
Dynamic atmospheric chamber systems: applications to trace gas emissions from soil and plant uptake

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Abstract: Atmospheric emissions, transport, transformation and deposition of trace gases may be simulated through chambers. The dynamic flow-through chamber system has been developed in response to a need to measure emissions of nitrogen, sulphur and carbon compounds for a variety of field applications. Oxides of nitrogen (NO, NO₂, NO_x) emissions have been measured from agricultural fertilised/unfertilised soils. Ammonia-nitrogen (NH₃-N) and reduced organic sulphur compound emissions have been measured using this same technique across a gas-liquid and soil-atmosphere interface at swine waste treatment anaerobic storage lagoons and in agricultural fields. Similar chamber systems have also been deployed to measure the uptake of nitrogen, sulphur, ozone and hydrogen peroxide gases by crops and vegetation to examine atmospheric-biospheric interactions. Emission measurements compare well with a coupled gas-liquid transfer with chemical reaction model as well as a US Environmental Protection Agency (EPA) WATER9 model.

Keywords: ammonia; dynamic chamber; emissions; hydrogen peroxide; hydrogen sulphide; nitrogen oxide.

Reference to this paper should be made as follows: Aneja, V.P., Blunden, J., Claiborn, C.S. and Rogers, H.H. (2006) 'Dynamic atmospheric chamber systems: applications to trace gas emissions from soil and plant uptake', *Int. J. Global Environmental Issues*, Vol. 6, Nos. 2/3, pp.253-269.

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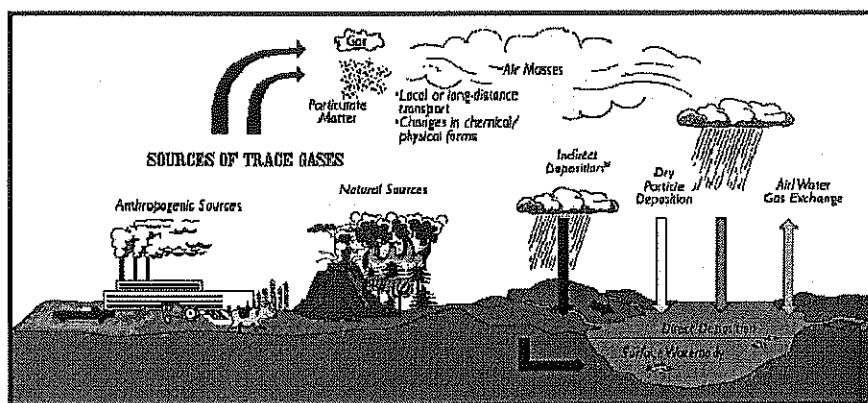
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1 Introduction

The biogeochemical cycling of trace gases on local, regional, and global scales is a complex system of emissions, transformations, transport, and deposition (Aneja et al., 2001b, Figure 1). Dynamic atmospheric chambers have been utilised to measure these atmospheric processes.

Figure 1 Atmospheric emissions, transport, transformation, and deposition of trace gases (Aneja et al., 2001b)



The dynamic flow-through chamber system is a technique that has been developed and modified over the past 25 years in an effort to measure earth-atmosphere and water-atmosphere fluxes of various compounds including biogenic sulphur, oxides of nitrogen, ammonia and methane as well as many other gases. Determination of fluxes for these different gaseous compounds is desirable in order to permit the accurate assessment of the relative roles of biogenic and anthropogenic sources in contributing to such phenomena as the atmospheric sulphate and nitrate burdens, acidity in precipitation due to biogenic emissions, and contribution to aerosol formation through atmospheric photochemical processes in localised areas downwind of suspect sources. Similar systems

have also been utilised to measure the exposure of plants to various air pollutants, and to quantify the uptake of trace gases by plants (e.g. Aneja, 1976; Claiborn and Aneja, 1993; Heck et al., 1987; Rogers et al., 1979).

Hill et al. (1978) initially developed this technique to measure biogenic sulphur fluxes from a salt marsh in Long Island, New York (Eklund, 1992). More recently, the dynamic flow-through chamber system has also been used over soils and liquid surfaces to measure various gaseous emissions such as ammonia (Aneja et al., 2000, 2001a,b; Roelle and Aneja, 2002), nitric oxide (Li et al., 1999; Roelle et al., 2001; Tabachow et al., 2002), methane (Chan et al., 1998; Zahn et al., 2001), VOCs (Sartin et al., 2002), carbon dioxide (Eriksen and Jensen, 2001; Knapp et al., 1997) and mercury (Carpi and Lindberg, 1998; Ferrara and Mazzolai, 1998; Gårdfeldt et al., 2001; Poissant et al., 2004) in a variety of environments. Currently, a study by the North Carolina State University Air Quality research group, in coordination with the US Department of Agriculture National Research Initiative, is underway to measure simultaneous emissions of nitric oxide, ammonia and hydrogen sulphide from swine waste treatment lagoons using this same technique.

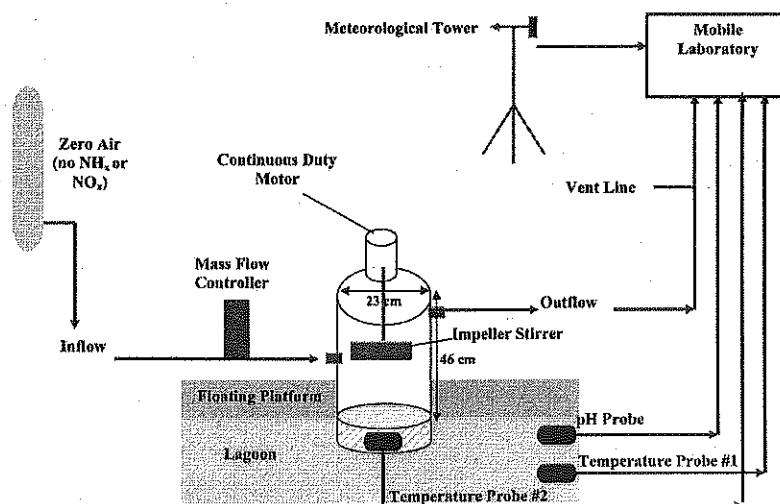
For measuring emissions, the chamber technique has the important advantage of association of a particular emission site and its measurable array of physical, chemical and microbiological properties with emissions of particular compounds or their reaction products. In addition, gas residence times in the chamber are in the order of minutes so that chemical transformations between emission and analysis may be minimised. The entire chamber system is small, lightweight and easily transportable, thus making a survey of trace gas flux very convenient. Finally, the system is relatively inexpensive and labour requirements are low. However, it should be noted that the chamber system cannot completely simulate the ambient environment. The chamber measures over a small area (requires extrapolation) and extrapolation to large scale may be a problem. For measuring fluxes to plants, the dynamic chamber is operated as a well-mixed chamber. Ideally, then, mixing is instantaneous, conditions are uniform throughout the chamber, and exit conditions are representative of internal, well-mixed conditions, thus simplifying calculations of fluxes and mathematical treatment of non-plant surface losses (Aneja, 1976; Claiborn and Aneja, 1993).

2 Dynamic flow-through chamber for emissions at the air-water/soil interface

In the dynamic emission flux chamber method, a continuously stirred tank reactor (CSTR; Levenspiel, 1962) with an open bottom is placed over an area of interest of soil, mud, or water (Hill et al., 1978). For flux measurements at the air-water interface, a floating platform is used to hold the chamber system above water. A hole is cut in the centre of the platform in which the chamber rests, allowing the bottom of the chamber to penetrate the water surface by 3–4 cm, thus forming a seal between the water surface and the air within the chamber and thereby providing a completely enclosed system in which to measure gaseous fluxes. Figure 2 depicts a schematic of a typical dynamic flow-through chamber system used to measure gaseous emissions from a swine waste treatment lagoon. For soil measurements, the chamber is placed onto a stainless steel ring which is then inserted into the soil. Flux measurements are begun after steady-state conditions have been reached within the chamber. The residence time, τ , is defined as the chamber volume, V , divided by the carrier flow rate, q . Based on the continuously stirred tank reactor (CSTR) theory

(Levenspiel, 1962), it takes ~ 6 residence times before steady-state concentrations are reached inside the chamber and sampling can be initiated. For example, if a chamber has an internal volume of 0.025 m^3 and the carrier gas flow rate is $5 \text{ L (} 0.005 \text{ m}^3 \text{) min}^{-1}$, it would take ~ 30 -minutes for steady-state conditions to be reached. The time it takes to reach steady state is dependent on the flow-rate and internal chamber volume and so may vary between different experiments.

Figure 2 Schematic of dynamic flow-through chamber system configured to measure emissions from a swine waste treatment lagoon



Over the years construction of the dynamic chamber has been modified and adapted in order to adhere to specific applications in a variety of environments (Eklund, 1992). A user's guide is available that summarises guidance on the design, construction, and operation of the US Environmental Protection Agency's (US EPA) recommended dynamic flux chamber (Kienbusch, 1986). For studies discussed in this paper, the chamber is generally constructed as follows. The dynamic flow-through chamber system is cylindrical in nature and built from Plexiglas material. Chamber dimensions may vary but are generally about 23 cm inner diameter (i.d.) and 46 cm in height. The entire closed system is lined on the inside with 5 ml fluorinated ethylene propylene (FEP) Teflon and stainless steel fittings in order to minimise chemical reactions with sample flow.

A carrier gas such as compressed zero-grade air is passed through a Teflon FEP sample line into the chamber. The in-flowing air is monitored by a mass flow controller and subsequently delivered into the chamber. The air inside the chamber is ideally well-mixed by a variable-speed motor-driven Teflon impeller stirrer (speeds generally range from ~ 40 – 100 rpm). The dynamic chamber system, with the continuous stirring provided by the impeller, meets the necessary criteria for performance as a continuously stirred tank reactor (CSTR). For performance as a CSTR, the chamber needs to be 'ideally' mixed (Aneja, 1976). In ideal mixing, the composition of any elemental volume within the chamber is assumed to be the same as that of any other volume within the

chamber. Tracer experiments (residence time distribution) have been used to test the flow and mixing characteristics of the system. The results of these mixing studies indicated that the dynamic chamber behaved as a 'perfect' mixer with negligible stagnancy or channelling (Aneja et al., 2000).

Johansson and Granat (1984) conducted research on pressure differences between the outside atmosphere and air within a chamber using a tilted water manometer, which indicated that pressure differences were below detectable limits (0.2 mm H₂O). Gao and Yates (1998) examined the carrier flow relationship to pressure changes inside the chamber and concluded that pressure deficit within the chamber should be minimised for accurate flux measurements. Arkinson (2003) conducted research on the temperature difference between the outside atmosphere and air within the chamber over a liquid surface. The results indicate that temperature differences are $\leq 0.4^\circ\text{C}$. Reichman and Rolston (2002) experimentally determined that, on a daily basis, average soil temperature perturbation at 1 cm depth was about 1.5°C , which is expected to cause only small errors in flux measurements.

The out-flowing gas also flows through a Teflon FEP sample line and is directed into a temperature controlled mobile laboratory, which houses the analytical instruments and data acquisition system. Here, the sample is analysed continuously for content of the gas of interest. A vent line is fitted to the exiting sample line to prevent pressurisation and is periodically bubble tested to check for under pressurisation and/or leaks in the enclosed system. Sample lines do not exceed 10 m.

3 Flux calculations

The following mass balance equation may be used for the dynamic flow-through chamber system and applied to any target gaseous substance of known concentration: where

$$\frac{dC}{dt} = \left(\frac{q[C_{air}]}{V} + \frac{JA}{V} \right) - (C) \left(\frac{LA_w}{V} + \frac{q}{V} \right) - R, \quad (1)$$

C Concentration of gas inside the chamber (ppbV)

C_{air} concentration of gas in carrier air (ppbV)

q Flow rate of compressed air through the chamber (lpm)

V Volume of the chamber (L)

A Emission surface area covered by chamber (m²)

A_w Inner surface area of the chamber of inner and upper wall surfaces (m²)

L Total loss of gas in the chamber per unit area (m min⁻¹) due to reaction with inner and upper walls of the chamber

h Internal height of the chamber (cm)

J Emission flux per unit area ($\mu\text{g} [\text{gas}] \text{m}^{-2} \text{s}^{-1}$)

R Gas phase reactions inside the chamber

Since zero-grade air is used as the carrier gas, C_{air} is equal to zero and gas phase reactions, R , are also assumed to be zero. Since the air inside the chamber is assumed to be well mixed by the impeller stirrer, C is assumed to be constant within the chamber. At steady-state conditions, the change of concentration with respect to time $\left(\frac{dC}{dt}\right)$ is expected to be zero. Therefore Equation (1) can be simplified as:

$$\frac{J}{h} = C_{eq} \left(\frac{LA_w}{V} + \frac{q}{V} \right). \quad (2)$$

Loss term, L , is determined experimentally while equilibrium-state gas concentration (C_{eq}), flow rate (q) and chamber dimensions (V and h) are all measured. Kaplan et al. (1988) has devised a method for calculating loss term by calculating the slope of the plot of $-\ln \left[\frac{C_{eq} - C(t)}{C_{eq} - C_0} \right]$ versus time (t). For this experiment, C_0 is the initial equilibrium state gaseous concentration measured by the chamber system at a constant flow rate (lpm). C_{eq} is the measured gaseous concentration at a second equilibrium state at an increased or reduced flow rate (lpm) into the chamber system. $C(t)$ depicts the gaseous concentration at any time, t , during the transition between the first and second equilibrium states. L is determined by:

$$L = \left(\text{slope} - \frac{q}{V} \right) \left(\frac{V}{A_w} \right) \quad (3)$$

4 Dynamic flow-through chamber for measuring trace gas uptake by plants

For measuring the uptake of gaseous species by plants, and for conducting exposure experiments, it is important to characterise the conditions very near the plant surface under study; thus, assuring well-mixed conditions for such experiments becomes very important. Claiborn and Aneja (1993) constructed a chamber with a 1:1 diameter to height ratio, which is ideal for well-mixed conditions (Uhl and Gray, 1966). The chamber is constructed of a Teflon-coated strap iron frame lined internally with a 5 ml thick Teflon film to allow for maximum light, and even light and temperature distribution. Mixing is provided by a Teflon-coated impeller mounted at the top of the frame, and aided by three Teflon-coated baffles. The entire system operates under slight negative pressure, and it is important to ensure that there are no leaks into the system, to allow for the proper determination of the air flow rate through the system and the initial concentration of the trace gas of interest. The air flow rate through the chamber provides one to two chamber exchange rates per minute. Sample ports are located on the inlet and outlet Teflon pipes. Separate sample manifolds are provided for CO_2 /water vapour and the trace gas of interest. Air samples from the chamber inlet and outlet are alternated automatically to the analysers so that concentration differences across the chamber are determined using the same analyser.

In order to determine trace gas uptake, wall losses to the empty chamber surfaces must first be determined. The fraction of the trace gas introduced into the chamber that is lost to the chamber walls during the chamber loss experiment (CL) is determined:

$$CL = \frac{C_{A_o,empty} - C_{A,empty}}{C_{A_o,empty}}, \quad (4)$$

where C_A and C_{A_o} are the outlet and inlet concentrations, respectively, of trace gas species A, and the subscript *empty* indicates the concentrations measured for the empty chamber experiment.

Then, total losses (i.e. to both plant surfaces and chamber surfaces) are measured, and the fractional loss (TL) of the trace gas entering the chamber is similarly determined:

$$TL = \frac{C_{A_o,exp} - C_{A,exp}}{C_{A_o,exp}}, \quad (5)$$

where C_A and C_{A_o} are, again, the outlet and inlet concentrations, respectively, of trace gas species A, and the subscript *exp* indicates the concentrations measured for the exposure chamber experiment.

5 Treatment of wall losses

For trace gases that are not very reactive (Seinfeld and Pandis, 1998), it is sufficient to subtract the wall losses from the empty chamber experiment from the total trace gas losses from the plant uptake experiments in order to determine the losses to plant surfaces, alone. In other words,

$$BL = TL - CL, \quad (6)$$

where BL is the fractional loss of the trace gas to plant biomass surfaces, alone. For very reactive and/or water-soluble species like hydrogen peroxide or nitric acid, however, a more rigorous method of correcting for wall losses must be employed (Claiborn and Aneja, 1993). By treating both wall losses and losses to plant surfaces as two parallel reactions, the fractional loss to the plant biomass surfaces, BL, is calculated from TL and CL,

$$Q_A = \frac{(BL)(F)C_{A_o,exp}}{A_L}. \quad (7)$$

From this equation, it is evident that for species for which the chamber losses are very low, it is acceptable to simply subtract the fractional loss to the walls (CL) from the fractional loss during the exposure chamber (TL).

Finally, fluxes to plant surfaces (Q_A) are calculated from the fractional loss to the plant surfaces (BL), the chamber air flow rate (F), the inlet trace gas concentration, and the plant surface area (A_L):

$$Q_A = \frac{(BL)(F)C_{A_0,exp}}{A_L} \quad (8)$$

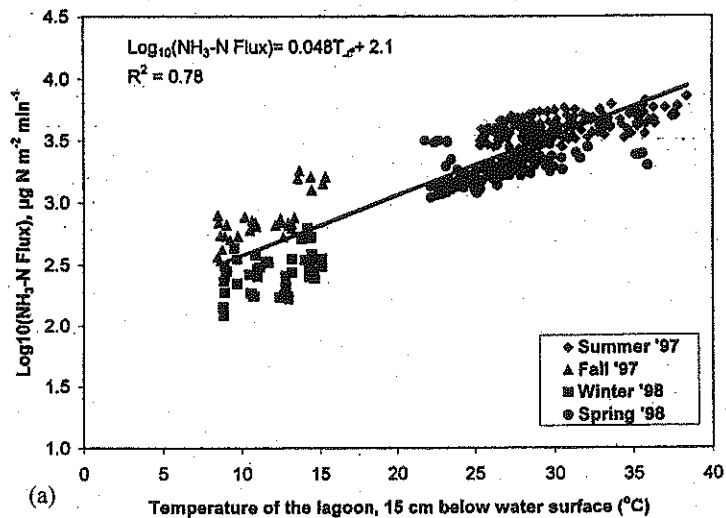
6 Results

The graphs in Figure 3 (a–d) provide results from some field studies undertaken to measure fluxes of selected S, N, and C compounds. Figure 3a (Aneja et al., 2001c) reports the log of hourly averaged ammonia-nitrogen flux from a swine waste treatment lagoon plotted against lagoon aqueous phase surface temperature during four different seasonal measurements. The graph shows an observed exponential ($r^2 = 0.78$) relationship between NH_3 and lagoon water temperature measured over the year. Figure 3b demonstrates the effect of soil temperature on NO from an agricultural (soybean) crop soil at four different sites. Figure 3c illustrates the temperature dependence of average H_2S emission fluxes from a salt marsh. The emission rate appears to increase semi-logarithmically with increasing temperature within a temperature range of $\sim 0^\circ\text{C}$ to $\sim 40^\circ\text{C}$. Finally, Figure 3d represents isoprene (hydrocarbon) concentration plotted against temperature during warm months (April – September). Please note that hydrocarbon concentrations (ppbV), which are treated as a surrogate for emission (since the concentration measurements are in close proximity to the source), are reported rather than flux calculations. The graph depicts the linear relationship between the logarithm of isoprene concentration and temperature using data from four sites. (It should be noted that a flux chamber was not used. However, Guenther et al. (1996) measured isoprene emissions from the leaves using a field portable dynamic chamber and observed that the flux is directly related to the concentration measurements near the source, suggesting that isoprene concentration measurements near the source are a surrogate for emissions).

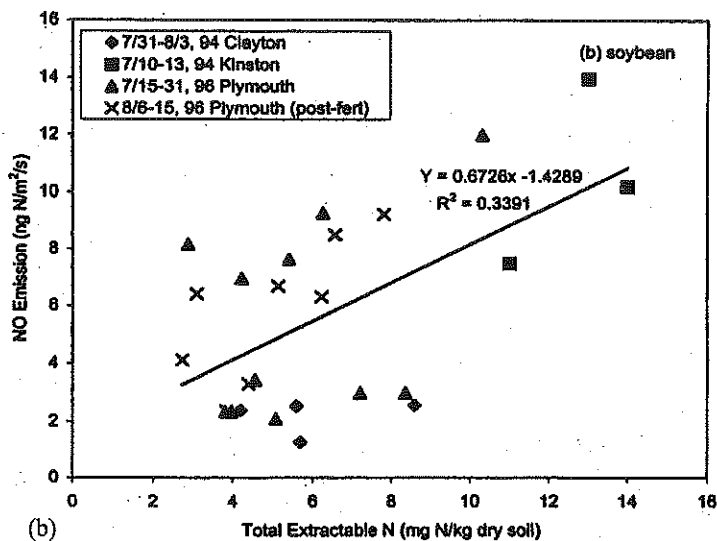
The well-mixed chamber was used to measure the uptake of gaseous hydrogen peroxide by spruce saplings (coniferous species). Hydrogen peroxide is a highly reactive and water-soluble atmospheric oxidant, for which wall losses are very significant (10–35% for one to two air exchanges per minute) and highly variable (standard deviation of 50% for daytime experiments) (Claiborn, 1991). A linear relationship between hydrogen peroxide flux and chamber concentration was apparent for the red spruce saplings (Figure 4). Note that there is no significant difference between daytime and night time fluxes. Average deposition velocities were calculated for these experiments, and ranged from $0.08 - 0.12 \text{ cm s}^{-1}$, and $0.08 - 0.12 \text{ cm s}^{-1}$ for daytime and nighttime values, respectively. In contrast, similar uptake experiments conducted on bean plants (broad leaf plants) resulted in deposition velocities ranging from 0.8 to 1.4 and 0.5 to 0.6 for days and nights, respectively (Claiborn, 1991).

Chamber experiments on uptake by plants provide useful information for deposition velocity determinations; however the deposition velocities over forest canopies will be different from those calculated in the chamber experiments. Deposition velocities take into account not only the resistance to mass transfer due to the plant itself, but also the resistance due to the forest canopy structure, and the turbulence over the canopy.

Figure 3 (a,b) Emissions of NH₃ (Aneja et al., 2000) and NO (Aneja et al., 2001c), respectively, versus temperature at various locations



(a)



(b)

Figure 3 (c,d) Emissions of H₂S (Hill et al., 1978) and mixing ratios for isoprene (Hagerman et al., 1997), respectively, versus temperature at various locations

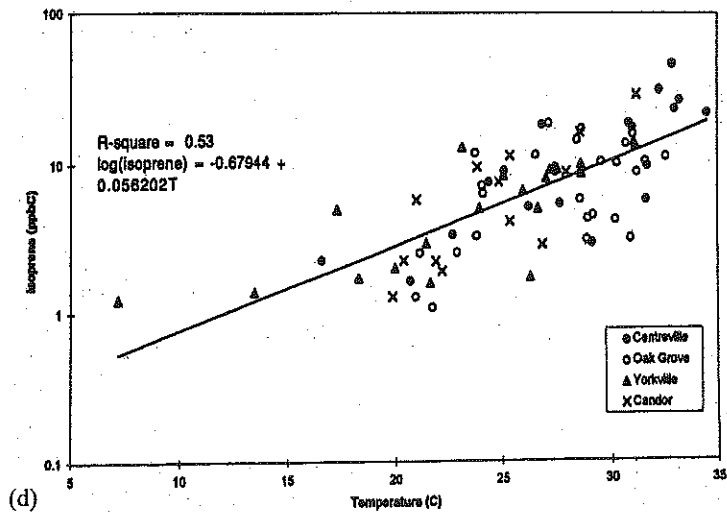
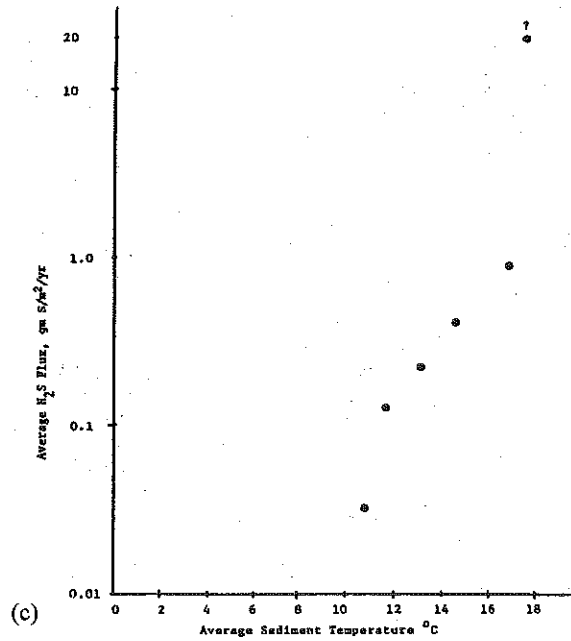
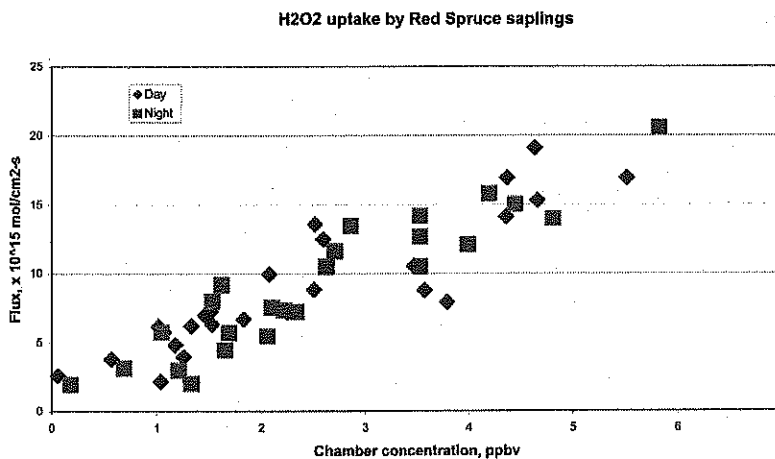


Figure 4 Fluxes of gaseous H₂O₂ to needles of red spruce saplings as measured in well-mixed chamber experiments. Diamonds denote daytime fluxes and squares denote night time fluxes (Claiborn, 1991)



7 Data comparisons

As with any type of experimental procedure, it is desirable to compare data acquired in field studies with controlled laboratory studies and/or related models. Various comparisons with models, as well as other measurement techniques, have been made with data collected via the dynamic flow-through chamber system. Some results are presented in the following discussion.

For model comparisons with the dynamic flow-through chamber system, Aneja et al., (2001d) developed a mass transport model based on the quiescent thin film concept (Danckwerts, 1970), which takes into account molecular diffusion and chemical reactions. Figure 5 shows the seasonal variation for ammonia emissions from a swine waste treatment lagoon using the dynamic chamber system experimental technique (Aneja et al., 2000) compared with subsequent modelling results. The coupled mass transfer model simulations corroborated experimental results utilising the dynamic chamber technique and validated the application to the flux equation (Aneja et al., 2001d). Also, Figure 6 shows how closely the recently developed WATER9 model, based on a set of air emission models that are documented in Air Emissions Models for Waste and Wastewater, US Environmental Protection Agency, (US EPA, 1994), compare with the same set of ammonia emissions data as discussed with the mass transport model previously.

Tabachow et al. (2002) measured nitric oxide (NO) emissions from unamended and municipal wastewater treatment plant biosolids-amended soil in both laboratory and *in situ* field experiments and compared the results. Based on matching soil temperatures (within 5°C) and water filled pore space (WFPS) ($\pm 5\%$), the ratio of volumetric soil water to total porosity of the soil, five of the six scenarios showed no statistically significant difference in the NO flux measurements from the lab vs the field studies.

Figure 5 Seasonal variation comparison of ammonia emissions between modelling results and dynamic chamber system experiments

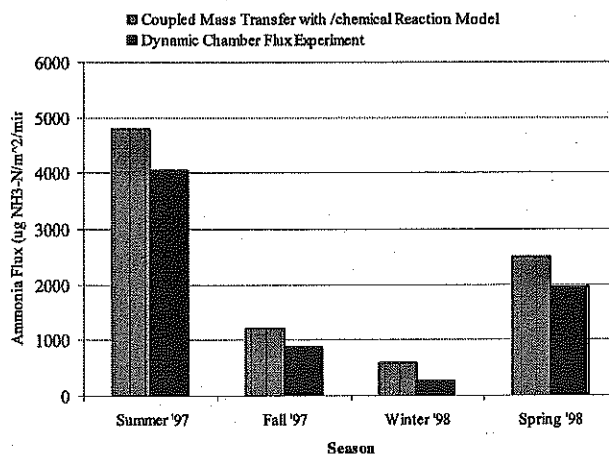
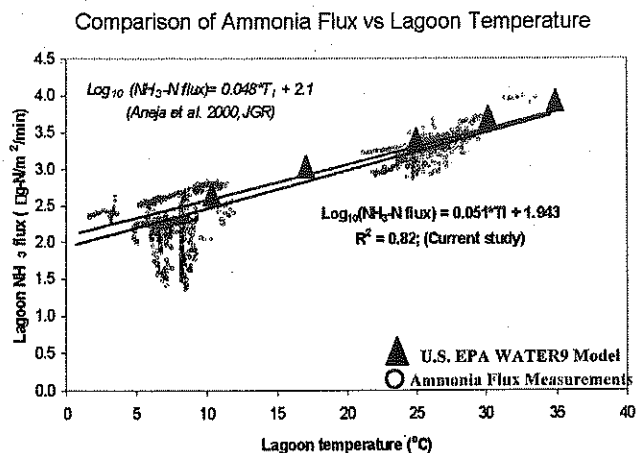
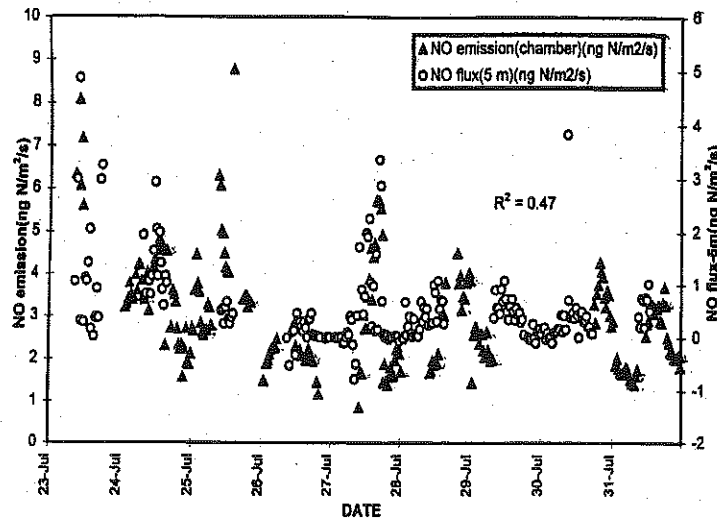


Figure 6 Ammonia flux versus lagoon temperature comparison between WATER9 modelling results and dynamic chamber experimental calculated results



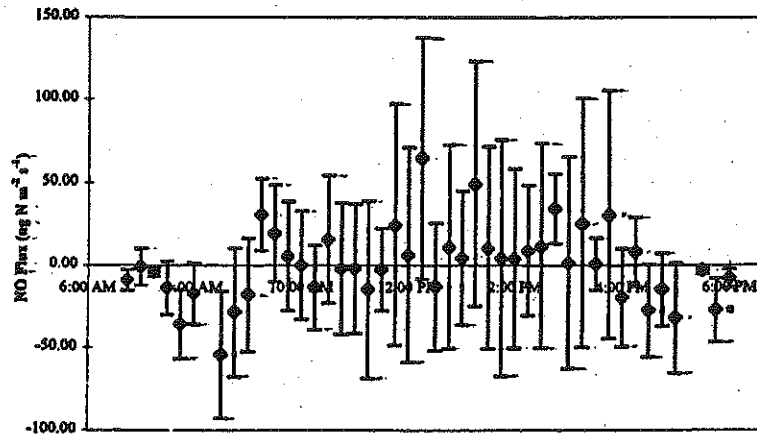
Nitric oxide emissions from soils on a soybean field, measured by the dynamic chamber system (Li et al., 1999), were compared in Figure 7 with NO fluxes calculated at a 5 m height utilising the eddy-correlation method (Gao et al., 1996). In spite of differences in flux magnitudes (note the different scales used), the two show qualitatively similar variations and structure with time. The values of NO soil emissions are greater than the NO fluxes at 5 m, which implies that some of the NO might have been converted to nitrogen dioxide (NO₂) through reactions with ozone and peroxy radicals by the time it reaches the 5 m height level from the source. R² = 0.47 indicates the degree of correlation between the two techniques, indicating that there may be other parameters for each technique that explain the variability in emissions.

Figure 7 Intercomparison of NO fluxes at 5 m height using the eddy-correlation technique with NO soil emissions using the dynamic chamber technique



In a coordinated research effort for Project NOVA 1995 (Natural Emissions of Oxidant Precursors: Validation of Techniques and Assessment) (Aneja, 1994), NO emissions were measured from fine sandy loam soil in a corn field using two different (dynamic and static) chamber techniques. One group from North Carolina State University used the dynamic chamber method while a group from NASA Langley Research Center used a closed box flux technique (static chamber), in which NO fluxes were calculated using the mixing ratio of NO (ppbv) versus time. (Anderson and Levine, 1987). Figure 8 depicts a difference plot of the composite averaged fluxes calculated by the dynamic chamber technique and the static chamber technique. Differences between the 400 measurements were calculated and the resulting Δ flux were analysed using the SAS statistical package. Due to time series activity in the data, PROC ARIMA (autoregressive integrated moving average) was used to model the data and test the hypothesis that the mean of these differences was zero, meaning that both chamber methods yield statistically identical values (SAS Institute, Inc., Cary, NC, USA). The statistical model which best fit the data consisted of a mean parameter and two autoregressive parameters. The SAS output produced a T-ratio = 0.15 for MU, the mean of differences. This value indicates that there is not enough evidence to statistically reject the hypothesis that the mean of the differences is zero. Therefore, as the hypothesis fails to be rejected, the conclusion must be that there was no statistical difference between NO flux as measured using the two different chamber methods (Roelle et al., 1999).

Figure 8 Difference of NO flux between two chamber techniques (dynamic chamber flux – static chamber flux) versus time of day. Vertical lines indicate one standard deviation of the NO flux measurements made from both chamber techniques



8 Conclusions

The dynamic flow-through chamber system has been successfully developed in response to a need to measure emissions of nitrogen, sulphur and carbon compounds for a variety of field applications. Dynamic chamber systems provide an inexpensive and versatile approach to measuring the fluxes and fate of trace gases into the atmosphere from land/water surfaces. For example, chamber techniques are often more appropriate than micrometeorological flux techniques when fetch requirements are difficult to meet, or when the goal is to understand not only flux rates, but also to identify the important underlying parameters and processes that influence the flux. Moreover, chamber systems have also been deployed to measure uptake of nitrogen, sulphur, ozone and hydrogen peroxide gases by crops and vegetation to examine atmospheric-biospheric interactions. Experimental results obtained by dynamic chamber systems have been effectively compared with independent flux models.

Acknowledgements

Funding of this research project was provided by the US Department of Agriculture (USDA) as a part of the National Research Initiative (NRI) under Contract No. 2003-05360. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

This paper was for oral presentation at NATO Advanced Research Workshop, Environmental Simulation Chambers: Application to Atmospheric Chemical Processes in Zakopane, Poland, October 2004.

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