IFA
Institut für Arbeitsschutz der
Deutschen Gesetzlichen Unfallversicherung

Proficiency testing for measuring of hazardous substances 2020

Proficiency testing for in-house and external measuring stations

Information for participants

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1 Introduction

During performance of a risk assessment, it is frequently necessary for the concentration of a hazardous substance in a workplace atmosphere to be measured. Under the German Occupational Safety and Health Act, overall responsibility for identifying and assessing hazards presented by hazardous substances at the workplace lies with the employer [1]. The measurements of hazardous substances required for this purpose may be performed by either internal (company) or external measuring stations.

A measuring station may be regarded as suitable if it possesses the necessary expertise and facilities. The suitability of external measuring stations is assured by accreditation. No provision is made for formal accreditation of inhouse measuring stations. They must however satisfy the requirements of Annex 1 of the Technical Rules for Hazardous Substances TRGS 402 [2]. To ensure that the quality parameters of analysis methods are observed, both internal and external measuring stations must employ quality assurance methods according to the state-of-the-art.

Participation in proficiency testing (PT) schemes can support quality assurance for measuring stations [3]. The Institute for Occupational Safety and Health of the German Social Accident Insurance (IFA) organises such PT schemes in co-operation with the Bundesverband der Messstellen für Umwelt- und Arbeitsschutz e.V. (BUA), the German association of environmental and OSH measurement bodies. The BUA recommends regular participation in these IFA's PT schemes.

Where possible, proficiency testing schemes are offered in which the entire analysis method can be tested. All PTs are conducted in accordance with the requirements of DIN EN ISO/IEC 17043 [4] and DIN ISO 13528 [5]. Participants will receive a detailed evaluation and a certificate of participation (see Annex).

2 Organisation of the proficiency testing schemes

Organisation and performance of the PT schemes, including evaluation and documentation of the results, are conducted by the IFA. For 2020, the PT schemes offered are intended to cover the widest possible range of hazardous substances. Participants can order ready-loaded samples or they can load their samples themselves at the dynamic test gas stream at the IFA. The PT schemes metals and inorganic acids (HCl, HNO_3, H_3PO_4, H_2SO_4) will be offered only as ready-loaded samples. For the PT scheme VOC the participants are required to prepare conditioned thermal desorption tubes and send them to the IFA for loading.

According to DIN EN 482 the minimum measuring range for a workplace air measurement method has to cover a range normally from 0.1 times to 2 times the occupational exposure limit value [6][7]. The adjusted concentration range in the PT schemes corresponds to this requirement. Information on the occupational exposure limit values are given in the database "GESTIS – International limit values" [8].

PT schemes are conducted once a year and always follow a similar procedure (see Fig. 1).

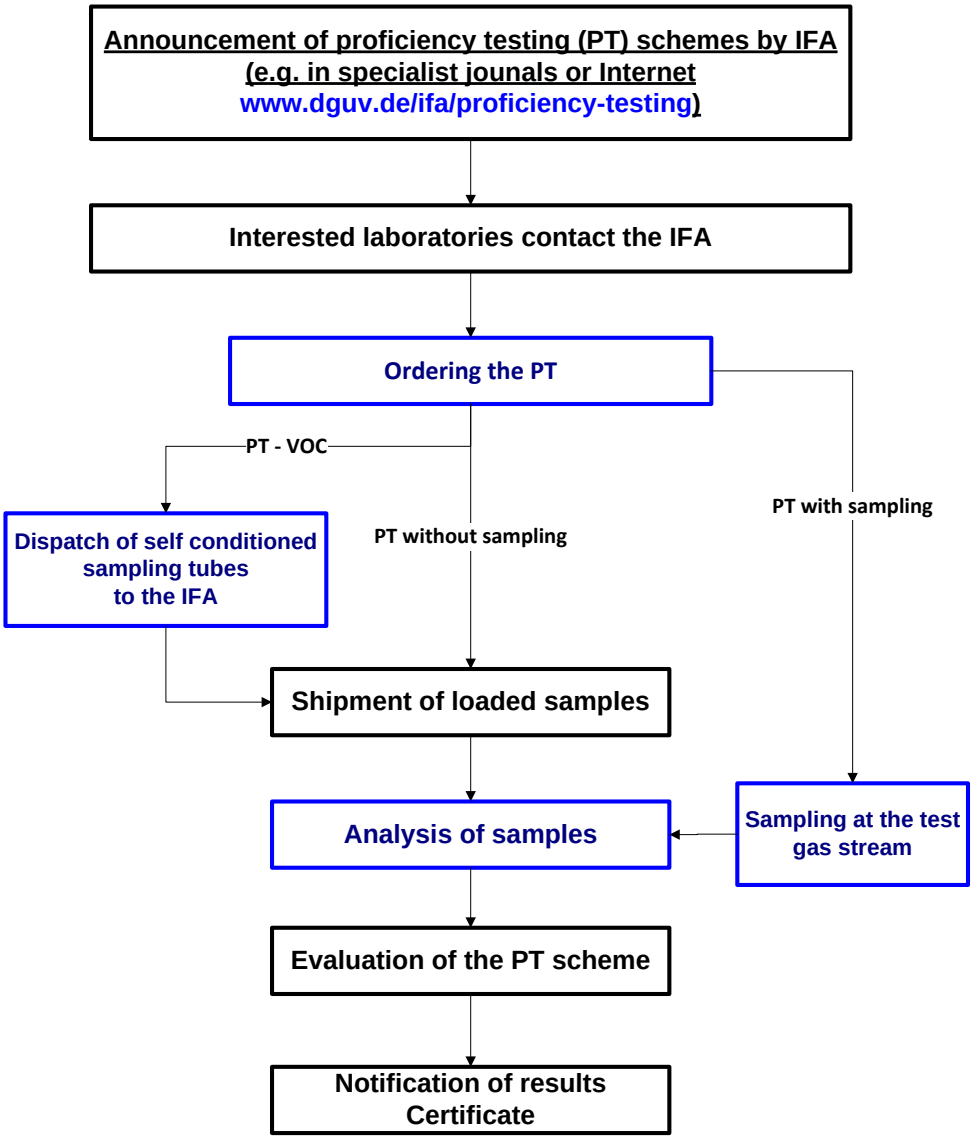


Fig. 1: Flow chart for IFA proficiency testing (PT) schemes

The following proficiency testing schemes (PT) are planned for 2020:

1. **PT Organic solvents**
   Date: -*
2. **PT Organic solvents with own sampling**
   Date: -*
3. **PT Inorganic acids (HCl, HNO_3/H_2SO_4, H_3PO_4)**
   Date: March 2020
4. **PT Inorganic acids with own sampling (HCl, HNO_3/H_2SO_4, H_3PO_4)**
   Date: -*
5. **PT Metals on filters**
   Date: April 2020
6. **PT Volatile organic compounds (VOC) with thermal desorption**
   Date: September 2020
7. **PT Volatile organic compounds (VOC) with thermal desorption with own sampling**
   Date: 01 to 02 September 2020
   Venue: IFA, Sankt Augustin
8. **PT Aldehydes**
   Date: November 2020
9. **PT Aldehydes with own sampling**
   1st series of experiments: 10 to 11 November 2020
   2nd series of experiments: 17 to 18 November 2020
   Venue: IFA, Sankt Augustin

*** At the end of 2019 the chemical and biological analytics section of the IFA moved into a new building. There, an enlarged and technically improved dynamic test gas stream will be set up. Due to a delay in delivery the PT schemes Organic solvents without and with own sampling and the PT scheme Inorganic acids with own sampling cannot be offered in 2020.**

There is a limited number of sample ports to load the sample tubes. Upon reaching the maximum number of participants we reserve the right to terminate the registration prematurely. On request you can be put on the waiting list if a PT is fully booked.

Please consider the registration deadlines indicated on the order forms.

Table 1 gives an overview of the fees charged for participation.

Table 1: Participation fees

Proficiency testing	Participation fee in €	Plus postage & packing
Inorganic acids (HCl, HNO_3/H_2SO_4, H_3PO_4)	505,-	see below
Metals on filters	470,-	see below
Volatile organic compounds (VOC) with thermal desorption	470,-	see below
Volatile organic compounds (VOC) with thermal desorption with own sampling	365,-	–
Aldehydes (including cartridges)	530,-	see below
Aldehydes (with own sampling)	365,-	–

Our prices do not include VAT and shipping costs.

Experience has shown that there were sometimes delays in sample shipping. For this reason the PT samples will be sent only by express. Postage and packing will be charged additionally (Table 2).

Table 2: Postage and packing fees

Region	Postage and packing in €
Germany and EU	15,-
non EU-countries	30,-
Overseas	50,-

Shipping costs will be indicated in the invoice separately.

The costs arising for the PT schemes are paid directly to the IFA.

Full information on the IFA's PT schemes is available on the Internet for interested parties and participants. The information can be found at www.dguv.de/ifa/proficiency-testing, and provides order forms for download. Complete and up-to-date information for participants is available here on short call.

3 Offered proficiency testing schemes

Information about the chemicals which are used for the preparation of the PT samples is available online.

3.1 Organic solvents

At the end of 2019 the chemical and biological analytics section of the IFA moved into a new building. Due to a delay in delivery of the enlarged dynamic test gas stream the PT schemes organic solvents and organic solvents with own sampling **cannot be offered in 2020.**

3.2 Inorganic acids (HCl, HNO_3, H_3PO_4, H_2SO_4)

The PT scheme for volatile inorganic acids (HCl, HNO_3) **with own sampling cannot be offered in 2020.**

The PT for inorganic acids consists of two parts:

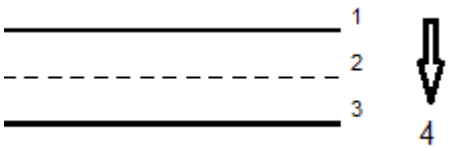
- Volatile inorganic acids: HCl, HNO_3

  Loading of the sample carriers for volatile inorganic acids is performed at the still existing dynamic test gas stream of the IFA. Alkaline impregnated quartz fibre filters are employed as sample carriers (figure 2).

- Non-volatile inorganic acids: H_3PO_4, H_2SO_4

  As sample carriers quartz-fibre filters are loaded with sulphuric acid and phosphoric acid. The samples are produced by means of piezoelectric microdispensing.

  After loading the filters are stabilised in 4 mL desorption solution c (Na_2CO_3) = 3.1 mmol/L; c ($NaHCO_3$) = 0.35 mmol/L).



1: pre-filter: salts of the acids are trapped on the pre-filter

2: spacer: to separate the filters

3: **sampling filter:** **alkali-impregnated quartz fiber filter**

This filter is to be analysed!

4: direction of air flow through the sampler

Fig. 2: Structure of the filter combination in the filter cassette for volatile inorganic acids

The concentrations to be measured for volatile and non-volatile inorganic acids will lie within the range between 1/10 and two times the AGW[1] value.

Since there is only a short-time limit value for nitric acid, the concentration of one test gas is in accordance with this limit value. The concentrations of the other two test gases are in accordance with the limit value of hydrochloric acid.

For HCl and HNO_3, the IFA recommends the analysis method for hazardous substances described in IFA Folder No. 6172 [9], DFG [10] or NIOSH [11].

For H_2SO_4 and H_3PO_4, a procedure according to IFA Folder 6173 [12], DFG [13] method or NIOSH [14] is recommended.

Ion chromatography should be employed for analysis.

3.2.1 Proficiency testing without own sampling

Each participant receives two sampling kits comprising of:

One sampling kit for HCI and HNO_3 comprising of

– Three loaded sample carriers (quartz fibre filters), and

– Two unloaded sample carriers for blank value adjustment.

In addition the participants will receive a sample kit for H_2SO_4 and H_3PO_4 comprising of

– Three loaded filters stabilised immediately after loading

– Two unloaded stabilised filters for blank value adjustment.

3.3 Volatile organic compounds (VOC) with thermal desorption

The PT is offered alternatively with and without own sampling. Participants may choose whether they wish to perform sampling for volatile organic compounds (VOC, Table 3) themselves, or to have their self conditioned sample carriers loaded by the IFA supplied to them [15].

The concentration of the individual substances to be measured will lie within the range between 10 to 200 µg/m³ for each substance. The concentrations of benzene will lie close to the acceptance concentration of 0.2 mg/m³ (60 ppb) [16].

The PT scheme consists of two runs with different concentrations of the same substances within the test gas. The test gas will contain up to ten different substances (see Table 3).

To exclude blank values in the basic gas and the test gas stream a blank value will be taken at the beginning of a test series.

Analysis has to be conducted by gas chromatography by means of thermal desorption according to DIN EN ISO 16017-1 [17], DIN EN ISO 16000-6 [18] or IFA Folder No. 8936 [19].

For benzene you can use the method according to IFA-Folder 6265, measurement method No. 2 [20].

[1] The German acronym AGW stands for Arbeitsplatzgrenzwert (Occupational Exposure Limit value – OEL). For carcinogenic substances and substances without AGW the database GESTIS – International limit values for chemical agents http://www.dguv.de/ifa/gestis-limit-values is used.

Table 3: Examples of substance selection in the PT scheme for VOC with thermal desorption

Substance group	Substances
Alkanes	n-decane, n-dodecane, n-heptane, n-hexadecane, n-hexane, n-nonane, n-octane, pentadecane, n-tetradecane, n-tridecane, n-undecane
Alcohols	butan-1-ol, 2-ethyl-1-hexanol
Aromates	benzene, 3-carene, cumene, ethylbenzene, limonene, α-pinene, toluene, 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, 1,3,5-trimethylbenzene, o-, m-, p-xylene
Esters and Ethers	2-butoxyethanol, 2-(2-butoxyethoxy)-ethyl acetate, 2-(2-butoxyethoxy)ethanol, 2-butoxyethyl acetate, n-butyl acetate, 2-ethoxyethyl acetate, ethyl acetate, 1-methoxy-2-propanol, 2-phenoxyethanol
Ketones and Aldehydes	butan-2-one, hexanal, acetophenone, 4-methyl-2-pentanone
Siloxanes	hexamethylcyclotrisiloxane, octamethylcyclotetrasiloxane

The substances to be analysed will be indicated to the participants in time.

The selected substances are related to indoor air environments (see Table 3).

The quantitative evaluation should be based on the substance, so please do not quote the results as toluene equivalent [21] .

Correction for the blank value:
In the IFA's experience, the blank value of the test gas stream can generally be ignored. However, substances which may simulate a blank reading are detected sporadically and non-reproducibly when using thermal desorption tubes. Several blank value samples are taken by the IFA for each test gas. If these samples show a significant blank value caused by the test gas stream, the participants are informed immediately via e-mail. Correction should be made for the blank value against participants' blank samples only if the detected substance concentrations can be attributed clearly to the participants' tubes.

3.3.1 Proficiency testing with own sampling

Up to 27 participants can carry out parallel sampling at the dynamic test gas stream at the IFA (figure follows).

The PT scheme consists of two runs with different concentrations of the same substances within the test gas. Each test will last 1.5 hours plus preparation time and run-in period.

The participants will receive detailed information about the PT scheme, a description of the apparatus, in particular of the sampling ports and a list of the substances to be analysed, in good time.

3.3.2 Proficiency testing without own sampling

The production of samples is conducted at the dynamic test gas stream of the IFA.
The particular feature of this PT scheme is that thermal desorption tubes (e.g. Tenax-TA) are employed for loading which must be conditioned by the participants prior to the PT.
Each participant (analysis laboratory) submits seven self conditioned thermal desorption

tubes to the IFA. Only personal air samplers are used at the IFA. The sample carrier should not generate a back pressure exceeding 5.0 kPa under the specified conditions. Where internally fabricated sample carriers are used, please supply an adapter (GL14/GL25 threaded connection) for the test gas stream.

The samples are loaded by the IFA before being returned to the participants.

The tubes and the blank value tubes will be loaded with a sampling volume of 2 l.

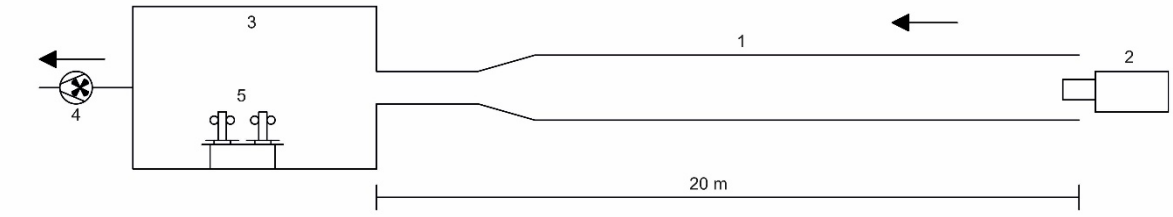
Each participant receives a sample kit comprising:

– Four (two as duplicates) loaded thermal desorption tubes
– Two thermal desorption tubes loaded with the basic gas of the test gas stream to exclude blank values, and
– One unloaded tube.

3.4 Metals on filters

Filter samples bearing metal particles will be shipped. Sampling is performed by means of the total dust sampling system (GSP) on cellulose-nitrate filters (pore size: 8 µm; diameter: 37 mm) [22].

These samples are produced at the Institute for the Research of Hazardous Substances (IGF) of the German Social Accident Insurance Institution for the raw materials and chemical industry (BG RCI) in Dortmund. Aqueous metal salt solutions are pyrolysed by means of a flame generator. The resulting metal oxide particles are passed at a defined air flow rate through a steel tube (wind tunnel) with a length of 20 m and a diameter of 0.5 m (Figure 3). This tube opens into a measurement chamber with a volume of approximately 20 m³ in which the sampling units are located. Homogeneous distribution of the particles is assured at all points within the measurement chamber [23].



1) Wind tunnel, L = 20 m 2) Particle generation 3) Measurement chamber 4) Fan 5) Sampling units

Fig. 3: IGF wind tunnel and measurement chamber

The concentrations are to be within a range of 0.1 to 2 times the applicable limit values (Table 4). In consideration of the analytic performance of the analysis methods to be employed, deviating concentration ranges are expected to be selected for cobalt, nickel and indium. These will be made known in good time.

Table 4: Limit values for metals in Germany

Metal	Limit value in mg/m³ (see [8], [24])	
Zn	0.1	[8]
Cu	0.01	[8]
Pb	0.1	[8]

Metal	Limit value in mg/m³ (see [8], [24])
Ni	0.006 [8]
Co	0.0005 [24]
In	0.0001 [8]
Mn	0.02 [8]

Each participant receives a sample kit comprising:

– Three loaded sample carriers, and

– Four unloaded sample carriers for blank value adjustment.

The analysis method (AAS methods, ICP) may be selected freely. The recommended digestion method is published by the DFG (the German Research Foundation) [25] and accordingly by the IFA folder [26] for the digestion of metal dusts.

In IFA folder 6015 an open and a microwave digestion can be used. In a comparative study it could be shown that both digestion methods can be used equivalently [27].

3.5 Aldehydes

The PT is offered alternatively with and without own sampling. Participants may choose whether they wish to perform sampling for aldehydes themselves, or to have loaded sample carriers supplied to them [28].

The PT scheme consists of three runs with different substances and concentrations within the test gas. It will contain up to four different aldehydes, formaldehyde, acetaldehyde, propionaldehyde and butyraldehyde.

The concentration for formaldehyde will lie in the range between 0.03 and 1 mg/m³, for each acetaldehyde, propionaldehyde and butyraldehyde in the range between 0.1 and 3 mg/m³.

Analysis should be conducted by HPLC according to IFA Folder No. 6045 [29] respectively DIN ISO 16000-3 [30].

3.5.1 Proficiency testing with own sampling

At the dynamic test gas stream at the IFA up to 27 participants can carry out parallel sampling (figure follows).

The PT scheme consists of three runs, each test will last approximately two hours plus preparation time and run-in period.

The participants will receive detailed information about the PT scheme, a description of the apparatus, in particular of the sampling ports and a list of the substances to be analysed, in good time.

3.5.2 Proficiency testing without own sampling

The production of samples for aldehydes is conducted at the dynamic test gas stream of the IFA.

The participants can choose between two sample carriers:

– Waters Sep-Pak DNPH-Cartridge (Prod. No. WAT047205) or

– Supelco LpDNPH S10 Cartridge (Prod. No. 21014).

Each participant receives a sample kit comprising:

– Three loaded sample carriers, and

– Two unloaded sample carriers for blank value adjustment.

4 Processing and documentation of the analysis results

Upon receipt of the samples, the duration of processing will be stated, which will generally be in the order of four weeks.

A USB stick is supplied with the samples for data recording.

Data are recorded by input of the results in a dedicated input program (RingDat of quo data, Dresden). The program, which has an interface in two languages (German or English), runs on all versions of MS WINDOWS from Windows 2000 onwards. Participants are required to print out and sign their results and return the signed copy to the IFA together with the data media. The data are entered into a database and anonymised for further analysis.
They are then analysed and displayed graphically with the aid of ProLab Plus. The IFA conducts statistical analysis.

Following completion of analysis, each participating test body receives

- confirmation of its participation, containing the following information
  - Laboratory number
  - Own laboratory result and z-score
  - Mean value of the samples following elimination of outliers (*Grubbs* test)
  - Reproducibility standard deviation
  - Reference value (mean value of the control samples for quality control)
- and a PT report containing the following information
  - Tabular and graphical presentation of the entire analysis results and the outliers
  - Tabular and graphical presentation of the z-score analysis
  - Alphabetical list of all participants
  - List of the analysis methods employed by the participants
  - and a certificate of attendance (see appendix)

5 Evaluation

5.1 Preliminary remarks regarding the analysis software

Since 2011, quo data's Prolab Plus software has been used for interpretation of the results of the PT schemes[2] . The interpretation methods employed in the software fully satisfy the requirements of DIN ISO 5725-2, -3, -5; DIN ISO 13528 and DIN 38402 A 45 [5], [31] to [34].

5.2 General statistical values

The PT schemes are evaluated in accordance with DIN ISO 5725-2. The basis for scoring of the participants is the evaluation by the "z-score" [31].

Previous to the calculation of the statistical values the outliers are determined. For determination of the outliers the *Grubbs* test is used.

For the PT scheme VOC the participants are supposed to indicate more than one individual value.
In this case, before calculating the individual mean value, the *Cochran*-test is used. I.e. if the difference between the results of a laboratory is too large, it is possible to disregard these results for further evaluation. The remaining individual results are averaged for each hazardous substance and weighed statistically, if applicable (see DIN ISO 5725-2 section 7.5.6 [31]).

5.3 Individual mean value

Individual mean values are only calculated for proficiency testing schemes with more than one individual value (PT scheme VOC).

Individual mean value:

$$c_{jk} = \frac{1}{n_{jk}} \sum_{i=1}^{n_{jk}} c_j$$

i	=	Index for individual values	n	=	Number of measurements
j	=	Index for the laboratory	c_{jk}	=	Individual mean value
k	=	Index for feature level	c_j	=	Individual value

5.4 Cochran outlier test

The dispersion of the individual values of laboratories are analysed with the outlier test according to *Cochran*:

[2] quo data, Gesellschaft für Qualitätsmanagement und Statistik mbH, 01187 Dresden/Germany, http://www.quodata.de/index.php?id=10

Cochran outlier test:

$$C = \frac{s_j^2}{\sum_{i=1}^{n} s_i^2}$$

C = Test value according *Cochran*

s_j = Maximum standard deviation of the tested row

n = Number of measurements

s_i = Standard deviation of the row

5.5 Total mean value

The total mean value C_k is then calculated, which generally serves as the reference value during the evaluation. At PT schemes with low number of participants the mean concentration found while analysing quality control samples can be defined as target concentration. This value is used for further statistical analyses such as the total standard deviation S_k, *Grubbs* test and z-score. Where certified materials are employed, the concentration stated on the certificate is defined as the reference value. The total mean value and total standard deviation are calculated from the data.

Total mean value:

$$C_k = \left(\frac{1}{N_k}\right) \sum_{i=1}^{N_k} C_{jk}$$

Total standard deviation:

$$S_k = \sqrt{\left(\frac{1}{N_k - 1}\right) \sum_{i=1}^{N_k} (C_{jk} - C_k)^2}$$

N_k = Number of individual mean values for the feature level k

S_k = Total standard deviation

C_{jk} = Individual mean value

C_k = Total mean value

5.6 Grubbs test for outliers

It is generally assumed that the body of data is subject to normal distribution. The outlier test is then performed at the 95% level (both sides α = 2.5%). The elimination of outliers ultimately produces a mean value which closely approximates to the "true value" of the sample.

In the *Grubbs* outlier test, the procedure is for all laboratory mean values to be calculated in the first instance and the total mean value and total standard deviation then to be determined from them. The difference is then formed between the individual mean values and the reference value/total mean value, the individual mean value with the greatest difference to the

reference value/total mean value being substituted marked * in the formula of the *Grubbs* outlier test. The test value t is compared to the tabular value for a significance level of α = 2.5% when considered from both sides (see above).

If it is confirmed as an outlier, it is removed from the body of data, and the total mean value and total standard deviation recalculated. The difference between the new total mean value/ reference value and the individual mean values is calculated and the greatest difference substituted in the *Grubbs* formula. If this value is also confirmed as an outlier, it is likewise removed. The procedure is repeated until no more outliers can be eliminated.

Grubbs outlier test:

$$t = \left| \frac{C_{jk}^{*} - C_k}{S_k} \right|$$

C_{jk}^{*} = Individual mean value

C_k = Total mean value/reference value

S_k = Total standard deviation

t = Test value

Should the data exhibit such strong variation that no outliers can be identified by means of the *Grubbs* test, they are defined by the deviation from the reference value. An outlier is recognized as such in this case when the deviation > ± 36%. Where the data material is heterogeneous, the limit can be increased to 50%, or identification of the outliers not be performed.

5.7 z-Score analysis

All individual mean values are considered in the z-score analysis, including the values identified as outliers in the *Grubbs* test.

The z-score of all participating laboratories, including those eliminated as outliers, is calculated with the aid of the outlier-free mean value and the outlier-free standard deviation.

The z-score can be regarded as the *quality characteristic* of the mean value of the individual laboratories (see below).

The z-score analysis is based upon the following formula:

z-score:

$$z = \frac{\left(C_{jk}^{*} - C_k\right)}{s}$$

C_{jk}^{*} = Individual mean value

C_k = Total mean value/reference value

s = Maximum permissible deviation from the reference value (as a rule 10%)

A permissible deviation of 10% is assumed for **s** during analysis of the PT scheme. Where data material exhibits strong scatter the permissible deviation may be increased to up to 20%.

The individual results are then evaluated as shown below:

		|z|	≤	1	Good result
1	≤	|z|	≤	2	Satisfactory result
2	≤	|z|	≤	3	Questionable result
3	≤	|z|			Extremely questionable result

A result for which |z| ≤ 2 is deemed satisfactory, i.e. the PT is deemed passed. Should the |z| score exceed 2, review of the analysis method employed is advisable.

The z-score can be used to determine further statistically important parameters, which shall not however be considered in any greater detail at this point.

5.8 Certificate

The certificate of participation is produced individually from each participant's results for all PT schemes. The certificate is produced in accordance with the following rules:

- All substances are stated on the certificate for which, averaged over all samples, a z-score ≤ 2 was attained.

  Calculation for the individual substance:
  Of three samples, at least two must yield individual z-scores < 2; a z-score distribution such as “2.9, 2.8, 0”, which purely mathematically would yield a mean value of 1.9, results in participation for the substance being deemed unsuccessful.

  No single sample may yield a z-score > 3.

- Participation in the PT is deemed successful when a z-score ≤ 2 (see above) is obtained for over 50% of the analysed substances.
- Should z-scores ≤ 2 be obtained for fewer than 50% of the analysed substances, the certificate merely confirms participation in the PT.

6 Literature

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7 Annex

IFA
Institut für Arbeitsschutz der
Deutschen Gesetzlichen Unfallversicherung

Company, place

department

has

successfully participated in the

Proficiency testing

„VOC 2017“

The following measurands have been successfully analysed:

1,2,4-Trimethylbenzene
1-Butanol
2-Ethoxyethyl acetate
Benzene
Cumene
m-Xylene
n-Heptane
Toluene

Sankt Augustin, 08.08.2017

Head of Unit Chemical agents II

Dr. D. Breuer