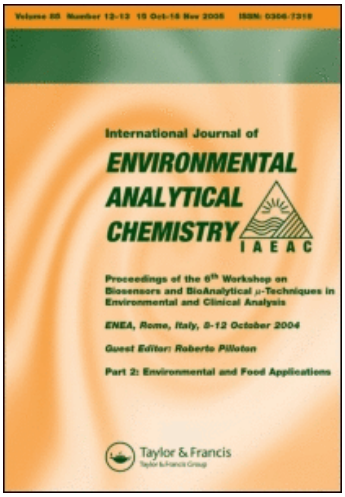

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NEW SAMPLING METHOD FOR THE DETERMINATION OF CONCENTRATIONS OF PARTICULATE MATERIAL IN GAS-CARRYING DUCTS

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A new sampling method for the quantitative determination of particulate material in gas-carrying ducts is presented. The method uses water as the collection and transportation medium instead of the normally applied in-(out)-stack gas filtration technique. The water is circulated in a closed-loop system formed by the sampling nozzle, probe tube and two impingers which act as collection vessels for the particulate material. The total mass of the particulate material is determined gravimetrically after evaporation of the water.

A good correlation exists between the data obtained with the standard in-stack filtration method and the described new sampling method. The new method will be especially useful in circumstances which cause severe problems with the classical filtration technique, such as high mass flow rates of particulate material and high vapour contents. The described method has successfully been applied to the determination of scrubber efficiencies.

KEY WORDS: Particulate material, stack-sampling.

INTRODUCTION

The determination of particulate material in gas streams in confined spaces like ducts, chimneys and flues is normally performed by in- or out-stack filtration using a sharp edged entry nozzle with isokinetic sampling[1–4]. To allow for a non-uniform distribution of the particulate concentration in the duct, samples are taken at a representative number of positions in the duct cross section. The particulate material entrained in the gas sample is separated by a filter medium and determined gravimetrically. The particulate concentration is calculated from the mass of sampled particulate material and the gas sample volume. The particulate mass flow rate is calculated from the particulate concentration and the duct gas volumetric flow rate.

For the separation of the particulate material, high retention flat filters are most widely applied. However, the use of these filters has several drawbacks. At high dust concentrations these filters can be blocked already after a short period of sampling. Moreover, water condensation may occur at high water contents of the duct gas. The use of an in-stack filter is not always a solution to avoid water condensation when the temperature of the duct gas is below its dew point (water saturation) or when the filter with filter holder cannot be heated in an external hot-box prior to its use in the duct. The

best way to avoid water condensation probably is the use of a heated probe tube and out-stack filter. However, losses of particulate material in the probe tube may result in too low concentrations and mass flow rates. Another draw-back of flat filters is the necessity to condition and weigh them before use. Moreover, they have to be handled with utmost care after sampling to avoid any loss of the collected particulate material. It has to be mentioned that the filtration technique is not very suitable for particle diameters $< 0.3 * 10^{-6}$ m.

In this paper, an alternative sampling method is described which overcomes the problems mentioned above for the use of flat filters. The method has been compared with the conventional method of sampling in a duct gas, under mild conditions. In addition, it has been applied under more severe conditions, i.e. high dust concentrations and high water vapour contents, in order to establish the efficiency of installations for the reduction of emissions.

EXPERIMENTAL

Apparatus

A Zambelli (Bareggio, Italy) Model 6000$^+$ automated sampling unit has been applied for isokinetic sampling. This system consists of a Model stack 4 sampling probe tube, with a sharp edged entry nozzle, a calibrated S-type pitot tube and a calibrated thermocouple. The instrument continuously measures the gas velocity of the duct gas near the entry nozzle and automatically adjusts the sampling flow rate to maintain isokinetic sampling conditions. Furthermore, the sampled total volume of duct gas is measured and expressed as Nl, i.e. litres under standard (0°C, 101.3 kPa, dry or wet) conditions. From the mass of the sampled particulate material and the total sampled volume, the concentration in the duct gas is calculated and expressed in mg/Nm^3 (dry).

In-stack gas filtration technique[1-4]

Figure 1 shows the set-up for isokinetic sampling of particulate material from a duct using an in-stack flat filter. The filters used were Whatmann GF/F 47 mm glass fibre filters obtained from Tamson (Zoetermeer, The Netherlands).

New water circulation technique

In Figure 2 the set-up for the isokinetic sampling of particulate material from a duct is presented, using a new particulate material collection concept. In this method pure water (treated in a Milli Q (Millipore, Bedford, USA) system) is pumped to the front of the sampling probe tube at a flow rate of approx. 35 ml/min by a Masterflex (Cole-Parmer, Chicago, USA) Model 7544–30 tube pump. The water is used as the collection and transportation medium for the particulate material, and is trapped in the first of a series of two impingers, each containing 200 ml of pure water.

The second impinger is used as a back-up. Since the water (with particulate material) from the first impinger is pumped to the sampling probe tube, a closed water circuit is maintained.

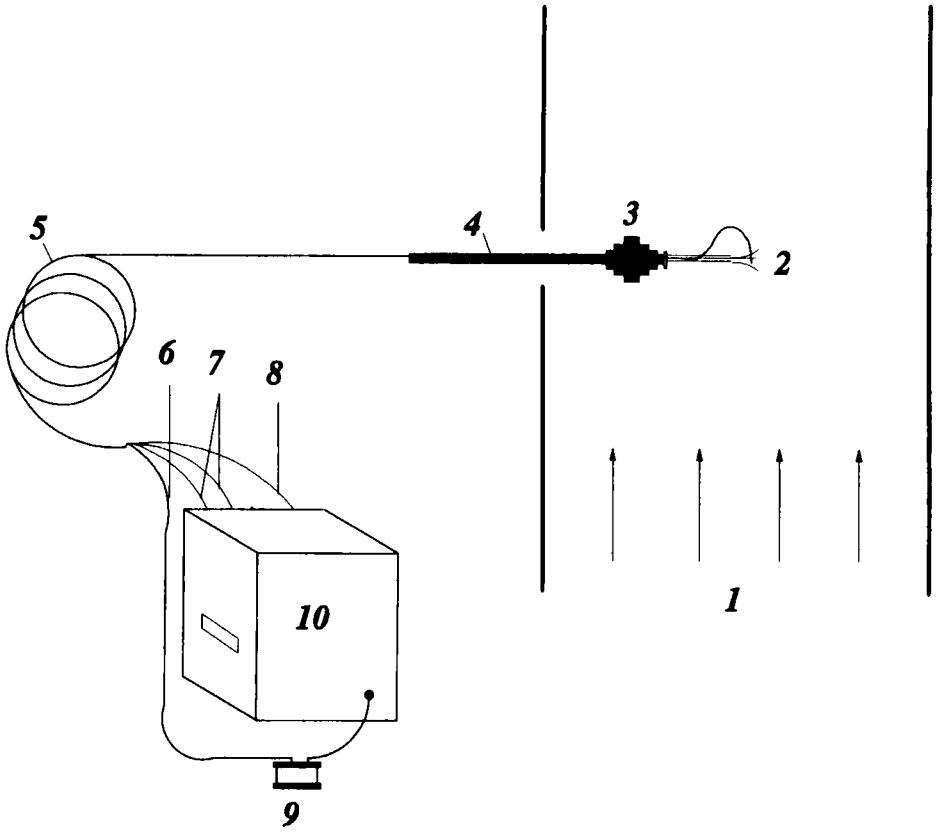

Figure 1 Set-up for isokinetic sampling of particulate material from a gas-carrying duct using an in-stack flat filter. 1, gas-carrying duct with particulates; 2, sharp-edged entry nozzle, S-type pitot tube and a thermocouple; 3, stainless steel filter holder accommodating a 47 mm Whatmann GF/F flat filter; 4, stainless-steel sampling probe tube containing a 1/2″ glass tube; 5, umbilical cord containing a sampling line (6), connected to sampling probe tube, two PVC lines (7) for measuring the dynamic and static pressure in connection with the pitot tube, and an electrical wire (8) connected to the thermocouple; 9, container with silica for drying the duct gas; 10, Zambelli 6000' automated isokinetic sampling unit.

A detailed flow chart of this new sampling system is presented in Figure 3. Pumping of water towards the sampling probe tube can be started immediately after starting the isokinetic sampling, but must be stopped approximately 1 min before the end of the sampling in order to avoid losses of water (containing particulate material) in the duct. This is performed by removing the suction 1/8" PTFE capillary (No. 4, Figure 3) from the water layer in the first impinger. At the end of the sampling period the sampling probe tube with entry nozzle and the 1/8" PTFE capillary for water transportation are flushed with pure water and the effluents are collected in the first impinger. The total mass of sampled particulate material (either as a suspension or as a solution) is quantitatively transferred to a glass beaker and evaporated on a hot water bath to almost dryness. The residue is transported quantitatively into a preconditioned (24 h at 60°C, 2 h in an desiccator) and accurately weighed PVC container (volume, approx. 30 ml). The samples are placed in an oven at 60°C until all the water has evaporated, dried in an desiccator for 2 h, and weighed. In addition, blank samples, i.e. 200 ml of pure water,

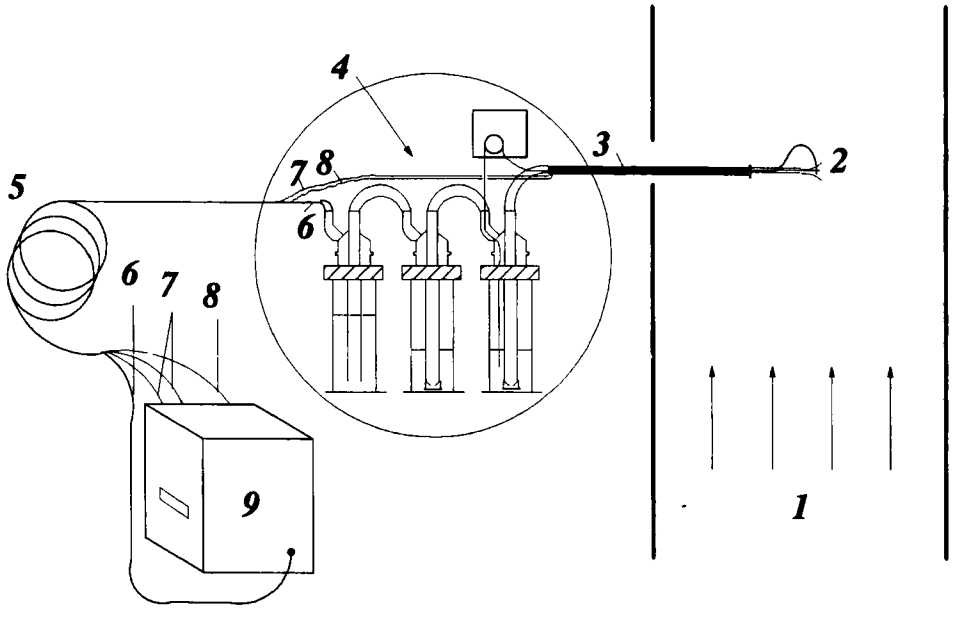

Figure 2 Set-up for isokinetic sampling of particulate material from a gas-carrying duct using the closed water circuit sampling system. 1, gas-carrying duct with particulates; 2, sharp-edged entry nozzle, S-type pitot tube and a thermocouple; 3, stainless-steel sampling probe tube containing a 1/2″ glass tube and a 1/8″ teflon capillary for transportation of a water medium to the front, i.e. where the entry nozzle is connected to the sampling probe tube; 4, closed water circuit sampling system, for details see Figure 3; 5, umbilical cord containing a sampling line (6), connected to the closed water circuit sampling system, two PVC lines (7) in connection with the pitot tube and an electrical wire (8), connected to the thermocouple; 9, Zambelli 6000⁺ automated isokinetic sampling unit.

were treated in the same way and the finally weighed mass (usually approx. 1 mg) was subtracted from the mass of particulate material found in the first impinger. Breakthrough of water with particulate material from the first to the second impinger was never observed; the dry mass in the second impinger was equal to that of 200 ml of pure water.

RESULTS AND DISCUSSION

In order to investigate its applicability, the new sampling technique has been compared with the conventional method using an in-stack flat filter. To this end a vertical gas-carrying duct has been used with a relatively stable mass flow rate of particulate material and mild conditions in terms of temperature and water vapour content. The concentration of particulate material has been determined in triplicate for each method by alternating sampling periods of approx. 45 min. Furthermore, samples have been taken for equal periods of time at the same pre-selected positions in the duct cross section.

The results are summarized in Table 1. From these results it is clear that both sampling methods correlate rather well, taking into account that the mass flow rate of particulate material is always subject to fluctuations. The observed lower concentration

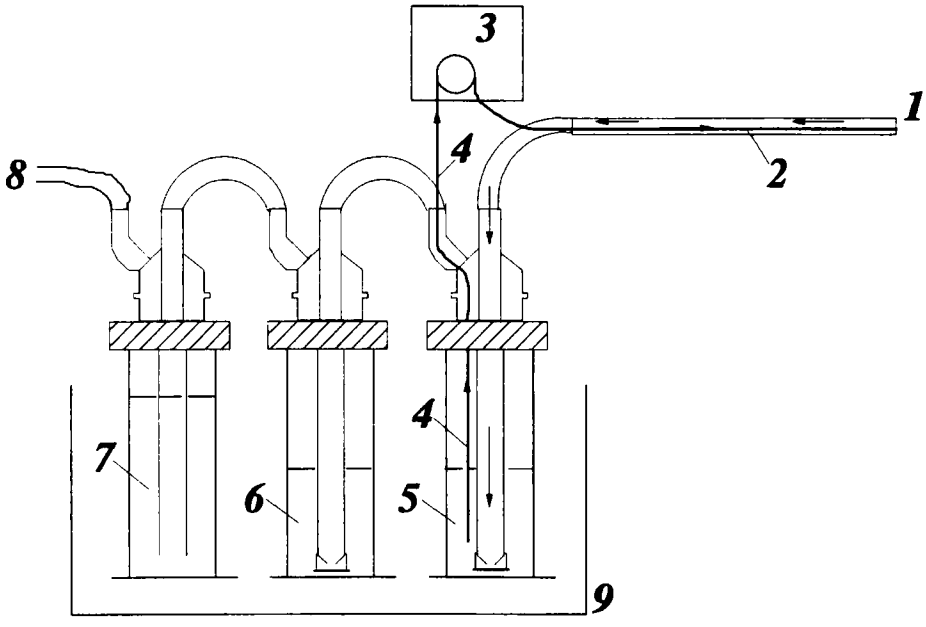

Figure 3 Detailed schematic presentation of the closed water circuit sampling system. 1, part of the stainless-steel sampling probe tube with a 1/2″ glass tube (inlet of the sampling system); 2, 1/8" PTFE capillary (inserted in the 1/2″ glass tube) for transportation of the water medium to the front of the sampling probe tube; 3, Cole-Parmer Masterflex 7544–30 tube pump, equipped with a head resulting in a flow rate of approximately 35 ml/min; 4, 1/8" PTFE capillary inserted in the 200-ml water medium (5) in the first impinger; 6, back-up impinger containing 200 ml pure water; 7, wash flask containing 300 g of silica for drying the sampled gas; 8, outlet of the sampling system, connected to the sampling line of the system (is No. 6 in Figure 2); 9, container for the accommodation of the sampling system, which can be filled with ice when the duct gas has temperatures above 100°C.

of particulate material between 13.00 h and 13.45 h may be explained by a temporary minor change of the process conditions. The average concentrations (with individual standard deviations) are 50.0 ± 10.5 mg/Nm^3 (dry) (2 df) and 58.6 ± 5.7 mg/Nm^3 (dry) (2 df) for the new (water circuit) and the conventional (flat filter) sampling method, respectively. From the F-test, i.e. $F_{2,2} = (10.5)^2/(5.70)^2 = 3.39$ with $F_{2,2}$ (1-sided, 0.95) = 19, it follows that both standard deviations can be pooled, giving:

$$s^2_{pooled} = [10.5^2\,(3-1) + 5.7^2\,(3-1)]/(3+3-2) = 71.37 \text{ and } s_{pooled} = 8.45 \text{ (4 df)}.$$

The (HO) hypothesis that both results are equal has been checked by the t-test, i.e. $t = (58.6-50.0)/s_{pooled} = 1.02$ with t (2-sided, 0.95, 4 df) = 2.78.

This confirms that the results obtained with the conventional and the new sampling method are equal with 95% confidence.

In order to test the new sampling method under more severe conditions, it has been applied to the determination of the efficiency of two installations used for the reduction of dust emissions. These measurements cannot be performed with the conventional sampling methods due to filter blockage, water condensation and loss of particulate material in the sampling probe tube.

Table 1 Determination of concentration of particulate material in a vertical gas-carrying duct (T = 47°C; water vapour content, 5 g/Nm^3) by the conventional sampling method using a glass fibre flat filter and the new sampling method using the new sampling method (closed water circuit).

Sampling method applied[1]	*Sampling period (h)*	*Total intake gas volume*[2] *(Nm^3 (dry))*	*Mass of particulate matter sampled*[3] *(mg)*	*Concentration of particulate matter*[4] *(mg/Nm^3 (dry))*
Flat filter	09.45–10.30	0.805	50.8	63.1
Water circuit	11.40–11.25	0.803	47.3	58.9
Flat filter	12.07–12.52	0.701	42.5	60.6
Water circuit	13.00–13.45	0.748	28.7	38.4
Flat filter	13.57–14.44	0.769	40.1	52.2
Water circuit	14.52–15.37	0.769	40.5	52.7

[1]Total sampling set-ups are presented in Figure 1 (using a flat filter) and Figure 2 (using a closed water circuit).
[2]Under standard conditions, i.e. 0°C and 101.3 kPa.
[3]Results obtained using the water circuit sampling method were corrected for the blank, i.e. 200 ml pure water containing 1.1 mg dry mass.
[4]Averages and individual standard deviations are:
flat filter: 58.6 ± 5.7 (= 59 ± 6) mg/Nm^3 (dry) (2 df) (= degrees of freedom), water circuit: 50.0 ± 10.5 (= 50 ± 11) mg/Nm^3 (dry) (2 df).

The results of these investigations are summarized in Tables 2 and 3. The results presented in Table 2 clearly demonstrate the applicability of the new sampling method at very high mass flow rates of particulate material and a high water vapour content. The ratios of the simultaneously measured particulate material concentration before and after the scrubber were very similar. For two different sampling periods values of 6995/1382 = 5.06 and 6519/1256 = 5.19 were obtained (Table 2).

The results of Table 3 illustrate that the described sampling method is also applicable to gas streams with a temperature of over 100°C, the boiling point of water. For such measurements the container with the impingers is filled with ice; through this, cold water is pumped to the front of the sampling probe thus preventing any problems due to boiling

Table 2 Determination of concentration of particulate material in a gas-carrying duct before (T = 74°C; water vapour content, 134 g/Nm^3) and after (T = 50.5°C; water vapour content, 66 g/Nm^3) a scrubber using the new sampling method.

Sampling point and period	*Total intake gas volume*[1] *(Nm^3 (dry))*	*Mass of particulate matter sampled*[2] *(mg)*	*Concentration of particulate matter (mg/Nm^3 (dry))*
Before scrubber 11.30–12.30 h	0.600	4197	6995
After scrubber 11.30–12.30 h	0.694	959	1382
Before scrubber 13.00–13.50 h	0.553	3603	6519
After scrubber 13.10–13.55 h	0.502	630	1256

[1]Under standard conditions, i.e. 0°C and 101.3 kPa;
[2]Corrected for the blank (i.e. 1.1 mg in 200 ml pure water); breakthrough of particulate material from the first to the second impinger was not observed.

Table 3 Determination of concentration of particulate material in a gas-carrying duct before (T = 115°C; water vapour content, 141 g/Nm^3) and after (T = 73.5°C; water vapout content, 123 g/Nm^3) a scrubber using the new sampling method.

Sampling point and period	*Total intake gas volume[1] (Nm^3 (dry))*	*Mass of particulate matter sampled[2] (mg)*	*Concentration of particulate matter (mg/Nm^3 (dry))*
Before scrubber 11.25–12.10 h	0.337	163.4	485
After scrubber 11.15–12.15 h	0.512	11.3	22.1
Before scrubber 13.32–14.17 h	0.302	174.1	576
After scrubber 13.30–14.30 h	0.575	12.3	21.4
Before scrubber 14.58–15.43 h	0.330	194.0	588
After scrubber 15.00–16.00 h	0.577	10.4	18.0

[1]Under standard conditions, i.e. 0°C and 101.3 kPa;
[2]Corrected for the blank (i.e. 1.1 mg in 200 ml pure water); breakthrough of particulate material from the first to the second impinger was not observed.

of the circulating water. From Table 3 it will be clear that even low amounts of particulate material, down to 20 mg/Nm^3 (dry), can be determined with a sampling time of only one hour.

CONCLUSIONS

A new method for the determination of particulate material in gas-carrying ducts has been presented. The method can be used under extreme conditions with respect to high particulate material concentrations and humidity, where the 'classical' in- (or out-)stack filter methods fail.

Under moderate conditions, for which the gas filtration technique is also applicable, both methods produce data which correlate well.

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