How High Accuracy is reached with Sonic Nozzles for Gas Calibrators

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Gas Calibration Needs

Gas analyser's calibration is required in many applications according legislation or quality systems management. This is the case for Air Pollution Monitoring or Continuous Emissions Monitoring devices installed in cabinets, measuring in continuous and remote locations. Measurements in trace ppm/ppb range are performed and analytical devices specifications need to be validated and corrected over the time. Rather than a single point calibration or validation, the goal is to perform a linearity validation throughout the entire measurement range.

In order to accomplish this task, a gas device should generate a range of different mixture concentrations in a very accurate and reproducible way, preferentially with automatic routines.

Two groups of mixtures generation for calibration purpose are available and described by ISO norms.

Gas Calibration Methods According the ISO

The first group is called gravimetric methods. According to the ISO 6142, a mixture is generated by cylinders weighing and the final mixture generation into a new cylinder. This method comes first in mind as mass is a primary standard and a short connection to this standard reduces uncertainties in the final result. The closer you are from a primary standard the better it is. Nevertheless field deployment of this method shows some limitations:

- only one unique concentration is available,
- some compounds cannot be stored in cylinders (formaldehyde),
- the critical stability of compounds at low concentration (SO2 in ppb range),
- high costs, when several mixtures are required

A second group of methods is described by the ISO 6145 and consist in several parts, under the general title 'Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods'. It includes: volumetric pumps, continuous syringe injection method, capillary calibration devices, critical orifices, thermal mass-flow controllers, diffusion method, saturation method, permeation method, electrochemical generation. Main benefits of dynamic methods are a better compatibility with industry requirements, mixture generation is done only when it is required and several concentrations (ranges) can be generated.

We will describe here the principle and specificities of the Part 6: sonic nozzles technology.

How do Sonic Nozzles Work?

A sonic nozzle works according the principle of critical flow (also referred to as 'choked'), an effect generated with compressible gases and a fluid dynamic conditions associated with the Venturi effect. When a flowing gas, at given conditions, passes through a restriction, such as the throat of a convergent-divergent nozzle, into a lower pressure environment, the fluid velocity increases. At initially subsonic upstream conditions, the conservation of mass principle requires the fluid velocity to increase as it flows through the smaller cross-sectional area of the restriction.

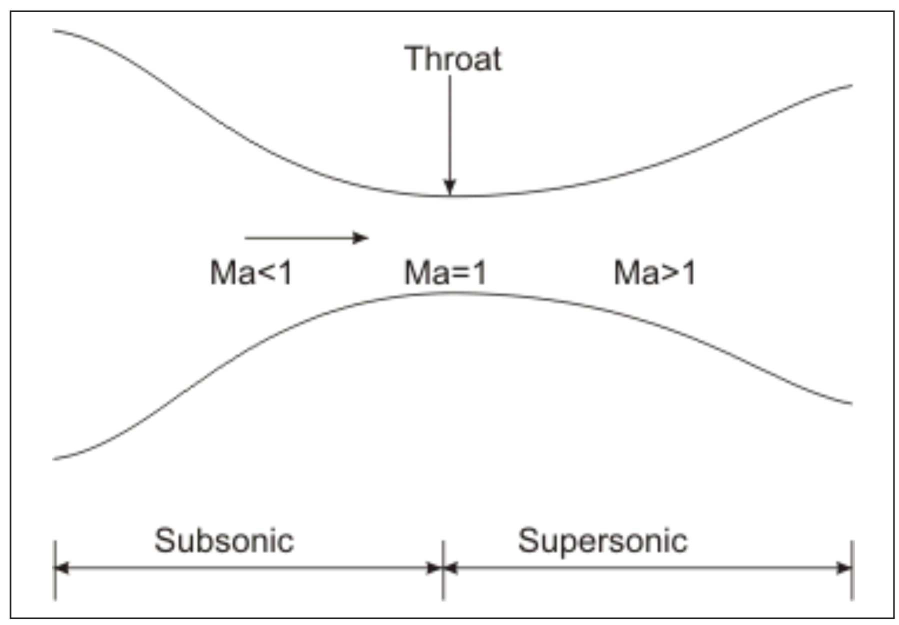


Figure 1. Gas speed across a Venturi

When the pressure ratio in-/outlet becomes higher than 2, the supersonic speed is reached into the restriction and the mass flow does not increase with a further decrease in the downstream pressure environment keeping the upstream pressure fixed, the critical flow is reached.

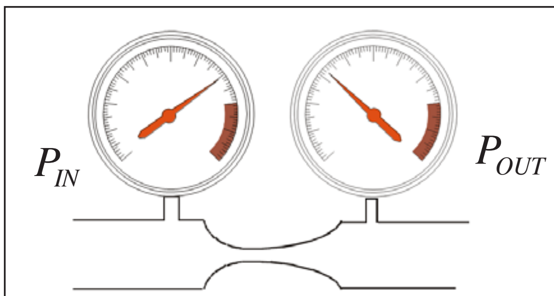
The critical flow of gases is used in many engineering applications because the mass flow rate is independent of the downstream pressure, depending only on the temperature and pressure on the upstream side of the restriction. For instance in de Laval nozzles used in rocket engines, to avoid loss of efficiency when exit pressure is lower than ambient (atmospheric); diving rebreathers, where precise constant mass flow gas addition is required at any depth and temperature conditions.

Finally it is also used in gas pipeline flow measurements and covered by the ISO standard 9300.

Sonic nozzles should not be mixed with capillary devices where the supersonic speed is not reached and then no critical flow conditions are generated.

Parameters of Importance

The critical flow is determined by the parameters of the equation above. The inlet pressure should carefully be managed to generate a precise flow.



$$Q = k\alpha \frac{P_{IN}}{\sqrt{T_{GAS}}}$$

$$P_{IN} \geq 2 \cdot P_{OUT}$$

Q = Gas flow of the nozzle
k = Gas constant
α = Geometrical constant
P_{IN} = Input pressure
T_{GAS} = Temperature of the gas

Figure 2. Gas flow at the critical speed parameters

Configuration of a Sonic Nozzle Calibrator

A basic sonic nozzle gas calibrator has two main lines, one for each gas to be mixed. A high precision pressure regulator maintains a constant inlet pressure, 3 bar at each gas inlet and with repeatability better than ± 1 mbar.

As one sonic nozzle can deliver only one flow, a combination of nozzles is created for each line in order to generate different concentrations. When 2 nozzles can generate 4 mixtures, up to 1024 concentrations steps can be reached by using 16 nozzles in different combinations (1024 = 216).

A dilution range from 1/1 up to 1/1000 can be generated.

Figure 3 shows the dilution point '26.6%' with a 4 sonic nozzles device (16 concentrations). The mechanical setup is configured to have all nozzles at the same temperature and to generate an homogeneous gas mixture.

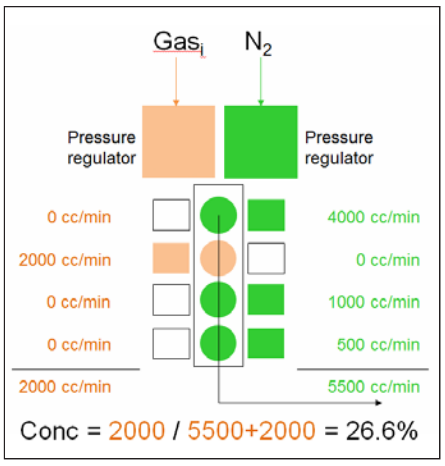


Figure 3. A four nozzle gas calibrator setup

Mechanical Setup and Performance of a Sonic Nozzle

Sonic nozzles are used either as stand-alone devices into gas circuits or, integrated in mixers/diluters. They are manufactured in nickel or gold for corrosion gases compatibility and can work up to 80°C and 10 bar maximal working pressure and 25 bar acceptable over pressure.

The nozzle is encapsulated into a metallic body for easy integration into the regulating device.

Side by Side Comparison Sonic Nozzle and Mass Flow Controller (MFC) Technologies

As the MFC is a well-known technology for gas diluters and calibrators, it is of interest to compare both methods.

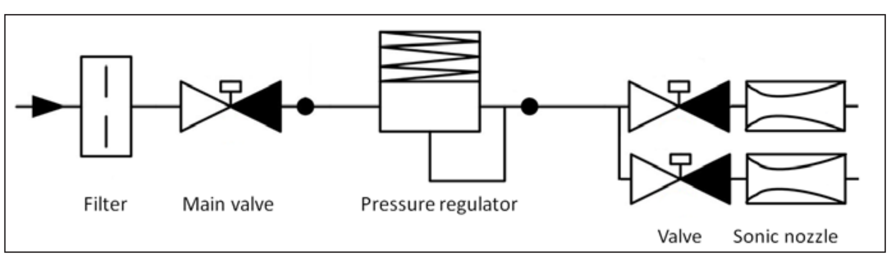


Figure 5. Sonic nozzle setup in a calibrator

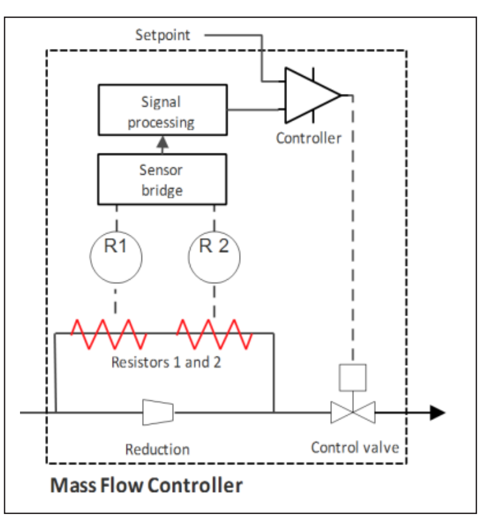


Figure 6. Mass Flow Controller setup

Figure 4. An encapsulated sonic nozzle

	Sonic Nozzle	Mass Flow Controller
Accuracy (of concentration)	≤ ± 0.2% to 0.25% *	≤ ± 1% % *
Repeatability (of concentration)	≤ ± 0.2% *	≤ ± 0.2% *
Dilution ratio range	1/1 to 1/1000 by steps	1/1 to 1/1000 continuous
Flow	Fixed	Variable
Number of gas sources	Only 2	Several
Flexibility	Regulation only	Regulation + Measurement
Flow regulation	Direct, by gas physics	Indirect, by ∆temperature
Regulation	Mechanical pressure	Flow by electronic valve
Warm up time	2 min	30-45 min
Moving parts	-	-
Sensitivity to contamination	No	Maybe affected
Available in portable enclosures	Yes	Yes
Recommended calibration	Every 18 month	Every 6-12 month

* Performances identified in ISO 17025 accredited laboratory

There is no real winner and each technology has pro and cons. When the MFC is more flexible by allowing a mix of several gases at the same time, the sonic nozzle generates only binary mixtures but at better accuracy and on the long term.

How High Accuracy is Reached with Sonic Nozzles?

High accuracy means low uncertainty. This can be generated by two ways: either by reducing the sources of uncertainty and/or reduce the uncertainty of remaining sources. The sonic nozzle combines both ways, assuming that the gas sources purity is constant:

Simplicity – No electronic signal measurement or flow regulation are needed as flow condition are blocked by gas physics conditions of the critical flow. The simple setup of a sonic nozzle calibrator is key in reducing potential sources of uncertainty. Only orifice diameter and pressure regulation remain.

Long-term stability – A high precision pressure regulator is a full metal device capable of maintaining the inlet pressure within variations of less than 2 mbar. Nozzle is made of nickel or gold with unaffected dimensions or surface properties over the time and even for corrosive gas applications. Both mechanical devices show excellent stability over years and no aging effect has being found. Their contribution to the device uncertainty is almost negligible.

Benefits

The sonic nozzle technology provides several benefits:

Metrological superior performances - The uncertainty and repeatability are better than 0.5% and across the entire dilution range of the device. Unlike Mass Flow Controllers, working below 5% of the dilution range is possible within the specifications

Flow rate is constant - not affected by downstream flow or pressure disturbances

Extended dilution capability – By combining several nozzles up to 1024 mixtures ratios can be generated. The a dilution ratio range goes from 1/1 up to 1/1000

Lower running cost – Even if acquisition costs are higher, long term stability of components reduce the frequency of validation and calibration of a calibrator

Proven performances – Finally accuracy performances of each are identified and accredited laboratories

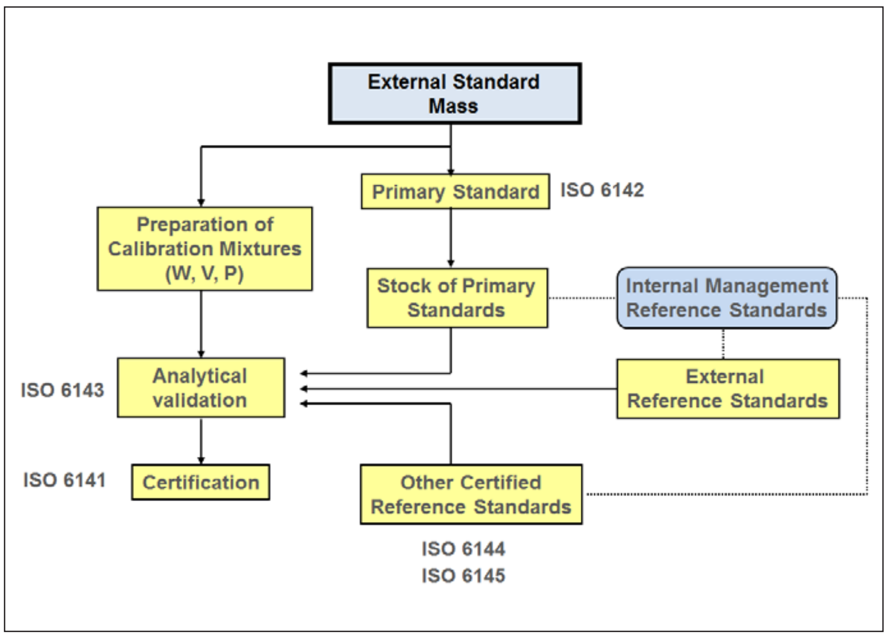


Figure 7. Flow chart of norms related to gas analysis

Traceability of Methods for Gas Analysis

As mentioned initially, gravimetric methods mentioned in the ISO 6142 have the shortest link to primary standards (the mass), but field deployment is not very convenient. The critical orifices principle, (mentioned in the ISO 6145 Part 6) although less direct maintains a full traceability with gas standards and methods as shown in *Figure 6*.

For gas analysis, four norms are involved:

ISO 6141:2000 - Requirements for certificates for calibration gases and gas mixtures

ISO 6142:2001 - Preparation of calibration gas mixtures -- Gravimetric method

ISO 6143:2001 - Comparison methods for determining and checking the composition of calibration gas mixtures

ISO 6144:2003 - Preparation of calibration gas mixtures -- Static volumetric method

ISO 6145:2009 - Preparation of calibration gas mixtures using dynamic volumetric methods

Conclusion

A gas calibrator with sonic nozzle technology involve single mechanical devices without any electronic measurement or regulation and see flow and then gas mixtures driven by the physics critical flow. Uncertainty sources are limited and provide a huge advantage for a calibrator having to work without frequent care and validation.

Over many years installed calibrators with sonic nozzles have shown superior reliability and metrological performances and therefore may be considered as an ideal transfer standard for gas calibration purpose.

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