**Hessisches Landesamt
für Naturschutz,
Umwelt und Geologie**

**Hessian Agency
for Nature Conservation,
Environment and Geology**

HESSEN

Annual Report 2022

Results of stack emission proficiency tests for substance ranges P, G, and O on the emission simulation apparatus in the year 2022

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.

DAkkS
Deutsche
Akkreditierungsstelle
D-EP-14551-01-00

Contents

0. About this Report

This report is a translation of „*Jahresbericht 2022 – Ergebnisse der Emissionsringversuche der Stoffbereiche P, G und O an der Emissionssimulationsanlage im Jahr 2022*" 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

In 2022, a total of 33 measuring institutes participated in the dust stack emission proficiency tests (substance range P) of the HLNUG, of which 27 participants were §29b measuring bodies and 6 volunteers. 26 of these participations were in the pandemic version of the proficiency test and were not assessed, 7 measuring institutes participated in the regular version of the proficiency test and were assessed. The success rate was 75% for the 4 assessed §29b measuring bodies, none of the 3 assessed voluntary participants passed the proficiency testing scheme.

In the gas stack emission proficiency tests (substance range G), a total of 34 measuring institutes participated in 2022, of which 28 participants were §29b measuring bodies and 6 volunteers. 25 of these participations were in the pandemic version of the proficiency test and were not assessed, 9 measuring institutes participated in the regular version of the proficiency test and were assessed. The success rate was 40% for the 5 assessed §29b measuring bodies, and of the 4 assessed voluntary participants, 25% passed the proficiency testing scheme.

In 2022, a total of 19 measuring sites took part in the odour stack emission proficiency tests (substance range O), of which 13 participated on the basis of an authorization in accordance with §29b BImSchG and 6 voluntarily. Here, 77% of the authorized and 33% of the voluntary participants 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 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.

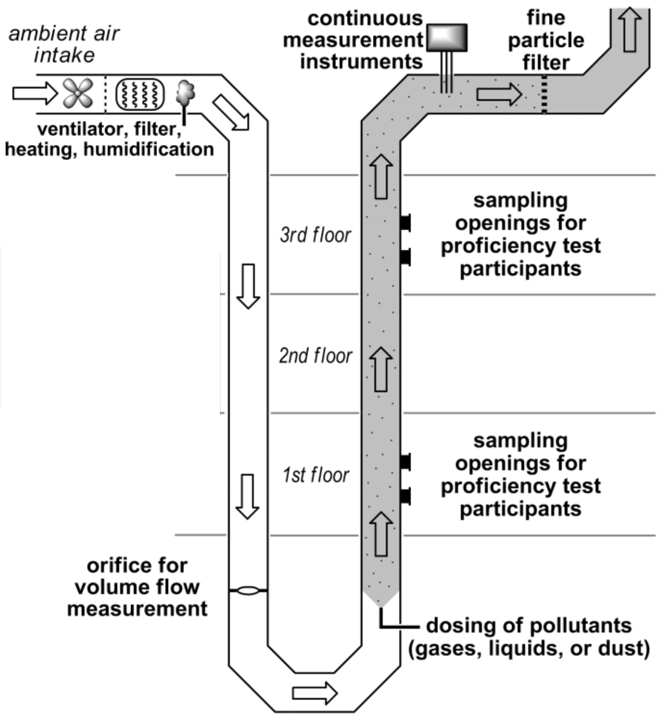
2.2 The Emission Simulation Apparatus

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 m^3/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 calibration gas generator for dosing 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 2022, 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
22G11	Gas (substance range G) - Pandemic version	01.02.2022	01.02.2022	1
22P11	Dust (substance range P) - Pandemic version	01.02.2022	01.02.2022	1
22G12	Gas (substance range G) - Pandemic version	02.02.2022	02.02.2022	2
22P12	Dust (substance range P) - Pandemic version	02.02.2022	02.02.2022	2
22G13	Gas (substance range G) - Pandemic version	03.02.2022	03.02.2022	2
22P13	Dust (substance range P) - Pandemic version	03.02.2022	03.02.2022	2
22G14	Gas (substance range G) - Pandemic version	04.02.2022	04.02.2022	1
22P14	Dust (substance range P) - Pandemic version	04.02.2022	04.02.2022	1
22G21	Gas (substance range G) - Pandemic version	22.02.2022	22.02.2022	2
22P21	Dust (substance range P) - Pandemic version	22.02.2022	22.02.2022	2
22G22	Gas (substance range G) - Pandemic version	23.02.2022	23.02.2022	1
22P22	Dust (substance range P) - Pandemic version	23.02.2022	23.02.2022	1
22G23	Gas (substance range G) - Pandemic version	24.02.2022	24.02.2022	2
22P23	Dust (substance range P) - Pandemic version	24.02.2022	24.02.2022	2
22G24	Gas (substance range G) - Pandemic version	25.02.2022	25.02.2022	2
22P24	Dust (substance range P) - Pandemic version	25.02.2022	25.02.2022	2
22G31	Gas (substance range G) - Pandemic version	15.03.2022	15.03.2022	2
22P31	Dust (substance range P) - Pandemic version	15.03.2022	15.03.2022	2
22G32	Gas (substance range G) - Pandemic version	16.03.2022	16.03.2022	2
22P32	Dust (substance range P) - Pandemic version	16.03.2022	16.03.2022	2
22G33	Gas (substance range G) - Pandemic version	17.03.2022	17.03.2022	1
22P33	Dust (substance range P) - Pandemic version	17.03.2022	17.03.2022	1
22G41	Gas (substance range G) - Pandemic version	05.04.2022	05.04.2022	2
22P41	Dust (substance range P) - Pandemic version	05.04.2022	05.04.2022	2
22G42	Gas (substance range G) - Pandemic version	06.04.2022	06.04.2022	2
22P42	Dust (substance range P) - Pandemic version	06.04.2022	06.04.2022	2
22G43	Gas (substance range G) - Pandemic version	07.04.2022	07.04.2022	2
22P43	Dust (substance range P) - Pandemic version	07.04.2022	07.04.2022	2
22G44	Gas (substance range G) - Pandemic version	08.04.2022	08.04.2022	1
22P44	Dust (substance range P) - Pandemic version	08.04.2022	08.04.2022	1
22O1	Odour (substance range O)	27.09.2022	27.09.2022	8
22O2	Odour (substance range O)	29.09.2022	29.09.2022	6
22O3	Odour (substance range O)	06.10.2022	06.10.2022	5
22P45	Dust (substance range P) - Pandemic version	07.10.2022	07.10.2022	1
22P5	Dust (substance range P)	07.11.2022	08.11.2022	5
22G5	Gas (substance range G)	08.11.2022	10.11.2022	6
22P6	Dust (substance range P)	21.11.2022	22.11.2022	2
22G6	Gas (substance range G)	22.11.2022	24.11.2022	3

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 (3 in the pandemic version)	
sampling	simultaneously for all participants (1st and 3rd 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 (not part of the pandemic version)
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 (six weeks in the pandemic version) 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	
sampling	simultaneously for all participants (1st and 3rd floor)	
basic conditions	2000 … 6000 m^3/h, flow velocity > 4 m/s, water vapour up to 50 g/m^3	
concentrations	approx. 50 … 50000 ou_E/m^3	
result submission	in ou_E/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, Dr. Egill Antonsson 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 SO_2 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 5. Detailed information is given in the results communications of the individual proficiency tests.

4.4 Measurement Methods

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: Compulsory 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) (not part of the pandemic version)
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_{ijk} is calculated from the mass concentration c_{ik} and the odour threshold $c_{0,k}$ of the component:

$$X_{ik} = \frac{c_{ik}}{c_{0,k}}\ \mathrm{ou_E/m^3}$$

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 $c_0 = 123\ \mu g/m^3$. 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 2022, in addition to *n*-butanol the components ‚organic solvent mixture' (ETX), tetrahydrothiophene (THT) and artificial pigsty (PIG) were used. For component ETX, the odour threshold $c_{0,k}$ could be determined with procedure a) from 207 measurements in the years 2017 to 2021, resulting in a consensus value of c_0 = 211 µg/m³. For the components THT and PIG consensus values had to be determined via procedure b). For THT, a value of c_0 = 0.445 µg/m³ and for PIG a value of c_0 = 242 µg/m³ was calculated, both on the basis of 39 measurements in 2022.

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 2022, as in the year before that, this was only necessary for component PIG, where σ_k had to be raised to a value of 0.24. 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. For components that are not part of these specifications, criteria were established by the HLNUG using a comparable procedure. The values are for the individual components:

Table 5: Precision criteria

No.	component	measurement mode	short designation	precision criterion σ_k in % of true value	maximum for standard uncertainty of assigned values [%]
			substance range P		
P1	dust	discontinuous	St	7.0	1,59
P2	Cadmium	discontinuous	Cd	10.0	1,86
P3	Cobalt	discontinuous	Co	10.0	1,91
P4	Chromium	discontinuous	Cr	10.0	1,89
P5	Copper	discontinuous	Cu	10.0	2,21
P6	Manganese	discontinuous	Mn	10.0	2,02
P7	Nickel	discontinuous	Ni	10.0	1,96
P8	Lead	discontinuous	Pb	10.0	1,88
P9	Vanadium	discontinuous	V	10.0	2,16
			substance range G		
G1	NO_x as NO_2	continuous	Nk	3.1	1.03
G2	CO	continuous	Kk	3.6	1.08
G3	TOC	continuous	Ck	3.3	1.08
G4	ethylbenzene	discontinuous	Ed	4.1	1.01
G5	toluene	discontinuous	Td	4.1	1.01
G6	sum of *o*-, *m*-, *p*-xylene	discontinuous	Xd	4.1	1.01
G7	SO_2	discontinuous	Sd	3.4	1.11
G8	formaldehyde	discontinuous	Fd	3.6	1.17
			substance range O		
O1	*n*-butanol	discontinuous	NBU	0.10 †	1.01
O2	solvent mixture	discontinuous	ETX	0.10 †	5.86
O3	tetrahydrothiophene	discontinuous	THT	0.10 †	6.70
O4	artificial pigsty odour	discontinuous	PIG	0.24 †	17.5

† In proficiency test O the precision criterion is not expressed in % of true value (see section 4.5.1)

4.5.3 Assessment Scheme

Interpretation of the z-scores

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

$\lvert z_{ijk}\rvert \leq 2$	satisfactory
$2 < \lvert z_{ijk}\rvert < 3$	questionable
$\lvert z_{ijk}\rvert \geq 3$	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) 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 interpetration 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

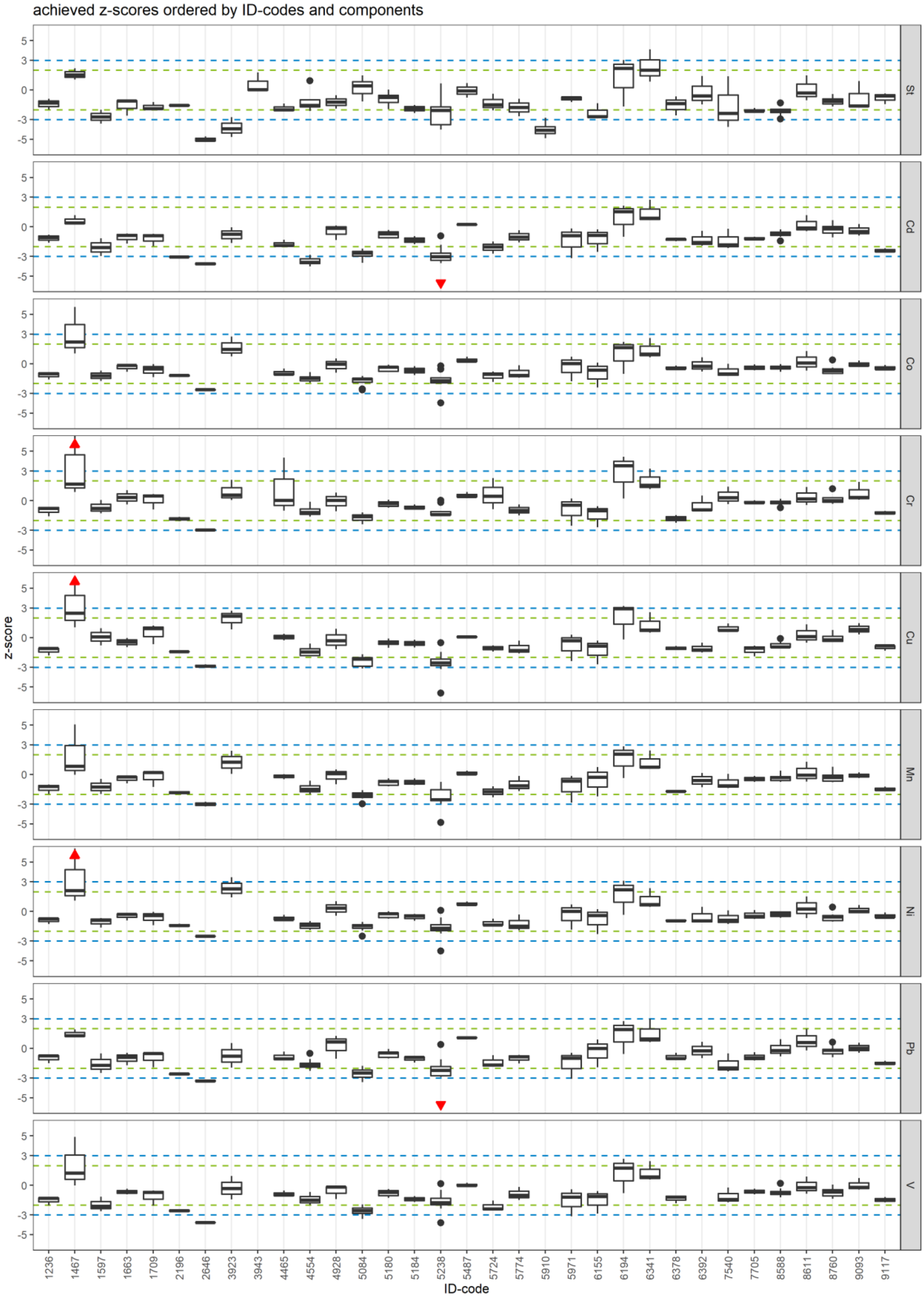
5.1 z-Scores

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.

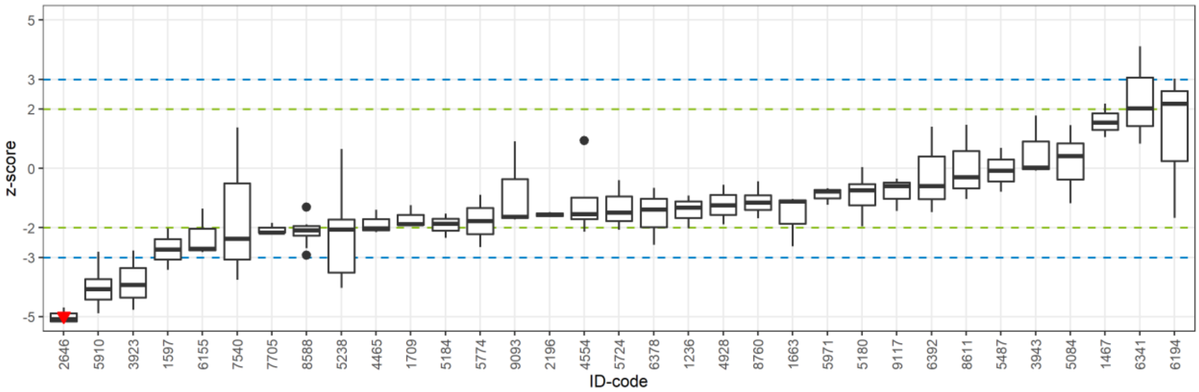
A list of the individual measurements of all participants can be found in a separate document (appendix to the annual report).

5.1.1 Dust Proficiency Test (Substance Range P)

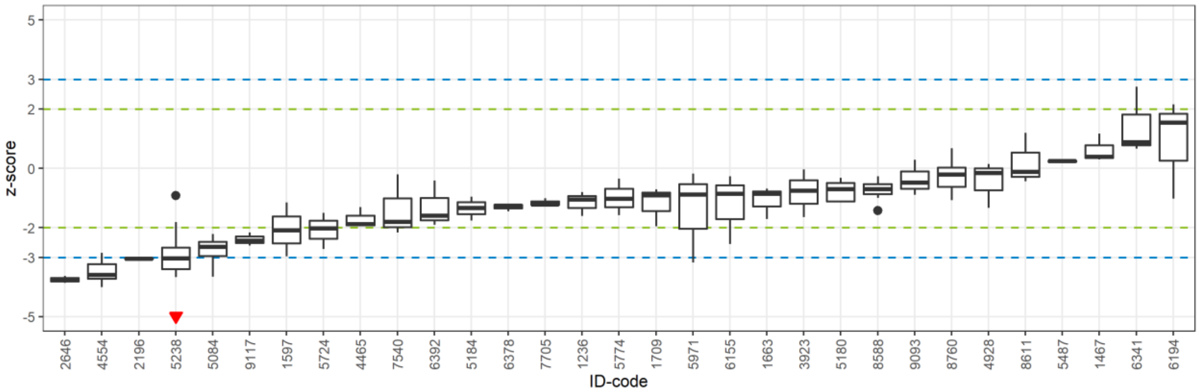


Scheme 2: Achieved z-scores dust proficiency test

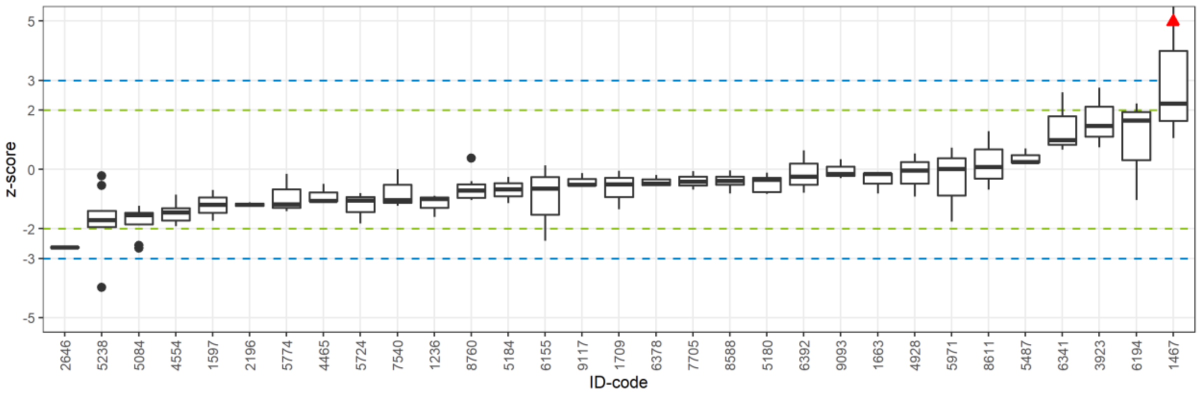
z-scores for component **dust concentration** (St)



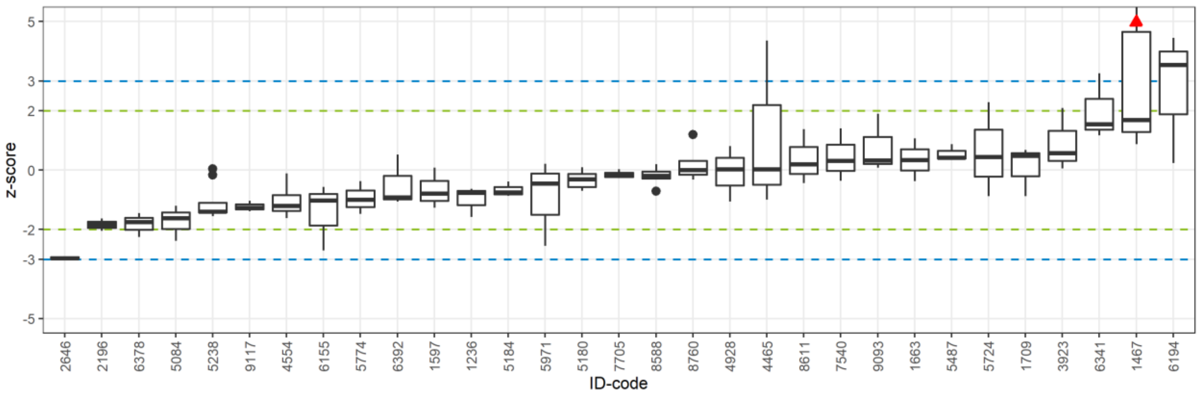
z-scores for component **Cadmium** (Cd)



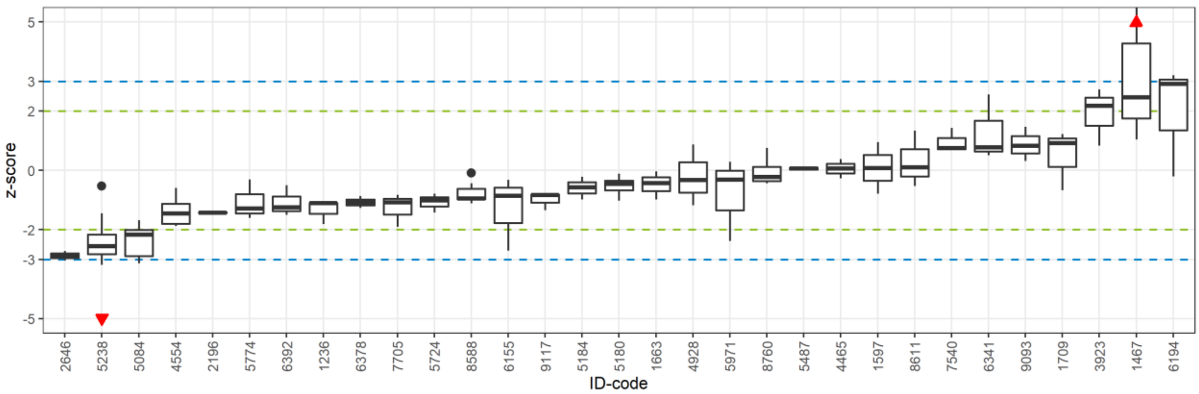
z-scores for component **Cobalt** (Co)



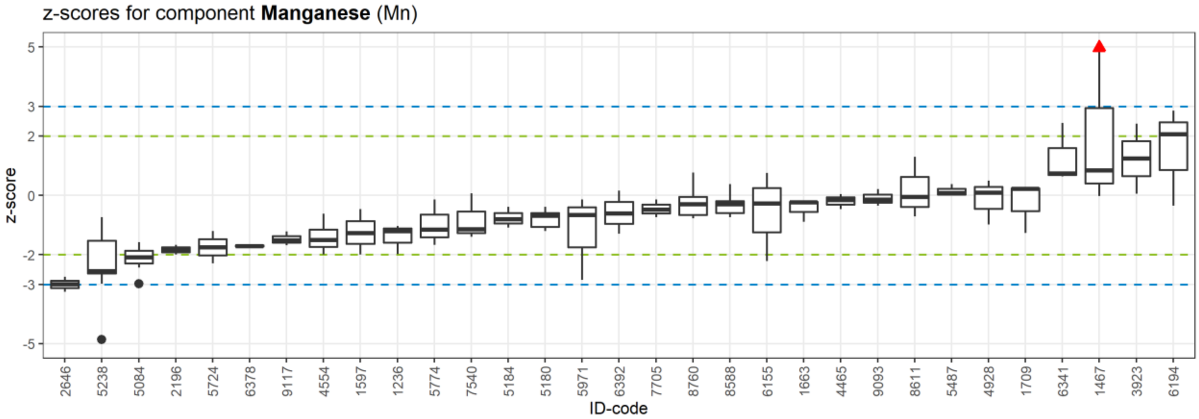
z-scores for component **Chromium** (Cr)

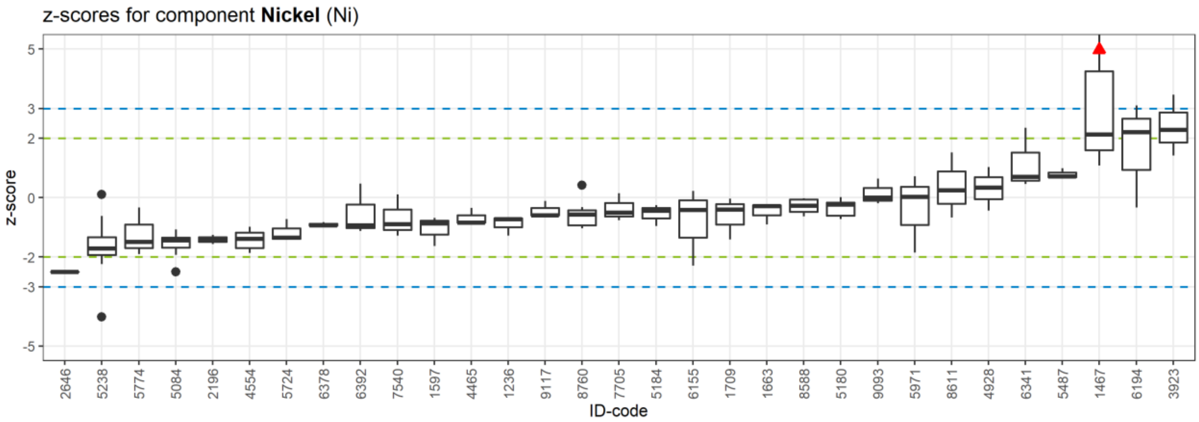


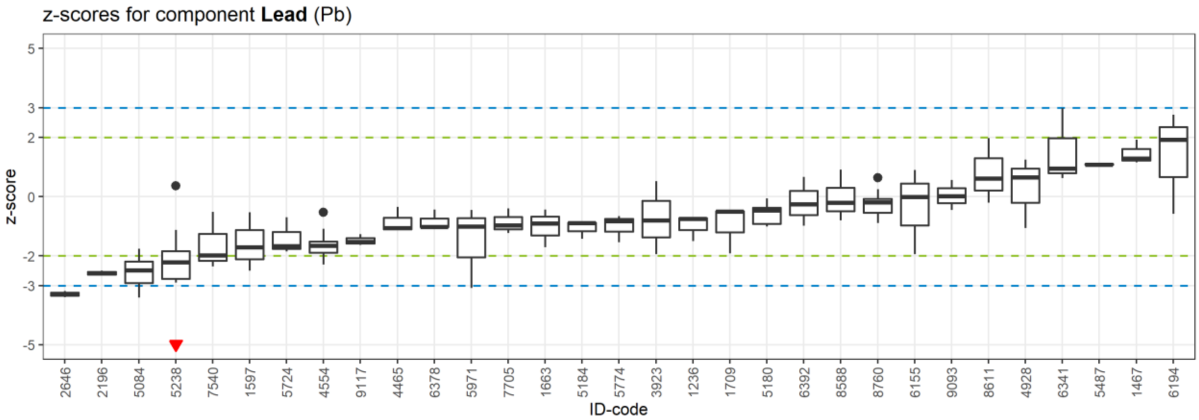
z-scores for component **Copper** (Cu)

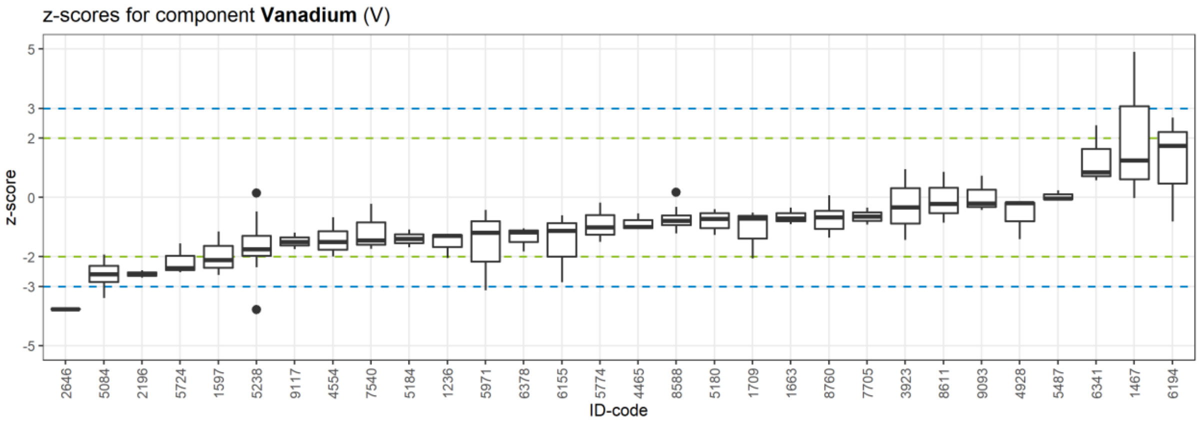


z-scores for component **Manganese** (Mn)

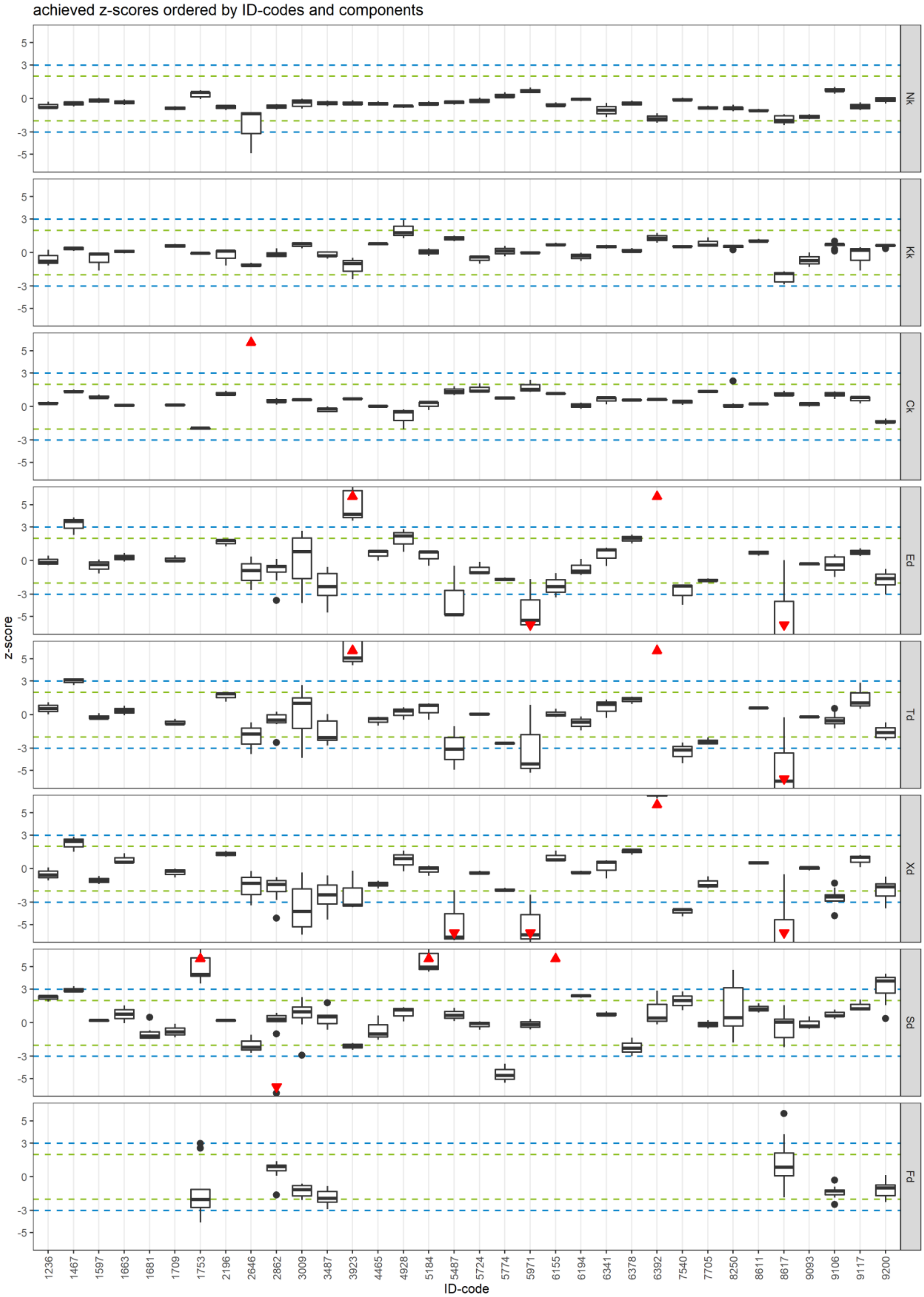



z-scores for component **Nickel** (Ni)
z-score
5
3
2
0
-2
-3
-5
2646 5238 5774 5084 2196 4554 5724 6378 6392 7540 1597 4465 1236 9117 8760 7705 5184 6155 1709 1663 8588 5180 9093 5971 8611 4928 6341 5487 1467 6194 3923
ID-code


z-scores for component **Lead** (Pb)
z-score
5
3
2
0
-2
-3
-5
2646 2196 5084 5238 7540 1597 5724 4554 9117 4465 6378 5971 7705 1663 5184 5774 3923 1236 1709 5180 6392 8588 8760 6155 9093 8611 4928 6341 5487 1467 6194
ID-code

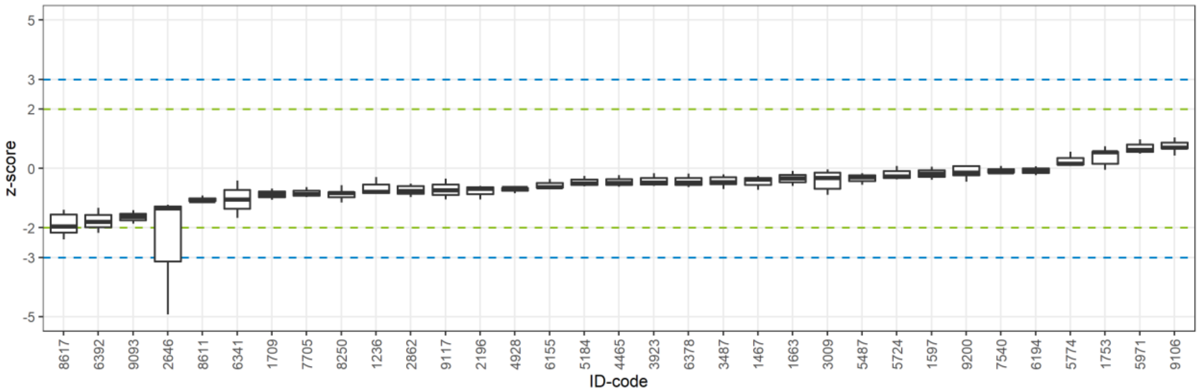

z-scores for component **Vanadium** (V)
z-score
5
3
2
0
-2
-3
-5
2646 5084 2196 5724 1597 5238 9117 4554 7540 5184 1236 5971 6378 6155 5774 4465 8588 5180 1709 1663 8760 7705 3923 8611 9093 4928 5487 6341 1467 6194
ID-code

5.1.2 Gas Proficiency Test (Substance Range G)

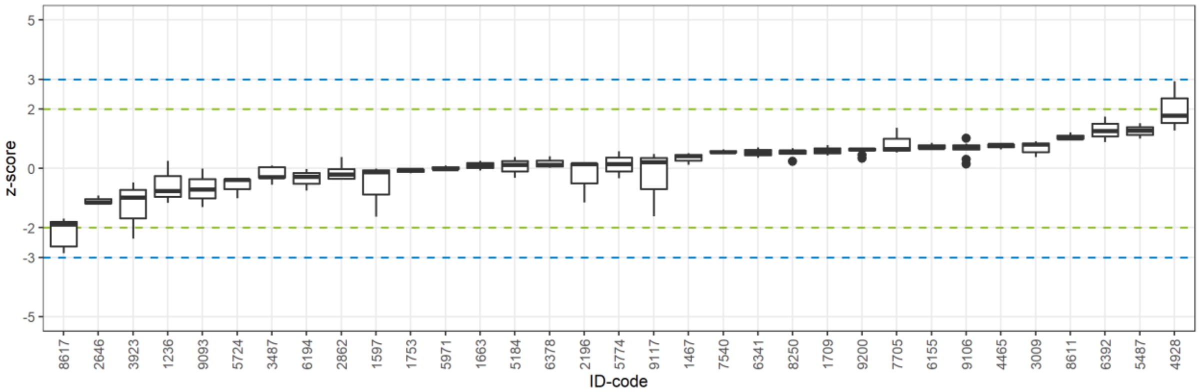


Scheme 3: Achieved z-scores gas proficiency test

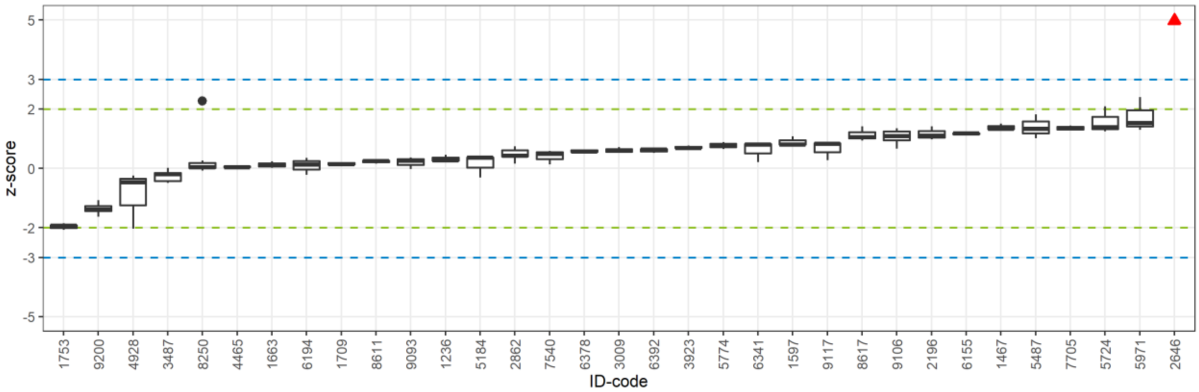
z-scores for component **NOx** (Nk)



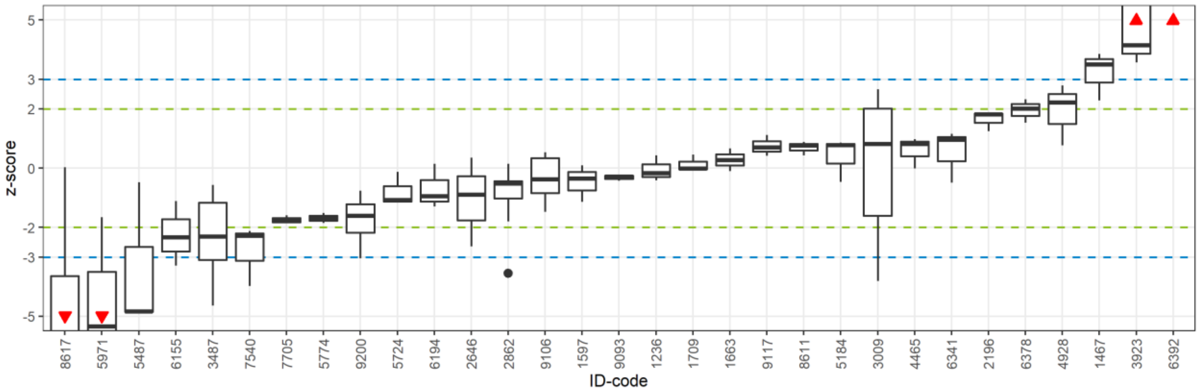
z-scores for component **carbon monoxide** (Kk)



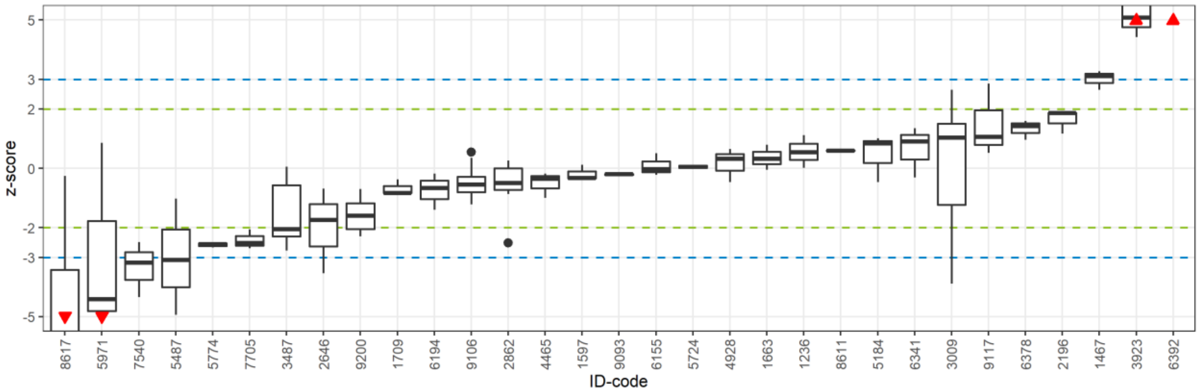
z-scores for component **TOC** (Ck)



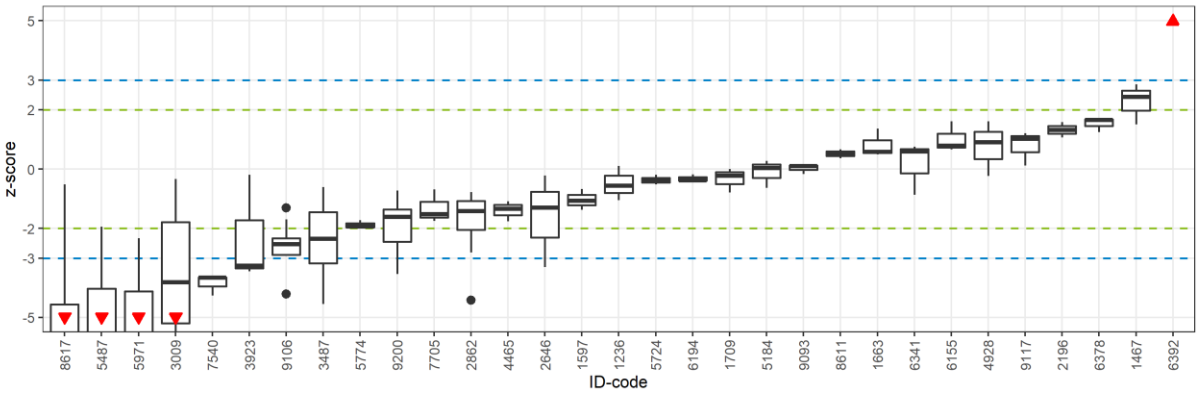
z-scores for component **ethylbenzene** (Ed)



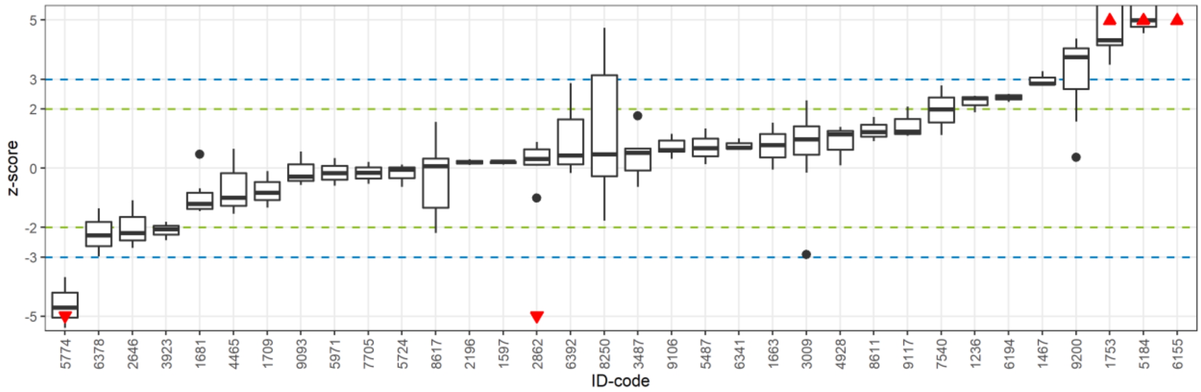
z-scores for component **toluene** (Td)



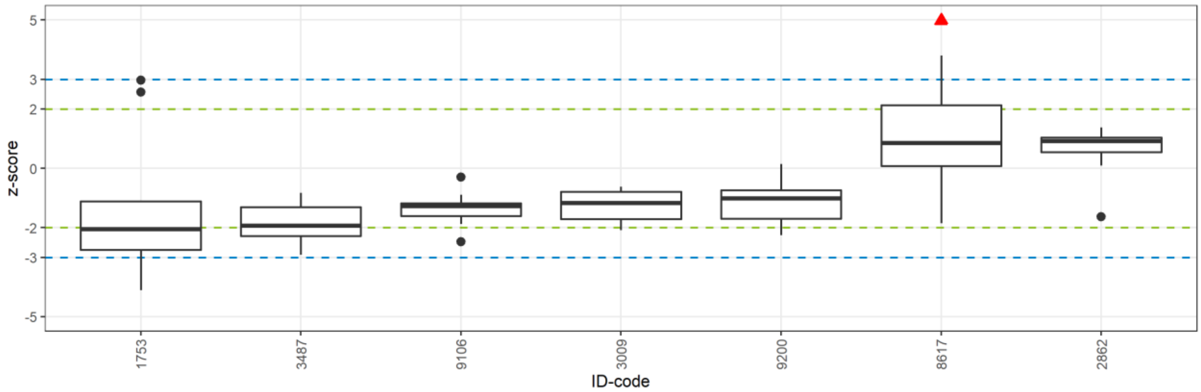
z-scores for component **xylene (sum)** (Xd)



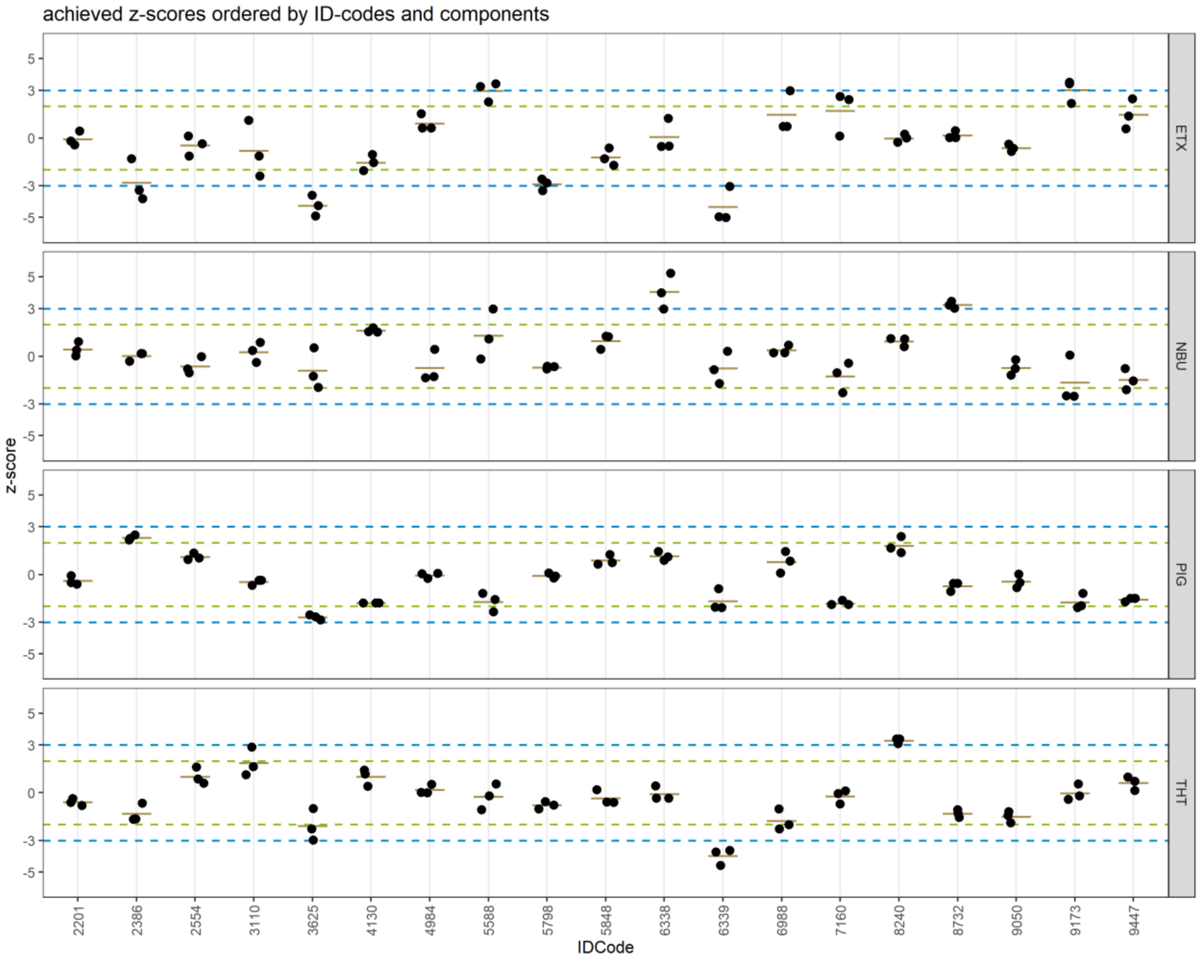
z-scores for component **SO2 discont.** (Sd)



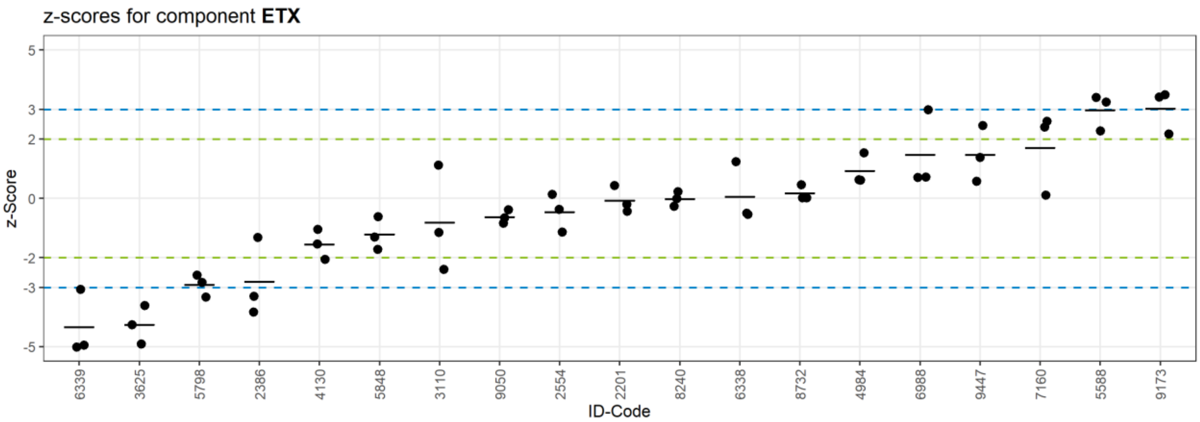
z-scores for component **formaldehyde** (Fd)

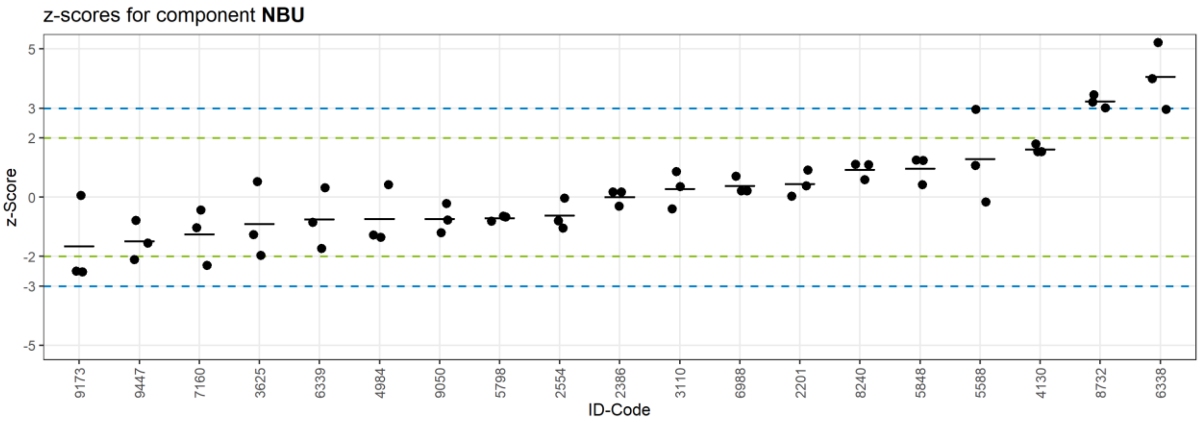


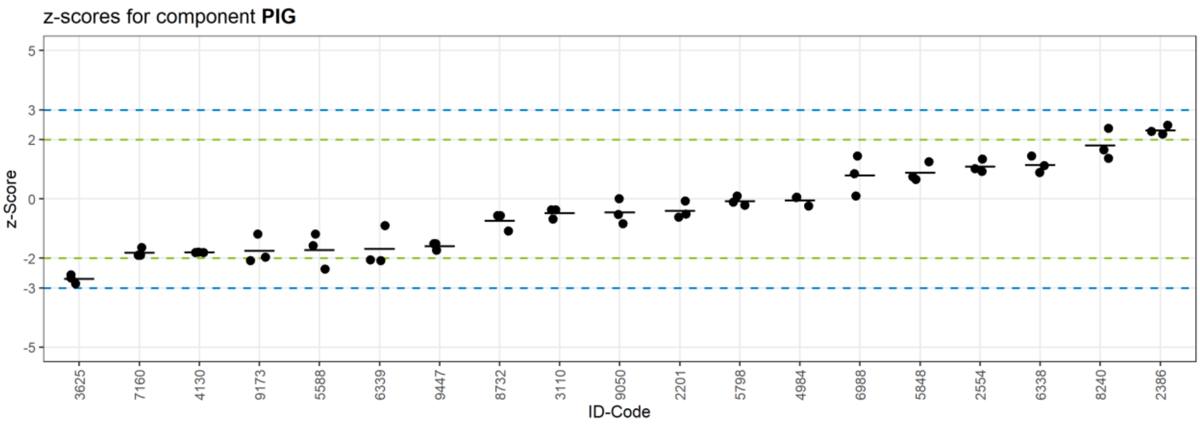
5.1.3 Odour Proficiency Test (Substance Range O)

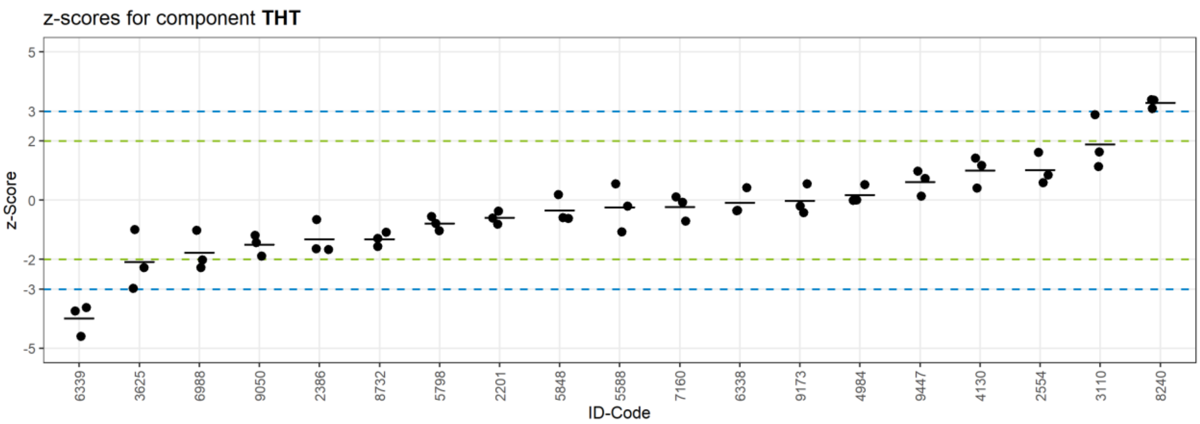


Scheme 4: Achieved z-scores odour proficiency test (only values in the range -5 ... 5 are shown)




z-scores for component NBU
z-Score
ID-Code
9173
9447
7160
3625
6339
4984
9050
5798
2554
2386
3110
6988
2201
8240
5848
5588
4130
8732
6338


z-scores for component PIG
z-Score
ID-Code
3625
7160
4130
9173
5588
6339
9447
8732
3110
9050
2201
5798
4984
6988
5848
2554
6338
8240
2386


z-scores for component THT
z-Score
ID-Code
6339
3625
6988
9050
2386
8732
5798
2201
5848
5588
7160
6338
9173
4984
9447
4130
2554
3110
8240

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 the gas (pandemic version) and odour proficiency testing schemes either no measurements were performed, or no assement criteria were defined. For each participant, only one (dust, pandemic version) or two (standard dust and gas proficiency tests) values are available per component, these are shown as dots. If for a participant two values are available, the mean value of these two is marked by a horizontal line.

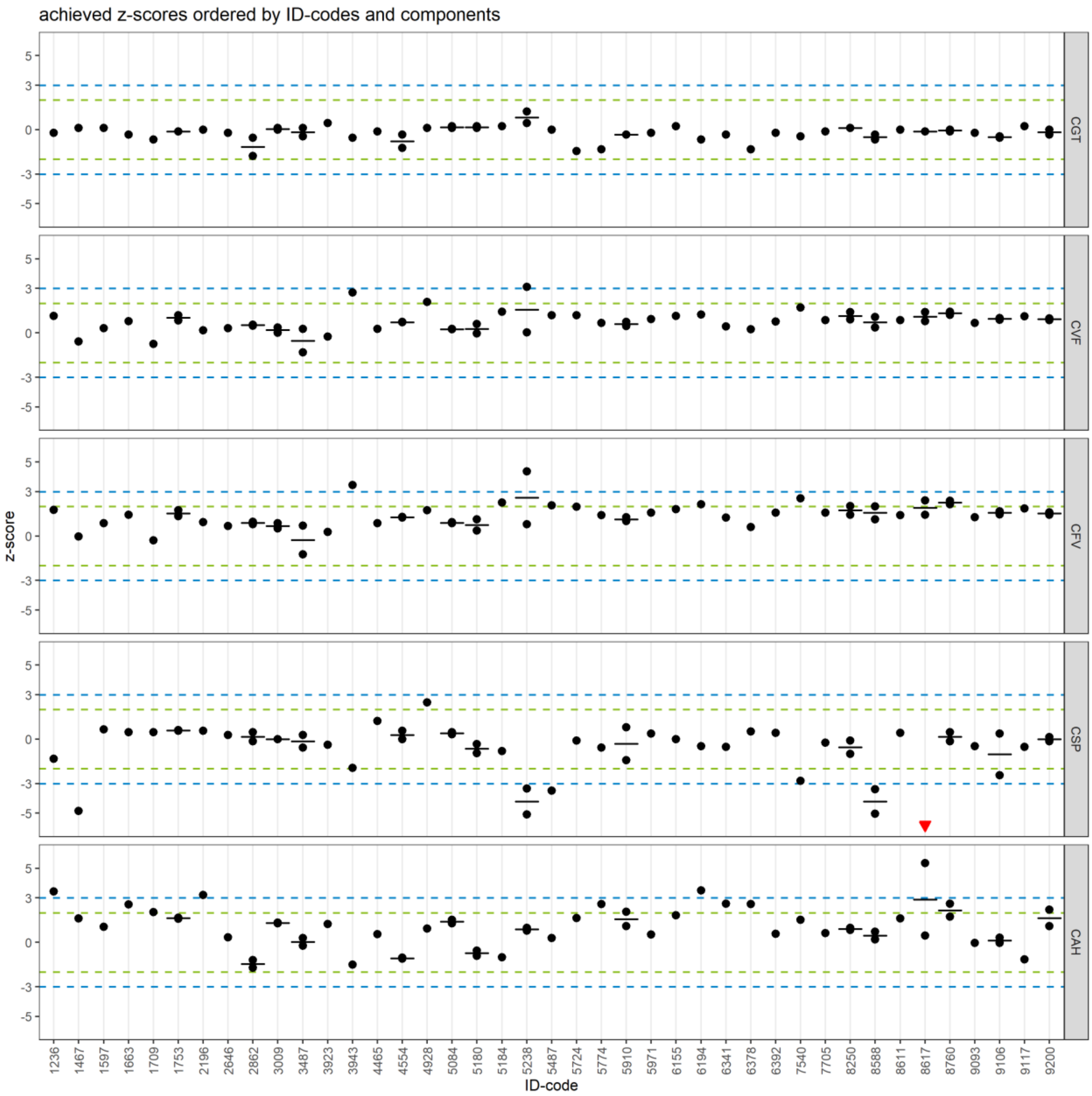
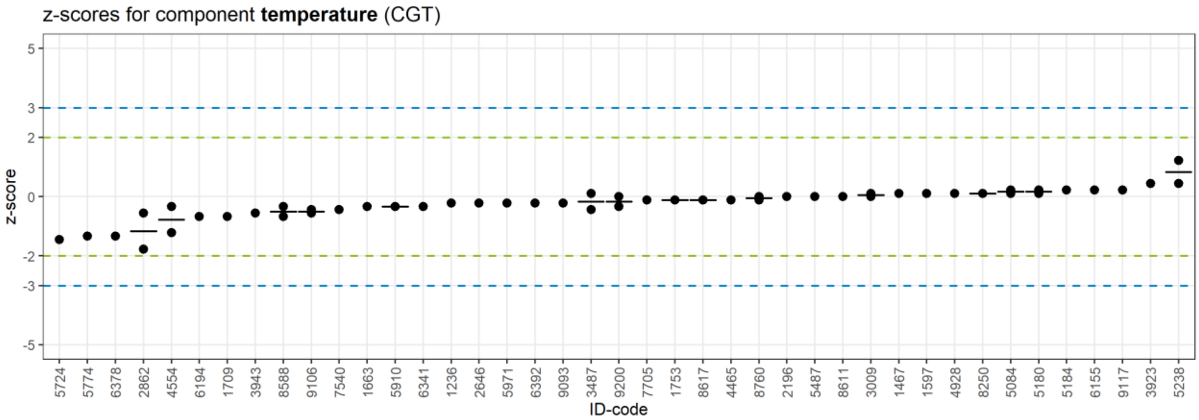
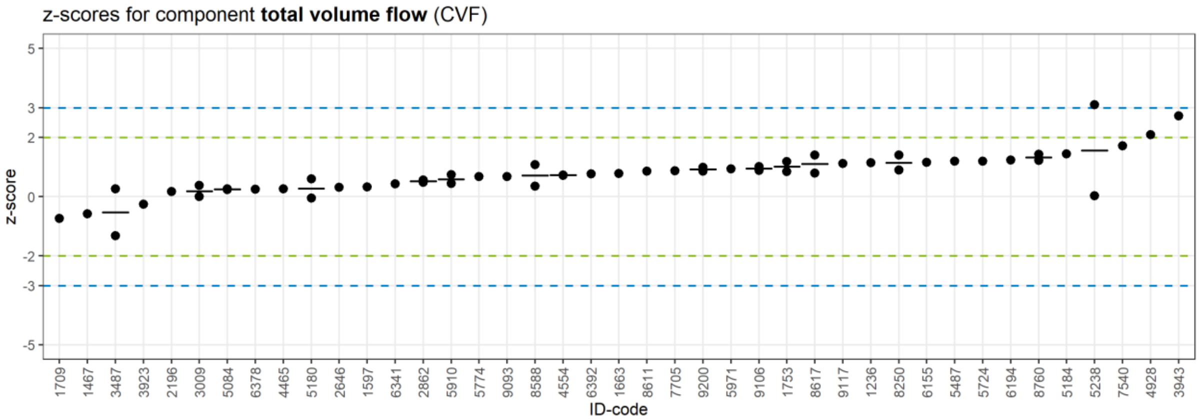
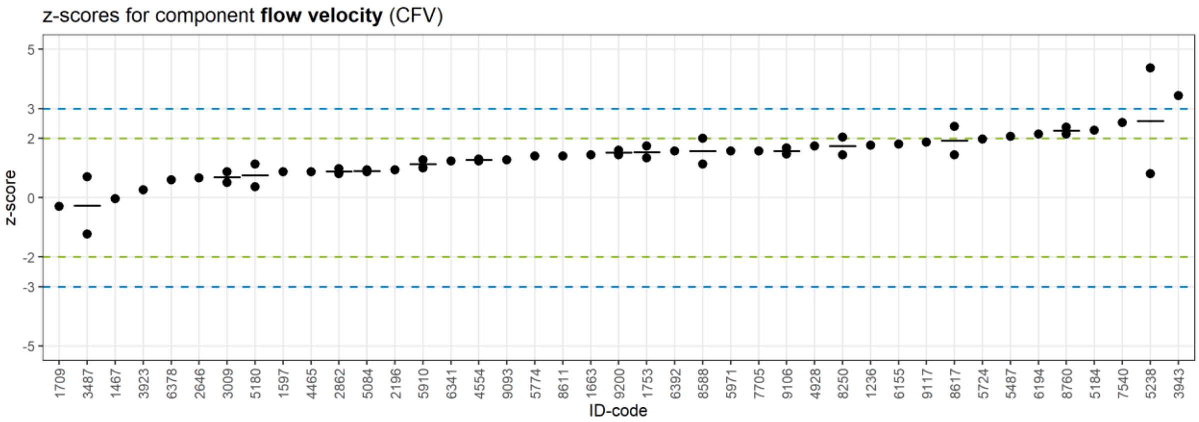
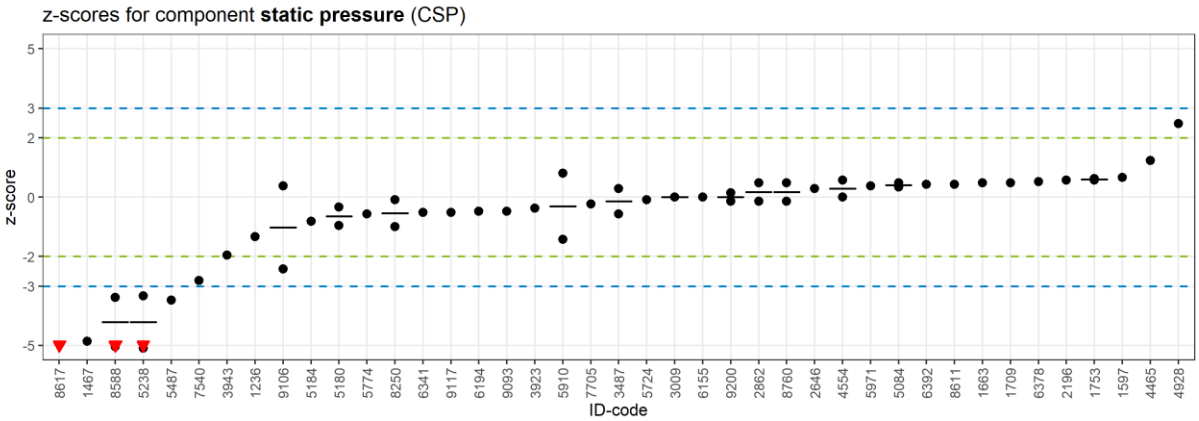


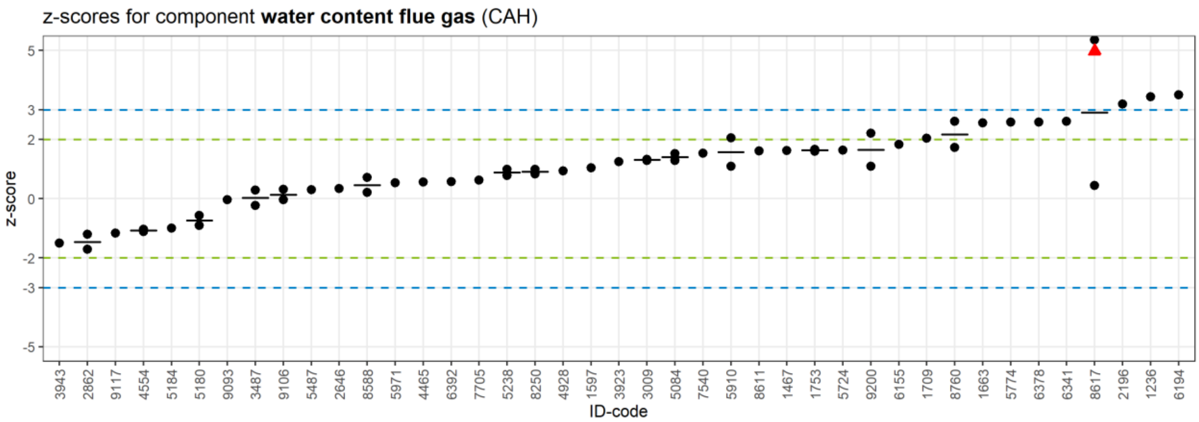
Abbildung 5: z-scores (or quotients from participant deviation and typical deviation) for gas flow conditions


z-scores for component **temperature** (CGT)
z-score
ID-code
5724 5774 6378 2862 4554 6194 1709 3943 8588 9106 7540 1663 5910 6341 1236 2646 5971 6392 9093 3487 9200 7705 1753 8617 4465 8760 2196 5487 8611 3009 1467 1597 4928 8250 5084 5180 5184 6155 9117 3923 5238


z-scores for component **total volume flow** (CVF)
z-score
ID-code
1709 1467 3487 3923 2196 3009 5084 6378 4465 5180 2646 1597 6341 2862 5910 5774 9093 8588 4554 6392 1663 8611 7705 9200 5971 9106 1753 8617 9117 1236 8250 6155 5487 5724 6194 8760 5184 5238 7540 4928 3943


z-scores for component **flow velocity** (CFV)
z-score
ID-code
1709 3487 1467 3923 6378 2646 3009 5180 1597 4465 2862 5084 2196 5910 6341 4554 9093 5774 8611 1663 9200 1753 6392 8588 5971 7705 9106 4928 8250 1236 6155 9117 8617 5724 5487 6194 8760 5184 7540 5238 3943

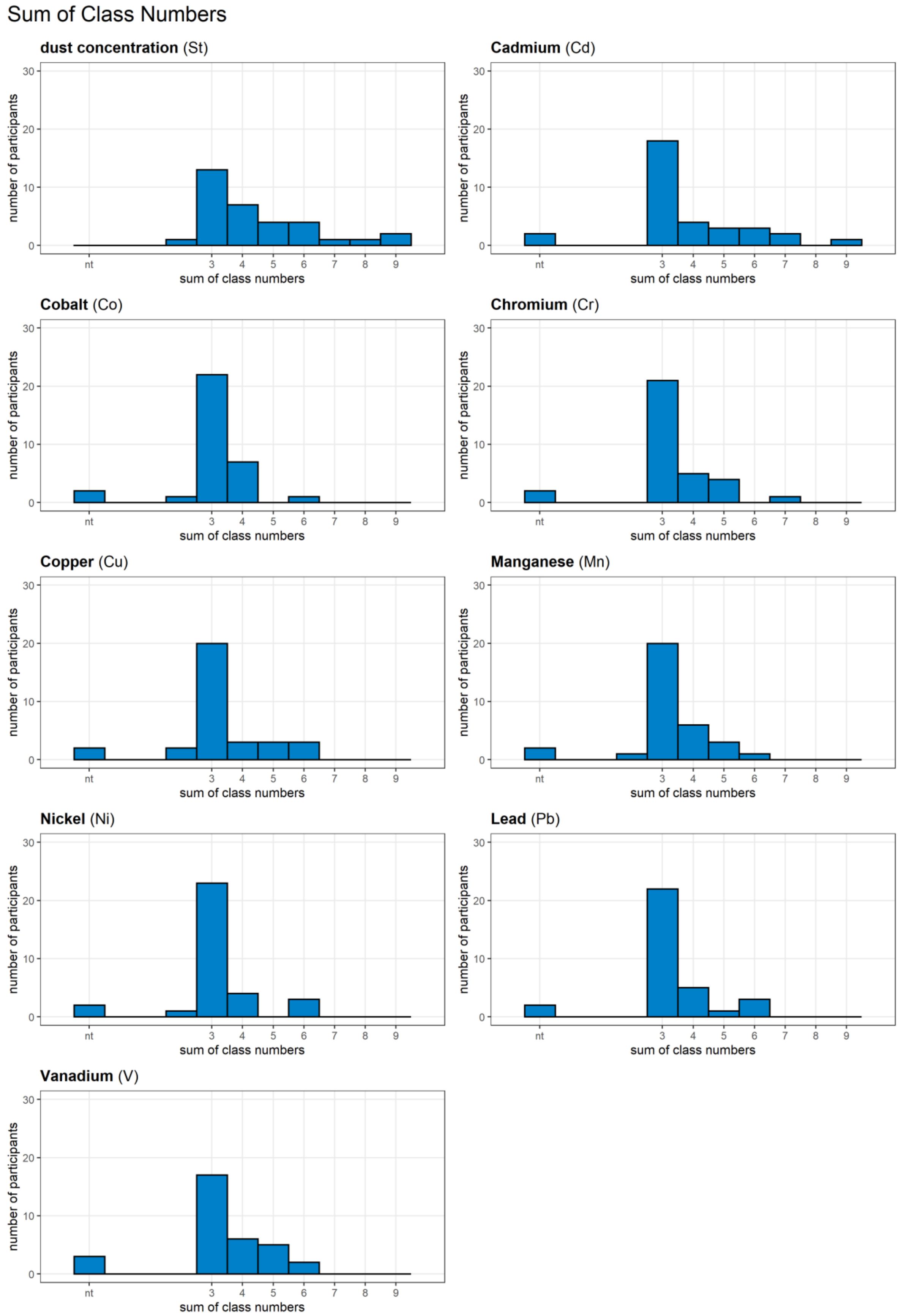




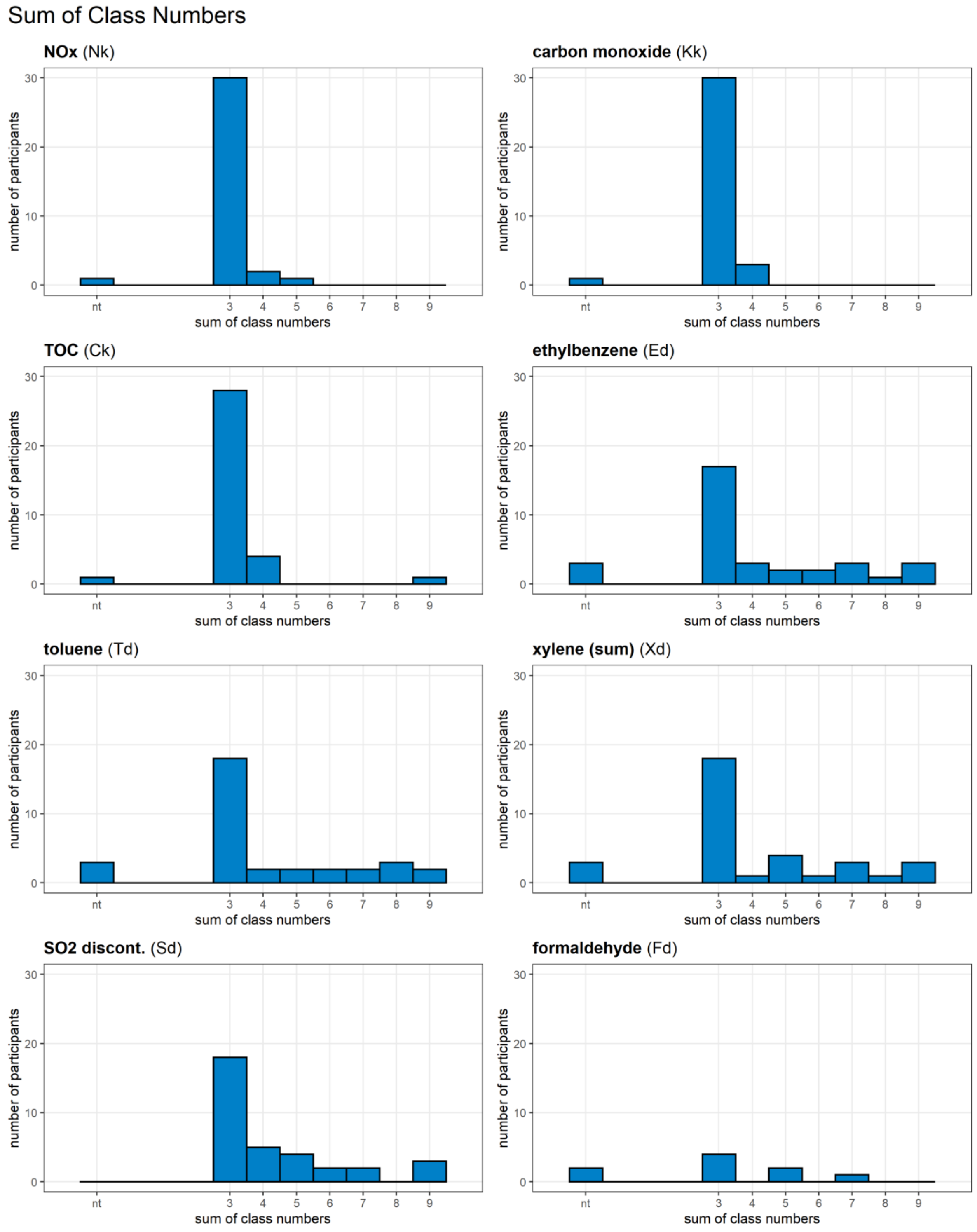
5.2 Sums of Class Numbers

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)

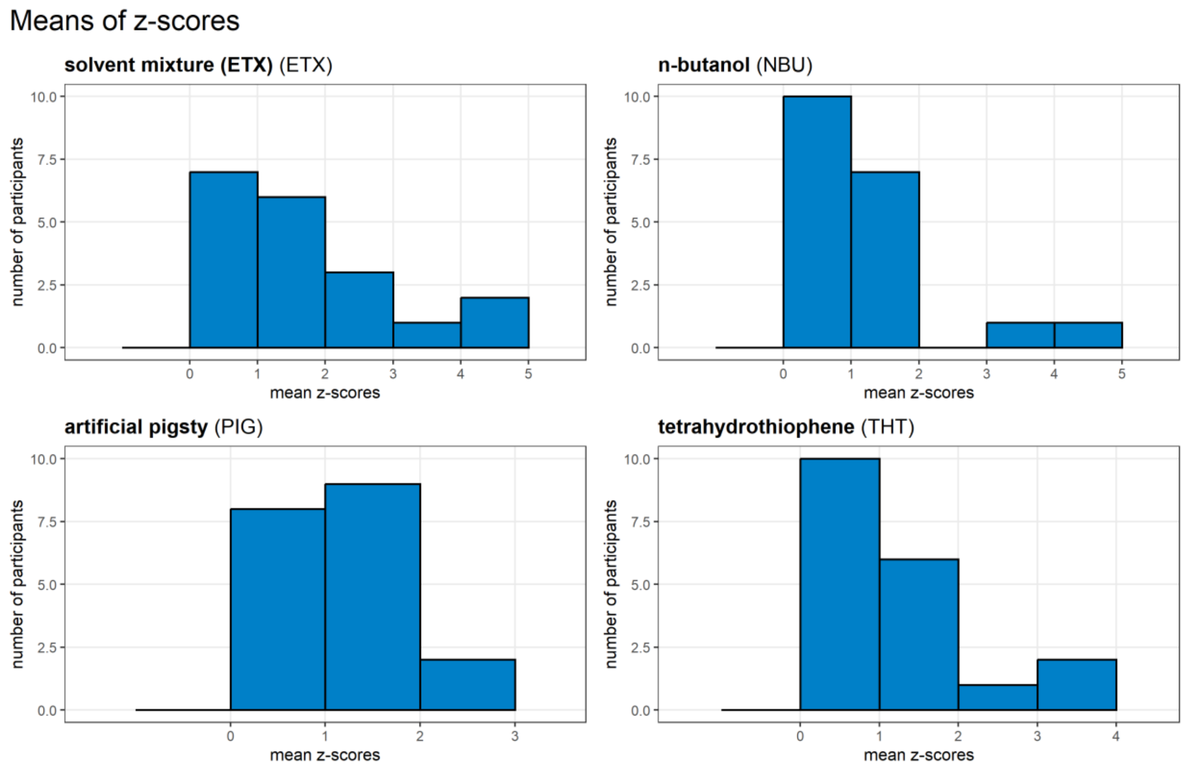


5.2.2 Gas Proficiency Test (Substance range G)



5.2.3 Odour Proficiency Test (Substance Range O)

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.



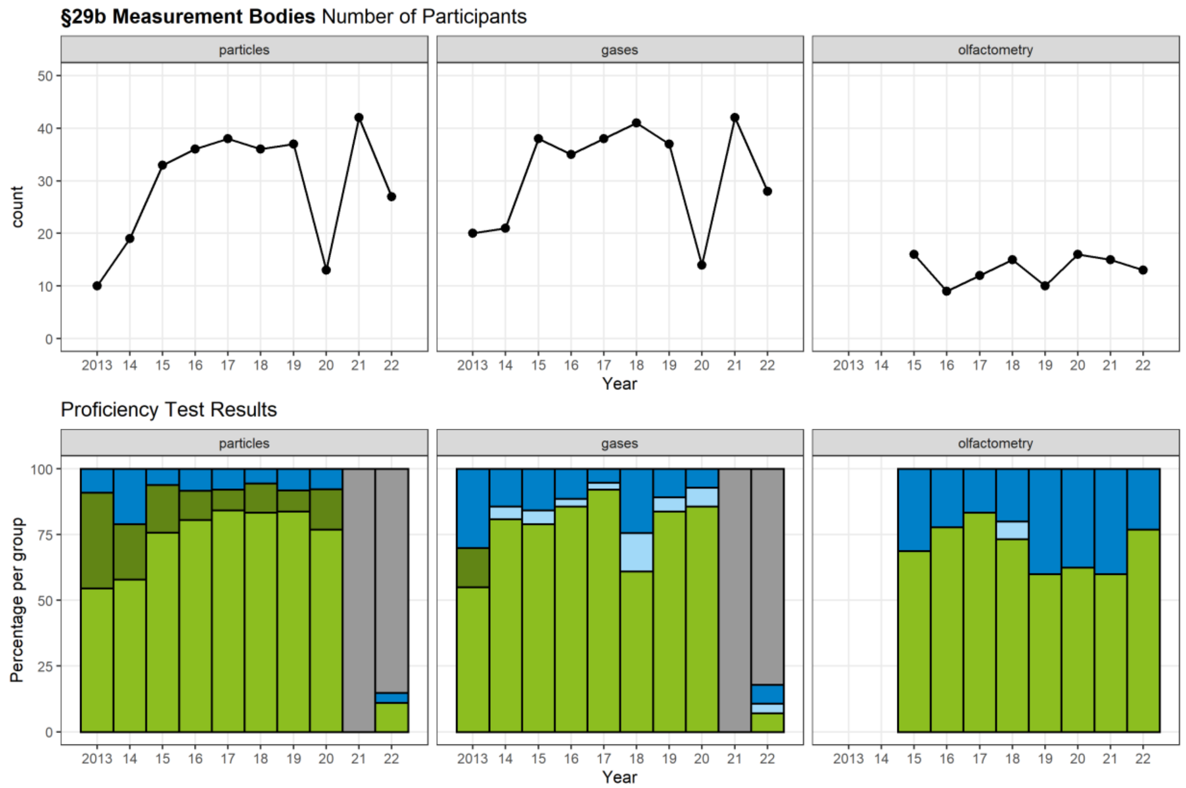
5.3 Theory Test

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.

All participants passed the theory test on the dust proficiency test in 2022, with a median score of 25 out of 33. The bottom quarter of the participants scored 22 points or less, the top quarter scored more than 28 points.

In 2022, 89% of all participants passed the theory test for the gas proficiency test, with a median score of 21 out of 33 points. The bottom quarter of the participants scored 19 points or less in the theory test, the top quarter scored 25 points or more.

6. Interpretation of Results



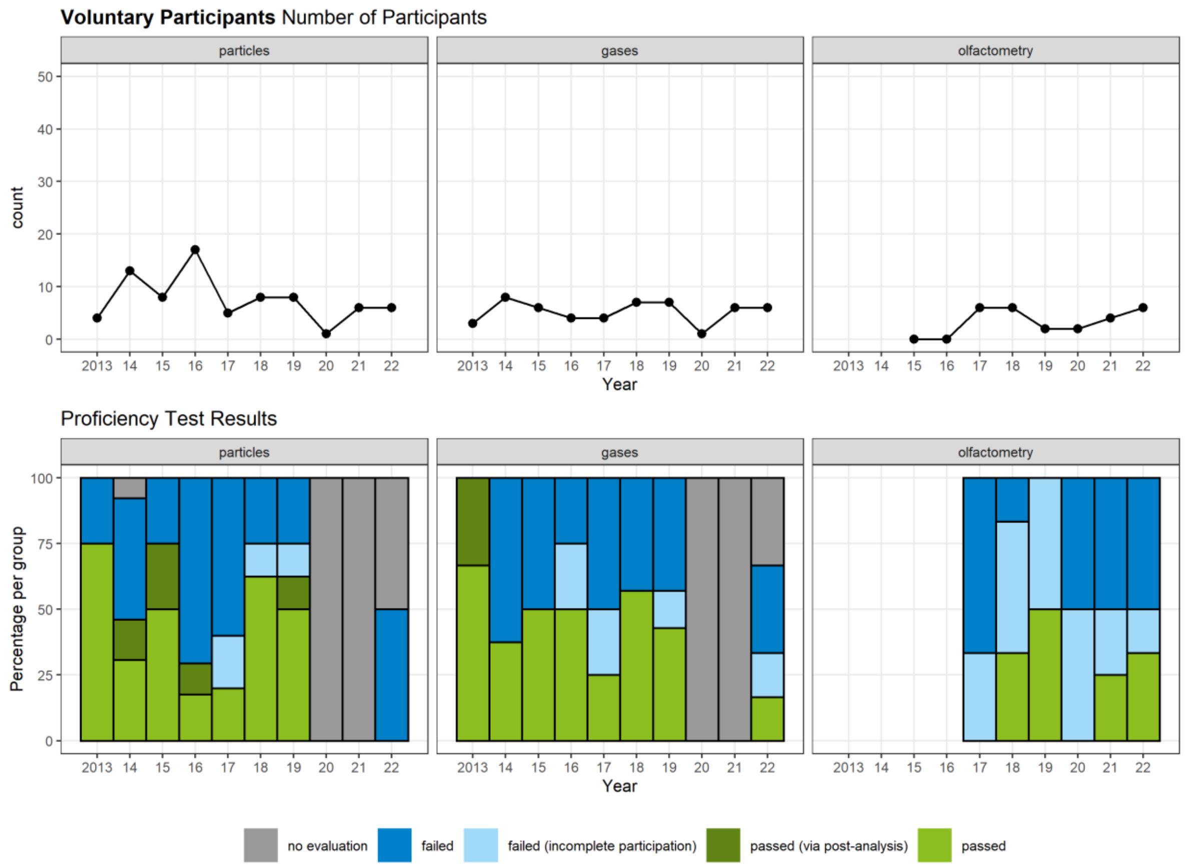


Table 6: Overview of results since 2018 (§29b-bodies)

year	proficiency test	passed	passed (via post-analysis)	failed	failed (incomplete participation)
2018	dust	30	4	2	-
	gas	25	-	10	6
	odour	11	-	3	1
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 (not evaluated)			
	gas (pandemic)	42 (not evaluated)			
	odour	9	-	6	-
2022	dust	3	-	1	-
	dust (pandemic)	23 (not evaluated)			
	gas	2	-	2	1
	gas (pandemic)	23 (not evaluated)			
	odour	10	-	3	-

Table 7: Overview of results since 2018 (voluntary participants)

year	proficiency test	passed	passed (via post-analysis)	failed	failed (incomplete participation)
2018	dust	5	-	2	1
	gas	4	-	3	-
	odour	2	-	1	3
2019	dust	4	1	2	1
	gas	3	-	3	1
	odour	1	-	-	1
2020	dust	-	-	-	-
	gas	-	-	-	-
	odour	-	-	1	1
2021	dust (pandemic)	6 (not evaluated)			
	gas (pandemic)	6 (not evaluated)			
	odour	1	-	2	1
2022	dust	-	-	3	-
	dust (pandemic)	3 (not evaluated)			
	gas	1	-	2	1
	gas (pandemic)	2 (not evaluated)			
	odour	2	-	3	1

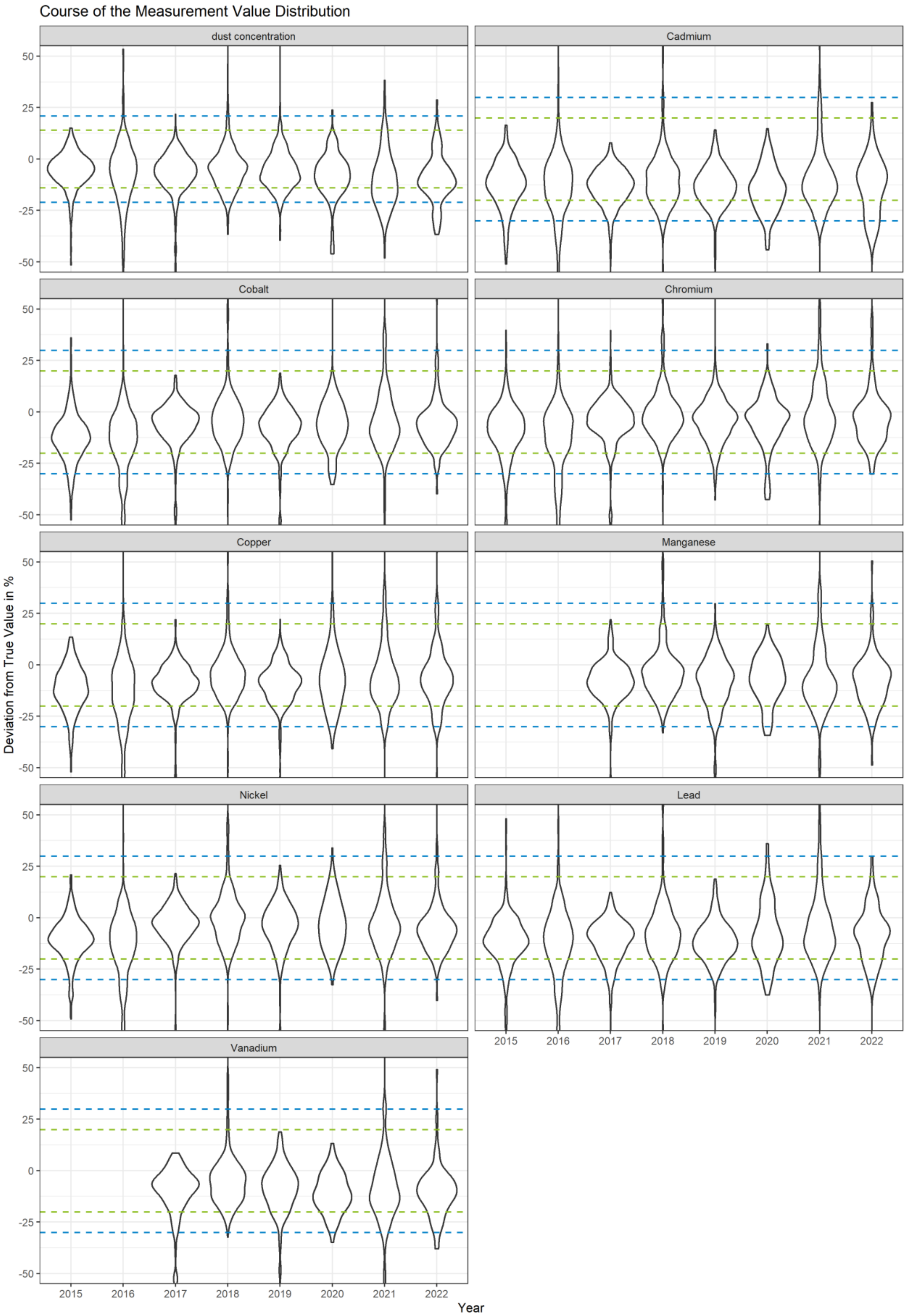
6.1 §29b Measuring Bodies

The year 2022 was again marked by the effects of the SARS-CoV-2 pandemic. While the odour proficiency tests could still be carried out essentially unchanged, the dust and gas proficiency tests were again initially carried out in the "pandemic version" created in 2021. In deviation from the stipulations of the LAI specifications, the number of measurements carried out and assessed was reduced from 9 to 3 and the component formaldehyde was not offered. At the same time, various evaluation criteria were adapted in both proficiency tests. The z-scores achieved by the participants are therefore only comparable with the previous years to a limited extent. Due to the deviations from the specifications, no overall evaluations were carried out for the dust and gas proficiency tests in the pandemic version. If this had been carried out, a total of 18 out of 23 (78%) of the authorized monitoring bodies would have passed the pandemic dust proficiency test and 14 out of 23 (61%) the pandemic gas proficiency test. If the results of the proficiency tests in November 2022, which could again be carried out completely in accordance with LAI specifications, are added, the overall pass rate is 78% (21 of 27 participations) for the dust proficiency test and 57% (16 of 28 participations) for the gas proficiency test. The pass rates are thus again slightly (dust) or significantly (gas) lower than in the years before the pandemic, but at least higher than in 2021.

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 2022 than in the years before the pandemic. In the pandemic version offered in 2021 and 2022, there were two decisive changes compared to the proficiency tests up to 2020: The number of measurements was reduced from 9 to 3, which should have no influence on the mean deviation of the submitted measured values from the target values. In addition, due to the hygiene concept, the participants had no opportunity for an exchange with other participants. Whether this had an influence on the submitted measured values cannot be verified. However, the changes in the deviation of the measurement results from the respective target values are striking.

In the years 2015 to 2020, 79% of the measured total dust concentrations (across all participants, both §29b monitoring bodies and volunteers) had a deviation of ≤ 14% from the target value (corresponding to a z-score of ≤ 2), 13% of the measured values had a deviation of 14-21% (z-score 2-3), and 8% of all measured values had a deviation of more than 21% from the target value (z score > 3). In 2021, however, only 52% of all measured values had a deviation of 14% or less from the target value, 27% were at 14-21% deviation, and 21% of all measured values had a deviation of more than 21%. In 2022, the results were slightly better again, but still not back to the level of 2015-2020. Last year, 65% of all measured values were at a maximum of 14% deviation from the target value, 21% were at 14-21% deviation, and 14% of all measured values were at a higher deviation.

A graphical representation of the distribution of the measured values in the dust proficiency tests of the past years can be seen in the following figure. Here, for each component and for each year since 2015, the distribution of the measured values 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 lie in the relevant range.

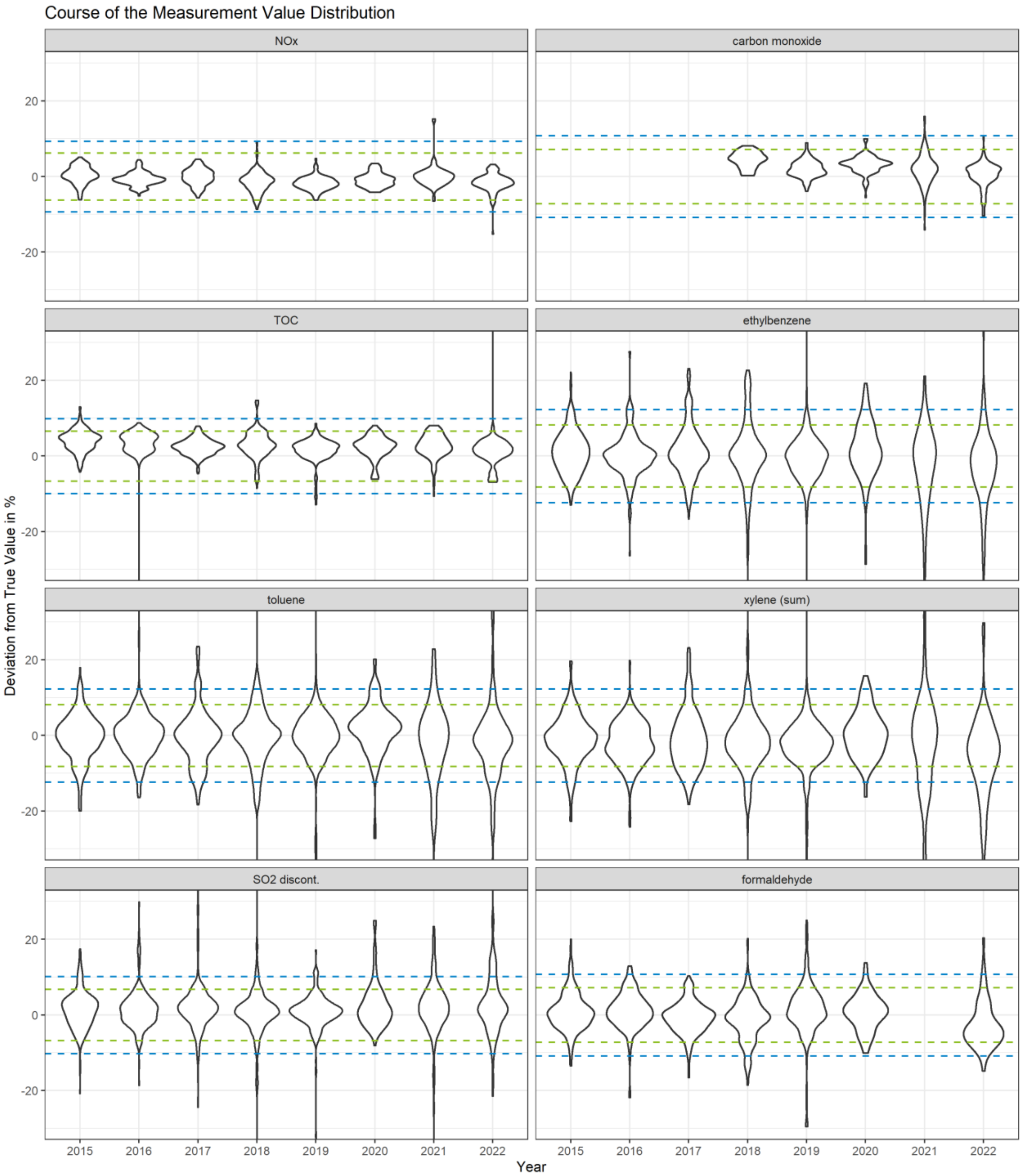


Scheme 6: Course of the measurement value distribution in the dust proficiency tests 2015-2022 (all participants)

The dust used in the proficiency tests cannot be the cause for the deterioration of the measurement results. In 2020, 2021 and 2022, the same two dusts were used for the majority of the proficiency tests. For dust A, in 2020 a total of 75% of all measured values for this dust (88 out of 117) were at less than 14% deviation from the nominal value, 16% were at 14-21% deviation and 9% at higher deviations. For the same dust, in 2021 only 51% of all measured values (40 out of 78) were less than 14% off target, 26% were 14-21% off target and 23% were even higher off target. In 2022, on the other hand, 67% of all measured values (42 out of 63) for the same dust had deviations of less than 14%, 16% had deviations between 14 and 21% and 17% of all measured values had even higher deviations. A similar picture can be seen for dust B, for which in 2021 only 54% of all measured values (35 out of 65) were at deviations of less than 14% from the target value, 28% were at deviations of 14-21% and 18% were at even higher deviations. In 2022, however, 77% of all measured values (30 of 39) for the same dust were less than 14% of the nominal value, 21% were in the range 14-21% and only 3% of all measured values had a deviation of more than 21% from the nominal value.

In the gas proficiency test, a similar development could be observed in recent years, but here limited to the discontinuous components and among these especially for the organic substances ethylbenzene, toluene and xylene. While the results for the continuous components NO_x, carbon monoxide and TOC were consistently very good (since 2015, 90-100% of all measured values have generally been in a range that corresponds to a z score < 2 according to current assessment criteria), the measurement results for the individual organic substances, similar to dust, deviated significantly from the values of the previous years in 2021 and 2022. The measurement results for the components ethylbenzene, toluene and xylene in the years 2015 to 2020 were on average 82% with a deviation of less than 8.2% from the target value (which corresponds to a z-score of ≤ 2 according to the current specifications), 11% of the measurement values were with a deviation of 8.2-12.3% (z-score 2-3), and 7% of all measurement values had a deviation of more than 12.3% from the target value (z score > 3). In 2021, however, only 62% of all measured values for these three components were at a deviation of less than 8.2%, 16% had a deviation of 8.2-12.3% and a full 22% of the measured values deviated by more than 12.3% from the target value. The readings for 2022 were only marginally better, with 68% of all readings deviating by up to 8.2% from the target value, 16% showing deviations of 8.2-12.3% and another 16% more than 12.3%. There are only slight differences between the three individual substances, but overall the measurement results for the component xylene, which must be determined as the sum of the isomers *o*-xylene, *m*-xylene and *p*-xylene, show somewhat greater deviations from the nominal value on average than the measurement results for the components ethylbenzene and toluene.

The following figure shows a graphical representation of the distribution of the measured values in the gas proficiency tests of the past years in the form of a "violin plot". The wider the shape shown, the more measurement results are in the relevant range.



Scheme 7: Course of the measurement value distribution in the gas proficiency tests 2015-2022 (all participants)

In 2022, a total of 10 out of 13 authorized measuring bodies (77%) passed the odour proficiency test. The results of the odour proficiency test were thus better than in the years 2019 to 2021. From a statistical point of view the number of test persons, which is usually 4, is in view of the uncertainty of individual test person results clearly too low, and is probably still the main cause of inadequate results in the odour proficiency test.

6.2 Voluntary Participants

The number of voluntary participations in the proficiency test varies from year to year; as a rule, there are about 8 participations in the dust proficiency test, 6 participations in the gas proficiency test and about 4 participations in the odour proficiency test. In 2022, there were 6 voluntary participations in each of the dust proficiency test, the gas proficiency test and the odour proficiency test. Due to the usually low number of voluntary participations in many years, the collected results of a year are extremely influenced by the performance of individual laboratories and thus a long-term comparison is only informative to a limited extent. It should also be noted that a participation of an authorized §29b monitoring site is counted as "voluntary" if this participation is not a "compulsory participation" but goes beyond the minimum required according to the 41st BImSchV at the monitoring bodies own request. Voluntary participation by §29b monitoring bodies usually leads to above-average results compared to other voluntary participants.

For the dust proficiency test in 2022, none of the 3 voluntary participations assessed was successful, for the gas proficiency test, this was the case for 1 of 4 (25%) voluntary participations. There were also 3 voluntary participations in the pandemic dust proficiency test and 4 voluntary participations in the pandemic gas proficiency test. If these participations had also been assessed, a total of one in 6 (17%) voluntary participants would have passed the dust proficiency test. Two other participants (33%) would have received the rating "failed (incomplete participation)" because they did not perform measurements for all required components. The gas proficiency test would have been passed by a total of 2 of the 6 (33%) voluntary participants if the pandemic proficiency tests had been fully assessed.

In the odour proficiency tests, 2 of 6 voluntary participants (33%) passed. One participant (17%) delivered correct measured values, but these were not determined within 6 h in accordance with guideline VDI 3880. The results were therefore classified as "failed (incomplete participation)".

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, measurements of the volume flow should actually have been carried out as an assessed component of the dust and gas proficiency tests since the middle of 2020. However, due to the SARS-CoV-2 pandemic and the resulting change in the proficiency test programme, this could not be implemented initially. In the pandemic version of the dust and gas proficiency tests, the gas flow conditions were only measured and evaluated before the dust measurements began. For the gas proficiency tests following the dust proficiency test in the afternoon, the gas flow conditions were kept constant and no new measurement was carried out by the participants. Instead of the planned 2 measured values, only one measured value per participant was determined. The gas flow conditions were not evaluated in the pandemic version of the proficiency tests due to the deviations from the LAI specifications. However, according to the assessment criteria specified there, all participants in the proficiency test (both §29b monitoring bodies and voluntary participants) would have passed this part of the proficiency test if they had been evaluated.

Since the resumption of the regular proficiency tests in November 2022, the gas flow conditions have been measured and assessed as provided for in the specifications of 2019. In 2022, values are thus available to the extent intended (two measurements on two different days under different conditions) for a total of 15 participations.

The values recorded in 2022 (see section 5.1.4) correspond to the observations of previous years: The measured values for temperature (CGT), volumetric flow (CVF) and flow velocity (CFV) show minimal deviations from the target values. For flue gas humidity (CAH) and especially for static pressure (CSP), there are overall larger deviations from the target 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 2022.

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 (or 3 in the pandemic version, respectively) 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 2022 annual report has only increased by approx. 10-15%, 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.

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.

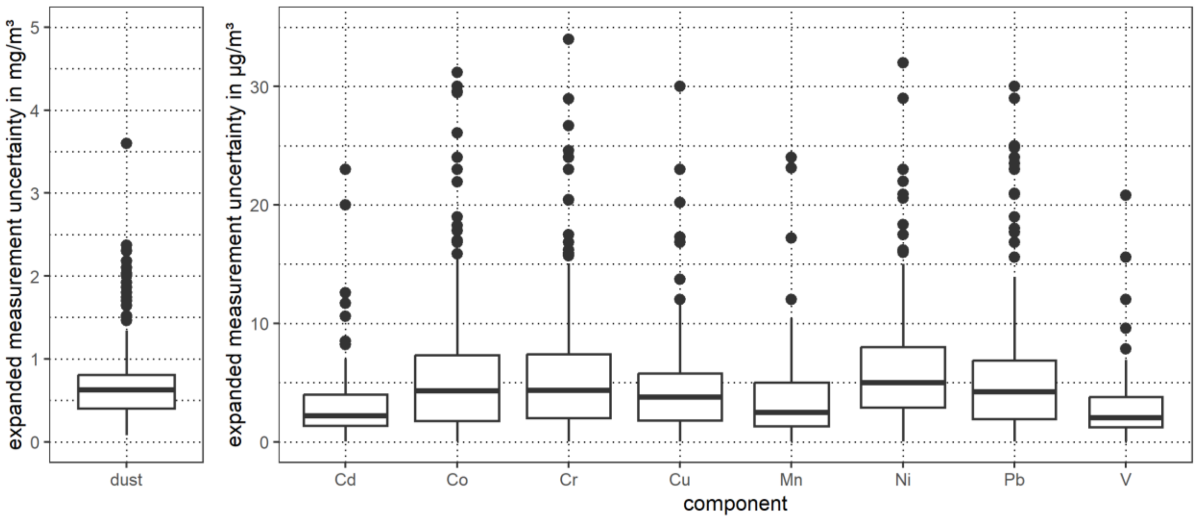


Table 8: Expanded measurement uncertainties reported by participants of the dust proficiency test

	dust [mg/m³]	**Cd** [µg/m³]	**Co** [µg/m³]	**Cr** [µg/m³]	**Cu** [µg/m³]	**Mn** [µg/m³]	**Ni** [µg/m³]	**Pb** [µg/m³]	**V** [µg/m³]
75th percentile	0.85	4.00	7.45	7.59	5.79	5.00	8.00	6.90	3.80
median	0.64	2.21	4.34	4.43	3.79	2.48	5.00	4.23	2.04
25th percentile	0.40	1.36	1.75	2.00	1.80	1.32	2.91	1.92	1.24
number of values	203	183	184	184	184	146	184	183	142

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.

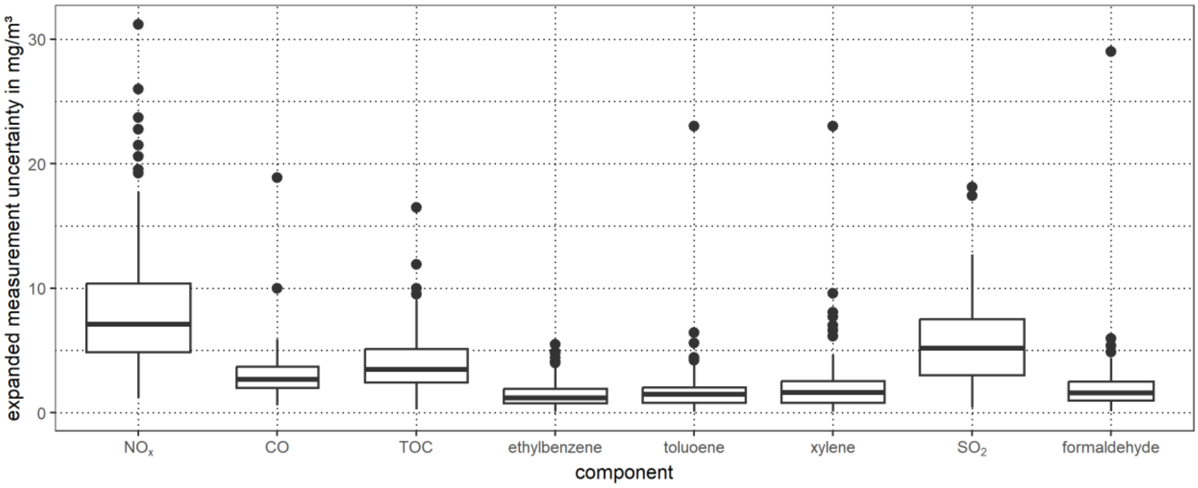


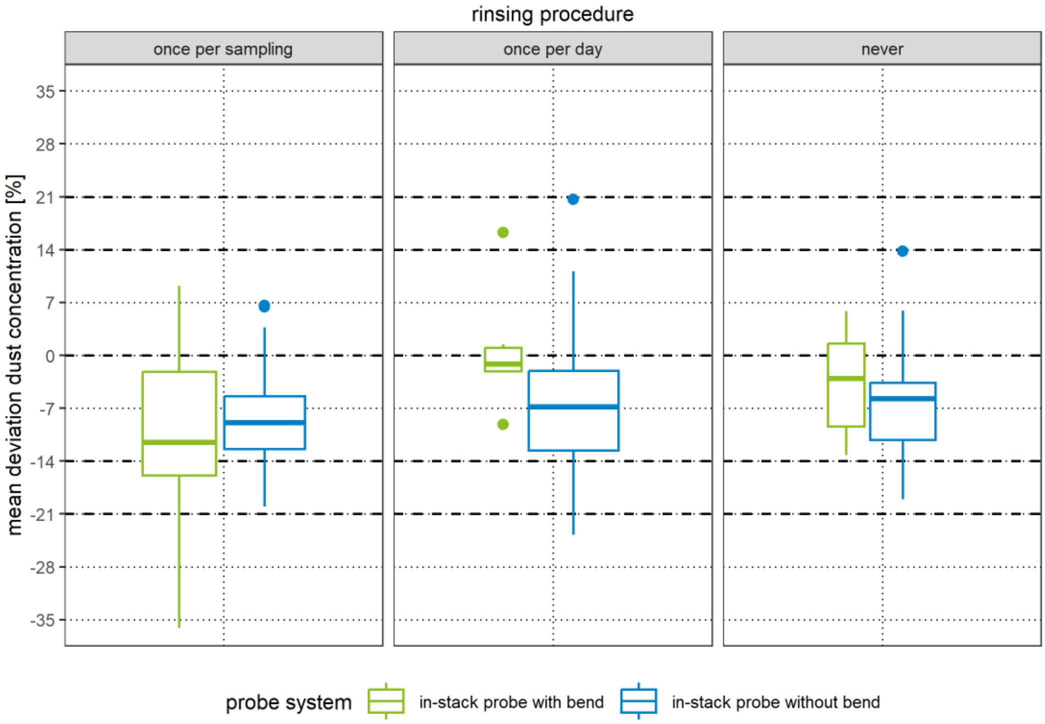
Table 9: Expanded measurement uncertainties reported by participants of the gas proficiency test

	NO_x as NO_2 [mg/m³]	CO [mg/m³]	TOC [mg/m³]	ethyl-benzene [mg/m³]	toluene [mg/m³]	sum of xylenes [mg/m³]	SO_2 [mg/m³]	form-aldehyde [mg/m³]
75th percentile	10.40	3.72	5.11	1.93	2.05	2.56	7.51	2.50
median	7.10	2.69	3.50	1.20	1.50	1.64	5.20	1.60
25th percentile	4.87	2.00	2.43	0.75	0.80	0.80	3.00	0.99
number of values	197	121	192	192	193	193	197	131

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 119 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.


rinsing procedure
once per sampling
once per day
never
mean deviation dust concentration [%]
35
28
21
14
7
0
-7
-14
-21
-28
-35
probe system
in-stack probe with bend
in-stack probe without bend

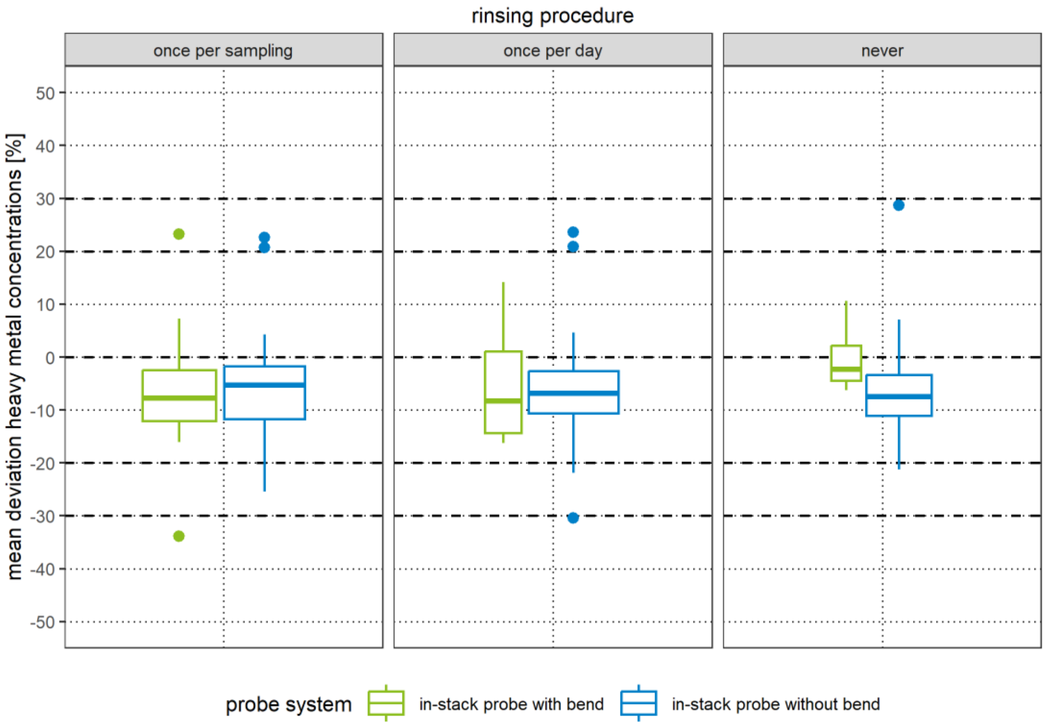

rinsing procedure
once per sampling
once per day
never
mean deviation heavy metal concentrations [%]
50
40
30
20
10
0
-10
-20
-30
-40
-50
probe system
in-stack probe with bend
in-stack probe without bend

Table 10: Correlation of dust measurement results with probe systems and rinsing procedures (2018-2022)

combi-nation	probe system	rinsing procedure	median of mean devia-tions total dust results	number of participants	median of mean devia-tions heavy metal results	number of participants
1 **(left)**	in-stack probe **with** bend(s)	after each sampling	-11.5%	24	-7.7%	24
2 **(centre)**		once per day	-1.1%*	6*	-8.3%	6*
3 **(right)**		no rinsing	-3.1%*	6*	-2.3%	4*
4 **(left)**	in-stack probe **without** bend	after each sampling	-8.9%	29	-5.3%	29
5 **(centre)**		once per day	-6.9%	35	-7.0%	35
6 **(right)**		no rinsing	-5.7%	19	-7.4%	19

*This combination was only indicated by approx. 3-5% of the participants. The median is clearly less meaningful here than for the other combinations.

Due to the relatively small number of cases, the results shown are significantly influenced by various influences of the respective laboratories. For example, the above-average results for combination 3 (probe with elbow that is not flushed: 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 flushing 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 flushing is carried out (combination 4, 5 and 6). At the same time, however, the measurement results for heavy metals are slightly better with this probe type when flushing after each measurement (combination no. 4) than when flushing daily (combination no. 5) or not flushing at all (combination no. 6).

The probe with bend (combination no. 1) performs significantly worse than the probe without bend (combination no. 4) for both dust and heavy metals when flushing 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 2022 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 the 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 5% of all participants are not listed here.

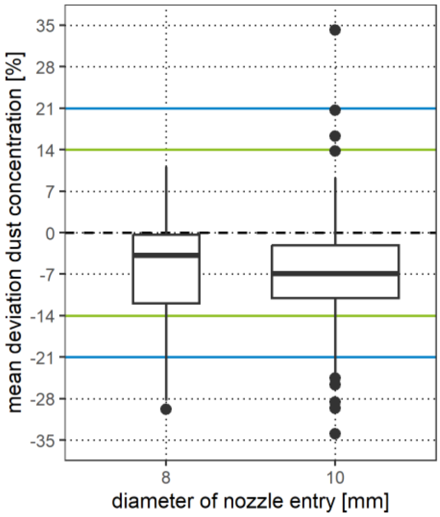


Table 11: Correlation of absolute means of z-scores for total dust with nozzle opening diameters (2016-2022)

diameter of nozzle opening	8 mm	10 mm
75th percentile	−0.4%	−2.1%
median	−3.8%	−6.9%
25th percentile	−11.9%	−11.0%
number of values	48	174

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 32 participants stated that heavy metal analysis was performed using AAS equipment, while 206 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 significantly more than those of the AAS users.

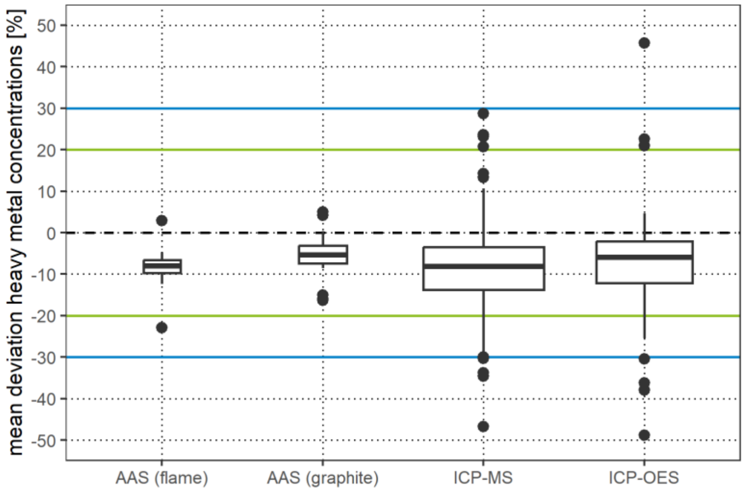


Table 12: Correlation of heavy metal results and analysis devices (2016-2022)

analysis device	flame-AAS	graphite furnace AAS	ICP-MS	ICP-OES
75th percentile	−6.6%	−3.1%	−3.5%	−2.1%
median	−8.0%	−5.3%	−8.1%	−5.9%
25th percentile	−9.7%	−7.4%	−13.8%	−12.1%
number of values	12	20	126	80

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 (CS_2). The majority of the participants reported that they had worked with CS_2. The average results of all participants were close to the target value.

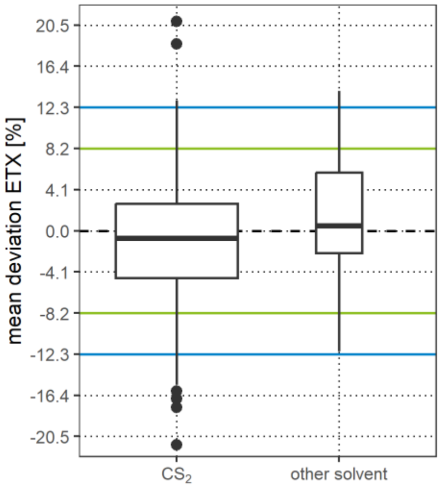


Table 13: Correlation of ETX measurement results with the desorption solvent (2016-2022)

solvent used in desorption	CS_2	other solvent
75th percentile	2.7%	5.8%
median	–0.7%	0.5%
25th percentile	–4.7%	–2.2%
number of values	203	29

7.6 Gas Chromatography Detectors

Gas chromatographs with either an FID detector or a mass spectrometer are usually used for the analysis of ETX samples.

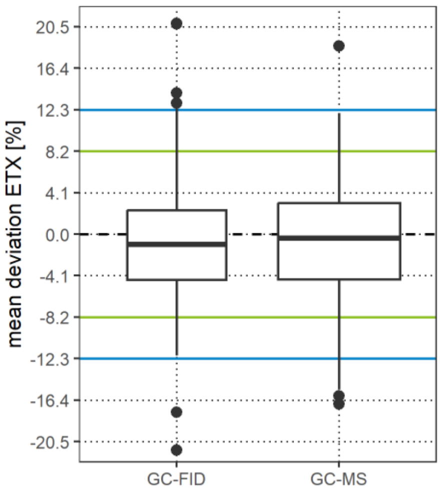


Table 14: Correlation of ETX measurement results with analytical instruments (2016-2022)

analytical instrument	GC-FID	GC-MS
75th percentile	2.4%	3.1%
median	–1.0%	–0.4%
25th percentile	–4.5%	–4.5%
number of values	92	140

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:

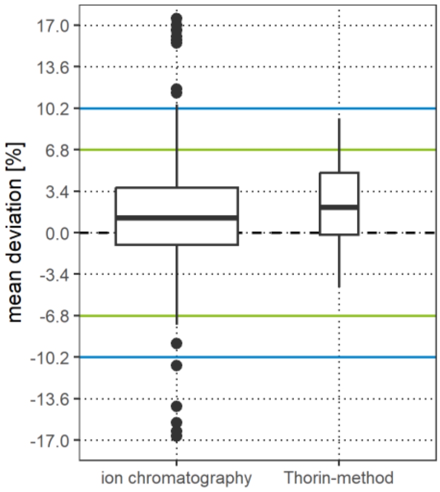


Table 15: Correlation of sulphur dioxide measurement results with the analytical method used (2016-2019)

method	ion chromatography	Thorin-method
75th percentile	3.7%	4.9%
median	1.2%	2.1%
25th percentile	–1.0%	–0.2%
number of values	223	22

The available results show a slightly smaller mean deviation for the ionic chromatography, 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 about 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:

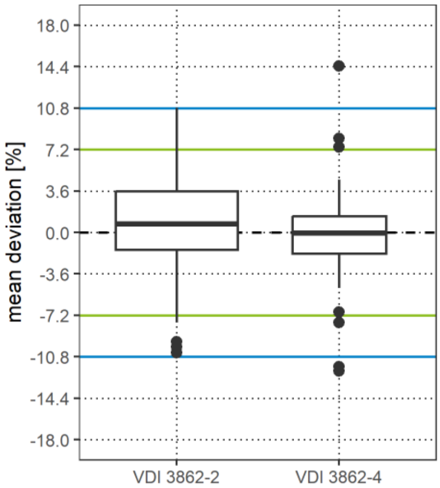


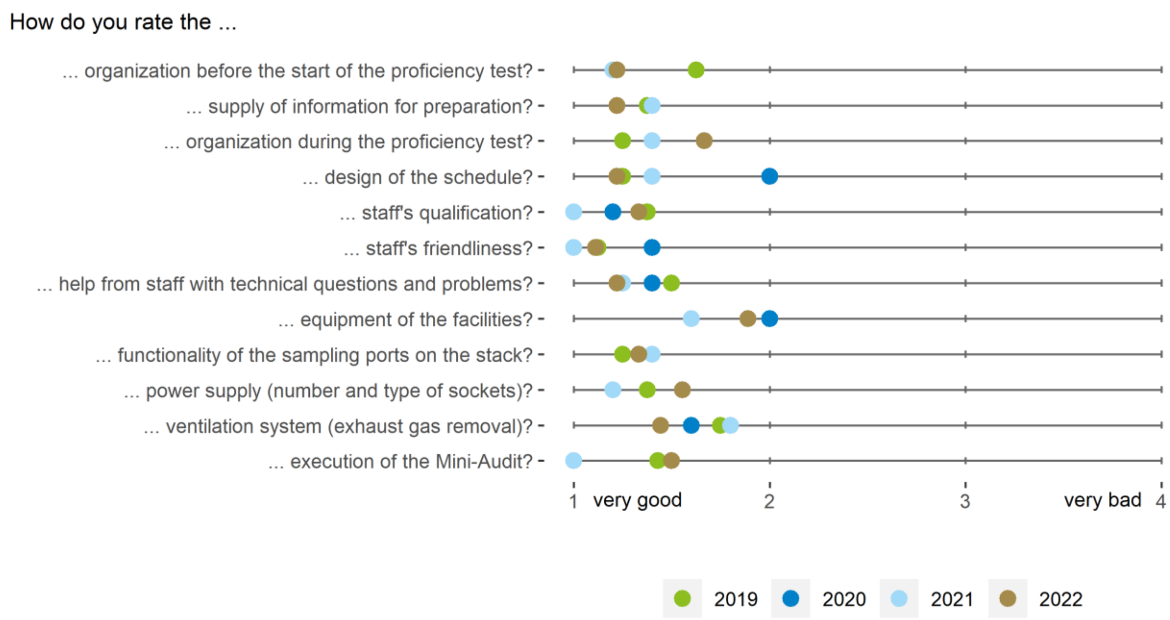
Table 16: Correlation of formaldehyde measurement results with the guidelines used

guideline (method)	VDI 3862 Part 2 (DNPH wash bottles)	VDI 3862 Part 4 (AHMT-procedure)
75th percentile	3.6%	1.4%
median	0.7%	0.0%
25th percentile	–1.5%	–1.8%
number of values	97	57

The DNPH wash bottle procedure apparently delivers on average comparable good values as the AHMT procedure, but spreads over a larger area.

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.



Unfortunately, last year there were only 9 responses from 6 different laboratories. However, these few feedbacks received also showed a high overall satisfaction of the participants with the proficiency testing scheme in 2022. The participants were particularly satisfied with the friendliness of the staff (average grade: 1.1). The worst ratings in comparison were given to the equipment of the facilities (average grade: 1.9). One participant rated the power supply, the equipment of the facilities and the organisation during the proficiency test with the worst possible grade 4 (very poor), however, without explaining this rating in more detail. Another participant criticised the condition of the olfactory room provided for olfactometry with regard to temperature and found it untidy. A third participant, on the other hand, was very positive about the space available in the measurement room as part of the "pandemic version".

Additional components suggested were hydrogen fluoride (HF) and hydrogen chloride (HCl) for the gas proficiency test, and the elements antimony, arsenic and tin for the dust proficiency test. If this wish should arise more frequently in the future, an implementation at ESA can be examined.

8. Concluding Remark

Due to the ongoing SARS-CoV-2 pandemic, the dust and gas proficiency tests in spring 2022 could again only be offered in the shortened "pandemic version". However, the odour proficiency tests in autumn, as well as further dust proficiency tests in November, could be carried out in full with the implementation of appropriate infection control measures. With a delay of two years, it was thus finally possible to fully implement the changes to the LAI specifications from May 2019. These include minor changes to the assessment criteria, the component spectrum and the timetable, but also the introduction of a theory test.

The measurement results in the dust and gas proficiency tests have improved significantly in 2022 compared to 2021 in some cases, but are still significantly worse for many components than in the years before the pandemic. In 2021, these proficiency tests took place exclusively in the pandemic version; in 2022, this was still the case for about three-quarters of all participations. The reduction in the number of measurements for the pandemic version of the proficiency tests from 9 to 3 should have no influence on the average deviation of the submitted measured values from the target values. Whether the higher dispersion of the measured values is related to the fact that the participants in the pandemic version basically had no contact with other participants cannot be verified. The meanwhile noticeable improvement of the measurement results also shows that the observed deviations are not fundamentally related to the way the proficiency test was conducted.

In the odour proficiency test, the participants achieved significantly better results overall in 2022 than in 2019 to 2021, and the pass rate here was again at the level of 2016 to 2018. As before, the main problem for participants in the odour proficiency test is probably the use of panels consisting of only 4 test persons. Under these circumstances, the measurement results of individual test persons have a massive influence on the sample result, whereby day-dependent fluctuations in perception of these individuals can easily lead to the failure of the laboratory's entire participation.

Unless something unexpected happens, the dust and gas proficiency tests in 2023 will again take place entirely in the "standard" version in accordance with the LAI specifications of 2019; the pandemic version will then no longer be offered by HLNUG. However, the knowledge gained in the pandemic version will certainly be incorporated into the next revision of the specifications, e.g. when discussing whether the number of measurements must necessarily be 9 or could also be 6.

Kassel, 9th February 2023

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