Final Report

Bilateral inter-comparison in the gas flow range from 0.5 m^3/h to 75 m^3/h with sonic nozzles

EURAMET Project No. 1511

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1. Introduction

The project EURAMET no.1511 was an inter-comparison among two laboratories with sonic nozzles and the one officially started in January 2021 and was concluded in June 2021. The planned time schedule is mentioned down in *table 1*. Each country took almost 3 weeks to perform the calibrations of sonic nozzles. The nominal range of flow rates was from 0.5 m^3/h to 75 m^3/h. The participating laboratories used their usual calibration procedure. The comparison was conducted with respect to guidelines[1)].

In the moment when this report is issued, no CIPM key comparison was finished in the field of low pressure gas flow in all the relevant flow rates. That is why this inter-comparison is EURAMET supplementary comparison.

Table 1 – Time schedule and participants

Country	Laboratory	Address of the place of calibration	e-mail telephone	Date of calibration	Responsible person
Slovak Republic	SMU Slovak Institute of Metrology	Slovak Institute of Metrology Karloveská 63, 842 55 Bratislava, Slovak Republic	vavrovic@smu.gov.sk +421 260 294 322	March 2021	Patrik Vávrovič
Czech Republic (PILOT LAB)	CMI Czech Metrology Institute	CMI Regional Inspectorate Pardubice Průmyslová 455, 530 03 Pardubice, Czech Republic	tvalenta@cmi.cz +420 466 670 728	March/April 2021	Tomáš Valenta

2. The instruments

Sonic nozzles were used for inter-comparison. The dimensional characteristics and marking stickers are specified in the pictures mentioned down.

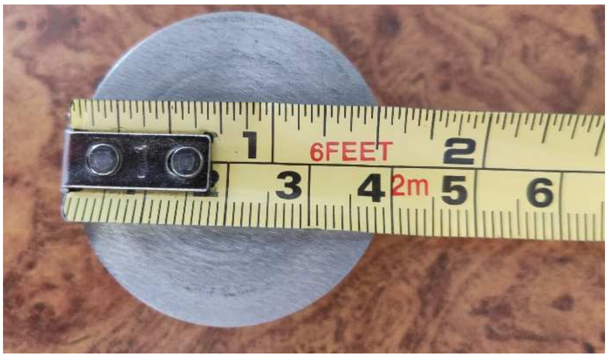
2.1. Sonic nozzle 75 m^3/h

1) - for CIPM key comparisons https://www.bipm.org/documents/20126/43742162/CIPM-MRA-G-11.pdf/9fe6fb9a-500c-9995-2911-342f8126226c
- for EURAMET comparisons – EURAMET Guide no.4 https://www.euramet.org/Media/news/G-GNP-GUI-004_Guide_on_Comparisons_web.pdf

2.2. Sonic nozzle 32 m^3/h

2.3. Sonic nozzle 16 m^3/h

2.4. Sonic nozzle 0.5 m^3/h



3. Calibration procedure

The calibration test procedure is mentioned in the document ***Wendt, G; Dietrich, H.; Jarosch, B.; Joest, R.; Natz, B.; Frössl, F.; Ruwe, M.: PTB testing instruction Volume 25: Gas meters – Test rigs with critical nozzles (English version 2000: 91 pages).***

The calibrations of a sonic nozzle with nominal flow rate **75 m^3/h** were performed according to the chapter *3.2.1 Determination of nozzle reference value* $Q_{v,20,dryAir}$ *(one point test).*

The calibrations of sonic nozzles with nominal flow rates **32 m^3/h, 16 m^3/h and 0.5 m^3/h** were performed according to the chapter *3.2.2 Determination of nozzle reference value* $Q_{v,20,tr,1000}$ *(two points test).*

The ambient temperature in laboratory had to be (21±1) °C and the relative humidity in laboratory had to be less than 80 % during the tests.

4. Test facility and obtained results

4.1. Slovak Republic

The primary standard of gas flow rate – the Bell Prover - was established by Slovak Institute of Metrology (SMU) during 1999 to 2002. The flow range of this standard is (from 1 to 65) m3/h, the expanded uncertainty of measurements is 0,12 %. This standard is listed in CMC tables BIPM (SK 13).

The standard works in the volumetric principle. The basic element of this equipment is the bell with accurately measured inner surface, which submerging into the closing liquid (oil with low viscosity and with low ability of evaporation in normal conditions of pressure and temperature). The bell replaces through the joined measuring instruments the exactly defined volume of air. Delivered volume of gas derived from the SI base units, the unit of the length and the time. The title of this equipment is „Primary standard with a bell“, or titled „Primary standard with a cubic gauge“ (shortly „PEZ“).

Range of flow rate: (1 to 65) m^3/h
Temperature: (20 ± 2)°C
Working pressure: atmospheric conditions
Uncertainty CMC (k=2): 0.12 % (NMI Service Identifier: SK13)

Place of calibration: Slovak Institute of Metrology
Karloveská 63, 842 55 Bratislava, Slovak Republic

Results:

Nozzle-ID s.n.	$Q_{V,20,dryAir}$	U(k=2)	p_{Test}
	[m^3/h]	[%]	[kPa]
01508	74.558	0.085	99.759

Nozzle-ID s.n.	$Q_{v.20.tr.1000}$	U(k=2)	c_{pE}
	[m^3/h]	[%]	[1/mbar]
JT-01-32000-1999	32.067	0.089	$1.07.10^{-5}$
JT-01-16000-1999	15.950	0.086	$1.07.10^{-5}$
JT-01-500-1999	0.493	0.127	$2.48.10^{-5}$

4.2. Czech Republic

Place of the test

Czech Metrology Institute, Gas Flow Department, Prumyslova 455, 530 03 Pardubice, Czech Republic

The test facility

A new national standard Bell Prover with the range from 0.5 m^3/h to 280 m^3/h was used for the calibrations of all the sonic nozzles. The bell was dimensionally very accurately evaluated by PTB. The manufacturer was company EP Ehrler Prüftechnik Engineering GmbH, Germany. The Bell Prover consists of:

- exactly dimensioned stainless steel bell
- connection system with switching device
- oil Shell Morlina 5
- fan, vacuum pump
- pressure vessel 2.7 m3
- control PC with software
- electronic digital thermometers with 0.01°C graduation scale, 4 pieces of manufacturer Temperaturmeßtechnik Geraberg GmbH,
- electronic digital pressure instruments with 1 Pa graduation scale , 5 pieces
  manufacturer PAROSCIENTIFIC, INC, 1 piece
  manufacturer YOKOGAWA, 3 pieces

manufacturer ROSEMOUNT, 1 piece

- incremental rulers with 0.001 mm graduation scale, 2 pcs
  producer HEDENHEIN
- timing circuit in a collecting unit serving as a stopwatch with a message of 0.001 s, 1 piece
  manufacturer Brehm + Jung
- hygrometer, 1 pc
  manufacturer JUMO

The nozzles were tested in sinking mode. Waiting time between measurements is 300 seconds. This Bell Prover is mentioned in CMC with NMI Service Identifier CZ21 and U(k=2)=0.07 %.

Results:

Nozzle-ID s.n.	$Q_{V,20,dryAir}$	U(k=2)	p_{Test}
	[m³/h]	[%]	[kPa]
01508	74.527	0.071	99.031

Nozzle-ID s.n.	$Q_{v.20.tr.1000}$	U(k=2)	c_{pE}
	[m³/h]	[%]	[1/mbar]
JT-01-32000-1999	32.092	0.071	$1.33.10^{-5}$
JT-01-16000-1999	15.950	0.071	$1.16.10^{-5}$
JT-01-500-1999	0.49350	0.071	$2.05.10^{-5}$

5. Determination of the reference values in determined flow rates

5.1 Description of the method

The reference value was determined in each flow rate separately, it means separately for each sonic nozzle. The method of determination of the reference value in each flow rate corresponds to the procedure A presented by M.G.Cox[2)]. Results from independent laboratories were taken into account for the determination of the key comparison reference value (KCRV) and of the uncertainty of the key comparison reference value.

5.1.1. The determination of the Key Comparison Reference Value (KCRV) and its uncertainty

The reference value y was be calculated as weighted mean of parameters (determined flow rates) $Q_{v,20,tr}$ or $Q_{v,20,tr,1000}$.

$$y = \frac{\frac{x_1}{u_{x1}^2} + \frac{x_2}{u_{x2}^2}}{\frac{1}{u_{x1}^2} + \frac{1}{u_{x2}^2}}, \qquad [1]$$

[2)] Cox M.G., *Evaluation of key comparison data*, Metrologia, 2002, **39**, 589-595 where x_1, x_2, x_{n3} are parameters $Q_{v,20,tr}$ or $Q_{v,20,tr,1000}$ of a sonic nozzle in different independent laboratories *1,2,3* [m³/h]

u_{x1}, u_{x2}, u_{x3} are standard uncertainties (not expanded) in different independent laboratories *1,2* [m³/h]

The standard uncertainty of the reference value u_y is given by

$$\frac{1}{u_y^2} = \frac{1}{u_{x1}^2} + \frac{1}{u_{x2}^2} \qquad [2]$$

The expanded uncertainty of the reference value $U(y)$ is

$$U(y) = 2.u_y \qquad [3]$$

The chi-squared test for consistency check will be performed using parameters $Q_{v,20,tr}$ or $Q_{v,20,tr,1000}$ of a sonic nozzle. At first the chi-squared value χ^2_{obs} will be calculated by

$$\chi^2_{obs} = \frac{(x_1 - y)^2}{u_{x1}^2} + \frac{(x_2 - y)^2}{u_{x2}^2} \qquad [4]$$

The degrees of freedom ν will be assigned

$$\nu = n - 1 \qquad [5]$$

where n is number of evaluated laboratories.

The consistency check will be failing if

$$Pr\{\chi^2_\nu > \chi^2_{obs}\} < 0,05 \qquad [6]$$

(The function *CHIINV(0,05;* ν*)* in MS Excel will be used. The consistency check will be failing if *CHIINV(0,05;* ν*)* $< \chi^2_{obs}$)

If the consistency check does not fail then y will be accepted as the key comparison reference value x_{ref} and $U(y)$ will be accepted as the expanded uncertainty of the key comparison reference value $U(x_{ref})$.

If the consistency check fails then the laboratory with the highest value of $\frac{(x_i - y)^2}{u_{xi}^2}$ will be excluded for the next round of evaluation and the new reference value y (WME), the new standard uncertainty of the reference value u_y and the chi-squared value χ^2_{obs} will be calculated again without the values of excluded laboratory. The consistency check will be calculated again, too. This procedure will be repeated till the consistency check will pass.

5.1.2. The determination of the differences "Lab to KCRV" and "Lab to Lab" as well as their uncertainties and Degrees of Equivalence

When the KCRV was determined, the differences between the participating laboratories and the KCRV were calculated according to

$$di = x_i - x_{ref} \qquad [7]$$

$$dij = x_i - x_j \qquad [8]$$

Based on these differences, the **D**egree **o**f **E**quivalence (*DoE*) was calculated according to:

$$Ei = \frac{di}{U(di)} \qquad [9]$$

and $$Eij = \frac{dij}{U(dij)} \quad , \quad \text{respectively.} \qquad [10]$$

The *DoE* is a measure for the equivalence of the results of any laboratory with the KCRV or with any other laboratory, respectively:

- The results of a laboratory is ***equivalent (passed) if*** **|*Ei*| *or* |*Eij*|≤*1.***
- The laboratory was determined as ***not equivalent (failed) if*** **|*Ei*| *or* |*Eij*| >*1.2***.
- For values of *DoE* in the range ***1* < |*Ei*| *or* |*Eij*| ≤ *1.2*** we define "**warning level**" were actions to check is recommended to the laboratory.

  The reason for such "warning level" is that we have to consider the confidence in the determination of the uncertainties (for the results of labs as well the KCRV). Conventionally we work at a 95% confidence level. Therefore in some comparisons a range up to |*E*| < *1.5* is used for these "warnings"[3)]. This is a reasonable value where stochastic influences dominate the uncertainty budgets. In the case of comparisons for gas flow, the smaller value 1.2 was chosen, which reflects the dominance of non-stochastic

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[3)] C. Ullner et al., *Special features in proficiency tests of mechanical testing laboratories*, and P. Robouch et al., *The „Naji Plot", a simple graphical tool for the evaluation of inter-laboratory comparisons*, parts of uncertainty compared to the stochastic parts. (The reproducibility is usually much better than the total uncertainty of a laboratory). [4)]

The calculation of the *DoE* needs the information about the uncertainty of the differences *di* and *dij* (equations [11] and [12]). To make statements about this, let us consider first the general problem of the difference of two values x_1 and x_2. If we look to the pure propagation of (standard) uncertainty we find:

$$u_{x_1-x_2}^2 = \begin{pmatrix} \frac{\partial(x_1-x_2)}{\partial x_1} & \frac{\partial(x_1-x_2)}{\partial x_2} \end{pmatrix} \begin{pmatrix} u_1^2 & \mathrm{cov} \\ \mathrm{cov} & u_2^2 \end{pmatrix} \begin{pmatrix} \frac{\partial(x_1-x_2)}{\partial x_1} \\ \frac{\partial(x_1-x_2)}{\partial x_2} \end{pmatrix} = u_1^2 + u_2^2 - 2.\mathrm{cov} \qquad [11]$$

Simply spoken, the (standard) uncertainty of the difference is the quadratic sum of the uncertainties of the inputs (u_1 and u_2) subtracting twice the covariance (*cov*) between the two input values.

Therefore, it is possible find the different cases in this comparison:

A) <u>Differences to the KCRV</u>

A1) *Independent laboratories with contribution to the KCRV*

The covariance between the result of a laboratory (with contribution to the KCRV) and the KCRV is the variance of the KCRV itself. [5)]

$$\Rightarrow u(di) = \sqrt{u_{xi}^2 + u_{xref}^2 - 2.u_{xref}^2} = \sqrt{u_{xi}^2 - u_{xref}^2} \qquad [12]$$

A2) *Independent laboratories without contribution to the KCRV*

There is no covariance between the result of a laboratory without contribution and the KCRV.

$$\Rightarrow u(di) = \sqrt{u_{xi}^2 + u_{xref}^2} \qquad [13]$$

B) <u>Differences Lab to Lab</u>

B1) *Independent laboratories*

There is no covariance between the results of two independent laboratory *i* and *j*

$$\Rightarrow u(dij) = \sqrt{u_{xi}^2 + u_{xj}^2} \qquad [14]$$

4) D.Dopheide, B.Mickan, R.Kramer, H.-J.Hotze, J.-P.Vallet, M.R.Harris, Jiunn-Haur Shaw, Kyung-Am Park, *CIPM Key Comparisons for Compressed Air and Nitrogen, CCM.FF-5.b – Final Report*, 07/09/2006

5) Cox M.G., *Evaluation of key comparison data*, Metrologia, 2002, **39**, 589-595

The equations from [12] to [14] use the standard uncertainties ($k = 1$). The expanded uncertainties $U(di)$ and $U(dij)$ (see equations [15],[16]) are determined by

$$U(di) = 2.u(di) \qquad [15]$$

$$U(dij) = 2.u(dij) \qquad [16]$$

5.2. Sonic nozzle with nominal flow rate 75m^3/h

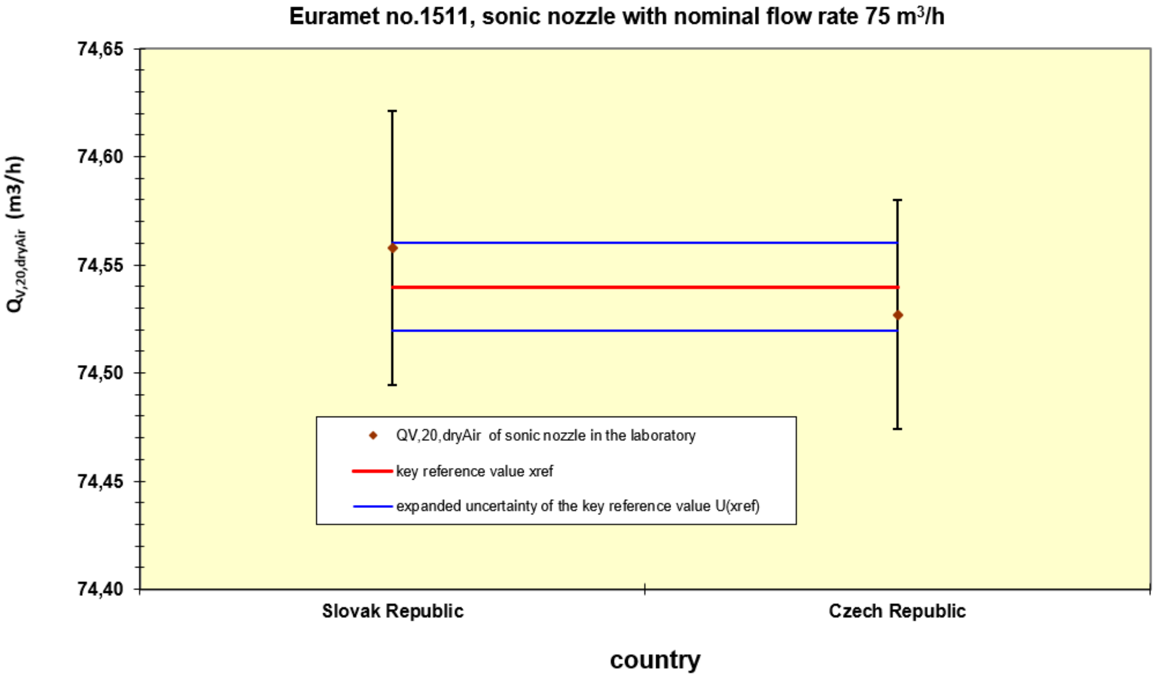
The first and last round of evaluation:

Country	$Q_{V,20,dryAir}$ x	Uncertainty $U(k=2)$	Uncertainty	$\frac{(x_i - y)^2}{\left(\frac{U(x_i)}{2}\right)^2}$	$1/u\text{^}2$
	(m^3/h)	(%)	(m^3/h)		
Slovak Republic	74.558	0.085	0.06337	0.332	995.941
Czech Republic	74.527	0.071	0.05291	0.232	1428.618

WME = y =	74.5397	m^3/h
U(y)=	0.02031	m^3/h
CHIINV	3.84146	
χ^2_{obs} =	0.56	

The consistency check passed because CHIINV > χ^2_{obs}

Country	$Q_{V.20.dryAir}$ x	Uncertainty $U(k=2)$	Uncertainty $U(k=2)$	di	$U(di)$	Ei
	(m^3/h)	(%)	(m^3/h)	(m^3/h)	(m^3/h)	
Slovak Republic	74.558	0.085	0.06337	0.0183	0.0600	0.30
Czech Republic	74.527	0.071	0.05291	-0.0127	0.0489	-0.26



5.3. Sonic nozzle with nominal flow rate 32 m³/h

The first and last round of evaluation:

Country	$Q_{v,20,tr,1000}$ x	Uncertainty $U(k{=}2)$	Uncertainty $U(k{=}2)$	$\frac{(x_i - y)^2}{\left(\frac{U(x_i)}{2}\right)^2}$	$1/u$^2
	(m³/h)	(%)	(m³/h)		
Slovak Republic	32.067	0.089	0.02854	1.145	4910.925
Czech Republic	32.092	0.071	0.02279	0.730	7704.593

WME = y = 32.0823 m³/h

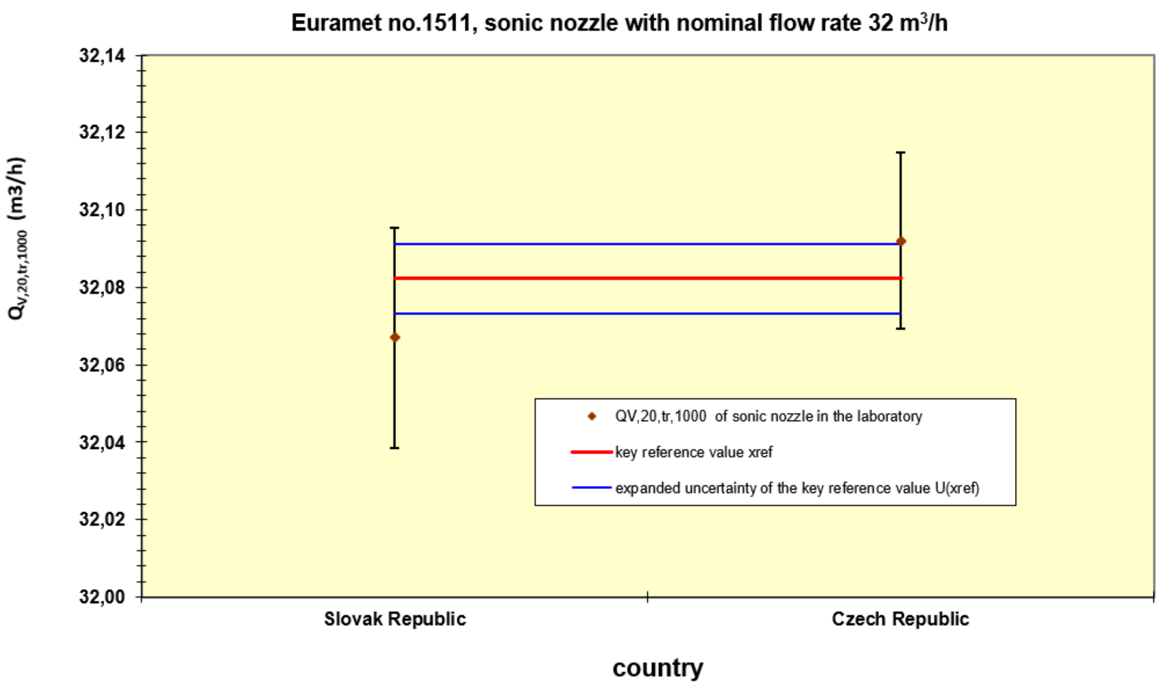
U(y)= 0.00890 m³/h

CHIINV 3.84146

χ^2_{obs} = 1.87

The consistency check passed because CHIINV > χ^2_{obs}

Country	$Q_{v,20,tr,1000}$ x	Uncertainty $U(k=2)$	Uncertainty $U(k=2)$	*di*	*U(di)*	*Ei*
	(m^3/h)	(%)	(m^3/h)	(m^3/h)	(m^3/h)	
Slovak Republic	32.067	0.089	0.02854	-0.0153	0.0271	-0.56
Czech Republic	32.092	0.071	0.02279	0.0097	0.0210	0.46



5.4. Sonic nozzle with nominal flow rate 16 m^3/h

The first and last round of evaluation:

Country	$Q_{v,20,tr,1000}$ x	Uncertainty $U(k=2)$	Uncertainty $U(k=2)$	$\frac{(x_i - y)^2}{\left(\frac{U(x_i)}{2}\right)^2}$	*1/u^2*
	(m^3/h)	(%)	(m^3/h)		
Slovak Republic	15.950	0.086	0.01372	0.000	21258.945
Czech Republic	15.950	0.071	0.01132	0.000	31190.470

WME = y = 15.9500 m³/h

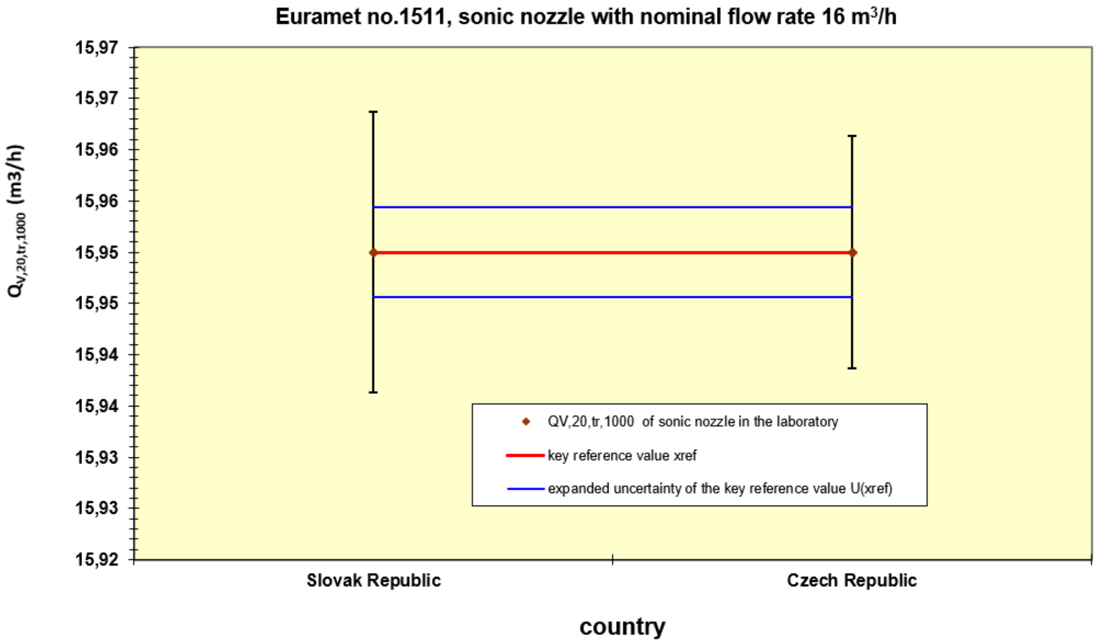
U(y)= 0.00437 m³/h

CHIINV 3.84146

χ^2_{obs} = 0.0000

The consistency check passed because CHIINV > χ^2_{obs}

Country	$Q_{v,20,tr,1000}$ x	Uncertainty $U(k=2)$	Uncertainty $U(k=2)$	di	$U(di)$	Ei
	(m³/h)	(%)	(m³/h)	(m³/h)	(m³/h)	
Slovak Republic	15.950	0.086	0.01372	0.0000	0.0130	0.00
Czech Republic	15.950	0.071	0.01132	0.0000	0.0104	0.00



5.5. Sonic nozzle with nominal flow rate 0.5 m³/h

The first and last round of evaluation:

Country	$Q_{v,20,tr,1000}$ x	Uncertainty $U(k=2)$	Uncertainty $U(k=2)$	$\frac{(x_i - y)^2}{\left(\frac{U(x_i)}{2}\right)^2}$	$1/u$^2
	(m³/h)	(%)	(m³/h)		
Slovak Republic	0.4930	0.127	0.00063	1.479	10203724.2
Czech Republic	0.4935	0.071	0.00035	0.463	32581342.8

WME = y = 0.49338 m^3/h

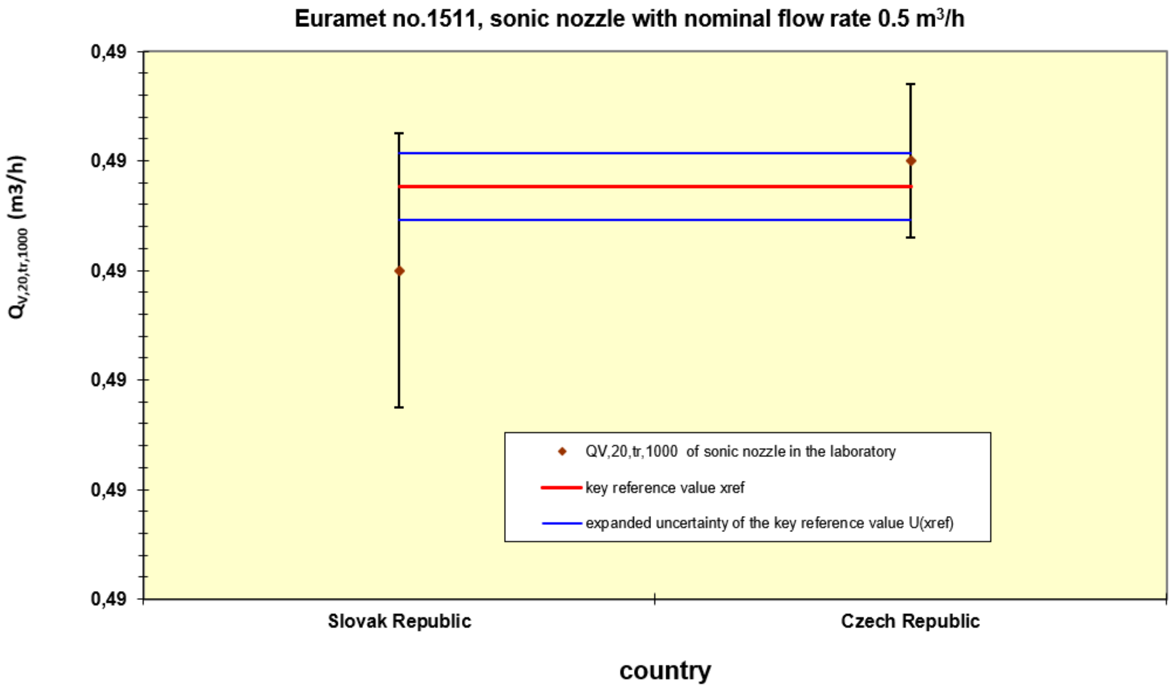
U(y)= 0.00015 m^3/h

CHIINV 3.84146

χ^2_{obs} = 1.94

The consistency check passed because CHIINV > χ^2_{obs}

Country	$Q_{v,20,tr,1000}$ *x*	Uncertainty *U(k=2)*	Uncertainty *U(k=2)*	*di*	*U(di)*	*Ei*
	(m^3/h)	(%)	(m^3/h)	(m^3/h)	(m^3/h)	
Slovak Republic	0.4930	0.127	0.00063	-0.00038	0.00061	-0.63
Czech Republic	0.4935	0.071	0.00035	0.00012	0.00032	0.38



6. Results

6.1. Czech Republic

Sonic nozzle nominal flow rate	$Q_{V,20,dryAir}$ or $Q_{v,20,tr,1000}$	uncertainty U(k=2)	uncertainty declared in CMC U(k=2)	key reference value x_{ref}	expanded uncertainty of the key refrence value $U(x_{ref})$	consistency check	di	Ei	result
m³/h	m³/h	%	%	m³/h	m³/h	-	m³/h	-	-
75	74.527	0.071	0.07	74.5397	0.02031	inside	-0.01270	-0,26	passed
32	32.092	0.071	0.07	32.0823	0.00890	inside	0.00970	0,46	passed
16	15.950	0.071	0.07	15.9500	0.00437	inside	0.00000	0,00	passed
0.5	0.4935	0.071	0.07	0.49338	0.00015	inside	0.00012	0,38	passed
						mean		0.15	passed

6.2. Slovak Republic

Sonic nozzle nominal flow rate	$Q_{V,20,dryAir}$ or $Q_{v,20,tr,1000}$	uncertainty U(k=2)	uncertainty declared in CMC U(k=2)	key reference value x_{ref}	expanded uncertainty of the key refrence value $U(x_{ref})$	consistency check	di	Ei	result
m³/h	m³/h	%	%	m³/h	m³/h	-	m³/h	-	-
75	74.558	0.085	0.12	74.5397	0.02031	inside	0.01830	0.30	passed
32	32.067	0.089	0.12	32.0823	0.00890	inside	-0.01530	-0.56	passed
16	15.950	0.086	0.12	15.9500	0.00437	inside	0.00000	0.00	passed
0.5	0.493	0.127	0.12	0.49338	0.00015	inside	-0.00038	-0.63	passed
						mean		-0.22	passed

7. Degree of equivalence between laboratories

The 14th CCM meeting (February, 2013) recommended that pair-wise degrees of equivalence no longer to be published in the KCDB and that information on pair-wise degrees of equivalence published in KC reports be limited to the equations needed to calculate them, with the addition of any information on correlations that may be necessary to estimate them more accurately.

8. Summary and conclusion

The summary of inter-comparison results is mentioned down in the table:

Sonic nozzle		Laboratory	
Serial number	Nominal flow rate (m^3/h)	**Czech Republic CMI**	**Slovak Republic SMU**
01508	75	passed	passed
JT-01-32000-1999	32	passed	passed
JT-01-16000-1999	16	passed	passed
JT-01-500-1999	0.5	passed	passed
Mean		**passed**	**passed**