

Annual Report 2023

Results of stack emission proficiency tests for substance ranges P, G, and O on the emission simulation apparatus in the year 2023

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0. About this Report

This report is a translation of „Jahresbericht 2023 – Ergebnisse der Emissionsringversuche der Stoffbereiche P, G und O an der Emissionssimulationsanlage im Jahr 2023“ 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 50 measuring institutes took part in HLNUG's dust emission proficiency tests (substance range P) in 2023, 43 of which were §29b measuring bodies and 7 volunteers. As in the past, the success rate of the §29b measuring bodies (75%) was significantly higher than that of the volunteers (33%).

A total of 51 measuring institutes took part in the gas emission proficiency tests (substance range G) in 2023, 46 of which were §29b measuring bodies and 5 volunteers. As in previous years, the success rate for the §29b measuring bodies (60%) was significantly higher than for the volunteers (25%).

A total of 13 measuring bodies took part in the odour emission proficiency tests (substance range O) in 2023, 10 of them on the basis of an authorisation in accordance with §29b BImSchG and 3 voluntarily. Here 70% of the authorised participants were successful and 67% of the volunteers.

In version 2 of this annual report, the assigned values for the gas flow conditions on two measurement days and the resulting z-scores were slightly corrected compared to version 1.

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 authorized 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 authorizing 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 authorization in accordance with §29b BImSchG.

Consequently, about 80-90% of the participants are laboratories authorized to perform measurements in accordance with §29b BImSchG (Federal Immission Control Act), or applicants for authorization 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.

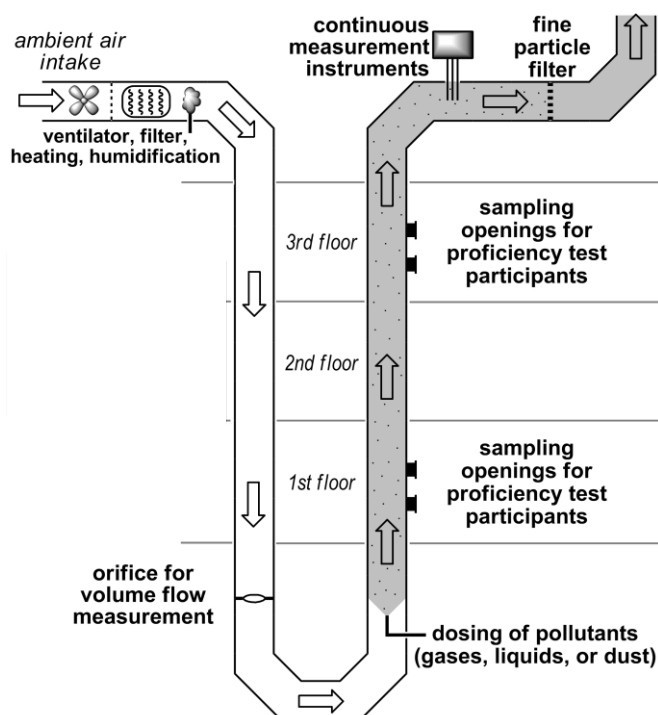
2.2 The Emission Simulation Apparatus

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 2023, 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
23P1	Dust (substance range P)	13.02.2023	14.02.2023	7
23G1	Gas (substance range G)	14.02.2023	16.02.2023	7
23P2	Dust (substance range P)	27.02.2023	28.02.2023	7
23G2	Gas (substance range G)	28.02.2023	02.03.2023	7
23P3	Dust (substance range P)	13.03.2023	14.03.2023	8
23G3	Gas (substance range G)	14.03.2023	16.03.2023	8
23P4	Dust (substance range P)	27.03.2023	28.03.2023	8
23G4	Gas (substance range G)	28.03.2023	30.03.2023	8
23P5	Dust (substance range P)	24.04.2023	25.04.2023	6
23G5	Gas (substance range G)	25.04.2023	27.04.2023	7
23O1	Odour (substance range O)	19.09.2023	19.09.2023	4
23O2	Odour (substance range O)	21.09.2023	21.09.2023	4
23O3	Odour (substance range O)	26.09.2023	26.09.2023	5
23P6	Dust (substance range P)	06.11.2023	07.11.2023	8
23G6	Gas (substance range G)	07.11.2023	09.11.2023	8
23P7	Dust (substance range P)	20.11.2023	21.11.2023	6
23G7	Gas (substance range G)	21.11.2023	23.11.2023	6

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	for each component 9 (+ 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	
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

	dust (substance range P)	gas (substance range G)
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	in ouE/m ³ , rounded to integers	

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 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 organizer, 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 optimized by specific heavy metal doping, grinding, sieving and drying steps. Finally, a complete homogenization 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 table **Fehler! Verweisquelle konnte nicht gefunden werden.** 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: Sequence of the stack emission proficiency tests of substance ranges P, G, and O

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 odours	(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) on the basis of 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 of the measurement, and σ_k is the 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 2023, in addition to *n*-butanol the components ‘organic solvent mixture’ (ETX), tetrahydrothiophene (THT) and artificial pigsty (PIG) were used. For components ETX and PIG, the odour threshold $c_{0,k}$ could be determined with procedure a). For ETX, a value was determined from 210 measurements in the years 2018 to 2022, resulting in a consensus value of $c_0 = 220 \mu\text{g}/\text{m}^3$. For PIG, from 84 measurements in the years 2021 and 2022 a consensus value of $c_0 = 194 \mu\text{g}/\text{m}^3$ was calculated. For the component THT, a consensus value had to be determined via procedure b). Here, a value of $c_0 = 0.486 \mu\text{g}/\text{m}^3$ was calculated on the basis of 30 measurements in 2023.

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 2023 this was necessary for components THT and PIG, where σ_k had to be raised to a value of 0.11 (THT) and 0.17 (PIG), respectively. 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. stand-ard uncertainty of assigned values [%]
P1	dust	St	7.0	1.57
P2	Cadmium	Cd	10.0	1.86
P3	Cobalt	Co	10.0	1.90
P4	Chromium	Cr	10.0	1.88
P5	Copper	Cu	10.0	2.19
P6	Manganese	Mn	10.0	2.01
P7	Nickel	Ni	10.0	1.94
P8	Lead	Pb	10.0	1.86
P9	Vanadium	V	10.0	2.14

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	5.25
O3	tetrahydrothiophene	THT	0.11	7.19
O4	artificial pigsty odour	PIG	0.17	12.1

4.5.3 Assessment Scheme

Interpretation of the z-scores

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

$$\begin{aligned}
 |z_{ijk}| \leq 2 & \quad \text{satisfactory} \\
 2 < |z_{ijk}| < 3 & \quad \text{questionable} \\
 |z_{ijk}| \geq 3 & \quad \text{unsatisfactory}
 \end{aligned}$$

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 $n = 3$) 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 $K_{jk} = 3$

For each component at least 6 measurement results must be submitted, otherwise the respective component is automatically evaluated as „failed“.

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”.

For the proficiency tests in the pandemic version, no overall assessment took place.

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 component volume flow was determined successfully, if

$$z_k < 3$$

is fulfilled. In this case, the component is rated “passed”. If this criterion is not met, the component is rated “failed”. If no measurement values were submitted, the component is rated “no participation”.

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

Communication of the evaluation of the participants’ results by HLNUG is done within six weeks after the last day for submission of results for the respective proficiency test. This evaluation is given to the participants in form of a general survey, including tables and diagrams, and quoting their unique ID-code.

5. Results

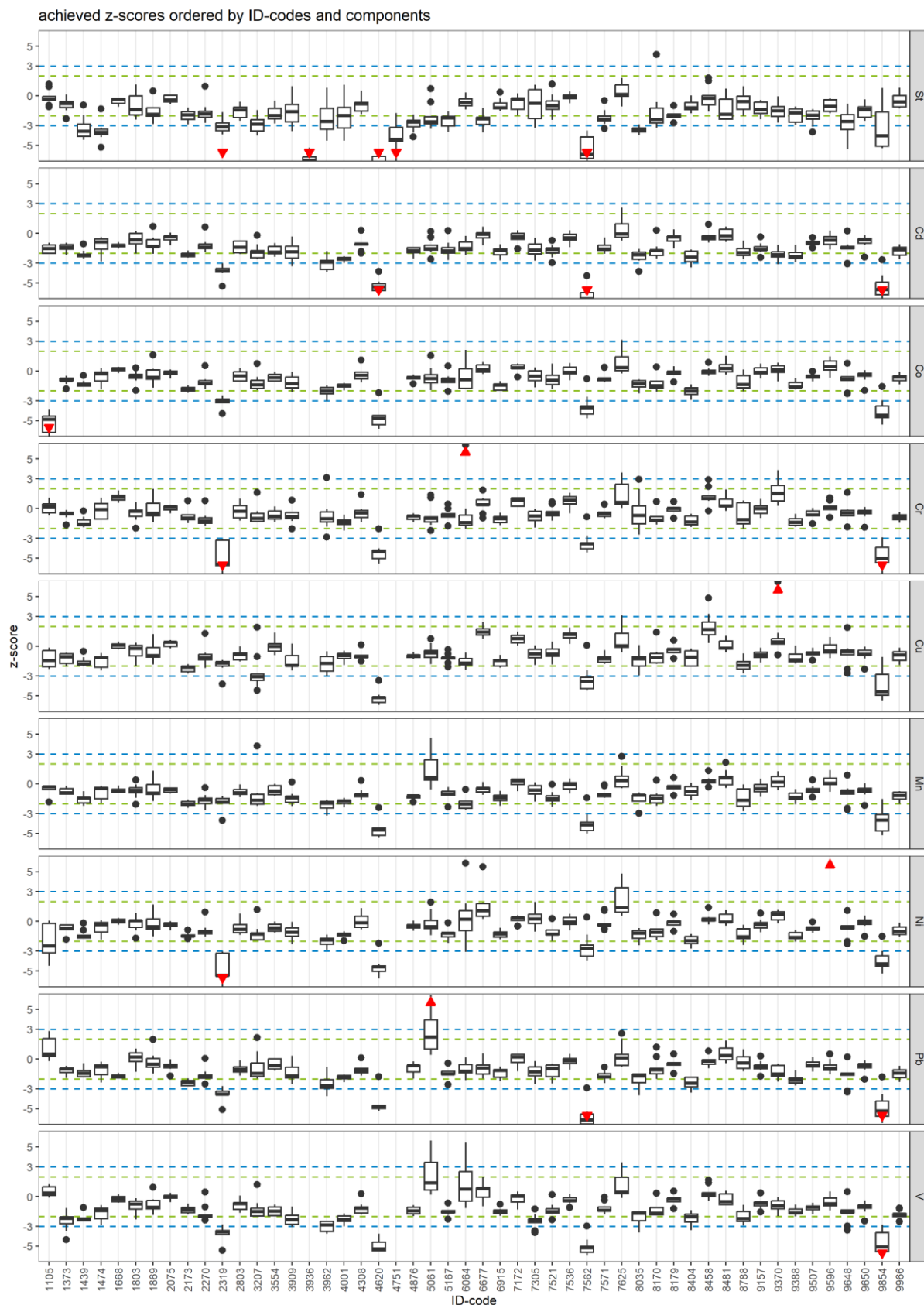
5.1 z-Scores

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 points in the diagram.

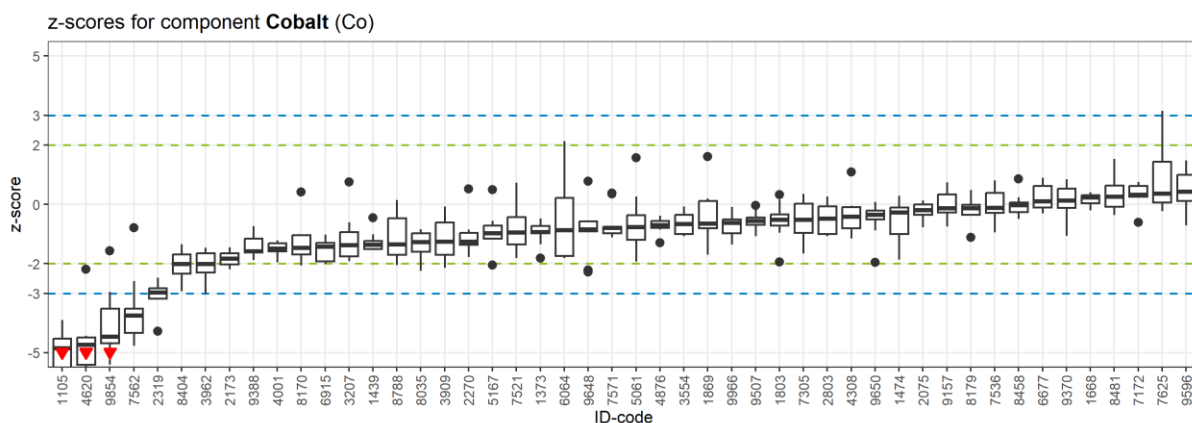
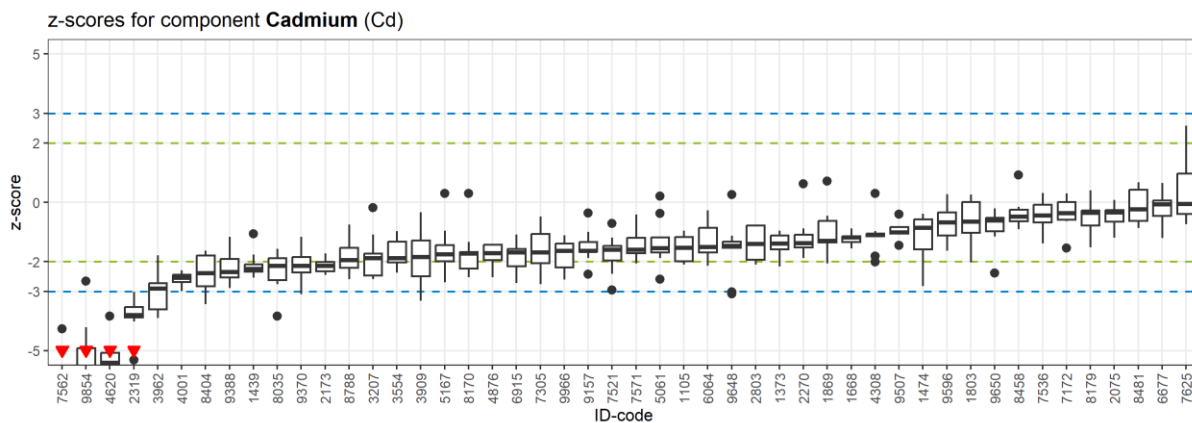
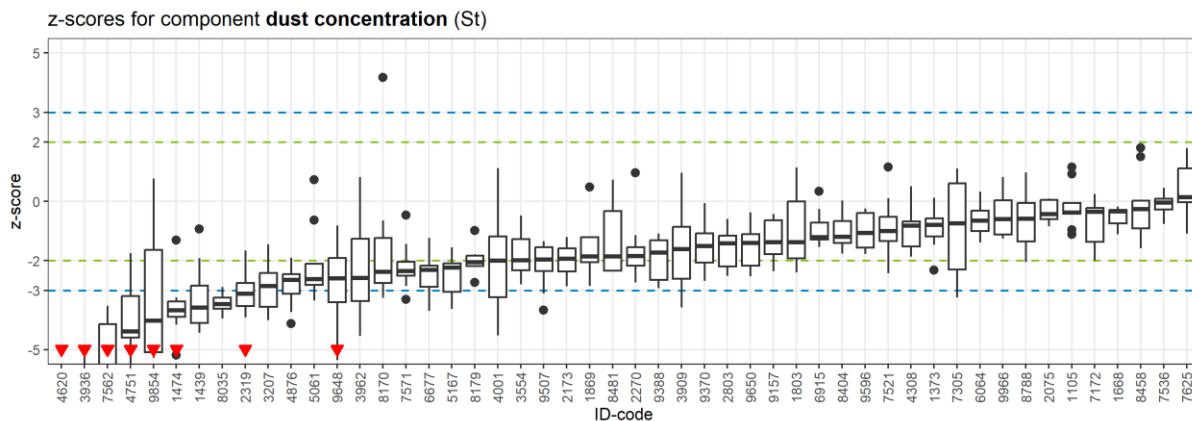
In order to be able to assess the performance of individual participants across all components and to get an impression of the quality of measurements for individual components, the diagrams are available in two different sorts; on the one hand as an overview on one page, on the other hand sorted according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

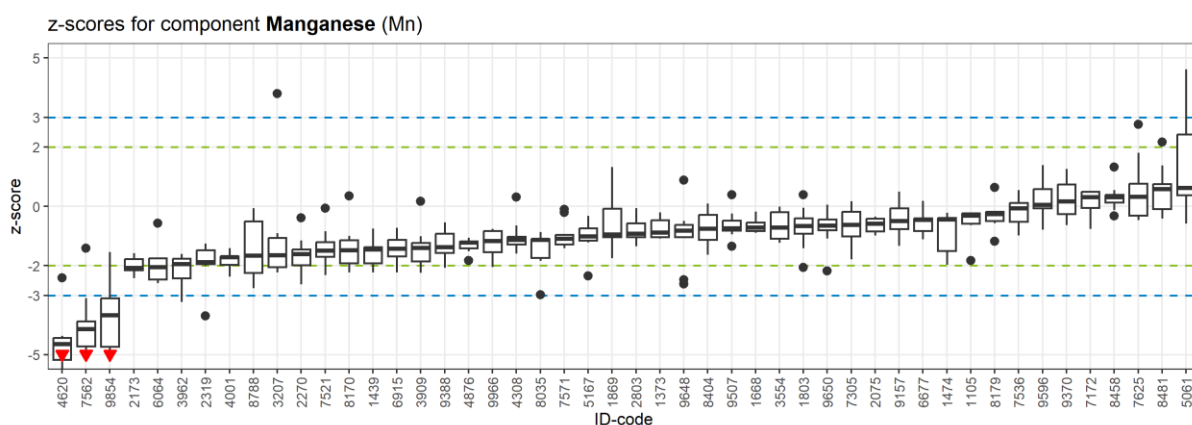
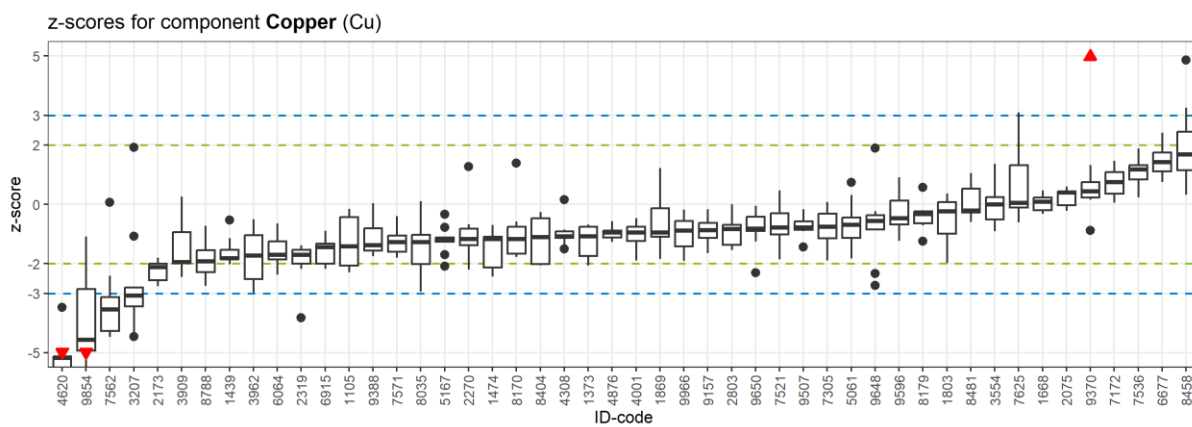
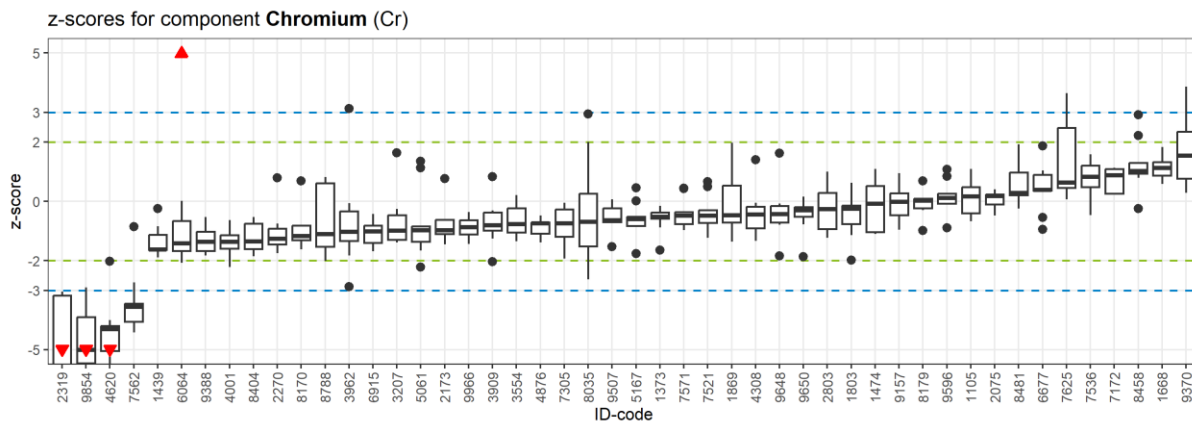
A list of the individual measurements of all participants can be found in a separate document (appendix to the annual report).

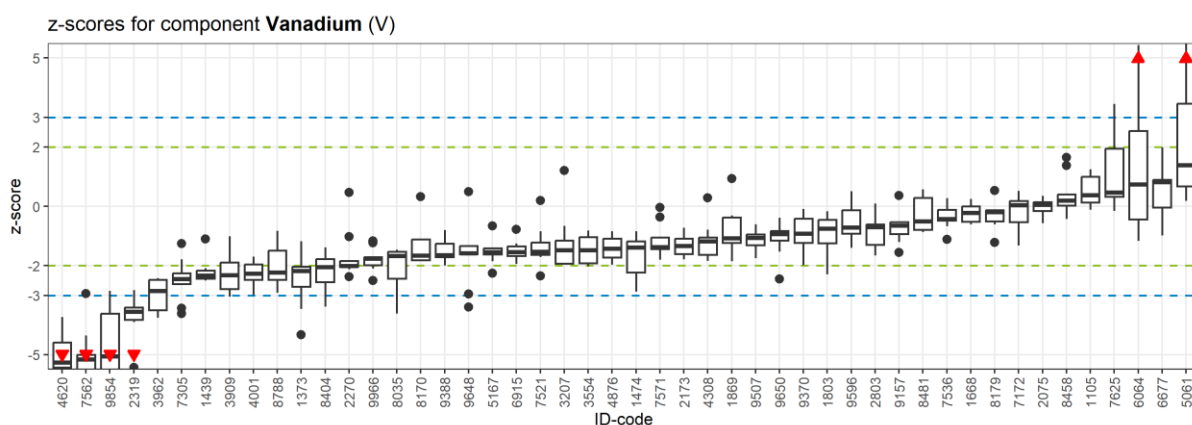
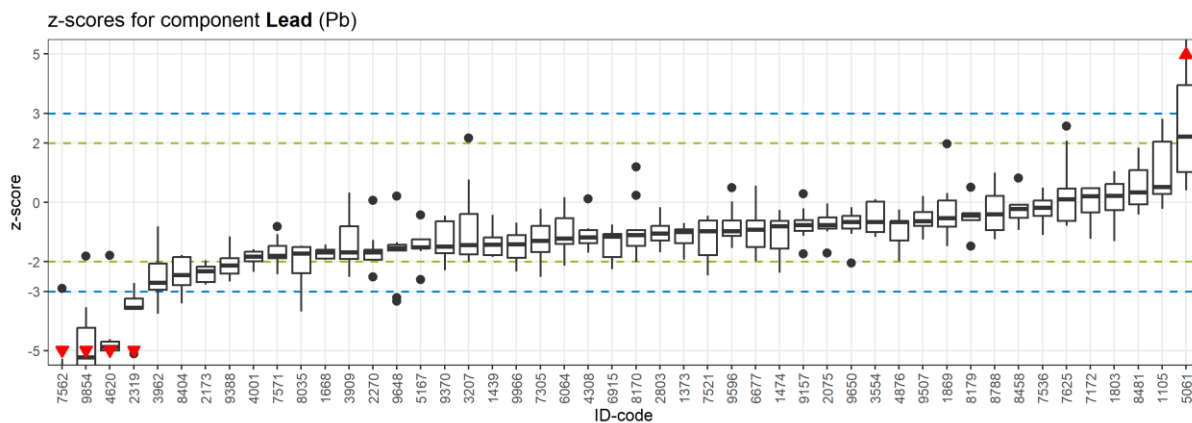
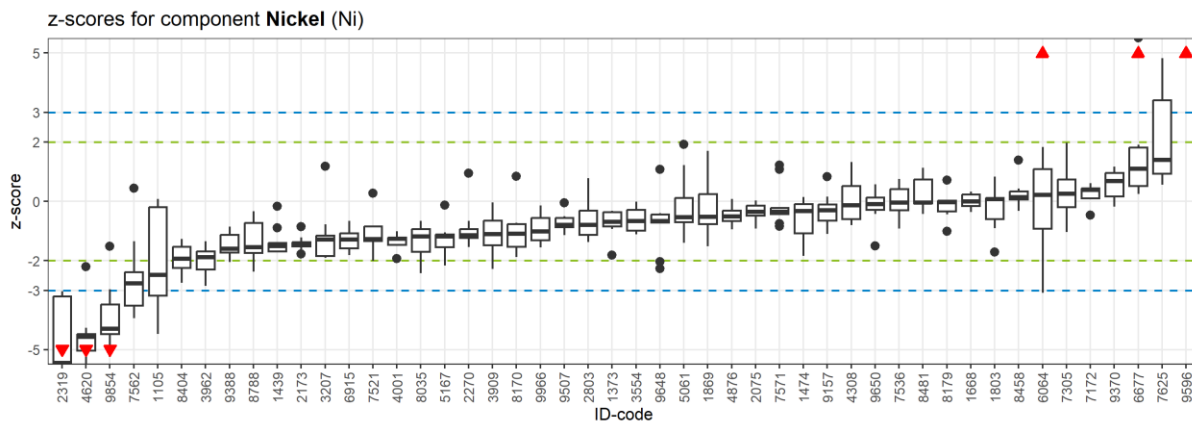
5.1.1 Dust Proficiency Test (Substance Range P)



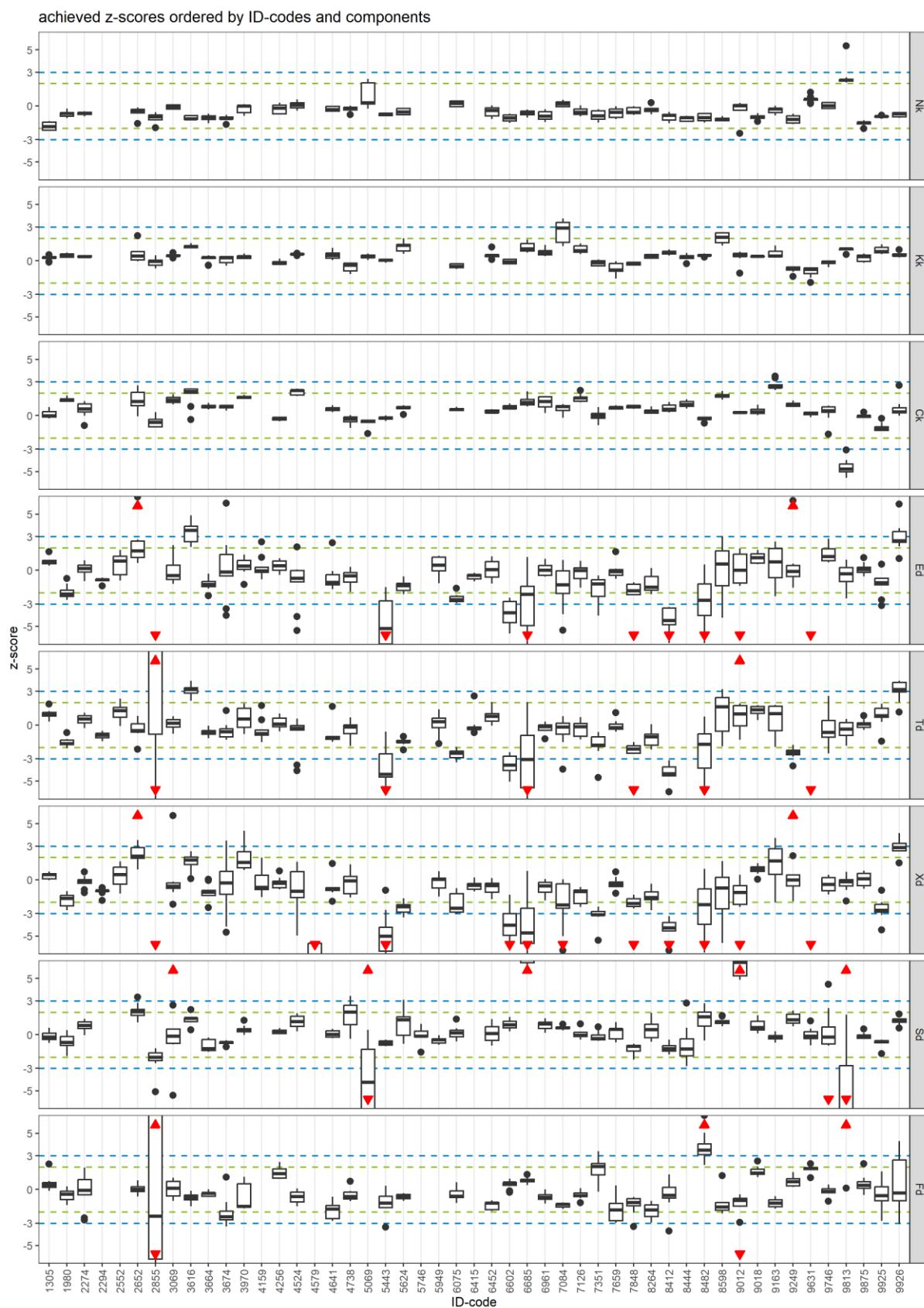
Scheme 2: Achieved z-scores dust proficiency test



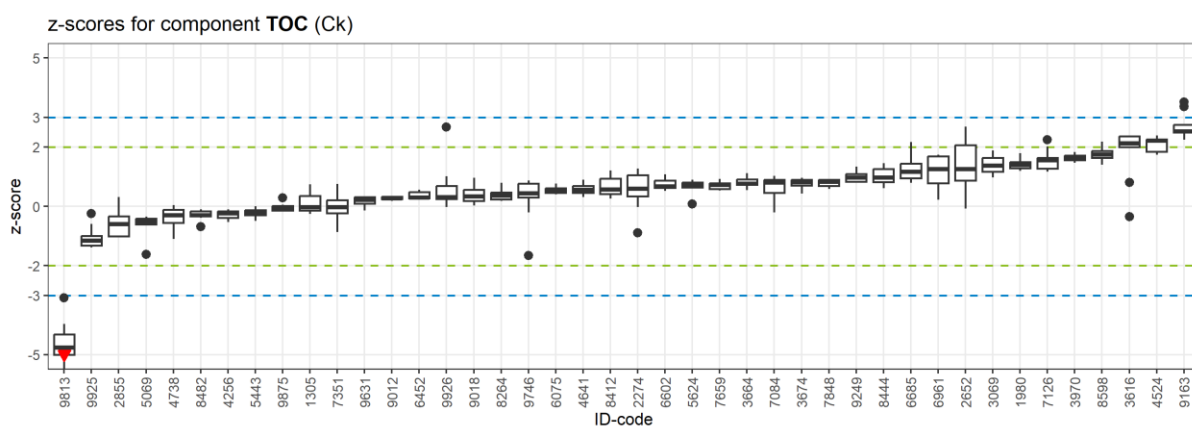
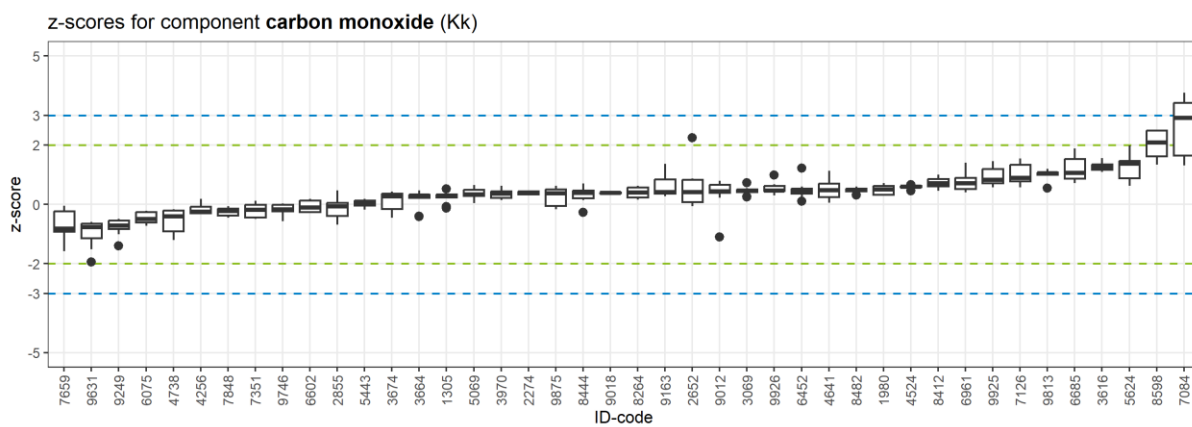
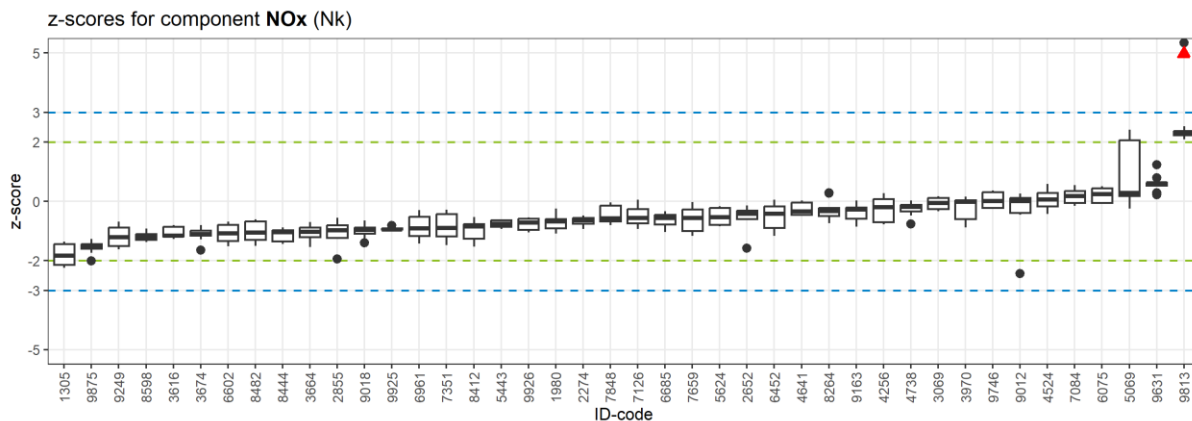


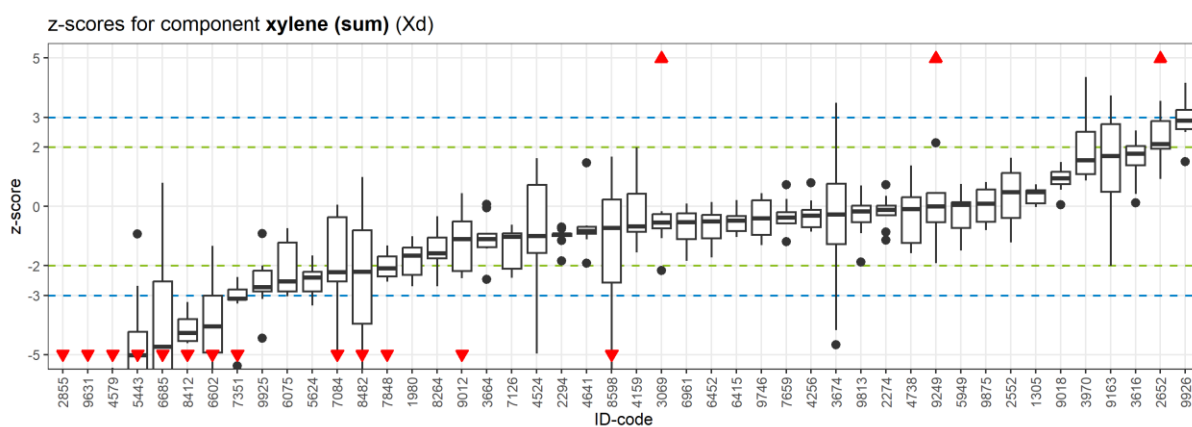
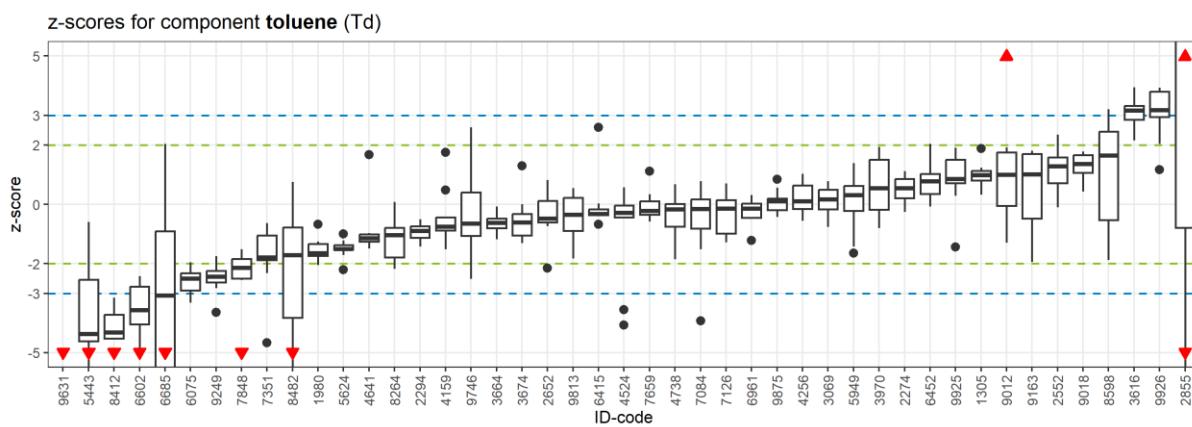
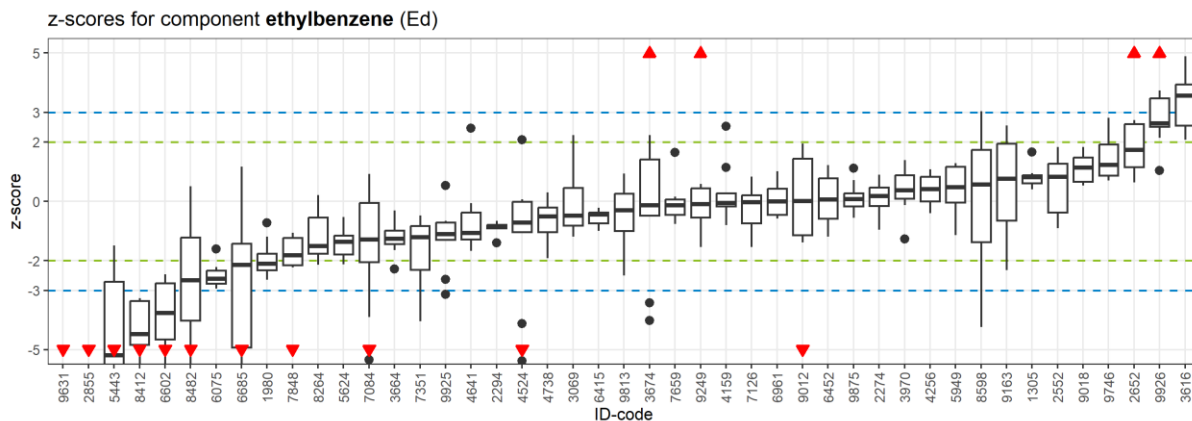


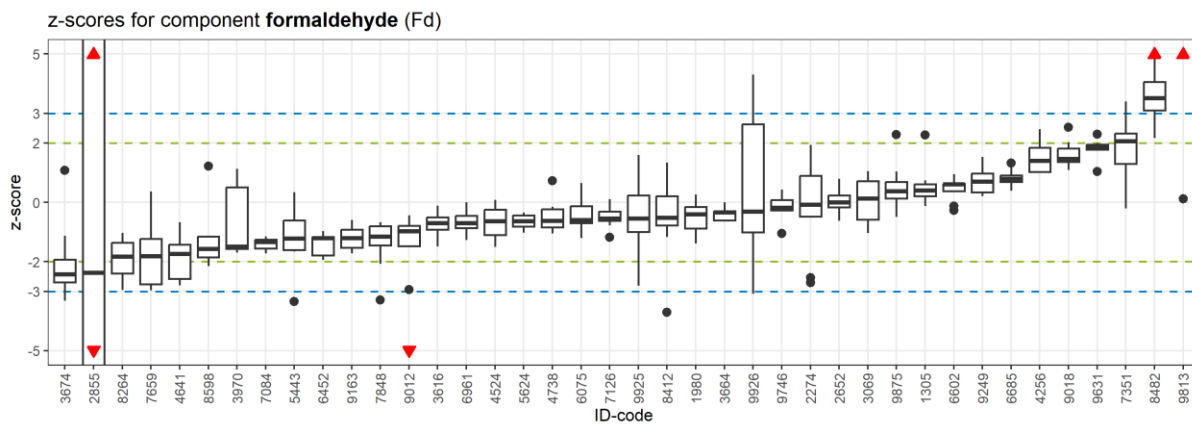
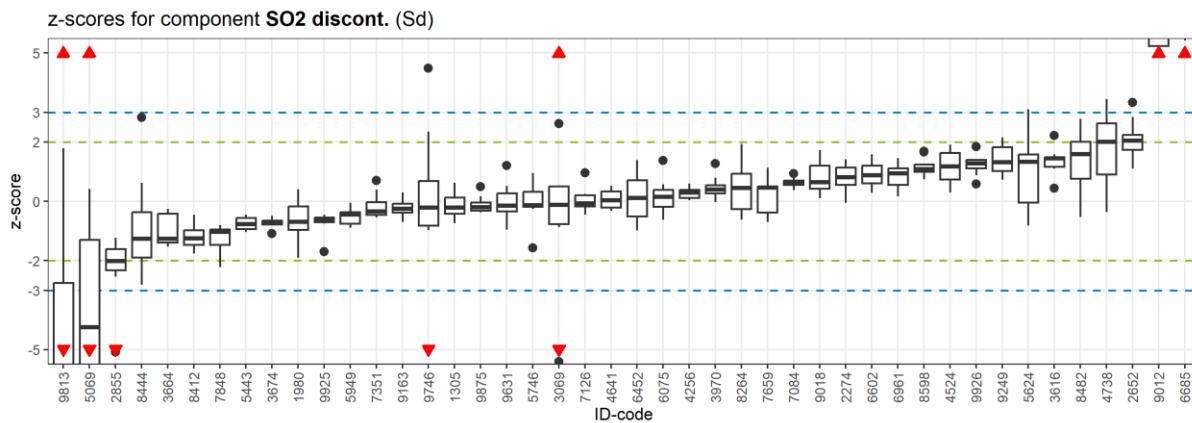
5.1.2 Gas Proficiency Test (Substance Range G)



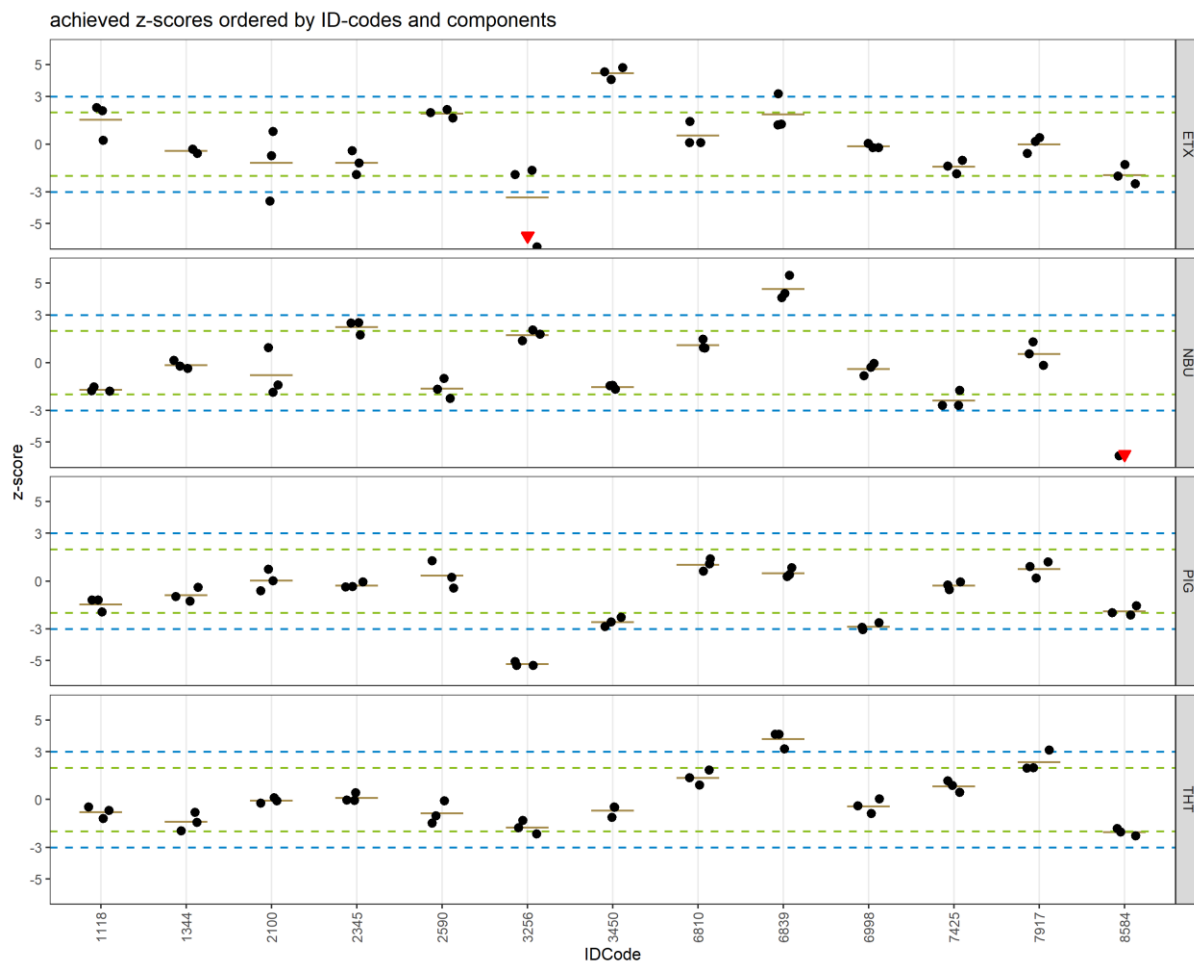
Scheme 3: Achieved z-scores gas proficiency test



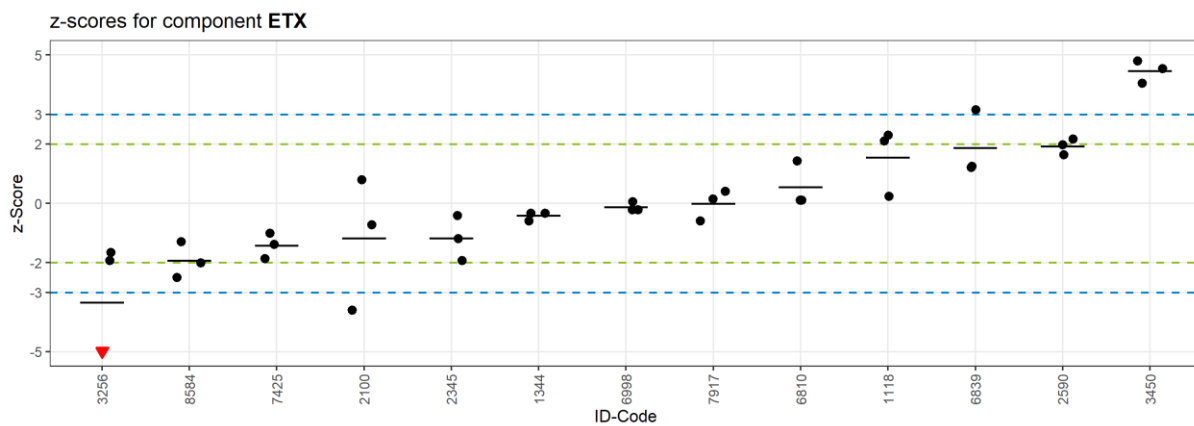


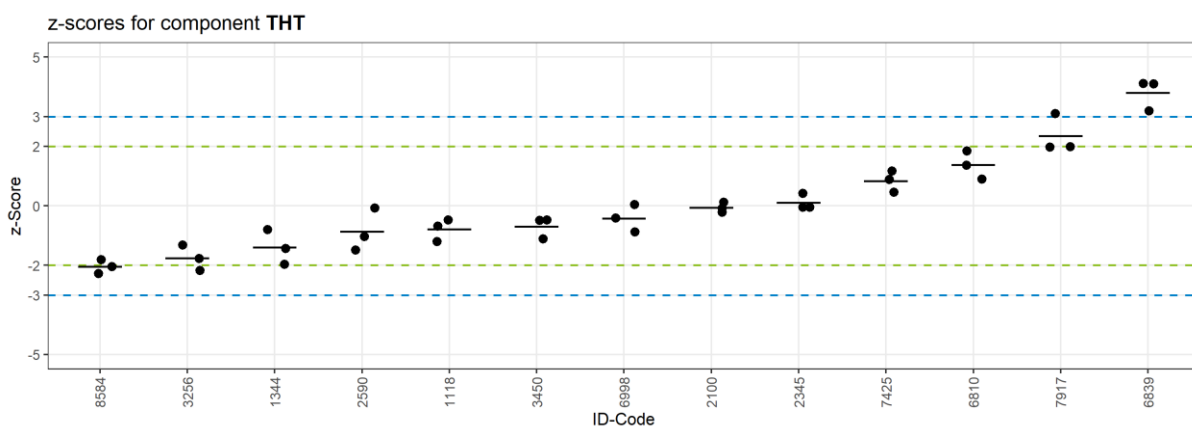
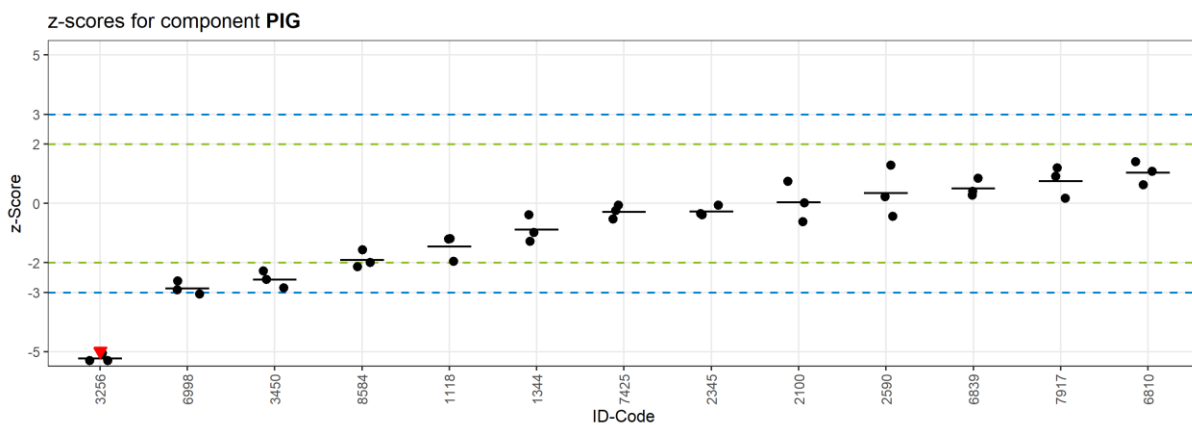
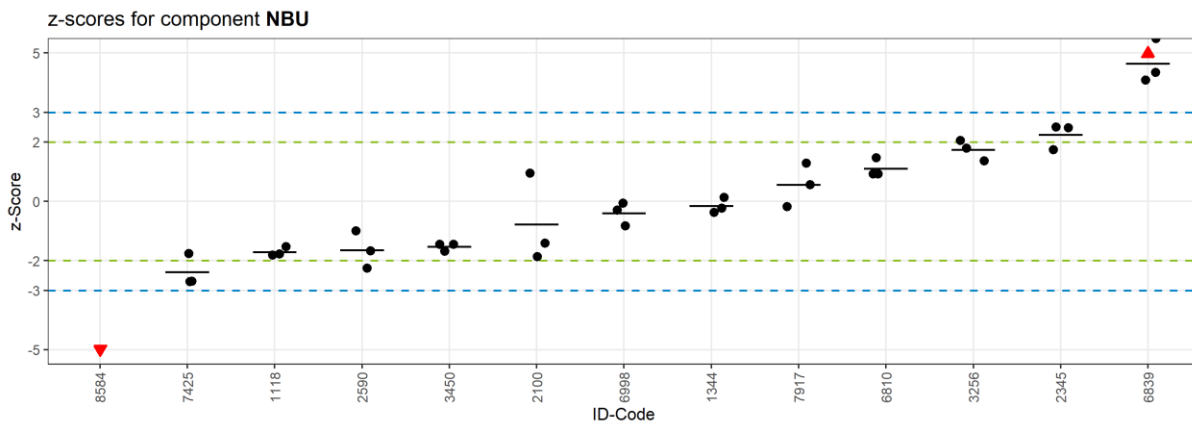


5.1.3 Odour Proficiency Test (Substance Range 0)



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 a point in each case. Mean values are indicated by a line.

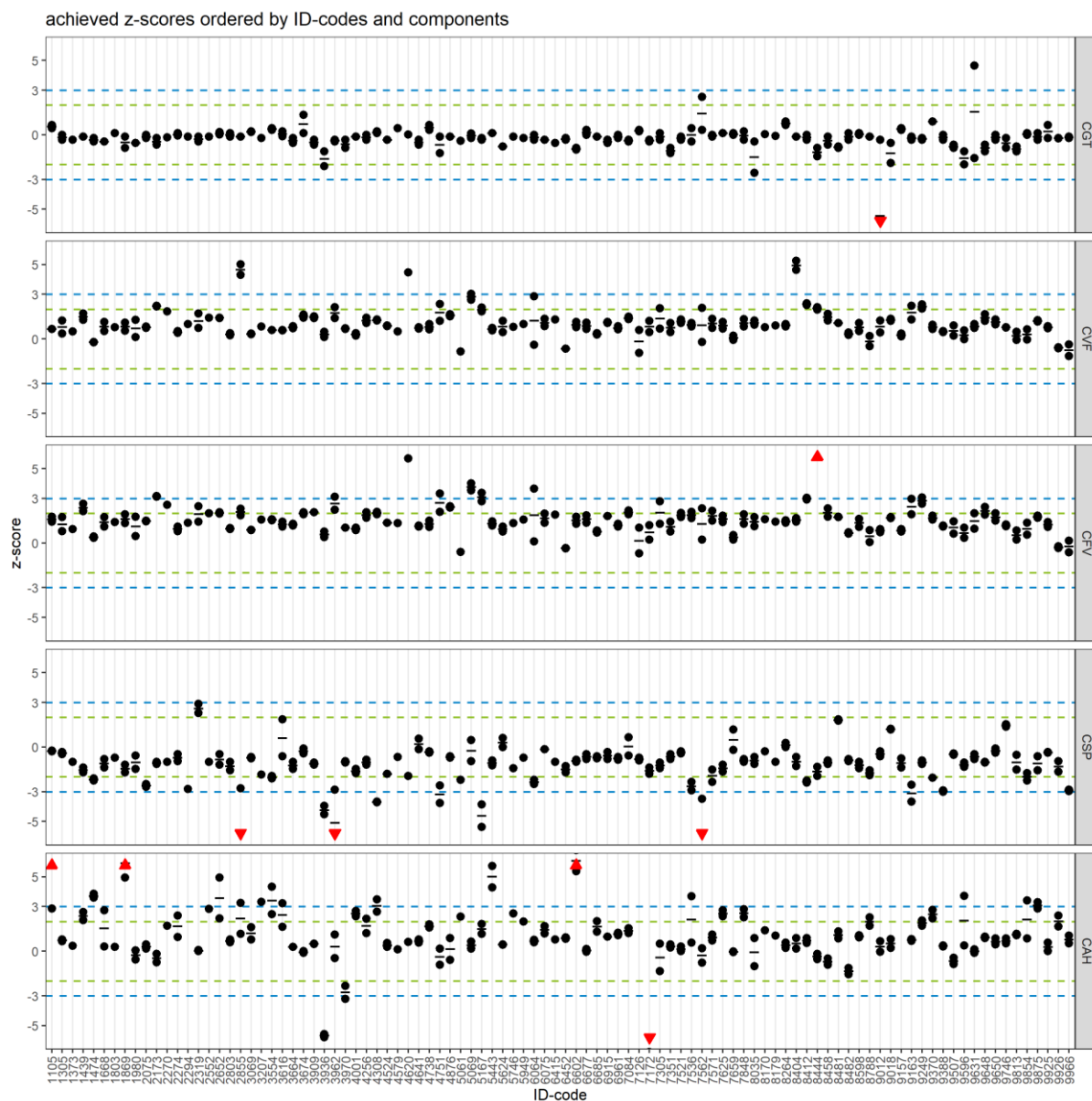
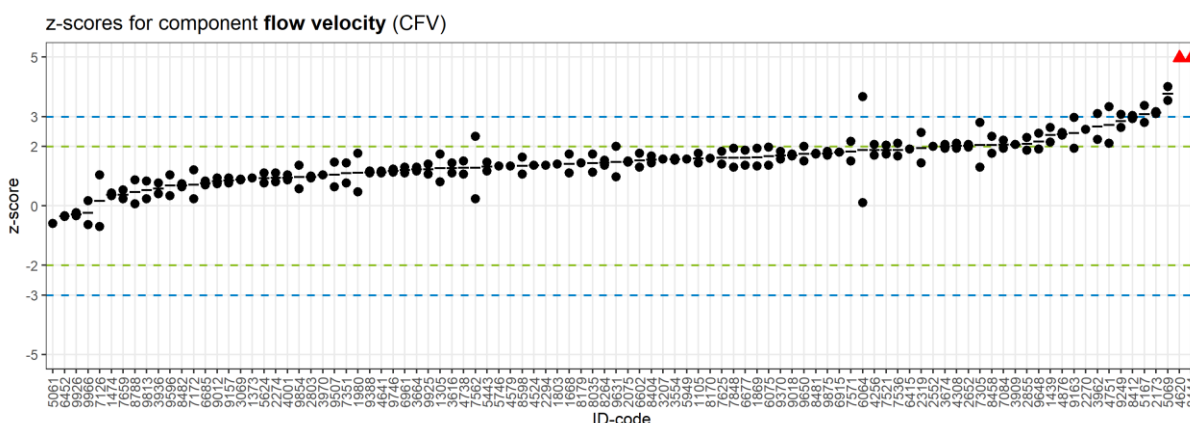
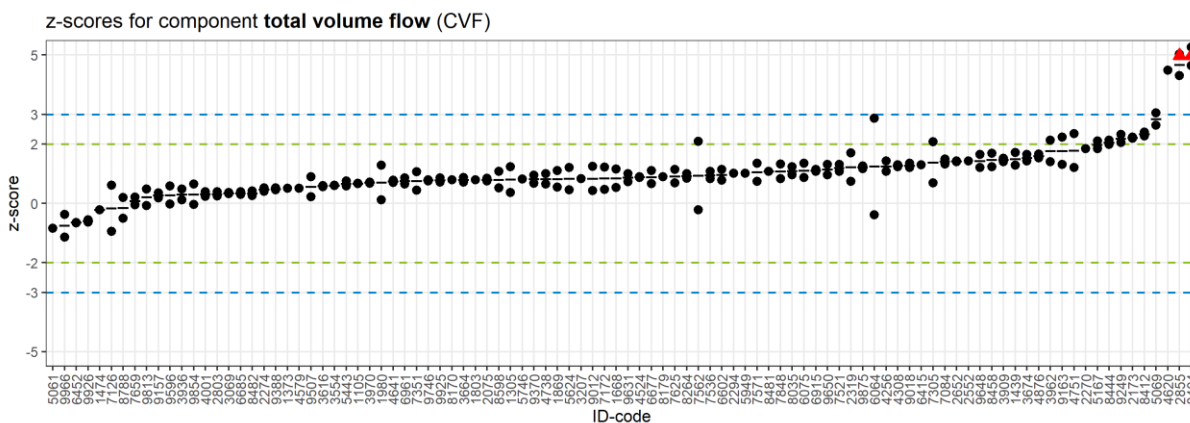
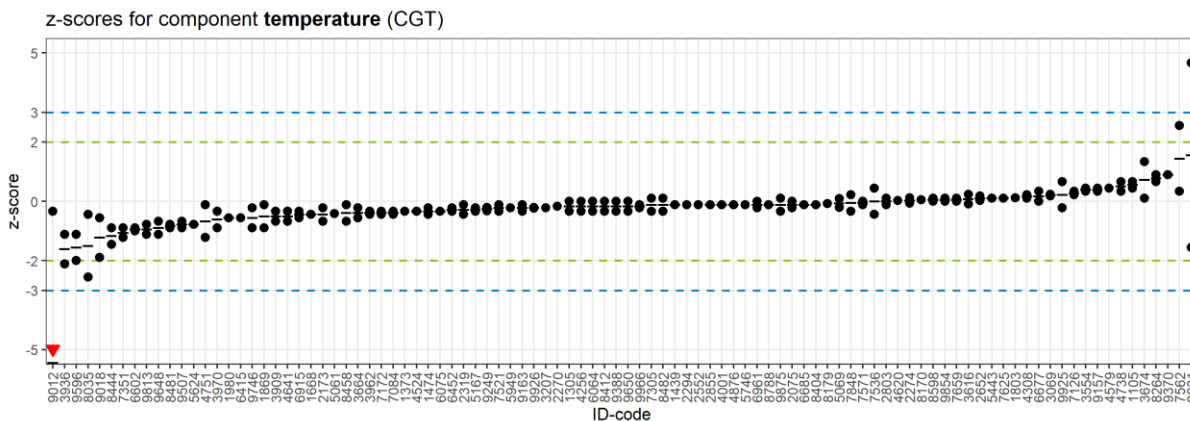
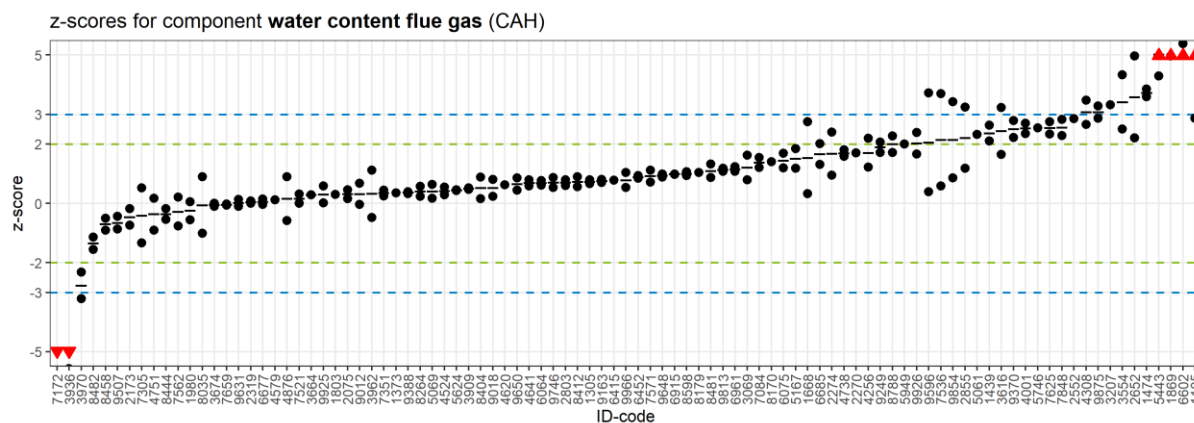
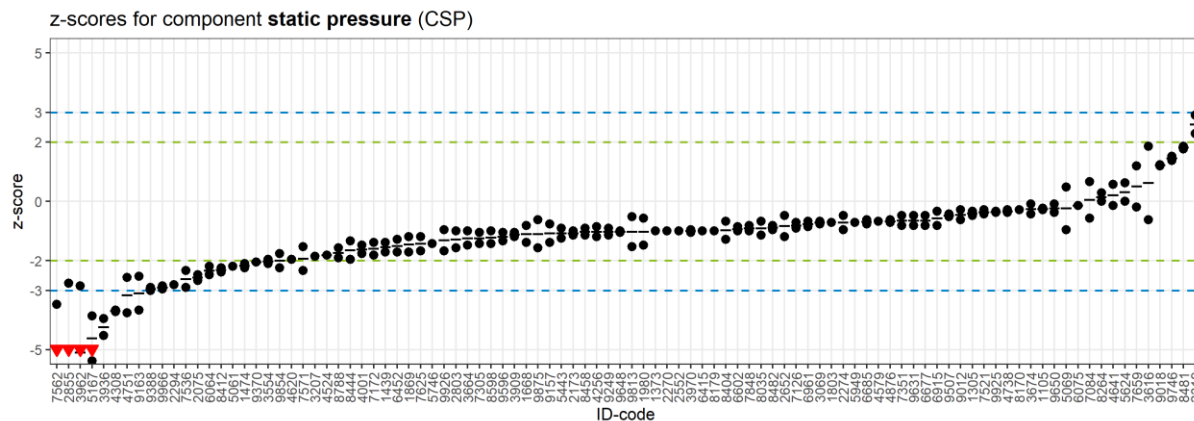


Abbildung 5: z-scores (or quotients from participant deviation and typical deviation) for gas flow conditions



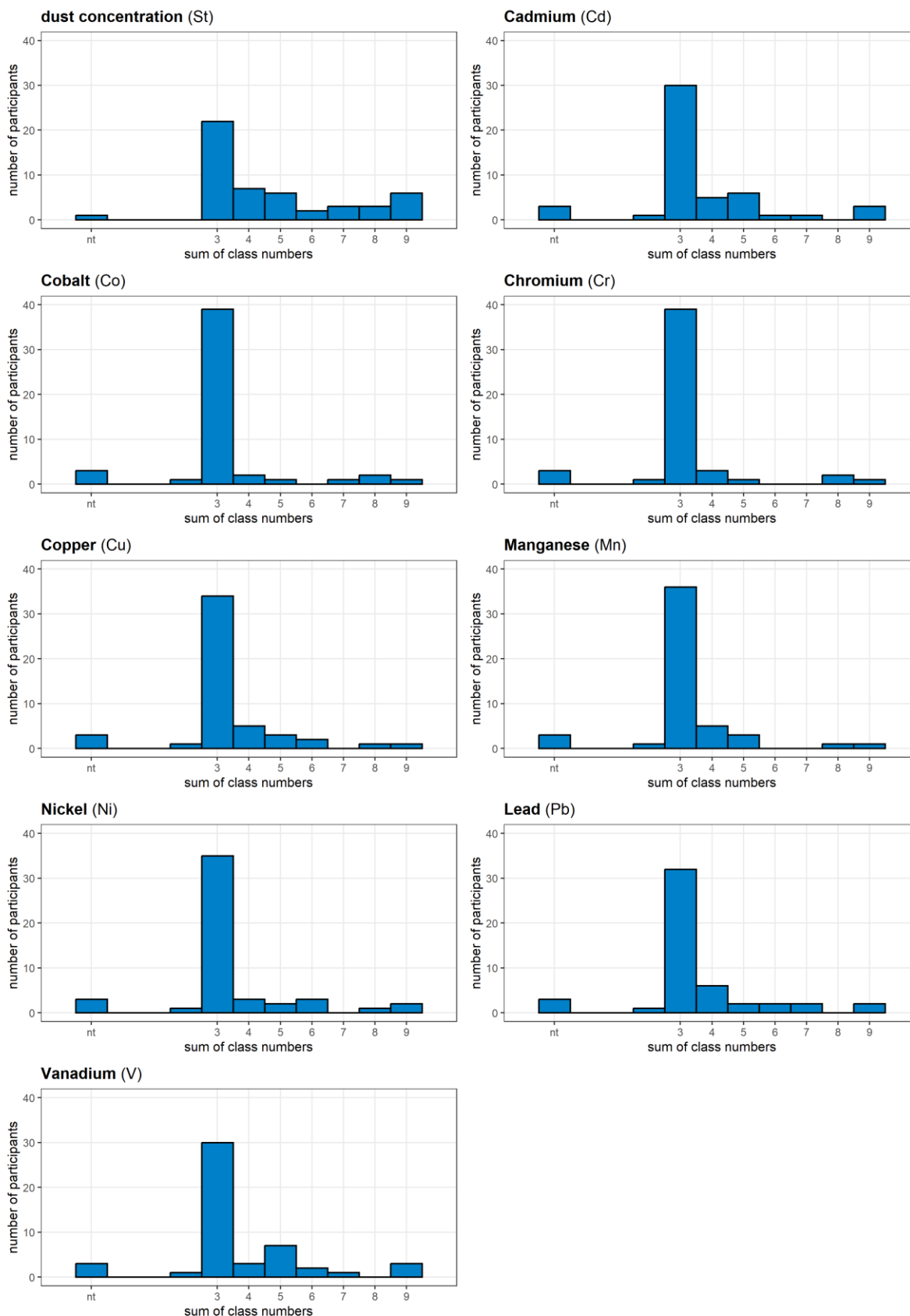


5.2 Sums of Class Numbers

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”.

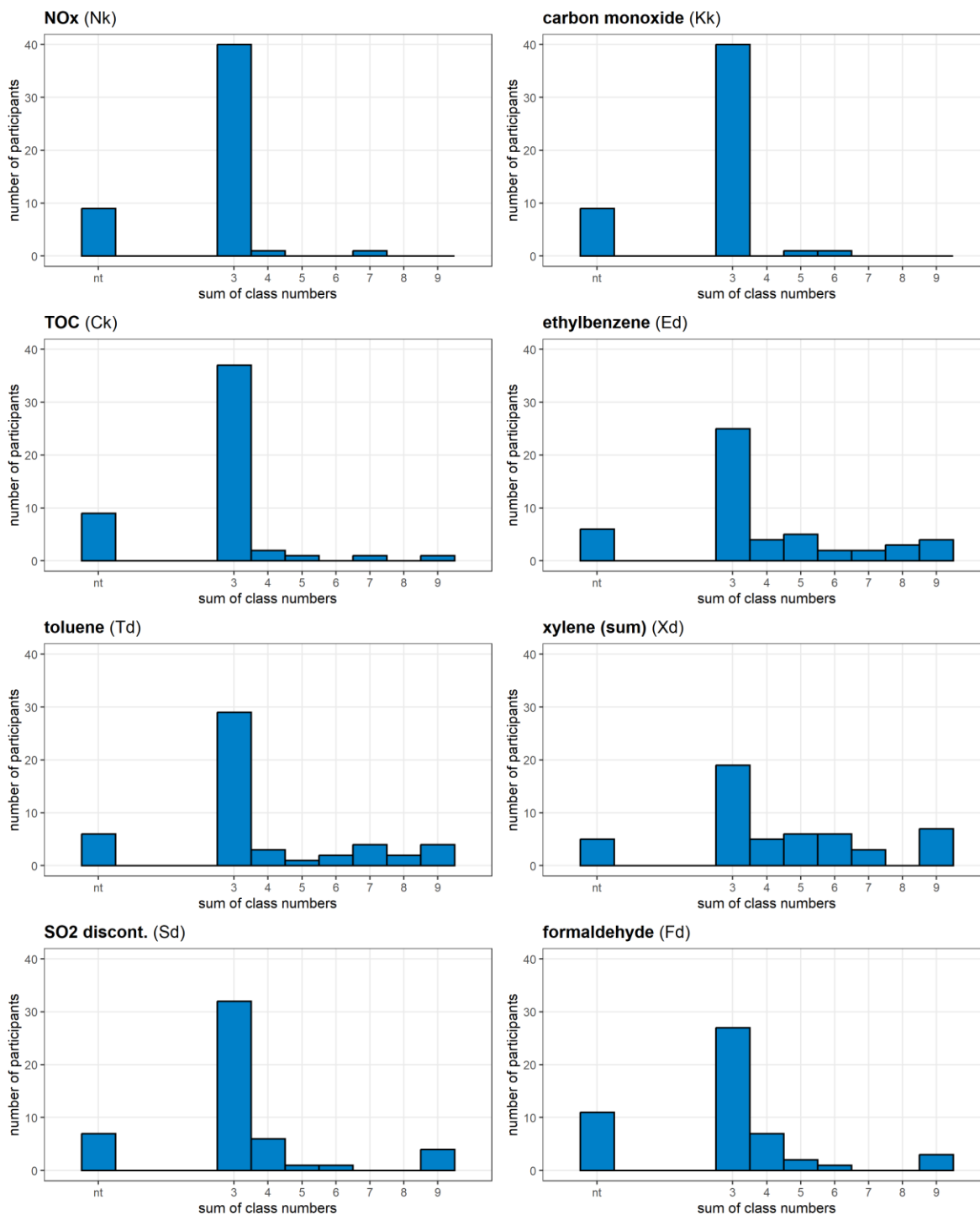
5.2.1 Dust Proficiency Test (Substance Range P)

Sum of Class Numbers



5.2.2 Gas Proficiency Test (Substance range G)

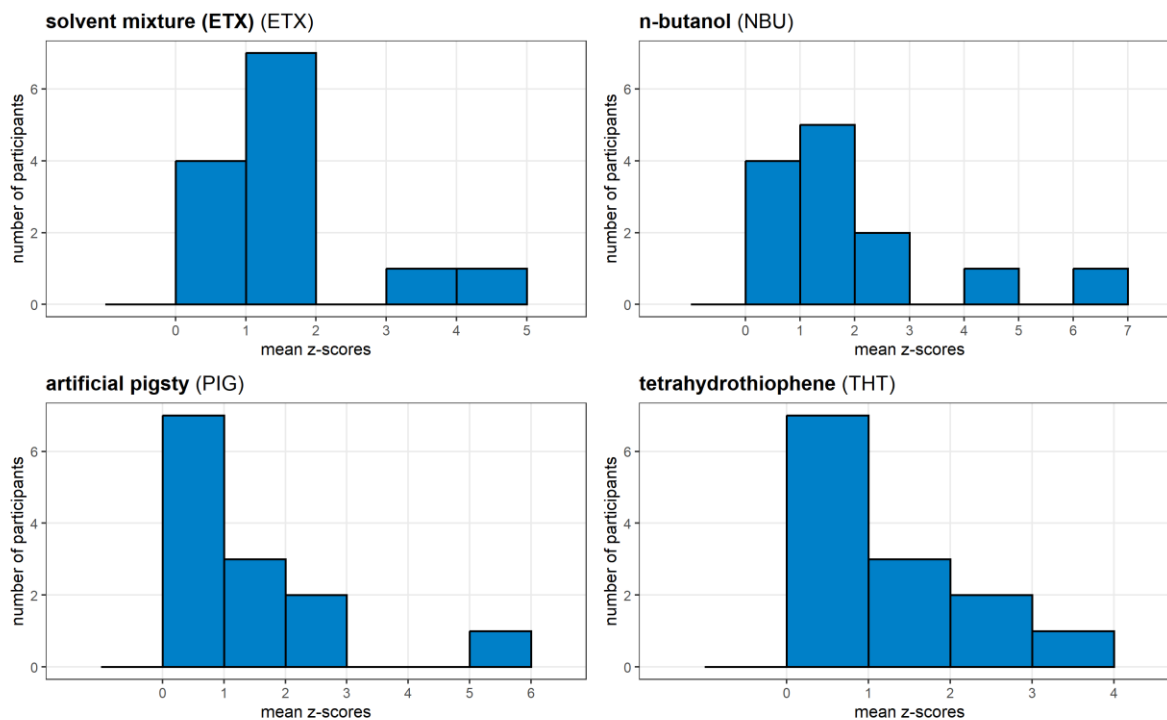
Sum of Class Numbers



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

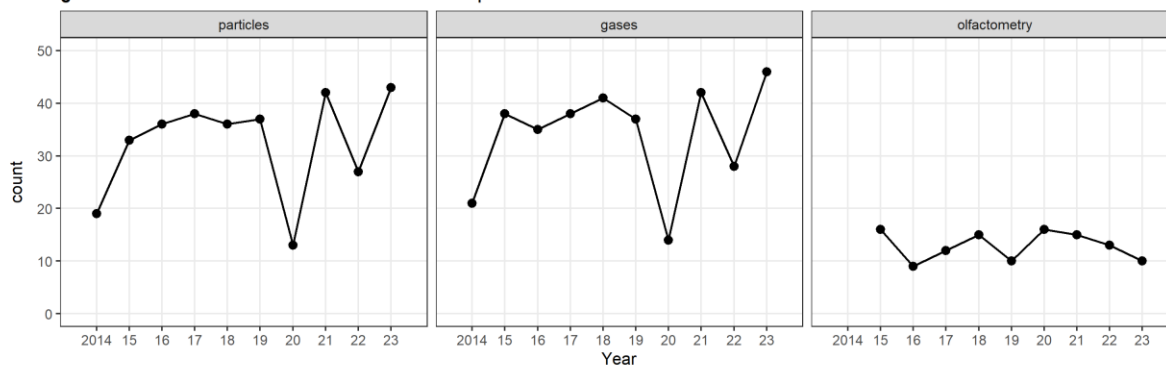
The new specifications of 2019 provide for the performance of a theory test for the dust and gas proficiency tests, which took the form of a 30-minute written test during the proficiency tests in November 2022. One person per participating laboratory could take part in this theory test. The contents 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, especially technical ones, were not permitted. The test consisted of a total of 15 questions each, which were weighted with 1 to 3 points. The number of points depended on the degree of difficulty of the question as well as on the significance 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, of which only one was correct in each case. For correct answers, the participants received the full number of points provided for the question; for incorrect answers, they received no points. The test was rated 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 rated 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 completed. The individual scores of the thematic sections of the test had no effect on the overall result.

A total of 87% of all participants passed the theory test for the dust proficiency test in 2023, 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 25 points or more.

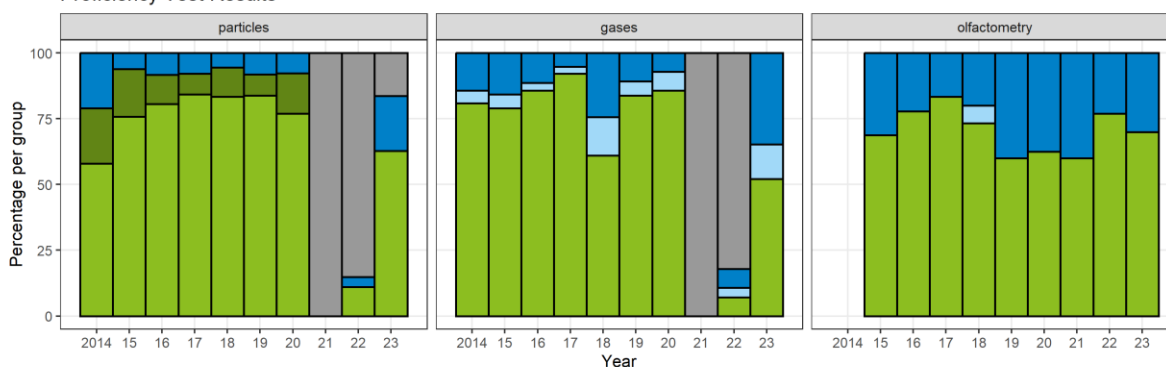
A total of 93% of all participants passed the theory test for the gas proficiency test in 2023, with a median score of 24 out of 33 points. The bottom quarter of participants scored 20 points or less, while the top quarter scored 28 points or more.

6. Interpretation of Results

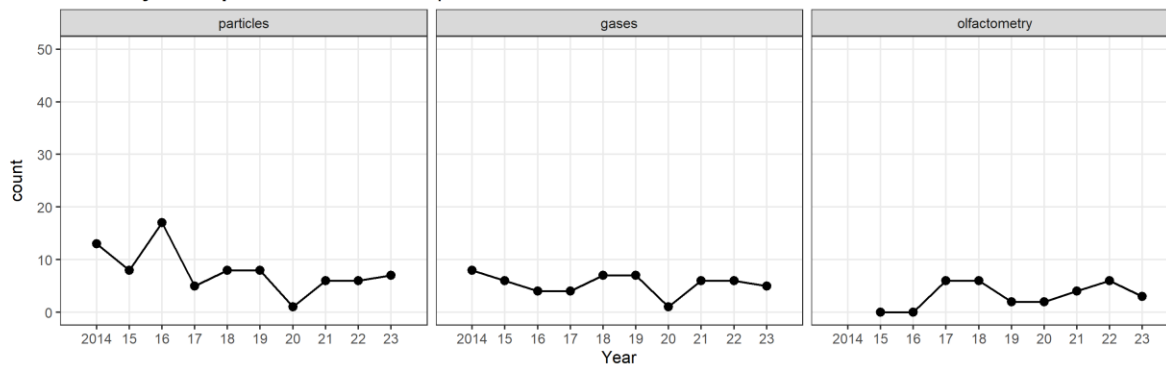
§29b Measurement Bodies Number of Participants



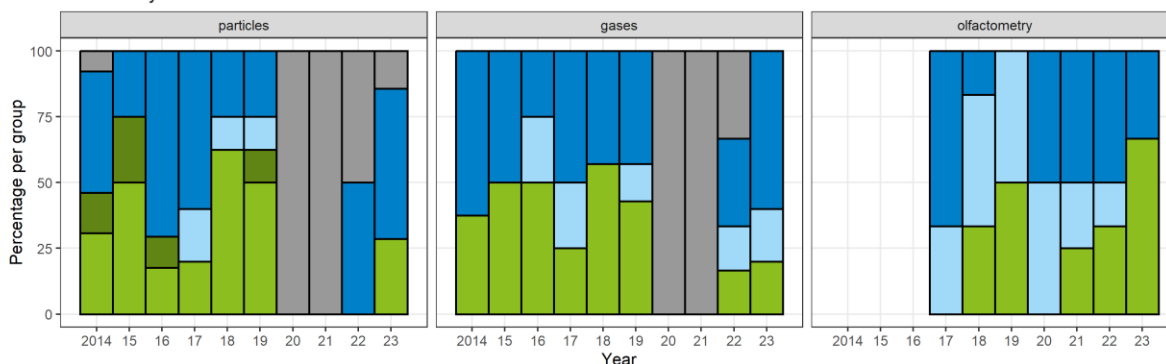
Proficiency Test Results



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 2019 (§29b-bodies)

year	proficiency test	passed	passed (post analysis)	failed	incomplete participation	not evaluated
2019	dust	31	3	3	-	-
	gas	31	-	4	2	-
	odour	6	-	4	-	-
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	-	-

Table 9: Overview of results since 2019 (voluntary participants)

year	proficiency test	passed	passed (post analysis)	failed	incomplete participation	not evaluated
2019	dust	4	1	2	1	-
	gas	3	-	3	1	-
	odour	1	-	-	1	-
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	-	-

6.1 §29b Measuring Bodies

Compared to the years 2020 to 2022, the effects of the SARS-CoV-2 pandemic receded into the background in 2023. All proficiency testing schemes could be carried out in full in accordance with the recognised LAI specifications.

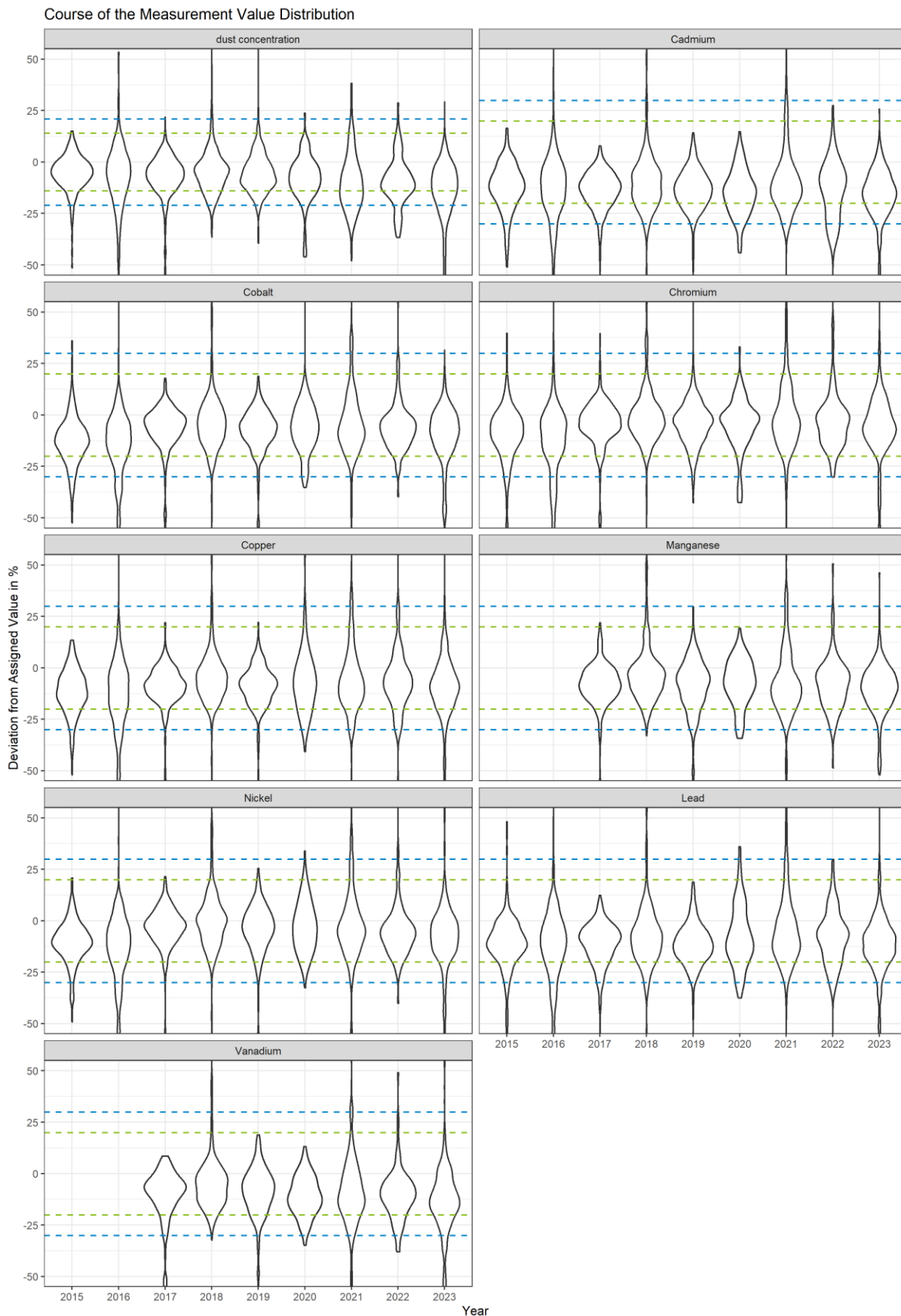
A total of 27 out of 43 (63%) of the notified measuring bodies passed the dust proficiency test. 9 (21%) of the notified measuring bodies failed. In the case of 7 (16%) participants, no evaluation could be carried out due to a technical defect in the stack simulator system. This means that 75% of the notified measuring bodies that were actually assessed passed the proficiency test. The pass rate was therefore again slightly below the average value from the years before the pandemic.

24 out of 46 (52%) of the notified measuring bodies passed the gas proficiency test. A total of 16 (35%) notified measuring bodies failed. A total of 6 (13%) participants only took part in and passed selected components as part of a repeat participation after a failed proficiency test. If these participants, who are counted as "failed" for formal reasons, are disregarded, 60% of the notified measuring bodies passed the proficiency testing scheme. The pass rate was therefore on a par with the previous year and again well below the average value from the years before the pandemic.

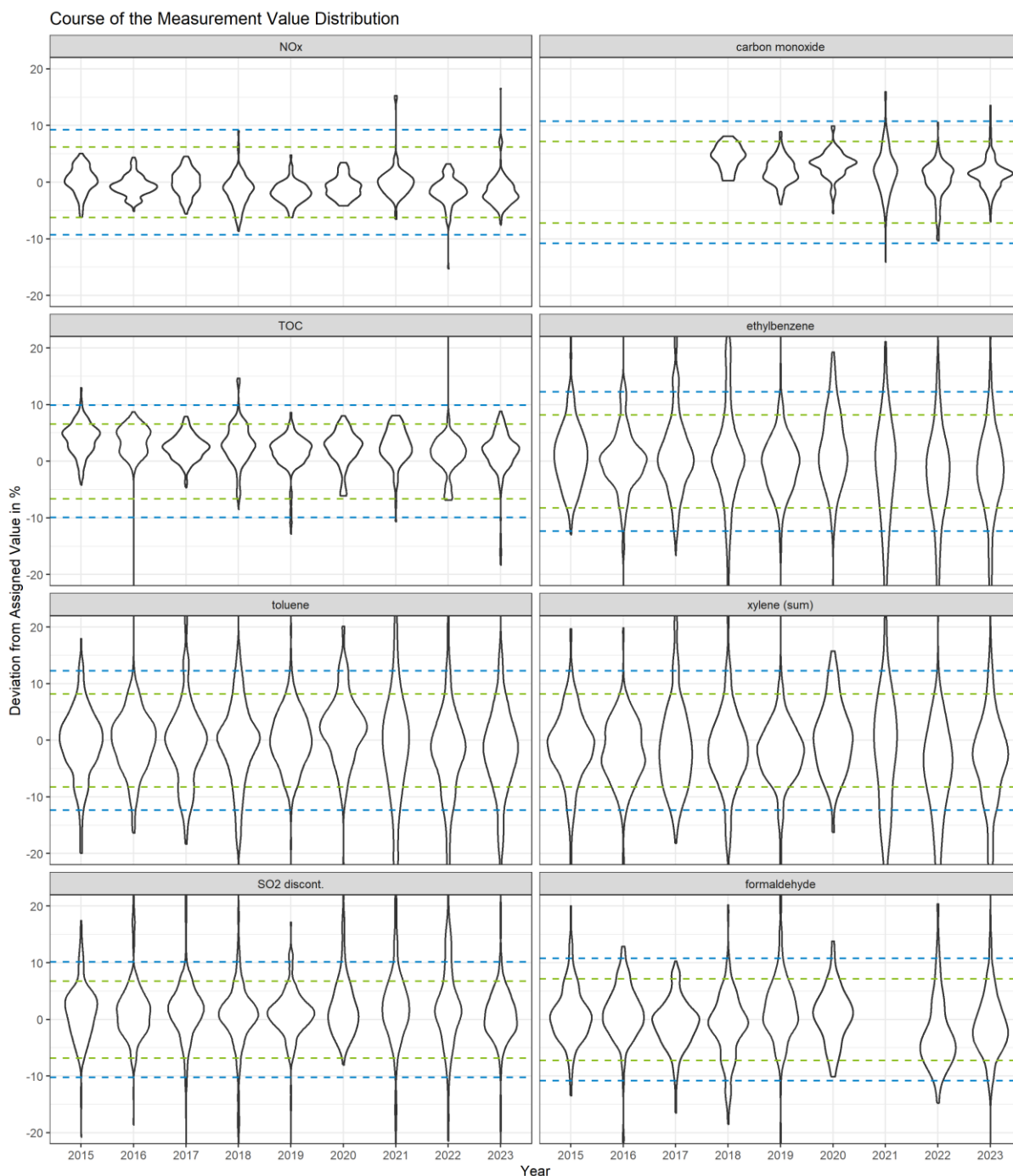
A comparison with the results from 2015 to 2020 shows that the relative deviations of the measured values from the target values for many dust and gas components were also greater on average in 2023 than in the years before the pandemic. Schemes 6 and 7 show a graphical representation of the distribution of the measured values in the interlaboratory tests of recent years. The distribution of the measured values for each component and for each year since 2015 is shown here 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 respective 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 reduced results observed in our proficiency tests: Deviations from isokinetics and the use of non-sharp-edged probes n (20). Meanwhile, the condition of the probe tips used in the dust proficiency test is 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 has been 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, among the unsuccessful proficiency test participants, there were a striking number who used thick-edged or even damaged probes.

In recent years, the results of the gas proficiency test have shown a similar development to the dust proficiency test, although in this case limited to the discontinuous components and, among these, 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 2023 deviated significantly from the values for 2015-2020 similar to dust (see Scheme 7).



Scheme 6: Course of the measurement value distribution in the dust proficiency tests 2015-2023 (all participants)



Scheme 7: Course of the measurement value distribution in the gas proficiency tests 2015-2023 (all participants)

In 2023, a total of 7 out of 10 notified measuring bodies (70%) passed the odour proficiency test. The results of the odour proficiency test were therefore better than in the years 2019 to 2021, but slightly worse than in 2022. Due to the comparatively low number of participants, however, there are already strong fluctuations in the pass rate due to the results of individual participants.

In view of the uncertainty of individual panel member results, the number of panel members 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.

When determining the odour concentration of *n*-butanol, one participant also selected a starting dilution level that was below the odour concentration to be measured (concentration in the sample: approx. 2430 ou_E/m³; starting dilution on the olfactometer: 2263 for sample 1, 1115 for samples 2 and 3). This means that the panel members already received a sample gas with an odour concentration of approx. 1.1 to 2.2 ou_E/m³ with the first dilution offered and should therefore have reported an odour perception with the first presentation. It is unclear why the olfactometry did not lead to an invalid measurement result for any of the 3 samples concerned. The participant in question submitted concentrations between approx. 400 and 800 ou_E/m³ as measurement results. A subsequent evaluation of the data available to us on olfactometry in the HLNUG proficiency tests led to the conclusion that this phenomenon is by no means an isolated case and that the start level on the olfactometer may influence the measurement result of the olfactometry in an unexpected way.

6.2 Voluntary Participants

The number of voluntary proficiency test participants fluctuates from year to year; as a rule, 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 2023, there were 7 voluntary participants in the dust proficiency test, 5 voluntary participants in the gas proficiency test and 4 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 many years is only of limited informative value.

In the dust proficiency test, a total of 2 out of 7 voluntary participants (29%) were successful in 2023, 4 (57%) participants did not pass the proficiency test. One (14%) voluntary participant could not be assessed due to a technical defect in the stack simulator system. Of the voluntary participants who were actually assessed, 33% passed the proficiency testing scheme.

In the gas proficiency test, 1 out of 5 (20%) of the voluntary participants passed the proficiency test, 3 (60%) were unsuccessful. Another participant only passed selected components, while he did not participate in the other components. Formally, this participation is 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 25%.

In the odour proficiency tests, 2 out of 3 voluntary participants (67%) passed, the third voluntary participant did not pass the proficiency test.

6.3 Gas Flow Conditions

For each proficiency test, the participants must also determine and specify the gas flow conditions. With the new LAI specifications, the measurements of the volume flow should actually have been carried out as an evaluated component of the dust and gas proficiency tests since mid-2020. However, since the resumption of the regular proficiency tests in November 2022, the gas flow conditions have now been measured and evaluated as provided for in the 2019 LAI specifications. This means that in 2023, values are available to the intended extent (two measurements on two different days under different conditions) for all participants in the dust and gas proficiency tests.

The values recorded in 2023 (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. In the case of flue gas humidity (CAH) and static pressure (CSP), there are overall larger deviations from the assigned values 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. The data received are summarised in the following tables and presented graphically. The database is based on feedback from participants from the years 2016 to 2023.

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. Since 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 z-scores of the participants were therefore established, with negative values also being included in the mean value. In addition, similar components such as heavy metals or organic solvents were combined to form a common mean value. This type of evaluation certainly represents a simplification of the problem and cannot show all the details. Thus, for example, different influences in different concentration ranges or high fluctuations between the individual results of a participant are completely ignored in this evaluation. However, the limitation to the mean values of the participants' z-scores allows a simple estimation of the effects of different methods on the mean deviation of the measured values from the assigned value.

For most evaluations, hardly any changes can be observed compared to the values in the last annual report. This is ultimately due to the fact that the data basis for the 2023 annual report has only increased slightly compared to the last annual report, while the values for most evaluations hardly differ from those of previous years. As a result, most findings become more robust 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 by no means proves causality. For example, it is quite conceivable that participants who use a certain device or procedure may happen to have other similarities that actually affect the measurement results, while the identified similarity actually plays no 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. Notified 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 sites are underrepresented.

7.1 Measurement Uncertainties

The participants' data on the absolute extended measurement uncertainties of their methods used in the dust proficiency test are shown in the following scheme. The median of the respective data as well as the 25th and 75th percentile are listed in the following table. This information should be understood as follows: Only a quarter of the participants indicated an uncertainty of measurement below the 25th percentile. Half of the participants indicated an uncertainty of measurement below or above the median. A quarter of the participants indicated an uncertainty of measurement greater than the 75th percentile.

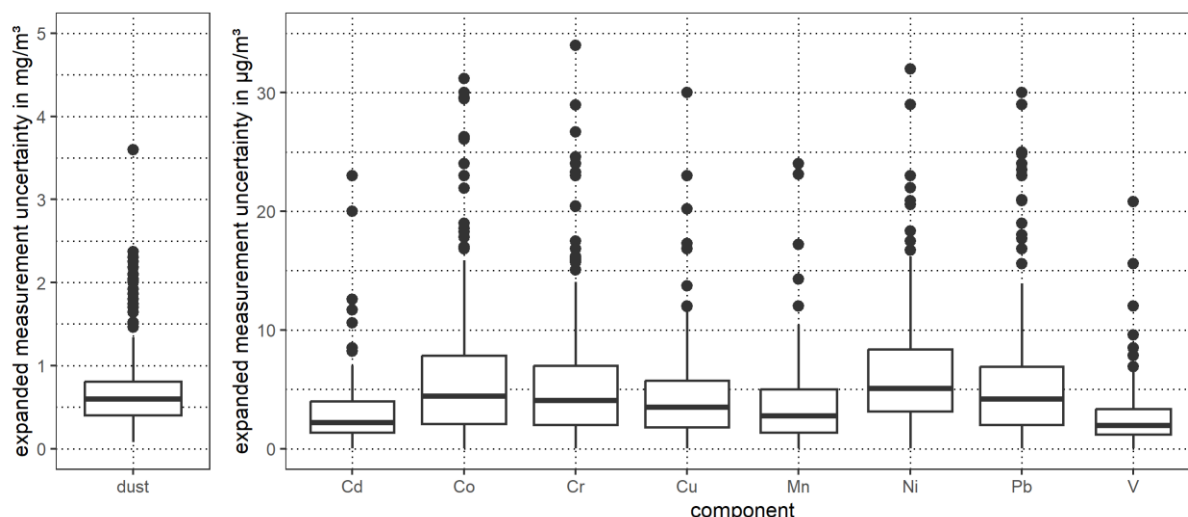


Table 10: Expanded measurement uncertainties reported by participants of the dust proficiency test

	dust [mg/m ³]	Cd [µg/m ³]	Co [µg/m ³]	Cr [µg/m ³]	Cu [µg/m ³]	Mn [µg/m ³]	Ni [µg/m ³]	Pb [µg/m ³]	V [µg/m ³]
75 th percentile	0,84	4,00	8,17	7,00	5,75	5,00	8,50	6,99	3,39
median	0,60	2,23	4,48	4,16	3,48	2,80	5,10	4,22	1,97
25 th percentile	0,40	1,35	2,07	2,00	1,80	1,37	3,15	1,99	1,19
number of values	250	226	227	227	227	189	227	226	185

For all information on absolute expanded measurement uncertainties, it should be noted that for reasons of comparability, participants were asked to give only one value for each method. The information may therefore refer to the highest concentration measured in the proficiency test and would be lower for lower concentrations. Nevertheless, these values should enable all participants to make an approximate assessment of how their own uncertainty of measurement relates to the uncertainty of measurement of other laboratories.

For the gas emission proficiency test, the following uncertainties were reported.

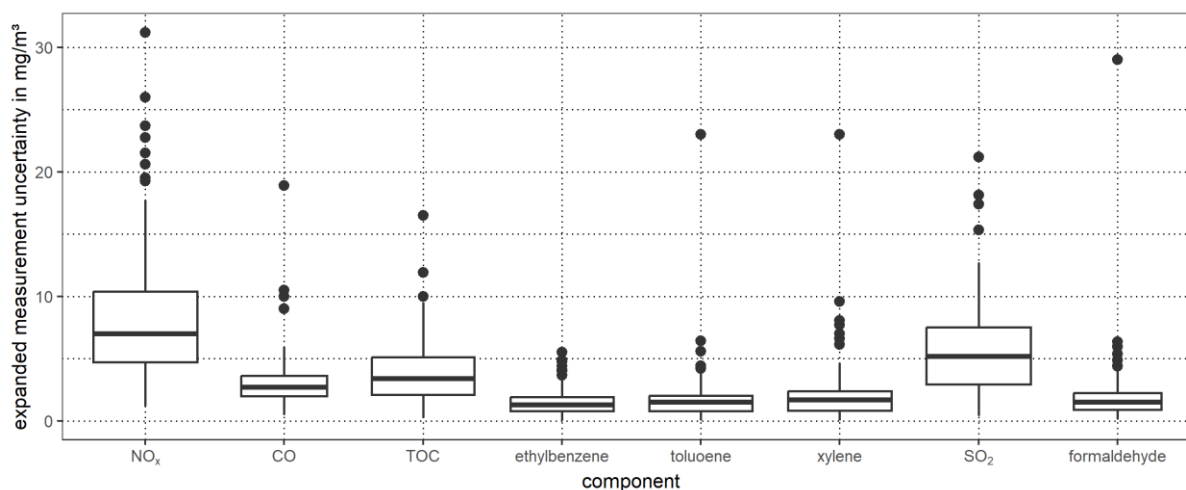


Table 11: Expanded measurement uncertainties reported by participants of the gas proficiency test

	NO _x as NO ₂ [mg/m ³]	CO [mg/m ³]	TOC [mg/m ³]	SO ₂ [mg/m ³]	form- aldehyde [mg/m ³]	ethyl- benzene [mg/m ³]	toluene [mg/m ³]	sum of xylenes [mg/m ³]
75 th percentile	10,40	3,63	5,10	1,91	2,04	2,41	7,53	2,24
median	7,03	2,70	3,40	1,32	1,50	1,70	5,20	1,50
25 th percentile	4,72	1,98	2,11	0,78	0,80	0,82	2,95	0,90
number of values	238	163	233	234	235	236	239	169

7.2 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 gooseneck is used, and whether this probe is rinsed after each sampling, every working day, or never. Four 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.

The data basis in this report is limited to the results since autumn 2018. In summer 2018, the query about the rinsing procedure was concretised with regard to frequency; since then a total of 164 participants gave corresponding information about their rinsing procedure. In previous years, only the basic rinsing procedure (yes/no) was queried, the data are therefore unfortunately not comparable.

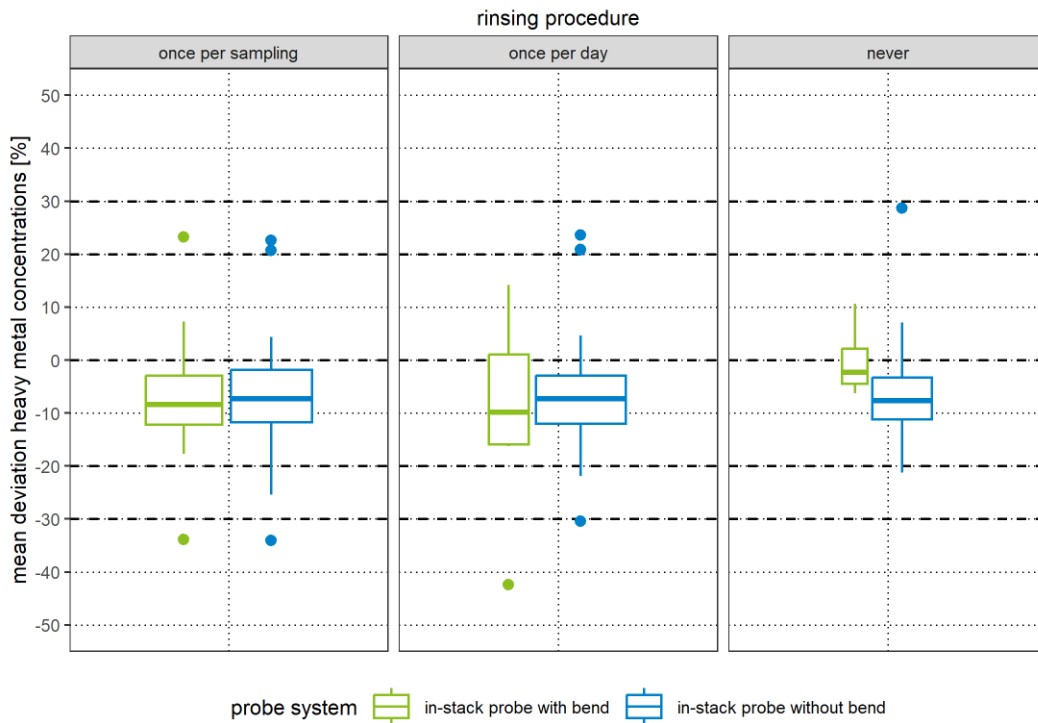
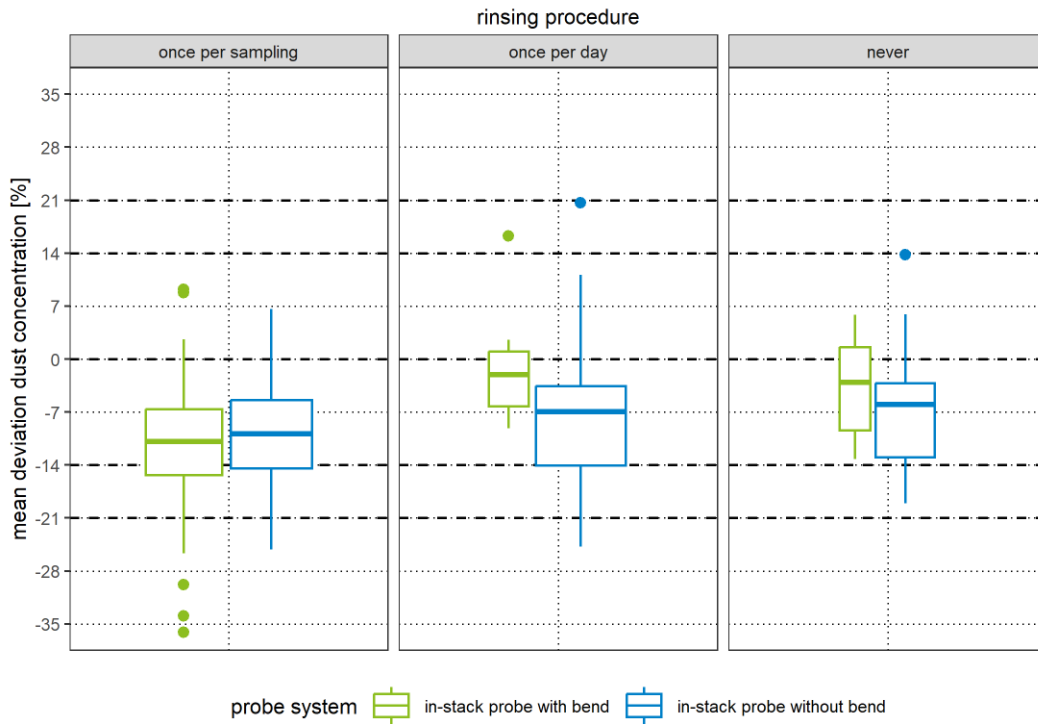


Table 12: Correlation of dust measurement results with probe systems and rinsing procedures (2018-2023)

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%	36	-8,4%	36
2 (centre)		once per day	-2,0%*	10*	-9,8%*	10*
3 (right)		no rinsing	-3,1%*	6*	-2,3%*	4*
4 (left)	in-stack probe without bend	after each sampling	-9,9%	41	-7,3%	41
5 (centre)		once per day	-7,0%	49	-7,4%	49
6 (right)		no rinsing	-6,0%	22	-7,6%	22

*This combination was only indicated by approx. 2-6% of the participants. The median is clearly 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 influences of single laboratories. The above-average results for combination 3 (probe with elbow that is not rinsed: right-hand figures, green) are unlikely to be representative of this type of sampling. Combination 3 is explicitly not in conformity with the standard, because with this probe geometry, dust adhesion to the inner surface of the probe is to be expected in any case, which can lead to significantly lower results if rinsing is not carried out.

It is striking that participants with a probe without a bend achieve better measurement results for dust concentrations the less frequently rinsing is carried out (combination 4, 5 and 6). At the same time, however, the rinsing frequency seems to have no significant effect on the heavy metal results.

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

On average, the results of the dust measurements in the proficiency tests of 2023 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).

7.3 Diameter of the Nozzle Opening in Dust Samplings

The information provided by the participants on the diameter of their probe’s nozzle opening does not indicate a clear trend. Regardless of the diameter, the measured values always seem to scatter over a wide range. Overall, however, the size of the nozzle opening does not seem to be a determining factor for the measurement results. Probe diameters that were mentioned by less than 15 participants (or 5% of all participants) are not listed here.

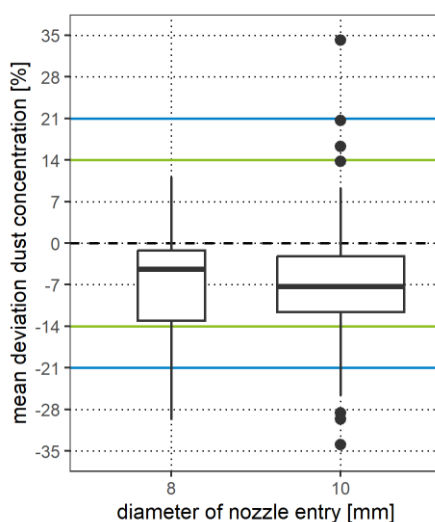


Table 13: Correlation of dust measurement results with nozzle opening diameters (2016-2023)

diameter of nozzle opening	8 mm	10 mm
75 th percentile	-1,2%	-2,2%
median	-4,4%	-7,2%
25 th percentile	-13,0%	-11,6%
number of values	56	201

7.4 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 35 participants stated that heavy metal analysis was performed using AAS equipment, while 249 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.

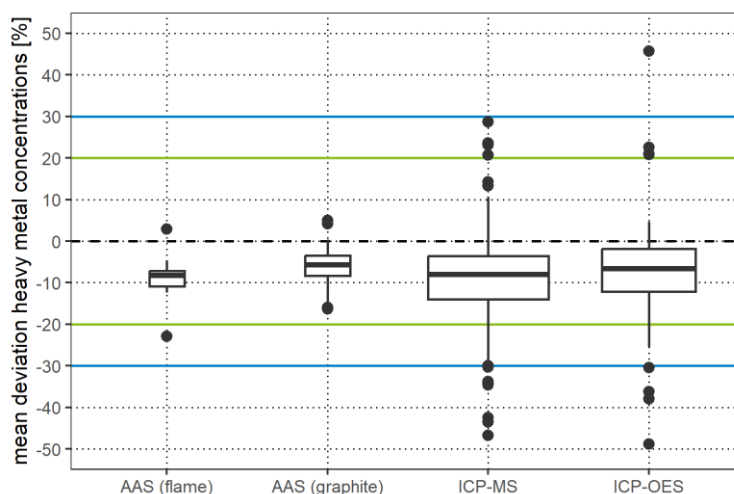


Table 14: Correlation of the mean deviation from the assigned value for heavy metal results and the used analysis devices (2016-2023)

analysis device	flame-AAS	graphite furnace AAS	ICP-MS	ICP-OES
75 th percentile	-7,2%	-3,4%	-3,6%	-1,9%
median	-8,2%	-5,6%	-8,0%	-6,6%
25 th percentile	-10,8%	-8,3%	-14,1%	-12,1%
number of values	13	22	155	94

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 (88%) reported that they had worked with CS₂. The average results of all participants were close to the target value.

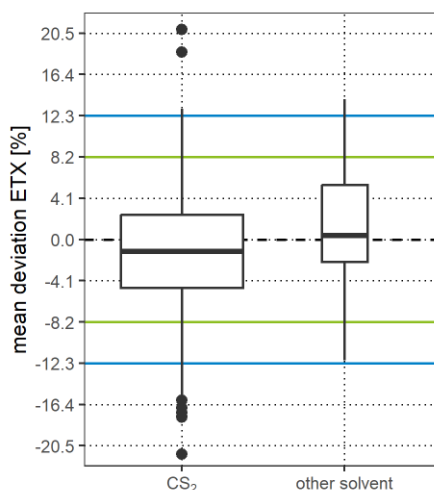


Table 15: Correlation of ETX measurement results with the desorption solvent (2016-2023)

solvent used in desorption	CS₂	other solvent
75 th percentile	+2,5%	+5,4%
median	-1,2%	+0,4%
25 th percentile	-4,8%	-2,2%
number of values	242	34

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.

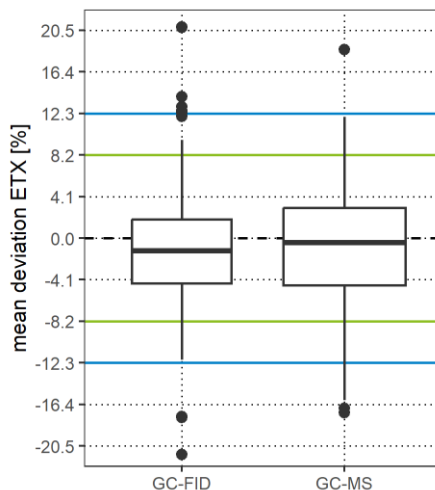


Table 16: Correlation of ETX measurement results with analytical instruments (2016-2023)

analytical instrument	GC-FID	GC-MS
75 th percentile	+1,8%	+3,0%
median	-1,2%	-0,4%
25 th percentile	-4,4%	-4,6%
number of values	110	166

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

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:

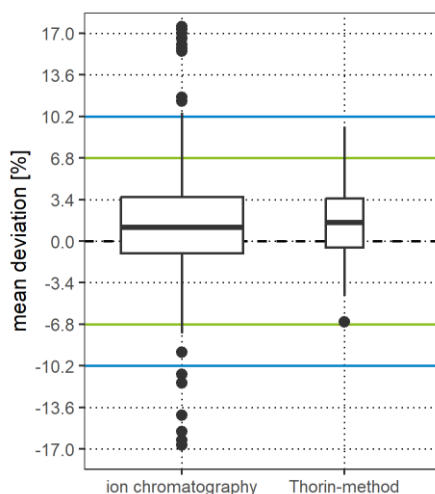


Table 17: Correlation of sulphur dioxide measurement results with the analytical method used (2016-2023)

method	ion chromatography	Thorin-method
75 th percentile	+3,6	+3,5
median	+1,1	+1,5
25 th percentile	-1,0	-0,5
number of values	263	25

The available results do not suggest a significant difference between the two methods, but the number of participants using the Thorin method is comparatively small. The higher dispersion of the IC method with various "outliers" may be due solely 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:

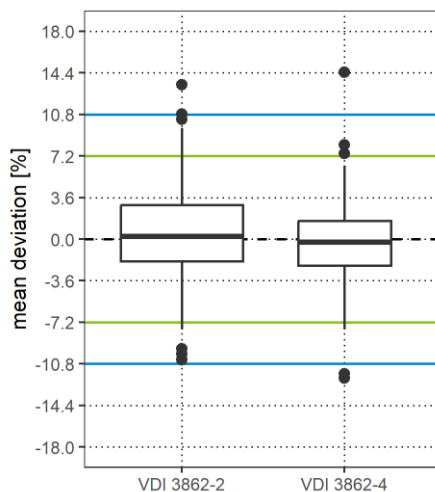


Table 18: Correlation of formaldehyde measurement results with the guidelines used

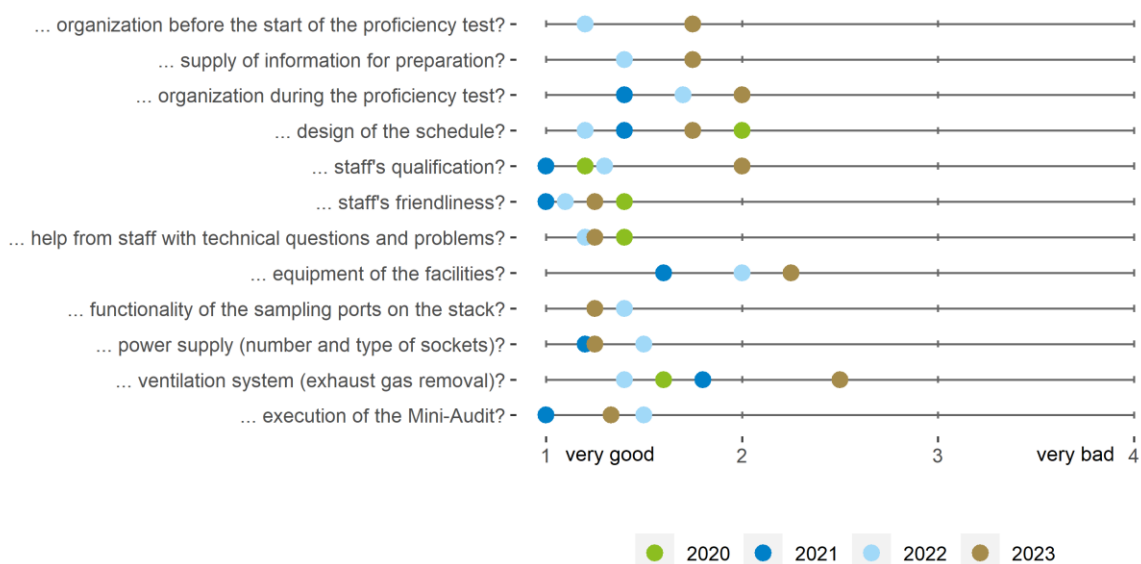
guideline (method)	VDI 3862 Part 2 (DNPH wash bottles)	VDI 3862 Part 4 (AHMT-procedure)
75 th percentile	+3,0	+1,6
median	+0,3	-0,2
25 th percentile	-1,9	-2,3
number of values	119	69

The DNPH wash bottle procedure apparently delivers on average comparable good values as the AHMT procedure.

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 ...



Unfortunately, there was only a total of 4 responses from 3 different participants last year. However, these few pieces of feedback received in 2023 also showed that the participants were highly satisfied overall with the organisation of the proficiency testing scheme. The participants were particularly satisfied with the friendliness and assistance of the staff, the functionality of the measurement openings and the power supply (average score: 1.3 in each case). The lowest ratings were given to the ventilation system (average rating: 2.5) and the equipment of the premises (average rating: 2.3).

One participant criticised the fact that in the gas proficiency test there were sometimes massive increases in TOC concentrations between different measurements that were not announced. The reason for this increase is the change of the ETX mixture after every third measurement, which is associated with a briefly increased dosing rate for flushing the pipes and leads to correspondingly increased concentrations between the sampling periods for the organic substances. The participants are now made aware of the change of solutions and the associated increase in concentration in the preliminary meeting for the proficiency test. In addition, the suggestion was made to start the following dosage in the gas proficiency test as soon as all participants have finished sampling. This is already done on Thursday, but on Wednesday this procedure can only be implemented to a limited extent for technical reasons. For the dust proficiency test, there was feedback that it was unclear whether the measurements in the proficiency test would be carried out at increased humidity. In this regard, reference can be made to the LAI specifications as well as the information sheet on the proficiency test and the measurement of the gas flow conditions by the participants. The participants were and are always informed in advance of significant changes to the conduction of the proficiency test.

It was also criticised that the participants on the 1st floor have less space for their equipment than the participants on the 3rd floor. Unfortunately, due to the number of participants, this problem cannot be easily solved. However, if the participants have a strong interest in a change, it could of course be considered whether the number of participants should be reduced from 8 to 6 per proficiency test (3 per measuring room). In return, the participation fees would have to be increased by at least 33% to approx. 3935 € for the dust proficiency test and approx. 4335 € for the gas proficiency test, in each case plus the expected increase to compensate for the general cost increases of recent years.

There was feedback on the odour proficiency test that the artificial pigsty odour "clogged" the olfactometer and should therefore be replaced by a different odour. There was also criticism that the odour proficiency test in the measuring room sometimes resulted in strong odour nuisance and that the odour concentrations in the proficiency test spread over too wide a range and should always be close to the usual limit values. With regard to the ventilation system, it should be noted that it fulfils the requirements for laboratories with an air exchange rate of at least 8 times per hour and is operated at the highest performance level during the proficiency test. An even more powerful extraction system would be neither economical nor practical, as it would only be used for the odour proficiency test and would not prevent the spread of odours in the measuring room, but would merely reduce it more quickly. This would not solve the fundamental problem of odour pollution in the measuring room. However, the suggestion can be taken up to further minimise the release of odorous substances in the measuring room through organisational measures. In future, the participating persons will be made aware of the need to keep all openings on the ESA closed, e.g. during the preliminary meeting for the proficiency test. In addition, the HLNUG support staff will be sensitised to this problem and will pay more attention to the implementation of this advice in future. Replacing the pigsty mixture with another odourant is out of the question, at least for the time being, as this odour has the greatest practical relevance in terms of the type of odour in the proficiency test and meets the wishes of the majority of participating measuring bodies for the preferential use of multi-component mixtures. Furthermore, the concentrations offered in the proficiency test must scatter over a certain range so that an effective evaluation of measurement results is possible. If this range is too small, participants could simply "guess" a concentration in the usual range and thus automatically pass the proficiency test. In addition, significantly higher odour concentrations are also measured, for example, in measurements to determine the odour reduction effect of systems. Measurement results of 10,000 ou_E/m³ and more are by no means rare in measurement reports in Hessen, and this is probably also the case for other federal states. The desire for concentrations of around 500 ou_E/m³ can therefore only be realised to a limited extent.

8. Concluding Remark

In contrast to the years 2020 to 2022, there were no significant restrictions due to the SARS-CoV-2 pandemic in 2023 when carrying out the proficiency tests at the HLNUG ESA. The dust and gas proficiency tests could therefore be carried out in full in accordance with the new LAI specifications from May 2019. Compared to the old versions from 2007 (dust) and 2014 (gas), these include minor changes to the assessment criteria, the range of components and the schedule, as well as the introduction of a theory test.

The measurement results in the dust and gas proficiency tests developed differently for the various components in 2023 compared to 2022. For many components, the measurement results are still significantly worse than in the years before the 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. For the organic components, the error in many cases is probably not to be found in the sampling, but rather in the sample preparation and especially in the analysis.

In the odour proficiency test, the participants once again achieved significantly better results overall in 2023 than in 2019 to 2021, with the pass rate similar to 2022 and 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 members. 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. In addition, the choice of starting dilution level on the olfactometer could also be a cause of inadequate odour measurement results.

Kassel, 26th February 2024

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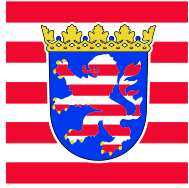
(Dezernatsleiter)

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**, S. 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**, S. 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, S. 100133. <https://doi.org/10.1016/j.aeaoa.2021.100133>*

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