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Recovery of Gaseous Emission from Ground Level Area Sources of Ammonia and Hydrogen Sulfide Using Dynamic Isolation Flux Chambers

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Abstract. *Controlled laboratory experiments were conducted to evaluate the recovery of ammonia (NH_3) and hydrogen sulfide (H_2S) emitted from ground level area sources (GLAS) using dynamic isolation flux chambers. H_2S (80–4,000 ppb) and NH_3 (5,000–40,000 ppb) were diffused through the flux chamber to simulate GLAS emissions while measuring the inlet and outlet flux chamber concentrations simultaneously. Results showed that the recovery of H_2S during a 30-minute sampling time was almost complete for concentrations greater than 2,000 ppb. At the lowest concentration of 80 ppb H_2S , about 92.55% of the gas could be recovered for the given sampling period. NH_3 emissions exhibited similar behavior between concentrations of 5,000–40,000 ppb. About 92.62% of NH_3 could be recovered within the same 30-minute sampling period and at the lowest concentration of 5,000 ppb. Complete recovery is achieved for concentrations greater than 40,000 ppb. Predictive equations were developed for gas recovery. These equations predicted that the maximum difference between chamber inlet and outlet concentrations of NH_3 or H_2S was 7% at the lowest concentration used for either gas. The use of 46 – 90 m of Teflon tubing with the flux chambers had no effect on gas adsorption measurements because recovery was completed almost instantaneously at the beginning of the tests.*

Keywords. Ammonia, hydrogen sulfide, flux chambers, ground level area sources, adsorption.

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Introduction

Ammonia and hydrogen sulfide are compounds emitted from animal feeding operations (AFO) that are gaining particular importance. Although these compounds are not regulated under the National Ambient Air Quality Standards (NAAQS), these substances are perceived to be a nuisance and could contribute to ozone formation in the ambient air and the formation of PM_{2.5}. Several methods were used to estimate ammonia emissions directly from livestock sources. A review presenting more than 11 techniques for measuring ammonia has been presented by Phillips et al.¹. One particular method is the use of isolation flux chambers.

Isolation flux chambers have been used to measure volatile gas emissions from ground level area sources. The technique involves enclosing a section of the emitting surface with a cover, ventilating this covered area at some known rate, and measuring the concentration in the exhaust air of the gas under study. The design of these enclosures varies as to the amount of area covered, the geometry of the enclosure, the ventilation rate of the enclosure, the design of the ventilating system, and the handling of air coming into the enclosure.² One concern about flux chambers has been the perceived volatile gas emission adsorption losses as well as variability in concentration measurements with air flow rates and level of concentrations.³ There are numerous factors that have to be accounted for in the measurements. The main focus of this particular study was primarily on the adsorption behavior of ammonia onto flux chambers and associated conveyance tubing. The other factors mentioned will be dealt with in the future.

Authors working on adsorption phenomena have accounted for adsorption losses through the use of differential equations. Mukhtar et al.⁴ discussed ammonia adsorption onto conveyance tubing and a regression equation was used, whereas a linear differential equation (eq 1) was used by Aneja et al.⁵.

$$\frac{d[C]}{dt} = \left[\frac{Q[C_o]}{V} + \frac{JA_i}{V} \right] - \left[\frac{LA_c[C]}{V} + \frac{Q[C]}{V} \right] - R \quad (1)$$

The variable C in equation (1) is the concentration of the gas monitored in the chamber; Q is the flow rate of the carrier gas through the chamber; C_o the concentration of the gas in ambient air, V is the volume of the chamber; J the emission flux; A_i the surface area covered by the chamber; A_c the inner surface area of the chamber; L the loss term by the chamber wall per unit area assumed first order in [C]; R the chemical production rate in the chamber; and h the internal height of the chamber.

When using ammonia-free air (zero air) as carrier gas, C_o = 0 and equation (1) simplifies to:

$$\frac{dC}{dt} = \frac{J}{h} - \left[\frac{L}{h} + \frac{Q}{V} \right] [C] \quad (2)$$

Concentration data from work with flux chambers are converted into gas emission rates from AFOs. Adsorption of NH₃ onto flux chambers and associated conveyance tubes might bring about errors in reporting emission rates and should thus be taken into consideration. This study evaluated the adsorption phenomena of NH₃ and H₂S onto flux chambers to determine whether any significant adsorption onto flux chambers and conveyance tubing were taken into account when reporting emission factors.

It has been reported by Klenbush et al.⁶ that flux chamber emission rate measurements made on soil cells range from 50% to 100% of the predicted emission rates. That is, the measured emission rates can be expected to be within a factor of one-half of the "true" emission rates. The

flux chamber accuracy based upon both the recovery tests and predictive modeling ranges from 50% to 124%. It was further noted that the sensitivity of this method depends on the detection limit of the analytical technique used (Klenbush et al.⁷).

The precision and accuracy of the flux chamber method was further researched by Gholson et al.⁸. They found this method to have compound-dependent negative bias ranging from 40-80% in their laboratory studies. The source of this bias was investigated but no definitive cause was determined.

Practical guidance to ensure the validity of fugitive VOC emission rates from flux chamber measurements has been presented by Eklund,⁹. However, no discussion regarding the sensitivity of the analytical instruments used was given and the potential adsorption onto tubing and chamber walls was also not addressed.

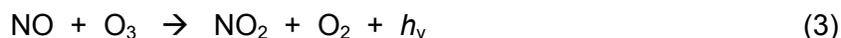
The adsorption loss of ammonia in flux chambers was accounted for by Aneja et al.¹⁰ by defining a loss term per unit surface area of the chamber (eq 1 and eq 2). The value of the total loss term was reported as 0.059 m/min and 0.079 m/min during summer and winter measurements, respectively. These losses were determined through surface loss experiments originally proposed by Kaplan et al.¹¹, which involved the application of an empirical equation to estimate the loss term similar to eq 2. The studies also reported that the chamber reached steady state conditions within 30 minutes of operation. In another study by Aneja et al.⁵, the reported loss term was 0.0276 m/min. Failure to report the correct adsorption loss term and the chemical production/destruction rate in association with the use of flux chamber methods may result to 5% and 50% reduction in flux values, respectively (Aneja et al.¹²).

The goal of this work is to have a better understanding of the adsorption phenomena of NH₃ and H₂S in flux chamber methods under controlled laboratory conditions and the optimization of the analytical method used to measure the gases. The specific objectives are as follows:

- a. To evaluate the recovery of NH₃ and H₂S due to adsorption from an isolation flux chamber sampling protocol;
- b. To determine the level of adsorption of ammonia and hydrogen sulfide onto flux chambers at different gas concentration; and
- c. To develop predictive equations to account for adsorption of gases over the range of concentrations used.

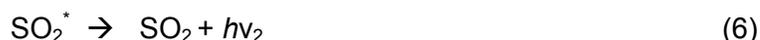
MATERIALS AND METHODS

In the laboratory controlled experiments, NH₃ was measured from the inlet and the outlet of a flux chamber using two analyzers (Model 17C Thermo Environmental Instruments, TEI, Franklin, MA) simultaneously. The analyzer measures NH₃ by the use of chemiluminescence based on the reaction of nitric oxide (NO) with ozone (O₃), given as eq 3.



The ammonia sample is drawn into the analyzer where it is converted to NO in a converter chamber. The NO then goes into the reaction chamber where it mixes with ozone (generated internally). The reaction creates a luminescence proportional to the concentration of NO.

For H₂S, the analysis is based on the quantitative oxidation of H₂S to SO₂ (eq 4):



The H₂S determination at the inlet and outlet of the flux chamber was also done using two analyzers (Model 340 Thermo Environmental Instruments, TEI, Franklin, MA) simultaneously. The measurement of H₂S is possible by a pulsed fluorescence technique involving eqs 5 and 6.

General Experimental Protocol

The steps in the general experimental procedure for this study are as follows:

1. **Stabilization** of the instruments, to ensure a good baseline that accounts for background and residual analyte
2. **Calibration** of instruments for NO₂ and NH₃ or SO₂ and H₂S over the concentrations to be used.
3. **Optimization** of converter efficiency to ensure that calibration factors are very close to 1.0 for all the ranges of desired concentration,
4. **Running of experiment** - begins by connecting the flux chambers to the gas lines, directing a portion of the inlet gas to the TEI and directing the outlet gas to the other TEI.
 - measurement of temperature and relative humidity with HOBO multi-parameter sensor (Onset Communications, Bourne, MA)
 - three replications
5. **Flushing** with zero grade air at the end of each test
6. **Post calibration** check

Laboratory Setup

Figure 1 shows the schematic of the setup to measure adsorption behavior of the gas sample (NH₃ or H₂S). Two NH₃ and two H₂S measuring instruments with five mass controllers (Allborg Instruments and Controls, Inc., Orangeburg, NY) were used through all the tests. One mass flow controller (MFC1) accurately controls the flow of high purity gas (Scott Specialty Gases, Houston, TX), and the other (MFC2) controls the amount of dilution from zero air generator (AADCO 737 Pure Air Generator, Village of Cleves, OH). Each TEI unit is also provided with mass flow controllers (MFC4 and MFC5) to accurately measure the exact amount of sampled gas mass flow rates through the units. All exhaust gases were directed to a fume hood. The sensitivity of the equipment is 2% full scale (at 100 ppm NH₃ and 10ppm H₂S, respectively) and no adsorption losses were reported if the difference in the outlet concentration and inlet concentration was within this range (TEI¹³).

A National Instruments LabVIEW (Version 6.1) program was used to control and regulate the flow and concentration of NH₃ and H₂S by using the programmable mass flow controllers (MFCs). Each gas to be tested was mixed well with zero grade air in a static mixer. The correct concentration of NH₃ or H₂S was then conveyed through the chamber at 7 liters per minute (LPM) during each experiment and this flow rate was also controlled by a mass flow controller (MFC3). The LabVIEW program allowed for the inlet NH₃ or H₂S concentration to be varied. The gas leaving the chamber was conveyed to the analyzer at a flow rate of 2 LPM regulated by a mass flow controller (MFC4).

The Isolation Flux Chamber

The flux chambers were custom-fabricated similar in design to the chambers described by Eklund et al.¹⁴. The schematic is shown in Figure 2. The dimensions of the flux chambers are as follows: diameter (a) = 49.5 cm, height of skirt (b) = 22.9 cm and provided with a semicircular dome with a height (c) = 16.5 cm. The total volume of the chamber is 64.5 liters and the cross sectional area ("foot print") of the base is 0.19 m². The flux chamber dome was made of acrylic and manufactured by Odotech, Inc., Montreal, Canada, following EPA recommendations. The bottom skirt was custom built by a local machine shop in College Station, TX and was made of stainless steel. The total surface area of the chamber (dome and wall) was about 0.745 m².

RESULTS AND DISCUSSION

It was observed throughout the course of the experiment that the performance of the TEI analyzers was affected by the concentration of gases. It was noted that at higher concentrations, the TEI could not be calibrated properly and even if the highest calibration factors were used, the known concentration of the gas could not be obtained. Thus, at higher concentrations, the converter efficiency of the unit had to be adjusted to display the correct concentration from certified gas source. This meant adjusting the factory set converter efficiency to reach the desired concentration. This was achieved by increasing the temperature of the converter so that more NH_3 or H_2S gases could be converted into NO_2 or SO_2 . Although the factory setting for the NH_3 converter temperature is fixed (about 775°C) this had to be adjusted to display the correct concentration of calibration gases. The acceptable range is between 300 to 1000°C . The optimum temperature is higher for higher concentration levels. Only one optimum converter temperature level was used for all the tests.

The converter efficiency was also affected by the level of oxygen available in the gas streams. At very high concentration when the dilution free air was very minimal, the desired inlet concentration could not be achieved and the concentration reading declined as the test progressed. Thus, there was a limit on the amount of dilution air required by the system.

NH_3 Recovery Tests

Figure 3 shows the adsorption-desorption curves for all the tests conducted for ammonia at concentrations between $5,000$ ppb to $40,000$ ppb. The differences in the inlet and outlet concentrations were well within the sensitivity of the gas sensing equipment at concentrations above $40,000$ ppm. Thus, tests using concentrations more than this level were discontinued. When the differences in inlet and outlet concentrations were calculated at the end of each run, the results are those shown in Table 1 and illustrated in Figure 4. To calculate the final outlet concentrations, the average of the last five minutes of the run was used. The adsorption curves were almost horizontal during the last five minutes of the experimental runs.

The highest difference in the inlet and outlet concentrations was observed at the lowest NH_3 concentration of $5,000$ ppb. About 92.62% of the ammonia dispersed through the flux chamber could be recovered at that level of concentration. The change in adsorption follows a logarithmic curve as shown in Figure 4. At $10,000$ ppb NH_3 about 95.73% of the ammonia could be recovered and the recovery was almost complete between $30,000$ to $40,000$ ppb. The concentration difference at this concentration range was about 2% and well within the sensitivity of the equipment.

The predictive equation to estimate percent un-recovered NH_3 is given below with a correlation (R^2) of 0.9378 .

$$\% \text{NH}_3 \text{ un-recovered} = -2.44 * \ln(\text{NH}_3 \text{ concentration}) + 27.68 \quad (7)$$

At $30,000$ ppb of NH_3 , the predicted un-recovered gas was about 2.5% and was similar to the actual data of 2.55% . The concentration wherein the un-recovered gas is well within the 2% sensitivity of the instruments based on this predictive equation is about $37,000$ ppb. Thus, at NH_3 concentrations greater than this, one can be confident that no adsorption losses may be reported following a 30 minute flux protocol sampling time. When correcting for un-recovered ammonia in the calculation of the emission factor, eq 7 may be used.

H_2S Recovery Tests

Figure 5 shows the adsorption-desorption behavior for H_2S as the gas was diffused through the flux chamber. All H_2S was recovered from the chamber at concentrations of $2,000$ ppb and 4000 ppb. The plot of the differences in inlet and outlet H_2S concentrations for the different runs in

Figure 6 shows that the recovery of H₂S increased as the concentration was increased. Table 2 shows the average recovery from all the tests for H₂S.

The percent recovery for H₂S ranged from 92.55% at the lowest inlet concentration of 80 ppb to 98.54% at inlet concentrations above 2,000 ppb. The un-recovered H₂S can also be estimated by the predictive eq 8 below with an R² of 0.968.

$$\% \text{ H}_2\text{S un-recovered} = -1.52 * \ln (\text{H}_2\text{S concentration}) + 13.64 \quad (8)$$

At 2,100 ppb of H₂S, the predicted un-recovered gas was at 2% and is the minimum level of detection by the equipment. At the lowest concentration of 80 ppb, the un-recovered gas percentage calculated from predictive equation was about 7% and 7.5% from the actual experimental runs (Table 2). When correcting for this effect in the calculation of the emission factor for H₂S, eq 8 may be used and recovery should be more than 90% even at a lowest concentration of 10 ppb.

Tests for NH₃ Adsorption on Teflon Tubing

The adsorption of NH₃ on 46 and 90 meters of Teflon tubing was also investigated for its possible effect on ammonia recovery from the flux chamber. Figures 7 and 8 illustrate the adsorption-desorption curves showing negligible adsorption of NH₃ onto 46 and 90 m of tubing. The outlet and inlet concentration are nearly identical at 10 ppm and 20 ppm NH₃. Further tests were performed at the ranges of concentrations used in the gas adsorption tests and there was no difference in inlet and outlet concentrations of NH₃. Thus, it was concluded that the adsorption of NH₃ on Teflon tubing of lengths up to 90 m have no effect on gas adsorption and recovery measurements from flux chambers. Thus, for estimating emission factors, the use of up to 90 m Teflon tubing will be of no consequence to gas adsorption over the entire concentration range provided the instruments (analyzers and flow controllers) were calibrated correctly.

CONCLUSIONS

This study has shown that 92.62% of ammonia diffused through the flux chamber could be recovered within the 30-minute sampling period at the lowest concentration of 5,000 ppb. Complete recovery is achieved on concentrations greater than 40,000 ppb. The recovery of hydrogen sulfide during the same sampling period was almost complete for concentrations greater than 2000 ppb. At the lowest concentration of 80 ppb H₂S, about 92.55% of the gas could be recovered and reported. The use of 46 m to 90 m of Teflon tubing had no effect on gas adsorption as recovery was completed almost instantaneously at the beginning of tests. Thus, in the calculation for determination of emission factors, no adsorption loss correction factor due to the Teflon tubing is needed.

The gas adsorption in flux chambers for both NH₃ and H₂S was about 7% at the lowest concentrations used in this study (5,000 ppb and 80 ppb for NH₃ and H₂S, respectively). The recovery at the chamber outlet increased as the gas concentration was increased. The adsorption behavior of NH₃ and H₂S could be explained by a predictive equation which could estimate percent adsorption depending on the average equilibrium concentration encountered at the chamber inlet during the test. It was theorized that at higher gas concentrations, the adsorption sites could easily be filled up within the 30-minute sampling period and thus adsorption become insignificant and should not affect calculations of the emission rates. At lower gas concentrations, the site saturation period may take longer and thus at the end of the 30 minutes of sampling time, the inlet and outlet concentrations are still quite different. The difference in concentrations may be reported as losses but in reality, there may not be any

adsorption loss if equilibrium conditions were achieved. But these would require sampling at longer periods.

The flux-chamber data may be used to estimate NH₃ and H₂S emission factors provided the actual recovery of these gases from the chamber is taken into account. For short term flux chamber gas measurements, a transfer function may be developed to account for the transition from a non-steady state condition to an equilibrium condition, thereby reducing the sampling time.

FUTURE WORK

Future experimental studies will be directed at developing transfer functions as a result of the use of flux chambers. Sampling period may be reduced substantially if this is done and one may not have to wait for longer equilibrium conditions to estimate gas emissions from GLAS. Uncertainty analysis will also be performed in conjunction with the use of flux chambers. By combining the results for this work and the uncertainty analysis study, we expect that emission factor calculations for GLAS will be improved. More important future work will be directed toward simulating the effect of sources depletion and the consequence of using different air flow rates, humidity and temperatures within the flux chambers to investigate how these factors affect concentration measurements.

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Table 1. Average NH₃ adsorption and recovery for different inlet concentrations.

NH ₃ Inlet Concentration (ppb)	Adsorption (%)				Standard Deviation (%)	NH ₃ Recovery (%)
	Trial 1	Trial 2	Trial 3	Mean		
5000	8.79	7.02	6.33	7.38	1.27	92.62
10000	4.98	4.20	3.64	4.27	0.67	95.73
20000	4.00	3.44	3.64	3.69	0.28	96.31
30000	2.91	2.40	2.35	2.55	0.31	97.45
40000	2.36	2.04	1.23	1.88	0.58	98.11

Table 2. Average H₂S adsorption and recovery for different inlet concentrations.

H ₂ S Inlet Concentration(ppb)	Adsorption (%)				Standard Deviation (%)	H ₂ S Recovery (%)
	Trial 1	Trial 2	Trial 3	Mean		

80	6.21	6.38	9.76	7.45	2.00	92.55
100	6.63	6.14	5.82	6.20	0.41	93.80
200	5.41	4.92	6.25	5.53	0.67	94.47
1000	3.75	3.01	2.92	3.23	0.46	96.77
2000	2.30	1.06	0.94	1.43	0.75	98.57
4000	1.12	1.88	1.39	1.46	0.39	98.54

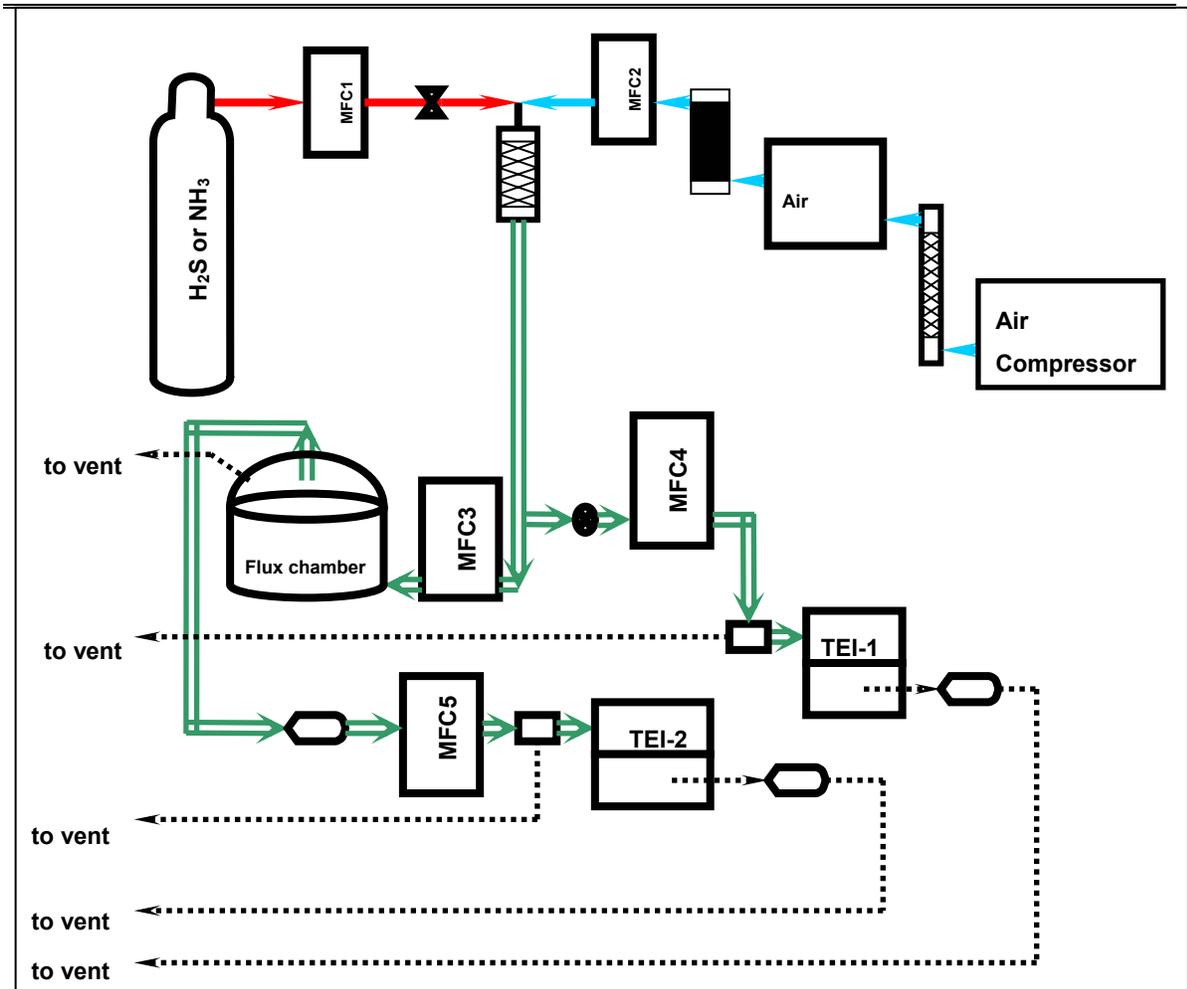
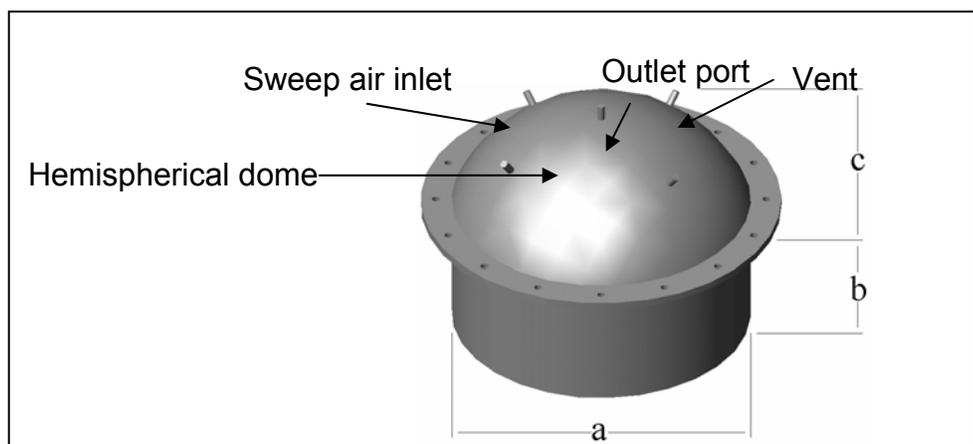


Figure 1. Schematic of experimental set-up for gas recovery tests.



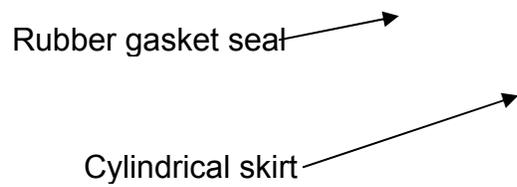


Figure 2. Schematic of the isolation flux chamber used in this study.

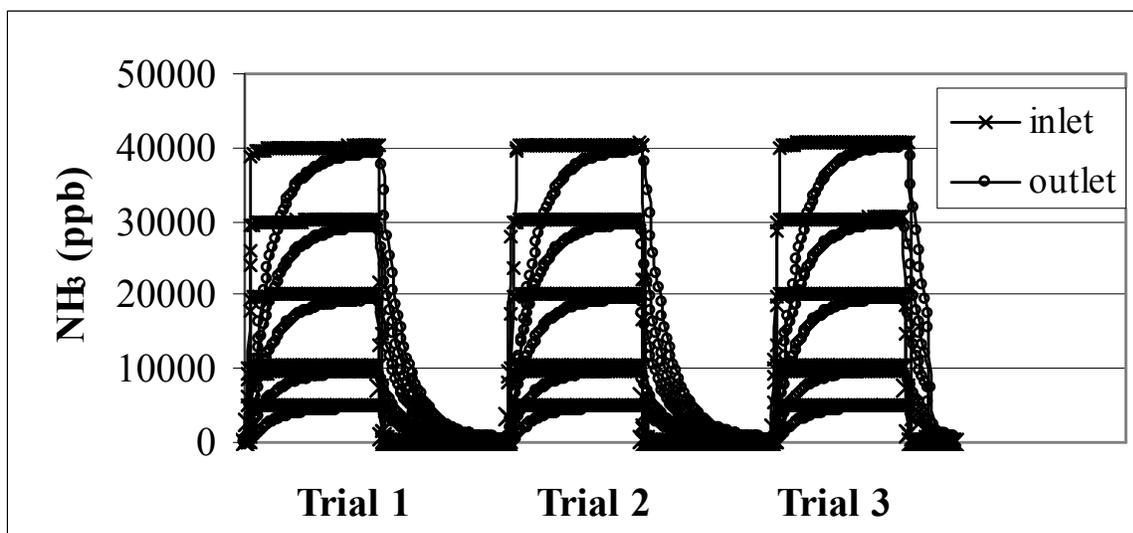


Figure 3. NH_3 adsorption – desorption curves between 5,000 – 40,000 ppb inlet concentrations.

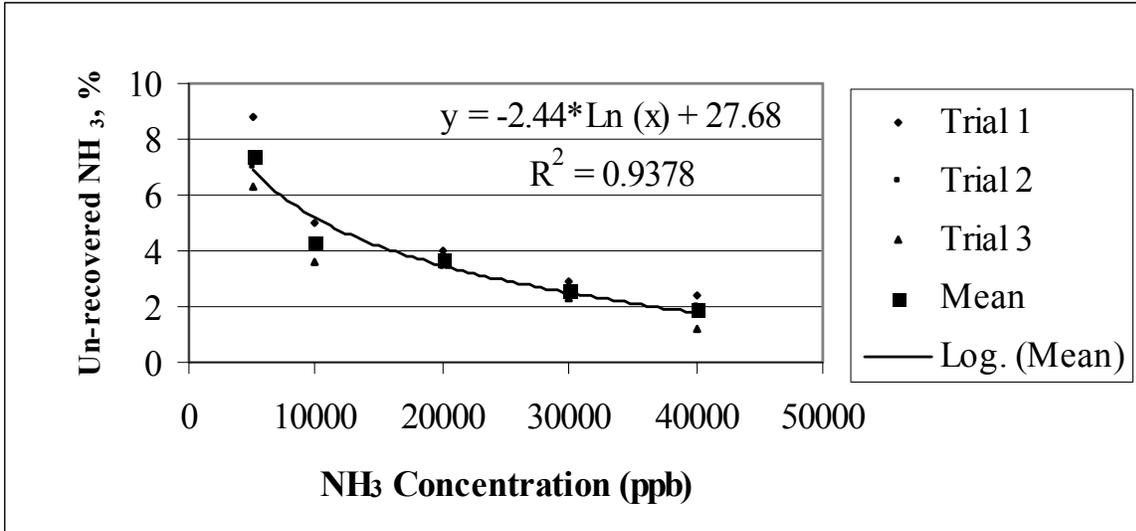


Figure 4. Plots of un-recovered NH₃ at inlet concentrations between 5,000 to 40,000 pb.

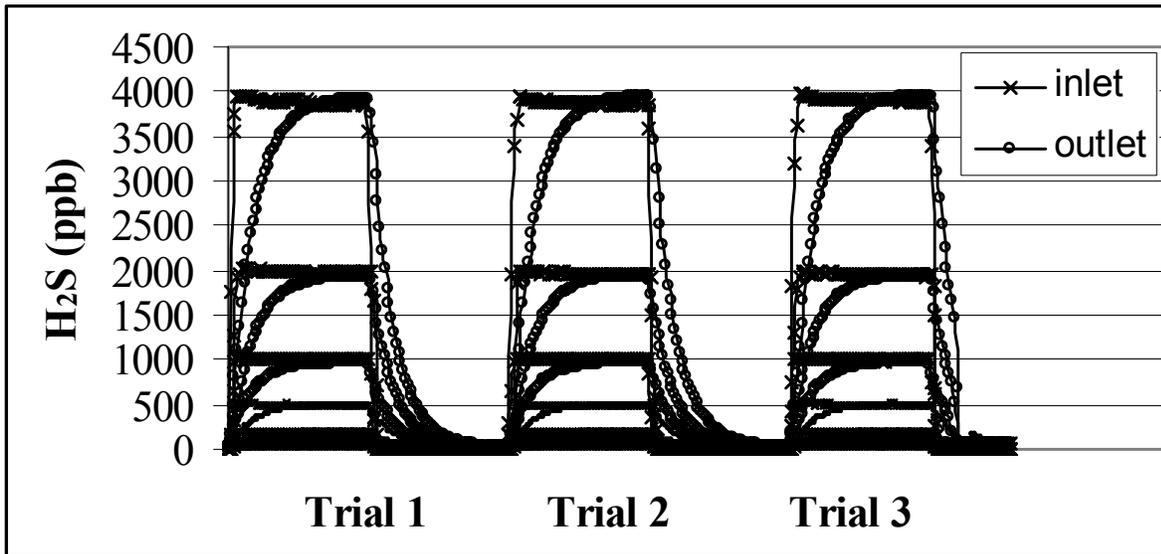


Figure 5. H₂S adsorption-desorption curves between 80 - 4000 ppb inlet concentrations.

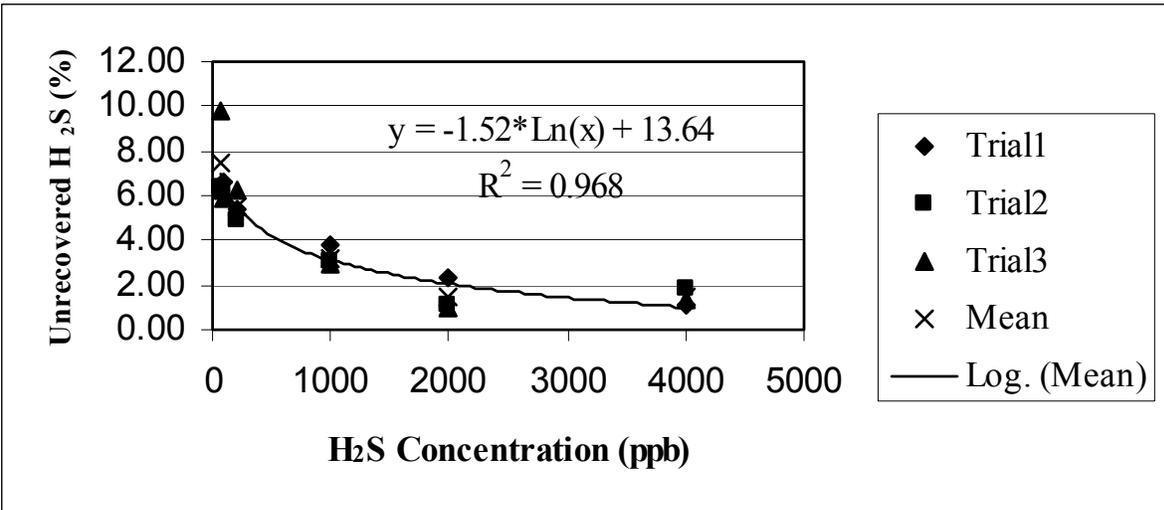


Figure 6. Plots of un-recovered H₂S at inlet concentrations between 80 to 4,000 ppb.

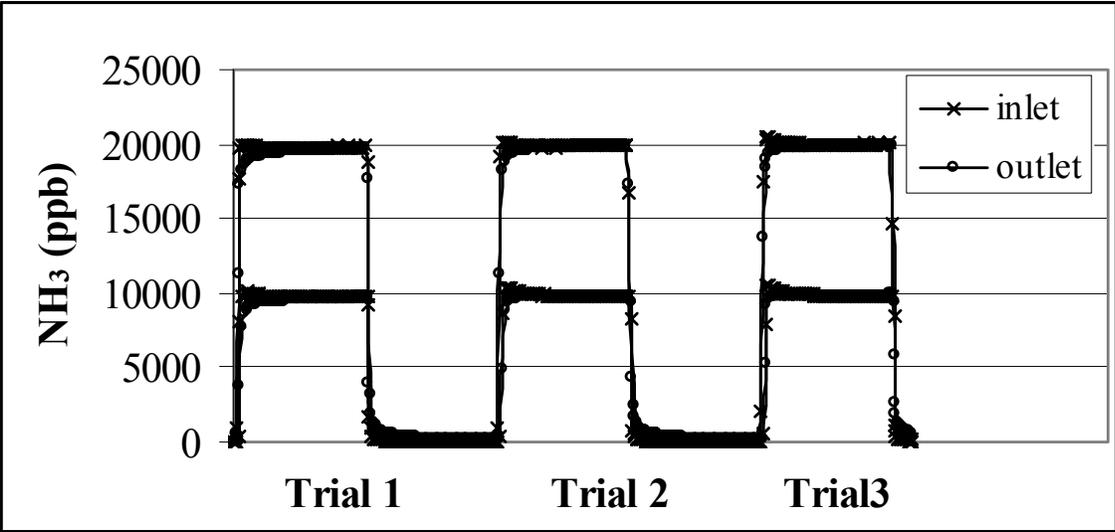


Figure 7. Adsorption – desorption curves of NH₃ on 90 m Teflon Tubing at concentrations of 10 and 20 ppm.

Figure 8. Adsorption – desorption curves of NH₃ on 46 m Teflon Tubing at



concentrations of 10 ppm.