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National Metrology Institute of South Africa

Flow Instability Evaluation at the NMISA Gas Flow Laboratory

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Your measure of excellence

Introduction

- NMISA is mandated to provide measurement traceability to the South African industry
- Gas Flow Laboratory - ISO/IEC 17025 accredited for volume gas flow measurements in the flow range 5 mL/min to 50 000 mL/min

Introduction

- ILCs and intermediate check measurements
  - Unstable behaviour resulting in $E_n > 1$
    - 5 mL/min to 50 mL/min
    - 40 000 mL/min up to 50 000 mL/min
- Possible causes for this flow instability were identified and measurements were performed to investigate and solve these instabilities

Introduction

- 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

NMISA Gas Flow Laboratory

- 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
- ISO/IEC 17025 accredited since March 2017
- Nitrogen gas is used as the calibration medium
- Typical instruments received for calibration
  - mass flow controllers
  - mass flow meters
  - bubble flow meters
  - rotameters

NMISA Gas Flow Laboratory

- Equipped with two secondary reference gas flow standards as highest-level standards
  - Bios ML-800 positive displacement piston prover
  - Fluke molbloc-L laminar flow elements (LFE)
- No primary gas flow standard
- 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

Flow instability evaluation
5 mL/min to 50 mL/min

- 5 mL/min to 50 mL/min with a CMC value of 2% of reading
- Reference standards and equipment used in this flow range are
  - Two piston prover flow cells
    - ML-800-3 (50 sccm)
    - ML-800-10 (500 sccm)
  - Two molbloc-L laminar flow elements
    - 5E1-VCR-V-Q (50 sccm)
    - 1E2-VCR-V-Q (100 sccm)
  - 50 sccm mass flow controllers

Flow instability evaluation
5 mL/min to 50 mL/min

- Possible causes of the instability in this flow range were identified as:
    - Stability of the reference standard
    - Flow disturbances in the gas flow stream
    - Tubing – length and diameter
    - Moisture entering the flow line
    - Restrictions (valves or adapters) in the flow line
    - Environmental conditions
    - Pressure drop over flow path
    - Stabilization time
    - Cleanliness of flow medium

Flow instability evaluation
5 mL/min to 50 mL/min

- Stability of the piston prover low flow cell ML-800-3 is suspected as being the main cause of the instability problem in this flow range
  - Filtration in the flow path
    - Filter in place prior to the flow cell to ensure clean (contamination free) nitrogen
    - Smooth out flow fluctuations
      - STABILIZATION TIME LONG

Flow instability evaluation
5 mL/min to 50 mL/min

- 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

Flow instability evaluation
5 mL/min to 50 mL/min

Flow instability evaluation
5 mL/min to 50 mL/min

- 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
    - 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

Flow instability evaluation

5 mL/min to 50 mL/min

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 1 - reference standards deviating from one another more than the calculated measurement uncertainty at flow rates 40 mL/min and above
- Table 2 - reference standards complying with the acceptance criteria in the flow range up to 50 mL/min
- Table 3 – four out of six measurement points - reference standards deviated more than the calculated measurement uncertainty

Flow instability evaluation

5 mL/min to 50 mL/min

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

- Tables 4 to 6 report measurements with the acceptance criteria for intermediate checks met

Flow instability evaluation

5 mL/min to 50 mL/min

- Tables 3 to 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

Flow instability evaluation
5 mL/min to 50 mL/min

- 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
- 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

Flow instability evaluation
40 000 mL/min to 50 000 mL/min

- 40 000 mL/min to 50 000 mL/min with a CMC value of 0,5% of reading
- Reference standards and equipment used in this flow range
  - One piston prover flow cell
    - ML-800-44 (50 SLM)
  - One molbloc-L laminar flow element
    - 3E4-VCR-V-Q (50 SLM)
  - 50 SLM mass flow controller

Flow instability evaluation
40 000 mL/min to 50 000 mL/min

- 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

Flow instability evaluation
40 000 mL/min to 50 000 mL/min

- 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

Flow instability evaluation
40 000 mL/min to 50 000 mL/min

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

- Tables 7 and 8
  - Measurements performed with the lengthened flow path using multiple stages pressure regulators inducing a pressure drop from 600 kPa to 300 kPa
- Table 7
  - Intermediate checks performed in the flow range 500 mL/min to 50 000 mL/min which indicated good agreement between the two reference standards
  - Deviations between the two reference standards agree within the calculated measurement uncertainty
- Table 8
  - 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
    - At 40 000 mL/min the acceptance criteria were not met

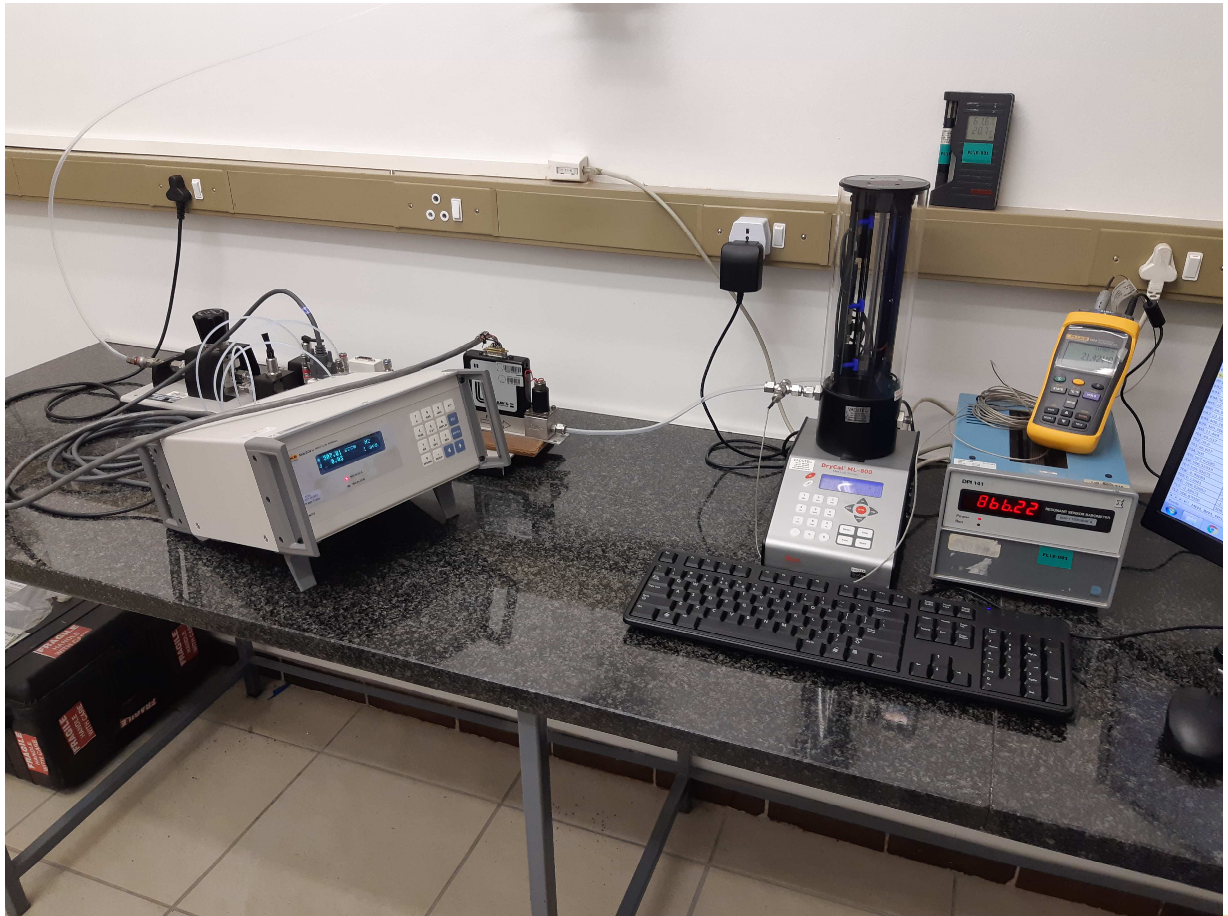
Temperature and pressure measurements at the location of the unit under test

- 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
  - reference thermometer (thermistor)
  - barometer

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

- To determine the preferred position
  - 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

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



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

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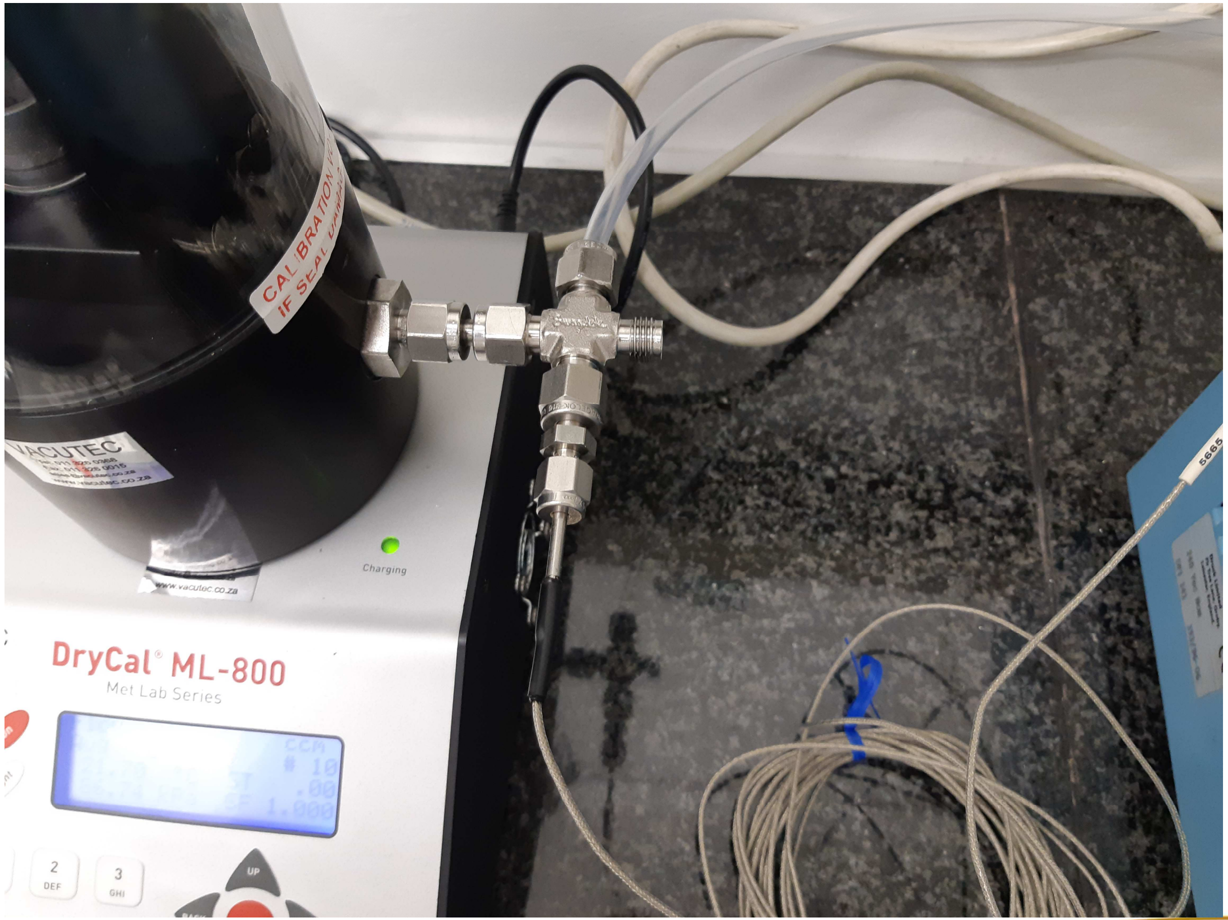


Temperature and pressure measurements at the location of the unit under test

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

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

Conclusion

5 mL/min to 50 mL/min

- 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

Conclusion

- It is recommended that one more set of measurements is performed in this flow range 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
    - 50 sccm versus 100 sccm laminar flow elements

Conclusion

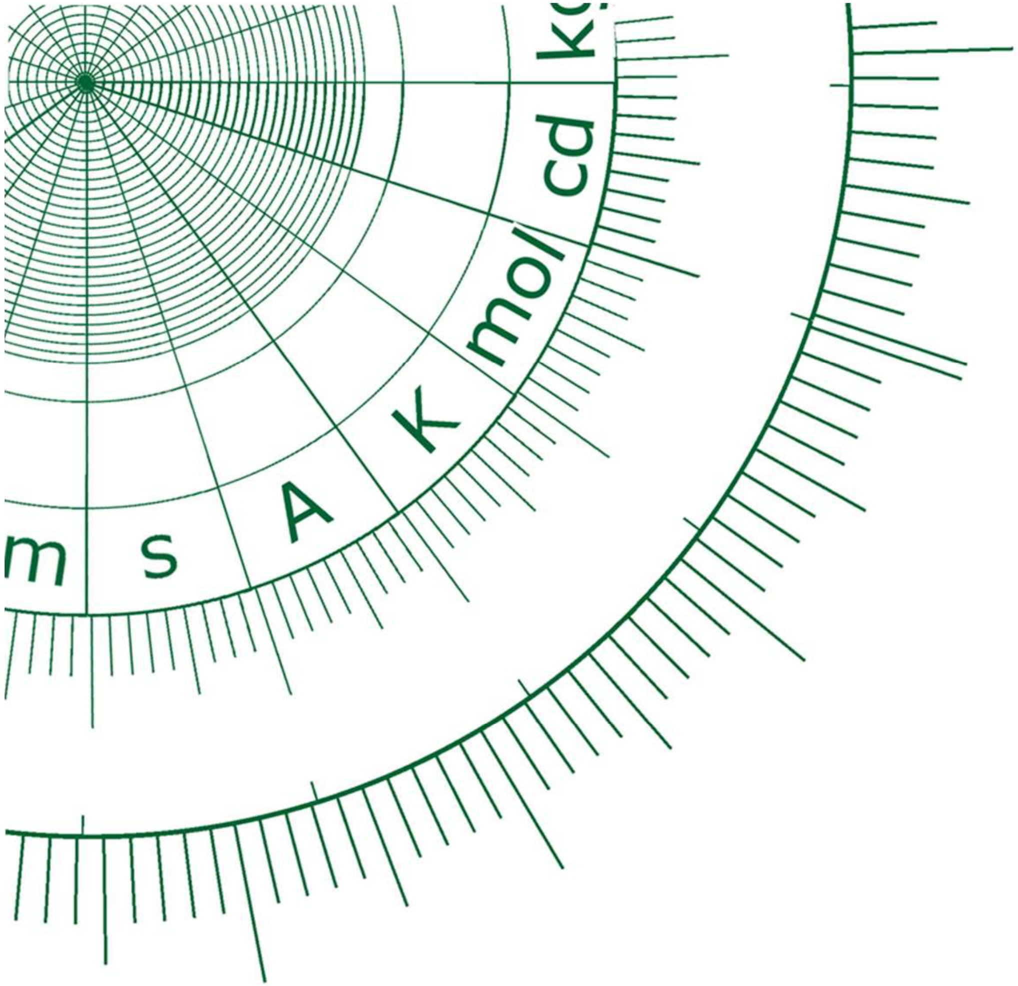
40 000 mL/min to 50 000 mL/min

- 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
  - 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

Conclusion

Temperature and pressure measurements

- 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



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Thank You

We measure what matters