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Evaluation of the Laboratory Comparison Exercise for NO, NO_2, SO_2, CO and O_3 4th–9th of October 2015, Langen (D)

EC Harmonization Program for Air Quality Measurements

Maurizio Barbiere, Friedrich Lagler, Annette Borowiak,
Volker Stummer, Hans-Guido Mücke, - 2015

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Contact information
European Commission
Institute for Environment and Sustainability
Maurizio Barbiere
Address: Joint Research Centre, Via Enrico Fermi 2749, TP 120, 21027 Ispra (VA), Italy
E-mail: maurizio.barbiere@jrc.ec.europa.eu
Tel.: +39 0332 783057

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In collaboration with:
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	NAME	VERSION	DATE
AUTHOR	M. BARBIERE	DRAFT 1	11/01/2016
AUTHOR	A. BOROWIAK	DRAFT 1.1	10/03/2016
AUTHOR	F. LAGLER	DRAFT 1.2	10/03/2016
AUTHOR	V. STUMMER	DRAFT 1.3	11/03/2016
AUTHOR	H-G. MÜCKE	DRAFT 1.4	06/04/2016
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1. Abstract

From the 4th to the 9th of October 2015 seven Laboratories of the World Health Organization (WHO) European-Region met for another joint JRC-ERLAP/WHO inter-laboratory comparison exercise (IE). They met at the National Air Quality Reference laboratory at the German Federal Environment Agency in Langen, Germany, to evaluate their proficiency in the analysis of inorganic gaseous pollutants (NO, NO2, SO2, CO and O3) covered by the European Air Quality Directive 2008/50 EC and recent revision 2015/1480/EC.
The proficiency evaluation, where each participant's bias was compared to two criteria, provides information on the current situation and capabilities to the European Commission and can be used by participants in their quality control system.
On the basis of criteria imposed by the European Commission, 73.2% of the results reported by the laboratories were good both in terms of measured values and reported uncertainties. Another 23.9% of the results had good measured values, but the reported uncertainties were too high and for 0.7% of the values the uncertainty was underestimated. 1.4% of the values were questionable and 0.7% were unsatisfactory.
Comparability of results among participants (reproducibility) at the highest concentration level, excluding outliers, is acceptable for CO and SO2 measurements while NO2, NO and O3 one showed less satisfactory results.

2. Executive Summary

From the 4th to the 9th of October 2015 seven Laboratories of the World Health Organization (WHO) European-Region met for another joint JRC-ERLAP/WHO inter-laboratory comparison exercise (IE).
The IE took place at the premises of UBA (D), the National Air Quality Reference laboratory of the German Federal Environment Agency in Langen, Germany, to evaluate their proficiency in the analysis of inorganic gaseous pollutants (NO, NO_2, SO_2, CO and O_3) covered by the European Air Quality Directive 2008/50/EC.

Most of the laboratories participating in the IE used automated instruments while one laboratory performed analysis using manual methods.

The proficiency evaluation, where each participant's bias was compared to two AQUILA based criteria, provides information on compliance with Data Quality Objectives and measurement capabilities of the National Air Quality Laboratories to the European Commission (AQUILA) and can be used by participants in the implementation of their laboratory's quality system.
In terms of the criteria (σ_p) imposed by the European Directive (that are not mandatory for WHO laboratories which do not belong to the EU), 73.2% of the results reported by WHO/AQUILA laboratories were considered satisfactory both in terms of measured values and evaluated uncertainties. Among the remaining results the majority presented satisfactory measured values but the evaluated uncertainties were either too high (23.9%) or too small (0.7%). Two reported values (1.4% of all) were questionable for the z-score and "not OK" for the En-number and one value was unsatisfactory (0.7%).

The comparability of results among all participants at the highest generated concentration levels, excluding outliers, was acceptable for CO and SO_2 measurements while O_3, NO and NO_2 measurements showed less satisfactory results.

Generally this proficiency evaluation confirmed the good performance of the involved laboratories with a high percentage of valid measurement and uncertainties.
The evaluation of reproducibility in comparison with previous IEs in Langen is confirming for SO2 a good performance and the results of CO are showing an improvement.
Some analytical difficulties for NO2 measurements continue and a performance decrease for NO and O_3 is noticed.

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Abbreviations

AQUILA	Network of National Reference Laboratories for Air Quality
CO	Carbon monoxide
DQO	Data Quality Objective
ERLAP	European Reference Laboratory for Air Pollution
EC	European Commission
GPT	Gas Phase Titration
IE	Inter-laboratory Comparison Exercise
IES	Institute for Environment and Sustainability
ISO	International Organization for Standardization
JRC	Joint Research Centre
NO	Nitrogen monoxide
NO_2	Nitrogen dioxide
NO_X	The oxides of nitrogen, the sum of NO and NO_2
NRL	National Reference Laboratory
O_3	Ozone
SO_2	Sulphur dioxide
WHO-CC	World Health Organization Collaborating Centre for Air Quality Management and Air Pollution Control, Berlin

Mathematical Symbols

symbol	*explanation*
α	converter efficiency (EN 14211)
E_n	E_n – number statistic (ISO 13528)
r	repeatability limit (ISO 5725)
R	reproducibility limit (ISO 5725)
σ_p	standard deviation for proficiency assessment (ISO 13528)
x*	robust average (Annex C ISO 13528)
s*	robust standard deviation (Annex C ISO 13528)
s_r	repeatability standard deviation (ISO 5725)
s_R	reproducibility standard deviation (ISO 5725)
$U_{X'}$	expanded uncertainty of the assigned/reference value (ISO 13528)
U_{xi}	expanded uncertainty of the participant's value
$u_{X'}$	standard uncertainty of the assigned/reference value (ISO 13528)
X	assigned/reference value (ISO 13528)
x_i	average of three values reported by the participant *i* (for particular parameter and concentration level) (ISO 5725)
$x_{i,j}$	j-the reported value of participant i (for particular parameter and concentration level) (ISO 5725)
z'	z'-score statistic (ISO 13528)

3. Introduction

The Directive 2008/50/EC [1] on ambient air quality and cleaner air for Europe sets a framework for a harmonized air quality assessment in Europe. Recently some annexes of the Directive were revised to include technical clarifications and updates on reference methods in the Commission Directive 2015/1480 [42].
One important objective of the Directive is that the ambient air quality shall be assessed on the basis of common methods and criteria. It deals with the air pollutants sulphur dioxide (SO_2), nitrogen dioxide (NO_2) and monoxide (NO), particulate matter, lead, benzene, carbon monoxide (CO) and ozone (O_3). Among others it specifies the reference methods for air pollution measurements and Data Quality Objectives (DQOs) for the accuracy of measurements.
The European Commission (EC) has supported the development and publication of reference measurement methods for CO [2], SO_2 [3], NO-NO_2 [4] and O_3 [5] as European standards. Appropriate calibration methods [6], [7] and [8] have been standardized by the International Organization for Standardization (ISO).

As foreseen in the Air Quality Directive, the European Reference Laboratory for Air Pollution (ERLAP) of the Institute for Environment and Sustainability (IES) at the Joint Research Centre (JRC) organizes inter-laboratory comparison exercises (IE) to assess and improve the status of comparability of measurements of National Reference Laboratories (NRL) of the Member States of the European Union [34], [35], [36], [37], [38], [39], [40], [41].
The World Health Organization Collaborating Centre for Air Quality Management and Air Pollution Control, Berlin (WHO CC) is carrying out similar activities since 1994 [9] [10], [31], [33], [36] and [39] but with a view to obtaining harmonized air quality data for health related studies. Their program integrates within the WHO EURO region, which includes public health institutes and other national institutes - especially from the Central Eastern Europe, Caucasus and countries from Central Asia.

Starting in 2004, it has been decided to bring together the efforts of both the JRC-ERLAP and WHO CC and to coordinate activities as far as possible, with a view to optimize resources and have better international harmonization.

The following report deals with the IE that took place from 4th to the 9th of October 2015 at the National Reference laboratory for Air Pollution, German Federal Environment Agency (UBA) in Langen, Germany, in joint cooperation with EC/ JRC/IES/ERLAP and WHO-CC.

Since 1990 ERLAP organizes IEs aiming at evaluating the comparability of measurements carried out by NRLs and promoting information exchange among the expert laboratories.
Currently, a more systematic approach has been adopted, in accordance with the Network of National Reference Laboratories for Air Quality (AQUILA) [11], aiming both at providing an alert mechanism for the purposes of the EU legislation and at supporting the implementation of laboratory quality systems by NRLs.
The methodology for the organization of IEs was developed by ERLAP in collaboration with AQUILA and is described in a paper on the organization of laboratory comparison exercises for gaseous air pollutants [12].
The AQUILA Network, managed by the JRC, provides expert judgement, promotes the harmonization of air quality measurements among European Countries and partners, coordinates the Quality Assurance and Quality Control (QA/QC) programs, method development and validation, participates in standardization activities, develops

common research projects and piloting studies and offers a forum for information exchange though training courses, workshops and conferences.
The evaluation scheme for the IEs was adopted by AQUILA in December 2008 and is applied to all IEs since then. It contains common criteria to alert the EC on possible performance failures which do not rely solely on the uncertainty claimed by participants. The evaluation scheme implements the z'-score method [13] with the uncertainty requirements for calibration gases stated in the European standards [2], [3], [4] and [5], which are consistent with the DQOs of European Directives.
According to the new amendment of the Air Quality Directives (detailed in Commission Directive 2015/1480/EC) [42] NRLs take part at least every three years in the Union-wide quality assurance program organized by the Commission's Joint Research Centre. If this participation produces unsatisfactory results then the National Laboratory should demonstrate at the next participation in the intercomparison satisfactory remediation measures, and provide a report to the Joint Research Centre [42]. In addition, considering that the evaluation scheme should be useful to participants for accreditation according to ISO 17025, they are requested to include their measurement uncertainty. Hence, participants' results (measurement values and uncertainties) are compared to the assigned values applying the E_n – number method [13].
Beside the proficiency of participating laboratories, the repeatability and reproducibility of standardized measurement methods [14], [15] and [16] are evaluated as well. These group evaluations are useful indicators of trends in measurement quality over different IE.

4. Inter-laboratory organization

The IE was announced in February 2015 to the members of the AQUILA network and the WHO CC representatives. Registration was opened in April 2015 and closed at the end of September 2015.
The participants were required to bring their own measurement instruments, data acquisition equipment and travelling standards (to be used for calibrations or checks during the IE).

The participants were invited to arrive on Sunday, 4th of October 2015, for the installation of their equipment. On the following morning the gas generation program started at 9:00 with NO mixture. On the 6th of October at 8:45 the zero air analysis for NO_2 measurement started. SO_2 and CO measurement was carried out on the following day starting at 8:45. O_3 was measured on Thursday the 8th of October from 8:45 am till 15:15 when the IE ended.

4.1. Participants

All participating laboratories belonged to institutions dealing with routine ambient air quality monitoring or to institutions involved in public health protection. The representatives came from following countries: Russian Federation, Croatia, Ukraine, Serbia, Former Yugoslav Republic of Macedonia and Germany. Further details are given in Table 1.

Country	Laboratory	Code	Network	Method
Serbia	Institute of Public Health (IPH_S)	A	AQUILA/ WHO	automatic
Ukraine	State Institution 'O.M. Marzeev Institute of Hygiene and Medical Ecology, Academy of Medical Sciences of Ukraine' (IHME)	B	WHO	Semi-auto/manual
Former Yugoslav Republic of Macedonia	Ministry of Environment and Physical Planning (MOEPP)	C	WHO	automatic
Russian Federation	State Environmental Institution 'Mosecommonitoring' (MOSECOM)	D	WHO	automatic
Croatia	Institute for Medical Research and Occupational Health (IMI)	E	AQUILA/ WHO	automatic
Croatia	Meteorological and Hydrological Service of Croatia (DHZ-TES)	F		
Germany	Federal Environment Agency (UBA)	G	AQUILA	automatic

Table 1: The list of participating organizations.

In Table 2 are reported the manufacturer and model of the instrumentation used by every participant during the inter-laboratory comparison exercise included those used in the evaluation of the assigned values.

As a whole, the instrumentation was manufactured by 4 different companies for all parameters analyzed.
The list contains the information reported by participants and by no means can be considered as an implicit or explicit endorsement of the organizers to any specific type of instrumentation.

Gas	Lab Code	Instrument
CO	A	HORIBA, 2008, APMA-370
	C	Thermo Environment TEI 48C
	D	Horiba, 2013, Carbon monoxide gas analyzer AP-370 model APMA 370
	E	HORIBA, APMA – 370, 2010
	F	EAS Envimet 300E
	G	HORIBA, 2008, APMA 370
NOX	A	HORIBA, 2008, APNA-370
	B	
	C	Thermo Environment TEI 42C
	D	Horiba, 2013, Nitrogen gas analyzer AP-370, model APNA 370
	E	HORIBA, APNA – 370, 2013
	F	EAS Envimet 200E
	G	HORIBA, 2009, APNA 370
O3	A	HORIBA, 2008, APOA-370
	B	
	C	Thermo Environment, TEI 49C
	D	Environnement S.A., 2011, Ozone gas analyzer O342M
	E	HORIBA, APOA – 370, 2013
	F	EAS Envimet 400E
	G	Thermo Sciientific, 2009, TE 49i
SO2	A	HORIBA, 2009, APSA-370
	B	
	C	Thermo Environment, TEI 43C
	D	
	E	HORIBA, APSA – 370, 2010
	F	EAS Envimet 100E
	G	HORIBA, 2012, APSA 370

Table 2: The list of instruments used by participants.

Semi-automatic method adopted by laboratory B:

- The NO_2 method is based on the interaction of nitrogen dioxide and sulfanilic acid with a formation of diazo compound which sets off an azo dye in reaction with ά-naphthylamin. Diazo compound colors the solution from light rose to red-violet. The amount of nitrogen dioxide is determined by color intensity (manual, photocolorimetric method, wave length of 540 nm). Range of measurements and error: 0.02 to 0.64 mg/m3; e= ± 25 %
- NO method is based on the oxidation of nitrogen oxide of chromic acid till dioxide and on the catching of the dioxide with the help of potassium iodine. The diazo compound is formed during the interaction of nitrogen dioxide with sulfanilic acid. This diazo compound is colored from light rose to red-violet while reacting with ά-naphthylamin. The amount of nitrogen dioxide is determined by color intensity (manual, photocolorimetric method, wave length of 540 nm). Range of measurements and error: 0.013 to 0.28 mg/m3; e= ± 25 %
- O_3 method is based on the displacement of iodine with ozone while ozone is absorbed by potassium iodine with a buffer based on boric acid. Extracted iodine is determined with a spectrometric measurement, wave length of 325 nm (manual, photo-colorimetric method). Range of measurements and error: 0.01 to 1.0 mg/m3; e= ± 25 %

4.2. Preparation of test mixtures

The facility of the UBA National Reference Laboratory is described in [9]. During this IE, gas mixtures were prepared for NO and NO_2, SO_2, CO and O_3 at concentration levels around limit values, critical levels and assessment thresholds set by European Air Quality Directive [1].

The test mixtures were prepared by the dilution of gases from cylinders containing high concentration of NO, NO_2, SO_2 or CO using thermal mass flow controllers [8]. O_3 was added using an ozone generator.

The participants were required to report three half-hour-mean measurements for each concentration level (run) in order to evaluate the repeatability of standardized measurement methods. Zero concentration levels were generated at least for one hour and one half-hour-mean measurement was reported. The sequence program of generated test gases is given in Table 3.

day	start time	duration	parameter	installation	calibration	Zero Air	NO	NO2	O3	CO	SO2
		h				nmol/mol	nmol/mol	nmol/mol	nmol/mol	mmol/mol	nmol/mol
4-Oct	15:00	3	/	X							
5-Oct	8:45	0.15	/		X						
5-Oct	9:00	2.5	NO			0					
5-Oct	11:45	1.5	NO				200				
5-Oct	13:30	1.5	NO				20				
6-Oct	8:45	1.00	NO2			0					
6-Oct	10:00	1.5	NO2					200			
6-Oct	11:45	1.5	NO2					100			
6-Oct	13:30	1.5	NO2					60			
6-Oct	15:15	1.5	NO2					20			
7-Oct	8:45	1	SO2			0					
7-Oct	10:00	1.5	SO2								130
7-Oct	11:45	1.5	SO2								45
7-Oct	13:30	1.5	SO2								20
7-Oct	15:15	1.5	SO2								5
7-Oct	17:00	1	CO			0					
7-Oct	18:00	2	CO							8	
7-Oct	20:00	2	CO							6	
7-Oct	22:00	2	CO							3	
8-Oct	0:00	2	CO							1	
8-Oct	2:00	2	CO							4,5	
8-Oct	8:45	1	O3			0					
8-Oct	10:00	1.5	O3						300		
8-Oct	11:45	1.5	O3						100		
8-Oct	13:30	1.5	O3						60		
8-Oct	15:15	1.5	O3						20		
9-Oct	8:45	0.15	evaluation								
9-Oct	9:00	3	dismantling								

Table 3: The sequence program of generated test gases with indicative pollutant concentrations

5. The evaluation of laboratory's measurement proficiency

To evaluate the participants measurement proficiency the methodology described in ISO 13528 [13] was applied. It has been agreed among the AQUILA members to take the measurement results of UBA as the assigned/reference values for the whole IE [12].
The traceability of UBA's measurement results and the method applied to validate them are presented in Annex A. In the following proficiency evaluations, the uncertainty of test gas homogeneity (Annex A) was added to the uncertainties of UBA's measurement results.

All data submitted by participating laboratories are reported in Annex B.
As it is described in the position paper [12], the proficiency of the participants was assessed by calculating two performance indicators.
The first performance indicator (z'-score) tests whether the difference between the participants measured value and the assigned/reference value remains within the limits of a common criterion.
The second performance indicator (E_n-number) tests if the difference between the participants measured values and the assigned/reference value remains within the limits of a criterion, that is calculated individually for each participant, from the uncertainty of the participants measurement result and the uncertainty of the assigned/reference value.

5.1. z'–score

The z'- score statistic is calculated according to ISO 13528 [13] as:

$$z' = \frac{x_i - X}{\sqrt{\sigma_p^2 + u_X^2}} = \frac{x_i - X}{\sqrt{(a \cdot X + b)^2 + u_X^2}} \qquad \textbf{Equation 1}$$

where 'x_i' is a participant's average value for each run, 'X' is the assigned/reference value, 'σ_p' is the 'standard deviation for proficiency assessment' and 'u_X' is the standard uncertainty of the assigned value. For '*a*' and '*b*' see Table 4.

In the European standards [2], [3], [4] and [5] the uncertainties for calibration gases used in ongoing quality control are prescribed. In fact, it is stated that the maximum permitted expanded uncertainty for calibration gases is 5% and that 'zero gas' shall not give instrument reading higher than the detection limit. As one of the tasks of NRLs is to supply calibration gas mixtures, the 'standard deviation for proficiency assessment' (σ_p) [13] is calculated in fitness-for-purpose manner from requirements given in European standards.
Over the whole measurement range σ_p is calculated by linear interpolation between 2.5% at the calibration point (75% of calibration range) and the limit of detection at zero concentration level. The limits of detection of studied measurement methods were evaluated from the data of previous IE. The linear function parameters of σ_p are given in Table 4:

Gas	σ_p=a·c+b a	b nmol/mol
SO_2	0.022	1
CO	0.024	100
O_3	0.020	1
NO	0.024	1
NO_2	0.020	1

Table 4: The standard deviation for proficiency assessment (σ_p).
σ_p is a linear function of concentration (c) with parameters: slope (a) and intercept (b).

The assessment of results in the z'-score evaluation is made according to the following criteria:

- $|z'| \leq 2$ are considered satisfactory.
- $2 < |z'| \leq 3$ are considered questionable.
- $|z'| > 3$ are considered unsatisfactory. Scores falling in this range are very unusual and are taken as evidence that an anomaly has occurred that should be investigated and corrected.

The results of z'-score evaluation are presented in bar plots (Figure 1 to Figure 5) in which the z'-scores of each participant are grouped together, and assessment criteria are presented as z'=±2 and z'=±3 lines. The laboratory G is used as reference value.

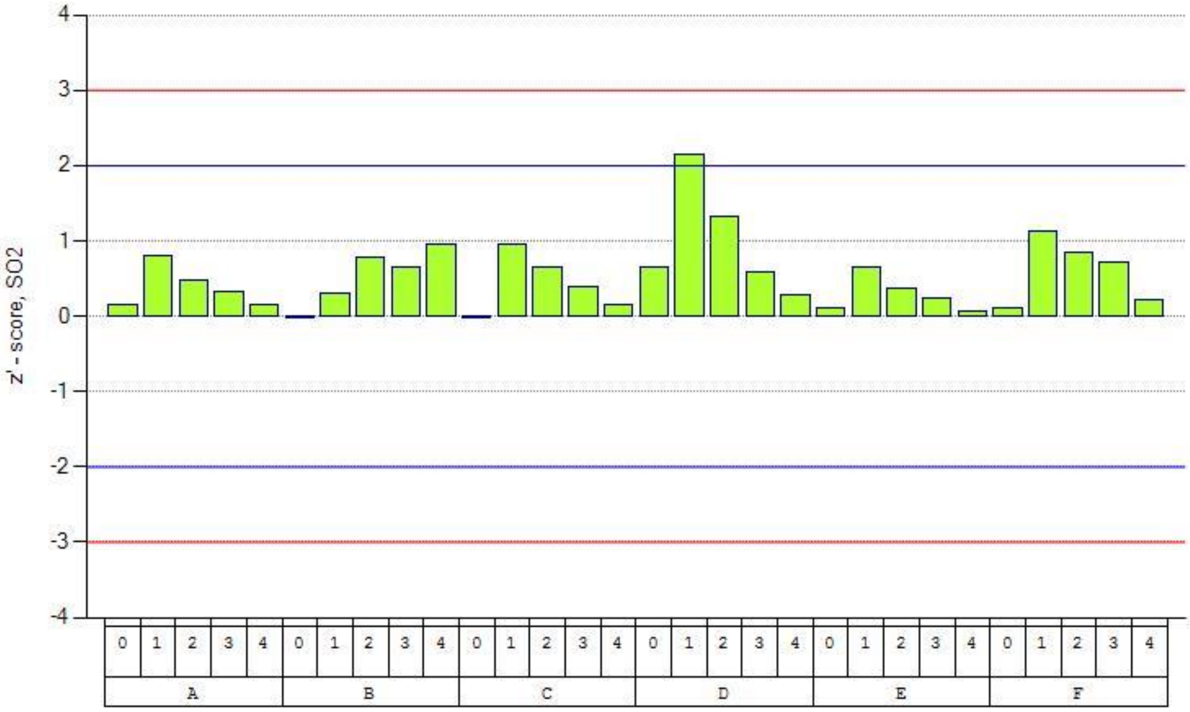

Figure 1: The z'-score evaluations of SO_2 measurements
Scores are given for each participant and each tested concentration level (run). Run number order (with nominal concentration) is: 0 (0 nmol/mol), 1 (130 nmol/mol), 2 (45 nmol/mol), 3 (20 nmol/mol), 4 (5 nmol/mol). The assessment criteria are presented as z'=±2 (blue line) and z'=±3 (red line). They represent the limits for the questionable and unsatisfactory results.

Figure 2: The z'-score evaluations of CO measurements

Scores are given for each participant and each tested concentration level (run). Run number order (with nominal concentration) is: 0 (0 µmol/mol), 1 (8 µmol/mol), 2 (6 µmol/mol), 3 (3 µmol/mol), 4 (1 µmol/mol), 5 (4,5 µmol/mol). The assessment criteria are presented as z'=±2 (blue line) and z'=±3 (red line). They represent the limits for the questionable and unsatisfactory results.

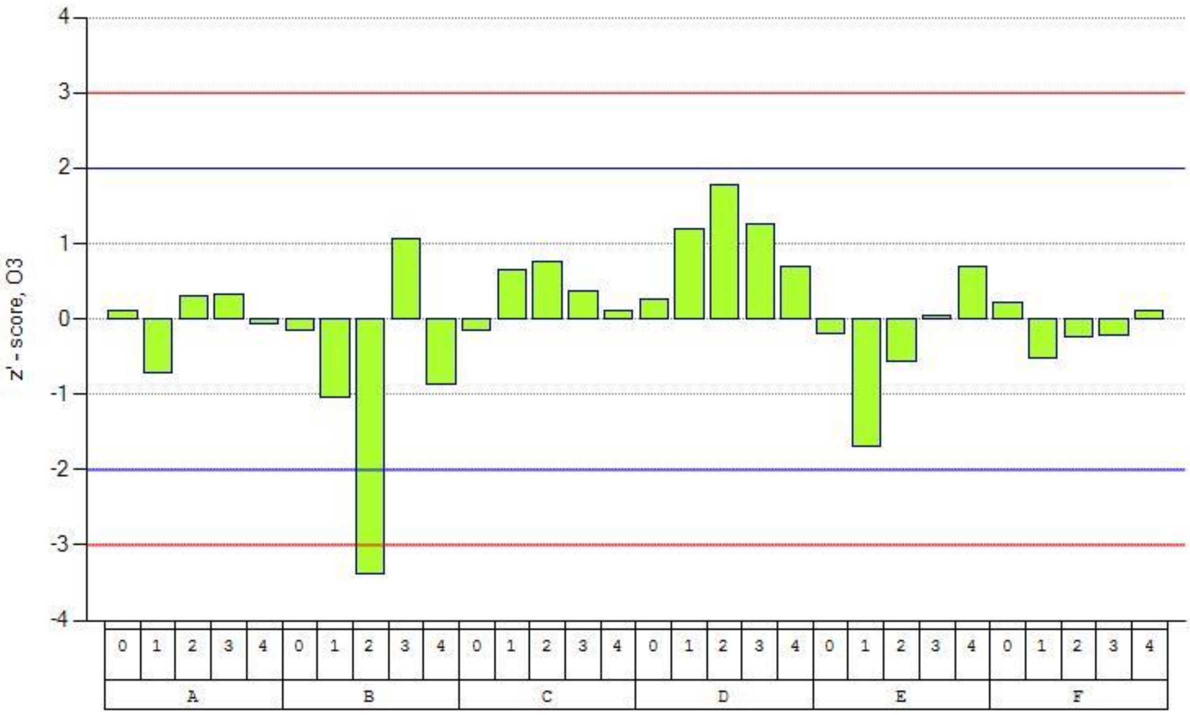

Figure 3: The z'-score evaluations of O_3 measurements

Scores are given for each participant and each concentration level (run). Run number order (with nominal concentration) is: 0 (0 nmol/mol), 1 (300 nmol/mol), 2 (100 nmol/mol), 3 (60 nmol/mol), 4 (20 nmol/mol). The assessment criteria are presented as z'=±2 (blue line) and z'=±3 (red line). They represent the limits for the questionable and unsatisfactory results.

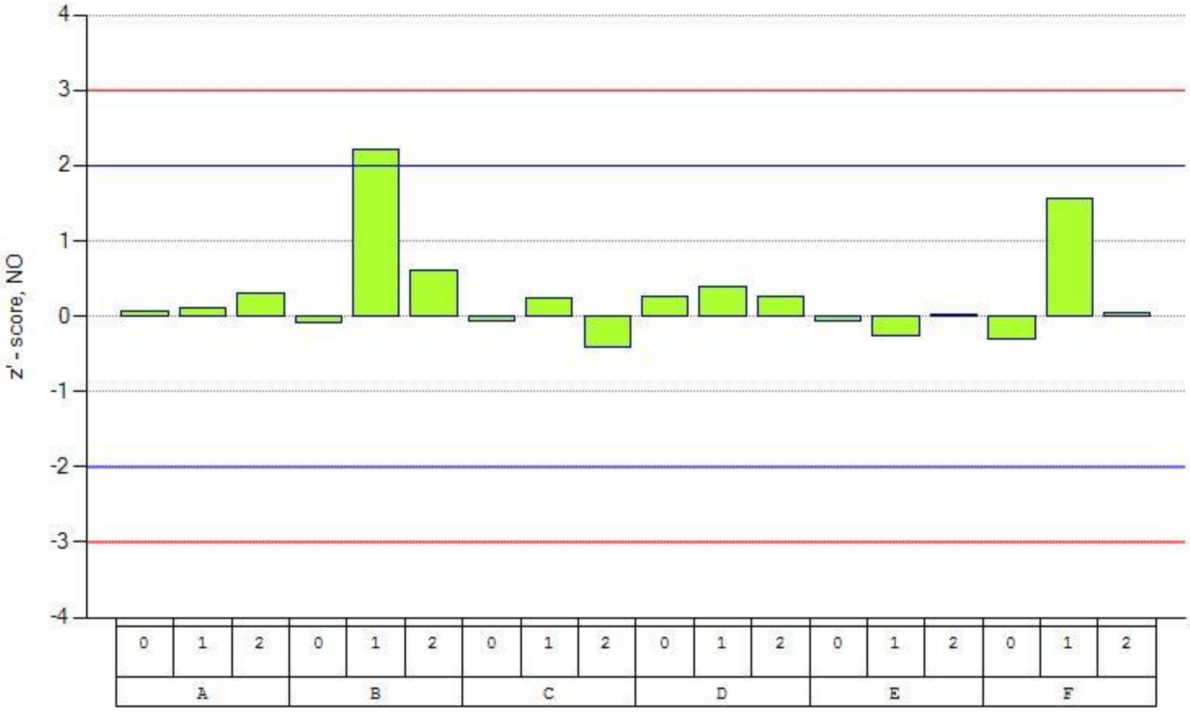

Figure 4: The z'-score evaluations of NO measurements
Scores are given for each participant and each tested concentration level (run). Run number order (with nominal concentration) is: 0 (0 nmol/mol), 1 (200 nmol/mol), 2 (20 nmol/mol). The assessment criteria are presented as z'=±2 (blue line) and z'=±3 (red line). They represent the limits for the questionable and unsatisfactory results.

Figure 5: The z'-score evaluations of NO_2 measurements
Scores are given for each participant and each concentration level (run). Run number order (with nominal concentration) is: 0 (0 nmol/mol), 1 (200 nmol/mol), 2 (100 nmol/mol), 3 (60 nmol/mol), 4 (20 nmol/mol). The assessment criteria are presented as z'=±2 (blue line) and z'=±3 (red line). They represent the limits for the questionable and unsatisfactory results.

5.2. *E_n* - number

The normalized deviations [13] (E_n) were calculated according to:

$$E_n = \frac{x_i - X}{\sqrt{U_{x_i}^2 + U_X^2}}$$ **Equation 2**

where 'X' is the assigned/reference value with an expanded uncertainty 'U_X' and 'x_i' is the participant's average value with an expanded uncertainty 'U_{Xi}'. Satisfactory results are the ones for which $|E_n| \leq 1$.

In Figure 6 to Figure 10 the bias of each participant (x_i-X) are plotted and error bars are used to show the value of denominator of equation 2 $\left(\sqrt{U_{x_i}^2 + U_X^2}\right)$. These plots represent also the E_n-number evaluations where, considering the E_n criteria ($|E_n| \leq 1$), all results with error bars touching or crossing the x-axis are considered satisfactory. Reported standard uncertainties (Annex B) being bigger than "standard deviation for proficiency assessments" (σ_p, Table 4) are considered not fit-for-purpose and are denoted with "*" in the x-axis of each figure. The En evaluation showed only one unsatisfactory result for one concentration of ozone measurement, as reported in table 5.

Parameter	Lab Code	Value	Run	En	En evaluation
O3	E	297.9	O3 _1	1.5	unsatisfactory

Table 5: Unsatisfactory results according to En number.

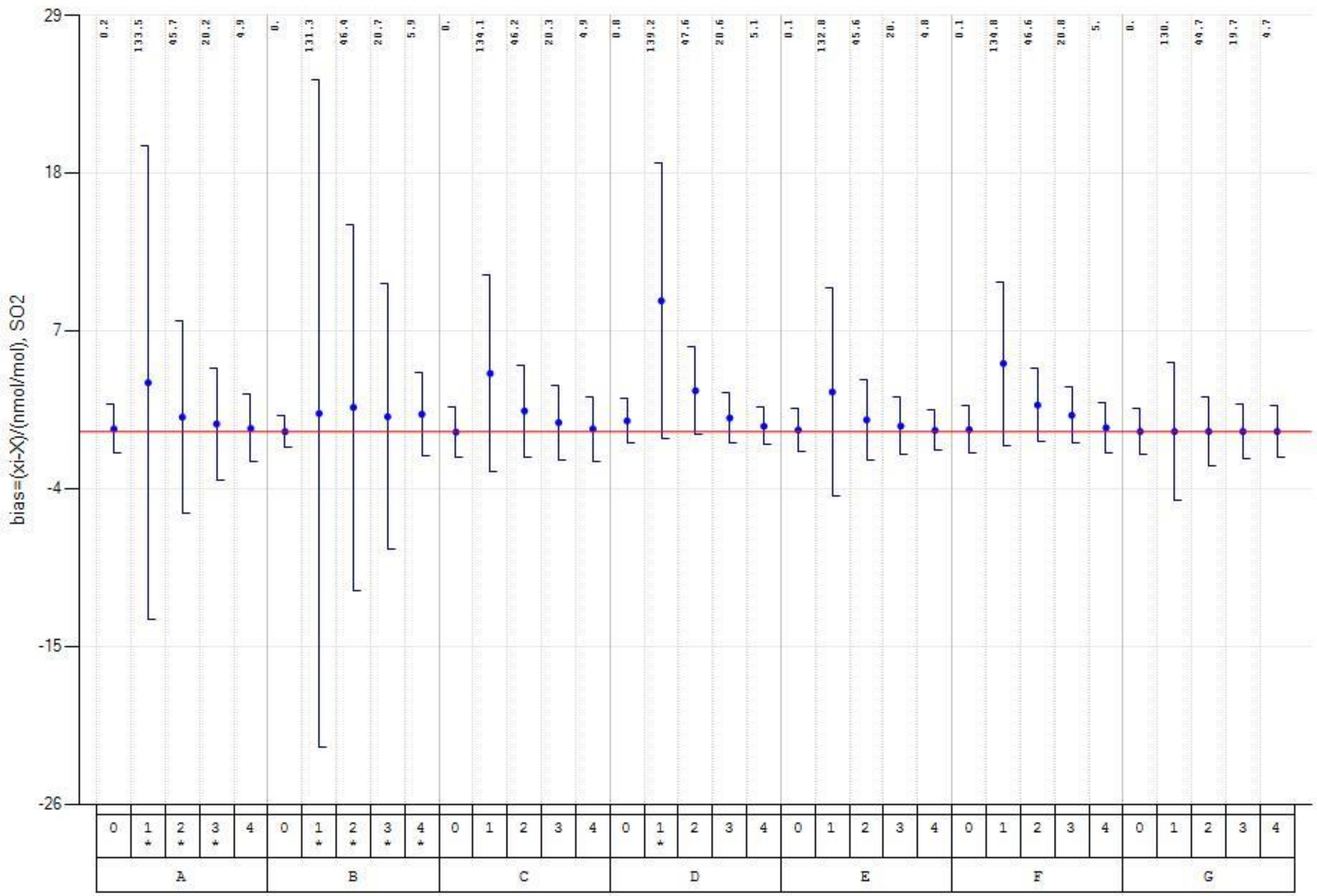

Figure 6: Bias of participant's SO_2 measurement results
Expanded uncertainty of bias for each run is presented as error bar. The results with error bars touching or crossing the x-axis are satisfactory. For each evaluation the run number (numbers 0 to 4) together with the participants rounded run average (nmol/mol) is given. The '*' mark indicates reported standard uncertainties bigger than σ_p.

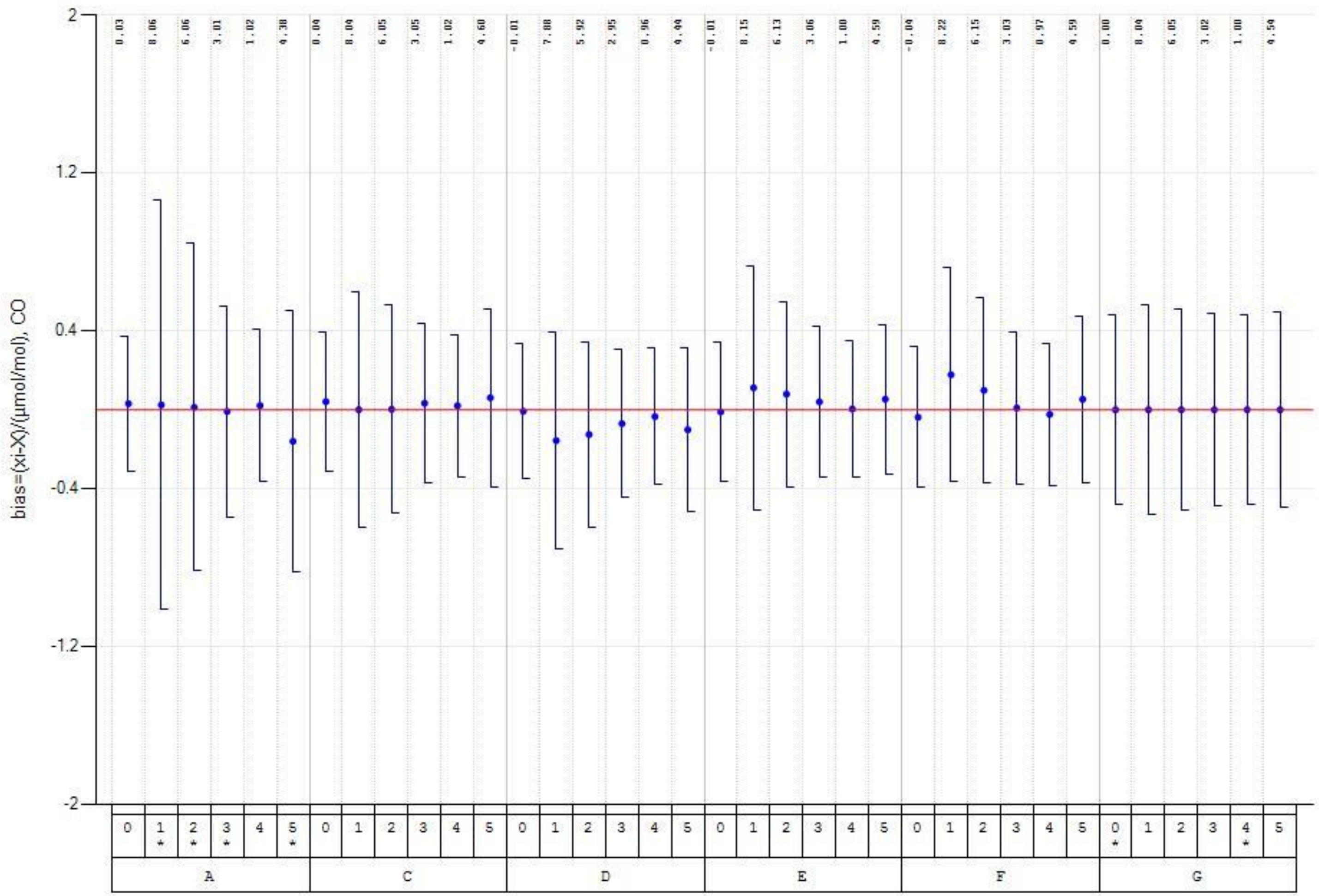

Figure 7: Bias of participant's CO measurement results
Expanded uncertainty of bias for each run is presented as error bar. Results with error bars touching or crossing the x-axis are satisfactory. For each evaluation the run number (numbers 0 to 5) together with the participants rounded run average (µmol/mol) is given. The '*' mark indicates reported standard uncertainties bigger than σ_p.

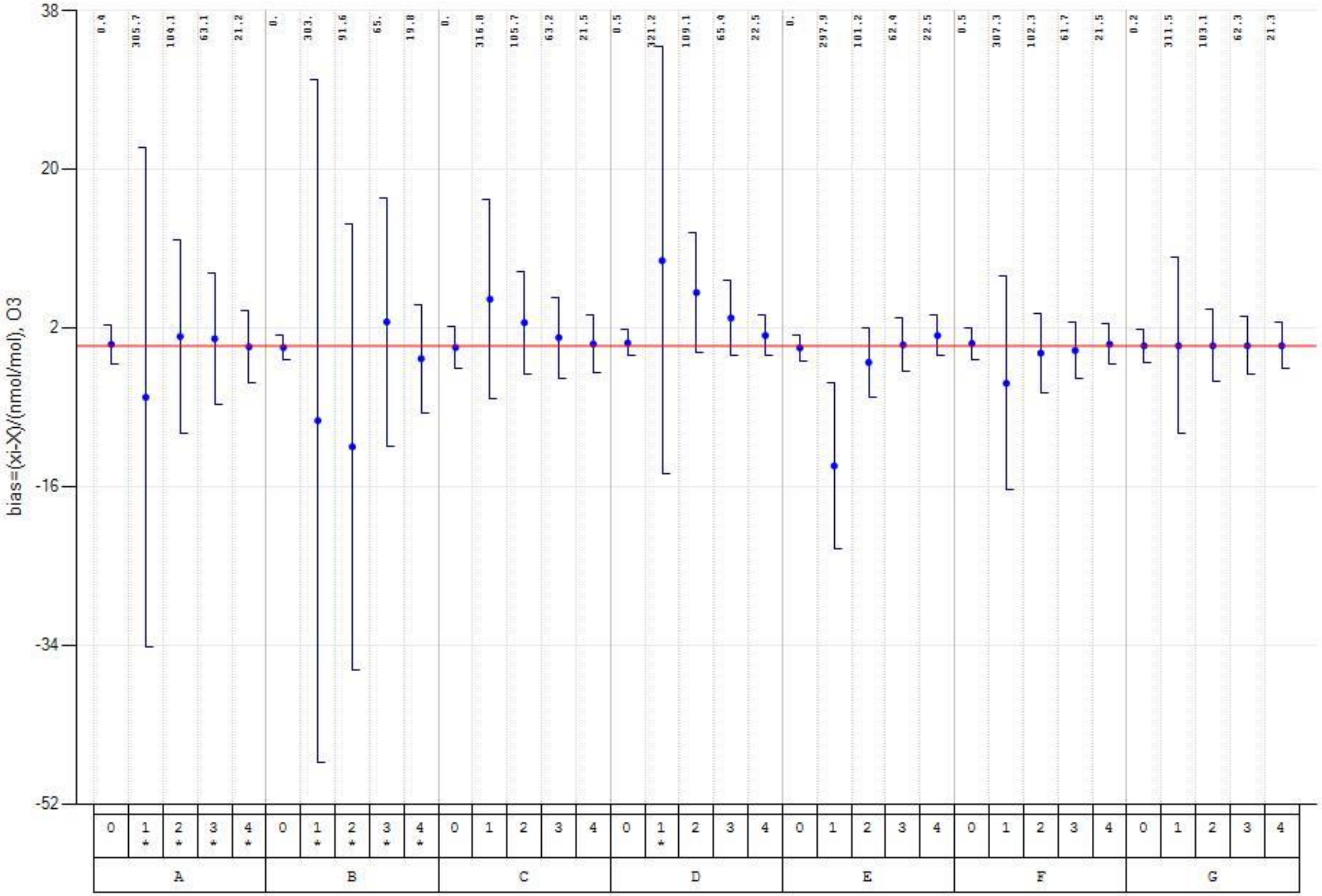

Figure 8: Bias of participant's O_3 measurement results
Expanded uncertainty of bias for each run is presented as error bar. Results with error bars touching or crossing the x-axis are satisfactory. For each evaluation the run number (numbers 0 to 4) together with the participants rounded run average (nmol/mol) is given. The '*' mark indicates reported standard uncertainties bigger than σ_p.

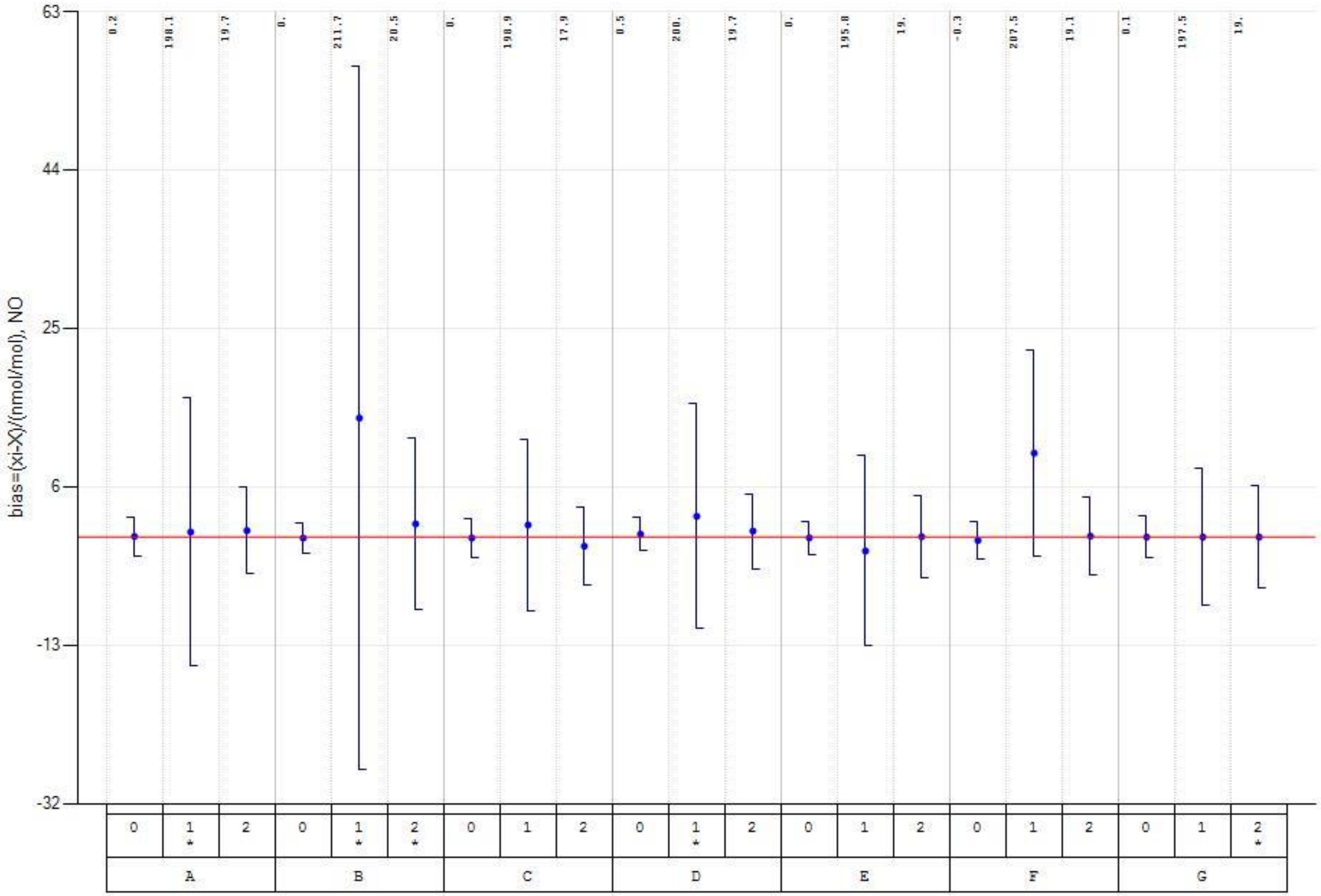

Figure 9: Bias of participant's NO measurement results
Expanded uncertainty of bias for each run is presented as error bar. Results with error bars touching or crossing the x-axis are satisfactory. For each evaluation the run number (numbers 0 to 2) together with the participants rounded run average (nmol/mol) is given. The '*' mark indicates reported standard uncertainties bigger than σ_p.

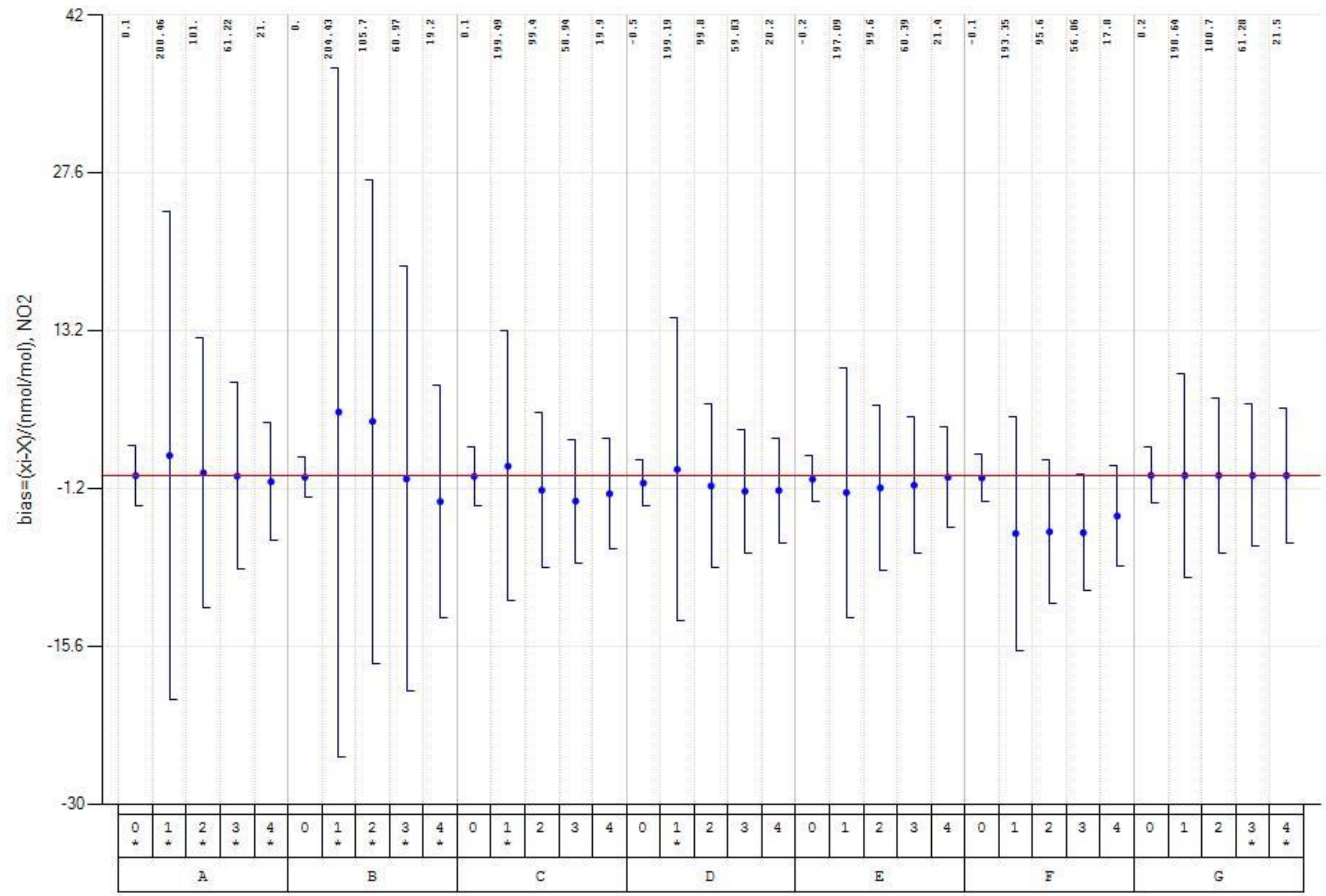

Figure 10: Bias of participant's NO_2 measurement results
Expanded uncertainty of bias is presented as error bar. Results with error bars touching or crossing the x-axis are satisfactory. For each evaluation the run number (0 to 4) together with the participants rounded run average (nmol/mol) is given. The '*' mark indicates reported standard uncertainties bigger than σ_p.

6. Discussion

For a general assessment of the quality of each result a decision diagram was developed (Figure 11) that divides results into seven categories (1 to 7). The description for each category is as follow:

- **1**: measurement result is completely satisfactory
- **2**: measurement result is satisfactory (z'-score satisfactory and En-number ok), but the reported uncertainty is too high.
- **3**: measured value is satisfactory (z'-score satisfactory), but the reported uncertainty is underestimated (En-number not ok).
- **4**: measurement result is questionable (z'-score questionable), but due to a high reported uncertainty can be considered valid (En-number ok).
- **5**: measurement result is questionable (z'-score questionable and En-number not ok).
- **6**: measurement result is unsatisfactory (z'-score unsatisfactory), but due to a high reported uncertainty can be considered valid (En-number ok).
- **7**: measurement result is unsatisfactory (z'-score unsatisfactory and En-number not ok).

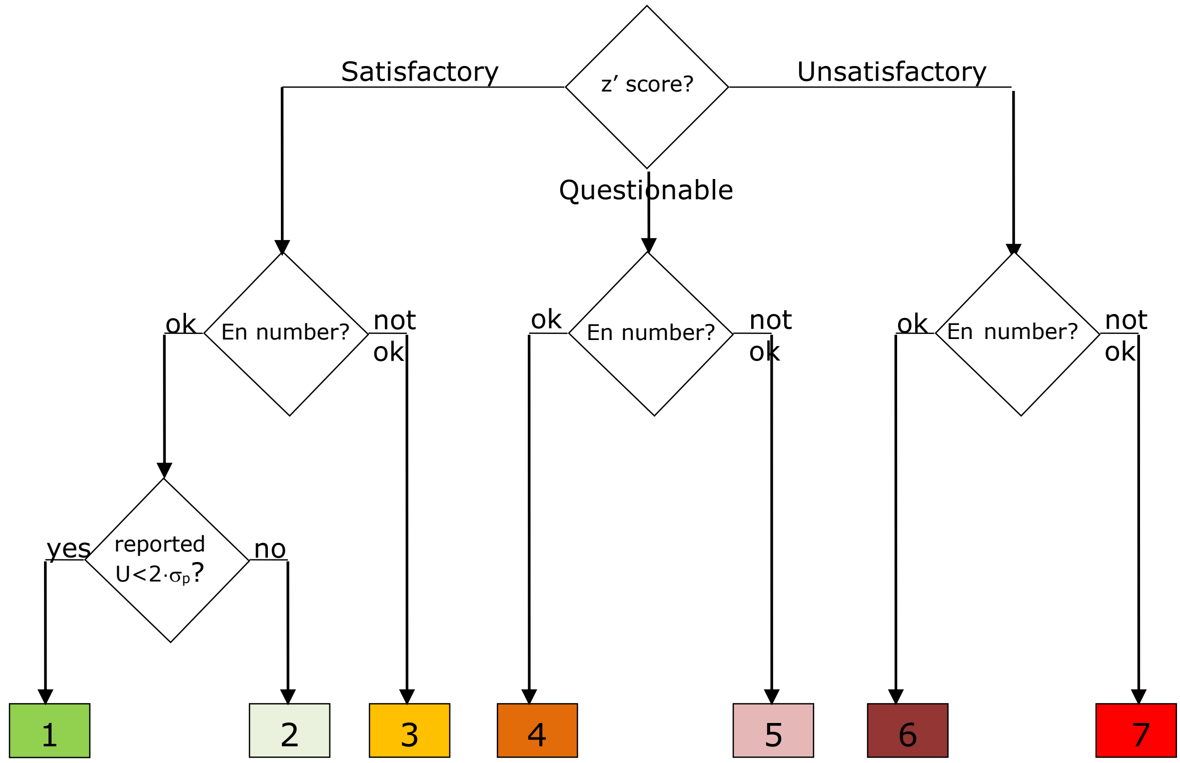

Figure 11: The decision diagram for general assessment of proficiency results.

The results of the IE were assigned to categories according to the diagram given in Figure 111 and are presented in the following Table 6.

Param.	Span	ref. conc. level	IE code					
			A	B	C	D	E	F
CO (µmol/mol)	0	-0.003	1	nd	1	1	1	1
	1	8.040	2	nd	1	1	1	1
	2	6.050	2	nd	1	1	1	1
	3	3.020	2	nd	1	1	1	1
	4	0.995	1	nd	1	1	1	1
	5	4.536	2	nd	1	1	1	1
NO (nmol/mol)	0	0.12	1	1	1	1	1	1
	1	197.47	2	4	1	2	1	1
	2	18.96	1	2	2	1	1	1
NO_2 (nmol/mol)	0	0.15	2	1	1	1	1	1
	1	198.64	2	2	1	2	1	1
	2	100.72	2	2	1	1	1	1
	3	61.28	2	2	1	1	1	1
	4	21.54	2	2	1	1	1	1
O_3 (nmol/mol)	0	0.20	1	1	1	1	1	1
	1	311.52	2	2	1	2	3	1
	2	103.08	2	6	1	1	1	1
	3	62.25	2	2	1	1	1	1
	4	21.30	2	2	1	1	1	1
SO_2 (nmol/mol)	0	0.01	1	1	1	1	1	1
	1	130.04	2	2	1	4	1	1
	2	44.74	2	2	1	1	1	1
	3	19.66	2	2	1	1	1	1
	4	4.73	1	2	1	1	1	1

Table 6: The general assessment of proficiency results. (n.d. not determined)

7. Conclusions

The proficiency evaluation scheme has provided an assessment of the participants measured values and their reported uncertainties.

As described in Table 7, in terms of criteria imposed by the European Directive (σ_p) the majority (73.2%, category '1') of the results reported by the laboratories falls into category '1' and are satisfactory both in terms of measured values and evaluated uncertainties. Among the remaining results the 24.6% are satisfactory values, but the evaluated uncertainties are either too high, category '2' (23.9%), or too small, category '3' (0.7%). Two results are found questionable for z'-score and valid for the En number (1.4% in category '4'). Only one result is found unsatisfactory for z'-score and valid for En-number (0.7% in category '6').

IE	Site	Categories %						
		1	2	3	4	5	6	7
Apr-08	Ispra (IT)	68.4	18.1	7.3	1.0	1.0	2.6	1.6
Oct-08 (I)	Ispra (IT)	37.9	40.8	14.2	0.6	3.6	1.0	1.9
Oct-08 (II)	Ispra (IT)	34.3	38.9	23.7	1.0	2.0	0.0	0.0
Sep-09	**Langen (DE)**	**60.8**	**29.9**	**3.1**	**4.1**	**1.0**	**1.0**	**0.0**
Oct-09	Ispra (IT)	85.0	5.7	7.5	0.4	1.4	0.0	0.0
Jun-10	Ispra (IT)	84.6	8.1	4.4	0.7	2.3	0.0	0.0
Sep-11	Ispra (IT)	86.1	7.9	5.4	0.0	0.3	0.0	0.3
Oct-11 (I)	Ispra (IT)	78.6	12.5	7.6	0.0	1.3	0.0	0.0
Oct-11 (II)	**Langen (DE)**	**59.4**	**39.9**	**0.0**	**0.7**	**0.0**	**0.0**	**0.0**
Jun-12	Ispra (IT)	92.2	0.5	7.3	0.0	0.0	0.0	0.0
Sep-13	**Langen (DE)**	**75.7**	**20.9**	**2.0**	**0.0**	**1.4**	**0.0**	**0.0**
Sep-13	Ispra (IT)	89.4	7.3	3.3	0.0	0.0	0.0	0.0
Oct-13	Ispra (IT)	86.8	8.9	3.6	0.4	0.4	0.0	0.0
May-14	Ispra (IT)	81.8	15.2	1.1	0.0	0.7	0.0	1.1
Oct-15	**Langen (DE)**	**73.2**	**23.9**	**0.7**	**1.4**	**0.0**	**0.7**	**0.0**

Table 7: Category summary

As in previous IE, the adopted criteria for high concentrations were the standard deviations for proficiency assessment, deriving from the European Standards' uncertainty requirements.

The reproducibility standard deviation obtained at this (Annex C) and previous IE [20], [21], [22], [23], [24], [25], [33], [34], [35], [36], [37], [38], [39], [40] and [41] is comparable to the mentioned criteria. On the other hand, the uncertainty criteria for zero levels were those set in AQUILA's position paper [12].

In the present IE a high share of '1' results can be observed confirming the good general performance of laboratories participating in this IE, in 2013 and 2011. It is remarkable the improvement in this IE for the only few results found questionable and unsatisfactory. In this exercise 97.9% of the results in the z'-score evaluations (Table 8) were satisfactory, 2 results were found questionable (1.4%) and 1 unsatisfactory (0.7%).

IE	*Site*	*Satisfactory (%)*	*Questionable (%)*	*Unsatisfactory (%)*
June/05	Ispra (IT)	94.7	2.3	3.0
June/07	Ispra (IT)	97.8	1.9	0.3
October/07	Essen (DE)	93.2	4.6	2.2
April/08	Ispra (IT)	93.8	2.1	4.1
October 2008_1	Ispra (IT)	92.9	4.2	2.9
October 2008_2	Ispra (IT)	97.0	3.0	0.0
September/09	**Langen (DE)**	**94.3**	**4.7**	**0.9**
October/09	Ispra (IT)	98.2	1.8	0.0
June/10	Ispra (IT)	97.0	3.0	0.0
September/11	Ispra (IT)	99.4	0.3	0.3
October/11	Ispra (IT)	98.7	1.3	0.0
October/11	**Langen (DE)**	**99.3**	**0.7**	**0.0**
June/12	Ispra (IT)	100.0	0.0	0.0
September/13	**Langen (DE)**	**98.6**	**1.4**	**0.0**
September/13	Ispra (IT)	100.0	0.0	0.0
October/13	Ispra (IT)	99.3	0.7	0.0
May/14	Ispra (IT)	98.1	0.7	1.1
October/15	**Langen (DE)**	**97.9**	**1.4**	**0.7**

Table 8: Z'-score summary

Comparability of results among the participants at the highest concentration level, excluding outliers, is acceptable for all pollutants measurements.

The relative reproducibility limits, at the highest studied concentration levels, are 9.8% for SO_2, 5.2% for CO, 10.7% for O_3, for NO 12.3% and for NO_2 7.9% almost all within the objective derived from criteria required by the EU legislation (σ_p see Table 4).
In Figure 42 is represented a deviation of Ozone from the objectives starting at the level of 60 ppb. In Figure 38 and Figure 39 a chart shows a deviation starting respectively of NO at 50 ppb and NO_2 at 20 ppb.

Laboratory B didn't report any values for CO measurements.

During this IE the performance of all NRL was generally satisfactory. Only one laboratory (B) had an unsatisfactory value for O_3 measurement at high concentration that requires a cause analysis.

8. References

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Annex A. Assigned values

The assigned values of tested concentration levels (run) were derived from UBA measurements which are calibrated against the certified reference values of CRMs and are traceable to international standards. In this perspective the assigned values are reference values as defined in the ISO 13528 [13].
UBA's SO_2, CO and NO analysers were calibrated according to the methodology described in the ISO 6143 [6]. The procedure and the device for generating primary calibration gases is described elsewhere [31]. Gas mixtures for the calibration experiment were produced from the reference mixtures by static volumetric dilution method ISO 6144 [34].
SO_2, CO and NO gas mixtures manufactured by Air Liquide and certified by UBA (U≤ 2%) were used as internal standards.
For the reference gas mixture composition evaluation and for the calibration experiment evaluation the computer application "GUM WORKBENCH" 0 was used.
For O_3 measurements, the primary standard NIST photometer SRP 29 was used.
UBA's measurement results were validated by comparison to the group statistics (x* and s*) for every parameter and concentration level of the IE. These statistics are calculated from participants, applying the robust method described in the Annex C of the ISO 13528 [13]. The validation is taking into account UBA's measurement result (X) and its standard uncertainty (u_X) as given in Equation 3:

$$\frac{\left|x^* - X\right|}{\sqrt{\frac{\left(1{,}25 \cdot s^*\right)^2}{p} + u_X^2}} < 2$$

Equation 3

Where 'x*' and 's*' represent robust average and robust standard deviation respectively and 'p' is the number of participants.

In Table 9 all inputs for Equation 3 are given and all UBA's measurement results are confirmed to be valid.

As a group evaluation robust average (x*) and robust standard deviation (s*) were calculated (applying the procedure described in Annex C of ISO 13528) for each run, and are presented in the following tables.

run	unit	X	uX'	x*	s*	p	val.
NO _0	nmol/mol	0.12	0.90	0.07	0.14	7	OK
NO _1	nmol/mol	197.47	2.92	199.22	2.70	7	OK
NO _2	nmol/mol	18.96	2.19	19.26	0.90	7	OK
NO2 _0	nmol/mol	0.15	0.90	-0.03	0.20	7	OK
NO2 _1	nmol/mol	198.64	3.32	199.04	2.18	7	OK
NO2 _2	nmol/mol	100.72	2.51	100.00	1.52	7	OK
NO2 _3	nmol/mol	61.28	2.30	60.17	1.25	7	OK
NO2 _4	nmol/mol	21.54	2.19	20.15	1.47	7	OK
O3 _0	nmol/mol	0.20	0.68	0.21	0.26	7	OK
O3 _1	nmol/mol	311.52	3.59	308.41	7.98	7	OK
O3 _2	nmol/mol	103.08	1.46	103.22	3.19	7	OK
O3 _3	nmol/mol	62.25	1.15	63.17	1.40	7	OK
O3 _4	nmol/mol	21.30	0.93	21.50	0.51	7	OK
SO2 _0	nmol/mol	0.01	0.56	0.11	0.14	7	OK
SO2 _1	nmol/mol	130.04	1.73	133.33	2.31	7	OK
SO2 _2	nmol/mol	44.74	0.85	46.11	0.74	7	OK
SO2 _3	nmol/mol	19.66	0.68	20.32	0.45	7	OK
SO2 _4	nmol/mol	4.73	0.63	4.96	0.19	7	OK
CO _0	µmol/mol	-0.003	0.169	-0.001	0.03	6	OK
CO _1	µmol/mol	8.040	0.189	8.067	0.10	6	OK
CO _2	µmol/mol	6.050	0.18	6.068	0.07	6	OK
CO _3	µmol/mol	3.020	0.172	3.026	0.03	6	OK
CO _4	µmol/mol	0.995	0.169	0.993	0.03	6	OK
CO _5	µmol/mol	4.536	0.176	4.550	0.06	6	OK

Table 9: The validation of assigned values (X)
by comparison to the robust averages (x*) taking into account the standard uncertainties of assigned values (uX'), and robust standard deviations (s*) as denoted by Equation 3.

The homogeneity of test gas was evaluated from measurements at the beginning and end of the distribution line. From the relative differences between beginning and end measurements, average and standard deviation were calculated, and the uncertainty of test gas due to lack of homogeneity was calculated as the sum of squares of these average and standard deviation (Equation 4). The upper and lower limits of bias due to homogeneity was evaluated to be smaller than 0.5% which constitutes the relative standard uncertainty of 0.3% of each concentration level. The standard uncertainties of assigned/reference values ($u_{X'}$) were calculated with Equation 4 and used in the proficiency evaluations of chapter 5.

$$u_{X'}^2 = u_X^2 + \left(X \cdot u_{\text{homogeneity}}\right)^2$$ **Equation 4**

Annex B. The results of the IE

In this annex are reported all participant's results, presented both in tables and graphs. For all concentrations generated (run), participants were asked to report 3 results representing 30 minutes averages (x_{ij}).
In this annex are presented the reported data and their uncertainty $u(x_i)$ and $U(x_i)$) expressed in mol/mol units.
For all the runs except concentration levels 0, also average (x_i) and standard deviation (s_i) of each participant are presented.
The assigned value is indicated on the graphs with the red line and the individual laboratories expanded uncertainties (Ux_i) are indicated with error bars.

Reported values for SO_2

	laboratories						
values	A	B	C	D	E	F	G
xi, 1	0.19	0.00	-0.03	0.75	0.12	0.14	0.01
u(xi)	0.64	0.00	0.68	0.55	0.48	0.59	0.56
U(xi)	1.28	0.00	1.37	1.11	0.96	1.18	1.12

Table 10: Reported values for SO2 run 0.

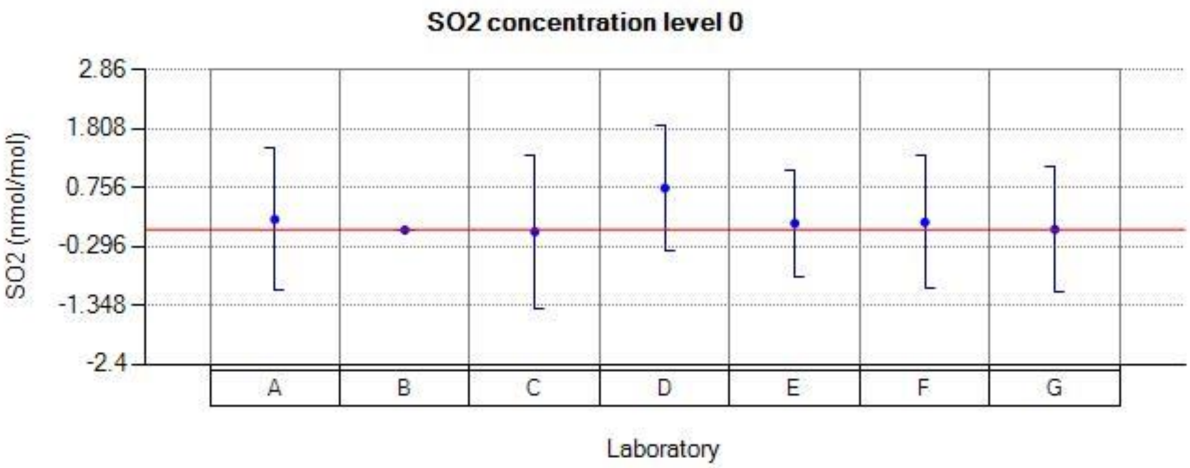


Figure 12: Reported values for SO_2 run 0.

	laboratories						
values	A	B	C	D	E	F	G
xi, 1	133.44	124.36	134.02	135.21	132.46	134.88	129.80
xi, 2	133.35	131.06	134.02	140.81	132.88	134.65	130.23
xi, 3	133.56	138.47	134.24	141.46	133.06	134.83	130.10
xi	133.45	131.29	134.09	139.16	132.80	134.78	130.04
si	0.10	7.05	0.12	3.43	0.30	0.12	0.22
u(xi)	8.06	7.22	2.95	4.46	3.21	2.27	1.69
U(xi)	16.12	22.98	5.90	8.93	6.42	4.54	3.37

Table 11: Reported values for SO_2 run 1.

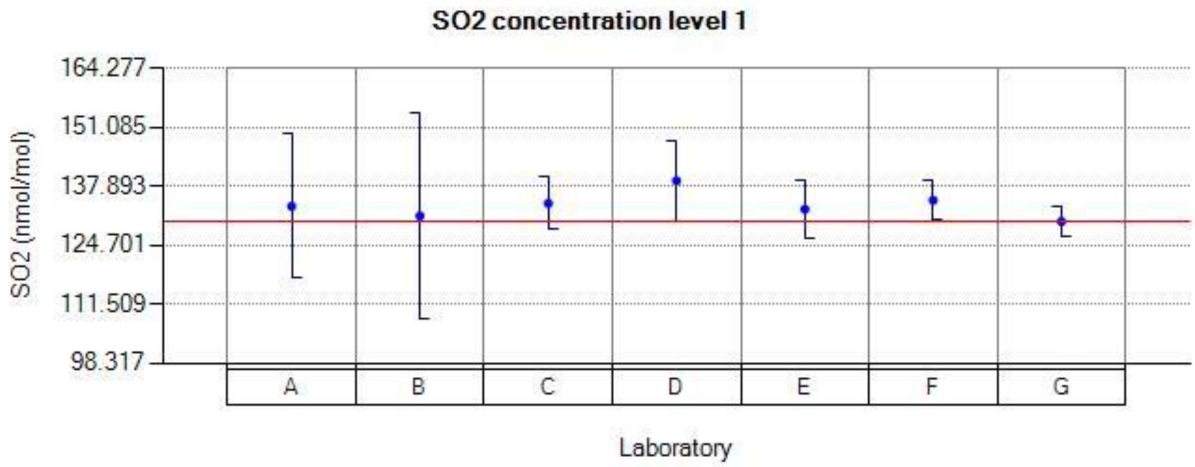


Figure 13: Reported values for SO_2 run 1.

	laboratories						
values	A	B	C	D	E	F	G
xi, 1	45.81	42.09	46.21	47.99	45.53	46.39	44.69
xi, 2	45.65	47.83	46.08	47.35	45.44	46.48	44.70
xi, 3	45.77	49.33	46.21	47.39	45.68	46.87	44.82
xi	45.74	46.41	46.16	47.57	45.55	46.58	44.73
si	0.08	3.82	0.07	0.35	0.12	0.25	0.07
u(xi)	3.24	3.97	1.35	1.27	1.14	0.98	0.83
U(xi)	6.47	12.64	2.70	2.53	2.28	1.96	1.67

Table 12: Reported values for SO_2 run 2.

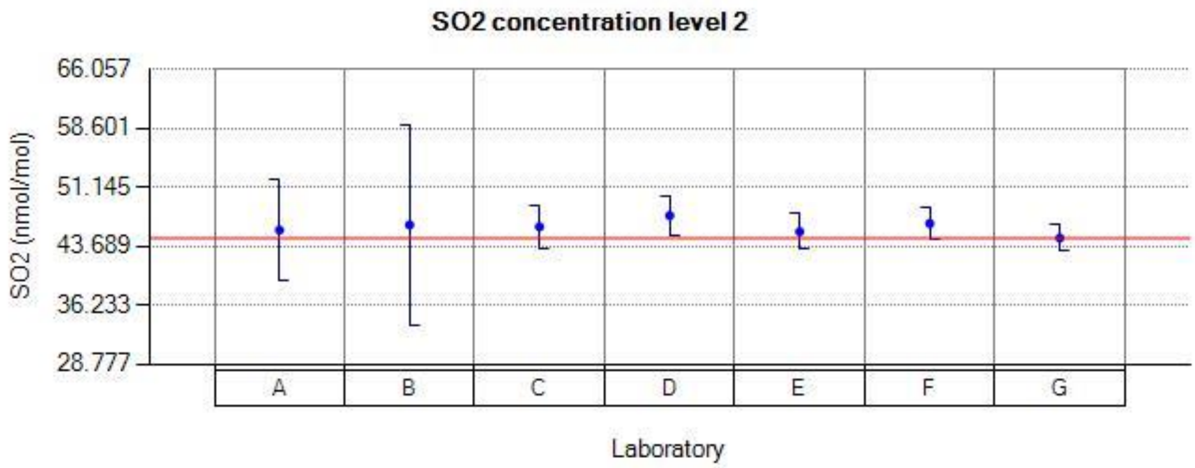


Figure 14: Reported values for SO_2 run 2.

	laboratories						
values	A	B	C	D	E	F	G
xi, 1	20.21	18.12	20.21	20.59	20.16	20.85	19.74
xi, 2	20.19	20.40	20.32	20.63	19.99	20.75	19.65
xi, 3	20.16	23.54	20.30	20.57	19.98	20.76	19.59
xi	20.18	20.68	20.27	20.59	20.04	20.78	19.66
si	0.02	2.72	0.05	0.03	0.10	0.05	0.07
u(xi)	1.83	2.88	1.09	0.53	0.74	0.71	0.68
U(xi)	3.66	9.17	2.18	1.05	1.48	1.42	1.36

Table 13: Reported values for SO_2 run 3.

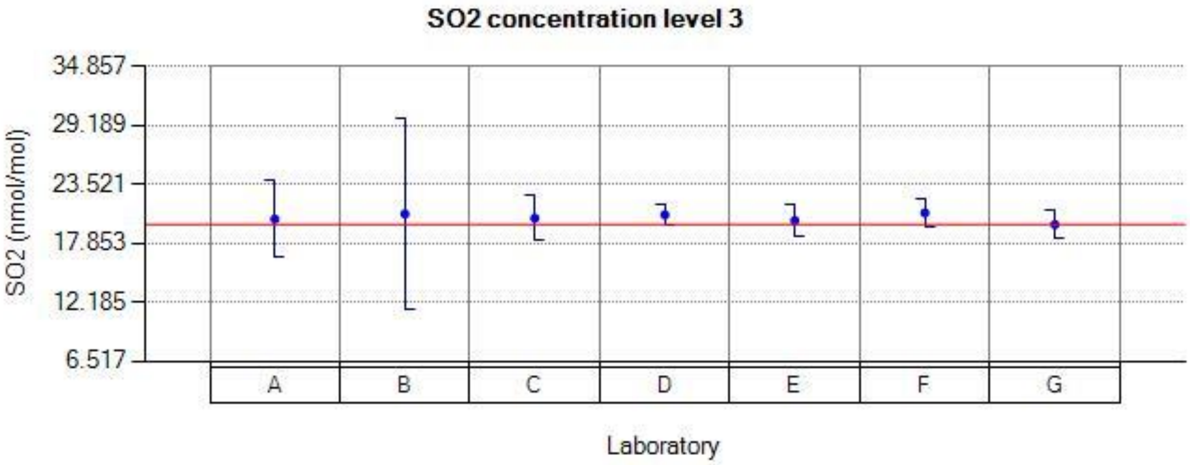


Figure 15: Reported values for SO_2 run 3.

	laboratories						
values	A	B	C	D	E	F	G
xi, 1	4.97	5.38	4.95	5.12	4.84	5.08	4.77
xi, 2	4.96	5.74	4.90	5.10	4.82	5.13	4.70
xi, 3	4.90	6.70	4.91	5.09	4.78	4.78	4.73
xi	4.94	5.94	4.92	5.10	4.81	4.99	4.73
si	0.03	0.68	0.02	0.01	0.03	0.18	0.03
u(xi)	0.99	0.82	0.93	0.13	0.31	0.62	0.63
U(xi)	1.98	2.63	1.87	0.26	0.62	1.24	1.26

Table 14: Reported values for SO_2 run 4.

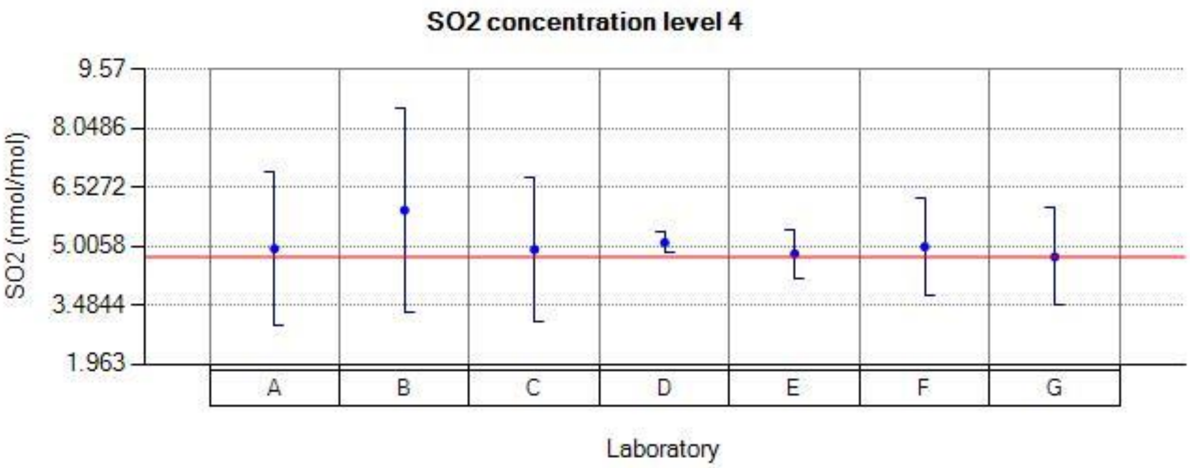


Figure 16: Reported values for SO_2 run 4.

Reported values for CO

	laboratories					
values	A	C	D	E	F	G
xi, 1	0.028	0.038	-0.011	-0.014	-0.041	-0.003
u(xi)	0.030	0.054	0.028	0.050	0.058	0.169
U(xi)	0.060	0.109	0.056	0.100	0.116	0.338

Table 15: Reported values for CO run 0.

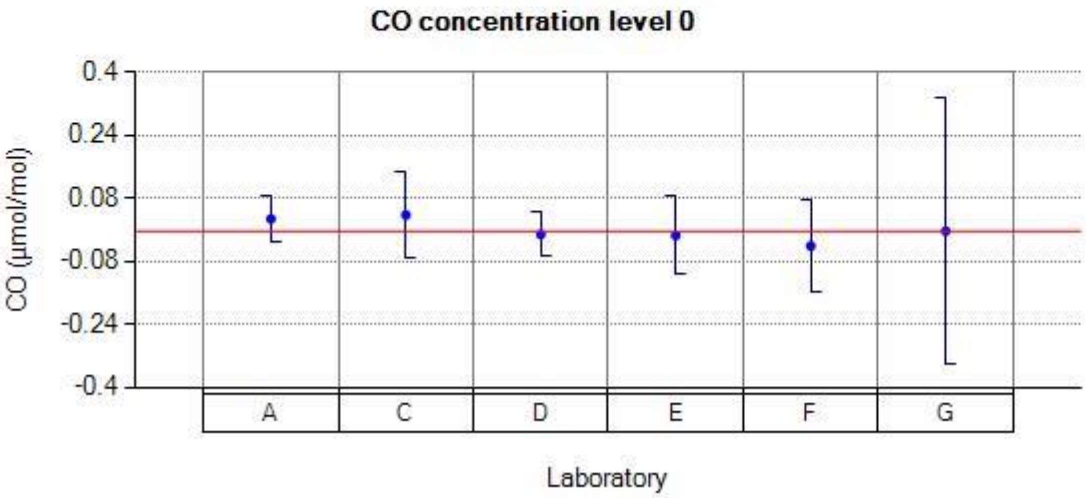
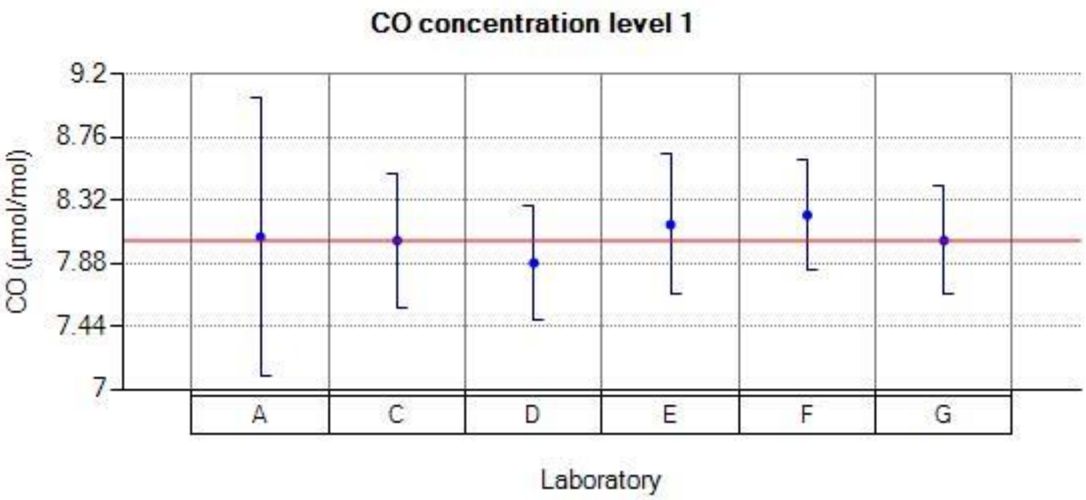


Figure 17: Reported values for CO run 0.

	laboratories					
values	A	C	D	E	F	G
xi, 1	8.063	8.037	7.871	8.142	8.213	8.005
xi, 2	8.065	8.039	7.886	8.153	8.228	8.056
xi, 3	8.065	8.043	7.891	8.158	8.209	8.058
xi	8.064	8.040	7.883	8.151	8.217	8.040
si	0.001	0.003	0.010	0.008	0.010	0.030
u(xi)	0.484	0.233	0.199	0.245	0.194	0.187
U(xi)	0.967	0.466	0.398	0.490	0.388	0.375

Table 16: Reported values for CO run 1.



v,

Figure 18: Reported values for CO run 1.

	laboratories					
values	A	C	D	E	F	G
xi, 1	6.061	6.060	5.924	6.129	6.152	6.049
xi, 2	6.063	6.048	5.924	6.129	6.147	6.050
xi, 3	6.063	6.047	5.925	6.129	6.144	6.050
xi	6.062	6.052	5.924	6.129	6.148	6.050
si	0.001	0.007	0.001	0.000	0.004	0.001
u(xi)	0.373	0.193		0.150	0.150	0.180
U(xi)	0.747	0.386	0.298	0.300	0.300	0.359

Table 17: Reported values for CO run 2.

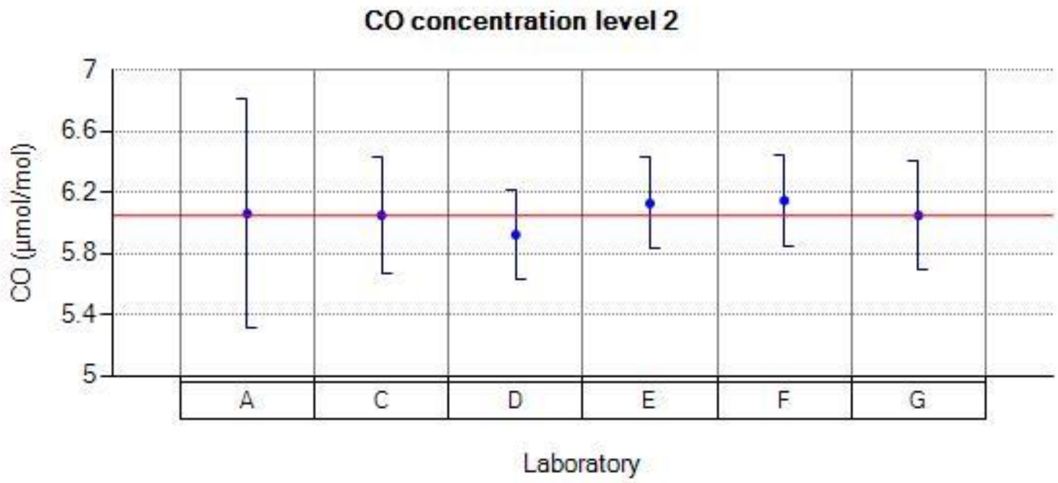


Figure 19: Reported values for CO run 2.

	laboratories					
values	A	C	D	E	F	G
xi, 1	3.012	3.057	2.950	3.060	3.036	3.020
xi, 2	3.013	3.050	2.950	3.060	3.027	3.020
xi, 3	3.011	3.053	2.949	3.059	3.023	3.021
xi	3.012	3.053	2.950	3.060	3.029	3.020
si	0.001	0.004	0.001	0.001	0.007	0.001
u(xi)	0.206	0.106	0.074	0.084	0.090	0.172
U(xi)	0.411	0.211	0.148	0.164	0.180	0.343

Table 18: Reported values for CO run 3.

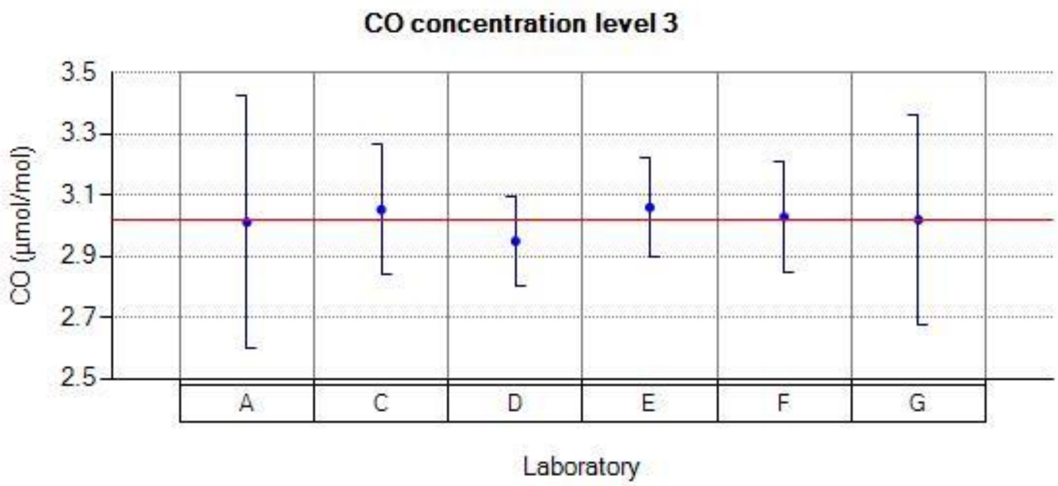


Figure 20: Reported values for CO run 3.

values	laboratories					
	A	C	D	E	F	G
xi, 1	1.017	1.012	0.961	0.998	0.973	0.995
xi, 2	1.017	1.015	0.960	0.998	0.974	0.995
xi, 3	1.015	1.019	0.959	0.999	0.966	0.994
xi	1.016	1.015	0.960	0.998	0.971	0.995
si	0.001	0.004	0.001	0.001	0.004	0.001
u(xi)	0.096	0.063	0.037	0.036	0.062	0.169
U(xi)	0.192	0.126	0.074	0.072	0.124	0.338

Table 19: Reported values for CO run 4.

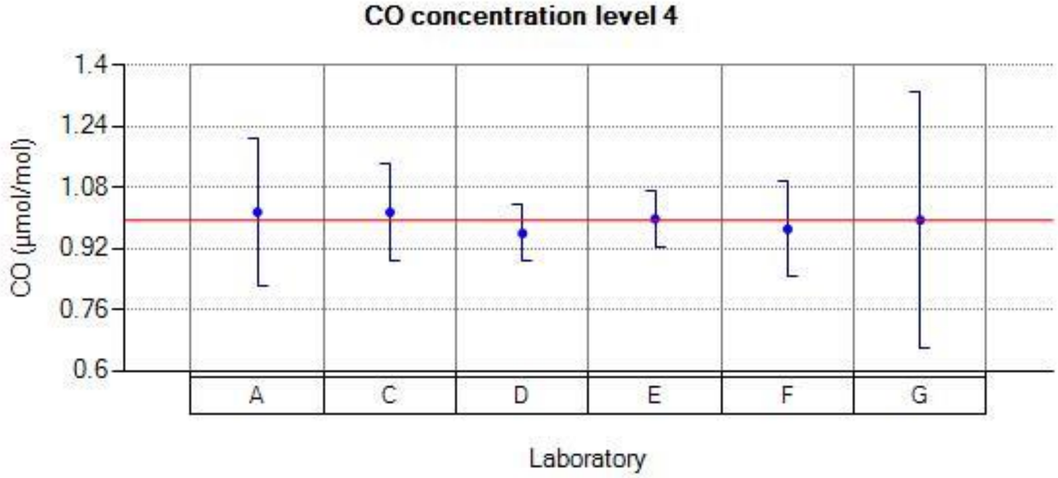


Figure 21: Reported values for CO run 4.

values	laboratories					
	A	C	D	E	F	G
xi, 1	4.376	4.594	4.432	4.590	4.592	4.535
xi, 2	4.376	4.598	4.436	4.590	4.593	4.537
xi, 3	4.377	4.599	4.437	4.590	4.581	4.537
xi	4.376	4.597	4.435	4.590	4.589	4.536
si	0.001	0.003	0.003	0.000	0.007	0.001
u(xi)	0.281	0.142	0.111	0.121	0.119	0.175
U(xi)	0.561	0.284	0.223	0.142	0.238	0.350

Table 20: Reported values for CO run 5.

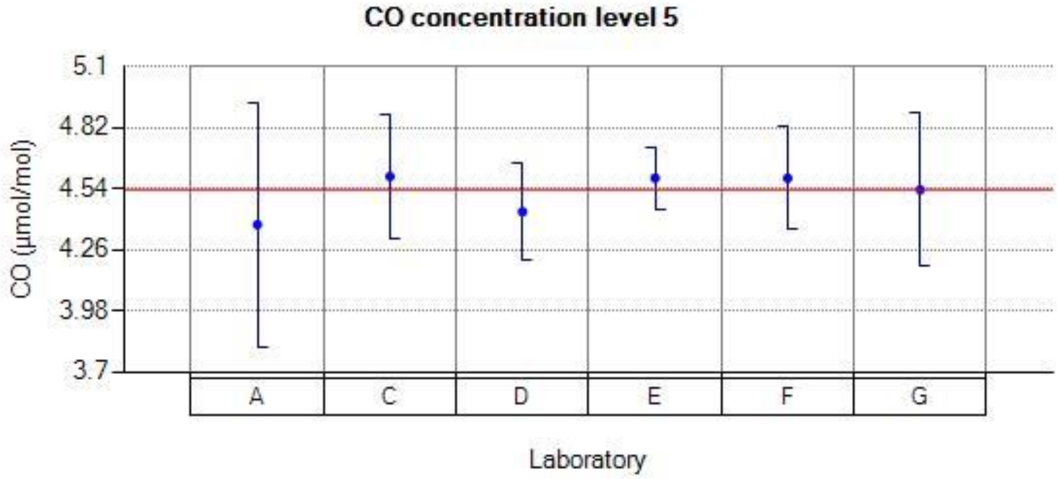


Figure 22: Reported values for CO run 5.

Reported values for O_3

	laboratories						
values	A	B	C	D	E	F	G
xi, 1	0.35	0.00	0.01	0.52	-0.04	0.46	0.20
u(xi)	0.90	0.00	1.00	0.32	0.31	0.58	0.68
U(xi)	1.80	0.00	2.00	0.64	0.52	1.16	1.36

Table 21: Reported values for O_3 run 0.

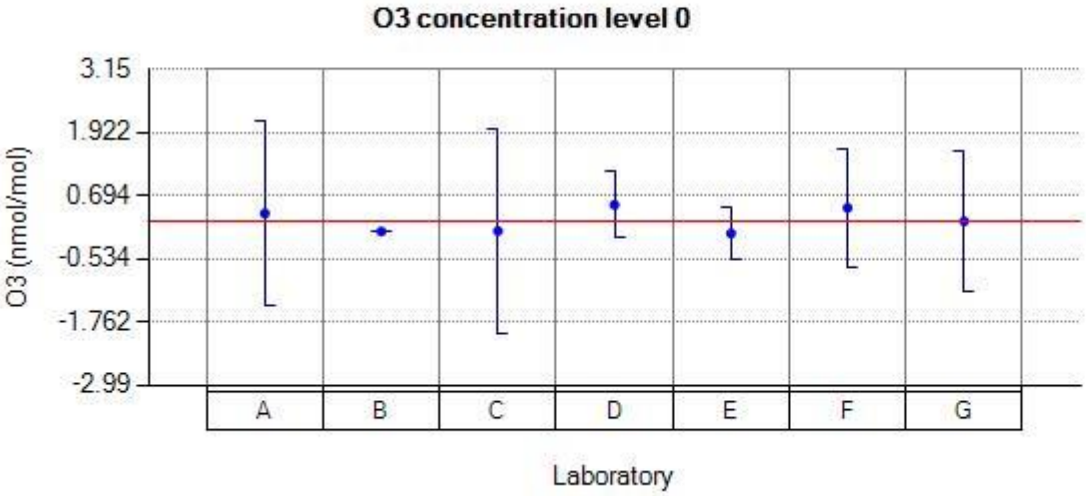


Figure 23: Reported values for O_3 run 0.

	laboratories						
values	A	B	C	D	E	F	G
xi, 1	305.45	298.53	313.05	313.27	290.17	303.70	309.10
xi, 2	305.73	316.43	316.77	322.53	300.02	307.53	311.50
xi, 3	305.87	294.16	320.57	327.73	303.48	310.54	313.97
xi	305.68	303.04	316.79	321.17	297.89	307.25	311.52
si	0.21	11.80	3.76	7.32	6.90	3.42	2.43
u(xi)	13.74	11.96	4.39	11.59	3.01	4.88	3.47
U(xi)	27.48	38.05	8.79	23.18	6.02	9.76	6.94

Table 22: Reported values for O_3 run 1

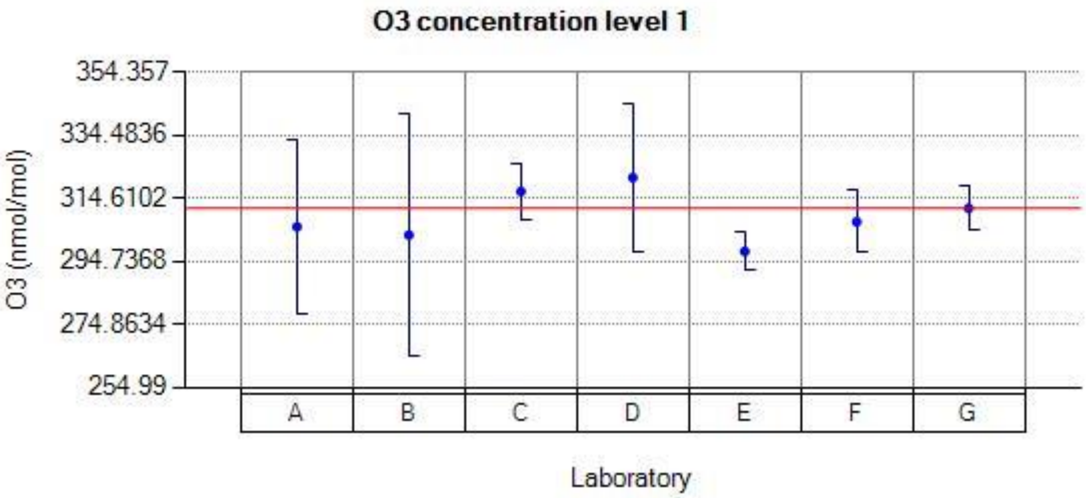


Figure 24: Reported values for O_3 run 1.

values	laboratories						
	A	B	C	D	E	F	G
xi, 1	104.05	92.36	106.48	109.47	101.19	102.92	103.50
xi, 2	104.14	98.97	105.78	109.07	100.86	102.21	103.04
xi, 3	104.19	83.54	104.85	108.81	101.53	101.62	102.70
xi	104.12	91.62	105.70	109.11	101.19	102.25	103.08
si	0.07	7.74	0.81	0.33	0.33	0.65	0.40
u(xi)	5.27	7.90	2.49	3.05	1.32	1.71	1.43
U(xi)	10.55	25.14	4.98	6.11	2.64	3.42	2.86

Table 23: Reported values for O_3 run 2.

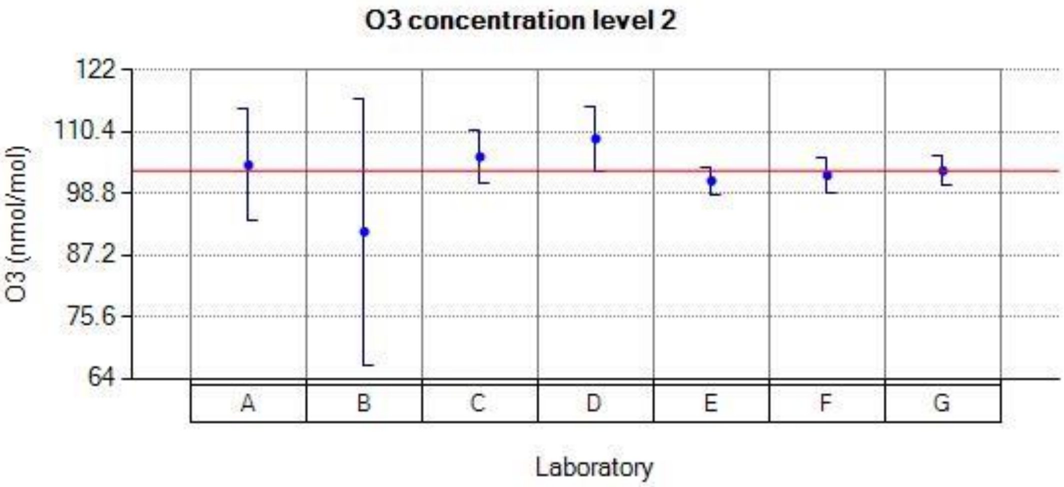


Figure 25: Reported values for O_3 run 2.

values	laboratories						
	A	B	C	D	E	F	G
xi, 1	63.00	69.66	63.30	66.20	62.58	61.79	62.20
xi, 2	63.06	63.57	63.13	65.00	62.20	61.66	62.29
xi, 3	63.09	61.61	63.11	65.00	62.28	61.69	62.26
xi	63.05	64.94	63.18	65.40	62.35	61.71	62.25
si	0.04	4.19	0.10	0.69	0.20	0.06	0.04
u(xi)	3.55	4.35	1.98	1.79	1.02	1.13	1.14
U(xi)	7.10	13.85	3.96	3.59	2.04	2.26	2.28

Table 24: Reported values for O_3 run 3.

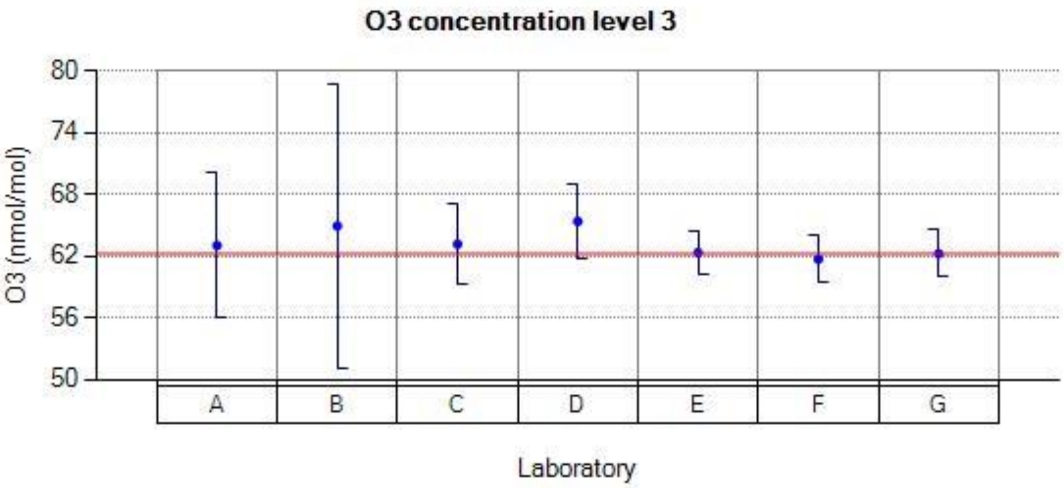


Figure 26: Reported values for O_3 run 3.

	laboratories						
values	A	B	C	D	E	F	G
xi, 1	21.38	18.18	21.54	22.67	22.54	21.57	21.32
xi, 2	21.10	21.53	21.50	22.00	22.47	21.39	21.31
xi, 3	21.10	19.82	21.49	22.75	22.40	21.45	21.28
xi	21.19	19.84	21.51	22.47	22.47	21.47	21.30
si	0.16	1.67	0.02	0.41	0.07	0.09	0.02
u(xi)	1.79	1.83	1.30	0.62	0.68	0.67	0.93
U(xi)	3.58	5.83	2.60	1.24	1.36	1.34	1.86

Table 25: Reported values for O_3 run 4.

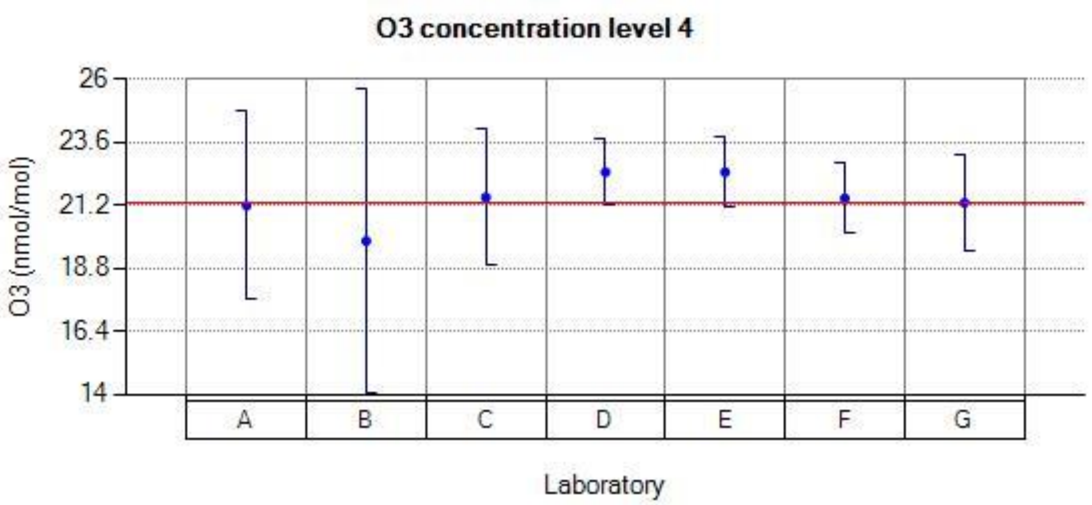


Figure 27: Reported values for O_3 run 4.

Reported values for NO

	laboratories						
values	A	B	C	D	E	F	G
xi, 1	0.20	0.00	0.02	0.47	0.04	-0.29	0.12
u(xi)	0.72	0.00	0.75	0.43	0.42	0.62	0.89
U(xi)	1.45	0.00	1.50	0.86	0.84	1.24	1.79

Table 26: Reported values for NO run 0.

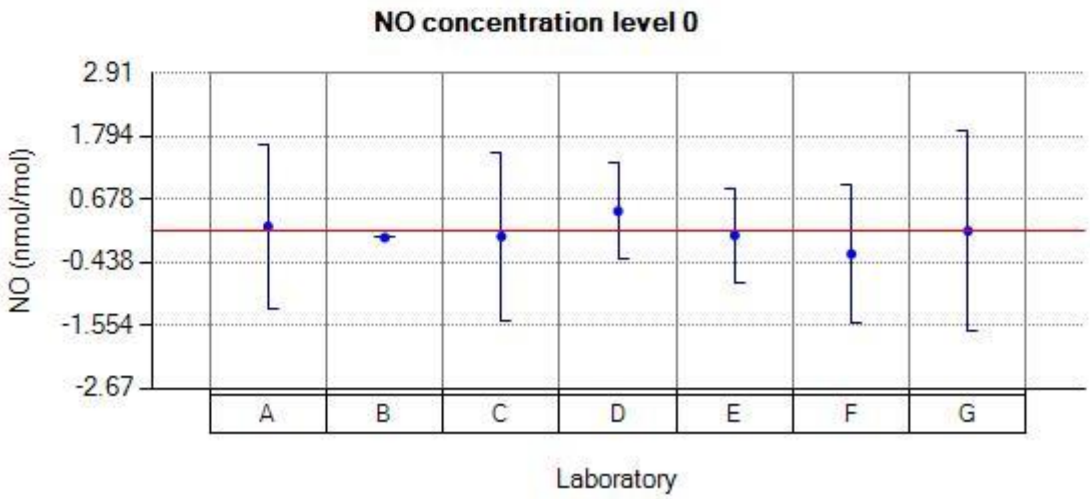


Figure 28: Reported values for NO run 0.

	laboratories						
values	A	B	C	D	E	F	G
xi, 1	198.45	196.92	199.52	200.35	195.64	206.40	197.53
xi, 2	197.77	221.00	198.86	199.71	195.89	207.65	197.31
xi, 3	198.03	217.29	198.38	199.83	195.86	208.57	197.56
xi	198.08	211.73	198.92	199.96	195.79	207.54	197.46
si	0.34	12.96	0.57	0.34	0.13	1.08	0.13
u(xi)	7.47	13.12	4.21	6.07	4.92	5.47	2.85
U(xi)	14.95	41.74	8.42	12.15	9.84	10.94	5.71

Table 27: Reported values for NO run 1.

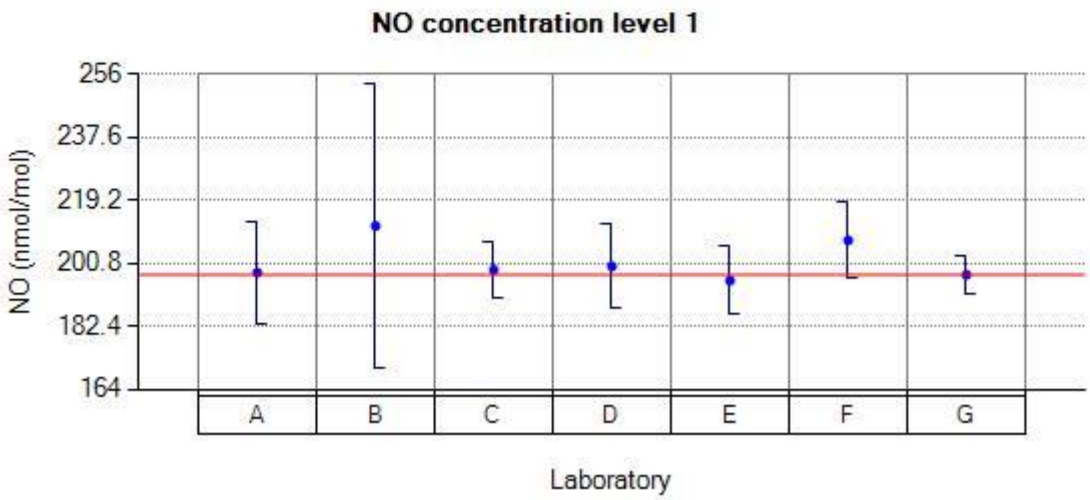


Figure 29: Reported values for NO run 1.

	laboratories						
values	A	B	C	D	E	F	G
xi, 1	19.80	18.56	17.96	19.96	19.09	18.96	18.96
xi, 2	19.60	19.33	17.81	19.64	19.00	19.26	18.96
xi, 3	19.83	23.73	17.83	19.43	18.95	18.99	18.96
xi	19.74	20.54	17.86	19.67	19.01	19.07	18.96
si	0.12	2.78	0.08	0.26	0.07	0.16	0.00
u(xi)	1.38	2.94	0.84	0.53	1.20	0.83	2.18
U(xi)	2.75	9.36	1.69	1.05	2.40	1.66	4.37

Table 28: Reported values for NO run 2.

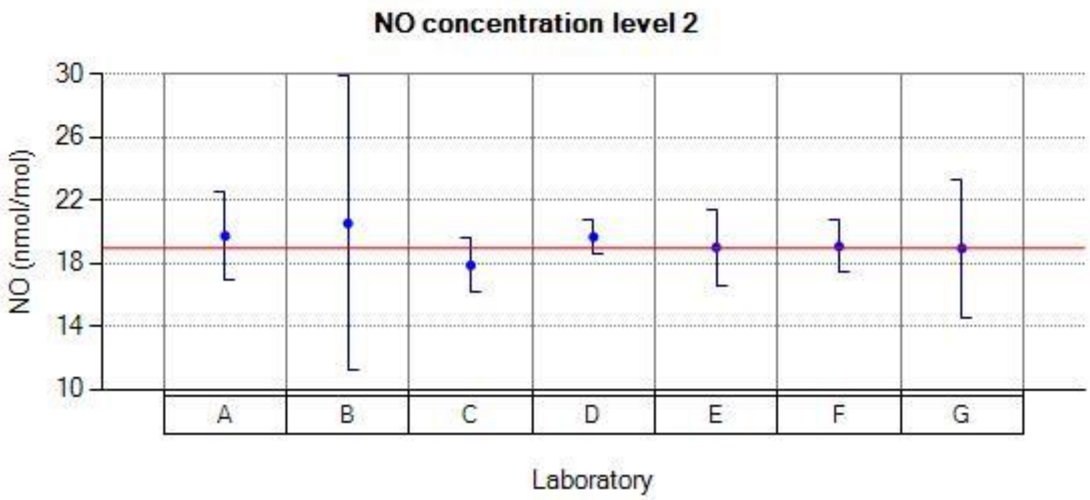


Figure 30: Reported values for NO run 2.

Reported values for NO_2

	laboratories						
values	A	B	C	D	E	F	G
xi, 1	0.14	0.00	0.07	-0.54	-0.19	-0.07	0.15
u(xi)	1.02	0.00	1.00	0.55	0.54	0.59	0.89
U(xi)	2.04	0.00	2.00	1.10	1.08	1.18	1.79

Table 29: Reported values for NO_2 run 0.

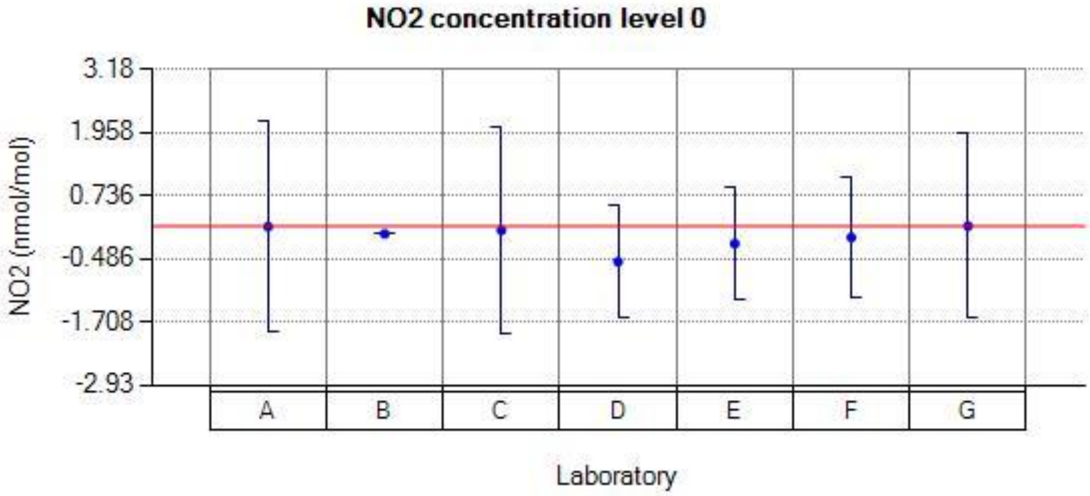


Figure 31: Reported values for NO_2 run 0.

	laboratories						
values	A	B	C	D	E	F	G
xi, 1	199.17	205.72	196.95	199.07	197.11	191.77	198.26
xi, 2	200.84	194.36	199.42	199.06	197.00	193.55	198.28
xi, 3	201.37	213.21	202.11	199.45	197.16	194.72	199.38
xi	200.46	204.43	199.49	199.19	197.09	193.34	198.64
si	1.14	9.49	2.58	0.22	0.08	1.48	0.64
u(xi)	10.60	9.66	5.17	6.04	4.62	4.19	3.27
U(xi)	21.21	30.72	10.34	12.09	9.24	8.38	6.54

Table 30: Reported values for NO_2 run 1.

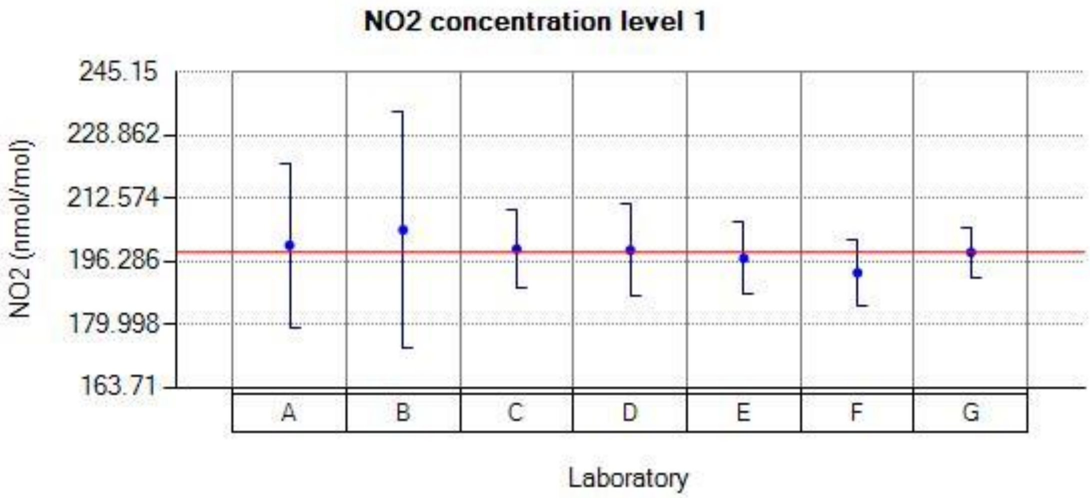


Figure 32: Reported values for NO_2 run 1.

	laboratories						
values	A	B	C	D	E	F	G
xi, 1	101.26	97.64	99.35	100.01	99.67	95.57	100.72
xi, 2	100.81	108.98	99.36	99.73	99.61	95.32	100.66
xi, 3	100.80	110.37	99.42	99.54	99.49	95.85	100.77
xi	100.95	105.66	99.37	99.76	99.59	95.58	100.71
si	0.26	6.98	0.03	0.23	0.09	0.26	0.05
u(xi)	5.59	6.76	2.50	2.76	2.83	2.13	2.49
U(xi)	11.19	21.50	5.00	5.53	5.66	4.26	4.99

Table 31: Reported values for NO_2 run 2.

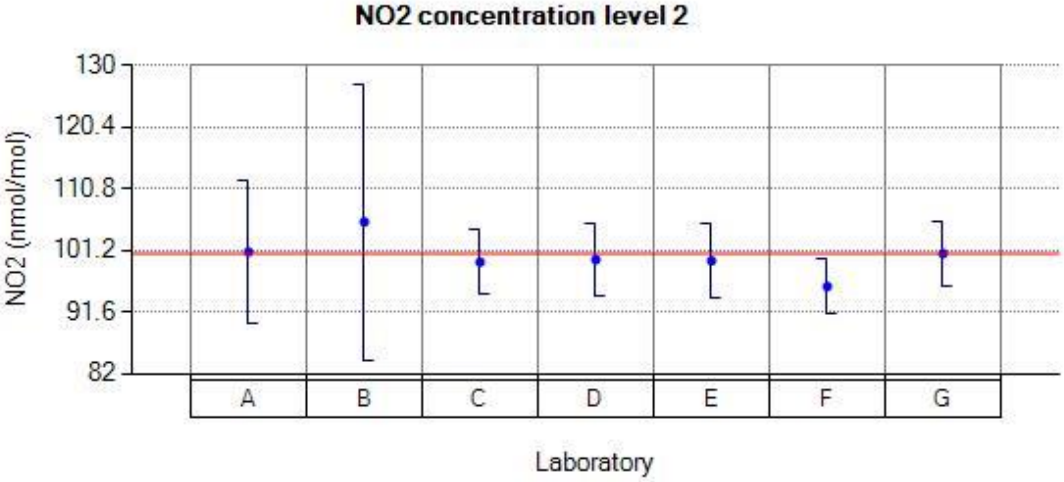


Figure 33: Reported values for NO_2 run 2.

	laboratories						
values	A	B	C	D	E	F	G
xi, 1	61.33	67.40	59.06	60.05	60.47	56.20	61.44
xi, 2	61.24	59.25	58.83	59.71	60.44	56.23	61.26
xi, 3	61.08	56.26	58.93	59.73	60.26	55.74	61.13
xi	61.21	60.97	58.94	59.83	60.39	56.05	61.27
si	0.12	5.76	0.11	0.19	0.11	0.27	0.15
u(xi)	3.59	5.92	1.63	1.60	2.12	1.37	2.30
U(xi)	7.18	18.84	3.26	3.19	4.24	2.74	4.59

Figure 34: Reported values for NO_2 run 3.

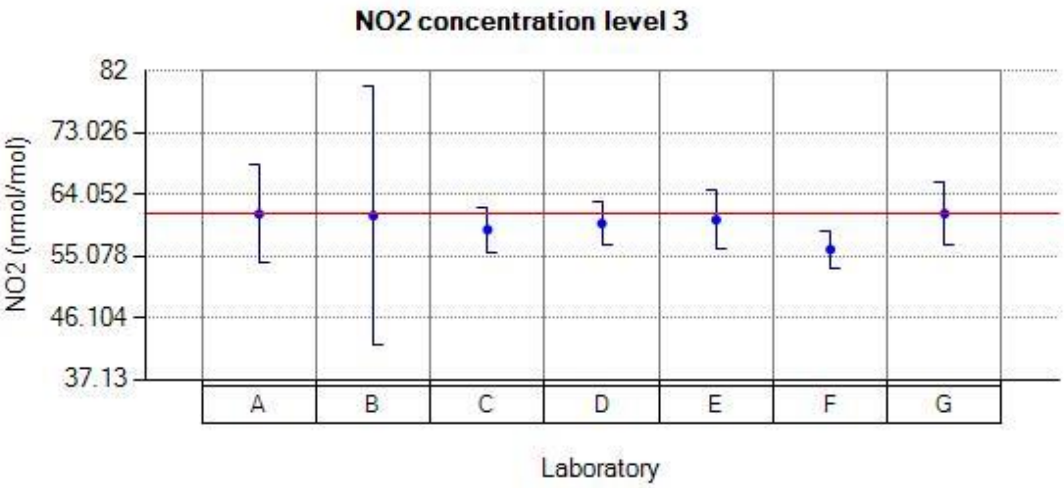


Figure 35: Reported values for NO_2 run 3.

	laboratories						
values	A	B	C	D	E	F	G
xi, 1	21.19	18.97	20.27	20.42	21.66	17.89	21.71
xi, 2	21.05	16.38	19.78	20.09	21.28	17.84	21.47
xi, 3	20.70	22.13	19.56	19.99	21.23	17.80	21.44
xi	20.98	19.16	19.87	20.16	21.39	17.84	21.54
si	0.25	2.88	0.36	0.22	0.23	0.04	0.14
u(xi)	1.57	3.04	1.21	0.93	0.62	0.72	2.19
U(xi)	3.13	9.67	2.42	1.86	1.24	1.44	4.38

Figure 36: Reported values for NO_2 run 4.

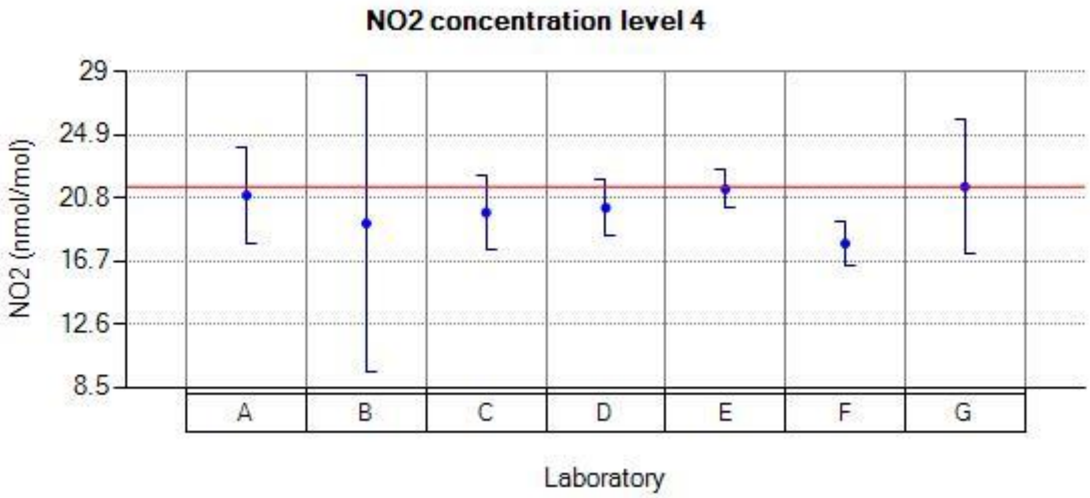


Figure 37: Reported values for NO_2 run 4.

Annex C. The precision of standardized measurement methods

For the main purpose of monitoring trends between different IE the precision of standardized SO_2, CO, O_3 and NO_X measurement methods [2], [3], [4] and [5] as implemented by NRLs was evaluated. The applied methodology is described in ISO 5725-part 1 [14], part 2 [15] and part 6 [16].

The precision experiment has involved a total of 7 laboratories the actual number of labs (p_j) varying from run to run (Table 32). Laboratory B didn't reported results for CO. For run 0 was requested only one value so repeatability cannot be evaluated. Five concentration levels were tested for CO, four levels for O_3, SO_2 and NO_2, and two for NO. Outlier tests were performed and results are reported in Annex D.
The repeatability standard deviation (s_r) was calculated in accordance with ISO 5725-2 as the square root of average within laboratory variance. The repeatability limit (r) is calculated using Equation 5 [16]. It represents the biggest difference between two test results found on an identical test gas by one laboratory using the same apparatus within the shortest feasible time interval.

$$r = t_{95\%,\nu} \cdot \sqrt{2} \cdot s_r$$ **Equation 5**

The reproducibility standard deviation (s_R) was calculated in accordance with ISO 5725-6 as the square root of sum of repeatability and between-laboratory variance. The reproducibility limit (R) is calculated using Equation 6 [16]. It represents the biggest difference between two measurements on an identical test gas reported by two laboratories.

$$R = t_{95\%,\nu} \cdot \sqrt{2} \cdot s_R$$ **Equation 6**

The repeatability standard deviation was evaluated with (p_j *(3-1)) degrees of freedom (ν) and reproducibility standard deviation with (p_j-1) degrees of freedom. The critical range student factors ($t_{\alpha,\nu}$) are reported in Table 32.

parameter	run	p_j	t critical value 95% for r	t critical value 95% for R
CO	1,2,3,4,5	6	2.179	2.571
NO	1,2	7	2.145	2.447
NO_2	1,2,3,4	7	2.145	2.447
O_3	1,2,3,4	7	2.145	2.447
SO_2	1,2,3,4	7	2.145	2.447

Table 32: Critical values of t used in the repeatability (r) and reproducibility (R) evaluation.

The repeatability (r) and reproducibility (R) limits of measurement methods are presented from Table 33 to Table 37 and from Figure 38 to Figure 42. It is also reported the 'reproducibility from common criteria (R (from σ_p))' calculated by substituting s_R in Equation 6 with a 'standard deviation for proficiency assessment' (Table 4). Comparison between R and R (from σ_p) serves to indicate that σ_p is realistic ([13] 6.3.1) or from the other point of view, that the general methodology implemented by NRLs is appropriate for σ_p. The green (R) and blue (r) line are representing a good performance if they run below the red line that represents the data quality objective of the IE.

NO data (nmol/mol) without outliers			
group average	repeatability limit : r	reproducibility limit : R	reproducibility limit (relative)
0.1		0.8	
19.3	3.2	4.2	
201.4	14.9	24.8	12.3%

Table 33: The R and r of NO standard measurement method.

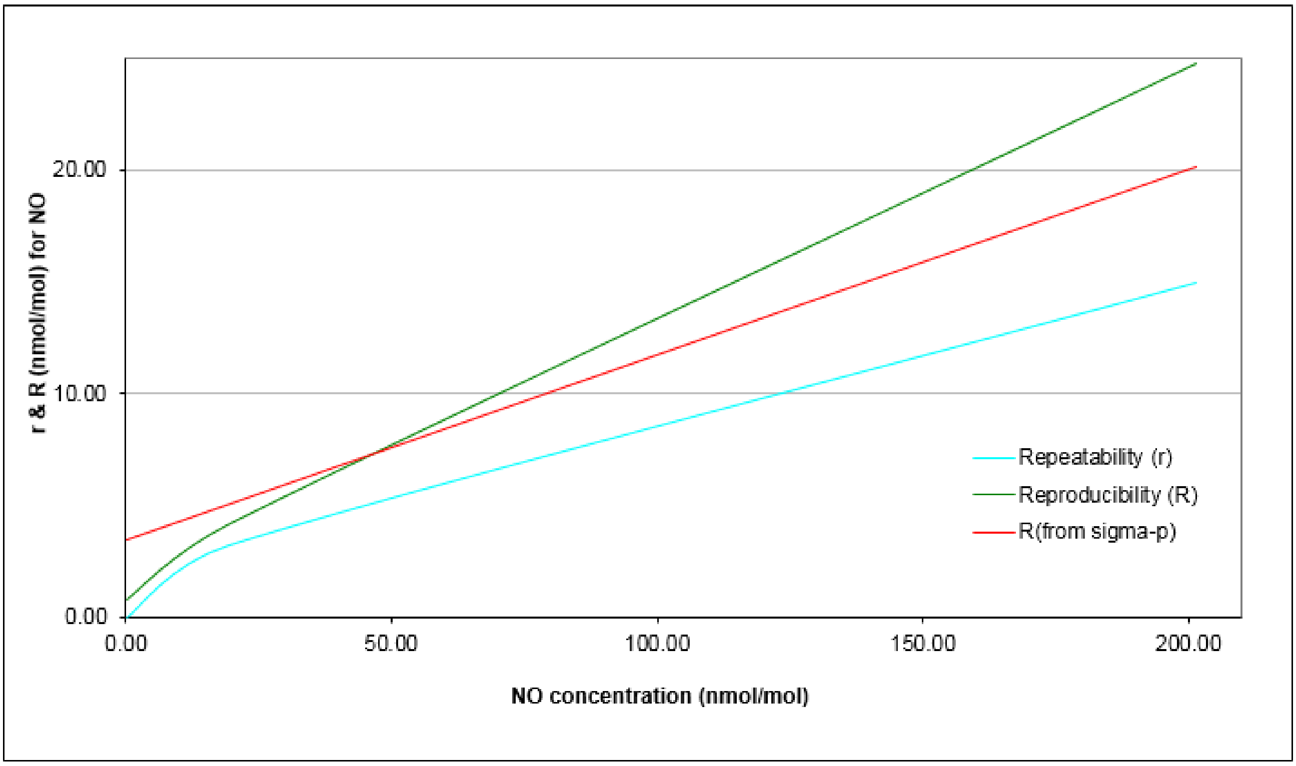


Figure 38: The R and r of NO standard measurement method as a function of concentration.

NO_2 data (µmol/mol) without outliers			
group average	repeatability limit: r	reproducibility limit: R	reproducibility limit (relative)
-0.1		0.8	
20.1	3.4	5.6	
59.8	6.6	8.9	
100.2	8.0	12.7	
199.0	11.5	15.8	7.9%

Table 34: The R and r of NO_2 standard measurement method.

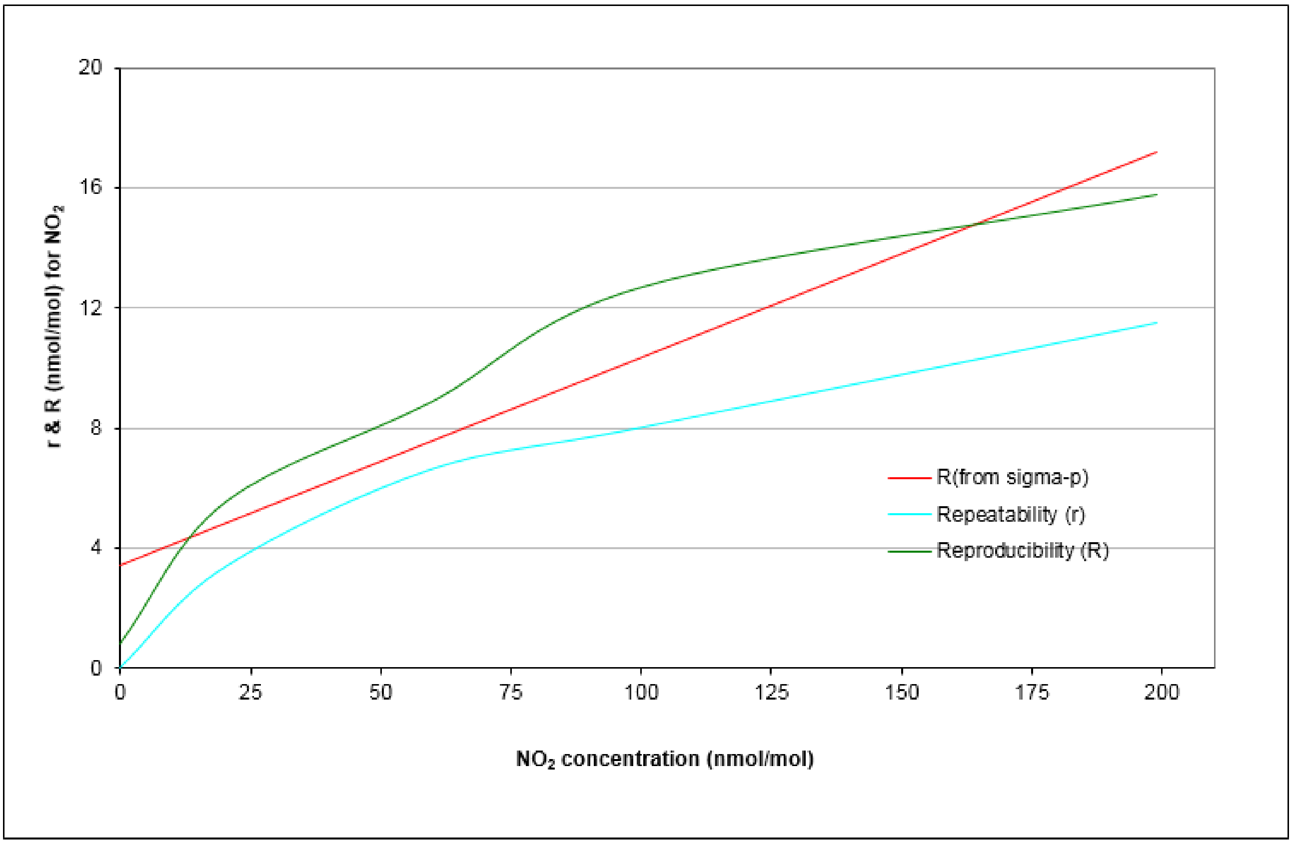


Figure 39: The R and r of NO_2 standard measurement method as a function of concentration.

SO_2 data (nmol/mol) without outliers			
group average	repeatability limit: r	reproducibility limit: R	reproducibility limit (relative)
0		0	
5.1	0.8	1.6	
20.3	3.1	3.2	
46.1	4.4	5.1	
133	9	13.1	13.4%

Table 35: The R and r of SO_2 standard measurement method.

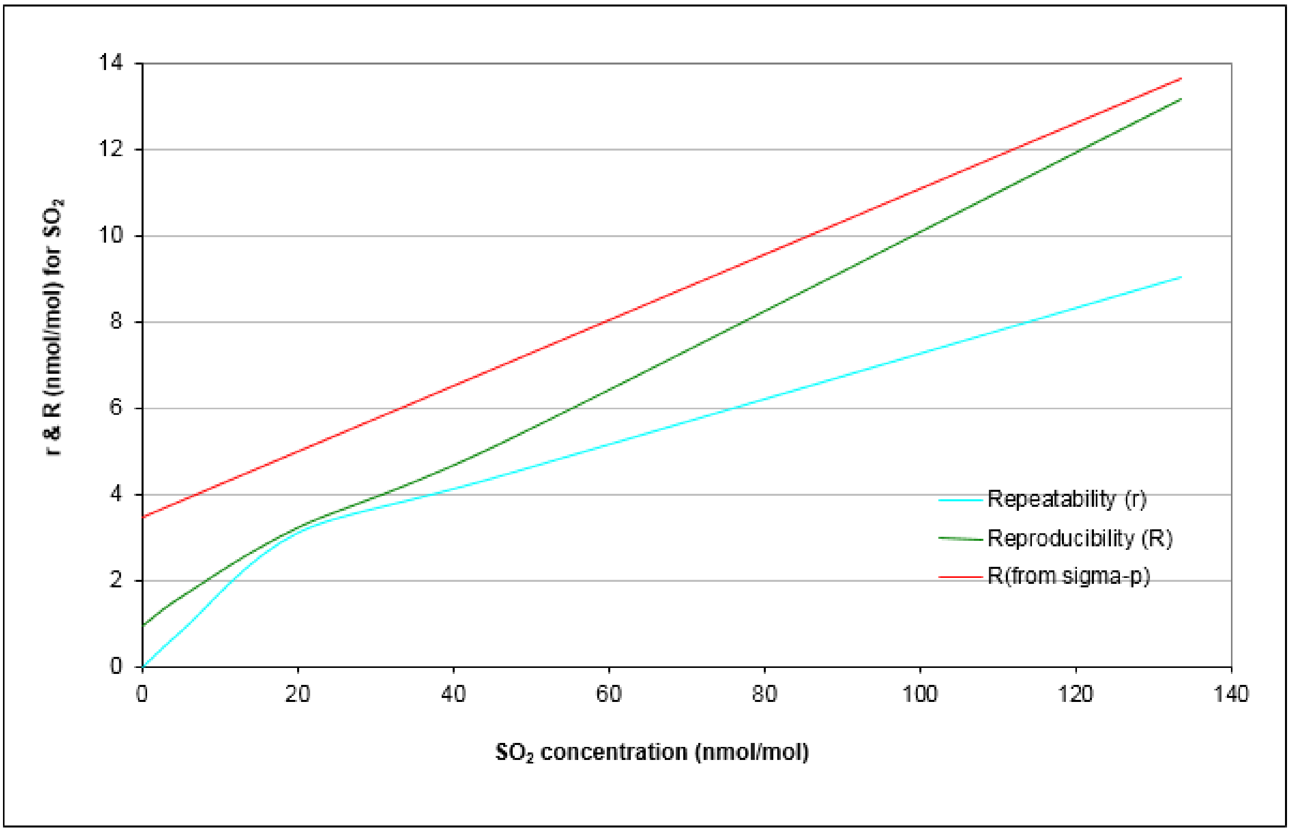


Figure 40: The R and r of SO_2 standard measurement method as a function of concentration.

CO data (nmol/mol) without outliers			
group average	repeatability limit: r	reproducibility limit: R	reproducibility limit (relative)
-0.001		0.106	
0.993	0.008	0.084	
3.021	0.01	0.144	
4.521	0.01	0.34	
6.061	0.011	0.287	
8.066	0.044	0.417	5.2%

Table 36: The R and r of CO standard measurement method.

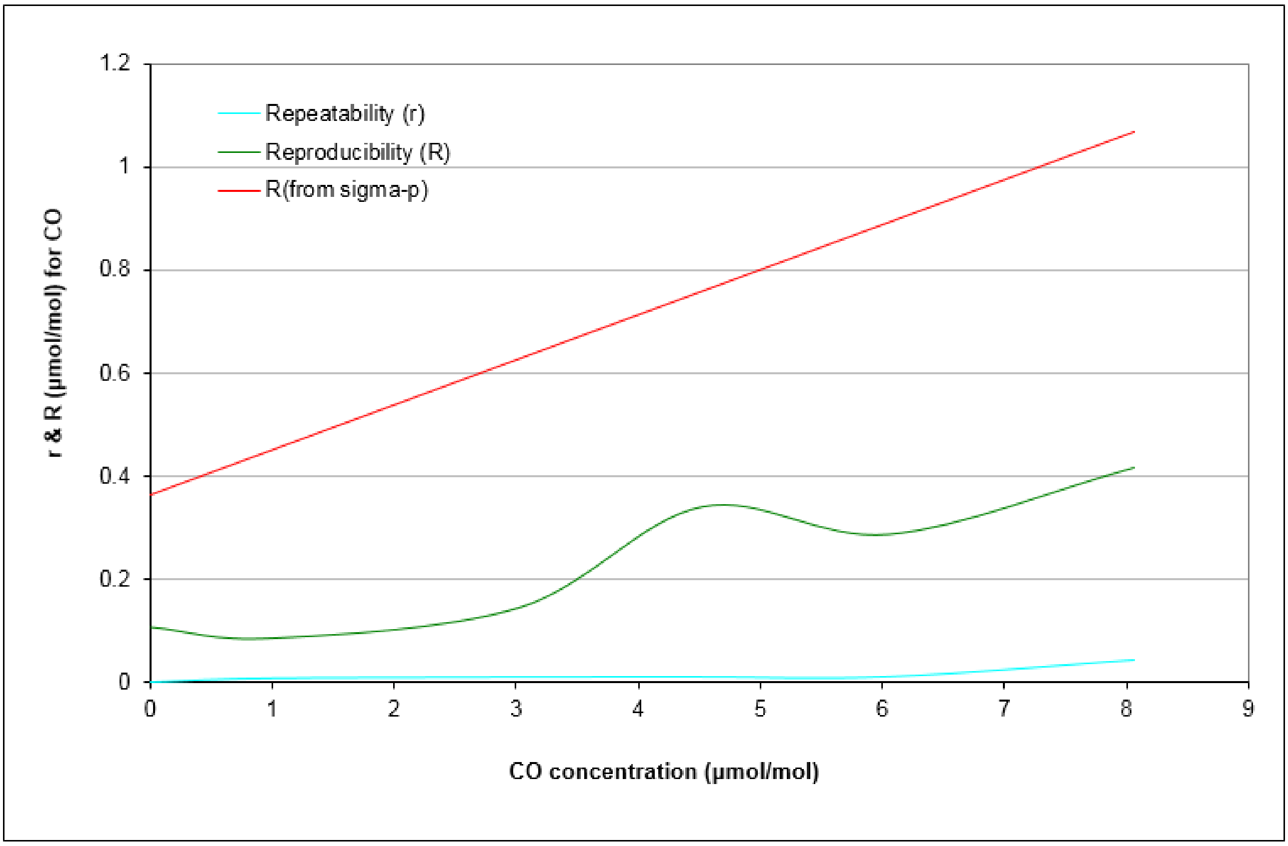


Figure 41: The R and r of CO standard measurement method as a function of concentration.

O_3 data (nmol/mol) without outliers			
group average	repeatability limit: r	reproducibility limit: R	reproducibility limit (relative)
0.2		0.8	
21.5	2	3.6	
63.3	4.9	6.6	
102.4	9	20.6	
309.1	18.9	33	10.7%

Table 37: The R and r of O_3 standard measurement method.

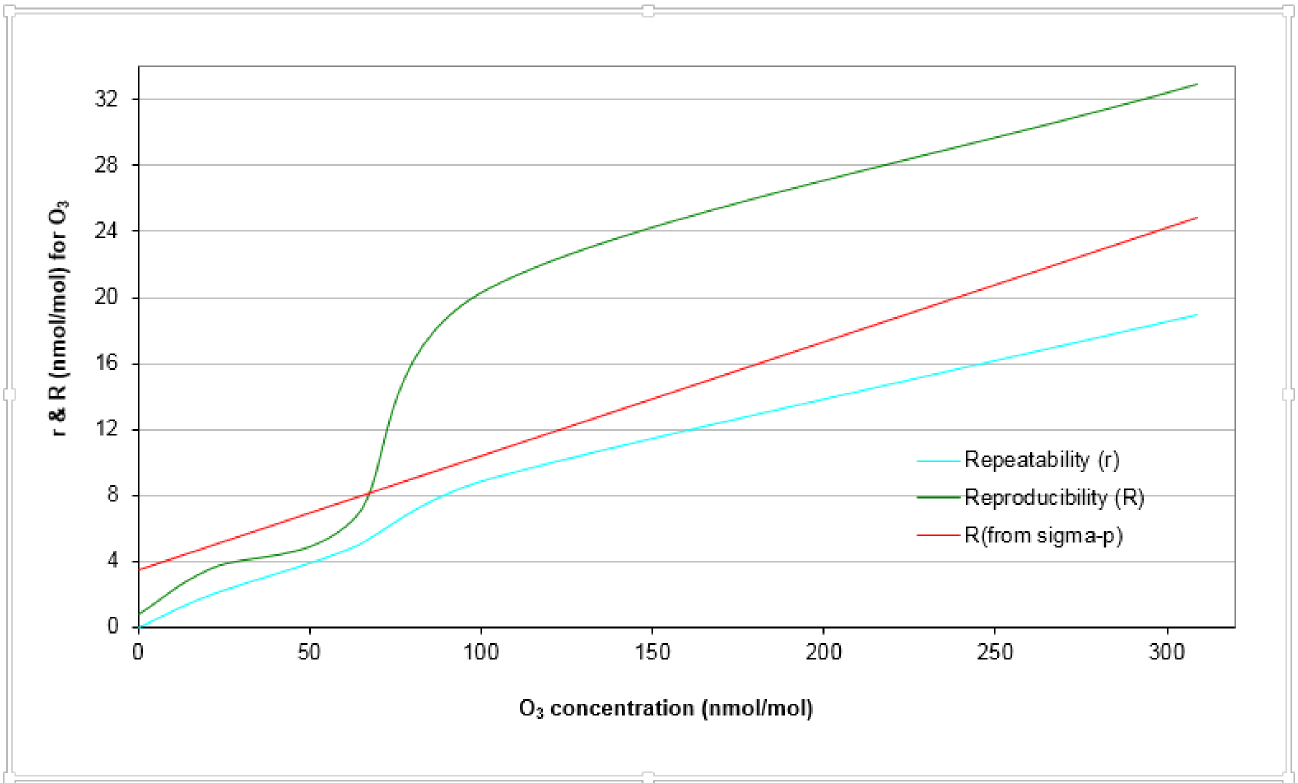


Figure 42: The R and r of O_3 standard measurement method as a function of concentration.

Annex D. Result analysis for consistency and outlier test

The precision evaluation (Annex C) focuses on data that are as much as possible the reflection of every day work of NRLs and thus represents the comparability of participant's standard operating procedures.
For that reason a procedure for the detection of exceptional errors (error during typing, slip in performing the measurement or the calculation, wrong averaging interval, malfunction of instrumentation, etc.) was applied. In this procedure were carried out tests for data consistency and statistical outliers as described in ISO 5725-2.
Laboratories showing some form of statistical inconsistency were requested to investigate the cause of discrepancies.
Laboratories were allowed to correct their results in case of identification of exceptional errors. Subsequently, data were considered definitive and "Grubb's one outlying observation test" was performed.
For runs where outliers were detected, outliers were removed and "Grubb's one outlying observation test" was repeated until no more outliers were observed. Statistical outliers obtained at this stage are not considered as due to extraordinary errors but due to significant difference in participant's standard operating procedure.

During this IE the statistical outliers presented in the table below are related to two results for SO2 and one of them is related to a zero level.

Laboratory	parameter	run	value	Gmax_1%	Gmax_5%
B	SO2	4	5.94	Not OK	Not OK
D	SO2	0	0.75	Not OK	Not OK

Table 38: "Genuine" statistical outliers according to Grubb's one outlying observation test.

The precision of standardized measurement methods reported in Annex C are calculated using the database without outliers.

According to Grubb's test results between a confidence level of 1 and 5% are considered straggler and they deserve a specific check.
In order to give useful information to the participants for judging their performance also the stragglers are reported in the following table:

Laboratory	parameter	run	value	Gmin_1%	Gmin_5%
F	NO2	3	65.67	OK	straggler

Table 39: Stragglers according to Grubb's one observation test.

Annex E. Laboratory accreditation certificate

In this annex is shown the accreditation certificate of the laboratory who organized this Inter-laboratory comparison and delivered the assigned value.

DAkkS
Deutsche
Akkreditierungsstelle

Deutsche Akkreditierungsstelle GmbH

Beliehene gemäß § 8 Absatz 1 AkkStelleG i.V.m. § 1 Absatz 1 AkkStelleGBV
Unterzeichnerin der Multilateralen Abkommen
von EA, ILAC und IAF zur gegenseitigen Anerkennung

Akkreditierung

Die Deutsche Akkreditierungsstelle GmbH bestätigt hiermit, dass das Prüflaboratorium

Umweltbundesamt
FG II 4.4 Experimentelle Untersuchungen zur Luftgüte -
Nationales EU-Luftqualitätsreferenzlabor
Paul-Ehrlich-Straße 29, 63225 Langen/Hessen

die Kompetenz nach DIN EN ISO/IEC 17025:2005 besitzt, Prüfungen in folgenden Bereichen durchzuführen:

physikalisch-chemische Untersuchungen von Luft

Die Akkreditierungsurkunde gilt nur in Verbindung mit dem Bescheid vom 09.12.2014 mit der Akkreditierungsnummer D-PL-14454-02 und ist gültig bis 08.12.2019. Sie besteht aus diesem Deckblatt, der Rückseite des Deckblatts und der folgenden Anlage mit insgesamt 2 Seiten.

Registrierungsnummer der Urkunde: **D-PL-14454-02-00**

Im Auftrag

Andrea Valbuena
Abteilungsleiterin

Berlin, 09.12.2014

Siehe Hinweise auf der Rückseite

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Die Akkreditierung erfolgte gemäß des Gesetzes über die Akkreditierungsstelle (AkkStelleG) vom 31. Juli 2009 (BGBl. I S. 2625) sowie der Verordnung (EG) Nr. 765/2008 des Europäischen Parlaments und des Rates vom 9. Juli 2008 über die Vorschriften für die Akkreditierung und Marktüberwachung im Zusammenhang mit der Vermarktung von Produkten (Abl. L 218 vom 9. Juli 2008, S. 30).
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Umweltbundesamt
FG II 4.4 Experimentelle Untersuchungen zur Luftgüte -
Nationales EU-Luftqualitätsreferenzlabor
Paul-Ehrlich-Straße 29, 63225 Langen/Hessen

Prüfungen in den Bereichen:

physikalisch-chemische Untersuchungen von Luft

verwendete Abkürzungen: siehe letzte Seite

Dem Laboratorium ist, ohne dass es einer vorherigen Information und Zustimmung der DAkkS bedarf, die Anwendung der hier aufgeführten genormten Prüfverfahren mit unterschiedlichen Ausgabeständen der Normen gestattet.

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DAkkS Deutsche Akkreditierungsstelle

Anlage zur Akkreditierungsurkunde D-PL-14454-02-00

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DIN EN 14212 2005-06	Luftqualität - Messverfahren zur Bestimmung der Konzentration von Schwefeldioxid mit Ultraviolett-Fluoreszenz (Abweichung: *Anwendung auf Prüfgase*) (*zurückgezogene Norm*)
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DIN EN 14626 2005-07	Luftqualität - Messverfahren zur Bestimmung der Konzentration von Kohlenmonoxid mit nicht-dispersiver Infrarot-Photometrie (Abweichung: *Anwendung auf Prüfgase*) (*zurückgezogene Norm*)
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verwendete Abkürzungen:

DIN	Deutsches Institut für Normung e. V.
EN	Europäische Norm
IEC	International Electrotechnical Commission
ISO	International Organization für Standardization

Gültigkeitsdauer: 09.12.2014 bis 08.12.2019 **Ausstellungsdatum: 09.12.2014** **Seite 2 von 2**

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