

Annual Report 2021

Results of stack emission proficiency tests for substance ranges P, G, and O on the emission simulation apparatus in the year 2021

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Contents

0.	About this Report	4
1.	Summary	4
2.	Introduction	4
2.1	Legal Background.....	4
2.2	The Emission Simulation Apparatus.....	4
3.	Organisational Information	6
4.	Execution of the Proficiency Tests	8
4.1	Description of the Test Objects	8
4.2	Preparation of the Test Objects.....	9
4.3	Metrological Traceability	9
4.4	Execution of the Measurements	10
4.5	Evaluation of the Proficiency Tests	10
4.5.1	Calculation of z-Scores.....	10
4.5.2	Criteria for Proficiency Assessment.....	12
4.5.3	Assessment Scheme.....	13
4.5.4	Communication of the Assessment Result.....	14
5.	Results	14
5.1	z-Scores	14
5.1.1	Dust (Substance Range P)	15
5.1.2	Gas (Substance Range G)	19
5.1.3	Odour (Substance Range O)	23
5.1.4	Gas flow conditions.....	25
5.2	Sums of Class Numbers	27
5.2.1	Dust (Substance Range P)	28
5.2.2	Gas (Substance range G)	29
5.2.3	Odour (Substance Range O)	30
6.	Interpretation of Results	31
6.1	§29b Measuring Bodies.....	32
6.2	Voluntary Participants	33
6.3	Basic Flow Conditions.....	34
7.	Optional Information from Participants	34
7.1	Measurement Uncertainties	35
7.2	Probes and Rinsing Procedures in Dust Sampling	36

7.3	Diameter of the Nozzle Opening in Dust Samplings	38
7.4	Analytical Instruments for Heavy Metals.....	39
7.5	Formaldehyde.....	40
7.6	Sulphur Dioxide.....	40
7.7	Solvents for Desorption of ETX.....	41
7.8	Gas Chromatography Detectors.....	42
7.9	Feedback from Participants	43
8.	Concluding Remark	44
9.	References	46

0. About this Report

This report is a translation of „Jahresbericht 2021 – Ergebnisse der Emissionsringversuche der Stoffbereiche P, G und O an der Emissionssimulationsanlage im Jahr 2021“ 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 2021, a total of 48 measuring institutes took part in both the dust stack emission proficiency tests (substance range P) and the gas stack emission proficiency tests (substance range G) of the HLNUG, of which 42 participants were §29b measuring bodies and 6 volunteers in both types of proficiency tests. All dust and gas proficiency tests in 2021 were carried out in the "pandemic version", an overall evaluation of the participant results did not take place.

A total of 19 measuring institutes took part in the odour stack emission proficiency tests (substance range O), of which 15 participated on the basis of an authorization in accordance with §29b BImSchG and 4 voluntarily. Here, 60% of the notified and 50% 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

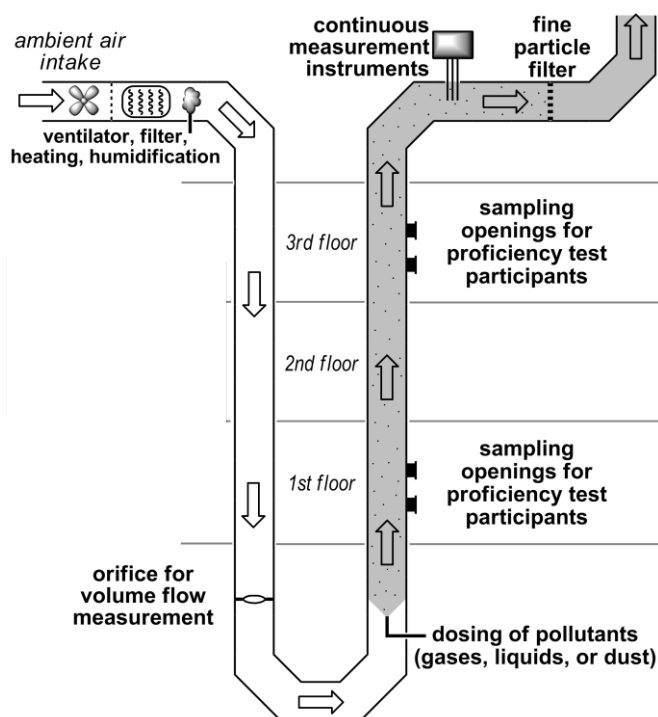
The prerequisite for carrying out stack emission proficiency tests is the ability to provide all participants at the same time with a stable and clearly defined simulated exhaust gas. For this purpose, HLNUG operates the Emission Simulation Apparatus (ESA, see scheme 1). It was designed as a model for 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 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 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 2021, the following proficiency tests of the substance ranges P, G, and O were carried out:

Table 1: Proficiency Tests organised by HLNUG in 2021

proficiency test	substance range	date	participants
21G11	Gas (substance range G) - pandemic version	02.02.2021	2
21P11	Dust (substance range P) - pandemic version	02.02.2021	2
21G12	Gas (substance range G) - pandemic version	03.02.2021	1
21P12	Dust (substance range P) - pandemic version	03.02.2021	1
21G13	Gas (substance range G) - pandemic version	04.02.2021	2
21P13	Dust (substance range P) - pandemic version	04.02.2021	2
21G14	Gas (substance range G) - pandemic version	05.02.2021	2
21P14	Dust (substance range P) - pandemic version	05.02.2021	2
21G21	Gas (substance range G) - pandemic version	16.02.2021	2
21P21	Dust (substance range P) - pandemic version	16.02.2021	2
21G22	Gas (substance range G) - pandemic version	17.02.2021	2
21P22	Dust (substance range P) - pandemic version	17.02.2021	2
21G23	Gas (substance range G) - pandemic version	18.02.2021	2
21P23	Dust (substance range P) - pandemic version	18.02.2021	2
21G24	Gas (substance range G) - pandemic version	19.02.2021	2
21P24	Dust (substance range P) - pandemic version	19.02.2021	2
21G31	Gas (substance range G) - pandemic version	02.03.2021	2
21P31	Dust (substance range P) - pandemic version	02.03.2021	2
21G32	Gas (substance range G) - pandemic version	03.03.2021	2
21P32	Dust (substance range P) - pandemic version	03.03.2021	2
21G33	Gas (substance range G) - pandemic version	04.03.2021	1
21P33	Dust (substance range P) - pandemic version	04.03.2021	1
21G34	Gas (substance range G) - pandemic version	05.03.2021	2
21P34	Dust (substance range P) - pandemic version	05.03.2021	2
21G41	Gas (substance range G) - pandemic version	16.03.2021	2
21P41	Dust (substance range P) - pandemic version	16.03.2021	2
21G42	Gas (substance range G) - pandemic version	17.03.2021	2
21P42	Dust (substance range P) - pandemic version	17.03.2021	2
21G43	Gas (substance range G) - pandemic version	18.03.2021	2
21P43	Dust (substance range P) - pandemic version	18.03.2021	2
21G44	Gas (substance range G) - pandemic version	19.03.2021	2
21P44	Dust (substance range P) - pandemic version	19.03.2021	2
21G51	Gas (substance range G) - pandemic version	20.04.2021	2
21P51	Dust (substance range P) - pandemic version	20.04.2021	2
21G52	Gas (substance range G) - pandemic version	21.04.2021	2
21P52	Dust (substance range P) - pandemic version	21.04.2021	2
21G53	Gas (substance range G) - pandemic version	22.04.2021	2
21P53	Dust (substance range P) - pandemic version	22.04.2021	2
21G54	Gas (substance range G) - pandemic version	23.04.2021	2
21P54	Dust (substance range P) - pandemic version	23.04.2021	2

proficiency test	substance range	date	participants
2101	Odour (substance range O)	21.09.2021	5
2102	Odour (substance range O)	23.09.2021	4
2103	Odour (substance range O)	28.09.2021	5
2104	Odour (substance range O)	30.09.2021	5
21G61	Gas (substance range G) - pandemic version	09.11.2021	2
21P61	Dust (substance range P) - pandemic version	09.11.2021	2
21P63	Dust (substance range P) - pandemic version	11.11.2021	1
21G64	Gas (substance range G) - pandemic version	12.11.2021	2
21P64	Dust (substance range P) - pandemic version	12.11.2021	2
21G71	Gas (substance range G) - pandemic version	23.11.2021	2
21P71	Dust (substance range P) - pandemic version	23.11.2021	2
21G72	Gas (substance range G) - pandemic version	24.11.2021	2
21P72	Dust (substance range P) - pandemic version	24.11.2021	1
21G73	Gas (substance range G) - pandemic version	25.11.2021	1
21P73	Dust (substance range P) - pandemic version	25.11.2021	1
21G74	Gas (substance range G) - pandemic version	26.11.2021	1
21P74	Dust (substance range P) - pandemic version	26.11.2021	1

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 10 including 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	SO ₂ : 20 ... 150 mg/m ³ NO _x as NO ₂ : 60 ... 450 mg/m ³ CO: 10 ... 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 TOC: 4 ... 100 mg/m ³
result submission	within six weeks after the end of the proficiency test, in mg/m ³ for dust concentrations and µg/m ³ for heavy metal concentrations respectively, relating to standard conditions (dry) and with two digits after decimal point.	within six 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 0)	
duration of each sampling	10 min
number of samplings	for each component 3
sampling	simultaneously for all participants (1 st and 3 rd 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 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, 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 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 5. 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	compulsory measurement procedure
P	dust	EN 13284-1 (10)
	heavy metals	EN 14385 (8)
G	SO ₂	EN 14791 (11)
	TOC	EN 12619 (12)
	ETX	CEN/TS 13649 (13)
	NO _x as NO ₂	EN 14792 (14)
	CO	EN 15058 (15)
O	4 odours	EN 13725 (16)

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_{ik} , which is the result of measurement i of component k , a z-score value z_{ik} is determined:

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

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 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:

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

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 2021, 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 186 measurements in the years 2016 to 2020, resulting in a consensus value of $c_0 = 224 \mu\text{g}/\text{m}^3$. For the components THT and PIG consensus values had to be determined via procedure b). For THT, a value of $c_0 = 0.503 \mu\text{g}/\text{m}^3$ and for PIG a value of $c_0 = 160 \mu\text{g}/\text{m}^3$ was calculated, both on the basis of 45 measurements in 2021.

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 2021 this was only necessary for

component PIG, where σ_k had to be raised to a value of 0.22. 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	1.56
P2	Cadmium	discontinuous	Cd	10.0	1.73
P3	Cobalt	discontinuous	Co	10.0	1.85
P4	Chromium	discontinuous	Cr	10.0	1.83
P5	Copper	discontinuous	Cu	10.0	2.16
P6	Manganese	discontinuous	Mn	10.0	1.97
P7	Nickel	discontinuous	Ni	10.0	1.90
P8	Lead	discontinuous	Pb	10.0	1.82
P9	Vanadium	discontinuous	V	10.0	2.12
substance range G					
G1	SO ₂	discontinuous	Sd	3.4	1.11
G2	NO _x as NO ₂	continuous	Nk	3.1	1.03
G3	CO	continuous	Kk	3.6	1.08
G4	(NA)				
G5	ethylbenzene	discontinuous	Ed	4.1	1.01
G6	toluene	discontinuous	Td	4.1	1.01
G7	sum of <i>o</i> -, <i>m</i> -, <i>p</i> -xylene	discontinuous	Xd	4.1	1.01
G8	TOC	continuous	Ck	3.3	1.08
substance range O					
O1	<i>n</i> -butanol	discontinuous	NBU	0.10 †	1.01
O2	solvent mixture	discontinuous	ETX	0.10 †	5.82
O3	tetrahydrothiophene	discontinuous	THT	0.10 †	5.75
O4	artificial pigsty odour	discontinuous	PIG	0.22 †	16.0

† 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 z-scores

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

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

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

The assessment of the individual component proceeds differently for the three substance ranges.

Substance Ranges P and G (Pandemic Version)

For the components in the dust and gas proficiency tests in the pandemic version, class numbers are assigned to the amounts of the z-scores of the results of the individual measurements according to the following scheme::

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

A component has been successfully determined if the associated sum of the class numbers is ≤ 6 . In this case, the participant receives the rating "passed" for the component. If the class sum exceeds the permissible value, the participant receives the rating "failed".

If only measured values for two measurements are submitted, the component is considered to be "passed" if the sum of the class numbers is a maximum of 4. If only one measurement value is submitted, the component is considered "passed" if the class number achieved with it is ≤ 2 . If sampling or measurements have been carried out for a component, but no measured value has been submitted, the component is assessed as "failed". If the participant has completely refrained from carrying out sampling or measurements for a component in the proficiency test, it will be marked "no participation".

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”.

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

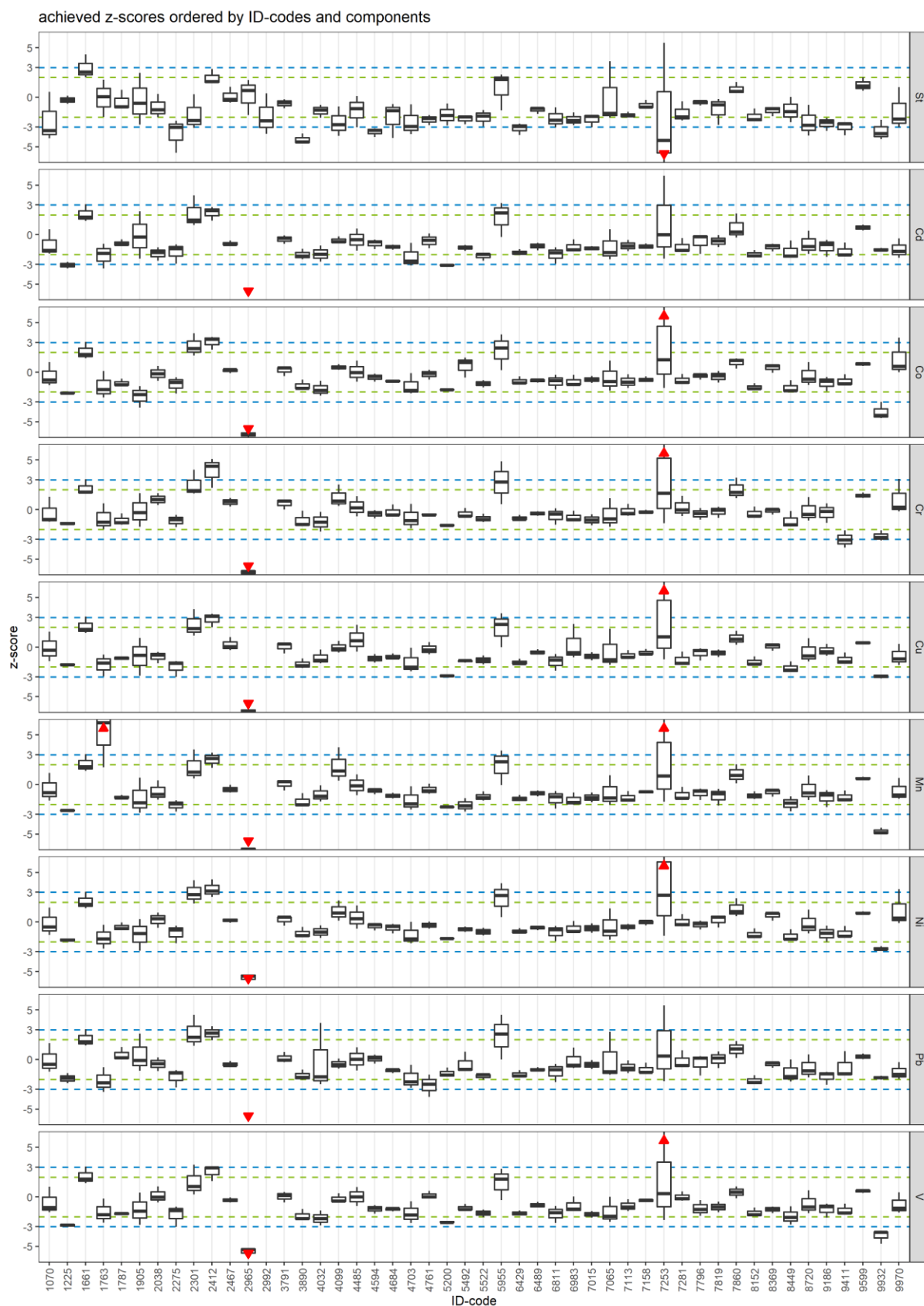
5.1 z-Scores

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.

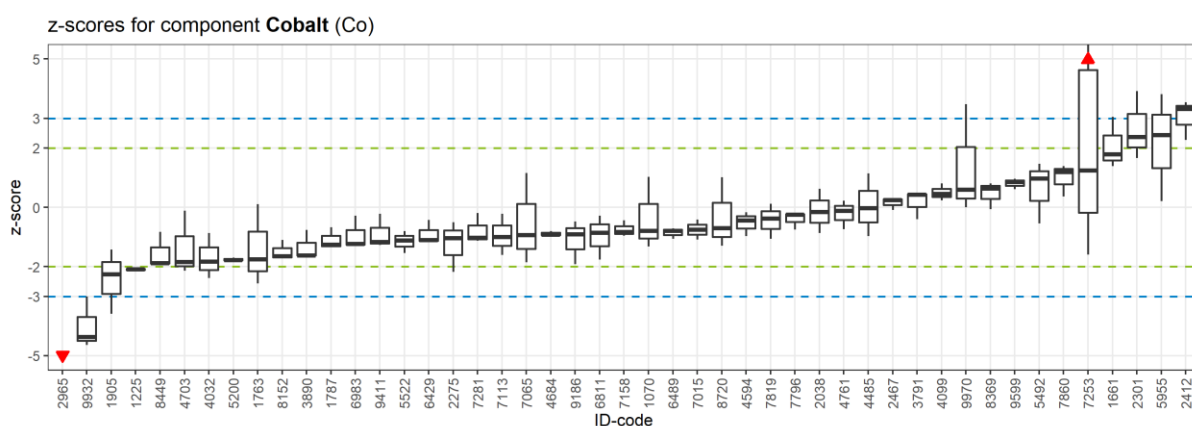
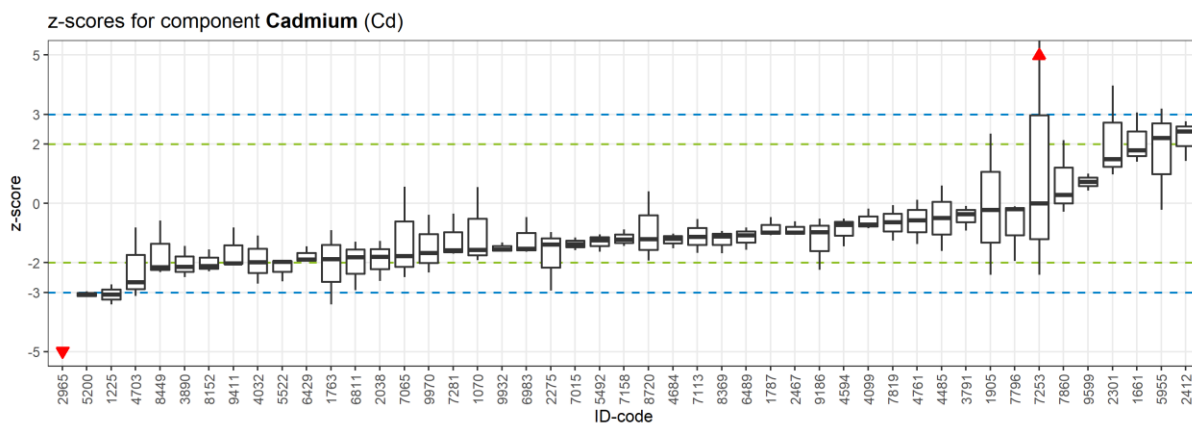
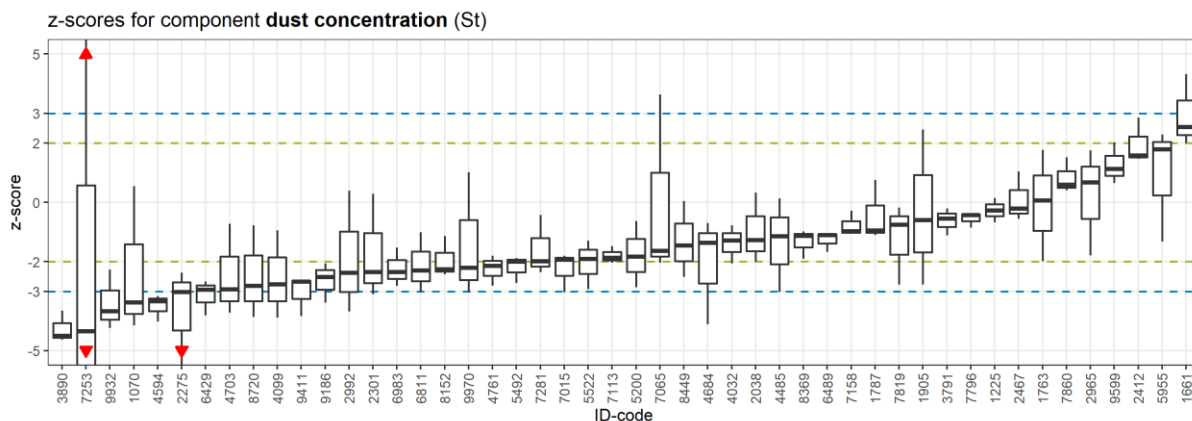
In order to be able to assess the performance of individual participants across all components and to get an impression of the quality of measurements for individual components, the diagrams are available in two different sorts; on the one hand as an overview on one page, on the other hand sorted according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

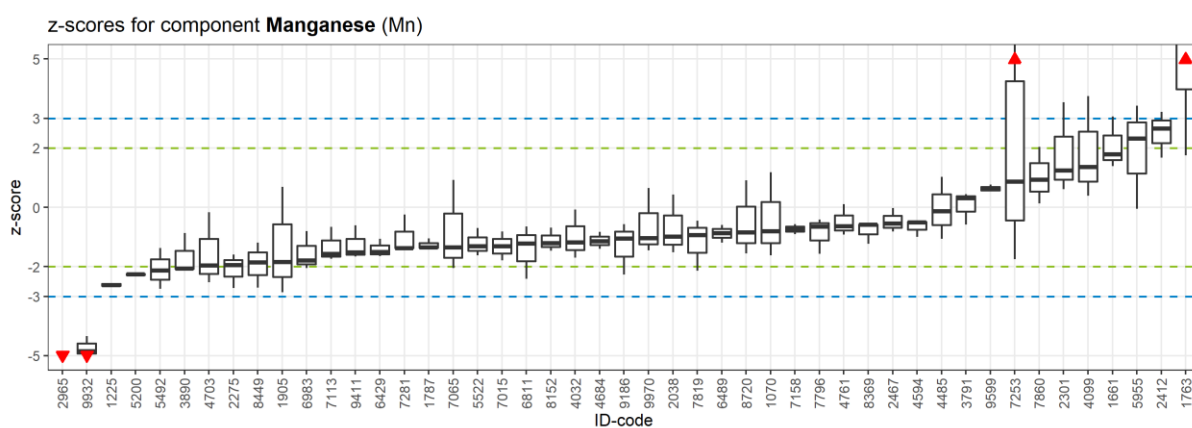
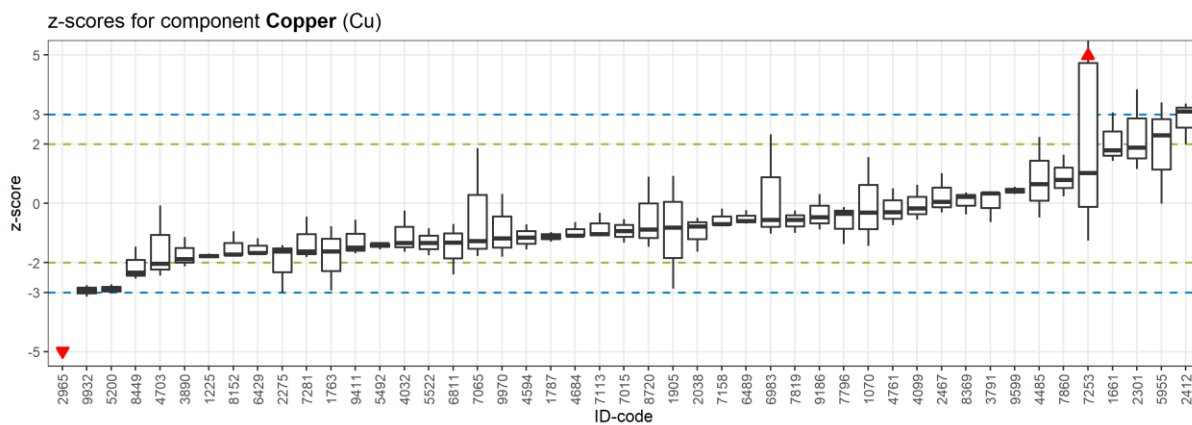
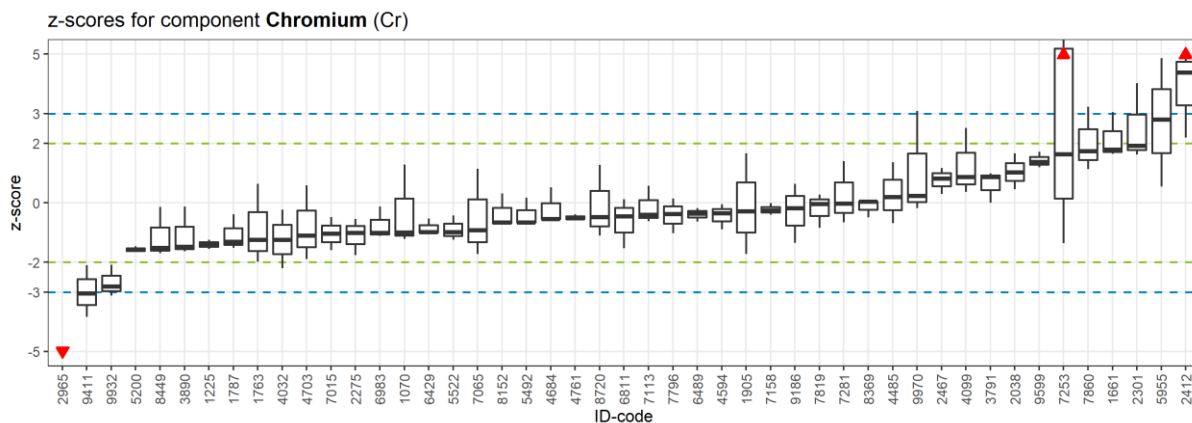
A list of the individual measurements of all participants can be found in a separate document (appendix to the annual report).

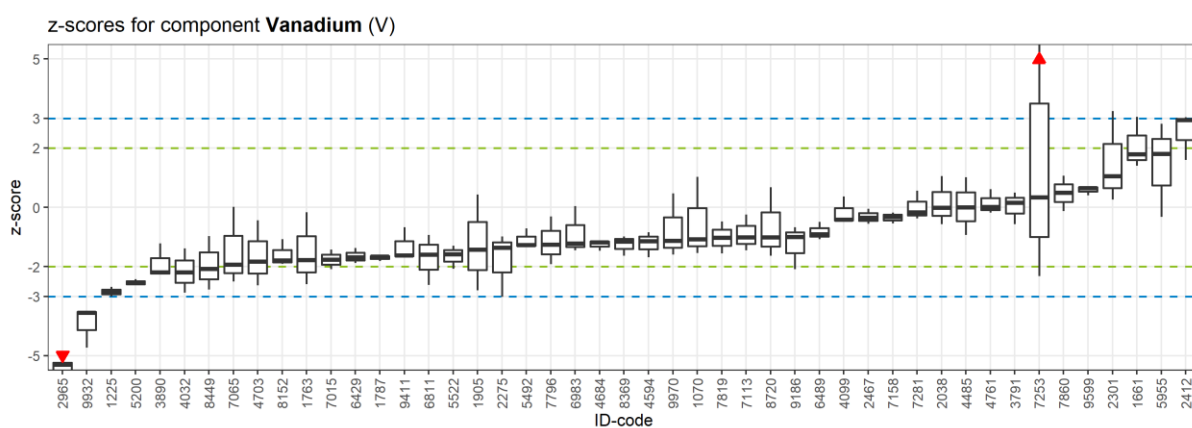
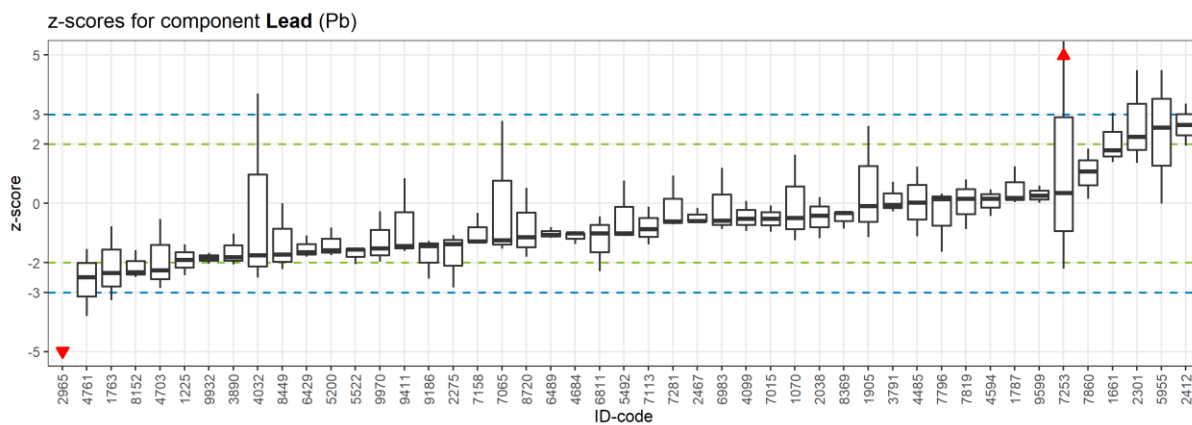
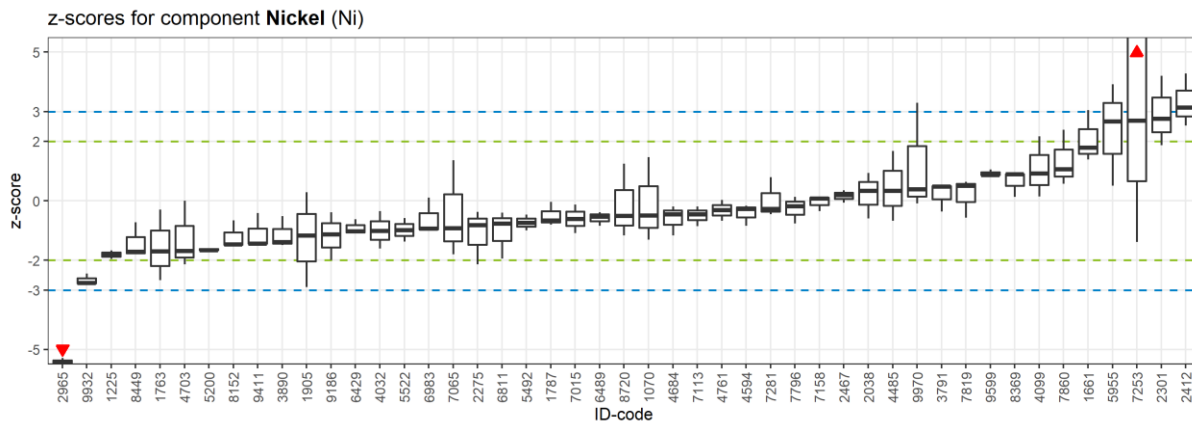
5.1.1 Dust (Substance Range P)



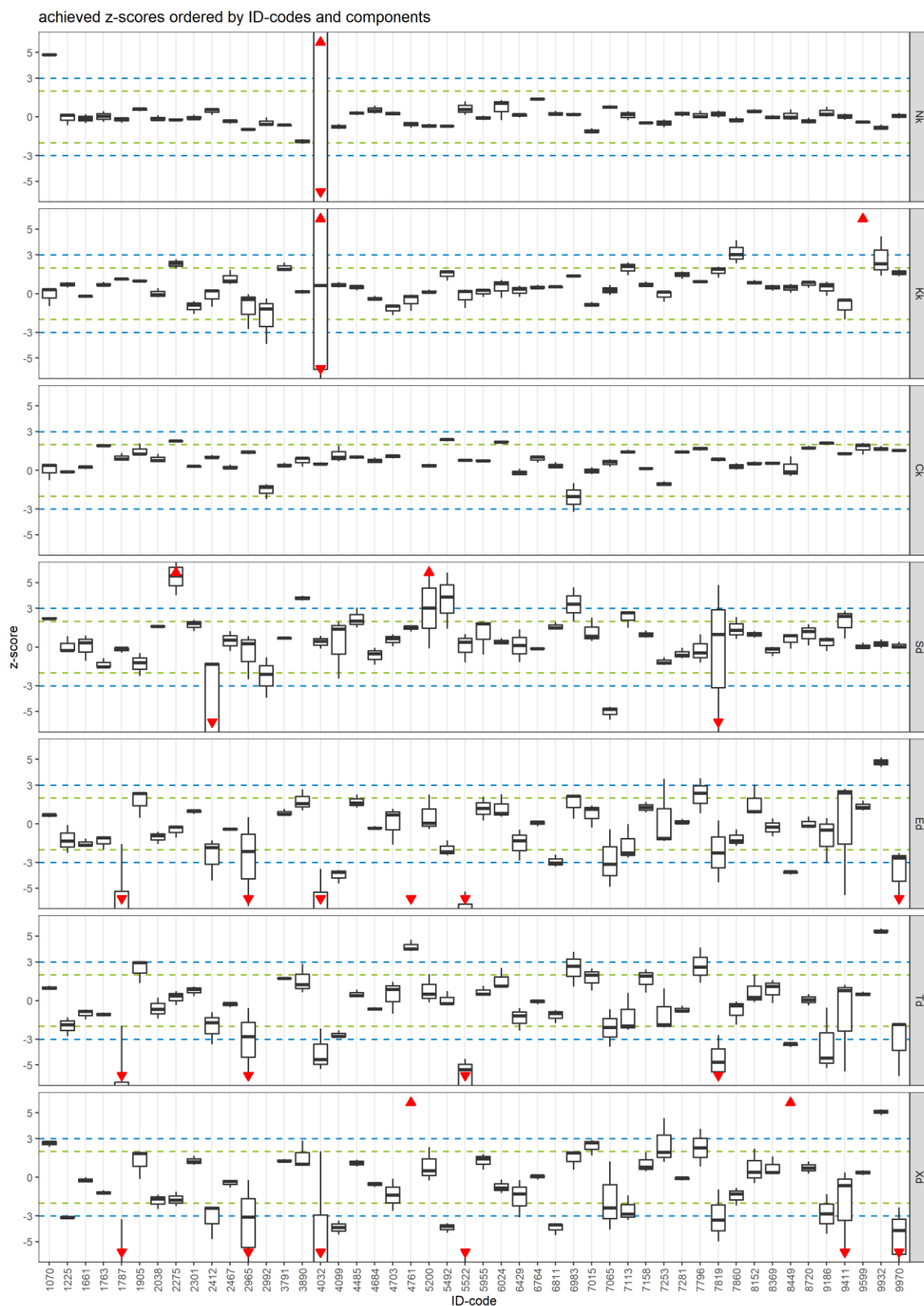
Scheme 2: Achieved z-scores dust proficiency test



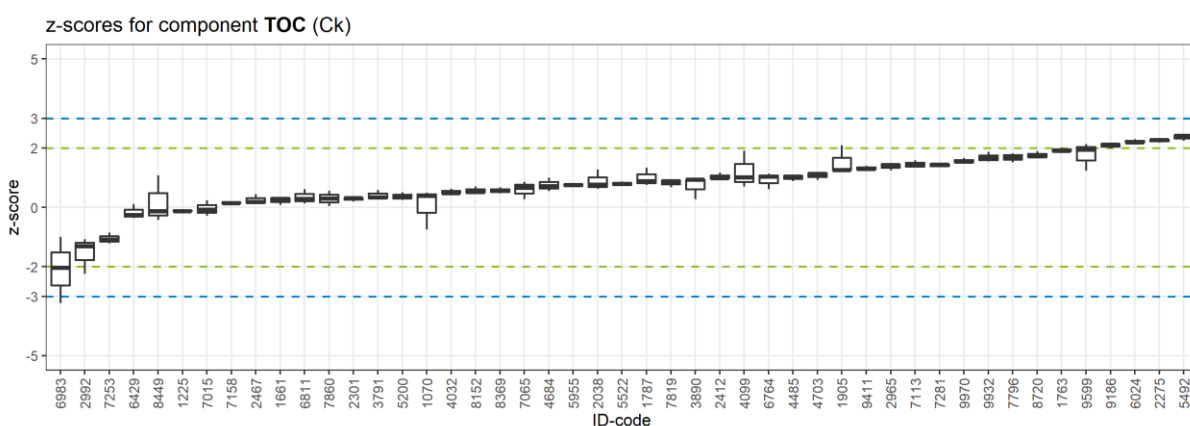
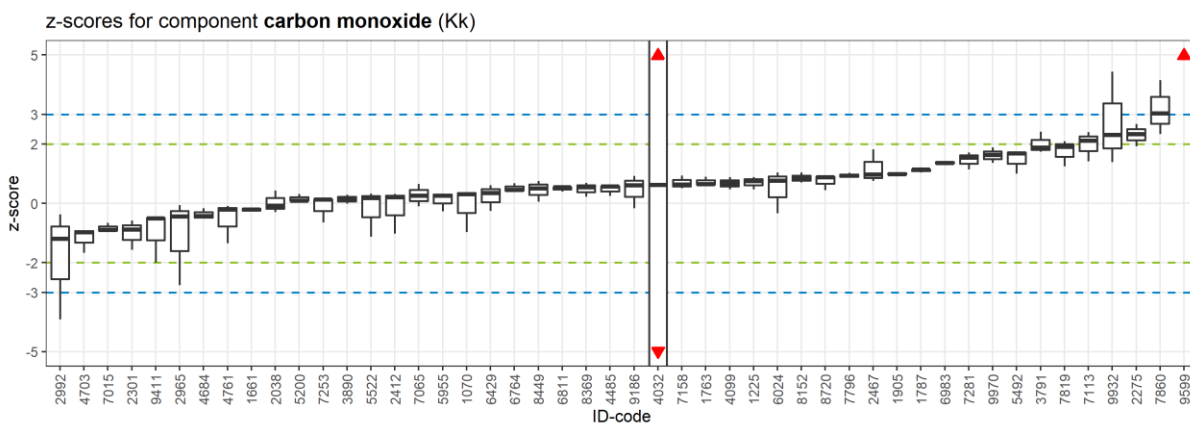
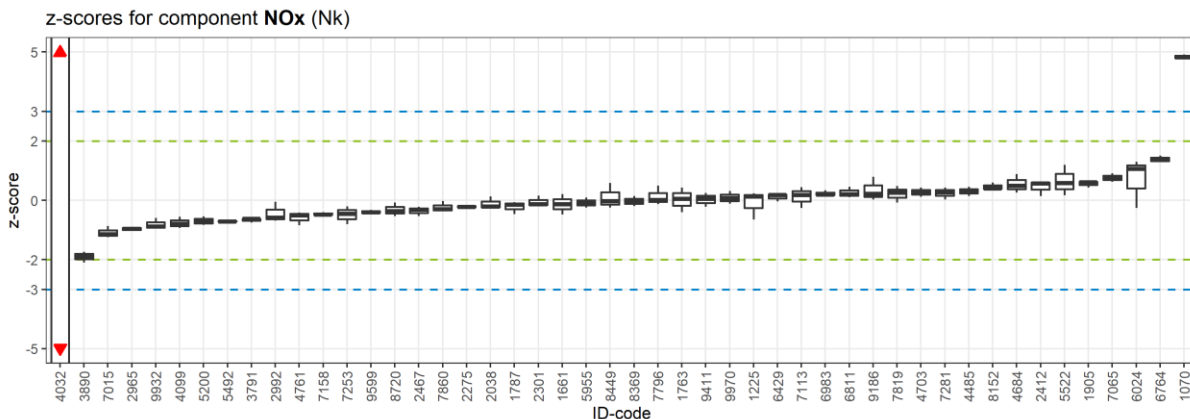


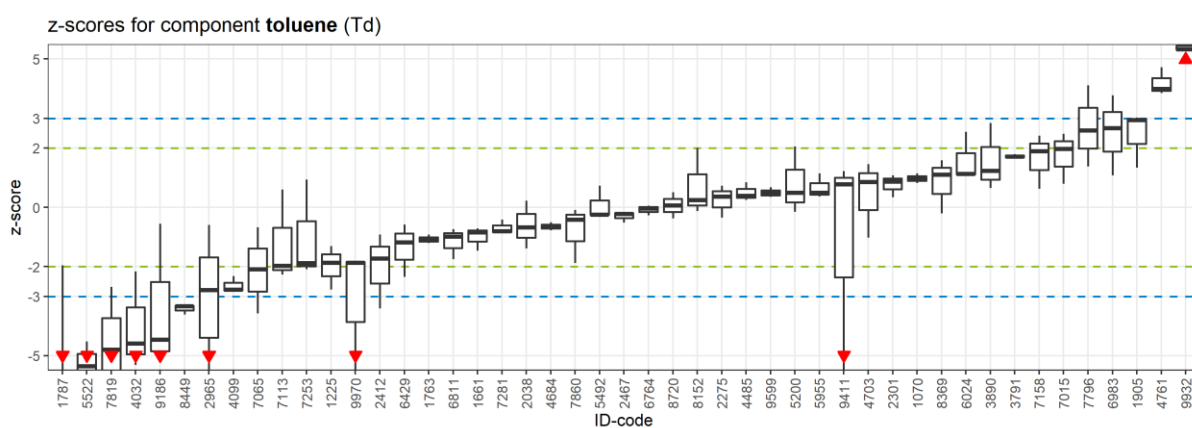
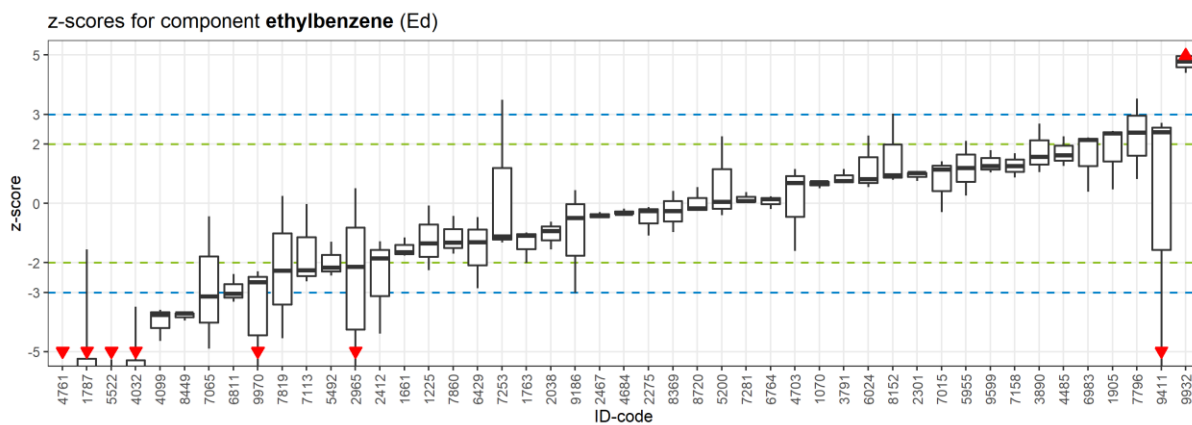
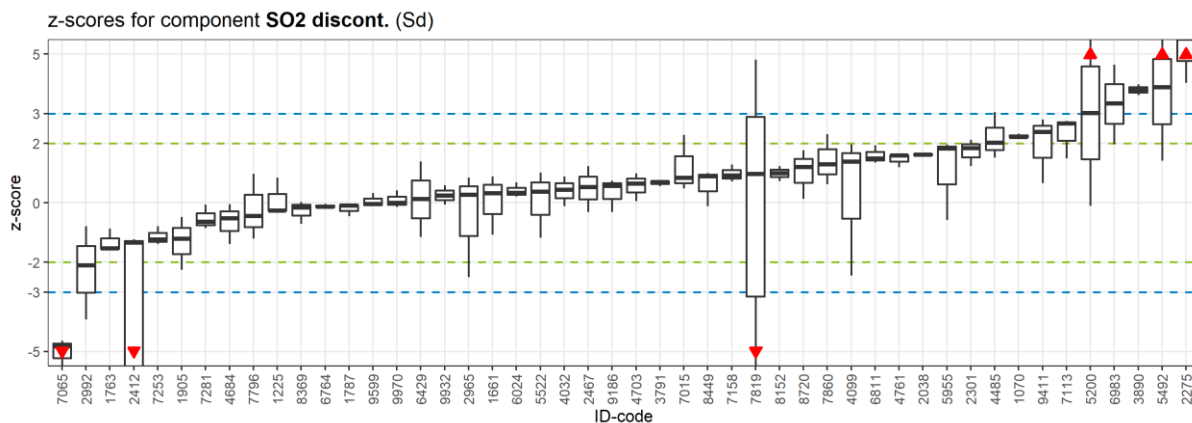


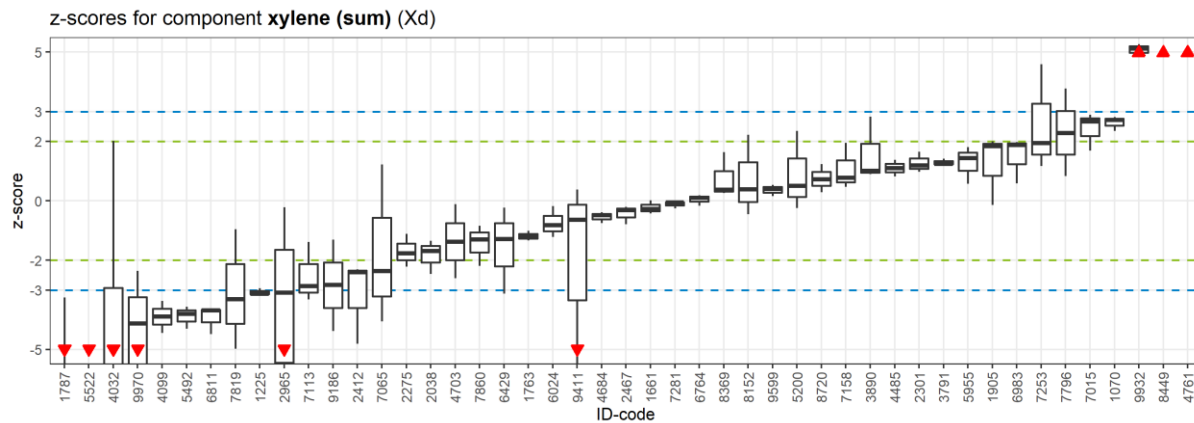
5.1.2 Gas (Substance Range G)



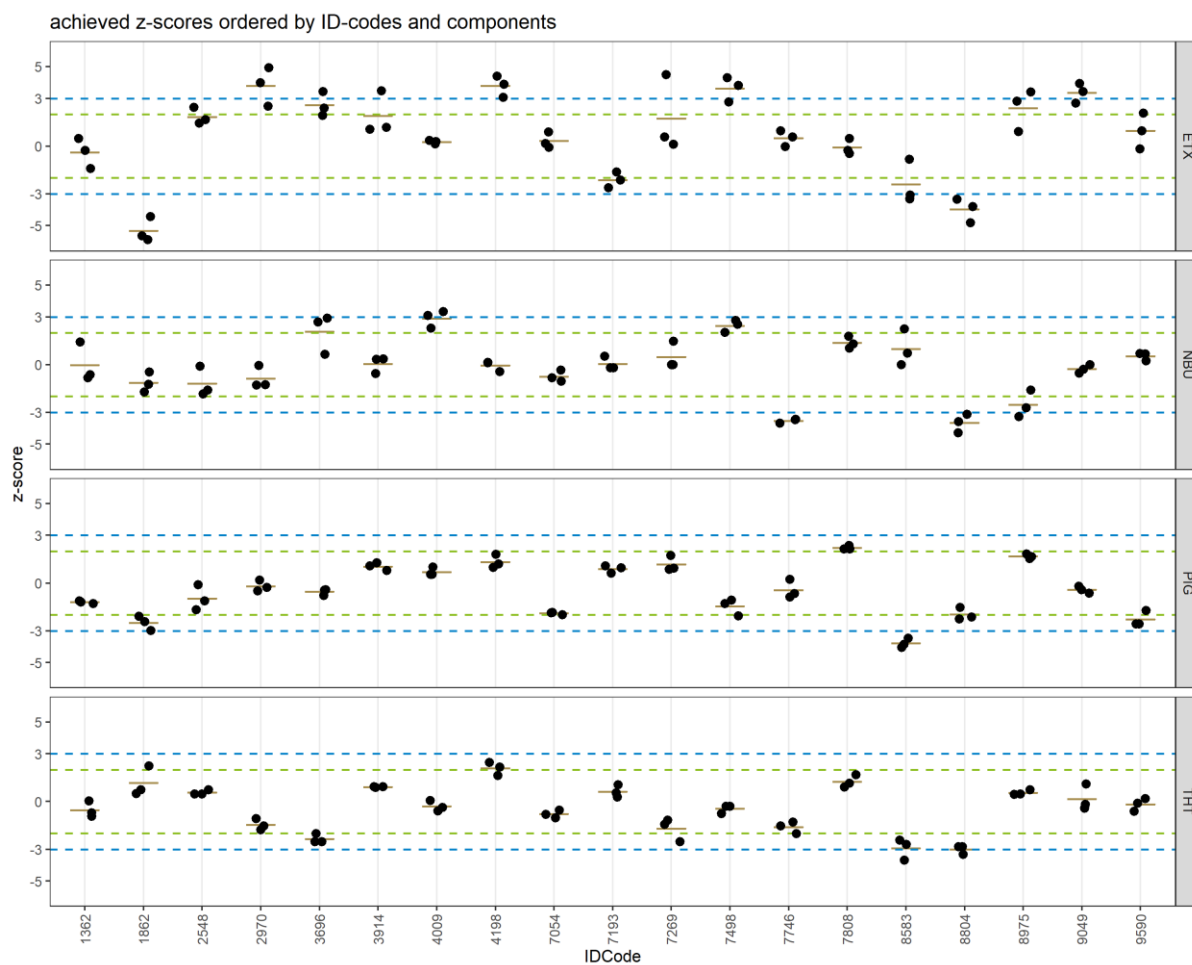
Scheme 3: Achieved z-scores gas proficiency test



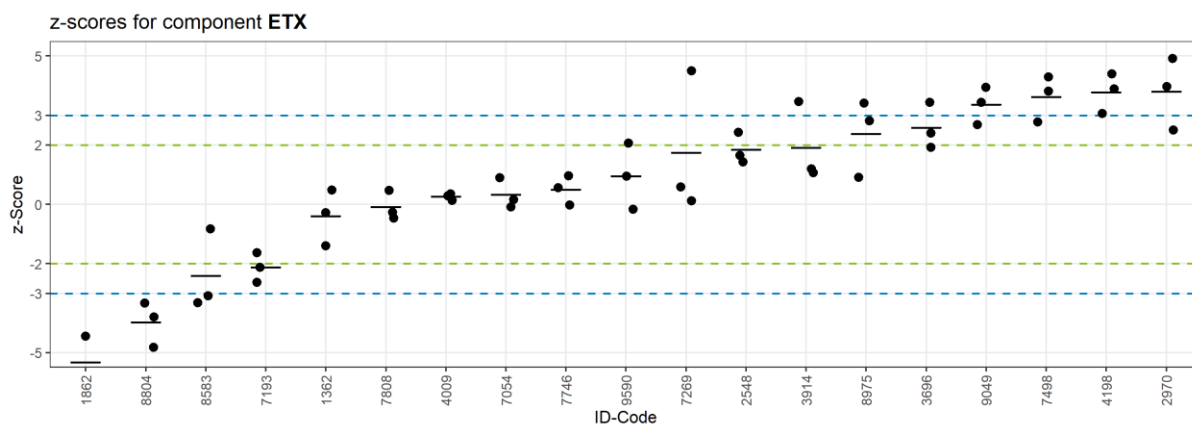


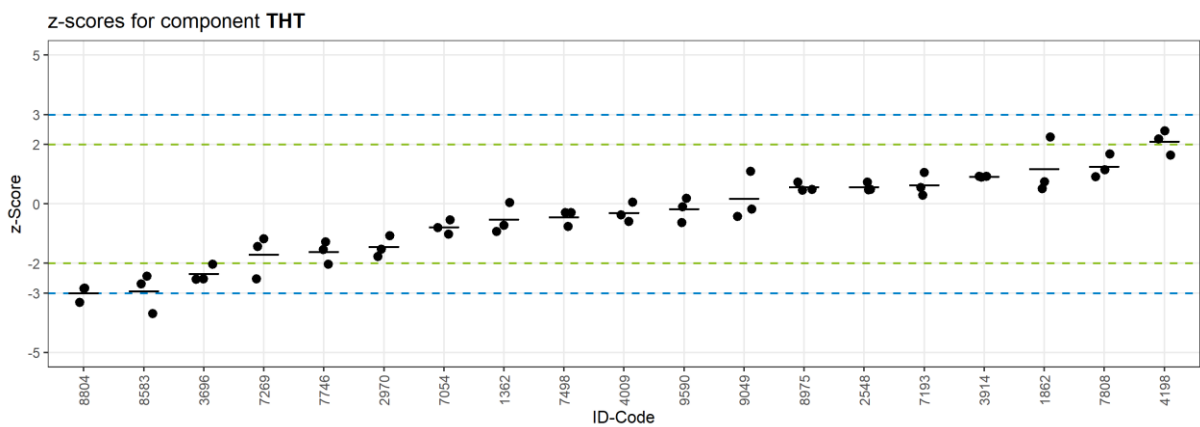
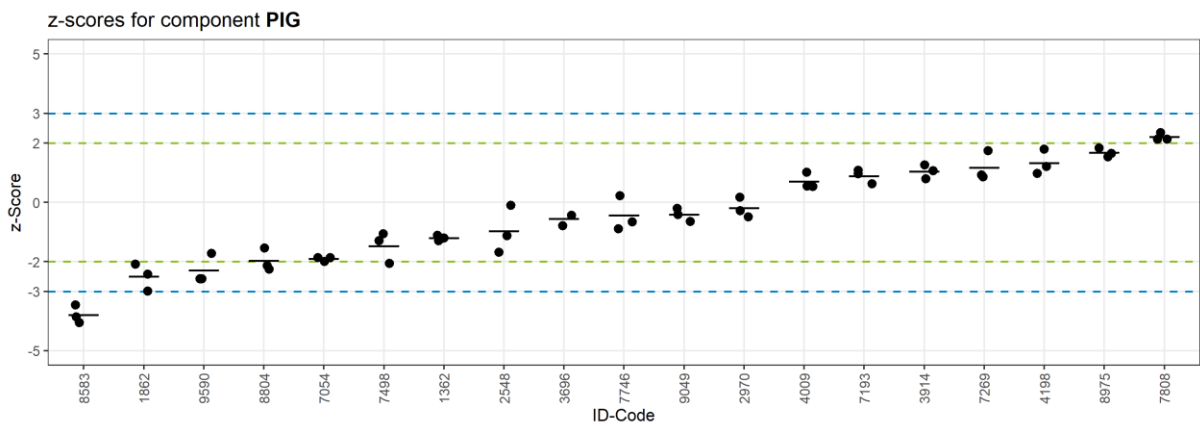
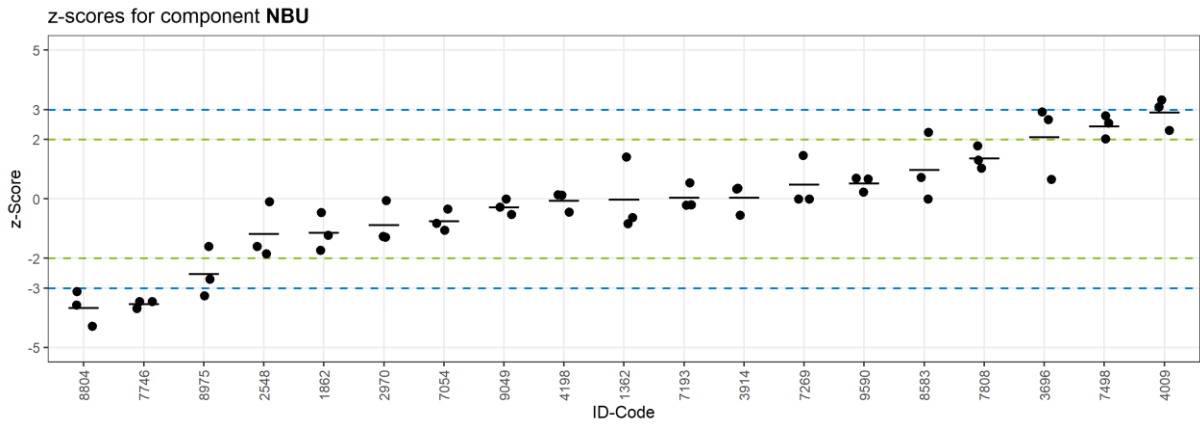


5.1.3 Odour (Substance Range 0)



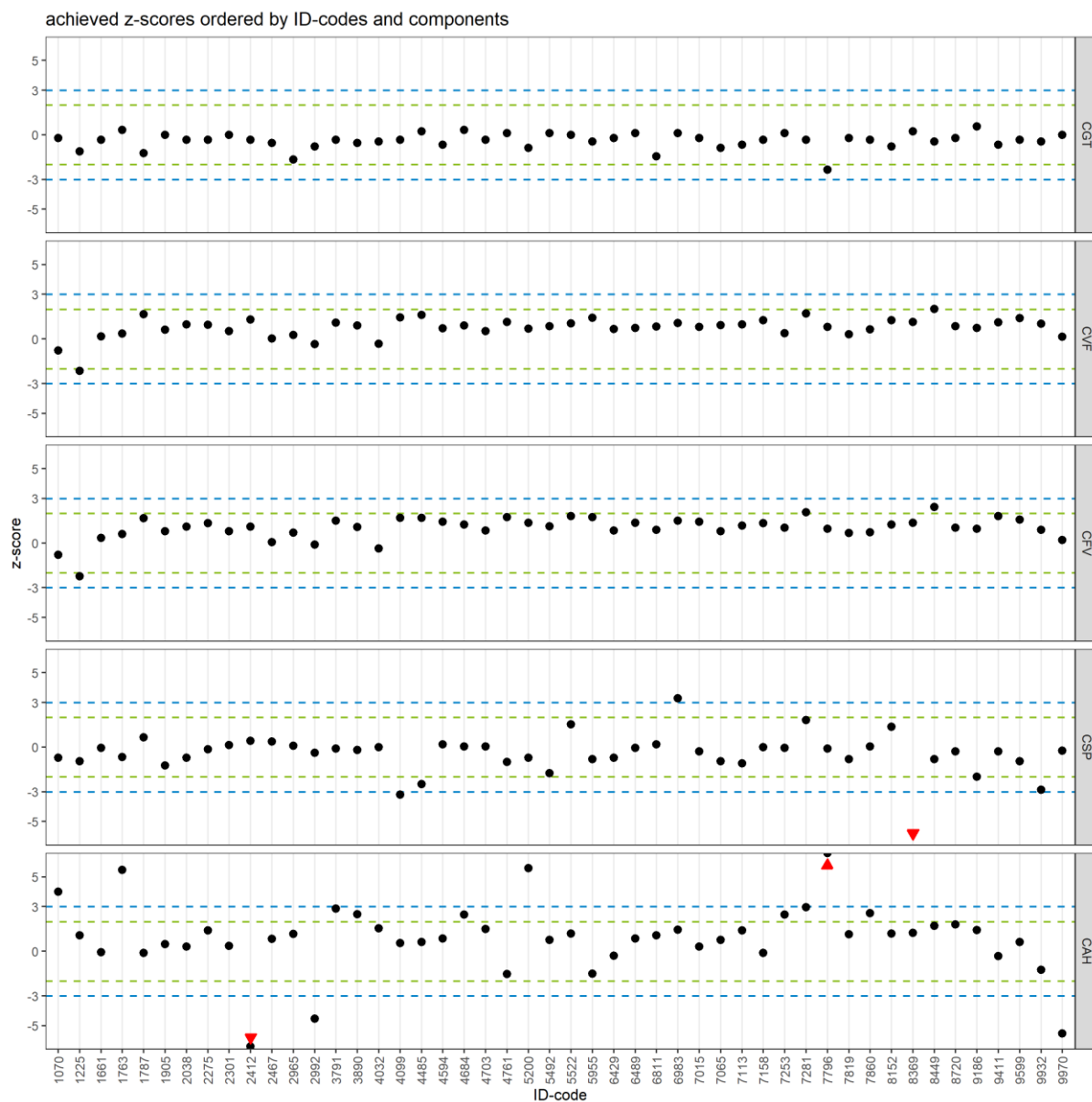
Scheme 4: Achieved z-scores odour proficiency test (only values in the range -5 ... 5 are shown)



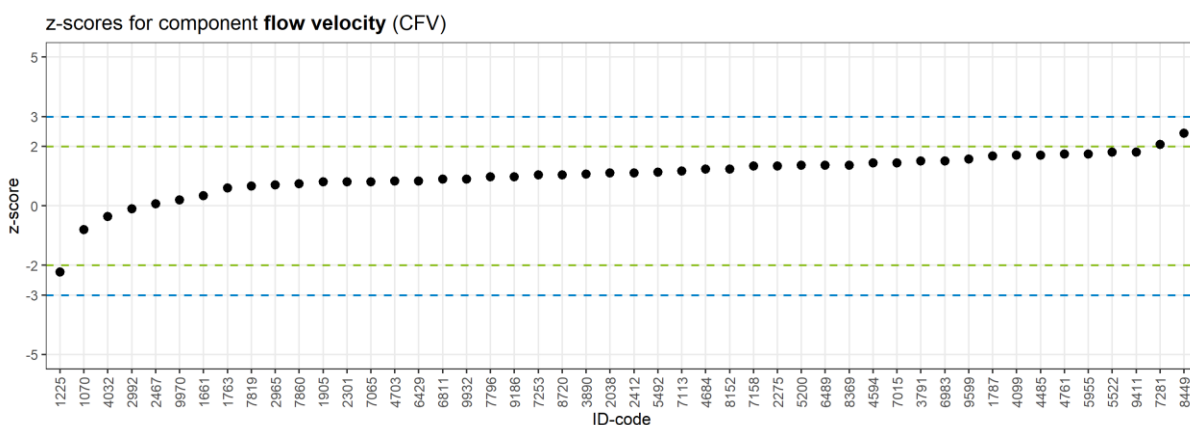
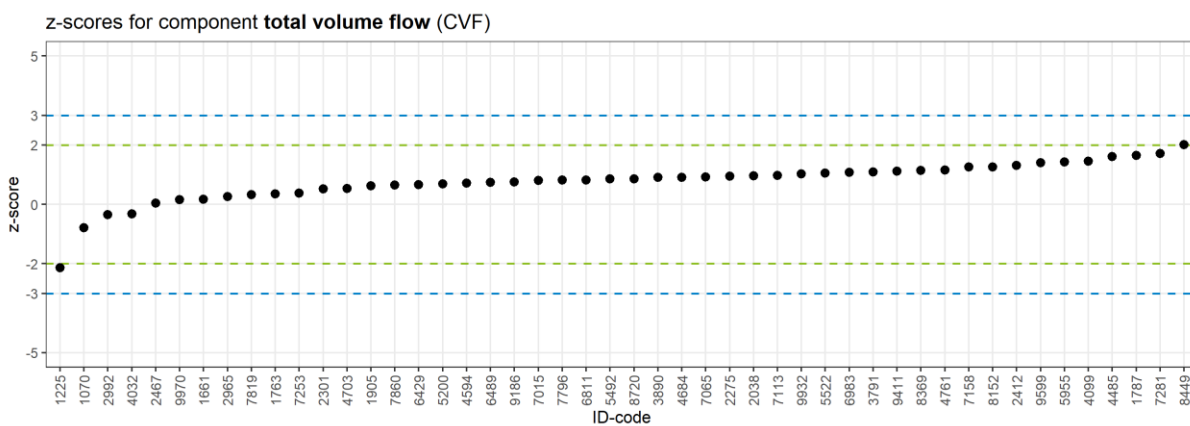
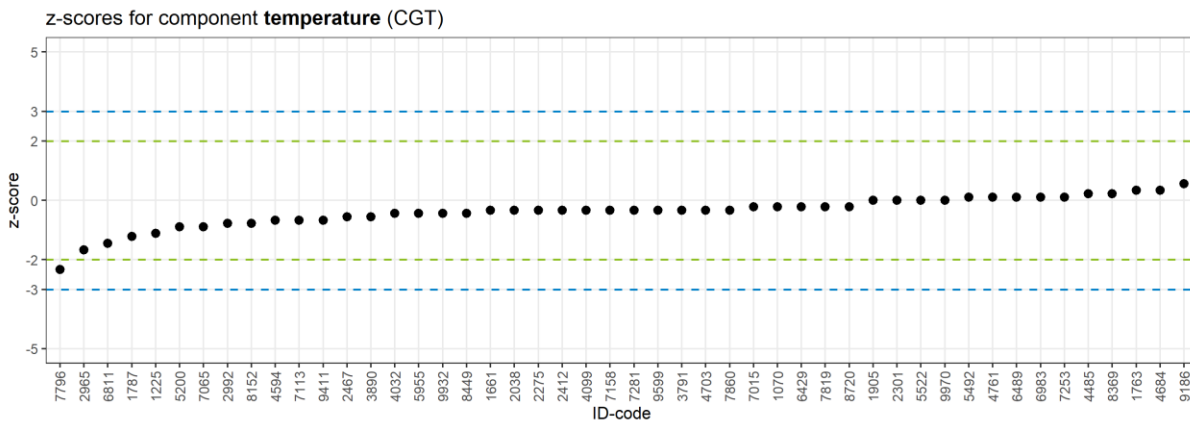


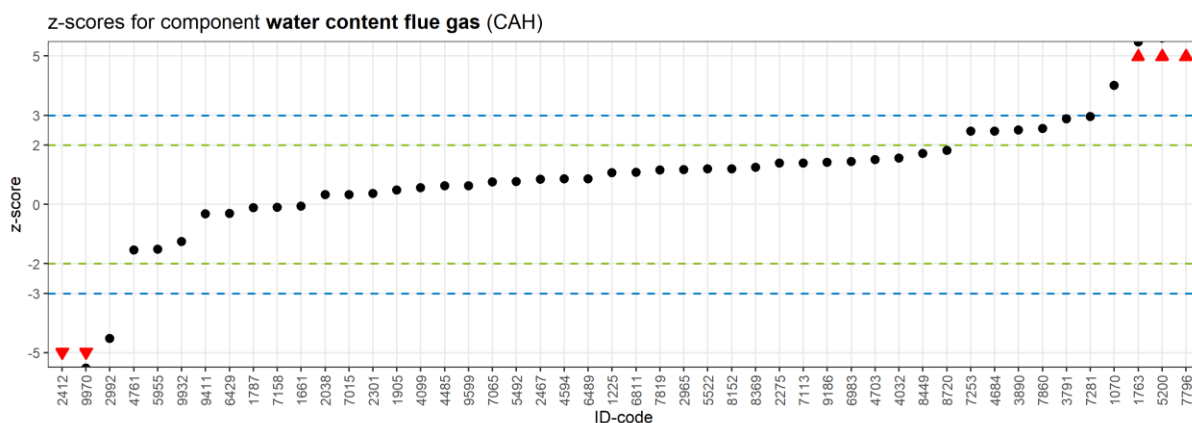
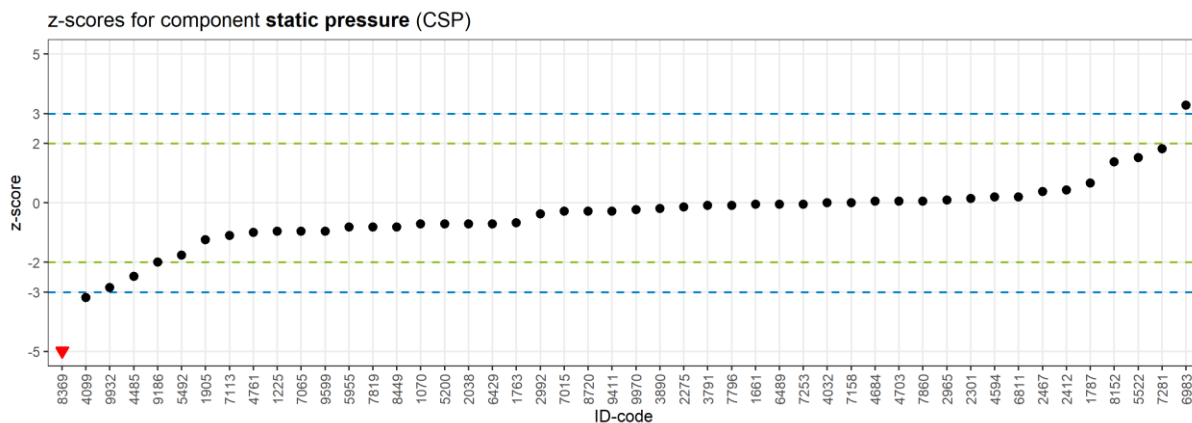
5.1.4 Gas flow conditions

The following diagrams show the results obtained by the participants in the dust proficiency tests for the measurement of the gas flow conditions. For the other types of proficiency tests, either no measurements were carried out or no evaluation criteria were defined. For each component, only one value is available per participant; this is shown as a point in each case.



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



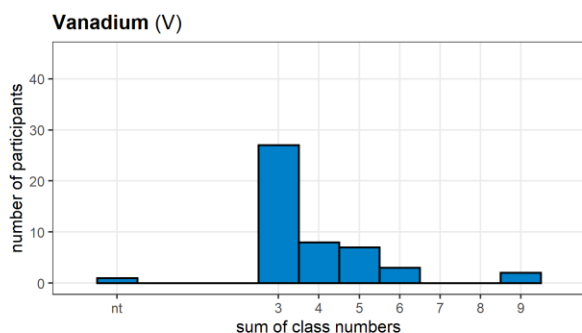
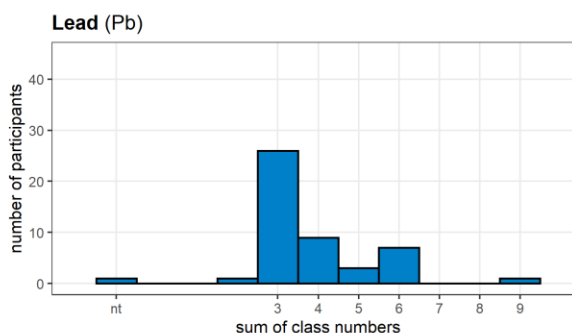
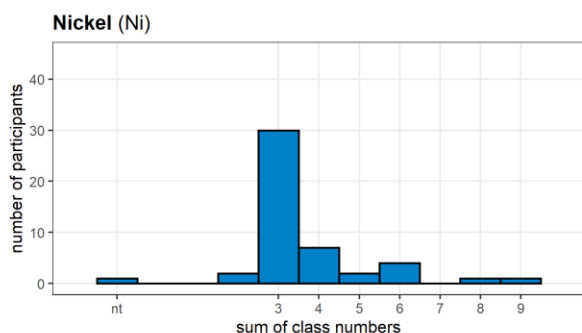
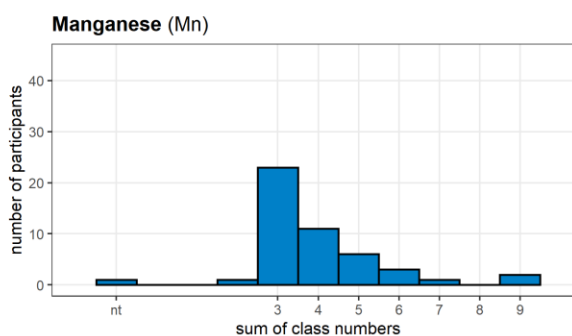
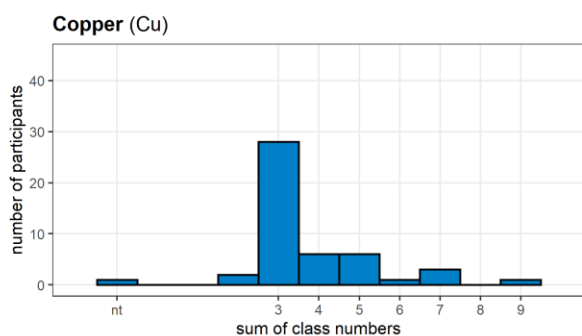
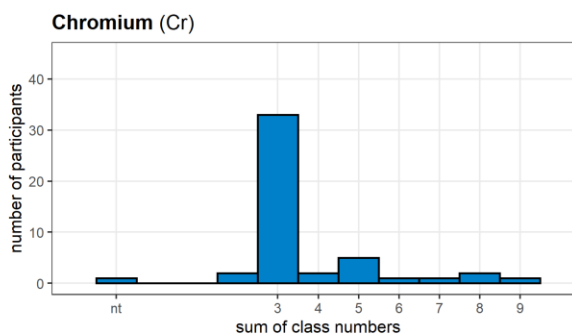
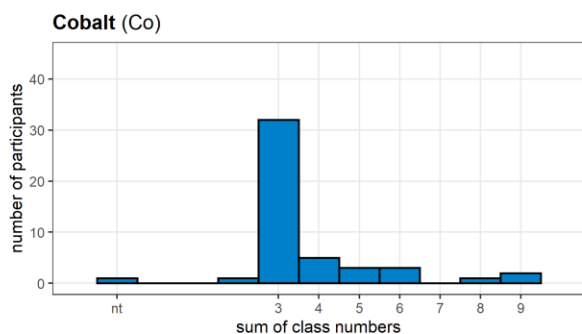
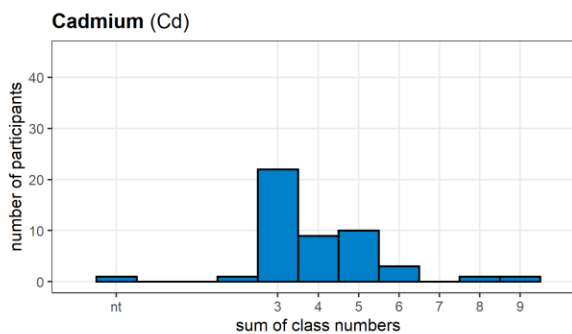
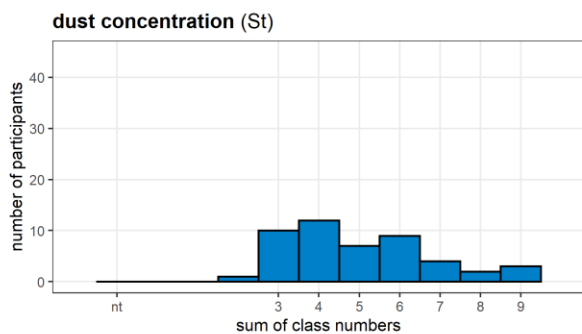


5.2 Sums of Class Numbers

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 **Fehler! Verweisquelle konnte nicht gefunden werden..** Participants that did not hand in results for a component are listed as “nt”.

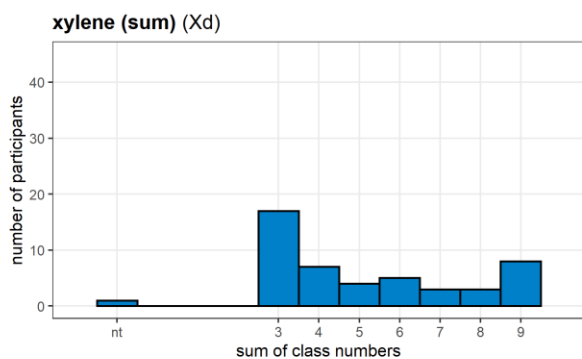
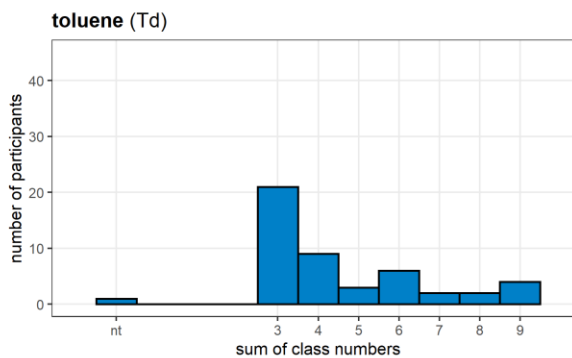
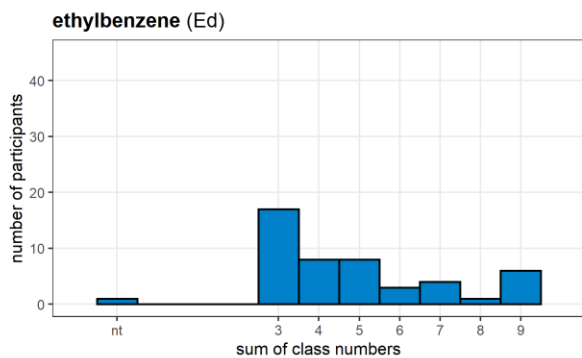
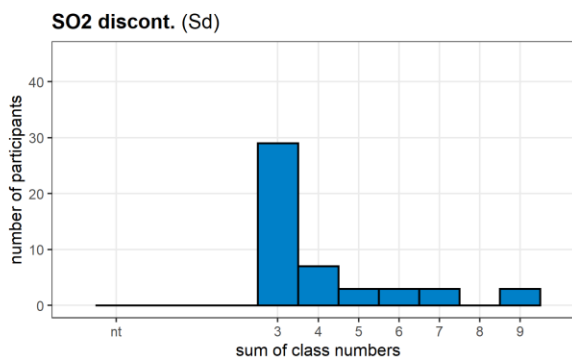
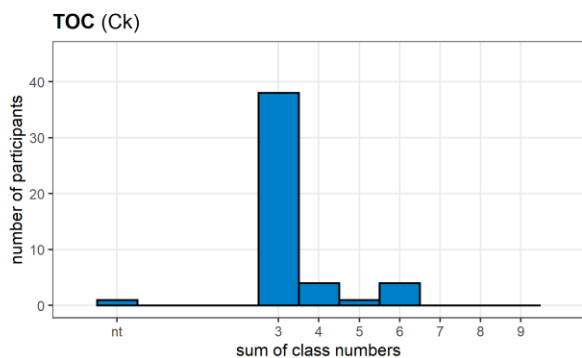
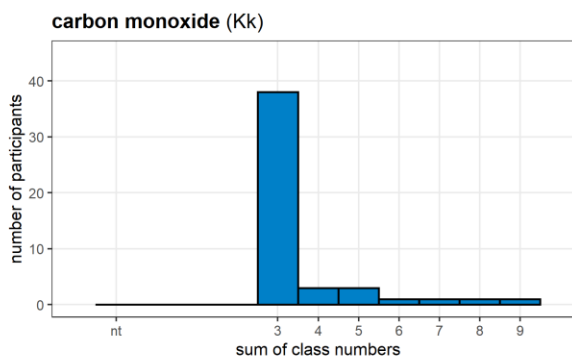
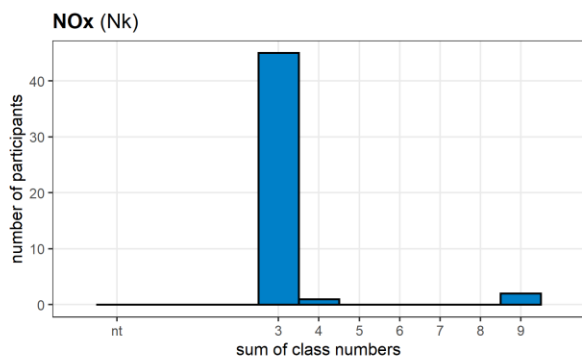
5.2.1 Dust (Substance Range P)

Sum of Class Numbers



5.2.2 Gas (Substance range G)

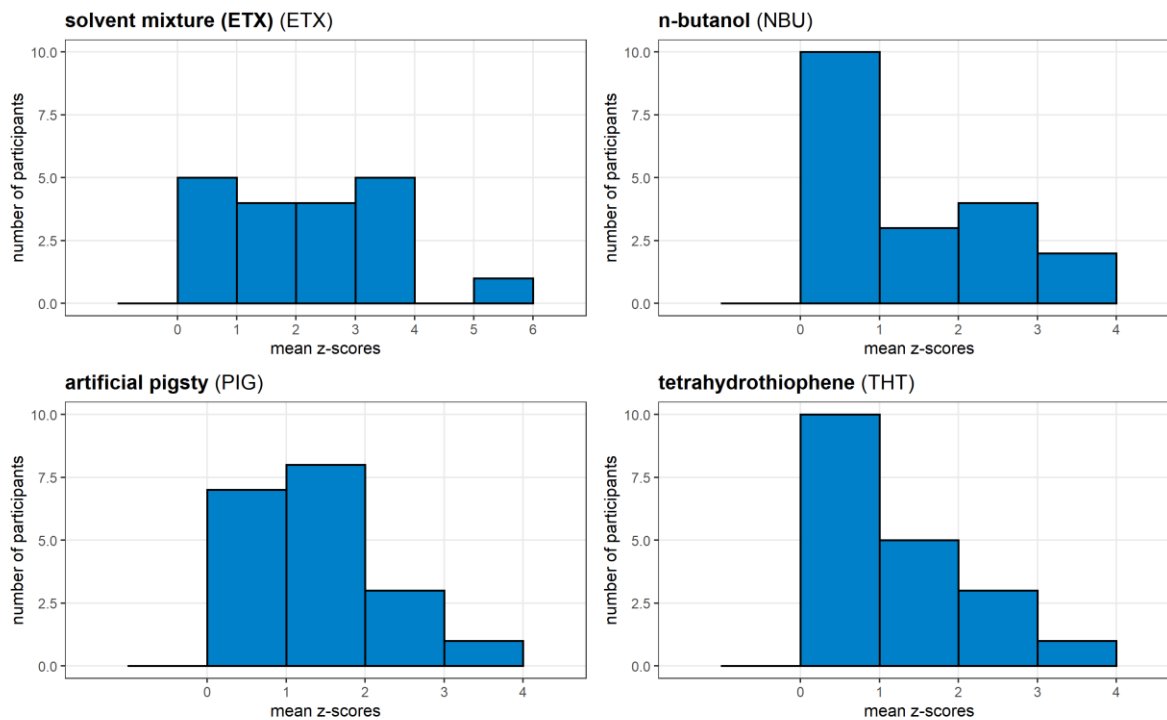
Sum of Class Numbers



5.2.3 Odour (Substance Range 0)

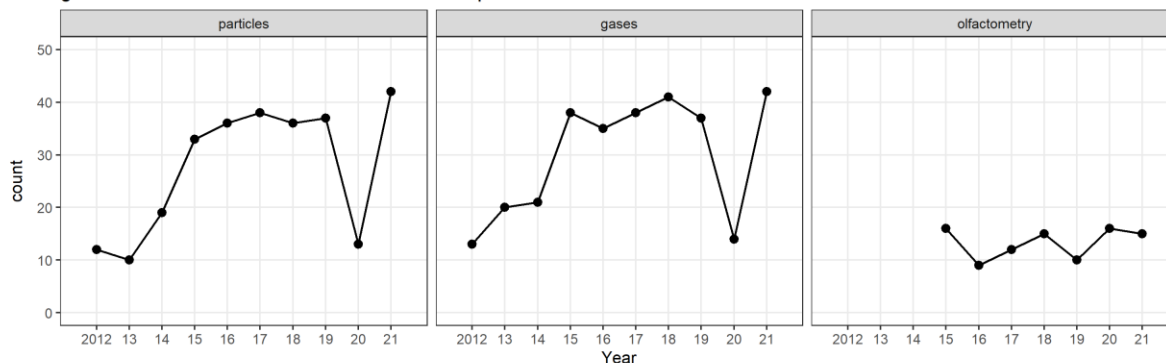
In odour emission proficiency tests, instead of sums of class numbers a mean value of z-scores is calculated. In the following histograms, the participants are allocated to a group by rounding down their mean z-score to the next lower integer.

Means of z-scores

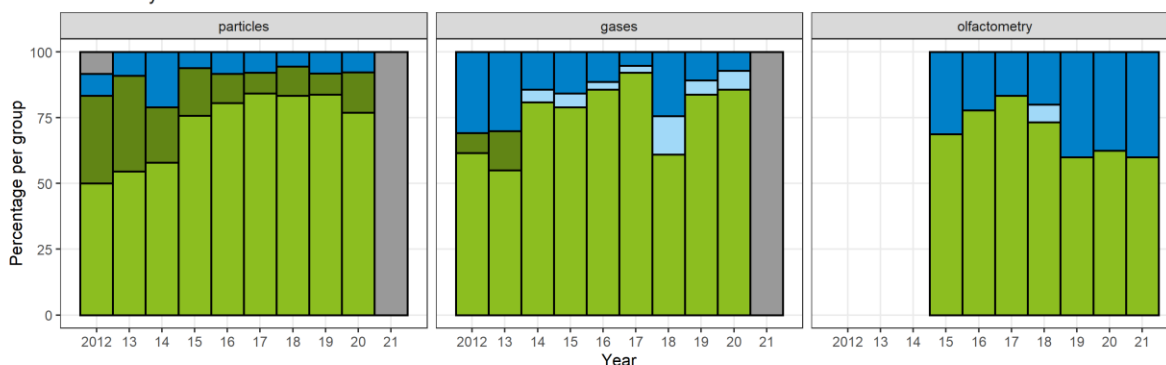


6. Interpretation of Results

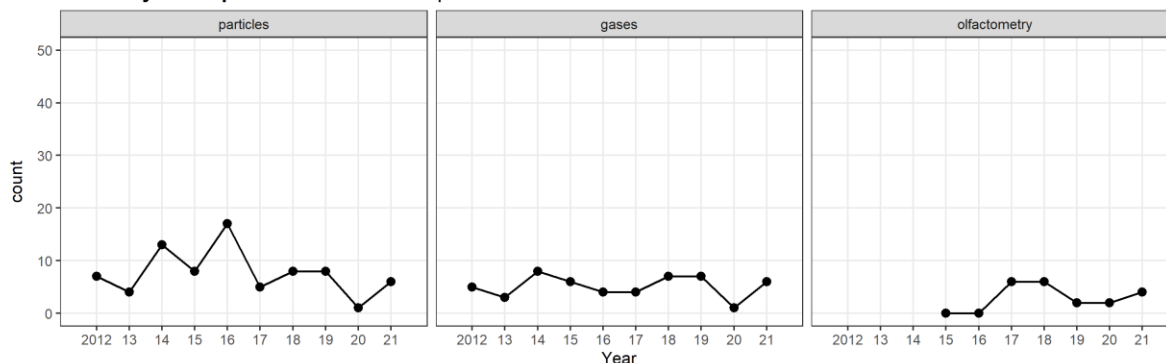
§29b Measurement Bodies Number of Participants



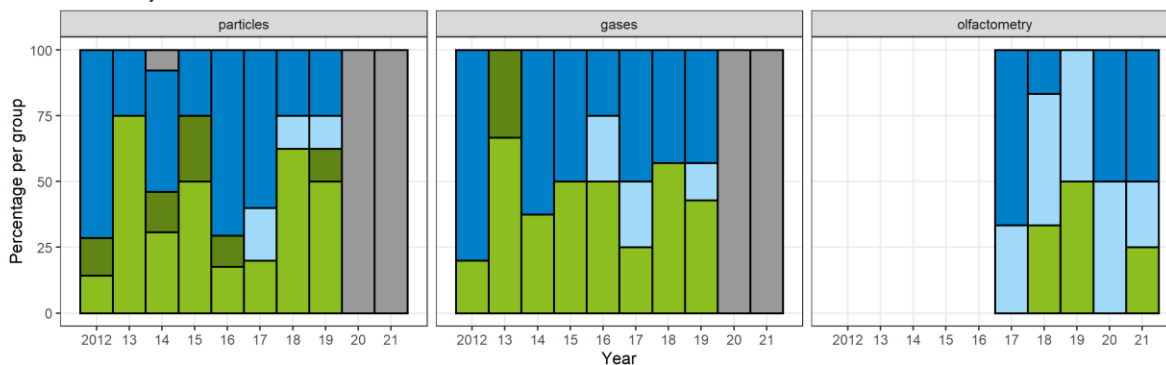
Proficiency Test Results



Voluntary Participants Number of Participants



Proficiency Test Results



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

Table 6: Overview of results since 2017 (§29b-bodies)

year	proficiency test	passed	passed (via post-analysis)	failed	failed (incomplete participation)
2017	gas	35	-	2	1
	odour	10	-	2	-
	dust	32	3	3	-
2018	gas	25	-	10	6
	odour	11	-	3	1
	dust	30	4	2	-
2019	gas	31	-	4	2
	odour	6	-	4	-
	dust	31	3	3	-
2020	gas	12	-	1	1
	odour	10	-	6	-
	dust	10	2	1	-
2021	gas		42 (not evaluated)		
	odour	9	-	6	-
	dust		42 (not evaluated)		

Table 7: Overview of results since 2017 (voluntary participants)

year	proficiency test	passed	passed (via post-analysis)	failed	failed (incomplete participation)
2017	gas	1	-	2	1
	odour	-	-	4	2
	dust	1	-	3	1
2018	gas	4	-	3	-
	odour	2	-	1	3
	dust	5	-	2	1
2019	gas	3	-	3	1
	odour	1	-	-	1
	dust	4	1	2	1
2020	gas	-	-	-	-
	odour	-	-	1	1
	dust	-	-	-	-
2021	gas		6 (not evaluated)		
	odour	1	-	2	1
	dust		6 (not evaluated)		

6.1 §29b Measuring Bodies

Due to the SARS-CoV-2 pandemic in 2020 the majority of the proficiency tests had to be cancelled and the number of participants in the dust and gas ring tests dropped significantly as a result. In 2021, however, the number of proficiency test participations by §29b measurement bodies returned to the pre-pandemic level. While the odour proficiency tests could continue to be carried out essentially unchanged, the dust and gas proficiency tests in 2021 took place exclusively in the new "pandemic version". In deviation from the LAI specifications, the number of measurements carried out and evaluated 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 LAI specifications, no overall evaluations were carried out for the dust and gas proficiency tests. If this had been carried out, 31 out of 42 (74%) of the authorized measuring bodies would have passed the dust proficiency test and 22 out of 42 (52%) the gas proficiency test. The pass rates are thus somewhat (dust) or significantly (gas) lower than in previous years. A direct comparison with the results from 2019 (the last year with a comparably large number of participants) shows that the relative deviations of the measured values from the target values were larger on average for practically all dust and gas components in 2021, and there were also a much larger number of extreme "outliers". In the pandemic version, there were two crucial changes compared to 2019: The number of measurements was reduced from 9 to 3, which should have no impact on the mean deviation of the submitted measurements from the target values. In addition, due to the hygiene concept, participants did not have the opportunity for an exchange with other participants. Whether this had an influence on the submitted measured values cannot be verified.

An evaluation of the results with the evaluation criteria of the previous LAI specifications would also have led to the overall result "passed" for 31 of the 42 (74%) authorized measuring bodies for the dust proficiency tests, and for the gas proficiency test this would have been the case for 26 of 42 (62%) of the §29b measuring bodies, whereby it must be pointed out that the component formaldehyde is completely missing here. The poor overall picture is thus demonstrably not an effect of the changed evaluation criteria, but a result of the objectively poorer performance of the participants in 2021.

In 2021, a total of 9 out of 15 authorized measuring bodies (60%) passed the odour proficiency test. The results of the odour proficiency test were thus comparable to the results of 2020 and 2019. Here, the determination of odour concentrations for the mixture of organic solvents (ETX) again seems to pose the greatest difficulties for the participants. Since measurement results both significantly above and significantly below the assigned values were determined, it can be concluded that the number of test persons, which is usually 4, is clearly too low from a statistical point of view in view of the uncertainty of individual test person results, plays a decisive role here.

6.2 Voluntary Participants

In 2020, due to the SARS-CoV-2 pandemic, not a single volunteer participant came to Kassel for a dust or gas proficiency test, either because the booked proficiency test had to be cancelled or because the changing travel restrictions and quarantine regulations had made it virtually impossible to travel. Fortunately, the number of voluntary proficiency test participants in 2021 almost returned to the pre-pandemic level. In total, there were 6 voluntary participants in each of the dust and gas proficiency tests, as well as 4 voluntary participants in the odour proficiency test.

Due to the deviations from the LAI specifications, no overall evaluations were carried out for the dust and gas proficiency tests, not even for the voluntary participants. If these had been carried out, 2 out of 5 (40%) of the volunteers would have passed both the dust and gas proficiency tests, while the sixth volunteer measured only some of the components in both proficiency tests (but successfully). These pass rates correspond roughly to what was observed in previous years. A direct comparison is difficult here, however, since the collected results of a year are extremely influenced by the performance of individual laboratories due to the usually small number of voluntary participations in many years. A long-term comparison is therefore only informative to a limited extent.

In the olfactometry proficiency tests, 2 of the 4 voluntary participants (50%) passed. Another participant (25%) delivered correct measured values, but these were not determined within 6 h in accordance with VDI 3880. The results were therefore classified as "failed (incomplete participation)".

6.3 Basic 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 evaluated 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. In the pandemic version of the dust and gas proficiency tests, the gas flow conditions were only measured before the dust measurements began. For the gas proficiency tests following the dust samplings, the gas flow conditions were kept constant and no new measurement was carried out by the participants in the afternoon. Instead of the planned 2 measured values, only one measured value per participant was determined. The values recorded in the dust proficiency tests (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 static pressure (CSP) there are individual "outliers", for flue gas humidity (CAH) there are several of them and also larger deviations from the target values overall.

The gas flow conditions were not evaluated due to the deviations from the LAI specifications. However, according to the assessment criteria specified there, all participants in the proficiency tests (both §29b measuring bodies and voluntary participants) would have passed this part of the proficiency test in the event of an evaluation.

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 2021.

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 (3 measurements in the pandemic version, respectively) 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, there are hardly any changes compared to previous years. This is ultimately due to the fact that the data basis has only increased by about a quarter since the 2019 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.

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.

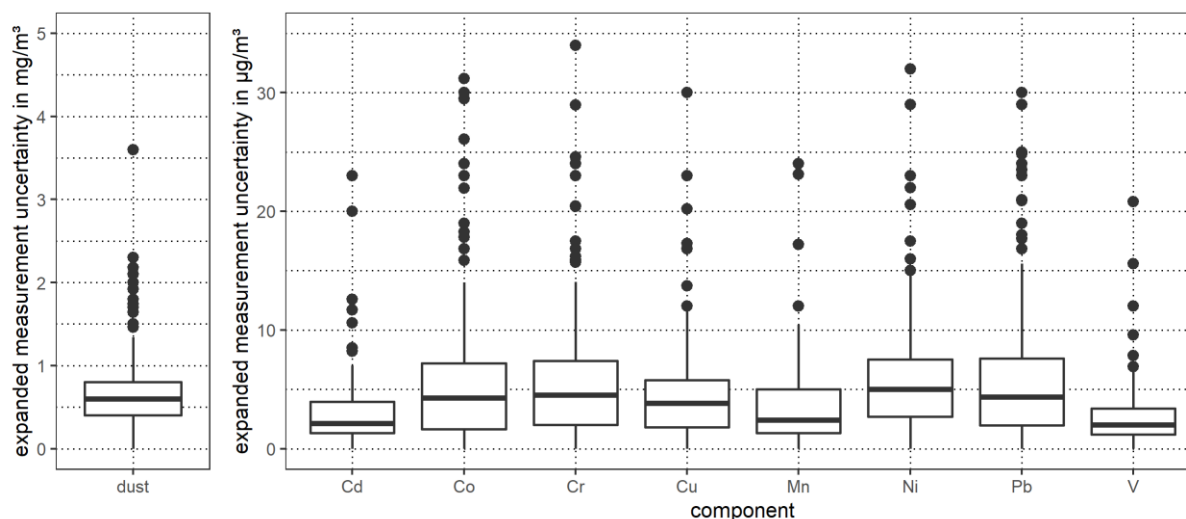


Table 8: Expanded measurement uncertainties reported by participants of the dust proficiency test

	dust [mg/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	0.81	4.00	7.50	7.98	5.88	5.00	7.78	8.06	3.80
median	0.62	2.22	4.40	4.71	3.93	2.52	5.00	4.83	2.04
25 th percentile	0.41	1.37	1.80	2.02	1.97	1.36	2.80	2.00	1.30
number of values	173	154	155	155	155	117	155	154	114

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.

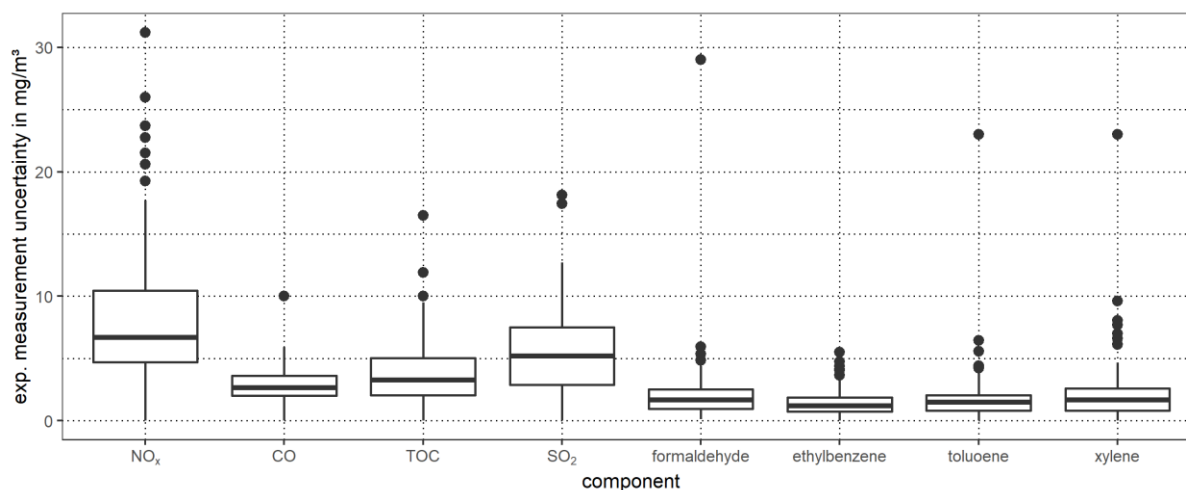


Table 9: Expanded measurement uncertainties reported by participants of the gas proficiency test

	NO _x as NO ₂ [mg/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	10.55	3.60	5.10	7.51	2.52	1.89	2.04	2.60
median	6.73	2.69	3.37	5.20	1.69	1.20	1.50	1.70
25 th percentile	4.87	2.00	2.25	2.90	0.97	0.75	0.80	0.80
number of values	169	93	164	169	125	166	167	167

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 89 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.

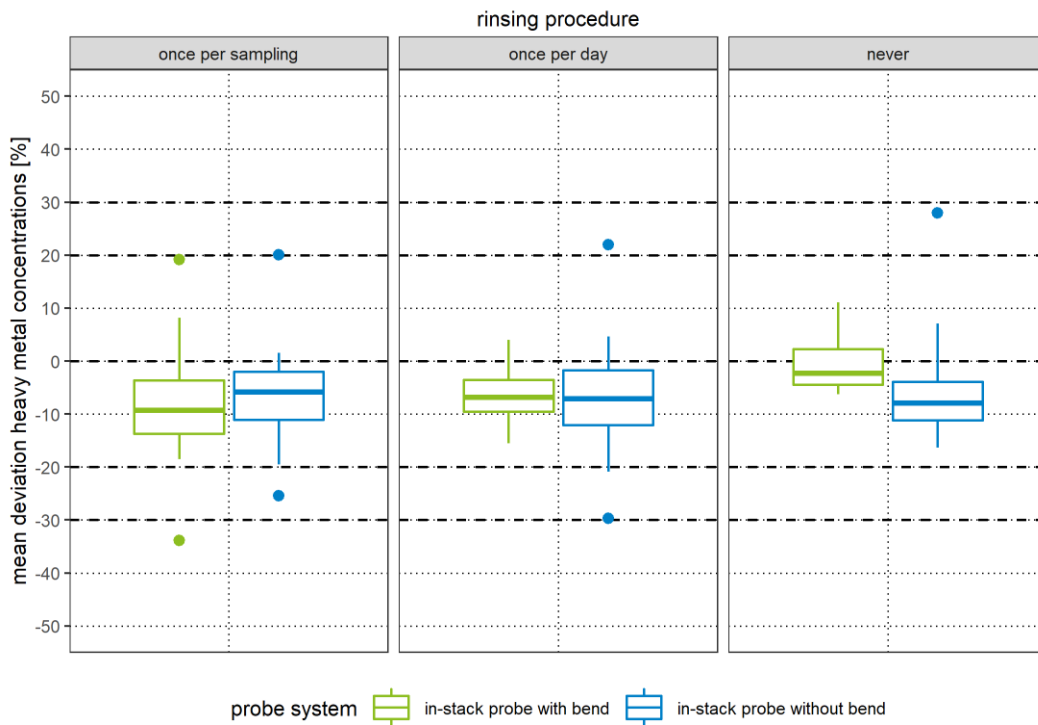
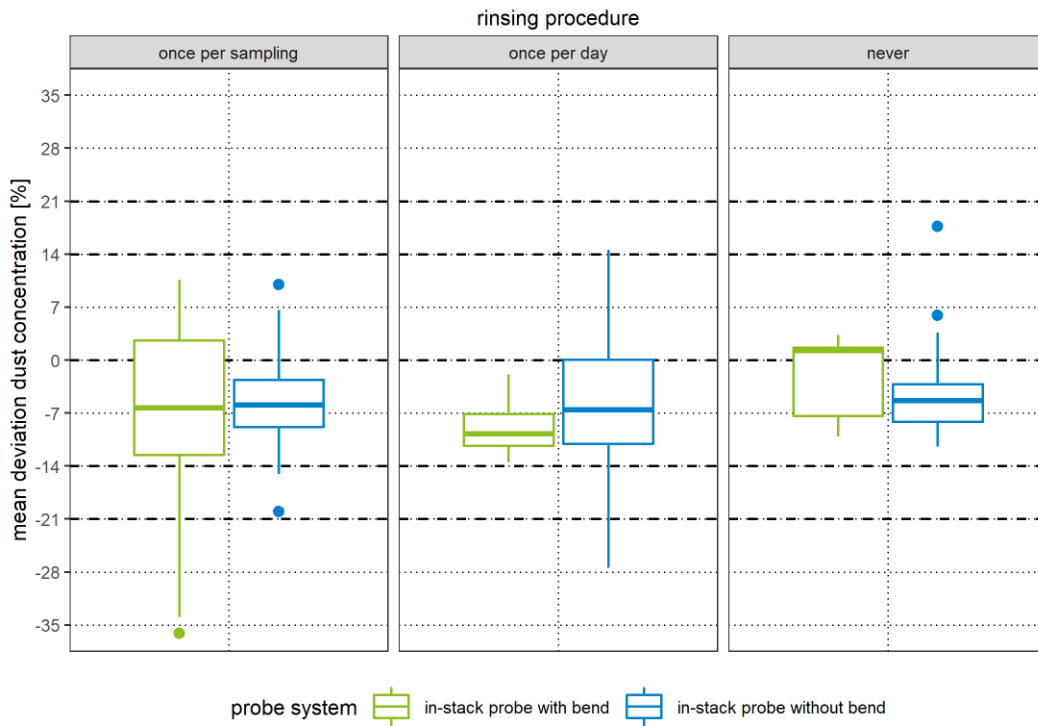


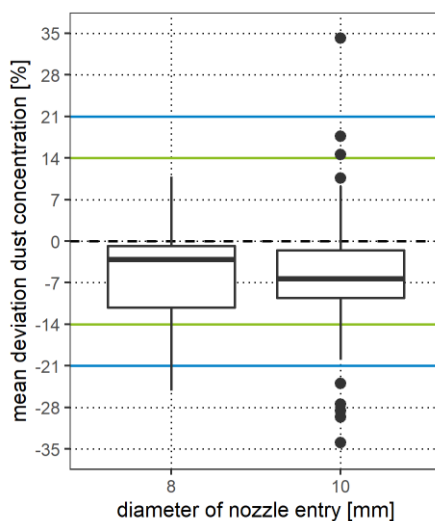
Table 10: Correlation of dust measurement results with probe systems and rinsing procedures (2018-2021)

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	-6.3%	17	-8.1%	17
2 (centre)		once per day	-9.7%	4	-6.9%	4
3 (right)		no rinsing	1.3%	5	-2.3%	4
4 (left)	in-stack probe without bend	after each sampling	-5.9%	22	-5.2%	22
5 (centre)		once per day	-6.7%	27	-7.0%	27
6 (right)		no rinsing	-5.3%	14	-7.5%	14

Due to the very small number of cases, the results shown are considerably influenced by various factors in the respective laboratories. The above-average results for combination 3 (probe with bend that is not rinsed: right-hand figures, green), for example, are unlikely to be representative of this type of sampling. Combination 3 is explicitly not in conformity with the standard, since with this probe geometry, dust deposits on the inner surface of the probe are to be expected in any case, which can lead to significantly lower results if rinsing is not carried out.

On average, the results of the dust measurements in the proficiency tests of 2021 also show significant lower findings, as did the results of the previous years. The HLNUG published a detailed investigation of this phenomenon and its probable cause in a scientific journal last year. (17).

7.3 Diameter of the Nozzle Opening in Dust Samplings

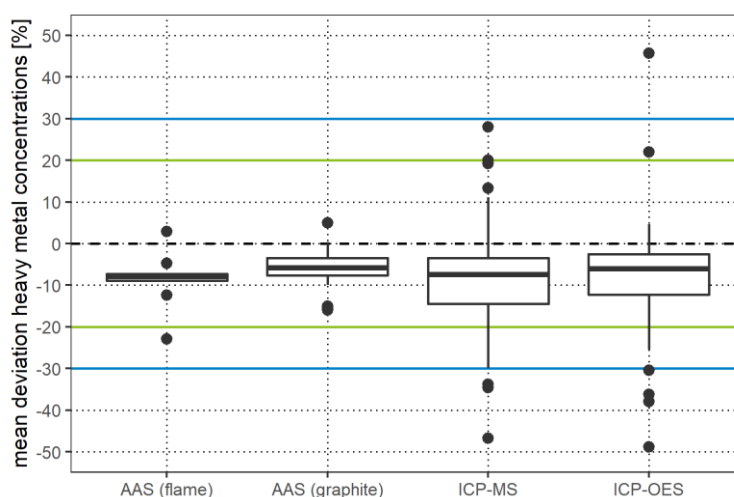


The information provided by the participants on the diameter of the probe’s nozzle opening does not indicate a clear trend. Regardless of the diameter, the measured values always seem to scatter over a wide range. Overall, however, the size of the nozzle opening does not seem to be a determining factor 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 (2016-2021)

diameter of nozzle opening	8 mm	10 mm
75 th percentile	-0.78%	-1.52%
median	-3.07%	-6.31%
25 th percentile	-11.20%	-9.56%
number of values	42	152

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 27 participants stated that heavy metal analysis was performed using AAS equipment, while 181 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.

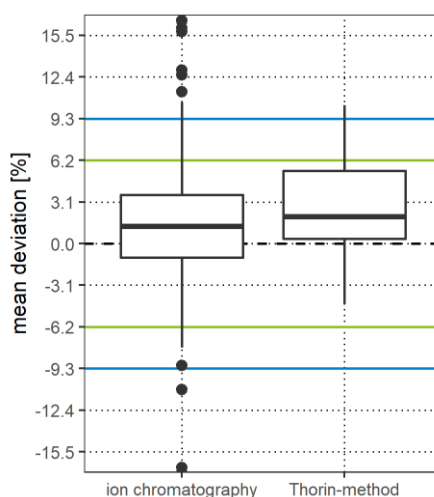
Table 12: Correlation of heavy metal results and analysis devices

analysis device	flame-AAS	graphite furnace AAS	ICP-MS	ICP-OES
75 th percentile	-7.21%	-3.49%	-3.47%	-2.34%
median	-7.85%	-5.81%	-7.44%	-6.00%
25 th percentile	-8.85%	-7.69%	-13.91%	-11.77%
number of values	10	17	107	74

7.5 Formaldehyde

Unfortunately, no measurements of formaldehyde concentrations could be carried out in 2021 as part of the gas proficiency tests. The data collected on this component in the past will not be reproduced here. The latest information can be found in the 2020 annual report.

7.6 Sulphur Dioxide



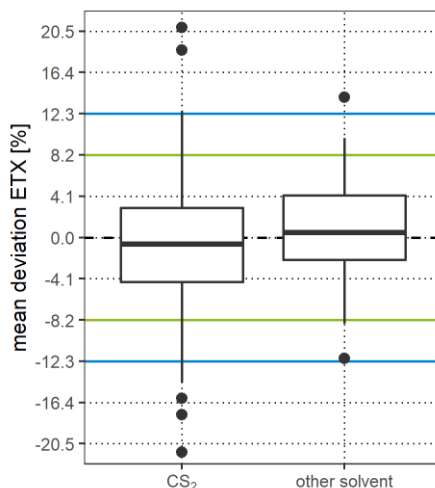
For the discontinuous determination of sulphur dioxide concentrations, participants can choose between analysis of the samples using ion chromatography or the Thorin method as part of the standard reference method. The following picture emerges from the information provided by the participants:

Table 13: Correlation of sulphur dioxide measurement results with the analytical method used (2016-2019)

method	ion chromatography	Thorin-method
75 th percentile	3.59%	5.41%
median	1.23%	2.01%
25 th percentile	-1.01%	0.36%
number of values	194	17

The available results show a slightly lower deviation on average for the ion chromatography method, but the number of participants using the Thorin method is comparatively small. The higher scatter of the IC method with various "outliers" is possibly solely due to the more than 10 times higher number of participants.

7.7 Solvents for Desorption of ETX



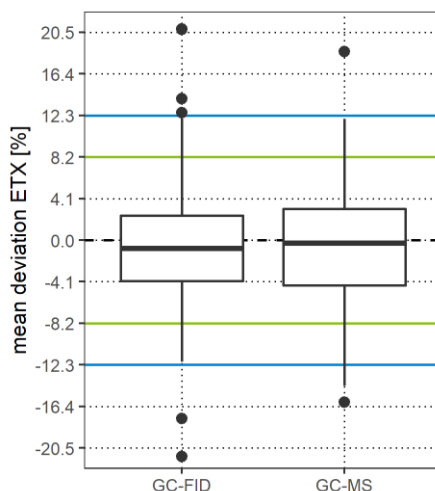
For the desorption of the solvents ethylbenzene, toluene and xylene (ETX) the participants can choose between other solvents or solvent mixtures besides the usual solvent carbon disulphide (CS₂). The majority of the participants reported that they had worked with CS₂. The average results of all participants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

In both the figure and the table, the deviations of the participants' measured values from the assigned value were summarised for all three components (ethylbenzene, toluene, xylenes).

Table 14: Correlation of ETX measurement results with the desorption solvent

solvent used in desorption	CS ₂	other solvent
75 th percentile	2.99%	4.16%
median	-0.62%	0.52%
25 th percentile	-4.46%	-2.20%
number of values	177	25

7.8 Gas Chromatography Detectors



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

Table 15: Correlation of ETX measurement results with analytical instruments

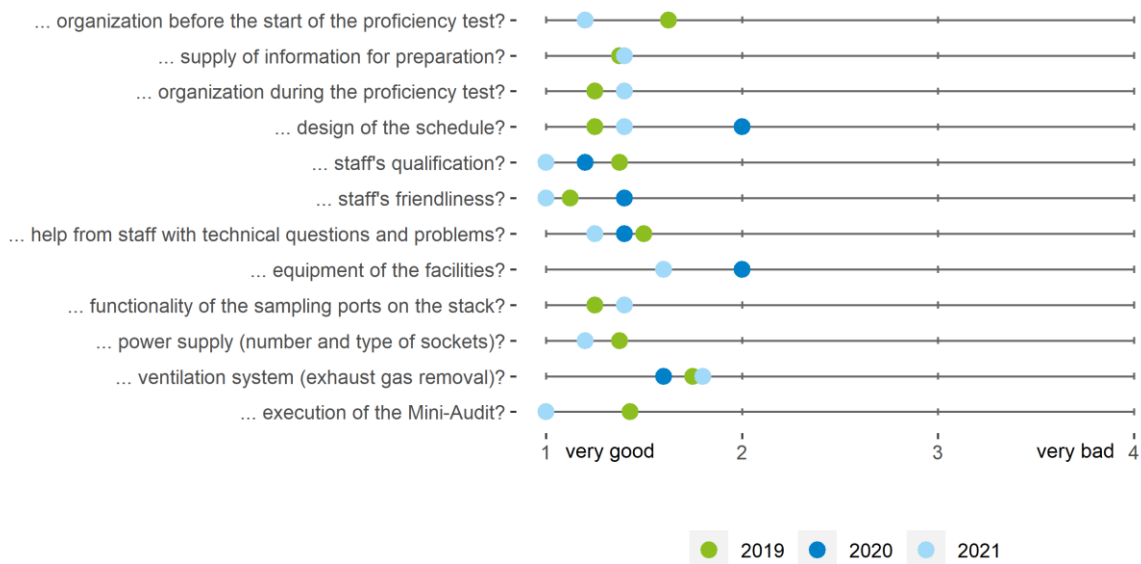
analytical instrument	GC-FID	GC-MS
75 th percentile	2.43%	3.09%
median	-0.80%	-0.29%
25 th percentile	-4.01%	-4.45%
number of values	81	121

For the overall sampling and analytical procedure, the participants achieved comparable results close to the target value with both detector variants. Again, the deviations of the participants' measured values from the assigned value for all three components (ethylbenzene, toluene, xylene) were summarised in the figure and in the table respectively.

7.9 Feedback from Participants

Since 2019 the 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) and 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, last year there were only 5 feedbacks from 3 different measuring institutes. However, these few feedbacks received also showed a high level of satisfaction of the participants with the proficiency testing scheme in 2021. The participants were particularly satisfied with the qualification and friendliness of the staff (average grade: 1.0). The only feedback on the execution of the mini-audit was also a 1.0, but no mini-audit took place in the “pandemic version” proficiency test in question, which is why this rating is difficult to interpret. The worst ratings in comparison were for the ventilation system (mean score: 1.8) and the equipment of the facilities (mean score: 1.6). One participant criticised the lack of cleanliness in the common room and the lack of WLAN. The fact that the common room was left in poor condition was apparently an isolated incident; moreover, this problem will probably resolve itself with the return to regular proficiency tests. WLAN access for proficiency test participants was finally installed at the end of 2021 and is now available to all participants free of charge. The same participant was very positive about the concept of the “pandemic version” and would like to see this version as the future “normal version” for dust and gas proficiency tests. In view of the procedure for establishing the specifications for our proficiency tests, this wish will not be realised in the near future. Moreover, during the last revision of the specifications in 2019, the reduction of the number of measurements per component from 9 to 6 already failed due to the resistance of the stakeholders of the measuring institutes. However, with the experience gained from the 3 measurements in the pandemic version, a compromise might be possible here in the next revision.

8. Concluding Remark

Due to the ongoing SARS-CoV-2 pandemic, the stack emission proficiency tests of the HLNUG could only be offered to a limited extent in 2021. It was possible to carry out the odour proficiency tests in autumn to the usual extent with appropriate hygiene measures, but the programme had to be changed to a “pandemic version” for the dust and gas proficiency tests. For this purpose, the number of measurements per component was reduced to 3 and the component formaldehyde was temporarily removed from the programme. The measurements of both proficiency test programmes could thus be carried out within one day, with a dust proficiency test in the morning and a gas proficiency test in the afternoon, with all components of the proficiency test being measured simultaneously in each case. This radical shortening of the programme made it possible to provide each participant with one of the two measuring rooms for their sole use.

A direct comparison of the results obtained by the participants in 2021 with the results from 2019 (the last year with a comparably large number of participants) shows that the relative deviations of the measured values from the target values were larger on average for practically all dust and gas components, and there were also a much larger number of extreme “outliers”. The reduction in the number of measurements 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. In any case, an evaluation of the results using the previous evaluation criteria shows that the poor overall picture is not an effect of the new criteria, but rather a consequence of the objectively worse measured values in 2021.

The proficiency tests will continue to be carried out in the pandemic version at least in the first half of 2022. With the additional data obtained in this way, it should be possible to make a statement in the next annual report as to whether the deterioration in the measurement results observed in 2021 actually represents a significant change or was only a temporary phenomenon.

Kassel, 12th May 2022

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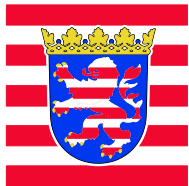
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