

Annual Report 2019

Results of stack emission proficiency tests for substance ranges P, G, and O on the emission simulation apparatus in the year 2019

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0. About this Report

This report is a translation of „Jahresbericht 2019 – Ergebnisse der Emissionsringversuche der Stoffbereiche P, G und O an der Emissionssimulationsanlage im Jahr 2019“ and was prepared with best care and attention. Nevertheless, the German version of this report shall be taken as authoritative. No guarantee can be given with respect to the English translation.

1. Summary

A total of 45 measuring institutes took part in HLNUG's dust emission proficiency tests (substance range P) in 2019, 37 of which were §29b measuring bodies and 8 volunteers. As in the past, the success rate of the §29b measuring bodies (92%) was significantly higher than that of the volunteers (63%).

A total of 44 measuring institutes took part in the gas emission proficiency tests (substance range G) in 2019, 37 of which were §29b measuring bodies and 7 volunteers. As in previous years, the success rate for the §29b measuring bodies (84%) was higher than that for the volunteers (73%).

A total of 12 measuring bodies took part in the odour emission proficiency tests (substance range O) in 2019, 10 of them on the basis of an authorisation in accordance with §29b BImSchG and 2 voluntarily. Here 60% of the authorised participants were successful and 50% of the volunteers.

The results of the dust emission proficiency tests are comparable with those of previous years. The results of the gas emission proficiency tests have been significantly better again this year after a significant deterioration in 2018. In the odour emission proficiency tests, an unusually large number of participants had difficulties with the component ETX this year, while the results for the component THT could not be evaluated.

2. Introduction

2.1 Legal Background

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

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

thorisation 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 stack 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 at which there are sampling openings for taking samples for emission measurements.

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

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. 5 – 11 m/s, moving a volume of approx. 2200 – 5000 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.

3. Organisational Information

In 2019, 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
19P1	Staub (Stoffbereich P) / dust (substance range P)	04.02.2019	05.02.2019	8
19G1	Gas (Stoffbereich G) / gas (substance range G)	06.02.2019	08.02.2019	8
19P2	Staub (Stoffbereich P) / dust (substance range P)	25.02.2019	26.02.2019	8
19G2	Gas (Stoffbereich G) / gas (substance range G)	27.02.2019	01.03.2019	8
19P3	Staub (Stoffbereich P) / dust (substance range P)	11.03.2019	12.03.2019	8
19G3	Gas (Stoffbereich G) / gas (substance range G)	13.03.2019	15.03.2019	8
19P4	Staub (Stoffbereich P) / dust (substance range P)	08.04.2019	09.04.2019	8
19G4	Gas (Stoffbereich G) / gas (substance range G)	10.04.2019	12.04.2019	8
19O1	Geruch (Stoffbereich O) / odour (substance range O)	24.10.2019	24.10.2019	7
19O2	Geruch (Stoffbereich O) / odour (substance range O)	29.10.2019	29.10.2019	5
19P5	Staub (Stoffbereich P) / dust (substance range P)	04.11.2019	05.11.2019	6
19G5	Gas (Stoffbereich G) / gas (substance range G)	06.11.2019	08.11.2019	5
19P6	Staub (Stoffbereich P) / dust (substance range P)	25.11.2019	26.11.2019	7
19G6	Gas (Stoffbereich G) / gas (substance range G)	27.11.2019	29.11.2019	7

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

	substance range P	substance range G
duration of each sampling	30 min	30 min (discontinuous samplings and TOC), 15 min (cont. measurements C ₃ H ₈ , CO, NO _x , SO ₂)
number of samplings	for each component 10, including introductory measurement	
sampling	simultaneously for all participants (1 st and 3 rd floor)	
basic conditions	not detailed in specifications	2000 ... 3500 m ³ /h 20 ... 40 °C

substance range P		substance range G	
concentrations	concentration of total dust: 1 ... 12 mg/m ³ in the following ranges: 1: 1 ... 4 mg/m ³ 2: 4 ... 7 mg/m ³ 3: 7 ... 12 mg/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 sum ETX: 4 ... 100 mg/m ³ formaldehyde: 4 ... 40 mg/m ³ TOC: 4 ... 100 mg/m ³ (ETX/propane) TOC: 5 ... 100 mg/m ³ (propane only)	
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 normal conditions (dry) and with one digit after decimal point.	within four weeks after the end of the proficiency test, in mg/m ³ relating to normal conditions (dry) and with one (components G1-3, G8, G9, see table 5) or two (G4-7 and G10, see table 5) digits after decimal point.	
submission procedure	results are entered into an Excel-file provided by HLNUG and handed in via e-mail.		
substance range O			
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 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
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Tel.: +49 – 561 – 2000 137
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 E-Mail: pt@hlnug.hessen.de

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

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

4. Execution of the Proficiency Tests

4.1 Description of the Test Objects

In contrast to proficiency tests by other providers, HLNUG's stack emission proficiency tests take place at an emission simulation apparatus and include sampling. 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 subject to interpretation for the stack emission proficiency tests at the ESA (4). Extensive investigations have shown that the standard deviations between the samples for the sampling points or measurement cross sections assigned to the participants reach the following maximum values:

Table 3: Maximum values of between samples standard deviations

variable	determined at	relative standard deviation between samples [%]
mass concentration of total dust and heavy metals	all available measurement planes (grid measurements)	1,58
mass concentrations of gases	lowest available measurement plane (point measurements)	0,15
mass concentrations of evaporated liquids	lowest available measurement plane (point measurements)	0,16

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

4.2 Preparation of the Test Objects

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

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

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

4.3 Metrological Traceability

The gaseous substances CO, NO, and propane are dosed using Coriolis flow sensors. The mass flows are measured and gravimetrically traced via suitable test weights and scales. 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 scales 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 basic 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	day of the test	component	compulsory measurement procedure
P	day 1 and 2	dust	(DIN) EN 13284-1 (10) / VDI 2066 Part 1 (11)
		heavy metals	not specified
G	day 1	formaldehyde	VDI 3862 part 2 (12), part 3 (13) or part 4 (14)
	day 2	SO ₂	(DIN) EN 14791 (15)
		TOC	(DIN) EN 12619 (16)
		ETX	(DIN) CEN/TS 13649 (17)
	day 3	SO ₂	using a suitability tested device
		TOC	(DIN) EN 12619 (16)
NO _x as NO ₂		(DIN) EN 14792 (18)	
		CO	(DIN) EN 15058 (19)
O	day 1	four odours	(DIN) EN 13725 (20)

4.5 Evaluation of the Proficiency Tests

4.5.1 Calculation of z-Scores

Substance Ranges P and G

The evaluation of the proficiency tests 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 criterion for proficiency assessment 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 criterion for proficiency assessment 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 to the standard DIN ISO 13528 (7). If less than nine measurement results for one particular component are available that fulfil the above mentioned criteria, neither a z-score-based evaluation nor a performance rating are possible.

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. Participants are informed about the increase of the precision criterion at the latest when the evaluation is communicated by HLNUG.

For the component THT, no proficiency assessment was carried out in 2019, contrary to the provisions of the LAI specifications for stack emission proficiency tests. The basis for determining odour threshold values from the measured values of previous years is the assumption that the measured values of the participants fluctuate statistically consistently around the value of the odour threshold. This prerequisite does not seem to be given for the component THT for the measured values in 2019, since almost all measured values submitted in the interlaboratory comparisons 1901 and 1902 corresponded to odour threshold values that are more than twice the consensus value for THT from the years 2015 to 2018. Practically all participant values in 2019 were scattered in an unusually narrow range for odour measurements. After intensive checks of the chemicals used and the dosing process, the HLNUG was able to rule out contamination or other changes in the THT used. A conclusive explanation for the distribution of the participants' values could not be found so far. The strict application of the assessment criteria according to the LAI specifications for the component THT would have resulted in only 3 of the 12 participants (25%) passing the proficiency test. Since the results for THT do not show a natural scattering, the associated consequences for the measuring bodies would have been disproportionate from the point of view of HLNUG. Against this background, no assessment was made for the component THT in 2019. In the result reports, only the odour threshold value calculated on the basis of the proficiency tests 1901 and 1902 and the odour concentrations calculated from these were given for the component THT. A calculation of z-scores and the assessment of the measurement results was waived.

The other components are not affected by this phenomenon, here the measured values of the participants showed the usual fluctuations around the average odour threshold values of the past years. The assessment of the participants was therefore based on the measurement results for the remaining three components *n*-butanol, solvent mixture and artificial pigsty.

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	1,66
P2	Cadmium	discontinuous	Cd	8,0	1,91
P3	Cobalt	discontinuous	Co	8,0	2,11
P4	Chromium	discontinuous	Cr	12,0	2,22
P5	Copper	discontinuous	Cu	8,0	2,11
P6	Manganese	discontinuous	Mn	10,0	2,24
P7	Nickel	discontinuous	Ni	8,0	1,93
P8	Lead	discontinuous	Pb	8,0	2,01
P9	Vanadium	discontinuous	V	10,0	2,90
substance range G					
G1	SO ₂	discontinuous	Sd	3,1	1,01
G2	SO ₂	continuous	Sk	3,9	1,01
G3	NO _x as NO ₂	continuous	Nk	3,1	1,01
G4	toluene	discontinuous	Td	5,6	1,01
G5	ethylbenzene	discontinuous	Ed	5,8	1,01
G6	sum of <i>o</i> -, <i>m</i> -, <i>p</i> -xylene	discontinuous	Xd	5,3	1,01
G7	formaldehyde	discontinuous	Fd	3,5	1,17
G8	TOC (propane, ETX)	continuous	Ck	3,3	1,08
G9	TOC (propane)	continuous	Pk	3,3	1,08
G10	CO	continuous	Kk	3,6	1,07
substance range O					
O1	<i>n</i> -butanol	discontinuous	NBU	0,10 [†]	1,01
O2	solvent mixture	discontinuous	ETX	0,11 [†]	7,31
O3	tetrahydrothiophene	discontinuous	THT	-	-
O4	artificial pigsty odour	discontinuous	PIG	0,18 [†]	13,0

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

4.5.3 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 components and overall results proceeds differently for the three substance ranges.

4.5.4 Assessment of Components and Overall Result

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“.

The evaluation of single components as well as the overall assessment differs between substance ranges P and G.

Dust Emission Proficiency Test

A component was determined successfully, if the respective sum of class numbers does not exceed 5. 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 and the sum of absolute z-scores does not exceed 5.2. Successful determinations are labelled “passed”, unsuccessful determinations are labelled “failed”.

The overall result of the proficiency test is “passed”, if the component total dust (no. P1) and at least 5 of the 6 mandatory heavy metal components (no. P2 to P5, no. P7, and no. P8) are rated “passed”, otherwise the overall result is “failed”.

Gas Emission Proficiency Test

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”.

With the publication of the new version of the guideline VDI 4220 (21), the component G2 (SO₂ continuous) became a voluntary component in November 2018. From this point on, the proficiency test was successfully passed if the components no. G1 and G3 to G8 were successfully determined.

The analytical part of the proficiency test, the analysis of a solution of ethylbenzene, toluene and xylene in carbon disulphide, could not be performed this year due to quality problems at the supplier of the standards used. An alternative supplier that meets the quality requirements of HLNUG was not available.

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”.

4.5.5 Communication of the Assessment Result

Communication of the evaluation of the participants’ results by HLNUG follows 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

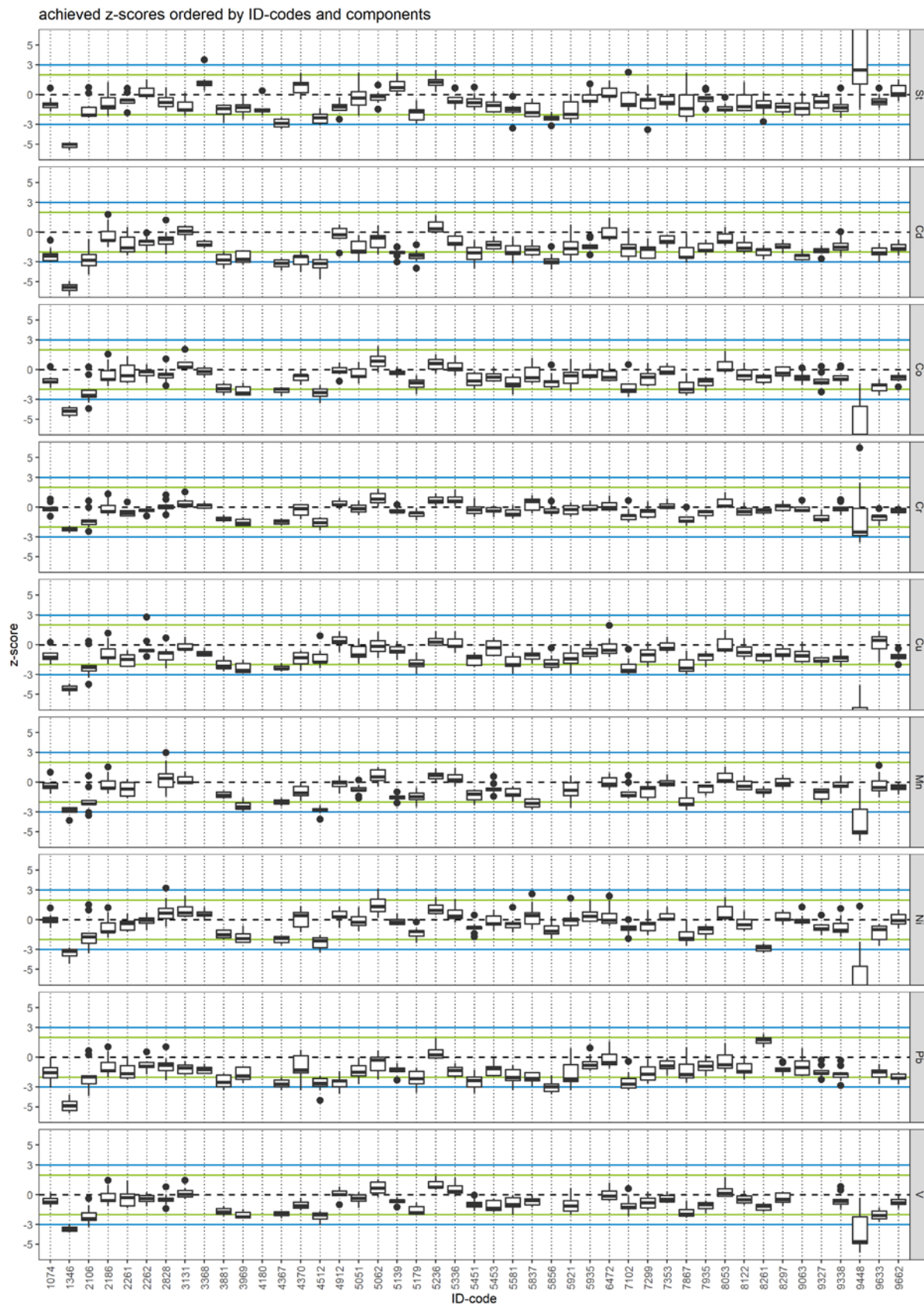
A compact overview of the z-scores achieved by the participants can be found in the following box whisker plots. The rectangle indicates values between the 25th and 75th percentile (interquartile distance), the continuous line in the rectangle indicates the median of the values. The "antennas" reach 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.

As each participant in the olfactometry proficiency test hands in only three measurement values per component, the measurement results are here presented in a different form. Instead of box whisker plots, the results of the odour measurements are displayed as dots, while a horizontal line indicates the mean value of a participant's results.

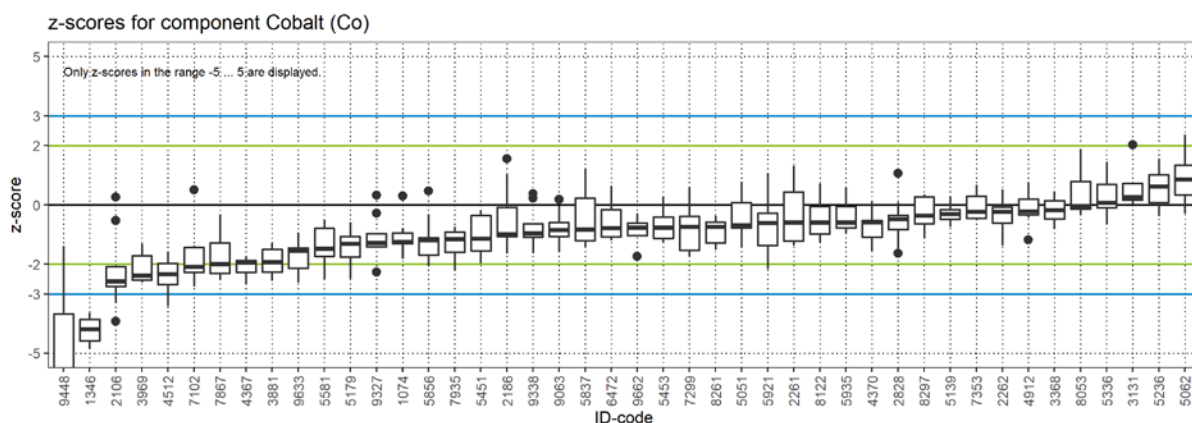
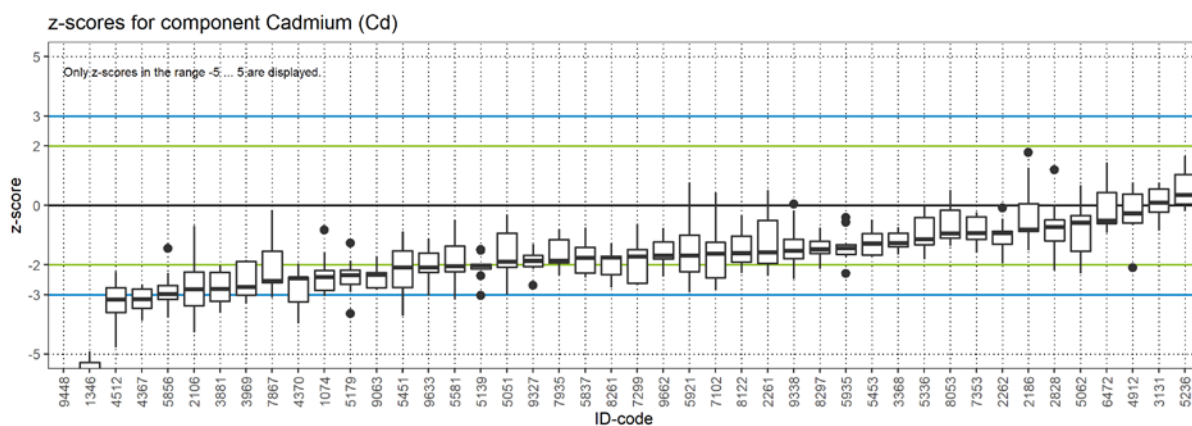
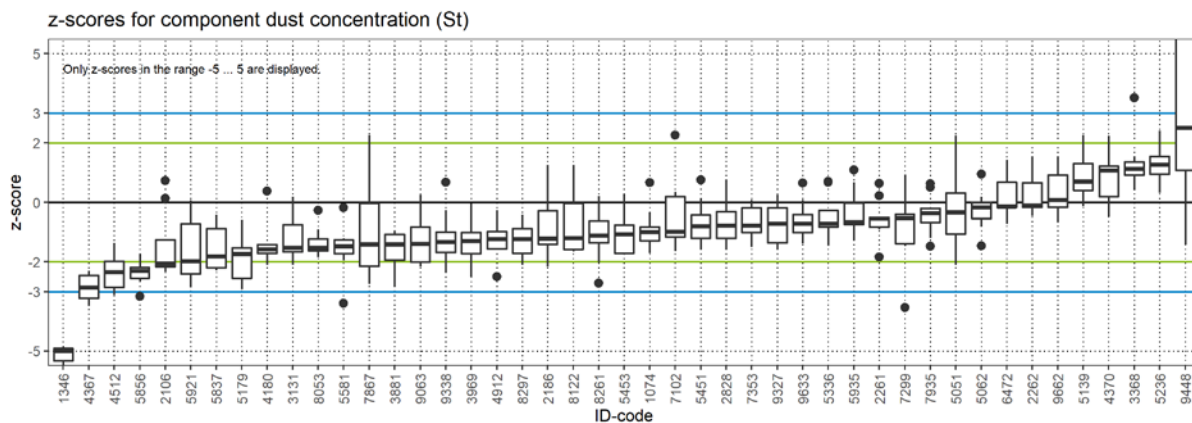
In order to be able to assess the performance of individual participants across all components and to get an impression of the quality of measurements for individual components, the diagrams are available in two different sorts; on the one hand as an overview on one page, on the other hand sorted according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

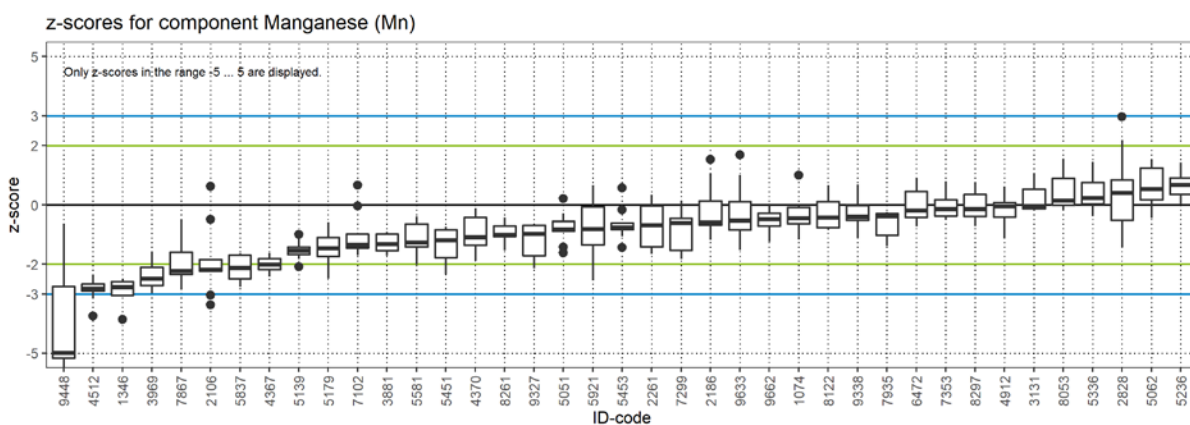
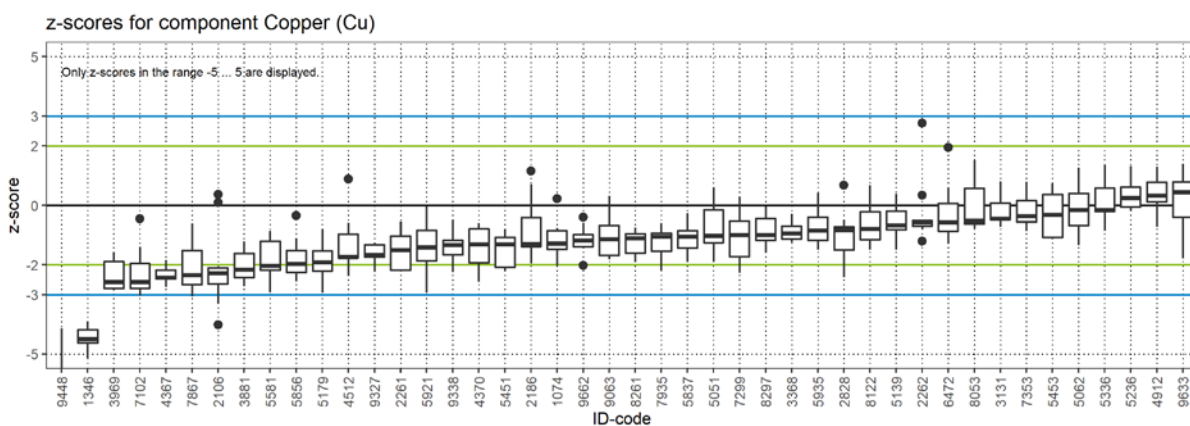
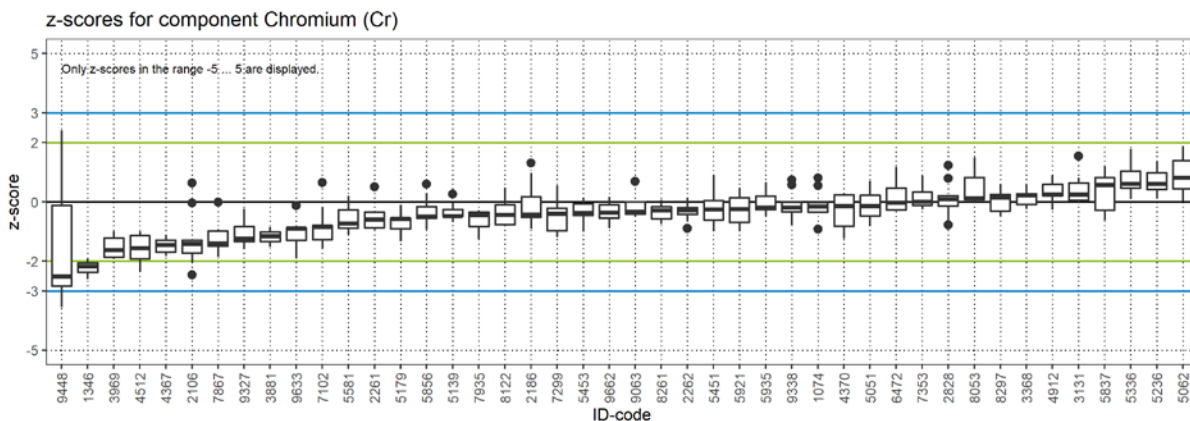
A list of the individual measurements of all participants can be found in a separate document (appendix to the annual report).

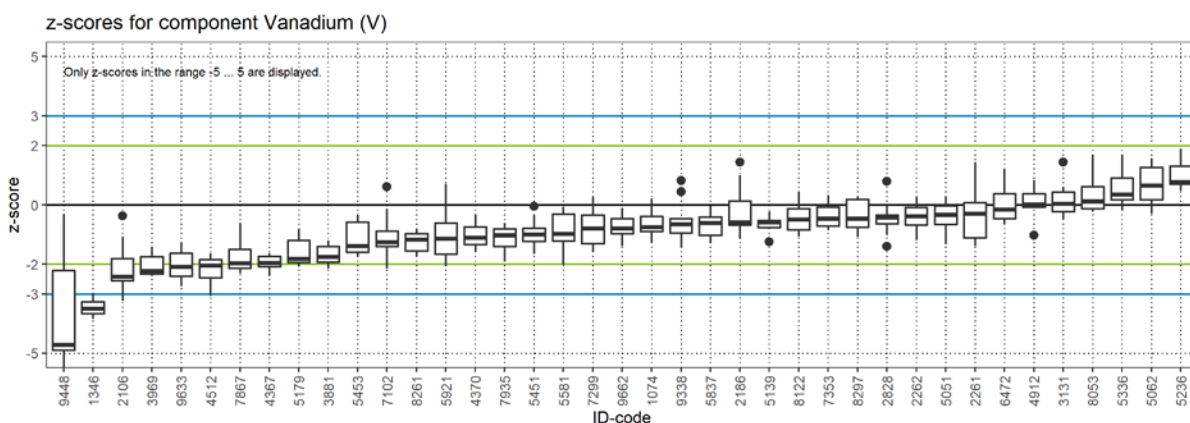
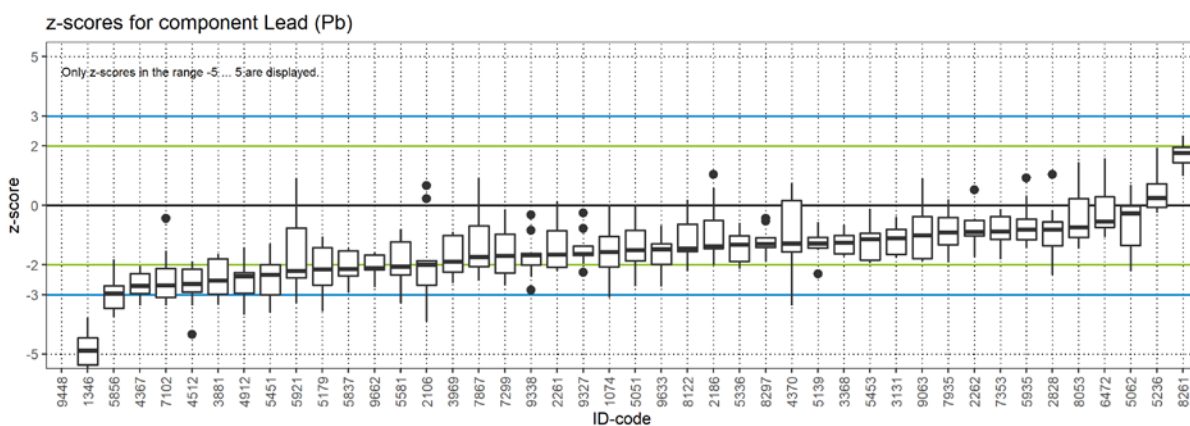
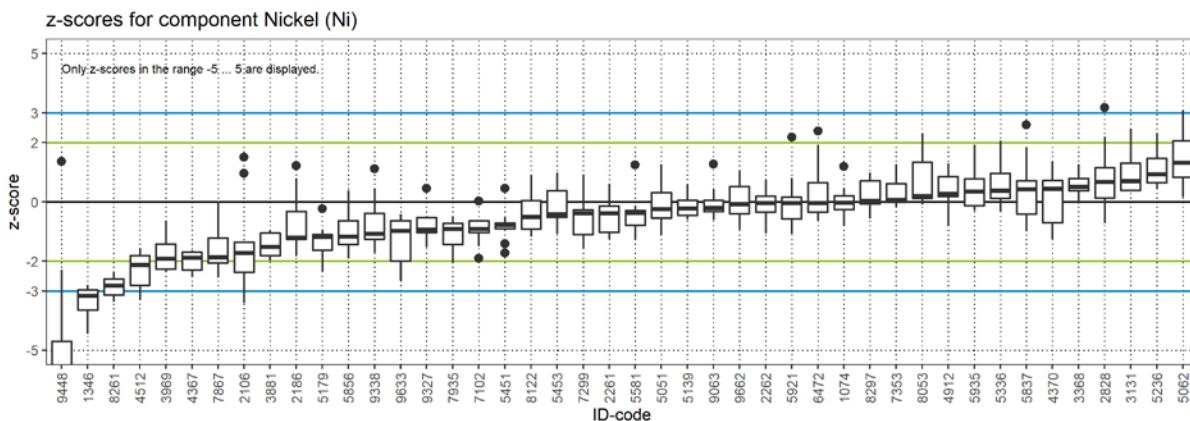
5.1.1 Substance Range P



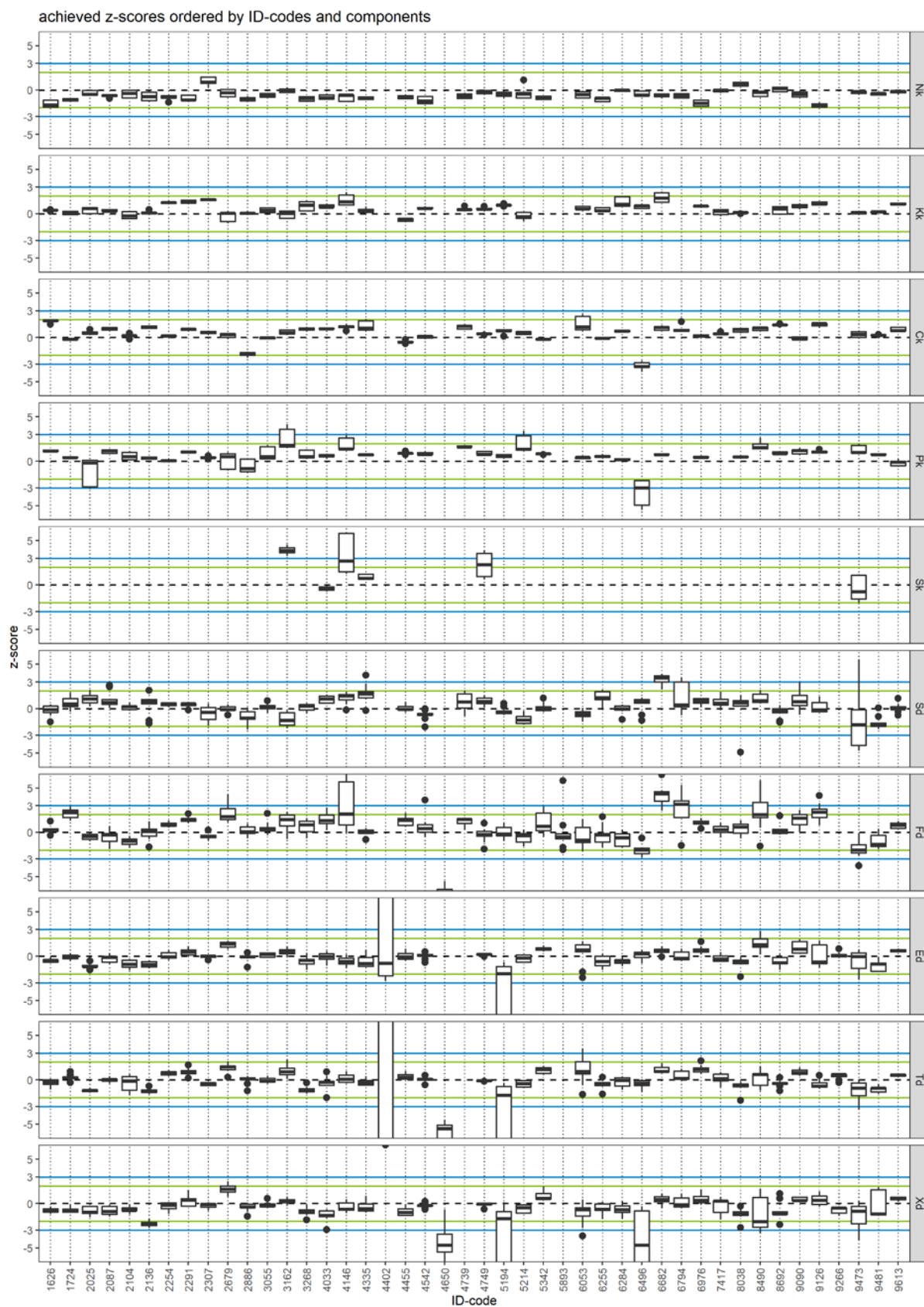
Scheme 2: Achieved z-scores dust proficiency test (only values in the range -5 ... 5 are shown)



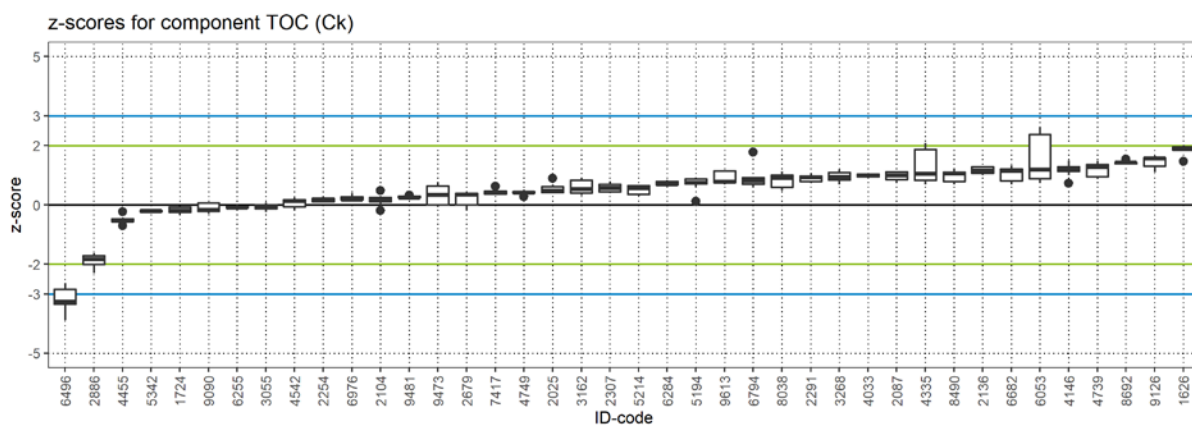
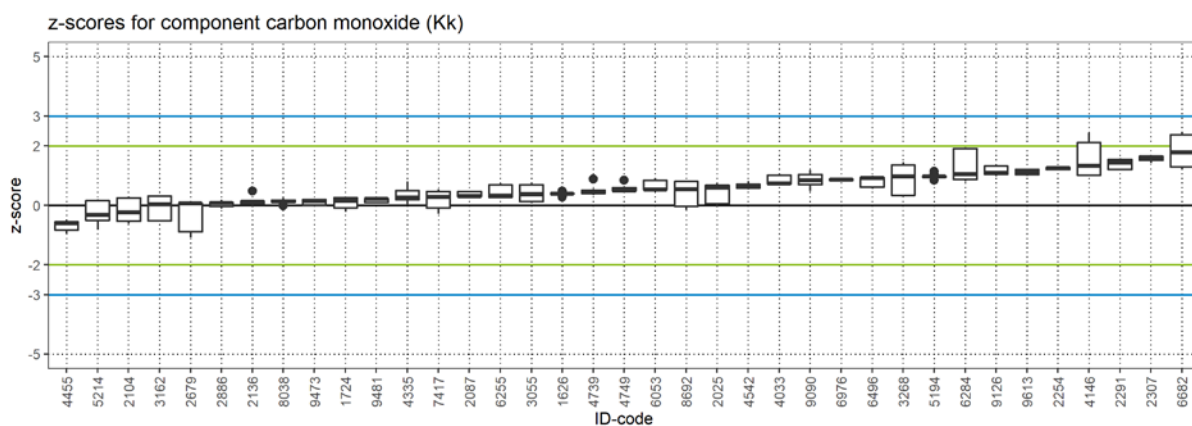
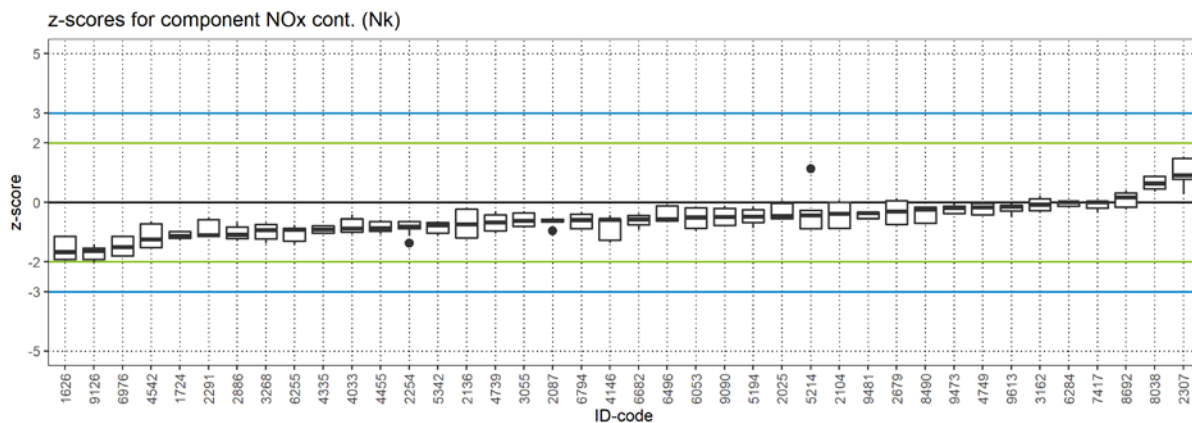


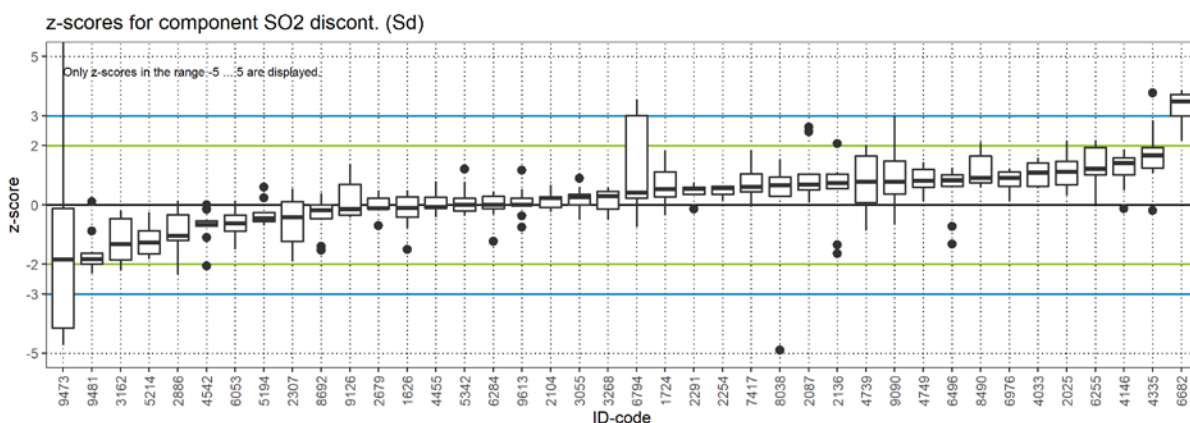
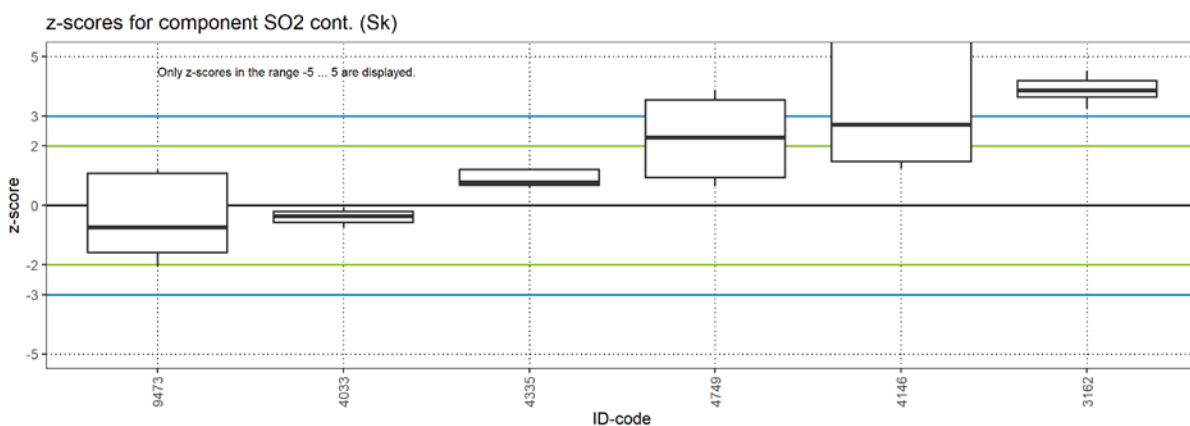
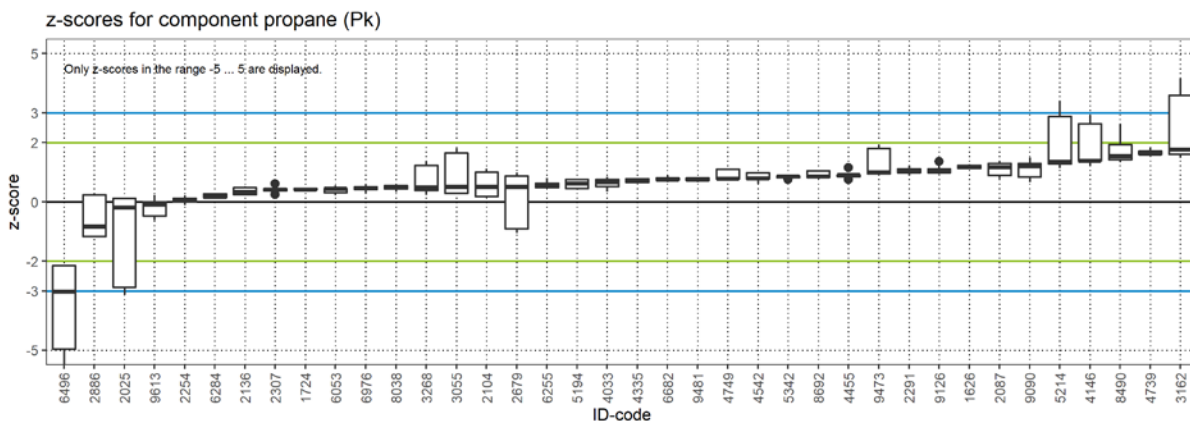


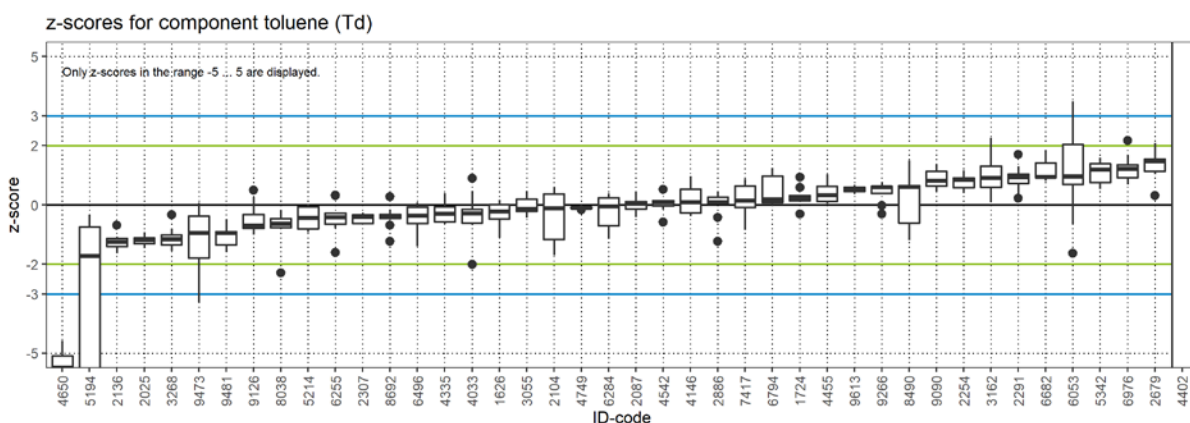
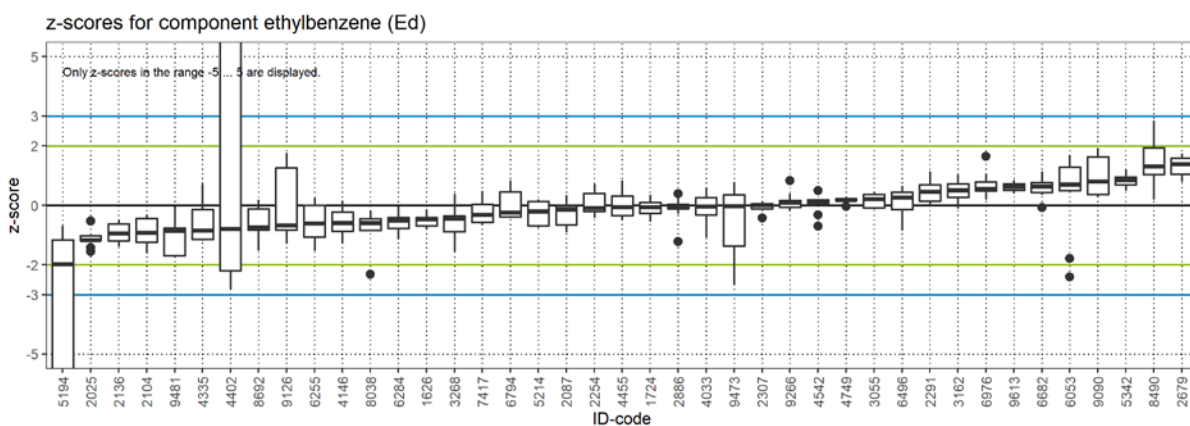
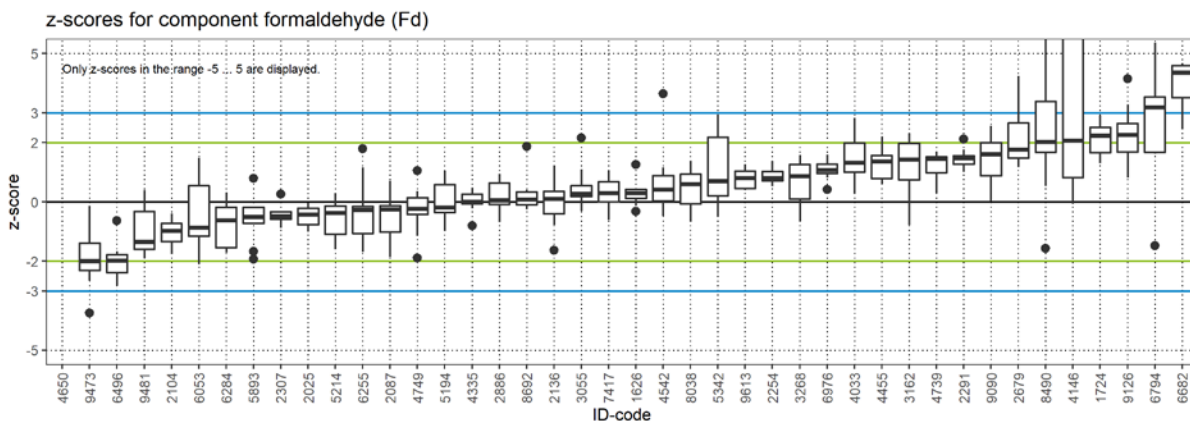
5.1.2 Substance Range G

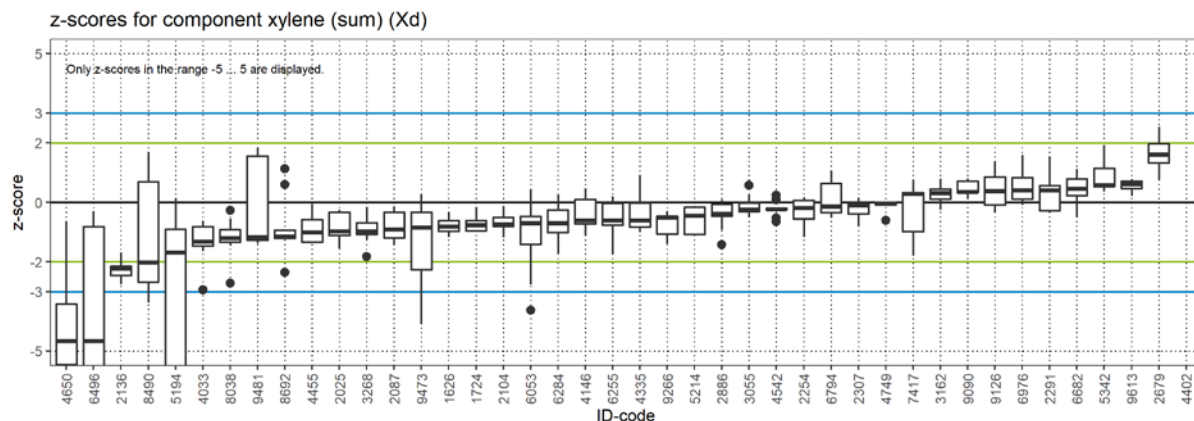


Scheme 3: Achieved z-scores gas proficiency test (only values in the range -5 ... 5 are shown)

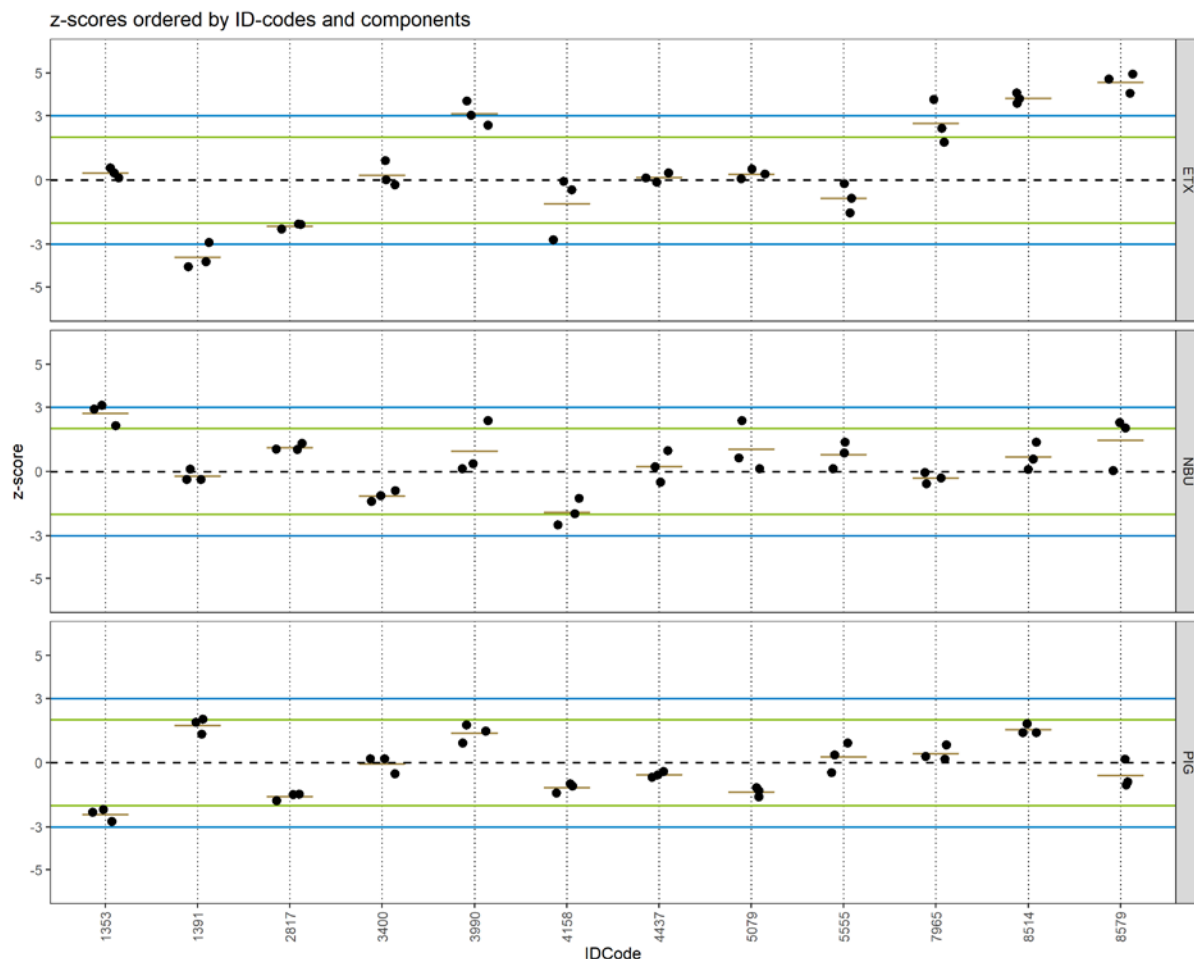


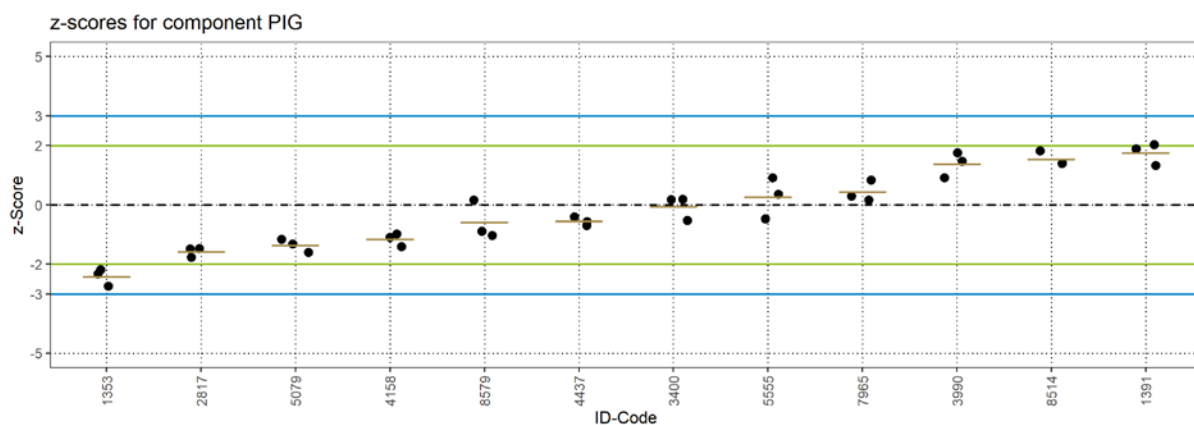
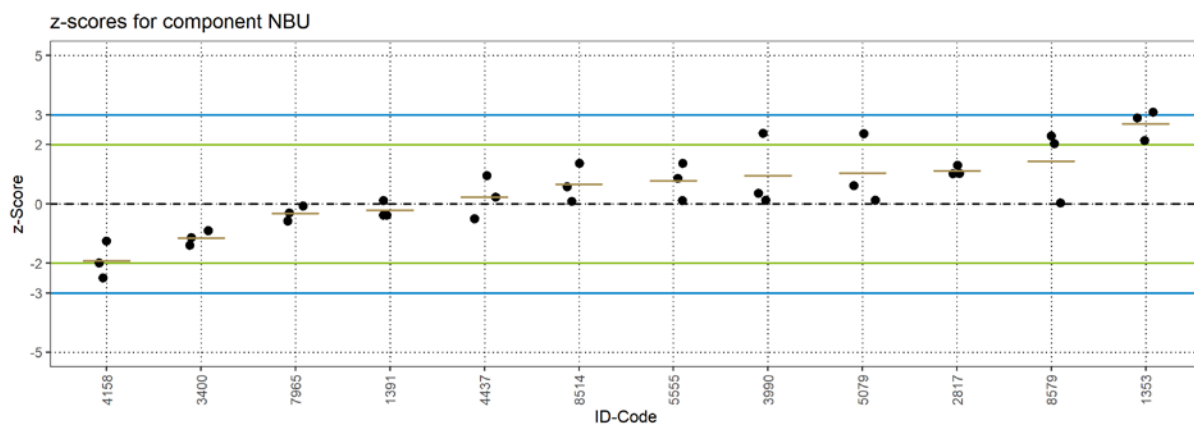
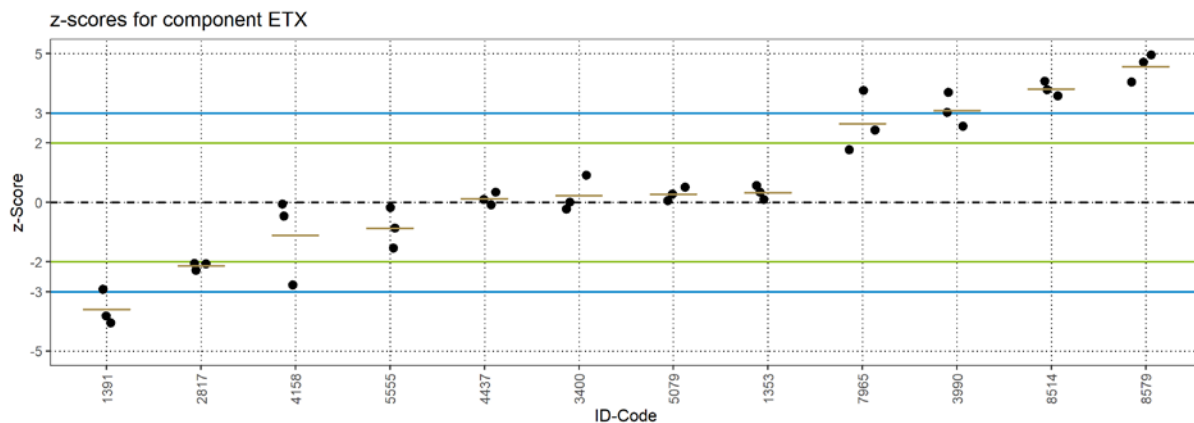






5.1.3 Substance Range 0

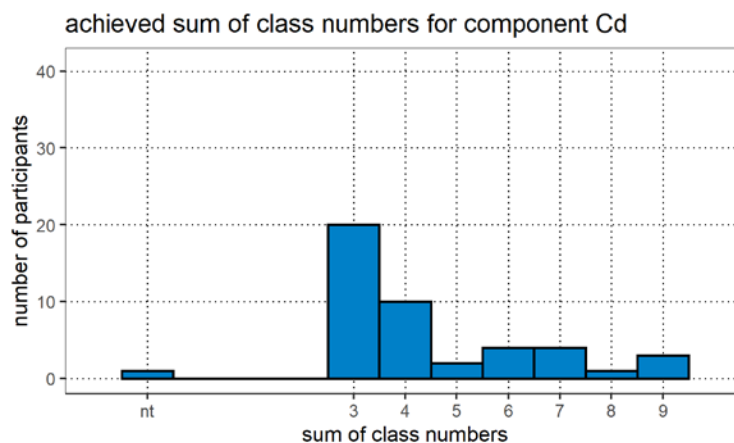
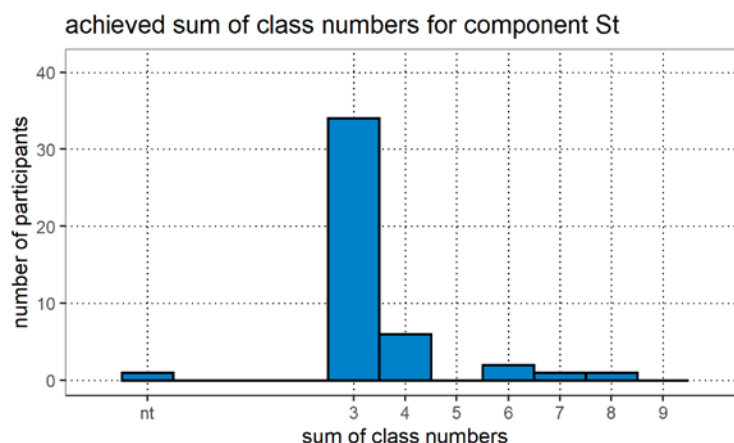


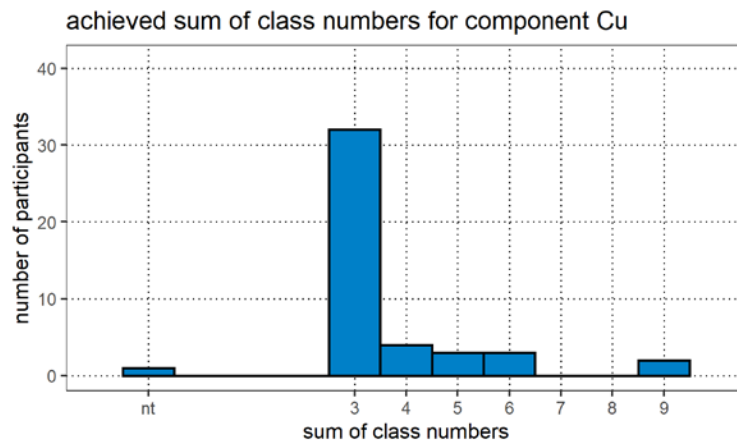
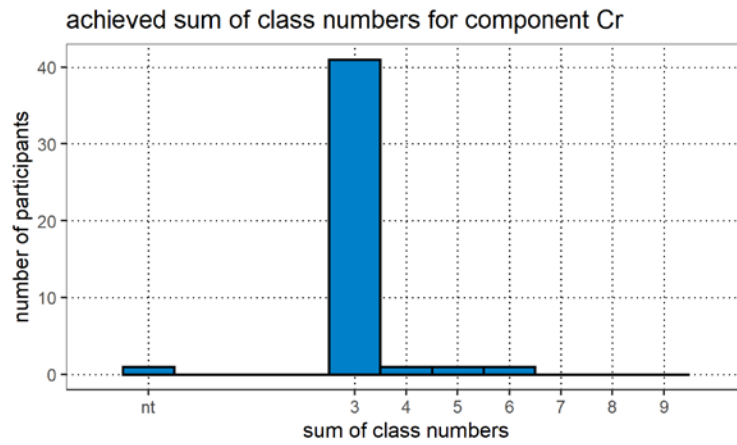
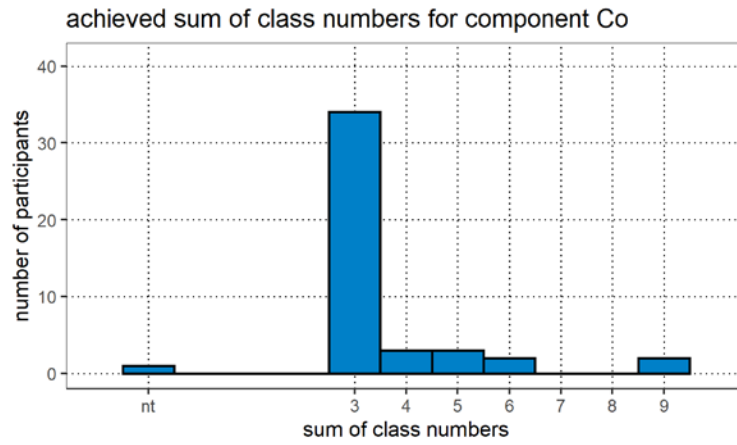


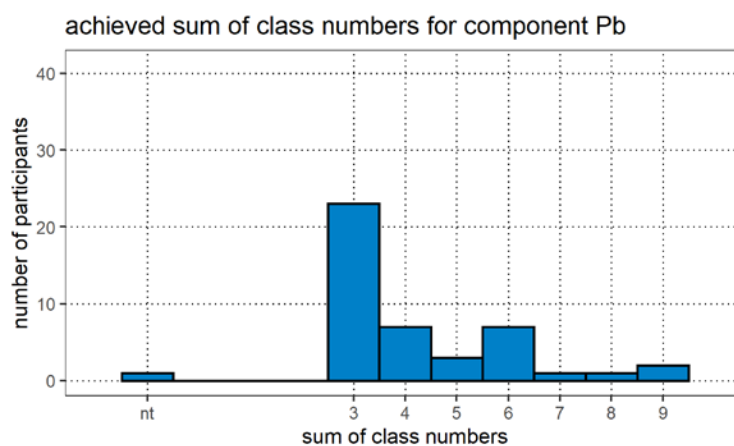
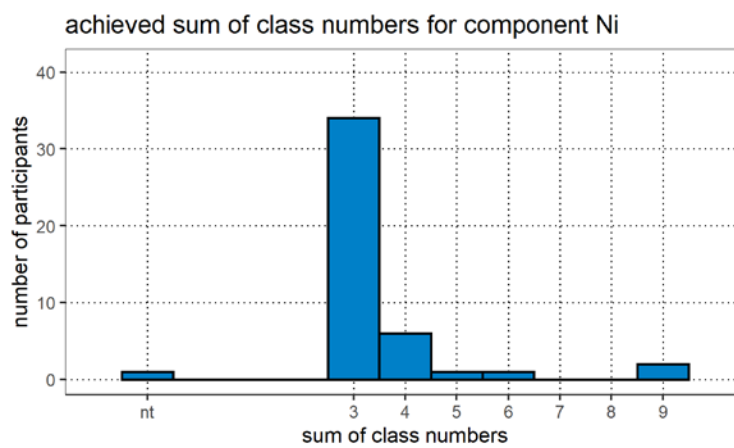
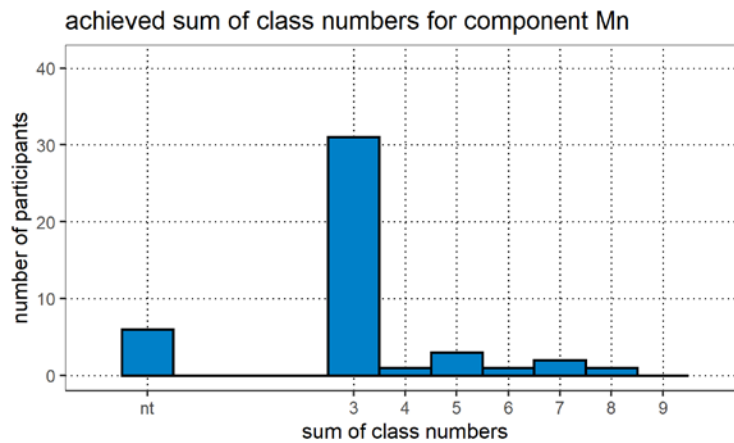
5.2 Sums of Class Numbers

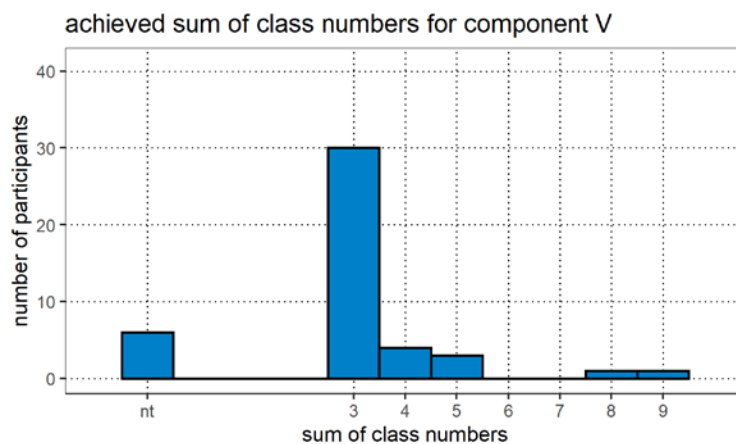
The following schemes show the sum of class numbers that the participants achieved for the different components in form of histogram charts. For the interpretation of the sums of class numbers, please see section 4.5. Participants that did not hand in results for a component are listed as “nt”.

5.2.1 Substance Range P

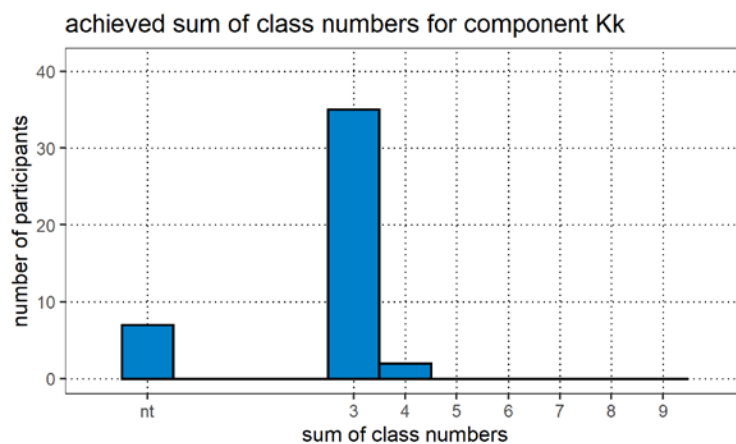
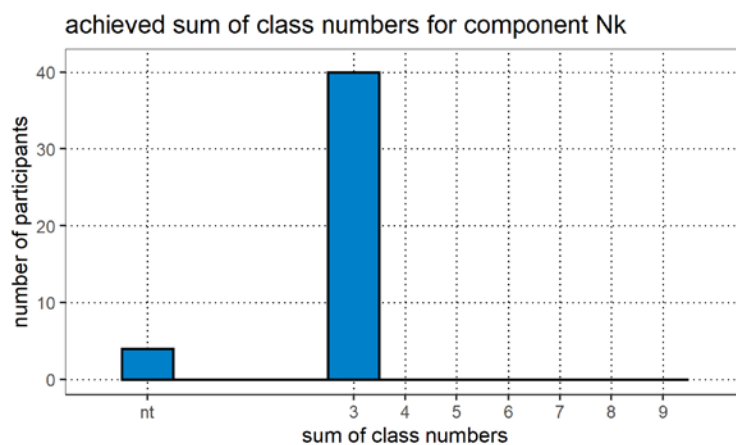


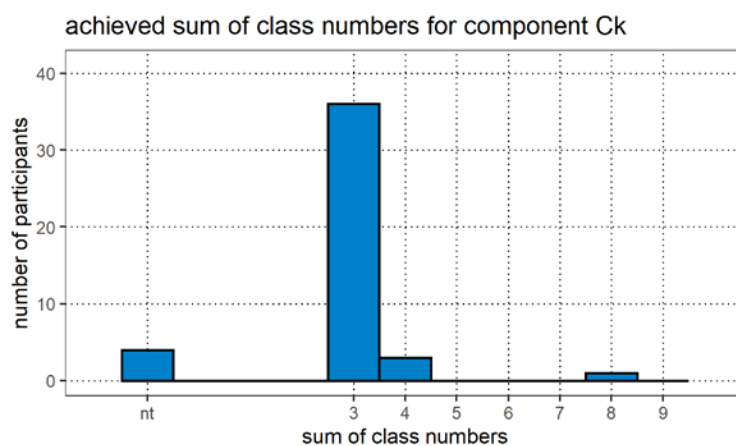
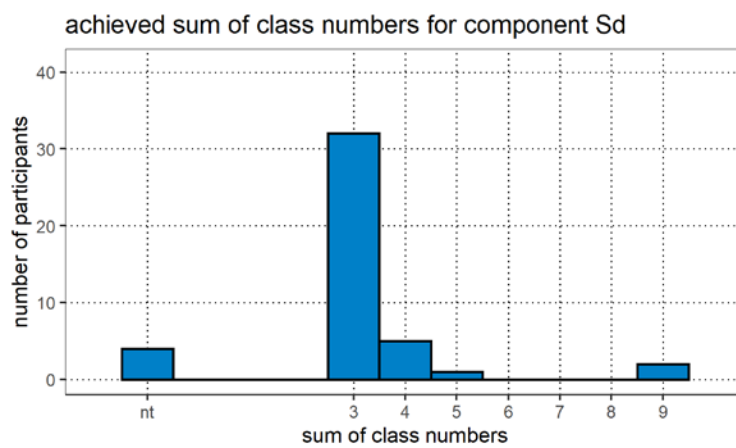
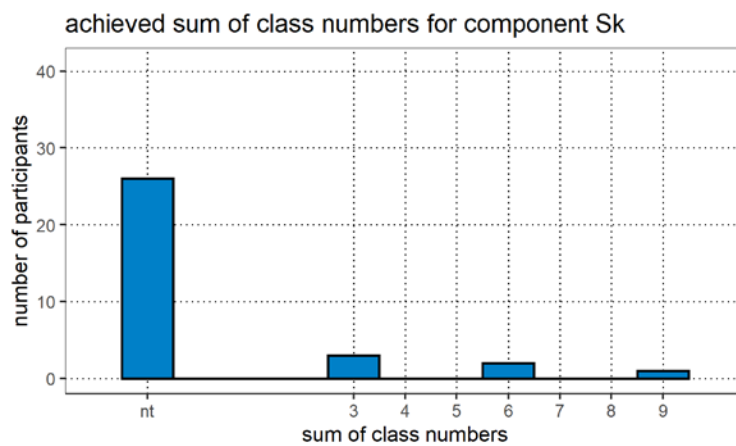


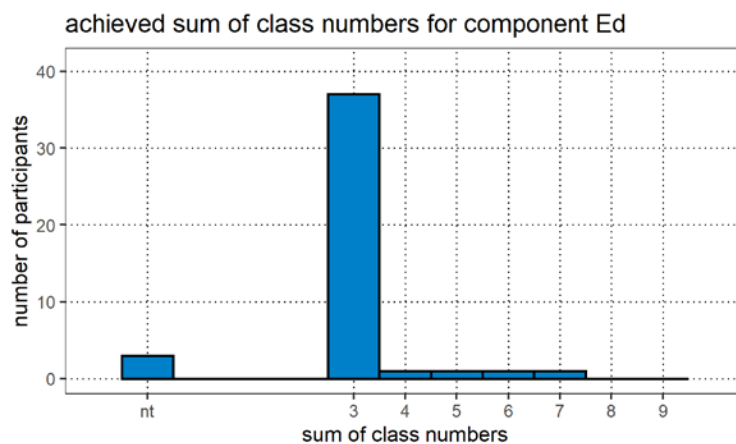
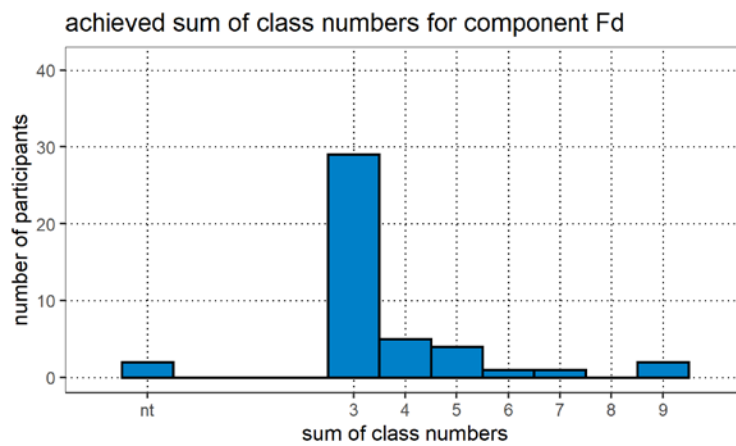
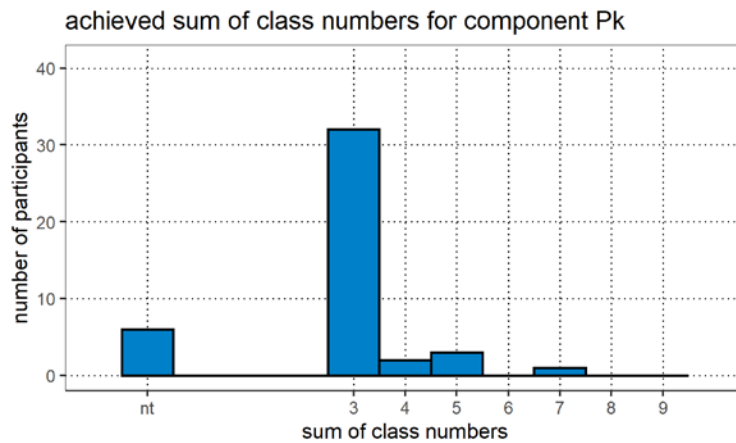


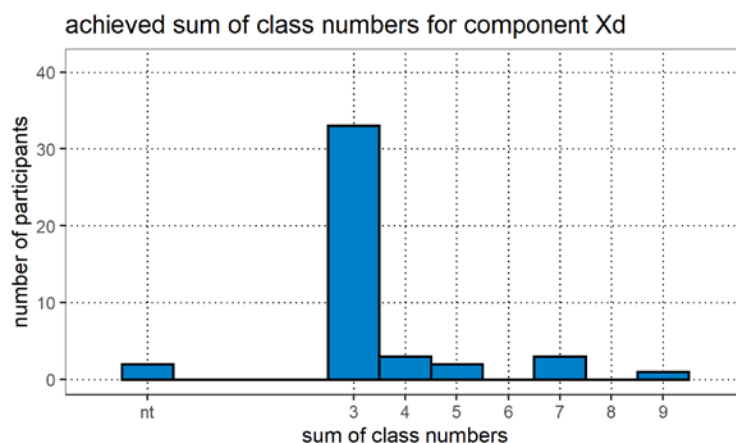
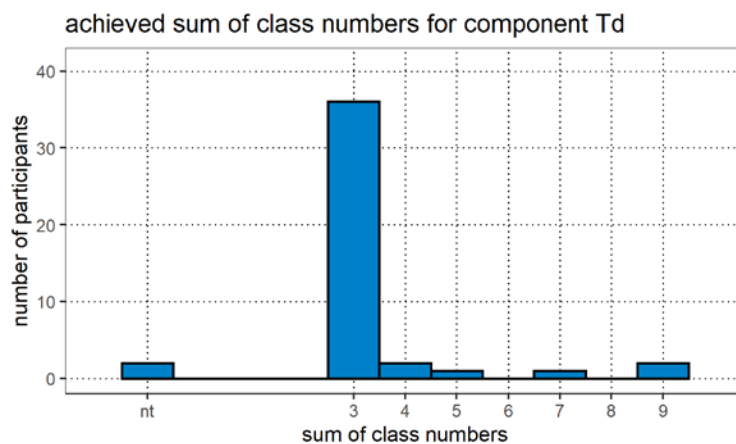


5.2.2 Substance Range G



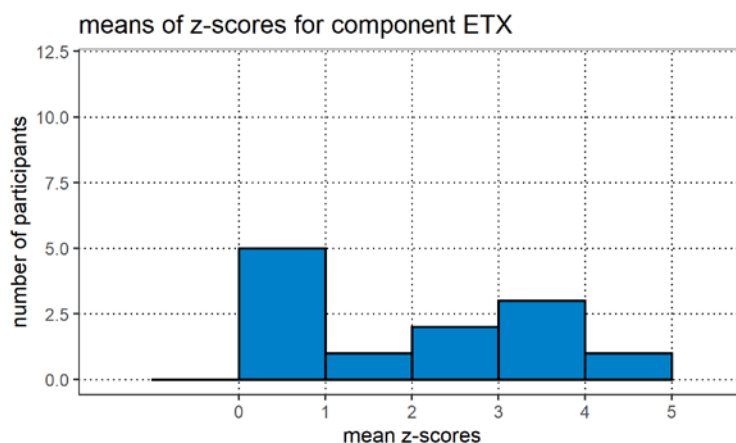


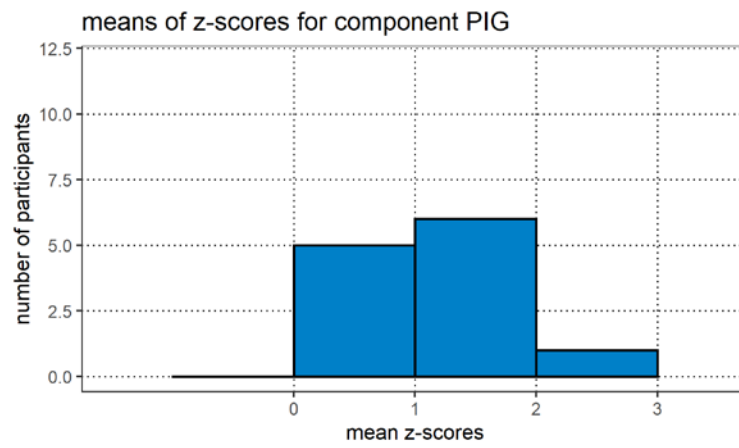
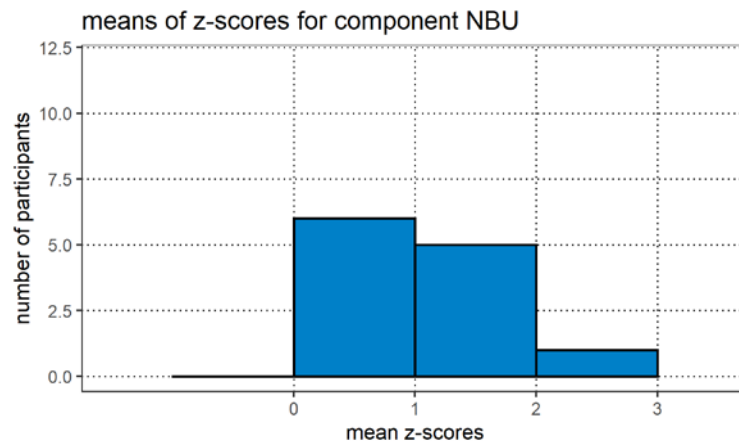




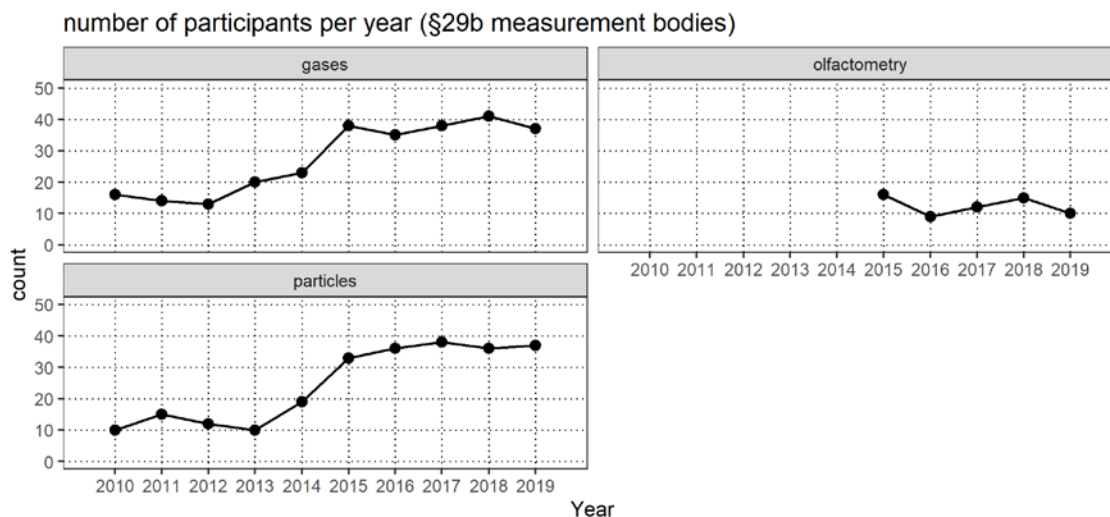
5.2.3 Substance Range 0

In odour emission proficiency tests, instead of sums of class numbers a mean value of z-scores is calculated. In the following schemes, the means of z-scores are displayed as histograms.

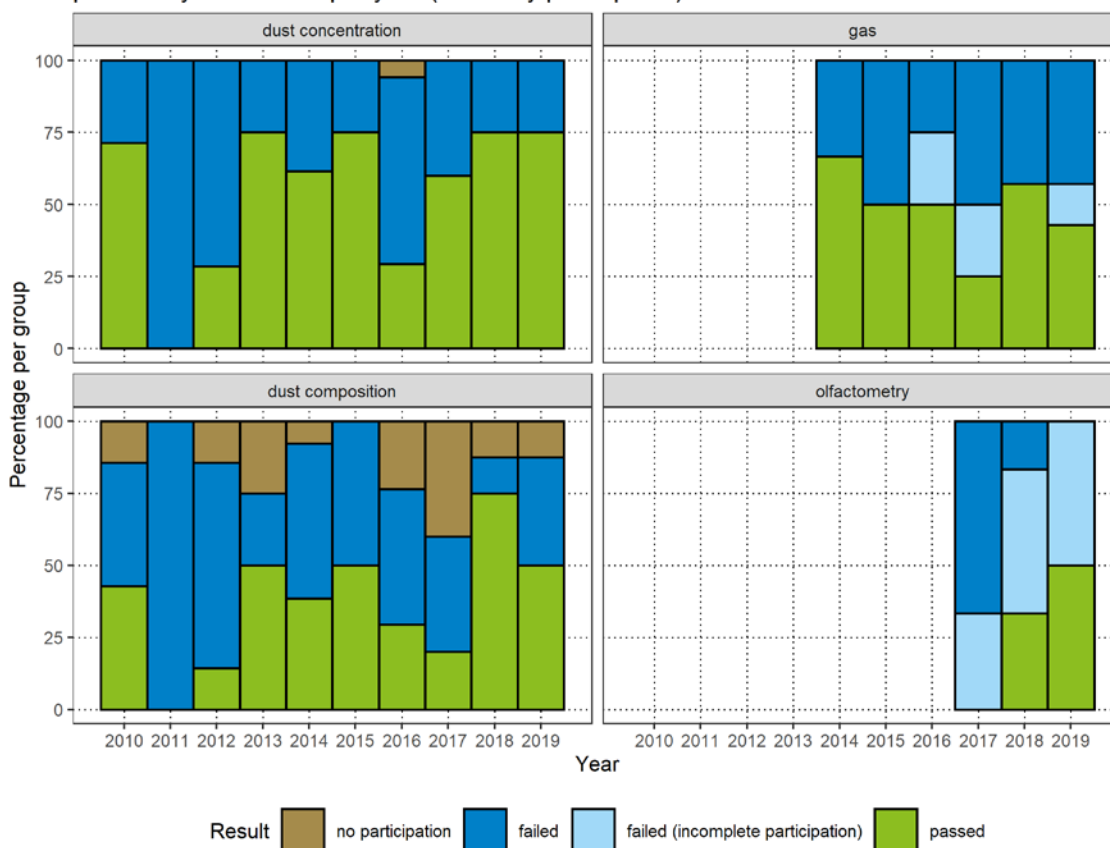




6. Interpretation of Results



proficiency test results per year (voluntary participants)



number of participants per year (voluntary participants)

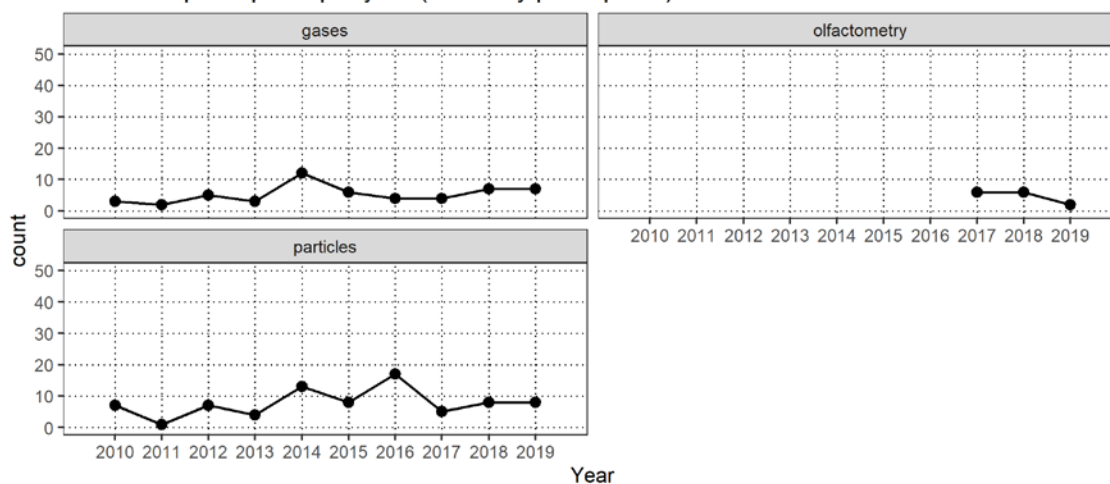


Table 6: Overview of results since 2015 (§29b-bodies)

year	components group	passed	failed	failed (incompl. participation)	no participation
2015	gas	30	6	2	-
	odour	11	5	-	-
	dust (total)	32	1	-	-
	dust composition	26	7	-	-
	dust composition (post-analysis)	6	-	-	-
2016	gas	30	4	1	-
	odour	7	2	-	-
	dust (total)	34	2	-	-
	dust composition	29	7	-	-
	dust composition (post-analysis)	4	1	-	-
2017	gas	35	2	1	-
	odour	10	2	-	-
	dust (total)	37	1	-	-
	dust composition	32	6	-	-
	dust composition (post-analysis)	3	2	-	-
2018	gas	25	10	5	1*
	odour	11	3	1	-
	dust (total)	35	1	-	-
	dust composition	30	6	-	-
	dust composition (post-analysis)	4	1	-	-
2019	gas	31	4	2	-
	odour	6	4	-	-
	dust (total)	34	3	-	-
	dust composition	31	6	-	-
	dust composition (post-analysis)	3	-	-	-

* One participant was absent without excuse at his proficiency test date.

Table 7: Overview since 2015 (voluntary participants)

year	components group	passed	failed	failed (incompl. participation)	no participation
2015	gas	3	3	-	-
	dust (total)	6	2	-	-
	dust composition	4	4	-	-
	dust composition (post-analysis)	2	-	-	-
2016	gas	2	1	1	-
	dust (total)	5	11	-	1
	dust composition	5	8	-	4
	dust composition (post-analysis)	2	-	-	-
2017	gas	1	2	1	-
	odour	-	4	2	-
	dust (total)	3	2	-	-
	dust composition	1	2	-	2
	dust composition (post-analysis)	-	1	-	-

year	components group	passed	failed	failed (incompl. participation)	no participation
2018	gas	4	3	-	-
	odour	2	1	3	-
	dust (total)	6	2	-	-
	dust composition	6	1	-	1
2019	gas	3	3	1	-
	odour	1	-	1	-
	dust (total)	6	2	-	-
	dust composition	4	3	-	1
	dust composition (post-analysis)	1	-	-	-

6.1 §29b Measuring Bodies

The number of proficiency test participations of §29b measuring bodies has approximately doubled between 2013 and 2015 and is now about 30-40 participations for gas and dust per year. Since 2015, the sum of all results of a year is therefore on a much broader statistical basis than in the years prior to 2014. Failures of individual participants therefore no longer distort the overall picture of a year as strongly as in the past.

In the dust proficiency test, the results of the 29b measuring bodies are still at a high level, 34 of 37 participants (92%) were successful in the dust (total) part of the proficiency test. The results for dust seem to stabilize on a high level, 31 of 37 participants (84%) passed this part of the interlaboratory test regularly, another 3 participants (8%) passed this part of the proficiency test only via post-analysis. In total, 92% of the participants in the dust proficiency test were successful.

In the gas proficiency test 31 of 37 (84%) of the §29b measuring bodies passed. After the unusually low rate in 2018, the pass rate has thus risen again to the previously observed level.

6 of 10 participants (60%) passed the odour proficiency test. The pass rate was thus lower than in previous years, although the total number of participants is also comparatively low. The component ETX (mixture of organic solvents) caused particular difficulties for the §29b measuring bodies in the odour proficiency test this year, all 4 unsuccessful measuring bodies had insufficient results for this component. A data analysis of the measurement results showed no abnormalities for the component ETX, unlike for THT. The reason for the bad performance of some participants as well as for the unusual value distribution for THT cannot be finally clarified. It is obvious, however, that the negligently low number of usually 4 test persons used for odour measurements plays a decisive role here, given the uncertainty of individual test person results from a statistical perspective.

6.2 Voluntary Participants

The number of voluntary participations in the proficiency tests varies from year to year, usually there are about 4 participations in the gas proficiency test and about 8 participations in the dust proficiency test. In 2019 there were 7 participants in the gas proficiency test and 8 voluntary participants in the dust proficiency test. Due to the mostly low number of voluntary participations, the collected results of one year are extremely influenced by the performance of individual laboratories in many years, a long-term comparison is only conditionally informative.

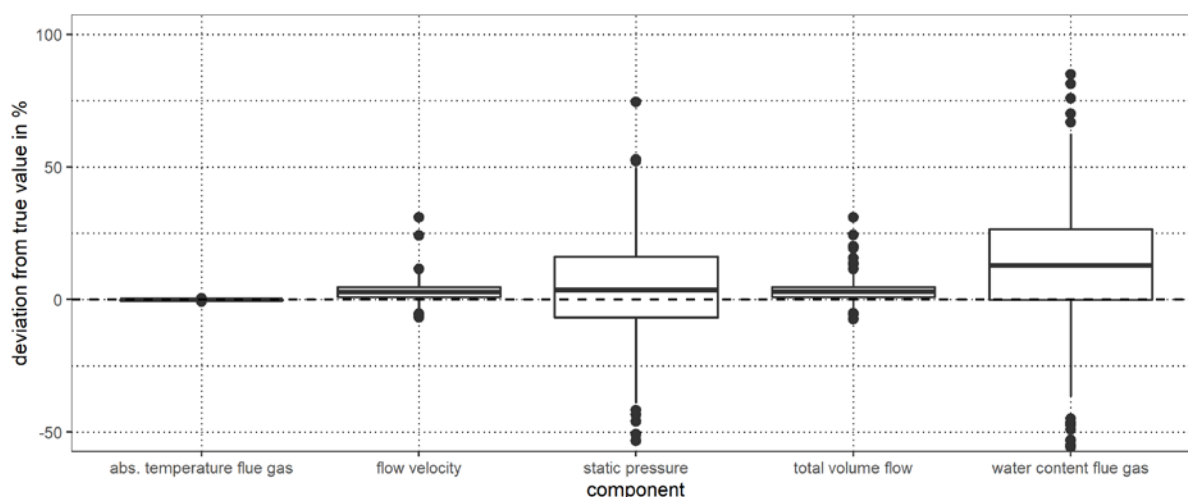
In 2019, a total of 6 out of 8 participations (75%) were successful in the proficiency test on dust (total). In the case of the dust composition, 4 of the 7 participants (57%) passed regularly, another participant (14%) only passed this part of the proficiency test via post-analysis. Thus, a total of 5 (63%) of the 8 voluntary participants in the dust proficiency test were successful. With these figures it should be noted that one participant only determined dust mass concentrations and did not determine heavy metal concentrations.

In the gas proficiency test, 3 of 7 (43%) of the voluntary participants passed the proficiency test. One participant was successful in principle, but did not take part in all mandatory components and therefore received the overall rating "failed (incomplete participation)".

In the odour proficiency tests, one out of two voluntary participants (50%) passed. The other participant (50%) delivered correct measured values, but these were not determined within 6 h according to VDI 3880. The results were therefore assessed as "failed (incomplete participation)".

6.3 Basic Flow Conditions

For each proficiency test, the participants must also determine and specify the basic flow conditions. The measured values of the participants are compared in the result communications with the target values determined by the HLNUG. The data basis for the evaluation in this report are the measured values received from proficiency test participants in 2019. A list of individual results is not given here, in the following scheme only a summary representation of the values is shown. The display is limited to relative deviations of the participant measured values from the respective setpoint value in order to be able to compare different proficiency tests with different flow conditions. Obviously incorrect measured values that deviate from the setpoint by orders of magnitude were removed from the data collective. This applies in particular to information on static pressure. Here, numerical values were often submitted that would be reasonably correct, e.g. in the unit Pa or as total pressure, but not as static pressure in the unit hPa, as asked for in the results submission.



As can be seen, the measured values for the exhaust gas temperature are always very close to the setpoint. In comparison, the measured values for the flow velocity and the volume flow show a significantly higher dispersion and many individual values that are far from the setpoint value.

Even more extremely, the measured values of the participants scatter for the static pressure and the water vapour concentration.

An evaluation of the absolute values of the relative deviations (positive and negative deviations do not cancel each other out here) can be found in the following table. The median of the absolute values of the respective deviations as well as the 25th and 75th percentile are listed there. This information should be understood as follows: Only a quarter of the readings showed a deviation below the 25th percentile. Half of the measurements showed a deviation below or above the median. One quarter of the measured values had a deviation (positive or negative) greater than the 75th percentile.

Table 8: Absolute relative deviations from true values for basic conditions (2019)

	absolute temperature [%]	flow velocity [%]	static pressure [%]	volume flow [%]	water vapour concentration [%]
75 th percentile	0,24	5,23	22,5	5,13	32,8
median	0,13	3,09	10,4	3,24	19,2
25 th percentile	0,08	1,54	5,0	1,71	8,6
number of values	227	227	227	226	222

The deviations from the assigned values were thus for most components relatively close to the results from the years 2016 to 2018. As in the previous year (19%), the deviations for water vapour concentration in 2019 were significantly higher than in 2016 (9%) and 2017 (12%).

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 2019.

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

For most evaluations, a certain stabilisation of the values can be observed. This is ultimately due to the fact that the data basis for the 2019 Annual Report has only increased by about one third,

while the values for most evaluations hardly differ from the values of previous years. As a result, most of the findings become more and more reliable and meaningful over time.

For all correlations presented in this report, it should be kept in mind that a correlation is merely an indication of a connection, but 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 reported an uncertainty of measurement below the 25th percentile. Half of the participants reported an uncertainty of measurement below or above the median. A quarter of the participants reported an uncertainty of measurement greater than the 75th percentile.

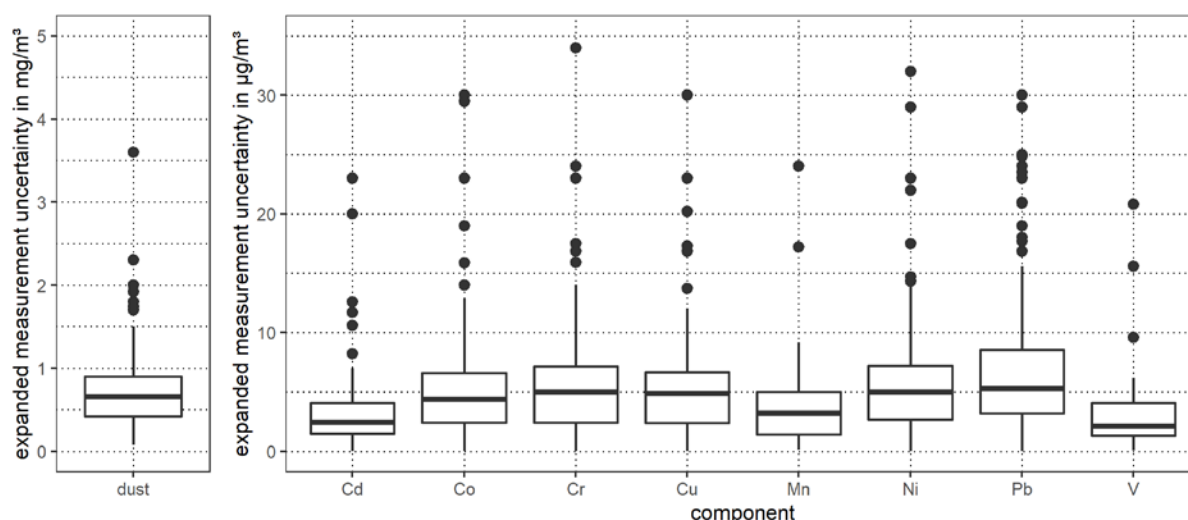


Table 9: Expanded measurement uncertainties reported by participants of the dust proficiency test (2016-2019)

	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	0,90	4,13	6,60	7,60	7,02	5,00	7,42	8,83	4,35
median	0,66	2,46	4,40	5,00	4,90	3,23	5,00	5,41	2,22
25 th percentile	0,42	1,46	2,40	2,40	2,33	1,38	2,65	3,18	1,30
number of values	127	110	111	111	111	73	111	110	70

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.

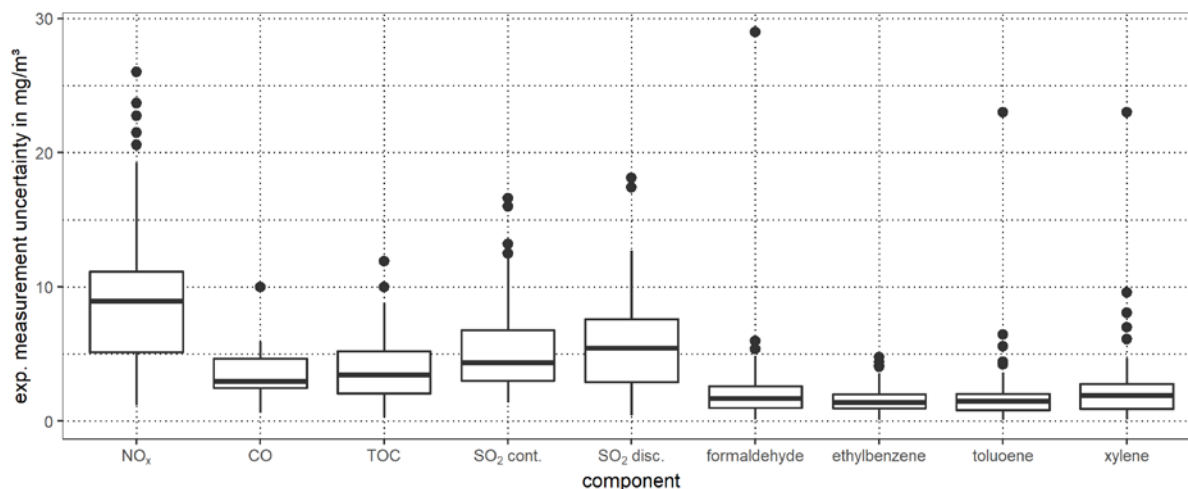


Table 10: Expanded measurement uncertainties reported by participants of the gas proficiency test (2016-2019)

	NO _x as NO ₂ [mg/m ³]	CO [mg/m ³]	TOC [mg/m ³]	SO ₂ (cont.) [mg/m ³]	SO ₂ (disc.) [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	11,13	4,65	5,21	6,79	7,60	2,60	2,04	2,04	2,78
median	8,95	2,99	3,45	4,36	5,42	1,70	1,50	1,50	1,94
25 th percentile	5,15	2,46	2,08	3,00	2,90	1,00	0,80	0,80	0,90
number of values	116	39	112	77	115	113	112	113	113

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. Two participants who stated that they rinse once at the end of the proficiency test were considered for the working day rinsing.

In this report, the data basis is limited to the results between autumn 2018 and the end of 2019. In summer 2018, the enquiry about the rinsing procedure was concretised with regard to frequency, and since then a total of 38 participants provided 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.

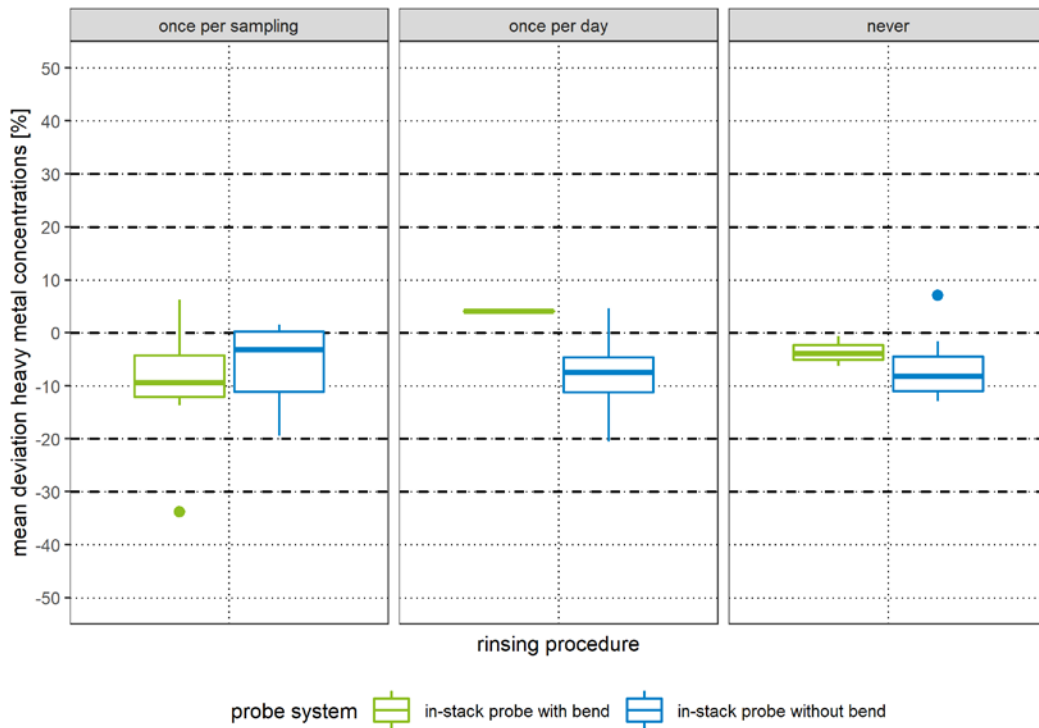
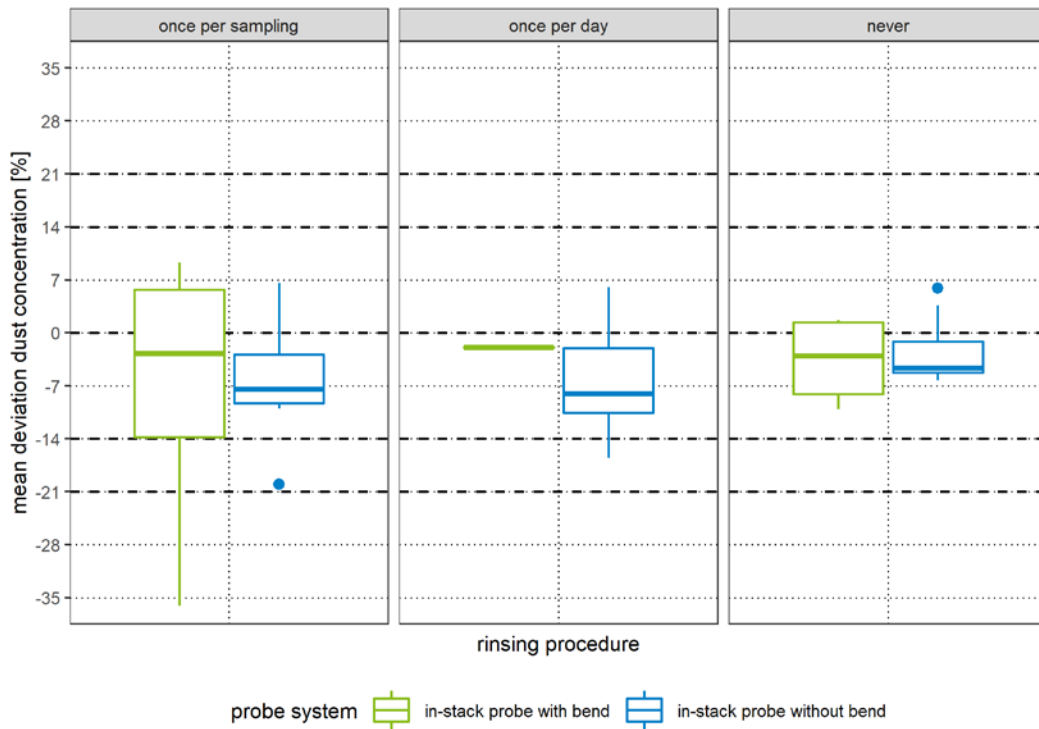


Table 11: Correlation of dust measurement results with probe systems and rinsing procedures (2018-2019)

combination	probe system	rinsing procedure	median of mean deviation total dust results	number of participants	median of mean deviation heavy metal results	number of participants
1	in-stack probe with bend(s)	after each sampling	-2,7%	7	-9,3%	7
2		once per day	-1,9%	1	+4,0%	1
3		no rinsing	-3,1%	4	-3,9%	3
4	in-stack without bend	after each sampling	-7,5%	10	-3,2%	10
5		once per day	-6,8%	8	-7,8%	8
6		no rinsing	-4,7%	8	-8,2%	8

Due to the very small number of cases, the results presented are considerably influenced by various factors of the respective laboratories. The above-average results for combination 3 (probe with bends that is not flushed: right figure, green), for example, are unlikely to be representative of this type of sampling. Combination 3 explicitly does not comply with the standard, since dust deposits on the inner surface of the probe are to be expected during sampling, which can lead to significantly lower results if no rinsing is performed.

7.3 Diameter of the Nozzle Opening in Dust Samplings

The information provided by the participants on the diameter of their probe's nozzle opening does not indicate a clear trend. Irrespective of the diameter, the measured values always seem to spread over a wide range. Overall, however, the size of the nozzle opening does not appear to be a determining factor for the measurement results. Opening diameters stated by less than 5% of all participants are not listed here.

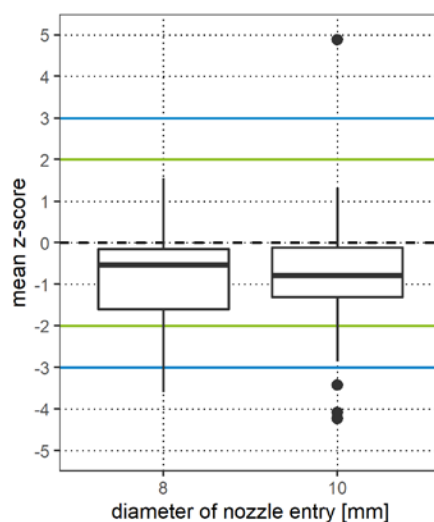


Table 12: Correlation of absolute means of z-scores for total dust with nozzle opening diameters (2016-2019)

diameter of nozzle opening	8 mm	10 mm
75 th percentile (z-score)	-0,15	-0,12
median (z-score)	-0,53	-0,79
25 th percentile (z-score)	-1,60	-1,31
number of values	30	114

7.4 Analytical Instruments for Heavy Metals

The information provided by the participants on the analytical instrument used for heavy metal analysis reveals only small differences between AAS and ICP users. A total of 21 participants stated that heavy metal analysis was performed using AAS equipment, while 133 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. In addition, ICP users seem to be more prone to massive underreporting than users of AAS devices.

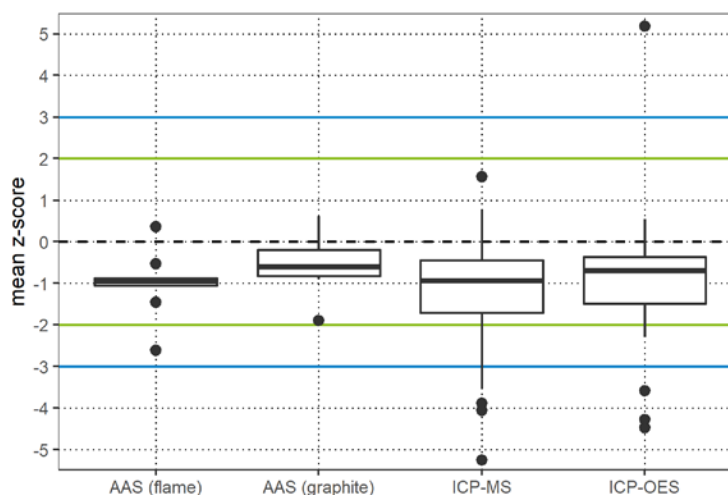


Table 13: Correlation of heavy metal results and analysis devices (2016-2019)

analysis device	flame-AAS	graphite fur- nace AAS	ICP-MS	ICP-OES
75 th percentile (z-score)	-0,88	-0,19	-0,45	-0,37
median (z-score)	-0,95	-0,60	-0,94	-0,69
25 th percentile (z-score)	-1,05	-0,82	-1,72	-1,49
number of values	9	12	81	52

7.5 Formaldehyde

For the measurement of formaldehyde concentrations, participants can choose from the guidelines VDI 3862 Parts 2 (12), 3 (13) and 4 (14). Only the procedures according to Part 2 and Part 4 were stated by more than 5% of the participants and are therefore shown in the following diagram and table. The following picture emerges from the information provided by the participants:

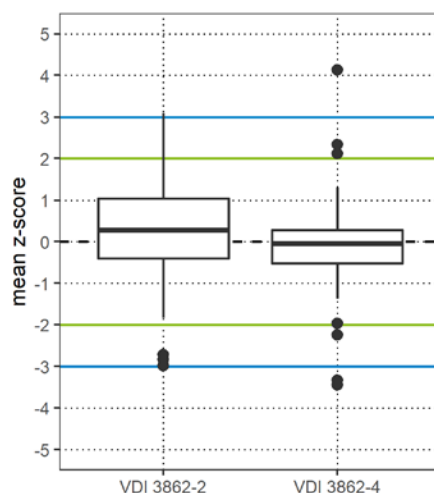


Table 14: Correlation of formaldehyde measurement results with the guidelines used (2016-2019)

guideline	VDI 3862 Part 2 (DNPH wash bottles)	VDI 3862 Part 4 (AHMT-procedure)
75 th percentile (z-score)	1,05	0,28
median (z-score)	0,27	-0,05
25 th percentile (z-score)	-0,40	-0,52
number of values	86	50

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

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:

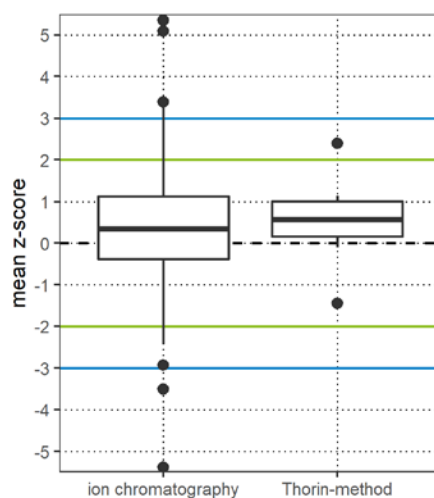


Table 15: Correlation of sulphur dioxide measurement results with the analytical method used (2016-2019)

method used	Ion chromatography	Thorin method
75 th percentile (z-score)	1,12	1,01
median (z-score)	0,34	0,57
25 th percentile (z-score)	-0,38	0,17
number of values	138	12

The available results do not suggest a significant difference between the two methods, but the number of participants using the Thorin method is comparatively small (8% of all participants). The higher dispersion of the IC method with various "outliers" may be due solely to the almost 12 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.

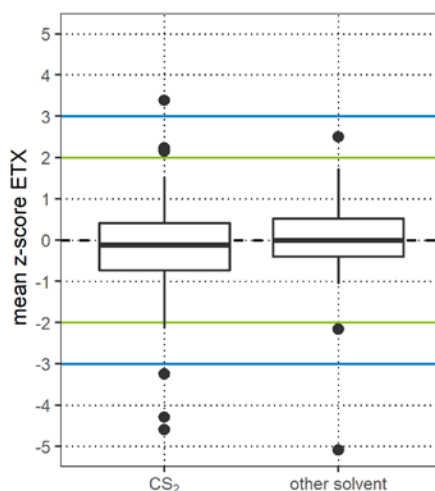


Table 16: Correlation of ETX measurement results with the desorption solvent (2016-2019)

solvent used in desorption	CS ₂	other solvent
75 th percentile (z-score)	0,41	0,52
median (z-score)	-0,12	-0,01
25 th percentile (z-score)	-0,73	-0,40
number of values	125	19

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. The information provided by the participants results in the following picture:

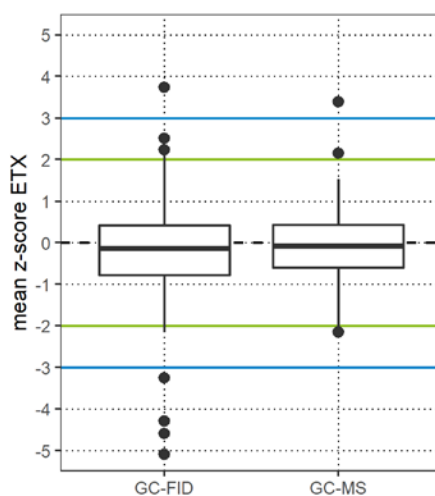


Table 17: Correlation of ETX measurement results with analytical instruments (2016-2019)

analytical instrument	GC-FID	GC-MS
75 th percentile (z-score)	0,42	0,43
median (z-score)	-0,14	-0,08
25 th percentile (z-score)	-0,78	-0,60
number of values	60	85

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

7.9 Feedback from Participants

Since the beginning of 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), and 3 (rather bad), to 4 (very bad). The mean value for the answers to the respective question is listed in the following table.

Table 18: Feedback from participants (2019)

How do you rate the...	mean value	rating
... organization before the start of the proficiency test (documents, e-mails)?	1,6	rather good
... supply of information for preparation (invitation letter, leaflets, etc.)?	1,4	very good
... organization during the proficiency test?	1,3	very good
... design of the schedule (sequence, start time, lunch breaks, end, etc.)?	1,3	very good
... staff's qualification?	1,3	very good
... staff's friendliness?	1,1	very good
... help from staff with technical questions and problems?	1,4	very good
... equipment of the facilities?	2,0	rather good
... functionality of the sampling ports on the stack?	1,3	very good
... power supply (number and type of sockets)?	1,4	very good
... ventilation system (exhaust gas removal)?	1,7	rather good
... execution of the Mini-Audit?	1,3	very good

Overall, the feedback received showed a high level of satisfaction of the participants with the current proficiency tests. The participants were particularly satisfied with the friendliness of the staff (average mark: 1.1). The worst ratings in comparison were given to the equipment of the facilities (average mark: 2.0). Individual participants stated "more space at the stack" as a suggestion for improvement. At present, this could only be achieved by halving the number of participants and doubling the participation fees.

CO₂ was suggested by one participant as an additional component. If this wish should arise more frequently in the future, an implementation at our ESA can be examined.

8. Concluding Remark

Since 2015, all sites of the measuring bodies authorized in accordance with §29b BImSchG have systematically and regularly participated in stack emission proficiency tests at HLNUG's emission simulation apparatus in Kassel. The results registered since then show a clear positive effect on the quality of the measurement results. Especially the measurement results for total dust mass concentrations and heavy metal mass concentrations show a clear improvement compared to the period before 2015. For the gaseous components, a significant deterioration of the results was observed in 2018. Since the results improved significantly again in 2019, it was obviously possible to correct errors quickly. It is at least questionable whether the quality deficits observed in 2018 in the results of HLNUG's stack emission proficiency tests in some §29b measuring bodies would have been noticed and corrected just as quickly in another way. This shows once again that the regular participation of all authorized bodies in stack emission proficiency tests is still important in order to detect erroneous trends early on and to maintain the quality of the measurement results in the regulated area at a consistently high level.

Kassel, 26th March 2020

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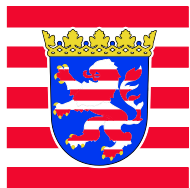
Head of Department
(Dezernatsleiter)

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