

The question of homogeneity inside a chimney: application of ISO 13528 to stack emission proficiency tests

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Received: 16 January 2015 / Accepted: 9 May 2015 / Published online: 27 May 2015
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Abstract The Hessian Agency for the Environment and Geology (HLUG) in Kassel, Germany, offers stack emission proficiency tests (PTs) on an in-house-constructed emission simulation apparatus (ESA). The PTs include gaseous (organic and inorganic) and particulate matter (heavy-metal-doped dust) emissions. A unique challenge arises when ISO 13528 is applied to these PTs: While homogeneity of the used gases, liquids, and dusts can easily be demonstrated, the equivalence of measurement points along the stack emission chimney is a completely different matter. Since each test item batch (in this case the pollution-doped ESA exhaust gas) only exists during one measurement, the standard procedure for determination of homogeneity (in this case equivalence of the sampling openings) is not applicable to determine compliance to the standard. To demonstrate the equivalence of the measurement points on our ESA, we devised a modified homogeneity check based on ISO 13528 annex B. This modified homogeneity check was successfully applied to our stack emission PT and should be applicable to sampling PTs in general.

Keywords Stack emissions · Proficiency tests · Sampling · Homogeneity check · ISO 13528 · ISO 17043

Presented at the Eurachem PTWorkshop, October 2014, Berlin, Germany.

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List of symbols

D	Particle diameter
g	Number of proficiency test items or sampling positions/times tested in a homogeneity check
m	Number of repeat measurements made per proficiency test item or repeat samplings made per sampling position/time
$q_3(D)$	Quantile of particles with a diameter $\leq D$
s	Standard deviation
s_r	Relative standard deviation
s_s	Estimate of between-sample standard deviation
s_{rs}	Estimate of relative between-sample standard deviation
s_w	Within-sample standard deviation
s_x	Standard deviation of sample averages
x	Measurement result (e.g., mass concentration)
\bar{x}	General average of x
σ_{pt}	Standard deviation for proficiency assessment

Introduction

Quality control in the field of industrial emission measurements is an important issue for human health and the environment. Since 1994, the Hessian Agency for the Environment and Geology (HLUG) offers stack emission proficiency tests on a self-made emission simulation apparatus (ESA, Fig. 1). The proficiency tests include gaseous (organic and inorganic) and particulate matter (heavy-metal-doped dust) emissions. Participation in such proficiency tests is mandatory by law (41. BImSchV, [1]) for institutes authorized in accordance with § 29b and § 26 BImSchG [2] to perform legally valid stack emission measurements in Germany.

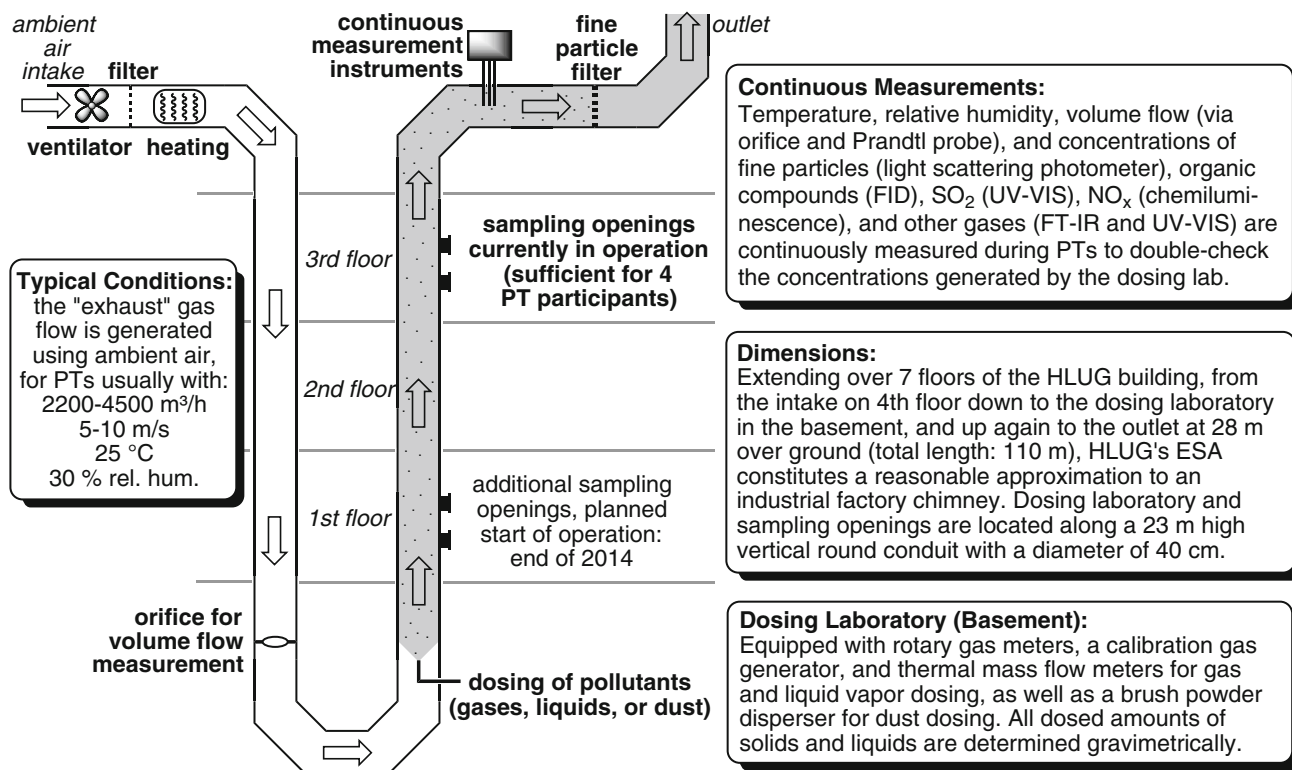


Fig. 1 Scheme of HLUG's emission simulation apparatus (ESA, simplified and not true to scale). For clarity, basement, ground floor, fourth floor, and attic are not specified. See Fig. 3 for exact locations of sampling openings

HLUG's ESA was designed to serve as a reasonable approximation to an industrial factory chimney. It has a total length of 110 m and extends over seven floors of the HLUG building in Kassel, Germany. Central part is a vertical, 23 m high, round conduit with a diameter of 40 cm. The measurement points for the proficiency test participants are positioned along this section of the ESA. The "stack emission" gas flow is generated using filtered and heated ambient air, usually operating at about 2200–4500 m³/h (5–10 m/s) and ca. 25 °C. Pollutants are added at the dosing laboratory in the basement, which is equipped with rotary gas meters, a calibration gas generator, and thermal mass flow meters for gas and liquid vapor dosing, as well as a brush powder disperser for dust dosing. Volume flow, pressure, temperature, and humidity, as well as concentrations of fine particles, SO₂, NO_x, organic components, and other gases, are continuously measured during proficiency tests to double check the concentrations generated by the dosing laboratory. Although gases and particles can generally be dosed together, HLUG currently only offers separate PTs for concentration measurements of particulate and of gaseous emission components.

Proficiency tests are currently offered for a maximum of four participants at a time on the third floor of the HLUG building in Kassel. An expansion to eight participants by

running the proficiency tests simultaneously with another four participants on the first floor is currently in preparation.

Standard ISO 17043 [3] states that a proficiency test provider must ensure that "every participant receives comparable proficiency test items," referring to ISO 13528 [4] for further details on the appropriate homogeneity checks. When this standard is applied to our proficiency tests, a unique challenge arises: Homogeneity of the used gases, liquids, and dusts can easily be demonstrated; the equivalence of the measurement points along the ESA, however, is more complicated. As in our case the "test item" is the pollutant-doped ESA "stack emission" volume flow, each "batch" only exists during one measurement. Consequently, the standard procedure for determination of homogeneity (in our case equivalence of the sampling openings) is not applicable to determine compliance to the standard. To demonstrate the equivalence of the measurement points on our ESA, we conducted a validation measurement program, interpreting ISO 13528 annex B according to our unique circumstances.

General application of ISO 13528 to sampling PTs

The reason for the requirement of a homogeneity check of the test items for a PT is that the property measured by the

participants may vary from sample to sample. If these variations are too big compared to the criterion for proficiency assessment, a participant may fail the test although (or even worse: because) the property for the received sample was measured correctly. Under these conditions, the results of highly proficient participants would be dominated by the randomly different properties of the received samples. As this situation would be both highly unfair and not helpful in determining the participant's performance, ISO 13528 states that the between-sample standard deviation (s_s) must meet the following relation to the criterion for proficiency assessment (σ_{pt}):

$$s_s \leq 0.3 \sigma_{pt} \quad (1)$$

The homogeneity check is carried out by the PT provider (or another single laboratory) by performing repeat determinations of the test items' relevant properties using randomly selected samples. The between-sample standard deviation s_s calculated from these results in accordance with ISO 13528 gives an estimate of the variations in those properties that are caused by actual differences between the samples and not by any errors that occurred during the measurement procedure.

If sampling is part of the PT, generally the same considerations apply. Here, however, the samples of the test item are not produced by the PT provider, but are taken by the participants during the PT. As usual, the provider must ensure that the samples' relevant properties do not depend on conditions that the participant does not control. This means for PTs including sampling that unless a participant makes a mistake during the sampling process, all samples taken during a PT should theoretically be homogeneous enough to result in a positive homogeneity check.

In reality, however, a homogeneity check performed by the PT provider (or another single laboratory), using the samples taken by the PT participants, would be difficult due to the fact that not all samplings will have been performed in the same way. The result would mix actual differences between the samples (that may be due to different time and/or position of the sampling) with differences caused by varying sampling techniques or sample treatment. Instead, analogous to the procedure that only covers the analysis, the proof of homogeneity for a sampling PT should be carried out by a single laboratory performing both the sampling and the analysis of the samples.

A general procedure for the assessment of the homogeneity of the sampling in accordance with ISO 13528 requires only minor changes to the procedure described in annex B. The steps defining the sample preparation, the selection of samples, and the preparation of replicates need to be replaced by:

- take $g \times m$ samples (with $g \geq 10$ and $m \geq 2$) that are representative for all possible correct samplings in the PT

Depending on the nature of the PT, this can mean taking $g \geq 10$ samples at random or at defined positions and/or times. The PT provider needs to devise a sampling plan that includes all relevant deviations that can occur during the PT. Ideally, the same sampling is repeated $m \geq 2$ times under the same conditions. An approximation to this may be a repetition under very similar conditions. When this is not possible, a number of $g \geq 20$ samples may be taken, and the standard deviation of the results may be used as s_s for the homogeneity check.

To minimize the impact of measurement uncertainties, it may be necessary to perform repeat determinations for all $g \times m$ samples when measuring the relevant properties of the test item. Where a measurement device is used to determine the test item's property directly, the $g \times m$ measurements should correspond to the procedure (e.g., regarding the measurement time) used by the participants.

The assessment of the obtained measurement results follows the procedure described in ISO 13528, annex B. If s_s does not exceed 30 % of σ_{pt} (see Eq. 1), the PT provider can assume that the sampling conditions for all PT participants are sufficiently equal. If this check should have a negative result, the sampling conditions are not equal for all participants and consequently the measurement results obtained from these samplings are not sufficiently comparable to allow a PT with the planned σ_{pt} .

Application of ISO 13528 to stack emission PTs

HLUG started a stack emission PT program in 1994, which right from the beginning included sampling. For the past 20 years, these PTs took place on the third floor of our office building in Kassel, Germany, where sampling can be carried out by four participants simultaneously (see Fig. 1). We recently decided to expand our capacities by installing additional sampling openings on the first floor, which would allow us to offer PTs for eight participants simultaneously. While all measurements on the third floor gave comparable results over the past 20 years, it soon turned out that measurements on the first floor gave significantly different results. Further investigations revealed that our dosing procedure was appropriate for measurements on the third floor, but the chemicals injected in the basement were not distributed homogeneous yet when the air flow reached the first floor. During the optimization of our dosing procedure, the modified homogeneity check of sampling positions as described in this paper proved to be a helpful tool in the assessment of our progress.

In our stack emission PTs, the concentrations of different kinds of pollutants are measured by the participants. The components used range from gases (such as SO₂, NO_x, and propane) over vapors of organic liquids (such as formaldehyde, ethyl benzene, toluene, and xylene) to heavy-metal-doped dusts. Gases and vapors require similar sampling techniques, that generally extract a partial volume flow from the chimney at a fixed position, which is either analyzed directly (using a continuous measurement device) or collected and analyzed later (discontinuous methods). Dust measurements are more complicated, as dusts show a particle size-dependent inertia, which hinders homogenization in a stack emission [5, 6]. To account for these known effects, the standard procedure for the measurement of dust mass concentrations in stack emissions is a discontinuous isokinetic grid measurement [7, 8], which partially compensates inhomogeneity effects [9]. Based on the different behavior and sampling techniques for gases/vapors on the one hand and dusts on the other hand, we devised two different sampling plans for the different pollutant types.

Homogeneity check for sampling of gaseous stack emissions

Since gases of similar molecular weight do not separate spontaneously, we concluded that once a homogeneous distribution of a gas in our ESA is achieved, the homogeneity of concentrations inside the stack emission does not decrease again, provided that no other gas (such as air) enters the volume flow. The same is true for vapors of organic liquids, which behave like gases provided their concentration is far below the condensation point. Our sampling plan for the measurement of concentrations of gaseous emissions consequently concentrated on the lowest sampling position available, which is passed by the pollutant-doped volume flow first. As the PT participants can perform the sampling on any position of the cross section of the ESA, we distributed the sampling positions for the homogeneity check equally across the cross section. The sampling plan for our ESA, which has an inner diameter of 40 cm, comprises 16 sampling positions as indicated in Fig. 2. The locations of these sampling positions were determined in accordance with the procedure for grid measurements described in VDI 2066 Part 1 [7].

Each of the 16 positions is to be sampled twice, which results in 16 × 2 samplings. This kind of repeat determination requires a constant concentration distribution across the cross section, which needs to be checked independently during the whole sampling process. If the concentrations at fixed points should change significantly over time, a different approach may be necessary.

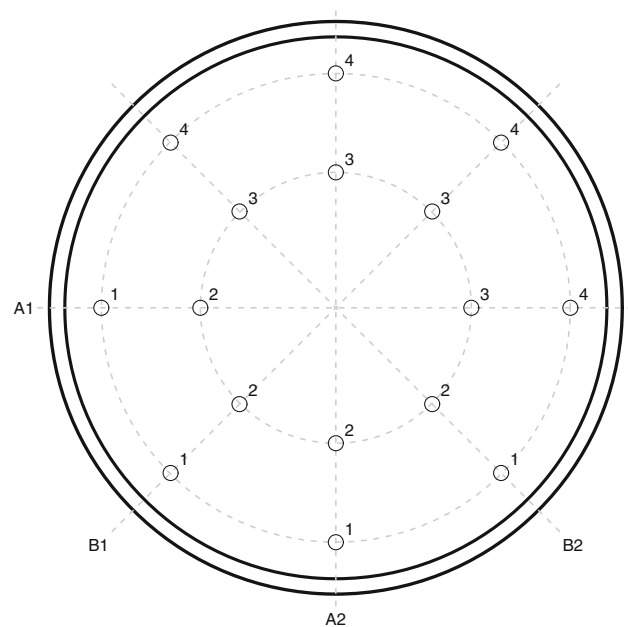


Fig. 2 Sampling positions for homogeneity check for gaseous stack emissions in HLUG's ESA (diameter: 40 cm). The axes B1 and B2 are 20 cm above axes A1 and A2. The sampling position of the reference FID was at the center of the chimney, 200 cm above axes A1 and A2 (see Fig. 3)

Homogeneity check for sampling of particulate stack emissions

As the sampling method used in our PT for the measurement of dust mass concentrations is a grid measurement on four points along the cross section of the chimney, the samplings for the homogeneity check need to be performed in the same way. Due to the size-dependent inertia of particles and its consequences for the homogeneity of the dust concentration, the sampling plan needs to represent all sampling positions available along the chimney.

The dust dosing in our ESA is carried out using a brush powder disperser. This device consists of a dust-filled cylinder, which is pressed at constant speed onto a rotating brush that conveys the dust into a pressurized air stream. Via this air stream, the dust is injected into the ESA. This system is able to produce a constant dust supply for ca. 50 min, but after that, the cylinder needs to be refilled. To measure the dust mass concentration, the cylinder is weighed before and after the dosing. This procedure allows a very precise determination of the dosed dust concentrations after its completion, but it cannot deliver a precisely predetermined concentration on demand. Consequently, we conducted our samplings at fairly similar dust concentrations and used the recovery value (measured dust concentration divided by dosed dust concentration in the ESA) to assess homogeneity of the samplings.

In this case, the repeat determinations are to be carried out as two subsequent measurements, assuming that the deviations between these give a good estimate of the random deviations between samplings. In our PTs, the described grid measurements are only performed using the following sets of axes (see Fig. 3): A1/A2 and C2/C4 on the first floor, and 2/3 and 9/10 on the third floor, respectively (two participants are measuring on the same level simultaneously using different axes). Consequently, the modified homogeneity check must be limited to grid measurements on these four positions. We therefore devised for our ESA a sampling plan that comprises 8×2 samplings on the first floor and 8×2 samplings on the third floor.

Experimental

General

The data used in this study are results of continuous and discontinuous concentration measurements. All concentrations relate to normal conditions (273.15 K, 1013.25 hPa), dry. The exact location of the sampling positions on HLUg's ESA is shown in Figs. 2 and 3.

Equipment and procedures

Measurements of volume concentrations of total organic carbon (TOC) were taken using two flame ionization detectors (FIDs) with electrically heated probe tubing (both FID 3006, Bernath Atomic/SICK AG, 79183 Waldkirch, Germany). The FIDs were adjusted using synthetic air as zero gas and propane in synthetic air as span gas. As the exact volume concentration of *m*-xylene was not relevant for this study, a response factor was not determined. The results of the FID measurements are therefore expressed as propane equivalent volume concentrations, which are proportional to the *m*-xylene volume concentration. Recorded were values at a rate of 1 Hz, reported are 15-min mean values.

Measurements of dust mass concentrations were taken isokinetically and in accordance with [8], using a vacuum pump (TLV 6(01), Rietschle/Gardner Denver Deutschland GmbH, 97616 Bad Neustadt, Germany) controlled by a volume flow measurement device (MPN-E, Breitfuß Messtechnik GmbH, 27243 Harpstedt, Germany), an absorption dryer (filled with ca. 750 g silica gel pellets), and an in-stack plane filter device (titanium ASTM B 348, nozzle diameter: 10 mm, Paul Gothe GmbH, 44789 Bochum, Germany), equipped with a quartz filter (MK 360, diameter: 50 mm, Munktell & Filtrak GmbH, 09471 Bärenstein, Germany). Weighing of the filters was carried

out using a precision balance (ME 235 P, Sartorius AG, 37075 Göttingen, Germany). Before weighing, the filters were dried for 2 h, at 180 °C prior to usage and at 160 °C after the dust sampling. Between drying and weighing, the filters were stored in a dessicator at 20 °C. The measurements were taken as 30-min grid measurements, divided into four 7.5-min samplings at positions 59 mm and 341 mm (distance to conduit wall at the sampling opening) on two orthogonal axes at the same level (see Fig. 3).

Dosing of pollutants

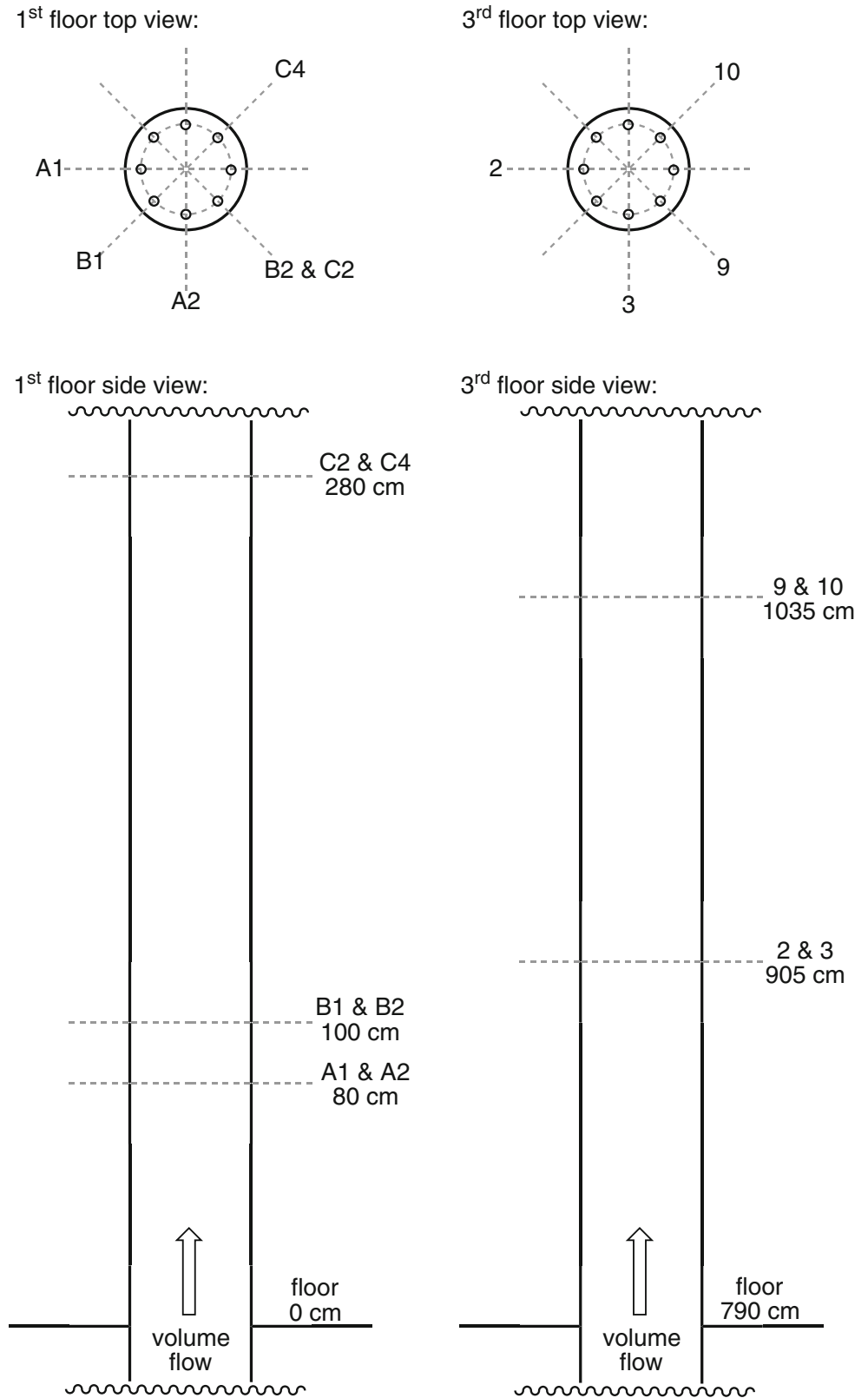
Dosing of *m*-xylene into the ESA was carried out using a calibration gas generator (HOVACAL digital 122, IAS GmbH, 58640 Iserlohn, Germany) in combination with a precision balance (LP 1200 S, Sartorius AG, 37075 Göttingen, Germany). The constancy of the dosing was double checked continuously using a flame ionization detector (Multi-FID 14, ABB Automation GmbH, 68309 Mannheim, Germany) near the end of the ESA conduit system (see Fig. 1). The volume flow was adjusted to 3270–3310 m³/h (normal conditions, dry) and constantly monitored using a gas flow measurement orifice (Blende 65856, Hartmann & Braun Meß- und Regeltechnik/ABB Automation Products GmbH, 63755 Alzenau, Germany) in combination with a measuring transducer (AVA 500, Schoppe & Faeser GmbH/ABB Automation Products GmbH, 63755 Alzenau, Germany).

Dosing of dust into the ESA was carried out using a brush powder disperser (RBG 1000, PALAS GmbH, 76229 Karlsruhe, Germany) and a precision balance (LC 1201 S, Sartorius AG, 37075 Göttingen, Germany). The constancy of the dosing was double checked continuously using a scattered-light photometer (LMS 181, PCME Ltd., St. Ives (Cambs.), UK) near the end of the ESA conduit system (see Fig. 1). The volume flow was adjusted to 4230–4270 m³/h (normal conditions, dry) and constantly monitored using the above-mentioned orifice.

Materials

m-Xylene (for synthesis, 99.8 %, Merck Chemicals GmbH, 65824 Schwalbach, Germany) was used unchanged. Iron blast furnace slag (Schneider-Strahlmittel STM-HOS 7.0 B030031, DF Druckluft-Fachhandel GmbH, 71083 Herrenberg, Germany) was ground using a ball mill and sieved. The resultant particle size distribution was measured via light-scattering particle size determination (Analysette 22, FRITSCH GmbH, 55743 Idar-Oberstein, Germany) with the following result (volume based): $q_3(2.2 \mu\text{m}) = 0.05$; $q_3(4.1 \mu\text{m}) = 0.10$; $q_3(20.7 \mu\text{m}) = 0.50$; $q_3(48.7 \mu\text{m}) = 0.90$; $q_3(58.5 \mu\text{m}) = 0.95$; $q_3(79.1 \mu\text{m}) = 0.99$.

Fig. 3 Location of sampling positions on HLUG’s ESA. Heights are indicated relative to first floor ground. Sampling positions for discontinuous grid measurements used for the determination of dust concentrations are indicated in the top view cross sections. The conduit is straight and vertical from –600 to 1700 cm



Results and Discussion

As we are currently still in the process of optimizing the dosing procedures for our ESA, we only conducted reduced sampling plans so far. To assess the homogeneity of sampling positions for gaseous emission measurements, *m*-xylene was used as a representative compound. The sampling plan was here reduced to 8×2 measurements (on the axes A1 and A2, see Fig. 2).

To assess the homogeneity of sampling positions for dust emission measurements, ground iron blast furnace slag was used as a representative material. Here, the sampling plan was reduced to 4×2 measurements on both the first and the third floors (adding up to 8×2 samplings).

Both sampling plans are not sufficient to meet the requirements of ISO 13528, but in our opinion, they already give a good estimate of the equivalence of the sampling positions. The following results were obtained after several optimization measures and are exemplary processed according to ISO 13528 annex B to show the general applicability of the modified homogeneity check as a tool to determine the equivalence of sampling positions.

Homogeneity check for sampling of gaseous emission measurements

Table 1 shows the results of flame ionization detector (FID) measurements of *m*-xylene-doped air at the positions indicated in Fig. 2. Measured was the volume

concentration of total organic carbon (TOC), expressed as propane equivalents.

Using the equations given in ISO 13528 annex B, we calculated the following values from these data:

- general average of volume concentrations of total organic carbon at sampling position: $\bar{x} = 42.683$ nL/L (C_3H_8 equivalents)
- standard deviation of sample averages: $s_x = 0.210$ nL/L (C_3H_8 equivalents)
- within-sample standard deviation: $s_w = 0.164$ nL/L (C_3H_8 equivalents)
- between-sample standard deviation: $s_s = 0.175$ nL/L (C_3H_8 equivalents)
- relative between-sample standard deviation: $s_{rs} = 0.409$ %

The volume concentrations obtained from the second FID at the reference position showed a relative standard deviation of $s_r = 0.36$ % ($s = 0.153$ nL/L, C_3H_8 equivalents), which is similar to the observed within-sample standard deviation. We therefore concluded that the concentration distribution shows sufficient stability to be assessed using the modified homogeneity check.

According to Eq. 1, the value of $s_{rs} = 0.41$ % allows for a criterion for proficiency assessment as low as 1.4 %. As the *m*-xylene vapor used in this study can be seen as a representative substrate for vapors in general, we feel confident that the use of any other evaporated liquid will result in similar values. Our current criteria for

Table 1 Results of TOC volume concentration measurements of *m*-xylene-doped air via FID (see Fig. 1 and 2 for the location of sampling positions)

Entry	Start of sampling (min after start of the first sampling)	TOC (nL/L C_3H_8 equivalents) at reference position (15-min mean values)	Sampling position	Number of sampling at this position	TOC (nL/L C_3H_8 equivalents) at sampling position (15-min mean values)
1	0	42.675	A1-1	1	43.255
2	179	42.452	A1-1	2	42.895
3	17	42.592	A1-2	1	43.090
4	197	42.475	A1-2	2	42.806
5	34	42.464	A1-3	1	42.666
6	214	42.490	A1-3	2	42.449
7	58	42.404	A1-4	1	42.632
8	230	42.522	A1-4	2	42.607
9	103	42.222	A2-1	1	42.454
10	250	42.612	A2-1	2	42.657
11	122	42.408	A2-2	1	42.470
12	266	42.741	A2-2	2	42.685
13	140	42.262	A2-3	1	42.386
14	283	42.596	A2-3	2	42.607
15	156	42.258	A2-4	1	42.542
16	300	42.635	A2-4	2	42.734

Table 2 Results of mass concentration measurements via discontinuous dust sampling (see Figs. 1, 3 for the location of sampling positions)

Entry	Sampling position	Number of sampling at this position	Dosed dust mass concentration (mg/m ³)	Measured dust mass concentration (mg/m ³)	Quotient measured/dosed dust mass concentration
1	A1/A2	1a	8.64	8.88	1.028
2	A1/A2	1b	8.69	8.76	1.008
3	A1/A2	2a	8.42	8.41	0.999
4	A1/A2	2b	8.43	8.40	0.997
5	A1/A2	3a	8.76	8.46	0.965
6	A1/A2	3b	8.70	8.75	1.006
7	A1/A2	4a	8.53	8.53	1.000
8	A1/A2	4b	8.61	8.64	1.003
9	2/3	1a	8.80	8.99	1.021
10	2/3	1b	9.27	9.20	0.992
11	2/3	2a	9.36	9.32	0.996
12	2/3	2b	9.36	9.39	1.004
13	2/3	3a	9.25	8.86	0.958
14	2/3	3b	9.37	9.05	0.966
15	2/3	4a	9.34	9.37	1.004
16	2/3	4b	9.18	9.50	1.035

proficiency assessment of concentration measurements of different gases and vaporized liquids range from 2.5 to 4.5 % and are derived from the participants' results of recent years. Based on the data obtained so far, we expect that the full sampling plan will result in a successful homogeneity check for all measurements currently applied in our PTs.

Another assessment of the obtained data according to EN 15259 [10] further proved the homogeneity of the concentration distribution along the cross section.

A similar assessment for propane, which (like other gases) is injected into our ESA in a slightly different way, resulted in a negative value for s_s^2 . This can be expected when the sampling positions are highly homogeneous. To assess homogeneity, in this case, we used s_x instead of s_s in Eq. 1. This leads to a higher requirement for the homogeneity check, as the effect of s_w (which generally lowers s_s) is not taken into account.

Homogeneity check for sampling of particulate emission measurements

Table 2 shows the results of discontinuous dust samplings at the positions indicated by Fig. 3. As the dust concentrations in our ESA can only be dosed constantly for the duration of one measurement, but not at a precisely predetermined value, the quotient of measured and dosed dust concentration was used for the homogeneity check.

Using the equations given in ISO 13528 annex B, we calculated the following values from these data:

- general average of quotients of measured and dosed dust mass concentrations: $\bar{x} = 0.9988$
- standard deviation of sample averages: $s_x = 0.0185$
- within-sample standard deviation: $s_w = 0.0159$
- between-sample standard deviation: $s_s = 0.0147$
- relative between-sample standard deviation: $s_{rs} = 1.47 \%$

According to Eq. 1, the value of $s_{rs} = 1.47 \%$ allows for a criterion for proficiency assessment as slow as 4.9 %. Our current criterion for proficiency assessment of the discontinuous measurement of dust concentrations, which is derived from the participants' results of recent years, is 7.0 %. Based on the data obtained so far, we expect that the full sampling plan will result in a successful homogeneity check.

Conclusion

By minor modification of the homogeneity check described in ISO 13528 annex B, we developed a versatile check for the determination of equivalence of different sampling positions and/or times used in proficiency tests. The modified homogeneity check was applied to HLUg's stack emission proficiency test by devising different sampling plans for gaseous and for particulate emission measurements. Preliminary results derived from partial execution of these sampling plans gave positive results. The full execution of the planned sampling program is currently in progress.

Acknowledgments We thank M. Altmann, S. Anis, R. Burzan, A. Fricke, M. Gerhold, I. Kämpken, H. Knoth, H. Neves and M. Scheibe (all HLUG) for their assistance in execution of the measurements for this study.

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