SMALL GAS MASS FLOW STANDARD COMPARISONS 1996 – 2001
WORKING GROUP MEETING / LNE 24, 25 SEPTEMBER 2001
REPORT

Final draft (26/04/2002)

SITUATION AND OBJECTIVES

In the last ten years, significant evolutions appeared in various National Metrology Institutes in the field of primary facilities for small gas mass flow calibration (10 ml/min to 10 l/min). The origin of the needs was diverse (Dynamic preparation of reference gas mixtures, semi-conductor process control, leak testing etc…) and the initiators of these actions came from different fields. The type(s) of solution implemented (Principle, applications) were also varied ; some were based on improvement of known methods, some others were new approaches.

As the new facilities were becoming operational, the various NMIs were keen to compare their methodologies to those of other colleagues.

For practical reasons, it appeared that the solutions to this need could be found in a set of bilateral comparisons organized in Europe in the framework of Euromet project 412 (Pilot LNE). As similar interest existed outside Europe, comparisons were enlarged to a worldwide scale.

The overall objective was that any participant would be « linked » directly or indirectly to all others.

The variety of principles used in the calibration equipement as well practical conditions could have led to difficulties. In fact, this variety was useful in demonstrating the robustness of the exercise.

Detailed results on the facilities and various bilatetal comparisons have been reported in papers listed in annexes 1 to 3. It was however found useful to draw some synthetic conclusions of the five years of work ; a working group meeting of the participants met for that purpose at the Laboratoire National d'Essais in Paris on the 24th and 25th of September 2001. Representatives of the following laboratories were present : NIST (USA), NMIJ (Japan), METAS (CH), IMGC (Italy), BNM-LNE (France), NMi (NL), CMI (CZ), BAM (Germany), DHI (USA) and BIPM.

SYNTHETIC CONCLUSIONS

Two categories of primary low gas flow standards based on completely different principles (gravimetric and volumetric) were involved. The participating laboratories calibrated laminar flow elements (LFEs) or sonic nozzles whose nominal working range was consistent with the measuring range of their primary or transfer standards.

RESULTS

- The relative differences between participants in the subrange 100 ml/min to 1 l/min are close and often less than 0.1 %. In the subrange 1 l/min to 10 l/min, the differences are similar but not so many laboratories were concerned. For flows higher than 10 l/min, the deviations do not increase significantly but only two participants were involved. The differences increase up to 0.3 % in the subrange 10 ml/min to 100 ml/min. Deviations up to 0.4 % are observed for the range below 10 ml/min.

  The between labs deviations are well inside the estimated uncertainties. Some laboratories whose experience is more recent should confirm this situation by additional comparisons.

  This global result appears quite satisfactory to the participants and can be well compared to other fields of flow calibrations.

- The sources of uncertainty to be considered, may be classified as follows to support the conclusions :

  1) Sources of uncertainty well understood and modelled.

  2) « black box » phenomena which can be evaluated by test or even as allowance for possible effects.

  3) Safety margin for presenting “commercial” capabilities and every-day working conditions.

  The presentations at the meeting of the calibration facilities and uncertainties by the various participants were a good contribution to harmonisation for type (1) sources listing and quantification.

  Nevertheless it became clear that, when comparing the results obtained by two laboratories, it is essential that correlated uncertainties be taken into account. The most typical source of correlation is use of the same transfer standard.

  Validation studies should aim at reducing type (2) sources to the minimum possible, by understanding all sources of uncertainty and treating them as type (1) sources.

  Interim solutions may however be necessary to prevent optimistic quotation of uncertainties.

  Type (3) sources of uncertainty are not to be considered in NMI comparisons report(s) and evaluation.

RECOMMENDATIONS FOR THE FUTURE

Range of work

In the range 10 ml/min to 10 l/min of nitrogen which was the main field of interest of the meeting, the sum of results obtained allows to reduce the number and frequency of future comparisons except for new-comers or if new methods are developed.

Some considerations should be given to other types of gas with specific interest in certain fields eg. gas analyser, semi-conductor process control, health, leak testing…

The range of smaller flow from 10 ml/min down to 0,01 ml/min should be carefully watched and fields of application identified. This range will necessitate implementation of new principles or adaptations ; examples were given during the meeting and visit : Micro-volumetry by NMi, Traced Gas by LNE.

For flow rate higher than 100 l/min, needs were reported, but some of the participants were concerned about risks of possible duplications with other already existing actions.

In support to this action, a tentative list to be completed is proposed in annex 4.

Organisation of future comparisons

It was generally recognized that the network of interested participants present at the meeting should be kept active and enlarged when appropriate.

A major step forward would be to link it to an existing structure (eg. the Working Group on Flow of the CCM[1]and/or the Working Group on Gas Metrology of the CCQM[2]); a minimum action would be that participants inform each others about future points of interest in the field.

It was also recommended that future comparisons would be optimised in their planning to minimise effects not related to the uncertainties of the primary facilities. The transfer standard should be used in conditions that give the best reproducibility (same points, same protocol); these conditions have to be defined and accepted before the exercice. Moreover appropriate hardware and logistical needs must also be considered before, in order that leaks or compatibility of electronics do not jeopardize the results.

Considering that a comparison involving travel of participant personnel complicates scheduling and increases costs, it was suggested that comparison of sensor(s) sent by post could be an interesting alternative. This will impose a more detailed definition of protocols and compatibility of the equipment.

[1] CIPM Consultative Committee for Mass and related quantitities
[2] CIPM Consultative Committee for Amount of Substance

ANNEX 1

Table of bilateral comparisons (1996-2001)

H ↓ / V →	BNM-LNE	METAS	PTB	IMGC	CMI	NMIJ	NIST
BNM-LNE	✶	2001-1 2cc-10l [1]	1998-9 50cc-1l [4]			1996-1 100cc-10l 1998-6 10cc-30l [5]	
METAS	1996-7 20cc-10l	✶			2001-2 10cc-10l	1998-10 [5]	
PTB	1999-6 20cc-1l [4]	2000-12 20cc-2l	✶			1998-6 2cc-1l [5]	
IMGC	2001-3 6cc-1l [2]			✶			2001-9 0.2cc-1l [8]
CMI					✶	1999	
NMIJ	1997-11 100cc-10l [7]					✶	+
NIST	1999-11 2cc-10l [3]					+ [5] [6]	✶

V = Visiting laboratory ; H = Host laboratory
cc = sccm = ml/min at 0 °C ; l = slm = l/min at 0 °C
+ = additional comparisons between NIST and NMIJ indicated for information

List of publications and internal reports :

[1] Niederhauser B., Barbe J. : *Bilateral comparison of primary low gas flow standards between the BNM-LNE and METAS,* Metrologia, 2002, 39, 573-578

[2] A primary standard piston prover for measurement of very small gas flows (G. Cignolo, F. Alasia, A. Capelli, G. La Piana, in preparation for the 5th ISFFM)

[3] Gas flow standards for the semiconductor industry (Internal progress report, Robert F. Berg, Pressure and Vacuum Group, 26 April 2000)

[4] 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, 197-202

[5] 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

[6] Wright J.D., Mattingly G.E., Nakao S., Yokoi Y. and Takamoto M.: *International comparison of a NIST primary standard with an NRLM transfer standard for small mass flow rates of nitrogen gas*, Metrologia, 1998, 35, 211-221

[7] Nakao S., Yokoi Y.,Hirayama T., Takamoto M, Barbe J. and Marschal A.: *Intercomparison Tests with NRLM for Small Mass Flow Rates of N_2, Ar and He in the range from 40 mg/min to 10 g/min,* In: Proceedings FLOMEKO'98, Lund, Sweden, June 15 - 17, 1998

[8] Berg R.F. and Cignolo G. : NIST - IMGC comparison of gas flows below 1 liter per minute (NIST report in preparation, 2002)

ANNEX 2

Small gas mass flow working group (participants and papers)

Participant	Paper	Author / Member
NIST (USA)	NIST Piston Prover Gas Flow Facilities in the range from 37 ml/min to 22 l/min	John D. Wright Tel : 301 975-5937 Fax : 301 258-9201 Email : john.wright@nist.gov
	Results of comparisons ; satisfactions and concerns	
	Water Bath Performance for a Primary Gas Flow Standard for 1 LPM to 2000 LPM	
	Model of a quartz capillary transfer standard for flow	Robert F. Berg Tel : 301 975 2466 Fax : 301 208 6962 Email : robert.berg@nist.gov
NMIJ (Japan)	The primary standard for low flow rates of NMIJ and calibration services in Japan	Shin-ichi Nakao Email : s.nakao@aist.go.jp
	Transfer standard using sonic nozzles of NMIJ	
	Results of comparisons ; satisfactions and concerns	
METAS (CH)	Swiss primary volumetric standard for low gas flows : experiences and progress	Bernhard Niederhauser Tel : +41 31 32 33 262 Fax : +41 31 32 33 210 Email : bernhard.niederhauser@metas.ch
	Results of comparisons ; satisfactions and concerns	
IMGC (Italy)	The 3-Litre capacity piston prover of the IMGC	Giorgio Cignolo Tel : +39 011 3977448 Fax : +39 011 3977437 Email : g.cignolo@imgc.cnr.it
BNM/LNE (France)	The primary gravimetric calibration systems in the range from 0,02 mg/s to 2 g/s Results of comparisons ; satisfactions and concerns	Jean Barbe / Alain Marschal Tél : + 33 1 40 43 37 80 Fax : + 33 1 40 43 37 37 Email : jean.barbe@lne.fr
NMi (NL)	The micro-flow facility in the range from 0.1 ml/h up to 20 ml/h The new servo-piston system developed at NMI as an alternative for the mercury piston provers	Mijndert van der Beek Tel : + 31 78 6332332 Fax : + 31 78 6332309 Email : mvanderbeek@nmi.nl
PTB (Germany) **unable to attend**	Comparison of the gas mass flow calibration systems of the BNM-LNE and the PTB	Dorothea Knopf Tel : + 49 531 592 3325 Fax : + 49 5317592 3015 Email : Dorothea.Knopf@ptb.de
DHI (USA)	Optimizing molbloc/molbox Use in Metrology	Pierre Delajoud / Martin Girard Email : delajoud@compuserve.com
BAM (Germany)	-	Hans-Joachim Heine Email :Hans-Joachim.Heine@bam.de
CMI (CZ)	-	Zdenek Krajicek Email : zkrajicek@cmi.cz
BIPM	-	Richard Davis Email : rdavis@bipm.org

ANNEX 3

Equivalence matrix (relative deviation divided by the relative uncertainty)

Nominal flow in the range from 100 ml/min to 10 l/min using nitrogen

H ↓ / V →	BNM-LNE	METAS	PTB	IMGC	CMI	NMIJ	NIST	Mean
BNM-LNE	*	**-0.04**	-0.37	0.13		**0.09**	0.00	-0.04
METAS	0.04	*	-0.44					-0.20
PTB	**0.37**	**0.44**	*					0.41
IMGC	**-0.13**			*			**0.28**	0.08
CMI					*			
NMIJ	-0.09					*		-0.09
NIST	**0.00**			-0.28			*	-0.14

Nominal flow in the range higher than 10 l/min

H ↓ / V →	BNM-LNE	METAS	PTB	IMGC	CMI	NMIJ	NIST	Mean
BNM-LNE	*					**0.03**		0.03
METAS		*						
PTB			*					
IMGC				*				
CMI					*			
NMIJ	-0.03					*		-0.03
NIST							*	

Nominal flow in the range below 100 ml/min

H ↓ / V →	BNM-LNE	METAS	PTB	IMGC	CMI	NMIJ	NIST	Mean
BNM-LNE	*	**-0.33**	-0.37	-0.03		**-0.26**	0.33	-0.13
METAS	0.33	*	-0.57					-0.12
PTB	**0.37**	**0.57**	*					0.47
IMGC	**0.03**			*			**-0.28**	-0.13
CMI					*			
NMIJ	0.26					*		0.26
NIST	**-0.33**			0.28			*	-0.03

V = Visiting laboratory ; H = Host laboratory