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Field Test of a Porous-Metal Denuder Sampler

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In this study, acidic/basic gas and particle concentrations determined with a porous-metal denuder and other samplers, that is, silica gel tube, impinger, honeycomb denuder system (HDS), Marple personal cascade impactor, and filter cassette, were compared in the ambient environment and different industrial sites in Taiwan. Results show that the concentrations determined by the denuder are very close to those of other samplers, with excellent correlation. The denuder also was tested successfully to determine H₂SO₄ size distribution in a lead-acid factory. One-way ANOVA shows no significant differences ($P > 0.05$) between the porous-metal denuder (PDS) and other samplers. The denuder is compact in size and suitable for sampling in the workplace and ambient environment.

INTRODUCTION

A diffusion denuder is a sampler that removes gases from an aerosol stream to measure their concentrations separately. Gas or vapor molecules diffuse rapidly to the wall of a diffusion sampler and adsorb onto the wall coated with a suitable material. The gas concentration can be determined by extracting the coated substrates and analyzing the samples (Koutrakis et al. 1993; Poon et al. 1994; Possanzini et al. 1983; Pui et al. 1990; Sioutas et al. 1996).

Various denuders were designed and reported in the last 20 years. Pui and colleagues (1990) designed a compact coiled denuder and compared the performance with an annular denuder (Possanzini et al. 1983). Koutrakis and colleagues (1993)

and Sioutas and colleagues (1996) developed a glass honeycomb denuder/filter pack system to collect atmospheric gases and particles. The system is considerably smaller than the annular denuder system and can be used easily for large field studies. Poon and colleagues (1994) developed a high-efficiency compact diffusion denuder using porous-metal discs. The smaller size of the denuder makes it possible to design a compact atmospheric and/or indoor denuder sampling system. Using the same porous-metal discs (diameter: 2.54 cm, pore size: 100 μm , thickness: 0.317 cm, P/N 1000, Mott Inc., Farmington, Connecticut), Tsai and colleagues (2001a) designed and tested a PDS in the laboratory. The entire casing and substrate support were made of Teflon, and sampling flowrate was fixed to be 2 L/min. The sampler consists of a two-stage cascade impactor (having cutoff aerodynamic diameters of 9.5 and 2.0 μm , respectively) to collect liquid particles followed by two porous-metal discs to collect basic and acidic gases, respectively. The denuder was tested for gas collection efficiency and capacity at gas concentration two times the permissible exposure limit (PEL, promulgated by Taiwan IOSH, Institute of Occupational Safety and Health). The test data indicated that the gas collection efficiency was high and the capacity was sufficient for the acidic/basic gas sampling in the workplace.

Tsai and colleagues (2001b) compared the collection efficiency and capacity of the denuder with the silica gel tube method and an additional impinger method at gas concentration two times the PEL in the laboratory. The collection efficiencies of the PDS coated with 5% sodium carbonate/glycerin on the PDS, for nitric acid, hydrogen chloride, and hydrogen fluoride were higher than those of the silica gel tube and the impinger when the sampling time was less than 3 hrs. For ammonia, the performance of the PDS, silica gel tube, and impinger was almost the same.

The PDS is considerably more compact, simpler in design, and easier to handle than the annular denuder system and the HDS for field sampling. In addition, the PDS can measure

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individual concentrations of gas and particulate simultaneously. In comparison, the Marple personal cascade impactor and filter cassette usually are used for particulate concentration measurement, and silica gel tube and impinger usually are used to measure gas concentration only. The silica gel tube is used for the collection of six inorganic acids including hydrofluoric, hydrochloric, phosphoric, hydrobromic, nitric, and sulfuric acids in a single sample, and the analysis is done by ion chromatography at the National Institute of Occupational Safety and Health (NIOSH 1994). The impinger is used in Method 2401 for sampling ammonia gas at the Taiwan Institute of Occupational Safety and Health (TIOSH 1994).

In this work the new PDS was designed and tested for monitoring acidic/basic gases and aerosols at the ambient environment around the Hsinchu Science Park and different industrial sites in Taiwan. The measured concentrations were compared with those obtained with other sampling techniques, that is, silica gel tube, filter cassette, impinger, and HDS. The denuder was tested also for size distribution of sulfuric acid particles in a lead-acid battery plant and compared with that measured by the eight-stage Marple personal cascade impactor (Rubow et al. 1987).

TEST SITES AND METHODS

Sampler Design

The denuder tested has a five-stage cascade impactor in front of two denuder discs. The schematic diagram is shown in Figure 1. The inner diameter of the denuder is 30.6 mm, and the total length is 136 mm. Each stage of the cascade impactor has a single round nozzle whose diameter is 7.2, 4.8, 3.6, 2.6, and 1.9 mm for stages 1 to 5, respectively. The cutoff aerodynamic diameter of stages 1 to 5 was tested to be 9.5, 6.7, 4.8, 3.2, and 2.0 μm , respectively. The sampling flowrate is 2 L/min. The inlet of the denuder is an annular slot based on the design of Wistchger and colleagues (1997). The gap of the slot is determined by the thickness of four pins.

Ambient Environment

To compare the ambient concentrations of inorganic acidic/basic aerosols and gases, the new PDS and HDSs were collocated at 1 m height at the surroundings of the Hsinchu Science Park in Taiwan, for a sampling period of 12.0 hrs. In the study, the substrates of the five-stage impactor were coated with silicon grease to prevent particle bounce. The impactor samples were not analyzed for ionic concentrations because of contamination by silicon grease. Instead, the concentration of fine particles collected after the filter of the denuder was analyzed and compared with those determined by the HDS.

Industrial Test Sites

(i) Lead-Acid Battery Factory

The new five-stage impactor PDS was tested with a silica gel tube and filter cassette for total sulfuric acid concentrations at the forming area of a lead-acid battery factory. The sulfuric acid droplets were collected on various impactor stages of the denuder, and the total concentration was the sum of the concentrations at the five stages. The Zefluor filter with a 37 cm diameter (2.0 μm pore size, Gelman Science) was used to collect acidic aerosol on the filter cassette at a flowrate of 2 L/min. The samplers were placed 0.5 m away from the forming trough and a distance of 1 m above the ground, for a sampling period of 1.0 hr. The size distribution of liquid sulfuric acid also was sampled simultaneously by a Marple personal impactor and compared with that determined by the PDS. Since liquid particles do not bounce, substrates were not coated (PE filters for the Marple personal impactor, and porous-metal substrates for the five-stage impactor of the denuder) and were extracted by the deionized distilled water. The Marple personal impactor was an eight-stage impactor with cut points ranging from 0.5 μm to 21 μm in aerodynamic diameter. The after filter of the Marple personal impactor was a PVC filter. The sampling flow rate is 2 L/min.

(ii) Sulfuric Acid Production Plant

This factory mainly produces 98% industrial and 125% fuming sulfuric acid. The samplers, that is, the new denuder, silica

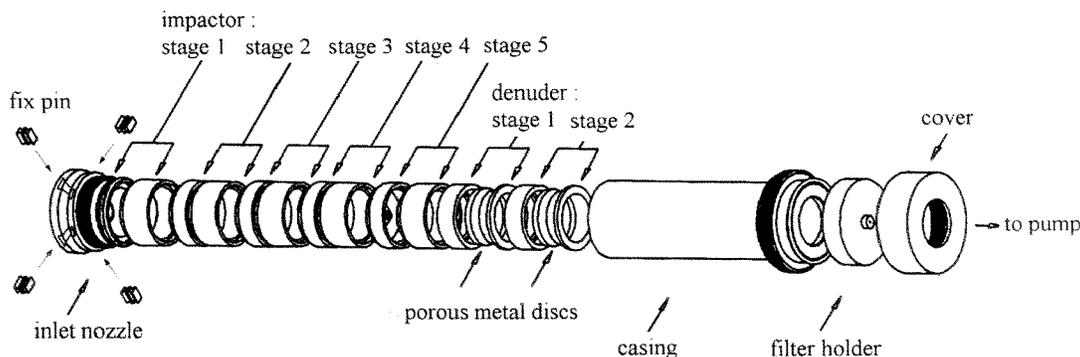


Figure 1. Schematic diagram of the porous-metal denuder sampler.

gel tube, and filter cassette were placed about 1 m away from the storage tank of the sulfuric acid and 1.5 m above from the ground for a sampling period of 2 hrs. In this factory, the concentration of sulfuric acid mist was comparatively low, because it was an outdoor sampling.

(iii) Semiconductor Factory

The samplers, including the new PDS, HDS, and silica gel tube were set up near the wastewater treatment plant, outside a semiconductor factory. Hydrofluoric acid was used to clean the wafer in the factory. For the treatment of wastewater, this factory uses CaCl_2 as a coagulator to capture F^- ions and thus form CaF_2 . Hydrochloric acid is added to the wastewater treating process in order to increase the coagulation efficiency in the pH range 5 to 7. Therefore, at the field site some acidic gases like HCl and HF apparently existed. The samplers were placed about a distance of 1 m above and away from the hydrofluoric acid wastewater reaction tank for 8-hr sampling.

(iv) Fertilizer Factory

This fertilizer factory produces melamine by injecting the urea into the reactor filled with ammonia gas. The samplers, including the new PDS, HDS, and impinger were placed near the ammonia storage tank with a distance of 1 m away and 1 m above the ground for 1-hr sampling. The study was mainly aimed at comparing the ammonia gas concentrations of different samplers.

Laboratory Analysis

(i) Porous-Metal Denuder

The porous-metal discs were coated with different solutions. For acidic gases, 10 ml, 5% (w/v) sodium carbonate, 1% (w/v) glycerol in 1:1 (v/v) methanol/water solution was used. For ammonia gas, 10 ml, 4% (w/v) citric acid in ethanol was used. The coating solution concentrations were higher than that of the denuder used for atmospheric sampling (Poon et al. 1994; Sioutas 1996) since the latter was found to be insufficient for the high gas concentration in the workplace. After coating, the porous-metal discs were dried by passing nitrogen gas through them. After sampling, the porous-metal discs were extracted with deionized distilled water in a low-pressure chamber (at 0.2 atm). The low-pressure chamber was used to help drive out air bubbles trapped in the porous-metal discs so that the adsorbed species can be extracted completely.

(ii) Honeycomb Denuder System

The components of the HDS include an impactor with the cutoff aerodynamic diameter at $2.5 \mu\text{m}$, a glass-transition section, two honeycomb denuders, a spacer, and a filter pack. The flowrate of the HDS is 10 L/min. The honeycombs of the HDS were coated using 1% (w/v) sodium carbonate, 1% (w/v) glycerol in a 1:1 (v/v) methanol/water solution for acid gases. For ammonia gas, 1% (w/v) citric acid in ethanol was used. A three-

stage filter pack was placed downstream of the denuders. The filter pack consists of a Teflon filter (Gelman Science, $2\text{-}\mu\text{m}$ pore size) to collect fine particles, a nylon filter (Gelman Science, $1\text{-}\mu\text{m}$ pore size) to collect HNO_3 and HCl, and a glass fiber filter (AP40, Millipore Inc.) coated with citric acid to collect NH_3 that volatilized from the collected particles on the Teflon filter. The concentrations of the samples were determined by an ion chromatograph (Model 4500i, Dionex Corp., California).

(iii) Silica Gel Tubes

The commercially available silica gel tubes (SKC 226-10-03, SKC, Inc.) were used for sampling inorganic acids. The silica gel tubes contained two sections of washed silica gel (first section: 400 mg placed by a thick glass fiber filter plug, second section: 200 mg retained by a urethane foam plug). The sampling flow rate was 0.5 L/min. The samples of the silica gel were analyzed according to the inorganic acids method (NIOSH Method 7903).

In this study, the Quality Assurance and Quality Control (QA/QC) procedure includes establishment of a calibration curve using standard solutions and a method detection limit (MDL), blank analysis, repeated analysis, and spike sample analysis. The MDL was determined as three times the standard deviation of repeated analysis at five times the lowest possible standard concentration. The MDL was 0.2, 0.11, 0.02, 0.05, 0.07, 0.09 ppb for HF, HCl, HNO_2 , HNO_3 , H_2SO_4 , and NH_3 gases, respectively, based on 12-hr sampling at 2 L/min. The blank values of porous-metal disc for the ion species were nondetectable. The results of precision analysis showed that the relative bias of detected values was below 5%. The recovery efficiencies were estimated using spike samples with the concentrations of two times the permissible exposure limit promulgated by Taiwan IOSH based on 15-min sampling at 2 L/min. The corresponding recovery efficiency from the porous-metal disc for the ion species F^- , Cl^- , NO_2^- , NO_3^- , SO_4^{2-} , and NH_4^+ were 95.3 ± 2.1 , 97.9 ± 0.9 , 97.3 ± 0.8 , 96.6 ± 0.7 , 95.3 ± 1.4 , and $96.9 \pm 1.3\%$, respectively.

RESULTS AND DISCUSSION

Particulate Concentrations

(i) Ambient Environment

In Figures 2a–2f we show that fine particle F^- , Cl^- , NO_2^- , NO_3^- , SO_4^{2-} , and NH_4^+ concentrations measured with the PDS plotted against those measured by the HDS in the ambient air of Hsinchu Science Park. The F^- , Cl^- , NO_2^- , NO_3^- , SO_4^{2-} , and NH_4^+ concentrations determined by the PDS and the HDS were highly correlated, with R^2 of 0.992, 0.996, 0.998, 0.999, 0.983, and 0.988, respectively. One-way ANOVA showed no significant differences ($P > 0.05$) for the six replicate samples taken with the PDS and HDS. The particulate concentrations and mean ratios of PDS and HDS are shown in Table 1. The mean values are close to 1, indicating the measurements are in agreement.

(ii) Lead-Acid Battery Factory

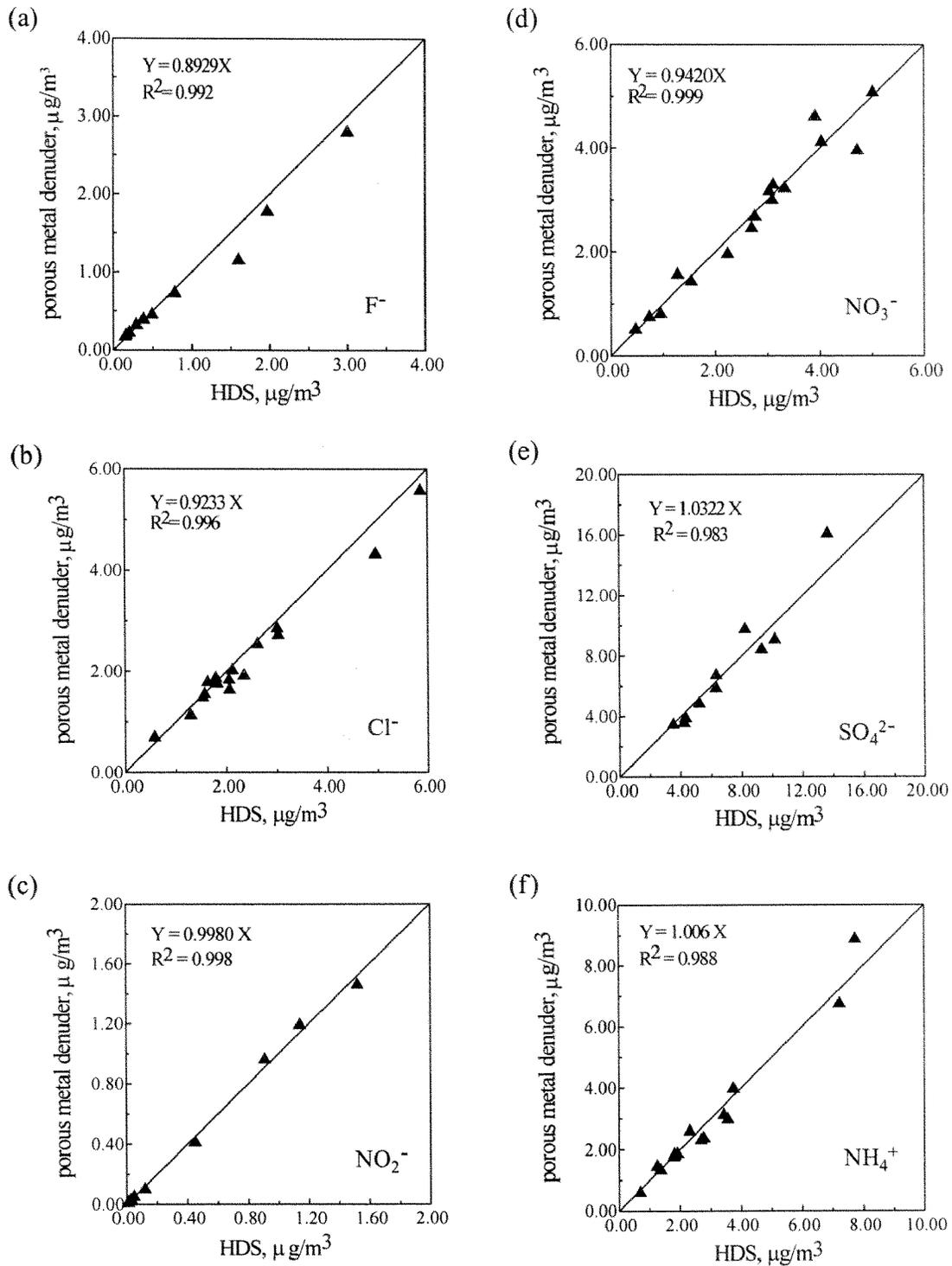


Figure 2. Fine particle (a) F^- , (b) Cl^- , (c) NO_2^- , (d) NO_3^- , (e) SO_4^{2-} , (f) NH_4^+ concentrations measured with the PDS in comparison with the HDS in the ambient air around the Hsinchu Science Park.

Figures 3a and 3b show the size distribution of the sulfuric acid mist in the lead-acid battery factory. The mass median aerodynamic diameter (MMAD) measured with the PDS and Marple personal cascade impactor was $6.92 \mu m$ ($\sigma_g = 1.27$)

and $6.99 \mu m$ ($\sigma_g = 1.45$), respectively. It indicates that the particle distribution has a single mode and the particles mainly distributed in $6.0\text{--}9.8 \mu m$ stage of the Marple personal cascade impactor in the sampling site. Similarly, the sulfuric acid

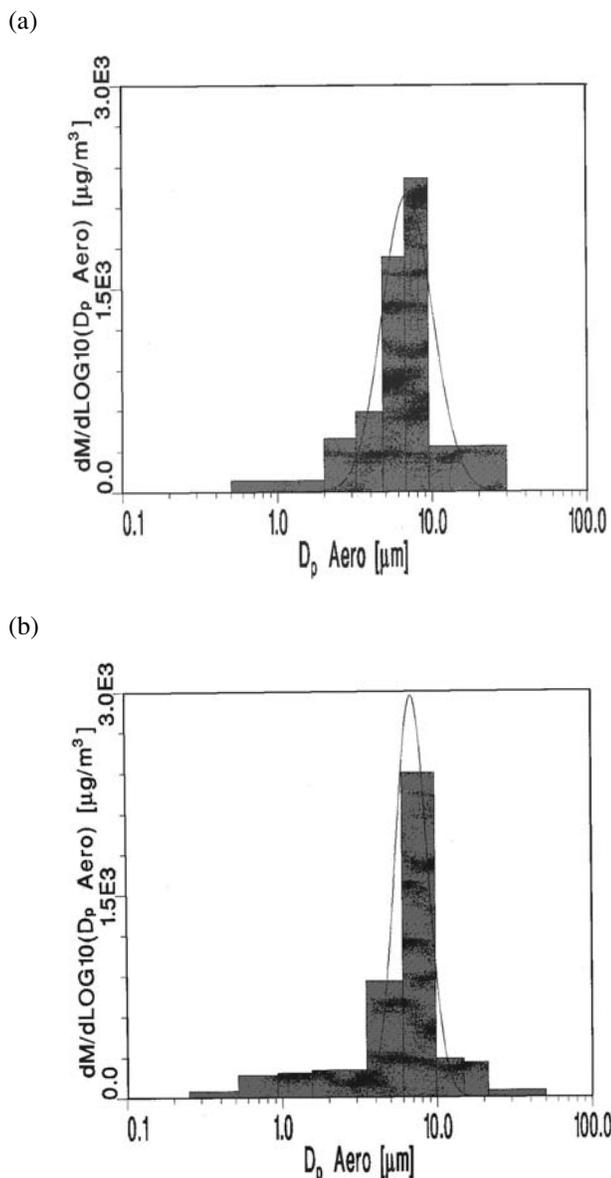


Figure 3. Sulfuric acid particle size distribution measured with (a) PDS, (b) Marple personal cascade impactor in the lead-acid battery factory.

mist distributed in 6.7–9.5 μm stage of the PDS. Figures 4a and 4b show the comparative study of total concentrations of sulfuric acid mist in the lead-acid battery and the sulfuric acid factory sampled with the PDS and the silica gel tube and filter cassette. For the lead-acid battery, the results indicate that the PDS has high correlation with other samplers with correlation coefficients 0.998 and 0.976 for the silica gel tube and the filter cassette, respectively. With the PDS, the sample concentration obtained was $1134.2 \pm 136.4 \mu\text{g}/\text{m}^3$ (average \pm standard deviation), whereas with the silica gel tube and filter cassette it was 1151.8 ± 137.9 and $1157.3 \pm 135.9 \mu\text{g}/\text{m}^3$, respectively.

Table 1

Comparison of particulate concentrations and mean concentration ratios of PDS and HDS

Species	Average concentration \pm SD, $\mu\text{g}/\text{m}^3$		Average concentration ratio \pm SD PDS/HDS
	PDS	HDS	
F^-	0.82 ± 0.86	0.90 ± 0.97	1.00 ± 0.14
Cl^-	2.23 ± 1.21	2.39 ± 1.34	0.95 ± 0.10
NO_2^-	0.43 ± 0.56	0.43 ± 0.56	0.96 ± 0.09
NO_3^-	2.66 ± 1.40	2.68 ± 1.39	1.00 ± 0.11
SO_4^{2-}	7.17 ± 3.90	7.13 ± 3.20	0.98 ± 0.12
NH_4^+	2.83 ± 2.13	2.87 ± 1.99	0.96 ± 0.10

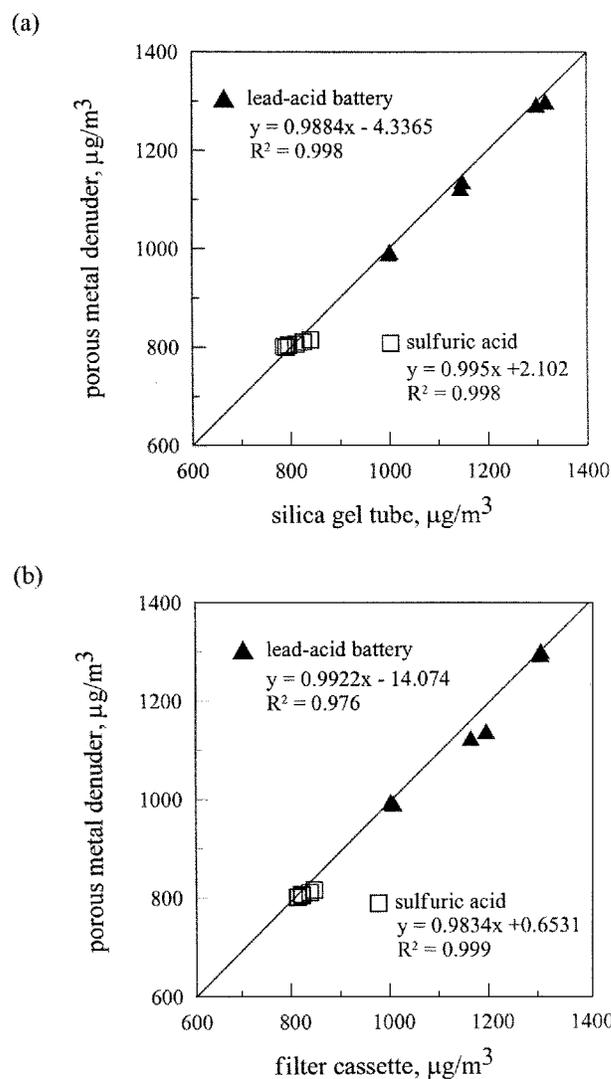


Figure 4. Total sulfuric acid mist concentration measured with the PDS in comparison with the (a) silica gel tube and (b) filter cassette in the lead-acid battery factory and sulfuric acid factory.

One-way ANOVA showed no significant differences ($P > 0.05$) for six replicate samples of the forming area of the lead-acid battery factory, monitored with the PDS, silica gel tube, and filter cassette. The filter of the cassette was extracted with 3 ml

of solution containing 10^{-4} M HClO_4 and 0.04 M KCl for pH meter. Concentrations of H^+ were then calculated by the pH of the sample using a standard calibration curve determined from known concentrations of H_2SO_4 . The nmol/nmol ratios of

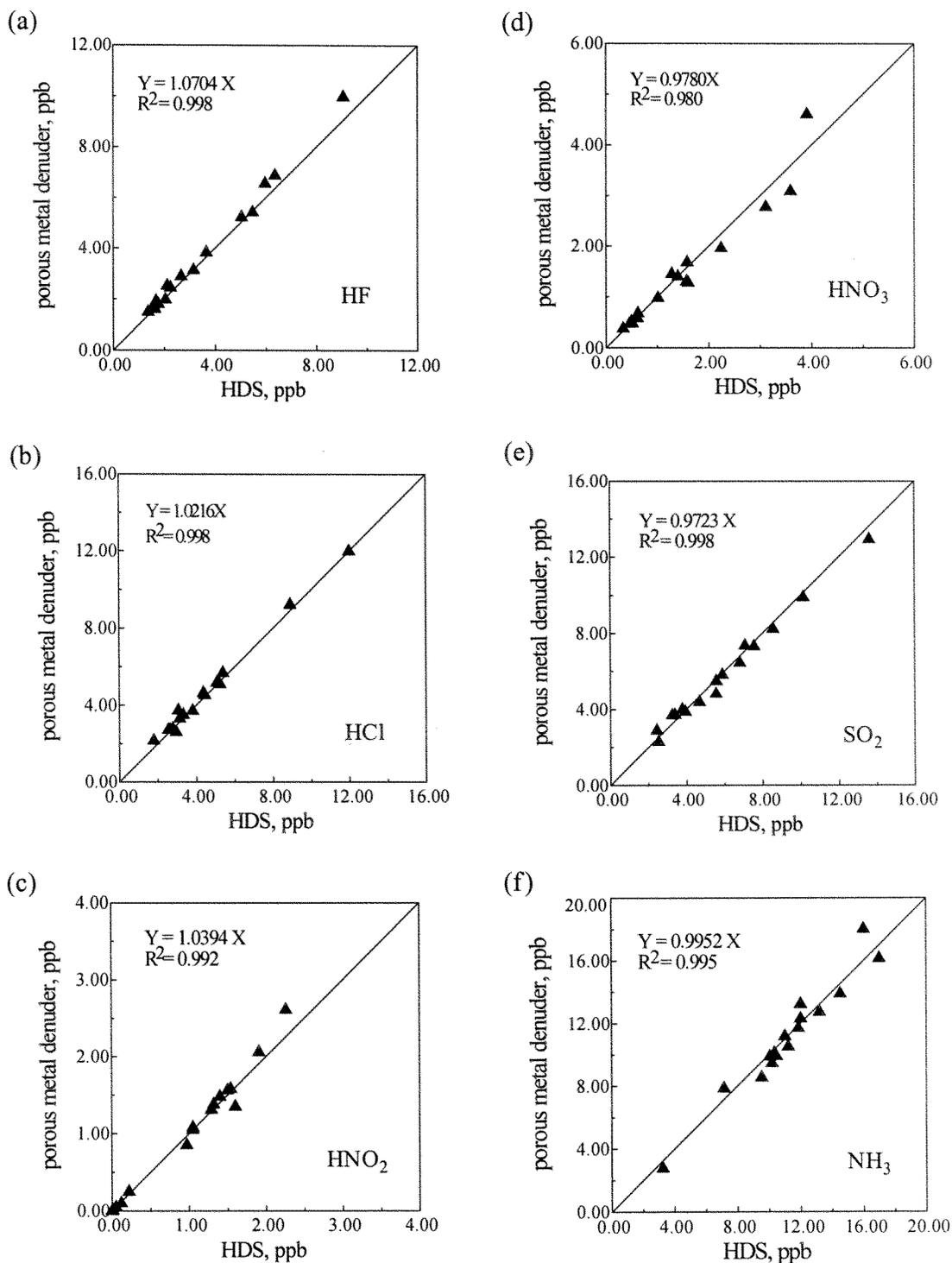


Figure 5. (a) HF, (b) HCl, (c) HNO_2 , (d) HNO_3 , (e) SO_2 , (f) NH_3 concentrations measured with the PDS in comparison with the HDS in the ambient air around the Hsinchu Science Park.

$2[SO_4^{2-}]/[H^+]$ are close to 1, indicating that the field particles mainly were of sulfuric acid mist and the H_2SO_4 existed in the droplet form.

(iii) Sulfuric Acid Factory

For the sulfuric acid factory, Figures 4a and 4b show that the H_2SO_4 acid mist concentrations measured by the PDS were found to be very close to the silica gel tube and filter cassette. The R^2 for the PDS with the silica gel tube and filter cassette was found to be 0.998 and 0.999, respectively. For the PDS, the sample concentration was $806.2 \pm 5.7 \mu g/m^3$ (average \pm standard deviation), whereas for the silica gel tube and filter cassette, the concentrations were $807.9 \pm 21.5 \mu g/m^3$ and $819.1 \pm 13.7 \mu g/m^3$, respectively. One-way ANOVA showed no significant differences ($P > 0.05$) for six replicate samples of the sulfuric acid factory, sampled with the PDS, silica gel tube, and filter cassette.

Gas Concentrations

(i) Ambient Environment

The HF, HCl, HNO_2 , HNO_3 , SO_2 , and NH_3 concentrations measured with the PDS plotted against those measured by means of the HDS in the ambient of the Hsinchu Science Park are shown in Figures 5a–5f, and the data obtained were in excellent correlation, with R^2 of 0.998, 0.998, 0.992, 0.980, 0.998, and 0.995, respectively. One-way ANOVA showed no significant differences ($P > 0.05$) for the six replicate samples taken with the PDS and HDS. The gas concentrations and mean ratios of PDS and HDS are shown in Table 2. The mean values are close to 1, indicating the measurements are in agreement.

(ii) Semiconductor Factory

In the wastewater treatment plant of the semiconductor factory, Hydrogen Fluoride (HF) gas concentrations measured with the PDS, HDS, and silica gel were found to be 0.00357 ± 0.00155 , 0.00352 ± 0.00152 , and 0.00345 ± 0.00145 ppm (average \pm standard deviation), respectively, from an 8-hr sampling, as shown in Figures 6a and 6b. The R^2 for the PDS with the HDS and silica gel tube was 0.995 and 0.998, respectively.

Table 2

Comparison of gas concentrations and mean concentration ratios of PDS and HDS

Species	Average concentration \pm SD, ppb		Average concentration ratio \pm SD PDS/HDS
	PDS	HDS	
HF	3.71 ± 2.44	3.48 ± 2.25	1.07 ± 0.06
HCl	4.60 ± 2.61	4.46 ± 2.62	1.04 ± 0.08
HNO_2	1.11 ± 0.76	1.09 ± 0.70	1.01 ± 0.09
HNO_3	1.48 ± 1.16	1.52 ± 1.15	0.99 ± 0.12
SO_2	5.83 ± 2.81	5.93 ± 3.01	1.00 ± 0.08
NH_3	11.14 ± 3.50	11.24 ± 3.26	0.98 ± 0.07

Figures 6a and 6b also show that HCl concentrations were 0.0067 ± 0.00321 ppm (average \pm standard deviation) for the PDS, 0.00654 ± 0.00301 ppm for the HDS, and 0.00654 ± 0.00293 ppm for the silica gel tube. The R^2 for the PDS with the HDS and silica gel tube was 0.998 and 0.995, respectively. One-way ANOVA showed no significant differences ($P > 0.05$) for six replicate samples of the semiconductor factory, monitored with the PDS, silica gel tube, and HDS.

(iii) Fertilizer Factory

The results of NH_3 concentration sampled by the PDS, compared with the HDS and impinger are shown in Figures 7a and 7b, respectively. The results indicate the NH_3 concentration measured with the PDS was 2.132 ± 0.049 ppm (average \pm standard deviation), whereas with the HDS and impinger it was 2.095 ± 0.054 and 2.155 ± 0.062 ppm, respectively. The R^2 was

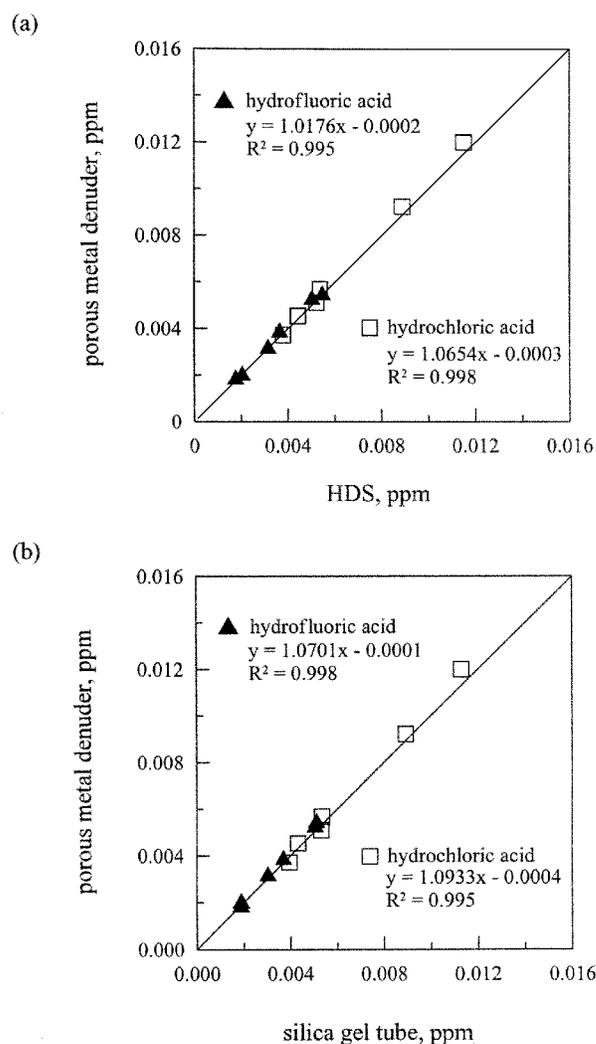


Figure 6. Hydrofluoric and hydrochloric acid gas concentrations measured with the PDS in comparison with the (a) HDS and (b) silica gel tube in the semiconductor factory.

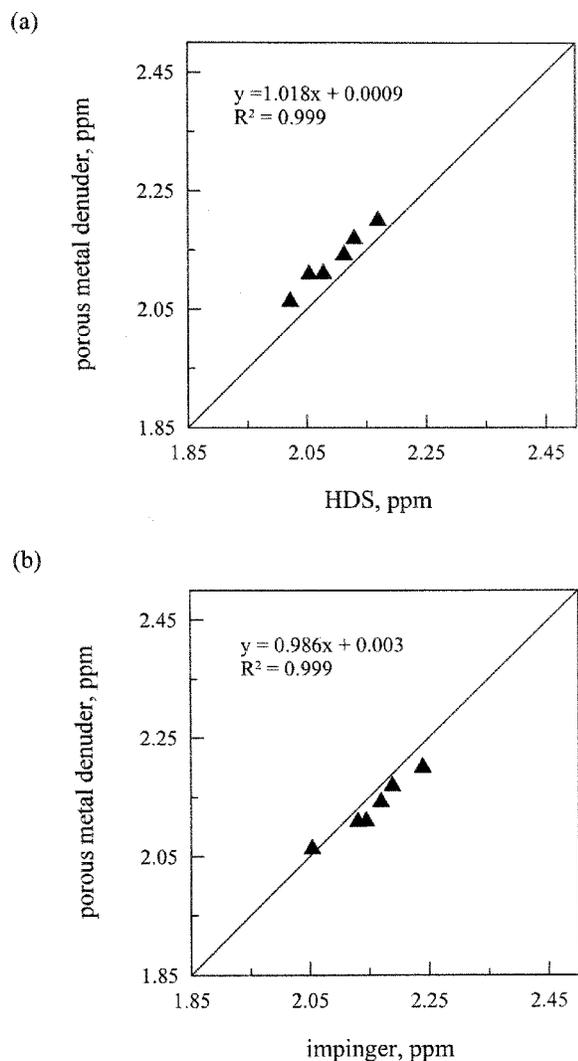


Figure 7. Ammonia gas concentration measured with the PDS in comparison with the (a) HDS and (b) impinger in the fertilizer factory.

calculated for the PDS with the HDS and impinger and found to be 0.999 and 0.999, respectively. One-way ANOVA shows no significant differences ($P > 0.05$) for six replicate samples of the fertilizer factory, sampled with the PDS, HDS, and impinger.

CONCLUSIONS

A PDS sampler has been designed and tested with collocated silica gel tube, impinger, HDS, Marple personal cascade impactor, and filter cassette in an ambient environment around the Hsinchu Science Park and different industrial sites, that is, lead-acid battery factory, sulfuric acid factory, fertilizer factory, and semiconductor factory in Taiwan.

The denuder was compared for the ambient acidic/basic aerosol and gas concentrations, sampled near the Hsinchu Science Park, with those of the HDS and found that the data are highly correlated with correlation coefficient (R^2) greater than 0.980. The results show that total concentrations of sulfuric acid mist measured with the new denuder, silica gel tube, and filter cassette in the lead-acid battery and sulfuric acid factory are very close to each other, with correlation coefficients 0.998 and 0.976 for the lead-acid battery and 0.998 and 0.999 for the sulfuric acid factory, respectively. The size distributions of sulfuric acid particles, sampled with the new denuder and Marple personal cascade impactor in the lead-acid battery factory also are very close.

The concentrations of gases, that is, hydrogen fluoride, hydrogen chloride, and ammonia, determined with the new denuder, HDS, silica gel tube, and impinger near the wastewater treatment tanks of the semiconductor and fertilizer factory also are close to one another and highly correlated.

This study shows that the proposed denuder sampler is applicable for a wide range of sampling concentrations of acidic/basic gases and aerosols. One-way ANOVA shows no significant differences ($P > 0.05$) between the new denuder and other well-established samplers.

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