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The New Static Expansion System Of METAS

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Abstract

The METAS static expansion system was built in 2004 and has been extensively characterised during the last two years.

The system consists of four expansion stages allowing the generation of calculable pressures ranging from 5·10-6 Pa up to 2000 Pa.

Uncertainty calculation yields a relative uncertainty of 0.001 (k=2) for the volumetric ratio of the expansion stage. The relative uncertainty on the pressure obtained after expansion is 0.003 above 10 Pa, it is still 0.01 at 2·10-4 Pa.

Measurement of the accommodation coefficient of two SRG from 10-5 Pa up to 0.1 Pa has shown no significant deviation of the accommodation coefficient over the full range of measurement and over a period of time of 18 months.

Keywords: Static expansion, pressure, vacuum

1. Introduction

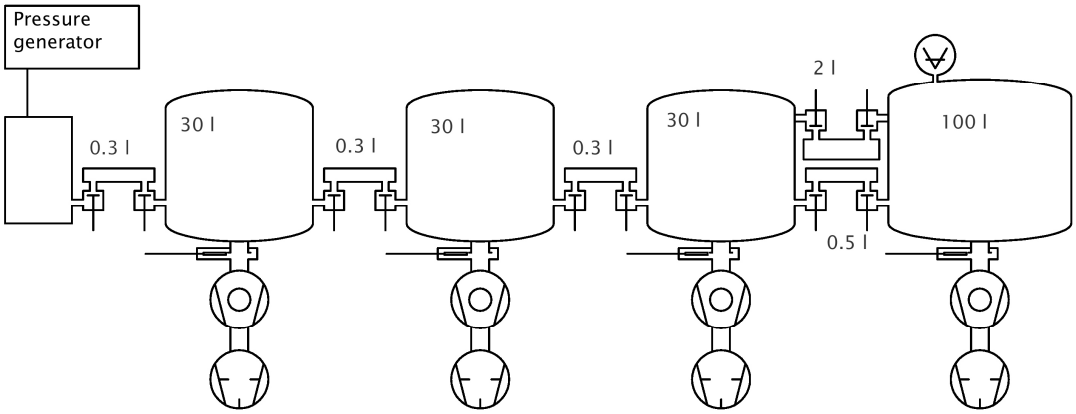
Static expansion systems have been used routinely for more than 30 years as a primary definition of low pressure [1-4]. They have a very large dynamic range that allows to cover a large pressure range that otherwise would need a digital piston gauge and a dynamic expansion system. The use of a static expansion system may seem complicated, with a lot of valve manipulation needed to achieve the final pressure. The complexity can be alleviated by a computer controlled automatic system. The characterisation of the key parameters of a static expansion system is easily achieved using standard measurement techniques usually available in a pressure calibration laboratory.

2. Measurement System

The static expansion system of METAS is a cascade of four static expansion stages. Each expansion stage is composed of a pair of chambers: a small chamber named the introduction chamber and a large chamber named the expansion chamber, whose volumetric ratio defines the expansion ratio. The three first expansion stages have an expansion ratio of 100 and the expansion chambers have a volume of 30 litres. The last expansion step has two introduction chambers with different volumes, allowing a selection between an expansion ratio of 50 or 200. The last expansion chamber has a volume of 100 litres and the maximum expansion ratio is 2·108.

The system is built according to ultra high vacuum (UHV) standards using metal gaskets at each flange. The valves have a polymer seal allowing a simpler actuation mechanism than a metal seal and a higher number of actuations before servicing. All the valves are of the same type and are mounted symmetrically, the seals oriented toward the introduction chamber. This way, any increase of pressure in the introduction chamber due to the deformation of the seal should be compensated at the opening of the next valve. Valves are actuated by compressed air injected from a remote cabinet in order to avoid any thermal dissipation close to the vacuum chambers. The valves open quickly but need about ten seconds to close, leaving time for pressure equalisation on both sides of the seal plate.

Fig1: Schematic view of the static expansion system of METAS



All the system is remotely controlled allowing automatic measurement cycles.

The rotary pumps are in a separate room to avoid any vibration or thermal load on the system. The room is air conditioned at 22°C with a temperature stability of 0.2 °C over 24 hours.

The two last expansion stages can be baked up to 150 °C. The residual pressure in the last expansion chamber after bakeout is as low as 4·10-8 Pa.

3. Generation Of The Initial Pressure

The initial pressure is generated using a digital pressure generator PPC3 from DHI with a range 1 kPa – 110 kPa. The same pressure generator is used for the volume ratio measurement and for the generation of the start pressure for the static expansion. The pressure sensor of the PPC3 is calibrated against a pressure balance above 7 kPa and against a digital piston manometer between 1 kPa and 7 kPa.

Before each expansion, the pressure generator stabilises the pressure at the value calculated as initial pressure for a few minutes. Then the regulation is stopped and the eventual drift of pressure is an indication of a thermal drift. The pressure increases when the valve of the first introduction chamber is closed because this implies a reduction of the volume containing the gas. The pressure

read after the closing of the valve is considered as the initial pressure that has been generated in the first introduction chamber.

4. Measurement Of The Temperature

The temperature of the chambers during the expansion process is an important influence factor that needs to be monitored precisely. A set of semiconductor negative thermal coefficient sensors (NTC) is placed on the outer wall of the chambers (6 NTC for each expansion chamber and 2 NTC for each introduction chamber). The NTC sensors have been calibrated by the temperature laboratory of METAS. Their uncertainty is 0.02 °C provided they are removed during the bakeout of the chambers.

5. Determination Of The Volume Ratio

The volume ratio of a static expansion system is a key parameter for the traceability of the generated pressure and is defined by the following equation,

$$\phi = \frac{V + v}{v}$$,

where Φ is the volume ratio, V is the sum of the volume of the expansion chamber and the volume of the next introduction chamber if there is a next expansion stage, v is the volume of the introduction chamber.

Table 1: Sources of uncertainty in the determination of the volume ratio

Input value	**Symbol**	**Typical value**	**Standard uncertainty**
Initial pressure	P_0	100 kPa	7 Pa
Final pressure	P_n	30 kPa	5 Pa
Initial temperature	T_0	295 K	0.1 K
Final temperature	T_n	295 K	0.1 K
Virial coefficient	$\beta(T)$	$5.0 \cdot 10^{-6}$ m^3/mol	$5.0 \cdot 10^{-7}$ m^3/mol
Temperature offset between the large and the small chamber	ΔT	1 K	0.1 K

The volume ratio of the system has been characterised using two different techniques, the well known accumulation technique developed at NPL at first for isothermal conditions [5] and extended later for non isothermal conditions [6] and

the depletion technique introduced by METAS [7]. Both techniques have been used at one year interval and the results assess the stability of the system and the reproducibility of the measurement.

Volume ratios obtained with the gas accumulation and gas depletion techniques are given in table 2 and show the good agreement between the two techniques. Additional work has also been made using argon to asses the hypothesis about thermalisation of the gas and the value of the virial coefficient. Once more the values obtained with argon are in agreement with the values obtained with nitrogen.

	Gas accumulation		Gas depletion		E_n factor (k=1)
	Φ	U(Φ) k=1	Φ	U(Φ) k=1	
Stage 1	98.435	0.060	98.415	0.050	0.26
Stage 2	97.996	0.060	97.996	0.050	0.01
Stage 3 and small introduction chamber 4	99.620	0.060	99.615	0.050	0.06
Stage 3 and large introduction chamber4	104.395	0.060	104.385	0.050	0.13
Stage 4 with small introduction chamber	202.716	0.120	202.706	0.100	0.06
Stage 4 with large introduction chamber	52.186	0.030	52.176	0.025	0.26

Table 2 : Values of the expansion ratio and uncertainty using the gas accumulation and gas depletion technique.

6. Determination Of The Volume Of The Chambers

The volume of the sensor to be calibrated or the volume of the barometer used for the determination of the volume ratio has an effect on the volume ratio of an expansion stage. This effect can be corrected if the volume of the chamber is

known with an uncertainty of a few percent. The volume of the expansion chamber was determined by measuring the change of the volume ratio when a chamber of known volume is connected to the expansion chamber. The volume of the additional chamber was determined by gravimetry using deionised water.

The volume of the expansion chamber is given by:

$$V = B\frac{\phi_0 - 1}{\phi_1 - \phi_0},$$

where Φ0 and Φ1 are respectively the volume ratio without and with the additional chamber and B is the volume of the additional chamber.

This indirect technique was chosen because it does not contaminate the chamber and measures the volume under realistic working condition.

7. Uncertainty Of The Volume Ratio Determination

The volume ratio of each expansion stage was determined using the accumulation technique as well as the depletion technique. The uncertainty contribution of the influence factors is summarised on table 1 while the best uncertainty on the volume ratio is given in table 2. The uncertainty of the volume ratio is strongly dependant on the number of expansion steps and the best value is reached after 30 expansions for the additive technique and after 200 expansions for the depletive technique (Φ=100). However the best uncertainty achieved is similar with both techniques. [7]

8. Generation Of The Low Pressure

Most calibrations are made using nitrogen because this gas has a relatively low virial coefficient, produces low adsorption on the walls and is easily available. A digital pressure regulator PPC3 working at levels up to 100 kPa is used for the generation of the starting pressure. In the case of a cascade of expansion stages with similar expansion ratio (100 in the case of the first three stages), the start pressure must have a dynamic range similar to the value of the expansion ratio. The choice of an expansion ratio of 50 or 200 at the last expansion step allows to generate any pressure ranging from 2·10-5 Pa to 2000 Pa with the start pressure kept between 4 kPa and 100 kPa. This gives a dynamic range for the initial pressure of 25 and diminishes the contribution of the relative uncertainty of the pressure generator to the final uncertainty. At each expansion stage a pause of a few minutes is made in order to allow the gas to thermalise with the wall of the chamber [7].

9. Uncertainty Of The Generated Pressure

The generated pressure is affected by several sources of uncertainties that are listed on table 3 in the general case of one expansion step starting from a pressure generated by the digital pressure generator. The uncertainty of the following expansion steps is calculated iteratively on the same principle with the following changes:

- The starting pressure and its uncertainty are given by the previous expansion.
- The uncertainty of the virial coefficient is relevant only for the first expansion and can be neglected for the expansions starting from pressures lower than 4 kPa.
- The contribution of the outgassing is lower for the next stages because of one or more of the following reasons: larger expansion chamber, bakeout prior to measurement, less exposition to high pressure.

Table 3: Example of uncertainty calculation for one stage of expansion.

Source of uncertainty	**Expected value**		**Uncertainty on the input value**		**Sensitivity coefficient**		**Contribution to the uncertainty**	
Pressure in the input chamber	10000	Pa	3	Pa	0.01		0.030	Pa
Volume ratio	100.00		0.05		-1.0	Pa	-0.050	Pa
Temperature of the introduction chamber	295.0	K	0.1	K	0.34	Pa/K	0.034	Pa
Temperature of the expansion chamber	295.00	K	0.1	K	-0.34	Pa/K	-0.034	Pa
Virial coefficient	$5.50 \cdot 10^{-6}$	m^3/mole	$5.50 \cdot 10^{-6}$	m3/mole	$-4.08 \cdot 10^{4}$	PaMole/m^3	-0.022	Pa
Outgassing	0	Pa	$4.0 \cdot 10^{-5}$	Pa	1.0		$4.0 \cdot 10^{-5}$	Pa
Final pressure	100	Pa						
Combined uncertainty						k=1	0.079	Pa
Expanded uncertainty						k=2	0.160	Pa

The relative uncertainty on the generated pressure increases with the number of expansion stages. Table 4 gives the maximal value of the uncertainty obtained after detailed calculation.

Pressure range	Uncertainty	Ranging	
	(k=2)	from	to
10 – 1000 Pa	0.003·p	0.03 Pa	3 Pa
0.1 – 10 Pa	0.004·p	$4 \cdot 10^{-4}$ Pa	$4 \cdot 10^{-2}$ Pa
0.001 – 0.1 Pa	0.006·p	$6 \cdot 10^{-6}$ Pa	$6 \cdot 10^{-4}$ Pa
$2 \cdot 10^{-4}$ – 10^{-3} Pa	0.01·p	$2 \cdot 10^{-6}$ Pa	$1 \cdot 10^{-5}$ Pa
10^{-5} – $2 \cdot 10^{-4}$ Pa	$2 \cdot 10^{-6}$ Pa	$2 \cdot 10^{-6}$ Pa	$2 \cdot 10^{-6}$ Pa

Table 4: Uncertainty of the static expansion system over the whole pressure range

10. Repeatability

Two spinning rotating gauges (SRG), and two different sensor electronics have been used to demonstrate the stability of the system using argon and nitrogen as gas,. on a time span of 18 months. Long term stability of the accommodation factor of the two SRG is summarised in table 5. The two sensors exhibit stability better than 0.4 % demonstrating a good reproducibility of the calibration chain (sensor and static expansion system). The accommodation coefficients are also equivalent when measured with two different sensor electronics and with two different gases.

Date	Sensor A: SN 191693			Sensor B: SN 191694		
	Readout Electronic	Accom. factor	Std. Dev.	Readout Electronic	Accom. factor	Std. Dev.
19.10.05	SRG2-CE	1.0000	0.0026	SRG2	1.0000	0.0042
15.03.06	SRG2-CE	0.9983	0.0026	SRG2	1.0000	0.0068
17.03.06	SRG2	0.9987	0.0057	SRG2-CE	0.9974	0.0031
29.01.07	SRG2-CE	0.9976	0.0051			

02.04.07	SRG2	1.0012	0.0072

Table 5: Accommodation factors of two SRG sensors with two different readout electronics over a time span of 18 months.

The value of the accommodation coefficient is supposed to be constant over the whole range of pressure for which the flow is molecular. Laminar flow effects are no longer negligible for pressure above 0.1 Pa while the sensitivity of the SRG becomes too small below 10-4 Pa. The normalised accommodation factor versus pressure is shown in Fig. 2.

A comparison was been made with Mittatekniikan Keskus (MIKES), the national metrological institute of Finland, using an MKS Baratron with 130 Pa of range as transfer standard. MIKES uses an DHI FPG system to characterise this pressure range. The comparison has shown the equivalence of the values of the two instruments.

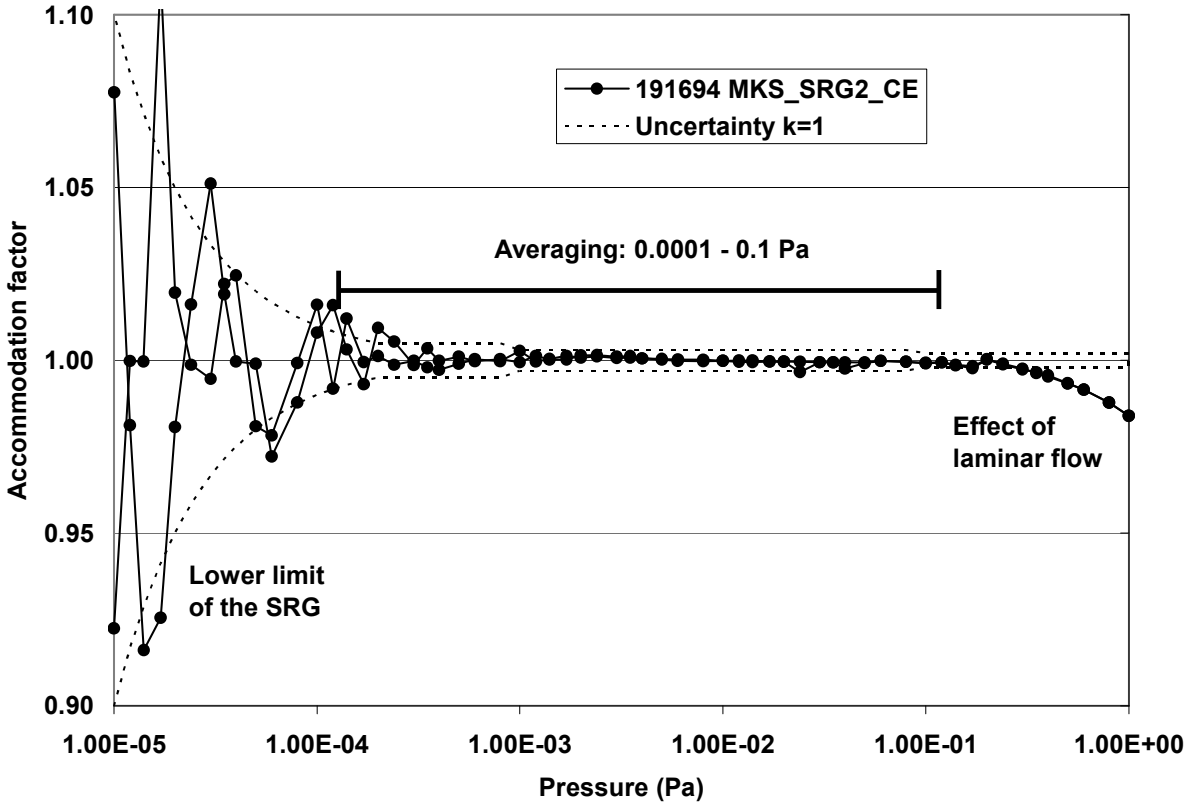


Fig.2: Normalised accommodation factor of a spinning rotating gauge versus pressure. The stability of the values ranging from 10-4 Pa to 0.1 Pa shows the consistency of the measurement system. The decrease above 0.1 Pa comes from the viscous effects and the discrepancy under 10-4 Pa is due to the lower limit of the sensor. The dotted line shows the uncertainty of the measurement; the continuous line shows the measured values

11. Conclusion

The static expansion system of METAS has been characterised using complementary techniques providing similar results. The measurement using a SRG shows that the accommodation coefficient is stable over all the range measurable under molecular flow. A comparison with a digital piston system has demonstrated the equivalence of the generated pressure from 10 to 100 Pa.

The new system will be used as a pilot instrument for an international comparison under the auspice of Euramet.

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