The Measurement of Nitrous Oxide Emissions from Soil by Using Chambers [and Discussion]

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The measurement of nitrous oxide emissions from soil by using chambers

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Small flux chambers are widely used to measure emissions of nitrous oxide, N_2O, from soil, the gas being determined by gas chromatography with an electron capture detector. The technique is relatively cheap, and is adaptable to a wide range of site conditions and emission rates: from the order of $1\ \mu g\ m^{-2}\ h^{-1}$ to more than $10\ mg\ m^{-2}\ h^{-1}$. Increasingly, systems are being automated, to get more information on short-term temporal variability and to collect data over long periods to improve estimates of total annual emissions. Such systems are being used in the field and with soil monoliths installed in a greenhouse. Large chambers 50–60 m^2 in area, with gas analysis by long-path infrared spectrometry, offer a way of overcoming small-scale spatial variability, and are useful in conditions where micrometeorological methods may not be applicable, or when long runs of data are needed from the same site. In studies with small closed chambers, we have measured N_2O emissions from grassland ranging from negligible values to about $4\ mg\ N_2O\text{–}N\ m^{-2}\ h^{-1}$ (nearly $1\ kg\ N_2O\text{–}N\ ha^{-1}\ d^{-1}$), with total losses in the range 0.14–5.1% of the nitrogen applied as fertilizer, depending on factors such as soil structure, water potential and temperature, and the chemical form of the fertilizer. Reasonable agreement can be obtained between chamber and micrometeorological flux measurements on homogeneous sites.

1. Introduction

Nitrous oxide, N_2O, is of environmental importance because it contributes to global warming (Houghton *et al.* 1990) and the depletion of the stratospheric ozone layer (Crutzen 1981). Soils have been identified by the Intergovernmental Panel on Climate Change (IPCC) as the major source of N_2O in the atmosphere (Houghton *et al.* 1990). The total emissions from all sources are estimated by Houghton *et al.* (1992) to be some 5–16 $Tg\ N_2O\text{–}N\ a^{-1}$, with 2.8–7.7 Tg attributed to soils – although these figures exclude temperate grassland soils.

Annual emissions of N_2O into the atmosphere are believed to have increased greatly during the last few decades (Houghton *et al.* 1990) and this is generally

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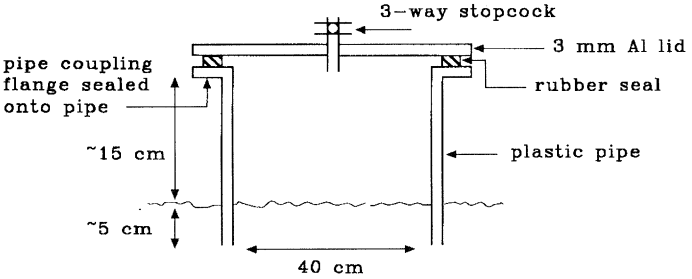

Figure 1. Cylindrical closed chamber (Clayton & Smith, unpublished work).

attributed to anthropogenic activity of various sorts. In spite of the wide uncertainty range for the flux of N_2O from agricultural land contained in the IPCC assessment, there is evidence that this flux may be one of the major contributors to the total global emissions of this gas, and one that is increasing with time as a result of expansion in the area of cultivated and disturbed land and an increase in the inputs of nitrogen in the form of mineral fertilizers and organic manures. Worldwide, about 77 Tg of nitrogen is added annually to soils as fertilizer (Granli & Bøckman 1994), and most of this is ultimately returned to the atmosphere as a result of denitrification to N_2 and N_2O. Even only a small proportion of N_2O would have a substantial effect on the global N_2O budget (Robertson 1993).

Measurements of N_2O between the atmosphere and land surfaces, whether in forests, natural grasslands or agricultural systems, have been based overwhelmingly on the use of small flux chambers, generally less than one square metre in area. A very limited amount of work has recently been carried out with much larger chambers. This paper describes the methods used, examines some of their advantages and disadvantages, and presents data obtained by these methods in some recent studies.

2. Chamber methods

(a) *Closed chambers*

The principle involved in measuring N_2O fluxes with so-called ‘closed chambers’ or ‘cover boxes’ is simple: a sealed enclosure is placed over the land surface and the change in N_2O concentration in the enclosure (usually an accumulation) is measured over periods of the order of one hour, normally by taking samples of a few millilitres and analysing the N_2O concentration directly by gas chromatography, using an electron capture detector (ECD; see, for example, Hutchinson & Mosier 1981).

A typical design is illustrated in figure 1, which shows the type of chamber used in much of our work in Edinburgh; each consists of a 20 cm length of polypropylene vent pipe (diameter 40 cm) fitted with a 4.5 cm wide outward-facing polyvinylchloride (PVC) flange at one end; the pipe is inserted about 5 cm into the soil, in a slot formed by a metal cutting ring. A $49 \times 49\ \mathrm{cm}^2$ square of 3 mm aluminium sheet with a circle of hollow-section rubber draught excluder on its underside is used as a lid, covering the flange and held in place by spring clips made from short lengths of square-section PVC electric cable ducting. The lid is fitted with a sampling port closed by a three-way tap. Sixty minutes after closing the lid, duplicate air samples are taken from the chamber, using airtight greased glass syringes suitable for sample storage for at least a few hours. Nitrous oxide is measured within this time by electron capture gas chromatography, using either a Poraplot Q or a Porapak Q column.

(*b*) *Open chambers*

In some circumstances, it is necessary to use so-called 'open chambers'. These are chambers through which a steady airstream is passed, and the gases emitted from the soil within the chamber are either measured directly in the emerging stream, or are adsorbed in some suitable trap, such as molecular sieve 5A, for subsequent release into a considerably smaller volume for analysis (see, for example, Egginton & Smith 1986). The open chamber/trapping method was formerly required for N_2O because gas chromatographic detectors were generally too insensitive to measure concentration increases directly in a closed chamber. This is no longer a problem, because of the improved sensitivity of present-day ECDs. However, the trapping method for N_2O is still useful when making, for example, simultaneous measurements of nitric oxide (NO) and N_2O emissions (Skiba *et al.* 1992); the NO is best determined by passing a rapid airstream through a chemiluminescence analyser, but in consequence the N_2O becomes diluted and requires concentration before analysis.

Each flux chamber only covers a very small area of land, and the marked spatial variability in N_2O emission which is generally observed (table 1) necessitates the use of as many replicate chambers as possible. The need to characterize temporal variability also increases the number of measurements that have to be made. These factors have encouraged the development of automated chamber systems combined with automated sampling and analysis, under computer control. Such systems have been described for N_2O measurements in a forest in Germany (Loftfield *et al.* 1992) and for measuring methane fluxes from rice paddies (Schutz *et al.* 1989). In our group, we are currently experimenting with automated chambers which can be linked to an automated sampling system, the whole assembly being powered by a battery pack and programmed to take a series of up to 24 samples over, say, one week at any one site.

An alternative to taking automation to the field is to transfer large intact soil monoliths from the field to a greenhouse or other indoor environment, fit them with an automated flux chamber/gas sampling/gas analysis system, and use them for detailed investigation of the effects of such variables as temperature, moisture content and nitrogen fertilizer input on N_2O flux (Thomson *et al.* 1994).

(*c*) *Large chambers*

Some recent developments (Galle *et al.* 1994; Smith *et al.* 1994*b*) have shown that N_2O fluxes can be measured from areas of land two to three orders of magnitude greater than those normally studied by the use of small chambers. A series of plastic hoops is covered with either tent fabric or polyethylene sheet to form a hemi-cylindrical chamber *ca.* 2 m in width and 25–30 m in length, rather like a large horticultural cloche. The accumulation of N_2O released from the land surface under the cover is measured with a Fourier transform infrared spectrometer (Galle *et al.* 1994), or with a relatively simple long-path infrared absorption spectrometer tuned to an N_2O absorption band at 2180–2200 cm^{-1} (figure 2). This device, the 'Hawk' gas monitor manufactured by Siemens Plessey, was designed

Table 1. *Spatial variability of N_2O fluxes and denitrification measured with small chambers*

experimental conditions	CV (%)	reference
random spacing (greater than 50 m); 0.13 m^2 and 0.49 m^2 chambers; ungrazed grassland ($n = 18$);	42–97%	Clayton *et al.* 1994
random spacing (greater than 50 m); 0.13 m^2 and 0.49 m^2 chambers; grazed grassland ($n = 6$)	59–183%	
grid, 7.1 m spacing; 0.008 m^2 chambers; ungrazed grassland	95% (mean)	Ambus & Christensen 1994
grid, 1–5 m spacing; 0.008 m^2 chambers; ungrazed grassland	50% (mean)	
grid, 1–2 m spacing; 0.012 m^2 chambers; N_2O and denitrification ($N_2O + N_2$) fluxes by C_2H_2 block method; fallow soil, irrigated	282–397% (N_2O) 161–508% ($N_2O + N_2$)	Folorunso & Rolston 1984

primarily for sensing leaks of gases from oil refineries and industrial sites, but by tuning to the appropriate wavelength it can be used to measure N_2O (Smith *et al.* 1994*b*).

3. Results and discussion

Some typical results obtained with conventional small chambers, mainly of the type illustrated in figure 1, are shown in figures 3–6. Figure 3 shows the relatively large fluxes of N_2O emitted from grassland on a heavy clay soil near Stirling in April 1992, following fertilization with 185 kg N ha^{-1} as NH_4NO_3. Emissions over a three-week period from an ungrazed area of the field corresponded to 1.7% of the nitrogen applied, while those from an area that had been grazed with cattle some months previously were 5.1%, three times greater. Measurements of soil moisture and mineral N contents in the two areas were not sufficiently different to account for the difference in the fluxes, but the surface of the grazed area was more heterogeneous than that of the ungrazed area, where livestock treading had produced many small puddled patches that could be expected to increase denitrification (Clayton *et al.* 1994).

Twenty-four closed chambers were used in this study; the coefficients of variation for the flux measurements are given in table 1. This study showed that spatial differences in emissivity are maintained over considerable periods; figure 4 shows the rank order of the chambers with respect to the N_2O fluxes measured over the three-week period of the experiment, with chamber 23, for example, giving the highest flux on all but two of the 15 sampling occasions.

Figure 5 shows emissions from grassland plots near Edinburgh fertilized and cut three times in each season, simulating the normal agricultural practice of

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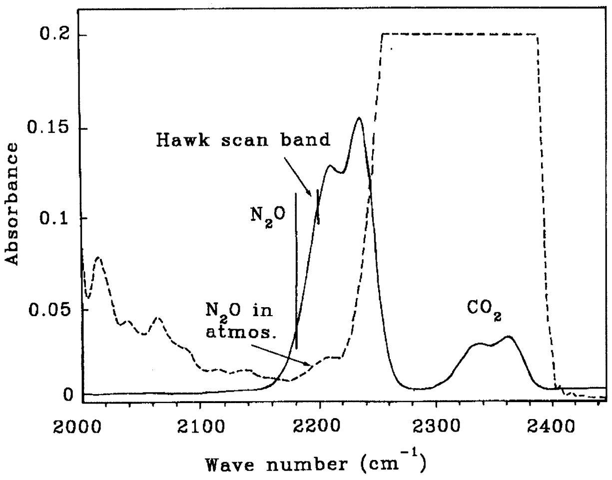

Figure 2. (*a*) Infrared absorption spectrum of 3 ppmv of N_2O in dry N_2 (continuous line) and of air (containing *ca.* 0.3 ppmv N_2O) at 26 °C and 32% humidity (broken line). Path 60 m (redrawn from figure provided by courtesy of Siemens-Plessey Controls Limited; after Smith *et al.* 1994*a*).

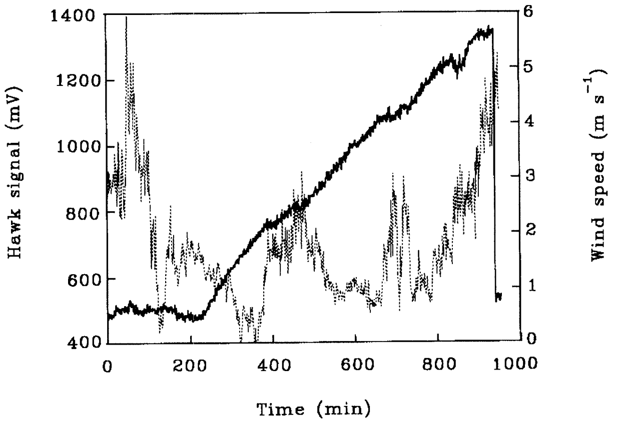

Figure 2. (*b*) N_2O accumulation in a large plastic chamber measured by Hawk long-path infrared spectrometer (continuous line), as affected by external wind speed (dotted line). Chamber closed after 230 min, opened after 930 min; cereal stubble site, Lammefjorden, Denmark, August 1993.

conserving grass for silage (McTaggart *et al.* 1994). This figure illustrates major differences in the pattern of emissions from different forms of nitrogen fertilizer. In figure 5*a*, it can be seen that there were large emissions of N_2O from plots receiving nitrogen as calcium nitrate in April, the first fertilization of the year. This is in contrast with the behaviour of the plots receiving urea in April (figure 5*b*), from which very low emissions of N_2O were observed. These effects contrasted with those observed following the 2nd and 3rd applications (in June and August), when the highest emissions were observed from plots receiving urea. The lowest emissions throughout were from plots receiving ammonium sulphate. These emission patterns, which were similar to those observed by Duxbury & McConnaughey (1986), suggest that denitrification was the predominant source of N_2O, in the relatively cool and wet conditions prevailing in April. Nitrification was likely to have been more important in the summer, but the differences be-

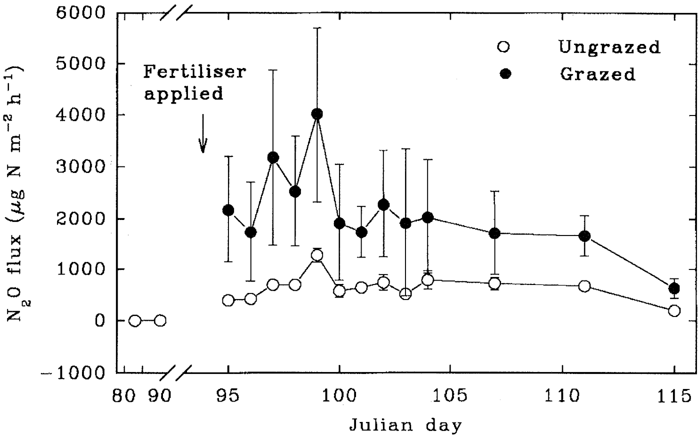

Figure 3. N_2O flux from ungrazed and grazed grassland, near Stirling, during the three weeks after fertilization; daily means and standard errors from 18 and 6 chambers, respectively (after Clayton *et al.* 1994).

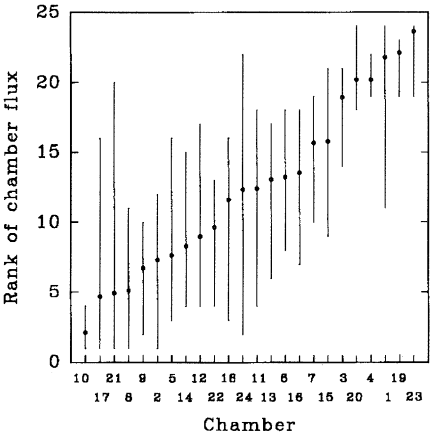

Figure 4. Rank order of 24 chambers with respect to N_2O fluxes from fertilized grassland measured over three-week period (15 measurement occasions). The bars show range and mean of rank for each chamber (Clayton *et al.* 1994).

tween urea and ammonium sulphate indicate that other factors are also involved. For example, the ammonium sulphate application also resulted in a significantly lower soil pH than that in the urea-treated plots, although a causal link between pH and emission rate has still to be established.

Figure 6 shows the impact of applying a nitrification inhibitor, dicyandiamide (DCD) with the nitrogen fertilizer. For both urea and ammonium sulphate, total emissions of N_2O over the season were roughly halved in the presence of DCD. It can also be seen in figure 6 that the total emissions from the 36 g N m^{-2} (360 kg N ha^{-1}) applied over the season ranged from only 50 mg N_2O–N m^{-2} (0.5 kg ha^{-1}) from ammonium sulphate plus DCD to about 500 mg N_2O–N m^{-2} (5 kg ha^{-1}) from unamended urea (i.e. from 0.14 to 1.4% of the fertilizer N applied). Emissions from nitrogen-fertilized cereal crops are generally lower than those from grassland, but here, too, the highest emissions have resulted from the

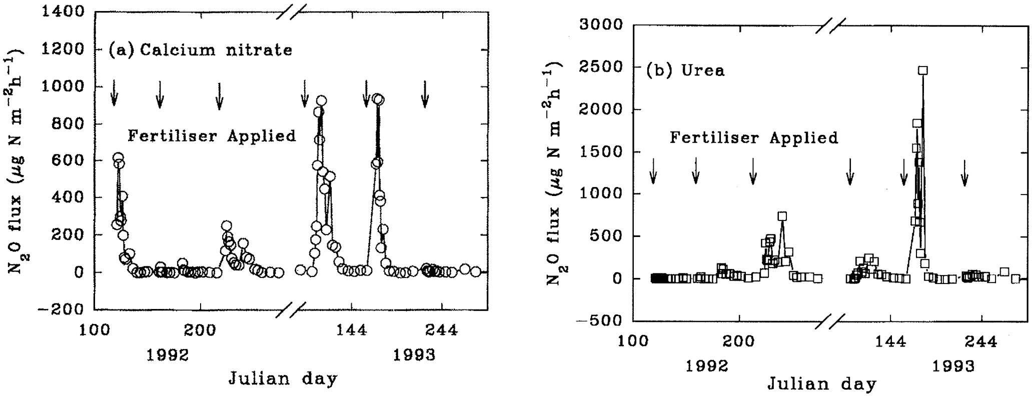

Figure 5. Daily N_2O–N emissions from fertilized grassland, near Edinburgh, over two growing seasons from April 1992. Fertilized (*a*) with calcium nitrate and (*b*) with urea.

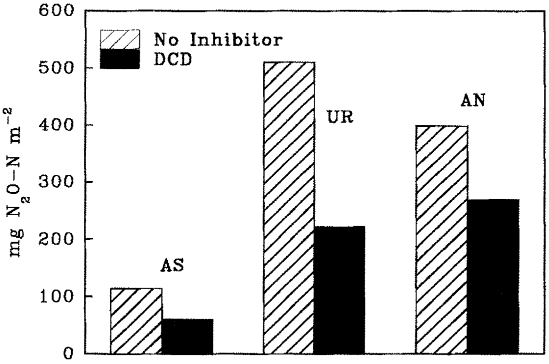

Figure 6. Total N_2O–N emissions from grassland with and without the nitrification inhibitor DCD; fertilized with 120 kg N ha^{-1} on three occasions during the 1993 growing season: AS, ammonium sulphate; UR, urea; AN, ammonium nitrate.

use of urea (McTaggart & Smith, unpublished data). As it is well known that in most circumstances crop yields are little affected by the chemical form in which the nitrogen is applied, these results are likely to be of considerable interest to those devising emission abatement strategies.

Several field experiments have compared the large plastic chamber/long-path infrared combination with small chambers and, at the other end of the scale, with micrometeorological measurements. At low windspeeds, it is enough to weight down the bottom of the plastic sheet with sandbags to obtain virtually linear increases in N_2O concentration in the large chambers with time, indicating negligible leakage. During windy periods, however, losses are significant (see figure 2*b*, above), but can be corrected for by a curve-fitting routine. Table 2 shows a comparison of results obtained by the three different methods for emissions from a cereal stubble field in Denmark in 1993. These results indicate that the different methods can yield results that are in reasonable agreement, provided that the area sampled by the chambers is representative of the micrometeorological fetch. Where this is not so, bias between the results can occur (Smith *et al.* 1994*a*).

The data from the Stirling site, summarized in figures 3 and 4 above, also showed that the N_2O flux from wet grassland was very sensitive to temperature.

Table 2. *Comparison of* N_2O *fluxes measured by small chambers, Hawk long-path infrared large chamber, and a micrometeorological (eddy correlation) method using tunable diode laser absorption spectrometry, all on the same cereal stubble site at Lammefjorden, Denmark*

date	method	flux ($\mu g\ N_2O\text{–}N\ m^{-2}\ h^{-1}$)
22 August	Hawk/large chamber	367
	small chambers (mean)	175
	eddy correlation	359
23/24 August	Hawk/large chamber	254
	small chambers (mean)	209
	eddy correlation	367
26/27 August	Hawk/large chamber	309
	small chambers (mean)	254
	eddy correlation	225

On one date, measurements were made from the chambers in both the morning and the afternoon, and the mean increase in flux in the afternoon was 91%, although the temperature had increased by less than 2 °C. This effect was also observed in experiments with a large chamber at the Edinburgh grassland site, and confirmed in another recent experiment. In the latter, small monoliths 15 cm deep were excavated from the Edinburgh site, using 40 cm flux chambers (see figure 1 above) as enclosures. These monoliths were fertilized with ammonium nitrate, watered to field capacity, brought to different temperatures, and the N_2O fluxes measured. The results (figure 7) show an exponential increase of flux with temperature, with a very high apparent Q_{10} value. One possible explanation for this is that increasing temperature increases the volume occupied by anaerobic microsites in which denitrification of nitrate to N_2O takes place, because of increasing respiratory consumption of oxygen in the soil (Tiedje *et al.* 1984). On this interpretation, the apparent Q_{10} would then be the product of the change in flux per unit volume of denitrifying soil and the change in the denitrifying volume.

Very different results have been obtained with fallow soil (sandy loam) in 1 m diameter monolith lysimeters located indoors in a greenhouse and fitted with automatic chambers (Thomson *et al.* 1994). Figure 8 shows the variation in N_2O flux caused by the diurnal variation in temperature when the soil in one of the lysimeters was maintained at about field capacity. Here the Q_{10} was only 1.6. It is evident that the relationship between N_2O emission and temperature is very complex, and further work will be necessary to achieve a full understanding of the processes involved.

4. Conclusions

Flux chamber methods for N_2O flux measurement have advantages over alternative methods in that they are robust, cheap and easy to operate, so that they are likely to be widely used for some time to come. This is in spite of their

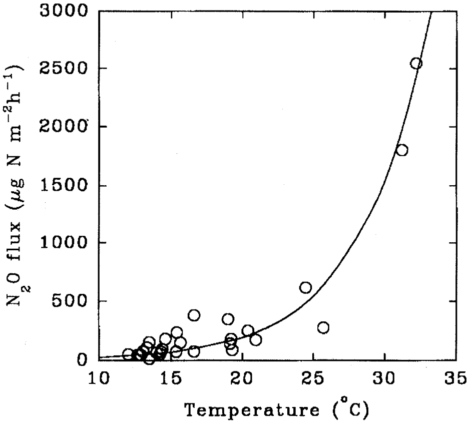

Figure 7. Relationship between soil temperature at a depth of 3 cm and N_2O flux from three replicate 40 cm diameter monoliths, all from a grassland site. Monoliths were rotated between three locations (outside, shaded; outside, in full sun; in greenhouse) to achieve the different temperatures. Regression: $y = e^{1.124+0.207x}$; $r^2 = 0.954$ ($Q_{10} \approx 8$). (Conen & Smith, unpublished data.)

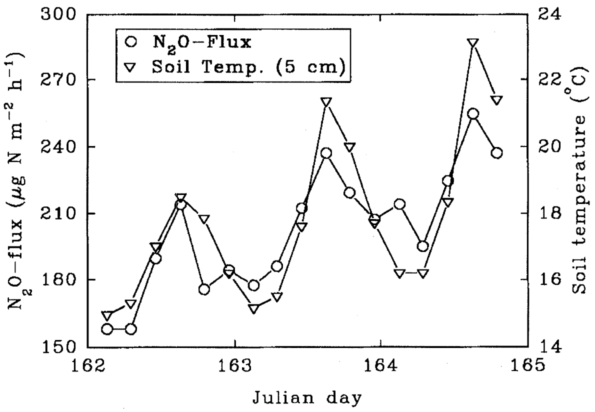

Figure 8. Relationship between N_2O flux and soil temperature at 5 cm depth in a lysimeter fertilized with 10 g N m^{-2} as calcium nitrate, June 1994. The lysimeter was installed in a greenhouse; the soil was a sandy loam.

disadvantages, which can include a high labour requirement, the need for a large number of replicate measurements to overcome spatial variability, and the possibility that the presence of a chamber may actually alter the flux by changing the environmental conditions.

Automatic systems are being introduced that allow intensive measurements to be made on a 24 h per day basis. This makes a major saving in labour, but the systems are costly and complex to construct and maintain. Furthermore, there is a need for a power supply, which precludes installation at some sites, and the spatial spread of chambers at a site is limited by lengths of cables and sampling lines. Individual battery operated chamber/sampler systems are being developed for use at dispersed or remote sites. Automatic systems in the laboratory allow intensive study of individual variables influencing fluxes, without the problems induced by the vagaries of the weather.

Large chambers 50–60 m^2 in area have some advantages. They can average fluxes over areas two to three orders of magnitude larger than conventional chambers; they are useful where site heterogeneity (e.g. the presence of plots with different treatments) rules out micrometeorological methods; and they can be used with a range of analytical techniques. However, the chambers are cumbersome to set up, and cannot be used in very windy conditions.

Chamber methods will probably continue to be useful to establish the effects of variables such as temperature, the supply of mineral nitrogen, and soil water content on N_2O fluxes. They may well be of particular value in ecosystems which are suspected of making a significant contribution to global emissions but which are in locations where site heterogeneity, difficulties of access or lack of infrastructure prevent the use of bulky and fragile micrometeorological equipment.

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Discussion

K. W. T. GOULDING (*Soil Science Department, Rothamsted Experimental Station, U.K.*). Dr Smith found a very large, sharp peak of N_2O production some time after the application of urea. He said that this N_2O could have been produced by nitrification of the NH_4^+ released by the urea, by denitrification of the resulting NO_3^-, or both. Such a sharp peak is very characteristic of denitrification. Is there any further data that would enable the source of the N_2O to be identified?

K. A. SMITH. There was relatively little emission of N_2O from urea in cool wet conditions in the spring, but much more from nitrate; under these conditions denitrification but not nitrification is favoured and we conclude that the former is the important mechanism. In contrast, in summer when temperatures are higher and the moisture content low, urea produces more N_2O than nitrate. These conditions are more conducive to nitrification, and less to denitrification, than conditions in the spring. This suggests that nitrification has become more important, but the experiment does not distinguish between N_2O emitted during nitrification and N_2O resulting from diffusion of the nitrite and nitrate produced by nitrification and anaerobic microsites where it can be denitrified. The emissions lasted for several weeks; the sharpness of the peaks in the figure is a consequence of the greatly compressed time axis to show two years' data. However, a very different factor influencing peak duration is the rapidly growing grass, which provides a strong sink for the mineral N and thus reduces the period when significant quantities are available for nitrification (or subsequent denitrification).

J. L. MONTEITH (*Institute of Terrestrial Ecology, Penicuik, U.K.*). Dr Smith emphasized the large variability of nitrous oxide emissions in the field which was suppressed by taking many samples. Has he been able to exploit the existence of variability to establish causal relations between flux, soil conditions, type of vegetation, etc.?

K. A. SMITH. It is clear that *temporal* variability is well correlated with soil conditions in some circumstances, e.g. N_2O fluxes are likely to be increased by increases in the concentration of mineral nitrogen (NH_4 or NO_3) present (such as occurs on fertilizer addition) or by wetting events – rainfall or irrigation.

However, the *spatial* variablity commonly observed in the field tends to make the identification of these relationships more difficult!

V. R. PHILLIPS (*Silsoe Research Institute, Bedford, U.K.*). Dr Neftel has reported that he had evidence that the emissions of nitrous oxide from livestock buildings might be far greater in total than the emissions from soils, and that he knows of no work in which the emissions from livestock buildings were being measured in detail.

The Silsoe Research Institute has recently begun a project, funded by the Ministry of Agriculture, Fisheries and Food, which measures emissions of nitrous oxide and methane from different types of livestock building and manure stores. The manure store work was in association with the Institute of Grassland and Environmental Research, North Wyke. It is too early yet to report figures on the Silsoe emission rate measurements.