

# NBS TECHNICAL NOTE 1035

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

# A Portable Calibration Densimeter for Use in Cryogenic Liquids

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# A Portable Calibration Densimeter for Use in Cryogenic Liquids

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NBS technical note

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#### A PORTABLE CALIBRATION DENSIMETER FOR USE IN CRYOGENIC LIQUIDS

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A portable densimeter designed specifically for calibrating liquefied natural gas densimeters but suitable for density measurement of a wide range of liquids, is described. The densimeter has been compared to the Density Reference System densimeter at the National Bureau of Standards and found to agree to well within the combined systematic error. The density results of this instrument and that of the DRS densimeter are statistically indistinguishable.

Key words: Densimeter; density reference system; liquid methane; LNG.

#### 1. INTRODUCTION

The cryogenic liquids Density Reference System [1] (DRS) at the National Bureau of Standards in Boulder, Colorado, originated as a device for testing calibration and performance of commercially built densimeters for liquefied natural gas (LNG) service. A number of commercial densimeters were tested with this system and the results reported [2]. All the densimeters tested functioned adequately. These tests, however, disclosed a need for better calibration methods for the commercial densimeters.

We implemented a transfer standard program at NBS as a method of providing a suitable calibration for commercially built LNG densimeters. In this program, a manufacturer or user of densimeters selects a commercial densimeter against which he will calibrate his densimeters by comparison in a homogeneous fluid. We periodically calibrate his selected densimeter in the DRS and this becomes the transfer standard.

We rebuilt the density reference system into a flexible configuration so densimeters of various designs could be more easily introduced and more rapidly calibrated [3]. The new DRS was designed also with the intention of demonstrating a type of system that could safely handle combustible gases in an industrial calibration laboratory since for best results, a densimeter should be calibrated in a fluid similar to that in which it will be used. After the DRS was rebuilt, the availability of an electronic balance capable of weighing 200 g to 0.001 g resulted in our rebuilding the DRS densimeter to incorporate this balance. We found this new balance both compact and sufficiently robust to suggest the possibility of constructing a portable silicon densimeter. This portable densimeter could then be installed in any commercial calibration facility to calibrate the reference densimeter of the facility. This method has the definite advantage that it permits us to test a whole facility rather than just the calibration densimeter used in the facility. The transporting of this portable densimeter should not add any additional uncertainty to the instrument since the reference weight makes it self-calibrating.

We have built such a portable densimeter and tested it in the DRS. We describe this densimeter and these comparison measurements in this report. From these results, we have generated an accuracy statement for this densimeter.

### 2. THE PORTABLE REFERENCE DENSIMETER

The Portable Reference Densimeter (PRD) shown in fig. 1 is very similar to the DRS densimeter described in ref. 3. The PRD is an Archimedes type densimeter consisting of a silicon single crystal float of accurately known weight that is weighed while immersed in the liquid whose density is desired. We weigh the 162 g silicon single crystal with a commercially built electronic balance capable of weighing 220 g to 1 mg. The sensing unit has been removed from the instrument case and installed in a 178 mm diameter closed aluminum case designed to withstand an internal pressure in excess of 1 MPa.

A simplified drawing of this instrument is shown in fig. 1. "A" is the silicon single crystal shown lifted from the suspension cage "B" and clamped between supports "C" and "D". The bottom supports "D" are raised when the 15 mm ID thin walled stainless steel tube "E" is lifted by a pneumatic cylinder actuating the bell crank mechanism "F". The upper supports are fixed to the 25.4 mm OD by 0.6 m long thin wall stainless steel tube "G". This length was dictated by the distance from the top plate to the sample liquid in the DRS where it was tested and may be changed. The cage "B" is suspended from the electronic balance sensing unit "H" by the 1.6 mm diameter tube "I". The balance "H" is mounted on a cantilevered plate "J" so that should gas tight balance case "K", capable of holding 10 bar interval pressure, flex when pressurized the leveling of the balance is unaffected. This permits a lighter construction for the balance case than used for the DRS densimeter. The tungsten reference weight





"L", used to monitor the balance calibration, is shown placed on its suspension cage "M". This cage replaces a section of the suspension tube "I". Some swivel joints in this suspension permits flexing but not rotation. A pneumatic-cylinder driven bell crank, similar to "F" but not shown in the drawing raises "N" which lifts the reference weight "L" off the suspension and clamps it against the fixed top support "O". The "O" ring seal at "P" connects this densimeter into an LNG sample container. This joint is a gland seal rather than a flange seal so the instrument can be tilted according to the bull's eye level "Q" to align the suspension "F" to tube "E". A gas tight seal "R" is provided for the electrical leads "S". The case "K" is clamped together with sufficient bolts through the flanges to withstand a 10 bar internal pressure.

The apparent mass of the reference weight is approximately the same as that of the silicon float when the latter is suspended in liquid methane. We compared this apparent mass value, corrected to a true mass value by using the temperature and pressure of the sample gas to determine the buoyant effect, to the true mass values of the tungsten as determined by precision weighing. This procedure provides a continual monitoring of the balance calibration. Tungsten was chosen for the reference weight because its high density reduces the magnitude of the buoyancy correction hence any effect of uncertainty in the gas density in the region of the reference is minimized.

#### 3. DENSITY MEASUREMENT AND SYSTEMATIC UNCERTAINTIES

The density of the liquid in which the float is immersed is given by the relation [3]:

$$\rho = \rho_{\rm S} (1 - (M_{\rm a} - M_{\rm a0})/M_{\rm s})$$
(1)

where  $M_s$  (=162.2502 ± 0.0005 g, 99% confidence interval) is the true mass of the silicon single crystal,  $M_a$  is the apparent mass value with the silicon on the suspension and immersed in the liquid,  $M_{a0}$  is the weight of the suspension in the liquid (this reading is zero ± 0.001 g as a general rule because the balance is zeroed in this configuration) and  $\rho_s$  is the density of the single crystal silicon at the liquid temperature and pressure. The silicon single crystal density at 110 K from ref. 2 is assumed to be 2.3308 ± 0.00007 g/cm<sup>3</sup>. This includes both the estimated experimental uncertainty in the density and the thermal expansivity.

The true mass values of the silicon crystals and the reference weights for the DRS and the PRD are given in table 1 along with an estimate of total uncertainty for each value. Total uncertainty is defined as t/- the sum of the systematic error limit plus the 99% confidence limit for the average of the n weighings. Each true mass value is an average of n weighings made on separate days. The systematic error in each case is the systematic uncertainty specified for the set of class S weights used.

The estimated errors for the various measured quantities are shown in table 2 which is similar to the same table in ref. 3. We have not included the error for temperature gradient and drift of a liquid sample [3] because we are now examining the systematic error of the densimeter only, not the densimeter plus sample container. As before [3], the total systematic error shown in table 2 is assumed to be the square root of the sum of the squares of the individual contributions.

If the densimeter is used in liquids at temperatures well removed from 110 K an expression for  $\rho_{S}(T)$  is related to the 0°C density by

$$\rho_{c}(T) = \rho_{c}(0^{\circ}C)(1 - 3 \beta(T) + \text{negligible terms})$$
(2)

where

$$\rho_{\rm s}(0^{\circ}{\rm C}) = 2.32940 \pm 0.00007 \, {\rm g/cm}^3$$

and the thermal expansivity

$$\beta(T) = - (L(273.2) - L(T))/L(273.2)$$
(3)

where L is a crystal dimension. Gibbons [4] gives values of  $\beta(T)$  for T = 40 to 300 K at intervals of 10 K. He reports the probable error for expansivity to be less than 0.5% at each point. Gibbons does not define probable error, but in one definition [6] it is 0.6475 times the standard deviation. This suggests that his estimated standard deviation for expansivity is less than 0.8% for each value. He also does not mention if inherent systematic error in his experimental apparatus was considered. Differences between his values and those of other experimenters are noted in the paper.

As a possible aid to interpolation in Gibbon's table for the expansivity of silicon the following fourth degree polynemial was fit to his values of  $\beta(T)$  for T = 70 to 300 K:

TABLE 1. Weight Values

			*99%	Systematic		
	Avg of n		Conf Limits	Error from	Total	% Total
	Measure-		for	Class S	Uncer-	Uncer-
	ments	<u>n</u>	Average	Weights	tainty	<u>tainty</u>
DRS Silicon Crystal	127.4351g	6	<u>+</u> .00022g	<u>+</u> .00020g	<u>+</u> .00042g	<u>+</u> .0003
DRS Reference Weight (brass)	102.9022g	1	<u>+</u> .00055g	<u>+</u> .00017g	<u>+</u> .00072g	<u>+</u> .0007
PRD Silicon Crystal	162.2502g	5	<u>+</u> .00025g	<u>+</u> .00024g	<u>+</u> .00049g	<u>+</u> .0003
PRD Reference Weight (tungsten)	132.2457g	1	<u>+</u> .00055g	<u>+</u> .00018g	<u>+</u> .00073g	<u>+</u> .0006

\*From t  $/\sqrt{n}$  where  $\sigma$  is estimated to be 0.00017g based on the pooled standard deviation of silicon weighings (9 degrees of freedom). The Student-t statistic for a 99% confidence interval in this case is 3.25.

# Table 2. Systematic Errors'

+

Variable, x	90/9X	<u>Max Magnitude</u>	δχ	<u> 20/0(%)*</u>
Silicon crystal mass	(ρ <sub>S</sub> - ρ)/M <sub>S</sub>	0.012 cm <sup>-3</sup>	$5 \times 10^{-4}$ g	0.0015
Apparent masses silicon	-p <sub>s</sub> /M <sub>s</sub>	0.015 cm <sup>-3</sup>	0.001 g	0.0038
Suspension	ρ <sub>s</sub> /M <sub>s</sub>	$0.015 \text{ cm}^{-3}$	0.001 g	0.0038
Silicon density	۵/۵ <sup>°</sup>	0.17	$7 \times 10^{-5} \text{ g/cm}^{-3}$	0.0030
TOTAL				0.006

\*for  $\rho$  = 400 kg/m<sup>3</sup>

$$\beta(T) = (-0.11620 - 7.72448 \times 10^{-4}T - 5.40814 \times 10^{-6}T^{2} + 6.2707 \times 10^{-8}T^{3} - 9.83286 \times 10^{-11}T^{4}) \times 10^{-3}$$

(4)

The residual standard deviation from this fit is 9.6 x  $10^{-7}$ . The target absolute residual of the polynomial values from those of Gibbons is 2.5 x  $10^{-6}$  which occurs for T = 240 K. This maybe an aberrant point, the next largest in absolute value is  $-1.7 \times 10^{-6}$  for T = 90 K. A plot of these residuals show a non-random pattern, so another method of interpolation may improve the fit. The estimated standard deviations for the values of  $\beta(T)$  predicted by the above polynomial range from 7.8 x  $10^{-7}$  at 70 K and 300 K to 3.4 x  $10^{-7}$  at 150 K. If we take three times the largest of these, 2.3 x  $10^{-6}$ , as our estimated random uncertainty for  $\beta(T)$  then the systematic error eq (4) introduces into the density determination is approximately  $|\partial_{\rho}/\partial_{\beta}|_{T=0^{\circ}C} \times (2.3 \times 10^{-6})$ , where  $|\partial_{\rho}/\partial_{\beta}|_{T=0^{\circ}C}$  is, differentiating eq (2), 3 x 2.3294 = 7.0 g/cm<sup>3</sup>. Thus, if eq (4) is used fo the  $\beta(T)$  calculation, another 1.6 x  $10^{-5}$  g/cm<sup>3</sup> (.007%) would be added in quadrature to the total in table 2. Even if this error were a factor of 10 larger, it would contribute little to the total systematic error.

4. CALCULATIONS OF THE RANDOM UNCERTAINTY FOR THE PORTABLE DENSIMETER

The PRD has been tested in the Density Reference System. The accuracy and precision of the DRS densimeter and the portable densimeter are essentially independent of liquid composition. Their accuracy and precision depend primarily upon the accuracy and precision of the balance and how well the weight and density of the silicon crystals are known. The ability to make an accurate comparison will, of course, depend upon conditions within the sample holder. The liquid needs to be relatively quiet, well mixed and of a fairly uniform temperature in the volume in which the measurements are being made.

The liquids chosen for comparing the two densimeters were saturated liquid methane and a saturated LNG like mixture. The DRS densimeter has been tested in those liquids [3]. The comparison of the two densimeters is based on data taken from two fillings of the liquid sample holder, for a total of 35 density values.

We first filled the DRS sample holder with liquid methane, took a series of measurements in the range of 110 to 125 K, then added nitrogen (about 1% to 2%) and propane (about 4% to 6%) to simulate LNG and took another series of measurements in the same temperature range.

The series of measurements for a liquid sample in the DRS were made by either heating or cooling the saturated liquid to a target temperature, waiting for the liquid to come to equilibrium, and taking the desired schedule of readings. The sample was stirred and the readings were repeated giving a duplicate set of readings for each temperature. It is reasonable to assume that the random error of the duplicate density values described above would correlate more closely than values obtained at another temperature.

Because of the large correlation between duplicate readings, only one of each of the duplicates can be usefully employed in the analysis. We have arbitrarily chosen to use the first of each duplicate in our analysis. The second of each would provide essentially the same information. Using both duplicates would complicate the analyses without providing any increase in information.

The model used to analyze the data is:

$$\Delta \rho_{ij} = \rho_{Tij} - \rho_{Dij} = \mu + \alpha_i + e_{ij}$$
<sup>(5)</sup>

where  $\rho_{\text{Tij}}$  is the jth density determined by the portable densimeter on the ith set of runs,  $\rho_{\text{Dij}}$  is the corresponding density value determined by the DRS densimeter,  $\mu$  represents any systematic difference between the two methods,  $\alpha_i$ represents a shift in the mean for measurements on the ith set of runs, and  $e_{ij}$ represents any random contribution to the jth measurement on the ith set of runs. A set of runs are those measurements made with a particular mixture in the liquid sample holder.

Figure 2 is a plot in chronological order of the  $\Delta \rho_{ij}$ . There are four sets of runs shown in this figure, two for methane and two for the LNG like mixture. The symbol "M" is for a methane value and "L" is for LNG. In this plot can be seen the extent of any shifts,  $\alpha_i$ , in the mean of the  $\Delta \rho_{ij}$  from one set of runs to the next. We know from our study of the DRS, ref. 3, that such shifts occur and appear to be random. In the DRS study, only methane data was used to study the variability of the  $\alpha_i$  because the comparison was between the DSR densimeter and the Haynes-Hiza [5] temperature relationship for saturated liquid methane. Because the Haynes-Hiza relationship depends on pure methane, the  $\alpha_i$  may have partly been the function of contaminants in the methane. The 99% upper bound on the standard deviation of the  $\Delta \rho_{ij}$ , taken from ref. 3, is .036 kg/m<sup>3</sup>. This is the bound we take to represent the variability from one set of runs to the next for each of the two Archimedes densimeters that we are



Reference System. The L and M symbols are for an LNG like mixture and Differences between the densities measured by the Portable Reference Densimeter and the Density Reference System densimeter in the Density

liquid methane, respectively.

Figure 2.

comparing; so that the 99% upper bound on the  $\Delta \rho_{ij}$  could be  $\sqrt{2} \times .036 \text{ kg/m}^3$ = .051 kg/m<sup>3</sup>. For the four sets of runs considered in this study, the standard deviation of the  $\alpha_i$  for the means of the  $\Delta \rho_{ij}$  is estimated to be 0.007 kg/m<sup>3</sup>. So we do not see any conflict in assuming, for the time being, that the new portable densimeter will behave in a manner like the DRS densimeter with respect to the variability in the  $\alpha_i$ .

Based on the  $\Delta \rho_{ij}$  shown in fig. 2, the  $e_{ij}$  has an estimated standard deviation of 0.0099 kg/m<sup>3</sup>. An upper 99% bound on the standard deviation is 0.014 kg/m<sup>3</sup>. If we were to divide this equally between the two densimeters, then the 99% upper bound for the standard deviation for variability within sets of runs would be 0.01 kg/m<sup>3</sup> for each densimeter. This value is less than expected based on the results presented in ref. 3, and suggests that our method of computing the density using the averaged sample temperature may have contributed a significant amount of the variability in our study of the DRS.

Each balance is calibrated by weighing its reference weight in air and adjusting the balance to read the correct apparent mass value in air, not the true mass value. Each reference weight has approximately the same apparent mass value as the corresponding silicon crystal when the latter is submerged in liquid methane.

As a test of the densimeter balance calibrations each reference weight is measured and recorded. At each sample temperature setting, the measured apparent mass value is corrected for gas buoyancy and compared to the true mass value. This correction is readily made for methane or methane-nitrogen samples but not for methane-propane-nitrogen samples because propane suppresses the methane vapor pressure by an uncertain amount and because the gas mixture is slow to reach equilibrium. The method of calculating the buoyancy correction is detailed in ref. 3. The reference weight of the PRD is located in ambient temperature gas. This arrangement and the tungsten weight reduce the buoyancy correction thus reducing associated uncertainties. The true mass value of the reference weight for the DRS densimeter is 102.9022 g with a total uncertainty from table 1 of + 0.72 mg. Comparison of the reference weight readings for methane and LNG, showed some differences which are attributed to the buoyancy corrections made for the two liquids. A 99% confidence interval for the average of the reference weight true mass values measured in the methane atmosphere is 102.9024 g + .0004 g and that for the average measured in the LNG vapor is 102.9015 + .0004 g.

While a statistical difference is present between these two values, the difference has no practical significance and both are consistent with the accepted true mass value of the reference weight.

The measured true mass value of the reference weight for the portable densimeter is 132.2457 g with a total uncertainty from table 1 of  $\pm$  0.73 mg. Comparison of the average true mass value of the reference weight measured in both methane and LNG atmospheres shows no significant difference between the two values. A 99% confidence interval for the combined average in methane and LNG atmospheres is 132.2455  $\pm$  .0003 g, which is consistent with the accepted true mass value for the reference weight.

The analyses of the reference weight true mass values taken during the comparison of the two densimeters show the balance of both densimeters to be both stable and accurate.

#### 5. CONCLUSIONS

From our experience with the improved DRS densimeter, we determined that a portable silicon densimeter of essentially the same design and similar accuracy is feasible. We have built such a densimeter and compared its performance to the DRS densimeter by installing it in the DRS. Its performance exceeds the performance of the DRS mainly because of the larger silicon crystal and the relocation and higher density of the reference weight.

The total uncertainty of a single measurement we estimate to be the systematic uncertainty added to three times the estimated within run and the between run variability added in quadrature:

$$3(.036^2 + .014^2)^{1/2} = 0.116 \text{ kg/m}^3$$
 (6)

At a density of 422.63 kg/m<sup>3</sup> (methane normal boiling point) the  $3\sigma$  uncertainty is .027% and, adding the systematic uncertainty of 0.006%, the total uncertainty of one measurement is 0.033%.

The comparison of the DRS and PRD densimeters has also shown the systematic uncertaintly of 0.022% for the DRS is over estimated. The agreement between the measured density values suggests that this sample temperature gradient used for the systematic error estimate in ref. 3 is too large. The estimated gradient is the dominant contribution to the systematic error estimate for the DRS [3]. The gradients across the sample in the region of the silicon crystals, whose centers are separated by 15 mm, must be much smaller than estimated in ref. 3.

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