Journal of the Air & Waste Management Association

Performance of the Annular Denuder System with Different Arrangements for HNO$_3$ and HNO$_2$ Measurements in Taiwan

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ABSTRACT

Experiments on different annular denuder system (ADS) arrangements for sampling nitrous acid (HNO_2) and nitric acid (HNO_3) gases were conducted in this study to evaluate their sampling artifacts. The evaluation basis is the one that employed one sodium chloride denuder for sampling HNO_3 gas and two sodium carbonate (Na_2CO_3) denuders for sampling HNO_2 gas, which is a commonly employed ADS arrangement in many field applications in the United States. A field study was conducted in Hsinchu, Taiwan, and the results indicated that this ADS arrangement may yield over 80% relative errors for HNO_3 gas. It also showed that the relative errors for HNO_2 gas can be less than 10% as sampled with only one Na_2CO_3 denuder. This is attributed to the fact that the ambient HNO_3 concentration measured in this study was relatively low while the HNO_2 concentration was high, as compared to typical concentrations of these two gases measured in the United States.

The sampling error of HNO_3 gas may be due to high concentrations of N-containing interfering species present in Taiwan's atmosphere. Because the relative sampling errors of HNO_3 and HNO_2 gases depend mainly on their concentrations in the atmosphere as well as concentrations caused by interfering species, the risk for high error while measuring low HNO_2 concentrations by only one Na_2CO_3 denuder is also possible. As a result, it is suggested that pretests are necessary to evaluate possible sources and degrees of sampling errors before field

sampling of HNO_2 and HNO_3 gases. The sampling errors of these two gases can, therefore, be minimized with a better arrangement of the ADS.

INTRODUCTION

The annular denuder system (ADS) is a widely employed sampling instrument for collecting reactive gases. The most widely employed ADS arrangement[1] is shown in Figure 1. It uses four annular denuders in series followed by a filter pack. The first denuder is coated with a sodium chloride (NaCl) aqueous solution for sampling nitric acid (HNO_3) gas. With the assumption of complete absorption of HNO_3 gas with one denuder and neglible interference, there is only one denuder used for HNO_3 gas sampling in Figure 1. The nitrous acid (HNO_2) gas is then collected by the second and third denuders coated with a sodium carbonate (Na_2CO_3) aqueous solution. Because significant interference is expected for HNO_2 gas sampling, any nitrogen dioxide (NO_2^-) or nitrate (NO_3^-) found in the third Na_2CO_3 denuder is used for the correction of HNO_2 concentration on the second denuder.

Laboratory studies[2,3] have demonstrated the high performance of an ADS for HNO_3 and HNO_2 gas sampling. They found that the ADS achieved very high HNO_3 and HNO_2 collection efficiencies. But they have also shown that the HNO_3 gas was subjected to a high potential of sampling biases in field studies.[4,5] Positive sampling artifacts may arise due to depositions of N-containing gases or particles, as well as the evaporation of HNO_3 from ammonium nitrate (NH_4NO_3) particles.[6-10]

Sampling errors in field measurements of HNO_3 and HNO_2 depend on the relative magnitude of their concentrations. For example, typical concentrations of HNO_3 gas in the United States are usually higher than or in the same order of magnitude as HNO_2 gas concentrations.[2,4,11-13] But in other areas such as Taiwan,[14] Korea,[15] and Portugal,[16] HNO_3 concentrations are detected to be very low, and they are usually lower than concentrations of HNO_2 gas. As a result, interference for the HNO_3 gas may be higher than that for HNO_2 gas measurements. Therefore, it may not be appropriate using the widely employed ADS arrangement

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IMPLICATIONS

The ADS has been commonly used for sampling acidic and basic gases. However, it has often been employed without a pre-evaluation of the sampling biases. If not properly arranged, the ADS may produce sizable errors due to complication of the atmospheres. This paper provides a detailed evaluation of the ADS arrangement for sampling HNO_3 and HNO_2 gases. It is shown that the proper ADS arrangement depends on the concentration of the target pollutant as well as concentrations caused by interfering species.

(as shown in Figure 1), which uses one NaCl denuder for collecting HNO_3 gas and two Na_2CO_3 denuders for collecting HNO_2 gas.

This is especially true when the atmosphere contains high concentrations of NH_4NO_3 particles, oxides of nitrogen (NO_x), peroxyacetyl nitrate (PAN), or other N-containing species. As demonstrated in the literature,[4,9,10] NH_4NO_3 may evaporate in the denuder, which leads to an excessive absorption of the HNO_3 gas. NO_x and PAN in the atmosphere could also be partially collected and converted to HNO_3.[6-8] Unfortunately, the ADS arrangement[1] has often been used without a pre-evaluation of its suitability.[13-15]

This paper attempts to experimentally evaluate the performance of different ADS arrangements for sampling HNO_3 and HNO_2 gases in Taiwan's atmosphere where the HNO_3 gas concentration is very low but the HNO_2 gas concentration is high. The comparison basis is the ADS arrangement shown in Figure 1. Sampling errors of the ADS arrangements are determined to demonstrate their applicability for measuring concentrations of these two gases in the atmosphere.

EXPERIMENTAL

Ambient 24-hr average samples were collected from August 1996 to May 1997. Measurements were made on the roof of the Institute of Environmental Engineering, National Chiao-Tung University (about 10 m above ground level), Hsinchu, Taiwan. A semiconductor industrial area is located about 1 km away from the sampling site. The semiconductor industry uses many acid and basic gases in its manufacturing processes. Due to low HNO_3 concentrations observed during the sampling period, several indoor measurements were also conducted with an artificial HNO_3 source. Twelve outdoor samples for HNO_2 gas and 29 outdoor and 7 indoor samples for HNO_3 gas were collected in this study.

The assembly of the ADS train used in this study was similar to the arrangement[1] shown in Figure 1, except that when sampling HNO_3 gas, three or four denuders were in a series and all were coated with 10 mL of 0.1% (w/v) NaCl in 1:9 methanol/water solution. And when sampling HNO_2 gas, the first denuder was coated with NaCl/methanol/water solution (to remove HNO_3 gas first) and the following three denuders were coated with 10 mL of 1% (w/v) Na_2CO_3, 1% (w/v) glycerol in 1:1 methanol/water solution. Other sampling conditions were in accordance with the U.S. Environmental Protection Agency's (EPA) manual (Compendium Chapter IP-9).[1]

The ADS train used in this study consisted of a Teflon-coated aluminum cyclone ($d_{50} < 2.5$ μm; University Research Glassware, [URG]), followed by three or four annular denuders (URG-2000-30B) and a filter pack. A
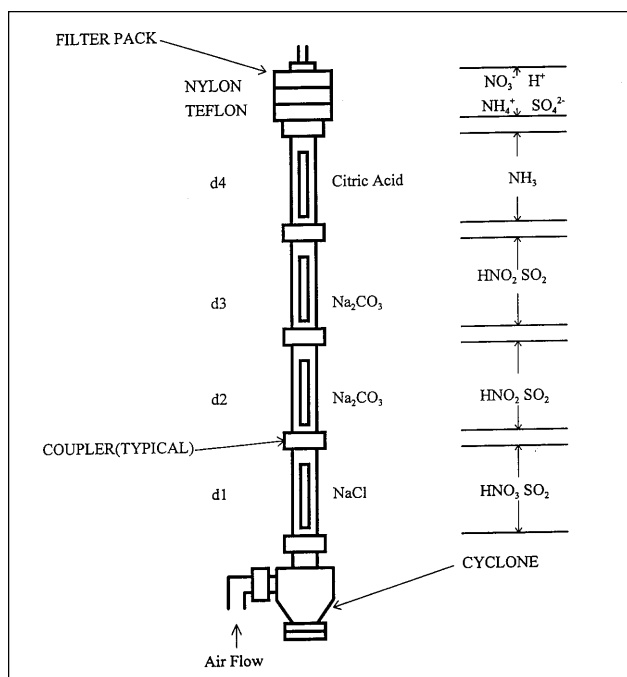

Figure 1. Schematic diagram of an ADS arrangement for evaluation basis (after EPA[1]).

housing box was used to protect the annular denuders and filter pack. The flow rate of the ADS was kept at 10.0 ± 0.2 L/min. After sampling, the denuders were extracted with 10 mL of ultrapure water (Millipore, Milli-Q water system). The extracts were stored at 4 °C and analyzed as NO_2^- and NO_3^- ion concentrations by ion chromatography (Dionex model 4500i) within 1 week. The resolution of the ion chromatography measurement was set at 10 μs/cm for sample concentration less than 1 mg/L or 30 μs/cm for that larger than 1 mg/L. The calibration curve was produced using five concentrations ranging from 0.2 to 5 mg/L. The R^2 values were over 0.995.

A quality assurance and quality control (QA/QC) program was performed during sampling and analysis to ensure that the measured artifact source was not from failures of the QA/QC program. Leak tests were performed for all joints before sampling to assure that no breakage occurred. Replicate results (parallel measurements of three identical ADS systems) of field samples showed that the first denuders (for both HNO_2 and HNO_3) could be within 15% of relative errors, and the downstream denuders were within 20% of relative errors. Spike analyses were also performed for the experiments. Additives at the concentration of 1 mg/L were added to the HNO_2 and HNO_3 samples, and the recovery percentages of spike analysis were within $100 \pm 15\%$ for the first denuders and within $100 \pm 25\%$ for the downstream denuders. The instrument detection limits (IDLs) were taken as three standard deviations of seven blank analytic samples, and the limits of quantification (LOQ) were taken as 10 standard deviations of 7 blank analytic samples.[1]

The method of detection limits (MDLs) were defined as the limits of quantification of the method, and they were the sum of LOQ and field blanks. The definitions of the above QA/QC parameters were in accord with those listed in the EPA manual for ADS sampling.[1]

Table 1 shows the IDLs, LOQ, and MDLs for HNO_3 and HNO_2 gas sampling in this study. One field blank sample was taken for every field sampling, and the concentrations of field blanks could not be detected by the ion chromatography. Because the field blank concentrations were well below the IDLs for both HNO_3 and HNO_2 species, values of LOQ and MDLs were the same. The values of LOQ and MDLs were 0.047 μg/m³ and 0.06 μg/m³, respectively, for HNO_3 and HNO_2 gases at a flow rate of 10 L/min for 24 hr, much lower than the values listed in Chapter IP-9 of the EPA manual[1] under the same sampling conditions.

METHODS FOR ESTIMATION OF ADS SAMPLING EFFICIENCY

Sampling errors for HNO_3 and HNO_2 measurements by an ADS are induced by the following two sources:

(1) Incomplete absorption of HNO_3 and HNO_2 gases at the denuder walls. This will result in less than 100% sampling efficiency. In addition, negative error may arise from adsorption of gases inside the cyclone.

(2) Perfect absorption of HNO_3 and HNO_2 gases, but with artifact sources of NO_3^- and NO_2^- due to N-containing gases or particles. These artifact sources will produce over 100% sampling efficiency. The sources of sampling errors could be from the deposition and evaporation of nitrate particles, or the absorption of NO_x, PAN, and other gases.[6-10,17,18]

Assuming that the concentrations of the target pollutant and the interfering species in the atmosphere are C_{tar} and C_{int}, respectively, and their absorption efficiencies in each denuder are η_{tar} and η_{int}, respectively, concentrations collected by each denuder are

first denuder $= C_{tar}\eta_{tar} + C_{int}\eta_{int}$ (1)

second denuder $= C_{tar}\eta_{tar}(1-\eta_{tar}) + C_{int}\eta_{int}(1-\eta_{int})$ (2)

third denuder $= C_{tar}\eta_{tar}(1-\eta_{tar})^2 + C_{int}\eta_{int}(1-\eta_{int})^2$ (3)

fourth denuder $= C_{tar}\eta_{tar}(1-\eta_{tar})^3 + C_{int}\eta_{int}(1-\eta_{int})^3$ (4)

The absorption efficiencies of HNO_3 and HNO_2 gases by NaCl solution and Na_2CO_3 solution, respectively, have been previously shown by the EPA manual[1] to approach unity. This result has also been demonstrated by many researchers.[2,3] Therefore, it is possible that the first denuder will almost absorb all target pollutant. Or, if the unabsorbed pollutant is not completely absorbed by the

Table 1. IDLs, LOQ, and MDLs of HNO_3 and HNO_2 species.

Species	IDLs	LOQ (μg/m³)[a]	Field Blank	MDLs
HNO_3	0.014	0.047	<IDL	0.047
HNO_2	0.018	0.060	<IDL	0.060

[a]Concentrations are in units of μg/m³ as sampled at a flow rate of 10 L/min for 24 hr.

first denuder it should be collected by the second denuder, and no target pollutant should be found in the third denuder.

Similarly, the absorption efficiency for the interferences (η_{int}) in each denuder should approach zero. But if the concentration of interfering species is high enough and the concentration of target pollutant is relatively low, it is still possible to produce a significant sampling error to the target pollutant. As a result, the concentration of interfering species collected by each denuder should be approximately the same as $C_{int}\eta_{int}$. Therefore, two approaches were made for estimating the sampling efficiency of HNO_3 and HNO_2 gases.

The first approach made was incomplete absorption of target pollutant by the first denuder. This approach assumes that the absorption of the target pollutant by the first denuder is not complete, but that the remaining target concentration will be completely collected by the second denuder. Therefore, the third denuder collects the interfering species only. As a result, eqs 2–4 can be further simplified to

second denuder $= C_{tar}(1-\eta_{tar})+ C_{int}\eta_{int}$ (5)

third denuder $= C_{int}\eta_{int}$ (6)

fourth denuder $= C_{int}\eta_{int}$ (7)

It is reasonable that when using three denuders in series, the concentration of target pollutant for the case of incomplete absorption (denoted as "$C_{tar,inc}$") can be theoretically calculated by subtracting double the concentration collected by the third denuder from the sum of those collected by the first and second denuders.

The second approach made was perfect absorption of target pollutant by the first denuder. This approach assumes that the target pollutant is completely collected by the first denuder; therefore, downstream denuders collect interferences only. As a result, eq 5 turns out to be the same as eqs 6 and 7. The target concentration for the case of perfect absorption (denoted as "$C_{tar,perf}$") is thus theoretically calculated as the difference between the first denuder and the average of downstream denuders. This approach has been adopted by EPA[1] to sample the HNO_2 gas. It employs two denuders for sampling HNO_2 gas, and the ambient HNO_2 concentration is obtained by subtracting concentration collected by the second denuder from

that collected by the first denuder. But for sampling HNO_3 gas, it uses only one denuder for sampling HNO_3 gas. That is, the assumption of perfect absorption of HNO_3 gas by the first denuder is made using the EPA method, and the interference for sampling HNO_3 gas is assumed negligible.

RESULTS AND DISCUSSION

Summary of HNO_3 and HNO_2 Measurements

HNO_3 Measurements. Table 2 shows the mean, standard deviation, range, and number of samples for HNO_3 measurements. The first denuder data represent the HNO_3 concentrations as measured by the ADS arrangement shown in Figure 1. The second, third, and fourth denuder measurements are sampling biases that have occurred due to either the incomplete absorption of HNO_3 or artifact sources from N-containing gases and particles. The outdoor HNO_3 concentrations collected at the first denuder range from 0.18 to 0.55 $\mu g/m^3$. As compared to measured data in the United States, the HNO_3 concentrations measured in this study are in the low range. The HNO_3 concentrations in the United States[11,13] could be higher than 30.0 $\mu g/m^3$, or as low as 0.00 $\mu g/m^3$.

In order to obtain higher HNO_3 concentrations, indoor sampling was also performed with an artificial HNO_3 source produced by opening a HNO_3 solution bottle near the ADS. The results of indoor sampling are also shown in Table 2. The indoor concentrations measured by the first denuder range from 1.10 to 9.64 $\mu g/m^3$. The mean concentrations of the second, third, and fourth denuders are 0.16, 0.14, and 0.13 $\mu g/m^3$, respectively, for indoor measurements; and 0.10, 0.08, and 0.07 $\mu g/m^3$,

respectively, for outdoor measurements. The HNO_3 concentrations collected from downstream denuders for indoor sampling are only slightly higher than those for outdoor sampling.

HNO_2 Measurements. Table 3 shows the mean, standard deviation, range, and number of samples for HNO_2 measurements. The pre-denuder data are for the collection of HNO_3 gas ahead of HNO_2 gas to avoid mixed collection of these two gas species. The difference between the first and second denuders represents the ambient HNO_2 concentration as measured by the ADS arrangement shown in Figure 1. It is seen that the denuder difference method gives a mean HNO_2 concentration of 4.01 $\mu g/m^3$. This is close to the average HNO_2 concentration collected by the first denuder, 4.32 $\mu g/m^3$, due to relatively low concentrations collected by the second denuders. As compared to measured data in the literature, the HNO_2 concentrations measured in this study are in the high range of typical HNO_2 concentrations of 0.09–4.6 $\mu g/m^3$ measured in the United States.[13] Moreover, the HNO_2 concentrations measured in this study are 1 order of magnitude higher than the HNO_3 concentrations.

Corrections Obtained Using More than One Denuder

HNO_3 Measurements. As observed previously in Table 2, nitrate concentrations collected by downstream denuders are similar and are 11–61% of the first denuder concentrations. Sampling biases may arise from field blanks. But field blank data were well below the IDLs, so it is suspected that downstream denuders should collect the N-containing interferents. This is supported by the fact that the nitrate concentrations in particles in Taiwan's atmosphere[14,19] are usually 1 order of

Table 2. HNO_3 concentrations measured with four denuders in series.

	Concentration ($\mu g/m^3$)			
	Mean	SD[a]	Range	N[b]
Outdoor measurement				
First denuder	0.37	0.12	0.18-0.55	29
Second denuder	0.10	0.02	0.06-0.13	29
Third denuder	0.08	0.02	<MDL-0.12	29
Fourth denuder	0.07	0.01	0.05-0.08	6
Indoor measurement[c]				
First denuder	4.19	2.91	1.10-9.64	7
Second denuder	0.16	0.02	0.13-0.19	7
Third denuder	0.14	0.02	0.12-0.15	7
Fourth denuder	0.13	0.04	0.08-0.17	5
Temperature (°C)	21	5	11-37	36
Relative humidity (%)	61	10	46-74	36

[a]Standard deviation; [b]Number of samples; [c]With an artificial HNO_3 source introduced.

Table 3. HNO_2 concentrations measured with four denuders in series.

	Concentration ($\mu g/m^3$)			
	Mean	SD[a]	Range	N[b]
Pre-denuder (for HNO_3)	0.35	0.08	0.22–0.48	12
First denuder	4.32	0.74	3.52–5.63	12
Second denuder	0.31	0.04	0.27–0.39	12
Third denuder	0.30	0.07	0.23–0.41	12
Denuder difference[c]	4.01	0.73	2.96–5.30	
Temperature (°C)	24	6	15–32	12
Relative humidity (%)	60	9	46–73	12

[a]Standard deviation; [b]Number of samples; [c]This gives the ambient HNO_2 concentration using the arrangement shown in Figure 1.[1] It is calculated by subtracting the concentration measured by the second denuder from that by the first denuder.

magnitude higher than those in the United States.[12] Another possibility is that nitric oxide (NO) or NO_2 were partially collected and measured as HNO_3.[6-8]

Figure 2 depicts the relationship of HNO_3 concentrations collected by the first denuder versus corrected concentrations obtained using additional denuders in series. The corrected HNO_3 concentrations obtained by two or three additional denuders are theoretically calculated using both assumptions of incomplete absorption ($C_{tar,inc}$) and perfect absorption ($C_{tar,perf}$) at the first denuder. The additional denuders are used for the correction of HNO_3 concentration. The line in Figure 2 is the 1:1 line where the two concentrations are the same. It is seen that if HNO_3 concentrations collected by the first denuder are high, the assumptions of incomplete absorption and perfect absorption at the first denuders lead to analogous results, and the HNO_3 concentration collected by the first denuder is very close to the corrected concentration obtained using additional denuders. But for low HNO_3 concentrations, high deviations are observed for both assumptions. This is especially true for perfect absorption assumption by the first denuder.

HNO_2 Measurements. Similar comparisons were also made for the HNO_2 measurements. It was observed that the corrected HNO_2 concentrations obtained using additional denuders are slightly less than concentrations collected by the first denuder; hence, the results are not shown. The use of one denuder only seems to lead to an error of about 6–9% for the HNO_2 concentration range studied (>3 $\mu g/m^3$). Therefore, depending on the concentration of HNO_2 in the atmosphere, the level of interference, and the degree of accuracy required for HNO_2 measurements,

one may not necessarily follow the arrangement[1] shown in Figure 1. For field sampling conducted in this study, only one denuder would be enough for HNO_2 sampling.

Relative Errors of HNO_3 Measurements

Results of relative errors under the perfect absorption assumption are shown in Figure 3a as a function of the HNO_3 gas collected by the first denuder. The relative error for the one-denuder system was calculated from the average concentrations of downstream denuders divided by the corrected HNO_3 concentration ($C_{tar,perf}$). The relative error for the two-denuder system was calculated from the difference between the second denuder and the average of downstream denuders divided by $C_{tar,perf}$. For the ADS arrangement where only one denuder is employed for HNO_3 sampling,[1] the estimated deviations (0.06–0.12 $\mu g/m^3$) from their corrected values are not related to concentrations collected by the first denuder. Thus, the relative errors are higher for HNO_3 concentrations of less than 1.0 $\mu g/m^3$.

The difference can be higher than 80% if the first denuder concentrations are about 0.2 $\mu g/m^3$. But if the ADS has adopted two denuders for HNO_3 sampling, by subtracting the concentrations of the second denuders from those of the first denuders, the relative errors can be significantly reduced as shown in Figure 3a. The estimated deviations (0.00–0.03 $\mu g/m^3$) from their corrected values for the two-denuder system are also not a function of the HNO_3 concentration collected by the first denuder; thus the relative errors are less than 30% for the first denuder concentrations of about 0.2 $\mu g/m^3$. In addition, they can be less than 10% for the first denuder concentrations of over 0.4 $\mu g/m^3$.
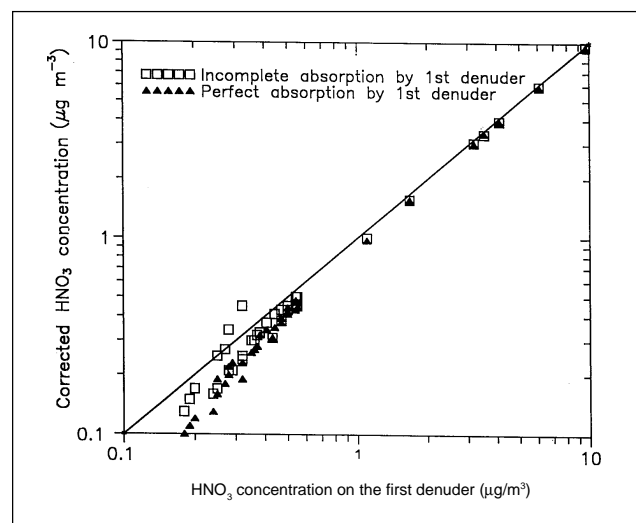

Figure 2. Relationship of HNO_3 concentrations collected by the first denuder versus corrected concentrations obtained using two or three additional denuders in series.

Figure 3a. Relative errors of one- and two-denuder systems as a function of HNO_3 concentration collected by the first denuder under the complete absorption assumption.
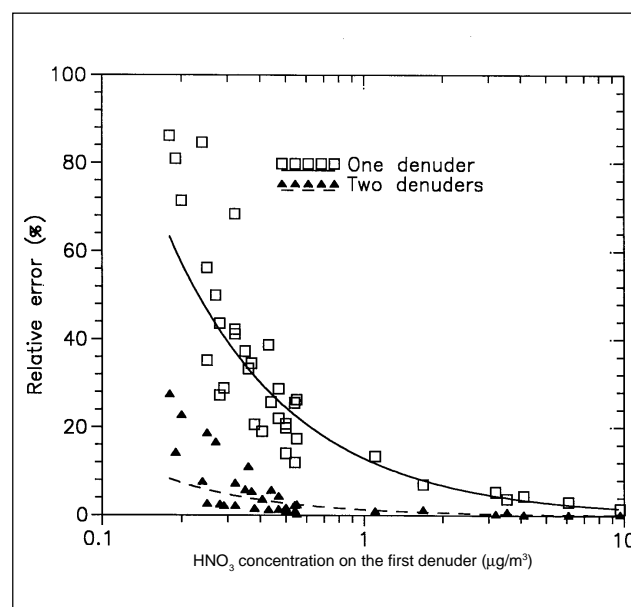
Figure 3b. Relative errors of one- and two-denuder systems as a function of HNO$_3$ concentration collected by the first denuder under the incomplete absorption assumption.
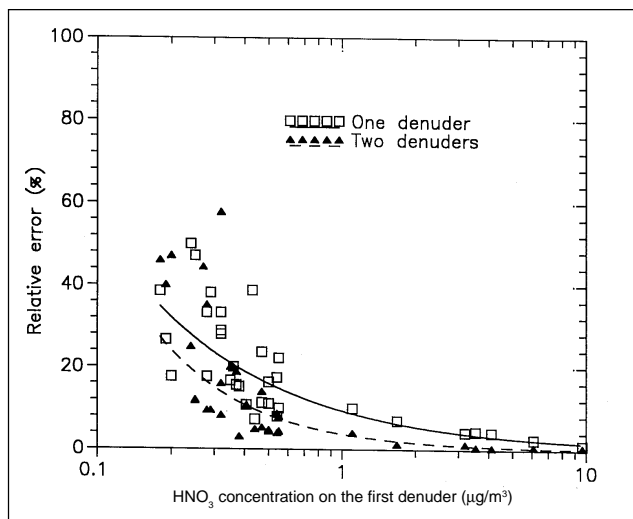
Figure 3b calculates and shows the sampling errors that can occur if the ambient HNO$_3$ gas is not completely absorbed by the first denuder, for both one- and two-denuder sampling systems. The calculation of the sampling errors for the one- and two-denuder systems is the same as that for the perfect absorption assumption case shown in Figure 3a, except that the corrected HNO$_3$ concentration is C$_{tar,inc}$ instead of C$_{tar,perf}$. It is seen that using two denuders in the ADS does not significantly reduce the relative sampling errors unless the HNO$_3$ gas concentration is over 0.5 μg/m^3. For some cases of low HNO$_3$ concentrations, using two denuders may even produce larger sampling errors. But incomplete absorption for low HNO$_3$ ambient concentration should not happen because it was demonstrated[2,3] that the denuder system has a very high absorption efficiency for HNO$_3$ gas, unless the HNO$_3$ gas concentration is high.[20]

CONCLUSIONS

Differences in the HNO$_3$ and HNO$_2$ gas concentrations obtained using different ADS arrangements have been evaluated in this study. The field measurements of HNO$_3$ gas indicated that using only one NaCl denuder can maintain the relative error to be less than 5% for the first denuder concentration of higher than 3.0 μg/m^3. But if the HNO$_3$ concentration measured at the first denuder was about 0.4 μg/m^3, the relative sampling error was approximately 40%. This may result in over 80% relative error for the first denuder concentration of about 0.2 μg/m^3, and using two NaCl denuders in the ADS for HNO$_3$ sampling may reduce the relative errors to within 30%, even for very low HNO$_3$ concentrations. Therefore, the two-denuder system is recommended for low concentrations of HNO$_3$ gas sampling.

The field measurements of HNO$_2$ gas by only one denuder can maintain the relative error to be less than about 10%, according to this study. Hence, it may not necessarily require two Na$_2$CO$_3$ denuders to collect HNO$_2$ gas. The spare denuder can then be used for the additional NaCl denuder required for HNO$_3$ sampling. But the risk for high error while measuring low HNO$_2$ concentrations by only one Na$_2$CO$_3$ denuder is also possible. Therefore, before field experiments of HNO$_2$ and HNO$_3$ sampling, pretests are suggested to evaluate possible sources and degrees of sampling errors. The sampling errors of these two gases can, therefore, be minimized with a better arrangement of the ADS.

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