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

D3.5

Interim report on validation of new or modified test protocols and test methods

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1	12/02/2024	Review	INRIM, UNIBO, FBK
2	11/03/2024	Review	INIG
3	24/03/2024	Review	INIG
4	16/05/2024	Final Review	INIG

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ACRONYMS AND SYMBOLS

DSO	Distribution System Operator
E_n	Index calculated in accordance with ISO/IEC 17043:2011
H2NG	Hydrogen added natural gas (hydrogen content in natural gas from 0 to 100%)
ILC	Interlaboratory comparison
Q_{min}	Minimum Flow Rate
Q_{max}	Maximum Flow Rate
SoA	State of Art
TDLAS	Tunable Diode Laser Absorption Spectroscopy
TSO	Transmission System Operator
U_{Lab}	Expanded uncertainty of determining the Laboratory result
U_{ref}	Expanded uncertainty of determining the result of the reference Laboratory
UUT	Unit Under Test
x	Laboratory result
X	Reference Laboratory result
Z	Compression factor

1. INTRODUCTION AND EXECUTIVE SUMMARY

This WP3 will focus on obtaining experimental validation to update existing research protocols and standards for measuring the quality and quantity of hydrogen and H2NG blends, addressing identified gaps. An experimental and rigorous approach is necessary before introducing new protocols or standards for measuring the quantity and quality of H2NG mixtures. The first objective of WP3 is to validate and confirm the possibility of using new testing protocols and methods to evaluate the performance of measurement equipment for f H2NG mixtures.

Task 3.2 is responsible for the experimental validation of new and/or modified test protocols and methods. The methodology validation activity is carried out on the devices selected in Task 2.1. Where possible, interlaboratory comparisons (ILCs) will be used. Once validated, the methodologies will be implemented in Task 3.3 for conducting testing activities. The cooperation of several research laboratories within the Consortium, along with partners involved in developing or modifying existing research methods, will ensure the achievement of the expected goals.

2. METHODOLOGY

This Interim Report summarizes the activities conducted during the period of 01.11.2023 – 30.04.2024 of Task T3.2 and has been edited in accordance with the following steps:

1. Analyzing the findings from WP2 related to specified test equipment. This analysis is crucial for selecting representative samples for interlaboratory comparisons.
2. Reviewing the findings from Task T3.1 concerning the preparation of test benches for testing, the research capabilities of individual partners/laboratories involved in Task T3.3 and analyzing the defined research procedures in Task T3.1. This analysis is vital for identifying suitable laboratories where ILCs will be conducted as part of the validation process.
3. Determining, for each type of measuring devices planned in the project, laboratories where ILC tests will be carried out.
4. Selecting appropriate samples for ILC tests and specifying the validation method for each type of measuring device.
5. Defining the experimental validation plan.

3. Experimental validation of new and/or modified test protocols and methods

Task 3.2 is responsible for the experimental validation of new and/or modified test protocols and methods. The methodology validation activity will be carried out on the devices selected in Task 2.1.

Test method validation is the process of providing objective evidence to confirm the suitability of a validated test method for its intended use. During validation, parameters characterizing the efficiency of the research method are examined - the validation tests and checks carried out allow us to learn about the capabilities and limitations of the measurement method. By definition, EN ISO/IEC 17025 [1] validation is the confirmation, by examination and provision of objective evidence, that specific requirements for a specifically intended task have been met. In other words, validation is a process that aims to confirm the reliability of the method used, i.e., it is intended to answer the question of whether the method used is correct and whether the obtained test results are reliable. Validation also means the process of confirming that the selected methods meet the requirements for their use and that they are able to detect the tested factor with appropriate precision and accuracy. It is recommended that the techniques used to determine the performance of the method should be one of the following or a combination thereof: calibration using reference standards or reference materials; comparison of results obtained by other methods; interlaboratory comparisons; systematic assessment of factors influencing the result; assessment of the uncertainty of results based on scientific understanding of the theoretical basis of the method and practical experience.

There are two types of tests in the project, i.e. performance and aging tests. Considering the specificity of the research planned in the project, it can be noted that most measurement methods are based on standardized methods with modifications to accommodate different gas mixtures as the medium. Aging tests focus on maintaining specific conditions, e.g. pressure and an appropriate gas mixture. Due to the limited number of laboratories involved in the tests, ILCs will be utilized wherever possible.

3.1 Devices intended for the testing campaign

Below, in clauses 3.1.1 to 3.1.7, based on current information from THOTH2 working and monthly meetings, as well as documents published on SharePoint and discussions among THOTH2 partners, the selected devices for testing in the T3.3 campaign are presented. The devices were chosen based on the SoA for TSO and DSO, and representative devices from these categories were then selected to validate the measurement methods through ILCs.

3.1.1 Gas meters

Table 1 and Table 2 present gas meters used by TSO and DSO, respectively, that were selected for testing. From these gas meters, objects for ILC tests were chosen in Chapter 3.3.

Table 1. Gas meters from TSO selected for testing

Rotary	Turbine	Ultrasonic
Common CGR-01 G100 Common CGR-01 G250 Itron Delta S1 G100 Itron Delta S3 G250	Honeywell SM-RI G160 Honeywell SM-RI G650 Itron Fluxi G160 Itron Fluxi G650	Honeywell Q.Sonic-4 (DN To be defined) KROHNE ALTOSONIC V12 DN100 KROHNE ALTOSONIC V12 DN300 SICK FLOWSIC600 DN100 SICK FLOWSIC600 DN300

Table 2. Gas meters from DSO selected for testing

Diaphragm	Thermal Mass	Ultrasonic
Honeywell BK-G40 Honeywell BK-G65 Sagemcom EG4 G4 (T conversion) Sagemcom EG4 G6 (T conversion) Sagemcom/Siconia/Sacofgas EG G10 (PT conversion) Sagemcom/Siconia/Sacofgas EG G16 (PT conversion)	Metersit Domusnext G6 Metersit Domusnext G25	Flonidan SciFlo G4 Flonidan SciFlo G6 Sagemcom Siconia ES4 EVO G4 Sagemcom Siconia ES4 EVO G6

3.1.2 Pressure transmitters

Table 3 shows the pressure transducers used by TSO and DSO, that were selected for testing. From these pressure transducers, objects for ILC tests were chosen in Section 3.3.

Table 3. Pressure transducers selected for testing

Gauge/Absolute
Rosemount 3051 C Rosemount 3051 T Yokogawa EJA 310A abs Yokogawa EJA 310E abs Yokogawa EJA 430 E gauge Aplisens APC 2000 ALW abs

3.1.3 Temperature transmitters

THOTH2 partners have agreed that, based on the installation conditions of the temperature sensors and their operating principle, the hydrogen content has no influence on their correct operation. Therefore, no experimental tests are planned for temperature transmitters, and validations are deemed unnecessary.

3.1.4 Volume conversion devices - Calculation of compression factor

The impact of hydrogen on volume conversion devices (flow computers) is considered in two aspects, i.e. regarding the resistance of pressure transducers (discussed in a separate clause),and concerning the accuracy of the method for converting the compression factor, Z for hydrogen content above the method range.

Based on the State of the Art (SoA) outlined in Deliverable report D1.1, operators typically use volume converters with algorithms compliant with SGERG-88 according to EN ISO 12213-3 [2] and AGA8-92DC according to EN ISO 12213-2 for converting to base conditions. [3]. For such algorithms, THOTH2 partners have agreed that the tests will testing will involve computer calculations (simulations) using these algorithms, compared to reference methods dedicated to calculating gases withhigher hydrogen content.

Furthermore, ENAGAS, one of the partners, will conduct volume conversion accuracy tests using the KROHNE SUMMIT 8800 flow computer. These tests will include evaluating Performance according to AGA8-92DC, Performance for SGERG-88, and Performance for Hydrogen gas (H_2).

3.1.5 Gas quality measuring devices (gas chromatographs)

Based on the analysis conducted within WP2, the consortium partners have decided to exclude gas analyzers from the scope of the THOTH2 project research. Instead, it has been agreed to focus on water dew point analyzers. The currently used gas analyzers by TSOs and DSOs are not designed to measure hydrogen content in gas, and therefore are inherently unsuitable for measuring NG-H2 blends. For this reason, investigating them in this context has been deemed unnecessary.

3.1.6 Water Dew Point Analyzers

Table 4 shows water dew point analysers used by TSO and DSO, that were selected for testing, from which the objects for ILC tests were chosen in Chapter 3.3.

Table 4. List of possible UUTs selected for the trace-water instruments tests in Task T3.3

UUT Model	Technology	UUT OWNER
Endress+Hauser J22	TDLAS	SNAM/ENAGAS
Michell PROMET EExd	Ceramic Metal-Oxide Moisture Sensor	GAZ-SYSTEM
Michell TDL 600	TDLAS	SNAM
Michell Transmet I.S.	Ceramic Moisture Sensor	GAZ-SYSTEM
SHAW SDT-Ex	Aluminum oxide technology sensors	INRIM
Michell Easidew PRO XP	Aluminum oxide technology sensors	INRIM

3.1.7 Leak detectors

Table 5 shows leak detectors used for TSO and DSO, that were selected for testing, from which objects for ILC testing were chosen in Section 3.3.

Table 5. Leak detectors selected for testing in Task T3.3

Model	**Technology**
MSA Safety Incorporated Altair 4	Catalytic sensor
MSA Safety Incorporated Altair 5	IR + Catalytic sensor
Dräger XAM 5000 4G	Catalytic + electrochemical sensors
GAZOMAT INSPECTRA LASER	Infrared sensors
GMI GT series 40	Semiconductor CH_4 0 - 100 % LEL, 1 % Catalytic Bead, CH_4 0 - 100 % Volume, 1 % Thermal Conductivity
GMI PS200	Catalytic sensors
Huber Günther & C. PROTHEO IR COMPACT	Infrared sensors
Huber Günther & C. METREX 2	Semiconductor + catalytic + thermal conductivity sensors
SENSIT HXG-3P	Semiconductor + catalytic + thermal conductivity sensors
SENSIT LZ-30	Infrared sensors

In addition, one ultrasonic gas leak detector is added to the list as a representative device to be tested for evaluating the performance of acoustic based sensors in the presence of H_2/NG blends. The potential device to be used can be either the Observer® (MSA) or the ULTRA-PRO (Distra).

3.2 Available infrastructures for testing campaign

Below, based on current information from THOTH2 working and monthly meetings, and documents published on SharePoint, including deliverable D3.1, the research capabilities of individual partners for testing specific types of devices are presented.

3.2.1 Gas meters

Tables 6 and Table 7 present the measurement capabilities of individual laboratories for planned calibration measurements of gas meters used by TSO and DSO using various gases, respectively. This data was used to select participants for the ILC tests outlined in Section 3.3. Partners who will be involved in the testing for specific gases in Task T3.3 are highlighted in bold within the tables.

Table 6. Calibration capability for TSO gas meters selected for testing in Task T3.3

Capability to perform calibration / Medium (at working pressure)		
H2NG and 100% H_2	**Air**	**NG**
DNV (up to 35 bar and 500 m^3/h)	**CESAME** (1 ÷ 40/50 bar and 5 ÷ 50,000 Nm^3/h)	**ENAGAS** (3 ÷ 50 barg and 10 ÷ 10,000 Nm^3/h)
		CESAME (1 ÷ 40 barg and 8 ÷ 80,000 Nm^3/h)
		GRTGAZ (1 ÷ 30 barg and 0,1 ÷ 2,000 Nm^3/h)
		GAS SYSTEM 1 to 30 barg and 0,1 ÷ 2,000 Nm^3/h)

Table 7. Calibration capability for DSO gas meters selected for testing in Task T3.3

Capability to perform calibration / Medium (at atmospheric pressure)			
25% H2NG	**100% H_2**	**Air**	**NG**
INIG (0,016 ÷ 100 m^3/h)	INIG (0,016 ÷ 100 m^3/h)	INIG (0,016 ÷ 1,000 m^3/h)	INIG (0,016 ÷ 100 m^3/h)
CESAME (-)	**METAS** (0,01 ÷ 50 m^3/h)	CESAME (5 ÷ 1,000 m^3/h)	CESAME (-)
GRTGAZ (-)	GRTGAZ (-)	ENAGAS (5 ÷ 10,000 m^3/h)	GRTGAZ (-)

3.2.2 Pressure transmitters

Table 8 presents the measurement capabilities of individual laboratories for planned calibration measurements of pressure transducers used by TSO and DSO. This data was used to select participants for the ILC tests outlined in Section 3.3.

Table 8. Calibration capability for pressure transducers selected for testing in Task T3.3

Partners	**Capability to perform calibration**
ENAGAS	Gauge: 0 Pa - 10 MPa Absolute: 80 kPa - 10 MPa
INIG	Gauge: -98 kPag – 13,4 MPa Absolute: 4 kPa – 13,5 MPa
GRTGaz	-

3.2.3 Volume conversion devices - Calculation of compression factor

Table 9 shows the possibilities of individual laboratories in relation to planned tests of volume conversion devices. This data was used to select participants for the ILC tests presented in Section 3.3.

Table 4. Calibration capability for volume conversion devices selected for testing in Task T3.3

Partners	Capability to perform tests
INIG	Tests using equations of state AGA8-92DC, SGERG-88, GERG-2008 [4]
ENAGAS	Tests using equations of state AGA8-92DC, SGERG-88, GERG-2008 [4]

3.2.4 Trace water sensors

Table 10 shows the measurement capabilities of individual laboratories in relation to planned tests of trace water sensors used by TSO and DSO. This data was used to select participants for the ILC tests presented in Section 3.3.

Table 105. Calibration capability for trace water sensors selected for testing in Task T3.3

Partners	Capability to perform tests
INRIM	-20 °Cfp to 95 °Cdp @ 1050 mbar in Air or N2 up to 2 l/min; -75 °Cfp to 0 °Cdp @ 1050 mbar in Air or N2 up to 1 l/min; -30 °Cfp to 70 °Cdp @ approx 1013 mbar (Ambient Pressure, not regulated) in Air up to 20 l/min; -50 °Cfp to 95 °Cdp @ approx 1013 mbar (Ambient Pressure, not regulated) in Air; -100 °Cfp to -20 °Cfp @ 1100 mbar in N2 (Pressure regulated between 200 mbar and 1100 mbar); '-55 °C to -10 °C pressure dew point

3.2.5 Leak detectors

Table 11 shows the measurement capabilities of individual laboratories in relation to planned tests of leak detectors used by TSO and DSO. This data was used to select participants for the ILC tests presented in Section 3.3.

Table 11. Calibration capability for leak detectors selected for testing in Task T3.3

Partners	Capability to perform tests
FBK	Test with calibrated mixtures with concentrations up to 4% v/v of CH_4 and H_2. Test flow range: 1 ÷ 20K sccm, Test pressure: 0/10 bar, Test temp.: 25 °C. Possibility of implementation of setup for measurements up to 100% v/v (to be measured only concentrations out of the explosive limit).
INIG	Mixtures from 0 to 100%, with any proportion of hydrogen and methane in the mixture. Pressure range of the mixture up to 2 bar, temperature 20°C.

3.3 Selecting samples for ILC tests, comparison participants and methodology

Below, in Sections 3.3.1 - 3.3.6, the measuring devices selected for ILC tests for experimental validation purposes are presented, along with details of comparison participants, conditions and acceptance criteria.

3.3.1 Gas meters

Table 12. Detailed data for ILC tests of gas meters in Task T3.2

ILC test sample	Participants	Gas medium used	Test conditions	ILC Acceptance Criterion
		TSO gas meters		
Itron Delta S3 G250 – rotary meter	ENAGAS CESAME GRTGAZ GS	NG	Errors at Q_{max}, $0,6Q_{max}$, $0,4Q_{max}$, $0,1\ Q_{max}$, $0,05Q_{max}$, Q_{min} (3 reps) Test flow range: 0,4 ÷ 400 m^3/h Test pressure: 16 bar, Test temp.: 15÷25 °C Silencers: Yes/No	En ≤ 1
Itron Fluxi G650 – turbine meter	ENAGAS CESAME GRTGAZ GS	NG	Errors at Q_{max}, $0,6Q_{max}$, $0,4Q_{max}$, $0,1\ Q_{max}$, $0,05Q_{max}$, Q_{min} (6 reps) Test flow range: 50 ÷ 1000 m^3/h Test pressure: 16 bar, Test temp.: 15÷25 °C Straightener: Yes/No	En ≤ 1
		DSO gas meters		
Sagemcom (EG) G6	INIG METAS	100% H_2	Errors at Q_{max}, $0,4Q_{max}$, $0,1\ Q_{max}$, Q_{min} (3 reps) Test flow range: 0,04 ÷ 6 m^3/h Test pressure: <10 kPag Test temp.: 15÷25 °C	En ≤ 1

Flonidan SciFlo G4	INIG GRTGAZ	25% H2NG	Errors at Q_{max}, $0{,}4Q_{max}$, $0{,}1\ Q_{max}$, Q_{min} (3 reps) Test flow range: 0,04 ÷ 6 m^3/h Test pressure: <10 kPag Test temp.: 15÷25 °C	En ≤ 1
Flonidan SciFlo G4	INIG GRTGAZ	NG	Errors at Q_{max}, $0{,}4Q_{max}$, $0{,}1\ Q_{max}$, Q_{min} (3 reps) Test flow range: 0,4 ÷ 65 m^3/h Test pressure: <10 kPag Test temp.: 15÷25 °C	En ≤ 1

3.3.2 Pressure transducers

Table 13. Detailed data for ILC tests of pressure transducers in Task T3.2

ILC test sample	Participants	Gas medium used	Test conditions	ILC Acceptance Criterion
Rosemount 3051	ENAGAS INIG (ILC option based on manufacturer's calibration results)	Inert (N_2)	Test pressure: P_{min}, P_2, P_3, P_4 P_{max}, and P_{max}, P_4, P_3, P_2, P_{min} Number of series: 1 Test temp.: 15÷25 °C Reading: one of, the current output/Hart output/display	En ≤ 1

3.3.3 Volume conversion devices - Calculation of compression factor

For the volume conversion devices, a comparison of Z results for several datasets obtained for the SGERG-88, AGA8-92DC, and GERG-2008 methods was assumed within the range of the applicability of the methods and also outside the range of the permissible hydrogen content.

Table 14. Detailed data for ILC tests of volume conversion devices in Task T3.2

ILC test method	Participants	Gas parameters / composition	ILC Acceptance Criterion
SGERG-88	ENAGAS INIG	TBD (min. 5 data sets)	The same *Z* value to 5 decimals
AGA8-92DC	ENAGAS INIG	TBD (min. 5 data sets)	
GERG-2008	ENAGAS INIG	TBD (min. 5 data sets)	

3.3.4 Trace water sensors

Tests of trace water sensors in the T3.3 test campaign will be carried out exclusively by the INRIM partner. It is not possible to perform ILCs in this case. Given that INRIM is the Italian National Metrology Institute, this confirms its competence and reliability of the research results.

3.3.5 Leak detectors

Table 15. Detailed data for ILC tests of Leak detectors in Task T3.2

ILC test sample	Participants	Gas medium used	Test conditions	ILC Acceptance Criterion
Altair 4 (MSA)	FBK/INIG	CH_4 and H_2, complement: clean air (zero gas)	Calibration curve and short-term stability tests in CH4 (H2 optional). Test conditions are reported in the leak detector testing protocol.	±5% result difference between FBK and INIG tests
SENSIT HXG-3P	FBK/INIG	CH_4 and H_2, complement: clean air (zero gas)	Calibration curve and short-term stability tests in CH4 (H2 optional). Test conditions are reported in the leak detector testing protocol.	±5% result difference between FBK and INIG tests
GAZOMAT INSPECTRA LASER/SENSIT LZ-30	FBK/INIG	CH_4 and H_2, complement: clean air (zero gas)	Calibration curve and short-term stability tests in CH4 (H2 optional). Test conditions are reported in the leak detector testing protocol.	±5% result difference between FBK and INIG tests

4. ILC principles

When carrying out ILCs for method and measurement validation purposes, small-scale comparisons will be carried out, following the guidelines outlined in document EA-4/21 INF:2018 [5] "Guidelines for the assessment of the appropriateness of small ILCs within the process of laboratory accreditation". A small comparison program should include the following arrangements:

- purpose and scope of interlaboratory comparison,
- quality standards,
- coordinating the program and participants,
- confidentiality,
- objects for comparison,
- measured quantity and test method,
- time frame,
- reporting of results,

- collusion and falsification of results,
- data analysis and results evaluation.

To evaluate the results of comparisons with the reference laboratory, the E_n index can be calculated in accordance with ISO/IEC 17043:2011 [6]:

$$E_{\mathrm{n}} = \frac{x - X}{\sqrt{{U_{\mathrm{Lab}}}^2 + {U_{\mathrm{ref}}}^2}}$$

where:

x- Laboratory result,
X- reference Laboratory result (with better CMC),
U_{Lab}- expanded uncertainty of determining the Laboratory result,
U_{ref}- expanded uncertainty of determining the result of the reference Laboratory.

In the case of ILC for gas meters, the method of determining indication errors and considerations regarding methods to compare results are presented in Annex 1 to D3.5.

5. CONCLUSION

After analyzing the research capabilities of individual partners in the project and selecting samples for testing, the method of conducting interlaboratory comparisons was demonstrated for each type of device covered by the project. In some cases, comparisons cannot be made due to the feasibility of conducting tests only in one laboratory. Below are the key takeaways for preparing for experimental validation:

- For gas meters used with TSO, it is possible to carry out ILC tests only with the use of natural gas in ENAGAS, CESAME, GRTGAZ, and GAZ SYSTEM. Calibration using hydrogen and H2NG mixture will only be performed by DNV.
- For gas meters used with DSO, ILC tests can be conducted as follows:
  - using 100% H_2 at METAS and INIG;
  - using natural gas and/or 25%H2/NG at INIG, GRTGAZ.
- For pressure transmitters, it is possible to carry out ILC tests at INIG and ENAGAS but also by comparing the manufacturer's calibration with the testing laboratory calibration.
- For volume converters, ILC tests will be carried out in the form of comparisons of the calculations of the compression factor Z at INIG and ENAGAS.
- Tests for gas analyzers will not be conducted.
- ILC tests are not possible for trace water sensors. The tests will be performed only by the National Metrology Institute INRIM.
- For leak detectors, it is possible to carry out ILC tests at INIG and FBK.

REFERENCES

[1] EN ISO/IEC 17025:2017 Testing and calibration laboratories

[2] EN ISO 12213:2013-3:2009 Natural gas - Calculation of compression factor - Part 3: Calculation using physical properties

[3] EN ISO 12213:2013-2:2006 Natural gas - Calculation of compression factor Part 2: Calculation using molar-composition analysis

[4] EN ISO 20765-2:2018 Natural gas - Calculation of thermodynamic properties - Part 2: Single-phase properties (gas, liquid, and dense fluid) for extended ranges of application

[5] EA-4/21 INF:2018 Guidelines for the assessment of the appropriateness of small interlaboratory comparisons within the process of laboratory accreditation

[6] ISO/IEC 17043:2023 Conformity assessment. General requirements for the competence of proficiency testing providers