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# Soil air concentration of N<sub>2</sub>O over 3 years of field experiments with animal manure and inorganic N-fertilizer

 $N_2$ O-indhold i jordluft ved 3 års markforsøg med anvendelse af husdyrgødning og uorganisk N-gødning

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### Summary

The concentration of  $N_2O$  in the soil air was investigated over a period of 3 years in a field experiment using animal manure as well as inorganic fertilizer. Variations in  $N_2O$  concentration are great and are mainly dependent on the soil nitrate content, climatic conditions and the availability of nitrogen and carbon in the animal manure.

In soil cropped with barley, periods with increased concentrations of  $N_2O$  (5–20 µl/l) have been seen in the spring and the early autumn while a low  $N_2O$  concentration was measured during the early summer (0.5–2 µl/l). Contrary to this, the  $N_2O$  concentration in soil cropped with beets reached levels of 20–120 µl/l during the summer decreasing to a minimum afterwards.

One of the years  $N_2O$  emission was measured 3 times. Values measured simultaneously for  $N_2O$  emission from the surface and for the concentration of  $N_2O$  in the soil air show that high levels of  $N_2O$  concentration are followed by simultaneous high levels of  $N_2O$  emission.

Key words: Nitrous oxide, nitrogen, denitrification, animal manure, inorganic N-fertilizer, soil air.

### Resumé

Jordluftens indhold af  $N_2O$  er målt gennem 3 år i markforsøg med anvendelse af husdyrgødning og handelsgødning. Variationerne i  $N_2O$ -indholdet er store, fortrinsvis bestemt af jordens nitratindhold, klimatiske forhold, samt tilgængeligheden af kulstof og kvælstof i husdyrgødningen.

Med byg som afgrøde er perioder med høje  $N_2O$ -indhold (5–20 µl/l) koncentreret om forår og tidligt efterår, mens der findes et lavt niveau for  $N_2O$ -koncentration i jordluften (0,5–2 µl/l) gennem forsommeren. I modsætning hertil er der i jordluft under afgrøde med roer i samme periode fundet et højt niveau (20–120 µl/l), mens indholdet igen falder til et minimum fra sensommer og i de første efterårsmåneder.

Overfladeafgivelse af  $N_2O$  er i et af årene blevet målt 3 gange. Disse målinger sammenholdt med målinger af jordluftens  $N_2O$ -koncentration viser, at høje niveauer for  $N_2O$ -koncentration samtidig giver høje niveauer for  $N_2O$ -afgivelse.

Nøgleord: Dinitrogenoxid, kvælstoftab, denitrifikation, husdyrgødning, handelsgødning, jordluft.

### Introduction

Nitrogen fertilizer supplied in agriculture is not fully utilized by crops. The added inorganic nitrogen may be immobilized in the soil, lost by leaching or transformed to gaseous nitrogen compounds (denitrification). In case of animal manure (slurry or manure), a great part of its content of nitrogen is in organic form and should be mineralized before it is available to the crops.

In lysimeter experiments with added <sup>15</sup>N-labelled nitrogen, it has been found that 14 to 19% of added nitrogen fertilizer could not be recovered in the crop, in the leaching water or in the soil after 3 years of cultivation. This part of the added nitrogen is assumed to be lost by transformation to gaseous forms (8).

The gaseous forms of nitrogen produced from nitrogen fertilizer are mainly N2 and N2O. A great number of microorganisms in the soil produce these gases by reduction of nitrate and some of them by oxidation of ammonium. The reduction of nitrate also called denitrification is an anaerobic respiration of nitrate via N2O as an intermediate to N2 as the end product. The nitrification process, which normally is an autotrophic, aerobic process can form N2O at bad aeration conditions (13). A lowering of the oxygen content in soil therefore may result in an increased formation of gaseous nitrogen originating in both processes. A soil with a high water content, sufficient inorganic nitrogen, and - for the denitrification process - an easily available carbon source, therefore will stimulate the microbial processes resulting in N<sub>2</sub>O and N<sub>2</sub>.

The content of N<sub>2</sub>O in the ambient atmosphere is low (0.3  $\mu$ l/l) for which reason a formation of N<sub>2</sub>O can be measured, either as the content of N<sub>2</sub>O in the soil air (2, 7), or as emission of N<sub>2</sub>O from soil surface (10, 14).

The content of  $N_2$  in the ambient atmosphere is 80% and makes a measurement of this product directly in the field impossible. Several methods for quantification of formation of  $N_2$  has been suggested. Using <sup>15</sup>N-tagged nitrogen, the <sup>15</sup>N enrichment of the  $N_2$  produced can be measured (13). This method has unfortunately a detection limit of 0.1–0.2 kg N/ha/day. Another method is based on the fact that acetylene inhibit the last step in the denitrification process, the transformation of N<sub>2</sub>O to N<sub>2</sub> (1). *Ryden et al.* (12) have described a method based on addition of acetylene to the soil, and measuring the accumulation of N<sub>2</sub>O in a chamber placed on the soil surface. A third method is incubation experiments in the laboratory in an N<sub>2</sub>-free atmosphere and gas chromatographic measurement of both gaseous products (9).

This paper decribes running measurements of the soil air content of  $N_2O$  during a three-year period in a field experiment with animal manure and inorganic fertilizer. The concentration measurements have been supplemented with a few measurements of surface emission of  $N_2O$ . Results should be regarded as the first stage of the





efforts to estimate the total loss of nitrogen to  $N_2O$  and  $N_2$  from the cultivated soil and its variation with the time of year and cultivation conditions.

### Sampling and measuring techniques

For sampling the soil atmosphere probes are permanently placed in the experimental plots at a depth of 15 cm during the growing season. 4 replicates are placed in each plot. In Fig. 1 the probe is shown together with the sampling pipette also used for the transport of the sample (by mail). The pipette is flushed with soil air from the air chamber in the bottom of the probe by a syringe on the top, and the air sampled is enclosed in the pipette between the 2 stopcocks.

After reaching the laboratory, the samples are transferred to gas chromatographic analysis via a lock. A part of the pipette content is transferred to the lock, and an adjustment of the vacuum to atmospheric pressure is made by supplying helium. A 1 ml sample is withdrawn with a gastight syringe.

The gas analyses for N<sub>2</sub>O have been performed with a Hewlett-Packard 5840 gas chromatograph with a Ni<sup>63</sup>-EC-detector. The column was a Poropak Q ( $6.4 \text{ mm} \times 2.0 \text{ m}$ ) at a temperature of 40°C, detector temperature 350°C, carrier gas flow 20 ml/min.

The emission of  $N_2O$  from the soil surface is based on a steady stream of ambient atmosphere passing through a perspex chamber placed on a frame in the upper 15 cm of the soil. The content of  $N_2O$  in the air from the chamber is measured on a gas chromatograph and sampled once an hour over a 24 hour period. The increased content of nitrous oxide in the air stream from the chamber related to the content in the ambient atmosphere will originate in the soil. This content of  $N_2O$  is combined with the air flow rate and the chamber area on the soil surface to calculate the emission of  $N_2O$  as an amount of  $N_2O$ -N per area per unit time. Details of the method are described by (4).

The water potential of the soil was registered with tensiometers (3) at the same depth, 15 cm.

Air temperature data have been obtained from a climatic station placed on the experimental area.

### Area for experiments and fertilizer treatments

The investigations were running at Askov Research Station in South Jutland. Measurements have been made in plots treated with fertilizer and with manure and cow slurry during several years. Soil texture are given in Table 1.

Teksturanalyse								
% clay	% silt	% fine sand	% coarse sand	% humus				
10.9	11.5	35.4	37.7	4.6				

Table 1. Particle size distribution

The concentration of  $N_2O$  in the soil air at depth of 15 cm has been measured regularly for 3 years most frequently during the growing season from spring to harvest. The measurements have been made in field plots treated with different quantities of animal manure, and inorganic N in NPK-fertilizer at two N-levels. The quantity and times of adding the inorganic fertilizer and the animal manure are shown in Table 2. In the first and third year the area was cropped with barley, while beets were grown in the area in the second year. Only 2 levels of slurry (25 and 100 t/ha) were measured in 1980.

### Results

### The $N_2O$ concentration

 $N_2O$  concentrations in soil air are shown in Fig. 2, 3 and 4 together with the air temperature and the soil water potential.

In 1980 the  $N_2O$  content decreased in the end of May to nearly atmospheric level in June and July with an increase to a maximum for this year (2–18  $\mu$ l/l  $N_2O$ ) in August, Fig. 2.

After the winter 1980-81 where low amounts were observed (measured in December), the concentration increased during 1981 to a maximum level in June-July, and decreased to low concentrations from August, i.e. an entirely different trend compared to the first year (Fig. 3).

Year		1980	1981	1982
Crop		barley	beets	barley
Cow slurry/manure added		Mar. 80	Dec. 80	Feb. 82
	at 25 t/ha slurry	62	42	47
The second secon	at 100 t/ha slurry	248	168	188
Inorganic N content kg/ha	at 400 t/ha slurry*	-	672	-
	at 100 t/ha manure	-	116	161
	at 25 t/ha slurry	40	20	35
O	at 100 t/ha slurry	160	80	140
Organic N content kg/na	at 400 t/ha slurry*	-	320	-
	at 100 t/ha manure	-	289	326
Chalked NH <sub>4</sub> NO <sub>3</sub> added		Apr. 80	Apr. 81	Apr. 82
Inorganic N content kg/ha	at 1 N	- 40	80	40
Inorganic N content kg/ha	at 4 N	160	320	160

### **Table 2.** Cropping and nitrogen amendment.Afgrøde og kvælstoftilførsel

\*) The 400 t/ha slurry is added every fourth year (Dec. 80).



Fig. 2. Air temperature, soil moisture tension, and  $N_2O$  concentration in soil air at a 15 cm depth during 1980. The crop was *spring barley*.

Lufttemperatur, vandmætningsgrad og  $N_2O$ -koncentration i jordluften i 15 cm dybde i 1980. Afgrøde: vårbyg. During the winter 1981-82 high values were measured (in November 81 and February 82).

In 1982 the seasonal variation was the same as in 1980: minimum concentration in June-July compared to the concentration levels in May and August.

### Climatic conditions during the experimental period

1980 was very wet with 414 mm precipitation from May to August, when the normal average is 261 mm. 1981 was also rather wet with 334 mm, while 1982 was dryer than normal with a precipitation of only 236 mm over the same period.

### Soil inorganic nitrogen

The amounts of  $NH_4^+$  and  $NO_3^-$  in the top soil layer are shown in Table 3. The content of  $NH_4^+$  varies very little between treatments, and during the measuring periods. The level is 1-5 mg  $NH_4^+$ -N/kg soil, with a few exceptions.

The  $NO_3^-$  content showed more pronounced variations with the type of fertilizer, fertilizing time as well as the crop uptake of nitrogen. After the slurry application 3 May 1980 an increased content of  $NO_3^-$  was found at the end of the month.

Applying manure and slurry 3 December 1980 did not result in an increased content of  $NO_3^-$  from the sampling 11 December 1980, but in





Lufttemperatur, vandmætningsgrad og N<sub>2</sub>O-koncentration i jordluften i 15 cm dybde i 1981. N-mængderne 1 N og 4 N er angivet i tabel 2. Afgrøde:  $\tau o e \tau$ .

June 1981 an increased nitrate content is found probably due to mineralization and nitrification of the organic nitrogen in the manure and slurry during the spring. In April 1982 an increased nitrate content was also found after application of organic fertilizer 2 months earlier.

In 1980 when the area was cropped with barley, the soil content of nitrate decreased from the

spring to a very low level in the midsummer, but increased again during the period of ripening.

The beets grown in 1981 did not exhaust the soil of nitrate during the early summer as barley did, but the soil content of nitrate was kept at a lower level during the autumn months compared to the barley.

	Fertilizer			Slurry					Ma	Manure			
Treatment	1 N		4 N		25	25 t/ha		100 t/ha		400 t/ha*		100 t/ha	
Date	NH₄+	NO <sub>3</sub>	$NH_4^+$	NO <sub>3</sub>	NH₄	NO <sub>3</sub>	$NH_4^+$	NO <sub>3</sub>	$NH_4^+$	NO <sub>3</sub>	NH₄+	NO <sub>3</sub>	
1980							_						
27 May	-	-	-	-	3.3	3.8	2.8	5.6	_	-	_	-	
7 July	-	_	-	-	3.9	0.3	3.6	0.8	-	-	-	-	
15 Aug.	-	-	_	-	3.3	0.8	3.1	3.6	-	-	_	-	
25 Sept.	-	-	_	-	4.7	1.6	5.1	5.0	-	-	-	-	
11 Dec.	3.0	0.7	2.0	1.1	2.4	1.4	4.5	1.2	-	-	-	-	
1981													
10 June	1.6	38	2.5	101	2.0	13	1.5	15	1.9	17.6	4.3	12.6	
25 June	2.5	0.7	2.8	2.2	2.9	1.5	4.1	2.7	2.8	1.0	4.2	1.2	
13 Oct.	1.8	1.5	1.3	1.4	2.0	1.3	2.1	1.3	2.6	1.5	8.0	4.4	
19 Nov.	1.4	1.6	1.2	1.3	1.4	1.8	1.2	2.2	1.5	1.7	2.9	2.8	
1982													
15 Feb.	2.3	2.7	2.7	1.6	3.4	1.9	2.3	2.8	3.2	1.5	4.8	2.7	
16 Apr.	2.1	4.8	2.2	4.7	2.5	5.5	2.5	191	2.3	5.9	12.4	94.7	
14 May	3.6	19	10	32	2.6	4.0	3.6	27	2.1	1.5	5.0	33.7	
9 Sept.	1.9	1.3	1.2	2.8	1.6	2.1	2.1	4.4	3.2	1.5	6.0	6.0	
18 Nov.		-	0.9	3.0	1.6	3.2	1.6	3.6	3.2	2.7	12.1	25.4	

# **Table 3.** Soil inorganic nitrogen in 0–20 cm depth (mg N/kg soil)The treatments are explained in Table 1.Jordens indhold af uorganisk N i 0–20 cm dybde (mg N/kg jord)Behandlingerne er forklaret i tabel 1.

\*) The 400 t/ha slurry is only given every fourth year (Dec. 1980).

### $N_2O$ emission from the soil surface

The N<sub>2</sub>O emission from the soil surface was measured 3 times in 4 of the treatments: 25 and 100 t slurry/ha and the fertilizer levels 1 N and 4 N. The results are shown in Table 4 with the corresponding measurements of the soil air. The emission of N<sub>2</sub>O was generally greatest for all treatments in the spring and with lower values in the summer and autumn. emission from surface and for the concentration of  $N_2O$  in soil air show that high levels of  $N_2O$ concentration are followed by simultaneous high levels for the  $N_2O$  emission. This is also described by (5).

### Discussion

In May 1980, when increased content of  $N_2O$  was found in the soil air in the slurry treated plots, the air temperature was 10°C and the water tension

The values measured simultaneously for N<sub>2</sub>O

Table 4. N<sub>2</sub>O-flux from soil surface (g N/ha/day) and N<sub>2</sub>O content in soil air (μl N<sub>2</sub>O/l). 1982. N<sub>2</sub>O overfladeafgivelse (g N/ha/døgn) og N<sub>2</sub>O indhold i jordluft (μl N<sub>2</sub>O/l). 1982.

Date	40 kg N/ha		160 kg N/ha		25 t sl	urry/ha	100 t slurry/ha	
	N <sub>2</sub> O-flux	$N_2O$ -conc.	N <sub>2</sub> O-flux	N <sub>2</sub> O-conc.	N <sub>2</sub> O-flux	$N_2O$ -conc.	N <sub>2</sub> O-flux	$N_2O$ -conc.
12 May	38	1.5	76	8.0	69	12	779	16
30 June	4.7	2.3	5.2	2.2	1.6	2.1	34	1.7
8 Sept.	0	0.7	3.1	0.9	3.5	0.8	10	1.4



Fig. 4. Air temperature, soil moisture tension, and N<sub>2</sub>O concentration in soil air at a 15 cm depth during 1982. The fertilizer amounts 1 N and 4 N are explained in Table 2. The crop was *spring barley*.

Lufttemperatur, vandmætningsgrad og N<sub>2</sub>O-koncentration i jordluften i 15 cm dybde i 1982. N-mængderne 1 N og 4 N er angivet i tabel 2. Afgrøde: vårbyg.

70 cBar (Fig. 2). The decrease in  $N_2O$  concentration during June and July cannot be explained from the above mentioned parameters, since the soil tension decreased from 70 to 0-10 cBar, and the soil temperature increased in the period when  $N_2O$  concentration decreased between the sampling in May and the sampling in June-July. The increase in the  $N_2O$  content in August-September is also unaccountable, solely by the soil moisture and the soil temperature, since these parameters did not change appreciably in this period.

However, the fluctuations in the soil air content of  $N_2O$  during the growing season can be related to the variations in the availability of nitrate. The nitrate is supplied via mineralization and nitrification of the organic nitrogen in the slurry and also directly from fertilizer applied. The mineralization probably proceeds relatively uniformly during the entire growing season. When barley is actively growing it exhausts the inorganic nitrogen pool in the soil, as seen from the soil content of  $NO_3^-$  (Table 3).

The major uptake of nitrate by the barley takes place in a 2 month period after (Table 3, and Fig. 2 and 4) the sprouting (11), i.e. in June and July. The low levels of  $NO_3^-$  and  $N_2O$  in this period make it probable that the microbial  $N_2O$  formation, i.e. the denitrification has been limited by the lack of nitrate. In the autumn 1980 an increased content of  $NO_3^-$  and  $N_2O$  was found, starting in August, when the barley ripened and therefore discontinued nutrient uptake. The decreasing temperature during October reduced the microbial activity resulting in a low  $N_2O$  content until spring 1981.

During the growth period 1981 the N<sub>2</sub>O content increased in May, when the soil temperature is increased to 5-10°C. As apposed to 1980 with a barley crop, the N<sub>2</sub>O concentration reaches a maximum in June-July followed by a decrease during August-September. This decrease took place in a period with high temperature. The reason for this must be that the nutrient uptake by the beet crop, starts substantially later than barley (C. Marcussen, pers. com.). Therefore, the competition between the microorganisms and the crop for the nitrate starts much later in fields cropped with beets than fields cropped with barley.

In the winter 1981-82, a few measurements showed remarkably high contents of  $N_2O$ . In spite of a temperature of 5-10°C below the autumn temperature in 1981, higher  $N_2O$  concentrations were measured. This is probably caused by the water status of the soil. During winter the soil is water saturated resulting in a very limited air exchange. Even a very small  $N_2O$  production will cause an accumulation of  $N_2O$ .

In 1982 the area was cropped with barley as in 1980. The precipitation in 1982 was substantially

less, compared to 1980. In spite of this, the content of  $N_2O$  was approximately of the same magnitude for the 2 slurry levels and showed the same seasonal fluctuations in these 2 years with barley. It is therefore concluded that a substantial part of the differences in the seasonal fluctuations in the soil air content of  $N_2O$  can be explained by the crop and its influence upon the amount of nitrate.

The seasonal fluctuations during 1981 and 1982 in the soil air content of  $N_2O$  with addition of 400 t slurry/ha, 100 t manure/ha and the fertilizer amounts 1 N and 4 N (Fig. 3 and 4) are all corresponding to the fluctuations described for addition of 25 and 100 t slurry/ha.

The fluctuations in the soil air content of  $N_2O$ during the growing season can primarily be used for a qualitative evaluation of the  $N_2O$  production. A semiquantitative evaluation of the denitrification in different soil amendment is found by comparing the  $N_2O$  content in the amendments on the same measurements dates. The water content has been the same in different amendments, and therefore the soil air exchange may be similar.

### Influence of the slurry treatments

From Fig. 2 it appears that 25 t slurry/ha give a lower content of  $N_2O$  in the soil air than 100 t slurry/ha in 1980. During 1981-82 the 100 t slurry/ha usually gave the highest values for  $N_2O$  concentration apart from 2 short periods in the summer 1981 and the winter 1981-82.

The use of 400 t slurry/ha in the winter 1980-81 did not give a higher content of  $N_2O$  than 25 and 100 t did (Fig. 1 and 2). Summarizing, the  $N_2O$  production varies only slightly with the slurry amounts. The manure applied in 1981 resulted in a  $N_2O$  content in the soil air at the same level as the 400 t slurry/ha (Fig. 3), while very high levels of  $N_2O$  was found in 1980 in the months following the manure application (Fig. 4).

The two inorganic fertilizer levels resulted in  $N_2O$  contents at the same level, and only slightly lower than the 100 t slurry/ha amendment in 1981 and 1982 resp. (Fig. 4). The organic matter in the slurry did not therefore generally stimulate the

 $N_2O$  production considerably beyond that level originating in the nitrate available.

## $N_2O$ content of the soil air related to the $N_2O$ production

Transport of a gas in soil occurs mainly by diffusion in the soil air (6) directed against parts with a lower concentration of the gas. Diffusion of gases in the soil solution is normally of the order of magnitude 3-4 times slower than in the soil air. In the field, where the pore volume will be more water filled with increasing depth, the transport of a gas will predominantly be from the site of production and towards the soil surface. The emission of  $N_2O$  from the soil surface will therefore normally be a good estimate of the production of  $N_2O$  in the top soil layer (0-30 cm), where the production mainly occurs. The concentration of  $N_2O$  in the soil air is, however, a result of the production of N<sub>2</sub>O in the soil as well as the velocity of the N<sub>2</sub>O transport in the soil air.

If the concentration of  $N_2O$  is related to the  $N_2O$  emission, it is possible to calculate the residence time for  $N_2O$  in the soil air. Hereby an estimate for duration of the period representative of the measurements can be obtained:

It is assumed that the  $N_2O$  concentration measured at 15 cm depth represents the  $N_2O$  content in 0-30 cm soil layer. With an air filled pore fraction of 25%, the values for  $N_2O$  emission stated in Table 4 indicate that the turn-over time varies from ¼ hour to 12 hours with an average of 2 hours for the  $N_2O$  content in 0-30 cm depth. The rather short turn-over time for  $N_2O$  indicate that the concentration of  $N_2O$  in the soil air qualitatively represents the  $N_2O$  actually produced in the soil.

### Conclusion

Measurements of the concentration of one of the denitrification products,  $N_2O$ , in soil air over a 3 year period have given us information on how cultivation methods influence the denitrification. Organic fertilizer may temporarily increase the  $N_2O$  concentration in soil air, but an important factor in the soil is the nitrate status of the soil,

and therefore the crop and its time for nitrate uptake greatly influences the denitrification and its seasonal variation.

Some simultaneous measurements of  $N_2O$  flux have shown that high surface losses of  $N_2O$  are generally combined with high concentrations of  $N_2O$  in the soil air. Registrations of the concentration of  $N_2O$  in the soil air, therefore, could be used to find the periods when denitrification occurs, and for a semiquantitative estimation of the influence of different cropping and amendments on denitrification. The equipment for sampling in the field, and the analysis for  $N_2O$  in the laboratory is simple. The method is therefore suitable for investigations on several areas, even placed in great distances from each other, as the sample can be mailed.

However, it is only possible to measure one of the products,  $N_2O$ , which means a limitation of the usability of the measuring results. With more knowledge of field emission of  $N_2O$  combined with experiments in the laboratory, especially the variations in the  $N_2O:N_2$  ratio (5), these  $N_2O$  concentration measurements can be used to evaluate the denitrification and its variation under many other cultivation conditions.

#### Litteratur

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