Purification, Purity, and Freezing Points of 30 Hydrocarbons of the API-Standard and API-NBS Series

By Anton J. Streiff, Janice C. Zimmerman, Laurel F. Soule, Marie T. Butt, Vincent A. Sedlak, Charles B. Willingham, and Frederick D. Rossini

This report describes the purification and determination of freezing points and purity of 30 hydrocarbons of the API-Standard and API-NBS series, including eight paraffins, six cycloparaffins, three aromatics, twelve olefins, and one acetylene.

I. Introduction

Previous reports described the purification and determination of freezing points and purity of 84 hydrocarbon compounds of the API-Standard and API-NBS series, which were produced as part of the cooperative program on Standard Samples of hydrocarbons of the National Bureau of Standards and the American Petroleum Institute [1, 2, 3, 4].

This report describes the purification and determination of freezing points and purity of an additional 30 hydrocarbon compounds under this cooperative program, including 8 paraffin hydrocarbons, 5 alkylcyclopentanes, 11 monoolefins, 3 diethylbenzenes, ethylcyclobutane, 1-butyne and cyclohexene. Four of these additional 30 compounds are second and purer lots of compounds, the first lots of which were described in the earlier reports.

The final lots of the material labeled API-Standard are sealed "in vacuum" in glass ampoules and made available as NBS Standard Samples of hydrocarbons, by the American Petroleum Institute and the National Bureau of Standards. The material labeled API-NBS is made available in appropriate small lots, through the American Petroleum Institute Research Project 44 at the National Bureau of Standards.

II. Materials

The starting materials were supplied as follows:

By the API Research Project 45 on the synthesis and properties of hydrocarbons of low molecular weight at the Ohio State University, Columbus, Ohio, under the supervision of C. E. Boord:

- Ethylcyclobutane.
- cis-1,3-Dimethylcyclopentane.
- trans-1,3-Dimethylcyclopentane (B).
- n-Butylcyclopentane.
- 1-Hexene (one-half).
- trans-2-Hexene.
- trans-3-Hexene.
- 3-Methyl-1-pentene.
- 2-Methyl-2-pentene.
- 2-Ethyl-1-butene (one-half).
- 1-Heptene.
- 1,3-Diethylbenzene (one-third).
- 1-Butyne (Ethylacetylene).

By the Hydrocarbon Laboratory at the Pennsylvania State College, State College, Pa., under the supervision of F. C. Whitmore:

- 2,3,3-Trimethylhexane. cis,cis,cis-1,2,3-Trimethylhexane.
- 2,2,4-Trimethylhexane. cis,trans,cis-1,2,3-Trimethylhexane.
- 2,3,3-Trimethylhexane. trans,trans,cis-1,2,3-Trimethylhexane.
- 2,3,5-Trimethylhexane.
- 3,3,4-Trimethylhexane. Trimethylcyclopentane.

1 This investigation was performed at the National Bureau of Standards as part of the work of the American Petroleum Institute Research Project 6 on the Analysis, purification, and properties of hydrocarbons.

2 Figures in brackets indicate the literature references at the end of this paper.

3 In this paper (B) following the name of a compound indicates that for the API-NBS series, it is a second (and usually slightly purer) sample of the given compound, the first sample of which will be labeled (A).

Purification, Purity, and Freezing Points
### Table 1. Information on the purification of 80 API–Standard and API–NBS hydrocarbons

<table>
<thead>
<tr>
<th>Compound ①</th>
<th>Laboratory ② providing starting material</th>
<th>Hydrocarbon charged for distillation</th>
<th>Volume</th>
<th>Purity</th>
<th>Kind</th>
<th>Azeotrope-forming substance ③</th>
<th>Amount of hydrocarbon in the azeotropic distillate</th>
<th>Distilling column number</th>
<th>Number of theoretical plates (approx.)</th>
<th>Reflux ratio ④ (approx.)</th>
<th>Rate of collection of distillate</th>
<th>Results plotted in figure</th>
<th>Volume selected of sample</th>
<th>API–Standard</th>
<th>API–NBS</th>
</tr>
</thead>
<tbody>
<tr>
<td>2,2-Dimethylpropane (Neopenone)</td>
<td>Univ. Oil Prod.</td>
<td>Ethylcyclobutane</td>
<td>2.00</td>
<td>0.98</td>
<td>Reg.</td>
<td>96.86±0.05</td>
<td>1</td>
<td>150</td>
<td>150/1</td>
<td>2.0</td>
<td>1</td>
<td>475</td>
<td>*500</td>
<td>250</td>
<td></td>
</tr>
<tr>
<td>2,2-Dimethylpropane (Neopenone)</td>
<td>Calif. Res. Corp.</td>
<td>Ethylcyclobutane</td>
<td>0.30</td>
<td>1.81</td>
<td>Reg.</td>
<td>98.83±0.05</td>
<td>h</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2,2,3-Trimeethylbutane (B)</td>
<td>General Motors</td>
<td>Ethylcyclobutane</td>
<td>0.50</td>
<td>2.44</td>
<td>Reg.</td>
<td>96.42±0.05</td>
<td>12</td>
<td>135</td>
<td>165/1</td>
<td>4.5</td>
<td>5</td>
<td></td>
<td>990</td>
<td>266</td>
<td></td>
</tr>
<tr>
<td>2,2,4-Trimeethylpentane (B)</td>
<td>APIRP45</td>
<td>Ethylcyclobutane</td>
<td>0.14</td>
<td>1.67</td>
<td>Reg.</td>
<td>98.96±0.05</td>
<td>12</td>
<td>135</td>
<td>165/1</td>
<td>4.5</td>
<td>10</td>
<td></td>
<td>795</td>
<td>156</td>
<td></td>
</tr>
<tr>
<td>2,2,3-Trimeethylpentane</td>
<td>Penn State</td>
<td>Ethylcyclobutane</td>
<td>1.04</td>
<td>1.54</td>
<td>Reg.</td>
<td>99.54±0.05</td>
<td>30</td>
<td>150</td>
<td>150/1</td>
<td>4.5</td>
<td>15</td>
<td>1040</td>
<td>213</td>
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<td>2,2,4-Trimeethylpentane</td>
<td>Penn State</td>
<td>Ethylcyclobutane</td>
<td>0.60</td>
<td>1.90</td>
<td>Reg.</td>
<td>98.96±0.05</td>
<td>14</td>
<td>125</td>
<td>125/1</td>
<td>4.5</td>
<td>28</td>
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<td>1158</td>
<td>345</td>
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<tr>
<td>2,2-Dimethylpropane</td>
<td>Calif. Res. Corp.</td>
<td>Ethylcyclobutane</td>
<td>0.30</td>
<td>1.81</td>
<td>Reg.</td>
<td>98.83±0.05</td>
<td>h</td>
<td></td>
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<tr>
<td>2,2-Dimethylpropane</td>
<td>Calif. Res. Corp.</td>
<td>Ethylcyclobutane</td>
<td>0.30</td>
<td>1.81</td>
<td>Reg.</td>
<td>98.83±0.05</td>
<td>h</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

① See footnote a of table 2.

③ The abbreviations are: Azeo., azethoporic; Reg., regular.

④ The abbreviations are: Cell., Celsolvent (ethylene glycol monoethyl ether); Me. Cell., methyl Celsolvent (ethylene glycol monomethyl ether); Me. Carb., methyl Carbitol (diethylene glycol monomethyl ether).

⑤ Approximate value obtained from the actual volume of hydrocarbon recovered by extracting the azethope—forming substance with water in separate funnels.


⑦ This is a second and improved API–Standard sample of this hydrocarbon.

⑧ This material was given its final purification by B. J. Mair by the method of adsorption. See references [6, 7].


⑩ One of two similar charges. Both cis and trans-1:3-dimethylokynopentane were obtained from this material (see fig. 17).

⑪ This charge consisted of material having substantially the same composition, from each of the two previous distillations (see footnote j), together with material from the first distillation of the concentrate of the trans isomer (see footnote z and fig. 21).

⑫ This charge consisted of material having substantially the same composition from each of the two previous distillations (see footnote j).

⑬ This is a second lot of cis, trans, cis-1,2,3-trimethylocyclopentane supplied by the Pennsylvania State College.

⑭ This material was divided into seven charges for purification by adsorption (see footnote h and fig. 21).

⑮ This is a second lot of 1,3-diethylbenzene supplied by the API Research Project 45.

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Table 1—Continued

<table>
<thead>
<tr>
<th>Compound a</th>
<th>Laboratory b providing starting material</th>
<th>Volume</th>
<th>Purity</th>
<th>Kind c</th>
<th>Presence of hydrocarbon in the isotropic distillate d</th>
<th>Reflux ratio e (approx.)</th>
<th>Rate of collection of distillate (ml/h)</th>
<th>Results plotted in figure</th>
<th>API-Standard</th>
<th>API-NBS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aromatics</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1,2-Diethylbenzene</td>
<td>NACA</td>
<td>3.60</td>
<td>97.6±0.06</td>
<td>Reg.</td>
<td>4</td>
<td>200</td>
<td>160/1</td>
<td>4.5</td>
<td>50</td>
<td>750</td>
</tr>
<tr>
<td>1,3-Diethylbenzene (B)</td>
<td>NACA</td>
<td>2.40</td>
<td>98.7±0.10</td>
<td>Azeo. Me. Corb.</td>
<td>73</td>
<td>8</td>
<td>135</td>
<td>145/1</td>
<td>8.5</td>
<td>32</td>
</tr>
<tr>
<td>1,4-Diethylbenzene</td>
<td>NACA</td>
<td>3.56</td>
<td>99.4±0.50</td>
<td>Reg.</td>
<td>9</td>
<td>135</td>
<td>165/1</td>
<td>4.5</td>
<td>34</td>
<td>110</td>
</tr>
</tbody>
</table>

Distillates

| Substance | Volume of selected sample | | | | | | | | | |
|-----------|---------------------------|--------|--------|-------|-------------------------|-------------------------|
| 1-Octene | Philips | 4.65 | Reg. | 3A | 200 | 160/1 | 4.5 | 37 | 1240 | 470 |
| 1-Hexene | API-NBS | 3.9 | 98.4±0.5 | Reg. | 9 | 135 | 165/1 | 4.5 | 38 | 1695 | 545 |
| 2,4,4-Trimethyl-1-pentene | API-NBS | 6.13 | 98.6±0.5 | Reg. | 13 | 135 | 155/1 | 8.0 | 40 | 1030 | 350 |
| 3-Methyl-1-pentene | API-NBS | 3.15 | 98.8±0.3 | Reg. | 4 | 200 | 160/1 | 4.5 | 48 | 1120 | 335 |
| 2-Methyl-1-pentene | General Motors | 3.06 | Reg. | 8 | 130 | 145/1 | 8.5 | 50 | 1045 | 250 |
| 2,6-Dimethyl-1-butene | API-NBS | 2.85 | 97.2±0.2 | Reg. | 12 | 135 | 165/1 | 4.5 | 51 | 1320 | 40 |
| 1-Nonene | API-NBS | 2.89 | 97.8±0.3 | Reg. | 12 | 135 | 165/1 | 4.5 | 36 | 1125 | 220 |
| 2,4,4-Trimethyl-1-pentene | NBS Auto Sec. | 6.00 | Reg. | 14 | 125 | 125/1 | 12.5 | 58 | 1350 | 350 |
| 2,4,4-Trimethyl-2-pentene | NBS Auto Sec. | 6.00 | Reg. | 14 | 125 | 125/1 | 12.5 | 58 | 1350 | 350 |
| Cyclohexene | API-NBS | 6.20 | 99.5±0.06 | Reg. | 10 | 135 | 165/1 | 4.5 | 63 | 2250 | 735 |

Acyclics

| Substance | Volume of selected sample | | | | | | | | | |
|-----------|---------------------------|--------|--------|-------|-------------------------|-------------------------|
| 1-Hexene | API-NBS | 2.95 | Reg. | 1 | 150 | 150/1 | 2.0 | 64 | 1125 | 265 |

Notes:
- a The total volume of the API-Standard sample was 1,475 ml.
- b One of two similar charges.
- c One of the two previous distillations (see footnote n).
- d One of two charges of similar material, one of which was 5.00 l. and the other 2.80 l.
- e One of three charges of similar material, two of which were 6.00 l. each, and the third of which was 5.30 l.
- f This charge consisted of material, having substantially the same composition, from each of the four previous distillations (see footnote m).
- g Obtained by purchase of commercially available material from the Eastman Kodak Co., Rochester, N. Y.
- h Obtained by purchase of commercially available material from the Connecticut Hard Rubber Co., New Haven, Conn.
- i One of two similar charges.
- j One of the two previous distillations (see footnote s).
- k One of two charges of similar material, one of which was 5.00 l. and the other 2.80 l.
- l One of three charges of similar material, two of which were 6.00 l. each, and the third of which was 5.30 l.
- m This charge consisted of material, having substantially the same composition, from each of the three previous distillations (see footnote w).
- n Obtained by purchase of commercially available material from the Connecticut Hard Rubber Co., New Haven, Conn.
- o The total volume of the API-Standard sample was 1,135 ml.
- p The total volume of the API-NBS sample was 290 ml.
- q One of four similar charges. Both 2,4,4-trimethyl-1-pentene and 2,4,4-trimethyl-2-pentene were obtained from this material (see fig. 58).
- r This charge consisted of material, having substantially the same composition, from each of the four previous distillations (see footnote cc).

Purification, Purity, and Freezing Points
By the National Advisory Committee for Aeronautics, through its Flight Propulsion Research Laboratory at Cleveland, Ohio:

1,2-Diethylbenzene.
1,3-Diethylbenzene (B) (two-thirds).
1,4-Diethylbenzene.

By the General Motors Corporation Research Laboratories, Detroit, Mich., through T. A. Boyd and W. G. Lovell:

2,2,3-Trimethylbutane (B).
3-Methyl-cis-2-pentene.

By the Hydrocarbons Research Laboratory, Automotive Section, National Bureau of Standards, through F. L. Howard and D. B. Brooks:

"Diisobutylene" (2,4,4-Trimethyl-1-pentene + 2,4,4-Trimethyl-2-pentene)

By the California Research Corporation, Richmond, Calif., through A. Kremser:

2,2-Dimethylpropane (Neopentane).

By the Phillips Petroleum Co., Bartlesville, Okla., through F. E. Frey:

1-Pentene (B).

By the Universal Oil Products Co., Riverside, Ill., through V. Haensel:

2,2-Dimethylpropane (Neopentane)

By the API Research Project 6 at the National Bureau of Standards, by purchase:

2,2,4-Trimethylpentane (B).
2-Ethyl-1-butene (one-half).
1-Hexene (one-half).

Cyclohexene.

Table 1 summarizes the amounts of the starting materials and gives some additional information as to source and purity.

Figure 1 to 64, inclusive, show graphically the results of the distillation and adsorption operations listed in table 1. These figures give, as appropriate, as a function of volume of hydrocarbon in the distillate or filtrate, the refractive index (nD at 25°C, to ±0.0001), the boiling point of the distillate (at the controlled pressure of 724.5 mm Hg, to ±0.01 deg C), the freezing point of selected fractions of hydrocarbon distillate (in air at 1 atm, usually, with a precision near ±0.003 deg C), and the purity of the hydrocarbon distillate. The letters W, X, Y, Z, indicate the disposition of the material as follows: W, returned to the laboratory supplying the material; X, blended for redistillation; Y, used for the API-Standard material; Z, used for the API-NBS material.

As demonstrated in the previous reports [2, 3, 4], the blending of fractions of distillate for the preparation of material of the highest purity can be done safely only on the basis of the freezing points of selected fractions. This is similarly true for the blending of fractions of filtrate from the adsorption process.

Table 2 gives the following information for each of the 30 compounds, except as otherwise indicated.

Footnotes to table 2.

1. (B) following the name of a compound indicates that for the API-NBS series, it is a second (and usually slightly purer) sample of the given compound, the first sample of which will be labeled (A).
3. The values in this column, except as otherwise indicated, were calculated as described in reference [8], using the values of the cryoscopic constants and freezing points for zero impurity given in the preceding columns.
4. Not determined in this investigation. From the 2 tables of the American Petroleum Institute Research Project 44, [9].
5. This is a second and improved API-Standard sample of this hydrocarbon.
6. Estimated by analogy with isomers subjected to similar purification.
7. This cryoscopic constant was determined by the procedure given on page 371 of reference [8].
**TABLE 2. Freezing points and purity of 30 API—Standard and API—NBS hydrocarbons**

<table>
<thead>
<tr>
<th>Compound</th>
<th>Kind of time-temperature observations used to determine freezing point</th>
<th>Freezing point of the actual selected sample in or at 1 atm.</th>
<th>Freezing point for zero impurity in or at 1 atm.</th>
<th>Cryoscopic constant</th>
<th>Calculated amount of impurity in the actual selected sample</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>API—Standard</td>
<td>API—NBS</td>
<td></td>
<td>A</td>
<td>API—Standard</td>
</tr>
<tr>
<td>1,2-Dimethylpropane (n-Decane)</td>
<td>F</td>
<td>-16.65</td>
<td>-16.55</td>
<td>±0.020</td>
<td>0.00395</td>
</tr>
<tr>
<td>1,2-Dimethylpentane (n-Decane)</td>
<td>F</td>
<td>-15.56</td>
<td>-15.55</td>
<td>±0.020</td>
<td>0.00395</td>
</tr>
<tr>
<td>2,2,3-Trimethylbutane (B)</td>
<td>F</td>
<td>-24.95</td>
<td>-24.93</td>
<td>±0.020</td>
<td>0.00430</td>
</tr>
<tr>
<td>1,2,4-Trimethylcyclohexane (B)</td>
<td>M</td>
<td>-107.39</td>
<td>-107.36</td>
<td>±0.013</td>
<td>0.0430</td>
</tr>
<tr>
<td>2,2,3-Trimethylhexane</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2,2,4-Trimethylhexane</td>
<td>M</td>
<td>-116.82</td>
<td>-116.80</td>
<td>±0.020</td>
<td>0.0447</td>
</tr>
<tr>
<td>2,3,5-Trimethylhexane</td>
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<td>-</td>
<td>-</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3,3,4-Trimethylhexane</td>
<td>M</td>
<td>-101.27</td>
<td>-101.20</td>
<td>±0.03</td>
<td>0.033</td>
</tr>
<tr>
<td>Cyclopentanes</td>
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<td></td>
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</tr>
<tr>
<td>3-Methylcyclopentane</td>
<td>M</td>
<td>-134.04</td>
<td>-133.90</td>
<td>±0.05</td>
<td>0.046</td>
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<td>trans-3,3-Dimethylcyclopentane (B)</td>
<td>M</td>
<td>-133.74</td>
<td>-133.70</td>
<td>±0.05</td>
<td>0.0459</td>
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<tr>
<td>cis, trans-3,3-Dimethylcyclopentane</td>
<td>M</td>
<td>-116.47</td>
<td>-116.45</td>
<td>±0.025</td>
<td>0.0245</td>
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<tr>
<td>M</td>
<td>-116.73</td>
<td>-116.72</td>
<td>±0.012</td>
<td>0.0365</td>
<td>0.2±0.04</td>
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<tr>
<td>n-Butylcyclopentane</td>
<td>M</td>
<td>-107.99</td>
<td>-107.98</td>
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<td>0.0424</td>
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<tr>
<td>Aromatics</td>
<td></td>
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<td></td>
</tr>
<tr>
<td>1,2-Diethylbenzene</td>
<td>F</td>
<td>-31.25</td>
<td>-31.24</td>
<td>±0.00</td>
<td>0.0299</td>
</tr>
<tr>
<td>1,3-Diethylbenzene (B)</td>
<td>M</td>
<td>-83.83</td>
<td>-83.83</td>
<td>±0.00</td>
<td>0.0369</td>
</tr>
<tr>
<td>1,4-Diethylbenzene</td>
<td>F</td>
<td>-42.87</td>
<td>-42.86</td>
<td>±0.00</td>
<td>0.0240</td>
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<tr>
<td>Olefins</td>
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<td></td>
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</tr>
<tr>
<td>1-Pentene (B)</td>
<td>M</td>
<td>-165.24</td>
<td>-165.25</td>
<td>±0.020</td>
<td>0.0600</td>
</tr>
<tr>
<td>1-Hexene</td>
<td>M</td>
<td>-130.26</td>
<td>-130.26</td>
<td>±0.005</td>
<td>0.0509</td>
</tr>
<tr>
<td>trans-2-Hexene</td>
<td>M</td>
<td>-132.00</td>
<td>-132.00</td>
<td>±0.020</td>
<td>0.0564</td>
</tr>
<tr>
<td>trans-3-Hexene</td>
<td>F and M</td>
<td>-113.44</td>
<td>-113.43</td>
<td>±0.005</td>
<td>0.055</td>
</tr>
<tr>
<td>3-Methyl-1-butene</td>
<td>F and M</td>
<td>-153.1</td>
<td>-153.1</td>
<td>±0.01</td>
<td>0.030</td>
</tr>
<tr>
<td>2-Methyl-2-butene</td>
<td>F and M</td>
<td>-135.08</td>
<td>-135.08</td>
<td>±0.015</td>
<td>0.0508</td>
</tr>
<tr>
<td>3-Methyl-2-pentene</td>
<td>M</td>
<td>-138.47</td>
<td>-138.46</td>
<td>±0.015</td>
<td>0.0509</td>
</tr>
<tr>
<td>2-Ethyl-1-butene</td>
<td>F and M</td>
<td>-15.55</td>
<td>-15.54</td>
<td>±0.010</td>
<td>0.0454</td>
</tr>
<tr>
<td>1-Heptene</td>
<td>M</td>
<td>-190.75</td>
<td>-190.67</td>
<td>±0.020</td>
<td>0.0509</td>
</tr>
<tr>
<td>2,4,4-Trimethyl-1-pentane</td>
<td>F and M</td>
<td>-93.50</td>
<td>-93.46</td>
<td>±0.010</td>
<td>0.0330</td>
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<tr>
<td>2,4,4-Trimethyl-2-pentane</td>
<td>F and M</td>
<td>-104.35</td>
<td>-104.35</td>
<td>±0.015</td>
<td>0.0300</td>
</tr>
<tr>
<td>Cyclohexenes</td>
<td>M</td>
<td>-103.51</td>
<td>-103.51</td>
<td>±0.015</td>
<td>0.0376</td>
</tr>
<tr>
<td>Acetylenes</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>1-Butyne (1-Butyne)</td>
<td>F and M</td>
<td>-125.75</td>
<td>-125.74</td>
<td>±0.020</td>
<td>0.035</td>
</tr>
</tbody>
</table>

Purification, Purity, and Freezing Points
cated: the kind of time-temperature curves, whether freezing or melting, used to determine the freezing point [8]; the freezing point of the actual sample, in air at 1 atm [8], for both the API-Standard and API-NBS lots; the calculated value of the freezing point for zero impurity [8]; the value of the cryoscopic constant, determined from the lowering of the freezing point on the addition of a known amount of an appropriate impurity [8]; and the resulting calculated amount of impurity in the API-Standard and the API-NBS materials.

Grateful acknowledgment is made to the other organizations and individuals listed in section II of this report for their contributions of materials for use in this work.

V. References

[9] American Petroleum Institute Research Project 44 at the National Bureau of Standards. Selected values of properties of hydrocarbons. Tables 1z, 2z, 3z, 5z, 6z, 7z, and 8z.

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**Figure 1.** Results of the first and only distillation of the first lot of 2,2-dimethylpropane (neopentane).

This material had not been fractionated by the supplier. Regular distillation at atmospheric pressure in Still 1 (5/2/45 to 5/25/45).

**Figure 2.** Results of the first and only distillation of a second lot of 2,2-dimethylpropane (neopentane).

This material had been fractionated by the supplier. Regular distillation at atmospheric pressure in Still 1 (1/21/46 to 2/28/46).
**Figure 3.** Results of the purification by adsorption of 2,2,3-trimethylbutane.
Adsonption in Column 10 (4/16/47); column 10 (5/8/47).

**Figure 4.** Results of the purification by adsorption of 2,2,4-trimethylpentane.
Adsonption in Column 10 (4/8/47).

**Figure 5.** Results of the first distillation of 2,2,3-trimethylhexane.
Regular distillation at 725 mm Hg in Still 12 (10/7/46 to 11/1/46).

**Figure 6.** Results of the second and final distillation of 2,2,3-trimethylhexane.
Azeotropic distillation with ethylene glycol monoethyl ether at 725 mm Hg in Still 3A (7/24/47 to 8/25/47).

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Figure 7. Results of the first distillation of 2,2,4-trimethylhexane.
Azeotropic distillation with ethylene glycol monoethyl ether at 725 mm Hg in Still 13 (10/21/44 to 11/15/44).

Figure 8. Results of the second distillation of 2,2,4-trimethylhexane.
Regular distillation at 725 mm Hg in Still 12 (9/5/45 to 9/20/45).

Figure 9. Results of the third and final distillation of 2,2,4-trimethylhexane.
Azeotropic distillation with ethylene glycol monoethyl ether at 725 mm Hg in Still 1A (1/8/47 to 2/9/47).

Figure 10. Results of the first distillation of 2,3,3-trimethylhexane.
Regular distillation at 725 mm Hg in Still 12 (8/21/46 to 9/11/46).

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FIGURE 11. Results of the second and final distillation of 2,3,3-trimethylhexane.
Azeotropic distillation with ethylene glycol monomethyl ether at 725 mm Hg in Still 4 (12/27/46 to 1/22/47).

FIGURE 12. Results of the first distillation of 2,3,5-trimethylhexane.
Regular distillation at 725 mm Hg in Still 12 (7/29/46 to 8/19/46).

FIGURE 13. Results of the second and final distillation of 2,3,5-trimethylhexane.
Azeotropic distillation with ethylene glycol monomethyl ether at 725 mm Hg in Still 11A (10/24/46 to 11/24/46).

FIGURE 14. Results of the first distillation of 3,3,4-trimethylhexane.
Regular distillation at 725 mm Hg in Still 12 (9/12/46 to 10/4/46).

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Figure 15. Results of the second and final distillation of 3,3,4-trimethylhexane.

Azeotropic distillation with ethylene glycol monophenyl ether at 725 mm Hg in Still 15A (8/19/47 to 9/8/47).

Figure 17. Results of the first distillation of cis and trans-1,3-dimethylcyclopentane.

Regular distillation at 725 mm Hg in Still 4 (7/3/46 to 7/27/46). One of two distillations of similar material. See footnote j of table 1. Fractions 1 to 167 (marked "x") were redistilled to obtain trans-1,3-dimethylcyclopentane (see fig. 21 and footnote z of table 1). Fractions 168 to 283 (marked "x'") were redistilled to obtain cis-1,3-dimethylcyclopentane (see fig. 18 and footnote k of table 1).
Figure 18. Results of the second distillation of cis-1,3-dimethyleclopentane. Regular distillation at 725 mm Hg in Still 15A (4/25/47 to 6/17/47). See footnote k of table 1.

Figure 19. Results of the third distillation of cis-1,3-dimethyleclopentane. Azeotropic distillation with ethanol at 725 mm Hg in Still 4 (6/26/47 to 8/18/47).
Figure 20. Results of the fourth and final distillation of cis-1,3-dimethylcyclopentane.

Azeotropic distillation with ethanol at 725 mm Hg in Still 2A (8/14/47 to 9/22/47).

Figure 21. Results of the second distillation of trans-1,3-dimethylcyclopentane.

Regular distillation at 725 mm Hg in Still 1A (2/14/47 to 4/3/47). See footnote z of table 1. The part marked “X’” was used as part of the charge for the concentrate of cis isomer. (See footnote k of table 1, and fig. 18.)
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Figure 22. Results of the third distillation of trans-1,3-dimethyleclopentane.
Azeotropic distillation with ethanol at 725 mm Hg in Still 3A (4/17/47 to 5/29/47).

Figure 23. Results of the fourth and final distillation of trans-1,3-dimethyleclopentane.
Azeotropic distillation with ethanol at 725 mm Hg in Still 2A (7/30/47 to 8/13/47).

Figure 24. Results of the first distillation of cis,cis,cis-1,2,3-trimethyleclopentane.
Regular distillation at 725 mm Hg in Still 4 (11/22/46 to 12/5/46).

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Figure 25. Results of the second and final distillation of cis,cis,cis-1,2,3-trimethylcyclopentane.
Azeotropic distillation with ethylene glycol monomethyl ether at 725 mm Hg in Still 1A (12/24/46 to 1/8/47).

Figure 26. Results of the first distillation of cis,trans,cis-1,2,3-trimethylcyclopentane.
Azeotropic distillation with ethylene glycol monomethyl ether at 725 mm Hg in Still 1A (12/26/46 to 1/3/47).

Figure 27. Results of the second and final distillation of cis,trans,cis-1,2,3-trimethylcyclopentane.
Azeotropic distillation with ethylene glycol monomethyl ether at 725 mm Hg in Still 4 (2/4/47 to 2/11/47). (See footnote m of table 1.)
Figure 28. Results of the first distillation of n-butylcyclopentane.
Regular distillation at 725 mm Hg in Still 14 (12/16/46 to 1/7/47).
Figure 29. Results of the second and final distillation of n-butyleclopentane.
Azeotropic distillation with ethylene glycol monoethyl ether at 725 mm Hg in Still A (2/25/47 to 4/14/47).

Figure 30. Results of the first distillation of 1,2-diethylbenzene.
Regular distillation at 725 mm Hg in Still 4 (4/2/47 to 5/8/47). The part of the distillate marked "X" received its final purification by adsorption. (See fig. 31 and footnote n of table 1.)
Figure 31. Results of the final purification of 1,2-diethylbenzene by adsorption.

See legend for figure 30 and footnote n of table 1. The part of the filtrate from each of the first 7 charges marked "X'" was blended together for subsequent charges 8 to 13.
Figure 32. Results of the first distillation of 1,3-diethyl-benzene.

Azotrope distillation with diethylene glycol monomethyl ether at 725 mm Hg in Still 8 (11/10/44 to 11/28/44).

Figure 33. Results of the second and final distillation of 1,3-diethylbenzene.

Regular distillation at 725 mm Hg in Still 12 (10/26/45 to 11/16/45). See footnote o of table 1.

Figure 34. Results of the first distillation of 1,4-diethylbenzene.

Regular distillation at 725 mm Hg in Still 9 (1/6/47 to 2/10/47). The distillate marked "X" was given its final purification by adsorption. (See results in columns 13 and 11 in fig. 36.)

Figure 35. Results of the second distillation of 1,4-diethylbenzene.

Azotrope distillation with diethylene glycol monomethyl ether at 725 mm Hg in Still 11A (4/17/47 to 5/19/47). The part of the distillate marked "X" was blended and divided into three similar charges for final purification by adsorption. (See results in columns 1, 2, and 3 in fig. 36.)
Figure 36. Results of the final purification of 1,4-diethylbenzene by adsorption.

See legends for figures 34 and 35 and footnote p of table 1.

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Figure 37. Results of the first and only distillation of 1-pentene.
Regular distillation at 725 mm Hg in Still 3A (1/21/47 to 3/24/47).
Figure 38. Results of the first distillation of the first lot of 1-hexene.
Regular distillation at 725 mm Hg in Still 9 (9/30/46 to 11/1/46).

Figure 39. Results of the second and final distillation of the first lot of 1-hexene.
Regular distillation at 725 mm Hg in Still 4 (2/13/47 to 3/4/47).
See footnote r of table 1.
Figure 40. Results of the first distillation of a second lot of 1-hexene.

Regular distillation at 725 mm Hg in Still 13 (12/9/46 to 1/20/47).
Figure 41. Results of the second and final distillation of the second lot of 1-hexene.

Azeotropic distillation with ethanol at 725 mm Hg in Still 9 (2/11/47 to 3/24/47). See footnote r of table 1.

Figure 42. Results of the first distillation of trans-2-hexene.

Regular distillation at 725 mm Hg in Still 11A (12/17/46 to 1/20/47). One of two distillations of similar material.

Figure 43. Results of the second and final distillation of trans-2-hexene.

Regular distillation at 725 mm Hg in Still 2A (3/13/47 to 4/7/47). See footnotes s and t of table 1.

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Figure 44. Results of the first distillation of trans-3-hexene.
Regular distillation at 725 mm Hg in Still 13 (7/20/46 to 9/3/46).

Figure 45. Results of the second and final distillation of trans-3-hexene.
Regular distillation at 725 mm Hg in Still 11A (11/27/46 to 12/16/46).
FIGURE 46. Results of the first distillation of 3-methyl-1-pentene.
Regular distillation at 725 mm Hg in Still 9 (11/4/46 to 12/30/46). One of two distillations of similar material. See footnote u of table 1.

FIGURE 47. Results of the second and final distillation of 3-methyl-1-pentene.
Regular distillation at 725 mm Hg in Still 11A (5/29/47 to 6/16/47).

FIGURE 48. Results of the first and only distillation of 2-methyl-2-pentene.
Regular distillation at 725 mm Hg in Still 4 (5/28/47 to 6/26/47).
Figure 49. Results of the first distillation of 3-methyl-cis-2-pentene.

Regular distillation at 725 mm Hg in Still 8 (12/12/45 to 1/21/46). One of three distillations of similar material. See footnote w of table 1.
Figure 50. Results of the second distillation of 3-methyl-cis-2-pentene.
Regular distillation at 726 mm Hg in still 2A (4/8/47 to 5/19/47). See footnotes w and x to table 1.

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Figure 51. Results of the third and final distillation of 3-methyl-cis-2-pentene.
Azeotropic distillation with ethanol at 725 mm Hg in Still 12A (6/26/47 to 7/17/47).

Figure 52. Results of the first distillation of the first lot of 2-ethyl-1-butene.
Regular distillation at 725 mm Hg in Still 12 (2/27/46 to 3/11/46).

Figure 53. Results of the second and final distillation of the first lot of 2-ethyl-1-butene.
Azeotropic distillation with ethanol at 725 mm Hg in Still 3A (3/25/47 to 4/17/47). See footnotes a and b to table 1.
Figure 54. Results of the first distillation of the second lot of 2-ethyl-1-butene.

Regular distillation at 725 mm Hg in Still 10 (12/20/46 to 2/27/47).

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**Figure 55.** Results of the second and final distillation of the second lot of 2-ethyl-1-butene.

Azeotropic distillation with ethanol at 725 mm Hg in Still 4 (5/8/47 to 5/28/47.) See footnotes aa and bb to table 1.

**Figure 56.** Results of the first distillation of 1-heptene.

Regular distillation at 725 mm Hg in Still 12 (4/30/46 to 5/31/46).

**Figure 57.** Results of the second and final distillation of 1-heptene.

Regular distillation at 725 mm Hg in Still 4 (12/8/46 to 12/20/46).
Figure 58. Results of the first distribution of 2,4,4-trimethyl-1-pentene and 2,4,4-trimethyl-2-pentene.

Regular distillation at 725 mm Hg in Still 14 (12/19/45 to 1/12/46). One of four distillations of similar material. See footnote cc of table 1. Fractions 41 to 74 (marked "x") were redistilled to obtain 2,4,4-trimethyl-1-pentene (see fig. 59). Fractions 109 to 131 (marked "x") were redistilled to obtain 2,4,4-trimethyl-2-pentene (see fig. 61).
Figure 59. Results of the second distillation of 2,4,4-trimethyl-1-pentene.

Regular distillation at 725 mm Hg in Still 13 (4/16/46 to 5/20/46). See footnote d to table 1

Figure 60. Results of the third and final distillation of 2,4,4-trimethyl-1-pentene.

Azeotropic distillation with ethylene glycol monomethyl ether at 725 mm Hg in Still 15A (6/26/47 to 7/30/47).
Figure 61. Results of the second distillation of 2,4,4-trimethyl-2-pentene.

Regular distillation at 725 mm Hg in Still 10 (4/16/46 to 5/31/46). See footnote dd to table 1.

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Figure 62. Results of the third and final distillation of 2,4,4-trimethyl-2-pentene.

Azeotropic distillation with ethanol at 725 mm Hg in Still 11A (6/17/47 to 8/8/47).

Figure 64. Results of the first and only distillation of 1-butylene.

Regular distillation at atmospheric pressure in Still 1 (9/6/45 to 11/8/45).
Figure 63. Results of the first and only distillation of cyclohexene.

Regular distillation at 725 mm Hg in Still 10 (10/15/46 to 12/19/46).

WASHINGTON, May 21, 1948.

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