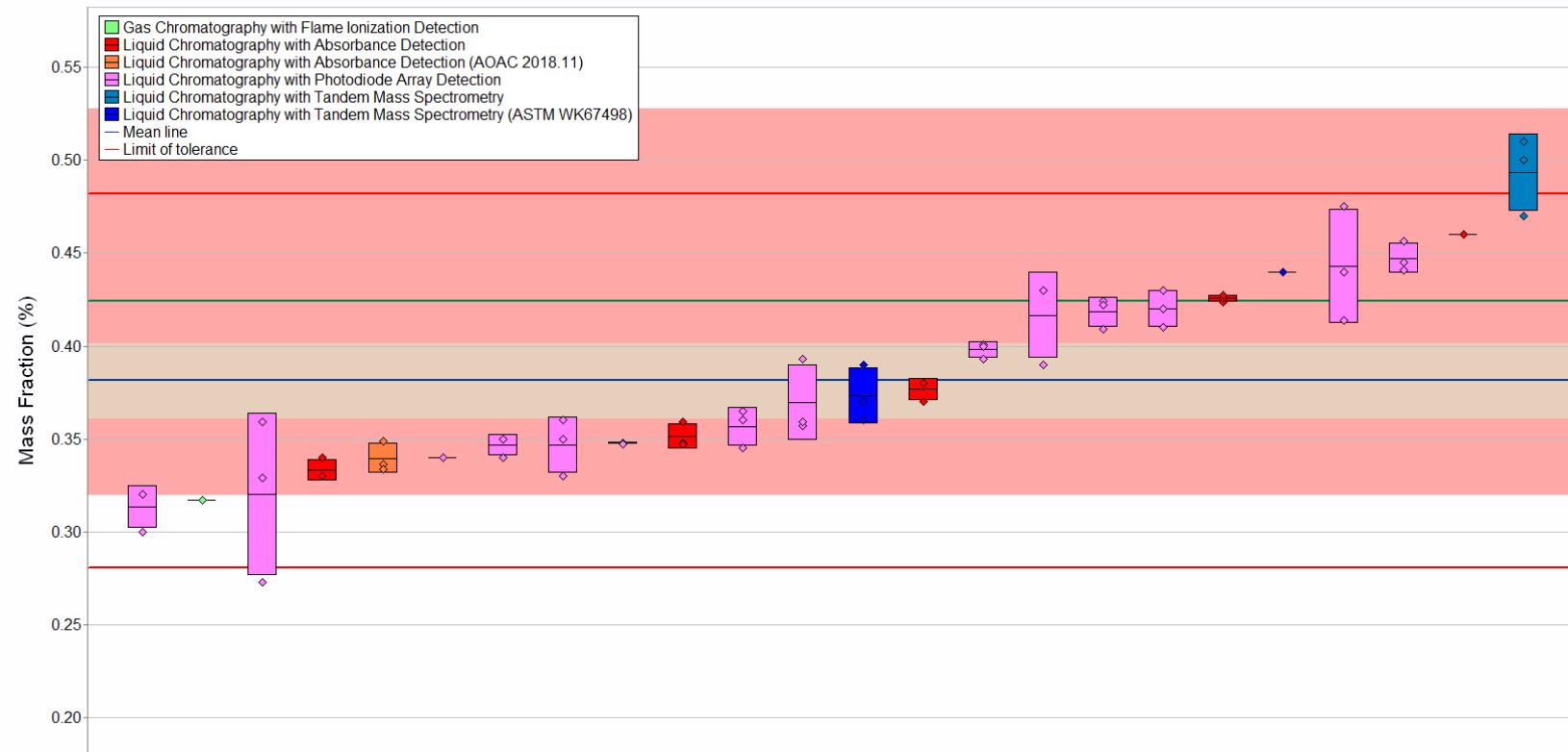
**Fig. 3-20. THCA in Plant Sample 4 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



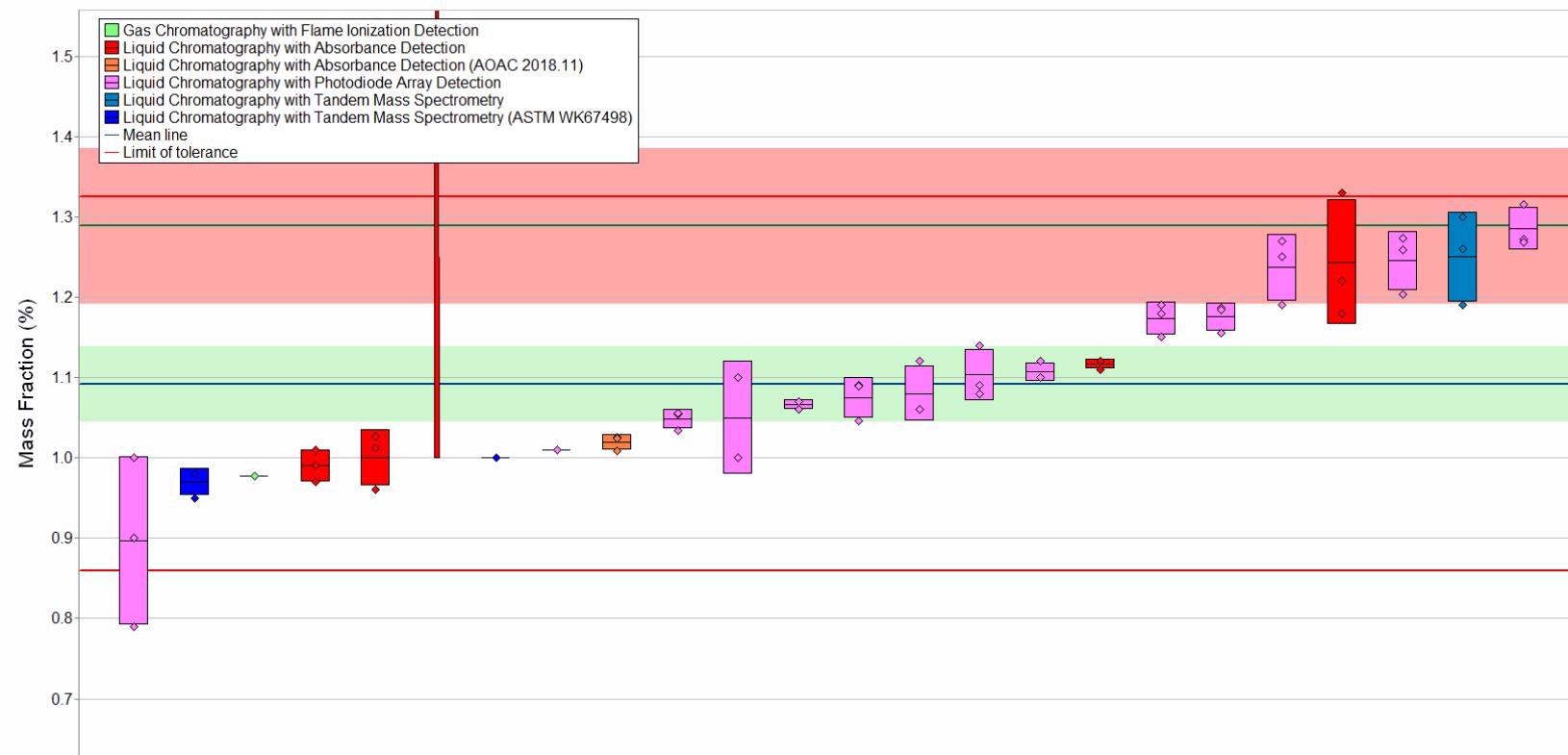
**Fig. 3-21. THCA in Plant Sample 6 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



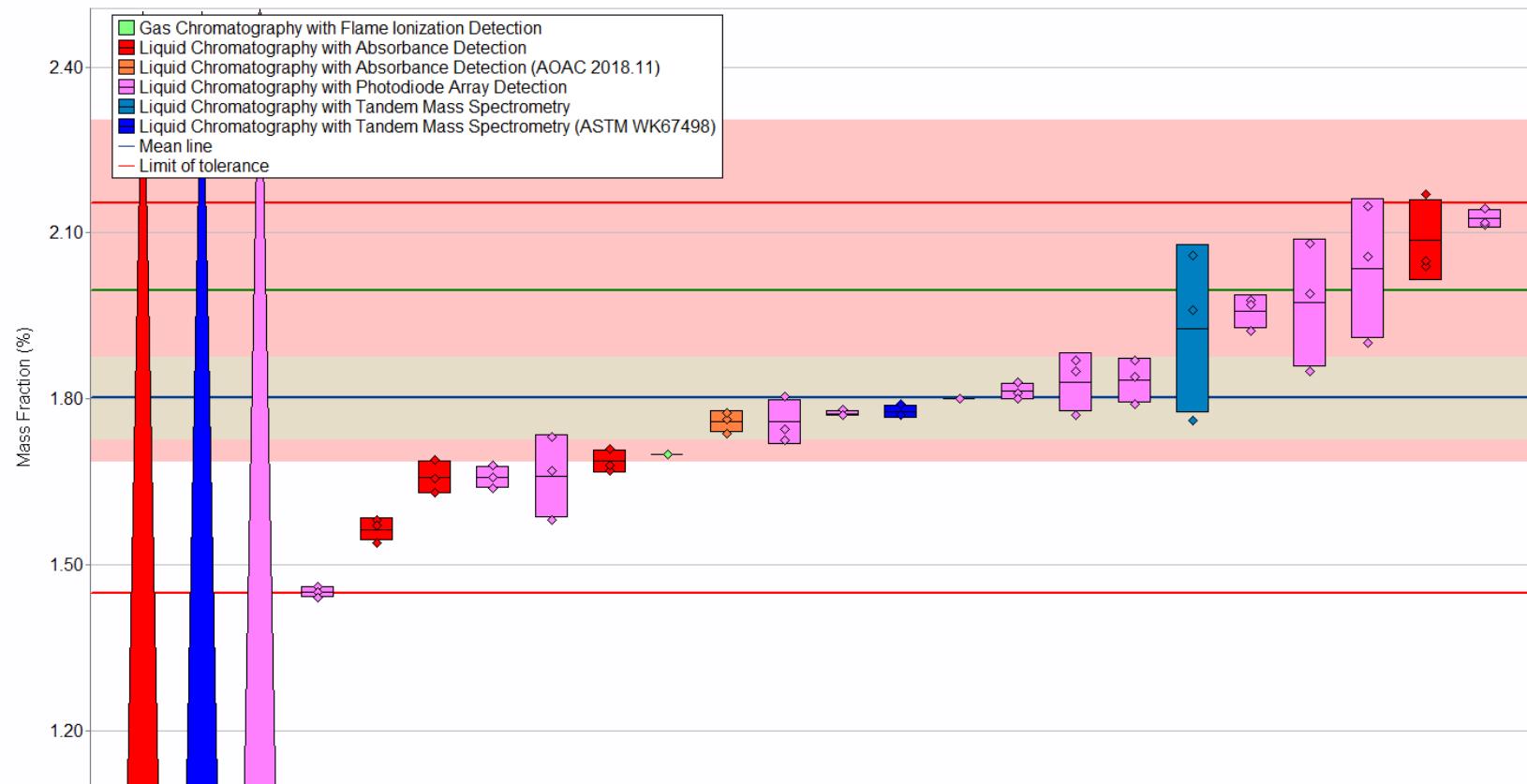
**Fig. 3-22. THCA in Plant Sample 2 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 3-23. THCA in Plant Sample 3 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 3-24. THCA in Plant Sample 5 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

### 3.4.2.3. Candidate Analytical Methods

NIST provided nine candidate standard methods for participants to use from AOAC International and ASTM International if an in-house analytical method was not available. Mass fractions were submitted by participating laboratories for THCA using an ASTM LC-MS/MS method and two AOAC LC-ABS methods, one of which included the option for MS detection. The within-(repeatability, %RSD<sub>r</sub>) and between-laboratory (reproducibility, %RSD<sub>R</sub>) variabilities are summarized for candidate method/sample pairs for which participants reported at least two independent measurements for a sample and at least five laboratories reported data (**Table 3-9**). Both AOAC methods were approved by an expert review panel using criteria established in SMPR 2017.002 for the quantitation of cannabinoids in dried cannabis plant samples [21], which requires %RSD<sub>r</sub> and %RSD<sub>R</sub> to be  $\leq 5\%$  and  $\leq 7\%$ , respectively, for THCA mass fractions between 0.1 % and 1 %. AOAC has since published SMPR 2019.003 for quantitation of cannabinoids in hemp plant samples [16], which requires %RSD<sub>r</sub> and %RSD<sub>R</sub> to be  $\leq 5\%$  and  $\leq 10\%$ , respectively, for THCA mass fractions between 0.05 % and 0.5 %. The requirements established in SMPR 2019.003 were used in this study for the AOAC and ASTM methods.

**Table 3-9. Within- and between-laboratory variabilities for THCA measurements using candidate standardized analytical methods.**

	<u>n<sup>a</sup></u>	NRC HEMP-1		Plant Sample 4			Plant Sample 6		
		%RSD <sub>r</sub>	%RSD <sub>R</sub>	<u>n<sup>a</sup></u>	%RSD <sub>r</sub>	%RSD <sub>R</sub>	<u>n<sup>a</sup></u>	%RSD <sub>r</sub>	%RSD <sub>R</sub>
<u>LC-Absorbance</u>									
AOAC 2018.10	10	1.6	21.4	10	4.8	16.2	9	5.8	16.5
AOAC 2018.11	8	2.5	18.8	8	4.3	10.1	8	4.7	13.6
<u>LC-MS/MS</u>									
ASTM WK67498	5	6.4	39.3	5	6.2	22.1	5	5.1	9.5

<sup>a</sup> n = number of laboratories

#### AOAC 2018.10

The %RSD<sub>r</sub> published for AOAC 2018.10 was 3.29 % for dried flowers containing 0.11 % THCA ( $n = 4$ , [22]), which was within the published requirement of  $\leq 5\%$  [21]. The average within-laboratory variability observed for the participants reporting use of AOAC 2018.10 was within the %RSD<sub>r</sub> published in the method and SMPR [16] for NRC HEMP-1 and Plant Sample 4, while reporting marginally outside of the recommended variability for Plant Sample 6. The THCA mass fractions in all plant samples were above the stated method LOQ (0.04 %) and laboratories should have achieved  $\leq 5\%$  RSD<sub>r</sub> for all samples. The THCA values reported by laboratories using AOAC 2018.10 were lower than the target value for NRC HEMP-1 (6 %) and Plant Sample 4 (8 %), and higher than the target value for Plant Sample 6 (10 %). The between-laboratory variabilities observed for THCA measured using AOAC 2018.10 in the hemp samples were above the 10 % requirement for %RSD<sub>R</sub> [16]. The observed reproducibility of AOAC 2018.10 had not been published at the time of this report.

### AOAC 2018.11

The %RSD<sub>r</sub> published for AOAC 2018.11 was reported from two separate analysts. The combined %RSD<sub>r</sub> was 1.3 % for dried plant material samples containing 0.085 % THCA ( $n = 10$ , [7]), which was within the published requirement of  $\leq 5\%$  [21]. The average within-laboratory variability observed for the participants reporting use of AOAC 2018.11 was consistent with the %RSD<sub>r</sub> published in the method and the SMPR [16]. The THCA mass fractions in all samples were above the published LOQ in AOAC 2018.11 for THCA in cannabis dried plant material (0.007 %). Laboratories using AOAC 2018.11 reported THCA values that were lower than the target value for NRC HEMP-1 (6 %) and Plant Sample 4 (7 %) and higher than the target value for Plant Sample 6 (16 %). The %RSD<sub>R</sub> observed for THCA measured using AOAC 2018.11 in the hemp samples were above the 10 % requirement for NRC HEMP-1 and Plant Sample 4 [16]. The observed reproducibility of AOAC 2018.11 had not been published at the time of this report.

### ASTM WK67498

The ASTM WK67498 method was developed following the AOAC [23] and ASTM [24] guidelines. The %RSD<sub>r</sub> published for ASTM WK67498 was between 1.7 % and 7.5 % based on measurements of 5 lots of the same hemp sample containing between 0.0968 % and 0.1262 % THCA ( $n = 3$ , [25]). The average within-laboratory variability observed for the participants reporting use of ASTM WK67498 was consistent with the %RSD<sub>r</sub> published in the method. However, the performance of ASTM WK67498 when compared to AOAC SMPR [16] does not meet the %RSD<sub>r</sub> requirement of  $\leq 5\%$  in one sample tested by the method authors [25] or in any samples in this study. Laboratories using ASTM WK67498 reported THCA values that were lower than the target value for NRC HEMP-1 (4 %), Plant Sample 4 (5 %), and higher than the target value for Plant Sample 6 (13 %). Observed reproducibility of ASTM WK67498 has not been published at the time of this report; however, the between-laboratory variabilities observed for THCA measured in the hemp samples were above the 10 % requirement published by AOAC [16].

### 3.4.3. Total Δ<sup>9</sup>-THC

#### 3.4.3.1. Within- and Between Laboratory Precision

Laboratories reporting quantitative results for total Δ<sup>9</sup>-THC in all plant samples demonstrated within-laboratory variability ( $\leq 4.5\%$ ) at or below the published expectation of  $\leq 5\%$  for cannabinoids in hemp [16], with higher variability reported for the hemp samples than the marijuana samples (**Table 3-6**). The majority of laboratories that analyzed total Δ<sup>9</sup>-THC in hemp (82 %) and marijuana (80 %) reported using LC. Unless a laboratory decarboxylated the THCA in the samples prior to analysis with LC, the total Δ<sup>9</sup>-THC values reported were based on calculation. For this reason, the within-laboratory variabilities for laboratories that used LC are not described further because they represent a weighted average of the Δ<sup>9</sup>-THC and THCA measurement variability discussed in the previous sections. If laboratories using LC and not decarboxylating THCA prior to analysis are required to report total Δ<sup>9</sup>-THC, the accuracy of the total Δ<sup>9</sup>-THC determination will depend entirely on the capability of the laboratory to accurately measure Δ<sup>9</sup>-THC and THCA.

The majority of laboratories that reported using gas chromatography (GC) to measure total  $\Delta^9$ -THC demonstrated repeatability at or below 5 % for NRC HEMP-1 (79 %), Plant Sample 4 (73 %), and Plant Sample 6 (86 %). Within-laboratory variabilities for total  $\Delta^9$ -THC values measured using GC in Plant Sample 2 (89 %), Plant Sample 3 (75 %), and Plant Sample 5 (75 %) were similar to the variabilities seen for measurements in the hemp samples. Higher precision ( $RSD_r \leq 3\%$ ) is recommended for plant samples with mass fractions of total  $\Delta^9$ -THC between 0.5 % and 5 % [16], which is applicable for Plant Sample 3 and Plant Sample 5.

The between-laboratory variabilities for total  $\Delta^9$ -THC ranged from 10.3 % to 30.9 % for all reported results. Laboratories that reported using GC to measure total  $\Delta^9$ -THC had between-laboratory variabilities ranging from 10.3 % to 45.5 %. Regardless of the analytical method used to assess total  $\Delta^9$ -THC mass fractions in the samples, all between-laboratory variabilities were outside the performance requirements [16]. However, between-laboratory precision is a metric intended to be more representative of the variability of multiple laboratories using a single analytical method and not multiple laboratories using multiple analytical methods. The between-laboratory variability for total  $\Delta^9$ -THC in plant samples for this exercise are comparable to the UK-PT program overall analyte relative standard deviations (10.6 % to 42.3 %) for total  $\Delta^9$ -THC in hemp [19].

In general, the between-laboratory variability increased with a decrease in total  $\Delta^9$ -THC mass fraction. Comparing results reported for total  $\Delta^9$ -THC in NRC HEMP-1 and Plant Sample 6, the majority of values cluster in an ellipse, indicating systematic errors such as calibration issues were prevalent for the two lowest mass fraction samples (**Fig. 3-25**). A similar trend was observed when comparing results reported for Plant Sample 3 and Plant Sample 5 (**Fig. 3-26**). These trends are consistent with the errors noted for the  $\Delta^9$ -THC and THCA measurements, which was expected considering the majority of laboratories calculated results for total  $\Delta^9$ -THC using  $\Delta^9$ -THC and THCA results. Potential sources of this type of calibration issue may be in the inaccurate assignment of calibrant purity (e.g., not considering potential impurities or moisture in the calibrant material) or extension of the calibration curve beyond the linear range. One approach to improve the calibration range is to conduct a screening experiment on the samples ahead of analysis to determine the most appropriate calibration curve for the sample. Prior to subsequent measurements, additional calibrant solutions may be prepared in that calibration range and other points can be excluded from the determination of the calibration curve to prevent bias.

Random errors likely occurred for laboratories with values outside the consensus and target ranges for total  $\Delta^9$ -THC in NRC HEMP-1 and Plant Sample 6 (**Fig. 3-25**). Laboratory B197 used infrared spectroscopy to determine total  $\Delta^9$ -THC and reported values approximately two to six times higher than the target values for total  $\Delta^9$ -THC in the three hemp samples. The total  $\Delta^9$ -THC values reported for all three hemp samples were similar ( $\approx 0.6\%$ ), which could indicate that the instrument could not accurately quantify total  $\Delta^9$ -THC values below 0.6 %. Laboratories must understand the limitations of the instruments being used so that those limits can be accurately reported. Laboratories B016, B053, and B164 all used LC methods to determine total  $\Delta^9$ -THC, indicating that the value was based on their  $\Delta^9$ -THC and THCA values. Each of the three labs recorded  $\Delta^9$ -THC and/or THCA values outside the consensus and target ranges for NRC HEMP-1 and Plant Sample 6, resulting in a high total  $\Delta^9$ -THC value. Additionally, calculations for total

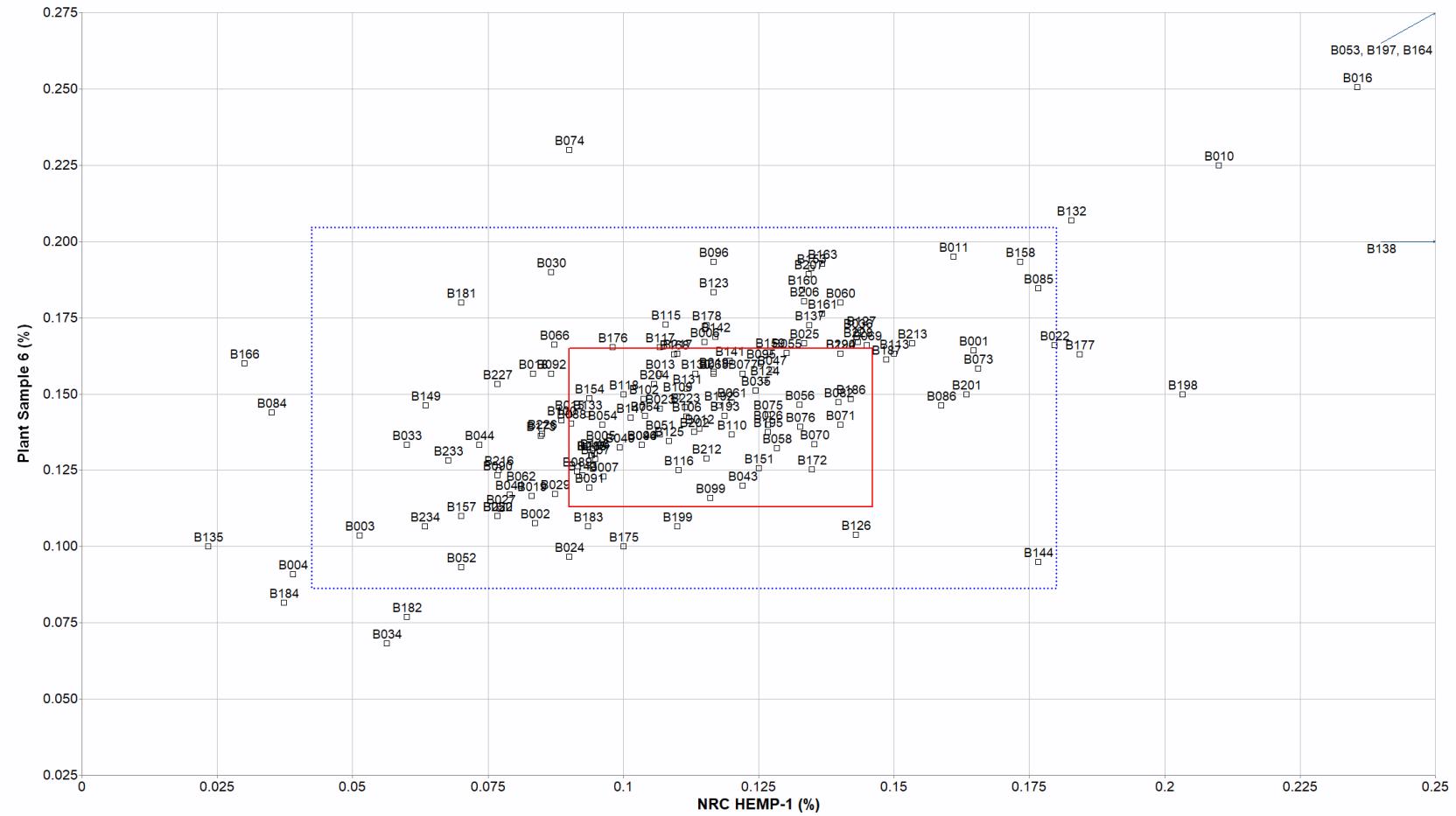
$\Delta^9$ -THC values should always be checked to ensure that Equation (8) is being properly utilized to convert the measured THCA value to  $\Delta^9$ -THC equivalents.

### 3.4.3.2. Accuracy

The individual participant, consensus, and target mass fraction results are presented in a tabular form in **Appendix B** and graphically in **Fig. 3-27** through **Fig. 3-34**. The majority of laboratories (75 %) reporting total  $\Delta^9$ -THC values in the hemp samples used either LC-ABS or LC-PDA as their analytical method. In contrast, laboratories measuring total  $\Delta^9$ -THC in marijuana reported use of both GC methods (40 %) and LC-ABS or LC-PDA (52 %). The consensus values for total  $\Delta^9$ -THC were between 6 % (NRC HEMP-1) and 13 % (Plant Sample 3) lower than the target values for all but Plant Sample 6, which had a consensus value approximately 4 % higher than the target value. The accuracy of the total  $\Delta^9$ -THC values reported by laboratories using LC methods was directly correlated to their ability to accurately quantify  $\Delta^9$ -THC and THCA. The overall trends in accuracy for total  $\Delta^9$ -THC measurements are similar to those for THCA measurements because THCA contributed more to the calculation of total  $\Delta^9$ -THC values than  $\Delta^9$ -THC.

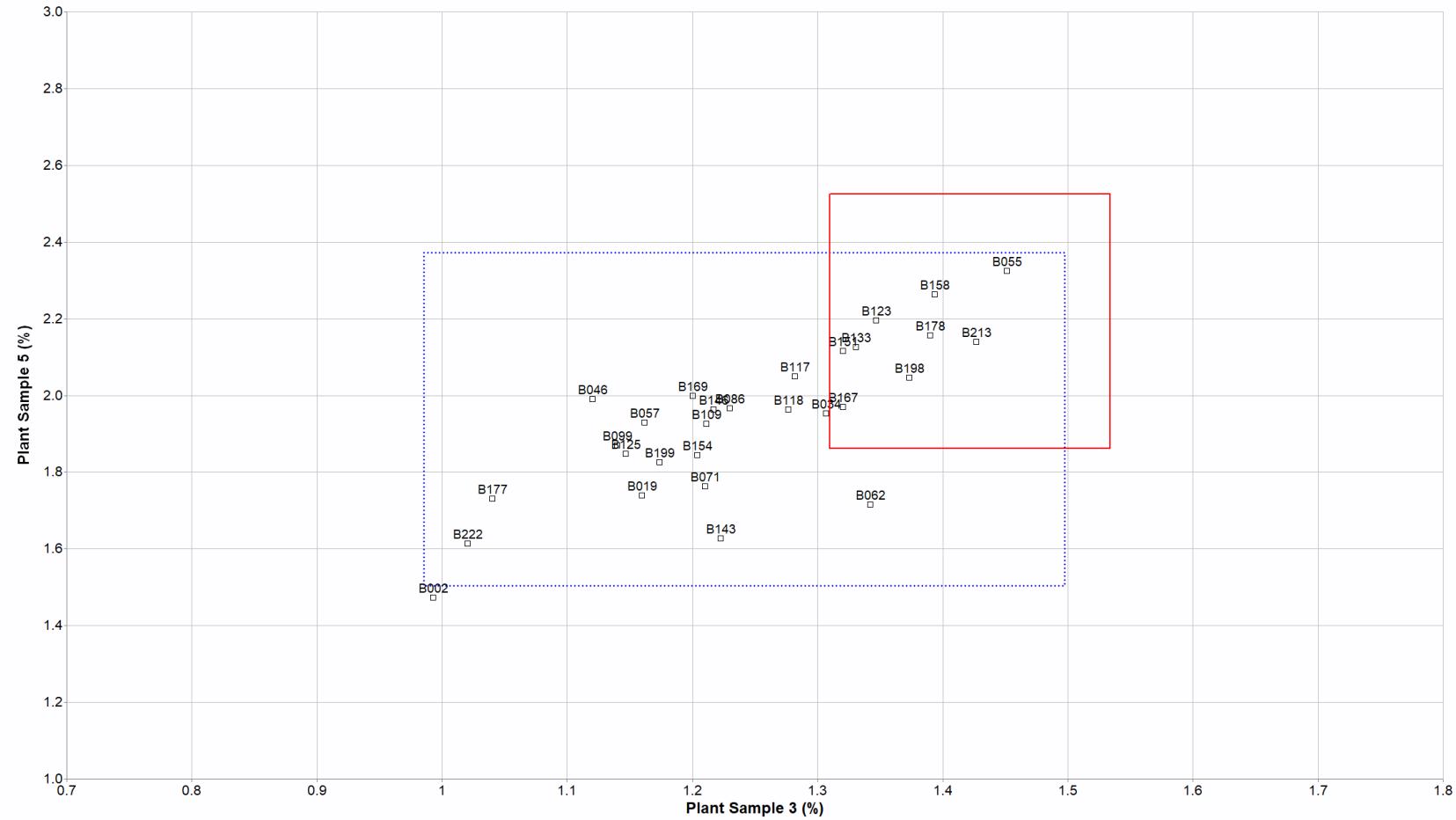
Similar to the overall data for total  $\Delta^9$ -THC, the values for total  $\Delta^9$ -THC measured using the GC methods were below the target value with the exception of NRC-HEMP 1 and Plant Sample 6. GC methods are expected to underestimate the total  $\Delta^9$ -THC concentration due to the incomplete decarboxylation of THCA in the GC inlet [26]. The GC-based total  $\Delta^9$ -THC values for NRC HEMP-1 and Plant Sample 6 were above the target value likely due to calibration bias at the lower end of the calibration curve.

With the exception of Plant Sample 3 (marijuana, **Fig. 3-32**) and Plant Sample 4 (hemp, **Fig. 3-28**), the consensus ranges for total  $\Delta^9$ -THC were within the target ranges similar to THCA. The GC-derived consensus ranges were closer to the target ranges for Plant Sample 3 (**Fig. 3-33**) and Plant Sample 4 (**Fig. 3-29**) than the overall consensus ranges because the between-laboratory variability for laboratories using GC methods was higher.



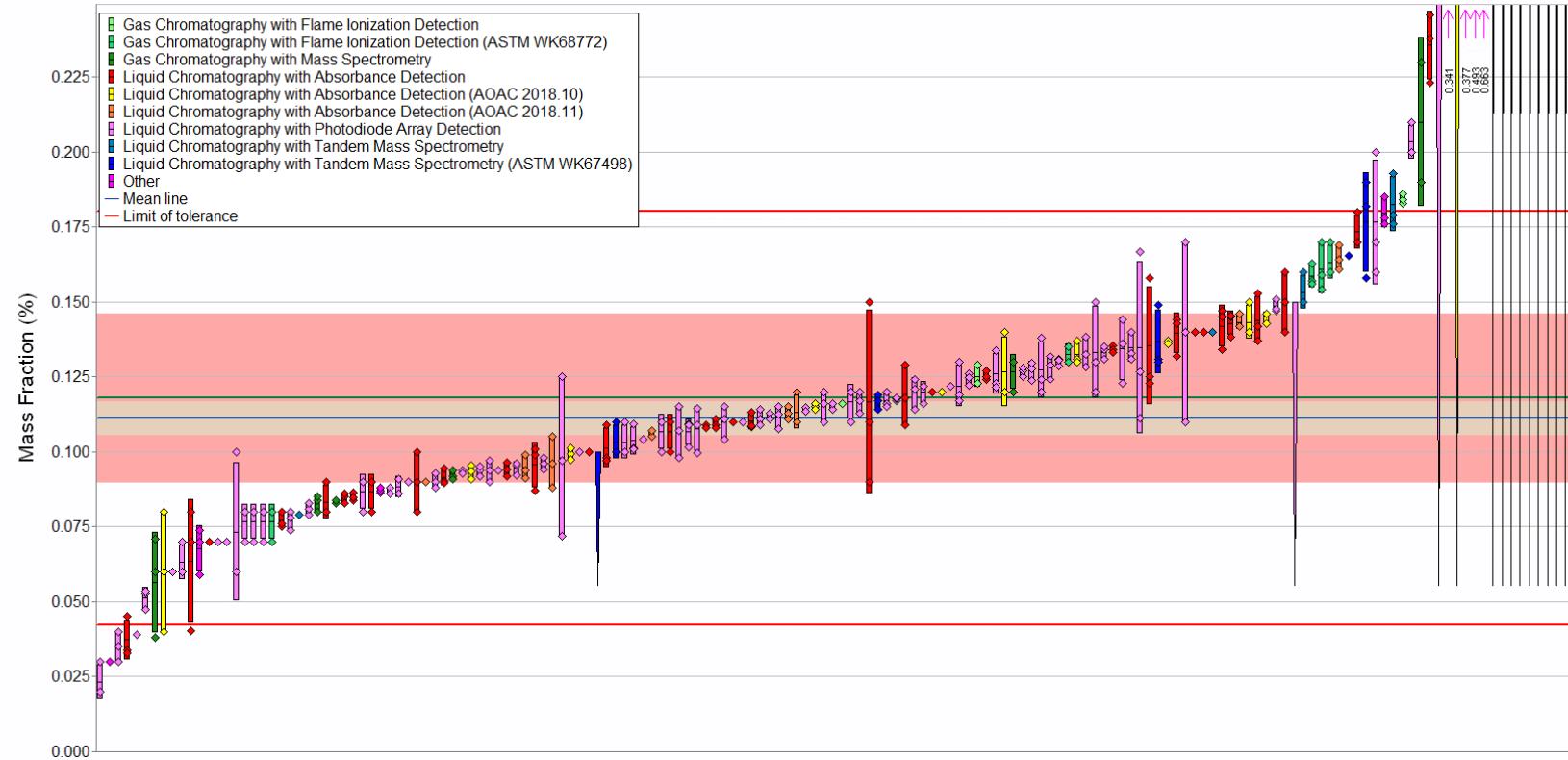
**Fig. 3-25. Laboratory means for total  $\Delta^9$ -THC in NRC HEMP-1 and Plant Sample 6 (sample/sample comparison view).**

In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (Plant Sample 6). The solid red box represents the target range of tolerance for the two samples, NRC HEMP-1 (x-axis) and Plant Sample 6 (y-axis), which encompasses the target values bounded by their uncertainties ( $U_{NIST}$ ) and represents the range that results in an acceptable  $Z_{NIST}$  score,  $|Z_{NIST}| \leq 2$ . The dotted blue box represents the consensus range of tolerance for NRC HEMP-1 (x-axis) and Plant Sample 6 (y-axis), calculated as the values above and below the consensus means that result in an acceptable  $Z'_{comm}$  score,  $|Z'_{comm}| \leq 2$ .



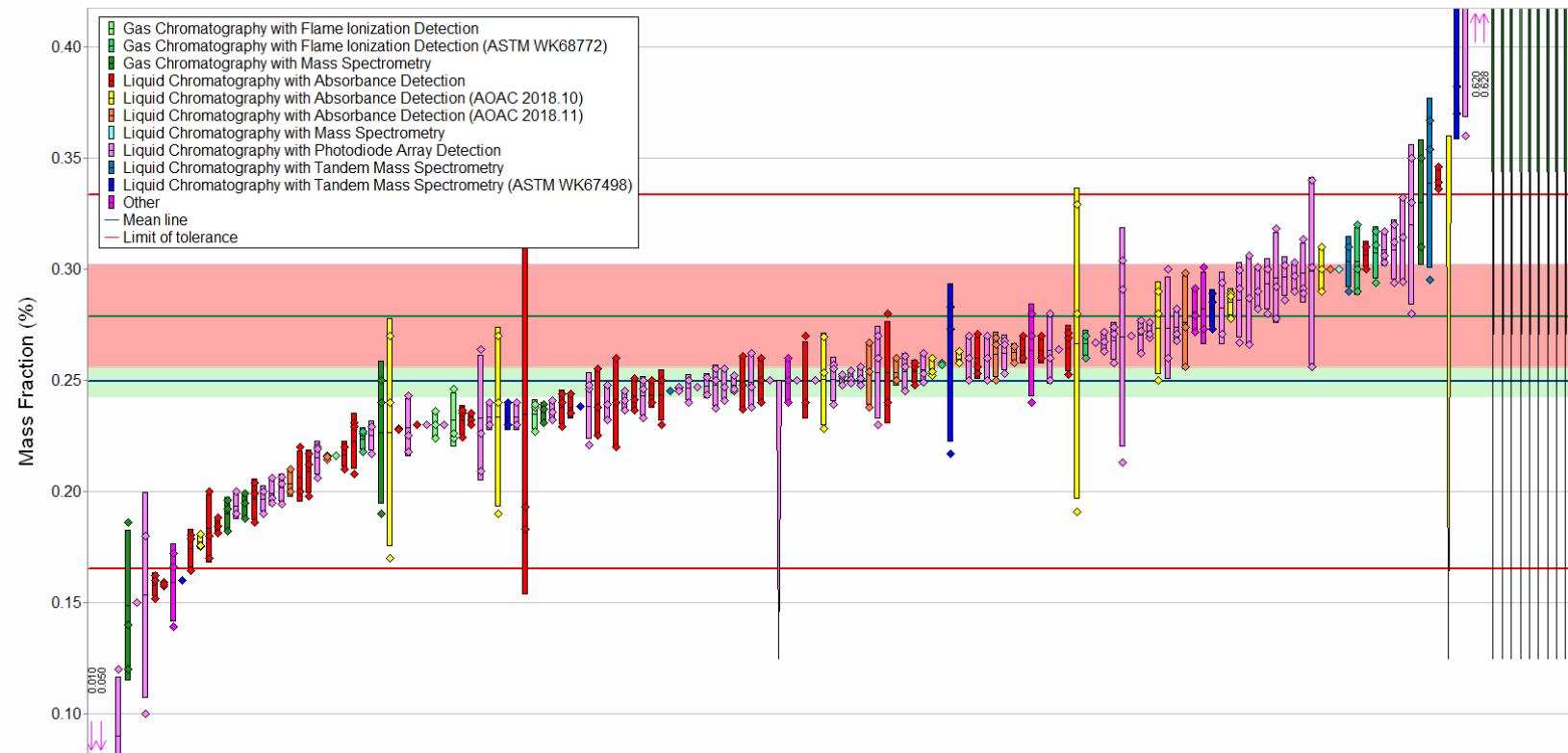
**Fig. 3-26. Laboratory means for total  $\Delta^9$ -THC in Plant Sample 3 and Plant Sample 5 (sample/sample comparison view).**

In this view, the individual laboratory mean for one sample (Plant Sample 3) is compared to the individual laboratory mean for a second sample (Plant Sample 5). The solid red box represents the target range of tolerance for the two samples, Plant Sample 3 (x-axis) and Plant Sample 5 (y-axis), which encompasses the target values bounded by their uncertainties ( $U_{NIST}$ ) and represents the range that results in an acceptable  $Z_{NIST}$  score,  $|Z_{NIST}| \leq 2$ . The dotted blue box represents the consensus range of tolerance for Plant Sample 3 (x-axis) and Plant Sample 5 (y-axis), calculated as the values above and below the consensus means that result in an acceptable  $Z'_{comm}$  score,  $|Z'_{comm}| \leq 2$ .



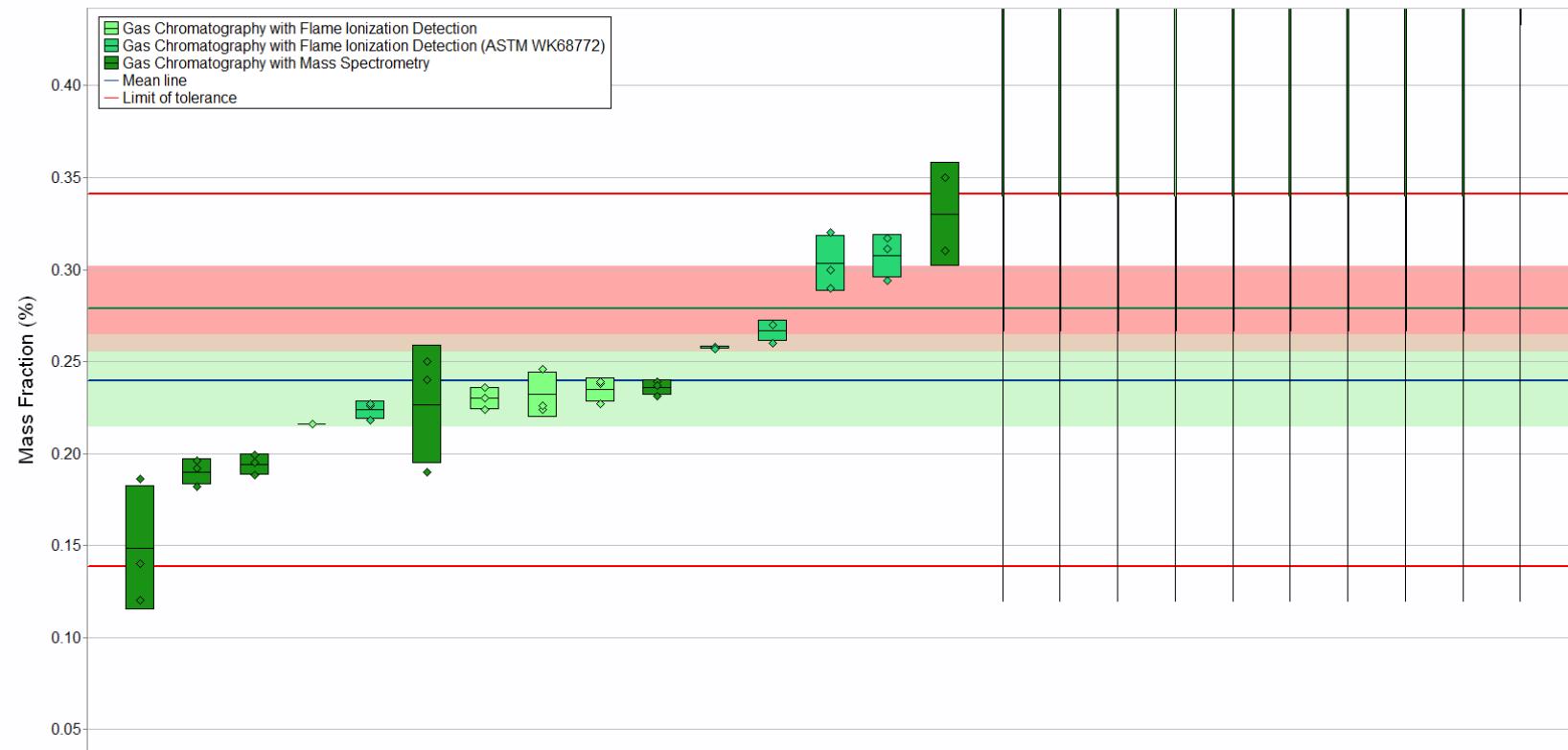
**Fig. 3-27. Total  $\Delta^9$ -THC in NRC HEMP-1 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



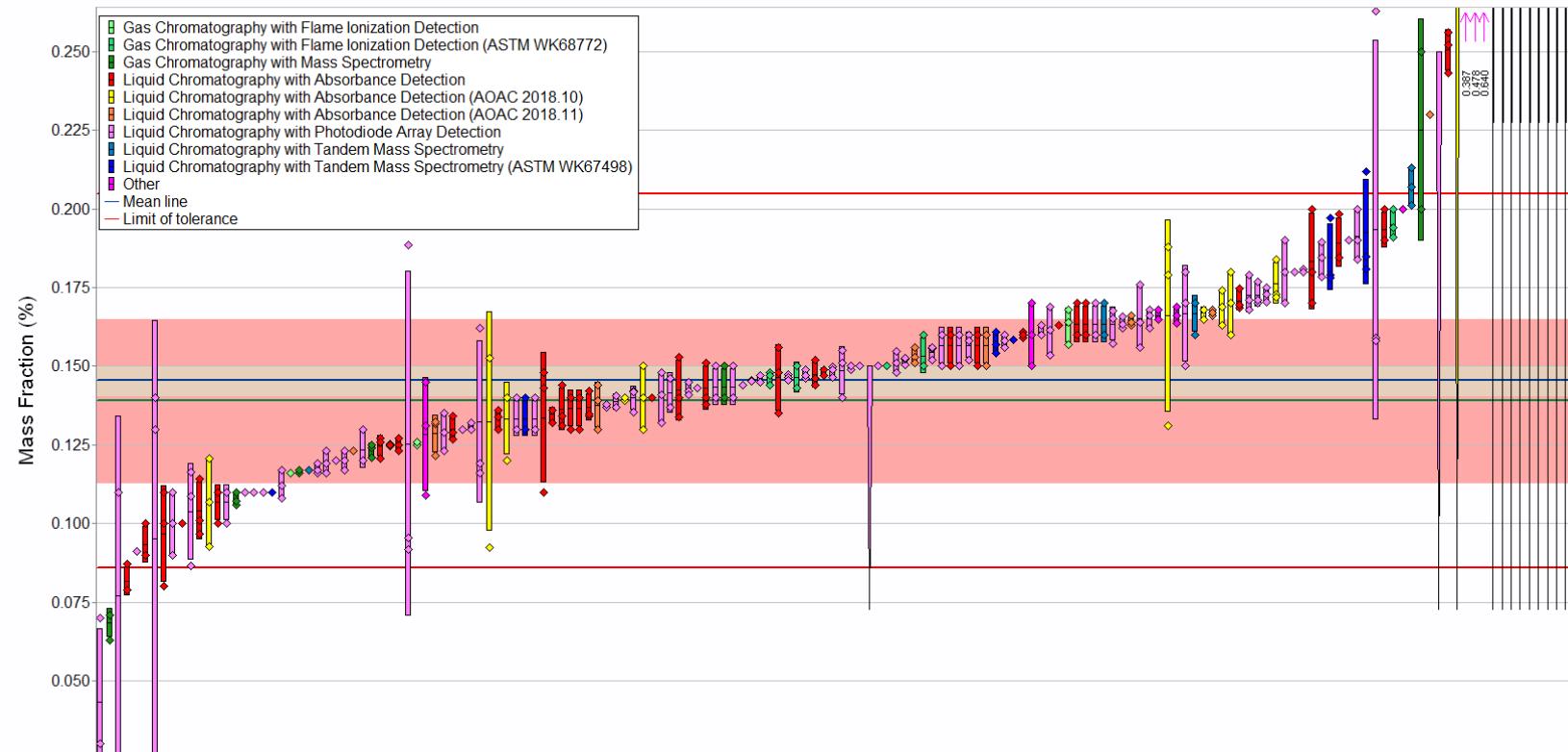
**Fig. 3-28. Total  $\Delta^9$ -THC in Plant Sample 4 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{comm}$  score,  $|Z'_{comm}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{NIST}$ ) and represents the range that results in an acceptable  $Z_{NIST}$  score,  $|Z_{NIST}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



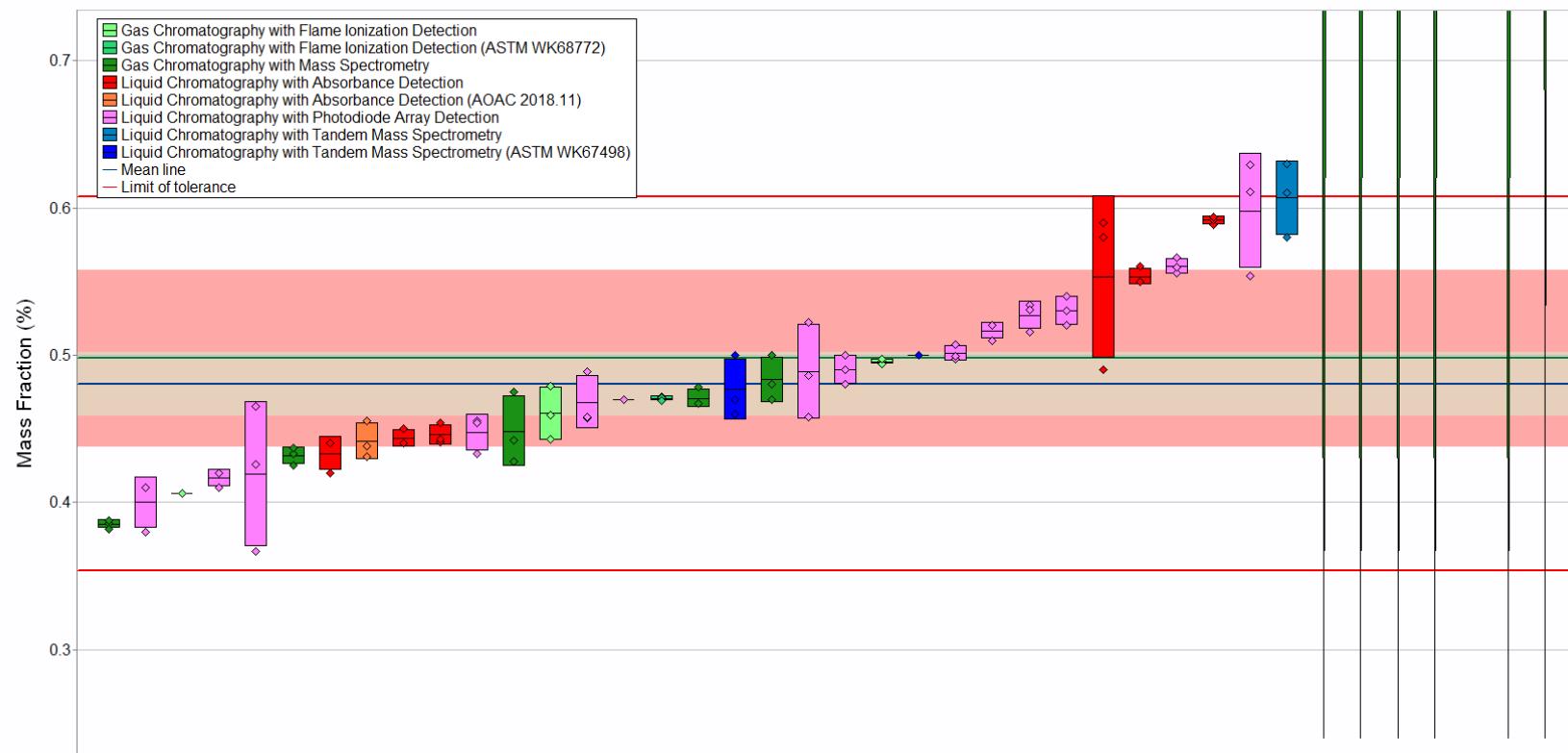
**Fig. 3-29. Total  $\Delta^9\text{-THC}$  in Plant Sample 4 (data summary view – GC methods).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



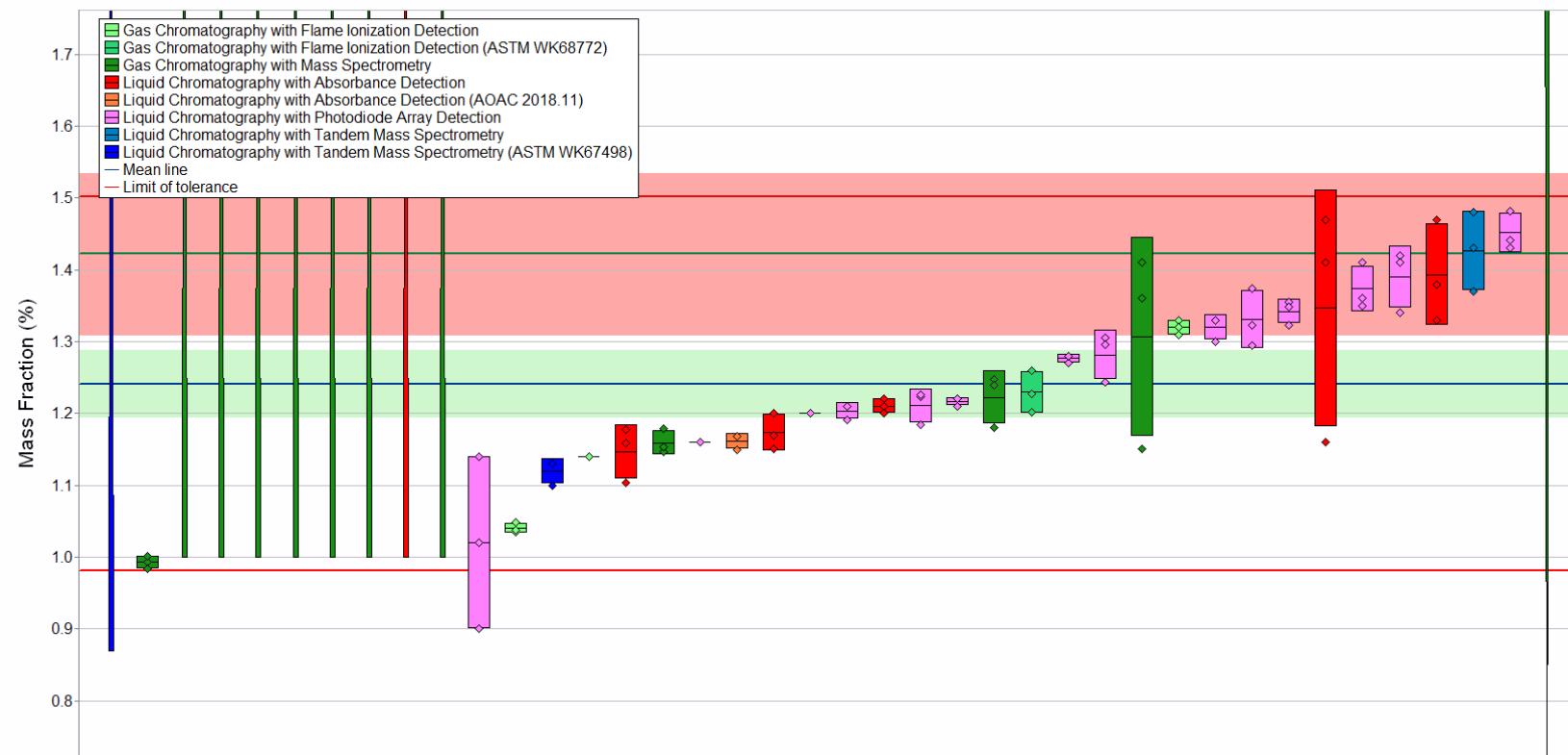
**Fig. 3-30. Total  $\Delta^9\text{-THC}$  in Plant Sample 6 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 3-31. Total  $\Delta^9$ -THC in Plant Sample 2 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 3-32. Total  $\Delta^9$ -THC in Plant Sample 3 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

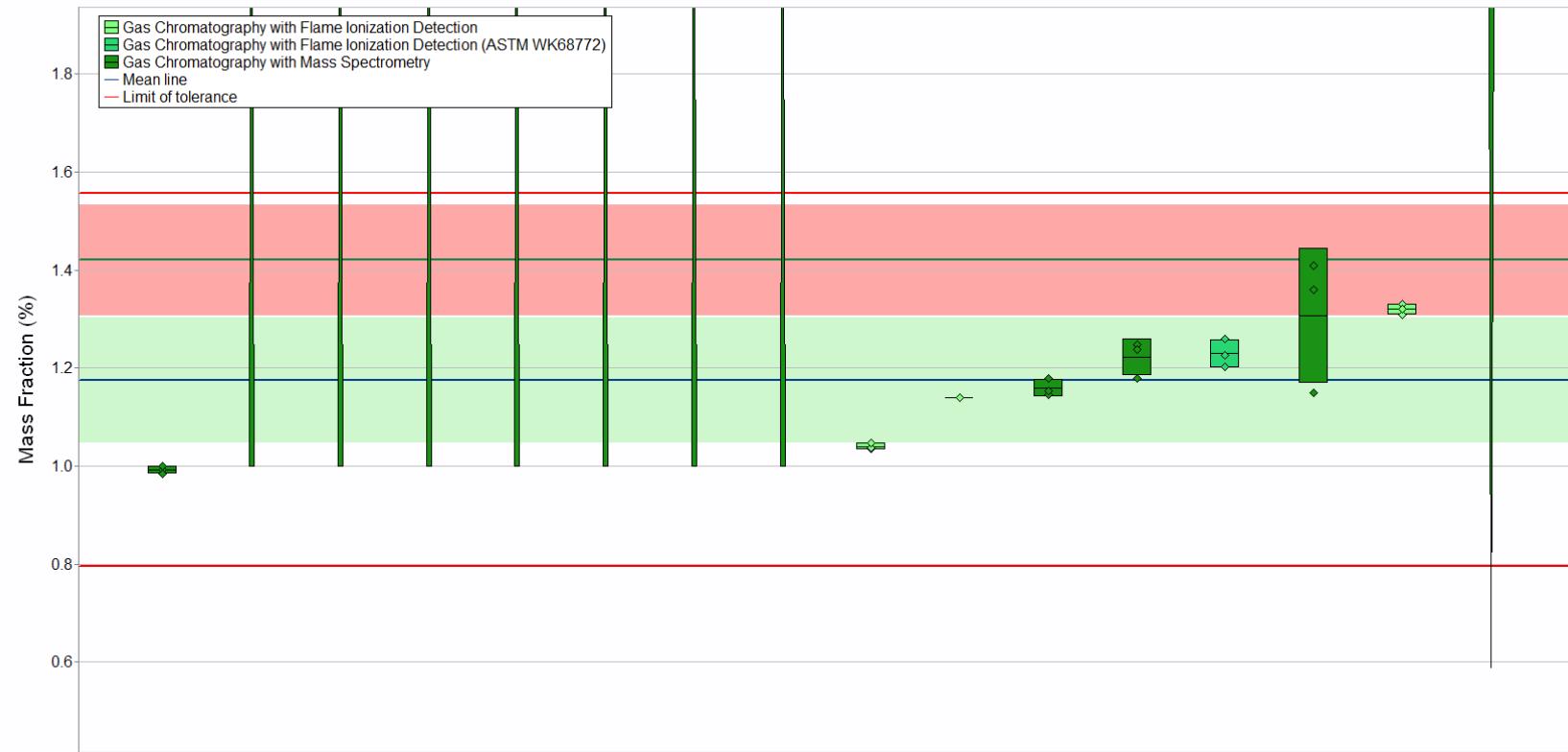
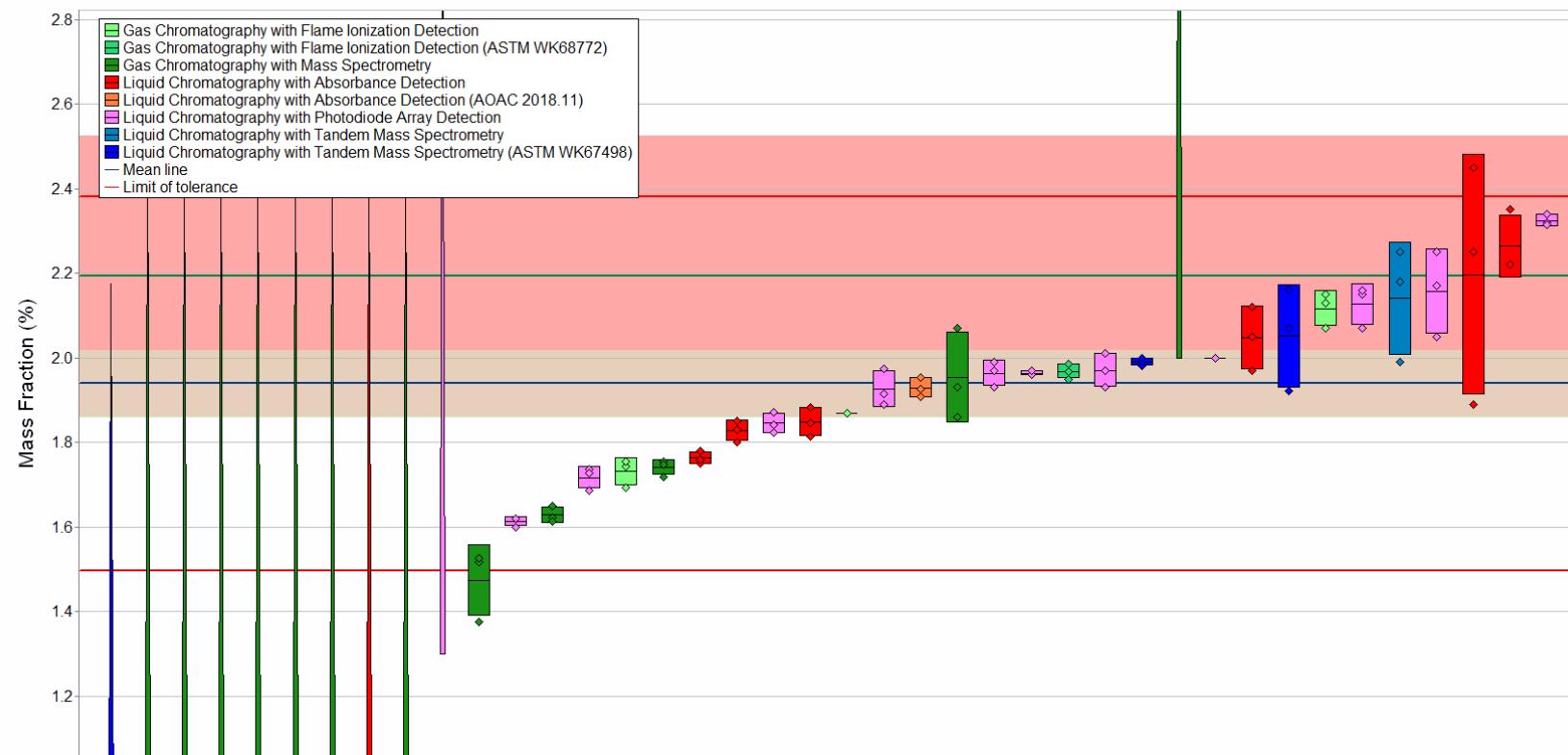


Fig. 3-33. Total  $\Delta^9$ -THC in Plant Sample 3 (data summary view – GC methods).

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



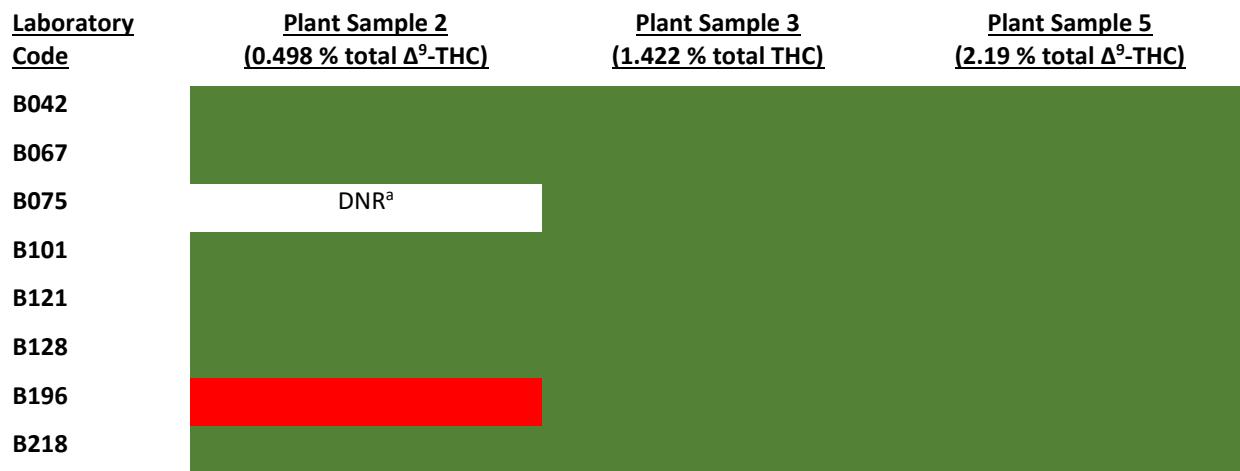
**Fig. 3-34. Total  $\Delta^9$ -THC in Plant Sample 5 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

Laboratories reporting qualitative results for the marijuana samples reported either a LOQ or a decision point threshold. The LOQ and decision point thresholds reported correspond with various jurisdictional legal definitions of hemp (1 % to 2 % total  $\Delta^9$ -THC) and are being used to differentiate between hemp and marijuana. **Table 3-10** illustrates the ability of the laboratories using LOQs or threshold values to correctly identify a plant sample from this study as hemp or marijuana based on their reported limits. Because the reporting limits used for laboratories that participated in this study were all greater than the 0.3 % total  $\Delta^9$ -THC federal limit, not all laboratories identified Plant Sample 2 to be marijuana. In the case of laboratory B196 for Plant Sample 2, the threshold value was 1 % total  $\Delta^9$ -THC, which was an overestimate of the total  $\Delta^9$ -THC in Plant Sample 2. For Plant Sample 3 and Plant Sample 5, all laboratories correctly captured the total  $\Delta^9$ -THC values.

**Table 3-10. Reporting for total  $\Delta^9$ -THC based on qualitative assessments.**

Red indicates that the laboratory incorrectly captured the percentage of total  $\Delta^9$ -THC in their method thresholds.  
Green indicates that laboratory correctly captured the percentage of total  $\Delta^9$ -THC in their method thresholds.



<sup>a</sup> DNM = did not report a result for this plant sample

### 3.4.3.3. Candidate Analytical Methods

NIST provided a list of nine candidate standard methods from AOAC International and ASTM International for participants to use if an in-house analytical method was not available. For total  $\Delta^9$ -THC, only candidate methods using GC without derivatization were reviewed. The within-(repeatability, %RSD<sub>r</sub>) and between-laboratory (reproducibility, %RSD<sub>R</sub>) variabilities are summarized for candidate method/sample pairs for which participants reported at least two independent measurements for a sample and at least five laboratories reported data (**Table 3-11**). The requirements established in SMPR 2019.003 will be used in this study for ASTM WK68772 GC-FID as performance requirements had not been previously established by ASTM.

**Table 3-11. Within- and between-laboratory variabilities for total Δ<sup>9</sup>-THC measurements using candidate standardized analytical methods.**

GC-FID	NRC HEMP-1			Plant Sample 4			Plant Sample 6		
	<u>n<sup>a</sup></u>	<u>%RSD<sub>r</sub></u>	<u>%RSD<sub>R</sub></u>	<u>n<sup>a</sup></u>	<u>%RSD<sub>r</sub></u>	<u>%RSD<sub>R</sub></u>	<u>n<sup>a</sup></u>	<u>%RSD<sub>r</sub></u>	<u>%RSD<sub>R</sub></u>
ASTM WK68772	5	4.4	19.6	5	3.3	22.9	5	3.4	5.6

<sup>a</sup> n = number of laboratories

#### ASTM WK68772

No method performance data was published for any of the analytes detailed in ASTM WK68772. The average within-laboratory variability observed for participants reporting use of ASTM WK68772 for total Δ<sup>9</sup>-THC measurement in all three hemp samples was within the %RSD<sub>r</sub> published in the SMPR [16], with only one participant reporting within-laboratory variability over 5 % for NRC HEMP-1. The total Δ<sup>9</sup>-THC values reported by laboratories using ASTM WK68772 were higher than the target value for NRC HEMP-1 (21 %) and Plant Sample 6 (7 %), and lower than the target value for Plant Sample 4 (3 %). The between-laboratory variabilities observed for total Δ<sup>9</sup>-THC measured using ASTM WK68772 in Plant Sample 6 were within the 10 % requirement and were above the 10 % requirement in NRC HEMP-1 and Plant Sample 4 % [16]; observed reproducibility of ASTM WK68772 had not been published at the time of this report.

#### 3.4.3.4. Examples and Recommendations

Quantitative analysis of total Δ<sup>9</sup>-THC by GC is challenging because of the heat-induced decarboxylation of THCA to Δ<sup>9</sup>-THC [27, 28, 29]. THCA converts to Δ<sup>9</sup>-THC after injection into the GC inlet because of the high temperatures, resulting in a single chromatographic peak corresponding to total Δ<sup>9</sup>-THC. Quantitative measurements of total Δ<sup>9</sup>-THC using GC are known to be inaccurate because the conversion is incomplete. The primary method for avoiding THCA decarboxylation in the GC inlet is derivatization to replace the carboxylic acid group on THCA with a more stable silicon-based group permitting separate quantification of both THCA and Δ<sup>9</sup>-THC [30, 31, 32]. Use of multiple derivatizing agents has been reported; however, the increased sample preparation time and more complex mass spectral interpretation limits its applicability to laboratories with high-throughput requirements for cannabis plant sample analysis. Many laboratories that utilize GC methods for cannabis samples have adopted other quantitative approaches that do not account for the incomplete conversion of THCA to Δ<sup>9</sup>-THC [33, 34, 35]. One approach is to assume that the decarboxylation rate is 100 % despite its inaccuracies, resulting in an underestimation of total Δ<sup>9</sup>-THC in the sample. Alternatively, the THCA can be decarboxylated in an oven prior to solvent extraction, assuming complete conversion of THCA to Δ<sup>9</sup>-THC. However, THCA does not decarboxylate to Δ<sup>9</sup>-THC at a 1:1 ratio, and other cannabinoids such as CBN can be formed in this process as it is an oxidative product of Δ<sup>9</sup>-THC [46]. Regardless of the GC method being used, the total Δ<sup>9</sup>-THC measurements using GC methods will likely be biased low given the incomplete decarboxylation of THCA. If the sample preparation does not include decarboxylation prior to extraction, the GC method should be evaluated for decarboxylation rates as part of the method validation. Decarboxylation rates can be determined by injecting a known concentration of THCA into the GC and determining the difference between

the expected and measured  $\Delta^9$ -THC concentrations. Because lower rates of decarboxylation effectively increase the number of marijuana samples being classified as hemp, GC methods are considered to be conservative and prevent false positive results.

Laboratories interested in using decision point GC methods to differentiate hemp from marijuana, based on a jurisdictional legal limit, typically use an internal standard (IS) added to the extract and a positive control prior to analysis. The positive control contains the cutoff value of  $\Delta^9$ -THC as established by the jurisdiction, and the ratio of the signal of  $\Delta^9$ -THC to the signal of the IS in the positive control is used as the decision point ratio above which unknown samples are considered marijuana. To improve accuracy of these methods, isotopically labeled analogs of  $\Delta^9$ -THC should be used as the IS. Both  $\Delta^9$ -THC- $d_3$  and  $\Delta^9$ -THC- $d_9$  are widely available.

### 3.5. Conclusions

In general, laboratories more accurately quantitated  $\Delta^9$ -THC in the higher mass fraction samples than in the lower mass fraction samples. Difficulties with lower mass fraction samples could be the result of higher influence from calibration bias at the lower end of the calibration curves and increased impact from chromatographic interferences when  $\Delta^9$ -THC mass fractions are low. For the higher  $\Delta^9$ -THC samples, the opposite calibration bias may exist due to the  $\Delta^9$ -THC mass fraction being at or near the upper calibration limit. Laboratories should use calibration curves that closely represent the extracted concentration of  $\Delta^9$ -THC in the samples to increase the accuracy of analysis or dilute high-concentration samples so that the  $\Delta^9$ -THC is within the calibration range. To prevent calibration bias, laboratories should use calibration standards that meet ISO standards, ensure all purity information is reviewed, and ensure that the calibrants are independently prepared and traceable to the SI, if possible. In order to reduce the impact of interfering matrix components on the analysis of  $\Delta^9$ -THC, chromatographic methods should be thoroughly evaluated with some frequency to ensure baseline separation of known cannabinoids with retention times similar to  $\Delta^9$ -THC.

The accuracy of THCA quantitation was not dependent upon the concentration in the samples. For all but Plant Sample 3 and Plant Sample 4, the THCA consensus range was within the target range. For Plant Sample 3 and Plant Sample 4, sample size variability among laboratories may have led to sample inhomogeneity at smaller sample sizes. Overall for THCA, laboratories had acceptable within-laboratory variability and the between-laboratory variability was higher than the published performance criteria, but also indicative of calibration bias, as seen in the linear trends presented in **Fig. 3-17** and **Fig. 3-18**.

The accuracy of the total  $\Delta^9$ -THC measurements was predicated on the accuracy of the  $\Delta^9$ -THC and THCA measurements for laboratories using LC and not decarboxylating prior to extraction, which was the majority of participants. For laboratories reporting use of GC, method validation should include evaluating of decarboxylation rates. To improve accuracy, unknown sample extracts should be run in tandem with positive controls, and all extracts and positive controls should be spiked with isotopically labeled  $\Delta^9$ -THC and/or THCA analogs prior to injection.

## 4. CBD, CBDA, and Total CBD

### 4.1. Study Overview

The promotion of CBD as a therapeutic treatment for a range of conditions, including seizures, has increased since the passing of the 2018 Farm Bill [36], with CBD sales reaching \$4.6 billion in the US in 2020 [37]. While CBD products have gained popularity, regulation and accurate labeling of CBD products remains uncertain [38]. CBD and CBDA are the primary cannabinoids found in hemp plant samples, with CBDA being the form produced by the plant. Similar to  $\Delta^9$ -THC, CBD is formed when its acidic counterpart, CBDA, is decarboxylated through prolonged exposure to heat or light [15]. CBD concentrations are typically reported as total CBD because both CBD and CBDA are found in plant material with CBDA being converted to CBD in finished products. Participants in this study were asked to use in-house analytical methods to determine mass fractions (%) of CBD, CBDA, and total CBD in six cannabis plant samples on an as-received basis. Through participation in this study, laboratories can better understand the performance of their in-house methods relative to those being used by others in the community and gage the accuracy of their measurements via comparison to target mass fractions assigned by NIST. Participant results will be used by NIST to gain knowledge on important challenges facing analytical methodologies in measuring CBD, CBDA, and total CBDA in cannabis samples.

### 4.2. Sample Information

The target values and uncertainties for CBD, CBDA, and total CBD in the hemp and marijuana plant samples are provided in **Table 4-1** on an as-received basis. The target values and uncertainties for NRC HEMP-1 were determined at NRC Canada and taken from the HEMP-1 COA [6]. The target values and uncertainties in the remaining five cannabis plant samples were determined at NIST using LC-PDA and/or LC-MS/MS measurements as described in Section 2.2.2. Participants were not given any information regarding the mass fractions of CBD, CBDA, or total CBD in the samples.

**Table 4-1. Example individualized data summary table for CBD, CBDA, and total CBD.**

Laboratory-specific results and Z-scores were provided to each participant separately from this report to protect laboratory identities.

Lab Code: (Code)		Exercise 2 – Cannabinoids in Cannabis Plant Samples						
Lab Code:	(Code)	1. Your Results		2. Community Results		3. Target		
	Sample	Units	$x_i$	$s_i$	$Z'_{\text{comm}}$	$Z_{\text{NIST}}$	$x_{\text{NIST}}$	$u$
CBD	NRC HEMP-1	%					0.541	0.070
CBD	Plant Sample 2	%					1.16	0.34
CBD	Plant Sample 3	%					1.20	0.19
CBD	Plant Sample 4	%					0.589	0.019
CBD	Plant Sample 5	%					1.40	0.063
CBD	Plant Sample 6	%					0.241	0.046
CBDA	NRC HEMP-1	%					1.460	0.080
CBDA	Plant Sample 2	%					15.13	0.34
CBDA	Plant Sample 3	%					13.82	0.32
CBDA	Plant Sample 4	%					8.11	0.42
CBDA	Plant Sample 5	%					12.99	0.22
CBDA	Plant Sample 6	%					4.21	0.46
Total CBD	NRC HEMP-1	%					1.82	0.12
Total CBD	Plant Sample 2	%					14.32	0.28
Total CBD	Plant Sample 3	%					13.25	0.28
Total CBD	Plant Sample 4	%					7.72	0.36
Total CBD	Plant Sample 5	%					12.79	0.20
Total CBD	Plant Sample 6	%					3.93	0.40

*Individual laboratory results will appear in this section; laboratory-specific results were provided to each participant separately from this report.*

$x_i$  Mean of reported values  
 $s_i$  Standard deviation of reported values  
 $Z'_{\text{comm}}$  Z-score with respect to community consensus  
 $Z_{\text{NIST}}$  Z-score with respect to NIST value

$N$  Number of quantitative values reported  
 $x^*$  Robust mean of reported values  
 $s^*$  Robust standard deviation

$x_{\text{NIST}}$  NIST-assessed value  
 $u$  standard uncertainty about the NIST-assessed value and assigned values by NRC Canada for HEMP-1

### 4.3. Reporting Statistics

The enrollment and reporting statistics for CBD, CBDA, and total CBD are described in **Table 4-2** for all plant samples. A total of 226 laboratories registered to participate in Exercise 2 of CannaQAP with approximately 77 % of participants signing up to report values for CBD, CBDA, and total CBD in the hemp samples (NRC HEMP-1, Plant Sample 4, and Plant Sample 6) and 13 % of participants signing up to report values for CBD, CBDA, and total CBD in the marijuana samples (Plant Sample 2, Plant Sample 3, and Plant Sample 5). The percentage of laboratories that requested samples and returned results for CBD, CBDA, and total CBD was between 76 % and 80 % for the hemp samples and between 48 % and 78 % for the marijuana samples.

**Table 4-2. Reporting statistics for the enrollment to measure CBD, CBDA, and total CBD.**

Cannabinoid	Number of Participants	Percent Reporting Results (%) for Hemp Samples		
		NRC HEMP-1	Plant Sample 4	Plant Sample 6
CBD	179	78.2	78.2	78.2
CBDA	171	80.1	79.5	80.1
Total CBD	171	77.8	78.4	78.4

Cannabinoid	Number of Participants	Percent Reporting Results (%) for Marijuana Samples		
		Plant Sample 2	Plant Sample 3	Plant Sample 5
CBD	33	60.6	60.6	60.6
CBDA	27	66.7	66.7	66.7
Total CBD	27	66.7	66.7	66.7

Participants were asked to either submit quantitative results or qualitative results based on LOQs or threshold values. For all but CBDA in NRC HEMP-1, 100 % of participants reporting results submitted quantitative results (**Table 4-3**). In the case of CBDA in NRC HEMP-1, one participant reported a LOQ value, and the analytical method used was “other” preventing additional insight. The CBD, CBDA, and total CBD values in all of the Exercise 2 plant samples were an order of magnitude higher than the recommended LOQ for analytical methods in the AOAC SMPR [16], which is likely why the majority of participants were able to report quantitative values.

**Table 4-3. Number of laboratories reporting quantitative mass fractions for CBD, CBDA, and total CBD.**

Samples	CBD	CBDA	Total CBD
NRC HEMP-1	140	136	133
Plant Sample 4	140	136	134
Plant Sample 6	140	137	134
Plant Sample 2	20	18	18
Plant Sample 3	20	18	18
Plant Sample 5	20	18	18

#### 4.4. Study Results and Discussion

The participant results are summarized in the community results section of **Table 4-1** and were based on the numerical results reported by participating laboratories summarized in **Appendix B**. The percent difference between the consensus and target values for CBD, CBDA, and total CBD in the cannabis plant samples is illustrated in **Fig. 4-1**. With the exception of NRC HEMP-1, the consensus values for CBD were biased high with respect to the target values, with the consensus value falling within a percent of the target value for Plant Sample 4. Conversely, the consensus values for CBDA in all samples except Plant Sample 6 were biased low with respect to the target values. For total CBD, the bias closely followed the CBDA bias because CBDA was a larger contributor in the calculation of total CBD values than CBD.

No trends were observed relating CBD, CBDA, and total CBD mass fractions and bias, suggesting that the differences between the target and consensus values were not due to cannabinoid extraction issues. Additionally, the observed biases do not support error due to the decarboxylation of CBDA into CBD over the duration of the study. Theoretically, the total CBD consensus values would be closer to the target value (errors close to zero in **Fig. 4-1**) if the

increased CBD and decreased CBDA mass fractions were only the result of decarboxylation. Based on a Bayesian model fitting of stability data the total CBD equivalent would decay approximately 4.5 % over the course of a month at 40 °C [17]. The total CBD decay in the plant samples for CannaQAP Exercise 2 should be less than 4.5 % because participants were instructed to store the study samples *below* room temperature and decay rates slow at lower temperatures. The analytical differences between the consensus and target values were more likely based on systematic errors from calibration or random errors from improper storage conditions or accounting. Further discussion of specific analyte bias is included for CBD (Section 4.4.1), CBDA (Section 4.4.2), and total CBD (Section 4.4.3) below.

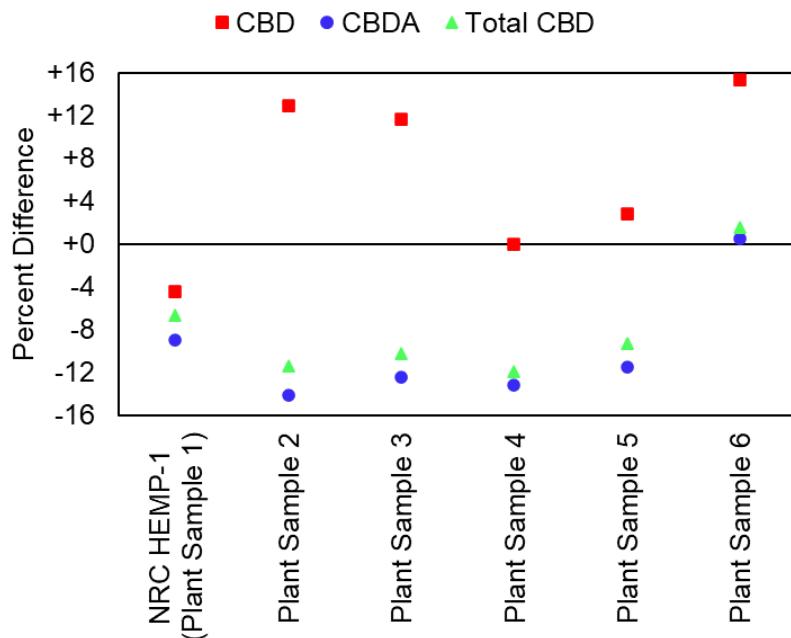


Fig. 4-1. Percent difference between the consensus mean and target value for CBD, CBDA, and total CBD.

The 0 % line represents no difference between the consensus mean and the target value.

**Table 4-4** details the within-laboratory (%RSD<sub>I</sub>) and between-laboratory (%RSD<sub>R</sub>) variabilities for CBD, CBDA, and total CBD in the six cannabis plant samples. The recommended within- and between-laboratory variabilities in the AOAC SMPR for quantitation of cannabinoids in hemp plant material are based on the mass fraction of analyte in the sample [16]. Participants had higher within-laboratory variabilities than are recommended in the AOAC SMPR for measurements in Plant Sample 4 (CBD, CBDA, and total CBD) and Plant Sample 2 (CBDA and total CBD). Higher than recommended within-laboratory variability for the study samples may be due to the use of smaller sample size for analysis since the sample may not be homogeneous below 0.5 g, or from pipetting errors that propagate over sample extraction and extract dilution. Between-laboratory variabilities were outside the recommended ranges for CBD, CBDA, and total CBD measurements of all plant samples. The AOAC method recommendations for reproducibility are intended for evaluation of a single method used in multiple laboratories and higher between-laboratory variabilities are expected when laboratories report values from multiple analytical methods.

**Table 4-4. Within-laboratory and between-laboratory variabilities for the determination of CBD, CBDA, and total CBD.**

<u>Samples</u>	<u>CBD</u>		<u>CBDA</u>		<u>Total CBD</u>	
	<u>%RSD<sub>r</sub></u>	<u>%RSD<sub>R</sub></u>	<u>%RSD<sub>r</sub></u>	<u>%RSD<sub>R</sub></u>	<u>%RSD<sub>r</sub></u>	<u>%RSD<sub>R</sub></u>
NRC HEMP-1	2.4 <sup>a</sup>	11.2 <sup>a</sup>	2.6 <sup>a</sup>	11.8 <sup>a</sup>	2.2 <sup>a</sup>	11.6 <sup>a</sup>
Plant Sample 4	4.9 <sup>a</sup>	12.2 <sup>a</sup>	4.1 <sup>c</sup>	11.2 <sup>c</sup>	4.2 <sup>c</sup>	12.0 <sup>c</sup>
Plant Sample 6	3.0 <sup>b</sup>	15.3 <sup>b</sup>	1.7 <sup>a</sup>	12.2 <sup>a</sup>	2.0 <sup>a</sup>	11.8 <sup>a</sup>
Plant Sample 2	2.1 <sup>a</sup>	14.1 <sup>a</sup>	2.3 <sup>c</sup>	13.8 <sup>c</sup>	2.2 <sup>c</sup>	13.6 <sup>c</sup>
Plant Sample 3	1.5 <sup>a</sup>	13.7 <sup>a</sup>	1.8 <sup>c</sup>	9.4 <sup>c</sup>	1.9 <sup>c</sup>	9.5 <sup>c</sup>
Plant Sample 5	1.6 <sup>a</sup>	13.9 <sup>a</sup>	1.6 <sup>c</sup>	12.0 <sup>c</sup>	1.6 <sup>c</sup>	12.0 <sup>c</sup>

<sup>a</sup> recommended %RSD<sub>r</sub> ≤ 3 % and % RSD<sub>R</sub> ≤ 8 % [16]

<sup>b</sup> recommended %RSD<sub>r</sub> ≤ 5 % and % RSD<sub>R</sub> ≤ 10 % [16]

<sup>c</sup> recommended %RSD<sub>r</sub> ≤ 2 % and % RSD<sub>R</sub> ≤ 6 % [16]

Most laboratories used a combination of solvent extraction for sample preparation and LC with either absorbance or photodiode array for analysis (**Table 4-5**). Approximately 94 % of laboratories used solvent extraction and the ≈ 5 % of laboratories reporting dilution also likely used solvent extraction prior to dilution because the samples were all plant material and could not be directly analyzed following only dilution. Due to the lack of data on actual solvents used for extraction as well as lack of detailed extraction procedures, no trends based on sample preparation were able to be assessed unless a candidate method was used. The results and discussion presented here will focus on comparability of different instrumental methods, chemical interferences, and calibration approaches. Additional information for sample preparation and LC instrumental methods are provided in **Appendix C** based on responses to a method questionnaire completed by 93 participants.

**Table 4-5. Percent of laboratories reporting specific sample preparation and analytical methods for the determination of CBD, CBDA, and total CBD.**

<u>Preparation Method</u>	<u>CBD</u>	<u>CBDA</u>	<u>Total CBD</u>
Solvent Extraction	93.1	95.2	93.0
Dilution	6.3	4.1	4.4
Other/No Response	0.6	0.6	2.6

<u>Analytical Method</u>	<u>CBD</u>	<u>CBDA</u>	<u>Total CBD</u>
LC-PDA	49.4	48.6	48.8
LC-ABS	37.3	38.2	35.2
LC-MS	1.5	0.9	0.9
LC-MS/MS	7.5	7.8	7.3
GC-FID	1.9	1.9	4.6
GC-MS	0.0	0.0	0.0
Other/No Response	2.5	2.6	3.3

#### 4.4.1. CBD

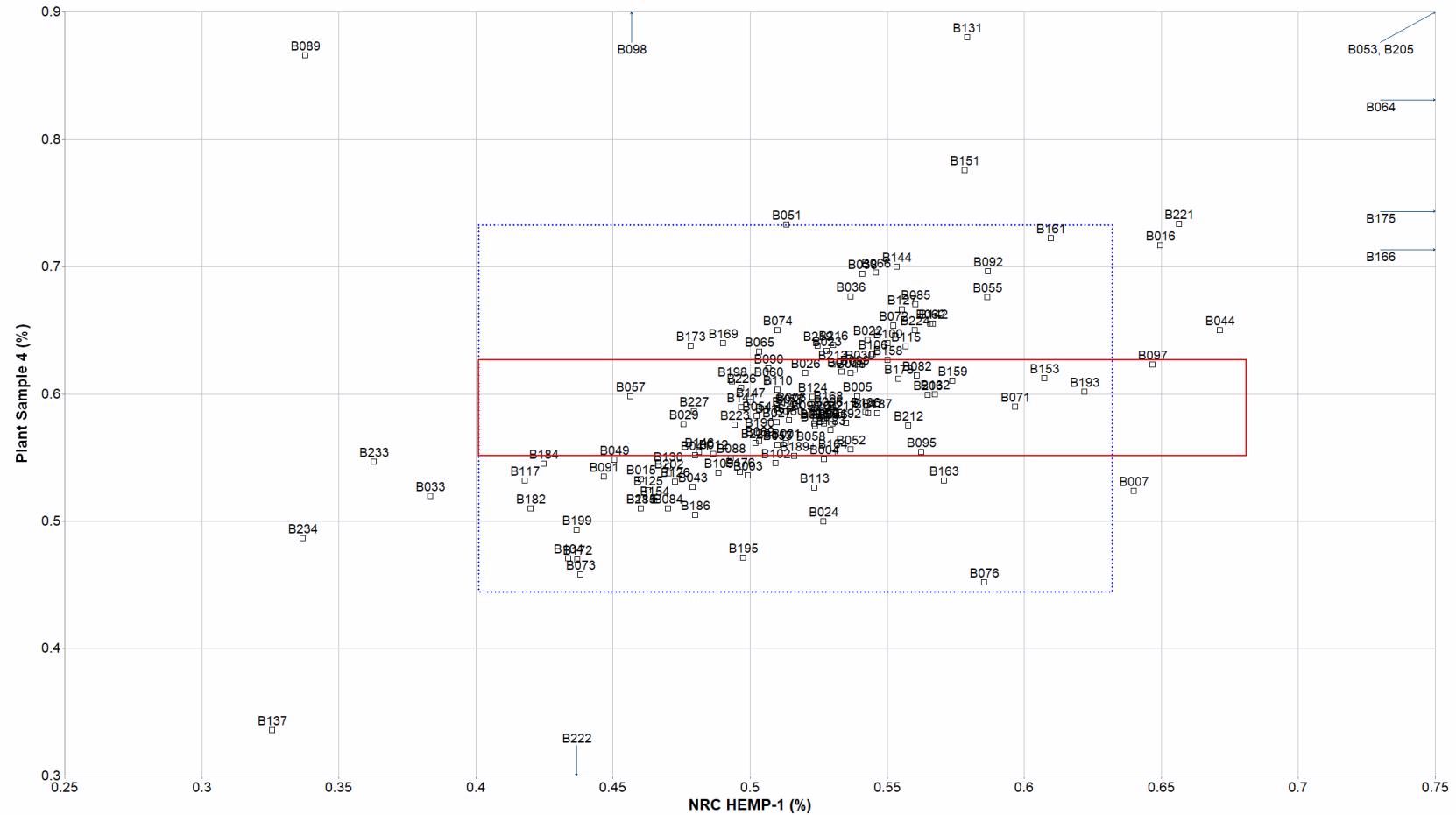
##### 4.4.1.1. Within- and Between-Laboratory Precision

Overall, laboratories reporting quantitative results for CBD in all plant samples demonstrated within-laboratory variability (repeatability, %RSD<sub>r</sub>) less than or equal to 4.9 %, with individual laboratories having within-laboratory variabilities as high as 75 %. The single laboratory with a

within-laboratory variability of 75% likely had a data entry error as one of the triplicate values was an order of magnitude lower than the other two entries. CBD target values in the six study samples ranged from 0.241 % to 1.40 %. The recommended within-laboratory variability for Plant Sample 6, according to the AOAC SMPR, was  $\leq 5\%$  and the value for the other 5 study samples was  $\leq 3\%$  [16]. Between 42 % and 85 % of participants had repeatability values within the recommended values, with smaller within-laboratory variabilities for CBD measurements in marijuana samples than in hemp samples (**Table 4-4**). The CBD values in NRC HEMP-1 and Plant Sample 4 were at the threshold percentage where the recommended within-laboratory variability decreased from 5 % to 3 %, likely reducing the percentage of laboratories meeting the recommended repeatability performance criteria. Because the CBD values in the samples required dilution to be within the calibration ranges used by most laboratories, laboratories with higher within-laboratory variabilities may have experienced pipetting errors during extract dilution.

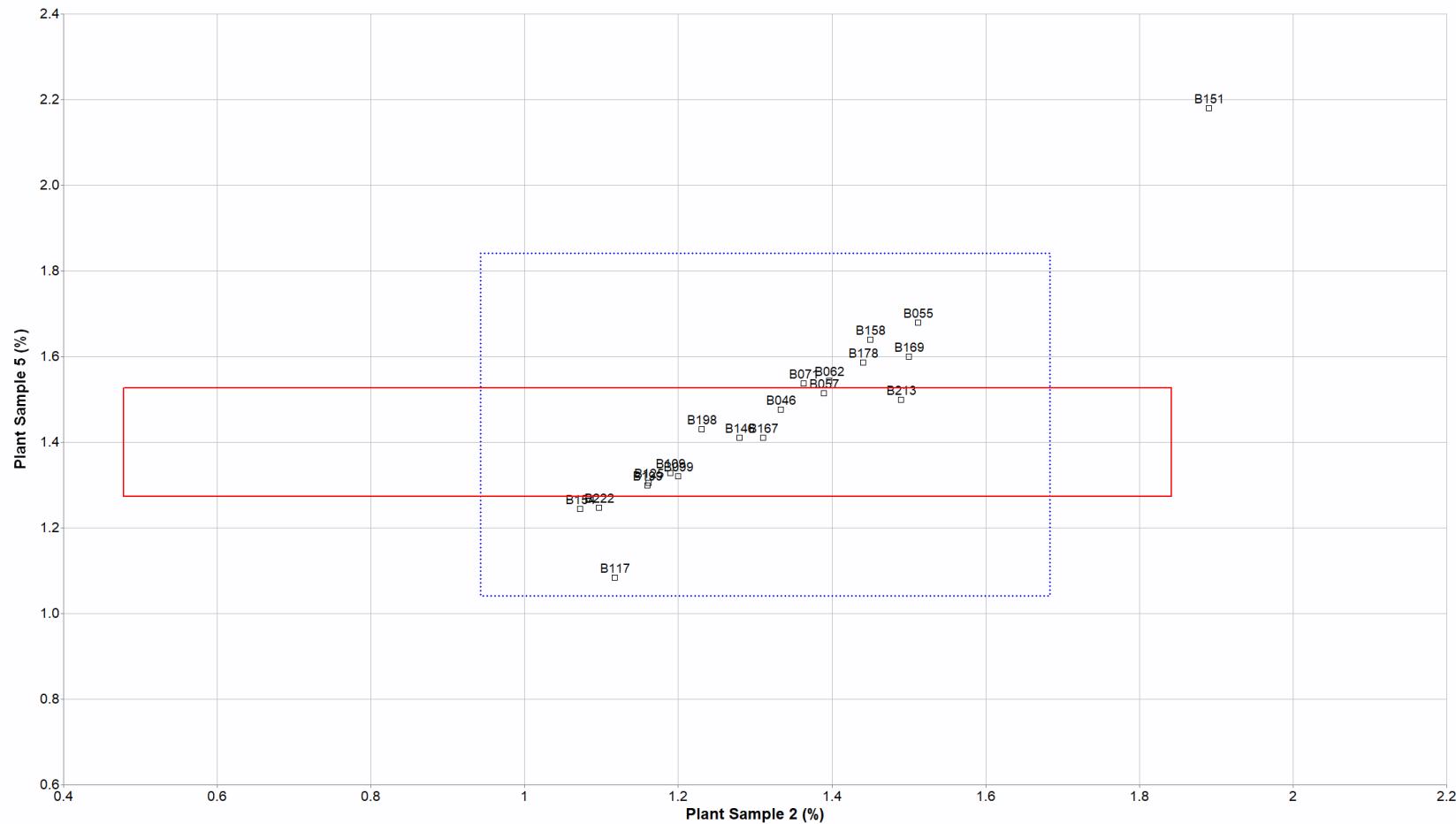
The between-laboratory variability (reproducibility,  $\%RSD_R$ ) was reasonable (11.2 % to 15.3 %) with respect to the published expectations (8 % to 10 %) considering the AOAC guidelines for between-laboratory precision are meant to be applied to variabilities from multiple laboratories using a single analytical method. The between-laboratory variabilities for CBD in all study samples for this exercise are comparable to the UK-PT program overall analyte RSDs for CBD in hemp, reported over the course of 16 studies (7.34 % to 12.6 %) [19].

To further examine the potential causes of between-laboratory variability, laboratory performance on two separate samples with similar analyte mass fractions was compared (**Fig. 4-2** and **Fig. 4-3**). NRC HEMP-1 and Plant Sample 4 both contained CBD mass fractions between 0.5 % and 0.6 %. The trend observed when comparing NRC HEMP-1 to Plant Sample 4 (**Fig. 4-2**) is moderately linear, which indicates that systematic between-laboratory errors were more pronounced than random errors. Possible systematic errors include calibration bias and global methodological issues, such as poor chromatographic separation of coeluting cannabinoids. Laboratories that were within the consensus range for one sample and outside the range for the other, such as B098, B222, B233 and B166, likely encountered random errors when preparing one but not both samples. For example, B222 and B166 may have made calculation errors when determining the CBD values for Plant Sample 4 (B222) and NRC HEMP-1 (B166). While B222 reported half the target value for Plant Sample 4, B166 reported twice the target value, both potentially errors in accounting for dilution. Other random errors that could impact variabilities include pipetting errors, sample massing errors, and potential for coeluting compounds in one sample and not another. When the CBD data from Plant Sample 2 was compared to Plant Sample 5, the majority of laboratories that reported either high or low relative to the target and consensus ranges did so consistently between samples, which is indicative of systematic errors such as calibration bias. Some ways to prevent calibration bias include purchasing standards from different manufacturers, checking the purity information of standards to ensure use of the correct analyte concentration, and preparing calibrants independently to avoid propagated errors from the use of serial dilutions.



**Fig. 4-2. Laboratory means for CBD in NRC HEMP-1 and Plant Sample 4 (sample/sample comparison view).**

In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (Plant Sample 4). The solid red box represents the target range of tolerance for the two samples, NRC HEMP-1 (x-axis) and Plant Sample 4 (y-axis), which encompasses the target values bounded by their uncertainties ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The dotted blue box represents the consensus range of tolerance for NRC HEMP-1 (x-axis) and Plant Sample 4 (y-axis), calculated as the values above and below the consensus means that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ .



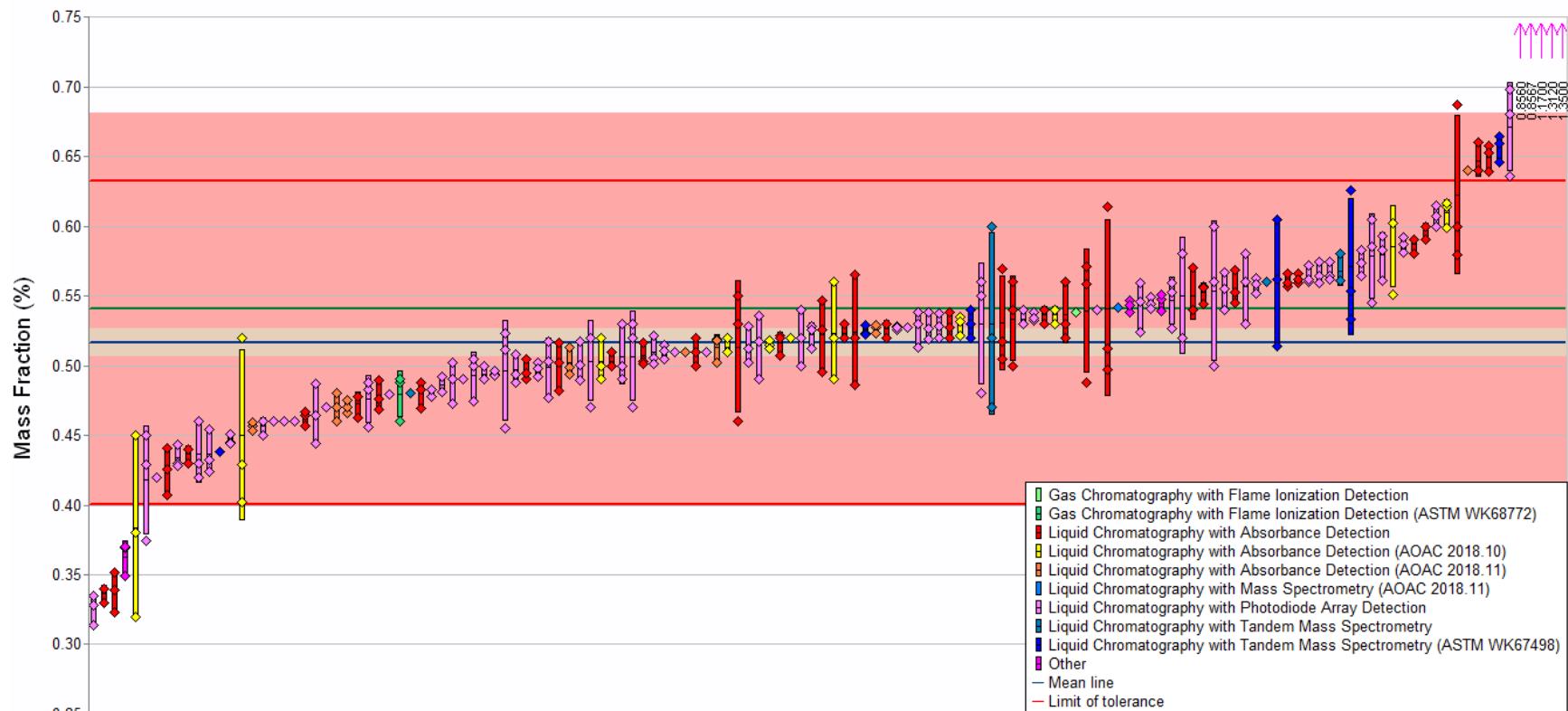
**Fig. 4-3. Laboratory means for CBD in Plant Sample 2 and Plant Sample 5 (sample/sample comparison view).**

In this view, the individual laboratory mean for one sample (Plant Sample 2) is compared to the individual laboratory mean for a second sample (Plant Sample 5). The solid red box represents the target range of tolerance for the two samples, Plant Sample 2 (x-axis) and Plant Sample 5 (y-axis), which encompasses the target values bounded by their uncertainties ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The dotted blue box represents the consensus range of tolerance for Plant Sample 2 (x-axis) and Plant Sample 5 (y-axis), calculated as the values above and below the consensus means that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ .

#### 4.4.1.2. Accuracy

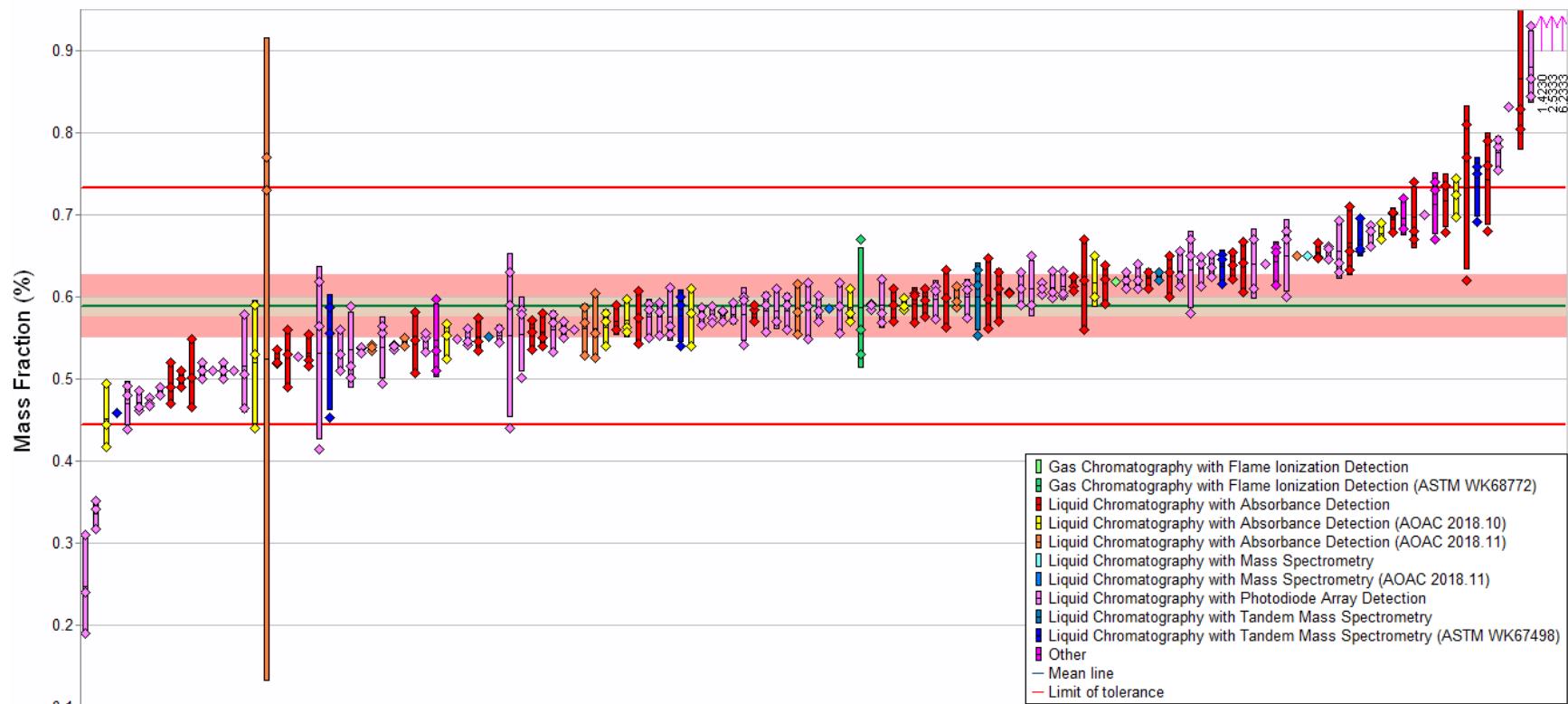
The individual participant, consensus, and target mass fraction results are presented in a tabular form in **Appendix B**, and graphically in **Fig. 4-4** through **Fig. 4-9**. The consensus ranges for CBD in all but one of the study samples were completely within the target ranges. The consensus range for CBD in Plant Sample 5 extended slightly above the target range. Between 80 % and 87 % of participants used either LC-ABS or LC-PDA as their analytical method across all samples and no trend was observed to suggest bias due to use of one method over another. Between 7 % (NRC HEMP-1) and 54 % (Plant Sample 4) of laboratories reporting quantitative results reported a mean result outside the target range for the three hemp samples and 5 % (Plant Samples 2 and 3) to 50 % (Plant Sample 5) of laboratories reported outside the target range for the three marijuana samples. The target range for Plant Sample 4 was much smaller than for the other plant samples, which is likely the reason a higher fraction of laboratories reported outside the target range for Plant Sample 4. However, for Plant Sample 5, 70% of the laboratories that reported CBD values outside the target range reported CBD values higher than the target range, which is consistent with systematic (calibration) errors seen for the marijuana samples in **Fig. 4-3**.

The target values for CBD in the six cannabis samples cover the typical range found in hemp and marijuana samples, permitting NIST to appropriately evaluate the different analytical methods. The consensus values for the plant samples were within 16 % of the target values, with participants performing similarly in the hemp and marijuana samples. Plant Sample 6 contained the smallest amount of CBD and yielded the highest percent difference between the consensus and target values. The majority of participants used a calibration range from 0.5 ppm to 100 ppm based on the calibration range data reported in the method questionnaire. Assuming the extraction masses and solvent volumes were similar to those used by NIST (Section **2.2.1**), CBD in the extracts was within the calibration range for NRC HEMP-1, Plant Sample 4, and Plant Sample 6, and would have required dilution for Plant Sample 2, Plant Sample 3, and Plant Sample 5. However, the CBD in all samples could have been measured in a diluted extract and still been within the majority of calibration curves. Laboratories may have measured CBD in diluted extracts for all samples, in which case the CBD value for Plant Sample 6 would have been on the lower end of the calibration curve, resulting in less accurate values for Plant Sample 6 than the other samples. When analyte concentrations fall in the tails of a calibration curve, the quantitative accuracy decreases. Analysts must recognize when an extract should either be diluted using a different dilution factor or measured directly in an extract and not diluted based on the calibration curve that was used. If the extract is outside of or at the tails of a calibration curve, the calibration curve should be adjusted to better suit the analyte concentration (Section **3.4.1.4**).



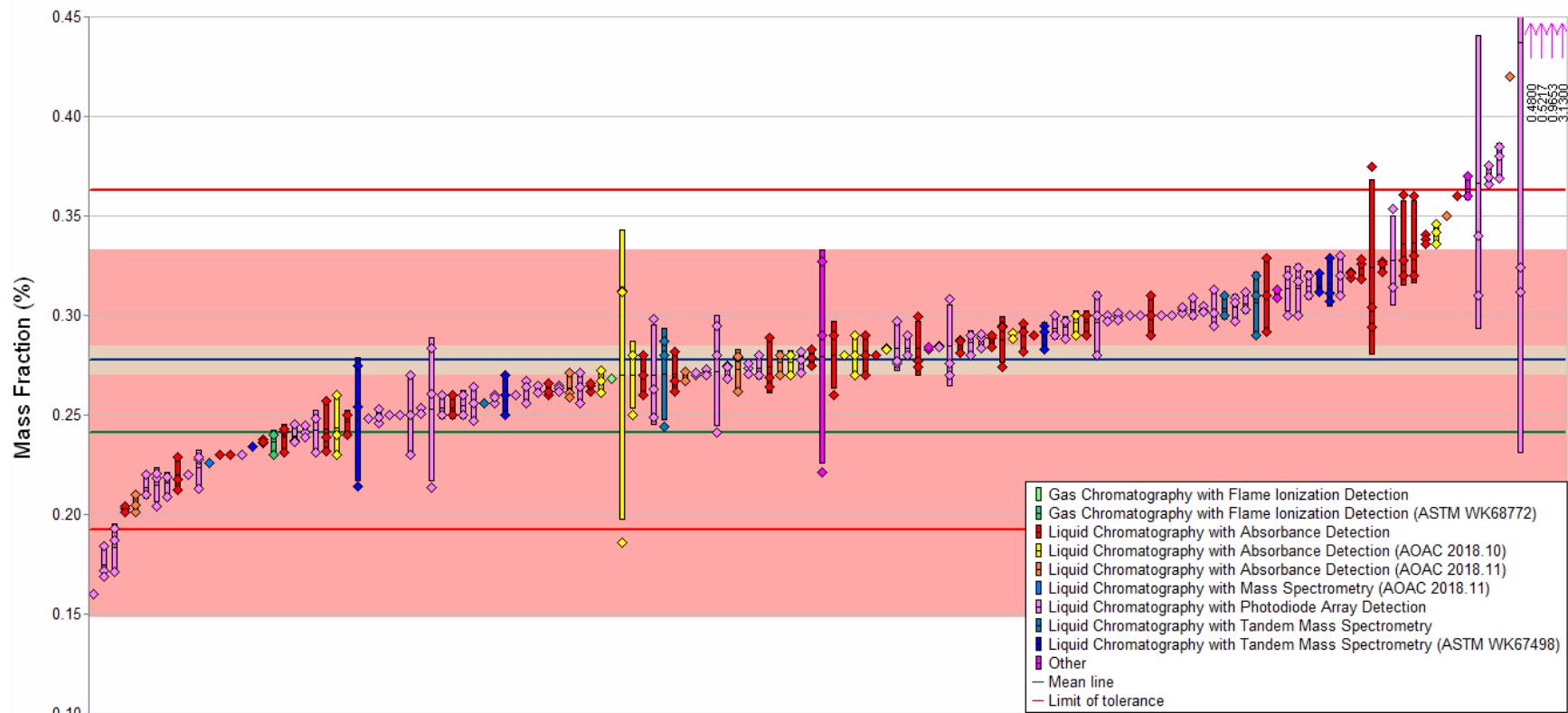
**Fig. 4-4. CBD in NRC HEMP-1 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



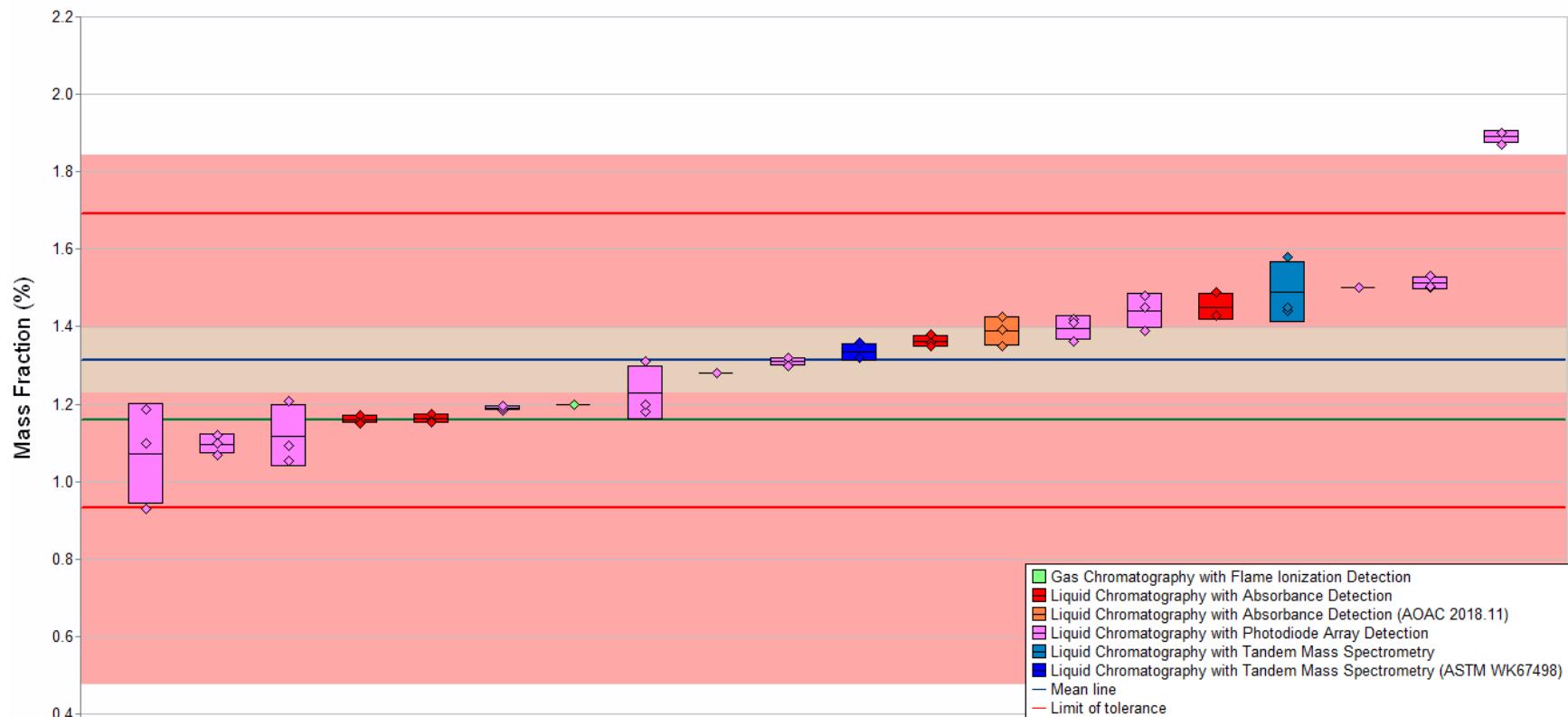
**Fig. 4-5. CBD in Plant Sample 4 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



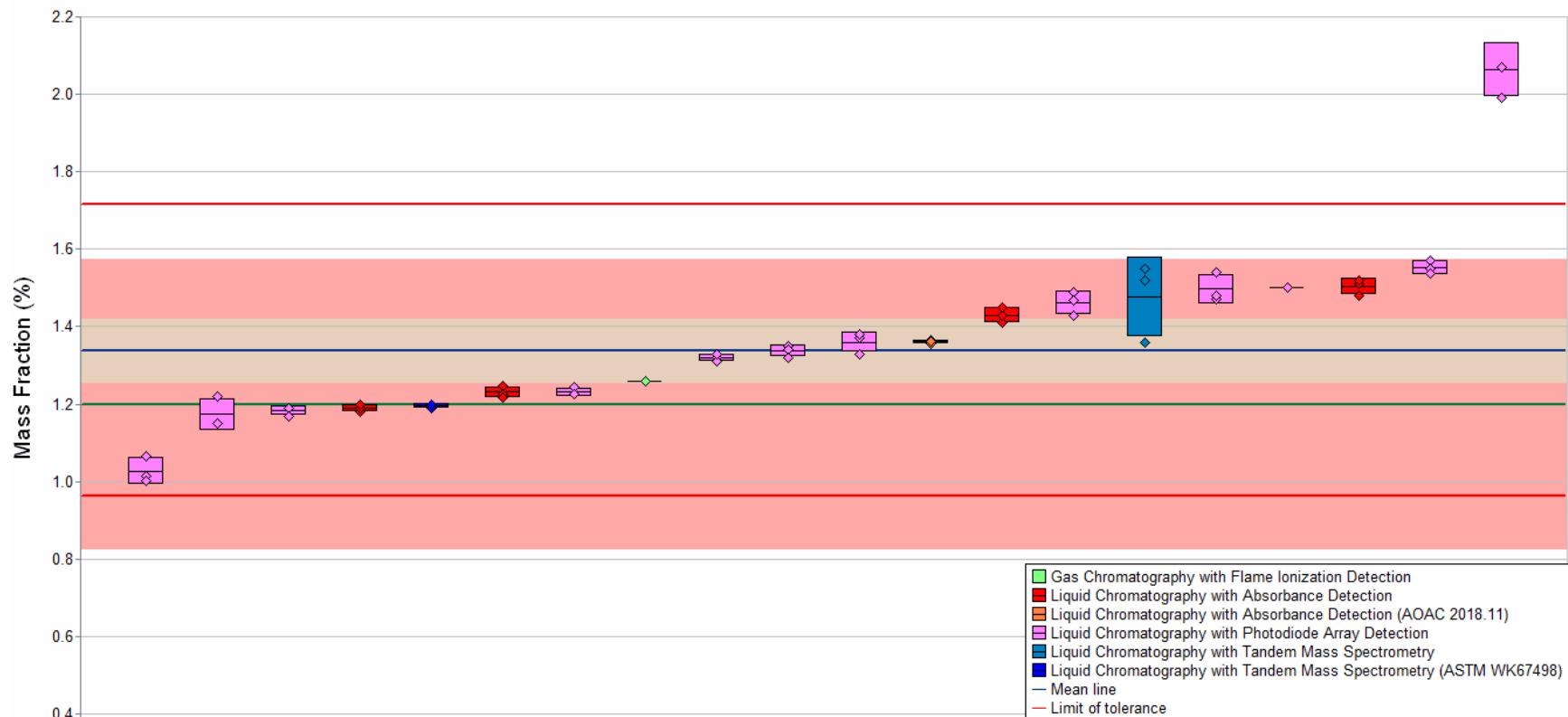
**Fig. 4-6. CBD in Plant Sample 6 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



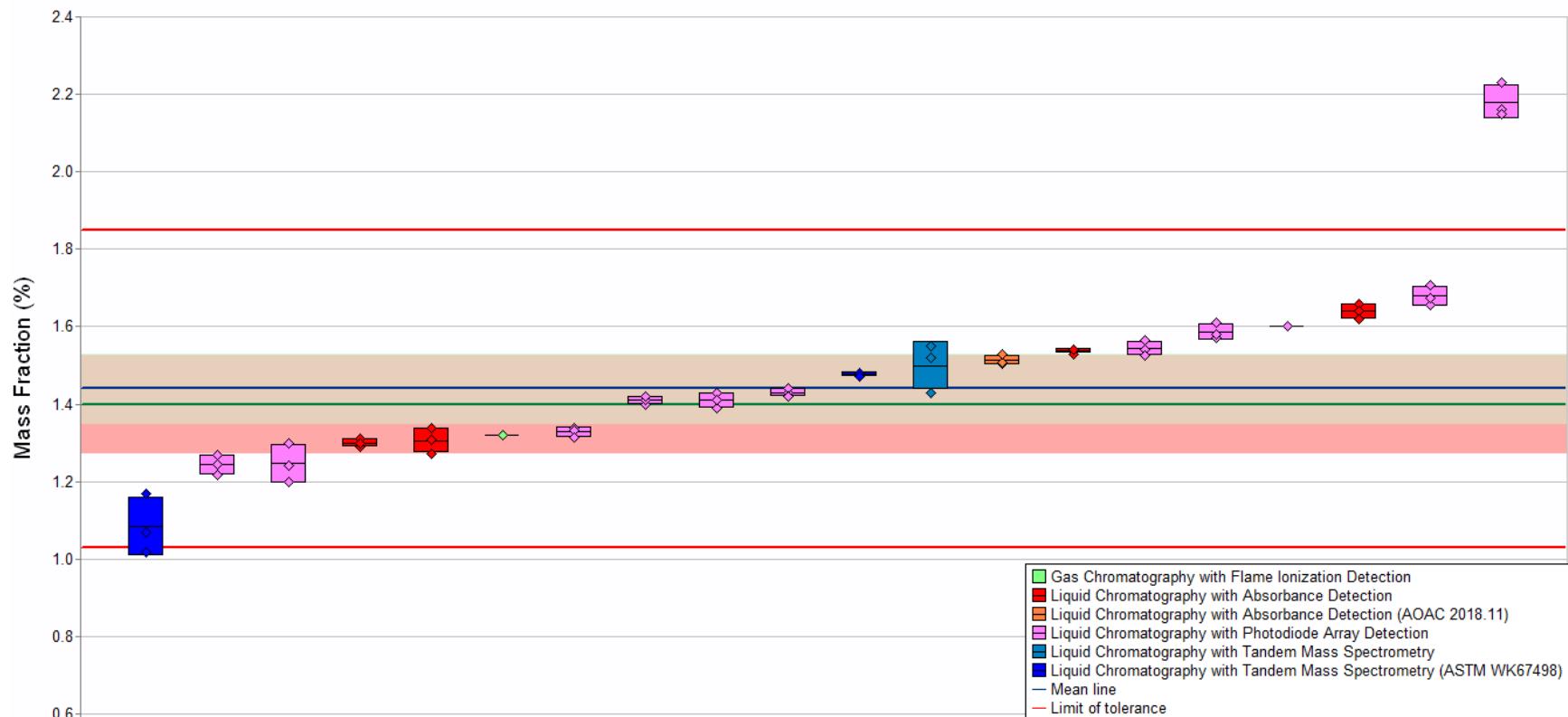
**Fig. 4-7. CBD in Plant Sample 2 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 4-8. CBD in Plant Sample 3 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 4-9. CBD in Plant Sample 5 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

#### 4.4.1.3. Candidate Analytical Methods

NIST provided a list of nine candidate standard methods from AOAC International and ASTM International for participants to use if an in-house analytical method was not available. Mass fractions were submitted by participating laboratories for CBD using an ASTM LC-MS/MS method and two AOAC methods. The within-laboratory (repeatability, %RSD<sub>r</sub>) and between-laboratory (reproducibility, %RSD<sub>R</sub>) variabilities are summarized for candidate method/sample pairs for which participants reported at least two independent measurements for a sample and at least five laboratories reported data (**Table 4-6**). Both AOAC methods were previously approved by an expert review panel using criteria established in SMPR 2017.002 for the quantitation of cannabinoids in dried cannabis plant samples [21], which requires %RSD<sub>r</sub> and %RSD<sub>R</sub> to be ≤ 5 % and ≤ 7 %, respectively, for CBD when the mass fraction is between 0.1 % to 1 %. AOAC has since published SMPR 2019.003 for quantitation of cannabinoids in hemp plant samples [16], which requires %RSD<sub>r</sub> and %RSD<sub>R</sub> to be ≤ 5 % and ≤ 10 % for CBD when its mass fraction is between 0.05 % to 0.5 %. For higher mass fraction CBD samples (CBD > 0.5 % to 5 %), the requirement is for %RSD<sub>r</sub> and %RSD<sub>R</sub> to be ≤ 3 % and ≤ 8 %, respectively [16]. In samples containing between 5 % and 35 %, the requirement is for %RSD<sub>r</sub> and %RSD<sub>R</sub> to be ≤ 2 % and ≤ 6 %, respectively [16]. Because ASTM has not published equivalent requirements, the requirements established in SMPR 2019.003 will be used in this study to be evaluated both AOAC and ASTM methods.

**Table 4-6. Within- and between-laboratory variabilities for CBD measurements using candidate standardized analytical methods.**

	<u>LC-Absorbance</u>	NRC HEMP-1			Plant Sample 4			Plant Sample 6		
		<u>n<sup>a</sup></u>	%RSD <sub>r</sub>	%RSD <sub>R</sub>	<u>n<sup>a</sup></u>	%RSD <sub>r</sub>	%RSD <sub>R</sub>	<u>n<sup>a</sup></u>	%RSD <sub>r</sub>	%RSD <sub>R</sub>
	AOAC 2018.10	11	2.7	7.8	11	5.4	10.9	11	2.7	7.4
	AOAC 2018.11	8	1.0	8.3	7	4.4	11.3	7	2.2	14.6
	<u>LC-MS/MS</u>									
	ASTM WK67498	5	3.4	7.5	5	6.2	18.0	5	3.9	14.8

<sup>a</sup> n = number of laboratories

#### AOAC 2018.10

The %RSD<sub>r</sub> published for AOAC 2018.10 was between 1.08 % and 4.74 % for CBD measured in 6 independent dried flower samples (n = 4 for each sample) at concentrations between 0.22 % and 0.70 % [22], which was within the published requirement of 5 % in SMPR 2017.002 [21]. The published %RSD<sub>r</sub> of 4.47 % for CBD in hemp flower at a mass fraction of 0.56 % was within the expectations for SMPR 2017.002 but did not meet expectations from the most current SMPR of 3 % [16]. The average within-laboratory variability observed for Exercise 2 hemp samples was within the reported variability in the published method [22] and %RSD<sub>r</sub> requirement of 3 % and 5 % [16] for NRC HEMP-1 and Plant Sample 6, respectively. The %RSD<sub>r</sub> for Plant Sample 4 for participants using AOAC 2018.10 was higher than the %RSD<sub>r</sub> published in the method and the 2019 SMPR (3 %, [16]). The CBD values reported by laboratories using AOAC 2018.10 were lower than the target value for NRC HEMP-1 (3 %) and Plant Sample 4 (1 %) and higher than the target value for Plant Sample 6 (16 %). The between-laboratory variabilities observed for CBD measured by AOAC 2018.10 in NRC HEMP-1 and Plant Sample 6 were within the 8 % and 10 % requirements,

respectively [16], while the between-laboratory variability for CBD in Plant Sample 4 was above the SMPR reproducibility requirement of 8 %. Observed reproducibility of AOAC 2018.10 had not been published at the time of this report.

#### **AOAC 2018.11**

The %RSD<sub>r</sub> published for AOAC 2018.11 was reported from two separate analysts. The combined %RSD<sub>r</sub> was 2.8 % for dried plant material samples containing 0.698 % CBD ( $n = 10$ , [7]), which was within the published requirements of  $\leq 5\%$  [21] and  $\leq 3\%$  [16]. The average within-laboratory variability for CBD measurements observed was consistent with the %RSD<sub>r</sub> published in the method and SMPR for NRC HEMP-1 and Plant Sample 6. The within-laboratory variability for Plant Sample 4 observed by participants (4.4 %) was above the recommended range of  $\leq 3\%$  [16]. Laboratories using the AOAC 2018.11 method reported CBD values that were lower than the target value for NRC HEMP-1 (8 %) and Plant Sample 6 (3 %) and higher than the target value for Plant Sample 4 (17 %). The between-laboratory variabilities observed for CBD measured in NRC HEMP-1 and Plant Sample 4 using AOAC 2018.11 were above the 8% requirement and the between-laboratory variability for Plant Sample 6 was above the 10 % requirement [16]. Observed reproducibility of AOAC 2018.11 had not been published at the time of this report.

#### **ASTM WK67498**

The ASTM WK67498 method was developed following the AOAC [23] and ASTM [24] guidelines. The %RSD<sub>r</sub> published for ASTM WK67498 was tested on 5 lots of the same hemp sample with %RSD<sub>r</sub> between 0.3 % and 3 % for hemp samples containing 0.484 % to 0.656 % CBD ( $n = 3$ , [25]), which were all within the published requirements of  $\leq 5\%$  or  $\leq 3\%$  based on the mass fraction of CBD in the hemp samples [16]. The average within-laboratory variability observed was higher than the %RSD<sub>r</sub> published in the method and SMPR [16]. Laboratories using the ASTM WK67498 method reported CBD values that were higher than the target value for NRC HEMP-1 (4 %), Plant Sample 4 (7 %), and Plant Sample 6 (19 %). The between-laboratory variabilities observed for CBD measured in the hemp samples were above the 8 % requirement for NRC HEMP-1 and Plant Sample 4 and above the 10 % requirement for Plant Sample 6 [16]. Observed reproducibility of ASTM WK67498 on hemp had not been published at the time of this report.

### **4.4.2. CBDA**

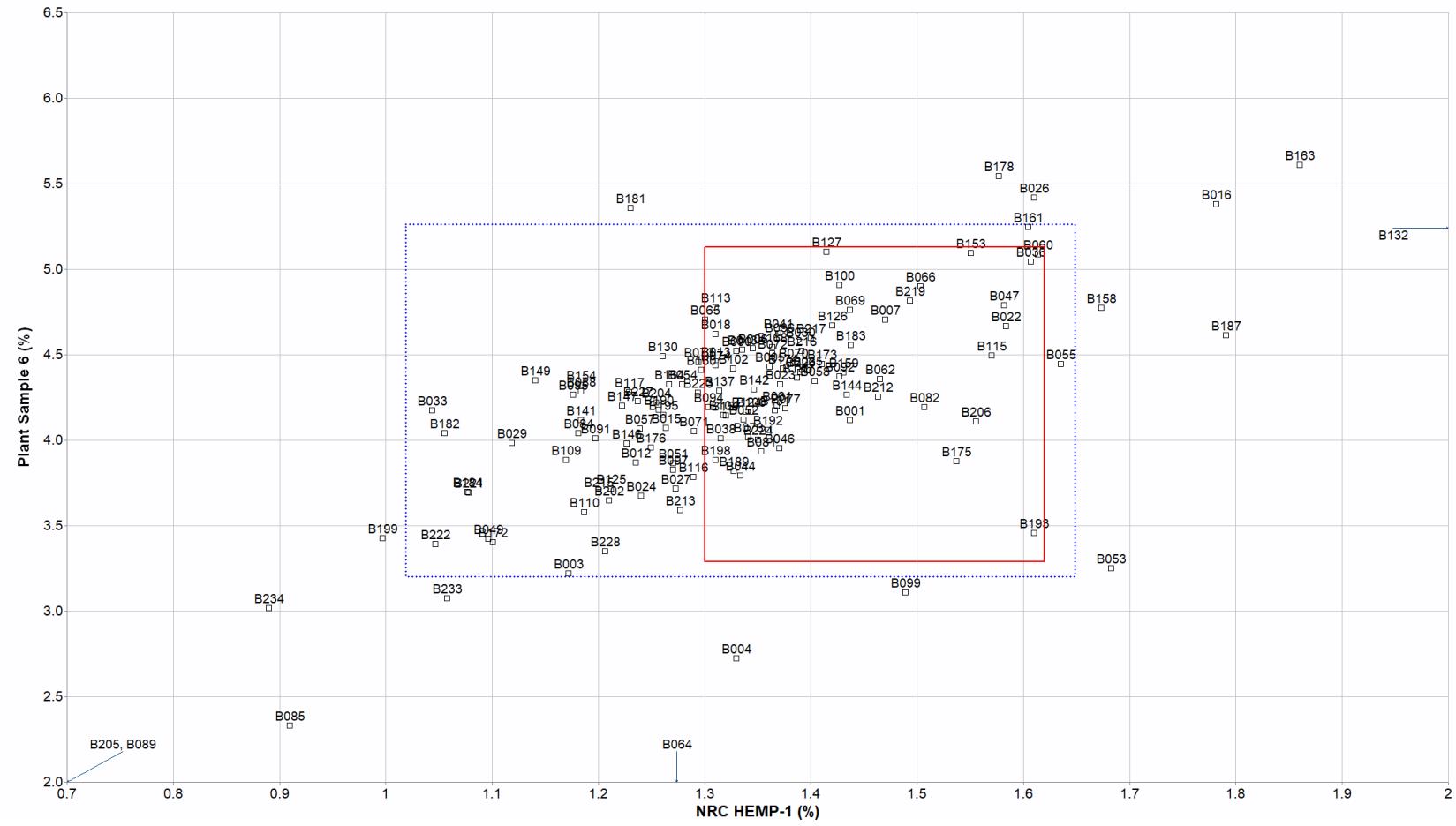
#### **4.4.2.1. Within- and Between-Laboratory Precision**

Overall, laboratories reporting quantitative results for CBDA in all plant samples demonstrated within-laboratory variability (repeatability, %RSD<sub>r</sub>) less than or equal to 4.1 %, with individual laboratories having within-laboratory variabilities at or below 26 %. CBDA values in the six study samples ranged from 1.46 % to 15.13 %. According to the AOAC SMPR 2019.003 [16], the recommended maximum within-laboratory variability for NRC HEMP-1 and Plant Sample 6 was 3 % and the value for the other 4 study samples was 2 %. Between 32 % and 78 % of participants had repeatability values for CBDA measurements within the recommended values for all samples. With the exception of Plant Sample 4, the within-laboratory variabilities were similar for hemp and marijuana samples, which could be due to the requirement to measure CBDA in diluted extracts across all samples (**Table 4-4**). Because the CBDA values in the samples required dilution

to be within the calibration ranges reported by laboratories that answered the method questionnaire (**Appendix C**), laboratories with higher within-laboratory variabilities may have experienced pipetting errors during extract dilution.

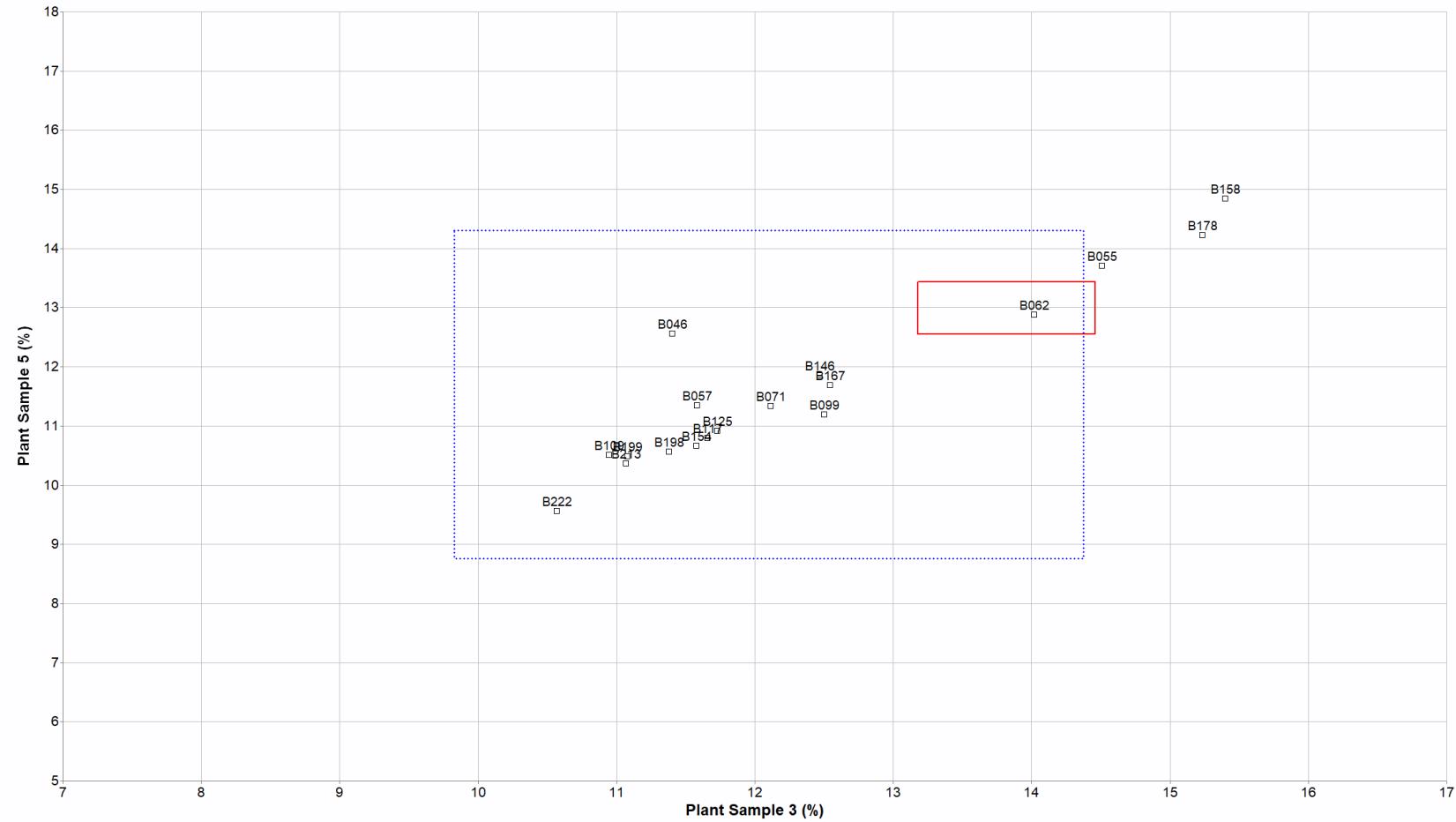
The between-laboratory variability was reasonable (9.4 % to 13.8 %) with respect to the published expectations (6 % to 8 %) considering the AOAC guidelines for between-laboratory precision are meant to be applied to variabilities from multiple laboratories using a single analytical method. The between-laboratory variabilities for CBD in all study samples for this exercise are comparable to the UK-PT program overall analyte RSDs for CBDA in hemp, reported over the course of 17 studies (8.60 % to 25.4 %) [19].

To further examine the potential causes of between-laboratory variability, laboratory performance on two separate samples with similar analyte mass fractions was compared (**Fig. 4-10** and **Fig. 4-11**). The CBDA concentrations in NRC HEMP-1 (1.46 %) and Plant Sample 6 (4.21 %) were below 5 %. The trend observed when comparing NRC HEMP-1 to Plant Sample 4 (**Fig. 4-10**) is slightly linear, which indicates a combination of random and systematic errors contributing to the between-laboratory variability. Possible systematic errors include calibration bias and global methodological issues, such as poor chromatographic separation of coeluting cannabinoids. More laboratories reported CBDA values below the target range for NRC HEMP-1 than Plant Sample 6. The CBDA concentration in a diluted NRC HEMP-1 extract may have been on the lower end of the calibration curve, resulting in less accurate and potentially lower values. Laboratories outside the consensus value of one sample and not the other, may have had random errors effecting the measurement of CBDA in one of the materials. For example, laboratory B004 was within the consensus range for NRC HEMP-1, but below the range for Plant Sample 6, possibly due to an incorrect dilution factor as the reported CBDA value was 1.5 times lower than the target value. Conversely, laboratory B187 reported a CBDA value within the consensus range for Plant Sample 6 and higher than the consensus range for NRC HEMP-1, possibly due to a missed dilution factor in the calculation of the CBDA concentration in NRC HEMP-1. When the CBDA data from Plant Sample 3 was compared to Plant Sample 5, the majority of laboratories that reported either high or low relative to the target and consensus ranges did so consistently between samples, which is indicative of systematic errors such as calibration bias. Some ways to prevent calibration bias include purchasing standards from different manufacturers, checking the purity information of standards to ensure use of the correct analyte concentration, and preparing calibrants independently to avoid propagation of errors from the use of serial dilutions.



**Fig. 4-10. Laboratory means for CBDA in NRC HEMP-1 and Plant Sample 6 (sample/sample comparison view).**

In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (Plant Sample 6). The solid red box represents the target range of tolerance for the two samples, NRC HEMP-1 (x-axis) and Plant Sample 6 (y-axis), which encompasses the target values bounded by their uncertainties ( $U_{NIST}$ ) and represents the range that results in an acceptable  $Z_{NIST}$  score,  $|Z_{NIST}| \leq 2$ . The dotted blue box represents the consensus range of tolerance for NRC HEMP-1 (x-axis) and Plant Sample 6 (y-axis), calculated as the values above and below the consensus means that result in an acceptable  $Z'_{comm}$  score,  $|Z'_{comm}| \leq 2$ .



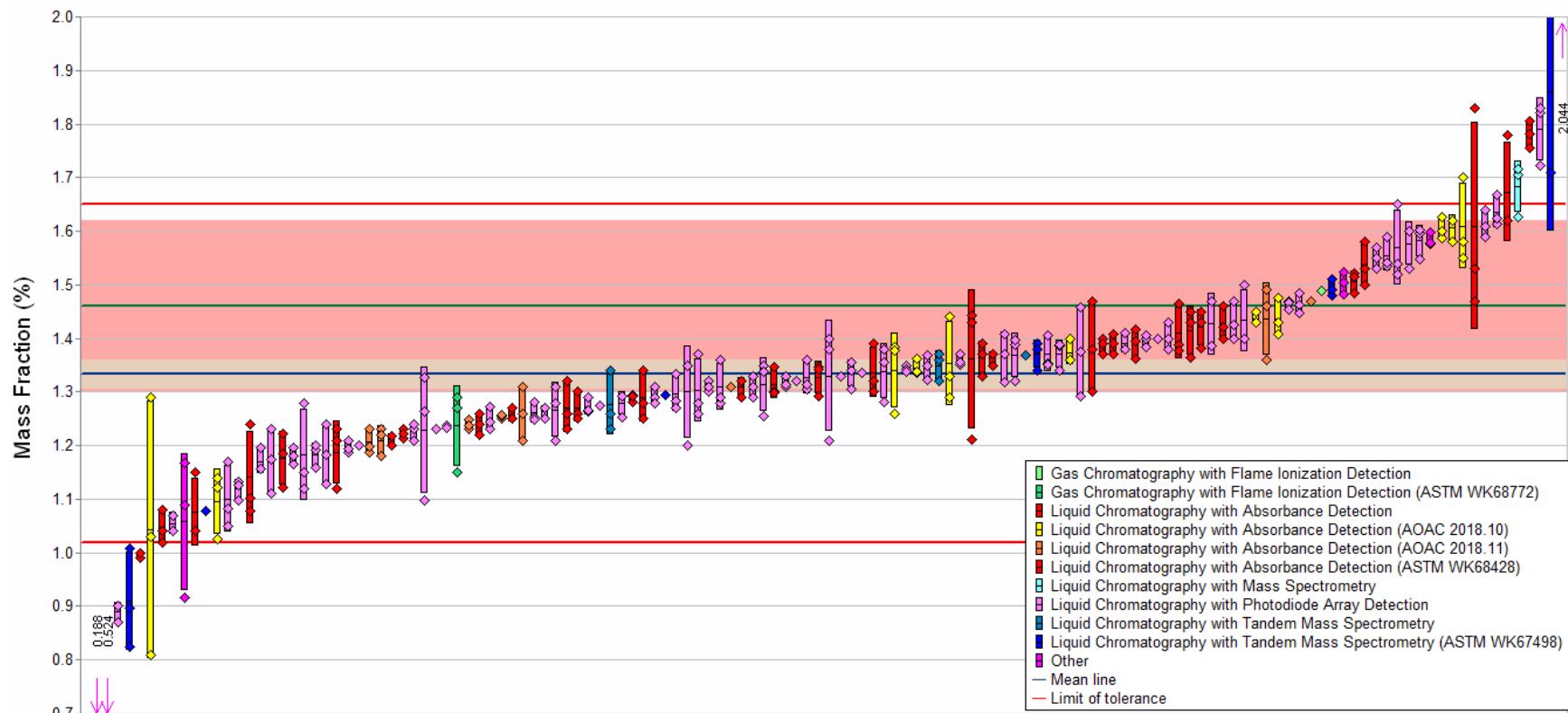
**Fig. 4-11. Laboratory means for CBDA in Plant Sample 3 and Plant Sample 5 (sample/sample comparison view).**

In this view, the individual laboratory mean for one sample (Plant Sample 3) is compared to the individual laboratory mean for a second sample (Plant Sample 5). The solid red box represents the target range of tolerance for the two samples, Plant Sample 3 (x-axis) and Plant Sample 5 (y-axis), which encompasses the target values bounded by their uncertainties ( $U_{NIST}$ ) and represents the range that results in an acceptable  $Z_{NIST}$  score,  $|Z_{NIST}| \leq 2$ . The dotted blue box represents the consensus range of tolerance for Plant Sample 3 (x-axis) and Plant Sample 5 (y-axis), calculated as the values above and below the consensus means that result in an acceptable  $Z'_{comm}$  score,  $|Z'_{comm}| \leq 2$ .

#### 4.4.2.2. Accuracy

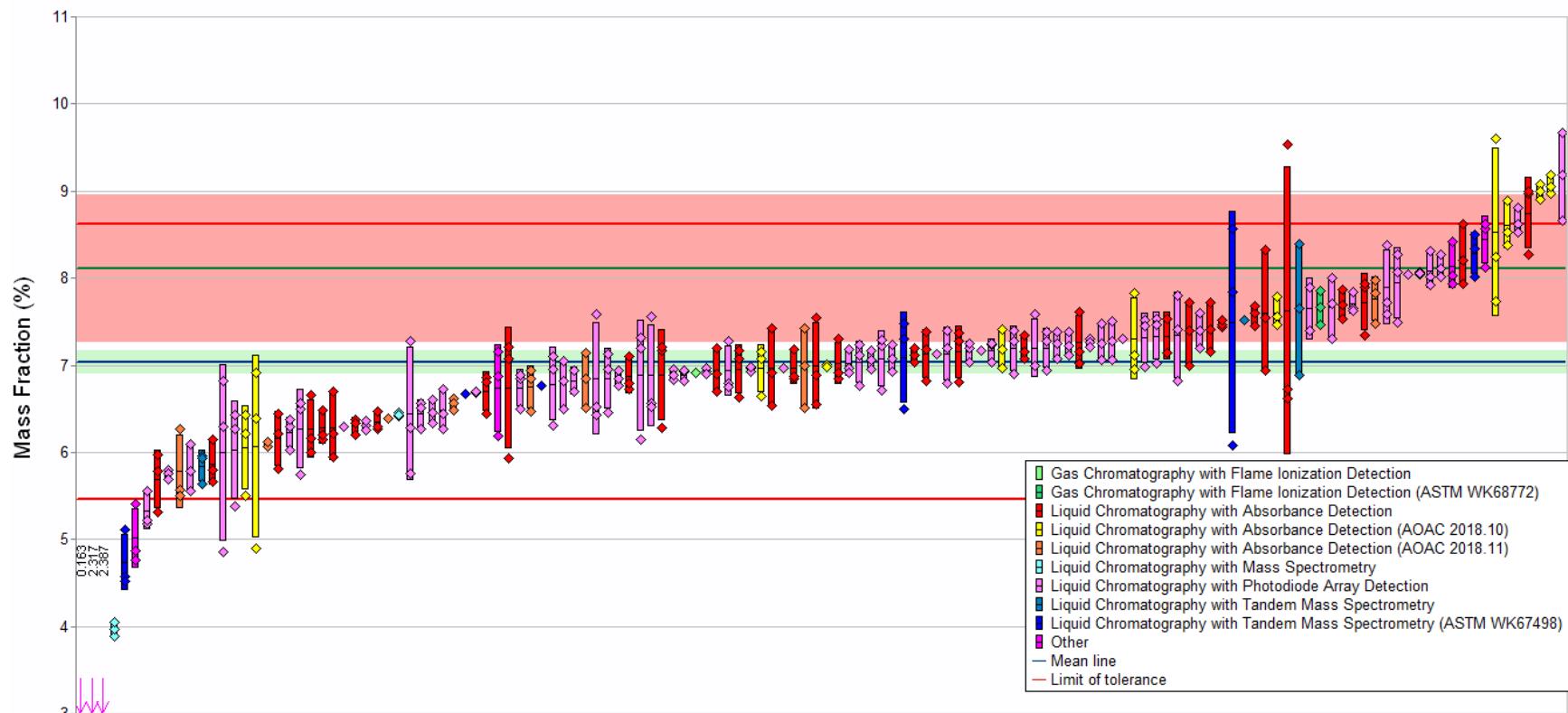
The individual participant, consensus, and target mass fraction results are presented in tabular form in **Appendix B** with examples shown in **Fig. 4-12** through **Fig. 4-17**. Between 78 % and 88 % of participants used either LC-ABS or LC-PDA as their analytical method across all samples and no trend was observed to suggest bias of one method over another. The consensus values for CBDA were between 9 % (NRC HEMP-1) and 14 % (Plant Sample 2) lower than the target values for all but Plant Sample 6, which had a consensus value consistent with the target value. Potential reasons for decreased CBDA mass fractions include decarboxylation of CBDA due to improper sample storage conditions, incomplete extraction, and calibration errors as discussed in Section **3.4.1.4**. The average CBDA value reported by participants using Cerilliant standards was 6.98 % compared to an average CBDA value of 7.13 % for participants reporting use of Cayman standards. The accuracy of analyte measurements depends heavily on the purity of the standards being used for calibration, calibrant range, and the preparation methods of the calibration curve. In lieu of an SI-traceable standard, laboratories should procure standards from multiple manufacturers and make the calibrants from independently prepared stock solutions.

With the exception of NRC HEMP-1 (**Fig. 4-12**) and Plant Sample 6 (**Fig. 4-14**), the consensus ranges for CBDA were below the target ranges. As mentioned previously, NRC HEMP-1 and Plant Sample 6 had the lowest CBDA mass fractions of all plant samples. Depending on the spacing of the calibration curve, the CBDA mass fraction for NRC HEMP-1 and Plant Sample 6 may have been quantitated using a different section of the calibration curve than the other four samples or even in a different dilution. Because of the similarity in percent difference between the target value and consensus value in the higher mass fraction CBDA samples (Plant Sample 4, Plant Sample 2, Plant Sample 3, and Plant Sample 5) and the prevalence of systematic error illustrated in **Fig. 4-11**, the consensus bias is likely a result of the calibration ranges that were used. Details regarding calibration curve and corresponding bias is described in Section **3.4.1.4**.



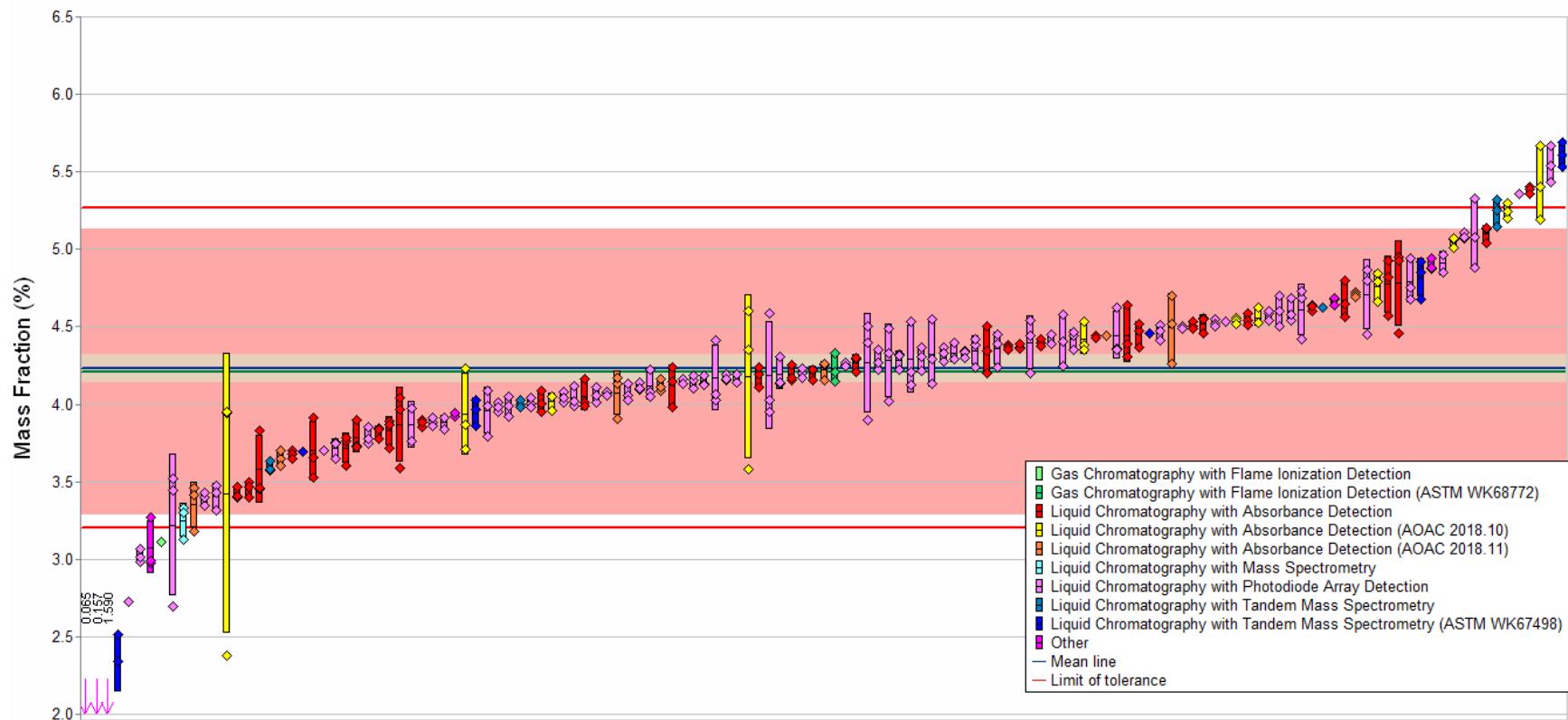
**Fig. 4-12. CBDA in NRC HEMP-1 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



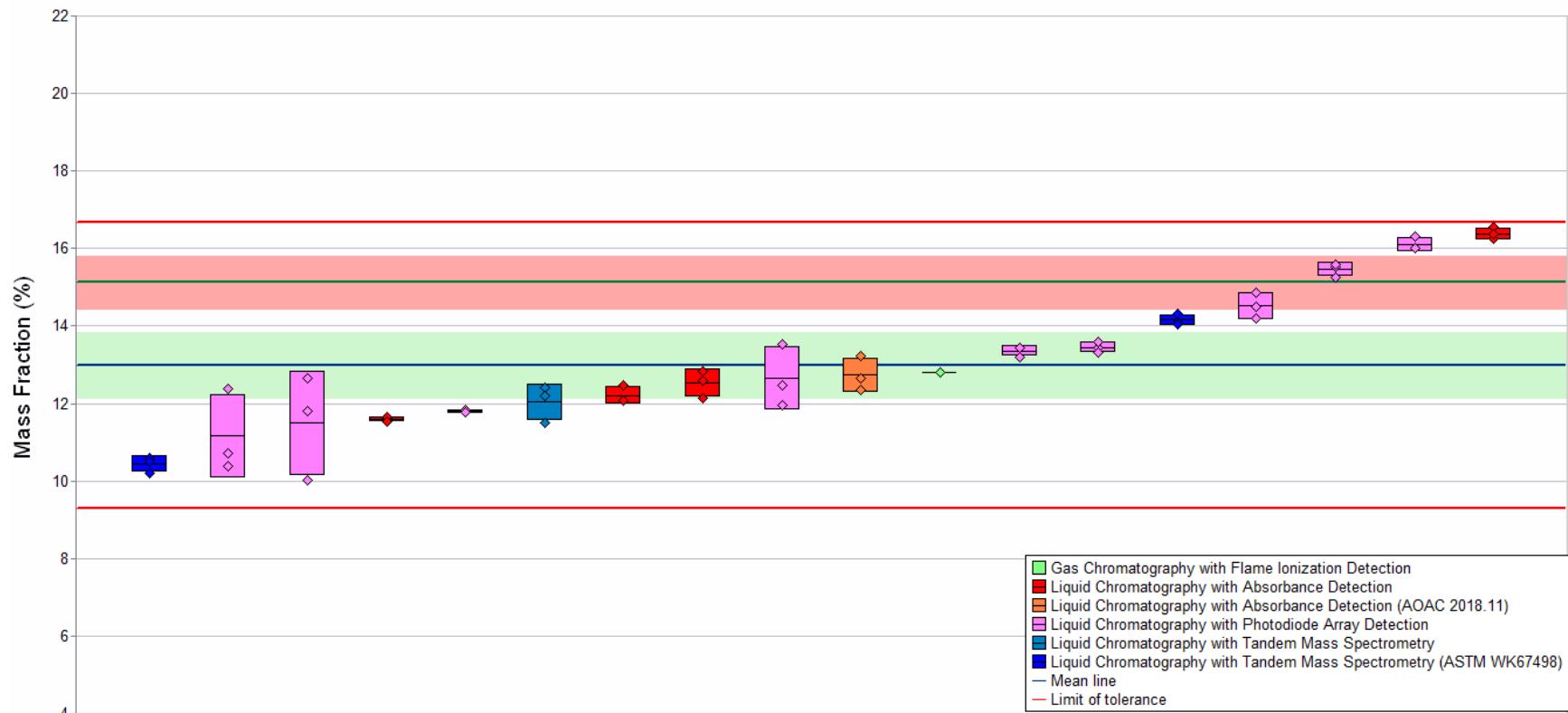
**Fig. 4-13. CBDA in Plant Sample 4 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



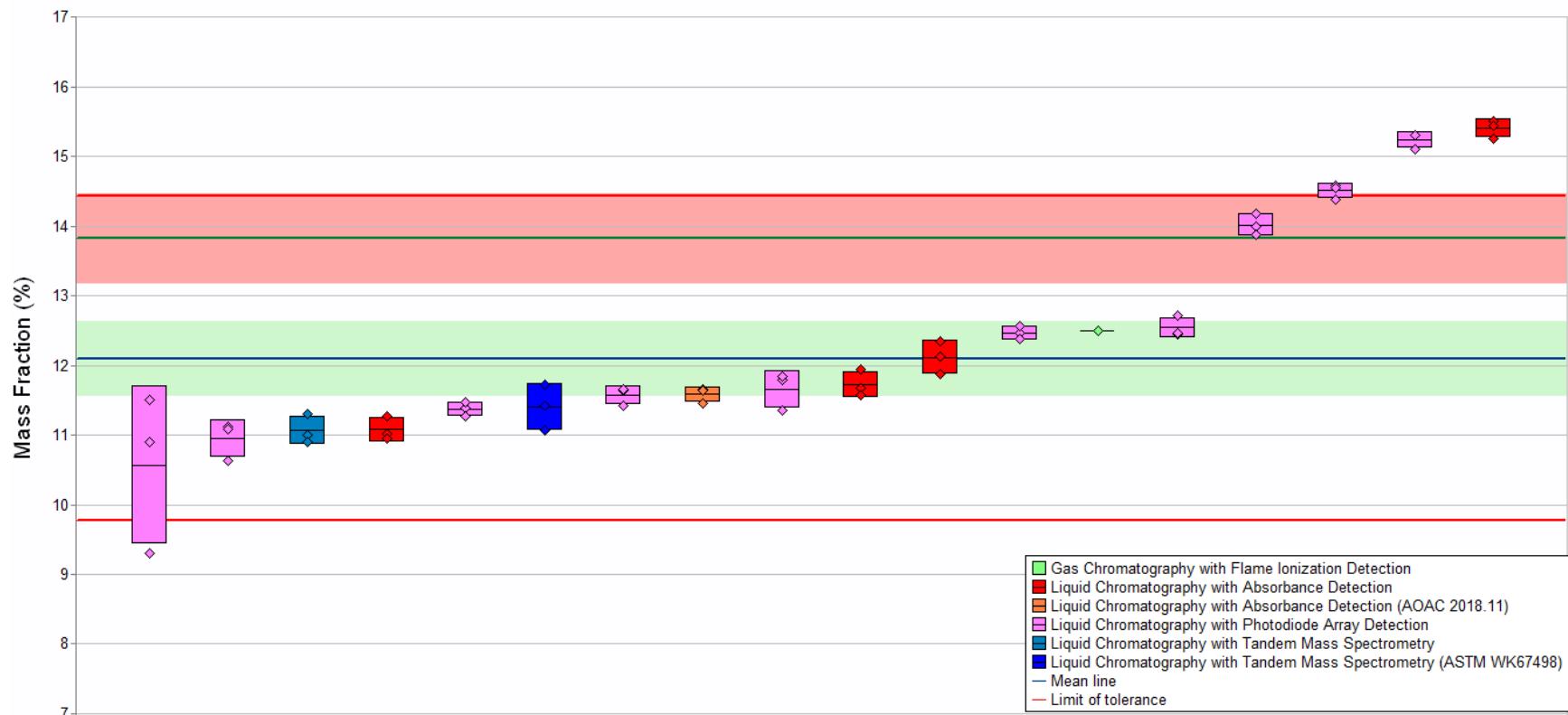
**Fig. 4-14. CBDA in Plant Sample 6 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



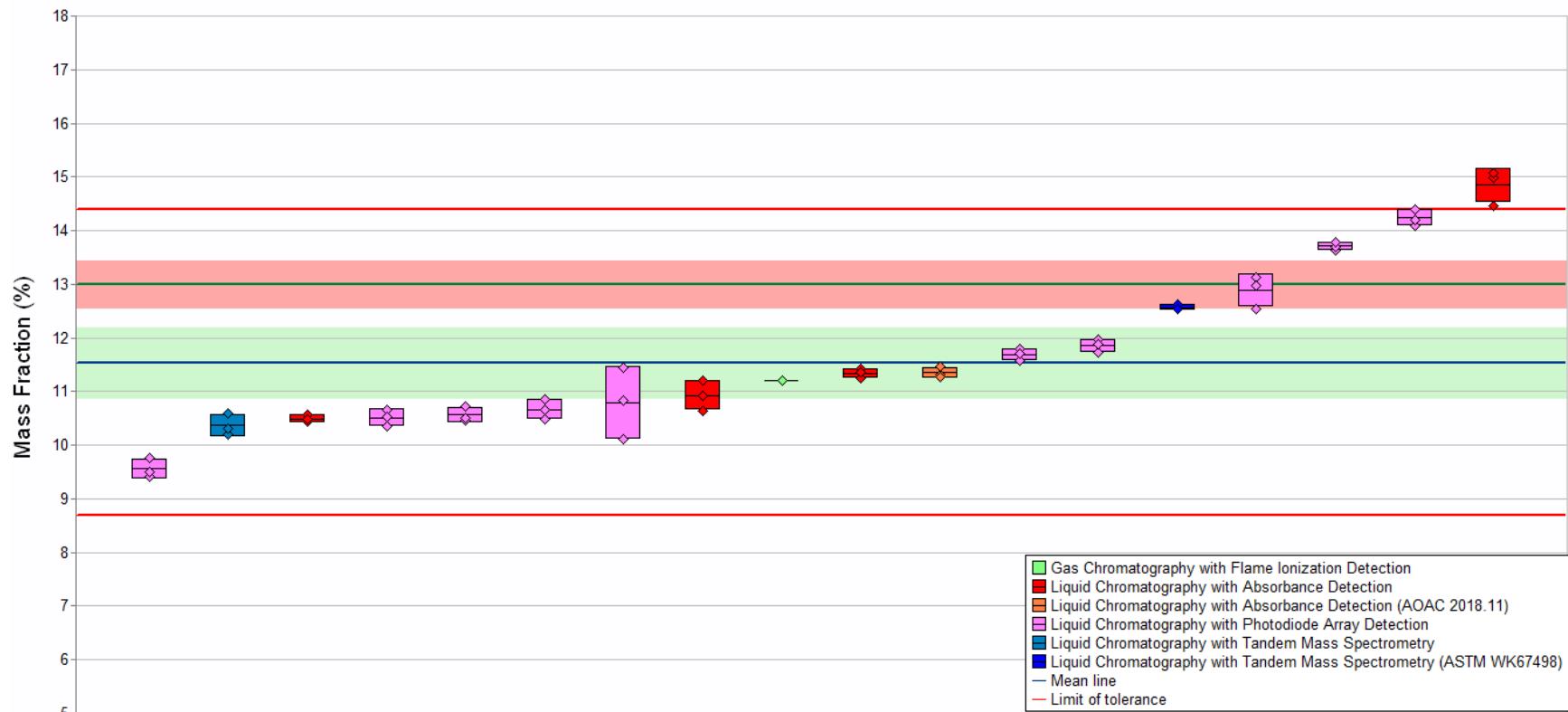
**Fig. 4-15. CBDA in Plant Sample 2 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 4-16. CBDA in Plant Sample 3 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 4-17. CBDA in Plant Sample 5 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

#### 4.4.2.3. Candidate Analytical Methods

NIST provided a list of nine candidate standard methods from AOAC International and ASTM International for participants to use if an in-house analytical method was not available. Mass fractions were submitted by participating laboratories for CBD using an ASTM LC-MS/MS method and two AOAC methods. The within-laboratory (repeatability, %RSD<sub>r</sub>) and between-laboratory (reproducibility, %RSD<sub>R</sub>) variabilities are summarized for candidate method/sample pairs for which participants reported at least two independent measurements for a sample and at least five laboratories reported data (**Table 4-7**). Both AOAC methods were previously approved by an expert review panel using criteria established in SMPR 2017.002 for the quantitation of cannabinoids in dried cannabis plant samples [21], which requires %RSD<sub>r</sub> and %RSD<sub>R</sub> to be  $\leq 4\%$  and  $\leq 5\%$ , respectively, for CBDA when the mass fraction is between 1% and 25%. AOAC has since published SMPR 2019.003 for quantitation of cannabinoids in hemp plant samples [16], which requires %RSD<sub>r</sub> and %RSD<sub>R</sub> to be  $\leq 3\%$  and  $\leq 8\%$  for samples containing  $> 0.5\%$  to 5% CBDA. In samples containing between 5% and 35% CBDA, the requirement is for %RSD<sub>r</sub> and %RSD<sub>R</sub> to be  $\leq 2\%$  and  $\leq 6\%$ , respectively [16]. Because ASTM has not published equivalent requirements, the requirements established in SMPR 2019.003 will be used in this study to evaluate both AOAC and ASTM methods.

**Table 4-7. Within- and between-laboratory variabilities for CBDA measurements using candidate standardized analytical methods.**

	NRC HEMP-1			Plant Sample 4			Plant Sample 6		
	n <sup>a</sup>	%RSD <sub>r</sub>	%RSD <sub>R</sub>	n <sup>a</sup>	%RSD <sub>r</sub>	%RSD <sub>R</sub>	n <sup>a</sup>	%RSD <sub>r</sub>	%RSD <sub>R</sub>
LC-Absorbance									
AOAC 2018.10	11	2.8	13.8	11	3.6	16.9	11	2.2	15.2
AOAC 2018.11	8	1.9	7.2	8	4.5	10.7	8	1.5	14.0
LC-MS/MS									
ASTM WK67498	5	2.9	42.0	5	6.9	27.0	5	3.4	50.6

<sup>a</sup> n = number of laboratories

#### AOAC 2018.10

The %RSD<sub>r</sub> published for AOAC 2018.10 was between 1.61% and 7.24% for CBDA measured in 7 independent dried flower samples (n = 4 for each sample) at concentrations between 3.61% and 8.14% [22], which was within the published requirement of 4% [21] and 3% [16] for 5 of the 7 cannabis samples measured. The average within-laboratory variability observed for NRC HEMP-1 and Plant Sample 6 was within the published variability of the method [22] and SMPR requirement of %RSD<sub>r</sub>  $\leq 3\%$  [16]. The %RSD<sub>r</sub> for Plant Sample 4 measurements was similar to the within-laboratory variabilities in the published method for dried flower samples containing  $> 5\%$  CBDA [22] but was not within the SMPR recommendation ( $\leq 2\%$ , [16]). The CBDA values reported by laboratories using AOAC 2018.10 were lower than the target value for NRC HEMP-1 (5%) and Plant Sample 4 (6.6%) and higher than the target value for Plant Sample 6 (7%). The between-laboratory variabilities observed for CBDA measured by AOAC 2018.10 in all hemp samples were higher than the recommended values [16]. Observed reproducibility of AOAC 2018.10 had not been published at the time of this report.

### AOAC 2018.11

The %RSD<sub>r</sub> published for AOAC 2018.11 was reported from two separate analysts. The combined %RSD<sub>r</sub> was 2.4 % for dried plant material samples containing 3.86 % CBDA ( $n = 10$ , [7]), which was within the published requirements of  $\leq 4\%$  [21] and  $\leq 3\%$  [16]. The average within-laboratory variability for CBDA measurements was consistent with the %RSD<sub>r</sub> published in the method and SMPR for NRC HEMP-1 and Plant Sample 6. The within-laboratory variability for Plant Sample 4 measurements (4.5 %) was above the recommended range of  $\leq 2\%$  [16]. Laboratories using the AOAC 2018.11 method reported CBDA values that were lower than the target value for NRC HEMP-1 (11 %), Plant Sample 4 (18 %), and Plant Sample 6 (2 %). The between-laboratory variabilities observed for CBDA measured in NRC HEMP-1 and Plant Sample 6 using AOAC 2018.11 were above the  $\leq 8\%$  requirement and the RSD<sub>R</sub> for Plant Sample 4 measurements was above the  $\leq 6\%$  requirement [16]. Observed reproducibility of AOAC 2018.11 had not been published at the time of this report.

### ASTM WK67498

The ASTM WK67498 method was developed following the AOAC [23] and ASTM [24] guidelines. The %RSD<sub>r</sub> published for ASTM WK67498 was tested on 5 lots of the same hemp sample with %RSD<sub>r</sub> between 0.7 % and 1.9 % for hemp samples containing 13.8 % to 17.5 % of CBDA ( $n = 3$ , [25]), which were all within the published requirements of  $\leq 2\%$  [16]. The average within-laboratory variability observed for the hemp samples in Exercise 2 was higher than the %RSD<sub>r</sub> published in the method and outside the required %RSD<sub>r</sub> except for CBDA measurements in NRC HEMP-1 [16]. Laboratories using the ASTM WK67498 method reported CBDA values that were lower than the target value for NRC HEMP-1 (8 %), Plant Sample 4 (15 %), and Plant Sample 6 (3 %). The between-laboratory variabilities observed for CBDA measured using ASTM WK67498 in the hemp samples were above the  $\leq 8\%$  requirement for NRC HEMP-1 and Plant Sample 6 and above the  $\leq 6\%$  requirement for Plant Sample 4 [16]. Observed reproducibility of ASTM WK67498 on hemp had not been published at the time of this report.

## 4.4.3. Total CBD

### 4.4.3.1. Within- and Between-Laboratory Precision

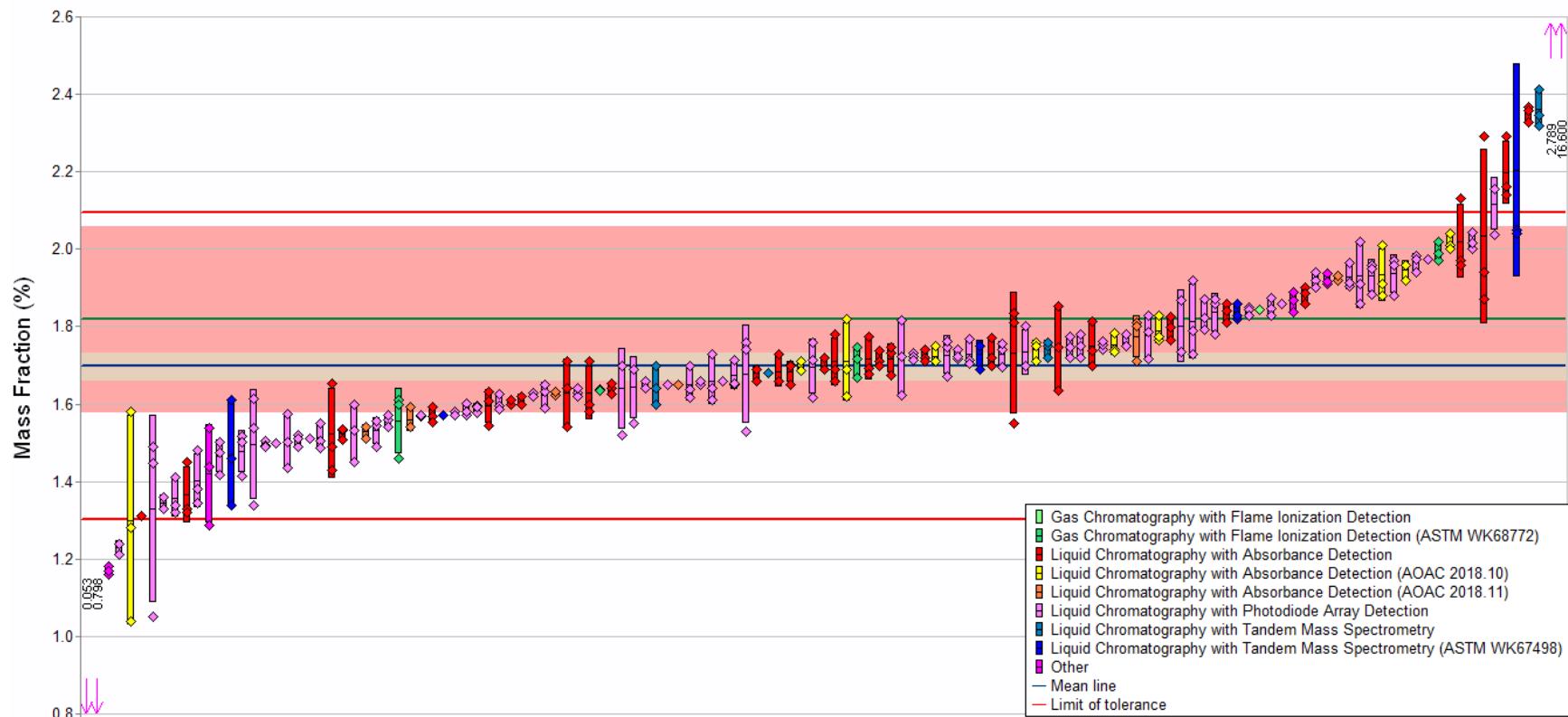
Laboratories reporting quantitative results for total CBD in all samples except for Plant Sample 2 and Plant Sample 4 demonstrated within-laboratory variability ( $\leq 2.2\%$ ) at or below the published expectation for cannabinoids in hemp [16] (**Table 4-4**). The majority of laboratories that analyzed total CBD in hemp (93 %) and marijuana (89 %) reported using LC. Unless a laboratory decarboxylated the CBDA in the samples prior to analysis with liquid chromatography (LC), the total CBD values reported were calculated. For this reason, the within-laboratory variabilities for laboratories that used LC are similar to the variabilities reported for CBD and CBDA. If total CBD is a calculated value, the uncertainty accounting must include the propagation of uncertainties from CBD and CBDA measurements that was not requested in this study. The total CBD measurements made by laboratories reporting use of LC are not described further because they represent a weighted average of the CBD and CBDA uncertainties, which have been discussed in the previous sections.

Only six laboratories reported values for total CBD using non-LC methods, five of which reported use of GC with flame ionization detection (FID) and one laboratory reported use of infrared spectroscopy. Five of the laboratories reported more than one measurement for total CBD in the hemp samples. Of those laboratories, 80 % had within-laboratory variabilities within the AOAC SMPR recommendation [16] for NRC HEMP-1 and Plant Sample 6 and none of the within-laboratory variabilities for Plant Sample 4 met the criteria. Two laboratories reported total CBD values using GC-FID for the marijuana samples. Only one laboratory reported more than one value for total CBD in the marijuana samples with within-laboratory variabilities between 0.8 % and 2.1 % for the three marijuana samples.

The between-laboratory variabilities for total CBD ranged from 9.5 % to 13.6 % for all reported results. Laboratories that reported using GC to measure total CBD in the hemp samples had between-laboratory variabilities ranging from 20.4 % to 45.5 %. Regardless of the analytical method used to assess total CBD mass fractions in the samples, all between-laboratory variabilities were outside the recommended performance criteria [16]; however, the criteria for between-laboratory precision is intended to evaluate multiple laboratories using a single analytical method, not multiple laboratories using multiple analytical methods. The between-laboratory variability for all reported values of total CBD in plant samples for this exercise are comparable to the UK-PT program overall analyte relative standard deviations (6.86 % to 15.4 %) for total CBD in hemp [19]. The laboratories using GC-FID had much higher between-laboratory variabilities, which is likely the result of the variability in CBDA decarboxylation rates in GC inlets between methods. CBDA decarboxylation in GC inlets varies with inlet temperature, liner composition, and frequency of liner replacement [39, 40].

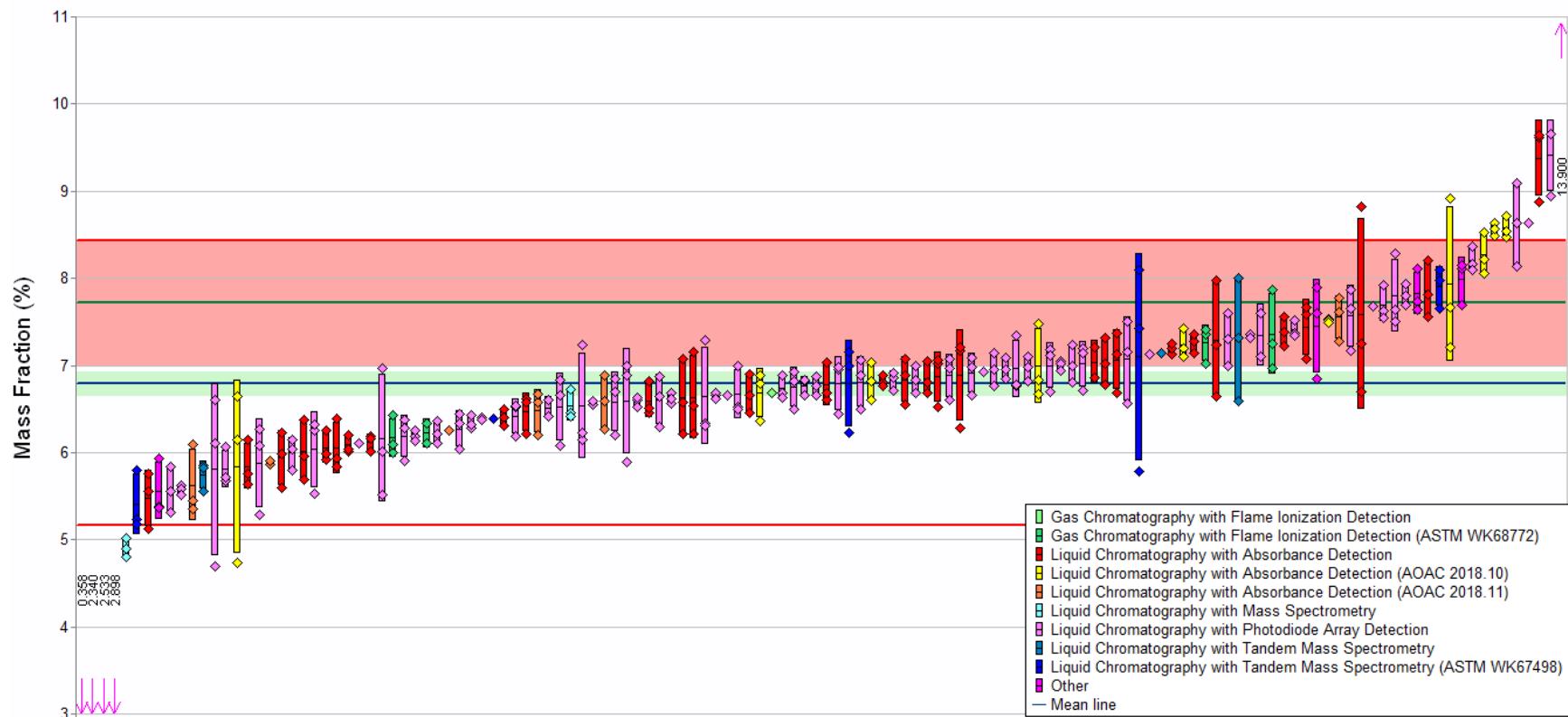
#### 4.4.3.2. Accuracy

The individual participant, consensus, and target mass fraction results are presented in a tabular form in **Appendix B** and graphically in **Fig. 4-18** through **Fig. 4-24**. The consensus values for total CBD were between 7 % (NRC HEMP-1) and 12 % (Plant Sample 5) lower than the target values for all but Plant Sample 6, which had a consensus value approximately 2 % higher than the target value. The accuracy of the total CBD values reported by laboratories using LC methods was directly correlated to their ability to accurately quantify CBD and CBDA. The overall trends in accuracy for total CBD measurements are similar to those for CBDA measurements because CBDA contributed more to the calculated total CBD values than CBD. Similar to the overall data for total CBD, the values for total CBD measurements by laboratories using GC-FID were 4 % to 13 % below the target value. GC methods are expected to underestimate the total CBD mass fraction due to the incomplete decarboxylation of CBDA in the GC inlet [40]. With the exception of NRC HEMP-1 (**Fig. 4-18**) and Plant Sample 6 (**Fig. 4-21**), the consensus ranges for total CBD were outside the target ranges, as was the case for CBDA. While the GC-derived and the overall consensus value for total CBD were similar for Plant Sample 4, the GC-derived consensus range partially overlapped with the target range due to the larger between-laboratory variability for laboratories using GC-FID (**Fig. 4-20**).



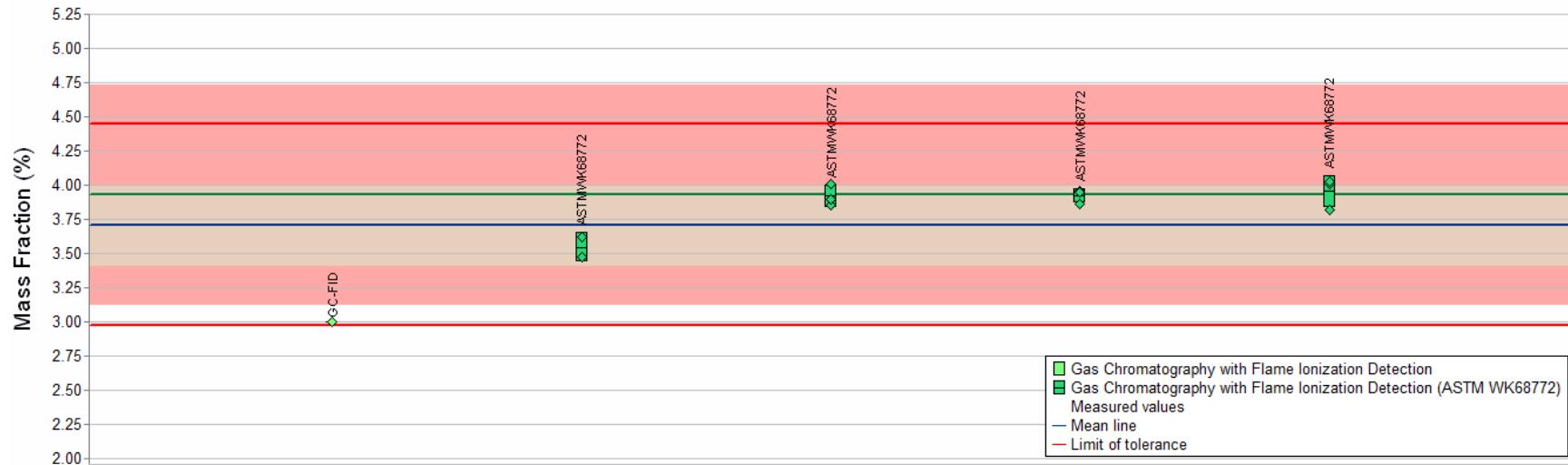
**Fig. 4-18. Total CBD in NRC HEMP-1 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 4-19. Total CBD in Plant Sample 4 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 4-20. Total CBD in Plant Sample 4 (data summary view – GC methods).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

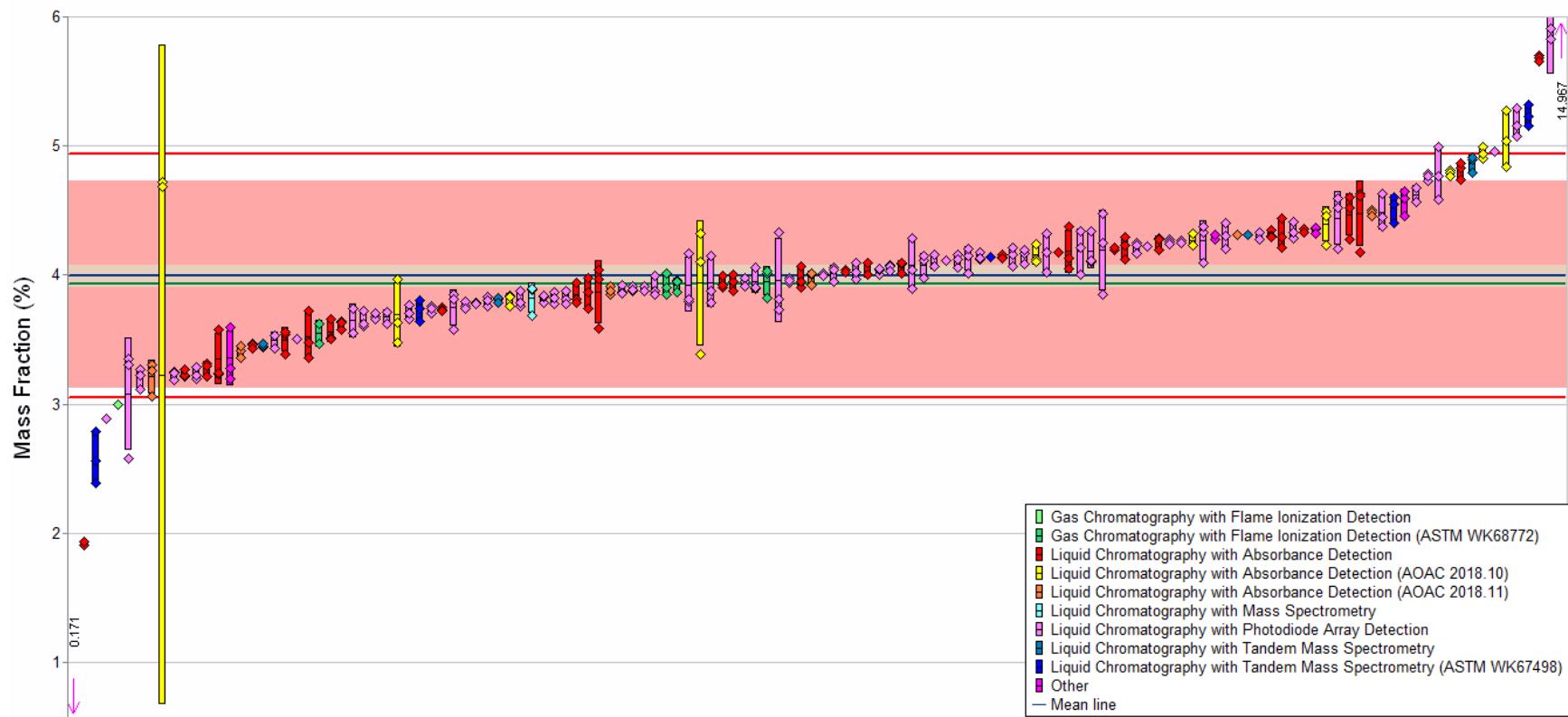
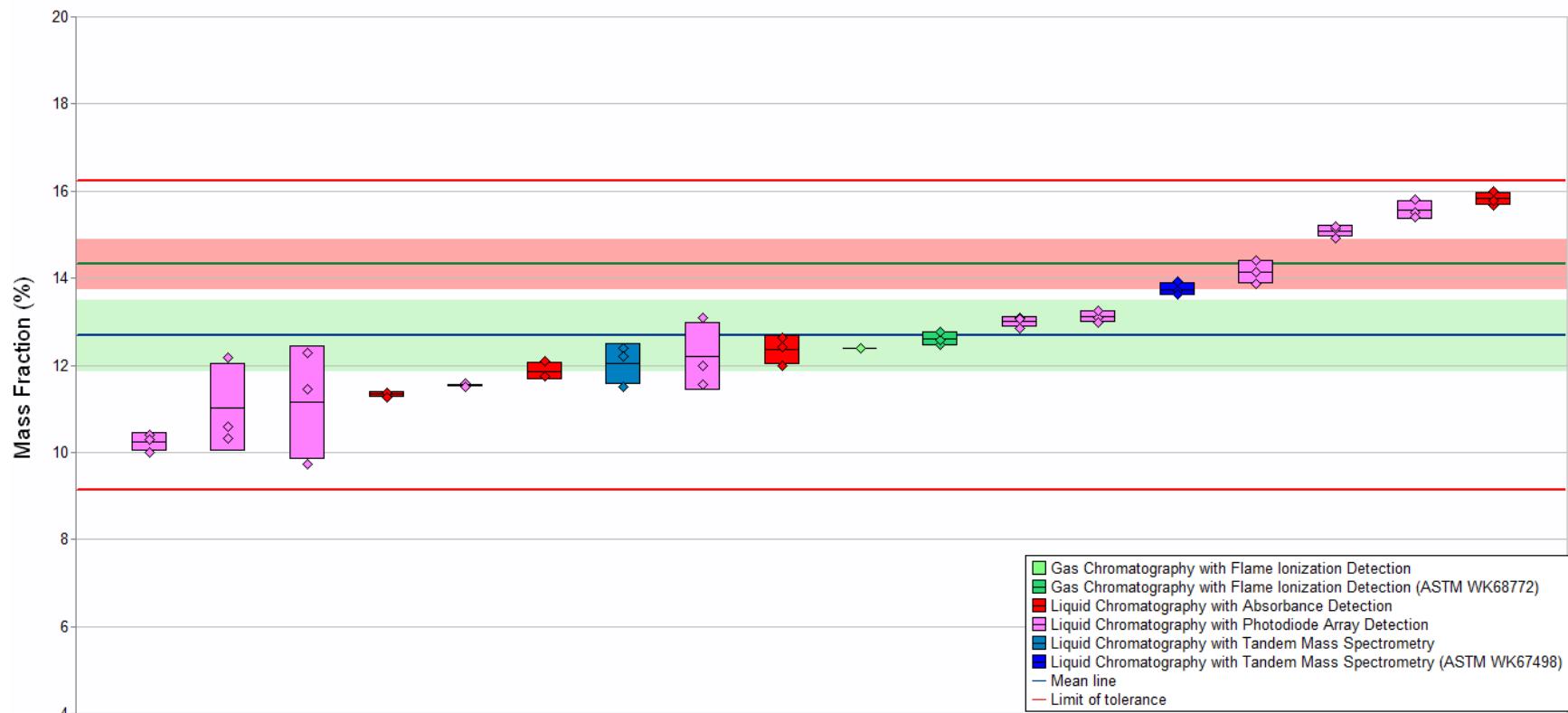


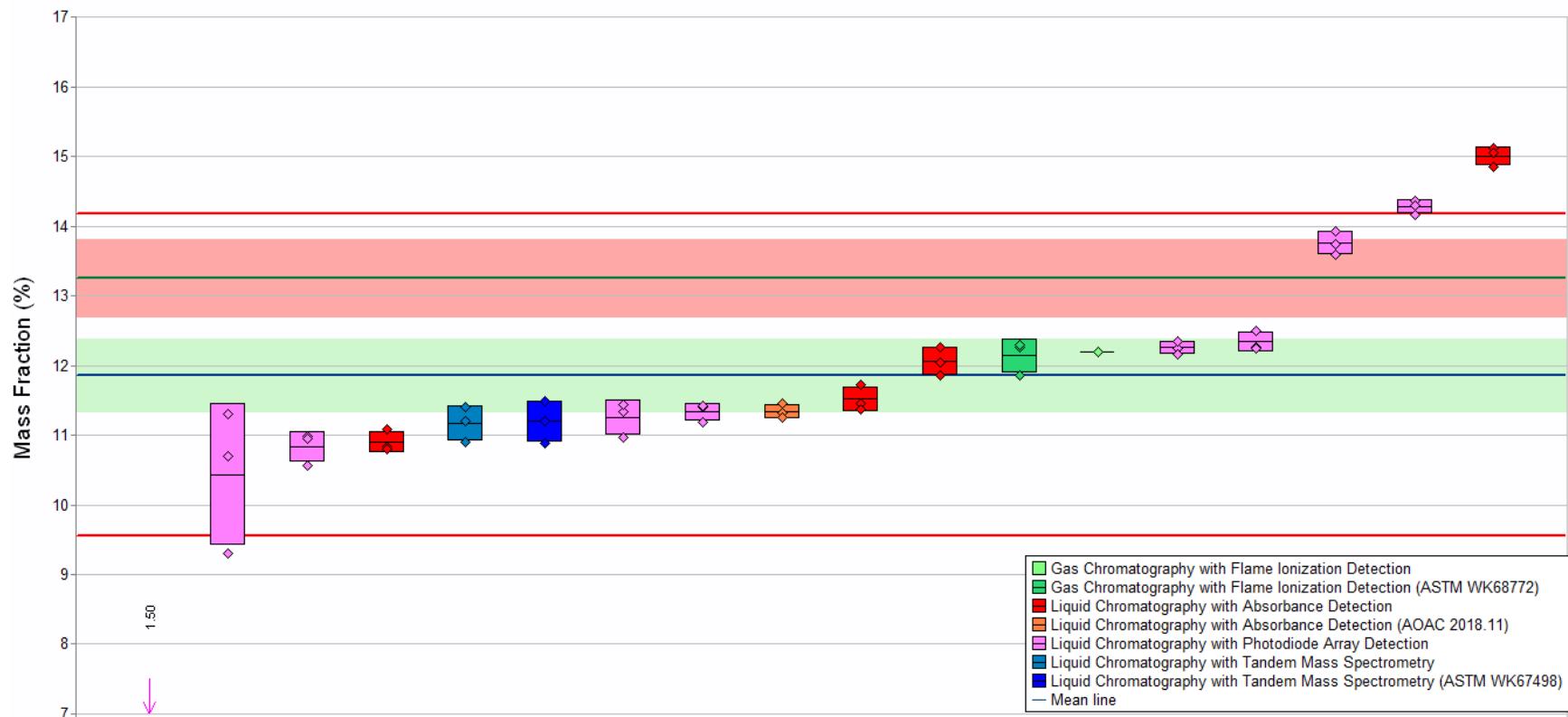
Fig. 4-21. Total CBD in Plant Sample 6 (data summary view – analytical method).

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



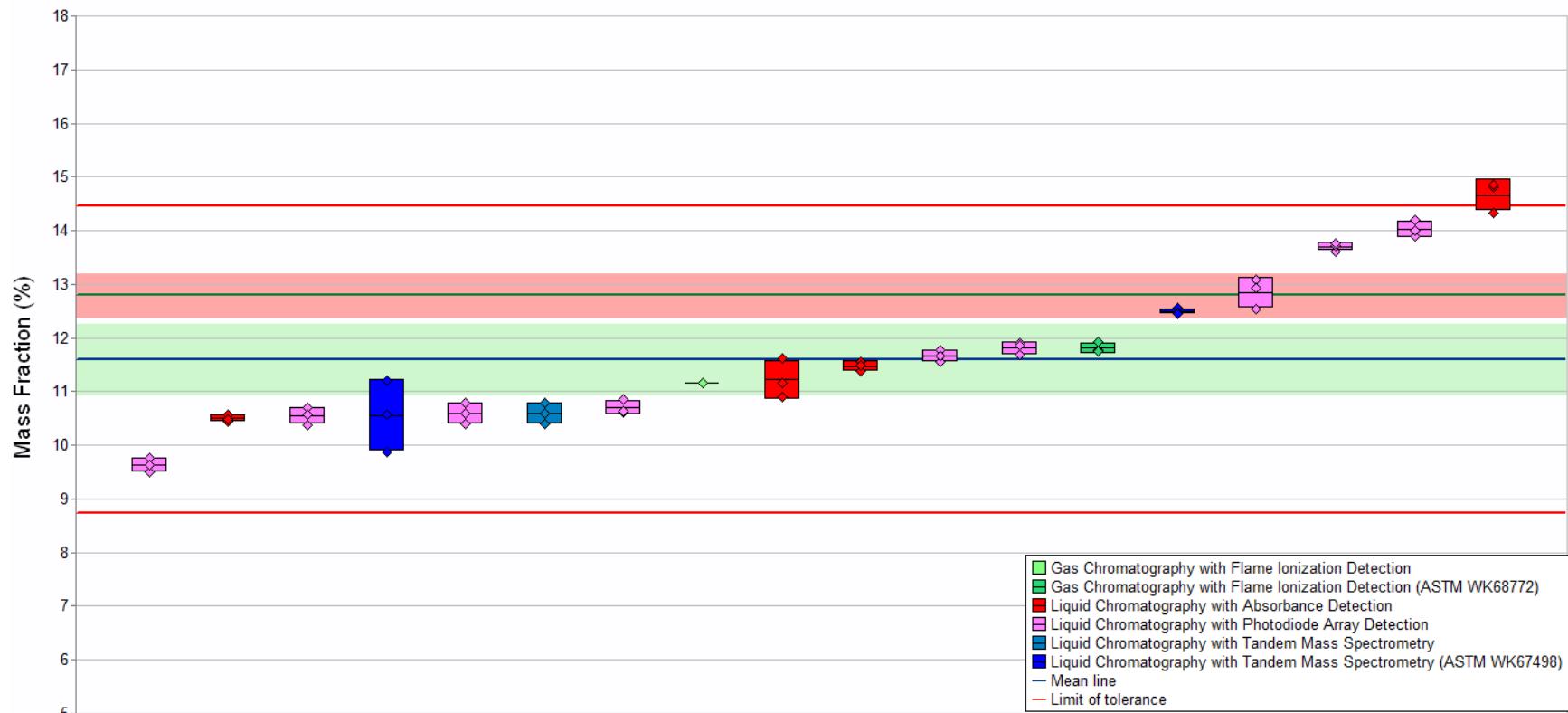
**Fig. 4-22. Total CBD in Plant Sample 2 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 4-23. Total CBD in Plant Sample 3 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 4-24. Total CBD in Plant Sample 5 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

#### 4.5. Conclusions

Laboratories that participated in Exercise 2 of CannaQAP successfully demonstrated the ability to measure CBD in the six plant samples, with consensus values within 15 % of the target values for all samples. Consensus values for CBDA were within 14 % of the target value for all plant samples; however, the consensus ranges for Plant Sample 4, Plant Sample 2, Plant Sample 3, and Plant Sample 5 were completely outside the target range. When this occurs, the consensus value is considered significantly different than the target value. Because CBD and to a greater extent CBDA concentrations in the sample extracts were likely outside the calibration range, reported values relied upon precise pipetting for dilution and correct accounting of the dilution factor in the calculation. Because sample dilutions performed volumetrically are less accurate than those done gravimetrically, participants should consider diluting samples by mass. If laboratories were using calibration curves that extended multiple orders of magnitude to quantify CBDA, the accuracy of the CBDA measurements would have been reduced compared to use of a more specific calibration curve. Laboratories should use calibration curves that closely represent the extracted mass fraction of CBD or CBDA in the samples to increase the accuracy of the analysis or dilute high-concentration samples to decrease their mass fractions to be within the calibration range. Additional steps laboratories can take to prevent calibration bias include independently preparing calibrants and ensuring standards are traceable to the SI, if possible. The overall total CBD measurements followed accuracy and precision trends for CBD and CBDA because the majority of laboratories used calculated values. For laboratories using GC-FID to determine total CBD, the total CBD values were generally lower than the overall CBD values, which indicates incomplete CBDA decarboxylation in the inlet. In addition to incomplete CBDA decarboxylation decreasing the accuracy of total CBD measurement, the variability in decarboxylation rates in the GC inlets also increased the between-laboratory precision. If the sample preparation does not include decarboxylation prior to extraction, the GC method should be evaluated for decarboxylation rates as part of the method validation.

## 5. Minor Cannabinoids

### 5.1. Study Overview

Potential health benefits and therapeutic applications have generated increasing interest in the non-psychoactive minor cannabinoids CBC, CBDV, CBG, CBN, and THCV, including anti-convulsant (CBC, CBDV, CBG, CBN, THCV), anti-inflammatory (CBG, CBN), antibacterial (CBN), cancer cell suppression (CBC, CBG), and neuroprotective (CBC, CBDV, CBG, CBN, and THCV) properties [41, 42]. Minor cannabinoids are typically present in the cannabis plant in their acidic forms, which undergo decarboxylation into the neutral forms after exposure to heat and/or light [42]. The two primary minor cannabinoid precursors are CBGA or CBGVA [43]; CBGA is enzymatically converted to CBCA, CBDA, and THCA and CBGVA is the precursor to CBDVA and THCVA. Due to the therapeutic properties of CBG, CBDV, and THCV and their low abundance in cannabis flowers, breeders have been developing strains with higher concentrations of CBGA, CBDVA, and THCVA [42]. Minimal research has been conducted on CBL and CBLA, which are often challenging to detect at extremely low levels in cannabis plants [44, 45]. CBL is not believed to be a decarboxylation product of CBLA, but as a degradation product of CBC during long-term storage or exposure to heat and/or light. Similarly, CBN is not formed through decarboxylation of CBNA, but instead CBN and CBNA are formed through the oxidation of  $\Delta^9$ -THC and THCA, respectively [46]. Reliable analytical methods for all minor cannabinoids will be a requirement as medicinal and recreational use of cannabis and cannabis-derived products continues to increase across the US. The most recent example of exploitation of a minor cannabinoid is in  $\Delta^8$ -THC specific products, which have similar psychoactive properties to cannabis-derived products containing  $\Delta^9$ -THC [47].  $\Delta^8$ -THC is an isomer of  $\Delta^9$ -THC that exists naturally in cannabis plant at extremely low levels. Recently, availability of specialized  $\Delta^8$ -THC containing cannabis products (e.g., smokable flower, vapes, edibles, etc.) has increased due to the development of a process to convert CBD into  $\Delta^8$ -THC and other cannabinoids [48]. Additionally, the chromatographic behavior and mass spectral fingerprint of  $\Delta^8$ -THC are similar to those of  $\Delta^9$ -THC, which has increased the need for laboratories to ensure that their analytical methods can appropriately distinguish between the two isomers preventing erroneous reporting of the presence of  $\Delta^9$ -THC.

Because of the increase in research and consumption of minor cannabinoids, participants in this study were asked to use in-house analytical methods to determine the mass fractions (%) of CBC, CBCA, CBDV, CBDVA, CBG, CBGA, CBL, CBLA, CBN, CBNA, THCA, THCVA, and  $\Delta^8$ -THC in three hemp plant and/or three marijuana plant samples. Through participation in this study, laboratories will be able to better understand the performance of their in-house methods relative to those being used by others in the community. Laboratories will also be able to use this data as part of their in-house method validation and/or as an analytical comparison to NIST values. Participant results will be used by NIST to gain knowledge on important challenges facing analytical methodologies in measuring minor cannabinoids in cannabis plant samples. In addition, NIST will use the results from this study to help in the design of specific studies with minor cannabinoids becoming more prevalent in cannabis plant samples and/or cannabis derived products (e.g., CBG, CBGA, and  $\Delta^8$ -THC).

## 5.2. Sample Information

The target values and uncertainties for minor cannabinoids in the hemp and marijuana samples are provided in **Table 5-1** through **Table 5-3** on an as-received basis. The target values and uncertainties for NRC HEMP-1 were determined at NRC Canada and taken from the HEMP-1 COA [6]. The target values and uncertainties in the remaining five cannabis plant samples were determined at NIST as described in Section 2. Participants were not given information regarding the mass fractions of minor cannabinoids in the samples prior to the study.

**Table 5-1. Example individualized data summary table for CBC, CBCA, CBDV, and CBDVA.**

Laboratory-specific results and Z-scores were provided to each participant separately from this report to protect laboratory identities.

*(Lab Name)*

### Exercise 2 – Cannabinoids in Cannabis Plant Samples

Lab Code:	(Code)	1. Your Results				2. Community Results			3. Target			
		Sample	Units	$x_i$	$s_i$	$Z'_{\text{comm}}$	$Z_{\text{NIST}}$	N	$x^*$	$s^*$	$x_{\text{NIST}}$	$u$
CBC	NRC HEMP-1	%						87	0.0309	0.0055	0.0325	0.0084
CBC	Plant Sample 2	%						14	0.121	0.022	0.1181	0.0072
CBC	Plant Sample 3	%						14	0.130	0.019	0.1207	0.0034
CBC	Plant Sample 4	%						100	0.056	0.013	0.0522	0.0042
CBC	Plant Sample 5	%						14	0.146	0.020	0.121	0.046
CBC	Plant Sample 6	%						82	0.0249	0.0081	0.0223	0.0028
CBCA	NRC HEMP-1	%						45	0.047	0.015	0.045	0.011
CBCA	Plant Sample 2	%						9	0.62	0.15		
CBCA	Plant Sample 3	%						9	0.59	0.16		
CBCA	Plant Sample 4	%						58	0.374	0.041		
CBCA	Plant Sample 5	%						9	0.55	0.13		
CBCA	Plant Sample 6	%						57	0.206	0.028		
CBDV	NRC HEMP-1	%						56	0.027	0.019	0.0188	0.0032
CBDV	Plant Sample 2	%						3	0.083	0.035		
CBDV	Plant Sample 3	%						3	0.078	0.027		
CBDV	Plant Sample 4	%						29	0.018	0.022		
CBDV	Plant Sample 5	%						4	0.088	0.046		
CBDV	Plant Sample 6	%						25	0.014	0.015		
CBDVA	NRC HEMP-1	%						58	0.084	0.020	0.0719	0.0054
CBDVA	Plant Sample 2	%						8	0.079	0.013		
CBDVA	Plant Sample 3	%						8	0.0730	0.0097		
CBDVA	Plant Sample 4	%						52	0.0388	0.0085		
CBDVA	Plant Sample 5	%						9	0.075	0.016		
CBDVA	Plant Sample 6	%						50	0.0260	0.0070		

$x_i$  Mean of reported values

$s_i$  Standard deviation of reported values

$Z'_{\text{comm}}$  Z'-score with respect to community consensus

$Z_{\text{NIST}}$  Z-score with respect to NIST value

N Number of quantitative values reported

$x^*$  Robust mean of reported values

$s^*$  Robust standard deviation

$x_{\text{NIST}}$  NIST-assessed value

$u$  standard uncertainty about the NIST-assessed value and assigned values by NRC Canada for HEMP-1

**Table 5-2. Example individualized data summary table for CBG, CBGA, CBL, and CBLA.**

Laboratory-specific results and Z-scores were provided to each participant separately from this report to protect laboratory identities.

**(Lab Name)**

**Exercise 2 – Cannabinoids in Cannabis Plant Samples**

Lab Code:	(Code)	1. Your Results				2. Community Results			3. Target			
		Sample	Units	$x_i$	$s_i$	$Z^{\text{comm}}$	$Z_{\text{NIST}}$	N	$x^*$	$s^*$	$x_{\text{NIST}}$	$u$
CBG	NRC HEMP-1	%						41	0.0122	0.0090	0.00478	0.00094
CBG	Plant Sample 2	%						10	0.057	0.015		
CBG	Plant Sample 3	%						11	0.074	0.016		
CBG	Plant Sample 4	%						70	0.0255	0.0082		
CBG	Plant Sample 5	%						12	0.081	0.020	0.0856	0.0010
CBG	Plant Sample 6	%						42	0.0104	0.0048		
CBGA	NRC HEMP-1	%						16	0.0128	0.0043	0.0117	0.0012
CBGA	Plant Sample 2	%						12	0.324	0.076		
CBGA	Plant Sample 3	%						12	0.349	0.042		
CBGA	Plant Sample 4	%						44	0.160	0.036		
CBGA	Plant Sample 5	%						12	0.369	0.045		
CBGA	Plant Sample 6	%						40	0.067	0.018		
CBL	NRC HEMP-1	%						17	0.0094	0.0040	0.0074	0.0014
CBL	Plant Sample 2	%						1				
CBL	Plant Sample 3	%						1				
CBL	Plant Sample 4	%						12	0.012	0.015		
CBL	Plant Sample 5	%						2	0.038	0.063		
CBL	Plant Sample 6	%						10	0.0013	0.0017		
CBLA	NRC HEMP-1	%						15	0.032	0.014	0.0187	0.0018
CBLA	Plant Sample 2	%						3	0.047	0.054		
CBLA	Plant Sample 3	%						3	0.043	0.037		
CBLA	Plant Sample 4	%						12	0.013	0.012		
CBLA	Plant Sample 5	%						3	0.054	0.062		
CBLA	Plant Sample 6	%						13	0.0114	0.0089		

$x_i$  Mean of reported values  
 $s_i$  Standard deviation of reported values  
 $Z^{\text{comm}}$   $Z'$ -score with respect to community consensus  
 $Z_{\text{NIST}}$   $Z$ -score with respect to NIST value

$N$  Number of quantitative values reported  
 $x^*$  Robust mean of reported values  
 $s^*$  Robust standard deviation

$x_{\text{NIST}}$  NIST-assessed value  
 $u$  standard uncertainty about the NIST-assessed value and assigned values by NRC Canada for HEMP-1

**Table 5-3. Example individualized data summary table for CBN, CBNA, THCV, THCVA, and  $\Delta^8$ -THC.**

Laboratory-specific results and Z-scores were provided to each participant separately from this report to protect laboratory identities.

*(Lab Name)*

**Exercise 2 – Cannabinoids in Cannabis Plant Samples**

Lab Code: <b>(Code)</b>	Sample	Units	<b>1. Your Results</b>				<b>2. Community Results</b>			<b>3. Target</b>	
			$x_i$	$s_i$	$Z'_{\text{comm}}$	$Z_{\text{NIST}}$	N	$x^*$	$s^*$	$x_{\text{NIST}}$	$u$
CBN	NRC HEMP-1	%					116	0.0485	0.0058	0.0490	0.0070
CBN	Plant Sample 2	%					6	0.0111	0.0093		
CBN	Plant Sample 3	%					11	0.0312	0.0077	0.0299	0.0020
CBN	Plant Sample 4	%					27	0.0017	0.0030		
CBN	Plant Sample 5	%					15	0.0679	0.0082	0.0647	0.0020
CBN	Plant Sample 6	%					24	0.0013	0.0020		
CBNA	NRC HEMP-1	%					33	0.0370	0.0082	0.0350	0.0036
CBNA	Plant Sample 2	%					3	0.0217	0.0050		
CBNA	Plant Sample 3	%					7	0.051	0.032		
CBNA	Plant Sample 4	%					16	0.0057	0.0054		
CBNA	Plant Sample 5	%					7	0.078	0.031		
CBNA	Plant Sample 6	%					19	0.0070	0.0052		
THCV	NRC HEMP-1	%	<i>Individual laboratory results will appear in this section; laboratory-specific results were provided to each participant separately from this report.</i>				22	0.0036	0.0051	0.00143	0.00020
THCV	Plant Sample 2	%					5	0.043	0.029		
THCV	Plant Sample 3	%					4	0.032	0.016		
THCV	Plant Sample 4	%					23	0.0066	0.0095		
THCV	Plant Sample 5	%					3	0.0259	0.0059		
THCV	Plant Sample 6	%					18	0.0004	0.0015		
THCVA	NRC HEMP-1	%					18	0.0081	0.0023	0.00728	0.00064
THCVA	Plant Sample 2	%					2	0.079	0.147		
THCVA	Plant Sample 3	%					4	0.0195	0.0020		
THCVA	Plant Sample 4	%					15	0.0052	0.0061		
THCVA	Plant Sample 5	%					4	0.0145	0.0026		
THCVA	Plant Sample 6	%					11	0.0024	0.0026		
$\Delta^8$ -THC	NRC HEMP-1	%					27	0.013	0.013		
$\Delta^8$ -THC	Plant Sample 2	%					2	0.07	0.19		
$\Delta^8$ -THC	Plant Sample 3	%					2	0.07	0.21		
$\Delta^8$ -THC	Plant Sample 4	%					17	0.0014	0.0027		
$\Delta^8$ -THC	Plant Sample 5	%					2	0.06	0.18		
$\Delta^8$ -THC	Plant Sample 6	%					17	0.0013	0.0030		

$x_i$  Mean of reported values

$s_i$  Standard deviation of reported values

$Z'_{\text{comm}}$  Z'-score with respect to community consensus

$Z_{\text{NIST}}$  Z-score with respect to NIST value

N Number of quantitative values reported

$x^*$  Robust mean of reported values

$s^*$  Robust standard deviation

$x_{\text{NIST}}$  NIST-assessed value

$u$  standard uncertainty about the NIST-assessed value and assigned values by NRC Canada for HEMP-1

### 5.3. Reporting Statistics

The enrollment and reporting statistics for minor cannabinoids are described in **Table 5-4** for six cannabis plant samples. A total of 226 laboratories registered to participate in Exercise 2 of CannaQAP with approximately 76 % of participants signing up to report values for at least one minor cannabinoid in the hemp samples (NRC HEMP-1, Plant Sample 4, and Plant Sample 6) and 12 % of participants signing up to report values for at least one minor cannabinoid in the marijuana samples (Plant Sample 2, Plant Sample 3, and Plant Sample 5). The percentage of laboratories that requested samples and returned results for minor cannabinoids was between 38 % and 85 % for hemp samples and between 55 % and 73 % for marijuana samples. Participants were asked to either submit quantitative mass fractions or qualitative results based on LOQs (**Table 5-5**). The minor cannabinoid mass fractions in the three hemp samples were one to four orders of magnitude lower than CBD and CBDA, which is why fewer number of participants were able to report quantitative results.

**Table 5-4. Reporting statistics for the enrollment to measure minor cannabinoids.**

<u>Analyte</u>	<u>Number of Participants</u>	<u>Percent Reporting Results (%) for Hemp Samples</u>		
		<u>NRC HEMP-1</u>	<u>Plant Sample 4</u>	<u>Plant Sample 6</u>
CBC	152	76.3	77.0	75.7
CBCA	85	69.4	69.4	69.4
CBDV	135	74.1	71.9	71.1
CBDVA	88	70.5	70.5	69.3
CBG	159	71.1	74.2	72.3
CBGA	53	77.4	84.9	83.0
CBL	69	65.2	65.2	65.2
CBLA	53	43.4	37.7	37.7
CBN	171	76.6	69.0	69.0
CBNA	69	60.9	56.5	56.5
THCV	138	69.6	69.6	68.8
THCVA	82	64.6	65.9	64.6
$\Delta^8\text{-THC}$	143	72.7	69.9	69.9

<u>Analyte</u>	<u>Number of Participants</u>	<u>Percent Reporting Results (%) for Marijuana Samples</u>		
		<u>Plant Sample 2</u>	<u>Plant Sample 3</u>	<u>Plant Sample 5</u>
CBC	23	60.9	60.9	63.6
CBCA	14	64.3	64.3	69.2
CBDV	20	65.0	65.0	68.4
CBDVA	14	64.3	64.3	69.2
CBG	24	62.5	62.5	65.2
CBGA	19	63.2	63.2	66.7
CBL	12	66.7	66.7	72.7
CBLA	11	54.5	54.5	60.0
CBN	29	58.6	58.6	60.7
CBNA	12	58.3	58.3	63.6
THCV	21	66.7	66.7	70.0
THCVA	15	60.0	60.0	64.3
$\Delta^8\text{-THC}$	24	62.5	62.5	65.2

**Table 5-5. Percent of reporting laboratories reporting quantitative mass fractions for minor cannabinoids.**

<u>Analyte</u>	<u>NRC HEMP-1 (%)</u>	<u>Plant Sample 4 (%)</u>	<u>Plant Sample 6 (%)</u>
CBC	75.0	85.5	71.3
CBCA	76.3	98.3	96.6
CBDV	56.0	29.9	26.0
CBDVA	93.5	83.9	82.0
CBG	36.3	59.3	36.5
CBGA	39.0	97.8	90.9
CBL	37.8	26.7	22.2
CBLA	65.2	60.0	65.0
CBN	88.5	22.9	20.3
CBNA	78.6	41.0	48.7
THCV	22.9	24.0	18.9
THCVA	34.0	27.8	20.8
$\Delta^8$ -THC	26.0	17.0	17.0

<u>Analyte</u>	<u>Plant Sample 2 (%)</u>	<u>Plant Sample 3 (%)</u>	<u>Plant Sample 5 (%)</u>
CBC	100	100	100
CBCA	100	100	100
CBDV	23.1	23.1	30.8
CBDVA	88.9	88.9	100
CBG	66.7	73.3	80.0
CBGA	100	100	100
CBL	12.5	12.5	25.0
CBLA	50.0	50.0	50.0
CBN	35.3	64.7	88.2
CBNA	42.9	100	100
THCV	35.7	28.6	21.4
THCVA	22.2	44.4	44.4
$\Delta^8$ -THC	13.3	13.3	13.3

Most laboratories used a combination of solvent extraction for sample preparation and LC with either absorbance or photodiode array detection for analysis (**Table 5-6**). The majority of laboratories reported use of solvent extraction (between 91 % and 95 %) and the remaining laboratories that reported use of dilution also likely used solvent extraction prior to dilution because the samples were all plant material. Due to the lack of data on actual solvents used for extraction as well as lack of detailed extraction procedures, no trends based on sample preparation were able to be assessed unless a candidate method was used. The results and discussion presented here focused on comparability of different instrumental methods, chemical interferences, and calibration approaches. Additional information on participant sample preparation and LC instrumental methods are provided in **Appendix C** based on responses to a method questionnaire completed by 93 participants.

**Table 5-6. Percent of laboratories reporting specific sample preparation and analytical methods for the determination of minor cannabinoids.**

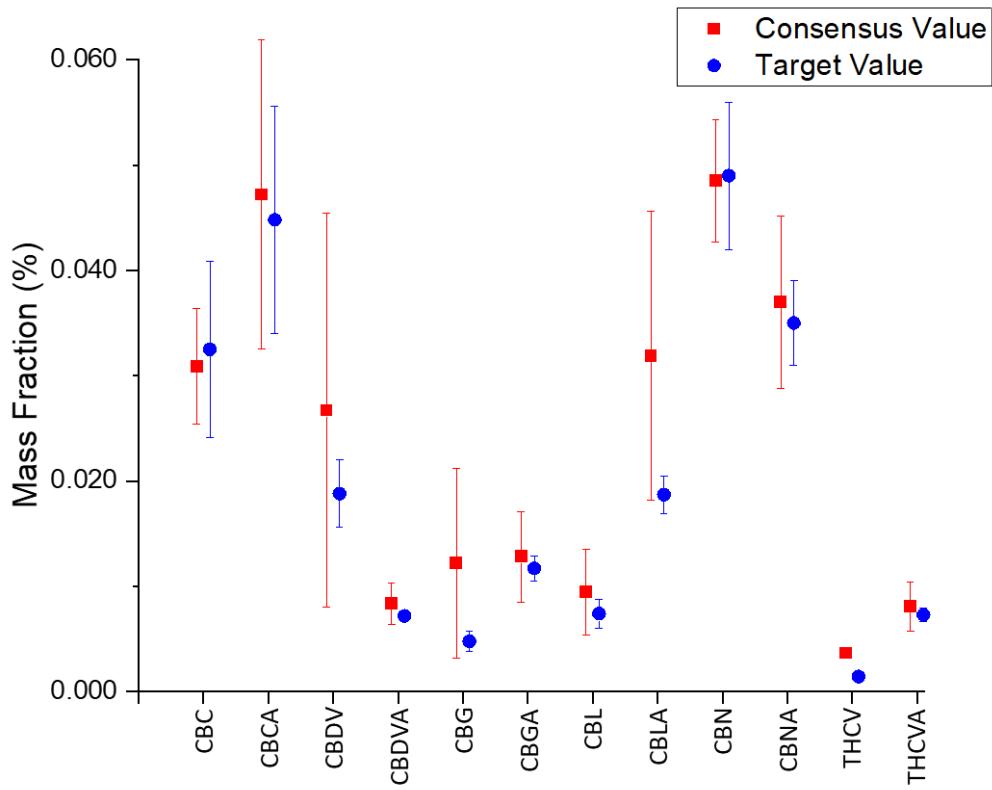
<u>Preparation Method</u>	<u>CBC</u>	<u>CBCA</u>	<u>CBDV</u>	<u>CBDVA</u>	<u>CBG</u>	<u>CBGA</u>	
Solvent Extraction	94.4	91.2	94.6	92.9	94.9	92.8	
Dilution	4.9	7.4	5.4	7.1	4.6	7.2	
Other/No Response	0.8	1.5	0.0	0.0	0.5	0.0	
<u>Preparation Method</u>	<u>CBL</u>	<u>CBLA</u>	<u>CBN</u>	<u>CBNA</u>	<u>THCV</u>	<u>THCVA</u>	<u>Δ<sup>8</sup>-THC</u>
Solvent Extraction	94.3	100	95.5	93.6	94.5	92.0	92.8
Dilution	5.7	0.0	4.3	6.4	5.5	8.0	6.9
Other/No Response	0.0	0.0	0.2	0.0	0.0	0.0	0.3
<u>Analytical Method</u>	<u>CBC</u>	<u>CBCA</u>	<u>CBDV</u>	<u>CBDVA</u>	<u>CBG</u>	<u>CBGA</u>	
LC-PDA	53.1	47.1	53.9	49.5	51.9	56.6	
LC-ABS	34.6	39.7	33.4	34.9	35.8	30.1	
LC-MS	1.3	2.0	1.5	1.9	1.0	1.2	
LC-MS/MS	7.2	6.9	8.4	9.4	7.4	6.6	
GC-FID	2.3	2.9	1.8	2.8	2.3	3.6	
GC-MS	0.0	0.0	0.0	0.0	0.0	0.0	
Other/No Response	1.5	1.5	0.9	1.4	1.5	1.8	
<u>Analytical Method</u>	<u>CBL</u>	<u>CBLA</u>	<u>CBN</u>	<u>CBNA</u>	<u>THCV</u>	<u>THCVA</u>	<u>Δ<sup>8</sup>-THC</u>
LC-PDA	47.2	50.6	49.5	40.4	52.6	49.7	50.1
LC-ABS	37.7	34.6	37.3	42.6	33.7	34.2	36.1
LC-MS	1.9	3.7	1.2	2.1	0.9	1.6	0.9
LC-MS/MS	9.4	3.7	7.7	10.6	9.1	9.6	7.7
GC-FID	3.8	7.4	2.9	4.3	1.8	3.2	3.2
GC-MS	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Other/No Response	0.0	0.0	1.4	0.0	1.8	1.6	2.0

#### 5.4. Study Results and Discussion

The participant results are summarized in the community results sections of **Table 5-1** through **Table 5-3**, which are based on the quantitative results reported by participating laboratories summarized in **Appendix B**. Target values for all minor cannabinoids except Δ<sup>8</sup>-THC were provided for NRC HEMP-1 using the certified mass fractions assigned by NRC Canada [6]. The remainder of the plant samples have assigned target values for CBC (all plant samples), CBG (Plant Sample 5), and CBN (Plant Sample 3 and Plant Sample 5), with no target values assigned in Plant Sample 2, Plant Sample 4, or Plant Sample 6 for the remaining minor cannabinoids due to limitations discussed in Section 2.2.3.

The participant results for the 13 minor cannabinoids in NRC HEMP-1 are summarized in the community results section of **Table 5-1**, **Table 5-2**, and **Table 5-3**. **Fig. 5-1** compares the target values and expanded uncertainties (error bars) for the minor cannabinoids in NRC HEMP-1 [6] to the consensus values and the robust estimate of the standard deviation of the reported values (error bars). With the exception of CBC and CBN, the consensus values for the minor cannabinoids were higher than the target values in NRC HEMP-1. The trend observed for the Δ<sup>9</sup>-THC and CBD acid/neutral pairs in which the consensus value for the acidic cannabinoid was lower than the target value and the consensus value for the neutral cannabinoid was higher than the target value

was not observed for the minor cannabinoid acid/neutral pairs. The consensus values for the majority of the minor cannabinoids compared well with the target values for NRC HEMP-1. Further discussion of specific analyte bias for minor cannabinoids in NRC HEMP-1 and other plant samples are included below.



**Fig. 5-1. Minor cannabinoid mass fraction (%) comparison between consensus and target values.**

CBDVA consensus and target mass fractions were divided by 10 for scale with the remaining cannabinoids.

#### 5.4.1. Within- and Between Laboratory Precision

**Table 5-7** details the within-laboratory ( $\%RSD_r$ ) and between-laboratory ( $\%RSD_R$ ) variabilities for minor cannabinoids in the six cannabis plant samples. The recommended within- and between-laboratory variabilities in the AOAC SMPR for quantitation of cannabinoids in hemp plant materials are based on the mass fraction of analyte in the sample [16]. The mass fraction of each minor cannabinoid in the hemp plant samples falls within the lowest mass fraction range (0.05 % to 0.5 %), indicating that the within-laboratory variability for minor cannabinoids in the hemp samples should be  $\leq 5$  % [16]. Of the laboratories that reported non-zero quantitative values for the minor cannabinoids, 49 % reported within-laboratory variabilities  $\leq 5$  %. The mass fractions of all minor cannabinoids except for CBCA in the marijuana samples was within the 0.05 % to 0.5 % range ( $\%RSD_r \leq 5$  %), with CBCA mass fractions in the 0.5 % to 5 % range ( $\%RSD_r \leq 3$  %) [16]. Of the laboratories that reported non-zero quantitative values for the minor cannabinoids, 71 % reported within-laboratory variabilities in the recommended range. The within-laboratory variabilities for the plant samples were heavily dependent on the mass fraction of the

cannabinoid in the sample, with higher variabilities reported for cannabinoids present at lower mass fractions in the plant samples (**Table 5-7**). Higher than recommended within-laboratory variability for the hemp samples is likely due to the majority of the minor cannabinoids having mass fractions near the method LOQs in the plant samples. Similar to the  $\Delta^9$ -THC in the hemp samples, as cannabinoid values approach the method LOQs, random errors accrue during sample preparation and analysis often contributing more significantly to the measurement variability.

Between-laboratory variabilities for the minor cannabinoid measurements were outside the recommended ranges for all plant samples [16]. The AOAC method recommendations are intended to evaluate reproducibility of a single method when used by multiple laboratories. Higher between-laboratory variabilities are expected when laboratories report values from multiple analytical methods and when the mass fraction of analyte in the sample is low. The between-laboratory variabilities for minor cannabinoids in Plant Sample 4 and Plant Sample 6 were higher on average than for the other plant materials, indicating the two plant samples were challenging for participants to analyze. The increase in between-laboratory variabilities for minor cannabinoids in these two plant samples compared to the other plant samples is likely twofold: the mass fractions of the minor cannabinoids in these samples are all low and both plant samples contain stems, which may be less homogeneous at smaller sample sizes than the other plant samples.

The between-laboratory variabilities were also higher than expected for a few minor cannabinoids across all plant types: CBDV, CBLA, THCV, and  $\Delta^8$ -THC. While the low mass fraction of each of these cannabinoids likely increased the between-laboratory variability, difficulties in accurately identifying CBDV, THCV, and  $\Delta^8$ -THC was also a potential contributing factor as was discussed in Section 3.4.1.4 for  $\Delta^8$ -THC and in Section 5.4.2 for CBDV and THCV. With each of these minor cannabinoids having known coeluting compounds, participants should take multiple steps, similar to those listed in Section 2.2.3, to ensure that the peak being integrated is the cannabinoid of interest. When analytes are difficult to accurately identify and thus quantitate, reduced accuracy results in high between-laboratory variability.

**Table 5-7. Within-laboratory and between-laboratory variabilities for the determination of minor cannabinoids.**

Samples	<u>CBC<sup>a</sup></u>		<u>CBCA</u>		<u>CBDV<sup>a</sup></u>		<u>CBDVA<sup>a</sup></u>	
	%RSD <sub>r</sub>	%RSD <sub>R</sub>	%RSD <sub>r</sub>	%RSD <sub>R</sub>	%RSD <sub>r</sub>	%RSD <sub>R</sub>	%RSD <sub>r</sub>	%RSD <sub>R</sub>
NRC HEMP-1	4.1	17.7	5.2 <sup>a</sup>	31.1 <sup>a</sup>	5.2	70.1	3.1	23.5
Plant Sample 4	4.9	22.1	5.2 <sup>a</sup>	10.9 <sup>a</sup>	4.0	117	4.6	21.9
Plant Sample 6	4.6	32.6	2.8 <sup>a</sup>	13.6 <sup>a</sup>	5.9	105	3.1	26.8
Plant Sample 2	4.6	18.2	2.6 <sup>b</sup>	24.8 <sup>b</sup>	5.6	42.1	3.6	16.4
Plant Sample 3	3.3	14.5	2.2 <sup>b</sup>	26.1 <sup>b</sup>	2.7	34.9	2.3	13.4
Plant Sample 5	3.4	13.6	2.0 <sup>b</sup>	23.0 <sup>b</sup>	1.8	52.4	2.6	20.8
Samples	<u>CBG<sup>a</sup></u>		<u>CBGA<sup>a</sup></u>		<u>CBL<sup>a</sup></u>		<u>CBLA<sup>a</sup></u>	
	%RSD <sub>r</sub>	%RSD <sub>R</sub>	%RSD <sub>r</sub>	%RSD <sub>R</sub>	%RSD <sub>r</sub>	%RSD <sub>R</sub>	%RSD <sub>r</sub>	%RSD <sub>R</sub>
NRC HEMP-1	5.4	74.1	6.3	33.6	6.4	43.0	5.0	42.9
Plant Sample 4	7.2	32.2	6.0	22.6	5.9	120	6.4	87.7
Plant Sample 6	6.7	46.8	5.1	26.7	40.4	128	4.3	78.0
Plant Sample 2	4.2	27.0	3.4	23.5	NDR	NDR	2.6	115
Plant Sample 3	4.4	21.4	2.7	12.1	NDR	NDR	2.1	84.5
Plant Sample 5	4.7	24.6	2.5	12.3	14.5	164	1.2	114
Samples	<u>CBN<sup>a</sup></u>		<u>CBNA<sup>a</sup></u>		<u>THCV<sup>a</sup></u>		<u>THCVA<sup>a</sup></u>	
	%RSD <sub>r</sub>	%RSD <sub>R</sub>	%RSD <sub>r</sub>	%RSD <sub>R</sub>	%RSD <sub>r</sub>	%RSD <sub>R</sub>	%RSD <sub>r</sub>	%RSD <sub>R</sub>
NRC HEMP-1	3.2	12.1	4.5	22.2	14.2	139	9.9	28.6
Plant Sample 4	16.7	179	17.5	94.1	12.7	145	13.0	117
Plant Sample 6	5.4	156	7.9	74.8	44.8	405	9.0	112
Plant Sample 2	1.0	84.0	8.0	23.1	2.7	66.4	13.3	187
Plant Sample 3	2.5	24.7	3.0	62.5	5.2	49.0	6.7	10.4
Plant Sample 5	2.1	12.1	4.9	39.5	5.7	22.9	4.2	18.1
<u>Δ<sup>8</sup>-THC<sup>a</sup></u>								
Samples	%RSD <sub>r</sub>	%RSD <sub>R</sub>						
NRC HEMP-1	5.6	97.6						
Plant Sample 4	52.7	197						
Plant Sample 6	77.6	241						
Plant Sample 2	1.1	284						
Plant Sample 3	2.0	305						
Plant Sample 5	2.4	303						

<sup>a</sup> recommended %RSD<sub>r</sub> ≤ 5 % and %RSD<sub>R</sub> ≤ 10 % [16]

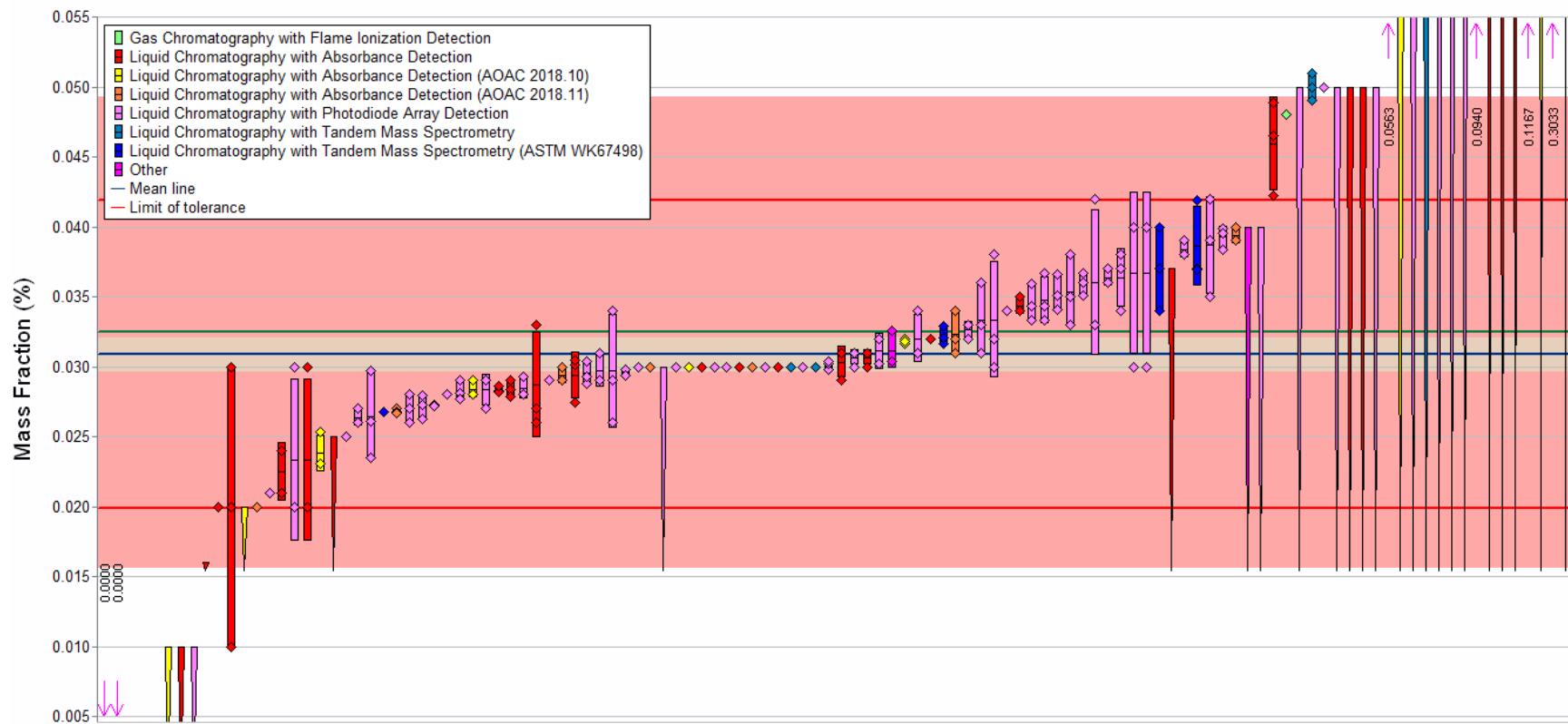
<sup>b</sup> recommended %RSD<sub>r</sub> ≤ 3 % and %RSD<sub>R</sub> ≤ 8 % [16]

NDR = No laboratories reported data for this analyte in this plant sample

## 5.4.2. Accuracy

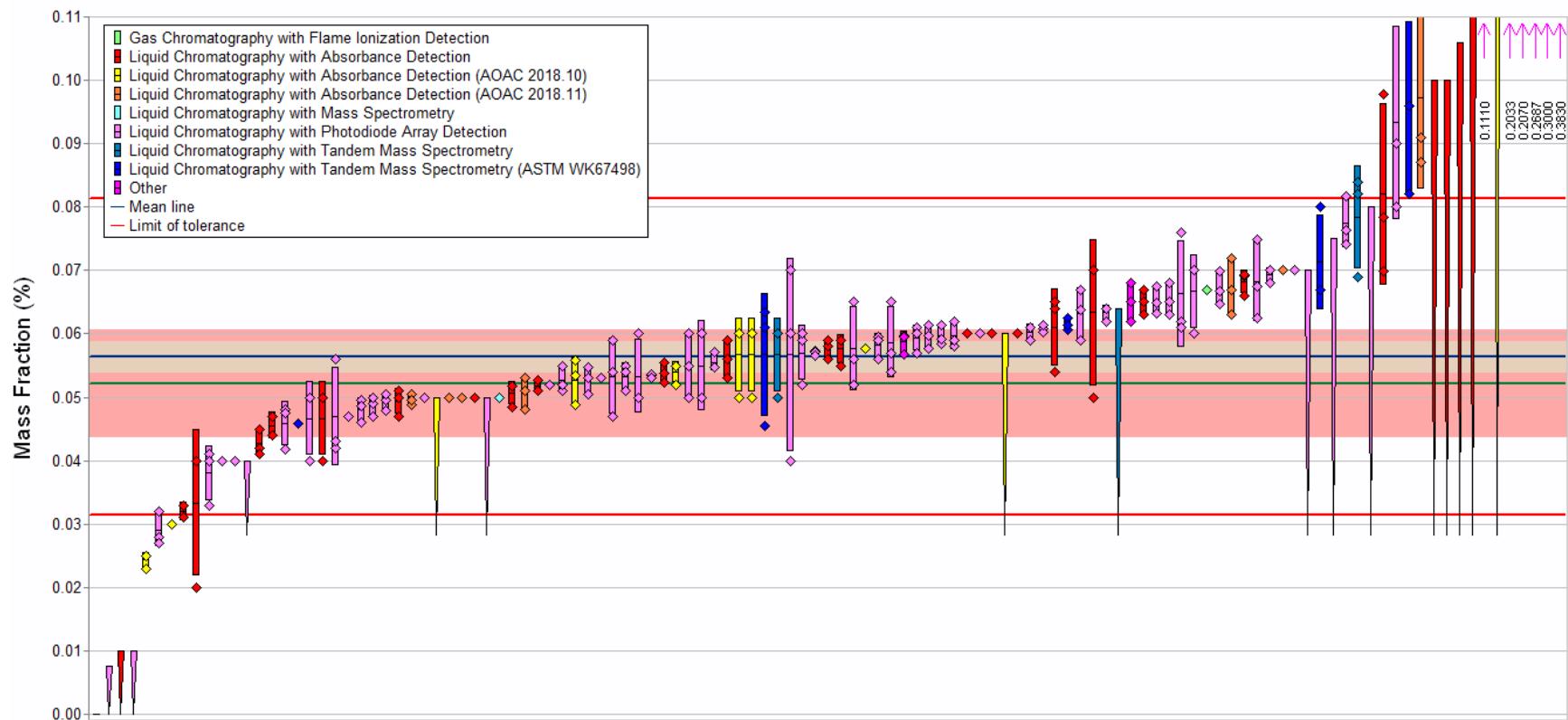
### 5.4.2.1. CBC and CBCA

Cannabichromene was the only minor cannabinoid with an assigned target value in all plant samples. The individual participant, consensus, and target mass fraction results are presented graphically in **Fig. 5-2** through **Fig. 5-7**. The consensus values for CBC were between 3 % and 21 % higher than the target values for Plant Sample 2 through Plant Sample 6, and 5 % lower for NRC HEMP-1. The consensus ranges for CBC in all but one of the study samples were completely within the target ranges. The consensus range for CBC in Plant Sample 3 extended slightly above the target range (**Fig. 5-6**). The only sample with an assigned CBCA mass fraction was NRC HEMP-1. Similar to CBC, the consensus range for CBCA was completely within the target range in NRC HEMP-1 (**Fig. 5-8**) and the consensus value was within 5 % of the target value. Because the consensus values for both the neutral and acid forms were higher than the target values in NRC HEMP-1, decarboxylation of CBCA due to improper storage is not indicated. These types of bias are usually a result of co-eluting interferences or calibration issues as discussed in Section **3.4.1.4**.



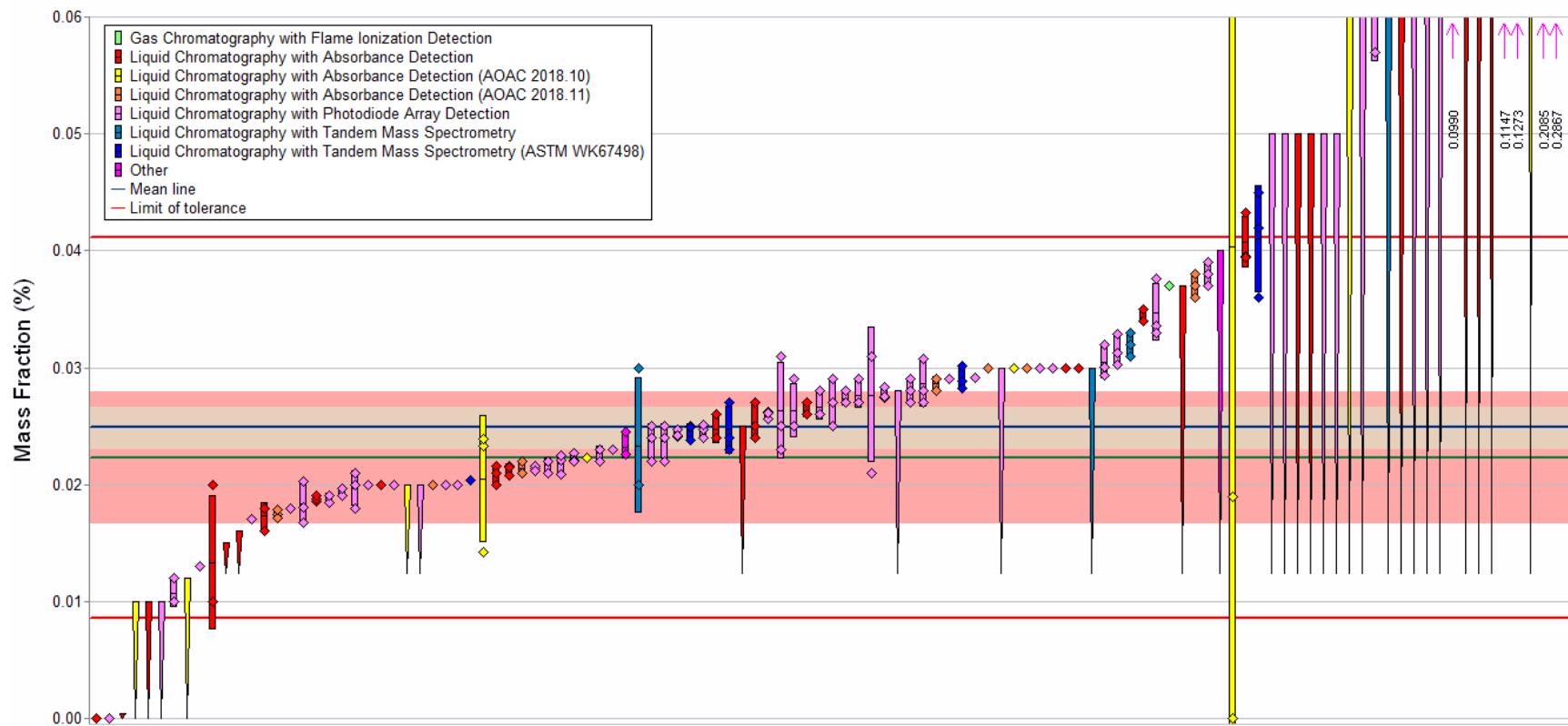
**Fig. 5-2. CBC in NRC HEMP-1 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



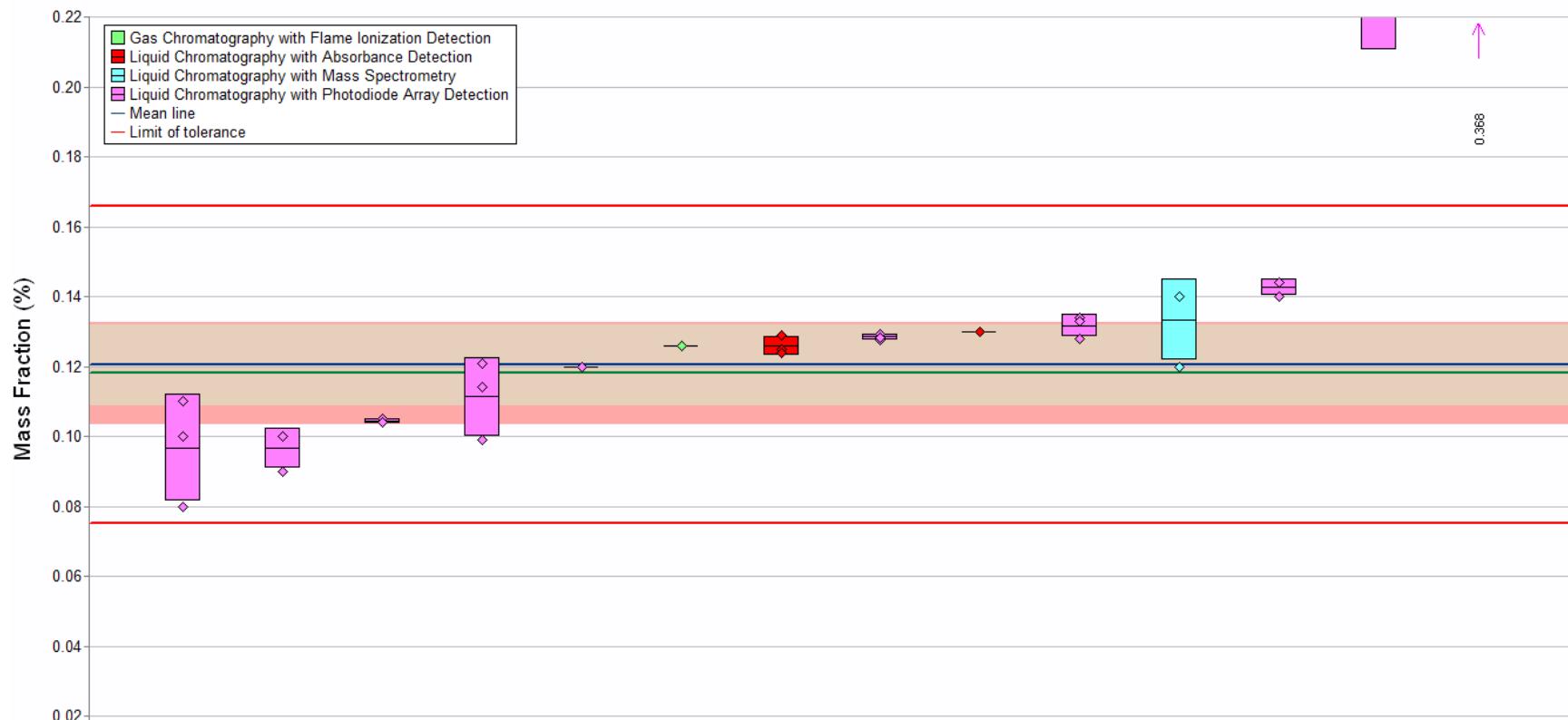
**Fig. 5-3. CBC in Plant Sample 4 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 5-4. CBC in Plant Sample 6 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 5-5. CBC in Plant Sample 2 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

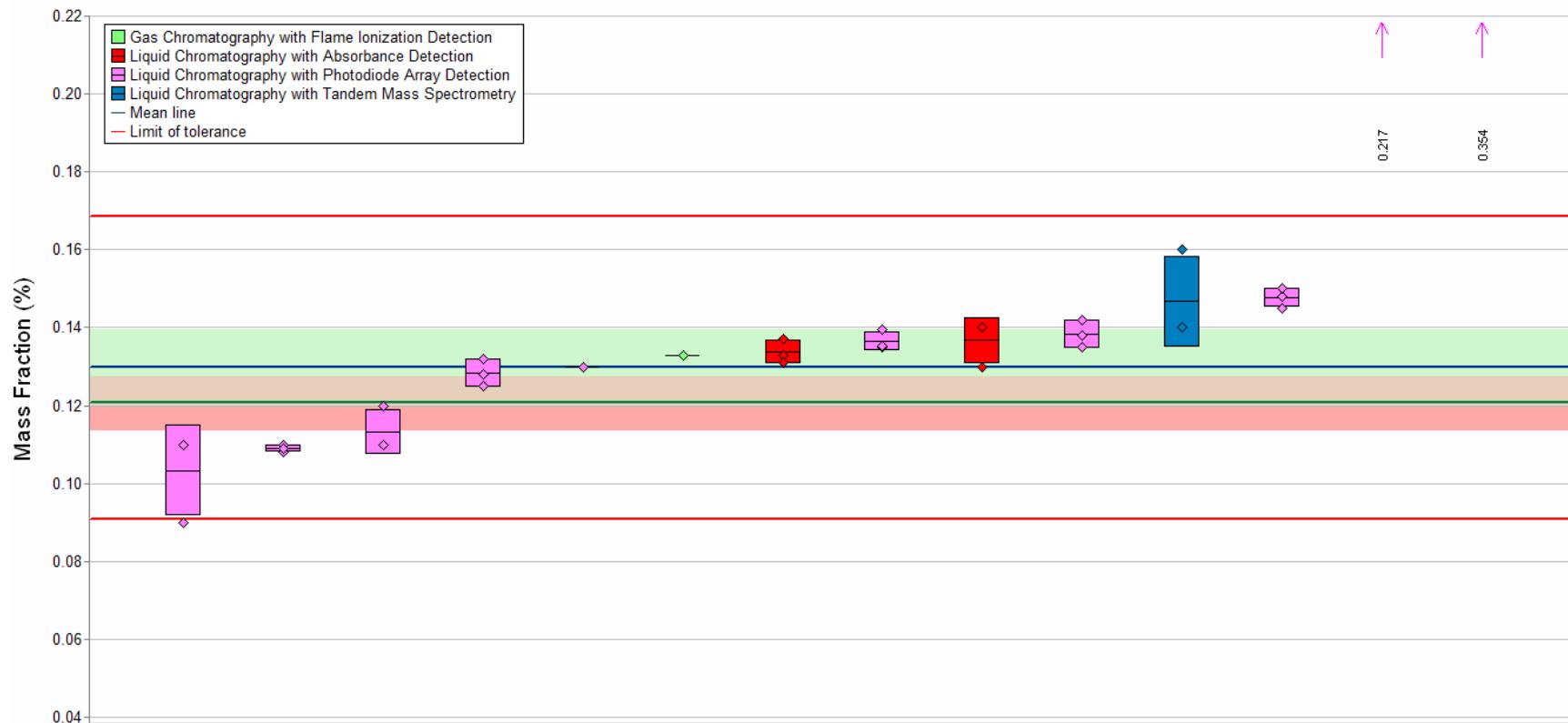
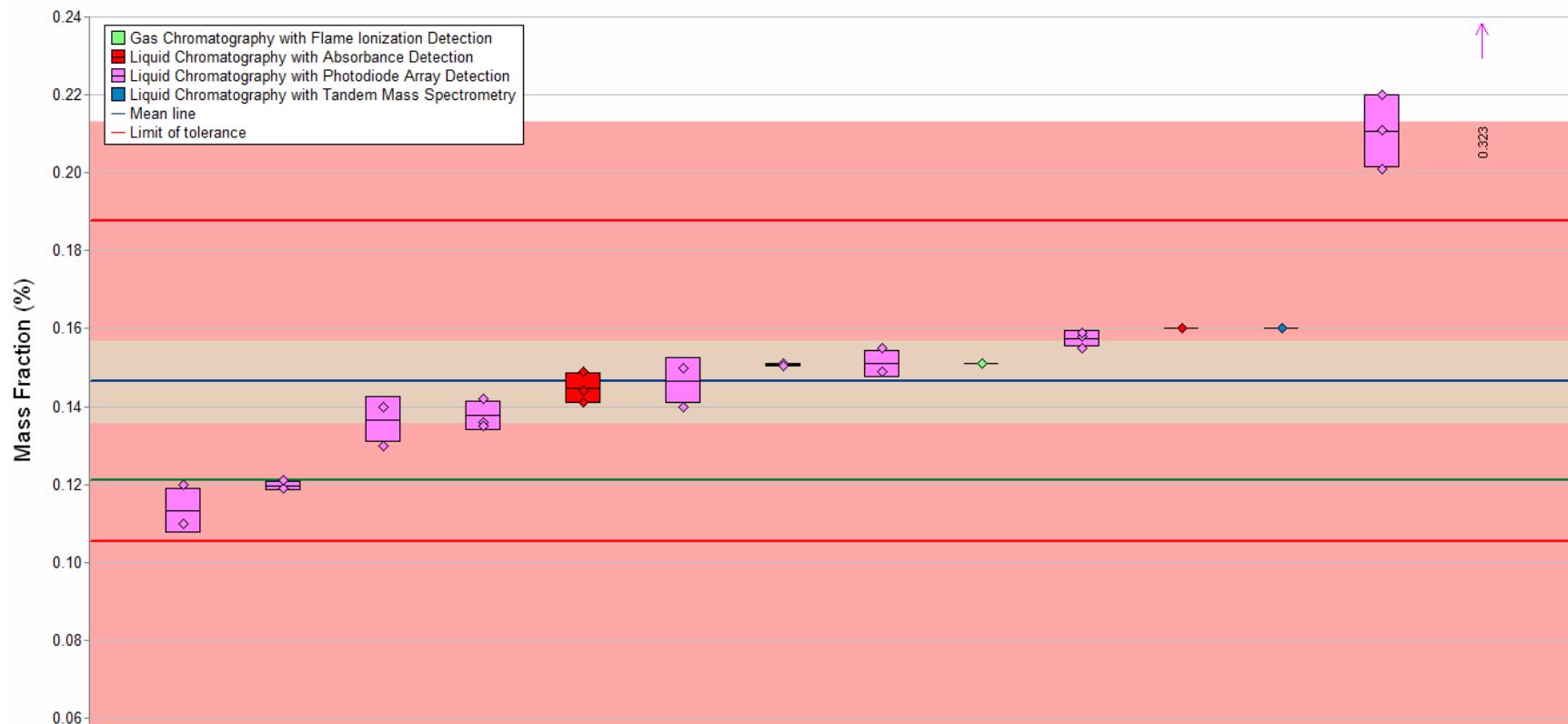


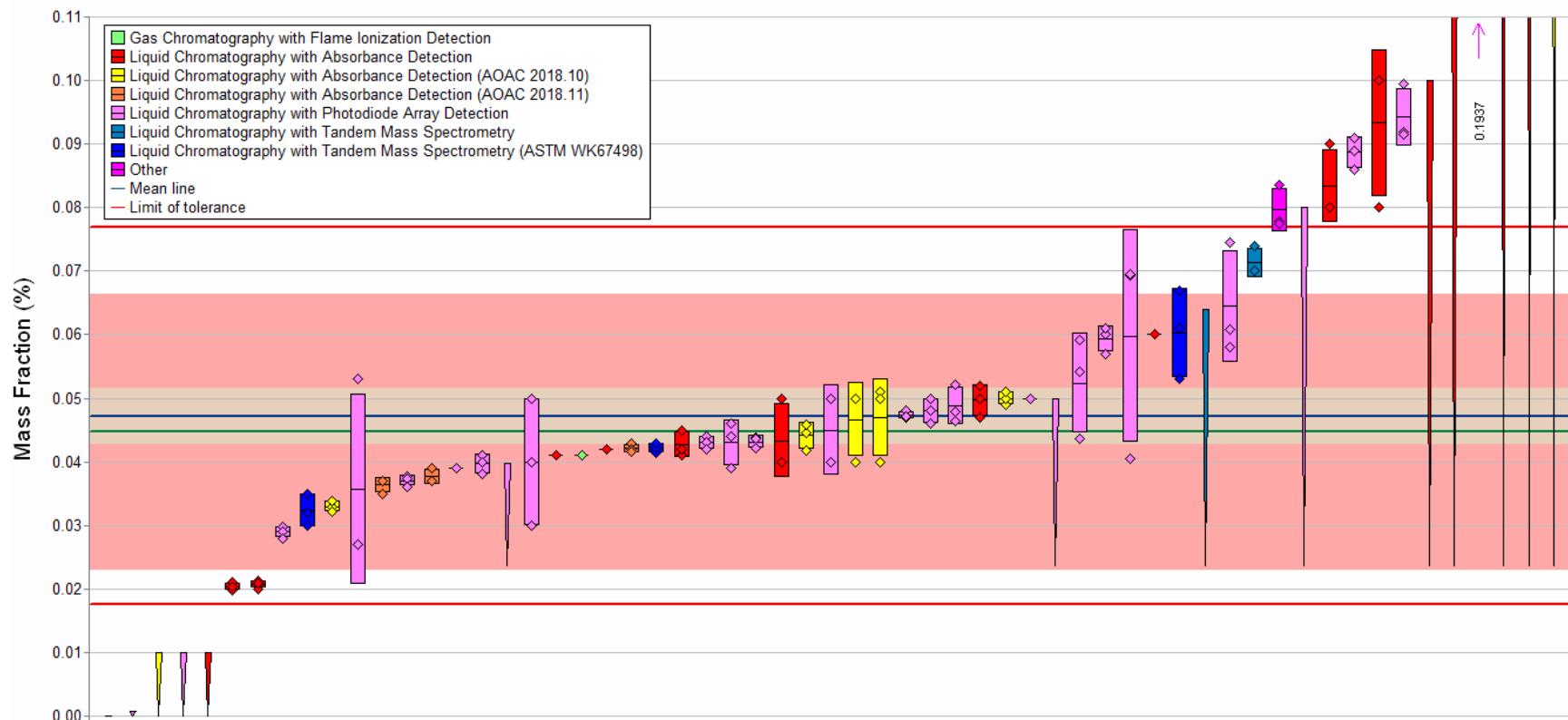
Fig. 5-6. CBC in Plant Sample 3 (data summary view – analytical method).

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 5-7. CBC in Plant Sample 5 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

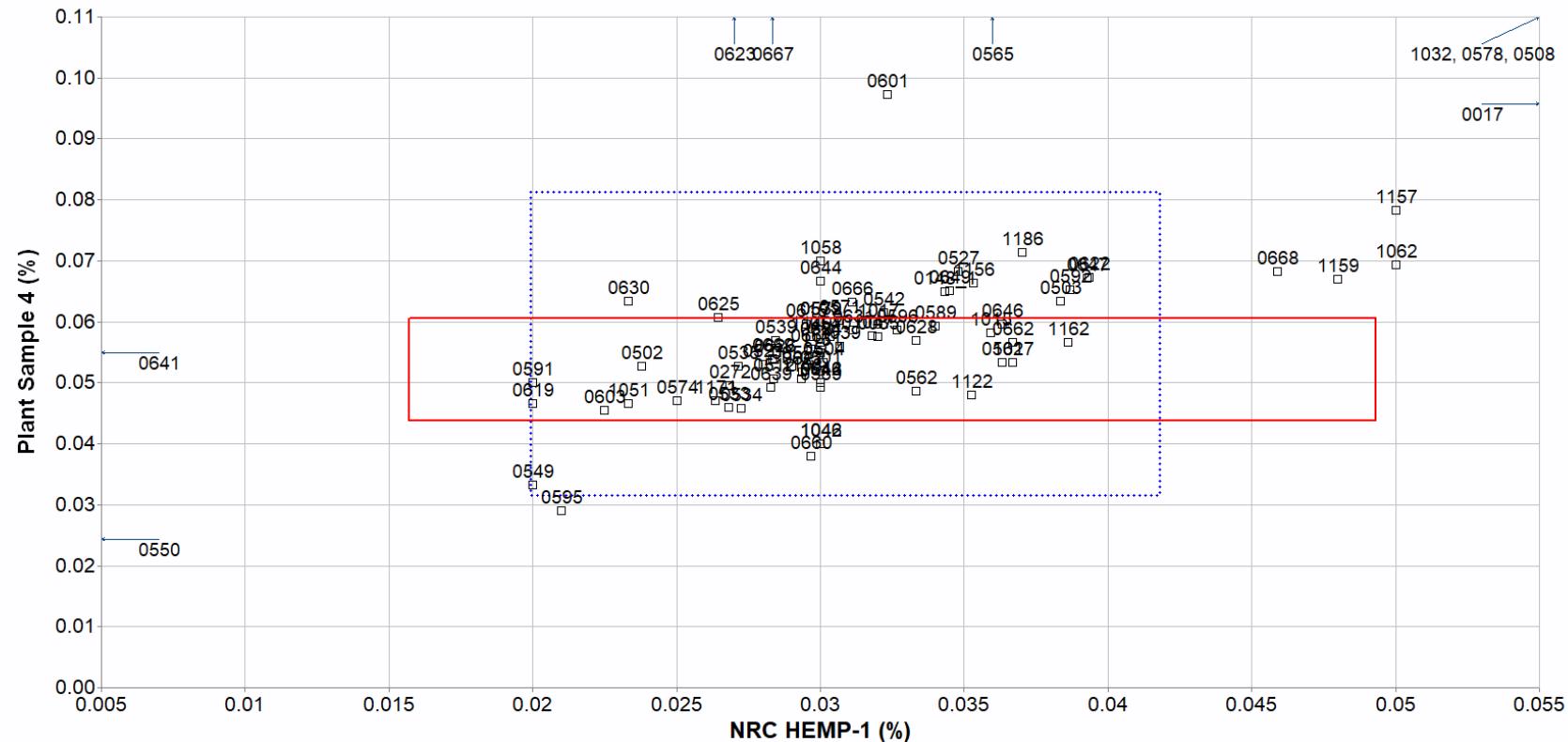


**Fig. 5-8. CBCA in NRC HEMP-1 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

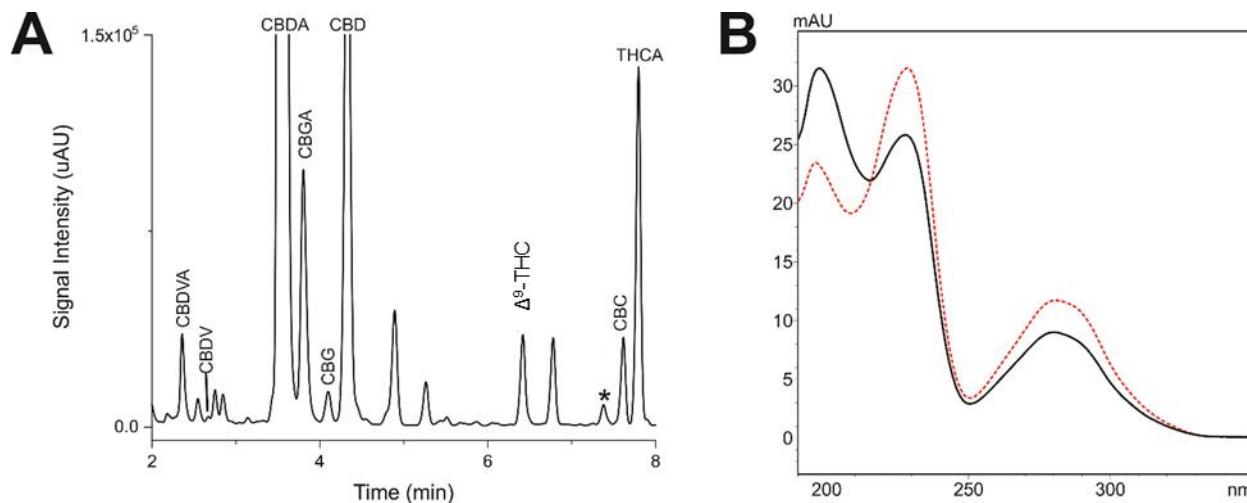
A higher percentage of laboratories reported values outside the target range for CBC in Plant Sample 2 (43 %), Plant Sample 3 (100 %), Plant Sample 4 (42 %), and Plant Sample 6 (46 %) than NRC HEMP-1 (9 %) and Plant Sample 5 (7 %). When comparing participant data for CBC in NRC HEMP-1 and Plant Sample 4 (**Fig. 5-9**), the CBC values were clustered centrally, with a higher number of participants overestimating the CBC values in Plant Sample 4 than NRC HEMP-1. This clustering is indicative of random errors being more impactful than systematic errors. The increased high bias for CBC measurements in the NIST plant samples compared to CBC measurements in NRC HEMP-1 could be due to co-elution of interfering cannabinoids, namely CBL and THCA (**Fig. 5-10** and **Fig. 5-11** in addition to calibration bias, as discussed in Section **3.4.1.4**). Approximately 87 % of participants reported use of either LC-ABS or LC-PDA as their analytical method for CBC measurements (**Table 5-6**). The monitoring wavelength for CBC in most cases is between 220 nm and 230 nm, which is not a selective range. The selectivity in LC-ABS and LC-PDA methods comes from the chromatographic separation of the cannabinoids, which should be thoroughly investigated in existing methods.

LC-PDA measurements for CBC completed at NIST were not included in the target value assignment because of the existence of a chromatographic interference. An example LC-UV chromatogram at 220 nm for Plant Sample 4 is shown in **Fig. 5-10 A**. CBC was tentatively identified in the chromatogram based on the retention time of the peak. However, when comparing the tentative CBC peak spectrum to the CBC spectrum of the calibration standard, a co-eluting species was observed (**Fig. 5-10 B**). Similar interferences were observed for CBC in Plant Sample 6, Plant Sample 2, Plant Sample 3, and Plant Sample 5 (data not shown). The co-eluting peak could not be identified through comparisons of retention times and absorbance spectra collected from other commercially available cannabinoid reference standards. Additionally, when using ACN and H<sub>2</sub>O as the mobile phase, as was reported by the majority of participants that responded to the method survey **Appendix C**, CBC and THCA elute closely. The impact of the percentage of ACN:H<sub>2</sub>O, and column temperature on CBC and THCA peak separation is illustrated in **Fig. 5-11**. When chromatographic methods do not baseline resolve CBC and THCA, analysis of samples with high THCA mass fractions may result in erroneous estimations of CBC and/or THCA mass fractions. Laboratories using LC methods should be sure to thoroughly evaluate all potential chromatographic cannabinoid interferences as commercial standards become available after they complete method development and validation. Coelution issues are resolvable by improving the baseline separation between analytes. Baseline separation is increasingly important for laboratories using UV absorbance detection because the absorbance spectra for cannabinoids are similar and absorbance detectors lack the specificity of mass spectrometers [20].



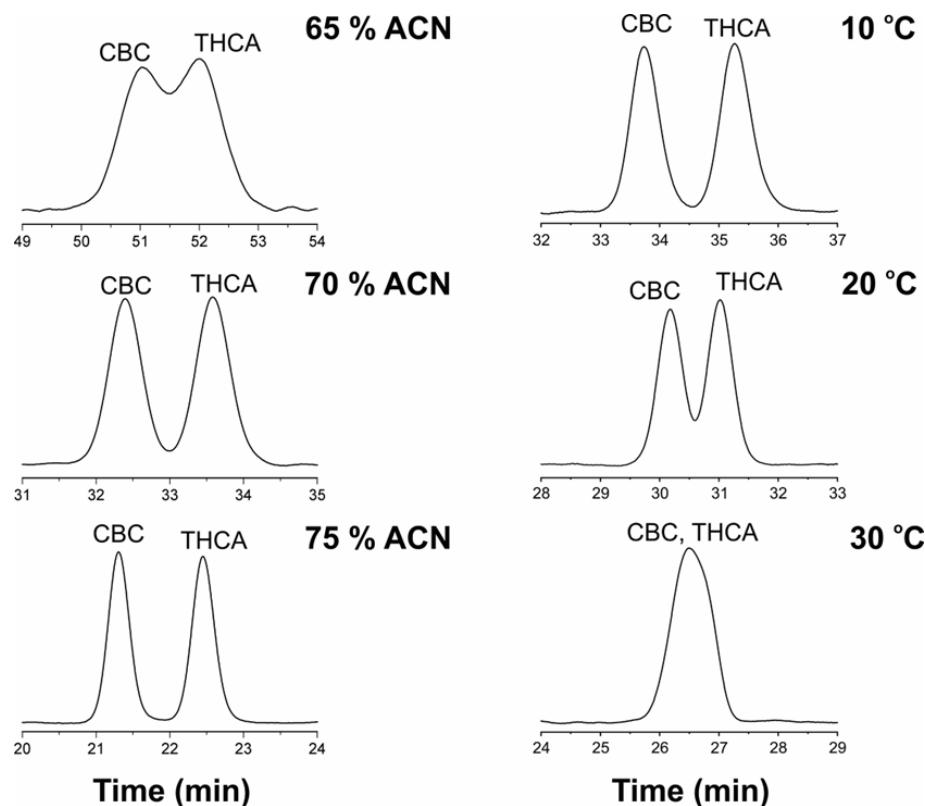
**Fig. 5-9. Laboratory means for CBC in NRC HEMP-1 and Plant Sample 4 (sample/sample comparison view).**

In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (Plant Sample 4). The solid red box represents the target range of tolerance for the two samples, NRC HEMP-1 (x-axis) and Plant Sample 4 (y-axis), which encompasses the target values bounded by their uncertainties ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The dotted blue box represents the consensus range of tolerance for NRC HEMP-1 (x-axis) and Plant Sample 4 (y-axis), calculated as the values above and below the consensus means that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ .



**Fig. 5-10. Chromatogram and absorbance spectra for tentative CBC peak.**

Panel A displays a LC-UV chromatogram at 220 nm for Plant Sample 4. Panel B displays the absorbance spectra for the tentatively identified CBC peak (solid black curve) and a CBC reference standard (dotted red curve). The chromatographic peak labeled “\*” was tentatively identified as CBL based on retention times of reference standards.



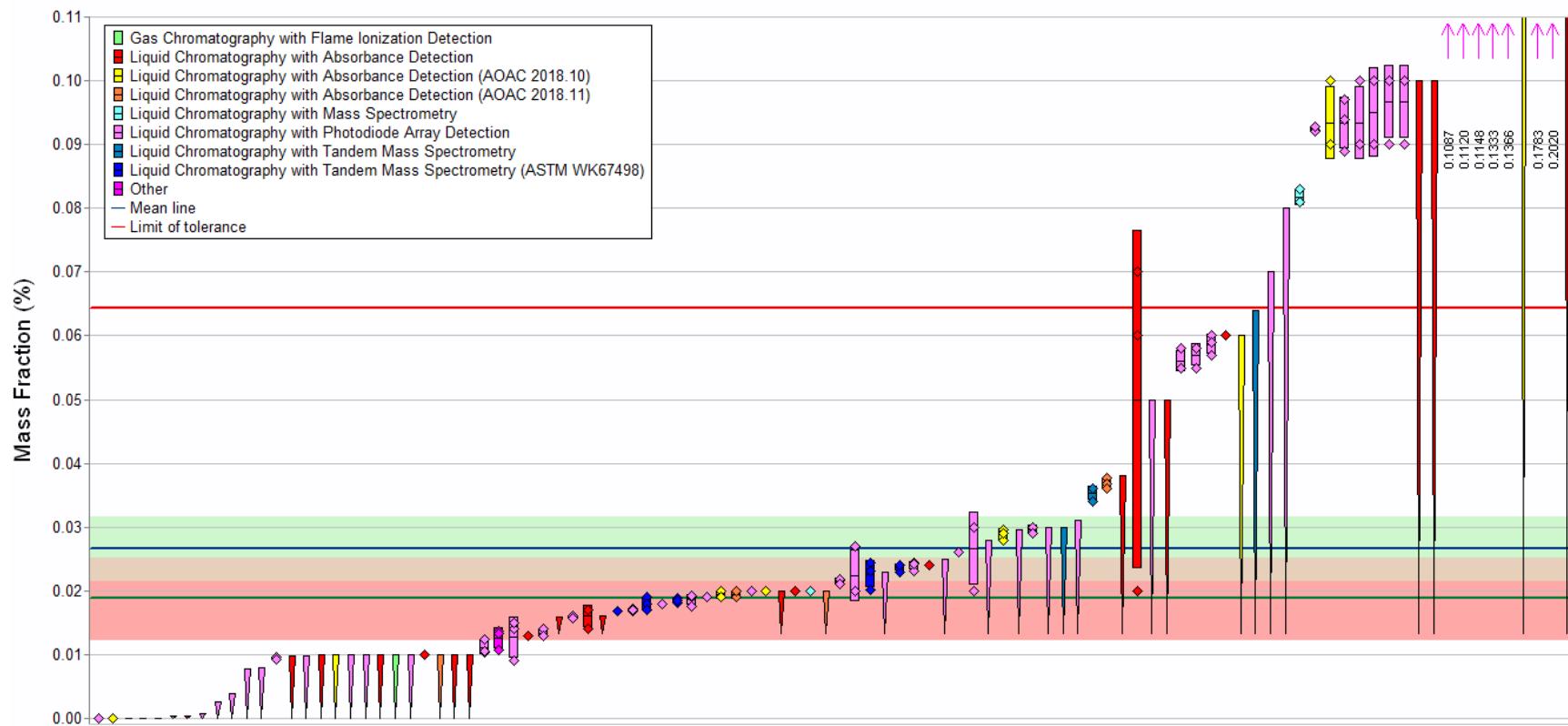
**Fig. 5-11. LC-UV chromatograms at 220 nm for CBC and THCA reference standards under different conditions.**

The panels to the left display chromatographic resolution at three ACN:H<sub>2</sub>O isocratic mobile phase compositions using a column temperature of 23 °C. The panels to the right display resolution at three column temperatures using a 75 % ACN:H<sub>2</sub>O mobile phase.

#### 5.4.2.2. CBDV and CBDVA

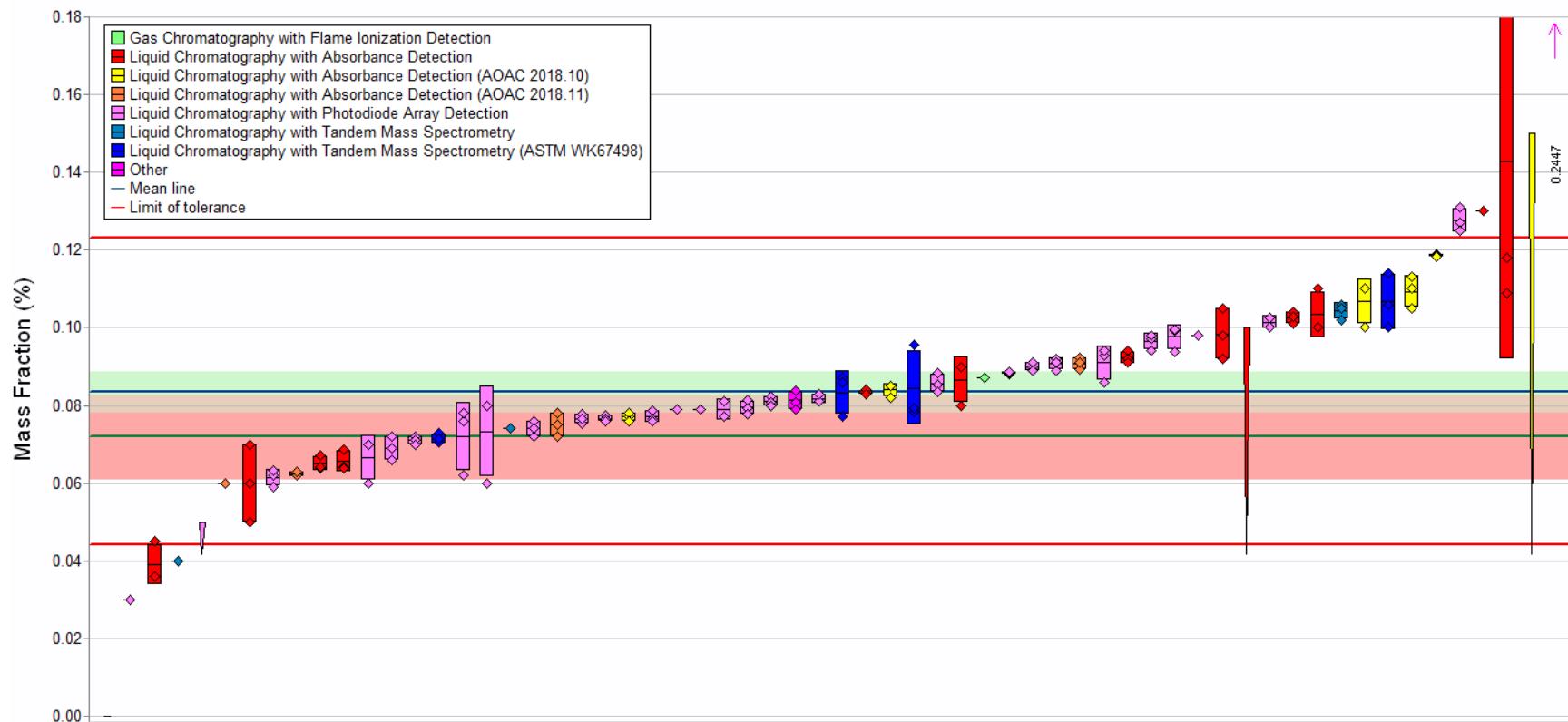
CBDV and CBDVA were only assigned target values in NRC HEMP-1. The individual participant, consensus, and target mass fraction results for CBDV and CBDVA in NRC HEMP-1 are presented graphically in **Fig. 5-12** (CBDV) and **Fig. 5-13** (CBDVA). The consensus values were higher than the target value for both CBDV (42 %) and CBDVA (16 %) in NRC HEMP-1. The target range did not contain the consensus value for either CBDV or CBDVA, with both consensus ranges extending above the target ranges. Approximately 55 % and 60 % of laboratories reporting quantitative results for CBDV and CBDVA in NRC HEMP-1, respectively, reported results outside the target range. Of laboratories reporting results outside of the target range, 84 % (CBDV) and 83 % (CBDVA) of laboratories reported results that were higher than the target range. Because the consensus values for both neutral and acid forms were higher than the target values in NRC HEMP-1, decarboxylation of CBDVA due to improper storage was not indicated. With a majority of laboratories reporting results higher than the target range, a combination of co-eluting interferences or calibration issues is likely, for one or both minor cannabinoids, similar to what was discussed in Section **3.4.1.4**.

The majority of participants reported use of either LC-ABS or LC-PDA as their analytical method for CBDV (87.3 %) and CBDVA (84.4 %) measurements (**Table 5-6**). The monitoring wavelength for CBDV and CBDVA in most cases is between 220 nm and 230 nm, which is not a selective range. The selectivity in LC-ABS and LC-PDA methods comes from the chromatographic separation of the cannabinoids, which should be thoroughly investigated in existing methods. CBDV in NRC HEMP-1 elutes closely with several peaks that have a maximum absorbance at 220 nm (**Fig. 3-13 A**), which could easily be misidentified as CBDV. Similarly, significant chromatographic interferences were observed at NIST for CBDV in Plant Sample 4 (**Fig. 5-14 A**), but not for CBDVA as confirmed in the absorbance spectra depicted in **Fig. 5-14 B**. Misidentification in the plant samples is more likely for CBDV because CBDV is present at or near the method LOQs and elutes closely with multiple interfering compounds. Because there were no likely coelutions observed for CBDVA, laboratories with measurements higher than the target range likely had calibration issues similar to those discussed in Section **3.4.1.4** for  $\Delta^9$ -THC.



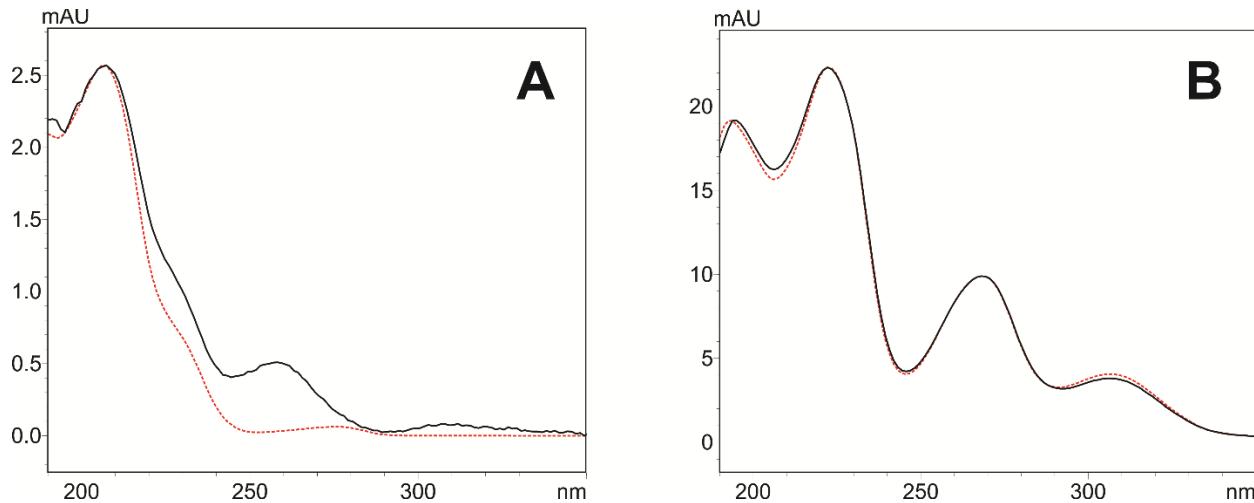
**Fig. 5-12. CBDV in NRC HEMP-1 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 5-13. CBDVA in NRC HEMP-1 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 5-14. Absorbance spectra for chromatographic peaks identified as CBDV and CBDVA in Plant Sample 4.**

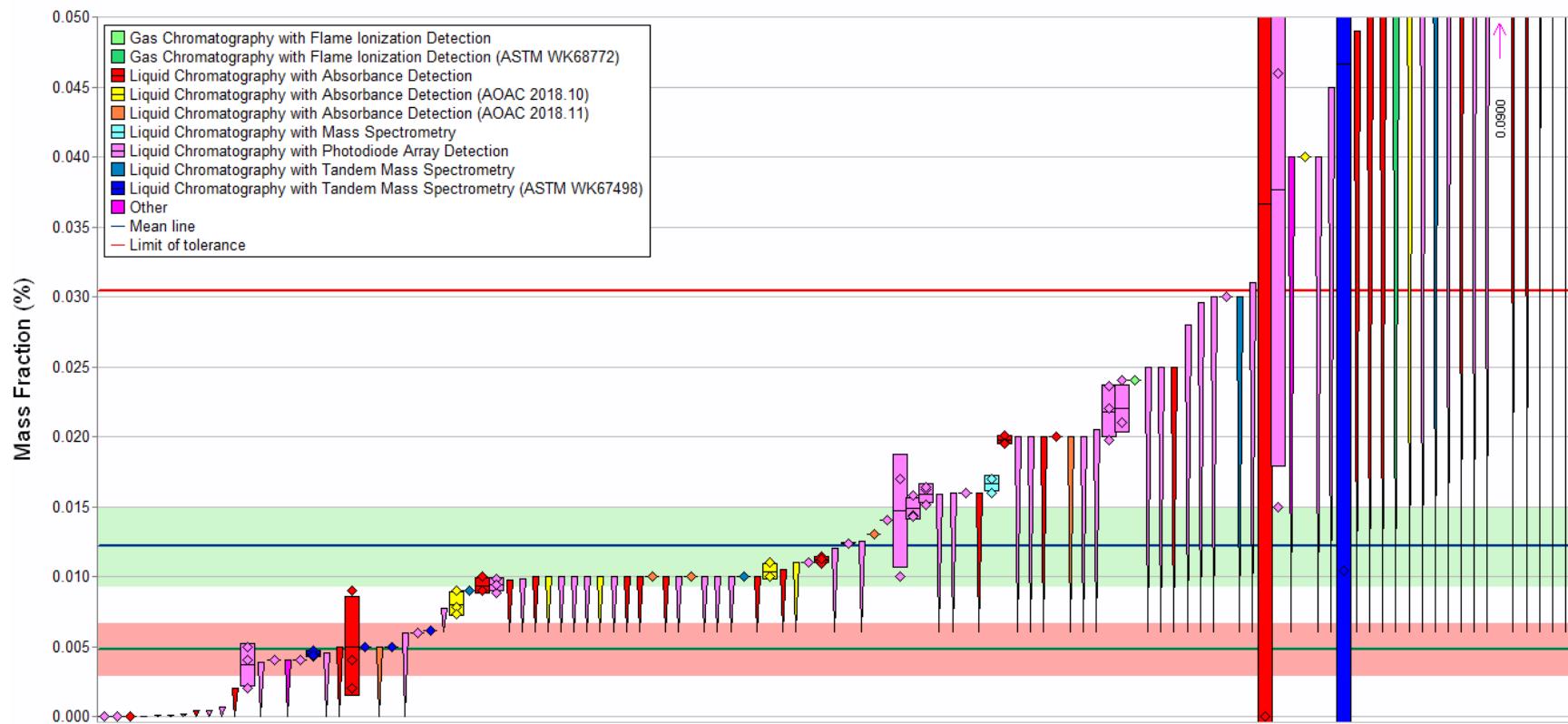
Panel A displays the absorbance spectra collected for the chromatographic peak identified as CBDV. Panel B displays the spectra for the peak identified as CBDVA. The solid black lines represent the spectra collected from the Plant Sample 4 chromatograms. The dotted red curves represent spectra of reference standards.

#### 5.4.2.3. CBG and CBGA

Target values for CBG were assigned in NRC HEMP-1 and Plant Sample 5. The individual participant, consensus, and target mass fraction results for CBG are presented graphically in **Fig. 5-15** and **Fig. 5-16**. The consensus values for CBG were 154 % higher than the target value for NRC HEMP-1 and 5 % lower than the target value for Plant Sample 5. The consensus range for NRC HEMP-1 was completely above the target range and the consensus range for Plant Sample 5 encompassed the target range. The only plant sample with an assigned target value for CBGA was NRC HEMP-1 (**Fig. 5-17**). The consensus value for CBGA was 9 % higher than the target value. While the consensus range extended higher than the target range, the consensus value was within the target range, indicating the consensus and target values agreed.

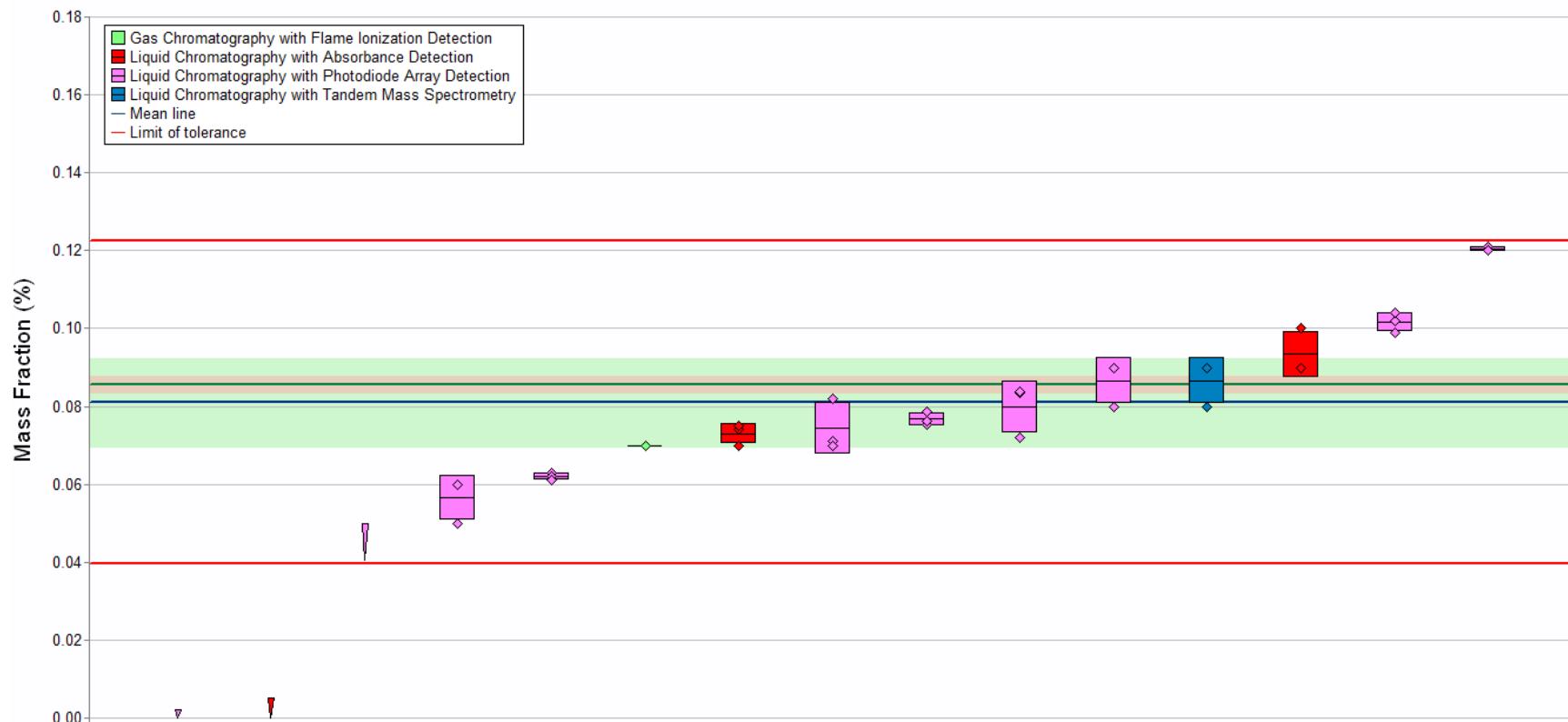
The CBG mass fraction in NRC HEMP-1 was an order of magnitude lower than in Plant Sample 5 and the recommended LOQ for cannabinoids in hemp [16]. The low mass fraction of CBG and CBGA in the samples increases the influence of coeluting compounds and calibration curve bias. Approximately 87 % of participants reported use of either LC-ABS or LC-PDA as their analytical method for CBG and CBGA measurements (**Table 5-6**). Similar to the other cannabinoids, the monitoring wavelength for CBG and CBGA in most cases is between 220 nm and 230 nm, which is not a selective range. The selectivity in LC-ABS and LC-PDA methods comes from the chromatographic separation of the cannabinoids, which should be thoroughly investigated in existing methods, especially when the analytes, such as CBG, are known to be present in low levels in the samples. The example LC-UV chromatogram shown in **Fig. 3-13 A** and **Fig. 5-10 A** for NRC HEMP-1 and Plant Sample 4 does not indicate chromatographic interference for either CBG or CBGA; however, absorbance comparisons in **Fig. 5-18** shows the presence of an interference for CBG and not CBGA in Plant Sample 4. Accuracy decreases when the amount of analyte in the sample nears the LOQ. Method LOQs must be determined appropriately and lower mass fraction analytes must be investigated using calibration curve ranges that do not extend orders of

magnitude beyond the unknown analyte concentration. If the CBG and/or CBGA concentrations in unknown samples appear to fall at or below the lowest calibration point, additional calibrants should be made and a new curve produced to quantitate the low mass fraction sample more accurately (Section 3.4.3.4).



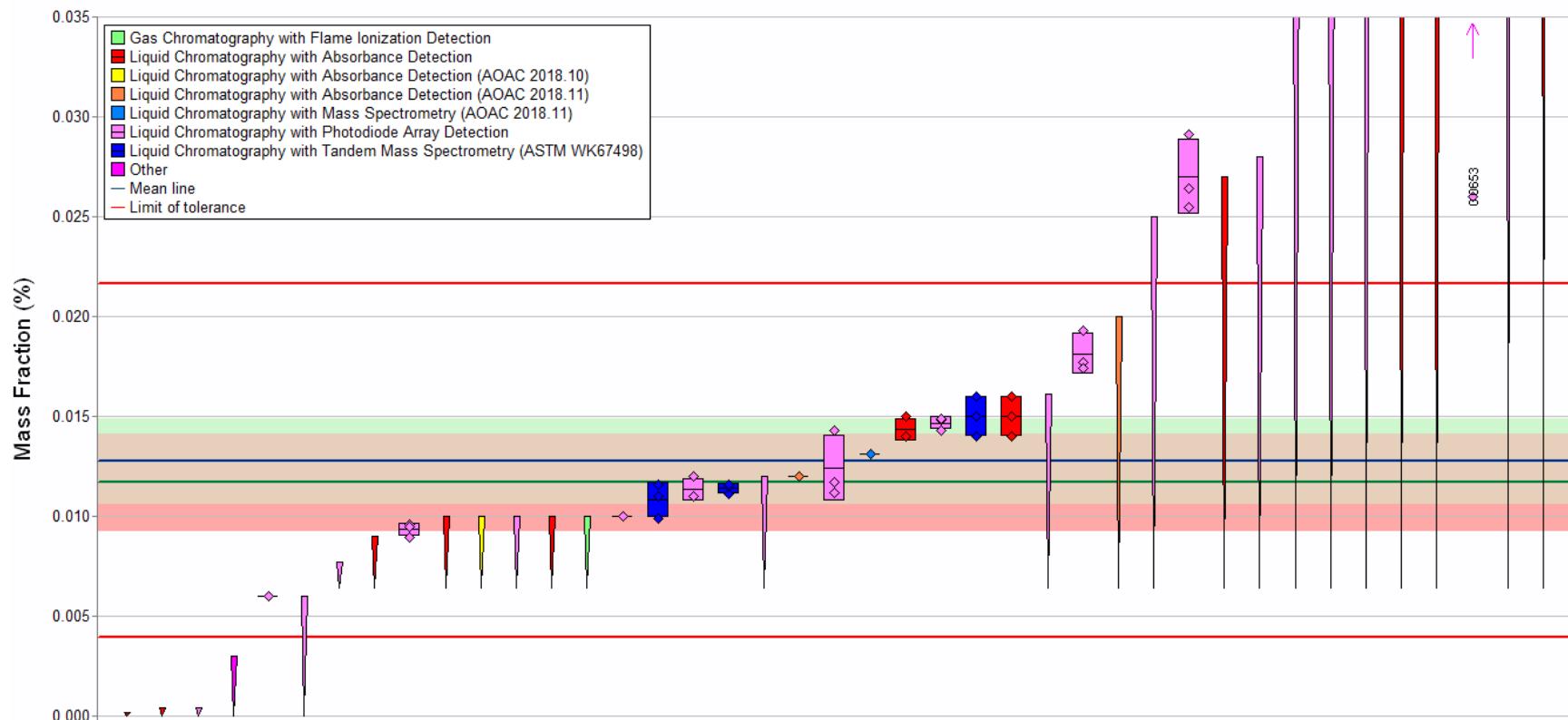
**Fig. 5-15. CBG in NRC HEMP-1 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



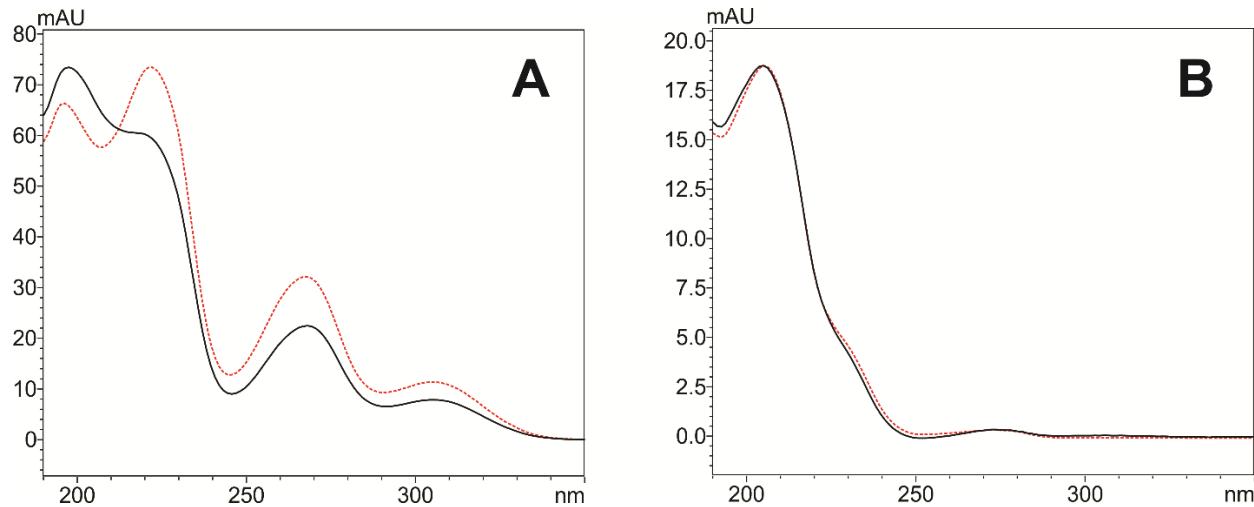
**Fig. 5-16. CBG in Plant Sample 5 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 5-17. CBGA in NRC HEMP-1 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



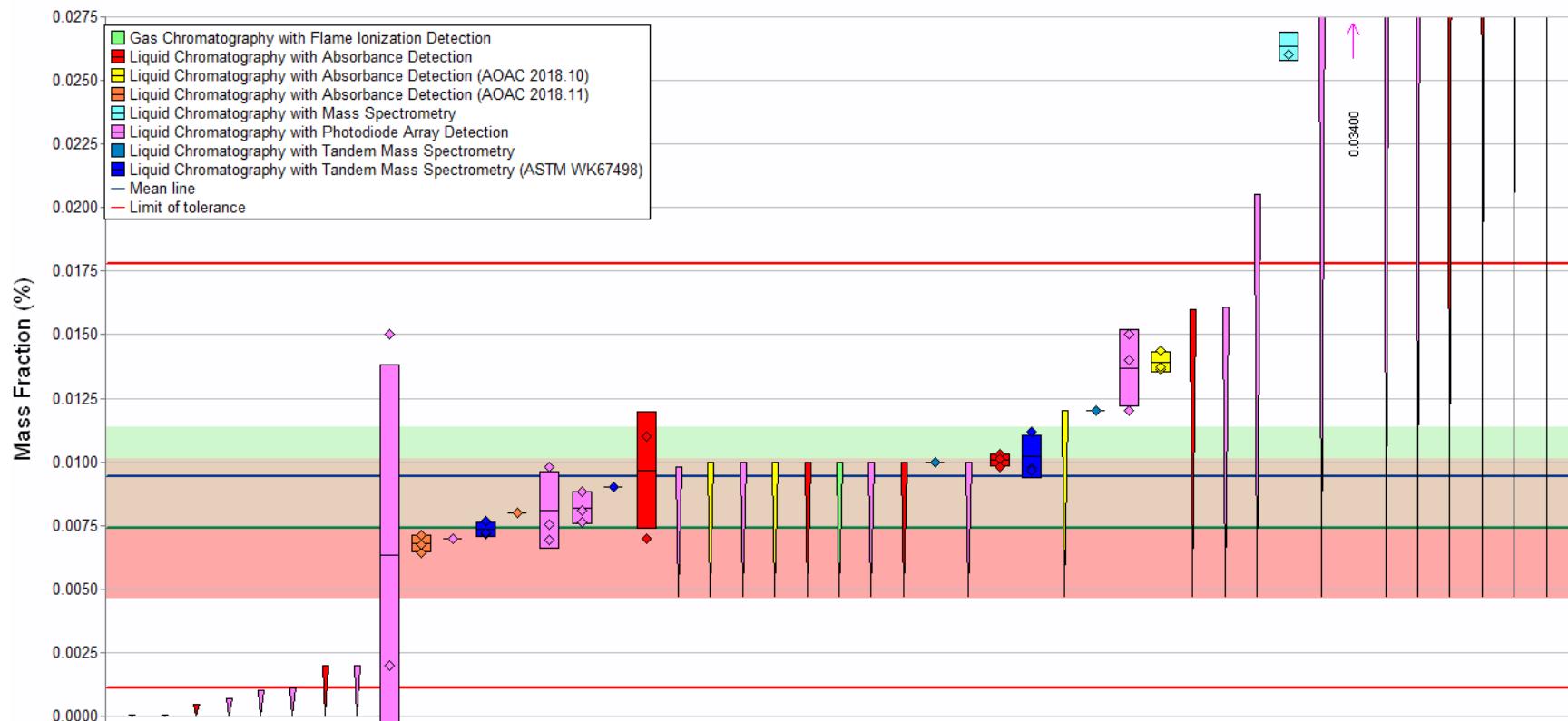
**Fig. 5-18. Absorbance spectra for chromatographic peaks identified as CBG and CBGA in Plant Sample 4.**

Panel A displays the absorbance spectra collected for the chromatographic peak identified as CBG. Panel B displays the spectra for the peak identified as CBGA. The solid black lines represent the spectra collected from the Plant Sample 4 chromatograms. The dotted red curves represent spectra of reference standards.

#### 5.4.2.4. CBL and CBLA

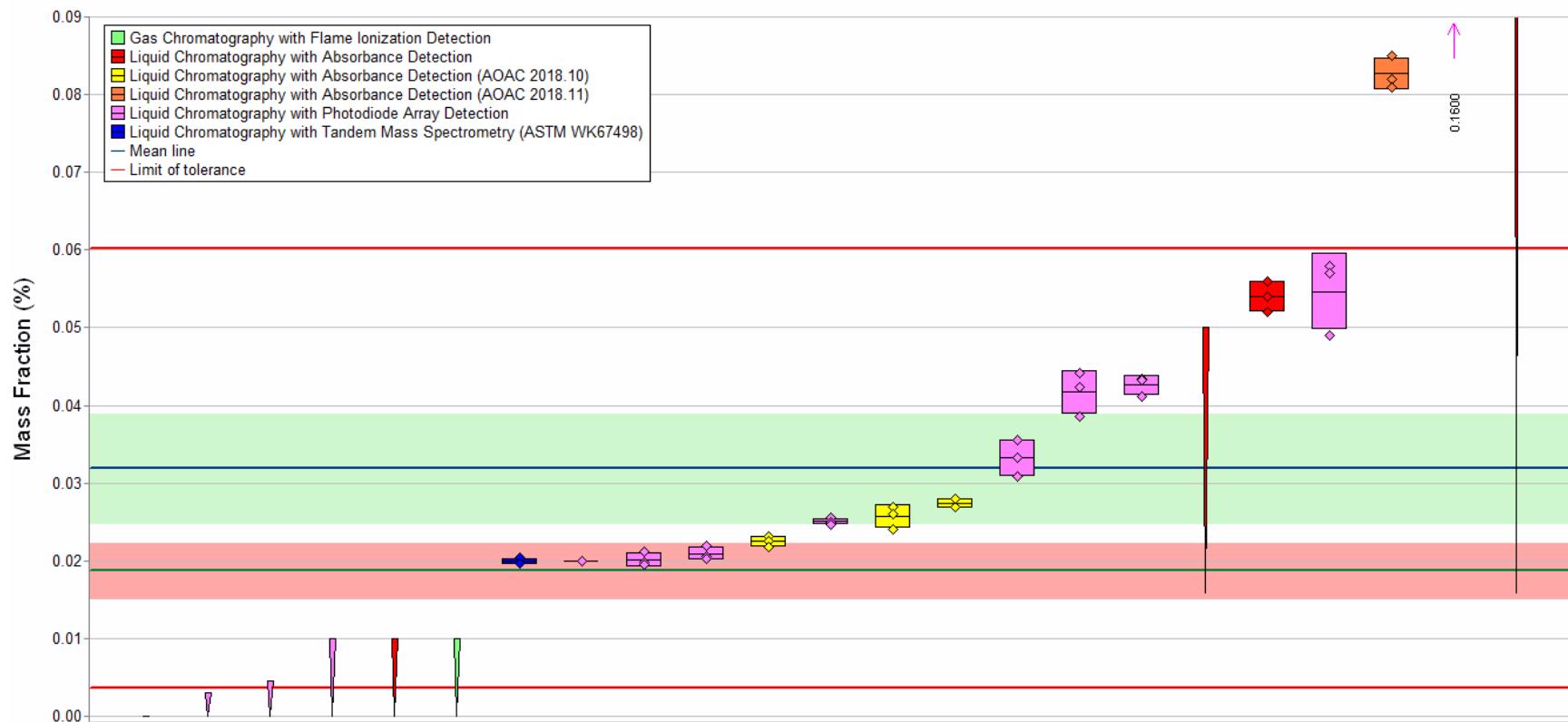
Assigned target values for CBL and CBLA were only available for NRC HEMP-1. The individual participant, consensus, and target mass fraction results for CBL and CBLA in NRC HEMP-1 are presented graphically in **Fig. 5-19** (CBL) and **Fig. 5-20** (CBLA). The consensus values were higher than the target value for both CBL (27 %) and CBLA (71 %) in NRC HEMP-1. The consensus value for CBL was within the target range, with the consensus range for CBL extending higher than the target range. Of the laboratories reporting values for CBL, 71 % reported values within the target range. The laboratories reporting values outside the target range for CBL all reported values above the target range. Of the laboratories reporting quantitative values for CBLA, 73 % reported values outside of and higher than the target range.

Similar to the other minor cannabinoids, the majority of laboratories (85 %) that reported values for CBL and CBLA reported use of either LC-ABS or LC-PDA. The higher consensus and participant values for CBL and CBLA are likely due to a combination of coeluting compounds and calibration bias, similar to what was discussed in Section 3.4.1.4. An example of a potential chromatographic interference for CBL is shown in the chromatogram of **Fig. 5-10 A**. CBL was tentatively identified based on retention time; however, the absorbance spectra shown in **Fig. 5-21** of the tentative CBL peak in Plant Sample 4 clearly shows the chromatographic peak is not CBL. Similar to CBL, CBLA elutes closely with CBCA, and could be mistaken for CBCA if the two cannabinoids are not baseline resolved. CBLA also elutes at the end of most methods, which may result in a broad, flat peak, making chromatographic separation more difficult. Analytical methods must be modified to improve baseline separation of all cannabinoids of interest from other cannabinoids and should be routinely reevaluated for potential chromatographic cannabinoid interferences as commercial standards become available, even after methods are validated and implemented.



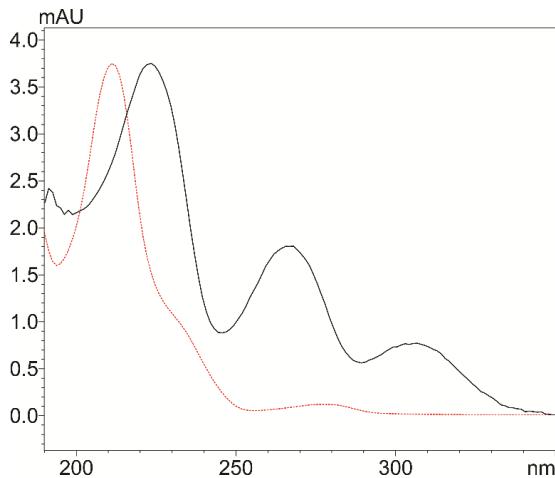
**Fig. 5-19. CBL in NRC HEMP-1 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 5-20. CBLA in NRC HEMP-1 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



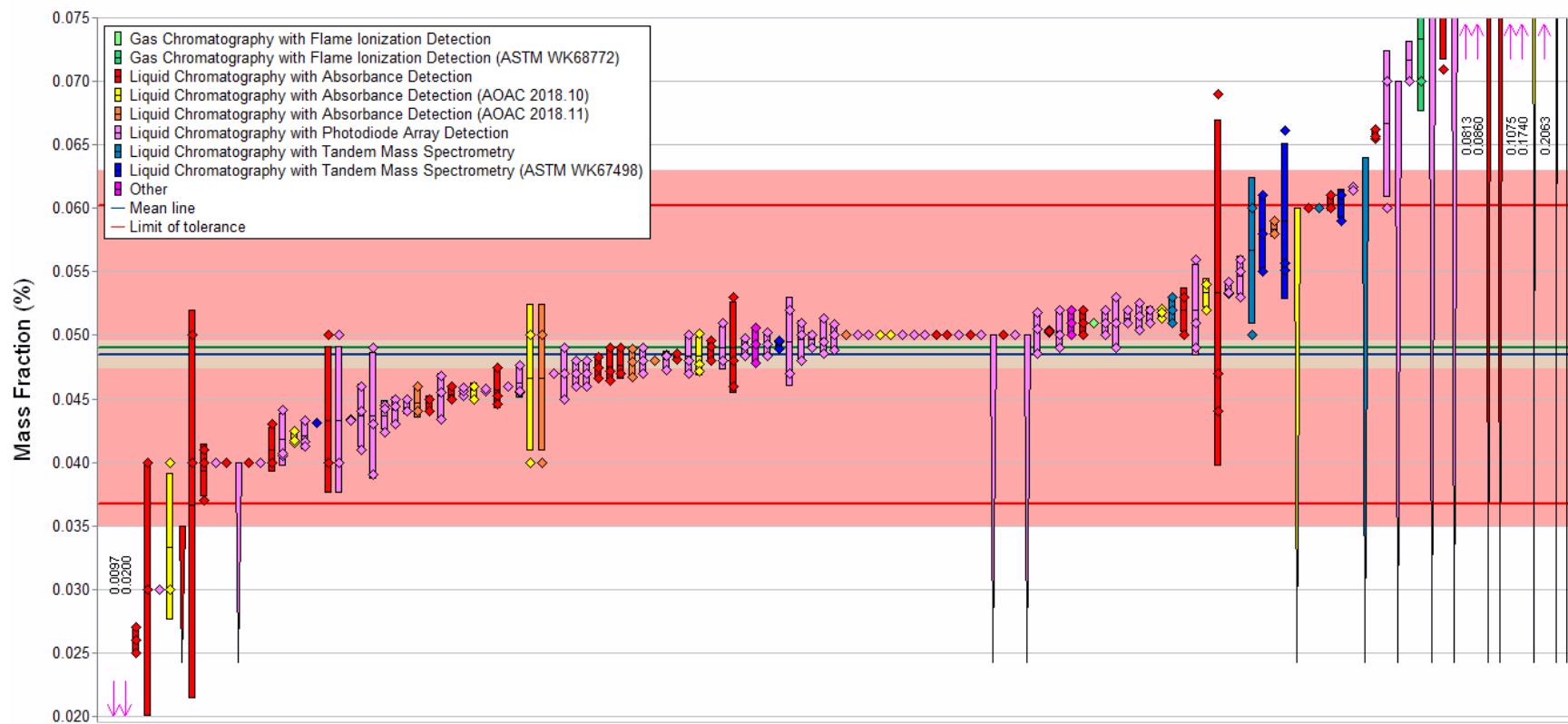
**Fig. 5-21. Absorbance spectra for chromatographic peak identified as CBL in Plant Sample 4.**

The absorbance spectra collected for the chromatographic peak identified as CBL is displayed as a solid black curve. The dotted red curve represents the spectrum of a CBL reference standard.

#### 5.4.2.5. CBN and CBNA

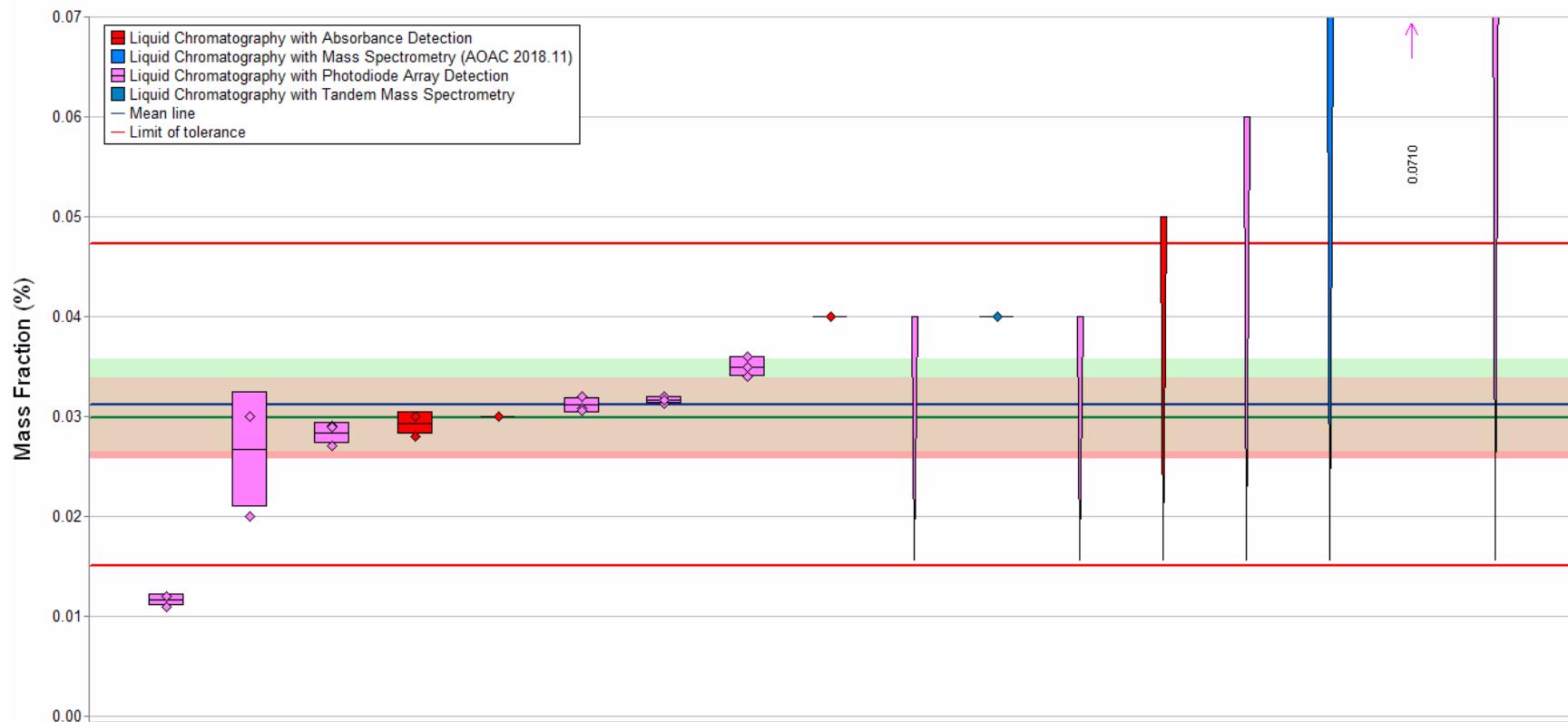
A target value was assigned for CBN in NRC HEMP-1, Plant Sample 3, and Plant Sample 5. The individual participant, consensus, and target mass fraction results are presented graphically in **Fig. 5-22** through **Fig. 5-24**. The consensus value for CBN in NRC HEMP-1 was 1 % lower than the target value and 4 % and 5 % higher than the target values in Plant Sample 3 and Plant Sample 5, respectively. The consensus values for CBN all three plant samples were within the target ranges, with the consensus ranges for Plant Sample 3 and Plant Sample 5 extending above the target range. The only sample with an assigned CBNA mass fraction was NRC HEMP-1. Similar to CBN in NRC HEMP-1, the consensus value and range for CBNA were completely within the target range (**Fig. 5-25**) and the consensus value was within 6 % of the target value.

In general, the consensus data agreed with the target values and ranges for all three plant samples. A larger fraction of participants reported values outside the target range for CBN in Plant Sample 3 (45 %) and Plant Sample 5 (67 %) than in NRC HEMP-1 (14 %), which is likely a result of the uncertainties associated with the target values for Plant Sample 3 and Plant Sample 5 being smaller than for that of NRC HEMP-1. For laboratories that reported values outside of and higher than the target range for Plant Sample 3 and Plant Sample 5, a calibration bias is unlikely considering the CBN mass fractions in NRC HEMP-1, Plant Sample 3, and Plant Sample 5 were similar. Coeluting compounds may have interfered with CBN quantitation in some participant methods. However, no evidence of interferences were observed in the NIST LC-PDA method for quantitation of CBN in Plant Sample 3 and Plant Sample 5, as was illustrated in **Fig. 5-26**.



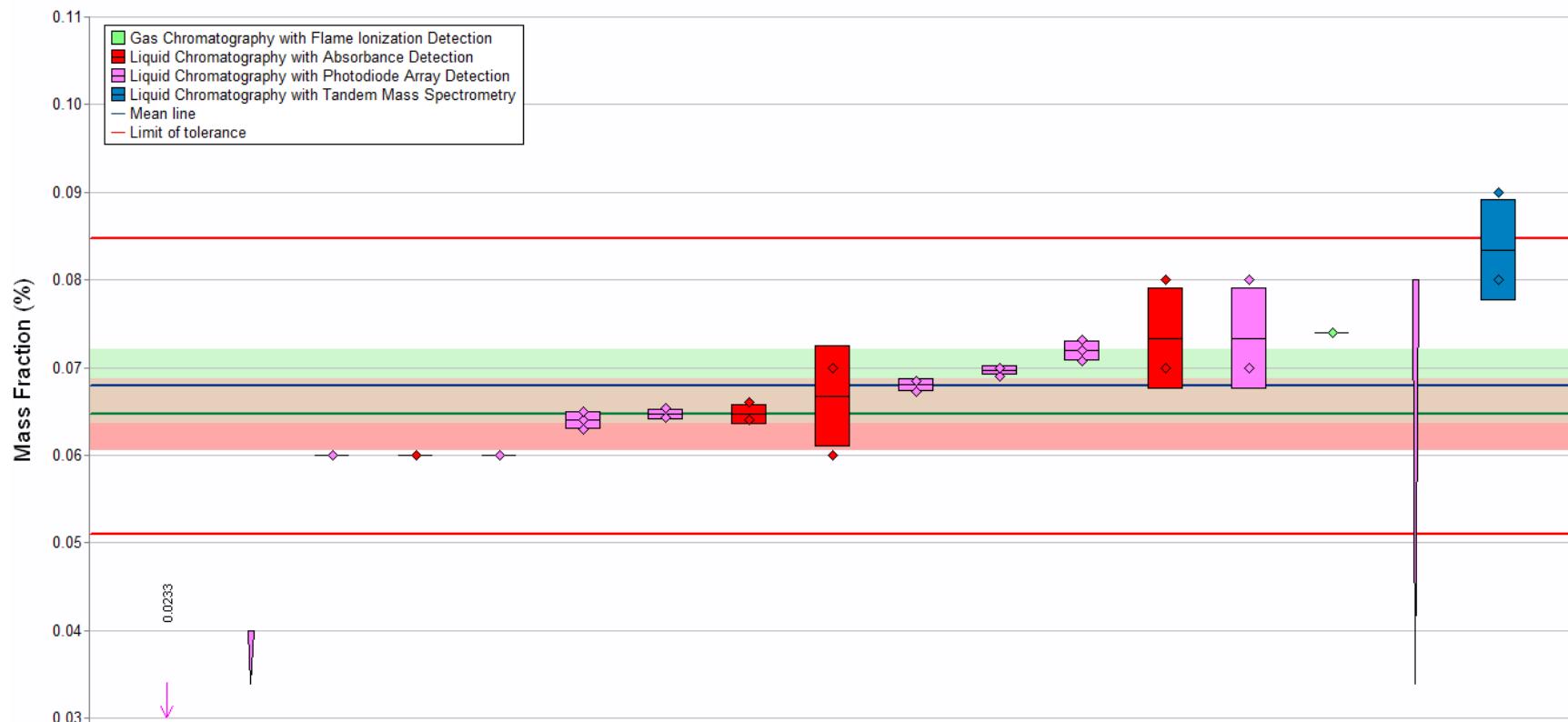
**Fig. 5-22. CBN in NRC HEMP-1 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



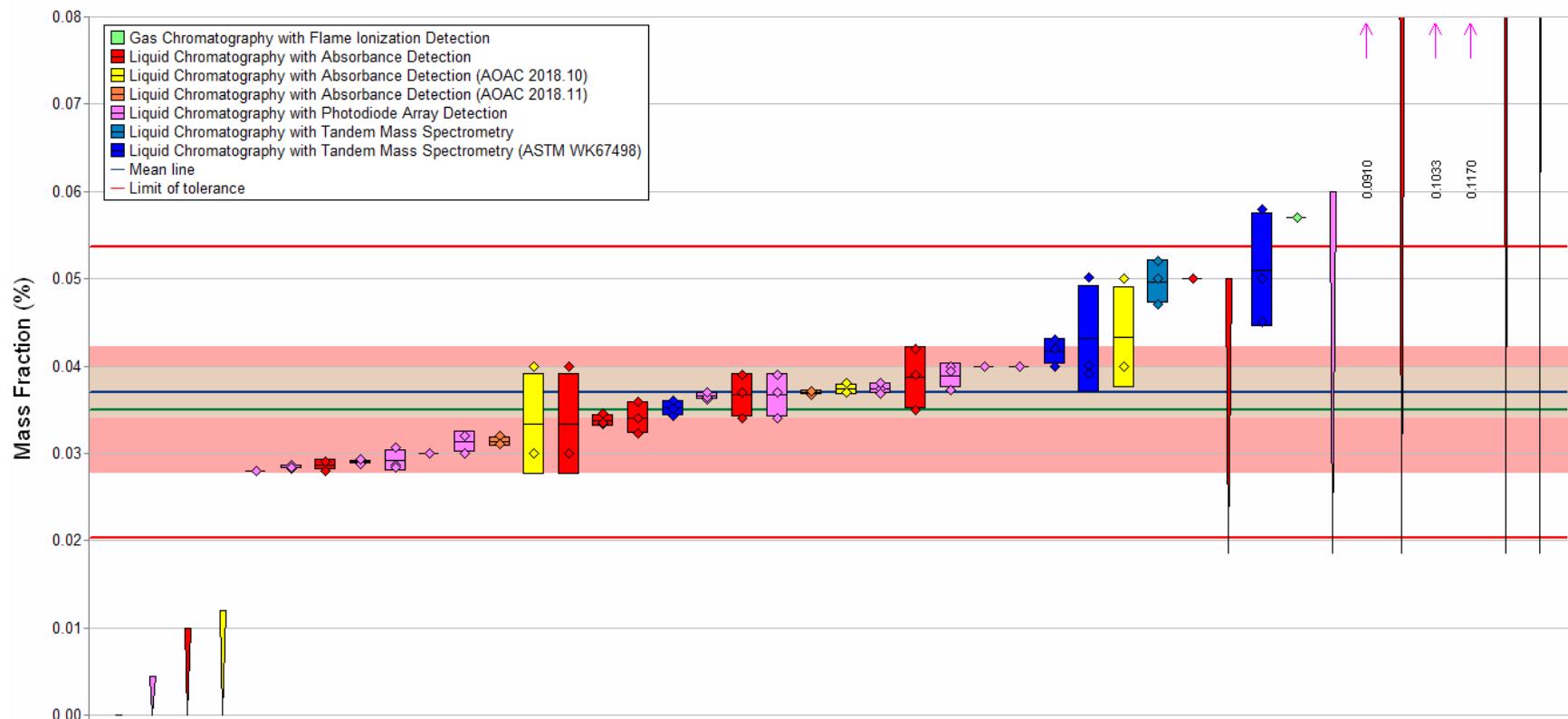
**Fig. 5-23. CBN in Plant Sample 3 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



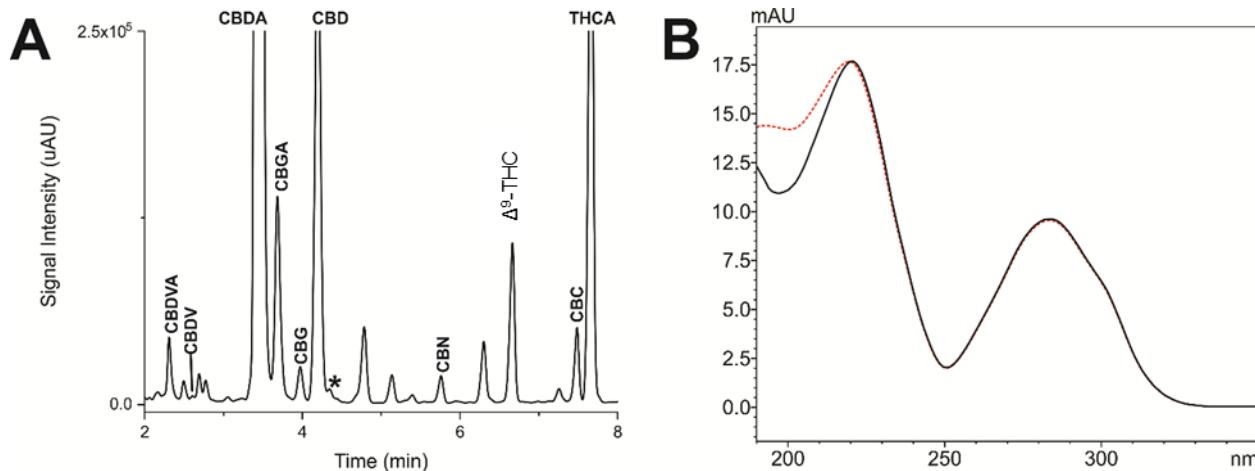
**Fig. 5-24. CBN in Plant Sample 5 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 5-25. CBNA in NRC HEMP-1 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 5-26. Chromatogram and absorbance spectra for tentatively identified CBN peak in Plant Sample 3.**

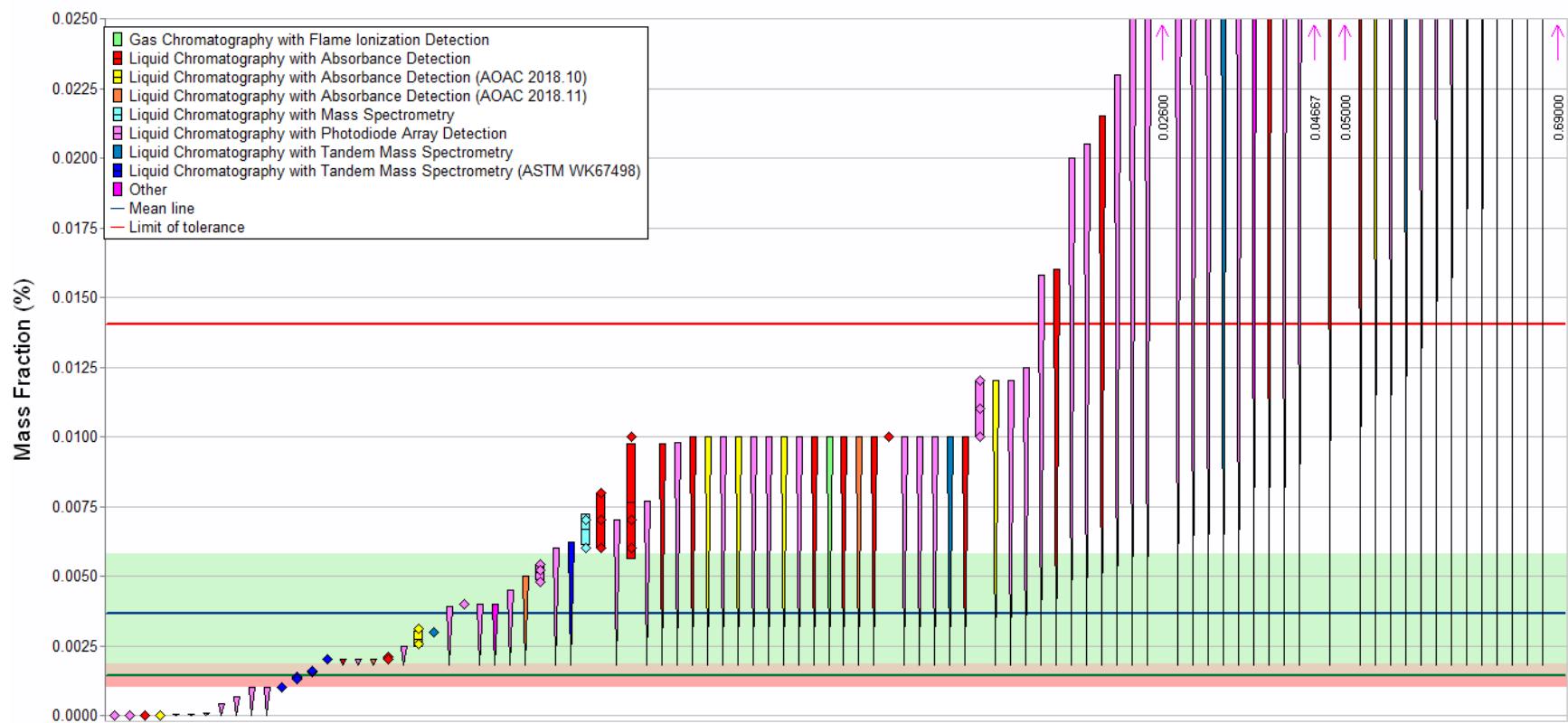
Panel A displays a LC-UV chromatogram at 220 nm for Plant Sample 3. Panel B displays the absorbance spectra for the tentatively identified CBN peak (solid black curve) and a CBN reference standard (dotted red curve). The chromatographic peak labeled “\*” was tentatively identified as THCV based on retention times of reference standards.

#### 5.4.2.6. THCV and THCVA

Target values for THCV and THCVA were assigned in NRC HEMP-1. The individual participant, consensus, and target mass fraction results for CBDV and CBDVA in NRC HEMP-1 are presented graphically in **Fig. 5-27** (THCV) and **Fig. 5-28** (THCVA). The consensus values were higher than the target values for THCV (155 %) and THCVA (11 %) in NRC HEMP-1. The consensus ranges for both THCV and THCVA extended above the target ranges in NRC HEMP-1, with the consensus value for THCVA falling within the target range. Approximately 91 % and 50 % of laboratories reporting quantitative results for THCV and THCVA in NRC HEMP-1, respectively, reported results outside the target range. Of laboratories reporting results outside of the target range, 80 % (THCV) and 100 % (THCVA) of laboratories reported results that were higher than the target range.

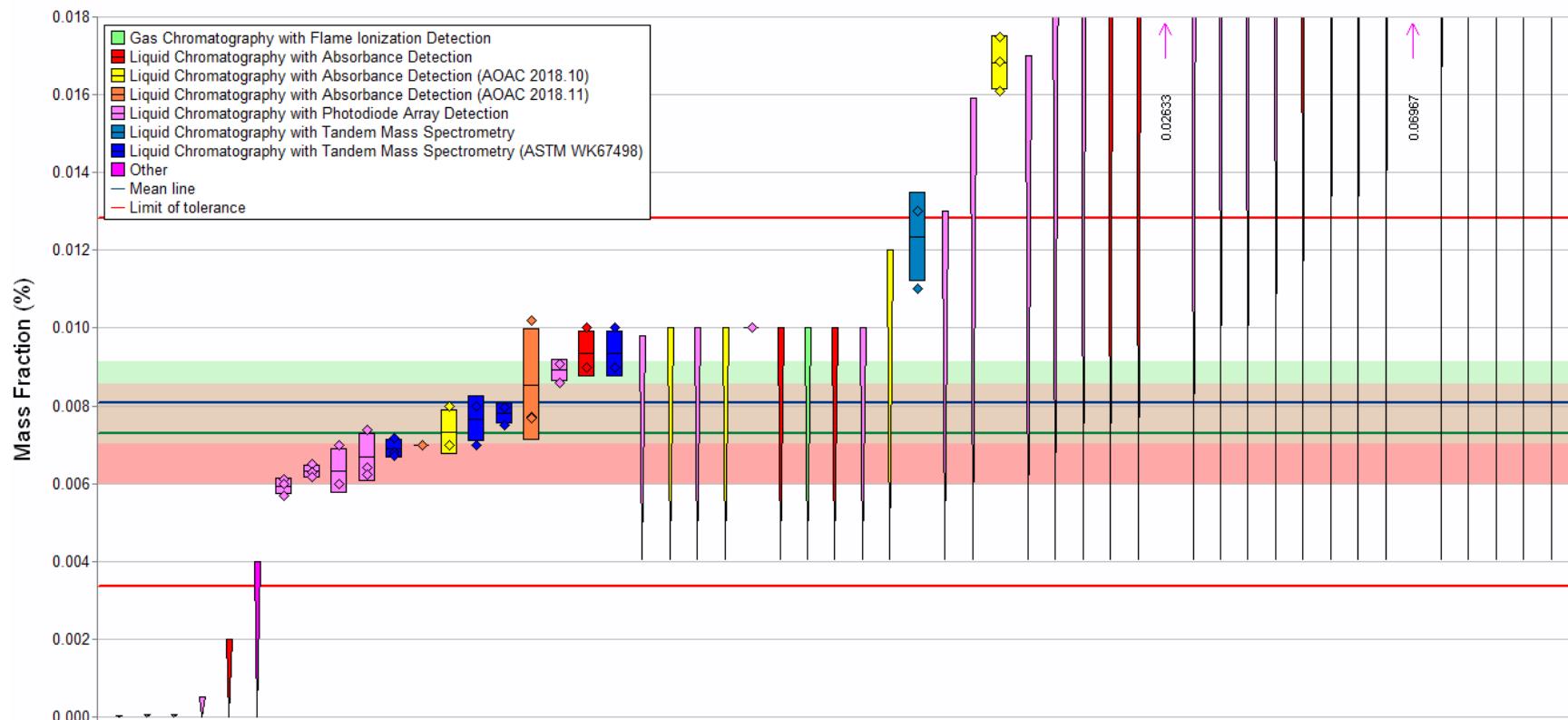
Similar to CBG and CBL, THCV and THCVA were present in NRC HEMP-1 at mass fractions an order of magnitude lower than the AOAC recommended LOQ for cannabinoids in hemp [16]. The low mass fraction of THCV and THCVA in the samples increases the influence of coeluting compounds and calibration curve bias. Approximately 85 % of participants reported use of either LC-ABS or LC-PDA as their analytical method for THCV and THCVA measurements (**Table 5-6**). Similar to the other cannabinoids, the monitoring wavelength for THCV and THCVA in most cases is between 220 nm and 230 nm, which is not a selective range. The selectivity in LC-ABS and LC-PDA methods comes from the chromatographic separation of the cannabinoids, which should be thoroughly investigated in existing methods, especially when the analytes, such as THCV and THCVA, are known to be present in low levels in the samples. An example is shown in **Fig. 5-29** of the spectrum collected over a peak that was initially identified as THCV in Plant Sample 3, as shown by the asterisk over the peak in the chromatogram in **Fig. 5-26 A**. As shown, the spectrum of the unknown peak did not align well with the THCV spectrum from the THCV standard. If laboratories are not taking steps to ensure that the cannabinoids detected in the analytical methods are verified, then analytes such as THCV can be misidentified.

Accuracy decreases when the amount of analyte in the sample nears the LOQ. In addition to taking steps to ensure the analyte of interest is confirmed, method LOQs must be determined appropriately and lower mass fraction analytes must be investigated using calibration curve ranges that do not extend orders of magnitude beyond the unknown analyte concentration. If the THCV and/or THCVA concentrations in unknown samples appear to fall at or below the lowest calibration point, additional calibrants should be made and a new curve produced to quantitate the low mass fraction sample more accurately (Section 3.4.3.4).



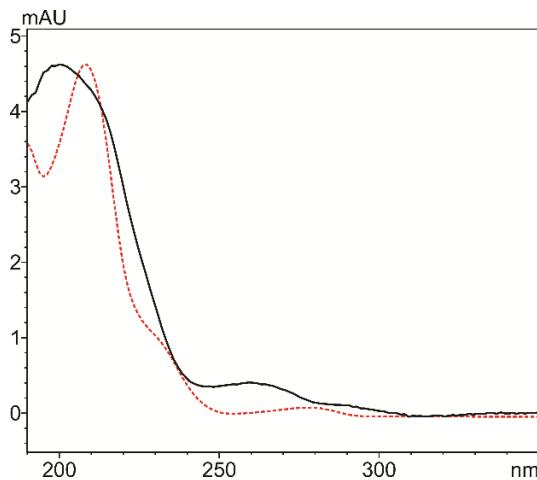
**Fig. 5-27. THCV in NRC HEMP-1 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 5-28. THCVA in NRC HEMP-1 (data summary view – analytical method).**

In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A downward triangle represents data reported as an LOQ or threshold value. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \leq 2$ . The red shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ( $U_{\text{NIST}}$ ) and represents the range that results in an acceptable  $Z_{\text{NIST}}$  score,  $|Z_{\text{NIST}}| \leq 2$ . The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



**Fig. 5-29. Absorbance spectra for chromatographic peak identified as THCV in Plant Sample 3.**

The absorbance spectra collected for the chromatographic peak identified as THCV is displayed as a solid black curve. The dotted red curve represents the spectrum of a THCV reference standard.

#### 5.4.2.7. $\Delta^8$ -THC

No plant samples were assigned target values for  $\Delta^8$ -THC, as the mass fractions for  $\Delta^8$ -THC were below the limits of quantitation in both NIST methods. Between 74.0 % and 86.7 % of laboratories reporting results reported values for  $\Delta^8$ -THC as below their LOQ. The consensus values of  $\Delta^8$ -THC in the six plant samples were the same order of magnitude as those of CBNA. Laboratories using LC-ABS or LC-PDA as their analytical methods may have had significant coelution from CBNA, falsely elevating their reported  $\Delta^8$ -THC values as shown in Fig. 3-14. While  $\Delta^8$ -THC was detected in NRC HEMP-1 by the NIST LC-MS/MS method (data not shown),  $\Delta^8$ -THC was not identified using the NIST LC-PDA method as is shown in Fig. 5-10 and Fig. 5-26 for Plant Sample 4 and Plant Sample 3, respectively.

#### 5.4.3. Candidate Analytical Methods

NIST provided a list of nine candidate standard methods from AOAC International and ASTM International for participants to use if an in-house analytical method was not available. The within- and between-laboratory variabilities for the minor cannabinoids are summarized for candidate method/sample pairs for which participants reported at least two independent measurements for a sample and at least three laboratories reported data (Table 5-8).

**Table 5-8. Within- and between-laboratory variabilities for minor cannabinoid measurements using candidate standardized analytical methods.**

Cannabinoids	NRC HEMP-1			Plant Sample 4			Plant Sample 6		
	<u>n</u> <sup>a</sup>	%RSD <sub>r</sub>	%RSD <sub>R</sub>	<u>n</u> <sup>a</sup>	%RSD <sub>r</sub>	%RSD <sub>R</sub>	<u>n</u> <sup>a</sup>	%RSD <sub>r</sub>	%RSD <sub>R</sub>
<b>AOAC 2018.10</b>									
CBC	5	1.2	23.3	7	4.4	19.4	7	3.9	48.2
CBCA	5	4.1	20.4	6	4.8	15.2	6	3.9	19.6
CBDV	6	2.4	91.6	3	1.3	175	3	4.1	200
CBDVA	5	2.3	18.5	4	3.3	6.3	5	10.7	260
CBG	3	7.6	71.8	6	2.1	3.9	3	9.5	123
CBN	9	1.9	10.6	4	4.6	325	NA	NA	NA
<b>AOAC 2018.11</b>									
CBC	7	1.8	18.7	7	4.9	10.8	4	2.2	54.5
CBG	NA	NA	NA	5	4.3	54.5	4	2.2	54.8
CBN	6	2.2	8.3	NA	NA	NA	NA	NA	NA
<b>ASTM WK67498</b>									
CBN	4	3.8	18.0	5	5.0	18.3	5	4.6	44.5

<sup>a</sup> n = number of laboratories

NA = not applicable because not enough laboratories reported data

#### *AOAC 2018.10*

Of the minor cannabinoids that met the requirements for assessment (**Table 5-8**), CBCA, CBDV, and CBDVA, were not included in the single laboratory validation of this candidate method [22]. The %RSD<sub>r</sub>s published in the method for CBC, CBG, and CBN measured in the independent cannabis flower samples were not consistently within the ≤ 5 % AOAC recommendation [21], with RSD<sub>r</sub>s ranging from 2.17 % to 7.72 % for CBC, from 1.06 % to 10.08 % for CBG, and from 2.77 % to 8.45 % for CBN [22]. The within-laboratory variability for CBC, CBCA, CBDV, and CBN by participants in this exercise was ≤ 5 % as recommended by AOAC [16] and within the published repeatabilities in the method [22]. The within-laboratory variability of CBG in NRC HEMP-1 and Plant Sample 6 was within the published method %RSD<sub>r</sub>s but above the recommended AOAC SMPR %RSD<sub>r</sub>; however, the concentration of CBG in the hemp plant materials was an order of magnitude lower than the method LOQ, which increases the difficulty of making precise measurements as the analyte signal is close to the background noise. The method LOQ was set using the Environmental Protection Agency method detection limit protocol [49]. For laboratories employing the AOAC 2018.10 method, the LOQs set in the method should be applied unless an independent LOQ is determined using an accepted protocol. While CBDVA was not included in the AOAC 2018.10 method development, the %RSD<sub>r</sub> for CBDVA in Plant Sample 6 was higher than recommended by AOAC [16]. Similar to CBG, the mass fraction of CBDVA in Plant Sample 6 was below the recommended LOQ in the AOAC SMPR, which would result in decreased precision. With the exception of CBDVA and CBG measurements in Plant Sample 4, all between-laboratory variabilities for the minor cannabinoids included in **Table 5-8** were above the AOAC recommended parameters of ≤ 10 % [16]. The lower mass fraction of all the minor cannabinoids in the hemp samples likely decreased the reproducibility of measurements in the samples. The reproducibility of AOAC 2018.10 had not been published at the time of this report.

### AOAC 2018.11

The average within-laboratory repeatability (%RSD<sub>r</sub>) published for AOAC 2018.11 was reported from two separate analysts for 11 cannabinoids in dried plant material, which included all of the minor cannabinoids in Exercise 2, except CBCA, CBL, CBLA, CBNA, THCV, and Δ<sup>8</sup>-THC [7]. The %RSD<sub>r</sub>s reported by the two analysts separately for the analysis of CBC, CBG, and CBN in cannabis dried plant matrix were within the recommended AOAC performance criteria [16, 21]. The mass fractions of CBC (0.0104 % to 0.0347 %) and CBN (0.0310 %) in the cannabis flower samples from the method [7] were within the range of at least one hemp sample included in Exercise 2. The mass fraction of CBG (0.0390 % to 0.0500 %) in the cannabis dried flower matrix from the published method [7] was an order of magnitude higher than the mass fraction of CBG in the Exercise 2 hemp samples. The %RSD<sub>r</sub>s for CBC, CBG, and CBN measurements by participants using AOAC 2018.11 were within both the published %RSD<sub>r</sub>s in the method [7] and the AOAC SMPR recommendation [16]. With the exception of CBN measurements in NRC HEMP-1, the between-laboratory variabilities (%RSD<sub>R</sub>) observed for the three minor cannabinoids in the hemp samples were above the 10 % requirement [16]. The mass fraction of CBC, CBG, and CBN in the hemp samples were below the recommended LOQ in the AOAC SMPR [16], which decreased the reproducibility of measurements in the samples. Observed reproducibility of AOAC 2018.11 had not been published at the time of this report.

### ASTM WK67498

The ASTM WK67498 method was developed following the AOAC [23] and ASTM [24] guidelines. The %RSD<sub>r</sub> published for ASTM WK67498 was based on 5 lots of the same hemp sample with %RSD<sub>r</sub> between 1.4 % and 4.9 % for hemp samples containing 0.377 % to 0.457 % of CBN ( $n = 3$ , [25]), which were all within the published requirements of  $\leq 5\%$  [16]. The average within-laboratory variability observed for the hemp samples in Exercise 2 was within the %RSD<sub>r</sub> published in the method [25] and in the SMPR [16]. The between-laboratory variabilities observed for CBDA measured using ASTM WK67498 in the hemp samples were above the  $\leq 8\%$  requirement for NRC HEMP-1 and Plant Sample 6 and above the  $\leq 6\%$  requirement for Plant Sample 4 [16]. Observed reproducibility of ASTM WK67498 on hemp had not been published at the time of this report. The between-laboratory variabilities (%RSD<sub>R</sub>) observed for CBN in the hemp samples were above the 10 % requirements established by AOAC [16, 21]. The mass fraction of CBN in the hemp samples was one to two orders of magnitude lower than the hemp samples used for the repeatability study in the method [25] and an order of magnitude lower than the AOAC recommended LOQ for cannabinoids in hemp [16] for Plant Sample 4 and Plant Sample 6. The between-laboratory variability of CBN measurements will be higher with such low mass fractions of CBN in the samples. Observed reproducibility of ASTM WK67498 had not been published at the time of this report.

## 5.5. Conclusions

Laboratories provided accurate consensus mass fractions for CBC, CBCA, CBGA, CBL, CBN, CBNA, and THCV in the plant samples with assigned target values for those cannabinoids. Participants had a more difficult time accurately quantitating CBDV, CBDVA, CBG, CBLA, and THCV. In general, the consensus values were higher than the target values, which was attributed to calibration bias

and potential interferences from coeluting species and/or misidentification of a cannabinoid altogether. Examples of NIST LC-PDA measurements of the Exercise 2 plant samples were used to illustrate potential difficulties with lower mass fraction cannabinoid analysis. To reduce the impact of interfering matrix components on the analysis of minor cannabinoids, chromatographic methods should be thoroughly evaluated and checked periodically to ensure baseline separation of known cannabinoids with similar retention times. Laboratories that report mean values outside the target range could experiencing calibration bias at the lower end of their calibration curve. Calibration curves should closely represent the extracted concentration of minor cannabinoids in the samples to increase the accuracy of the analysis.

## References

- [1] ISO (2017) ISO Guide 35:2017 Reference materials - Guidance for characterization and assessment of homogeneity and stability. <https://www.iso.org/standard/60281.html>.
- [2] Barber CA, Bryan Sallee CE, Burdette CQ, Kotoski SP, Phillips MM, Wilson WB, Wood LJ (2022) Cannabis Laboratory Quality Assurance Program: Exercise 2 Moisture Final Report. (National Institute of Standards and Technology, Gaithersburg, MD), NIST Internal Report (IR) NIST IR 8449. <https://doi.org/10.6028/NIST.IR.8449>.
- [3] Barber CA, Bryan Sallee CE, Burdette CQ, Kotoski SP, Phillips MM, Wilson WB, Wood LJ (2022) Cannabis Quality Assurance Program: Exercise 2 Toxic Elements Final Report. (National Institute of Standards and Technology, Gaithersburg, MD), NIST Interagency/Internal Report (IR) NIST IR 8452. <https://doi.org/10.6028/NIST.IR.8452>.
- [4] ISO 13528:2022, Statistical methods for use in proficiency testing by interlaboratory comparisons, pp. 53–4. <https://www.iso.org/standard/78879.html>.
- [5] Beauchamp CR, Camara JE, Carney J, Choquette SJ, Cole KD, DeRose PC, Duewer DL, Epstein MS, Kline MC, Lippa KA, Lucon E, Molloy J, Nelson MA, Phinney KW, Polakoski M, Possolo A, Sander LC, Schiel JE, Sharpless KE, Toman B, Winchester MR, Windover D (2021) *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*. (US Department of Commerce, Washington, DC), NIST Special Publication (SP) 260-136. <https://doi.org/10.6028/NIST.SP.260-136-2021>
- [6] NRC CRM HEMP-1 Certificate of Analysis. <https://nrc.canada.ca/en/certifications-evaluations-standards/certified-reference-materials/list/153/html>. Accessed 12 August 2022.
- [7] Vaclavik L, Benes F, Fenclova M, Hricko J, Krmela A, Svobodova V, Hajslova J, Mastovska K. (2019) Quantitation of cannabinoids in *Cannabis* dried plant materials, concentrates, and oils using liquid chromatography-diode array detection technique with optional mass spectrometric detection: single-laboratory validation study, first action 2018.11, *J AOAC Int* 102(6): 1822-1833. <https://doi.org/10.5740/jaoacint.18-0426>.
- [8] Abdur-Rahman M, Phillips MM, Wilson WB. *Cannabis Quality Assurance Program; Exercise 1 Final Report*. NIST IR 8385:1-194 (2021) <http://doi.org/10.6028/NIST.IR.8385>.
- [9] Wilson WB, Abdur-Rahman M. Chromatographia 5: 115-125 (2022) <https://doi.org/10.1007/s10337-021-04114-y>.
- [10] Wilson WB, Urbas AA, Abdur-Rahman M, Romares A, Mistek-Morabito E, Forensic Chemistry 37: 100550 (2024). <https://doi.org/10.1016/j.forc.2024.100550>.
- [11] University of Kentucky Hemp Proficiency Testing Program. <https://www.rs.uky.edu/regulatory/hpt/reports/HM20NOV-1CertOfAnal.pdf>. Accessed 12 August 2022.
- [12] Lemmon EW, Bell IH, Huber ML, and McLinden MO, "Thermophysical Properties of Fluid Systems" in NIST Chemistry WebBook, NIST Standard Reference Database Number 69, Eds. P.J. Linstrom and W.G. Mallard, National Institute of Standards and Technology, Gaithersburg MD, 20899, <https://webbook.nist.gov>, (retrieved May 3, 2023).

- [13] European Reference Materials Application Note 1.  
[https://crm.jrc.ec.europa.eu/graphics/cms\\_docs/erm1\\_english.pdf](https://crm.jrc.ec.europa.eu/graphics/cms_docs/erm1_english.pdf). Accessed 12 August 2022.
- [14] Koepke A, Lafarge T, Possolo A, Toman, B. (2020) NIST Consensus Builder. National Institute of Standards and Technology, US Department of Commerce.  
<https://consensus.nist.gov/app/nicob>. Accessed 6 February 2024.
- [15] Moreno-Sanz, G. (2016) Can you pass the acid test? Critical review and novel therapeutic perspectives of Δ9-Tetrahydrocannabinolic Acid A. *Cannabis and Cannabinoid Research* 1(1): 124-130. <https://doi.org/10.1089/can.2016.0008>.
- [16] Official Methods of Analysis of AOAC INTERNATIONAL, 22nd Ed. (2019) AOAC SMPR 2019.003 – Standard Method Performance Requirements (SMPRs®) for Quantitation of Cannabinoids in Plant Materials of Hemp (Low THC Varieties Cannabis sp.) (AOAC INTERNATIONAL, Gaithersburg, MD), [https://www.aoac.org/wp-content/uploads/2020/11/SMPR202019\\_003.pdf](https://www.aoac.org/wp-content/uploads/2020/11/SMPR202019_003.pdf), Accessed last March 19, 2023.
- [17] Meija, J, McRae, G, Miles, CO, Melanson, JE. (2022) Thermal stability of cannabinoids in dried cannabis: a kinetic study. *Analytical and Bioanalytical Chemistry*, 414:377-384. <https://doi.org/10.1007/s00216-020-03098-2>.
- [18] Reason, DA, Crainger, MNC, Lane, JR. (2022) Optimal storage conditions of commercial cannabis crops. *Industrial & Engineering Chemistry Research*, 61, 14691-14701. <https://doi.org/10.1021/acs.iecr.2c02071>
- [19] University of Kentucky College of Agriculture, Food and Environment, Hemp Proficiency Testing Summary Reports, Hemp Scheme Summary Statistics, 2019 – 2022. <https://www.rs.uky.edu/regulatory/hpt/reports.php>. Accessed 24 of July 2023.
- [20] Lazarjani, MP, Torres, S, Hooker, T, Fowlie, C, Young, O, Seyfoddin, A. (2020) Methods for quantitation of cannabinoids: a narrative review. *Journal of Cannabis Research*, 2: 35. <https://doi.org/10.1186/s42238-020-00040-2>.
- [21] AOAC SMPR® 2017.002:Standard Method Performance Requirements (SMPRs) for Quantitation of Cannabinoids in Dried Plant Materials, *Journal of AOAC INTERNATIONAL*, Volume 100, Issue 4, 1 July 2017, Pages 1204–1207, [https://doi.org/10.5740/jaoacint.SMPR2017\\_002](https://doi.org/10.5740/jaoacint.SMPR2017_002), Accessed last March 19, 2023.
- [22] Mudge, EM, Murch, SJ, Brown, PN. (2017) Leaner and greener analysis of cannabinoids. *Analytical and Bioanalytical Chemistry*, 409, 3153-3163. <https://doi.org/10.1007/s00216-017-0256-3>.
- [23] Dr. Latimer, George W, Jr. (ed.), 'Guidelines for Dietary Supplements and Botanicals', in Dr. George W Latimer, Jr. (ed.), *Official Methods of Analysis of AOAC INTERNATIONAL*, 22 (New York, 2023; online edn, Oxford Academic, 4 Jan. 2023), <https://doi.org/10.1093/9780197610145.005.011>. Accessed 5 of Jan. 2024.
- [24] ASTM International (2019) ASTM D8282-19 – Standard Practice for Laboratory Test Method Validation and Method Development (ASTM International, West Conshohocken, PA). <https://dx.doi.org/10.1520/D8282-19>
- [25] McRae, G, Melanson, JE. (2020) Quantitative determination and validation of 17 cannabinoids in cannabis and hemp using liquid chromatography-tandem mass

- spectrometry. *Analytical and Bioanalytical Chemistry*, 412, 7381-7393. doi: <https://doi.org/10.1007/s00216-020-02862-8>.
- [26] Sgro, S, Lavezzi, B, Caprari, C, Polito, M, D'Elia, M, Lago, G, Furlan, G, Girotti, S, Ferri, EN. (2021) Delta9-THC determination by the EU official method: evaluation of measurement uncertainty and compliance assessment of hemp samples. *Analytical and Bioanalytical Chemistry*, 413 (13), 3399 – 3410. <https://doi.org/10.1007/s00216-021-03283-x>.
- [27] C. Citti, F. Russo, S. Sgro, A. Gallo, A. Zanotto, F. Forni, M.A. Vandelli, A. Lagana, C.M. Montone, G. Gigli, G. Cannazza, Pitfalls in the analysis of phytocannabinoids in cannabis inflorescence, *Anal Bioanal Chem* 412(17) (2020) 4009-4022. <https://doi.org/10.1007/s00216-020-02554-3>.
- [28] Dussy FE, Hamberg C, Luginbuhl M, Schwerzmann T, Briellmann TA, Isolation of Delta-9-THCA-A from hemp and analytical aspects concerning the determination of Delta-9-THC in cannabis products, *Forensic Sci Int* 149(1) (2005) 3-10. <https://doi.org/10.1016/j.forsint.2004.05015>.
- [29] Citti C, Braghierioli D, Vandelli MA, Cannazza G, Pharmaceutical and biomedical analysis of cannabinoids: A critical review, *J Pharm Biomed Anal* 147 (2018) 565-579. <https://doi.org/10.1016/j.jpba.2017.06.003>.
- [30] Beres T, Cernochova L, Cavar Zeljkovic S, Benicka S, Gucky T, Bercak M, Tarkowski P, Intralaboratory comparison of analytical methods for quantification of major phytocannabinoids, *Anal Bioanal Chem* 411(14) (2019) 3069-3079. <https://doi.org/10.1007/s00216-019-01760-y>.
- [31] Cardenia V, Gallina T, Scappini S, Rubino RC, Rodriguez-Estrada MT. Development and validation of a fast gas chromatography/mass spectrometry method for the determination of cannabinoids in Cannabis sativa L, *J Food Drug Anal* 26(4) (2018) 1283-1292. <https://doi.org/10.1016/j.jfda.2018.06.001>.
- [32] Fodor B, Molnár-Perl I, The role of derivatization techniques in the analysis of plant cannabinoids by gas chromatography mass spectrometry, *TrAC Trends in Analytical Chemistry* 95 (2017) 149-158. <https://doi.org/10.1016/j.trac.2017.07.022>.
- [33] Nahar L, Guo M, Sarker SD, Gas chromatographic analysis of naturally occurring cannabinoids: A review of literature published during the past decade, *Phytochemical Analysis* 31(2) (2020) 135-146. <https://doi.org/10.1002/pca.2886>.
- [34] Leghissa A., Hildenbrand ZL, Schug KA, A review of methods for the chemical characterization of cannabis natural products, *J Sep Sci* 41(1) (2018) 398-415. <https://doi.org/10.1002/jssc.201701003>.
- [35] Pourseyed ML, Torres S, Hooker T, Fowlie C, Young O, Seyfoddin A, Methods for quantification of cannabinoids: a narrative review, *J Cannabis Res* 2(1) (2020) 35.
- [36] Agricultural Improvement Act of 2018, H.R.2, 115th Congress (2018). <https://www.congress.gov/115/bills/hr2/BILLS-115hr2enr.pdf>. Accessed 6 of February 2024.
- [37] Sill M. (2021). The future of the CBD industry in 2022 and beyond. Forbes Business Council, Oct. 21, 2021. <https://www.forbes.com/sites/forbesbusinesscouncil/2021/10/21/the-future-of-the-cbd-industry-in-2022-and-beyond/?sh=28ad923525fd>. Accessed 6 of February 2024.

- [38] Miller OS, Elder Jr. EJ, Jones KJ, Gidal, BE. (2022). Analysis of cannabidiol (CBD) and THC in nonprescription consumer products: implications for patients and practitioners. *Epilepsy & Behavior*, 127, 108514. <https://doi.org/10.1016/j.yebeh.2021.108514>.
- [39] Cheng, Y, Kerrigan, S. (2023). Factors Influencing the in situ formation of Δ9-THC from cannabidiol during GC-MS analysis. *Drug Test Anal*. <https://doi.org/10.1002/dta.3617>.
- [40] Cheng, Y, Kerrigan, S. (2023). Differentiation of hemp from marijuana using a qualitative decision-point assay. *Forensic Chemistry*, 37. <https://doi.org/10.1016/j.forc.2023.100541>.
- [41] Stone N, Murphy A, England T, O'Sullivan S. *Br J Pharmacol*. 177: 4330-4352 (2020) <https://doi.org/10.1111/bph.15185>.
- [42] Walsh KB, McKinney AE, Holmes, AE. (2021). Minor cannabinoids: biosynthesis, molecular pharmacology and potential therapeutic uses. *Front Pharmacol*. Nov. 29; <https://doi.org/10.3389/fphar.2021.777804>.
- [43] Zagozen M, Cerenak A, Kreft S. *Acta Pharm.* 71: 355-364 (2021) <https://doi.org/10.2478/acph-2021-0021>.
- [44] Wang Y, Avula B, ElSohly M, Radwan M, Wang M, Wanas A, Mehmedic Z, Khan I. *Planta Med* 84: 260-266 (2018) <https://doi.org/10.1055/s-0043-124873>.
- [45] Gul W, Gul S, Radwan M, Wanas A, Mehmedic Z, Khan I, Sharaf M, ElSohly M. *J AOAC Intern* 98(6): 1523-1528 (2015) <https://doi.org/10.5740/jaoacint.15-095>.
- [46] Repka MA, Munjal M, ElSohly MA, Ross SA. *Drug Dev Ind Pharm* 32(1): 21-32 (2006) <https://doi.org/10.1080/03639040500387914>.
- [47] <https://emergency.cdc.gov/han/2021/han00451.asp>
- [48] Golomebek P, Muller M, Barthlott I, Scroll C, Lachenmeir DW. *Toxics*. 8 (2020) <https://doi.org/10.3390/toxics8020041>.
- [49] Environmental Protection Agency. Guidelines establishing test procedures for the analysis of pollutants; procedures for detection and quantification. In: 40 CFR pt. 136, Appendix D, rev. 1.11. 2002. <https://www.gpo.gov/fdsys/pkg/CFR-2011-title40-vol23/pdf/CFR-2011-title40-vol23-part136-appD.pdf>

## Appendix A. List of Symbols, Abbreviations, and Acronyms

**ABS**

absorbance detection

**ACN**

acetonitrile

**CannaQAP**

Cannabis Laboratory Quality Assurance Program

**CBC**

Cannabichrome

**CBCA**

cannabichromenic acid

**CBD**

cannabidiol

**CBDA**

cannabidiolic acid

**CBDV**

cannabidivarin

**CBDVA**

cannabidivarinic acid

**CBG**

cannabigerol

**CBGA**

cannabigerolic acid

**CBL**

cannabicyclol

**CBLA**

cannabicyclolic acid

**CBN**

cannabinol

**CBNA**

cannabinolic acid

**CRM**

certified reference material, a reference material that delivers a certified value for at least one property

**ESI**

electrospray ionization

**EtOH**

ethanol

**FA**

formic acid

**FID**

flame ionization detection

**GC**

gas chromatograph

**IS**

internal standard

**LC**

liquid chromatography

**LOQ**

limits of quantitation

**MeOH**

methanol

**MF**

mass fraction, expressed as a percentage

**MRM**

multiple reaction monitoring mode

**MS/MS**

tandem mass spectrometry

***n***

number of laboratories providing results of a given type

**NRC**

National Research Council of Canada

**PA**

phosphoric acid

**PDA**

photodiode array detection

**PTFE**

polytetrafluoroethylene

**QL**

quantification limit

**RM**

Reference Material, a reference material provided by NIST

**RSD**

relative standard deviation

**s\***

a robust estimate of standard deviation of participant values

**SD**  
standard deviation

**SI**  
International System of Units

**SMPR**  
Standard Method Performance Requirements

**SRM®**  
Standard Reference Material®, a CRM provided by NIST

**THCA**  
 $\Delta^9$ -tetrahydrocannabinolic acid

**THCV**  
tetrahydrocannabivarin

**THCVA**  
tetrahydrocannabivarinic acid

**UHPLC**  
ultra-high pressure liquid chromatograph

**UK-PT**  
University of Kentucky Proficiency Testing Program

**$U_{95}$**   
an approximate 95 % level of confidence expanded uncertainty on a certified value

**$U_{NIST}$**   
an approximate 95 % level of confidence expanded uncertainty assigned by NIST staff on a non-certified

**$x^*$**   
the consensus mean value of measurand  $X$

**$x_i$**   
a value for measurand  $X$  reported by the  $i^{\text{th}}$  participant

**$x_{NIST}$**   
the NIST value for measurand  $X$

**$Z'_{\text{comm}}$**   
Z-score with respect to community consensus value and its uncertainty

**$Z_{NIST}$**   
Z-score with respect to a target value and uncertainty assigned by NIST measurements

**%RSD<sub>r</sub>**  
relative within-laboratory repeatability precision

**%RSD<sub>R</sub>**  
relative between-laboratory reproducibility precision

**$\Delta^8\text{-THC}$**   
 $\Delta^8$ -tetrahydrocannabinol

**$\Delta^9\text{-THC}$**

$\Delta^9$ -tetrahydrocannabinol

**$\sigma_{\text{PT}}$**

standard deviation for proficiency assessment

**Appendix B. Tables of participant data.**

<u>Cannabinoids</u>	<u>Hemp Plant Samples</u>	<u>Marijuana Plant Samples</u>
$\Delta^9$ -THC	<b>Table B-1</b>	<b>Table B-2</b>
THCA	<b>Table B-3</b>	<b>Table B-4</b>
Total $\Delta^9$ -THC	<b>Table B-5</b>	<b>Table B-6</b>
CBD	<b>Table B-7</b>	<b>Table B-8</b>
CBDA	<b>Table B-9</b>	<b>Table B-10</b>
Total CBD	<b>Table B-11</b>	<b>Table B-12</b>
CBC	<b>Table B-13</b>	<b>Table B-14</b>
CBCA	<b>Table B-15</b>	<b>Table B-16</b>
CBDV	<b>Table B-17</b>	<b>Table B-18</b>
CBDVA	<b>Table B-19</b>	<b>Table B-20</b>
CBG	<b>Table B-21</b>	<b>Table B-22</b>
CBGA	<b>Table B-23</b>	<b>Table B-24</b>
CBL	<b>Table B-25</b>	<b>Table B-26</b>
CBLA	<b>Table B-27</b>	<b>Table B-28</b>
CBN	<b>Table B-29</b>	<b>Table B-30</b>
CBNA	<b>Table B-31</b>	<b>Table B-32</b>
THCV	<b>Table B-33</b>	<b>Table B-34</b>
THCVA	<b>Table B-35</b>	<b>Table B-36</b>
$\Delta^8$ -THC	<b>Table B-37</b>	<b>Table B-38</b>

**Table B-1. Data summary table for  $\Delta^9$ -THC in three hemp samples.**

Data in red font have been flagged as a data entry of zero or results that include text (e.g., "< LOQ" or "present").

Data in blue font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

Note: This table spans multiple pages; the NIST values and consensus values are included on all pages for convenience.

Target Consensus	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
				0.0318	0.0086				0.0663	0.0040				0.0295	0.0029
				0.037	0.014				0.068	0.014				0.0310	0.0088
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B001	0.072	0.075	0.075	0.0740	0.0017	0.065	0.07	0.072	0.0690	0.0036	0.034	0.033	0.034	0.0337	0.0006
B003						0.08317	0.08277	0.0713	0.0791	0.0067	0.03923	0.0342	0.02649	0.0333	0.0064
B004	0.039	0.039	0.039	0.0390	0.0000	0.065	0.065	0.065	0.0650	0.0000	0.019	0.019	0.019	0.0190	0.0000
B005	< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1		
B006	0.033	0.033	0.032	0.0327	0.0006	0.062	0.06	0.061	0.0610	0.0010	0.029	0.029	0.028	0.0287	0.0006
B007															
B008															
B009															
B012	0.04018	0.04158		0.0409	0.0010	0.05854	0.06079		0.0597	0.0016	0.02624	0.02581		0.0260	0.0003
B013	0.03	0.02	0.03	0.0267	0.0058	0.06	0.06	0.06	0.0600	0.0000	0.03	0.03	0.03	0.0300	0.0000
B014															
B015	0.02	0.02	0.02	0.0200	0.0000	0.05	0.05	0.05	0.0500	0.0000	0.02	0.02	0.02	0.0200	0.0000
B016	0.092	0.1042	0.0929	0.0964	0.0068	0.1025	0.1043	0.1032	0.1033	0.0009	0.0631	0.0565	0.0571	0.0589	0.0036
B018	<0.01	<0.01	<0.01			0.11	0.1	0.11	0.1067	0.0058	<0.01	<0.01	<0.01		
B020															
B021															
B022	0.082	0.077	0.077	0.0787	0.0029	0.088	0.082	0.083	0.0843	0.0032	<0.04	<0.04	<0.04		
B023	0.0295	0.0274	0.0261	0.0277	0.0017	0.0654	0.0663	0.0645	0.0654	0.0009	0.0304	0.0299	0.0295	0.0299	0.0005
B024	0.03	0.03	0.02	0.0267	0.0058	0.04	0.05	0.03	0.0400	0.0100	0.01	0.02		0.0150	0.0071
B025	0.04	0.07	0.05	0.0533	0.0153	0.1	0.12	0.08	0.1000	0.0200	0.05	0.06	0.04	0.0500	0.0100
B026	0.02	0.04	<0.01	0.0300	0.0141	0.06	0.04	0.05	0.0500	0.0100	<0.01	<0.01	<0.01		
B027	<0.025	<0.025	<0.025			0.0582	0.0588	0.0566	0.0579	0.0011	<0.025	<0.025	<0.025		
B028															
B029	<0.030	<0.030	<0.030			0.075	0.075	0.073	0.0743	0.0012	<0.030	<0.030	<0.030		
B030	<0.05	<0.05	<0.05			0.1	0.11	0.11	0.1067	0.0058	0.06	0.06	0.06	0.0600	0.0000
B031															
B032															
B033	< 0.5	< 0.5	< 0.5			0.06	0.06	0.07	0.0633	0.0058	< 0.5	< 0.5	< 0.5		
B035	0.04249	0.04415	0.04247	0.0430	0.0010	0.06473	0.06305	0.06264	0.0635	0.0011	0.02714	0.03188	0.0305	0.0298	0.0024
B036	0.04	0.05	0.05	0.0467	0.0058	0.07	0.08	0.07	0.0733	0.0058	0.04	0.03	0.03	0.0333	0.0058
B037															
B038															
B039															
B041	<0.064					0.069			0.0690		<0.064				
B042															
B043	0.043					0.073			0.0730		0.035				0.0350
B044	<0.075	<0.075	<0.075	0.0430		<0.075	<0.075	<0.075			<0.075	<0.075	<0.075		

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.0318	0.0086				0.0663	0.0040				0.0295	0.0029
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B045	detected	detected				detected	detected				detected	detected			
B046	0.03	0.03	0.03	0.0300	0.0000	0.06	0.06	0.06	0.0600	0.0000	0.03	0.03	0.03	0.0300	0.0000
B047	0.052	0.033	0.041	0.0420	0.0095	0.063	0.07	0.063	0.0653	0.0040	0.034	0.033	0.03	0.0323	0.0021
B048															
B049	0.03036	0.03075	0.02884	0.0300	0.0010	0.06012	0.06818	0.07249	0.0669	0.0063	0.02016	0.03094	0.03246	0.0279	0.0067
B051	0.03	0.03	0.03	0.0300	0.0000	0.07	0.08	0.09	0.0800	0.0100	0.02	0.04	0.04	0.0333	0.0115
B052	< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08		
B053	0.09	0.095	0.099	0.0947	0.0045	0.199	0.187	0.2	0.1953	0.0072	0.112	0.113	0.12	0.1150	0.0044
B054	0.02461	0.02757	0.02823	0.0283	0.0042	0.06845	0.06553	0.06628	0.0668	0.0015	0.03167	0.03129	0.02603	0.0297	0.0032
B055	0.037	0.0336	0.0349	0.0352	0.0017	0.0761	0.0718	0.0722	0.0734	0.0024	0.0281	0.0292	0.033	0.0301	0.0026
B057	0.0212	0.0199	0.0191	0.0201	0.0011	0.0573	0.0559	0.0575	0.0569	0.0009	0.023	0.0241	0.0218	0.0230	0.0012
B058	0.0415	0.0421	0.0393	0.0410	0.0015	0.069	0.0681	0.0639	0.0670	0.0027	0.0344	< 0.0297	< 0.0296	0.0344	
B059	< 1.5	< 1.5	< 1.5			< 1.5	< 1.5	< 1.5			< 1.5	< 1.5	< 1.5		
B060	0.06	0.06	0.06	0.0600	0.0000	0.08	0.08	0.1	0.0867	0.0115	0.04	0.04	0.03	0.0367	0.0058
B061	0.038	0.04	0.039	0.0390	0.0010	0.065	0.064	0.069	0.0660	0.0026	0.026	0.027	0.027	0.0267	0.0006
B062	< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08		
B063															
B064	0.033			0.0330		0.077			0.0770		0.027			0.0270	
B065	0.033	0.035	0.035	0.0343	0.0012	0.067	0.069	0.071	0.0690	0.0020	0.033	0.034	0.033	0.0333	0.0006
B066	< 0.003	< 0.003	< 0.003			0.0759	0.0733	0.0675	0.0722	0.0043	0.034	0.0309	0.0361	0.0337	0.0026
B068															
B069	0.05	0.05	0.05	0.0500	0.0000	0.12	0.08	< 0.01	0.1000	0.0283	0.04	0.04	< 0.01	0.0400	0.0000
B070	0.047	0.063	0.04	0.0500	0.0118	0.112	< 0.111	< 0.111	0.1120		< 0.067	< 0.067	< 0.067		
B071	0.03	0.02	0.02	0.0233	0.0058	0.05	0.05	0.05	0.0500	0.0000	0.02	0.02	0.02	0.0200	0.0000
B072	0.036	0.033	0.034	0.0343	0.0015	0.07	0.075	0.073	0.0727	0.0025	0.035	0.035	0.035	0.0350	0.0000
B073	0.0241			0.0241		0.0543			0.0543		0.0231			0.0231	
B074	0.04	0.04	0.04	0.0400	0.0000	0.06	0.06	0.06	0.0600	0.0000	0.07	0.07	0.07	0.0700	0.0000
B076	0.05353	0.05127	0.05001	0.0516	0.0018	0.0635	0.06371	0.06392	0.0637	0.0002	0.02832	0.02817	0.02895	0.0285	0.0004
B077															
B078															
B079															
B081	< 0.36	< 0.36	< 0.36			< 0.36	< 0.36	< 0.36			< 0.36	< 0.36	< 0.36		
B082	0.055	0.056	0.044	0.0517	0.0067	0.059	0.059	0.061	0.0597	0.0012	0.026	0.03	0.033	0.0297	0.0035
B084	0.04	0.03	0.035	0.0350	0.0050	0.07	0.07	0.07	0.0700	0.0000	0.03	0.03	0.03	0.0300	0.0000
B085	0.051	0.051	0.043	0.0483	0.0046	0.098	0.108	0.094	0.1000	0.0072	0.041	0.033	0.035	0.0363	0.0042
B086															
B087															
B088	0.041	0.036	0.039	0.0387	0.0025	0.061	0.064	0.072	0.0657	0.0057	0.028	0.03	0.024	0.0273	0.0031
B089	0.0307	0.0303	0.0291	0.0300	0.0008	0.043	0.0429	0.0481	0.0447	0.0030	0.032	0.0311	0.0263	0.0298	0.0031
B090	< 0.05	< 0.05	< 0.05			0.06	0.06	0.07	0.0633	0.0058	< 0.05	< 0.05	< 0.05		
B091	0.019	0.019	0.02	0.0193	0.0006	0.046	0.043	0.046	0.0450	0.0017	0.012	0.015	0.015	0.0140	0.0017
B092	0.01	0.01	0.01	0.0100	0.0000	0.05	0.06	0.05	0.0533	0.0058	0.01	0.02	0.02	0.0167	0.0058
B094	0.04	0.04	0.04	0.0400	0.0000	0.06	0.07	0.06	0.0633	0.0058	0.03	0.03	0.03	0.0300	0.0000

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.0318 0.037	0.0086 0.014				0.0663 0.068	0.0040 0.014				0.0295 0.0310	0.0029 0.0088
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B095	0.048	0.049	0.048	0.0483	0.0006	0.067	0.064	0.068	0.0663	0.0021	0.037	0.037	0.037	0.0370	0.0000
B096	0.034	0.039	0.033	0.0353	0.0032	0.054	0.059	0.059	0.0573	0.0029	0.028	0.027	0.027	0.0273	0.0006
B097	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
<b>B098</b>						<b>0.01</b>	<b>0.04</b>	<b>0.01</b>	<b>0.0200</b>	<b>0.0173</b>					
B099	0.034			0.0340		0.06			0.0600					0.0270	
B100	<0.04	<0.04				0.07	0.07		0.0700		<0.04	<0.04			
B101	<1	<1	<1			<1	<1	<1			<1	<1	<1		
B102	0.0311	0.0281	0.0277	0.0290	0.0019	0.0592	0.0594	0.0608	0.0598	0.0009	0.0304	0.0293	0.029	0.0296	0.0007
B104	<0.25	<0.25	<0.25			<0.25	<0.25	<0.25			<0.25	<0.25	<0.25		
B105															
B106	0.034	0.032	0.033	0.0330	0.0010	0.066	0.061	0.062	0.0630	0.0026	0.026	0.029	0.026	0.0270	0.0017
B108															
B109	0.03	0.027	0.027	0.0280	0.0017	0.064	0.065	0.056	0.0617	0.0049	0.03	0.03	0.03	0.0300	
B110	0.055	0.055	0.056	0.0553	0.0006	0.06	0.07	0.07	0.0667	0.0058	0.03	0.03	0.03	0.0300	
B111															
<b>B113</b>	<b>0.07</b>	<b>0.09</b>	<b>0.08</b>	<b>0.0800</b>	<b>0.0100</b>	0.07	0.06	0.06	0.0633	0.0058	0.04	0.04	0.04	0.0400	
B114															
B115	0.0169	0.0163	0.0186	0.0173	0.0012	0.0535	0.0485	0.0513	0.0511	0.0025	0.0155	0.017	0.0156	0.0160	0.0008
B116	0.031	0.0366	0.0313	0.0330	0.0032	0.0603	0.0613	0.0595	0.0604	0.0009	0.0276	0.0261	0.0262	0.0266	0.0008
<b>B117</b>	0.02	0.021	0.022	0.0210	0.0010	<b>0.037</b>	<b>0.042</b>	<b>0.035</b>	<b>0.0380</b>	<b>0.0036</b>	0.021	0.015	0.017	0.0177	0.0031
B120															
B121															
B122															
B124	0.0405	0.0445	0.037	0.0407	0.0038	0.0625	0.0655	0.0655	0.0645	0.0017	0.032	0.032	0.034	0.0327	0.0012
B125	0.034	0.035	0.036	0.0350	0.0010	0.06	0.06	0.06	0.0600		0.027	0.027	0.026	0.0267	0.0006
<b>B126</b>	<b>0.081</b>	<b>0.08</b>	<b>0.079</b>	<b>0.0800</b>	<b>0.0010</b>										
B127	0.0551	0.0544	0.0537	0.0544	0.0007	0.0657	0.067	0.0712	0.0680	0.0029	0.0351	0.0357	0.0364	0.0357	0.0007
B129															
B130	0.04	0.04	0.04	0.0400		0.06	0.06	0.06	0.0600		0.03	0.03	0.03	0.0300	
<b>B131</b>	0.0376	0.0376	0.0364	0.0372	0.0007	<b>0.0995</b>	<b>0.1032</b>	<b>0.0981</b>	<b>0.1003</b>	<b>0.0026</b>	0.0405	0.0413	0.042	0.0413	0.0008
B132	0.043	0.041	0.041	0.0417	0.0012	0.088	0.086	0.071	0.0817	0.0093	0.037	0.037	0.036	0.0367	0.0006
<b>B133</b>	<0.02	<0.02	<0.02			0.08	0.07	0.1	0.0833	0.0153	<0.02	<0.02	<0.02		
B135	0.02	0.03	0.02	0.0233	0.0058	0.05	0.05	0.05	0.0500		0.02	0.02	0.02	0.0200	
B136	0.038			0.0380		0.062			0.0620		0.036			0.0360	
B137	0.047	0.04	0.039	0.0420	0.0044	0.081	0.073	0.082	0.0787	0.0049	0.039	0.039	0.042	0.0400	0.0017
B140	0.01	0.01	0.01	0.0100		0.07	0.06	0.07	0.0667	0.0058	<0.01	<0.01	<0.01		
B141	0.04	0.04	0.032	0.0373	0.0046	0.061	0.066	0.063	0.0633	0.0025	0.034	0.032	0.033	0.0330	0.0010
B142	0.04	0.041	0.04	0.0403	0.0006	0.084	0.061	0.061	0.0687	0.0133	0.039	0.038	0.036	0.0377	0.0015
B144	0.04	0.04	0.04	0.0400		0.08	0.08	0.08	0.0800		0.04	0.05	0.04	0.0433	0.0058
B146	0.0274	0.0263	0.029	0.0276	0.0014	0.0577	0.064	0.0628	0.0615	0.0033	0.0271	0.0247	0.0241	0.0253	0.0016
B147	0.031	0.035	0.029	0.0317	0.0031	0.061	0.066	0.073	0.0667	0.0060	0.032	0.03	0.035	0.0323	0.0025
B148															
B149	0.0251	0.031	0.023	0.0264	0.0041	0.05353	0.061	0.054	0.0562	0.0042	0.03	0.021	0.02	0.0237	0.0055

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.0318 0.037	0.0086 0.014				0.0663 0.068	0.0040 0.014				0.0295 0.0310	0.0029 0.0088
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B151	0.014	0.014	0.014	0.0140		0.086	0.086	0.085	0.0857	0.0006	0.026	0.032	0.028	0.0287	0.0031
B152															
B153	0.036	0.037	0.033	0.0353	0.0021	0.082	0.077	0.087	0.0820	0.0050	0.038	0.039	0.038	0.0383	0.0006
B154	0.024	0.022	0.023	0.0230	0.0010	0.054	0.058	0.072	0.0613	0.0095	0.025	0.032	0.029	0.0287	0.0035
B157	<0.05					0.06			0.0600		<0.05	<0.05			
B158	<0.05	<0.05	<0.05			0.06	0.07	0.07	0.0667	0.0058	<0.05	<0.05	<0.05		
B159	0.0525	0.0556	0.0513	0.0531	0.0022	0.0833	0.076	0.0763	0.0785	0.0041	0.0422	0.0413	0.0425	0.0420	0.0006
B160	0.04917	0.04298	0.04587	0.0460	0.0031	0.09551	0.09279	0.09405	0.0941	0.0014	0.04894	0.0506	0.05242	0.0507	0.0017
B161	0.047	0.047	0.046	0.0467	0.0006	0.074	0.069	0.075	0.0727	0.0032	0.032	0.035	0.035	0.0340	0.0017
B163	0.0523	0.0458	0.0458	0.0480	0.0038	0.0936	0.097	0.0761	0.0889	0.0112	0.0522	0.0534	0.0487	0.0514	0.0024
B164	0.12	0.11	0.11	0.1133	0.0058	0.08	0.13	0.16	0.1233	0.0404	0.1	0.08	0.1	0.0933	0.0115
B165															
B166	0.03	0.03	0.03	0.0300		0.06	0.06	0.06	0.0600		0.03	0.03	0.03	0.0300	
B167															
B168	0.0312	0.031	0.0312	0.0311	0.0001	0.0609	0.0601	0.0576	0.0595	0.0017	0.0313	0.031	0.031	0.0311	0.0002
B169	<0.15	<0.15				<0.15	<0.15				<0.15	<0.15			
B170															
B171	0.02	0.02		0.0200		0.08	0.07		0.0750	0.0071	0.04	0.04		0.0400	
B172	0.09936	0.06568	0.0488	0.0713	0.0257	0.12808	0.14848	0.11456	0.1304	0.0171	<0.031	<0.031	0.0932	0.0932	
B173	<0.0152	<0.0147	<0.0146			0.04016	0.04219	0.0431	0.0418	0.0015	0.01568	0.01509	0.01529	0.0154	0.0003
B174	<0.06	<0.06	<0.06			<0.06	0.06	0.07	0.0650	0.0071	<0.06	<0.06	<0.06		
B175	<0.05	<0.05	<0.05			0.1	0.09	0.09	0.0933	0.0058	<0.05	<0.05	<0.05		
B176	<0.0002	<0.0002	<0.0002			0.086	0.089	0.088	0.0877	0.0015	0.044	0.046	0.046	0.0453	0.0012
B178	0.0266	0.0266	0.0208	0.0247	0.0033	0.0679	0.0707	0.0678	0.0688	0.0016	0.0345	0.0331	0.0332	0.0336	0.0008
B181	<0.0077					0.09			0.0900		0.05			0.0500	
B182	<0.05	<0.05				0.08	0.08	0.08	0.0800		<0.05	<0.05	<0.05		
B183	0.0351	0.0387	0.0365	0.0368	0.0018	0.063	0.0644	0.0619	0.0631	0.0013	0.0491	0.0306	0.043	0.0409	0.0094
B184	<0.047	<0.047	<0.047			0.067	0.071	0.071	0.0697	0.0023	<0.047	<0.047	<0.047		
B185															
B186	0.071	0.069	0.06	0.0667	0.0059	0.062	0.065	0.068	0.0650	0.0030	0.031	0.031	0.031	0.0310	
B187	<0.06	<0.06	<0.06			0.0709	0.071	0.0793	0.0737	0.0048	<0.06	<0.06	<0.06		
B188															
B189	0.0377	0.0342	0.0346	0.0355	0.0019	0.065	0.0604	0.0612	0.0622	0.0025	0.0344	0.0339	0.0327	0.0337	0.0009
B190	0.06	0.06	0.06	0.0600		0.07	0.07	0.07	0.0700		0.04	0.04	0.04	0.0400	
B192	0.03224	0.03197	0.03227	0.0322	0.0002	0.06465	0.06517	0.06718	0.0657	0.0013	0.03101	0.03151	0.03078	0.0311	0.0004
B193	<0.113	<0.102	<0.119			<0.116	<0.107	<0.113			<0.122	<0.125	<0.121		
B194															
B195	0.052	0.053	0.055	0.0533	0.0015	0.066	0.066	0.068	0.0667	0.0012	0.026	0.026	0.025	0.0257	0.0006
B196															
B198	0.13	0.13	0.13	0.1300		0.08	0.07	0.09	0.0800	0.0100	<0.07	<0.07	<0.07		
B199	0.05	0.05	0.05	0.0500		0.06	0.05	0.05	0.0533	0.0058	0.02	0.02	0.02	0.0200	
B200															
B202	0.0332	0.028	0.0278	0.0297	0.0031	0.0663	0.0646	0.0676	0.0662	0.0015	0.0252	0.0257	0.0262	0.0257	0.0005

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4				Plant Sample 6					
Target Consensus				0.0318 0.037	0.0086 0.014				0.0663 0.068	0.0040 0.014				0.0295 0.0310	0.0029 0.0088
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B204	0.031	0.031	0.033	0.0317	0.0012	0.068	0.061	0.065	0.0647	0.0035	0.03	0.03	0.032	0.0307	0.0012
B205	0.063	0.065	0.055	0.0610	0.0053	0.192	0.182	0.184	0.1860	0.0053	0.091	0.085	0.085	0.0870	0.0035
B206	0.035	0.033	0.034	0.0340	0.0010	0.057	0.053	0.063	0.0577	0.0050	0.028	0.027	0.024	0.0263	0.0021
B207	0.0487	0.0481	0.0477	0.0482	0.0005	0.1118	0.1123	0.111	0.1117	0.0007	0.0513	0.05	0.0672	0.0562	0.0096
B208															
B210															
B212	0.04	0.042	0.041	0.0410	0.0010	0.062	0.064	0.056	0.0607	0.0042	0.028	0.027	0.027	0.0273	0.0006
B213	0.04	0.03	0.03	0.0333	0.0058	0.08	0.07	0.07	0.0733	0.0058	0.03	0.03	0.03	0.0300	0.0000
B215	0.023			0.0230		0.057			0.0570		0.026				0.0260
B216	<0.044	<0.044	<0.044			0.06	0.058	0.059	0.0590	0.0010	<0.044	<0.044	<0.044		
B217	0.03	0.03	0.03	0.0300	0.0000	0.07	0.08	0.07	0.0733	0.0058	0.03	0.03	0.03	0.0300	0.0000
B219	0.0324	0.0333	0.0326	0.0328	0.0005	0.0743	0.0779	0.0771	0.0764	0.0019	0.0333	0.032	0.0329	0.0327	0.0007
B220															
B221	0.033	0.033	0.034	0.0333	0.0006	0.075	0.08	0.084	0.0797	0.0045	0.034	0.035	0.033	0.0340	0.0010
B222	<0.04	<0.04	<0.04			<0.04	<0.04	<0.04			<0.05	<0.05	<0.05		
B223	0.0347	0.0342	0.0359	0.0349	0.0009	0.0556	0.059	0.0601	0.0582	0.0023	0.0258	0.0258	0.0265	0.0260	0.0004
B224	0.03	0.03	0.03	0.0300		0.07	0.07	0.07	0.0700		0.03	0.03	0.03	0.0300	
B226	<0.0152	<0.0149	<0.015			0.0375	0.04037		0.0389	0.0020	0.01543	0.01486	0.01505	0.0151	0.0003
B227	0.025	0.029	0.025	0.0263	0.0023	0.11	0.115	0.12	0.1150	0.0050	0.044	0.044	0.052	0.0467	0.0046
B228	0.051	0.05	0.05	0.0503	0.0006	0.078	0.073	0.068	0.0730	0.0050	0.039	0.039	0.038	0.0387	0.0006
B229	<0.10					<0.10					<0.10				
B230															
B233	0.023	0.018	0.022	0.0210	0.0026	0.039	0.042	0.041	0.0407	0.0015	0.021	0.021	0.014	0.0187	0.0040
B234	0.01	0.02	0.02			0.04	0.04	0.04	0.0400	0.0000	0.01	0.01	0.01	0.0100	0.0000

**Table B-2. Data summary table for  $\Delta^9$ -THC in three marijuana samples.**

Data in red font have been flagged as a data entry of zero or results that include text (e.g., "&lt; LOQ" or "present").

Data in blue font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

Target Consensus	Plant Sample 2					Plant Sample 3					Plant Sample 5					
				0.138 0.151	0.023 0.031				0.292 0.293	0.014 0.046				0.442 0.420	0.032 0.075	
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B045	detected	detected					detected	detected				detected	detected			
B046	0.15	0.16	0.15	0.1533	0.0058	0.27	0.26	0.27	0.2667	0.0058	0.43	0.43	0.43			
B055	0.1687	0.1697	0.1659	0.1681	0.0020	0.3257	0.3281	0.3185	0.3241	0.0050	0.4636	0.4605	0.4565	0.4300	0.0000	
B057	0.1433	0.149	0.1386	0.1436	0.0052	0.268	0.2697	0.265	0.2676	0.0024	0.3848	0.3801	0.3968	0.4602	0.0036	
B059	<1.5	<1.5	<1.5			<1.5	<1.5	<1.5			<1.5	<1.5	<1.5	0.3872	0.0086	
B062	0.152	0.156	0.148	0.1520	0.0040	0.314	0.31	0.311	0.3117	0.0021	0.466	0.463	0.445			
B071	0.11	0.1	0.1	0.1033	0.0058	0.24	0.23	0.23	0.2333	0.0058	0.28	0.28	0.28	0.4580	0.0114	
B099	0.128			0.1280		0.279			0.2790		0.379			0.2800	0.0000	
B101	<1	<1	<1			>1	>1	>1			>1	>1	>1	0.3790		
B109	0.135	0.139	0.131	0.1350	0.0040	0.268	0.267	0.27	0.2683	0.0015	0.392	0.385	0.375			
B117	0.095	0.105	0.101	0.1003	0.0050	0.188	0.19	0.191	0.1897	0.0015	0.255	0.277	0.266	0.3840	0.0085	
B125	0.139	0.136	0.139	0.1380	0.0017	0.276	0.271	0.261	0.2693	0.0076	0.396	0.384	0.401	0.2660	0.0110	
B133	0.22	0.23	0.25	0.2333	0.0153	0.39	0.39	0.37	0.3833	0.0115	0.51	0.53	0.52	0.3937	0.0087	
B135	0.11	0.11	0.11	0.1100		0.24	0.27	0.25	0.2533	0.0153	0.38	0.38	0.4	0.5200	0.0100	
B140	0.17	0.17	0.17	0.1700		0.28	0.3	0.32	0.3000	0.0200	0.57	0.54	0.53	0.3867	0.0115	
B146	0.144	0.144	0.143	0.1437	0.0006	0.281	0.278	0.278	0.2790	0.0017	0.406	0.399	0.413	0.5467	0.0208	
B151	0.213	0.216	0.212	0.2137	0.0021	0.382	0.383	0.381	0.3820	0.0010	0.509	0.525	0.534	0.4060	0.0070	
B154	0.138	0.15	0.127	0.1383	0.0115	0.285	0.284	0.284	0.2843	0.0006	0.388	0.386	0.398	0.5227	0.0127	
B158	0.15	0.15	0.15	0.1500	0.0000	0.3	0.3	0.29	0.2967	0.0058	0.44	0.43	0.42	0.3907	0.0064	
B167	0.16	0.16	0.16	0.1600	0.0000	0.29	0.3	0.29	0.2933	0.0058	0.36	0.37	0.36	0.4300	0.0100	
B169	0.16	0.16		0.1600		0.31	0.32		0.3150	0.0071	0.44	0.44		0.3633	0.0058	
B171	0.15	0.15		0.1500		0.31	0.31		0.3100		0.46	0.44		0.4400		
B178	0.163	0.161	0.157	0.1603	0.0031	0.302	0.308	0.297	0.3023	0.0055	0.43	0.425	0.423	0.4500	0.0141	
B198	0.19	0.19	0.19	0.1900		0.41	0.39	0.41	0.4033	0.0115	0.59	0.6	0.58	0.4260	0.0036	
B199	0.15	0.15	0.15	0.1500		0.3	0.3	0.31	0.3033	0.0058	0.45	0.46	0.45	0.5900	0.0100	
B207	0.217	0.2188	0.219	0.2183	0.0011	0.3845	0.391	0.3951	0.3902	0.0053	0.4615	0.4558	0.4626	0.4533	0.0058	
B213	0.18	0.17	0.18	0.1767	0.0058	0.32	0.34	0.32	0.3267	0.0115	0.46	0.44	0.45	0.4600	0.0037	
B222	0.13	0.12	0.13	0.1267	0.0058	0.27	0.21	0.24	0.2400	0.0300	0.34	0.35	0.34	0.4500	0.0100	
B229	0.12			0.1200		0.21			0.2100		0.28			0.3433	0.0058	

**Table B-3. Data summary table for THCA in three hemp samples.**

Data in **blue** font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .  
*Note: This table spans multiple pages; the NIST values and consensus values are included on all pages for convenience.*

Target Consensus	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6					
	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD
Lab	A	B	C			A	B	C			A	B	C		Avg	SD
B001	0.101	0.107	0.102	0.1033	0.0032	0.211	0.223	0.225	0.2197	0.0076	0.15	0.148	0.148	0.1487	0.0012	
B003	0.060315	0.05399	0.06104	0.0584	0.0039	0.13732	0.1413	0.14044	0.1397	0.0021	0.08775	0.08474	0.06847	0.0803	0.0104	
B004	0.059	0.059	0.059	0.0590	0.0000	0.202	0.202	0.202	0.2020	0.0000	0.073	0.073	0.073	0.0730	0.0000	
B005	0.113	0.115	0.1	0.1093	0.0081	0.227	0.233	0.2112	0.2237	0.0113	0.155	0.149	0.152	0.1520	0.0030	
B006	0.093	0.095	0.093	0.0937	0.0012	0.209	0.205	0.206	0.2067	0.0021	0.143	0.143	0.14	0.1420	0.0017	
B007	0.1	0.11	0.12	0.1100	0.0100	0.25	0.23	0.23	0.2367	0.0115	0.14	0.14	0.14	0.1400	0.0000	
B008																
B009																
B012	0.08491	0.0819		0.0834	0.0021	0.2107	0.2173		0.2140	0.0047	0.13038	0.1267		0.1285	0.0026	
B013	0.09	0.09	0.09	0.0900	0.0000	0.21	0.19	0.2	0.2000	0.0100	0.15	0.15	0.14	0.1467	0.0058	
B014																
B015	0.09	0.08	0.09	0.0867	0.0058	0.21	0.2	0.21	0.2067	0.0058	0.14	0.14	0.14	0.1400	0.0000	
B016	0.1651	0.1466	0.1416	0.1511	0.0124	0.072	0.0685	0.0613	0.0673	0.0055	0.2006	0.2028	0.1932	0.1989	0.0050	
B018	0.1	0.1	0.1	0.1000	0.0000	0.12	0.12	0.13	0.1233	0.0058	0.18	0.18	0.18	0.1800	0.0000	
B020																
B021																
B022	0.107	0.1	0.099	0.1020	0.0044	0.253	0.225	0.225	0.2343	0.0162	0.145	0.147	0.146	0.1460	0.0010	
B023	0.091	0.0932	0.0862	0.0901	0.0036	0.211	0.2142	0.2099	0.2117	0.0022	0.1308	0.1319	0.132	0.1316	0.0007	
B024	0.07	0.08	0.07	0.0733	0.0058	0.16	0.17	0.16	0.1633	0.0058	0.1	0.11	0.09	0.1000	0.0100	
B025	0.09	0.09	0.09	0.0900	0.0000	0.26	0.26	0.23	0.2500	0.0173	0.13	0.14	0.12	0.1300	0.0100	
B026	0.11	0.11	0.12	0.1133	0.0058	0.26	0.24	0.26	0.2533	0.0115	0.15	0.16	0.14	0.1500	0.0100	
B027	0.0912	0.0844	0.0888	0.0881	0.0034	0.2201	0.2243	0.2184	0.2209	0.0030	0.133	0.1282	0.1237	0.1283	0.0047	
B028																
B029	0.1	0.1	0.099	0.0997	0.0006	0.227	0.224	0.212	0.2210	0.0079	0.133	0.134	0.136	0.1343	0.0015	
B030	0.1	0.1	0.09	0.0967	0.0058	0.21	0.22	0.22	0.2167	0.0058	0.15	0.15	0.15	0.1500	0.0000	
B031																
B032	0.05	0.07	0.09	0.0700	0.0200	0.15	0.21	0.23	0.1967	0.0416	0.13	0.16	0.16	0.1500	0.0173	
B033	0.09108	0.09343	0.09344	0.0927	0.0014	0.20726	0.20985	0.20985	0.2090	0.0015	0.13768	0.14003	0.13756	0.1384	0.0014	
B035	0.11	0.1	0.11	0.1067	0.0058	0.25	0.26	0.26	0.2567	0.0058	0.16	0.15	0.16	0.1567	0.0058	
B036																
B037	0.099			0.0990		0.191	0.218	0.204	0.2043	0.0135	0.137	0.126	0.122	0.1283	0.0078	
B038																
B039	0.09			0.0900		0.201			0.2010		0.133			0.1330		
B041																
B042	0.09			0.0900		0.198			0.1980		0.136			0.1360		
B043	0.11	0.07	0.07	0.0833	0.0231	0.23	0.22	0.22	0.2233	0.0058	0.15	0.16	0.15	0.1533	0.0058	
B044	0.09	0.09	0.08	0.0867	0.0058	0.19	0.2	0.2	0.1967	0.0058	0.12	0.12	0.12	0.1200	0.0000	
B045	0.101	0.107	0.102	0.1033	0.0032	0.211	0.223	0.225	0.2197	0.0076	0.15	0.148	0.148	0.1487	0.0012	

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.0979	0.0084				0.2403	0.0096				0.126	0.022
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B046	0.060315	0.05399	0.06104	0.0584	0.0039	0.13732	0.1413	0.14044	0.1397	0.0021	0.08775	0.08474	0.06847	0.0803	0.0104
B047	0.052	0.033	0.041	0.0420	0.0095	0.063	0.07	0.063	0.0653	0.0040	0.034	0.033	0.03	0.03233	0.00208
B048															
B049	0.03036	0.03075	0.02884	0.0300	0.0010	0.06012	0.06818	0.07249	0.0669	0.0063	0.02016	0.03094	0.03246	0.02785	0.00671
B051	0.03	0.03	0.03	0.0300	0.0000	0.07	0.08	0.09	0.0800	0.0100	0.02	0.04	0.04	0.03333	0.01155
B052	< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08		
<b>B053</b>	<b>0.09</b>	<b>0.095</b>	<b>0.099</b>	<b>0.0947</b>	<b>0.0045</b>	<b>0.199</b>	<b>0.187</b>	<b>0.2</b>	<b>0.1953</b>	<b>0.0072</b>	<b>0.112</b>	<b>0.113</b>	<b>0.12</b>	<b>0.11500</b>	<b>0.00436</b>
B054	0.02461	0.02757	0.03283	0.0283	0.0042	0.06845	0.06553	0.06628	0.0668	0.0015	0.03167	0.03129	0.02603	0.02966	0.00315
B055	0.037	0.0336	0.0349	0.0352	0.0017	0.0761	0.0718	0.0722	0.0734	0.0024	0.0281	0.0292	0.033	0.03010	0.00257
B057	0.0212	0.0199	0.0191	0.0201	0.0011	0.0573	0.0559	0.0575	0.0569	0.0009	0.023	0.0241	0.0218	0.02297	0.00115
B058	0.0415	0.0421	0.0393	0.0410	0.0015	0.069	0.0681	0.0639	0.0670	0.0027	0.0344	< 0.0297	< 0.0296	0.03440	
B059	<1.5	<1.5	<1.5			<1.5	<1.5	<1.5			<1.5	<1.5	<1.5		
B060	0.06	0.06	0.06	0.0600	0.0000	0.08	0.08	0.1	0.0867	0.0115	0.04	0.04	0.03	0.03667	0.00577
B061	0.038	0.04	0.039	0.0390	0.0010	0.065	0.064	0.069	0.0660	0.0026	0.026	0.027	0.027	0.02667	0.00058
B062	< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08		
B063															
B064	0.033			0.0330		0.077			0.0770		0.027			0.02700	
B065	0.033	0.035	0.035	0.0343	0.0012	0.067	0.069	0.071	0.0690	0.0020	0.033	0.034	0.033	0.03333	0.00058
B066	<0.003	<0.003	<0.003			0.0759	0.0733	0.0675	0.0722	0.0043	0.034	0.0309	0.0361	0.03367	0.00262
B068															
<b>B069</b>	<b>0.05</b>	<b>0.05</b>	<b>0.05</b>	<b>0.0500</b>	<b>0.0000</b>	<b>0.12</b>	<b>0.08</b>	<b>&lt;0.01</b>	<b>0.1000</b>	<b>0.0283</b>	<b>0.04</b>	<b>0.04</b>	<b>&lt;0.01</b>	<b>0.04000</b>	<b>0.00000</b>
<b>B070</b>	<b>0.047</b>	<b>0.063</b>	<b>0.04</b>	<b>0.0500</b>	<b>0.0118</b>	<b>0.112</b>	<b>&lt;0.111</b>	<b>&lt;0.111</b>	<b>0.1120</b>		<b>&lt;0.067</b>	<b>&lt;0.067</b>	<b>&lt;0.067</b>		
B071	0.03	0.02	0.02	0.0233	0.0058	0.05	0.05	0.05	0.0500	0.0000	0.02	0.02	0.02	0.02000	0.00000
B072	0.036	0.033	0.034	0.0343	0.0015	0.07	0.075	0.073	0.0727	0.0025	0.035	0.035	0.035	0.03500	0.00000
B073	0.0241			0.0241		0.0543			0.0543		0.0231			0.02310	
<b>B074</b>	<b>0.04</b>	<b>0.04</b>	<b>0.04</b>	<b>0.0400</b>	<b>0.0000</b>	<b>0.06</b>	<b>0.06</b>	<b>0.06</b>	<b>0.0600</b>	<b>0.0000</b>	<b>0.07</b>	<b>0.07</b>	<b>0.07</b>	<b>0.07000</b>	<b>0.00000</b>
B076	0.05353	0.05127	0.05001	0.0516	0.0018	0.0635	0.06371	0.06392	0.0637	0.0002	0.02832	0.02817	0.02895	0.02848	0.00041
B077															
B078															
B079															
B081	<0.36	<0.36	<0.36			<0.36	<0.36	<0.36			<0.36	<0.36	<0.36		
B082	0.055	0.056	0.044	0.0517	0.0067	0.059	0.059	0.061	0.0597	0.0012	0.026	0.03	0.033	0.02967	0.00351
B084	0.04	0.03	0.035	0.0350	0.0050	0.07	0.07	0.07	0.0700	0.0000	0.03	0.03	0.03	0.03000	0.00000
<b>B085</b>	<b>0.051</b>	<b>0.051</b>	<b>0.043</b>	<b>0.0483</b>	<b>0.0046</b>	<b>0.098</b>	<b>0.108</b>	<b>0.094</b>	<b>0.1000</b>	<b>0.0072</b>	<b>0.041</b>	<b>0.033</b>	<b>0.035</b>	<b>0.03633</b>	<b>0.00416</b>
B086															
B087															
B088	0.041	0.036	0.039	0.0387	0.0025	0.061	0.064	0.072	0.0657	0.0057	0.028	0.03	0.024	0.02733	0.00306
B089	0.0307	0.0303	0.0291	0.0300	0.0008	0.043	0.0429	0.0481	0.0447	0.0030	0.032	0.0311	0.0263	0.02980	0.00306
B090	<0.05	<0.05	<0.05			0.06	0.06	0.07	0.0633	0.0058	<0.05	<0.05	<0.05		
B091	0.019	0.019	0.02	0.0193	0.0006	0.046	0.043	0.046	0.0450	0.0017	0.012	0.015	0.015	0.01400	0.00173
B092	0.01	0.01	0.01	0.0100	0.0000	0.05	0.06	0.05	0.0533	0.0058	0.01	0.02	0.02	0.01667	0.00577
B094	0.04	0.04	0.04	0.0400	0.0000	0.06	0.07	0.06	0.0633	0.0058	0.03	0.03	0.03	0.03000	0.00000
B095	0.048	0.049	0.048	0.0483	0.0006	0.067	0.064	0.068	0.0663	0.0021	0.037	0.037	0.037	0.03700	0.00000

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4				Plant Sample 6					
Target Consensus				0.0979 0.091	0.0084 0.020				0.2403 0.215	0.0096 0.030				0.126 0.137	0.022 0.021
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B096	0.034	0.039	0.033	0.0353	0.0032	0.054	0.059	0.059	0.0573	0.0029	0.028	0.027	0.027	0.02733	0.00058
B097	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
<b>B098</b>						<b>0.01</b>	<b>0.04</b>	<b>0.01</b>	<b>0.0200</b>	<b>0.0173</b>					
B099	0.034			0.0340		0.06			0.0600		0.027			0.02700	
B100	0.104	0.098		0.1010	0.0042	0.255	0.229		0.2420	0.0184	0.166	0.156		0.1610	0.0071
B102	0.0891	0.0835	0.0831	0.0852	0.0034	0.1973	0.2008	0.2054	0.2012	0.0041	0.1362	0.1364	0.134	0.1355	0.0013
B104	<0.25	<0.25	<0.25			<0.25	<0.25	<0.25			<0.25	<0.25	<0.25		
B106	0.09	0.09	0.089	0.0897	0.0006	0.205	0.186	0.187	0.1927	0.0107	0.132	0.132	0.131	0.1317	0.0006
B108															
B109	0.097	0.096	0.088	0.0937	0.0049	0.208	0.209	0.188	0.2017	0.0118	0.135	0.137	0.137	0.1363	0.0012
B110	0.075	0.076	0.074	0.0750	0.0010	0.21	0.2	0.2	0.2033	0.0058	0.12	0.11	0.11	0.1133	0.0058
B111															
B113	0.1	0.09	0.08	0.0900	0.0100	0.21	0.19	0.2	0.2000	0.0100	0.14	0.15	0.15	0.1467	0.0058
B114															
B115	0.094	0.1054	0.109	0.1028	0.0078	0.2765	0.2746	0.2859	0.2790	0.0061	0.1759	0.1793	0.1785	0.1779	0.0018
B116	0.0884	0.0875	0.0884	0.0881	0.0005	0.1997	0.1998	0.1878	0.1958	0.0069	0.1116	0.1123	0.1127	0.1122	0.0006
B117	0.089	0.098	0.105	0.0973	0.0080	0.233	0.243	0.241	0.2390	0.0053	0.164	0.161	0.181	0.1687	0.0108
B120															
B121															
B122															
B124	0.1065	0.0895	0.0965	0.0975	0.0085	0.228	0.236	0.2407	0.2349	0.0064	0.141	0.137	0.139	0.1390	0.0020
B125	0.084	0.084	0.082	0.0833	0.0012	0.199	0.196	0.194	0.1963	0.0025	0.124	0.124	0.121	0.1230	0.0017
B126	0.0728	0.0736	0.06751	0.0713	0.0033	0.2058	0.1875	0.2037	0.1990	0.0100	0.1304	0.1151	0.1102	0.1186	0.0105
B127	0.1113	0.0997	0.0952	0.1021	0.0083	0.2128	0.2305	0.228	0.2238	0.0096	0.1521	0.1585	0.1516	0.1541	0.0038
B129															
B130	0.09	0.09	0.09	0.0900		0.21	0.22	0.21	0.2133	0.0058	0.15	0.15	0.14	0.1467	0.0058
B131	0.0883	0.0855	0.0812	0.0850	0.0036	0.2186	0.2399	0.2181	0.2255	0.0124	0.1254	0.1266	0.126	0.1260	0.0006
<b>B132</b>	<b>0.171</b>	<b>0.154</b>	<b>0.157</b>	<b>0.1607</b>	<b>0.0091</b>	0.317	0.305	0.256	0.2927	0.0323	0.193	0.2	0.187	0.1933	0.0065
B133	0.11	0.11	0.11	0.1100		0.22	0.25	0.24	0.2367	0.0153	0.16	0.17	0.16	0.1633	0.0058
B135	<0.06	<0.06	<0.06			0.21	0.21	0.2	0.2067	0.0058	0.11	0.11	0.11	0.1100	
B136															
B137	0.11	0.094	0.111	0.1050	0.0095	0.251	0.209	0.294	0.2513	0.0425	0.151	0.147	0.156	0.1513	0.0045
B141	0.096	0.092	0.093	0.0937	0.0021	0.202	0.223	0.209	0.2113	0.0107	0.148	0.147	0.144	0.1463	0.0021
B142	0.091	0.085	0.086	0.0873	0.0032	0.267	0.263	0.247	0.2590	0.0106	0.154	0.149	0.145	0.1493	0.0045
<b>B144</b>	<b>0.15</b>	<b>0.18</b>	<b>0.14</b>	<b>0.1567</b>	<b>0.0208</b>	0.26	0.21	0.2	0.2233	0.0321	0.1	0.11	0.11	0.1067	0.0058
B146	0.0775	0.0752	0.0766	0.0764	0.0012	0.177	0.21	0.26	0.2157	0.0418	0.12	0.12	0.121	0.1203	0.0006
B147	0.077	0.085	0.077	0.0797	0.0046	0.198	0.193	0.195	0.1953	0.0025	0.138	0.125	0.113	0.1253	0.0125
B148															
B149	<0.0237	0.061	0.066	0.0635	0.0035	0.19	0.218	0.223	0.2103	0.0178	0.1314	0.135	0.161	0.1425	0.0162
B151	0.08	0.082	0.081	0.0810	0.0010	0.173	0.171	0.165	0.1697	0.0042	0.096	0.092	0.096	0.0947	0.0023
B152															
B153	0.1112	0.131	0.14	0.1274	0.0147	0.252	0.262	0.262	0.2587	0.0058	0.173	0.165	0.185	0.1743	0.0101
B154	0.079	0.086	0.077	0.0807	0.0047	0.176	0.192	0.219	0.1957	0.0217	0.132	0.14	0.139	0.1370	0.0044

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.0979	0.0084				0.2403	0.0096				0.126	0.022
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B157	0.08			0.0800		0.22			0.2200		0.13	0.13		0.1300	
B158	0.17	0.15	0.15	0.1567	0.0115	0.29	0.27	0.27	0.2767	0.0115	0.19	0.19	0.19	0.1900	0.0000
B159	0.0859	0.0845	0.0826	0.0843	0.0017	0.2049	0.2184	0.2231	0.2155	0.0094	0.1366	0.1391	0.1406	0.1388	0.0020
B160	0.10158	0.09739	0.09865	0.0992	0.0021	0.23388	0.216	0.22335	0.2244	0.0090	0.14758	0.1529	0.15636	0.1523	0.0044
B161	0.102	0.103	0.103	0.1027	0.0006	0.246	0.238	0.242	0.2420	0.0040	0.16	0.157	0.171	0.1627	0.0074
B163	0.11	0.0966	0.0957	0.1008	0.0080	0.216	0.201	0.161	0.1927	0.0284	0.152	0.181	0.151	0.1613	0.0170
B164	0.31	0.29	0.29	0.2967	0.0115	0.32	0.39	0.41	0.3733	0.0473	0.34	0.34	0.33	0.3367	0.0058
B165															
B166	<0.02	<0.02	<0.02			0.18	0.21	0.22	0.2033	0.0208	0.14	0.13	0.12	0.1300	0.0100
B167															
B168	0.0908	0.0885	0.087	0.0888	0.0019	0.228	0.215	0.204	0.2157	0.0120	0.151	0.15	0.15	0.1503	0.0006
B169	<0.15	<0.15				0.17	0.17		0.1700		<0.15	<0.15			
B170															
B172	0.07672	0.06952	0.07112	0.0725	0.0038	0.18624	0.17232	0.17368	0.1774	0.0077	0.1048	0.10872	0.10888	0.1075	0.0023
B173	0.0797	0.08213	0.0780	0.0800	0.0020	0.22394	0.2271	0.2370	0.2294	0.0068	0.1461	0.13209	0.13540	0.1379	0.0074
B174	0.09	0.09	0.09	0.0900		0.2	0.2	0.23	0.2100	0.0173	0.14	0.14	0.14	0.1400	
B175	0.11	0.12	0.12	0.1167	0.0058	0.19	0.17	0.18	0.1800	0.0100	0.11	0.11	0.11	0.1100	
B176	0.082	0.088	0.086	0.0853	0.0031	0.208	0.214	0.21	0.2107	0.0031	0.135	0.139	0.137	0.1370	0.0020
B178	0.107	0.102	0.102	0.1037	0.0029	0.232	0.241	0.229	0.2340	0.0062	0.162	0.156	0.157	0.1583	0.0032
B181	0.08			0.0800		0.21			0.2100		0.15			0.1500	
B182	0.07	0.07		0.0700		0.19	0.2	0.19	0.1933	0.0058	0.13	0.13	0.13	0.1300	
B183	0.0637	0.0648	0.0651	0.0645	0.0007	0.1279	0.133	0.1296	0.1302	0.0026	0.0816	0.0707	0.0727	0.0750	0.0058
B184	0.039	0.051	0.037	0.0423	0.0076	0.149	0.167	0.16	0.1587	0.0091	0.09	0.09	0.099	0.0930	0.0052
B185															
B186	0.088	0.087	0.085	0.0867	0.0015	0.186	0.197	0.214	0.1990	0.0141	0.135	0.135	0.132	0.1340	0.0017
B187	0.1241	0.1227	0.1199	0.1222	0.0021	0.255	0.2774	0.2883	0.2736	0.0170	0.1458	0.1501	0.1385	0.1448	0.0059
B188															
B189	0.11	0.1049	0.1072	0.1074	0.0026	0.2145	0.21	0.2069	0.2105	0.0038	0.1445	0.147	0.153	0.1482	0.0044
B190	0.09	0.09	0.09	0.0900		0.23	0.22	0.22	0.2233	0.0058	0.14	0.15	0.14	0.1433	0.0058
B192	0.09674	0.09758	0.09788	0.0974	0.0006	0.21322	0.2137	0.20606	0.2110	0.0043	0.13254	0.131	0.13079	0.1314	0.0010
B193	0.124	0.147	0.135	0.1353	0.0115	0.237	0.264	0.262	0.2543	0.0150	0.16	0.158	0.173	0.1637	0.0081
B194															
B195	0.083	0.085	0.084	0.0840	0.0010	0.204	0.206	0.211	0.2070	0.0036	0.127	0.128	0.128	0.1277	0.0006
B196															
B198	0.08	0.08	0.08	0.0800		0.21	0.2	0.22	0.2100	0.0100	0.12	0.13	0.12	0.1233	0.0058
B199	0.05	0.06	0.05	0.0533	0.0058	0.17	0.15	0.15	0.1567	0.0115	0.08	0.08	0.09	0.0833	0.0058
B200															
B202	0.0931	0.0964	0.0946	0.0947	0.0017	0.226	0.22	0.225	0.2237	0.0032	0.135	0.119	0.129	0.1277	0.0081
B204	0.084	0.085	0.084	0.0843	0.0006	0.228	0.202	0.216	0.2153	0.0130	0.14	0.138	0.141	0.1397	0.0015
B205	0.014	0.013	0.014	0.0137	0.0006	0.001	<0.001	<0.001	0.0010		<0.001	<0.001	<0.001		
B206	0.113	0.116	0.11	0.1130	0.0030	0.292	0.275	0.293	0.2867	0.0101	0.174	0.176	0.177	0.1757	0.0015
B207	0.0988	0.098	0.0976	0.0981	0.0006	0.2669	0.2586	0.2566	0.2607	0.0055	0.1522	0.1537	0.1496	0.1518	0.0021
B208															

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4				Plant Sample 6					
Target Consensus				0.0979 0.091	0.0084 0.020				0.2403 0.215	0.0096 0.030				0.126 0.137	0.022 0.021
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B210															
B212	0.087	0.082	0.085	0.0847	0.0025	0.191	0.188	0.183	0.1873	0.0040	0.122	0.116	0.109	0.1157	0.0065
B213	0.14	0.14	0.14	0.1400	0.0000	0.27	0.25	0.28	0.2667	0.0153	0.16	0.15	0.16	0.1567	0.0058
B215	0.81			0.8100		0.2			0.2000		0.12			0.1200	
B216	0.091	0.086	0.086	0.0877	0.0029	0.214	0.227	0.228	0.2230	0.0078	0.142	0.145	0.141	0.1427	0.0021
B217	0.09	0.09	0.09	0.0900	0.0000	0.23	0.2	0.22	0.2167	0.0153	0.16	0.15	0.16	0.1567	0.0058
B219	0.0933	0.0976	0.0959	0.0956	0.0022	0.226	0.24	0.237	0.2343	0.0074	0.145	0.139	0.142	0.1420	0.0030
B220															
B221	0.058	0.056	0.059	0.0577	0.0015	0.228	0.222	0.199	0.2163	0.0153	0.142	0.148	0.118	0.1360	0.0159
B222	0.09	0.08	0.09	0.0867	0.0058	0.08	0.13	0.09	0.1000	0.0265	0.12	0.12	0.12	0.1200	
B223	0.0871	0.0857	0.0892	0.0873	0.0018	0.203	0.213	0.216	0.2107	0.0068	0.135	0.138	0.135	0.1360	0.0017
B224	0.12	0.12	0.12	0.1200		0.26	0.26	0.26	0.2600		0.15	0.15	0.16	0.1533	0.0058
B226	0.0833	0.08221	0.0808	0.0821	0.0013	0.2176	0.21355		0.2156	0.0029	0.1443	0.13665	0.13661	0.1392	0.0044
B227	0.059	0.06	0.05	0.0563	0.0055	0.21	0.208	0.22	0.2127	0.0064	0.12	0.12	0.12	0.1200	
B228	0.108	0.105	0.105	0.1060	0.0017	0.251	0.229	0.214	0.2313	0.0186	0.145	0.147	0.147	0.1463	0.0012
B229	0.11			0.1100		0.19			0.1900		0.13			0.1300	
B230															
B233	0.051	0.051	0.037	0.0463	0.0081	0.1	0.13	0.125	0.1183	0.0161	0.124	0.11	0.095	0.1097	0.0145
B234	0.05	0.05	0.05	0.0500	0.0000	0.16	0.14	0.15	0.1500	0.0100	0.09	0.1	0.1	0.0967	0.0058

**Table B-4. Data summary table for THCA in three marijuana samples.**

Target Consensus	Plant Sample 2					Plant Sample 3					Plant Sample 5				
	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B046	0.37	0.39	0.36	0.3733	0.0153	0.95	0.98	0.98	0.970	0.017	1.77	1.77	1.79	1.777	0.012
B055	0.4409	0.4448	0.4563	0.4473	0.0080	1.272	1.3158	1.2683	1.285	0.026	2.115	2.1438	2.1175	2.125	0.016
B057	0.3364	0.349	0.3337	0.3397	0.0082	1.026	1.024	1.008	1.019	0.010	1.737	1.763	1.775	1.758	0.019
B062	0.393	0.401	0.4	0.3980	0.0044	1.187	1.184	1.155	1.175	0.018	1.979	1.971	1.922	1.957	0.031
B071	0.38	0.38	0.37	0.3767	0.0058	1.12	1.11	1.12	1.117	0.006	1.67	1.68	1.71	1.687	0.021
B099	0.317			0.3170		0.977			0.977		1.7			1.700	
B109	0.365	0.36	0.345	0.3567	0.0104	1.09	1.046	1.089	1.075	0.025	1.804	1.745	1.726	1.758	0.041
B117	0.414	0.475	0.44	0.4430	0.0306	1.203	1.273	1.259	1.245	0.037	1.9	2.149	2.057	2.035	0.126
B125	0.359	0.348	0.347	0.3513	0.0067	1.027	1.012	0.961	1.000	0.035	1.655	1.63	1.689	1.658	0.030
B133	0.39	0.43	0.43	0.4167	0.0231	1.06	1.12	1.06	1.080	0.035	1.87	1.85	1.77	1.830	0.053
B135	0.34	0.35	0.35	0.3467	0.0058	>1.01	1.01	>1.00	1.010		>1.02	>1.02	>1.02		
B146	0.357	0.393	0.359	0.3697	0.0202	1.07	1.06	1.07	1.067	0.006	1.77	1.78	1.77	1.773	0.006
B151	0.348	0.348	0.347	0.3477	0.0006	1.12	1.1	1.1	1.107	0.012	1.81	1.8	1.83	1.813	0.015
B154	0.329	0.359	0.273	0.3203	0.0437	1.054	1.056	1.034	1.048	0.012	1.657	1.639	1.679	1.658	0.020
B158	0.46	0.46	0.46	0.4600	0.0000	1.22	1.33	1.18	1.243	0.078	2.17	2.04	2.05	2.087	0.072
B167	0.41	0.43	0.42	0.4200	0.0100	1.15	1.18	1.19	1.173	0.021	1.84	1.87	1.79	1.833	0.040
B169	0.34	0.34		0.3400		1	1.1		1.050	0.071	1.8	1.8		1.800	
B178	0.424	0.422	0.409	0.4183	0.0081	1.27	1.25	1.19	1.237	0.042	2.08	1.99	1.85	1.973	0.116
B198	0.35	0.36	0.33	0.3467	0.0153	1.14	1.09	1.08	1.103	0.032	1.67	1.73	1.58	1.660	0.075
B199	0.33	0.34	0.33	0.3333	0.0058	0.97	0.99	1.01	0.990	0.020	1.54	1.58	1.57	1.563	0.021
B207	0.4237	0.4257	0.4275	0.4256	0.0019	>1.0	>1.0	>1.0	>1.0		>1.0	>1.0	>1.0		
B213	0.5	0.47	0.51	0.4933	0.0208	1.26	1.3	1.19	1.250	0.056	1.96	1.76	2.06	1.927	0.153
B222	0.32	0.3	0.32	0.3133	0.0115	1	0.79	0.9	0.897	0.105	1.46	1.45	1.44	1.450	0.010
B229	0.44			0.4400		1			1.000		>1.0				
B046	0.37	0.39	0.36	0.3733	0.0153	0.95	0.98	0.98	0.970	0.017	1.77	1.77	1.79	1.777	0.012
B055	0.4409	0.4448	0.4563	0.4473	0.0080	1.272	1.3158	1.2683	1.285	0.026	2.115	2.1438	2.1175	2.125	0.016
B057	0.3364	0.349	0.3337	0.3397	0.0082	1.026	1.024	1.008	1.019	0.010	1.737	1.763	1.775	1.758	0.019
B062	0.393	0.401	0.4	0.3980	0.0044	1.187	1.184	1.155	1.175	0.018	1.979	1.971	1.922	1.957	0.031
B071	0.38	0.38	0.37	0.3767	0.0058	1.12	1.11	1.12	1.117	0.006	1.67	1.68	1.71	1.687	0.021
B099	0.317			0.3170		0.977			0.977		1.7			1.700	
B109	0.365	0.36	0.345	0.3567	0.0104	1.09	1.046	1.089	1.075	0.025	1.804	1.745	1.726	1.758	0.041
B117	0.414	0.475	0.44	0.4430	0.0306	1.203	1.273	1.259	1.245	0.037	1.9	2.149	2.057	2.035	0.126
B125	0.359	0.348	0.347	0.3513	0.0067	1.027	1.012	0.961	1.000	0.035	1.655	1.63	1.689	1.658	0.030
B133	0.39	0.43	0.43	0.4167	0.0231	1.06	1.12	1.06	1.080	0.035	1.87	1.85	1.77	1.830	0.053
B135	0.34	0.35	0.35	0.3467	0.0058	>1.01	1.01	>1.00	1.010		>1.02	>1.02	>1.02		
B146	0.357	0.393	0.359	0.3697	0.0202	1.07	1.06	1.07	1.067	0.006	1.77	1.78	1.77	1.773	0.006
B151	0.348	0.348	0.347	0.3477	0.0006	1.12	1.1	1.1	1.107	0.012	1.81	1.8	1.83	1.813	0.015

**Table B-5. Data summary table for total  $\Delta^9$ -THC in three hemp samples.**

Data in blue font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

Note: This table spans multiple pages; the NIST values and consensus values are included on all pages for convenience.

Target Consensus	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6					
	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD
Lab	A	B	C		A	B	C		A	B	C		A	B	C	
B001	0.161	0.169	0.164	0.1647	0.0040	0.25	0.266	0.269	0.2617	0.0102	0.166	0.163	0.164	0.1643	0.0015	
B002	0.084	0.083	0.084	0.0837	0.0006	0.192	0.182	0.196	0.1900	0.0072	0.11	0.106	0.107	0.1077	0.0021	
B003	0.0529	0.04735	0.05353	0.0513	0.0034	0.2036	0.20669	0.19447	0.2016	0.0064	0.11619	0.10852	0.08654	0.1037	0.0154	
B004	0.039	0.039	0.039	0.0390	0.0000	0.267	0.267	0.267	0.2670	0.0000	0.091	0.091	0.091	0.0910	0.0000	
B005	0.099	0.101	0.087	0.0957	0.0076	0.199	0.204	0.186	0.1963	0.0093	0.136	0.13	0.134	0.1333	0.0031	
B006	0.115	0.116	0.114	0.1150	0.0010	0.263	0.258	0.258	0.2597	0.0029	0.168	0.168	0.165	0.1670	0.0017	
B007	0.088	0.096	0.105	0.0963	0.0085	0.21	0.2	0.2	0.2033	0.0058	0.123	0.123	0.123	0.1230	0.0000	
B008																
B009																
B010	0.19	0.23		0.2100	0.0283	0.31	0.35		0.3300	0.0283	0.2	0.25		0.2250	0.0354	
B011	0.154	0.159	0.17	0.1610	0.0082	0.311	0.317	0.294	0.3073	0.0119	0.191	0.2	0.194	0.1950	0.0046	
B012	0.11465	0.1134		0.1140	0.0009	0.2433	0.2514		0.2474	0.0057	0.1406	0.1369		0.1388	0.0026	
B013	0.11	0.1	0.11	0.1067	0.0058	0.24	0.23	0.23	0.2333	0.0058	0.16	0.16	0.15	0.1567	0.0058	
B014																
B015	0.09	0.09	0.09	0.0900	0.0000	0.05	0.05	0.05	0.0500	0.0000	0.14	0.14	0.15	0.1433	0.0058	
B016	0.2458	0.2379	0.2231	0.2356	0.0115	0.162	0.16	0.1519	0.1580	0.0053	0.256	0.2523	0.2433	0.2505	0.0065	
B018	0.09	0.08	0.08	0.0833	0.0058	0.22	0.21	0.22	0.2167	0.0058	0.15	0.16	0.16	0.1567	0.0058	
B019	0.08	0.084	0.085	0.0830	0.0026	0.195	0.199	0.188	0.1940	0.0056	0.116	0.117	0.117	0.1167	0.0006	
B020																
B021																
B022	0.185	0.178	0.176	0.1797	0.0047	0.301	0.273	0.273	0.2823	0.0162	0.165	0.165	0.168	0.1660	0.0017	
B023	0.1093	0.1091	0.1017	0.1067	0.0043	0.2504	0.2542	0.2486	0.2511	0.0029	0.1451	0.1456	0.1453	0.1453	0.0003	
B024	0.09	0.1	0.08	0.0900	0.0100	0.18	0.2	0.17	0.1833	0.0153	0.1	0.11	0.08	0.0967	0.0153	
B025	0.12	0.15	0.13	0.1333	0.0153	0.33	0.35	0.28	0.3200	0.0361	0.17	0.18	0.15	0.1667	0.0153	
B026	0.12	0.14	0.12	0.1267	0.0115	0.29	0.25	0.28	0.2733	0.0208	0.14	0.15	0.13	0.1400	0.0100	
B027	0.08	0.074	0.0779	0.0773	0.0030	0.251	0.256	0.248	0.2517	0.0040	0.117	0.112	0.108	0.1123	0.0045	
B028																
B029	0.088	0.088	0.086	0.0873	0.0012	0.274	0.271	0.258	0.2677	0.0085	0.116	0.117	0.119	0.1173	0.0015	
B030	0.09	0.09	0.08	0.0867	0.0058	0.28	0.3	0.3	0.2933	0.0115	0.19	0.19	0.19	0.1900	0.0000	
B031																
B033	0.04	0.06	0.08	0.0600	0.0200	0.19	0.24	0.27	0.2333	0.0404	0.12	0.14	0.14	0.1333	0.0115	
B034	0.038	0.06	0.071	0.0563	0.0168	0.12	0.186	0.14	0.1487	0.0338	0.071	0.063	0.071	0.0683	0.0046	
B035	0.12237	0.12609	0.12473	0.1244	0.0019	0.24649	0.24709	0.24541	0.2463	0.0009	0.14789	0.15469	0.15115	0.1512	0.0034	
B036	0.14	0.14	0.15	0.1433	0.0058	0.29	0.31	0.3	0.3000	0.0100	0.18	0.16	0.17	0.1700	0.0100	
B037																
B039																
B041	0.079	<1	<1	0.0790		0.245	<1	<1	0.2450		0.117	<1	<1		0.1170	
B042	<1															

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4				Plant Sample 6					
Target Consensus				0.118	0.014				0.279	0.012				0.139	0.013
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B043	0.122			0.1220		0.247			0.2470		0.12			0.1200	
B044	0.1	0.06	0.06	0.0733	0.0231	0.2	0.19	0.19	0.1933	0.0058	0.13	0.14	0.13	0.1333	0.0058
B046	0.1	0.11	0.1	0.1033	0.0058	0.23	0.24	0.23	0.2333	0.0058	0.13	0.14	0.13	0.1333	0.0058
B047	0.138	0.12	0.124	0.1273	0.0095	0.256	0.261	0.245	0.2540	0.0082	0.156	0.16	0.158	0.1580	0.0020
B048															
B049	0.09926	0.1011	0.09741	0.0993	0.0018	0.22807	0.25331	0.26949	0.2503	0.0209	0.09228	0.15254	0.15256	0.1325	0.0348
B050	0.08	0.08	0.07	0.0767	0.0058	0.2	0.19	0.2	0.1967	0.0058	0.11	0.11	0.11	0.1100	0.0000
B051	0.11	0.11	0.1	0.1067	0.0058	0.26	0.25	0.24	0.2500	0.0100	0.14	0.14	0.13	0.1367	0.0058
B052	0.07	0.07	0.07	0.0700	0.0000	< 0.08	< 0.08	< 0.08	0.2500	0.0100	0.1	0.09	0.09	0.0933	0.0058
B053	0.329	0.342	0.352	0.3410	0.0115	0.616	0.615	0.654	0.6283	0.0222	0.457	0.479	0.497	0.4777	0.0200
B054	0.09605	0.09418	0.09806	0.0961	0.0019	0.2554	0.25017	0.23741	0.2477	0.0093	0.1422	0.14195	0.1354	0.1398	0.0039
B055	0.1309	0.1288	0.1307	0.1301	0.0012	0.3019	0.2862	0.3015	0.2965	0.0090	0.1653	0.1573	0.1676	0.1634	0.0054
B056	0.135	0.13		0.1325	0.0035	0.258	0.257		0.2575	0.0007	0.143	0.15		0.1465	0.0049
B057	0.099	0.094	0.0912	0.0947	0.0040	0.2159	0.2143	0.2156	0.2153	0.0009	0.1317	0.1324	0.1217	0.1286	0.0060
B058	0.129	0.132	0.124	0.1283	0.0040	0.268	0.266	0.253	0.2623	0.0081	0.162	0.119	0.116	0.1323	0.0257
B059															
B060	0.14	0.17	0.11	0.1400	0.0300	0.27	0.26	0.23	0.2533	0.0208	0.18	0.17	0.19	0.1800	0.0100
B061	0.116	0.122	0.122	0.1200	0.0035	0.253	0.249	0.262	0.2547	0.0067	0.146	0.149	0.147	0.1473	0.0015
B062	0.079	0.081	0.083	0.0810	0.0020	0.206	0.197	0.195	0.1993	0.0059	0.117	0.123	0.12	0.1200	0.0030
B063															
B064	0.104			0.1040		0.264			0.2640		0.143			0.1430	
B065	0.11	0.12	0.12	0.1167	0.0058	0.25	0.26	0.27	0.2600	0.0100	0.16	0.16	0.15	0.1567	0.0058
B066	0.0865	0.0872	0.088	0.0872	0.0008	0.2911	0.2791	0.2718	0.2807	0.0097	0.1661	0.1638	0.169	0.1663	0.0026
B067	<1	<1	<1			<1	<1	<1			<1	<1	<1		
B068															
B069	0.146	0.146	0.143	0.1450	0.0017	0.329	0.28	0.191	0.2667	0.0700	0.179	0.188	0.131	0.1660	0.0306
B070	0.125	0.158	0.123	0.1353	0.0197	0.328	0.193	0.183	0.2347	0.0810	0.143	0.11	0.148	0.1337	0.0206
B071	0.14	0.14	0.14	0.1400	0.0000	0.23	0.23	0.23	0.2300	0.0000	0.14	0.14	0.14	0.1400	0.0000
B073	0.1655			0.1655		0.2382			0.2382		0.1583			0.1583	
B074	0.09	0.09	0.09	0.0900	0.0000	0.3	0.3	0.3	0.3000	0.0000	0.23	0.23	0.23	0.2300	0.0000
B075	0.12	0.13	0.13	0.1267	0.0058	0.25	0.19	0.24	0.2267	0.0321	0.15	0.14	0.14	0.1433	0.0058
B076	0.137	0.131	0.13	0.1327	0.0038	0.252	0.26	0.254	0.2553	0.0042	0.139	0.139	0.14	0.1393	0.0006
B077	0.13	0.119	0.117	0.1220	0.0070	0.218	0.243	0.225	0.2287	0.0129	0.16	0.152	0.158	0.1567	0.0042
B078															
B079															
B081	<0.36	<0.36	<0.36			<0.36	<0.36	<0.36			<0.36	<0.36	<0.36		
B082	0.144	0.143	0.132	0.1397	0.0067	0.253	0.271	0.256	0.2600	0.0096	0.146	0.144	0.152	0.1473	0.0042
B083	<1	<1				<1	<1				<1	<1			
B084	0.04	0.03	0.035	0.0350	0.0050	0.2452	0.2366	0.241	0.2409	0.0043	0.144	0.144	0.144	0.1440	0.0000
B085	0.19	0.182	0.158	0.1767	0.0167	0.382	0.448	0.37	0.4000	0.0420	0.197	0.179	0.178	0.1847	0.0107
B086	0.163	0.157	0.156	0.1587	0.0038	0.226	0.218	0.227	0.2237	0.0049	0.148	0.147	0.144	0.1463	0.0021
B088	0.09	0.088	0.093	0.0903	0.0025	0.232	0.239	0.248	0.2397	0.0080	0.141	0.148	0.132	0.1403	0.0080
B089	0.0902	0.0945	0.0895	0.0914	0.0027	0.1593	0.1573	0.1585	0.1584	0.0010	0.1272	0.1258	0.1207	0.1246	0.0034

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.118	0.014				0.279	0.012				0.139	0.013
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B090	0.07	0.08	0.08	0.0767	0.0058	0.25	0.25	0.25	0.2500	0.0000	0.13	0.12	0.12	0.1233	0.0058
B091	0.094	0.092	0.095	0.0937	0.0015	0.22	0.206	0.219	0.2150	0.0078	0.116	0.123	0.119	0.1193	0.0035
B092	0.09	0.09	0.08	0.0867	0.0058	0.28	0.24	0.24	0.2533	0.0231	0.16	0.16	0.15	0.1567	0.0058
B093	<1				<1						<1				
B094	0.1	0.1	0.11	0.1033	0.0058	0.23	0.24	0.23	0.2333	0.0058	0.14	0.13	0.13	0.1333	0.0058
B095	0.124	0.127	0.125	0.1253	0.0015	0.24	0.229	0.244	0.2377	0.0078	0.159	0.161	0.16	0.1600	0.0010
B096	0.117	0.12	0.113	0.1167	0.0035	0.239	0.257	0.255	0.2503	0.0099	0.263	0.159	0.158	0.1933	0.0603
<b>B098</b>							<b>0.01</b>		<b>0.0100</b>		<b>0.03</b>	<b>0.03</b>	<b>0.07</b>	<b>0.0433</b>	<b>0.0231</b>
B099	0.116			0.1160		0.216			0.2160		0.116			0.1160	
B100	0.091	0.086		0.0885	0.0035	0.294	0.271		0.2825	0.0163	0.146	0.137		0.1415	0.0064
B102	0.1092	0.1013	0.1008	0.1038	0.0047	0.2322	0.2355	0.2409	0.2362	0.0044	0.1498	0.1489	0.1465	0.1484	0.0017
B104	<0.25	<0.25	<0.25			<0.25	<0.25	<0.25			<0.25	<0.25	<0.25		
B106	0.113	0.111	0.111	0.1117	0.0012	0.246	0.224	0.226	0.2320	0.0122	0.142	0.145	0.141	0.1427	0.0021
B108															
B109	0.115	0.111	0.104	0.1100	0.0056	0.246	0.248	0.221	0.2383	0.0150	0.149	0.149	0.15	0.1493	0.0006
B110	0.12	0.12	0.12	0.1200		0.25	0.24	0.24	0.2433	0.0058	0.14	0.14	0.13	0.1367	0.0058
B111															
B113	0.16	0.15	0.14	0.1500	0.0100	0.26	0.22	0.24	0.2400	0.0200	0.16	0.17	0.16	0.1633	0.0058
B114															
B115	0.0998	0.109	0.1145	0.1078	0.0074	0.2968	0.2901	0.3028	0.2966	0.0064	0.1703	0.175	0.173	0.1728	0.0024
B116	0.1085	0.1133	0.1088	0.1102	0.0027	0.2354	0.2365	0.2242	0.2320	0.0068	0.1254	0.1246	0.125	0.1250	0.0004
B117	0.098	0.107	0.115	0.1067	0.0085	0.241	0.255	0.247	0.2477	0.0070	0.164	0.156	0.176	0.1653	0.0101
B118	0.1	0.1	<0.10	0.1000		0.25	0.25	0.24	0.2467	0.0058	0.15	0.15	0.15	0.1500	
B120															
B121	<2					<2					<2				
B122															
B123	0.11	0.09	0.15	0.1167	0.0306	0.27	0.26	0.26	0.2633	0.0058	0.17	0.2	0.18	0.1833	0.0153
B124	0.1339	0.1229	0.1216	0.1261	0.0068	0.262	0.272	0.277	0.2703	0.0076	0.1557	0.1521	0.1559	0.1546	0.0021
B125	0.108	0.109	0.108	0.1083	0.0006	0.235	0.232	0.23	0.2323	0.0025	0.136	0.136	0.132	0.1347	0.0023
B126	0.1451	0.1454	0.1382	0.1429	0.0041	0.1804	0.1644	0.1786	0.1745	0.0088	0.1143	0.1009	0.0967	0.1040	0.0092
B127	0.1527	0.1419	0.1372	0.1439	0.0079	0.2524	0.2692	0.2712	0.2643	0.0103	0.1685	0.1747	0.1694	0.1709	0.0034
B128	<1	<1	<1			<1	<1	<1			<1	<1	<1		
B129															
B130	0.11	0.12	0.11	0.1133	0.0058	0.25	0.26	0.25	0.2533	0.0058	0.16	0.16	0.15	0.1567	0.0058
B131	0.115	0.1125	0.1076	0.1117	0.0038	0.2913	0.3136	0.2893	0.2981	0.0135	0.1504	0.1523	0.1525	0.1517	0.0012
B132	0.193	0.176	0.179	0.1827	0.0091	0.367	0.354	0.295	0.3387	0.0384	0.207	0.213	0.201	0.2070	0.0060
B133	0.094	0.093	0.093	0.0933	0.0006	0.266	0.287	0.306	0.2863	0.0200	0.14	0.15	0.14	0.1433	0.0058
B134	<1	<1	<1			<1	<1	<1			<1	<1	<1		
B135	0.02	0.03	0.02	0.0233	0.0058	0.23	0.23	0.23	0.2300		0.11	0.1	0.09	0.1000	0.0100
B136															
B137	0.144	0.123	0.136	0.1343	0.0106	0.301	0.256	0.34	0.2990	0.0420	0.171	0.168	0.179	0.1727	0.0057
<b>B138</b>	<b>0.49</b>	<b>0.5</b>	<b>0.49</b>	<b>0.4933</b>	<b>0.0058</b>	0.25	0.24	0.26	0.2500	0.0100	0.2	0.2	0.2	0.2000	
B141	0.124	0.121	0.114	0.1197	0.0051	0.238	0.262	0.247	0.2490	0.0121	0.163	0.16	0.16	0.1610	0.0017

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.118	0.014				0.279	0.012				0.139	0.013
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B142	0.12	0.116	0.115	0.1170	0.0026	0.318	0.292	0.278	0.2960	0.0203	0.174	0.169	0.163	0.1687	0.0055
B143	0.092	0.094	0.091	0.0923	0.0015	0.239	0.237	0.231	0.2357	0.0042	0.121	0.124	0.125	0.1233	0.0021
B144	0.17	0.2	0.16	0.1767	0.0208	0.3	0.26	0.26	0.2733	0.0231	0.13	0.015	0.14	0.0950	0.0695
B145															
B146	0.0954	0.0923	0.0962	0.0946	0.0021	0.213	0.304	0.291	0.2693	0.0492	0.132	0.13	0.13	0.1307	0.0012
B147	0.098	0.109	0.097	0.1013	0.0067	0.235	0.235	0.244	0.2380	0.0052	0.153	0.14	0.134	0.1423	0.0097
B148															
B149	0.04041	0.08	0.07	0.0635	0.0206	0.23	0.25	0.25	0.2433	0.0115	0.148	0.135	0.156	0.1463	0.0106
B151	0.129	0.123	0.123	0.1250	0.0035	0.236	0.23	0.224	0.2300	0.0060	0.125	0.126	0.126	0.1257	0.0006
B152															
B153	0.133	0.131	0.14	0.1347	0.0047	0.303	0.306	0.317	0.3087	0.0074	0.19	0.184	0.2	0.1913	0.0081
B154	0.094	0.097	0.09	0.0937	0.0035	0.209	0.226	0.264	0.2330	0.0282	0.14	0.155	0.151	0.1487	0.0078
B157	0.07			0.0700		0.25			0.2500		0.11	0.11		0.1100	
B158	0.18	0.17	0.17	0.1733	0.0058	0.31	0.31	0.3	0.3067	0.0058	0.19	0.19	0.2	0.1933	0.0058
B159	0.1278	0.1297	0.1237	0.1271	0.0031	0.263	0.2675	0.2719	0.2675	0.0045	0.162	0.1633	0.1658	0.1637	0.0019
B160	0.13826	0.12839	0.13239	0.1330	0.0050	0.30062	0.28222	0.28993	0.2909	0.0092	0.17837	0.18469	0.18955	0.1842	0.0056
B161	0.137	0.137	0.136	0.1367	0.0006	0.289	0.278	0.288	0.2850	0.0061	0.173	0.172	0.184	0.1763	0.0067
B162															
B163	0.149	0.131	0.13	0.1367	0.0107	0.283	0.273	0.217	0.2577	0.0356	0.185	0.212	0.181	0.1927	0.0169
<b>B164</b>	<b>0.4</b>	<b>0.37</b>	<b>0.36</b>	<b>0.3767</b>	<b>0.0208</b>	<b>0.36</b>	<b>0.47</b>	<b>0.52</b>	<b>0.4500</b>	<b>0.0819</b>	<b>0.4</b>	<b>0.37</b>	<b>0.39</b>	<b>0.3867</b>	<b>0.0153</b>
B165															
B166	0.03	0.03	0.03	0.0300		0.24	0.27	0.28	0.2633	0.0208	0.17	0.16	0.15	0.1600	0.0100
B167															
B168	0.111	0.109	0.108	0.1093	0.0015	0.261	0.248	0.237	0.2487	0.0120	0.163	0.163	0.163	0.1630	
B169	<0.15	<0.15				0.15	0.15		0.1500		<0.15	<0.15			
B170															
B172	0.16664	0.12665	0.11117	0.1348	0.0286	0.29141	0.2996	0.26688	0.2860	0.0170	0.09191	0.09535	0.18869	0.1253	0.0549
B173	0.08506	0.08619	0.08281	0.0847	0.0017	0.23656	0.24137	0.25097	0.2430	0.0073	0.14386	0.13094	0.13404	0.1363	0.0067
B174	0.12	0.12	0.12	0.1200		0.17	0.24	0.27	0.2267	0.0513					
B175	0.1	0.1	0.1	0.1000		0.27	0.24	0.24	0.2500	0.0173	0.1	0.1	0.1	0.1000	
B176	0.072	0.125	0.097	0.0980	0.0265	0.269	0.276	0.271	0.2720	0.0036	0.162	0.168	0.166	0.1653	0.0031
B177	0.183	0.184	0.186	0.1843	0.0015	0.238	0.227	0.239	0.2347	0.0067	0.157	0.164	0.168	0.1630	0.0056
B178	0.12	0.116	0.11	0.1153	0.0050	0.271	0.282	0.268	0.2737	0.0074	0.177	0.17	0.171	0.1727	0.0038
B181	0.07			0.0700		0.27			0.2700		0.18			0.1800	
B182	0.06	0.06		0.0600		0.25	0.25	0.25	0.2500		0.11	0.11	0.011	0.0770	0.0572
B183	0.091	0.0956	0.0936	0.0934	0.0023	0.1752	0.181	0.1756	0.1773	0.0032	0.1207	0.0926	0.1067	0.1067	0.0141
B184	0.034	0.045	0.033	0.0373	0.0067	0.198	0.217	0.212	0.2090	0.0098	0.079	0.079	0.087	0.0817	0.0046
B185															
B186	0.147	0.145	0.134	0.1420	0.0070	0.225	0.238	0.255	0.2393	0.0150	0.149	0.149	0.147	0.1483	0.0012
B187	0.1509	0.1472	0.1476	0.1486	0.0020	0.2945	0.3143	0.3321	0.3136	0.0188	0.1616	0.169	0.1536	0.1614	0.0077
B188															
B189	0.0964	0.092	0.0941	0.0942	0.0022	0.1881	0.1842	0.1814	0.1846	0.0034	0.1267	0.1289	0.1342	0.1299	0.0039
B190	0.14	0.14	0.14	0.1400		0.27	0.26	0.26	0.2633	0.0058	0.16	0.17	0.16	0.1633	0.0058

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.118	0.014				0.279	0.012				0.139	0.013
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B191															
B192	0.11708	0.11755	0.1181	0.1176	0.0005	0.25164	0.25259	0.24789	0.2507	0.0025	0.14725	0.1464	0.14548	0.1464	0.0009
B193	0.109	0.129	0.118	0.1187	0.0100	0.208	0.231	0.229	0.2227	0.0127	0.14	0.138	0.151	0.1430	0.0070
B194															
B195	0.125	0.127	0.128	0.1267	0.0015	0.245	0.246	0.252	0.2477	0.0038	0.137	0.138	0.138	0.1377	0.0006
B196	<1					<1					<1				
B197	0.66	0.66	0.67	0.6633	0.0058	0.59	0.67	0.6	0.6200	0.0436	0.65	0.64	0.63	0.6400	0.0100
B198	0.21	0.2	0.2	0.2033	0.0058	0.26	0.25	0.28	0.2633	0.0153	0.15	0.15	0.15	0.1500	
B199	0.11	0.11	0.11	0.1100		0.22	0.2	0.2	0.2067	0.0115	0.1	0.11	0.11	0.1067	0.0058
B200															
B201	0.17	0.16	0.16	0.1633	0.0058	0.27	0.27	0.26	0.2667	0.0058	0.15	0.15	0.15	0.1500	
B202	0.115	0.113	0.111	0.1130	0.0020	0.265	0.258	0.265	0.2627	0.0040	0.144	0.13	0.139	0.1377	0.0071
B204	0.105	0.105	0.107	0.1057	0.0012	0.267	0.238	0.254	0.2530	0.0145	0.153	0.151	0.156	0.1533	0.0025
B206	0.134	0.135	0.131	0.1333	0.0021	0.312	0.294	0.32	0.3087	0.0133	0.18	0.181	0.18	0.1803	0.0006
B207	0.1353	0.134	0.1332	0.1342	0.0011	0.3458	0.339	0.336	0.3403	0.0050	0.1847	0.1847	0.1983	0.1892	0.0079
B208															
B210															
B211															
B212	0.116	0.114	0.116	0.1153	0.0012	0.229	0.229	0.217	0.2250	0.0069	0.135	0.129	0.123	0.1290	0.0060
B213	0.16	0.15	0.15	0.1533	0.0058	0.31	0.29	0.31	0.3033	0.0115	0.17	0.16	0.17	0.1667	0.0058
B215	0.094			0.0940		0.23			0.2300		0.13			0.1300	
B216	0.08	0.075	0.076	0.0770	0.0026	0.248	0.256	0.258	0.2540	0.0053	0.125	0.127	0.123	0.1250	0.0020
B217	0.11	0.11	0.11	0.1100		0.27	0.25	0.26	0.2600	0.0100	0.17	0.16	0.16	0.1633	0.0058
B218	<1	<1	<1			<1	<1	<1			<1	<1	<1		
B219	0.114	0.119	0.117	0.1167	0.0025	0.273	0.289	0.285	0.2823	0.0083	0.161	0.154	0.157	0.1573	0.0035
B220															
B221															
B222	0.08	0.07	0.08	0.0767	0.0058	0.07	0.12	0.08	0.0900	0.0265	0.11	0.11	0.11	0.1100	
B223	0.111	0.109	0.114	0.1113	0.0025	0.233	0.246	0.25	0.2430	0.0089	0.145	0.147	0.145	0.1457	0.0012
B224	0.14	0.14	0.14	0.1400		0.3	0.3	0.3	0.3000		0.16	0.16	0.17	0.1633	0.0058
B226	0.08658	0.08369	0.08476	0.0850	0.0015	0.22841	0.22766		0.2280	0.0005	0.14201	0.13415	0.13486	0.1370	0.0043
B227	0.08	0.08	0.07	0.0767	0.0058	0.29	0.3	0.32	0.3033	0.0153	0.15	0.15	0.16	0.1533	0.0058
B228	0.146	0.142	0.142	0.1433	0.0023	0.298	0.274	0.256	0.2760	0.0211	0.166	0.168	0.167	0.1670	0.0010
B229	<0.10					0.16			0.1600		0.11			0.1100	
B230															
B233	0.074	0.07	0.059	0.0677	0.0078	0.139	0.172	0.166	0.1590	0.0176	0.145	0.131	0.109	0.1283	0.0181
B234	0.06	0.07	0.06	0.0633	0.0058	0.18	0.18	0.1	0.1533	0.0462	0.1	0.11	0.11	0.1067	0.0058

**Table B-6. Data summary table for total  $\Delta^9$ -THC in three marijuana samples.**

Target Consensus	Plant Sample 2					Plant Sample 3					Plant Sample 5				
	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B002	0.386	0.388	0.382	0.385	0.003	0.993	0.984	1.001	0.993	0.009	1.518	1.375	1.526	1.473	0.085
B019	0.425	0.433	0.437	0.432	0.006	1.146	1.153	1.179	1.159	0.017	1.754	1.719	1.748	1.740	0.019
B034	0.475	0.442	0.428	0.448	0.024	1.41	1.15	1.36	1.307	0.138	2.07	1.86	1.93	1.953	0.107
B042	<1	<1	<1	>1	>1	>1	>1	>1	>1	>1	>1	>1	>1	1.94	0.22
B046	0.47	0.5	0.46	0.477	0.021	1.1	1.13	1.13	1.120	0.017	1.98	1.99	2	1.990	0.010
B055	0.5554	0.5598	0.5661	0.560	0.005	1.4412	1.4821	1.4308	1.451	0.027	2.3185	2.3406	2.3135	2.324	0.014
B057	0.4383	0.4551	0.4313	0.442	0.012	1.168	1.168	1.149	1.162	0.011	1.908	1.926	1.954	1.929	0.023
B062	0.497	0.507	0.499	0.501	0.005	1.355	1.348	1.323	1.342	0.017	1.736	1.728	1.686	1.717	0.027
B067	<1	<1	<1	>1	>1	>1	>1	>1	>1	>1	>1	>1	>1	1.94	0.22
B071	0.44	0.44	0.42	0.433	0.012	1.22	1.21	1.2	1.210	0.010	1.75	1.76	1.78	1.763	0.015
B075	0.48	0.5	0.47	0.483	0.015	>1	>1	>1	>1	>1	>1	>1	>1	1.94	0.22
B086	0.472	0.471	0.469	0.471	0.002	1.26	1.202	1.227	1.230	0.029	1.95	1.968	1.985	1.968	0.018
B099	0.406			0.406		1.14			1.140		1.87			1.870	
B109	0.455	0.454	0.433	0.447	0.012	1.223	1.184	1.226	1.211	0.023	1.974	1.915	1.889	1.926	0.044
B117	0.458	0.522	0.486	0.489	0.032	1.243	1.306	1.296	1.282	0.034	1.921	2.162	2.069	2.051	0.122
B118	0.52	0.51	0.52	0.517	0.006	1.28	1.27	1.28	1.277	0.006	1.97	1.99	1.93	1.963	0.031
B121	<2			<2		>2			>2		>2			1.94	0.22
B123	0.59	0.49	0.58	0.553	0.055	1.41	1.16	1.47	1.347	0.164	2.45	1.89	2.25	2.197	0.284
B125	0.454	0.441	0.443	0.446	0.007	1.177	1.159	1.104	1.147	0.038	1.847	1.814	1.882	1.848	0.034
B128	<1	<1	<1	>1	>1	>1	>1	>1	>1	>1	>1	>1	>1	1.94	0.22
B133	0.554	0.611	0.629	0.598	0.039	1.323	1.374	1.295	1.331	0.040	2.15	2.16	2.07	2.127	0.049
B135	0.41	0.42	0.42	0.417	0.006	>1.13	1.16	>1.13	1.160		>1.28	>1.28	>1.30		
B143	0.467	0.478	0.467	0.471	0.006	1.18	1.248	1.239	1.222	0.037	1.649	1.623	1.612	1.628	0.019
B146	0.457	0.489	0.458	0.468	0.018	1.22	1.21	1.22	1.217	0.006	1.96	1.96	1.97	1.963	0.006
B151	0.496	0.494	0.497	0.496	0.002	1.33	1.31	1.32	1.320	0.010	2.07	2.13	2.15	2.117	0.042
B154	0.426	0.465	0.367	0.419	0.049	1.21	1.21	1.191	1.204	0.011	1.841	1.824	1.871	1.845	0.024
B158	0.55	0.56	0.55	0.553	0.006	1.38	1.47	1.33	1.393	0.071	2.35	2.22	2.22	2.263	0.075
B167	0.52	0.54	0.53	0.530	0.010	1.3	1.33	1.33	1.320	0.017	1.97	2.01	1.93	1.970	0.040
B169	0.47	0.47		0.470		1.2	1.2		1.200		2	2		2.000	
B177	0.443	0.479	0.459	0.460	0.018	1.035	1.037	1.048	1.040	0.007	1.693	1.744	1.755	1.731	0.033
B178	0.534	0.531	0.516	0.527	0.010	1.42	1.41	1.34	1.390	0.044	2.25	2.17	2.05	2.157	0.101
B196	>1			>1							>1			1.94	0.22
B198	0.49	0.5	0.48	0.490	0.010	1.41	1.35	1.36	1.373	0.032	2.05	2.12	1.97	2.047	0.075
B199	0.44	0.45	0.44	0.443	0.006	1.15	1.17	1.2	1.173	0.025	1.8	1.85	1.83	1.827	0.025
B207	0.5885	0.5921	0.5939	0.592	0.003	>1.0	>1.0	>1.0	>1.0		>1.0	>1.0	>1.0	>1.0	
B213	0.61	0.58	0.63	0.607	0.025	1.43	1.48	1.37	1.427	0.055	2.18	1.99	2.25	2.140	0.135
B218	<1	<1	<1	>1	>1	>1	>1	>1	>1	>1	>1	>1	>1	2.140	0.135
B222	0.41	0.38	0.41	0.400	0.017	1.14	0.9	1.02	1.020	0.120	1.62	1.62	1.6	1.613	0.012
B229	0.5			0.500		>0.87					>0.87			1.613	
B128	<1	<1	<1	<1		>1	>1	>1			>1	>1	>1		

**Table B-7. Data summary table for CBD in three hemp samples.**

Data in **red** font have been flagged as a data entry of zero or results that include text (e.g., “< LOQ” or “present”). Data in **blue** font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

*Note: This table spans multiple pages; the NIST values and consensus values are included on all pages for convenience.*

Target Consensus	NRC HEMP-1 (Plant Sample 1)						Plant Sample 4						Plant Sample 6					
				0.541	0.070					0.589	0.019					0.241	0.046	
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD		
B001		0.502	0.519	0.518	0.5130	0.0095	0.529	0.569	0.587	0.5617	0.0297	0.261	0.259	0.271	0.2637	0.0064		
B003		0.50319	0.47665	0.51755	0.4991	0.0208	0.53724	0.5391	0.53192	0.5361	0.0037	0.26088	0.28382	0.21328	0.2527	0.0360		
B004		0.527	0.527	0.527	0.5270	0.0000	0.549	0.549	0.549	0.5490	0.0000	0.16	0.16	0.16	0.1600	0.0000		
B005		0.571	0.558	0.488	0.5390	0.0446	0.633	0.598	0.563	0.5980	0.0350	0.296	0.282	0.292	0.2900	0.0072		
B006		0.515	0.518	0.512	0.5150	0.0030	0.598	0.584	0.589	0.5903	0.0071	0.288	0.291	0.288	0.2890	0.0017		
B007		0.64	0.64	0.64	0.6400	0.0000	0.77	0.73	0.071	0.5237	0.3925	0.35	0.35	0.35	0.3500	0.0000		
B009																		
B012		0.48137	0.49165		0.4865	0.0073	0.544	0.562		0.5530	0.0127	0.25367	0.25058		0.2521	0.0022		
B013		0.51	0.51	0.51	0.5100	0.0000	0.55	0.56	0.57	0.5600	0.0100	0.3	0.3	0.3	0.3000	0.0000		
B014																		
B015		0.46	0.46	0.46	0.4600	0.0000	0.53	0.51	0.56	0.5333	0.0252	0.25	0.25	0.26	0.2533	0.0058		
B016		0.6389	0.6575	0.6522	0.6495	0.0096	0.7357	0.7356	0.6787	0.7167	0.0329	0.3406	0.3385	0.3356	0.3382	0.0025		
B018		0.54	0.54	0.53	0.5367	0.0058	0.62	0.56	0.67	0.6167	0.0551	0.29	0.29	0.29	0.2900	0.0000		
B020																		
B021																		
B022		0.547	0.538	0.543	0.5427	0.0045	0.66	0.614	0.654	0.6427	0.0250	0.284	0.283	0.284	0.2837	0.0006		
B023		0.5264	0.5386	0.5186	0.5279	0.0101	0.6402	0.648	0.6134	0.6339	0.0181	0.2995	0.3014	0.2977	0.2995	0.0019		
B024		0.53	0.53	0.52	0.5267	0.0058	0.49	0.5	0.51	0.5000	0.0100	0.23	0.23	0.23	0.2300	0.0000		
B026		0.52	0.52	0.52	0.5200	0.0000	0.65	0.6	0.6	0.6167	0.0289	0.3	0.3	0.29	0.2967	0.0058		
B027		0.5044	0.5143	0.5105	0.5097	0.0050	0.5817	0.5696	0.5829	0.5781	0.0074	0.2373	0.2451	0.2362	0.2395	0.0049		
B028																		
B029		0.483	0.488	0.456	0.4757	0.0172	0.564	0.611	0.554	0.5763	0.0304	0.219	0.219	0.209	0.2157	0.0058		
B030		0.54	0.54	0.54	0.5400	0.0000	0.61	0.64	0.62	0.6233	0.0153	0.3	0.3	0.3	0.3000	0.0000		
B031																		
B032																		
B033		0.32	0.38	0.45	0.3833	0.0651	0.44	0.53	0.59	0.5200	0.0755	0.25	0.28	0.28	0.2700	0.0173		
B035		0.52804	0.52556	0.5272	0.5269	0.0013	0.58707	0.5773	0.5661	0.5768	0.0105	0.27123	0.27786	0.28167	0.2769	0.0053		
B036		0.54	0.53	0.54	0.5367	0.0058	0.69	0.67	0.67	0.6767	0.0115	0.28	0.28	0.28	0.2800	0.0000		
B037																		
B038		0.614	0.512	0.497	0.5410	0.0637	0.678	0.702	0.703	0.6943	0.0142	0.375	0.294	0.304	0.3243	0.0442		
B041		0.48			0.4800		0.552			0.5520		0.256			0.2560			
B042																		
B043		0.479			0.4790		0.527			0.5270		0.248			0.2480			
B044		0.68	0.698	0.636	0.6713	0.0319	0.68	0.67	0.6	0.6500	0.0436	0.45	0.31	0.34	0.3667	0.0737		
B045		detected	detected				detected	detected				detected	detected					
B046		0.52	0.53	0.54	0.5300	0.0100	0.59	0.6	0.54	0.5767	0.0321	0.26	0.27	0.25	0.2600	0.0100		
B047		0.559	0.546	0.524	0.5430	0.0177	0.583	0.602	0.57	0.5850	0.0161	0.276	0.297	0.277	0.2833	0.0118		

	NRC HEMP-1 (Plant Sample 1)						Plant Sample 4				Plant Sample 6				
Target Consensus				0.541	0.070				0.589	0.019				0.241	0.046
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B048															
B049	0.5195	0.429	0.4025	0.4503	0.0613	0.524	0.5535	0.567	0.5482	0.0220	0.18567	0.3124	0.31192	0.2700	0.0730
B051	0.55	0.53	0.46	0.5133	0.0473	0.62	0.81	0.77	0.7333	0.1002	0.29	0.3	0.31	0.3000	0.0100
B052	0.53	0.56	0.52	0.5367	0.0208	0.58	0.54	0.55	0.5567	0.0208	0.25	0.25	0.24	0.2467	0.0058
B053	1.25	1.325	1.361	1.3120	0.0566	1.388	1.416	1.465	1.4230	0.0390	0.939	0.964	0.993	0.9653	0.0270
B054	0.50032	0.51714	0.48954	0.5023	0.0139	0.59064	0.60098	0.55672	0.5828	0.0232	0.26453	0.26328	0.26153	0.2631	0.0015
B055	0.581	0.5868	0.5917	0.5865	0.0054	0.6872	0.6621	0.6797	0.6763	0.0129	0.2888	0.2836	0.2904	0.2876	0.0036
B057	0.4564	0.4592	0.4532	0.4563	0.0030	0.6127	0.5876	0.5944	0.5982	0.0130	0.2787	0.2792	0.2615	0.2731	0.0101
B058	0.528	0.526	0.512	0.5220	0.0087	0.578	0.568	0.533	0.5597	0.0236	0.264	0.271	0.256	0.2637	0.0075
B060	0.53	0.5	0.49	0.5067	0.0208	0.61	0.59	0.63	0.6100	0.0200	0.3	0.28	0.31	0.2967	0.0153
B061	0.513	0.53	0.538	0.5270	0.0128	0.572	0.571	0.593	0.5787	0.0124	0.267	0.264	0.256	0.2623	0.0057
B062	0.564	0.574	0.559	0.5657	0.0076	0.661	0.659	0.645	0.6550	0.0087	0.3	0.3	0.297	0.2990	0.0017
B063															
B064	0.856			0.8560		0.831			0.8310		0.48			0.4800	
B065	0.47	0.52	0.52	0.5033	0.0289	0.58	0.67	0.65	0.6333	0.0473	0.32	0.32	0.3	0.3133	0.0115
B066	0.5478	0.5391	0.5508	0.5459	0.0061	0.7199	0.6835	0.6826	0.6953	0.0213	0.309	0.313	0.313	0.3117	0.0023
B068															
B069	0.5	0.52	0.49	0.5033	0.0153	0.58	0.57	0.54	0.5633	0.0208	0.28	0.27	0.29	0.2800	0.0100
B070	0.5	0.56	0.54	0.5333	0.0306	0.638	0.592	0.622	0.6173	0.0234	0.31	0.292	0.329	0.3103	0.0185
B071	0.59	0.6	0.6	0.5967	0.0058	0.57	0.59	0.61	0.5900	0.0200	0.3	0.29	0.3	0.2967	0.0058
B072	0.557	0.544	0.556	0.5523	0.0072	0.648	0.666	0.647	0.6537	0.0107	0.327	0.326	0.322	0.3250	0.0026
B073	0.4382			0.4382		0.4581			0.4581		0.234			0.2340	
B074	0.51	0.51	0.51	0.5100	0.0000	0.65	0.65	0.65	0.6500	0.0000	0.42	0.42	0.42	0.4200	0.0000
B076	0.55106	0.60219	0.60246	0.5852	0.0296	0.4939	0.41771	0.44432	0.4520	0.0387	0.28313	0.28332	0.28319	0.2832	0.0001
B077	0.536	0.517	0.49	0.5143	0.0231	0.584	0.591	0.59	0.5883	0.0038	0.276	0.27	0.308	0.2847	0.0204
B078															
B079															
B081	0.56	0.52	0.49	0.5233	0.0351	0.58	0.61	0.54	0.5767	0.0351	0.26	0.24	0.23	0.2433	0.0153
B082	0.566	0.557	0.559	0.5607	0.0047	0.613	0.624	0.607	0.6147	0.0086	0.26	0.262	0.266	0.2627	0.0031
B084	0.47	0.47	0.47	0.4700	0.0000	0.52	0.5	0.51	0.5100	0.0100	0.25	0.25	0.25	0.2500	0.0000
B085	0.605	0.562	0.514	0.5603	0.0455	0.656	0.696	0.659	0.6703	0.0223	0.275	0.214	0.254	0.2477	0.0310
B086															
B087															
B088	0.474	0.5	0.505	0.4930	0.0166	0.541	0.546	0.562	0.5497	0.0110	0.264	0.256	0.247	0.2557	0.0085
B089	0.3229	0.3517	0.3387	0.3378	0.0144	0.8038	0.8282	0.9652	0.8657	0.0870	0.513	0.5253	0.5269	0.5217	0.0076
B090	0.47	0.53	0.52	0.5067	0.0321	0.62	0.61	0.63	0.6200	0.0100	0.3	0.29	0.29	0.2933	0.0058
B091	0.445	0.451	0.444	0.4467	0.0038	0.588	0.502	0.515	0.5350	0.0464	0.246	0.253	0.249	0.2493	0.0035
B092	0.58	0.59	0.59	0.5867	0.0058	0.74	0.68	0.67	0.6967	0.0379	0.36	0.36	0.36	0.3600	0.0000
B094	0.52	0.5	0.54	0.5200	0.0200	0.57	0.61	0.57	0.5833	0.0231	0.28	0.27	0.27	0.2733	0.0058
B095	0.559	0.566	0.562	0.5623	0.0035	0.557	0.535	0.572	0.5547	0.0186	0.319	0.322	0.321	0.3207	0.0015
B096	0.537	0.528	0.52	0.5283	0.0085	0.555	0.617	0.588	0.5867	0.0310	0.3	0.3	0.309	0.3030	0.0052
B097	0.64	0.66	0.64	0.6467	0.0115	0.63	0.61	0.63	0.6233	0.0115	0.28	0.27	0.26	0.2700	0.0100
B098	0.46	0.45	0.46	0.4567	0.0058	2.33	2.96	2.31	2.5333	0.3696	0.22	0.22	0.22	0.2200	0.0000

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.541	0.070				0.589	0.019				0.241	0.046
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B099	0.538			0.5380		0.619			0.6190		0.268			0.2680	
B100	0.58	0.52		0.5500	0.0424	0.67	0.61		0.6400	0.0424	0.32	0.31		0.3150	0.0071
B102	0.5211	0.5055	0.5013	0.5093	0.0104	0.5334	0.5496	0.5551	0.5460	0.0113	0.2733	0.2756	0.2705	0.2731	0.0026
B104	0.43	0.428	0.443	0.4337	0.0081	0.486	0.461	0.466	0.4710	0.0132	0.187	0.193	0.171	0.1837	0.0114
B105															
B106	0.549	0.544	0.541	0.5447	0.0040	0.655	0.613	0.626	0.6313	0.0215	0.303	0.301	0.304	0.3027	0.0015
B108															
B109	0.502	0.49	0.473	0.4883	0.0146	0.556	0.564	0.494	0.5380	0.0383	0.271	0.27	0.271	0.2707	0.0006
B110	0.5	0.51	0.52	0.5100	0.0100	0.57	0.61	0.63	0.6033	0.0306	0.25	0.25	0.26	0.2533	0.0058
B111															
B113	0.53	0.52	0.52	0.5233	0.0058	0.56	0.49	0.53	0.5267	0.0351	0.26	0.29	0.29	0.2800	0.0173
B114															
B115	0.53	0.56	0.58	0.5567	0.0252	0.6356	0.6245	0.6518	0.6373	0.0137	0.3024	0.305	0.3016	0.3030	0.0018
B116	0.5027	0.5015	0.5168	0.5070	0.0085	0.5839	0.5907	0.5693	0.5813	0.0109	0.2378	0.2357	0.2362	0.2366	0.0011
B117	0.374	0.429	0.45	0.4177	0.0392	0.414	0.618	0.564	0.5320	0.1057	0.675	0.324	0.312	0.4370	0.2062
B120															
B122															
B124	0.4955	0.547	0.5255	0.5227	0.0259	0.5725	0.6135	0.6067	0.5976	0.0220	0.263	0.298	0.249	0.2700	0.0252
B125	0.464	0.467	0.457	0.4627	0.0051	0.536	0.518	0.52	0.5247	0.0099	0.242	0.243	0.231	0.2387	0.0067
B126	0.4774	0.4779	0.4626	0.4726	0.0087	0.5234	0.5159	0.5545	0.5313	0.0205	0.229	0.2178	0.2126	0.2198	0.0084
B127	0.5448	0.5526	0.5683	0.5552	0.0120	0.633	0.6551	0.7107	0.6663	0.0400	0.3201	0.3275	0.3606	0.3361	0.0216
B129															
B130	0.46	0.48	0.47	0.4700	0.0100	0.54	0.55	0.54	0.5433	0.0058	0.28	0.28	0.27	0.2767	0.0058
B131	0.593	0.5831	0.5612	0.5791	0.0163	0.8657	0.9296	0.8445	0.8799	0.0443	0.3755	0.3659	0.3695	0.3703	0.0048
B132	0.561	0.58	0.561	0.5673	0.0110	0.633	0.614	0.553	0.6000	0.0418	0.287	0.244	0.28	0.2703	0.0231
B136	0.542			0.5420		0.586			0.5860		0.226			0.2260	
B137	0.335	0.314	0.328	0.3257	0.0107	0.341	0.317	0.351	0.3363	0.0175	0.172	0.169	0.184	0.1750	0.0079
B141	0.508	0.488	0.494	0.4967	0.0103	0.568	0.621	0.58	0.5897	0.0278	0.297	0.297	0.288	0.2940	0.0052
B142	0.574	0.564	0.562	0.5667	0.0064	0.693	0.63	0.642	0.6550	0.0335	0.385	0.38	0.369	0.3780	0.0082
B144	0.56	0.5	0.6	0.5533	0.0503	0.7	0.7	0.7	0.7000		0.3	0.3	0.3	0.3000	
B146	0.483	0.478	0.483	0.4813	0.0029	0.501	0.584	0.578	0.5543	0.0463	0.231	0.248	0.248	0.2423	0.0098
B147	0.502	0.516	0.482	0.5000	0.0171	0.61	0.596	0.575	0.5937	0.0176	0.295	0.294	0.274	0.2877	0.0118
B148															
B149	0.565	0.52	0.486	0.5237	0.0396	0.543	0.574	0.607	0.5747	0.0320	0.282	0.267	0.262	0.2703	0.0104
B151	0.605	0.585	0.545	0.5783	0.0306	0.783	0.791	0.754	0.7760	0.0195	0.301	0.295	0.313	0.3030	0.0092
B152															
B153	0.6	0.607	0.615	0.6073	0.0075	0.602	0.604	0.632	0.6127	0.0168	0.317	0.3	0.324	0.3137	0.0123
B154	0.464	0.487	0.444	0.4650	0.0215	0.464	0.506	0.579	0.5163	0.0582	0.241	0.28	0.295	0.2720	0.0279
B157	0.51			0.5100		0.56			0.5600		0.26	0.25		0.2550	0.0071
B158	0.57	0.54	0.54	0.5500	0.0173	0.65	0.63	0.6	0.6267	0.0252	0.27	0.29	0.28	0.2800	0.0100
B159	0.5831	0.5739	0.5642	0.5737	0.0095	0.6028	0.6175	0.6104	0.6102	0.0074	0.314	0.3538	0.3144	0.3274	0.0229
B160	0.5282	0.5122	0.5024	0.5143	0.0130	0.6004	0.5414	0.596	0.5793	0.0329	0.29699	0.30901	0.30666	0.3042	0.0064
B161	0.614	0.599	0.616	0.6097	0.0093	0.725	0.697	0.745	0.7223	0.0241	0.336	0.346	0.342	0.3413	0.0050

	NRC HEMP-1 (Plant Sample 1)						Plant Sample 4				Plant Sample 6				
Target Consensus				0.541	0.070				0.589	0.019				0.241	0.046
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B163	0.626	0.553	0.533	0.5707	0.0490	0.587	0.556	0.453	0.5320	0.0701	0.329	0.307	0.311	0.3157	0.0117
B164	0.48	0.56	0.55	0.5300	0.0436	0.44	0.59	0.63	0.5533	0.1002	0.31	0.33	0.32	0.3200	0.0100
B165															
B166	1.18	1.16	1.17	1.1700	0.0100	0.67	0.73	0.74	0.7133	0.0379	0.36	0.37	0.36	0.3633	0.0058
B167															
B168	0.52	0.538	0.527	0.5283	0.0091	0.604	0.601	0.568	0.5910	0.0200	0.326	0.318	0.328	0.3240	0.0053
B169	0.49	0.49		0.4900		0.64	0.64		0.6400		0.3	0.3		0.3000	
B170															
B172	0.4544	0.424	0.43208	0.4368	0.0157	0.49176	0.47944	0.4392	0.4701	0.0275	0.2044	0.2184	0.2208	0.2145	0.0089
B173	0.47616	0.48986	0.4687	0.4782	0.0107	0.62176	0.6385	0.65388	0.6380	0.0161	0.29915	0.27714	0.27394	0.2834	0.0137
B174	0.51	0.51	0.52	0.5133	0.0058	0.57	0.58	0.61	0.5867	0.0208	0.27	0.28	0.28	0.2767	0.0058
B175	0.91	0.83	0.83	0.8567	0.0462	0.68	0.79	0.76	0.7433	0.0569	0.33	0.36	0.32	0.3367	0.0208
B176	0.523	0.455	0.511	0.4963	0.0363	0.541	0.535	0.54	0.5387	0.0032	0.256	0.26	0.259	0.2583	0.0021
B178	0.567	0.555	0.54	0.5540	0.0135	0.599	0.631	0.606	0.6120	0.0168	0.312	0.303	0.303	0.3060	0.0052
B181	0.46			0.4600		0.51			0.5100		0.25			0.2500	
B182	0.42	0.42		0.4200		0.5	0.52	0.51	0.5100	0.0100	0.26	0.26	0.26	0.2600	
B183	0.5347	0.5318	0.5213	0.5293	0.0071	0.5624	0.5967	0.5567	0.5719	0.0216	0.2726	0.261	0.2668	0.2668	0.0058
B184	0.426	0.441	0.407	0.4247	0.0170	0.507	0.582	0.547	0.5453	0.0375	0.264	0.269	0.289	0.2740	0.0132
B185															
B186	0.488	0.469	0.483	0.4800	0.0098	0.465	0.501	0.549	0.5050	0.0421	0.203	0.204	0.201	0.2027	0.0015
B187	0.5596	0.5267	0.5524	0.5462	0.0173	0.5483	0.5889	0.6177	0.5850	0.0349	0.2279	0.229	0.2129	0.2233	0.0090
B188															
B189	0.5212	0.5073	0.5195	0.5160	0.0076	0.5736	0.5345	0.5457	0.5513	0.0201	0.2616	0.2647	0.2657	0.2640	0.0021
B190	0.51	0.5	0.5	0.5033	0.0058	0.59	0.56	0.56	0.5700	0.0173	0.28	0.28	0.28	0.2800	
B192	0.53217	0.53811	0.53445	0.5349	0.0030	0.57489	0.58835	0.5688	0.5773	0.0100	0.26494	0.261	0.26113	0.2624	0.0022
B193	0.6	0.687	0.579	0.6220	0.0573	0.597	0.647	0.561	0.6017	0.0432	0.239	0.232	0.257	0.2427	0.0129
B195	0.502	0.498	0.492	0.4973	0.0050	0.477	0.47	0.467	0.4713	0.0051	0.241	0.245	0.239	0.2417	0.0031
B196															
B198	0.49	0.49	0.5	0.4933	0.0058	0.59	0.59	0.65	0.6100	0.0346	0.29	0.29	0.28	0.2867	0.0058
B199	0.44	0.43	0.44	0.4367	0.0058	0.52	0.47	0.49	0.4933	0.0252	0.23	0.23	0.23	0.2300	
B200															
B202	0.466	0.47	0.475	0.4703	0.0045	0.541	0.534	0.538	0.5377	0.0035	0.21	0.201	0.205	0.2053	0.0045
B204	0.527	0.523	0.529	0.5263	0.0031	0.615	0.554	0.582	0.5837	0.0305	0.275	0.279	0.283	0.2790	0.0040
B205	1.45	1.28	1.32	1.3500	0.0889	6.55	6.09	6.06	6.2333	0.2747	3.31	3.11	2.97	3.1300	0.1709
B206	0.56	0.572	0.562	0.5647	0.0064	0.615	0.574	0.609	0.5993	0.0221	0.284	0.285	0.284	0.2843	0.0006
B208															
B210															
B212	0.563	0.558	0.552	0.5577	0.0055	0.592	0.584	0.55	0.5753	0.0223	0.271	0.273	0.27	0.2713	0.0015
B213	0.47	0.52	0.6	0.5300	0.0656	0.62	0.62	0.63	0.6233	0.0058	0.32	0.31	0.29	0.3067	0.0153
B215	0.46			0.4600		0.51			0.5100		0.23			0.2300	
B216	0.569	0.517	0.505	0.5303	0.0340	0.606	0.642	0.667	0.6383	0.0307	0.289	0.29	0.284	0.2877	0.0032
B217	0.53	0.54	0.53	0.5333	0.0058	0.6	0.56	0.59	0.5833	0.0208	0.29	0.28	0.28	0.2833	0.0058
B219	0.523	0.522	0.529	0.5247	0.0038	0.616	0.652	0.645	0.6377	0.0191	0.283	0.295	0.292	0.2900	0.0062

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4				Plant Sample 6					
Target Consensus				0.541 0.517	0.070 0.058				0.589 0.589	0.019 0.072				0.241 0.278	0.046 0.043
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B220															
B221	0.664	0.659	0.646	0.6563	0.0093	0.692	0.75	0.759	0.7337	0.0364	0.312	0.321	0.312	0.3150	0.0052
B222	0.42	0.46	0.43	0.4367	0.0208	0.24	0.31	0.19	0.2467	0.0603	0.25	0.27	0.23	0.2500	0.0200
B223	0.494	0.493	0.496	0.4943	0.0015	0.553	0.581	0.593	0.5757	0.0205	0.268	0.275	0.274	0.2723	0.0038
B224	0.56	0.56	0.56	0.5600		0.65	0.65	0.65	0.6500		0.3	0.31	0.3	0.3033	0.0058
B226	0.49052	0.50497	0.49458	0.4967	0.0075	0.60563	0.60404		0.6048	0.0011	0.28126	0.28741	0.28703	0.2852	0.0034
B227	0.488	0.49	0.46	0.4793	0.0168	0.67	0.53	0.56	0.5867	0.0737	0.24	0.24	0.23	0.2367	0.0058
B228	0.513	0.499	0.494	0.5020	0.0098	0.604	0.555	0.526	0.5617	0.0394	0.272	0.272	0.267	0.2703	0.0029
B230															
B233	0.369	0.37	0.349	0.3627	0.0118	0.534	0.51	0.597	0.5470	0.0449	0.327	0.221	0.29	0.2793	0.0538
B234	0.33	0.34	0.34	0.3367	0.0058	0.49	0.49	0.48	0.4867	0.0058	0.21	0.21	0.22	0.2133	0.0058

**Table B-8. Data summary table for CBD in three marijuana samples.**

Data in red font have been flagged as a data entry of zero or results that include text (e.g., “< LOQ” or “present”). Data in blue font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

Target Consensus	Plant Sample 2					Plant Sample 3					Plant Sample 5					
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
		detected	detected	detected	detected	detected	detected	detected	detected	detected	detected	detected	detected	detected	detected	
B045		1.32	1.36	1.32	1.333	0.023	1.2	1.19	1.2	1.197	0.006	1.48	1.48	1.47	1.477	0.006
B046		1.5015	1.5313	1.503	1.512	0.017	1.5716	1.5502	1.5375	1.553	0.017	1.6551	1.707	1.6754	1.679	0.026
B055		1.391	1.426	1.35	1.389	0.038	1.356	1.366	1.361	1.361	0.005	1.506	1.508	1.529	1.514	0.013
B057		1.418	1.41	1.361	1.396	0.031	1.49	1.467	1.429	1.462	0.031	1.54	1.565	1.527	1.544	0.019
B062		1.36	1.38	1.35	1.363	0.015	1.41	1.43	1.45	1.430	0.020	1.53	1.54	1.54	1.537	0.006
B071		1.2			1.200		1.26			1.260		1.32			1.320	
B099		1.183	1.191	1.195	1.190	0.006	1.244	1.226	1.225	1.232	0.011	1.339	1.331	1.313	1.328	0.013
B109		1.092	1.207	1.053	1.117	0.080	1.013	1.001	1.066	1.027	0.035	1.017	1.168	1.068	1.084	0.077
B117		1.175	1.157	1.152	1.161	0.012	1.247	1.227	1.218	1.231	0.015	1.337	1.273	1.307	1.306	0.032
B125		1.28	1.28	1.28	1.280		1.32	1.31	1.33	1.320	0.010	1.4	1.41	1.42	1.410	0.010
B146		1.9	1.9	1.87	1.890	0.017	2.07	2.13	1.99	2.063	0.070	2.16	2.23	2.15	2.180	0.044
B151		1.1	1.186	0.929	1.072	0.131	1.19	1.189	1.169	1.183	0.012	1.27	1.218	1.243	1.244	0.026
B154		1.49	1.43	1.43	1.450	0.035	1.51	1.48	1.52	1.503	0.021	1.66	1.64	1.62	1.640	0.020
B158		1.31	1.32	1.3	1.310	0.010	1.35	1.34	1.32	1.337	0.015	1.43	1.39	1.41	1.410	0.020
B167		1.5	1.5		1.500		1.5	1.5		1.500		1.6	1.6		1.600	
B169		1.48	1.45	1.39	1.440	0.046	1.54	1.47	1.48	1.497	0.038	1.61	1.57	1.58	1.587	0.021
B178		1.18	1.31	1.2	1.230	0.070	1.33	1.37	1.38	1.360	0.026	1.43	1.44	1.42	1.430	0.010
B198		1.16	1.17	1.15	1.160	0.010	1.18	1.19	1.2	1.190	0.010	1.29	1.31	1.3	1.300	0.010
B199		1.44	1.45	1.58	1.490	0.078	1.52	1.55	1.36	1.477	0.102	1.43	1.52	1.55	1.500	0.062
B213		1.12	1.07	1.1	1.097	0.025	1.22	1.15	1.15	1.173	0.040	1.2	1.24	1.3	1.247	0.050
B222		detected	detected	detected	detected	detected	detected	detected	detected	detected	detected	detected	detected	detected	detected	
B045		1.32	1.36	1.32	1.333	0.023	1.2	1.19	1.2	1.197	0.006	1.48	1.48	1.47	1.477	0.006
B046		1.5015	1.5313	1.503	1.512	0.017	1.5716	1.5502	1.5375	1.553	0.017	1.6551	1.707	1.6754	1.679	0.026
B055		1.391	1.426	1.35	1.389	0.038	1.356	1.366	1.361	1.361	0.005	1.506	1.508	1.529	1.514	0.013
B057		1.418	1.41	1.361	1.396	0.031	1.49	1.467	1.429	1.462	0.031	1.54	1.565	1.527	1.544	0.019
B062		1.36	1.38	1.35	1.363	0.015	1.41	1.43	1.45	1.430	0.020	1.53	1.54	1.54	1.537	0.006
B071		1.2			1.200		1.26			1.260		1.32			1.320	
B099		1.183	1.191	1.195	1.190	0.006	1.244	1.226	1.225	1.232	0.011	1.339	1.331	1.313	1.328	0.013

**Table B-9. Data summary table for CBDA in three hemp samples.**

Data in **blue** font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .  
*Note: This table spans multiple pages; the NIST values and consensus values are included on all pages for convenience.*

Target Consensus	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
				1.460	0.080				8.11	0.42				4.21	0.46
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg
B001	1.49	1.46	1.36	1.437	0.068	6.47	6.84	6.94	6.750	0.248	4.16	4.09	4.11	4.120	0.036
B003	1.17313	1.11052	1.23148	1.172	0.060	5.79094	5.75336	5.68722	5.744	0.053	3.52319	3.44622	2.6942	3.221	0.458
B004	1.33	1.33	1.33	1.330	0.000	7.125	7.125	7.125	7.125	0.000	2.726	2.726	2.726	2.726	0.000
B005	1.442	1.429	1.212	1.361	0.129	7.277	7.366	6.803	7.149	0.303	4.426	4.44	4.427	4.431	0.008
B006	1.335	1.362	1.339	1.345	0.015	7.002	7.002	6.979	6.994	0.013	4.555	4.542	4.515	4.537	0.020
B007	1.47	1.47	1.47	1.470	0.000	7.97	7.83	7.48	7.760	0.252	4.72	4.71	4.69	4.707	0.015
B009															
B012	1.23828	1.23228		1.235	0.004	6.6996	6.93555		6.818	0.167	3.975	3.76395		3.869	0.149
B013	1.32	1.3	1.31	1.310	0.010	6.34	6.46	6.61	6.470	0.135	4.51	4.47	4.41	4.463	0.050
B014															
B015	1.27	1.25	1.27	1.263	0.012	6.29	6.03	6.37	6.230	0.178	4.06	4.03	4.13	4.073	0.051
B016	1.8055	1.7822	1.7553	1.781	0.025	8.9686	8.9942	8.2761	8.746	0.407	5.4019	5.3861	5.3576	5.382	0.022
B018	1.36	1.28	1.29	1.310	0.044	7.54	6.94	8.32	7.600	0.692	4.64	4.6	4.63	4.623	0.021
B020															
B021															
B022	1.597	1.576	1.578	1.584	0.012	8.569	8.128	8.62	8.439	0.271	4.678	4.688	4.638	4.668	0.026
B023	1.385	1.3873	1.3405	1.371	0.026	7.3046	7.2654	7.2132	7.261	0.046	4.293	4.3948	4.2927	4.327	0.059
B024	1.22	1.24	1.26	1.240	0.020	5.94	6.7	6.22	6.287	0.384	3.7	3.68	3.65	3.677	0.025
B026	1.58	1.7	1.55	1.610	0.079	9.19	8.97	9.05	9.070	0.111	5.4	5.67	5.19	5.420	0.241
B027	1.2889	1.2627	1.2655	1.272	0.014	6.9479	6.8358	6.8798	6.888	0.056	3.7575	3.7435	3.6468	3.716	0.060
B028															
B029	1.132	1.127	1.097	1.119	0.019	6.947	7.097	6.303	6.782	0.422	3.919	4.049	3.989	3.986	0.065
B030	1.38	1.38	1.41	1.390	0.017	7.06	7.51	7.27	7.280	0.225	4.59	4.6	4.54	4.577	0.032
B031															
B033	0.81	1.03	1.29	1.043	0.240	4.9	6.39	6.91	6.067	1.043	3.58	4.6	4.35	4.177	0.532
B035	1.38319	1.39964	1.40651	1.396	0.012	6.92903	6.96388	6.90529	6.933	0.029	4.3864	4.44817	4.38663	4.407	0.036
B036	1.62	1.58	1.62	1.607	0.023	8.9	9.08	8.99	8.990	0.090	5.06	5.01	5.07	5.047	0.032
B037															
B038	1.3	1.347	1.298	1.315	0.028	5.935	7.204	7.077	6.739	0.699	4.084	3.949	4.007	4.013	0.068
B041	1.369			1.369		7.516			7.516		4.626			4.626	
B043	1.335			1.335		7.298			7.298		4.532			4.532	
B044	1.306	1.34	1.355	1.334	0.025	7.05	6.82	6.5	6.790	0.276	3.75	3.78	3.85	3.793	0.051
B046	1.34	1.39	1.38	1.370	0.026	7.3	7.48	6.49	7.090	0.527	3.97	4.03	3.86	3.953	0.086
B047	1.596	1.602	1.547	1.582	0.030	7.481	7.245	7.061	7.262	0.211	4.676	4.944	4.755	4.792	0.138
B048															
B049	1.14	1.1225	1.026	1.096	0.061	5.5065	6.2195	6.423	6.050	0.481	2.3805	3.944	3.948	3.424	0.904
B051	1.32	1.26	1.23	1.270	0.046	7.17	7.21	6.28	6.887	0.526	4.04	3.97	3.59	3.867	0.242
B052	1.32	1.39	1.3	1.337	0.047	7.61	7.02	7.15	7.260	0.310	3.98	4.24	4.15	4.123	0.132

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				1.460 1.33	0.080 0.16				8.11 7.04	0.42 0.79				4.21 4.23	0.46 0.52
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B053	1.627	1.705	1.716	1.683	0.049	3.888	3.963	4.043	3.965	0.078	3.124	3.332	3.299	3.252	0.112
B054	1.2919	1.29157	1.25312	1.279	0.022	7.52642	7.45883	7.01912	7.335	0.275	4.34689	4.3393	4.30089	4.329	0.025
B055	1.6678	1.6141	1.6233	1.635	0.029	8.1076	8.018	8.2687	8.131	0.127	4.6234	4.358	4.3547	4.445	0.154
B057	1.249	1.23	1.237	1.239	0.010	6.619	6.564	6.489	6.557	0.065	4.136	4.17	3.905	4.070	0.144
B058	1.43	1.4	1.38	1.403	0.025	7.39	7.2	6.79	7.127	0.307	4.38	4.42	4.24	4.347	0.095
B060	1.59	1.64	1.61	1.613	0.025	8.07	8.04	8.06	8.057	0.015	5.11	5.07	5.08	5.087	0.021
B061	1.32	1.388	1.396	1.368	0.042	6.969	6.909	7.182	7.020	0.143	4.201	4.233	4.169	4.201	0.032
B062	1.462	1.484	1.448	1.465	0.018	7.655	7.625	7.837	7.706	0.115	4.39	4.449	4.242	4.360	0.107
B063															
B064	1.274			1.274		7.173			7.173		0.065			0.065	
B065	1.2	1.35	1.35	1.300	0.087	6.82	7.8	7.41	7.343	0.493	4.87	4.8	4.45	4.707	0.225
B066	1.5036	1.4812	1.5245	1.503	0.022	8.4213	8.0349	7.9384	8.132	0.256	4.8765	4.8793	4.9423	4.899	0.037
B068															
B069	1.43	1.45	1.43	1.437	0.012	7.41	7.18	6.97	7.187	0.220	4.84	4.66	4.79	4.763	0.093
B070	1.3	1.47	1.38	1.383	0.085	7.298	6.848	6.911	7.019	0.244	4.47	4.367	4.52	4.452	0.078
B071	1.34	1.28	1.25	1.290	0.046	6.2	6.38	6.33	6.303	0.093	4.16	4.01	3.99	4.053	0.093
B072	1.37	1.33	1.39	1.363	0.031	7.08	7.34	7.15	7.190	0.135	4.5	4.53	4.49	4.507	0.021
B073	1.2939			1.294		6.7634			6.763		4.4565			4.457	
B074	1.31	1.31	1.31	1.310	0.000	6.39	6.39	6.39	6.390	0.000	4.44	4.44	4.44	4.440	0.000
B076	1.38466	1.26026	1.37777	1.341	0.070	9.60382	7.73584	8.23985	8.527	0.966	4.05199	3.95779	4.04932	4.020	0.054
B077	1.459	1.376	1.292	1.376	0.084	6.975	7.511	7.445	7.310	0.292	4.028	3.948	4.587	4.188	0.348
B078															
B079															
B081	1.44	1.33	1.29	1.353	0.078	7.08	7.16	6.64	6.960	0.280	4.23	3.87	3.71	3.937	0.266
B082	1.521	1.485	1.514	1.507	0.019	7.598	7.673	7.448	7.573	0.115	4.158	4.169	4.254	4.194	0.053
B084	1.166	1.196	1.181	1.181	0.015	6.701	6.69	6.698	6.696	0.006	4.01	4.08	4.04	4.043	0.035
B085	1.007	0.897	0.824	0.909	0.092	4.575	5.107	4.521	4.734	0.324	2.516	2.342	2.138	2.332	0.189
B086															
B088	1.159	1.192	1.2	1.184	0.022	7.033	7.197	7.246	7.159	0.112	4.324	4.314	4.223	4.287	0.056
B089	0.5238	0.5259	0.5234	0.524	0.001	2.2876	2.3467	2.3165	2.317	0.030	1.5925	1.575	1.6037	1.590	0.014
B090	1.21	1.4	1.38	1.330	0.104	7.25	7.08	7.38	7.237	0.150	4.55	4.52	4.5	4.523	0.025
B091	1.21	1.188	1.193	1.197	0.012	7.585	6.43	6.519	6.845	0.643	4.007	4.041	3.982	4.010	0.030
B092	1.46	1.42	1.4	1.427	0.031	6.15	5.8	5.66	5.870	0.252	4.37	4.36	4.39	4.373	0.015
B094	1.28	1.26	1.37	1.303	0.059	6.79	7.27	6.75	6.937	0.289	4.31	4.13	4.14	4.193	0.101
B095	1.222	1.186	1.121	1.176	0.051	6.651	6.158	6.002	6.270	0.339	4.292	4.3	4.205	4.266	0.053
B096	1.406	1.353	1.351	1.370	0.031	6.707	7.223	7.292	7.074	0.320	4.685	4.542	4.582	4.603	0.074
B097	1.26	1.3	1.25	1.270	0.026	7.03	7.12	7.19	7.113	0.080	3.89	3.87	3.72	3.827	0.093
B098											4.07	4.08	4.06	4.070	0.010
B099	1.489			1.489		6.917			6.917		3.11			3.110	
B100	1.468	1.385		1.427	0.059	7.897	7.394		7.646	0.356	4.965	4.855		4.910	0.078
B102	1.3607	1.3062	1.314	1.327	0.029	6.9236	6.8122	6.9333	6.890	0.067	4.4432	4.4631	4.349	4.418	0.061
B104	1.33	1.309	1.313	1.317	0.011	7.031	7.242	7.246	7.173	0.123	4.104	4.154	4.186	4.148	0.041
B106	1.35	1.343	1.337	1.343	0.007	7.23	6.921	7.076	7.076	0.155	4.192	4.152	4.163	4.169	0.021

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				1.460 1.33	0.080 0.16				8.11 7.04	0.42 0.79				4.21 4.23	0.46 0.52
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B108															
B109	1.195	1.157	1.157	1.170	0.022	6.495	6.558	5.744	6.266	0.453	3.884	3.858	3.914	3.885	0.028
B110	1.23	1.21	1.12	1.187	0.059	7.2	6.7	6.9	6.933	0.252	3.45	3.46	3.83	3.580	0.217
B111															
B113	1.32	1.32	1.29	1.310	0.017	7.43	6.54	6.91	6.960	0.447	4.46	4.95	4.93	4.780	0.277
B114															
B115	1.52	1.54	1.65	1.570	0.070	6.31	6.25	6.36	6.307	0.055	4.503	4.5	4.487	4.497	0.009
B116	1.2949	1.2804	1.2929	1.289	0.008	7.1791	7.3773	6.8152	7.124	0.285	3.7327	3.9012	3.7224	3.785	0.100
B117	1.097	1.263	1.327	1.229	0.119	6.264	6.559	6.514	6.446	0.159	4.332	4.023	4.489	4.281	0.237
B120															
B122															
B124	1.2805	1.3805	1.356	1.339	0.052	8.074	8.277	7.487	7.946	0.410	4.029	4.415	4.063	4.169	0.214
B125	1.218	1.217	1.2	1.212	0.010	6.465	6.265	6.297	6.342	0.107	3.765	3.783	3.602	3.717	0.100
B126	1.43	1.449	1.382	1.420	0.035	7.404	7.154	7.717	7.425	0.282	4.8	4.65	4.563	4.671	0.120
B127	1.4295	1.45	1.3638	1.414	0.045	7.3411	7.8957	7.9316	7.723	0.331	5.0387	5.1304	5.1368	5.102	0.055
B129															
B130	1.26	1.31	1.21	1.260	0.050	6.99	7.42	6.51	6.973	0.455	4.52	4.7	4.26	4.493	0.221
B131	1.4088	1.3706	1.3182	1.366	0.045	7.7154	8.3787	7.5895	7.895	0.424	4.1399	4.1941	4.1921	4.175	0.031
<b>B132</b>	<b>2.112</b>	<b>1.984</b>	<b>2.037</b>	<b>2.044</b>	<b>0.064</b>	8.397	7.648	6.88	7.642	0.759	5.254	5.318	5.145	5.239	0.087
B136															
B137	1.348	1.256	1.337	1.314	0.050	6.766	6.843	6.933	6.847	0.084	4.123	4.537	4.21	4.290	0.218
B141	1.12	1.15	1.28	1.183	0.085	5.385	6.433	6.27	6.029	0.564	4.081	4.05	4.22	4.117	0.091
B142	1.368	1.349	1.322	1.346	0.023	7.584	6.997	6.999	7.193	0.338	4.307	4.366	4.217	4.297	0.075
B144	1.4	1.4	1.5	1.433	0.058	7.4	7.2	7.6	7.400	0.200	3.9	4.4	4.5	4.267	0.321
B146	1.23	1.21	1.24	1.227	0.015	6.14	7.31	7.19	6.880	0.644	3.95	4.01	3.98	3.980	0.030
B147	1.214	1.23	1.222	1.222	0.008	6.718	6.619	9.537	7.625	1.657	4.225	4.225	4.153	4.201	0.042
B148															
B149	1.24	1.103	1.079	1.141	0.087	6.626	7.07	7.162	6.953	0.287	4.503	4.202	4.348	4.351	0.151
B152															
B153	1.531	1.57	1.549	1.550	0.020	8.013	7.916	8.305	8.078	0.202	5.078	4.88	5.329	5.096	0.225
B154	1.183	1.239	1.129	1.184	0.055	5.762	6.28	7.276	6.439	0.769	4.132	4.292	4.55	4.325	0.211
B157	1.32			1.320		6.96			6.960		4.16	4.13		4.145	0.021
B158	1.78	1.62	1.62	1.673	0.092	8.62	8.2	7.93	8.250	0.348	4.57	4.82	4.93	4.773	0.184
B159	1.4685	1.4245	1.3989	1.431	0.035	7.1203	7.3844	7.2257	7.243	0.133	4.2029	4.5414	4.4439	4.396	0.174
B160	1.3342	1.2844	1.2704	1.296	0.034	7.128	6.4526	6.957	6.846	0.351	4.2434	4.4088	4.5816	4.411	0.169
B161	1.587	1.601	1.626	1.605	0.020	8.895	8.384	8.527	8.602	0.264	5.202	5.296	5.242	5.247	0.047
<b>B163</b>	<b>2.16</b>	<b>1.71</b>	<b>1.71</b>	<b>1.860</b>	<b>0.260</b>	8.56	7.84	6.08	7.493	1.276	5.69	5.53	5.61	5.610	0.080
B164	1.21	1.28	1.31	1.267	0.051	4.86	6.3	6.82	5.993	1.015	4.28	4.37	4.33	4.327	0.045
B165															
B166	<0.02	<0.02	<0.02			6.18	6.87	7.16	6.737	0.503	3.94	3.92	3.94	3.933	0.012
B167															
B168	1.37	1.37	1.35	1.363	0.012	6.86	6.81	6.44	6.703	0.229	4.59	4.54	4.51	4.547	0.040
B170															

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				1.460	0.080				8.11	0.42				4.21	0.46
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B172	1.17	1.04904	1.08216	1.100	0.063	6.09352	5.78	5.55728	5.810	0.269	3.31608	3.47544	3.42712	3.406	0.082
B173	1.38812	1.46575	1.37737	1.410	0.048	7.53136	7.68897	7.87114	7.697	0.170	4.64297	4.3896	4.30479	4.446	0.176
B174	1.36	1.36	1.4	1.373	0.023	6.95	7.12	7.83	7.300	0.467	4.38	4.35	4.53	4.420	0.096
B175	1.58	1.53	1.5	1.537	0.040	6.85	6.87	7.18	6.967	0.185	3.9	3.85	3.88	3.877	0.025
B176	1.231	1.273	1.243	1.249	0.022	6.266	6.731	6.436	6.478	0.235	3.792	4.088	3.992	3.957	0.151
B178	1.6	1.6	1.53	1.577	0.040	8.62	8.81	8.53	8.653	0.143	5.67	5.43	5.54	5.547	0.120
B181	1.23			1.230		8.04			8.040		5.36			5.360	
B182	1.04	1.07		1.055	0.021	6.49	6.85	6.88	6.740	0.217	3.99	4.12	4.02	4.043	0.068
B183	1.4746	1.4074	1.4289	1.437	0.034	7.5626	7.7822	7.4663	7.604	0.162	4.621	4.5298	4.523	4.558	0.055
B184	1.04	1.15	1.04	1.077	0.064	5.81	6.44	6.21	6.153	0.319	3.53	3.66	3.91	3.700	0.193
B185															
B186	1.4	1.39	1.37	1.387	0.015	6.55	6.89	7.54	6.993	0.503	4.38	4.37	4.35	4.367	0.015
B187	1.8204	1.7228	1.829	1.791	0.059	8.6571	9.1799	9.6671	9.168	0.505	4.6825	4.7326	4.4229	4.613	0.166
B188															
B189	1.3466	1.2929	1.3427	1.327	0.030	6.4825	6.1449	6.1963	6.275	0.182	3.7786	3.8445	3.8384	3.821	0.036
B190	1.25	1.25	1.27	1.257	0.012	7.1	6.73	6.79	6.873	0.199	4.19	4.24	4.11	4.180	0.066
B192	1.3515	1.37006	1.35641	1.359	0.010	7.06146	7.1669	6.94876	7.059	0.109	4.11058	4.01303	4.05735	4.060	0.049
B193	1.53	1.83	1.47	1.610	0.193	7.72	7.4	6.99	7.370	0.366	3.5	3.4	3.47	3.457	0.051
B195	1.281	1.253	1.248	1.261	0.018	6.973	6.929	6.976	6.959	0.026	4.135	4.189	4.126	4.150	0.034
B196															
B198	1.33	1.31	1.29	1.310	0.020	6.56	6.52	7.56	6.880	0.589	3.91	3.84	3.91	3.887	0.040
B199	0.99	1	1	0.997	0.006	5.97	5.31	5.78	5.687	0.340	3.4	3.41	3.47	3.427	0.038
B200															
B202	1.23	1.22	1.18	1.210	0.026	6.07	6.12	6.12	6.103	0.029	3.65	3.7	3.6	3.650	0.050
B204	1.251	1.255	1.257	1.254	0.003	7.145	6.511	6.843	6.833	0.317	4.251	4.155	4.258	4.221	0.058
<b>B205</b>	<b>0.232</b>	<b>0.168</b>	<b>0.165</b>	<b>0.188</b>	<b>0.038</b>	<b>0.165</b>	<b>0.164</b>	<b>0.16</b>	<b>0.163</b>	<b>0.003</b>	<b>0.155</b>	<b>0.158</b>	<b>0.158</b>	<b>0.157</b>	<b>0.002</b>
B206	1.534	1.589	1.542	1.555	0.030	7.38	6.938	7.27	7.196	0.230	4.093	4.139	4.101	4.111	0.025
B208															
B210															
B212	1.469	1.466	1.454	1.463	0.008	7.39	7.278	6.899	7.189	0.257	4.253	4.269	4.244	4.255	0.013
B213	1.34	1.23	1.26	1.277	0.057	5.63	5.96	5.93	5.840	0.182	3.57	3.58	3.63	3.593	0.032
B215	1.2			1.200		6.3			6.300		3.7			3.700	
B216	1.416	1.394	1.363	1.391	0.027	7.433	7.465	7.511	7.470	0.039	4.56	4.545	4.461	4.522	0.053
B217	1.4	1.4	1.4	1.400	0.000	8	7.3	7.7	7.667	0.351	4.7	4.5	4.6	4.600	0.100
B219	1.48	1.49	1.51	1.493	0.015	8.02	8.34	8.5	8.287	0.244	4.92	4.85	4.68	4.817	0.123
B220															
B221	1.078	1.078	1.078	1.078		6.673	6.673	6.673	6.673		3.696	3.696	3.696	3.696	
<b>B222</b>	<b>1.02</b>	<b>1.08</b>	<b>1.04</b>	<b>1.047</b>	<b>0.031</b>	<b>2.11</b>	<b>2.33</b>	<b>2.72</b>	<b>2.387</b>	<b>0.309</b>	<b>3.35</b>	<b>3.4</b>	<b>3.43</b>	<b>3.393</b>	<b>0.040</b>
B223	1.29	1.28	1.31	1.293	0.015	6.77	7.11	7.23	7.037	0.239	4.22	4.35	4.27	4.280	0.066
B224	1.36	1.32	1.37	1.350	0.026	6.46	6.41	6.43	6.433	0.025	4	4.03	3.98	4.003	0.025
B226	1.39101	1.40711	1.37124	1.390	0.018	7.53359	7.13833		7.336	0.279	4.40071	4.41771	4.37844	4.399	0.020
B227	1.29	1.27	1.15	1.237	0.076	7.67	7.46	7.86	7.663	0.200	4.33	4.15	4.21	4.230	0.092
B228	1.232	1.188	1.199	1.206	0.023	6.264	5.572	5.499	5.778	0.422	3.457	3.182	3.415	3.351	0.148

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4				Plant Sample 6					
Target Consensus				1.460	0.080				8.11	0.42				4.21	0.46
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B230															
B233	0.917	1.168	1.088	1.058	0.128	5.402	4.864	4.767	5.011	0.342	3.27	2.973	2.987	3.077	0.168
B234	0.9	0.9	0.87	0.890	0.017	5.56	5.18	5.22	5.320	0.209	2.98	3.01	3.07	3.020	0.046

**Table B-10. Data summary table for CBDA in three marijuana samples.**

Target Consensus	Plant Sample 2					Plant Sample 3					Plant Sample 5				
	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Lab															
B046	14.11	14.31	14.04	14.15	0.14	11.73	11.06	11.42	11.40	0.34	12.63	12.53	12.53	12.56	0.06
B055	15.5282	15.2613	15.6005	15.46	0.18	14.5846	14.3802	14.5542	14.51	0.11	13.6403	13.7048	13.7829	13.71	0.07
B057	12.64	13.22	12.34	12.73	0.45	11.45	11.65	11.64	11.58	0.11	11.31	11.28	11.47	11.35	0.10
B062	14.188	14.503	14.871	14.52	0.34	14.181	13.998	13.869	14.02	0.16	12.538	13.132	12.977	12.88	0.31
B071	12.6	12.84	12.15	12.53	0.35	12.12	12.35	11.87	12.11	0.24	11.24	11.42	11.35	11.34	0.09
B099	12.8			12.80		12.5			12.50		11.2			11.20	
B109	11.785	11.84	11.767	11.80	0.04	10.635	11.116	11.083	10.94	0.27	10.669	10.527	10.344	10.51	0.16
B117	11.947	13.539	12.458	12.65	0.81	11.353	11.787	11.832	11.66	0.26	10.103	11.456	10.841	10.80	0.68
B125	12.46	12.076	12.073	12.20	0.22	11.938	11.67	11.577	11.73	0.19	11.201	10.647	10.928	10.93	0.28
B146	13.45	13.43	13.2	13.36	0.14	12.46	12.37	12.57	12.47	0.10	11.72	11.96	11.89	11.86	0.12
B154	11.791	12.654	10.022	11.49	1.34	11.639	11.662	11.423	11.57	0.13	10.856	10.478	10.662	10.67	0.19
B158	16.54	16.24	16.37	16.38	0.15	15.51	15.25	15.44	15.40	0.13	14.99	15.07	14.47	14.84	0.33
B167	13.43	13.6	13.33	13.45	0.14	12.71	12.45	12.46	12.54	0.15	11.79	11.58	11.7	11.69	0.11
B178	16.3	16	16	16.10	0.17	15.3	15.1	15.3	15.23	0.12	14.4	14.1	14.2	14.23	0.15
B198	10.72	12.39	10.39	11.17	1.07	11.39	11.27	11.47	11.38	0.10	10.46	10.73	10.51	10.57	0.14
B199	11.62	11.65	11.53	11.60	0.06	11.01	10.95	11.27	11.08	0.17	10.43	10.57	10.48	10.49	0.07
B213	11.5	12.4	12.2	12.03	0.47	11.3	11	10.9	11.07	0.21	10.6	10.2	10.3	10.37	0.21
B222	10.6	10.2	10.5	10.43	0.21	11.5	10.9	9.3	10.57	1.14	9.77	9.42	9.5	9.56	0.18

**Table B-11. Data summary table for total CBD in three hemp samples.**

Data in **red** font have been flagged as a data entry of zero or results that include text (e.g., “< LOQ” or “present”). Data in **blue** font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

*Note: This table spans multiple pages; the NIST values and consensus values are included on all pages for convenience.*

Target Consensus	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B001	1.81	1.8	1.71	1.773	0.055	6.2	6.57	6.67	6.480	0.248	3.91	3.85	3.88	3.880	0.030
B003	1.53203	1.45058	1.59756	1.527	0.074	5.61589	5.5848	5.51961	5.573	0.049	3.35072	3.30615	2.57609	3.078	0.435
B004	1.86	1.86	1.86	1.860	0.000	7.674	7.674	7.674	7.674	0.000	2.887	2.887	2.887	2.887	0.000
B005	1.836	1.811	1.551	1.733	0.158	7.014	7.059	6.529	6.867	0.294	4.18	4.175	4.175	4.177	0.003
B006	1.686	1.712	1.686	1.695	0.015	7.526	7.514	7.496	7.512	0.015	4.808	4.797	4.768	4.791	0.021
B007	1.92	1.93	1.93	1.927	0.006	7.77	7.61	7.28	7.553	0.250	4.5	4.48	4.46	4.480	0.020
B009															
B011	1.97	2.02	1.99	1.993	0.025	7.25	7.87	6.96	7.360	0.465	4.01	3.82	4.03	3.953	0.116
B012	1.5673	1.5724		1.570	0.004	6.4197	6.6		6.510	0.127	3.74	3.552		3.646	0.133
B013	1.66	1.65	1.66	1.657	0.006	6.11	6.22	6.36	6.230	0.125	4.25	4.23	4.17	4.217	0.042
B014															
B015	1.58	1.58	1.57	1.577	0.006	6.04	5.79	6.15	5.993	0.184	3.81	3.78	3.88	3.823	0.051
B016	2.3658	2.3588	2.3272	2.351	0.021	9.6138	9.6393	8.8713	9.375	0.436	5.7006	5.683	5.652	5.679	0.025
B018	1.73	1.66	1.66	1.683	0.040	7.23	6.65	7.97	7.283	0.662	4.36	4.33	4.33	4.340	0.017
B020															
B021															
B022	1.936	1.909	1.916	1.920	0.014	8.116	7.686	8.153	7.985	0.260	4.354	4.362	4.319	4.345	0.023
B023	1.741	1.7553	1.6942	1.730	0.032	7.0463	7.0198	6.9394	7.002	0.056	4.0645	4.1556	4.0624	4.094	0.053
B024	1.6	1.62	1.62	1.613	0.012	5.69	6.38	5.96	6.010	0.348	3.47	3.46	3.43	3.453	0.021
B026	1.91	2.01	1.88	1.933	0.068	8.71	8.47	8.54	8.573	0.123	5.04	5.27	4.84	5.050	0.215
B027	1.63	1.62	1.62	1.623	0.006	6.68	6.56	6.62	6.620	0.060	3.53	3.53	3.43	3.497	0.058
B028															
B029	1.476	1.502	1.418	1.465	0.043	6.656	6.835	6.081	6.524	0.394	3.655	3.769	3.707	3.710	0.057
B030	1.75	1.75	1.78	1.760	0.017	6.8	7.23	7	7.010	0.215	4.33	4.33	4.28	4.313	0.029
B031															
B033	1.04	1.28	1.58	1.300	0.271	4.73	6.14	6.65	5.840	0.995	3.39	4.32	4.1	3.937	0.486
B035	1.7411	1.75305	1.76071	1.752	0.010	6.66382	6.68462	6.62204	6.657	0.032	4.1181	4.17891	4.12874	4.142	0.032
B036	1.96	1.92	1.96	1.947	0.023	8.49	8.63	8.56	8.560	0.070	4.72	4.68	0.28	3.227	2.552
B037															
B041	1.681			1.681		7.143			7.143		4.313			4.313	
B042															
B043	1.65			1.650		6.924			6.924		4.222			4.222	
B044	1.86	1.87	1.78	1.837	0.049	6.87	6.65	6.3	6.607	0.287	3.6	3.62	3.72	3.647	0.064
B046	1.69	1.75	1.75	1.730	0.035	6.99	7.16	6.23	6.793	0.495	3.74	3.8	3.64	3.727	0.081
B047	1.959	1.951	1.882	1.931	0.042	7.144	6.956	6.763	6.954	0.191	4.376	4.633	4.447	4.485	0.133
B048															
B049	0.05417	0.05275	0.05126	0.053	0.001	0.32034	0.36757	0.38682	0.358	0.034	0.11411	0.19951	0.19933	0.171	0.049

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				1.82	0.12				7.72	0.36				3.93	0.40
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B051	1.71	1.64	1.54	1.630	0.085	7.17	7.21	6.28	6.887	0.526	4.04	3.97	3.59	3.867	0.242
B052	1.69	1.78	1.66	1.710	0.062	7.25	6.7	8.82	7.590	1.100	3.74	3.98	3.87	3.863	0.120
<b>B053</b>	<b>2.678</b>	<b>2.821</b>	<b>2.868</b>	<b>2.789</b>	<b>0.099</b>	4.801	4.896	5.015	4.904	0.107	3.682	3.889	3.89	3.820	0.120
B054	1.63331	1.64984	1.58853	1.624	0.032	7.19131	7.14237	6.71249	7.015	0.263	4.07676	4.06884	4.03341	4.060	0.023
B055	2.0437	2.0024	2.0153	2.020	0.021	7.7976	7.6939	7.9313	7.808	0.119	4.3435	4.1056	4.1095	4.186	0.136
B056	1.639	1.635	1.637	1.603	6.104	6.338			6.221	0.165	3.47	3.622		3.546	0.107
B058	1.78	1.75	1.72	1.750	0.030	7.06	6.88	6.49	6.810	0.291	4.11	4.15	3.98	4.080	0.089
B060	1.92	1.94	1.9	1.920	0.020	7.35	7.32	7.32	7.330	0.017	4.78	4.73	4.77	4.760	0.026
B061	1.67	1.747	1.763	1.727	0.050	6.684	6.63	6.892	6.735	0.138	3.951	3.976	3.912	3.946	0.032
B062	1.846	1.875	1.828	1.850	0.024	7.374	7.345	7.518	7.412	0.093	4.15	4.202	4.017	4.123	0.095
B063															
B064	1.973			1.973		7.122			7.122		4.11			4.110	
B065	1.52	1.7	1.7	1.640	0.104	6.56	7.51	7.16	7.077	0.480	4.59	4.52	4.2	4.437	0.208
B066	1.8665	1.8381	1.8878	1.864	0.025	8.1054	7.7301	7.6446	7.827	0.245	4.4587	4.5922	4.6474	4.566	0.097
B068															
B069	1.74	1.782	1.734	1.752	0.026	7.027	6.817	6.604	6.816	0.212	4.491	4.234	4.457	4.394	0.140
B070	1.636	1.852	1.747	1.745	0.108	7.039	6.597	6.683	6.773	0.234	4.23	4.122	4.293	4.215	0.086
B071	1.77	1.72	1.7	1.730	0.036	6.01	6.19	6.16	6.120	0.096	3.94	3.81	3.79	3.847	0.081
B073	1.5729			1.573		6.3896			6.390		4.1424			4.142	
B074	1.65	1.65	1.65	1.650	0.000	6.25	6.25	6.25	6.250	0.000	4.31	4.31	4.31	4.310	0.000
B076	1.71	1.76	1.75	1.740	0.026	8.916	7.202	7.671	7.930	0.886	3.837	3.754	3.834	3.808	0.047
B077	1.816	1.724	1.623	1.721	0.097	6.7	7.178	7.12	6.999	0.261	3.809	3.732	4.331	3.957	0.326
B078															
B079															
B081	1.82	1.69	1.62	1.710	0.101	6.79	6.89	6.36	6.680	0.282	3.97	3.63	3.48	3.693	0.251
B082	1.901	1.86	1.887	1.883	0.021	7.276	7.353	7.139	7.256	0.108	3.907	3.918	3.997	3.941	0.049
B084	1.49	1.52	1.51	1.507	0.015	6.4	6.37	6.38	6.383	0.015	3.76	3.83	3.8	3.797	0.035
B085	1.612	1.459	1.338	1.470	0.137	5.231	5.803	5.18	5.405	0.346	2.791	2.556	2.392	2.580	0.201
B086	1.748	1.716	1.669	1.711	0.040	6.004	6.086	6.423	6.171	0.222	3.961	3.946	3.865	3.924	0.052
B088	1.49	1.545	1.557	1.531	0.036	6.709	6.858	6.917	6.828	0.107	4.056	4.039	3.951	4.015	0.056
<b>B089</b>	<b>0.7823</b>	<b>0.8129</b>	<b>0.7978</b>	<b>0.798</b>	<b>0.015</b>	<b>2.81</b>	<b>2.8862</b>	<b>2.9968</b>	<b>2.898</b>	<b>0.094</b>	<b>1.9096</b>	<b>1.9066</b>	<b>1.9334</b>	<b>1.917</b>	<b>0.015</b>
B090	1.53	1.76	1.74	1.677	0.127	6.98	6.82	7.1	6.967	0.140	4.28	4.26	4.24	4.260	0.020
B091	1.506	1.493	1.491	1.497	0.008	7.24	6.142	6.232	6.538	0.610	3.761	3.798	3.741	3.767	0.029
B092	1.86	1.84	1.81	1.837	0.025	6.14	5.76	5.63	5.843	0.265	4.19	4.19	4.21	4.197	0.012
B094	1.64	1.61	1.73	1.660	0.062	6.52	6.99	6.49	6.667	0.280	4.06	3.89	3.9	3.950	0.095
B095	1.631	1.607	1.545	1.594	0.044	6.392	5.937	5.837	6.055	0.296	4.084	4.094	4.01	4.063	0.046
B096	1.767	1.715	1.704	1.729	0.034	6.437	6.952	6.983	6.791	0.307	4.409	4.283	4.327	4.340	0.064
<b>B098</b>						<b>2.33</b>	<b>2.96</b>	<b>2.31</b>	<b>2.533</b>	<b>0.370</b>	3.78	3.79	3.78	3.783	0.006
B099	1.844			1.844		6.69			6.690		3			3.000	
B100	1.867	1.735		1.801	0.093	7.596	7.095		7.346	0.354	4.674	4.568		4.621	0.075
B102	1.7144	1.651	1.6536	1.673	0.036	6.6053	6.5238	6.6356	6.588	0.058	4.1699	4.1897	4.0845	4.148	0.056
B104	1.597	1.576	1.594	1.589	0.011	6.652	6.812	6.836	6.767	0.100	3.786	3.835	3.842	3.821	0.031
B106	1.733	1.722	1.714	1.723	0.010	6.996	6.683	6.832	6.837	0.157	3.979	3.942	3.955	3.959	0.019

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				1.82	0.12				7.72	0.36				3.93	0.40
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B108															
B109	1.55	1.505	1.488	1.514	0.032	6.252	6.315	5.531	6.033	0.436	3.678	3.654	3.704	3.679	0.025
B110	1.71	1.6	1.58	1.630	0.070	6.9	6.46	6.66	6.673	0.220	3.23	3.24	3.58	3.350	0.199
B111															
B113	1.69	1.69	1.66	1.680	0.017	7.08	6.22	6.58	6.627	0.432	4.18	4.63	4.61	4.473	0.254
B114															
B115	1.86	1.91	2.02	1.930	0.082	6.19	6.13	6.25	6.190	0.060	4.265	4.266	4.2498	4.260	0.009
B116	1.6396	1.6257	1.652	1.639	0.013	6.887	7.068	6.553	6.836	0.261	3.51511	3.661	3.5045	3.560	0.087
B117	1.337	1.537	1.614	1.496	0.143	5.907	6.371	6.277	6.185	0.245	4.474	3.853	4.249	4.192	0.314
B120															
B122															
B124	1.618	1.758	1.7147	1.697	0.072	7.653	7.872	7.173	7.566	0.358	3.796	4.17	3.812	3.926	0.211
B125	1.532	1.534	1.509	1.525	0.014	6.206	6.012	6.042	6.087	0.104	3.544	3.561	3.39	3.498	0.094
B126	1.731	1.748	1.674	1.718	0.039	7.016	6.78	7.322	7.039	0.272	4.438	4.295	4.214	4.316	0.113
B127	1.7988	1.8245	1.7646	1.796	0.030	7.0726	7.5812	7.6683	7.441	0.322	4.74	4.8279	4.8666	4.812	0.065
B129															
B131	1.8285	1.7851	1.7173	1.777	0.056	7.6321	8.2777	7.5005	7.803	0.416	4.0062	4.0441	4.0459	4.032	0.022
<b>B132</b>	<b>2.413</b>	<b>2.32</b>	<b>2.347</b>	<b>2.360</b>	<b>0.048</b>	7.996	7.321	6.586	7.301	0.705	4.895	4.908	4.792	4.865	0.064
B136															
B137	1.517	1.415	1.501	1.478	0.055	6.274	6.319	6.431	6.341	0.081	3.788	4.148	3.876	3.937	0.188
B141	1.489	1.05	1.446	1.328	0.242	5.29	6.263	6.075	5.876	0.516	3.88	3.85	3.99	3.907	0.074
B142	1.774	1.747	1.721	1.747	0.027	7.344	6.766	6.78	6.963	0.330	4.162	4.209	4.067	4.146	0.072
<b>B144</b>	<b>1.79</b>	<b>1.73</b>	<b>1.92</b>	<b>1.813</b>	<b>0.097</b>	<b>9.65</b>	<b>9.65</b>	<b>8.94</b>	<b>9.413</b>	<b>0.410</b>	<b>7.14</b>	<b>5.91</b>	<b>5.83</b>	<b>6.293</b>	<b>0.734</b>
B146	1.56	1.54	1.57	1.557	0.015	5.89	6.99	6.88	6.587	0.606	3.7	3.76	3.74	3.733	0.031
B147	1.567	1.594	1.554	1.572	0.020	6.502	6.401	6.308	6.404	0.097	4.001	3.999	4.091	4.030	0.053
B148															
B149	1.653	1.49	1.43	1.524	0.115	6.8	6.77	6.89	6.820	0.062	3.9	3.95	4.07	3.973	0.087
B152															
B153	1.942	1.984	1.974	1.967	0.022	7.629	7.547	7.915	7.697	0.193	4.77	4.586	4.997	4.784	0.206
B154	1.501	1.574	1.434	1.503	0.070	5.517	6.013	6.96	6.163	0.733	3.894	4.044	4.285	4.074	0.197
B157	1.66			1.660		6.66			6.660		3.91	3.88		3.895	0.021
B158	2.13	1.97	1.96	2.020	0.095	8.21	7.81	7.56	7.860	0.328	4.28	4.52	4.6	4.467	0.167
B159	1.871	1.8232	1.791	1.828	0.040	6.8473	7.0936	6.9473	6.963	0.124	3.9999	4.3366	4.2117	4.183	0.170
B160	1.69829	1.63862	1.61654	1.651	0.042	6.85166	6.20033	6.69729	6.583	0.340	4.01845	4.17553	4.32472	4.173	0.153
B161	2.01	2	2.04	2.017	0.021	8.53	8.05	8.22	8.267	0.243	4.9	4.99	4.94	4.943	0.045
B163	2.52	2.05	2.04	2.203	0.274	8.09	7.43	5.78	7.100	1.190	5.32	5.16	5.23	5.237	0.080
B164	1.55	1.69	1.69	1.643	0.081	4.7	6.11	6.61	5.807	0.990	4.06	4.16	4.12	4.113	0.050
B165															
B166	1.18	1.16	1.17	1.170	0.010	6.85	7.6	7.9	7.450	0.541	4.31	4.28	4.31	4.300	0.017
B167															
B168	1.72	1.74	1.71	1.723	0.015	6.62	6.57	6.21	6.467	0.224	4.35	4.3	4.29	4.313	0.005
B170															
B172	1.48049	1.34401	1.38113	1.402	0.071	5.83578	5.5485	5.31293	5.566	0.262	3.1126	3.26636	3.22638	3.202	0.080

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				1.82	0.12				7.72	0.36				3.93	0.40
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B173	1.69354	1.77532	1.67665	1.715	0.053	7.22676	7.38173	7.55687	7.388	0.165	4.37104	4.12682	4.04924	4.182	0.168
B174	1.71	1.71	1.75	1.723	0.023	6.67	6.83	7.48	6.993	0.429	4.12	4.1	4.24	4.153	0.076
B175	2.29	2.16	2.14	2.197	0.081	6.68	6.81	7.05	6.847	0.188	3.75	3.73	3.72	3.733	0.015
B176	1.602	1.571	1.582	1.585	0.016	6.036	6.438	6.336	6.270	0.209	3.581	3.846	3.81	3.746	0.144
B178	1.97	1.96	1.88	1.937	0.049	8.16	8.36	8.09	8.203	0.140	5.29	5.07	5.16	5.173	0.111
B181	1.5			1.500		8.64			8.640		4.96			4.960	
B182	1.33	1.36		1.345	0.021	6.19	6.52	6.54	6.417	0.197	3.76	3.88	3.79	3.810	0.062
B183	1.8279	1.7661	1.7744	1.789	0.034	7.1948	7.4218	7.1046	7.240	0.163	4.3252	4.2336	4.2335	4.264	0.053
B183	1.8279	1.7661	1.7744	1.789	0.034	7.1948	7.4218	7.1046	7.240	0.163	4.3252	4.2336	4.2335	4.264	0.053
B184	1.33	1.45	1.32	1.367	0.072	5.6	6.23	5.99	5.940	0.318	3.36	3.48	3.72	3.520	0.183
B185															
B186	1.72	1.69	1.69	1.700	0.017	6.21	6.54	7.16	6.637	0.482	4.04	4.02	4.02	4.027	0.012
B187	2.1562	2.0375	2.1564	2.117	0.069	8.1406	8.6397	9.0957	8.625	0.478	4.3344	4.3795	4.0918	4.269	0.155
B188															
B189	1.7022	1.6512	1.6971	1.684	0.028	6.2587	5.9236	5.9798	6.054	0.179	3.5755	3.6363	3.632	3.615	0.034
B190	1.61	1.6	1.61	1.607	0.006	6.82	6.46	6.51	6.597	0.195	3.95	4	3.88	3.943	0.060
B192	1.71743	1.73965	1.72402	1.727	0.011	6.76779	6.87372	6.66286	6.768	0.105	3.86992	3.78043	3.81943	3.823	0.045
B193	1.94	2.29	1.87	2.033	0.225	7.37	7.13	6.69	7.063	0.345	3.31	3.21	3.3	3.273	0.055
B195	1.625	1.597	1.587	1.603	0.020	6.592	6.547	6.586	6.575	0.024	3.867	3.919	3.857	3.881	0.033
B196															
B197	16.4	16.7	16.7	16.600	0.173	13.7	14.3	13.7	13.900	0.346	15.2	15	14.7	14.967	0.252
B198	1.66	1.64	1.64	1.647	0.012	6.34	6.31	7.29	6.647	0.557	3.71	3.62	3.71	3.680	0.052
B199	1.31	1.31	1.31	1.310		5.75	5.12	5.55	5.473	0.322	3.21	3.22	3.27	3.233	0.032
B200															
B202	1.54	1.54	1.51	1.530	0.017	5.86	5.9	5.91	5.890	0.026	3.41	3.45	3.36	3.407	0.045
B204	1.624	1.623	1.631	1.626	0.004	6.881	6.264	6.584	6.576	0.309	4.003	3.923	4.017	3.981	0.051
B206	1.905	1.965	1.914	1.928	0.032	7.088	6.658	6.985	6.910	0.225	3.874	3.915	3.881	3.890	0.022
B208															
B210															
B211															
B212	1.851	1.843	1.828	1.841	0.012	7.073	6.967	6.6	6.880	0.248	4	4.017	3.992	4.003	0.013
B213	1.64	1.6	1.7	1.647	0.050	5.56	5.85	5.83	5.747	0.162	3.44	3.45	3.47	3.453	0.015
B215	1.51			1.510		6.1			6.100		3.5			3.500	
B216	1.812	1.739	1.7	1.750	0.057	7.125	7.189	7.254	7.189	0.065	4.288	4.276	4.197	4.254	0.049
B217	1.7	1.8	1.7	1.733	0.058	7.6	7	7.3	7.300	0.300	4.4	4.2	4.3	4.300	0.100
B219	1.82	1.83	1.86	1.837	0.021	7.65	7.97	8.1	7.907	0.232	4.6	4.55	4.4	4.517	0.104
B220															
B221															
B222	1.32	1.41	1.34	1.357	0.047	2.09	2.35	2.58	2.340	0.245	3.19	3.25	3.24	3.227	0.032
B223	1.63	1.62	1.64	1.630	0.010	6.49	6.82	6.93	6.747	0.229	3.97	4.09	4.02	4.027	0.060
B224	1.75	1.72	1.76	1.743	0.021	6.46	6.73	6.41	6.533	0.172	3.81	3.82	3.79	3.807	0.015
B226	1.71043	1.739	1.69716	1.716	0.021	7.21259	6.86435		7.038	0.246	4.14068	4.16174	4.12692	4.143	0.018
B227	1.61	1.6	1.46	1.557	0.084	7.35	7.02	7.41	7.260	0.210	4.01	3.85	3.9	3.920	0.082

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4				Plant Sample 6					
Target Consensus				1.82	0.12				7.72	0.36				3.93	0.40
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B228	1.593	1.541	1.542	1.559	0.030	6.094	5.442	5.349	5.628	0.406	3.304	3.063	3.262	3.210	0.129
B230															
B233	1.286	1.538	1.437	1.420	0.127	5.937	5.374	5.363	5.558	0.328	3.597	3.194	3.277	3.356	0.213
B234	1.24	1.24	1.21	1.230	0.017	6.06	5.67	5.71	5.813	0.215	3.2	3.22	3.29	3.237	0.047

**Table B-12. Data summary table for total CBD in three marijuana samples.**

Data in blue font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

Target Consensus	Plant Sample 2					Plant Sample 3					Plant Sample 5				
	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg
Lab	A	B	C			A	B	C			A	B	C		
B042															
B046	13.7	13.91	13.63	13.75	0.15	11.48	10.89	11.21	11.19	0.30	12.55	12.47	12.46	12.49	0.05
B055	15.1197	14.9155	15.1846	15.07	0.14	14.3623	14.1616	14.3015	14.28	0.10	13.6176	13.7261	13.763	13.70	0.08
B062	13.86	14.129	14.402	14.13	0.27	13.927	13.743	13.592	13.75	0.17	12.536	13.081	12.925	12.85	0.28
B071	12.41	12.64	12	12.35	0.32	12.04	12.26	11.86	12.05	0.20	11.38	11.56	11.49	11.48	0.09
B079															
B086	12.475	12.571	12.776	12.61	0.15	11.851	12.267	12.295	12.14	0.25	11.768	11.925	11.748	11.81	0.10
B099	12.4			12.40		12.2			12.20		11.16				11.16
B109	11.518	11.574	11.515	11.54	0.03	10.571	10.975	10.945	10.83	0.23	10.696	10.563	10.385	10.55	0.16
B117	11.569	13.081	11.979	12.21	0.78	10.97	11.339	11.443	11.25	0.25	9.878	11.215	10.575	10.56	0.67
B125	12.102	11.748	11.74	11.86	0.21	11.717	11.462	11.371	11.52	0.18	11.16	11.61	10.891	11.22	0.36
B146	13.08	13.06	12.86	13.00	0.12	12.25	12.16	12.35	12.25	0.10	11.68	11.9	11.85	11.81	0.12
B148															
B154	11.44	12.283	9.718	11.15	1.31	11.397	11.416	11.187	11.33	0.13	10.791	10.407	10.594	10.60	0.19
B158	15.99	15.68	15.79	15.82	0.16	15.11	14.85	15.05	15.00	0.14	14.81	14.86	14.32	14.66	0.30
B167	13.09	13.25	12.99	13.11	0.13	12.5	12.26	12.25	12.34	0.14	11.77	11.55	11.67	11.66	0.11
B170															
<b>B178</b>	15.8	15.5	15.4	15.57	0.21	<b>1.54</b>	<b>1.47</b>	<b>1.48</b>	<b>1.50</b>	<b>0.04</b>	14.2	13.9	14	14.03	0.15
B196															
B198	10.58	12.18	10.32	11.03	1.01	11.32	11.25	11.45	11.34	0.10	10.61	10.85	10.64	10.70	0.13
B199	11.35	11.38	11.26	11.33	0.06	10.83	10.79	11.08	10.90	0.16	10.44	10.58	10.49	10.50	0.07
B210															
B213	11.5	12.4	12.2	12.03	0.47	11.4	11.2	10.9	11.17	0.25	10.8	10.4	10.6	10.60	0.20
B222	10.4	10	10.3	10.23	0.21	11.3	10.7	9.31	10.44	1.02	9.76	9.5	9.63	9.63	0.13

**Table B-13. Data summary table for CBC in three hemp samples.**

Data in **red** font have been flagged as a data entry of zero or results that include text (e.g., “< LOQ” or “present”).

Data in **blue** font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

*Note: This table spans multiple pages; the NIST values and consensus values are included on all pages for convenience.*

Target Consensus	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B001	0.031	0.032	0.034	0.0323	0.0015	0.087	0.091	0.114	0.0973	0.0146	0.029	0.029	0.028	0.02867	0.00058
B003															
B004	0.03	0.03	0.03	0.0300	0.0000	0.052	0.052	0.052	0.0520	0.0000	0.013	0.013	0.013	0.01300	0.00000
B005	< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1		
B006	0.028	0.029	0.028	0.0283	0.0005	0.055	0.055	0.052	0.0540	0.0017	< 0.012	< 0.012	< 0.012		
B007	0.03	0.03	0.03	0.0300	0.0000	0.07	0.07	0.07	0.0700	0.0000	0.03	0.03	0.03	0.03000	0.00000
B009															
B013	0.03	0.04	0.04	0.0366	0.0057	0.06	0.05	0.05	0.0533	0.0058	< 0.028	< 0.028	< 0.028		
B015	0.02	0.02	0.03	0.0233	0.0057	0.05	0.04	0.05	0.0467	0.0058	0.02	0.02	0.02	0.02000	0.00000
B016	< 0.0004	< 0.0004	< 0.0004			0.0784	0.0978	0.0698	0.0820	0.0143	< 0.0004	< 0.0004	< 0.0004		
B018	0.02	0.02	0.02	0.0200	0.0000	0.05	0.04	0.05	0.0467	0.0058	0.02	0.02	0.02	0.02000	0.00000
B020															
B021															
B022	< 0.04	< 0.04	< 0.04			0.068	0.062	0.065	0.0650	0.0030	< 0.04	< 0.04	< 0.04		
B023	0.028	0.0293	0.028	0.0284	0.0007	0.0574	0.0572	0.0565	0.0570	0.0005	0.0256	0.0262	0.0261	0.02597	0.00032
B024	0.02	0.03	0.01	0.0200	0.0100	0.04	0.04	0.02	0.0333	0.0115	0.01	0.02	0.01	0.01333	0.00577
B026	< 0.01	< 0.01	< 0.01			0.03	0.03	0.03	0.0300	0.0000	< 0.01	< 0.01	< 0.01		
B027	0.0297	0.0235	0.0261	0.0264	0.0031	0.0608	0.0602	0.0613	0.0608	0.0006	0.0293	0.0301	0.032	0.03047	0.00139
B028															
B029	< 0.030	< 0.030	< 0.030			0.059	0.054	0.047	0.0533	0.0060	< 0.030	< 0.030	< 0.030		
B030	0.03	0.03	0.03	0.0300	0.0000	0.05	0.05	0.05	0.0500	0.0000	0.02	0.02	0.02	0.02000	0.00000
B031															
B032															
B033	< 0.5	< 0.5	< 0.5			0.06	0.05	0.06	0.0567	0.0058	< 0.5	< 0.5	< 0.5		
B035	0.02979	0.03021	0.03034	0.0301	0.0002	0.05337	0.0536	0.05303	0.0533	0.0003	0.0241	0.02467	0.02422	0.02433	0.00030
B036	< 0.02	< 0.02	< 0.02			< 0.05	< 0.05	< 0.05			< 0.02	< 0.02	< 0.02		
B037															
B038															
B041	< 0.064					< 0.064					< 0.064				
B043	0.028			0.0280		0.053			0.0530		0.023			0.02300	
B044	< 0.075	< 0.075	< 0.075			< 0.075	< 0.075	< 0.075			< 0.075	< 0.075	< 0.075		
B047	0.036	0.031	0.033	0.0333	0.0025	0.059	0.06	0.052	0.0570	0.0044	0.027	0.027	0.029	0.02767	0.00115
B048															
B049	0.02531	0.02303	0.02305	0.0238	0.0013	0.04874	0.05344	0.05591	0.0527	0.0036	0.01419	0.02331	0.02386	0.02045	0.00543
B051	0.02	0.02	0.03	0.0233	0.0057	0.05	0.07	0.07	0.0633	0.0115	0.00	0.00	0.00	0.00000	0.00000
B052	< 0.10	< 0.10	< 0.10			< 0.10	< 0.10	< 0.10			< 0.10	< 0.10	< 0.10		
B053	0.114	0.116	0.12	0.1166	0.0031	0.205	0.201	0.215	0.2070	0.0072	0.097	0.1	0.1	0.09900	0.00173

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.0325	0.0084				0.0522	0.0042				0.0223	0.0028
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B054	0.0273	0.02791	0.02625	0.0271	0.0008	0.05308	0.05477	0.05051	0.0528	0.0021	0.01964	0.01902	0.01966	0.01944	0.00037
B055	0.0367	0.036	0.0351	0.0359	0.0008	0.0596	0.056	0.059	0.0582	0.0019	0.0227	0.0221	0.022	0.02227	0.00038
<b>B056</b>	<b>0.094</b>	<b>0.094</b>		<b>0.0940</b>	<b>0.0000</b>	<b>0.383</b>	<b>0.383</b>		<b>0.3830</b>	<b>0.0000</b>	<b>0.203</b>	<b>0.214</b>		<b>0.2085</b>	<b>0.0078</b>
B058	0.0302	0.032		0.0311	0.0013	0.0638	0.0669	0.0589	0.0632	0.0040	0.0291			0.0291	
B060	0.03	0.04	0.04	0.0367	0.0058	0.06	0.04	0.07	0.0567	0.0153					
B061	0.031	0.031	0.034	0.0320	0.0017	0.058	0.059	0.062	0.0597	0.0021	0.027	0.028	0.027	0.0273	0.0006
B062	< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08		
B063															
<b>B064</b>	<b>&lt;0.0001</b>					<b>&lt;0.0001</b>					<b>0.00001</b>			<b>0.0000</b>	
B065	0.038	0.039	0.038	0.0383	0.0006	0.062	0.064	0.064	0.0633	0.0012	0.028	0.029	0.027	0.0280	0.0010
B066	0.0304	0.0326	0.0304	0.0311	0.0013	0.0598	0.0567	0.0596	0.0587	0.0017	0.0226	0.0245	0.0226	0.0232	0.0011
B068															
B069	0.03	0.03	0.03	0.0300	0.0000	0.05	0.06	0.06	0.0567	0.0058	0.03	0.03		0.0300	0.0000
B070	0.026	0.033	0.027	0.0287	0.0038	<0.111	<0.111	<0.111			<0.067	<0.067	<0.067		
B072	0.032	0.032	0.032	0.0320	0.0000	0.058	0.059	0.056	0.0577	0.0015	0.024	0.026	0.024	0.0247	0.0012
B073	0.0268					0.0268		0.0459			0.0459		0.0204		0.0204
B074	0.02	0.02	0.02	0.0200	0.0000	0.05	0.05	0.05	0.0500	0.0000	0.03	0.03	0.03	0.0300	0.0000
B076	0.03164	0.03195	0.03183	0.0318	0.0002	0.05769	0.05775	0.05765	0.0577	0.0000	0.02233	0.02231	0.02232	0.0223	0.0000
B077															
B078															
B079															
B081	<0.15	<0.15	<0.15			<0.15	<0.15	<0.15			<0.15	<0.15	<0.15		
B082	0.03	0.03	0.03	0.0300	0.0000	0.047	0.051	0.05	0.0493	0.0021	0.018	0.016	0.018	0.0173	0.0012
<b>B084</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.0000</b>	<b>0.0000</b>	<b>0.06</b>	<b>0.05</b>	<b>0.055</b>	<b>0.0550</b>	<b>0.0050</b>	<b>0.03</b>	<b>0.03</b>	<b>0.03</b>	<b>0.0300</b>	<b>0.0000</b>
B085	0.037	0.04	0.034	0.0370	0.0030	0.067	0.08	0.067	0.0713	0.0075	0.024	0.027	0.023	0.0247	0.0021
B088	0.032	0.03	0.038	0.0333	0.0042	0.047	0.049	0.05	0.0487	0.0015	0.02	0.021	0.018	0.0197	0.0015
B090	<0.05	<0.05	<0.05			0.07	0.07	0.07	0.0700	0.0000	<0.05	<0.05	<0.05		
B091	0.021	0.021	0.021	0.0210	0.0000	0.032	0.028	0.027	0.0290	0.0026	0.01	0.012	0.01	0.0107	0.0012
B094	0.03	0.03	0.03	0.0300	0.0000	0.06	0.07	0.07	0.0667	0.0058	0.03	0.03	0.03	0.0300	0.0000
B095	0.035	0.034	0.034	0.0343	0.0006	0.065	0.063	0.067	0.0650	0.0020	0.034	0.034	0.035	0.0343	0.0006
B096	0.033	0.033	0.032	0.0327	0.0006	0.054	0.065	0.057	0.0587	0.0057	0.031	0.025	0.023	0.0263	0.0042
B097	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B098						0.09	0.11	0.08	0.0933	0.0153					
<b>B099</b>	<b>0.048</b>			<b>0.0480</b>		0.067			0.0670		0.037			0.0370	
B100	0.03	0.03		0.0300		0.06	0.05		0.0550	0.0071	<0.02	<0.02			
B102	0.029	0.0281	0.0277	0.0283	0.0007	0.0478	0.0495	0.0504	0.0492	0.0013	0.0214	0.0216	0.0212	0.0214	0.0002
B106	0.029	0.029	0.029	0.0290		0.055	0.051	0.052	0.0527	0.0021	0.023	0.023	0.022	0.0227	0.0006
B108															
<b>B109</b>	<b>0.028</b>	<b>0.027</b>	<b>0.026</b>	<b>0.0270</b>	<b>0.0010</b>	<b>0.213</b>	<b>0.213</b>	<b>0.184</b>	<b>0.2033</b>	<b>0.0167</b>	<b>0.113</b>	<b>0.115</b>	<b>0.116</b>	<b>0.1147</b>	<b>0.0015</b>
B110	0.03	0.03	0.03	0.0300		0.06	0.06	0.06	0.0600		0.03	0.03	0.03	0.0300	
B111															
B114															
B115	0.034	0.037	0.038	0.0363	0.0021	0.0585	0.0591	0.0613	0.0596	0.0015	0.0308	0.027	0.028	0.0286	0.0020

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.0325	0.0084				0.0522	0.0042				0.0223	0.0028
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B116	0.0283	0.0282	0.0286	0.0284	0.0002	0.0517	0.0517	0.0485	0.0506	0.0018	0.0186	0.0188	0.0191	0.0188	0.0003
B117	0.027	0.029	0.029	0.0283	0.0012	0.108	0.113	0.112	0.1110	0.0026	0.059	0.057	0.065	0.0603	0.0042
B120															
B124	0.031	0.03	0.031	0.0307	0.0006	0.061	0.061	0.059	0.0603	0.0012	0.028	0.026	0.026	0.0267	0.0012
B125	0.031	0.031	0.029	0.0303	0.0012	0.059	0.059	0.055	0.0577	0.0023	0.025	0.027	0.024	0.0253	0.0015
B126															
B129															
B130	0.03	0.03	0.03	0.0300		0.05	0.05	0.05	0.0500		0.02	0.02	0.02	0.0200	
B132	0.051	0.049	0.05	0.0500	0.0010	0.084	0.082	0.069	0.0783	0.0081	0.032	0.033	0.031	0.0320	0.0010
B137	0.029	0.029	0.031	0.0297	0.0012	0.041	0.033	0.04	0.0380	0.0044	0.017	0.017	0.017	0.0170	
B141	0.027	0.026	0.026	0.0263	0.0006	0.042	0.056	0.043	0.0470	0.0078	0.025	0.024	0.022	0.0237	0.0015
B142	<0.0007	<0.0007	<0.0007			<0.0075	<0.0075	<0.0075			0.025	0.024	0.022	0.0237	0.0015
B144	0.05	0.05	0.05	0.0500		0.07	0.07	0.068	0.0693	0.0012	0.038	0.039	0.037	0.0380	0.0010
B146	0.0273	0.0272	0.0272	0.0272	0.0001	0.0418	0.048	0.0476	0.0458	0.0035	0.0185	0.0191	0.0191	0.0189	0.0003
B147	<0.016	<0.016	<0.016			0.045	0.042	0.041	0.0427	0.0021	<0.016	<0.016	<0.016		
B148															
B149	<0.015	0.021	0.024	0.0225	0.0021	<0.015	0.044	0.047	0.0455	0.0021	<0.015	<0.015	<0.015		
B152															
B153	0.035	0.038	0.033	0.0353	0.0025	0.076	0.062	0.061	0.0663	0.0084	0.029	0.025	0.025	0.0263	0.0023
B154	0.029	0.034	0.026	0.0297	0.0040	0.052	0.056	0.065	0.0577	0.0067	0.031	0.031	0.021	0.0277	0.0058
B157	<0.05					0.06			0.0600		<0.05	<0.05			
B158	<0.05	<0.05	<0.05			0.06	0.06	0.06	0.0600	0.0000	<0.05	<0.05	<0.05		
B159	0.0298	0.0298	0.0294	0.0297	0.0002	0.0571	0.0547	0.0547	0.0555	0.0014	0.024	0.0247	0.0251	0.0246	0.0006
B160	0.03658	0.03409	0.0351	0.0353	0.0013	0.04951	0.04604	0.04858	0.0480	0.0018	0.02083	0.02205	0.02247	0.0218	0.0009
B161	0	0	0	0.0000		0.023	0.025	0.025	0.0243	0.0012	0	0.019	0.102	0.0403	0.0542
B163	0.0419	0.037	0.0369	0.0386	0.0029	0.0634	0.0611	0.0455	0.0567	0.0097	0.0302	0.0288	0.0282	0.0291	0.0010
B164	0.32	0.3	0.29	0.3033	0.0153	0.29	0.31	0.3	0.3000	0.0100	0.3	0.29	0.27	0.2867	0.0153
B165															
B167															
B172	0.03664	0.03336	0.03432	0.0348	0.0017	0.06744	0.07488	0.0624	0.0682	0.0063	0.01672	0.01808	0.02024	0.0183	0.0018
B173	0.02838	0.029	0.02782	0.0284	0.0006	0.0538	0.05237	0.05541	0.0539	0.0015	0.02162	0.01992	0.02101	0.0208	0.0009
B174	<0.06	<0.06	<0.06			<0.06	<0.06	<0.06			<0.06	<0.06	<0.06		
B175	<0.05	<0.05	<0.05			0.05	0.05	0.05	0.0500		<0.05	<0.05	<0.05		
B178	0.0359	0.0343	0.0333	0.0345	0.0013	0.0633	0.0675	0.0648	0.0652	0.0021	0.0329	0.0303	0.0313	0.0315	0.0013
B181	0.03					0.04			0.0400		0.02			0.0200	
B182	<0.05	<0.05				<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B184	<0.025	<0.025	<0.025			<0.025	0.033	0.031	0.0320	0.0014	<0.025	<0.025	<0.025		
B185															
B186	0.031	0.03	0.031	0.0307	0.0006	0.053	0.056	0.059	0.0560	0.0030	0.026	0.026	0.027	0.0263	0.0006
B187	<0.06	<0.06	<0.06			0.0742	0.0763	0.0818	0.0774	0.0039	<0.06	<0.06	<0.06		
B188															
B189	0.04654	0.04225	0.0489	0.0459	0.0034	0.06919	0.06608	0.0694	0.0682	0.0019	0.04326	0.03948	0.03942	0.0407	0.0022
B190	0.03	0.03	0.03	0.0300		0.06	0.06	0.06	0.0600		0.03	0.03	0.03	0.0300	

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4				Plant Sample 6					
Target Consensus				0.0325	0.0084				0.0522	0.0042				0.0223	0.0028
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B192	0.03838	0.0399	0.03953	0.0393	0.0008	0.06465	0.06981	0.06681	0.0671	0.0026	0.03757	0.03361	0.033	0.0347	0.0025
B193	<0.105	<0.0951	<0.112			<0.108	<0.0998	<0.106			<0.114	<0.117	<0.113		
B195	0.035	0.042	0.039	0.0387	0.0035	0.063	0.065	0.068	0.0653	0.0025	0.025	0.027	0.029	0.0270	0.0020
B196															
B198	< 0.07	< 0.07	< 0.07			<0.07	<0.07	<0.07			<0.07	<0.07	<0.07		
B200															
B202	0.027	0.0268	0.0267	0.0268	0.0002	0.0504	0.0488	0.0496	0.0496	0.0008	0.0174	0.0171	0.0178	0.0174	0.0004
B204	0.029	0.029	0.03	0.0293	0.0006	0.053	0.048	0.051	0.0507	0.0025	0.021	0.021	0.022	0.0213	0.0006
<b>B205</b>	0.042	0.033	0.033	0.0360	0.0052	<b>0.262</b>	<b>0.271</b>	<b>0.273</b>	<b>0.2687</b>	<b>0.0059</b>	<b>0.12</b>	<b>0.132</b>	<b>0.13</b>	<b>0.1273</b>	<b>0.0064</b>
B206	0.036	0.037	0.036	0.0363	0.0006	0.055	0.051	0.054	0.0533	0.0021	0.022	0.022	0.021	0.0217	0.0006
B208															
B210															
B212	0.034	0.034	0.034	0.0340		0.061	0.06	0.057	0.0593	0.0021	0.029	0.029	0.029	0.0290	
B213	0.03	0.03	0.03	0.0300	0.0000	0.06	0.06	0.05	0.0567	0.0058	< 0.03	< 0.03	< 0.03		
B215	0.025			0.0250		0.047			0.0470		0.018			0.0180	
B216	<0.037	<0.037	<0.037			0.054	0.064	0.065	0.0610	0.0061	<0.037	<0.037	<0.037		
B217	0.03	0.03	0.03	0.0300	0.0000	0.04	0.04	0.04	0.0400	0.0000	0.02	0.02	0.02	0.0200	0.0000
B219	0.0316	0.0329	0.0321	0.0322	0.0007	0.0606	0.0625	0.0614	0.0615	0.0010	0.025	0.0238	0.0249	0.0246	0.0007
B220															
<b>B221</b>	<b>0.058</b>	<b>0.055</b>	<b>0.056</b>	<b>0.0563</b>	<b>0.0015</b>	<b>0.096</b>	<b>0.082</b>	<b>0.109</b>	<b>0.0957</b>	<b>0.0135</b>	0.036	0.045	0.042	0.0410	0.0046
B222	<0.04	<0.04	<0.04			<0.04	<0.04	<0.04			<0.05	<0.05	<0.05		
B223	0.0288	0.0293	0.0304	0.0295	0.0008	0.0576	0.0597	0.0614	0.0596	0.0019	0.0274	0.0283	0.0275	0.0277	0.0005
B224	0.03	0.03	0.03	0.0300		0.05	0.05	0.05	0.0500		0.02	0.03	0.02	0.0233	0.0058
B226	0.03015	0.0305	0.02742	0.0294	0.0017	0.05107	0.05263		0.0518	0.0011	0.02158	0.02075	0.0215	0.0213	0.0005
B228	0.04	0.039	0.039	0.0393	0.0006	0.072	0.067	0.063	0.0673	0.0045	0.036	0.038	0.037	0.0370	0.0010
B230															
B234	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		

**Table B-14. Data summary table for CBC in three marijuana samples.**

Data in blue font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

Target Consensus	Plant Sample 2					Plant Sample 3					Plant Sample 5				
	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B032				0.1181	0.0072				0.1207	0.0034				0.121	0.046
B055	0.1294	0.1276	0.1283	0.121	0.022	0.1394	0.1349	0.1354	0.1366	0.0025	0.1512	0.1504	0.1504	0.1507	0.0005
B062	0.128	0.134	0.133	0.1317	0.0032	0.138	0.142	0.135	0.1383	0.0035	0.149	0.155	0.149	0.1510	0.0035
B079				0.1260		0.133			0.1330		0.151			0.1510	
B099	0.126														
B109	0.379	0.365	0.359	0.3677	0.0103	0.362	0.343	0.357	0.3540	0.0098	0.343	0.313	0.313	0.3230	0.0173
B117	0.212	0.241	0.223	0.2253	0.0146	0.211	0.22	0.219	0.2167	0.0049	0.201	0.22	0.211	0.2107	0.0095
B125	0.129	0.125	0.124	0.1260	0.0026	0.137	0.133	0.131	0.1337	0.0031	0.149	0.141	0.144	0.1447	0.0040
B146	0.105	0.104	0.104	0.1043	0.0006	0.11	0.108	0.109	0.1090	0.0010	0.119	0.119	0.121	0.1197	0.0012
B148															
B154	0.114	0.121	0.099	0.1113	0.0112	0.132	0.128	0.125	0.1283	0.0035	0.142	0.136	0.135	0.1377	0.0038
B158	0.13	0.13	0.13	0.1300	0.0000	0.14	0.14	0.13	0.1367	0.0058	0.16	0.16	0.16	0.1600	0.0000
B167	0.12	0.12	0.12	0.1200	0.0000	0.13	0.13	0.13	0.1300	0.0000	0.14	0.14	0.13	0.1367	0.0058
B178	0.144	0.144	0.14	0.1427	0.0023	0.148	0.15	0.145	0.1477	0.0025	0.158	0.159	0.155	0.1573	0.0021
B196															
B198	0.1	0.11	0.08	0.0967	0.0153	0.12	0.11	0.11	0.1133	0.0058	0.14	0.15	0.15	0.1467	0.0058
B210															
B213	0.14	0.12	0.14	0.1333	0.0115	0.14	0.16	0.14	0.1467	0.0115	0.16	0.16	0.16	0.1600	0.0000
B222	0.1	0.09	0.1	0.0967	0.0058	0.11	0.09	0.11	0.1033	0.0115	0.12	0.11	0.11	0.1133	0.0058

**Table B-15. Data summary table for CBCA in three hemp samples.**

Data in **blue** font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

*Note: This table spans multiple pages; the NIST values and consensus values are included on all pages for convenience.*

Target Consensus	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	
Lab	A	B	C			A	B	C			A	B	C		
B001	0.037	0.039	0.037	0.0377	0.0012	0.379	0.393	0.401	0.3910	0.0111	0.224	0.221	0.224	0.2230	0.0017
B003															
B004															
B005	< 0.1	< 0.1	< 0.1			0.39	0.395	0.366	0.3837	0.0155	0.208	0.209	0.212	0.2097	0.0021
B006	0.049	0.05	0.051	0.0500	0.0010	0.412	0.407	0.394	0.4043	0.0093	0.229	0.238	0.229	0.2320	0.0052
B009															
B015	0.03	0.05	0.04	0.0400	0.0100	0.36	0.35	0.38	0.3633	0.0153	0.21	0.21	0.21	0.2100	0.0000
B020															
B021															
B023	0.048	0.047	0.0471	0.0474	0.0006	0.3675	0.3635	0.361	0.3640	0.0033	0.193	0.1959	0.1907	0.1932	0.0026
B024	0.1	0.1	0.08	0.0933	0.0115	0.37	0.4	0.38	0.3833	0.0153	0.21	0.21	0.2	0.2067	0.0058
B026	<0.01	<0.01	<0.01			0.29	0.37	0.39	0.3500	0.0529	0.19	0.19	0.2	0.1933	0.0058
B028															
B030	0.05	0.05	0.05	0.0500	0.0000	0.4	0.42	0.41	0.4100	0.0100	0.23	0.24	0.23	0.2333	0.0058
B035	0.04208	0.04378	0.04363	0.0432	0.0009	0.36021	0.36531	0.36271	0.3627	0.0026	0.20572	0.20713	0.20604	0.2063	0.0007
B037															
B041	<0.064					0.362			0.3620		0.202			0.2020	
B043	<0.01					0.347			0.3470		0.211			0.2110	
B048															
B049	0.03291	0.03389	0.03217	0.0330	0.0009	0.30969	0.35819	0.37732	0.3484	0.0349	0.11393	0.20091	0.20008	0.1716	0.0500
B051	0.05	0.04	0.04	0.0433	0.0058	0.36	0.37	0.3	0.3433	0.0379	0.2	0.19	0.15	0.1800	0.0265
B053	0.205	0.189	0.187	0.1937	0.0099	1.301	1.238	1.364	1.3010	0.0630	0.852	0.85	0.883	0.8617	0.0185
B054	0.03773	0.03738	0.03604	0.0371	0.0009	0.38209	0.37982	0.35719	0.3730	0.0138	0.19681	0.19662	0.19541	0.1963	0.0008
B058	0.0609	0.0745	0.0581	0.0645	0.0088	0.394	0.397	0.366	0.3857	0.0171	0.22	0.21	0.195	0.2083	0.0126
B062	< 0.08	< 0.08	< 0.08			0.403	0.412	0.41	0.4083	0.0047	0.209	0.212	0.199	0.2067	0.0068
B064	<0.0001					0.372			0.3720		0.192			0.1920	
B066	0.0778	0.0774	0.0836	0.0796	0.0035	0.4273	0.3949	0.3841	0.4021	0.0225	0.2167	0.2191	0.2134	0.2164	0.0029
B069	0.04	0.05	0.05	0.0467	0.0058	0.47	0.49	0.47	0.4767	0.0115	0.25	0.25	0.26	0.2533	0.0058
B070	<0.222	<0.222	<0.222			0.415	0.362	0.35	0.3757	0.0346	0.273	0.27	0.308	0.2837	0.0211
B076	0.04465	0.04179	0.04588	0.0441	0.0021	0.49061	0.4051	0.42874	0.4415	0.0442	0.1954	0.19635	0.19915	0.1970	0.0019
B078															
B079															
B081	<0.35	<0.35	<0.35			<0.35	<0.35	<0.35			<0.35	<0.35	<0.35		
B082	0.041	0.045	0.042	0.0427	0.0021	0.377	0.395	0.403	0.3917	0.0133	0.196	0.191	0.189	0.1920	0.0036
B084	0.05	0.046	0.048	0.0480	0.0020	0.35	0.34	0.345	0.3450	0.0050	0.19	0.19	0.19	0.1900	0.0000
B085	0.067	0.061	0.053	0.0603	0.0070	0.674	0.596	0.541	0.6037	0.0668	0.306	0.22	0.22	0.2487	0.0497
B095	0.041	0.041	0.041	0.0410	0.0000	0.365	0.35	0.371	0.3620	0.0108	0.22	0.223	0.216	0.2197	0.0035
B096	0.091	0.086	0.089	0.0887	0.0025	0.335	0.353	0.362	0.3500	0.0137	0.214	0.213	0.213	0.2133	0.0006

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4				Plant Sample 6					
Target Consensus				0.045 0.047	0.011 0.015				0.374	0.041				0.206 0.204	0.028 0.025
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B097	<0.01	<0.01	<0.01			0.36	0.36	0.38	0.3667	0.0115	0.18	0.17	0.18	0.1767	0.0058
B099	0.041			0.0410		0.363			0.3630		0.172			0.1720	
B100	0.05	0.04		0.0450	0.0071	0.42	0.36		0.3900	0.0424	0.24	0.21		0.2250	0.0212
B102	0.0404	0.0693	0.0695	0.0597	0.0167	0.3455	0.3563	0.371	0.3576	0.0128	0.205	0.2069	0.202	0.2046	0.0025
B108															
B111															
B114															
B115	0.044	0.043	0.042	0.0430	0.0010	0.4089	0.4	0.4007	0.4032	0.0049	0.2852	0.284	0.277	0.2821	0.0044
B117	0.027	0.053	0.027	0.0357	0.0150	0.017	0.033	<0.002	0.0250	0.0113	0.017	0.016	0.016	0.0163	0.0006
B125	0.052	0.047	0.05	0.0497	0.0025	0.355	0.368	0.369	0.3640	0.0078	0.196	0.212	0.203	0.2037	0.0080
B132	0.074	0.07	0.07	0.0713	0.0023	0.412	0.386	0.334	0.3773	0.0397	0.22	0.221	0.217	0.2193	0.0021
B137	0.06	0.057	0.061	0.0593	0.0021	0.333	0.357	0.376	0.3553	0.0215	0.235	0.235	0.233	0.2343	0.0012
B141	0.041	0.038	0.04	0.0397	0.0015	0.336	0.368	0.351	0.3517	0.0160	0.21	0.204	0.203	0.2057	0.0038
B142	<0.0007	<0.0007	<0.0007			0.458	0.363	0.38	0.4003	0.0507	0.227	0.223	0.212	0.2207	0.0078
B146	0.0297	0.0291	0.028	0.0289	0.0009	0.267	0.288	0.306	0.2870	0.0195	0.152	0.142	0.145	0.1463	0.0051
B148															
B149	<0.127	<0.127	<0.127			0.358	0.373	0.431	0.3873	0.0386	0.26	0.219	0.263	0.2473	0.0246
B154	0.046	0.044	0.039	0.0430	0.0036	0.298	0.324	0.375	0.3323	0.0392	0.19	0.201	0.201	0.1973	0.0064
B158	0.06	0.06	0.06	0.0600	0.0000	0.33	0.31	0.31	0.3167	0.0115	0.17	0.17	0.17	0.1700	0.0000
B159	0.0437	0.0591	0.0542	0.0523	0.0079	0.4262	0.4435	0.4297	0.4331	0.0091	0.2361	0.2455	0.2408	0.2408	0.0047
B161	0.04	0.051	0.05	0.0470	0.0061	0.423	0.404	0.422	0.4163	0.0107	0.225	0.229	0.253	0.2357	0.0151
B165															
B173	0.02099	0.01986	0.02033	0.0204	0.0006	0.35484	0.35933	0.36482	0.3597	0.0050	0.18651	0.17119	0.17562	0.1778	0.0079
B174															
B175	0.08	0.08	0.09	0.0833	0.0058	0.4	0.36	0.4	0.3867	0.0231	0.19	0.19	0.19	0.1900	
B178	0.0479	0.0522	0.0464	0.0488	0.0030	0.406	0.422	0.401	0.4097	0.0110	0.238	0.229	0.233	0.2333	0.0045
B185															
B187	0.0918	0.0915	0.0995	0.0943	0.0045	0.4048	0.4325	0.441	0.4261	0.0189	0.2219	0.2258	0.2133	0.2203	0.0064
B192	<0.0397	<0.0397	<0.0397			0.35551	0.3564	0.34669	0.3529	0.0054	0.19385	0.19607	0.1892	0.1930	0.0035
B193	<0.281	<0.254	<0.298			0.329	0.378	0.318	0.3417	0.0319	<0.304	<0.312	<0.301		
B196															
B200															
B202	0.0428	0.0419	0.0416	0.0421	0.0006	0.341	0.34	0.344	0.3417	0.0021	0.17	0.163	0.162	0.1650	0.0044
B204	0.037	0.035	0.037	0.0363	0.0012	0.357	0.315	0.339	0.3370	0.0211	0.188	0.188	0.186	0.1873	0.0012
B208															
B210															
B215	0.039			0.0390		0.37			0.3700		0.2			0.2000	
B216	<0.040	0.042	0.042	0.0420		0.362	0.388	0.39	0.3800	0.0156	0.195	0.212	0.207	0.2047	0.0087
B219	0.0414	0.0422	0.0428	0.0421	0.0007	0.374	0.398	0.391	0.3877	0.0123	0.212	0.206	0.201	0.2063	0.0055
B221	0.03	0.032	0.035	0.0323	0.0025	1.81	1.87	1.69	1.7900	0.0917	0.118	0.123	0.105	0.1153	0.0093
B222	<0.05	<0.05	<0.05			0.14	0.15	0.1	0.1300	0.0265	0.17	0.18	0.17	0.1733	0.0058
B226	0.02126	0.02002	0.02082	0.0207	0.0006	0.33913	0.3452		0.3422	0.0043	0.18319	0.17736	0.17848	0.1797	0.0031
B230															

**Table B-16. Data summary table for CBCA in three marijuana samples.**

Target Consensus	Plant Sample 2					Plant Sample 3					Plant Sample 5					
	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD
Lab	A	B	C			A	B	C			A	B	C		Avg	SD
B062	0.789	0.784	0.76	0.778	0.016	0.74	0.754	0.708	0.734	0.024	0.663	0.689	0.668	0.673	0.014	
B079																
B099	0.647			0.647		0.631			0.631		0.586				0.586	
B117	0.057	0.067	0.055	0.060	0.006	0.054	0.061	0.061	0.059	0.004	<0.003	0.066	0.056	0.061	0.007	
B125	0.669	0.666	0.656	0.664	0.007	0.648	0.644	0.633	0.642	0.008	0.594	0.577	0.58	0.584	0.009	
B146	0.521	0.509	0.547	0.526	0.019	0.453	0.45	0.429	0.444	0.013	0.348	0.416	0.444	0.403	0.049	
B148																
B154	0.599	0.644	0.505	0.583	0.071	0.595	0.595	0.584	0.591	0.006	0.54	0.52	0.529	0.530	0.010	
B158	0.59	0.6	0.61	0.600	0.010	0.58	0.56	0.57	0.570	0.010	0.54	0.54	0.53	0.537	0.006	
B178	0.774	0.769	0.751	0.765	0.012	0.728	0.728	0.713	0.723	0.009	0.658	0.66	0.648	0.655	0.006	
B196																
B210																
B222	0.57	0.53	0.56	0.553	0.021	0.62	0.5	0.56	0.560	0.060	0.53	0.48	0.53	0.513	0.029	

**Table B-17. Data summary table for CBDV in three hemp samples.**

Data in red font have been flagged as a data entry of zero or results that include text (e.g., “< LOQ” or “present”).

Data in blue font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

Note: This table spans multiple pages; the NIST values and consensus values are included on all pages for convenience.

Target Consensus	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	
Lab	A	B	C			A	B	C			A	B	C		
B003															
<b>B004</b>	0.026	0.026	0.026	0.0260	0.0000	0.003	0.003	0.003	0.0030	0.0000	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.0000</b>	<b>0.0000</b>
B005	< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1		
B006	0.019	0.02	0.019	0.0193	0.0006	< 0.012	< 0.012	< 0.012			< 0.012	< 0.012	< 0.012		
B007															
B009															
B012	0.00953	0.00925		0.0094	0.0002										
B013	<0.028	<0.028	<0.028			<0.057	<0.059	<0.060			<0.028	<0.028	<0.028		
<b>B015</b>	<b>0.1</b>	<b>0.09</b>	<b>0.1</b>	<b>0.0967</b>	<b>0.0058</b>	0.02	0.02	0.03	0.0233	0.0058	0.03	0.03	0.03	0.0300	0.0000
B016	< 0.0004	< 0.0004	< 0.0004			< 0.0004	< 0.0004	< 0.0004			< 0.0004	< 0.0004	< 0.0004		
B018	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B020															
B021															
B023	0.0159	0.0161	0.0158	0.0159	0.0002	<0.0037	<0.0037	<0.0037			<0.0037	<0.0037	<0.0037		
B024		0.06		0.0600											
<b>B026</b>	<b>0.13</b>	<b>0.14</b>	<b>0.13</b>	<b>0.1333</b>	<b>0.0058</b>	0.04	0.04	0.04	0.0400	0.0000	0.02	0.02	0.03	0.0233	0.0058
B027	<0.025	<0.025	<0.025			<0.025	<0.025	<0.025			<0.025	<0.025	<0.025		
B028															
B029	<0.030	<0.030	<0.030			<0.030	<0.030	<0.030			0.042	0.047	0.035	0.0413	0.0060
B030	0.02	0.02	0.02	0.0200	0.0000	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B031															
<b>B033</b>	<b>0.1</b>	<b>0.09</b>	<b>0.09</b>	<b>0.0933</b>	<b>0.0058</b>	< 0.5	< 0.5	< 0.5			< 0.5	< 0.5	< 0.5		
B035	<0.0025	<0.0025	<0.0025			<0.005	<0.005	<0.005			<0.005	<0.005	<0.005		
B036	<0.01	<0.01	<0.01			<0.02	<0.02	<0.02			<0.01	<0.01	<0.01		
B037															
B038															
B041	<0.064					<0.064					<0.064				
B043	<0.01					<0.01					<0.01				
<b>B045</b>	<b>detected</b>	<b>detected</b>				<b>not detected</b>	<b>not detected</b>				<b>not detected</b>	<b>not detected</b>			
B047	< 0.01	< 0.01	< 0.01			< 0.01	< 0.01	< 0.01			< 0.01	< 0.01	< 0.01		
B048															
<b>B051</b>	0.07	0.06	0.02	0.0500	0.0265	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.0000</b>	<b>0.0000</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.0000</b>	<b>0.0000</b>
<b>B052</b>	< 0.10	< 0.10	< 0.10			<b>0.12</b>	< 0.10	< 0.10	<b>0.1200</b>		< 0.10	< 0.10	< 0.10		
B053	0.081	0.083	0.081	0.0817	0.0012	0.01	0.01	0.011	0.0103	0.0006	0.007	0.006	0.007	0.0067	0.0006
B054	0.0123	0.01027	0.01056	0.0110	0.0011	<0.0001	<0.0001	<0.0001			<0.0001	<0.0001	<0.0001		
<b>B055</b>	<b>0.0923</b>	<b>0.0921</b>	<b>0.0928</b>	<b>0.0924</b>	<b>0.0004</b>	<0.0432	<0.0432	<0.0432			<0.0432	<0.0432	<0.0432		
B058	< 0.0295	< 0.0296	< 0.0296			< 0.0292	< 0.0295	< 0.0298			< 0.0289	< 0.0297	< 0.0296		

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4				Plant Sample 6					
Target Consensus				0.0188 0.027	0.0032 0.019				0.018	0.022				0.014	0.015
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B060															
B061	0.02	0.027	0.02	0.0223	0.0040	<0.004	<0.004	<0.004			<0.004	<0.004	<0.004		
B062	< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08		
B063															
B064	<0.0000					<0.0000					<0.0000				
B066	0.0137	0.0107	0.0133	0.0126	0.0016	<0.003	<0.003	<0.003			<0.003	<0.003	<0.003		
B068															
B069	0.02	0.02	0.02	0.0200	0.0000	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B070	<0.222	<0.222	<0.222			<0.111	<0.111	<0.111			<0.067	<0.067	<0.067		
B073	0.0168			0.0168		<0.0062					<0.0062				
B076	0.02963	0.02787	0.02911	0.0289	0.0009	0.01427	0.01358	0.01381	0.0139	0.0004	0.0093	0.00999	0.0096	0.0096	0.0003
B077															
B078															
B079															
B081	<0.16	<0.16	<0.16			<0.16	<0.16	<0.16			<0.16	<0.16	<0.16		
B082	0.017	0.017	0.014	0.0160	0.0017	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
<b>B084</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.0000</b>	<b>0.0000</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.0000</b>	<b>0.0000</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.0000</b>	<b>0.0000</b>
B085	0.019	0.018	0.017	0.0180	0.0010	0.003	0.002	0.003	0.0027	0.0006	0.002	< 0.002	< 0.002	0.0020	
<b>B088</b>	0.03	0.03	0.029	0.0297	0.0006	<b>0.246</b>	<b>0.273</b>	<b>0.286</b>	<b>0.2683</b>	<b>0.0204</b>	<0.006	<0.006	<0.006		
<b>B090</b>	<0.05	<0.05	<0.05			<b>0</b>	<b>0</b>	<b>0</b>	<b>0.0000</b>		<b>0</b>	<b>0</b>	<b>0</b>	<b>0.0000</b>	
B091	<0.023	<0.023	<0.023			<0.117	<0.117	<0.117			<0.065	<0.065	<0.065		
B094	0.03	0.02	0.03	0.0267	0.0058	< 0.02	< 0.02	< 0.02			< 0.02	< 0.02	< 0.02		
B095	0.024	0.024	0.024	0.0240	0.0000	< 0.002	< 0.002	< 0.002			< 0.002	< 0.002	< 0.002		
B097	<0.01	<0.01	<0.01			0.08	0.07	0.08	0.0767	0.0058	<0.01	<0.01	<0.01		
B099	<0.01					<0.01					<0.01				
B100	<0.03	<0.01				<0.01	<0.01				<0.01	<0.01			
B102	< 0.0001	< 0.0001	< 0.0001			0.0266	0.026	0.0264	0.0263	0.0003	< 0.0001	< 0.0001	< 0.0001		
B106	0.018	0.018	0.018	0.0180		<0.001	<0.001	<0.001			<0.001	<0.001	<0.001		
<b>B109</b>	<b>0.208</b>	<b>0.201</b>	<b>0.197</b>	<b>0.2020</b>	<b>0.0056</b>	0.079	0.079	0.071	0.0763	0.0046	<b>0.068</b>	<b>0.068</b>	<b>0.068</b>	<b>0.0680</b>	
B110	<0.02	<0.02	<0.02			<0.02	<0.02	<0.02			<0.02	<0.02	<0.02		
B111															
<b>B113</b>	0.01	0.01	0.01	0.0100		0.01	0.01	<b>0</b>	0.0100	0.0000	<b>0</b>	<b>0</b>	<b>0</b>	<b>0.0000</b>	
B114															
B115	0.055	0.055	0.058	0.0560	0.0017	0.01206	0.01198	0.01145	0.0118	0.0003	0.01	0.01	0.01	0.0100	0.0000
<b>B116</b>	<b>0.1156</b>	<b>0.1142</b>	<b>0.1147</b>	<b>0.1148</b>	<b>0.0007</b>	0.0328	0.0324	0.0307	0.0320	0.0011	0.0304	0.0303	0.0305	0.0304	0.0001
B117	0.014	0.015	0.009	0.0127	0.0032	<0.002	<0.002	<0.002			<0.002	<0.002	<0.002		
B120															
B122															
B125	0.013	0.013	0.013	0.0130		<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B126															
B129															
B130	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B132	0.036	0.034	0.036	0.0353	0.0012	0.007	0.006	0.006	0.0063	0.0006	0.004	0.004	0.004	0.0040	

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.0188 0.027	0.0032 0.019				0.018	0.022				0.014	0.015
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B137	0.058	0.055	0.058	0.0570	0.0017	0.023	0.025	0.024	0.0240	0.0010	0.023	0.021	0.023	0.0223	0.0012
<b>B141</b>	<b>0.112</b>	<b>0.105</b>	<b>0.109</b>	<b>0.1087</b>	<b>0.0035</b>	0.028	0.038	0.037	0.0343	0.0055	0.043	0.04	0.041	0.0413	0.0015
B142	<0.0007	<0.0007	<0.0007			<0.0075	<0.0075	<0.0075			<0.0007	<0.0007	<0.0007		
B146	0.019	0.0176	0.0192	0.0186	0.0009	0.0205	0.0243	0.0207	0.0218	0.0021	< 0.0159	< 0.0161	< 0.0161		
B147	<0.016	<0.016	<0.016			<0.016	<0.016	<0.016			<0.016	<0.016	<0.016		
B148															
B149	<0.0159	<0.0159	<0.0159			<0.0159	<0.0159	<0.0159			<0.0159	<0.0159	<0.0159		
B152															
B153	<0.0004	<0.0004	<0.0004			<0.0004	<0.0004	<0.0004			<0.0004	<0.0004	<0.0004		
<b>B154</b>	<b>0.094</b>	<b>0.097</b>	<b>0.089</b>	<b>0.0933</b>	<b>0.0040</b>	0.026	0.029	0.037	0.0307	0.0057	0.037	0.038	0.039	0.0380	0.0010
B158	<0.05	<0.05	<0.05			<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B159	<0.0099	<0.0100	<0.0098			<0.0102	<0.0101	<0.0099			<0.0097	<0.0095	<0.0102		
<b>B160</b>	<b>&lt;LOQ</b>	<b>&lt;LOQ</b>	<b>&lt;LOQ</b>			<b>&lt;LOQ</b>	<b>&lt;LOQ</b>	<b>&lt;LOQ</b>			<b>&lt;LOQ</b>	<b>&lt;LOQ</b>	<b>&lt;LOQ</b>		
<b>B161</b>	<b>0</b>	<b>0</b>	<b>0</b>	<b>0.0000</b>		<b>0</b>	<b>0</b>	<b>0</b>	<b>0.0000</b>		<b>0</b>	<b>0</b>	<b>0</b>	<b>0.0000</b>	
B163	0.0244	0.0202	0.0232	0.0226	0.0022	0.00337	0.00268	0.00233	0.0028	0.0005	0.00194	0.0016	0.00215	0.0019	0.0003
<b>B164</b>	<b>0.09</b>	<b>0.09</b>	<b>0.1</b>	<b>0.0933</b>	<b>0.0058</b>	<0.001	0.06	0.06	0.0600		0.06	<0.001	0.05	0.0550	0.0071
B165															
B172	< 0.031	< 0.031	< 0.031			< 0.031	< 0.031	< 0.031			< 0.031	< 0.031	< 0.031		
B173	< 0.0101	< 0.0098	< 0.0097			< 0.01	< 0.0101	< 0.01020			< 0.0101	< 0.0098	< 0.0099		
B174	< 0.06	< 0.06	< 0.06			< 0.06	< 0.06	< 0.06							
B178	0.0244	0.0232	0.0243	0.0240	0.0007	<0.0006	<0.0006	<0.0006			<0.0006	<0.0006	<0.0006		
B181	<0.0077					<0.0077									
<b>B182</b>	<b>0.09</b>	<b>0.1</b>		<b>0.0950</b>	<b>0.0071</b>	<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B184	<0.038	<0.038	<0.038			<0.038	<0.038	<0.038			<0.038	<0.038	<0.038		
B185															
B186	<0.0001	<0.0001	<0.0001			<0.0001	<0.0001	<0.0001			<0.0001	<0.0001	<0.0001		
<b>B187</b>	<b>0.1373</b>	<b>0.1311</b>	<b>0.1414</b>	<b>0.1366</b>	<b>0.0052</b>	<0.06	<0.06	<0.06			<0.06	<0.06	<0.06		
B188															
B189	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B190	0.02	0.02	0.02	0.0200											
B192	0.02166	0.02179	0.02099	0.0215	0.0004	<0.0202	<0.0202	<0.0202			<0.0203	<0.0203	<0.0203		
<b>B193</b>	<b>0.152</b>	<b>0.25</b>	<b>0.133</b>	<b>0.1783</b>	<b>0.0628</b>	<0.0854	<0.0787	<0.0833			<0.0898	<0.0924	<0.0889		
B195	0.014	0.013	0.013	0.0133	0.0006	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B198	< 0.07	< 0.07	< 0.07			<0.07	<0.07	<0.07			<0.07	<0.07	<0.07		
B200															
B202	0.0378	0.0368	0.036	0.0369	0.0009	<0.005	<0.005	<0.005			<0.005	<0.005	<0.005		
B204	0.019	0.019	0.02	0.0193	0.0006	<0.002	<0.002	<0.002			<0.002	<0.002	<0.002		
B205	0.06	0.059	0.057	0.0587	0.0015	0.029	0.031	0.029	0.0297	0.0012	0.014	0.016	0.016	0.0153	0.0012
<b>B206</b>	<b>0.111</b>	<b>0.112</b>	<b>0.113</b>	<b>0.1120</b>	<b>0.0010</b>	<b>0.127</b>	<b>0.117</b>	<b>0.119</b>	<b>0.1210</b>	<b>0.0053</b>	<b>0.07</b>	<b>0.073</b>	<b>0.074</b>	<b>0.0723</b>	<b>0.0021</b>
B208															
B210															
B212	0.019	0.019	0.019	0.0190		<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B213	< 0.03	< 0.03	< 0.03			<0.03	<0.03	<0.03			<0.03	<0.03	<0.03		

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4				Plant Sample 6					
Target Consensus				0.0188 0.027	0.0032 0.019				0.018	0.022				0.014 0.014	0.015 0.0001
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B215	<0.0039														
B217	< 0.007	< 0.007	< 0.008			< 0.007	< 0.007	< 0.007			< 0.008	< 0.007	< 0.007		
B219	0.0182	0.0189	0.0184	0.0185	0.0004	0.00272	0.00283	0.00276	0.0028	0.0001	0.00152	0.00139	0.00142	0.0014	0.0001
B221	0.024	0.023	0.024	0.0237	0.0006	0.003	0.003	0.004	0.0033	0.0006	0.002	0.002	0.002	0.0020	
B222	0.09	0.1	0.1	0.0967	0.0058	<0.04	<0.04	<0.04			<0.05	<0.05	<0.05		
B223	0.0172	0.0169	0.017	0.0170	0.0002	<0.0125	<0.0125	<0.0125			<0.0125	<0.0125	<0.0125		
B224	0.02	0.02	0.02	0.0200		<0.03	<0.03	<0.03			<0.01	<0.01	<0.01		
B226	< 0.0102	< 0.0099	< 0.01			< 0.0097	< 0.0099	< 0.0099			< 0.0099	< 0.0099	< 0.0099		
B228	< 0.02	< 0.02	< 0.02			< 0.02	< 0.02	< 0.02			< 0.02	< 0.02	< 0.02		
B230															

**Table B-18. Data summary table for CBDV in three marijuana samples.**

Data in red font have been flagged as a data entry of zero or results that include text (e.g., “< LOQ” or “present”).

Target Consensus	Plant Sample 2					Plant Sample 3					Plant Sample 5				
	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	
Lab	A	B	C		A	B	C		A	B	C		Avg	SD	
B045	detected	detected													
B055	< 0.0432	< 0.0432	< 0.0432		<0.0432	<0.0432	<0.0432		<0.0432	<0.0432	<0.0432				
B062	< 0.08	< 0.08	< 0.08		< 0.08	< 0.08	< 0.08		< 0.08	< 0.08	< 0.08				
B079															
B099	<0.01				<0.01				<0.01						
B109	0.152	0.157	0.157	0.155	0.003	0.157	0.154	0.154	0.155	0.002	0.158	0.152	0.153	0.154	0.003
B117	<0.002	<0.002	<0.002			<0.002	<0.002	<0.002			<0.002	<0.002	<0.002		
B125	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B146	0.052	0.0523	0.0521	0.052	0.000	0.0526	0.051	0.0528	0.052	0.001	0.0589	0.0593	0.059	0.059	0.000
B148															
B154	0.061	0.068	0.055	0.061	0.007	0.065	0.06	0.063	0.063	0.003	0.061	0.055	0.056	0.057	0.003
B158	<0.05	<0.05	<0.05			<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B178	<0.0006	<0.0006	<0.0006			<0.0006	<0.0006	<0.0006			<0.0006	<0.0006	<0.0006		
B198	<0.07	<0.07	<0.07			<0.07	<0.07	<0.07			<0.07	<0.07	<0.07		
B210															
B213	< 0.03	< 0.03	< 0.03			< 0.03	< 0.03	< 0.03			< 0.03	< 0.03	< 0.03		
B222	<0.05	<0.05	<0.05			<0.05	<0.05	<0.05			0.08	0.08	0.08	0.080	

**Table B-19. Data summary table for CBDVA in three hemp samples.**

Data in **red** font have been flagged as a data entry of zero or results that include text (e.g., “< LOQ” or “present”).

Data in **blue** font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

*Note: This table spans multiple pages; the NIST values and consensus values are included on all pages for convenience.*

Target Consensus	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg
Lab	A	B	C			A	B	C			A	B	C		
B001	0.075	0.078	0.072	0.0750	0.0030	0.059	0.061	0.062	0.0607	0.0015	0.031	0.031	0.031	0.0310	0.0000
B003															
B004	0.03	0.03	0.03	0.0300	0.0000	0.051	0.051	0.051	0.0510	0.0000	0.025	0.025	0.025	0.0250	0.0000
B005	< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1		
B006	0.077	0.078	0.076	0.0770	0.0010	0.044	0.042	0.043	0.0430	0.0010	0.028	0.028	0.028	0.0280	0.0000
B009															
B012	0.10261	0.10027		0.1014	0.0017	0.03201	0.03317		0.0326	0.0008	0.017	0.01991		0.0185	0.0021
B015	0.08	0.08	0.06	0.0733	0.0115	0.03	0.03	0.03	0.0300	0.0000	0.02	0.02	0.02	0.0200	0.0000
B020															
B021															
B023	0.0972	0.098	0.0941	0.0964	0.0021	0.0473	0.0484	0.0361	0.0439	0.0068	0.0251	0.032	0.0317	0.0296	0.0039
B024	0.13	0.13	0.13	0.1300	0.0000	0.04	0.05	0.04	0.0433	0.0058					
B026	0.1	0.11	0.11	0.1067	0.0058	0.05	0.04	0.04	0.0433	0.0058	0.02	0.03	0.02	0.0233	0.0058
B027	0.0788	0.0766	0.076	0.0771	0.0015	0.0409	0.04	0.0408	0.0406	0.0005	0.0256	0.0263	0.0257	0.0259	0.0004
B028															
B030	0.07	0.06	0.07	0.0667	0.0058	0.03	0.04	0.03	0.0333	0.0058	<0.02	<0.02	<0.02		
B031															
B035	0.08789	0.08845	0.08867	0.0883	0.0004	0.03179	0.03269	0.03475	0.0331	0.0015	0.0243	0.02452	0.02358	0.0241	0.0005
B037															
B041	0.074			0.0740		<0.064					<0.064				
B043	0.098			0.0980		0.041			0.0410		0.029				0.0290
B048															
<b>B051</b>	0.07	0.06	0.05	0.0600	0.0100	0.03	0.03	0.03	0.0300	0.0000	0.02	<b>0.00</b>	0.02	0.0133	0.0115
<b>B053</b>	<b>0.24</b>	<b>0.246</b>	<b>0.248</b>	<b>0.2447</b>	<b>0.0042</b>	<b>0.146</b>	<b>0.142</b>	<b>0.151</b>	<b>0.1463</b>	<b>0.0045</b>	<b>0.112</b>	<b>0.113</b>	<b>0.117</b>	<b>0.1140</b>	<b>0.0026</b>
B054	0.06203	0.06334	0.05911	0.0615	0.0022	0.0389	0.03809	0.03656	0.0378	0.0012	0.02582	0.02545	0.0249	0.0254	0.0005
B055	0.0793	0.0778	0.0813	0.0795	0.0018	0.0427	0.0401	0.0416	0.0415	0.0013	0.0253	0.024	0.0251	0.0248	0.0007
B058	0.0823	0.0807	0.0799	0.0810	0.0012	0.0329	0.0321		0.0325	0.0006	<0.0289	<0.0297	<0.0296		
<b>B064</b>	0.079			0.0790		<0.00001					<b>0.00001</b>				<b>0.0000</b>
B066	0.0809	0.0791	0.0837	0.0812	0.0023	0.039	0.034	0.0347	0.0359	0.0027	0.021	0.0251	0.0253	0.0238	0.0024
B069	0.113	0.11	0.105	0.1093	0.0040	<0.01	<0.01	<0.01			<0.01	0.038	<0.01	0.0380	
B070	0.092	0.105	0.098	0.0983	0.0065	<0.111	<0.111	<0.111			<0.067	<0.067	<0.067		
B076	0.11889	0.11848	0.1182	0.1185	0.0003	0.04499	0.04495	0.04519	0.0450	0.0001	0.02879	0.02841	0.02935	0.0288	0.0005
B078															
B079															
B081	<0.15	<0.15	<0.15			<0.15	<0.15	<0.15			<0.15	<0.15	<0.15		
B082	0.094	0.092	0.091	0.0923	0.0015	0.037	0.042	0.044	0.0410	0.0036	0.027	0.028	0.028	0.0277	0.0006
B084															

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.0719	0.0054				0.0388	0.0085				0.0260	0.0070
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B085	0.087	0.086	0.077	0.0833	0.0055	0.047	0.047	0.041	0.0450	0.0035	0.029	0.023	0.022	0.0247	0.0038
B088	0.079	0.079	0.079	0.0790	0.0000	0.034	0.034	0.036	0.0347	0.0012	0.021	0.021	0.02	0.0207	0.0006
B095	0.083	0.084	0.083	0.0833	0.0006	0.034	0.033	0.035	0.0340	0.0010	0.026	0.027	0.027	0.0267	0.0006
B097	0.09	0.09	0.08	0.0867	0.0058	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B099	0.087			0.0870		0.036			0.0360		0.023			0.0230	
B100	0.081	0.077		0.0790	0.0028	0.042	0.039		0.0405	0.0021	0.031	0.028		0.0295	0.0021
B102	0.0882	0.0853	0.0836	0.0857	0.0023	0.035	0.0361	0.0361	0.0357	0.0006	0.0263	0.0262	0.0255	0.0260	0.0004
B111															
B114															
B115	0.081	0.081	0.083	0.0817	0.0012	0.0438	0.0428	0.0415	0.0427	0.0012	0.0253	0.0295	0.0293	0.0280	0.0024
B117	0.062	0.076	0.078	0.0720	0.0087	0.039	0.038	0.037	0.0380	0.0010	0.097	0.036	0.035	0.0560	0.0355
B125	0.101	0.104	0.103	0.1027	0.0015	0.039	0.039	0.039	0.0390		0.029	0.029	0.029	0.0290	
B129															
B130	0.06	0.06	0.06	0.0600		0.04	0.03	0.03	0.0333	0.0058	0.02	0.02	0.03	0.0233	0.0058
B132	0.106	0.102	0.105	0.1043	0.0021	0.054	0.05	0.043	0.0490	0.0056	0.034	0.034	0.033	0.0337	0.0006
B137	<0.0001	<0.0001	<0.0001			<0.0001	<0.0001	<0.0001			<0.0001	<0.0001	<0.0001		
B141	0.076	0.072	0.074	0.0740	0.0020	0.035	0.038	0.036	0.0363	0.0015	0.027	0.027	0.026	0.0267	0.0006
B142	0.09	0.091	0.089	0.0900	0.0010	0.042	0.039	0.044	0.0417	0.0025	0.03	0.029	0.027	0.0287	0.0015
B146	0.0778	0.0753	0.0766	0.0766	0.0013	0.032	0.0374	0.0365	0.0353	0.0029	0.0216	0.0213	0.0215	0.0215	0.0002
B148															
B149	0.045	0.036	0.036	0.0390	0.0052	0.021	0.019	0.02	0.0200	0.0010	<0.0230	<0.0230	<0.0230		
B153	0.093	0.086	0.094	0.0910	0.0044	0.044	0.05	0.048	0.0473	0.0031	0.034	0.034	0.035	0.0343	0.0006
B154	0.069	0.072	0.066	0.0690	0.0030	0.029	0.041	0.037	0.0357	0.0061	0.023	0.021	0.025	0.0230	0.0020
B158	0.11	0.1	0.1	0.1033	0.0058	0.07	0.07	0.06	0.0667	0.0058	0.05	0.05	0.05	0.0500	0.0000
B159	0.0721	0.071	0.0698	0.0710	0.0012	0.0354	0.0332	0.0336	0.0341	0.0016	0.0219	0.0223	0.0223	0.0222	0.0002
B161	0.085	0.082	0.085	0.0840	0.0017	0.045	0.041	0.044	0.0433	0.0021	0.041	0.033	0.032	0.0353	0.0049
B163	0.0955	0.0785	0.0794	0.0845	0.0096	0.0414	0.037	0.0278	0.0354	0.0069	0.0307	0.0291	0.0283	0.0294	0.0012
B165															
B173	0.06418	0.0688	0.06382	0.0656	0.0028	0.03026	0.02875	0.03257	0.0305	0.0019	0.01675	0.01535	0.01515	0.0157	0.0009
B174															
B178	0.131	0.127	0.125	0.1277	0.0031	0.106	0.053	0.052	0.0703	0.0309	0.0416	0.0387	0.0394	0.0399	0.0015
B185															
B187	0.0992	0.0939	0.0996	0.0976	0.0032	<0.06	<0.06	<0.06			<0.06	<0.06	<0.06		
B192	0.07733	0.07659	0.07585	0.0766	0.0007	0.0362	0.03641	0.03545	0.0360	0.0005	0.02441	0.024	0.02411	0.0242	0.0002
B193	0.118	0.201	0.109	0.1427	0.0507	0.0702	0.0616	0.063	0.0649	0.0046	<0.0438	0.045	<0.0434	0.0450	
B195	0.092	0.089	0.091	0.0907	0.0015	0.033	0.034	0.043	0.0367	0.0055	0.02	0.021	0.021	0.0207	0.0006
B200															
B202	0.0892	0.0922	0.0912	0.0909	0.0015	0.0401	0.0386	0.0393	0.0393	0.0008	0.0245	0.0259	0.0274	0.0259	0.0015
B204	0.062	0.062	0.063	0.0623	0.0006	0.039	0.031	0.037	0.0357	0.0042	0.026	0.025	0.026	0.0257	0.0006
B208															
B210															
B215															
B219	0.0704	0.0728	0.0714	0.0715	0.0012	0.038	0.0392	0.0384	0.0385	0.0006	0.026	0.0254	0.0251	0.0255	0.0005

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4				Plant Sample 6					
Target Consensus				0.0719 0.084	0.0054 0.020				0.0388	0.0085				0.0260 0.0297	0.0070 0.0006
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B221	0.114	0.1	0.106	0.1067	0.0070	0.043	0.047	0.048	0.0460	0.0026	0.03	0.029	0.03	0.0297	0.0006
B222	<0.05	<0.05	<0.05			<0.04	<0.04	<0.04			<0.05	<0.05	<0.05		
B224	0.04	0.04	0.04	0.0400		0.02	0.02	0.02	0.0200		0.01	0.01	0.01	0.0100	
B226	0.06703	0.06376	0.0642	0.0650	0.0018	0.02973	0.02916		0.0294	0.0004	0.01628	0.0158	0.01568	0.0159	0.0003
B230															

**Table B-20. Data summary table for CBDVA in three marijuana samples.**

Data in blue font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

Target Consensus	Plant Sample 2					Plant Sample 3					Plant Sample 5				
	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B055	0.0773	0.0762	0.0759	0.0765	0.0007	0.0736	0.0728	0.0727	0.0730	0.0005	0.0684	0.0695	0.0675	0.0685	0.0010
B079				0.0650		0.064			0.0640		0.061			0.0610	
B099	0.065														
B117	0.076	0.077	0.07	0.0743	0.0038	0.07	0.067	0.065	0.0673	0.0025	0.061	0.063	0.059	0.0610	0.0020
B125	0.077	0.074	0.074	0.0750	0.0017	0.076	0.075	0.074	0.0750	0.0010	0.075	0.072	0.073	0.0733	0.0015
B146	0.0701	0.0697	0.07	0.0699	0.0002	0.0698	0.0691	0.0709	0.0699	0.0009	0.0812	0.0815	0.0817	0.0815	0.0003
B148															
B154	0.08	0.085	0.064	0.0763	0.0110	0.066	0.075	0.067	0.0693	0.0049	0.072	0.07	0.067	0.0697	0.0025
B158	0.1	0.1	0.1	0.1000	0.0000	0.1	0.1	0.1	0.1000	0.0000	0.11	0.11	0.11	0.1100	0.0000
<b>B178</b>	<b>0.107</b>	<b>0.127</b>	<b>0.103</b>	<b>0.1123</b>	<b>0.0129</b>	0.106	0.127	0.104	0.1123	0.0127	0.106	0.117	0.115	0.1127	0.0059
B210															
B222	<0.05	<0.05	<0.05			<0.05	<0.05	<0.05			0.07	0.06	0.06	0.0633	0.0058

**Table B-21. Data summary table for CBG in three hemp samples.**

Data in **red** font have been flagged as a data entry of zero or results that include text (e.g., “< LOQ” or “present”).

Data in **blue** font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

Note: This table spans multiple pages; the NIST values and consensus values are included on all pages for convenience.

Target Consensus	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4				Plant Sample 6			
	Avg	SD	Avg	SD	Avg	Avg	SD	SD	Avg	Avg	SD	SD	
Lab	A	B	C			A	B	C		A	B	C	
B001	0.013	0.013	0.013	0.0130	0.0000	0.059	0.06	0.06	0.05967	0.00058	0.013	0.013	0.013
B003						0.11233	0.10533	0.11094	0.10953	0.00371	0.04492	0.04853	0.0374
B004	0.014	0.014	0.014	0.0140	0.0000	0.023	0.023	0.023	0.02300	0.00000	0.005	0.005	0.005
B005	< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1
B006	< 0.011	< 0.011	< 0.011			0.022	0.023	0.024	0.02300	0.00100	< 0.012	< 0.012	< 0.012
B007													
B009													
B012						0.02016	0.0182		0.01918	0.00139			
B013	<0.028	<0.028	<0.028			<0.057	<0.059	<0.060			<0.028	<0.028	<0.028
B015						0.02	0.02	0.02	0.02000	0.00000			
B016	< 0.0004	< 0.0004	< 0.0004			< 0.0004	< 0.0004	< 0.0004			< 0.0004	< 0.0004	< 0.0004
B018	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01
B020													
B021													
B022	<0.04	<0.04	<0.04			<0.04	<0.04	<0.04			<0.04	<0.04	<0.04
B023	0.0144	0.0158	0.0143	0.0148	0.0008	0.0274	0.0277	0.0214	0.02550	0.00355	<0.01497	<0.0149	<0.0149
B024													
B026	<0.01	<0.01	<0.01			0.01	0.01	0.01	0.01000		<0.01	<0.01	<0.01
B027	<0.025	<0.025	<0.025			<0.025	<0.025	<0.025			<0.025	<0.025	<0.025
B028													
B029	<0.030	<0.030	<0.030			0.04	0.046	0.043	0.04300	0.00300	<0.030	<0.030	<0.030
B030	<0.01	<0.01	<0.01			0.02	0.02	0.03	0.02333	0.00577	<0.01	<0.01	<0.01
B031													
B032													
B033	< 0.5	< 0.5	< 0.5			<0.5	< 0.5	< 0.5			< 0.5	< 0.5	< 0.5
B035	0.01239	0.01235	0.01239	0.0124	0.0000	0.03057	0.02334	0.02735	0.02709	0.00362	0.01102	0.01023	0.00897
B036	0.04	0.04	0.04	0.0400	0.0000	0.04	0.04	0.05	0.04333	0.00577	0.03	0.03	0.03
B037													
B038													
B041	<0.064					<0.064					<0.064		
B043	<0.01					<0.01					<0.01		
B044	0.011	<0.075	<0.075			<0.075	<0.075	<0.075			<0.075	<0.075	<0.075
B045	not detected	not detected				detected	detected				detected	detected	
B047	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01
B048													
B049													
B051	0.00	0.00	0.11	0.0367	0.0635	0.02	0.00	0.04	0.02000	0.02000	0.00	0.00	0.00

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.00478	0.00094				0.0255	0.0082				0.0104	0.0048
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B052	< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08		
B053	0.017	0.016	0.017	0.0167	0.0006	0.093	0.093	0.099	0.0950	0.00346	0.041	0.039	0.042	0.04067	0.00153
B054	<0.0001	<0.0001	<0.0001			0.01689	0.02101	0.0203	0.01940	0.00220	0.00606	0.00499	0.00618	0.00574	0.00065
B055	< 0.0812	< 0.0812	< 0.0812			0.0239	0.0275	0.0251	0.02550	0.00183	< 0.0812	< 0.0812	< 0.0812		
B058	< 0.0295	< 0.0296	< 0.0296			< 0.0292	< 0.0295	< 0.0298			< 0.0289	< 0.0297	< 0.0296		
B060															
B061	<0.004	<0.004	0.004	0.0040		0.018	0.02	0.023	0.02033	0.00252	0.009	0.004	<0.004	0.00650	0.00354
B062	< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08		
B063															
B064	<0.0001					<0.0001					<0.0001				
B066	<0.004	<0.004	<0.004			0.0327	0.0326	0.0301	0.03180	0.00147	0.011	0.009	0.0095	0.00983	0.00104
B068															
B069	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B070	<0.222	<0.222	<0.222			<0.111	<0.111	<0.111			<0.067	<0.067	<0.067		
B071	<0.005	<0.005	<0.005			<0.005	<0.005	<0.005			<0.005	<0.005	<0.005		
B072	0.009	0.009	0.01	0.0093	0.0006	0.028	0.029	0.028	0.02833	0.00058	0.012	0.012	0.012	0.01200	0.00000
B073	0.0061			0.0061		0.0221			0.02210		0.0094			0.00940	
B074	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B076	0.00778	0.009	0.00729	0.0080	0.0009	0.00659	0.00595	0.00701	0.00652	0.00054	0.0016	0.00064	0.00079	0.00101	0.00051
B077															
B078															
B079															
B081	<0.31	<0.31	<0.31			<0.31	<0.31	<0.31			<0.31	<0.31	<0.31		
B082	0.004	0.002	0.009	0.0050	0.0036	0.012	0.015	0.019	0.01533	0.00351	<0.02	<0.02	<0.02		
B084	0.00	0.00	0.00	0.0000	0.0000	0.02	0.02	0.02	0.02000	0.00000	0.01	0.01	0.01	0.01000	0.00000
B085	0.005	0.005	0.005	0.0050	0.0000	0.027	0.027	0.025	0.02633	0.00115	0.01	0.009	0.009	0.00933	0.00058
B088	<0.006	<0.006	<0.006			0.015	0.015	0.014	0.01467	0.00058	<0.006	<0.006	<0.006		
B090	0	0	0	0.0000		<0.05	<0.05	<0.05			0	0	0	0.00000	
B091	<0.016	<0.016	<0.016			<0.083	<0.083	<0.083			<0.046	<0.046	<0.046		
B094	< 0.02	< 0.02	< 0.02			0.02	0.02	0.02	0.02000		< 0.02	< 0.02	< 0.02		
B095	< 0.002	< 0.002	< 0.002			< 0.002	< 0.002	< 0.002			< 0.002	< 0.002	< 0.002		
B096	0.021	0.024	0.021	0.0220	0.0017	0.029	0.042	0.029	0.03333	0.00751	< 1	0.027	< 1	0.02700	
B097	< 0.01	< 0.01	< 0.01			< 0.01	< 0.01	< 0.01			< 0.01	< 0.01	< 0.01		
B098															
B099	0.024			0.0240		0.027			0.02700		< 0.01				
B100	< 0.02	< 0.02				< 0.05	< 0.05				< 0.02	< 0.02	< 0.02		
B102	0.0162	0.0151	0.0164	0.0159	0.0007	0.0221	0.0227	0.0235	0.02277	0.00070	< 0.0001	< 0.0001	< 0.0001	0.01000	
B106	0.004	0.004	0.004	0.0040		0.026	0.024	0.025	0.02500	0.00100	0.01	0.01	0.01	0.01000	
B108															
B109	< 0.012	< 0.012	< 0.012			0.02	0.02	0.018	0.01933	0.00115	< 0.012	< 0.012	< 0.012		
B110	< 0.02	< 0.02	< 0.02			0.02	0.03	0.03	0.02667	0.00577	< 0.02	< 0.02	< 0.02		
B111															
B113	0	0	0	0.0000		0.02	0.02	0.02	0.02000		0.01	0.01	0.01	0.01000	

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.00478	0.00094				0.0255	0.0082				0.0104	0.0048
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B114															
B115															
B116	0.0109	0.0114	0.0113	0.0112	0.0003	0.0274	0.0275	0.0262	0.02703	0.00072	0.0103	0.0098	0.0099	0.01000	0.00026
B117	0.015	0.046	0.052	0.0377	0.0199	<0.002	<0.002	<0.002			<0.002	<0.002	<0.002		
B120															
B122															
B124	0.016	0.016	0.016	0.0160		0.032	0.033	0.031	0.03200	0.00100	0.013	0.013	0.013	0.01300	
B125	<0.01	<0.01	<0.01			0.024	0.023	0.023	0.02333	0.00058	<0.01	<0.01	<0.01		
B126															
B129															
B130	0.01	0.01	0.01	0.0100		0.02	0.02	0.02	0.02000		0.01	0.01	0.01	0.01000	
B131	<0.0250	<0.0250	<0.0250			<0.0250	<0.0250	<0.0250			<0.0250	<0.0250	<0.0250		
B132	0.009	0.009	0.009	0.0090		0.039	0.037	0.033	0.03633	0.00306	0.015	0.015	0.014	0.01467	0.00058
B136															
B137	0.017	0.01	0.017	0.0147	0.0040	0.029	0.027	0.033	0.02967	0.00306	0.014	0.011	0.039	0.02133	0.01537
B141	<0.0045	<0.0045	<0.0045			0.037	0.039	0.028	0.03467	0.00586	0.01	0.009	0.01	0.00967	0.00058
B142	<0.0007	<0.0007	<0.0007			0.041	0.018	0.023	0.02733	0.01210	0.012	0.012	0.011	0.01167	0.00058
B144	<0.001	0.006	<0.001	0.0060		0.03	0.03	0.03	0.03000		0.015	0.01	0.014	0.01300	0.00265
B146	<0.0157	<0.0160	<0.0159			0.037	0.0482	0.048	0.04440	0.00641	<0.0158	<0.0160	<0.0160		
B147	<0.016	<0.016	<0.016			0.026	0.024	0.026	0.02533	0.00115	<0.016	<0.016	<0.016		
B148															
B149	<0.0105	<0.0105	<0.0105			0.014	0.01	0.011	0.01167	0.00208	<0.0105	<0.0105	<0.0105		
B152															
B153	<0.0005	<0.0005	<0.0005			<0.0005	<0.0005	<0.0005			<0.0005	<0.0005	<0.0005		
B154	<0.045	<0.045	<0.045			0.025	0.029	0.033	0.02900	0.00400	<0.045	0.016	0.015	0.01550	0.00071
B158	<0.05	<0.05	<0.05			<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B159	<0.0099	<0.0100	<0.0098			<0.0102	<0.0101	<0.0099			<0.0097	<0.0095	<0.0102		
B160	0.02199	0.01979	0.02364	0.0218	0.0019	0.02953	0.02893	0.03219	0.03022	0.00174	0.01639	0.01844	0.01629	0.01704	0.00121
B161	0.011	0.01	0.01	0.0103	0.0006	0.028	0.028	0.029	0.02833	0.00058	0.011	0.013	0.013	0.01233	0.00115
<b>B163</b>	<b>0.119</b>	<b>0.0104</b>	<b>0.0104</b>	<b>0.0466</b>	<b>0.0627</b>	0.0346	0.0319	0.0254	0.03063	0.00473	0.0165	0.0155	0.0155	0.01583	0.00058
B164	<0.001	0.03	<0.001	0.0300		<0.001	0.03	0.032	0.03100	0.00141	<0.001	<0.001	<0.001		
B165															
B167															
<b>B168</b>	0.0196	0.0201	0.0195	0.0197	0.0003	<b>0.0564</b>	<b>0.0559</b>	<b>0.0534</b>	<b>0.05523</b>	<b>0.00161</b>	<b>0.0409</b>	<b>0.0407</b>	<b>0.0408</b>	<b>0.04080</b>	<b>0.00010</b>
B172	<0.031	<0.031	<0.031			<0.031	<0.031	<0.031			<0.031	<0.031	<0.031		
B173	<0.0101	<0.0098	<0.0097			<0.15	<0.1522	<0.1531			<0.0101	<0.0098	<0.0099		
B174	<0.06	<0.06	<0.06			<0.06	<0.06	<0.06			<0.06	<0.06	<0.06		
B175	<0.05	<0.05	<0.05			<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B176	<0.0002	<0.0002	<0.0002			0.036	0.024	0.031	0.03033	0.00603	<0.0002	<0.0002	<0.0002		
B178	0.00987	0.00884	0.00945	0.0094	0.0005	0.0272	0.0279	0.027	0.02737	0.00047	0.0125	0.0119	0.0121	0.01217	0.00031
B181	<0.0077					<0.0077					<0.0077				
<b>B182</b>	<b>0.09</b>	<b>0.09</b>		<b>0.0900</b>		<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B184	<0.049	<0.049	<0.049			<0.049	<0.049	<0.049			<0.049	<0.049	<0.049		

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.00478	0.00094				0.0255	0.0082				0.0104	0.0048
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B185															
B186	<0.0001	<0.0001	<0.0001			0.023	0.024	0.026	0.02433	0.00153	0.01	0.009	0.009	0.00933	0.00058
B187	<0.06	<0.06	<0.06			<0.06	<0.06	<0.06			<0.06	<0.06	<0.06		
B188															
B189	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B190	0.02	0.02	0.02	0.0200		0.03	0.03	0.03	0.03000		0.01	0.01	0.01	0.01000	
B192	<0.0205	<0.0205	<0.0205			0.0238	0.02397	0.02302	0.02360	0.00051	<0.0203	<0.0203	<0.0203		
B195	<0.01	<0.01	<0.01			0.02	0.02	0.021	0.02033	0.00058	<0.01	<0.01	<0.01		
B196															
B198	<0.07	<0.07	<0.07			<0.07	<0.07	<0.07			<0.07	<0.07	<0.07		
B200															
B202	<0.005	<0.005	<0.005			0.0212	0.0214	0.023	0.02187	0.00099	0.0061	0.006	0.00569	0.00593	0.00021
B204	0.01	0.01	0.01	0.0100		0.032	0.029	0.03	0.03033	0.00153	0.014	0.013	0.014	0.01367	0.00058
<b>B205</b>	0.005	0.004	0.002	0.0037	0.0015	<b>0.121</b>	<b>0.105</b>	<b>0.106</b>	<b>0.11067</b>	<b>0.00896</b>	<b>0.037</b>	<b>0.034</b>	<b>0.036</b>	<b>0.03567</b>	<b>0.00153</b>
B206	< 0.01	< 0.01	< 0.01			< 0.02	< 0.02	< 0.02			< 0.01	< 0.01	< 0.01		
B208															
B210															
B212	<0.01	<0.01	<0.01			0.032	0.029	0.027	0.02933	0.00252	0.012	0.014	0.013	0.01300	0.00100
B213	< 0.03	< 0.03	< 0.03			< 0.03	< 0.03	< 0.03			< 0.03	< 0.03	< 0.03		
B215	<0.0039					0.016			0.01600		<0.0038				
B216	<0.025	<0.025	<0.025			<0.025	<0.025	<0.025			<0.025	<0.025	<0.025		
B217	< 0.01	< 0.01	< 0.01			< 0.04	< 0.04	< 0.04			< 0.01	< 0.01	< 0.01		
B219	0.00431	0.00474	0.00435	0.0045	0.0002	0.0257	0.0271	0.0262	0.02633	0.00071	0.0101	0.00946	0.0095	0.00969	0.00036
B220															
B221	0.005	0.005	0.005	0.0050		0.027	0.027	0.031	0.02833	0.00231	0.011	0.011	0.011	0.01100	
B222	<0.04	<0.04	<0.04			<0.04	<0.04	<0.04			<0.05	<0.05	<0.05		
B223	<0.0125	<0.0125	<0.0125			0.0252	0.0252	0.0259	0.02543	0.00040	<0.0125	<0.0125	<0.0125		
B224	0.01	0.01	0.01	0.0100		0.03	0.03	0.03	0.03000		0.01	0.01	0.01	0.01000	
B226	< 0.0102	< 0.0099	< 0.01			< 0.0097	< 0.0098				< 0.0098	< 0.0098	< 0.0099		
B227	< 0.05	< 0.05	< 0.05			< 0.05	< 0.05	< 0.05			< 0.05	< 0.05	< 0.05		
B228	< 0.02	< 0.02	< 0.02			0.04	0.032	0.029	0.03367	0.00569	< 0.02	< 0.02	< 0.02		
B230															
B234	<0.02	<0.02	<0.02			0.03	0.03	0.03	0.03000	0.00000	<0.02	<0.02	<0.02		

**Table B-22. Data summary table for CBG in three marijuana samples.**

Data in red font have been flagged as a data entry of zero or results that include text (e.g., "< LOQ" or "present").

Target Consensus	Plant Sample 2					Plant Sample 3					Plant Sample 5				
	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B032															
B045	detected	detected				detected	detected				detected	detected			
B055	0.0475	0.0509	0.0484	0.0489	0.0018	0.0689	0.0628	0.0644	0.0654	0.0032	0.0834	0.0721	0.0839	0.0798	0.0067
B062	< 0.08	< 0.08	< 0.08			0.094	0.091	0.088	0.0910	0.0030	0.102	0.104	0.099	0.1017	0.0025
B071	<0.005	<0.005	<0.005			<0.005	<0.005	<0.005			<0.005	<0.005	<0.005		
B079															
B099	0.048			0.0480		0.065			0.0650		0.07			0.0700	
B109	< 0.056	< 0.056	< 0.060			< 0.059	< 0.056	< 0.060			0.063	0.062	0.061	0.0620	0.0010
B117	<0.002	<0.002	<0.002			<0.002	<0.002	<0.002			<0.002	<0.002	<0.002		
B125	0.053	0.053	0.052	0.0527	0.0006	0.069	0.069	0.067	0.0683	0.0012	0.074	0.07	0.075	0.0730	0.0026
B146	0.0978	0.0991	0.0967	0.0979	0.0012	0.117	0.114	0.118	0.1163	0.0021	0.12	0.121	0.12	0.1203	0.0006
B148															
B154	0.052	0.05	0.044	0.0487	0.0042	0.065	0.066	0.065	0.0653	0.0006	0.071	0.07	0.082	0.0743	0.0067
B158	0.07	0.06	0.07	0.0667	0.0058	0.08	0.09	0.08	0.0833	0.0058	0.09	0.1	0.09	0.0933	0.0058
B167	0.04	0.04	0.03	0.0367	0.0058	0.05	0.05	0.05	0.0500	0.0000	0.06	0.06	0.05	0.0567	0.0058
B178	0.0582	0.0543	0.053	0.0552	0.0027	0.0745	0.0722	0.0701	0.0723	0.0022	0.0754	0.0787	0.0762	0.0768	0.0017
B196															
B198	0.07	<0.07	< 0.07	0.0700		0.07	0.08	0.08	0.0767	0.0058	0.09	0.09	0.08	0.0867	0.0058
B210															
B213	0.06	0.06	0.06	0.0600	0.0000	0.08	0.09	0.07	0.0800	0.0100	0.09	0.08	0.09	0.0867	0.0058
B222	<0.04	<0.04	<0.04			<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		

**Table B-23. Data summary table for CBGA in three hemp samples.**

Data in **red** font have been flagged as a data entry of zero or results that include text (e.g., “< LOQ” or “present”).

Data in **blue** font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

Note: This table spans multiple pages; the NIST values and consensus values are included on all pages for convenience.

Target Consensus	NRC HEMP-1 (Plant Sample 1)				Plant Sample 4				Plant Sample 6			
	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	0.067	0.018
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	Avg	SD
B012						0.13932	0.14785		0.1436	0.0060	0.06	0.056
B013	<0.028	<0.028	<0.028			0.16	0.17	0.17	0.1667	0.0058	0.08	0.08
<b>B016</b>	<0.0004	<0.0004	<0.0004			0.2859	0.2473	0.2639	0.2657	0.0194	<b>0.1233</b>	<b>0.1274</b>
B018	<0.01	<0.01	<0.01			0.15	0.14	0.17	0.1533	0.0153	0.07	0.07
B024						0.12	0.13	0.11	0.1200	0.0100	0.04	0.05
B027	<0.025	<0.025	<0.025			0.1557	0.1507	0.1481	0.1515	0.0039	0.0562	0.0581
B028											0.0557	0.0567
B036	<0.01	<0.01	<0.01			<0.02	<0.02	<0.02			<0.01	<0.01
B038						0.133	0.151	0.125	0.1363	0.0133		
B043	<0.01					0.136					0.052	
B054	0.00894	0.0096	0.00944	0.0093	0.0003	0.14505	0.14509	0.13688	0.1423	0.0047	0.0558	0.0564
B055	0.0112	0.0143	0.0117	0.0124	0.0017	0.1623	0.1525	0.1596	0.1581	0.0051	0.056	0.0585
B062	<0.08	<0.08	<0.08			0.224	0.222	0.242	0.2293	0.0110	0.116	0.115
B063											0.12	0.1170
B066	<0.003	<0.003	<0.003			0.1494	0.1431	0.1453	0.1459	0.0032	0.0539	0.0605
B073	0.0131			0.0131		0.1244			0.1244		0.0516	
B077						0.226	0.221	0.219	0.2220	0.0036	0.12	0.116
B079											0.12	0.1187
B085	0.016	0.015	0.014	0.0150	0.0010	0.193	0.209	0.141	0.1810	0.0356	0.063	0.058
<b>B092</b>	<0.0002	<0.0002	<0.0002			0.28	0.25	0.23	0.2533	0.0252	<b>0.11</b>	<b>0.13</b>
<b>B096</b>	<b>0.0264</b>	<b>0.0291</b>	<b>0.0255</b>	<b>0.0270</b>	<b>0.0019</b>	0.153	0.179	0.167	0.1663	0.0130	0.074	0.079
B097	<0.01	<0.01	<0.01			0.12	0.12	0.13	0.1233	0.0058	<0.01	<0.01
B099	<0.01					0.17					0.044	
B109	<0.012	<0.012	<0.012			0.156	0.152	0.138	0.1487	0.0095	0.064	0.06
<b>B117</b>	<b>0.026</b>	<b>0.078</b>	<b>0.092</b>	<b>0.0653</b>	<b>0.0348</b>	0.195	0.304	0.285	0.2613	0.0582	<b>0.322</b>	<b>0.206</b>
B125	0.015	0.014	0.014	0.0143	0.0006	0.16	0.151	0.156	0.1557	0.0045	0.069	0.068
<b>B146</b>	<0.0158	<0.0161	<0.0161			0.215	0.266	0.26	0.2470	0.0279	0.107	0.11
B148											0.111	0.1093
B149	<0.009	<0.009	<0.009			0.188	0.179	0.18	0.1823	0.0049	0.073	0.067
B153	<0.0004	<0.0004	<0.0004			0.167	0.165	0.167	0.1663	0.0012	0.08	0.079
B154	0.006	0.006	0.006	0.0060		0.154	0.161	0.189	0.1680	0.0185	0.006	0.052
B157	<0.05					0.18			0.1800		0.08	0.08
B158	<0.05	<0.05	<0.05			0.17	0.16	0.16	0.1633	0.0058	0.07	0.08
B163	0.0116	0.00987	0.011	0.0108	0.0009	0.123	0.109	0.1	0.1107	0.0116	0.057	0.0499
B175	<0.05	<0.05	<0.05			0.16	0.17	0.16	0.1633	0.0058	<0.05	0.06
B178	0.0193	0.0177	0.0174	0.0181	0.0010	0.207	0.207	0.2	0.2047	0.0040	0.0931	0.0877
B181	<0.0077					0.11			0.1100		0.06	

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4				Plant Sample 6					
Target Consensus				0.0117	0.0012				0.160	0.036				0.067	0.018
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B184	<0.027	<0.027	<0.027			0.115	0.12	0.116	0.1170	0.0026	0.059	0.049	0.053	0.0537	0.0050
B186	0.016	0.014	0.015	0.0150	0.0010	0.134	0.132	0.138	0.1347	0.0031	0.063	0.063	0.065	0.0637	0.0012
B193	<0.0990	<0.0894	<0.105			0.134	0.143	0.143	0.1400	0.0052	<0.107	<0.110	<0.106		
B195	0.011	0.011	0.012	0.0113	0.0006	0.158	0.166	0.174	0.1660	0.0080	0.065	0.068	0.072	0.0683	0.0035
B196															
B198	< 0.04	< 0.04	< 0.04			0.17	0.13	0.15	0.1500	0.0200	0.05	0.05	0.05	0.0500	
B204	0.012	0.012	0.012	0.0120	0.0000	0.144	0.129	0.136	0.1363	0.0075	0.057	0.056	0.059	0.0573	0.0015
B210															
B217	< 0.006	< 0.005	< 0.006			0.15	0.14	0.15	0.1467	0.0058	0.06	0.06	0.06	0.0600	
B219	0.0111	0.0115	0.0116	0.0114	0.0003	0.143	0.153	0.151	0.1490	0.0053	0.0596	0.0569	0.0588	0.0584	0.0014
B222	<0.04	<0.04	<0.04			0.07	0.1	0.06	0.0767	0.0208	<0.05	<0.05	<0.05		
B223	0.0143	0.0148	0.0149	0.0147	0.0003	0.165	0.171	0.173	0.1697	0.0042	0.0726	0.0761	0.0733	0.0740	0.0019
B228	< 0.02	< 0.02	< 0.02			0.214	0.193	0.176	0.1943	0.0190	0.092	0.093	0.089	0.0913	0.0021
B234	0.01	0.01	0.01	0.0100	0.0000	0.2	0.18	0.19	0.1900	0.0100	0.08	0.09	0.09	0.0867	0.0058

**Table B-24. Data summary table for CBGA in three marijuana samples.**

Data in blue font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

Target Consensus	Plant Sample 2					Plant Sample 3					Plant Sample 5					
	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	
Lab	A	B	C			A	B	C			A	B	C			
B055	0.284	0.28	0.2805	0.282	0.002	0.3255	0.323	0.3242	0.324	0.001	0.3432	0.3423	0.3414	0.342	0.001	
B062	0.415	0.43	0.444	0.430	0.015	0.491	0.469	0.482	0.481	0.011	0.463	0.456	0.453	0.457	0.005	
B079																
B099	0.236			0.236		0.29			0.290		0.308			0.308		
B109	0.276	0.267	0.269	0.271	0.005	0.317	0.31	0.311	0.313	0.004	0.325	0.322	0.317	0.321	0.004	
B117	0.497	0.554	0.487	0.513	0.036	0.479	0.495	0.51	0.495	0.016	0.49	0.522	0.467	0.493	0.028	
B125	0.301	0.289	0.285	0.292	0.008	0.339	0.326	0.323	0.329	0.009	0.35	0.329	0.341	0.340	0.011	
B146	0.469	0.47	0.463	0.467	0.004	0.501	0.497	0.499	0.499	0.002	0.498	0.507	0.504	0.503	0.005	
B148																
B154	0.292	0.312	0.214	0.273	0.052	0.34	0.343	0.336	0.340	0.004	0.347	0.335	0.362	0.348	0.014	
B158	0.29	0.28	0.29	0.287	0.006	0.32	0.33	0.32	0.323	0.006	0.35	0.34	0.33	0.340	0.010	
B178	0.378	0.364	0.358	0.367	0.010	0.411	0.41	0.4	0.407	0.006	0.414	0.412	0.406	0.411	0.004	
B196																
B198	0.27	0.27	0.22	0.253	0.029	0.34	0.31	0.34	0.330	0.017	0.34	0.35	0.34	0.343	0.006	
B210																
B222	0.33	0.31	0.32	0.320	0.010	0.38	0.31	0.34	0.343	0.035	0.36	0.37	0.36	0.363	0.006	

**Table B-25. Data summary table for CBL in three hemp samples.**

Data in **blue** font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

*Note: This table spans multiple pages; the NIST values and consensus values are included on all pages for convenience.*

Target Consensus	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
				0.0074	0.0014				0.012	0.015				0.0013	0.0017
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B003															
B004															
B005	< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1		
B006	< 0.012	< 0.012	< 0.012			< 0.012	< 0.012	< 0.012			< 0.012	< 0.012	< 0.012		
B015															
B016	< 0.0004	< 0.0004	< 0.0004			< 0.0004	< 0.0004	< 0.0004			< 0.0004	< 0.0004	< 0.0004		
B020															
B026	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B028															
B035	0.0089	0.0081	0.0076	0.0081	0.0006	<0.005	<0.005	<0.005			<0.005	<0.005	<0.005		
B037						<0.01					<0.01				
B043	<0.01														
B048															
<b>B053</b>	<b>0.026</b>	<b>0.026</b>	<b>0.027</b>	<b>0.0263</b>	<b>0.0006</b>	0.002	0.002	0.002	0.0020	0.0000	0.002	0.001	0.002	0.00167	0.00058
B054	0.00979	0.00754	0.00692	0.0081	0.0015	<0.0007	<0.0007	<0.0007			<0.0007	<0.0007	<0.0007		
B058	< 0.0295	< 0.0296	< 0.0296			< 0.0292	< 0.0295	< 0.0298			< 0.0289	< 0.0297	< 0.0296		
B061	0.002	0.002	0.015	0.0063	0.0075	0.01	0.011	0.013	0.0113	0.0015	0.005	0.002	<0.004	0.00350	0.00212
B064	<0.0001					<0.0001					<0.00001				
B069	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
<b>B076</b>	0.01362	0.01437	0.01374	0.0139	0.0004	0.04037	0.04217	0.04034	0.0410	0.0010	<b>0.02782</b>	<b>0.02691</b>	<b>0.02716</b>	<b>0.02730</b>	<b>0.00047</b>
B078															
B079															
B081	<0.39	<0.39	<0.39			<0.39	<0.39	<0.39			<0.39	<0.39	<0.39		
B082	0.011	0.007	0.011	0.0096	0.0023	<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B084															
B095	< 0.002	< 0.002	< 0.002			< 0.002	< 0.002	< 0.002			< 0.002	< 0.002	< 0.002		
B097	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B099	<0.01					<0.01					<0.01				
B100	<0.01	<0.01				<0.01	<0.01				<0.01	<0.01			
B102	< 0.0001	< 0.0001	< 0.0001			< 0.0001	< 0.0001	< 0.0001			< 0.0001	< 0.0001	< 0.0001		
B111															
B114															
B115															
B116	0.0103	0.0101	0.0098	0.0100	0.0002	0.0026	0.0022	0.0021	0.0023	0.0003	0.0014	0.0012	0.0012	0.00127	0.00012
B117	<0.002	<0.002	<0.002			<0.002	<0.002	<0.002			<0.002	<0.002	<0.002		
B125	<0.01	<0.01	<0.01			0.011	0.011	0.01	0.0107	0.0006	<0.01	<0.01	<0.01		
B132	0.012	0.012	0.012	0.0120	0.0015	0.001	0.001	0.001	0.0010		0.001	0.001	0.001	0.00100	
<b>B137</b>	0.012	0.014	0.015	0.0137	0.0015	0.028	0.021	0.022	0.0237	0.0038	<b>0.023</b>	<b>0.023</b>	<b>0.026</b>	<b>0.02400</b>	<b>0.00173</b>

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4				Plant Sample 6					
Target Consensus				0.0074	0.0014				0.012	0.015				0.0013	0.0017
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B141	0.007	0.007	0.007	0.0070		<0.0045	<0.0045	<0.0045			<0.0045	<0.0045	<0.0045		
B142	<0.0007	<0.0007	<0.0007			<0.0075	<0.0075	<0.0075			<0.0007	<0.0007	<0.0007		
B146	< 0.0158	< 0.0161	< 0.0161			< 0.0161	< 0.0159	< 0.0159			< 0.0159	< 0.0161	< 0.0161		
B147	<0.016	<0.016	<0.016			<0.016	<0.016	<0.016			<0.016	<0.016	<0.016		
B148															
B154	<0.045	<0.045	<0.045			<0.045	<0.045	<0.045			<0.045	<0.045	<0.045		
B158	<0.05	<0.05	<0.05			<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B159	<0.0099	<0.0100	<0.0098			<0.0102	<0.0101	<0.0099			<0.0097	<0.0095	<0.0102		
<b>B161</b>	<b>0.035</b>	<b>0.034</b>	<b>0.033</b>	<b>0.0340</b>	<b>0.0010</b>	<b>0.068</b>	<b>0.064</b>	<b>0.064</b>	<b>0.0653</b>	<b>0.0023</b>	<b>0.028</b>	<b>0.03</b>	<b>0.035</b>	<b>0.03100</b>	<b>0.00361</b>
B163	0.0112	0.0097	0.0096	0.0102	0.0009	0.0009	0.0012	0.0009	0.0010	0.0002	0.0009	0.0009	0.0009	0.0009	0.00004
B164	<0.001	<0.001	<0.001			<0.001	0.04	0.03	0.0350	0.0071	<0.001	<0.001	<0.001		
B165															
B174															
B178	<0.0011	<0.0011	<0.0011			<0.0011	<0.0011	<0.0011			<0.0011	<0.0011	<0.0011		
B185															
B187															
B192	<0.0205	<0.0205	<0.0205			<0.0202	<0.0202	<0.0202			<0.0203	<0.0203	<0.0203		
B193	<0.0878	<0.0792	<0.0931			<0.0903	<0.0832	<0.0881			<0.0949	<0.0977	<0.0940		
B200															
B202	0.00644	0.00714	0.00676	0.0068	0.0004	<0.005	<0.005	<0.005			<0.005	<0.005	<0.005		
B204	0.008	0.008	0.008	0.0080	0.0000	<0.002	<0.002	<0.002			<0.002	<0.002	<0.002		
B208															
B210															
B219	0.00715	0.00769	0.0072	0.0074	0.0003	0.00051	0.00052	0.00053	0.0005		0.00046	0.00041	0.00043	0.00043	0.00003
B221	0.009	0.009	0.009	0.0090		0.0005	0.0005	0.0005	0.0005		0.00040	0.00050	0.00050	0.00047	0.00006
B222	<0.04	<0.04	<0.04			<0.04	<0.04	<0.04			<0.05	<0.05	<0.05		
B224	0.01	0.01	0.01	0.0100		<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B230															
B234	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		

**Table B-26. Data summary table for CBL in three marijuana samples.**

Target Consensus	Plant Sample 2					Plant Sample 3					Plant Sample 5					
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B079																
B099	<0.01						<0.01					<0.01				
B117	<0.002	<0.002	<0.002				<0.002	<0.002	<0.002			<0.002				
B125	0.022	0.022	0.023				0.02	0.02	0.02	0.02000		0.021	0.022	0.022	0.022	0.001
B146	< 0.0159	< 0.0161	< 0.0159				< 0.0161	< 0.0161	< 0.0161			< 0.0160	< 0.0160	< 0.0160		
B148																
B154	<0.045	<0.045	<0.045				<0.045	<0.045	<0.045			<0.045	<0.045	<0.045		
B158	<0.05	<0.05	<0.05				<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B178	<0.0011	<0.0011	<0.0011				<0.0011	<0.0011	<0.0011			<0.0011	<0.0011	<0.0011		
B210																
B222	<0.04	<0.04	<0.04				<0.05	<0.05	<0.05			0.05	<0.05	0.06	0.055	0.007

**Table B-27. Data summary table for CBLA in three hemp samples.**

Data in **red** font have been flagged as a data entry of zero or results that include text (e.g., “< LOQ” or “present”).

Data in **blue** font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

*Note: This table spans multiple pages; the NIST values and consensus values are included on all pages for convenience.*

Target Consensus	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B001	0.082	0.085	0.081	0.0827	0.0021				0.013	0.012				0.0114	0.0089
B003				0.0187	0.0018										
B004				0.032	0.014										
B006	0.027	0.027	0.028	0.0273	0.0006	0.023	0.022	0.018	0.0210	0.0026	0.019	0.02	0.019	0.0193	0.0006
B007															
B009															
B015	0.02	0.02	0.02	0.0200	0.0000										
B020															
B028															
B035	0.04109	0.04341	0.04326	0.0426	0.0013	0.01512	0.01674	0.01663	0.0162	0.0009	0.01645	0.01708	0.01696	0.0168	0.0003
B037															
B043	<0.01					<0.01									
B048															
B053	0.158	0.166	0.156	0.1600	0.0053	0.603	0.628	0.623	0.6180	0.0132	0.422	0.425	0.443	0.4300	0.0114
B054	0.02113	0.01974	0.01946	0.0201	0.0009	0.00918	0.00958	0.00939	0.0094	0.0002	0.0092	0.00889	0.00881	0.0090	0.0002
B058															
B064	<0.0001					<0.0001									
B070															
B076	0.02318	0.02255	0.02179	0.0225	0.0007	0.00826	0.00864	0.00859	0.0085	0.0002	0.00828	0.00862	0.00862	0.0085	0.0002
B078															
B079															
B084															
B097	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B099	<0.01					<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B102	0.0219	0.0206	0.0203	0.0209	0.0009	0.0092	0.0097	0.0123	0.0104	0.0017	0.0107	0.0104	0.0102	0.0104	0.0003
B111															
B114															
B115	0.0386	0.0441	0.0424	0.0417	0.0028										
B117	<0.003	<0.003	<0.003			<0.003	<0.003	<0.003			<0.003	<0.003	<0.003		
B125	0.052	0.056	0.054	0.0540	0.0020	0.034	0.037	0.046	0.0390	0.0062	0.023	0.023	0.025	0.0237	0.0012
B132															
B137	0.058	0.049	0.057	0.0547	0.0049	0.007	0.006	0.007	0.0067	0.0006	0.008	0.008	0.008	0.0080	
B141	<0.0045	<0.0045	<0.0045			<0.0045	<0.0045	<0.0045			<0.0045	<0.0045	<0.0045		
B142															
B146	0.0333	0.0309	0.0356	0.0333	0.0024	0.0267	0.0247	0.0254	0.0256	0.0010	0.017	0.0179	0.0188	0.0179	0.0009
B148															
B158	<0.05	<0.05	<0.05			<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4				Plant Sample 6					
Target Consensus				0.0187 0.032	0.0018 0.014				0.013	0.012				0.0114 0.0089	
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B161	0.024	0.027	0.026	0.0257	0.0015	0	0	0	0.0000		0.012	0	0	0.0040	0.0069
B165															
B174															
B178	0.0255	0.025	0.0247	0.0251	0.0004	0.0112	0.011	0.0107	0.0110	0.0003	0.0122	0.0109	0.0123	0.0118	0.0008
B185															
B193	<0.187	<0.169	<0.199			<0.192	<0.177	<0.188			<0.202	<0.208	<0.200		
B200															
B202															
B208															
B210															
B219	0.0196	0.0204	0.0197	0.0199	0.0004	0.00723	0.00768	0.00742	0.0074	0.0002	0.00791	0.00741	0.00749	0.0076	0.0003
B221															
B230															

**Table B-28. Data summary table for CBLA in three marijuana samples.**

Target Consensus	Plant Sample 2					Plant Sample 3					Plant Sample 5				
	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B079	<0.01					<0.01					<0.01				
B099	<0.003	<0.003	<0.003			<0.003	<0.003	<0.003			<0.003	<0.003	<0.003		
B117	0.062	0.073	0.075	0.070	0.007	0.062	0.064	0.064	0.063	0.001	0.08	0.088	0.088	0.085	0.005
B125	0.0498	0.0511	0.0498	0.050	0.001	0.0488	0.0479	0.047	0.048	0.001	0.06	0.0601	0.0609	0.060	0.000
B146	0.0196	0.0189	0.0197	0.019	0.000	0.0184	0.0178	0.0188	0.018	0.001	0.0176	0.0171	0.017	0.017	0.000
B148															
B158															
B178															
B210															

**Table B-29. Data summary table for CBN in three hemp samples.**

Data in red font have been flagged as a data entry of zero or results that include text (e.g., "< LOQ" or "present").

Data in blue font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

*Note: This table spans multiple pages; the NIST values and consensus values are included on all pages for convenience.*

Target Consensus	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B001	0.044	0.046	0.044	0.0447	0.0012										
B003	0.05053	0.05177	0.04854	0.0503	0.0016										
<b>B004</b>	0.046	0.046	0.046	0.0460	0.0000	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.0000</b>	<b>0.0000</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.0000</b>	<b>0.0000</b>
B005	< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1		
B006	0.045	0.046	0.046	0.0457	0.0006	< 0.012	< 0.012	< 0.012			< 0.012	< 0.012	< 0.012		
B007	0.05	0.05	0.05	0.0500	0.0000										
B009															
B012	0.04343	0.04325		0.0433	0.0001										
B013	0.05	0.05	0.05	0.0500	0.0000	< 0.057	< 0.059	< 0.060			< 0.028	< 0.028	< 0.028		
B014															
B015	0.04	0.04	0.04	0.0400	0.0000										
<b>B016</b>	<b>0.082</b>	<b>0.0798</b>	<b>0.0709</b>	<b>0.0776</b>	<b>0.0059</b>	< 0.0004	< 0.0004	< 0.0004			< 0.0004	< 0.0004	< 0.0004		
B018	0.04	0.04	0.04	0.0400	0.0000	< 0.01	< 0.01	< 0.01			< 0.01	< 0.01	< 0.01		
B020															
B021															
B022	0.052	0.05	0.051	0.0510	0.0010	< 0.04	< 0.04	< 0.04			< 0.04	< 0.04	< 0.04		
B023	0.05	0.0509	0.0488	0.0499	0.0011	< 0.0037	< 0.0037	< 0.0037			< 0.0037	< 0.0037	< 0.0037		
<b>B024</b>	<b>0.03</b>	<b>0.04</b>	<b>0.02</b>	<b>0.0300</b>	<b>0.0100</b>										
B026	0.04	0.05	0.05	0.0467	0.0058	< 0.01	< 0.01	< 0.01			< 0.01	< 0.01	< 0.01		
B027	0.0502	0.0493	0.0484	0.0493	0.0009	< 0.025	< 0.025	< 0.025			< 0.025	< 0.025	< 0.025		
B028															
B029	0.044	0.046	0.041	0.0437	0.0025	< 0.030	< 0.030	< 0.030			< 0.030	< 0.030	< 0.030		
B030	0.05	0.05	0.05	0.0500		< 0.02	< 0.02	< 0.02			< 0.02	< 0.02	< 0.02		
B031															
B032															
B033	< 0.5	< 0.5	< 0.5			< 0.5	< 0.5	< 0.5			< 0.5	< 0.5	< 0.5		
B035	0.04528	0.04558	0.04584	0.0456	0.0003	< 0.005	< 0.005	< 0.005			< 0.005	< 0.005	< 0.005		
B036	0.03	0.04	0.03	0.0333	0.0058	< 0.02	< 0.02	< 0.02			< 0.01	< 0.01	< 0.01		
B037															
B038															
B041	< 0.064					< 0.064					< 0.064				
B042															
B043	0.047					< 0.01					< 0.01				
B044	< 0.075	< 0.075	< 0.075			< 0.025	< 0.025	< 0.025			< 0.025	< 0.025	< 0.025		
<b>B045</b>	<b>detected</b>	<b>detected</b>	<b>detected</b>			<b>not detected</b>	<b>not detected</b>	<b>not detected</b>			<b>not detected</b>	<b>not detected</b>	<b>not detected</b>		
B047	0.056	0.055	0.053	0.0547	0.0015	< 0.01	< 0.01	< 0.01			< 0.01	< 0.01	< 0.01		
B048															

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.0490	0.0070				0.0017	0.0030				0.0013	0.0020
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B049	0.04156	0.0425	0.04169	0.0419	0.0005										
<b>B051</b>	0.04	0.04	0.05	0.0433	0.0058	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.0000</b>	<b>0.0000</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.0000</b>	<b>0.0000</b>
B052	< 0.10	< 0.10	< 0.10			< 0.10	< 0.10	< 0.10			< 0.10	< 0.10	< 0.10		
<b>B053</b>	<b>0.201</b>	<b>0.207</b>	<b>0.211</b>	<b>0.2063</b>	<b>0.0050</b>	0.006	0.006	0.006	0.0060	0.0000	0.005	0.005	0.005	0.0050	0.0000
B054	0.04447	0.04426	0.0424	0.0437	0.0011	0.00034	0.00034	0.00033	0.0003	0.0000	<0.0001	<0.0001	<0.0001		
B055	0.0486	0.0513	0.0495	0.0498	0.0014	<0.0217	<0.0217	<0.0217			<0.0217	<0.0217	<0.0217		
<b>B056</b>	<b>0.109</b>	<b>0.106</b>		<b>0.1075</b>	<b>0.0021</b>	<b>0.014</b>	<b>0.015</b>		<b>0.0145</b>	<b>0.0007</b>	<b>0.013</b>	<b>0.013</b>		<b>0.0130</b>	<b>0.0000</b>
B058	0.0498	0.0491	0.0484	0.0491	0.0007	< 0.0292	< 0.0295	< 0.0298			< 0.0289	< 0.0297	< 0.0296		
B060	0.04	0.04	0.05	0.0433	0.0058										
B061	0.047	0.048	0.05	0.0483	0.0015	<0.004	<0.004	<0.004			<0.004	<0.004	<0.004		
B062	< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08		
B063															
B064	<0.0001					<0.0001					<0.0001				
B065	0.051	0.053	0.052	0.0520	0.0010	0.0015	0.0015	0.0017	0.0016	0.0001	0.0011	0.0011	0.001	0.0011	0.0001
B066	0.0478	0.0493	0.0506	0.0492	0.0014	<0.004	<0.004	<0.004			<0.004	<0.004	<0.004		
B068															
B069	0.05	0.05	0.05	0.0500		<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B070	0.044	0.069	0.047	0.0533	0.0137	<0.111	<0.111	<0.111			<0.067	<0.067	<0.067		
<b>B071</b>	0.05	0.04	0.02	0.0367	0.0153	0.01	0.01	0.01	0.0100	0.0000	<b>0.01</b>	<b>0.01</b>	<b>0.01</b>	<b>0.0100</b>	<b>0.0000</b>
B072	0.051	0.05	0.052	0.0510	0.0010	<0.005	<0.005	<0.005			<0.005	<0.005	<0.005		
B073	0.0431			0.0431		<0.0031					<0.0031				
B074	0.05	0.05	0.05	0.0500	0.0000	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B076	0.05207	0.05131	0.05183	0.0517	0.0004	0.00249	0.00253	0.00262	0.0025	0.0001	0.00206	0.002	0.00201	0.0020	0.0000
B077															
B078															
B079															
B081	<0.2	<0.2	<0.2			<0.2	<0.2	<0.2			<0.2	<0.2	<0.2		
B082	0.05	0.053	0.053	0.0520	0.0017	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
<b>B084</b>	0.05	0.05	0.05	0.0500	0.0000	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.0000</b>	<b>0.0000</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.0000</b>	<b>0.0000</b>
B085	0.058	0.061	0.055	0.0580	0.0030	0.002	0.002	0.002	0.0020	0.0000	< 0.002	< 0.002	< 0.002		
B086															
B088	0.048	0.051	0.048	0.0490	0.0017	<0.013	<0.013	<0.013			<0.013	<0.013	<0.013		
B089	0.048	0.0496	0.0491	0.0489	0.0008	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
<b>B090</b>	<0.05	0.05	<0.05	0.0500		<b>0</b>	<b>0</b>	<b>0</b>	<b>0.0000</b>		<b>0</b>	<b>0</b>	<b>0</b>	<b>0.0000</b>	
B091	0.051	0.051	0.052	0.0513	0.0006	<0.118	<0.118	<0.118			<0.065	<0.065	<0.065		
B094	0.05	0.05	0.05	0.0500		< 0.02	< 0.02	< 0.02			< 0.02	< 0.02	< 0.02		
B095	0.06	0.061	0.06	0.0603	0.0006	< 0.002	< 0.002	< 0.002			< 0.002	< 0.002	< 0.002		
<b>B096</b>	0.051	0.056	0.049	0.0520	0.0036	<1	<1	<1			<1	<b>0.083</b>	<1		<b>0.0830</b>
B097	0.05	0.05	0.05	0.0500	0.0000	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B098															
<b>B099</b>	0.051			0.0510		<0.01					<0.01				
B100	0.052	0.047		0.0495	0.0035	<0.007	<0.007	<0.007			<0.007	<0.007	<0.007		
B102	0.0433	0.0416	0.0413	0.0421	0.0011	< 0.0001	< 0.0001	< 0.0001			< 0.0001	< 0.0001	< 0.0001		

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.0490	0.0070				0.0017	0.0030				0.0013	0.0020
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B104	<0.25	<0.25	<0.25			<0.25	<0.25	<0.25			<0.25	<0.25	<0.25		
B105															
B106	0.045	0.044	0.044	0.0443	0.0006	<0.001	<0.001	<0.001			<0.001	<0.001	<0.001		
B108															
B109	0.052	0.05	0.049	0.0503	0.0015	< 0.012	< 0.012	< 0.011			< 0.012	< 0.012	< 0.012		
B110	0.045	0.046	0.045	0.0453	0.0006	<0.02	<0.02	<0.02			<0.02	<0.02	<0.02		
B111															
B113	0.05	0.05	0.05	0.0500		0	0	0	0.0000		0.01	0	0	0.0033	0.0058
B114															
B115	0.045	0.047	0.049	0.0470	0.0020										
B116	0.0481	0.0481	0.0486	0.0483	0.0003	0.0015	0.0013	0.0014	0.0014	0.0001	0.00090	0.00090	0.00080	0.0009	0.0001
B117	0.009	0.011	0.009	0.0097	0.0012	<0.002	<0.002	<0.002			<0.002	<0.002	<0.002		
B120															
B122															
B124	0.046	0.047	0.048	0.0470	0.0010						<0.01	<0.01	<0.01		
B125	0.045	0.045	0.044	0.0447	0.0006	<0.01	<0.01	<0.01							
B126	0.026	0.027	0.025	0.0260	0.0010										
B127	0.0466	0.0483	0.0475	0.0475	0.0009	<0.010	<0.010	<0.010			<0.010	<0.010	<0.010		
B129															
B130	0.05	0.05	0.04	0.0467	0.0058	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B131	0.0468	0.0455	0.0434	0.0452	0.0017	0.0808	0.0938	0.0785	0.0844	0.0083	0.0256	0.0259	0.0264	0.0260	0.0004
B132	0.083	0.079	0.082	0.0813	0.0021	0.002	0.002	0.001	0.0017	0.0006	0.001	0.001	0.001	0.0010	
B136															
B137	0.05	0.048	0.051	0.0497	0.0015	0.028	0.024	0.029	0.0270	0.0026	0.016	0.014	0.022	0.0173	0.0042
B141	0.048	0.046	0.047	0.0470	0.0010	<0.0045	<0.0045	<0.0045			<0.0045	<0.0045	<0.0045		
B142	0.052	0.051	0.05	0.0510	0.0010	<0.0075	<0.0075	<0.0075			<0.0007	<0.0007	<0.0007		
B144	0.03	<0.001	0.03	0.0300		0.031	0.027	0.024	0.0273	0.0035	<0.001	<0.001	<0.001		
B146	0.045	0.043	0.0444	0.0441	0.0010	<0.0161	<0.0159	<0.0159			<0.0159	<0.0161	<0.0161		
B147	0.046	0.048	0.053	0.0490	0.0036	<0.016	<0.016	<0.016			<0.016	<0.016	<0.016		
B148															
B149	0.04	0.041	0.037	0.0393	0.0021	<0.0205	<0.0205	<0.0205			<0.0205	<0.0205	<0.0205		
B151	<0.040	<0.040	<0.040			<0.040	<0.040	<0.040			<0.040	<0.040	<0.040		
B152															
B153	0.07	0.072	0.073	0.0717	0.0015	<0.0005	<0.0005	<0.0005			<0.0005	<0.0005	<0.0005		
B154	0.051	0.053	0.049	0.0510	0.0020	<0.045	<0.045	<0.045			<0.045	<0.045	<0.045		
B157	0.05			0.0500		<0.05					<0.05	<0.05			
B158	0.05	0.05	0.05	0.0500	0.0000	<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B159	0.0485	0.0484	0.0473	0.0481	0.0007	<0.0102	<0.0101	<0.0099			<0.0097	<0.0095	<0.0102		
B160	0.04765	0.04552	0.04559	0.0463	0.0012	0.0027	0.00266	0.00253	0.0026	0.0001	0.00215	0.00198	0.00193	0.0020	0.0001
B161	0.054	0.052	0.054	0.0533	0.0012	0	0	0	0.0000		0	0	0	0.0000	
B163	0.0661	0.0551	0.0557	0.0590	0.0062	0.00224	0.00221	0.00168	0.0020	0.0003	0.00198	0.00186	0.00184	0.0019	0.0001
B164	0.06	0.07	0.07	0.0667	0.0058	<0.001	<0.001	<0.001			<0.001	<0.001	<0.001		
B165															

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.0490	0.0070				0.0017	0.0030				0.0013	0.0020
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B167															
B168	0.0502	0.0504	0.0503	0.0503	0.0001	0.00179	0.00227	0.00222	0.0021	0.0003	< 0.01	< 0.01	< 0.01	0.0017	0.0000
B172	0.04416	0.04048	0.04072	0.0418	0.0021	< 0.031	< 0.031	< 0.031			< 0.031	< 0.031	< 0.031		
B173	0.04523	0.04741	0.04455	0.0457	0.0015	< 0.01	< 0.0102	< 0.0102			< 0.0101	< 0.0099	< 0.0099		
B174	<0.06	<0.06	<0.06			<0.06	<0.06	<0.06			<0.06	<0.06	<0.06		
B175	0.06	0.06	0.06	0.0600		<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B176	0.049	0.039	0.043	0.0437	0.0050	<0.0002	<0.0002	<0.0002			<0.0002	<0.0002	<0.0002		
B178	0.0525	0.0515	0.0504	0.0515	0.0011	<0.0006	<0.0006	<0.0006			<0.0006	<0.0006	<0.0006		
B181	0.05			0.0500		<0.0077					<0.0077				
B182	<0.05	<0.05				<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B183	0.0477	0.0501	0.0472	0.0483	0.0016	<0.06	<0.06	<0.06			<0.06	<0.06	<0.06		
B184	<0.035	<0.035	<0.035			<0.035	<0.035	<0.035			<0.035	<0.035	<0.035		
B185															
B186	0.049	0.047	0.047	0.0477	0.0012	0.002	0.002	0.002	0.0020		0.001	0.001	0.001	0.0010	
B187	0.0614	0.0617	0.0614	0.0615	0.0002										
B188															
B189	0.0662	0.0655	0.0657	0.0658	0.0004	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B190	0.05	0.05	0.05	0.0500											
B192	0.05418	0.05324	0.05341	0.0536	0.0005	<0.0202	<0.0202	<0.0202			<0.0203	<0.0203	<0.0203		
B193	<0.0925	0.174	<0.0982	0.1740		<0.0951	<0.0877	<0.0928			<0.100	<0.103	<0.0991		
B195	0.049	0.047	0.048	0.0480	0.0010	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B196															
B198	<0.07	<0.07	<0.07			<0.07	<0.07	0.07	0.0700		<0.07	<0.07	<0.07		
B199	0.04	0.04	0.04	0.0400		0	0	0	0.0000		0	0	0	0.0000	
B200															
B202	0.0489	0.0467	0.0479	0.0478	0.0011	<0.005	<0.005	<0.005			<0.005	<0.005	<0.005		
B204	0.048	0.048	0.048	0.0480		<0.002	<0.002	<0.002			<0.002	<0.002	<0.002		
B205	0.089	0.086	0.083	0.0860	0.0030	0.027	0.029	0.026	0.0273	0.0015	0.016	0.017	0.016	0.0163	0.0006
B206	0.05	0.05	0.049	0.0497	0.0006	<0.02	<0.02	<0.02			<0.01	<0.01	<0.01		
B208															
B210															
B212	0.052	0.052	0.051	0.0517	0.0006	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B213	0.06	0.05	0.06	0.0567	0.0058	<0.03	<0.03	<0.03			<0.03	<0.03	<0.03		
B215	0.04			0.0400		<0.0039					<0.0038				
B216	0.043	0.04	0.04	0.0410	0.0017	<0.017	<0.017	<0.017			<0.017	<0.017	<0.017		
B217	0.05	0.05	0.05	0.0500		<0.007	<0.007	<0.007			<0.008	<0.007	<0.007		
B219	0.0489	0.0495	0.0496	0.0493	0.0004	0.00224	0.00231	0.00218	0.0022	0.0001	0.00145	0.00132	0.00135	0.0014	0.0001
B220															
B221	0.059	0.061	0.061	0.0603	0.0012	0.002	0.002	0.002	0.0020	0.0000	0.002	0.002	0.002	0.0020	0.0000
B222	<0.05	<0.05	<0.05			<0.04	<0.04	<0.04			<0.05	<0.05	<0.05		
B223	0.0456	0.0456	0.0458	0.0457	0.0001	<0.0125	<0.0125	<0.0125			<0.0125	<0.0125	<0.0125		
B224	0.06	0.06	0.06	0.0600		<0.02	<0.02	<0.02			<0.01	<0.01	<0.01		
B226	0.04721	0.04903	0.04644	0.0476	0.0013	<0.0097	<0.0098				<0.0098	<0.0098	<0.0098		

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4				Plant Sample 6					
Target Consensus				0.0490 0.0485	0.0070 0.0058				0.0017 0.0030				0.0013 0.0020		
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B227	0.07	0.08	0.07	0.0733	0.0058	<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B228	0.059	0.058	0.058	0.0583	0.0006	< 0.02	< 0.02	< 0.02			< 0.02	< 0.02	< 0.02		
B230															
B234	0.02	0.02	0.02	0.0200	0.0000	<0.003	<0.003	<0.003			<0.003	<0.003	<0.003		

**Table B-30. Data summary table for CBN in three marijuana samples.**

Data in red font have been flagged as a data entry of zero or results that include text (e.g., “< LOQ” or “present”).

Data in blue font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

Target Consensus	Plant Sample 2					Plant Sample 3					Plant Sample 5					
	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD		
Lab	A	B	C		A	B	C	A	B	C	A	B	C	Avg	SD	
B032																
B042																
<b>B045</b>	not detected	not detected				detected	detected				detected	detected				
B055	0.0041	0.0041	0.004	0.0041	0.0001	0.0313	0.032	0.0316	0.0316	0.0004	0.0731	0.0719	0.0708	0.0719	0.0012	
B062	< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08			
<b>B071</b>	0.02	0.02	0.02	0.0200	0.0000	0.04	0.04	0.04	0.0400	0.0000	0.07	0.07	0.06	0.0667	0.0058	
B079																
B086																
<b>B099</b>	0.043			0.0430		0.071			0.0710		0.074			0.0740		
B109	< 0.056	< 0.056	< 0.060			< 0.059	< 0.056	< 0.060			0.064	0.065	0.063	0.0640	0.0010	
<b>B117</b>	<0.002	<0.002	0.007	0.0070		0.011	0.012	0.012	0.0117	0.0006	0.02	0.026	0.024	0.0233	0.0031	
B125	<0.01	<0.01	<0.01			0.03	0.03	0.028	0.0293	0.0012	0.064	0.064	0.066	0.0647	0.0012	
B146	< 0.0159	< 0.0161	< 0.0159			0.0291	0.0271	0.0289	0.0284	0.0011	0.0643	0.0653	0.0643	0.0646	0.0006	
B148																
B151	< 0.040	< 0.040	< 0.040			< 0.040	< 0.040	< 0.040			< 0.040	< 0.040	< 0.040			
B154	<0.045	<0.045	<0.045			0.034	0.036	0.035	0.0350	0.0010	0.069	0.07	0.07	0.0697	0.0006	
B158	<0.05	<0.05	<0.05			<0.05	<0.05	<0.05			0.08	0.07	0.07	0.0733	0.0058	
B167						0.03	0.03	0.02	0.0267	0.0058	0.06	0.06	0.06	0.0600	0.0000	
B178	0.00462	0.00443	0.00407	0.0044	0.0003	0.0308	0.032	0.0306	0.0311	0.0008	0.0684	0.0684	0.0672	0.0680	0.0007	
B196																
<b>B198</b>	<0.07	<0.07	<0.07			<0.07	<0.07	<0.07			0.07	0.07	0.08	0.0733	0.0058	
B199	0.01	0.01	0.01	0.0100	0.0000	0.03	0.03	0.03	0.0300		0.06	0.06	0.06	0.0600	0.0000	
B210																
B213	< 0.03	< 0.03	< 0.03			0.04	0.04	0.04	0.0400	0.0000	0.08	0.08	0.09	0.0833	0.0058	
B222	<0.04	<0.04	<0.04			<0.04	<0.04	<0.04			0.06	0.06	0.06	0.0600		

**Table B-31. Data summary table for CBNA in three hemp samples.**

Data in **red** font have been flagged as a data entry of zero or results that include text (e.g., "< LOQ" or "present").

Data in **blue** font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

*Note: This table spans multiple pages; the NIST values and consensus values are included on all pages for convenience.*

Target Consensus	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6					
				0.0350 0.0370	0.0036 0.0082				0.0057	0.0054				0.0070 0.0052		
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B003																
B004																
B005	< 0.1 < 0.012	< 0.1 < 0.012	< 0.1 < 0.012				< 0.1 < 0.011	< 0.1 < 0.011	< 0.1 < 0.011			< 0.1 < 0.011	< 0.1 < 0.011	< 0.1 < 0.011		
B006																
B007																
B009																
B015	0.03	0.03	0.03	0.0300	0.0000											
B020																
B023	0.0374	0.038	0.0369	0.0374	0.0006	<0.0148	<0.0148	<0.0147			<0.0149	<0.0149	<0.0149			
B026	0.03	0.03	0.04	0.0333	0.0058	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			
B028																
B030	0.04	0.04	0.04	0.0400		<0.03	<0.03	<0.03			<0.03	<0.03	<0.03			
B035	0.03617	0.03646	0.03697	0.0365	0.0004	0.00838	0.00823	0.00747	0.0080	0.0005	0.00684	0.00766	0.00691	0.0071	0.0005	
B037																
B043	0.028			0.0280		<0.01					0.003			0.0030		
B048																
<b>B051</b>	0.03	0.04	0.03	0.0333	0.0058	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.0000</b>	<b>0.0000</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.0000</b>	<b>0.0000</b>	
<b>B053</b>	<b>0.109</b>	<b>0.122</b>	<b>0.12</b>	<b>0.1170</b>	<b>0.0070</b>	0.017	0.015	0.017	0.0163	0.0012	0.018	0.018	0.018	0.0180	0.0000	
B054	0.02893	0.02882	0.02925	0.0290	0.0002	0.00502	0.00488	0.0046	0.0048	0.0002	0.00406	0.00413	0.00386	0.0040	0.0001	
B058																
B064	<0.0001					<0.0001					<0.0001					
B069	0.04	0.04	0.05	0.0433	0.0058	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			
B070	0.037	0.034	0.039	0.0367	0.0025	<0.111	<0.111	<0.111			<0.067	<0.067	<0.067			
B078																
B079																
B081	<0.28	<0.28	<0.28			<0.28	<0.28	<0.28			<0.28	<0.28	<0.28			
<b>B082</b>	0.039	0.042	0.035	0.0387	0.0035	<b>0.051</b>	<b>0.043</b>	<b>0.037</b>	<b>0.0437</b>	<b>0.0070</b>	0.019	0.019	0.017	0.0183	0.0012	
<b>B084</b>	0.04	0.04	0.04	0.0400	0.0000	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.0000</b>	<b>0.0000</b>	<b>0.00</b>	<b>0.01</b>	<b>0.005</b>	0.0050	0.0050	
B085	0.058	0.05	0.045	0.0510	0.0066	0.008	0.007	0.007	0.0073	0.0006	0.006	0.005	0.005	0.0053	0.0006	
B097	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			
B099	0.057			0.0570		<0.01	<0.01	<0.01			0.021			0.0210		
B102	0.0306	0.0286	0.0284	0.0292	0.0012	< 0.0001	< 0.0001	< 0.0001			< 0.0001	< 0.0001	< 0.0001			
B111																
B114																
B115	0.034	0.037	0.039	0.0367	0.0025											
<b>B117</b>	<b>0.087</b>	<b>0.091</b>	<b>0.095</b>	<b>0.0910</b>	<b>0.0040</b>	<0.002	<0.002	<0.002			<0.003	<0.003	<0.003			
B125	0.028	0.029	0.029	0.0287	0.0006	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.0350	0.0036				0.0057	0.0054				0.0070	0.0052
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B132	0.052	0.047	0.05	0.0497	0.0025	0.008	0.008	0.006	0.0073	0.0012	0.007	0.007	0.007	0.0070	
B137	0.106	0.1	0.104	0.1033	0.0031	0.029	0.029	0.032	0.0300	0.0017	0.022	0.022	0.008	0.0173	0.0081
B141	<0.0045	<0.0045	<0.0045			0.008	0.011	0.007	0.0087	0.0021	<0.0045	0.063	<0.0045	0.0630	
B142															
B146	0.0282	0.0286	0.0284	0.0284	0.0002	< 0.0161	< 0.0159	< 0.0159			< 0.0159	< 0.0161	< 0.0161		
B148															
B154	0.032	0.032	0.03	0.0313	0.0012	0.254	0.19	0.241	0.2283	0.0338	<0.045	0.149	0.173	0.1610	0.0170
B158	0.05	0.05	0.05	0.0500	0.0000	<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B161	0.038	0.037	0.037	0.0373	0.0006	0	0	0	0.0000		0	0	0	0.0000	
B163	0.0501	0.0401	0.0391	0.0431	0.0061	0.0062	0.00591	0.00429	0.0055	0.0010	0.00595	0.00557	0.00552	0.0057	0.0002
B165															
B173	0.03406	0.03586	0.03225	0.0341	0.0018	< 0.01	< 0.0101	< 0.0102			< 0.0101	< 0.0098	< 0.0099		
B174															
B175	<0.05	<0.05	<0.05			<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B178	0.04	0.0394	0.0373	0.0389	0.0014	0.00621	0.00663	0.0068	0.0065	0.0003	0.00613	0.00583	0.00609	0.0060	0.0002
B185															
B187	<0.06	<0.06	<0.06			<0.155	<0.143	<0.151			<0.163	<0.168	<0.162		
B193	<0.151	<0.136	<0.160												
B196															
B200															
B202	0.0367	0.0371	0.0371	0.0370	0.0002	<0.005	<0.005	<0.005			0.00734	0.00598	0.0059	0.0064	0.0008
B204	0.031	0.032	0.031	0.0313	0.0006	<0.002	<0.002	<0.002			<0.002	<0.002	<0.002		
B208															
B210															
B215															
B219	0.0343	0.0361	0.0351	0.0352	0.0009	0.00573	0.00618	0.0058	0.0059	0.0002	0.00481	0.0046	0.00449	0.0046	0.0002
B221	0.04	0.043	0.042	0.0417	0.0015	0.006	0.006	0.007	0.0063	0.0006	0.005	0.005	0.005	0.0050	
B224															
B226	0.03328	0.03352	0.03452	0.0338	0.0007	< 0.0097	< 0.0098				< 0.0098	< 0.0098	< 0.0099		
B230															

**Table B-32. Data summary table for CBNA in three marijuana samples.**

Data in **blue** font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

Target Consensus	Plant Sample 2					Plant Sample 3					Plant Sample 5					
	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD
Lab	A	B	C			A	B	C			A	B	C		Avg	SD
B079																
B099	0.035				0.0350		0.066		0.0660		0.079		0.0790			
B117	<0.002	<0.002	<0.002			0.051	0.054	0.056	0.0537	0.0025	0.113	0.123	0.116	0.1173	0.0051	
B125	<0.01	<0.01	<0.01			0.016	0.018	0.015	0.0163	0.0015	0.044	0.046	0.042	0.0440	0.0020	
B146	< 0.0159	< 0.0161	< 0.0159			0.0336	0.0323	0.0332	0.0330	0.0007	0.0632	0.0645	0.0671	0.0649	0.0020	
B148																
<b>B154</b>	0.013	0.014	0.012	0.0130	0.0010	0.036	0.134	0.216	0.1287	0.0901	<b>0.214</b>	<b>0.85</b>	<b>0.068</b>	<b>0.3773</b>	<b>0.4158</b>	
B158	<0.05	<0.05	<0.05			0.05	0.05	0.05	0.0500	0.0000	0.09	0.08	0.09	0.0867	0.0058	
B178	0.0142	0.0137	0.0373	0.0217	0.0135	0.0396	0.0401	0.0391	0.0396	0.0005	0.0764	0.0783	0.0785	0.0777	0.0012	
B196																
B210																

**Table B-33. Data summary table for THCV in three hemp samples.**

Data in **red** font have been flagged as a data entry of zero or results that include text (e.g., “< LOQ” or “present”).

Data in **blue** font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

*Note: This table spans multiple pages; the NIST values and consensus values are included on all pages for convenience.*

Target Consensus	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6					
				0.0014 0.0036	0.0002 0.0051				0.0066	0.0095				0.0004	0.0015	
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B003																
B004	0.004	0.004	0.004	0.0040	0.0000	0.006	0.006	0.006	0.0060	0.0000	0.00	0.00	0.00	0.00000	0.00000	
B005	< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1			
B006	< 0.012	< 0.012	< 0.012			< 0.011	< 0.011	< 0.011			< 0.012	< 0.012	< 0.012			
B007																
B009																
B012																
B013	<0.028	<0.028	<0.028			<0.057	<0.059	<0.060			<0.028	<0.028	<0.028			
B015																
B016	0.66	0.71	0.7	0.6900	0.0265	0.79	0.76	0.73	0.7600	0.0300	0.35	0.36	0.37	0.36000	0.01000	
B018	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			
B020																
B022	<0.04	<0.04	<0.04			<0.04	<0.04	<0.04			<0.04	<0.04	<0.04			
B023	0.0048	0.0054	0.0052	0.0051	0.0003	<0.0037	<0.0037	<0.0037			<0.0037	<0.0037	<0.0037			
B024																
B026	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			
B027	<0.025	<0.025	<0.025			<0.025	<0.025	<0.025			<0.025	<0.025	<0.025			
B028																
B029	<0.030	<0.030	<0.030			<0.030	<0.030	<0.030			<0.030	<0.030	<0.030			
B030	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			
B031																
B033	< 0.5	< 0.5	< 0.5			< 0.5	< 0.5	< 0.5			< 0.5	< 0.5	< 0.5			
B035	<0.0025	<0.0025	<0.0025			<0.005	<0.005	<0.005			<0.005	<0.005	<0.005			
B036	<0.01	<0.01	<0.01			<0.03	<0.03	<0.03			<0.01	<0.01	<0.01			
B037																
B038																
B041	<0.064					<0.064					<0.064					
B043	<0.01					<0.01					<0.01					
B044	<0.025	<0.025	<0.025			<0.025	<0.025	<0.025			<0.025	<0.025	<0.025			
B047	< 0.01	< 0.01	< 0.01			< 0.01	< 0.01	< 0.01			< 0.01	< 0.01	< 0.01			
B048																
B051	0.05	0.04	0.05	0.0467	0.0058	0.00	0.00	0.03	0.0100	0.0173	0.02	0.00	0.02	0.01333	0.01155	
B052	< 0.10	< 0.10	< 0.10			< 0.10	< 0.10	< 0.10			< 0.10	< 0.10	< 0.10			
B053	0.006	0.007	0.007	0.0067	0.0006	0.001	0.001	0.002	0.0013	0.0006	0.001	0.001	0.001	0.00100	0.00000	
B054	<0.0001	<0.0001	<0.0001			<0.0001	<0.0001	<0.0001			<0.0001	<0.0001	<0.0001			
B055	<0.0851	<0.0851	<0.0851			0.0859	0.0864	0.0833	0.0852	0.0017	0.0376	0.036	0.0374	0.03700	0.00087	
B057																

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.0014	0.0002				0.0066	0.0095				0.0004	0.0015
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B058	< 0.0295	< 0.0296	< 0.0296			< 0.0292	< 0.0295	< 0.0298			< 0.0289	< 0.0297	< 0.0296		
B060															
B061	<0.004	<0.004	<0.004			<0.004	0.003	0.005			<0.004	<0.004	<0.004		
B062	< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08		
B063															
B064	<0.0001					<0.0001					<0.0001				
B066	<0.004	<0.004	<0.004			<0.004	<0.004	<0.004			<0.004	<0.004	<0.004		
B068															
B069	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B070	<0.222	<0.222	<0.222			<0.111	<0.111	<0.111			<0.067	<0.067	<0.067		
B073	<0.0062					<0.0062					<0.0062				
B076	0.00309	0.00257	0.00257	0.0027	0.0003	0.00948	0.00996	0.01012	0.0099	0.0003	0.00149	0.00166	0.00181	0.00165	0.00016
B077															
B078															
B079															
B081	<0.24	<0.24	<0.24			<0.24	<0.24	<0.24			<0.24	<0.24	<0.24		
B082	0.007	0.008	0.006	0.0070	0.0010	<0.02	<0.02	<0.02			<0.02	<0.02	<0.02		
<b>B084</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.0000</b>	<b>0.0000</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.0000</b>	<b>0.0000</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00000</b>	<b>0.00000</b>
B085	0.002	0.002	0.002	0.0020	0.0000	<0.002	>0.002	>0.002			<0.002	<0.002	<0.002		
B088	<0.010	<0.010	<0.010			<0.010	<0.010	<0.010			<0.010	<0.010	<0.010		
<b>B090</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00000</b>	<b>0.00000</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00000</b>	<b>0.00000</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00000</b>	<b>0.00000</b>
B091	<0.023	<0.023	<0.023			<0.119	<0.119	<0.119			<0.065	<0.065	<0.065		
B094	< 0.02	< 0.02	< 0.02			< 0.02	< 0.02	< 0.02			< 0.02	< 0.02	< 0.02		
B095	< 0.002	< 0.002	< 0.002			< 0.002	< 0.002	< 0.002			< 0.002	< 0.002	< 0.002		
B097	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B098															
B099	<0.01					<0.01					<0.01				
B100	<0.007	<0.007				<0.007	<0.007				<0.007	<0.007			
B102	<0.0001	<0.0001	<0.0001			<0.0001	<0.0001	<0.0001			<0.0001	<0.0001	<0.0001		
B106	<0.001	<0.001	<0.001			0.011	0.01	0.01			0.0103	0.0006	<0.001	<0.001	<0.001
B108															
B109	<0.012	<0.012	<0.012			<0.012	<0.012	<0.011			<0.012	<0.012	<0.012		
B111															
<b>B113</b>	<b>0</b>	<b>0</b>	<b>0</b>	<b>0.0000</b>		<b>0.01</b>	<b>0.01</b>	<b>0.01</b>	<b>0.0100</b>		<b>0</b>	<b>0</b>	<b>0</b>	<b>0.00000</b>	
B114															
B115															
<b>B116</b>	0.0021	0.002	0.002	0.0020	0.0001	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.0000</b>	<b>0.0000</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00000</b>	<b>0.00000</b>
B117	<0.002	<0.002	<0.002			<0.002	<0.002	<0.002			<0.002	<0.002	<0.002		
B120															
B122															
B125	<0.01	<0.01	<0.01			0.011	0.01	<0.01			0.0105	0.0007	<0.01	<0.01	<0.01
B129															
B130	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.0014 0.0036	0.0002 0.0051				0.0066	0.0095				0.0004 0.0015	0.0010 0.02200
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B132	0.003	0.003	0.003	0.0030		0.001	0.001	0.001	0.0010		0.001	0.001	0.001	0.00100	
B137	0.012	0.011	0.01	0.0110	0.0010	0.026	0.026	0.034	0.0287	0.0046	0.022	0.022	0.022	0.02200	
B141	<0.0045	<0.0045	<0.0045			<0.0045	<0.0045	<0.0045			<0.0045	<0.0045	<0.0045		
B142															
B146	< 0.0156	< 0.0159	< 0.0158			< 0.0158	< 0.0156	< 0.0156			< 0.0157	< 0.0158	< 0.0159		
B147	<0.016	<0.016	<0.016			<0.016	<0.016	<0.016			<0.016	<0.016	<0.016		
B148															
B149	<0.0215	<0.0215	<0.0215			<0.0215	<0.0215	<0.0215			<0.0215	<0.0215	<0.0215		
B152															
B153	<0.0004	<0.0004	<0.0004			<0.0004	<0.0004	<0.0004			<0.0004	<0.0004	<0.0004		
B154	<0.045	<0.045	<0.045			<0.045	<0.045	<0.045			<0.045	<0.045	<0.045		
B158	<0.05	<0.05	<0.05			<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B159	<0.0099	<0.0100	<0.0098			<0.0102	<0.0101	<0.0099			<0.0097	<0.0095	<0.0102		
B161	0	0	0	0.0000		0	0	0	0.0000		0	0	0	0.00000	
B163	0.00155	0.00158	0.00161	0.0016	0.00000	0.00046	0.00051	0.00055	0.0005	0.00000	0.00043	0.00048	0.00034	0.00042	0.00008
B164	<0.001	<0.001	<0.001			<0.001	<0.001	<0.001			<0.001	<0.001	<0.001		
B165															
B167															
B172	< 0.031	< 0.031	< 0.031			0.04544	0.04776	0.04456	0.0459	0.0017	< 0.031	< 0.031	< 0.031		
B173	< 0.0101	< 0.0098	< 0.0097			< 0.01	< 0.0101	< 0.0102			< 0.0101	< 0.0098	< 0.0099		
B174	<0.06	<0.06	<0.06			<0.06	<0.06	<0.06			<0.06	<0.06	<0.06		
B178	<0.0007	<0.0007	<0.0007			<0.0007	<0.0007	<0.0007			<0.0007	<0.0007	<0.0007		
B181	<0.0077					<0.0077					<0.0077				
B182	0.05	0.05		0.0500		<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B184	<0.040	<0.040	<0.040			<0.040	<0.040	<0.040			<0.040	<0.040	<0.040		
B185															
B186	0.01	0.007	0.006	0.0077	0.0021	<0.0001	<0.0001	<0.0001			<0.0001	<0.0001	<0.0001		
B187	<0.06	<0.06	<0.06			<0.06	<0.06	<0.06			<0.06	<0.06	<0.06		
B188															
B189	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B190	0.01	0.01	0.01	0.0100		0.01	0.01	0.01	0.0100		0.01	0.01	0.01	0.01000	
B192	<0.0205	<0.0205	<0.0205			<0.0202	<0.0202	<0.0202			<0.0203	<0.0203	<0.0203		
B193	<0.200	<0.180	<0.212			<0.206	<0.189	<0.201			<0.216	<0.222	<0.214		
B195	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B198	<0.07	<0.07	<0.07			<0.07	<0.07	<0.07			<0.07	<0.07	<0.07		
B200															
B206	0.026	0.025	0.027	0.0260	0.0010	0.019	0.019	0.021	0.0197	0.0012	0.019	0.019	0.014	0.01733	0.00289
B208															
B210															
B212	<0.01	<0.01	<0.01			0.014	0.013	0.012	0.0130	0.0010	<0.01	<0.01	<0.01		
B213	<0.03	<0.03	<0.03			<0.03	<0.03	<0.03			<0.03	<0.03	<0.03		
B215	<0.0039					<0.0039									
B216	<0.053	<0.053	<0.053			<0.053	<0.053	<0.053			<0.053	<0.053	<0.053		

	NRC HEMP-1 (Plant Sample 1)				Plant Sample 4				Plant Sample 6						
Target Consensus				0.0014 0.0036	0.0002 0.0051				0.0066	0.0095				0.0004 0.0004	0.0015
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B217	< 0.01	< 0.01	< 0.01			< 0.01	< 0.01	< 0.01			< 0.01	< 0.01	< 0.01		
B219	0.00133	0.00139	0.00129	0.0013	0.0001	0.00036	0.00037	0.00037	0.0004		0.0002	0.00018	0.00018	0.00019	
B221	0.001	0.001	0.001	0.0010		0.0004	0.0005	0.0004	0.0004	0.0001	0.0002	0.0002	0.0002	0.00020	
B222	<0.04	<0.04	<0.04			<0.04	<0.04	<0.04			<0.05	<0.05	<0.05		
B223	<0.0125	<0.0125	<0.0125			<0.0125	<0.0125	<0.0125			<0.0125	<0.0125	<0.0125		
B224	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B226	< 0.0102	< 0.0099	< 0.01			< 0.0097	< 0.0099	< 0.0099			< 0.0099	< 0.0099	< 0.0099		
B230															
B234	<0.006	<0.006	<0.006			<0.006	<0.006	<0.006			<0.006	<0.006	<0.006		

**Table B-34. Data summary table for THCV in three marijuana samples.**

Target Consensus	Plant Sample 2					Plant Sample 3					Plant Sample 5				
	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B055	0.0169	0.0209	0.019	0.019	0.002	0.0173	0.0169	0.0204	0.0182	0.0019	0.0263	0.0219	0.0272	0.0251	0.0028
B062	< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08		
B099	<0.01					<0.01					<0.01				
B109	0.056	0.056	0.06	0.057	0.002	< 0.059	< 0.056	< 0.060			< 0.059	< 0.059	< 0.059		
B117	<0.002	<0.002	<0.002			<0.002	<0.002	<0.002			<0.002	<0.002	<0.002		
B125	0.022	0.021	0.021	0.021	0.001	0.023	0.024	0.024	0.0237	0.0006	0.023	0.022	0.024	0.0230	0.0010
B146	0.0315	0.0323		0.032	0.001	0.0279	0.0293	0.0329	0.0300	0.0026	0.0297	0.0308	0.0283	0.0296	0.0013
B154	<0.045	<0.045	<0.045			<0.045	<0.045	<0.045			<0.045	<0.045	<0.045		
B158	<0.05	<0.05	<0.05			<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B167	< 0.01	< 0.01	< 0.01			< 0.01	< 0.01	< 0.01			< 0.01	< 0.01	< 0.01		
B178	<0.0007	<0.0007	<0.0007			<0.0007	<0.0007	<0.0007			<0.0007	<0.0007	<0.0007		
B198	0.11	0.11	0.11	0.110		0.07	0.07	0.07	0.0700		<0.07	<0.07	<0.07		
B213	< 0.03	< 0.03	< 0.03			< 0.03	< 0.03	< 0.03			< 0.03	< 0.03	< 0.03		
B222	<0.04	<0.04	<0.04			<0.04	<0.04	<0.04			<0.05	<0.05	<0.05		

**Table B-35. Data summary table for THCVA in three hemp samples.**

Data in red font have been flagged as a data entry of zero or results that include text (e.g., “< LOQ” or “present”).

Data in blue font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

Note: This table spans multiple pages; the NIST values and consensus values are included on all pages for convenience.

Target Consensus	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	
Lab	A	B	C			A	B	C	Avg	SD	A	B	C	Avg	SD
B003															
B004															
B005	< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1		
B006	< 0.012	< 0.012	< 0.012			< 0.012	< 0.012	< 0.012			< 0.012	< 0.012	< 0.012		
B009															
B015															
B020															
B023	0.0061	0.006	0.0057	0.0059	0.0002	<0.0037	<0.0148	<0.0037			<0.0037	<0.0037	<0.0037		
<b>B024</b>						<b>0.03</b>	<b>0.03</b>		<b>0.0300</b>	<b>0.0000</b>					
<b>B026</b>	<0.01	<0.01	<0.01			<b>0.03</b>	<b>0.04</b>	<b>0.03</b>	<b>0.0333</b>	<b>0.0058</b>	<0.01	<0.01	<0.01		
B027	<0.025	<0.025	<0.025			<0.025	<0.025	<0.025			<0.025	<0.025	<0.025		
B028															
B030	<0.04	<0.04	<0.04			<0.04	<0.04	<0.04			<0.04	<0.04	<0.04		
B035	0.0065	0.00628	0.00618	0.0063	0.0002	<0.005	<0.005	<0.005			<0.005	<0.005	<0.005		
B037															
B041	<0.064					<0.064					<0.064				
B043	<0.01					<0.01					<0.01				
B048															
<b>B053</b>	<b>0.025</b>	<b>0.026</b>	<b>0.028</b>	<b>0.0263</b>	<b>0.0015</b>	0.004	0.005	0.005	0.0047	0.0006	0.003	0.003	0.004	0.00333	0.00058
B054	0.0074	0.0064	0.00623	0.0067	0.0007	<0.0005	<0.0005	<0.0005			<0.0005	<0.0005	<0.0005		
B057															
B058	< 0.0295	< 0.0296	< 0.0296			< 0.0292	< 0.0295	< 0.0298			< 0.0289	< 0.0297	< 0.0296		
B062	< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08		
B064	<0.0001					<0.0001					<0.0001				
B066	<0.004	<0.004	<0.004			<0.004	<0.004	<0.004			<0.004	<0.004	<0.004		
B069	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B070	<0.222	<0.222	<0.222			<0.111	<0.111	<0.111			<0.067	<0.067	<0.067		
<b>B076</b>	<b>0.01748</b>	<b>0.01686</b>	<b>0.01609</b>	<b>0.0168</b>	<b>0.0007</b>	0.00469	0.00499	0.00501	0.0049	0.0002	0.00293	0.003	0.00325	0.00306	0.00017
B078															
B079															
B081	<0.21	<0.21	<0.21			<0.21	<0.21	<0.21			<0.21	<0.21	<0.21		
B082	0.009	0.009	0.01	0.0093	0.0006	0.013	<0.03	<0.03	0.0130		<0.03	<0.03	<0.03		
<b>B084</b>	0.01	0.01	0.01	0.0100	0.0000	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.0000</b>	<b>0.0000</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00000</b>	<b>0.00000</b>
B085	0.01	0.009	0.009	0.0093	0.0006	0.002	0.002	0.002	0.0020	0.0000	<0.002	<0.002	<0.002		
B088	<0.013	<0.013	<0.013			<0.013	<0.013	<0.013			<0.013	<0.013	<0.013		
B095	<0.002	<0.002	<0.002			<0.002	<0.002	<0.002			<0.002	<0.002	<0.002		
B097	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4				Plant Sample 6					
Target Consensus				0.00728	0.00064				0.0052	0.0061				0.0024	0.0026
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B099	<0.01					<0.01					<0.01				
B100	<0.005	<0.017				<0.005	<0.005				<0.005	<0.005			
B102	< 0.0001	< 0.0001	< 0.0001			< 0.0001	< 0.0001	< 0.00001			< 0.0001	< 0.0001	< 0.00001		
B111															
B114															
B115															
<b>B117</b>	<b>0.048</b>	<b>0.076</b>	<b>0.085</b>	<b>0.0696</b>	<b>0.0193</b>	<b>0.024</b>	<b>0.101</b>	<b>0.077</b>	<b>0.0673</b>	<b>0.0394</b>	<b>0.058</b>	<b>0.046</b>	<b>0.056</b>	<b>0.05333</b>	<b>0.00643</b>
<b>B125</b>	<b>&lt;0.01</b>	<b>&lt;0.01</b>	<b>&lt;0.01</b>			<b>&lt;0.01</b>	<b>&lt;0.01</b>	<b>&lt;0.01</b>			<b>&lt;0.01</b>	<b>&lt;0.01</b>	<b>&lt;0.01</b>		
B129															
B132	0.013	0.011	0.013	0.0123	0.0012	0.003	0.003	0.002	0.0027	0.0006	0.002	0.002	0.002	0.00200	
B137	<0.0001	<0.0001	<0.0001			0.013	0.019	0.013	0.0150	0.0035	0.007	0.007	0.009	0.00767	0.00115
B141	0.007	0.006	0.006	0.0063	0.0006	<0.0045	<0.0045	<0.0045			<0.0045	<0.0045	<0.0045		
B142															
B146	< 0.0157	< 0.0160	< 0.0159			< 0.0159	< 0.0158	< 0.0157			< 0.0158	< 0.0160	< 0.0160		
B148															
B149	<0.0260	<0.0260	<0.0260			<0.026	<0.026	<0.026			<0.0260	<0.0260	<0.0260		
B153	<0.0005	<0.0005	<0.0005			<0.0005	<0.0005	<0.0005			<0.0005	<0.0005	<0.0005		
<b>B154</b>	<b>&lt;0.045</b>	<b>&lt;0.045</b>	<b>&lt;0.045</b>			0.014	0.016	0.017	0.0157	0.0015	<b>&lt;0.045</b>	<b>&lt;0.045</b>	<b>&lt;0.045</b>		
B158	<0.05	<0.05	<0.05			<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B159	<0.0099	<0.0100	<0.0098			<0.0102	<0.0101	<0.0099			<0.0097	<0.0095	<0.0102		
<b>B161</b>	<b>0.007</b>	<b>0.008</b>	<b>0.007</b>	<b>0.0073</b>	<b>0.0006</b>	<b>0</b>	<b>0</b>	<b>0</b>	<b>0.0000</b>		<b>0</b>	<b>0</b>	<b>0</b>	<b>0.00000</b>	
B163	0.00796	0.00796	0.00749	0.0078	0.0003	0.00144	0.00157	0.00119	0.0014	0.0002	0.0013	0.00131	0.00108	0.00123	0.00013
B165															
B174	<0.06	<0.06	<0.06			<0.06	<0.06	<0.06			<0.06	<0.06	<0.06		
<b>B178</b>	<b>0.00908</b>	<b>0.00858</b>	<b>0.00909</b>	<b>0.0089</b>	<b>0.0003</b>	<b>&lt;0.0006</b>	<b>&lt;0.0006</b>	<b>&lt;0.0006</b>			<b>&lt;0.0006</b>	<b>&lt;0.0006</b>	<b>&lt;0.0006</b>		
B185															
B187	<0.06	<0.06	<0.06			<0.06	<0.06	<0.06			<0.06	<0.06	<0.06		
B192	<0.0205	<0.0205	<0.0205			<0.0202	<0.0202	<0.0202			<0.0203	<0.0203	<0.0203		
B193	<0.202	<0.182	<0.212			<0.208	<0.192	<0.203			<0.218	<0.225	<0.216		
B195	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B200															
B202	0.0102	0.00772	0.00769	0.0085	0.0014	<0.005	<0.005	<0.005			0.00543	0.00607	0.00559	0.00570	0.00033
B204	0.007	0.007	0.007	0.007	0.000	<0.002	<0.002	<0.002			<0.002	<0.002	<0.002		
B208															
B210															
B215															
B216	<0.025	<0.025	<0.025			<0.025	<0.025	<0.025			<0.025	<0.025	<0.025		
B219	0.0068	0.00716	0.00672	0.0069	0.0002	0.00129	0.00136	0.00128	0.0013	0.0000	0.00099	0.00087	0.00089	0.00092	0.00006
B221	0.007	0.008	0.008	0.0077	0.0006	0.001	0.002	0.001	0.0013	0.0006	0.001	0.001	0.001	0.00100	
B222	<0.04	<0.04	<0.04			<0.04	<0.04	<0.04			<0.05	<0.05	<0.05		
B230															

**Table B-36. Data summary table for THCVA in three marijuana samples.**

Data in blue font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

Target Consensus	Plant Sample 2				Plant Sample 3				Plant Sample 5						
	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD			
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B062	< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08			< 0.08	< 0.08	< 0.08		
B099	<0.01					<0.01					<0.01				
B117	0.121	0.132	0.102	0.12	0.02	0.011	0.04	0.015	0.02200	0.01572	0.013	0.019	0.013	0.0150	0.0035
B125	<0.01	<0.01	<0.01			0.012	0.012	0.011	0.01167	0.00058	0.014	0.014	0.015	0.0143	0.0006
B146	< 0.0158	< 0.0159	< 0.0158			< 0.0159	< 0.0160	< 0.0160			< 0.0159	< 0.0159	< 0.0158		
B154	0.041	0.043	0.034	0.04	0.00	0.041	0.043	0.043	0.04233	0.00115	0.025	0.025	0.025	0.0250	
B158	<0.05	<0.05	<0.05			<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B178	<0.0006	<0.0006	<0.0006			0.0114	0.0115	0.0116	0.01150	0.00010	<0.0006	<0.0006	<0.0006	<0.0006	
B222	<0.04	<0.04	<0.04			<0.04	<0.04	<0.04			<0.05	<0.05	<0.05	<0.0006	<0.0006

**Table B-37. Data summary table for  $\Delta^8$ -THC in three hemp samples.**

Data in red font have been flagged as a data entry of zero or results that include text (e.g., "< LOQ" or "present").

Data in blue font have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable  $Z'_{\text{comm}}$  score,  $|Z'_{\text{comm}}| \geq 2$ .

Note: This table spans multiple pages; the NIST values and consensus values are included on all pages for convenience.

Target Consensus	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4				Plant Sample 6					
			0.013	0.013				0.0014	0.0027			0.0013	0.0030		
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B003															
B004	0.00	0.00	0.00	0.0000	0.0000						0.00	0.00	0.00	0.00000	0.00000
B005	< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1		
B006	0.021	0.02	0.02	0.0203	0.0006	0.03	0.043	0.04	0.0376	0.0068	0.017	0.017	0.031	0.02167	0.00808
B007															
B009															
B012															
B015															
B016	< 0.0004	< 0.0004	< 0.0004			< 0.0004	< 0.0004	< 0.0004			< 0.0004	< 0.0004	< 0.0004		
B018	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B020															
B022	<0.04	<0.04	<0.04			<0.04	<0.04	<0.04			<0.04	<0.04	<0.04		
B023	<0.0037	<0.0037	<0.0038			<0.0037	<0.0037	<0.0037			<0.0037	<0.0037	<0.0037		
B024			0.02	0.0200											
B026	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B027	<0.025	<0.025	<0.025			<0.025	<0.025	<0.025			<0.025	<0.025	<0.025		
B028															
B029	<0.030	<0.030	<0.030			<0.030	<0.030	<0.030			<0.030	<0.030	<0.030		
B030	<0.06	<0.06	<0.06			<0.06	<0.06	<0.06			<0.06	<0.06	<0.06		
B031															
B033	< 0.5	< 0.5	< 0.5			< 0.5	< 0.5	< 0.5			< 0.5	< 0.5	< 0.5		
B035	<0.0025	<0.0025	<0.0025			<0.005	<0.005	<0.005			<0.005	<0.005	<0.005		
B036	<0.02	<0.02	<0.02			<0.06	<0.06	<0.06			<0.02	<0.02	<0.02		
B037															
B038															
B043	<0.01					<0.01					<0.01				
B044	<0.025	<0.025	<0.025			<0.025	<0.025	<0.025			<0.025	<0.025	<0.025		
B045	not detected	not detected				not detected	not detected				not detected	not detected			
B047	< 0.01	< 0.01	< 0.01			< 0.01	< 0.01	< 0.01			< 0.01	< 0.01	< 0.01		
B048															
B049	0.01036	0.00701	0.00694	0.0081	0.0020										
B051	0.00	0.00	0.00	0.0000	0.0000	0.00	0.00	0.00	0.0000	0.0000	0.00	0.00	0.00		
B052	< 0.10	< 0.10	< 0.10			< 0.10	< 0.10	< 0.10			< 0.10	< 0.10	< 0.10		
B053	0.003	0.003	0.002	0.0027	0.0006	0.001	0.001	0.001	0.0010	0.0000	0.001	0.001	0.001	0.0010	0.0000
B054	<0.0001	<0.0001	<0.0001			<0.0001	<0.0001	<0.0001			<0.0001	<0.0001	<0.0001		
B055	<0.0933	<0.0933	<0.0933			<0.0933	<0.0933	<0.0933			<0.0741	<0.0741	<0.0741		
B056	0.011	0.009		0.0100	0.0014	0.002	0.002		0.0020	0.0000					

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.013	0.013				0.0014	0.0027				0.0013	0.0030
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B057															
B058	< 0.0295	< 0.0296	< 0.0296			< 0.0292	< 0.0295	< 0.0298			< 0.0289	< 0.0297	< 0.0296		
B060															
B061	0.028	0.03	0.038			0.0320	0.0053	<0.004	<0.004	<0.004					
B062	< 0.08	< 0.08	< 0.08					< 0.08	< 0.08	< 0.08					
B063															
B064	<0.0002							<0.0002							
B066	<0.004	<0.004	<0.004					<0.004	<0.004	<0.004					
B068															
B069	<0.01	<0.01	<0.01					<0.01	<0.01	<0.01					
B070	<0.222	<0.222	<0.222					<0.111	<0.111	<0.111					
B071	0.02	0.02	0.02			0.0200	0.0000	<0.005	<0.005	<0.005					
B073	<0.0062							<0.0062							
B077															
B078															
B079															
B081	<0.45	<0.45	<0.45					<0.45	<0.45	<0.45					
B082	<0.08	<0.08	<0.08					<0.08	<0.08	<0.08					
B084	0.00	0.00	0.00	0.0000	0.0000			0.00	0.00	0.00	0.0000	0.0000	0.00	0.00	0.00000
B085	< 0.002	< 0.002	< 0.002					< 0.002	< 0.002	< 0.002					
B088	<0.013	<0.013	<0.013					<0.013	<0.013	<0.013					
B090	0	0	0	0.0000				0	0	0	0.0000		0	0	0.00000
B094	0.04	0.04	0.04	0.0400				< 0.08	< 0.08	< 0.08					
B095	< 0.002	< 0.002	< 0.002					< 0.002	< 0.002	< 0.002					
B097	<0.01	<0.01	<0.01					<0.01	<0.01	<0.01					
B098															
B099	<0.01					0.061			0.0610		0.028				0.02800
B100	<0.005	<0.005				<0.005	<0.005				<0.005	<0.005			
B102	< 0.0001	< 0.0001	< 0.0001			< 0.0001	< 0.0001	< 0.0001			< 0.0001	< 0.0001	< 0.0001		
B106	0.035	0.035	0.033			0.0343	0.0012	0.001	0.002	0.002	0.0016	0.0005	0.001	0.001	0.00100
B109	< 0.012	< 0.012	< 0.012					< 0.012	< 0.012	< 0.011					
B110	<0.02	<0.02	<0.02					<0.02	<0.02	<0.02					
B111															
B113	0	0	0	0.0000				0	0	0	0.0000		0	0	0.00000
B114															
B115															
B116	0.0051	0.0049	0.0051	0.0050	0.0001			0.00	0.00	0.00	0.0000	0.0000	0.0104	0.0046	0.0038
B117	<0.002	<0.002	<0.002					0.006	0.013	<0.002	0.0095	0.0049	0.005	>0.002	>0.002
B120															
B122															
B125	<0.01	<0.01	<0.01					<0.01	<0.01	<0.01					
B126															
B129															

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4					Plant Sample 6				
Target Consensus				0.013	0.013				0.0014	0.0027				0.0013	0.0030
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B130	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B132	<0.0002	<0.0002	<0.0002			<0.0002	<0.0002	<0.0002			<0.0002	<0.0002	<0.0002		
<b>B137</b>	0.016	0.015	0.013	0.0147	0.0015	<b>0.044</b>	<b>0.043</b>	<b>0.045</b>	<b>0.0440</b>	<b>0.0010</b>	<b>0.024</b>	<b>0.028</b>	<b>0.031</b>	<b>0.02767</b>	<b>0.00351</b>
B141	0.009	0.008	0.008	0.0083	0.0006	<0.0045	0.007	<0.0045			<0.0045	<0.0045	<0.0045		
B142	<0.0007	<0.0007	<0.0007			<0.0075	<0.0075	<0.0075			<0.0007	<0.0007	<0.0007		
B144	0.002	0.001	0.002	0.0017	0.0006	<0.001	<0.001	<0.001			<0.001	<0.001	<0.001		
B146	<0.0158	<0.0161	<0.0161			<0.0161	<0.0159	<0.0159			<0.0159	<0.0161	<0.0161		
B147	<0.016	<0.016	<0.016			<0.016	<0.016	<0.016			<0.016	<0.016	<0.016		
B148															
B149	<0.027	<0.027	<0.027			<0.0279	<0.0279	<0.0279			<0.0279	<0.0279	<0.0279		
B152															
B153	<0.0004	<0.0004	<0.0004			<0.0004	<0.0004	<0.0004			<0.0004	<0.0004	<0.0004		
B154	<0.045	<0.045	<0.045			<0.045	<0.045	<0.045			<0.045	<0.045	<0.045		
B158	<0.05	<0.05	<0.05			<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B159	<0.0099	<0.0100	<0.0098			<0.0102	<0.0101	<0.0099			<0.0097	<0.0095	<0.0102		
B160	0.02496	0.02059	0.02306	0.0229	0.0022	0.00278	0.0025	0.00242	0.0025	0.0002	0.00207	0.00219	0.00285	0.00237	0.00042
<b>B161</b>	<b>0</b>	<b>0</b>	<b>0</b>	<b>0.0000</b>		<b>0</b>	<b>0</b>	<b>0</b>	<b>0.0000</b>		<b>0</b>	<b>0</b>	<b>0</b>	<b>0.00000</b>	
B163	<0.0027	<0.0027	<0.0027			<0.0027	<0.0027	<0.0027			<0.0027	<0.0027	<0.0027		
B164	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B165															
B168	0.00595	0.0045	0.00463	0.0050	0.0008	0.00139	0.00121	0.00121	0.0013	0.0007	0.00107	0.00115	0.00117	0.00113	0.00005
B169	<0.15	<0.15				<0.15	<0.15				<0.15	<0.15			
<b>B172</b>	0.03592	0.03536	<0.031	0.0356	0.0004	<0.031	<0.031	<0.031			<0.031	<0.031	<b>0.04176</b>	<b>0.04176</b>	
B173	<0.0151	<0.0147	<0.0146			<0.01	<0.0101	<0.0102			<0.0101	<0.0098	<0.0099		
B174	<0.06	<0.06	<0.06			<0.06	<0.06	<0.06			<0.06	<0.06	<0.06		
B175	<0.05	<0.05	<0.05			<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B178	<0.0010	<0.0010	<0.0010			<0.0010	<0.0010	<0.0010			<0.0010	<0.0010	<0.0010		
B181	<0.0077					<0.0077					<0.0077				
B182	<0.05	<0.05				<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B184	<0.035	<0.035	<0.035			<0.035	<0.035	<0.035			<0.035	<0.035	<0.035		
B185															
B186	<0.0001	<0.0001	<0.0001			<0.0001	<0.0001	<0.0001			<0.0001	<0.0001	<0.0001		
B187															
B188															
B189	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B190	0.01	0.01	0.01	0.0100											
B192	0.02779	0.02786	0.0281	0.0279	0.0002	<0.0202	<0.0202	<0.0202			<0.0203	<0.0203	<0.0203		
B193	<0.280	<0.252	<0.297			<0.288	<0.265	<0.281			<0.302	<0.311	<0.300		
B195	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B196															
B198	<0.07	<0.07	<0.07			<0.07	<0.07	<0.07			<0.07	<0.07	<0.07		
B200															
B202	<0.0100	<0.0100	<0.0100			<0.0100	<0.0100	<0.0100			<0.0100	<0.0100	<0.0100		

	NRC HEMP-1 (Plant Sample 1)					Plant Sample 4				Plant Sample 6					
Target Consensus				0.013	0.013				0.0014	0.0027				0.0013	0.0030
Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
B204	<0.002	<0.002	<0.002			<0.002	<0.002	<0.002			<0.002	<0.002	<0.002		
B206	< 0.01	< 0.01	< 0.01			< 0.02	< 0.02	< 0.02			< 0.01	< 0.01	< 0.01		
B208															
B210															
B212	0.041	0.04	0.039			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B213	< 0.03	< 0.03	< 0.03			< 0.03	< 0.03	< 0.03			< 0.03	< 0.03	< 0.03		
B215	<0.0039					<0.0039					<0.0038				
B216	<0.077	<0.077	<0.077			<0.077	<0.077	<0.077			<0.077	<0.077	<0.077		
B217	< 0.06	< 0.06	< 0.06			< 0.007	< 0.007	< 0.007			< 0.007	< 0.006	< 0.007		
B219	<0.0002	<0.0002	<0.0002			<0.0002	<0.0002	<0.0002			<0.0002	<0.0002	<0.0002		
<b>B221</b>	<b>0</b>	<b>0</b>	<b>0</b>	<b>0.0000</b>		<b>0</b>	<b>0</b>	<b>0</b>	<b>0.0000</b>		<b>0</b>	<b>0</b>	<b>0</b>	<b>0.00000</b>	
B222	<0.04	<0.04	<0.04			<0.04	<0.04	<0.04			<0.05	<0.05	<0.05		
B223	<0.0125	<0.0125	<0.0125			<0.0125	<0.0125	<0.0125			<0.0125	<0.0125	<0.0125		
B224	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		
B226	< 0.0152	< 0.0148	< 0.015			< 0.0097	< 0.0098				< 0.0098	< 0.0098	< 0.0099		
B227	<0.05	<0.05	<0.05			<0.05	<0.05	<0.05			<0.05	<0.05	<0.05		
B228	< 0.02	< 0.02	< 0.02			< 0.02	< 0.02	< 0.02			< 0.02	< 0.02	< 0.02		
B230															
B234	0.02	0.02	0.02	0.0200	0.0000	<0.01	<0.01	<0.01			<0.01	<0.01	<0.01		

**Table B-38. Data summary table for  $\Delta^8$ -THC in three marijuana samples.**

Data in red font have been flagged as a data entry of zero or results that include text (e.g., “< LOQ” or “present”).

Target Consensus	Plant Sample 2					Plant Sample 3					Plant Sample 5				
	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	Avg	SD	
Lab	A	B	C		A	B	C	A	B	C	A	B	C	Avg	SD
B045	not detected	not detected													
B055	< 0.0933	< 0.0933	< 0.0933		< 0.0933	< 0.0933	< 0.0933				< 0.0933	< 0.0933	< 0.0933		
B062	< 0.08	< 0.08	< 0.08		< 0.08	< 0.08	< 0.08				< 0.08	< 0.08	< 0.08		
B071	<0.005	<0.005	<0.005		<0.005	<0.005	<0.005				<0.005	<0.005	<0.005		
B099	0.112				0.11		0.116				0.098				
B109	< 0.056	< 0.056	< 0.060		< 0.059	< 0.056	< 0.060				< 0.059	< 0.059	< 0.059		0.10
B117	0.024	0.025	0.024	0.02	0.00	0.022	0.02	0.021	0.02	0.00	0.018	0.019	0.017	0.02	0.00
B125	<0.01	<0.01	<0.01		<0.01	<0.01	<0.01				<0.01	<0.01	<0.01		
B146	< 0.0159	< 0.0161	< 0.0159		< 0.0161	< 0.0161	< 0.0161				< 0.0160	< 0.0160	< 0.0160		
B154	<0.045	<0.045	<0.045		<0.045	<0.045	<0.045				<0.045	<0.045	<0.045		
B158	<0.05	<0.05	<0.05		<0.05	<0.05	<0.05				<0.05	<0.05	<0.05		
B169	<0.15	<0.15			<0.15	<0.15					<0.15	<0.15			
B178	<0.0010	<0.0010	<0.0010		<0.0010	<0.0010	<0.0010				<0.0010	<0.0010	<0.0010		
B198	<0.07	<0.07	<0.07		<0.07	<0.07	<0.07				<0.07	<0.07	<0.07		
B213	< 0.03	< 0.03	< 0.03		< 0.03	< 0.03	< 0.03				< 0.03	< 0.03	< 0.03		
B222	<0.04	<0.04	<0.04		<0.04	<0.04	<0.04				<0.05	<0.05	<0.05		

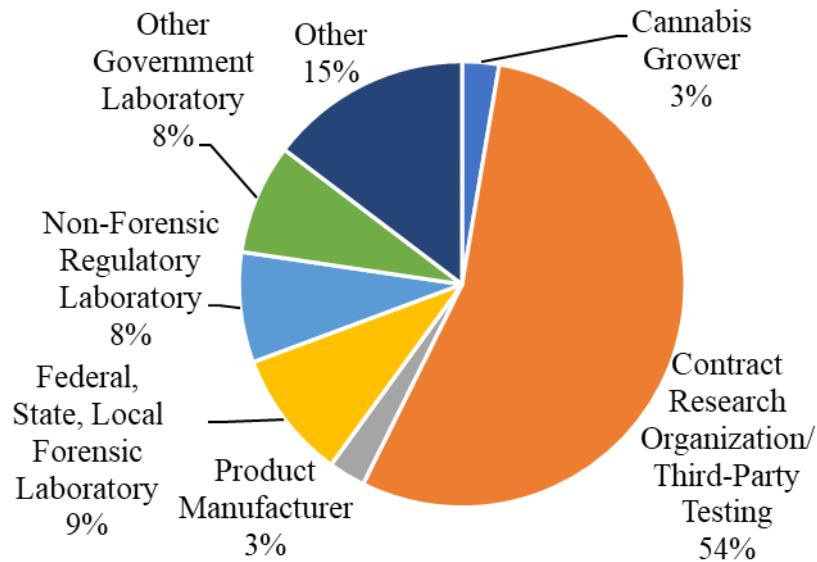
### Appendix C. Method questionnaire

Ninety-three laboratories provided information in the method questionnaire for the entire study. The laboratory codes of these participants are shown below and the following sections correspond to laboratories who only participated in the cannabinoid portion of the study.

B003	B041	B092	B146	B172	B205
B005	B053	B093	B147	B173	B206
B007	B054	B100	B151	B175	B207
B008	B056	B101	B153	B178	B212
B011	B060	B102	B154	B181	B213
B012	B061	B104	B155	B182	B216
B015	B064	B109	B157	B186	B217
B016	B066	B113	B158	B187	B218
B022	B071	B117	B159	B190	B219
B024	B072	B123	B163	B192	B222
B027	B073	B126	B164	B193	B223
B029	B077	B135	B166	B195	B224
B030	B082	B138	B167	B198	B228
B033	B083	B141	B168	B202	
B034	B085	B142	B169	B203	
B035	B088	B144	B171	B204	

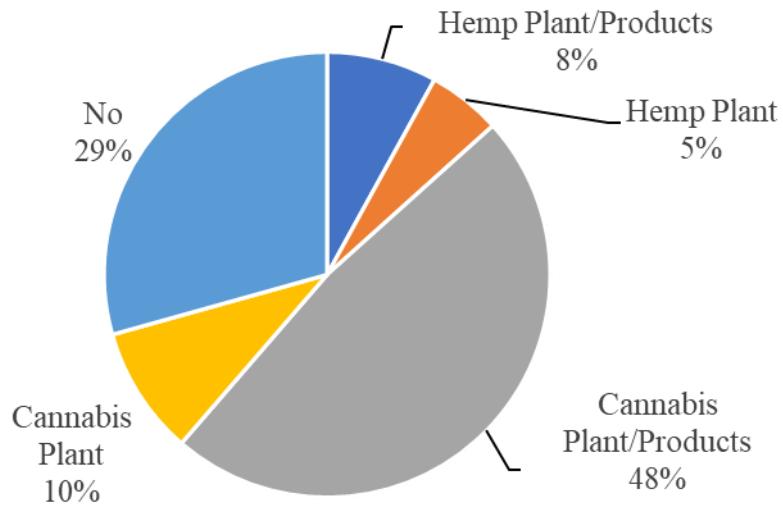
## C.1. General Laboratory Questions

### C.1.1. Participant sectors



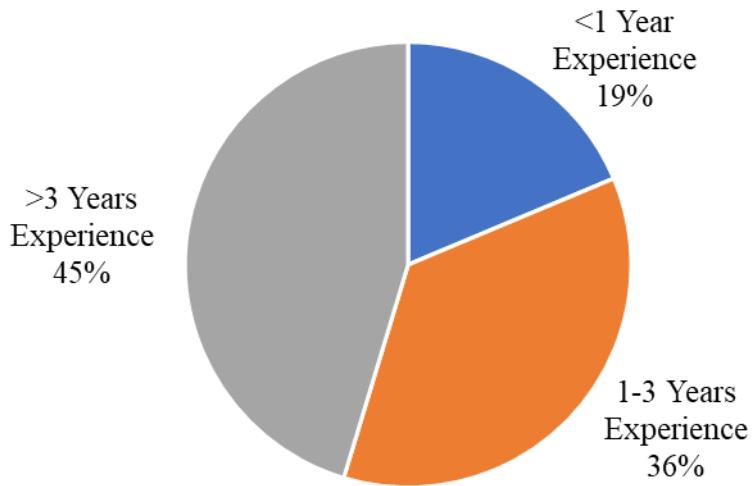
<b>Cannabis Grower</b>	B024	B126								
<b>Contract Research Organization</b>	B007	B012	B015	B016	B029	B030	B035	B041	B054	B060
<b>Third-Party Testing</b>	B061	B064	B066	B072	B088	B100	B102	B104	B113	B141
	B142	B144	B153	B157	B159	B166	B172	B173	B178	B182
	B186	B187	B190	B193	B206	B212	B213	B216	B217	B223
<b>Product Manufacturer</b>	B056	B138								
<b>Forensic Lab</b>	B093	B101	B135	B167	B171	B198	B218			
<b>Non-Forensic Regulatory Lab</b>	B003	B022	B027	B082	B147	B195				
<b>Other Government Lab</b>	B011	B085	B109	B117	B146	B205				
<b>Other</b>	B005	B071	B073	B092	B151	B154	B158	B163	B164	B168
	B169									

### C.1.2. Accreditation



<b>Hemp Plant/Products</b>	B030	B072	B088	B187	B206	B212				
<b>Hemp Plant Only</b>	B011	B135	B198	B224						
<b>Cannabis Plant/Products</b>	B003	B012	B015	B022	B027	B029	B035	B041	B056	B060
	B061	B064	B066	B082	B100	B102	B109	B113	B144	B147
	B151	B153	B157	B159	B167	B168	B169	B173	B178	B186
	B195	B213	B216	B217	B218	B223				
<b>Cannabis Plant Only</b>	B016	B093	B101	B104	B142	B171	B182			
<b>No</b>	B005	B007	B024	B054	B071	B073	B085	B092	B117	B126
	B138	B141	B146	B154	B158	B163	B164	B166	B172	B190
	B193	B205								

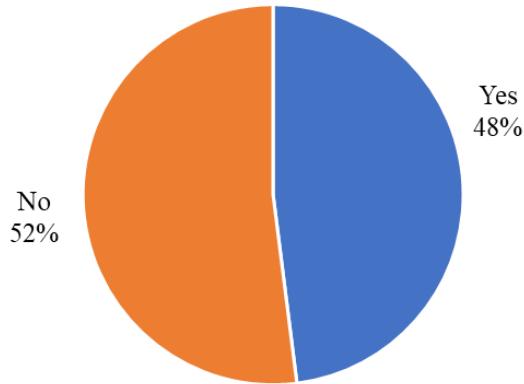
### C.1.3. Level of Experience



<b>&lt; 1 year</b>	B005	B071	B088	B101	B117	B135	B138	B154	B168	B171
	B182	B187	B206	B218						
<b>1-3 years</b>	B007	B011	B024	B035	B060	B073	B085	B092	B093	B102
	B146	B158	B163	B164	B166	B167	B169	B172	B173	B178
	B193	B198	B205	B213	B217	B223	B224			
<b>&gt; 3 years</b>	B003	B012	B015	B016	B022	B027	B029	B030	B041	B054
	B056	B061	B064	B066	B072	B082	B100	B104	B109	B113
	B126	B141	B142	B144	B147	B151	B153	B157	B159	B186
	B190	B195	B212	B216						

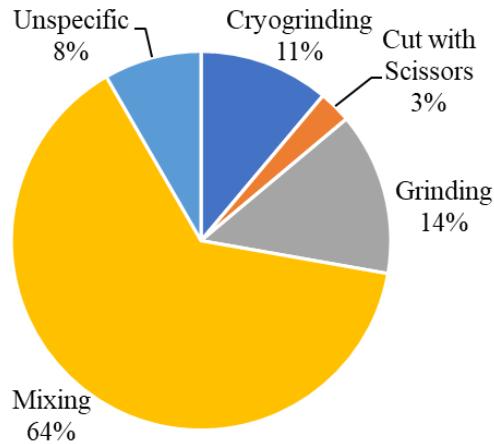
## C.2. Sample Preparation Questions

### C.2.1. Homogenization



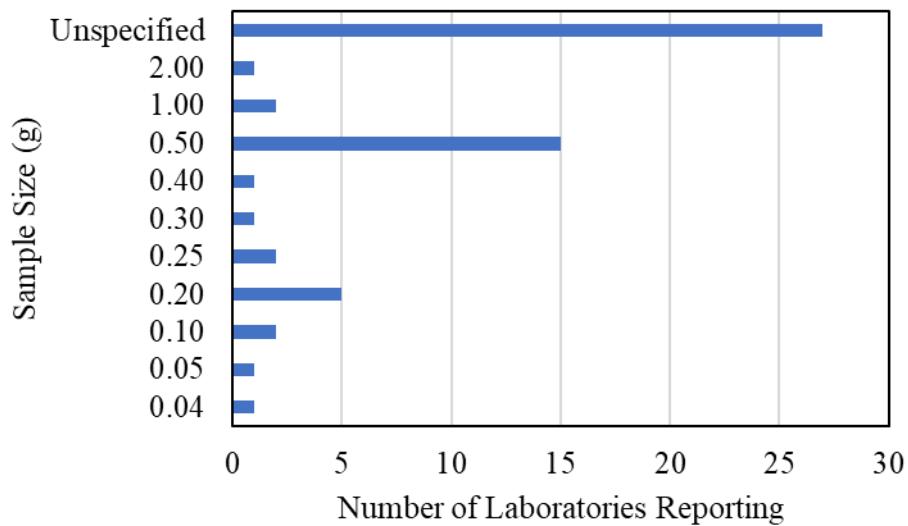
Yes	B011	B012	B015	B027	B029	B030	B035	B054	B056	B064
	B066	B071	B073	B088	B100	B104	B109	B126	B135	B144
	B146	B157	B159	B163	B169	B173	B186	B187	B190	B193
	B195	B198	B206	B212	B213	B218				
No	B003	B005	B007	B016	B022	B024	B041	B060	B061	B072
	B082	B085	B092	B093	B101	B102	B113	B117	B138	B141
	B142	B147	B151	B153	B154	B158	B164	B166	B167	B168
	B171	B172	B178	B182	B205	B216	B217	B223	B224	

### C.2.2. Homogenization method



<b>Cryogrinding</b>	B027	B056	B159	B187						
<b>Cut with Scissors</b>	B015									
<b>Grinding</b>	B066	B157	B169	B193	B206					
<b>Mixing</b>	B011	B012	B029	B030	B035	B054	B064	B071	B088	B100
	B109	B126	B135	B144	B146	B163	B173	B186	B190	B198
<b>Unspecified</b>	B212	B213	B218							
	B073	B104	B195							

### C.2.3. Sample Size

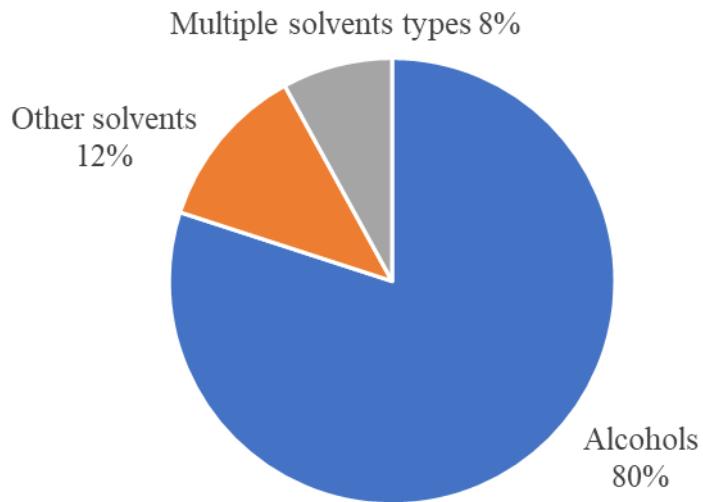


<b>0.04 g (1.7 %)</b>	B101
<b>0.05 g (1.7 %)</b>	B093
<b>0.1 g (3.4 %)</b>	B113 B117
<b>0.2 g (8.6 %)</b>	B005 B007 B102 B159 B212
<b>0.25 g (3.4 %)</b>	B073 B142
<b>0.3 g (1.7 %)</b>	B166
<b>0.4 g (1.7 %)</b>	B205
<b>0.5 g (25.9 %)</b>	B003 B012 B015 B016 B035 B060 B082 B100 B104 B146 B147 B151 B168 B172 B223
<b>1 g (3.4 %)</b>	B157 B187
<b>2 g (1.7 %)</b>	B041
<b>Unspecified (46.6 %)</b>	B011 B024 B027 B029 B054 B064 B085 B088 B092 B126 B135 B138 B144 B153 B158 B163 B164 B169 B171 B178 B182 B186 B193 B206 B213 B216 B218

### C.3. Extraction Procedure

All laboratories that tested the six cannabis samples (hemp and marijuana) reported using the same sample extraction methods for both sample sets.

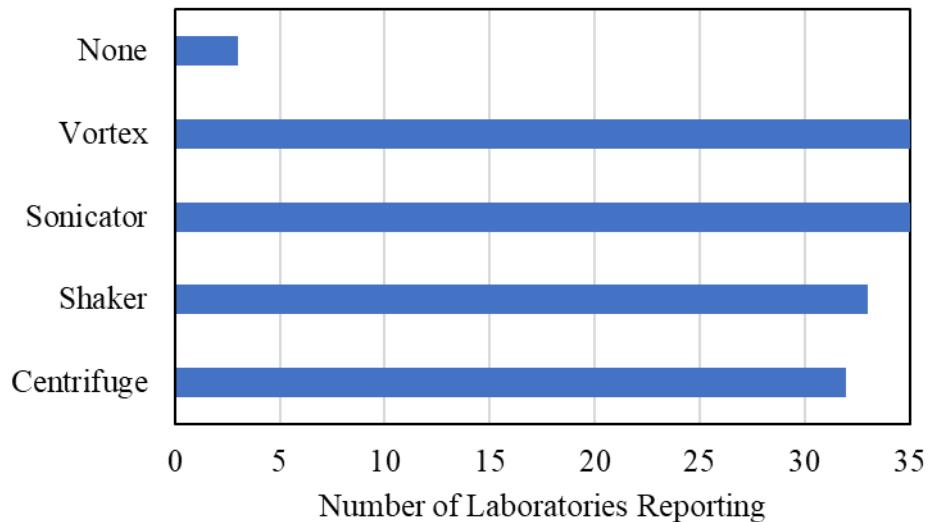
#### C.3.1. Extraction Solvent



Extraction with alcohol (ethanol, methanol, etc.)	Extraction with other solvent (hexane, DMSO, etc.)	Extraction with multiple solvent composition				
B003	B060	B109	B157	B182	B029	B024
B005	B061	B113	B158	B187	B054	B056
B007	B064	B117	B159	B193	B085	B072
B011	B066	B126	B163	B198	B088	B093
B012	B071	B138	B164	B205	B135	B166
B015	B073	B141	B167	B206	B153	B186
B016	B082	B142	B168	B212	B190	
B022	B092	B144	B169	B213	B195	
B027	B100	B146	B171	B216	B217	
B030	B101	B147	B172	B218		
B035	B102	B151	B173	B223		
B041	B104	B154	B178	B224		

### C.3.2. Extraction Equipment

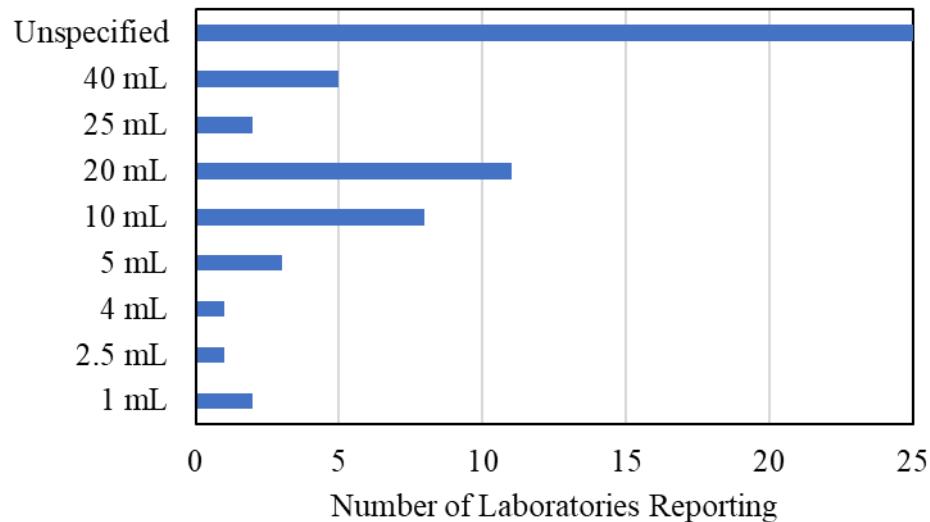
Note: Some laboratories reported using multiple pieces of equipment (up to 4)



<b>Centrifuge</b>	B005	B016	B022	B035	B041	B071	B072	B092	B101	B102
	B104	B109	B126	B147	B154	B157	B158	B159	B163	B166
	B167	B169	B171	B178	B182	B187	B195	B205	B212	B213
	B216	B223								
<b>Shaker</b>	B005	B011	B015	B016	B027	B030	B041	B061	B064	B071
	B072	B085	B092	B100	B109	B113	B141	B146	B157	B158
	B159	B163	B166	B171	B172	B173	B178	B182	B190	B195
	B212	B216	B223							
<b>Sonicator</b>	B003	B007	B012	B016	B022	B024	B035	B054	B060	B066
	B072	B082	B088	B102	B109	B117	B135	B142	B147	B151
	B153	B159	B163	B164	B166	B167	B168	B169	B186	B212
	B213	B216	B217	B223	B224					
<b>Vortex</b>	B003	B012	B016	B022	B024	B027	B035	B054	B061	B071
	B073	B082	B092	B093	B101	B102	B109	B126	B147	B151
	B153	B154	B163	B166	B167	B168	B169	B187	B195	B198
	B205	B206	B213	B217	B218					
<b>None</b>	B029	B056	B144							

### C.3.3. Extraction Volume

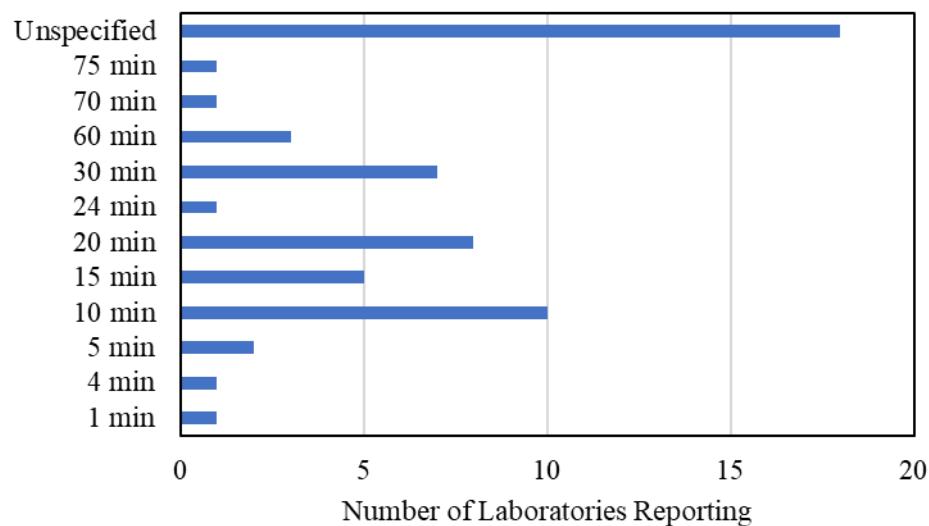
Note: A few laboratories reported performing repeated extractions.



<b>1 mL (3.4 %)</b>	B113	B163								
<b>2.5 mL (1.7 %)</b>	B166									
<b>4 mL (1.7 %)</b>	B101									
<b>5 mL (5.2 %)</b>	B088	B093	B205							
<b>10 mL (13.8 %)</b>	B015	B016	B024	B073	B157	B159	B168	B193		
<b>20 mL (19.0 %)</b>	B005	B007	B054	B060	B082	B102	B104	B117	B147	B172
<b>25 mL (3.4 %)</b>	B187									
<b>40 mL (8.6 %)</b>	B003	B012	B035	B142	B146	B151	B212			
<b>Unspecified (43.1 %)</b>	B011	B027	B029	B041	B064	B085	B092	B100	B126	B135
	B138	B144	B153	B158	B164	B169	B171	B178	B182	B186
	B206	B213	B216	B218	B223					

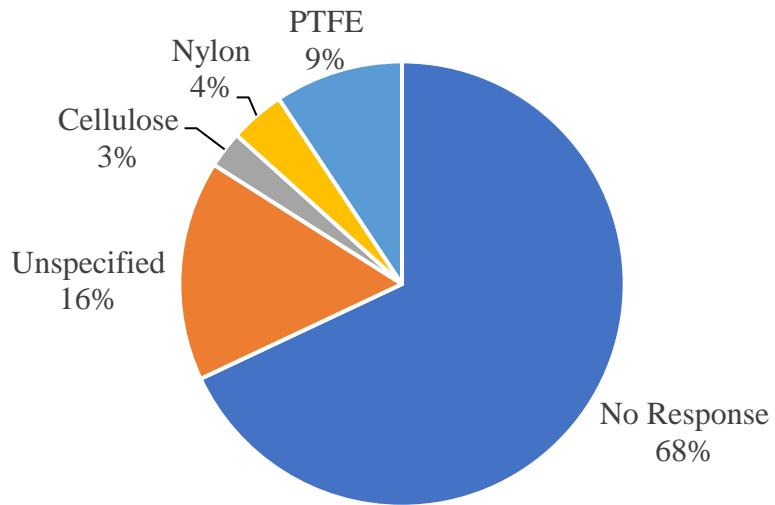
### C.3.4. Extraction Time

Note: Extraction times here include the vortexing, shaking, and/or sonication times reported.



<b>1 min (1.7 %)</b>	B126
<b>4 min (1.7 %)</b>	B157
<b>5 min (3.4 %)</b>	B011 B193
<b>10 min (17.2 %)</b>	B005 B093 B101 B147 B158 B168 B172 B187 B218 B223
<b>15 min (8.6 %)</b>	B007 B035 B113 B146 B164
<b>20 min (13.8 %)</b>	B024 B060 B064 B088 B102 B135 B142 B213
<b>24 min (1.7 %)</b>	B012
<b>30 min (12.1 %)</b>	B003 B082 B092 B117 B159 B182 B206
<b>60 min (5.2 %)</b>	B073 B151 B212
<b>70 min (1.7 %)</b>	B166
<b>75 min (1.7 %)</b>	B163
<b>Unspecified (31.0 %)</b>	B015 B016 B027 B029 B041 B054 B085 B100 B104 B138 B144 B153 B169 B171 B178 B186 B205 B216

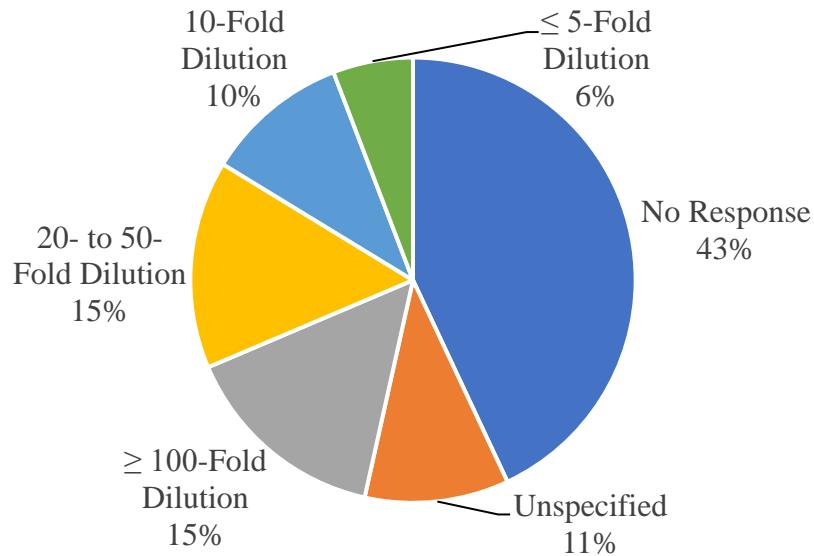
### C.3.5. Filtration



				Type of Materials			
No Response				Unspecified	Cellulose	Nylon	PTFE
B011	B085	B159	B213	B003	B005	B024	B054
B015	B093	B163	B216	B007	B172	B164	B088
B022	B100	B166	B217	B012		B187	B092
B029	B101	B167	B219	B016			B117
B030	B102	B169	B223	B027			B126
B035	B104	B171	B224	B060			B168
B041	B109	B173		B135			B182
B056	B113	B178		B144			
B061	B138	B186		B146			
B064	B141	B190		B147			
B066	B142	B193		B153			
B071	B151	B195		B205			
B072	B154	B198					
B073	B157	B206					
B082	B158	B212					

### C.3.6. Dilution

Note: Some laboratories reported using multiple dilutions

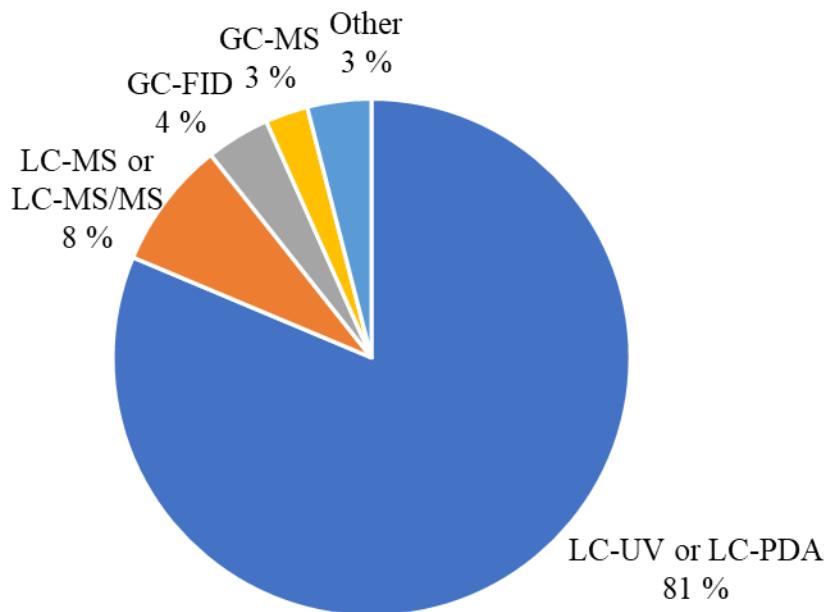


			Dilutions				
			Not Specified	≥100-Fold	20- to 50-Fold	10-Fold	≤ 5-Fold
No Response							
B007	B101	B195	B016	B003	B005	B060	B054
B011	B109	B198	B035	B012	B012	B073	B102
B015	B117	B205	B041	B073	B024	B142	B113
B022	B135	B212	B082	B100	B088	B144	B151
B027	B141	B217	B085	B144	B102	B146	B182
B029	B153	B218	B138	B159	B104	B159	
B030	B154	B224	B169	B163	B126	B163	
B056	B158		B213	B168	B147	B168	
B061	B166		B216	B187	B157	B193	
B064	B167			B193	B164		
B066	B171			B206	B172		
B071	B173			B223	B182		
B072	B178						
B092	B186						
B093	B190						

## C.4. Analytical Methods Questions

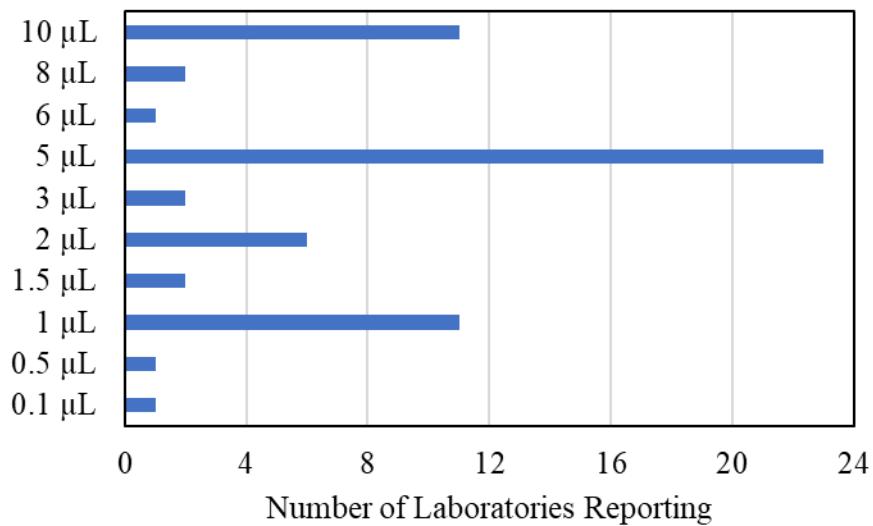
### C.4.1. Instrumental Techniques

All laboratories that tested the six cannabis samples (hemp and marijuana) reported using the same analytical methods for both sample sets. Because 89 % of the laboratories reported use of LC based method, the following subsections will only focus on LC based methods.



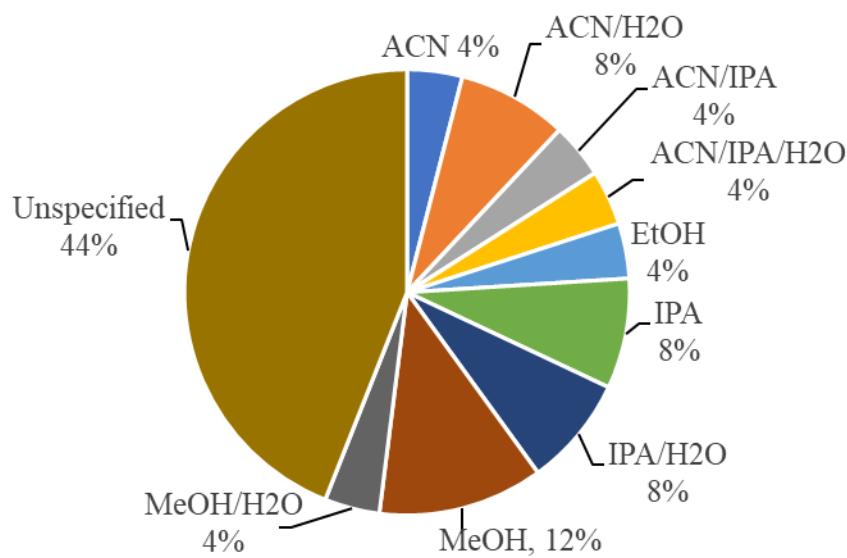
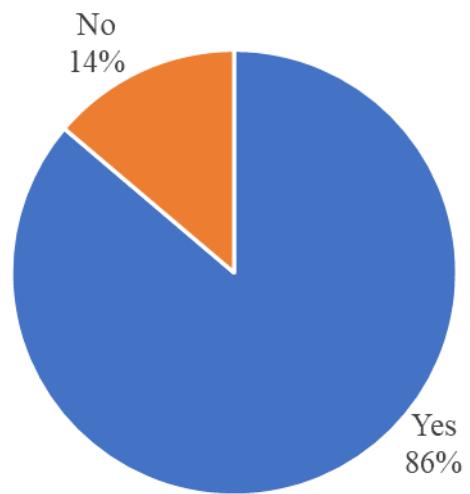
LC-UV LC-PDA		LC-MS LC-MS/MS		GC-MS	GC-FID	Other
B003	B092	B168	B041	B101	B011	B138
B005	B100	B169	B073	B218	B056	B151
B007	B102	B171	B085		B093	B166
B012	B104	B172	B163			
B015	B109	B173	B213			
B016	B113	B178	B224			
B022	B117	B182				
B024	B126	B186				
B027	B135	B187				
B029	B141	B190				
B030	B142	B193				
B035	B144	B195				
B054	B146	B198				
B060	B147	B205				
B061	B153	B206				
B064	B154	B212				
B066	B157	B216				
B071	B158	B217				
B072	B159	B223				
B082	B164					
B088	B167					

#### C.4.2. Injection Volumes



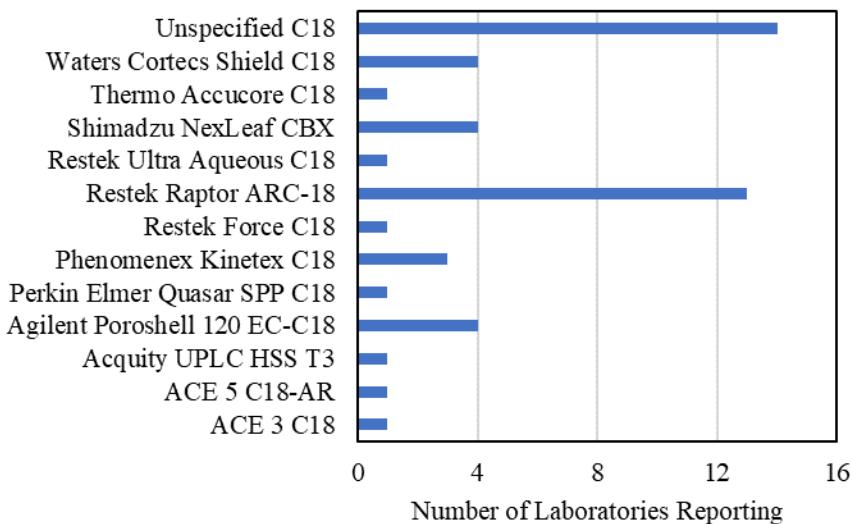
<b>0.1 <math>\mu\text{L}</math> (1.7 %)</b>	B003
<b>0.5 <math>\mu\text{L}</math> (1.7 %)</b>	B216
<b>1 <math>\mu\text{L}</math> (18.3 %)</b>	B027    B035    B054    B085    B117    B135    B146    B153    B158    B159 B164
<b>1.5 <math>\mu\text{L}</math> (3.3 %)</b>	B144    B244
<b>2 <math>\mu\text{L}</math> (10.0 %)</b>	B060    B092    B157    B163    B193    B195
<b>3 <math>\mu\text{L}</math> (3.3 %)</b>	B041    B064
<b>5 <math>\mu\text{L}</math> (38.3 %)</b>	B005    B007    B015    B024    B061    B066    B071    B072    B073    B088 B100    B142    B104    B147    B167    B171    B172    B173    B190    B205 B212    B213    B223
<b>6 <math>\mu\text{L}</math> (1.7 %)</b>	B022
<b>8 <math>\mu\text{L}</math> (3.3 %)</b>	B012    B030
<b>10 <math>\mu\text{L}</math> (18.3%)</b>	B016    B113    B126    B141    B154    B169    B182    B186    B187    B198 B206

### C.4.3. Needle Wash Solvent



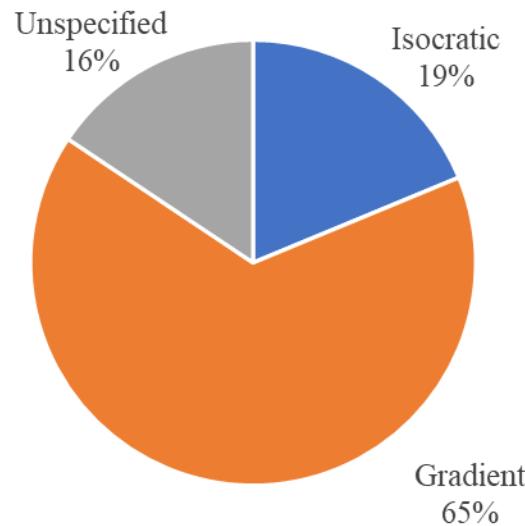
<b>No Wash</b>	B007	B142	B172	B193
<b>ACN</b>	B147			
<b>ACN/H<sub>2</sub>O</b>	B158	B168		
<b>ACN/IPA</b>	B187			
<b>ACN/IPA/H<sub>2</sub>O</b>	B024			
<b>EtOH</b>	B104			
<b>IPA</b>	B035	B157		
<b>IPA/H<sub>2</sub>O</b>	B178	B182		
<b>MeOH</b>	B102	B135	B171	
<b>MeOH/H<sub>2</sub>O</b>	B117			
<b>Unspecified</b>	B003	B005	B027	B060
				B071
				B072
				B113
				B126
				B144
				B163
				B164

#### C.4.4. Analytical Columns



<b>ACE 3 C18 (2.0 %)</b>	B169
<b>ACE 5 C18-AR (2.0 %)</b>	B198
<b>Acquity UPLC HSS T3 (2.0 %)</b>	B163
<b>Agilent Poroshell 120 EC-C18 (8.2 %)</b>	B005    B172    B030    B060
<b>Perkin Elmer Quasar SPP C18 (2.0 %)</b>	B187
<b>Phenomenex Kinetex C18 (6.1 %)</b>	B003    B193    B206
<b>Restek Force C18 (2.0 %)</b>	B164
<b>Restek Raptor ARC-18 (26.5 %)</b>	B022    B027    B035    B041    B088    B102    B104    B113 B146    B147    B158    B159    B186
<b>Restek Ultra Aqueous C18 (2.0 %)</b>	B144
<b>Shimadzu NexLeaf CBX (8.2 %)</b>	B024    B126    B223    B171
<b>Thermo Accucore C18 (2.0 %)</b>	B073
<b>Waters Cortecs Shield C18 (8.2 %)</b>	B092    B135    B205    B168
<b>Unspecified C18 (28.6 %)</b>	B007    B012    B015    B066    B071    B072    B117    B153 B154    B157    B173    B178    B182    B190

#### C.4.5. Mobile Phase Programs

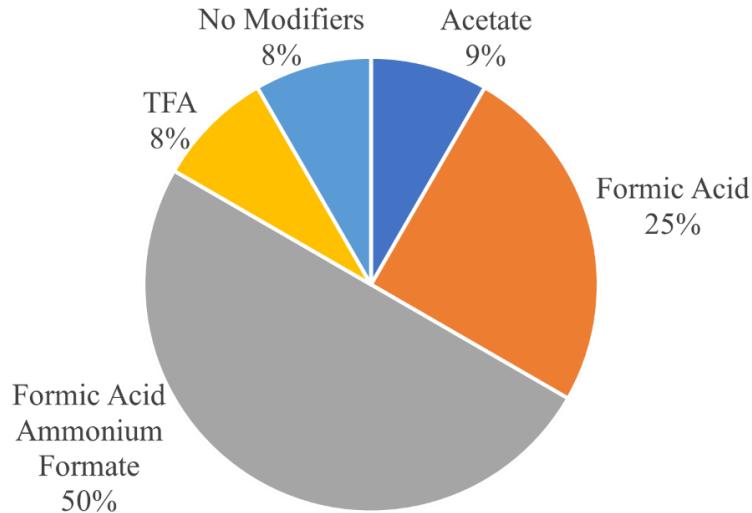


<b>Isocratic Mobile Phase</b>	B012	B088	B135	B141	B154	B164	B169	B178	B187	B193
	B198	B205								
<b>Gradient Mobile Phase</b>	B003	B005	B007	B016	B022	B024	B027	B054	B060	B061
	B066	B071	B072	B073	B085	B092	B100	B102	B104	B113
	B117	B126	B144	B146	B147	B153	B157	B158	B159	B163
	B168	B171	B172	B182	B190	B195	B206	B212	B213	B216
	B223	B224								
<b>Unspecified</b>	B015	B029	B030	B035	B041	B142	B167	B173	B186	B217

#### C.4.6. Isocratic Separation Conditions

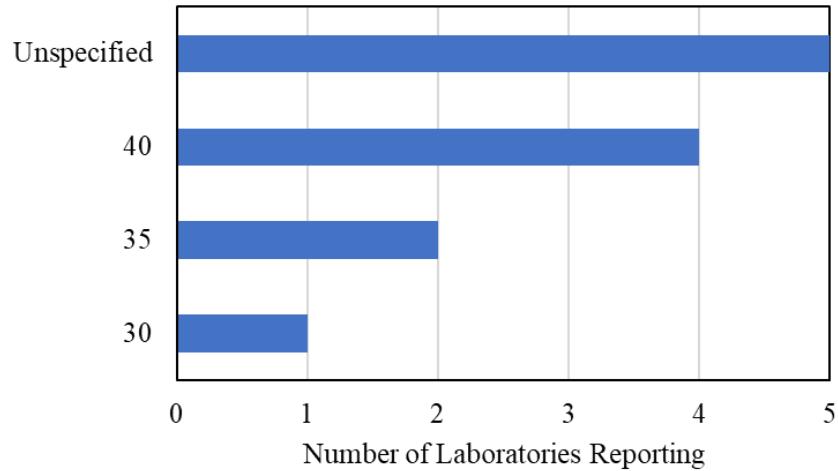
All laboratories using an isocratic mobile phase reported use of ACN:H<sub>2</sub>O.

##### C.4.6.1. Organic Modifiers



<b>Acetate</b>	B198
<b>Formic Acid</b>	B012    B135    B164
<b>Formic Acid/Ammonium Formate</b>	B088    B141    B178    B187    B193    B205
<b>TFA</b>	B169
<b>No Modifier</b>	B154

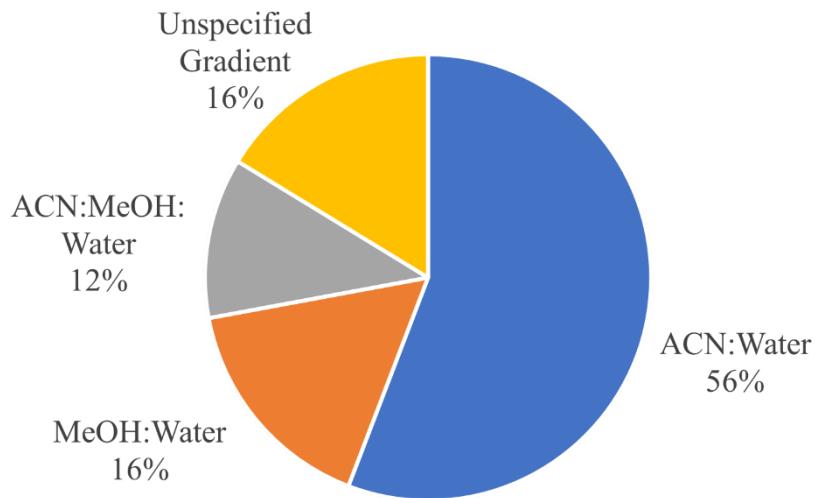
#### C.4.6.2. Column Temperatures (°C)



<b>30 °C (8.3 %)</b>	B088
<b>35 °C (16.7 %)</b>	B164      B169
<b>40 °C (33.3 %)</b>	B012      B187      B193      B205
<b>Unspecified (41.7 %)</b>	B135      B141      B154      B178      B198

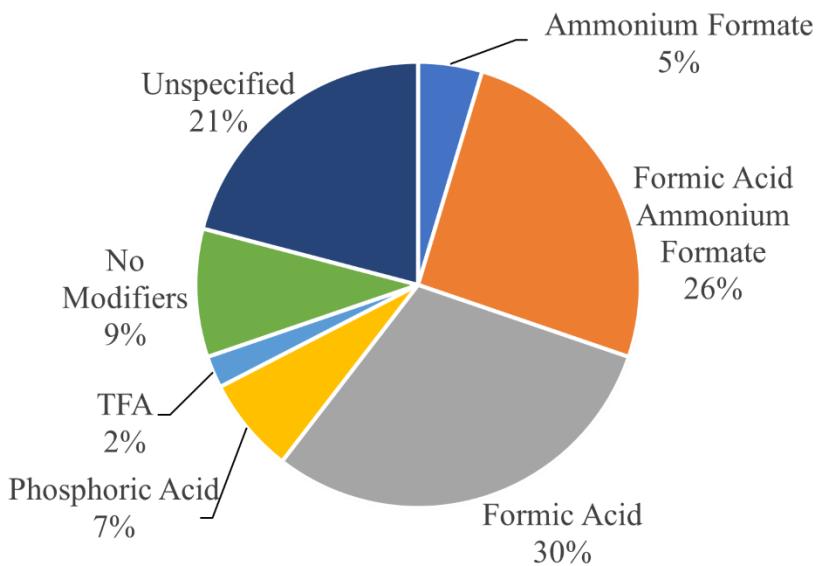
## C.4.7. Gradient Separation Conditions

### C.4.7.1. Mobile Phases



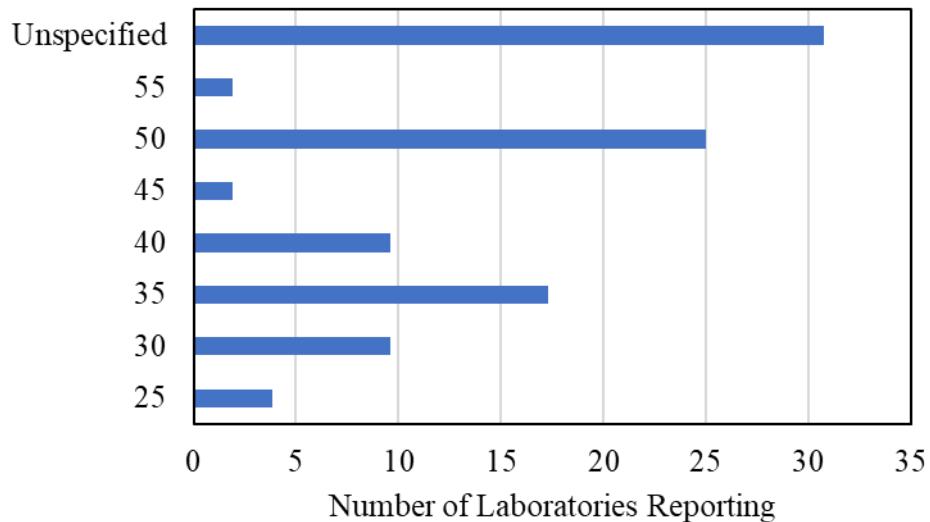
<b>ACN:Water</b>	B016	B022	B024	B027	B073	B092	B100	B102	B104	B113
	B117	B126	B146	B147	B157	B158	B159	B163	B168	B171
	B190	B212	B223	B224						
<b>MeOH:Water</b>	B003	B007	B071	B144	B153	B172	B213			
<b>ACN:MeOH:Water</b>	B005	B041	B054	B060	B206					
<b>Unspecified</b>	B061	B066	B072	B085	B182	B195	B216			

#### C.4.7.2. Organic Modifiers



<b>Ammonium Formate</b>	B153	B224									
<b>Formic Acid/</b> <b>Ammonium Formate</b>	B005	B027	B054	B073	B102	B117	B146	B147	B158	B159	
<b>Formic Acid</b>	B171	B003	B041	B060	B071	B092	B104	B157	B163	B172	B182
<b>Phosphoric Acid</b>	B206	B024	B126	B223	B213						
<b>TFA</b>	B190	B168									
<b>No Modifiers</b>	B007	B016	B100	B113							
<b>Unspecified</b>	B144	B022	B212	B061	B066	B072	B085	B195	B216		

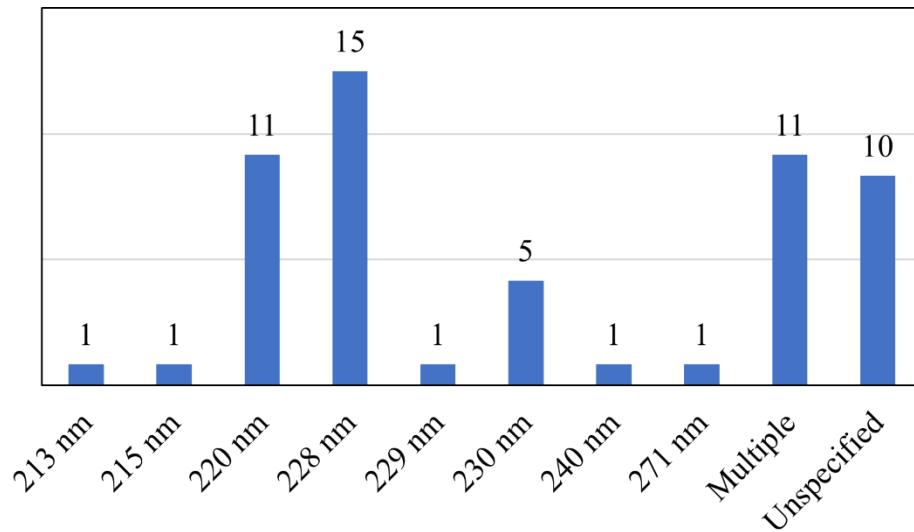
#### C.4.7.3. Column Temperature (°C)



<b>25 °C (3.8 %)</b>	B054	B073								
<b>30 °C (9.6 %)</b>	B005	B146	B158	B159	B206					
<b>35 °C (17.3 %)</b>	B024	B102	B113	B126	B153	B157	B212	B223	B224	
<b>40 °C (9.6 %)</b>	B003	B035	B041	B085	B100					
<b>45 °C (1.9 %)</b>	B163									
<b>50 °C (25.0 %)</b>	B007	B030	B060	B066	B071	B104	B144	B168	B171	B172
<b>55 °C (1.9 %)</b>	B117									
<b>Unspecified (30.8 %)</b>	B022	B015	B016	B027	B029	B061	B072	B092	B142	B147
	B167	B186	B190	B195	B216	B217				

#### C.4.8. Detector Wavelength

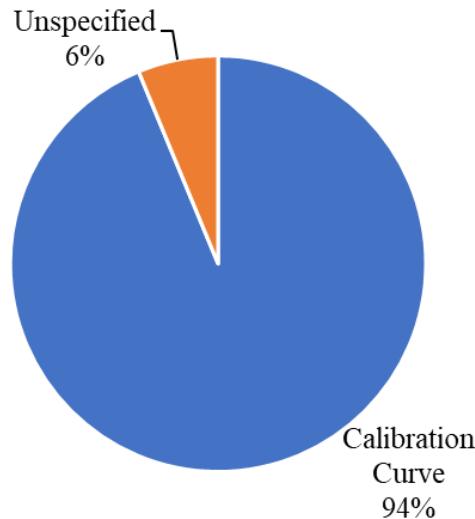
Note: Some laboratories reported using multiple distinct wavelengths (up to 5)



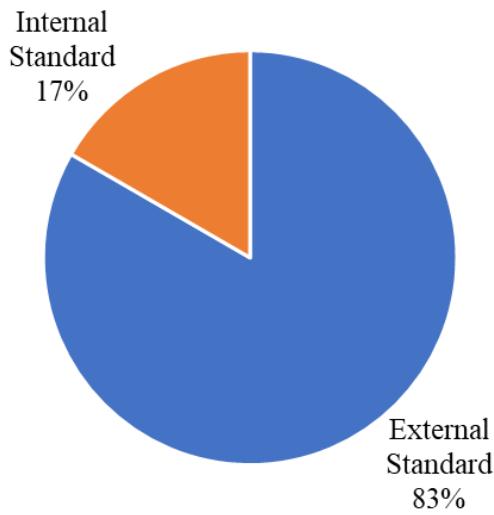
<b>213 nm (1.8 %)</b>	B092
<b>215 nm (1.8 %)</b>	B142
<b>220 nm (19.3 %)</b>	B024 B060 B104 B126 B147 B157 B169 B171 B178 B186 B190
<b>228 nm (26.3 %)</b>	B007 B035 B061 B071 B088 B102 B117 B146 B158 B159 B172 B187 B193 B205 B206
<b>229 nm (1.8 %)</b>	B153
<b>230 nm (8.8 %)</b>	B005 B066 B144 B173 B182
<b>240 nm (1.8 %)</b>	B198
<b>271 nm (1.8 %)</b>	B135
<b>Multiple Wavelengths (19.3 %)</b>	B003 B012 B022 B027 B030 B054 B100 B113 B141 B164 B216
<b>Unspecified (17.5 %)</b>	B015 B016 B029 B072 B154 B168 B195 B212 B217 B223

## C.5. Calibration Questions

### C.5.1. Calibration Methods

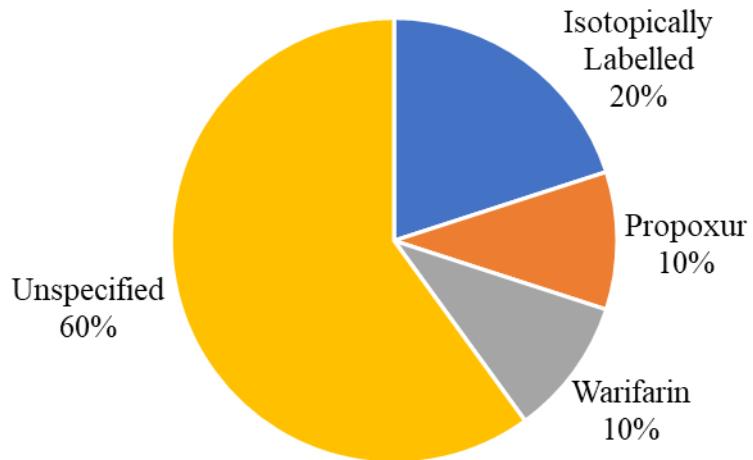


<b>Calibration Curve (93.8 %)</b>	B003 B005 B007 B012 B015 B016 B024 B027 B030 B035 B041 B054 B060 B061 B066 B071 B072 B073 B085 B088 B092 B100 B102 B104 B113 B117 B126 B135 B141 B142 B144 B146 B147 B153 B154 B157 B158 B159 B163 B164 B167 B168 B169 B171 B173 B178 B182 B186 B187 B190 B193 B195 B198 B205 B206 B212 B213 B216 B223 B224
<b>Unspecified (6.3 %)</b>	B022 B029 B172 B217



<b>External Standard (83.3 %)</b>	B003 B005 B007 B012 B015 B016 B024 B027 B030 B035 B054 B060 B061 B066 B071 B072 B085 B088 B092 B100 B102 B104 B113 B117 B126 B135 B141 B144 B146 B147 B153 B154 B157 B159 B164 B168 B169 B173 B186 B187 B190 B193 B195 B198 B205 B206 B212 B216 B223 B224
<b>Internal Standard (16.7 %)</b>	B041 B073 B142 B158 B163 B167 B171 B178 B182 B213

### C.5.2. Internal Standards

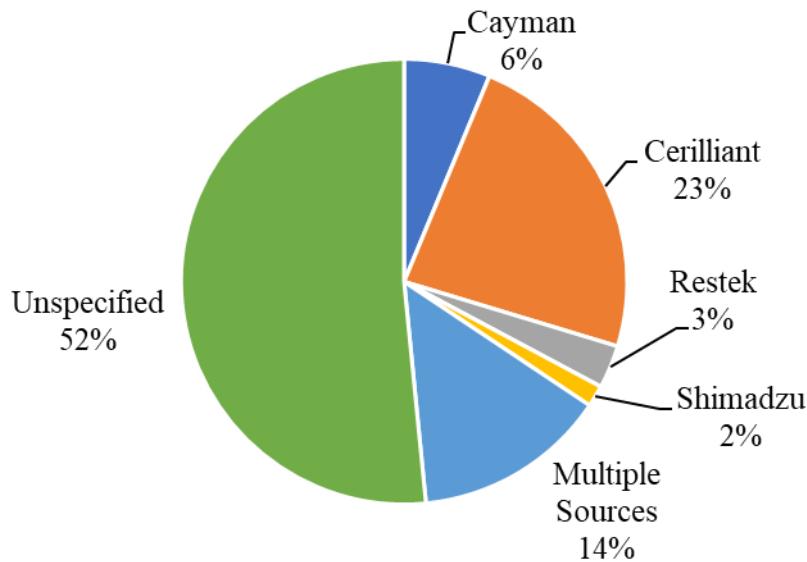


---

<b>Isotopically Labelled</b>	B073	B163
<b>Propoxur</b>	B142	
<b>Warfarin</b>	B041	
<b>Unspecified</b>	B158	B167 B171 B178 B182 B213

---

### C.5.3. Source of Calibrants



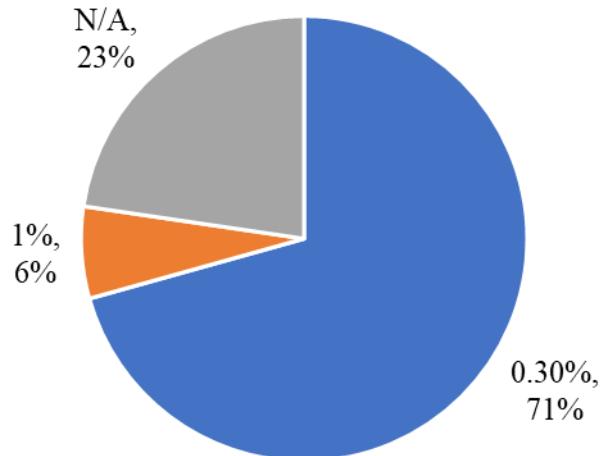
<b>Cayman</b>	B060	B126	B135	B182						
<b>Cerilliant</b>	B072	B092	B117	B142	B146	B154	B157	B158	B159	B168
	B169	B172	B187	B190	B193					
<b>Restek</b>	B071	B164								
<b>Shimadzu</b>	B024									
<b>Multiple Sources<sup>a</sup></b>	B003	B012	B022	B027	B088	B100	B113	B147	B171	
<b>Unspecified</b>	B005	B007	B015	B016	B029	B030	B035	B041	B054	B061
	B066	B073	B085	B102	B104	B141	B144	B153	B163	B167
	B173	B178	B186	B195	B198	B205	B206	B212	B213	B216
	B217	B223	B224							

<sup>a</sup> Consist of a combination from at least two of the following: Absolute Standards, Cayman, Cerilliant, Lipomed, and Restek.

## C.6. Sample Classification Questions

### C.6.1. Cutoff/Threshold Points for Distinguishing Hemp and Marijuana

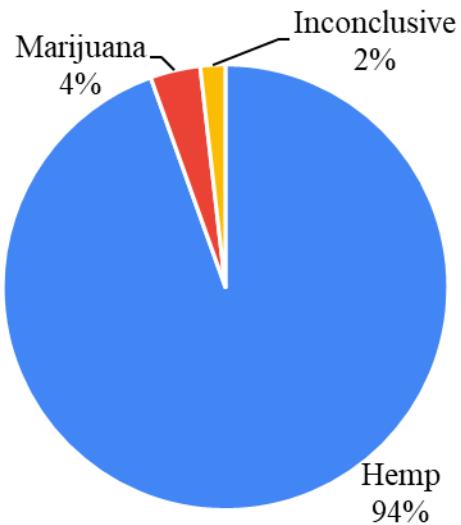
Note: Cutoff not specified as either  $\Delta^9$ -THC or Total  $\Delta^9$ -THC Mass Fractions.



<b>0.30 %</b>	B003	B005	B011	B015	B016	B022	B024	B030	B035	B054
	B056	B060	B064	B066	B071	B072	B073	B088	B100	B102
	B104	B109	B113	B117	B135	B138	B141	B144	B146	B147
	B157	B159	B163	B164	B168	B169	B171	B172	B173	B178
	B182	B186	B187	B190	B193	B198	B205	B206	B212	B216
	B217	B223	B224							
<b>1 %</b>	B093	B101	B158	B213	B218					
<b>NA</b>	B007	B012	B027	B029	B041	B061	B082	B085	B092	B126
	B142	B151	B153	B154	B166	B167	B195			

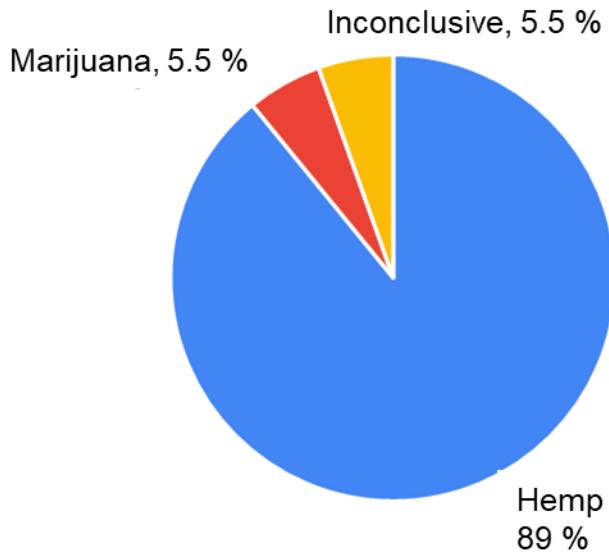
## C.6.2. Laboratories Classification of Hemp Samples

### C.6.2.1. NRC HEMP-1 (Plant Sample 1)



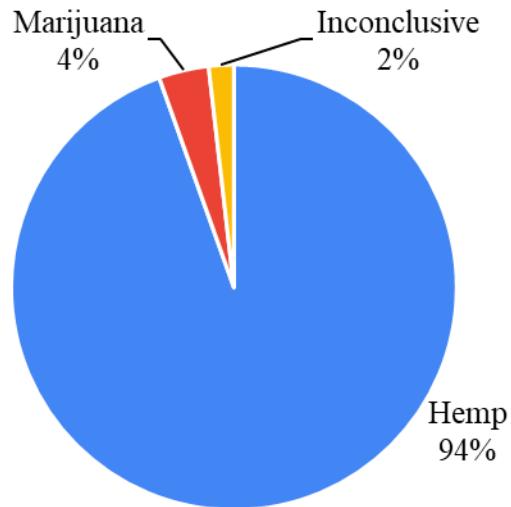
<b>Hemp</b>	B003	B005	B007	B015	B016	B022	B024	B030	B035	B054
	B060	B064	B066	B071	B088	B092	B100	B102	B104	B109
	B113	B117	B126	B141	B142	B144	B146	B147	B153	B154
	B157	B158	B159	B164	B168	B171	B172	B173	B178	B182
	B186	B187	B190	B193	B195	B198	B205	B206	B212	B216
	B217	B223								
<b>Marijuana</b>	B012	B082								
<b>Inconclusive</b>	B072									

### C.6.2.2. Plant Sample 4



<b>Hemp</b>	B003	B005	B007	B015	B016	B024	B030	B035	B054	B060
	B064	B066	B071	B088	B092	B100	B102	B104	B109	B113
	B117	B126	B141	B144	B146	B147	B153	B154	B157	B158
	B159	B168	B171	B172	B173	B178	B182	B186	B187	B190
	B193	B195	B198	B205	B206	B212	B216	B217	B223	
<b>Marijuana</b>	B012	B082	B164							
<b>Inconclusive</b>	B022	B072	B142							

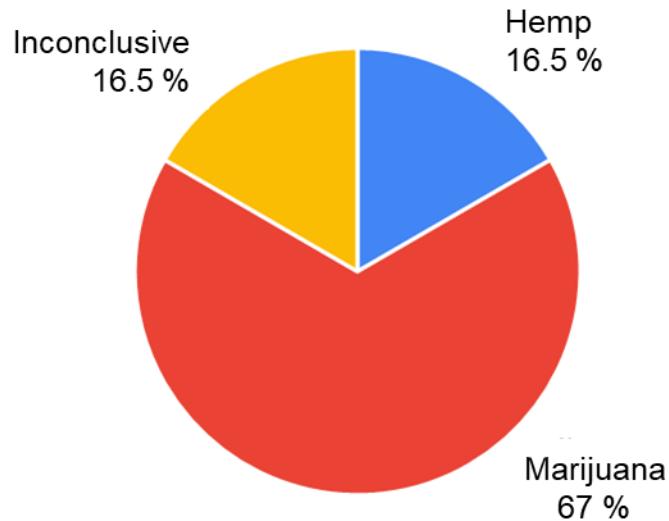
### C.6.2.3. Plant Sample 6



<b>Hemp</b>	B003 B005 B007 B015 B016 B022 B024 B030 B035 B054 B060 B064 B066 B071 B088 B092 B100 B102 B104 B109 B113 B117 B126 B141 B142 B144 B146 B147 B153 B154 B157 B158 B159 B164 B168 B171 B172 B173 B178 B182 B186 B187 B190 B193 B195 B198 B205 B206 B212 B216 B217 B223
<b>Marijuana</b>	B012 B082
<b>Inconclusive</b>	B072

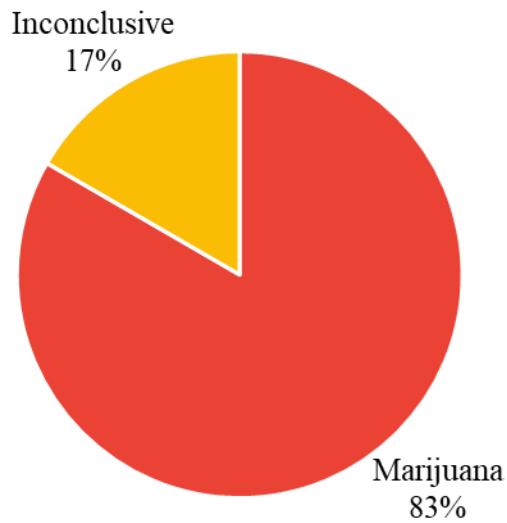
### C.6.3. Laboratories Classification of Marijuana Samples

#### C.6.3.4. Plant Sample 2



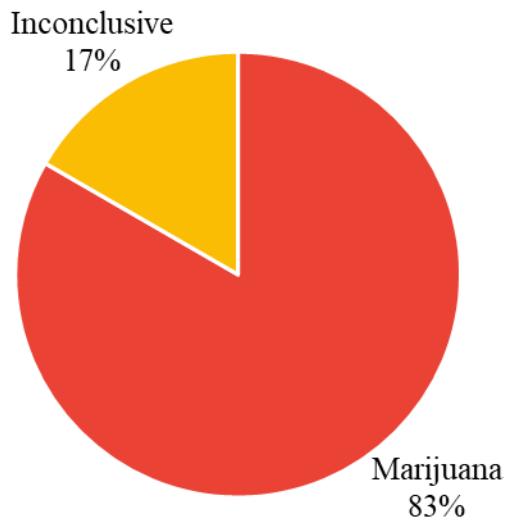
Hemp	B158	B171						
Marijuana	B071	B109	B117	B146	B154	B167	B178	B198
Inconclusive	B072	B088						

#### C.6.3.5. Plant Sample 3



Hemp										
Marijuana	B071	B109	B117	B146	B154	B158	B167	B171	B178	B198
Inconclusive	B072	B088								

### C.6.3.6. Plant Sample 5



Hemp	Marijuana	Inconclusive
<b>Marijuana</b>	B071 B109 B117 B146 B154 B158 B167 B171 B178 B198	
<b>Inconclusive</b>	B072 B088	