A Two-Stage Virtual Impactor for In-Stack Sampling of PM$_{2.5}$ and PM$_{10}$ in Flue Gas of Stationary Sources

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ABSTRACT

Two ISO standard methods for in-stack sampling and measurement of PM$_{2.5}$ and PM$_{10}$ mass concentrations in flue gas from stationary sources were published in 2009 (ISO 23210, conventional cascade impactors) and 2012 (ISO 13271, virtual impactors). The performances of these two methods in terms of PM$_{2.5}$ separation efficiency and the accuracy of measured mass concentration were compared at the same sampling point and conditions both using laboratory-scale model flue gas with different dust concentrations and using real flue gas sampled from a test facility for pulverized coal combustion. The virtual impactor showed very satisfactory performance for PM$_{2.5}$ mass concentration and separation efficiency within the investigated range of mass concentrations and ambient conditions. The conventional cascade impactor method overestimated PM$_{2.5}$ mass concentration by more than 25% due to particle bounce and re-entrainment of coarse particles from the collection plates. During in-stack PM$_{2.5}$ sampling from coal combustion flue gas with reactive components at high temperature, the use of greased plates with the conventional impactor caused overestimation of mass concentration, even when grease with high temperature endurance was used. The use of a quartz-fiber filters on the impaction plates reduced overestimation but particle bounce and re-entrainment still remained.

Keywords: PM$_{2.5}$; PM$_{10}$; Mass concentration; Stationary sources; Virtual impactor; Conventional impactor; Particle bounce.

INTRODUCTION

PM$_{2.5}$ particulates invade the pulmonary alveoli and are considered to cause various diseases (heart disease, lung disease, etc.); they are therefore being closely monitored around the world as a severe environmental issue (Dockery et al., 1993; Lighty et al., 2000; Pope et al., 2009). The specialized cancer agency of the World Health Organization, the International Agency for Research on Cancer (IARC), classified particulate matter as carcinogenic to humans (Group 1) in 2013 (Loomis et al., 2013).

In 2012, the U.S. environmental standard for annual average PM$_{2.5}$ concentration was revised from 15 to 12 µg m$^{-3}$. New environmental standards were also established in Japan (2009) and China (2012). Recently published data for atmospheric PM$_{2.5}$ mass concentration substantially exceed the above environmental quality standard in East- and South Asian areas. For example, official data from the Chinese Ministry of Environmental Protection for the year 2013 report average annual PM$_{2.5}$ air pollution mass concentration of 90.1 µg m$^{-3}$ in Beijing, which exceeds the Chinese national standard (35 µg m$^{-3}$). The environmental impacts of anthropogenic PM$_{2.5}$ emissions, such as from mobile and stationary sources, need to be determined in order to institute appropriate reduction measures.

Conversely, the contribution from stationary sources has received somewhat limited attention despite several studies of fine particulate emissions from stationary sources (Mohr et al., 1996; Senior et al., 2000). However, since PM$_{2.5}$ air pollution emerged as a serious problem, there is greater necessity for reliable measurement of PM$_{2.5}$ and PM$_{10}$ mass concentration emitted from stationary sources. Two new standard methods for measurement of PM$_{2.5}$ and PM$_{10}$ mass concentration from stationary sources were deliberated by ISO TC146/SC1/WG20 from 2004. Based on long-term discussions, one standard method using conventional impactors with impaction plates was published as ISO 23210: 2009, and another method using impactors with virtual
impaction surface was also published as ISO 13271: 2012. The PM$_{2.5}$ and PM$_{10}$ separation efficiencies of both impaction methods comply with ISO 7708: 1995, which describes the ratios of particles entering the human body through breathing, as shown in Fig. 1 for corresponding ISO 7708 curves.

Conventional impactors have particle collection plates as the impaction surface, on which particles within a certain size interval are collected following inertial size-separation. The bounce (Dzubay et al., 1976) and re-entrainment (Markowski, 1984) of coarse particles at the collection plates frequently result in substantial errors in the measured mass concentrations, as shown in Fig. 1. These phenomena are suppressed to some extent by the use of greased impaction surfaces (Turner and Hering, 1987) as applied to environmental PM$_{2.5}$ measurements (Hillamo and Kauppinen, 1991; Demokritou et al., 2004; Saarikoski et al., 2008; Kumar and Gupta, 2015). However, for in-stack measurements, commercial greases that maintain effective stickiness and constant weight under various flue gas conditions, high temperature and in the presence of reactive components have proven to be unreliable. Although porous media (typically quartz-fiber filters) were used instead (Barr et al., 1982; Huang and Tsai, 2002; John et al., 2003; Saarikoski et al., 2008; Huang and Lin, 2010), no quantitative data are available on measurement overestimation during in-stack uses at high temperatures. Because of above reasons, the limit of permissible deviation by the bounce and re-entrainment of coarse particles larger than 5.0 µm in diameter is about 30% in ISO 23210 for conventional impactors.

A virtual impactor design (Conner, 1966) achieves inertial size-separation via a space of relatively slow-moving air within a cavity of a receiving nozzle, and thereby avoids particle bounce and re-entrainment. Various modifications and improvements to this type of impactors (McFarland et al., 1978; Masuda et al., 1979; Loo and Cork, 1988; Kauppinen et al., 1989; Sioutas et al., 1994; Gotoh and Masuda, 2001) include a multiple-nozzle virtual impaction surface impactor (hereinafter “VIS impactor”) that was originally built for use in the Space Shuttle (Szymanski and Liu, 1989), and then subsequently applied to ambient air PM$_{10}$ and PM$_{2.5}$ sampling (Prasserttachato et al., 2006). However, no previous studies have compared the performance of conventional and virtual impactors using the same flue gas loaded with fine particles at a relatively high temperature.

The objective of this work is to verify the performance and applicability of the two-stage VIS impactor under a variety of relatively high dust concentrations and temperatures using laboratory-generated aerosol for in-stack measurement of stationary sources. In addition, by using real flue gas from a pulverized coal combustion test facility, the PM$_{2.5}$ separation efficiency and measured PM$_{2.5}$ mass concentration were compared for both impactor types. For a conventional cascade impactor with impaction substrates, the effect of using quartz-fiber filters or grease for collecting coarse particles at high temperature on measured PM$_{2.5}$ mass concentration is discussed.

**METHODS**

**Impactor Type 1: Two-Stage VIS Impactor**

The principle of separation at nozzles and the key design parameters for a VIS impactor are shown in Fig. 2. In its basic configuration, the separation stage consists of particle acceleration nozzles and particle collection nozzles whose diameters are indicated in Fig. 2 by $D_0$ and $D_1$ respectively. Each separation stage includes six pairs of particle acceleration and collection nozzles facing coaxially and
Fig. 2. Principle of operation of a virtual impaction surface (VIS) impactor.

located equidistantly in a circular arrangement. The particle-laden gas enters the particle acceleration nozzles and accelerates depending on $D_0$ and the total gas volume flow rate $Q_0$. Only a part of the gas stream leaving the acceleration nozzles enters the particle collection nozzles. The flow rate through the particle collection nozzles $Q_1$, which is called the minor flow rate, typically amounts to 10% of the total flow rate. The remaining major part of the flow ($Q_2$) is redirected and bypasses the particle collection nozzles. Consequently, particles larger than a certain aerodynamic size (cut-off size) are within the minor flow, received by the particle collection nozzles and collected on a filter. Fine particles smaller than this cut-off size stay in the major stream and are directed into the next separation stage. The arrangements of the nozzles and gas-passage in the VIS impactor are shown in Fig. 3. There are two separation stages (first stage: 10 µm cut-off, second stage: 2.5 µm cut-off). Particles larger than 10 µm are pre-cut and collected on the filter of the first stage; those of 2.5 µm to 10 µm are sampled on the filter of the second stage; and PM$_{2.5}$ are sampled on the PM$_{2.5}$ collection filter. The impactor components were constructed of stainless steel for its ability to withstand high temperature and corrosive gas.

The design of the impactor is based on impactor theory (Marple and Liu, 1974; Marple and Willeke, 1976). The separation characteristics of impactors are specified by means of the Stokes number $Stk$ at the particle separation nozzle. $Stk$ corresponds to the ratio of particle stopping distance to nozzle diameter and is given by

$$Stk = \frac{\rho_p d_p^2 U C_c}{9 \mu D_0} \tag{1}$$

where $\rho_p$, $d_p$, $U$, $C_c$, $\mu$, and $D_0$ are particle density, particle diameter, gas velocity through a particle acceleration nozzle, Cunningham’s correction factor, viscosity, and diameter of a particle acceleration nozzle, respectively.

Substitution of appropriate values of $Stk$ at 50% cut-off diameter, $\rho_p = 1000$ kg m$^{-3}$, and the value for 50% cut-off diameter $d_p$ (10 µm or 2.5 µm) into Eq. (1) gives a relationship between $U$ and $D_0$ at each separation stage. The main design parameters of a VIS impactor are shown.

Fig. 3. Cross-section diagram of VIS impactor for sampling of PM$_{2.5}$ and PM$_{10}$ in flue stack of stationary emission source.
in Table 1. The VIS impactor of the described dimensions is operated at 12.5 L min⁻¹ (for ambient air) in total suction flow entering the device. Distribution of the total suction flow showed minor gas flows of 1.0 L min⁻¹ for PM₁₀ cut-off stage, and 1.2 L min⁻¹ for PM₂.₅ cut-off stage; the gas flow rate through the PM₂.₅ collection filter was 10.3 L min⁻¹ (for ambient air). The 50% cut-off Stokes numbers Stk₅₀ for the stages of the present VIS impactor were 0.47 and 0.50 for PM₁₀ and PM₂.₅ cut-off stages respectively. The VIS impactor was calibrated in an earlier publication (Prassertachato et al., 2006). The VIS impactor separation performance using mono-dispersed particles is shown in Fig. 1. To calculate separation efficiencies, aerosol particles with aerodynamic diameters from 1 µm to 20 µm were generated from oleic acid tagged with uranine using a vibrating orifice aerosol generator (Szymanski and Liu, 1989). The aerosols generated were diluted with filtered air and charge-neutralized before being introduced into the impactor. The uranine concentration was measured using a fluorometer. The impactor performed well in measurements using monodispersed particles, with less than 5% deviation from ISO 7708.

In order to maintain the designed cut-off diameters in hot in-stack sampling situations, the suction gas flow rate was determined by keeping Stk constant regardless of variations in temperature, pressure, and gas composition from ambient air conditions. To approach the conditions to isokinetic sampling, a series of impactor entry nozzles of appropriate shape and differing inner diameters were prepared and selected in order to provide the best sampling conditions with reference to stack gas velocity and suction gas flow rate at the sampling point.

### Impactor Type 2: Two-Stage Conventional Cascade Impactor

A conventional cascade impactor with impaction plates (GMU-Cascade Impactor Johnas II, Paul Gothe, Bochum, Germany) was used for comparison with the VIS impactor. The GMU impactor has two separation stages (first stage: 10 µm cut-off, second stage: 2.5 µm cut-off). The first and second stages have 6 and 12 separation nozzles respectively. ISO 23210 indicates that this method is applicable to dust concentrations of less than 40 mg m⁻³ half-hourly average under standard conditions. Suction gas flow rate for the GMU impactor is 43.4 L min⁻¹ for ambient air sampling. Separation performance data for the GMU impactor (John et al., 2003) are also indicated in Fig. 1, for reference. In general, coating the impaction plates with grease limits particle bounce and re-entrainment at the impaction plates and improves the sampling performance of the impactor. However, in stacks at high temperatures in reactive atmospheres, substantial errors may occur in PM₁₀ and PM₂.₅ mass concentration measurements, caused by adhesion degradation and/or weight change of the coated grease.

In this work, the GMU impactor characteristics were tested with the impaction stages equipped with either quartz-fiber filters or greased metal plates as impaction surfaces. The grease used in these experiments was L200 Demnum Grease (Daikin Industries, Ltd.). The manufacturer information indicates that mass loss due to evaporation of this grease is less than 1% over a period of 22 h at 200°C in air. The amount of grease used for a coated stage was 1.5 (± 10%) mg cm⁻².

### Testing Facilities and Operation Conditions

#### Model Aerosol Stream Using Test Ash Powder

In order to evaluate the performance of the VIS impactor at high temperature and differing dust concentrations, a model aerosol stream simulating some industrial conditions was produced using a dust generation system equipped with a fluidized-bed aerosol generator (Model 3211, KANOMAX, Osaka, Japan) and a dilution tank in which aerosol mass concentration was reduced to one tenth of its initial value by the addition of clean, dry air. The model aerosol was composed of Type 1 testing powder #10 (Fly ash, mass-based median particle diameter of 5.2 µm, geometric standard deviation of 1.2, particle density of 2000–2300 kg m⁻³) (JIS Z 8901, 1995) and air. Particle mass concentration in the aerosol was controlled by the particle feed rate of the aerosol generator and ranged from 3 mg m⁻³ to 60 mg m⁻³. The sampling period for the impactors was chosen to obtain statistically reliable data, and was typically several minutes. Temperature was varied from 20°C to 200°C by heating.
the aerosol stream lines and the impactor using tape heaters.

Particle mass concentration and size distribution were examined periodically by substituting the impactor with a filter holder equipped with a plain glass-fiber filter to sample particles under similar conditions to the impactors. Particle size distribution was measured by a laser diffraction particle size analyzer (MT-3000, NIKKISO Co., Ltd., Japan) using a well dispersed liquid suspension of particles collected on a plain filter with the assistance of ultrasonication. A typical mass-based size distribution is shown in Fig. 4 as a function of the aerodynamic particle size. For the conversion of particle size (obtained via laser diffraction method) to aerodynamic particle size, values of particle density $\rho_p = 2150 \text{ kg m}^{-3}$ and dynamic shape factor $\phi = 1$ were used in the calculations. Although particle size distribution obtained in this way is not necessarily exactly the same as that for particles in airborne state, it shows a typical case of the agglomerates broken into their primary form by the ultrasonic wave. PM$_{2.5}$ mass fraction of the model aerosol calculated by the data in Fig. 4 and ISO 7708 separation curve was 20% of the total model aerosol and remained consistent irrespective of the experimental conditions.

Gravimetric analysis of impactor samples was carried out by weighing the substrates carefully before and after sampling on a microbalance (Mettler Toledo XS105, minimum scale value: 0.01 mg, automatic calibration) in a clean, laboratory room at constant humidity. The specifications of the microbalance were adequate for conducting the measurements. The filters weighed approximately 100 mg and the impaction substrates 400 mg; and the mass of the particles collected was over 1 mg. It is known that the stability of the filters and impaction substrates is important for PM$_{2.5}$ emission measurement (Kauppinen and Pakkanen, 1990). Before sampling, the filters and greased substrates were baked in an oven for 3 h at 220°C, in order to ensure substrate weight stability during in-stack sampling and subsequent evaluation.

**Pulverized Coal Combustion Test Facility**

In order to evaluate the performance and suitability of both types of impactors for industrial sampling of flue gas from pulverized coal combustion, a test facility was chosen for further experimental studies. The gas flow line is shown in Fig. 5. The coal combustion rate of the test facility was about 300 kg h$^{-1}$. This facility has two lines for a gas treatment system. Downstream of selective catalytic de-NO$_x$ equipment, one line has an electrostatic precipitator and gypsum limestone de-SO$_x$ equipment, whose structures are similar to those of actual commercial plants. This line is used for the experimental study of flue gas treatment. The second line has only a bag-filter, and is used to control the gas flow rate to the first line during the experiment. The measurement point was located downstream of the electrostatic precipitator. The inner diameter of the exhaust gas ducting was 0.15 m, and had a sampling port of inside diameter 0.15 m. Because the ducting diameter is small compared with the impactor length, neither a VIS impactor nor a GMU impactor can be placed directly in the flue gas ducting. The sampling port length was extended to 0.35 m and each impactor, equipped with a bent suction nozzle, was placed in a sampling port. Temperature-controlled waterproof rubber heaters were attached to the impactor to maintain it at the same temperature as that at the sampling point.

Prior to measuring PM$_{2.5}$ and PM$_{10}$ mass concentrations, the total dust concentration, and the main operational data for the flue gas were determined in accordance with JIS Z 8808. The results were: total dust concentration: 3.7 mg m$^{-3}$, gas velocity: 32.3 m s$^{-1}$, temperature: 127°C, water content: 11.5%, CO$_2$ content: 13.7%, and O$_2$ content: 5.5%. All particle concentrations indicated in the present work are
Fig. 5. Schematic gas flow line of a pulverized coal combustion test facility.

based on the gas volume at standard conditions (0°C and 1013 hPa). Alternating measurements were made of the VIS and GMU impactors equipped with either filters or grease-coated metal plates. Prior to PM$_{2.5}$ and PM$_{10}$ sampling the impactors were preheated for approximately 20 minutes, with the entry nozzle tip facing the opposite direction to the flue gas stream without gas suction. Based on the operational data, the suction gas flow rate was adjusted to keep each impactor constant at the recommended $St_k_{50}$. ISO 23210 and ISO 13271 suggest 90–130% ratio of entry nozzle gas velocity to sampling point gas velocity in order to maintain sampling error within ±15% based on the model proposed by Davies (1968). To meet this guideline, the entry nozzle inner diameter was chosen as 2.5 mm for the VIS impactor and 5 mm for the GMU impactor. Sampling times for the VIS and GMU impactors were 1 h and 0.5 h, giving total sampled gas volume of 0.54 m$^3$ and 0.84 m$^3$ at standard conditions (0°C, 1013 hPa), respectively. Gravimetric analysis of impactor samples used the same method as that for the model aerosol experiments.

RESULTS AND DISCUSSION

Test by Model Aerosol Stream

The relationship between the measured PM$_{2.5}$ concentration and total particle concentration is shown in Fig. 6. The effects of temperature on measured PM$_{2.5}$ mass concentration were also examined. Total particle concentration is calculated by summing the three fractions of particle concentrations. The lower black dashed line in Fig. 6 indicates the estimated PM$_{2.5}$ concentration assuming the separation efficiency indicated in ISO 7708 (cf. Fig. 1) and using model aerosol particle size distribution indicated in Fig. 4. In the case of the VIS impactor, measured PM$_{2.5}$ concentrations show very good agreement with the PM$_{2.5}$ line throughout the dust concentration range. For the GMU impactor, measured PM$_{2.5}$ concentrations exceed the PM$_{2.5}$ line. The GMU results were much improved when the impaction plates were coated with grease compared with those obtained using quartz-fiber filters, but the values were still higher than those obtained by the VIS impactor. The value when using the filter was more than double that obtained with the VIS impactor, and approximately 30% greater when using greased plates under the described experimental conditions. The upper light-gray dashed line in Fig. 6 shows particle concentration at the PM$_{2.5}$ filter if the GMU impactor had achieved the separation efficiency for using quartz-fiber filters as impaction plates (John et al., 2003; cf. Fig. 1). An agreement was obtained between the estimated values (the upper light-gray dashed line) and the experimental results for the GMU impactor when grease was used. To emphasize the issue, SEM images of particles sampled on PM$_{2.5}$ collection filters are shown in Fig. 7. The filters sampled at total particle concentrations of 10 mg m$^{-3}$ and 30 mg m$^{-3}$ were used for evaluation. The size of the circles near the scale bars (Fig. 7) represents an aerodynamic diameter of 2.5 µm, which corresponds to 1.7 µm actual particle diameter. In images for the VIS impactor, particles larger than 2.5 µm aerodynamic diameter were rarely observed at either of the investigated dust concentrations, thereby confirming a well-defined separation performance. On GMU filters, however, a substantial number of particles larger than 2.5 µm aerodynamic diameter were rarely observed at either of the investigated dust concentrations, thereby confirming a well-defined separation performance. When the GMU impactor was operated with greased plates, although there were fewer coarse particles present on the incorrect impactor stage at low dust concentration, there were still considerable numbers of coarse particles observed at high dust concentrations.

Evidently, even when using greased plates, particle bounce
Fig. 6. Comparison of PM$_{2.5}$ concentration present in model aerosol (lower dashed line) and that measured by VIS impactor and by GMU impactor with quartz-fiber filters or greased plates as impaction surface. PM$_{2.5}$ concentration was assessed using the actual powder size distribution (Fig. 4) and impactor cut-off efficiency (Fig. 1).

Fig. 7. SEM images of model aerosol particles collected on PM$_{2.5}$ collection filter; VIS impactor (a-1 and a-2), GMU impactor with glass-fiber filter impaction plates (b-1 and b-2), GMU impactor with greased plates (c-1 and c-2); aerosol total concentration 30 mg m$^{-3}$ (a-1, b-1, c-1), 10 mg m$^{-3}$ (a-2, b-2, c-2). The size of the circle next to the scale bar represents the aerodynamic cut-off diameter at PM$_{2.5}$ separation stage.
was not entirely eliminated in the experiments with the model aerosol stream at room temperature, even though these experimental conditions were much less demanding than actual in-stack sampling under industrial conditions. In the VIS design, errors due to bounce and re-entrainment were practically eliminated and were not observed, because the collected aerosol is not impacting a plate but instead enters receiving nozzles into a cavity before being collected on a filter. The VIS impactor is evidently better suited to in-stack measurement of stationary sources than conventional cascade impactors using the impaction plates design.

Test in Pulverized Coal Combustion Test Facility

Mass concentrations were calculated from the weight of each particle fraction and total suction gas volume (see Fig. 8) for the VIS impactor and the GMU impactor with quartz-fiber filters and greased plates. The totals for each of the stages were very similar, thereby confirming the appropriate selection of sampling inlet. Compared with the VIS impactor, the GMU design shows higher concentration of particles smaller than 2.5 µm and lower concentration of particle sizes 2.5–10 µm. Under the experimental conditions described in this study, the PM$_{2.5}$ value obtained using the filter was 24% greater than that of the VIS impactor. And that using the greased impaction plates was 52% greater than that of the VIS impactor.

The performance differences between the three methods were evaluated via SEM images of PM$_{2.5}$ particles collected on filters; typical cases are shown in Fig. 9. For the VIS impactor, almost all particles had an aerodynamic diameter smaller than 2.5 µm, which is highly consistent with that obtained using model aerosol. Contrary to the VIS impactor, the images from the GMU impactor show quite a number of coarse particles larger than 2.5 µm present on the PM$_{2.5}$ collection filter, which was attributed to particle bounce from the impaction surface and re-entrainment. Coating the impaction surface with grease was expected to reduce these artifacts. However, the results were similarly poor to the fiber filter case. This is likely due to temperature degradation of the grease coating and the greater hardness of the metal impaction surface. The fact that the VIS design has no impaction plates and does not require any grease coating has advantages for ease of use and eliminates, de facto, bounce effects. Consequently, the measured PM$_{2.5}$ and PM$_{10}$ fractions are not biased by the measurement method. It is therefore evident that the VIS impactor is better suited to in-stack measurement of stationary sources than is a conventional cascade impactor using impaction plates.

CONCLUSIONS

Two ISO methods—conventional and virtual impactors—were compared for in-stack sampling and measurement of PM$_{2.5}$ mass concentration from stationary sources. The VIS impactor showed remarkably good performance within the investigated range of mass concentrations and ambient parameters. The conventional impactor overestimated PM$_{2.5}$ concentrations due to particle bounce and re-entrainment, even when an adhesive coating such as grease was applied to the impaction plates. For high-temperature coal combustion flue gas, the use of greased plates in the conventional impactor caused the overestimation of PM$_{2.5}$ concentration. Using quartz-fiber filters somewhat improved the performance compared with that of the greased plates; however, coarse particle bounce and re-entrainment persisted, resulting still in substantial overestimation of PM$_{2.5}$ values. Evidently, the method of virtual particle separation employed in the VIS impactor provides superior performance over the conventional cascade impactor for in-stack measurement of PM$_{10}$/PM$_{2.5}$ from stationary sources.

Fig. 8. Concentration of coal combustion dust collected on VIS impactor and GMU impactor (with quartz-fiber filters or greased plates) at PM$_{10}$ and PM$_{2.5}$ separation stages.
Fig. 9. SEM images of coal ash particles collected on PM$_{2.5}$ collection filter by VIS impactor and GMU impactor (with quartz-fiber filters or greased plates). The size of the circle next to the scale bar represents the aerodynamic cut-off diameter at PM$_{2.5}$ separation stage.

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