Hessisches Landesamt
für Naturschutz,
Umwelt und Geologie

Hessian Agency
for Nature Conservation,
Environment and Geology

HESSEN

Annual Report 2017

Results of proficiency tests for substance ranges P, G, and O on the emission simulation apparatus for quality assurance in stack emission measurements in the year 2017

HLNUG Department I3 is accredited for performing testing services according to
DIN EN ISO/IEC 17025 and DIN EN ISO/IEC 17043.
The accreditation is valid for the testing procedures listed in the certificate.

DAkkS Deutsche Akkreditierungsstelle D-PL-14551-02-00

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

Contents

1. About this Report

This report is a translation of „*Jahresbericht 2017 – Ergebnisse der Ringversuche der Stoffbereiche P, G und O an der Emissionssimulationsanlage zur Qualitätssicherung von Emissionsmessungen im Jahr 2017*" 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.

2. Summary

A total of 43 measuring institutes took part in HLNUG's dust emission proficiency tests (substance range P) in 2017, 38 of which were §29b measuring bodies and 5 volunteers. As in the past, the success rate of the §29b measuring bodies (92%) was significantly higher than that of the volunteers (20%).

A total of 42 measuring institutes took part in the gas emission proficiency tests (substance range G) in 2017, 38 of which were §29b measuring bodies and 4 volunteers. As in previous years, the success rate for the §29b measuring bodies (92%) was significantly higher than for the volunteers (25%).

A total of 18 measuring bodies took part in the odour emission proficiency tests (substance range O) in 2017, 12 of them on the basis of an authorization in accordance with §29b BImSchG and 6 voluntarily. Here 83% of the authorized participants were successful and 0% of the volunteers.

3. Introduction

3.1 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 (1) Federal Immission Control Act) in Germany. The proficiency tests presented in this annual report are accredited according to DIN EN ISO/IEC 17043 (2) and are recognized 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 our 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 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.

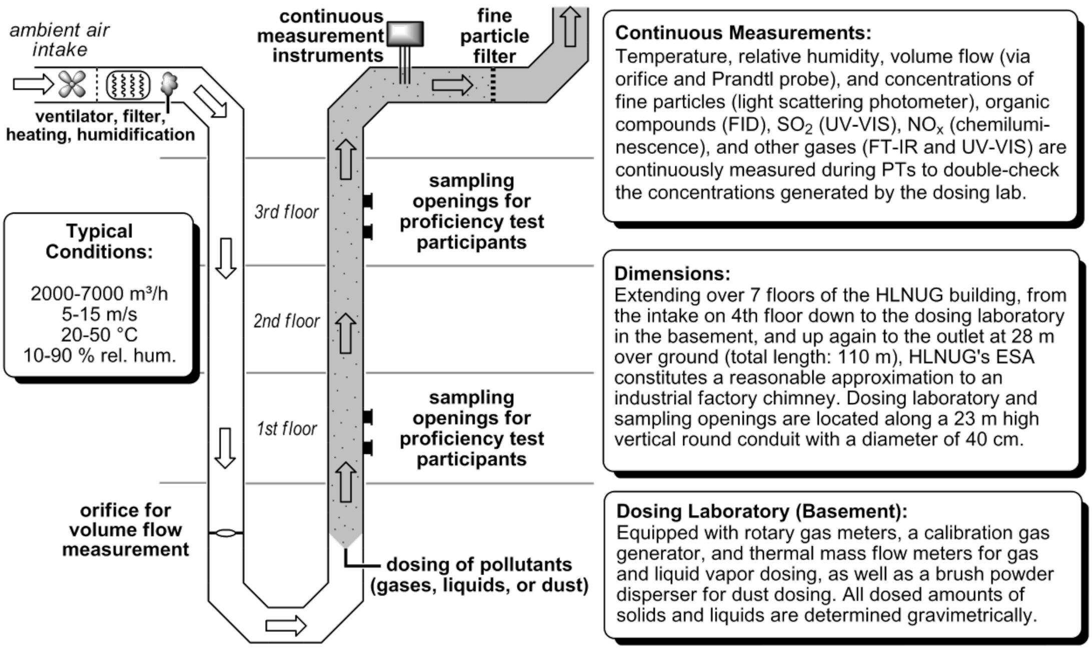
3.2 The Emission Simulation Apparatus

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 at which there are measuring openings 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. 5 – 11 m/s, moving a vloume of approx. 2200 – 5000 m^3/h through the system.

The air pollutants to be measured by the participants in the proficiency test are dispensed into the air flow in the dosing laboratory in the cellar. For this purpose, the dosing laboratory is equipped with various drum gas meters for dosing different gases, a calibration gas generator for dosing liquids and a brush dosing unit for dosing dusts. Various measuring instruments continuously measure volume flow, pressure, temperature and humidity as well as the concentrations of organic compounds (as TOC) and various other components in order to constantly check the concentrations generated by the dosing laboratory



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

4. Organizational Information

In 2017, the following proficiency tests of the substance ranges P, G, and O were carried out:

Table 1: Proficiency Tests organized by HLNUG

proficiency test	substance range	start	end	participants
RV 471S	particulate emissions (substance range P)	30.01.2017	31.01.2017	7
RV 472G	gaseous emissions (substance range G)	01.02.2017	03.02.2017	7
RV 473S	particulate emissions (substance range P)	13.02.2017	14.02.2017	7
RV 474G	gaseous emissions (substance range G)	15.02.2017	17.02.2017	8
RV 475S	particulate emissions (substance range P)	06.03.2017	07.03.2017	8
RV 476G	gaseous emissions (substance range G)	08.03.2017	10.03.2017	8
RV 477S	particulate emissions (substance range P)	27.03.2017	28.03.2017	8
RV 478G	gaseous emissions (substance range G)	29.03.2017	31.03.2017	8
RV 479O	odour emissions (substance range O)	28.09.2017	28.09.2017	6
RV 480O	odour emissions (substance range O)	05.10.2017	05.10.2017	7
RV 481S	particulate emissions (substance range P)	06.11.2017	07.11.2017	7
RV 482G	gaseous emissions (substance range G)	08.11.2017	10.11.2017	6
RV 483S	particulate emissions (substance range P)	27.11.2017	28.11.2017	6
RV 484G	gaseous emissions (substance range G)	29.11.2017	01.12.2017	5
RV 485O	odour emissions (substance range O)	04.10.2017	04.10.2017	5

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

	substance range P	substance range G
duration of each sampling	30 min	30 min (dicontinuous samplings and TOC), 15 min (continuous measurements of propane, NO_x, SO_2)
number of samplings	for each component 10 including introductory measurement	
sampling	simultaneously for all participants (1st and 3rd floor)	
basic conditions	not detailed in specifitations	2000 … 3500 m^3/h 20 … 40 °C
concentrations	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 als NO_2: 60 … 450 mg/m^3 sum ETX: 4 … 100 mg/m^3 formaldeyhde: 4 … 40 mg/m^3 TOC: 4 … 100 mg/m^3 (ETX/propane) TOC: 5 … 100 mg/m^3 (propane only)
result submission	swithin 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.	swithin 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 2) or two (G4-7, see table 2) digits after decimal point.

	substance range P	substance range G
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 (1st and 3rd floor)
basic conditions	2000 … 6000 m^3/h, flow velocity > 4 m/s, water vapour up to 50 g/m^3
concentrations	approx. 50 … 50000 ou_E/m^3
result submission	on the day of the proficiency test, until 19:00 Uhr
submission procedure	results are entered into an Excel-file provided by HLNUG and handed in personally on the same day

The proficiency tests were organized 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*)

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: emission@hlnug.hessen.de

Technically responsible for the execution of the proficiency tests are currently:
Dr. Dominik Wildanger, Dr. Jens Cordes and Benno Stoffels.

5. Execution of the Proficiency Tests

5.1 Particulate Standards

In contrast to the pure substances used in gas and odour proficiency tests, no reference materials are available on the market in sufficient quantities for particulate substances. For this reason, reference dusts are produced in the HLNUG for proficiency tests in substance range P.

The matrix is an industrial dust which is optimized by targeted heavy metal doping, grinding, screening and drying steps. A complete homogenization of the dust standard is finally achieved by intensive mixing of the batch.

The determination of the conventionally correct value ("true value") of the heavy metal concentration of a doped dust sample is based on the data from ring analyses carried out by laboratories of various state institutes. The robust mean value from the individual values of the ring analyses is regarded as the true 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 checked according to DIN ISO 13528 (1).

5.2 Execution of the Measurements

Each participant determines the mass concentrations (substance range P and G), or the odour concentrations (substance range O) respectively, of all components in accordance with (DIN) EN 15259:2008-01 (2). In addition, the basic conditions (flue gas velocity, volume flow, temperature, humidity, and static pressure) are measured before the start of the sampling.

Determination of dust and dust composition (substance range P)

The measurements are performed as follows:

day of the test	component	compulsory measurement procedure
day 1 and 2	dust	(DIN) EN 13284-1 (3) and/or VDI 2066 part 1 (4)
	heavy metals	not specified

Determination of gaseous emissions (substance range G)

The measurements are performed as follows:

day of the test	component	compulsory measurement procedure
day 1	formaldehyde	VDI 3862 part 2, 3 or 4 (5) (6) (7)
day 2	SO_2	(DIN) EN 14791 (8)
	TOC	(DIN) EN 12619 (9)
	ETX	(DIN) EN 13649 (10)
day 3	SO_2	using a suitability tested device
	TOC	(DIN) EN 12619 (9)
	NO_x as NO_2	(DIN) EN 14792 (11)

Determination of odour emissions (substance range O)

The measurements are performed as follows:

day of the test	component	compulsory measurement procedure
day 1	four odours	(DIN) EN 13725

5.3 Evaluation

5.3.1 Calculation of z-Scores

Substance range P and G

The evaluation of the proficiency test is carried out 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:

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

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

Substance range O

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

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

In this equation, X_{ik} is the assigned value of the measurement, and σ_k is the precision criterion for component *k*.

The assigned value X_{ijk} is calculated from the mass concentration c_{ik} and the odour threshold $c_{0,k}$ of the component:

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

The dosed mass concentration c_{ik} is determined for each measurement based on the meas-urement data of the dosing device and the volume flow. The odour threshold $c_{0,k}$ of *n*-butanol is $c_0 = 123\ \mu\text{g/m}^3$. 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 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' measure-ment results. Provided that the sampling was carried out within 14 days, results of sev-eral proficiency tests can be taken into account. Solely results of participants are con-sidered, 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. 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 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. 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.

The precision criteria for the different components are listed in the following table:

Table 2: Components, short designations and precision criteria

No.	component	measurement mode	short designa-tion	precision criterion $\boldsymbol{\sigma_k}$ in % of true value
		substance range P		
P1	dust	discontinuous	St	7,0
P2	Cadmium	discontinuous	Cd	8,0
P3	Cobalt	discontinuous	Co	8,0
P4	Chromium	discontinuous	Cr	12,0
P5	Copper	discontinuous	Cu	8,0
P6	Nickel	discontinuous	Ni	8,0
P7	Lead	discontinuous	Pb	8,0
		substance range G		
G1	SO_2	discontinuous	Sd	3,1
G2	SO_2	continuous	Sk	3,9
G3	NO_x as NO_2	continuous	Nk	3,1
G4	toluene	discontinuous	Td	5,6
G5	ethylbenzene	discontinuous	Ed	5,8
G6	sum of *o*-, *m*-, *p*-xylene	discontinuous	Xd	5,3
G7	formaldehyde	discontinuous	Fd	3,5
G8	TOC (propane, ETX)	continuous	Ck	3,3

No.	component	measurement mode	short designation	precision criterion $\boldsymbol{\sigma_k}$ in % of true value
substance range G – components not required for passing				
G9	TOC (propane only)	continuous	Pk	3,3
G10	toluene (standard)	laboratory analysis	STDT	4,5
G11	ethylbenzene (standard)	laboratory analysis	STDE	4,5
G12	*o*-, *m*-, *p*-xylene (standard)	laboratory analysis	STDX	4,5
substance range O				
-	all odours	discontinuous	e.g. NBU, AAC, …	0,1†

† In proficiency test O the precision criterion is not expressed in % of true value (see section 5.3.1)

5.3.2 Evaluation of z-Scores

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

$$|z_{ijk}| \leq 2 \qquad \text{result satisfactory}$$

$$2 < |z_{ijk}| < 3 \qquad \text{result questionable}$$

$$|z_{ijk}| \geq 3 \qquad \text{result unsatisfactory}$$

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

For the substance ranges P and G, in the next step of the evaluation 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:

$$z_{jk} \leq 2 \qquad \text{results in } K_{jk} = 1$$

$$2 < z_{jk} < 3 \qquad \text{results in } K_{jk} = 2$$

$$z_{jk} \geq 3 \qquad \text{results in } K_{jk} = 3$$

For each component in gas and dust emission proficiency tests, at least 6 measurement results must be submittet, otherwise the respective component is automaticalle evaluated as „failed“.

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

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 result for the component total dust (P1) is also the result for the test part “Dust Concentration”. The overall result of the test part “Dust Composition” is “passed”, if at least 5 out of 6 heavy metal components (P2 to P7) were determined successfully, otherwise it is “failed”.

The overall result of the proficiency test is “passed”, if both test part results are “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”.

The overall result of the proficiency test is “passed”, if all compulsory components (G1 to G8) are “passed”, otherwise the overall result is “failed”.

For the analytical part of the proficiency test (which does not include sampling), z-Scores are calculated following the scheme described above. A component was here determined successfully, if

$$|z_k| < 3$$

The overall result of the analytical part is “passed”, if all 3 components of the standard are “passed”, otherwise the overall result is “failed”.

Odour emission proficiency test

Forthe 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”.

5.3.3 Communication of the Evaluation

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, quoting their unique ID-code.

6. Results

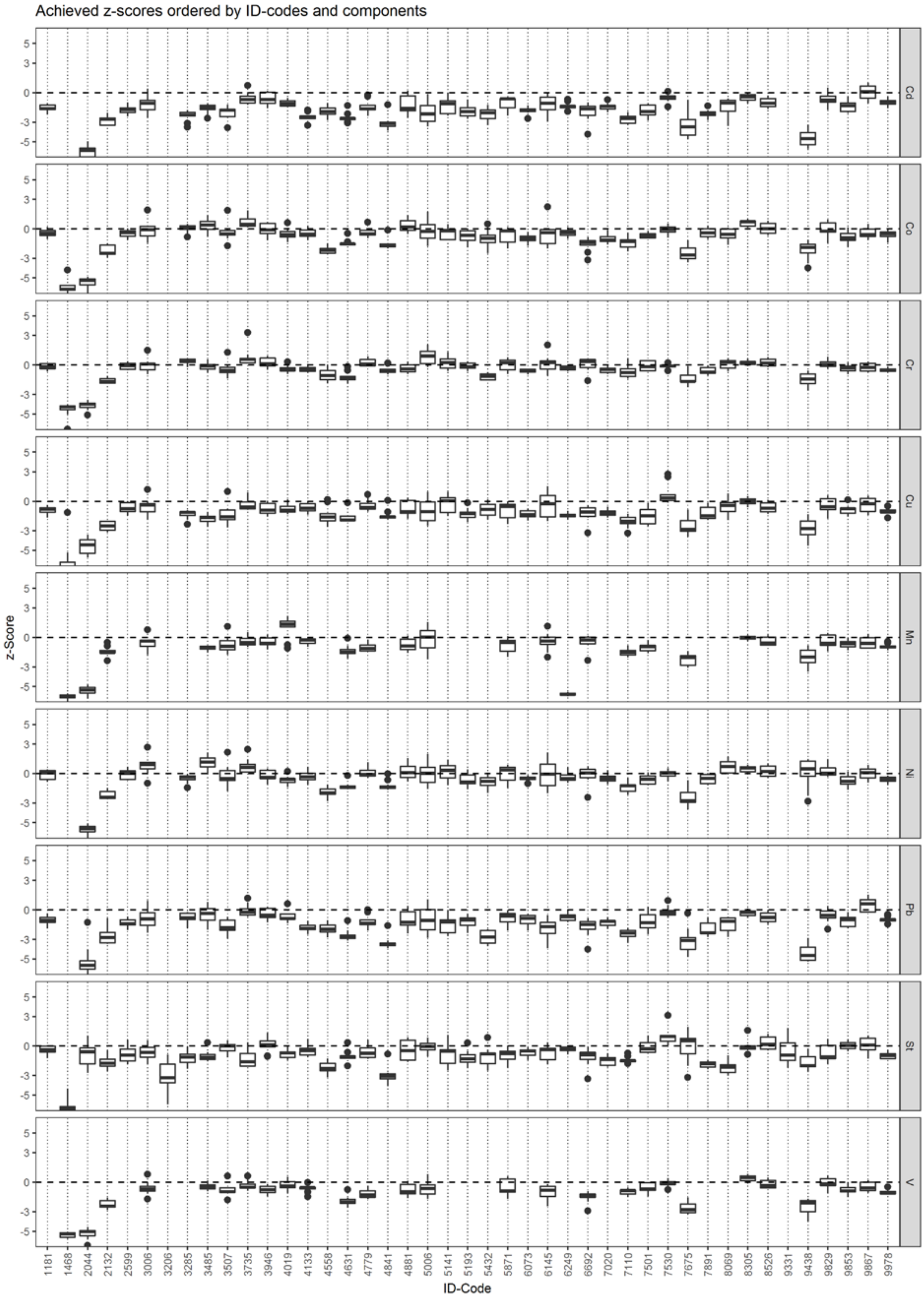
6.1 z-Scores

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.

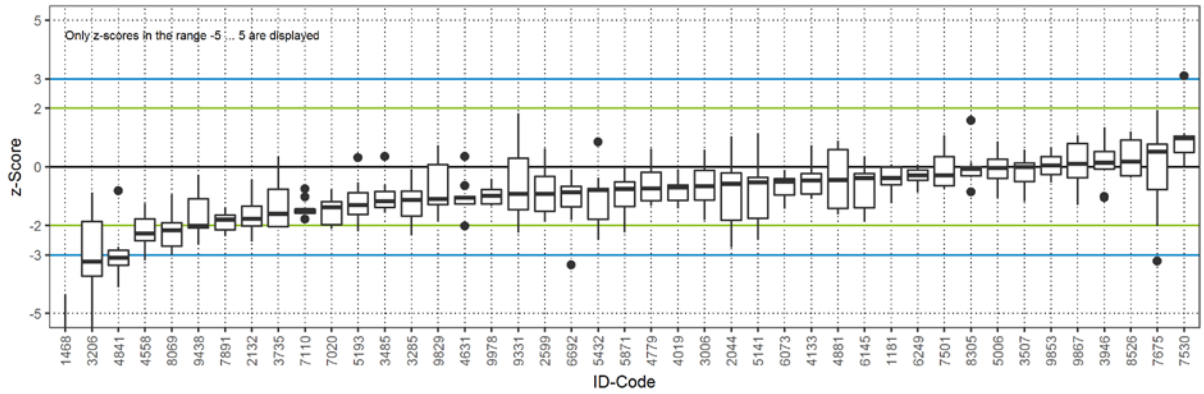
A list of the individual measurements of all participants can be found in a separate document (appendix to the annual report).

6.1.1 Substance Range P

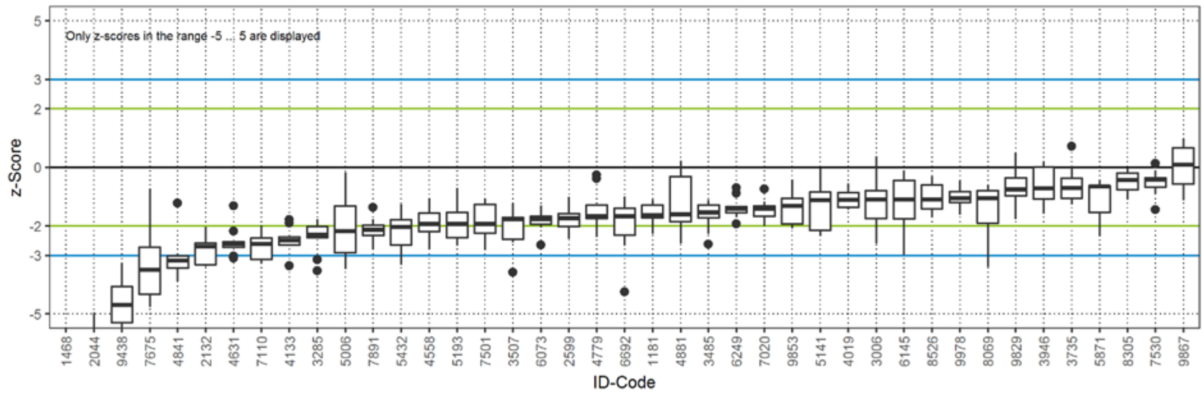


Scheme 2: Achieved z-scores dust proficiency test (only values in the range -5 … 5 are shown)

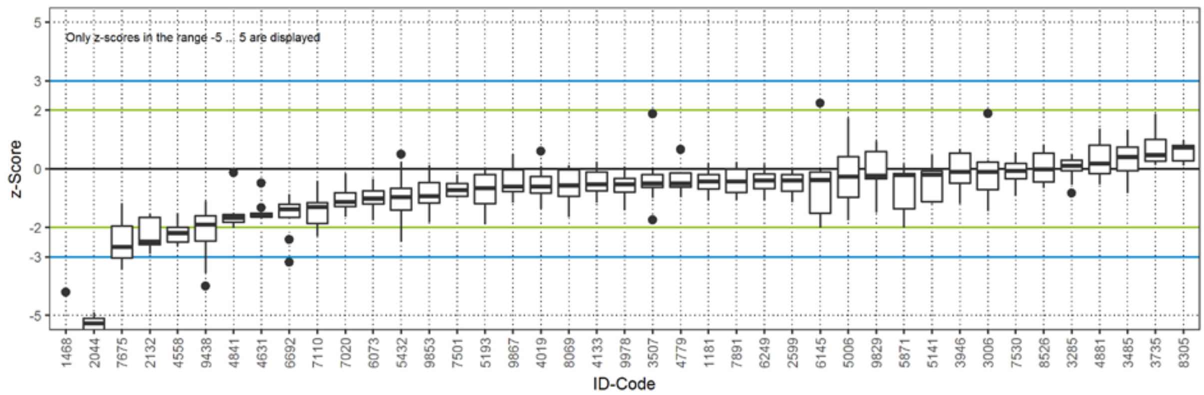
z-scores for component dust concentration (St)



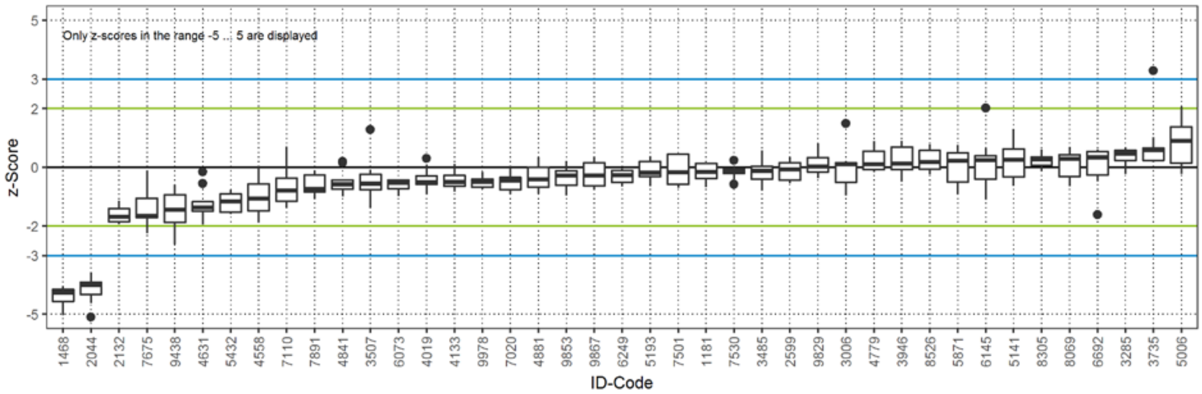
z-scores for component Cadmium (Cd)



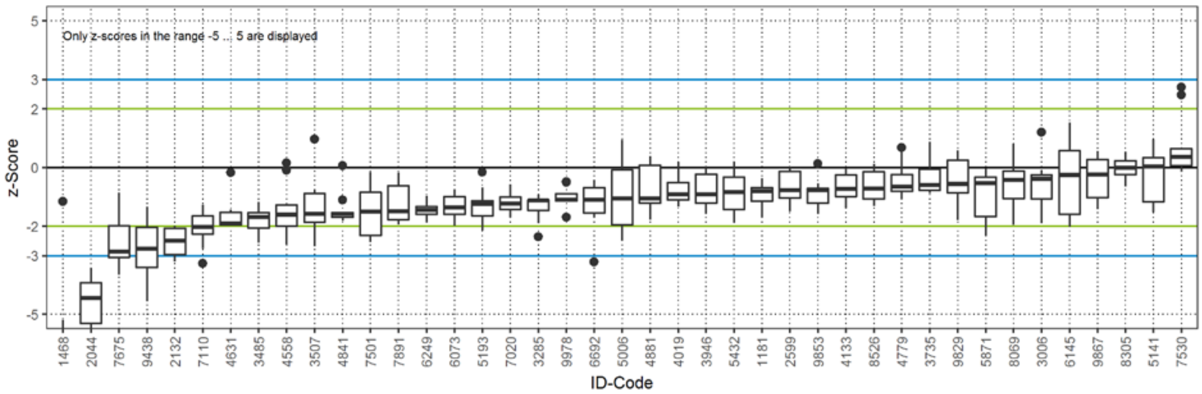
z-scores for component Cobalt (Co)



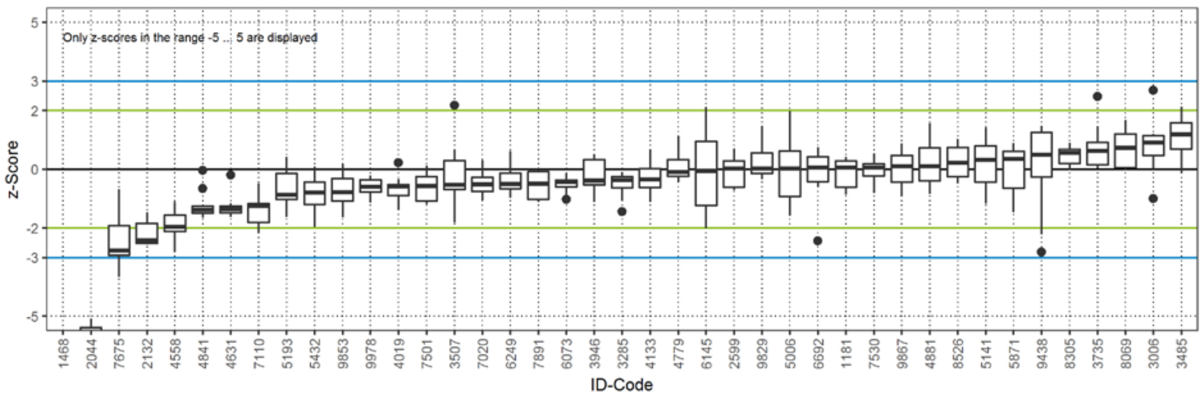
z-scores for component Chromium (Cr)



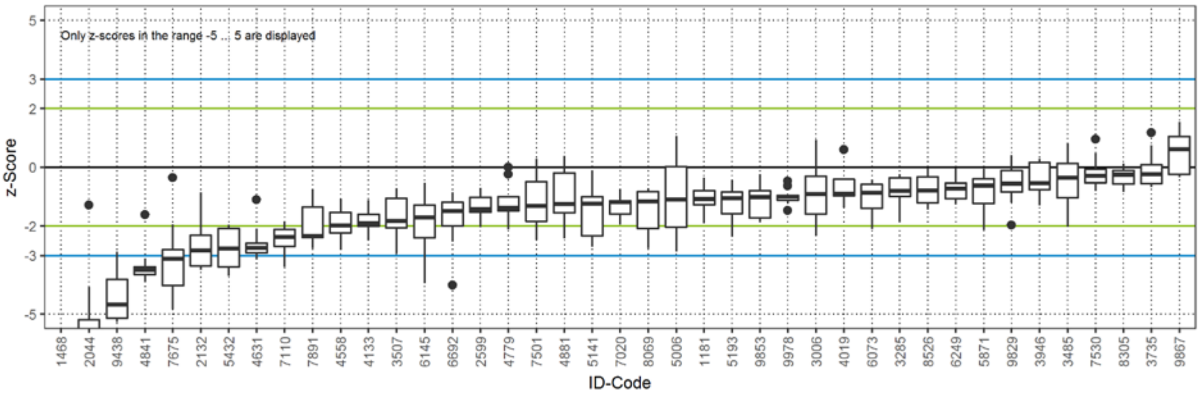
z-scores for component Copper (Cu)



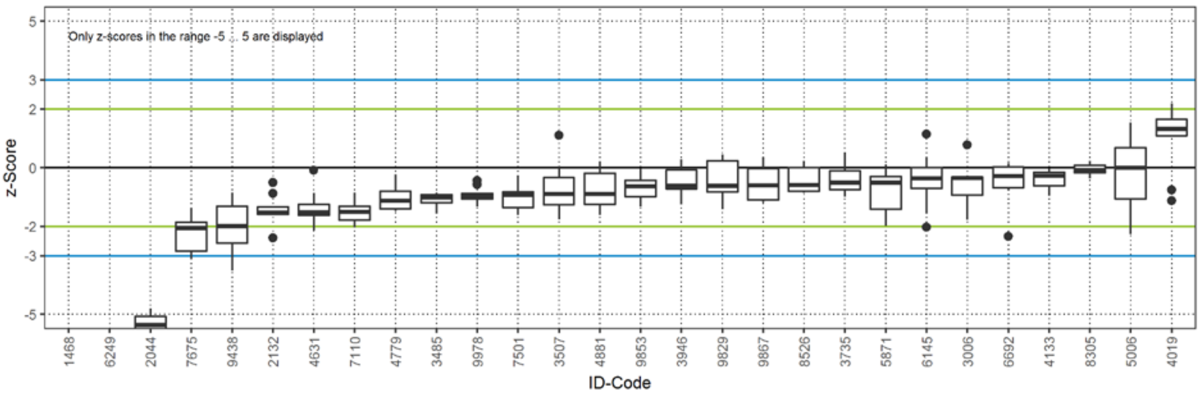
z-scores for component Nickel (Ni)



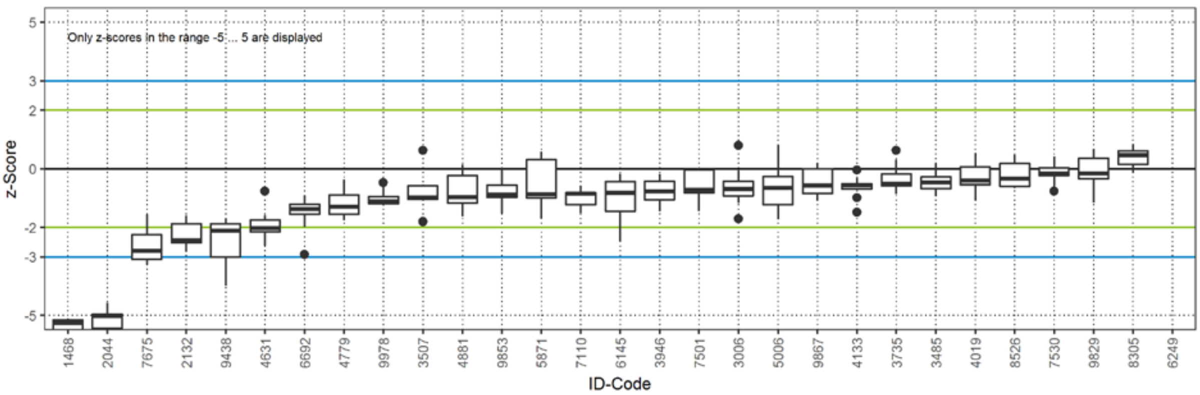
z-scores for component Lead (Pb)



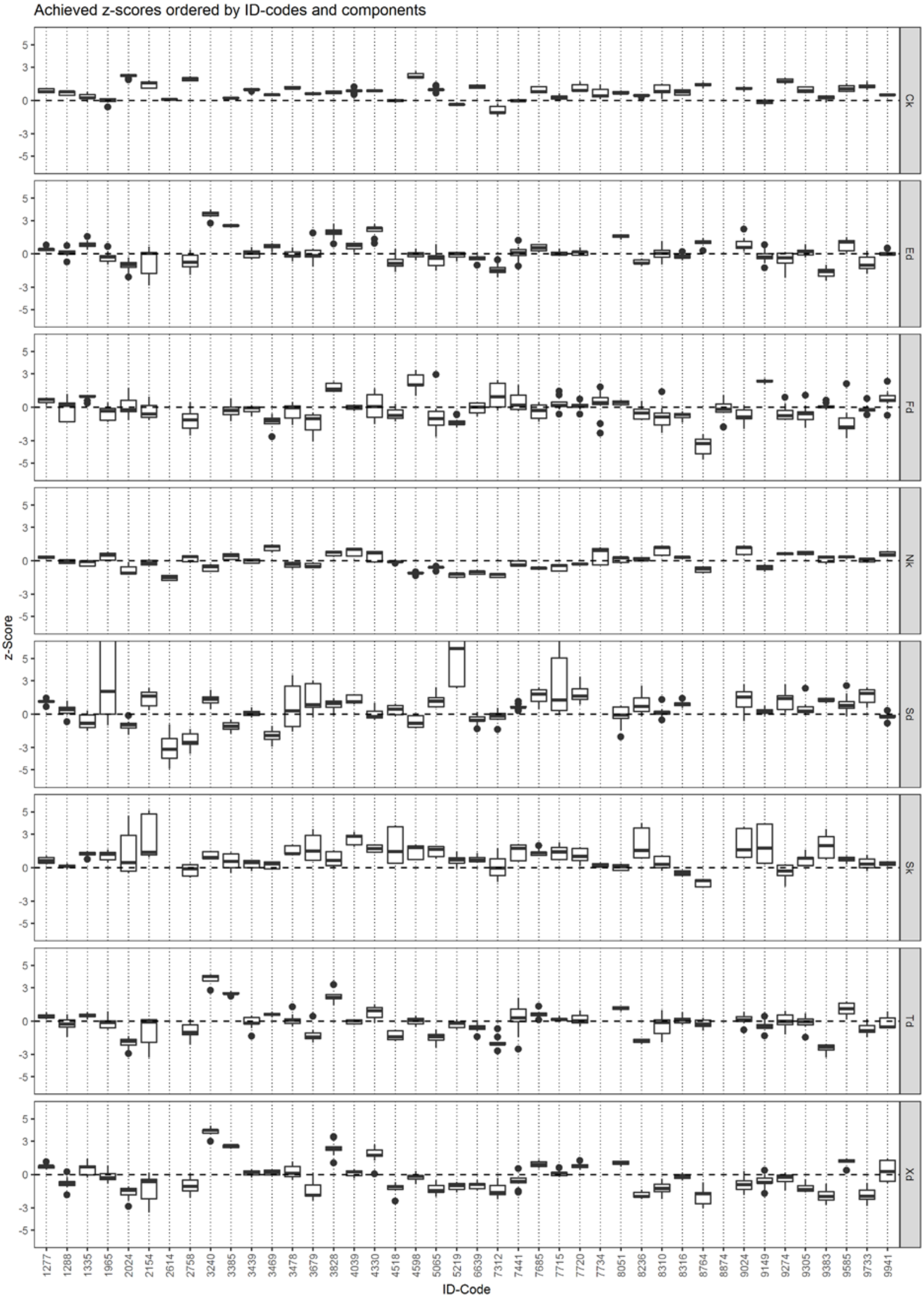
z-scores for component Manganese (Mn)



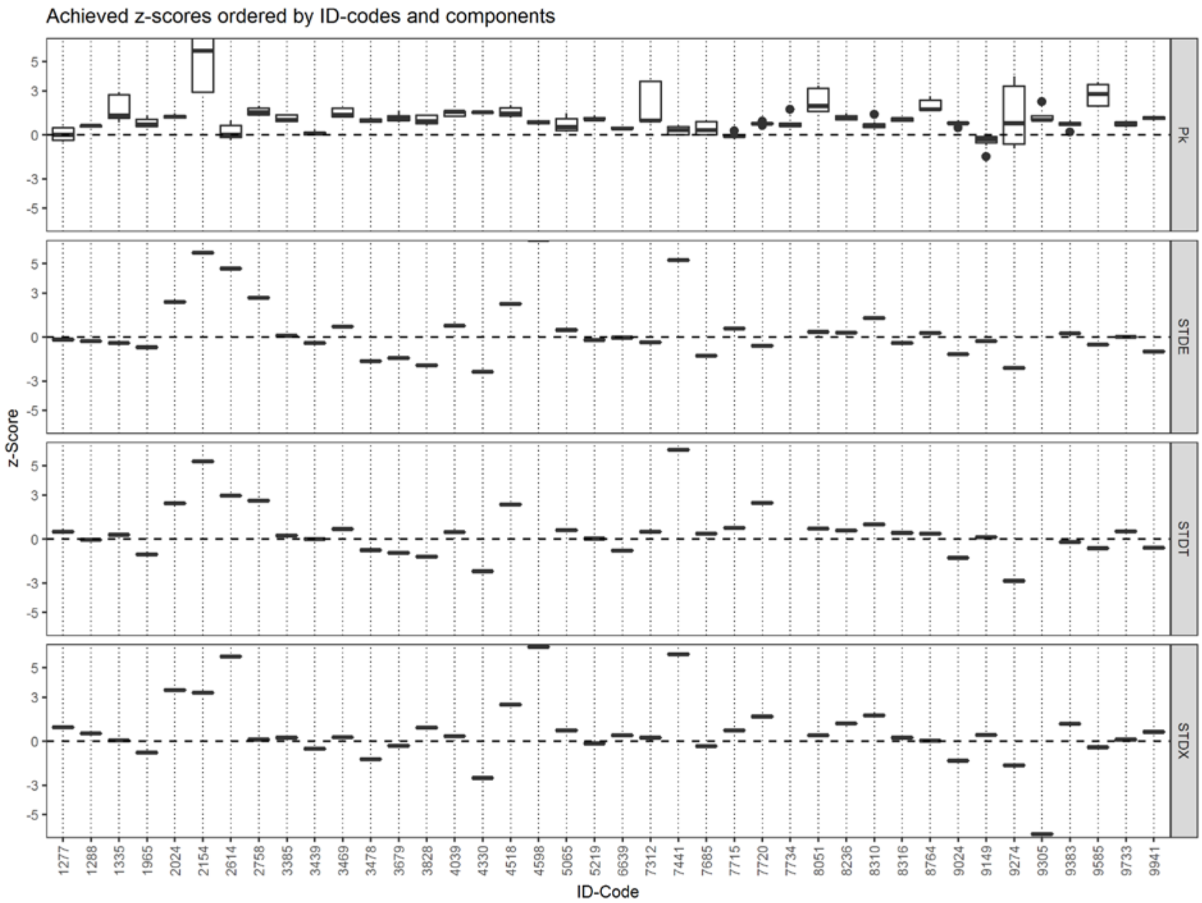
z-scores for component Vanadium (V)



6.1.2 Substance Range G

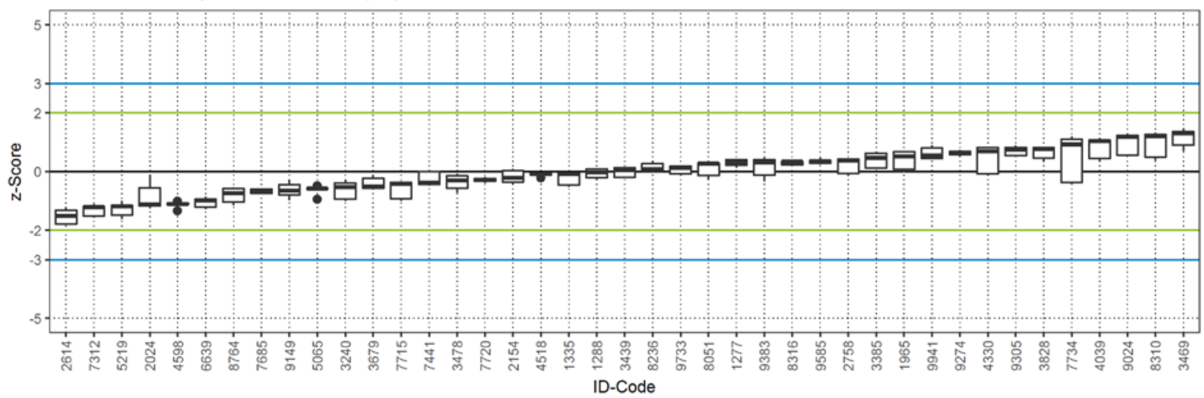


Scheme 3: Achieved z-scores gas proficiency test (only values in the range -5 … 5 are shown)

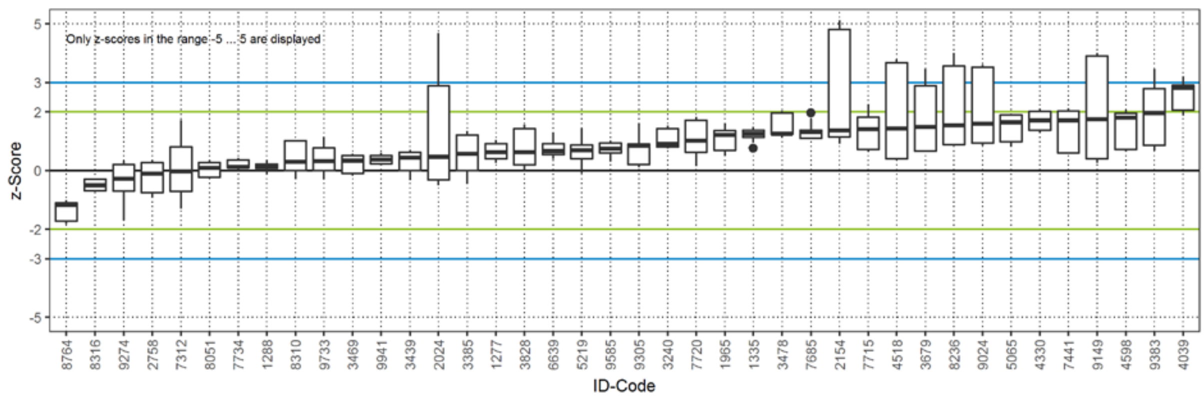


Scheme 4: Achieved z-scores gas proficiency test, components not required for passing (only values in the range -5 … 5 are shown)

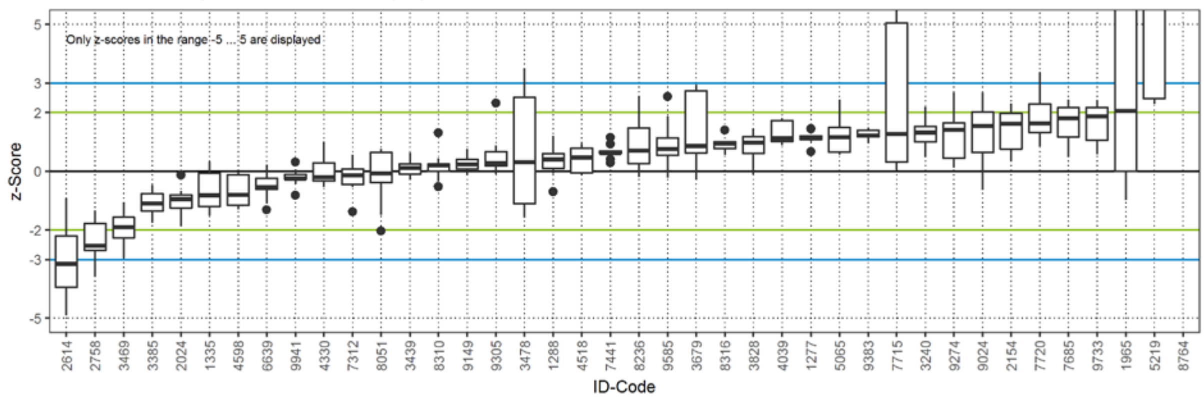
z-scores for component NOx cont. (Nk)

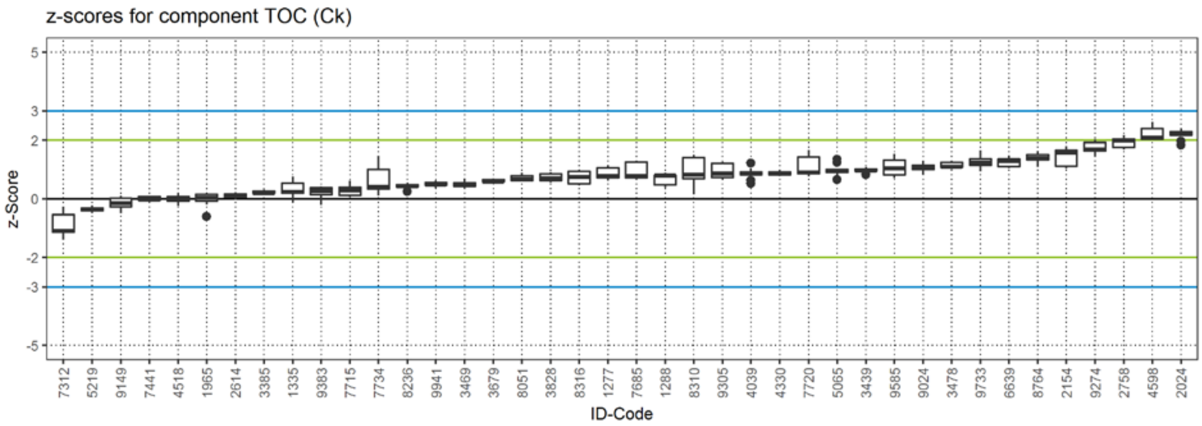


z-scores for component SO2 cont. (Sk)



z-scores for component SO2 discont. (Sd)




z-scores for component TOC (Ck)
z-Score
ID-Code
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5219
9149
7441
4518
1965
2614
3385
1335
9383
7715
7734
8236
9941
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3679
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3828
8316
1277
7685
1288
8310
9305
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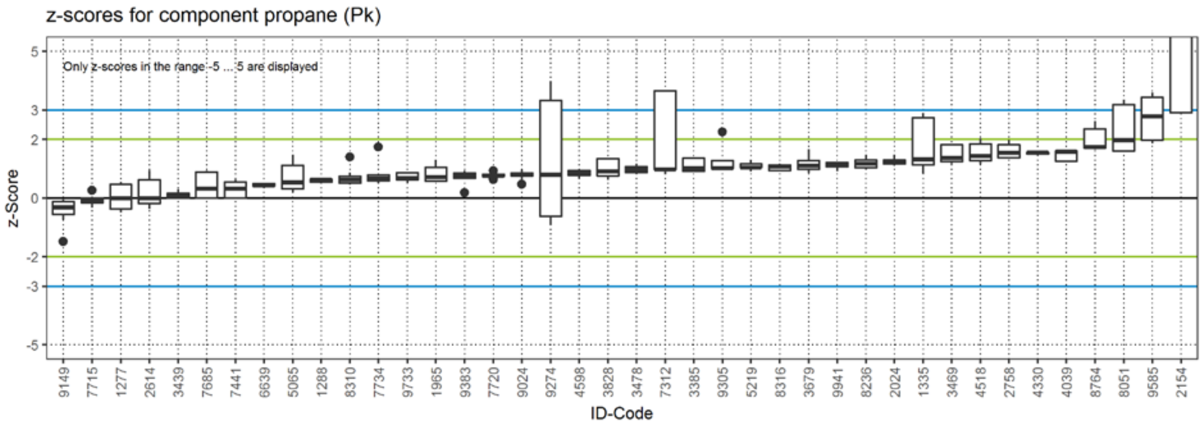
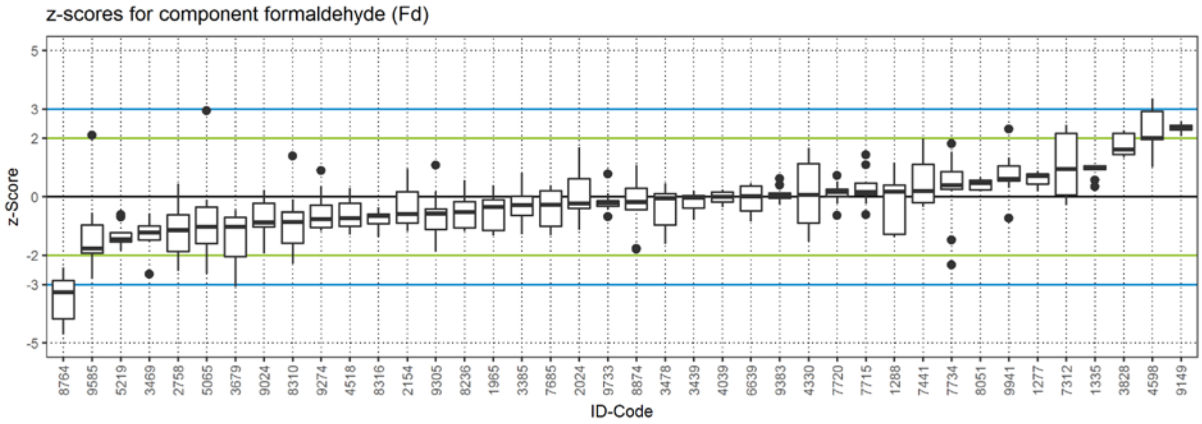
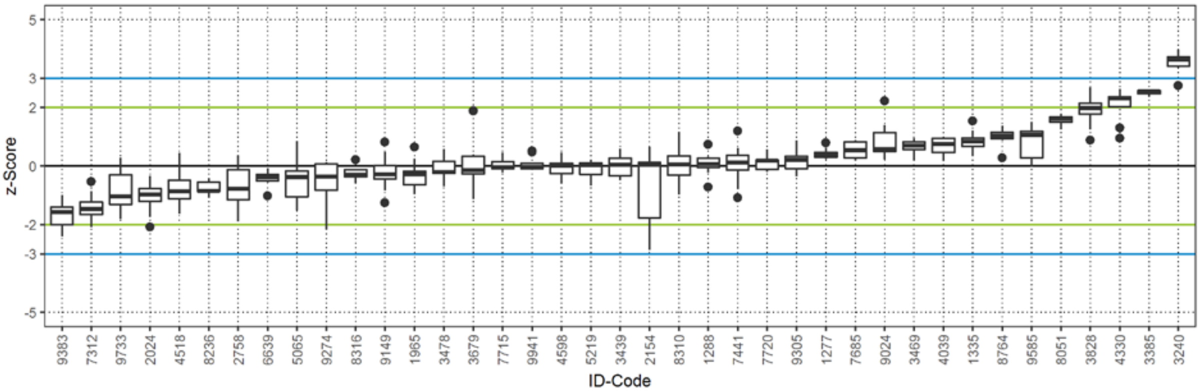
9274
2758
4598
2024

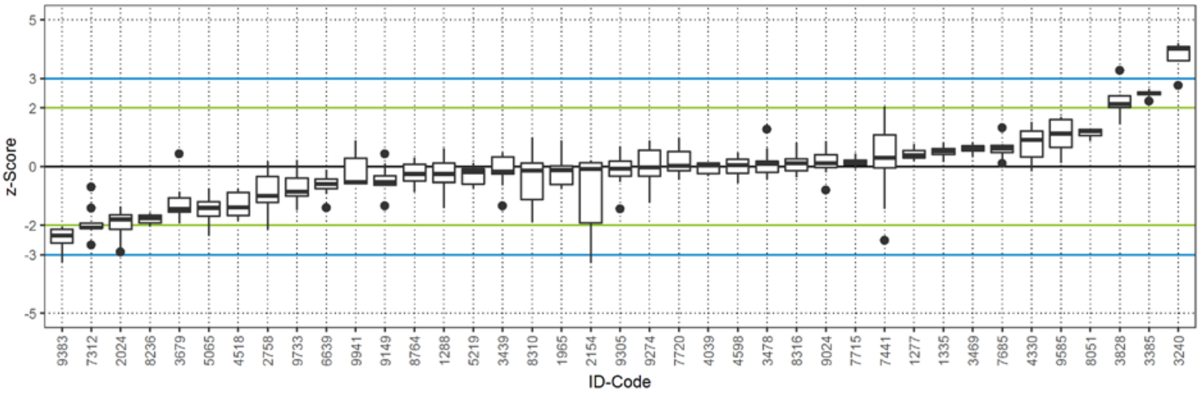

z-scores for component propane (Pk)
Only z-scores in the range -5 ... 5 are displayed
z-Score
ID-Code
9149
7715
1277
2614
3439
7685
7441
6639
5065
1288
8310
7734
9733
1965
9383
7720
9024
9274
4598
3828
3478
7312
3385
9305
5219
8316
3679
9941
8236
2024
1335
3469
4518
2758
4330
4039
8764
8051
9585
2154


z-scores for component formaldehyde (Fd)
z-Score
ID-Code
8764
9585
5219
3469
2758
5065
3679
9024
8310
9274
4518
8316
2154
9305
8236
1965
3385
7685
2024
9733
8874
3478
3439
4039
6639
9383
4330
7720
7715
1288
7441
7734
8051
9941
1277
7312
1335
3828
4598
9149

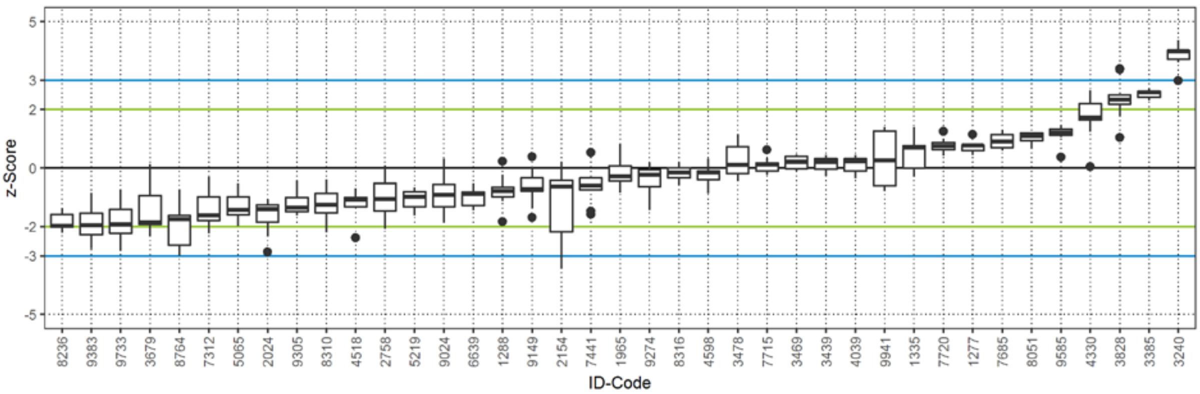
z-scores for component ethylbenzene (Ed)



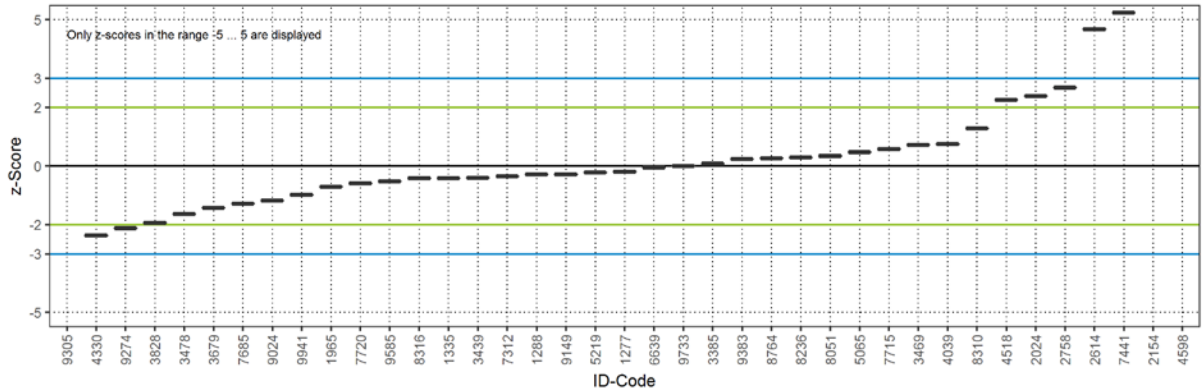
z-scores for component toluene (Td)



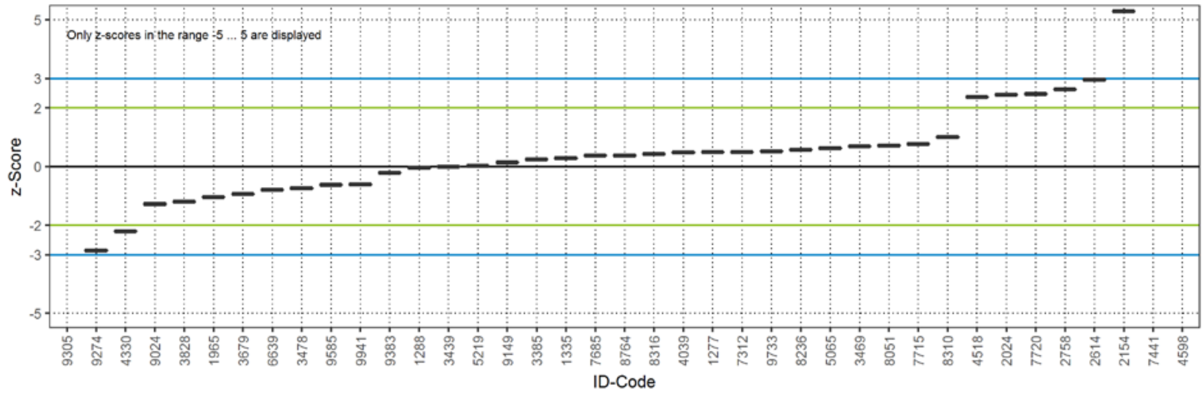
z-scores for component xylene (sum) (Xd)



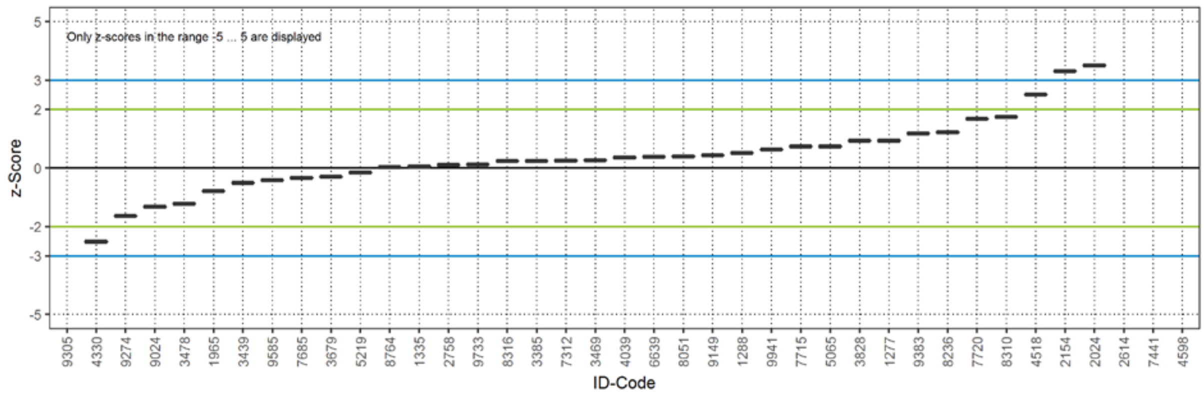
z-scores for component ethylbenzene (standard) (STDE)



z-scores for component toluene (standard) (STDT)

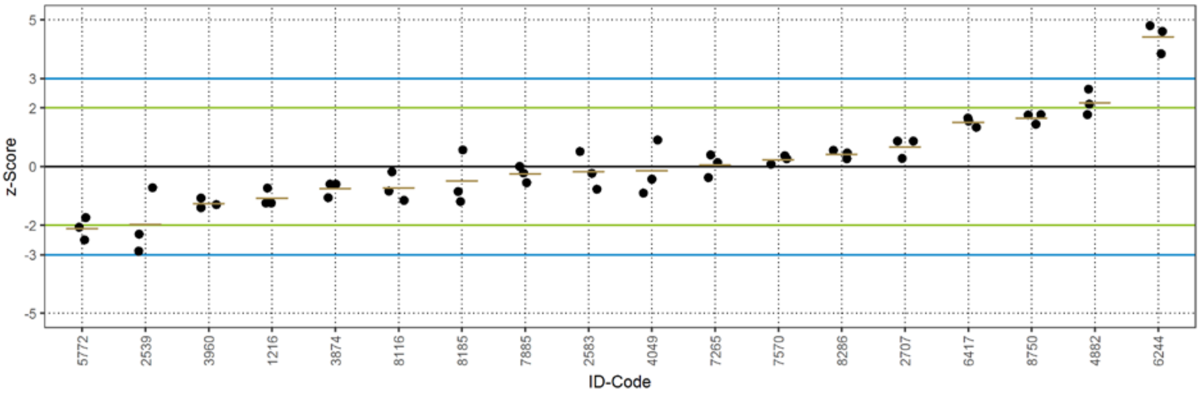


z-scores for component xylene (standard) (STDX)

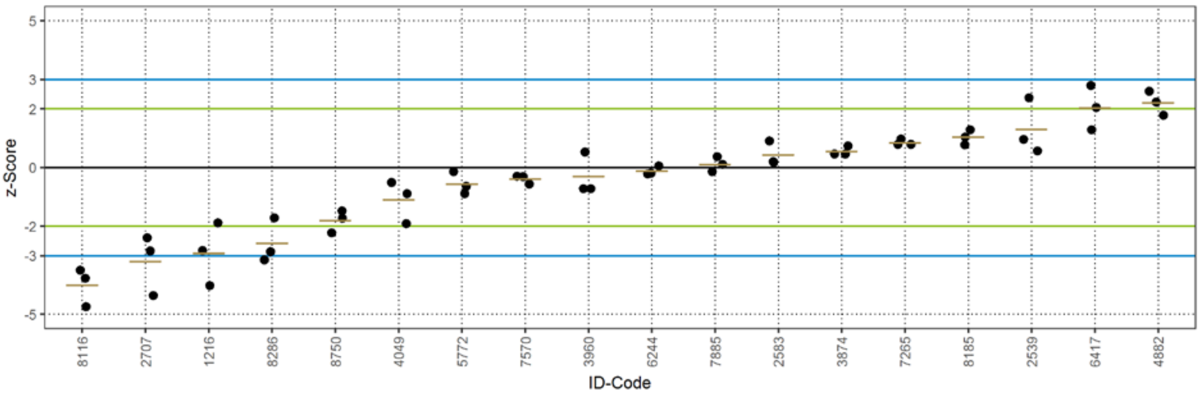


6.1.3 Substance Range O

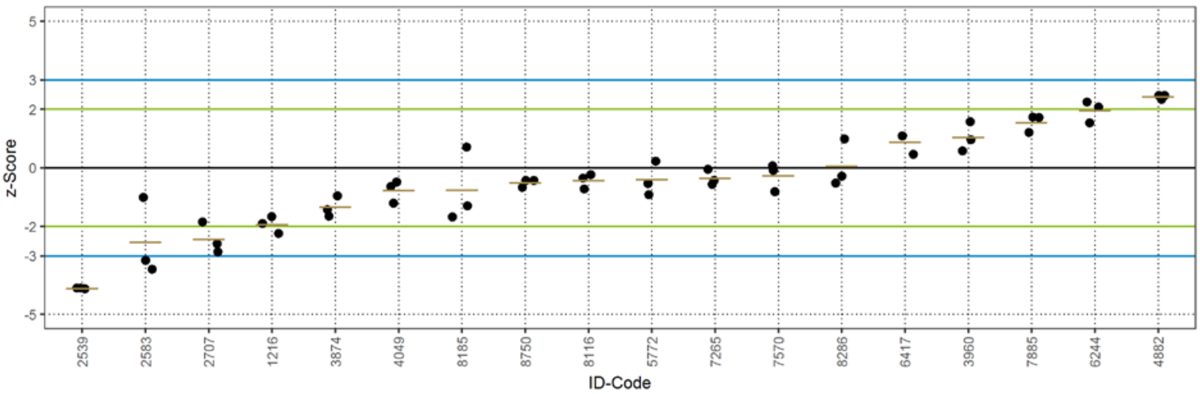
z-scores for component ETX



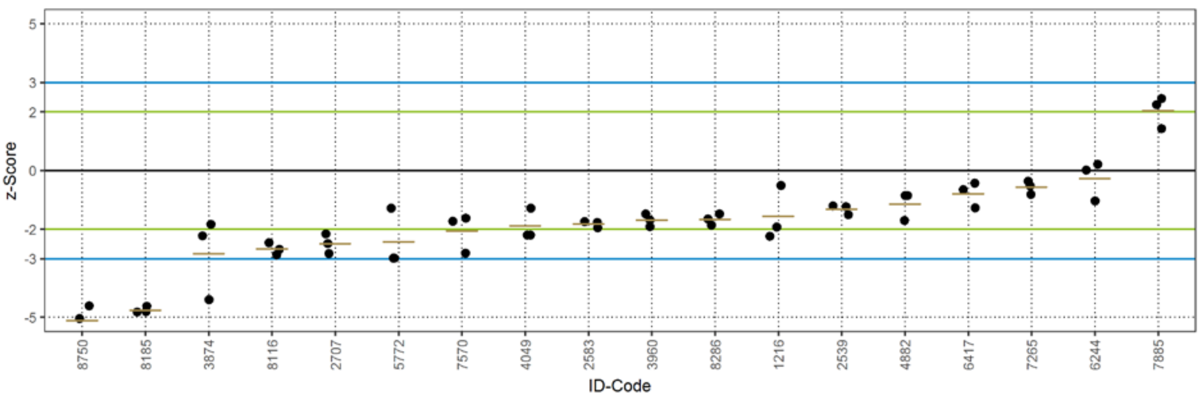
z-scores for component NBU



z-scores for component PIG



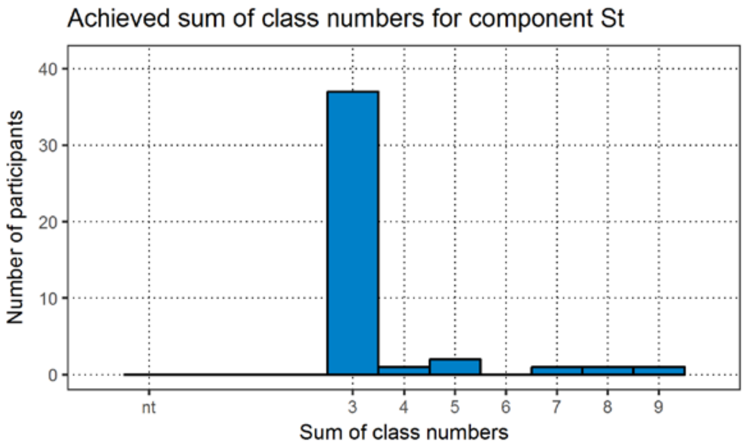
z-scores for component THT

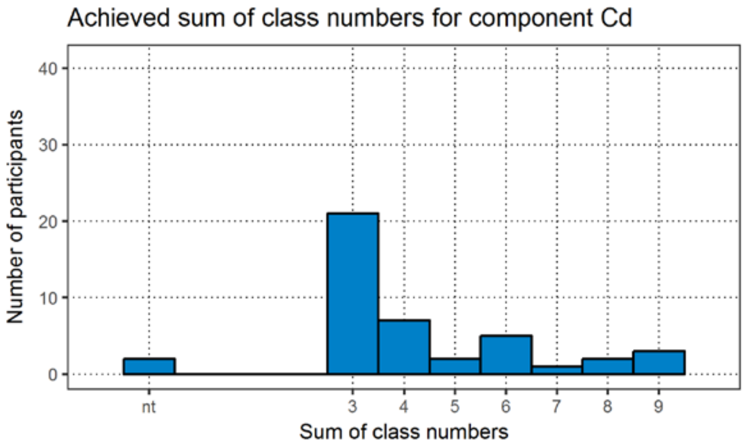


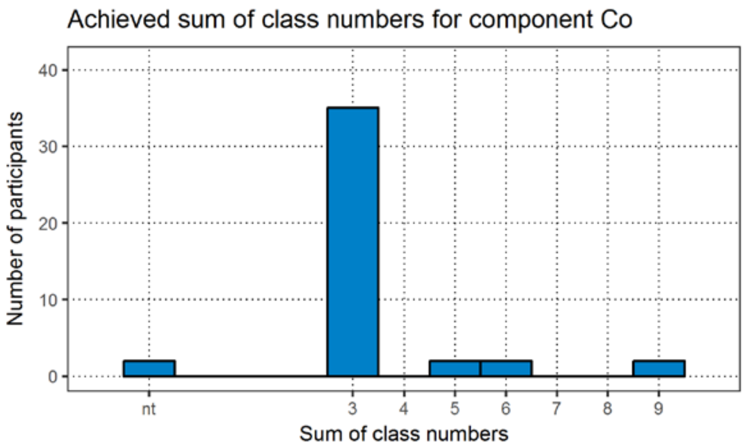
6.2 Acheived Sums of Class Numbers

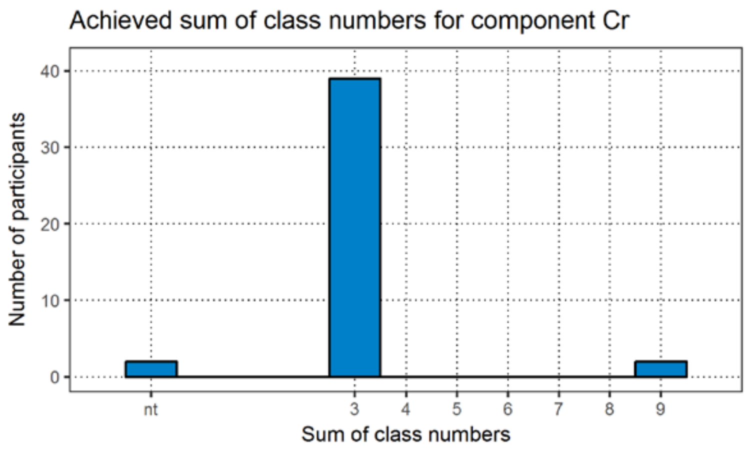
The following schemes show the sum of class numbers that the participants achieved for the different components in form of histogramme charts. For the interpretation of the sums of class numbers, please see section 4.3.2.

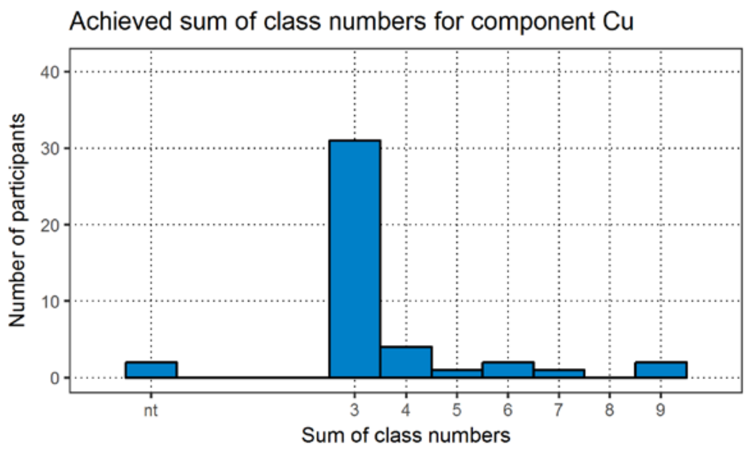
6.2.1 Substance Range P

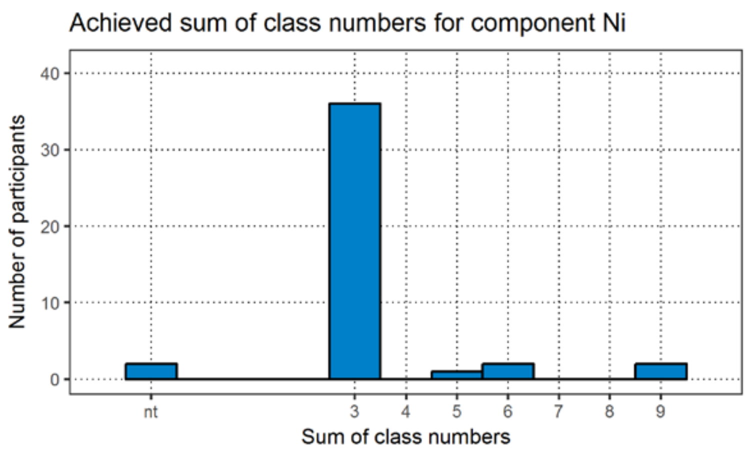


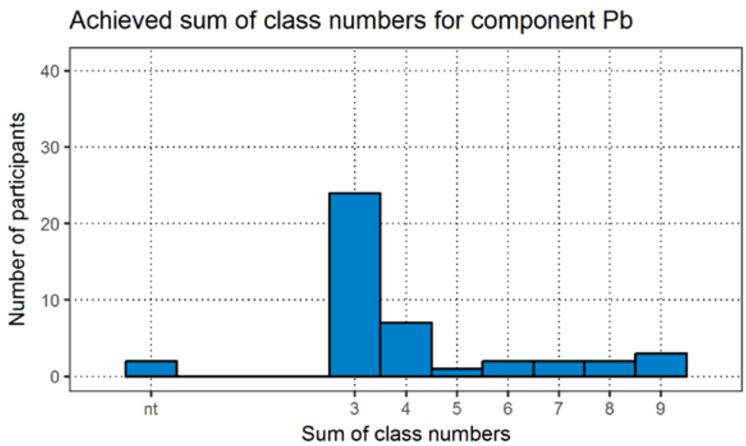


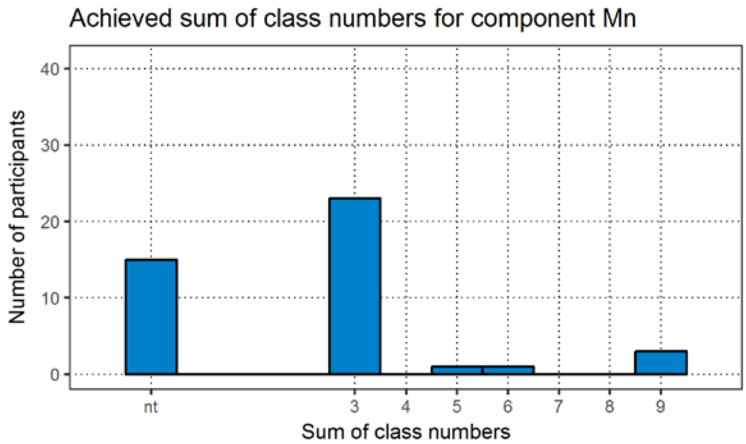


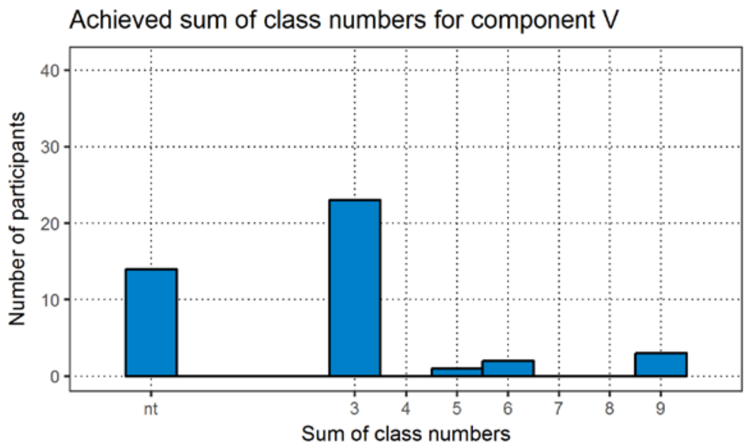

Achieved sum of class numbers for component Cr
Number of participants
0
10
20
30
40
nt
3
4
5
6
7
8
9
Sum of class numbers


Achieved sum of class numbers for component Cu
Number of participants
0
10
20
30
40
nt
3
4
5
6
7
8
9
Sum of class numbers

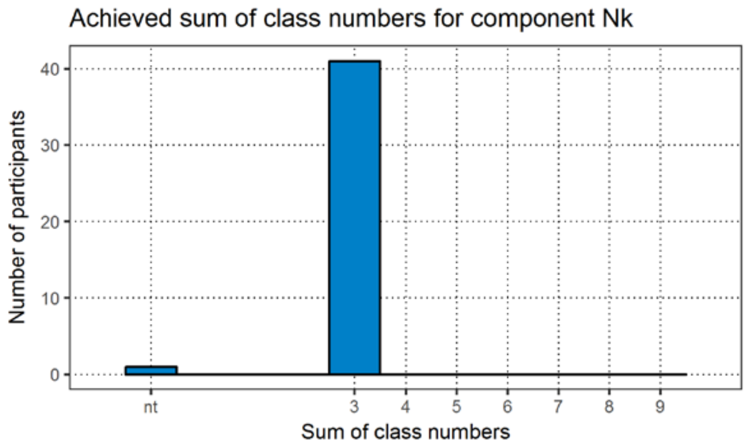

Achieved sum of class numbers for component Ni
Number of participants
0
10
20
30
40
nt
3
4
5
6
7
8
9
Sum of class numbers

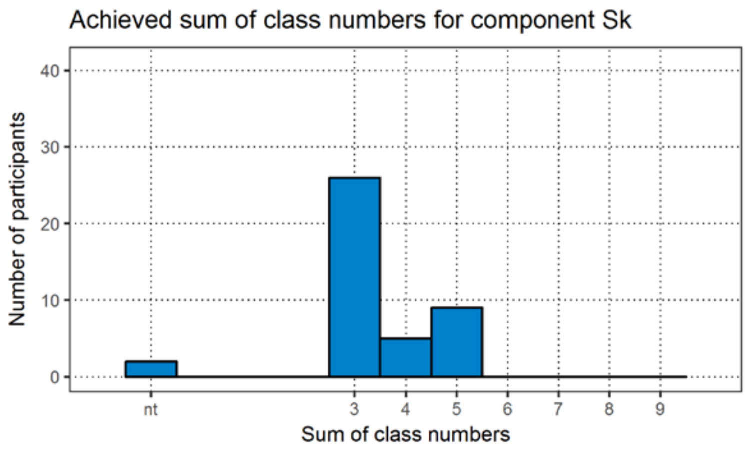

Achieved sum of class numbers for component Pb
Number of participants
0
10
20
30
40
nt
3
4
5
6
7
8
9
Sum of class numbers

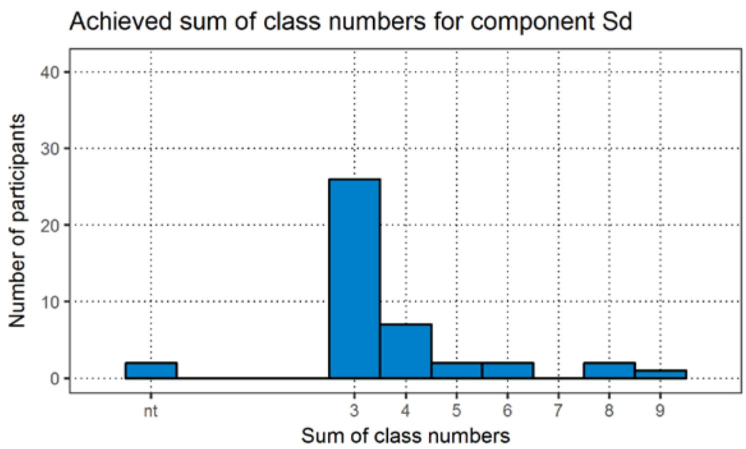

Achieved sum of class numbers for component Mn
Number of participants
0
10
20
30
40
nt
3
4
5
6
7
8
9
Sum of class numbers

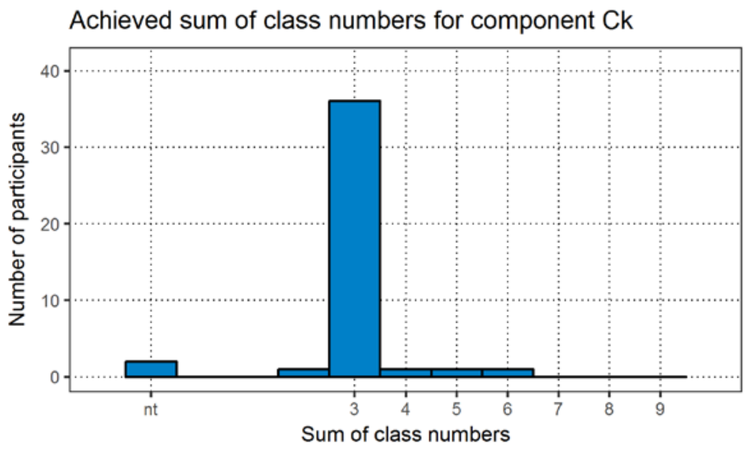

Achieved sum of class numbers for component V
Number of participants
0
10
20
30
40
nt
3
4
5
6
7
8
9
Sum of class numbers

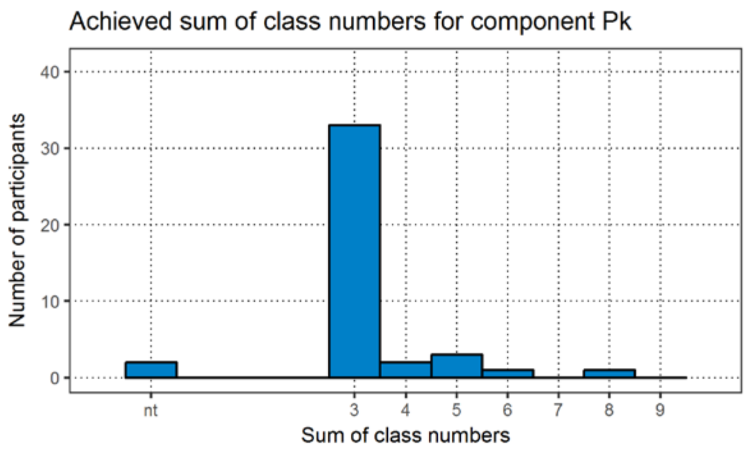
6.2.2 Substance Range G

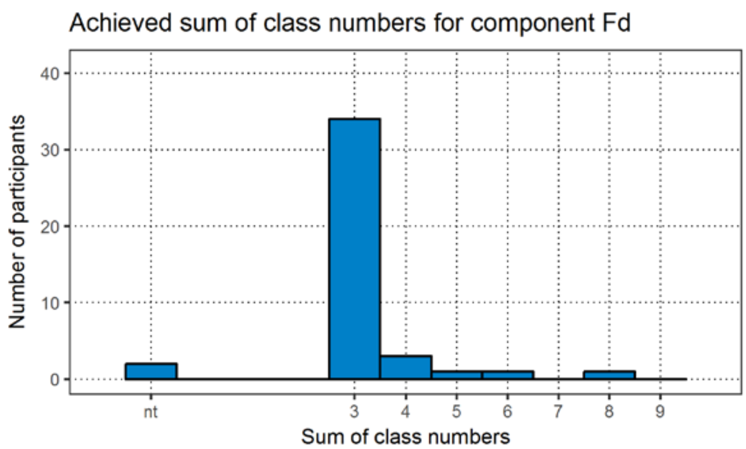


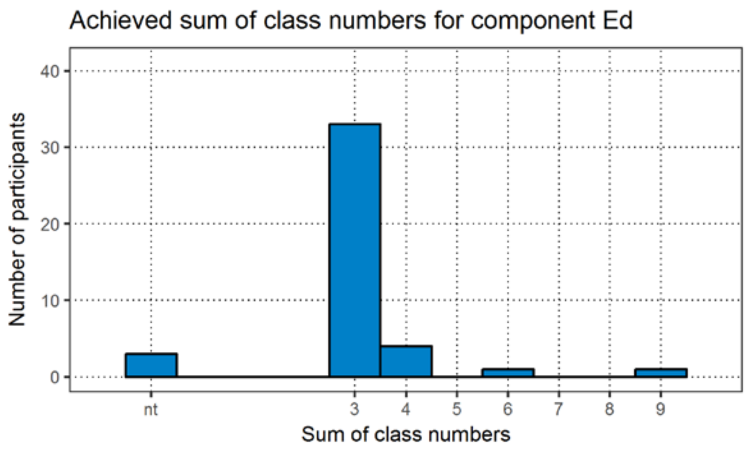


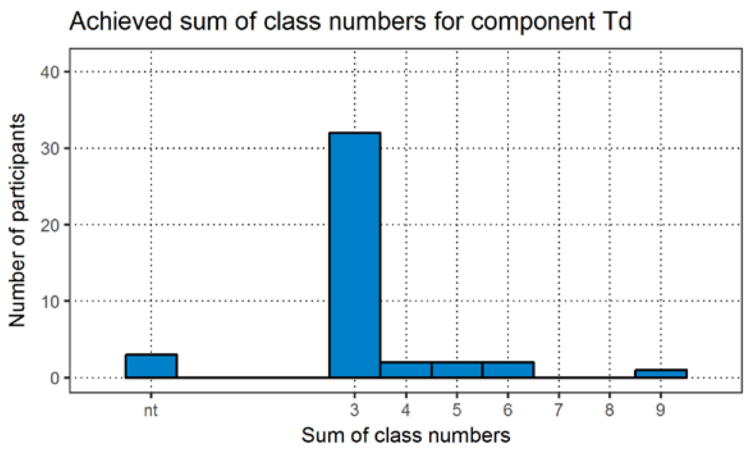


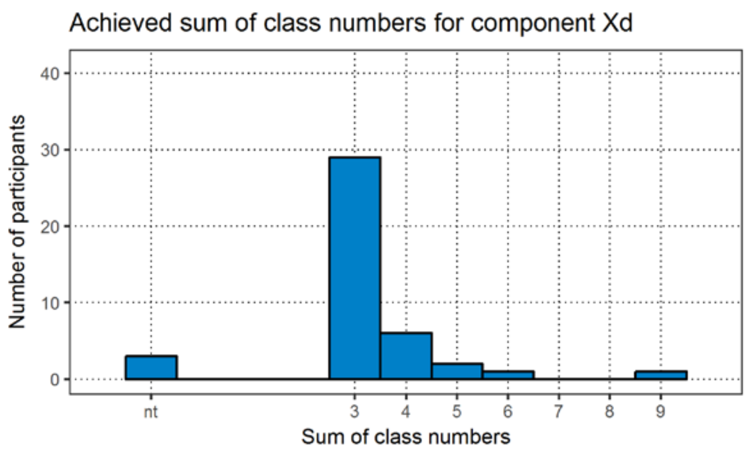

Achieved sum of class numbers for component Ck
Number of participants
40
30
20
10
0
nt
3
4
5
6
7
8
9
Sum of class numbers


Achieved sum of class numbers for component Pk
Number of participants
40
30
20
10
0
nt
3
4
5
6
7
8
9
Sum of class numbers


Achieved sum of class numbers for component Fd
Number of participants
40
30
20
10
0
nt
3
4
5
6
7
8
9
Sum of class numbers

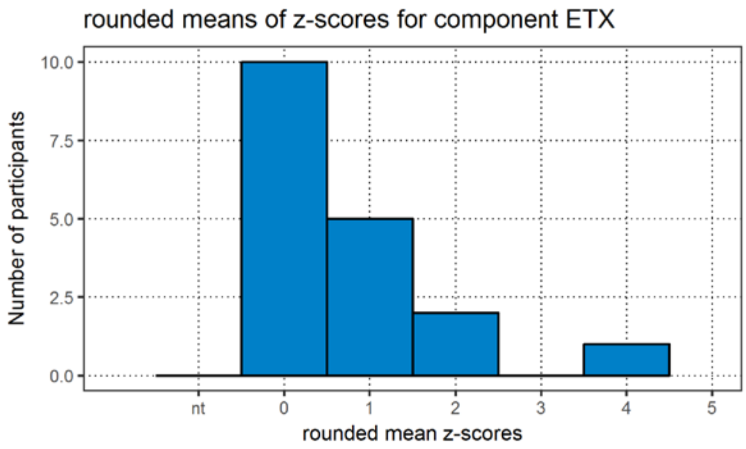

Achieved sum of class numbers for component Ed
Number of participants
Sum of class numbers
0
10
20
30
40
nt
3
4
5
6
7
8
9

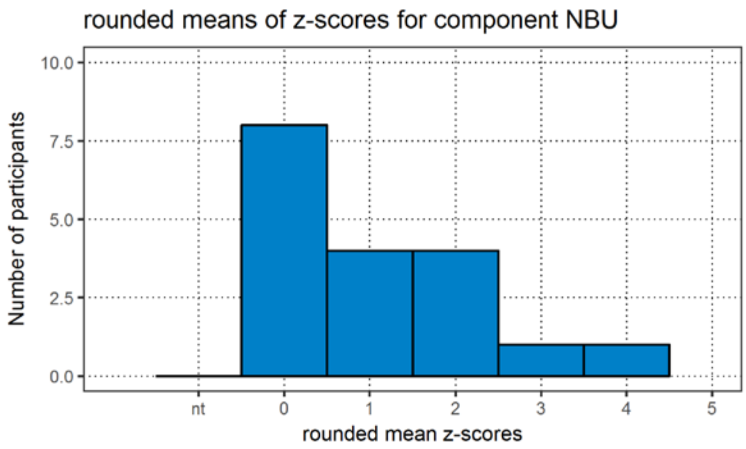

Achieved sum of class numbers for component Td
Number of participants
Sum of class numbers
0
10
20
30
40
nt
3
4
5
6
7
8
9

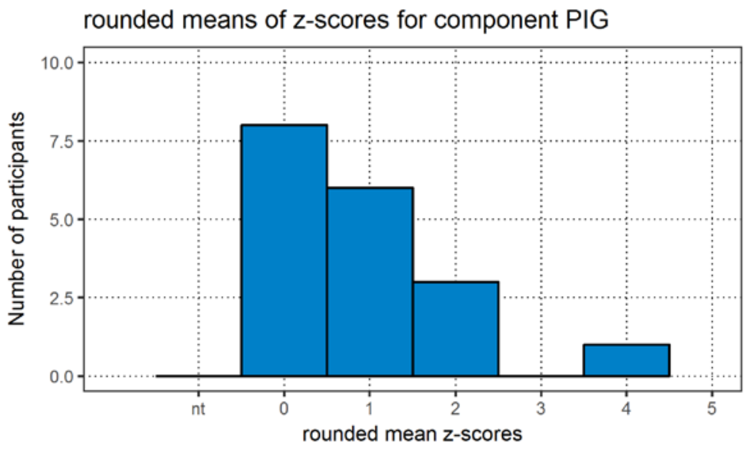

Achieved sum of class numbers for component Xd
Number of participants
Sum of class numbers
0
10
20
30
40
nt
3
4
5
6
7
8
9

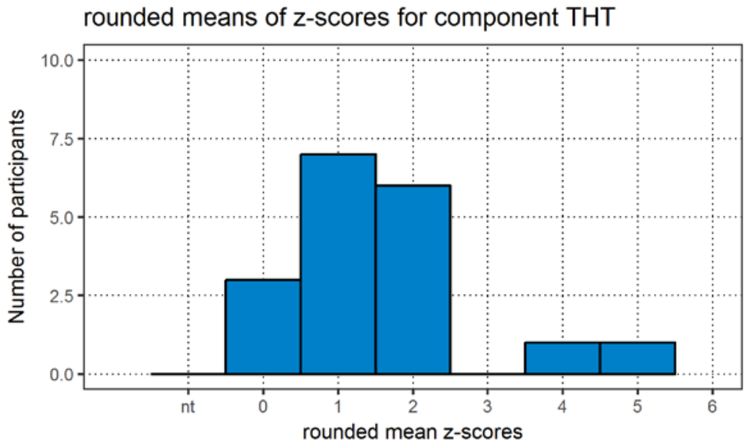
6.2.3 Substance Range O

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







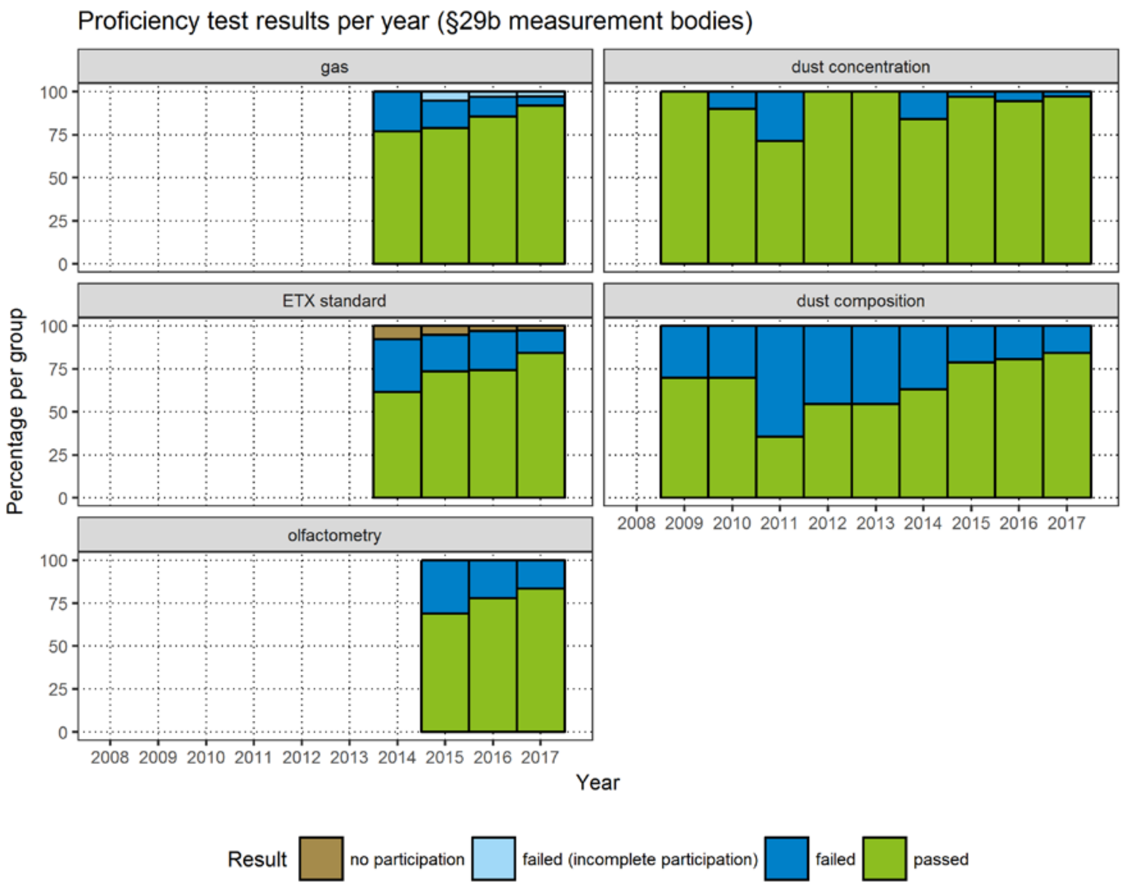


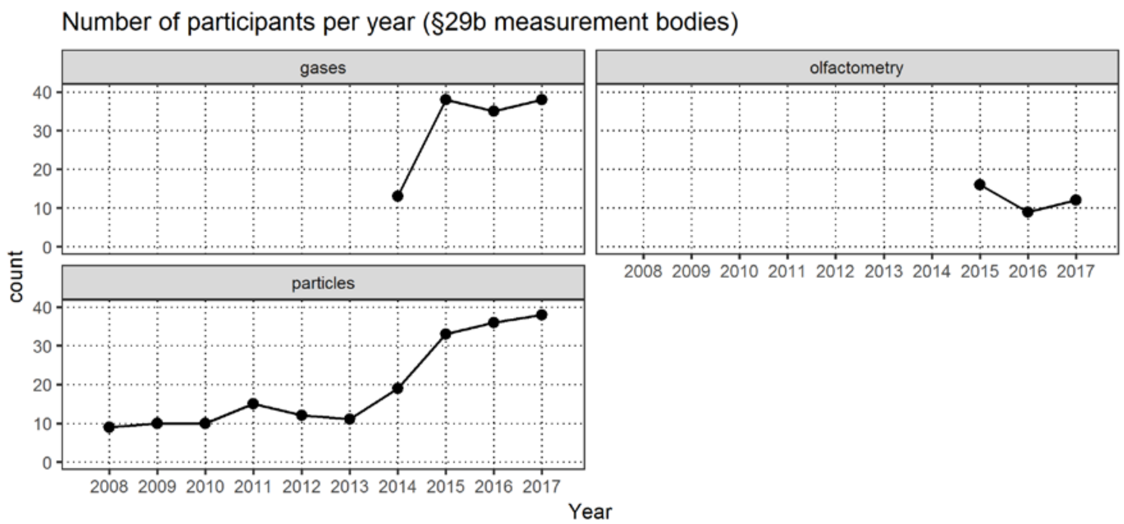
6.3 Component Group Results

43 institutes participated in the (total) dust measurements. 40 of them passed this proficiency test section. 43 institutes took part in the component group “dust composition”. 33 of them passed this section of the proficiency test. 6 institutes took part in the follow-up analysis. 3 of them passed this analysis. 42 institutes participated in the gas emission proficiency test. 36 of them passed this proficiency test section.

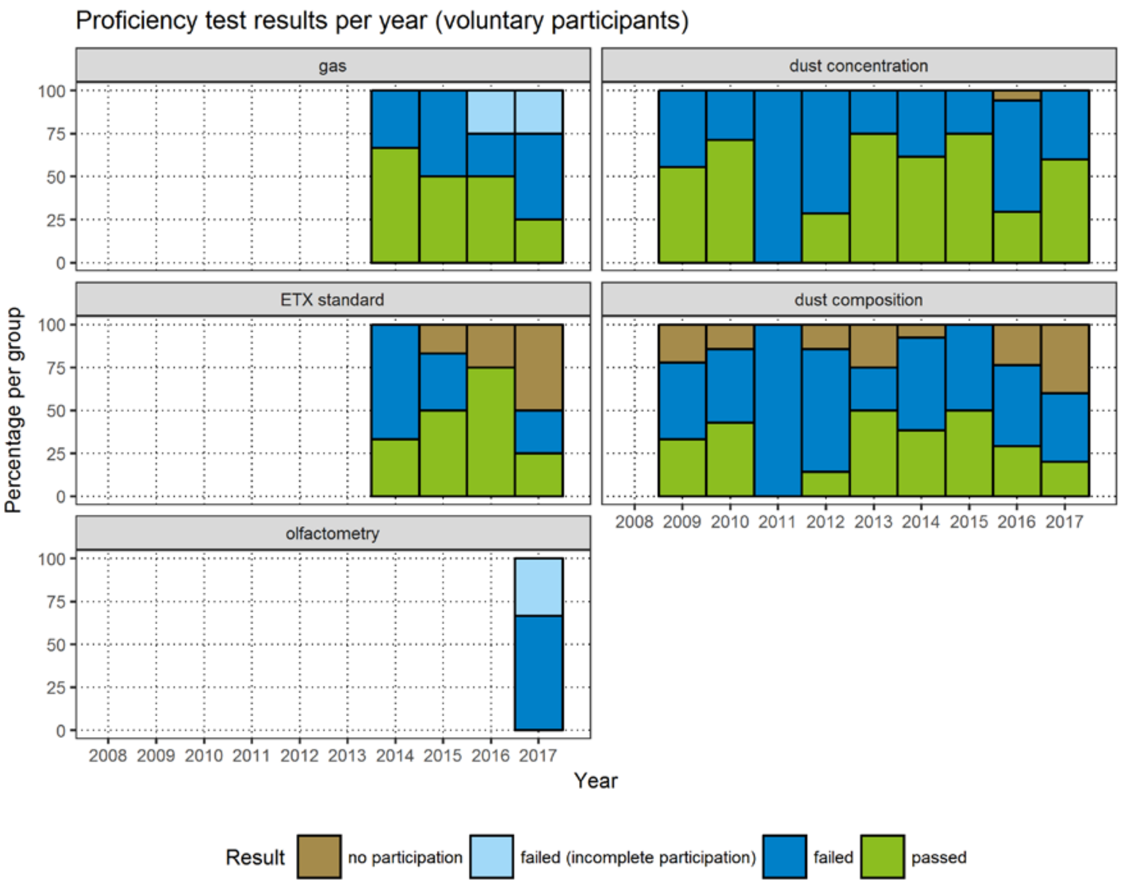
A breakdown of the results by proficiency test round must unfortunately be dispensed with at this point. Since the participants in a proficiency test know the identity of the other participants, participants could with some effort obtain the ID code of another participant without their knowledge or consent if the results were broken down by proficiency test round. To prevent this, the German accreditation body DAkkS asked the HLNUG not to establish a connection between results, ID codes and proficiency test rounds in public reports.

7. Interpretation





In the gas proficiency test, it should be noted that the evaluation scheme changed fundamentally in 2014. In the diagrams in this report only the participations according to the new rules are shown, older participations are not considered.



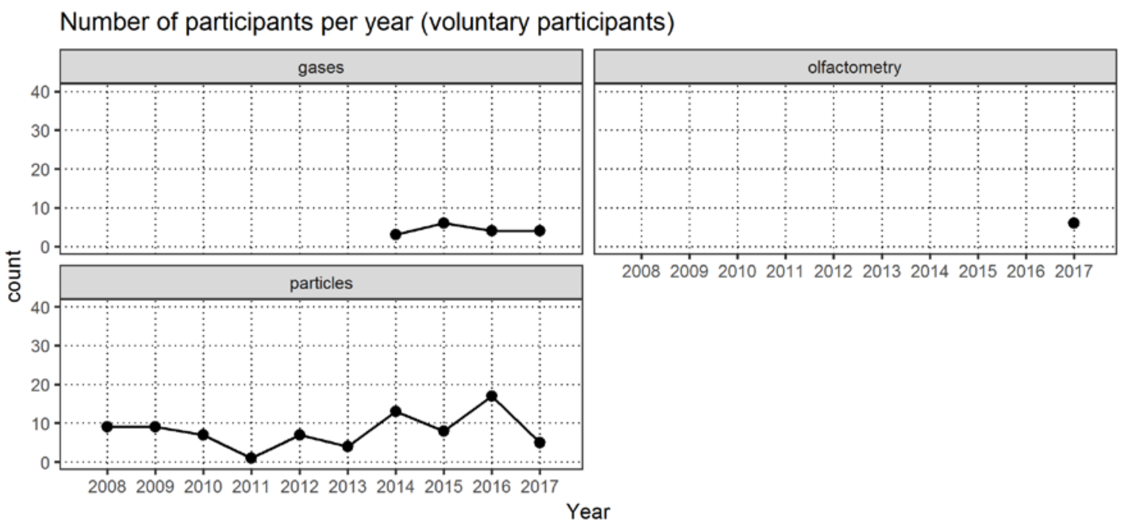


Table 3: Overview of results since 2015 (§29b-laboratories)

year	components group	passed	failed	failed (incomplete participation)	no participation
2015	ETX-standard	28	8	-	2
	gas	30	6	2	-
	odour	11	5	-	-
	dust (total)	32	1	-	-
	dust composition	26	7	-	-
	dust composition (post analysis)	6	-	-	-

year	components group	passed	failed	failed (incomplete participation)	no participation
2016	ETX-standard	26	8	-	1
	gas	30	4	1	-
	odour	7	2	-	-
	dust (total)	34	2	-	-
	dust composition	29	7	-	-
	dust composition (post analysis)	4	1	-	-
2017	ETX-standard	32	5	-	1
	gas	35	2	1	-
	odour	10	2	-	-
	dust (total)	37	1	-	-
	dust composition	32	6	-	-
	dust composition (post analysis)	3	2	-	-

Table 4: Overview since 2015 (voluntary paricipants)

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

7.1 Evaluation of Measurement Results

§29b-Measuring Bodies

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

In the dust proficiency test, the results of the 29b measuring bodies 2017 are at a consistently high level; 37 out of 38 participants (97%) were in the proficiency test section „dust concentration“ successful. The positive trend of the last few years with regard to „dust composition“ has continued; 32 of 38 participants (84%) passed this proficiency test part at the first attempt; another 3 participants (8%) passed this proficiency test part via the follow-up analysis. Overall, 92% of the participations in the dust proficiency test were successful.

35 of 38 (92%) of the §29b measuring bodies passed the gas emission proficiency test. One laboratory (3%) did not pass only because it did not measure all mandatory components (incomplete participation). 32 of 37 (86%) of the §29b measuring bodies have successfully completed the analysis of the ETX standard, one measuring body did not participate in this analysis.

The results of the §29b measuring bodies on the odour proficiency tests are comparable with the results of the previous year. 10 out of 12 participants (83%) passed the odour proficiency test.

Voluntary participants

The number of voluntary proficiency test participations varies from year to year, as a rule there are approx. 4 participations in the gas proficiency test and approx. 8 participations in the dust proficiency test. Due to the usually small number of voluntary participations over many years, the collected results for one year are extremely marked by the performance of individual laboratories; a comparison over many years is only partially revealing. In 2017 there were 4 participants in the gas proficiency test, 5 in the dust proficiency test, and for the first time there were any voluntary participants in the odour proficiency test.

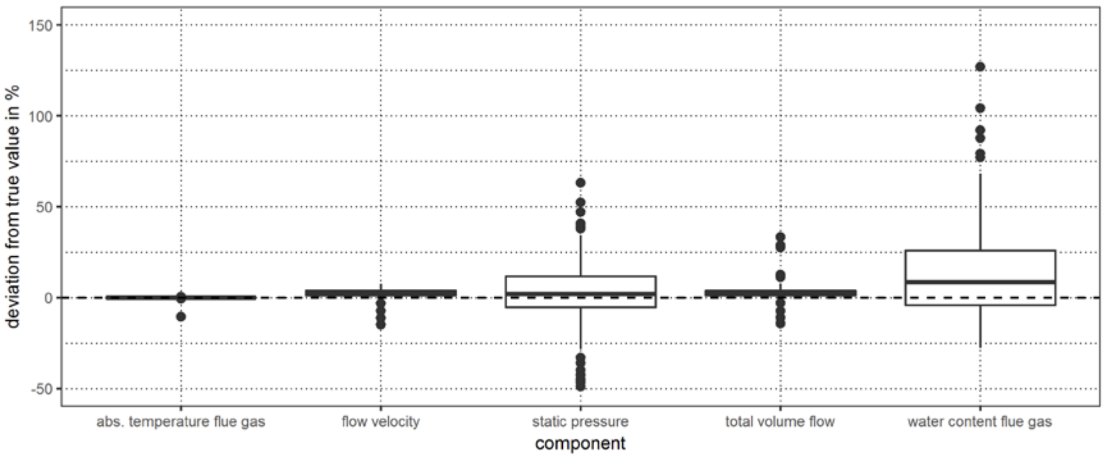
In the „dust concentration“ proficiency test section, a total of 3 out of 5 participations (60%) were successful in 2017. One in three participants (33%) passed the „dust composition“ test section, while two participants did not take part in this part of the proficiency test. One voluntary participant participated unsuccessfully in the follow-up analysis. Overall, one (20%) of the 5 voluntary participations in the dust proficiency test was successful.

In the gas proficiency test, one in 4 (25%) of the voluntary participants passed the proficiency test, whereby one laboratory did not pass simply because it did not measure all mandatory components (incomplete participation). The analysis of the ETX standard solution was successfully completed by one in 2 (50%) of the voluntary participants, 2 laboratories did not participate in this analysis.

In the odour proficiency tests there were a total of 6 voluntary participants, none of whom were rated as "successful". However, it must be pointed out that 2 (33%) of these voluntary participants achieved acceptable measurement results, but did not meet the requirements of the 41st BImSchV, since the olfactometry was not carried out according to VDI 3880. The results of these voluntary participants are therefore not comparable with the other participants; the respective participations were classified as "incomplete participation" and were not "successful" in the sense of the 41st BImSchV.

7.2 Basic 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 2017. 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 5: Absolute relative deviations from true values for basic conditions

	absolute temperature [%]	water vapour concentration [%]	static pressure [%]	flow velocity [%]	volume flow [%]
75th percentile	0.18	26.72	20.25	3.87	4.08
median	0.10	11.98	7.14	2.55	2.53
25th percentile	0.03	5.62	4.16	1.41	1.48
number of values	207	203	196	200	210

7.3 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 obtained are summarized in tables and presented graphically below. The database is based on feedback from participants in 2016 and 2017.

For some components, the participants in the proficiency test have a certain freedom in the choice of different process parameters. Based on the voluntary information provided by the participants, an attempt was made to determine correlations between the methods, equipment, etc. used and the results obtained. Since 9 measurements are always carried out for each component at different concentrations, it is difficult to make a clear statement about the quality of a process. Therefore, correlations to the average z-scores of the participants were established for a simple and clear representation, whereby negative values are also included in the mean value. In addition, similar components such as heavy metals or organic solvents were combined to a common mean value where appropriate. This type of evaluation certainly simplifies the problem and cannot reproduce all the details. For example, different influences in different concentration ranges or high fluctuations between the individual results of a participant are completely ignored in this evaluation. The limitation to the mean values of the z-scores of the participants, however, allows a simple estimation of the effects of different methods on the mean deviation of the measured values from the target value.

7.3.1 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.

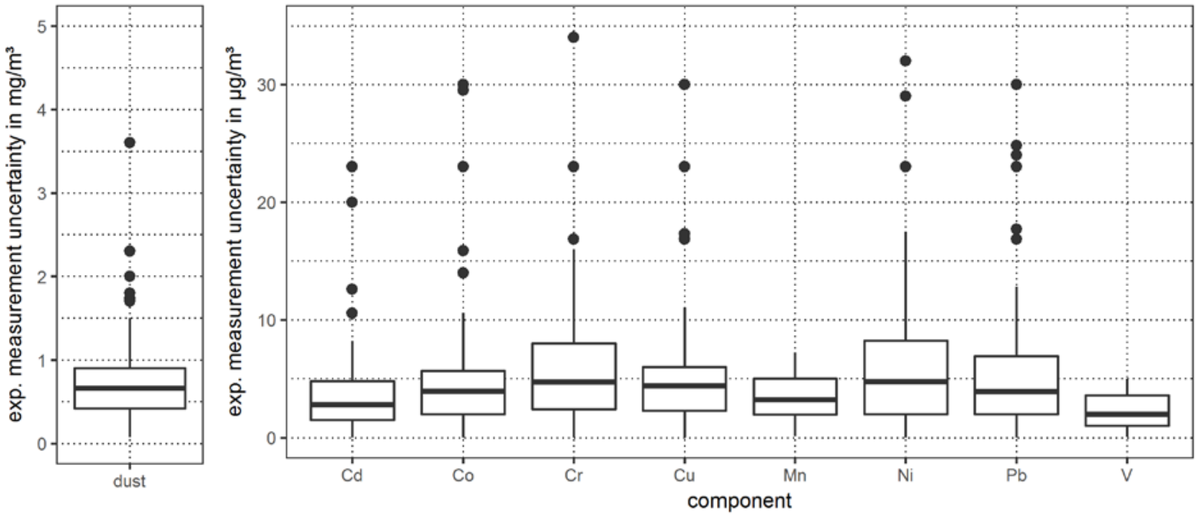


Table 6: Expanded measurement uncertainties reported by participants of the dust proficiency test

	dust [mg/m³]	Cd [µg/m³]	Co [µg/m³]	Cr [µg/m³]	Cu [µg/m³]	Mn [µg/m³]	Ni [µg/m³]	Pb [µg/m³]	V [µg/m³]
75th percentile	0.91	4.83	5.66	8.00	6.00	5.00	8.24	6.92	3.59
median	0.66	2.81	3.96	4.72	4.40	3.23	4.79	3.94	2.00
25th percentile	0.42	1.50	2.00	2.40	2.27	1.95	2.00	2.00	1.00
number of values	61	49	49	49	49	17	49	48	15

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

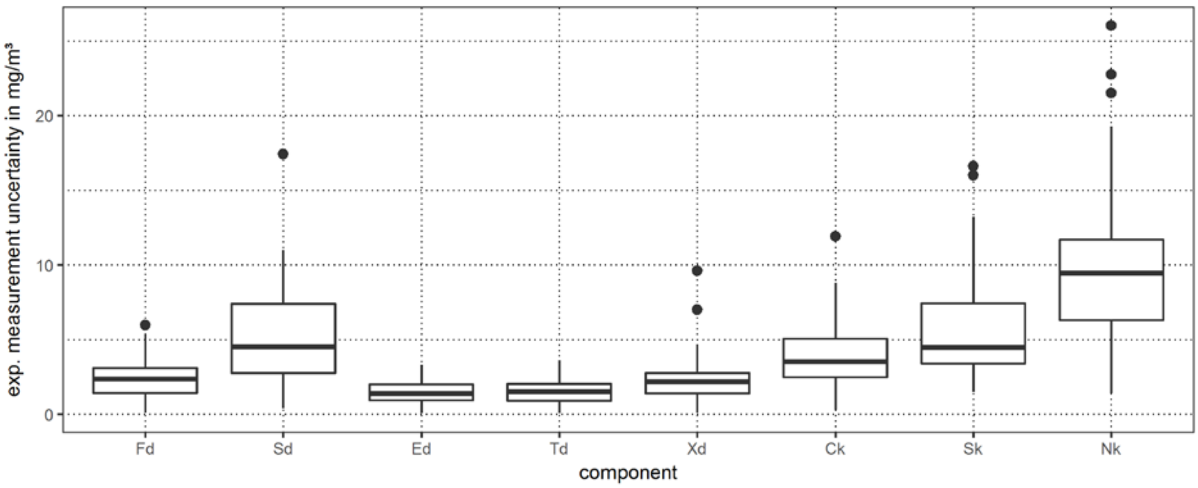


Table 7: Expanded measurement uncertainties reported by participants of the gas proficiency test

	formal-dehyde [mg/m³]	SO_2 (disc.) [mg/m³]	ethyl-benzene [mg/m³]	toluene [mg/m³]	sum of xylenes [mg/m³]	TOC [mg/m³]	SO_2 (cont.) [mg/m³]	NO_x as NO_2 [mg/m³]
75th percentile	3.10	7.40	2.00	2.04	2.78	5.08	7.43	11.71
median	2.36	4.53	1.40	1.53	2.20	3.53	4.50	9.45
25th percentile	1.44	2.75	0.96	0.92	1.40	2.50	3.40	6.30
number of values	48	51	49	49	49	50	46	52

For all information on absolute extended measurement uncertainties, it should be noted that for reasons of comparability, the participants were asked to provide only one value for each method. Therefore, this figure may refer to the highest concentration measured in the proficiency test and would be lower for smaller concentrations. Nevertheless, these values should allow everyone involved to make an approximate assessment of how their own measurement uncertainty relates to the measurement uncertainties of other laboratories.

7.3.2 Probe and Rinsing in Dust Samplings

In the correlation of probe systems and rinsing procedures, the participant field of the dust proficiency tests is essentially divided into 7 groups, depending on whether an in-stack probe with or without gooseneck is used, whether this probe is rinsed after each sampling or not and whether the rinsing residues are taken into account in the measurement result or not. The representation here was limited to the combinations indicated by at least four participants.

In this report, the data basis covers the results from 2016 and 2017 and is thus significantly larger than in the previous year's report. The evaluations for 3 of the 7 combinations of probe system and rinsing procedure are now based on a relatively solid amount of data, the statements for the other 4 combinations are comparatively uncertain.

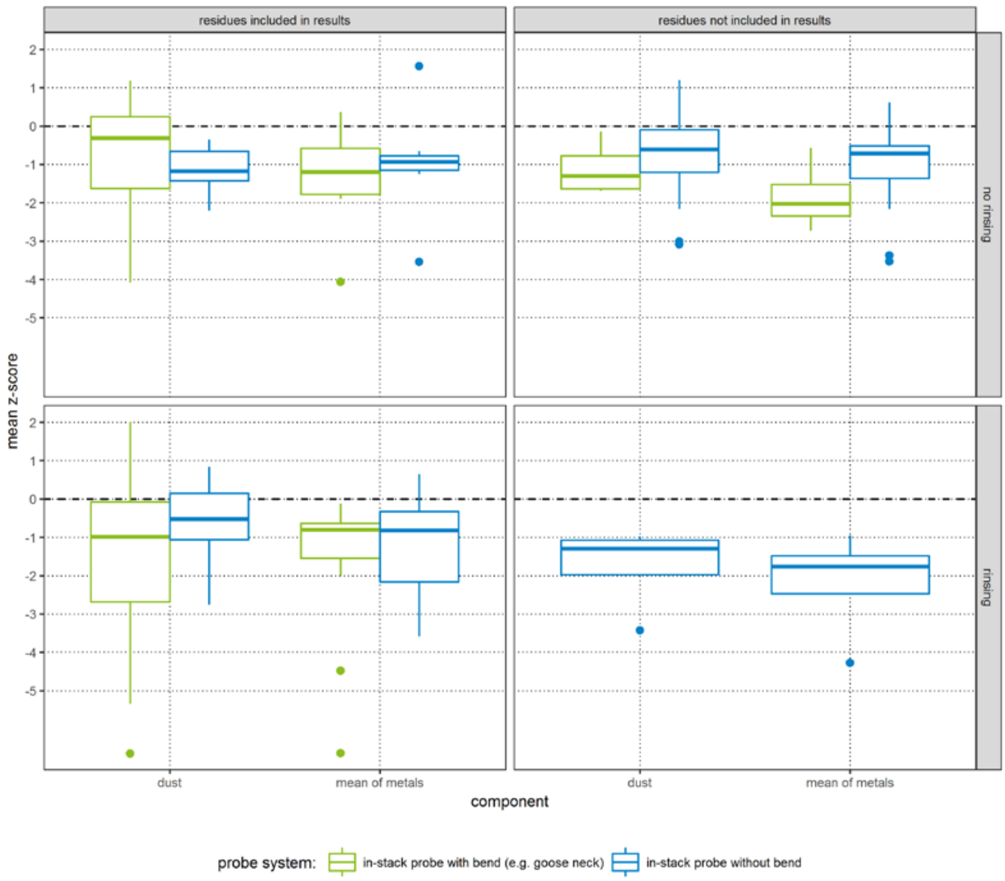


Table 8: Correltaion of dust measurement results with probe systems and rinsing procedures

combi-nation	probe system and rinsing procedure	median of mean z-scores total dust results	number of participants	median of mean z-scores heavy metal results	number of participants
1	in-stack probe without bend, rinsing after each sampling, residiues included in results	-0.52	15	-0.82	14
2	in-stack probe without bend, rinsing after each sampling, residiues **not** included in results	-1.30	4	-1.76	4
3	in-stack probe without bend, **no** rinsing after each sampling, residiues included in results	-1.18	7	-0.94	8
4	in-stack probe without bend, **no** rinsing after each sampling, residiues **not** included in results	-0.61	25	-0.71	24
5	in-stack probe with goose neck, rinsing after each sampling, residiues included in results	-0.99	17	-0.80	13
6	in-stack probe with goose neck, **no** rinsing after each sampling, residiues included in results	-0.31	7	-1.20	7
7	in-stack probe with goose neck, **no** rinsing after each sampling, residiues **not** included in results	-1.30	4	-2.02	4

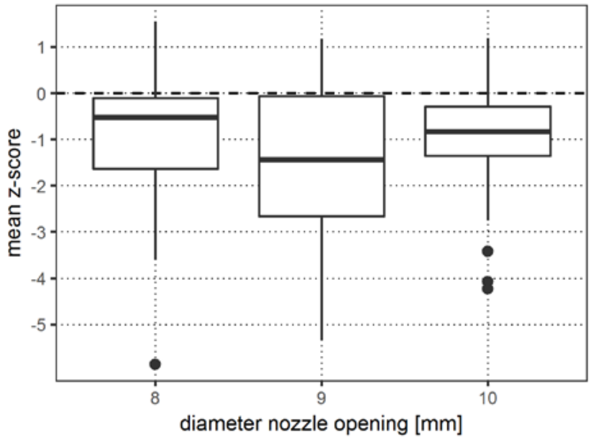
The data provided by the participants indicate that the best results for dust concentrations in the proficiency test is obtained using one of the three following combinations. The overall best results are apparently achieved when using an in-stack probe without bend, if the probe is rinsed after each sampling and the rinsing residues thus obtained are taken into account in the measurement result (combination 1). This procedure delivers results for total dust that are relatively close to the target value and have comparatively little scattering. The deviations in the case of heavy metals are somewhat more scattered here, but also show very slight deviations on average. The combination of system and rinsing procedure used by most participants, the use of an in-stack probe without bend and without rinsing (combination 4), leads to comparable results with slightly higher dispersion. Comparable deviations with similar scattering are achieved in the determination of heavy metal concentrations if a probe with gooseneck is used, this is rinsed after each sampling and the rinsing residues are taken into account in the measurement result (combination 5). However, the measured values for the total dust concentrations show a clear tendency towards results clearly below the target value with a very high dispersion of the measured values.

The three combinations described above were all reported by 13 to 25 participants. The following four combinations were only reported by 4 to 8 participants, the statements are correspondingly less reliable.

Significantly worse results than with the combinations considered so far were achieved by participants with in-stack probes without bend, who stated that they did not rinse after each measurement but nevertheless took rinsing residues into account in the result (combination 3). This means, for example, that rinsing was performed only once per working day and the residues were distributed proportionally to the samples taken up to that point. It is not surprising that results significantly below the target values are achieved when working with an in-stack probe without a bend and rinsing after each measurement, but the residues are not taken into account in the result (combination 2). Participants who use an in-stack gooseneck probe and refrain from rinsing achieved comparably poor results (combination 7). It should be noted that combinations 2 and 7 do not represent standard-compliant procedures in accordance with EN 13284-1. A mixed picture results from the use of a gooseneck probe and a (presumed) daily rinsing with consideration of the residues (combination 6). On average, the best results for total dust are apparently achieved here. However, the values here are also among the most scattered, and the results for heavy metals are also rather below average.

7.3.3 Diameter of the Nozzle Opening in Dust Sampling

The information provided by the participants on the diameter of their probe‘s nozzle opening does not show a clear trend. Regardless of the diameter, the measured values for the total dust concentration always seem to spread over a wide range.



Nozzle opening diameters mentioned by only 3 or fewer participants are not listed here. In the following table, the values were evaluated based on the absolute values of the z-scores.

Table 9: Correlation of absolute means of z-scores for total dust with nozzle opening diameters

diameter of nozzle opening	8 mm	9 mm	10 mm
75th percentile	1.63	2.66	1.35
median	0.83	1.43	0.91
25th percentile	0.26	1.19	0.37
number of values	21	6	52

Here, too, there is no clear connection. Only the scattering of the results seems to be somewhat smaller for users of 10 mm probe openings than for other participants.

7.3.4 Analytical Instruments for Heavy Metals

The information provided by the participants on the analytical instrument used for heavy metal analysis shows a certain difference between AAS and ICP users. A total of 11 participants stated that their heavy metal analysis was carried out using AAS instruments (including 5 flame-AAS and 6 graphite furnaces). On average, all participants achieved comparable z-scores for the heavy metals, irrespective of the analysis device used. However, the measured values of ICP users scatter much more than those of AAS users. In addition, ICP users seem to be more prone to massive underreporting than users of AAS devices.

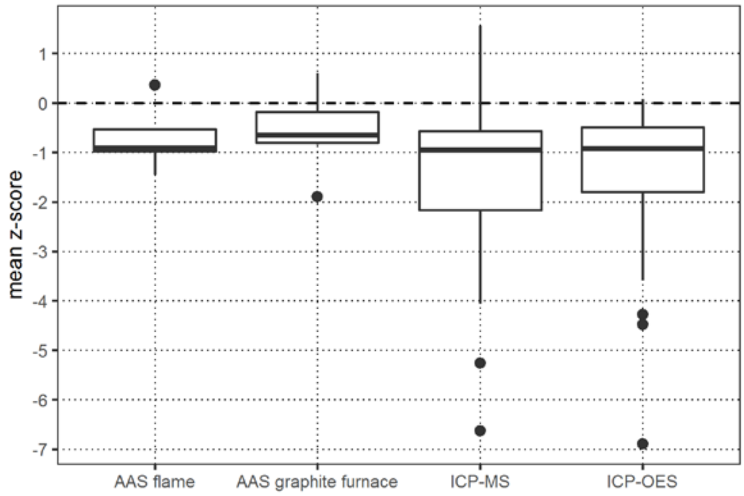


Table 10: Correlation of heavy metal results and analysis devices

analysis device	flame-AAS	graphite furnace AAS	ICP-MS	ICP-OES
75th percentile	-0.53	-0.19	-0.57	-0.49
median	-0.91	-0.65	-0.95	-0.92
25th percentile	-0.98	-0.81	-2.17	-1.80
number of values	5	6	41	26

In this correlation - as in all the other correlations presented in this report - it goes without saying that a correlation is only an indication of a connection, but by no means proves a causality. For example, it is quite conceivable that the participants using an AAS device might happen to have other similarities that actually affect the measurement results, while the analysis device actually plays no role at all.

7.3.5 Formaldehyde

For the measurement of formaldehyde concentrations, participants can choose from the guidelines VDI 3862 Parts 2, 3 and 4. The following picture emerges from the information provided by the participants:

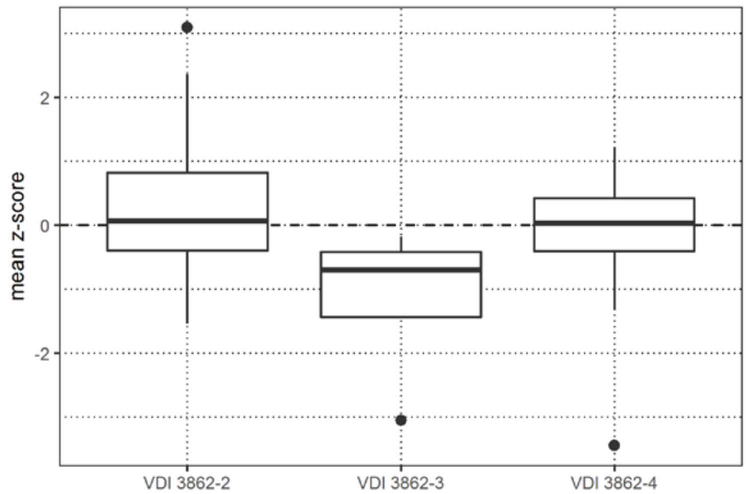


Table 11: Correlation of formaldehyde measurement results with the guidelines used

guideline	median of mean z-scores formaldehyde results	number of participants
VDI 3862 Part 2 (DNPH washing bottles)	0.07	43
VDI 3862 Part 3 (DNPH cartridges)	-0.70	4
VDI 3862 Part 4 (AHMT-procedure)	0.03	24

The DNPH washing bottle procedure apparently provides on average comparable good values to the AHMT procedure, but spreads over a much wider range. The DNPH cartridge method was only given by 4 participants, all had rather disappointing results.

7.3.6 Sulfur dioxide

For the discontinuous determination of sulfur 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:

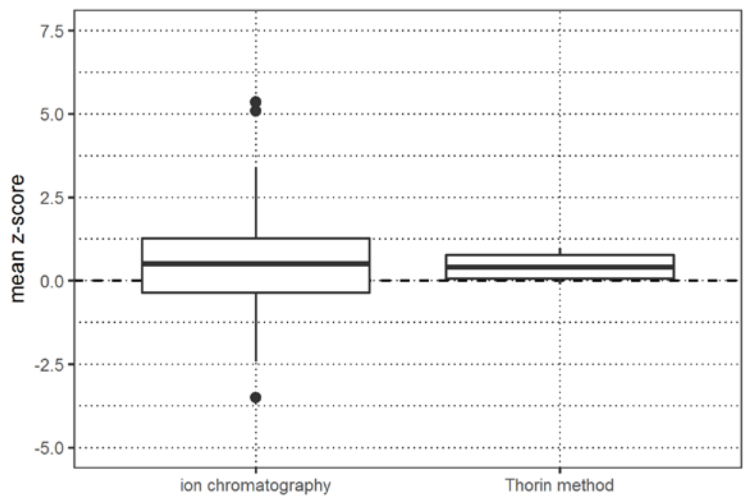


Table 12: Correlation of sulphur dioxide measurement results with the analytical method used

method used	median of mean z-scores discontinuous SO_2 -results	number of participants
Ion chromatography	0.51	68
Thorin method	0.70	5

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.

7.3.7 Solvent for Desorption of ETX

For the desorption of the solvents ethylbenzene, toluene and xylene (ETX), the participants can also choose other solvents or solvent mixtures in addition to the usual solvent carbon disulfide. The overwhelming majority of the participants stated that they had worked with CS_2. The results of these participants are on average close to the target value, but also vary relatively strongly. Participants with other desorption solvents achieved slightly better results with slightly less dispersion.

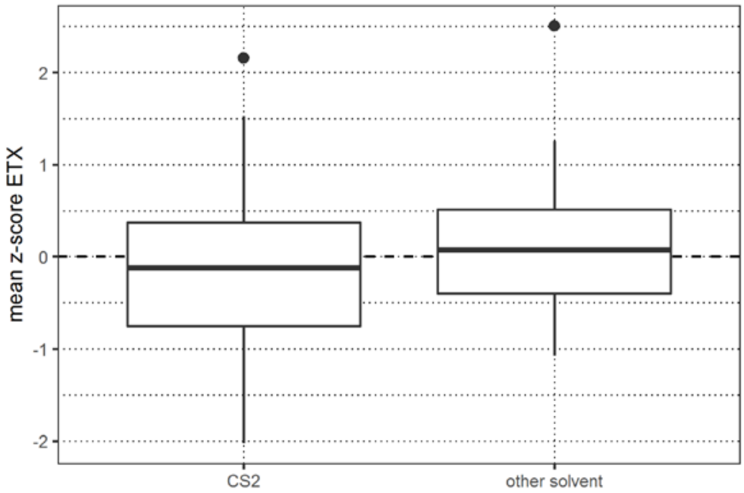


Table 13: Correlation of ETX measurement results with the desorption solvent

solvent used in desorption	median of mean z-scores ETX results	number of participants
CS_2	-0.12	59
other solvent	0.03	10

7.3.8 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:

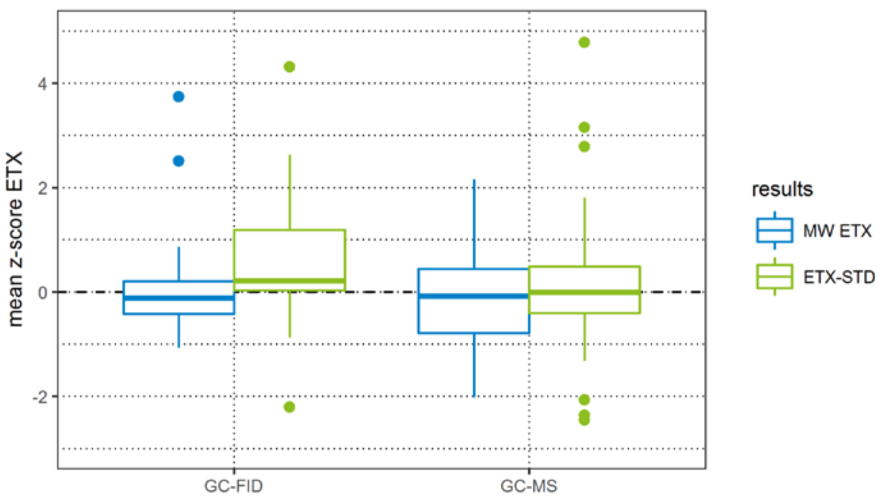
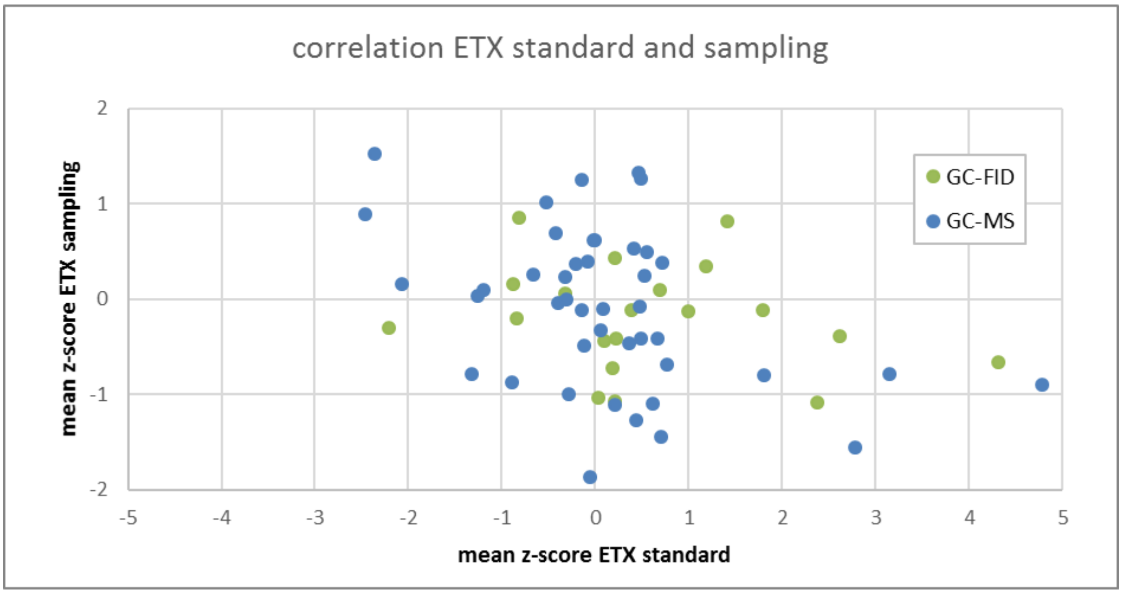


Table 14: Correlation of ETX measurement results with analytical instruments

analytical instrument	median of mean z-scores ETX including sampling	median of mean z-scores ETX standard solution	number of participants
GC-FID	-0.12	0.22	23
GC-MS	-0.08	-0.02	47

The results here are surprisingly inconsistent. For the overall method of sampling and analysis, the participants achieved comparable results with both detector variants close to the target value, although the measured values of the GC-MS users scatter somewhat more than those of the GC-FID users. In analysing the ETX standard, GC-MS users achieve on average results equivalent to those obtained from sampling. However, GC-FID users apparently tend to have measurement results far above the true values in some cases. It is also surprising that there does not appear to be a clear correlation between the results of sampling measurements and those of the pure analytical standard, as the following figure shows.



A high error in the results with sampling and a small error in the standard (points along the y-axis) can be explained by a combination of sampling errors and flawless analysis. The results with small errors in sampling but high errors in the standard (points along the x-axis) can only be explained by an error-free execution of the overall procedure (sampling + analysis) with simultaneous errors in the pure analysis of the standard. Here, for example, an incorrect calculation of desorption rates would be conceivable. Overall, however, all conceivable combinations of positive and negative findings can be observed in the data, relatively independent of the detector used.

8. Concluding Remark

Since 2015, the participation frequency of the authorized measuring bodies has increased significantly compared to previous years due to the systematic and regular participation of all locations. The results registered since then show a clear positive effect on the quality of the measurement results. Nevertheless, many measurement results are still far outside the acceptable range. This shows that regular participation in stack emission proficiency tests is still important in order to detect faulty trends at an early stage and to maintain the quality of emission measurement results in the regulated sector at a consistently high level.

Kassel, 18 June 2018

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