

Annual Report 2024

Results of stack emission proficiency tests for substance ranges P, G, and O on the emission simulation apparatus in the year 2024

HLNUG Department I3 is accredited for performing testing services according to DIN EN ISO/IEC 17043. The accreditation is valid for the testing procedures listed in the certificate.



Jede Veröffentlichung oder Vervielfältigung (im Ganzen oder in Auszügen) bedarf der vorherigen schriftlichen Genehmigung durch das Hessische Landesamt für Naturschutz, Umwelt und Geologie.

Any publication or reproduction (in whole or in part) requires the prior written permission by Hessisches Landesamt für Naturschutz, Umwelt und Geologie.

Contents

0.	About this Report	4
1.	Summary	4
2.	Introduction	4
2.1	Legal Background.....	4
2.2	The Emission Simulation Apparatus.....	5
3.	Organisational Information	6
4.	Execution of the Proficiency Tests	8
4.1	Description of the Test Objects	8
4.2	Preparation of the Test Objects.....	8
4.3	Metrological Traceability	9
4.4	Execution of the Measurements	9
4.5	Evaluation of the Proficiency Tests	10
4.5.1	Calculation of z-Scores.....	10
4.5.2	Criteria for Proficiency Assessment.....	11
4.5.3	Assessment Scheme.....	12
4.5.4	Communication of the Assessment Result.....	14
5.	Results	14
5.1	z-Scores	14
5.1.1	Dust Proficiency Test (Substance Range P).....	15
5.1.2	Gas Proficiency Test (Substance Range G)	19
5.1.3	Odour Proficiency Test (Substance Range O).....	23
5.1.4	Gas flow conditions.....	25
5.2	Sums of Class Numbers	27
5.2.1	Dust Proficiency Test (Substance Range P).....	28
5.2.2	Gas Proficiency Test (Substance Range G)	29
5.2.3	Odour Proficiency Test (Substance Range O).....	30
5.3	Theory Test.....	31
6.	Interpretation of Results	32
6.1	§29b Measuring Bodies.....	34

6.2	Voluntary Participants	37
6.3	Gas Flow Conditions	37
7.	Optional Information from Participants.....	38
7.1	Probes and Rinsing Procedures in Dust Sampling	38
7.2	Diameter of the Nozzle Opening in Dust Samplings	41
7.3	Analytical Instruments for Heavy Metals.....	42
7.4	Chemicals in the Digestion Solution	44
7.5	Solvents for Desorption of ETX.....	46
7.6	Gas Chromatography Detectors.....	47
7.7	Sulphur Dioxide.....	48
7.8	Formaldehyde.....	49
7.9	Feedback from Participants	50
8.	Concluding Remark	52
9.	References	53

0. About this Report

This report is a translation of „Jahresbericht 2024 – Ergebnisse der Emissionsringversuche der Stoffbereiche P, G und O an der Emissionssimulationsanlage im Jahr 2024“ and was prepared with best care and attention. Nevertheless, the German version of this report shall be taken as authoritative. No guarantee can be given with respect to the English translation.

1. Summary

A total of 61 measuring institutes took part in HLNUG's dust emission proficiency tests (substance range P) in 2024, 34 of which were §29b measuring bodies and 27 of which were volunteers. As in the past, the success rate for the §29b measuring bodies (81%) was significantly higher than that for the volunteers (15%).

A total of 47 measuring institutes took part in the gas emission proficiency tests (substance range G) in 2024, 39 of which were §29b measuring bodies and 8 volunteers. As in previous years, the success rate for the §29b measuring bodies (69%) was significantly higher than for the volunteers (20%).

A total of 17 measuring bodies took part in the odour emission proficiency tests (substance range O) in 2024, 11 of them on the basis of an authorisation in accordance with §29b BImSchG and 6 of them voluntarily. Here, 82% of the authorised participants were successful and 50% of the volunteers were successful.

2. Introduction

2.1 Legal Background

The stack emission proficiency tests offered at the Emission Simulation Apparatus (ESA) of Hessisches Landesamt für Naturschutz, Umwelt und Geologie (HLNUG, Hessian Agency for Nature Conservation, Environment and Geology) in Kassel were developed for the quality control of measuring bodies authorised to perform measurements in accordance with §29b Bundes-Immissionsschutzgesetz (BImSchG, Federal Immission Control Act (1)) in Germany. The proficiency tests presented in this annual report are accredited according to DIN EN ISO/IEC 17043 (2) and are recognised by all authorising authorities in Germany within the meaning of §16 Para. 4 No. 7a of the 41. Bundes-Immissionsschutzverordnung (41. BImSchV (3), 41st Federal Immission Control Ordinance). Regular successful participation in these stack emission proficiency tests is therefore a prerequisite for maintaining an authorisation in accordance with §29b BImSchG.

Consequently, about 80-90% of the participants are laboratories authorised to perform measurements in accordance with §29b BImSchG (Federal Immission Control Act), or applicants for authorisation in accordance with BImSchG. Nevertheless, other measuring institutes can also participate in the HLNUG stack emission proficiency tests, e.g. laboratories that do not perform measurements in the regulated sector in Germany but still want to check the quality of their emission measurements.

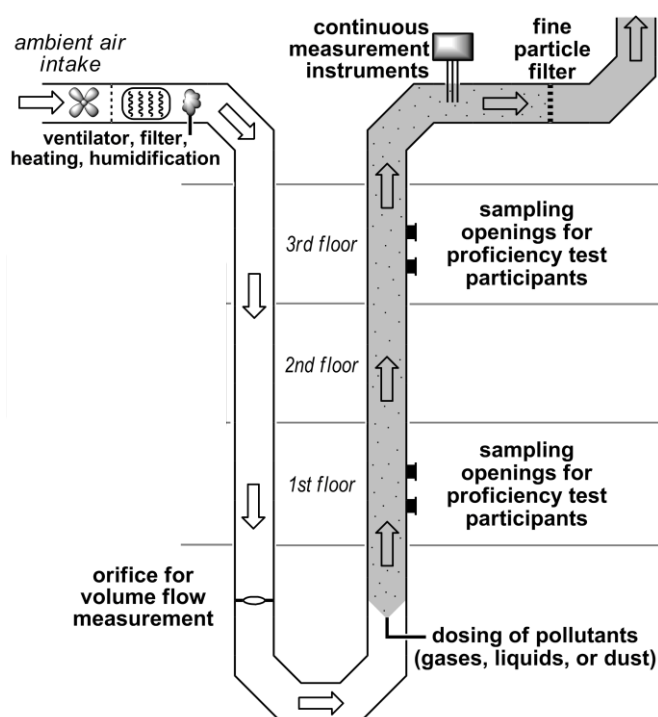
2.2 The Emission Simulation Apparatus

The prerequisite for carrying out stack emission proficiency tests is the ability to provide all participants at the same time with a stable and clearly defined simulated exhaust gas. For this purpose, HLNUG operates the Emission Simulation Apparatus (ESA, see scheme 1). It was designed as a model for an industrial flue gas chimney. It serves not only to carry out emission proficiency tests but also to carry out model investigations in the field of emission measurement technology.

The ESA has a total length of 110 m and extends over all seven floors of the HLNUG building in Kassel. The heart of this system is a vertical, 23 m high round stainless steel conduit with an inner diameter of 40 cm. This part of the ESA is the actual chimney substitute, equipped with sampling ports for taking samples for emission measurements.

The test atmosphere in the form of simulated exhaust gas is created by drawing in ambient air, pumping it through the system, heating it and adding precisely metered quantities of pollutants. The exhaust gas typically flows through the ESA at approx. 4 – 15 m/s, moving a volume of approx. 2000 – 6000 m³/h through the system.

The air pollutants to be measured by the participants in the proficiency test are dispensed into the air flow in the dosing laboratory in the basement. For this purpose, the dosing laboratory is equipped with various Coriolis mass flow meters for dosing different gases, a dosing system for liquids, and a brush dosing unit for dosing dusts. The concentrations of air-polluting substances generated in the dosing laboratory are constantly monitored by continuous measurement.



Scheme 1: Scheme of HLNUG's emission simulation apparatus (simplified and not true to scale)

3. Organisational Information

In 2024, the following proficiency tests of the substance ranges P, G, and O were carried out:

Table 1: Proficiency Tests organised by HLNUG

proficiency test	substance range	start	end	participants
24P1	Staub (dust)	05.02.2024	06.02.2024	7
24G1	Gas	06.02.2024	08.02.2024	8
24P2	Staub (dust)	19.02.2024	20.02.2024	7
24G2	Gas	20.02.2024	22.02.2024	7
24P3	Staub (dust)	04.03.2024	05.03.2024	7
24G3	Gas	05.03.2024	07.03.2024	7
24P4	Staub (dust)	18.03.2024	19.03.2024	8
24G4	Gas	19.03.2024	21.03.2024	8
24P7	Staub (dust)	22.04.2024	23.04.2024	6
24G7	Gas	23.04.2024	25.04.2024	6
24P81	Staub Kurzversion (dust short version)	11.06.2024	11.06.2024	4
24P82	Staub Kurzversion (dust short version)	12.06.2024	12.06.2024	4
24P83	Staub Kurzversion (dust short version)	13.06.2024	13.06.2024	3
24O3	Geruch (odour)	19.09.2024	19.09.2024	1
24O1	Geruch (odour)	24.09.2024	24.09.2024	7
24O2	Geruch (odour)	26.09.2024	26.09.2024	9
24P5	Staub (dust)	04.11.2024	05.11.2024	7
24G5	Gas	05.11.2024	07.11.2024	7
24P6	Staub (dust)	18.11.2024	19.11.2024	8
24G6	Gas	19.11.2024	21.11.2024	4

These proficiency tests were organised and carried out under the following conditions (see specifications for the respective substance ranges for details):

Table 2: Characteristics of HLNUG’s stack emission proficiency tests

	dust (substance range P)	gas (substance range G)
duration of each sampling		30 min
number of samplings	standard-version: for each component 9 (+ introductory measurement) short version: for each component 6 (+ introductory measurement)	
sampling	simultaneously for all participants (1 st and 3 rd floor)	
basic conditions	volume flow: 2000 ... 6000 m ³ /h (standard conditions, dry) mean flow velocity: 4 ... 15 m/s (operating conditions, wet) temperature: 20 ... 50 °C water vapour concentration: 0 ... 50 g/m ³ (standard conditions, dry) static pressure: 0 ... 10 hPa	

	dust (substance range P)	gas (substance range G)
concentrations	dust (total): 1 ... 15 mg/m ³ heavy metals: 1 ... 200 µg/m ³	NO _x as NO ₂ : 60 ... 450 mg/m ³ CO: 10 ... 100 mg/m ³ TOC: 4 ... 100 mg/m ³ ethylbenzene: 1 ... 40 mg/m ³ toluene: 1 ... 40 mg/m ³ xylene (sum of isomers): 1 ... 40 mg/m ³ SO ₂ : 20 ... 150 mg/m ³ formaldehyde: 2 ... 20 mg/m ³
result submission	within six weeks after the end of the proficiency test, in mg/m ³ for dust concentrations and µg/m ³ for heavy metal concentrations respectively, relating to standard conditions (dry) and with two digits after decimal point.	within four weeks after the end of the proficiency test, in mg/m ³ , relating to standard conditions (dry) and with two digits after decimal point.
submission procedure	results are entered into an Excel-file provided by HLNUG and handed in via e-mail.	
	odour (substance range O)	
duration of each sampling	10 min	
number of samplings	for each component 3	
basic conditions	2000 ... 6000 m ³ /h, flow velocity > 4 m/s, water vapour up to 50 g/m ³	
concentrations	approx. 50 ... 50000 ouE/m ³	
result submission	within one week after the proficiency test, in ouE/m ³ , rounded to integers	
submission procedure	results are entered into an Excel-file provided by HLNUG and handed in via e-mail.	

The proficiency tests were organised by:

Hessisches Landesamt für Naturschutz, Umwelt und Geologie
(Hessian Agency for Nature Conservation, Environment and Geology)

Dezernat I3 – Luftreinhaltung: Emissionen
(Department I3 – Air Pollution Control: Emission)

The location of the proficiency tests was:

Hessisches Landesamt für Naturschutz, Umwelt und Geologie
 Ludwig-Mond-Str. 33
 34121 Kassel
 - GERMANY -

Tel.: +49 – 561 – 2000 137

Fax: +49 – 561 – 2000 225

E-Mail: pt@hlnug.hessen.de

Technically responsible for the execution of the proficiency tests are currently:

Dr. Jens Cordes, Benno Stoffels and Prof. Dr. Dominik Wildanger.

4. Execution of the Proficiency Tests

4.1 Description of the Test Objects

In contrast to proficiency tests by other providers, HLNUG's stack emission proficiency tests take place at a stack simulator and include the sampling procedure. The test object in our proficiency tests is therefore the exhaust gas flow in the duct during the measurement period (see section 2.2). The test objects therefore only exist during the measurement, and the usual specifications for homogeneity and stability are therefore subject to interpretation for the stack emission proficiency tests at the ESA (4). Extensive investigations have shown that the standard deviations between the samples for the sampling points or measurement cross sections assigned to the participants reach the following maximum values:

Table 3: Maximum values of between samples standard deviations

variable	determined at	relative standard deviation between samples [%]
mass concentration of total dust and heavy metals	all available measurement planes (grid measurements)	1.58
mass concentrations of gases	lowest available measurement plane (point measurements)	0.15
mass concentrations of evaporated liquids	lowest available measurement plane (point measurements)	0.16

All determined between samples standard deviations are well below the criteria for the proficiency assessment of the participants. This ensures that all participants in the proficiency test will find comparable sampling conditions. The position of the sampling, i.e. the measurement plane assigned by the organiser, has no significant influence on the mass concentrations measured by the participant. An equivalent to the stability test in conventional proficiency tests does not exist at the ESA, as the test objects are not stored after the assigned values have been determined. Instead, the assigned values are determined individually for each test object during its generation, and thus during the simultaneous measurement by the participants.

4.2 Preparation of the Test Objects

The exhaust gas flow sampled by the participants in the ESA is generated by adding the test substances to be measured to the air flow generated by the system. Gases are added as pure substances, evaporated liquids either also as pure substances or as solutions in other evaporable liquids. Sometimes these liquids are also dosed as a homogeneous mixture of different pure substances (5).

In contrast to the pure substances in gas and odour proficiency tests, no reference materials are available on the market in sufficient quantities for particulate substances. Therefore, for proficiency tests of the substance range P, the certified reference materials produced by HLNUG according to DIN EN ISO 17034 (6) are used. The matrix here is an industrial dust, which is optimised by specific heavy metal doping, grinding, sieving and drying steps. Finally, a complete homogenisation of the dust standard is achieved by intensive mixing of the batch.

The determination of the conventionally correct value ("assigned value") of the heavy metal concentration of a doped dust batch is based on the data from interlaboratory analyses carried

out by laboratories of various German state institutes. The robust mean value from the individual values of the interlaboratory comparisons is regarded as the assigned heavy metal content value of the dust standard. The dust is subject to a homogeneity and stability test and verification, which is repeated at certain intervals. Homogeneity and stability of the test dusts are verified according to DIN ISO 13528 (7).

4.3 Metrological Traceability

The gaseous substances CO, NO and propane are dosed using Coriolis flow sensors. The mass flows are measured and gravimetrically traced via suitable test weights and balances. During dosing, liquids are taken from a container located on a balance. The mass flow is also recorded here by recording the weight values, and the balances used are metrologically traced via suitable test weights. The mass flows for SO₂ and dust are determined by differential weighing of the containers used. The assigned values of the heavy metal concentrations in the dust are determined by competent laboratories using various analytical instruments within the framework of interlaboratory comparisons. Within the scope of these interlaboratory comparisons, a total digestion of the dust is carried out in accordance with DIN EN 14385 (8), as well as an analysis using calibrated measuring equipment. This calibration is carried out by means of element solutions of known traceable composition. Consequently, the heavy metal concentrations in the test dusts used are metrologically traceable. The volume flow is determined by means of an orifice plate, which is regularly checked by means of metrologically traceable measuring instruments. By calculating from metrologically traceable mass flows and metrologically traceable volume flows, all mass concentrations indicated are also metrologically traceable. The maximum values of the relative standard uncertainty of the assigned values can be found in the tables 5 to 7. Detailed information is given in the results communications of the individual proficiency tests.

4.4 Execution of the Measurements

Each participant determines the mass concentration of the emission components in accordance with (DIN) EN 15259 (9). In addition, the gas flow conditions must be recorded before the actual sampling begins. This includes exhaust gas velocity/flow rate, exhaust gas temperature and humidity as well as the air pressure in the system.

Table 4: Mandatory measurement methods

substance range	component	measurement method
P	dust	(DIN) EN 13284-1 (10)
	heavy metals	(DIN) EN 14385 (8)
G	NO _x as NO ₂	(DIN) EN 14792 (11)
	CO	(DIN) EN 15058 (12)
	TOC	(DIN) EN 12619 (13)
	ETX	(DIN) CEN/TS 13649 (14)
	SO ₂	(DIN) EN 14791 (15)
	formaldehyde	VDI 3862 part 2 (16), part 3 (17) or part 4 (18)
O	four odourants	(DIN) EN 13725 (19)

4.5 Evaluation of the Proficiency Tests

4.5.1 Calculation of z-Scores

Substance Ranges P and G

The evaluation of the proficiency test is carried out in accordance with the respective specifications (for substance ranges P and G) using the z-score procedure. For the measurement value x_{ijk} , which is the result of measurement i of concentration level j of component k , a z-score value z_{ijk} is determined:

$$z_{ijk} = \frac{x_{ijk} - X_{ijk}}{\sigma_k \cdot X_{ijk}}$$

In this equation, X_{ijk} is the assigned value (target value) of the measurement, and σ_k is the criterion for proficiency assessment (precision criterion) for component k . The assigned value is calculated from measurement data of the dosing devices and the volume flow.

Substance Range O

For odour emission proficiency tests, the evaluation is carried out on the basis of the z-score procedure, using logarithmised values:

$$z_{ik} = \frac{1}{\sigma_k} \cdot \log_{10} \left(\frac{x_{ik}}{X_{ik}} \right)$$

In this equation, X_{ik} is the assigned value of the measurement, and σ_k is the precision criterion for component k . The assigned value X_{ik} is calculated from the mass concentration c_{ik} and the odour threshold $c_{0,k}$ of the component:

$$X_{ik} = \frac{c_{ik}}{c_{0,k}} \text{ ou}_E/\text{m}^3$$

The dosed mass concentration c_{ik} is determined for each measurement based on the measurement data of the dosing device and the volume flow. The odour threshold $c_{0,k}$ of n -butanol is $c_0 = 123 \mu\text{g}/\text{m}^3$. The thresholds of all other components are deduced from results of proficiency test participants according to the following procedure:

- a) A consensus value is calculated from the measurement results reported by at least 20 participants in at least two different proficiency tests previously run by HLNUG. Here, solely results of participants are taken into account, who achieved the result 'passed' for the component n -butanol in the respective proficiency test. The consensus value is obtained by the robust mean of the logarithmic values according the standard DIN ISO 13528 (7) and is updated on a regular basis by including new results. This calculation is restricted to measurements of the past five years as long as the above mentioned requirements are met.
- b) If not enough measurement results of former proficiency tests are available to determine the consensus value of a component by means of the procedure described under a), an alternative method is used: Here, the consensus value of a component offered during a proficiency test is subsequently calculated from the participants' measurement results.

Provided that the sampling was carried out within 14 days, results of several proficiency tests can be taken into account. Solely results of those participants are considered, who achieved the result ‘passed’ for the component *n*-butanol in the respective proficiency test. The consensus value is obtained by the robust mean of the logarithmic values according the standard DIN ISO 13528 (7). If less than nine measurement results for a particular component are available that fulfil the above mentioned criteria, neither a z-score-based evaluation nor a performance rating are possible.

In the odour stack emission proficiency tests in 2024, in addition to *n*-butanol the components ‘organic solvent mixture’ (ETX), tetrahydrothiophene (THT) and artificial pigsty (PIG) were used. For the components ETX and PIG, the odour threshold $c_{0,k}$ could be determined with procedure a). A consensus value of $c_0 = 198 \mu\text{g}/\text{m}^3$ was obtained for ETX from 186 measurements in the years 2019 to 2023. For the component PIG, 114 individual measurements from the years 2021 to 2023 resulted in a consensus value of $c_0 = 208 \mu\text{g}/\text{m}^3$. For the component THT, a consensus value had to be determined via procedure b). This resulted in a value of $c_0 = 0.509 \mu\text{g}/\text{m}^3$ based on 36 individual measurements in 2024.

If the uncertainty of a true value u_k determined in compliance with DIN ISO 13528 (7) results in a value for which with $\sigma_k = 0.10$ the following condition is not met:

$$\sigma_k \geq \frac{1}{0.3} \cdot \log_{10}(1 + u_k)$$

Then σ_k is adjusted in accordance with DIN ISO 13528 (7). In doing so, σ_k is recalculated precisely to two decimal places, so that the condition above is fulfilled. In 2024 this was necessary for the components THT and PIG, where σ_k had to be raised to a value of 0.14. The participants were informed about this along with their results evaluation.

4.5.2 Criteria for Proficiency Assessment

The criteria for the proficiency assessment of the participants (precision criteria) σ_k were defined as values from findings in accordance with section 6.3 of DIN ISO 13528 (7) by the German Federation/Federal States Working Group on Immission Control (LAI) and published within the framework of the specifications for stack emission proficiency tests. The values are for the individual components:

Table 5: Precision criteria dust proficiency test

No.	component	short designation	precision criterion σ_k in % of true value	max. standard uncertainty of assigned values [%]
P1	dust	St	7.0	1.55
P2	Cadmium	Cd	10.0	1.86
P3	Cobalt	Co	10.0	1.88
P4	Chromium	Cr	10.0	1.86
P5	Copper	Cu	10.0	2.18
P6	Manganese	Mn	10.0	1.99
P7	Nickel	Ni	10.0	1.92
P8	Lead	Pb	10.0	1.84
P9	Vanadium	V	10.0	2.13

Table 6: Precision criteria gas proficiency test

No.	component	short designation	precision criterion σ_k in % of true value	max. standard uncertainty of assigned values [%]
G1	NO _x as NO ₂	Nk	3.1	1.03
G2	CO	Kk	3.6	1.08
G3	TOC	Ck	3.3	1.08
G4	ethylbenzene	Ed	4.1	1.01
G5	toluene	Td	4.1	1.01
G6	sum of <i>o</i> -, <i>m</i> -, <i>p</i> -xylene	Xd	4.1	1.01
G7	SO ₂	Sd	3.4	1.11
G8	formaldehyde	Fd	3.6	1.17

Table 7: Precision criteria odour proficiency test

No.	component	short designation	precision criterion σ_k	max. standard uncertainty of assigned values [%]
O1	<i>n</i> -butanol	NBU	0.10	1.01
O2	solvent mixture	ETX	0.10	5.09
O3	tetrahydrothiophene	THT	0.14	9.96
O4	artificial pigsty odour	PIG	0.14	9.55

4.5.3 Assessment Scheme

Interpretation of the z-scores

The z-scores can be interpreted using the following scheme:

$ z_{ijk} \leq 2$	satisfactory
$2 < z_{ijk} < 3$	questionable
$ z_{ijk} \geq 3$	unsatisfactory

Generally, for each measurement resulting in a z-score of more than two, a causal research is advised.

The assessment of the individual component proceeds differently, depending on the substance range of the proficiency test.

Substance Ranges P and G

For the components in the dust and gas proficiency test, the mean value z_{jk} of the absolute values of the n z-scores of one concentration level (usually $n = 3$ for the standard version and $n = 2$ for the short version) is calculated:

$$z_{jk} = \sum_{i=1}^n \frac{|z_{ijk}|}{n}$$

Based on z_{jk} , to each concentration level a class number K_{jk} is assigned according to the following scheme:

$z_{jk} \leq 2$	results in $K_{jk} = 1$
$2 < z_{jk} < 3$	results in $K_{jk} = 2$
$z_{jk} \geq 3$	results in $K_{jk} = 3$

In the standard version of the proficiency test, for each component at least 6 measurement results must be submitted, otherwise the respective component is automatically evaluated as „failed“. There is no minimum number of measurement results for the short version.

A component was determined successfully, if the respective sum of class numbers does not exceed 6. If in justified single cases only values for two concentration levels were submitted, the component was determined successfully if the sum of class numbers does not exceed 4. Successful determinations are labelled “passed”, unsuccessful determinations are labelled “failed”. The overall result for the proficiency test is “passed”, if all components in the respective scheme (P1 to P9 for dust and G1 to G8 for gas) were rated “passed”. If one of these components was rated “failed”, the overall result is also “failed”. If a participant chose not to take part in the measurements for one or components, the overall result is “failed (incomplete participation)”, provided that all other components were assessed as “passed”.

No overall assessment is made for the proficiency tests in the short version.

Odour Emission Proficiency Test

For the evaluation of odour measurements, the mean value z_k of the absolute values of the n z-scores (usually $n = 3$) of one component is calculated:

$$z_k = \sum_{i=1}^n \frac{|z_{ik}|}{n}$$

A component was determined successfully, if

$$z_k < 3$$

is fulfilled. In this case, the component is rated “passed”. If this criterion is not met or if no measurement result was submitted in due time, the component is rated “failed”. The overall result of the proficiency test is “passed”, if all components were determined successfully. If one or more components are rated “failed”, the overall result is “failed”.

Gas Flow Conditions

For the measurement of the gas flow conditions in the dust and gas proficiency tests, only two measurement values per component are submitted and evaluated. The interpretation of the z-scores described above applies here as well. For the gas flow conditions, the mean value z_k of the absolute values of the n z-scores (usually $n = 2$) of one component is calculated:

$$z_k = \sum_{i=1}^n \frac{|z_{ik}|}{n}$$

The volume flow component is assessed as "passed" if the condition

$$z_k < 3$$

is fulfilled, otherwise the component is assessed as "failed". If no measured values were submitted, the component is marked as "not participated".

The proficiency test part Gas Flow Conditions is rated "passed", if the component volume flow is rated "passed". If this component was rated "failed", the proficiency test part Gas Flow Conditions is also rated "failed". If a participant did not participate in the component volume flow, the proficiency test part Gas Flow Conditions is noted as "not evaluated".

4.5.4 Communication of the Assessment Result

The results were sent to the proficiency testing scheme participants as a short report in tabular and diagram form, stating the respective participant number (ID code), no later than six weeks after the submission deadline.

5. Results

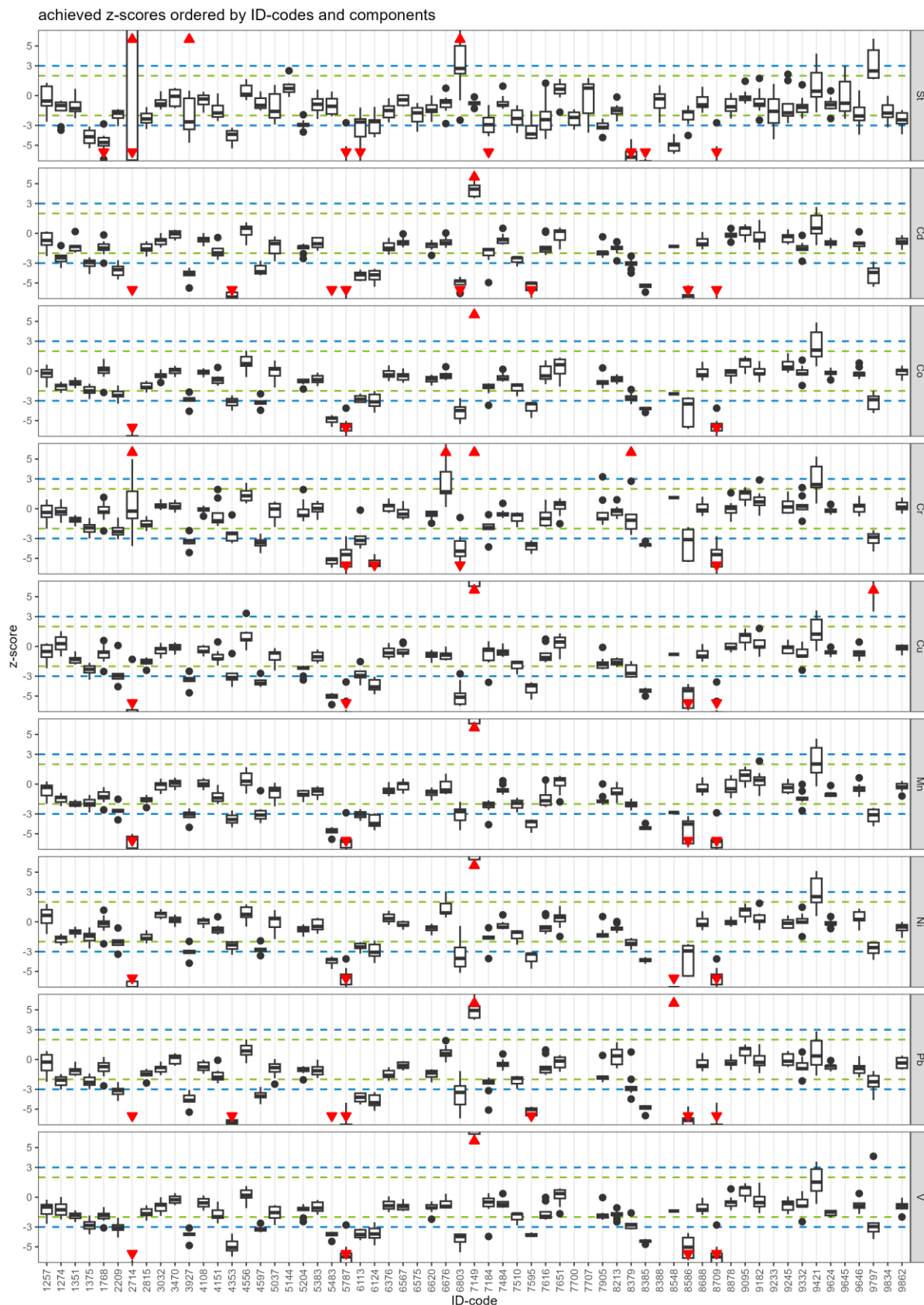
5.1 z-Scores

A compact overview of the z-scores achieved by the participants can be found in the following box whisker plots. The rectangle indicates values between the 25th and 75th percentile (interquartile distance), the continuous line in the rectangle indicates the median of the values. The "antennas" reach from the upper edge of the rectangle to the highest and from the lower edge to the lowest value, which is still within 1.5 times the interquartile distance. Values outside this range are entered separately as dots in the diagram.

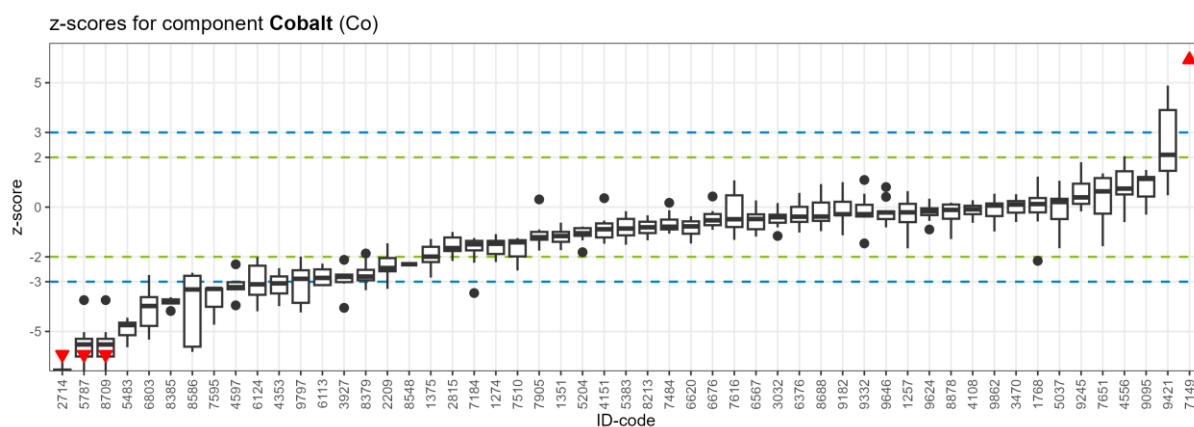
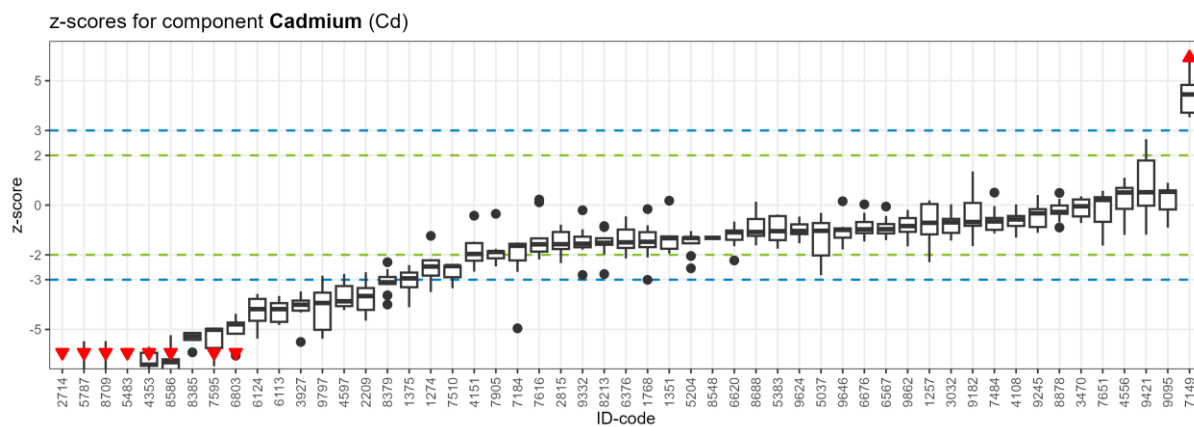
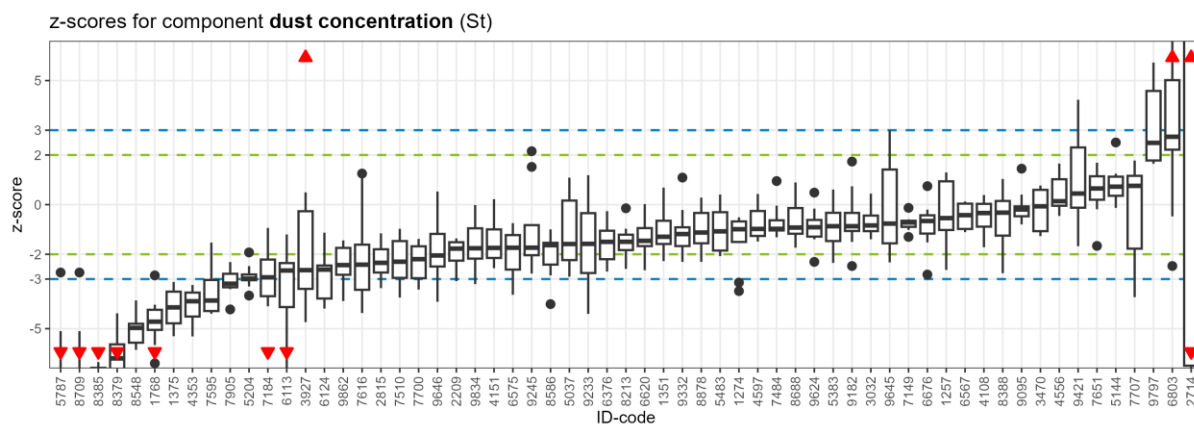
In order to be able to assess the performance of individual participants across all components and to get an impression of the quality of measurements for individual components, the diagrams are available in two different sorts; on the one hand as an overview on one page, on the other hand sorted according to the respective median of the achieved z-scores.

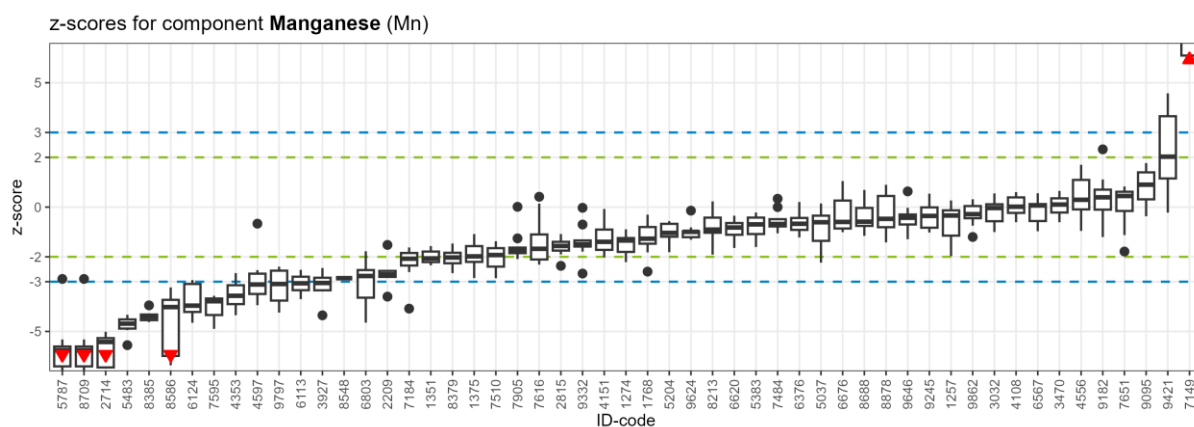
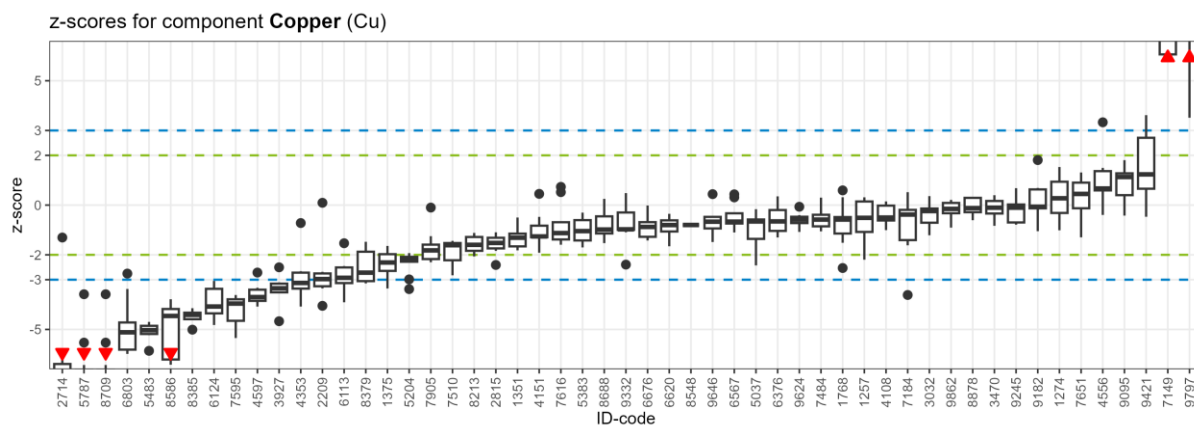
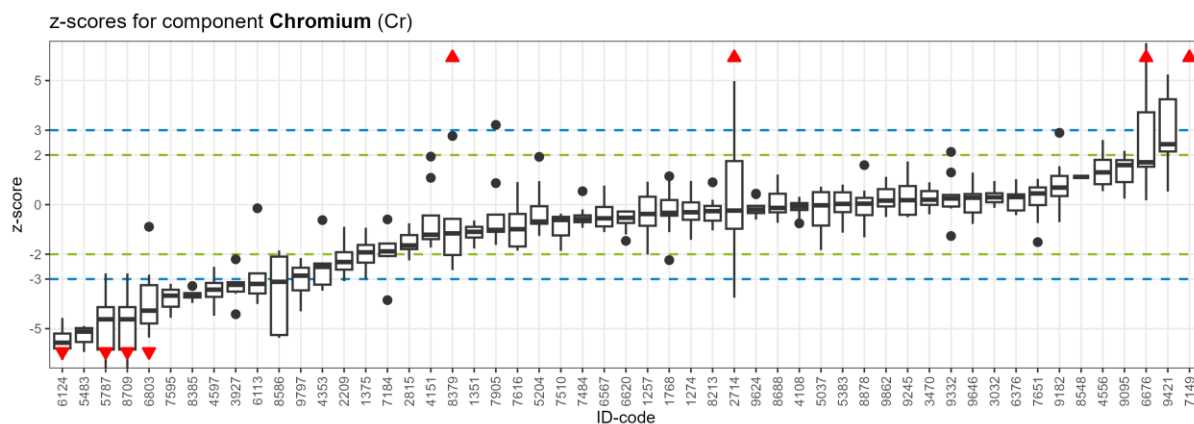
A list of the individual measurements of all participants can be found in a separate document (appendix to the annual report).

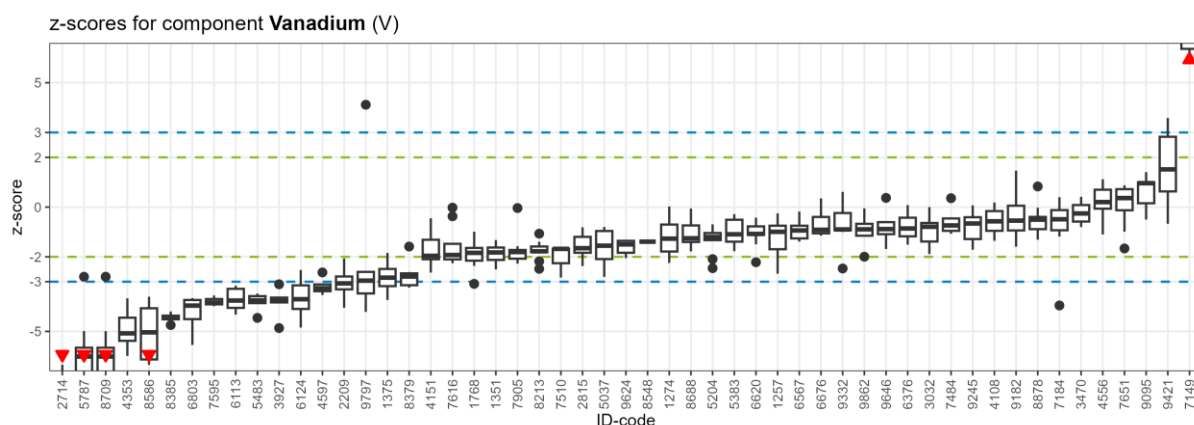
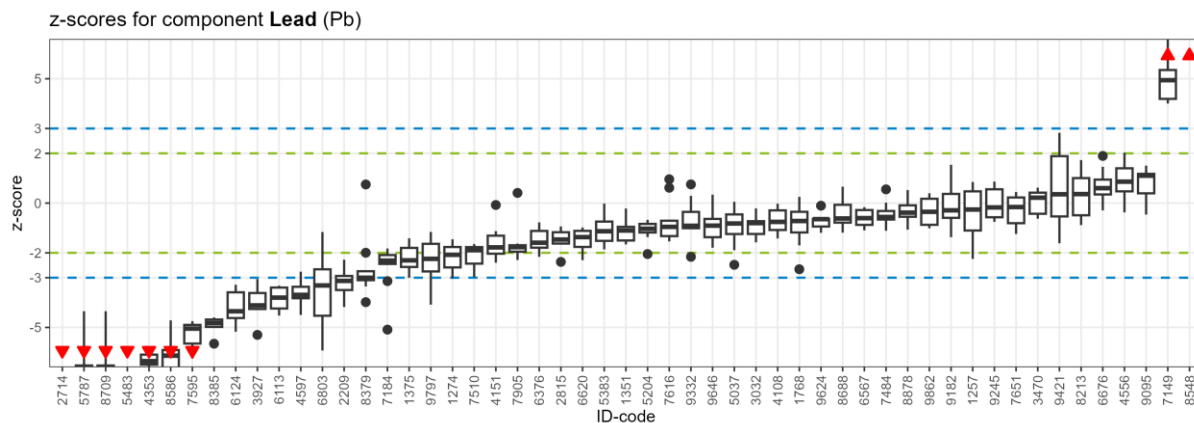
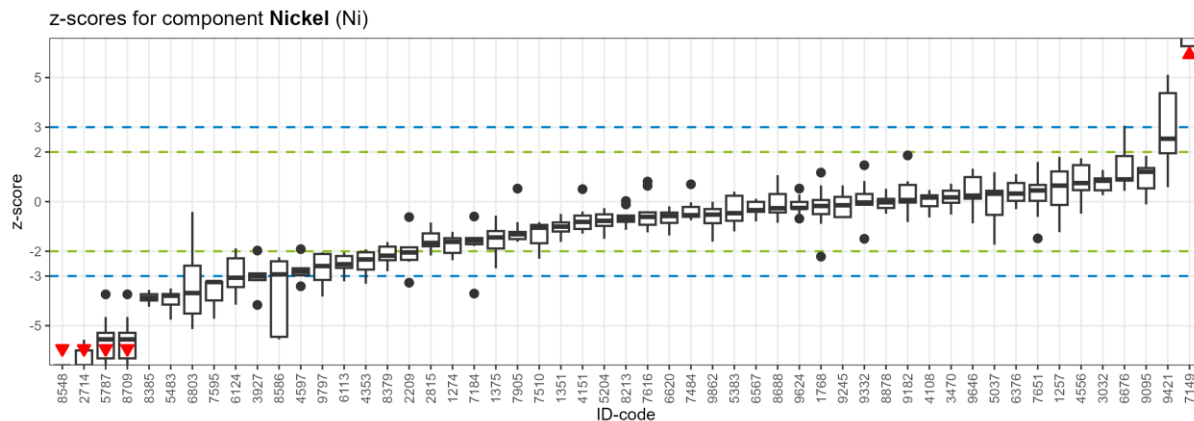
5.1.1 Dust Proficiency Test (Substance Range P)



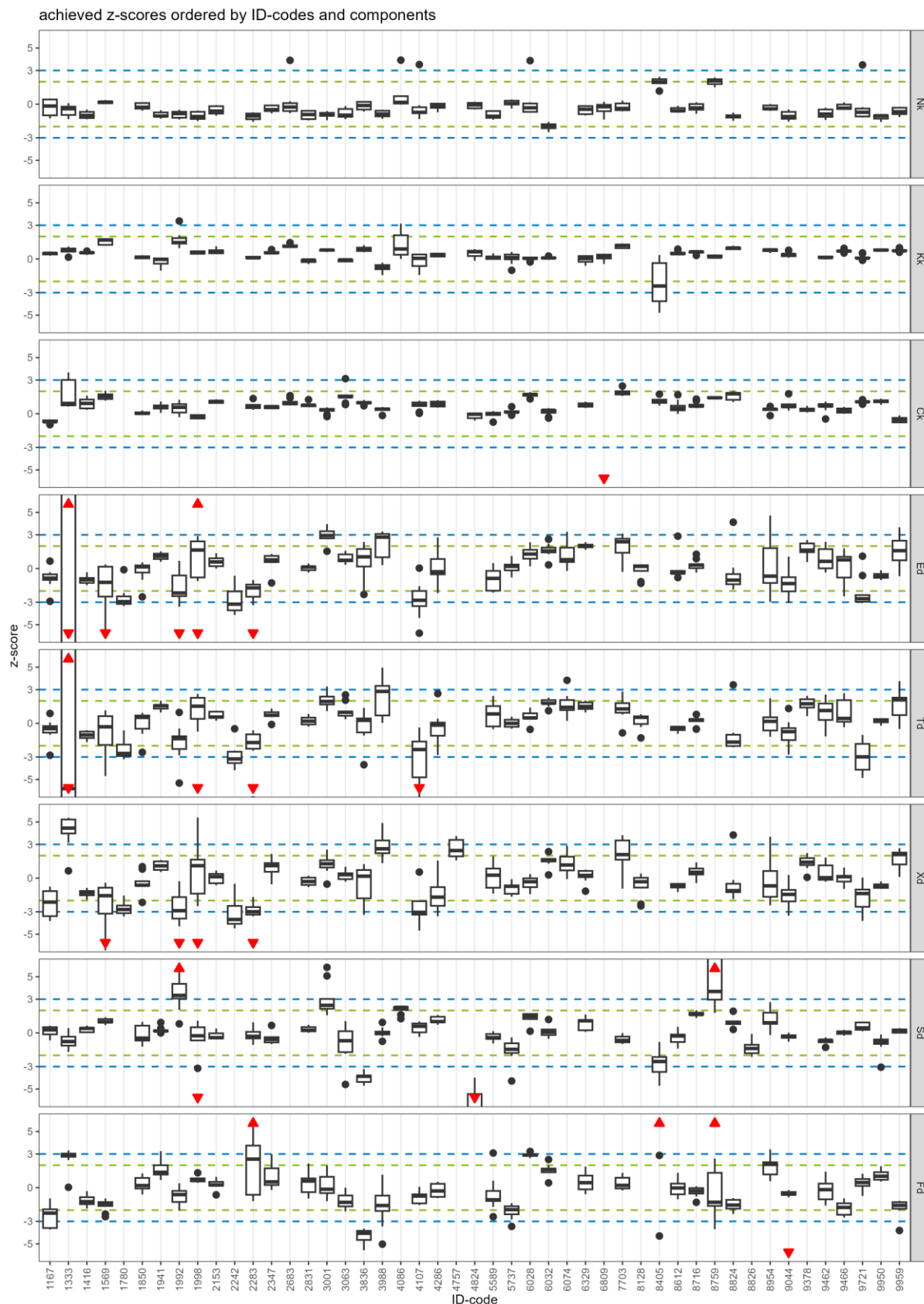
Scheme 2: Achieved z-scores dust proficiency test



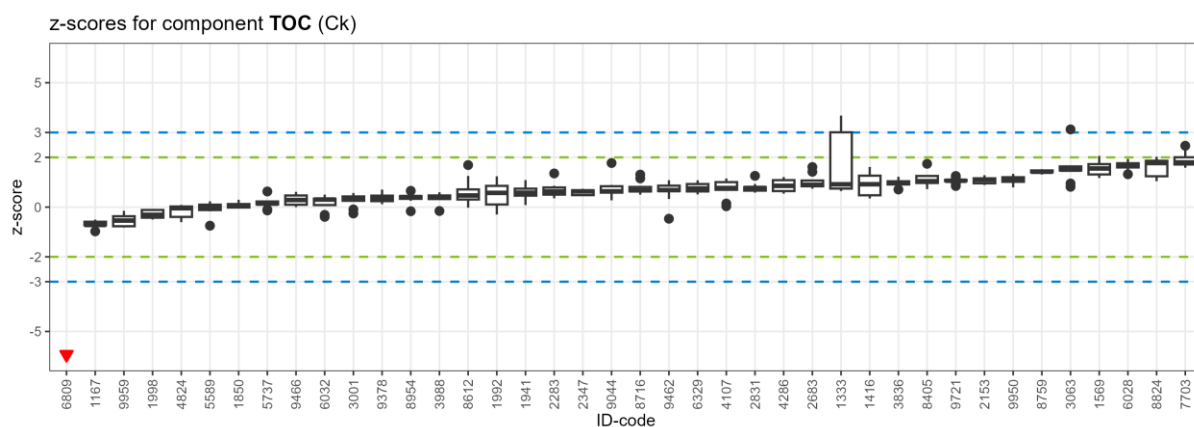
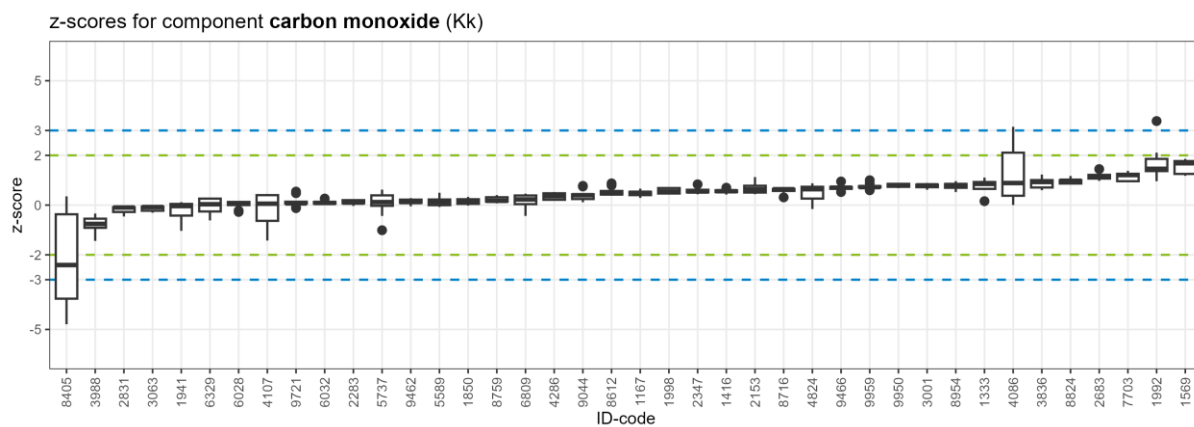
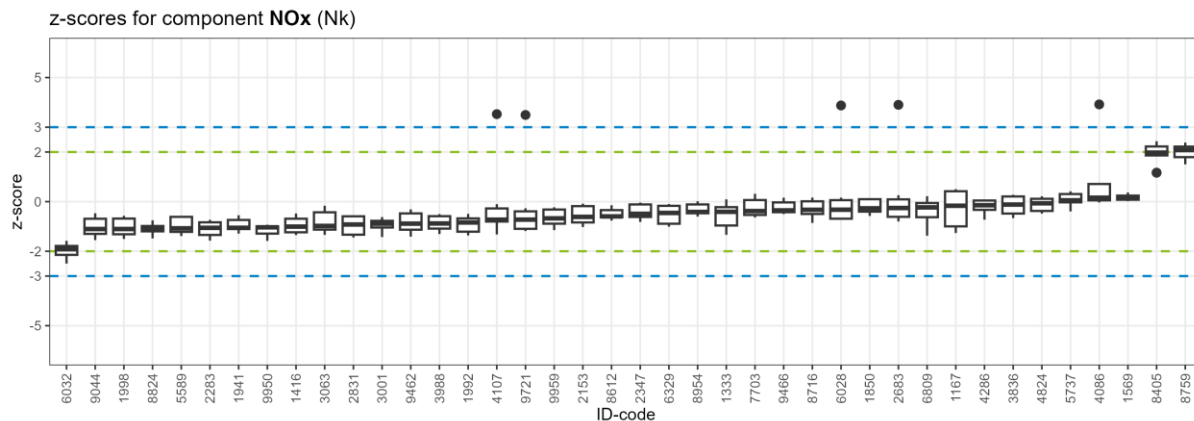


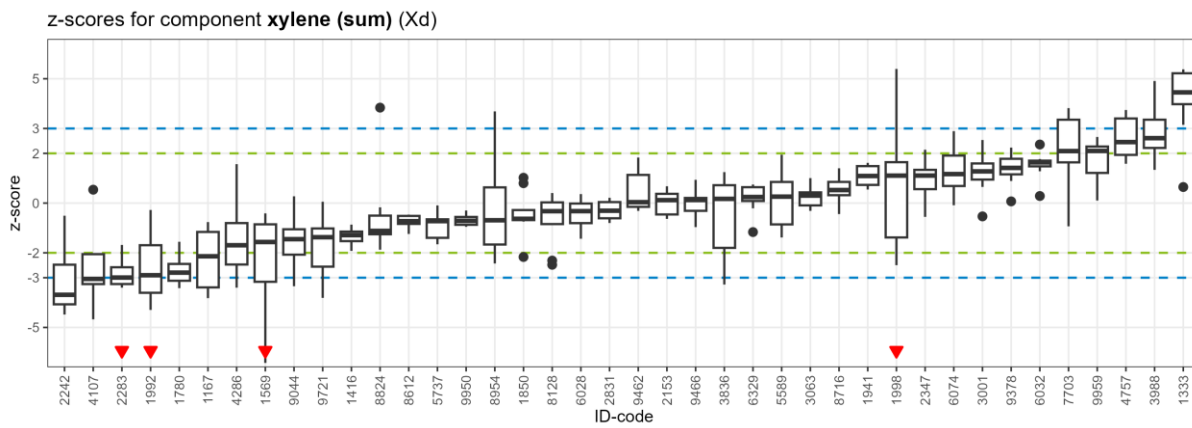
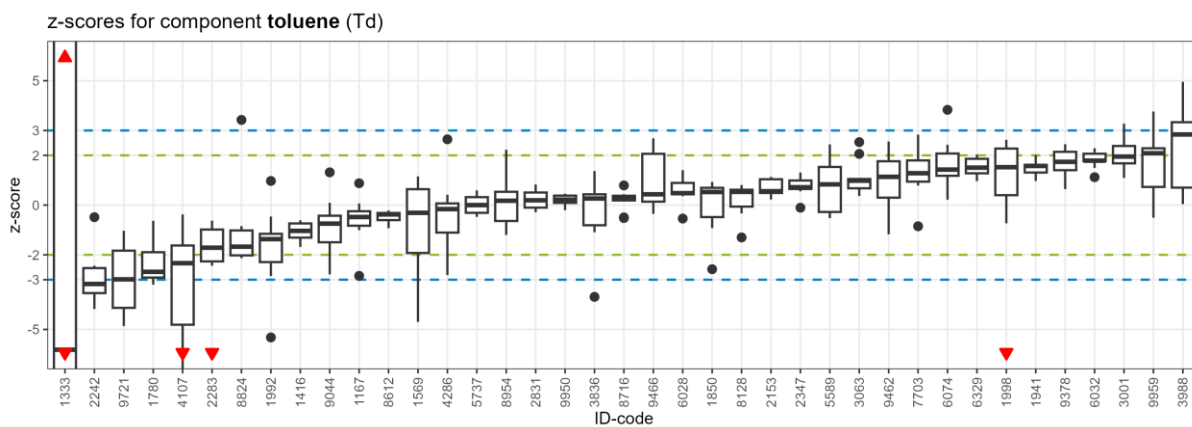
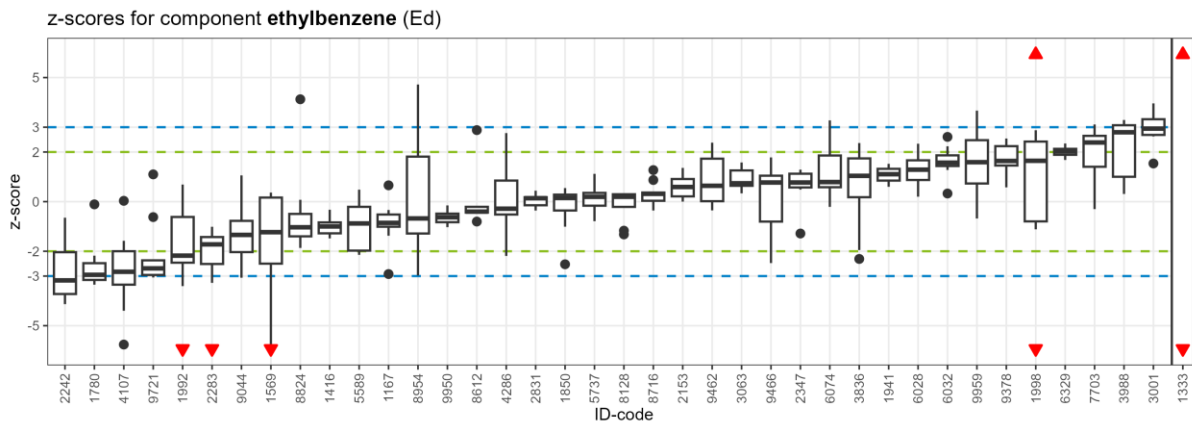


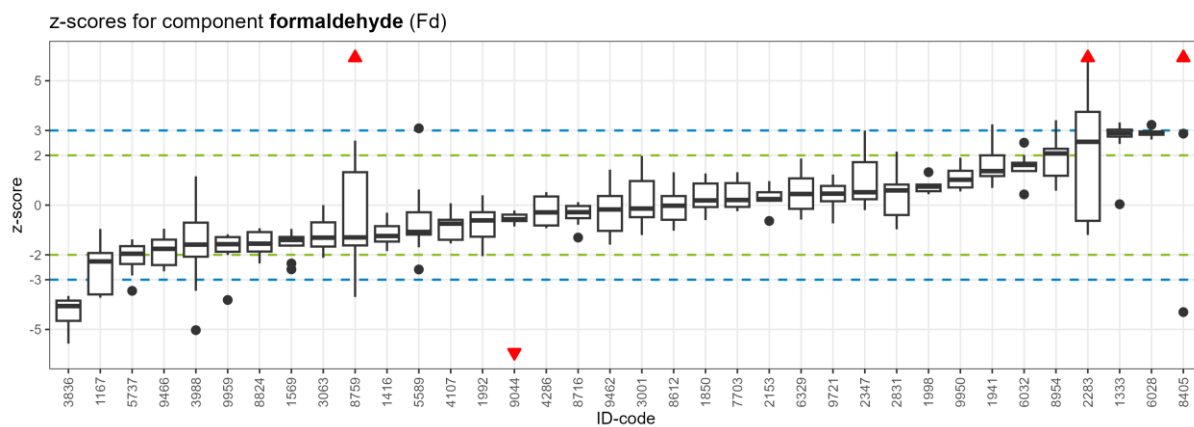
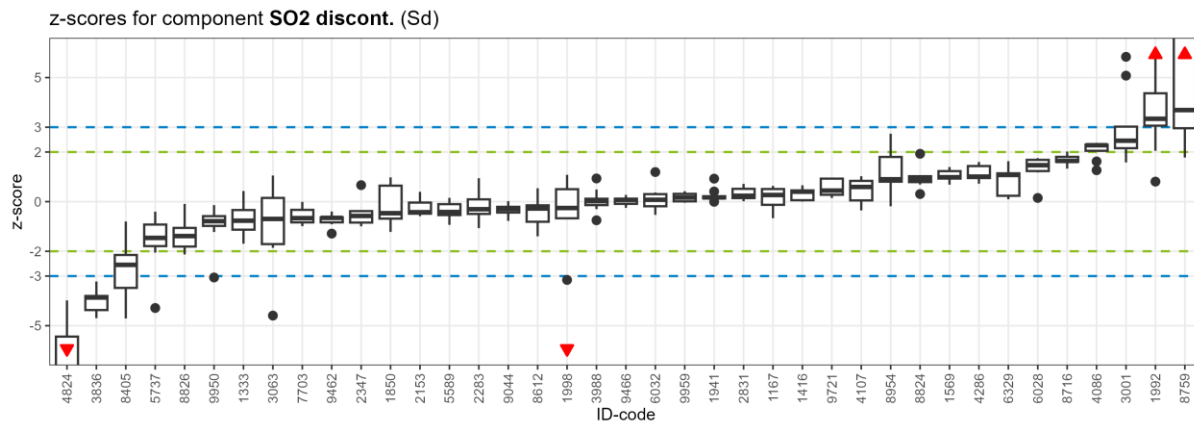
5.1.2 Gas Proficiency Test (Substance Range G)



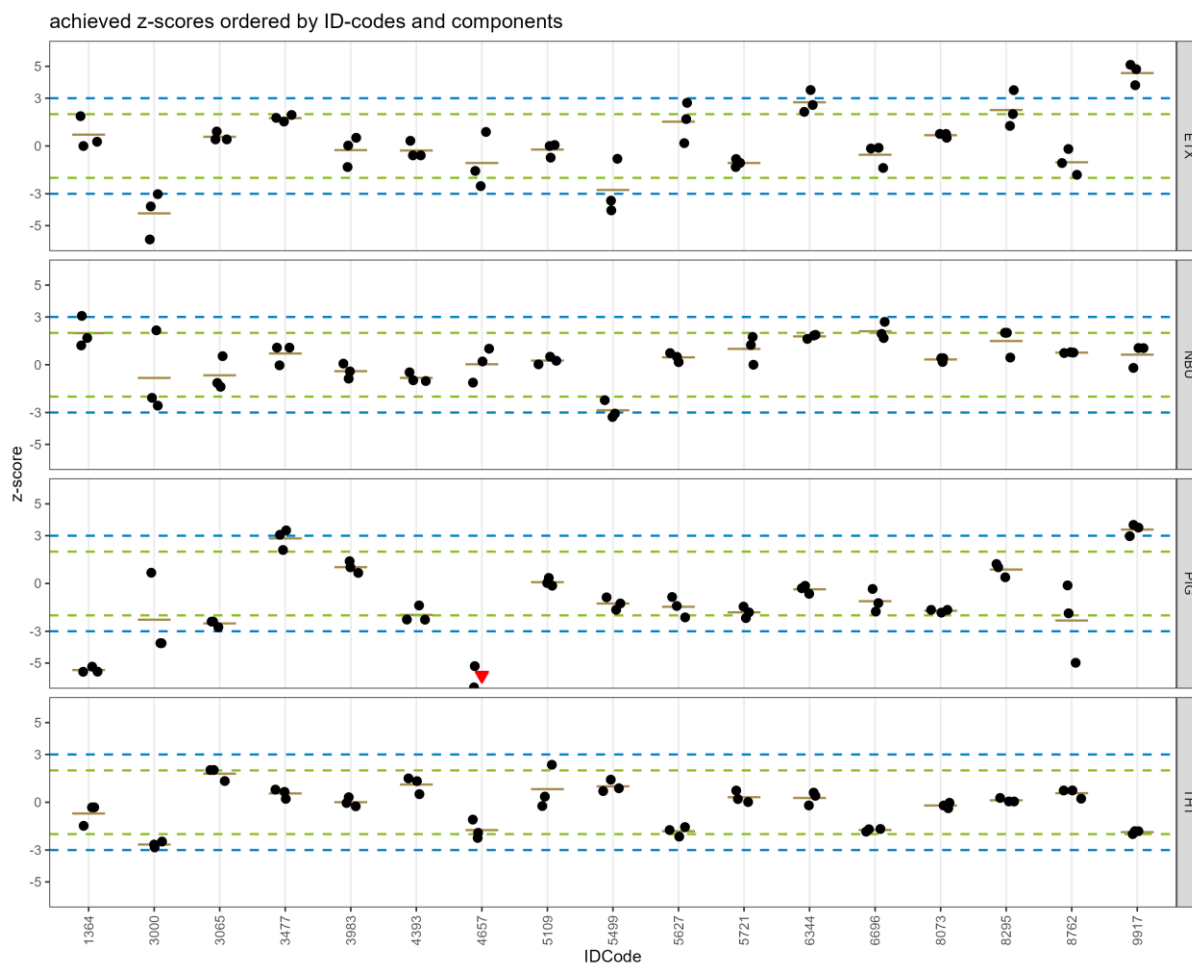
Scheme 3: Achieved z-scores gas proficiency test



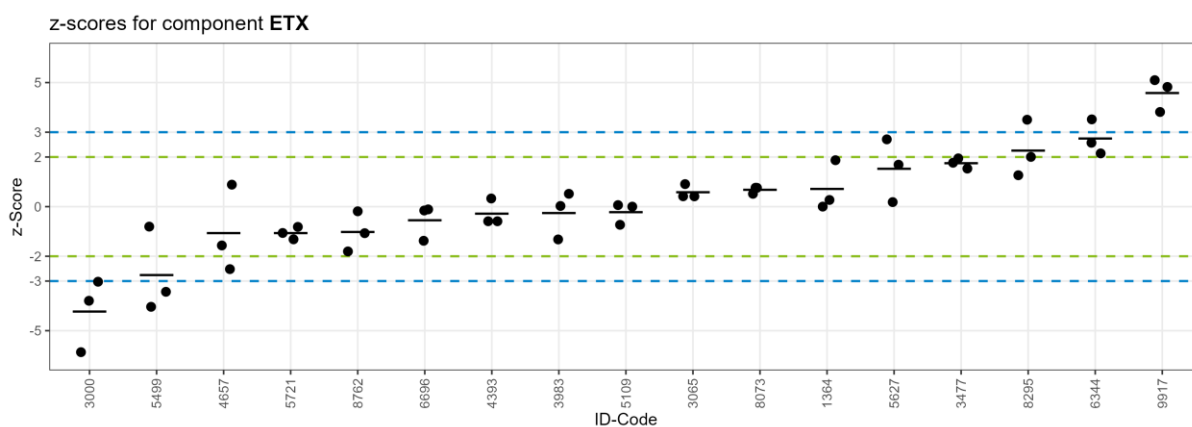


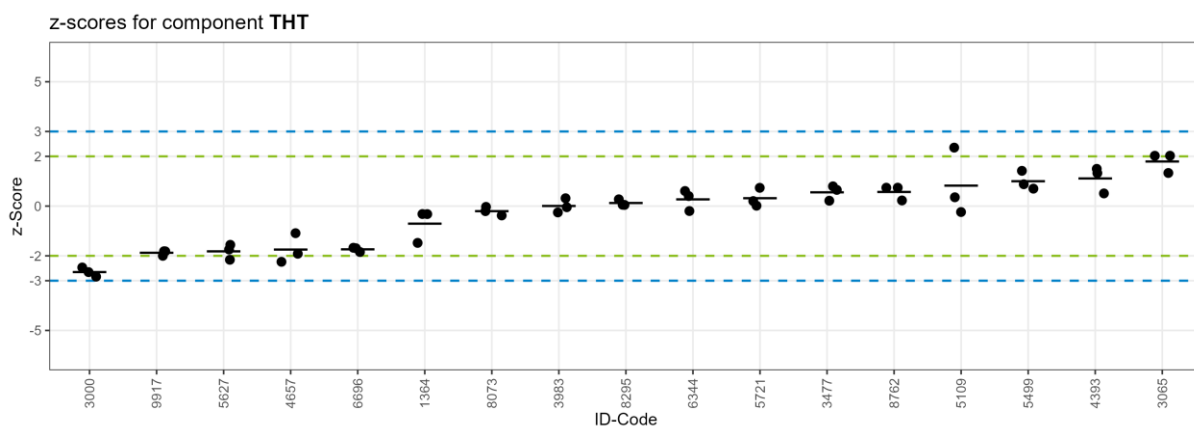
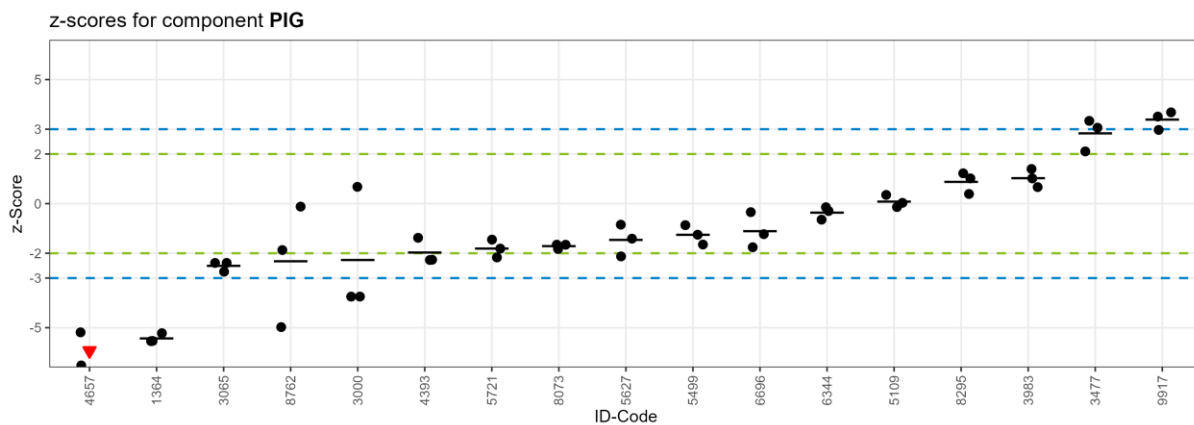
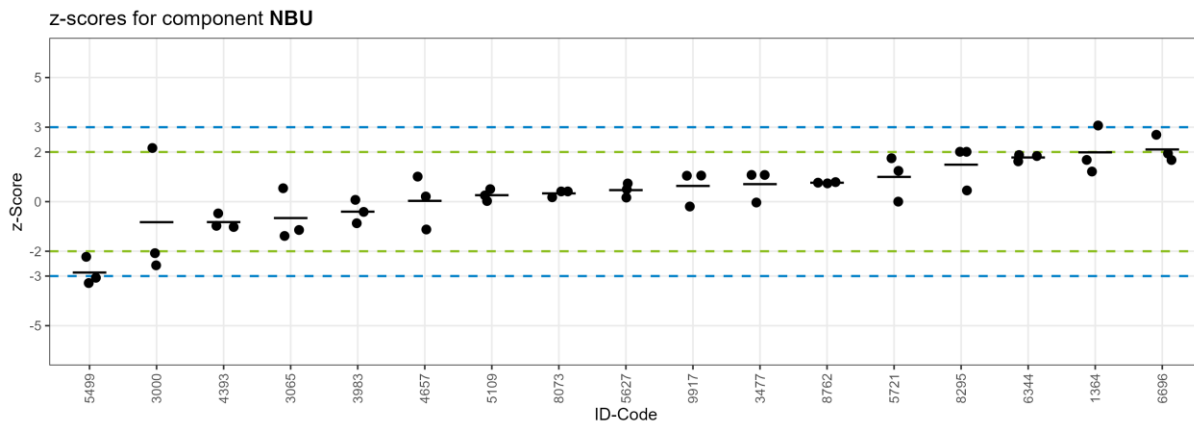


5.1.3 Odour Proficiency Test (Substance Range 0)



Scheme 4: Achieved z-scores odour proficiency test (only values in the range -5 ... 5 are shown)





5.1.4 Gas flow conditions

The following diagrams show the results obtained by the participants in the dust and gas proficiency tests for the measurement of the gas flow conditions. For each component, only two values are available per participant; these are shown as dots. The mean value of the two values is marked by a dash. The mean value of the two values is marked by a dash.

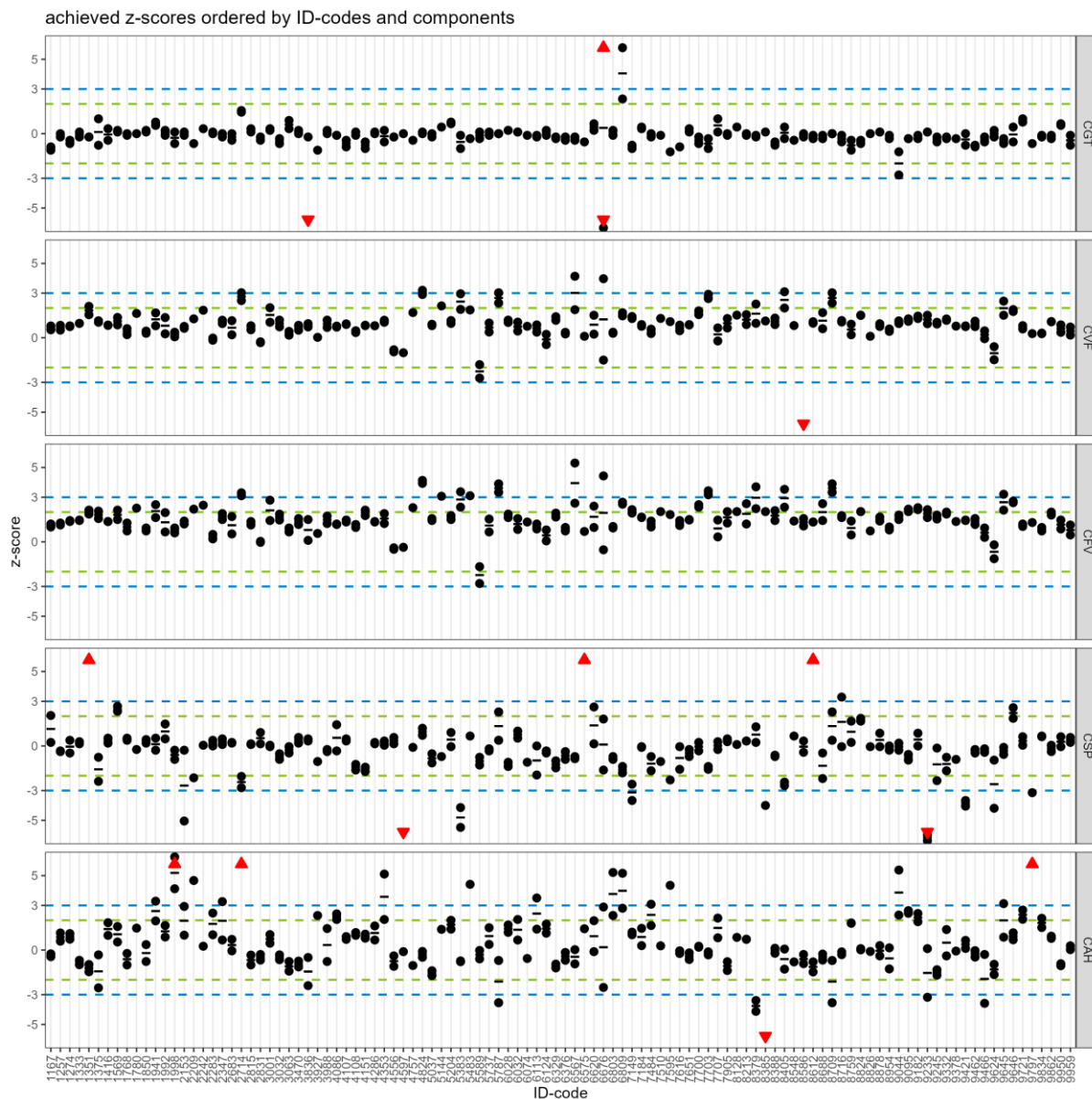
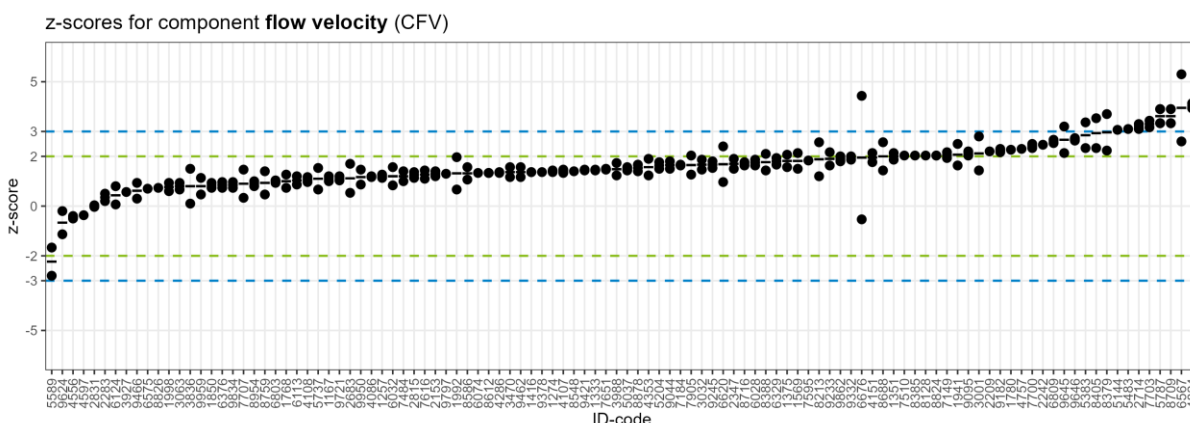
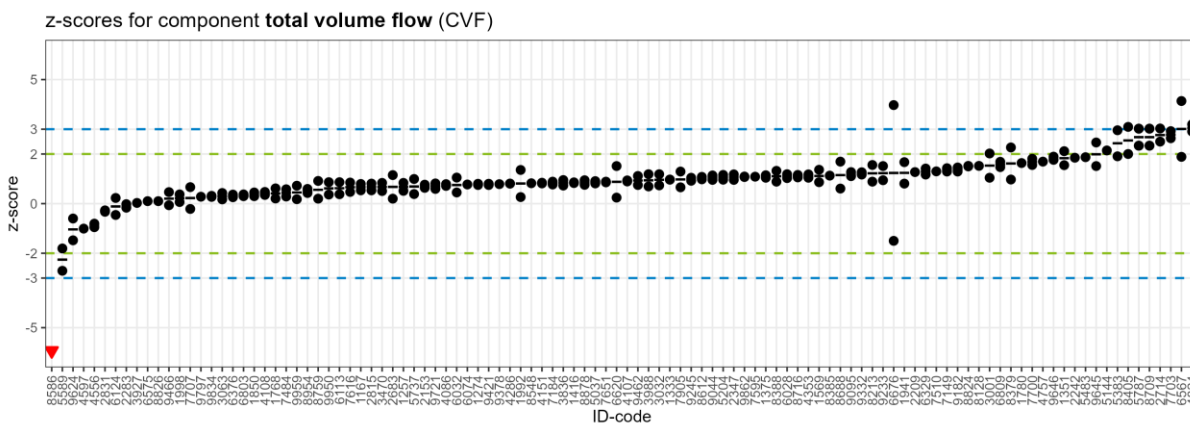
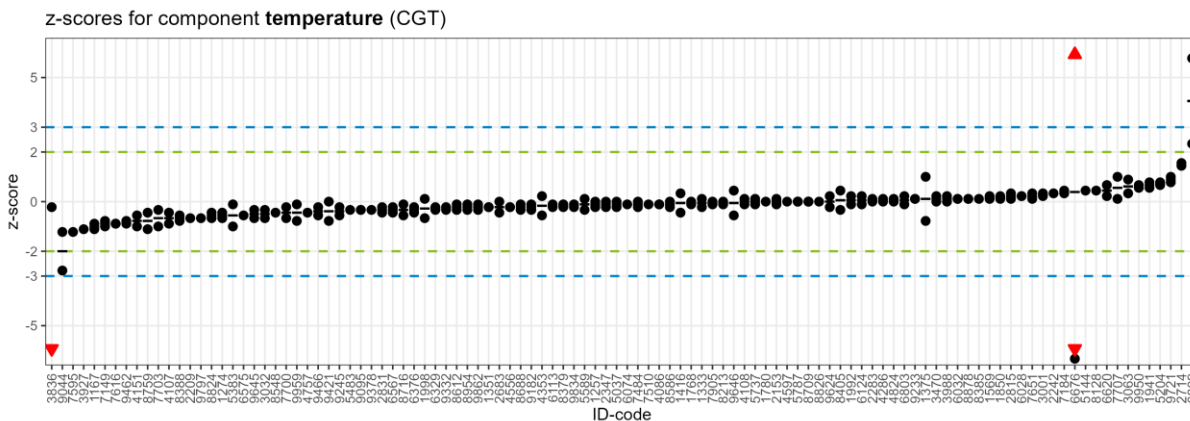
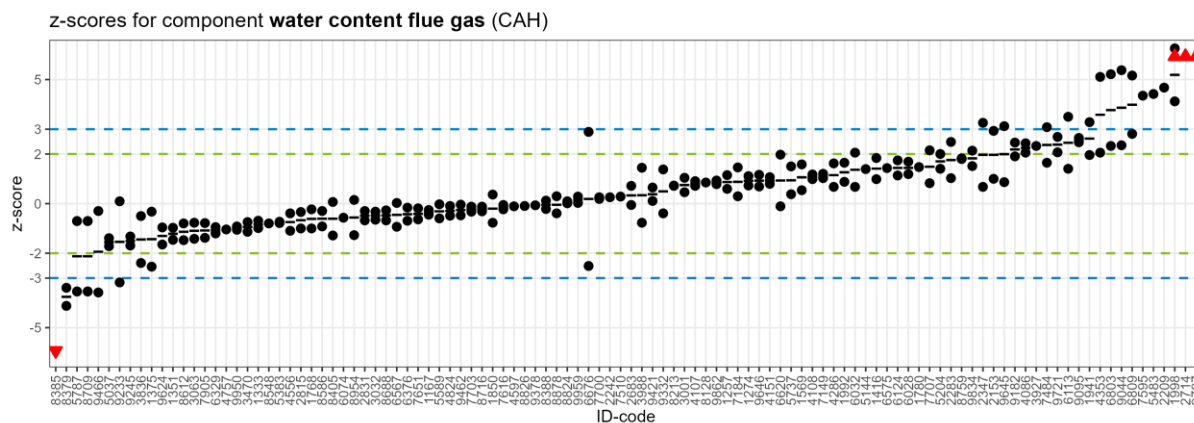
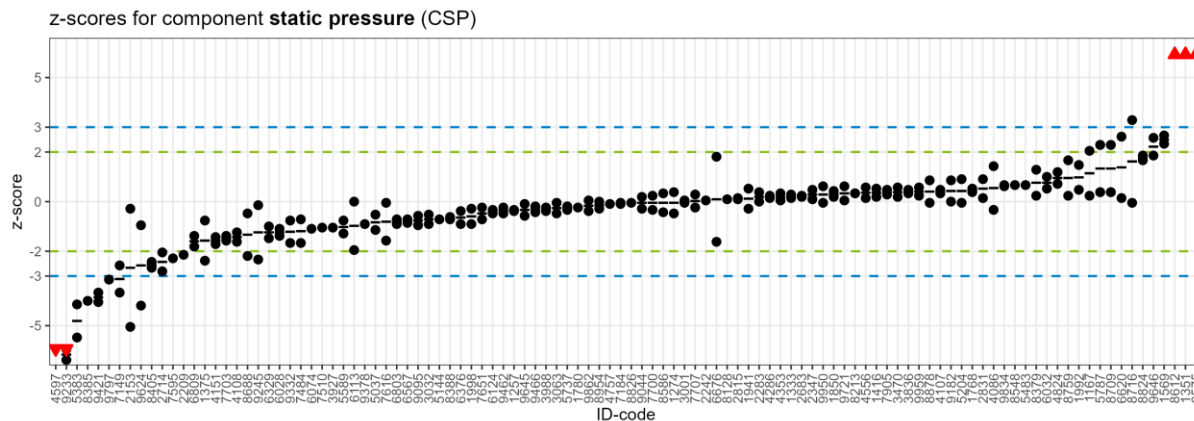


Abbildung 5: z-scores (or quotients from participant deviation and typical deviation) for gas flow conditions



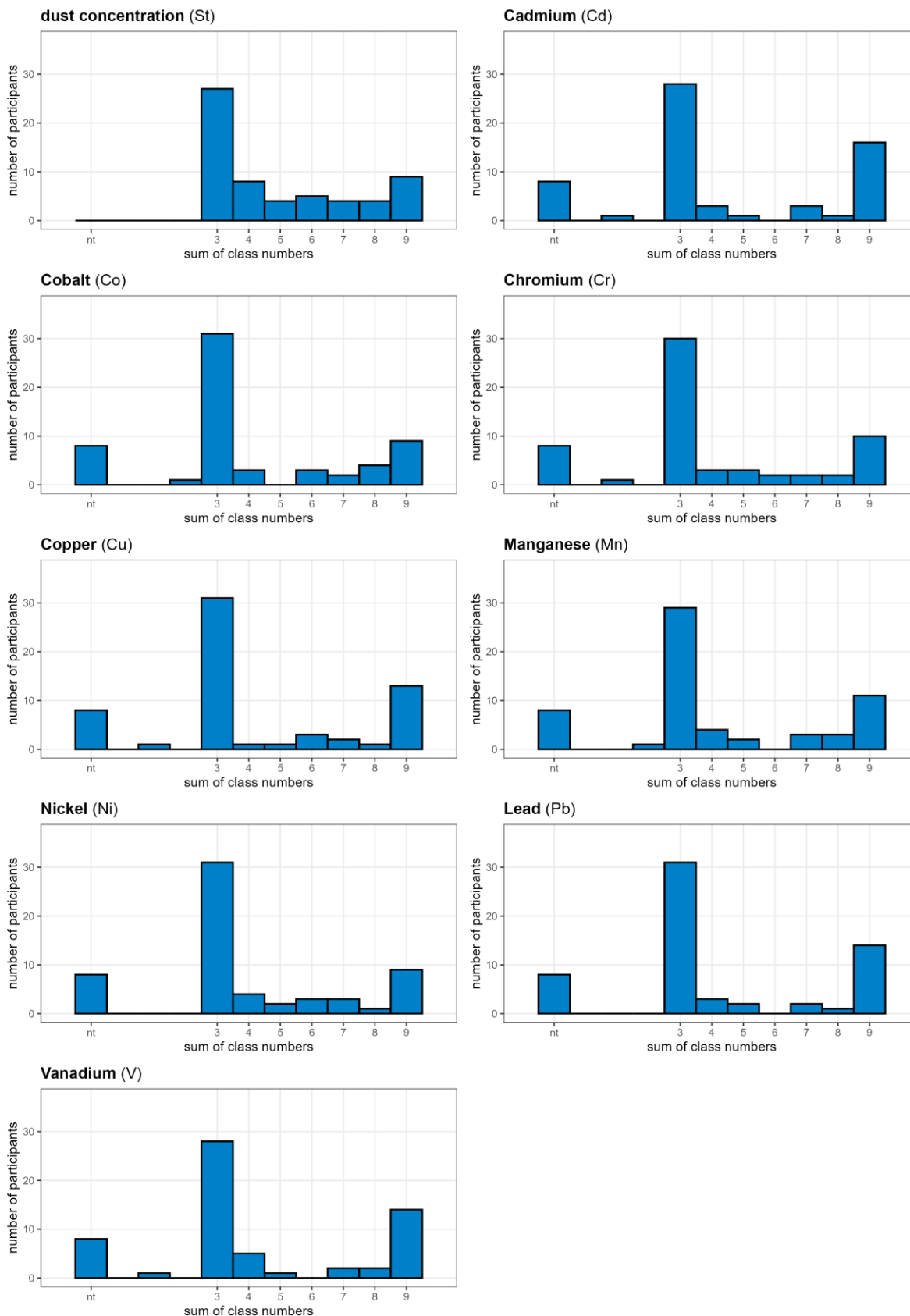


5.2 Sums of Class Numbers

The following schemes show the sum of class numbers that the participants achieved for the different components in form of histogram charts. For the interpretation of the sums of class numbers, please see section 4.5.3. Participants that did not hand in results for a component are listed as “nt”.

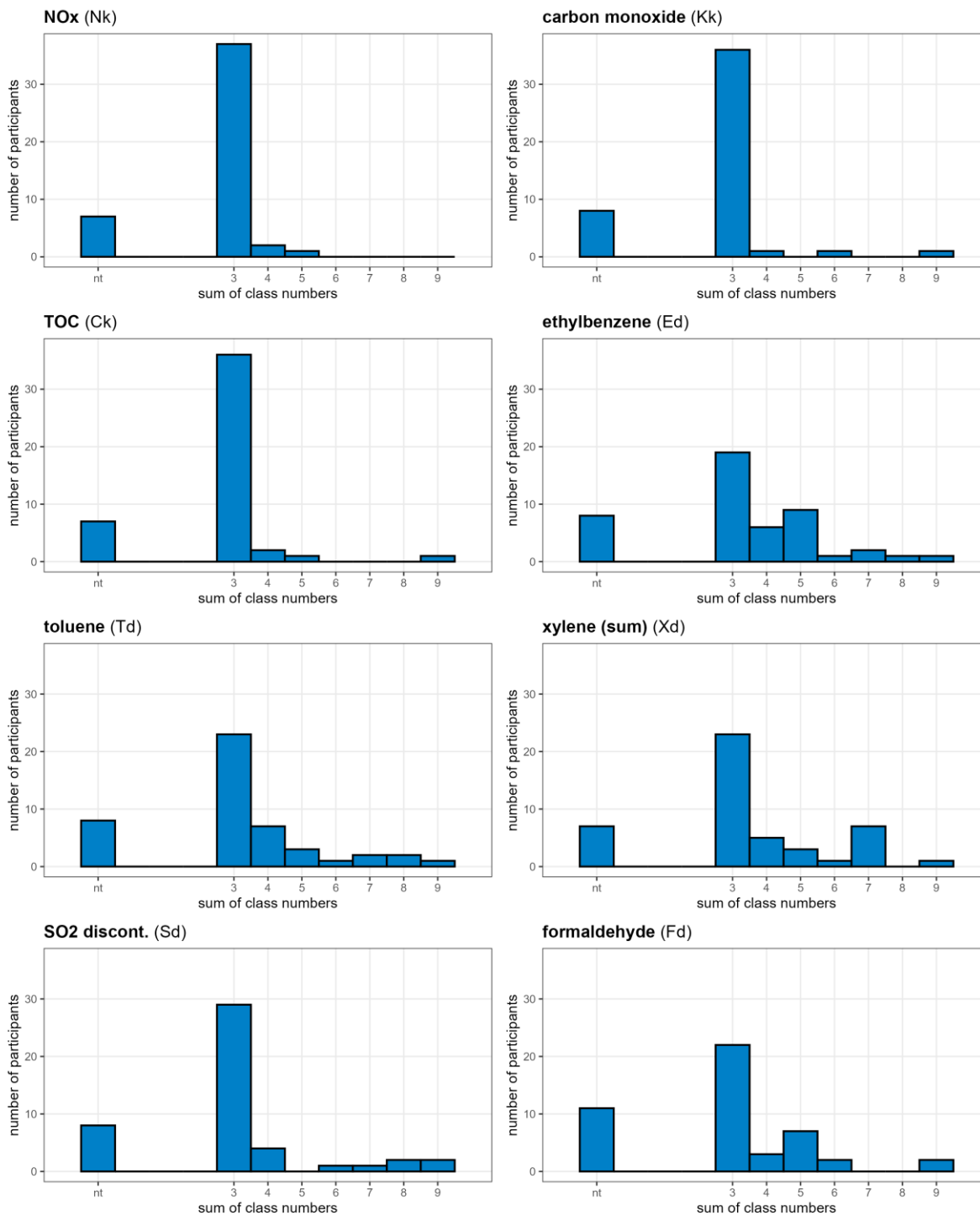
5.2.1 Dust Proficiency Test (Substance Range P)

Sum of Class Numbers



5.2.2 Gas Proficiency Test (Substance Range G)

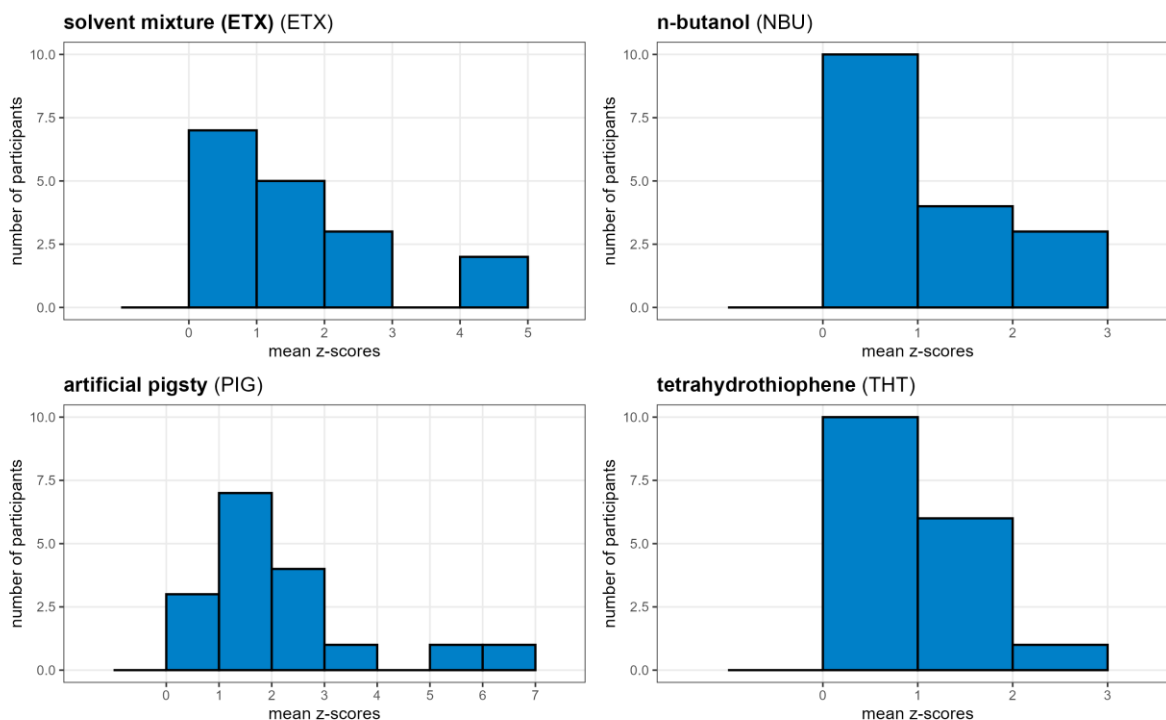
Sum of Class Numbers



5.2.3 Odour Proficiency Test (Substance Range 0)

In odour emission proficiency tests, instead of sums of class numbers a mean value of z-scores is calculated. In the following histograms, the participants are allocated to a group by rounding down their mean z-score to the next lower integer.

Means of z-scores



5.3 Theory Test

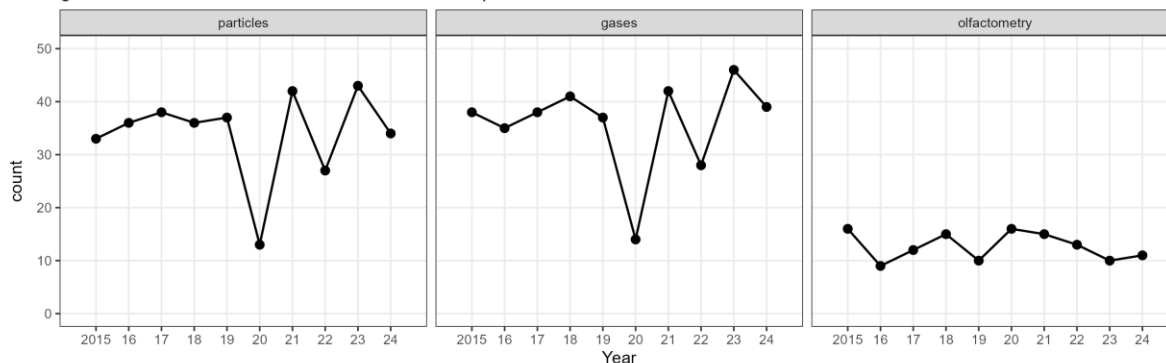
The new specifications of 2019 provide for the performance of a theory test for the dust and gas proficiency tests, which took the form of a 30-minute written test (available in German language only). One person per participating laboratory could take part in this theory test. The contents of the tests for all participants were the requirements of the standards and guidelines applied in the respective proficiency testing scheme. For the execution of the test, each participant was provided with a folder containing the standards as a reference book. Other aids, in particular technical ones, were not permitted. The test consisted of a total of 15 questions, which were weighted with 1 to 3 points. The number of points depends on the difficulty of the question and the importance of the question for the reliability of measured values in emission measurements. In total, a maximum of 33 points could be achieved in the test. There were 4 possible answers to each question, only one of which was correct. For correct answers, participants received the full number of points allocated to the question; for incorrect answers, they received no points. The test was graded as "passed" overall if at least half of the maximum possible score was achieved. If less than half of the maximum points were achieved, the test was graded as "failed". The test was divided into 3 thematic sections, for each of which section-specific assessments were made. In each section, 5 questions on one standard were to be answered. The individual scores of the thematic sections of the test had no effect on the overall result.

A total of 81% of all participants passed the theory test for the dust proficiency test in 2024, with a median score of 23 out of 33 points. The bottom quarter of participants scored 19 points or less, while the top quarter scored 27 points or more.

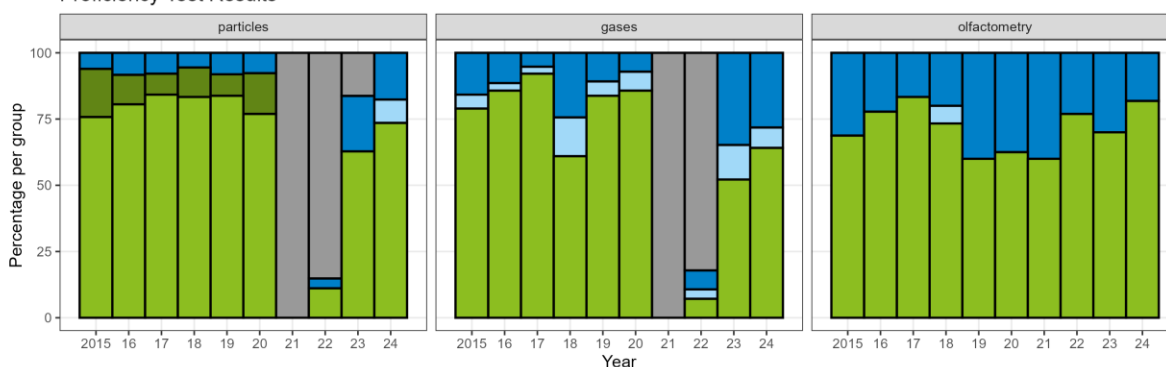
A total of 98% of all participants passed the theory test for the gas proficiency test in 2024, with a median score of 25 out of 33 points. The bottom quarter of participants scored 22 points or less, while the top quarter scored 27 points or more.

6. Interpretation of Results

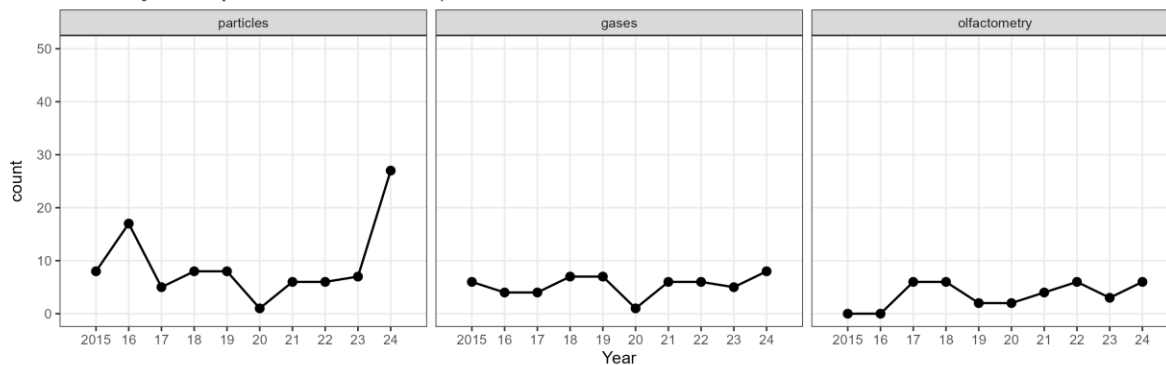
§29b Measurement Bodies Number of Participants



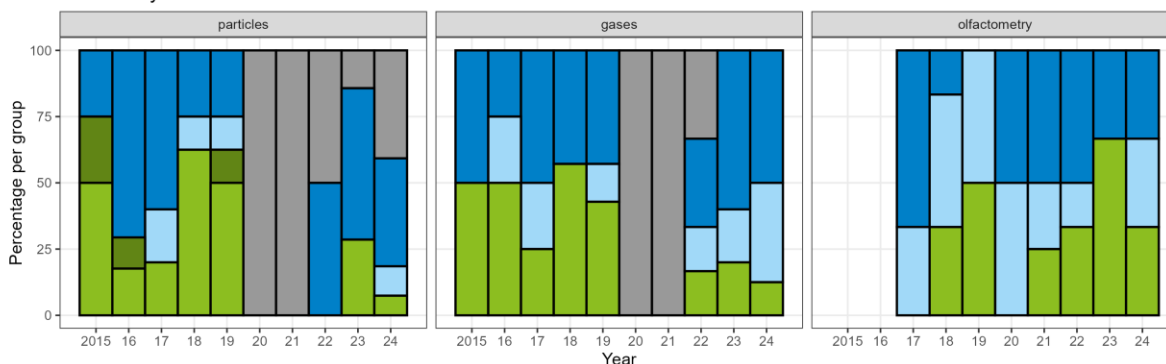
Proficiency Test Results



Voluntary Participants Number of Participants



Proficiency Test Results



no evaluation
 failed
 failed (incomplete participation)
 passed (via post-analysis)
 passed

Table 8: Overview of results since 2024 (§29b-bodies)

year	proficiency test	passed	passed (post analysis)	failed	incomplete participation	not evaluated
2020	dust	10	2	1	-	-
	gas	12	-	1	1	-
	odour	10	-	6	-	-
2021	dust (pandemic)					42
	gas (pandemic)					42
	odour	9	-	6	-	-
2022	dust	3	-	1	-	-
	dust (pandemic)					23
	gas	2	-	2	1	-
	gas (pandemic)					23
	odour	10	-	3	-	-
2023	dust	27	-	9	-	7
	gas	24	-	16	6	-
	odour	7	-	3	-	-
2024	dust	25	-	6	3	-
	gas	25	-	11	3	-
	odour	9	-	2	-	-

Table 9: Overview of results since 2024 (voluntary participants)

year	proficiency test	passed	passed (post analysis)	failed	incomplete participation	not evaluated
2020	dust	-	-	-	-	-
	gas	-	-	-	-	-
	odour	-	-	1	1	-
2021	dust (pandemic)					6
	gas (pandemic)					6
	odour	1	-	2	1	-
2022	dust	-	-	3	-	-
	dust (pandemic)					3
	gas	1	-	2	1	-
	gas (pandemic)					2
	odour	2	-	3	1	-
2023	dust	2	-	4	-	1
	gas	1	-	3	1	-
	odour	2	-	1	-	-
2024	dust	2	-	11	3	-
	dust (short version)					11
	gas	1	-	4	3	-
	odour	2	-	2	2	-

6.1 §29b Measuring Bodies

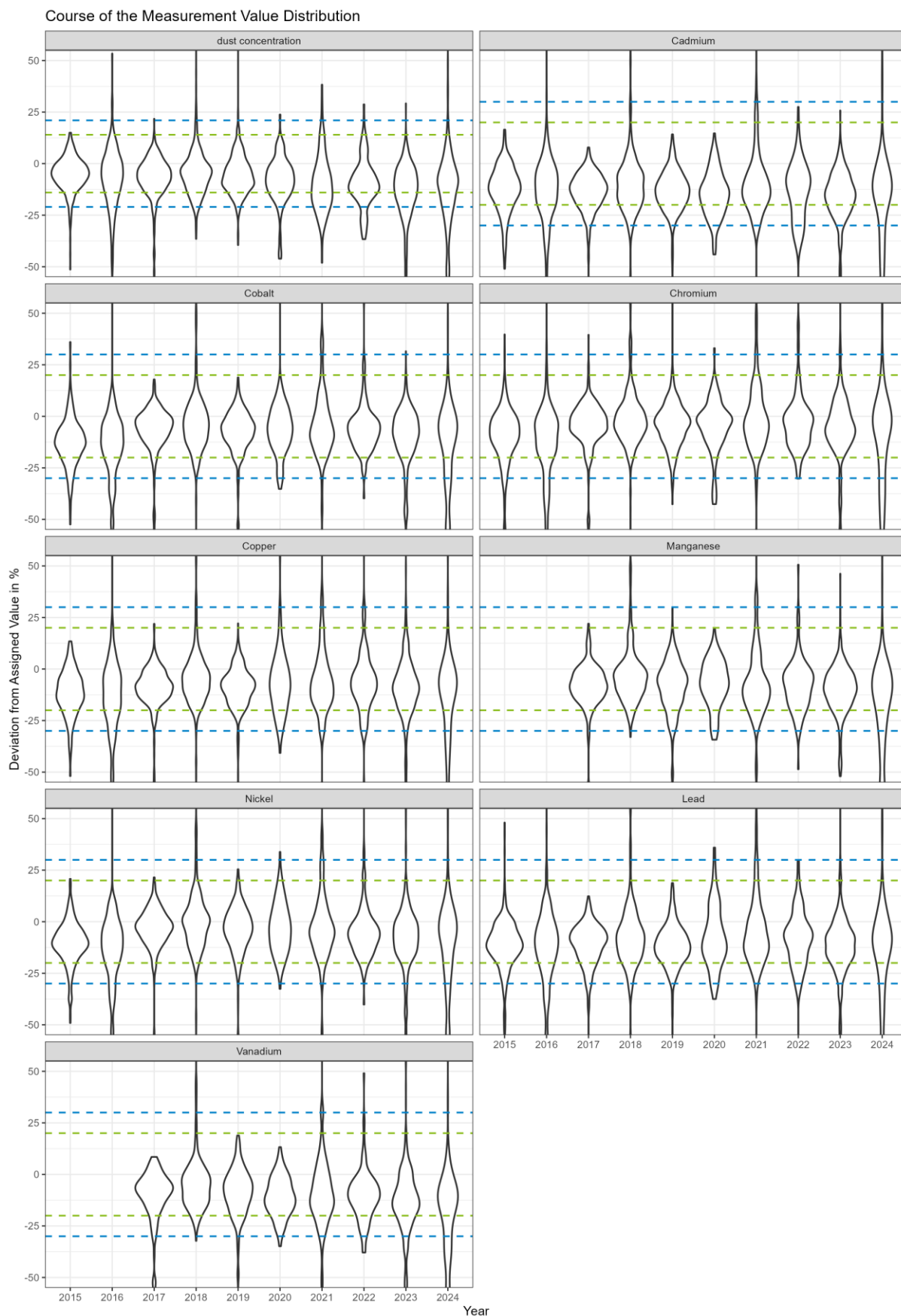
A total of 25 out of 34 (74%) of the authorised measuring bodies passed the dust proficiency test. Six (18%) of the authorised measuring bodies failed. A further 3 (8%) only submitted part of the measurement results as part of a repeat participation after a failed proficiency test, but passed them all. If these participants, which are counted as "failed" proficiency tests for formal reasons, are disregarded, 81% of the authorised measuring bodies passed the proficiency test. The pass rate was thus above the 2023 figure (75%), and once again reached the average value of approx. 82% from 2016 to 2020 (before the SARS-CoV-2 pandemic).

The gas proficiency test was passed by 25 out of 39 (64%) of the authorised measuring bodies. 11 (28%) of the authorised measuring bodies failed. A further 3 (9%) only took part in selected components as part of a repeat participation after a failed proficiency test and passed these. If these participants, which are counted as "failed" proficiency tests for formal reasons, are disregarded, 69% of the notified measuring bodies passed the proficiency testing scheme. The pass rate was therefore higher than the previous year's figure (60%), but once again well below the average figure from 2016 to 2020 before the SARS-CoV-2 pandemic (approx. 87%).

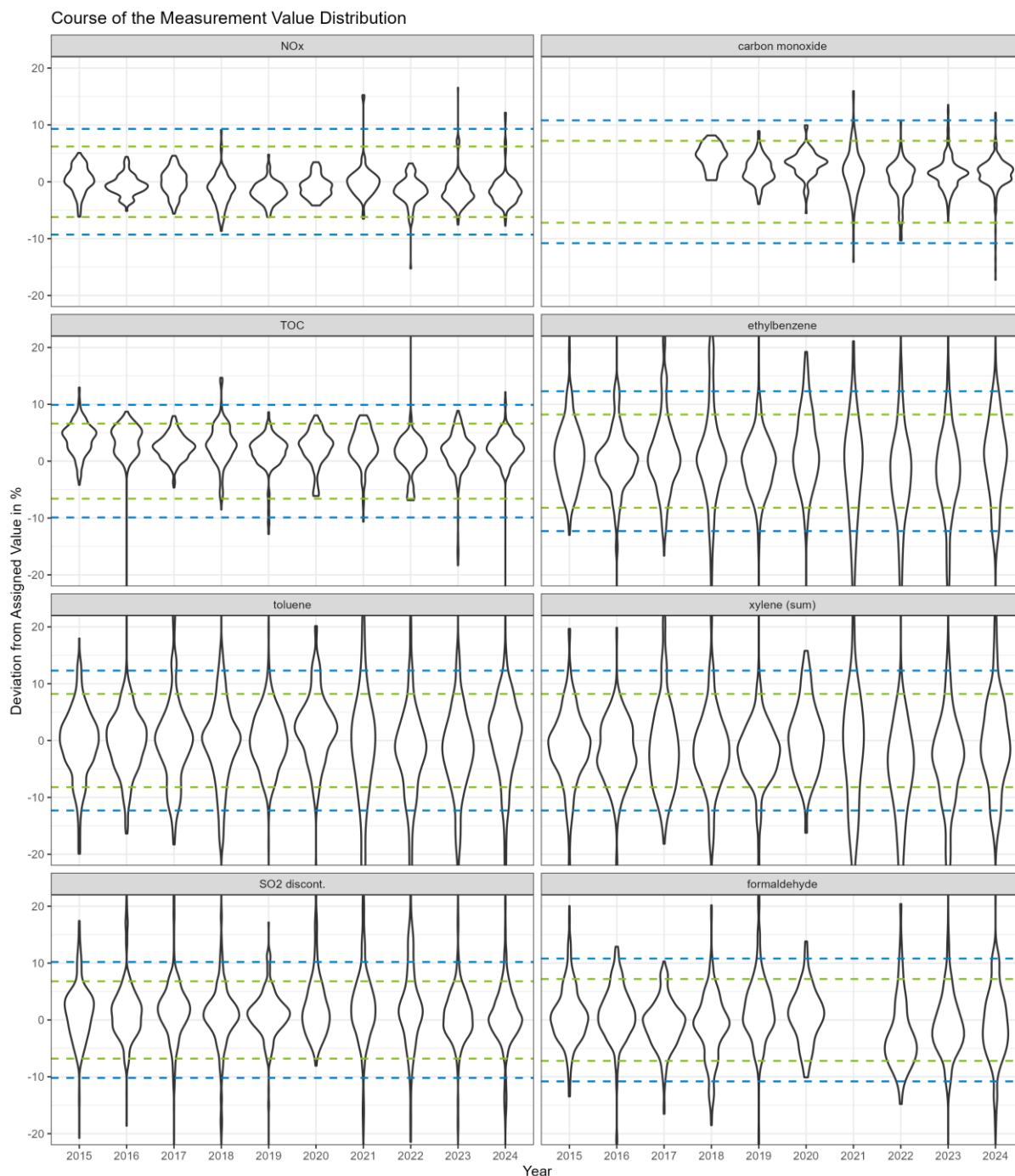
A comparison with the results from 2015 to 2020 shows that the relative deviations of the measured values from the target values for many dust and gas components in 2024 were again greater on average than in the years before the pandemic. Schemes 6 and 7 show a graphical representation of the distribution of the measured values in the proficiency tests of recent years. Here, the distribution of the measured values for each component and for each year since 2015 is shown according to their deviation from the respective target value in the form of a "violin plot", a combination of a "box plot" and a "kernel density plot". The wider the shape shown, the more measurement results are in the relevant range.

A key finding of the HLNUG investigations into dust sampling in accordance with DIN EN 13284-1 is that two factors are essentially responsible for the lower findings observed in our proficiency tests: Deviations from isokinetics and the use of non-sharp-edged probes (20). The condition of the probe tips used in the dust proficiency test is now documented photographically. In fact, these images confirm that the use of clearly non-sharp-edged probe tips, e.g. with dents or notches or generally with thicker-than-average edges, leads to significantly below-average measurement results. According to theory, thick edges lead to turbulence at the probe tip, which results in reduced recovery rates, an effect that HLNUG was able to confirm with its own measurements. Asymmetrical damage (dents and notches) is likely to cause a similar, probably even more pronounced effect. In fact, a striking number of the unsuccessful participants in the proficiency test used thick-edged or even damaged probes.

In recent years, the results of the gas proficiency test have shown a similar trend to the dust proficiency test, although in this case limited to the discontinuous components, in particular the organic substances ethylbenzene, toluene and xylene. While there were consistently very good results for the continuous components NO_x, CO and TOC, the measurement results for the individual organic substances in the years 2021 to 2024 deviated significantly from the values for the years 2015-2020, similar to dust (see Scheme 7).



Scheme 6: Course of the measurement value distribution in the dust proficiency tests 2015-2024 (all participants)



Scheme 7: Course of the measurement value distribution in the gas proficiency tests 2015-2024 (all participants)

A total of 9 out of 11 authorised measuring bodies (82%) passed the odour proficiency test in 2024. The results of the odour proficiency test were therefore better than in all previous years since 2017, although the comparatively low number of participants means that the pass rate fluctuates greatly depending on the results of individual participants.

In view of the uncertainty of individual panel member results, the number of panelists used - usually 4 - is clearly too low from a statistical point of view and is probably still the main reason for inadequate results in the odour proficiency test.

6.2 Voluntary Participants

The number of voluntary proficiency test participants fluctuates from year to year; on average, there are around 8 participants in the dust proficiency test, around 6 participants in the gas proficiency test and around 4 participants in the odour proficiency test. In the year 2024, however, there was an unusually high number of 27 voluntary participants in the dust proficiency test, 11 of whom took part in the new short version of the dust proficiency test. There were 8 voluntary participants in the gas proficiency test and 6 in the odour proficiency test. Due to the usually low number of voluntary participants in many years, the collected results of a year are extremely influenced by the performance of individual laboratories. A comparison over several years is therefore only of limited value.

In the dust proficiency test, a total of 2 of the 16 voluntary participants (13%) were successful in 2024, 11 (69%) voluntary participants did not pass the proficiency test. A total of 3 (19%) voluntary participants correctly determined the dust masses but did not analyse the heavy metal concentrations. These participants were formally categorised as "failed (incomplete participation)". If these are disregarded, the pass rate for voluntary participants in the dust proficiency test this year was 15%. The 11 participants in the short version were not assessed as passed/failed.

In the gas proficiency test, 1 out of 8 (13%) of the voluntary participants passed the proficiency test, 4 (50%) were unsuccessful. Three further voluntary participants (38%) only passed selected components, while they did not participate in the other components. Formally, these participations are counted as "failed (incomplete participation)". If only the voluntary participants who took part fully in the gas proficiency test are considered, the pass rate is 20%.

In the odour proficiency test, 2 out of 6 voluntary participants (33%) passed, and 2 voluntary participants (33%) failed. Two further voluntary participants (33%) delivered correct measurements, but these were not determined within 6 hours in accordance with guideline VDI 3880. The overall results were therefore classified as "failed (incomplete participation)". Excluding these two participations, the pass rate was 50%.

6.3 Gas Flow Conditions

For each proficiency test, the participants must also determine the gas flow conditions. The values recorded in 2024 (see section 5.1.4) correspond to the observations of previous years: The measured values for temperature (CGT), volume flow (CVF) and flow velocity (CFV) show minimal deviations from the assigned values. The participants generally tend to have higher measurement results for the mean flow velocity (approx. +0.5 m/s). One possible explanation for this is insufficient sealing of the sampling ports during the measurement recording, which leads to an increased volume flow compared to the overall period. However, the fact that the volume flow is generally only measured at 4 points with an identical radius in the duct cross-section may also play a role. Although this method conforms to the standard, it does not necessarily lead to correct values if the flow velocity at the selected radius does not correspond exactly to the mean value of the flow velocity in the entire duct cross-section. The tendency towards increased measurement results for the volume flow is a direct consequence of the deviations in the flow velocity, as the latter variable is calculated from the former. In both cases, however, the deviations observed are usually manageable. For flue gas humidity (CAH) and static pressure (CSP), the measurement results are much more scattered in comparison, here there are larger deviations from the assigned values overall and individual "outliers".

7. Optional Information from Participants

All participants were asked to provide additional information on their measurements on a voluntary basis together with the measurement results. Here, too, the information is not listed; the data received is summarised in tables and presented graphically below. The data basis here is the feedback from participants from the years 2016 to 2024, unless otherwise stated.

For some components, the participants in the proficiency test have a certain freedom in the choice of various process parameters. Based on the participants' voluntary data, an attempt was made to determine correlations between the methods, equipment, etc. used and the results obtained. As 6-9 measurements are always carried out at different concentrations for each component, it is difficult to make a clear statement about the quality of a procedure. For a simple and clear presentation, correlations to the mean deviations of the participants were therefore established, with negative values also being included in the mean value. Furthermore, similar components such as heavy metals or organic solvents were combined to form a common mean value. This type of analysis certainly represents a simplification of the problem and cannot reflect all details. For example, different influences in different concentration ranges or high fluctuations between the individual results of a participant remain completely unconsidered in this evaluation. However, the restriction to the mean values of the participants' deviations allows a simple estimation of the effects of different methods on the mean deviation of the measured values from the assigned value.

A certain stabilisation of the values can be observed for most evaluations. This is ultimately due to the fact that the data basis for the annual report 2024 has only increased slightly, while the values for most analyses hardly differ from those of previous years. As a result, most of the findings are becoming increasingly reliable and meaningful over time.

For all correlations presented in this report, it should be kept in mind that a correlation is merely an indication of a connection, but in no way proves causality. For example, it is entirely conceivable that the participants using a certain device or a certain method could have other things in common that actually have an effect on the measurement results, while the identified commonality does not actually play any role at all.

Another aspect that should be taken into account with this data is that although the figures are representative of the proficiency testing scheme participation, they are not necessarily representative of the respective measurement method. Authorised measuring bodies that do not pass the proficiency test due to high deviations from the assigned values are promptly requested to participate again. As a result of these repeated participations, the measurement results of less reliable measuring bodies are disproportionately included in the data, while the measurement results of very reliable laboratories are underrepresented.

7.1 Probes and Rinsing Procedures in Dust Sampling

For the correlation of probe systems and rinsing procedures, the field of participants in the dust proficiency tests is divided into 6 groups, depending on whether an in-stack probe with or without bend (elbow or gooseneck) is used, and whether this probe is rinsed after each sampling, every working day, or never. Participants who stated that they rinse once at the end of the proficiency test were considered to rinse once at the end of each working day for this analysis.

The data basis in this report covers the results since autumn 2018. In summer 2018, the query about the rinsing procedure was specified in terms of frequency; since then a total of 218 participants have provided relevant information about their rinsing procedure. In previous years,

the survey only asked whether rinsing was carried out (yes/no), so the data is unfortunately not comparable.

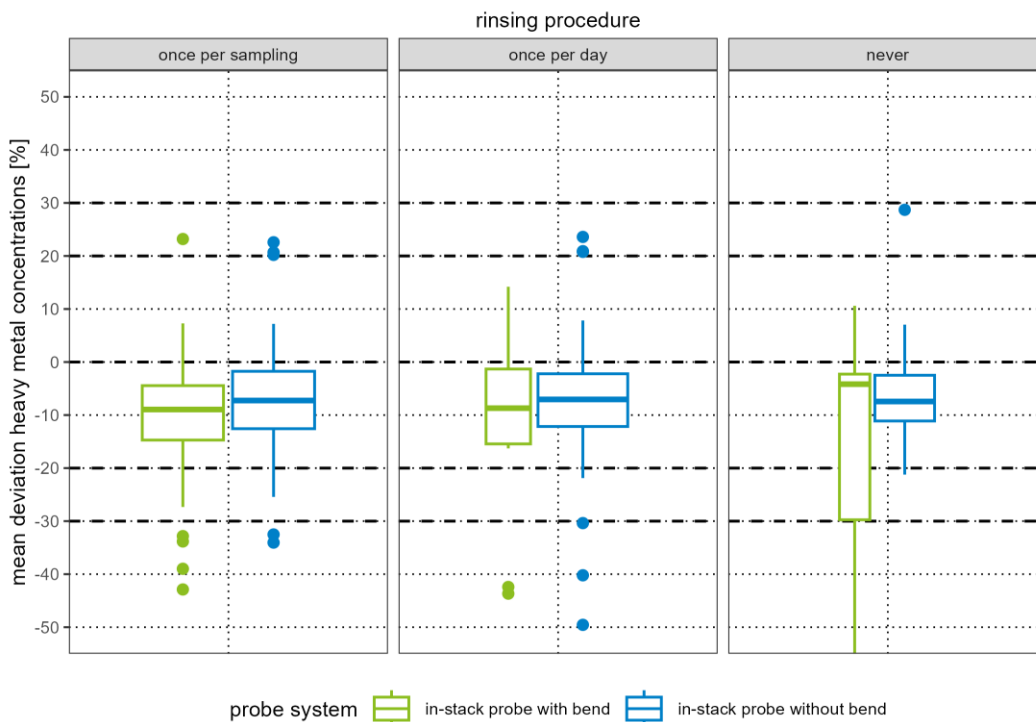
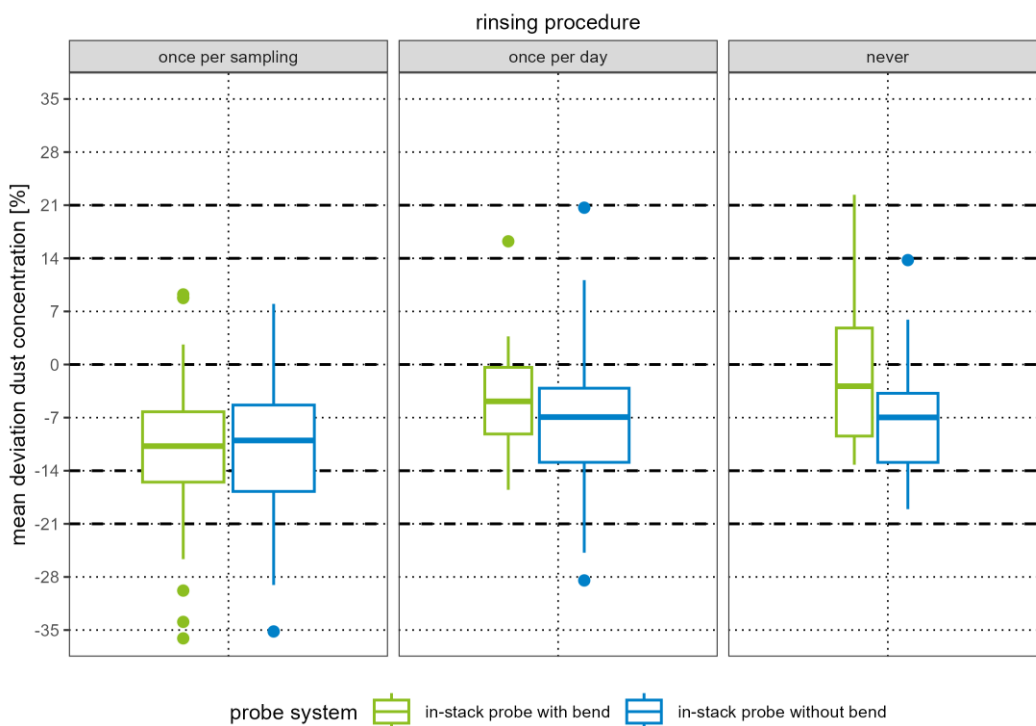


Table 10: Correlation of dust measurement results with probe systems and rinsing procedures (2018-2024)

combination	probe system	rinsing procedure	median of mean deviations total dust results	number of participants	median of mean deviations heavy metal results	number of participants
1 (left)	in-stack probe with bend(s)	after each sampling	-10.8%	51	-8.9%	50
2 (centre)		once per day	-4.9%*	17*	-8.7%*	15*
3 (right)		no rinsing	-2.9%*	10*	-4.2%*	7*
4 (left)	in-stack probe without bend	after each sampling	-10.0%	51	-7.3%	51
5 (centre)		once per day	-7.0%	61	-7.1%	60
6 (right)		no rinsing	-7.0%	28	-7.4%	27

*This combination was only stated by approx. 3-8% of participants. The median is significantly less meaningful here than for the other combinations.

Due to the relatively small number of cases, some of the results shown are significantly influenced by individual results from a small number of laboratories. The above-average results for combination 3 (probe with bend that is not rinsed: right-hand figures, green) are unlikely to be representative of this type of sampling. Combination 3 is explicitly not standard-compliant, as with this probe geometry, dust deposits to the inner surface of the probe are to be expected in any case, which can lead to significantly lower results if rinsing is not carried out.

It is noticeable that participants with a probe without a bend achieve better measurement results for the dust concentration if rinsing is not carried out after every measurement (combination 4, 5 and 6). However, the rinsing frequency hardly seems to have any effect on the measurement results for heavy metals with this type of probe.

Probes with bends perform slightly worse than probes without bend (combination no. 4) for both dust and heavy metals when rinsed after each measurement (combination no. 1). For the other rinsing procedures, there are not enough measurement results available for probes with bends to make a reliable statement.

If, in addition to the rinsing procedure, the data is also narrowed down according to the probe diameter used (see also section 7.2), the trend in the measurement results for total dust between the different probe types becomes even clearer. Selected in this way, the mean deviation in dust measurements for participants who worked with a 10 mm nozzle is -9.1% for combination 1 (32 participants, probe with bend in front of the filter, which is rinsed after each sampling), -9.9% for combination 4 (32 participants, probe without bend, rinsed after each sampling), -8.0% for combination 5 (25 participants, probe without bend, rinsed every working day) and -6.0% for combination 6 (20 participants, probe without bend, no rinsing).

On average, the results of the dust measurements in the proficiency tests of 2024 also show significantly lower results, as did the results of the previous years. The HLNUG has published a detailed investigation of this phenomenon and its probable cause in a scientific journal in 2021. (20).

7.2 Diameter of the Nozzle Opening in Dust Samplings

Based on the data collected since 2016 on the measurement results submitted and the diameters of the probe nozzle openings used (approx. 360 participants), a trend can now be identified. The majority of participants (approx. two thirds) use nozzles with a diameter of 10.0 mm. Most of the remaining participants can be divided into two groups: About a quarter of the participants use probes with a diameter between 8.0 and 9.9 mm. Most of the remaining participants (less than 10%) use a probe diameter of less than 8.0 mm, despite the standard specification to the contrary. A direct comparison shows that the participants with 10 mm probes achieved the best results on average. Similar results were achieved with probe diameters between 8 and 10 mm. However, the results using probe diameters of less than 8 mm are significantly lower and show a higher scatter.

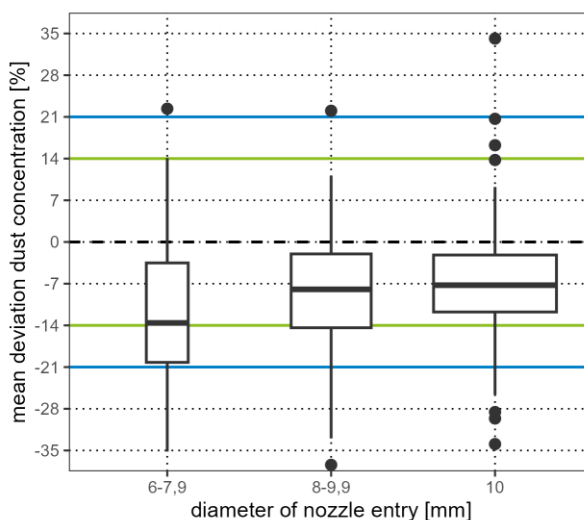


Table 11: Correlation of dust measurement results with nozzle opening diameters (2016-2024)

Diameter of the probe opening	6 to 7.9 mm	8 to 9.9 mm	10 mm
75 th percentile	-3.5%	-2.0%	-2.2%
median	-13.6%	-8.0%	-7.3%
25 th percentile	-20.2%	-14.4%	-11.8%
number of values	26	95	224

Probe diameters < 6 mm or > 10 mm were mentioned by fewer than 18 participants (or 5% of all participants) and are not listed here.

7.3 Analytical Instruments for Heavy Metals

The information provided by the participants on the analytical instrument used for heavy metal analysis reveals little difference between AAS and ICP users. A total of 37 participants stated that heavy metal analysis was performed using AAS equipment, while 294 participants stated that they used an ICP instrument. On average, all participants achieved comparable z-scores for the heavy metals, regardless of the analytical instrument used. However, the measured values of the ICP users scatter more than those of the AAS users.

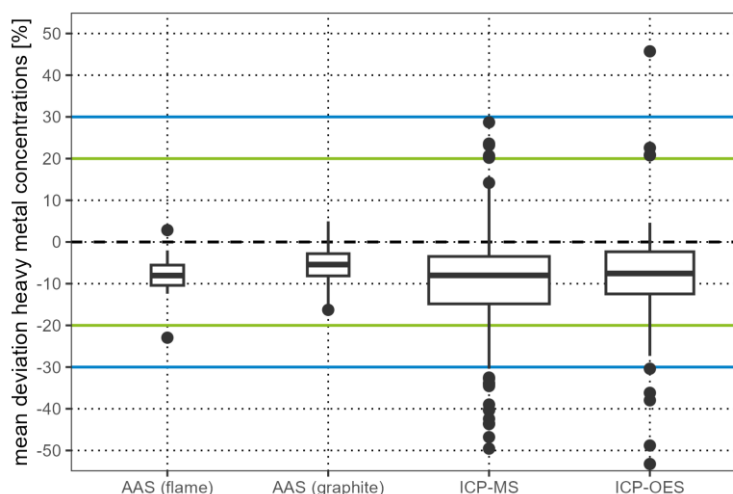


Table 12: Correlation of the mean deviation from the assigned value for heavy metal results and the used analysis devices (2016-2024)

analysis device	flame-AAS	graphite furnace AAS	ICP-MS	ICP-OES
75 th percentile	-5.5%	-2.8%	-3.5%	-2.3%
median	-8.0%	-5.4%	-8.0%	-7.5%
25 th percentile	-10.4%	-8.1%	-14.8%	-12.5%
number of values	14	23	192	102

If the mean recovery of the measured values for heavy metals is corrected by the mean recovery of the measured values for total dust, a similar picture emerges in principle. However, the median values of the deviations in this calculation are around the zero value, which indicates that the lower findings in the total dust are the determining error in the heavy metal concentrations. This observation is not really surprising, as the lack of dust mass in the samples must naturally lead to proportionally lower findings for the heavy metals. The key finding here is that other sources of error probably do not play a major role.

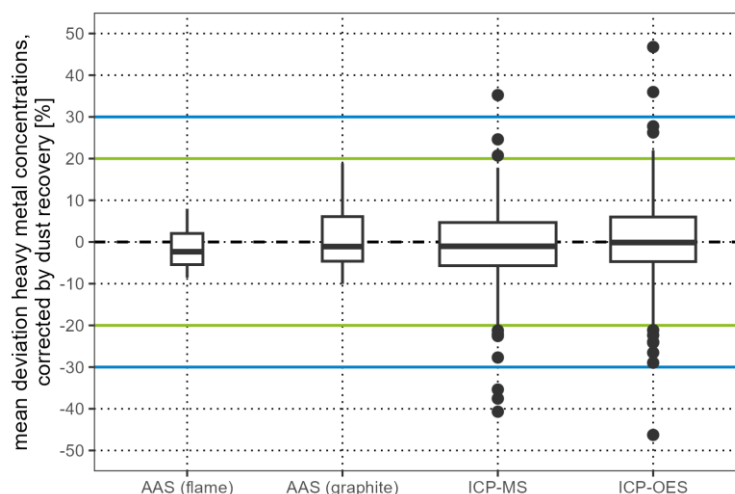


Table13 : Correlation of the mean deviation of the heavy metal concentrations from the target value with the analysers used, corrected in each case for the mean deviation of the total dust concentration (2016-2024)

analysis device	flame-AAS	graphite furnace AAS	ICP-MS	ICP-OES
75 th percentile	+2.0%	+6.1%	+4.7%	+6.0%
median	-2.3%	-1.1%	-1.0%	-0.1%
25 th percentile	-5.4%	-4.6%	-5.7%	-4.7%
number of values	14	23	191	102

7.4 Chemicals in the Digestion Solution

The standardised method sets minimum requirements for digestion for heavy metal analysis, but there is relatively wide latitude in the composition of the digestion solution. Since 2024, participants have therefore been asked to provide information on the combination of chemicals they use. The vast majority of participants use the combination of hydrofluoric acid (HF) and nitric acid (HNO₃) specified as a minimum requirement in the standardised procedure, with around half of them also adding hydrogen peroxide (H₂O₂). The measurement results of these participants are basically similar, but the addition of hydrogen peroxide appears to lead to a greater scattering of results.

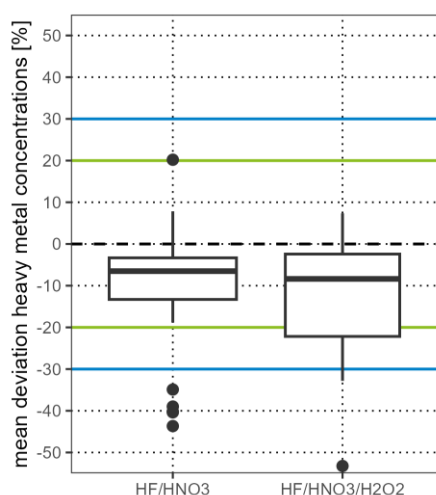


Table14 : Correlation of the mean deviation of the heavy metal concentrations from the target value with the chemicals used in the digestion solution (2024)

Chemicals	HF and HNO ₃	HF, HNO ₃ and H ₂ O ₂
75th percentile	-3.3%	-2.4%
Median	-6.5%	-8.4%
25th percentile	-13.3%	-22.2%
Number of values	26	21

Only a small minority (3 participants, not shown in the diagram) have so far stated that they use a combination of tetrafluoroboric acid (HBF₄), nitric acid (HNO₃) and hydrochloric acid (HCl). The measurement results of this group show unusually large deviations (-43% on average), but only very little data is available here, which also does not come from authorised measuring bodies, but from voluntary participants.

The measurement results for the digestion composition were also analysed with additional correction for the deviations in the respective total dust concentration. Similar to the analysis with regard to the analytic instruments, it can be seen that the lower results observed for the heavy metals can essentially be attributed to the errors in dust recovery.

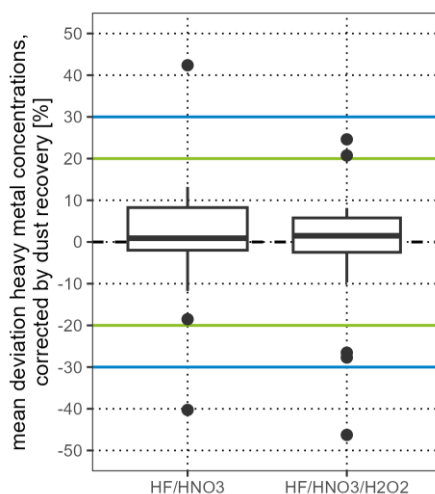


Table15 : Correlation of the mean deviation of the heavy metal concentrations from the target value with the chemicals used in the digestion solution, corrected in each case by the mean deviation of the total dust concentration (2024)

Chemicals	HF and HNO ₃	HF, HNO ₃ and H ₂ O ₂
75th percentile	+8.3%	+5.8%
Median	+0.9%	+1.5%
25th percentile	-2.0%	-2.5%
Number of values	26	21

However, for the three participants who used a combination of tetrafluoroboric acid (HBF₄), nitric acid (HNO₃) and hydrochloric acid (HCl) for the digestion, there was still an average deviation of -19%, even when corrected for the recovery of the total dust concentrations.

7.5 Solvents for Desorption of ETX

For the desorption of the solvents ethylbenzene, toluene and xylene (ETX) the participants can choose between other solvents or solvent mixtures besides the usual solvent carbon disulphide (CS₂). The majority of the participants reported that they had worked with CS₂. The average results of all participants were close to the target value.

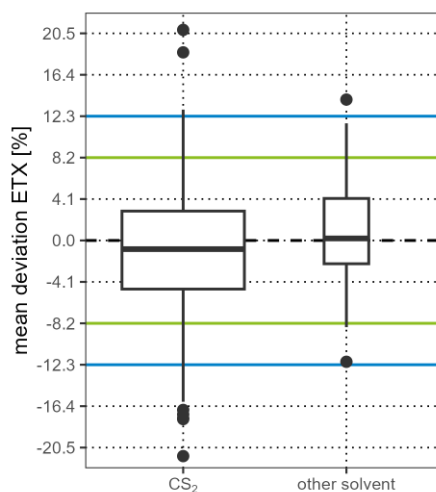


Table 16: Correlation of mean deviations of ETX measurement results with the desorption solvent (2016-2024)

solvent used in desorption	CS ₂	other solvent
75 th percentile	+2.9%	+4.2%
median	-0.9%	+0.2%
25 th percentile	-4.8%	-2.3%
number of values	278	37

7.6 Gas Chromatography Detectors

Gas chromatographs with either an FID detector or a mass spectrometer (MS) are usually used for the analysis of ETX samples. The information provided by the participants gives the following picture:

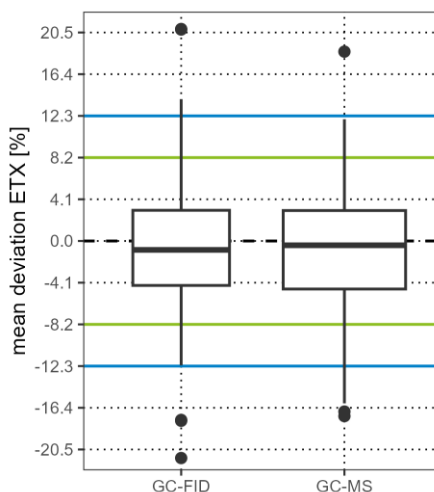


Table 17: Correlation of mean deviations of ETX measurement results with analytical instruments (2016-2024)

analytical instrument	GC-FID	GC-MS
75 th percentile	+3.0%	+3.0%
median	-0.9%	-0.4%
25 th percentile	-4.4%	-4.7%
number of values	121	166

For the overall sampling and analysis procedure, the participants achieved comparable results close to the target value with both detector variants.

Overall, there appears to be a trend towards analysis with GC-MS devices. In the period 2016-2019, 41% of participants still stated that they worked with a GC-FID. In the period 2020-2024, this was only the case for 36% of participants.

It is noticeable here that there has apparently been a recent deterioration in the measurement results of participants using GC-MS detectors. The 25th percentile (i.e. the lower quarter of the results) for GC-MS users is -7.2% if only the results since 2020 are analysed, while the other medians and percentiles have hardly changed. For the measurement results in the period 2016-2019, the 25th percentile was still -3.2%. This difference is due to a number of unusually large underreporting cases that entered the results statistics in recent years. It was not investigated whether this is a deterioration in participants who have been working with GC-MS devices for a long time or participants who have only recently switched from GC-FID to GC-MS.

7.7 Sulphur Dioxide

For the discontinuous determination of sulphur dioxide concentrations, participants can choose between analysis of the samples using ion chromatography or the Thorin method as part of the standard reference method. The following picture emerges from the information provided by the participants:

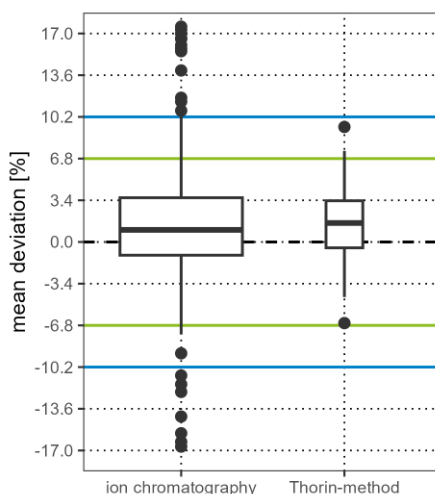


Table 18: Correlation of mean deviations of sulphur dioxide measurement results with the analytical method used (2016-2019)

method	ion chromatography	Thorin-method
75 th percentile	+3,6%	+3,4%
median	+1,0%	+1,5%
25 th percentile	-1,1%	-0,5%
number of values	299	27

No significant difference between the two methods can be recognised in the present results. However, the number of participants using the Thorin method is comparatively small. The somewhat higher dispersion of the IC method with various "outliers" is possibly solely due to the more than 10 times higher number of participants.

7.8 Formaldehyde

For the measurement of formaldehyde concentrations, participants can choose from the guidelines VDI 3862 Parts 2 (16), 3 (17) and 4 (18). Only the procedures according to Part 2 and Part 4 were used by more than 5% of the participants and are therefore shown in the following diagram. The following picture emerges from the information provided by the participants:

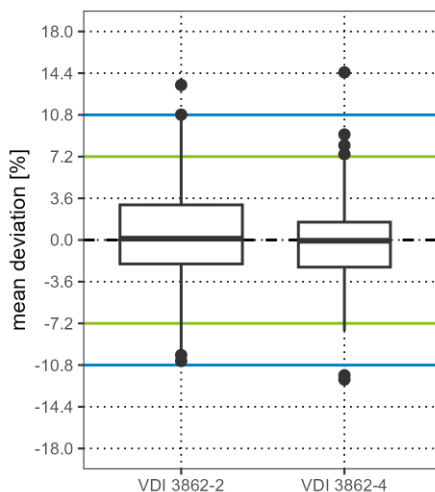


Table 19: Correlation of the mean deviation of the formaldehyde measurement results with the guidelines used (2016-2024)

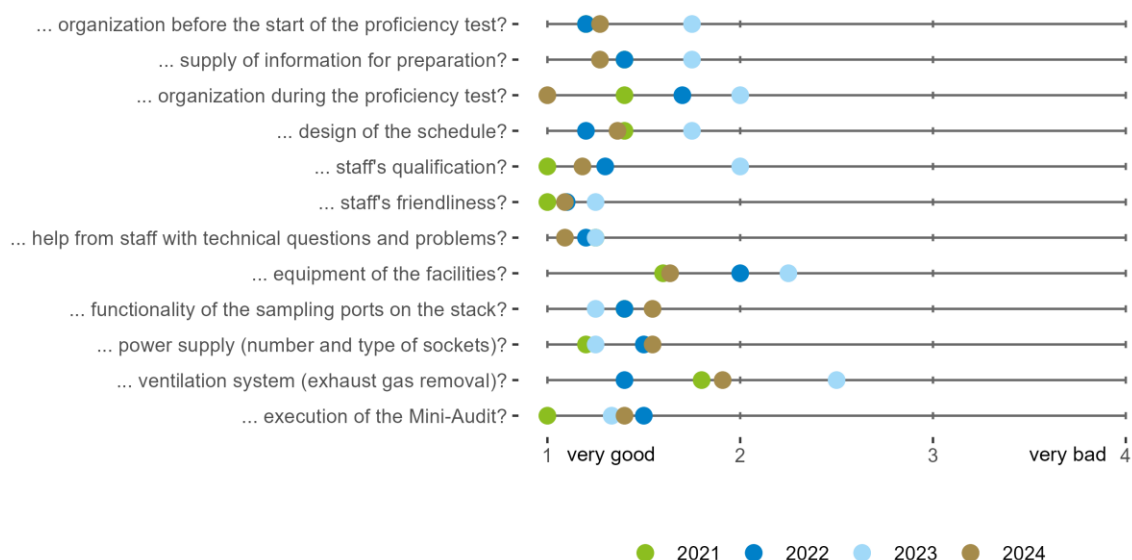
guideline (determination method)	VDI 3862 Part 2 (DNPH wash bottles)	VDI 3862 Part 4 (AHMT-procedure)
75 th percentile	+3,0%	+1,5%
median	+0,1%	-0,1%
25 th percentile	-2,1%	-2,3%
number of values	138	78

The DNPH wash bottle method apparently delivers values comparable to the AHMT method on average.

7.9 Feedback from Participants

Since 2019 HLNUG provides an online feedback questionnaire for its proficiency test participants. The possible ratings for the questions range from 1 (very good), over 2 (rather good), 3 (rather bad) to 4 (very bad). The mean value for the answers to the respective question is shown in the following scheme.

How do you rate the ...



There was a total of 11 responses last year. As in previous years, the feedback received in 2024 showed that the participants were generally very satisfied with the organisation of the proficiency testing scheme. The participants were particularly satisfied with the organisation during the proficiency testing scheme (average grade: 1.0), the friendliness of the staff and the assistance provided by the staff with technical questions and problems (average grade: 1.1 in each case). As in the previous year, the lowest ratings were given to the ventilation system (average rating: 1.9) and the equipment in the rooms (average rating: 1.6).

One participant complained that the door code did not work most of the time. There was apparently a technical problem with a stuck number key, which led to repeated incorrect entries.

Another participant noted that carrying out the formaldehyde measurements on Thursday was problematic for participants with long journeys, as the AHMT procedure requires the analysis to be carried out within 48 hours. However, these measurements were scheduled for Thursday for this very reason, as very few participants have the capacity to have the samples brought to the laboratory during the ongoing proficiency test. After the measurements on Thursday, the samples can be delivered to the laboratory in person by the samplers either on Thursday evening or Friday morning. With a measurement start on Thursday at approx. 9.30 a.m., the analysis for the AHMT procedure for the first samples must be completed by 10.00 a.m. on Saturday. Given the usual travel routes of our participants, we assume that the samples will realistically reach the laboratory by 12:00 noon on Friday and can be processed and analysed during Friday afternoon. If the formaldehyde measurements in the proficiency test were brought forward to Wednesday, a few laboratories would have an advantage if the samples were transported to the laboratory by courier. In this case, the samples would have to be analysed by Friday morning. The working hours in the laboratory would therefore essentially shift from Friday to Thursday. All other participants would

suddenly have the problem that transport by the samplers would no longer be practicable. At most, the samples could be processed on Friday morning under time pressure if the return journey was made on Thursday evening. A return journey on Friday would no longer be an option. For most participants, moving the formaldehyde measurements from Thursday to Wednesday would therefore be the worse alternative.

One participant in the odour proficiency test requested that HLNUG provide a pump for emptying the sampling bags during conditioning. However, we only provide the emission source in the proficiency test; all aspects of sampling are the responsibility of the participants. This also includes all equipment that is not usually provided by the plant operator at the sampling location.

There was also criticism of the fact that no free coffee was offered to participants despite the relatively high participation fees. From a purely financial point of view, we completely agree with this, but unfortunately a difficulty arises here from the fact that HLNUG is not a private company, but a public authority. According to the legal interpretation of our administration, the authorised measuring bodies are obliged by law to participate in our proficiency test. As participation is therefore not a free decision, at least for these companies, the fees must not include any services that are not absolutely necessary for the fulfilment of the purpose. Therefore, according to this interpretation, we cannot use even a small part of the fees to provide coffee or other drinks to the participants on site. If the provision of drinks for the participants is desired, another method of financing must therefore be found.

Another participant pointed out that the assessment criteria in the gas proficiency test were in some cases significantly more demanding than the minimum requirements for measurement uncertainty in accordance with the 17th Federal Immission Control Ordinance (17. BImSchV, based on the IED). It should be noted that the various standard reference methods only formulate minimum requirements with regard to measurement uncertainty. However, the assessment criteria in our proficiency tests are based on the expectations of the notifying authorities and the responsible higher-level environmental authorities. For the monitoring of emissions in Germany, it is expected that the measurements required for this are carried out according to the current state of the art. When carried out by experts and using appropriate equipment, most methods achieve measurement uncertainties that are significantly better than the minimum requirements of the standard. This is reflected accordingly in the assessment criteria of our proficiency tests.

HCl, HF, mercury and significantly higher water vapour concentrations were proposed as additional components. We are already working on increased moisture concentrations. However, HCl, HF and mercury are not so easy to integrate into our proficiency testing programme. However, if the desire to include these components in the programme should arise more frequently in the future, implementation at the ESA can be examined.

8. Concluding Remark

The measurement results in the dust and gas proficiency tests have generally improved in 2024 compared to 2023. However, the measurement results for many components are still worse than in the years before the SARS-CoV-2 pandemic. This mainly concerns the discontinuous components such as dust and the individual organic substances (ethylbenzene, toluene and xylene). In the case of total dust, in addition to errors in isokinetics, the use of thick-edged and/or damaged probe tips could be a possible cause of inadequate results. In the case of organic components, the error in many cases is probably not to be found in the sampling, but rather in the sample preparation and above all in the analysis.

In the odour proficiency test, the participants once again achieved significantly better results overall in 2024 than in 2019 to 2021, with the pass rate, similar to 2022 and 2023, back at the level of 2016 to 2018. As before, the main problem for participants in the odour proficiency test is likely to be the use of panels of only 4 test subjects. Under these circumstances, the measurement results of individual panel members have a massive influence on the sample result, which means that daily fluctuations in the perception of these individuals can easily lead to the failure of the entire laboratory.

During the last revision of the specifications for our dust and gas proficiency tests in 2019, we aimed to reduce the number of assessed measurements from 9 to 6. In our experience, the overall result is not affected by the cancellation of 3 measurements. Unsuccessful participations are almost always characterised by systematic errors that occur regardless of the number of measurements. For the measuring personnel, this shortening would have had the advantage of significantly relaxing the schedule, which would have made travelling on a Sunday unnecessary, for example. Unfortunately, this request failed due to strong opposition from representatives of the measuring bodies. In order to make a better proficiency testing offer possible, at least for the voluntary participants, we have now further developed the programme originally planned for all measuring bodies as a "short version" of the dust and gas proficiency test. This version of the interlaboratory comparison, which has been shortened to 6 evaluated measurements, is now offered to those participants who do not wish to be authorised in accordance with the 41st BImSchV. This short version is planned with measurements only from Tuesday to Thursday, leaving Monday and Friday completely free for arrival and departure. As the short version does not fulfil the requirements of the LAI specifications and therefore cannot be used for an authorisation in accordance with the 41st BImSchV, this new programme unfortunately has no benefit for the majority of our participants, the authorised measuring bodies. However, we have recently observed increased interest in our proficiency tests from countries such as Austria, Italy and Greece. These laboratories generally do not aim for an authorisation in Germany and may well be interested in a shortened proficiency testing programme.

Kassel, 27th March 2025

gez. J. Cordes

Dr. Jens Cordes

Technical Supervisor
Proficiency Testing

(Fachlich Verantwortlicher
Ringversuche)

gez. B. Stoffels

Benno Stoffels

Deputy Technical Supervisor
Proficiency Testing

(Stellvertretender Fachlich
Verantwortlicher Ringversuche)

gez. D. Wildanger

Prof. Dr. Dominik Wildanger

Head of Department

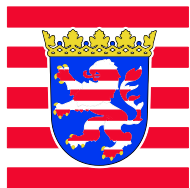
(Dezernatsleiter)

9. References

1. Gesetz zum Schutz vor schädlichen Umwelteinwirkungen durch Luftverunreinigungen, Geräusche, Erschütterungen und ähnliche Vorgänge (Bundes-Immissionsschutzgesetz – BImSchG) in der Fassung der Bekanntmachung vom 17. Mai 2013 (BGBl I, 2013, Nr. 25, S. 1274–1311). (*Act on the Prevention of Harmful Effects on the Environment Caused by Air Pollution, Noise, Vibration and Similar Phenomena (Federal Immission Control Act – BImSchG) in the version promulgated on 17 May 2013 (BGBl I, 2013, p. 1274)*)
2. DIN EN ISO/IEC 17043:2010-05 - Konformitätsbewertung - Allgemeine Anforderungen an Eignungsprüfungen (ISO/IEC 17043:2010); Deutsche und Englische Fassung EN ISO/IEC 17043:2010. (*Conformity assessment - General requirements for proficiency testing (ISO/IEC 17043:2010); German and English version EN ISO/IEC 17043:2010*). Berlin : Beuth-Verlag.
3. Einundvierzigste Verordnung zur Durchführung des Bundes-Immissionsschutzgesetzes (Bekanntgabeverordnung – 41. BImSchV) vom 2. Mai 2013 (BGBl I, 2013, Nr. 21, S. 1001–1010). (*Forty-first Ordinance on the implementation of the Federal Immission Control Act (Ordinance on authorisation – 41st BImSchV) of 2 May 2013 (BGBl I, 2013, p. 973)*)
4. J. Cordes, B. Stoffels, D. Wildanger. *The question of homogeneity inside a chimney: application of ISO 13528 to stack emission proficiency tests. Accred Qual Assur.* 20, 2015, p. 287.
<https://doi.org/10.1007/s00769-015-1139-y>
5. S. Stöckel, J. Cordes, B. Stoffels, D. Wildanger. *Scents in the stack: olfactometric proficiency testing with an emission simulation apparatus. Environ. Sci. Pollut. Res.* 25, 2018, p. 24787.
<https://doi.org/10.1007/s11356-018-2515-z>
6. DIN EN ISO 17034:2017-04 - Allgemeine Anforderungen an die Kompetenz von Referenzmaterialherstellern (ISO 17034:2016); Deutsche und Englische Fassung EN ISO 17034:2016. (*General requirements for the competence of reference material producers (ISO 17034:2016); German and English version EN ISO 17034:2016*). Berlin : Beuth-Verlag.
7. DIN ISO 13528:2020-09 - Statistische Verfahren für Eignungsprüfungen durch Ringversuche (ISO 13528:2015, korrigierte Fassung 2016-10-15); Text Deutsch und Englisch. (*Statistical methods for use in proficiency testing by interlaboratory comparisons (ISO 13528:2015, Corrected version 2016-10-15); Text in German and English*). Berlin : Beuth-Verlag.
8. DIN EN 14385:2004-05 - Emissionen aus stationären Quellen - Bestimmung der Gesamtemission von As, Cd, Cr, Co, Cu, Mn, Ni, Pb, Sb, TI und V; Deutsche Fassung EN 14385:2004. (*Stationary source emissions - Determination of the total emission of As, Cd, Cr, Co, Cu, Mn, Ni, Pb, Sb, TI and V; German version EN 14385:2004*). Berlin : Beuth-Verlag.
9. DIN EN 15259:2008-01: Luftbeschaffenheit - Messung von Emissionen aus stationären Quellen - Anforderungen an Messstrecken und Messplätze und an die Messaufgabe, den Messplan und den Messbericht; Deutsche Fassung EN 15259:2007. (*Air quality - Measurement of stationary source emissions - Requirements for measurement sections and sites and for the measurement objective, plan and report; German version EN 15259:2007.*). Berlin : Beuth-Verlag.
10. DIN EN 13284-1:2018-02: Emissionen aus stationären Quellen - Ermittlung der Staubmassenkonzentration bei geringen Staubkonzentrationen - Teil 1: Manuelles gravimetrisches Verfahren; Deutsche Fassung EN 13284-1:2017. (*Stationary source emissions. Determination of low range mass concentration of dust. Manual gravimetric method; German version EN 13284-1:2017.*). Berlin : Beuth-Verlag.
11. DIN EN 14792:2017-05 - Emissionen aus stationären Quellen - Bestimmung der Massenkonzentration von Stickstoffoxiden - Standardreferenzverfahren: Chemilumineszenz; Deutsche

- Fassung EN 14792:2017. (*Stationary source emissions. Determination of mass concentration of nitrogen oxides. Standard reference method. Chemiluminescence; German version EN 14792:2017.*). Berlin : Beuth-Verlag.
12. DIN EN 15058:2017-05 - Emissionen aus stationären Quellen - Bestimmung der Massenkonzentration von Kohlenmonoxid - Standardreferenzverfahren: Nicht-dispersive Infrarotspektrometrie; Deutsche Fassung EN 15058:2017. (*Stationary source emissions. Determination of the mass concentration of carbon monoxide. Standard reference method: non-dispersive infrared spectrometry; German version EN 15058:2017.*). Berlin : Beuth-Verlag.
 13. DIN EN 12619:2013-04: Emissionen aus stationären Quellen - Bestimmung der Massenkonzentration des gesamten gasförmigen organisch gebundenen Kohlenstoffs – Kontinuierliches Verfahren mit dem Flammenionisationsdetektor; Deutsche Fassung EN 12619:2013. (*Stationary source emissions. Determination of the mass concentration of total gaseous organic carbon. Continuous flame ionisation detector method; German version EN 12619:2013.*). Berlin : Beuth-Verlag.
 14. DIN CEN/TS 13649:2015-03 - DIN SPEC 33969:2015-03 - Emissionen aus stationären Quellen - Bestimmung der Massenkonzentration von gasförmigen organischen Einzelverbindungen - Sorptive Probenahme und Lösemittelextraktion oder thermische Desorption; Deutsche Fassung CEN/TS 13649:2014. (*Stationary source emissions. Determination of the mass concentration of individual gaseous organic compounds. Sorptive sampling method followed by solvent extraction or thermal desorption; German Version CEN/TS 13649:2014.*). Berlin : Beuth-Verlag.
 15. DIN EN 14791:2017-05 - Emissionen aus stationären Quellen - Bestimmung der Massenkonzentration von Schwefeloxiden - Standardreferenzverfahren; Deutsche Fassung EN 14791:2017. (*Stationary source emissions. Determination of mass concentration of sulphur oxides. Standard reference method; German version EN 14791:2017.*). Berlin : Beuth-Verlag.
 16. VDI 3862 Blatt 2:2000-12 - Messen gasförmiger Emissionen - Messen aliphatischer und aromatischer Aldehyde und Ketone nach dem DNPH-Verfahren - Gaswaschflaschen-Methode. (*Gaseous emission measurement - Measurement of aliphatic and aromatic aldehydes and ketones by DNPH method - Impinger method*). Berlin : Beuth-Verlag.
 17. VDI 3862 Blatt 3:2000-12 - Messen gasförmiger Emissionen - Messen aliphatischer und aromatischer Aldehyde und Ketone nach dem DNPH-Verfahren - Kartuschen-Methode. (*Gaseous emission measurement - Measurement of aliphatic and aromatic aldehydes and ketones by DNPH method - Cartridges method*). Berlin : Beuth-Verlag.
 18. VDI 3862 Blatt 4:2001-05 - Messen gasförmiger Emissionen - Messen von Formaldehyd nach dem AHMT-Verfahren. (*Gaseous emission measurement - Measurement of formaldehyde by the AHMT method*). Berlin : Beuth-Verlag.
 19. DIN EN 13725:2022-06 - Emissionen aus stationären Quellen – Bestimmung der Geruchsstoffkonzentration durch dynamische Olfaktometrie und die Geruchsstoffemissionsrate; Deutsche Fassung EN 13725:2022. (*Stationary source emissions – Determination of odour concentration by dynamic olfactometry and odour emission rate; German version EN 13725:2022*). Berlin : Beuth-Verlag.
 20. E. Antonsson, J. Cordes, B. Stoffels, D. Wildanger. *The European Standard Reference Method systematically underestimates particulate matter in stack emissions. Atmos. Environ, X, 12, 2021, p. 100133.* <https://doi.org/10.1016/j.aeaoa.2021.100133>

HESSEN



Hessisches Landesamt für Naturschutz, Umwelt und Geologie

Hessian Agency for Nature Conservation, Environment and Geology

Dezernat I3 – Luftreinhaltung: Emissionen

Department I3 – Air Pollution Control: Emissions

Ludwig-Mond-Straße 33

34121 Kassel

– GERMANY –