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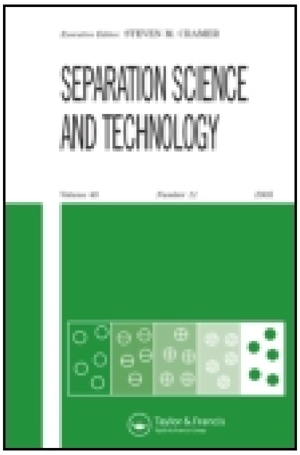

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Adsorption Capacity of a Nylon Filter of Filter Pack System for HCl and HNO_3 Gases

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ABSTRACT

Laboratory and field studies were conducted to evaluate the collection capacity of the nylon filter (Nylasorb membrane, 47 mm diameter, 1.0 μm pore size, Gelman Laboratory, USA) for HCl and HNO_3. In ambient sampling, the field results show that one piece of the nylon filter in the filter pack of an annular denuder system (ADS) or a honeycomb denuder system (HDS) has enough capacity to adsorb HCl and HNO_3 gases evaporated from the particles collected on the Teflon filter. The laboratory

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results show that the individual capacity of the nylon filter to adsorb HCl gas is 8, 160, and 240 μg and that of HNO_3 gas is 160, 1400, 1900 μg at 20% ± 5%, 55% ± 5%, and 80% ± 5% relative humidity, respectively. At low humidity, the efficiency of the nylon filter for HCl gas will be decreased substantially when HNO_3 co-exists. It is decreased to as little as 35–50% when relative humidity is low, at 20% ± 5% for the added amount of HNO_3 is from 40 to 145 μg. A monolayer adsorption theory was successfully employed to estimate the reduction of adsorbing capacity to adsorb HCl gas when HNO_3 gas co-exists. There is no significant effect on the adsorbing efficiency of HNO_3 when HCl co-exists; the collection efficiency is greater than 95% at all relative humidities.

Key Words: Nylon filter; Filter pack system; HCl; HNO_3.

INTRODUCTION

The annular denuder/filter pack system (ADS) and honeycomb denuder system (HDS) are commercial instruments and commonly used in atmospheric research. The filter pack of the ADS consists of a Teflon filter to collect fine particles, two nylon filters to collect HCl and HNO_3, and a glass fiber filter coated with citric acid to collect NH_3 released from the collected particles (Possanzini et al.[1]). Instead of using two nylon filters, the filter pack of HDS contains only one nylon filter following the Teflon filter (Koutrakis et al.,[2] Sioutas et al.[3]). The nylon filter is used to collect HCl and HNO_3 evaporated from particulate NH_4NO_3 and NH_4Cl collected on the Teflon filter (Stelson et al.,[4] Doyle et al.,[5] Stelson and Seinfeld,[6] Pio and Harrison[7]).

$$NH_4Cl(p) \leftrightarrows HCl(g) + NH_3(g) \tag{1}$$

$$NH_4NO_3(p) \leftrightarrows HNO_3(g) + NH_3(g) \tag{2}$$

Appel et al.[8] evaluated the collection efficiency of nylon filters for HNO_3 gas at ambient levels (25 $\mu g/m^3$) in the laboratory. The results showed that the collection efficiency of the nylon filters for HNO_3 was greater than 97% when the collected amount of HNO_3 was less than 250 μg. Karakas et al.[9] used the nylon filter (47 mm in diameter and 0.45 μm pore size, Sartorius) to adsorb HNO_3 gas at room temperature and 50–60% relative humidity (RH) at the sampling flow rate of 15 L/min. The results showed that the nylon filter had collection efficiency greater than 95% and collection capacity of the nylon filter for HNO_3 gas was 3500 μg. The collection efficiency of the nylon filter (47 mm in diameter and 0.45 μm pore size, Sartorius) for HCl gas was studied by William et al.[10] The collection efficiency was generally better than 80% at 18–24°C and 10–80% RH at sampling flow rate of 0.6–6.0 L/min when the total collected HCl was less than 100 μg. However, the collection efficiency was decreased at higher Cl^- loading. For example, the collection efficiency was 64% for Cl^- loading of 1336 μg. When dual nylon filters were used, the collection efficiency was greater than 90% at all humidity (10–80% RH), flow rates (0.6–6.0 L/min) and Cl^- loading levels (<100 μg). Tsai et al.[11] compared the acidic aerosol concentrations measured by the ADS and HDS in Hsinchu, Taiwan. Incomplete adsorption of HCl and HNO_3 gas by the first nylon filter in the filter pack of the ADS was observed. About 14% of the total Cl^- and NO_3^- were measured in the second nylon filter of the filter pack of the ADS.

In this study, ambient sampling was performed to evaluate the capacity of the nylon filters to adsorb HCl and HNO_3 gases using an ADS and a HDS. In the laboratory, an experiment was conducted to determine the collection capacity of nylon filter (Nylasorb membrane, 47 mm diameter, 1.0 μm pore size, Gelman Laboratory, USA) of ADS and HDS to adsorb individual or mixed HCl and HNO_3 gases evaporated from particles on the Teflon filter at different RHs. A possible adsorption of HCl and HNO_3 gases on the Teflon filter of the filter pack was also investigated.

METHODS

Laboratory Analysis

To determine the collection capacity of the nylon filter and Teflon filter to adsorb HCl and HNO_3 gases, test gas was generated in the laboratory as described in Tsai et al.[12] The Experimental setup is shown in Fig. 1. Aerating clean air through a bubbler containing a known concentration of acidic solution generated the desirable test gas concentration. A hot plate was used to heat up the bubbler to facilitate gas generation. The generated concentration of HCl and HNO_3 gases was about 200 and 800 $\mu g/m^3$ (about 140 and 320 ppb), respectively. The relative bias of the gas concentration was below 10%. Heating tape was used to wrap the bubblers and connecting tubes to maintain a constant temperature throughout the sampling system to prevent gas condensation on the wall. The mixed gases were then divided into three branches: one went to the filter pack system to test the capacities of the two-stage nylon filter (Nylasorb membrane, 47 mm diameter, 1.0 μm pore size, Gelman Laboratory, USA) or Teflon filter (Zefluor membrane, 47 mm diameter, 2.0 μm pore size, Gelman Laboratory, USA) at 10 L min^{-1}; the other went to an impinger to examine the concentration of the test gas in 2 L min^{-1}, and the third went to exhaust for temperature and RH measurement. After sampling, the nylon filters were extracted with 10 ml of anion eluent (1.8 mM of Na_2CO_3 and 1.7 mM of $NaHCO_3$) to extract the Cl^- and NO_3^- efficiently. The Teflon filters were

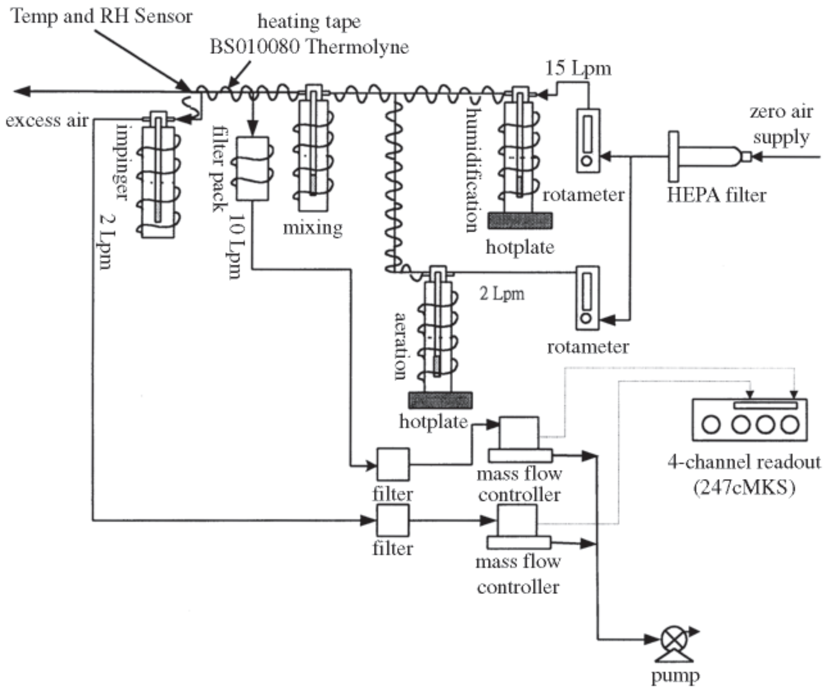

Figure 1. Test setup for measuring the gas capacity of Nylon filter and Teflon filter.

extracted with 10 mL of DI water. The concentrations of the test gas collected on the two-stage filters (first section: C_1, second section: C_2) and impinger was determined by an ion chromatograph (Model 4500i, Dionex Corp., CA). The acid gas collection efficiency of the filter (η, %) was calculated as

$$\eta, \% = \frac{C_1}{C_1 + C_2} \times 100 \tag{3}$$

Ambient Sampling

A HDS and an ADS were collocted at 11 m above ground level on the roof of the Institute of Environmental Engineering, National Chiao Tung University, located in Hsinchu, Taiwan for a sampling period of 24 hr to compare the ambient concentrations of HCl and HNO_3 gases collected on the nylon filters in filter pack system. A total of 10 ambient samples were collected from May 2001 to June 2001 at a sampling flow rate of $10\,L\,/min^{-1}$.

The daily average temperature was from 25°C to 34°C and the daily average relative humidity was from 40% to 82%.

The components of the HDS include an impactor with a cutoff aerodynamic diameter at 2.5 μm, a glass-transition section, two honeycomb denuders, a spacer, and a filter pack (Koutrakis et al.,[2] Sioutas et al.,[3]). The first-stage honeycombs of the HDS were coated using 1% (w/v) sodium carbonate, 1% (w/v) glycerol in 1 : 1 (v/v) methanol/water solution for acid gases. For ammonia gas, 2% (w/v) citric acid in ethanol was used in the second-stage honeycombs. A three-stage filter pack was placed downstream of the denuders. The filter pack consists of a Teflon filter (Gelman Science, 2 μm pore size) to collect fine particles, a nylon filter (Gelman Science, 1- μm 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., CA).

Details of the ADS sampler were described by the US Environmental Protection Agency (EPA).[13] The ADS consists of a cyclone (model URG-2000-30EN) with the cutoff aerodynamic diameter at 2.5 μm ($10\,\mathrm{L\,min^{-1}}$ flow rate) to remove fine particles and four denuders to collect acidic and basic gas species. As in the case of HDS, following the fourth denuders is a PTFE Teflon filter pack, containing a Teflon filter upstream of two nylon filters (to collect HNO_3 and HCl) and a citric acid coated glass fiber filter (to collect NH_3).

In this study, the filter packs of both the HDS and ADS were used to examine the collection capacity of the nylon filter to adsorb HNO_3 and HCl gases. QA/QC procedure in this study included establishment of calibration curve using standard solutions and 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 recovery efficiencies were estimated using spike samples with the sampling concentrations based on sampling volume. The results of MDL, blank analysis, and recovery efficiency of filters were shown in Table 1. The results of precision analysis showed that the relative bias of detected values was below 4%.

RESULTS AND DISCUSSION

Laboratory Results of Adsorbing Capacity of Nylon Filter for Individual Gas

The gas collection efficiencies of nylon filter were first tested in our laboratory. The individual collection capacity of nylon filter is defined as the

Table 1. Method detection limit, blank analysis, and recovery efficiency of filters.

			Blank analysis		Recovery efficiency ± SD[b](%)	
Species	n[c]	MDL[a] (ppbv)	Teflon filter	Nylon filter	Teflon filter	Nylon filter
Cl^-	7	0.021	ND[d]	ND	107.2 ± 2.6	95.4 ± 1.5
NO_3^-	7	0.018	ND	ND	100.1 ± 4.5	98.5 ± 2.5

[a] MDL, Method detection limit based on $10\,\mathrm{L\,min^{-1}}$ and 24 hr.
[b] SD, Standard deviation.
[c] n, Number of samples.
[d] ND, Detected value <MDL.

maximum amount of HCl and HNO_3 gases that can be collected on the filter with a collection efficiency greater than 95% (breakthrough line). Figure 2 shows that the individual collection efficiencies of the nylon filter for HCl gas when the temperature is 30°C ± 2°C and the RH is 20% ± 5%, 55% ± 5%, and 80% ± 5%, respectively. For RH of 80% ± 5%, the gas collection efficiency of the nylon filters are about 100% for the initial gas loading smaller than 200 μg. The collection efficiency decreased to 85.4% for gas loading increases to 332 μg. The capacity of nylon filter to adsorb HCl gas was found to be 240 μg. Similarly, the collection efficiency decreased to 80.0% as the gas loading increases to 233 μg and the capacity was found to be 160 μg for a RH of 55% ± 5%. For a RH of 20% ± 5%, the collection efficiency decreased drastically, from 100% to 80.4%, when the gas loading increased from 6 to 10 μg and the capacity was found to be only 8 μg.

A similar conclusion can be reached in the case of adsorbing HNO_3 gas by the nylon filter. Figure 3 shows that the individual collection efficiencies of the nylon filter for HNO_3 gas at 30°C ± 2°C and 20% ± 5%, 55% ± 5%, and 80% ± 5% RH, respectively. It is seen that the collection efficiencies remain constant and then drop below 95%, except for a RH of 20% ± 5%. The capacity of nylon filter to adsorb HNO_3 gas is found to be 160, 1400, and 1900 μg at 20% ± 5%, 55% ± 5%, and 80% ± 5% RH, respectively. The results show that the individual capacity of a nylon filter to adsorb HNO_3 gas was higher than that of HCl gas at different relative humidities. We infer that the nylon filter is more selective to adsorb HNO_3 than HCl. In addition, the individual capacities of nylon filter to adsorb HNO_3 and HCl gas increase with an increasing RH. It may be because that a hydrogen bond is formed by the reaction of water vapor and the function group (–CONH) of the nylon filter. The water molecules laden the surface of the filters and become better

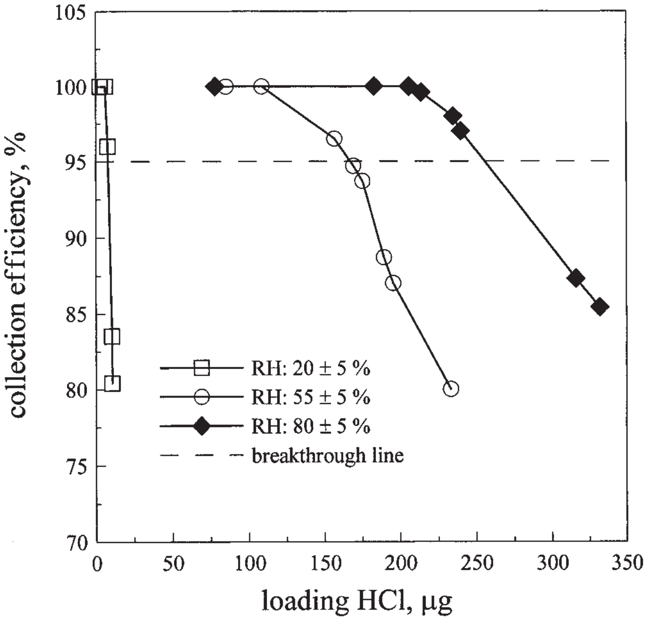

Figure 2. Individual collection efficiency of the Nylon filter for different loading HCl gases.

adsorbing sites for HNO_3 and HCl gas. The following reactions of gases with water vapor may take place on the filter surface.

$$HCl_{(g)} + H_2O_{(s)} \rightarrow Cl^- + H_3O^+ \quad (4)$$

$$HNO_{3(g)} + H_2O_{(s)} \rightarrow NO_3{}^- + H_3O^+ \quad (5)$$

Therefore, a high relative humidity causes an increase in the adsorption capacity for HNO_3 and HCl gas of the nylon filter.

Laboratory Results of Adsorbing Capacity of Nylon Filter for Mixed Gas

The possible interference of co-existing gas on the collection efficiency of a nylon filter was determined. Figure 4 shows that the collection efficiency for HCl gas of the nylon filter when HNO_3 is present at three relative humidities of 20% ± 5% [Fig. 4(a)], 55% ± 5% [Fig. 4(b)], and 80% ± 5% [Fig. 4(c)].

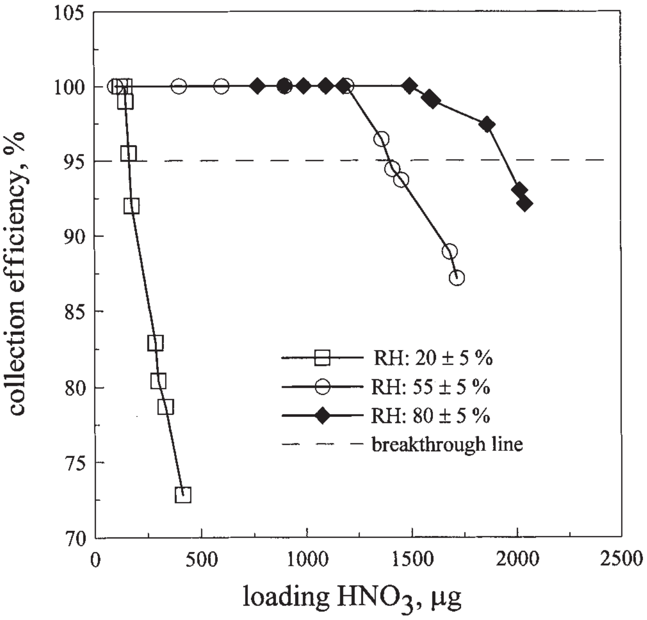

Figure 3. Individual collection efficiency of the Nylon filter for different loading HNO_3 gases.

When the added amount of HNO_3 was from 40 to 145 μg, the collection efficiency for HCl gas of the nylon filter was decreased substantially to 35–50% at the testing RH of 20% ± 5%, as shown in Fig. 4(a), while the collection efficiency was greater than 95% for HCl gas only. For a higher testing RH, such as 55% ± 5% [Fig. 4(b)] and 80% ± 5% [Fig. 4(c)], the interference by co-existing HNO_3 gas on the collection efficiency of HCl gas for the nylon filter was less severe. The collection efficiency was decreased to 65–97%, when the added amount of HNO_3 was from 203 to 1310 μg for 55% ± 5% RH; and it was 67–100%, when the added amount of HNO_3 was from 97 to 2150 μg for 80% ± 5% RH.

In comparison, there is no significant effect for the nylon filter to adsorb HNO_3 gas when HCl co-exists. The collection efficiency was greater than 95%, as shown in Fig. 5(a) (20% ± 5%), Fig. 5(b) (55% ± 5%) and Fig. 5(c) (80% ± 5%), when the added amount of HCl was from 1 to 4, 18 to 127, and 44 to 195 μg for 20% ± 5%, 55% ± 5%, and 80% ± 5% RH, respectively. It is seen that the influence of the RH on the collection efficiency for the nylon filter to adsorb HNO_3 gas when HCl co-exists can be also neglected.

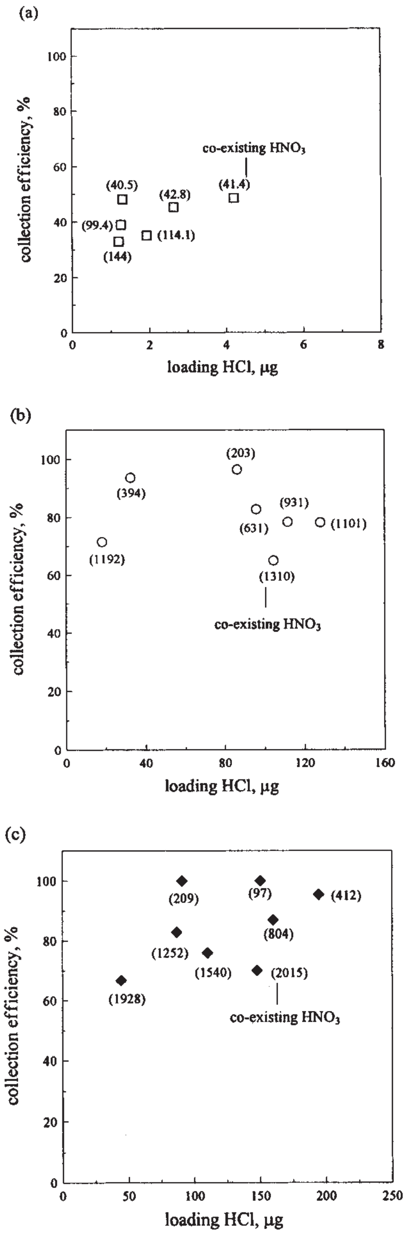

Figure 4. Collection efficiency for HCl gas of the nylon filter when HNO$_3$ is present (HNO$_3$ is added) (a) 20% ± 5%, (b) 55% ± 5%, and (c) 80% ± 5%.

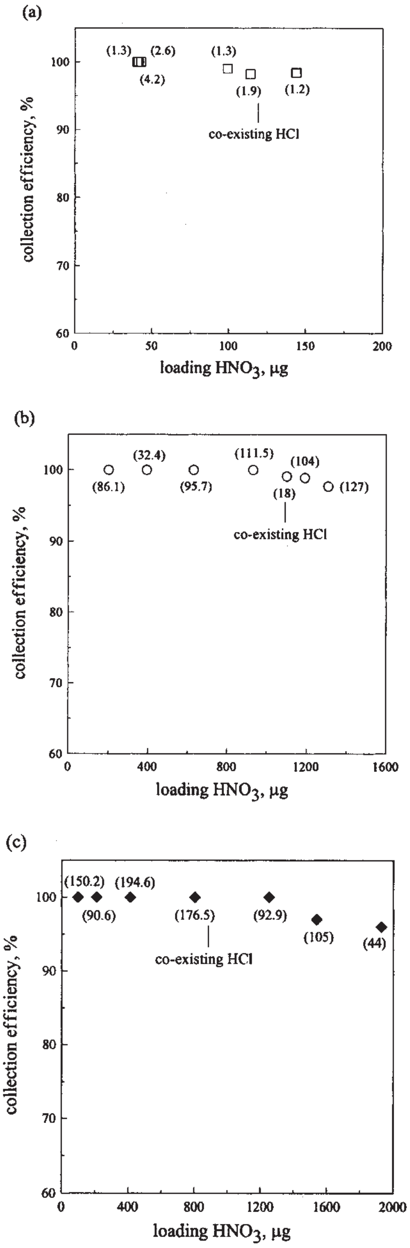

Figure 5. Collection efficiency for HNO_3 gas of the Nylon filter when HCl is present (a) 20% ± 5%, (b) 55% ± 5%, and (c) 80% ± 5%.

To further explain the interference by co-existing HNO_3 gas on the collection efficiency of HCl gas for a nylon filter, this study employed a monolayer adsorption theory to estimate the capacity of the nylon filter to adsorb HCl gas when HNO_3 gas coexists based on the following assumptions:

1. The total adsorption site of the nylon filter is equal to the individual collection capacity of the nylon filter to adsorb HNO_3 gases.
2. The nylon filter is more selective to adsorb HNO_3 than HCl.
3. Before the filter is saturated, the adsorption of HCl and HNO_3 gases by the nylon filter is a monolayer adsorption process.
4. When HCl and HNO_3 gases co-exist, the interference of co-existing gas on the collection efficiency of the nylon filter is the same.

A form of this theoretical equation can be expressed mathematically as follows:

$$m'_{HCl} = \left(1 - \frac{w_{HNO_3}}{m_{HNO_3}}\right) \times m_{HCl} \qquad (6)$$

where m'_{HCl} (μg) is the collection capacity of the nylon filter to adsorb HCl gas when HNO_3 coexists, m_{HCl} (μg) is the individual collection capacity of nylon filter to adsorb HCl gas, w_{HNO_3} (μg) is the added amount of HNO_3 gas, and m_{HNO_3} (μg) is the individual collection capacity of the nylon filter to adsorb HNO_3 gas.

Figure 6 shows the relationship between the collection efficiency of the nylon filter to adsorb HCl gas when HNO_3 co-exists (η'_{HCl}, %) and the interference by co-existing HNO_3 gas on the collection capacity of HCl gas ($m_{HCl} - m'_{HCl}$, μg) at different RHs. It is seen that the collection efficiency for HCl gas when HNO_3 co-exists decreases with increasing interference by co-existing HNO_3 gas on the collection capacity of HCl gas. When the RH is increased, the influence of interference by HNO_3 gas on the collection efficiency of HCl gas of the nylon filter is not apparent. It is due to the fact that the capacities of the nylon filter to adsorb HNO_3 and HCl gas increase with an increased RH.

Laboratory Results of Teflon Filter to Adsorb HCl and HNO_3

It is interesting to know whether the prefilter (Teflon filter) of the filter pack system has a possible interference to adsorb HCl and HNO_3 gases on particulate concentration measurement. Table 2 shows that the gas adsorption amount (μg) of the Teflon filter for HCl and HNO_3 gas when temperature was

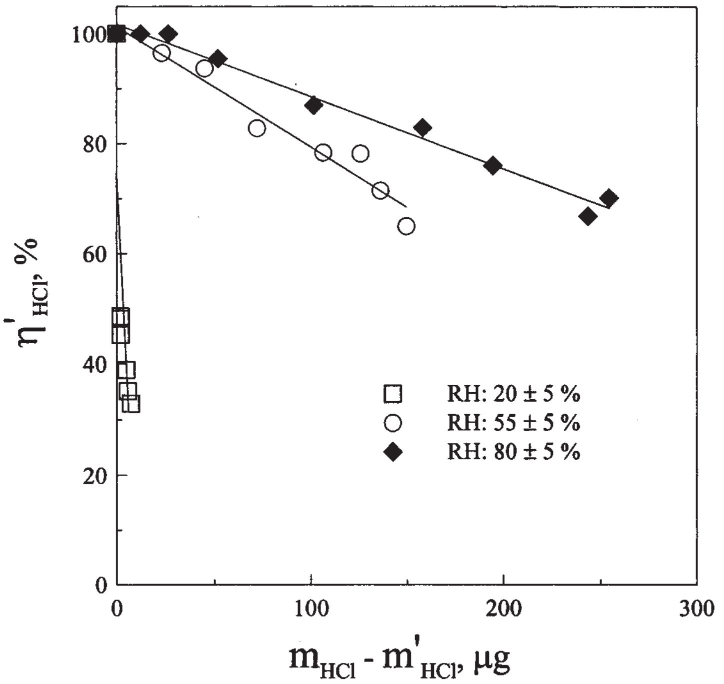

Figure 6. Relationship between the collection efficiency of the Nylon filter to adsorb HCl gas when HNO_3 co-exists (η'_{HCl}, %) and the interference by co-existing HNO_3 gas on the collection capacity of HCl gas ($m_{HCl} - m'_{HCl}$, µg) at different RHs.

30°C ± 2°C and RH was 20% ± 5% and 80% ± 5% at different sampling periods. When the RH was low, such as 20% ± 5%, the gas amounts collected by the Teflon filter were less than MDL for 1, 2, and 3 hr sampling periods. For high RH, such as 80% ± 5%, the adsorption amounts of HCl gas by the Teflon filter were still less than MDL, but those of HNO_3 gas were 2.8 ± 0.5, 4.1 ± 0.5, and 4.4 ± 0.3 µg (about 1.87 ± 0.33, 1.37 ± 0.17, and 0.98 ± 0.07 ppb) for 1, 2, and 3 hr sampling periods. It indicates that the water molecule, which is retained by the surface of the Teflon filter in high RH, will collect HNO_3 gas. Also, the adsorption amounts of HNO_3 for 2 and 3 hr sampling periods are close to each other due to the fact that the retained water molecule approaches saturation to adsorb HNO_3 gas. In addition, we calculated the percentage of total HNO_3 collected on the Teflon filter and found those were 6.2%, 3.4%, and 1.8% for 1, 2, and 3 hr sampling periods at 80% ± 5% RH. It indicates that the interference to adsorb HNO_3 by the Teflon filter can be neglected for atmosphere sampling in a general period of 24 hr.

Table 2. Adsorption mass of Teflon filter for HCl and HNO_3 gas.

Time (hr)	n^c	RH:[a] 20% ± 5% [mean ± SD[b] (μg)]		RH: 80% ± 5% [mean ± SD (μg)]	
		HCl	HNO_3	HCl	HNO_3
1	3	ND[d]	ND	ND	2.8 ± 0.5
2	3	ND	ND	ND	4.1 ± 0.5
3	3	ND	ND	ND	4.4 ± 0.3

[a] RH; Relative humidity.
[b] SD, Standard deviation.
[c] *n*, Number of test samples.
[d] ND, Detected value <MDL.

Field Results

It is important to know whether the nylon filters of an ADS and a HDS have sufficient capacity to adsorb HCl and HNO_3 gases for ambient sampling. Two nylon filters were used in the filter pack of ADS and HDS to absorb HCl and HNO_3 gases. The results of the field experiments are given in Table 3. The concentrations of HCl and HNO_3 gases on the first nylon filter of the ADS were 0.15 ± 0.16 and 0.14 ± 0.12 ppb. Those on the first nylon filter of the HDS were 0.20 ± 0.22 ppb and 0.14 ± 0.10 ppb, respectively. Both of the gas concentrations on the second nylon filters of the ADS and HDS were less than method detection limiting (0.021 and 0.018 ppb for HCl and HNO_3). The concentrations of Cl^- and NO_3^- particulate on the Teflon filter of the ADS were 0.38 ± 0.35 and 0.73 ± 0.44 $\mu g/m^3$, and those of the HDS were 0.47 ± 0.45 and 0.69 ± 0.49 $\mu g/m^3$, respectively. Because of the low NO_3^-

Table 3. Concentrations of HCl and HNO_3 in Nylon filters of ADS and HDS in field test.

Species	*n*	ADS[a] [average ± SD (ppb)]		HDS[b] [average ± SD (ppb)]	
		First nylon filter	Second nylon filter	First nylon filter	Second nylon filter
HCl	10	0.15 ± 0.16	ND	0.20 ± 0.22	ND
HNO_3	10	0.14 ± 0.12	ND	0.14 ± 0.10	ND

[a] ADS, annular denuder system.
[b] HDS, honeycomb denuder system.

and Cl^- concentrations in particulate, one piece of the nylon filter in the filter pack of the ADS and HDS has enough capacity to adsorb HCl and HNO_3 gases for ambient sampling.

CONCLUSIONS

This study evaluated whether the collection capacity of a nylon filter of an annular denuder system is enough to adsorb HCl and HNO_3 gases evaporated from particles on a Teflon filter. In the collection capacity test, the individual capacity of nylon filter to adsorb HNO_3 gas was found to be higher than that of HCl gas at different humidities. In the interference test, experiment data show that the collection efficiency of the nylon filter for HCl gas was decreased substantially when HNO_3 was present. A monolayer adsorption theory was employed to estimate the theoretical capacity of the nylon filter to adsorb HCl gas when HNO_3 gas coexists. The results showed that the collection efficiency of the nylon filter for HCl gas decreases with increasing interference of HNO_3 gas. However, there is no significant effect of nylon filter to adsorb HNO_3 when HCl co-exists, the collection efficiency of the nylon filter for HNO_3 gas is greater than 95%.

The possible collection of the Teflon filter for HCl and HNO_3 gases was also investigated. The results show that HCl and HNO_3 collected on the Teflon filter can be neglected in a general period of 24 hr. In ambient sampling, the results show that one piece of the nylon filter in the filter packs of the ADS and HDS has enough capacity to adsorb HCl and HNO_3 gases.

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