

THE APPLICATION OF CRITICAL NOZZLES IN SERIES FOR THE DETERMINATION OF SMALL FLOW RATES AND THE GENERATION OF GAS MIXTURES

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Abstract:

Because of the low uncertainty and very good long term stability the application of critical nozzles became very important especially for test rigs. The majority of nozzles are used in the so-called “suck mode” using atmospheric air as test medium. In order to use nozzles for the generation of different flow rates as well as to generate gas flows with different kinds of gases the application with increased input pressure became more important. An other field of extended application is the usage for small flow rates. Nozzles which have a shape in accordance to ISO 9300 are available with throat diameters down to 80 μm . Below this value the shapes have to be simplified. Even for simplified shapes the mechanical manufacturing of nozzles allows at the time being throat diameters of $d = 15 \mu\text{m}$.

In order to extend the flow rates to values smaller than the flow rates possible with a single nozzle the use of two nozzles in series is a possible approach. The paper shows that the properties of nozzles, especially the very high reproducibility, is the main precaution for this solution.

The paper will describe an arrangement of two micro nozzles in series. The throat diameters are $d_1 = 15 \mu\text{m}$ and $d_2 = 25 \mu\text{m}$, respectively. The generated flow rates are compared with LFE measurements. The main source of uncertainty is the reproducibility of the pressure transducers. A nozzle bridge and its potential to decrease this source of uncertainty will be proposed and discussed.

Gas mixtures for the calibration of sensors are usually manufactured by gravimetric methods. The so called dynamic generation is based on the mixture of two or more flows of gas components with known flow rates. The arrangement of two nozzles in series is an interesting approach for two-component mixtures. The flow rate of the main component is given by the flow rate of the upstream nozzle. The second component is inputted at the connection point of the two nozzles. The amount of this additional flow can be determined by the pressure change at the inlet of the second nozzle. The application of two nozzles in series for generating nitrogen-methane mixtures will be described using a third nozzle for calibrating the mixing unit. In order to get experience with this kind of application the generated mixtures were compared with measurement results of a commercially available gas analyser. The results show the feasibility of this kind of application.

Keywords: critical nozzles, flow rate determination, gas mixture, micro nozzle

1. Introduction

The application of critical nozzles is well-known for the generation of constant flow rates [1]. Nozzles are easy to handle and show a very good long term stability which makes them very popular for the application at test rigs. In the last years the usage for different gases as well as for higher input pressures became a topic of interest [2].

In the last years, the extension of the application range of sonic nozzles to smaller flow rates and the application for different kinds of gases were areas of research and development at PTB.

The international standard ISO 9300 covers two types of Venturi shapes for critical nozzles. These types allow the usage of nozzles under critical conditions also for large back pressure ratios p_{back}/p_1 up to 0,85. Also for throat diameters below the diameter range covered by the standard these kinds of nozzles show reliable results but the usable back pressure ratio is much lower than for large nozzles. The lowest possible throat diameter is limited by the production technology. Nozzles with a shape in accordance to ISO 9300 are currently available for flow rates down to $Q_V = 100 \text{ cm}^3/\text{min}$.

In order to reach smaller flow rates, mechanically manufactured nozzles with simplified shapes were investigated by different groups. At PTB, mechanically drilled micro nozzles were investigated [3].

The manufacturing of nozzles on silicon chips is an approach to use nozzles for smaller flow rates as well as to reduce production costs for larger numbers [6].

For the measurement of flow rates down to $Q_V = 0,1 \text{ cm}^3/\text{min}$ laminar flow elements (LFE) are available. Capillaries with diameters of $d = 10 \text{ }\mu\text{m}$ were used as transfer standards for such devices. The capillaries work for these applications as LFE but with very large difference pressures [5]. In the case of LFE inlet and output pressures have to be determined, whereas for critical nozzles, only the inlet pressure is important. Also the temperature is an important source of uncertainty.

The use of nozzles in series is known for the calibration of one nozzle by an other one with known parameters. In order to provide a transfer standard with low uncertainty and increased reliability a device with two interchangeable nozzles was realised and applied for key comparisons [7].

The paper describes the idea to use nozzles in series for the determination of flow rates smaller than flows achievable with one nozzle at comparable input conditions. If a flow is inputted at the connection point, the second nozzle will allow to determine the sum of flow rates. In principle this approach is possible by different kinds of devices but especially nozzles are very well suited. Only two pressures have to be measured, the temperature has a comparable low influence, and very compact devices are possible. A realisation on chip is easily possible.

A further field of application of critical nozzles is the generation of gas mixtures by mixing the known flow rates generated by flow controllers for each component. First investigations show that critical nozzles in series are suited for this application. An important advantage of nozzles is the possibility of calibrating a nozzle with one kind of gas and using it for other gases with low uncertainty only by replacing the thermodynamic gas parameters in the calculation formula.

2. Description of the nozzle behaviour

The flow rate of a nozzle is given by the geometrical dimensions, namely by the throat area A , the used gas (R_1 specific gas constant, c_1^* critical flow factor), the discharge coefficient c_D (which is a function of the Reynolds number Re), the pressure p_1 , and the temperature T_1 at the inlet of the nozzle.

$$Q_{m1} = A_1 \cdot c_{D1} \cdot c_1^* \frac{p_1}{\sqrt{R_1 \cdot T_1}} \quad (1)$$

Eq. (1) is valid only if the nozzle is used in the critical mode (choked) which requires that the critical pressure ratio is reached. The pressure p_1 and the temperature T_1 have to be measured under stagnation conditions [2]. Aschenbrenner has developed a simplification of equ. (1) which

is useful for the application of nozzles with humid air at atmospheric pressure [1]. For the application on different gases eq. (1) may be written as follows:

$$Q_m = A_{eff} \cdot \psi \quad (2)$$

The effective area A_{eff} depends on the thickness of the boundary layer especially in the throat. Mickan at al. proposed a set of equations (2) ... (4) for the effective area which is applicable for laminar boundary conditions, that means for Re numbers (calculated using the throat diameter as the characteristic length) below $Re = 10^6$.

The Re - dependency of the discharge coefficient C_D leads to the consideration of the gas viscosity μ in eq. (3). The real gas behaviour is incorporated by the critical flow factor c^* .

$$A_{eff} = a + b \cdot x \quad (3)$$

$$x = \sqrt{\frac{\mu}{\psi}} \quad (4)$$

$$\psi = c^* \cdot \frac{p_D}{\sqrt{R \cdot T_D}} \quad (5)$$

$$\psi = c^* \cdot \frac{p_D}{\sqrt{R \cdot T_D}} = c^* \cdot \sqrt{\frac{M}{8314.41}} \frac{p_D}{\sqrt{T_D}} \quad (5a)$$

By introducing the molar mass M eq. (5) can be written as shown in (5a).

In order to reach low flow rates micro nozzles with a simplified shape are used at PTB. Fig. 1 shows the outline of this kind of nozzle which are manufactured by a mechanical process.

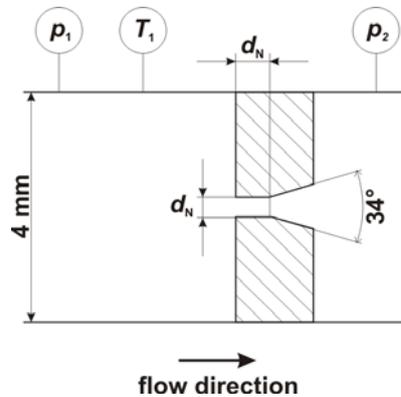


Fig. 1: Outline of micro nozzles used by PTB

Fig. 2 shows a nozzle with a diameter of $d = 20 \mu\text{m}$ photographed by an electron microscope. During application the flow was directed from the cylindrical part to the conical one.

As described in [3] this direction of operation leads to a discharge coefficient which is nearly independent from the back pressure ratio (below a certain value of this ratio) as it is usual for nozzles with a shape described in ISO 9300.

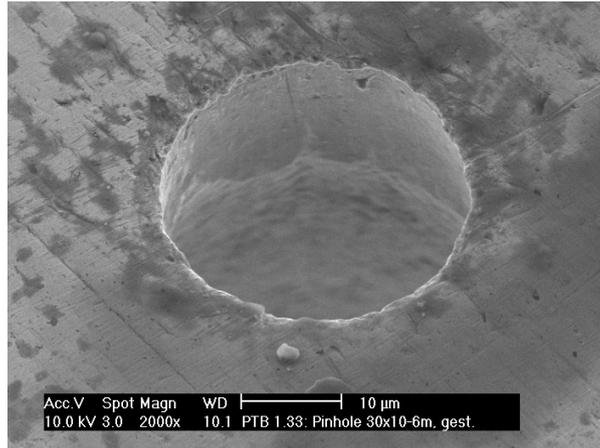


Fig. 2: Picture of a micro nozzle photographed by an electron microscope.

The picture shows a circular shape and sharp edges, but currently this kind of manufacturing does not allow throat diameters below $d = 15 \mu\text{m}$.

The calibration results of micro nozzles show a behaviour which can be described by the parameters a and b (see eq. 3 to 5) but a correction in respect to the ratio between input and output pressure becomes necessary for the lowest diameters. This behaviour has to be investigated.

3. Usage of 2 nozzles in series

If a second nozzle is connected in series downstream to a first one, then both nozzles will work in the critical mode if the total pressure ratio and the ratio of the throat areas A_1/A_2 are chosen appropriately. Such an arrangement allows the calibration of one nozzle by another one. Also the application of 2 nozzles may be advantageous for comparison measurements because two independent sets of measurement results for temperature and pressure are available for the flow rate determination and uncertainty analysis.

$$r_p = \frac{p_2}{p_1} = \frac{A_1}{A_2} \frac{c_{D1}}{c_{D2}} \frac{c_1^*}{c_2^*} \sqrt{\frac{R_2 \cdot T_2}{R_1 \cdot T_1}} \quad (7)$$

If the gas flowing through both nozzles is the same, then R_1 and R_2 are equal. If the flow rates are very small then the influence of the Joule-Thompson effect on the temperature is negligible in comparison to the thermal properties of the piping. It is possible to keep both temperatures constant and equal by an appropriate design and isolation of the piping, or by using the nozzles in a temperature-stabilised water bath, for example.

If the pressure ratio p_1/p_2 is measured for input pressures up to $p_1 = 3,5 \text{ bar}$, the ratio of the critical flow functions is nearly constant. Hence the result represents the ratio of the discharge coefficients.

Fig.3 shows this ratio for a micro nozzle of $d_{N1} = 15 \mu\text{m}$ throat diameter in series with a nozzle of $d_{N2} = 25 \mu\text{m}$.

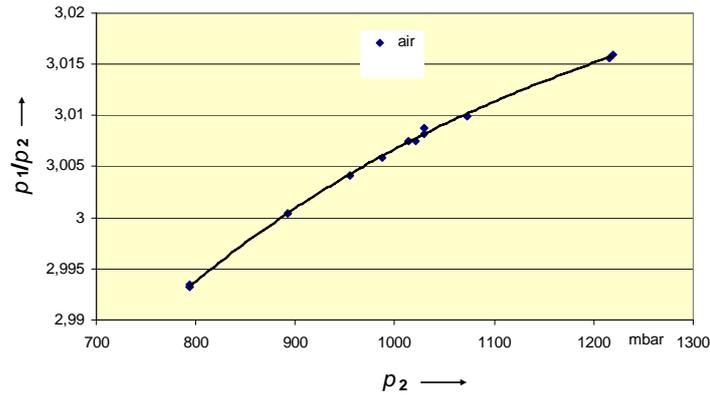


Fig. 3: Input pressure ratio for 2 micro nozzles in series

The Re - numbers are between $Re_{D2} = 350$ and $Re_{D2} = 520$ for the downstream nozzle ($d_{N2} = 25 \mu\text{m}$) and between $Re_{D1} = 580$ and $Re_{D1} = 870$ for the upstream nozzle, respectively.

3.1 Application of two nozzles in series with air for a flow rate generation

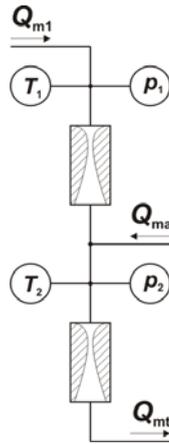
In order to measure flow rates lower than the flow rate defined by the throat area of a nozzle the use of two nozzles is a possible approach. If downstream of the first nozzle, that means at the connection point of both nozzles, an additional flow Q_{ma} will be fed in, then the pressure at this point will change. The flow rate inserted at the connection point can be derived as the difference of the flow rates determined by both nozzles. In principle it is necessary to measure both temperatures but the temperature T_1 and T_2 will not differ if the gas temperature of the additional flow is nearly the same as the temperature at T_1 , and if the energy input by Joule-Thompson effect is negligible.

$$Q_{ma} = A_2 c_{D2} c_2^* \frac{p_2}{\sqrt{R \cdot T_2}} - A_1 c_{D1} c_1^* \frac{p_1}{\sqrt{R \cdot T_2}} \quad (8)$$

For small flow rates and proper design of the nozzle holders, the assumption of equal temperatures for both nozzles is reasonable. If the same kind of gas flows through both nozzles, the pressure ratio r_p defined in eq. (7) is only a function of pressure. Eq. (8) may be simplified:

$$Q_{ma} = [p_2 - p_1 r_p(p_1)] \frac{c_{D2} c_2^* A_2}{\sqrt{R \cdot T}} \quad (9)$$

Eq. (9) shows the importance of the pressure determination for the uncertainty of the arrangement. For the zero point adjustment the flow may be switched of. Then the ratio r_p may be adjusted. The additional flow Q_{ma} is determined correctly as long as both nozzles are operating in the critical mode. By using a vacuum pump for the generation of a sufficiently low output pressure p_3 of the second nozzle and a pressure regulator for the stabilisation of p_1 it is possible to adjust p_2 near to atmospheric conditions. This may be advantageous for the generation of constant flows through a MUT.



*Fig. 4: Arrangement of 2 nozzle in series
(if no additional flow Q_{ma} is inserted at the connection point the arrangement allows the calibration of a nozzle by another nozzle with known parameters)*

Fig. 5 shows the result of an LFE calibration by the arrangement in accordance to Fig. 4. At the lowest flow rate ($Q_{ma} = 0,0017$ mg/s), the influence of the zero point drift of the difference pressure determination leads to larger deviations. With increasing flow rates the deviation shifts to positive values. Probably, this is caused by the increase of the pressure p_2 if the flow rate is increasing. This means, the back pressure ratio has an influence on the flow rate Q_{m1} generated by the upstream nozzle. Numerical investigations show a movement of the area characterised by a Mach number of $M = 1$ in the outlet of the nozzle [3] which causes probably this phenomenon.

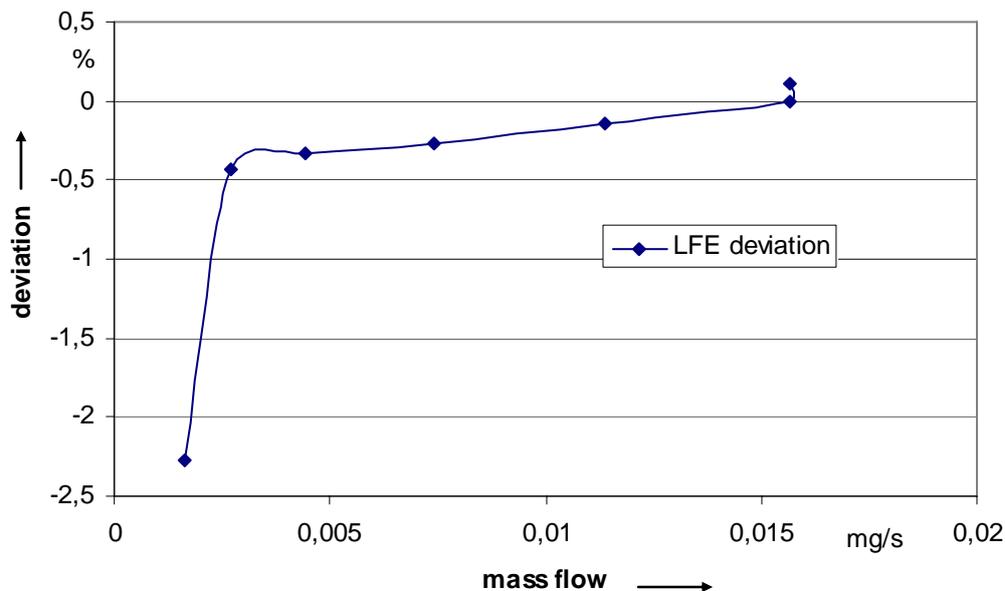


Fig. 5: Result of an LFE calibration by 2 nozzle in series

3.1 Application of a nozzle bridge for the generation of a flow rate

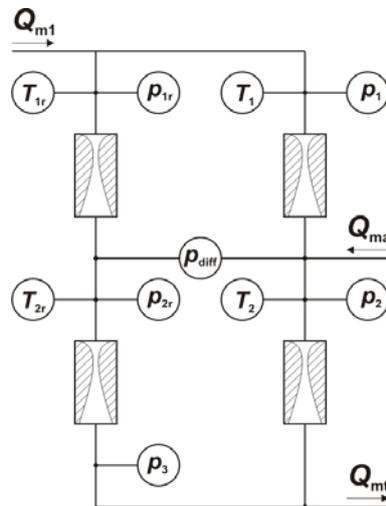


Fig. 6: Application of a nozzle bridge for the determination of flows

The use of nozzles in a bridge (Fig. 6) allows it to monitor the pressure at the connection point of to nozzles series in difference to the bridge wing where an additional flow is inserted. This allows the use of difference pressure transducers in combination with an absolute pressure transducer. The pressure range of the difference pressure transducer can be optimised in respect to the required flow rate range of the flow inserted at the connection point. As described in chapter 5 such an arrangement will allow to reduce the uncertainty.

4 The use of nozzles in series for gas mixing

Nozzles in series may be used also for the generation of gas mixtures if another kind of gas is used as the additional flow Q_{ma} . In principle the dependency of the critical flow factor c_m^* as a function of the gas composition is needed for this application. On the other hand, the arrangement may be used for the determination of c_m^* itself, provided Q_{ma} is measured separately.

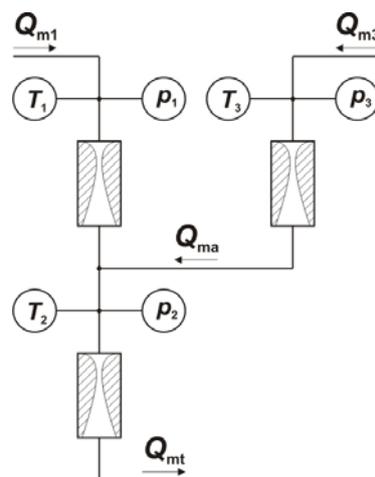


Fig. 7: Application of nozzles in series for gas mixing

Fig. 7 shows the test setup. The main component of the gas mixture was nitrogen which flew through nozzle 1 and 2. At the outlet of nozzle 2 a slightly increased overpressure against atmospheric pressure was applied, given by the pressure drop of the connected infrared detector.

The flow through the infrared methane detector was split from the total flow Q_{mt} at the outlet of nozzle 2. The second component (methane) was mixed in via nozzle 3. Nozzle 3 was a micro nozzle. In table 1 the parameters of the nozzles are listed.

During the first step the pressure change at the connection point of nozzle 1 and 2 was calibrated by the flow rate determined by nozzle 3. In order to match the range of the nozzle mixer to the measuring range of the infrared detector $x_{\text{CH}_4} = 0 \dots 1800$ ppm the flow rate generated by nozzle 3 was selected to generate a concentration of 0,36 mol %. Then the pressure change at the connection point was $\Delta p_2 = 7$ mbar at a $p_2 = 2100$ mbar. After calibration the pressure p_3 was reduced to reach the wished concentration. Then nozzle 3 was not under critical condition anymore but it was usable as a flow resistor.

According to their specifications, the pressure transducers used for the determination of p_1 and p_2 have an uncertainty of $U = 10^{-4} p_{\text{max}} = 0,7$ mbar. Because of the low values of Δp_2 during the measurements the results are inside the estimated uncertainty.

Table 1 Nozzle parameter for mixing methane into nitrogen

parameter	nozzle 1	nozzle 2	nozzle 3
diameter	0,5 mm	1 mm	20 μm
shape	ISO 9300 torodial	ISO 9300 torodial	in acc. to Fig. 1
a	2,1080E-07	7,79705E-7	6,6040E-10
b	-3,363E-05	-1,94432E-5	-1,4241E-07
$Q_{20,\text{tr},1000}$	145 l/h	537 l/h	0,43 l/h

Table 2 Gas data for mixing methane into nitrogen

value	for nitrogen N_2	for methane CH_4	unit
critical flow factor c^* (5 bar ... 7bar)	$0,6863 \pm 0,0004$	$0,6736 \pm 0,0012$	--
critical flow factor c^* (3 bar ... 5bar)	$0,6858 \pm 0,0003$	$0,6724 \pm 0,0011$	--
molar mass M	28,0154	16,043	g/mol
specific gas constant R	296,78	518,26	kg/kmol
dynamic viscosity μ	$(17,6 \pm 0,23) \cdot 10^{-6}$	$(10,05 \pm 0,3) \cdot 10^{-6}$	Pa·s

The values for c^* and μ are valid between 15 °C and 25 °C.

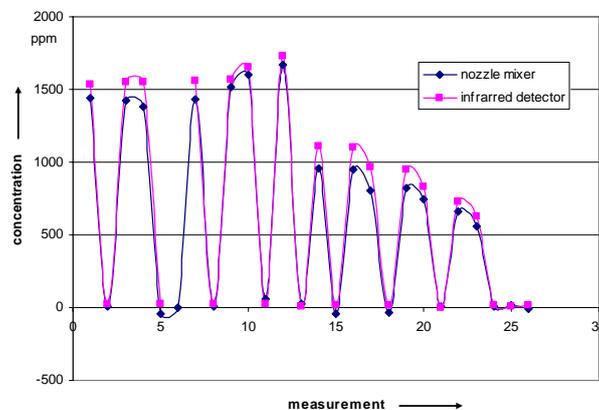


Fig 8: Comparison of the concentration determination by the nozzle mixer with an infrared detector

First comparison measurements of the nozzle mixer with a commercially available infrared detector are summarised in Fig. 8.

If the concentration of the second component is higher, the nozzle mixer will provide lower uncertainties. The usable pressure range of p_2 depends mainly on the critical back pressure range of nozzle 1 and 2. A concentration range of the second component $x_{\text{CH}_4} = 2\% \dots 20\%$ is recommended. In order to reach lower concentration ranges a second dilution may be a solution.

5. Considerations on uncertainty

In the case of nozzles in series the uncertainty for the flow rate generation will be influenced by the opportunity to set Q_{ma} to zero by switching of the additional flow rate at the connection point. This means that the discharge coefficient of one nozzle may be adjusted. Because of the long term stability of the nozzle dimensions, the uncertainty caused by drifts is small. The uncertainty of the base nozzle influences the result with a sensitivity of $c = 1$. As mentioned above, a good temperature stability may be reached by a proper design. Further the sensitivity of the temperature is relatively low because of the square root in the equation. Therefore, the uncertainty of the pressure determination is one of the main sources of uncertainty. Because of the low flow rates, the leakage test requires considerable efforts. A main problem is also the time constant for changes of the pressure p_2 at the inlet of nozzle 2 (connection point). During the experiments the total volume at the connection point V_{12} was around $V_{12} = 1\text{cm}^3$. After a 10% change of the input pressure at nozzle 1 a stabilisation time of 6 min was necessary to reach stable pressure ratios r_p . By an improved design this volume can be reduced by a factor of ten.

With the definition of r_p in eq. (7), the eq. (9) finally results in:

$$Q_{\text{ma}} = \left(1 - \frac{p_2}{p_{2,0}}\right) \cdot Q_{m1} = \frac{\Delta p_2}{p_{2,0}} Q_{m1} \quad (10)$$

with p_2 – pressure at nozzle #2, actually measured;
 $p_{2,0}$ – pressure at nozzle #2 when $Q_{\text{ma}} = 0$;
 Q_{m1} – mass flow rate through the upstream nozzle #1;
 $\Delta p_2 = p_{2,0} - p_2$

By application of the rules for the propagation of uncertainty given by the GUM we get:

$$u_{\text{rel}}^2(Q_{\text{ma}}) = u_{\text{rel}}^2(Q_{m1}) + c_1^2 \cdot u_{\text{rel}}^2(p_{2,0}) + c_2^2 \cdot u_{\text{rel}}^2(p_2) - 2 \cdot c_1 \cdot c_2 \cdot \text{cov}(p_2, p_{2,0}) \quad (11)$$

with the sensitivity coefficients

$$c_1 = c_2 = \frac{p_2}{\Delta p_2} \quad (12)$$

The (relative) uncertainty of the pressure measurement can be split in two parts: a stochastic part $u_{\text{rel,repeat}}$ which represents the repeatability of the measurement and a part $u_{\text{rel,static}}$ covering the uncertainty impact of influence factors which are constant in time. This shall be expressed as:

$$u_{\text{rel}}^2(p_2) = u_{\text{rel,repeat}}^2(p_2) + u_{\text{rel,static}}^2(p_2) \quad (13)$$

[the expression for $p_{2,0}$ is similar and it is assumed that $u_{\text{rel}}(p_2) = u_{\text{rel}}(p_{2,0})$].

With the conventional relation of the covariance with the correlation coefficient r and its estimation by comparing the repeatability with the total uncertainty we get:

$$\begin{aligned} \text{cov}(p_2, p_{2,0}) &= r \cdot u_{rel}^2(p_2); \quad r = 1 - \frac{u_{rel,repeat}(p_2)}{u_{rel}(p_2)} \\ \Rightarrow \text{cov}(p_2, p_{2,0}) &= u_{rel,static}(p_2) \end{aligned} \quad (14)$$

This expression allows to pool the uncertainty for Q_{ma} mentioned in eq. (10) as follows:

$$u_{rel}^2(Q_{ma}) = u_{rel}^2(Q_{m1}) + 2 \left(\frac{p_2}{\Delta p_2} \right)^2 \cdot u_{rel,repeat}^2(p_2) \quad (15)$$

Eq. (15) shows that beside the calibration uncertainty of the nozzle flow rate $u_{rel}(Q_{m1})$ the main source of uncertainty is given by the short term stability of the pressure transducers. An estimation of this figure is possible by investigating the repeatability of the pressure ratio r_p .

6. Conclusions

Taking into account future improvements in the state of the art of producing small nozzles, the application of nozzles in series will allow to

- realise gas measuring devices with a flow rate range which is 10 times smaller than the base flow rate of the nozzles used in series
- generate two-component gas mixtures in a concentration range up to 20 %

The technique needs further investigation, in particular the influence of the back pressure ratio on the flow rate of micro nozzles has to be considered. Nozzles in series are an alternative to capillaries for the generation of flow rates below $Q = 1 \text{ cm}^3/\text{min}$. If nozzles are used in a bridge, a differential pressure transducer may be used instead of the second absolute pressure transducer in order to achieve lower uncertainties. The arrangement allows even smaller concentrations (below 2 %) if a further dilution is applied.

7. Symbols

Table 3: Symbols

symbol	description	unit
A_{eff}	effective throat area	m^2
a	$A_{\text{eff}} (\text{Re} \Rightarrow \infty)$	m^2
b	coefficient for Re- behavior A_{eff}	$\text{m}^{1.5}$
c	sensitivity coefficient of uncertainty influences	--
x_{CH_4}	concentration	mol/mol
μ	dynamic viscosity	$\text{Pa}\cdot\text{s}$
ψ	mass flow density in the nozzle throat	$\text{kg}/\text{m}^2/\text{s}$
c^*	critical flow factor	--
p	pressure	bar

p_D	nozzle input pressure	Pa
T_D	nozzle input temperature	K
M	molar mass	kg/kmol
r	correlation coefficient	--
r_p	pressure ratio	--
R	specific gas constant = 8314.41/M	kJ/(kg·K)
Q_m	mass flow rate	kg/s
$Q_{20, tr, 1000}$	volume flow rate at $T=293,15$ K, $p_D=1000$ mbar, dry air	m ³ /h
u	uncertainty (expansion factor $k=1$)	
u_{rel}	relative uncertainty ($k=1$)	--
ρ	density	kg/ m ³

8. References

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