Design and calibration of critical flow Venturi nozzles for high-pressure hydrogen applications

M.A de Huu[1], R. Maury[2]
[1]Federal Institute of Metrology METAS, Lindenweg 50, 3003 Bern-Wabern, Switzerland
[2]CESAME-EXADEBIT SA, Poitiers, France
E-mail (corresponding author): marc.dehuu@metas.ch

Abstract

In this paper, we present the strategy behind the design and manufacture of sets of Critical Flow Venturi Nozzles (CFVN) with various sizes, shapes and surface roughnesses, as well as their dimensional calibration to test the applicability of ISO 9300 for high-pressure hydrogen aplications. Some of the built nozzles and nozzle holders will be used for high-pressure measurements with hydrogen and underwent a baseline metrological check at lower pressure using inert gases to ensure that their low-pressure characteristics are known before performing the measurements with high-pressure hydrogen. Furthermore, all the nozzles are being dimensionally calibrated using an ultra-precise 3D coordinate measuring machine and will yield a database of reference data for these nozzles. As there are currently no test rigs with traceable standards that are able to directly perform the calibration of CFVN with high-pressure hydrogen, an alternative method needed to be developed and a traceability scheme with an expected uncertainty around 1 % using a calibrated Coriolis meter with hydrogen is presented.

1. Introduction

Meters are available for use with gaseous hydrogen at 100 MPa but traceable calibration services area lacking, except for hydrogen dispensers at refuelling stations. Critical flow Venturi nozzles (CFVN) are very reliable and cost-effective flow standards, which are used in many laboratories as reference flow meters. Unfortunately, ISO 9300 [1] is not applicable for hydrogen with sufficient accuracy as no adequate database of nozzle calibrations with hydrogen is available.

The overall objective of the EMPIR MetHyInfra project is to establish a metrological infrastructure for the calibration of flow meters for the measurement of hydrogen flow at pressure up to 90 MPa and flow rates up to several kg/min. The specific objectives related to CFVN are the development of and the investigation into calibration methods of CFVN and master meters to be used as standards for gaseous hydrogen at such high pressures. The measuring models for CFVN are defined in ISO 9300. The MetHyInfra project will test the applicability of ISO 9300 models for the measurement of hydrogen flow in uncharted territory by analysing CFVN behaviour in comparison with dependencies described in ISO 9300. It is also the aim of this project to define a standardised method for the dimensional characterisation of CFVN.

2. Design of CFVN

ISO 9300 specifies the geometry and method of use of CFVN. It is applicable to CFVN in which the gas flow accelerates to the critical velocity at the throat (equal to the local speed of sound) and only for steady flow of single-phase gases. The standard also gives general requirements on material, surface finish of the throat and inlet and overall design (geometry).

To study how the requirements on surface finish, geometry and size affect the measurement results from CFVN, several type of nozzles, where such parameters vary, have been manufactured. The objective is to have access to sets of nozzles with different parameters (diameter, roughness and shape) and to analyse how they affect the measurement results with a comparison of the discharge coefficient.

The pressure and mass flow rate range implies that the diameter of the nozzles is in the range of (1 to 3) mm. Mechanical tolerances indicated in ISO 9300 will be very hard to control as these are relative numbers and would yield very small absolute numbers given the size of the nozzles, especially the contour shape and the average roughness.

2.1 Design requirements for high-pressure applications with hydrogen

The design of CFVNs for high-pressure applications with hydrogen must take safety aspects into consideration. The following requirements apply:

- Design for pressure up to 100 MPa,
- Design for hydrogen application but no long-term use,
- Fullfill ISO 9300 requirements as much as possible,
- The material shall not be subject to corrosion in the intended service,
- The material shall be dimensionally stable and have repeatable thermal expansion

characteristics so that appropriate throat diameter correction can be made.

Furthermore, for easier handling, the CFVN should be embedded in a nozzle holder with standard threads for high-pressure applications which are proven in industry. Assuming no long-term use with hydrogen, hydrogen embrittlement is not considered an issue.

Based on these requirements, the material for the nozzles and the holders is stainless steel of type 1.4404 (grade 316 L).

2.2 Nozzle geometries

According to ISO 9300, there are two designs of standard CFVN: toroidal throat and cylindrical throat nozzles. Regarding surface finish, three ranges of surface roughness have been manufactured: Ra < 0.1 µm, 0.4 < Ra < 0.6 µm and 0.9 < Ra < 1.1 µm, the lowest roughness corresponding to a polished surface. Finally, two different throat diameters (1 mm and 2 mm) have been realised. This gives a total of twelve nozzles made out of two shapes, two throat sizes and three surface finishes. Technical drawings for the 1 mm diameter nozzles are shown in Figure 1. A set of simlar drawings has been realised for the nozzles with 2 mm diameter.

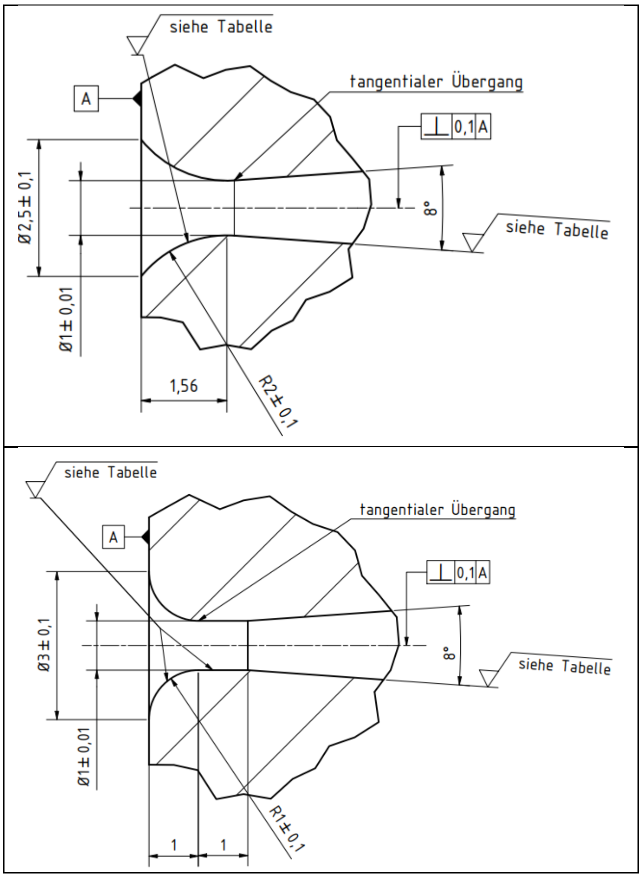

Figure 1: Technical drawings of the nozzle with 1 mm diameter for the toroidal shape (top) and cylindrical shape (bottom).

Two sets of nozzles have been manufactured: One set (Set A) for high-pressure applications up to 100 MPa and one set (Set B) for low-pressure applications up to 20 MPa. Set B contains all twelve design of nozzles while Set A consist of twins of a pair of nozzles from Set B. Table 1 gives an overview of the manufactured nozzles to be used in this project.

Table 1: Design characteristics of the 14 nozzles to be used

Designation	Surface	Throat diameter	Design
CFVN1	Ra < 0.1 µm	1 mm	CYL
CFVN2	Ra < 0.1 µm	1 mm	TOR
CFVN3	Ra < 0.1 µm	2 mm	CYL
CFVN4	Ra < 0.1 µm	2 mm	TOR
CFVN5	(0.4 < Ra < 0.6) µm	1 mm	CYL
CFVN6	(0.4 < Ra < 0.6) µm	1 mm	TOR
CFVN7	(0.4 < Ra < 0.6) µm	2 mm	CYL
CFVN8	(0.4 < Ra < 0.6) µm	2 mm	TOR
CFVN9	(0.9 < Ra < 1.1) µm	1 mm	CYL
CFVN10	(0.9 < Ra < 1.1) µm	1 mm	TOR
CFVN11	(0.9 < Ra < 1.1) µm	2 mm	CYL
CFVN12	(0.9 < Ra < 1.1) µm	2 mm	TOR
CFVN13	*Ra < 0.1 µm*	*1 mm*	*CYL*
CFVN14	*Ra < 0.1 µm*	*1 mm*	*TOR*

One pair of CFVN has been built twice (CFVN1 and CFVN13, CFVN2 and CFVN14) in order to perform a comparison between high-pressure measurements with hydrogen and low-pressure measurements with alternative fluids. The comparison of the Cd value (discharge coefficient) with a proper C* (critical flow function) estimation will provide valuable input for a possible alternative traceability route.

Each nozzle for the high-pressure measurements is mounted in a dedicated nozzle holder to avoid any manipulation. Connections are standard threads for high-pressure applications. For the set of low-pressure nozzles, three dedicated nozzle holders have been manufactured, as these nozzles will be shipped to three different laboratories for comparison measurements using substitute gases to hydrogen. Each laboratory then has its own nozzle holder.

Each holder is equipped with two pressure tappings, one located 1 D upstream of the front of the nozzle and one located 7 D downstream of the back of the nozzle, where D is the diameter of the upstream piping. A temperature probe can be inserted upstream (6 D) of the nozzle.

3. Dimensional calibration

The shape and throat diameter of nozzles are key geometric parameters for comparing measured and calculated values of discharge coefficient according to ISO 9300.

Figure 2 shows the METAS ultra-precise micro-coordinate measuring machine (µCMM) that is part of the measuring infrastructure of the METAS Length laboratory. This machine has a position measurement uncertainty as low as 50 nm (k=2) per probed point. A detailed description of the METAS uCMM can be found in Ref. [5].

The uCMM records the three dimensional geometry of workpieces, in this case CFVN. The position of individual surface points is measured by probing the CFVN surface with a precisely known probing element, here a sapphire sphere, with a maximum number of 300 probing points per mm.

Figure 2: The METAS micro-coordinate measuring machine

For this project, the 0.5 mm diametre sapphire probe makes 8 radial measurements spaced 45 degrees apart, starting from the inlet plane and moves along the flow direction toward the divergent section. Additional measurements are taken at fixed axial positions to probe the circularity of the nozzle and determine the throat diameter at the smallest section. A graphical overview of the measurement procedure is shown in Figure 3, where one can identify the 8 radial positions along which the nozzle shape is probed along the flow direction. Also indicated are 8 axial positions, represented by the circles, where the circularity of the nozzle is probed at various fixed positions along the flow axis.
Data are currently being analysed to extract several parameters like throat diameter, throat shape, and deviation from ideal shape. Further parameter will most certainly also be considered. A data reduction analysis is also planned to allow identifying the minimum number of measuring points needed for a dimensional calibration.
As of writing of this paper, measurements are still ongoing. In the end, all the nozzles will have been dimensionally calibrated with a high number of probing points and form a set of high quality data for nozzles with different characteristics.

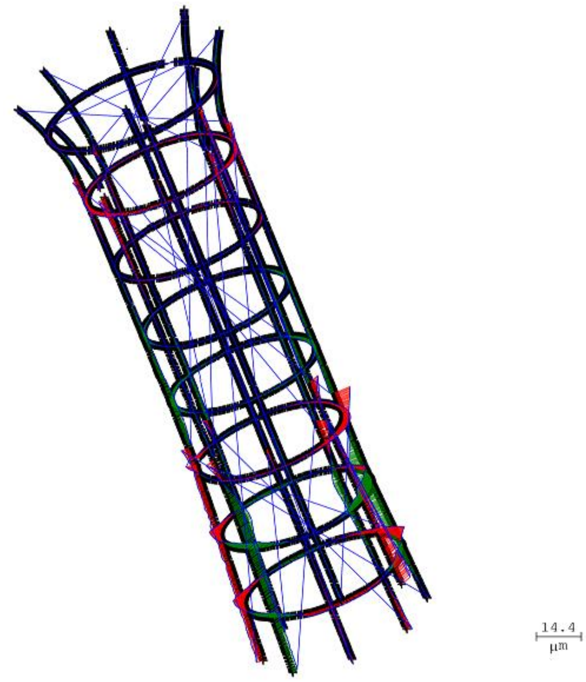

Figure 3: Measurement procedure followed by the 3D coordinate measuring machine: 8 radial and axial positions. Nozzle inlet is at the bottom of the figure; the scale only applies to the red and green lines in the figure as they indicate deviation from circularity.

4. Determination of Cd values at low pressure

First flow measurements with some CFVN have been peformed and are reported here. The main objective of these measurements is to determine the discharge coefficients of the CFVN and perform a quick metrological check before being used for the high-pressure measurements.
The characterisation of the CFVN has been carried out using nitrogen gas at an inlet pressure range fom 300 kPa to 3000 kPa. A schematic of the measurement setup and the nozzle holder are shown in Figure 4. The gas source consists of a bundle of pressure cylinders filled up with gaseous nitrogen at 300 bar. A two-stage pressure regulator allows adjusting the inlet pressure to the CFVN. The nitrogen gas passes a plate heat exchanger (WT) located before the nozzle to bring the expanding gas to room temperature (22 °C). Temperature at the inlet of the nozzle is measured by a Pt100 sensor inserted into the nozzle holder. Inlet and outlet pressure at the nozzle are measured using the intended pressure tappings in the nozzle holder. A needle valve located downstream of the CFVN allows adjusting back pressure and was used to determine the maximum back pressure of the nozzle to ensure choked flow. Finally, the nitrogen gas passes through another passive heat exchanger, not shown in Figure 4, and volumetric flow rate is determined by a reference meter at ambient conditions. Volume flow rate is converted to

mass flow rate using nitrogen gas density determined using pressure and temperature information at the reference meter and calculated values from the NIST Chemistry WebBook [3]. A leak test at the maximum measuring pressure was performed before the start of the measurements.

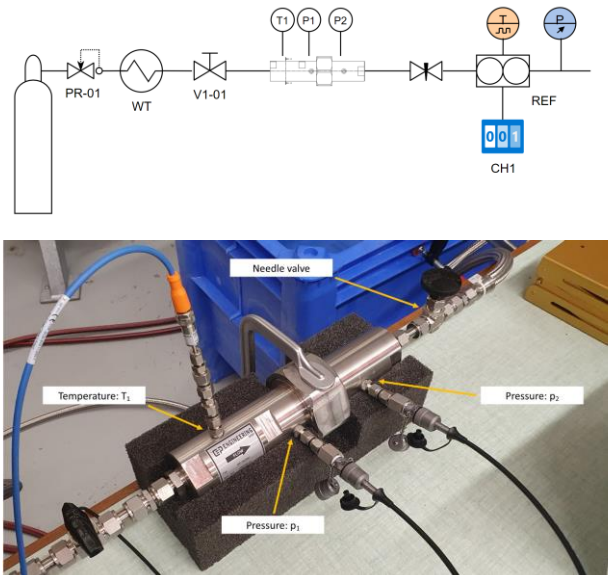

Figure 4: Top) Schematic overview of the measurement setup, Bottom) Nozzle holder with the temperature and pressure probes, flow direction is from left to right.

Preliminary results for one of the nozzles are presented in Figure 5 where calculated and measured Cd coefficients are shown. The coefficients for toroidal nozzles from Table 1 of ISO 9300 (a = .09959, b = 2.72 and n = 0.5) have been used to calculated Cd. The experimental uncertainty of the measurements is 0.3 %. The nominal diameter of the nozzle (1 mm) was taken to determine its Cd value.

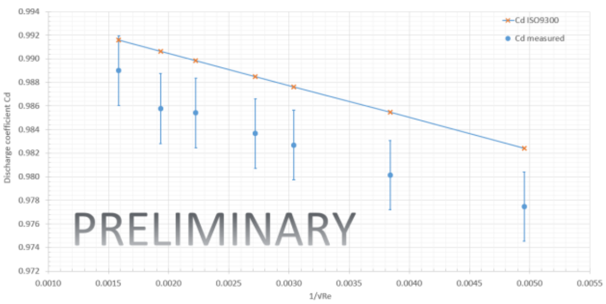

Figure 5: Calculated and measured Cd coefficients for a nozzle from this project.

Agreement is reasonable, the shapes of the Cd curves are very similar but present a small offset. This indicates that the low-pressure behaviour for this nozzle is well described by ISO 9300.

5. Traceability scheme for high-pressure CFVN with hydrogen

The high-pressure measurements with hydrogen will be performed using a closed-loop test rig where pure hydrogen will be circulating and pressure can be adjusted. The high-pressure nozzle holders containing their respective high-pressure nozzles will be mounted in a dedicated flow line housing pressure regulators, valves, heat exchangers and a Coriolis meter as reference meter.
A reference flow standard is required for the calibration of the Coriolis meter. The most appropriate reference flow device is a gravimetric primary standard designed for use with hydrogen refuelling stations. Several members of the MetHyInfra consortium have developed these systems and demonstrated measurements uncertainty of 0.3 % (k=2) or better in mass of hydrogen. The mobile flow standard built by CESAME will be used owing to its 200 L collection volume and will allow maximum flow rates for the Coriolis meter calibration.
The Coriolis meter will be calibrated with hydrogen over a flow rate range (0.3 to 1.5) kg/min delivered by a hydrogen refuelling station using the CESAME gravimetric system as reference standard. The Coriolis meter will be installed in the "warm region" of the refuelling station, upstream of the heat exchanger and pressure ramp controller. In this region, temperature is near ambient and the pressure is expected to be consistently high, typically around 90 MPa. Pressure at the meter may fluctuate depending on the operating state of the refuelling stations but the flow meter is relatively insensitive to pressure, any fluctuation in pressure during the calibration will not introduce significant error. The objective is to obtain a calibration curve for the Coriolis meter with an uncertainty of 0.6 % (k=2). This calibration meter will then be mounted in the closed-loop test rig as reference for the high-pressure calibration of the CFVN and ensure traceabiliy.

6. Conclusion

The development of the hydrogen infrastructure worldwide needs reliable and traceable metrological testing and calibrating equipment. CFVN are one key element of such an infrastructure and need dedicated investigations as to their behaviour for hydrogen flow, especially up to high pressures. The MetHyInfra project aims at offering part of the solutions for high-pressure applications with hydrogen of CFVN. Here, a systematic investigation of CFVN characteristics and how they affect hydrogen flow measurements is carried out. First, dedicated nozzles and holders for use up to 100 MPa with hydrogen have been designed. After undergoing a dimensional calibration using a 3D coordinate measuring machine, the built nozzles will be calibrated using substitute gases at low pressure and hydrogen at high pressures. Traceability for the high-pressure measurements is guaranteed by using a Coriolis meter as reference meter that has been calibrated by a gravimetric standard designed for use at hydrogen refuelling stations. The MetHyInfra project is ongoing and will deliver valuable data for the users of CFVN with hydrogen and lead to an improvement of ISO 9300.

7. Acknowledgment

This project 20IND11 MetHyInfra has received funding from the EMPIR programme co-financed by the Participating States and from the European Union's Horizon 2020 research and innovation programme.

References

[1] ISO 9300: *Measurement of gas flow by means of critical flow Venturi nozzles*, 2005.

[2] Meli F et al., “Ultra precision micro-CMM using a low force 3D touch probe”, in *Proceedings of SPIE, Conf. on Recent Developments in Traceable Dimensional Measurements III,* Vol. 5879, 240-247, 2005.

[3] NIST Web Chemistry webbook, https://webbook.nist.gov.