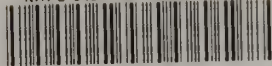


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U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards
Center for Absolute Physical Quantities
Washington, DC 20234

August 1983

Sponsors Report

Prepared for:

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U.S. DEPARTMENT OF COMMERCE, Malcolm Baldrige, *Secretary*
NATIONAL BUREAU OF STANDARDS, Ernest Ambler, *Director*



ADVANCED MASS CALIBRATION IN STATE LABORATORIES*

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Washington, D. C. 20234

ABSTRACT

This report describes the initial phase of a development program to improve maintenance of mass reference standards in State Weights and Measures Laboratories. Four one-kilogram objects of special design were calibrated by five State Laboratories, a highly qualified "check" laboratory, and NBS. The results indicate that certain improvements should be made in critical areas of the recalibration process. Errors associated with air buoyancy correction, for example, are significant; for this reason, it is necessary to improve the methods and instrumentation used for the measurement of air pressure, air temperature, and relative humidity in State Laboratories. Proposed future work is focused upon the air buoyancy correction, the simplification of calculation procedures, and the provision of NBS Reference Standards to improve the values assigned to the State Reference Standards.

The work reported was based upon the collaboration of the NBS Length and Mass Measurements and Standards Division, the NBS Office of Weights and Measures, Sandia Laboratories, and the five participating State Laboratories.

*This report includes privileged information and it is intended for internal NBS use only.

PURPOSE

Recent measurement control programs coordinated by the Office of Weights and Measures have disclosed some apparent anomalies in mass measurements in State programs that may be the result of inconsistencies in the mass standards that are in the possession of the State laboratories. The measurements reported here were designed to detect problems that would be encountered if the State laboratories were given the opportunity to perform measurements necessary to recalibrate State reference standards for mass and to determine the techniques that should be developed to perform this task most effectively.

METHOD

A. General

A weighing scheme has been devised using four one-kilogram weights, some with special features, to examine the following aspects of the participating State laboratories:

1. The accuracy of the State Kilogram Mass Standards
2. Laboratory techniques and environmental influences on assigned values
3. Adequacy of the required instrumentation
4. Data reduction difficulties and degree of automation

Cleaning of test weights by State Laboratories was specifically excluded from the measurement procedures.

In the measurement sequence, Division 523 cleaned the weights by vapor degreasing and calibrated the circulating weights before and after calibration by each State. This procedure enables early detection of a mass change in any weight due to accident or mistreatment.

Fives states participated in the measurements:

1. California
2. Colorado
3. Nevada
4. New York
5. Utah.

Sandia laboratory also participated at the request of NBS in order to provide Division 523 with additional measurement confirmation in areas for which the associated instrumentation is critical to success. Unlike most State laboratories, the Sandia laboratory maintains a high level of competence in pressure and temperature measurements that are important in mass calibrations involving significant buoyancy corrections.[1]*

B. The measurement format

Each participating laboratory was asked to assign mass values to the four one-kilogram test weights by comparison with its primary kilogram standards using a specified format. The four weights, designated A1, S2, R2, and H2 were paired as follows:

Pair 1	A1	S2
Pair 2	R2	H2

Each pair was to be calibrated by performing all possible comparisons with the two State kilogram standards. This procedure yields a total of six intercomparisons per pair, shown in table I, which were to be accomplished by the method known as double substitution weighing. Air density parameters were observed at the beginning and end of the sequence involving pair 1 and during the third and sixth observations for pair 2. The sequence of observations was modified for pair 2 to insure that air density parameters were noted during the most sensitive comparisons

(see table I). State metrologists were not given special instructions concerning the measurement of the air density parameters: temperature, barometric pressure, or relative humidity.

THE ARTIFACTS

The NBS mass standards used by Division 523 (B1, D2) for the NBS measurements were manufactured by the Troenmer Co. in the late 1960's. These weights are made of highly polished stainless steel with height nearly equal to diameter. Each weight is constructed as one-piece with an integral lifting knob machined into the object. Each State one-kilogram standard is also made from one piece of stainless steel with height nearly equal to diameter. However, lifting is provided by a pinched waist, i.e., "hour glass shape". The State standards were manufactured by several firms from an alloy of nominal density 8.0 g/cm^3 furnished by NBS.

The package sent to each State contained four one-kilogram weights. The test weight designated A1 is identical to the NBS standards described above (B1, D2). Weights S2, R2, and H2 were specially manufactured by Division 523 from stainless steel and were designed with highly unusual features to detect various weighing errors. S2 is a weight made from three concentric tubes supported by a tee-shaped base for simultaneous lifting of the individual pieces. This weight has an extremely large surface area. Weight R2 is a thick-walled tubular weight with a surface area which approximates that of H2. H2, although made of stainless steel, is a hermetically sealed hollow weight of average density 2.9 g/cm^3 and, consequently, it has a much larger volume than R2. Table II lists the important artifact characteristics.

DATA REDUCTION

The instructions supplied to each State contained the information required to perform a complete data reduction and assignment of the mass values to the four one-kilogram weights. These instructions included:

1. Least-squares matrix coefficients
2. Standard deviation calculation for the least-squares fit
3. Volumes and thermal expansion coefficients of the weights
4. Fractional weight calibration report for the weight set supplied in the package
5. Air density formula.

Each State was also asked to return the raw data to NBS with sufficient information to permit the use of the NBS 1182 computer and the mass code[2] for data reduction.

RESULTS

Each laboratory completed the measurements and reduced the data according to the instructions provided by NBS. The results varied in detail for many reasons including round-off errors attributed to hand calculators and the omission of thermal expansion corrections. Two laboratories made mathematical errors in the data reduction and were given the opportunity to correct them. The results of the State calculation, although not used, are given in table IV. These data indicate that most states had problems calculating the mass of H₂ as compared to the NBS calculated values.

Table III presents the NBS reduced values assigned to the artifacts, the series standard deviations, and corresponding F ratios, check

standard agreement, and T values. For the moment, consider the check standard values. Each series was solved based on the value of a single kilogram standard and not the sum of the two standards as is the usual method. The value assigned to the second kilogram standard, referred to here as the "check standard," is compared to its "accepted value" and any difference is given in the appropriate columns of table III. Based on the statistical "T" test, all values except one are within the measurement error limits. Of course, if the Standard and check standard change mass an equal amount, the difference would remain constant and incorrect mass values would evade the check.

All States performed the weighings on balances of the same make and model; Sandia and NBS have more modern models that in principle and performance, however, do not differ significantly from those belonging to the State laboratories. Standard deviations (Series S.D. in table III) for these instruments are given for each weight pairing. Experience with balances of this type indicates that a 60 μ g standard deviation is quite normal and better than the manufacturer's specifications. The calibration data are in agreement with this experience and indicate that all balances functioned normally during these measurements.

NBS assigned seven values to each weight before shipment and upon return from each laboratory. Prior to each calibration, all weights were vapor degreased and allowed several days to equilibrate. At least 24 hours before calibration the weights and standards were stored in an annex to the balance case to allow for further temperature equilibration. In table III the mean values for each of the weights are given with the corresponding standard deviations. The standard deviation values are typical of the methods and instrumentation used in these measurements.

It is useful to look at the mass values assigned by each participating laboratory by examining the differences between these values and the NBS mean value, as shown in table V. The reader should keep in mind that weights A1, S2, and R2 have nearly the same volume as the standards (see table I) and therefore most of the buoyant force is self-cancelling. Weight A1 closely resembles the standards in every way whereas S2 has a much larger surface area and it should be relatively more sensitive to surface related problems.

Using the range of values for A1 as a normal reference, it may be noted that the ranges of values for S2 and R2 are significantly larger; that is most likely caused by the thermal sensitivity of the weight geometry. The range of values assigned to weight H2 is much larger than the ranges for the other three weights. Moreover, the mean values assigned to H2 by the States show a large offset from the corresponding NBS value whereas the mean values of the other weights agree well within the uncertainty limits discussed in the next section. Although we could expect thermal problems to cause weight H2 to behave similarly to S2 and R2, additional errors in the buoyancy correction due to the lower average density of H2 are dominant here. With the techniques used by NBS and Sandia in this work the agreement between NBS and Sandia is typical of the agreement we should expect for a weight such as H2[3].

In summary, it is evident that a somewhat smaller range in the assigned values for weights A1, S2, and R2 could be economically achieved by the States with improved thermal control. Improvements in the range of assigned values for weights such as H2 will require significant improvements in the capability of States to measure air temperature, pressure, and relative humidity. These and other improvements are discussed in the last section of this report.

UNCERTAINTY ESTIMATES

Before the beginning of this program, the NBS standards B1 and D2 used in this work were suspected to have lost mass. A recent comparison with the platinum prototype kilograms does indicate a mass loss of 76 and 40 micrograms, respectively. It is also known that the two NBS working kilograms originally used in the calibration of all the State one-kilogram standards have changed. However, there are two partially compensating effects: mass is increasing from the lack of cleaning and, at the same time, there is a mass loss from abrasion that occurs with use. Therefore at this time it is premature to place realistic uncertainties on the accuracy of the measurements reported here. However it is possible to estimate a limit for the offset between the State kilograms and the NBS standards used in this work.

Recent recalibration of the NBS working kilograms that were initially used to calibrate the State kilograms (not B1 and D2) indicates a mass loss of 100 micrograms from the accepted value. In the early 1970's other measurements indicated that even larger departures from the accepted values could have been possible when the working standards were relatively clean. Therefore, there might be an offset between the State assigned weight value for weight A1 and the value assigned by NBS. At most, due to this cause, the State values should not be in disagreement with values assigned to A1 by NBS by more than 125 micrograms. Normally the uncertainty of weights such as A1, as assigned from a "four ones series" (see table I) where the restraint is a pair of standard kilograms is, in general

$$\text{Uncertainty} = 3(0.6124 \times \text{S.D.}) + \text{S.E.}$$

where S.E. is the systematic error for the standards and S.D. is the standard deviation of the least squares adjustment. For an ordinary one-kilogram NBS calibration this amounts to about 100 μg but it can vary with the balance used to make the measurement. For most of the participating laboratories an estimate based on the NBS calibration certificate for a State standard would be

$$3(0.6124 \times 60) + 100 = 210 \mu\text{g} .$$

FUTURE DIRECTION

There are two areas that must be improved to advance this project. The first priority is to reduce the work load on the staff of Division 523 and the State metrologists and the second priority is to recalibrate the State one-kilogram standards with improved techniques. The first priority will be met by reducing the number of measurements made at NBS and by shipping a portable microcomputer with data reduction software as a part of the weight package so that each participant can reduce the data using validated methods in his own laboratory.

Other improvements that will be made in future measurements include: a prescribed procedure for thermal conditioning of the weights, the addition of calibrated air density instrumentation in the weight package and the addition of two one-kilogram standard masses that will enable the States to recalibrate their one-kilogram standards.

ACKNOWLEDGMENTS

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Table I

The sequence of observations known as a "four ones series"[4] for both pairs of artifacts calibrated by the State participants in terms of their standards.

OBSERVATION ORDER	OBSERVED DIFFERENCE	KILOGRAMS PAIR 1			
		STATE 1	STATE 2	A2	S2
1*	1	+	-		
2	2	+		-	
3	3	+			-
4	4		+	-	
5	5		+		-
6*	6			+	-

		KILOGRAMS PAIR 2			
		STATE 1	STATE 2	R2	H2
1	1	+	-		
2	2	+		-	
4	3	+			-
3*	4		+	-	
5	5		+		-
6*	6			+	-

*Air density parameters recorded.

Table II

Artifact Characteristics

KILOGRAM DESIGNATION	VOLUME (cm ³) @ 20°C	ASSUMED THERMAL COEFFICIENT CUBIC (ppm)	APPROXIMATE SURFACE AREA (cm ²)
B1	127.3852	45	145
D2	127.6250	45	145
STATE 1	125*	45	145
STATE 2	125*	45	145
A1	127.384	45	145
S2	126.545	45	660
R2	126.392	45	270
H2	337.666	45	270

*Assumed based on nominal mass and a density of 8.0 g/cm³ at 20 °C.

Table III

Calibration Results

LAB	DATE	A1 1000	S2 999	R2 999	H2 1000	Pair #1			Pair #2				
						SERIES S.D.	F RATIO ⁴	CHECK STD. ACCEPT - OBS	T VALUE ⁵	SERIES S.D.	F RATIO	CHECK STD. ACCEPT - OBS	T VALUE
NBS	4/82	.009028 g	.988438 g	.994439 g	.004012 g	29 µg	0.242	-16 µg	-0.74	101 µg	2.842	-21 µg	-0.55
NBS	6/82	.009170	.988631	.994594	.003863	34	0.132	-42	0.55	22	0.317	-24	0.99
NBS	7/82	.009181	.988596	.994493	.003891	18	0.090	-92	2.16	27	0.208	-64	1.53
NBS	9/82	.009119	.988629	.994531	.003789	58	0.942	-39	0.93	58	0.919	-10	0.24
NBS	1/83	.009153	.988575	.994467	.003854	25	1.548	-73	0.02	75	0.180	-1	1.72
NBS	2/83	.009113	.988525	.994469	.003854	31	0.273	-50	-1.17	42	0.490	-126	2.97
NBS	4/83	.009157	.988479	.994529	.003895	34	1.379	-84	0.68	70	0.324	-28	1.97
\bar{X}		.009132 g	.988553 g	.994503 g	.003880			-56.6				-39.1	
S.D.		52.0 µg	74.9 µg	52.3 µg	67.9 µg			27.3				43	
New York	5/82	.009295	.988625	.994655	.005980	21	0.263	-86	3.04 ³	35	0.350	-62	1.45
CA	6/82	.009301	.988789	.994742	.004544	47	0.612	-4	0.09	42	0.493	-31	0.73
Nevada	8/82	.009070	.988476	.994471	.004643	79	1.747	-71	-1.68	65	1.163	84	-1.97
Sandia ¹	11/82	.009102	.988739	.994436	.004143	61	2.741	14	-2.63	60	2.849	2	-1.589
Colorado	2/83	.009068	.988602	.994580	.004642	54	1.172	4	-0.89	65	0.816	-38	0.08
Utah ²	3/83	.009239	.988689	.994788	.003816	34	0.323	-38	0.90	54	0.157	5	-0.12

¹Sandia used weight pairing A1-R2, S2-H2.²Utah submitted extra measurements that are not included.³Check out of control.⁴Critical value is 3.79 for probability = 0.01.⁵Critical value is 3

Table IV

State participants data reduction.

Kilograms:	Assigned Mass Values			
	A1	S2	R2	H2
	1000 g	999 g	999 g	1000 g
New York	.009302	.988628	.994656	.005920
California	.009303	.988787	.994744	.004497
Nevada	.009107	.988485	.994433	.004384
Colorado	.009076	.988551	.994543	.003347
Utah	.009244	.988679	.994780	.004115

Table V

NBS values minus participant values (as calculated by Division 523)

LABORATORIES IN MEASUREMENT ORDER	KILOGRAM DESIGNATION				
	A1	S2	R2	H2	
New York	-163 μg	-72 μg	-152 μg	-2100 μg	
California	-169	-236	-239	-664	
Nevada	62	77	32	-763	
Sandia	30	-186	67	-263	
Colorado	64	-49	-77	-762	
Utah	-107	-136	285	64	
\bar{X}	=	-47	-100	-14	-748
RANGE	=	233	313	524	2164

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11. ABSTRACT <i>(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)</i> <p>This report describes the calibration of four one-kilogram objects by five State laboratories. The project is of mutual interest to the Length and Mass Measurements and Standards Division (523) and to the Office of Weights and Measures (120). The work is ongoing at the present time, however, the initial phase is completed and reported here. Also discussed is the proposed direction of future work.</p>			
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