NOVEL METHODS OF TESTING FOR MEASUREMENT OF NATURAL GAS AND HYDROGEN MIXTURES

THOTH2

D3.1

Test rigs preparation and reporting procedure for experimental activities

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Revision Version	Date	Changes	Changes made by Partner
0	23/10/2023	First release	UNIBO
1	30/10/2023	Review by the partners: correction of the description of the test benches	CESAME, ENAGAS, FBK, NaTran, GS, INIG, INRIM, METAS
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4	20/02/2025	Addition of safety procedures for testing	CESAME, ENAGAS, FBK, NATRAN, GS, INIG, INRIM, METAS

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ACRONYMS AND SYMBOLS

Acronym	Definition
A	Area, in m^2
BMC	Best measurement capacity
CFVN	Critical flow venturi nozzles
CMH	Chilled-mirror hygrometer
CRDS	Cavity ring-down spectroscopy
CMC	Calibration and measurement capability
CRV	Comparison Reference Value
CTC	Climatic test chamber
DoE	Degree of Equivalence
DMP	Data Management Plan
DN	Nominal diameter
DSO	Distribution System Operator
DUT	Device under test
FCV	Flow control valve
H2NG	Hydrogen and natural gas
HE	Heat exchanger
HF	High frequency
ID	Instrumentation Diagram
ILAC	International Laboratory Accreditation Cooperation
ILC	Interlaboratory comparison
LF	Low frequency
LFP	Low frost-point
MRA	Mutual Recognition Arrangement
MUT	Meter under test
MV	Manual valve
NG	Natural Gas
OIML	International Organization of Legal Metrology
PFD	Process Flow Diagram
PHG	Primary humidity generators
PLC	Programmable Logic Controller
PR	Pressure reducer
PRT	Platinum resistance thermometers
PRV	Pressure regulating Valve
PTR-MS	Proton transfer reaction mass spectrometer
PV	Pneumatic valve
RH	Relative humidity
RTD	Resistance Temperature Detector
SPRT	Standard platinum resistance thermometers
SV	Shut-off valve
TOF	Time of flight detector
TPHG	Transportable precision humidity generator
TS	Transfer standards
TSO	Transmission System Operator

U	Expanded Calibration Uncertainty, in °C
URV	Upper Range Value
USM	Ultrasonic meter
UUT	Unit Under Test
WME	Weighted Mean Error
WS	Working standards

Symbol	Definition
Ar	Argon
CH_4	Methane
CO_2	Carbon dioxide
H_2	Hydrogen
HF	High frequency
I	Electric current, in mA
I_{max}	Maximum electric current, in mA
I_{min}	Minimum electric current, in mA
k	Coverage factor
Kr	Krypton
M	Molar mass of the gas, in kg/kmol
N_2	Nitrogen
P	Pressure, in bar
P_{max}	Maximum pressure, in bar
P_{min}	Minimum pressure, in bar
Q	Mass flow rate, in kg/s
Q_{max}	Maximum volumetric flow rate, in m^3/h
Q_{min}	Minimum volumetric flow rate, in m^3/h
R	Ideal gas constant, in J/(kmol·K)
Re	Reynolds number
T	Absolute temperature, in K
V	Volume, in m^3
Z	Compressibility factor
ρ	Density, in kg/m^3

EXECUTIVE SUMMARY

Starting from the testing protocols developed in Task 2.2, Task 3.1 provides a description of the laboratories and of the operative procedures developed to test the state of the art (SoA) natural gas (NG) transmission and distribution sectors' measuring devices that have been prioritized in the Task 2.1.

Since experimental activities will be performed in several test facilities, information about the measurement equipment, the materials, and the procedures to respect the protocol and safety requirements are described in this deliverable for each measuring device category: gas meters, pressure transmitters, trace water humidity sensors, leak detectors, and volume converters.

Regarding **transmission gas meters**, ageing and limit tests will be performed. Ageing will be performed at different hydrogen (H_2) concentration, i.e., 25%vol. and 100%vol. respectively in CESAME and NaTran testing facilities accordingly with the protocols developed in Task 2.2. As shown in Table 1, different testing conditions can be achieved. Once ageing is the completed, the gas meters will be calibrated in different laboratories ensuring interlaboratory comparison (ILC) accordingly with the proposed indications reported in Deliverable D2.2. The available testing conditions declared by the laboratories in the THOTH2 consortium are provided in Table 2.

Table 1. Testing facilities adopted for ageing.

Parameter	CESAME	NaTran
Operative fluid	CH_4; H2CH4 mixture up to 100%vol.	NG; H2NG mixture up to 100%vol.
Available pressure range	0 - 35 bar	up to 80 bar

Table 2. Testing facilities involved for calibration.

Parameter	CESAME	Enagás	Gaz-System	NaTran
Fluid	Air	NG	NG	NG
Operational mode	Open loop	Closed loop	Closed loop	Open loop
Pressure	1 - 50 barg	3 - 50 barg	8 - 54 barg	< 30 barg
Flow rate	5 - 50000 Nm³/h	10 - 10000 m³/h	8 - 6000 m³/h	0.1 - 2000 Sm³/h
Uncertainty	0.21%	0.23 - 0.26 %	0.22 - 0.29 %	-
Max diameter of the Device Under Test (DUT)	400 mm	DN600	DN300 (+ DN350 & 400)	DN80

As for transmission gas meters, also **distribution gas meters** will be tested in different laboratories with different hydrogen and natural gas mixtures (H2NG). The distribution gas meters will be aged at different H2NG mixtures and subsequently calibrated with three different fluids. For this purpose, ageing will be performed in INIG at two different concentrations, i.e., at 25%vol and 100%vol. H2. Once ageing is completed, periodical

calibration will be performed in METAS, INIG and NaTran test benches with different fluids. The main characteristics of the involved laboratories are shown in Table 3 and Table 4.

Table 3. Testing facility adopted for ageing of the distribution gas meters.

Parameter	INIG
Fluid	H2NG mixture: 25%vol; 100% vol
Operational mode	flow / static
Pressure	2 - 8 kPa

Table 4. Testing facility adopted for the calibration of the distribution gas meters.

Parameter	NaTran	INIG	METAS
Fluid	The same for gas transmission flow meters. For domestic gas meters up to 16 Std.m³/h a new bench is currently in process.	25%vol H2NG	pure H2
Operational mode		open / close loop	open loop
Pressure		< 10 kPa	2 - 50 bar
Flow rate		< 100 Sm³/h	0.071 - 120 Nm³/h
Uncertainty		0.22 - 0.27 %	< 0.3%
Nominal size of the DUT		G65	G40

Tests on the selected **pressure transmitters** will involve two laboratories. INIG and CESAME will perform ageing at 25%vol and pure H2 respectively, instead just INIG will verify the effect of the ageing through means of calibration.

Regarding the **trace water humidity sensor** tests will be developed in INRIM, the Italian National Metrological Institute. The Istituto Nazionale di Ricerca Metrologica (INRIM) is the Italian National Metrology Institute. In its role as the National Metrological Institute, INRIM provides the measurement traceability by means of a comprehensive measurement, testing and calibration portfolio with over 400 services available in the field of mechanics, thermodynamics, time and frequency, electricity, photometry, and acoustics. The expertise, competence and facilities shared in this project include the development of primary standards and measurement techniques for humidity in gases, the measurement of thermo-physical properties of real gas mixtures, as well as the measurement of surface and air temperature. The humidity laboratory shares a broad range of calibration and testing facilities to provide measurement traceability to sensors and analysers for trace water measurements in different gas matrices including energy-relevant gases.

Tests on the selected **leak detectors** will take place in two different laboratories, FBK and INIG. FBK has a long-term experience on the development of flow sensors and solid-state gas sensors, specifically chemoresistive gas sensors. Specifically, the testing activity will be performed in the Gas Qualification Laboratory (Laboratorio Qualifica Gas). INIG will instead perform test through the GU_84 test bench. The test bench allows to generate the desired gas volume flow rates and to simulate atmospheric conditions that could be present in the field.

The selected **volume converter** will be experimentally tested in Enagás Metrology and Innovation Centre (CMI), Zaragoza.

The detailed description of the laboratories and the operative and safety procedures are reported in the following chapters. Specifically, this deliverable is divided in nine sections and an appendix:

- The first section indicates the rules of good practices that should be followed to ensure rigorous results relating to transportation and testing conditions.
- Chapters 2 to 7 describe the testing laboratories and the procedures for each measuring device, i.e., gas transmission and distribution meters, pressure transmitters, trace water humidity sensors, leak detectors, and volume converters.
- Chapter 8 shows the data recorded during the experimental testing activities.
- Finally, Chapter 9 reports the main conclusions.

The list of public documentation and links regarding the measuring devices installed in the testing benches and described in the report are reported in the Appendix.

1. COMMON RULES FOR THE MEASURING DEVICES TO BE TESTED

While specific procedures are detailed in the referring section, some general rules will apply to all the measuring devices to be tested and will be ensured by the THOTH2 consortium.

1.1. Delivery, handling and transport of the measuring devices to be tested.

The devices should be delivered to the test site in their normal packing, together with the accessories and all manuals normally supplied. In fact, as the workload distribution shows, partners will have to exchange the meters between them. To do so, an agreed procedure for that exchange has to be ensured.

Special attention should be given to avoiding damages during handling and transportation that could negatively influence the results of the test.

As a general rule, the measuring devices must be handled with care during transportation, packing/unpacking and mounting/dismounting operations.

To delivery meters to another partner, the partner who is sending the device is responsible with respect to the following requirements:

- A sufficient number of photos of the measuring device/s have to be taken before packing to demonstrate that no damage is present.
- The measuring devices and any other associated equipment have to be packed securely.
  - Proper and sufficient materials, including, for example, shrink wrap, crinkle paper, or packing peanuts could be used to avoid damages by filling the empty space in the box and cushioning the item.
- A photo of the package has to be taken before shipping.
- Labels mentioning the set has to be created and attached to the package.
  - If applicable to the measuring device technology under test, during loading/unloading of the travel meters slinging will correspond to the appropriate markings. Places to attach slings must be marked with 

labels, places marked 

<u>must not</u> be used for slinging, centre of gravity is marked with 

label.
- The package containing the measuring device has to be shipped.
  - If applicable and when possible, the measuring devices should be preferably transported as "fragile goods". When the measuring devices will be delivered as "fragile goods", fragile sticker and clear shipping label alert handlers to a delicately packaged item. Multiple stickers on all sides of the package should be used.
- The partner to whom the measuring devices have been sent has to be informed by email containing the photos taken as described above.

Once the partner has received the package, the following instructions will be adopted:

- To document that no damage has occurred during the transport and handling upon arrival and prior to departure, the visual check of the transportation packages, including lock mechanisms, will be performed, condition of the shock and tipping indicators (if appropriate) will be checked and documented. For this purpose, photos of the package have to be taken once it arrives at the site.

- The measuring devices will be unpacked with care.
- The measuring device will be visually checked. A sufficient number of photos of the measuring devices have to be taken.
    - Any observed damages or nonconformities will be documented.
- The Record of transportation and handling will be filled in, signed, scanned and sent to the Task 3.3 leader together with the photographs.
- The measuring device will be stored until the date on which the tests take place according to the storage conditions defined in the protocols.

1.2. Testing conditions

The testing activities must be performed to ensure that:

- All the testing activities shall be carried out in accordance with this procedure, by an authorized and experienced person.
- The measuring instruments shall be used for their intended purpose and under the conditions specified by the manufacturers and the procedures reported in this document.
- All the measuring instruments shall be used with the required range and accuracy class and with valid calibration certificates.
- For those testing activities in which the presence of air could influence the results, the measuring station shall be airtight to avoid contamination and/or safety hazards.
- A constant temperature in the room and in the area of the reference and calibration instruments and calibration equipment shall be maintained, if applicable and relevant to the testing conclusion.
- All safety mitigation and protective measures must be taken in order to minimize any risk due to the presence of explosive and flammable gases.

2. TESTING OF GAS TRANSMISSION FLOW METER IN THE PRESENCE OF H_2

Gas transmission meters selected in Task 2.1 (Deliverable D2.1) will be tested in accordance with the protocol developed in Task 2.2 (Deliverable D2.2).

Testing activities include ageing and calibration for gas transmission meters. As described in the next sections, calibrations will be performed in different laboratories, while ageing will be performed at CESAME and NaTran facilities.

Objective of the test: The tests will consist i) in limit tests, i.e., tests about the accuracy of state-of-the-art technology (e.g., turbine, rotary and ultrasonic gas meters) considering different H_2 concentrations conditions up to pure H_2, and ii) in ageing to assess H_2 impact.

Operative procedures adopted during the experimental activities: Two activities will be performed to achieve the objective:

1. Ageing of the devices with 100%vol. and 25%vol. H_2 in NG. The test duration and the ageing conditions are indicated in Deliverable D2.2.
2. Calibration of the measuring devices. The test conditions are indicated in Deliverable D2.2. Different testing fluids will be used for calibration:
    a. CESAME: air
    b. Enagas: NG
    c. Gaz System: pure NG
    d. NaTran: NG
    e. Other facility outside the consortium: 100%vol H_2.

In the next sections, information about the measurement equipment, materials, and operational procedures to respect the metrology requirements indicated in WP2 is provided. The safety measures adopted throughout the testing activities are also included in the discussion.

2.1. Ageing of the gas transmission meters: introduction

The ageing of the gas transmission meters will be performed at CESAME and NaTran at the testing conditions shown in Table 5.

Table 5. The operative conditions of performed ageing.

Ageing	Laboratory	Fluid	Pressure (bar)	Temperature (C)	Period of time
1	CESAME	H2NG (25%vol)	Nominal pressure + or – 15%	Ambient temperature	4+4

2.2. Ageing of the gas transmission meters: test bench and main components

THOTH2 gas transmission meters' ageing testing benches are described in the next sections.

2.2.1. Cesame ageing test bench

CESAME-EXADEBIT is an internationally recognized laboratory specialized in the field of gas flow metering. With a strength coming from more than 30 years' experience in this area, CESAME-EXADEBIT holds the French national reference for the cubic meter of gas under high pressure and historically carries out legal verifications for gas network Transmission System Operator (TSO)'s and Distribution System Operator (DSO)'s, as well as calibrations and tests for various industries - gas, chemical, aeronautics, mechanical, with ad hoc ISO 17020 and ISO 17020 accreditations.

As a Designated Institute of the Laboratoire national de métrologie et d'essais (LNE), i.e., the French National Metrology Institute for high pressure gas flow metering (referred as "LNE-LADG" for this purpose), CESAME acts in the framework of EURAMET (European association of Metrology) under the aegis of the Bureau international des poids et mesures (BIPM) for maintaining and improving the national flow unit reference through continuous comparisons with other national metrology institutes all over the world, participating to several joint research program addressing emerging metrology needs and developing the corresponding references. For instance, Cesame-Exadebit has developed since 2021 one of the first four mobile references in Europe for the calibration (or legal verification) of H_2 dispensers for vehicles.

Regarding ageing testing activities, the CESAME test bench is designed to supply different meters with H2 and/or H2NG at the operating pressure for the desired time. Until the test is completed, the meter is visually inspected, recalibrated and the results are compared with those prior to the test phase.

This test lab is designed to supply gas (methane, i.e., CH_4, H_2 or a mixture of the two) non-stop to meters of any type. It can operate dynamically (in a closed loop) or statically. Like its predecessor, it is installed in a well-ventilated room for safety reasons and is equipped with two gas detectors (H_2 + CH_4) which are linked to a valve system that cuts off the supply in the event of a leak.

The main installed components are:

- Hydrogen bundles / CH_4 + H_2 mixtures
- Pneumatic valves
- Manual valves
- Pressure sensors
- H2 detectors
- Meters (turbine and piston types)
- Venting system.

The Process Flow Diagram (PFD) of the plant is shown in Figure 1. From the left of the scheme, pure H2 or H2 blends are supplied to the testing bench through a pressure reducer (PR) that reduces the gas pressure as it leaves the cylinder from 200 bar or 150 bar to a pressure selected before starting the test and between 0 and 35 bar in accordance with the testing pressure set point.

Downstream the pressure reducer, the gas passes through the pneumatic valve "PV" and along the hose (F1) is supplied to the testing bench. It has to be noted that also nitrogen (N_2) gas can be supplied to the testing bench connecting the cylinders to the dedicated connection fitting indicated as "EN2".

Several operative conditions can be simulated as described below:

- **Static ageing mode.** In this configuration, the H_2 cylinders are connected to the testing bench and the manual valve "MVB" is open. In this configuration:

- The manual valve “MV2” is opened to allow H_2 or H_2 blends to enter the meter or any other measuring device to be aged.
- The manual valves “MV1” and “MV3” are closed, avoiding H_2 or H_2 blends to exit the testing bench. In case of leakage, the gas pressure is maintained by the pressure reducer.

- **Dynamic ageing mode.** Compared to the static mode, dynamic mode requires a closed-loop bench. Although it will not be used in the THOTH2 project, in the dynamic ageing mode, the manual valve “MV3” is opened, and gas is recirculated back through the hose (F4) by a blower.
- **N_2 passivation (i.e., flushing).** In the case of N_2 passivation/flushing, the following operations are performed:
    - The N_2 cylinders are connected to the testing bench by connecting the hose to the connection fitting “EN2”.
    - The manual valves “MV1”, “MV2” and “MV3” are opened to pass N_2 to all parts of the circuit removing any trace of H_2 or H_2 blends before operating on the testing bench.

If gas is detected by detectors installed in the room or if the value of pressure measured in the circuit by the pressure transmitter “P” exceeds the authorized threshold, an alarm is sent to the control system in order to automatically cut off gas supply by closing the “PV” valve. N_2 purging is then performed by the operator.

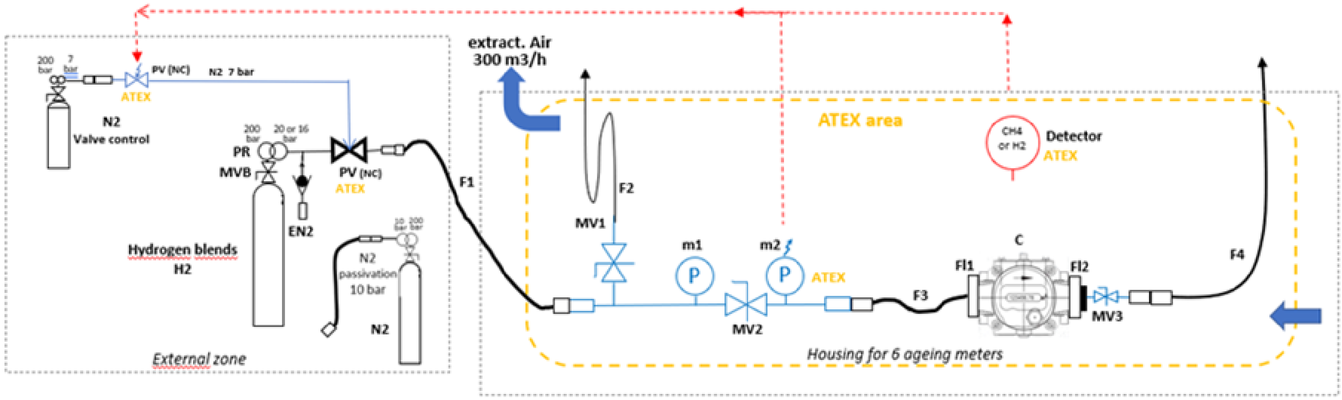

Figure 1. The PFD of the ageing test lab.

2.2.2. NaTran ageing test bench: FENHYX – 100% H_2

The aim of the platform FenHYx is to test all types of devices, innovative processes and pipes used on the gas network for the transmission and distribution of H2NG mixtures up to 100% H_2, and at different pressure levels. This will enable to overcome the technical challenges for H_2 development, and the industrialization of innovations in this field.

The PFD of the ageing bench is shown in Figure 3 while the main operative conditions are shown in Table 6. A 3D drawing is shown in Figure 2.

Table 6. Main operative conditions of the FenHYx test bench.

Pressure	up to 80 bar
Temperature	-20°C to +60°C
Gas mixtures	Natural gas, H2NG mixture up to 100%, 100% CO2
Number of gas meters simultaneously	12
Operating mode	Semiautomatic

The FENHYX static aging bench is designed to supply simultaneously up to 12 meters with H_2 and/or H2NG at the operating pressure, and for a long time period (up to 24 months).

For safety reasons, the lab is installed in a well-ventilated room and is equipped with many gas detectors (H_2 + CH_4), which are linked to the controlling automatism of the bench.

The main installed components are:

- Hydrogen cylinders / H2NG mixtures / Helium / Nitrogen
- Pneumatic valves
- Pressure sensors
- Temperature sensors (PT100)
- Gas detectors: H_2 and CH_4
- Venting system
- Alarm system.

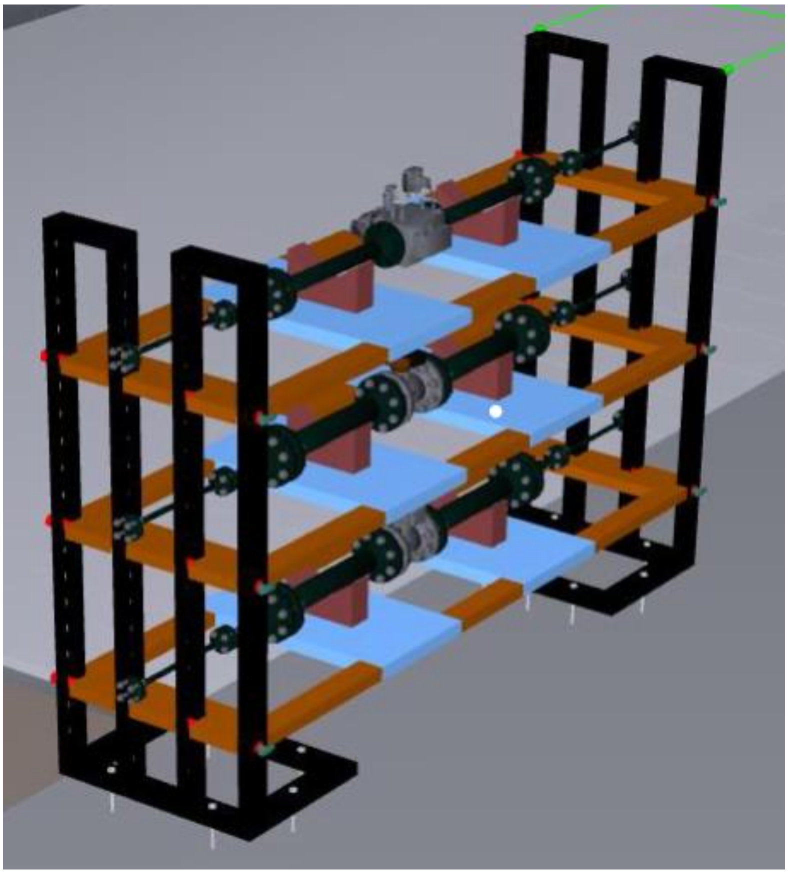

Figure 2. FENHYX – Static ageing bench: 3D drawing (NatRan).

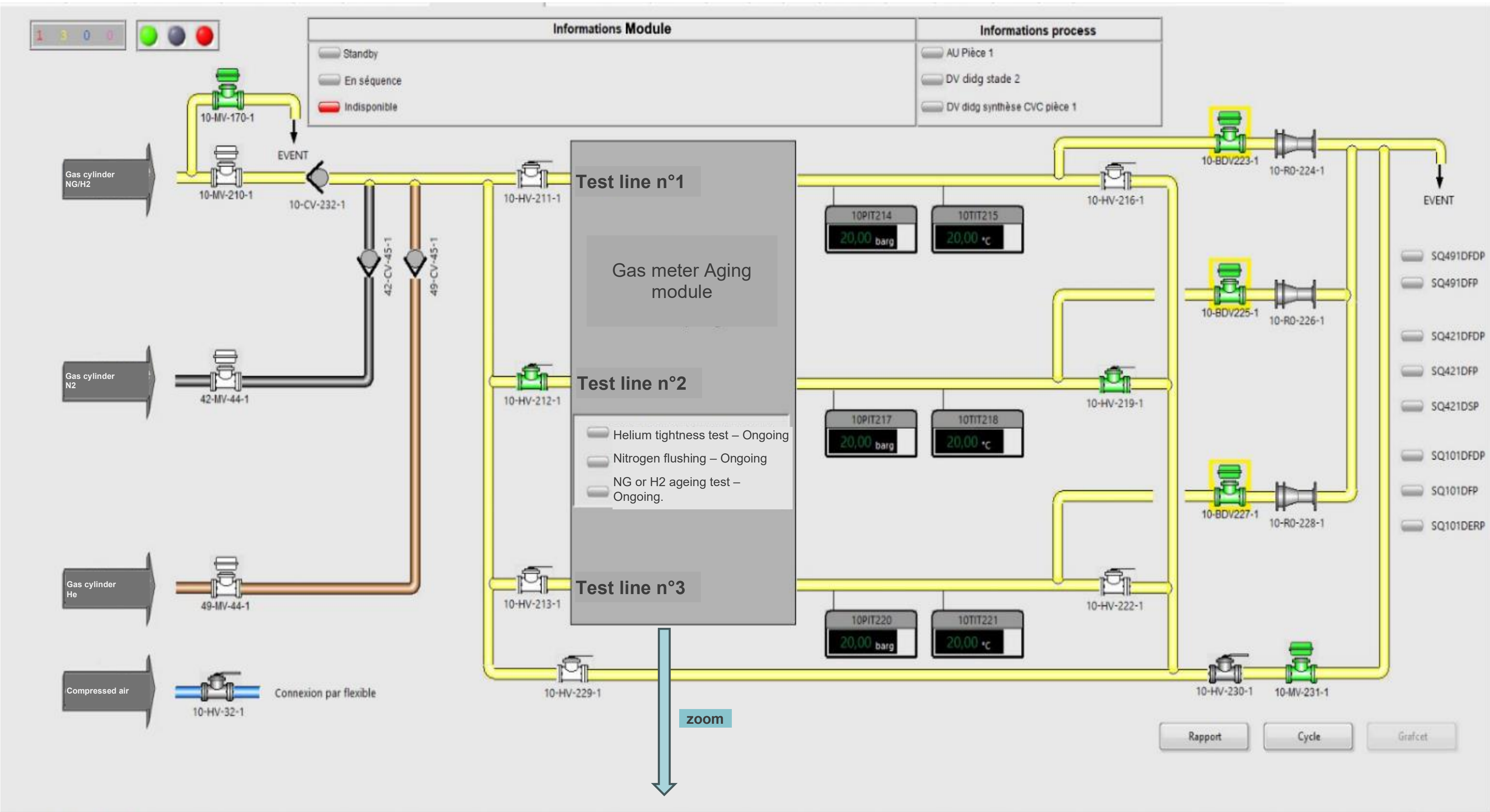

Figure 3. Simplified Process Flow Diagram of the aging testing bench (NaTran).

2.3. Ageing of the gas transmission meters: testing procedure

The testing procedure to complete the ageing of the gas transmission meters is reported in Sections 2.3.1 (CESAME) and 2.3.2 (NaTran).

2.3.1. Cesame ageing test procedure

Referring to the schematic shown in Figure 1, the ageing test refers to the steps 2 and 4 of the procedure:

1. Calibration of all the gas meters with different gases (H_2, H2NG, Air, NG) as references;
2. Exposure to H_2/H2NG for a defined period of the meters (see Deliverable 2.2);
3. Calibration using different gases (H_2, H2NG, Air, NG same measuring equipment as point 1 ***but*** only for some meters) (see Deliverable 2.2);
4. Exposure to H_2/H2NG for another defined period (see Deliverable 2.2);
5. Calibration using different gases (H_2, H2NG, Air, NG same measuring equipment as point 1 ***but*** only for some meters).

The steps relating to meters' calibration (1, 3 and 5) will be detailed in Section 2.4 while those referring to ageing are reported in Figure 4.

For safety reasons, the test installation will be located in a well-ventilated room with a safety system to detect the H_2 and CH_4 content in the air. For testing purposes, the ambient conditions will be maintained at the set value as defined in the testing protocol.

Safety rules during ageing test:

As the equipment is under pressure in the test room with hydrogen (H2) and methane (CH4), it is considered to be an ATEX zone, for which reason we have taken the following requirements into account:

1. Access to the test room is authorised only to a few people from the company known as 'authorised personnel', subject to the following conditions:
   - Put on ATEX clothing and PPE
   - To be equipped with a portable gas detector and oxygen meter (test their operation beforehand).
   - Leave all non-ATEX mobile phones and electrical equipment outside and impose these rules on any visitors.
   - Use an ATEX mobile phone (for photos or to call another authorised operator).
   - Use the safety sign at the entrance to the premises to indicate that work is ongoing.
2. The electrical equipment in the room is all ATEX certified and that which is not ATEX certified is located outside the room.
3. The room is equipped with an extractor which renews the air in it a certain number of times an hour so that in the event of a leak, however large, we can renew it in a very short time to avoid explosions.
4. The room is fitted with two gas detectors (one for H2, the other for CH4).
5. The valves used to interrupt the link between the gas supply and the test bench in the room are controlled by the detection. Above the authorised value of gas in the volume, the valves are closed to isolate the bench.
6. An internal procedure has been put in place in the event of a suspected anomaly or incident:
   - Reasons:
     - Absence of permanent air extraction noise in the proximity of the test room.
     - Permanent noise/whistling coming from the test room or the gas cylinder storage area.
     - Fire emanating from the test room or gas cylinder storage area.

- Absence of electricity in the premises.
- Flooding of the test room.
- Partial or total collapse of the roof of the gas cylinder storage area.

- Actions to be taken by authorised personnel:
  - Emergency stop on the control panel at the front of the test room OR if this is not possible (fire in the test room), turn down the 2 right-hand isolating switches in the general electrical panel.
  - If possible, close the gas cylinder valves.
  - Control the fire if possible or, failing that, call the fire brigade (18).
  - Finally, ask the technical manager in charge of the tests for assistance in defining the procedure to be followed.

2.3.2. NaTran ageing test procedure

Referring to the schematic shown in Figure 3, the ageing test refers to the steps 2 and 4:

1. Verification of the installation "tightness". If a positive result is achieved, then the meter is calibrated on one of NaTran ISO 17025 calibration benches with different gases (H_2, H2NG, Air, NG). This calibration is considered as the reference. Specifically, before the start of the ageing test, every meter is calibrated with NG or H2NG mixture up to 100%vol.
2. The meter is installed on the "static ageing" bench and exposed to H_2 or H2NG mixture at the defined pressure and during the defined period of the meters.
3. The meter is recalibrated periodically on the above-mentioned calibration bench with the same gas as for the "reference" calibration. The calibration frequency is defined in the THOTH2 testing procedure.
4. The meter is reinstalled on the "static ageing" bench to continue its ageing test.
5. At the end of the total ageing period, the meter is recalibrated for the last time, and all the calibration curves are compared.

The steps relating to meters' calibration (1, 3 and 5) will be detailed in Section2.4.

Description of the implementation of point 2 and point 4:

As for CESAME, similar safety rules regarding ventilation and explosive gas monitoring sensors apply. Furthermore, ambient conditions are maintained and recorded.

The ageing procedure is shown in Figure 5 will be adopted.

- <u>Flushing</u>. An N_2 source is connected to the connection stub to flush the test installation. Attention must be given to not exceeding the maximum pressure of the meters. Once the flushing is completed, the manual valves are closed to ensure no outside air enters the installation. Once the flushing is completed, the manual valves are closed to ensure no outside air enters the installation.

- <u>Supply of the testing gas</u>. Open the H_2/H2NG cylinder, adjust the pressure reducer to the required set point pressure after reopening the previously closed valves ("MV1", "MV2" and "MV3"), as indicated by the pressure gauge on the pressure reducer.

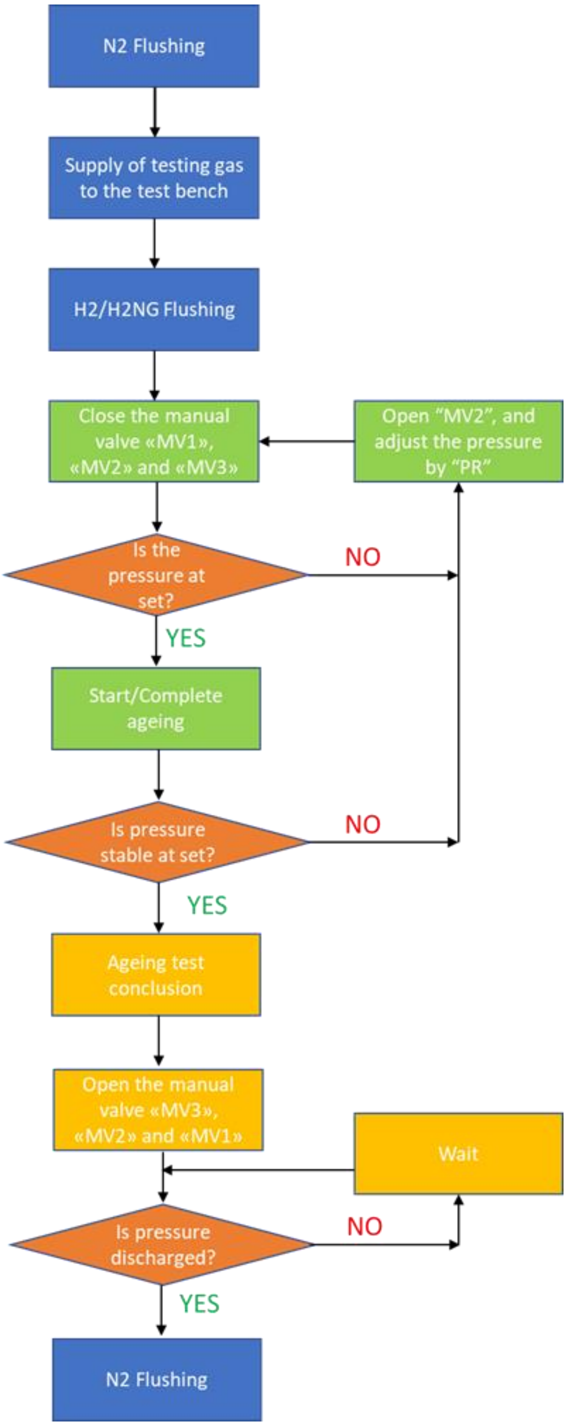

Figure 4. The ageing procedure at CESAME.

After replacing at least three volumes of the test installation (or after letting the gas flow through the circuit for ten seconds or more), the valves are closed in the following sequence "MV3", "MV2" and last "MV1".
Check the pressure on the pressure gauges installed in the circuit, which should be equal to + or - 15% of the required set point. If not, use the pressure reducer to adjust the pressure to the required value and open the valve "MV2".
Note the beginning of the aging process of the meters with H_2/H2NG.

- Pressure check. Testing pressure has to be regularly checked by the pressure gauge in the test installation for a drop in pressure and, if necessary, top up the pressure to the required value. For this purpose, the test installation will be equipped with various manometers for pressure measurement (as it is shown on the diagram) to check the pressure in the circuit and ensure that the required working pressure is available during the test.

- Completion of the test. Once the defined period of ageing is completed, the test installation should be emptied (atmospheric pressure in the installation could be verified with the manometer) by opening the manual valves after having closed the H_2/H2NG supply. The following opening sequence will be adopted "MV3", "MV2" and "MV1".
Once valves are opened and before handling the meters, N_2 flushing will be repeated to minimize any risk occurring when meters are dismantled due to the presence of H_2/H2NG gas.

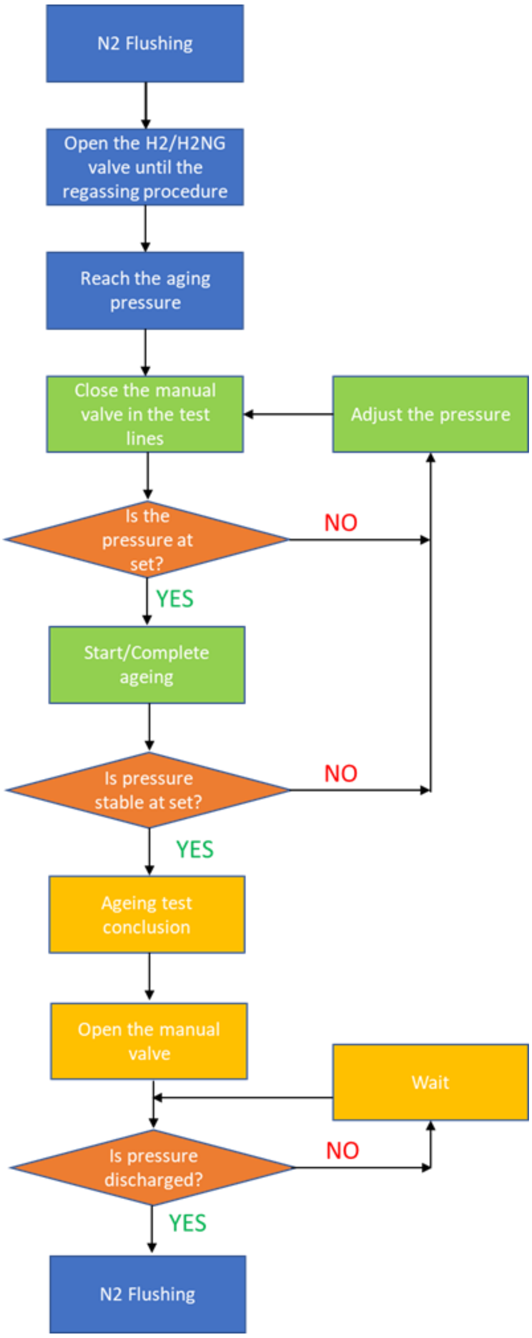

Figure 5. The ageing procedure at NaTran.

- Flushing. Connect an N_2 source to the connection stub to flush the test installation, paying attention to not exceeding the maximum pressure of the meters. After having completed the flushing of the test installation, the N_2 inlet valves ("42-MV-44-1") are closed.

- Supply of the testing gas. Open the H_2/H2NG valve ("10-MV-210-1") until the end of the regassing procedure. Open the venting valve until the pressure in the test line is at 1 barg. Once the pressure is reached the downstream valves are closed to start pressurization. Open the H_2/H2NG valve until reaching the ageing pressure, using the pressure sensor. Check the pressure on the pressure gauges installed in the circuit, which should be equal to ± 15% of the required testing pressure. If not, use the H_2/H2NG valve to adjust the pressure to the required value. Note the beginning of the ageing process of the meters with H_2/H2NG.

- Pressure check. Testing pressure has to be regularly checked by the pressure gauge in the test installation for a drop in pressure and, if necessary, top up the pressure to the required value. For this purpose, two times a day the research technician achieves a visual inspection of the gas meter, and the pressure level is controlled on the bench synoptic. If the pressure is lower than the pressure test, an alarm is triggered on the synoptic and the research technician repressurizes the test line.

- Completion of the test. Once the defined period of ageing is completed, the test installation should be emptied (atmospheric pressure in the installation could be verified with the manometer) by opening valves.
  Once valves are opened and before handling the meters, N_2 flushing will be repeated to minimize any risk occurring when meters are dismantled due to the presence of H_2/H2NG gas.

  As shown the procedure developed and implemented in NaTran will be very similar to those implemented in CESAME.

Safety rules during ageing test:

A hydrogen leak in the ambient air has a higher probability of ignition compared to natural gas due to its explosivity range. Therefore, the prevention of leak risk (through preventive measures such as detection or forced ventilation) is central to the risk prevention approach. As a reminder:

- A hydrogen/air mixture containing between 4% and 8% hydrogen can ignite upon contact with an ignition source.
- A hydrogen/air mixture with more than 8% hydrogen can explode upon contact with an ignition source.

The origins of gas fluid leaks (hydrogen or any other pressurized fluid) in the "Lab FenHYx" installations can be categorized as follows:

- Leaks by permeation due to the very small size of hydrogen molecules, which facilitates the migration of gas through materials.
- Leaks due to the progressive wear of materials (corrosion, mechanical fatigue).
- Leaks due to sealing defects (flanges, seals, instrumentation).
- Leaks resulting from the rupture of the walls of a pressurized equipment.

The safety study resulted in:

- The definition of an ATEX zoning plan.
- The technical definition of the gas detection system, fire detection system, and air extraction system for the entire building.

Type of leak	Detection method	Control methods
Gas permeation & micro-leak at connection	Permanent multi-gas detection for test halls. (No permanent detection for external piping)	Natural ventilation and mechanical ventilation of premises
Low to moderate flow leak in confined space	2 thresholds set on the gas detection system (15 and 30% LEL). At threshold 1: - Audible alarm - Visual signaling (indicator lights)	
High flow rate in confined space	At threshold 2 (30% LEL): - Audible alarm - Emergency shutdown and global depressurization, power cut, as per APS instructions	

The ultimate safety scenario is an emergency shutdown with depressurization and power cut. This trigger can be initiated by the Safety PLC or manually. The only equipment remaining powered are those related to safety (safety PLC, safety valve power supply, ATEX ventilation, and all types of detectors).

Since the installation is not physically connected to the GRTgaz transport network (including adjacent installations of the Alfortville network interconnection), the safety shutdown of the Lab FenHYx is completely neutral in terms of effect for the Operations Directorate. This action has the effect of:

- Depressurizing and venting all gas supply lines on the site (helium, nitrogen, natural gas, hydrogen) and isolating the B50 cylinders stored on the external platform.
- Stopping all operating equipment, depressurized in the same way.
- Interrupting the power supply to all test equipment (except the PLC and detection and safety equipment).

In addition to the above prevention measures and complementing the gas detection devices and pressure level measurement systems, the planned maintenance plan will include a dedicated action in the form of an annual leak detection campaign on the flanges and fittings of high-pressure piping.

2.4. Calibration of gas transmission meters: introduction

The gas transmission meters will be calibrated in CESAME, ENAGAS, Gaz System, and NaTran. Additional testing benches could be evaluated in the case that gases and operative conditions different from those available in the THOTH2 consortium test benches will be necessary.

The following sections provide the main information about the testing benches and the calibration procedures implemented.

2.5. Calibration of gas transmission meters: test benches and main components

The calibration of the gas transmission meters will be performed in different testing benches as described in the following sections.

2.5.1. Cesame calibration test bench

The “M1” test rig uses dry air under pressure, and it allows to carry out tests under pressure up to 40/50 barg depending on the nozzle used. Furthermore, also significant flow rates can be managed in the test rig at low pressures, i.e., at 1 barg. Flow rates between about 5 Nm^3/h and 50000 Nm^3/h are possible. Critical flow sonic nozzles, used as a reference, ensure to obtain low uncertainties which are about 0.21% (k=2).

A picture of the M1 test rig is shown in Figure 6, while Figure 7 shows the synoptic of the test bench.

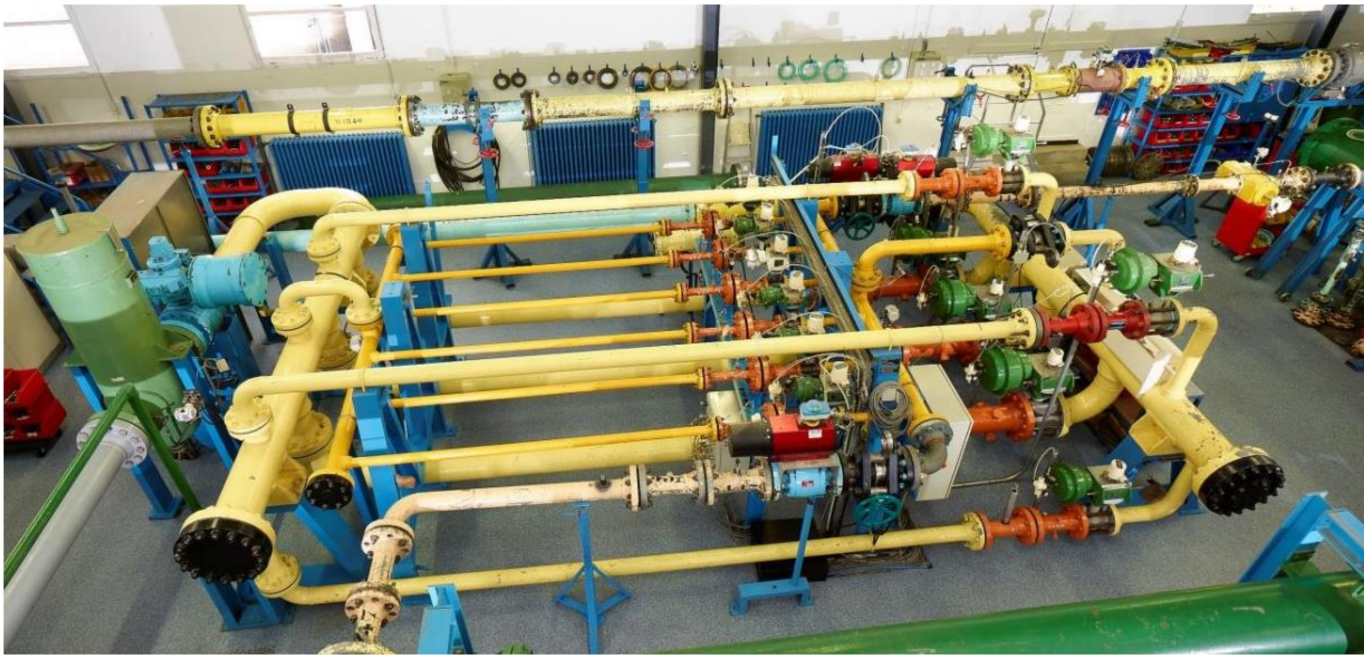

Figure 6. A photo of the CESAME test bench.

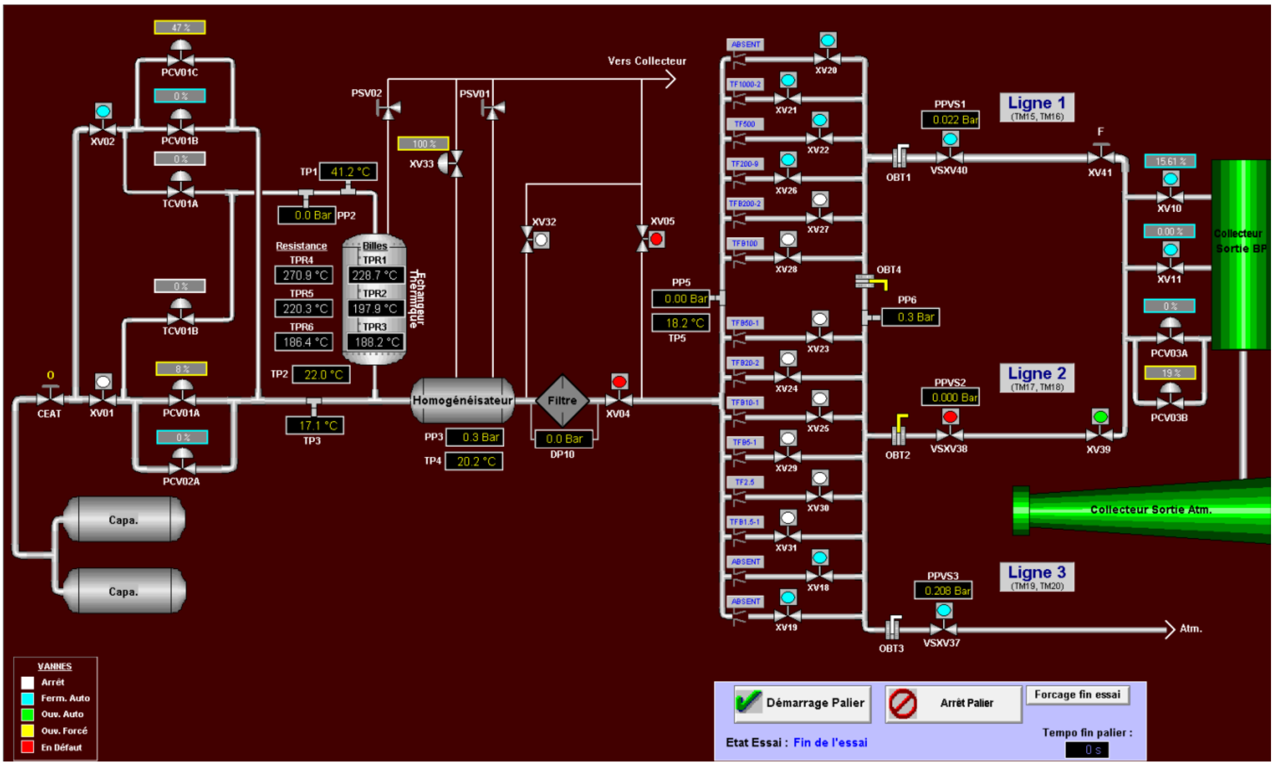

Figure 7. Synoptic of the test bench at CESAME.

The main specifications are given in Table 7 below.

Table 7. Test rig technical specifications.

Specifications	Value
Operation mode	Open loop
Static pressure range	In the range between 1 to 50 bar for the nozzles n° 1 to 9 In the range between 1 to 40 bar for n° 10/11 and 12.
Maximum flow rate	600 $m^3\ h^{-1}bar^{-1}$ (30 000 Sm^3/h) for [40, 50] barg 2000 $m^3\ h^{-1}bar^{-1}$ (80 000 Sm^3h^{-1}) for [1, 40] barg
Minimum flow rate	8 $m^3\ h^{-1}$ in standard conditions
Maximum diameter of the Device Under Test (DUT)	400 mm
Maximum length of straight tube upstream and downstream the DUT	40 m
Accredited uncertainty (k=2)	Critical flow sonic nozzle: 0.21%

The simplified Process Flow Diagram (PFD) of the calibration bench is shown in Figure 8. As shown, the calibration bench uses a dedicated compressed air generation and storage system essentially comprising the following components:

- A compression system.
- Three storage tanks totalling 100 m^3 at a pressure of 200 bar (1).
- Auxiliaries: coolers, desiccants, oil separators.
- Shut-off and control valves with dual valve system (cold air - hot air) for low and high flow rates (2) and (3).
- Accumulation heater in which part of the air used is reheated (4).
- Mixer in which heated air is mixed with cold air coming directly from the tanks (5).
- Five micrometer filter (6).
- A double bundle of pipes carrying 12 nozzles to create the flow through the device to be tested. Shut-off valves behind each nozzle allow the flow rate to be varied for each nozzle configuration.
- One set of 12 sonic nozzles numbered from 1 to 12 with nominal flow rates of 1.5, 2.5, 5, 10, 20, 50, 100, 200, 200, 500, 1000, 1000 $m^3\ h^{-1}\ bar^{-1}$ (7).
- Three test lines of different lengths and diameters, including one for atmospheric pressure (8).
- Pressure-relief valves to regulate the pressure in the appliance under test (9).
- A 10 m^3 capacity, 2 m diameter buffer tank for safety valve flow tests (10).

By opening the shut-off (2) and control (3) valves, a stable pressure can be obtained upstream of the sonic nozzles (7). Depending on the device being tested, one or more nozzles may be used at the same time. The pressure in the device (8) downstream of the nozzles is regulated by the downstream control valves (9). The air is then released into the atmosphere via an acoustic airlock.

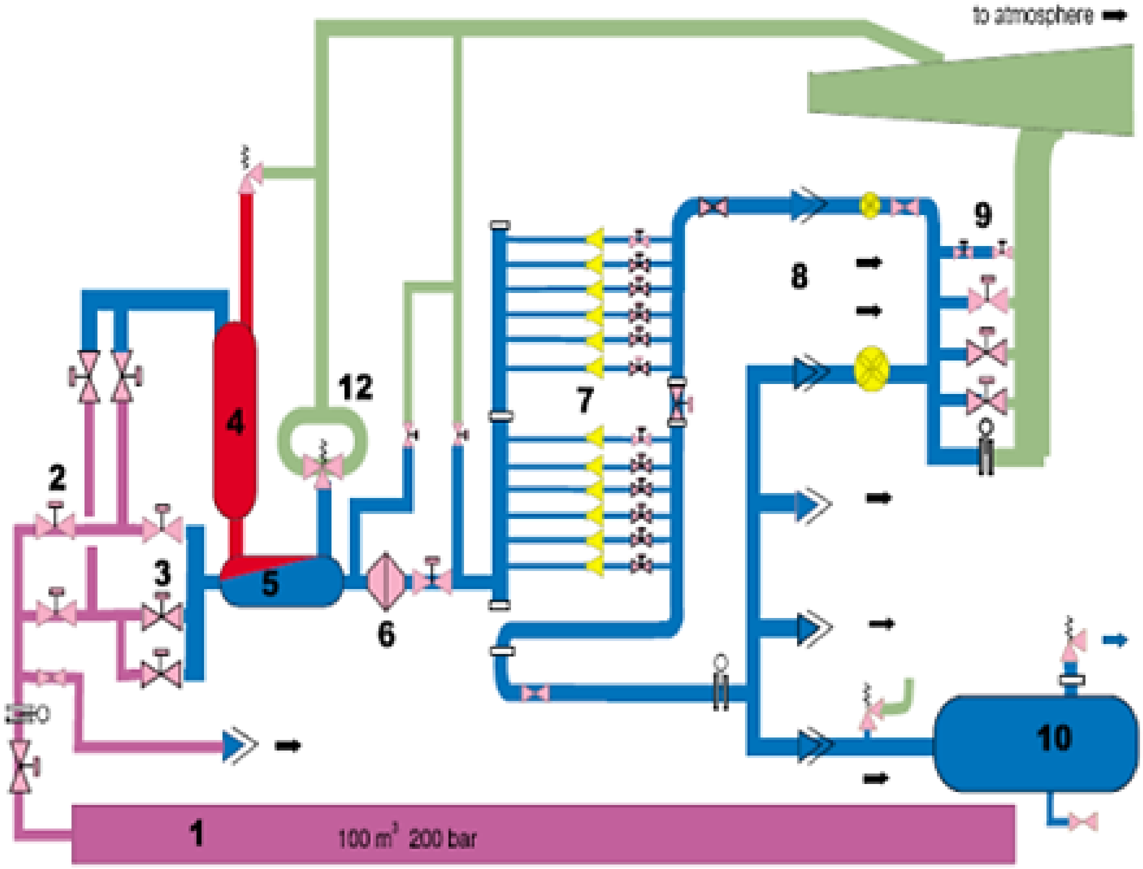

Figure 8. The calibration bench: Process Flow Diagram (PFD).

2.5.2. ENAGAS calibration test bench

Description of the lab

Enagás central Laboratory (Zaragoza, Spain) has three specialized laboratories accredited by the National Accreditation Body, ENAC:

- Gas meter laboratory.
- Gas analysis and quality laboratory.
- Instrumentation laboratory.

The *gas meter laboratory* includes:

- High-Pressure Meter Calibration Laboratory (LACAP) - Figure 9.
- Air Meter Calibration Laboratory.
- Household meters tests for end users.

Additionally, a new calibration laboratory ready for operation with hydrogen and natural gas is under development. A general description of the main gas meter facilities is reported in the following sections.

Figure 9. Photo of Enagás High-Pressure Meter Calibration Laboratory (LACAP).

A general description of High-Pressure Meter Calibration Laboratory (LACAP) is included as this will be the gas meter laboratory used in the THOTH2 project.

High-Pressure Meter Calibration Laboratory (LACAP)

Technical details

- Test bench type: Closed loop, operating independently from the gas transmission pipeline.
- Fluid: Natural gas, proceeding from near gas transmission pipeline.
- Fluid flow: With an axial blower, actuated by an electrical motor with a frequency variator.
- Gas temperature control: Heat exchangers gas/cool water.
- Calibration runs: Two (not working at the same time).

Main installed components

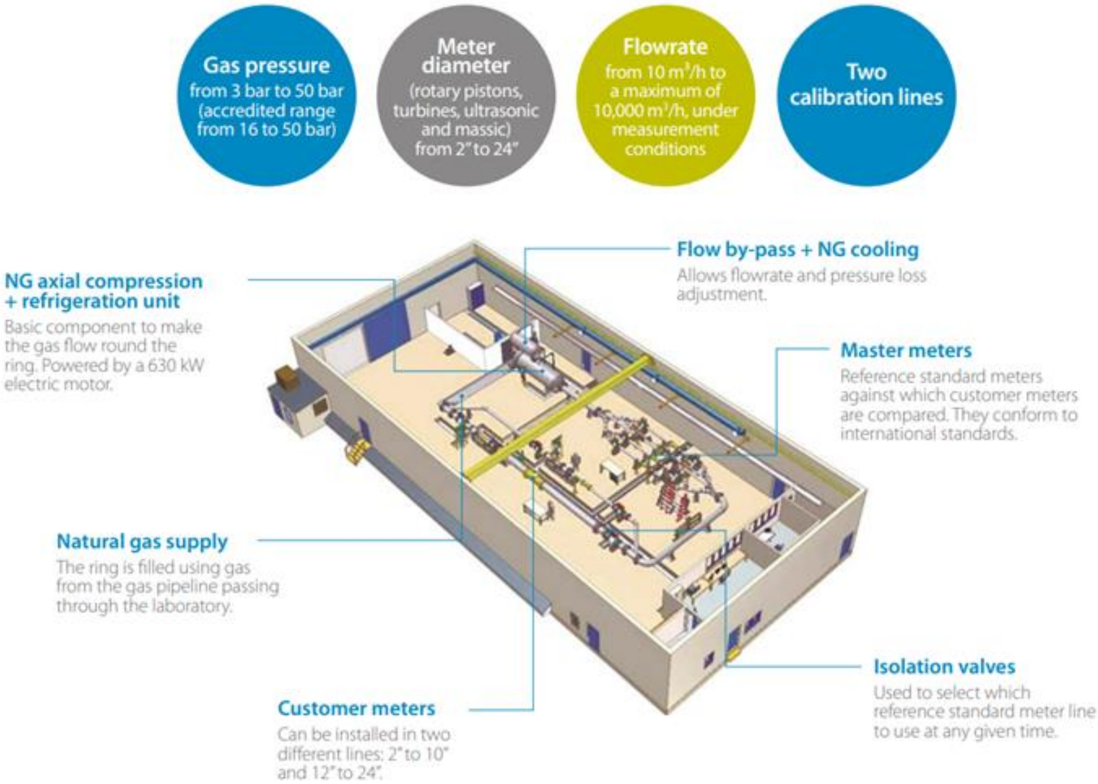

Figure 10. 3D Model of Enagás High-Pressure Meter Calibration Laboratory (LACAP).

Installed measuring devices

Calibration capability

- Reference measurement system, working standards (WS):
  - 4 Turbine G-1600 DN250 ANSI300.
  - 1 Turbine G-400 DN150 ANSI300.
  - 1 Turbine G-160 DN80 ANSI300.
- Traceability to International standards, through transfer standards (TS):
  - 1 Turbine G-1600 DN250 ANSI300.
  - 1 Turbine G-400 DN150 ANSI300.
  - 1 Turbine G-160 DN80 ANSI300.
- Gas meters tested (DUT): Turbine, Ultrasonic (USM), Rotary Pistons, Vortex and Coriolis (under special study).
- Measured signals:
  - 2 × High frequency pulses (HF NAMUR), 0-5 kHz.
  - 1 × Low frequency pulses (LF), REED contact.
  - 1 × Current output: 4-20 mA.
  - 1 × Serial RS485/232 (optional If manufacturer software available, mainly for USM).
- Spools: available with minimum lengths of 5D and 10D for all the nominal diameters.

- Length of calibration runs: 5D+ DUT (3D) +10D.
- Gas composition: continuous on-line measurement at process conditions (gas chromatography).
- Best Measurement Capacity (BMC), uncertainty range on meter error (%) depending on flow rates and pressures: 0.23 - 0.26 % (www.enac.es).
- The Enagás Central Laboratory is supported be a Quality Control System accredited by ENAC (EN ISO/IEC 17025).
- CEM (Spanish National Metrology Institute) associated Laboratory since October 2014.

Hydraulic design

- Test pressure range: 3 to 50 barg (accredited range: 16 to 50 barg).
- Gas temperature range: 18 to 32 ºC.
- Flowrate range: 10 to 10.000 m^3/h (actual conditions).
- Nominal diameters (inches): 2, 3, 4, 6, 8, 10, 12, 16, 20 and 24 (DN50 to DN600).
- Flanges (ANSI): 150, 300, 600.

2.5.3. Gaz-System calibration test bench

The Gas Meter Calibration Laboratory (LWG) operating within the structures of the Polish National TSO GAZ-SYSTEM was launched in 2017. It is the only laboratory in Poland offering calibration of gas meters utilizing high-pressure natural gas as the working medium. The laboratory is accredited by the Polish Center for Accreditation (PCA) for the calibration of turbine and ultrasonic gas meters. The main specifications are given in Table 8 below. The general view is shown on Figure 11 below.

Table 8. LWG technical specifications.

Specifications	Value
Operation mode	Closed loop
Static pressure range	(8 ÷ 54) barg
Volumetric flow range	(8 ÷ 6000) m^3/h
Gas temperature range	(16 ÷ 24) °C
Nominal diameter of the tubing	Nominal: DN50, DN80, DN100, DN150, DN200, DN250 i DN300; Optional: DN350 i DN400
Accredited uncertainty (k=2)	Calibration of turbine meters: CMC = 0,22% for flow rates (1600 ÷ 4000) m^3/h CMC = 0,28% for flow rates (8 ÷ 1600) m^3/h Calibration of ultrasonic meters: CMC = 0,22% for flow rates (1600 ÷ 6000) m^3/h CMC = 0,29% for flow rates (13 ÷ 1600) m^3/h
CMC: Calibration and Measurement Capability	

Figure 11. A photo of the Gas Meter Calibration Laboratory (LWG) by Gaz-System. Source: [1].

LWG is designed for operation in two different modes: in-line and closed-loop. The selection of one of the two operative modes depends on the expected operative conditions:

- When the flow rate is up to 6 000 m^3/h and a pressure drop across the loop is lower than 1 bar, closed-loop mode is used. In this condition, almost 400 kW is required to power supply the blower. This mode ensures better repeatability due to the stability of the calibration conditions, and, therefore is preferable and is used for day-to-day calibrations.
- When the beforementioned conditions are exceeded, the inline mode is possible. The flow is generated by the machines installed in the downstream compressor station. Pressure drops up to 10 bar through the loop can be ensured. This mode may be used in the case of testing mass flowmeters and other specific equipment with higher pressure drop.

The main installed components are shown in Figure 12.

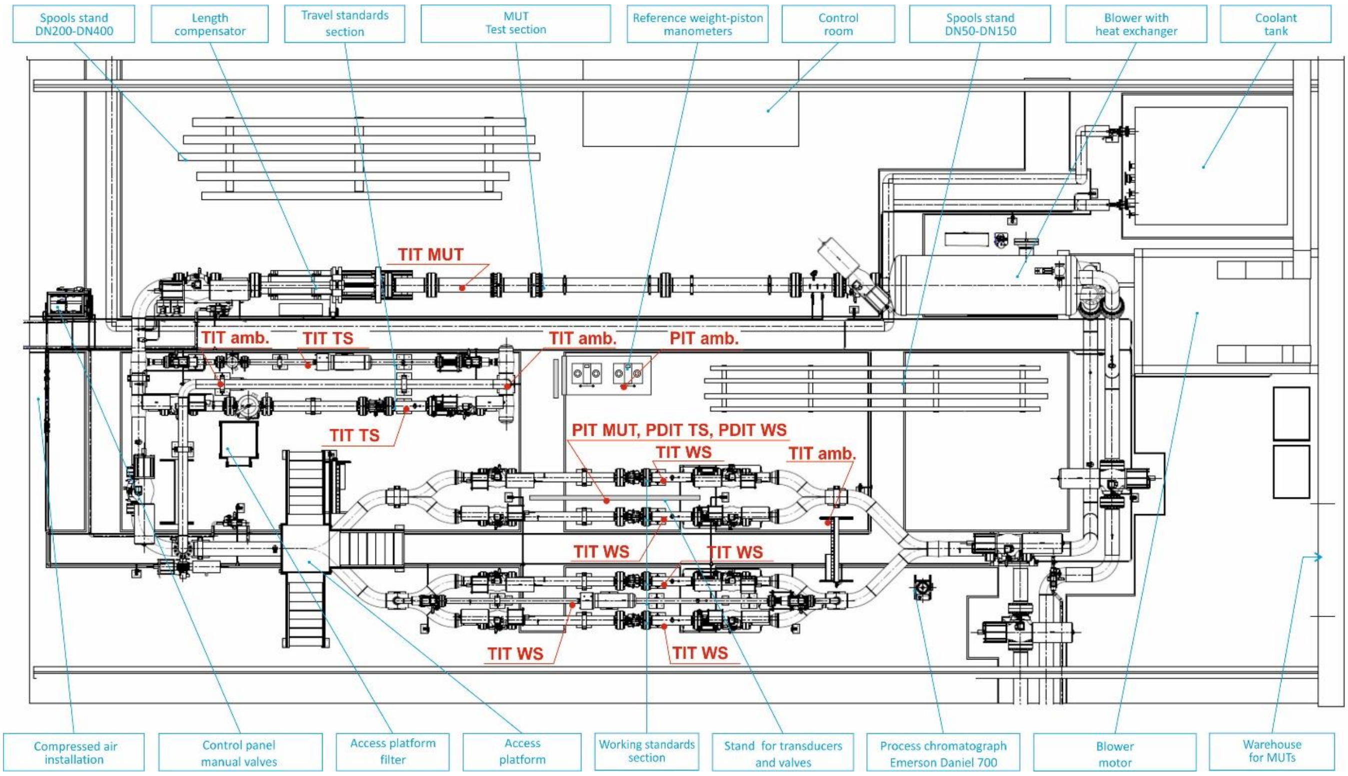

Figure 12. Main installed components and measuring devices of the Gas Meter Calibration Laboratory LWG.
TIT MUT – temperature sensor meter-under-test, TIT TS – temperature sensor transfer standard, TIT WS - temperature sensor working standard, TIT amb. PIT amb. – temperature and pressure sensors ambient conditions, PIT MUT – pressure transducer meter-under-test, PDIT TS – differential pressure transducer «transfer standard to working standard", PDIT WS – differential pressure transducer «meter-under-test to working standard".

The simplified functional layout of the facility is given in Figure 13 below. As shown in Figure 13, in the closed loop configuration, the shut-off valves SV1, SV2, SV3 and SV4 are opened. The gas is moved by the blower and passes through a heat exchanger. Due to compression, in fact, gas has to be cooled down to the set temperature in a dead band between ± 0,1 K during calibration. Once it is cooled down, it passes through the meter under test (MUT) and through the chosen working standards WS1 to WS5. The flow rate through the facility is controlled by varying the rotational speed of the blower up to 6.000 m3/h. Otherwise, in the in-line mode, the valve SV4 is closed while SV1, SV2 and SV3 are maintained open. The gas is compressed by the compressor CS and is injected into the infrastructure by means of the valve SV2. The flow control valves FCV1 and FCV2 regulate the flow rate. Specifically, FCV1 is opened to bypass part of the flow rate directly to the valve SV1. On the other hand, the gas passed through FCV2 follows the same path as the closed loop configuration. Finally, gas is injected back upstream of the compressor through the valve SV3 and SV1.

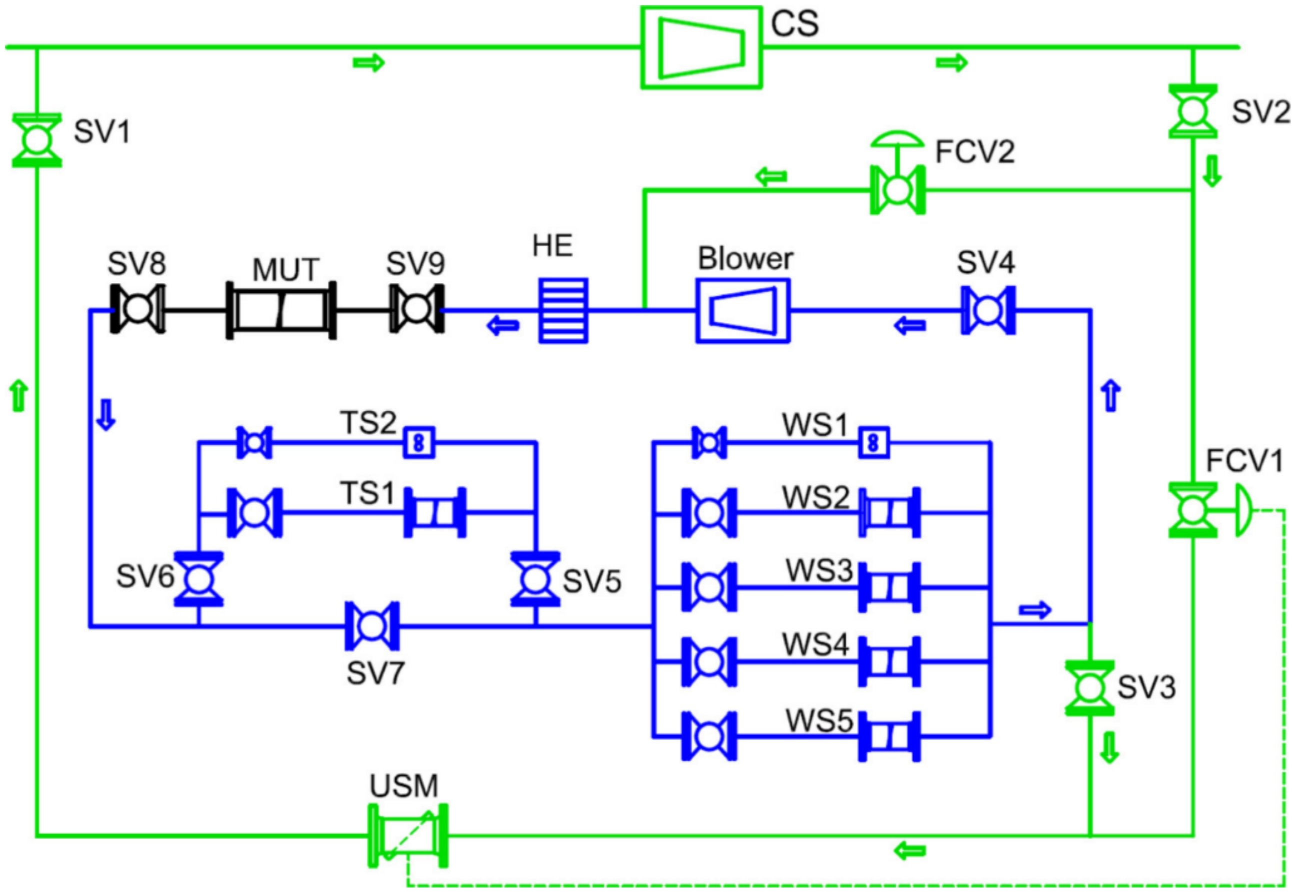

Figure 13. The Process Flow Diagram of the infrastructure available at LWG by Gaz System. CS – one or more of the compressor station machines, Blower – high pressure closed loop blower, MUT – meter under test, WS1 – rotary working standard, WS2 – WS5 – turbine working standards, TS1 – turbine transfer standard, TS2 – rotary transfer standards, SV1 – SV9 – shut off valves, FCV1, FCV2 – flow control valves, HE – heat exchanger. Source: [1]

2.5.4. NaTran calibration test bench

A new test bench has been installed in the R&D department RICE of NaTran. The BMC (Bench (B) of Mid-size (M) gas meter (C)) bench is dedicated to calibrating gas meter with different gas (natural gas – French transmission grid, H2NG, 100% H_2, biogas, carbon dioxide CO_2 - soon).

Figure 14. A photo of the H2 NaTran facility (BMC).

Main specifications of the BMC specification are given in Table 9.

Table 9. BMC technical specifications.

Specifications	Value
Operation mode	Open loop
Static pressure range	Up to 30 barg (ANSI600 flanges)
Volumetric flow range	Up to 2000 Sm^3/h by means of 8 calibrated sonic nozzles
Nominal diameter of the tubing where the meters can be installed	DN20, DN25, DN50, DN80

A simplified PFD of the BMC test bench is shown in Figure 15. As shown in the figure, the BMC bench is composed by:

- 1x 1µm filter
- 1x DN150 buffer upstream the sonic nozzles,
- 1x pressure reducer to regulate the sonic nozzles upstream pressure,
- A combination of 8x different sizes and calibrated sonic nozzles for the reference flowrate,
- Three different sizes control valve outlet the bench to regulate the pressure and flowrate at the meter on test.
- 1x high-precision pressure balances for nozzles upstream pression measurement,
- 1x master densitometer to measure the gas density upstream the nozzles,
- 10x PT100 (class A) temperature sensors
- 1x Digiquartz® pressure sensor

NaTran shall perform calibrations utilizing compressed natural gas of the following meters:

- KROHNE ALTOSONIC V12 DN100
- ITRON Dresser Delta S3-Flow DN150/G250

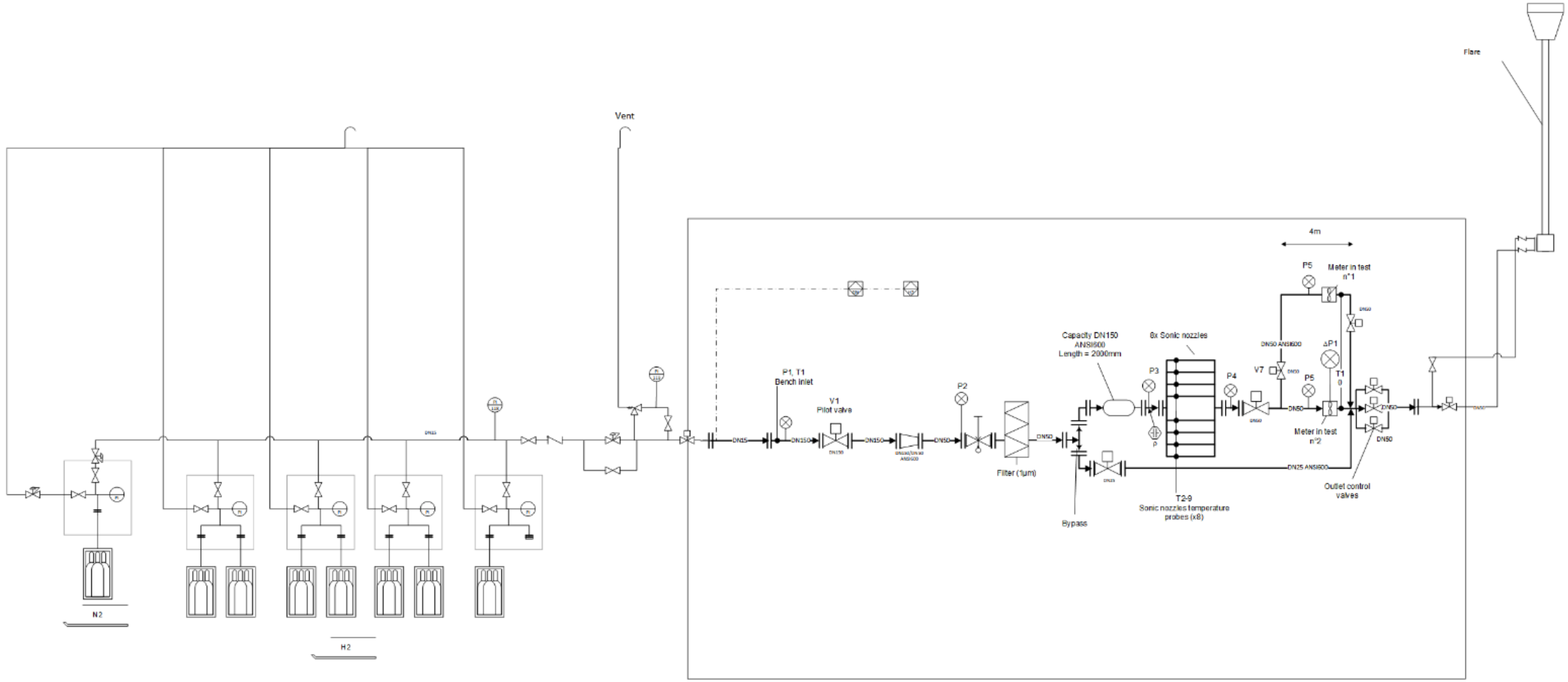

Figure 15. Simplified Process Flow Diagram of the NaTran test lab.

2.6. Calibration of the gas transmission meters: testing procedure

The calibration procedures adopted in the THOTH2 testing benches are described in the next sections.

2.6.1 Cesame calibration test procedure

In accordance with Figure 16, the principle used for the flowmeter calibration bench consists of several steps until calculation convergence is reached.

1. The mass flow rate through the device to be tested is determined using venturi nozzles operating under critical conditions, which have been calibrated beforehand.
2. The mass flow rate of nozzles operating with air is determined from the pressures and temperatures measured under shutdown conditions at the nozzle inlet, and the flow coefficients A, Cd of the nozzles in accordance with standard ISO 9300.
3. The principle is a comparison method consisting of comparing a reference standard flow established using nozzles operating under sonic conditions with a device flow placed downstream of these nozzles.
4. Iterative flow calculation method. The iterative method is interrupted when the relative error is smaller than 10^{-6}.

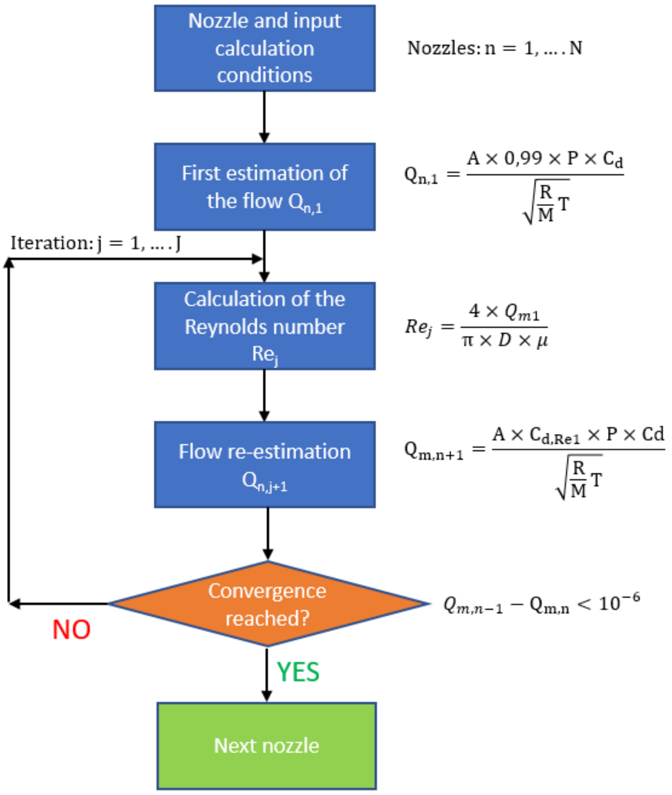

Figure 16. The methodology performed for the flow meter calibration.

Where:
$Q_{n,1}$ is the first estimation of the mass flow rate in kg/s.
$Q_{n,j+1}$ is the estimation of the mass flow rate at the "j+1" th iteration.
A is the sonic nozzle flow discharge area in m².
P is the upstream pressure in Pa.
Re_j is the Reynolds number calculated for the j-th iterative flow.
R is the ideal gas constant and equal to 8314 J/(kmol · K).
T is the testing temperature in K.
M is the molar mass of the gas in kg/kmol.

From an operative point of view, a dedicated test procedure is implemented. Specifically, the following checks are carried out before each test:

- Check of the assembly conditions.

- Tightness check (external and internal leaks, etc.).
- Check that the installation is operating correctly (valves, etc.).
- The various pressure and temperature sensors are checked (fluid temperatures at the nozzle and apparatus must be between 10 and 28°C) and humidity has to be less than 5%.
- Check the frequency delivered by the device.

During the performance of the test, the following activities have to be completed:

- Acquisition of the various parameters that are necessary to perform the calculation.
- Processing and display of measurement results.
- Carrying out the next stage.
- End of the test and validation of the calibration curve by the test manager
- Dismantling the meter in accordance with the instructions for dismantling meters by the manufacturer.

CESAME shall perform calibrations utilizing compressed air of the following meters:

- ITRON Delta S1 G100
- ITRON Delta S3 G250
- COMMON CGR-01/G100
- ELSTER SMRI/G160
- ITRON FLUXI/G160
- ITRON FLUXI/G650

Safety rules during calibration test

Meter calibration at CESAME is carried out using the M1 air test facility.
For safety reasons, people working on this bench must comply with the following requirements:

- Wearing PPE (gloves, safety shoes)
- Earmuffs must be worn during tests
- Use of tools reserved for skilled personnel

As for bench safety, there's an emergency stop button that must be activated by a competent person in response to a situation that could become critical and compromise safety and/or property. Such situations include:

- Unplanned or uncontrolled pressure increase
- Obligation to leave the premises as soon as possible

When this emergency stop button is pressed, the following results are obtained:

- All mechanical components are switched off
- Drain all circuits
- Heater off

2.6.2 ENAGAS calibration test procedure

The operating and calibration procedure in Enagás Laboratory consists of the following steps:

- Delivery of meters, spools and accessories in the Laboratory warehouse.
- Inspection for external damage and checking of technical characteristics and documents. If necessary, issues reporting.
- Transfer to the Laboratory building for temperature assimilation purposes.
- Visual check of meter (and customer spools if any) for internal damage or special dirt that could affect the calibration or any part of the test bench. If necessary, issues reporting.
- Install DUT and spools in the suitable calibration run. If delivered by the customer, a flow conditioner can be installed upstream the DUT (minimum at 10D).
- Connect the pressure and temperature transmitters. Absolute pressure is measured at DUT pr-connection. Gas temperature is measured with Resistance Temperature Detector (RTD). Optionally, pressure loss across the DUT is measured.
- Pressurize the loop at the setup value (or at the higher if more than one pressure is required). Later, pressurize and leak-test the calibration run.
- Connection and check the DUT signal outputs to be used.
- After checking the required signals, the ball valves in the calibration run are opened, and the maximum flow rate, Q_{max} is reached. After that, wait for flow rate and gas temperature stabilization in the loop.
- Start calibration at Q_{max} ±5 %. The calibration computer records all the signals from the Programmable Logic Controller (PLC). Gas volume passes through WS and DUT, and error % at the flow rate is calculated. Three repetitions of the error calculation are recorded.
- Stabilization, measurements, repetitions and error calculation are performed in the same way for the consecutive flow rates until Q_{min} is reached. Standard calibration is for 6 flow rates: 100 %, 70 %, 40 %, 25 %, 10 % and 5 % of Q_{max}.
- Other flow rates and number of repetitions can be performed under request.
- If another pressure has been required, the loop and calibration run pressure is decreased. All the process is repeated in the same way until the minimum calibration pressure is achieved.
- After each calibration, the Weighted Mean Error (WME) is calculated, following International Organization of Legal Metrology (OIML) R137. If necessary, meter adjustment is performed to reach the WME as near to zero as possible.
- If more than one calibration pressure has been required, the adjustment is performed at the pressure indicated by the customer (usually the nearest to the pressure in field operation).
- Verify the adjustment at one flow rate (check point); more check- points can be tested under request.
- Depressurize the calibration run, inertize with nitrogen and remove DUT, spool pieces and accessories.
- Issue the Calibration Certificate following EN ISO/IEC 17025.
- Box up DUT and accessories, using the same packaging, boxes, etc. the goods arrived with.
- Storage in the Laboratory warehouse until collection.

Safety rules during calibration test

Enagás LACAP Laboratory is accredited by the National Accreditation Body (ENAC) according to the UNE-EN ISO/IEC 17025 standard, and safe operation of gas meters calibration is carried out according to procedures and instructions, and controlled by specific design and systems:

1. Procedures and instructions, stablishing for example:

- Authorisation for access to the calibration room (ATEX area) is required.
- Special activities shall be performed by two operators.
- ATEX clothing and personal protective equipment (PPE) is required.
- Use of portable gas detectors is required during the works.
- Access with non-ATEX devices (mobile phones, etc.) I not possible.

2. Design and systems:
   - All equipment in the calibration room has ATEX area certification.
   - Ventilation system for constant air renovation in the room. The operation of this system is controlled in case of gas or smoke alarm.
   - Gas detection system.
   - Smoke detection system.
   - Emergency stop in the calibration area and fire alarm button.
   - Venting, blanketing and vacuum system.
   - Control and monitoring system. This system is specially designed requiring signals to enable the operation of instruments, etc.
   - Alarms.
   - Non sparking/ATEX Tools.

Enagás shall perform calibrations utilizing compressed natural gas of the following meters:

- KROHNE ALTOSONIC V12 DN300
- Emerson Rosemount 3418 DN300
- ITRON Dresser Delta S3-Flow DN150/G250
- Honeywell Q.Sonic-max DN100

2.6.3 Gaz-System calibration test procedure

The flowchart illustrating the process of calibration is shown on the Figure 17 below. Calibrations are performed in accordance with the validated calibration procedures JB-PW-01 and JB-PW-04 (up-to-date versions), in the permanent premises of LWG. Both calibration procedures utilize the use of the specially selected/constructed reference standard meters (4 x turbine meters SM-RI-2-X G1000 and 1 x turbine meter CGT-2 G160) to calibrate the meter under test (MUT) in a close loop mode. The error determination is based on the comparison of the gas volume indicated by the reference meter(s) and the gas volume indicated by the MUT, after calculations of corrections concerning temperature and pressure differences in the reference meter(s) and the MUT. The calibrations are performed by means of the high frequency pulse output of the MUT. Reported calibration uncertainty is evaluated based on the laboratory's uncertainty budget including the CMC (type B uncertainty components determined by non-statistical methods) and the repeatability of the MUT (type A components determined by statistical methods).

Re-calibrations of the laboratory's reference meters (1 x rotary-piston prover RM-TS IRPP G250 and 1 x turbine meter SM-RI-X G1000) are carried out on a yearly basis (currently in PTB, Germany, previously in FORCE Technology, Denmark) ensuring traceability to the harmonized European gas cubic meter for natural gas established by Eurega Consortium. As shown in the previous section, the CMC uncertainty of these calibrations ranges from 0,15% to 0,18%. Pressure, differential pressure and temperature sensors/transmitters are re-calibrated within one-year or two-year intervals in the accredited Polish laboratories, traceable to the Polish National Metrology Institute GUM.

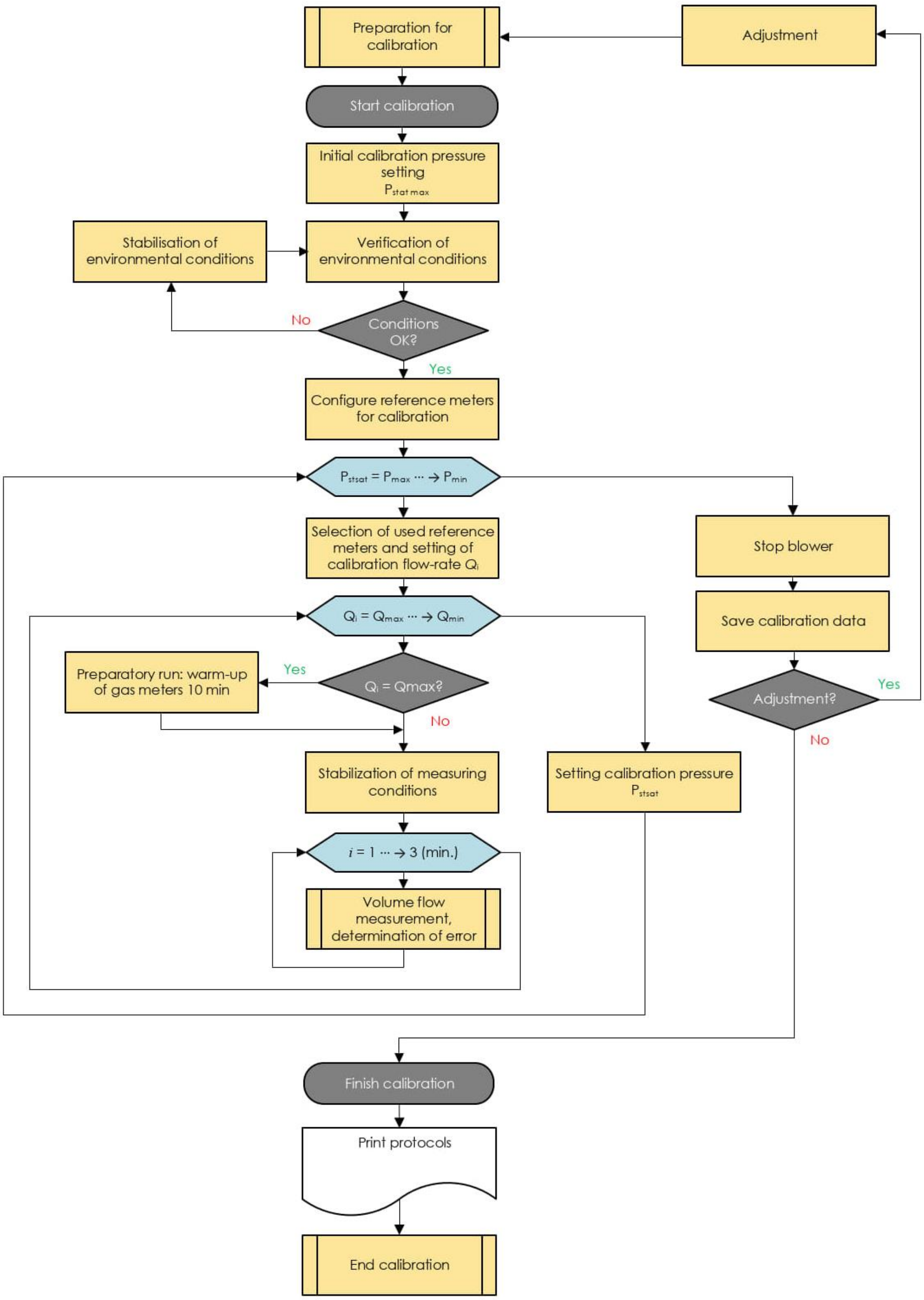

Figure 17. The Flowchart of the Gas Meter Calibration Laboratory LWG.

Gaz-System shall perform calibrations utilizing compressed NG of the following meters:

- Rotary meter ITRON Delta S3 G250
- Rotary meter COMMON CGR-01 G250
- Turbine meter Elster SM-RI G650
- Turbine meter ITRON Fluxi G650

During the calibrations following requirements apply:

- The lubricating mechanism (oil pump) of the turbine meters will not be used.
- During the calibration nominal pressure shall be maintained to within ±0,4 bar of the set value;
- During the calibration nominal flow shall be maintained within ±3% of the set value;
- Only designated High-Frequency (HF) interfaces of the meters will be used for data acquisition;
- The measurement time for each flow point will correspond to the statistically significant number of HF pulses, registered by the laboratory's data acquisition system. The Minimum number of registered HF pulses shall amount to 10'000.
- The meters will be calibrated in the temperature range appropriate to laboratory's standard procedure, provided corresponding uncertainty corrections are implemented. Together with corresponding inlet /outlet spool pieces and pressure dumpers, if appropriate. In fact, in closed loop with turbine master meters, the influence of pulsations produced by rotary gas meter operations is significant. Therefore, inlet/outlet pressure dumpers have to be properly sized and installed.
- The temperature sensor will be mounted in the outlet spool piece, two times the nominal diameter (2xDN) downstream of the turbine meter, and 2xDN upstream of the rotary meter;
- The pressure sensor will be connected to the appropriate pressure tapping on the meter body marked "Pr".
- Each meter will be conditioned at its maximum calibration flow rate for at least 10 minutes before starting the first measurement.
- Prior to the calibrations, the meters will be stored for at least 8 hours on the premises with the ambient temperature converging with the calibration temperature (temperature difference of less than ±2 °C is recommended).
- After calibration is finished, the inlet and outlet of the travel meters will be sealed with the appropriate stickers, then meters will be sheathed into the transportation boxes or fixed on pallets, prepared for shipping and stored according to the LWG internal procedures until dispatching.

Safety rules during calibration test

Within GAZ-SYSTEM, laboratory activities are carried out in compliance with a comprehensive set of safety procedures and instructions. A detailed procedural framework, titled *"Organization of Work on Operation of the Transmission Network",* governs various aspects of operations, including but not limited to:

- Authorization and responsibility matters.
- Designation of hazard zones.
- Principles of work organization, encompassing hazardous gas work, maintenance work, emergency work, non-standard tasks, particularly hazardous work, and construction activities conducted in explosion hazard zones.
- Work orders, including issuance, documentation, and supervision.

- Procedures for work performed by external contractors.
- Occupational health and safety requirements, including safety closures, protective equipment, and work tools.
- Fire protection and environmental protection measures, including fire extinguishing systems and methane detection systems.

In addition, a complete set of exploitation instructions is in place for specific laboratory equipment, covering, but not limited to, the following categories:

- Fire protection equipment.
- Gas detection systems.
- Alarm and signalization devices.
- Lifting and handling equipment.
- Access control and monitoring systems.

Furthermore, dedicated guidelines are being implemented for the operation of individual laboratory systems and machinery, including pressurized vessels, blowers, valves, piping, data acquisition (DAQ) systems with measuring instruments, computer systems, networks, and gas meters from various manufacturers.

A specialized *"Fire Safety Instruction Based on the Technological Process of the Gas Meter Calibration Laboratory (LWG)"* is also implemented, ensuring safety protocols are tailored to the specific equipment and processes used within the laboratory.

Additionally, calibration procedures incorporate safety mechanisms and algorithms designed to mitigate risks and prevent contingencies or emergencies. While these procedures do not contain a separate section exclusively dedicated to safety, they are developed with safety considerations integrated throughout, referencing the relevant procedures and instructions addressing specific safety concerns.

The existing safety framework is suited to operations involving high-pressure natural gas. However, in the case of hydrogen or hydrogen-natural gas mixtures, establishing specific safety requirements necessitates a thorough understanding of the system's architecture, including hydrogen supply, mixing, and recovery or venting processes. These safety aspects must be determined during the design phase.

3. TESTING OF GAS DISTRIBUTION FLOW METER IN THE PRESENCE OF H_2

The testing of the gas distribution meters selected during the activities of Task 2.1 and indicated in Deliverable D2.1 will be performed in accordance with the protocol developed in Task 2.2 and indicated in Deliverable D2.2.

Testing activities include ageing and calibration. Specifically, calibrations will be performed in NaTran, INIG, and METAS test benches while ageing will be performed at the INIG facility as for workload distribution defined in WP2.

Objective of the test: The testing activity will consist of limit tests, i.e., tests about the accuracy of state-of-the-art technology (e.g., diaphragm, ultrasonic and thermal mass) under different H_2 concentration conditions.

Operative procedures adopted during the experimental activities: Two activities will be performed to achieve the objective:

1. Ageing of the devices with 100%vol. and 25%vol. H2 in NG or CH_4 based on the final decision taken in accordance with the Manufacturers. It has to be noted that using CH_4 will allow reproducibility regardless of the composition of the NG, that slightly varies in EU countries based on its origin. The selection of the testing gas will be indicated in the final report including the experimental results. The test duration and conditions are indicated in the specific protocol (Deliverable D2.2).
2. Calibration of the measuring devices. Different testing fluids will be used for calibration:
    a. NaTran: pure NG
    b. METAS: pure H_2
    c. INIG: 25%vol H_2 in NG.

The detailed testing plan and the procedure are reported in Deliverable D2.2.

In the next sections, information about the measurement equipment, the materials, and the operative procedures to respect the metrology requirements indicated in WP2 will be shown. The safety measures adopted throughout the testing activities are also included in the discussion.

3.1. Ageing of the gas distribution meters: introduction

Ageing of the gas distribution meters will be performed in INIG according to the protocol described in Deliverable D2.2.

The operative conditions maintained during ageing are reported in Table 10.

Table 10. The operative conditions of performed ageing.

Ageing	Laboratory	Fluid	Pressure (kPa)	Temperature (C)	Period of time
1	INIG	25% H_2/CH_4 (vol/vol)	2 ÷ 8	ambient	4+4
2	INIG	100% H2	2 ÷ 8	ambient	4+4

3.2. Ageing of the gas distribution meters: test bench and main components

The Stand Gas Meter Test for H2 (SGTW) test of INIG bench will be used. The test bench enables to perform durability (aging) tests of gas meters and measuring equipment using mixtures of CH4 with H2 and 100% H2 in both flow and static conditions in a closed loop. A picture of the SGTW is shown in Figure 18.

Figure 18. SGTW test bench for gas meters and measuring equipment ageing test.

The PFD of the test bench is shown in Figure 19. The installation was made of seamless pipes and fittings using steel intended for flammable fuels connected by welding. The inlet and outlet manifolds are made of 2" pipes, while the branches to the gas meters are made of 1 1/4" pipes. The piping system was integrated into three four-level racks, which enabled stable installation of the tested gas meters. Regulating and flow-cutting ball valves are placed on the branches of the installation's collectors. In order to measure the temperature at the inlet to the gas meters, connectors with a built-in measuring sleeve for mounting temperature sensors were made.

The installation is also equipped with an analogue pressure measurement on the supply manifold. The station operates on overpressure generated by a side-channel blower equipped with additional cleaning filters located at the blower inlet. The pressure in the installation does not exceed 10 kPa, and during operation, it varies in the range of 2÷8 kPa. To eliminate the problem of transmitting vibrations from the blower supplying the entire system, compensators were installed on the supply side and on the side discharging gas from the blower. The required volume flow is obtained by smoothly adjusting the blower speed using a frequency converter.

The test bench consists of three installations that operate in a closed system. Filled with gas from a gas cylinder (for 100% H_2 and for 25%H_2/CH_4), each installation circulates gas in the amount of approximately 0.15 m^3 enriched with H_2. The datasheet of the blowers adopted and their performance curves are reported in the Appendix.

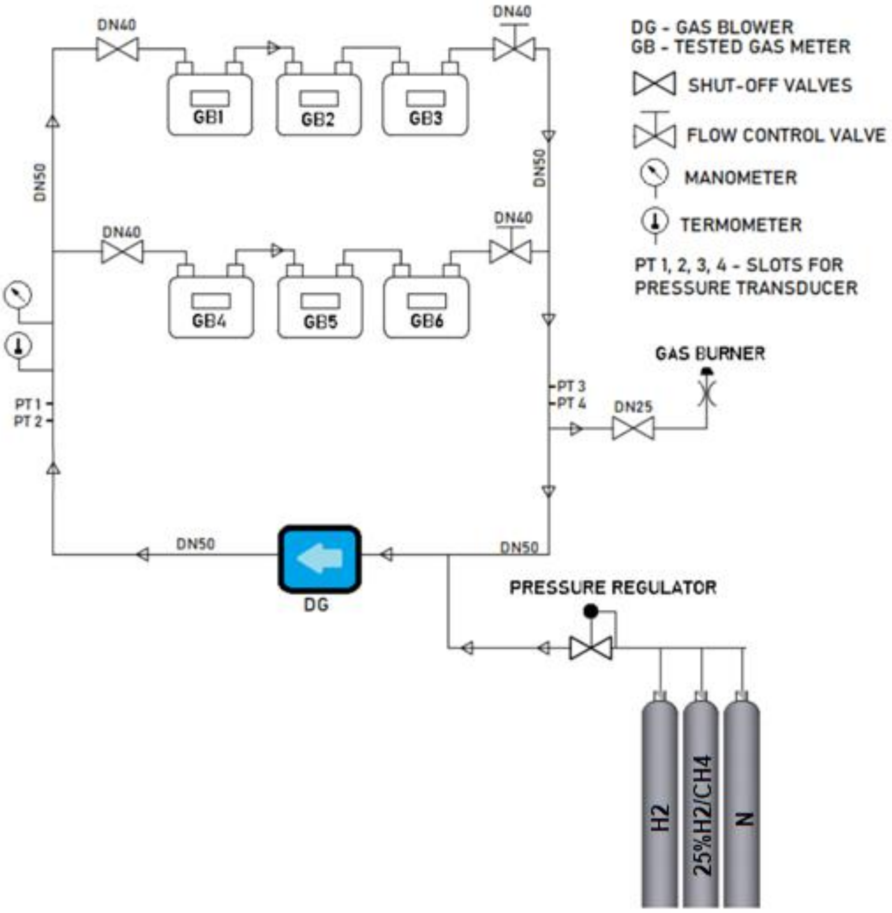

Figure 19. Process Flow Diagram of an SGTW test bench for gas meters and measuring equipment ageing test.

The list of the main installed components in the SGTW test bench is reported in Table 11.

Table 11. List of the main equipment of the SGTW test bench.

Equipment type	Producer	Type	Range
Blower	Verdichter-Blower Effepizeta	CL 7/01 VG MS	up to 65 m^3/h up to 130 m^3/h
Gas mixer (gas cylinders)	INiG	-	max. 7 gas components
Switching and regulating valves	other	-	-
Pressure regulator	other	-	2 kPa

The instrumentation is indicated in Table 12.

Table 12. List of the main measuring devices of the SGTW test bench.

Equipment type	Producer	Type	Range
Pressure Gauge	WIKA	-	0 ÷ 10 kPa
Thermometer	KFM	-	-10 ÷ 50 °C

3.3. Ageing of the gas distribution meters: testing procedure

The ageing test refers to the step 2 and 4:

1. Calibration of all the gas meters with different gases (25% H_2/CH_4) as references.
2. Exposure to 100% H_2 and 25% H_2/CH_4 for a defined period of the meters (See D2.2).
3. Calibration using different gases (25% H_2/CH_4 same measuring equipment as point 1 (See D2.2).
4. Exposure to 100% H_2 and 25% H_2/CH_4 for another defined period (See D2.2).
5. Calibration using different gases (25% H_2/CH_4 same measuring equipment as point 1).

Other steps refer to calibration and are described in Section 3.4.

Description of the implementation of point 2 and point 4:

- In the case of indoor testing, the test installation should be located in a well-ventilated room with a safety system to detect the hydrogen/methane content in the air;
- The test installation should be equipped with a control manometer for pressure measurement to check the pressure in the circuit and ensure that the required working pressure is available;
- Maintain/record required environmental conditions.
- Connect a nitrogen source to the connection stub to flush the test installation, not exceeding the maximum pressure of the meters.
- After flushing the test installation with nitrogen, close the manual valves so that no air enters the installation.
- Open the H_2 or H_2/CH_4 cylinder, adjust the pressure reducer after reopening the previously closed valves to the required working pressure, as indicated by the pressure gauge on the pressure reducer.
- After replacing at least 3 volumes of the test installation (or after letting the gas flow through the circuit for ten seconds or more), close the valves.
- Check the pressure on the pressure gauges installed in the circuit, which should be equal to the required working pressure. If not, use the pressure reducer to adjust the pressure to the required value.
- Note the beginning of the ageing process of the meters with H_2 or H_2/CH_4.
- Regularly check the pressure gauge in the test installation for a drop in pressure and, if necessary, top up the pressure to the required value.
- After the fixed period of ageing, the test installation should be emptied (the manometer could verify the atmospheric pressure in the installation) and flushed with nitrogen, and then the meters could be dismantled.
- Proceed to the calibration of the meters with the gas mentioned above.

Safety rules during ageing test:

- In the case of gas meter ageing test using a mixture of hydrogen with methane and 100% hydrogen, the test are performed in open location (covered from above by a tin roof) perfectly ventilated, so that the risk of creating an explosive atmosphere during an uncontrolled gas leak is negligible.
- All electrical devices are located outside the gas meter installation location.
- Open flames and smoking are strictly prohibited in the vicinity of the testing installation;
- The use of spark-producing devices, including mobile phones, is strictly prohibited in the vicinity of the testing installation;
- All safety mitigation and protective measures must be taken in order to minimize any risk due to the presence of explosive and flammable gases.
- All the testing activities shall be carried out in accordance by an authorized and experienced person;
- The measuring instruments shall be used for their intended purpose and under the conditions specified by the manufacturers and the procedures;
- All the measuring instruments shall be used with the required range and accuracy class and with valid calibration certificates;

3.4. Calibration of the gas distribution meters: introduction

The gas distribution meters will be calibrated in NaTran, INIG and METAS test benches with different fluids.

The calibration will be performed to verify at least the accuracy and time stability (error drift after every ageing period) of the tested gas distribution meters using different gases. More details about the results are reported in Section 8.

3.5. Calibration of the gas distribution meters: test benches

The calibration of the gas distribution meters will be performed in different testing benches as described in the following sections.

3.5.1. NaTran calibration test bench

The same bench as described Section 2.5.4 is used for the distribution gas meter calibration from 0.1 Std.m^3/h to 2000 Std.m^3/h.

For domestic gas meters up to 16 Std.m^3/h a new bench is currently in process, the starting date is the first quarter of 2025. At this moment, only the Piping & Instrumentation Diagram (P&ID) is available (Figure 20). The bench works with dry air / NG /H2NG mixtures /100% H_2/other industrial gases.

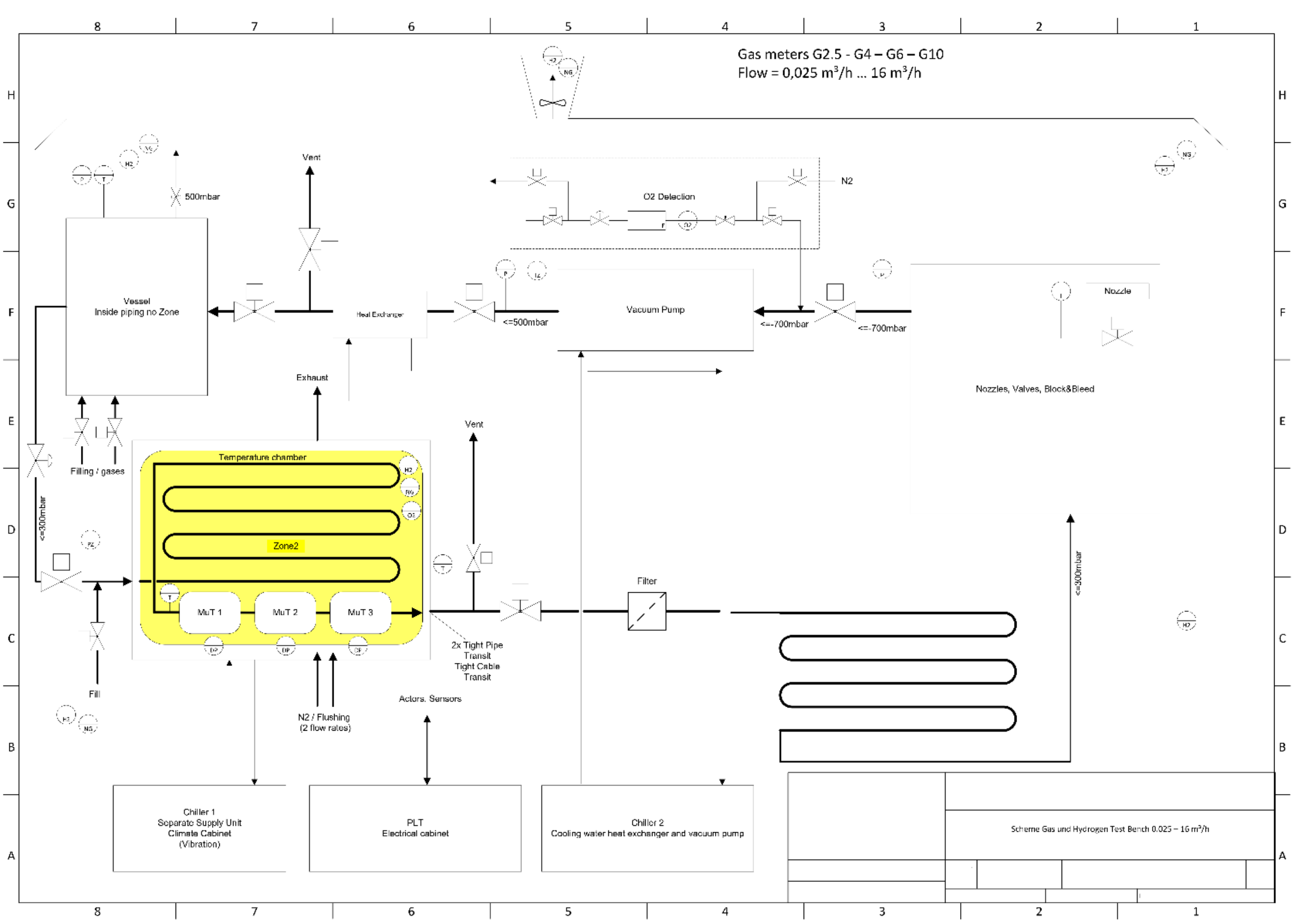

Figure 20. P&ID of the domestic gas meter calibration bench of NaTran.

NaTran shall perform calibrations of the following DSO meters:

- Sagemcom/Siconia ES4evo G4
- Sagemcom/Siconia ES4evo G6
- Flonidan SciFlo G4
- Flonidan SciFlo G6

3.5.2. INIG calibration test bench

The SG25-G test bench is used to perform tests on errors of indications at reference temperature and errors of indications at declared temperature limits using gas mixtures containing H_2 (25%vol). A picture and the PFD of the test bench are shown respectively in Figure 21 and Figure 22.

The test bench is filled with the appropriate medium using a gas cylinder. The gas flow in the loop is forced by an intrinsically safe side-channel blower with speed control achieved by changing the inverter settings or changing the settings of the control valves. The station is equipped with control gas meters: rotary type CGR-

05 manufactured by Common, laboratory wet drum NB15 by Rombach, and wet drum type BSM-1 manufactured by Bessel. Gas meters use the volumetric method for measurement and are generally insensitive to changes in gas composition. The datasheets of the control gas meters are reported in the Appendix.

The gas temperature is measured using Pt100 temperature sensors with accuracy class A according to PN-EN 60751. The test rig uses five pressure transmitters: two of the PC-28 type (gauge pressure in the control gas meter and the tested gas meters) and three of the APR-2000GDP type (differential pressure in the MUT) manufactured by Aplisens. Atmospheric pressure is measured using a VAISALA digital barometer type PTB330.

Figure 21. SG25-G test bench for the gas meter metrology test.

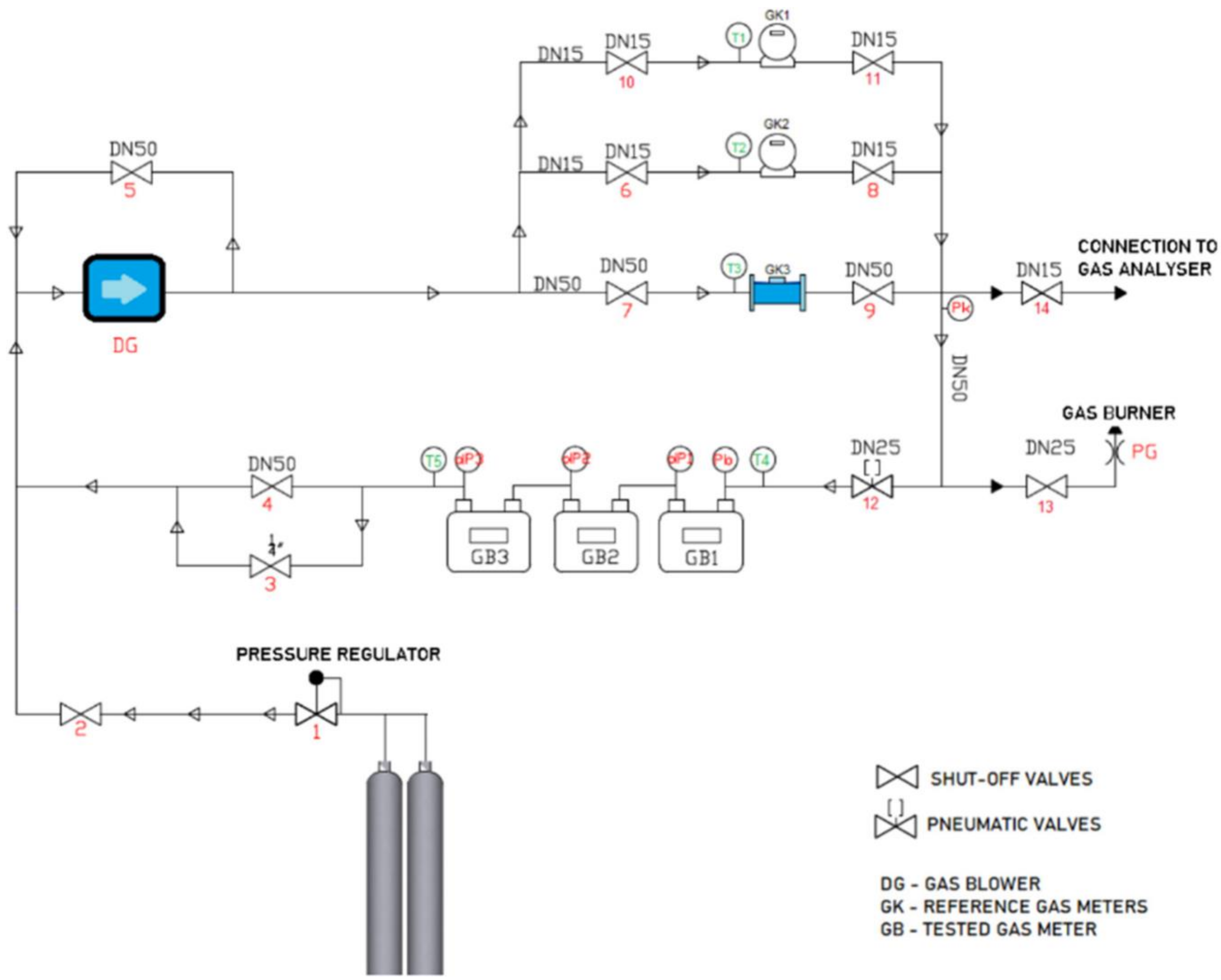

Figure 22. Process Flow Diagram of a SG25-G test bench for a gas meter metrology test.

The list of the main installed components in the SG25-G test bench is reported in Table 13.

Table 13. List of the main equipment of the SG25-G test bench.

Component type	Producer
Blower	Verdichter-Blower
Gas cylinders	SIAD or other
Switching and regulating valves	other

The list of the instrumentation installed is reported in Table 14.

Table 14. List of the main measuring devices of the SG25-G test bench.

Equipment type	Producer	Type	Range
Wet gas meter	Bessel	BSM-1	0,02 ÷ 1,25 [m³/h]
Wet gas meter	Elster	NB15	1 ÷ 16 [m³/h]
Rotary gas meter	Common	CGR-05	0,5 ÷ 100 [m³/h]
Gauge pressure transducers	Aplisens	PC-28 PD Exi	0 ÷ 10 [kPa]
Differential pressure transducers	Aplisens	APR-2000G PD Exi	0 ÷ 2,5 [kPa]
Digital barometer	Vaisala	PTB330	50,0 ÷ 110,0 [kPa]
Temperature sensors	Termoaparatura Wrocław	Pt100	-50 ÷ +260 [°C]

The main specifications of the SG25-G are given in Table 15 below.

Table 15. SG25-G technical specifications.

Specifications	Value
Operation mode	Open / Closed loop
Static pressure range	Up to 10 kPa
Volumetric flow range	Up to 100 Sm³/h
Nominal size of the meters that can be installed	G65
Accredited uncertainty (k=2)	Wet gas meter: 0,25% Wet gas meter (Elster): 0,27% Rotary gas meter: 0,22%

3.5.3. METAS calibration test bench

METAS will perform calibration with pure H2 in a new facility that is a pressure, volume temperature and time (pVTt) primary gas flow standard with an expected expanded uncertainty of 0.3 % (k=2). The standard will span the flow range from 0.1 L/min to 2000 L/min using several collection tanks and a diverter valve system. The standard measures flow by collecting gas in a tank of known volume during a measured time interval.

The characteristics of the main components installed in the pVTt test bench are indicated from Table 16 to Table 21.

Table 16. Pressure vessels, heat exchanger.

Producer	Type	min. / max. pressure PS bar	min. / max. temp TS °C	V l	Test pressure PT bar	PED cat.
Barth+Höpfinger	10 L Tank	-1 / +100	0 / +100	10	155	III
Barth+Höpfinger	50 L Tank	-1 / +100	0 / +100	50	155	IV
Barth+Höpfinger	200 L Tank 1	-1 / +100	0 / +100	196	155	IV
Barth+Höpfinger	200 L Tank 2	-1 / +100	0 / +100	196	155	IV

Barth+Höpfinger	200 L Tank 3	-1 / +100	0 / +100	196	155	IV
Alfa Laval	AXP27-50L	-1 / +130	-20 / +150	1.25	195	II
		-1 / +130	-20 / +150	1.2	195	II

Table 17. Safety equipment.

ID	Equipment	Type	Producer	PS bar	Qv H2 Nm3/h	D0 mm	PED cat.
V-81	Safety valve	Typ 4384 H2	Leser	63	5550	10	IV

The outlet from the safety valve is routed to the outside via a separate pipe with an internal diameter of 18 mm.

Table 18. Tubing and piping.

OD mm	Wall thickness mm	ID mm	PS bar	Type	Producer
(1/8") 3.175	(0.028") 0.71	1.76	586	SS-T2-S-028	Swagelok
(1/2") 12.7	(0.049") 1.25	10.2	255	SS-T8-S-049	Swagelok

All lines of the H2-PVTt standard are made of seamless stainless-steel tubing from Swagelok.

The diameter of the H2 flow lines is 1/2" and the pressure measuring lines are made of 1/8" tubes.

Table 19. Valves.

ID	Equipment	ID / mm	PS / bar	min. TS / °C	max. TS / °C	Type	Producer
V-11	Solenoid valve	8	350	-20	80	16138032	ValEvo
V-2x	Check valve		355	-23	93	SS-CHS8	Swagelok
V-3x	Needle valve ½"	9.5	295	-53	93	SS-18RS8	Swagelok
V-4x	Ball valve ½"	10.3	413	-40	121	SS-AFSS8	Swagelok
V-5x V-13	Ball valve ½" actuated	10.3	127	-28	65	SS-63TS8	Swagelok
V-6x	3-way ball valve ½"	10.3	103	10	65	SS-45XS8	Swagelok
V-64	3-way ball valve 1/8"	2.36	172	-53	148	SS-41GXS2	Swagelok
V-9x	Ball valve 1/8"	2.36	172	-53	148	SS-41GS2	Swagelok

Table 20. Pressure regulators.

ID	Equipment	P_{in} bar	P_{out} bar	min. T_S °C	max. T_S °C	Type	Producer
PR-01	Pressure Regulating Valve (PRV)	300	80			BT2000 300	Messer
PR-11		300	10			BT2000 10	Messer
PR-12		400	50	-15	80	RSHN4	Swagelok
PR-13		70	28	-15	80	RSN4	Swagelok
PR-14		70	7	-15	80	RSFD8	Swagelok
PR-15		69.8	17.2	-15	80	KHF1	Swagelok

Table 21. Instrumentation.

ID	Equipment	FS Range bar abs	Overpressure bar	Acc. %FS	Temp. °C	Type	Producer
P1	P-Transmitter	0 to 1	2	0.02	-40...90	PAA-33XEi	Keller AG
P2	P-Transmitter	0 to 3	5	0.02	-40...90	PAA-33XEi	Keller AG
P3	P-Transmitter	0 to 10	20	0.02	-40...90	PAA-33XEi	Keller AG
P4	P-Transmitter	0 to 100	200	0.02	-40...90	PAA-33XEi	Keller AG
P5	P-Transmitter	0 to 10	20	0.02	-40...90	PAA-33XEi	Keller AG
P6	P-Transmitter	0 to 60	120	0.02	-40...90	PAA-33XEi	Keller AG

All temperatures (T1 to T10) are measured with intrinsically safe, class A Pt100 resistance thermometers. The control system is based on a National Instrument (NI) cRIO controller, and the calibration software is written in LabView.

The new pVTt system is under construction. pVTt systems have been used as primary gas flow standard by several national metrology institutes and other laboratories for more than 40 years. The standard consists of a flow source (pressurised bottles), valves for diverting the flow, collection tanks, a vacuum pump, pressure and temperature sensors, and a critical flow Venturi nozzle which will isolate the meter under test from the pressure variations in the downstream piping and tanks. The main specifications are indicated in Table 22.

Table 22. pVTt technical specifications.

Specifications	Value
Operation mode	Open loop
Pressure range	(2 to 50) bar, upstream of critical nozzle, close to atmospheric downstream
Mass flow rate range	(0.1 to 168) g/min = (1.2 to 2000) NL/min = (0.071 to 120) Nm3/h, for the primary standard, increased flow range using secondary standards
Temperature range	Ambient conditions
Nominal size of the meters that can be tested	G40
Accredited uncertainty (k=2)	< 0.3%
Type of gases	H2, with future options to use premixed H2 blends supplied in gas cylinders
Density of H_2 @ 1013 hPa and 20 °C (STP): 0.084 kg/m^3 = 0.084 g/L	

The P&ID of the infrastructure is shown in Figure 23.

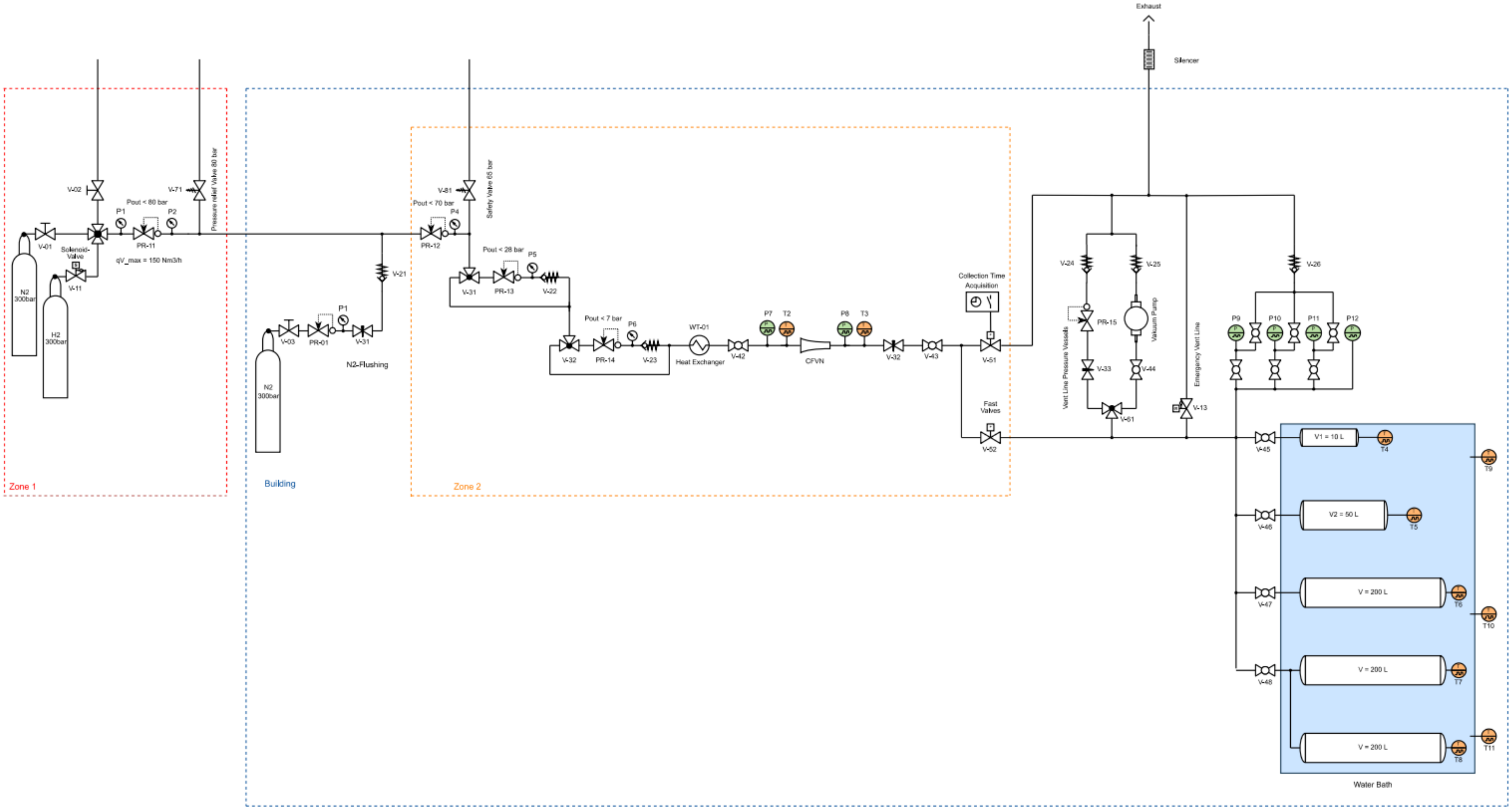

Figure 23: P&ID of the METAS pVTt system.

3.6. Calibration of the gas distribution meters: testing procedure

In the next section the testing procedures adopted in the three laboratories will be described.

3.6.1. INIG calibration test procedure

The flowchart illustrating the process of calibration is shown on the Figure 24 below.

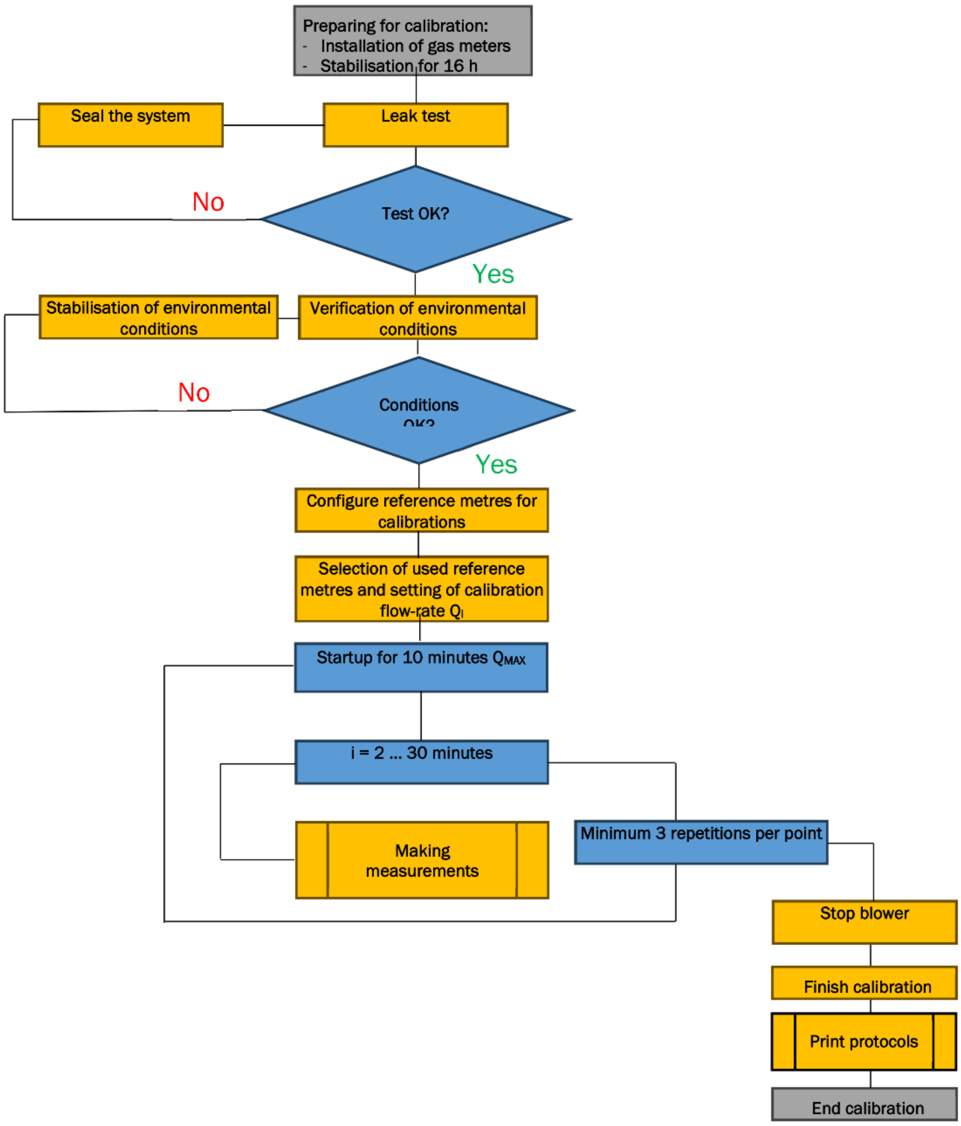

Figure 24. Flowchart of the gas meter calibrations procedure.

During the calibrations following requirements apply:

- During the calibration nominal pressure shall be maintained to within ±0,15 kPa of the set value;
- During the calibration nominal flow shall be maintained within ±5% of the set value;
- The temperature sensor will be mounted in the stub of the first and last gas meter to be tested and on the reference gas meter;

- The pressure measurement of the test gas meter shall be connected at the inlet of the first test gas meter;
- The differential pressure measurement will be connected to the inlet and outlet of each gas meter tested;
- Each meter will be conditioned at the maximum calibration flow rate for at least 10 minutes prior to the first measurement;
- Prior to calibration, the meters will be stored for at least 16 hours in rooms with an ambient temperature coinciding with the calibration temperature (recommended temperature difference less than ±2°C);
- Once calibration is complete, the inlet and outlet of the meters will be sealed with appropriate caps and the meters will then be prepared for dispatch.

Safety rules during calibration test:

- In the case of gas meter calibration using a mixture of hydrogen with methane and 100% hydrogen, the laboratory conducts measurements in a well-ventilated room with a leak detection safety system dedicated to H2NG mixtures and hydrogen in the atmosphere. When the system is activated, the operation of the measuring installation and the ventilation of the room are stopped.
- All electrical devices, such as a circulation blower or frequency converter, are located outside the Ex zone. Where possible, intrinsically safe devices (in ATEX version) are used.
- Open flames and smoking are strictly prohibited in the vicinity of the testing installation;
- The use of spark-producing devices, including mobile phones, is strictly prohibited in the vicinity of the testing installation;
- All safety mitigation and protective measures must be taken in order to minimize any risk due to the presence of explosive and flammable gases.
- All the testing activities shall be carried out in accordance by an authorized and experienced person;
- The measuring instruments shall be used for their intended purpose and under the conditions specified by the manufacturers and the procedures;
- All the measuring instruments shall be used with the required range and accuracy class and with valid calibration certificates;

INIG shall perform calibrations of the following meters:

- Sacofgas EG G10
- Sacofgas EG G16
- Honeywell BK G40
- Honeywell BK G65
- Metersit Domusnext G6
- Metersit Domusnext G25
- Sagemcom/Siconia EF4evo G4
- Sagemcom/Siconia EF4evo G6

3.6.2. METAS calibration test procedure

The gas distribution meters will be calibrated against the critical flow venturi nozzles (CFVN), where the pressure at the outlet of the CFVN will be close to atmospheric conditions. The CFVN will first be calibrated against the pVTt primary standard according to the following procedure.

1. Start the control system, the calibration software and the water bath circulation pump.
2. Purge the test section with nitrogen, depressurize and isolate the test section.
3. Install the nozzle under test (or another DUT upstream of the nozzle), perform a leak test.
4. Define the calibration points in the calibration and control software
    a. (DUT name, nozzle ID, inlet pressure, collection time, no. repetitions)
5. Select and connect the pressure transmitters and the collection volume according to the calibration point (nozzle inlet pressure, flow rate and final collection tank pressure).
6. Close the tank valve V-52, open the bypass valve V-51, open the H2 solenoid valve V-11 and set the nozzle inlet pressure manually according to the calibration point and establish stable flow conditions.
7. Evacuate the collection tank volume to an initial tank pressure lower than 0.1 mbar.
8. Wait for pressure and temperature in the tank to stabilise and acquire initial values P_T^i and T_T^i for the tank. These values are needed to calculate the initial density and the initial mass of gas in the tank m_T^i.
9. Close the bypass valve and during the dead-end time when both bypass and tank valves are closed, obtain a start time t_i. At the same time, acquire the initial pressure and temperature in the inventory volume P_I^i and T_I^i. These values will be needed with the inventory volume V_I to obtain the initial mass in the inventory volume m_I^i. Shortly after the bypass valve is fully closed, open the tank valve.
10. Acquire the nozzle inlet temperature and pressure during the whole collection interval.
11. Wait for the tank to fill to a prescribed upper pressure and then close the tank valve and obtain the stop time t^f during the dead-end time. At the same time, acquire the pressure and temperature in the inventory volume P_I^f and T_I^f and hence the final mass in the inventory volume m_I^f. Open the bypass valve.
12. Wait for stability and acquire P_T^f and T_T^f for the tank and hence m_T^f.

The average mass flow rate $\dot{m}$ can be calculated from the mass balance for the total volume comprising tank and inventory volume during collection time as shown in Eq. (3.1).

$$Q = \frac{\left(m_T^f - m_T^i\right) + \left(m_I^f - m_I^i\right)}{t^f - t^i} \tag{3.1}$$

Assuming no volume changes between initial and final conditions, one obtains Eq. (3.2).

$$Q = \frac{V_T \cdot \left(\rho_T^f - \rho_T^i\right) + V_I \cdot \left(\rho_I^f - \rho_I^i\right)}{t^f - t^i} \tag{3.2}$$

where ρ is the gas density determined via a real gas equation of state $\rho = \frac{PM}{ZRT}$, where *M* is the molar mass of the gas, *R* is the ideal gas constant and Z is the compressibility factor.

With the use of dead-end time, where both valves are closed, there is no question about loss of mass during the diversion. However, both valves must be fast because when both are closed, mass accumulation in the inventory volume leads to a pressure rise that depends on the mass flow and the size of the inventory volume.

The pressure in the inventory volume must always be low enough that critical conditions apply to the CFVN (see Figure 25).

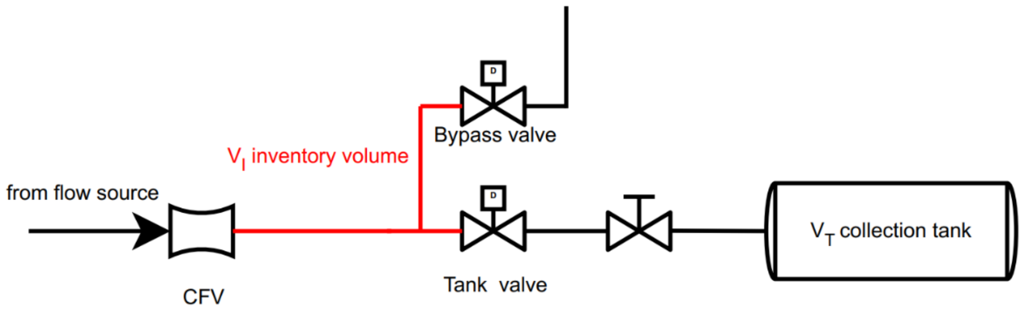

Figure 25: Schematic of the volumes of the PVTt standard.

Safety rules during calibration test:

- The test rig shall be operated by an authorized and experienced person;
- The test rig shall be using a ground-monitoring system to make sure that all electronic equipment in the EX-Zone (Zone 2) is at the same potential;
- All electronics in the EX-Zone are ATEX rated for the appropriate zone and gas type
- A fume hood with a dedicated air circulation system encloses the EX-Zone;
- Hydrogen detectors are placed under the fume hood and in the room housing the fume hood and serve as leak-detection systems. When the first trigger level of the system is activated, the hydrogen feeding line is closed and the ventilation of the room is activated. If the second trigger level is activated, the tanks containing hydrogen are vented into the atmosphere.
- After the measurements, the test rig is flushed with Nitrogen for inertisation;
- Open flames and smoking are strictly prohibited in the vicinity of the testing installation;
- The use of spark-producing devices, including mobile phones, is strictly prohibited in the vicinity of the testing installation;
- All safety mitigation and protective measures must be taken in order to minimize any risk due to the presence of explosive and flammable gases.
- All the testing activities shall be carried out in accordance with an authorized and experienced person;
- The measuring instruments shall be used for their intended purpose and under the conditions specified by the manufacturers and the procedures;
- All the measuring instruments shall be used with the required range and accuracy class and with valid calibration certificates;

METAS shall perform calibrations of the following meters:

- Sagemcom/Siconia EF4evo G4
- Sagemcom/Siconia EF4evo G6

4. TESTING OF PRESSURE TRANSMITTERS IN THE PRESENCE OF H_2

The testing of the pressure transmitters selected during the activities of Task 2.1 and indicated in Deliverable D2.1 will be performed in accordance with the protocol developed in Task 2.2 and indicated in Deliverable D2.2.

Objective of the test: The testing activity aims to evaluate the long-term drift and the hysteresis of the measuring devices in presence of pure H_2 or H2NG mixtures.

Operative procedures adopted during the experimental activities: Two activities will be performed to achieve the objective:

1. Ageing of the devices with H_2 and H2NG. The test duration and conditions are indicated in the specific protocol (Deliverable D2.2).
2. Calibration of the measuring devices. The calibration activity will be performed in accordance with the relevant international standards.

The detailed testing plan is reported in Deliverable D2.2.

In the next sections, information about the measurement equipment, the materials, and the operative procedures to respect the metrology requirements indicated in WP2 will be shown. The safety measures adopted throughout the testing activities are also included in the discussion. Furthermore, the test report and documentation to ensure comparable information and data in the experimental activity will be indicated.

4.1. Ageing of the pressure transmitters: introduction

Ageing of pressure transmitters will be performed in INIG and CESAME according to the protocol described in Deliverable D2.2.

The operative conditions maintained during ageing are reported in Table 23.

Table 23. The operative conditions of performed ageing.

Ageing	Laboratory	Fluid	Pressure (bar)	Temperature (C)	Period of time
1	INIG	25% H2/CH4(vol/vol)	65 ± 1,625	20 ± 5	4 + 4
2	CESAME	H2 & any other mixture of CH4/H2	1 to 65 barg	In accordance with the protocol	4 + 4

4.2. Ageing of the pressure transmitters: test bench and main components

4.2.1 INIG ageing test bench

The ageing test bench developed in INIG is shown in Figure 26. The ageing components consist of a sealed steel pressure installation to which the transducers under test are connected. The pressure source is a gas cylinder with a ready-made gas mixture together with a pressure regulator. A pressure gauge with a pressure recording function is mounted on the test installation. The installation is equipped with shut-off and bleed valves.

The installation is made of AISI 316 stainless steel with LET-LOK- type connections. Ball valves type H-800 made of AISI 316 stainless steel with PFA ball seal and PTFE stem seal with an operating pressure of up to 172 bar and an operating temperature range from -54°C to +149°C. The valves are designed to operate in fully open or fully closed positions. To connect the pressure transducers to the installation, there are T-pieces and straight connectors with ½" thread made of AISI 316 stainless steel.

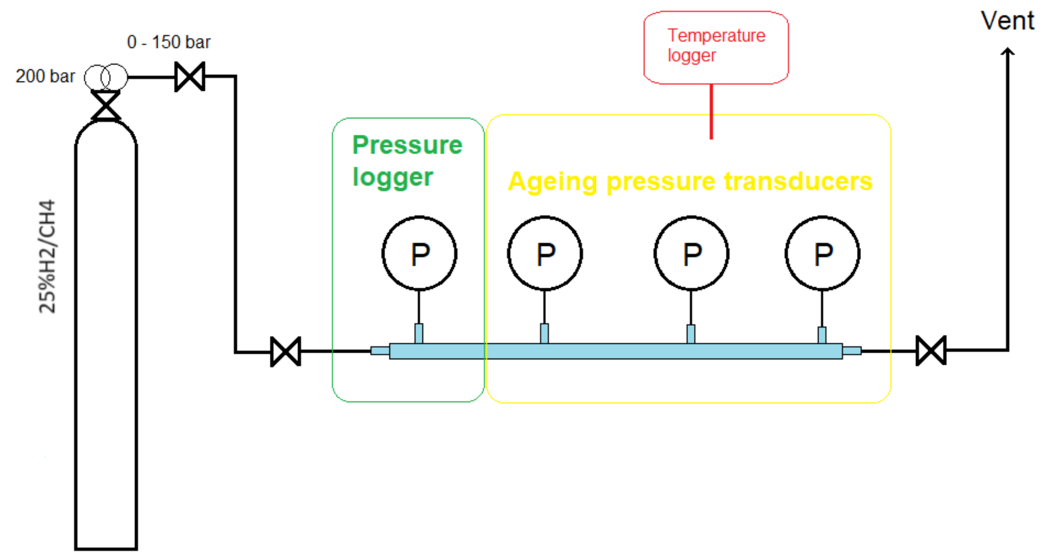

Figure 26. The ageing installation for pressure transducers test.

4.2.2. CESAME ageing test bench

The pressure sensors are mounted together on a manifold which is mounted on a line in the same way as gas meters (Figure 27).

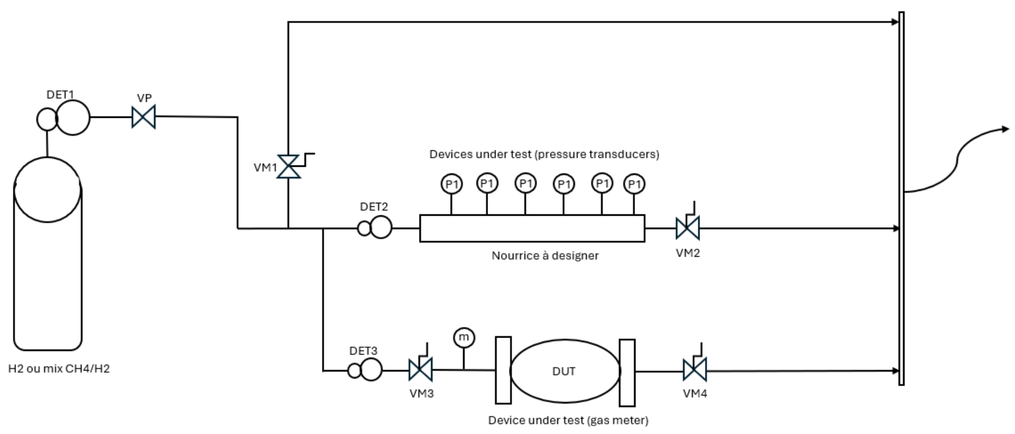

Figure 27. Example showing how pressure sensors are fitted in the ageing circuit of gas meters.

4.3. Ageing of the pressure transmitters: testing procedure

4.3.1 INIG ageing test procedure

General notes:

- Ageing conditions: if performed outdoors, no change in the ambient temperature conditions is possible. However, according to the manufacturer, normal changes in ambient temperature should not affect the operation of the transmitter, and only the medium and pressure are crucial for the transmitter's operation. However, to ensure test reproducibility, ageing should be performed indoor (room temperature) if possible.

The phases of the testing are reported below, while the ageing exposure process of pressure transmitters refers to steps 2 and 4:

1. Calibration with nitrogen.
2. Exposure to 25% H_2/CH_4 for a period of 3 months.
3. Calibration using nitrogen, N_2 (same measuring equipment as point 1).
4. Exposure to 25% H_2/CH_4 for a period of 3 months.
5. Calibration using nitrogen (same measuring equipment as point 1).

Description of the implementation of point 2 and point 4:

- The test installation should be equipped with a control manometer for pressure measurement, with an accuracy class appropriate to the intended working pressure;
- Maintain/record required environmental conditions;
- Connect the tested pressure transmitters with the appropriate pressure range to the test installation. Agree on the pressure range, number of transmitters and mounting position;
- Connect a nitrogen source to the connection stub to flush the test installation, not exceeding the maximum pressure of the transmitters;
- After flushing the test installation with nitrogen, close the valves so that no air enters the installation;
- Connect a burner to the release valve or ensure safe release to the atmosphere (in the case of small installation capacity);
- Connect the H_2/CH_4 source to the connection stub and start filling the test installation while burning the gas from the release valve;
- After replacing at least 3 volumes of the test installation, close the release valve;
- Use the pressure regulator to set the required operating pressure in the installation and then close the valve on the connection pipe. An acceptable deviation from the required pressure must be agreed;
- Close the gas cylinder securely;
- Note the beginning of the ageing process of the H2/CH4 transducers. Additionally, according to manufacturers' opinion it can be considered, for example, to release the pressure in the testing installation once a week and refill it with fresh H_2/CH_4, so as to simulate force the pressure on the membrane;
- Regularly check the pressure gauge in the test installation for a drop in pressure and, if necessary, top up the pressure to the required value;
- After a period of 4 months, the medium from the test installation should be ventilated by connecting a burner to the release port or ensuring a safe release to the atmosphere (in the case of a small installation capacity).

Once verified that the test installation is at atmospheric pressure, the transmitters can be dismantled and tested the accuracy using nitrogen (N_2).

Safety rules during ageing test

- Transmitter ageing test should be performed with power disconnected (limiting the risk associated with the lack of ATEX Class IIC approval for the pressure transducers under test);
- Test is performed in a well-ventilated room with a leak detection safety system dedicated to H2NG mixtures and hydrogen in the atmosphere. When the system is activated, the operation of the measuring installation and the ventilation of the room are stopped;
- Open flames and smoking are strictly prohibited in the vicinity of the testing installation;
- The use of spark-producing devices, including mobile phones, is strictly prohibited in the vicinity of the testing installation;
- All safety mitigation and protective measures must be taken in order to minimize any risk due to the presence of explosive and flammable gases.
- All the testing activities shall be carried out in accordance by an authorized and experienced person;
- The measuring instruments shall be used for their intended purpose and under the conditions specified by the manufacturers and the procedures;
- All the measuring instruments shall be used with the required range and accuracy class and with valid calibration certificates;

4.3.2 CESAME ageing test procedure

The procedure adopted for the ageing test is the same adopted for the gas meters.

4.4. Calibration of pressure transmitters: introduction

The instruction provided in EN 61298-1 “*Process measurement and control devices - General methods and procedures for evaluating performance – Part 1: General considerations*” is used as reference for the instructions developed to testing pressure transmitters accordingly with THOTH2 protocols.

Since INIG operates in the field of accreditation and International Laboratory Accreditation Cooperation Mutual Recognition Arrangement (ILAC MRA) measurement [2], and it makes interlaboratory comparisons on an ongoing basis, the Consortium agrees to not proceed with intercomparison among THOTH2 partners and to perform calibration of the pressure transmitters only at the INIG test bench.

ILAC is an international organisation of accreditation bodies worldwide that accredit conformity assessment bodies in the following areas: testing laboratories, calibration laboratories, medical laboratories, inspection bodies, and organisers of proficiency tests. ILAC MRA supports the international recognition of test results, calibration certificates and inspection certificates.

Calibrations of the laboratory’s reference equipment (1 x Druck Pace 6000, 1 x Druck DPI 620 and 1 x Multimeter Fluke 8508A) are carried out on a yearly basis (currently in Druck Standards Laboratory – England, PGNiG Orlen Group – Poland, Fluke Precision Measurement – Germany).

The INiG Laboratory conducts periodic interlaboratory comparisons as part of its accreditation, both in the field of pressure, pressure transducers and electrical quantities. The same equipment is used for these comparisons as will be used for testing within the THOTH2 project. The last interlaboratory comparisons INiG were conducted in the field of electrical quantities in 2023 with The Railway Research Institute IK (Poland) and in the field of pressure in 2022 with the Plum laboratory (Poland) with positive results.

4.5. Calibration of pressure transmitters: test bench and main components

The calibration of the pressure transmitters will be performed at INIG in the S01/PR test bench. The S01/PR test bench is used to test the accuracy of pressure transducers.

The PFD of the test bench is indicated in Figure 28 As depicted, nitrogen gas is used as fluid during the calibration for safety reasons. Nitrogen gas is stored in bottles (1) connected to the device under test. Pressure regulators (2) are installed at the exit of the bottle to ensure a stable pressure upstream the measuring device. In accordance with the Upper Range Value (URV) defined in the calibration procedure, the input signal to the measuring devices under test, will be set by the PACE 6000 two-channel pressure calibrator (4).

The required overpressure, for pressure in the range of 0 to 71 bar, is set to the pressure transmitter using the PACE 6000. Specifically, channel A is used for pressure from 0 to 71 bar, while channel B is from 0 to 8 bar. Based on the testing pressure, channel A will be used during the experimental testing. Even if it will not be used in the testing activities performed during the THOTH2 project, also the DRUCK DPI multifunction calibrator is available at the INIG testing facility. In the case of pressure in the range from 0 to 135 bar, the pressure is directly set through the reducer and controlled using the DRUCK DPI multi-function calibrator and the Vaisala PTB330 digital barometer.

In the case of transducer tests where a specific transducer ambient temperature is required to be maintained, the tested transducer is placed in a climatic chamber produced by Votsch or on a laboratory table (depending on the test) and the pressure pipe and electrical wires of the output signal are connected.

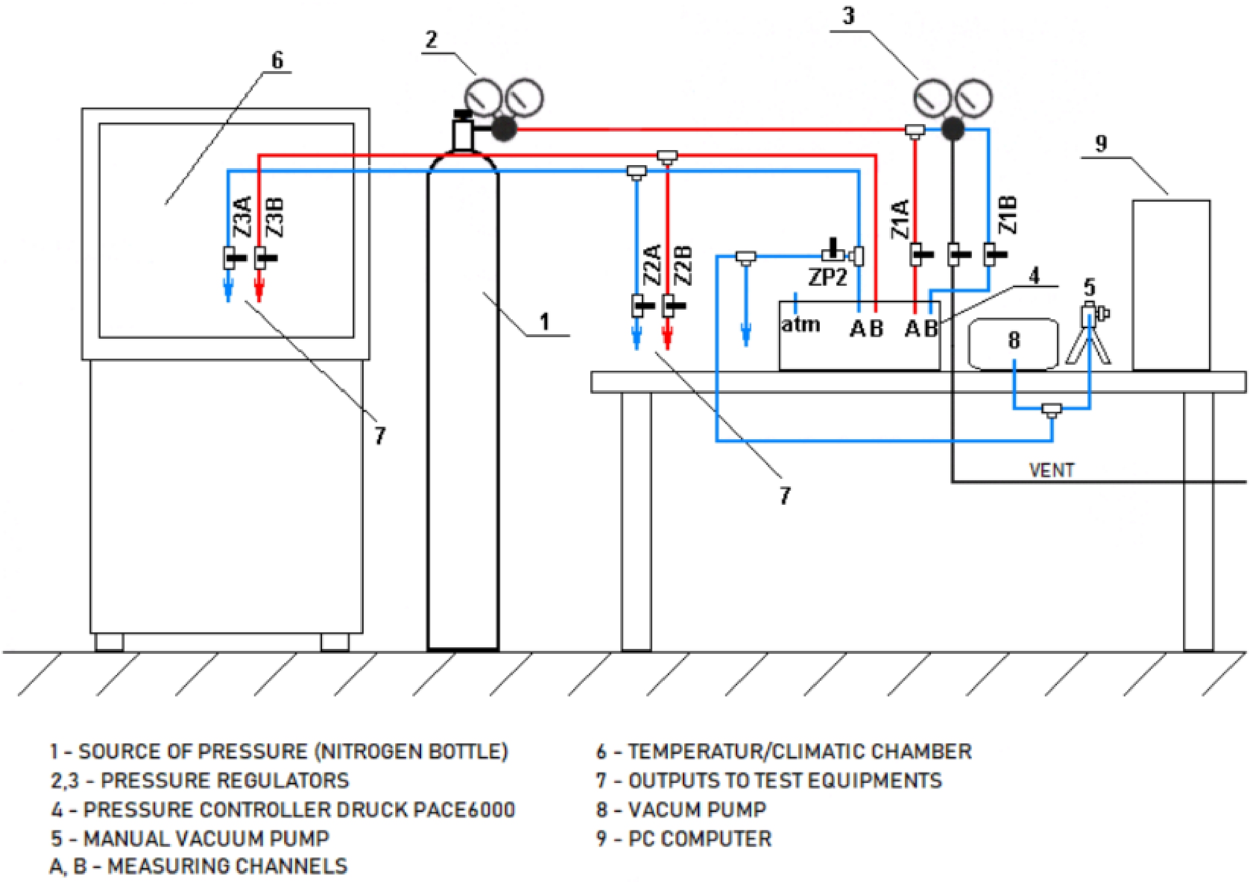

Figure 28. Process Flow Diagram of a S01/PR test bench for pressure transducers test.

The output signal for the converters can be a voltage or current signal measured with a FLUKE 8508A reference multimeter, or a HART signal read by a device using the HART communication protocol.

The Calibration and Measurement Capability (CMC) for INIG for the calibration of pressure transducers is reported in Table 24.

Table 24. CMC of INIG for absolute and gauge pressure measurement

Absolute pressure			Gauge pressure	
Pressure	CMC INIG		Pressure	CMC INIG
(0,04 ÷ 1,1) bar	1,7*10^{-4} bar		(-0,98 ÷ 0,0) bar	1,8*10^{-4} bar
(1,1 ÷ 2) bar	2,6*10^{-4} bar		(0,0 ÷ 6) bar	8,2*10^{-4} bar
(2 ÷ 6) bar	7,0*10^{-4} bar		(6 ÷ 17,5) bar	2,4*10^{-3} bar
(6 ÷ 17,5) bar	2,4*10^{-3} bar		(17,5 ÷ 70) bar	7,7*10^{-3} bar
(17,5 ÷ 70) bar	7,7*10^{-3} bar		(70 ÷ 135) bar	36*10^{-3} bar
(70 ÷ 100) bar	29*10^{-3} bar			
(100 ÷ 135) bar	33*10^{-3} bar			

4.6. Calibration of pressure transmitters: testing procedure

The flowchart illustrating the process of calibration is shown on the Figure 29 below.

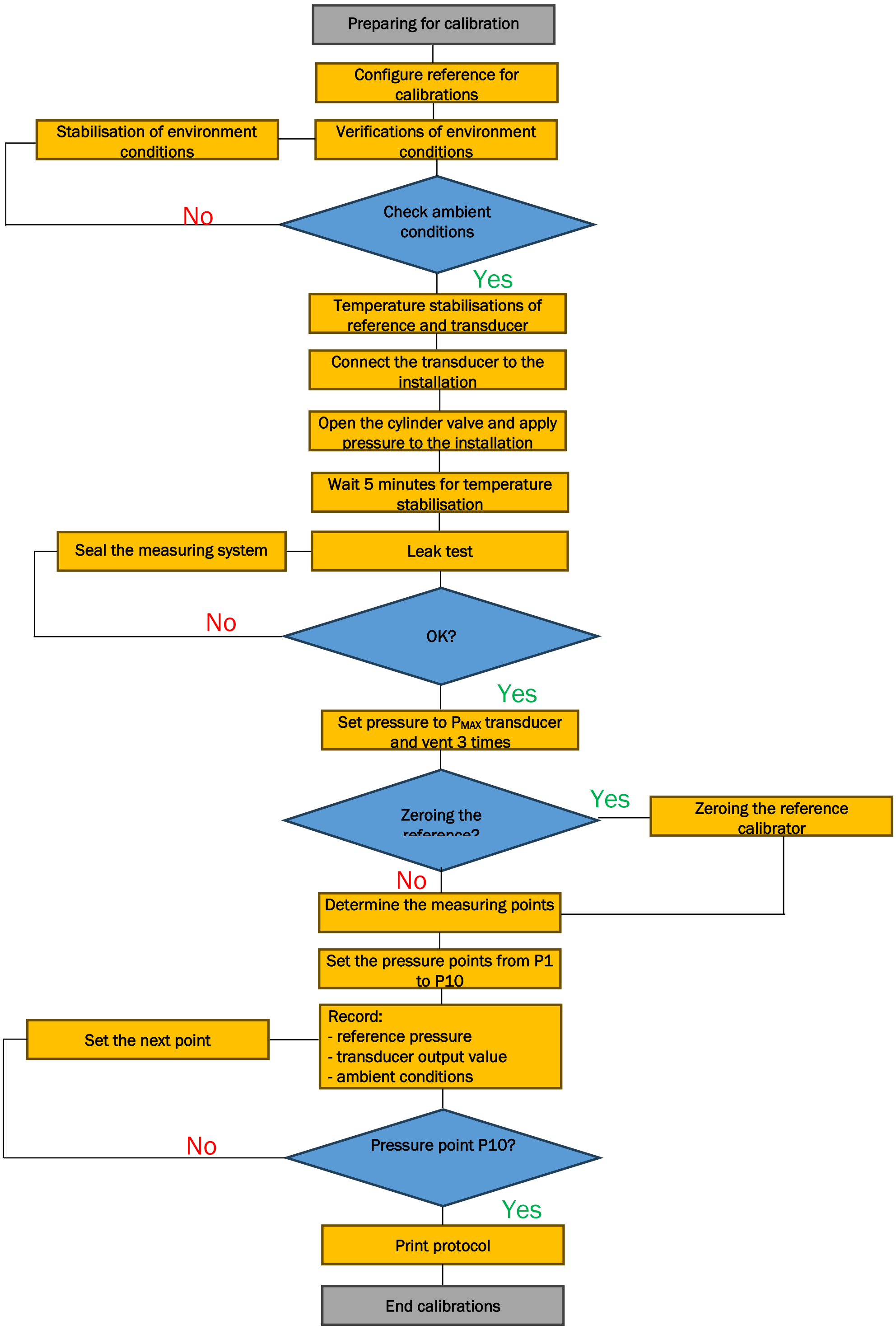

Figure 29. Pressure transmitters' calibration procedure.

The procedure defines methods of calibration of pressure transducers in the pressure range (0,04 ÷ 135) bar abs and (-0,98 ÷ 134) bar gauge.

Measuring instruments and auxiliary equipment used in the calibration of pressure transducers:

- Pressure calibrator Druck Pace 6000.
- Multifunctional calibrator Druck DPI 620 + pressure modules PM 620.
- Digital barometer VAISALA PTB330.
- Reference Multimeter Fluke 8508A.
- Nitrogen cylinder, regulators, valves.

Points of calibrations:

P_{min}	P_2	P_3	P_4	P_{max}
1 ⇨	2	3	4	5 ⇩
10	9	8	7	⇦ 6

where P_2, P_3, and P_4 are calculated according to Eq. (4.1)–Eq. (4.3).

$$P_2 = \frac{3P_{\min} + P_{\max}}{4} \quad (4.1)$$

$$P_3 = \frac{P_{\min} + P_{\max}}{2} \quad (4.2)$$

$$P_4 = \frac{3P_{\max} + P_{\min}}{4} \quad (4.3)$$

For each measurement point, five measurements of the reference pressure and five measurements of the transducer output value should be performed.

Equations describing the correction of the pressure transducer under test:

The following equations will be applied based on the type of output from the multimeter.

For “current” output, Eq. (4.4) and Eq. (4.5) apply:

$$\Delta P = P_{\mathrm{k}} - (P(I_{\mathrm{k}}) + \delta P_{\mathrm{powt}} + \delta P_{\mathrm{hist}} + \delta P_{\mathrm{tamb}}) \text{ [bar]} \quad (4.4)$$

$$P(I_{\mathrm{k}}) = \frac{P_{\max} - P_{\min}}{I_{\max} - I_{\min}}(I_{\mathrm{k}} - I_{\min}) + P_{\min} \text{ [bar]} \quad (4.5)$$

Where:

- $P(I_{\mathrm{k}})$ is the pressure as a function of the measured current at the transducer output, [bar];
- P_{k} is the correct value of pressure, [bar];
- $[P_{\min}; P_{\max}]$ is the pressure range set at the transducer, [bar];
- I_{k} is the measured electric current value at transducer output, [mA];
- $[I_{\min}; I_{\max}]$ is the electric current range at transducer output, [mA];
- δP_{powt} is the correction related to the repeatability of indication of the calibrated transducer;
- δP_{hist} is the correction related to the hysteresis of the gauge to be calibrated;

- δP_{tamb} is the correction related to the influence of ambient temperature on the accuracy of the calibration transducer.

For “voltage” output, Eq. (4.6) and Eq. (4.7) apply:

$$\Delta P = P_{\mathrm{k}} - (P(U_{\mathrm{k}}) + \delta P_{\mathrm{powt}} + \delta P_{\mathrm{hist}} + \delta P_{\mathrm{tamb}}) \ \ [\mathrm{bar}] \qquad (4.6)$$

$$P(U_{\mathrm{k}}) = \frac{P_{\mathrm{max}} - P_{\mathrm{min}}}{U_{\mathrm{max}} - U_{\mathrm{min}}}(U_{\mathrm{k}} - U_{\mathrm{min}}) + P_{\mathrm{min}} \ \ [\mathrm{bar}] \qquad (4.7)$$

where:

- $P(I_{\mathrm{k}})$ is the pressure as a function of the measured current at the transducer output, [bar];
- $P(U_{\mathrm{k}})$ is the pressure as a function of the voltage measured at the transducer output, [bar];
- P_{k} is the correct value of pressure, [bar];
- $[P_{\mathrm{min}}; P_{\mathrm{max}}]$ is the pressure range set at the transducer, [bar];
- U_{k} is the measured voltage value at transducer output [V];
- $[U_{\mathrm{min}}; U_{\mathrm{max}}]$ is the voltage range at transducer output, [V];
- δP_{powt} is the correction related to the repeatability of indication of the calibrated transducer;
- δP_{hist} is the correction related to the hysteresis of the gauge to be calibrated;
- δP_{tamb} is the correction related to the influence of ambient temperature on the accuracy of the calibration transducer.

Safety rules during calibration test:

- Calibration of pressure transducers should be carried out on inert gases and there is no risk of explosion;
- All activities should be carried out in accordance by an authorized and experienced person;
- The measuring instruments shall be used for their intended purpose and under the conditions specified by the manufacturers and the procedures;
- All the measuring instruments shall be used with the required range and accuracy class and with valid calibration certificates;

5. TESTING OF TRACE WATER SENSORS IN THE PRESENCE OF H_2

The testing of the trace water sensors selected during the activities of Task 2.1 and indicated in Deliverable D2.1 will be performed in accordance with the protocol developed in Task 2.2 and indicated in Deliverable D2.2.

Testing and calibration activities will be performed only in INRIM test benches are based on primary and reference standards for humidity in gases. INRIM (Istituto Nazionale di Ricerca Metrologica) is the Italian National Metrology Institute.

Objective of the test: The testing activity aims to evaluate the performance of market devices installed at DSO/TSO partners, via repeat calibrations, and identify any potential influence of hydrogen on their measurement performance and operation.

Operative procedures adopted during the experimental activities: Six main activities will be performed to achieve the above objective:

1. Understanding the actual measurement performance of the trace water sensors (Units Under Test, UUTs) installed and routinely used at DSO/TSO partners' premises by means of a so-called "common-practice" laboratory calibration and an associated investigation of historical data if and when available;
2. Ageing/operation of the UUTs in the field, as they are returned from the calibration, for about 8 months;
3. Investigation of possible ageing/drift due to the operation condition of the UUTs by repeating the calibration at point 1;
4. Understanding of actual measurement performance of the UUTs by means of a so-called "revised-practice" laboratory calibration in the presence of pure H_2 or blended with NG;
5. Ageing of the UUTs in the field for approximately 5 months;
6. Investigation of possible aging/drift due to the operation condition of the UUTs by repeating the calibration at point 4.

The detailed testing plan is reported in Deliverable D2.2.

In the next sections, information about the measurement equipment, the materials, and the operative and safety procedures to respect the metrology requirements indicated in WP2 will be shown. Furthermore, the test report and documentation to ensure comparable information and data in the experimental activity will be indicated.

5.1. Calibration of the Units Under Test – Common practice

The first and second calibrations of the Units Under Test as described at points 1 and 3 listed above will be performed according to the protocol described in Deliverable D2.2.

The common-practice calibration will be implemented according to the calibration by comparison method, where the reading of the UUTs is compared against a reference value (coming from a reference standard instrument or a primary standard generator). The difference between the UUTs reading and the reference value (the calibration correction) and its associated expanded uncertainty correspond to the results of the calibration.

5.2. Testing bench used for common practice calibration

The expertise, competence and facilities shared by INRIM include the development of primary standards and measurement techniques for humidity in gases, the measurement of thermo-physical properties of real gas mixtures, as well as the measurement of air temperature. The humidity laboratory shares a broad range of calibration and testing facilities to provide measurement traceability to sensors and analysers for trace water measurements in different gas matrices including energy-relevant gases.

5.2.1. PRIMARY HUMIDITY GENERATORS, INRIM-01 and INRIM-02

The INRIM-01 and INRIM-02 primary humidity generators (PHG), shown in Figure 30 and Figure 32, are recirculating-type generators that operate according to the single-pressure and single-temperature principle. They cover the dew/frost point temperature range from -85 °C to 95 °C.

In both systems, the working pressure is kept constant to better than 0.05% for any value between 1000 hPa and 1200 hPa, typically at 1050 hPa, by using an electronic pressure controller. The pressure controller keeps the pressure stable and constant even when a small gas flow, in the range between 0.5 L/min and 4 L/min in standard conditions, is drawn from the generator to feed the dew-point hygrometers under calibration and then is vented to the ambient. The small flow vented through the dew-point hygrometers is then replaced by the same amount of dry gas coming from a pressurized nitrogen/air gas source.

The reference dew-point is established within an isothermal saturator; the dew-point temperature is measured at the saturator outlet using a pair of calibrated platinum resistance thermometers (PRTs) traceable to ITS-90. A PRT is immersed in the water/ice while the other is in contact with the gas flow, respectively. Neglecting any possible difference between the evaporation and condensation temperatures, which for pure water (demineralized water) is below 0.001 °C in both liquid and solid phases, the dew-point temperature is the parameter describing the saturation of the gas stream over water, while the frost-point temperature describes the saturation over ice.

Chilled-mirror hygrometers or laser-based analysers under calibration are connected to the generator outlet either via a heated flexible hose (held at a temperature at least 10 °C higher than the dew-point temperature for dew-point temperature higher than the ambient temperature) or by a rigid electro-polished stainless tubing.

For calibration of aluminium oxide sensors, a suitable sensor holder is used. The holder hosts a single sensor in calibration and is fed by the generator outlet, while the flow rate is controlled by a needle valve downstream the sensor. A manifold affording multiple parallel sensor calibrations can be fitted to the generator outlet.

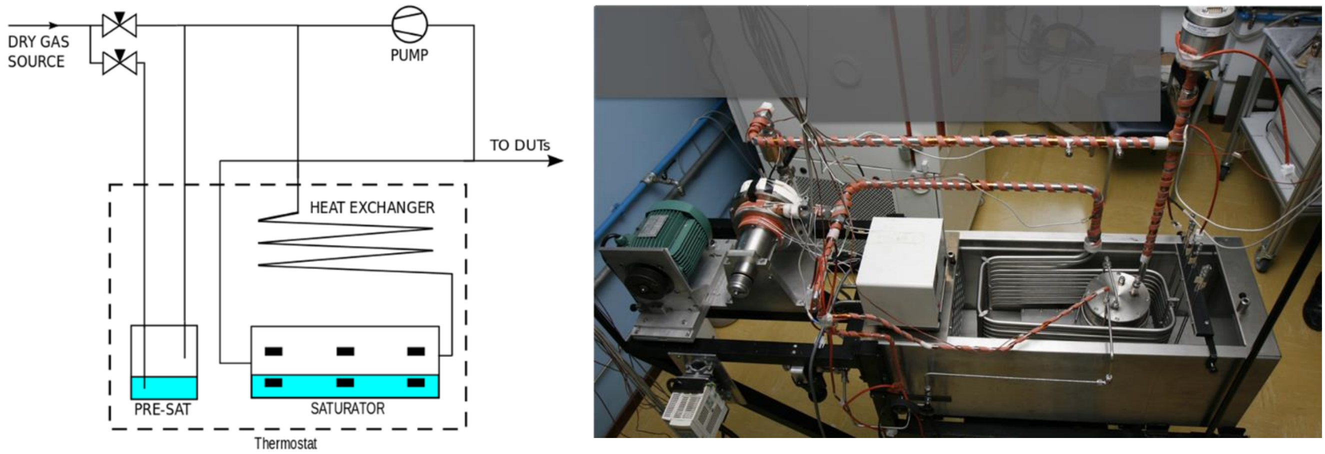

Figure 30. Simplified diagram and a picture of the PHG GENERATOR INRIM-01.

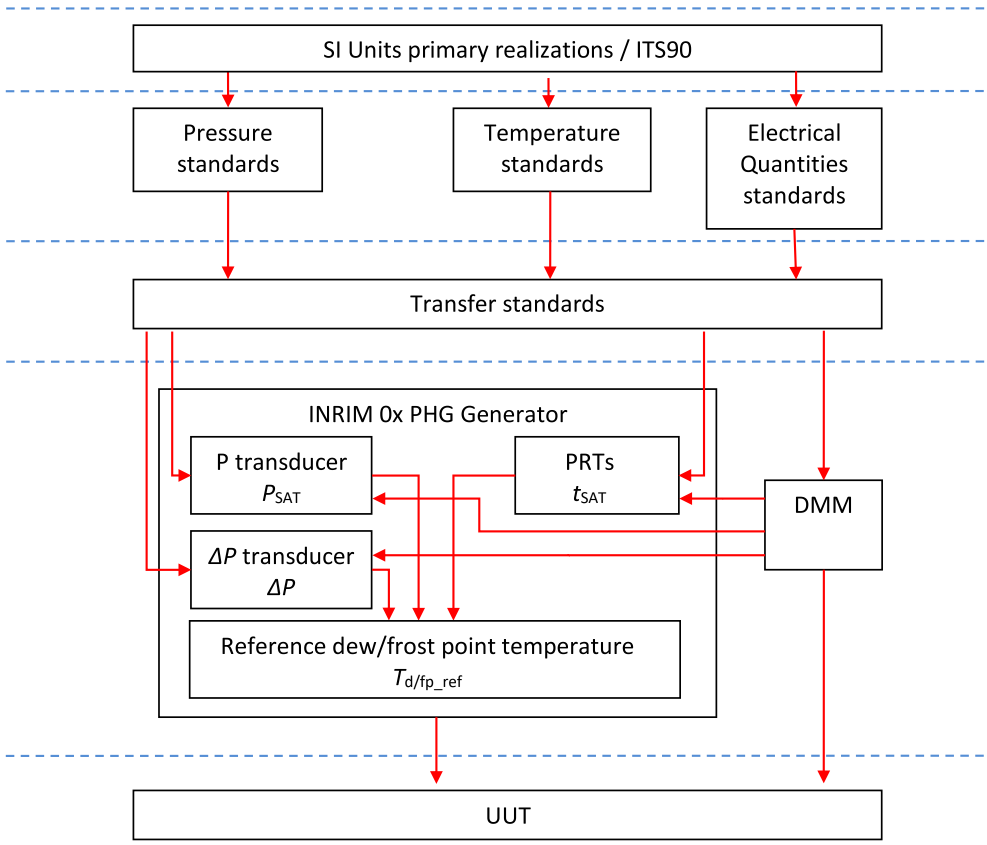

Figure 31. Traceability diagram of the PHG GENERATOR INRIM-01.

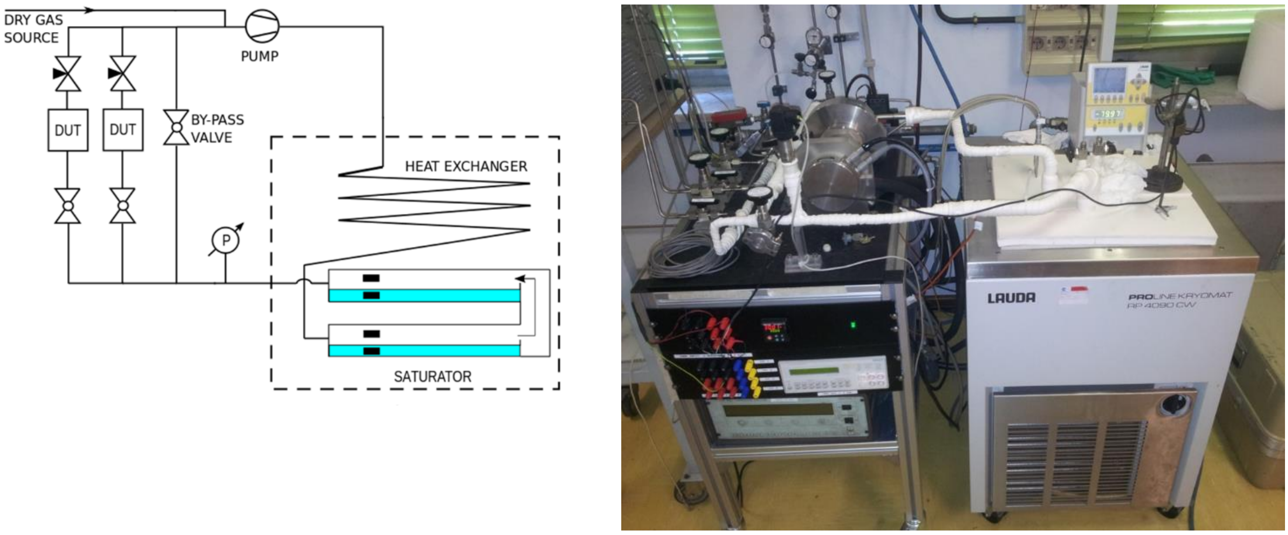

Figure 32. The simplified diagram and a picture of the PHG GENERATOR INRIM-02.

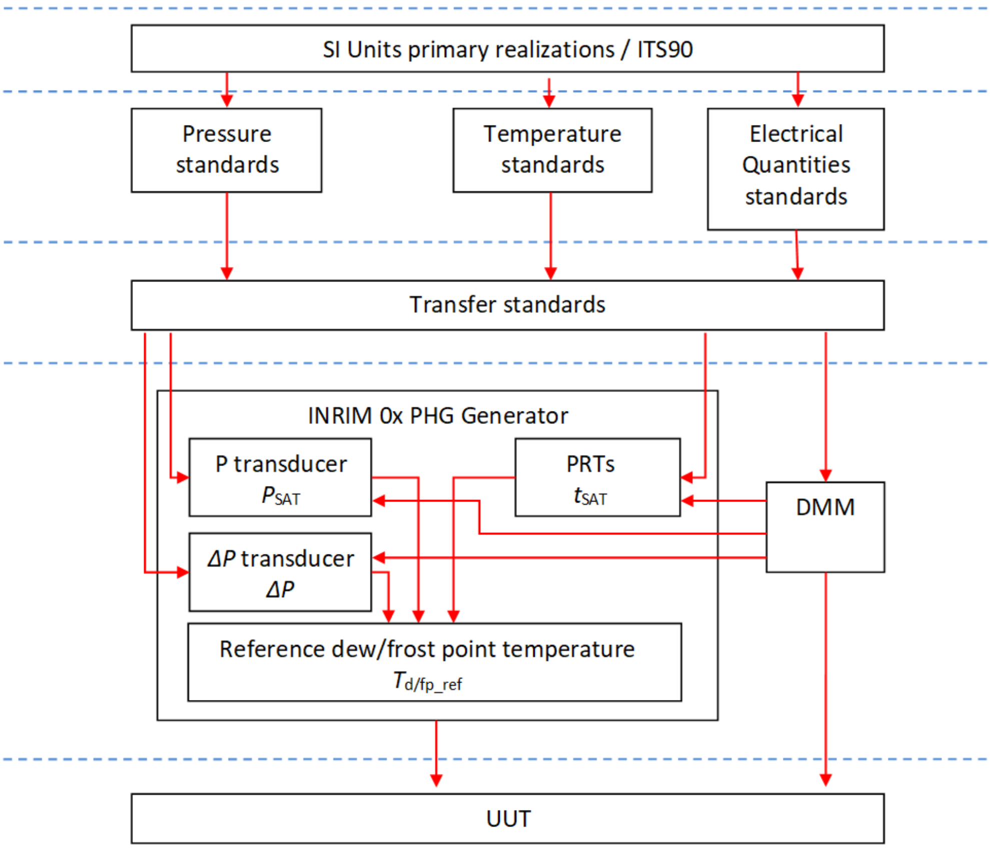

Figure 33. Traceability diagram of the PHG GENERATOR INRIM-02.

5.2.2. LOW-FROST-POINT HUMIDITY GENERATOR, INRIM-03

The INRIM-03 low frost-point (LFP) humidity generator shown in Figure 34 is a single-pressure, single-temperature generator that operates by saturating a nitrogen flow gas over an isothermal, uniform, ice layer in the frost-point temperature range between -100 °C and -20 °C and in the pressure range from 200 hPa to 0.68 MPa. Its operation involves several components, including a heat exchanger, an isothermal saturator, a thermostatic bath, a temperature measurement system with standard platinum resistance thermometers (SPRTs) read by a precision resistance bridge, a gas drying system based on molecular sieves, pressure gauges and a back-pressure control system. A trace water analyser based on cavity ring-down spectroscopy (CRDS) or a chilled-mirror hygrometer (CMH) are used to monitor the generator operation.

A dry N2 gas stream flows through a helicoidally shaped heat exchanger to reach a set temperature. Then, it enters a multipass isothermal saturator where it flows over ice passageways until the gas is saturated with water vapour. The entire system is hosted in a thermostatic bath to maintain a constant temperature which uses ethanol as a heat transfer medium. The saturation temperature measurements are carried out by means of the SPRT in contact with the outlet gas stream, while a platinum resistance thermometer (PRT) immersed in the ethanol bath, at the same depth as the SPRT, ensures a continuous measurement of the bath temperature and a periodic check of the temperature uniformity. These temperature data are acquired with the resistance bridge. The outlet gas from the LFP humidity generator is supplied to the instruments/ analysers/ sensors under calibration through electro-polished tubing to ensure minimal interaction between the gas and the tube walls. To further minimise any absorption/desorption effects, the tubing is trace-heated.

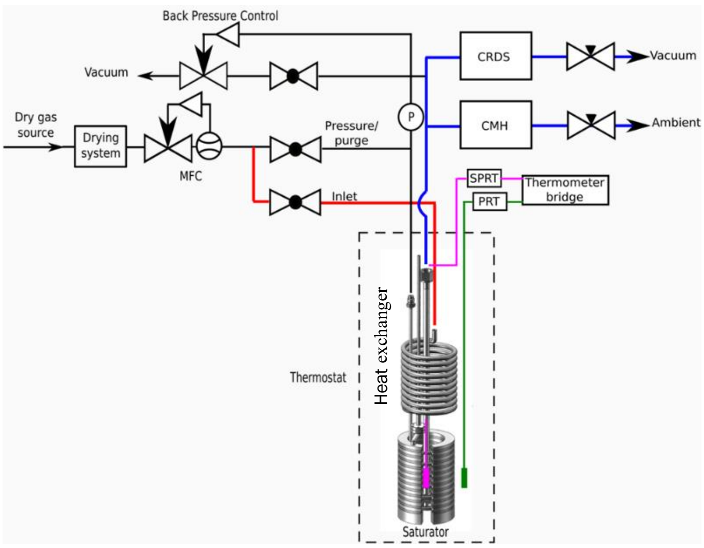

Figure 34. Simplified diagram and a photograph of the LFP HUMIDITY GENERATOR INRIM-03 Mark1.

The traceability diagram for the LFP Humidity Generator INRIM-03 Mark1 is shown in Figure 35.

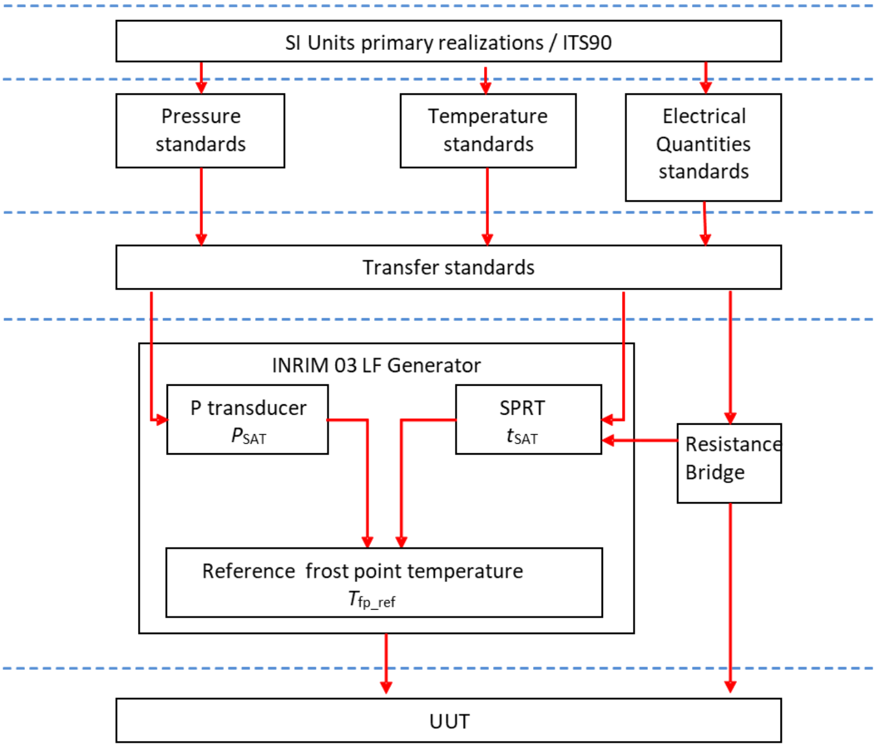

Figure 35. Traceability diagram of the LFP HUMIDITY GENERATOR INRIM-03 Mark1.

5.2.3. CLIMATIC TEST CHAMBER CTS C-30/400/S

The climatic test chamber CTS C-30/400/S shown in Figure 36 is a custom-made climatic chamber designed for calibration and testing, in a wide range of environmental and climatic conditions, for sensor calibration and for testing products, materials, and components. The operating temperature ranges from -30 °C to 95 °C, while the dew-point temperature ranges from -50 °C to 90 °C and relative humidity ranges from 2 %rh to 98 %rh (see Figure 37 for details about the operating range). The custom-made CTS C-30/400/S is designed as a double coaxial chamber where the outer chamber is equipped with heating, cooling and humidification systems which allow for accurate control of temperature, humidity, and flow. The heating system uses an electric heater to raise the temperature inside the outer chamber. The heater is controlled by a thermostat, which ensures that the desired temperature is maintained. The cooling system uses a refrigeration unit to lower the temperature inside the outer chamber. The refrigeration unit is controlled by a thermostat which ensures that the desired temperature is maintained. The humidification system uses a mixed-flow water vapour generator to control the humidity inside the outer chamber, while the humidification system is controlled by an impedance hygrometer which ensures that the desired humidity is maintained in the outer as well as in the inner chamber. The outer and inner chambers are also equipped with a variety of sensors that monitor temperature, humidity, and other environmental conditions in real-time. These data are then used by the chamber's control system to adjust the cooling, heating, humidification, and dehumidification systems to ensure that the desired environmental conditions are maintained.

Figure 36. A photo of the climatic chamber CTS C-30/400/S.

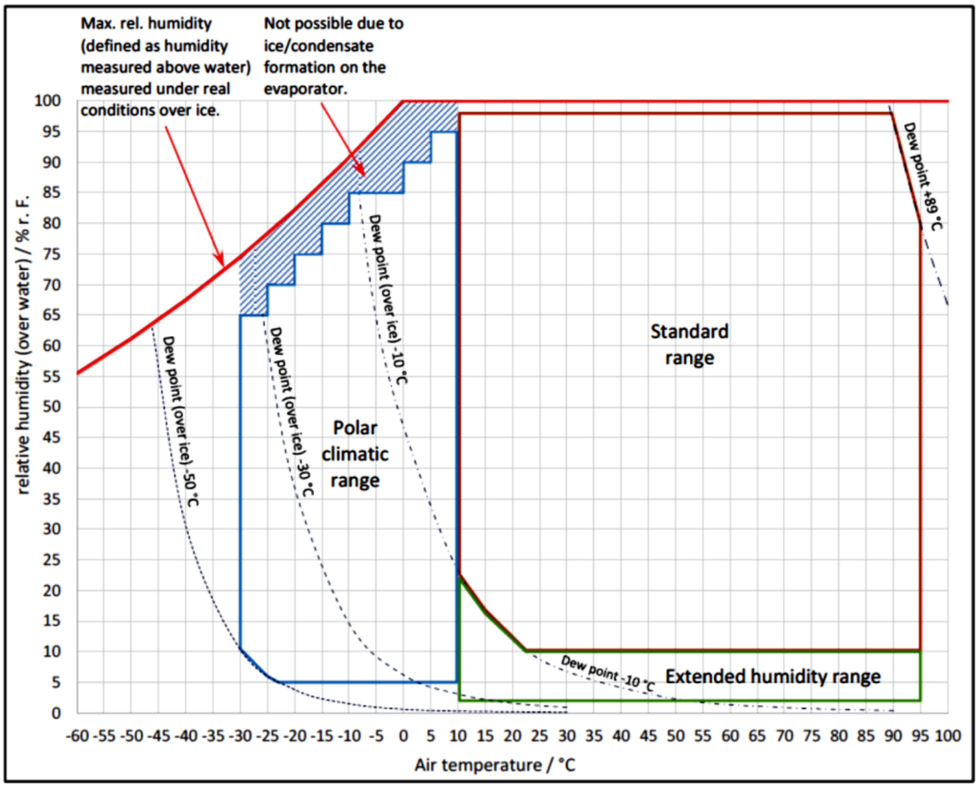

Figure 37. The working range of the climatic chamber.

5.3. Calibration of the Units Under Test – Revised practice

The calibrations of the Units Under Test (UUTs) as described at points 4 and 6 listed above will be performed according to the protocol described in Deliverable D2.2

The so-called “revised-practice” calibration will be implemented by comparison against a novel traceable calibration system for moist H2 operating from 0.2 MPa to 5.5 MPa. The reference value is compared against the reading of the UUTs. The difference between the UUTs reading and the reference value (the calibration correction), along with its associated uncertainty corresponds to the result of the calibration. The calibration will be carried out at three different frost-point temperatures in the range between -60 °C and -10 °C at pressures up to 3 MPa. The corresponding water vapor amount fraction ranges from 0.5 µmol/mol to 100 µmol/mol.

5.4. Testing bench used for revised practice calibration

A novel humidity standard able to generate moist H2 with the traceable amount of water vapour is being under development at INRIM under the EU Metrology Partnership project 21GRD05 MeT4H2 “Metrology for the hydrogen supply chain” (see Figure 38). The system consists of a transportable, single-pass, precision humidity generator (TPHG) to provide state-of-the-art onsite traceability to trace water measurements in hydrogen. It is a saturation-based generator designed at INRIM capable of generating a moist hydrogen stream with pressure dew-point in the range between -60 °C and -10 °C at any pressure between 0.2 MPa and 6 MPa. At 6 MPa, the amount of water fraction corresponds to 0.3 µmol/mol to 55 µmol/mol, while at 3 MPa, the amount of water fraction corresponds to 0.5 µmol/mol to 100 µmol/mol. Its development and commissioning are underway, together with its metrological testing and validation. Further to its operation as a moist H_2 generator, the system has been designed for a flexible, multi-gas, multi-pressure operation in order to be easily retrofitted to enable trace water sensors testing and calibration in CH_4/H_2 gas blends.

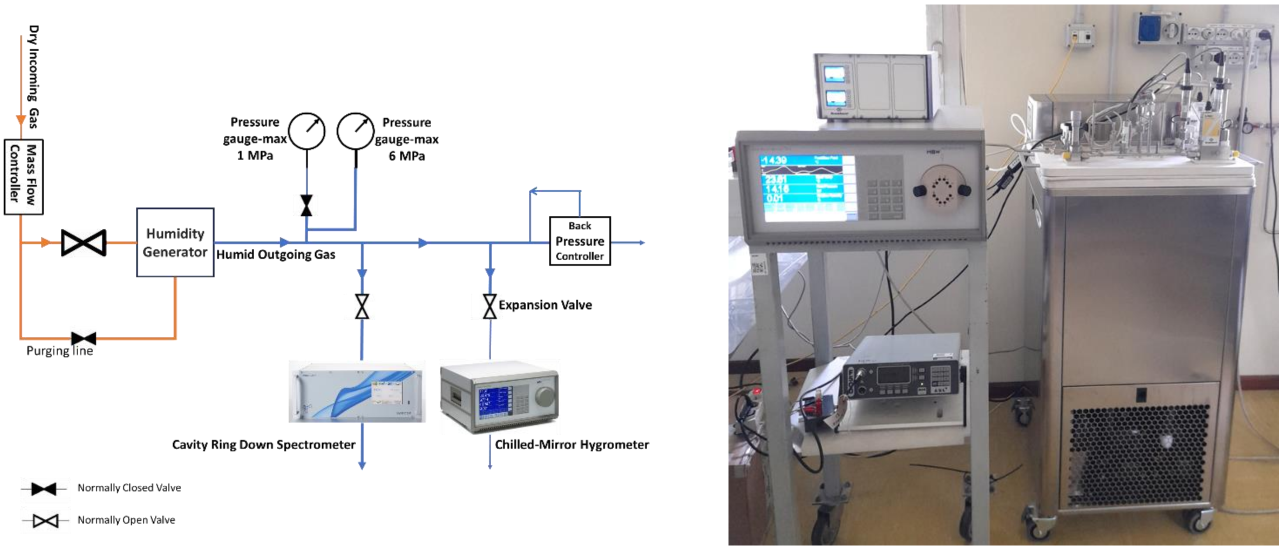

Figure 38. Simplified diagram and a picture of the moist-H2 transportable precision humidity generator (TPHG).

5.5. Estimation of ageing of trace water sensors in field operation

In order to investigate how the operating condition affects the UUTs performance over time, a suitable time interval (ageing) between the first and second calibration, for both common and revised practices, is established. During this period, the UUTs are returned to the respective users and will work under their normal operating conditions and environment.

The ageing period is eight months for the common-practice calibration and five months for the revised-practice calibration. The sensor drift and its uncertainty will be estimated in terms of the difference between the calibration results of two consecutive calibrations at the same calibration point and the combined uncertainty of the difference.

For sensors having a historical record of traceable calibrations or control charts available, the most recent sensor drift will be compared and contrasted with the present testing to support the sensor performance studies.

5.6. Safety procedure adopted during tests

The following safety measures were adopted both during the aging phases and calibration activities involving pure hydrogen or blends. They **do not apply** to measurements carried out in in paragraph 5.1 in nitrogen.

- **Operational environment and leak detection:**
  - All measurements are carried out in a well-ventilated, safety-isolated environment, under an aspiration hood equipped with hydrogen leak detection systems.
  - In case of detection of a hydrogen leak, the system automatically shut off the electrical power and start the room ventilation while the H2 ventilation under the hood is kept working.
- **Electrical and equipment safety:**

    - All electrical devices were located outside hazardous (Ex) zone.
    - Only intrinsically safe (ATEX-certified) equipment can be used in the Ex zone.
- **Ignition source control:**
    - Open flames, smoking, and spark-producing devices (including mobile phones) are strictly prohibited near the testing areas.
- **Personnel and procedure compliance:**
    - All testing activities are carried out by authorized and experienced personnel.
    - Only calibrated instruments with valid certification and appropriate accuracy classes were used.
- **Emergency preparedness and safety infrastructure:**
    - Advanced safety systems, such as hydrogen-specific sensors and hood with forced ventilation are in place to ensure a rapid and effective response in case of leaks or incidents.
    - Emergency protocols, including evacuation procedures and emergency shutdown processes, are adopted to protect personnel and equipment from potential hazards.
    - Risk assessment procedures are regularly updated and personnel trained on them.

6. TESTING OF LEAK DETECTORS IN THE PRESENCE OF H_2

The testing of the leak detectors selected during the activities of Task 2.1 and indicated in Deliverable D2.1 will be performed in accordance with the protocol developed in Task 2.2 and indicated in Deliverable D2.2.

<u>Objective of the test</u>: The test activity focuses on reliability, reproducibility, and stability to determine how the presence of H_2 may impact device performance and characteristics. Furthermore, the second objective of the test is to identify potential shortcomings of the current standards used for testing and acceptance of leak detectors and to provide recommendations to overcome such limitations.

<u>Operative procedures adopted during the experimental activities</u>: Four main activities will be performed to achieve the objective:

1. Preparation of the experimental setup in accordance with current standards [3-5] and the detector manufacturer's guidelines. Gas flushing of the leak detectors.
2. Evaluate, as a reference, the detector performance in monitoring methane leakages, including the calibration curve, short- and long-term stability, alarm setpoint check, RH% impact, response/recovery times and high gas concentration operation.
3. In the presence of the H2NG mixture, replicate the same measurements made with methane, using different percentages of hydrogen and methane. Analyse and compare the data collected under the two different conditions.
4. Evaluate, if allowed, the potential H_2 poisoning of detectors.

The test specifications, durations and conditions are indicated in the specific testing protocol (Deliverable D2.2).

In the following chapters, information will be provided on the measurement equipment, materials, and operating procedures to meet the metrological requirements stated in WP2. The safety measures adopted throughout the testing activities are also included in the discussion. In addition, the test report and documentation will be given to ensure comparable information and data in the experimental activity.

6.1. Preparation of the experimental setup and testing procedure

The experimental setup will be prepared in accordance with current standards [3-5] and the detector manufacturer's guidelines. Tests of leak detectors will be performed in FBK and INIG in accordance with the protocol described in Deliverable D2.2.

The operative conditions controlled during the tests are reported in Table 25.

Table 25. Measurement parameters adjustable directly and indirectly in FBK and INIG setups.

Directly controlled setup parameters					Indirectly controlled setup parameters	
Gas cylinder concentration [ppm mol]	Gas flow [sccm]	Temperature [°C]	Injection time [s]	Gas mixture composition	Gas concentrations [ppm mol]	RH% [%]

6.1.1. FBK test bench and main components

FBK has a long-term experience in the development of flow sensors and solid-state gas sensors, specifically chemoresistive gas sensors. In order to be able to characterize, calibrate and validate the gas sensors internally produced, a dedicated lab has been set up, i.e., the Gas Qualification Laboratory (Laboratorio

Qualifica Gas). The Gas Qualification Laboratory of FBK has been designed to provide tools, systems and facilities useful for the development, integration and testing of gas sensing devices and flow sensors. In particular, it includes:

- gas test benches, equipped with mass flow controllers and cylinders with certified concentrations useful to characterize, test and calibrate the chemical gas sensor, either purchased from commercial suppliers or produced in the FBK clean rooms. The test benches and all the safety procedures implemented in the Gas Qualification Laboratory enable tests up to concentrations lower than the LFL of flammable/explosive gases.
- analytical tools (a quadrupole mass spectrometer and a proton transfer reaction mass spectrometer), useful as gold standards for the determination of unknown gas mixtures, or as a reference to check the composition of prepared gas mixtures in the lab.

To date, the management, all the labs, procedures and processes of the Sensors and Devices center of FBK are in compliance with the standard UNI EN ISO 9001/2015. In addition, management software (Fablims) has been implemented to ensure process quality. In this software, all tools, procedures, processes and items to be tested and/or developed are recorded to standardise operations and improve process control and reproducibility. Furthermore, the registration of non-conformities has been foreseen, allowing for targeted improvements in the processes and enhancing overall efficiency and effectiveness through root cause analysis.

In addition to the Gas Qualification Lab setups, a suitable test bench has been developed for performing measurements at concentration ≥ LFL of H_2 and CH_4. The test bench has been implemented in the laboratory of the Hydrogen technologies and Resilient Energy Systems (HyRES) Unit of the Sustainable Energy (SE) centre of FBK.

Description of the lab

Gas test benches for measurements below the lower flammability limit

The Gas Qualification Laboratory is a 5 x 6 m^2 lab located in the east building of FBK at Via Sommarive 18 (Trento, Italy). Currently, the lab hosts four gas test benches:

- MKS gas test bench, equipped with six MKS mass flow controllers, six electrovalves and a multi-channel flow ratio/pressure controller model 647C manufactured by MKS;
- Brooks gas test bench, equipped with five Brooks mass flow controllers, five electrovalves and a multi-channel flow ratio/pressure controller model 0260 manufactured by Brooks;
- Alicat gas test bench, equipped with three Alicat mass flow controllers and a multi-channel flow ratio/pressure controller manufactured by Alicat;
- A portable gas test bench, placed on a wheeled bench and equipped with calibrated four mass flow controllers from manufacturers, four electrovalves and a customized multi-channel flow ratio/pressure controller.

A general scheme of the test benches at FBK is reported in Figure 39. The simplified P&ID is shown in Figure 40. All four gas test benches can be driven using a dedicated communication and data acquisition application, developed using LABVIEW software, which allows to communicate and to drive mass flow controllers, including setting, saving and uploading automatic gas flow injections, and storing, in real -time, actual flow data related to the gas test benches.

In addition, four different gas chambers equipped with related read-out electronic boards are available. In particular, the gas chambers have been designed for hosting and testing solid-state gas sensors, while the electronic read-out can be used to collect gas sensor data, as well as to communicate and set the working parameters of the devices investigated. The gas chambers are placed in a climate chamber, in order to make the tests at different temperatures and relative humidity (%).

Outside the lab is present a gas box, containing seven dual-stage pressure reducers which can host, simultaneously, seven different gas bottles, typically at an initial pressure of 200 bar. The gas box is equipped with electropolished stainless steel tubes with a diameter of 6 mm, dedicated to the transport of the gas mixtures.

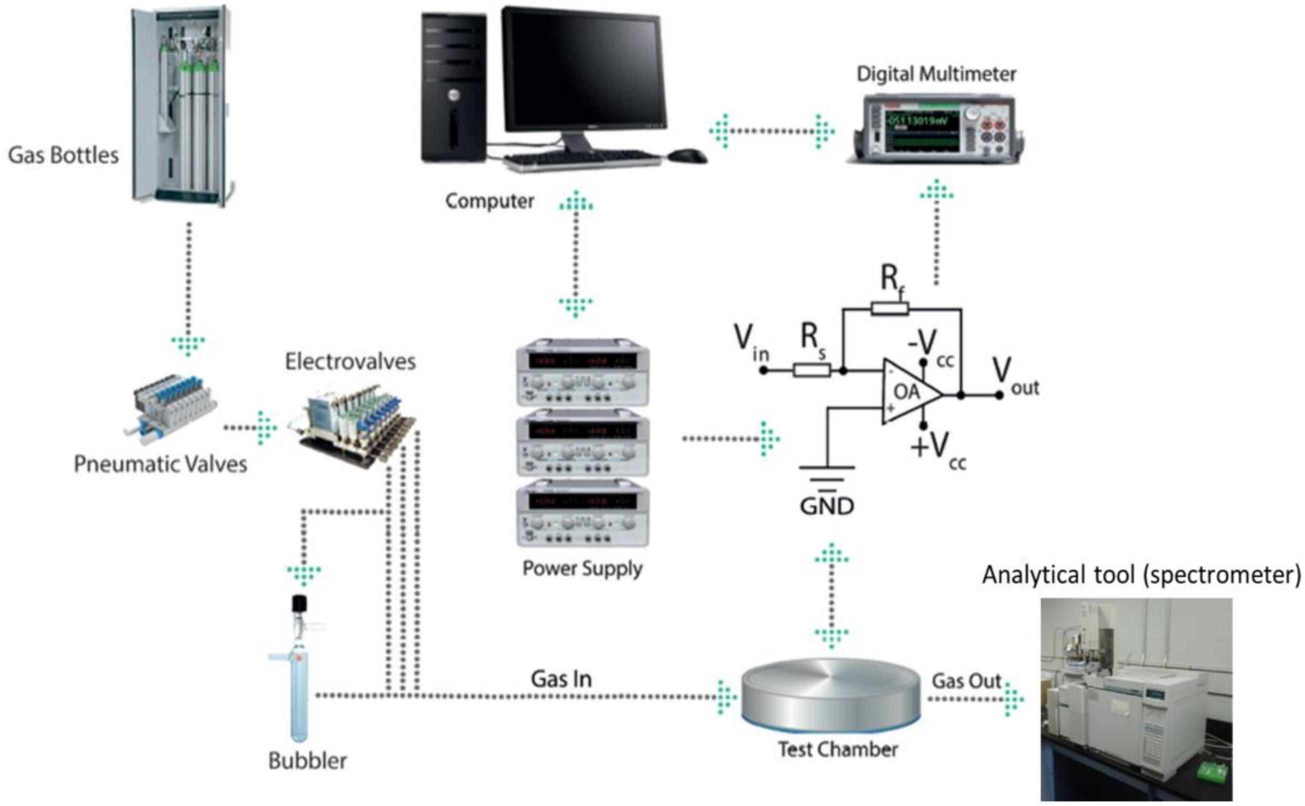

Figure 39. Process Flow Diagram of the test bench.

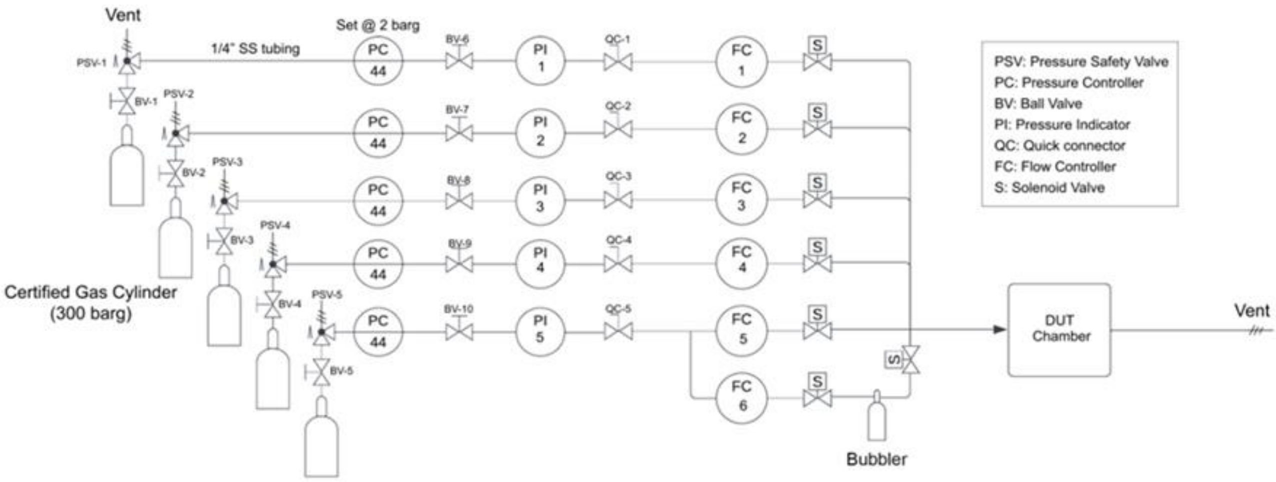

Figure 40. Piping and Instrumentation Design of the test bench.

Main installed components

Gas mixtures are obtained by mixing different gases contained in certified gas cylinders at a pressure up to 250 barg. To control the gas mixture composition, a maximum of eighteen mass flow controllers can be used simultaneously. Of course, the maximum number of gases to be used in the mixture is limited by the number of dual-stage pressure reducers, which are seven.

The main installed components are:

- Gas cylinders with certified concentration of gases.
- Seven dual-stage pressure reducers, which work from 250 to 1 bar.
- Seven MKS, five Brooks, three Alicat and four Brooks (from different manufacturers) mass flow controllers.
- Multi-channel flow ratio/pressure controllers manufactured by MKS (model 647C), Brooks (model 0260) and Alicat.
- A dedicated software for driving the gas test benches.
- A climatic chamber.
- Sealed gas chambers equipped with suitable electronic readout boards, for testing solid state gas sensors and commercial devices.
- Bubblers, filled with deionized water, useful for introducing controlled levels of RH% in the gas flow.
- A temperature/humidity sensor (Sensirion SHT40) for measuring RH% and temperature of the gas mixture flow.

Installed analytical measuring devices

Currently, at the Gas Qualification Lab, there are two analytical tools available:

- A quadrupole mass spectrometer, specifically a Pfeiffer Vacuum QMS 200 equipped with a tungsten grid ion source. A custom-made differential pumping system has been added enabling constant fluxing of gas inside the gas phase analysis chamber with an Alicat MC-series mass flow controller through a capillary stainless-steel tube (120 μm internal diameter);

- A Proton Transfer Reaction Mass spectrometer (PTR-MS) equipped with a Time-of Flight detector (TOF) from Kore Technology. The tool is equipped with a Hollow Cathode Glow Discharge Ion Source (GD), which could be started with water for proton reaction or with other gas (argon Ar and krypton Kr) for charge transfer reaction.

<u>Gas test bench for measurements above the lower flammability limit</u>

A general overview of the setup for sensor testing above the lower flammability limit is described in Figure 41. Combustible gasses (methane and hydrogen) come in different cylinders with molar ratios defined in D2.1 and are mixed by the mass flow controllers (Bronkhorst) with different ratios of air in the sensor chamber, where the detectors are situated. The chamber vent is under continuous nitrogen flow and in case of detonation, a pressure release valve directs the hot gasses to a secondary vent. Moreover, pressure and temperature sensors are connected to actuated valves that allow passage of gasses only when the operator starts the experiments, and only if the sensors are not in a triggered state (i.e. below safety setpoint). The sensor chamber is a steel pressure vessel rated to 7 bars.

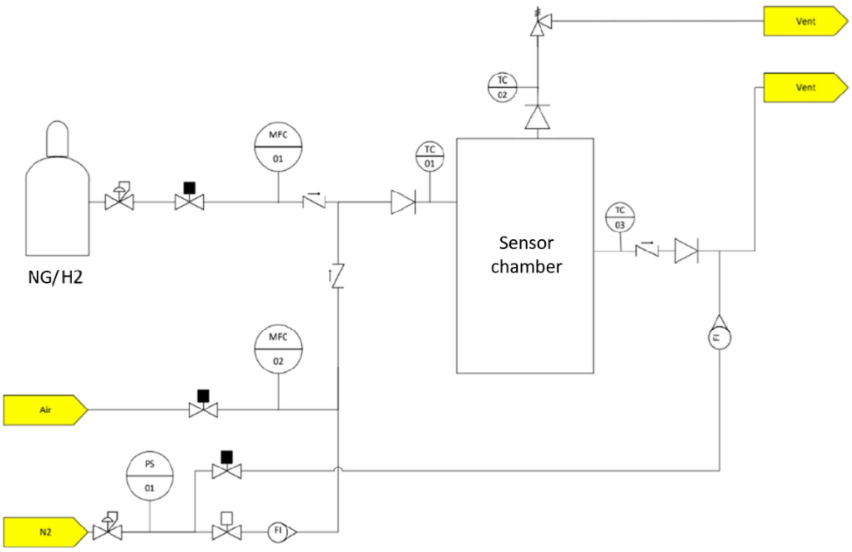

Figure 41. Piping and Instrumentation Design of the test bench

<u>Safety procedures adopted during the experimental campaign</u>

To ensure the safe operation of the test benches and the gas mixing systems to be used for testing the leak detectors, several key safety considerations will be implemented.
Suitable test benches will be used to evaluate the leak detectors at concentrations below the Lower Flammability Limit (LFL) at the Gas Qualification Laboratory. To ensure safety, only cylinders containing flammable or explosive gases with certified concentrations below the LFL will be employed. The test environment is continuously monitored with temperature, humidity, and pressure sensors, which are connected to a loop system with a shut-off valve. This setup helps prevent the creation of conditions that could lower the LFL within the testing range. Additionally, the gas flow controller used in the test setup will be

regularly inspected and calibrated to maintain accurate gas compositions and minimize any deviations from target values.
In the case of tests above the lower flammability limit, the key consideration was on compartmentalizing the sensor chamber, where an explosion event is most probable, with flame arrestors. The camera is also equipped with a pressure release valve to direct possible combustion byproducts in a separate vent. Combustible gasses (methane and hydrogen) come premixed in 1 L, 40 bar cylinders, and the mass flow controllers are used to maintain flow at ambient pressure in the chamber. Pressure and temperature sensors in the chamber are connected to an Emergency Shut Down system (ESD) that shut solenoid valves on the NG/H2 and air lines. Moreover, the ESD is also connected to a solenoid valve that opens the nitrogen purge line to flush the chamber. The entire setup is installed in a ventilated test bench to address possible leaks from connections, and all the lines are controlled for leaks with a portable hydrogen/helium sniffer at regular intervals. The test bench is enclosed in a steel cage in case of chamber failure.

In both laboratories. personnel working with the test setup will adhere to strict safety protocols, including the use of personal protective equipment such as flame-resistant clothing, ear mufflers, gloves and gas masks, when needed. Regular safety drills are conducted at FBK, managed by the FBK safety office, to ensure all personnel are familiar with emergency procedures, including evacuation plans and gas shut-off protocols.

Components and electrical equipment used in the test area will comply with ATEX directives to minimize ignition risks in potentially explosive atmospheres.

6.1.2. INIG test bench and main components

GU_84 test bench

The primary function of the GU_84 test bench is to generate specified gas volume flow rates. The schematic flow diagram of the test bench is shown in Figure 42. The task of generating a specific gas volume flow rate is achieved through the MCR flow controller - 50 l/min - D - 5M - 5IN - RS485 MODBUS - HC – GAS. The flow controller enables the generation of prescribed emission levels of methane, carbon dioxide, hydrogen, and their mixtures.

Additionally, the test bench is equipped with a set of devices to simulate atmospheric conditions that may interfere with measurements (Table 26):

- Active background heating mat: This mat allows for heating the background to a temperature 10°C above ambient, simulating conditions where the background temperature is elevated.
- Three Monsoon EXO TERRA misting nozzles: These nozzles enable adjustments to the test bench's operating parameters, including air humidity and misting to simulate varying environmental conditions.
- Piezoelectric buzzer: This buzzer generates noise at a frequency of 3.7 kHz ± 0.5 kHz with a sound level of approximately 82 dB, simulating ambient noise conditions.
- AABCOOLING Super Silent Fan 12 Pro: This fan serves as a wind generator, capable of producing wind speeds of up to 5 m/s to simulate air movement.

Table 26. Main characteristics of the installed equipment.

Equipment	Producer	Type	Range
Flow regulator	Alicat	MCR	0,005-50 l/min,
Thermohygrobarometer	Adafruit	BME280	-40 - 85 °C ± 1 °C

			10 - 100 % RH ± 3 %RH 300-1100 hPa ± 1 hPa
Spray nozzles	Exo Terra	Monsoon	from air humidity up to 98% in 2% steps
Heating mat	INiG's own design	-	from ambient temperature up to 10°C above ambient temperature in 0.5°C steps
Fan	AABCOOLING	Super Silent Fan 12 Pro	from 0 to 5 m/s in steps of 0.2 m/s.

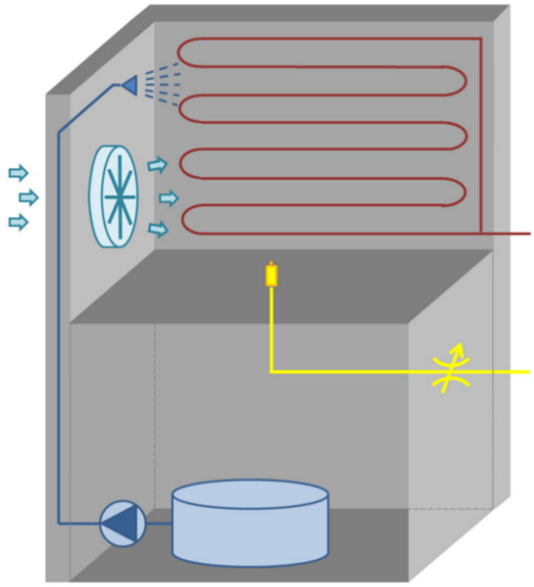

Figure 42. Scheme of the test bench for leakage detection devices.

<u>The Modular Gas Mixer for making gas mixtures</u>

The Modular Gas Mixer is shown in Figure 43. The invention, which is a part of the mixing plant, can be used during experiments, research and development works carried out with the use of gas-burning devices, gas fittings and control and measuring apparatus, as well as during certification tests of gas devices.

In addition, the mixing plant can be used on industrial lines where there is a need to use a multi-component gas mixture in a technological process in which, apart from the percentage composition, other parameters are also controlled, e.g., energy (Wobbe index, heat of combustion) or physical (density of the mixture).

The component gases are fed from the installation or tanks to the mixer to each module.

Figure 43. Modular gas mixer for preparing gas mixtures.

The mixer works in two modes:

1. In the first mode, two independent segments form independently two mixtures.
2. In the second mode, as a single multi-component mixer, the amount of ingredients resulting from the number of modules in both segments can be mixed.

The mixer gives the possibility of independent operation of several devices or receiving installations at the same time, allowing, if necessary, to increase the number of components in the mixture without additional costs of expansion and downtime in the event of occasionally usage of gas mixtures with a larger number of components.

Safety procedures adopted during the experimental campaign

To ensure the safe operation of the GU_84 test bench and the modular gas mixer, several key safety considerations will be implemented.
The test bench operates e.g. with methane, carbon dioxide, hydrogen, and their mixtures, requiring strict control of gas flow rates and concentrations to prevent hazardous conditions such as leaks or unintended combustion. The gas flow controller and Modular Gas Mixer will be regularly inspected and calibrated to maintain precise gas compositions, minimizing deviations from target values.

The test environment will be continuously monitored for temperature, humidity, and pressure variations using thermohygrobarometers, ensuring stable and controlled testing conditions. Additionally, safety mechanisms

such as automatic shut-off valves, ventilation systems, and gas detectors will be integrated to promptly detect and mitigate potential gas leaks.
Personnel working with the test setup will adhere to strict safety protocols, including the use of personal protective equipment such as gas masks, flame-resistant clothing, and gloves. Regular safety drills will be conducted to ensure all personnel are familiar with emergency procedures, including evacuation plans and gas shut-off protocols.

Special attention will be given to the simulation of environmental conditions using heating mats, misting nozzles, and wind generators, ensuring that these elements do not interfere with safe gas handling. Electrical equipment used in the test area will comply with ATEX directives to minimize ignition risks in potentially explosive atmospheres.

6.2. Testing procedure

As reported in the testing protocol (D2.2), for each device to be tested, different sensing performances, both in the presence and of NG (methane) and H_2/NG blends will be investigated, namely:

- Calibration curves;
- Short term stability;
- Long term stability;
- Alarm setpoint check;
- RH% test;
- Response and recovery evaluation;
- High gas concentration operation;
- H_2 poisoning;
- LOD evaluation for ultrasonic leak detectors.

In the following sections the details of the testing procedures that will be adopted at FBK and INIG are described.

6.2.1. FBK testing procedure

Besides the type of measures to be performed, the following main steps will be taken for performing the tests:

1. Check and validation of the setup conditions;
2. Installation of the device to be tested in the measuring setup;
3. Steps for performing the specific test.

Check of the setup conditions

All the testing measurements will be performed in a dynamic condition, as reported in D2.2. Therefore, the test benches are equipped with cylinders with certified concentrations of gases, dual-stage pressure reducers, stainless steel tubes and mass flow controllers to manage and regulate the gas mixture composition and gas flow to be injected into the device to be tested. To check and validate the setup conditions before proceeding with the measurement, the following steps will be carried out:

1. Opening the cylinder valve and checking the cylinder pressure at the high-pressure gauge in the dual-stage pressure reducer. If pressure <30 bar, continue with the next step.

2. Close the cylinder valve and valve of the dual-stage pressure reducer to leave the first section of the line under pressure.
3. Re-check the pressure after one hour, if it varies by less than 2% of the initial pressure value, proceed to the next step.
4. By using the regulators present in the gas lines, adjust the pressure of the second section of the gas line (from the dual-stage pressure reducer to the gas chamber/device allocation) to 1.5-2 barg.
5. Close the valves upstream and downstream the second section of the gas line, to leave it in pressure.
6. Re-check the line pressure at the low-pressure gauges after 20 minutes. If it varies by less than 10% of the initial pressure value, proceed to the next step.
7. After reopening the valves along the line, from the cylinder to the exhaust, flow 100 sccm of the test gas (in the alternative, dry air) from the mass flow controllers. Check, using the reference mass flow controller downstream, that the flow is correct (<2% error from nominal value) and stable with a 10-minute test injection.

If all these steps are passed successfully, it is possible to proceed with the device installation in the measuring setup. Eventual non-conformities identified in the setup conditions will be recorded in the dedicated section of the internal management software (Fablims).

The following scheme represents the steps to be performed during the setup condition check (Figure 44):

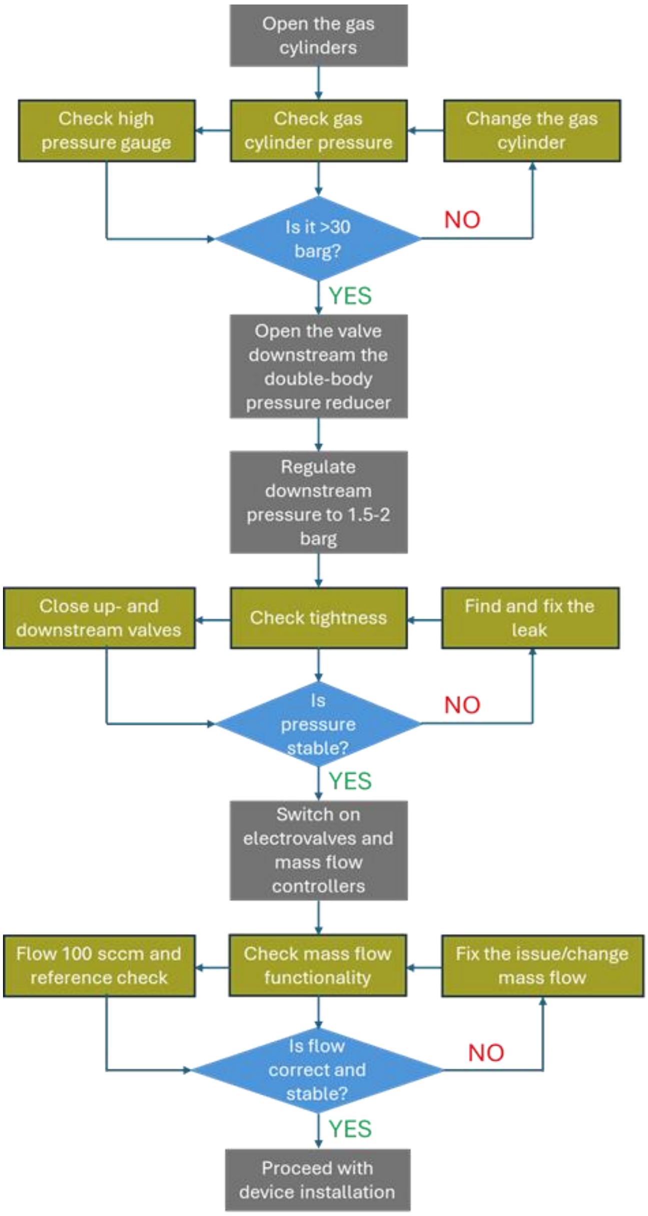

Figure 44. Setup condition check scheme.

Installation of the device to be tested in the measuring setup

The device to be tested will be prepared and mounted as near to typical use as possible, in accordance with the instruction manual, including all necessary interconnections, initial adjustments and initial calibrations. If any, relevant standard guidelines will be strictly followed. The FBK procedure includes the following steps:

1. Registration of the device to be tested in Fablims, the internal management software.
2. Depending on the characteristics of the device, installation is done in a targeted manner:
    a. Devices equipped with an internal pump: an outlet for excess gas will be provided. An inlet flow limiter to the setup will be added. Verify that the flow rate of the equipment is 10% higher than the flow rate at which the flow failure signal is activated.

b. Verify that the excess flow at the outlet must be high enough to prevent reverse flow. Typically, an excess flow of about 20% is achieved.
c. Devices without an internal pump: an excess gas outlet is not needed. The gas flow is let pass through a device, which will be connected to an exhaust tube downstream. Verify that the exhaust flow is equal to the upstream flow entering the device.
d. Devices having integral sensors: the entire equipment will be exposed to the test conditions without the removal of any normally attached parts, including any sampling probe for tests. For this purpose, such devices will be placed in a sealed chamber connected to the flowing system (upstream) and to an exhaust (downstream).
e. For all the other sensors: where possible, the flowing system will be directly connected with the inlet of the sensor contained in the leak detectors using a suitable connector.

3. Verify that the sealed chamber (in the case of devices with integral sensors) or the connector (in the case of the other devices, when applicable) are tight and leak-proof. To do this:
   a. Bring the setup in which the sensor to be tested was installed to a pressure of 1.5-2 barg.
   b. Close the valves upstream and downstream of the device and wait 10 minutes.
   c. Check that the pressure change is less than 5% of the initial value.

If all these steps are passed successfully, it is possible to proceed with the device specific test. Eventual non-conformities identified in the setup conditions will be recorded in the dedicated section of the internal management software (Fablims).
The following scheme (Figure 45) represents the steps to be performed during the device installation in the test setup (gas bench).

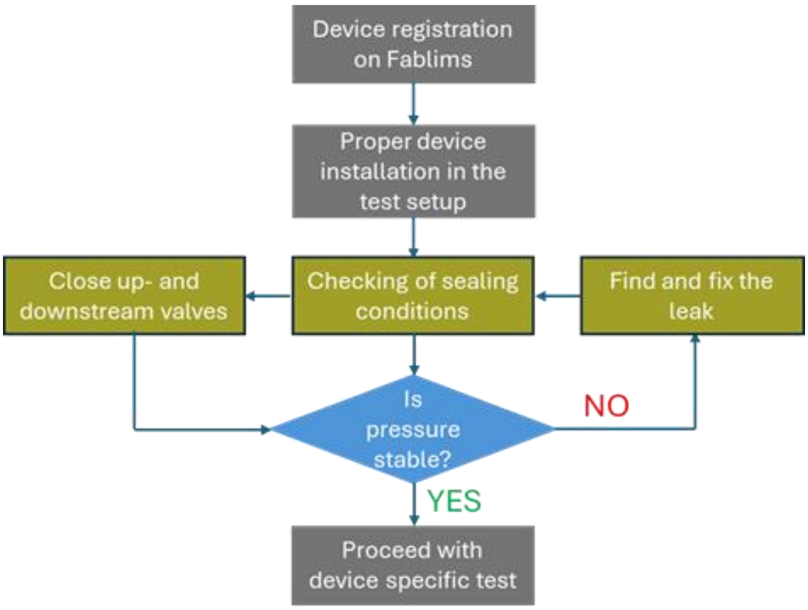

Figure 45. Scheme of steps to be performed for device installation in the test setup.

<u>Steps for performing the specific test</u>

1. Registration of the test to be performed, and the related process, on Fablims.
2. Switch on the controllers of the test setup and leak detector to be tested.
3. Check that the recording and transmission (where possible) of data by the device work properly.

4. Check that the environmental sensors (humidity and temperature) are working correctly.
5. Create a folder on the PC in use for saving the data of the measurement.
6. Write the injection file (.txt) and upload it to the custom-made software, prepared in Labview, for setup control.
7. Allow the equipment adequate acclimation time. Each time the equipment is subjected to a different test condition, the equipment must be allowed to stabilize under the new conditions before measurements are taken. Before taking measurements, the equipment must stabilize under the new conditions.
8. Open the cylinders required to perform the test.
9. Stabilize the sensor for at least one hour using zero air as a reference. During this step period, use the temperature and humidity sensor contained in the setup to check that:
    a. the gas flowing into the leak detector is at a temperature constant to ±2 °C within the range of 15°C to 25°C, a temperature which should be kept constant throughout the duration of each test, unless otherwise specified for the particular test;
    b. the gas flowing into the leak detector is at a relative humidity (RH%) over the range of 20% to 80% throughout each test unless otherwise specified for the particular test. The humidity of zero gas and test gas shall be controlled to within ±10% RH%.
10. Carry out the test that is to be performed.
11. Check, using the experimental setup management software and the related datalog, that the test was carried out correctly and that there were no issues.
12. Check that the detector has collected and transmitted (where possible) data throughout the measurement.
13. Carry out an analysis of the collected data.
14. If no problems were found that would lead to the experiment being repeated, restore the setup to carry out the next test.

Data collected during the experiment will be properly saved in the dedicated PC folder and uploaded in Fablims, along with potential issues encountered during the test.

The following scheme (Figure 46) represents the steps to be performed for executing a specific test for the leak detectors.

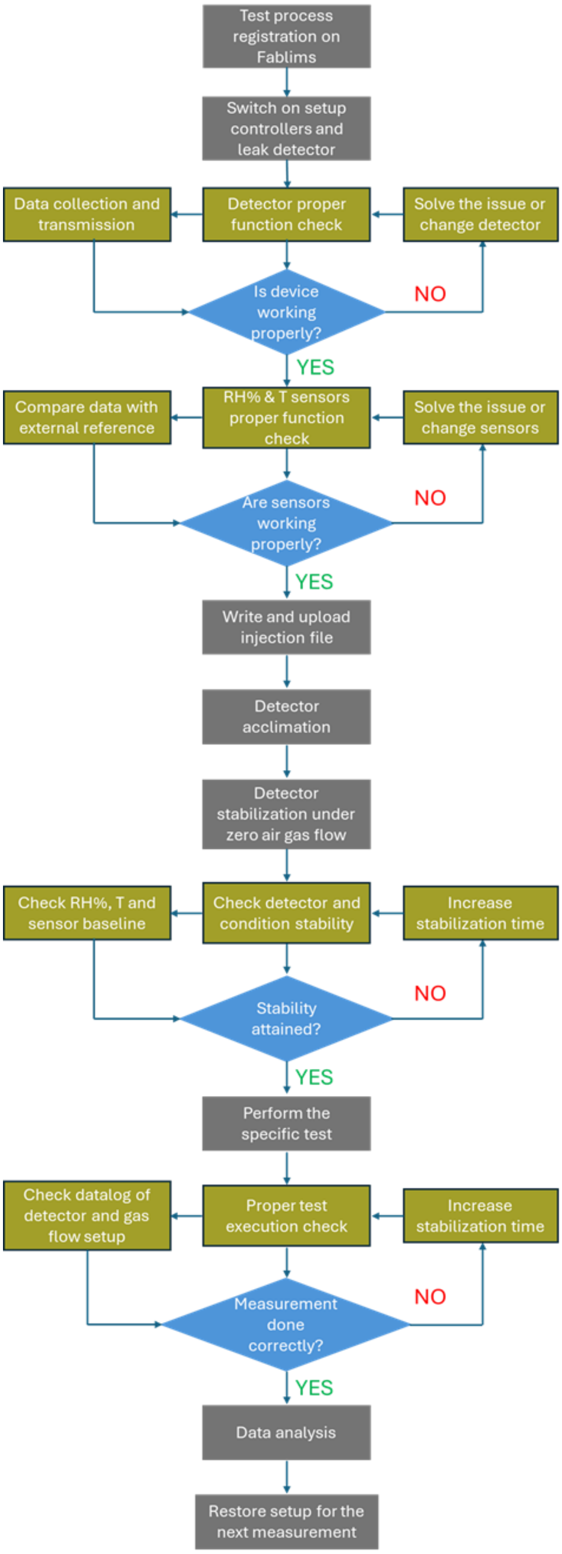

Figure 46. Scheme of the approach used at FBK for performing a specific test for a leak detector.

6.2.2. INIG testing procedure

The testing procedure will involve the following primary steps in addition to defining the type of measures to be conducted:

1. Verification and validation of the setup conditions.
2. Installation of the device to be tested within the measurement setup.
3. Execution of the specific test procedures.

<u>Verification and validation of the setup conditions</u>

Due to the necessity of conducting tests under dynamic conditions, the measurement setup will be powered by gas mixtures obtained using the gas modular mixer (Figure 43).

The INiG procedure outlines the following steps for checking the settings on the measurement platform:

Gas Mixture Composition Control Before Measurements:

- The composition of the gas mixture supplied to the setup will be chromatographically verified using a laboratory gas chromatograph from PerkinElmer with an Arnel 1115 system.
  - The supplied gas mixture composition will be deemed correct if the target value falls within the range determined by the chromatographic measurement result, accounting for uncertainty.

Control of Target Temperature and Humidity Parameters in the Measurement Area:

- Target temperature and humidity parameters in the measurement area will be monitored using an additional/external thermo-hygrometer.
  - The measured relative humidity (RH) value should not deviate by more than 5% RH from the target value.
  - The measured air temperature should not deviate by more than 2°C from the target value.

Control of Gas Flow Rate:

- The desired gas flow rate will be regulated at the setup using a mass flow regulator. Before commencing measurements, it is essential to verify and record the gas composition introduced into the regulator.
  - The gas composition should align with the chromatographic analysis result.

<u>Device installation in the measurement setup</u>

The device under test will be prepared and installed to closely simulate typical usage as outlined in the instruction manual. This process includes establishing all necessary interconnections, making initial adjustments, and performing required calibrations.

Devices with integral sensors cannot be tested using the INiG measurement station setup. For devices equipped with internal pumps and those without, the installation procedure involves registering the device in the sample log and assigning a unique sample number.

Once registered, the device will be positioned on the test stand with the power on, ensuring that the sample intake system aligns directly over the gas flow nozzle. For devices featuring acoustic sensors, they will be situated outside the measurement area with the distance from the gas flow nozzle adjusted according to the manufacturer's guidelines.

During installation, efforts will be made to replicate typical operational conditions specified in the instruction manual. This encompasses setting up all required connections, making initial adjustments, and conducting any necessary calibrations to prepare the device for testing.

Execution of the specific test procedures

This stage will be executed as follows:

- Record the following details in the measurement protocol: sample number, type of test being conducted, date, and identification of the individual(s) conducting the test.
- Power on the controllers of the test setup and the leak detector to be tested.
- Ensure proper functioning of data recording and transmission (where applicable) by the device.
- Configure the desired test conditions on the control panel of the measurement setup.
- Allow sufficient time for the equipment to acclimate. It is important that the equipment stabilizes under the new conditions before measurements are taken.
- Stabilize the sensor for at least one hour using zero air as a reference. During this period, use the temperature and humidity sensor within the setup to ensure the following:
  - The gas flowing into the leak detector maintains a constant temperature within ±2°C within the range of 15°C to 25°C, which should remain consistent throughout each test unless specified otherwise.
  - The gas flowing into the leak detector maintains a relative humidity (RH%) between 20% and 80% throughout each test unless specified otherwise. The humidity of the zero gas and test gas should be controlled within ±5% RH%.
- Supply the gas with the assumed and verified composition from the gas mixer to the measurement station.
- Proceed with conducting the intended test.
- Continuously record in the measurement protocol the prevailing conditions at the station and the device readings.
- Analyse the collected data thoroughly.
- If no issues are detected that would necessitate repeating the experiment, restore the setup for the next test.

All experimental data will be appropriately saved in the designated PC folder.

7. TESTING OF GAS VOLUME CONVERSION

The testing of the gas volume converter selected during the activities of Task 2.1 and indicated in the Deliverable D2.1 will be performed in accordance with the protocol developed in Task 2.2 and indicated in Deliverable D2.2.

<u>**Objective of the test:**</u> The test is separated into two activities:

- Error evaluation of current implemented methods AGA8 and SGERG-88 for NG:H2 admixtures up to 30 vol-% of hydrogen and pure hydrogen.
- Testing of flow computer with EU-type examination certificate for methods AGA8, SGERG-88 and method valid for pure hydrogen.

<u>Operative procedures adopted during the experimental activities</u>: in the next sections, information about the measurement equipment, the materials, and the operative procedures to respect the metrology requirements indicated in WP2 will be shown. Furthermore, the test report and documentation to ensure comparable information and data in the experimental activity will be indicated.

The detailed protocol is reported in Deliverable D2.2.

7.1. Volume conversion testing room

Error evaluation will be performed with INIG tool. For the purpose of interlaboratory comparisons (ILCs) calculations of selected points (at least 6 points for tested methods AGA8, SGERG88 and GERG2008) will be performed with the Enagás tool. Calculations will be made based on the composition of Gas 1 from the EN ISO 12213-2 standard. ILC acceptance criterion is established in the same compressibility factor value to 5 decimals.

Flow computer testing will be performed in Enagás Metrology and Innovation Centre (CMI), Zaragoza (Figure 47).

Figure 47. Photo of Enagás instrumentation testing room.

7.2. Volume conversion testing procedure

This testing procedure has the purpose of checking the register of the selected flow computer (compressibility factor, conversion factor, error, etc.) for different hydrogen concentrations (inside and outside of limits defined in standards for compressibility factor calculation). It is not considered detailed testing according to standard EN 12405 'Gas meters, Conversion devices, Part 1. Volume conversion'.

The testing procedure in Enagás Laboratory consists of the following steps:

- Delivery of flow computer in Laboratory.
- Inspection for external damage and checking of technical characteristics and documents. If necessary, issues reporting.
- Installation and connection of flow computer signals.
- Checking of signals.
- Configuration of a method for compressibility factor calculation.
- Configuration of gas composition.
- Configuration of pressure.
- Configuration of temperature.
- Compressibility factor, conversion factor, error, etc. register.
- For other compositions, pressure and temperature, the process is repeated in the same way.
- Disconnection of the flow computer.
- Box up flow computer using the same packaging, boxes, etc. the goods arrived with and storage until collection.

8. TEST REPORT AND DOCUMENTATION

To ensure intercomparison and proper experimental data management, Task 2.2 and Task 3.1 agreed to define the general rules for the creation of reports and other types of documentation to be shared among partners.

Specifically, the results will be gathered by the task leader and will be shared in proper formats according to the Data Management Plan (DMP) after having been treated with different partners for validation.

Some general rules will apply to all the measuring device technology as reported below:

- Detailed description of the measurement setup used (if different from what is already specified in the testing protocol), accompanied by diagrams and photos.
- Detailed description of the exact configuration of the device, including use of or removal of the optional accessories.
- Description of how the tested device was installed in the measurement setup, correlated with pictures.

The additional guidelines for each measuring device technology are presented in the following sections.

Before the report or the datasets are released to the public, the data will be anonymised.

8.1. Gas transmission flow meters' report guidelines

As agreed in Task 2.2 and indicated in deliverable D2.2, rules will be followed in reporting the results of the experimental activities. Specifically, the results will be gathered by the task leader and will be shared in proper formats according to the Data Management Plan (DMP) after having been treated with different partners for validation.

Different information and data shall be reported for ageing and calibration. Several data will be recorded in the laboratories depending on the specific adopted procedure in order to ensure comparable results. Some of the most important data are listed below while the definitive list will be defined in Task 3.3.

8.1.1. Ageing

The data included in the report should include:

- Start and end date of the aging;
- Fluid composition;
- Aging fluid conditions, i.e., ageing pressure and temperature;
- Aging operative conditions, i.e., static or dynamic
- Anomalies or errors occurred during the aging in order to investigate the potential causes and avoid their repetition

8.1.2. Calibration

As a result of each test, the calibration certificate will be issued annexed with the calibration protocol. The calibration/testing uncertainty at each experimental point and a statement about the confidence interval (e.g., k=2 or 95 %) will be provided.

- Data referring to the DUT:
  - Owner of the meter

- Manufacturer
- Model
- Set number
- Meter number
- Type, e.g., turbine, rotary, ultrasonic, etc.;
- Calibre (if applicable);
- Year of manufacturing and of last calibration (if applicable)
- Nominal diameter (DN);
- Nominal pressure (PN);
- Maximum flowrate (Q_{max})
- Minimum flowrate (Q_{min})
- Transitional flowrate (Q_t)

- Data referring to the results of the calibration:
  - Date and time of calibration;
  - Name of the responsible laboratory
  - Nominal calibration pressure
  - Testing fluid
  - Uncertainty (k=2)
  - Nominal flow point Q_{nom};
  - Errors of the meter;
  - Average error;
  - Number of repetitions.
  - Testing final results, i.e., succeed or not succeed;

8.2. Gas distribution flow meters' report guidelines

As agreed in Task 2.2 and indicated in deliverable D2.2, rules will be followed in reporting the results of the experimental activities. Different information and data shall be reported for ageing and calibration.

Several data will be recorded in the laboratories depending on the specific adopted procedure in order to ensure comparable results. Some of the most important data are listed below while the definitive list will be defined in Task 3.3.

8.2.1. Ageing

The list of data for calibration are reported below:

- Start and end date of the aging;
- Fluid composition;
- Aging fluid conditions, i.e., ageing pressure and temperature;
- Aging operative conditions, i.e., static or dynamic
- Anomalies or errors occurred during the aging in order to investigate the potential causes and avoid their repetition

8.2.2. Calibration

As a result of each test, the calibration certificate will be issued annexed with the calibration protocol. The calibration/testing uncertainty at each experimental point and a statement about the confidence interval (e.g., k=2 or 95 %) will be provided.

Several data will be recorded in the laboratories depending on the specific adopted procedure in order to ensure comparable results.

- Data referring to the DUT:
  - Owner of the meter
  - Manufacturer
  - Model
  - Set number
  - Meter number
  - Type, e.g., diaphragm, ultrasonic, thermal mass.;
  - Accuracy class;
  - Calibre (if applicable);
  - Year of manufacturing and of last calibration (if applicable)
  - Max/Nom pressure (PN);
  - Maximum flowrate (Q_{max})
  - Minimum flowrate (Q_{min})
  - Transitional flowrate (Q_t)
  - PT &/or T correction
  - Base temperature (T_b)
  - Base pressure (p_b).

- Data referring to the results of the calibration
  - Date and time of calibration;
  - Name of the responsible laboratory
  - Nominal calibration pressure
  - Testing fluid
  - Nominal flow point Q_{nom};
  - Average error;
  - Number of calibration points;
  - Number of repetitions;
  - Error;
  - Uncertainty (k=2);
  - Testing final results, i.e., succeed or not succeed;

8.3. Pressure transmitters' report guidelines

As agreed in Task 2.2 and indicated in deliverable D2.2, rules will be followed in reporting the results of the experimental activities. Different information and data shall be reported for ageing and calibration. In the following section, the minimum set of data to be recorded is indicated.

8.3.1. Ageing

The list of data for calibration are reported below:

- Start and end date of the aging;
- Fluid composition;
- Aging fluid conditions, i.e., ageing pressure and temperature;
- Aging operative conditions, i.e., static or dynamic

- Anomalies or errors occurred during the aging in order to investigate the potential causes and avoid their repetition

8.3.2. Calibration

As a result of each test, the calibration certificate will be issued annexed with the calibration protocol. The calibration/testing uncertainty at each experimental point and a statement about the confidence interval (e.g., k=2 or 95 %) will be provided. The data below will be recorded for each calibration point:

- Pressure indicated by the reference instrument;
- Indication of the calibration instrument, i.e., the pressure reading or the measured analogue value;
- Ambient conditions, i.e., temperature, relative humidity and pressure;
- Tightness of the installation, i.e., Pass / Not pass;
- Indication parameters:
    - of the instrument to be calibrated
    - of the instruments in the measuring system

8.4. Trace water sensors' report guidelines

For each water dewpoint sensor or analyser (trace water sensor) calibration, a traceable calibration certificate will be issued following INRIM calibration procedures. The calibration procedures refer to INRIM Calibration and Measurement Capabilities (CMC) of humidity primary and secondary standards recognized at the international level by the MRA and available in the BIPM KCDB.

Alongside the detailed information about the sensor (owner, type, s/n) and the measurement conditions and calibration time and date, the calibration data reported in each calibration certificate will include the following information (Table 27):

Table 27. Data reported for trace water.

Calibration point	t_{REF} (°C)	t_{UUT} (°C)	t_{UUT} - t_{REF} (°C)	U (°C)
1	X	X	X	X
2	X	X	X	X
3	X	X	X	X

with the symbol meaning:

t_{REF}	=	reference frost-point temperature
t_{UUT}	=	frost-point temperature read by the unit under test
U	=	expanded calibration uncertainty

and the following details about the uncertainty estimates:

The calibration uncertainty estimated the uncertainty of the applied conditions, the resolution and the short-term repeatability of the unit under calibration. The long-term stability and reproducibility of the condensate on the mirror were not considered. The expanded calibration uncertainty U will be expressed as the standard uncertainty multiplied by the coverage factor k = 2, which corresponds to a confidence interval of approximately 95 % for a normal probability distribution. The coverage factor, *k* is the numerical factor used as a multiplier of the combined standard uncertainty in order to obtain an expanded uncertaintyLeak detectors' report guidelines

8.5. Leak detectors' report guidelines

The report will be drafted following the guidelines provided in reference standards, where applicable. While these standards do not offer detailed instructions on organizing technical reports, but rather provide general suggestions, the structure of the report is determined by the project partners responsible for testing the leak detectors in the THOTH2 project (Work Package 3), i.e., FBK and INIG.

The report will be organized as follows, for each device tested:

- Graphs and detailed description of the technical specifications of the measurement carried out (e.g., trend of methane concentration vs. time, etc.).
- Data/graphs of the response and behaviour of the sensor during the measurement performed.
- For measurements carried out in the presence of hydrogen, analytical comparison of the data obtained with respect to what was obtained in measurements carried out in the presence of methane.
- "Final considerations" section, containing insights into any potential improvements, recommendations, or areas for further development based on the test results. Overall, this section aims to offer a conclusive and critical assessment of the sensor's performance, drawing on the data and observations gathered during the test session.
- Any further discussions/comments/notes, including non-conformities identified and reported in Fablims.

The report will be uploaded to the internal management/quality software (Fablims). Anonymisation of the data will be carried out before making it available to the public.

8.6. Gas volume converters' report guidelines

A test report will be organized as follows:

- Detailed description of the testing setup used.
- Tables with a detailed description of the configuration of the device for each condition.
- Tables with the register for each condition (compressibility factor, conversion factor, error, etc.).
- Any error or failure occurring during the testing activities.

Before the report is released to the public, the data will be anonymised.

9. CONCLUSION

Information on the test benches of the consortium participants chosen for the experimental activities was presented in this deliverable. Four laboratories will be involved in testing gas transmission meters, three for low-pressure and low flowrate gas distribution meters, two for pressure transmitters and leak detectors, and one for trace water humidity sensors and volume converters.

Since the tested measuring devices will be transported among laboratories, particular attention will be paid to handling and checking their status before starting the testing procedure to avoid misleading results. For this purpose, a preliminary acceptance procedure has been provided, and it will be confirmed or reviewed in case issues are noted during the performance of Task 3.3.

As shown, each laboratory will rigorously adopt the protocol provided in Task 2.2 through a dedicated procedure described in the document. Even if not described in deep detail, attention will be given to safety aspects. Before starting the text with H2 or any other flammable mixture, an internal procedure will be adopted to minimize the risk of accidents or any other events that could damage any operator or things. Furthermore, the indications and technical suggestions provided by the manufacturers once the use of measuring device in the presence of H2 or H2NG is adopted.

REFERENCES

[1] Turkowski, M., Dyakowska, E., Szufleński, P., Jakubiak, T. *Construction of the new gas meter high pressure calibration facility – Technical and metrological problems*. Flow Measurement and Instrumentation, vol. 60, pp. 57-60.

[2] https://ilac.org/about-ilac/role/

[3] Explosive atmospheres — Part 29-1: Gas detectors — Performance requirements of detectors for flammable gases, EN 60079-29-1:2016+A11:2022, European Standard, 2022.

[4] Explosive atmospheres — Part 29-2: Gas detectors — Selection, installation, use and maintenance of detectors for flammable gases and oxygen, EN 60079-29-2:2015, European Standard, 2015.

[5] Fixed Ultrasonic Gas Leak Detectors (UGLD) — General requirements and test methods, EN 50724:2023, European Standard, 2023.

APPENDIX

The list of useful links to public documentation regarding the measuring equipment installed in the testing benches and cited in the deliverable is reported below.

Digital barometer:

Vaisala PTB330: https://www.vaisala.com/sites/default/files/documents/WEA-MET-ProductSpotlight-PTB330-B212229EN-A.pdf

Flow controller:

Alicat MC-series: https://www.comhas.com/wp-content/uploads/2016/12/Alicat_Mass_Controller_Specs.pdf

Alicat 50 SLPM: https://store.alicat.com/products/mcp-50slpm-d

Gas chromatograph:

PerkinElmer model 1115: https://www.perkinelmer.com/it/product/arnel-model-1115ppc590-narl8105

Quadrupole mass spectrometer:

Pfeiffer Vaccum QMG 200: https://www.pfeiffer-vacuum.com/global/en/shop/categories/QUADRUPOLE_MASS_SPECTROMETER

Multi-channel flow ratio & pressure controller:

MKS 647C: https://download.siliconexpert.com/pdfs/2016/8/9/15/13/3/550/mksi_/manual/561030335513839647.pdf

Brooks 0260: https://cdn.brooksinstrument.com/-/media/brooks/documentation/products/accessories-and-software/secondary-electronics/0260/secondary-electronics-data-sheet-0260.pdf?rev=5f521df13a9c46c98b2187480d852655&sc_lang=en&hash=7d2691727407e0eafdcdccef f60e95a7

Multifunction calibrator:

Druck DPI 620: https://www.epsas.it/wp-content/uploads/2023/08/ULTIMA-VERSIONE-Druck-DPI620G-Datasheet-EN.pdf

Multimeter:

Fluke 8508A: https://www.testequipmenthq.com/datasheets/FLUKE-8508A-Datasheet.pdf

Pressure controller:

Druck Pace 6000: https://www.bakerhughes.com/it/druck/pressure-controllers-and-indicators-pace

Pressure transmitter:

Aplisens PC-28: https://aplisens.com/pdf/produkty/PC-28.pdf

Aplisens APR-2000GDP: https://aplisens.com/pdf/produkty/APR-2000G.pdf

Keller AG 33X-Ei series: https://www.kraftteknik.com/uploaded/katalog/33xei_seri.pdf

Rotary piston gas meter:

Elester RM-TS IRPP: https://docuthek.kromschroeder.com/download.php?lang=de&doc=34215

Metrixitalia CGR-05: https://www.metrixitalia.it/pdf/CGR-FX%20Pistoni%20Rotanti%20ENG.pdf

Temperature / humidity sensor:

Sensirion SHT40: https://sensirion.com/products/catalog/SHT40/

Temperature / humidity / pressure Sensor:

Adafruit BME280 I2C: https://www.adafruit.com/product/2652#description

Time-of-Flight detector:

Kore Technology: https://kore.co.uk/service-support/introduction-to-tof-ms/

Turbine meter:

Honeywell SM-RI-X: https://prod-edam.honeywell.com/content/dam/honeywell-edam/pmt/hps/products/pmc/gas/distribution/turbine-and-quantometers/sm-ri-x/pmt-hps-elster-sm-ri-x-datasheet-en.pdf

Riels CGT-2: https://www.riels.it/en/cgt-cgt-02-turbine-gas-meters#:~:text=The%20CGT%2D02%20uses%20a,help%20to%20minimize%20pressure%20losses

Wet gas meter:

Bessel BSM series: https://www.bessel.it/wp-content/uploads/2022/07/scheda-tecnica-BSM-web.pdf

Elser NB15: https://docuthek.kromschroeder.com/download.php?lang=de&doc=11583