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U.S. DEPARTMENT OF COMMERCE/National Bureau of Standards

# Handbook for the Quality Assurance of Metrological Measurements

John K. Taylor and Henry V. Oppermann



The National Bureau of Standards<sup>1</sup> was established by an act of Congress on March 3, 1901. The Bureau's overall goal is to strengthen and advance the nation's science and technology and facilitate their effective application for public benefit. To this end, the Bureau conducts research and provides: (1) a basis for the nation's physical measurement system, (2) scientific and technological services for industry and government, (3) a technical basis for equity in trade, and (4) technical services to promote public safety. The Bureau's technical work is performed by the National Measurement Laboratory, the National Engineering Laboratory, the Institute for Computer Sciences and Technology, and the Institute for Materials Science and Engineering.

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- Reactor Radiation

<sup>1</sup>Headquarters and Laboratories at Gaithersburg, MD, unless otherwise noted; mailing address Gaithersburg, MD 20899.

<sup>2</sup>Some divisions within the center are located at Boulder, CO 80303.

<sup>3</sup>Located at Boulder, CO, with some elements at Gaithersburg, MD.

# Handbook for the Quality Assurance of Metrological Measurements

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## PREFACE

This handbook is the third of a series developed by the National Bureau of Standards for use in the management of the national weights and measures measurement system. The other two publications are:

NBS Handbook 143, "State Weights and Measures Laboratories, Program Handbook," and

NBS Special Publication 686, "State Weights and Measures Laboratories, Program Description and Directory."

State weights and measures laboratories are in the forefront in assuring equity in the marketplace. A key to their success in that role is their ability to provide high accuracy calibrations and measurements. Those calibrations and measurements must be traceable to the national standards and must be in statistical control. Statistical control depends on a quality measurement assurance program which, in turn, depends on a competent staff, and adequate facilities and equipment.

This handbook provides the basis for the quality measurement assurance program by documenting good laboratory practices (GLPs), good measurement practices (GMPs), and standard operations procedures (SOPs). Those following the guidance in this handbook will be assured that their measurement data are properly evaluated and limits of random and systematic error assigned.

State laboratories are expected to study and use the material in this handbook, tailoring the material to fit their unique requirements. Although the handbook was written with the specific needs of the state laboratories in mind, it should be useful to the wider audience of laboratories engaged in high accuracy metrological measurements.

The authors encourage the users to comment on their experience in the use of this material so that it can be improved.

Albert D. Tholen  
Office of Weights and Measures  
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Table 9.1 Use of Range to Estimate Standard Deviation

Number of Sets of Replicates k	Factor Degrees of Freedom	Number of Replicates in a Set				
		2	3	4	5	6
1	*					
	$d_2^*$	1.41	1.91	2.24	2.48	2.67
3	$\nu$	1.00	1.98	2.93	3.83	4.68
	*					
5	$d_2^*$	1.23	1.77	2.12	2.38	2.58
	$\nu$	2.83	5.86	8.44	11.1	13.6
10	*					
	$d_2^*$	1.19	1.74	2.10	2.36	2.56
15	$\nu$	4.59	9.31	13.9	18.4	22.6
	*					
20	$d_2^*$	1.16	1.72	2.08	2.34	2.55
	$\nu$	8.99	18.4	27.6	36.5	44.9
∞	*					
	$d_2^*$	1.15	1.71	2.07	2.34	2.54
	$\nu$	13.4	27.5	41.3	54.6	67.2
	*					
	$d_2^*$	1.14	1.70	2.07	2.33	2.54
	$\nu$	17.8	36.5	55.0	72.7	89.6
	*					
	$d_2^*$	1.13	1.69	2.06	2.33	2.53

$$\bar{R}/d_2^* \rightarrow \sigma$$

Intermediate values for  $d_2^*$  and  $\nu$  may be obtained by interpolation, or from the reference from which this table was adapted.

Example: If 10 sets of measurements were made and each set consisted of two measurements (duplicates), the value for  $d_2^*$  would be 1.16; if 15 sets of measurements were made and each set consisted of three measurements (triplicates) the value for  $d_2^*$  would be 1.71.

Adapted from Lloyd S. Nelson, J. Qual. Tech. 7 No. 1, January 1975.

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Table 9.2 Z-Factors for Two-sided Confidence Intervals  
for the Normal Distribution

<u>Confidence Level, %</u>	<u>Z Factor</u>
50	0.68
67	1.00
75	1.15
90	1.645
95	1.960
95.28	2.000
99.00	2.575
99.74	3
99.9934	4
99.99995	5
100 - $10^{-9}$	6
100 - $10^{-12}$	7
100 - $10^{-15}$	8
100 - $10^{-18.9}$	9
100 - $10^{-23}$	10



Table 9.3 Student t-Variate

*	80%	90%	95%	98%	99%	99.73% (z=3)
df	t <sub>.90</sub>	t <sub>.95</sub>	t <sub>.975</sub>	t <sub>.99</sub>	t <sub>.995</sub>	t <sub>.9985</sub>
1	3.078	6.314	12.706	31.821	63.657	235.80
2	1.886	2.920	4.303	6.965	9.925	19.207
3	1.638	2.353	3.182	4.541	5.841	9.219
4	1.533	2.132	2.776	3.747	4.604	6.620
5	1.476	2.015	2.571	3.365	4.032	5.507
6	1.440	1.943	2.447	3.143	3.707	4.904
7	1.415	1.895	2.365	2.998	3.499	4.530
8	1.397	1.860	2.306	2.896	3.355	4.277
9	1.383	1.833	2.262	2.821	3.250	4.094
10	1.372	1.812	2.228	2.764	3.169	3.957
11	1.363	1.796	2.201	2.718	3.106	3.850
12	1.356	1.782	2.179	2.681	3.055	3.764
13	1.350	1.771	2.160	2.650	3.012	3.694
14	1.345	1.761	2.145	2.624	2.977	3.636
15	1.341	1.753	2.131	2.602	2.947	3.586
16	1.337	1.746	2.120	2.583	2.921	3.544
17	1.333	1.740	2.110	2.567	2.898	3.507
18	1.330	1.734	2.101	2.552	2.878	3.475
19	1.328	1.729	2.093	2.539	2.861	3.447
20	1.325	1.725	2.086	2.528	2.845	3.422
25	1.316	1.708	2.060	2.485	2.787	3.330
30	1.310	1.697	2.042	2.457	2.750	3.270
40	1.303	1.684	2.021	2.423	2.704	3.199
60	1.296	1.671	2.000	2.390	2.660	3.130
∞	1.282	1.645	1.960	2.326	2.576	3.000

\*Columns to be used in calculating corresponding two-sided confidence interval. From: NBS Handbook 91 p. T-5; Last column from B.J. Joiner, J. Research NBS.

Table 9.4. Critical values for the F-Test,  $F_{0.975}$ 

$n_1$  = degrees of freedom for numerator

$n_2 \backslash n_1$	1	2	3	4	5	6	7	8	9	10	12	15	20	24	30	40
1	647.8	799.5	864.2	899.6	921.8	937.1	948.2	956.7	963.3	968.6	976.7	984.9	993.1	997.2	1001	1006
2	38.51	39.00	39.17	39.25	39.30	39.33	39.36	39.37	39.39	39.40	39.41	39.43	39.45	39.46	39.46	39.47
3	17.44	16.04	15.44	15.10	14.88	14.73	14.62	14.54	14.47	14.42	14.34	14.25	14.17	14.12	14.08	14.04
4	12.22	10.65	9.98	9.60	9.36	9.20	9.07	8.98	8.90	8.84	8.75	8.66	8.56	8.51	8.46	8.41
5	10.01	8.43	7.76	7.39	7.15	6.98	6.85	6.76	6.68	6.62	6.52	6.43	6.33	6.28	6.23	6.18
6	8.81	7.26	6.60	6.23	5.99	5.82	5.70	5.60	5.52	5.46	5.37	5.27	5.17	5.12	5.07	5.01
7	8.07	6.54	5.89	5.52	5.29	5.12	4.99	4.90	4.82	4.76	4.67	4.57	4.47	4.42	4.36	4.31
8	7.57	6.06	5.42	5.05	4.82	4.65	4.53	4.43	4.36	4.30	4.20	4.10	4.00	3.95	3.89	3.84
9	7.21	5.71	5.08	4.72	4.48	4.32	4.20	4.10	4.03	3.96	3.87	3.77	3.67	3.61	3.56	3.51
10	6.94	5.46	4.83	4.47	4.24	4.07	3.95	3.85	3.78	3.72	3.62	3.52	3.42	3.37	3.31	3.26
11	6.72	5.26	4.63	4.28	4.04	3.88	3.76	3.66	3.59	3.53	3.43	3.33	3.23	3.17	3.12	3.06
12	6.55	5.10	4.47	4.12	3.89	3.73	3.61	3.51	3.44	3.37	3.28	3.18	3.07	3.02	2.96	2.91
13	6.41	4.97	4.35	4.00	3.77	3.60	3.48	3.39	3.31	3.25	3.15	3.05	2.95	2.89	2.84	2.78
14	6.30	4.86	4.24	3.89	3.66	3.50	3.38	3.29	3.21	3.15	3.05	2.95	2.84	2.79	2.73	2.67
15	6.20	4.77	4.15	3.80	3.58	3.41	3.29	3.20	3.12	3.06	2.96	2.86	2.76	2.70	2.64	2.59
16	6.12	4.69	4.08	3.73	3.50	3.34	3.22	3.12	3.05	2.99	2.89	2.79	2.68	2.63	2.57	2.51
17	6.04	4.62	4.01	3.66	3.44	3.28	3.16	3.06	2.98	2.92	2.82	2.72	2.62	2.56	2.50	2.44
18	5.98	4.56	3.95	3.61	3.38	3.22	3.10	3.01	2.93	2.87	2.77	2.67	2.56	2.50	2.44	2.38
19	5.92	4.51	3.90	3.56	3.33	3.17	3.05	2.96	2.88	2.82	2.72	2.62	2.51	2.45	2.39	2.33
20	5.87	4.46	3.86	3.51	3.29	3.13	3.01	2.91	2.84	2.77	2.68	2.57	2.46	2.41	2.35	2.29
21	5.83	4.42	3.82	3.48	3.25	3.09	2.97	2.87	2.80	2.73	2.64	2.53	2.42	2.37	2.31	2.25
22	5.79	4.38	3.78	3.44	3.22	3.05	2.93	2.84	2.76	2.70	2.60	2.50	2.39	2.33	2.27	2.21
23	5.75	4.35	3.75	3.41	3.18	3.02	2.90	2.81	2.73	2.67	2.57	2.47	2.36	2.30	2.24	2.18
24	5.72	4.32	3.72	3.38	3.15	2.99	2.87	2.78	2.70	2.64	2.54	2.44	2.33	2.27	2.21	2.15
25	5.69	4.29	3.69	3.35	3.13	2.97	2.85	2.75	2.68	2.61	2.51	2.41	2.30	2.24	2.18	2.12
26	5.66	4.27	3.67	3.33	3.10	2.94	2.82	2.73	2.65	2.59	2.49	2.39	2.28	2.22	2.16	2.09
27	5.63	4.24	3.65	3.31	3.08	2.92	2.80	2.71	2.63	2.57	2.47	2.36	2.25	2.19	2.13	2.07
28	5.61	4.22	3.63	3.29	3.06	2.90	2.78	2.69	2.61	2.55	2.45	2.34	2.23	2.17	2.11	2.05
29	5.59	4.20	3.61	3.27	3.04	2.88	2.76	2.67	2.59	2.53	2.43	2.32	2.21	2.15	2.09	2.03
30	5.57	4.18	3.59	3.25	3.03	2.87	2.75	2.65	2.57	2.51	2.41	2.31	2.20	2.14	2.07	2.01
40	5.42	4.05	3.46	3.13	2.90	2.74	2.62	2.53	2.45	2.39	2.29	2.18	2.07	2.01	1.94	1.88
60	5.29	3.93	3.34	3.01	2.79	2.63	2.51	2.41	2.33	2.27	2.17	2.06	1.94	1.88	1.82	1.74
120	5.15	3.80	3.23	2.89	2.67	2.52	2.39	2.30	2.22	2.16	2.05	1.94	1.82	1.76	1.69	1.61
$\infty$	5.02	3.69	3.12	2.79	2.57	2.41	2.29	2.19	2.11	2.05	1.94	1.83	1.71	1.64	1.57	1.48

$n_2$  = degrees of freedom for denominator

For use for a one-tailed test of equality of standard deviation estimate at 2.5% level of confidence, or for a two-tailed test at 5% level of confidence.

Table 9.5. Critical Values for the F-test,  $F_{0.99}$ 

$\nu_2$	$\nu_1$	Degrees of freedom for numerator									
		1	2	3	4	5	6	7	8	9	10
10		10.04	7.56	6.55	5.99	5.64	5.39	5.20	5.06	4.94	4.85
11		9.65	7.21	6.22	5.67	5.32	5.07	4.89	4.74	4.63	4.54
12		9.33	6.93	5.95	5.41	5.06	4.82	4.64	4.50	4.39	4.30
13		9.07	6.70	5.74	5.21	4.86	4.62	4.44	4.30	4.19	4.10
14		8.86	6.51	5.56	5.04	4.69	4.46	4.28	4.14	4.03	3.94
15		8.68	6.36	5.42	4.89	4.56	4.32	4.14	4.00	3.89	3.80
16		8.53	6.23	5.29	4.77	4.44	4.20	4.03	3.89	3.78	3.69
17		8.40	6.11	5.18	4.67	4.34	4.10	3.93	3.79	3.68	3.59
18		8.29	6.01	5.09	4.58	4.25	4.01	3.84	3.71	3.60	3.51
19		8.18	5.93	5.01	4.50	4.17	3.94	3.77	3.63	3.52	3.43
20		8.10	5.85	4.94	4.43	4.10	3.87	3.70	3.56	3.46	3.37
22		7.95	5.72	4.82	4.31	3.99	3.76	3.59	3.45	3.35	3.26
24		7.82	5.61	4.72	4.22	3.90	3.67	3.50	3.36	3.26	3.17
26		7.72	5.53	4.64	4.14	3.82	3.59	3.42	3.29	3.18	3.09
28		7.64	5.45	4.57	4.07	3.75	3.53	3.36	3.23	3.12	3.03
30		7.56	5.39	4.51	4.02	3.70	3.47	3.30	3.17	3.07	2.98
35		7.42	5.27	4.40	3.91	3.59	3.37	3.20	3.07	2.96	2.88
40		7.31	5.18	4.31	3.83	3.51	3.29	3.12	2.99	2.89	2.80
45		7.23	5.11	4.25	3.77	3.45	3.23	3.07	2.94	2.83	2.74
50		7.17	5.06	4.20	3.72	3.41	3.19	3.02	2.89	2.78	2.70
55		7.12	5.01	4.16	3.68	3.37	3.15	2.98	2.85	2.75	2.66
60		7.08	4.98	4.13	3.65	3.34	3.12	2.95	2.82	2.72	2.63
65		7.04	4.95	4.10	3.62	3.31	3.09	2.93	2.80	2.69	2.61
70		7.01	4.92	4.07	3.60	3.29	3.07	2.91	2.78	2.67	2.59
75		6.99	4.90	4.05	3.58	3.27	3.05	2.89	2.76	2.65	2.57
80		6.96	4.88	4.04	3.56	3.25	3.04	2.87	2.74	2.64	2.55
85		6.94	4.86	4.02	3.55	3.24	3.02	2.86	2.73	2.62	2.54
90		6.93	4.85	4.01	3.53	3.23	3.01	2.84	2.72	2.61	2.52
95		6.91	4.84	3.99	3.52	3.22	3.00	2.83	2.70	2.60	2.51
100		6.90	4.82	3.98	3.51	3.21	2.99	2.82	2.69	2.59	2.50
105		6.88	4.81	3.97	3.50	3.20	2.98	2.81	2.69	2.58	2.49
110		6.87	4.80	3.96	3.49	3.19	2.97	2.81	2.68	2.57	2.49
115		6.86	4.79	3.96	3.49	3.18	2.96	2.80	2.67	2.57	2.48
120		6.85	4.79	3.95	3.48	3.17	2.96	2.79	2.66	2.56	2.47
$\infty$		6.63	4.61	3.78	3.32	3.02	2.80	2.64	2.51	2.41	2.32

For use for a one-tailed test of equality of standard deviation estimates at 1% level of confidence, or for a two-tailed test at 2% level of confidence.

Table 9.6. Factors for Two-sided Tolerance Intervals  
for the Normal Distribution

$\begin{matrix} P \\ n \end{matrix}$	$\gamma = 0.95$					$\gamma = 0.99$				
	0.75	0.90	0.95	0.99	0.999	0.75	0.90	0.95	0.99	0.999
2	22.858	32.019	37.674	48.430	60.573	114.363	160.193	188.491	242.300	303.054
3	5.922	8.380	9.916	12.861	16.208	13.378	18.930	22.401	29.055	36.616
4	3.779	5.369	6.370	8.299	10.502	6.614	9.398	11.150	14.527	18.383
5	3.002	4.275	5.079	6.634	8.415	4.643	6.612	7.855	10.260	13.015
6	2.604	3.712	4.414	5.775	7.337	3.743	5.337	6.345	8.301	10.548
7	2.361	3.369	4.007	5.248	6.676	3.233	4.613	5.488	7.187	9.142
8	2.197	3.136	3.732	4.891	6.226	2.905	4.147	4.936	6.468	8.234
9	2.078	2.967	3.532	4.631	5.899	2.677	3.822	4.550	5.966	7.600
10	1.987	2.839	3.379	4.433	5.649	2.508	3.582	4.265	5.594	7.129
11	1.916	2.737	3.259	4.277	5.452	2.378	3.397	4.045	5.308	6.766
12	1.858	2.655	3.162	4.150	5.291	2.274	3.250	3.870	5.079	6.477
13	1.810	2.587	3.081	4.044	5.158	2.190	3.130	3.727	4.893	6.240
14	1.770	2.529	3.012	3.955	5.045	2.120	3.029	3.608	4.737	6.043
15	1.735	2.480	2.954	3.878	4.949	2.060	2.945	3.507	4.605	5.876
16	1.705	2.437	2.903	3.812	4.865	2.009	2.872	3.421	4.492	5.732
17	1.679	2.400	2.858	3.754	4.791	1.965	2.808	3.345	4.393	5.607
18	1.655	2.366	2.819	3.702	4.725	1.926	2.753	3.279	4.307	5.497
19	1.635	2.337	2.784	3.656	4.667	1.891	2.703	3.221	4.230	5.399
20	1.616	2.310	2.752	3.615	4.614	1.860	2.659	3.168	4.161	5.312
21	1.599	2.286	2.723	3.577	4.567	1.833	2.620	3.121	4.100	5.234
22	1.584	2.264	2.697	3.543	4.523	1.808	2.584	3.078	4.044	5.163
23	1.570	2.244	2.673	3.512	4.484	1.785	2.551	3.040	3.993	5.098
24	1.557	2.225	2.651	3.483	4.447	1.764	2.522	3.004	3.947	5.039
25	1.545	2.208	2.631	3.457	4.413	1.745	2.494	2.972	3.904	4.985
26	1.534	2.193	2.612	3.432	4.382	1.727	2.469	2.941	3.865	4.935
27	1.523	2.178	2.595	3.409	4.353	1.711	2.446	2.914	3.828	4.888

From: NBS Handbook 91, p T-11



Table 9.7. Factors for Computing Two-sided Confidence Limits for  $\sigma$

Degrees of Freedom $\nu$	$\alpha = .05$		$\alpha = .01$		$\alpha = .001$	
	$B_U$	$B_L$	$B_U$	$B_L$	$B_U$	$B_L$
1	17.79	.3576	86.31	.2969	844.4	.2480
2	4.859	.4581	10.70	.3879	33.29	.3291
3	3.183	.5178	5.449	.4453	11.65	.3824
4	2.567	.5590	3.892	.4865	6.938	.4218
5	2.248	.5899	3.175	.5182	5.085	.4529
6	2.052	.6143	2.764	.5437	4.128	.4784
7	1.918	.6344	2.498	.5650	3.551	.5000
8	1.820	.6513	2.311	.5830	3.167	.5186
9	1.746	.6657	2.173	.5987	2.894	.5348
10	1.686	.6784	2.065	.6125	2.689	.5492
11	1.638	.6896	1.980	.6248	2.530	.5621
12	1.598	.6995	1.909	.6358	2.402	.5738
13	1.564	.7084	1.851	.6458	2.298	.5845
14	1.534	.7166	1.801	.6549	2.210	.5942
15	1.509	.7240	1.758	.6632	2.136	.6032
16	1.486	.7308	1.721	.6710	2.073	.6116
17	1.466	.7372	1.688	.6781	2.017	.6193
18	1.448	.7430	1.658	.6848	1.968	.6266
19	1.432	.7484	1.632	.6909	1.925	.6333
20	1.417	.7535	1.609	.6968	1.886	.6397
21	1.404	.7582	1.587	.7022	1.851	.6457
22	1.391	.7627	1.568	.7074	1.820	.6514
23	1.380	.7669	1.550	.7122	1.791	.6568
24	1.370	.7709	1.533	.7169	1.765	.6619
25	1.360	.7747	1.518	.7212	1.741	.6668
26	1.351	.7783	1.504	.7253	1.719	.6713
27	1.343	.7817	1.491	.7293	1.698	.6758
28	1.335	.7849	1.479	.7331	1.679	.6800
29	1.327	.7880	1.467	.7367	1.661	.6841
30	1.321	.7909	1.457	.7401	1.645	.6880
31	1.314	.7937	1.447	.7434	1.629	.6917
32	1.308	.7964	1.437	.7467	1.615	.6953
33	1.302	.7990	1.428	.7497	1.601	.6987
34	1.296	.8015	1.420	.7526	1.588	.7020
35	1.291	.8039	1.412	.7554	1.576	.7052
36	1.286	.8062	1.404	.7582	1.564	.7083
37	1.281	.8085	1.397	.7608	1.553	.7113
38	1.277	.8106	1.390	.7633	1.543	.7141
39	1.272	.8126	1.383	.7658	1.533	.7169
40	1.268	.8146	1.377	.7681	1.523	.7197
41	1.264	.8166	1.371	.7705	1.515	.7223
42	1.260	.8184	1.365	.7727	1.506	.7248
43	1.257	.8202	1.360	.7748	1.498	.7273
44	1.253	.8220	1.355	.7769	1.490	.7297
45	1.249	.8237	1.349	.7789	1.482	.7320
46	1.246	.8253	1.345	.7809	1.475	.7342
47	1.243	.8269	1.340	.7828	1.468	.7364
48	1.240	.8285	1.335	.7847	1.462	.7386
49	1.237	.8300	1.331	.7864	1.455	.7407
50	1.234	.8314	1.327	.7882	1.449	.7427

From: NBS Handbook 91, p T-34



Table 9.8. Density of Air-free Water in g/cm<sup>3</sup> as a  
Function of Celsius Temperature

T	0.0°C	0.1°C	0.2°C	0.3°C	0.4°C	0.5°C	0.6°C	0.7°C	0.8°C	0.9°C
.0	.999840	.999846	.999853	.999859	.999865	.999871	.999877	.999883	.999888	.999893
1.0	.999899	.999903	.999908	.999913	.999917	.999921	.999925	.999929	.999933	.999937
2.0	.999940	.999943	.999946	.999949	.999952	.999954	.999956	.999959	.999961	.999963
3.0	.999964	.999966	.999967	.999968	.999969	.999970	.999971	.999971	.999972	.999972
4.0	.999972	.999972	.999972	.999971	.999971	.999970	.999969	.999968	.999967	.999965
5.0	.999964	.999962	.999960	.999958	.999956	.999954	.999951	.999949	.999946	.999943
6.0	.999940	.999937	.999933	.999930	.999926	.999922	.999918	.999914	.999910	.999906
7.0	.999901	.999896	.999892	.999887	.999881	.999876	.999871	.999865	.999860	.999854
8.0	.999848	.999842	.999835	.999829	.999822	.999816	.999809	.999802	.999795	.999787
9.0	.999780	.999773	.999765	.999757	.999749	.999741	.999733	.999725	.999716	.999707
10.0	.999699	.999690	.999681	.999672	.999662	.999653	.999643	.999634	.999624	.999614
11.0	.999604	.999594	.999583	.999573	.999562	.999552	.999541	.999530	.999519	.999507
12.0	.999496	.999485	.999473	.999461	.999449	.999437	.999425	.999413	.999401	.999388
13.0	.999376	.999363	.999350	.999337	.999324	.999311	.999297	.999284	.999270	.999256
14.0	.999243	.999229	.999215	.999200	.999186	.999172	.999157	.999142	.999128	.999113
15.0	.999098	.999083	.999067	.999052	.999036	.999021	.999005	.998989	.998973	.998957
16.0	.998941	.998925	.998908	.998892	.998875	.998858	.998841	.998824	.998807	.998790
17.0	.998773	.998755	.998738	.998720	.998702	.998684	.998666	.998648	.998630	.998612
18.0	.998593	.998575	.998556	.998537	.998519	.998500	.998480	.998461	.998442	.998422
19.0	.998403	.998383	.998364	.998344	.998324	.998304	.998284	.998263	.998243	.998222
20.0	.998202	.998181	.998160	.998139	.998118	.998097	.998076	.998055	.998033	.998012
21.0	.997990	.997968	.997947	.997925	.997903	.997881	.997858	.997836	.997814	.997791
22.0	.997768	.997746	.997723	.997700	.997677	.997654	.997630	.997607	.997584	.997560
23.0	.997536	.997513	.997489	.997465	.997441	.997417	.997392	.997368	.997344	.997319
24.0	.997294	.997270	.997245	.997220	.997195	.997170	.997145	.997119	.997094	.997068
25.0	.997043	.997017	.996991	.996966	.996940	.996913	.996887	.996861	.996835	.996808
26.0	.996782	.996755	.996723	.996702	.996675	.996648	.996621	.996593	.996566	.996539
27.0	.996511	.996484	.996456	.996428	.996401	.996373	.996345	.996316	.996288	.996260
28.0	.996232	.996203	.996175	.996146	.996117	.996088	.996060	.996031	.996001	.995972
29.0	.996943	.995914	.995884	.995855	.995825	.995795	.995765	.995736	.995706	.995676
30.0	.995645	.995615	.995585	.995554	.995524	.995493	.995463	.995432	.995401	.995370
31.0	.995339	.995308	.995277	.995246	.995214	.995183	.995151	.995120	.995088	.995056
32.0	.995024	.994992	.994960	.994928	.994896	.994864	.994831	.994799	.994766	.994734
33.0	.994701	.994668	.994635	.994602	.994569	.994536	.994503	.994470	.994436	.994403
34.0	.994369	.994336	.994302	.994268	.994234	.994201	.994167	.994132	.994098	.994064
35.0	.994030	.993995	.993961	.993926	.993891	.993857	.993822	.993787	.993752	.993717
36.0	.993682	.993647	.993611	.993576	.993541	.993505	.993469	.993434	.993398	.993362
37.0	.993326	.993290	.993254	.993218	.993182	.993146	.993109	.993073	.993036	.993000
38.0	.992963	.992926	.992889	.992852	.992815	.992778	.992741	.992704	.992667	.992629
39.0	.992592	.992554	.992517	.992479	.992442	.992404	.992366	.992328	.992290	.992252

\*Based on the work of H. Wagenbreth and W. Blanke, PTB - Mitteilungen 6-71.

Table 9.9. Density of Air,  $\text{g}/\text{cm}^3$ , as Function of Temperature and Pressure\*

Barometric Pressure				Temperature, C				
kPa	mbars	mm Hg	18	20	22	24	26	28
77.33	773	580	0.00092	0.00092	0.00091	0.00091	0.00090	0.00090
79.99	800	600	0.00095	0.00095	0.00094	0.00093	0.00093	0.00092
82.66	827	620	0.00099	0.00098	0.00097	0.00096	0.00096	0.00095
85.33	853	640	0.00102	0.00101	0.00100	0.00100	0.00099	0.00098
87.99	880	660	0.00105	0.00104	0.00103	0.00103	0.00102	0.00101
90.66	907	680	0.00108	0.00107	0.00107	0.00106	0.00105	0.00104
93.33	933	700	0.00111	0.00110	0.00110	0.00109	0.00108	0.00107
95.99	960	720	0.00114	0.00114	0.00113	0.00112	0.00112	0.00110
98.66	987	740	0.00118	0.00117	0.00116	0.00115	0.00114	0.00113
101.32	1013	760	0.00121	0.00120	0.00119	0.00118	0.00117	0.00117
103.99	1040	780	0.00124	0.00123	0.00122	0.00121	0.00121	0.00120
106.66	1067	800	0.00127	0.00126	0.00125	0.00124	0.00124	0.00123

Computed for air at 50% relative humidity.

\*An equation to compute the density of air at any pressure, temperature, and relative humidity is given in Section 8 of the Appendix to SOP No. 2.

Table 9.10. Coefficient of Expansion

Material	Coefficient of Expansion	
	Linear cm/°C	Cubical cm <sup>3</sup> /°C
aluminum	.000024	
brass	.000019	.000054
cast iron	.000010	.000030
copper	.000017	.000050
diamond	.00000118	.0000035
fused silica (quartz)	.0000005	.0000016
glass, borosilicate (TlCA)		.000010
glass, borosilicate (TlCB)		.000015
glass, soda-lime		.000025
invar	.0000004	.0000012
lead	.000029	.000087
length bench	.0000106	
mercury		.000018
platinum	.000011	.000027
polycarbonate plastic		.00045
polypropylene plastic		.00024
polystyrene plastic		.00021
steel, stainless	.000018	.000048
steel, tape, mild	.0000116	.0000335
steel, low carbon	.000012	.000035
steel, template	.000012	.000035
water (20 °C)		.000021

Table 9.11. A Short Table of Random Numbers

46	96	85	77	27	92	86	26	45	21	89	91	71	42	64	64	58	22	75	81	74	91	48	46	18
44	19	15	32	63	55	87	77	33	29	45	00	31	34	84	05	72	90	44	27	78	22	07	62	17
34	39	80	62	24	33	81	67	28	11	34	79	26	35	34	23	09	94	00	80	55	31	63	27	91
74	97	80	30	65	07	71	30	01	84	47	45	89	70	74	13	04	90	51	27	61	34	63	87	44
22	14	61	60	86	38	33	71	13	33	72	08	16	13	50	56	48	51	29	48	30	93	45	66	29
40	03	96	40	03	47	24	60	09	21	21	18	00	05	86	52	85	40	73	73	57	68	36	33	91
52	33	76	44	56	15	47	75	78	73	78	19	87	06	98	47	48	02	62	03	42	05	32	55	02
37	59	20	40	93	17	82	24	19	90	80	87	32	74	59	84	24	49	79	17	23	75	83	42	00
11	02	55	57	48	84	74	36	22	67	19	20	15	92	53	37	13	75	54	89	56	73	23	39	07
10	33	79	26	34	54	71	33	89	74	68	48	23	17	49	18	81	05	52	85	70	05	73	11	17
67	59	28	25	47	89	11	65	65	20	42	23	96	41	64	20	30	89	87	64	37	93	36	96	35
93	50	75	20	09	18	54	34	68	02	54	87	23	05	43	36	98	29	97	93	87	08	30	92	98
24	43	23	72	80	64	34	27	23	46	15	36	10	63	21	59	69	76	02	62	31	62	47	60	34
39	91	63	18	38	27	10	78	88	84	42	32	00	97	92	00	04	94	50	05	75	82	70	80	35
74	62	19	67	54	18	28	92	33	69	98	96	74	35	72	11	68	25	08	95	31	79	11	79	54
91	03	35	60	81	16	61	97	25	14	78	21	22	05	25	47	26	37	80	39	19	06	41	02	00
42	57	66	76	72	91	03	63	48	46	44	01	33	53	62	28	80	59	55	05	02	16	13	17	54
06	36	63	06	15	03	72	38	01	58	25	37	66	48	56	19	56	41	29	28	76	49	74	39	50
92	70	96	70	89	80	87	14	25	49	25	94	62	78	26	15	41	39	48	75	64	69	61	06	38
91	08	88	53	52	13	04	82	23	00	26	36	47	44	04	08	84	80	07	44	76	51	52	41	59
68	85	97	74	47	53	90	05	90	84	87	48	25	01	11	05	45	11	43	15	60	40	31	84	59
59	54	13	09	13	80	42	29	63	03	24	64	12	43	28	10	01	65	62	07	79	83	05	59	61
39	18	32	69	33	46	58	19	34	03	59	28	97	31	02	65	47	47	70	39	74	17	30	22	65
67	43	31	09	12	60	19	57	63	78	11	80	10	97	15	70	04	89	81	78	54	84	87	83	42
61	75	37	19	56	90	75	39	03	56	49	92	72	95	27	52	87	47	12	52	54	62	43	23	13
78	10	91	11	00	63	19	63	74	58	69	03	51	38	60	36	53	56	77	06	69	03	89	91	24
93	23	71	58	09	78	08	03	07	71	79	32	25	19	61	04	40	33	12	06	78	91	97	88	95
37	55	48	82	63	89	92	59	14	72	19	17	22	51	90	20	03	64	96	60	48	01	95	44	84
62	13	11	71	17	23	29	25	13	85	33	35	07	69	25	68	57	92	57	11	84	44	01	33	66
29	89	97	47	03	13	20	86	22	45	59	98	64	53	89	64	94	81	55	87	73	81	58	46	42
16	94	85	82	89	07	17	30	29	89	89	80	98	36	25	36	53	02	49	14	34	03	52	09	20
04	93	10	59	75	12	98	84	60	93	68	16	87	60	11	50	46	56	58	45	88	72	50	46	11
95	71	43	68	97	18	85	17	13	08	00	50	77	50	46	92	45	26	97	21	48	22	23	08	32
86	05	39	14	35	48	68	18	36	57	09	62	40	28	87	08	74	79	91	08	27	12	43	32	03
59	30	60	10	41	31	00	69	63	77	01	89	94	60	19	02	70	88	72	33	38	88	20	60	86
05	45	35	40	54	03	98	96	76	27	77	84	80	08	64	60	44	34	54	24	85	20	85	77	32
71	85	17	74	66	27	85	19	55	56	51	36	48	92	32	44	40	47	10	38	22	52	42	29	96
80	20	32	80	98	00	40	92	57	51	52	83	14	55	31	99	73	23	40	07	64	54	44	99	21
13	50	78	02	73	39	66	82	01	28	67	51	75	66	33	97	47	58	42	44	88	09	28	58	06
67	92	65	41	45	36	77	96	46	21	14	39	56	36	70	15	74	43	62	69	82	30	77	28	77
72	56	73	44	26	04	62	81	15	35	79	26	99	57	28	22	25	94	80	62	95	48	98	23	86
28	86	85	64	94	11	58	78	45	36	34	45	91	38	51	10	68	36	87	81	16	77	30	19	36
69	57	40	80	44	94	60	82	94	93	98	01	48	50	57	69	60	77	69	60	74	22	05	77	17
71	20	03	30	79	25	74	17	78	34	54	45	04	77	42	59	75	78	64	99	37	03	18	03	36
89	98	55	98	22	45	12	49	82	71	57	33	28	69	50	59	15	09	25	79	39	42	84	18	70
58	74	82	81	14	02	01	05	77	94	65	57	70	39	42	48	56	84	31	59	18	70	41	74	60
50	54	73	81	91	07	81	26	25	45	49	61	22	88	41	20	00	15	59	93	51	60	65	65	63
49	33	72	90	10	20	65	28	44	63	95	86	75	78	69	24	41	65	86	10	34	10	32	00	93
11	85	01	43	65	02	85	69	56	88	34	29	64	35	48	15	70	11	77	83	01	34	82	91	04
34	22	46	41	84	74	27	02	57	77	47	93	72	02	95	63	75	74	69	69	61	34	31	92	13

From: NBS Handbook 91, p T-82





## 10. GLOSSARY

Accuracy - closeness to the true or accepted (or nominal) value. (See inaccuracy).

Apparent mass vs brass - the mass of normal brass (density  $8.4 \text{ g/cm}^3$  at  $0^\circ\text{C}$  with a cubical coefficient of expansion of  $0.000054/^\circ\text{C}$ ) that produces the same balance reading as the object under test when the comparison is made at  $20^\circ\text{C}$  in air with a density of  $1.2 \text{ mg/cm}^3$ .

Bias - see systematic error

Calibration - comparison of a measurement standard or instrument with another standard or instrument to detect, correlate, report, or eliminate by adjustment any inaccuracy of the compared.

Check standard - an artifact that can be systematically measured to provide a data base whereby the performance characteristics of a measurement system may be judged. The check standard must be sufficiently similar to the items tested to permit valid inferences to be drawn from measurements on the former to those on the latter (see transfer standard).

Correction - that quantity when added algebraically to the nominal value defines its true value. Thus, if the true value is larger than the nominal value, the correction required is positive.

Conventions - a system of rules, generally accepted, for use in reporting measurement differences. The following conventions have been adopted for use when reporting differences (d) between nominal values (N) and the actual values (T) of standards (S) and test items (X):

$$\begin{aligned}S_T &= S_N + C_S \\X_T &= X_N + C_X\end{aligned}$$

Likewise, in comparing two values,

$$X - S = d$$

If d is positive, X exceeds S.

Density - the ratio of the mass of a portion of a substance to its volume; i.e. density = mass/volume.

Drainage Time - An arbitrarily specified time used in volumetric calibrations to permit the drainage of a reproducible amount of liquid from a volume standard or object calibrated and/or to establish a "wet down" condition. It is measured after flow cessation (which see).

Flow cessation - The moment when a full stream "breaks" and becomes a trickle or drip; important when measuring drainage time (which see).

Inaccuracy - deviation from the true or accepted (or nominal) value. Inaccuracy may result from both imprecision and bias in the measurement process.

Limiting mean - The value approached by the average of a sequence of independent measurements of the same quantity as the number of measurements included in the set approaches infinity.

Mass - the measure of the inertial property of a body. Qualitatively it can simply be thought of as the quantity of material in a body.

Measurement Assurance Program (MAP) - A quality assurance program for a measurement process that quantifies the total uncertainty of the measurements (both random and systematic components of error) with respect to national or other designated standards.

Measurement Process - A sequence of operations whose purpose is to assign a number(s) that represents how much of a certain property a given substance or object has.

Offset - The difference between a laboratory's assignment for a transfer standard and the NBS assigned value.

Precision - the degree of mutual agreement of independent measurements of a single quantity yielded by repeated applications of a process under specified conditions. It is quantitatively stated by a precision measure such as the standard deviation, for example.

Prover - a volumetric standard larger than 50 L in capacity.

Quality assessment - the procedures and activities utilized to verify that a quality control system is operating within acceptable limits, and to evaluate the quality of the product/measurement produced.

Quality control - the procedures and activities developed and implemented to produce products/measurements of desired quality.

Random errors - errors that vary in a non-reproducible way (fortuitously) around the limiting mean. For a large set of measurements, the errors are distributed evenly above and below the average. Also, small errors occur more frequently than large ones. These errors can be treated statistically by use of the laws of probability.

Standard deviation - a measure of the dispersion of individual members of a population about the limiting mean of the population. It is described by the symbol,  $\sigma$ , and is mathematically defined as the square root of the arithmetic mean of the squares of deviations of the various items from the arithmetic mean of the whole.

Standard deviation, - also called sample standard deviation (symbol,  $s$ ), a  
estimated a measure of the dispersion of individual members of a  
sample about the mean of the sample. If the sample is  
randomly selected from the population, the sample  
standard deviation is an estimate of the population  
standard deviation. (see Section 7 for calculations).

Standard deviation, - also called "between" standard deviation,  $s_b$ , a measure  
long term of the variability of measurements made at different  
times when using the same measurement procedure.

Standard deviation, - also called "within" standard deviation,  $s_w$ , a measure  
short term measure of the variability of a measurement process  
during a short period of time, usually the time necessary  
to complete one calibration/measurement using a particu-  
lar sequence of operations. It is useful in deciding how  
the precision of measurement is improved by replication  
at a given time.

Statistical control - a term that describes the operation of a measurement  
system in a stable and statistically predictable manner.  
A system so operating will produce measurements randomly  
distributed about a limiting mean with a fixed standard  
deviation.

Systematic errors - errors that are reproducible and tend to bias a result in  
one direction. Their causes can be assigned, at least in  
principle, and they can have both constant and variable  
components. These errors cannot be treated statistically.

Test measure - a volumetric standard up to and including 50 L (10 gal) in  
capacity.

Tolerance - the maximum allowable departure of a standard from its nominal  
value.

Traceability - the ability to relate an individual measurement result to  
national standards of measurement.

Transfer standard - an NBS calibrated artifact for use in evaluating a  
measurement process. It is most useful in evaluating the  
offset of a measurement process or reference standards.

True mass - the mass of a body. The adjective "true" is frequently used with  
the word "mass" to clearly differentiate from the apparent mass of  
a body.

Uncertainty - allowance assigned to a measured value to include two major  
components of error: bias and random error.





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11. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)  The general concept of quality assurance for metrological measurements is discussed. A number of Good Laboratory Practices (GLPs) and Good Measurement Practices (GMPs) related to metrology are compiled. Twenty recommended Standard Operations Procedures (SOPs) for high-accuracy mass, length, and volumetric calibrations made most frequently by State weights and measures laboratories are included. The statistical techniques useful for evaluating measurement quality are reviewed. Control charts most useful for metrological measurements are discussed.			
12. KEY WORDS (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons) Control charts; good laboratory practices; good measurement practices; length calibration; mass calibration; metrology; quality assurance; statistical techniques; volumetric calibration.			
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The practices and procedures presented in the handbook are based largely on the research and experience of numerous scientists at NBS, both named and unnamed, whose individual and collective goals have been to develop and utilize metrological procedures of the highest quality to serve the ever demanding needs of modern technology.

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## 1. INTRODUCTION

The quality of data must be known and established beyond a reasonable doubt before it can be used logically in any application. Data ordinarily are obtained for use in some decision process. In every such case, the questions of how good and how sure must be answered in quantitative terms. Data quality may be judged by comparison of its uncertainty with acceptable uncertainties related to its use. If the uncertainty of the data is negligible or relatively small, it may be considered to be of good or acceptable quality; if too large it may be considered poor quality or even useless. Obviously, the uncertainty must be known for data to be used in any logical way. Because absolute certainty can never be achieved, the limits of uncertainty are key issues and they must be estimated. It is the goal of laboratory quality assurance programs to provide the basis by which measurements can be carried out to produce reproducible results for which statistically supported uncertainty statements can be made.

The question of measurement assurance has been of prime concern to the National Bureau of Standards throughout the entire period of its existence. When calibration capabilities were transferred to the States, NBS also initiated a number of activities to assure the continuing quality of calibration data. These included the presentation to the States of primary standards of the highest quality, the development of minimum specifications for State laboratory facilities, presentation of basic and advanced training courses for metrologists, initiation and continuation of a series of laboratory problems to assist in the maintenance of competence, and a plan for certification of the capabilities of State weights and measures laboratories (21).

The system for certification of laboratories includes the development and implementation of a quality assurance program as a prime requirement. This handbook brings together, for the first time, much of the essential information needed in the development of a viable program. A brief overview of quality assurance as it is related to measurement programs is presented. A collection of good laboratory practices (GLPs) is presented next, followed by a series of documents describing good measurement practices (GMPs) related to physical measurements.

One of the features of the handbook is a compilation of standard operations procedures (SOPs) for calibrations and measurements made frequently by the State laboratories. These SOPs are detailed instructions for carrying out such measurements according to procedures recommended by the National Bureau of Standards. They comprise much of the instructional material of the basic and intermediate metrology seminars offered by NBS. The SOPs follow a standard format designed to specify all of the important steps to be followed in a sequential manner. SOPs are a basic part of any quality assurance program and are considered to be essential for systematic measurement leading to the attainment of a state of statistical control. The adoption and use of the SOPs included here will fully meet the requirement of the NBS Certification Plan for State Weights and Measures Laboratories for the use of documented test procedures.

A chapter containing miscellaneous subjects includes a discussion of how NBS supports the national measurement system (of which the State laboratories are key components), a discussion of the theory of tolerance testing as

contrasted to calibration, a simplified discussion of the concepts of mass and apparent mass, and a practical overview of the method of construction and use of control charts which are a basic part of any quality assurance program.

The handbook concludes with a review of the statistical techniques that are most often useful for the evaluation of measurement data, a collection of tables of statistical and physical data, and a glossary of a number of terms used in physical metrology and quality assurance. A bibliography of papers and books selected for their specific applicability to the quality assurance of measurements is included, as well.

This publication is not intended to replace but rather to complement basic sources of information. Indeed, it is assumed that the user is already familiar with elementary physics and metrology and has taken the NBS basic seminar, at the least. For example, equations are presented but not derived, since their derivation is presumed to be already familiar. In fact, if the reader has any difficulty in following the material presented here, it is recommended that he or she consult the corresponding material in the manuals of the basic or intermediate seminars or in general text books of physics.

As in any collection, the choice of material included is somewhat arbitrary. The selection principles followed included judgments of the frequency of need and degree of availability. The convenience resulting from collection of scattered materials into a single volume was a major consideration. Material that is readily available in numerous locations, was considered to be common knowledge, or was believed to be only rarely needed was intentionally excluded.

Because of the selectivity and the personal judgment involved, it is likely that some users of the handbook will find gaps in the information or may find some that is included to be of limited usefulness. Reader feedback on this Handbook is welcomed and will be very useful in influencing the contents of future editions.

One section that will be updated and added to is that containing the SOPs. The Office of Weights and Measures intends to develop SOPs for all measurements commonly made by State laboratories. The present edition contains SOPs for the measurements most frequently made. The Handbook is organized so that additional SOPs, GLPs, GMPs, and supplemental material may be easily inserted at a future date.

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NOTE: Certain commercial equipment, instruments, or materials are identified in this handbook to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.



## 2. CONCEPTS OF ACCURACY, PRECISION, AND STATISTICAL CONTROL

Accuracy is an intuitively understandable and desirable requirement for measurements. Data which are known to be inaccurate or whose accuracy is unknown have little appeal to most users. Yet precision is sometimes confused with accuracy and the agreement of successive results can inspire a degree of confidence that the measurements may not merit.

Accuracy, the closeness of a measured value to the true value, includes the concepts of bias and precision and is judged with respect to the use to be made of the data. A measurement process must be unbiased to be capable of producing accurate values. It must be sufficiently precise, as well, or else the individual results will be inaccurate due to unacceptable variability. The following discussion is presented to clarify these concepts. The term uncertainty is used widely in describing the results of measurement and denotes an estimate of the bounds of inaccuracy. Strictly speaking, the actual error of a reported value is usually unknowable. However, limits of error ordinarily can be inferred, with some risk of being incorrect, from the precision and reasonable limits for the possible bias of the measurement process.

The concept of precision is concerned with the variability of the individual results of replicate measurements. A process which shows a small scatter is said to be precise and vice versa. Obviously such judgments are subjective and based on the intended use of the data. What might be considered as very precise for one purpose could be grossly imprecise for another. Under constant conditions, random errors are responsible for the observed scatter of measured values. These may be reduced to the point at which they are negligible with respect to the tolerable error of the measured value, or are limited by inherent characteristics of the instrumentation or the methodology used. The averages of several series of measurements will show a smaller variability than the individual values and the grand average of such is expected to approach a limiting value (limiting mean) as the number of measurements is increased.

The concept of bias is concerned with whether or not the limiting mean differs from the true (or accepted) value of the property measured. Here again, judgment is ordinarily involved since it is impossible to eliminate all error or even to know if this has been achieved. Such decisions are thus based on whether or not bias exists for all practical purposes.

In the case of individual measurements, each will exhibit some degree of inaccuracy, that is to say it will deviate from the true value. This will occur because of random error together with any bias of the measurement system. Indeed, it is highly improbable that any individual measurement made by an unbiased measurement system will be accurate, since the probability of zero random error is zero. Many individual values may appear to have the correct value but this is due to truncation resulting from insensitivity of the measurement process or from rounding of the data.

A measurement process should be sufficiently precise to minimize the number of replicate measurements required for the intended use. A very precise system may need only a few measurements, even one, to provide data that would not be significantly improved by further replication. Also, a measurement system must be sufficiently precise to identify whether or not biases of a

comparable magnitude are present in the system. While possible in principle, an unbiased measurement process of low precision may be incapable of providing accurate data, from a practical point of view, because of the large number of measurements required to reduce the uncertainty due to the random error to reasonable limits.

Precision may be evaluated by the redundant process of replicate measurement. Results on a single object or material may be used for this purpose, or the information obtained on a number of objects or samples (even duplicate measurements) may be pooled. Accordingly, there is no reason why a laboratory cannot evaluate its own precision without external assistance [25]. While reference standards may be helpful in this regard, they are not necessary for this purpose.

In order to properly estimate precision, a large number of measurements over an extended period of time are required. A small number of measurements tend to underestimate the standard deviation since small random errors are more probable than large ones and less likely to be observed during a limited set of measurements. Also, it is common experience that it is much easier to repeat a measurement on a given occasion than to reproduce it over a period of time. The repeatability, or short term standard deviation is needed to answer questions about the number of repetitive measurements that may be required while the long term standard deviation, or reproducibility is needed to answer such questions as the agreement of data obtained at different times, or the statistical control of a measurement process.

Though precise measurements can serve useful purposes when limited comparisons are required, accuracy is more often an essential requirement. Whenever the true value of the measured quantity is needed, or when data from different laboratories, different methodologies, or that from the same laboratory using the same method over a period of time needs to be interrelated, bias can be a serious problem. Bias of a measurement process can only be evaluated by comparison of a measured value with the "true" value of the parameter that is measured. This requires the use of a reference standard whose value is known within acceptable limits of uncertainty. Since the measured value typically will differ somewhat from the reference value, a statistically based decision must be made on the significance of any observed difference.

The precision of a measurement system may be influenced by a number of factors, each having its own precision. The precision of each factor, quantified in terms of the variance, contributes to the precision of the process. The variance is simply the square of the standard deviation,  $s$ . In measurement processes, the variances of the individual steps,  $s_1^2$ , add up to define the variance of the process, i.e.,  $s^2 = s_1^2 + s_2^2 + s_3^2 + \dots + s_n^2$ . Some of the steps (or factors) can be easily identified and the individual variances estimated. As steps are identifiable, improvements conceivably can be made when there are "assignable causes" for undesirable imprecision. Because of addition in quadrature, it is evident that one or a few sources of variance can be the major contributors to the total variance. Knowledge of the magnitude of the individual variances can indicate both directions for improvement and possible sources of trouble when "out-of-control" measurements occur.



It is conceivable that variance can be reduced to very low levels, with diligent effort. Laboratories commonly improve their precision as they gain experience with their methodology. Ordinarily, a laboratory will improve its quality control practices to the point where the precision attained is adequate for a particular application or when peer performance has been attained. Because measurement must be pragmatic, cost-benefit decisions will often dictate how far to go. For example, it is a matter of record that laboratories using the same methodology will differ in their precisions. This may be due to differences in levels of skill but also to different levels of tolerance for permissible error.

Unlike random errors, systematic errors or biases from several sources are not necessarily randomly distributed; hence one must consider that biases can add up algebraically. That is to say, the total bias  $B = B_1 + B_2 + \dots + B_n$ . Thus, a large number of small biases can equal or even exceed a large bias from a single source. While the effect of random error decreases as the number of measurements,  $n$ , is increased ( $s_{\bar{x}} = s_x/\sqrt{n}$ ), the effect of bias is independent of the number of measurements.

A stable measurement system is expected to generate reproducible data. Statistical control may be defined as the attainment of a state of predictability. Under such a condition, the mean of a large number of measurements will approach a limiting value (limiting mean) and the individual measurements should have a stable distribution, described by their standard deviation. Under such a condition, the limits within which any new measured value would be expected to lie can be predicted with a specified probability, the confidence limits for a measurement or mean of set of measurements can be calculated, and the number of measurements required to obtain a mean value with a given confidence may be estimated.

It is axiomatic that attainment of statistical control is the first objective of a measurement process. This is just another way of stating that it must achieve stability. Yet, it has the further connotation that the data produced are statistically describable. Eisenhart has stated -- "Until a measurement operation has been 'debugged' to the extent that it has attained a state of statistical control it cannot be regarded in any logical sense as measuring anything at all" (12).

When a measurement system is altered or disturbed, a new or modified measurement system may result with a limiting mean and/or a standard deviation different from the previous values. During normal use of a measurement system, changes can occur as well, unbeknown to the laboratory personnel. A well designed quality assurance program will monitor the system for such changes and indicate when corrective actions are required.

Modern quality assurance is based on the premise that measurement can be established as a process that can be in a state of statistical control, achievable by applying the principles of quality control [7]. The output of such a process can be described statistically and limits can be assigned for the confidence of single measurements. In addition, bounds for bias can be estimated intelligently; hence, limits of uncertainty can be established for the data. Such data can be used to make decisions with statistically supported confidence since its quality is known.



The uncertainty statement is a necessary and critical part of reporting the results of calibration. As stated by Croarkin [11]:

The uncertainty statement assigns credible limits to the accuracy of the reported value stating to what extent that value may differ from its reference base. In practice it quantifies the magnitude of any possible discrepancy between the value actually obtained in the laboratory and the value which would be obtained at NBS for the same property of an object. An uncertainty provides both a measure of the worth of the values reported by the measurement laboratory and an estimate of the systematic error accruing to any organization that makes use of these values.

The uncertainty statement is composed of (i) all sources of systematic error that contribute to the offset from the reference base and (ii) a limit to random error that quantifies the variability that is inherent in the measurement process as it transfers from a "known" or calibrated artifact or measurement system to an "unknown."

The estimate of the standard deviation of a measurement process is the appropriate statistic for quantifying random error. Limits to random error are computed so as to cover a large percentage of possible measurement outcomes; i.e., limits to random error can be computed at the 99.73 percent confidence level. In some applications, the factor three is sufficient to achieve this confidence level; for this handbook where results may depend on relatively small number of measurements, exact limits based on Student's t-distribution are recommended.

The systematic errors included in the uncertainty statements in the SOPs are based upon the uncertainties associated with the reference standards. It is assumed that other sources of systematic error are negligible. If this assumption is not true, the systematic error from other sources must be included in the uncertainty statement. Consequently, the SOPs recommend that the uncertainty associated with a reported value be computed as the sum of possible bias due to the uncertainty in the values reported for the reference standards plus the appropriate t-statistic for the desired confidence level times the standard deviation of the measurement process.

The uncertainty associated with a measurement reported by a laboratory may be used as part of the basis of quality assessment. The confidence level associated with an uncertainty statement permits a user of the data to understand "how good" the reported value is and whether or not it is "good enough" for the user's needs. The uncertainty statements from different laboratories can be compared for the same types of measurements. Since the uncertainty statement represents credible limits on both random and systematic errors, the overall measurement capability of different laboratories can be compared. The validity of reported uncertainty statements can then be evaluated by other means, such as round robin testing. If the limits for random and systematic errors comprising the uncertainty statement are reported separately, the relative magnitudes of these errors for different laboratories may be compared to determine how well each component is controlled. This may provide a useful insight into the quality of a laboratory's measurement process.

### 3. QUALITY ASSURANCE IN A METROLOGY LABORATORY

#### Introduction

The limits of uncertainty of measurement data are of concern to both the serious metrologist and to the user of measurements. They permit the strength and weakness of each measured value to be evaluated. They support valid data and prevent the over interpretation of poor data. Precision estimates may be based on the results of replicate measurements while limits for bias depend on a critical analysis of sources of error of the measurement process. In research investigations, all of the above is done. In practical measurement situations, repetition must be minimized due to time and cost considerations, and bias is often evaluated on the basis of past experience. In fact, the objective of a good metrologist should be to conduct operations so that "individual measurements are good enough for their intended use" [7].

The experience of metrologists has demonstrated that data reliability is best achieved by a well-designed and operational quality assurance program. For this reason, most certification plans, and the NBS system for Certification of Capability of State Measurement Laboratories [21] require the existence of a quality assurance program as one of the criteria for certification.

#### What is Quality Assurance

Quality assurance consists of two separate but related activities, quality control and quality assessment. Both must be operational and coordinated. The following definitions are offered.

Quality assurance: A system of activities whose purpose is to provide to the producer or user of a product or a service the assurance that it meets defined standards of quality with a stated level of confidence.

Quality control: The overall system of activities whose purpose is to control the quality of a product or service so that it meets the needs of users. The aim is to provide quality that is satisfactory, adequate, dependable, and economic.

Quality assessment: The overall system of activities whose purpose is to provide assurance that the overall quality control job is being done effectively. It involves a continuing evaluation of the products produced and the performance of the production system.

Quality assurance is based on the premise that measurements can be made systematically by what may be called a measurement process, analogous to a manufacturing process. The product of a measurement process is measurement data that can be envisioned to attain a high degree of reproducibility similar to the reproducibility of the products of a well-controlled production process. In each case, reproducibility is obtained by quality control of the process. While the techniques used may be different, the concept is the same in both situations.

The quality of the product in each case can be evaluated by the second aspect of quality assurance, which may be called quality assessment. In the manufacturing process, the product is tested for conformance with specifications. In the measurement process, the assessment may be made by replicate measurements and by the measurement of a check standard. Ordinarily, and especially in the case of measurement, the production output is sampled and evidence is accumulated and maintained using control charts, for example, to verify the stability of the process and to set limits on the reliability of the data.

### Quality Control

Anything that may affect the production process must be optimized and stabilized to the extent necessary and possible, if reproducible products are to be obtained. In measurement processes, it is widely recognized that quality is influenced by many factors that can be classified in three categories: management practices; personnel; technical operations [21].

Management Practices: A well-managed laboratory is essential for reliable measurements. Both the calibre of the management staff and the policies it develops can influence data quality. Management sets the goals of the laboratory, provides resources and staff, and supervises the laboratory activities. Good management recognizes its responsibility to train and maintain staff competence, and develops policy and provides resources to accomplish this.

Needless to say, management must be skilled in its management responsibilities, with the skill requirement dependent on the size and complexity of the laboratory. In technical organizations, managerial skill is not enough. Managers must have a high level of technical competence, at least in the general aspects of the laboratory's operations. They must be able to evaluate the general quality of the laboratory output and to develop and administer the quality assurance aspect of its operations.

Personnel: A competent staff is an absolute necessity for quality measurements. This is often overlooked in today's highly mechanized and automated laboratories. Each member must have an educational background, supplemented with specific training and experience, sufficient for the duties to be performed. Each person must understand the responsibilities of his/her position (by suitable position descriptions and indoctrination) and must have the personal desire to perform them at a high level of competence.

Laboratory personnel are critical factors in the operational aspects of quality assurance [24]. Not only must they perform technical operations intelligently and skillfully, but in the full spirit of the quality assurance requirements. Strict adherence to appropriate GLPs, GMPs, and SOPs (see below), is essential and quality assessment must be carried out in the spirit of its purpose -- to realistically evaluate the measurement process and its outputs.

Technical Operations: All technical operations must be carried out in a reliable and consistent manner. The first requirement is for the use of suitable and properly maintained equipment and facilities. The equipment to be used ordinarily is specified in the SOPs (see below) but maintenance usually is



the responsibility of the operator. At least, the operator must verify its serviceability at the time of use. Calibration and calibration standards are closely related to equipment and these must be "operational" as well. Maintenance of facilities includes good housekeeping as well as environmental control. Both are essential for good measurements and both can affect data quality, introducing both systematic and random error.

Consistent and reliable data are dependent on the use of GLPs, GMPs, and SOPs as discussed in the following. GLPs (Good Laboratory Practices) denote those practices that the metrological community has developed to facilitate and promote reliable and reproducible measurements. GLPs are general and relate to most if not all of the activities and operations that a laboratory may conduct. For example, recording and maintaining data and records is related to all of the measurements of a laboratory. Because carelessness and inconsistencies can introduce error or uncertainties and raise questions of reliability, the procedures used for record keeping should be the subject of a GLP.

GMPs (Good Measurement Practices) describe recommended ways that specific technical operations are carried out that are closely related to but are not in themselves methods of measurement. GLPs may address some of the steps in SOPs (see later) that are assumed to be part of the art of measurement, hence are included only as general instructions. The method of reading a meniscus is an example. Because variability in such operations can introduce imprecision or bias, both within and between laboratories, critical ones are subjects for GMPs.

SOPs (Standard Operations Procedures) describe procedures to carry out methods for specific measurements. They consist of step-by-step instructions and all critical operations are specified. SOPs are central to the concept of measurement as a process. To qualify as a process, a measurement must be carried out in a highly specific and consistent manner. The SOP defines the modus operandi of the process. The term standard has several connotations in this regard. It may be, but is not necessarily, a method produced by the consensus action of a standards - writing organization. When such a method is available, its use should be given serious consideration. Whether or not one is used, an SOP adopted by a laboratory becomes the procedure to be followed precisely when that kind of measurement is made; thus it becomes the "standard method" for that laboratory.

### Quality Assessment

The term quality assessment describes those activities and procedures utilized to monitor the effectiveness of the quality control program and to evaluate the quality of the data output. There are both internal and external approaches for quality assessment and some of the features of each are described below.

Internal Approaches: Repetitive measurement is the key to evaluation of precision. Repetitive measurements of a test item (or sample) are always useful but this is expensive since at least 7 repetitions are required to estimate a standard deviation within any reasonable limits of uncertainty and 30 are desirable (see Chapter 8.7). Pooling of duplicate measurement data of

the same item or of similar items can be used to evaluate precision (see Chapter 8.4). At least 15 such duplicates are needed to estimate a standard deviation with reasonable confidence such as would be needed to establish control chart limits (see Chapter 7.4) which is a recommended way to use such data.

A check standard of reasonable stability can be used to monitor both precision and bias, if its value is known with sufficient accuracy. Historical data on a laboratory's own check standard can be used to develop a control chart and thus monitor and assess measurement precision.

The frequency of use of any of the above internal approaches to assess quality depends on several considerations. For an ongoing process, historical information on its stability will provide guidance together with the risks that are involved. Obviously all data taken within the period between the last known in-control and first known out-of-control are suspect. A prudent metrologist will design quality assessment procedures that will minimize such a risk.

Measurement operations carried out intermittently or occasionally present difficult problems for their quality assurance. In such cases, a sufficient number of preliminary measurements need to be made to assure that the process is in statistical control. This could require more effort than the actual measurements of the test item.

External Approaches: Any laboratory can evaluate its own precision and should do so before seeking external evaluation of its measurement accuracy. Until it has acquired the capability to do so, a laboratory is virtually unqualified to perform reliable measurements. The use of an externally provided check standard, certified by or on the basis of NBS traceability is an excellent way to evaluate the bias of a measurement system already demonstrated to be in statistical control. Participation in a MAP (measurement assurance program) [11] or in an RMMP (regional measurement management program) is an elegant way for measurement quality assessment. Participation in less formalized round robins provides other opportunities for quality assessment. Again, it is emphasized that the attainment of acceptable precision, based on a laboratory's internal quality assessment program is a prerequisite for meaningful participation in any external quality assessment activity.

Laboratory Audits: Audits are a valuable technique for quality assurance and may be of both internal and external origin. System audits consist of appropriate inspections to assess the adequacy of various aspects of the quality assurance system including facilities, equipment, records, and control charts. Some audits even include investigation of the qualifications of staff.

The objective of a system audit is to determine the operational characteristics of a laboratory's quality assurance practices. Internal audits usually use the laboratory's stated quality assurance policy or program as the basis of comparison. Externally conducted audits may use external standards for this purpose. The details of either type of audit are beyond the scope of this presentation but guidance will be found in the literature [26].

Laboratories are encouraged to conduct internal system audits at a level of scrutiny exceeding that of any external audit. When this is done, there



should be few surprises when an external audit is conducted. Indeed a good system of internal audits with adequate records thereof should minimize the need for external audits. The NBS system for Certification of the Capability of State Measurement Laboratories recognizes this in making self-appraisal a part of the certification process [21].

Performance audits consist of activities used to quantitatively evaluate measurement proficiency. A laboratory's internal quality assessment program is essentially an ongoing internal performance audit. External performance audits (MAPs and RMMPs are elegant examples of such) can identify bias that might be difficult to evaluate, internally.

#### Documentation

One aspect of quality assurance that merits emphasis is that of documentation. All data must be technically sound and legally defensible (that is to say, supportable by evidence of unquestionable reliability). Accordingly, a metrologist must keep adequate and accurate records on such things as:

- What is measured
- Who measures
- When measurements are made
- How measurements are made
  - Equipment
  - Calibration
  - Methodology
- Data obtained
- Calculations
- Quality assurance support
- Reports

Good metrologists have historically kept such information and a well-managed laboratory will automatically acquire and manage it. Its quality assurance program should address in detail the way that documentation is to be maintained.

#### Quality Assurance Program Document

The various aspects of the quality assurance practices that should be followed in a laboratory should be developed and described in a quality assurance program document [25]. This document will formally declare

management's commitment to all aspects of quality assurance and its support and enforcement of good laboratory practices and the quality assurance plan to be followed.

The quality assurance document should describe the maintenance procedures for facilities and equipment, the control charts to be maintained and the records of "out-of-control" that should be kept. The document should indicate the procedure to be followed for review of test reports and the mechanism by which the quality of data is assessed. Matters of safety and safe laboratory practices should be addressed.

An example of a quality assurance program document suitable for a State laboratory is given in reference [21].

### Responsibilities

A quality assurance program is only as effective as it is systematically implemented. Ordinarily, this means that a formal QA program must be established that documents the policy and the procedures to be followed. The establishment of policy is the responsibility of management. The development of QA procedures is a joint responsibility in which the technical staff has a major role because of its superior knowledge of technical requirements. When quality output is the objective of all concerned, the quality assurance program is not a disciplinary document but one that sets forth the way in which there is common belief that the work of the laboratory should be done.

#### 4. GOOD LABORATORY PRACTICES

- GLP No. 1 Measurement Control for Mass Calibration
- GLP No. 2 Minimum Requirements for the Physical Facilities for Weights and Measures Laboratory
- GLP No. 3 Care of State Standards
- GLP No. 4 Periodic Recalibration of State Standards
- GLP No. 5 Laboratory Administration
- GLP No. 6 Pre-requisites for Testing Measurement Standards and Equipment
- GLP No. 7 Systems Requirements - Military Standard 45662-Calibration
- GLP No. 8 Sealing of Equipment
- GLP No. 9 Reporting Mass Values and Uncertainties
- GLP No. 10 Purity of Water
- GLP No. 11 Painting Weights
- GLP No. 12 Laboratory Records
- GLP No. 13 Drying Containers



MEASUREMENT CONTROL FOR MASS CALIBRATION

Measurement assurance is knowing within the limits of a measurement process that a measurement is valid with respect to its accuracy and precision. This requires that both the standards and the measurement process must be in a state of statistical control. This implies that the variability of the measurement process is known so that the reported uncertainties are valid. The National Bureau of Standards is providing technical guidance and support to the State weights and measures laboratories to develop measurement control programs that provide measurement assurance. The objectives of these programs are to:

- establish and update the uncertainty statements for measurements
- document the validity of routine measurements
- monitor the laboratory standards
- monitor the metrologist

While other programs have been in use to meet these objectives, the control programs developed for measurement assurance greatly increase the comprehensiveness of the program with a minimum amount of additional workload for the State laboratories.

The State laboratories typically provide measurement services in the areas of mass, volume, and length. Some laboratories provide services in other measurement areas. The bulk of their workload is in tolerance testing and calibrating mass standards. The mass calibration area demands the greatest precision and is the first area in which a measurement control program was developed and implemented.

Almost all mass calibrations performed in State laboratories are on weight sets from 1 mg to 100 g. Normally, two balances are used to calibrate these weights. A measurement control program in this area consists of the metrologist intercomparing the State primary mass standards during the time a weight set is being calibrated. Three decades (sets) of State standards are intercompared in this process; the 100 g versus summation 50, 30, 20 g, the 10 g versus summation 5, 3, 2 g, and the 100 mg versus summation 50, 30, 20 mg. The 100 g decade is intercompared on a large capacity balance used in the calibration process and the 10 g and 100 mg decades are intercompared on a microbalance. These three additional measurements serve to meet all the objectives of a control program.

First, the measurements made on the standards must agree with the NBS reported values within the limits of the measurement process variability. If the measurements agree with the NBS values, this provides documentation that the measurement process is in control and the measurements made on the



standards in the weight set are assumed to be valid.\* If the results do not agree with the NBS values, then all the measurements made during the time of test must be repeated since they cannot be assumed to be valid.

The results of the measurements made on the known standards are plotted on control charts. The control charts are initially established by inter-comparing the standards over time. The variability in the data establishes the precision of the measurement process. Because the measurements are made throughout the year, all the parameters likely to affect the measurement process will be experienced and the combined effect reflected in the control chart. Any new measurements must be within the control limits on the charts for the measurement system to be in control.

The control charts are also used to monitor the stability of the standards. If the standards are changing, this will be evident by a trend in the data which can be readily observed from the control chart. The continuous collection of data will reveal this drift early in its development and permit the metrologist to take corrective action before the drift causes a serious problem.

If more than one person is performing the measurement, the data can be used also to determine if there is a difference in results dependent upon the operator of the balance. Ideally, there should not be a significant difference in results between operators, but data collected in this manner can be used to determine whether or not this is the case.

When data are collected over several years, the data on the standards are statistically analyzed to compare the results of previous years to the results of the current year. First, the average of the data on each decade of standards is compared using the t-test. Next, the variances are compared using the F-test. If the results are consistent, they are combined and a new control chart is made based upon the updated data. If problems or changes are revealed, they are investigated and corrective action taken. This establishes a continuous, comprehensive, internal measurement control program in a State laboratory.

The final aspect of the measurement control program is to verify that the results among State laboratories agree and that the results agree with NBS. This part of the program is accomplished through the Regional Measurement Management Program (RMMP) groups. These groups consist of the metrologists of the State laboratories in a geographic part of the country. Five groups are operating throughout the country.

These RMMP's perform round-robin testing on NBS calibrated standards in addition to conducting technical meetings to discuss test methods and address regional problems. The RMMP's develop round-robin experiments, coordinate the movement of the standards to the member labs, and analyze the data with guidance from NBS. The NBS assists in the investigation of problems. This

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\*Some laboratories use weighing designs that utilize check standards in each measurement of an unknown weight to document the validity of each measurement. This increases the workload considerably. The method above provides documentation with a minimum amount of additional work.

approach minimizes the NBS resources needed for this purpose. This establishes measurement control among laboratories with the NBS as the unifying base for the country. Formal NBS training is incorporated with the regional meetings.

The strength of the measurement assurance approach is that it is applicable to a wide range of measurements and is flexible enough to permit each measurement control program to be tailored to the particular needs of a given measurement area. The sophistication of a control program varies with the criticality of the measurement. If a measurement has relatively large tolerances and the laboratory equipment is very precise, a very simple measurement control system can be used. The advantage of a properly designed measurement assurance program is that a large amount of information can be obtained with a minimum amount of work.



MINIMUM REQUIREMENTS FOR THE PHYSICAL FACILITIES FOR  
WEIGHTS AND MEASURES LABORATORY

The physical facilities of a State weights and measures laboratory should meet the following minimum requirements, based on the space requirements for the equipment issued in the New State Standards Program. Many laboratories have acquired additional equipment and find that more space is necessary for efficient operations. In high production laboratories, even more space will be required.

General

1. Location. - The laboratory must be in a suitable location with respect to accessibility and freedom from excessive external vibration sources.
2. Temperature and Relative Humidity. - Facilities must be provided for the control of temperature to plus or minus 1 °C and maintain relative humidity between 35 and 55%.
3. Illumination. - The laboratory should be clean and well-lighted. The Lighting Handbook of the Illumination Engineering Society recommends 100 footcandles at bench level for precise laboratory measurements. Seventy footcandles at bench level is considered to be the minimum for a State weights and measures laboratory.
4. Space. - The overall amount and the general layout of the space should be adequate to facilitate the orderly conduct of the measurement operations.

Office space should be suitable and adapted to the needs of the measurement programs. Adequate filing equipment should be available to provide for the orderly maintenance of records.

Adequate storage space should be provided to accommodate test items awaiting test and/or disposal. Locked, dust-tight storage should be available to properly store all primary standards. Secondary standards should be housed to provide ready accessibility and yet to prevent damage due to environmental or other sources.

Facilities should be maintained in a clean and orderly manner to facilitate the measurement program and to inspire confidence in the users of the laboratory's services.

5. Miscellaneous. - Suitable equipment should be available and used to move the largest test objects in and out of the building and around balances without subjecting the balance or the weights to damage.

## The Mass Laboratory

1. Isolation from Vibration. - The laboratory must be free from any sources of vibration that would adversely affect the kind of measurement services that are offered. (See recommendation 5 below concerning location for installation of precision balances.)
2. Air Currents. - Air currents and air drafts must be held to the practicable minimum. Doors and windows must seal tightly. Heating and cooling ducts must be so positioned that air currents will not be directed toward the balances.
3. Isolation. - The mass laboratory, if possible, should be located away from external walls and should be isolated from any main pedestrian traffic flow.
4. Size. - The small mass laboratory must be at least 200 square feet in area (at least 350 square feet is recommended), with a minimum width of 12 feet. The large mass laboratory must be at least 350 square feet in area (at least 600 square feet is recommended). (When individual laboratories are combined in a single space, floor plans should be submitted to the Office of Weights and Measures for evaluation). In the case of multiple use, no type of use should jeopardize other uses of the space.
5. Location of Precision Balances.
  - a. Either the basement or the ground level of a building is the preferred location for balances.
  - b. Locations exposed to sunlight or to temperature fluctuations should be avoided.
  - c. In the case of high vibration areas, independent piers, with separate footings not in contact with the floor or other structural footings, are desirable. In this case, insulation must be soft and remain soft and should not be inserted until after the pier has set.
  - d. The balances should not be located in a building subject to high-level (low-frequency) vibration such as from jackhammers, jaw crushers, forges, and shakers.
  - e. If possible, locations adjoining streets with heavy bus or truck traffic should be avoided.



### The Length Laboratory

Size. - The length laboratory must be at least 120 square feet in area, with a minimum width of 6 feet, and a clear straight wall not less than 20 feet in length.

### The Volume Laboratory

1. Water Supply. - The volume laboratory must have hot and cold running water at a counter-top sink, and a source for distilled water must be available.
2. Size. - The volume laboratory must be at least 120 square feet in area, with a minimum width of 10 feet.



CARE OF STATE STANDARDS

State standards must be handled as primary standards whose integrity must be maintained and which must be protected at all times by an adequate chain of custody. Storage space for standards should be provided which is locked to prevent their unauthorized use and which is constructed to protect them from adverse environmental damage.

All standards should be properly stored at all times. Mass standards (30 kg and 50 lb) and smaller should be stored in their cases or in cabinets. They should not be left out when not in use, such as over-night. The 500-pound stainless steel and similar standards should be covered with plastic or stored in wood cases to protect them from dust and dirt in the air, and they should rest on a clean pad or blocks. All mass standards are to be placed on suitable pads to avoid abrading the bottoms.

Mass standards should never be handled with bare hands. Lifting tools, or clean gloves or cloths are always to be used. If the weights should become soiled, proper cleaning methods are to be used. Ordinarily, this will involve dusting with a camel-hair brush. If standards appear to be visibly dirty, or if there is reason to suspect contamination by dirt, the cleaning procedures described in GMP 5 or GMP 6 should be followed.

Standards, such as volumetric pipets, should be clamped securely in their holding brackets. They should be protected from physical damage as well as from dust and dirt, to the extent possible.





PERIODIC RECALIBRATION OF STATE STANDARDS

A number of States have provisions in their weights and measures laws requiring the periodic submission of their State standards to NBS for recalibration. Those provisions are based on an early version of the Model Law (1911) which was considered appropriate for the circumstances that prevailed prior to the establishment of the New State Standards Program by NBS. Periodic recalibration is now considered by NBS to be unnecessary and undesirable.

Standards of mass, volume, and length, fabricated from modern materials, kept in the controlled environment of a State metrology laboratory under the custody of trained metrologists, are generally stable and not subject to change. Moreover, the cooperative NBS-State audit programs would identify any such change in ample time for corrective action in the unlikely event that such a change should occur. These same programs provide the necessary traceability chain at a level of confidence sufficient for the need.

Moreover, the process of packing, shipping, and unpacking exposes the standards to unnecessary hazards that could result in damage, compromising their integrity. The return and recalibration could take several months causing an inavailability of State services which would be disruptive to the performance of the mission of the State laboratories.

In order to develop a policy for the guidance of and implementation by all 50 States regarding this subject, the following actions are recommended:

1. All States should recognize the fact that periodic return of their State standards to NBS for recalibration is unnecessary and undesirable for the reasons stated above,
2. Appropriate steps should be taken to amend any State laws that require periodic submission of the standards to NBS for recalibration purposes and,
3. References to the periodic recalibration of State standards in the law such as,

"He (the director) shall maintain the State standards in good order and shall submit them, at least once in ten years, to the National Bureau of Standards for certification,"

should be removed. In lieu thereof the wording of Sections 3 and 6 of the present Uniform Law should be substituted:

SECTION 3. PHYSICAL STANDARDS. -- Weights and measures that are traceable to the U. S. prototype standards supplied by the Federal Government, or approved as being satisfactory by the National Bureau of Standards, shall be the State primary standards of weights and measures, and shall be maintained in such calibration as prescribed by the National Bureau of Standards. All secondary standards may be prescribed by the director and shall be verified upon their initial receipt, and as often thereafter as deemed necessary by the director.

SECTION 6. POWERS AND DUTIES OF THE DIRECTOR. -- The director shall:

6.1 Maintain traceability of the State standards to the National Bureau of Standards.

The approach described above is recommended by NBS because each State that participates in the Office of Weights and Measures Laboratory Certification Program has the capability of certifying its own State standards with the necessary documentation referencing the national standards. The Laboratory Certification Program provides interaction between the State standards laboratories and NBS, assuring satisfactory laboratory conditions suitable for primary standards in addition to the proper use of NBS procedures in standards calibration. Thus, each State program is evaluated and, if found in compliance, is certified as being capable of performing the measurements listed on the laboratory certificate.

LABORATORY ADMINISTRATION

The following administrative practices are presented for guidance and are recommended to provide for the management of a metrology laboratory and to facilitate good measurements:

1. Maintenance of Standards and Instruments.

- 1.1. Standards of mass, length, and volume must be maintained with greatest care in order to preserve their accuracy.
- 1.2. Balances must receive regular maintenance and, because they are easily damaged, must be operated by no one other than a competent, trained technologist.

2. Laboratory Environment

- 2.1. For precision measurement and to preserve accuracy of standards, it is necessary to control laboratory temperature ( $\pm 1^{\circ}\text{C}$ ), relative humidity (35-55%), and to minimize other possible disturbing influences such as vibration and air currents.
- 2.2. In order to maintain a clean, well-controlled environment, access to the laboratory must be restricted to authorized personnel only. Traffic through the laboratory must be minimized.

3. Filing System

- 3.1. A practical filing system should be instituted that will enable the administrator to effectively maintain his/her records (calibration data, reports, etc.).
- 3.2. Separate files should be established to include the following categories:
  - 3.2.1. "Mass," "Length," "Volume," "Balances," "Laboratory Equipment." (Reference material.)
  - 3.2.2. "Laboratory Tests and Calibrations." (Annual data sheets, and reports.)
  - 3.2.3. "State Standards." (Reference standards, reports of calibration and tests.)
  - 3.2.4. "Reports of Calibrations and Tests."
  - 3.2.5. "Correspondence." (Regarding calibrations and tests.)

4. Numbering System for Tests.

- 4.1. All calibrations and tests should be numbered consecutively. A system consisting of digits indicating the year of the test, followed by a consecutive number for that particular year is recommended.
- 4.2. It is further recommended that the State name, abbreviation, or initial should precede the test number (Example: Test No. NY 86-123).
- 4.3. A master record should be kept of all test numbers, items tested, names of submitters, dates received, and dates returned.

5. Laboratory Services Promotion.

- 5.1. Contacts should be established and services made available to weights and measures jurisdictions, other local and State government agencies, industry, and educational and research organizations. The laboratory can provide valuable measurement services to the "grass roots" institutions of the States.

6. Training and Professional Organizations.

- 6.1. In order to provide a technically competent service to the State, the laboratory must be staffed by a skilled metrologist who can apply himself/herself to the profession on a full-time basis.
- 6.2. A continuous educational program must be pursued in order to develop and maintain the necessary professional skills.
- 6.3. Participation in NBS training programs is required.
- 6.4. Participation in related professional organizations that offer educational programs that may prove beneficial for the laboratory technologist and his/her laboratory activity is recommended.



PREREQUISITES FOR TESTING MEASUREMENT STANDARDS AND EQUIPMENT

All standards and equipment submitted for calibration or tolerance testing should be of good appearance and in good operating condition. It is futile to test and/or calibrate any item that does not meet the generally recognized minimum requirements set forth below. When items received do not meet these qualifications, they should be returned to the submitter for appropriate action.

Mass Standards

Weights and other devices must be free from all foreign matter, such as dirt, rust, concrete, grease, and other adhering substances. Old seals should be removed and plugs loosened unless "as found" values are needed. Spacers in adjustment cavities shall be furnished by the submitter.

Class "C" or Class "F" test weights, 50 pounds or heavier, must be freshly painted with an approved type paint, unless they are in extremely good condition. No painting or touching up should be done by laboratory personnel (unless special arrangements are made).

Cast Iron Weights

Cast iron weights should be cleaned thoroughly with a wire brush. If a weight is covered with numerous applications of paint that is beginning to chip, the weight should be cleaned with paint remover. Do not sand or shot blast or sandpaper cast iron weights. This method of cleaning removes metal as well as paint and could result in the weight being rejected.

Cast iron weights should be freshly painted with a good quality, thin coat of aluminum paint (not an enamel). (See also GLP No. 11).

Stainless Steel or Plated Weights

Stainless steel or plated weights should be washed in alcohol or with detergent and water to remove all extraneous matter. Care should be taken to avoid leaving a soapy film on the weight and to prevent the entrance of water into the adjustment cavity.

Apothecary or Metric Weights

Small weights (up to 100 grams) or weights that are calibrated, should not be cleaned in the field. If cleaning is necessary, it can be done better within the facilities in the laboratory. (See GMP No. 5).

Volume Standards

Test measures must be free from all foreign matter. Dents must be removed, and all leaks repaired. Unless a measure is in extremely good condition, it must be freshly painted with an approved type paint. Gauge tubes, reading scales, and other test measure components must be in working order, and in a reasonable good state of repair.

### Small Liquid Measures (Glassware and Nester Sets)

Small liquid measures should be thoroughly cleaned with soap and water and rinsed thoroughly to prevent a soap film residue.

### Field Test Measures

If a field test measure has been damaged, all dents should be removed and all leaks should be stopped. Some 5-gallon and smaller measures have plastic sight gauges that yellow with age. These should be replaced as necessary.

All field test measures should be freshly painted using a color appropriate for the intended use.

### Balances and Scales

All balances and scales should be cleaned and in working order when they are submitted, with oil in dash pots. Any loose parts should be tightened and broken parts replaced.

### Tank Trucks

All tank trucks shall be clean on the exterior. All safety valves must be operating properly when the truck arrives. All markers shall be loosened. Since this work is scheduled by appointment, trucks must arrive at the appointment time. Truck drivers or customer-designated personnel shall assist laboratory personnel as requested.

MILITARY STANDARD 45662

CALIBRATION SYSTEMS REQUIREMENTS

1. Scope

- 1.1 Scope. This standard provides for the establishment and maintenance of a calibration system to control the accuracy of the measuring and test equipment used to assure that supplies and services presented to the Government for acceptance are in conformance with prescribed technical requirements.
- 1.2 Applicability. This standard applies to all contracts under which the contractor is required to maintain measuring and test equipment in support of contract requirements.
- 1.3 Significance. This standard and any procedure or document executed in implementation thereof shall be in addition to and not in derogation of other contract requirements.

2. Reference Documents (Not Applicable)

3. Definitions

- 3.1 Calibration. Comparison of a measurement standard or instrument of known accuracy with another standard or instrument to detect, correlate, report, or eliminate by adjustment, any variation in the accuracy of the item being compared.
- 3.2 Measuring and test equipment. All devices used to measure, gauge, test, inspect, or otherwise examine items to determine compliance with specifications.
- 3.3 Measurement standard (reference). Standards of the highest accuracy order in a calibration system which establish the basic accuracy values for that system.
- 3.4 Measurement standard (transfer). Designated measuring equipment used in a calibration system as a medium for transferring the basic value of reference standards to lower echelon transfer standards or measuring and test equipment.
- 3.5 Traceability. The ability to relate individual measurement results to national standards or nationally accepted measurement systems through an unbroken chain of comparisons.

4. General Statements of Requirements

- 4.1 General. The contractor shall establish or adapt and maintain a system for the calibration of all measuring and test equipment used in fulfillment of his contractual requirements. The calibration system shall be coordinated with his Inspection or Quality Control Systems

and shall be designed to provide adequate accuracy in use of measuring and test equipment. All measuring and test equipment applicable to the contractor, whether used in the contractor's plant or at another source, shall be subject to such control as is necessary to assure conformance of supplies and services to contractual requirements. The calibration system shall provide for the prevention of inaccuracy by ready detection of deficiencies and timely positive action for their correction. The contractor shall make objective evidence of accuracy conformance readily available to the Government representative.

4.2 Quality assurance provisions. All operations performed by the contractor in compliance with this standard will be subject to Government verification at unscheduled intervals. Verification will include but not be limited to the following:

- a. Surveillance of calibration operations for conformance to the established system.
- b. Review of calibration results as necessary to assure accuracy of the system. The contractor's gauges, and measuring and testing devices shall be made available for use by the Government when required to determine conformance with contract requirements. If conditions warrant, contractor's personnel shall be made available for operation of such devices and for verification of their accuracy and condition.

## 5. Detailed Statements of Requirements.

5.1 Calibration system description. The contractor shall provide and maintain a written description of his calibration system covering measuring and test equipment and measurement standards to satisfy each requirement of this standard. The portion dealing with measuring and test equipment shall prescribe calibration intervals and sources and may be maintained for the documents normally used by the contractor to define his inspection operations. The description for calibration of measurement standards shall include a listing of the applicable measurement standards, both reference and transfer, and shall provide nomenclature, identification number, calibration interval and source, and environmental conditions under which the measurement standards will be applied and calibrated. The description of the calibration system and applicable procedures and reports of calibration shall be available to the Government representative.

5.2 Adequacy of standards. Standards established by the contractor for calibrating the measuring and test equipment used in controlling product quality shall have the capabilities for accuracy, stability, range, and resolution required for the intended use.

5.3 Environmental controls. Measuring and test equipment and measurement standards shall be calibrated and utilized in an environment controlled to the extent necessary to assure continued measurements of required accuracy giving due consideration to temperature, humidity, vibration, cleanliness, and other controllable factors affecting precision measurement. When applicable, compensating corrections



shall be applied to calibration results obtained in an environment which departs from standard conditions.

- 5.4 Intervals of calibration. Measuring and test equipment and measurement standards shall be calibrated at periodic intervals established on the basis of stability, purpose, and degree of usage. Intervals shall be shortened as required to assure continued accuracy as evidenced by the results of preceding calibrations and may be lengthened only when the results of previous calibrations provide definite indications that such action will not adversely affect the accuracy of the system. The contractor shall establish a system for the mandatory recall of standards and measuring and test equipment within established time limits or interval frequencies.
- 5.5 Calibration procedures. Written procedures shall be prepared or provided and utilized for calibration of all measuring and test equipment and measurement standards used to assure the accuracy of measurements involved in establishing product conformance. The procedures may be a compilation of published standard practices or manufacturer's written instructions and need not be rewritten to satisfy the requirements of this standard. As a minimum, the procedures shall specify the accuracy of the instrument being calibrated and the accuracy of the standards used. The procedure shall require that calibration be performed by comparison with higher accuracy level standards.
- 5.6 Out of Tolerance Evaluations.
- 5.6.1 Evaluation of suspect product. The contractor shall establish a procedure for the analysis of the impact of out of tolerance measuring and test equipment on product quality. The impact on quality of products examined or tested by equipment found to be out of tolerance during calibration will be determined and appropriate corrective action taken to correct product quality. Records of the result of the analysis and the corrective actions taken to maintain the required quality of the product shall be maintained and be available to the Government representative.
- 5.6.2 Evaluation of calibration system accuracy. The contractor shall establish a procedure to evaluate the adequacy of the calibration system based on out of tolerance data generated from calibrating test and measuring equipment. The procedure shall include but not be limited to adjustment of calibration frequency, adequacy of the measuring or test instrument, calibration procedures and measuring or test procedures. The procedures shall specifically provide for the identification and prevention of use of any equipment which does not perform satisfactorily.

## 5.7 Calibration Sources.

5.7.1 Domestic contracts. Measuring and test equipment shall be calibrated by the contractor or a commercial facility utilizing standards whose calibration is certified as being traceable to the National standards, has been derived from accepted values of natural physical constants, or has been derived by the ratio type of self-calibration techniques. Standards requiring calibration by a higher level standards laboratory shall be calibrated by a commercial facility capable of providing the required service, a Government laboratory under arrangements made by the contracting officer, or by the National Bureau of Standards. All standards used in the calibration system shall be supported by certificates, reports, or data sheets attesting to the date, accuracy, and environmental or other conditions under which the results furnished were obtained. Statements of certification shall contain as a minimum, the requirements prescribed in paragraph 5.8. All subordinate standards and measuring and test equipment shall be supported by like data when such information is essential to achieving the accuracy control required by this standard. In those cases where no data is required, a suitably annotated calibration label on the item shall be sufficient to satisfy the support data requirements of this paragraph. Certificates or reports from other than the National Bureau of Standards or Government laboratory shall attest to the fact that the standards used in obtaining the results have been compared at planned intervals with the National standard either directly or through a controlled system utilizing the methods outlined above. The contractor shall be responsible for assuring that the sources providing calibration services, other than the National Bureau of Standards or a Government laboratory, are in fact capable of performing the required service to the satisfaction of this standard. All certificates and reports shall be available for inspection by authorized Government representatives.

5.7.2 Foreign contracts. The provisions in paragraph 5.7.1 shall apply with the exception that the National standards laboratories of countries whose standards are compared with International or U. S. National standards may be utilized.

5.8 Application and Records. The application of the above requirements will be supported by records designed to assure that established schedules and procedures are followed to maintain the accuracy of all measuring and test equipment, and supporting standards. The records shall include an individual record of calibration or other means of control for each item of measuring and test equipment and measurement standards, providing description or identification of the item, calibration interval, date of last calibration, and calibration results of out of tolerance conditions. In addition, the individual record of any item whose accuracy must be reported via a calibration report or certificate will quote the report or certificate number for ready reference. These records shall be available for review by authorized Government personnel.

- 5.9 Calibration status. Measuring and test equipment and standards shall be labeled or some other suitable means shall be established for monitoring the equipment to assure adherence to calibration schedules. The system shall indicate date of last calibration, by whom calibrated and when the next calibration is due. The system may be automated or manual. Items which are not calibrated to their full capability or which require functional check only shall be labeled to indicate the applicable condition.
- 5.10 Control of subcontractor calibration. The contractor is responsible for assuring that the subcontractor's calibration system conforms to this standard to the degree necessary to assure compliance with contractual requirements.
- 5.11 Storage and handling. All measuring and test equipment shall be handled, stored and transported in a manner which shall not adversely affect the calibration or condition of the equipment.
- 5.12 Amendments and revisions. Whenever this standard is amended or revised subsequent to a contractually effective date, the contractor may follow or authorize his subcontractor to follow the amended or revised military standard provided no increase in price or fee is involved. The contractor shall not be required to follow the amended or revised standard except as a change in the contract. If the contractor elects to follow the amended or revised military standard, he shall notify the contracting officer in writing of this election.

## 6. Miscellaneous

- 6.1 Contract data requirements. The following Data Item Descriptions shall be utilized when the contract cites this standard and requires the contractor to develop and deliver data. The approved Data Item Description (DD Form 1664) required in connection with this standard and listed on the DD Form 1423 are as follows:

<u>Data Requirements</u>	<u>Applicable DID</u>	<u>This Standard Reference Para.</u>
Calibration System Description	DI-R-7064	5.1
Equipment Calibration Procedures	DI-R 7065	5.5
Procedures, Array, Calibration	UDI-T-23934	5.5
Calibration-- Maintenance Test Data	UDI-T-20340A	5.1 and 5.6
Reports; Test Procedures and Results, Calibration of Test Coupons for Propulsion Shafting	UDI-T-23801	5.1

(Copies of Data Item Description required by the Contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)





## SEALING OF EQUIPMENT

### Types of Seals and Their Locations

Most weights and measures jurisdictions require that all equipment officially approved for commercial use (with certain exceptions to be pointed out later) be suitably marked or sealed to show approval. The seal of approval should be as conspicuous as circumstances permit and should be of such a character and so applied that it will be reasonably permanent. Uniformity of position of the seal on similar types of equipment is also desirable as a further aid to the public.

The official will need more than one form of seal to meet the requirements of different kinds of equipment. Good quality, weather-resistant, water-adhesive, or pressure-sensitive seals or decalcomania seals are recommended for fabric-measuring devices, liquid-measuring devices, taximeters, and most scales, because of their permanence and good appearance. Steel stamps are most suitable for liquid and dry measures, for some types of linear measures, and for weights. An etched seal, applied with suitable etching ink, is excellent for steel tapes, and greatly preferable to a seal applied with a steel stamp. The only practicable seal for a graduate is one marked with a diamond or carbide pencil, or one etched with glass-marking ink. For a vehicle tank, the official may wish to devise a relatively large seal, perhaps of metal, with provision for stamping data relative to compartment capacities, the whole to be welded or otherwise permanently attached to the shell of the tank. In general, the lead-and-wire seal is suitable for use as an approval seal.

### Exceptions

Commercial equipment such as measure-containers, milk bottles, and lubricating-oil bottles are not tested individually because of the time element involved. Because manufacturing processes for these items are closely controlled, an essentially uniform product is produced by each manufacturer. The official normally tests samples of these items prior to their sale within his jurisdiction and subsequently makes spot checks by testing samples selected at random from new stocks.

Another exception to the general rule for sealing approved equipment is found in certain very small weights whose size precludes satisfactory stamping with a steel die.

## SEALING FIELD STANDARDS

The sealing of a field standard means that the standard or the carrying case for the standard is marked to indicate that the standard complies with the specifications for that particular type of standard. The mark aids the metrologist, the weights and measures official, and the service technician to identify standards that have been certified. In the case of adjustable standards, the seal is applied in such a manner that the seal must be destroyed before any adjustment can be made. This discourages unauthorized adjustments



to the standard. Frequently, the sealing mark includes the year in which the standard was last adjusted or certified so that this information is available immediately to the user of the standard.

A seal may be the authorized mark applied by the metrologist to a standard or a lead and wire security seal applied to the adjusting mechanism of a standard in a manner that prevents further adjustment without breaking the seal. The lead and wire seal is clamped to the standard using a hand press that imprints the authorized mark into the lead portion of the lead and wire security seal.

A weight with an adjusting cavity and sealing cap should be sealed following adjustment by stamping the authorized mark on the sealing cap following adjustment. Weights without sealing caps generally should not be sealed by stamping the weights since this mars the surface of the standard. Rather, the carrying case should be marked to identify that the weights in the set have been certified. This latter method of sealing is recommended for single piece and knob weights that do not have sealing caps.

Whenever a weight is adjusted, the sealing cap should be marked. Weights with lead sealing caps can be stamped each year the weights are certified by over stamping the previous stamp if adjustment is not necessary. Over stamping should not be done on aluminum sealing caps since the underlying mark will remain. In the case of aluminum caps, the original seal on the cap will indicate that the weight has not been adjusted since the date of the seal. The latest test report from the laboratory that tested the weight will disclose the date of test and certifies the weight to the appropriate tolerance class.

Metal graduated neck type volumetric field standards usually have a means of adjusting the graduated scale. A lead and wire security seal is normally used to seal the standard. It may be necessary to use more than one lead and wire seal to seal the adjustments on the standard.

Glass volume standards and metal length standards must be marked with an appropriate marking instrument. The mark must be placed on an area that will not interfere with the integrity and readability of the standard. Although these standards do not have adjustments, they should be tested periodically. Steel measuring tapes may change length over time. Recent certification reports may carry greater significance in the event they are needed in court.

REPORTING MASS VALUES AND UNCERTAINTIES

A mass calibration is not complete until the uncertainty associated with the calibration is determined and reported. In mass calibration, the uncertainty is taken as the sum of uncertainties associated with the standards plus three times the pooled standard deviation of the balance.

The guideline for reporting corrections to weights and the uncertainty is as follows:

The uncertainty should be reported to two significant figures. The correction should be reported to the last figure affected by the uncertainty.

Zeros which follow a decimal point, when there are only zeros ahead of the decimal point, are not considered significant figures.

The following rules should be used in rounding data, consistent with its significance:

1. When the digit next beyond the one to be retained is less than five, the retained figure is kept unchanged. For example: 2.541 becomes 2.5 to two significant figures.
2. When the digit next beyond the one to be retained is greater than five, the retained figure is increased by one. For example: 2.453 becomes 2.5 to two significant figures.
3. When the digit next beyond the one to be retained is exactly five, and the retained digit is even, it is left unchanged and conversely. Thus, 3.450 becomes 3.4 but 3.550 becomes 3.6 to two significant figures.
4. When two or more figures are to the right of the last figure to be retained, they are to be considered as a group in rounding decisions. Thus, in 2.4(501), the group (501) is considered to be  $>5$  while for 2.5(499), (499) is considered to be  $<5$ .

Several examples will be given to illustrate the proper method of reporting corrections and uncertainties.

#### Example 1

Suppose the correction for a weight is computed to be 1.3578 mg and the uncertainty is 0.5775 mg. The uncertainty is first rounded to two significant figures, that is, 0.58 mg. Then the correction is stated to be 1.36 mg. Notice that the uncertainty and the correction are stated to the same number of decimal places. The correction is reported as 1.36 mg  $\pm$  0.58 mg.

#### Example 2

Suppose the correction for a weight is computed to be 1.3578 and the uncertainty is 0.0135 mg. The uncertainty is first rounded to two significant figures, that is, 0.014 mg. (The first zero after the decimal point does not count.) Then the correction is rounded to the same number of decimal places as the uncertainty statement, that is, 1.358 mg. The correction is reported as  $1.358 \text{ mg} \pm 0.014 \text{ mg}$ .

#### Example 3

Suppose the correction for a weight is computed to be 4.3415 mg and the uncertainty is 2.0478 mg. The uncertainty is first rounded to two significant figures, that is, 2.0 mg. (Notice there are two significant figures present. The zero is a significant figure since it follows a non-zero number.) The correction is then rounded to the same number of decimal places as the uncertainty statement, that is, 4.3 mg. The correction is reported as  $4.3 \text{ mg} \pm 2.0 \text{ mg}$ .

#### Example 4

Suppose the correction for a weight is computed to be 285.41 mg and the uncertainty is 102.98 mg. Convert the values to grams. The uncertainty is first rounded to 0.10 g. The first nonzero digit (1) is the first significant figure and the remaining digits are rounded to the nearest number following the first nonzero digit. The correction is then rounded to the point where the rounding occurred in the uncertainty statement. The correction should be rounded to 0.29 g. The correction is reported as  $0.29 \text{ g} \pm 0.10 \text{ g}$ .

#### Example 5

Suppose the correction for a weight is computed to be 285.41 mg and the uncertainty is 33.4875 mg. The uncertainty is first rounded to two significant figures, that is 33 mg. The correction is then rounded to the same number of decimal places as the uncertainty statement, that is, 285 mg. The correction is reported as  $285 \text{ mg} \pm 33 \text{ mg}$ .

#### Example 6

Suppose the correction for a weight is computed to be 0.31653 lb and the uncertainty is 0.00565 lb. The uncertainty is first rounded to two significant figures, that is, 0.0056 lb. The correction is then rounded to the same number of decimal places as the uncertainty statement, that is, 0.3165 lb. The correction is reported as  $0.3165 \pm 0.0056 \text{ lb}$ .

Rather than state the uncertainty value with each correction, it is also proper to place the correction values in a column headed by the words "Mass Correction" or "Apparent Mass versus  $8.0 \text{ g/cm}^3$  Correction," etc., and place the uncertainties (without plus or minus signs) in a column headed "Uncertainty".



PURITY OF WATER

Water is used in two ways in the metrology laboratory. It may be used as a cleaning fluid or it may be used as a standard of volume for purposes of calibration. In each case, it must be clean; in the latter case it must be pure, as well.

Cleanliness of water may be achieved by removal of physical contaminating substances, especially by filtration. City water is ordinarily clean but may become dirty from the distribution system and especially from prolonged standing in some kinds of pipes and tubing. Hoses used to transfer water from and into large vessels and tanks may need internal cleaning, as well. Flushing to remove visible contamination is all that is usually required.

Clean water is all that is necessary when making measurements by volumetric transfer since only volumetric comparisons are involved. It is obvious that dirty water could cause a number of problems, including the leaving of residues in the volumetric vessels which could cause volumetric errors or could soil their interior, as a minimum. When in doubt of the cleanliness of the water supply, simple filters should be attached to the transfer lines used.

Pure water requires the removal of chemical contaminants and this may be achieved by distillation or by ion exchange systems. Pure water but not water of the highest purity ordinarily is all that is needed for the accuracy levels of state laboratories. Appreciable levels of dissolved salts may be reached before the density of water is changed significantly.

ASTM Type IV Water\* is recommended as fully adequate for calibration purposes. Such water may be produced by distillation or by ion exchange with relatively inexpensive equipment. Moreover, sales literature will usually specify whether the equipment will provide water of the above quality. An ion-exchange system is recommended for its simplicity and ease of operation. It can operate intermittently (on demand) and requires little or no maintenance except for change of cartridges, the need for which will be indicated.

A relatively small system (1-10 gal/hr) is adequate and it may be used on demand or to fill a small (5-10 gal) storage bottle to assure a continuous supply of calibration water. There are a number of commercial sources for such equipment.

There are two broad types of ion-exchange systems. Pressure cartridge systems (PCS) operate directly from line pressure (up to 100 psi) and need no special operation precautions. The less expensive alternative operates from the water line through a needle valve to produce a desired flow rate through the cartridge. In this system, the outlet must not become valved-off or the

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\*ASTM D 1193, Standard Specification for REAGENT WATER, ASTM, 1916 Race St., Philadelphia, PA 19103.

water pressure can build up and burst the cartridge. It is common practice to plumb directly from the output of this cartridge to a storage tank without using a valve in between. The unit is operated by simply turning the shut-off valve located at the water supply tap.



PAINTING WEIGHTS

Large weights should be painted both for their protection and to preserve their mass integrity. Unprotected weights are subject to corrosion. Furthermore, when corrosion is present, the extent and any changes resulting from it may be difficult to estimate.

Thin even coats of aluminum paint are recommended for this purpose. Spray applications are best if large weights or a number of small weights are to be painted. In preparation for painting, a weight should be cleaned and loose scale should be removed. The painting should be done before the weights are calibrated or tolerance tested.

Painting should be done in an area reserved for this purpose, or at least in a place which is removed from laboratory measurement operations. The weights should be protected from dust or dirt pick-up while the coating is drying.



LABORATORY RECORDS

Laboratory records, and especially laboratory data, are the only tangible outputs of a measurement laboratory. They form the basis for decision by the laboratory as well as the users of its outputs. Also, they provide the basis for evaluating measurement performance and for justifying courses of action. Accordingly they must be recorded with care and in such a manner as to make them understandable for both present and possible future use by both the original metrologist and by anyone else familiar with the art of the measurement process. The following guidelines are based upon the experience of the metrology community.

All data should be recorded in notebooks or on data sheets specifically designed for the purpose. In either case, a system of numbering or other means of identification should be used so that references and cross references can be made unequivocally. All entries should be made in ink (blue-black, or black) of a permanent variety. Pencil or pen with water-soluble ink is not acceptable.

All entries must be dark and clear enough for photocopy. Numbers should be made with sufficient deliberation so that they are definitive. For example, 4 and 9 or 3 and 8 can be difficult to distinguish if carelessly formed. Erasures are not permitted in notebooks or on data sheets. If an error is made it should be crossed out and the correct data recorded. Initials and dates in the margin should verify who made any change and the date thereof. In the case of extensive revisions, the rationale for such may need to be stated.

Clear identification of what was tested, how the measurements were made, and by whom are prime requirements. References to original sources such as calibration certificates, previous test results, and SOP's are satisfactory where applicable, but such references must be accurate. Any deviations from standard practices should be documented.

The identity of the sample or item tested must be unquestionable. This may require what is commonly called a chain-of-custody. This consists of a system of safeguards that need to be followed and a way to verify that the operations required have been carried out in the case of a given item or sample. This is always important but especially so when testing is done in a delayed manner, or where transportation and/or storage is involved. In the latter case, any possibility of change or alteration by natural or other causes needs to be eliminated.

The idea of a chain means that there should be positive and unbroken evidence that the sample has been safeguarded. Any gaps in this could open the test results to questions. In general, sample or test items are considered to be in custody when they are in the possession of an individual or in a sealed or locked storage area or shipment container. A custody form may need to be devised, in legally sensitive cases verifying, with signature and dates, the custody of the sample or item at all times.



DRYING CONTAINERS

Vessels calibrated "to contain" must be dried of all measurable water in order to obtain an "empty" weight. The drying process should not contaminate the container otherwise it will need to be recleaned before further calibration or volumetric use. The following is presented as guidance when drying is required.

Drain as much of the residual water as practical before starting any drying process. If time is not a consideration, a glass tube may be inserted into the container to pass clean dry air through it to evaporate the residual water film. A filter or dust trap may be necessary to pre-clean the air used. Alternately, air may be sucked through the tube connected to a vacuum pump with some danger of drawing in dirty air from the surroundings. Clean absorbent cotton placed at the neck opening can minimize the entrance of foreign matter.

Solvent cleaning may be used with alcohol as the preferred medium. Preliminary rinsing with acetone will remove large amounts of water, with which it is infinitely miscible, but this solvent often contains impurities such as traces of oils that could deposit on the container walls. Accordingly, a final alcohol rinse is recommended, even if acetone is used to remove most of the water. The alcohol is allowed to drain as much as possible, followed by air drying as before.

Some metal containers have been known to be internally coated to minimize corrosion. Accordingly, it should be ascertained that such coatings, when present, are not affected by alcohol or acetone, otherwise, solvent treatments should not be used.

The external surfaces of all containers should be clean when gravimetric calibration is used. Otherwise, any removal of external dust or dirt during the measurement process could cause errors of unknown magnitude. Cleaning should be done using detergents, as possible. If oily or greasy deposits need to be removed, solvents may be used, provided it is ascertained that they do not affect any external coatings on the container. The external surfaces should be dry before any calibrations are attempted.





## 5. GOOD MEASUREMENT PRACTICES

- GMP No. 1 Reading Turning Points on an Equal-Arm Balance
- GMP No. 2 Reading the Center of Graduations When Using a Microscope
- GMP No. 3 Method of Reading a Meniscus
- GMP No. 4 Adjusting the Optical Scale on a Single-Pan Mechanical Balance
- GMP No. 5 Weight Cleaning Procedures
- GMP No. 6 Cleaning Metal Volumetric Measures
- GMP No. 7 Cleaning Precision Glassware
- GMP No. 8 Recommended Form for Reporting Tape Calibrating
- GMP No. 9 Equations for Metallic Tapes
- GMP No. 10 Weighing Operations



READING TURNING POINTS ON AN EQUAL-ARM BALANCE

The damping of the beam oscillations of an equal-arm balance is generally very slow. Consequently, it is practical to read the turning points (the highest and lowest value of the beam oscillation) on the graduated scale and use the sum of the turning points as the observation value rather than wait for the beam to come to rest. This GMP requires that the graduated scale must be numbered such that adding weights to the left arm increases the readings.

Suppose the graduated scale has twenty graduations which are numbered from 0 to 20. The loads on the balance arms should be adjusted so that the sum of the turning points is approximately twice the midscale reading. In this example, the sum of the turning points should be within one division of twenty. Turning points should be estimated to at least 0.1 division in this example which is typical of the Russell balance. This means the final rest point is approximately ten which is the midscale reading. Motion should be induced to the beam so that the turning points can be read easily. Care should be taken so that the beam does not hit its limiting stops during its normal oscillation when turning points are being read. The amount of the beam oscillation is not critical although a span of from three to ten divisions is adequate.

Once motion has been induced for the beam oscillation, wait for at least one complete oscillation cycle for the beam motion to stabilize. After this time, the turning points can be read. The readings may begin with either the high or low turning point. The turning points for at least two consecutive oscillation cycles should be recorded. The turning points should reveal a consistent pattern of slow damping; that is, the turning points should gradually be converging to the eventual rest point. For example, if the last high turning point was greater than the previous high turning point (assuming the readings are normally dropping on previous readings), this would indicate that something has interfered with beam oscillation and the last reading was not valid with respect to the previous readings. Under these circumstances, turning points should continue to be read until a consistent damping pattern has been obtained. In some cases, the balance oscillation will dampen so slowly that the same readings may be obtained for several oscillations before a decrease is observed. These readings are valid and may be used to compute the sum of the turning points.

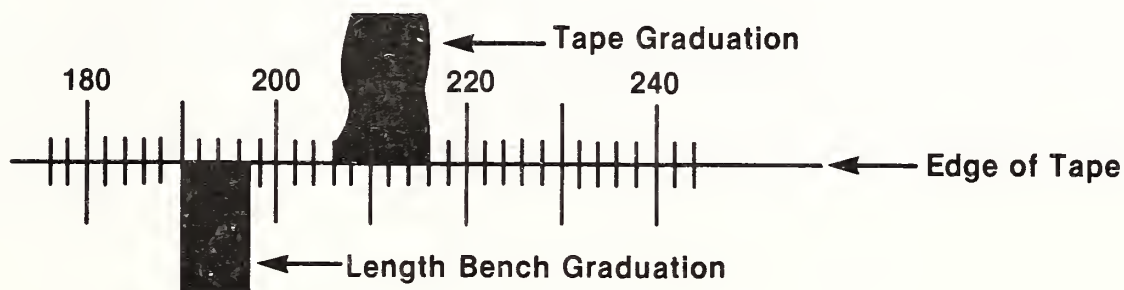
When at least four satisfactory turning points have been obtained (two high and two low turning points), all but the last three readings should be discarded. This will leave two high and one low turning point or vice versa. The two readings for the high or low turning points, as the case may be, should be averaged and added to the single turning point to obtain the sum of the turning points. The sum should be carried to two decimal places if the second decimal place digit is nonzero for the example above. As an example, the following readings were obtained: 15.5, 4.3, 15.4, 4.4. The sum of the turning points is computed as  $(4.3 + 4.4) \div 2 + 15.4 = 19.75$ .





READING THE CENTER OF GRADUATIONS WHEN USING A MICROSCOPE

The microscope furnished with the length bench has a reticle on which each graduation represents 0.002 inch. The difference in length between two tapes is measured from the center of the graduations of interest. The tape should be placed on the length bench such that the reading edge of the tape partially covers the graduations marked on the length bench. Place the microscope so that both the edges of the tape and the edge of the bench graduations are in the field-of-vision of the microscope and within reading range of the graduated reticle. Position the microscope so that part of the graduations on the reticle overlap the bottom portion of the graduation on the tape and part overlaps the graduation on the length bench. This setting is illustrated below. Do not move the microscope once the comparison between X and S has begun.



Determine the center of a graduation by first determining the width of the graduation, dividing the width by two and adding this value to the reticle reading for the left edge of the graduation. In the illustration above, let the length bench be the standard, S, and the tape be the unknown, X. The left edge of the tape graduation is at 206 (0.206 inch); the right edge is at 216 (0.216 inch). The width of the graduation is 0.010 inch. Adding half this value to 206 gives the center of the graduation at 211. Following the same procedure for the length bench graduation shows the left edge of the graduation at 190 and the right edge at 197. The center of the graduation is 193.5. The difference between the two tapes is  $211 - 193.5$  which is 0.0175 inch. Since X is longer than S, d is positive. Using the equation  $X - S = d$ ,

$$X - S = 0.0175 \text{ inch.}$$

or

$$X = S + 0.0175 \text{ inch.}$$

If it is necessary to calibrate a rigid rule to the ends of the rule, the edge of ends of the rule are reference points for the measurement. In these cases, there will be only a single value read from the microscope reticle for each end of the rule since the center of the graduation is assumed to be the end of the rules.

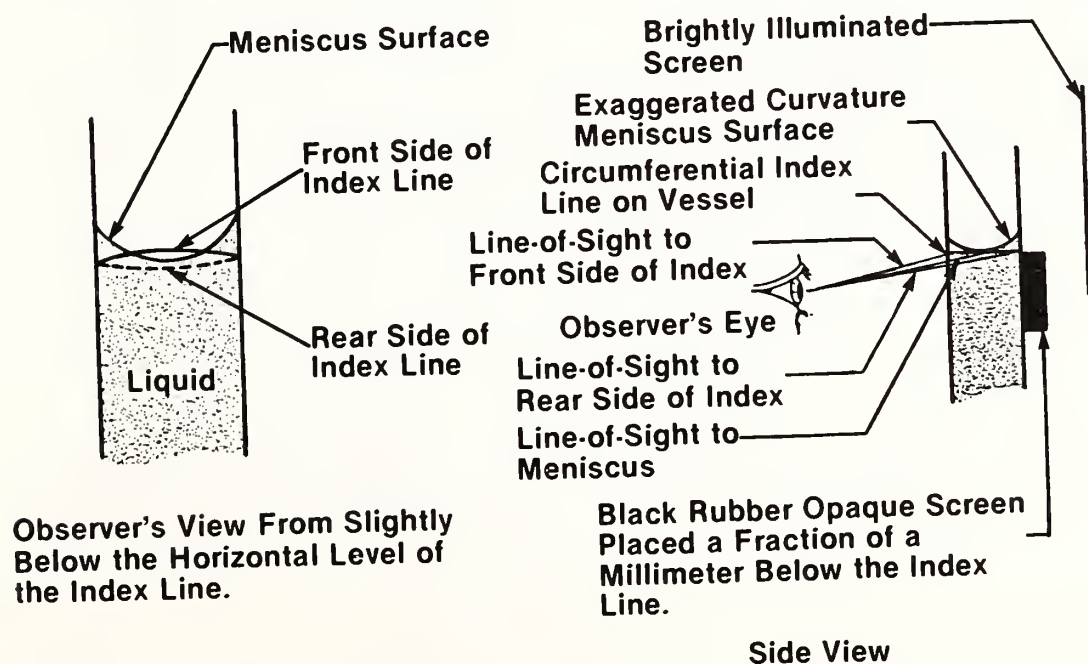


METHOD OF READING A MENISCUS

## Using Water or Other Wetting Liquid

In all apparatus where the volume is defined by a meniscus, the reading or setting is made on the lowest point of the meniscus. In order that the lowest point may be observed, it is necessary to place a shade of some dark material immediately below the meniscus, which renders the profile of the meniscus dark and clearly visible against a light background. A convenient device for this purpose is a collar-shaped section of thick black rubber tubing, cut open at one side and of such size as to clasp the tube firmly.

The position of the lowest point of the meniscus with reference to the graduation line is such that it is in the plane of the middle of the graduation line. This position of the meniscus is obtained by making the setting in the center of the ellipse formed by the graduation line on the front and the back of the tube as observed by having the eye slightly below the plane of the graduation line. This is illustrated below. The setting is accurate if, as the eye is raised and the ellipse narrows, the lowest point of the meniscus remains midway between the front and rear portions of the graduation line. By this method it is possible to observe the approach of the meniscus from either above or below the line to its proper setting.



The meniscus formed by a non-wetting liquid, such as mercury, is convex. The highest point of such a meniscus is used to make the reading. The reading of a mercurial barometer is a classical example of this kind. In making the reading, the observer's eye should be normal to and in the line of sight with the meniscus. The illumination is adjusted to get a sharp definition of the meniscus. Elimination of parallax error is very important and can be judged by slight fluctuations of eye level that do not affect the reading.

The curvature of a meniscus is related to the surface tension of the liquid and inversely related to the diameter of the tubing in which it is formed. When reading any meniscus, it is important to ascertain that it is in an equilibrium position. Tapping of sight glasses and/or small motions of containers may be used to induce slight displacements and the return to the same reading is evidence of a stable meniscus.

ADJUSTING THE OPTICAL SCALE ON A SINGLE PAN MECHANICAL BALANCE

When a single pan balance is used for comparing unknown weights to reference standards, it is desirable to adjust the optical scale so that all readings obtained in the intercomparison will be above zero. For the double substitution weighing design, the readings should begin in the first quarter of the optical scale. For a modified substitution, it is most useful to set the optical scale at its midpoint. An upscale reading can be obtained by several methods depending upon the features of the balance and the value of the weights being used in the intercomparison.

- I. Zero-adjust knob: If the coarse zero adjustment has sufficient range, the optical scale reading can be increased or decreased to obtain the desired reading. The fine zero adjustment is used to obtain the final reading.
- II. Tare-adjustment knob: If the balance has a tare adjustment feature, it can be used easily to reduce the optical scale reading. The tare adjustment also can be used effectively for weights that would normally give readings near zero when the optical scale starts at zero. This is accomplished by dialing in built-in weights such that the optical scale reading is at the maximum. The tare adjustments can then be used to reduce the reading on the optical scale.
- III. Small weights: If the weights under test would normally give readings near zero, small weights can be placed on the balance pan to increase the reading on the optical scale. These weights must remain on the pan throughout the intercomparison.

Small weights can be used in combination with the zero and tare adjustments to obtain the desired setting.

- IV. Internal zero adjustment: In some cases, the balance may be used to test weights that would normally give readings at the maximum range of the optical scale with the maximum setting of the built-in weights. An example of this would be the testing of a 1-kg weight on the Mettler CB 1000 balance. In this case there is no additional range on the optical scale to permit the performance of the sensitivity test portion of the weighing design. Under this condition, a sufficient amount of optical scale range can be obtained by adjusting the zero adjustment balance ball that is inside the balance. This adjustment would then permit the external zero adjust knobs to be used to obtain the final setting.

Once the desired optical scale setting has been obtained and the intercomparison has begun, no further adjustments should be made until the intercomparison has been completed.





WEIGHT CLEANING PROCEDURES\*

It is essential that weights being calibrated as well as the standards used be clean if the calibration is to be accurate and meaningful. Therefore, a cleaning procedure should be considered as a part of every calibration.

A. Categories of Weights

For cleaning purposes, weights may be divided into four categories:

1. One-piece weights.

This category will include all one-piece weights except lacquered weights, sheet metal weights, and small wire weights.

2. Screw-knob weights.

This category will include all weights with adjusting cavities except lacquered weights.

3. Lacquered weights.

This category includes all lacquered or painted weights.

4. Sheet metal weights and wire weights.

B. Cleaning Procedures

1. One-piece weights.

One-piece weights, one gram and larger, are steam cleaned. The weights are either held or placed in a jet of steam and manipulated so that the entire surface of the weight is subjected to the cleaning action of the steam long enough to clean it. A superficial steaming is not enough. The weight is then dried, either by evaporation or careful wiping with a soft non-abrasive material such as high grade cheese cloth, free from oil and other substances that could leave a residue on the weights. Care must be exercised that no water spots are left on the weights as they dry. Visible particles on the weights should be brushed or wiped off before steam cleaning them. If a steam generator is not available, one-piece weights may be cleaned either by immersing them in a hot or boiling distilled water bath in a non-metallic container, or according to the procedures for screw-knob weights. Occasionally, a weight will have foreign material adhering to it that requires the use of solvents. Ethyl alcohol is a good general solvent. If alcohol does not remove the material, other solvents may be used. Alcohol is then used to remove any film left by the other solvents. The weights are then steam cleaned as outlined above.

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\*From: H. E. Almer, "Weight Cleaning Procedures" NBSIR-74-443 (1974).

2. Screw-knob weights.

Weights in this category are usually cleaned by wiping with a soft non-abrasive material such as high grade cheesecloth, free from oils or other substances that would leave a residue of any kind on the surface. Occasionally, a weight will have foreign matter adhering to it that requires cleaning by using solvents, applied with a cloth. Ethyl alcohol is a good general solvent. If alcohol does not remove the foreign material, other solvents may be used. Alcohol is then used to remove any film left by the other solvents.

3. Lacquered weights.

The cleaning of lacquered weights requires special care because their protective lacquer coating is soluble in most solvents. Lacquered weights are cleaned by wiping with a soft non-abrasive material, free from oils and other substances that would contaminate the weights, or by brushing with a soft brush such as a camel hair brush, or both. A rubber bulb type syringe may be used to blow off lint or other small particles, however, be careful not to touch the weights with the nozzle. An electrostatic charge may be placed on the surfaces of the weights during the cleaning process or while handling them. This could be especially troublesome in a very dry atmosphere. If reliable mass values are to be obtained, the charge must be bled off of the weights before calibration.

4. Sheet metal weights.

Either of the following two procedures may be used in cleaning sheet metal and other small one-piece weights.

a. Two-step method.

First, the weights are placed in an acetone bath agitated to help loosen any foreign material. A soft brush, such as a camel hair brush, may be used to agitate the weights. The weights are removed from the acetone, allowed to dry and then steam cleaned. For steam cleaning, the weights are held in front of a jet of steam with forceps until the entire surface has been covered with steam. (See Note on next page). In order that the portion of the surface under the forceps may be steamed, the weight is set down and picked up again with the forceps holding the weight at a different spot than the first time; the weight is again steamed. Do not allow the weights to touch the steam nozzle. A low ash filter paper should be used for drying the sheet metal weights. A circular disk is folded unsymmetrically. The main body of the weight is placed between the folds of the paper with the turned up edge of the weight protruding. The main body of the weight is dried by pressing lightly on the top of the paper. The turned up edge is brushed lightly with a piece of filter paper. In some cases, it may be necessary to brush the body of the weight with filter paper to remove drops of water. Care must be exercised that no water spots are left on the weights as they dry.

b. Three-step method.

First, the weights are placed in an acetone bath and agitated with a soft brush to help loosen any foreign material adhering to the weights. The weights are removed from the acetone, allowed to dry, then placed in an ethyl alcohol bath and agitated. The weights are removed from the alcohol bath, allowed to dry, and then steam cleaned and dried as outlined in the two step procedure.

Note: The smaller fractional weights, say smaller than 1g, may be placed in a hot or boiling distilled water bath for the final cleaning instead of steam cleaning them. A hot or boiling distilled water bath also may be used for the final cleaning of all sheet metal weights when a steam generator is not available.

C. Cleaning Interval

1. Weights under test.

The weights under test are cleaned before calibration.

2. Standards.

Standards need not be cleaned every time they are used. If the standards are handled carefully, and kept under a reasonably dust tight cover, when not in use, in a clean atmosphere, the interval between cleanings may be several months, or until the calibration procedure checks indicate that the standards are changing and may need cleaning. Under less favorable conditions the interval may be only a few weeks.

D. Temperature Equilibrium

Newly cleaned weights should be allowed to come to temperature equilibrium before they are calibrated. This may take several hours for the larger weights that have been steam cleaned.

Generally, laboratory weights will come to temperature equilibrium over night.

E. Storage

Usually, weights are not placed in the balance immediately after cleaning, but are stored for varying periods. The weights should be stored under cover so that they will stay clean. Weights, one gram and larger, may be stored on a tray lined with filter paper and covered with an inverted glass dish. The smaller weights may be stored in a small glass dish covered with a watch glass. In both cases, the container should be labeled with the weight identification. When the weights are to be moved, carry the tray or dish in a level position so that the weights do not slide around.

When the weights are placed in the balance, they should be carefully brushed to remove any particles that may be on them. A small bulb type rubber syringe is useful in removing lint and other small particles from



weights. The particles are blown off the weights. Therefore, neither the nozzle nor any other part of the syringe need touch the weights, and care must be taken that they do not.

#### F. Brushes

All of the equipment used in cleaning and handling weights should, of course, be clean. But, brushes require special attention because they are easily contaminated and often are the last cleaning device used before the weights are calibrated. Only soft brushes, such as camel hair brushes, should be used on weights.

The brushes are cleaned by washing with soap and water, then rinsing in ethyl alcohol and allowed to dry in air. When drying the brushes, place them so that the bristles do not touch anything. New brushes are cleaned before using to remove any oil or other matter that might contaminate the weights. Used brushes are cleaned as often as necessary to be sure that the brushes themselves do not contaminate the weights. Store cleaned brushes in containers that will keep them clean until needed. When handling the brushes, do not touch the bristles, as oil from the skin will contaminate them. When the brushes are laid down, place them so that the bristles do not touch anything.

#### G. Steam Generator

A steam generator may be purchased from a laboratory supply house or one can be constructed.

A simple steam generator may be constructed from a 500 mL wash-bottle, a two hole stopper, some glass tubing, a hot plate for the heat source, a large graduate, and some flexible tubing.

#### H. Vapor Degreaser

Weights may be cleaned by vapor degreasing, using apparatus such as shown in Figure 1.

The apparatus consists of a stainless steel cylinder placed on a hot plate. The cylinder has several loops of copper tubing coiled around the outside center of the cylinder as cooling coils. The cylinder also has a cover to minimize the escape of vapors of the solvents used in the cleaning process. A basket is suspended from the cover inside the cylinder. A single weight or several small weights are placed in the basket. Solvent (e.g. ACS grade trichloro-ethylene) is placed in the cylinder to a depth of about 2.5 cm. The solvent is heated to boiling. Cold water is circulated through the cooling coils. The solvent condenses on the weight, thereby cleaning the weight, and drops off into the liquid at the bottom of the cylinder. The cleaning process is run for 15-20 minutes. The standards are then air dried and allowed to return to room temperature. The weights, when removed from the degreaser, are hot so that residual solvent evaporates in a few seconds. (Caution: Weights should be removed from the degreaser immediately after cleaning to prevent possible electrolytic corrosion.)



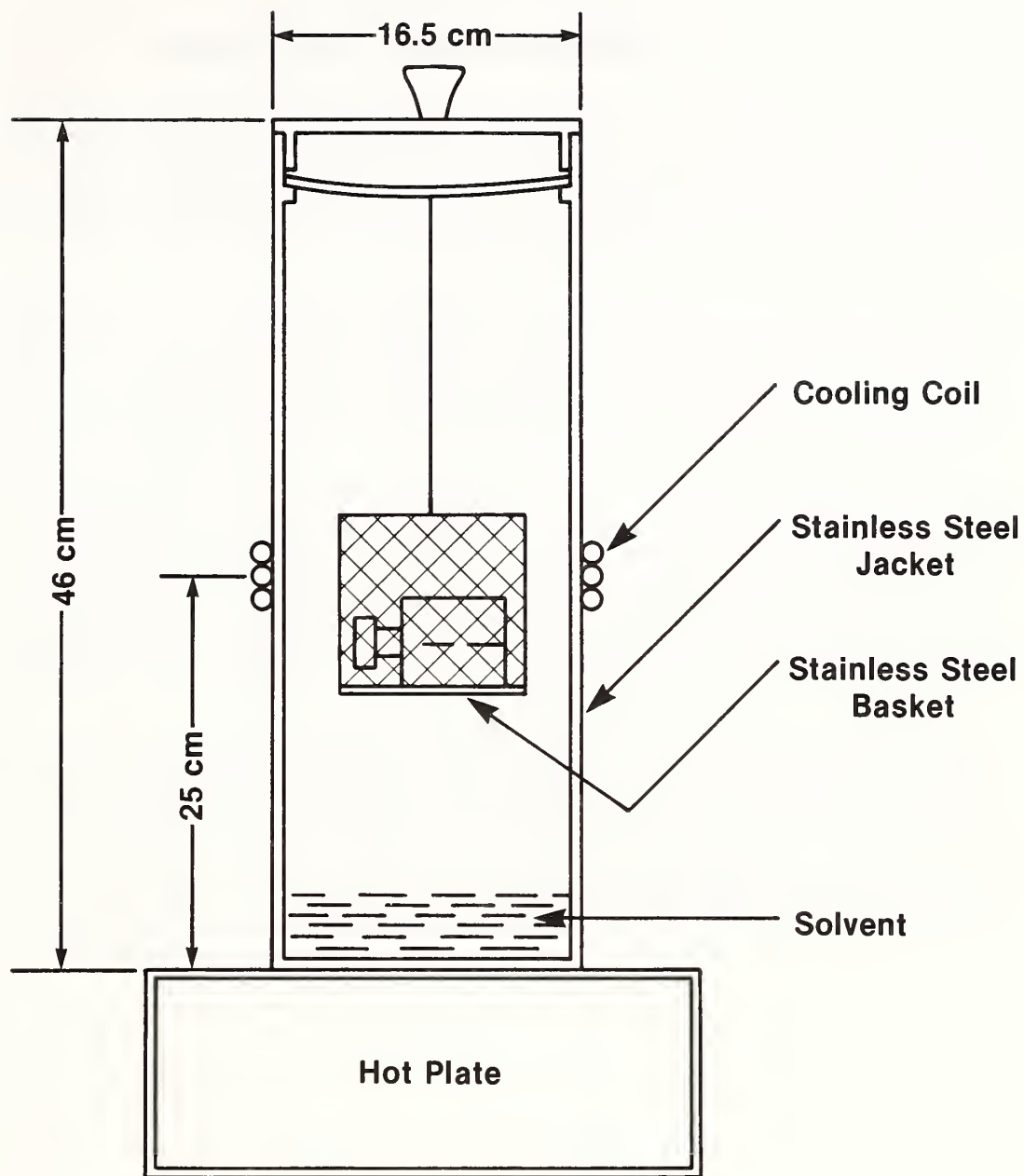


Figure 1. Vapor Degreaser



CLEANING METAL VOLUMETRIC MEASURES

All volumetric test measures, including laboratory standards and those to be calibrated or tolerance tested, must be clean at the time of measurement. Drainage is adversely affected by greasy and oily surfaces. Corroded surfaces raise questions which impair future use so that containers, so damaged, may not be worth testing.

Metal measuring vessels are best cleaned by using a non-foaming dishwashing detergent. Warm water is most effective both for cleaning and rinsing, which must be thorough. Ordinarily, three rinses will be sufficient, but this should be confirmed by testing the final rinse for any visual evidence of detergent. If warm water cleaning and rinsing is used, the vessel must be equilibrated to test temperatures before use.

The cleaning operation involves addition of a liberal amount of the detergent solution and vigorous shaking, swirling, or other motion so that the solution contacts the entire inner surface of the vessel. Depending on the condition of the surface, this operation should be repeated one or more times to insure removal of oily films and residues. As much as possible of the detergent should be drained prior to the water rinses to facilitate the latter operation.

Vessels should be drained completely after use and stored in a dry place. They should be covered to prevent entrance of dust and foreign matter. Metal containers further should be protected from damage by denting and/or droppage. Once such a vessel has been visibly dented, it is difficult to ascertain whether additional changes have occurred. Even when dents have been removed and the vessel has been recalibrated, any visual deformities can compromise future use. Accordingly, protection from damage while stored or when in use should be a major concern of the metrologist.



CLEANING PRECISION GLASSWARE

It has been found that some methods of cleaning glassware prior to calibration or use are ineffective or detrimental.

Laboratory detergents that contain phosphates can leave a deposit on the glass which will cause water to "bead" on the surface making it appear to be dirty, and making it difficult to "set" a meniscus on a line.

Once this phosphate deposit has occurred, it is very difficult to remove. It may be removed with hot (approx. 65 °C) sodium dichromate--sulfuric acid cleaning solution. However, use of this hot solution is hazardous and is not recommended unless proper safety equipment is available.

There are a number of suitable detergents (both liquid and powder), available from laboratory supply houses, which do not contain phosphates. The catalog descriptions usually indicate whether or not they contain phosphates. Examples are "Thomas Cleaning Compound" available from Arthur H. Thomas Co., Swedesboro, New Jersey, and "Liqui-Nox" available from several sources.

If acetone is used to remove oil or grease film, always follow with alcohol before drying or rinsing with water. Acetone, if allowed to dry, also leaves a film deposit.

The above solvents need not be used if the glass does not have an oil or grease film, nor if the cleaning is to be done with cold sodium dichromate-sulfuric acid solution. This solution at room temperature is suitable for the metrology laboratory and field glassware.

However, if laboratory glassware is to be calibrated for chemical laboratories, the metrologist should inquire if this cleaning method is acceptable because it can leave a deposit, which can be detected in some chemical analysis operations.

As an alternative, nitric acid, either concentrated or diluted, may be used. Some laboratories store some of their glassware in a weak solution of nitric acid to avoid contamination between uses.

It must be stressed that proper safety precautions be followed where appropriate including the use of eye shields, rubber gloves, and hoods, when using these cleaning procedures.

These cleaning agents do not preclude the use of other suitable methods, of which there are several. Some have special applications that would not normally be encountered in the weights and measures laboratory, or by field inspectors.



### Cleaning Methods\*

Frequently it is desirable to give glassware a preliminary rinse or soak with an organic solvent such as xylene or acetone to remove grease, followed by a water rinse. The rinsing with water must be done thoroughly if acid will be used later to clean the glassware.

Unless autoclaving is necessary, glassware should be cleaned as soon as possible after use to avoid setting and caking of residues. Pipets, for example, may be placed in a jar containing a weak antiseptic solution, immediately after use. Autoclaving is necessary to disinfect glassware that may have been used to contain potentially dangerous biological fluids.

A selection of general cleaning methods follows:

1. Fill with sulphuric acid-dichromate mixture and let stand. After removal of the mixture, rinse with distilled water at least six times. To make the cleaning mixture, dissolve 60-65 grams of sodium or potassium dichromate by heating in 30-35 mL of water, cool and slowly add concentrated sulphuric acid to make one liter of solution. This solution is available from laboratory supply companies.

Note: Extreme care should be exercised in handling acidic solutions.

2. Scrub with a 1% to 2% hot solution of a detergent. Rinse well after brushing. A number of suitable commercial washing compounds are available.
3. Fuming sulphuric acid (very hazardous material) is an excellent cleaning agent. Usually, cleaning can be accomplished by use of a comparatively small amount of acid, manipulating the vessel so that the acid comes in contact with all of the surface, and immediately emptying and rinsing.

When a piece is badly contaminated with stopcock grease (except silicone grease), it may be necessary to rinse with acetone once or twice before using one of the above methods. For silicone grease, the acetone can be omitted and the piece soaked for 30 minutes in fuming sulphuric acid. Warm decahydronaphthalene (decalin) also has been suggested as a solvent for silicone grease. In this case, let the piece soak for 2 hours, drain, and rinse once or twice with acetone.

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\*While the metrologist is not ordinarily faced with the problems for which these procedures are necessary, they are given here in the event that they may be needed.

### Cautions

Care should be exercised when using most cleaning solutions because they can cause skin irritations or severe burns on contact. Dilute solutions become concentrated as the water evaporates; therefore, always flush the exposed area immediately with large quantities of water.

Goggles should be worn to protect the eyes from splashes and rubber gloves to protect the hands. It is advisable to wear an acid resistant laboratory coat or a rubber apron to protect clothing when using strong acids for cleaning. The glassware should be handled gently to avoid breakage and also to prevent spilling acids and other cleaning fluids. All cleaning should be done in a laboratory sink or on an acid-proof laboratory bench, to the extent possible.

Some of the cleaning materials mentioned leave minute traces or residues unless the rinsing process is carried out thoroughly. While such traces may not be harmful if the object of cleaning is to prepare the glassware for calibration, they can give trouble when the glassware is used in certain laboratory operations. For example, manganese and chromium compounds, even in extreme dilution, may retard or inhibit growth of micro-organisms, and traces of phosphorus may interfere with delicate tests for this element. When glassware is to be calibrated, the final rinsings must be with distilled or deionized water.

If an article is to be dried after cleaning as is necessary for all vessels marked "To Contain", acetone, followed by ethylalcohol (American Chemical Society Specification) may be used. Drying may be hastened by blowing clean, dry air into the vessel (or sucking the air through the vessel).

Efficient air filters must be provided to remove any particles of oil or dirt from compressed air used for drying purposes.



RECOMMENDED FORM FOR REPORTING TAPE CALIBRATIONSEXAMPLE

For: 100-Foot Iron-Nickel Alloy Tape (or Steel)  
XYZ Co.

Test No. \_\_\_\_\_

Submitted by:

This tape has been compared with the standards of \_\_\_\_\_ which are traceable to NBS. The horizontal distances between the centers of graduations of the indicated intervals have the following lengths at 68 °F (20 °C) when the tape is subjected to horizontally applied tensions and supported on a horizontal flat surface:

<u>Tension</u> (pounds)	<u>Interval</u> (feet)	<u>Length</u> (feet)	<u>Uncertainty</u> (feet)
20 (or 10)	0 to 100		
30	0 to 100		

Note that reported lengths may be converted to catenary suspension support lengths by use of the "Equations for Metallic Tapes" (GMP No. 9).

The uncertainty of the reported length value is based upon the limits imposed by the standards used for the calibration of the tape, the length of the interval, the character of the tape graduations and the repeatability of the measurement process where three standard deviations is taken as the limit of random error.

Measurements were made to the centers of the graduations at the edge of the tape ribbon nearest the observer with the zero mark to the left of the observer.

The average AE value for this tape is \_\_\_\_\_ pounds, where AE is the product of the average cross-sectional area of the tape ribbon and its Young's Modulus of Elasticity.

The average weight per foot of this tape is 0.0 \_\_\_\_\_ pound.

The assumed coefficient of thermal expansion for Invar tapes is 0.00000022 per °F (0.0000004 per °C). For steel tapes, the assumed coefficient is 0.00000645 per °F (0.0000116 per °C).

The exact relationship between the International System of Units and the U. S. customary units of length is one foot equals 0.3048 meter.





EQUATIONS FOR METALLIC TAPES

The horizontal straight-line distance,  $L$ , of a tape interval can be computed by the following equation for an applied tension,  $P$ , and temperature,  $T$ , when the tape is supported for  $N$  number of equidistant catenary suspensions

$$L = L_s + \ln(P - P_s)/AE + \ln(T - T_s)\alpha - \ln(W \cdot L_n/N \cdot P)^2/24, \quad (1)$$

where  $L_s$  is the calibrated length of the tape interval on a flat surface at  $T_s$  and  $P_s$

$L_n$  is the designated nominal length of the tape interval

$P_s$  is the standard tension applied to the tape interval for  $L_s$

$T_s$  is the standard temperature of the tape interval for  $L_s$ , 68 °F (20 °C)

$AE$  is the average cross-sectional area times Young's Modulus of Elasticity

$W$  is the average weight per unit length of the tape ribbon

$\alpha$  is the coefficient of thermal expansion of the tape ribbon.

While the tape is supported entirely on a horizontal flat surface,  $N = \infty$ , the general equation is reduced to

$$L = L_s + \ln(P - P_s)/AE + \ln(T - T_s)\alpha, \quad (2)$$

and the distance,  $L$ , of the tape interval can be set to the designated nominal length,  $L_n$ , for determining the tension of accuracy\*,  $P_o$ , while the tape is supported on a flat surface, by writing equation (2) as follows:

$$L_n = L_s + \ln(P_o - P_s)/AE + \ln(T - T_s)\alpha,$$

from which

$$P_o = P_s + AE(L_n - L_s)/L_n - AE(T - T_s)\alpha \quad (3)$$

or 
$$P_s = P_o - AE(L_n - L_s)/L_n + AE(T - T_s)\alpha.$$

Substituting the last equation for  $P_s$  in the general equation (1), we have

$$L = L_n + \ln(P - P_o)/AE - \ln(W \cdot L_n/N \cdot P_c)^2/24,$$

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\*Tension of accuracy is defined as that tension which must be applied to the tape interval to produce its designated nominal length at the observed temperature of the tape.

The distance, L, of the tape interval again can be set to the designated nominal length, L<sub>n</sub>, for determining the tension of accuracy, P<sub>c</sub>, while the tape is supported in catenary suspensions, by writing this equation as follows:

$$L_n = L_n + L_n(P_c - P_o)/AE - L_n(W \cdot L_n/N \cdot P_c)^2/24,$$

$$\text{from which } P_c^2 (P_c - P_o) = AE(W \cdot L_n/N)^2/24 \quad (4)$$

$$\text{or } P_c^2 [P_c - P_s - AE(L_n - L_s)/L_n + AE(T - T_s)\alpha] = AE(W \cdot L_n/N)^2/24. \quad (5)$$

The value of P<sub>c</sub> can be solved by first determining the right side of the equals sign in equations (4) or (5) then substituting various values for P<sub>c</sub> until the left side approaches the right side within the desired limits. If the value is greater than the right side, reduce the value of P<sub>c</sub>.

WEIGHING OPERATIONS

Good laboratory techniques and practices, in addition to good standards and balances, are required to obtain good mass measurements. Errors can arise from a variety of sources, such as balance drift, air currents, lack of thermal equilibrium, magnetic or electrostatic fields, and to manipulative skill using the balance. The magnitude and significance of these errors will vary depending upon the laboratory environment, the balance, and the accuracy required for the measurement. Different methods of weighing are used to eliminate or reduce the effect of sources of error. Particular sources of error and ways to eliminate the errors are discussed with the weighing procedures. The accuracy required for a measurement and the criticality of the measurement often influence the choice of the weighing method used in the measurement. Regardless of the procedure used, there are several practices to be followed to promote good measurements.

Environment and Preparation

First, the balance should be installed in a laboratory having good temperature and humidity control. The requirements for temperature stability increase as more precision is needed in the measurement. For high precision measurement, the temperature should not vary by more than  $\pm 1$  °C in a 24-hour period throughout the year. General laboratory measurement can probably withstand temperature variations of  $\pm 2$  °C per day. Varying temperatures result in balance drift and unequal temperatures for the air, balance, and objects to be weighed. The relative humidity should be maintained between 40 and 60%. If the relative humidity falls significantly below 40%, static electricity may be generated both in and near the balance. This can result in erratic readings and make precision weighing impossible.

The test objects, balances, reference standards, and air should all be in thermal equilibrium. When possible, the objects to be weighed and the reference standards should be placed either in the balance chamber or adjacent to the balance so all can come to the same thermal equilibrium. If there is a lack of thermal equilibrium, convection currents will be generated when an object is on the balance pan and an erroneous reading may result. These types of errors are likely to go unnoticed so care must be taken to reduce the likelihood of their occurrence. Tests have shown that these types of errors depend upon the temperature differences among the objects and the balance, and on the geometry of the objects being weighed. On 20-g standards of greatly different geometries, studies have shown that errors as large as 0.2 mg can occur for a 4 °C temperature difference among the standards and the balance.

The balance must be installed in an area free from air currents. Balances should be installed away from heating/cooling vents. It is not adequate to merely close vents when weighing is being done because the resulting disruption of the temperature control system may result in temperature changes near the balances.

## Handling Weights

The masses of standard weights or objects weighed can be affected significantly by the manner in which they are handled. Human contact can leave grease or oily films that affect the mass at the time of measurement and even can cause permanent damage due to corrosion.

All small weights should never be touched but handled using forceps, clean gloves, or swatches of cloth. In the latter case, the cloth must be lint free. Instructions for cleaning weights and for removing adhering foreign material are described in GMP No. 5.

Large weights cause special problems. Fork lifts, portable cranes, hoists, and even over-head cranes may need to be used to move the weights within the laboratory and on or off the balances. Laboratory personnel need to become expert in using such equipment, as necessary, to avoid damage to the laboratory facilities, to the balances used, and even to the weights, themselves. The problem of temperature equilibrium for large weights is qualitatively the same as for small weights and needs consideration on an individual basis.

Large weights must be clean at the time of use but cleaning may be a problem. Minimally, they should be inspected to assure that foreign material is not present. Cleaning is addressed further in GMP No. 5.

## Operation

Analytical balances are high precision instruments and should be operated carefully. Avoid shock loading the balance. The dials on mechanical balances should be turned slowly and gently. Excessive shock can damage a balance. Careful balance operation will improve the repeatability of measurements.

Mechanical analytical balances are provided with partial and full release positions. The partial release position is used when determining if an unknown load will provide an on-scale reading. The balance beam has limited movement in this position. The partial release position provides some protection to the balance when the dialed-in weights are not close to the actual mass placed on the pan. It is a good practice to arrest the pan each time a dial is being changed to protect the balance from shock loading. It is acceptable to change the dial representing the smallest built-in weights when in the partial release position, because the small weight changes should not result in shock loading of the balance.

When releasing the pan to either the full or partial release position, the action should be done slowly and carefully. The objective is to minimize disturbances to the balance as much as possible.

Similarly, all loads should be placed on the balance pan carefully and centered on the pan.

All balances should be exercised before taking readings. A load equal to the load to be measured should be placed on the balance, a reading taken, and the pan arrested, if appropriate, or the weight removed from electronic balances. This operation should be repeated several times before readings are



taken for measurement purposes. Once the balance has been "warmed-up", better repeatability will be achieved. Balances can be very accurate even when used without being exercised first, but improved results can be obtained by going through a "warm-up" procedure. If the larger variation present in initial weighings on a balance that has not been exercised are not significant to the measurement, the warm-up procedure may be ignored.

To determine the repeatability of measurements when a balance has not been exercised versus its performance after being exercised, and to determine how much warm-up time is required, begin measurements starting with the unexercised balance and record the readings. Repeat a series of measurements until you have obtained several measurements after the balance has been exercised. This test can be repeated over several days using the same objects and the results compared. The readings obtained when using an unexercised balance are likely to show a slightly larger variation than those obtained after the balance has been exercised. Balance drift is likely to be larger initially and then reach a steady state when the balance has been "warmed-up".

#### Comparison Weighing - Mechanical Balance

Most routine laboratory measurements can be performed to sufficient accuracy by using the direct reading of mass values. When higher precision is required, a comparison of the unknown object to a known mass standard must be made. A comparison weighing eliminates the errors of the built-in weights, reduces disturbances during the measurement because dial settings are not changed during the measurement, and can cancel the effect of drift by selecting the appropriate weighing design.

The purpose of comparison weighing is to compare the unknown, X, to a standard, S, thereby eliminating the built-in weights from the measurement. In comparison weighing the built-in weights act only as counterweights; they do not affect the difference measured between X and S. Consequently, the dial settings must not be changed during a comparison measurement; otherwise the built-in weights would be part of the measurement.

When comparison measurements are made on a single pan mechanical balance, all readings are taken from the optical scale. The unknown and the standard must have nearly the same mass so that the difference between them can be measured on the optical scale. If the mass of the unknown and the standard are significantly different, small mass standards are used as trim weights with either the unknown or the reference standard or both to obtain an observed mass difference that is significantly less than one-fourth the range of the optical scale.

As part of a comparison measurement, the mass value of a scale division is determined by performing a sensitivity determination. The small mass standard used as part of the weighing design to calibrate the optical scale is called the sensitivity weight. The sensitivity weight should have a mass between  $1/5$  and  $1/2$  the range of the optical scale, inclusive. Additionally, the sensitivity weight should have a mass that is at least twice the observed mass difference between the standard and the unknown. Since the maximum size of the sensitivity is limited to one-half the range of the optical scale, it may be necessary to carry tare weights with the standard and the unknown to assure that the observed difference between them is less than one-half the mass of the



sensitivity weight. For high accuracy measurements, the value of the sensitivity weight should include its correction from its nominal value and the air buoyancy effect on the sensitivity weight.

The first readings for the standard and the unknown in a comparison on a single pan balance should fall in the first quarter of the optical scale but well ahead of zero so the balance drift will not result in negative values for any readings. Although negative numbers may be used in calculations, they are avoided to simplify calculations and reduce calculation errors. Because the sensitivity weight may have a mass as large as one-half the range of the optical scale and the measured difference between the standard and the unknown may be as large as one-fourth the range of the optical scale, it is necessary to obtain the first two readings in the first quarter of the optical scale so all readings will remain on-scale for the measurement. In this way it is not necessary to change the dial settings to measure the difference between the standard and the unknown.

#### Comparison Weighing - Electronic Balance

Measurements made on a full electronic balance are simplified because there are no built-in weights to consider. Although many electronic balances are equipped with a built-in calibration weight, the weight is not involved in the comparison weighing.

The principles for comparison weighing on a full electronic balance are the same as when using a single pan mechanical balance. The balance indications are used to measure the mass difference between the standard and the unknown and a sensitivity weight is used to establish the mass value for a digital division on the balance. Since there are no built-in weights in the full electronic balance, the entire range of the digital indications can be considered for "optical scale" of the balance.

For comparison weighing the standard and the unknown should be "nearly the same mass." Since a full electronic balance has a much larger range for indicating mass values, the masses do not have to be as close together as when a mechanical balance is being used. When using an electronic balance, the difference in mass between the standard and unknown should be less than 0.05% of the balance capacity. Tare weights that are known standards should be used if the masses are not sufficiently close together. The sensitivity weight used to determine the mass per digital division should have a mass that is at least 10 to 20 times the mass difference between the standard and the unknown but not exceeding 1% of the balance capacity. For high precision weighing air buoyancy corrections must be made for all objects used in the intercomparison.

#### Magnetic and Electrostatic Fields

Care must be taken when weighing magnets or objects having electrostatic charges. A magnet has a magnetic field that will likely affect results of a measurement. The magnet is attracted to ferromagnetic materials in the balance and balance chamber. The magnetic field may also affect the magnetic field generated in an electronic balance that utilizes the principle of magnetic force restoration as its method of measurement.

Weights made of ferromagnetic material can become magnetized during manufacture or during use if they are placed in a strong magnetic field. Magnetized weights can result in measurement errors that may go undetected. If a measurement problem is suspected, the weights should be checked for magnetism and should be demagnetized if they are found to be magnetized.

If magnets or magnetized material must be weighed, the material should be placed in a magnetically shielded container to prevent the magnetic field from generating measurement errors. If balance design and conditions permit, another alternative is to position the magnetized material a relatively large distance from the balance pan using a non-ferromagnetic object on the pan to serve as a platform for the magnetic. Since the strength of the magnetic field drops off at a rate of the cube of the distance from the magnetic, it may be possible to effectively isolate the magnet from other ferromagnetic material in the balance.

Electrostatic fields can also cause errors in measurements. If a static charge exists in a mechanical balance, the balance may give erratic readings and lack repeatability. If the object being weighed contains a significant electrostatic charge, it may result in measurement errors and may leave a static charge on the balance. Electrostatic charges are of a particular concern when plastic containers are placed on the balance.

Care should be taken to remove electrostatic charges from objects being weighed by grounding the objects, if necessary, before placing them on the balance. To prevent the build-up of static electricity in a balance, the relative humidity in the laboratory should be maintained between 40 and 60 percent. The water vapor in the air will serve to drain off electrostatic charges from the balance.

Balances utilizing the magnetic force restoration principle for weighing should be checked to verify that the magnetic field generated by the magnetic cell in the balance does not exist around the balance pan. If the shielding of the magnetic cell is insufficient, measurement errors may occur when weighing ferromagnetic objects or when the balance is placed on a surface comprised of ferromagnetic material.



## 6. STANDARD OPERATIONS PROCEDURES

- SOP No. 1 Recommended Standard Operations Procedure for Preparation of Calibration/Test Reports
- SOP No. 2 Applying Air Buoyancy Correction
- SOP No. 3 Recommended Standard Operations Procedure for Weighing by Double Substitution Using an Equal-Arm Balance
- SOP No. 4 Recommended Standard Operations Procedure for Weighing by Double Substitution Using a Single Pan Mechanical Balance, Full Electronic Balance or a Balance with Digital Indications and Built-in Weights
- SOP No. 5 Recommended Standard Operations Procedure for Using a 3-1 Weighing Design (Double Substitution)
- SOP No. 6 Recommended Standard Operations Procedure for Weighing by Transposition
- SOP No. 7 Recommended Standard Operations Procedure for Weighing by Single Substitution Using a Single Pan Mechanical Balance, a Full Electronic Balance, or a Balance with Digital Indications and Built-in Weights
- SOP No. 8 Recommended Standard Operations Procedure for Tolerance Testing of Mass Standards by Modified Substitution
- SOP No. 9 Recommended Standard Operations Procedure for Control Charts for Calibration of Mass Standards
- SOP No. 10 Recommended Standard Operations Procedure for Calibration of Rigid Rules
- SOP No. 11 Recommended Standard Operations Procedure for Calibration of Metal Tapes, Bench Method
- SOP No. 12 Recommended Standard Operations Procedure for Calibration of Steel Tapes, Tape-to-Tape Method
- SOP No. 13 Recommended Standard Operations Procedure for Calibration of Volumetric Ware, Gravimetric Method
- SOP No. 14 Recommended Standard Operations Procedure for Gravimetric Calibration of Volumetric Ware Using and Electronic Balance
- SOP No. 15 Recommended Standard Operations Procedure for Calibration of Intermediate and Large Volume Standards, Gravimetric Method
- SOP No. 16 Recommended Standard Operations Procedure for Calibration of Measuring Flask, Volume Transfer Method

- SOP No. 17 Recommended Standard Operations Procedure for Control Charts for Calibration of Measuring Flasks, Volume Transfer Method
- SOP No. 18 Recommended Standard Operations Procedure for Calibration of Graduated Neck Volumetric Field Standards, Volumetric Transfer Method
- SOP No. 19 Recommended Standard Operations Procedure for Calibration of Large Metal Provers, Volumetric Method
- SOP No. 20 Recommended Standard Operations Procedure for Control Charts for Calibration of Neck-Type Provers, Volumetric Method
- SOP No. 21 Recommended Standard Operations Procedure for Calibration of LPG Provers



SOP No. 1

Recommended Standard Operations Procedure  
for  
Preparation of Calibration/Test Reports

1. Introduction

- 1.1. Test reports are the visible outputs of the testing laboratory. They should be prepared with utmost care to insure that they accurately convey all information pertaining to the testing so that reports may be used with maximum benefit by all concerned. Carefully prepared test reports will contain or refer to all information necessary to justify the test results.
- 1.2. The test report may consist of filling in the blanks in a form in the case of a routine measurement. A more detailed report, including narrative information, may be required for special calibrations or tests.
- 1.3. Regardless of the final form, the test report should contain the basic information described in the following sections.

2. Content

2.1. Client

Identify the individual/organization requesting the calibration/test.

2.2. Purpose of test

Describe clearly what is required to be done.

2.3. Description of test/calibration items

Include information that will positively identify the item(s), including manufacturer's name, model number, serial numbers, etc. It is recommended that a laboratory number be assigned and attached to each test item at the time of its acceptance for testing. The use of the laboratory number will facilitate the internal control of test items during the testing process.

2.4. Test Method

Describe how the test(s) was/were made by reference to SOP(s). In the absence of SOPs, brief but informative descriptions of the methodology should be included.

2.5. Test Results

Present test results in tabular or other convenient form.

## 2.6. Limits of uncertainty

State the limits of uncertainty for the test results and the basis for their assignment.

## 2.7. Traceability/In-tolerance

Identify the reference standards used and their traceability to national standards.

State that instrumentation used was/was not in tolerance at time of the calibration measurements. See Appendix B for a suitable format for this purpose.

## 2.8. References (optional but recommended).

Include references to determine where original data may be located in case of future need. Also, include name of person(s) who made the test measurements. These may be on a blind copy.

## 2.9. Conclusions

When test is of more than routine significance, present the conclusions that may be drawn from the test results, and especially any limitations on the results, as necessary or desirable.

## 2.10. Signatures

Laboratory director or his/her designated alternate must approve and sign all test reports. Other signatures may be required, at the discretion of the laboratory director. Each signer accepts his/her share of responsibility for the contents of the report.

# 3. Recording

3.1. Give each test report a characteristic identification number according to a system developed by the laboratory director.

3.2. File all test reports in a systematic manner for ease of retrieval, as necessary.

3.3. Retain copies of all test reports for a minimum period of five years, until superseded by a subsequent report, or until deemed by the laboratory director as having no future value.

APPENDIX A.

Recommended Format for Routine Test Report

Test Report  
Issued by  
Name of Testing Laboratory  
Laboratory Report No. \_\_\_\_\_

Test Item(s)/Lab No(s): \_\_\_\_\_  
\_\_\_\_\_

Submitted by: \_\_\_\_\_

Date: \_\_\_\_\_

Purpose of Test: \_\_\_\_\_  
\_\_\_\_\_

Test Results\*:

As Found

After Adjustment

Reference Information:

Test Method: \_\_\_\_\_

Traceability: \_\_\_\_\_

Data Reference: \_\_\_\_\_

Test Results Certified by (name, title, date): \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

\*Report, as appropriate

APPENDIX B.

Recommended Format for Certificate of Traceability

A B C COMPANY

123 Utopia Street  
Anywhere, USA

CERTIFICATE OF TRACEABILITY

Name of Device:

Model:

Serial No:

Submitted by:

The calibration was performed on (date)  
The ambient conditions were °C and % relative humidity.  
The item tested was/was not in tolerance at time of calibration. Any out of tolerance data are attached.

Data:

The primary standards to which the above data are traceable are identified in this report. The calibration of these standards is traceable to the National Bureau of Standards. The cycling and certification of all standards of measurement at this facility meet the requirements of MIL-STD-45662.

<u>Name of</u> <u>Standard</u>	<u>NBS</u> <u>Report</u>	<u>Date</u> <u>Calibrated</u>	<u>Date</u> <u>Due</u>
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Test Results Certified by (name, title, date): \_\_\_\_\_

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Recommended Standard Operations Procedure  
for  
Applying Air Buoyancy Corrections

1. Introduction

1.1. Purpose

Frequently, the largest source of error in mass measurement, if uncorrected, is the effect of air buoyancy on the objects being compared. This SOP provides the equations to be used to correct for the buoyant effect of air. The significance of the air buoyancy correction depends upon the accuracy required for the measurement, the magnitude of the air buoyancy correction relative to other sources of error in the overall measurement process, and the precision of the mass measurement. An air buoyancy correction should be made in all high accuracy mass determinations. The Appendix to this SOP provides a brief theoretical discussion of this subject.

1.2. Prerequisites

1.2.1. The (true) mass values or corrections must be available for the standards used in the measurement process. The mass standards must be traceable to NBS.

1.2.2. The thermometer, barometer, and hygrometer used must be calibrated and in good operating condition as verified by periodic tests or cross-checks to other standards.

2. Methodology

2.1. Scope, Precision, Accuracy

This procedure is applicable to all weighings utilizing a comparison of mass standards. The precision will depend upon the accuracy of the thermometer, barometer, and hygrometer used to determine the air density. When the calculations for the air density and air buoyancy corrections are made, a sufficient number of decimal places must be carried so the error due to the rounding-off of numbers is negligible relative to the error in the measurement process. Typically, carrying six to eight decimal places is sufficient, but this will depend upon the precision of the measurement.



## 2.2. Apparatus/Equipment Required\*

- 2.2.1. \*Thermometer accurate to 0.10 °C to determine air temperature.
- 2.2.2. \*Barometer accurate to 0.5 mm of mercury (66.5 Pa) to determine air pressure.
- 2.2.3. \*Hygrometer accurate to 10 percent to determine relative humidity.

## 2.3. Estimating the Magnitude of the Air Buoyancy Correction

The magnitude of the air buoyancy correction, MABC, of a measurement can be estimated using the following formula:

$$MABC = (\rho_A - \rho_n)(V_x - V_s)$$

where  $\rho_A$  = air density at the time of the measurement  
 $\rho_n$  = density of "normal" air; i.e., 1.2 mg/cm<sup>3</sup>  
 $V_x$  = volume of the unknown standard X  
 $V_s$  = volume of the reference standard S

This information can be used to decide on the importance of the air buoyancy correction for a particular measurement.

## 2.4. Procedure

- 2.4.1. Record the temperature, pressure, and relative humidity at the start and at the end of the measurement process. If these parameters change significantly during the measurement process, it may be necessary to use average values to compute the air density.
- 2.4.2. Determine the air density using Table 9.9 or the equation given in Section 8 of the Appendix to this SOP.

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\*The thermometer, barometer, and hygrometer are used to determine the air density at the time of measurement. The air density is used to make the buoyancy correction. The accuracies specified are recommended for high precision calibration. Less accurate equipment can be used with only a small degradation in the overall accuracy of the measurement.

### 3. Calculations

- 3.1. Calculate the mass,  $M_x$ , of the unknown standard, X, using the following equation.

$$M_x = \frac{M_s \left( 1 - \frac{\rho_A}{\rho_s} \right) + d}{\left( 1 - \frac{\rho_A}{\rho_x} \right)}$$

If tare weights were carried with X and S, use the following equation.

$$M_x = \frac{M_s \left( 1 - \frac{\rho_A}{\rho_s} \right) + d + M_{t_s} \left( 1 - \frac{\rho_A}{\rho_{t_s}} \right) - M_{t_x} \left( 1 - \frac{\rho_A}{\rho_{t_x}} \right)}{\left( 1 - \frac{\rho_A}{\rho_x} \right)}$$

The symbols not previously defined are as follows:

$d$  = measured difference between X and the reference standard, S, using one of the weighing designs given in other SOPs

$M_s$  = true mass of the reference standard, S

$\rho_s$  = density of the reference standard, S

$\rho_x$  = density of the unknown standard, X

$M_{t_s}$  = true mass of the tare weights carried with S

$\rho_{t_s}$  = density of the tare weights carried with S

$M_{t_x}$  = true mass of the tare weights carried with X

$\rho_{t_x}$  = density of the tare weights carried with X

- 3.2. If reporting the apparent mass,  $AM_x$ , versus  $8.0 \text{ g/cm}^3$ , compute it using the following.

$$AM_x \text{ vs } 8.0 = M_x \frac{1 - \frac{0.0012 \text{ g/cm}^3}{\rho_x}}{0.999850}$$

- 3.3. If reporting the apparent mass,  $AM_x$ , versus brass, compute it using the following.

$$AM_x \text{ vs brass} = M_x \frac{1 - \frac{0.0012 \text{ g/cm}^3}{\rho_x}}{0.999857}$$

- 3.4. The apparent mass values are related by the following:

$$AM_x \text{ vs 8.0} = 1.000007 (AM_x \text{ vs brass})$$

$$AM_x \text{ vs brass} = 0.999993 (AM_x \text{ vs 8.0})$$

#### 4. Assignment of Uncertainty

The uncertainty in determining the air buoyancy correction is usually negligible relative to the precision of the measurement process itself. Consequently, the uncertainty for the measurement is based upon the uncertainty for the measurement process used.

#### Tolerances for Measurements Related to Air Density Estimation

<u>Variable</u>	Uncertainty of Air Density Values	
	$\pm 0.1\%$	$\pm 1.0\%$
Relative humidity	$\pm 11.3\%$	.....
Air temperature	$\pm 0.29 \text{ }^\circ\text{C}$	$\pm 2.9 \text{ }^\circ\text{C}$
Air pressure	$\pm 101 \text{ Pa}$	$\pm 1010 \text{ Pa}$
Air pressure	$\pm 0.76 \text{ mm Hg}$	$\pm 7.6 \text{ mm Hg}$

## Appendix to SOP No. 2

### The Basic Theory of Air Buoyancy Corrections

Richard S. Davis\*

#### 1. Introduction

In performing measurements of mass, the balance or scale used acts as a force transducer. The force produced by an object to be weighed in air has two components: one proportional to the mass of the object, and the other proportional to its volume. The latter component, or buoyant force, may under some circumstances be large enough to require correction. The following shows under what circumstances buoyancy corrections are required as well as how they are made.

#### 2. Scope

The method for applying buoyancy corrections presented below applies to mass measurements made in air. The density of air is computed from auxiliary measurements of temperature, pressure and relative humidity after which the buoyancy corrections are calculated directly from the Principle of Archimedes. The following weighing situations are considered.

##### 2.1. Two-Pan Balance

##### 2.2. Single-Pan Balance

##### 2.2.1. With Built-In Weights

##### 2.2.2. With Electronic Control

#### 3. Summary of Method

In general, buoyancy corrections are applied to mass measurements by calculating the difference in volume between the unknown weight and the standard, multiplying this volume difference by the density of air at the balance or scale, and adding the product to the mass of the standard. The density of air is computed from an equation of state for air together with measured values for the temperature, pressure and relative humidity of the air, or it may be obtained with lesser accuracy, from Table 9.9.

#### 4. Significance and Use

Buoyancy corrections generally must be applied when determining the mass of an unknown object to high accuracy. The corrections may become important even at modest accuracies if the unknown object whose mass is to be determined has a density that differs widely from that of the standards (weighing of water, for example). Many mass standards are calibrated in

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\*NBS Center for Basic Standards

terms of a so-called "apparent mass" scale. (See Chapter 7.3.) Use of this scale does not, as is sometimes assumed, indiscriminately eliminate the need for buoyancy corrections.

## 5. Terminology

### 5.1. Weighing by Substitution

The procedure by which one determines the assembly of standard weights that will produce nearly the same reading on a one-pan balance as does the unknown object. The balance thus serves as a comparator. A two-pan balance may be used in this mode if one of the pans contains a counterpoise and the standards and unknown are substituted on the second pan. (See SOP No. 3.)

### 5.2. Mass

The term "mass" is always used in the strict Newtonian sense as a property intrinsic to matter. Mass is the proportionality constant between a force on a material object and its resulting acceleration. This property is sometimes referred to as "true mass", "vacuum mass", or "mass in vacuum" to distinguish it from apparent mass.

### 5.3. Apparent Mass<sup>1</sup>

The mass of material of a specified density that would exactly balance the unknown object if the weighing were carried out at a temperature of 20 °C in air of density  $1.2 \times 10^{-3}$  g/cm<sup>3</sup>. The mass,  $M_N$ , of an object, N, is related to its apparent mass  $M_{N,A}$  by the equation

$$M_{N,A} = \frac{M_N \left( 1 - \frac{1.2 \times 10^{-3}}{\rho_N} \right)}{1 - \frac{1.2 \times 10^{-3}}{\rho_B}}$$

where

$\rho_N$  = density of the object N at 20 °C in g/cm<sup>3</sup>

$\rho_B$  = density of the apparent mass scale at 20°C in g/cm<sup>3</sup>

There are at present two apparent mass scales in wide use. The older is based on  $\rho_B = 8.4000$  with a coefficient of volumetric expansion of 0.000054/°C and the more recent specifies  $\rho_B = 8.0000$  at 20 °C. The quantity  $M_{N,A}$  is thus a function of the particular apparent mass scale which has been used in its calculation.

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<sup>1</sup>Pontius, P.E. Mass and Mass Values, NBS Monograph 133, 1974, pp 12-23, 26-33.



#### 5.4. Sensitivity

The response of a balance under load to an additional small weight:

$$S = \frac{m_s - \rho_A v_s}{\Delta R}$$

where

$S$  = balance sensitivity

$m_s$  = mass of the small, additional weight

$\rho_A$  = density of air at the balance

$v_s$  = volume of the small, additional weight

$\Delta R$  = change in balance reading due to the addition of the small weight

#### 6. Apparatus

In order to ascertain the density of air at the balance, the following apparatus is necessary: thermometer, barometer, and hygrometer. Ideally, these should be placed in or next to the balance case. It may only be practical, however, for the thermometer to be so placed. In addition, a desk or hand-held calculator will be extremely useful.

#### 7. Procedure

Weigh the unknown object as directed by the balance manufacturer or in accordance with accepted procedure. Record the temperature, pressure and relative humidity of the air in the balance at the time of weighing. Do not correct the barometric pressure to sea level.

#### 8. Calculation

The density of air can be calculated from the following formula:<sup>2</sup>

$$\rho_A = \frac{0.46460(P - 0.0037960 U_{e_s})}{(273.15 + t)} \times 10^{-3} \quad (1)$$

where

$\rho_A$  = density of air, g/cm<sup>3</sup>

$P$  = barometric pressure, mm Hg

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<sup>2</sup>Jones, F.E., "The Air Density Equation and the Transfer of the Mass Unit," Journal of Research, National Bureau of Standards, Vol. 83, 1978, p. 419.

U = relative humidity, percent

t = temperature, °C

$$e_s = 1.3146 \times 10^9 \exp (-5315.56/(t + 273.15))$$

A table of  $e_s$  values is provided in Table 2 of this appendix.

Small errors (of order 0.01 percent) in this equation occur for locations well above sea level or under conditions in which the concentration of carbon dioxide differs greatly from the global average. The reader is directed to the reference in footnote 2 for a more general formulation of the equation. Alternatively, the density of air may be obtained from Table 9.9 if and accuracy of  $\pm 0.5\%$  for air density is sufficient.

Calculate the density of air at the balance during the weighing. Then determine the mass of the unknown,  $M_x$ , as follows:

8.1. If a two-pan balance is used:

$$M_x = M_s + \rho_A(V_x - V_s) + S\Delta M_{opt} \quad (2a)$$

or

$$M_x = \frac{M_s \left( 1 - \frac{\rho_A}{\rho_s} \right) + S\Delta M_{opt}}{1 - \frac{\rho_A}{\rho_x}} \quad (2b)$$

or

$$M_x = \frac{M_s - \rho_A V_s + S\Delta M_{opt}}{1 - \frac{\rho_A}{\rho_x}} \quad (2c)$$

where

$M_x$  = mass of the unknown object

$M_s$  = mass of the standard weights

$\rho_s$  = density of the standard weights =  $\frac{M_s}{V_s}$

$\Delta M_{opt}$  = off-balance indication read on the optical scale

$V_x$  = volume of the unknown object

$V_s$  = volume of the standard weights

$\rho_x$  = Density of the unknown object =  $\frac{M_x}{V_x}$

Volumes and densities are, in general, a function of temperature. The thermal coefficients of volumetric expansion of the unknown object and the standard may be significant in very accurate work<sup>3</sup>. The coefficient of volumetric expansion is usually estimated as three times the linear coefficient of expansion of the weight material.

The error in  $M_x$  incurred by ignoring the buoyancy correction is  $\rho_A(V_x - V_s)$ . To estimate quickly whether such an error is of consequence in a particular measurement, (assume  $\rho_A = 1.2 \times 10^{-3}$ ).

If the mass and volumes of the standards have been adjusted to an apparent mass scale, then

$$M_x \approx \frac{AM_s \left( 1 - \frac{\rho_A}{\rho_B} \right) + S\Delta M_{opt}}{1 - \frac{\rho_A}{\rho_x}} \quad (3)$$

where

$AM_s$  = apparent mass of the standard.

The symbol  $\approx$  signifies that the relation is approximate.

- 8.2. If a single-pan balance with built-in weights is used, it is probable that the built-in weights have been adjusted on an apparent mass scale<sup>4</sup>. Determine which apparent mass scale has been used and calculate the mass of the unknown from the equation

$$M_x \approx \frac{M_D \left( 1 - \frac{\rho_A}{\rho_B} \right) + SM_{opt}}{1 - \frac{\rho_A}{\rho_x}} \quad (4)$$

$M_D$  = mass indicated by dial readings

$M_{opt}$  = mass indicated on the optical scale.

---

<sup>3</sup>In general,  $V(t) = V_{20} [1 + \beta(t - 20^\circ \text{C})]$  where  $t$  is the temperature of the weight,  $V_{20}$  is the volume at  $20^\circ \text{C}$ , and  $\beta$  is the coefficient of volumetric expansion.

<sup>4</sup>Schoonover, R. M. and Jones, F. E., "Air Buoyancy in High-Accuracy Weighing on Analytical Balances," *Anal. Chem.*, 53, 1981, p. 900.

Equation 4 assumes the optical scale sensitivity,  $S$ , has been determined with a known sensitivity weight as part of the weighing procedure. This is good practice. If a sensitivity weight has not been used, however, set  $S = 1$  and include  $M_{\text{opt}}$  with  $M_D$ .

If the balance has been used only as a comparator, that is, to compare the mass of the unknown object with that of some external standard, then

$$M_x = M_s + \rho_A(V_x - V_s) + S\Delta M'_{\text{opt}}$$

where  $\Delta M'_{\text{opt}}$  = difference in optical scale reading between observations of the standard and the unknown.

For some balances, operation requires that the user restore the balance to null by means of a manually controlled dial. The portion of the mass reading controlled by this dial should be treated, for purposes of buoyancy corrections, as an optical scale.

- 8.3. If a single-pan balance with full-range electronic control is used, the following should be noted. As part of its calibration the electronic gain has been adjusted by means of a weight of known mass. For example, if the range of electronic control is 100 g, the electronics have been adjusted so that a 100-g standard weight produces a readout of precisely 100 g. This procedure effectively builds an apparent mass calibration into the balance. The reference density of the apparent mass scale is the density of the standard mass used for the calibration and the reference air density is the air density at the time of calibration.

The mass of an unknown object weighed on the balance is then

$$M_x = \frac{M_R \left( 1 - \frac{\rho_c}{\rho_c} \right)}{1 - \frac{\rho_A}{\rho_x}} \quad (5)$$

where

$M_R$  = readout displayed on the balance

$\rho_c$  = density of air at the time of balance calibration

$\rho_c$  = density of the standard used to calibrate the balance (or  $\rho_B$  if the apparent mass of the standard was used instead of the true mass).

If the balance includes both an electronic control system and built-in weights, the buoyancy considerations for the built-in weights are as described in section 8.2 and the considerations for the electronically determined mass are those given directly above.

8.4. Top-loading balances may be considered a form of single-pan balance and the appropriate procedure for buoyancy corection followed.

## 9. Precision

The contribution of the random error of evaluation of  $\rho_A$  to the precision of mass measurement may be estimated as follows:

For mechanical balances, or electronic balances used in weighing by substitution, the contribution is:

$$\delta\rho_A (V_x - V_s)$$

where

$$\begin{aligned} \delta\rho_A &= \text{random error of evaluation of } \rho_A \\ V_s &= \text{volume of standards, if weighing by substitution} \\ V_s &= \frac{M_D}{D_B}, \text{ if using the built-in weights on a single-pan} \\ &\quad \text{balance.} \\ V_x &= \text{volume of object weighed} \end{aligned}$$

For electronic balances used in the direct-reading mode, the contribution is:

$$\delta\rho_A V_x$$

The quantity  $\delta\rho_A$  will have contributions from the measurements of temperature, pressure and relative humidity which are required for the calculation of  $\rho_A$ . Equation (1) may be used to estimate the effects of imprecision in measurements of P, t, and U. It is unrealistic, however, to expect  $\delta\rho_A/\rho_A$  ever to be less than 0.05 percent even using the best techniques available.

## 10. Accuracy

Inattention to problems of buoyancy most often results in systematic errors. For a substitution weighing, for example, the buoyancy correction is of order  $\rho_A(V_x - V_s)$ . While this quantity may be significant to a measurement, day to day variation in  $\rho_A$  (usually no more than 3 percent) may not be noticeable and hence need not be measured. For the most accurate work, on the other hand, not only must  $\rho_A$  be accurately determined but volumes of the unknown and standard may need to be measured to better than 0.05 percent -- the minimum systematic uncertainty attainable in the calculation of  $\rho_A$ .

If the standards have been calibrated in terms of apparent mass, complete neglect of buoyancy corrections will produce an error in the measured result  $M_x$  of order

$$AM_s (1.2 \times 10^{-3} - \rho_A) \left( \frac{1}{\rho_s} - \frac{1}{\rho_B} \right) + \left( \frac{\rho_B - \rho_x}{\rho_B} \right) \left( \frac{\rho_A}{\rho_x} \right) \quad (7)$$

This error is often unacceptably large.



Use of equation (3), on the other hand, introduces only an error of approximately

$$AM_s (1.2 \times 10^{-3} - \rho_A) \left( \frac{1}{\rho_s} - \frac{1}{\rho_B} \right) \quad (8)$$

It is a requirement for manufacture that the actual density of standard weights be near enough to the assumed density of the apparent mass scale to which they are adjusted that the magnitude of (8) will always be small under normal conditions in laboratories near sea level.

The fact that there are two apparent mass scales widely used --one based on density  $8.0 \text{ g cm}^{-3}$  and an older one based on  $8.4 \text{ g cm}^{-3}$  --means that some caution is required on the part of the user. For the most accurate work, the apparent mass scale should be abandoned in favor of substitution weighing with standards of known mass and volume.

The user must decide the accuracy required of the particular mass measurement and choose a buoyancy correction technique commensurate with that accuracy.

The same considerations which apply to the accuracy of buoyancy corrections in weighing by substitution are easily extended to the other types of weighing indicated above.

There are many factors which effect the accuracy of a mass measurement. The above has dealt only with those arising from problems of buoyancy.

## EXAMPLES

### EXAMPLE 1

The weight set of Table 1 is used with an equal-arm balance to find the mass of a piece of single-crystal silicon. The following weights were used to balance the silicon: 10 g, 3 g. The balance pointer showed the silicon side to be light by 3.5 divisions. The 10 mg weight of Table 1 was used to find the sensitivity of the balance. When the weight was added, the pointer moved 10.3 divisions. At the time of the weighing, the following measurements were taken:

$$P = 748.1 \text{ mm Hg}$$

$$t = 22.3 \text{ }^{\circ}\text{C}$$

$$U = 37\%$$

What is the mass of the silicon?

ANSWER:

From Eq. 1, or Table 2, calculate

$$\begin{aligned}e_S &= 1.3146 \times 10^9 \exp (-5315.56 / (22.3 + 273.15)) \\&= 20.2 \text{ mm Hg}\end{aligned}$$

$$\rho_A = \frac{0.46460(748.1 - 0.0037960 \times 37 \times 20.2)}{273.15 + 22.3} \times 10^{-3}$$

$$\rho_A = 1.172 \times 10^{-3} \text{ g/cm}^3$$

The density of silicon at 20 °C is 2.3291 g/cm<sup>3</sup> and its coefficient of linear expansion is 0.0000026/°C.

Make use of Eq. 2c and Table 1

$$\begin{aligned}M_S &= 10.0001264 + 3.0000459 \\&= 13.000172 \text{ g}\end{aligned}$$

$$\begin{aligned}V_S &= (1.267 + 0.380) \\&= 1.647 \text{ cm}^3\end{aligned}$$

Calculate the sensitivity:

$$S = \frac{0.010003 \text{ g} - 0.00370 \times 1.172 \times 10^{-3}}{10.3}$$

$$= \frac{0.009999}{10.3} = 0.971 \text{ mg/Division}$$

$$\begin{aligned}M_X &= \frac{13.000172 - 1.647 \times 1.172 \times 10^{-3} + 0.971 \times 10^{-3}(-3.5)}{1 - \frac{1.172 \times 10^{-3}}{2.3291}} \\&= 13.001385 \text{ g}\end{aligned}$$

Note that the thermal expansion is insignificant in this example.

## EXAMPLE 2

Let us again consider the weighing performed in Example 1. This time, all we know about our weight set is that it has been adjusted to the 8.4 apparent mass scale at the best available commercial tolerance.

Using Eq. (3),

$$M_X = \frac{13.00 \left[ 1 - \frac{1.172 \times 10^{-3}}{8.3909} \right] + 0.010 \left[ 1 - \frac{1.172 \times 10^{-3}}{8.3909} \right] \left[ \frac{-3.5}{10.3} \right]}{1 - \frac{1.172 \times 10^{-3}}{2.3291}}$$

$$= 13.001329 \text{ g}$$

For routine weighing, it is sometimes satisfactory to assume that the temperature is 20 °C and the density of air is  $1.2 \times 10^{-3}$ . Had this been done, the computed value for the silicon would be

$$M_X = \frac{13 \left[ 1 - \frac{1.2 \times 10^{-3}}{8.3909} \right] + 0.010 \left[ 1 - \frac{1.2 \times 10^{-3}}{8.3909} \right] \left[ \frac{-3.5}{10.3} \right]}{1 - \frac{1.2 \times 10^{-3}}{2.3291}}$$

$$= 13.001442 \text{ g}$$

which is within 100 µg of the answer found in Example 1.

### EXAMPLE 3

Another piece of silicon is measured on a single-pan microbalance. The balance weights were adjusted by the manufacturer to the 8.0 apparent mass scale. The sensitivity of the balance has been determined to be exactly 1.000. This particular laboratory is well above sea level. At the time of the weighing, the following measurements were recorded:

$$P = 612.3 \text{ mm Hg}$$

$$t = 23.4 \text{ °C}$$

$$U = 23\%$$

The balance reading was 15.00 g on the built-in weights and 0.000358 g on the optical screen. What is the mass of the silicon?

### ANSWER

First, calculate  $e_S$  and  $\rho_A$ :

$$e_S = 21.59 \text{ mm Hg}$$

$$\rho_A = 0.956 \times 10^{-3} \text{ g cm}^{-3}$$

Then, use Eq. 4:

$$M_X = \frac{(15.00 + 0.000358) \left(1 - \frac{0.956 \times 10^{-3}}{8.0}\right)}{1 - \frac{0.956 \times 10^{-3}}{2.3291}}$$
$$= 15.004724 \text{ g.}$$

#### EXAMPLE 4

The built-in weights in Example 3 are actually stainless steel of density 7.78 g cm<sup>-3</sup> at 20 tC. What is the approximate error caused by using the apparent mass scale?

ANSWER

Using (8), the error is approximately

$$15\text{g}(0.2 \times 10^{-3} \text{g cm}^{-3}) \left( \frac{1}{7.78 \text{g cm}^{-3}} - \frac{1}{8.0 \text{cm}^{-3}} \right) = 11 \text{ micrograms}$$

This discrepancy, though larger than the precision of the best analytical balances, is actually well within the tolerance of Class 1 weights.

TABLE 1

<u>Mass</u> (g)	<u>Uncertainty</u> (g)	<u>Vol at 20 °C</u> (cm <sup>3</sup> )	<u>Coef of Exp</u>
100.00094070	.00002535	12.67439	.000045
50.00046277	.00001550	6.33719	.000045
30.00029259	.00001361	3.80232	.000045
20.00015779	.00001113	2.53487	.000045
10.00012644	.00001330	1.26744	.000045
5.00004198	.00000688	.63372	.000045
3.00004588	.00000459	.38023	.000045
2.00000627	.00000333	.25349	.000045
1.00001438	.00000300	.12674	.000045
.49995376	.00000160	.03012	.000020
.29996145	.00000115	.01807	.000020
.19994984	.00000087	.01205	.000020
.09996378	.00000091	.00602	.000020
.04998659	.00000072	.00301	.000020
.02999100	.00000077	.00181	.000020
.02000570	.00000066	.00741	.000069
.01000277	.00000086	.00370	.000069
.00499706	.00000070	.00185	.000069
.00300299	.00000076	.00111	.000069
.00200197	.00000066	.00074	.000069
.00100083	.00000086	.00037	.000060



TABLE 2

Temp (°C)	$e_s$ (mm Hg)
15.0	12.80
15.5	13.22
16.0	13.65
16.5	14.09
17.0	14.54
17.5	15.00
18.0	15.48
18.5	15.97
19.0	16.48
19.5	17.00
20.0	17.54
20.5	18.09
21.0	18.65
21.5	19.23
22.0	19.83
22.5	20.44
23.0	21.07
23.5	21.72
24.0	22.38
24.5	23.07
25.0	23.77
25.5	24.49
26.0	25.23
26.5	25.99
27.0	26.77
27.5	27.57
28.0	28.39
28.5	29.23
29.0	30.09
29.5	30.98



Recommended Standard Operations Procedure

for

Weighing by Double Substitution

Using an Equal-Arm Balance

1. Introduction

1.1. Purpose

The double substitution procedure is one in which a standard and an unknown weight are intercompared twice to determine the average difference between the two weighings. Any effects of inequality of arms and linear drift are eliminated by the weighing sequence used. Accordingly, the procedure is especially useful for high accuracy calibrations.

1.2. Prerequisites

1.2.1. Mass standards must be available with calibration certificates traceable to NBS.

1.2.2. The balance used must be in good operating condition as verified by a valid control chart or preliminary experiments to ascertain its performance quality.

1.2.3. The operator must be experienced in precision weighing techniques.

2. Methodology

2.1. Scope, Precision, Accuracy

This method is applicable to all weighings utilizing an equal arm balance. Because considerable effort is involved, it is most useful for calibrations of the highest accuracy. The precision will depend upon the sensitivity of the balance and the care exercised in making the required weighings. The accuracy will depend on the accuracy of calibration of the standard weights and the precision of the intercomparison.

2.2. Summary

The standard is balanced with respect to a counterweight. The standard is then replaced by the test weight which is weighed with respect to the same counterweight. A small weight (called a sensitivity weight) is added to the test weight and these are weighed. The standard and the same sensitivity weight are then weighed. The latter two weighings provide both second weighings of

the standard and test weights as well as a determination of the sensitivity of the balance under the load conditions. All weighings are made at regularly spaced time intervals to average out any effects due to linear instrument drift.

### 2.3. Apparatus/Equipment

- 2.3.1. Precision equal-arm balance with sufficient capacity and sensitivity for the calibrations planned. The index scale of the balance is conveniently numbered from 0 to 20 with 10 as the center division, although other numbering systems such as 0 to 200 are possible. A system in which the center division is 0 is not recommended since the negative readings that result can cause observational and/or computational problems. The graduations are so numbered that the addition of a small weight to the left arm will increase the scale reading.
- 2.3.2. Standard weights with valid calibrations, traceable to NBS. The sensitivity weight may be a calibrated secondary standard. The use of secondary standards as sensitivity weights reduces wear on the primary mass standards.
- 2.3.3. Counterweights, (uncalibrated), of approximately the same mass as the standard weights. Lead shot in a suitable container is useful for this purpose.
- 2.3.4. Small calibration weights (usually decimal fractions) to be used as tare weights.
- 2.3.5. Equipment capable of loading and unloading weights on the balance without damage to either (especially important in the case of large weights).
- 2.3.6. Stop watch or other timing device to observe time of each trial measurement.

### 2.4. Symbols

The following symbols are used in this procedure:

- S = standard weight
- X = weight calibrated
- T = counterweight
- t = small calibrated weight. A subscript s or x is used to indicate the larger weight with which it is associated.
- sw = small calibrated weight used to evaluate the sensitivity of the balance.
- M = the mass of a specific weight. Subscripts s, x, t, sw, are used to identify the weight
- AM = the apparent mass of a specific weight. Subscripts s, x, t, sw, are used to identify the weight.

## 2.5. Procedure

### 2.5.1. Preliminary Procedure

Conduct preliminary measurements (without recording data) to determine the values for  $T$ ,  $t_s$ ,  $t_x$ , and  $sw$  for use in measurement procedures, 2.5.2, or 2.5.3. This will serve to warm up the balance and facilitate the actual measurements in which the trials should be observed at regular time intervals. Estimate the time required to complete a trial (see 2.5.2) and maintain essentially equal intervals between successive trials (see 2.5.2.5). In a series of calibrations, this preliminary procedure is only required when new values need to be determined for  $T$ ,  $t_s$ ,  $t_x$ , and  $sw$ .

### 2.5.2. Measurement Procedure, Option A

Measurement No.	Left Pan (arm)	Right Pan (arm)	Observation
1	$S + t_s$	$T$	$O_1$
2	$X + t_x$	$T$	$O_2$
3	$X + t_x + sw$	$T$	$O_3$
4	$S + t_s + sw$	$T$	$O_4$

The turning points,  $O_1$ ,  $O_2$ ,  $O_3$ , and  $O_4$  are observed as described in GMP No. 1. All observations should be recorded on suitable data sheets, such as those in the Appendix.

2.5.2.1. Observation 1. Place the standard weight(s),  $S$ , equivalent to the nominal weight of the test weight, on the left pan (arm) of the balance and add a slight excess of counter weight ( $T$ ) to the right pan (arm). Add tare weight,  $t_s$ , to the left pan (arm) to obtain an approximate balance. Record the sum of the turning points and the time.

2.5.2.2. Observation 2. Remove weight(s)  $S$  and replace with test weight,  $X$ . Adjust tare weight,  $t_x$ , to obtain an approximate balance condition within one division of the sum of turning points obtained for  $O_1$  (for a 0 to 20 division graduated scale). (If  $S$  and  $X$  are approximately equal,  $t_x$  may equal  $t_s$ .) Record the sum of the turning points and the time.

2.5.2.3. Observation 3. Add a small sensitivity weight,  $sw$ , to the weights of observation 2. The value of  $sw$  should be such as to change the turning points by about 4 divisions on a 0 to 20 scale (40 divisions on a 0 to 200 scale). Record the sum of the turning points and the time.



2.5.2.4. Observation 4. Remove weight X and  $t_x$  and replace with S and  $t_s$ . Weight sw remains on the balance pan. Record the sum of the turning points and the time.

2.5.2.5. Calculate the time intervals between the successive trials. These should not differ from one another by more than  $\pm 20\%$ . If this difference is exceeded, reject the data and take a new series of measurements that will so agree.

### 2.5.3. Measurement Procedure, Option B

Measurement No.	Left Pan (arm)	Right Pan (arm)	Observation
1	X + $t_x$	T	O <sub>1</sub>
2	S + $t_s$	T	O <sub>2</sub>
3	S + $t_s$ + sw	T	O <sub>3</sub>
4	X + $t_x$ + sw	T	O <sub>4</sub>

Measurements for Option B are made as described in Option A except that X, S,  $t_x$ , and  $t_s$  are interchanged appropriately.

## 3. Calculations

3.1. Calculate the sums of the turning points, O<sub>1</sub>, O<sub>2</sub>, O<sub>3</sub> and O<sub>4</sub> (Ref. GMP No. 1).

3.2. Calculate the correction,  $C_x$ , required for the test weight, as follows, according to the optional sequence used. In each case,  $C_s$  is the apparent mass correction required for the standard weight used, including that for the tare weights as appropriate.

3.2.1. No air buoyancy correction. Calculate the apparent mass correction,  $C_x$ , for the test weight as follows, according to the optional sequence used. In each case, the appropriate apparent mass corrections for the standard weight(s),  $C_s$ , the tare weights  $AM_{t_s}$  and  $AM_{t_x}$ , and the sensitivity weights,  $AM_{sw}$ , are included. The symbols  $N_s$  and  $N_x$  refer to the nominal values of S and X, respectively.

#### 3.2.1.1. Optional Sequence A

$$C_x = C_s + AM_{t_s} - AM_{t_x} + \frac{(O_2 - O_1 + O_3 - O_4)}{2} \frac{(AM_{sw})}{(O_3 - O_2)} + N_s - N_x$$

#### 3.2.1.2. Optional Sequence B

$$C_x = C_s + AM_{t_s} - AM_{t_x} + \frac{(O_1 - O_2 + O_4 - O_3)}{2} \frac{(AM_{sw})}{(O_3 - O_2)} + N_s - N_x$$

### 3.2.2. Air Buoyancy Correction

3.2.2.1. Calculate the air density,  $\rho_A$ , as described in section 8 of the Appendix to SOP No. 2 or obtain from Table 9.9.

3.2.2.2. Calculate the mass  $M_X$  of the test weight X and its mass correction  $C'_X$  using the mass of the standard weight(s), the tare weights and the sensitivity weights according to the optional sequence used.

#### 3.2.2.2.1. Optional Sequence A

$$M_X = \frac{M_s \left(1 - \frac{\rho_A}{\rho_s}\right) + M_{t_s} \left(1 - \frac{\rho_A}{\rho_{t_s}}\right) - M_{t_x} \left(1 - \frac{\rho_A}{\rho_{t_x}}\right) + \frac{(0_2 - 0_1 + 0_3 - 0_4)}{2} \cdot \frac{M_{sw} \left(1 - \frac{\rho_A}{\rho_{sw}}\right)}{0_3 - 0_2}}{\left(1 - \frac{\rho_A}{\rho_X}\right)}$$

#### 3.2.2.2.2. Optional Sequence B

$$M_X = \frac{M_s \left(1 - \frac{\rho_A}{\rho_s}\right) + M_{t_s} \left(1 - \frac{\rho_A}{\rho_{t_s}}\right) - M_{t_x} \left(1 - \frac{\rho_A}{\rho_{t_x}}\right) + \frac{(0_1 - 0_2 + 0_4 - 0_3)}{2} \cdot \frac{M_{sw} \left(1 - \frac{\rho_A}{\rho_{sw}}\right)}{0_3 - 0_2}}{\left(1 - \frac{\rho_A}{\rho_X}\right)}$$

3.2.2.3. Calculate the mass correction  $C'_X$ , as follows:

$$C'_X = M_X - N_X$$

where  $N_X$  is the nominal value for X.

3.2.2.4. Calculate the apparent mass of X ( $AM_X$ ), versus the desired reference density of 8.0 g/cm<sup>3</sup> or brass. It is recommended that the apparent mass versus 8.0 g/cm<sup>3</sup> be reported unless otherwise requested. The density,  $\rho_X$ , must be in g/cm<sup>3</sup>.

#### 3.2.2.4.1. Apparent mass versus 8.0 g/cm<sup>3</sup>

$$AM_X \text{ vs } 8.0 = M_X \frac{\left(1 - \frac{.0012}{\rho_X}\right)}{0.999850}$$

#### 3.2.2.4.2. Apparent mass versus brass

$$AM_x \text{ vs brass} = M_x \frac{1 - \frac{.0012}{\rho_x}}{.999857}$$

#### 4. Assignment of Uncertainty

The limits of uncertainty,  $U$ , include estimates of the uncertainty of the mass standards used,  $U_s$ , plus the uncertainty of measurement,  $U_m$ , at the 99.73% level of confidence. The latter is estimated by

$$ts$$

where  $s$  is the standard deviation of measurement and  $t$  is obtained from Table 9.3.

Then

$$U = \pm [U_s + ts]$$

##### 4.1. Precision of Measurement Known from Control Chart Performance. (See SOP No. 9.)

The value for  $s$  is obtained from the control chart limits and current knowledge that the measurements are in a state of statistical control. This will need to be ascertained by measurement of at least one check standard while the above measurements are in progress.

Use the value of  $t$  (corresponding to a probability level of 99.73%) from Table 9.3 appropriate for the number of degrees of freedom,  $\nu$ , on which the control limits of the control chart are based.

##### 4.2. Precision Estimated from Series of Measurements

Measure a stable test object at least 7 times, no two measurements of which may be made on a single day. Calculate the mean and the standard deviation in the conventional manner. The latter is the value of  $s$  that is used in Section 4.1. In this case select the value for  $t$  from Table 9.3 based on the number of degrees of freedom involved in computing  $s$ .

Note: Repetitive measurements made on the same day estimate only the short-term standard deviation.

#### 5. Report

##### 5.1. Report results as described in SOP No. 1, Preparation of Calibration/Test Reports.

# Appendix

## Double Substitution Data Sheet for Equal-Arm Balance (Option A)

Test No.: \_\_\_\_\_ Sheet No.: \_\_\_\_\_ Date: \_\_\_\_\_

Item Identification: \_\_\_\_\_ Balance: \_\_\_\_\_

Standard Identification: \_\_\_\_\_ Observer: \_\_\_\_\_

Temperature: \_\_\_\_\_ Pressure: \_\_\_\_\_ Rel. Hum.: \_\_\_\_\_

$C_S(C'_S) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_  $AM_{sw}(M_{sw}) =$  \_\_\_\_\_

$\rho_S =$  \_\_\_\_\_  $\rho_X =$  \_\_\_\_\_  $\rho_{sw} =$  \_\_\_\_\_

$AM_{t_S}(M_{t_S}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_  $AM_{t_X}(M_{t_X}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_

$\rho_{t_S} =$  \_\_\_\_\_  $\rho_{t_X} =$  \_\_\_\_\_

Time \_\_\_\_\_ Balance standard deviation = \_\_\_\_\_

Measurement No.	Weights	Turning Points		
	Left Arm	Low	High	Sum
1	$S + t_S$			$O_1 =$
2	$X + t_X$			$O_2 =$
3	$X + t_X + sw$			$O_3 =$
4	$S + t_S + sw$			$O_4 =$

Time \_\_\_\_\_

# Appendix

## Double Substitution Data Sheet for Equal-Arm Balance (Option B)

Test No.: \_\_\_\_\_ Sheet No.: \_\_\_\_\_ Date: \_\_\_\_\_

Item Identification: \_\_\_\_\_ Balance: \_\_\_\_\_

Standard Identification: \_\_\_\_\_ Observer: \_\_\_\_\_

Temperature: \_\_\_\_\_ Pressure: \_\_\_\_\_ Rel. Hum.: \_\_\_\_\_

$C_S(C'_S) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_  $AM_{sw}(M_{sw}) =$  \_\_\_\_\_

$\rho_S =$  \_\_\_\_\_  $\rho_X =$  \_\_\_\_\_  $\rho_{sw} =$  \_\_\_\_\_

$AM_{t_S}(M_{t_S}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_  $AM_{t_X}(M_{t_X}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_

$\rho_{t_S} =$  \_\_\_\_\_  $\rho_{t_X} =$  \_\_\_\_\_

Time \_\_\_\_\_ Balance standard deviation = \_\_\_\_\_

Measurement No.	Weights	Turning Points		
	Left Arm	Low	High	Sum
1	$X + t_X$			$O_1 =$
2	$S + t_S$			$O_2 =$
3	$S + t_S + sw$			$O_3 =$
4	$X + t_X + sw$			$O_4 =$

Time \_\_\_\_\_



# Appendix

## Double Substitution Data Sheet for Equal-Arm Balance (Option A)

Test No.: 137 Sheet No.: 1 Date: 8/27/86

Item Identification: 1000 lb No. 623 Balance: Russell

Standard Identification: #11 and #22 Observer: HO

Temperature: 23.5°C Pressure: 746.1 mm Hg Rel. Hum.: 45%

$\theta_s(C'_s) =$  0.0047 lb  $\pm$  .0030 lb  $\overline{AM}_{sw}(M_{sw}) =$  0.01 lb

$\rho_s =$  8.0 g/cm<sup>3</sup>  $\rho_x =$  7.0 g/cm<sup>3</sup>  $\rho_{sw} =$  7.8 g/cm<sup>3</sup>

$\overline{AM}_{t_s}(M_{t_s}) =$  3.625  $\pm$  .000075 lb  $\overline{AM}_{t_x}(M_{t_x}) =$  3.642 lb  $\pm$  negligible

$\rho_{t_s} =$  8.0 g/cm<sup>3</sup>  $\rho_{t_x} =$  7.8 g/cm<sup>3</sup>

Time 10:45 a Balance standard deviation = 0.0018 lb

Measurement No.	Weights	Turning Points		
	Left Arm	Low	High	Sum
1	S + t <sub>s</sub>	<del>5.1</del> 5.2	<del>15.3</del> 15.2 15.2	O <sub>1</sub> = 20.4
2	X + t <sub>x</sub>	<del>5.8</del> 5.9 5.9	<del>14.1</del> 14.1	O <sub>2</sub> = 20.0
3	X + t <sub>x</sub> + sw	<del>6.1</del> 6.2	<del>17.6</del> 17.5 17.4	O <sub>3</sub> = 23.65
4	S + t <sub>s</sub> + sw	<del>6.3</del> 6.4 6.4	<del>16.3</del> 16.2	O <sub>4</sub> = 22.6

Time 11:05 a

$$\rho_a = 1.163 \text{ mg/cm}^3 = 0.001163 \text{ g/cm}^3$$

$$M_X = \frac{M_s \left(1 - \frac{\rho_A}{\rho_s}\right) + M_{t_s} \left(1 - \frac{\rho_A}{\rho_{t_s}}\right) - M_{t_x} \left(1 - \frac{\rho_A}{\rho_{t_x}}\right) + \frac{(0_2 - 0_1 + 0_3 - 0_4)}{2} \cdot \frac{M_{sw} \left(1 - \frac{\rho_A}{\rho_{sw}}\right)}{0_3 - 0_2}}{1 - \frac{\rho_A}{\rho_x}}$$

$$M_X = \left[ 1000.0047 \left(1 - \frac{.001163}{8.0}\right) + 3.625 \left(1 - \frac{.001163}{8.0}\right) - 3.642 \left(1 - \frac{.001163}{7.8}\right) + \frac{20.0 - 20.4 + 23.65 - 22.6}{2} \cdot \frac{.01 \left(1 - \frac{.001163}{7.8}\right)}{23.65 - 20.0} \right] / \left(1 - \frac{.001163}{7.0}\right)$$

$$M_X = \frac{999.8593243 + 3.6244730 - 3.6414570 + .0008903}{.99983386}$$

$$M_X = 1000.009372 \text{ lb}$$

$$\text{Uncertainty} = t_s + U_s \quad \text{Degrees of freedom in } s = 20$$

$$\text{Uncertainty} = 3.422 (.0018) + (.0030 + .000075)$$

$$\text{Uncertainty} = 0.0092346 \text{ lb}$$

$$M_X = 1000.0094 \pm 0.0092 \text{ lb}$$

$$C'_X = M_X - N_X = 0.0094 \pm 0.0092 \text{ lb}$$

$$AM_X \text{ vs } 8.0 = M_X \frac{\left(1 - \frac{.0012}{\rho_x}\right)}{0.999850}$$

$$AM_X \text{ vs } 8.0 = 1000.0094 \frac{\left(1 - \frac{.0012}{7.0}\right)}{.999850}$$

$$AM_X \text{ vs. } 8.0 = 999.9880 \pm .0092 \text{ lb}$$

$$C_X = AM_X \text{ vs } 8.0 - N_X$$

$$C_X = 999.9880 - 1000 = -.0120 \pm .0092 \text{ lb}$$

SOP No. 4

Recommended Standard Operations Procedure

for

Weighing by Double Substitution Using a Single-Pan Mechanical  
Balance, a Full Electronic Balance, or a Balance with Digital  
Indications and Built-In Weights

1. Introduction

1.1. Purpose

The double substitution procedure is one in which a standard and an unknown weight are intercompared twice to determine the average difference between the two weights. Errors in any built-in weights or in the balance indications are eliminated by using the balance only as a comparator and by calibrating the balance indications over the range of use for the measurement. Accordingly, the procedure is especially useful for high accuracy calibrations.

1.2. Prerequisites

1.2.1. Mass standards must be available with calibration certificates traceable to NBS.

1.2.2. The balance used must be in good operating condition as verified by a valid control chart or preliminary experiments to ascertain its performance quality.

1.2.3. The operator must be experienced in precision weighing techniques.

2. Methodology

2.1. Scope, Precision, Accuracy

This method is applicable to all weighings utilizing a single-pan mechanical balance, a full electronic balance, or a balance that combines digital indications with the use of built-in weights. The precision will depend upon the sensitivity of the balance and the care exercised to make the required weighings. The accuracy will depend upon the accuracy of the calibration of the standard weights and the precision of the intercomparison.

2.2. Summary

The balance is adjusted if necessary, to obtain balance indications for all measurements that will be within the range of the optical scale or digital indications of the balance without changing the dial settings for the built-in weights, if present. The standard and the

test weight are each weighed. A small calibrated weight, called a sensitivity weight, is added to the test weight and these are weighed. The standard and the same sensitivity weight are then weighed. The latter two weighings provide both second weighings of the standard and the test weight as well as a determination of the sensitivity of the balance under the load conditions at the time of the intercomparison. All weighings are made at regularly spaced time intervals to average out any effects due to instrument drift.

The double substitution procedure is the same for all of the balances mentioned above but the adjustment of the balance to prepare for the intercomparison and the selection of the sensitivity weight varies slightly depending upon the balance used. When steps specific to a particular balance are required, they are given in subsections of the procedure identified by a, b, and c along with the balance type.

### 2.3. Apparatus/Equipment Required

- 2.3.1. Precision balance with sufficient capacity and sensitivity for the calibrations planned.
- 2.3.2. Standard weights with valid calibrations traceable to NBS. The sensitivity weight may be a calibrated secondary standard. The use of secondary standards as sensitivity weights reduces wear on the primary mass standards.
- 2.3.3. Small standard weights with valid calibrations traceable to NBS to be used as tare weights.
- 2.3.4. Uncalibrated weights to be used to adjust the balance to the desired reading range.
- 2.3.5. Forceps to handle the weights or gloves to be worn if the weights are moved by hand.
- 2.3.6. Stop watch or other timing device to observe the time of each measurement.
- 2.3.7. \*Thermometer accurate to 0.10 °C to determine air temperature.
- 2.3.8. \*Barometer accurate to 0.5 mm of mercury (66.5 Pa) to determine air pressure.
- 2.3.9. \*Hygrometer accurate to 10 percent to determine relative humidity.

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\*The thermometer, barometer, and hygrometer are used to determine the air density at the time of the measurement. The air density is used to make an air buoyancy correction. The accuracies specified are recommended for high precision calibration. Less accurate equipment can be used with only a small degradation in the overall accuracy of the measurement.

## 2.4. Symbols

The following symbols are used in this procedure:

- S - standard weight
- X - weight calibrated
- T - counterweight
- t - small calibrated weight. A subscript s or x is used to indicate the larger weight with which it is associated.
- sw - small calibrated weight used to evaluate the sensitivity of the balance.
- M - the mass of a specific weight. Subscripts s, x, t, sw, are used to identify the weight
- AM - the apparent mass of a specific weight. Subscripts s, x, t, sw, are used to identify the weight.

## 2.5. Procedure

### 2.5.1. Preliminary Procedure

Place the test weight and standards in the balance chamber or near the balance overnight to permit the weights and the balance to attain thermal equilibrium. Conduct preliminary measurements to obtain an approximate value for the difference between the standard and the unknown, to determine where the readings occur on the balance, to determine if tare weights are required, to determine the sensitivity weight that must be used, and to determine the time interval required for the balance indication to stabilize.

Tare weights are rarely needed for high precision mass standards. If tare weights are required, carry tare weights,  $t_s$  and  $t_x$ , with the standard and the unknown, S and X, respectively. The tare weights must be calibrated standards. The standard and its tare weight,  $S + t_s$ , should be "nearly the same mass" as the unknown with its tare weight,  $X + t_x$ . "Nearly the same mass" depends upon the balance used. Select  $t_s$  and  $t_x$  such that the difference in mass between  $S + t_s$  and  $X + t_x$  is:

- a. Single-pan mechanical balance - less than  $\frac{1}{4}$  the range of the optical scale.
- b. Full electronic balance - less than 0.05 percent of the balance capacity.
- c. Combination balance - less than  $\frac{1}{4}$  the range of the digital indications.

Select a sensitivity weight that is:

- a. Single-pan balance - between  $\frac{1}{4}$  and  $\frac{1}{2}$  the range of the optical scale, and at least three times the mass difference between X and S.



- b. Full electronic balance - at least 10 to 20 times the mass difference between X and S but not exceeding 1 percent of the balance capacity.
- c. Combination balance - between  $\frac{1}{4}$  and  $\frac{1}{2}$  the range of the digital indications, and at least three times the mass difference between X and S.

Adjust the single pan balance or the combination balance so the first two readings of the double substitution fall in the first quarter of the optical scale or digital indications. The zero adjustment and tare adjustment may be used. Small weights may be placed on the balance pan to reach the desired reading range. These weights remain on the pan throughout the double substitution. Once the balance has been adjusted to the desired position, neither the balance dials, the zero and tare adjustments, nor the small weights placed on the balance pan are to be changed during the measurement.

If the balance is equipped with a pan arrestment mechanism, arrest the pan between each observation.

#### 2.5.2. Measurement Procedure, Option A

Measurement No.	Weights on Pan	Observation
1	$S + t_s$	$O_1$
2	$X + t_x$	$O_2$
3	$X + t_x + sw$	$O_3$
4	$S + t_s + sw$	$O_4$

All observations should be recorded on suitable data sheets, such as those in the appendix. Record the laboratory ambient temperature, barometric pressure, and relative humidity.

2.5.2.1. Trial 1. Place the standard weight(s), S, along with  $t_s$  on the balance pan. If equipped with a pan arrestment mechanism, release the balance pan. When the pan is released, start the stop-watch and record observation  $O_1$  once the balance indication has stabilized.

2.5.2.2. Trial 2. Remove weight(s) S and  $t_s$  and replace with test weight X and its tare weight,  $t_x$ . Release the pan, time the interval, and record observation  $O_2$ .

2.5.2.3. Trial 3. Add the sensitivity weight, sw, to the weights of observation 2. Release the pan, time the interval, and record observation  $O_3$ .

2.5.2.4. Trial 4. Remove weights X and  $t_x$  and replace with S and  $t_s$ . The sensitivity weight,  $sw$ , remains on the balance pan. Release the pan, time the interval, and record observation  $0_4$ .

2.5.2.5. If repeated double substitutions are performed, the time intervals between successive trials should not differ from one another by more than  $\pm 20\%$ . If this difference is exceeded, reject the data and take a new series of measurements that will so agree.

### 2.5.3. Measurement Procedure, Option B

Measurement No.	Weights on Pan	Observation
1	$X + t_x$	$0_1$
2	$S + t_s$	$0_2$
3	$S + t_s + sw$	$0_3$
4	$X + t_x + sw$	$0_4$

Measurements for Option B are made as described in Option A except that X, S,  $t_x$ , and  $t_s$  are interchanged appropriately.

## 3. Calculations

3.1. No air buoyancy correction. Calculate the apparent mass correction,  $C_x$ , for the test weight as follows, according to the optional sequence used. In each case, the apparent mass corrections for the standard weight(s),  $C_s$ , the tare weights,  $AM_{t_s}$  and  $AM_{t_x}$ , and the sensitivity weight,  $AM_{sw}$  are included. The symbols  $N_s$  and  $N_x$  refer to the nominal values of S and X, respectively.

### 3.1.1. Optional Sequence A

$$C_x = C_s + AM_{t_s} - AM_{t_x} + \frac{(0_2 - 0_1 + 0_3 - 0_4)}{2} \left( \frac{AM_{sw}}{0_3 - 0_2} \right) + N_s - N_x$$

### 3.1.2. Optional Sequence B

$$C_x = C_s + AM_{t_s} - AM_{t_x} + \frac{(0_1 - 0_2 + 0_4 - 0_3)}{2} \left( \frac{AM_{sw}}{0_3 - 0_2} \right) + N_s - N_x$$

## 3.2. Air Buoyancy Correction

3.2.1. Calculate the air density,  $\rho_A$ , as described in section 8 of the Appendix to SOP No. 2 or obtain from Table 9.9.

3.2.2. Calculate the mass  $M_x$  of the test weight, and its mass correction  $C'_x$  using the mass of the standard weight(s), the tare weights and the sensitivity weights according to the optional sequence used.

### 3.2.2.1. Optional Sequence A

$$M_x = \frac{M_s \left(1 - \frac{\rho_A}{\rho_s}\right) + M_{t_s} \left(1 - \frac{\rho_A}{\rho_{t_s}}\right) - M_{t_x} \left(1 - \frac{\rho_A}{\rho_{t_x}}\right) + \frac{(0_2 - 0_1 + 0_3 - 0_4)}{2} \cdot \frac{M_{sw} \left(1 - \frac{\rho_A}{\rho_{sw}}\right)}{0_3 - 0_2}}{1 - \frac{\rho_A}{\rho_x}}$$

### 3.2.2.2. Optional Sequence B

$$M_x = \frac{M_s \left(1 - \frac{\rho_A}{\rho_s}\right) + M_{t_s} \left(1 - \frac{\rho_A}{\rho_{t_s}}\right) - M_{t_x} \left(1 - \frac{\rho_A}{\rho_{t_x}}\right) + \frac{(0_1 - 0_2 + 0_4 - 0_3)}{2} \cdot \frac{M_{sw} \left(1 - \frac{\rho_A}{\rho_{sw}}\right)}{0_3 - 0_2}}{1 - \frac{\rho_A}{\rho_x}}$$

3.2.3. Calculate the mass correction  $C'_x$ , as follows:

$$C'_x = M_x - N_x$$

where  $N_x$  is the nominal value for X.

3.2.3.1. Calculate the apparent mass of x versus the desired reference density of 8.0 g/cm<sup>3</sup> or brass. It is recommended that the apparent mass versus 8.0 g/cm<sup>3</sup> be reported unless otherwise requested. The density of X,  $\rho_x$ , must be in g/cm<sup>3</sup>.

3.2.3.1.1. Apparent mass versus 8.0 g/cm<sup>3</sup>

$$AM_x \text{ vs. } 8.0 = M_x \frac{\left(1 - \frac{.0012}{\rho_x}\right)}{0.999850}$$

3.2.3.1.2. Apparent mass versus brass

$$AM_x \text{ vs. brass} = M_x \frac{\left(1 - \frac{.0012}{\rho_x}\right)}{0.999857}$$

#### 4. Assignment of Uncertainty

The limits of uncertainty,  $U$ , include estimates of the uncertainty of the mass standards used,  $U_s$ , plus the uncertainty of measurement,  $U_m$ , at the 99.73% level of confidence. The latter is estimated by

$$ts$$

where  $s$  is the standard deviation of measurement and  $t$  is obtained from Table 9.3.

Then 
$$U = \pm [U_s + ts]$$

##### 4.1. Precision of Measurement Known from Control Chart Performance. (See SOP No. 9.)

The value for  $s$  is obtained from the control chart data. Statistical control will need to be verified by measuring at least one check standard while the above measurements are in progress.

Use the value of  $t$  (corresponding to a probability level by 99.73%) from table 9.3 appropriate for the number of degrees of freedom,  $\nu$ , on which the control limits of the control chart are based.

##### 4.2. Precision Estimated from Series of Measurements

Measure a stable test object at least 7 times, no two measurement of which may be made on a single day. Calculate the mean and the standard deviation in the conventional manner. The latter is the value of  $s$  that is used in Section 4.4. In this case select the value for  $t$  from table 9.3 based on the number of degrees of freedom involved in computing  $s$ .

Note: Repetitive measurements made on the same day estimate only the short-term standard deviation.

#### 5. Report

##### 5.1. Report results as described in SOP No. 1, Preparation of Calibration/Test Reports.

# Appendix

## Double Substitution Data Sheet for Single Pan Balance (Option A)

Test No.: \_\_\_\_\_ Sheet No.: \_\_\_\_\_ Date: \_\_\_\_\_

Item Identification: \_\_\_\_\_ Balance: \_\_\_\_\_

Standard Identification: \_\_\_\_\_ Observer: \_\_\_\_\_

Temperature: \_\_\_\_\_ Pressure: \_\_\_\_\_ Rel. Hum.: \_\_\_\_\_

$C_s(C'_s) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_  $AM_{sw}(M_{sw}) =$  \_\_\_\_\_

$\rho_s =$  \_\_\_\_\_  $\rho_x =$  \_\_\_\_\_  $\rho_{sw} =$  \_\_\_\_\_

$AM_{t_s}(M_{t_s}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_  $AM_{t_x}(M_{t_x}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_

$\rho_{t_s} =$  \_\_\_\_\_  $\rho_{t_x} =$  \_\_\_\_\_

Time \_\_\_\_\_ Balance standard deviation = \_\_\_\_\_

Measurement No.	Weights	Observations
1	$S + t_s$	$O_1 =$
2	$X + t_x$	$O_2 =$
3	$X + t_x + sw$	$O_3 =$
4	$S + t_s + sw$	$O_4 =$

Time \_\_\_\_\_



# Appendix

## Double Substitution Data Sheet for Single Pan Balance (Option B)

Test No.: \_\_\_\_\_ Sheet No.: \_\_\_\_\_ Date: \_\_\_\_\_

Item Identification: \_\_\_\_\_ Balance: \_\_\_\_\_

Standard Identification: \_\_\_\_\_ Observer: \_\_\_\_\_

Temperature: \_\_\_\_\_ Pressure: \_\_\_\_\_ Rel. Hum.: \_\_\_\_\_

$C_S(C'_S) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_  $AM_{sw}(M_{sw}) =$  \_\_\_\_\_

$\rho_S =$  \_\_\_\_\_  $\rho_X =$  \_\_\_\_\_  $\rho_{sw} =$  \_\_\_\_\_

$AM_{t_s}(M_{t_s}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_  $AM_{t_x}(M_{t_x}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_

$\rho_{t_s} =$  \_\_\_\_\_  $\rho_{t_x} =$  \_\_\_\_\_

Time \_\_\_\_\_ Balance standard deviation = \_\_\_\_\_

Measurement No.	Weights	Observations
1	$X + t_x$	$O_1 =$
2	$S + t_s$	$O_2 =$
3	$S + t_s + sw$	$O_3 =$
4	$X + t_x + sw$	$O_4 =$

Time \_\_\_\_\_

# Appendix

## Double Substitution Data Sheet for Single Pan Balance (Option A)

Test No.: 123 Sheet No.: 1 Date: 8/24/86

Item Identification: 10 g Set 4 Balance: micro

Standard Identification: 10g state standard Observer: HO

Temperature: 22.3 Pressure: 753.5 mm Hg Rel. Hum.: 45%

$G_S(C'_S) =$  -0.679 mg  $\pm$  0.014 mg  $\Delta M_{SW}(M_{SW}) =$  4.9773 mg

$\rho_S =$  8.0 g/cm<sup>3</sup>  $\rho_X =$  7.84 g/cm<sup>3</sup>  $\rho_{SW} =$  8.5 g/cm<sup>3</sup>

$AM_{t_S}(M_{t_S}) =$  0  $\pm$              $AM_{t_X}(M_{t_X}) =$  0  $\pm$            

$\rho_{t_S} =$              $\rho_{t_X} =$            

Time 8:35 a Balance standard deviation = 0.0029 mg

Measurement No.	Weights	Observations
1	S + t <sub>s</sub>	O <sub>1</sub> = 1.268
2	X + t <sub>x</sub>	O <sub>2</sub> = 1.821
3	X + t <sub>x</sub> + sw	O <sub>3</sub> = 6.798
4	S + t <sub>s</sub> + sw	O <sub>4</sub> = 6.245

Time 8:41 a

$$\rho_a = 1.180 \text{ mg/cm}^3 = 0.00180 \text{ g/cm}^3$$

$$M_x = \frac{M_s \left(1 - \frac{\rho_A}{\rho_s}\right) + M_{t_s} \left(1 - \frac{\rho_A}{\rho_{t_s}}\right) - M_{t_x} \left(1 - \frac{\rho_A}{\rho_{t_x}}\right) + \frac{(0_2 - 0_1 + 0_3 - 0_4)}{2} \cdot \frac{M_{sw} \left(1 - \frac{\rho_A}{\rho_{sw}}\right)}{0_3 - 0_2}}{1 - \frac{\rho_A}{\rho_x}}$$

$$M_x = \frac{(9.999321\text{g}) \left(1 - \frac{.00118}{8}\right) + 0 - 0 + \frac{1.821 - 1.268 + 6.798 - 6.245}{2} \cdot \frac{.0049773 \left(1 - \frac{.00118}{8.5}\right)}{6.798 - 1.821}}{1 - \frac{.00118}{7.84}}$$

$$M_x = \frac{9.9978461 + .000552957}{.99984949} = 9.9999041\text{g}$$

$$C'_x = M_x - N_x = 9.9999041\text{g} - 10\text{g} = -0.0959\text{mg}$$

$$\text{Uncertainty} = t_s + U_s \quad \text{Degrees of freedom in } s = 30$$

$$\text{Uncertainty} = 3.270 (.0029) + .014\text{mg}$$

$$\text{Uncertainty} = 0.023483\text{mg}$$

$$C'_x = -0.096 \pm 0.023\text{mg}$$

$$\begin{aligned} AM_x \text{ vs } 8.0 &= M_x \frac{\left(1 - \frac{\rho_n}{\rho_x}\right)}{\left(1 - \frac{\rho_n}{8.0}\right)} \\ &= \frac{9.999904\text{g} \left(1 - \frac{.0012}{7.84}\right)}{\left(1 - \frac{.0012}{8}\right)} \\ AM_x \text{ vs } 8.0 &= \frac{9.999904 \cdot .9998469}{.999850} = 9.99987338 \text{ g} \end{aligned}$$

$$C_x = AM_x \text{ vs } 8.0 - N_x = 9.99987338 \text{ g} - 10 \text{ g}$$

$$C_x = -0.126616 \text{ mg}$$

$$C_x = -0.127 \pm 0.023 \text{ mg}$$

# Appendix

## Double Substitution Data Sheet for Single Pan Balance (Option B)

Test No.: 125 Sheet No.: 1 Date: 8/24/86

Item Identification: 1 troy ounce Set 1 Balance: CB 100

Standard Identification: 30g state standard Observer: HO

Temperature: 22.3 Pressure: 753.5 mm Hg Rel. Hum.: 45%

$C_s (C'_s) =$  0.407 mg  $\pm$  0.022 mg  $AM_{sw} (M_{sw}) =$  49.916 mg

$\rho_s =$  8.0 g/cm<sup>3</sup>  $\rho_x =$  7.84 g/cm<sup>3</sup>  $\rho_{sw} =$  8.5 g/cm<sup>3</sup>

$AM_{t_s} (M_{t_s}) =$  1.1003596g  $\pm$  0.0063mg  $AM_{t_x} (M_{t_x}) =$  0  $\pm$        

$\rho_{t_s} =$  8.04 g/cm<sup>3</sup>  $\rho_{t_x} =$        

Time 9:00 a Balance standard deviation = 0.018 mg

Measurement No.	Weights	Observations
1	$X + t_x$	$O_1 = 20.93$
2	$S + t_s$	$O_2 = 17.21$
3	$S + t_s + sw$	$O_3 = 67.08$
4	$X + t_x + sw$	$O_4 = 70.81$

Time 9:05 a

$$C_x = C_s + AM_{t_s} - AM_{t_x} + \frac{O_1 - O_2 + O_4 - O_3}{2} \cdot \frac{AM_{sw}}{O_3 - O_2} + N_s - N_x$$

$$C_x = 0.407\text{mg} + 1.1003596\text{g} - 0 + \frac{20.93 - 17.21 + 70.81 - 67.08}{2} \cdot \frac{49.916 \text{ mg}}{67.08 - 17.21} + 30\text{g} - 1 \text{ t oz}$$

$$C_x = 0.407 \text{ mg} + 1100.3596 \text{ mg} + 3.7284359 \text{ mg} + 30 \text{ g} - 31.1034768 \text{ g}$$

$$C_x = 1104.495036 \text{ mg} - 1.1034768 \text{ g}$$

$$C_x = 1.018236 \text{ mg}$$

$$\text{Uncertainty} = t_s + U_s \quad \text{Degrees of freedom in } s = 60$$

$$\text{Uncertainty} = 3.130 (0.018 \text{ mg}) + (0.022 + 0.0063 \text{ mg})$$

$$\text{Uncertainty} = 0.08464 \text{ mg}$$

$$C_x = 1.018 \text{ mg} \pm 0.085 \text{ mg}$$

$$C_x = 0.0000327 \text{ t oz} \pm 0.0000027 \text{ t oz}$$





Recommended Standard Operations Procedure

for

Using a 3-1 Weighing Design (Double Substitution)

1. Introduction

1.1. Purpose

The 3-1 weighing design is a combination of three intercomparisons of three weights of equal nominal value; a standard, an unknown weight, and a second standard called a check standard. The weights are intercompared using the double substitution procedure which can be performed using an equal-arm, single-pan mechanical, full electronic, and a combination balance utilizing built-in weights and a digital indication. The specific SOP for the double substitution procedure for each balance is to be followed. The 3-1 weighing design provides two methods of checking the validity of the measurement. Hence, the procedure is especially useful for high accuracy calibrations in which it is critical to assure that the measurements are valid and well documented.

1.2. Prerequisites

1.2.1. Mass standards must be available with calibration certificates traceable to NBS.

1.2.2. The balance used must be in good operating condition as verified by a valid control chart or preliminary experiments to ascertain its performance quality.

1.2.3. The operator must be experienced in precision weighing techniques.

2. Methodology

2.1. Scope, Precision, Accuracy

This method can be performed on any type of balance using the appropriate double substitution SOP for the particular balance. Because considerable effort is involved, this weighing design is most useful for calibrations of the highest accuracy. The weighing design utilizes three double substitutions to calibrate a single unknown weight. This introduces redundancy into the measurement process and permits two checks on the validity of the measurement; one on accuracy and the other on balance repeatability. A least-squares best fit analysis is done on the measurements to assign a value to the unknown weight. The precision will depend upon the sensitivity of the balance and the care exercised to make the required weighings. The accuracy will depend upon the accuracy of the calibration of the standard weights and the precision of the intercomparison.

## 2.2. Summary

A standard weight, an unknown weight, and a check standard are intercompared in a specific order using the double substitution procedure. The balance and the weights must be prepared according to the appropriate double substitution SOP for the particular balance being used. Once the balance and weights have been prepared, all readings must be taken from the reading scale of the balance without adjusting the balance or weights in any way. Within a double substitution all weighings are made at regularly spaced time intervals to average out any effects due to instrument drift. Because of the amount of effort required to perform the 3-1 weighing design, the procedure includes the air buoyancy correction.

## 2.3. Apparatus/Equipment Required

- 2.3.1. Precision balance with sufficient capacity and sensitivity for the calibrations planned.
- 2.3.2. Standard weights with valid calibrations traceable to NBS. The sensitivity weight may be a calibrated secondary standard. The use of a secondary standard as a sensitivity weight reduces wear on the primary mass standards.
- 2.3.3. Small standard weights with valid calibrations traceable to NBS to be used as tare weights.
- 2.3.4. Uncalibrated weights to be used to adjust the balance to the desired reading range.
- 2.3.5. Forceps to handle the weights or gloves to be worn if the weights are moved by hand.
- 2.3.6. Stop watch or other timing device to observe the time of each measurement.
- 2.3.7. \*Thermometer accurate to 0.10 °C to determine air temperature.
- 2.3.8. \*Barometer accurate to 0.5 mm of mercury (66.5 Pa) to determine air pressure.
- 2.3.9. \*Hygrometer accurate to 10 percent to determine relative humidity.

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\*The thermometer, barometer, and hygrometer are used to determine the air density at the time of the measurement. The air density is used to make an air buoyancy correction. The accuracies specified are recommended for high precision calibration. Less accurate equipment can be used with only a small degradation in the overall accuracy of the measurement.

## 2.4. Procedure

2.4.1. Place the test weight and standards in the balance chamber or near the balance overnight to permit the weights and the balance to attain thermal equilibrium. Conduct preliminary measurements to determine the tare weights that may be required, the size of the sensitivity weight required, adjust the balance to the appropriate reading range of the balance indications, and to exercise the balance. Refer to the appropriate double substitution SOP for details.

### 2.4.2. Measurement Procedure

Record the pertinent information for the standard,  $S$ , unknown,  $X$ , and check standard,  $S_c$ , as indicated on a suitable data sheet such as the one in the Appendix of this SOP. Record the laboratory ambient temperature, barometric pressure, and relative humidity. Perform the measurements in the order shown below.

Double Substitution	Measurement No.	Weights on Pan	Observation
1	1	$S + t_s$	$0_1$
	2	$X + t_x$	$0_2$
	3	$X + t_x + sw$	$0_3$
	4	$S + t_s + sw$	$0_4$
2	5	$S + t_s$	$0_1$
	6	$S_c + t_{s_c}$	$0_2$
	7	$S_c + t_{s_c} + sw$	$0_3$
	8	$S + t_s + sw$	$0_4$
3	9	$X + t_x$	$0_1$
	10	$S_c + t_{s_c}$	$0_2$
	11	$S_c + t_{s_c} + sw$	$0_3$
	12	$X + t_x + sw$	$0_4$

where

$t_s$  = calibrated tare weights carried with  $S$   
 $t_x$  = calibrated tare weights carried with  $X$   
 $t_{s_c}$  = calibrated tare weights carried with  $S_c$   
 $sw^c$  = calibrated sensitivity weight

### 3. Calculations

3.1. Calculate the air density,  $\rho_A$ , as described in section 8 of the Appendix to SOP No. 2 or use Table 9.9.

3.2. Calculate the measured differences,  $a_1$ ,  $a_2$ , and  $a_3$ , for the weights used in each double substitution using the following formula:

$$a = \frac{(0_1 - 0_2 + 0_4 - 0_3)}{2} \cdot \frac{M_{sw} \left(1 - \frac{\rho_A}{\rho_{sw}}\right)}{0_3 - 0_2}$$

where

$M_{sw}$  = mass of the sensitivity weight

$\rho_{sw}$  = density of the sensitivity weight

3.3. Calculate the short-term standard deviation (S.D.) for the 3-1 weighing design. This standard deviation has one degree of freedom.

$$\text{Short term S.D.} = 0.577(a_1 - a_2 + a_3)$$

3.4. Compute the F statistic which compares the short-term S.D. to the pooled short-term S.D. (See chapter 8.4 for the 3-1 weighing designs performed on the balance used.)

$$F\text{-statistic} = \frac{(\text{short-term S.D.})^2}{(\text{Pooled short-term S.D.})^2}$$

The F-statistic so computed must be less than the F-value obtained from an F-table at 99% confidence level (Table 9.5) to be acceptable. The F-value is obtained from the F-table for numerator degrees of freedom equal one, and denominator degrees of freedom equal to the number of degrees of freedom in the pooled short-term S.D. If the data fails the F-test and the source of the error cannot be determined conclusively, the measurement must be repeated.

3.5. Compute the least-squares measured difference  $d_x$  for X from S.

$$d_x = \frac{-2a_1 - a_2 + a_3}{3}$$

3.6. Compute the mass  $M_x$  of X.

$$M_x = \frac{M_s \left(1 - \frac{\rho_A}{\rho_s}\right) + d_x + M_{t_s} \left(1 - \frac{\rho_A}{\rho_{t_s}}\right) - M_{t_x} \left(1 - \frac{\rho_A}{\rho_{t_x}}\right)}{\left(1 - \frac{\rho_A}{\rho_x}\right)}$$



where

$\rho_A$  = air density

$M_i$  = Mass for weight i

$\rho_i$  = density for weight i

- 3.7. Calculate the apparent mass of X versus the desired reference density of 8.0 g/cm<sup>3</sup> or brass. It is recommended that the apparent mass versus 8.0 g/cm<sup>3</sup> be reported unless otherwise requested. The density of X,  $\rho_X$ , must be in g/cm<sup>3</sup>. (See SOP No. 2)

- 3.7.1. Apparent mass versus 8.0 g/cm<sup>3</sup>

$$AM_X \text{ vs. } 8.0 = \frac{M_X \left( 1 - \frac{.0012}{\rho_X} \right)}{0.999850}$$

- 3.7.2. Apparent mass versus brass

$$AM_X \text{ vs. brass} = \frac{M_X \left( 1 - \frac{.0012}{\rho_X} \right)}{0.999857}$$

- 3.8. Compute the least-squares measured difference  $d_{s_c}$  of  $S_c$  from S.

$$d_{s_c} = \frac{-a_1 - 2a_2 - a_3}{3}$$

- 3.9. Compute the mass of  $S_c$ ,  $M_{s_c}$ .

$$M_{s_c} = \frac{M_s \left( 1 - \frac{\rho_A}{\rho_s} \right) + d_{s_c} + M_{t_s} \left( 1 - \frac{\rho_A}{\rho_{t_s}} \right) - M_{t_{s_c}} \left( 1 - \frac{\rho_A}{\rho_{t_{s_c}}} \right)}{1 - \frac{\rho_A}{\rho_{s_c}}}$$

The mass determined for the check standard should be plotted on the control chart and must lie within the control limits. If it does not, and the source of error cannot be found, the measurement must be repeated.

#### 4. Assignment of Uncertainty

The limits of uncertainty,  $U$ , include estimates of the uncertainty of the mass standards used,  $U_s$ , plus the uncertainty of measurement,  $U_m$ , at the 99.73% level of confidence. The latter is estimated by

$$ts$$

where  $s$  is the standard deviation of measurement and  $t$  is obtained from Table 9.3.

Then

$$U = \pm [U_s + ts]$$

##### 4.1. Precision of Measurement Known from Control Chart Performance (See SOP No. 9.)

The value for  $s$  is obtained from the control chart data for 3-1 weighing designs and double substitution measurements. Statistical control needs to be verified by the measurement of the check standard in the 3-1 design.

Use the value of  $t$  (corresponding to a probability level of 99.73%) from Table 9.3 appropriate for the number of degrees of freedom,  $\nu$ , on which the control limits of the control chart are based.

#### 5. Report

##### 5.1. Report results as described in SOP No. 1, Preparation of Calibration/Test Reports.

# Appendix

## 3-1 Weighing Design When Tare Weights Are Used (Densities used to compute air buoyancy correction) (Air buoyancy correction on the tare weights)

Date	_____	Temperature	_____
Balance	_____	Pressure	_____
Load	_____	Relative humidity	_____
$M_{sw} =$		$\rho_{sw} =$	$\rho_A =$
$S =$		$X_{nom} =$	$S_c =$
$C_s =$			$C_{s_c} =$
$\rho_s =$		$\rho_x =$	$\rho_{s_c} =$
$M_{t_s} =$		$M_{t_x} =$	$M_{t_{s_c}} =$
$\rho_{t_s} =$		$\rho_{t_x} =$	$\rho_{t_{s_c}} =$
Pooled short-term SD =		Balance SD =	

$$a = \begin{bmatrix} 0_1 & -0_2 & +0_4 & -0_3 \\ \hline & & 2 & \end{bmatrix} \begin{bmatrix} M_{sw} \left(1 - \frac{\rho_A}{\rho_{sw}}\right) \\ \hline 0_3 & -0_2 \end{bmatrix}$$

### DOUBLE SUBSTITUTIONS

$S - X = a_1$	$S - S_c = a_2$	$X - S_c = a_3$
$S + t_s$	$S + t_s$	$X + t_x$
$X + t_x$	$S_c + t_{s_c}$	$S_c + t_{s_c}$
$X + t_x + sw$	$S_c + t_{s_c} + sw$	$S_c + t_{s_c} + sw$
$S + t_s + sw$	$S + t_s + sw$	$X + t_x + sw$
$a_1 =$	$a_2 =$	$a_3 =$

$$\text{short-term S.D.} = 0.577 (a_1 - a_2 + a_3)$$

$$F\text{-test} = \frac{(\text{short-term S.D.})^2}{(\text{Pooled short-term S.D.})^2} < \text{value from F-table 9.5}$$

$$d_x = \frac{-2a_1 - a_2 + a_3}{3}$$

$$M_x = \frac{M_s \left(1 - \frac{\rho_A}{\rho_s}\right) + d_x + M_{t_s} \left(1 - \frac{\rho_A}{\rho_{t_s}}\right) - M_{t_x} \left(1 - \frac{\rho_A}{\rho_{t_x}}\right)}{1 - \frac{\rho_A}{\rho_x}}$$

$$AM_x \text{ vs } \rho_{\text{ref}} = \frac{M_x \left(1 - \frac{.0012}{\rho_x}\right)}{1 - \frac{.0012}{\rho_{\text{ref}}}}$$

where  $\rho_{\text{ref}}$  refers to the apparent  
mass reference density

$$d_{s_c} = \frac{-a_1 - 2a_2 - a_3}{3}$$

$$M_{s_c} = \frac{M_s \left(1 - \frac{\rho_A}{\rho_s}\right) + d_{s_c} + M_{t_s} \left(1 - \frac{\rho_A}{\rho_{t_s}}\right) - M_{t_{s_c}} \left(1 - \frac{\rho_A}{\rho_{t_{s_c}}}\right)}{1 - \frac{\rho_A}{\rho_{s_c}}}$$

# Example

## 3-1 Weighing Design When Tare Weights Are Used (Densities used to compute air buoyancy correction) (Air buoyancy correction on the tare weights)

Date	8/18/86	Temperature	21.7
Balance	CB 1000	Pressure	753.5 mm Hg
Load	1 kg	Relative humidity	45%
$M_{sw} = 50.086 \text{ mg}$		$\rho_{sw} = 8.41 \text{ g/cm}^3$	$\rho_A = 1.182 \text{ mg/cm}^3$
$S = 1. \text{ kg state standard}$		$X_{nom} = 1 \text{ kg}$ set 401	$S_c = 1.. \text{ kg state standard}$
$C'_s = -1.5 \text{ mg} \pm 0.098 \text{ mg}$			$C_{s_c} = 2.3 \text{ mg}$
$\rho_s = 8.0 \text{ g/cm}^3$		$\rho_x = 7.84 \text{ g/cm}^3$	$\rho_{s_c} = 8.0 \text{ g/cm}^3$
$M_{t_s} = 0$		$M_{t_x} = 0$	$M_{t_{s_c}} = 0$
$\rho_{t_s} =$		$\rho_{t_x} =$	$\rho_{t_{s_c}} =$

Pooled short-term SD = 0.023 mg      Balance SD = 0.10 mg

$$a = \left[ \frac{0_1 - 0_2 + 0_4 - 0_3}{2} \right] \left[ \frac{M_{sw} \left( 1 - \frac{\rho_A}{\rho_{sw}} \right)}{0_3 - 0_2} \right]$$

DOUBLE SUBSTITUTIONS

$S - X = a_1$		$S - S_c = a_2$		$X - S_c = a_3$	
$S + t_s \quad 10.0$		$S + t_s \quad 10.3$		$X + t_x \quad 15.5$	
$X + t_x \quad 15.3$		$S_c + t_{s_c} \quad 14.0$		$S_c + t_{s_c} \quad 14.1$	
$X + t_x + sw \quad 65.3$		$S_c + t_{s_c} + sw \quad 64.1$		$S_c + t_{s_c} + sw \quad 64.0$	
$S + t_s + sw \quad 60.1$		$S + t_s + sw \quad 60.4$		$X + t_x + sw \quad 65.6$	
$a_1 = -5.25829$		$a_2 = -3.69845$		$a_3 = 1.50538$	

short-term S.D. = 0.577 ( $a_1 - a_2 + a_3$ ) = -0.03142

F-test =  $\frac{(\text{Short-term S.D.})^2}{(\text{Pooled short-term S.D.})^2} < \text{value from F-table 9.5}$

F-test =  $\frac{(-.03142)^2}{(0.023)^2} = 1.87 < 7.31$       Passes F-test  
Degrees of freedom = 40



$$d_x = \frac{-2a_1 - a_2 + a_3}{3} = \frac{-2(-5.25829) - (-3.69845) + 1.50538}{3} = 5.24014 \text{ mg}$$

$$M_x = \frac{M_s \left(1 - \frac{\rho_A}{\rho_s}\right) + d_x + M_{t_s} \left(1 - \frac{\rho_A}{\rho_{t_s}}\right) - M_{t_x} \left(1 - \frac{\rho_A}{\rho_{t_x}}\right)}{\left(1 - \frac{\rho_A}{\rho_x}\right)}$$

$$M_x = \frac{999.9985g \left(1 - \frac{.001182}{8}\right) + .00524014g}{1 - \frac{.001182}{7.84}} = 1000.006757$$

$$AM_x \text{ vs } \rho_{\text{ref}} = \frac{M_x \left(1 - \frac{.0012}{\rho_x}\right) 1000.006757g \left(1 - \frac{.0012}{7.84}\right)}{1 - \frac{.0012}{\rho_{\text{ref}}}} = \frac{1000.006757g \left(1 - \frac{.0012}{7.84}\right)}{1 - \frac{.0012}{8}} = 1000.003695g$$

where  $\rho_{\text{ref}}$  refers to the apparent

mass reference density  $\rho_{\text{ref}} = 8.0 \text{ g/cm}^3$

Uncertainty =  $t_s + U_s$                       Degrees of freedom = 40

Uncertainty =  $3.199 (0.10 \text{ mg}) + 0.098 \text{ mg}$

Uncertainty =  $0.42 \text{ mg}$

$M_x = 1000.00676 \text{ g} \pm 0.42 \text{ mg}$

$C'_x = 6.76 \text{ mg} \pm 0.42 \text{ mg}$

$$d_{s_c} = \frac{-a_1 - 2a_2 - a_3}{3}$$

$$d_{s_c} = \frac{-(-5.25829) - 2(-3.69845) - 1.50538}{3} = 3.71660 \text{ mg}$$

$$M_{s_c} = \frac{M_s \left( 1 - \frac{\rho_A}{\rho_s} \right) + d_{s_c} + M_{t_s} \left( 1 - \frac{\rho_A}{\rho_{t_s}} \right) - M_{t_{s_c}} \left( 1 - \frac{\rho_A}{\rho_{t_{s_c}}} \right)}{\left( 1 - \frac{\rho_A}{\rho_{s_c}} \right)}$$

$$M_{s_c} = \frac{999.9985 \text{ g} \left( 1 - \frac{.001182}{8} \right) + .00371660 \text{ g}}{1 - \frac{.001182}{8}} = 1000.002217 \text{ g}$$

$$M_{s_c} = 1000.00222 \text{ g} \pm 0.42 \text{ mg}$$

$$G'_{s_c} = 2.22 \text{ mg} \pm 0.42 \text{ mg}$$



Recommended Standard Operations Procedure

for

Weighing by Transposition

1. Introduction

1.1. Purpose

This method eliminates possible error in comparison of weights when using an equal-arm balance, due to a small inequality in the length of the arms. While this procedure is written to describe the measurement of masses of weights for calibration purposes, it may be used for general mass determinations, whenever accurate weighing is required. (Note: This is a single - transposition procedure, hence does not eliminate the effects of linear drift).

1.2. Prerequisites

- 1.2.1. Verify that standard weights are available with valid certificates of calibration.
- 1.2.2. Verify that the balance to be used is in good working order. This may be verified by maintenance of a valid control chart. Otherwise, a series of trial measurements should be made to ascertain that the balance is functional and that its sensitivity is adequate for the requirements of the measurement.
- 1.2.3. Verify the ability of the technician to make precision weighings and that he/she has had training in this specific procedure.

2. Methodology

2.1. Scope, Precision, Accuracy

The measurement principle is applicable to weighing with any equal-arm balance. The range of weights that can be calibrated will depend upon the capacity of the balance used. The precision of intercomparison will depend upon the sensitivity of the balance, and the care exercised in making the required weighings. The accuracy will depend on the accuracy of calibration of the standard weights and the precision of intercomparison.

2.2. Summary

The masses of a standard and test weight are intercompared. The loads are then interchanged and a second intercomparison is made. A small calibrated weight is then added to one of the masses and a

third weighing is made to determine the sensitivity of the balance under the load conditions.

### 2.3. Apparatus/Equipment

- 2.3.1. Equal arm balance of capacity and sensitivity sufficient for the weights tested. The index scale of the balance is conveniently numbered 0 to 20 with 10 as the center division, although other numbering systems such as 0 to 200 are possible. The graduations should be numbered so that addition of a weight to the left arm will increase the balance readings. A system in which the center division is 0 is not recommended since the negative readings that result can cause observational and computational problems.
- 2.3.2. Standard weights with valid calibrations, traceable to NBS.
- 2.3.3. Small calibrated weights (usually decimal fractions) to be used as tare weights.
- 2.3.4. Equipment capable of loading and unloading weights on balance without damage to either (especially important in the case of large weights).

### 2.4. Symbols

The following symbols are used in this procedure.

- S = standard weight
- X = weight calibrated
- T = counterweight
- t = small calibrated. A subscript s or x is used to indicate the larger weight with which it is associated.
- sw = small calibrated weight used to evaluate the sensitivity of the balance.
- M = the mass of a specific weight. Subscripts s, x, t, sw, are used to identify the weight.
- AM = the apparent mass of a specific weight. Subscripts s, x, t, sw together with numerical sub-subscripts as necessary are used to identify the weight.



## 2.5. Procedure

2.5.1. The following weighing sequences may be used:

### Option A

Measurement No.	Left Arm	Right Arm	Observation
1	$X + t_{x_1}$	$S + t_{s_1}$	$O_1$
2	$S + t_{s_2}$	$X + t_{x_2}$	$O_2$
3	$S + t_{s_2} + sw$	$X + t_{x_2}$	$O_3$

### Option B

Measurement No.	Left Arm	Right Arm	Observation
1	$S + t_{s_1}$	$X + t_{x_1}$	$O_1$
2	$X + t_{x_2}$	$S + t_{s_2}$	$O_2$
3	$X + t_{x_2} + sw$	$S + t_{s_2}$	$O_3$

## 2.5.2. Preliminary Procedures

Conduct preliminary measurements (without recording data) to determine the values for  $t_s$  and  $t_x$  as described in 2.5.3. This will serve to warm up the balance and to facilitate the measurements. In a series of calibrations, this step may be minimized or eliminated after the first series of measurements. Depending on the corrections for S and X, weights  $t_s$  and  $t_x$  may be unnecessary in some cases.

## 2.5.3. Measurement Procedure (Option A)

All observations should be recorded on suitable data sheets such as those in the Appendix. Record the laboratory ambient temperature, barometric pressure, and relative humidity.

2.5.3.1. Observation 1. Place the test weight(s) on the left arm and the standard(s) on the right arm. Add  $t_x$ , and/or  $t_s$  (small calibrated weights) as necessary to obtain an approximate balance. Read and record all data and the sum of the turning points  $O_1$  (see GMP No. 1).

2.5.3.2. Observation 2. Place the standard weight(s) on the left arm and the test weight(s) on the right arm with appropriate small weights, as was done in Observation 1. The tare weights carried with S and X may have to be changed so that  $O_2$  is within one division of  $O_1$  on a 0 to 20 division graduated

scale. Read and record all data and the sum of the turning points,  $O_2$ .

- 2.5.3.3. Observation 3. Without disturbing weights already in place from Observation 2, add a small calibrated sensitivity weight (sw) to the left arm, sufficient to displace the turning points 3 to 5 divisions on a 20 division graduated scale. Record the sum of the turning points,  $O_3$ .

#### 2.5.4. Measurement Procedure (Option B)

Conduct 3 measurements as in 2.5.3.1, 2.5.3.2, and 2.5.3.3 using the weighing schedule Option B.

### 3. Calculations

#### 3.1. No Air Buoyancy Correction

The following equations may be used to calculate the corrections required for the test weight(s). The calibrated apparent mass values for  $t_s$ ,  $t_x$ , and sw must be used.  $C_s$  is the apparent mass correction for the standard weight(s), S. The symbols  $N_s$  and  $N_x$  refer to the nominal values of S and X, respectively.

##### Option A

$$C_x = C_s + \frac{AM_{t_{s1}} + AM_{t_{s2}} - AM_{t_{x1}} - AM_{t_{x2}}}{2} + \frac{O_1 - O_2}{2} \cdot \frac{AM_{sw}}{O_3 - O_2} + N_s - N_x$$

##### Option B

$$C_x = C_s + \frac{AM_{t_{s1}} + AM_{t_{s2}} - AM_{t_{x1}} - AM_{t_{x2}}}{2} + \frac{O_2 - O_1}{2} \cdot \frac{AM_{sw}}{O_3 - O_2} + N_s - N_x$$

#### 3.2. Air Buoyancy Correction

- 3.2.1. Calculate the air density,  $\rho_A$ , as described in Section 8 of the Appendix to SOP No. 2 or obtain from Table 9.9.

- 3.2.2. Calculate the mass,  $M_x$ , of the test weight X, using the mass of the standard weight(s), the tare weights, and the sensitivity weights according to the optional sequence used. It is assumed that the densities of the combination of tare weights used do not differ significantly in the weighing design.

Symbols used:  $\rho_A$  = air density  
 $M_i$  = mass for weight i  
 $\rho_i$  = density for weight i

### 3.2.2.1. Optional Sequence A

$$M_X = \frac{M_S \left(1 - \frac{\rho_A}{\rho_S}\right) + \frac{O_1 - O_2}{2} \cdot \frac{M_{SW} \left(1 - \frac{\rho_A}{\rho_{SW}}\right)}{O_3 - O_2} + \frac{M_{t_{S1}} + M_{t_{S2}}}{2} \left(1 - \frac{\rho_A}{\rho_{t_S}}\right) - \frac{M_{t_{X1}} + M_{t_{X2}}}{2} \left(1 - \frac{\rho_A}{\rho_{t_X}}\right)}{1 - \frac{\rho_A}{\rho_X}}$$

### 3.2.2.2. Optional Sequence B

$$M_X = \frac{M_S \left(1 - \frac{\rho_A}{\rho_S}\right) + \frac{O_2 - O_1}{2} \cdot \frac{M_{SW} \left(1 - \frac{\rho_A}{\rho_{SW}}\right)}{O_3 - O_2} + \frac{M_{t_{S1}} + M_{t_{S2}}}{2} \left(1 - \frac{\rho_A}{\rho_{t_S}}\right) - \frac{M_{t_{X1}} + M_{t_{X2}}}{2} \left(1 - \frac{\rho_A}{\rho_{t_X}}\right)}{1 - \frac{\rho_A}{\rho_X}}$$

### 3.2.2.3. Calculate the mass correction, $C'_X$ , as follows:

$$C'_X = M_X - N_X$$

when  $N_X$  is the nominal value for X

### 3.2.2.4. Calculate the apparent mass of X versus the desired reference density of 8.0 g/cm<sup>3</sup> or brass. It is recommended that the apparent mass versus 8.0 g/cm<sup>3</sup> be reported unless otherwise requested. The density of X, $\rho_X$ , must be in g/cm<sup>3</sup>.

#### 3.2.2.4.1. Apparent mass versus 8.0 g/cm<sup>3</sup>

$$AM_X \text{ vs. } 8.0 = M_X \frac{(1 - \frac{.0012}{\rho_X})}{0.999850}$$

#### 3.2.2.4.2. Apparent mass versus brass

$$AM_X \text{ vs. brass} = M_X \frac{(1 - \frac{.0012}{\rho_X})}{0.999857}$$

#### 4. Assignment of Uncertainty

The limits of uncertainty,  $U$ , include estimates of the uncertainty of the mass standards,  $U_s$ , plus the uncertainty of measurement,  $U_m$ , at the 99.73% level of confidence. The latter is estimated by

$$ts$$

where  $s$  is the standard deviation of measurement and  $t$  is obtained from Table 9.3.

Then 
$$U = \pm [U_s + ts]$$

##### 4.1. Precision of Measurement Known from Control Chart Performance (SOP No. 9)

The value for  $s$  is obtained from the control chart data for transposition measurements. Statistical control will need to be verified by measurement of at least one check standard while the above measurements are in progress.

Use the value of  $t$  (corresponding to a probability level of 99.73%) from Table 9.3 appropriate for the number of degrees of freedom,  $\nu$ , on which the control limits of the control chart are based.

##### 4.2. Precision Estimated from a Series of Measurements

Measure a stable test object at least 7 times, no two measurements of which may be made on a single day. Calculate the mean and a standard deviation in the conventional manner. The latter is the value of  $s$  that is to be substituted in the equation given in Section 4. In this case select the value for  $t$  from Table 9.3 based on the number of degrees of freedom involved in computing  $s$ .

Note: Repetitive measurements made on the same day evaluate only the short-term standard deviation.

#### 5. Report

##### 5.1. Report results as described in SOP No. 1, Preparation of Calibration/Test Reports

# Appendix

## Transposition Data Sheet for Equal-Arm Balance (Option A)

Test No.: \_\_\_\_\_ Sheet No.: \_\_\_\_\_ Date: \_\_\_\_\_

Item Identification: \_\_\_\_\_ Balance: \_\_\_\_\_

Standard Identification: \_\_\_\_\_ Observer: \_\_\_\_\_

Temperature: \_\_\_\_\_ Pressure: \_\_\_\_\_ Rel. Hum.: \_\_\_\_\_

$C_s(C'_s) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_  $AM_{sw}(M_{sw}) =$  \_\_\_\_\_

$\rho_s =$  \_\_\_\_\_  $\rho_x =$  \_\_\_\_\_  $\rho_{sw} =$  \_\_\_\_\_

Time \_\_\_\_\_ Balance standard deviation = \_\_\_\_\_

Measure- ment No.	Weights		Turning Points		Sum
	Left Arm	Right Arm	Low	High	
1	$X + t_{x1}$ $t_{x1} =$	$S + t_{s1}$ $t_{s1} =$			$O_1 =$
2	$S + t_{s2}$ $t_{s2} =$	$X + t_{x2}$ $t_{x2} =$			$O_2 =$
3	$S + t_{s2} + sw$	$X + t_{x2}$			$O_3 =$

Time \_\_\_\_\_

$AM_{t_{s1}}(M_{t_{s1}}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_

$AM_{t_{x1}}(M_{t_{x1}}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_

$AM_{t_{s2}}(M_{t_{s2}}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_

$AM_{t_{x2}}(M_{t_{x2}}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_

$\rho_{t_s} =$  \_\_\_\_\_

$\rho_{t_x} =$  \_\_\_\_\_



# Appendix

## Transposition Data Sheet for Equal-Arm Balance (Option B)

Test No.: \_\_\_\_\_ Sheet No.: \_\_\_\_\_ Date: \_\_\_\_\_

Item Identification: \_\_\_\_\_ Balance: \_\_\_\_\_

Standard Identification: \_\_\_\_\_ Observer: \_\_\_\_\_

Temperature: \_\_\_\_\_ Pressure: \_\_\_\_\_ Rel. Hum.: \_\_\_\_\_

$C_S(C'_S) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_  $AM_{sw}(M_{sw}) =$  \_\_\_\_\_

$\rho_S =$  \_\_\_\_\_  $\rho_X =$  \_\_\_\_\_  $\rho_{sw} =$  \_\_\_\_\_

Time \_\_\_\_\_ Balance standard deviation = \_\_\_\_\_

Measure- ment No.	Weights		Turning Points		Sum
	Left Arm	Right Arm	Low	High	
1	$S + t_{s1}$ $t_{s1} =$	$X + t_{x1}$ $t_{x1} =$			$O_1 =$
2	$X + t_{x2}$ $t_{x2} =$	$S + t_{s2}$ $t_{s2} =$			$O_2 =$
3	$X + t_{x2} + sw$	$S + t_{s2}$			$O_3 =$

Time \_\_\_\_\_

$AM_{t_{s1}}(M_{t_{s1}}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_  $AM_{t_{x1}}(M_{t_{x1}}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_

$AM_{t_{s2}}(M_{t_{s2}}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_  $AM_{t_{x2}}(M_{t_{x2}}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_

$\rho_{t_s} =$  \_\_\_\_\_  $\rho_{t_x} =$  \_\_\_\_\_

# Appendix

## Transposition Data Sheet for Equal-Arm Balance (Option A)

Test No.: 135 Sheet No.: 1 Date: 8/26/86

Item Identification: 1000 lb No. 4211 Balance: Russell

Standard Identification: #11 and #22 Observer: HO

Temperature: 23.5°C Pressure: 745.8 mm Hg Rel. Hum.: 50%

$C_s(C'_s) =$  0.0047 lb  $\pm$  0.0030 lb  $AM_{sw}(M_{sw}) =$  0.01 lb

$\rho_s =$  8.0 g/cm<sup>3</sup>  $\rho_x =$  7.0 g/cm<sup>3</sup>  $\rho_{sw} =$  7.8 g/cm<sup>3</sup>

Time 1:15 p Balance standard deviation = 0.0018 lb

Measure- ment No.	Weights		Turning Points		Sum
	Left Arm	Right Arm	Low	High	
1	$X + t_{x1}$	$S + t_{s1}$	4.0	<del>16.3</del>	$O_1 = 20.35$
	$t_{x1} = 3.6171b$	$t_{s1} = 3.6251b$	4.1	16.3	
2	$S + t_{s2}$	$X + t_{x2}$	<del>5.6</del>	<del>14.1</del>	$O_2 = 19.75$
	$t_{s2} = $	$t_{x2} = 3.6121b$	5.7	14.1	
3	$S + t_{s2} + sw$	$X + t_{x2}$	<del>8.4</del>	<del>16.0</del>	$O_3 = 24.3$
			8.4	15.9	

Time 1:40 p

$AM_{t_{s1}}(\overline{M_{t_{s1}}}) =$  3.625  $\pm$  .0000751b  $AM_{t_{x1}}(\overline{M_{t_{x1}}}) =$  3.617  $\pm$  negligible

$AM_{t_{s2}}(\overline{M_{t_{s2}}}) =$  3.625  $\pm$  .0000751b  $AM_{t_{x2}}(\overline{M_{t_{x2}}}) =$  3.612  $\pm$  negligible

$\rho_{t_s} =$  8.0 g/cm<sup>3</sup>  $\rho_{t_x} =$  7.8 g/cm<sup>3</sup>

$$C_x = C_s + \frac{AM_{ts1} + AM_{ts2} - AM_{tx1} - AM_{tx2}}{2} + \frac{O_1 - O_2}{2} \cdot \left( \frac{AM_{sw}}{O_3 - O_2} \right)$$

$$C_x = .0047 + \frac{3.625 + 3.625 - 3.617 - 3.612}{2} + \frac{20.35 - 19.75}{2} \cdot \frac{0.01}{24.3 - 19.75}$$

$$C_x = .0047 + .0105 + .000659$$

$$C_x = 0.015859 \text{ lb}$$

$$\text{Uncertainty} = t_s + U_s \quad \text{Degrees of freedom in } s = 20$$

$$\text{Uncertainty} = 3.422 (0.0018) + (.0030 + .000075)$$

$$\text{Uncertainty} = 0.0092346 \text{ lb}$$

$$C_x = 0.0159 \pm 0.0092 \text{ lb}$$

Recommended Standard Operations Procedure  
for

Weighing by Single Substitution

Using a Single Pan Mechanical Balance,

a Full Electronic Balance, or a

Balance with Digital Indications and Built-In Weights

1. Introduction

1.1. Purpose

The single substitution procedure is one in which a standard and an unknown weight are intercompared once to determine the difference between the two weights. Errors in any built-in weights or in the balance indications are eliminated by using the balance only as a comparator and by calibrating the balance indications over the range of use for the measurement. This procedure is suitable for calibration when moderate accuracy is required.

1.2. Prerequisites

1.2.1. Mass standards must be available with calibration certificates traceable to NBS.

1.2.2. The balance used must be in good operating condition as verified by a valid control chart or preliminary experiments to ascertain its performance quality.

1.2.3. The operator must be experienced in precision weighing techniques.

2. Methodology

2.1. Scope, Precision, Accuracy

This method is applicable to all weighings utilizing a single pan mechanical balance, a full electronic balance, or a balance that combines digital indications with the use of built-in weights. The precision will depend upon the sensitivity of the balance and the care exercised to make the required weighings. The accuracy will depend upon the accuracy of the calibration of the standard weights and the precision of the intercomparison.

## 2.2. Summary

The balance is adjusted if necessary, to obtain balance indications for all measurements that will be within the range of the optical scale or digital indications of the balance without changing the dial settings for the built-in weights, if present. The standard and the test weight are each weighed. A small calibrated weight, called a sensitivity weight, is added to the test weight and these are weighed.

The single substitution procedure is the same for all of the balances mentioned above but the adjustment of the balance to prepare for the intercomparison and the selection of the sensitivity weight to be used varies slightly depending upon the balance used. When steps specific to a particular balance are required, they are given in subsections of the procedure identified by a, b, and c along with the balance type.

## 2.3. Apparatus/Equipment Required

- 2.3.1. Precision balance with sufficient capacity and sensitivity for the calibrations planned.
- 2.3.2. Standard weights with valid calibrations traceable to NBS. The sensitivity weight may be a calibrated secondary standard. The use of secondary standards as sensitivity weights reduces wear on the primary mass standards.
- 2.3.3. Small standard weights with valid calibrations traceable to NBS to be used as tare weights.
- 2.3.4. Uncalibrated weights to be used to adjust the balance to the desired reading range.
- 2.3.5. Forceps to handle the weights or gloves to be worn if the weights are moved by hand.
- 2.3.6. Stop watch or other timing device to observe the time of each measurement.
- 2.3.7. \*Thermometer accurate to 0.10 °C to determine air temperature.
- 2.3.8. \*Barometer accurate to 0.5 mm of mercury (66.5 Pa) to determine air pressure.
- 2.3.9. \*Hygrometer accurate to 10 percent to determine relative humidity.

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\*The thermometer, barometer, and hygrometer are used to determine the air density at the time of the measurement. The air density is used to make an air bouyancy correction. The accuracies specified are recommended for high precision calibration. Less accurate equipment can be used with only a small degradation in the overall accuracy of the measurement.



## 2.4. Symbols

The following symbols are used in this procedure.

- S = standard weight
- X = weight calibrated
- T = counterweight
- t = small calibrated. A subscript s or x is used to indicate the larger weight with which it is associated.
- sw = small calibrated weight used to evaluate the sensitivity of the balance.
- M = the mass of a specific weight. Subscripts s, x, t, sw, are used to identify the weight.
- AM = the apparent mass of a specific weight. Subscripts s, x, t, sw together with numerical sub-subscripts as necessary are used to identify the weight.

## 2.5. Procedure

### 2.5.1. Preliminary Procedure

Place the test weight and standards in the balance chamber or near the balance overnight to permit the weights and the balance to attain thermal equilibrium. Conduct preliminary measurements to obtain an approximate value for the difference between the standard and the unknown, to determine where the readings occur on the balance, to determine if tare weights are required, to determine the sensitivity weight that must be used, and to determine the time interval required for the balance indication to stabilize.

Tare weights are rarely needed for high precision mass standards. If tare weights are required, carry tare weights,  $t_s$  and  $t_x$ , with the standard and the unknown, S and X, respectively. The standard and its tare weight,  $S + t_s$ , should be "nearly the same mass" as the unknown with its tare weight,  $X + t_x$ . "Nearly the same mass" depends upon the balance used. Select  $t_s$  and  $t_x$  such that the difference in mass between  $S + t_s$  and  $X + t_x$  is:

- a. Single-pan mechanical balance - less than  $\frac{1}{4}$  the range of the optical scale.
- b. Full electronic balance - less than 0.05 percent of the balance capacity.
- c. Combination balance - less than  $\frac{1}{4}$  the range of the digital indications.

Select a sensitivity weight that is:

- a. Single-pan mechanical balance - between  $\frac{1}{4}$  and  $\frac{1}{2}$  the range of the optical scale, inclusive, and at least three times the mass difference between  $X+t_x$  and  $S+t_s$ .

- b. Full electronic balance - at least 10 to 20 times the mass difference between  $X+t_x$  and  $S+t_s$  but not exceeding 1 percent of the balance capacity.
- c. Combination balance - between  $\frac{1}{4}$  and  $\frac{1}{2}$  the range of the digital indications, inclusive, and at least three times the mass difference between  $X+t_x$  and  $S+t_s$ .

Adjust the single pan balance or the combination balance so the first two readings of the single substitution fall in the first quarter of the optical scale or digital indications. The zero adjustment and tare adjustment may be used. Small weights may be placed on the balance pan to reach the desired reading range. These weights remain on the pan throughout the double substitution. Once the balance has been adjusted to the desired position, neither the balance dials, the zero and tare adjustments, nor the small weights placed on the balance pan are to be changed during the measurement.

If the balance is equipped with a pan arrestment mechanism, arrest the pan between each observation.

#### 2.5.2. Measurement Procedure, Option A

Measurement No.	Weights on Pan	Observation
1	$S + t_s$	$O_1$
2	$X + t_x$	$O_2$
3	$X + t_x + sw$	$O_3$

All observations should be recorded on a suitable data sheets such as those in the Appendix. Record the laboratory ambient temperature, barometric pressure, and relative humidity.

- 2.5.2.1. Measurement No. 1. Place the standard weight(s),  $S$ , along with  $t_s$  on the balance pan. If equipped with a pan arrestment mechanism, release the balance pan. When the pan is released, start the stop-watch and record observation  $O_1$  once the balance indication has stabilized.
- 2.5.2.2. Measurement No. 2. Remove weight(s)  $S$  and  $t_s$  and replace with test weight  $X$  and its tare weight,  $t_x$ . Release the pan, time the interval until the balance indications have stabilized, and record observation  $O_2$ .
- 2.5.2.3. Measurement No. 3. Add the sensitivity weight,  $sw$ , to the weights of observation 2. Release the pan, time the interval, and record observation  $O_3$ .

2.5.2.4. If repeated single substitutions are performed, the time intervals between successive trials should not differ from one another by more than  $\pm 20\%$ . If this difference is exceeded, reject the data and take a new series of measurements that will so agree.

### 2.5.3. Measurement Procedure, Option B

Measurement No.	Weights on Pan	Observation
1	$X + t_x$	$O_1$
2	$S + t_s$	$O_2$
3	$S + t_s + sw$	$O_3$

Measurements for Option B are made as described in Option A except that  $X$ ,  $S$ ,  $t_x$ , and  $t_s$  are interchanged appropriately.

## 3. Calculations

3.1. No air buoyancy correction. Calculate the apparent mass correction,  $C_x$ , for the test weight as follows, according to the optional sequence used. In each case, the apparent mass corrections for the standard weight(s) and the tare weights are included.

Symbols used:  $AM_i$  = apparent mass of weight  $i$   
 $N_s$  = nominal value of  $S$   
 $N_x$  = nominal value of  $X$

### 3.1.1. Optional Sequence A

$$C_x = C_s + AM_{t_s} - AM_{t_x} + \frac{(O_2 - O_1)(AM_{sw})}{(O_3 - O_2)} + N_s - N_x$$

### 3.1.2. Optional Sequence B

$$C_x = C_s + AM_{t_s} - AM_{t_x} + \frac{(O_1 - O_2)(AM_{sw})}{(O_3 - O_2)} + N_s - N_x$$

## 3.2. Air Buoyancy Correction

3.2.1. Calculate the air density,  $\rho_A$ , as described in Section 8 of the Appendix to SOP No. 2 or obtain from Table 9.9.

3.2.2. Calculate the mass of the test weight  $X$ ,  $M_x$ , using the mass of the standard weight(s) and the tare weights and the sensitivity weight according to the optional sequence used.

Symbols used:  $\rho_A$  = air density  
 $M_i$  = mass of weight  $i$   
 $\rho_i$  = density of weight  $i$

### 3.2.2.1. Optional Sequence A

$$M_X = \frac{M_S \left(1 - \frac{\rho_A}{\rho_S}\right) + M_{t_S} \left(1 - \frac{\rho_A}{\rho_{t_S}}\right) - M_{t_X} \left(1 - \frac{\rho_A}{\rho_{t_X}}\right) + (O_2 - O_1) \left( \frac{M_{sw} \left(1 - \frac{\rho_A}{\rho_{sw}}\right)}{(O_3 - O_2)} \right)}{1 - \frac{\rho_A}{\rho_X}}$$

### 3.2.2.2. Optional Sequence B

$$M_X = \frac{M_S \left(1 - \frac{\rho_A}{\rho_S}\right) + M_{t_S} \left(1 - \frac{\rho_A}{\rho_{t_S}}\right) - M_{t_X} \left(1 - \frac{\rho_A}{\rho_{t_X}}\right) + (O_1 - O_2) \left( \frac{M_{sw} \left(1 - \frac{\rho_A}{\rho_{sw}}\right)}{O_3 - O_2} \right)}{1 - \frac{\rho_A}{\rho_X}}$$

3.2.3. Calculate the mass correction  $C'_X$ , for X as follows

$$C'_X = M_X - N_X$$

where  $N_X$  is the nominal mass of X.

3.3. Calculate the apparent mass of X versus the desired reference density of 8.0 g/cm<sup>3</sup> or brass. It is recommended that the apparent mass versus 8.0 g/cm<sup>3</sup> be reported unless otherwise requested. The density of X,  $\rho_X$ , must be in g/cm<sup>3</sup>.

3.3.1. Apparent mass versus 8.0 g/cm<sup>3</sup>

$$AM_X \text{ vs. } 8.0 = M_X \frac{1 - \frac{.0012}{\rho_X}}{0.999850}$$

3.3.2. Apparent mass versus brass

$$AM_X \text{ vs. brass} = M_X \frac{1 - \frac{.0012}{\rho_X}}{0.999857}$$

#### 4. Assignment of Uncertainty

The limits of uncertainty,  $U$ , include estimates of the uncertainty of the mass standards used,  $U_s$ , plus the uncertainty of measurement,  $U_m$ , at the 99.73% level of confidence. The latter is estimated by

$$ts$$

where  $s$  is the standard deviation of measurement and  $t$  is obtained from Table 9.3.

Then 
$$U = \pm [U_s + ts]$$

##### 4.1. Precision of Measurement Known from Control Chart Performance (SOP No. 9.)

The value for  $s$  is obtained from the control chart data. Statistical control will need to be verified by measurement of at least one check standard while the above measurements are in progress.

Use the value of  $t$  (corresponding to a probability level of 99.73%) from Table 9.3 appropriate for the number of degrees of freedom,  $\nu$ , on which the control limits of the control chart are based.

##### 4.2. Precision Estimated from Series of Measurements

Measure a stable test object at least 7 times, no two measurements of which may be made on a single day. Calculate the mean and the standard deviation in the conventional manner. The latter is the value of  $s$  that is used in Section 4.1. In this case select the value for  $t$  from Table 9.3 based on the number of degrees of freedom involved in computing  $s$ .

Note: Repetitive measurements made on the same day estimate only the short-term standard deviation.

#### 5. Report

##### 5.1. Report results as described in SOP No. 1, Preparation of Calibration/Test Reports.



# Appendix

## Single Substitution Data Sheet for Single Pan Balance (Option A)

Test No.: \_\_\_\_\_ Sheet No.: \_\_\_\_\_ Date: \_\_\_\_\_

Item Identification: \_\_\_\_\_ Balance: \_\_\_\_\_

Standard Identification: \_\_\_\_\_ Observer: \_\_\_\_\_

Temperature: \_\_\_\_\_ Pressure: \_\_\_\_\_ Rel. Hum.: \_\_\_\_\_

$C_S(C'_S) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_  $AM_{SW}(M_{SW}) =$  \_\_\_\_\_

$\rho_S =$  \_\_\_\_\_  $\rho_X =$  \_\_\_\_\_  $\rho_{SW} =$  \_\_\_\_\_

$AM_{t_S}(M_{t_S}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_  $AM_{t_X}(M_{t_X}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_

$\rho_{t_S} =$  \_\_\_\_\_  $\rho_{t_X} =$  \_\_\_\_\_

Time \_\_\_\_\_ Balance standard deviation = \_\_\_\_\_

Measurement No.	Weights	Observations
1	$S + t_S$	$O_1 =$
2	$X + t_X$	$O_2 =$
3	$X + t_X + sw$	$O_3 =$

Time \_\_\_\_\_

# Appendix

## Single Substitution Data Sheet for Single Pan Balance (Option B)

Test No.: \_\_\_\_\_ Sheet No.: \_\_\_\_\_ Date: \_\_\_\_\_

Item Identification: \_\_\_\_\_ Balance: \_\_\_\_\_

Standard Identification: \_\_\_\_\_ Observer: \_\_\_\_\_

Temperature: \_\_\_\_\_ Pressure: \_\_\_\_\_ Rel. Hum.: \_\_\_\_\_

$C_s(C'_s) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_  $AM_{sw}(M_{sw}) =$  \_\_\_\_\_

$\rho_s =$  \_\_\_\_\_  $\rho_x =$  \_\_\_\_\_  $\rho_{sw} =$  \_\_\_\_\_

$AM_{t_s}(M_{t_s}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_  $AM_{t_x}(M_{t_x}) =$  \_\_\_\_\_  $\pm$  \_\_\_\_\_

$\rho_{t_s} =$  \_\_\_\_\_  $\rho_{t_x} =$  \_\_\_\_\_

Time \_\_\_\_\_ Balance standard deviation = \_\_\_\_\_

Measurement No.	Weights	Observations
1	$X + t_x$	$O_1 =$
2	$S + t_s$	$O_2 =$
3	$S + t_s + sw$	$O_3 =$

Time \_\_\_\_\_

# Appendix

## Single Substitution Data Sheet for Single Pan Balance (Option A)

Test No.: 150 Sheet No.: 1 Date: 8/27/86

Item Identification: 50 g Set 7 Balance: CB 100

Standard Identification: 50-g state standard Observer: HO

Temperature: 22.6°C Pressure: 751.7 mm Hg Rel. Hum.: 50%

$C_S(\bar{C}'_S) =$  0.255 mg  $\pm$  0.033 mg  $AM_{sw}(\bar{M}_{sw}) =$  49.916 mg

$\rho_S =$  8.0 g/cm<sup>3</sup>  $\rho_X =$  7.95 g/cm<sup>3</sup>  $\rho_{sw} =$  8.5 g/cm<sup>3</sup>

$AM_{t_s}(\bar{M}_{t_s}) =$  0  $\pm$              $AM_{t_x}(\bar{M}_{t_x}) =$  0  $\pm$            

$\rho_{t_s} =$              $\rho_{t_x} =$            

Time 3:40 p Balance standard deviation = 0.018 mg

Measurement No.	Weights	Observations
1	$S + t_s$	$O_1 = 12.62$
2	$X + t_x$	$O_2 = 12.51$
3	$X + t_x + sw$	$O_3 = 62.37$

Time 3:45 p

$$C_x = C_s + AM_{t_s} - AM_{t_x} + \frac{(O_2 - O_1) AM_{sw}}{O_3 - O_2}$$

$$C_x = 0.255 \text{ mg} + 0 - 0 + \frac{(12.51 - 12.62) 49.916 \text{ mg}}{62.37 - 12.51}$$

$$C_x = 0.255 \text{ mg} + (-0.110124)$$

$$C_x = 0.144876$$

$$\text{Uncertainty} = t_s + U_s \quad \text{Degrees of freedom in } s = 60$$

$$\text{Uncertainty} = 3.130 (.018 \text{ mg}) + 0.022 \text{ mg}$$

$$\text{Uncertainty} = 0.07834 \text{ mg}$$

$$C_x = 0.145 \pm 0.078 \text{ mg}$$





SOP No. 8

Recommended Standard Operations Procedure

for

Tolerance Testing of Mass Standards

by

Modified Substitution

1. Introduction

1.1. This SOP describes procedures to be followed for determining whether or not mass standards are within the tolerances specified for a particular class of standards. The procedures described permit the metrologist to report that the weights under test were compared against a State standard with the results reported on the laboratory report form. The comparison is important because built-in weights of a balance do not represent laboratory standards unless they have been formally calibrated. If a State law requires that field weights be compared against the State standards, this procedure can be used to fulfill this requirement.

1.2. Prerequisites

1.2.1. Mass standards must be available with calibration certificates traceable to NBS.

1.2.2. The balance used must be in good operating condition.

1.2.3. The operator must be experienced in precision weighing techniques.

2. Methodology

2.1. Scope, Precision, Accuracy

This method is applicable to all mass tolerance testing. The precision of the tolerance determination will not be a factor, provided the uncertainty of the measurement is no more than one-tenth of the permissible tolerance of the mass standard tested.

2.2. Summary

The mass to be tested is compared with a calibrated standard by a modified substitution procedure. The comparison may be made using a single-pan, an equal-arm, or a full-electronic balance. The reference standard is placed on the balance to obtain a convenient reference point and a sensitivity test is conducted. The error (departure from nominal value) of the weight tested is determined by comparing its reading to the reading obtained for the reference

standard. A weight is considered to be within tolerance when its error does not exceed the tolerance established for the particular class of weight.

### 2.3. Apparatus/Equipment

2.3.1. Single-pan, equal arm, or full-electronic balance with sufficient capacity for the load tested and with readability equal to or less than one-tenth of the acceptable tolerance tested.

2.3.2. Mass standards calibrated with an accuracy of one-tenth or less than the tolerance tested. The calibration must be traceable to NBS.

2.3.3. Calibrated sensitivity weights.

2.3.4. Counterweights, T, uncalibrated, of approximately the same mass as the standard weights (for option C).

### 2.4. Procedure - Option A Use of Single-Pan Balance

2.4.1. Select a reference standard of the same nominal value as the weight under test. Place the standard on the balance pan. Adjust the optical scale reading (See GMP No. 4) to approximately midscale and record reading.

2.4.2. Add a sensitivity weight equal to approximately one-quarter full scale reading and record reading.

2.4.3. Calculate the value of a scale division. If it is within  $\pm 2\%$  of nominal value (usual case) the nominal value of a division can be used for tolerance testing.

2.4.4. Remove the sensitivity weight and adjust the optical scale to account for corrected value of standard used.

Example: Suppose that the nominal range of the optical scale is 100 mg and that the reference standard has a correction of -2.5 mg. The optical scale is adjusted to read 47.5 mg when the standard is on the pan. Under this condition, the reading 50.0 mg represents the nominal mass of the standard.

2.4.5. Remove Standard

2.4.6. Place weight to be tested on balance pan and read optical scale. The error in the weight is the amount by which the indication deviates from the mid-scale reading. If the weight indication is more than the mid-scale value, the weight is heavy by the indicated difference; if the indication is less than the mid-scale value, the weight is light. Record the error.

- 2.4.7. After several weights have been tested (no more than 10) put the standard on the balance pan and record the reading. The difference between this indication and the previous one for the standard indicates a balance drift. This drift will normally be very small. If the drift ever exceeds 25% of the tolerance applicable to the weights under test or affect a measurement result to the extent that a weight may be out of tolerance, the measurement should be repeated and more frequent checks of the standard should be made.
- 2.4.8. Readjust the optical scale at any time that a significant difference is observed when rechecking a standard.
- 2.5. Procedure - Option B  
Use of Full Electronic Balance
- 2.5.1. Select a reference standard of the same nominal value as the weight under test. Place the standard on the pan. If the standard is light, add small calibrated weights with the standard, equivalent to the correction for the standard. Record reading. If the weight is heavy do nothing at this point but follow instructions of 2.5.5. Zero the balance so errors can be read directly from the balance indications.
- 2.5.2. Add a calibrated sensitivity weight ( $sw \geq 2$  times the tolerance) and record reading. Verify whether the nominal scale division is within  $\pm 2\%$  of actual value. In this case, the nominal value of the scale division may be used.
- 2.5.3. Remove sensitivity weight and zero the balance.
- 2.5.4. Remove all weights from balance pan.
- 2.5.5. Place weight to be tested on the balance pan. If (and only if) the standard used is heavy, add small calibrated weights equal to the correction required for the standard and carry these along with every weight tested. Record balance reading which indicates, directly, the error of the weight tested. If the reading is positive, the weight is heavy by the indicated amount; if the reading is negative, the weight is light by that amount.
- 2.5.6. After several weights have been tested (no more than 10) recheck the zero as in 2.5.3 and record the reading. The difference between this indication and the previous one for the standard indicates a balance drift. This drift will normally be small. If the drift should ever exceed 25% of the tolerance applicable to the weight under test or affect a measurement result to the extent that a weight may be out of tolerance, the measurement should be repeated and more frequent checks of the standard should be made.
- 2.5.7. Readjust the zero at any time that a significant difference is observed when rechecking a standard.

2.6. Procedure - Option C  
Use of Equal Arm Balance

- 2.6.1. Select a reference standard of the same nominal value as the weight under test. Place the standard on the left balance pan together with small calibrated weights equal to the correction required for the standard, provided it is light. If (and only if) the standard is heavy, do nothing further at this point but follow instructions in 2.6.4. Add sufficient counter weights to the right pan to obtain a sum of turning points of approximately twice midscale value. If necessary, number the graduated scale such that adding a weights to the left pan will increase the balance reading. Record turning point as  $O_1$ .
- 2.6.2. Add an appropriate calibrated sensitivity weight to the left pan and record turning point as  $O_2$ . Calculate the sensitivity =  $AM_{sw}/(O_2 - O_1)$  where  $AM_{sw}$  is the apparent mass of the sensitivity weight.
- 2.6.3. Remove all weights from the left pan.
- 2.6.4. Place weight to be tested on the left pan. If the standard used in 2.6.1 was heavy, add small correction weights to the left pan, equivalent to the correction required for the standard. Add small calibrated weights as required to left or right pan to obtain an approximate balance and record turning point as  $O_3$ .
- 2.6.5. Calculate error of weight tested as follows.

- 2.6.5.1. If added weight, AW, is placed on left pan

$$\text{ERROR} = (O_3 - O_1) \frac{AM_{sw}}{O_2 - O_1} - AW$$

- 2.6.5.2. If added weight, AW, is placed on right pan

$$\text{ERROR} = (O_3 - O_1) \frac{AM_{sw}}{O_2 - O_1} + AW$$

- 2.6.6. After several weights have been tested (no more than 10), recheck turning point  $O_1$ , as described in 2.6.1. Only a small difference should be observed. If this is significant, use new value for  $O_1$ , in subsequent measurements. If this change should ever exceed 25% of the tolerance applicable or affect a measurement result to the extent that a weight may be out of tolerance, the measurement should be repeated and more frequent checks of the standard should be made.



## 2.7. Tolerance Evaluation

- 2.7.1. Compare error in weight tested with the tolerance for the class of weights to which it belongs. If error is numerically smaller than tolerance, the weight is considered to be within tolerance. If error is larger than permissible weight is considered to be outside of tolerance and appropriate action should be taken. It is recommended that weights with errors within 10% of the tolerance limit be adjusted.





## SOP No. 9

### Recommended Standard Operations Procedure

#### for

### Control Charts for Calibration of Mass Standards

#### 1. Introduction

1.1. The purpose of this SOP is to describe procedures for the development of control charts and their use for demonstrating attainment of statistical control of a mass calibration process.

#### 1.2. Prerequisites

The use of this SOP requires that appropriate apparatus, methodology, and standards are available, and that the laboratory thoroughly understands the basic principles of the measurement process used and has had sufficient experience to perform the necessary operations required for the measurements of concern.

#### 2. Summary

An appropriate check standard is weighed at established intervals and the results are plotted on a chart. The abscissa represents the sequence of measurements and the ordinate the measured values. A central line (Chapter 7.4) is drawn, indicating the mean of the measured values and control limits are indicated within which the results of measurements are expected to be randomly distributed, based on statistical considerations. The system is considered to be in statistical control when the above conditions are met. The statistical information on which the control limits are based can be used to calculate confidence limits for measurements made while the system is demonstrated to be in a state of statistical control.

#### 3. Procedure

##### 3.1. Definition of System Monitored

The system monitored is considered to consist of the balance that is used, the SOP, the laboratory environment, the operator, and any other sources that could contribute to the variance or bias of the measurement data. Any of the above that can be considered to be constant or negligible contributors to the variance may be consolidated and monitored by a single control chart. Any that cannot be so considered (for example: different balance; different SOP) may require individual control charts.

The question of variability of the precision of a balance, with load always needs to be considered. For single-pan constant-load balances this is not a question. For equal-arm balances, the precision is a function of load and a separate control chart is required (in

principle) for every load tested. Practically, this is not feasible, hence control charts utilizing reference standards at 2 or 3 intervals, appropriately spaced within the range of test are satisfactory.

### 3.2. Selection of Check Standards

3.2.1. The check standards used in high precision calibration measurement should be stable and will normally be primary standards. For lower order calibrations, the check standards should simulate the laboratory's primary standards to the extent feasible. They should be calibrated with an accuracy equal to or better than the potentiality of the process monitored. Check standards should be cared for in the same way as for primary standards to prevent their damage or deterioration. Lower order check standards should be recalibrated every two years.

#### 3.2.2. Recommended Check Standards for Typical Test Situations

<u>Balance</u>	<u>Range of Measurement</u>	<u>Check Standard(s)</u>
Ultramicrobalance		
3g- capacity	1 mg - 3 g	1 g
Microbalance		
20 - or 30 g capacity	1 mg - 20 or 30 g	100 mg and 10 g
100 or 160 g capacity	20 - 100 g	100 g
1 kg capacity	100 g - 1 kg	1000 g
3 kg capacity	1 kg - 3 kg	3 kg or 5 lb
30 kg capacity	3 kg - 30 kg	30 kg or 50 lb
Russell balance	60 lb - 2500 lb	1000 lb, and 2500 lb

### 3.3. Establishing Control Chart Parameters

3.3.1. The control chart parameters (see Chapter 7.4) consist of the central line which is the best estimate of the mean of measurements of the check standard, and control and warning limits that represent probabilistic limits for the distribution of results around the central line. These parameters are evaluated on the basis of a reasonable number of initial measurements and updated as additional measurement data are accumulated.

3.3.2. Make at least 7 (minimum number) and preferably at least 12 independent measurements of the check standard under the same conditions that will be used to make routine measurements. No two measurements should be made in the same day. This is

necessary to estimate the long-term standard deviation to the extent feasible.

Calculate the mean,  $\bar{x}$  and the estimate of the standard deviation,  $s$  in the conventional manner.

Establish the control chart parameters as follows:

Central Line =  $\bar{x}$   
Upper Control Limit =  $\bar{x} + 3s$   
Upper Warning Limit =  $\bar{x} + 2s$   
Lower Warning Limit =  $\bar{x} - 2s$   
Lower Control Limit =  $\bar{x} - 3s$

### 3.3.3. Upgrading Control Chart Parameters

Upgrade control chart parameters when a significant amount of additional data is available or when the previously determined parameters are no longer pertinent, due to changes in the system.

Note: Ordinarily, upgrading is merited when the amount of new data is equal to that already used to establish the parameters in use, or when at least 7 additional data points have been accumulated.

Calculate  $\bar{x}$  and  $s$  for the new set of data and examine for significant differences from the former using the  $t$  test and  $F$  test, respectively (See Chapter 8). If either is significantly different, determine the reason for it as possible and decide on whether corrective actions are required. If none are required, establish new parameters using the most recent data. If no significant differences are found, pool all data and calculate new control chart parameters based on all existing data.

### 3.4. Frequency of Measurement

The check standard should be measured with sufficient frequency to minimize the risk of loss of data during the period from last-known-in to first-known-out of control. It is good practice to measure the check standard at least one time during each period that a set of test measurements is made. For critical calibrations or those of highest accuracy, it is desirable to alternate measurements of test materials and check standards or incorporate the standard in the calibration design.

Whenever a long period of inactivity has occurred, it is good practice to make a series of measurements of the check standard and to plot the results on a control chart to demonstrate attainment of statistical control, prior to beginning a calibration program.

Control charts should be maintained in as close to real time as feasible to effectively monitor the measurement process.

#### 4. Use of Control Charts

##### 4.1. Monitoring a Measurement Process

Use the following criteria to interpret control chart results.

- 4.1.1. If plotted points are randomly distributed within the warning limits, decide that the system is in control.
- 4.1.2. If a plotted point is outside the warning limits but within the control limits, remeasure the check standard. The remeasured value must be within the warning limits to merit the decision of "in control". If outside of the warning limits, examine the data for arithmetic errors. If none are found, consider the system "out of control". Reject all data obtained since last "in-control" measurement and take corrective action. Accept no further data until the system is demonstrated to be in-control as indicated by at least two successive measurements of the check standard within the warning limits.

If a plotted point is outside of the control limits and arithmetically correct, the system is considered to be out of control. Data are rejected, corrective actions must be taken and re-attainment of statistical control demonstrated, as above, before data may be accepted.

##### 4.2 Transfer of Measurement Statistics

- 4.2.1. Absence of a significant difference between the central line and the accepted value for the check standard may be accepted as evidence of insignificant bias at the level of confidence of the statistical test used. As long as the system remains in control, this conclusion is valid.
- 4.2.2. The estimate of the standard deviation,  $s$ , used to establish the control limits may be used to calculate confidence intervals for all pertinent measurements made while the system is in control. The value of the test weight is said to be within the limits

$$\bar{y} \pm \frac{ts}{\sqrt{n}}$$

where  $n$  represents the number of measurements of the test weight,  $\bar{y}$  represents the mean of the measurements on the test weight and  $t$  is based on the number of degrees of freedom on which  $s$  is based and the confidence level required for the interval.

Note: The familiar 2s and 3s limits (zs limits) are conservative and may be used for all practical purposes instead of the ts limits. For  $n \geq 25$ ,  $t \rightarrow z$  and the difference between the two sets of limits is negligible.



Recommended Standard Operations Procedure

for

Calibration of Rigid Rules

1. Introduction

- 1.1. This SOP describes the procedure to be followed for the calibration of rigid rules by comparison with the 18-inch metal rule issued to each State as the State primary standard. The interval from 1 inch to 13 inches is calibrated. It is assumed that the rigid rules have the same coefficient of expansion.

The maximum length of rule that can be directly compared to the standard rule is 12 inches. However, longer lengths can be calibrated in segments of 12 inches with reference to the standard rule.

The accuracy of calibration of standard rules is within 0.0001 inch. The precision of intercomparison and the accuracy of the standard limit the uncertainty of calibration to 0.002 inch under optimum conditions.

1.2. Prerequisites

- 1.2.1. The calibration certificate of the primary standard must be available and referenced.
- 1.2.2. The technician should be expert in making length measurements and particularly in estimating scale differences, using a reticle eyepiece.

2. Methodology

2.1. Summary

A rigid rule (test rule) is calibrated by comparing intervals on it with intervals of the standard rule. A reticle eye piece (ocular micrometer) is used for this purpose. Test rules longer than the standard rule may be calibrated in segments, using the last calibrated graduation as the zero graduation mark for the succeeding segments.

2.2. Equipment/Apparatus

- 2.2.1. Length bench or similar flat surface on which to lay the test rule and the standard rule.
- 2.2.2. Standard rule with valid calibration certificate.

- 2.2.3. Microscope with reticle eye piece to compare graduations. The reticle should have graduation intervals of 0.002 inch or less.

## 2.3. Procedure

- 2.3.1. Both the test rule and the standard rule must be in temperature equilibrium with the length laboratory.
- 2.3.2. Place the test rule and the standard rule on the length bench or similar flat surface, parallel to one another with the reading edges adjacent. It is not necessary to have the "zero" graduations in coincidence.

Ordinarily, this will require that one rule reads left-to-right (increasing units) while the other reads right-to-left (decreasing units). In this case, for convenience of calculation, the standard rule is placed in the right-to-left position. A worksheet to reverse the calibration on the standard rule is provided at the end of this SOP. Place shims under one of the rules as necessary so that the upper surfaces of both are in the same plane.

- 2.3.3. Place the ocular micrometer on the bench in the vicinity of the zero position and align it so that its scale is parallel to the scales under test. (See GMP No. 2 on "Reading the Center of Graduations when Using a Microscope" for instructions on how to make readings.) All observations should be recorded on a suitable data sheet such as the one included in the Appendix.

- 2.3.3.1. Observe and record readings of left and right sides of the corresponding graduation of the standard rule. The average of these readings will give a value for A.

- 2.3.3.2. Observe and record readings of the left and right sides of the corresponding graduation of the test rule. The average of these readings will give a value for B. If the "zero" graduation is the end of the rule, only the reading for the end of the rule is taken.

- 2.3.4. Move the ocular microscope successively to each graduation that needs to be calibrated and record readings similarly as in 2.3.3.1 and 2.3.3.2. The readings of the standard will be recorded as values for C and readings for the test rule will be for D. Caution: Be sure that the rules are not disturbed by movement of the microscope.

2.3.5. Return ocular microscope to the zero graduation and repeat readings. Accept all previous data if present zero reading does not disagree with previous reading by more than 0.002 inch; otherwise, discard and repeat entire sequence of readings until a satisfactory set is obtained.

2.3.6. Make a second set of measurements as directed in 2.3.3., 2.3.4., and 2.3.5.

### 3. Calculations

3.1. Calculate the center of each graduation (the mean of the left and right edges of each graduation) at each interval measured. Record each value in the appropriate space of the data sheet (See the appendix of this SOP). The center of the "zero" graduation on the standard rule is A; for the test rule it is B. The centers of graduations for the standard and the test rule are recorded as C and D, respectively, for each interval measured. For each trial the values for A and B are used to compute the measured difference between the standard and the test rule for each interval measured in that trial.

3.2. Calculate the measured differences,  $d_1$  and  $d_2$ , for each trial for each interval measured and record on the data sheet. The measured difference  $d_i = A_i - B_i - C_i + D_i$  for each trial where  $i$  is the trial number 1 or 2.

3.3. Calculate the range,  $R$ , in the measured differences as  $R = |d_1 - d_2|$  for each interval and record on the data sheet.

3.4. Record the length of the standard,  $L_s$ , for each interval measured.

3.5. Calculate the length,  $L_x$ , of the test rule for each interval measured using the mean of the measured differences,  $L_x = (d_1 + d_2)/2 + L_s$ . Record the values for  $L_x$  on the data sheet.

### 4. Assignment of Uncertainty

4.1. The uncertainty,  $U_C$ , of a correction (the mean of two measurements) is given by the expression

$$U_C = \pm \left[ U_s + \frac{ts}{\sqrt{2}} \right]$$

where  $U_s$  is the uncertainty assigned for the correction for the standard and  $s$  represents the estimate of the standard deviation of a single measurement. The value for  $t$  corresponding to a probability level of 99.73% and the number of degrees of freedom on which  $s$  is based will be found in Table 9.3.

- 4.1.1. If control charts are not maintained, the standard deviation may be estimated from the average range,  $\bar{R}$ , of the ranges,  $R$  (absolute differences of the measured corrections) for the  $n$  intervals. Thus,

$$\bar{R} = \frac{\sum R}{n}$$

and

$$s = \bar{R}/d_2^*$$

The factor  $d_2^*$  is obtained by interpolation of the values in Table 9.1. The degrees of freedom,  $\nu$ , associated with  $s$  should be taken as one-half the degrees of freedom shown below the appropriate  $d_2^*$  factor in the same table.<sup>†</sup>

- 4.1.2. If control charts are maintained and the measurement system is in a state of statistical control, the value of  $s$  on which the control limits are based may be used in the equation of 4.1. The corresponding value for  $\nu$  is used when selecting the value for  $t$ .

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<sup>†</sup>Degrees of freedom are reduced approximately by the factor one-half because the same zero interval measurements are used for each interval estimate. Values should be rounded to the nearest whole number.

# Worksheet to Reverse the Calibration of the Rigid Rule Standard

To reverse the calibration on the State standard rigid rule, subtract the lengths for each calibrated interval from the length from the 1-inch to the 13-inch graduation.

Example:

Length from 13 to 12 = Length from 1 to 13 - length from 1 to 12

Length from 13 to 12 = 11.9998 - 10.9997 = 1.0001 inch

Notation:

$L_i$  = length of interval  $i$

$C_j$  = calibrated length of the standard from the 1-inch graduation to graduation  $j$

$C_{13}$  = calibrated length of the standard from the 1-inch graduation to the 13-inch graduation

Nominal Length Of Interval (inches)	Calculation To Be Performed	Length of Interval (inches)
1	$L_1 = C_{13} - C_{12} = \underline{\hspace{1cm}} - \underline{\hspace{1cm}} = \underline{\hspace{1cm}}$	
2	$L_2 = C_{13} - C_{11} = \underline{\hspace{1cm}} - \underline{\hspace{1cm}} = \underline{\hspace{1cm}}$	
3	$L_3 = C_{13} - C_{10} = \underline{\hspace{1cm}} - \underline{\hspace{1cm}} = \underline{\hspace{1cm}}$	
4	$L_4 = C_{13} - C_9 = \underline{\hspace{1cm}} - \underline{\hspace{1cm}} = \underline{\hspace{1cm}}$	
5	$L_5 = C_{13} - C_8 = \underline{\hspace{1cm}} - \underline{\hspace{1cm}} = \underline{\hspace{1cm}}$	
6	$L_6 = C_{13} - C_7 = \underline{\hspace{1cm}} - \underline{\hspace{1cm}} = \underline{\hspace{1cm}}$	
7	$L_7 = C_{13} - C_6 = \underline{\hspace{1cm}} - \underline{\hspace{1cm}} = \underline{\hspace{1cm}}$	
8	$L_8 = C_{13} - C_5 = \underline{\hspace{1cm}} - \underline{\hspace{1cm}} = \underline{\hspace{1cm}}$	
9	$L_9 = C_{13} - C_4 = \underline{\hspace{1cm}} - \underline{\hspace{1cm}} = \underline{\hspace{1cm}}$	
10	$L_{10} = C_{13} - C_3 = \underline{\hspace{1cm}} - \underline{\hspace{1cm}} = \underline{\hspace{1cm}}$	
11	$L_{11} = C_{13} - C_2 = \underline{\hspace{1cm}} - \underline{\hspace{1cm}} = \underline{\hspace{1cm}}$	
12	$L_{12} = C_{13} - C_1 = \underline{\hspace{1cm}} - \underline{\hspace{1cm}0} = \underline{\hspace{1cm}}$	



# Appendix

## Rigid Rule Calibration Data Sheet

Test No.: \_\_\_\_\_ Date: \_\_\_\_\_

Item Identification: \_\_\_\_\_

Standard Identification: \_\_\_\_\_

Temperature: \_\_\_\_\_ Observer: \_\_\_\_\_

Rule	Grad.	Trial 1			Trial 2		
		Left	Right	Center of Graduation	Left	Right	Center of Graduation
S	_____	_____	_____	A = _____	_____	_____	A = _____
X	_____	_____	_____	B = _____	_____	_____	B = _____
S <sub>1</sub>	_____	_____	_____	C <sub>1</sub> = _____	_____	_____	C <sub>1</sub> = _____
X <sub>1</sub>	_____	_____	_____	D <sub>1</sub> = _____	_____	_____	D <sub>1</sub> = _____
				d <sub>1</sub> = A - B - C + D = _____	d <sub>2</sub> = A - B - C + D = _____		
				Range =  d <sub>1</sub> - d <sub>2</sub>   = _____	L <sub>s</sub> = _____		
				Length of X, L <sub>x</sub> = (d <sub>1</sub> + d <sub>2</sub> ) / 2 + L <sub>s</sub> = _____			
S <sub>2</sub>	_____	_____	_____	C <sub>2</sub> = _____	_____	_____	C <sub>2</sub> = _____
X <sub>2</sub>	_____	_____	_____	D <sub>2</sub> = _____	_____	_____	D <sub>2</sub> = _____
				d <sub>1</sub> = A - B - C + D = _____	d <sub>2</sub> = A - B - C + D = _____		
				Range =  d <sub>1</sub> - d <sub>2</sub>   = _____	L <sub>s</sub> = _____		
				Length of X, L <sub>x</sub> = (d <sub>1</sub> + d <sub>2</sub> ) / 2 + L <sub>s</sub> = _____			
S <sub>3</sub>	_____	_____	_____	C <sub>3</sub> = _____	_____	_____	C <sub>3</sub> = _____
X <sub>3</sub>	_____	_____	_____	D <sub>3</sub> = _____	_____	_____	D <sub>3</sub> = _____
				d <sub>1</sub> = A - B - C + D = _____	d <sub>2</sub> = A - B - C + D = _____		
				Range =  d <sub>1</sub> - d <sub>2</sub>   = _____	L <sub>s</sub> = _____		
				Length of X, L <sub>x</sub> = (d <sub>1</sub> + d <sub>2</sub> ) / 2 + L <sub>s</sub> = _____			
S <sub>4</sub>	_____	_____	_____	C <sub>4</sub> = _____	_____	_____	C <sub>4</sub> = _____
X <sub>4</sub>	_____	_____	_____	D <sub>4</sub> = _____	_____	_____	D <sub>4</sub> = _____
				d <sub>1</sub> = A - B - C + D = _____	d <sub>2</sub> = A - B - C + D = _____		
				Range =  d <sub>1</sub> - d <sub>2</sub>   = _____	L <sub>s</sub> = _____		
				Length of X, L <sub>x</sub> = (d <sub>1</sub> + d <sub>2</sub> ) / 2 + L <sub>s</sub> = _____			
S <sub>5</sub>	_____	_____	_____	C <sub>5</sub> = _____	_____	_____	C <sub>5</sub> = _____
X <sub>5</sub>	_____	_____	_____	D <sub>5</sub> = _____	_____	_____	D <sub>5</sub> = _____
				d <sub>1</sub> = A - B - C + D = _____	d <sub>2</sub> = A - B - C + D = _____		
				Range =  d <sub>1</sub> - d <sub>2</sub>   = _____	L <sub>s</sub> = _____		
				Length of X, L <sub>x</sub> = (d <sub>1</sub> + d <sub>2</sub> ) / 2 + L <sub>s</sub> = _____			

# Worksheet to Reverse the Calibration of the Rigid Rule Standard

To reverse the calibration on the State standard rigid rule, subtract the lengths for each calibrated interval from the length from the 1-inch to the 13-inch graduation.

Example:

Length from 13 to 12 = Length from 1 to 13 - length from 1 to 12

Length from 13 to 12 = 11.9998 - 10.9997 = 1.0001 inch

Notation:

$L_i$  = length of interval  $i$

$C_j$  = calibrated length of the standard from the 1-inch graduation to graduation  $j$

$C_{13}$  = calibrated length of the standard from the 1-inch graduation to the 13-inch graduation

Nominal Length Of Interval (inches)	Calculation To Be Performed	Length of Interval (inches)
1	$L_1 = C_{13} - C_{12} = \underline{11.9998} - \underline{10.9995} = \underline{1.0003}$	
2	$L_2 = C_{13} - C_{11} = \underline{11.9998} - \underline{9.9984} = \underline{2.0014}$	
3	$L_3 = C_{13} - C_{10} = \underline{11.9998} - \underline{8.9989} = \underline{3.0009}$	
4	$L_4 = C_{13} - C_9 = \underline{11.9998} - \underline{7.9991} = \underline{4.0007}$	
5	$L_5 = C_{13} - C_8 = \underline{11.9998} - \underline{6.9992} = \underline{5.0006}$	
6	$L_6 = C_{13} - C_7 = \underline{11.9998} - \underline{5.9995} = \underline{6.0003}$	
7	$L_7 = C_{13} - C_6 = \underline{11.9998} - \underline{4.9995} = \underline{7.0003}$	
8	$L_8 = C_{13} - C_5 = \underline{11.9998} - \underline{3.9992} = \underline{8.0006}$	
9	$L_9 = C_{13} - C_4 = \underline{11.9998} - \underline{2.9993} = \underline{9.0005}$	
10	$L_{10} = C_{13} - C_3 = \underline{11.9998} - \underline{1.9995} = \underline{10.0003}$	
11	$L_{11} = C_{13} - C_2 = \underline{11.9998} - \underline{0.9998} = \underline{11.0000}$	
12	$L_{12} = C_{13} - C_1 = \underline{11.9998} - \underline{0} = \underline{11.9998}$	

## Appendix

## Rigid Rule Calibration Data Sheet

Test No.: 141 Date: 8/27/86  
 Item Identification: #365 6-inch rule  
 Standard Identification: #5451 State Standard  
 Temperature: 22.4 °C Observer: HO

Rule	Grad.	Trial 1			Trial 2		
		Left	Right	Center of Graduation	Left	Right	Center of Graduation
S	<u>13</u>	<u>.182</u>	<u>.186</u>	A = <u>.184</u>	<u>.208</u>	<u>.212</u>	A = <u>.210</u>
X	<u>0</u>	<u>.183</u>	<u>---</u>	B = <u>.183</u>	<u>.209</u>	<u>---</u>	B = <u>.209</u>
S <sub>1</sub>	<u>12</u>	<u>.208</u>	<u>.212</u>	C <sub>1</sub> = <u>.210</u>	<u>.195</u>	<u>.200</u>	C <sub>1</sub> = <u>.1975</u>
X <sub>1</sub>	<u>1</u>	<u>.209</u>	<u>.215</u>	D <sub>1</sub> = <u>.212</u>	<u>.197</u>	<u>.203</u>	D <sub>1</sub> = <u>.200</u>
d <sub>1</sub> = A-B-C+D = <u>0.003"</u>				d <sub>2</sub> = A-B-C+D = <u>0.0035"</u>			
Range =  d <sub>1</sub> - d <sub>2</sub>   = <u>0.005"</u>				L <sub>s</sub> = <u>1.00355"</u>			
Length of X, L <sub>x</sub> = (d <sub>1</sub> +d <sub>2</sub> )/2 + L <sub>s</sub> = <u>1.00355"</u>							
S <sub>2</sub>	<u>10</u>	<u>.194</u>	<u>.198</u>	C <sub>2</sub> = <u>.196</u>	<u>.216</u>	<u>.220</u>	C <sub>2</sub> = <u>.218</u>
X <sub>2</sub>	<u>3</u>	<u>.196</u>	<u>.202</u>	D <sub>2</sub> = <u>.199</u>	<u>.218</u>	<u>.224</u>	D <sub>2</sub> = <u>.221</u>
d <sub>1</sub> = A-B-C+D = <u>0.004"</u>				d <sub>2</sub> = A-B-C+D = <u>0.004"</u>			
Range =  d <sub>1</sub> - d <sub>2</sub>   = <u>0</u>				L <sub>s</sub> = <u>3.0009"</u>			
Length of X, L <sub>x</sub> = (d <sub>1</sub> +d <sub>2</sub> )/2 + L <sub>s</sub> = <u>3.0009"</u>							
S <sub>3</sub>	<u>7</u>	<u>.224</u>	<u>.229</u>	C <sub>3</sub> = <u>.2265</u>	<u>.192</u>	<u>.197</u>	C <sub>3</sub> = <u>.1945</u>
X <sub>3</sub>	<u>6</u>	<u>---</u>	<u>.231</u>	D <sub>3</sub> = <u>.231</u>	<u>---</u>	<u>.201</u>	D <sub>3</sub> = <u>.201</u>
d <sub>1</sub> = A-B-C+D = <u>0.0055"</u>				d <sub>2</sub> = A-B-C+D = <u>0.0075"</u>			
Range =  d <sub>1</sub> - d <sub>2</sub>   = <u>0.0020"</u>				L <sub>s</sub> = <u>6.0003"</u>			
Length of X, L <sub>x</sub> = (d <sub>1</sub> +d <sub>2</sub> )/2 + L <sub>s</sub> = <u>6.0068"</u>							
S <sub>4</sub>	<u>---</u>	<u>---</u>	<u>---</u>	C <sub>4</sub> = <u>---</u>	<u>---</u>	<u>---</u>	C <sub>4</sub> = <u>---</u>
X <sub>4</sub>	<u>---</u>	<u>---</u>	<u>---</u>	D <sub>4</sub> = <u>---</u>	<u>---</u>	<u>---</u>	D <sub>4</sub> = <u>---</u>
d <sub>1</sub> = A-B-C+D = <u>---</u>				d <sub>2</sub> = A-B-C+D = <u>---</u>			
Range =  d <sub>1</sub> - d <sub>2</sub>   = <u>---</u>				L <sub>s</sub> = <u>---</u>			
Length of X, L <sub>x</sub> = (d <sub>1</sub> +d <sub>2</sub> )/2 + L <sub>s</sub> = <u>---</u>							
S <sub>5</sub>	<u>---</u>	<u>---</u>	<u>---</u>	C <sub>5</sub> = <u>---</u>	<u>---</u>	<u>---</u>	C <sub>5</sub> = <u>---</u>
X <sub>5</sub>	<u>---</u>	<u>---</u>	<u>---</u>	D <sub>5</sub> = <u>---</u>	<u>---</u>	<u>---</u>	D <sub>5</sub> = <u>---</u>
d <sub>1</sub> = A-B-C+D = <u>---</u>				d <sub>2</sub> = A-B-C+D = <u>---</u>			
Range =  d <sub>1</sub> - d <sub>2</sub>   = <u>---</u>				L <sub>s</sub> = <u>---</u>			
Length of X, L <sub>x</sub> = (d <sub>1</sub> +d <sub>2</sub> )/2 + L <sub>s</sub> = <u>---</u>							

## SOP No. 11

### Recommended Standard Operations Procedure for

### Calibration of Metal Tapes

### Bench Method

## 1. Introduction

### 1.1. Purpose of Calibration

Metal tapes are used by contractors, surveyors, and others for building layouts, measurement of land areas, establishment of land boundaries, and similar purposes. Inaccuracies in such measurements can cause structural misalignments, boundary controversies, and other problems. The test method described here provides a procedure to calibrate such tapes to four decimal places in the case of measurements in inches, and to 6 decimal places for measurements expressed in units of meters. The calibrated length bench is used as the standard.

### 1.2. Prerequisites

- 1.2.1. Verify that the calibrations for the length bench scale and the thermometers are available and valid at the time of measurement.
- 1.2.2. Verify that the ocular microscope used in measuring differences in lengths is in good working order.
- 1.2.3. Verify that the technician has been trained in the use of this method. Note that the precision and accuracy attainable depend upon the care exercised in aligning the tape on the length bench, and the skill acquired in the use of an optical micrometer to measure scale differences.

## 2. Methodology

### 2.1. Summary

The tape to be calibrated is laid over the bench scale and sufficient tension is applied to insure that it lies flat on the bench. Differences between the graduation on the tape and that of the bench scale are measured using an ocular micrometer. The temperature of the tape is observed and corrections applied to the reference temperature of 20 °C, as required, using formulas or tables which are provided. All data are recorded, using the forms contained in Appendix A. Typical lengths to be tested on a 100-foot tape when using the 16-foot length bench are: every foot through 10 feet and then 15, 20, 30, 40, 45, 50, 60, 70, 75, 80, 90, and 100 foot lengths.

## 2.2. Apparatus

- 2.2.1. Two thermometers, capable of indicating temperatures in the range of 15 to 30 °C, and accurate to  $\pm 0.5$  °C.
- 2.2.2. A 16-foot, (5-meter) bench with attached calibrated length scale.
- 2.2.3. Optical microscope with ocular scale having dimensions spaced at 0.002 inch intervals.
- 2.2.4. Clamps and weights to apply an appropriate tension to the tape under calibration. (See "Tension Specifications", in Appendix C.3.

## 2.3. Procedure

- 2.3.1. Clean the tape by first wiping with a soft cloth, and then with a soft cloth saturated with alcohol to remove protective oil film. (See Appendix C, Section C.1.)
- 2.3.2. Lay two thermometers (see 2.2.1) on the bench at intervals of 1/3 and 2/3 of the length of the bench to determine its temperature at the time of the test.
- 2.3.3. Place the clamp at the zero end of the bench so that the zero mark on the tape is near the zero graduation on the length bench. Note: When the ring is part of the measuring tape, a special clamp-holder must be used. See Appendix C.5 for further information on this matter.
- 2.3.4. Lay the tape flat on the bench. The tape should extend well beyond the end roller of the bench to permit tension to be applied. Slide one end of a fabric strap onto another tape clamp. Fasten this tape clamp to the tape on the portion that extends below the end roller. Hang the tension weight from the bottom of the fabric strap. Check to see that the tape is lying straight on the bench and parallel to the bench scale. Adjust, if necessary. Apply tension using a weight of 10 pounds, unless other tension is desired. (See Appendix C.3.)
- 2.3.5. Adjust the tape clamp on the zero end of the bench so that the tape zero graduation coincides with the center of the zero graduation of the bench. Note: Some tapes are calibrated using the one foot mark as the reference point, rather than the zero graduation. In this case, align the 1-foot mark of the tape with the zero graduation mark of the bench and interpret all following instructions accordingly.
- 2.3.6. Check all alignments and coincidence of zero graduations before proceeding with calibration. Use the lateral adjustments at the left end of the bench to facilitate alignment. Caution! Take care that tape is not touched or



disturbed during the following sequence of measurements.  
Record all observations on a suitable data sheet such as the one included in the Appendix.

- 2.3.7. Record the temperatures indicated by the two thermometers.
- 2.3.8. Place the ocular microscope on the bench in the vicinity of the zero position and align it so that its scale is parallel to the tape under test. (See GMP No. 2 for instructions on how to make readings.)
  - 2.3.8.1. Observe readings of left and right sides of zero graduation of tape and record to the nearest .001 inch.
  - 2.3.8.2. Observe readings of left and right sides of zero graduation of bench and record to the nearest .001 inch.
- 2.3.9. Move ocular microscope successively to each graduation that needs to be calibrated and record readings similarly as in 2.3.8.1 and 2.3.8.2.
- 2.3.10. Return ocular microscope to the zero graduation and repeat readings to verify that the tape has not moved. Accept all previous data if present zero reading does not disagree with previous reading by more than 0.001 inch; otherwise, discard all previous data and repeat entire sequence of readings until a satisfactory set is obtained.
- 2.3.11. Remove the tension weights, move the tape, hang the tension weights back on the fabric strap, and realign the zero marks on the tape and the bench to coincidence.
- 2.3.12. Make a second set of measurements as directed in 2.3.8, 2.3.9, and 2.3.10.
- 2.3.13. Record the temperatures indicated by the two thermometers.
- 2.3.14. After all measurements are completed, apply a thin film of oil to the tape.

#### 2.4. Calculations

- 2.4.1. Calculate  $X_m$  and  $S_m$  for each series of measurements and for each scale interval, as follows:

$$X_m = \frac{(\text{right edge reading of tape} + \text{left edge reading of tape})}{2}$$

$$S_m = \frac{(\text{right edge reading of bench} + \text{left edge reading of bench})}{2}$$

Record at appropriate places on the data/worksheet.

2.4.2. Calculate  $d = X_m - S_m$  for each scale interval, and record on the data/worksheet.

2.4.3. Obtain the correction to the standard,  $C_s$ , from the calibration certificate for the length bench scale and tabulate. Note: If the 1-foot mark or other mark is used as the reference mark, the correction must be made, taking this into account.

2.4.4. Calculate the temperature correction,  $K$ , from the equation

$$K = L_n [(T-20) (a_s - a_x)]$$

where

$a_s$  = coefficient of linear expansion for the standard bench ( $10.63 \times 10^{-6}/^{\circ}\text{C}$ )

$a_x$  = coefficient of linear expansion for the tape ( $11.60 \times 10^{-6}/^{\circ}\text{C}$ )

$L_n$  = nominal length of tape interval under test

In this equation,  $T$  is the average of the four temperature readings described in Sections 2.3.7 and 2.3.11. In the case of steel tapes, the value for  $K$  may be obtained from the table in Appendix B.

2.4.5. Calculate a correction,  $C_x$ , for each trial and each scale interval.

$$C_x = d + C_s + K$$

2.4.6. Calculate and report the mean,  $\bar{C}_x$ , of the two corrections for each interval.

2.4.7. Compute the absolute difference between the two corrections for each interval and record as  $R$  on the worksheet.

2.4.8. Compute the length of the tape under the 10 lb. load as

$$L_o = L + \bar{C}_x$$

### 3. Determination of AE Value

3.1. Replace the tension weight (10 pound) used in 2.3.4 with a 20 pound weight (or with one 10 pounds heavier than that used in the original calibration).

3.2. Adjust the coincidence of the zero graduation as was done in 2.3.5.

3.3. Adjust the tape so that it lies flat and parallel to the bench scale.

3.4. Check the zero graduation coincidence and readjust if necessary.

- 3.5. Record zero readings as instructed in 2.8.
- 3.6. Record readings at the longest scale interval of the bench measured under the original tension, as instructed in 2.3.9.
- 3.7. Take a second series of measurements as described in 3.5 and 3.6.
- 3.8. Repeat appropriate calculations and compute the length of the tape under 20 lb. load as

$$L_1 = L + \bar{C}_x$$

- 3.9. Compute the AE value using the equation

$$AE = \frac{Q_1 - Q_0}{L_1 - L_0} L_n$$

where

AE = cross sectional area times Young's modulus of elasticity  
 $Q_0$  = the lower load applied to the tape (e.g., 10 lb.)  
 $Q_1$  = the higher load applied to the tape (e.g., 20 lb.)  
 $L_0$  = the length of the tape under load  $Q_0$  (mean of two measurements)  
 $L_1$  = length of the tape under load  $Q_1$  (mean of two measurements)  
 $L_n$  = nominal length of the interval under test

#### 4. Determination of Weight-per-Unit Length

- 4.1. Weigh the tape and reel (or case) to  $\pm 0.1$  g. ( $W_1$ )
- 4.2. Remove the tape from the reel or case and weigh the empty reel or case to  $\pm 0.1$  g. ( $W_C$ )
- 4.3. Measure the length of any blank ends on the tape to  $\pm 0.01$  foot. ( $L_B$ )
- 4.4. Use the nominal value 2.5 g for the weight of the loop normally used on steel tapes. ( $W_L$ ) If a larger or smaller loop is used, its weight should be estimated or the weight of the loop should be obtained from the tape manufacturer.
- 4.5. Compute the weight-per-unit length using the equation

$$\text{Weight/length} = \frac{W_1 - W_C - W_L}{L_T + L_B}$$

where

$W_1$  = weight of tape plus reel (or case)  
 $W_C$  = weight of reel (or case)  
 $W_L$  = weight of loop (2.5 g)  
 $L_T$  = graduated length of tape  
 $L_B$  = length of blank ends

## 5. Assignments of Uncertainty

- 5.1. The uncertainty,  $U_c$ , of a correction (the mean of two measurements) is given by the expression

$$U_c = \pm U_s + \frac{ts}{\sqrt{2}}$$

Where  $U_s$  is the uncertainty assigned for the correction for the standard and  $s$  represents the estimate of the standard deviation of a single measurement. The value for  $t$  (corresponding to a probability level of 99.73%) and the number of degrees of freedom on which  $s$  is based will be found in Table 9.3.

- 5.1.1. If control charts are not maintained,  $s$  may be estimated from the average range,  $\bar{R}$ , of the ranges,  $R$  (absolute differences of the measured corrections) over all  $n$  intervals. Thus,

$$\bar{R} = \frac{\sum R}{n}$$

and  $s$  is estimated by

$$s = \bar{R}/d_2^*$$

The factor  $d_2^*$  is obtained by interpolation of the values in Table 9.1. The degrees of freedom,  $\nu$ , associated with  $s$  should be taken as one-half the degrees of freedom shown below the appropriate  $d_2^*$  factor in the same table.‡

- 5.1.2. If control charts are maintained and the measurement system is in a state of statistical control, the value of  $s$  on which the control limits are based may be substituted in the equation of 5.1 and the corresponding value for  $\nu$  is used when selecting the value of  $t$  to be used.

- 5.1.2.1. If control charts are maintained, the second trial may be eliminated with a somewhat larger random uncertainty of the measurement. An  $R$  control chart (difference of duplicates) is recommended and only the zero and one other scale interval (randomly selected) are measured in duplicate. The difference in the  $C_x$  values ( $R$ ) obtained in these control measurements is plotted on the control chart and indication of measurement process control permits

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‡Degrees of freedom are reduced approximately by the factor one-half because the same zero interval measurement, are used for each interval estimated. Values should be rounded to the nearest whole number.

the assignment of the process standard deviation to all measurements made at that time in calibrating the rule. The uncertainty,  $U_c$ , becomes

$$U_c = \pm [U_s + ts]$$

Note: See Chapter 7.4 for further information on the maintenance and use of R control charts.



# APPENDIX A

## CALIBRATION OF METAL TAPES - BENCH METHOD

TEST ITEM \_\_\_\_\_ TAPE MFGR \_\_\_\_\_

SUBMITTED BY \_\_\_\_\_ DATE \_\_\_\_\_

TEMPERATURE: START: TEMP 1 \_\_\_\_\_ TEMP 2 \_\_\_\_\_

FINISH: TEMP 3 \_\_\_\_\_ TEMP 4 \_\_\_\_\_ AVERAGE TEMP \_\_\_\_\_

TENSION \_\_\_\_\_ SUPPORT \_\_\_\_\_

OBSERVER \_\_\_\_\_ SHEET NO. \_\_\_\_\_

COEFFICIENT OF EXPANSION OF UNKNOWN,  $a_x$  = \_\_\_\_\_

COEFFICIENT OF EXPANSION OF STANDARD,  $a_s$  = \_\_\_\_\_

$$C_x = d + C_s + K ; K = L_n [(T - 20)(a_s - a_x)]$$

NOTE: Coefficient of expansion for steel tape =  $11.60 \times 10^{-6}/^{\circ}\text{C}$   
Coefficient of expansion for length bench =  $10.63 \times 10^{-6}/^{\circ}\text{C}$

Inter- val $x_1$ $s_1$	T r i a l	X			S			$d^*$ $x_m - s_m$	Corr'n to Stand. $C_s$	$K^{**}$ Temp. Corr'n	Corr'n to X $C_x$	Range of $C_x$
		Left Edge	Right Edge	Mean $x_m$	Left Edge	Right Edge	Mean $x_s$					
X	1											
S	2											
Average, $\bar{C}_x$												
X	1											
S	2											
Average, $\bar{C}_x$												
X	1											
S	2											
Average, $\bar{C}_x$												
X	1											
S	2											
Average, $\bar{C}_x$												

\*Carry average difference to at least four decimal places if average is in inches, carry to at least six decimal places if average is in meters.

\*\*Carry to at least four decimal places if in inches; at least six decimal places if in meters.

# APPENDIX A

## CALIBRATION OF METAL TAPES - BENCH METHOD

TEST ITEM Surveyor's Tape 100 ft TAPE MFR Lufkin

SUBMITTED BY ABC Company DATE 8/29/86

TEMPERATURE: START: TEMP 1 22.3 °C TEMP 2 22.4 °C

FINISH: TEMP 3 22.4 °C TEMP 4 22.5 °C AVERAGE TEMP 22.4 °C

TENSION 10-lb load SUPPORT throughout

OBSERVER HO SHEET NO. 1

COEFFICIENT OF EXPANSION OF UNKNOWN,  $a_x = 11.60 \times 10^{-6} / ^\circ\text{C}$

COEFFICIENT OF EXPANSION OF STANDARD,  $a_s = 10.63 \times 10^{-6} / ^\circ\text{C}$

$$C_x = d + C_s + K ; K = L_n [(T - 20)(a_s - a_x)]$$

NOTE: Coefficient of expansion for steel tape =  $11.60 \times 10^{-6} / ^\circ\text{C}$

Coefficient of expansion for length bench =  $10.63 \times 10^{-6} / ^\circ\text{C}$

Interval $x_i$ $s_i$	T r i a l	X			S			$d^*$ $x_m - s_m$	Corr'n to Stand. $C_s$	$K^{**}$ Temp. Corr'n	Corr'n to X $C_x$	Range of $C_x$
		Left Edge	Right Edge	Mean $x_m$	Left Edge	Right Edge	Mean $x_s$					
X 0-10'	1	.204	.218	.211	.215	.221	.218	-.007	+.002	.00028	-.00472	.0025"
S 0-10'	2	.182	.197	.1895	.196	.202	.199	-.0095	+.002	.00028	-.00722	
Average, $C_x$											-.00597	
X 0-15'	1	.166	.182	.174	.184	.190	.187	-.013	+.003	.00042	-.00958	.0020"
S 0-15'	2	.198	.214	.206	.218	.224	.221	-.015	+.003	.00042	-.01158	
Average, $C_x$											-.01058	
X 15-20'	1	.160	.174	.167	.168	.174	.171	-.004	-.001	.00014	-.00486	.0015"
S 0-5'	2	.144	.159	.1515	.154	.160	.157	-.0055	-.001	.00014	-.00636	
From 0-20' $C_x = -.01058 + (-.00561) = -.0162"$											Average, $C_x$	-.00561
X 15-30'	1	.216	.232	.224	.219	.225	.222	.002	+.003	.00042	.00542	.0020"
S 0-15'	2	.140	.156	.148	.145	.151	.148	.000	+.003	.00042	.00342	
From 0-30' $C_x = -.01058 + .00442 = -.0062"$											Average, $C_x$	.00442

\*Carry average difference to at least four decimal places if average is in inches, carry to at least six decimal places if average is in meters.

\*\*Carry to at least four decimal places if in inches; at least six decimal places if in meters.

# APPENDIX B-1

## TEMPERATURE CORRECTION FACTOR FOR CALIBRATION OF STEEL TAPES (ALL VALUES IN INCHES X 10<sup>4</sup>)

L <sub>n</sub> Feet	Temperature, °C											
	19	20	21	22	23	24	25	26	27	28	29	30
1	0.1	0	-0.1	-0.2	-0.3	-0.5	-0.6	-0.7	-0.8	-0.9	-1.0	-1.2
2	0.2	0	-0.2	-0.5	-0.7	-0.9	-1.2	-1.4	-1.6	-1.9	-2.1	-2.3
3	0.3	0	-0.3	-0.7	-1.0	-1.4	-1.7	-2.1	-2.4	-2.8	-3.1	-3.5
4	0.5	0	-0.5	-0.9	-1.4	-1.9	-2.3	-2.8	-3.3	-3.7	-4.2	-4.7
5	0.6	0	-0.6	-1.2	-1.7	-2.3	-2.9	-3.5	-4.1	-4.7	-5.2	-5.8
6	0.7	0	-0.7	-1.4	-2.1	-2.8	-3.5	-4.2	-4.9	-5.6	-6.3	-7.0
7	0.8	0	-0.8	-1.6	-2.4	-3.3	-4.1	-4.9	-5.7	-6.5	-7.3	-8.1
8	0.9	0	-0.9	-1.9	-2.8	-3.7	-4.7	-5.6	-6.5	-7.4	-8.4	-9.3
9	1.0	0	-1.0	-2.1	-3.1	-4.2	-5.2	-6.3	-7.3	-8.4	-9.4	-10.5
10	1.2	0	-1.2	-2.3	-3.5	-4.7	-5.8	-7.0	-8.1	-9.3	-10.5	-11.6
15	1.7	0	-1.7	-3.5	-5.2	-7.0	-8.7	-10.5	-12.2	-14.0	-15.7	-17.5
20	2.3	0	-2.3	-4.7	-7.0	-9.3	-11.6	-14.0	-16.3	-18.6	-21.0	-23.3
30	3.5	0	-3.5	-7.0	-10.5	-14.0	-17.5	-21.0	-24.4	-27.9	-31.4	-34.9
40	4.7	0	-4.7	-9.3	-14.0	-18.6	-23.3	-27.9	-32.6	-37.2	-41.9	-46.6
45	5.2	0	-5.2	-10.5	-15.7	-21.0	-26.2	-31.4	-36.7	-41.9	-47.1	-52.4
50	5.8	0	-5.8	-11.6	-17.5	-23.3	-29.1	-34.9	-40.7	-46.6	-52.4	-58.2
60	7.0	0	-7.0	-14.0	-21.0	-27.9	-34.9	-41.9	-48.9	-55.9	-62.9	-69.8
70	8.1	0	-8.1	-16.3	-24.4	-32.6	-40.7	-48.9	-57.0	-65.2	-73.3	-81.5
75	8.7	0	-8.7	-17.5	-26.2	-34.9	-43.6	-52.4	-61.1	-69.8	-78.6	-87.3
80	9.3	0	-9.3	-18.6	-28.0	-37.2	-46.6	-55.9	-65.2	-74.5	-83.8	-93.1
90	10.5	0	-10.5	-21.0	-31.4	-41.9	-52.4	-62.9	-73.3	-83.8	-94.3	-104.8
100	11.6	0	-11.6	-23.3	-34.9	-46.6	-58.2	-69.8	-81.5	-93.1	-104.8	-116.4

For example: For L<sub>n</sub> = 20 feet at 25 °C, the temperature correction is -0.00116 inches.

# APPENDIX B-2

## VALUES FOR K TEMPERATURE CORRECTION FOR CALIBRATION OF STEEL TAPES GRADUATED IN METERS (ALL VALUES IN METERS X 10<sup>6</sup>)

L <sub>n</sub> Meters	Temperature, °C											
	19	20	21	22	23	24	25	26	27	28	29	30
0.1	0.1	0	-0.1	-0.2	-0.3	-0.4	-0.5	-0.6	-0.7	-0.8	-0.9	-1.0
0.2	0.2	0	-0.2	-0.4	-0.6	-0.8	-1.0	-1.2	-1.4	-1.6	-1.7	-1.9
0.3	0.3	0	-0.3	-0.6	-0.9	-1.2	-1.5	-1.7	-2.0	-2.3	-2.6	-2.9
0.4	0.4	0	-0.4	-0.8	-1.2	-1.6	-1.9	-2.3	-2.7	-3.1	-3.5	-3.9
0.5	0.5	0	-0.5	-1.0	-1.5	-1.9	-2.4	-2.9	-3.4	-3.9	-4.4	-4.9
0.6	0.6	0	-0.6	-1.2	-1.7	-2.3	-2.9	-3.5	-4.1	-4.7	-5.2	-5.8
0.7	0.7	0	-0.7	-1.4	-2.0	-2.7	-3.4	-4.1	-4.8	-5.4	-6.1	-6.8
0.8	0.8	0	-0.8	-1.6	-2.3	-3.1	-3.9	-4.7	-5.4	-6.2	-7.0	-7.8
0.9	0.9	0	-0.9	-1.7	-2.6	-3.5	-4.4	-5.2	-6.1	-7.0	-7.9	-8.7
1.0	1.0	0	-1.0	-1.9	-2.9	-3.9	-4.8	-5.8	-6.8	-7.8	-8.7	-9.7
2.0	1.9	0	-1.9	-3.9	-5.8	-7.8	-9.7	-11.6	-13.6	-15.5	-17.4	-19.4
3.0	2.9	0	-2.9	-5.8	-8.7	-11.6	-14.6	-17.5	-20.4	-23.3	-26.2	-29.1
4.0	3.9	0	-3.9	-7.8	-11.6	-15.5	-19.4	-23.3	-27.2	-31.0	-34.9	-38.8
5	4.8	0	-4.8	-9.7	-14.6	-19.4	-24.2	-29.1	-34.0	-38.8	-43.6	-48.5
10	9.7	0	-9.7	-19.4	-29.1	-38.8	-48.5	-58.2	-67.9	-77.6	-87.3	-97.0
15	14.6	0	-14.6	-29.1	-43.6	-58.2	-72.8	-87.3	-101.8	-116.4	-131.0	-145.5
20	19.4	0	-19.4	-38.8	-58.2	-77.6	-97.0	-116.4	-135.8	-155.2	-174.6	-194.0
25	24.2	0	-24.2	-48.5	-72.8	-97.0	-121.2	-145.5	-169.8	-194.0	-218.2	-242.5
30	29.1	0	-29.1	-58.2	-87.3	-116.4	-145.5	-174.6	-203.7	-232.8	-261.9	-291.0

For example: For L<sub>n</sub> = 4 meters, at 25 °C the temperature correction is -0.0000194 meters.

## APPENDIX C

### Supplemental Information

#### C.1. Cleaning

To clean a steel tape before calibration, first wipe the tape with a soft cloth. Then with a soft cloth saturated with alcohol to remove the film of oil used to protect the tape.

After calibration, a thin film of light oil, such as sewing machine oil, should be applied to the tape for protection.

#### C.2. Tolerances

The tolerances for measuring tapes are those stated in the Federal Specification, "Tapes, Measuring (General Use)" which is Federal Spec. GGG-T-106D dated November 12, 1969. Steel tapes of lengths 25, 50, 75, and 100 feet fall under Type II class B. The tolerances are stated in section 3.8.2.6 and given below for reference.

3.8.2.6. Accuracy. The inaccuracy in the length of the ribbon, when supported on a horizontal surface with a tension of 10 pounds at a temperature of 68 °F or 20 °C shall not exceed 0.050 inch for the 75-foot length, and 0.100 inch for the 100-foot length.

Tolerances for a 30-meter tape are given below.

<u>Length Interval</u>	<u>Tolerance</u>
0 through 15 meters	1.27 mm (0.050 inch)
15 through 22 meters	1.91 mm (0.075 inch)
22 through 30 meters	2.54 mm (0.100 inch)

#### C.3. Tension Specifications

The length of a tape will be affected by the temperature of the tape, the tension applied to the tape, and the manner in which the tape is supported. The tape will stretch when tension is applied and will return to its normal length when the tension is removed, provided the tape has not been permanently deformed when it was stretched. The tensions at which steel tapes are to be calibrated, expressed in terms of the load in pounds (or kilograms) to be applied to obtain the tension, are as follows:

lengths less than 25 feet (10 m)	3 1/2 lb (2 kg)
lengths of 25 ft through 100 ft (10 m - 30 m)	10 lb (5 kg)
lengths greater than 100 feet (30 m)	20 lb (10 kg)

The loads should be accurate within 0.1 lb (45 g).



#### C.4. Methods of Support and Tension Considerations

Tapes calibrated in a State laboratory are normally supported on a horizontal surface throughout the entire length of the tape. Also, tapes may be calibrated and used when supported in catenary types of suspension. In these cases, the tape is supported at equidistant points because the weight of the tape affects its length. The weight of the tape increases the tension and the "sag" causes the horizontal length to be shorter than when the tape is supported throughout its length. Equations are given in GMP No. 10 to compute the horizontal straight-line distance of a tape supported at N number of equidistant catenary suspensions and for computing the tension of accuracy, defined as the tension that must be applied to the tape interval to produce its designated nominal length at the observed temperature of the tape.

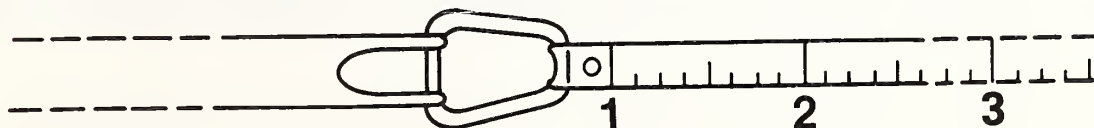
It is sufficient to provide the user of a steel tape with the calibrated length of the tape under standard temperature and tension conditions, the weight per unit-length of the tape, and the AE value for the tape. This information will enable the user to compute the values desired using the equations cited above.

#### C.5. Zero Reference Point

Metal measuring tapes submitted to a State laboratory for calibration normally will be made of steel. Generally, these tapes will have a ring on the end of the tape. For maximum calibration and measurement accuracy, a tape should have a blank end between the ring and the zero graduation. The zero graduation is then more precisely defined and more easily referenced for calibration and use.

Tapes that have the ring as part of the measuring portion of the tape are more difficult to calibrate than a tape with a blank end. When the ring is part of the measuring portion of the tape, the zero reference point shall be the outside end of the ring unless otherwise specified. It is more difficult to obtain a good zero reference setting on the ring due to its curvature and to parallax in reading the edge of the ring against a reference mark. Additionally, the ring may become permanently deformed in use and change the length of the tape. For these tapes the NBS normally calibrates from the 1-foot mark over the length of the tape and then calibrates from the ring to the 1-foot mark. These values are reported separately so the user can obtain maximum measurement accuracy by using the 1-foot graduation as the zero reference point.

When the ring is part of the measuring range of the tape, a special holder for the ring is needed to clamp the tape to the length bench. A strap with an open area in the middle is needed to permit the end of the ring to be seen. The strap is slipped through the ring and the strap is clamped to the length bench. An example is illustrated below.



The edge of the tape to be calibrated (the reading edge) is the edge nearest the observer when the zero graduation is to the observer's left. When viewed through a microscope, some graduations will appear to have irregular edges. The portion of the graduation to be used for calibration is the portion of the graduation at the bottom of the reading edge of the tape. This provides a reference point that can be repeated and referenced by others. Do not attempt to estimate the "best overall" edge of a graduation because this is not easily repeatable and cannot be accurately reproduced by others. If the graduations to be calibrated do not reach to the edge of the tape, the tape should not be calibrated.

#### C.6. Temperature Considerations

The reference temperature for length calibrations is 20 °C. The length of the tape can be determined at any other temperature T by using the equation:

$$L_T = L_{20} [1 + a(T - 20)]$$

where

$L_T$  = length of the tape at a temperature T  
 $L_{20}$  = length of the tape at 20 °C  
 $a$  = linear coefficient of expansion

Since  $a$  is always positive, it can be seen that for temperatures above 20 °C, the tape is longer than it is at 20 °C. For temperatures below 20 °C, the tape is shorter than it is at 20 °C.

If two length standards have different coefficients of expansion because they are made of different materials, the lengths of the tapes will change at different rates as the temperature changes. If two tapes are being compared at a temperature other than 20 °C, these lengths must be corrected back to 20 °C for calibration.

If two length standards are being compared and they have the same coefficient of expansion, then as the temperature changes the lengths will change by the same amount. Hence, if the standards are compared at a temperature other than 20 °C, the relationship between the two standards will be the same as if they were being compared at 20 °C; thus, no temperature correction is needed.

steel tape	$11.60 \times 10^{-6}/^{\circ}\text{C}$	$(6.45 \times 10^{-6}/^{\circ}\text{F})$
length tape	$10.63 \times 10^{-6}/^{\circ}\text{C}$	$(5.91 \times 10^{-6}/^{\circ}\text{F})$
invar tape	$4.0 \times 10^{-7}/^{\circ}\text{C}$	$(2.2 \times 10^{-7}/^{\circ}\text{F})$

#### C.7. Invar Tapes

Invar is an alloy of nickel and steel. Invar tapes are used to obtain measurements of greater accuracy than can be made with steel tapes, because invar has a very low coefficient of expansion. It has the added benefit of being very slow to tarnish from exposure to the atmosphere. However, invar tapes require very careful handling to prevent twists and kinks.

The load to be applied to an invar tape to maintain the desired tension is normally 20 lb. A load of 40 lb is used for the higher tension to determine the AE value (described below). For metric tapes, the normal load is 5 kg. A load of 10 kg is used to determine the AE value.

#### C.8. AE Value

The AE value (area elongation value) for a tape is determined by first calibrating the tape under its normal tension. The load is then increased by 10 or 20 lb and one length interval is recalibrated to determine the length of the tape under the increased tension. The AE factor is then computed as:

$$AE = \frac{Q_1 - Q_0}{L_1 - L_0} L_n$$

where AE = cross-sectional area times Young's modulus of elasticity  
Q<sub>0</sub> = the lower load applied to the tape (e.g., 10 lb)  
Q<sub>1</sub> = the higher load applied to the tape (e.g., 20 lb)  
L<sub>0</sub> = length of the tape under load Q<sub>0</sub>  
L<sub>1</sub> = length of the tape under load Q<sub>1</sub>  
L<sub>n</sub> = nominal length of the interval under test

For example, suppose a 100 foot tape is calibrated from 0 to 100 feet with a load of 10 lb applied to the tape. Suppose this length was 99.992 feet. Suppose the load was increased to 20 lb and the new length was found to be 100.004 feet. The AE value is:

$$AE = \frac{(20 \text{ lb} - 10 \text{ lb}) 100 \text{ feet}}{(100.004 - 99.992) \text{ feet}} = \frac{1000 \text{ lb feet}}{0.012 \text{ feet}} = 83333 \text{ lb}$$

It is recommended that the AE value be determined over the longest interval that is convenient to measure. This minimizes the error in the AE value because of the better readability of the change in length.

#### C.9. Weight per Unit Length

The weight per unit length of a tape can be determined as follows:

1. Weigh the tape and reel (or case).
2. Remove the tape from the reel or case and weigh the empty reel (or case).
3. Measure the length of any blank ends on the tape and add this to the measuring length of the tape.
4. Correct for the weight of the loop on the tape. The weight of the loop that is normally used on steel tapes is approximately 2.5 grams.

The weight per unit length is then computed as:

$$\text{weight per unit length} = \frac{\text{weight of loaded reel} - \text{weight of empty reel} - \text{weight of loop}}{\text{length of tape} + \text{length of blank ends}}$$



## SOP No. 12

### Recommended Standard Operations Procedure for

### Calibration of Steel Tapes

### Tape-to-Tape Method

#### 1. Introduction

##### 1.1. Purpose of Test

The accuracy of a surveyor's measurement often must be provable to 100 ppm (sometimes in a court of law). A significant feature of such proof is the knowledge of the accurate length of the tape used. This procedure describes a technique which will provide such information, by comparison of a tape with a calibrated standard tape.

##### 1.2. Prerequisites

1.2.1. Verify that a valid calibration certificate is available for the standard tape used.

1.2.2. Verify the availability of all equipment necessary to make the test.

1.2.3. Verify that the person performing the calibration is capable of making basic measurements of length, using steel tapes and has been trained in the use of this procedure.

#### 2. Methodology

##### 2.1. Scope, Precision, Accuracy

The method is applicable to the calibration of metal tapes such as used by surveyors, builders, and contractors. The overall length and specified intermediate intervals may be checked by the technique. The accuracy is limited by the accuracy of the calibration of the standard tape and by the precision of intercomparison. The latter should be within  $\pm 0.001$  foot, corresponding to 10 parts per million in a 100 foot tape. The method is limited to calibration of steel tapes (because the tension is specified as the result of a 10-pound load).

##### 2.2. Summary

This procedure is based upon the method developed by C. Leon Carroll Jr., National Bureau of Standards, NBSIR 74-451, "Field Comparisons of Steel Surveyors' Tapes."

The tape to be calibrated is stretched out parallel to a standard tape on a reasonably flat surface. Paper scales (graph paper), graduated in millimeters are used at the zero end and at each



specified interval of calibration to measure any differences between the two tapes. The length of the tape undergoing calibration is computed from the known length of the standard tape and the observed differences between the test tape and the standard.

Calibrations are usually made at each 1-foot interval for the first 10 feet, and at each 10-foot interval to the full length of the tape.

## 2.3. Equipment

2.3.1. Standard tape, calibrated to within  $\pm 0.001$  foot, traceable to NBS.

2.3.2. Pieces of graph paper (10 x 10 to the centimeter, i.e., millimeter graduations), approximately 5 cm in width by 15 cm in height. Number the horizontal centimeter graduations, 0, 1, 2, etc.

2.3.3. Equipment, such as shown in Figure 1, to apply a load to the tapes under test, consisting of:

2.3.3.1. Spring scales (two) one capable of indicating a load of 10 pounds and the other to 20 pounds. The scales should be calibrated with an accuracy of  $\pm 0.1$  lb. This may be done by the arrangement shown in Figure 2.

2.3.3.2. Turnbuckles, suitable for adjusting tension on the tapes.

2.3.3.3. Swivel connectors to prevent axial twisting of the tapes.

2.3.3.4. Magnifying glass to aid in reading the graph paper values.

## 2.4. Procedure

2.4.1. Set up an experimental arrangement similar to that shown in Figure 1. The ends of the tapes (not shown) are held in place by suitable anchor pins.

2.4.2. Stretch the test tape and standard tape parallel to each other on a reasonably flat surface such as the corridor of a building or the surface of a parking lot. The evenness of the surface is less important than the parallelism of the tapes. The two tapes should be separated by a constant distance of about 1 to 3 centimeters. The zero and test intervals of the two tapes should not be in coincidence but rather displaced by one or two centimeters, as indicated in the detail of Figure 1.

- 2.4.3. Use the turnbuckles to apply equal loads of 10 pounds to the two tapes as indicated by the spring scales. (Note the use of swivels to prevent axial twisting.)
- 2.4.4. Place a piece of the graph paper under the zero interval and each interval to be calibrated as shown in the detail in Figure 1. Adjust the tapes and the paper so that the former are precisely aligned with the lateral rulings of the paper. It is convenient but not necessary for these to be the bold centimeter rulings of the paper. Note the amount of separation of the tapes at the zero interval and make corresponding adjustments at each calibration interval of interest. In this way, parallelism of the two tapes is easily verified.
- 2.4.5. Make final adjustment of tensions on the tapes and recheck for parallelism at all test points before taking the readings described in 2.4.6. Do not disturb during the measurement sequence.
- 2.4.6. Read the distances A, B, C, and D as indicated in the detail of Figure 1. Note that A and B are for the zero (or first) interval and are the same for all test intervals. C and D have subscripts 1, 2, etc. corresponding to the interval, i, calibrated. Make all readings to the center of the graduation mark tested and estimate to the closest 0.1 mm. Record all readings in centimeters.
- 2.4.7. Record all measurements on an observation sheet such as that provided in the Appendix of this SOP as First Trial.
- 2.4.8. Release the tension to the tapes and reapply it.
- 2.4.9. Displace each piece of graph paper a few millimeters, then readjust the load, check for parallelism, and record a second series of measurements as Second Trial.
- 2.4.10. Readjust as in 2.4.8 and record a third series of measurements as Third Trial.

## 2.5. Calculations

- 2.5.1. Calculate and record  $A-B-C+D$  for each trial, then record the value of R, the range of these values (difference of highest and lowest) for each scale interval. The range should not exceed 0.15 cm. Sum the values for A, B, C, D for the three trials to use when calculating the length, L, of each interval.
- 2.5.2. The value obtained from  $\Sigma A - \Sigma B - \Sigma C + \Sigma D$  must equal the sum of the column A - B - C + D, otherwise an error has been made in the calculations.

- 2.5.3. Calculate the length of the test tape at each calibration interval according to the following equation:

$$L = S + \frac{k}{3} \Sigma (A - B - C + D)$$

where L = length of test tape at the calibration interval

S = length of standard tape at the calibration interval

k = Conversion factor, tape interval/scale interval, i.e.,

k = 0.032808 ft/cm for tapes graduated in feet

and k = 0.010000 m/cm for tapes graduated in meters

## 2.6. Temperature Correction

No temperature correction is required, provided the test tape and the standard tape are at the same temperature. This will be the case when the measurements are made inside a building. Tapes of the same color would be expected to attain the same temperature, even in sunlight. However, black and white tapes have shown temperature differences of as much as 8 °C when exposed to direct sunlight. In such cases, the temperature difference, even if measured, would be uncertain due to variability of exposure along the length of the tape. Accordingly, calibrations in the laboratory are preferred, when possible.

## 2.7. Measurement of the AE Value

2.7.1. Apply a load of 20 pounds to the test tape while maintaining a load of 10 pounds on the standard tape.

2.7.2. Measure the difference of length over a relatively large interval as described in 2.4. (ordinarily, the maximum interval is chosen).

2.7.3. Calculate the AE value using the equation

$$AE = \frac{10 L_n}{\frac{k}{3} [\Sigma(A-B-C+D)_{20} - \Sigma(A-B-C+D)_{10}]}$$

where  $L_n$  is the nominal value for the interval and all other symbols are the same as those used earlier. The subscripts 10 and 20 signify the readings observed for the respective tensions applied to the test tape.

## 2.8. Determination of Weight-per-Unit Length

- 2.8.1. Weigh the tape and reel (or case) to  $\pm 0.1$  g. ( $W_1$ )
- 2.8.2. Remove the tape from the reel or case and weigh the empty reel or case to  $\pm 0.1$  g. ( $W_C$ )
- 2.8.3. Measure the length of any blank ends on the tape to  $\pm 0.01$  foot. ( $L_B$ )
- 2.8.4. Use the nominal value 2.5 g for the weight of the loop normally used on steel tapes. ( $W_L$ ) If a larger or smaller loop is used, its weight should be estimated or the weight of the loop should be obtained from the tape manufacturer.
- 2.8.5. Compute the weight-per-unit length using the equation

$$\text{Weight/length} = \frac{W_1 - W_C - W_L}{L_T + L_B}$$

where

$W_1$  = weight of tape plus reel (or case)  
 $W_C$  = weight of reel (or case)  
 $W_L$  = weight of loop (2.5 g)  
 $L_T$  = graduated length of tape  
 $L_B$  = length of blank ends

## 2.9. Assignment of Uncertainty

Calculate the uncertainty,  $U_C$  of the calibration, using the following equation

$$U_C = U_S + U_I$$

where  $U_S$  = uncertainty of the standard

$U_I$  = uncertainty of the intercomparison measurement.

The uncertainty of intercomparison can be estimated from the average range,  $R$ , of the three replicate measurements at each calibration point, as follows:

$$\bar{R} = \frac{(R_1 + R_2 + R_3 + \dots + R_k)}{k}$$

where  $k$  = number of points calibrated. The estimate of the standard deviation,  $s$ , is calculated as follows

$$s = \bar{R}/d_2^*$$

The value for  $d_2^*$  to be used can be found in Table 9.1. The degrees of freedom  $\nu$  associated with  $s_R$  should be taken as one-half the degrees of freedom shown below the appropriate  $d_2^*$  factor in the same Table.<sup>§</sup>

The uncertainty of intercomparison,  $U_I$ , is calculated as follows:

$$U_I = \frac{t \bar{R}}{\sqrt{3} d_2^*}$$

The value for  $t$  which can be found in Table 9.3 depends on the number of degrees of freedom,  $\nu$ , associated with  $s$  and the confidence level desired. For example, for 19 calibration points,  $\nu \approx 18$  and  $t = 3.475$  for a 99.73% confidence level.

Accordingly, in this case,

$$U_I = \frac{3.475 \bar{R}}{\sqrt{3} d_2^*}$$

This uncertainty would be applicable to each of the intervals calibrated.

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<sup>§</sup>Degrees of freedom are reduced approximately by the factor one-half because the same zero measurements are used for each interval estimated. Values should be rounded to the nearest whole number.



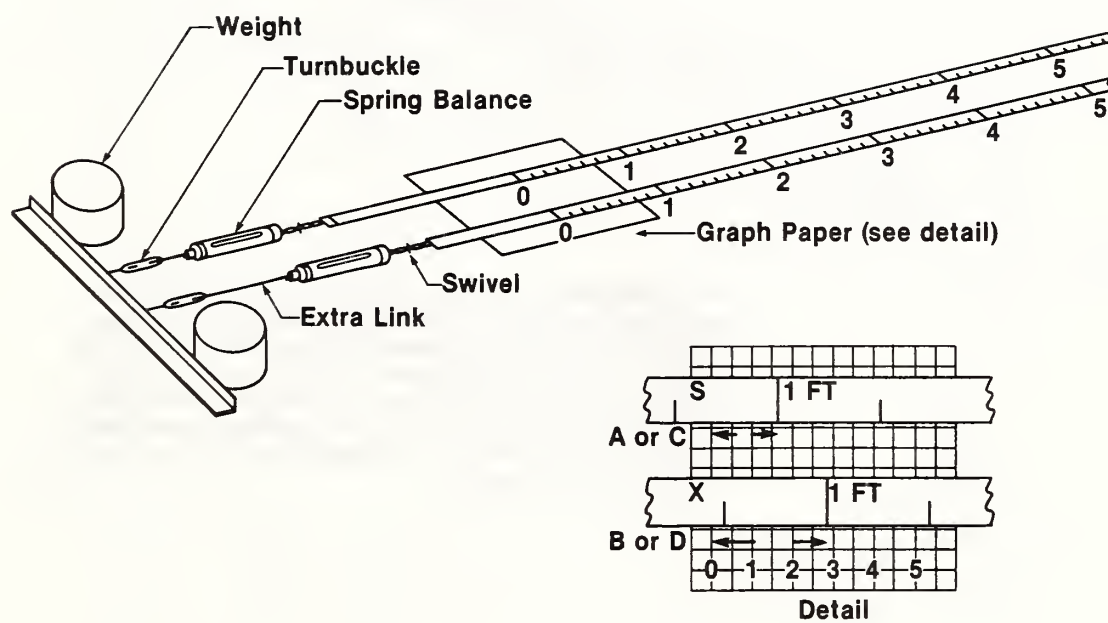


Figure 1. Experimental Arrangement

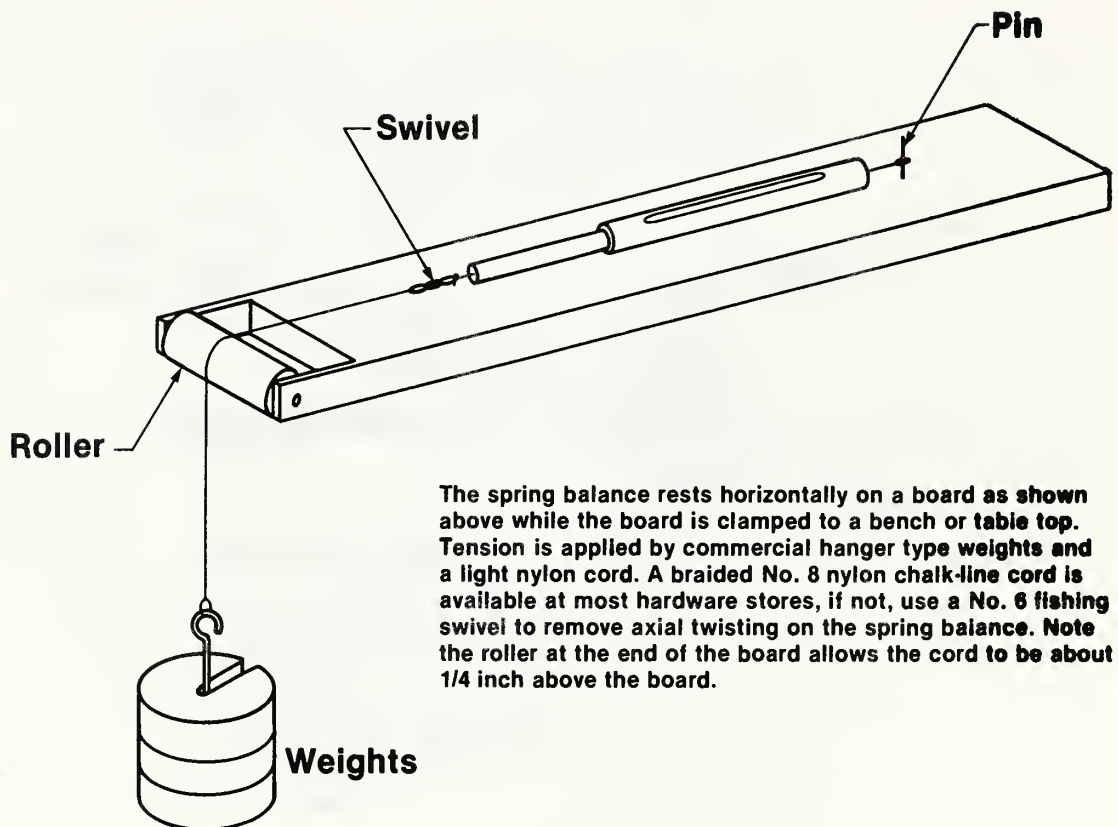


Figure 2. Calibration of Spring Scales.

# Appendix

## OBSERVED DATA AND CALCULATIONS OF THE LENGTH (L) OF A STEEL SURVEYOR TAPE BASED ON A COMPARISON TO A CALIBRATED STANDARD TAPE

### Standard Tape

Manufacturer \_\_\_\_\_  
 Serial No. \_\_\_\_\_  
 Temp. Coef. Exp.,  $\alpha$ = \_\_\_\_\_  
 Weight/Length \_\_\_\_\_ AE= \_\_\_\_\_ LB  
 Material/Color/Finish \_\_\_\_\_

### Test Tape

Manufacturer \_\_\_\_\_  
 Serial No. \_\_\_\_\_  
 Temp. Coef. Exp.,  $\alpha$ = \_\_\_\_\_  
 Weight/Length \_\_\_\_\_ AE= \_\_\_\_\_ LB  
 Material/Color/Finish \_\_\_\_\_  
 Applied Load For Tension \_\_\_\_\_ LB

### Observed Data (in cm)

Interval	Trial	A	B	C	D	A-B-C+D			
	1						$\frac{k}{3}\Sigma(A-B-C+D)$	S	L
	2								
	3								
	$\Sigma$								
Range, R									

Interval	Trial	A	B	C	D	A-B-C+D			
	1						$\frac{k}{3}\Sigma(A-B-C+D)$	S	L
	2								
	3								
	$\Sigma$								
Range, R									

Interval	Trial	A	B	C	D	A-B-C+D			
	1						$\frac{k}{3}\Sigma(A-B-C+D)$	S	L
	2								
	3								
	$\Sigma$								
Range, R									

Interval	Trial	A	B	C	D	A-B-C+D			
	1						$\frac{k}{3}\Sigma(A-B-C+D)$	S	L
	2								
	3								
	$\Sigma$								
Range, R									

Interval	Trial	A	B	C	D	A-B-C+D			
	1						$\frac{k}{3}\Sigma(A-B-C+D)$	S	L
	2								
	3								
	$\Sigma$								
Range, R									

Interval	Trial	A	B	C	D	A-B-C+D			
	1						$\frac{k}{3}\Sigma(A-B-C+D)$	S	L
	2								
	3								
	$\Sigma$								
Range, R									

Interval	Trial	A	B	C	D	A-B-C+D			
	1						$\frac{k}{3}\Sigma(A-B-C+D)$	S	L
	2								
	3								
	$\Sigma$								
Range, R									

# Appendix

## OBSERVED DATA AND CALCULATIONS OF THE LENGTH (L) OF A STEEL SURVEYOR TAPE BASED ON A COMPARISON TO A CALIBRATED STANDARD TAPE

### Standard Tape

### Test Tape

Manufacturer Lufkin  
 Serial No. #5678  
 Temp. Coef. Exp.,  $\alpha = 11.60 \times 10^{-6}/^{\circ}\text{C}$   
 Weight/Length 0.01471b/ft AE= 128000LB  
 Material/Color/Finish steel/black/gloss

Manufacturer Lufkin  
 Serial No. #1234  
 Temp. Coef. Exp.,  $\alpha = 11.60 \times 10^{-6}/^{\circ}\text{C}$   
 Weight/Length 0.01471b/ft AE=        LB  
 Material/Color/Finish steel/white/gloss  
 Applied Load For Tension 10 LB

### Observed Data (in cm)

Interval	Trial	A	B	C	D	A-B-C+D			
0-30'	1	0.83	3.08	2.43	4.65	-0.03	$\frac{k}{3}\Sigma(A-B-C+D)$	S	L
	2	0.22	2.44	3.37	5.55	-0.04			
	3	0.69	2.93	2.03	4.23	-0.04			
	$\Sigma$	1.74	8.45	7.83	14.43	-0.11	- .00120 ft	30.0012	30.0000 ft
Range, R						0.01			

Interval	Trial	A	B	C	D	A-B-C+D			
0-50'	1	0.83	3.08	5.12	7.28	-0.09	$\frac{k}{3}\Sigma(A-B-C+D)$	S	L
	2	0.22	2.44	5.99	8.12	-0.09			
	3	0.69	2.93	5.84	8.03	-0.05			
	Σ	1.74	8.45	16.95	23.43	-0.23			
Range, R						0.04			

### AE test

Interval	Trial	A	B	C	D	A-B-C+D	Load on unknown = 20 lb		
0-50'	1	1.80	2.54	4.82	5.62	0.06	$\frac{k}{3}\Sigma(A-B-C+D)$	S	L
	2	1.25	1.98	5.34	6.17	0.10			
	3	0.60	1.38	4.39	5.17	0.00			
	$\Sigma$	3.65	5.90	14.55	16.96	0.16	0.00175 ft	50.00167	50.00342 ft
Range, R						0.10	( 10 )		

$$AE = \left( \frac{10}{50.00342 - 49.99915} \right) 50$$

$$AE \approx 117000 \text{ lb}$$





SOP No. 13

Recommended Standard Operations Procedure

for

Calibration of Volumetric Ware, Gravimetric Method\*

1. Introduction

1.1. Purpose of Test

Volumetric flasks, pipets, graduated cylinders, burets, and similar glass and plastic volumetric ware such as those used in chemical and clinical laboratories need to be calibrated to ensure accuracy of measurement. This SOP describes procedures for calibration of such ware with capacities ranging from 0.1 cm<sup>3</sup> to 2000 cm<sup>3</sup>. The method is extendable, in principle, to larger volumes, limited only by the capacity and physical dimensions of the weighing apparatus. It is not recommended for volumetric ware with capacities less than 0.1 cm<sup>3</sup>.

1.2. Prerequisites

1.2.1. Verification of Calibration/Calibrants

Verify the validity of calibration of the apparatus required as follows:

- a. Thermometers
- b. Balance/Weights
- c. Supply of distilled water

1.2.2. Verification of Equipment

Verify that balance is in good operating condition.

1.2.3. Verification of Ability to Test

The skills required by this procedure are as follows:

- a. ability to weigh precisely
- b. ability to read and adjust a water meniscus
- c. thorough understanding of the method.

It should be verified that the person performing the test has acquired these skills by previous experience.

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\*Based on the work of J. Lembeck "The Calibration of Small Volumetric Glassware," NBSIR 74-461 (1974).

## 2. Methodology

### 2.1. Scope, Precision, Accuracy

The procedure is applicable for the calibration of any size of measuring container that, when filled with water, will not overload the balance used. The precision of calibration will depend on the care exercised in adjusting the various volumes, and strict adherence to the various steps of the procedure. The accuracy attainable will depend on the uncertainties of the standard weights and the air buoyancy and thermal expansion corrections that are made.

### 2.2. Summary

This procedure is based on a determination of the mass of water contained in or delivered from the vessel that is calibrated. The volumetric determination is calculated from the above measurements and a knowledge of the temperature pressure, and relative humidity of the air, and the temperature of the water that is weighed.

### 2.3. Equipment and Standards

2.3.1. Balance having sufficient capacity to weigh the loaded vessel. The sensitivity of the balance will be a limiting factor in the accuracy of the measurement.

2.3.2. Mass standards or built-in weights calibrated with adequate accuracy. Ordinarily, weights with NBS class S-1 tolerances are required.

2.3.3. Thermometer, calibrated to  $\pm 0.1$  °C for measuring the temperature of the water.

2.3.4. Barometer, calibrated to  $\pm 6$  mm Hg. (Alternatively, the existing barometric pressure may be obtained from the local weather service.)

2.3.5. Distilled or deionized water (See GLP No. 10).

### 2.4. General Considerations

2.4.1. All glassware must be meticulously cleaned, prior to calibration. When clean, the walls will be uniformly wetted. Instructions for cleaning are given in GMP No. 7. An exception is plastic ware which will not be wetted. Follow manufacturer's instructions for cleaning such vessels. Do not use materials that will attack, discolor, or swell plastic ware.

2.4.2. Calibrations are critically dependent on the setting of a meniscus. See GMP No. 3 for guidance in reading a meniscus.

2.4.3. Use water, stored in a large container, that is temperature equilibrated with the laboratory air.

## 2.5. Calibration Procedure for Burets

- 2.5.1. Clamp the buret vertically on a support stand. Also clamp a plain glass test tube, large enough to hold a thermometer, in the vicinity of the buret.
- 2.5.2. Fill buret with water and test for absence of leaks from the tip and stopcock.
- 2.5.3. Drain and record delivery time, defined as the time of unrestricted flow from the zero mark to the lowest graduation mark with the stopcock fully open.
- 2.5.4. Fill the buret slightly above zero mark with temperature equilibrated water and also the test tube that holds the thermometer. Record water temperature.
- 2.5.5. Set the meniscus on the zero mark and touch the tip with the wetted wall of a beaker to remove any excess water.
- 2.5.6. Fully open the stopcock and discharge contents of buret into a weighed weighing flask. The tip should be in contact with the wall of the flask. When the level in the buret is within a few millimeters above the line being calibrated, slow the discharge and make an accurate setting. When the setting is completed, move the flask horizontally to break contact with the tip. Recheck the setting.
- 2.5.7. Stopper and weigh the flask.
- 2.5.8. Check temperature of water in the test tube.
- 2.5.9. Test the next interval in the same manner - from the zero mark to the next interval of test.
- 2.5.10. For burets with a specified waiting time, empty as in 2.5.6 to within a few millimeters of the calibration mark. Pause for the specified waiting time (e.g. 30 s) then adjust the meniscus to the graduation line as in 2.5.6.

## 2.6. Calibration Procedure for Pipets (one-mark)

- 2.6.1. Fill the pipet to the index mark and measure the delivery time with the tip in contact with the internal surface of a beaker.
- 2.6.2. Refill the pipet by suction, slightly above the index line. Record the water temperature. Wipe tip with filter paper, then slowly lower level to index line, using a stopcock or pinch clamp for fine control. The tip must be in contact with the wetted wall of a beaker while this setting is being made. Do not remove any water remaining on tip at this time.

- 2.6.3. Hold pipet in a vertical position and deliver water into a previously weighed weighing flask, with the tip in contact with its inside wall or neck.
- 2.6.4. After flow has ceased, wait 2 seconds then remove the pipet from contact with the flask.
- 2.6.5. Stopper the flask and weigh with its contained load.
- 2.7. Calibration of Flasks (To Contain)
  - 2.7.1. Clean and dry flask, then stopper and weigh.
  - 2.7.2. Place an appropriate sized funnel in neck and almost fill flask while maneuvering to wet the entire neck below the stopper.
  - 2.7.3. Let stand for 2 minutes then adjust to set meniscus at calibration line.
  - 2.7.4. Determine temperature of water by putting some in a beaker or test tube containing a thermometer.
  - 2.7.5. Weigh filled flask.
- 2.8. Calibration of Flasks (To Deliver)
  - 2.8.1. Clean but do not dry flask.
  - 2.8.2. Fill flask to reference line as described in 2.7, then weigh full vessel, with cap or stopper.
  - 2.8.3. Empty flask over a 30-second period by gradually inclining it so as to avoid splashing. When main drainage has ceased, hold in vertical position for 30 seconds unless another drain time is specified, then touch off the drop of water adhering to the top of the flask.
  - 2.8.4. Place stopper or cap on flask and reweigh.
- 2.9. Calibration of Other Volumetric Glassware
  - 2.9.1. Measuring Pipet

Calibrate in a manner similar to that used to calibrate burets (2.5).
  - 2.9.2. Graduated Cylinders

Calibrate in a manner similar to that used for flasks (2.7; 2.8).



### 3. Calculations

- 3.1. Calculate the volume from the weight of water, contained or delivered, as follows:

$$V_{20} = (I_L - I_E) (Q) \left( \frac{1}{\rho_W - \rho_A} \right) \left( 1 - \frac{\rho_A}{\rho_B} \right) [1 - \alpha(T-20)]$$

where:

$I_L - I_E$  = the difference, in grams, obtained from the balance indications associated with the empty vessel and the loaded vessel.

$Q$  = the apparent mass conversion factor defined by the expression

$$Q = \frac{\rho_B (D_{20} - .0012)}{D_{20} (\rho_B - .0012)}$$

where  $\rho_B$  is the density of the balance weights in g/cm<sup>3</sup>, and  $D_{20}$  is the apparent mass scale to which the weights are adjusted. The factor has a maximum value of 1.000013 hence may be considered as unity for most calibrations.

$\rho_W$  = density of water at the temperature of measurement

$\rho_A$  = density of air at the conditions of calibration

$\alpha$  = the thermal cubical coefficient of expansion for the vessel being calibrated

$T$  = temperature of calibration, °C

- 3.2. The equation given in 3.1 may be simplified to

$$V_{20} = (I_L - I_E) Z$$

where  $Z$  represents the product of all other factors.

- 3.3. Values for  $Z$  as a function of temperature and pressure for use with the equation of 3.2 will be found in Tables 1 and 2 of this SOP when  $\rho_B = 7.78 \text{ g/cm}^3$  and  $D_{20} = 8.3909 \text{ g/cm}^3$ .
- 3.4. Values for the density of water and air, and for the cubical expansion will be found in Tables 9.8, 9.9, and 9.10. These should be used in connection with equation 3.1 for cases where the tabulated  $Z$  factors do not apply.

#### 4. Precision and Accuracy

- 4.1. The precision attainable will depend on the precision of the weighings and the setting of the meniscus. Experience has shown the following to be experimentally realizable.

Vessel	Nominal Size, cm <sup>3</sup>	Reproducibility, cm <sup>3</sup>
Transfer Pipet	1	0.002
	2	0.002
	5	0.002
	10	0.003
	15	0.005
	25	0.005
	50	0.007
	100	0.010
Flasks	10	0.005
	25	0.005
	50	0.007
	100	0.011
	200	0.014
	250	0.017
	500	0.021
Burets	1000	0.042
	10	0.003
	25	0.005
	50	0.007
	100	0.012

- 4.2. The accuracy attained will depend on the accuracy of the weighings and of the temperature of the water.

Individual biases can result from idiosyncrasies in reading menisci and in improper observance of drainage times.

Problems of internal cleanliness can cause large and unpredictable errors due to drainage of the vessels being calibrated.

Table 1: Values of Z in Eq 3.2 as a Function of Temperature and Pressure for  
Use in Calibration of Borosilicate Glassware<sup>A</sup>

Barometric Pressure			Temperature,															
kPa	mbar	mm Hg	19	20	21	22	23	24	25	26	27	28						
77.33	773	580	1.00243	1.00262	1.00281	1.00302	1.00324	1.00347	1.00371	1.00396	1.00422	1.00449						
79.99	800	600	1.00245	1.00264	1.00283	1.00304	1.00326	1.00349	1.00374	1.00399	1.00424	1.00451						
82.66	827	620	1.00248	1.00267	1.00287	1.00308	1.00330	1.00353	1.00377	1.00402	1.00428	1.00455						
85.33	853	640	1.00251	1.00270	1.00290	1.00311	1.00333	1.00356	1.00380	1.00405	1.00431	1.00458						
87.99	880	660	1.00254	1.00272	1.00292	1.00313	1.00335	1.00358	1.00382	1.00408	1.00433	1.00460						
90.66	907	680	1.00256	1.00275	1.00295	1.00316	1.00338	1.00361	1.00385	1.00410	1.00436	1.00463						
93.33	933	700	1.00259	1.00278	1.00298	1.00319	1.00341	1.00364	1.00388	1.00413	1.00439	1.00466						
95.99	960	720	1.00262	1.00281	1.00301	1.00322	1.00344	1.00367	1.00391	1.00416	1.00442	1.00468						
98.66	987	740	1.00265	1.00284	1.00304	1.00324	1.00346	1.00370	1.00393	1.00418	1.00444	1.00471						
101.32	1013	760	1.00268	1.00286	1.00306	1.00327	1.00349	1.00372	1.00396	1.00421	1.00447	1.00474						
103.99	1040	780	1.00270	1.00289	1.00309	1.00330	1.00352	1.00375	1.00399	1.00424	1.00450	1.00477						
106.66	1067	800	1.00273	1.00291	1.00312	1.00333	1.00355	1.00378	1.00402	1.00427	1.00452	1.00479						

<sup>A</sup>Values of Z assume a relative humidity of 50%.

Table 2: Values of Z in Eq 3.2 as a Function of Temperature and Pressure for  
Use in Calibration of Soda-Lime Glassware<sup>A</sup>

Barometric Pressure			Temperature,															
kPa	mbar	mm Hg	19	20	21	22	23	24	25	26	27	28						
77.33	773	580	1.00244	1.00262	1.00279	1.00299	1.00319	1.00341	1.00363	1.00387	1.00411	1.00437						
79.99	800	600	1.00246	1.00264	1.00281	1.00301	1.00321	1.00343	1.00366	1.00390	1.00414	1.00439						
82.66	827	620	1.00250	1.00267	1.00285	1.00305	1.00326	1.00347	1.00369	1.00393	1.00417	1.00443						
85.33	853	640	1.00253	1.00270	1.00288	1.00308	1.00328	1.00350	1.00372	1.00396	1.00420	1.00446						
87.99	880	660	1.00256	1.00272	1.00290	1.00310	1.00330	1.00352	1.00374	1.00399	1.00422	1.00448						
90.66	907	680	1.00258	1.00275	1.00293	1.00313	1.00333	1.00355	1.00377	1.00401	1.00425	1.00451						
93.33	933	700	1.00261	1.00278	1.00296	1.00316	1.00336	1.00357	1.00380	1.00404	1.00428	1.00454						
95.99	960	720	1.00264	1.00281	1.00299	1.00319	1.00339	1.00361	1.00383	1.00407	1.00431	1.00456						
98.66	987	740	1.00267	1.00284	1.00302	1.00321	1.00341	1.00364	1.00385	1.00409	1.00433	1.00459						
101.32	1013	760	1.00270	1.00286	1.00304	1.00324	1.00344	1.00366	1.00388	1.00412	1.00436	1.00462						
103.99	1040	780	1.00272	1.00289	1.00307	1.00327	1.00347	1.00369	1.00391	1.00415	1.00439	1.00465						
106.66	1067	800	1.00275	1.00291	1.00310	1.00330	1.00350	1.00372	1.00394	1.00418	1.00441	1.00467						



Recommended Standard Operations Procedure

for

Gravimetric Calibration of Volumetric Ware Using an Electronic Balance

1. Introduction

1.1. Purpose of Test

This procedure describes the calibration of either the "to deliver" or "to contain" volume of measuring containers that may be used as volumetric measuring standards.

1.2. Prerequisites

- 1.2.1. Verify that valid calibration certificates are available for the standard masses to be used.
- 1.2.2. Verify the availability of an adequate supply of distilled or deionized water.
- 1.2.3. Verify that an electronic balance of sufficient capacity is available and in good operating condition.
- 1.2.4. Verify that the person performing the calibration is trained and proficient in carrying out the test procedure.

2. Methodology

2.1. Scope, Precision, Accuracy

The procedure is applicable for the calibration of any size of measuring container that, when filled with water, will not overload the electronic balance used. Typical containers range in capacity from 1 mL to 20 L. The precision of calibration will depend on the care exercised in adjusting the various volumes, and strict adherence to the various steps of the procedure. The accuracy attainable will depend on the uncertainties of the standard weights and the air buoyancy and thermal expansion corrections that are made.

2.2. Summary

The electronic balance used is first calibrated by weighing a standard mass. The volumetric vessel to be calibrated is then weighed dry or "wetted down", depending on whether the calibration is to be made on a "to contain" or "to deliver" basis. The container is filled with pure water of known temperature and re-weighed. The difference in mass is used to calculate the capacity of the container.



### 2.3. Equipment and Standards

- 2.3.1. Electronic balance with capacity sufficient for the required weighings. The resolution and repeatability should be at least that of the acceptable uncertainty of the calibration.
- 2.3.2. Supply of deionized or distilled water.
- 2.3.3. Calibrated thermometer, readable to  $\pm 0.1$  °C.
- 2.3.4. Barometer or other means to ascertain air pressure at time of weighings to  $\pm 133$  Pa ( $\pm 1$  mm Hg).
- 2.3.5. Standard calibrated masses for calibrating the scale of the balance.

### 2.4. Procedure

#### 2.4.1. Cleanliness check

Verify that all containers to be calibrated are clean as evidenced by uniform drainage of water. No water droplets should remain on any interior surface as the water drains from the container. A reproducible "wet-down" weight is evidence for cleanliness in cases where it is not possible to visually check for uniform drainage. Use GMP No. 6 or 7 to clean vessels as necessary.

#### 2.4.2. Drying procedure.

Use GMP No. 7 as the procedure to dry any container to be calibrated on a "to-contain" basis.

#### 2.4.3. Wet-down

Fill the container to capacity with distilled or deionized water, then empty over a 30-second period while avoiding splashing. Drain for 30 seconds unless another drain time is specified.

#### 2.4.4. Weighings

2.4.4.1. Zero the balance and record reading as  $O_1$ . Place a standard mass,  $m_s$ , on the balance platform ( $m_s$  should be slightly larger than the mass of the filled vessel). Record reading as  $O_2$ .

2.4.4.2. Place dry or "wet-down" container on balance platform, as appropriate, and record reading as  $O_3$ .\*

---

\*When calibrating "to deliver" vessels,  $O_4$  may be measured before  $O_3$ .

Caution: All containers must be dry on the outside for all weighings.

2.4.4.3. Fill container to its reference mark, carefully adjusting the meniscus to minimize filling error (See GMP No. 3). Weigh the filled vessel and record reading as  $O_4$ . Read temperature of the water used to fill the container.

2.4.4.4. Immediately after weighing, check temperature of water in container. If the temperature differs by more than 0.2 °C from that of 2.4.4.3, refill and reweigh.

2.4.4.5. Record air temperature and barometric pressure at time of above measurements.

2.4.4.6. Make a duplicate determination.

### 3. Calculations

3.1. Compute the volume,  $V_t$ , for each determination using the equation:

$$V_t = (O_4 - O_3) \left( \frac{m_s}{O_2 - O_1} \right) \left( 1 - \frac{\rho_A}{\rho_s} \right) \left( \frac{1}{\rho_w - \rho_A} \right)$$

where

$m_s$  = mass of standard used in 2.4.4.1

$\rho_s$  = density of  $m_s$

$\rho_A$  = density of air at temperature and pressure of calibration from Table 9.9

$\rho_w$  = density of water at temperature of calibration, from Table 9.8

$V_t$  = represents either the "to contain" or "to deliver" volume, depending on whether  $O_3$  represents a dry or a "wet down" container at the temperature of measurement.

3.2. Compute  $V_t$  for each trial and the mean,  $\bar{V}_t$  for the duplicate measurements.

3.3. Compute  $V_{20}$ , the volume at 20 °C, using the expression:

$$V_{20} = \bar{V}_t [1 - \alpha (T-20)]$$

where  $\alpha$  is the cubical coefficient of expansion of the container being calibrated, (see Table 9.10), and T is the average of the temperature of the water at the two determinations.

#### 4. Assignment of Uncertainties

- 4.1. The calibration uncertainty,  $U$ , for the mean,  $\bar{V}_t$ , is estimated, using the expression

$$U = \pm [U_s + U_m]$$

where

$U_s$  = uncertainty of standards  
 $U_m$  = uncertainty of measurement

- 4.2. The uncertainty of measurement,  $U_m$ , may be estimated (for the mean of duplicates) by

$$U_m = \frac{ts}{\sqrt{2}}$$

where  $s$  is the estimate of the long-term standard deviation of a single measurement. The value for  $t$  (corresponding to a probability level of 99.73%) is obtained from Table 9.3 corresponding to the number of degrees of freedom on which  $s$  is based.

The standard deviation,  $s$ , may be estimated by measuring a volume standard at least 7 times, no two measurements of which may be made on a single day. Calculate the mean and the standard deviation in the conventional manner. The latter is the value of  $s$  that is used. In this case select the value for  $t$  from Table 9.3 based on the number of degrees of freedom involved in computing  $s$ .

Note: Repetitive measurements made on the same day estimate only the short-term standard deviation.

Recommended Standard Operations Procedure for  
Calibration of Intermediate and Large Volume Standards  
Gravimetric Method\*

1. Introduction

1.1. Purpose of Calibration

This procedure is applicable to calibrations where high accuracy is needed, or where the volumetric transfer method is not possible. It is especially useful for the capacity range of 5 to 100 gallons.

1.2. Prerequisites

- 1.2.1. Verify that a balance of adequate readability, sensitivity and capacity is available.
- 1.2.2. Verify that appropriate calibrated mass standards are available.
- 1.2.3. Verify that an adequate supply of pure water is available.
- 1.2.4. Verify that the person performing the calibration measurements is trained and proficient in using this procedure and in the accurate weighings needed.

2. Methodology

2.1. Summary

This procedure may be used to determine three calibration constants:

- a. The containment volume,  $V_C$ , which is the volume of water required to fill the vessel at a specified temperature (usually 60 °F).
- b. The delivery volume,  $V_D$ , which is the volume of water that may be poured or drained from the vessel at a specified temperature (usually 60 °F) under specified conditions (see note 1).
- c. The neck constant,  $K$ , which relates the true volume of the neck to the value indicated by the neck scale.

The mass of water required to fill the vessel is measured at an observed temperature. The known density of water and coefficient of expansion of the vessel are used to calculate the volume of the vessel at a reference temperature.

---

\*Based on the work of J. Houser "Procedure for the Calibration of Volumetric Test Measures" NBSIR 73-287.

The precision attained will depend on the sensitivity of the balance and on the care exercised in carrying out the various operations of the procedure. Cleanliness of the vessel and strict adherence to the prescribed drainage procedure are critical for high precision. The accuracy of calibration depends on all of the above plus the uncertainties of the various weighings, the volumetric temperature corrections, the proper correction for air buoyancy, and the purity of the water used as the standard. The metrologist must consider critically all of the above factors when assigning uncertainty limits to the calibrated volumes.

With careful observance of good measurement practices, an overall relative uncertainty of  $\pm 0.02$  percent should be attainable.

## 2.2. Apparatus

- 2.2.1. Equal-arm balance of adequate capacity and sensitivity (standard deviation of 500 mg or better).
- 2.2.2. Weights (calibrated to at least 1:50,000) as appropriate for the accuracy requirements.
- 2.2.3. Thermometer calibrated to  $\pm 0.1$  °C.
- 2.2.4. Barometer (calibrated to  $\pm 2$  mm) or means to ascertain atmospheric pressure to  $\pm 2$  mm of mercury (266 Pa).
- 2.2.5. Precision ball bearings for neck calibration (2.5 in, grade 50) or, alternatively, a calibrated buret (0-50 mL) or other volumetric measuring device.
- 2.2.6. Supply of distilled or deionized water, meeting ASTM Type IV specification (See GLP No. 10). The water should be stored in the laboratory for about one day, prior to use, to come to temperature equilibrium with the laboratory.
- 2.2.7. Watch with second hand or other timing device to control drainage times.
- 2.2.8. Cap to place over neck of the vessel.

## 2.3. Procedure

### 2.3.1. Preliminary Operations

- 2.3.1.1. Clean interior of the vessel with biodegradable low-sudsing detergent (see GMP No. 6) with gentle scrubbing as necessary. Rinse several times with clean water to remove all detergent, followed by pure water. Check for leaks and for uniform drainage.



- 2.3.1.2. Clean and dry outside of container to remove any material which might subsequently dislodge and cause an erroneous weighing.
- 2.3.1.3. Dry inside by rinsing with small amounts of alcohol or by drawing clean, filtered air through the vessel (several hours required).
- 2.3.2. Empty Weight
  - 2.3.2.1. Weigh empty vessel and cap using any method of high accuracy. The method of double substitution (SOP No. 3 Option A) is recommended. This is mass  $m_1$ .
  - 2.3.2.2. Record air temperature and pressure needed for subsequent buoyancy corrections.
- 2.3.3. Filled Weight
  - 2.3.3.1. Fill vessel with pure water to the zero point on the neck scale. Be sure vessel is level. Bounce the liquid to disturb meniscus to assure it has reached equilibrium level. Record water temperature and readjust level if necessary. Be sure that no water remains on the outside of the vessel when it is weighed. (See note 2.)
  - 2.3.3.2. Weigh filled vessel and cap as in 2.3.2. This is mass  $m_2$ .
- 2.3.4. Drained Weight
  - 2.3.4.1. Drain vessel using appropriate discharge/pour and drain times (see note 1). Replace cap.
  - 2.3.4.2. Weigh drained vessel and cap as in 2.3.2. This is mass  $m_3$ .
- 2.3.5. Neck Calibration (Option I)
  - 2.3.5.1. Fill vessel with water to a scale division,  $d_a$ , near the bottom of the scale range. Record reading.
  - 2.3.5.2. Add a suitable number of precision ball bearings to the vessel, successively, and observe intermediate scale readings  $d_b$ ,  $d_c$ , etc., and a final reading  $d_f$  near top of scale.
  - 2.3.5.3. A plot of scale readings with respect to number of spheres should be linear and will be a gross check of the validity of this calibration.

2.3.5.4. Calculate the scale factor, K, using the expression

$$K = \frac{n \pi d^3}{8(d_f - d_a)} F$$

where  $n$  = total number of spheres used to increase scale reading from  $d_a$  to  $d_f$

$d$  = diameter of a sphere

$F$  = factor to correct volume units of sphere to those of vessel.

### 2.3.6. Neck Calibration (Option II)

2.6.3.1. Fill vessel with water to a scale division,  $d_a$ , near the bottom of the scale range. Record reading.

2.6.3.2. Make appropriate successive additions of water from a calibrated buret or other suitable volumetric measuring device. Record scale readings after each addition.

2.6.3.3. A plot of scale readings with respect to volume added should be linear and will be a gross check of the validity of this calibration.

2.6.3.4. Calculate the scale factor, K, using the expression

$$K = \frac{V}{(d_f - d_a)} F$$

where  $V$  = the total volume of water added to increase scale reading from  $d_a$  to  $d_f$

$F$  = factor to correct volume units of buret (or volumetric device) to scale units of vessel.

## 3. Calculations

3.1. To calculate containment volume,  $V_C$ , at temperature of measurement,  $t$ ,

$$V_{Ct} = \frac{m_2 - m_1 + \rho_1 V_{m1} - \rho_2 V_{m2}}{\rho_w - \rho_2}$$

3.2. To calculate delivered volume,  $V_D$ , at temperature of measurement,  $t$

$$V_{D_t} = \frac{m_2 - m_3 + \rho_2 V_{m_2} - \rho_3 V_{m_3}}{\rho_w - \rho_2}$$

3.3. To correct volumes to reference temperature,  $t_R$

$$V_{t_R} = V_t [1 + \alpha (t_R - t)]$$

3.4. To correct volume measured in  $\text{cm}^3$  to gallon

$$V_{\text{gal}} = 0.00026417 V_{\text{cm}^3}$$

3.5. Symbols

$m_1$  = weight of empty vessel in air  
 $m_2$  = weight of filled vessel in air  
 $m_3$  = weight of drained vessel in air  
 $\rho_1$  = density of air at time empty vessel is weighed  
 $\rho_2$  = density of air at time filled vessel is weighed  
 $\rho_3$  = density of air at time drained vessel is weighed  
 $V_{m_1}$  = volume of weight,  $m_1$   
 $V_{m_2}$  = volume of weight,  $m_2$   
 $V_{m_3}$  = volume of weight,  $m_3$   
 $\rho_w$  = density of water at temperature of filling  
 $\alpha$  = cubical coefficient of expansion of the vessel

3.6. Physical Constants

3.6.1. Density of Air

Value at temperature and pressure of weighings obtained from Table 9.9, or for higher accuracy work, calculated from the equation given in Section 8 of the Appendix to SOP No. 2.

3.6.2. Density of Water

Values for the density of water are given in Table 9.8.

3.6.3. Coefficient of Expansion

Values for the cubical coefficient of expansion of various materials are given in Table 9.10.

4. Assignment of Uncertainties

The uncertainty may be estimated based upon the contributions of the possible sources of error. Several measurements may be made to obtain an estimate of repeatability of the procedure.

4.1. The precision attainable will depend on the precision of the weighings and the setting of the meniscus.

- 4.2. The accuracy attained will depend on the accuracy of the weighings and of the temperature of the water.

Individual biases can result from idiosyncrasies in reading menisci and in improper observance of drainage times.

Problems of internal cleanliness can cause large and unpredictable errors due to drainage of the vessels being calibrated.

#### NOTES

1. Pour and drain times. It is not possible to completely drain a filled container, because some of the contents will remain as a film or other-wise. By strict adherence to a specified procedure, the residual contents can be held essentially constant so that reproducible calibration constants can be obtained. The conditions conventionally selected are as follows:

- a. For bottom-drain containers: open drain valve fully and allow contents to discharge at maximum rate. When flow ceases, wait 30 seconds, close valve, and touch off any drops adhering to spout.
- b. For pour-type containers: pour contents by gradually tilting container to an 85° angle, so that virtually all is delivered in 30 seconds. Allow to drain for an additional 10 seconds, then touch-off any drops adhering to the lip.

The above described instruction must be precisely followed during calibration and use of the calibrated vessels.

2. A suitable cap (an empty glass beaker, for example) should be placed on the top of open vessels to minimize evaporation losses. If used, the cap should be included in all weighings.
3. When a slicker-plate standard is calibrated, the plate should be used to fix the water level in it. This plate should be weighed along with the standard during each such operation.

Recommended Standard Operations Procedure for

Calibration of Measuring Flasks

Volume Transfer Method

1. Introduction

1.1. Purpose of Test

This procedure describes a method for calibration of the "to deliver" volume of measuring flasks that, in turn, will be used as volumetric measurement standards.

1.2. Prerequisites

- 1.2.1. Verify that valid calibration certificates are available for the standard pipets and the standard burets used in the test.
- 1.2.2. Verify the availability of an adequate supply of distilled or deionized water.
- 1.2.3. Verify that the burets and pipets are clean and in good operational condition.
- 1.2.4. Verify that the person performing the calibration is trained and proficient in carrying out this test procedure.

2. Methodology

2.1. Scope, Precision, Accuracy

The method is applicable for the calibration of any size of measuring flask for which standard pipets of comparable volume are available. Typical flasks have volumes in the range of 1 gill to 1 gallon. The precision of calibration will depend on the care exercised in adjusting the various volumes and strict adherence to the various steps of the procedure. Typical standard deviations of 0.25 to 2.0 minims should be achievable depending on the capacity of the flask calibrated. The accuracy will depend on the accuracy of calibration of the several volumetric standards used together with the precision of the intercomparison. Clean glassware and strict adherence to the drainage instructions are essential for precise and accurate results.

2.2. Summary

The flask to be calibrated is given an initial wet-down, then almost filled with water delivered from a calibrated pipet. Additional water is added from a calibrated buret until the meniscus coincides with the calibration mark of the flask. The sum of such volumes delivered into the wet flask is equivalent to its "to deliver"



volume. It is assumed that the flask is marked with a 10-second drain time. If another drain time is marked on the flask, it should be used. If the drain time is not marked on the flask, a 30-second drain time should be used.

## 2.3. Equipment

- 2.3.1. Standard pipet(s) of suitable volume with calibration certificate(s) traceable to NBS.
- 2.3.2. Standard buret(s) of 120 minims or 10 mL capacity, with calibration certificate traceable to NBS.
- 2.3.3. Supply of distilled or deionized water.
- 2.3.4. Calibrated thermometer, readable to 0.1 °C, if calibration of other than borosilicate glassware is to be done.

## 2.4. Procedure

### 2.4.1. Cleanliness check

Verify that all glassware, including the standards used and the vessels to be calibrated, is internally clean, as evidenced by uniform drainage of water. No water droplets should remain on the internal surfaces as the water drains from the vessels. If this occurs, the glassware must be cleaned with suitable agents such as a sodium dichromate-sulfuric acid solution and rinsed with pure water until uniform drainage is obtained. (See GMP No. 7)

### 2.4.2. Wet down

- 2.4.2.1. Fill the standard pipet to overflowing, then drain into an empty vessel. Check for uniform drainage. Touch-off the pipet outlet tip against the container wall to remove excess droplets and to establish a constant tip retention volume. The pipet and bore of the delivery side of the stopcock should appear to be "empty" and the delivery tip should contain a small volume of water retained in it. This is the "wet down" condition of the pipet. It must be done at the beginning of each test sequence or each test day.
- 2.4.2.2. Refill the pipet and discharge it into the flask to be calibrated. Empty the flask in a 30 second period by gradually inclining the flask so as to avoid splashing of the walls as much as possible. When the main drainage stream has ceased, the flask will be nearly vertical. Allow an additional 10 second drainage after discharge of its contents, then touch off the rim of the flask to remove any drops adhering to it. At the same time, check that

uniform drainage has been achieved. This establishes the "to deliver" condition of the flask. Note that some small amount of water will remain in the flask. Each flask to be calibrated must be given this "wet down" treatment.

#### 2.4.3. Conditioning the buret

2.4.3.1. Fill the standard buret to overflow and drain several times to verify uniform drainage. Refill with water. Note that the stopcock bore and delivery tip should be filled with water at all times, in contrast to the condition for the pipet.

2.4.3.2. Touch-off the delivery tip against the wall of the receiving vessel, to remove any droplet adhering to the external surface of the former. This is an operation that must be done every time a measured volume of water is delivered from the buret.

#### 2.4.4. Calibration

2.4.4.1. Fill the standard pipet to overflow. The delivery bore and the delivery tip will be empty, except for the small volume retained in the latter. Place the inside of the flask neck in contact with the tip of the pipet or buret, to avoid splashing but in a manner that does not block the flow of water. Do this whenever transfers are involved.

2.4.4.2. Deliver the contents of the pipet into the "wet down" flask. This should nearly fill the flask to the calibration line.

2.4.4.3. Add water to the flask from the buret until the meniscus coincides with the calibration mark. (See GMP No. 3 for instructions on how to read a meniscus.) Read the volume delivered, and record on a suitable data sheet such as the one in the Appendix.

2.4.4.4. Empty the measuring flask as described in 2.4.2.2 to re-establish "wet down."

#### 2.4.5. Replicate Measurement

2.4.5.1. Repeat the procedure described in 2.4.4.

2.5. Record all data using the form given in the Appendix or a similar format.

### 3. Computations

- 3.1. Compute each individual "to deliver" volume,  $V_{TD}$ ,

$$V_{TD} = V_P + V_B$$

where  $V_P$  = "to deliver" calibrated volume of the standard pipet  
 $V_B$  = volume of water delivered from the buret, corrected for any calibration values

Report the average volume  $V_{TDM}$  as the value for the flask.

### 4. Assignment of Uncertainty

- 4.1. The calibration uncertainty,  $U$ , is estimated, using the expression

$$U = \pm [U_s + U_m]$$

where  $U_s$  = uncertainty of the calibration standards used  
(ordinarily, the uncertainty of  $V_P$ )

$U_m$  = uncertainty of the measurement process

- 4.2. The uncertainty,  $U_m$ , of  $V_{TDM}$  is estimated from the expression

$$U_m = ts$$

- 4.3. If a control chart has been maintained for this measurement procedure, the value for  $s$  may be based on the control chart (see Recommended Standard Operations Procedure, SOP No. 17, Control Charts for Calibration of Measuring Flasks), and  $t$  is obtained from Table 9.3, corresponding to the 99.73% level of confidence and the number of degrees of freedom associated with the standard deviation  $s$ .

When a control chart is not available, the value of  $s$  may be estimated from the results of at least 12 sets of duplicate calibrations as described in SOP No. 17.

### NOTES

1. The volumetric standards are made of borosilicate glass. If borosilicate glassware is to be calibrated, no temperature correction is required for the calibration process, provided the water temperature is the same (to within 0.5 °C) while it is in the standard pipet, buret, and the flask.
2. For calibration of other kinds of glassware, not only should the above temperature condition be realized but the water temperature must also be known. The water used must be stored in the laboratory until its temperature is equilibrated with its surroundings and the temperature of the discharged water is measured, using the calibrated thermometer. The "to deliver" volume of the flask, corrected to the standard temperature of  $V_{TD}$  20 °C, is computed, using the expression

$$V_{TD\ 20^\circ C} = V_{TDM} [1 - (t-20)(a_s - a_x)]$$

where

$V_{TDM}$  = observed average volume as computed in 3.1

$t$  = temperature of water at time of calibration

$a_s$  = cubical coefficient of expansion of borosilicate glass,  
0.000010/°C

$a_x$  = cubical coefficient of expansion of glass of flask, e.g.,  
0.000025/°C for soda-lime glass

# APPENDIX

## CALIBRATION OF MEASURING FLASK (VOLUME TRANSFER METHOD)

Test No.: \_\_\_\_\_ Date: \_\_\_\_\_ Observer: \_\_\_\_\_

Vessel Calibrated: \_\_\_\_\_

Vessel Identification No.: \_\_\_\_\_

Requested By: \_\_\_\_\_

### STANDARDS USED

Standard Pipet No.: \_\_\_\_\_ Calibrated Volume,  $V_p$ : \_\_\_\_\_

Standard Buret No.: \_\_\_\_\_

Calibration Corrections: \_\_\_\_\_

	TRIAL NO. 1	TRIAL NO. 2
$V_p$	_____	_____
Buret	_____	_____
Final Reading	_____	_____
Initial Reading	_____	_____
Difference	_____	_____
Correction Required	_____	_____
Corrected Volume $V_B$	_____	_____
$V_{TD} = V_p + V_B$	_____	_____
Average $V_{TDM}$	_____	

### TEMPERATURE CORRECTION

Water Temperature: \_\_\_\_\_

Average Water Temperature,  $t$ : \_\_\_\_\_

$a_s =$  \_\_\_\_\_  $a_x =$  \_\_\_\_\_

$(a_s - a_x) =$  \_\_\_\_\_  $t - 20\text{ }^{\circ}\text{C} =$  \_\_\_\_\_

$A = (a_s - a_x) (t - 20\text{ }^{\circ}\text{C}) =$  \_\_\_\_\_  $B = 1 - A =$  \_\_\_\_\_

$V_{TD20\text{ }^{\circ}\text{C}} = V_{TDM} \times B =$  \_\_\_\_\_



## SOP No. 17

### Recommended Standard Operations Procedure for Control Charts for Calibration of Measuring Flasks Volume Transfer Method

#### 1. Introduction

##### 1.1. Purpose

This procedure may be used to develop and maintain control charts to monitor the statistical control of the volume transfer method for calibration of measuring flasks, especially when using the Recommended Standard Operations Procedure (SOP No. 16) for this purpose. The same principles may be applied to the development of control charts for other calibration procedures.

##### 1.2. Prerequisites

1.2.1. The verification procedure is the same as that required by SOP No. 16.

1.2.2. The quality assurance reference flask (QARF) (see 3.1) must be scrupulously clean at each time of use. Verify that this is true or take corrective actions.

#### 2. Summary

A reference volumetric flask (or a series of such) is obtained and calibrated several times initially to establish a reliable mean value and to estimate the standard deviation of calibration. All such calibrations are made using SOP No. 16 or an equivalent procedure. Directions for preparing and using an  $\bar{X}$  and an R control chart are given. The  $\bar{X}$  control chart monitors the process with respect to both systematic and random errors while the R control chart monitors its short-term precision. When the calibration process is judged to be in a state of statistical control, the calibrations made at that time may be considered to be valid and the process standard deviation may be used, as appropriate, to establish confidence intervals for the calibrations made using the SOP.

#### 3. Equipment

3.1. A quality assurance reference flask(s) (QARF) is required, constructed of borosilicate glass and dimensionally similar to the flasks under calibration. A pint and a  $\frac{1}{2}$  gallon flask or a  $\frac{1}{2}$  pint and 1 quart are recommended.

3.2. All equipment designated in Section 2.3 of SOP No. 16.

#### 4. Procedure

##### 4.1. Initial Measurements

- 4.1.1. Calibrate the QARF a minimum of 12 times. A calibration is defined as the result of duplicate measurements as required by the SOP. Calibrations may be made on successive days, but no two may be made on any single day.
- 4.1.2. Tabulate the measurement data using the notation and a form such as the one contained in the Appendix of this SOP.
- 4.1.3. Calculate the means of the two trials  $\bar{X}_i$  and the ranges  $d_i$  for the  $k$  tests.
- 4.1.4. Calculate the average difference  $|\bar{d}|$  of the trials, for the  $k$  tests as follows:

$$|\bar{d}| = \Sigma |d| / k$$

- 4.1.5. Estimate the total standard deviation  $s$ , based on  $\bar{X}_i$ , the average measured volume of the QARF on each of  $k$  occasions as follows:

$$s = \sqrt{\frac{\Sigma (\bar{X}_i - \bar{X})^2}{k-1}}$$

##### 4.2. Construction of Control Charts

Construct the following control charts using the data of section 4.1.

- 4.2.1. Construct an  $\bar{X}$  control chart having the following control limits.

$$\text{Central Line} = \bar{X}$$

$$\text{Lower warning limit (LWL)} = \bar{X} - 2 s$$

$$\text{Lower control limit (LCL)} = \bar{X} - 3 s$$

$$\text{Upper warning limit (UWL)} = \bar{X} + 2 s$$

$$\text{Upper control limit (UCL)} = \bar{X} + 3 s$$

- 4.2.2. Construct an  $R$  control chart for duplicate measurements having the following control limits. Note that  $R$  (the range) and  $|d|$  (absolute difference of duplicate measurements) are equivalent for duplicate measurements.

$$\text{Central Line} = \bar{R}$$

$$\text{LCL} = \text{LWL} = 0$$

$$\text{UWL} = 2.512 \bar{R}$$

$$\text{UCL} = 3.267 \bar{R}$$

#### 4.3. Use of Control Charts

- 4.3.1. An appropriate QARF is recalibrated each time the laboratory performs calibrations using the SOP. If the calibrations extend over several days, the QARF is recalibrated, daily. The values of  $\bar{X}$  and R for each recalibration of the QARF are plotted on the respective control charts, preferably in sequential order. The limits on the charts are such that 95% of the values should fall within the warning limits and rarely should a value fall outside of the control limits, provided that the system is in a state of statistical control.
- 4.3.2. If the plotted value of  $\bar{X}$  lies outside of the control limits and the corresponding value on the R chart is within the control limits, a source of systematic error is suspected. Poor drainage is a possible source of bias for low values and changes of drainage technique should be considered as a cause for high values. Sources of error can result from the use of the volumetric standards as well as of the vessels being calibrated. Changes in criteria for reading menisci are also possible sources of bias.
- 4.3.3. If the values for the R chart fall outside of the warning limits but inside of the control limits, decrease in precision is indicated. Cleanliness and procedural problems should be investigated.
- 4.3.4. No calibration data should be accepted when the system is out of control.
- 4.3.5. If the plotted values for either  $\bar{X}$  or d are outside of the warning limits but inside of the control limits, a second set of duplicate calibrations should be made. If the new values are within the warning limits, the process may be considered to be in control. If they lie outside of the warning limits, lack of control is indicated. Corrective actions should be taken and attainment of control demonstrated before calibration measurements are considered to be acceptable.
- 4.3.6. Even while the system is in an apparent state of control, incipient troubles may be indicated when the control data show short- or long-term trends, shifts, or runs. The t-test and F-test may be used in judgment of the significance of such observations (see Chapters 8.9, 8.10, and 8.11).

#### 5. Interpretation of Control Chart Data

- 5.1. Demonstration of "in control" indicates that the calibration process is consistent with the past experience of the laboratory. That is to say, there is no reason to believe that excessive systematic error or changes in precision have occurred.

- 5.2. To the extent appropriate, the precision of measurement of the QARF may be extended to the calibration of other glassware. This means that the process standard deviation,  $s$  is transferable to all similar measurements made by the measurement system.
- 5.3. Extension of the  $s$  for the QARF to other calibrations assumes that all aspects of its calibration correspond to those for the other calibration. If the volumes of the respective glassware are comparable as well as the sizes of necks and fiducial lines, this may be justified. It may be necessary to have a series of QARF's with corresponding control charts to monitor the calibration process for a range of volumetric calibrations.

# Appendix

Control Chart Data  
 QARF No. \_\_\_\_\_  
 Nominal Capacity \_\_\_\_\_  
 Laboratory \_\_\_\_\_

Test No.	Date	V <sub>TD</sub> , X <sub>1</sub> Trial No. 1	V <sub>TD</sub> , X <sub>2</sub> Trial No. 2	V <sub>TDM</sub> ( $\bar{X}$ )	d  =  Trial No. 1 - Trial No. 2 *
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
SUM				$\Sigma \bar{X}$	$\Sigma  d $

$$k^{**} = \frac{\bar{X}}{s} = \frac{\Sigma \bar{X}}{k} =$$

$$\bar{R} = \frac{\Sigma |d|}{k} = \quad \text{UWL} = 2.512 \bar{R} =$$

$$s = \sqrt{\frac{\Sigma (\bar{X}_i - \bar{X})^2}{k - 1}} \quad \text{UCL} = 3.267 \bar{R} =$$

\*This is the range, R, of the two trials and is actually the larger value minus the smaller value.

\*\*k is the number of tests used to calculate the control limits.





## SOP No. 18

### Recommended Standard Operations Procedure for Calibration of Graduated Neck-Type Metal Volumetric Field Standards Volumetric Transfer Method

#### 1. Introduction

##### 1.1. Purpose of Test

This procedure may be used to calibrate small non-pressurized, graduated neck-type, metal field standards such as the 5-gallon standard used by weights and measures officials to test liquid dispensing equipment, such as gasoline pumps, for example.

##### 1.2. Prerequisites

- 1.2.1. Verify that the standards used have been calibrated and that a valid certificate is available.
- 1.2.2. Verify the cleanliness of the water to be used.
- 1.2.3. Verify that the person performing the calibrations is trained and proficient in carrying out this test procedure.

#### 2. Methodology

##### 2.1. Scope, Precision, Accuracy

This procedure is applicable for the calibration of a small test measure within the limitations of the standards available. The precision attainable will depend on the care used in the various volumetric adjustments and readings, in the strict observance of drainage times, and the internal cleanliness of the various volumetric vessels which can influence their drainage characteristics. The accuracy will depend on the uncertainty of the calibrations of the standards used.

##### 2.2. Summary

Water is delivered from the standard to the vessel under calibration. Because the "to deliver" volume of the latter is calibrated, the delivery must be into a "wet down" vessel. The gauge scale is adjusted to a correct reading, as necessary, and then sealed.

##### 2.3. Equipment

- 2.3.1. Slicker-plate standard made of stainless steel, with volume equal to that of the vessel to be calibrated.
- 2.3.2. Supply of clean water

## 2.4. Procedure

### 2.4.1. Cleanliness Verification

Fill and drain both standard and vessel to be calibrated and check for any soiling that would affect drainage, as evidenced by clinging droplets, greasy films, and the like. Clean either or both with detergent and water, as necessary, and rinse thoroughly. (See GMP No. 6)

2.4.2. Fill vessel with water to its nominal level and dump contents during a  $30 \pm 5$  second period then drain for a 10-second period after cessation of flow. Touch off any adhering drop from the neck. This constitutes the "wet-down" condition.

2.4.3. Fill slicker-plate standard with water, raised by surface tension slightly higher than the rim. Use slicker-plate to strike off a precise volume, checking to see that no air bubbles are entrained during the leveling process.

2.4.4. Open valve at base and transfer water from the standard to the wet-down vessel. Allow a 30-sec drain period after cessation of flow.

2.4.5. Level vessel (or suspend it by its handle, if appropriate) and read scale on its neck. Record reading.

2.4.6. Make a duplicate determination which should agree with the former within  $\pm 0.2 \text{ in}^3$ . If excess disagreement, check all vessels for cleanliness and repeat duplicate determinations until satisfactory agreement is obtained.

2.4.7. Adjust the scale of the vessel as described in 3.3. Seal the scale adjustment device.

## 3. Calculations

3.1. Because the water temperature is usually reasonably close to 60 °F, the coefficients of expansion of the standard and the test vessel are sufficiently close together, and the deliveries and readings are made over a short period of time, no temperature corrections are made.

3.2. Within the accuracy requirements, no corrections arising from dissimilarities of the standard and vessel are necessary.

3.3. The average of the duplicate readings is used to adjust the scale of the vessel to the correct reading which is set at the calibrated volume of the slicker-plate standard at 60 °F.

Note: If the accuracy requirements necessitate a temperature correction, the temperature of the water used must be measured and the correction is made according to the procedure given in SOP No. 19.

#### 4. Assignment of Uncertainty

- 4.1. The calibration uncertainty,  $U$ , is estimated, using the expression

$$U = \pm [U_s + U_m]$$

where  $U_s$  = uncertainty of the calibration standards used  
(ordinarily, the uncertainty of  $V_p$ )

$U_m$  = uncertainty of the measurement process

- 4.2. The uncertainty,  $U_m$ , of  $V_{TDM}$  is estimated from the expression

$$U_m = ts$$

- 4.3. If a control chart has been maintained for this measurement procedure, the value for  $s$  may be based on the control chart (see Recommended Standard Operations Procedure, SOP No. 17, Control Charts for Calibration of Measuring Flasks), and  $t$  is obtained from Table 9.3, corresponding to the 99.73% level of confidence and the number of degrees of freedom associated with the standard deviation  $s$ .

When a control chart is not available, the value of  $s$  may be estimated from the results of at least 12 sets of duplicate calibrations as described in SOP No. 17.





Recommended Standard Operations Procedure for  
Calibration of Large Neck-Type Metal Provers Volumetric Method

1. Introduction

1.1. Purpose of Test

This procedure is used to calibrate large neck-type metal provers (10 gal and larger).

1.2. Prerequisites

1.2.1. Verify that the standards to be used have been calibrated and that a valid certificate is available.

1.2.2. Verify cleanliness of water to be used.

1.2.3. Verify that the person performing the calibrations is trained and proficient in carrying out this procedure.

2. Methodology

2.1. Scope, Precision, Accuracy

This procedure is applicable for the calibration of any size metal prover within the limitations of the standards available. The precision attainable will depend on strict adherence to the procedure, to the care in volumetric adjustments, and to the number of transfers, in the case of multiple transfers. The accuracy will depend on the standards used.

2.2. Summary

Water is delivered from a volumetric standard to the prover being calibrated. Depending on the respective volumes, multiple transfers may be required. While these should be minimized, a maximum number of 15 transfers is permitted. The temperature cannot be considered to be constant during multiple transfers, hence the temperature of the water for each transfer must be measured. Because of the large volumes, the difference in thermal expansion of the respective vessels must be considered.

2.3. Equipment

2.3.1. Calibrated volumetric standard.

2.3.2. Thermometer, calibrated to 0.5 °C.

2.3.3. Supply of clean water.

## 2.4. Procedure

### 2.4.1. Cleanliness Verification

Fill and drain both standard and prover to be calibrated and check for visual evidence of soiling and of improper drainage. If necessary clean with detergent and water (see GMP No. 6).

2.4.2. Fill prover with water and level it. Drain water, then wait 30 seconds after cessation of flow, before closing drain valve. This establishes "wet down" condition.

2.4.3. Fill standard and carefully adjust to reference mark or record the neck reading.

2.4.4. Measure and record temperature, then discharge into prover. Wait 30 seconds after cessation of flow to attain specified drainage, then close delivery valve.

2.4.5. Repeat step 2.4.4 as many times as necessary to fill prover to its nominal level. Level the prover if necessary and record the neck reading. Measure temperature of water in prover and record.

2.4.6. Discharge water from prover to obtain "wet down" as in 2.4.2, then repeat calibration.

2.4.7. Perform calculations as described in section 3.0. Compare duplicate results which should agree within 0.02 percent. If excessive disagreement is found, clean and take other corrective actions as necessary, then recalibrate until duplicate determinations agree within 0.02 percent.

2.4.8. Adjust scale and seal equipment.

## 3. Calculations

The basic equation for use to calculate the volume of the prover is as follows:

$$Z_{60} = \frac{\rho_A(U_{60}[1+\alpha(t_A-60)]+\Delta_A)+\rho_B(U_{60}[1+\alpha(t_B-60)]+\Delta_B)+\dots+\rho_N(U_{60}[1+\alpha(t_N-60)]+\Delta_N)}{\rho_Q[1+\beta(t_Q-60)]}$$

where

- $\rho_{A,B,\dots, N}$  - density of water at the several transfers
- $t_{A,B,\dots, N}$  - temperature of water at the several transfer
- $\alpha$  - coefficient of cubical expansion of the standard
- $\beta$  - coefficient of cubical expansion of the prover
- $U_{60}$  - calibrated volume of the standard at 60 °F
- $Z_{60}$  - volume delivered from the prover at 60 °F
- $t_Q$  - temperature of water in prover after final transfer
- $\Delta_A, \Delta_B, \dots, \Delta_N$  - volume difference between water level and the reference mark on the standard where the subscripts A, B, ..., N represent each delivery as above. If the water level is below the reference line,  $\Delta$  is negative. If the water level is at the reference line,  $\Delta$  is zero

#### 4. Assignment of Uncertainty

- 4.1. The calibration uncertainty,  $U$ , is estimated, using the expression

$$U = \pm [U_s + U_m]$$

where  $U_s$  = uncertainty of the calibration standards

$U_m$  = uncertainty of the measurement process

- 4.2. The uncertainty,  $U_m$ , of  $Z_{60}$  is estimated from the expression

$$U_m = \frac{ts}{\sqrt{2}}$$

- 4.3. If a control chart has been maintained for this measurement procedure, the value for  $s$  may be based on the control chart (see Recommended Standard Operations Procedure, SOP No. 20, Control Charts for Calibration of Neck-Type Provers), and  $t$  is obtained from Table 9.3, corresponding to the 99.73% level of confidence and the number of degrees of freedom associated with the standard deviation.

When a control chart is not available, the value of  $s$  may be estimated from the results of at least 12 sets of duplicate calibrations as described in SOP No. 20.



## SOP No. 20

### Recommended Standard Operations Procedure for Control Charts for Calibration of Neck-Type Provers (Volumetric Method)

#### 1. Introduction

##### 1.1. Purpose

This procedure describes a process to be followed to monitor the statistical control of the volumetric method for the calibration of neck-type provers, especially when using the Recommended Standard Operation Procedures (SOP) No. 19 for this purpose. The same principles may be adapted to the development of control charts for other calibration procedures.

##### 1.2. Prerequisites

The verification procedure is the same as that required by the SOP No. 19.

#### 2. Summary

Because of the size and cost of neck-type provers, it is not practical to have check standards remain in the laboratory for the purposes of measurement control. However, it is practical to maintain a range (R) chart for each size prover to establish the precision of the measurement process. Directions for preparing and using an R control chart that monitors the precision of the test procedure are given. It is assumed that provers of the same nominal capacity and design will have similar characteristics with respect to the repeatability of tests. Since it is not practical to run a sufficient number of tests on each unknown prover to determine the repeatability, the absolute difference between two test results on individual provers of the same nominal size are graphed on the same R chart to reflect the repeatability of measurement of the provers tested in the laboratory.

#### 3. Equipment

All equipment is designated in Section 2.3 of SOP No. 19.

#### 4. Procedure

##### 4.1. Data Collection

4.1.1. Conduct two calibrations on each prover. A minimum of 12 provers must be tested before a reasonably adequate data base is established.

4.1.2. Tabulate the measured errors as determined by each of the two trials using a form such as the one contained in the Appendix. (If the prover is adjusted after the first trial to indicate zero, the first trial reading is zero.)



4.1.3. Calculate the absolute difference  $|d|$  of the two trials and the summation  $\Sigma|d|$ . Note that  $|d| = R$ , the range of the two trials.

4.1.4. Calculate the average range of the trials,  $\bar{R}$ , for the  $k$  tests as follows:

$$\bar{R} = \frac{\Sigma|d|}{k}$$

#### 4.2. Construction of the R Control Chart

4.2.1. Construct an R control chart having the following limits:

Central Line =  $\bar{R}$

Lower control and warning limits LCL = LWL = 0

Upper warning limit UWL =  $2.512\bar{R}$

Upper Control limit UCL =  $3.267\bar{R}$

4.2.2. The recommended format for construction of R control charts is given in Chapter 7.4.

#### 4.3. Use of Control Charts

4.3.1. Two trials are run on each prover submitted to the laboratory for certification. The values for R are plotted on the appropriate control chart, preferably in sequential order. The limits of the charts are such that 95 percent of the values should fall within the warning limits and rarely should a value fall outside the control limits, provided that the system is in a state of statistical control.

4.3.2. If the values plotted on the R chart fall outside of the control limit, a decrease in precision is indicated. Cleanliness and procedural problems should be investigated.

4.3.3. No calibration data should be accepted when the system is out of control.

4.3.4. If a plotted value for R is outside of the warning limit but inside the control limit, a second set of duplicate calibrations should be made. If the new value for R is within the warning limit, the process may be considered in control. If it lies outside of the warning limit, lack of control is indicated. Corrective actions should be taken and attainment of control demonstrated before calibration measurements are considered to be acceptable.

4.3.5. Even while the system is in an apparent state of control, incipient troubles may be indicated when the control data show short- or long-term trends, shifts, or runs.

## 5. Interpretation of Control Chart Data

- 5.1. Demonstration of "in control" indicates that the calibration process is consistent with the past experience of the laboratory. That is to say, there is no reason to believe that excessive changes in precision have occurred.
- 5.2. The accuracy is inferred from a consideration of control of the sources of bias. These include drain characteristics of the prover, time required to drain the prover, and prover design (geometry and piping).
- 5.3. To the extent appropriate, the precision of measurement of provers may be extended to the calibration of other provers of the same capacity and design. Care must be exercised in this approach because each prover is custom made and has unique metrological characteristics. The lack of precision in the test of a prover may indicate a defect in that particular standard and may not reflect inconsistency in the measurement process.
- 5.4. It is expected that the maximum imprecision (the upper control limit) of the volumetric method of prover calibration will be less than one-half of the prover tolerance. If this is not the case, contact the NBS Office of Weights and Measures to discuss the problem.

# Appendix

Control Chart Data  
Prover Capacity \_\_\_\_\_  
Laboratory \_\_\_\_\_

Test No.	Date	V <sub>TD</sub> , X <sub>1</sub> Trial No. 1	V <sub>TD</sub> , X <sub>2</sub> Trial No. 2	V <sub>TDM</sub> ( $\bar{X}$ )	d  =  Trial No. 1 - Trial No. 2 *
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
SUM				$\Sigma \bar{X}$	$\Sigma  d $

$$k^{**} = \underline{\hspace{2cm}} \quad \bar{R} = \frac{\Sigma |d|}{k} = \underline{\hspace{2cm}}$$

$$UWL = 2.512\bar{R} = \underline{\hspace{2cm}} \quad UCL = 3.267\bar{R} = \underline{\hspace{2cm}}$$

\*This is the range, R, of the two trials and is actually the larger value minus the smaller value.

\*\*k is the number of tests used to calculate the control limits.

Recommended Standard Operations Procedure for  
Calibration of LPG Provers

1. Introduction

1.1. Purpose of Test

This procedure may be used to calibrate or ascertain the accuracy of a volume standard used to test systems designed to measure and deliver liquefied petroleum gas (LPG) in the liquid state by definite volume, whether installed in a permanent location or mounted on a vehicle. A schematic diagram of such a prover is shown in Figure 1, together with numbers, e.g., 1, to clarify the various operations described in the procedure. The parts labeled A, B, and C are hose connections used in meter testing.

1.2. Prerequisites

- 1.2.1. Verify that valid calibration certifications are available for the standard prover and the thermometers to be used.
- 1.2.2. Verify the availability of an adequate supply of clean water.
- 1.2.3. Verify that the person performing the calibration is trained and proficient in carrying out this procedure.

2. Methodology

2.1. Scope, Precision, Accuracy

This procedure is applicable for the calibration of LPG provers with capacities of 20 to 100 gal. Provers of the latter capacity are encountered most frequently, hence the procedure is written with that in mind. The changes necessary for testing provers of other capacities will be obvious, hence are not described. The agreement of duplicate measurements made within a short period of time on a given 100-gal prover should be within 5 in<sup>3</sup> (0.02 gal). The accuracy will depend on the uncertainty in the volume of the standard, on the care exercised in making the various measurements and temperature readings, and the corresponding corrections. The overall uncertainty, in the case of an experienced technician, is not expected to exceed 10 in<sup>3</sup>.

2.2. Summary

The procedure is a modification of one described by M.W. Jensen in NBS Handbook 99, "Examination of Liquefied Petroleum Gas Liquid-Measuring Devices." The LPG prover is calibrated with a known volume of water delivered into it from a standard prover of calibrated volume. The LPG prover is pressurized and the liquid level is

measured at each of several values of applied pressure. The calibration thus defines the capacity of the prover over its expected range of operational pressure.

## 2.3. Equipment

- 2.3.1. Standard calibrated prover (hereinafter referred to as the standard) of minimum volume of 10 gallon but preferably of the same volume as the LPG prover (hereinafter referred to as the prover). Multiple transfers (not exceeding 15) are permissible, but this practice should be avoided or minimized to the extent possible.
- 2.3.2. Calibrated 1-gallon flask to calibrate neck of prover.
- 2.3.3. Thermometers(2) accurately calibrated to 0.5 °C.
- 2.3.4. Supply of clean water, preferably soft water (filtered if necessary).
- 2.3.5. Sturdy platform of sufficient height to hold standard and to permit transfer of water from it to the prover by gravity flow.
- 2.3.6. Clean pipe or tubing of large diameter facilitate transfer of water from standard to prover.

## 2.3. Procedure

### 2.4.1. Preliminary Operations

- 2.4.1.1. Install and level standard on raised platform. Provide pipe or tubing for delivery of water by most direct route to prover.
- 2.4.1.2. Position and level prover where it can be reached from the elevated standard by the shortest feasible delivery system.
- 2.4.1.3. Remove the plug and relief valve (1) in the top, and extend the pipe into the hole. This may require the use of a reducer and a short length of hose (about 1 inch in diameter). If this is a tight fit, open the vapor return line valve (2) to provide an air bleed.

Use the prover inlet line (3) as a gravity drain. If necessary, remove the brass fitting on the end and connect a hose or pipe to make the necessary drain line.



#### 2.4.2. Cleanliness Check

Both the standard and the prover must be internally clean. This should be verified by checking that water drains properly from them. If necessary, either or both should be cleaned with water and detergent (see GMP No. 6) to attain good drainage characteristics.

#### 2.4.3. Neck Calibration

2.4.3.1. Fill the prover with water from the standard. Check the prover levels and adjust if necessary. Check the prover system for leaks. This is a wet-down run.

2.4.3.2. Bleed the liquid level down to a graduation near the bottom of the upper neck. "Rock" the prover to "bounce" the liquid level, momentarily, to ensure that it has reached an equilibrium level. Read and record this setting. This is in preparation for calibration of the neck scale.

2.4.3.3. Remove the fill hose or pipe from the top and insert a funnel.

2.4.3.4. Recheck the scale reading, then add one gallon of water from a suitable standard; record scale reading.

2.4.3.5. Repeat 2.4.3.4 by successive additions until water is near the top of the scale (the neck capacity is usually about 5 gallons). The closer the water is to the top of the neck, the harder it may be to "bounce" the liquid in the gauge.

2.4.3.6. Calculate and check accuracy of the neck scale. The error should be less than 2 percent. If more than this, the scale should be replaced or a correction factor should be computed and applied to all future readings.

This factor is calculated as follows:

$$\text{Neck scale correction factor} = \frac{\text{Volume of water added to neck}}{\text{Final scale reading} - \text{Initial scale reading}}$$

#### 2.4.4. Body Calibration

2.4.4.1. Drain the prover using its inlet valve as a control. When the liquid reaches the top of the lower gage glass, close the valve and allow the water to drain from the interior of the prover into

the lower neck for 30 seconds. Then bleed slowly with the bleed valve (4) until the bottom of the liquid meniscus reaches the zero graduation. (This step should be started during the 30 second drain period but should not be completed before the end of the drain period.)

2.4.4.2. Transfer 100 gallons (or other suitable volume) from the standard in the usual manner, and record the standard and prover temperature readings. If multiple transfers are required, record temperature of the standard at the time of each transfer, but that of the prover after the final transfer. "Rock" the prover to "bounce" the liquid in the upper gauge glass before reading. Record scale reading.

2.4.4.3. Drain as described in 2.4.4.1 and make another test run. Record temperatures and final scale reading. The two test runs are expected to agree within one-half the prover tolerance (5 in<sup>3</sup> on a 100-gallon prover). If significantly different from this, continue until replicates agree within these limits.

#### 2.4.5. Pressure Correction

2.4.5.1. Replace the relief valve and plug in the top of the prover using suitable pipe joint compound or tape. Allow water from 2.4.4.2 to remain in prover.

2.4.5.2. Use a cylinder of nitrogen and a proper pressure regulator.

Connect the cylinder to the vapor return fitting (2) near the top of the neck. This may require fashioning a connection with copper tubing and existing fittings.

Make sure all valves are closed except the vapor return valve. Check the scale reading then slowly introduce pressure until the prover gauge reads 50 psig. Lightly tap the gauge to vibrate the needle as it has a tendency to stick.

"Bounce" the liquid in the neck, then read and record the liquid level at this applied pressure.

2.4.5.3. Repeat step 2.4.5.2 at 100, 150, and 200 psig. Other pressure points in between those listed may be tested if so desired. There should be a drop of approximately 10 to 15 in<sup>3</sup> for each 50 psig.

2.4.5.4. Repeat step 2.4.5.2 as the pressure is bled down to 150, 100, 50, and 0 psig (atmospheric pressure). The readings should agree with those previously obtained within approximately 2 in<sup>3</sup>. If the data are not linear with respect to pressure, repeat the series of measurements above to verify the nonlinearity of the readings.

#### 2.4.6. Prover Adjustments and Corrections

2.4.6.1. The internal pressure and hence the volume of the prover may vary during use. Accordingly, a pressure correction must be made using the data of steps 2.4.5.

To minimize the amount of correction needed when the prover is in use, the prover should be adjusted to indicate exactly its nominal capacity with 100 pounds of internal pressure. (An internal pressure of 100 psig is suggested as being convenient.) If the actual volume of the prover is not near a convenient whole gallon value and cannot easily be adjusted to a whole gallon value, a prover correction value can be computed (see 3.4) and added to the pressure correction values to obtain a set of combined prover and pressure correction values to be computed. The pressure correction is computed in 3.4.

2.4.6.2. With the pressure in the prover at 100 psig, adjust the upper scale to read the nominal volume. This is accomplished by adjusting the upper scale so that the water level reading is  $Z_{60} - V_{NOM} - 7.8 \text{ in}^3$ . (The factor 7.8 in<sup>3</sup> corrects for the compressibility of the water, for a prover of 100 gal capacity. For provers of other capacities, the factor is  $0.078 V_{\text{prover}}$ . If the upper scale is not adjustable, see 2.4.6.3.

2.4.6.3. For provers with an adjustable lower scale, compute the prover correction,  $L_C$  at 100 psig as follows.

$$L_C = Z_{60} - V_{NOM} - 7.8 \text{ in}^3 - \text{upper scale reading at 100 psig.}$$

If the prover correction is negative, move the bottom scale down to increase the prover volume. If the prover correction is positive, move the bottom scale up to decrease the prover volume. The distance  $h$  that the bottom scale is to be moved is

$$h = \frac{4|\text{Prover correction at 100 psig}|}{\pi d^2}$$

where d is the inside diameter of the lower neck of the prover.

#### 2.4.7. Final Operations

2.4.7.1. Seal the bottom and top scales as appropriate.

2.4.7.2. Drain prover, then remove plug (5) at the lower neck to facilitate drainage below the lower gauge. If time permits, let the prover drain overnight.

2.4.7.3. With the nitrogen cylinder connected, blow nitrogen through the prover to remove remaining moisture. Be sure to blow out drain line.

2.4.7.4. If water has entered the pump-off system, pour several gallons of alcohol into the prover and pump the alcohol through the system to remove the water to prevent it from freezing in the pump when LP gas is used.

### 3. Calculations

#### 3.1. Single Delivery

Calculate  $Z_{60}$ , the volume of the prover at 60 °F, using the following equation:

$$Z_{60} = \frac{\rho_A (U_{60} [1 + \alpha (t_A - 60)] + \Delta_A)}{\rho_Q [1 + \beta (t_Q - 60)]}$$

See 3.3 for explanation of the symbols

#### 3.2. Multiple Deliveries

Calculate  $Z_{60}$ , the volume of the prover at 60 °F, using the following equation

$$Z_{60} = \frac{\rho_A (U_{60} [1 + \alpha (t_A - 60)] + \Delta_A) + \rho_B (U_{60} [1 + \alpha (t_B - 60)] + \Delta_B) + \dots + \rho_N (U_{60} [1 + \alpha (t_N - 60)] + \Delta_N)}{\rho_Q [1 + \beta (t_Q - 60)]}$$



### 3.3. Symbols Used in 3.1 and 3.2

$Z_{60}$  = volume of the unknown vessel at 60 °F

$U_{60}$  = volume of the standard vessel at 60 °F

$\rho_A, \rho_B, \dots, \rho_N$  = density of the water in the standard where  $\rho_A$  is the density of the water for the first delivery,  $\rho_B$  is the density of the water for the second delivery, and so on until all N deliveries are completed

$\Delta_A, \Delta_B, \dots, \Delta_N$  = volume difference between water level and the reference mark on the standard where the subscripts A, B, ..., N represent each delivery as above. If the water level is below the reference line,  $\Delta$  is negative. If the water level is above the reference line,  $\Delta$  is positive. If the water level is at the reference line,  $\Delta$  is zero

$t_A, t_B, \dots, t_N$  = temperature of water for each delivery with the subscripts as above

$\alpha$  = coefficient of cubical expansion for the standard

$\beta$  = coefficient of cubical expansion for the prover

$t_Q$  = temperature of the water in the filled prover

$\rho_Q$  = density of the water in the prover

Note: Values for the density of water at the respective temperatures may be found in Table 9.8.

### 3.4. Pressure Corrections

Compute the pressure correction,  $P_{corr}$ , at each pressure that the prover was read by correcting for the compressibility of the water. The equation is

$$P_{corr} = \text{Scale reading at 100 psig} - \text{scale reading at other pressure} \\ + (\text{water compressibility factor}) \frac{(100 - \text{other pressure})}{100}$$

where the water compressibility factor is 7.8 in<sup>3</sup> or 0.03376 gal for a 100 gallon prover. The compressibility factor is given in both cubic inches and gallons so the proper unit can be selected depending upon the unit used for the scale readings.

Plot the pressure corrections. If the corrections versus the pressure is linear, make a straight line best fit of the data and interpolate to obtain the pressure corrections for any desired pressure. If the data is nonlinear, then perform a straight line interpolation between adjacent pressure readings to obtain pressure corrections at any desired intermediate pressures. A best fit curve can be drawn for the nonlinear data and the pressure corrections interpolated from the graph for intermediate pressures.



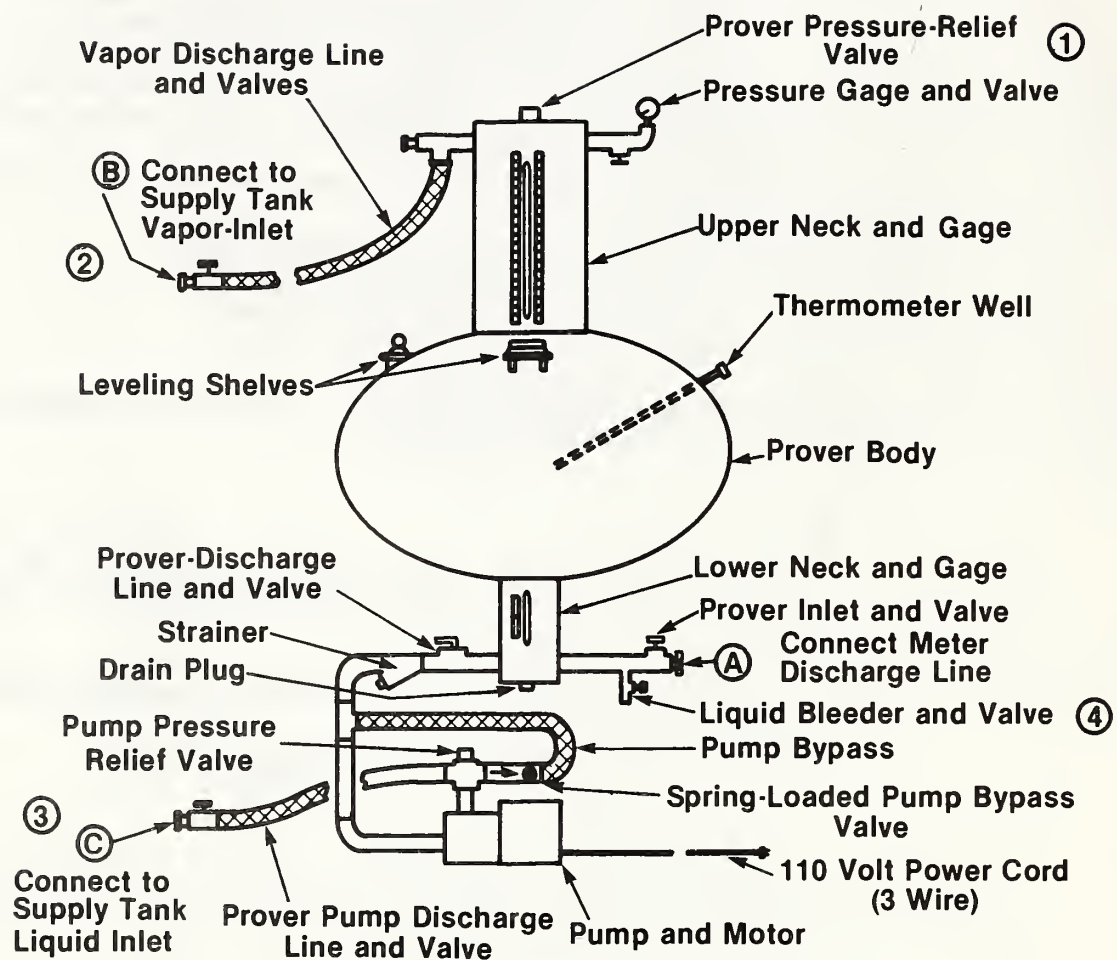


Figure 1. Volumetric LP Gas Meter Prover (Schematic).

## 7. MISCELLANEOUS SUBJECTS

### 7.1 NBS Support for the National Measurement System

Measurements are essential for almost every aspect of human existence. They are the keystone of our modern civilization. Measurements are always estimates of the true value of some property and have some degree of uncertainty which ordinarily needs to be known. Moreover, measurements made by various individuals and/or at various times need to be relatable and compatible. This requires that measurements must be made systematically in what may be called a national measurement system. The basic operational aspects of the national measurement system are depicted in Figure 7.1. The upper part of the figure represents physical measurements while the lower part relates to chemical measurements. Essentially one can visualize samples of materials that are measured to obtain data on some physical or chemical property that is needed for an end use. The measurement is made with respect to some physical or chemical standard. The basis for all measurements are the seven basic units shown in the table below. These are internationally accepted and NBS maintains national primary standards compatible with the international standards for fixing the magnitude of the units.

Table -- Units of Measurements

#### Basic

Length -	meter
Mass -	kilogram
Time -	second
Electric Current	ampere
Temperature -	kelvin
Amount of Substance	mole
Luminous Intensity	candela

#### Supplementary

Plane Angle -	radian
Solid Angle -	steradian

A hierarchy of standards can be envisioned, each relatable and hence traceable to the ones above it as shown in Figure 7.2. It is possible to use working standards for applied measurements with calibrations traceable to the national standards in virtually all areas of physical measurements. The traceability may be because of NBS calibrations or by measurements by others (a State weights and measures laboratory, for example) using higher hierarchy standards with certified values traceable to NBS. Obviously, the intercomparison measurements that are required need to be made with a high degree of reliability since the uncertainty of measurement must be added to the uncertainty of the calibrated value of the standards used.

The propagation of error in a measurement chain is illustrated in Figure 7.2. After several iterations, a lower hierarchy standard (LHS) may have an intolerable uncertainty for some uses due to the uncertainties associated with intervening calibration steps so that a user may need to have a particular

working standard calibrated closer to the primary standard (see chain C in Figure 7.2). In such a case, a calibration laboratory higher in the hierarchy must provide the service (or use of a standard higher in the hierarchy is another possibility). Of course, NBS provides calibration services in some cases.

In consideration of the above, it should be noted that physical standards may need to meet rigid specifications as to their form, material, workmanship, and stability to qualify for use as upper hierarchy standards.

Physical measurements may be considered in two general classes. The first consists of those made to fix the values of measurement standards and can be called calibration and tolerance testing. The second is applied measurement and includes all but those in the first. Indeed, one might consider that the first is really a special class of the second kind. In both cases, measurement essentially is a comparison of an unknown with a standard and the uncertainty of the result includes that of the standard used together with the uncertainty of the intercomparison. Whether for calibration or for application, it behooves every metrologist and every measurement laboratory to minimize the uncertainty of its measurements. Improvements can be sought in two directions. Higher quality standards will reduce the systematic error (heavy lines of Figure 7.2) while improvements in precision (dotted lines of 7.2) can result from better quality control and/or increasing the number of replications. While replication can make random error uncertainties small, there is a practical upper limit to which this can be done. In high quality measurement, the two kinds of uncertainties may equal each other but the systematic should not exceed the random component. Reduction of systematic uncertainty to one-third of the random uncertainty is often practical in which case it does not contribute appreciable error to a measurement process.

NBS supports the measurement process as shown by Figure 7.3 by maintaining the basic primary standards and by calibrating or otherwise providing routes to them for traceability of working standards. NBS conducts fundamental research to increase understanding of measurement in its broadest interpretation. Along with others, NBS investigates and develops new and or improved methods of measurement and provides reference materials to evaluate the measurement process.

The responsibility for the reliability of any specific measurement is that of the metrologist/laboratory that reports it. A measured value without limits of uncertainty (error bars) is virtually useless since such limits are always needed in any application and are not implicit in the measurement process.

The measurement process should follow the procedure outlined in figure 7.3. The measurement laboratory uses existing methodology, appropriate calibrations and quality control techniques to attain statistical control of the measurement process. When acceptable precision is attained, the laboratory can evaluate its bias and set limits of uncertainty for the data. If either bias or precision are unacceptable, assignable causes should be sought and appropriate corrective actions should be taken.

Any laboratory can and should evaluate its own precision of measurement and its ability to do so is a measure of its competence. Evaluation of bias can be extremely difficult and facilitated by externally provided calibrations, reference materials, definitive measurements, and other approaches. Obviously, biases smaller than the precision of measurement will be difficult to identify.

A laboratory must be capable of evaluating its own precision and maintaining its measurement system in a state of statistical control. Otherwise, it must be considered as incapable of providing the services it offers. Furthermore, a laboratory should assign limits of uncertainty and maintain documented evidence for the basis of such assignment. In on-going measurement processes, control charts provide the basis for such assignment. In other cases, the redundant process of repetition is the only means to make such an assignment.

In addition to the above, NBS provides a limited amount of support for the measurement system in the form of education and training. Basic metrological information is contained in a number of papers. Statistical treatment of measurement data is discussed in a group of papers contained in NBS Special Publication 300 (17) and in NBS Handbook 91 (19). NBS presents a number of seminars in several areas of metrology. Information about them and the current schedule can be obtained from the NBS Office of Measurement Services (16). Specialized training for metrologists of State Weights and Measures laboratories is possible from the NBS Office of Weights and Measures.



TABLE 7.1

OPERATION OF A MEASUREMENT PROCESS WITH NBS SUPPORT

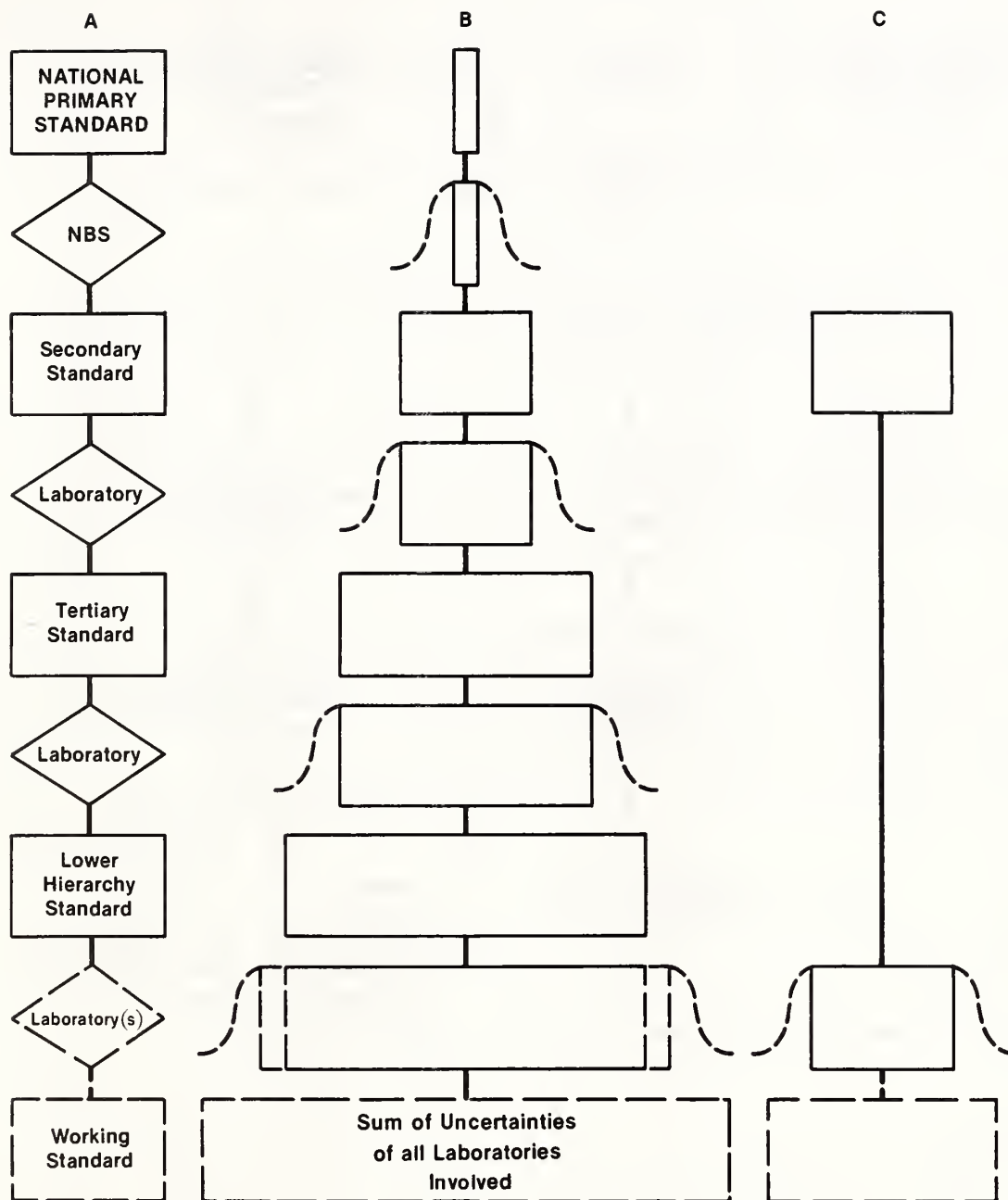
1. NBS provides
  - 1.1 Fundamental research to extend frontiers of methodology
  - 1.2 Basic research to develop new methodology
  - 1.3 Applied research to improve existing methodology
  - 1.4 Reference materials
  - 1.5 Calibration services
  - 1.6 Other approaches for evaluating bias, e.g., definitive methods
2. Measurement laboratory
  - 2.1 Utilizes existing methodology
  - 2.2 Applies appropriate quality control techniques
  - 2.3 Demonstrates statistical control of the measurement process
  - 2.4 Evaluates precision
    - 2.4.1 Precision acceptable
      - 2.4.1.1 Evaluates bias using reference materials, calibration services, other approaches
        - 2.4.1.1.1 Limits of bias acceptable
          - 2.4.1.1.1.1 Reports measured value with assigned limits of accuracy
        - 2.4.1.1.2 Limits of bias unacceptable
          - 2.4.1.1.2.1 Assignable cause identified to improve methodology or quality control. Material remeasured
          - 2.4.1.1.2.2 Assignable cause not identified
            - 2.4.1.1.2.2.1 Seek other methods of measurement, or measurement not possible and major research required
            - 2.4.1.1.2.2.2 Report biased measured value with precision, only, reported
    - 2.4.2 Precision unacceptable
      - 2.4.2.1 Assignable cause(s) identified to improve methodology and/or quality control; remeasure
      - 2.4.2.2 Assignable cause(s) not identified; measurement not possible; seek other methodology or major research required



Notes:

1. Measurement laboratories have sole responsibility for evaluating their precision and can do so with little or no NBS support  
  
NBS can provide education/training/workshops to assist laboratories to achieve quality control/statistical control  
  
NBS can develop GLP's, GMP's, SOP's in critical areas of national concern (including industrial productivity)
2. Measurement laboratories can utilize 1.4, 1.5, and 1.6, with little or no NBS assistance, to evaluate bias
3. NBS has responsibility to establish and maintain the National Measurement System which involves 1.1, 1.2, 1.4, 1.5, and 1.6
4. NBS engages in state-of-the-art R & D in 1.2 and 1.3 to develop 1.4, 1.5, and 1.6. Much of this is transferable to measurement laboratories.
5. NBS cannot attest for the precision or accuracy of measurement values reported by others than itself.
6. NBS can make judgments whether a measurement process is or is not potentially capable of reliable measurements, but the demonstration of such capability is the responsibility of the measurement laboratory and must be supported by an adequate quality assurance program.
7. The development and maintenance of an adequate quality assurance program is the sole responsibility of the measurement laboratory. This is a prerequisite for offering and providing measurement services. NBS can assist by conducting research in quality assurance techniques, development of reliable methodology and conducting seminars or workshops, all intended to make measurement assurance self-sustaining.
8. If a measurement assurance program requires extensive NBS direct involvement, it needs to be redesigned to eliminate such involvement and or to remove the reason why such involvement is necessary.





A. Calibration Chain  
 B. Propagation of Uncertainty  
 C. Alternate Calibration of Chain Uncertainty

FIGURE 7.2 PROPAGATION OF CALIBRATION UNCERTAINTY



## 7.2 Tolerance

Tolerances for weighing and measuring devices are established on the following basis: tolerance values are so fixed that the permissible errors are sufficiently small that there is no serious injury to either the buyer or seller of commodities, yet not so small as to make manufacturing or maintenance cost of equipment disproportionately high. With respect to the standards used to test weighing and measuring devices, the error in the standard should be less than 25 percent of the smallest tolerance to be applied to the device when the standard is used, otherwise, a correction factor must be applied to the standard so used.

Whenever a customer's standard is tested in the laboratory and returned to the user, the standard should be expected to remain within tolerance until the time it is scheduled to be recertified. Whenever a standard is out of tolerance or near the tolerance limit such that it is likely to go out of tolerance before it is expected to be retested, the standard should be adjusted as closely as practical to zero error. The rate with which the standard changes its value depends upon the material of which it is made, the frequency of use, the care it receives, and the environment to which it is subjected. Any standard that is damaged or is subject to an incident that may have changed its value significantly, resulting in a question of its validity, should be removed from use until it can be tested in the laboratory.

The tolerance applicable to the device for which the standard is to be used is not the only consideration for establishing the tolerance for the standard. An additional factor is that the tolerance on the standard should not be an excessively large fraction of the value of the standard. The standard should be a reasonably accurate representation of its nominal value. A determination of what is "reasonably accurate" depends upon the type of standard, its nominal value, its use, and the accuracy required in the measurement process in which the measuring device (under test) will be used.

### Tolerances for Commercial Equipment

Acceptance and Maintenance Tolerances - The official tolerances prescribed by a weights and measures jurisdiction for commercial equipment are the limits of inaccuracy officially permissible within that jurisdiction. It is recognized that an errorless value or performance of mechanical equipment is unattainable. Tolerances are established, therefore, to fix the range of inaccuracy within which equipment will be officially approved for commercial use. In the case of classes of equipment on which the magnitude of the errors of value or performance may be expected to change as a result of use, two sets of tolerances are established: acceptance tolerances and maintenance tolerances. Acceptance tolerances are applied to new or newly reconditioned or adjusted equipment, and are smaller than (usually one-half of) the maintenance tolerances. Maintenance tolerances thus provide an additional range of inaccuracy within which equipment will be approved on subsequent tests, permitting a limited amount of deterioration before the equipment will be officially rejected for inaccuracy and before reconditioning or adjustment will be required. In effect, there is assured a reasonable period of use for equipment after it is placed in service before reconditioning will be officially required. The foregoing comments do not apply, of course, when only a single set of tolerance values is established, as is the case with equipment such as glass milk bottles and



graduates, which maintain their original accuracy regardless of use, and single service measure-containers, which are used only once.

Tolerances and Adjustments - Tolerances are primarily accuracy criteria for use by the regulatory official. However, when equipment is being adjusted for accuracy, either initially or following repair or official rejection, the effect should be to adjust as closely as practicable to zero error. Equipment owners should not take advantage of tolerances by deliberately adjusting their equipment to have a value or to give performance at or close to the tolerance limit. Nor should the repairman or serviceman bring equipment merely within tolerance range when it is possible to adjust closer to zero error.

Tolerances for Standards - A general principle that has long been recognized by the National Bureau of Standards is that the error in a standard used by a weights and measures official should be known and corrected for when the standard is used; or if the standard is to be used without correction, its error should not be greater than  $1/3$  of the smallest tolerance to be applied when the standard is used. The reason for this is to keep at a minimum the proportion of the tolerance on the item tested that will be used up by the error of the standard. Expressed differently, the reason is to give the item being tested as nearly as practicable the full benefit of its own tolerance.

Field testing operations are complicated to some degree when corrections to standards are applied. Except for work of relatively high precision, it is recommended that the accuracy of standards used in testing commercial weighing and measuring equipment be so established and maintained that the use of corrections is not necessary. Also, whenever it can readily be done, it will be desirable to reduce the error on a standard below the  $1/3$  of the smaller tolerance previously mentioned.

The numerical values of the tolerances recommended by the National Bureau of Standards for the standards of length, mass, and capacity used by weights and measures officials may be obtained upon request from the Office of Weights and Measures of the National Bureau of Standards.

When Corrections Should Be Made - When testing a measuring device the weights and measures official has expressly only one official duty, and that is merely to determine whether equipment is or is not suitable for commercial use. If a device conforms to all of the official requirements, the official seals it to indicate approval. If it does not conform to all official requirements, he is required only to reject it and prohibit its use until the device is brought into proper conformance.

Some officials contend that it is justifiable for the official to make minor corrections and adjustments in order to correct faulty equipment if there is no service agency nearby or if the owner or operator depends on this single device and would be "out of business" during the repair of the device.

Adjustments should be made, with the permission of the owner or his representative, only when the official is thoroughly competent to make such an adjustment and when he is certain that the real cause of the inaccuracy will be corrected thereby and is not due to faulty installation or a defective part. He should never undertake major repairs, or even minor corrections if the services of commercial agencies are readily available.

Gauging - In the majority of cases, when the weights and measures official tests commercial equipment, he is verifying the accuracy of a value or the accuracy of the performance as previously established either by himself or by someone else. There are times, however, when the test of the official is the initial test on the basis of which the calibration of the device is first determined or its performance first established. The most common example of such gauging is in connection with vehicle tanks, the compartments of which are used as measures. Frequently the official makes the first determination on the capacities of the compartments of a vehicle tank, and his test results are used to determine the proper settings of the compartment indicators for the exact compartment capacities desired. Adjustments of the position of an indicator under these circumstances are clearly not the kind of adjustments discussed in the preceding paragraph.

Inspection versus Testing - A distinction may be made between the inspection and the testing of commercial equipment that should be useful in differentiating between the two principal groups of official requirements--specifications and performance requirements. Although frequently the term inspection is loosely used to include everything that the official has to do in connection with commercial equipment, it is useful to limit the scope of that term primarily to examinations made to determine compliance with design, maintenance, and use requirements. The term testing may then be limited to those operations carried out to determine the accuracy of value or performance of the equipment under examination by comparison with the actual physical standards of the official.

Accuracy of Standards - The accuracy of testing apparatus should invariably be verified prior to the official use of the apparatus. Standards should be reverified as often as circumstances require. By their nature, metal volumetric standards are more susceptible to damage in handling than are standards of some other types. Whenever damage to a standard is known or suspected to have occurred, and whenever repairs that might affect the accuracy of a standard have been made, the standard should be recalibrated. Routine recalibration of standards, particularly volumetric standards, even when a change of value is not anticipated, should be made with sufficient frequency to affirm their continued accuracy, so that the official may never be in an indefensible position with respect to the accuracy of his testing apparatus. If use is made of secondary standards, such as special fabric testing tapes, these should be verified much more frequently than such basic standards as steel tapes or volumetric provers to demonstrate their constancy of value or performance.

Accurate and dependable results cannot be obtained with faulty or inadequate standards. If either serviceman or official is poorly equipped, it cannot be expected that their results will check consistently. Disagreements between servicemen and officials can be avoided, and the servicing of commercial equipment can be indefensibly improved if servicemen and officials will give equal attention to the adequacy and maintenance of their testing apparatus.

### 7.3 Distinction Between Mass and Apparent Mass

The mass of an object can be simply defined as the quantity of matter that comprises the object. The mass of an object remains constant regardless of its location. Thus, the mass does not vary as the object is moved from one part of the country to another although the forces acting on the mass may change. In mass measurement, the term "true mass" is frequently used to mean the mass of an object. The adjective "true" is redundant and will not be used in the remainder of this paper.

The purpose of mass measurement is to determine the mass of an unknown object; i.e., the unknown object is calibrated by comparing its mass to that of a known mass standard. A mass calibration is performed in air as are virtually all mass measurements. Thus, when two objects are compared, each object is being subjected to a lifting force equal to the mass of air displaced by the object times the force of gravity in addition to the downward force on each object resulting from the earth's gravity. The mass of air displaced by an object depends on the density of the air and the volume of the object. Since all mass measurements are made in air and mass calibrations are performed by comparing an unknown standard to a known standard, the mass of a standard is frequently reported as the apparent mass of the standard. The apparent mass of an object is the mass of a (hypothetical) reference standard of a specified density that will produce a balance reading equal to that produced by the object if the measurements are made at 20 °C in air with a density of 1.2 mg/cm<sup>3</sup>. Whenever the term "apparent mass" is used, it is necessary to specify the density of the (normally hypothetical) reference standard against which the unknown standard is being compared. This statement of the density of the reference standard, called the reference density, is necessary because the apparent mass value depends in part upon the volume of the hypothetical reference standard. The reference density of 8.0 g/cm<sup>3</sup> is normally used to report the apparent mass of a standard or object. This is called the apparent mass versus 8.0 g/cm<sup>3</sup>. In the past, the apparent mass was reported against the density of brass at 20 °C. This density is 8.3909 g/cm<sup>3</sup>. This apparent mass value is referred to as the apparent mass versus brass.

The definition of apparent mass versus a reference density specifies the conditions under which the apparent mass of a standard or object is to be determined. To compute apparent mass, it is necessary to know:

1. the density of the hypothetical reference standard;
2. the mass of the hypothetical reference standard which is the mass of a reference standard that will give the same balance reading as the unknown standard under specified conditions;
3. the temperature (20 °C) at which the "comparison" of the masses is made;
4. the density of the air (1.2 mg/cm<sup>3</sup>) in which the "comparison" is made; and
5. the density of the standard being calibrated.

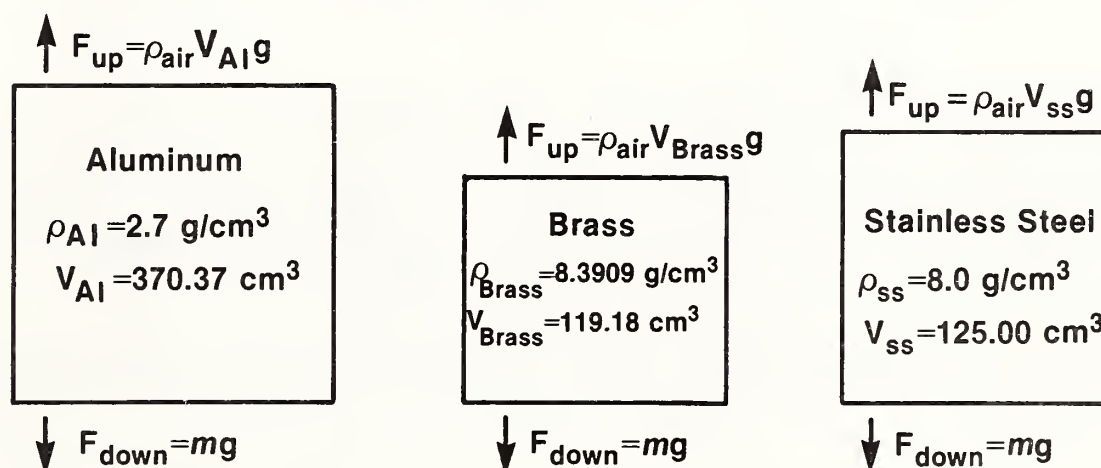
The density of the object being calibrated must be known since its volume is involved in the apparent mass determination. It is the difference in the volumes between the object being calibrated and the hypothetical reference standard that determines the apparent mass of an object versus a specified reference density.



The apparent mass versus  $8.0 \text{ g/cm}^3$  of an object can be defined as the mass the object would appear to have if it was compared against a standard which has density of  $8.0 \text{ g/cm}^3$  and a mass giving the same balance reading as the object when the comparison is made at  $20^\circ\text{C}$  in air having a density of  $1.2 \text{ mg/cm}^3$ .

The definition for apparent mass versus brass is exactly the same except that the density of brass at  $20^\circ\text{C}$  ( $8.3909 \text{ g/cm}^3$ ) is substituted for  $8.0 \text{ g/cm}^3$ . An example will be given to illustrate the apparent mass concept.

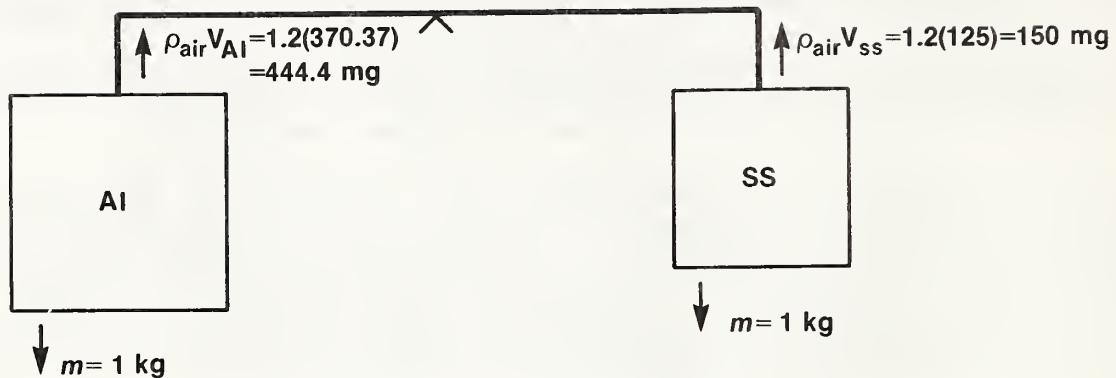
Suppose that we have three weights made of aluminum (Al), brass, and stainless steel (SS). Assume that the densities of these weights are  $2.7 \text{ g/cm}^3$ ,  $8.3909 \text{ g/cm}^3$ , and  $8.0 \text{ g/cm}^3$ ; respectively, and that all three weights have a mass of exactly  $1 \text{ kg}$ . The weights then have volumes of  $370.37 \text{ cm}^3$ ,  $119.18 \text{ cm}^3$  and  $125.00 \text{ cm}^3$ , respectively. When the weights are in air, there will be a lifting force due to the effect of the displaced air and a downward force due to the mass ( $m$ ) of the weight.



It can be seen that the larger the volume, the greater the upward force. Since gravity appears in all terms when the weights are intercompared, the gravity factor ( $g$ ) cancels.

Suppose now that a perfect equal-arm balance exists and is used to compare the weights in both a vacuum and in air. Suppose that the aluminum weight is placed on one pan of the balance and the stainless steel weight is placed on the other pan and the balance and weights are placed in a vacuum. Because there is no air present, there is no lifting force so the weights will appear to have equal masses since only the downward force is acting on the weights. If the aluminum weight was compared in a vacuum against the brass weight, the two weights would also appear to have equal masses. The same would be true if the brass weight was compared to the stainless steel weight. Thus, the mass of an object can be visualized as the mass an object would appear to have when it is compared in a vacuum against a known standard.

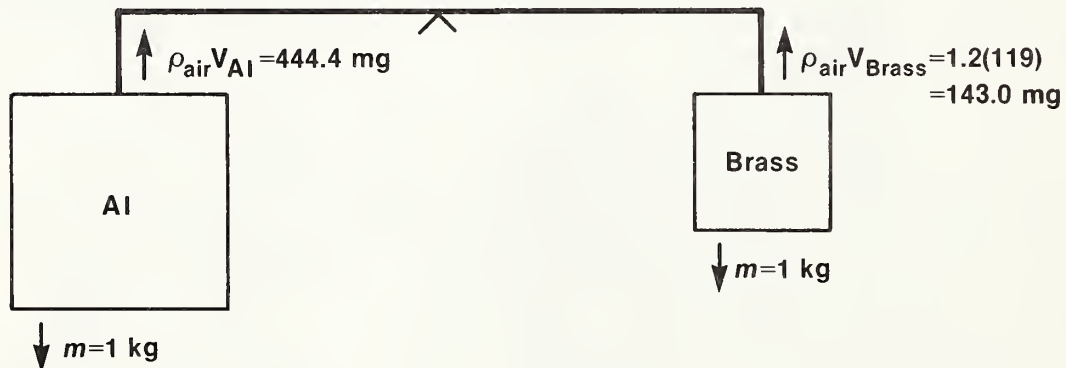
Now suppose that the aluminum and stainless steel weights are compared on the balance in air that has a density of  $1.2 \text{ mg/cm}^3$ . The air will exert a lifting force on each weight as described earlier. This can be illustrated in mass units (since gravity cancels out) as



The aluminum weight experiences a much larger lifting effect than the stainless steel weight, namely, 444.4 mg versus 150 mg. Thus, the aluminum weight appears to be 294.4 mg lighter than the stainless steel weight. This is simply the air density times the difference in weight volumes. Hence, the apparent mass of the aluminum weight versus  $8.0 \text{ g/cm}^3$  is approximately

$$1 \text{ kg} - 294.4 \text{ mg} = 999.7056 \text{ g}$$

If the aluminum weight was compared in air against the brass weight, the situation is illustrated as

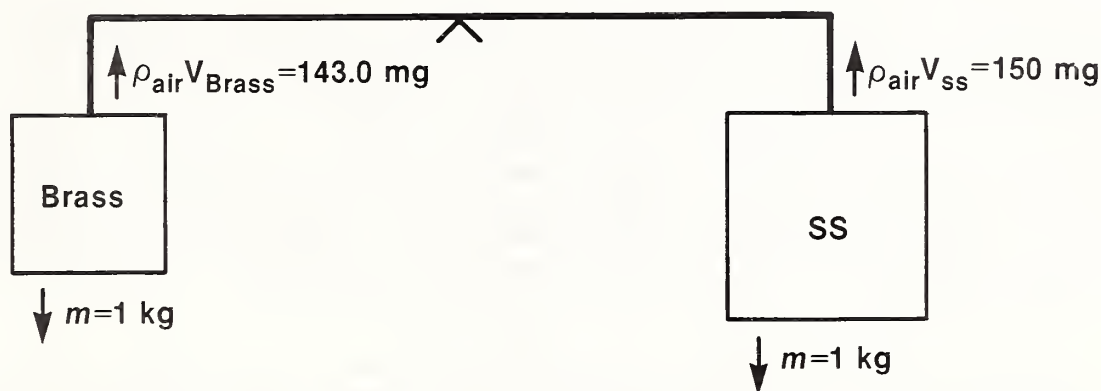


This means the aluminum weight appears to be 301.4 mg lighter than the brass weight. Hence the apparent mass of the aluminum weight versus brass is approximately

$$1 \text{ kg} - 301.4 \text{ mg} = 999.6986 \text{ g}$$



To carry the example further, suppose the brass weight and the stainless steel weight were compared in air with a density of  $1.2 \text{ mg/cm}^3$ . The effect can be illustrated as



Using the stainless steel weight as the reference standard, the brass weight appears to be 7 mg heavier than the stainless steel weight. This is due to the difference in the lifting effect of air on the weights. The lifting effect is less on the brass weight because its volume is less than that of the stainless steel weight. This condition can also be reported using the brass weight as the reference standard. In this case, the stainless steel weight appears to be 7 mg lighter than the brass weight. Hence, we can say that the apparent mass of the brass weight versus  $8.0 \text{ g/cm}^3$  is  $1 \text{ kg} + 7 \text{ mg}$  or  $1000.007 \text{ g}$ . Similarly, the apparent mass of the stainless steel weight versus brass is  $1 \text{ kg} - 7 \text{ mg}$  or  $999.993 \text{ g}$ . Thus, the apparent mass of a weight depends upon the density chosen for the reference density. This is particularly clear for the results for the aluminum weight. Thus, the apparent mass versus a reference density is the mass an object would appear to have if it was compared against a standard of a specified density and having a mass that would give the same reading as the object when the comparison is made at  $20^\circ \text{C}$  in air with a density of  $1.2 \text{ mg/cm}^3$ .

The equations recommended for use to compute the apparent mass of an object are given in SOP No. 2.

## 7.4 Control Charts

Control charts provide a graphical means to decide whether a measurement system has attained and is maintaining a state of statistical control. They can indicate drifts warn of the initiation of potential problems and reveal the need for corrective actions.

Control charts were first proposed in 1934 by Walter Shewhart for use in statistical process control. Since that time, various modifications of his original format have been proposed and used. Cameron and co-workers at NBS [7] were among the first to use control charts for monitoring measurement processes, using essentially the Shewhart format. A general discussion of control charts and extensive tables useful for calculating control limits will be found in ASTM Manual for Presentation of Data and Control Chart Analysis [3]. Croarkin discusses control charts as they are used in measurement assurance programs [11]. The following discussion reviews the kinds of control charts considered to be most useful for laboratory use.

Several kinds of control charts will be found to be useful. The simplest is based on the repetitive measurement of a stable test object and either the results of single measurements ( $\bar{X}$  chart) or the means of several ( $\bar{\bar{X}}$  chart) are plotted with respect to sequence or time of measurement. The results should be randomly distributed about the mean ( $\bar{X}$ ) in the case of an  $\bar{X}$  chart and about the mean of means ( $\bar{\bar{X}}$ ) in the case of an  $\bar{\bar{X}}$  chart when the measurement system is in a state of statistical control. Furthermore, the results should lie within defined limits, based on statistical considerations.

There is nothing really wrong in maintaining a simple  $\bar{X}$  chart. However, an  $\bar{\bar{X}}$  chart is preferable to an  $\bar{X}$  chart because average values will indicate a change in performance more conclusively than individual values. This advantage must be evaluated against the increased effort required to maintain the former. An  $\bar{\bar{X}}$  chart based on the average of two measurements is a good compromise when possible.

In addition to the above, there are precision control charts in which either the standard deviation, estimated at various times, or the range,  $R$ , of a set of measurements is plotted and interpreted similarly. Because of the economy of effort, an  $R$  chart is preferable to an  $s$  chart. When a property-value control chart ( $\bar{X}$  chart or  $\bar{\bar{X}}$  chart) and a precision chart are maintained in parallel, diagnosis of out-of control situations as due to imprecision or bias and the identification of assignable causes for such are facilitated.

### X Control Chart

Single measurements are made of a stable test object, at least once on each test day or at least monthly (if a measurement system is to be maintained in statistical control over a period of time). The results are plotted sequentially and the process is considered to be in control when they are randomly distributed within limits as defined below.

## Initial Control Limits, X Chart

### Central Line, $\bar{X}$

Measure the test object on at least 12 occasions (recommended) but no more frequently than daily, i.e. never twice on the same day. The initial central line is the mean of the  $n$  measurements,  $X_i$  for  $i=1, \dots, n$ .

$$\text{Central line, } \bar{X} = \frac{\sum X_i}{n}$$

Calculate  $s_x$  the estimate of the standard deviation of  $X$  in the usual manner. Note that this is an estimate of the long-term standard deviation. Calculate the upper and lower control and warning limits as:

$$\begin{aligned} \text{UCL} &= \bar{X} + 3s_x \\ \text{UWL} &= \bar{X} + 2s_x \\ \text{LWL} &= \bar{X} - 2s_x \\ \text{LCL} &= \bar{X} - 3s_x \end{aligned}$$

When so set, approximately 95% of the plotted points should fall between the warning limits (LWL and UWL) and rarely should any fall outside of the control limits (LCL and UCL) if the system is in a state of statistical control. The control limits are conservative.

### $\bar{X}$ Control Chart

A stable test object is measured in replicate, periodically. It is recommended that these should be duplicate measurements made at least once on each test-day or at least monthly, whichever is the more frequent. The means of the measurements,  $\bar{X}$ , are plotted sequentially. Statistical control is judged when the plotted points are randomly distributed within the control limits, determined as outlined below.

## Initial Control Limits, $\bar{X}$ Chart

### Central Line $\bar{\bar{X}}$

Measure the test object, in duplicate on at least 12 occasions (recommended) and no more frequently than daily, i.e. never twice on the same day. The initial central line is the mean of the means of  $n$  duplicate measurements,  $\bar{X}_i$  for  $i=1, \dots, n$ .

$$\text{central line, } \bar{\bar{X}} = \frac{\sum \bar{X}_i}{n}$$

Calculate  $s_{\bar{x}}$ , the estimate of the standard deviation of  $\bar{X}$  in the usual manner.

Note that this is a long-term standard deviation of the mean of  $n$  measurements and will ordinarily be larger than the short-term (within day) standard deviation which may be calculated from the value of  $\bar{R}$  (see later).

However, if the long-term exceeds the short-term standard deviation by more than a factor of 2, the quality control should be improved to decrease the former to more acceptable values.

#### Control Limits

$$UCL = \bar{\bar{X}} + 3s_{\bar{X}}$$

$$UWL = \bar{\bar{X}} + 2s_{\bar{X}}$$

$$LWL = \bar{\bar{X}} - 2s_{\bar{X}}$$

$$LCL = \bar{\bar{X}} - 3s_{\bar{X}}$$

The limits are set so that approximately 95% of the points should fall within the warning limits (LWL and UWL) and rarely should any fall outside of the control limits (LCL and UCL) if the system is in a state of statistical control. The limits are conservative.

#### R Control Chart

The absolute differences (R) of duplicate measurements of the test object, and also of similar test specimens may be plotted sequentially to evaluate the precision of the measurement process. This constitutes an R (range) control chart. Note that the range is related to the short-term standard deviation, i.e. the repeatability of measurements over a relatively short period of time.

#### Initial Control Limits, R Chart

##### Central Line $\bar{R}$

The observed ranges  $R_i$  (absolute values) for k sets (at least 12 is recommended) of duplicate measurements are averaged to obtain a value for  $\bar{R}$ .

$$\bar{R} = \frac{\sum R_i}{k}$$

The control limits for duplicate measurements are as follows:

$$UCL = 3.267 \bar{R}$$

$$UWL = 2.512 \bar{R}$$

$$LWL = 0$$

$$LCL = 0$$

The control limits have the same significance as in the case of an  $\bar{X}$  or  $\bar{X}$  chart.

For triplicate measurements, the control limits are:

$$UCL = 2.512 \bar{R}$$

$$UWL = 2.050 \bar{R}$$

$$LWL = 0$$

$$LCL = 0$$



## Updating Control Charts

After additional control data are accumulated (at least as much as originally used) the control limits may be updated. A  $t$  test is made to see whether the second set of data for  $\bar{X}$  or  $\bar{X}$  is significantly different from the first set (see Chapter 8.11). If not, all data may be combined to obtain a new and more robust estimate of  $\bar{X}$  or  $\bar{X}$ . If the second set is significantly different from the first, only the latter should be used in revising the control chart.

The value for the standard deviation  $s$  for the second set of determinations should likewise be compared with the first estimate using the  $F$  test (see Chapter 8.9) to decide whether to pool it with the first (see Chapter 7.4) or to use it separately in setting new control limits. A smaller value for  $s$ , may result from improvement of the precision as the result of a learning experience, for example. A larger value for  $s$  could be due to an original poor estimate of the standard deviation of the measurement process, or to a decrease of precision resulting from an assignable cause(s). In either case, the reason should be ascertained.

If the values of  $R$  show no systematic trends, and if  $\bar{R}$  has not changed significantly, all of the values of  $R$  may be combined to obtain an updated estimate of  $\bar{R}$ , from which updated control limits can be computed. Judgment of the significance of apparent changes in  $\bar{R}$  can be made by computing the corresponding values of  $s$  (see Table 9.1) and conducting an  $F$  test (see Chapter 8.9).

## Interpretation of Control Chart Data

Plotted points should be randomly distributed within the warning limits when the system is in a state of statistical control. If a plotted point lies outside of the warning limits, a second set of measurements should be made. If this point lies outside of the warning limits, corrective action is required and demonstrated attainment of control is necessary before measurements may be reported with confidence. Barring blunders, one point outside of the control limits is reason for corrective action. The nature of the corrective action to be taken, in either case, will depend on the kind of measurement made. If the  $X$  or  $\bar{X}$  point is outside the limits but the  $R$  point is not, a source of bias should be sought and eliminated. If the  $R$  point is outside of limits,  $X$  or  $\bar{X}$  will probably but not necessarily be outside, as well (note that compensating fluctuations could cancel one another). Sources of extraordinary random error should be sought and eliminated, before any possible bias can be detected.

Control charts may be used to evaluate the uncertainty of measurement in some cases. When an appropriate control chart is maintained, an  $X$  or  $\bar{X}$  chart may be used to evaluate bias and to document the standard deviation of the measurement process. Then the values for  $s$  on which the control limits are based may be used in calculations of confidence limits for measurement values (see Chapter 8.6).





## 8. STATISTICAL TECHNIQUES

The purpose of statistics in metrology is to summarize experimental data, to provide the basis for assessing its quality, and to provide a basis for making probabilistic decisions in its use. The essential basic statistical information for describing a simple data set is:

The mean of the set	$\bar{x}$
The standard deviation of the set	$s$
The number of individuals in the set	$n$

If the set is a random sample of the population from which it was derived, if the measurement process is in statistical control, and if all of the observations are independent, then  $s$  is an estimate of the population standard deviation,  $\sigma$ , and  $\bar{x}$  is an unbiased estimate of the mean,  $\mu$ .

The population consists of all of the possible measurements that could have been made, under the test conditions, for a stable test sample. In this regard, the metrologist must be aware that any changes in the measurement system (known or unknown) possibly could result in significant changes in its operational characteristics and hence the values of the mean and standard deviation. Whenever there is doubt, statistical tests should be made to determine the significance of any apparent differences before statistics may be combined.

The following discussion reviews some statistical techniques useful when interpreting measurement data. In presenting this information, it is assumed that the reader is already familiar with basic statistical concepts. For a detailed discussion of the following techniques and others not presented here, it is recommended that the reader consult NBS Handbook 91 - Experimental Statistics, by Mary G. Natrella [19]. Also, that handbook contains complete statistical tables from which the tables contained in Chapter 9 of this handbook were taken.

### 8.1 Estimation of Standard Deviation from a Series of Measurements on a Given Object

Given  $n$  measurements

$$x_1, x_2, x_3 \dots x_n$$

Mean ,  $\bar{x}$

$$\bar{x} = \frac{x_1 + x_2 + x_3 + \dots + x_n}{n}$$

Standard deviation estimate,  $s$

$$s = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}}$$

The estimate,  $s$ , is based on  $n-1$  degrees of freedom

## 8.2 Estimation of Standard Deviation from the Differences of k Sets of Duplicate Measurements

Given k differences of duplicate measurements

$$d_1, d_2, d_3 \dots d_k,$$

a useful formula for estimating the standard deviation is:

$$s_d = \frac{\sum d_i^2}{2k}$$

$s_d$  is based on k degrees of freedom

Note  $d_1 = x_1 - x_1'$ , for example.

The values  $d_1, d_2$  etc., may be differences of duplicate measurements of the same sample (or object) at various times, or they may be the differences of duplicate measurements of several similar samples (or objects).

## 8.3 Estimation of Standard Deviation from the Average Range of Several Sets of Measurements

The range, R, is defined as the difference between the largest and smallest values in a set of measurements.

Given

$$R_1, R_2, R_3 \dots R_k$$

Mean,  $\bar{R}$

$$\bar{R} = \frac{R_1 + R_2 + R_3 + \dots + R_k}{k}$$

Standard deviation can be estimated by the formula

$$s_R = \bar{R}/d_2^*$$

The value of  $d_2^*$  will depend on the number of sets of measurements used to calculate  $\bar{R}$ , and on the number of measurements in each set, i.e., 2 for duplicates, 3 for triplicates, etc. Consult a table such as Table 9.1 for the appropriate value of  $d_2^*$  to use. The effective number of degrees of freedom for  $s_R$  will be found in the table.

## 8.4 Pooling Estimates of Standard Deviations

Estimates of the standard deviation obtained at several times may be combined (pooled) to obtain a better estimate based upon more degrees of freedom. The following equation may be used for this purpose:

$$s_p = \frac{(n_1 - 1)s_1^2 + (n_2 - 1)s_2^2 + \dots + (n_k - 1)s_k^2}{(n_1 - 1) + (n_2 - 1) + \dots + (n_k - 1)}$$

$s_p$  will be based on

$(n_1 - 1) + (n_2 - 1) + \dots + (n_k - 1)$  degrees of freedom.

### 8.5 "Within" and "Between" Standard Deviation

The estimation of the within-series,  $s_w$ , and between-series,  $s_b$  standard deviation, (also referred to as short-term and long-term standard deviations in the applications described here) is an important way to characterize a measurement process. The former provides guidance as to how many repetitions of a measurement are required to obtain a result on a single occasion with a given precision while the latter is a better estimate of the precision of replication of a result on various occasions, which is a more realistic evaluation of measurement variability.

To estimate these standard deviations, sets of measurements may be made on several occasions. To simplify the calculations, each set should consist of the same number of measurements. For most measurements, it is recommended that duplicate measurements be made on at least 12 separate occasions when estimating  $s_w$  and  $s_b$ .

Given  $k$  sets of duplicate measurements made on  $k$  occasions

#### Tabulate

Occasion	Measured Values		Range	Mean
1	$x_1$	$x_1'$	$R_1$	$(x_1 + x_1')/2 = \bar{x}_1$
2	$x_2$	$x_2'$	$R_2$	$(x_2 + x_2')/2 = \bar{x}_2$
3	$x_3$	$x_3'$	$R_3$	$(x_3 + x_3')/2 = \bar{x}_3$
.	.	.	.	.
.	.	.	.	.
.	.	.	.	.
$k$	$x_k$	$x_k'$	$R_k$	$(x_k + x_k')/2 = \bar{x}_k$

1. Calculate  $\bar{R} = \frac{R_1 + R_2 + \dots + R_k}{k}$

2. Calculate  $s_w = s_R = \bar{R}/d_2^*$

Note: One may use the procedure of 8.2 to calculate  $s_w$  if preferred.

3. Calculate  $s_{\bar{x}}$  as follows:

$$\bar{x} = \frac{\bar{x}_1 + \bar{x}_2 + \dots + \bar{x}_k}{k}$$

$$s_{\bar{x}} = \sqrt{\frac{\sum (\bar{x}_i - \bar{x})^2}{k-1}}$$

4. Calculate  $s_b$  (for the case of duplicate measurements)

$$s_b = \sqrt{s_{\bar{x}}^2 - \frac{s_w^2}{2}}$$

Note that  $s_b$  is an estimate of the long term component of the standard deviation of a single measurement. The long term standard deviation of the mean of  $n$  measurements taken at a single occasion is estimated by:

$$\sqrt{s_b^2 + \frac{s_w^2}{n}}$$

### 8.6 Confidence Interval for the Mean

The estimation of the confidence interval for the mean of  $n$  measurements is one of the most frequent statistical calculations. The formula used will depend on whether the population standard deviation,  $\sigma$ , is known or whether it is estimated on the basis of measurements of a sample(s) of the population.

#### Using Population Standard Deviation, $\sigma$

Strictly speaking,  $\sigma$ , is never known for a measurement process. However, the formula for use in such a case is:

$$\bar{x} \pm \frac{z\sigma}{\sqrt{n}}$$

where  $\bar{x}$  = sample mean  
 $\sigma$  = known standard deviation  
 $n$  = number of measurements of sample  
 $z$  = standard normal variate, depending on the confidence level desired

For 95% confidence  $z = 1.960$ ; for 99.7% confidence  $z = 3.0$   
 For other confidence levels, see Table 9.2



### Using Estimate of Standard Deviation, $s$

In the usual situation,  $s$  is known, based on  $\nu$  degrees of freedom and the formula for use is:

$$\bar{x} \pm \frac{ts}{\sqrt{n}}$$

where  $\bar{x}$  = sample mean  
 $s$  = estimate of standard deviation  
 $n$  = number of measurements on which mean is based  
 $t$  = Student's  $t$  value, based on the confidence level desired and the degrees of freedom associated with  $s$  (see Table 9.3).

Note that  $t \rightarrow z$  as  $n \rightarrow \infty$ . For many practical purposes, the standard deviation may be considered as known when estimated by at least 30 degrees of freedom.

### 8.7 Confidence Interval for $\sigma$

The standard deviation,  $\sigma$ , is ordinarily not known but, instead, an estimated value based on a limited number of measurements, using procedures such as have been described above. Such estimates may be pooled, as appropriate, to obtain better estimates. In any case, the uncertainty of the estimated value of the standard deviation may be of interest and can be expressed in the form of a confidence interval, computed as indicated below.

The interval is asymmetrical because the standard deviation is ordinarily underestimated when small numbers of measurements are involved. The reason for this is that large deviations occur infrequently in a limited measurement process. Indeed, it is the general experience of metrologists that a few measurements appear to be more precise than they really are.

The basic information required to compute the interval is an estimate of the standard deviation,  $s$ , and the number of degrees of freedom on which the estimate is based. The relationships to use are:

Lower limit	$B_L s$
Upper limit	$B_U s$
Interval	$B_L s$ to $B_U s$

The values for  $B_L$  and  $B_U$  depend upon the confidence level and degrees of freedom associated with  $s$ . Values for use in calculating the confidence level are given in Table 9.7. A more extensive table (Table A-20), will be found in NBS Handbook 91 [19].

## 8.8 Statistical Tolerance Intervals

Statistical tolerance intervals define the bounds within which a percentage of the population is expected to lie with a given level of confidence. For example, one may wish to define the limits within which 95% of measurements would be expected to lie with a 95% confidence of being correct. The interval is symmetrical and is computed using the expression

$$\bar{x} \pm ks$$

where  $k$  depends on three things

$p$  = the proportion or percentage of the individual measurements to be included

$\gamma$  = the confidence coefficient to be associated with the interval

$n$  = the number of measurements on which the estimate,  $s$ , is based

Table 9.6 may be used to obtain values for  $k$  for frequently desired values of  $\gamma$  and  $p$ . A more extensive table is Table A-6 found in NBS Handbook 91 [19].

## 8.9 Comparing Estimates of a Standard Deviation (F Test)

This test may be used to decide whether there is sufficient reason to believe that two estimates of a standard deviation are significantly different. It consists in calculating the ratio of the variances (square of the standard deviation) and comparing it with tabulated values. Unless the computed ratio is larger than the tabulated value, there is no reason to believe that the respective standard deviations are significantly different.

The  $F$  ratio is calculated using the equation

$$F = \frac{s_L^2}{s_S^2}$$

where  $s_L$  is the numerically larger value and  $s_S$  is the smaller value of the two estimates under consideration.

The critical value of  $F$  will depend on the significance level chosen for the decision (test) and the number of degrees of freedom associated with  $s_L$  and  $s_S$ , respectively.

Table 9.4 contains critical values for  $F$  at the 95% level of confidence. The tabulated values of  $F$  are those expected not to be exceeded with 95% confidence on the basis of chance, alone. As an example, if both the numerator and the denominator values for  $s$  were each based on 9 degrees of freedom, an  $F$  value of no larger than 4.03 is expected with 95% confidence, due to the uncertainties of the  $s$  values, themselves. Table A-5 of NBS Handbook 91 [19] contains values for  $F$  for other confidence levels.

The F test is useful in comparisons of the precision of methods, equipment, laboratories, or metrologists, for example. An inspection of Table 9.4 will show that when either of the values of  $s$  is based on a small number of degrees of freedom, the F value is large. Consequently, the significance of decisions on small changes in precision can be supported statistically only by a relatively large number of measurements. If such changes are suspected but the data requirement is difficult to meet, the decision may need to be made on the basis of information about the measurement process itself.

The F test is also useful when deciding whether estimates of the standard deviation made at various times are significantly different. Such questions need to be answered when deciding on whether to revise control limits of a control chart, for example.

#### 8.10 Comparing a Set of Measurements with a Given Value

The question may arise as to whether a measured value agrees or is in significant disagreement with a stated value for the object measured. The decision can be based on whether or not the confidence interval for the measured value encompasses the stated value. The confidence interval is calculated using the expression

$$\bar{x} \pm \frac{ts}{\sqrt{n}}$$

as already described in Section 8.6. In using this expression,  $n$  represents the number of measurements used to calculate the mean,  $\bar{x}$ , and  $t$  depends on the degrees of freedom,  $\nu$ , associated with  $s$  and the confidence level needed when making the decision. Note that one may use historical data for estimating  $s$ , such as a control chart for example, in which case  $\nu$  will represent the degrees of freedom associated with establishment of the control limits and may be considerably larger than  $n-1$ .

#### 8.11 Comparing Two Sets with Regard to Their Means

This discussion is concerned with a decision as to whether the means of two measured values,  $\bar{x}_A$  and  $\bar{x}_B$ , are in agreement. The data sets used for this purpose may consist of the following:

$\bar{x}_A$	$\bar{x}_B$
$s_A$	$s_B$
$n_A$	$n_B$

The first question to be resolved is whether  $s_A$  and  $s_B$  can be considered to be different estimates of the same standard deviation or whether they do, indeed, differ. An F test may be used for this purpose. However, it will be recalled that this is not sensitive to small real differences, so the decision may need to be based on physical considerations such as the known stability of the measurement process, for example.

### Case I

Confirming (or assuming) that  $s_A$  and  $s_B$  are not significantly different, they are pooled, as already described (but repeated here for convenience) and used to calculate a confidence interval for the difference of the means. If this is larger than the observed difference, there is no reason to believe that the means differ. The steps to follow when making the calculation described above are:

Step 1. Choose  $\alpha$  the level of significance for the test

Step 2. Calculate the pooled estimate of the standard deviation,  $s_p$

$$s_p = \sqrt{\frac{(n_A - 1)s_A^2 + (n_B - 1)s_B^2}{(n_A - 1) + (n_B - 1)}}$$

$s_p$  will be estimated with  $n_A + n_B - 2$  degrees of freedom

Step 3. Calculate the respective variances of the means

$$V_A = \frac{s_p^2}{n_A} \quad V_B = \frac{s_p^2}{n_B}$$

Step 4. Calculate the uncertainty of  $|X_A - X_B| = \Delta$

$$U_\Delta = t \sqrt{(V_A + V_B)}$$

using a value for  $t$  based on  $\alpha/2$  and  $\nu = n_A + n_B - 2$

Step 5. Compare  $U_\Delta$  with  $\Delta$

If  $U_\Delta \geq \Delta$ , there is no reason to believe that  $\Delta$  is significant at the level of confidence chosen.



## Case II

Confirming (or assuming) that  $s_A$  and  $s_B$  are significantly different, their individual values are used to calculate  $U_\Delta$  as outlined below.

Step 1. Choose  $\alpha$ , the level of significance for the test

Step 2. Calculate the respective variances of the means

$$v_A = \frac{s_A^2}{n_A} \quad v_B = \frac{s_B^2}{n_B}$$

Step 3. Calculate the uncertainty,  $U_\Delta$ , of  $|X_A - X_B| = \Delta$

$$U_\Delta = t^* \sqrt{(v_A + v_B)}$$

using  $t^*$  based on  $\alpha/2$  and  $f$ , the effective number of degrees of freedom calculated as described in Step 4

Step 4. Calculate  $f$ , the effective number of degrees of freedom as follows:

$$f = \frac{(v_A + v_B)^2}{\frac{v_A^2}{n_A + 1} + \frac{v_B^2}{n_B + 1}} - 2$$

Step 5. Compare  $U_\Delta$  with  $\Delta$

If  $U_\Delta \geq \Delta$ , there is no reason to believe that  $\Delta$  is significant at the level of confidence chosen.

## 8.12 Use of Random Numbers

Conducting operations in random sequences can avert problems of bias due to the order in which they are done. For example, in the measurement of a series of items it might be difficult to determine whether systematic trends in the measured values were due to differences in the items or to measurement system drift, unless the items were measured in random order.

Tables of random numbers are a convenient means for randomizing measurement operations. The operations, test objects, and other matters requiring randomization may be assigned serial numbers, and the order of selection is determined by use of a table, as described below. When the number of operations or test items is less than 100, a table such as Table 9.11, reproduced from NBS Handbook 91 [19], may be used conveniently. One may start from any randomly selected position in the table and proceed from it in any pre-determined arbitrary manner. If the first number encountered is not that of one of the items, one ignores it and proceeds until a valid one is encountered. This becomes the first item in the sequence. Continuing, the items are selected as their serial number is encountered. If a number already selected is encountered, it is passed over until an unselected number is reached. The procedure is continued until all items are randomly selected.



As an example, select 10 specimens (numbered 01 to 10) in random order. Start from a randomly selected place, say column 2, row 5 of Table 9.11. Proceed from this point along the table as one would read a book. The starting number is 14 which is not usable. The first useful number encountered is 08, the next 03, and so on. Using the above described procedure, the following random order was found:

Specimen No.

08, 03, 09, 05, 06, 02, 07, 10, 04, 01

Order

1, 2, 3, 4, 5, 6, 7, 8, 9, 10