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Cryogenic Flow Research Facility Provisional Accuracy Statement

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PREFACE

It is the policy of the National Bureau of Standards to use the International System of Units (SI) in its reports unless this usage would lead to confusion or a lack of understanding. In this report SI units have not been used because it is standard practice in the industry to use English units and the use of SI units would be unduly cumbersome. The following conversions are given so the reader can convert the values in the report to SI units if he desires.

$$\text{feet}^3 \times 0.028317 = \text{meter}^3$$

$$\text{gallons} \times 0.0037854 = \text{meters}^3$$

$$\text{gallons/minute} \times 0.00006309 = \text{meters}^3/\text{second}$$

$$\text{inches} \times 0.0254 = \text{meters}$$

$$\text{pounds} \times 0.45359 = \text{kilograms}$$

$$\text{pounds/inch}^2 \times 6894.757 = \text{newtons/meter}^2$$

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CRYOGENIC FLOW RESEARCH FACILITY
PROVISIONAL ACCURACY STATEMENT

J. W. Dean, J. A. Brennan, D. B. Mann, and C. H. Kneebone

The National Bureau of Standards and the Compressed Gas Association have jointly sponsored a research program on cryogenic flow measurement. A cryogenic flow research facility was constructed and was first used to evaluate commercially available cryogenic flowmeters operating with liquid nitrogen on a positive displacement principle.

The performance of the flow facility was simultaneously being evaluated during the meter tests. This is a summary report of the performance evaluation of the flow facility. An accuracy statement is given for both totalized mass and volumetric flow.

Key Words: Accuracy statement; cryogenic; flow facility; liquid nitrogen; measurement.

1. Introduction

The historical background, design, and operation of the Cryogenic Flow Research Facility have been given in Mann [1971]. This report describes the present status of our knowledge of our ability to make cryogenic flow measurements after a year of operation of the facility. The error analysis presented here details just the contribution of the flow facility itself to the uncertainty of flow measurements. Subsequent reports will deal with the problems and uncertainties in using the facility in research measurements and in evaluation of flowmeters. The uncertainties given here are, in a sense, limits to the discrimination possible in those investigations.

2. Provisional Accuracy Statement

The following accuracy statements should be considered provisional and are currently restricted to flow rates between 20 and 100 gpm, pressures from 50 to 100 psig, and temperatures of 80 to 90 K, and for

ideal conditions where operational or equipment malfunctions are not present. As results of future tests become known, the accuracy statements will be improved.

2.1 Mass Flow

At this time the uncertainty of the measurement of totalized mass flow is estimated to be ± 0.18 percent. This figure includes an uncertainty of ± 0.12 percent for known sources of systematic errors plus an uncertainty of ± 0.06 percent for random error. The estimated uncertainty due to the random error is three times the standard deviation calculated from 23 applications of the calibrated masses over a period of three months.

2.2 Volumetric Flow

At this time the uncertainty of the measurement of totalized volumetric flow of liquid nitrogen is estimated to be ± 0.47 percent. This figure includes an additional uncertainty of ± 0.39 percent for known sources of systematic error in the density determination, which is added in quadrature to the uncertainty due to systematic error in the mass measurement. The major contribution to the uncertainty in liquid nitrogen volumetric flow measurement is the uncertainty in the liquid density, which can only be improved by new PVT property measurements.

When the objective of using the accuracy statement is in the mediation between seller and buyer, where both have accepted the same values of density, the uncertainty in the density need not be considered. Under this condition the uncertainty of the measurement of totalized volumetric flow is ± 0.18 percent, because the contribution of the pressure and temperature measurements error is negligible.

3. Methods of Obtaining the Accuracy Statement

The estimated uncertainty of a measurement is composed of the estimated imprecision plus an estimate of the limits to possible systematic error. The methods available for determining the imprecision of cryogenic flow measurement are:

- 1) Measurement of the static imprecision from repeated applications of the calibration weights.
- 2) Measurement of the dynamic imprecision of the combined meter and flow system.
- 3) Measurement of the dynamic imprecision of the flow system by series testing two or more meters and performing an analysis of variance calculation with the resulting data.

In measuring static imprecision, the flow facility is brought to equilibrium pressure and temperature by circulating liquid nitrogen through the weigh tank. Repeated application of the calibration weights combined with the reading of load cell output under these conditions allows the calculation of the static imprecision of mass measurement. However, liquid is not being accumulated in the weigh tank and a different imprecision may be associated with this action. Thus, the static imprecision measurement does not include all the desired information. It is the static imprecision that is most commonly reported in the literature for cryogenic flow facilities.

Measuring the dynamic imprecision of the combined meter and flow system includes any effect of accumulating liquid in the weigh tank, but it also includes the contribution of the volumetric meter imprecision. Meters have substantial imprecisions that tend to mask the contribution of the flow system. At best, meters can only indicate an imprecision that is larger than the true flow system imprecision. Since only volumetric cryogenic flowmeters were available, the imprecision of the

density measurement required to compare the performance of these volumetric meters to the gravimetric flow system is included.

The best method of separating the dynamic imprecision of the flow system from the imprecision of the meters is to perform a series meter test and to make the analysis of variance calculation. Modification of the flow facility to permit series testing of two meters is underway. The imprecision reported are found by methods (1) and (2) using the most consistent meter data available.

Systematic errors may be investigated by:

1. Propagating the effect of the indicated systematic errors in the calibration of the measuring system components.
2. Plotting the deviations between the meter and flow system performance as a function of the flow system parameters.
3. Using a flowmeter as a transfer device and intercomparing the results of tests of the same meter on different facilities.
4. Changing components of the measuring system and comparing results.

The critical components of the measurement system have been calibrated by the appropriate groups within NBS.

The deviations of all meters evaluated have been plotted as a function of pressure, liquid temperature, density, weight accumulated, degree of liquid subcooling, mass flow rate, and the order in which the data were taken. The presence of a dependency on any of these parameters for all meters would indicate the presence of a systematic error.

Some of the volumetric meters tested on the cryogenic flow research facility had previously been evaluated on private industrial cryogenic flow facilities. Where appropriate, data obtained on the NBS

and the private facilities are compared. Discrepancies indicate a systematic error, but the systematic error cannot be charged to either facility without further information.

The technique of changing components of the flow facility and comparing the results has not been used to determine the presence of systematic error because the components are unique. Experimental evidence from the first three of the above four methods are discussed below.

4. Weigh System Calibration

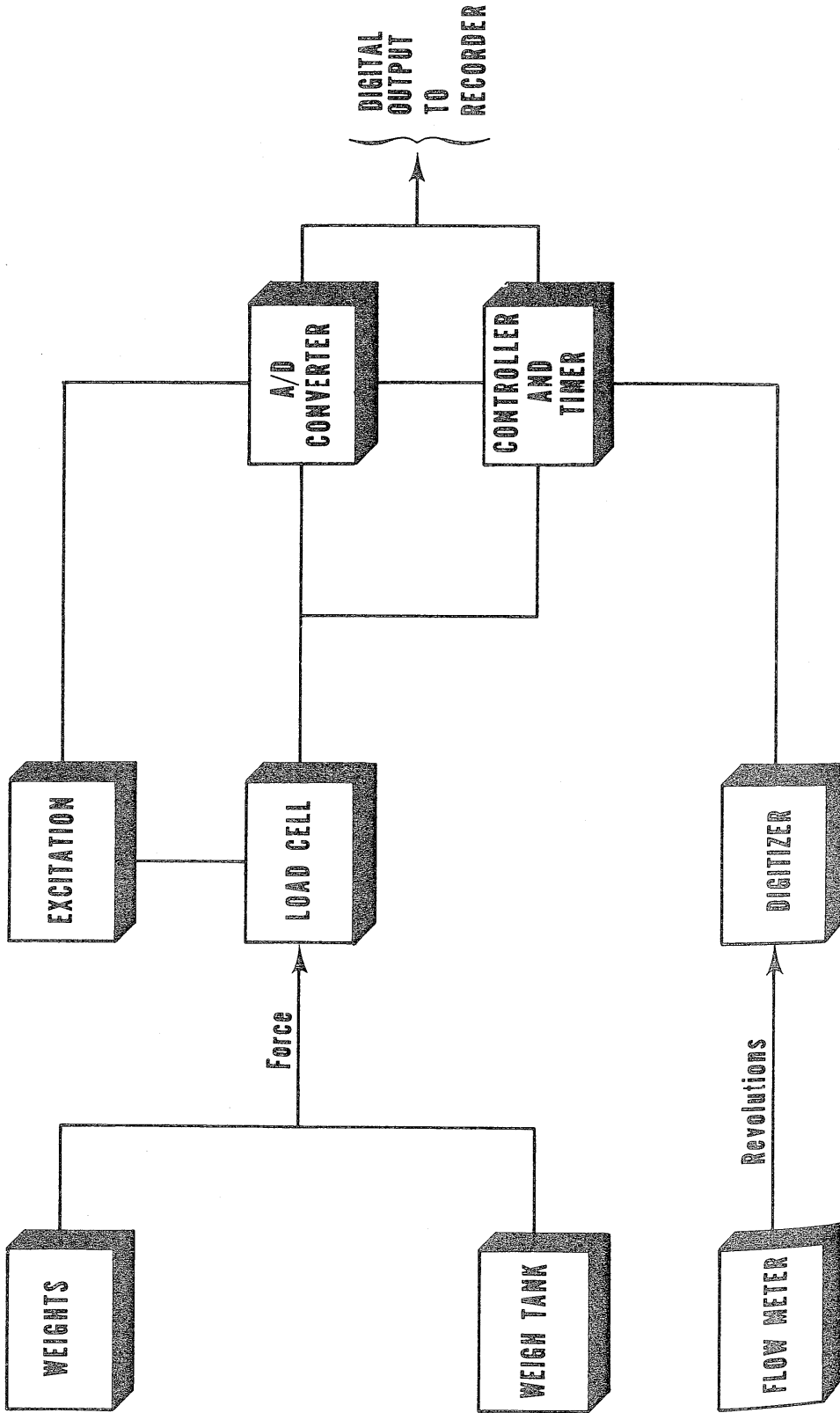
A schematic diagram of the flow measurement system is seen in figure 1. Either the calibration weights or the weigh tank can be supported from the load cell. The load cell receives a 10 volt excitation and produces an analog output voltage proportional to the applied weight. Both the excitation and the output signal are read by an integrating digital voltmeter converting the analog signal to a digital signal that is recorded. The flowmeter under test either generates, or is adapted to generate, a digital output. Integer pulses are counted, timed, and used to synchronize the reading of the load cell at the start and end of the test draft by the controller.

The performance of the weigh system is determined by the measurement of the load cell sensitivity by the operation of the calibrator. The load cell sensitivity is described by the relationship

$$S = \frac{E_2 - E_1}{WV} \quad (1)$$

where E_2 and E_1 are the load cell output voltages measured by an integrating digital voltmeter. W is the weight of the brass cylindrical

Measurement System



masses of the calibrator, and V is the load cell excitation voltage measured by the same integrating digital voltmeter. An estimate of the uncertainty of the ability to determine the load cell sensitivity requires a knowledge of the systematic and random errors associated with the brass cylindrical masses and the integrating digital voltmeter-load cell system. The masses have been calibrated yearly and are used to calibrate the voltmeter-load cell combination.

Recalibration of the four nominal 250-pound brass cylindrical masses required that the calibrator be removed from the catch and weigh tanks and disassembled. After recalibrating the masses and re-assembly of the calibrator, the calibrator was placed on a test table and the load cell sensitivity was determined using the brass cylindrical masses and four additional cast iron nominal 50-pound masses. The calibrator was then installed on the catch and weigh tanks and the load cell sensitivity determined and compared to the value obtained on the test table.

The following sections describe the calibrations and calculations performed in order to better estimate the uncertainty of the load cell sensitivity. These results are then used to calculate the uncertainty in the mass determination.

4.1 Mass Calibration

The four nominal 250-pound brass cylinders used to perform the in-place calibration of the load cell were calibrated against a set of mass standards maintained in Boulder by the NBS Electromagnetics Division. The set of standards consists of one 25-pound and two 50-pound brass masses, and a set of smaller stainless steel masses which have been compared to the National Standard of Mass. The test reports and descriptive information are included in Appendix A.

The above mass standards were used to calibrate a working set of masses consisting of cast iron nominal 50-pound blocks. This calibration was done on a Russell [1955] equal arm balance.

Our nominal 250-pound brass cylinders were calibrated on the same Russell equal arm balance against the brass, the cast iron, and the small stainless steel masses as required. This measurement did not make a correction for the buoyancy of air and the difference of density of the weights. This results in a possible systematic error estimated to be +0.002 percent. The total uncertainty in the 150 pounds of standard masses used in the comparison is less than ± 0.00008 percent in 250 pounds. The uncertainty in the 100 pounds of cast iron masses is somewhat larger, but still sufficiently small that the total uncertainty in the 250 pound brass cylinders is conservatively estimated to be less than ± 0.01 percent.

Additional information available on the stability of the mass standards includes independent calibration of the 50-pound masses in 1965 and 1971 which agrees to within ± 0.001 percent, and calibrations of the 250-pound brass cylinders used in sensitivity calculations, in 1968 and 1970, agreed to within ± 0.001 percent.

Since the load cell senses force, it is necessary to determine what forces were exerted by the individual masses under the conditions of the experiment. Important experimental conditions were the air pressure of 12.3 psia at 70° F and local gravity of 979.615 cm/s², both measured at the Boulder Laboratories.

4.1.1 Gravitational and Buoyancy Corrections

Gravitational corrections were made on all masses by the expression

$$W_B = \frac{g_B}{g_S} W_S \quad (2)$$

where

- g_B = 979.615 cm/s² at the experiment
- g_S = 980.665 cm/s² standard gravity
- W_B = weight at Boulder without buoyancy correction
- W_S = weight at standard conditions of 45° latitude sea level without buoyancy correction.

By definition W_S is equal to the masses certified by NBS. The value of g_B/g_S is equal to 0.998929.

In order to make buoyancy corrections for the calibration masses, the density of each mass must be known. The buoyancy corrections made here are approximations based on handbook values of the density. The buoyancy force is equal to the volume of the masses times the density of the displaced gas.

The density of air at 12.3 psia and 70° F is 0.063 lbs/ft³ or

$$F_b = \text{Mass/density of brass} \times 0.063 \text{ lbs/ft}^3.$$

The density of brass has been taken as 520 lbs/ft³. Thus, the weight of the flow facility cylindrical brass masses at Boulder are:

Mass Number		Boulder Weight (lb _f)
1	255.34 (0.998929 - 0.063/520)	255.03
2	247.72 (0.998929 - 0.063/520)	247.42
3	252.33 (0.998929 - 0.063/520)	252.03
4	247.53 (0.998929 - 0.063/520)	247.23

4.2 Voltmeter-Load Cell Calibration

The voltmeter-load cell calibration mechanism was reassembled and placed on a steel table with a hole cut in its center. This hole allowed a weigh pan to be attached to the load cell string. Four nominal 50-pound working masses were obtained from the NBS Boulder weight calibration laboratory. Thus, a total of 1200 pounds of mass in 50-pound increments was available to calibrate the load cell. A statistically designed calibration was performed by repeated application of the calibrated masses sequenced as follows: 0, 200, 50, 150, and 100 pounds.

The calibration mechanism was operated with the above masses in the described sequence with 10 ± 0.001 volt energizing the load cell. The millivolt output reading of the load cell was recorded with its corresponding applied force. The results are shown in table 1.

The sensitivity value and the uncertainty of the sensitivity value needs to be known in order to estimate the uncertainty associated with weighing a test draft of liquid. Therefore, sensitivity values are determined daily using the brass masses. Table 2 lists the typical results obtained. Since the load cell is within the pressure environment, it is subjected to system pressure. The sensitivity is a function of this pressure as shown in table 3. The uncertainty of the sensitivity value is estimated to be less than ± 0.02 percent.

The data in tables 1, 2, and 3 are consistent with the uncertainty due to non-linearity specified by the manufacturer of ± 0.05 percent (see Appendix B).

TABLE 1
 Voltmeter-Load Cell Calibration Data

Weight lb.	Voltage mV
0.00	0.944
255.03	8.616
502.45	16.061
754.48	23.646
1001.71	31.087
754.48	23.648
502.45	16.064
255.03	8.617
199.76	6.953
454.79	14.627
702.21	22.071
954.24	29.657
1201.47	37.098
954.24	29.661
702.21	22.077
454.79	14.630
199.76	6.956
49.94	2.446
304.97	10.120
552.39	17.564
804.42	25.149
1051.65	32.589
804.42	25.152
552.39	17.567
304.97	10.120
49.94	2.447
149.82	5.450
404.85	13.124
652.27	20.571
904.30	28.157
1151.53	35.598
904.30	28.158
652.27	20.573
404.85	13.127
149.82	5.452
99.88	3.950
354.91	11.624
602.33	19.069
854.36	26.653
1101.59	34.094
854.36	26.656
602.33	19.071
354.91	11.625
99.88	3.950

TABLE 2
Sensitivity Determinations

Date	Excitation Voltage (volts)	System Pressure (psig)	Sensitivity (mV/V - lb)		
			First Mass	Second Mass	Both Masses
11/18/70	10.0045	93.5	0.00300222	0.00300285	0.00300253
19	10.0057	94.1	0.00300225	0.00300007	0.00300118
20	10.0057	94.4	0.00300225	0.00300209	0.00300217
23	10.0054	59.2	0.00300469	0.00300783	0.00300677
12/07/70	10.0043	88.4	0.00300267	0.00300210	0.00300239
08	10.0048	67.2	0.00300487	0.00300518	0.00300502
09	10.0051	69.1	0.00300517	0.00300711	0.00300613
11	10.0043	75.6	0.00300463	0.00300332	0.00300398
14	10.0046	90.3	0.00300219	0.00300282	0.00300250
15	10.0049	88.2	0.00300327	0.00300112	0.00300221
16	10.0048	59.6	0.00300487	0.00300640	0.00300562
17	10.0047	70.6	0.00300412	0.00300360	0.00300386
21	10.0046	51.5	0.00300532	0.00300767	0.00300648
22	10.0041	58.0	0.00300508	0.00300661	0.00300583
01/19/71	10.0024	81.7	0.00300363	0.00300389	0.00300376
20	10.0014	60.4	0.00300511	0.00300459	0.00300485
21	10.0024	92.2	0.00300010	0.00300227	0.00300117
22	19.9984	74.7	0.00300444	0.00300388	0.00300416
25	10.0018	80.4	0.00300381	0.00300366	0.00300374
26	10.0028	75.9	0.00300234	0.00300670	0.00300463
26	10.0026	77.2	0.00300357	0.00300625	0.00300489
27	10.0037	77.3	0.00300402	0.00300430	0.00300416
28	10.0026	69.4	0.00300514	0.00300625	0.00300569

TABLE 3

Least Squares Estimate of Sensitivity
as a Function of Pressure
(50 - 100 psig)

Mass	Sensitivity, mV/V - lb
First	0.00301053 - 0.00000009 P
Second	0.00301510 - 0.00000014 P
Both	0.00301278 - 0.00000011 P

5. Static Imprecision of the Weigh System

The sensitivity of the load cell is measured each day the flow research facility is operated. This is done with the pump running, liquid passing through the weigh tank, and operating temperatures and pressures established. Known weights are placed on the load cell, the voltage recorded, and the load cell sensitivity calculated. The calculated sensitivities are normalized by applying the pressure correction. The standard deviation is calculated from data obtained over several months of operation, and the uncertainty due to imprecision is then calculated on three times the standard deviation. Since liquid is not accumulated in the weigh tank as the known weights are applied, dynamic effects are not included. The uncertainty calculated in this manner is defined as the uncertainty due to static imprecision.

Results from the load cell calibration are shown in figure 2 and the uncertainty due to static imprecision is ± 0.06 percent.

6. Imprecision of the Density

The liquid density is found by measuring the pressure and temperature and calculating the density from a state equation. The liquid nitrogen density data used are given by an equation of state defined by Strobridge [1962]. At the time Strobridge did the work, there were very few experimental data in the liquid region. The data available were located along the saturated liquid line or at high pressures (above 100 atm). No experimental data were available in the liquid region of current interest.

Recently, new experimental work has been done for nitrogen in the saturated and subcooled liquid region. This work has been performed by Streett [1968] and Terry [1969]. Terry reports that unpublished work

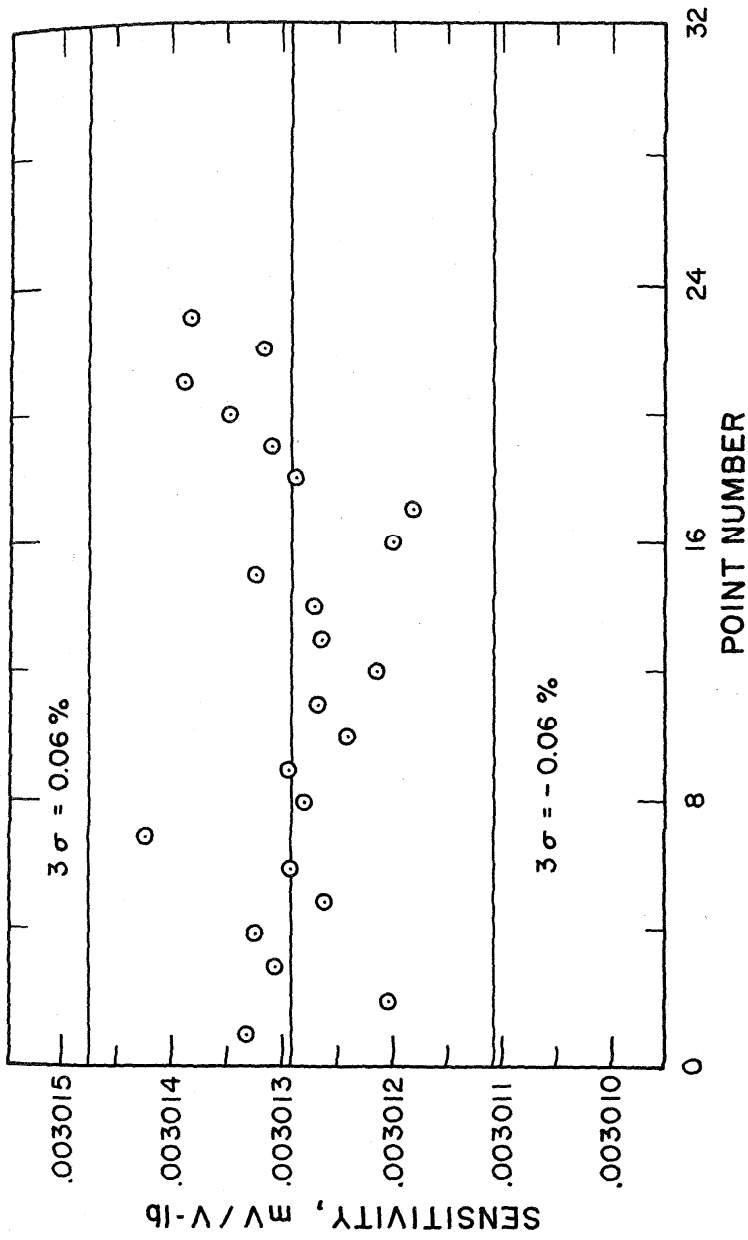


Figure 2. Load Cell Control Chart.

of the British Oxygen Company (BOC) on saturated liquid data for molar volumes is in agreement with his work to ± 0.05 percent. A comparison was made between Strobridge's calculated density data and the new experimental data in the range from 77 to 95 K and pressures to 100 psia. The data of Streett, Terry, and BOC give larger molar volumes on the order of 0.3 to 0.4 percent over this region. These differences are within the average uncertainty in density of ± 0.39 percent claimed by Strobridge.

The above comparison indicates that the density values, in the stated pressure and temperature range given by Strobridge, might be brought into agreement with the new data by multiplying Strobridge's density data by 0.9965. This correction has not been made in the present meter performance work in order to facilitate intercomparison with measurements using the data of Strobridge.

Temperature and pressure measurements must be made to determine the density. The density error associated with these measurements may be estimated from the isothermal compressibility and the temperature coefficient of the liquid. The isothermal compressibility is defined as

$$\alpha = \frac{1}{\rho} \left(\frac{\partial \rho}{\partial P} \right)_T$$

and the temperature coefficient as

$$\beta = \frac{1}{\rho} \left(\frac{\partial \rho}{\partial T} \right)_P$$

Assuming that the temperature error is not more than ± 0.1 K and the pressure error is not more than ± 0.25 psi, then the resulting density errors for liquid nitrogen are:

Table 4

Errors Due to Pressure and Temperature Measurements

Liquid	α - %/psi	% density error	β - %/K	% density error
N ₂	0.0022	0.0005	0.6	0.06

The pressure measurement is made with a strain gauge type pressure transducer that is calibrated against a test gauge having a stated accuracy of 0.1 percent of full scale. Table 4 shows that the uncertainty in the pressure measurement contributes very little to the uncertainty in the density.

The temperature measurement requires more care. The temperature measurement system consists of a constant current source of 0.001 amp feeding a standard resistance, specified at ± 0.02 percent, and a platinum resistance thermometer. The platinum resistance thermometer has been calibrated against a NBS secondary standard thermometer at the hydrogen, nitrogen, and ice point. The interpolation between these temperatures is done by the three point method of Corruccini [1960]. It is estimated that the uncertainty of the calibration and the interpolation does not exceed ± 0.05 K, which results in a density uncertainty of ± 0.03 percent. The stability of the current source is monitored by reading the voltage drop across the standard resistor on the 0.1 volt scale of the voltmeter. The current is adjusted to within ± 0.05 percent which is the capability of the voltmeter. This translates into a possible density error of ± 0.01 percent.

The stability of the constant current source has been checked over a period of 15 days throughout each day. The uncertainty of the current reading, calculated on three times the standard deviation, is ± 0.0036 percent, which results in an uncertainty in the density of ± 0.002 percent.

The thermometer protrudes into the flow stream with liquid sweeping around the tubular support of the platinum sensor. Flow velocities are between 0.5 to 5 ft/s passing the thermometer. The velocity head of liquid passing the thermometer is about 0.3 ft. Thus, the pressure on the downstream side of the thermometer tube should not be depressed more than 0.25 psi below the liquid static pressure. Since the static pressure is 10 to 75 psi above the liquid vapor pressure, it is believed that liquid cavitation does not contribute to the thermometer imprecision.

From the above discussion of the instrumentation, it is seen that the major uncertainty in the density measurement is in the uncertainty of the state equation for nitrogen.

7. Systematic Errors

Four different types of positive displacement, volumetric cryogenic flowmeters have been evaluated. The deviation between the meter reading and the flow system measurement has been plotted for the parameters: liquid temperature, weight of liquid accumulated, gas space temperature, degree of liquid subcooling, pressure, mass flow rate, and the order in which the data were taken. Of all these parameters, only liquid temperature consistently showed a trend for all meters. The deviations were consistently higher at high liquid temperatures. This effect was traced to a systematic error made in calculating the buoyancy correction.

Previously an analysis of the gas in the catch and weigh tank had shown a predominance of helium. Thus, the buoyancy calculation was consequently made on the basis of helium. That gas analysis proved to be in error, and subsequent analysis of the gas showed that at higher

temperatures the higher vapor pressure of liquid nitrogen causes a predominance of nitrogen vapor in the gas space. Therefore, an equation of state for the helium-nitrogen gas mixture was written into the data reduction program to calculate the buoyancy of the accumulated liquid.

Once the buoyancy correction is made, there remains the question of how well the correction has been made. The ability to make this correction depends upon how well the average temperature of the displaced gas is measured and the uncertainty of the state equation for the helium-nitrogen mixture. Currently, there is a problem in obtaining a uniform temperature environment for the weigh tank. When liquid is dumped from the weigh tank, gas is displaced from the catch tank. This gas prefers to circulate up the outside of the weigh tank neck, across the warm flange, and down the inside of the weigh tank neck, rather than through the vents provided. This action causes a temperature gradient to exist across the gas space of the catch and weigh tank. The temperature of the displaced gas is taken as the average temperature across the gas space as measured by two platinum resistance thermometers. The uncertainty of the buoyancy force for the temperature extremes encountered can be quite large; therefore, tests are not conducted until temperatures are within specified limits. Large errors in this area would be indicated by a systematic deviation as a function of gas space temperature. Since there is no such deviation apparent in the data, it is believed that errors from this source are not large.

The binary mixture state equation is believed accurate to within 13 to 5 percent in the range of interest. Errors from this source would be a function of liquid temperature and would result in only a ± 0.07 percent error in the liquid weight at the higher temperatures. The effect

would be much less at lower temperatures. The absence of a systematic trend as a function of liquid temperature is evidence that errors from this source are not large.

Considering both of the above mentioned sources of error, the estimate of limits to the systematic error associated with the buoyancy calculations is ± 0.1 percent. This is the largest source of error in the mass measurement. Improvement will be made in this area when better temperature control is obtained.

The liquid diffuser is submerged in the weigh tank giving a buoyancy force that is equal to the product of the diffuser volume and the liquid density. Again a large density error will result in only a small weighing error. However, the density of the liquid and the volume of the diffuser are reasonably well known. It is estimated that the resulting uncertainty in weighing from this source does not exceed ± 0.03 percent.

Table 5 summarizes the available evidence on estimated limits to systematic error from all known or suspected sources. The source and limits to the magnitude of the error are listed according to the type and amount of information available on that error source. The estimated limit to total systematic error has been computed by adding the components in quadrature, i. e. ,

$$\sqrt{(0.01)^2 + (0.05)^2 + \text{-----} + (0.01)^2} = \pm 0.12\%.$$

This procedure may be justified whenever there is no "correlation" among the several sources of error. For example, the error due to the voltmeter is positive, this does not influence the chances that any of the other errors will be positive or negative.

Table 5
Systematic Errors

	Source of Error	Error Estimated From	Error %	Comment
Mass Measurement	Weights	Calibration	± 0.01	See appendix A
	Non-linearity of Voltmeter-Load Cell	Manf. & Measurement	± 0.05	See Appendix B and Text
	Calibration of Voltmeter-Load Cell	Measurement	± 0.02	Table 3
	Temperature on Load Cell	Measurement	± 0.00	Operates at design temperature
	Buoyancy of Liquid	Calculated	± 0.10	Can be improved by reducing the temperature gradient in the weigh tank
	Buoyancy of Diffuser	Calculated	± 0.03	Caused by the uncertainty in the liquid density
	Reaction Forces	Calculated	± 0.01	Estimated from dynamic equations
	Impact Forces	Calculated		Designed to be eliminated
		Total	± 0.12	By quadrature
	Density Measurement	Thermometer Calibration and Interpolation	Calibration	± 0.03
Thermometer Current Source		Calibration	± 0.01	
Pressure		Calculated	± 0.00	Not significant
State Equation of Liquid Nitrogen		Strobridge [1962]	± 0.39	Error primarily in experimental measurements
		Total	± 0.39	By quadrature

8. Interlaboratory Comparison

It is quite informative to have the same meter checked by another flow facility operating on a different principle. Such comparisons help to locate unsuspected sources of systematic error and to lend confidence to the validity of an accuracy statement. Extensive data have been taken on two positive displacement type meter elements at NBS and at a static gravimetric flow facility operated by a meter supplier. Due to the possibility of leaks occurring in these particular meters, in the meter bowls, or in the meter element-bowl joints which bypass the measuring chamber, it is necessary to calibrate such devices as units - element and bowl combined. Unfortunately, this was not discovered until the calibration data had been taken, with elements calibrated in different bowls by NBS and the meter supplier. The result is an uncertainty of ± 0.5 percent in the differences between NBS and supplier calibrations which were negligible by comparison (less than 0.1 percent). Despite this loss of resolution, the results indicate that there is no major (> 0.5 percent) discrepancy between the two measurement facilities.

Present plans include the use of fixed meter element/meter bowl configurations to establish future interlaboratory comparison.

9. Tests of Meters

When the facility is used to test meters the resulting uncertainty necessarily incorporate a component of variation arising from the performance of the meter itself. Four different types of meters have been tested, some with substantial imprecision. The tests of these meters were evaluated through a pressure range of 50 to 100 psig, a temperature range of 80 to 90 K, and a flow range of 20 to 50 gpm. The resulting standard deviation was based on 36 points taken on the meter with

best performance. The uncertainty due to combined dynamic imprecision of the volumetric meter, the density measurement required to compare the meter to the mass system and the cryogenic flow facility, calculated on three times the standard deviation, is ± 0.3 percent.

10. Provisional Accuracy Statement Summary

As may be garnered from the above discussions, it is a tedious and difficult process to demonstrate the validity of an accuracy statement. It seems desirable at this time to gather a moderate amount of additional evidence prior to issuing a formal accuracy statement. The following statements summarize the accuracy information presently available on the NBS gravimetric flow system.

At this time the uncertainty of the measurement of totalized mass flow is estimated to be ± 0.18 percent. This figure includes an uncertainty of ± 0.12 percent for known sources of systematic errors plus an uncertainty of ± 0.06 percent for random error. The estimated uncertainty due to the random error is three times the standard deviation calculated for 23 applications of the calibrated masses over a period of three months.

At this time the uncertainty of the measurement of totalized volumetric flow of liquid nitrogen is estimated to be ± 0.47 percent. This figure includes an additional uncertainty of ± 0.39 percent for known sources of systematic error in the density determination. The major contributor to the uncertainty of liquid nitrogen volumetric flow measurement is the uncertainty in the liquid density.

When the objective of using the accuracy statement is in the mediation between seller and buyer, where both have accepted the same values of density, the uncertainty in the density need not be considered.

Under this condition the uncertainty of the measurement of totalized volumetric flow is ± 0.18 percent, because the contribution of the pressure and temperature measurements error is negligible.

This accuracy statement should be considered provisional and is currently restricted to flow rates between 20 and 100 gpm, pressures from 50 to 100 psig, and temperatures from 80 to 90 K, for ideal conditions where operational or equipment malfunctions are not present. The error analysis is continuing and will be updated as more information is acquired.

L. A. K
Xenon,
Tetraflu
Thermo

11. References

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5. T. R. Strobridge, "The Thermodynamic Properties of Nitrogen from 64 to 300 K between 0.1 and 200 Atmospheres," *NBS Tech. Note* 129 (1962).
6. M. J. Terry, J. T. Lynch, M. Bunclark, K. R. Mansell, and L. A. K. Staveley, "The Densities of Liquid Argon, Krypton, Xenon, Oxygen, Nitrogen, Carbon Monoxide, Methane, and Carbon Tetrafluoride Along the Orthobaric Liquid Curve," *J. Chem. Thermodynamics*, 1 (1969), p. 413.

APPENDIX A

Standard Masses Report of Calibration

For Section 212.31

Item Set of mass standards 50LB-6, 50LB-26, and 25LB-26
Stated density 50LB-6 8.3909 G per cm³ at 20C
50LB-26 8.3909 G per cm³ at 20C
25LB-26 8.3909 G per cm³ at 20C

The above items have the mass values shown with reference to the national standard of mass. See attached supplement for limitations in the use of apparent mass value and uncertainty from error in density.

Item	Nominal	App. Mass Corr. Microlb.	True Mass Corr. Microlb.	Uncertainty Microlb.	Vol. at 20 (cm) ³
50LB-6	50.0	116.7	116.8	44.0	2702.8
50LB-26	50.0	- 8.4	- 8.4	44.0	2702.8
25LB-26	25.0	6.9	6.9	40.0	1351.4

The uncertainty figure is an expression of the overall uncertainty using three standard deviations as a limit to the effect of random errors of measurement, the magnitude of systematic errors from known sources being negligible.

For the Director,

Signed by
Paul E. Pontius, Chief
Mass and Volume Section
Metrology Division

Test completed June 4, 1965
Washington, D. C. 20234

UNITED STATES DEPARTMENT OF COMMERCE
 NATIONAL BUREAU OF STANDARDS
 Washington 25, D. C.

Comp No. 3009
 Set A

NATIONAL BUREAU OF STANDARDS

TEST REPORT

on

Set of Avoirdupois Weights

Maker: W & L. E. Gurley, Inventory No. NBS 96850
 Submitted by: National Bureau of Standards
 Division 2, Section 6

Material and Assumed Density: 10 lb to 0.002 lb, stainless steel,
 7.92 g/cm³ at 20°C; 0.001 lb to 0.0001 lb, 80% Ni - 20% Cr, 8.4 g/cm³
 at 20°C.

This set of weights is accepted for calibration and conforms to Class S-1 specifications for design and construction. It does not conform to Class S-1 requirements for accuracy of adjustment in that the weights designated (0.2 lb), (0.05 lb), (0.02 lb), and (0.005 lb) have corrections larger than the tolerances for those denominations. All weights of this set are adjusted within Class A tolerances.

This certifies that the values are correct within the accuracy specified for the Class S-1 calibration.

The weights from 10 lb to 0.05 lb, inclusive, are marked with the letters SA.

<u>Designation</u>	<u>Apparent Mass vs. Brass</u>			
(10 lb).	10	lb	+12	mg
(10 ").	10	"	+ 0	"
(5 ")	5	"	+22	"
(2 ").	2	"	- 2.0	"
(2 ").	2	"	+ 4.3	"
(1 ")	1	"	+ 2.2	"
(0.5 ")	0.5	"	+ 2.2	"
(0.2 ").	0.2	"	- 1.16	"
(0.2 ").	0.2	"	+ 0.16	"
(0.1 ")	0.1	"	- 0.43	"

Comp. No. 3009, Set A, (continued)

<u>Designation</u>	<u>Apparent Mass vs. Brass</u>	
(0.05 lb)	0.05 lb	+ 0.50 mg
(0.02 ")	0.02 "	- 0.13 "
(0.02 ")..	0.02 "	- 0.64 "
(0.01 ")	0.01 "	+ 0.05 "
(0.005 ")	0.005 "	+ 0.141 "
(0.002 ")	0.002 "	- 0.048 "
(0.002 ")..	0.002 "	- 0.065 "
(0.001 ")	0.001 "	+ 0.014 "
(0.0005 ")	0.0005"	+ 0.023 "
(0.0002 ")	0.0002"	+ 0.012 "
(0.0002 ")..	0.0002"	- 0.037 "
(0.0001 ")	0.0001"	+ 0.014 "

L. B. Macurdy
Chief, Mass Unit
Mass and Scale Section
Metrology Division

Test completed:
July 12, 1961

CLC:bvh

Supplement for Precise Laboratory Standards of Mass

1. BASIS FOR VALUES. Values under the heading *Apparent Mass versus Brass* are those which the weights appear to have when compared in air under *Normal Conditions* against *Normal Brass* standards, no correction being made for the buoyant effect of the air. *Normal Conditions* are 20° C and air density of 1.2 mg/cm³. *Normal Brass* has a density of 8.4 g/cm³ at 0° C and coefficient of cubical expansion of 0.000 054/deg C. True Mass values are those which might be observed in air of zero density, i. e., in a vacuum.

2. ACCURACY. The accuracy is the extent to which the measured values, as given, are in agreement with the true values of the mass of the weights. The divergence of the measured value from the true value is the inaccuracy.

The methods of weight calibration employed at the National Bureau of Standards include checks against numerical errors, observational blunders and other sources of uncertainty that would detract from the accuracy. The methods involve more than the minimum number of observations necessary to obtain the values, and derive the values from more than one standard, with some mathematical treatment to obtain the best value. A statistical estimate of the inaccuracy includes allowance for uncertainty or bias in the standards and or the precision of the weighings. The estimated bias of the standards is small relative to the precision and is included in the limits stated below for the inaccuracy of values under the various classes. These limits are at least three times the standard deviations of the calibrated values. In rounding off the computed values those digits that are uncertain by 15 units or more are dropped by rounding off to one less decimal place.

Class J: For microbalance weights adjusted to nominal values in a series which permits appropriate intercomparisons of the weights the inaccuracy is within 0.2 microgram. For weights of odd amounts the inaccuracy is within 0.3 microgram.

Class M: 1 part in 10⁶ for weights 10 g and above; 0.01 mg for weights 10 g to 1 g, inclusive; 0.001 mg for weights below 1 g.

Class S and S-1: 3 parts in 10⁶ for weights 10 g and above; 0.03 mg for weights 10 g to 1 g, inclusive; 0.003 mg for weights below 1 g. For 1 g weights of such materials as platinum, tantalum or gold the inaccuracy is within 0.01 mg.

3. VOLUMES. Under Class M the volumes are determined for weights of 1 g and larger as the basis for computation of True Mass values. In other cases True Mass values are computed by the use of assumed densities. When the density is assumed to be that of normal brass, only the Apparent Mass values are given.

4. COMPUTATION OF TRUE MASS OF OBJECTS. If it is desired to compute the true mass of an object it is necessary to make proper allowance for any systematic effects. Since air exerts an appreciable buoyant effect it is necessary to make an appropriate correction to the results of a weighing in air. When the volumes of the object and the weights are known it is convenient to use formula I.

$$I. \quad M_n = M_s + \rho(V_n - V_s),$$

where M_n = true mass of unknown object, M_s = true mass of standard weights necessary to bring the balance into equilibrium, ρ = air density, V_n = volume of unknown mass, and V_s = volume of standards. When apparent mass values are used for M_s , the volume, V_s , is the volume of normal brass.

When the volume of the unknown mass has not been measured the true mass may be computed from the densities by the approximate formula II.

$$II. \quad M_n = M_s \left[1 + \rho \left(\frac{1}{D_n} - \frac{1}{D_s} \right) \right]$$

where D_n and D_s are densities.

For values of $D_n = 2$ g/cm³ or greater and for $D_s = 8.4$ g/cm³, formula II gives values correct within 1 part in one million. For values of D_n less than 2 g/cm³ or for weighing to higher precision than 1 in 10⁶ the following exact formulae may be required.

$$III. \quad M_n = M_s \left(\frac{1 - \frac{\rho}{D_s}}{1 - \frac{\rho}{D_n}} \right)$$

The precision needed for ρ is determined by the size of the difference between V_n and V_s . For quartz versus brass $V_n - V_s$ is +0.258 cm³ per gram. With a precision of weighing of 4 in 10⁷, and with ρ known to 0.2 percent the weighing will be correct to 1 part in 10⁶. For a precise computation of ρ it is necessary to determine the temperature, pressure, and relative humidity of the air and to apply the following corrections: (1) Instrument correction for barometer, (2) barometric reduction from local to standard gravity, (3) reduction of barometric pressure to the pressure for dry air of equivalent density, and (4) thermometer correction to international temperature scale. Instructions and tables for computing the various terms required for the density of the air are given in the Smithsonian Meteorological Tables.

When "Apparent Mass versus Brass" is used for M_s , the standards must then be assumed to have the same density as normal brass. This assumption introduces no error when weighings are made under normal conditions. At altitudes to 6,000 ft. apparent mass values and the assumed volumes of the weights may still be used with actual air densities since the discrepancies arising from the assumption regarding volume do not exceed the accuracy of the Class S calibration.

For higher accuracy than the accuracy of the Class S calibration or under more extreme conditions it may be desirable to compute the true mass values for the weights in order to provide a more precise determination of the buoyant effect of the air. When true mass values are used for M_s , then the actual density of the standards must be used for D_s .

True mass values of the weights may be computed by applying the following corrections to the "Apparent Mass" values:

Material	Density	Correction
	<i>g/cm³</i>	<i>mg/g</i>
Aluminum.....	2.7	+ 0.302
Stainless steel.....	7.8	+ .011
Stainless steel.....	8.0	+ .007
Brass.....	8.4	.000
Tantalum.....	16.6	-.071
Gold.....	18.0	-.076
Platinum.....	21.5	-.087

Corrections are to be added to apparent mass to get true mass.

APPENDIX B

Load Cell Manufacturers Certificate

Certificate of Calibration

Date 3-22-68

Transducer Model No. 36-233-BGA Range 1000 lb Serial No. 92693A

Customer NBS, Colorado

Customer P.O. No. 71615-8 Sales No. 1198

Excitation Voltage 10 volts ac/dc

Input Resistance (Ri) 398.6 ohms

Output Resistance (Ro) 351.9 ohms

Sensitivity

Compression 3.002 mv/v
Tension 3.007 mv/v

THESE FACTORS ARE WITHIN ± 0.10 % OF NOMINAL 3 MV/V F.S. OUTPUT IN THE DIRECTION OF STANDARDIZATION.

Linearity (L) within 0.05 %F.S.

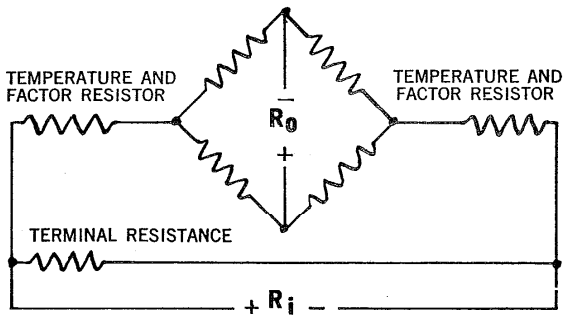
Hysteresis (H) within 0.03 %F.S.

Repeatability (R) within 0.02 %F.S.

Combined L,H,R, within 0.07 %F.S.

Double Shunt Calibration with 58,158 ohms 100.31 %F.S. in compression
100.13 %F.S. in tension

Wiring Diagram



STANDARDIZED IN COMPRESSION

Terminal Connections

- A-F Neg. Output
- E-J Pos. Output
- B-K Neg. Input
- D-H Pos. Input
- Shield

Connector PC1H-12-10P
Bendix

THIS TRANSDUCER WAS MANUFACTURED TO RIGID QUALITY CONTROLLED STANDARDS AND CALIBRATED WITH DEAD WEIGHTS, A DEAD WEIGHT AND LEVER SYSTEM LOAD APPLICATOR OR LABORATORY STANDARD TRANSDUCER CALIBRATED BY THE NATIONAL BUREAU OF STANDARDS OR TRACEABLE THERETO.

[Signature]
QUALITY CONTROL