



# Proficiency Testing Protocol for Inorganic gaseous pollutants measurement

13-16 April 2026

20-23 April 2026

27-30 April 2026

Joint Research Centre (ERLAP)  
Ispra (Italy)



## 1 INTRODUCTION

The comparison exercise (PT) evaluate proficiency in the analysis of inorganic gaseous pollutants covered by European Air Quality Directives [1] ( $\text{SO}_2$ ,  $\text{CO}$ ,  $\text{NO}$ ,  $\text{NO}_2$  and  $\text{O}_3$ ).

PT are organised with a view to harmonising air quality measurements across the EU and in order to assure the implementation of Air Quality Directives by the responsible bodies in EU Member States.

ERLAP (European Reference Laboratory for Air Pollution) has been organising PT for National Air Quality Reference Laboratories (NRLs) since the early '90s.

ERLAP is part of the Joint Research Centre site of Ispra (I) of the European Commission and has an implemented quality system accredited ISO 17043 [2] and compliant to ISO 17025 [3] for gaseous pollutants testing.

This document describes the proficiency testing scheme for the measurement of inorganic gases in purified ambient air, as performed by the European Reference Laboratory for Air Pollution (ERLAP) of the JRC, according to the requirements of the ISO/IEC 17043 [2] standard.



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### 3 SCOPE OF THE PROFICIENCY TESTING SCHEME

ISO/IEC 17043 [2] defines proficiency test as the evaluation of participant performance against pre-established criteria by means of PT.

Accreditation to ISO/IEC 17025 [3] requires laboratories to participate in a Proficiency Testing (PT) scheme, provided that a suitable scheme is available.

These PT are carried out in order to compare calibration standards and measurement capabilities, and to facilitate exchange of technical information amongst the national experts.

The interlaboratory comparison exercises will cover the goals of both organizations, focusing on harmonization and quality control of air pollution measurements performed by environmental institutes and National Reference Laboratories.

Additionally, these exercises provide valuable information on the robustness and performance of reference methods used to measure the considered pollutants. Evaluation of the precision of standardized measurement method laid down in ISO 5725-2 [6] could be implemented to evaluate the reproducibility of the method.

The purpose of proficiency testing (PT) is to make sure that measurements are accurate and comparable, and that they meet the data quality standards set out in the Air Quality Directive [1]. PT also helps national experts share technical information with each other. The same Directive [1] provides guidelines for organizing PT. It requires the establishment of community-wide quality assurance programs and sets the minimum frequency for National Reference Laboratories (NRLs) to participate in these programs.

By taking part in the exercise, participants can detect possible problems in their analytical chain and take remedial actions (if necessary), or else prove the reliability of their analyses.

### 4 PT PROVIDER

The European Reference Laboratory for Air Pollution (ERLAP) of the European Commission's Directorate-General for Joint Research Centre (DG JRC) assumes full legal responsibility for all activities related to the proficiency testing (PT) scheme. ERLAP operates independently and does not utilize any externally provided products or services for the inorganic gases PT scheme. ERLAP is authorized and impartial in conducting inter-laboratory comparisons, in accordance with ISO/IEC 17043 [2]. Additionally, ERLAP possesses the necessary expertise and experience to perform measurements of SO<sub>2</sub>, NO, NO<sub>2</sub>, NOx, O<sub>3</sub>, and CO in purified ambient air, compliant with ISO/IEC 17025 [3].

ERLAP conducts these PT activities at the Joint Research Centre – Directorate C, Unit C05, Building 100, Room 0102. The ERLAP team comprises the following members:

Annette Borowiak: PT Coordinator (Office Tel: +39 0332 789956)

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**4.1 Accreditation**

ERLAP Laboratory use ISO/IEC 17043 [2] to implement a quality system aimed at improving their ability to consistently produce valid exercises. It is also the basis for accreditation from an Accreditation Body. Since the standard is about competence, accreditation is simply formal recognition of a demonstration of that competence. A prerequisite for a laboratory to become accredited is to have a documented Quality Management System. The usual contents of the quality manual follow the outline of the ISO/IEC 17043 [2] and is also compliant to ISO/IEC 17025 [3] standards.

## 5 PARTICIPANTS

The National Reference Laboratories (NRLs), representing the EU Member States, are required to participate to a PT at least once every three years.

To provide a true evaluation and monitoring of performance the overall design of the PT is encouraging the participating laboratories to adopt an analytical testing procedure as they would apply in their routine analytical work.

In ERLAP, the supply bench can accommodate up to 10 laboratories, each equipped with 4 instruments, including the PT provider, for the inter-laboratory comparison of inorganic gases.

Keeping in mind that the assigned value is obtained through ERLAP results and due to the fact that ERLAP has an institutional obligation established by the Air Quality Directive, the organization of this type of proficiency test does not require a minimum number of participants. However, to fully cover the purposes of these exercises, in the case of few participants, the PT provider will seek to invite other laboratories or postpone the test, always in agreement with the registered participants.

All participation requests are accepted according to the following criteria:

- Priority 1 to laboratories that have not participated in the last 3 years
- Priority 2 to laboratories with unsatisfactory results during the previous ILC
- Priority 3 NRL laboratories with the condition first come first served
- Priority 4 other (external) laboratories if places available.



## 6 STEPS OF THE INORGANIC GASES PT

### 6.1 Timetable:

The typical timetable for this type of PT is detailed in paragraph 7.

### 6.2 PT steps:

Access is provided through the Proficiency Testing Data Acquisition Platform (PT-DAP) of JRC-ERLAP: <https://erlap-intercomparison.jrc.ec.europa.eu>

The opening and deadline for the registration is clearly reported.

#### a. Protocol consultation

- Review and understand of the PT protocol before proceeding with registration is asked to the possible participants.

#### b. Registration to the platform

- New users: register to PT-DAP (one-time, two-step procedure)
- Existing users: Log in using EU Login credentials

#### c. Expression of interest

- Expression of interest in participating to the PT Inorganic gases exercise
- Receive notification of successful application; contact PT coordinator if not received
- Receive notification of acceptance or rejection (within a month from application closure) applicants may also *delete* their application.

#### d. Entrance to JRC site

Due to specifics of the JRC Ispra site, all participants must register their presence via the JRC REM (Register for Events Module) event registration system: <https://web.jrc.ec.europa.eu/remjrc/screen/meetings>.

Each laboratory participant (max. 2 people per laboratory) has to register on the website. Entrance permits will be provided based on the identity information submitted via the website.

The entrance permit allows visitors to stay in the JRC from 8.00 am till 19.00 pm, but arrival and departure needs to be agreed with ERLAP before.

All non EU participants have to send their passports to the ERLAP Committee no later than 40 days before the PT.

#### e. Participants transport

Transports to/from airport and to/from hotel/JRC will be organized only for those who have requested them in the system before the deadline. Transport is organised only if it is accepted the accommodation suggested in the REM web application. In case the participants prefer a different accommodation it can be organized personally but it is kindly requested to fill in the online registration form accordingly.

#### f. Equipment shipment



All the equipment used during the PT has to go through the custom control of the JRC.

Equipment must be labelled with the address of the proficiency provider (see Paragraph 4). When the equipment arrives at the custom office in Ispra from an EU-member state, a delivery note (list of goods) and a pro-forma invoice (value of goods) must be provided with the equipment and a note "MATERIAL FOR TEST" must be included in all documents. Equipment arriving from outside the EU need a Carnet ATA to pass the custom.

When the equipment is shipped the reference persons of the PT in Ispra (Claudia Tarricone and Maurizio Barbiere) shall be informed. The last day before leaving the equipment must be packed with the appropriate labels indicating destination and reference name, further the delivery from our customs office has to be booked.

#### **g. Own equipment transport**

Upon arrival the participant has to go through the Customs entrance of the JRC (Opening hours Mon-Thu: 08:30 - 11:45 and 13:30 - 17:00, Fri: 08:30 - 11:45 and 13:30 - 15:45), which is about 500 m to the West of the main entrance.

For both (Shipment and Own transport) the list of goods and the pro-forma invoice is requested.

Example:

Material for test

- 1 NOx analyser
- 1 SO2 analyser
- 1 CO analyser
- 3 gas cylinders
- 1 Notebook

Estimated value: 50.000 Euro

From the customs the participants can drive directly to the ERLAP laboratory in building 100. The papers received from custom (FATTURA PROFORMA) need to be kept for taking the equipment out of the JRC on the last day.

#### **h. Installation and testing process**

On the first day of the proficiency testing (PT) exercise, the ERLAP Team will be available to address any questions or concerns participants may have regarding the various PT phases. Additionally, all participants are strongly encouraged to familiarize themselves with the safety and security protocols in effect at the JRC Ispra site.

For this PT exercise, gas mixtures will be prepared to include sulfur dioxide (SO<sub>2</sub>), carbon monoxide (CO), ozone (O<sub>3</sub>), nitrogen oxide (NO), and nitrogen dioxide (NO<sub>2</sub>). Participating laboratories are required to conduct measurements continuously using their own automatic analyzers, which must be calibrated with their respective reference standards and according to their own procedures. Manual



measurement methods are not permissible for this exercise in Ispra. All measurement results should be recorded using each participant's data acquisition systems.

#### **i. Data Submission**

The reporting of results within the requested time scale and in the specified units is part of the performance assessment.

Participants are requested to submit their results and uncertainties via the secure pages on our PT-DAP web site (PT-DAP: <https://erlap-intercomparison.jrc.ec.europa.eu>).

Each participant has confidentially a unique User ID and Password to access these pages.

The participants are asked to report three half-hour-mean measurements (steps) for each concentration level (run). Zero concentration levels are generated for one hour and one half-hour-mean measurement has to be reported.

The uncertainties are written with the same number of significant figures and in the same units as the result. The estimate should also state the level of confidence associated with the coverage factor (k).

Submitted data are saved in a central database and subsequently used for calculations and comparisons. Uploaded data are then used for data evaluation and writing the final report about the inter-laboratory comparison.

After submitting their data, participants will receive a notification confirming successful submission. They have the flexibility to manage their data and also withdraw their participation from the exercise at any time before the data submission deadline.

#### **j. Performance evaluation**

Concluded data submission the PT provider verify the entire dataset to identify any obvious blunders. Involved laboratory will be notified if any blunders will be found between their results and they have the opportunity to modify their data. Afterward the statistical evaluation will be performed as described in paragraph 13.

#### **k. Reporting**

Once the performance assessment will be over, the report will be produced. As soon as all the internal revisions, approval and authorization will be completed the participants will have the opportunity to comment on formal aspects of the report. The final report will be officially published on the JRC Publications Repository, and the URL to access the record will be provided when it becomes available.

Participants are encouraged to offer feedback on the service provided, ideally upon completion of the PT exercise. Additionally, if there are any complaints or appeals, participants can use the form provided in Annex 1 of this document to send them to the PT provider.

## 7 TIMETABLE

The proficiency testing (PT) for inorganic gases is typically organized over two consecutive years, followed by a break in the third year. Below is a summary of the timetable for this PT exercise:

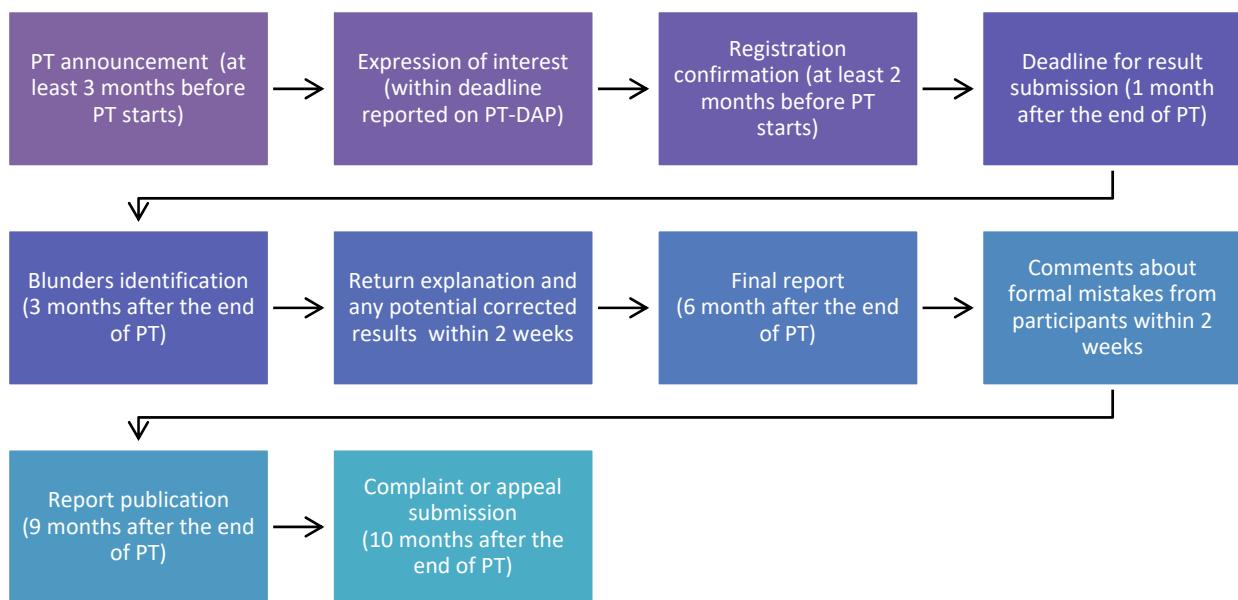


Figure 1: PT timetable for inorganic gases

## 8 WEB APPLICATION

The JRC-ERLAP PT-DAP platform for proficiency testing scheme and data acquisition is a web application that allows stakeholders/users to access PT schemes organised by ERLAP for variables of air quality relevance.

This web application adopts the European Commission's identity and access management service, EU Login.

### Authentication: EU login account

- open <https://erlap-intercomparison.jrc.ec.europa.eu> Proficiency Testing Data Acquisition Platform (PT-DAP) and click "SUBSCRIBE" (Figure 2);
- if you are a new user and do not have an EU Login account yet, create an account;
- fill in all mandatory fields (i.e. First Name, Last Name, e-mail, confirm e-mail and e-mail language) and acknowledge the privacy statement, then click on "Create an account";
- you can now sign in using your e-mail and password.

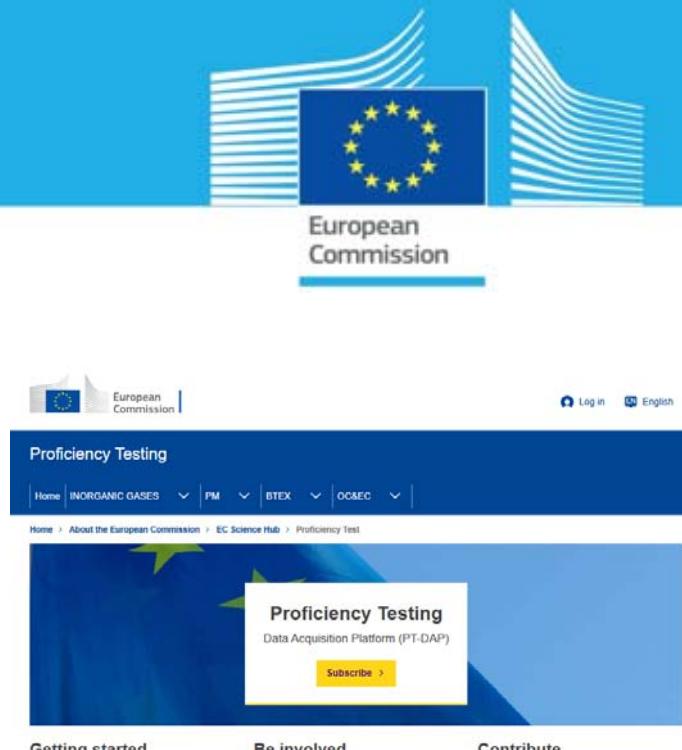


Figure 2: PT DAP web application

#### Subscription to the JRC-ERLAP PT-DAP:

- fill in the subscription form, accept the privacy statement, enter the verification code and send;
- you receive an e-mail; click on the “validation link” to confirm your e-mail and access the JRC-ERLAP PT-DAP;

#### Application-Registration to the Inorganic gases PT exercise:

- select the Inorganic gases scheme section and apply for the available exercise;
- duly fill in the application form (details on applicant, shipping, measurement instrument and method);
- accept all procedures, terms and conditions as described in the PT plan;
- you receive a notification of successful application; if not, contact PT coordinator (Par.2);
- you receive a notification either accepting or rejecting your application;
- in this section you may also delete your application.

#### Submission of data and metadata:

access the PT-DAP inorganic gases section, select the relevant PT-inorganic gases exercise:

- click “SUBMIT” to submit your data with\_uncertainties and metadata:



- from the drop down list, "SELECT" one measurand (i.e., NO, NO<sub>2</sub>, O<sub>3</sub>, SO<sub>2</sub>, CO) and the relative measurement instrument unique identifier;
- upload the CSV file with the data with unit format  $\mu\text{g}/\text{m}^3$  for NO, NO<sub>2</sub>, SO<sub>2</sub> and O<sub>3</sub> while in mg/m<sup>3</sup> for CO (conversion factor as reported in EN standard)
- the CSV file is automatically verified for format and completeness issues; in case of a well formed file, data are shown in a table (cells highlighted in yellow indicate missing entries, left intentionally or mistakenly empty); otherwise, error details are provided;
- repeat it for each pollutant.

You receive a notification of successful data submission, if not, contact PT coordinator (Par.2).

You may manage your data any time before the "data submission" closure, accessing the PT Inorganic gases exercise.

- or click "WITHDRAW" to withdraw your participation from the PT exercise.

To ensure consistency in the results, all participants are asked to adhere to the rounding rules specified in Table 1.

Value x	Number of decimals	Example: before rounding	Example: after rounding
$x \geq 10$	integer	17.83	18
$1 \leq x < 10$	1 decimal	2.345	2.3
$0.1 \leq x < 1$	2 decimals	0.865	0.87
$0.01 \leq x < 0.1$	3 decimals	0.0419	0.042

Table 1 Rounding rules for pollutants without an environmental objective.

### Feedback

- At the completion of the PT exercise, each participant should leave a feedback on the provided service

The web application and the database are hosted and maintained at the JRC-ERLAP in Ispra (IT). The use of the web application and database is regulated by European Commission Legal Notice and JRC Privacy Statement.

## 9 CONFIDENTIALITY

The results of the Proficiency Testing (PT) are published in accordance with the agreements outlined in the document AQUILA-N37 [4], which has been approved by all National Reference Laboratories (NRLs).

To ensure the confidentiality of laboratory information, ERLAP implements the following measures:



- Any administrative information supplied by a laboratory remains confidential and will not be shared with third parties.
- Access to ERLAP facilities is strictly limited to members of Unit C5 and authorized personnel, such as cleaning, maintenance, safety, and security staff.
- Confidential passwords to access the web application for data submission are issued once registration for the PT is complete. These passwords grant access to the web interface and the online questionnaire and remain valid until the PT is concluded. Laboratories have the option to change their passwords online at any time.
- For their first participation in a PT organized by ERLAP, participants are required to sign the LAB-REC-2000 form, which is a confidentiality involvement form.

By expressing interest in participating, each applicant agrees to these privacy and confidentiality policies. To ensure permanent and public access to the PT results, a digital object identifier (DOI) is associated with the report.

## **10 COLLUSION AND DATA FALSIFICATION**

While proficiency testing (PT) is designed to help participants improve their performance, there is a risk that some may attempt to misleadingly present their analytical capabilities. Collusion between participants and the falsification of results are contrary to professional ethics. Such behavior undermines the benefits of PT for both the participating laboratories and the organizer, defeating the purpose of genuine participation.

ERLAP conducts its PT program with the assumption that participants perform analyses and report results with scientific rigor. However, ERLAP is committed to preventing any instances of collusion or result falsification. To ensure integrity, an ERLAP representative will be present in the laboratory whenever a participant is on-site.

Through this protocol, every participant is made aware of the consequences of detected collusion or falsification. Collusion and falsification are unethical and constitute scientific fraud. If a clear situation of collusion or falsification is identified, the involved laboratories will be contacted. Upon confirmation of the evidence, these laboratories will be excluded from the PT data evaluation.

## **11 FEES**

Participation in this intercomparison circuit does not involve any registration fee. Transportation from/to airport and hotel is provided by the PT provider only under the conditions outlined in paragraph 6. Accommodation and travel costs for personnel, as well as the transportation of equipment, are managed and paid by the participant. No certificate will be issued for the participation to this PT.

## **12 PT ITEM**

The test mixtures will be prepared by diluting gases from cylinders containing high concentrations of NO, SO<sub>2</sub>, or CO using thermal mass flow controllers. Ozone (O<sub>3</sub>) will be generated using an ozone generator, and nitrogen dioxide (NO<sub>2</sub>) will be



produced using the gas phase titration method under conditions of NO excess or through dynamic dilution of gas cylinder.

The test gases will be prepared with a particular emphasis on low concentration levels, extending up to the limit or threshold values specified. The tested concentration levels will be below 10  $\mu\text{mol/mol}$  for CO, 720 nmol/mol for NO, and 200 nmol/mol for  $\text{NO}_2$ ,  $\text{O}_3$ , and  $\text{SO}_2$ . These test gases will be dry and free of particulate matter.

The standard methods to be employed are as follows: EN 14212 for sulphur dioxide ( $\text{SO}_2$ ), EN 14211 for nitrogen oxides (NO,  $\text{NO}_2$ ), EN 14625 for ozone ( $\text{O}_3$ ), and EN 14626 for carbon monoxide (CO).

Participating laboratories are required to bring their own analysers and traveling standards, including zero air, for calibrations or checks during the exercise. If a participant encounters any issues meeting these requirements or faces technical, logistical, or administrative problems, they must contact ERLAP promptly.

Participants are required to report three half-hour mean measurements for each concentration level (run). For zero concentration levels, which will be generated for one hour, participants need to report one half-hour mean measurement.

In table 2 is shown the PT scheduling.

day	Start time	Step	Component	NO ( $\mu\text{g}/\text{m}^3$ )	NO <sub>2</sub> ( $\mu\text{g}/\text{m}^3$ )	O <sub>3</sub> ( $\mu\text{g}/\text{m}^3$ )	CO ( $\text{mg}/\text{m}^3$ )	SO <sub>2</sub> ( $\mu\text{g}/\text{m}^3$ )
I	9:00		/	INSTALLATION				
II	8:00		/	CALIBRATION				
II	11:00	0	NO-NO <sub>2</sub> -O <sub>3</sub>	ZERO AIR				
II	12:00	1	NO-NO <sub>2</sub>	X	X			
II	14:00	2	NO-NO <sub>2</sub>	X	X			
II	16:00	1	O <sub>3</sub>			X		
II	18:00	3	NO-NO <sub>2</sub>	X	X			
II	20:00	4	NO-NO <sub>2</sub>	X	X			
II	22:00	2	O <sub>3</sub>			X		
III	0:00	5	NO-NO <sub>2</sub>	X	X			
III	2:00	6	NO-NO <sub>2</sub>	X	X			
III	4:00	3	O <sub>3</sub>			X		
III	6:00	7	NO-NO <sub>2</sub>	X	X			X
III	8:00	8	NO-NO <sub>2</sub>	X	X			X
III	10:00	4	O <sub>3</sub>			X		
III	12:00	9	NO-NO <sub>2</sub>	X	X			X
III	14:00	10	NO-NO <sub>2</sub>	X	X			X
III	16:00	5	O <sub>3</sub>			X		
III	< 18:00			CALIBRATION				
III	20:00	0	CO-SO <sub>2</sub>				ZERO AIR	
III	21:00	1	CO-SO <sub>2</sub>				X	X
III	23:00	2	CO-SO <sub>2</sub>				X	X
IV	01:00	3	CO-SO <sub>2</sub>				X	X
IV	03:00	4	CO-SO <sub>2</sub>				X	X
IV	05:00	5	CO-SO <sub>2</sub>				X	X
IV	07:00			ZERO AIR not to be reported				
IV	08:00			END				

Table 2 : gas mixture generation plan

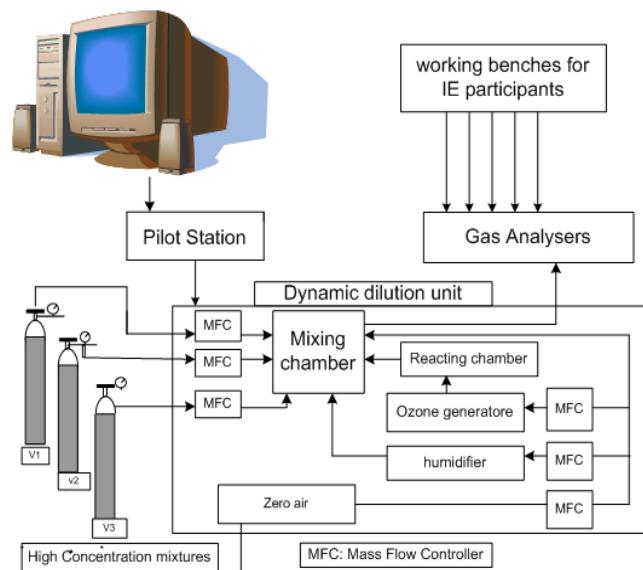


Figure 3: Test gas generator facility in ERLAP

In Figure 4 the facility available during this type of PT is illustrated for each participant.

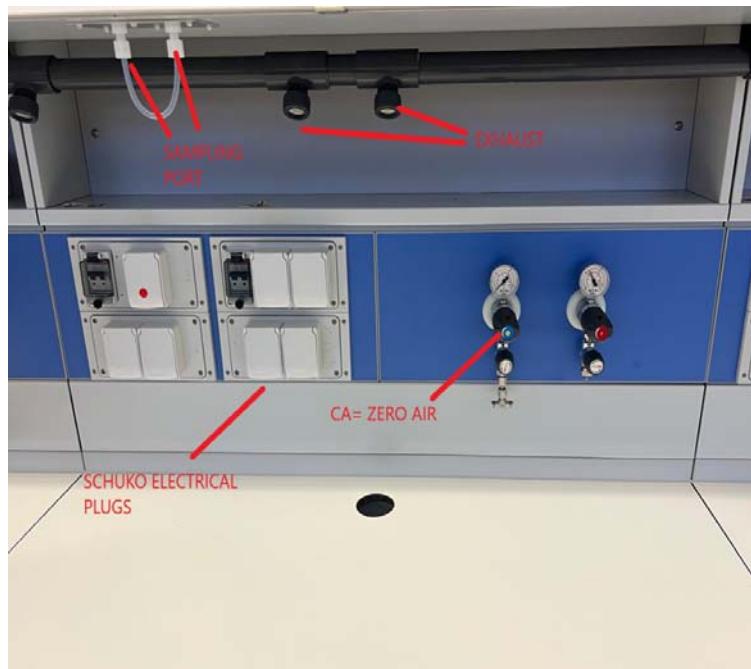


Figure 4: ERLAP laboratory facility for the PT

Every participant will have a working area where the instrumentation can be installed (Fig.4). Electrical plugs and gas supply are available as well. As shown in figure 4 there is a sampling line (white tube) to connect to the instrument for measurement during the PT. The dark gray pipe is to connect the exhaust of the instrument. It is recommended to keep the sampling line closed when it is not used.

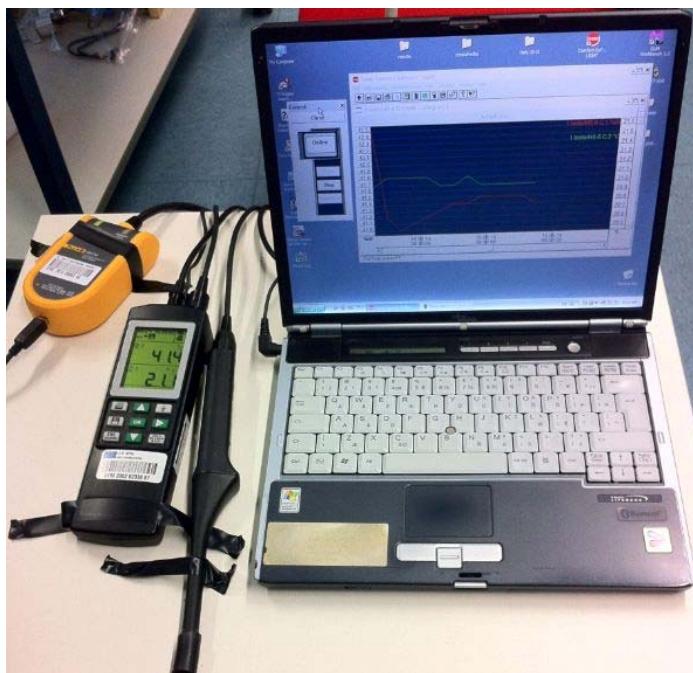


Figure 5: temperature and time during PT

Time, temperature and line-voltage (Figure 5) will be registered during the PT with proper devices. All data recorded are available on request to the participants of the PT.

At the end of the exercise every laboratory has the responsibility to run all QA/QC foreseen by their procedure.

## 12.1 Homogeneity

In ERLAP, the homogeneity of test gas mixtures across the entire working bench is assessed by comparing the measurements from all ports used by participants during the PT with the last port (Port 20) of the distribution line. During this evaluation, a consistent concentration of ozone is employed. Measurements are conducted using two different ozone analyzers (Thermo Fisher Scientific 49i), both calibrated against a Standard Reference Photometer (SRP) primary standard, operating in parallel.

The test is considered acceptable when the difference of the two measurements is below 0.5%. This test is part of the equipment checks run before a PT organised by ERLAP in Ispra.



The standard uncertainties of the reference value ( $u_x$ ) are calculated with equation 1;  $u_x'$  is the analytical uncertainty of the reference value,  $X$  is the reference value and  $u_{\text{homogeneity}}$  is the contribution of the homogeneity test result to the uncertainty budget of the reference value.

$$u_x^2 = u_{X'}^2 + (X u_{\text{homogeneity}}) \quad \text{equation 1}$$

The homogeneity is considered as a component of the uncertainty associated with the assigned value of the PT item and is therefore taken into account in the evaluation of participants' performance.



## 13 PERFORMANCE ASSESSMENT

The primary goal of these proficiency tests (PT) is to verify the accuracy of the results submitted by participating laboratories. This is typically achieved by comparing the participants' results to an estimated assigned value.

The z score or z' score method as defined in ISO 13528 [5] will be used to demonstrate the capacity of NRLs perform in accordance with the EN standard. When there is concern about the uncertainty of the assigned value ( $u(x_{pt})$ ) for example.  $u(x_{pt}) > 0.3\sigma_{pt}$ , then z' score is applied. The z and z' score will be calculated as described in ISO:13528 (§ 9.4, 9.5) [5] through the following equation 2 and 3.

$$z = \frac{x_i - X}{\sqrt{\sigma_{pt}^2}} \quad \text{equation 2}$$

$$z' = \frac{x_i - X}{\sqrt{u^2 + \sigma_{pt}^2}} \quad \text{equation 3}$$

$z$  = z-score

$z'$  = z'-score

$x_i$  = participant average values

$X$  = reference value

$u$  = uncertainty of the reference value

$\sigma_{pt}$  = Standard deviation for proficiency assessment

$a$  = slope see table 3

$b$  = intercept see table 3

The following criteria for assessing performance through z or z' score results are:

- Absolute values  $\leq 2$  are considered satisfactory.
- Absolute values between 2 and 3 are considered questionable.
- Absolute values  $\geq 3$  are considered unsatisfactory.

Scores falling in the range of 2 to 3 indicate that the cause of the event should be investigated and corrected by the participating NRL.

Another indicative performance indicator, called the En-score (equation 4), can be reported and it assesses if the difference between a participant's measured values and the assigned stays within limits that are individually calculated for each participant. These limits are derived from the uncertainty of the participant's measurement result and the uncertainty of the assigned or reference value.

$$En = \frac{x_i - X}{\sqrt{u_x^2 + u_X^2}} \quad \text{equation 4}$$

$En$  = En-score

$x_i$  = participant average values

$X$  = reference value



$U_x$ = expanded uncertainty of the participants

$U_x$ = expanded uncertainty of the reference value

The  $En$  evaluation allows to determine if the differences between participating NRLs result and the assigned value fall within the assigned uncertainty and NRL uncertainty, as long as  $|En| < 1$ . Scores of  $|En| \geq 1$  suggest the need to revisit the uncertainty estimates or can indicate an incorrect measurement result.

Other parameters may be used in addition to evaluate the laboratory's performances (Difference (D), Difference % (D%), ...)

### 13.1 Data review

Before applying any statistical procedures during the initial evaluation of the Proficiency Testing (PT) exercise data, the PT provider conducts a thorough review of the entire dataset to identify any obvious errors or blunders. These may include reporting results in incorrect units, mixing results from different PT items, or transcription/typing errors. If any blunders are detected, the PT provider contacts the involved participants to request clarification and, if necessary, corrections.

### 13.2 Assigned value

To calculate z-scores, an assigned value and its standard deviation is established for each proficiency testing (PT) item.

Typically, for this PT the measurements conducted by ERLAP are used as the assigned values ( $X$ ). These assigned values for the tested concentration levels are derived from a calibration process using certified reference material (CRM) gases, ensuring strict traceability to international standards as outlined in ISO 13528 [5]. For NO, CO, and SO<sub>2</sub>, the gas mixtures used in the calibration experiments are produced through dynamic dilution of CRM gases. This process utilizes mass flow controllers and a molbloc/molbox system to measure the flow rates accurately. The efficiency of the NO<sub>2</sub> converter is evaluated using a Gas Phase Titration (GPT) method.

The uncertainty calculation for these gases includes contributions from the CRM, zero gas (which is also based on CRM), flow rates, drift, converter efficiency, repeatability, and homogeneity.

For ozone (O<sub>3</sub>), the calibration is based on the NIST Photometer, and the uncertainty calculation encompasses the NIST Photometer, drift, repeatability, and homogeneity.

### 13.3 Standard deviation for proficiency assessment

Among the available methods for determining the standard deviation for proficiency assessment ( $\sigma_{pt}$ ), the approach of calculating  $\sigma_{pt}$  from data obtained in multiple rounds of a proficiency testing scheme was chosen. The Standard deviation for proficiency assessment is defined by:

$$\sigma_{pt} = (a \cdot x_{pt} + b) \quad \text{Equation 5}$$



$\sigma_{pt}$  = Standard deviation for proficiency assessment

$x_{pt}$  = assigned value

a = slope (see table 3)

b = intercept (see table 3)

In the NO/NO<sub>2</sub>, SO<sub>2</sub>, O<sub>3</sub> CO EN Standards [7, 8, 9, 10] the uncertainties for calibration gases used in ongoing quality control are described. In fact, the 'standard deviation for proficiency assessment' ( $\sigma_{pt}$ ) [5] is derived in a fitness-for-purpose manner from requirements given in the EN standards, where it is stated that the maximum permitted expanded uncertainty for calibration gases at the calibration point (75% of certification range) is 5%.

Considering the measurement of NO<sub>2</sub> is obtained from the difference of concentration between NOx and NO channels, the  $\sigma_{pt}$  is calculated, as linear interpolation, considering both the contributions of the two channels at span concentration. Over the whole measurement range,  $\sigma_{pt}$  is calculated by linear interpolation between the value at the calibration point and zero. For zero, instead of detection limit criteria, the specifications for purity of zero gas used in type approval as defined in EN Standards, are taken.

	$\sigma_{pt}$ (zero) nmol/mol	$\sigma_{pt}$ (cal point) nmol/mol	$\sigma_{pt}$ nmol/mol = a · [Assigned value] nmol/mol + b	
			a	b
				nmol/mol
SO <sub>2</sub>	1	7.1	0.022	1
CO	100	1613	0.024	100
O <sub>3</sub>	1	4.7	0.020	1
NO	1	18.0	0.024	1
NO <sub>2</sub>	1.4	6.9	0.028	1.4

Table 3 Standard deviation for proficiency assessment  $\sigma_{pt}$ .

### 13.4 Evaluation of performance

During this PT, National Reference Laboratories (NRLs) are required to demonstrate an overall satisfactory performance in the z or z' score evaluations. This means that no more than 20% of their results can be unsatisfactory (z score or z' score  $\leq 2$ ), and fewer than 40% can be questionable for each parameter. If an NRL has more than one unsatisfactory result or more than two questionable results out of five gas concentrations, their overall evaluation will be deemed unsatisfactory.

In the event of unsatisfactory results, the national laboratory must show that remediation measures have been successfully implemented during their next participation in the intercomparison. They are also required to submit a report to the Joint Research Centre outlining these measures.

## 14 METHOD REPRODUCIBILITY

For informative purposes only, the data from this PT exercise are also used to determine the reproducibility of the EN standard measurement method [7,8,9,10] according to the ISO 5725-2.



## 15 PT REPORT

The report detailing the results of the statistical evaluation will be distributed to all participants. This report is considered valid only in its entirety and not in isolated parts. Individual participant performance reports will not be provided.

Before finalizing the report, laboratories that show blunders will be asked to investigate the causes of these discrepancies. Laboratories are permitted to correct their results if significant errors are identified. Once the data are considered definitive, the performance indicators will be calculated.

Upon completion of the statistical data evaluation, including the approval and authorization process for publication, a final version 0 of the report will be sent to the participants as outlined in paragraph 6. At this stage, submitted data cannot be altered, and only minor formal corrections will be allowed. Following the release of the final version 0, a new version, if necessary, of the report will be published and distributed. This report will be made public, and the results of participating laboratories will no longer be confidential.

All records and reports from any proficiency testing (PT) organized by ERLAP will be retained for at least four years.



## **16 RELEVANT REFERENCES:**

- 1) Directive (EU) 2024/2881/EC of the European Parliament and of the Council of 23 October 2024 on ambient air quality and cleaner air for Europe.
- 2) ISO 17043:2023, Conformity assessment - General requirements for proficiency testing
- 3) ISO/IEC 17025:2017, General requirements for the competence of testing and calibration laboratories.
- 4) AQUILA protocol N 37: Organization of Inter-Comparison Exercises for gaseous air pollution for EU National Air Quality Reference Laboratories and Laboratories of the WHO EURO Region
- 5) ISO 13528:2022, Statistical Methods for use in Proficiency Testing by Inter-Laboratory Comparisons
- 6) ISO 5725-2:2019, Accuracy (trueness and precision) of measurement methods and results – Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method
- 7) EN 14211:2024 Ambient air — Standard method for the measurement of the concentration of nitrogen dioxide and nitrogen monoxide by chemiluminescence
- 8) EN 14212:2024 Ambient air — Standard method for the measurement of the concentration of sulfur dioxide by ultraviolet fluorescence
- 9) EN 14625:2024 Ambient air — Standard method for the measurement of the concentration of ozone by ultraviolet photometry
- 10) EN 14626:2024 Ambient air — Standard method for the measurement of the concentration of carbon monoxide by non-dispersive infrared spectroscopy



## 17 ANNEX 1: APPEAL/COMPLAINT FORM

*This form should be addressed to the PT provider team.*

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Laboratory and participant name:

PT scheme: Inorganic gases YYYY-n

If you would like to file an appeal<sup>§</sup>/complaint, please use the box below:

Description of Appeal/Complaint:

Date:

Name and Function:

Signature:

<sup>§</sup>Errors made by the participants in data entry cannot be corrected after the report is issued and these errors are not grounds for appeal.