

Flow Instability Evaluation at the NMISA Gas Flow Laboratory

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Abstract

Interlaboratory comparison and verification measurements performed in the Gas Flow Laboratory indicated stability problems in the flow ranges below 50 mL/min and above 30 L/min. Initially the stability problems in the low flow range below 50 mL/min were attributed to the flow cell used as reference standard. The unstable measurements above 30 L/min were attributed to the lack of sufficient pressure drop in the flow path from the gas cylinder to the measuring point. However, further measurements performed indicated that this might not be the cause of the instabilities experienced. To further investigate the matter, a second reference standard was acquired, and the flow path was lengthened and equipped with more pressure regulators to ensure better pressure control and a larger pressure drop. This paper discusses the methods to determine the causes of the instability, the measurements performed, analysis of the measurement results and measures implemented to eliminate further problems.

This paper includes a discussion of the possible flow instability generated by the insertion of thermistors in the flow path to perform temperature measurements of volume flow devices. The Flow Laboratory regularly receives volumetric flow devices and to calculate standardised flow conditions, it is necessary to measure the gas temperature and pressure at the unit under test (UUT) location. Emphasis is given to the determination of the immersion depth and measurement position of the temperature sensor in the flow path to ensure no, or negligible, disturbances in the gas flow path.

1. Introduction

The National Metrology Institute of South Africa (NMISA), is mandated to provide measurement traceability to the South African industry. The NMISA Gas Flow Laboratory is ISO/IEC 17025 accredited for volume gas flow measurements in the flow range 5 mL/min to 50 000 mL/min.

Interlaboratory comparison and intermediate check measurements performed in the flow ranges 5 mL/min to 50 mL/min and 40 000 mL/min up to 50 000 mL/min have indicated unstable behaviour resulting in E_n values greater than one and reference standards deviating with more than the calculated measurement uncertainty [1]. Possible causes for this flow instability were identified and measurements were performed to investigate and solve these instabilities.

As the laboratory receives numerous volumetric flow devices (bubble flow meters) for calibration, temperature and pressure measurements at the

flow device's location must be performed to enable standard flow condition calculations. Measurements were performed with the thermistor positioned in the flow path at the inlet and the outlet of the unit under test to determine the best location for these measurements. Measurements were performed with the unit under test positioned to the outlet of the reference standard.

2. The NMISA Gas Flow Laboratory

The Gas Flow Laboratory is part of the Physical Metrology group of NMISA. Gas flow (volume gas flow) calibration services are offered in the flow range 5 mL/min to 50 000 mL/min. The laboratory is ISO/IEC 17025 accredited since March 2017. Nitrogen gas is used as the calibration medium. Typical instruments received for calibration include mass flow controllers, mass flow meters, bubble flow meters and rotameters.

The Flow Laboratory is equipped with two secondary reference gas flow standards as

highest-level standards, a Bios ML-800 positive displacement piston prover and Fluke molbloc-L laminar flow elements (LFE). As the laboratory is not equipped with a primary gas flow standard, the secondary reference standards are calibrated by National Metrology Institutes with relevant calibration and measurement capabilities (CMCs) in the BIPM key comparison database (KCDB). Traceability to international standards is therefore imported from these National Metrology Institutes [1].

3. Flow instability evaluation

The flow ranges 5 mL/min to 50 mL/min and 40 000 mL/min to 50 000 mL/min were investigated.

3.1 Flow range 5 mL/min to 50 mL/min

Currently the NMISA Gas Flow Laboratory is ISO/IEC 17025 accredited to perform measurements in the flow range 5 mL/min to 50 mL/min with a CMC value of 2% of reading. The measurements performed in this flow range indicate unstable behaviour.

Reference standards and equipment used in this flow range are:

- a) Two piston prover flow cells, ML-800-3 (50 sccm) and ML-800-10 (500 sccm)
- b) Two molbloc-L laminar flow elements, 5E1-VCR-V-Q (50 sccm) and 1E2-VCR-V-Q (100 sccm)
- c) 50 sccm mass flow controllers

Possible causes of the instability in this flow range were identified as:

- a) Stability of the reference standard
- b) Flow disturbances in the gas flow stream
- c) Tubing – length and diameter
- d) Moisture entering the flow line
- e) Restrictions (valves or adapters) in the flow line
- f) Environmental conditions
- g) Pressure drop over flow path
- h) Stabilization time
- i) Cleanliness of flow medium

Currently, the stability of the piston prover low flow cell, ML-800-3 (50 sccm), is suspected as being the main cause of the instability problem in this flow range. Since acquiring this flow cell, it has been sent back to the manufacturer twice because of malfunction (sticky piston) and instability problems. Communication with the manufacturer resulted in advice regarding filtration in the flow path. It seems the filter in place prior to the flow cell to ensure clean (contamination free) nitrogen

entering the flow cell and to smooth out flow fluctuations has a price. Stabilization time is long. What is long? The laboratory's calibration procedures prescribe a stabilization time of at least one hour before performing calibrations. As the calibration procedures prescribe four sets of measurements (ten readings per measurement set) per calibration point, a longer than one-hour stabilization time may cause the calibration of an instrument run over more than one day.

The Gas Flow Laboratory has been renovated to lengthen the flow path from the gas cylinder to the inlet of the unit under test. Lengthening the flow path includes using multiple stages pressure regulators to improve stabilization of the gas flow and implement a more sufficient pressure drop – 600 kPa to 500/400 kPa to 400/300 kPa. Before renovating the laboratory, ¼" tubing of approximately 1,5 meters was used to directly connect the outlet of the nitrogen gas cylinder to the inlet of the measuring path. The laboratory layout with lengthened flow path is shown in Figure 1.



Figure 1: Laboratory layout.

Intermediate checks between similar and overlapping flow cells and laminar flow elements were performed to investigate the flow instability in this flow range. Firstly, piston prover flow cells and laminar flow elements of the same flow ranges were compared – 50 sccm: ML-800-3 against 5E1-VCR-V-Q and 500 sccm: ML-800-10 against 5E2-VCR-V-Q. Thereafter the two flow cells ML-800-3 (50 sccm) and ML-800-10 (500 sccm) were compared to the 1E2-VCR-V-Q (100 sccm) laminar flow element [3].

Tables 1 to 6 report intermediate check results between different reference standards.

Table 1: Reference standards: LFE 5E1-VCR-V-Q (S/N 7029) versus ML-800-3 (S/N 128014).

Reference Value (Bios)	Indicated Value (LFE)	Correction	Expanded Uncertainty (k=2)	Meet Acceptance Criteria
mL/min	mL/min	mL/min	mL/min	
5.182	5.177	0.005	0.020	Yes
15.150	15.084	0.066	0.110	Yes
25.145	25.157	-0.012	0.165	Yes
30.042	30.150	-0.108	0.190	Yes
39.688	40.061	-0.373	0.172	No
48.953	49.437	-0.484	0.207	No

Table 2: Reference standards: LFE 5E2-VCR-V-Q (S/N 7030) versus ML-800-10 (S/N 135209).

Reference Value (Bios)	Indicated Value (LFE)	Correction	Expanded Uncertainty (k=2)	Meet Acceptance Criteria
mL/min	mL/min	mL/min	mL/min	
6.030	6.064	-0.034	0.043	Yes
50.444	50.527	-0.084	0.151	Yes
149.767	149.897	-0.130	0.414	Yes
250.001	250.272	-0.271	0.673	Yes
299.658	299.965	-0.307	0.795	Yes
399.407	399.826	-0.420	1.056	Yes
498.464	498.880	-0.416	1.315	Yes

Table 3: Reference standards: LFE 1E2-VCR-V-Q (S/N 7481) versus ML-800-3 (S/N 128014).

Reference Value (LFE)	Indicated Value (Bios)	Correction	Expanded Uncertainty (k=2)	Meet Acceptance Criteria
mL/min	mL/min	mL/min	mL/min	
5.478	5.491	-0.012	0.016	Yes
14.999	15.052	-0.053	0.043	No
25.205	25.282	-0.077	0.106	Yes
30.710	30.840	-0.130	0.099	No
40.494	40.654	-0.160	0.110	No
49.438	49.643	-0.205	0.123	No

Table 4: Reference standards: LFE 1E2-VCR-V-Q (S/N 7481) versus ML-800-10 (S/N 135209).

Reference Value (LFE)	Indicated Value (Bios)	Correction	Expanded Uncertainty (k=2)	Meet Acceptance Criteria
mL/min	mL/min	mL/min	mL/min	
5.375	5.388	-0.013	0.015	Yes
14.902	14.920	-0.018	0.028	Yes
25.114	25.137	-0.023	0.044	Yes
30.623	30.640	-0.017	0.055	Yes
40.412	40.425	-0.013	0.072	Yes
49.363	49.375	-0.012	0.088	Yes

Table 5: Reference standards: LFE 1E2-VCR-V-Q (S/N 7481) versus ML-800-3 (S/N 128014).

Reference Value (LFE)	Indicated Value (Bios)	Correction	Expanded Uncertainty (k=2)	Meet Acceptance Criteria
mL/min	mL/min	mL/min	mL/min	
5.478	5.491	-0.012	0.023	Yes
14.999	15.052	-0.053	0.085	Yes
25.205	25.282	-0.077	0.282	Yes
30.710	30.840	-0.130	0.234	Yes
40.494	40.654	-0.160	0.248	Yes
49.438	49.643	-0.205	0.247	Yes

Table 6: Reference standards: LFE 1E2-VCR-V-Q (S/N 7481) versus ML-800-10 (S/N 135209).

Reference Value (LFE)	Indicated Value (Bios)	Correction	Expanded Uncertainty (k=2)	Meet Acceptance Criteria
mL/min	mL/min	mL/min	mL/min	
5.375	5.388	-0.013	0.019	Yes
14.902	14.920	-0.018	0.035	Yes
25.114	25.137	-0.023	0.047	Yes
30.623	30.640	-0.017	0.060	Yes
40.412	40.425	-0.013	0.075	Yes
49.363	49.375	-0.012	0.098	Yes

Table 1 reports measurements with the two reference standards deviating from one another more than the calculated measurement uncertainty at flow rates 40 mL/min and above. Table 2 indicates reference standards complying with the acceptance criteria in the flow range up to 50 mL/min. Table 3 shows measurement results with four out of six measurement points where the reference standards deviated more than the calculated measurement uncertainty. Table 1 and Table 3 report measurements with the ML-800-3 flow cell as one of the two comparing reference

standards. Table 4 to Table 6 report measurements with the acceptance criteria for intermediate checks met.

Table 3 to Table 6 indicate results for measurements performed comparing the ML-800-3 and ML-800-10 flow cells against the same laminar flow element, 1E2-VCR-V-Q, at the same measurement points over the flow range 5 mL/min to 50 mL/min. However, the measurement uncertainty for the measurements in Table 3 and Table 4 were calculated evaluating the repeatability of the error using the standard deviation of the mean and in Table 5 and Table 6 evaluating the repeatability of the error based on maximum deviation, using the standard deviation value instead of the standard deviation of the mean.

The ML-800-10 flow cell complied with the acceptance criteria irrespective of how the repeatability of the error is evaluated in the uncertainty measurement evaluation or against which other reference standard it is compared to. The ML-800-3 flow cell complied only where the repeatability of the error was treated based on maximum deviation, using the standard deviation value and not the standard deviation of the mean - only complying when the measurement uncertainties have been increased.

3.2 Flow range 40 000 mL/min to 50 000 mL/min
Currently the NMISA Gas Flow Laboratory is ISO/IEC 17025 accredited to perform measurements in the flow range 40 000 mL/min to 50 000 mL/min with a CMC value of 0,5% of reading. The measurements performed in this flow range indicate unstable behavior.

Reference standards and equipment used in this flow range:

- a) One piston prover flow cell, ML-800-44 (50 SLM)
- b) One molbloc-L laminar flow element, 3E4-VCR-V-Q (50 SLM)
- c) 50 SLM mass flow controller

Of all the possible causes of instability, insufficient pressure drop over the flow path has been identified as being the main cause of the instability problem in this flow range.

Again, intermediate checks between similar and overlapping flow cells and laminar flow elements were performed to investigate the flow instability in this flow range. Firstly, the piston prover flow cell and laminar flow element of the same flow range

were compared – 50 SLM: ML-800-44 against 3E4-VCR-V-Q. Thereafter the two flow cells, ML-800-44 (50 SLM) and ML-800-75 (100 SLM) were compared.

Tables 7 to 8 report intermediate check results between the different reference standards.

Table 7: Reference standards: LFE 3E4-VCR-V-Q (S/N 7028) versus ML-800-44 (S/N 128098).

Reference Value (Bios)	Indicated Value (LFE)	Correction	Expanded Uncertainty (k=2)	Meet Acceptance Criteria
mL/min	mL/min	mL/min	mL/min	
534.06	542.25	-8.19	20.57	Yes
5028.22	5036.06	-7.84	23.54	Yes
15053.53	15061.14	-7.61	45.24	Yes
25008.40	25006.61	1.79	66.27	Yes
30037.83	30041.92	-4.09	69.27	Yes
40007.98	40000.79	7.18	94.72	Yes
49272.40	49255.09	17.31	115.00	Yes

Table 8: Reference standards: ML-800-44 (S/N 128098) versus ML-800-75 (S/N 136463).

Reference Value (ML-800-44)	Indicated Value (ML-800-75)	Correction	Expanded Uncertainty (k=2)	Meet Acceptance Criteria
mL/min	mL/min	mL/min	mL/min	
15154.85	15133.83	21.02	35.24	Yes
25042.28	24979.90	62.38	69.27	Yes
30154.23	30084.93	69.30	93.93	Yes
40205.38	40017.05	188.32	130.21	No
47642.75	47463.08	179.68	259.41	Yes

Table 7 and Table 8 report measurements performed with the lengthened flow path using multiple stages pressure regulators inducing a pressure drop from 600 kPa to 300 kPa.

Table 7 reports intermediate checks performed in the flow range 500 mL/min to 50 000 mL/min which indicated good agreement between the two reference standards, the ML-800-44 flow cell and the 3E4-VCR-V-Q laminar flow element. Deviations between the two reference standards agree within the calculated measurement uncertainty.

An alternative method was used to check measurement results above 10 000 mL/min. Two flow cells, ML-800-44 and ML-800-75, with overlapping flow ranges, were used to perform the measurements being reported in Table 8. At 40 000 mL/min the acceptance criteria were not met.

4. Temperature and pressure measurements at the location of the unit under test

To calibrate volume flow meters, the NMISA Gas Flow Laboratory measures the temperature and pressure of the gas in the flow path at the location of the UUT to enable standard flow condition calculations. A reference thermometer (thermistor) and barometer are used respectively [2].

To determine the preferred position for measuring the temperature and pressure, measurements were performed with the thermistor positioned to the inlet and to the outlet of the unit under test. The unit under test (piston prover) was connected to the outlet of the reference standard (laminar flow element); the outlet of the unit under test was open to atmosphere. A ¼" union cross adapter is used for the connection of the thermistor and barometer. As the flow path is only a ¼" in width, there is no other option than to position the thermistor in the middle of the connector.

Figures 2 and 3 show the calibration setup with the thermistor and barometer connected to the inlet of the unit under test.



Figure 2: Thermistor and barometer connected to the inlet of the unit under test.



Figure 3: Thermistor and barometer connected to the inlet of the unit under test.

Table 9 reports calibration results with the thermistor connected to the inlet of the unit under test.

Table 9: Thermistor positioned at the inlet of unit under test.

Flow values are standardised to a reference temperature of 0 °C and reference pressure of 101.325 kPa				
UNIT UNDER TEST		Reference Flow Rate (mL/min)	Correction (Standardised Flow Rate) (mL/min)	Expanded Uncertainty (k=2) (mL/min)
Volumetric Flow Rate (mL/min)	Calculated Standardised Flow Rate (mL/min)			
639.736	508.183	505.691	-2.492	1.539
1270.918	1008.986	1004.486	-4.500	2.631
1904.123	1509.965	1504.523	-5.442	2.877

Figures 4 and 5 show the calibration setup with the thermistor and barometer connected to the outlet of the unit under test.



Figure 4: Thermistor and barometer connected to the outlet of the unit under test.



Figure 5: Thermistor and barometer connected to the outlet of the unit under test.

Table 10 reports calibration results with the thermistor connected to the outlet of the unit under test.

Table 10: Thermistor positioned at the outlet of unit under test.

Flow values are standardised to a reference temperature of 0 °C and reference pressure of 101.325 kPa

UNIT UNDER TEST		Reference Flow Rate (mL/min)	Correction (Standardised Flow Rate) (mL/min)	Expanded Uncertainty (k=2) (mL/min)
Volumetric Flow Rate (mL/min)	Calculated Standardised Flow Rate (mL/min)			
635.981	505.952	504.738	-1.214	0.965
1264.635	1005.602	1003.919	-1.683	1.778
1894.333	1505.862	1504.006	-1.856	2.829

The correction values differ between the inlet and outlet measurement positions with more than the calculated measurement uncertainty. For volumetric instruments like bubble flow meters the temperature and pressure measurements can only be measured at the inlet of the instrument.

5. Conclusion

From the measurements performed, it can be concluded that the behaviour of the ML-800-3 flow cell is indeed the cause of E_n values larger than one and reference standards deviations exceeding the calculated measurement uncertainty in the low flow range. It is recommended that one more set of measurements is performed in the flow range 5 mL/min to 50 mL/min to assign the reason for the instability behaviour in this flow range to the ML-800-3 flow cell. This set of measurements will compare the two molbloc-L laminar flow elements; 5E1-VCR-V-Q and 1E2-VCR-V-Q, thus the

50 sccm laminar flow element against the 100 sccm laminar flow element over this flow range.

The measurement results in the flow range above 40 000 mL/min showed satisfactory results. However, it is recommended that measurements are again performed in this flow range with the two reference standards, ML-800-44 flow cell and 3E4-VCR-V-Q laminar flow element. As these two reference standards are currently the only available instruments for the flow range up to 50 000 mL/min, it is recommended that measurements are performed using a needle valve to control the flow rate instead of a mass flow controller.

More measurements must be performed to investigate the unit under test temperature and pressure measurement location. These measurements must also be performed using the piston prover as reference standard where instruments are positioned in line with the reference standard.

References

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