

# Comparisons of Viscous-sealed Provers with LNE and Studies of Piston – Cylinder Leakage

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## Abstract:

Dry piston provers (DryCals) are used for high-precision primary calibration of transfer standards and MFCs. In the course of our NVLAP ISO 17025 accreditation, a large amount of validation had been done at flows above 1000 sccm. However, we desired additional validation at low flows.

We submitted a set of 0.15% production DryCals to LNE (Laboratoire National de Métrologie et d'Essais), in Paris, for comparison with their dynamic gravimetric primary standard, using LFEs as short-term transfer devices in the course of a calibration. Their tests used flows from 5 to 30,000 sccm, and were repeated after five months. Results were well within our combined uncertainty claims and showed insignificant drift. However, these were routine calibrations, and the dynamic gravimetric method does not possess low enough uncertainty to adequately validate 0.15% when used with an intervening LFE.

This is especially true at the lowest flows, where piston leakage tare is significant. We have performed static gravimetric tests of our master low-flow DryCal at 175 sccm with a test uncertainty of 0.025% and 0.005% deviation, but we are not certain that such tests are feasible at 5 sccm.

As the most significant departure from linearity at the lowest flows was leakage tare, we conducted additional studies at Bios. The studies showed that leakage was constant with flow for all species of gas tested, remained constant for 14 months and had low enough uncertainty to validate our accuracy at the lowest flows.

## 1. Introduction

The DryCal ML-800 (shown in Figure 1) is a piston prover for measuring the flow of gases in the 5 sccm to 50,000 sccm range. It differs from earlier provers in that the piston is sealed by the viscosity of the gas under test, eliminating other sealing methods such as the use of mercury rings. This allows the instrument to be small, automatic and portable, with accuracy suitable for standards laboratories. Three interchangeable flow cells are available: 5 to 500 sccm (ML-800-10), 50 to 5,000 sccm (ML-800-24) and 500 to 50,000 sccm (ML-800-44) For brevity, we will use the nomenclature "10", "24" and "44" for the three cell sizes; sccm to represent a flow of one cubic centimeter per minute at conditions of 0 °C and 101.325 kPa; and slm to represent 1000 sccm.

We have published extensive descriptions of the DryCal's operating principles and construction, as well as detailed uncertainty analyses. We have also published extensive inter-laboratory comparisons in order to validate the instrument and our analyses. However, validation is an ongoing process, and we have continued to evaluate the DryCal in new flow regimes, with new comparison methodologies and with new gas species.



Figure 1 – DryCal ML-800  
(attached to a mass flow controller)

## 2. Review of operating principles

A functional diagram of a DryCal piston prover is shown in Figure 2. Inlet gas ordinarily flows through the bypass valve to the outlet. When a reading is to begin, the bypass valve closes and the incoming gas displaces a piston that moves through a cylinder. After the piston has been allowed adequate time to accelerate, the time needed to pass from one optical sensor to another is measured. To the degree that the volume of the device is well known, we can derive the flow from measured primary dimensions (length and time). Temperature and absolute pressure sensors are added to obtain standardized readings.

The unique attribute of the DryCal design is its use of the viscosity of the gas under test to form a seal. Using piston-cylinder clearances of approximately 9 microns, leakage is minimized to the point of insignificance at all but the lowest flows in the instrument's range.

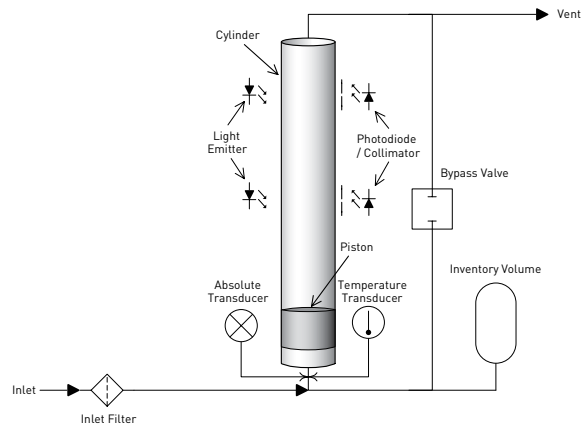


Figure 1: Met Lab Series Piston Prover

Figure 2 – Piston Power

## 3. Prior Inter-Laboratory Comparisons

We have, in the past, reported extensively on comparisons of the DryCal with other laboratories, particularly with NIST (National Institute of Standards and Technology, U.S.A.) and NMIJ (National Metrological Institute of Japan). We had shown that the DryCal was transportable and possessed low drift by comparing the same cell at NMIJ in Japan and at NIST in the USA five months later (Figure 3) with better than 0.1% correlation.

We had shown that additional DryCals could be manufactured and dimensionally calibrated by comparing an old and a newly-manufactured cell at NIST (Figure 4). We had also conducted a multi-laboratory comparison using two different sizes of flow cells, with the Youden plot showing about  $\pm 0.05\%$  consistency between the cells (Figure 5).

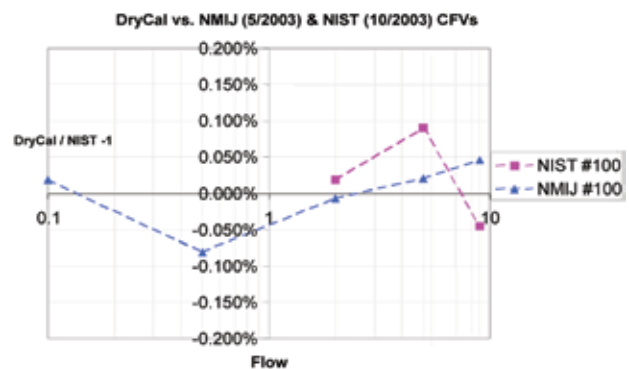


Figure 3 – DryCal vs. NMIJ and NIST CFVs

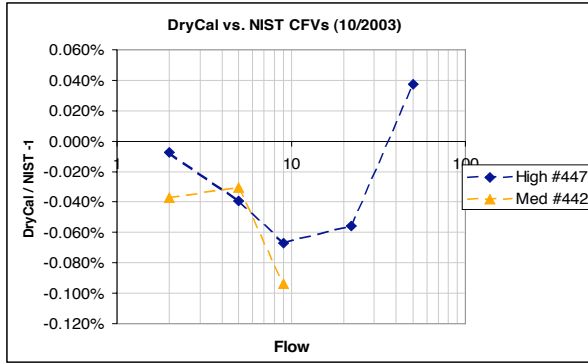


Figure 4 – New and old DryCals vs. NIST CFVs

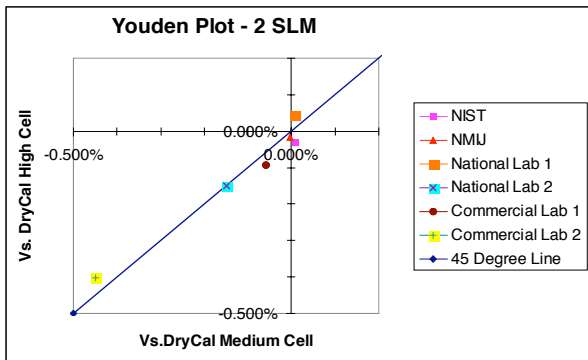


Figure 5 – Multi-laboratory comparison using 24 and 44 sized cells

## 4. LNE Comparison

Our previous inter-laboratory comparisons offered a very high degree of confidence in our prover design and calibration. Its accuracy was confirmed in a substantial number of comparisons. However, our accuracy at flows lower than 1 slm had been validated only at one laboratory (NMIJ) and below 100 sccm by inference alone. Also, the evaluations had only been conducted with the two larger-sized (24 and 44) cells. Therefore, we wished to validate our provers over their full flow range of 5 sccm to 50 slm using all three sizes of cells. LNE graciously offered to perform such an evaluation.

### 4.1. Method

LNE uses a laminar flow element as a short-term transfer standard calibrated by a dynamic gravimetric system (Appendix I). All three sizes of ML-800 DryCal cells were left with LNE for five months. They were evaluated over their full rated ranges against LNE's standards initially, and again after approximately five

months. Thus, they were tested against LNE for both accuracy and long-term stability. It is noteworthy that this was the first time we were able to compare DryCal to a national laboratory at flows below 100 sccm.

### 4.2. Results

The complete data and calibration certificates issued by LNE are available on our website. Here, we will present the results in graphical form. We plotted the results of our comparison against flow as two plots, one for 12 October 2004 and one for 25 March 2005. We also plotted the uncertainty claimed by LNE on their calibration certificates. The results for our ML-800-10, -24 and -44 cells are presented in Figures 6, 7 and 8, respectively.

All of our comparison results were well within LNE's stated uncertainties, which seem to be very conservative. The long term repeatability drift over a five month period was near zero for the two higher-flow comparisons and about  $\pm 0.1\%$  for the lowest flow comparison.

### 4.3 Interpretation of Results

Our worst-case disagreement was about  $\pm 0.2\%$  for the two larger (24 and 44) cells. However, prior comparisons at NIST and NMIJ left us confident that the DryCals were well within their specifications. The lowest-flow (10) cell disagreed with LNE by  $\pm 0.1\%$  and  $\pm 0.25\%$  at its lowest flow of 5 sccm.

However, these were routine calibrations, and the dynamic gravimetric method does not possess low enough uncertainty to adequately validate 0.15% when used with an intervening LFE.

We were confident of our mid-span uncertainty, having performed static gravimetric tests of our master low-flow DryCal at 175 sccm with a test uncertainty of 0.025% and 0.005% deviation. However, we were not certain that such tests were feasible at 5 sccm, and we wished to validate our uncertainty at that flow rate.

As the most significant departure from linearity at the lowest flows would result from errors in leakage tare (which the DryCal's microcontroller adds into the reading), we conducted additional studies at Bios.

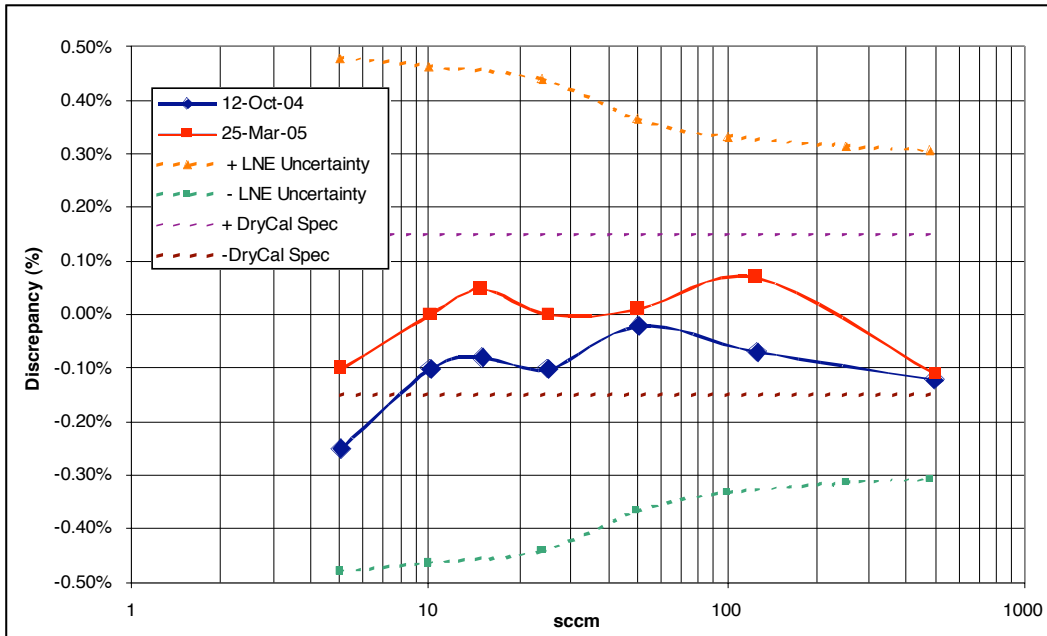


Figure 6 – LNE results for size 10 cell

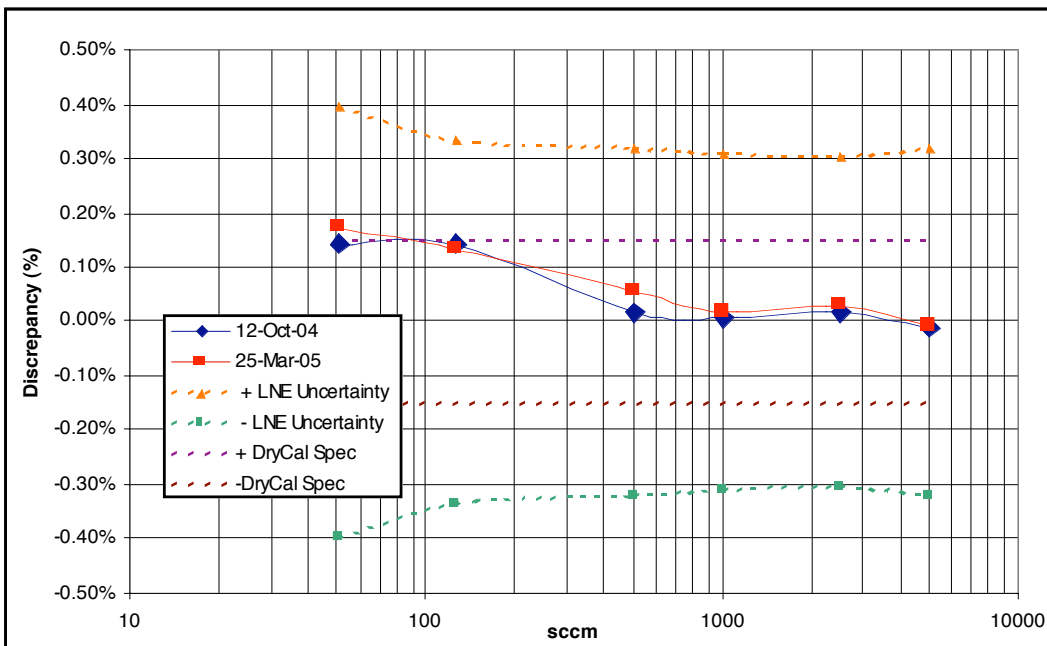


Figure 7 – LNE results for size 24 cell

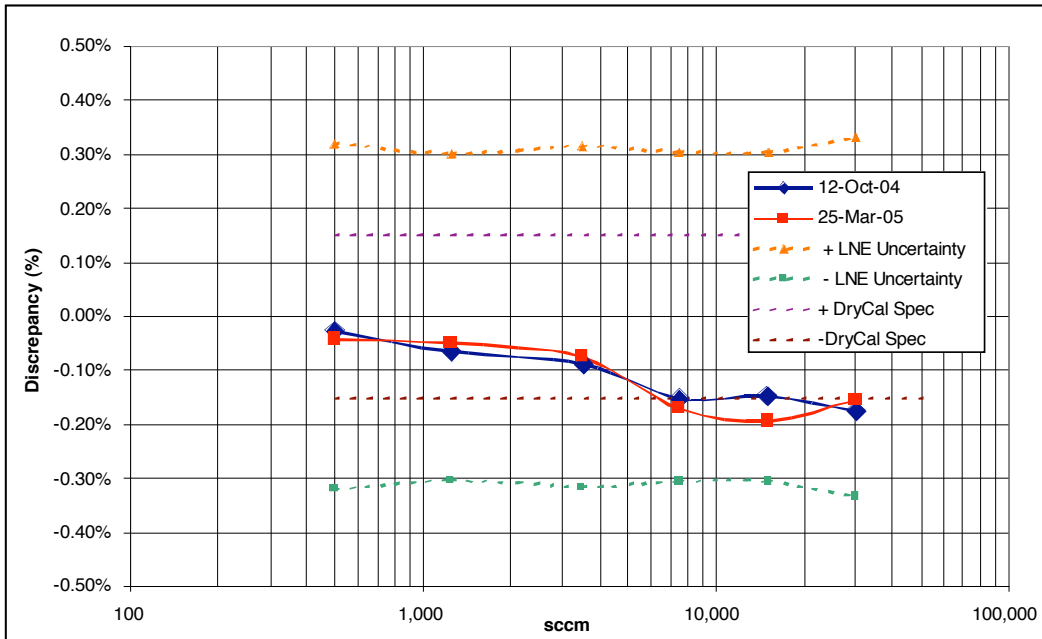


Figure 8 - LNE results for size 44 cell

## 5.0. Leakage Tare Studies

Because the piston seal is formed by the viscosity of the gas under test, there is necessarily some leakage past the piston skirt. The amount is small, approximately 0.1 sccm for our smallest (10) cell, but we must adjust for the leakage to achieve the lowest flow measurements at a given uncertainty level. We do this by adding the tare value of leakage to our observed measurements. Our low-flow uncertainty depends upon the uncertainty of our leakage tare measurements. The quality of these measurements thus becomes the limiting factor in how low a flow we can measure at a given uncertainty.

While averaging a number of leak-test readings will reduce the uncertainty of the tare value, this cannot remedy faulty test methodology. If, for example, the leakage is not constant with flow, there will be a systemic error if leakage is not measured dynamically. For this reason, we wished to measure leakage at our lowest specified flow, currently 5 sccm.

The most straightforward approach to dynamic leak testing would be to supply a well-known flow of the desired value (5 sccm) and to observe the difference in readings between the DryCal under calibration and the flow standard. Unfortunately, this would require a primary standard many times more accurate than the

DryCal, which, in its most advanced form, is accredited at 0.076% combined expanded uncertainty. Standards of such (0.02%) uncertainty simply do not exist in a practical enough form for our production purposes.

Thus, we needed a non-comparative, stand-alone, approach to leak measurement. In seeking this, our leak test methodology progressed from a near-static technique, to a summed-flow technique and finally to a successive inversion technique. At the same time, in order to facilitate more reproducible leakage tests, we built a new flow laboratory with  $\pm 0.25^\circ\text{C}$  stability.

### 5.1. Near-static

Our earliest method was a near-static test. The inlet (or outlet) of the cell was blocked, the bypass valve closed and the cell inverted. Gravity would cause the piston to descend at its leak rate. By timing the descent (which took hours), we could calculate the flow.

The problem with such a test, other than the time it takes, is that it indicates leakage at such a low flow rate (~0.1 sccm) that it may not be representative of leakage at the flows actually used in the instrument (>5 sccm). Therefore, this method was never considered for practical calibration of the DryCal.

## 5.2. Dynamic summation

Our first approach to dynamic leakage testing was a dynamic summation method. Gas was introduced to two flow restrictors and allowed to stabilize fully. Flow was measured for each of the two flow streams and again for both flow streams simultaneously. Since the leakage would subtract from each of the measurements, we could obtain leakage by subtracting the sum of the two individual flows from the single reading of the combined flow:

$$\dot{Q}_l = (\dot{Q}_1 + \dot{Q}_2 - \dot{Q}_l) - (\dot{Q}_1 - \dot{Q}_l) - (\dot{Q}_2 - \dot{Q}_l)$$

This technique is illustrated in Figure 5 and typical results are shown in Table 1.

Table 1 – Dynamic summation typical results

Flow A	Flow AB	Flow B	Leakage
4.090	8.371	4.139	0.142
4.098	8.395	4.136	0.161
4.095	8.378	4.133	0.150
4.101	8.385	4.138	0.146
4.104	8.400	4.139	0.157
4.107	8.397	4.145	0.145
4.102	8.395	4.144	0.149
4.104	8.392	4.136	0.152
4.101	8.387	4.141	0.145
4.101	8.380	4.138	0.141
Average Leakage			0.149
ST Dev (u) max.			0.00625
U (2 X)			0.013

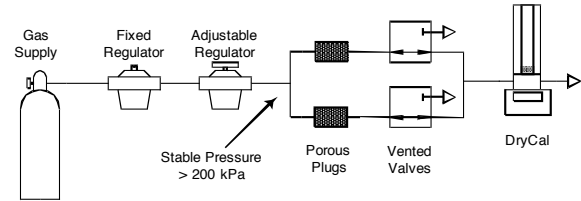


Figure 9 – Dynamic summation method

Tests on a number of cells confirmed that we were progressing in the right direction. We noted that dynamic leakage was higher than static leakage (with either the inlet or outlet port blocked) in every case, significantly for a low-flow (10) cell as shown in Figure 10 and more dramatically with a high-flow (44) cell as shown in Figure 11.

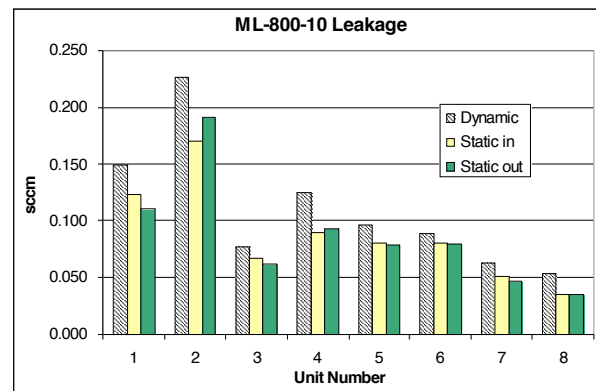


Figure 10 – Near-static vs. dynamic leakage measurement, size 10

The problem with this method is that it depends upon the constancy of leakage with flow for its results. We “laddered” such tests, progressing in flow for each cell to evaluate constancy. Although the results appeared constant with flow, we wished to use a method that would use more nearly equal flows than with the summation method.

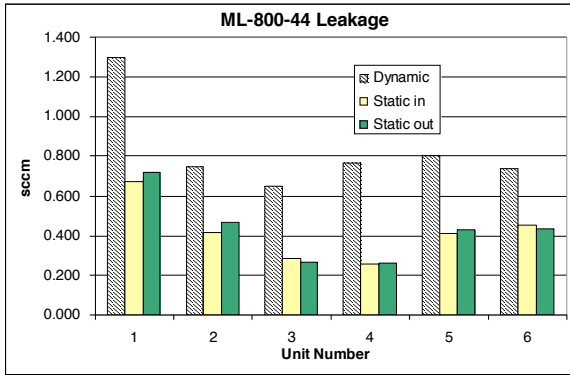


Figure 11 - Near-static vs. dynamic leakage measurement, size 44

### 5.3. Dynamic inversion

To measure dynamic leakage at a nearly constant flow rate, we devised a dynamic inversion test. The cell under test is attached to its base with a flexible cable. Using a stable flow, readings are taken with the cell in alternating upright and inverted positions (Figure 12). Since leakage subtracts from the upright readings and adds to the inverted ones, leakage is half the magnitude of the difference between successive readings. Typical results are shown in Table 2.

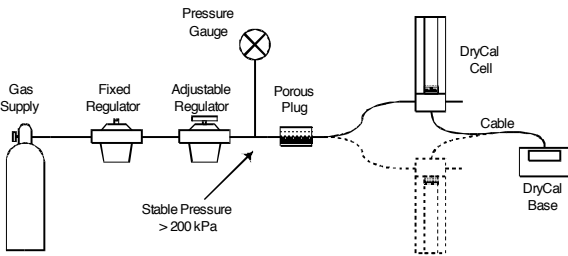


Figure 12- Dynamic inversion method

### 5.4. Linearity of leakage with flow

The dynamic inversion method yielded very consistent leakage readings that were almost constant with flow, except, as noted, for near-zero flows. This is illustrated for the three gas species nitrogen (Figure 13), hydrogen (Figure 14) and xenon (Figure 15). Each chart shows the results of its specific gas on five different measuring cells at a number of dynamic flow points. Not only is the average leakage constant, but the leakage of each individual cell is constant with flow. This validates our using a single tare value and adding it to our readings at any flow.

Table 2 – Dynamic leakage successive-inversion method

Position	Flow (sccm)	Leakage (sccm)
Upright	5.0330	
Inverted	5.2780	0.123
Upright	5.0370	0.121
Inverted	5.2820	0.123
Upright	5.0330	0.125
Inverted	5.2780	0.123
Upright	5.0330	0.123
Inverted	5.2800	0.124
Upright	5.0360	0.122
Inverted	5.2810	0.123
Upright	5.0350	0.123
Average Leakage		0.123
Standard Deviation (u)		0.00102

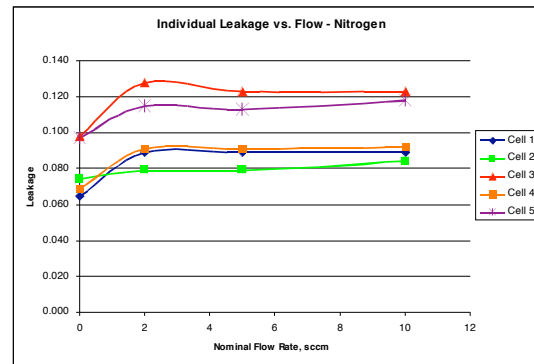


Figure 13 – Leakage vs. flow for nitrogen

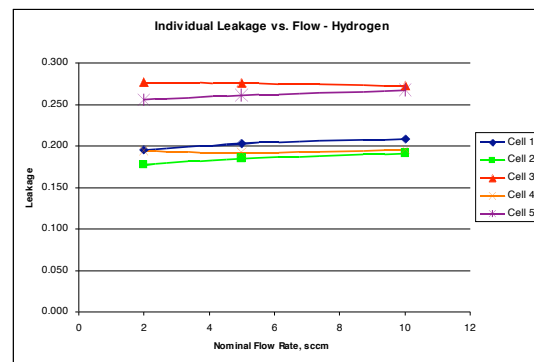


Figure 14 – Leakage vs. flow for hydrogen



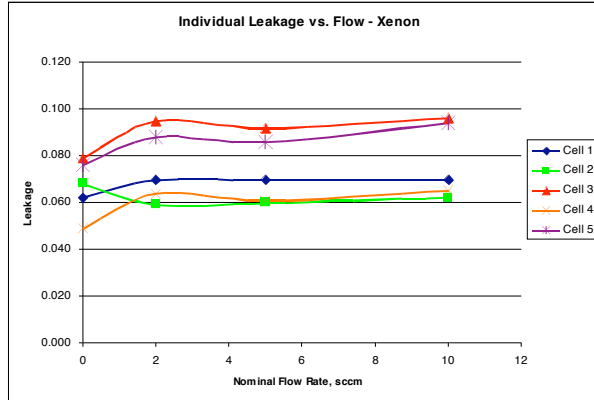


Figure 15 – Leakage vs. flow for xenon

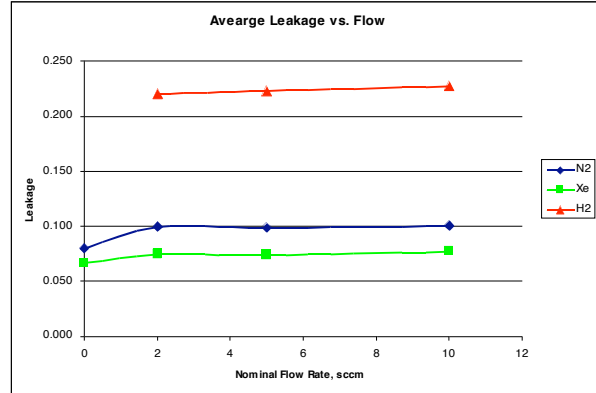


Figure 16 – Average leakage vs. flow by species

### 5.5. Consistency of leakage

From Table 2, we can see that the leakage measurements are quite consistent. Their standard deviation is two orders of magnitude smaller than the leakage 0.00102 sccm for a leakage of 0.123 sccm. The uncertainty of the tare value is further reduced by averaging a number of readings, with the uncertainty of the tare value of leakage reduced by the square root of the number of readings averaged. Unfortunately, this benefit only accrues to the tare value and it is irreducible by further averaging of working flow readings. When the cell is used for actual measurements, the uncertainty of the leakage appears a second time, now in each reading. This (and only this) additional leakage uncertainty can be reduced by averaging working readings.

Leakage tare proved remarkably consistent with time and with travel. After 14 months, the units tested at LNE were again calibrated for leakage. Even though the summation method was used in October 2004 and the inversion method was used in December 2005, the leakage tare values differed by only -0.0028 sccm, which represents 0.056% of our 5 sccm lowest rated flow.

### 5.6. Relative leakage by species

As we had seen earlier, the leakage of each individual cell was quite constant with flow. However, as is to be expected, it is quite different for each species. The average values for five cells, each using nitrogen, hydrogen and xenon illustrate this in Figure 16. Thus we need to correct the leakage tare value used in the DryCal for each gas species. Provision was made in the DryCal design for a leakage correction factor (LCF) to be entered. The LCF is simply the ratio of the leakage of the gas under test to that of nitrogen.

## 6. Conclusions

The tests at LNE supported our uncertainty analysis for all of our three sizes of cells.

The mid-span accuracy of our low-flow cell was further established by our internal 0.025% static gravimetric test at 175 sccm. Meanwhile, our leakage studies established that our leakage tare measurements methods and the tare's long term reproducibility were adequate to support our  $\pm 0.015\%$  uncertainty claims at our lowest rated flow levels. In effect, gravimetric tests validated the cell's slope and leakage tare tests its intercept.

In the future, it would be of interest to perform a direct comparison of the dynamic gravimetric method with the Drycal, eliminating the intervening uncertainty of a transfer LFE.

### Acknowledgement

The author would like to thank Mr. Jean Barbe of LNE for his excellent work in evaluating the ML-800 system.

## APPENDIX I

### LNE Primary Low Gas Mass Flow Standards

[Courtesy of LNE]

#### Components

The dynamic primary gravimetric standards at LNE have already been described in [1] and [2]. It mainly consists of three balances with different ranges and a synchronized real time measurement of the time differences. In table 1 the range specifications of the three systems are summarized.

Table 1. Ranges of the three gravimetric systems

System #	Capacity of cylinder (l)	Balance range (kg)	Balance resolution (mg)	Mass flow range (mg/s)	Volume flow range (ml/min)
1 small	0,4	1,2	0,1	0,02 ... 4	1 ... 200
2 medium	4	8	10	4 ... 200	200 ... 10000
3 large	11	16	100	200 ... 2000	10000 ... 100000

The Sartorius electronic balances are calibrated against masses traceable to French mass standard.

The time measurement is done by the HP 53131A counter calibrated with a SR620 counter traceable to French time standard.

### Experimental Setup

Figure 1 provides a schematic diagram of a mass flow calibration system used for calibrating a molbloc laminar flowmeter.

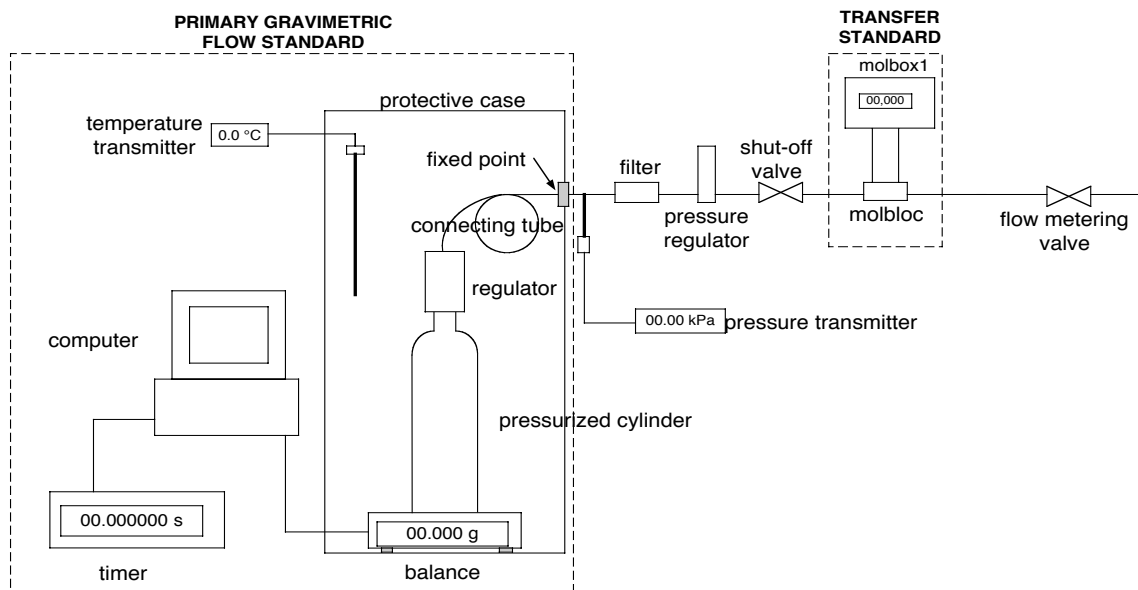


Figure 1. Schematic of experimental setup

The gas cylinder pressurised to an initial pressure of 15 MPa (150 bar) continuously releases the gas under 600 kPa (6 bar) set by adjusting a regulator. The cylinder is placed on the pan of an electronic balance placed on a concrete block and enclosed into a protective case to isolate it from the room.

Gas flows through the flexible connecting tube before leaving the case. A pressure sensor is placed on the level of the fixed point in order to verify the pressure stability in the tube and thus avoid mass variation and unstable 'Bourdon' effects that could disturb the weighing of gas mass lost by the cylinder.

Then, gas flows to the transfer standard. The pressure regulator connected upstream of the transfer standard allows a constant relative pressure between 100 kPa and 500 kPa to be regulated. The transfer standard is also used to display the instantaneous flow, the pressure and the temperature of the gas in the circuit. The proper operation of the reference standards can be checked for possible leakage in the gas circuit and stability of the generated reference flow.

The needle valve connected downstream of the flowmeter allows to adjust the flow at various measuring points.

When the generated reference flow is stabilized, a measurement is initiated. The actual mass automatically sent by the balance is read by a PC that starts the timer. When the measurement of the gas mass loss is completed, the PC interrupts the timer and the corresponding time interval is determined. Then, the mean mass flow rate is calculated.

## Uncertainty Evaluation

The uncertainty associated with the mean mass flow determination takes into account the uncertainties associated with the mass loss (difference of two readings of the balance) and with the time interval corresponding to that difference. Table 2 gives the uncertainty analysis for flowrates between 8 mg/s and 250 mg/s (400 cm<sup>3</sup>/min and 12 dm<sup>3</sup>/min at 0 °C and 101,325 kPa with nitrogen).

Table 2. Uncertainty budget for the primary standard between 8 mg/s and 250 mg/s

Uncertainty component $x_i$	Standard uncertainty in parameter $u(x_i)$	Sensitivity coefficient $c_i$	$c_i \cdot u(x_i)$
Mass loss (10 g to 50 g)	8,0 mg	1/t	(1/t)·8,0
Time interval (200 s to 1200 s)	0,072 s	m/t <sup>2</sup>	(m/t <sup>2</sup> )·0,072
Reproducibility	$8 \cdot 10^{-4} \cdot q_m$	1	$8 \cdot 10^{-4} \cdot q_m$
Leakage out of the bench	$5,8 \cdot 10^{-4}$ mg/s	1	$5,8 \cdot 10^{-4}$

Quantity:  $q_m$

Expanded Uncertainty:  $1,8 \cdot 10^{-3} \cdot q_m$

Coverage Factor: 2,0

Coverage: 95,45%

## References

Nakao S. and al.: Intercomparison Tests of the NRLM Transfer Standard with the Primary standards of NIST, BNM-LNE, OFMET and PTB for Small Mass Flow Rates of Nitrogen Gas, In: Proceedings Metrologie 99, 9th International Metrology Congress, Bordeaux, France, October 18 - 21, 1999

Knopf D., Barbe J., Richter W. and Marschal A.: Comparison of the gas mass flow calibration systems of the BNM-LNE and the PTB, Metrologia, 2001, 38, 197-

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