Proficiency Testing for Determination of Particulate Matter on Dust Testing Train in Ostrava (Czech Republic)

1: In the Energy Research Center workplace at VŠB - Technical University of Ostrava, an experimental dust testing train has been built which enables researchers to set a comparative reference particulate matter concentration in a vertical pipeline of 313 mm inner diameter and c. 7 m length.
This equipment is primarily used for proficiency testing for laboratories dealing with particulate matter (dust emissions) measuring. Laboratories can participate in the Proficiency testing scheme (PT scheme) for dust measuring which has been performed in the Energy Research Center every year since 2009.
The Proficiency testing (PT) is an evaluation of a participant's performance compared with predetermined criteria with the aid of interlaboratory comparison of tests. A need for lasting confidence in a laboratory performance is not essential only for laboratories and their clients, but also for other interested parties, such as regulatory bodies, accreditation bodies of an accrediting laboratory, and other institutions specifying laboratory requirements.

"Proficiency testing (PT) belongs among the most important methods for verification of laboratory testing quality. PT is used for verification of a laboratory competence to perform (authorised or accredited) tests of emissions measuring."

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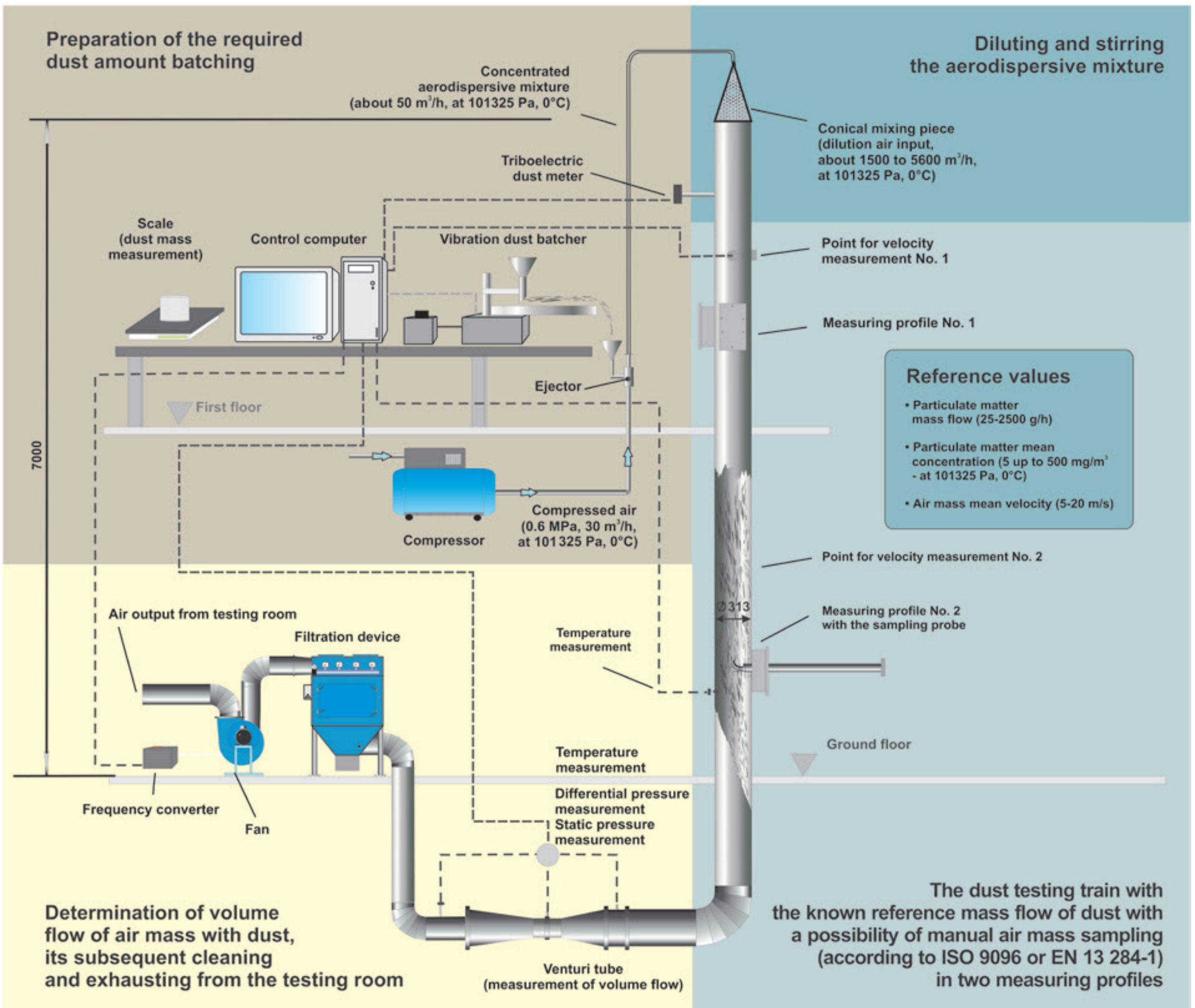


Fig 1. Dust train scheme

2 : Principles of preparation of the dust mixture (aerosol) in the dust testing train

The dust testing train allows preparation of a flowing dust environment so that spatial homogeneity as well as time stability of the flowing aerosol medium can be achieved to the utmost degree. For one determination, the aerosol environment is usually prepared for a minimum period of one hour. The train allows preparation of the reference ("veritable") value of the following factors in the vertical pipeline:

- Particulate matter mass flow (25 – 2500 g/h)
- Particulate matter mean concentration in the measuring profile (5 up to 500 mg/m^3 at 0 °C and 101 325 Pa)
- Air mass mean velocity in the measuring profile (5 up to 20 m/s)

2.1 : Section 1- Preparation of the required dust amount batching

Preparation of the concentrated aerodispersive mixture is ensured by a vibration batcher, which spills a known amount of dust into a funnel from which the air and dust mixture (aerosol) is transported to an ejector. The ejector sucks-in the batched dust with air and mixing of this mixture with ejector jet air occurs in a diffuser.

Fig 2. Vibration dust batcher for the dust train

2.2 : Section 2 and 3 – Diluting and stirring the aerodispersive mixture and the dust testing train with the known reference mass flow of dust, with a possibility of air mass sampling in two measuring profiles

This mixture is transported by way of diameter 20 mm pipeline through a conical mixing piece (perforated sheet) to an inlet in the testing train sampling section which is formed by diameter 313 mm vertical pipeline of c. 7 m length. On the vertical sampling section, there are two locations enabling a representative air mass sample take-off for determination of the mean concentration of particulate matter and its mass flow.

2.3 : Section 4 – Determination of volume flow of air mass with dust, its subsequent cleaning and exhausting from the testing room

Further, the air mass is lead through the Venturi tube which serves for the exact determination of the air mass volume flow. Behind the Venturi tube there is a filtration device where a substantial part of the batched dust is captured. The filtration device is subsequently connected with an exhausting fan which represents a source of suction for the dust testing train.

2.4 : Reference values determination

The particulate matter mass flow reference value is determined on the basis of the batched dust weighed amount and batching time. The particulate matter mean concentration reference value in the measuring profile is determined from the particulate matter mass flow and air mass volume flow in the dust testing train. Air mass flow rate is controlled with the help

Fig 4. Covered exhausting fan on the left, filtration device on the right

of a frequency converter which alters the rotational speed of the exhausting fan. The dust batching continuity is controlled with the aid of an output signal from the triboelectric dust meter positioned at the upper part of the vertical pipeline. In order to control the entire process of the dust mixture preparation and to store the measured data, there is a PC automatically regulating the air mass flow in the dust train and also regulating the dust batching to the dust train.

3 : Utilisation of the dust testing train

The dust testing train is used above all for proficiency testing of laboratories for particulate matter emissions measuring. However, the equipment has also been used for other applications, research or education purposes, e.g.:

- Validation of devices for on line determination of dust concentration;
- Verification of a possibility to run the dust train with a pulverised coal extreme flow in short-time modes (c. 5 min), when pulverised coal mass flows of c. 135 kg/h have been achieved; this mode has been used for a calibration of dust meters monitoring the dangerous "explosive" threshold dust concentrations.
- On the dust train, series of experimental tests have been performed in order to determine influence of aerosol (non) isokinetic sampling on the measured dust concentration for the batched dust defined granulometry.
- Experimental tests have been performed in order to determine the influence of an isokinetic sampling probe mouth shape on dust concentration measuring.
- Other utilisation of the dust train is e.g. to verify dust meters functionality (to a kind, shape, size and colour of particulate matter) or to determine properties of filtration materials for the industrial filtration and other.

Fig 3. Diluting section on the left, measuring profile 1 (upper) in the middle, measuring profile 2 (lower) on the right

4 : Proficiency testing in a field of emissions monitoring

Proficiency testing (PT) belongs among the most important methods for verification of laboratory testing quality. PT is used for verification of a laboratory competence to perform (authorised or accredited) tests of emissions measuring.

The Association of Laboratories for Emissions Measuring (the ALEM, www.alme.cz) has been organizing PT's since 1996, when they were originally only intended for the ALEM members.

In 1998 the tests were made available for all persons interested and from that year they have been made public regularly in the Proficiency testing scheme (PT scheme) on the Czech Accreditation Institute (CAI) websites (Český institut pro akreditaci o.p.s. - ČIA, www.cia.cz). In 2003 the ALEM gained a status of the CAI pilot workplace and in 2012 the ALEM applied for accreditation as a proficiency testing provider which it gained that year.

Gradually, from the original limited scope of testing, a system has been developed which allows covering of participants' essential requirements for proficiency testing in the emissions measuring field in a relatively broad scope. In general, it is true that a regular inspection of accreditation criteria, performed by an accreditation body and a successful participation in PT connected with it, results in a continuous improvement of work and in a substantial enhancement of the quality of measuring a respective subject. And that is the very aim of each provider of proficiency testing, and also of the ALEM.

At present, the ALEM offers the following tests within the Proficiency testing scheme:

Accredited:

- ALME-OR-01: Determination of selected gaseous components (CO, NO, SO^2),
- ALME-OR-02: Determination of propane concentration in a reference material (RM),
- ALME-OR-03: Determination of ammonia concentration in RM,
- ALME-OR-06: Determination of particulate matter (TSP) mass flow in flowing air mass.

Nonaccredited:

- ALME-OR-04: Determination of heavy metals (As, Pb, Cr, Cu, V) in RM (fly ash)
- ALME-OR-05: Determination of fluorides and chlorides in a solution
- ALME-OR-07: Determination of BTEX in an emission sample (a small vial containing activated carbon)
- ALME-OR-08: Determination of PAH in RM (fly ash)
- ALME-OR-09: Determination of halogenderivate concentration in an emission sample (a small vial containing activated carbon)
- ALME-OR-10: Determination of gaseous HCl in RM (gas)

5 : PT for dust emissions measuring

The first PT for dust emissions measuring in the Czech Republic was organized by the ALEM in 2004. The PT was carried out in a chimney of one large industrial source of dust emissions. An advantage of this source was a considerable time stability and homogeneity of the aerosol. A crucial disadvantage was not knowing the veritable dust concentration in the aerosol, a large diameter of the measuring profile and a low dust concentration. This way it was possible to verify, whether participants were measuring the dust emissions accurately, but not whether they were measuring them properly.

In 2008 the dust testing train was built in the Energy Research Center at the VŠB - Technical University Ostrava (ERC, http://vec.vsb.cz/) by which the principal disadvantage of the PT for determination of dust concentration in emissions (aerosol) performed so far has been removed. Since 2009 this PT has been performed in the ERC under the auspices of the ALEM.

As mentioned in the chapter above, in 2012 the ALEM has gained from the CAI the Certificate of Accreditation no. 314/2012 for "Proficiency testing scheme" (PT scheme) in a field of emissions measuring (waste gas) and sampling, among others also for the test: ALME-OR-06: Determination of particulate matter (TSP) mass flow in flowing air mass.

The PT has been performed by means of PT scheme in compliance with EN ISO/IEC 17043 and in accordance with guidelines for accreditation MPA 30-03-12 par.4.

Since 2012 the participation in the ALME-OR-06 PT scheme is an acknowledged test for all the European Union member states.

6 : Successfulness assessment method for participants in the PT for particulate matter measuring

Participants perform three independent measurements of all monitored values (particulate matter mass flow, particulate matter mean concentration, air mass mean velocity in a profile, air mass flow in a profile), where the main comparative value is the particulate matter mass flow and the air mass mean velocity in the profile (other values are evaluated for reference only for a PT participant need).

Successfulness of participants in the PT for particulate matter emissions measuring is evaluated for each value separately, and a so-called Z-score is determined (in compliance with EN ISO/IEC 17043) with the aid of the measured results of the participants can be evaluated statistically.

$$Z = \left| \frac{x - X}{X \cdot \sigma} \right|$$

x is a value measured by a participant within the respective measurement

X is a reference value relevant for the respective participant within the respective measurement

σ is a standard deviation for proficiency assessment (an estimation of the method variability determining acceptable results) This value is determined by the PT organiser.

If the Z-score resulting value was ≤ 2, the given parameter measurement is successful.

During years of organising these PT's the criteria have been adapted depending on the proficiency testing provider's decision.

In 2009 and 2010 the criteria were relatively moderate and to achieve Z-score value ≤ 2 at least once of three measured values was enough to pass out the PT successfully. Since 2011 the successfulness criterion has been made stricter and for passing out the PT successfully Z-score value ≤ 2 needs to be achieved in all three independent measurements for the given value.

7 : Results of participants of the PT for particulate matter measuring in 2009 - 2013

Every year measuring groups from the Czech Republic, Slovak Republic, Poland, Serbia and Germany have been coming over for comparative measurements. Successfulness of participants is reviewed in the table below.

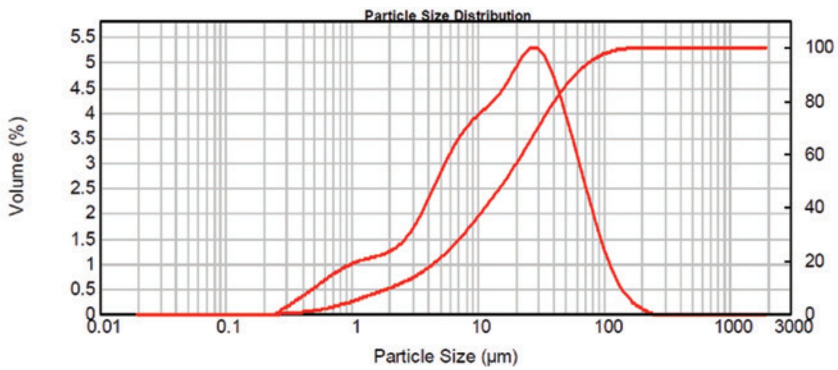
Table 1. Successfulness of PT participants from 2009 - 2013

		Particulate matter mass flow			Particulate matter mean concentration			Air mass mean velocity		
Year	Number of participants	Successful participants %	Mean value g/h	σ %	Successful participants %	Mean value mg/m³	σ %	Successful participants %	Mean value m/s	σ %
2009	47	91,5	310	15	83,0	96	15	95,9	13,0	10
2010	37	86,5	93	15	86,5	30	15	100,0	12,9	10
2011	27	70,4	88	15	77,8	29	15	88,9	12,2	8
2012	26	80,8	104	15	76,9	40	15	92,3	10,7	8
2013	28	85,7	60	20	64,3	20	15	89,3	12,6	8

Note: The larger (massy) particles are contained in aerosol, the more substantial are the aerosol isokinetic sampling influences on the measured particulate matter concentration accuracy.

8 : Parameters of dust batched to the dust train within ALME-OR-06

Fly ash captured in the last section of the electrostatic separator behind a hard coal burning boiler with a wet bottom furnace is used for batching within the PT. This fly ash is dried and particles of granulometry > 200 μm are removed. Granulometric analysis of this dust is shown in the following chart. This dust is relatively coarse in comparison with typical industrial sources dust emissions, which allows better examining of the ability of PT participants to cope with aerosol isokinetic sampling principles.



The first decile d(0.1)= 2 μm; median d(0.5) = 16 μm; the ninth decile d(0.9) = 60 μm

Chart: Granulometric analysis of fly ash used within the PT (determined on MASTERSIZER 2000 apparatus)

If needed, the dust can be processed to the demanded granulometry with the help of sieves. Any non-explosive, non-radioactive, non-toxic, dry, loose and non-adhesive solid particles of size c. from 5 up to 200 μm can be batched to the dust testing train. The used dust can be polydispersive or, in case of need, pseudo monodispersive (i.e. with a noticeable proportion of particles of a certain size).

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