Recovery of Fertilizer Nitrogen Under Field Conditions Using Nitrogen-15

J. N. Carter, O. L. Bennett, and R. W. Pearson

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ABSTRACT

An experiment was conducted under field conditions to study the movement and recovery of N^{15}-enriched nitrogen fertilizer added to soil in the forms of $NaNO_3$ and $(NH_4)_2SO_4$. The plots consisted of the soil mass inside cylinders 30 or 60 cm in diameter, which were pressed into the soil to a depth of 45–60 cm. These plots were either seeded to sudangrass (*sorghum sudanense*) or left uncropped. The plant tops, roots, and the soil, by 15-cm layers, were analyzed for total N and excess N^{15} content.

Recovery of added N^{15} from the 32 plots ranged between 96.3 and 101.8%, and averaged 99.0%. There was no significant difference in recovery of the fertilizer N due to the percent of excess N^{15} in the fertilizer, size of plot, exposure to natural rainfall, cropping, N source, or the time of application of the fertilizer. Wide variation in recovery of excess N^{15} occurred with core sampling and it was found necessary to remove, weigh, mix and subsample the entire soil mass from within the rims. The small amounts of unrecovered N were probably due to procedural errors in the field and laboratory, and leaching and root growth beyond the sampling zone. There was no indication of any N loss in the gaseous form.

The fate of applied fertilizer N was followed under field conditions using N^{15}-enriched nitrogen fertilizer applied to the soil *in situ* enclosed by large steel cylinders. Total recovery of the fertilizer N from these experiments after 8 weeks ranged from 88 to 96%, but dropped as low as 77% after 10 months. Recovery of the fertilizer N was greater from an ammonium source than from nitrate. Better recovery of fertilizer was obtained when the plots were cropped to sudangrass rather than left fallow. Doubling the rate of application of the fertilizer N from 336 to 672 kg/ha did not affect percent of total recovery. The 4 to 12% of fertilizer N unrecovered after 8 weeks appears to be due to a loss in the gaseous form, since special precautions were taken to restrict leaching and to eliminate errors in sampling and analysis.

MANY LABORATORY and greenhouse studies have shown that gaseous losses of N from soils may occur under a variety of conditions, the amount lost depending mostly upon the form of N added, pH, moisture content of the soil, and degree of aeration (2, 3, 4, 5, 8, 13). Under field conditions it is known

that the recovery of added fertilizer N by the fertilized crop is seldom more than 50% (10, 12). Part of the remaining N is recovered by subsequent crops (10), part is probably lost through leaching beyond the root zone, some may be incorporated in soil organic matter, and presumably the rest escapes as gasses. In lysimeter studies where the amount of N removed through leaching can be measured, the average gaseous losses of N in various experiments have been estimated to be about 15% (1, 7, 9). Gaseous losses of N under field conditions in undisturbed soil have not been accurately determined, chiefly because of the experimental difficulties. It has not been feasible to collect and measure the evolved gasses directly. It is also difficult to measure leaching losses, except in lysimeters. Furthermore, analyses for changes in total soil N have been unsatisfactory because of the difficulty of sampling, and also because ordinary chemical methods for determining total N are not sufficiently accurate to measure the small losses in the presence of the relatively large quantities of soil N.

Through the use of N^{15} tagging it becomes possible to measure, with satisfactory accuracy, the amount of fertilizer N remaining in the soil or plant and hence to estimate fertilizer losses. The method, of course, does not help in measuring total gaseous loss because it does not measure loss of untagged soil N. Also, the isotope is too expensive for use on large field plots or at high enrichment levels. Much can be learned, however, from small plot experiments about the influence of soil factors on gaseous loss, movement in soil, and plant uptake of mineralized soil nitrogen in soil if accurate techniques are available for fertilizer salts labeled at low enrichment levels.
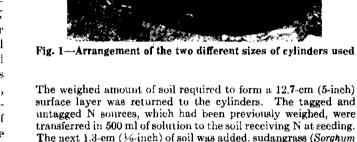
The purpose of the first phase of this study was to develop procedures that accurately measure the fertilizer N in the soil, and thus permit reliable estimates of gaseous loss, at a reasonable cost of N^{15}. The objective of the second phase was to utilize such procedures in the evaluation of soil and fertilizer factors on N loss by volatilization under field conditions.

PART I   PROCEDURE STUDY
Materials and Methods

The field experiment, made up of 32 plots, was conducted at Thorsby, Alabama, on a gently sloping, well-drained Greenville fine sandy loam which had an initial pH of 5.9 and an N content of 0.045%. The experiment involved 16 randomized treatments replicated twice.

The entire experimental area was first treated with 2,240 kg (1 ton/acre) of ground limestone and 1,120 kg (1,000 lb/acre) of 0-14-14 fertilizer per hectare. After these materials had been disked into the soil, metal cylinders, used for confining the area to be fertilized with N^{15}, were pressed into the ground with little soil disturbance. Two different sizes and arrangements of cylinders were used, as shown in Fig. 1. In the first, the cylinders, which were 60 cm (24 inches) in diameter and 60 cm long, were pressed into the ground. The N^{15} source was applied to the entire area of the cylinder. In the second arrangement, a similar cylinder, 60 cm in length and diameter, was pressed into the soil around another one 30 cm (12 inches) in diameter and 45 cm (18 inches) long. In these double-ring tests, the tagged N source was applied to the soil in the center cylinder only. The outer ring of soil in the double-ring units was fertilized with nontagged N at the same rate in all cases.

In order to get a uniform surface soil in the various cylinders, the top 15 cm (6 inches) of soil was removed from all cylinders, combined, passed through a 0.64-cm (1/4-inch) screen, and mixed.

Fig. 1—Arrangement of the two different sizes of cylinders used

The weighed amount of soil required to form a 12.7-cm (5-inch) surface layer was returned to the cylinders. The tagged and untagged N sources, which had been previously weighed, were transferred in 500 ml of solution to the soil receiving N at seeding. The next 1.3-cm (1/2-inch) of soil was added, sudangrass (Sorghum sudanense) was seeded where required, and then covered with the final 1.3-cm of soil. Nitrogen applied during growth of the crop was added as a solution to the soil surface.

Sodium nitrate and ammonium sulfate, tagged at the 8 and 16% excess N^{15} levels, were applied to both cropped and fallow soils. The N sources were applied either at seeding time or when the plants were 20 to 25 cm (8 to 10 inches) high, which was 24 days after planting. The rate of application in all cases was 280 kg (250 lb/acre) of N per ha.

Leaching was restricted in all except four of the plots by protecting the cylinders with a movable plastic cover suspended several feet above the soil surface. This cover was placed over the plots only at infrequent intervals for short periods of time when heavy rains were expected. Water was added by irrigation in quantities adequate for good crop growth, but not sufficient to cause a movement of N below the root zone. The amount of water required was determined by sampling extra plots provided for the purpose. The moisture level of the fallow and cropped plots was kept approximately the same.

The plants were harvested at 8 weeks when they were just beginning to flower. They were cut at ground level and dried at 65C for 72 hr, weighed, ground to 20-mesh size in a Wiley Mill, and sampled.

The soil in the cylinders was sampled both by core-sampling and by removing the soil by layers, mixing, and subsampling. The soil in the 30-cm cylinders was removed and sampled by 15-cm increments down to the 45-cm level. The 45- to 60-cm layer was core-sampled by taking three equally spaced 1.9-cm (3/4-inch) cores. In the case of the 30-cm cylinder exposed to natural rainfall, one additional core sampling was taken at the 60- to 75-cm depth. The 60-cm cylinders were sampled to a depth of 15-cm by taking 7 equally spaced 1.9-cm core samples, then removing the remaining soil in the 15-cm layer and mixing for an additional sample. This sampling procedure was then followed for the 15- to 30-cm and 30- to 45-cm layers. Seven equally spaced 1.9-cm core samples only were taken of the 45- to 60-cm layers, because very small amounts of the N^{15} were expected to be

Table 1—The effect of soil pH, drying temperature, and time of sampling on the recovery of sodium nitrate and ammonium sulfate fertilizer N[15] from soil

Soil pH	Fertilizer treatment	Duration of incubation	N[15] recovered				
			Drying temperatures				
			30C	50C	65C	80C	95C
			%				
4.5-5.0	NaNO₃	0	97.1	97.6	98.3	98.5	96.1
		8 weeks	97.4	96.4	95.6	96.9	95.6
	(NH₄)₂SO₄	0	101.2	101.8	99.4	99.0	97.9
		8 weeks	100.2	101.8	100.6	100.3	98.7
5.5-6.0	NaNO₃	0	96.5	97.2	98.6	98.2	97.7
		8 weeks	94.9	94.9	96.1	94.4	93.6
	(NH₄)₂SO₄	0	100.5	97.8	95.2	95.1	92.4
		8 weeks	98.0	97.9	98.3	97.5	93.3
6.5-7.0	NaNO₃	0	98.8	99.4	98.9	98.6	100.0
		8 weeks	96.8	97.9	97.2	98.9	97.5
	(NH₄)₂SO₄	0	99.3	92.5	89.9	90.1	85.7
		8 weeks	97.4	96.5	98.1	97.9	95.5
7.5-8.0	NaNO₃	0	97.9	99.0	99.5	99.0	99.9
		8 weeks	97.2	97.7	97.1	98.1	97.6
	(NH₄)₂SO₄	0	85.0	74.9	70.7	69.1	64.8
		8 weeks	94.6	94.2	92.7	95.7	94.8

present. Plant samples were taken between the two cylinders of the two-ring plots, but soil samples were not taken.

Each soil layer, weighing between 13.6 and 84.4 kg (30 and 186 lb), depending on cylinder size, was passed through a 0.64-cm screen, mixed, and approximately 2.3-kg (5-lb) subsamples taken. Each subsample was then dried, using a maximum heat of 65C. One-half of this subsample was ground to a fine powder in a pulverizer equipped with ceramic plates, and a final subsample of 114 g (4 ounces) taken for analysis. The roots were removed only from the top 15-cm layer of soil. Samples for soil moisture determination were weighed and dried to a constant weight at 105C.

Analyses for total nitrogen and for N[15] content were made on both plant and soil samples. Total nitrogen was determined by the macro-Kjeldahl method, modified to include nitrates by the use of salicylic acid. The presence of nitrites was not found in any of the samples.

In the Kjeldahl procedure, 40 ml of H_2SO_4, containing 114 g of salicylic acid per 4.1 kg (9 lb) of acid, was added to the dry soil or plant material. The sample and acid mixture was then gently shaken until thoroughly mixed. The mixture was allowed to sit for a period of 12 to 14 hours before continuing. Five grams of $Na_2S_2O_3 \cdot 5H_2O$ was then added to the sample, shaken, let stand for 5 min, and then heated for 10 min. Following cooling of the sample, 10 g of anhydrous Na_2SO_4 and 1 piece of no. 16 copper wire 1.9-cm long were added and then the sample was

Table 2—Effect of sampling technique on the recovery of fertilizer N[15] in the crop and soil

Treatment*	N[15] recovered	
N source	Complete sampling	Core sampling
	%	%
At Seeding		
NaNO₃	100.9	116.2
	98.5	111.0
(NH₄)₂SO₄	101.0	109.2
	98.4	118.0
To Growing Plants		
NaNO₃	100.9	117.2
	100.1	106.2
(NH₄)₂SO₄	100.9	137.2
	101.1	86.4

* All plots sampled were 60-cm in diameter, cropped to sudangrass, received 8% excess N[15], and were covered.

digested for a period of 2 hours. Following cooling of the sample, 350 ml of distilled water was added.

The ammonia was distilled into 4% boric acid solution using a specially designed connecting bulb that gives an additional distillation of the ammonia and prevents any NaOH from contaminating the distillate. The samples were titrated, using $0.1N$ H_2SO_4 with a microburette and methyl red–methylene blue as an indicator.

Duplicate samples were then combined and redistilled into a small excess of H_2SO_4. The excess water in the samples was evaporated and the N[15] content determined with a Consolidated Electrodynamics Corporation Mass Spectrometer, Model 21–620, equipped with a glass inlet sample system (11). (Mention in this publication of commercially manufactured equipment does not imply endorsement by the USDA over similar equipment not mentioned.)

The amount of excess N[15] recovered was determined from the product of the dry weight of the soil or plant material, percent N in this material, and the percent excess N[15] in the distillate of the total N determination. The N[15] was considered to be excess in the soil and plant material if the values obtained from the mass spectrometer analysis were above those received on untreated samples taken from the same general area. The amount of excess N[15] in the plant, and the various soil layers were added together to give total excess N[15] recovery. The average weight of the soil in the various layers of the 60-cm cylinder was used to determine the excess N[15] recovered by core sampling. In all other cases, the actual weight of the material involved was used. All calculations were on an oven-dry basis.

In order to evaluate possible sources of error or loss of N during sampling and analysis, a laboratory study was carried out on surface soil from the experimental area. Fertilizer N recovery, both at zero time and after 8 weeks, was measured under a range of sample drying temperatures. Various modifications of the Kjeldahl procedure were also tested.

Results and Discussion

The laboratory study showed that the method of handling and drying the samples and performing the N analysis gave satisfactory recovery of the fertilizer N in the sample (Table 1). Significant losses of ammonia N did occur from ammonium sulfate, especially at high drying temperatures, but the conditions that caused this loss did not occur in the field experiments.

Recovery of the ammonium N from neutral or alkaline soils was generally higher after 8 weeks incubation than at zero time in the laboratory experiment. This increase in recovery

Table 3—Recovery of fertilizer N^{15} after 8 weeks from soil fertilized with N^{15}-labeled sodium nitrate

Treatment			N^{15} recovered						
N^{15}–N used	Diameter of plots	Cropping system	Plant tops	Plant roots plus 0–15 cm	15–30 cm	30–45 cm	45–60 cm	60–75 cm	Total
%	cm		————————————————————————% ————————————————————————						
			N applied at seeding						
8	30	Fallow	—	21.8	61.2	16.0	0.3	—	99.3
			—	8.1	69.0	22.5	0.1	—	99.7
8	30	Sudangrass	42.6	26.3	29.0	1.3	0.5	—	99.7
			47.7	20.5	26.9	1.5	0.6	—	97.2
8	60	Sudangrass	40.6	26.3	33.2	0.4	0.4	—	100.9
			36.0	26.6	34.7	0.7	0.6	—	98.6
16	30	Sudangrass	42.4	25.7	28.7	0.9	0.5	—	98.2
			42.7	26.3	25.8	1.3	0.2	—	96.3
			N applied to growing plants						
8	30	Sudangrass	39.6	25.6	35.3	1.1	0.2	—	101.8
			40.8	20.3	37.5	0.7	0.3	—	99.6
8	60	Sudangrass	31.7	24.9	43.5	0.7	0.1	—	100.9
			32.3	41.6	25.1	0.7	0.4	—	100.1
16	30	Sudangrass	41.3	20.7	36.6	0.6	0.3	—	99.5
			42.5	32.1	23.0	0.6	0.5	—	98.7
16	30	Sudangrass*	55.0	12.4	24.9	4.0	0.7	0.6	97.6
			48.3	13.8	29.6	5.3	0.6	0.5	98.1

* Uncovered plots, all other plots covered during periods of heavy rainfall.

Table 4—Recovery of fertilizer N^{15} after 8 weeks from soil fertilized with N^{15}-labeled ammonium sulfate

Treatment			N^{15} recovered						
N^{15}–N used	Diameter of plots	Cropping system	Plant tops	Plant roots plus 0–15 cm	15–30 cm	30–45 cm	45–60 cm	60–75 cm	Total
%	cm		————————————————————————% ————————————————————————						
			N applied at seeding						
8	30	Fallow	—	51.3	38.1	7.7	0.4	—	97.5
			—	53.8	37.1	6.1	0.2	—	97.2
8	30	Sudangrass	56.4	34.8	7.2	0.7	0.7	—	99.8
			53.0	41.7	4.3	0.3	0.4	—	99.7
8	60	Sudangrass	46.5	43.1	9.2	0.7	1.5	—	101.0
			50.1	38.5	8.1	0.5	1.2	—	98.4
16	30	Sudangrass	57.1	31.7	6.8	0.9	0.5	—	97.0
			59.1	35.8	2.7	0.6	1.0	—	99.2
			N applied to growing plants						
8	30	Sudangrass	47.0	51.4	1.0	0.5	0.4	—	100.3
			25.9	66.9	3.7	0.3	0.4	—	97.2
8	60	Sudangrass	37.0	61.8	0.7	0.7	0.7	—	100.9
			42.9	55.8	1.4	0.3	0.7	—	101.1
16	30	Sudangrass	45.1	51.4	0.7	0.8	0.7	—	98.7
			47.0	49.1	1.0	0.8	0.3	—	98.2
16	30	Sudangrass*	49.3	44.1	1.6	0.8	0.5	0.0	96.3
			52.6	42.4	1.3	0.9	0.7	0.6	98.5

* Uncovered plots, all other plots covered during period of heavy rainfall.

was due to the nitrification that took place during incubation, making less ammonia available for loss in the gaseous form.

The variation and inadequacy of core sampling in a study of this nature is emphasized by the data in Table 2. Wide variations in recovery of excess N^{15} occurred with both sources of N, but seemed to be greater with ammonium sulfate. Contributing factors were the unevenness of the original application of the N, the difficulty of getting a good sample when roots were present, and the inaccuracy of determining the total weight of the soil in the layer sample. Probably no reasonable core sampling procedure would be sufficiently accurate in this experimental arrangement. It is shown that it is better to remove, weight, mix, and subsample the entire soil mass from within the rims than to depend upon core sampling.

The recovery of N^{15} from the field plots varied from 96.3 to 101.8%, and averaged 99.0% of that added (Tables 3 and 4). There were no significant differences in recovery of added N caused by treatment, such as cropping, time of application of N, the two different N sources, size of plot, and exposure to

natural rainfall. The part of the experiment dealing with total percent recovery of N^{15} had a coefficient of variation of 1.50 and an average difference of 1.41% between replications.

In Tables 3 and 4, it is shown that the results obtained were just as accurate from the 8% enrichment level of N^{15} additions as from the 16% level.

Tables 3 and 4 show that there were only insignificant differences in the results from the two sizes of rims. There are many disadvantages to using the larger size of rim, including the increased cost of N^{15} and cylinders, the greater amounts of both plant tops and soil that must be sampled, and the difficulty of placing larger rims in undisturbed soil.

The double-rim system was used to detect any movement of any N^{15}-fertilizer below the center rim and uptake by surrounding plants. None was detected, indicating that in this soil the single cylinder 45 cm deep was adequate.

The recovery of nitrogen was as good from the uncovered as from the covered plots (Tables 3 and 4). Rainfall during the period of the experiment was about 30% below normal.

The covered plots received a total of 11.4 cm (4.5 inches) of water, mainly from irrigation during the growing season. The uncovered plots received 20.8 cm (8.2 inches) of water, from both irrigation and natural rainfall, during the same period. There seemed to be a slightly greater downward movement of N in the profile on the uncovered plots, particularly on those treated with sodium nitrate. The movement of N did not go beyond the sampling zone and so did not affect recovery of the N.

Total recovery of the N^{15} was about the same whether applied at seeding time or when the plants were 20 to 25 cm high. As would be expected, there was more uptake of fertilizer N by the crop when the fertilizer was applied at the time of seeding. In this case the fertilizer N was available to the plant for a much longer time.

The movement of N depended upon the source of nitrogen, cropping system, and whether the plots were covered (Tables 3 and 4). There was very little downward movement of N from ammonium sulfate in the cropped soils. Most of the N was either taken up by the plant or remained in the 0- to 15-cm level. In the fallow plots there was slightly more downward movement from the ammonium source, but very little moved below 45 cm. There was greater downward movement of nitrate N in both the cropped and fallow plots. In the cropped soil, most of the N was found in the plant and 0- to 30-cm layer, with slightly greater movement in the uncovered plots. Again, there was greater movement of N in the fallow plots. In all cases, only a trace of fertilizer N was found in the 45-60-cm and 60- to 75-cm (24- to 30-inch) layers. This N^{15} was probably carried to the lower depth in the cropped plots by numerous roots which were found in the zone.

This experiment shows clearly that the techniques developed and used are reliable enough to determine small changes in the fertilizer N balance of the soil. A single cylinder, approximately 30 cm in diameter, pressed into the soil to a depth of 45 to 60 cm, constituted a satisfactory plot and adequately confined the added N. The amount of water each cylinder receives should be controlled to prevent any leaching below the sampling zone. The N source need not be tagged at a level above 8% excess N^{15} when the rate of application of the N is at a high level. The concentration of the N^{15} in the upper 45 cm of soil was ample to make an accurate analysis. There could be advantages to using a fertilizer containing a higher percent N^{15} where lower rates of N were applied, where the N^{15} was analyzed in the lower soil layers, and when using soils higher in N.

Almost complete recovery of the added N was obtained in all treatments. There was no indication that any of the added N was lost either by leaching or in the gaseous form. The small differences reported are within the experimental errors of the procedures used.

There was considerable range in the amount of the N^{15} fertilizer found in the crop and in various soil layers depending upon the cropping system and the form of N used. Despite this variation in location of the added N, satisfactory recovery of all excess N^{15} was possible. The methods described permit detection of small changes of added fertilizer N in the plant and soil. The added N fertilizer can be traced in its transformation throughout the soil profile as well as in the plant.

PART II EFFECT OF NITROGEN RATE, SOURCE AND SOIL pH ON RECOVERY OF APPLIED NITROGEN

Materials and Methods

The experimental plan consisted of 35 randomized treatments in duplicate. The experimental and analytical procedures used in this study were essentially as described in Part I.

The treated areas consisted of 30-cm diameter metal cylinders, pressed into the soil to a depth of 60-cm. The top 15-cm of soil was removed from the cylinders and the weighed amount of soil of the proper pH returned at the time of fertilizer addition. Soil of pH 5.5–6.0 and 6.5–7.0 was selected from nearby plots that had been untreated for a number of years, and had approximately the same N content. The soil of pH 4.5–5.0 was obtained by treating the soil of pH 5.5–6.0 with sulfuric acid, thoroughly leaching, and incubating the treated soil for 2 weeks before application to the experimental plots.

Ammonium sulfate and sodium nitrate, both labeled at 8% excess N^{15} for the 336 kg/ha-(300 lb/acre) N rate and 4% excess N^{15} for the 672 kg-(600 lb) rate were applied in 500 ml of solution. Unlabeled N fertilizer, at the rate of 336 kg N/ha, was applied to the soil outside the treated cylinders. Sudangrass was then seeded on the cropped plots and on the surrounding area outside the treated cylinders.

Leaching was prevented by use of movable plastic shelters during rainstorms, with adequate moisture being maintained by supplemental irrigation.

Plots receiving the nitrification inhibitor 2-chloro-6-(trichloromethyl) pyridine were handled separately from the other treatments (6). The chemical was applied in solution to the upper 22.5 cm (9 inches) of the soil at a rate to give a final 5-ppm concentration in the soil. The method used to obtain this concentration was to first prepare a 10% solution of the chemical in acetone (10 g made to 100 ml). This solution was then diluted with distilled water to give a final concentration of 50-ppm solution of the chemical in water (1 ml of acetone, chemical combination per 2,000 ml distilled water). The chemical-water mixture was violently agitated during addition of the chemical in acetone in order to get it into solution. A total of 640 ml of this solution was added to the surface of the subsoil followed by the addition of 1,360 ml to the surface 15 cm or 13.6 kg (30 lb) of soil. The treated cylinders were covered with aluminum foil for 36 hours before further treatment. The upper 10 cm (4 inches) of this soil was then removed and N^{15} fertilizer was applied in the same manner as the other plots.

At the end of an 8-week growing period the crop was harvested and the soil removed from each plot in 15-cm increments to a depth of 45 cm. Core samples were taken by 15-cm increments from 45 to 90 cm (18 to 36 inches).

Results and Discussion

Total recovery of fertilizer N from these experiments after eight weeks ranged from 88 to 96% (Table 5). This recovery value was lower than in the first year's study, probably because of slight differences in soil conditions or conditions that were not measured in this experiment. There was greater recovery of the N^{15} fertilizer from the ammonium than from the nitrate source of N. Also, total recovery of the N was greater from both sources of N when the plots were cropped rather than when left fallow. The soil pH, fertilizer concentration, and the addition of 2-chloro-6-(trichloromethyl) pyridine had no significant consistent effect upon the percentage recovery of the N^{15}. The unrecovered fertilizer N, after eight weeks, appears to have been the result of N loss in the gaseous form, since precautions were taken to prevent leaching and to reduce errors in sampling and analysis to a minimum.

Plant uptake of the added N^{15} was slightly greater from the

ammonium than from the nitrate source (Table 5). There was a greater uptake at the higher soil pH levels on the ammonium-treated plots. Also, there was an increase in the total amount of fertilizer taken up when higher rates of the fertilizer were used.

The loss of N increased with time as was indicated by soil samples taken two and ten months after applying 336 kg of N/ha from sodium nitrate to soil at pH of 5.5-6.0 (Table 6). A total of 10% of the added N was lost in the 2 warm months following application in July; followed by a 13% loss (77.1% N recovery) during the 8 fall, winter, and spring months that followed. There was no evidence of loss of N by leaching in any of these treated cylinders as indicated by amounts recovered at various soil depths. This indicates a slow rate of gaseous loss of N during all seasons that is more rapid during the warmer periods of the year.

In a supplemental experiment, a 14-month incubation was also tested. During the last 4 months of this test, however, the cylinders were closely covered by plastic sheets. Under these conditions an additional loss of 32% of the added N occurred. Such a loss appears inconsistently high, and suggests that anaerobic conditions may have been inadvertently induced, leading to excessive denitrification.

There was an increased loss of N from the plots that were not protected with a plastic cover (Table 7). Significant quantities of the N^{15} were found in the lowest soil layers sampled, indicating that leaching of the N was occurring. This loss of N was probably due to leaching of the fertilizer below the sampling zone. The uncovered plots received 20.0 cm (7.88 inches) of water from irrigation and natural rainfall during the growing season. The covered plots received 17.1 cm (6.73 inches) of water, mainly from irrigation sources during the same period.

The application of 2-chloro-6-(trichloromethyl) pyridine to soils receiving ammonium fertilizer had little effect on total recovery or plant uptake, but had considerable effect upon movement of the fertilizer N (Table 8). Nitrification of ammonium fertilizer was decreased on the treated soil with little movement of ammonium in the soil. The N^{15} fertilizer that did move into the lower horizons was generally in the nitrate form. The greatest advantage of adding the chemical to retard nitrification was to hold the N in the root zone for longer periods of time.

There was very little difference between the total N technique and the N^{15} technique for determining recovery of fertilizer nitrogen by the plant (Table 9). This close agreement between the two techniques was favored by the low N content of the soil and the large concentration of fertilizer used. Additional work is needed to show that close agreement would exist on soils of higher nitrogen content and lower levels

Table 5—Effect of N source and rate on recovery of N^{15} after 2 months in cropped and fallow soils at different pH levels

Treatment		N^{15} recovered (0-60 cm soil)		
			Cropped soil	
N/ha	Soil pH	Fallow soil total*	Plant tops	Total*
kg		%	%	%
NaNO₃				
336	4.5-5.0	90.5	43.9	93.1
	5.5-6.0	90.2	41.6	94.8
	6.5-7.0	93.2	45.3	94.5
672	4.5-5.0	88.5	22.8	91.6
	5.5-6.0	89.9	24.0	93.5
	6.5-7.0	92.3	24.2	94.0
(NH₄)₂SO₄				
336	4.5-5.0	95.1	40.7	93.7
	5.5-6.0	93.2	49.2	93.0
	6.5-7.0	93.2	49.8	93.0
672	4.5-5.0	95.2	29.2	96.2
	5.5-6.0	91.9	32.6	95.0
	6.5-7.0	92.2	36.4	95.5

* LSD 5% = 2.4, 1% = 3.3. Average difference of 1.48% between replications.

Table 6—Effect of time on recovery and movement of added fertilizer N^{15}

Time of sampling*	N^{15} recovered					
	0-15 cm	15-30 cm	30-45 cm	45-60 cm	60-75 cm	Total
	%					
2 months (fall)	20.2	51.1	20.0	0.3	—	91.6
	22.3	50.3	15.9	0.3	—	88.8
10 months (spring)	24.8	21.6	25.3	3.2	0.7	75.6
	26.4	20.9	27.6	3.3	0.5	78.7

* All plots were fallow and received 336 kg N per ha as NaNO₃ applied to soil of pH 5.5-6.0. Plots were protected with a plastic cover.

Table 7—Effect of covering plots on recovery and movement of added fertilizer N^{15} after 2 months

Treatments*		N^{15} recovered					
Nitrogen source	Special treatment	Plant tops	Plant roots plus 0-15 cm	15-30 cm	30-45 cm	45-60 cm	Total
		%					
NaNO₃	Covered	24.0	17.2	46.9	5.1	0.3	93.5
	Uncovered	21.2	5.7	8.9	30.0	5.6	71.4
(NH₄)₂SO₄	Covered	32.6	38.6	22.6	0.6	0.6	95.0
	Uncovered	30.2	11.3	23.0	14.5	4.4	83.4

* All plots received 672 kg N per ha applied to soil of pH 5.5-6.0.

Table 8—Effect of 2-chloro-6-(trichloromethyl) pyridine on the recovery, movement, plant uptake, and nitrification of fertilizer N^{15} in fallow and cropped soils

Treatment		N^{15} recovered in plants and soil						Nitrification‡	
$(NH_4)_2SO_4$	Chemical*	Plant tops	Plant roots† plus 0–15 cm	15–30 cm	30–45 cm	46–60 cm	Total	0–15 cm	15–30 cm
kg N/ha					——%——				
					Fallow				
336	–	—	59.4	30.3	3.5	0.0	93.2	68.9	93.9
	+	—	82.2	11.8	1.2	0.0	95.2	29.1	90.2
672	–	—	54.2	35.5	2.2	0.0	91.9	38.9	75.6
	+	—	83.3	7.4	0.5	0.0	91.2	18.3	88.1
					Cropped				
336	–	49.2	21.4	21.9	0.8	0.3	93.6	22.3	75.4
	+	51.1	38.4	1.7	0.5	1.0	92.7	4.5	27.0
672	–	32.6	38.6	22.6	0.6	0.6	95.0	13.1	47.9
	+	29.6	56.9	8.1	0.4	0.1	95.1	2.0	66.2

* 2-chloro-6-(trichloromethyl) pyridine.
† The 0–15 cm soil layer included plant stubbles in addition to roots in soil.
‡ The percentage of total N recovered for a given depth found as NO_2 and NO_3.

Table 9—Comparison of total N and excess N^{15} for determining N fertilizer recovery by sudangrass

Rate of application	Soil pH	N recovered			
		$NaNO_3$		$(NH_4)_2SO_4$	
		Using total N	Using excess N^{15}	Using total N	Using excess N^{15}
kg N/ha			——%——		
336	4.5–5.0	43.2	43.9	42.2	40.7
	5.5–6.0	43.4	41.7	49.9	49.2
	6.5–7.0	46.0	45.6	51.7	49.8
672	4.5–5.0	21.5	22.8	38.1	29.2
	5.5–6.0	24.8	24.1	32.3	32.6
	6.5–7.0	23.1	24.2	35.7	36.4

of N fertilizer. Only then would it be possible to detect whether or not the methods normally used in the field for determining recovery of fertilizer N are reliable.

The losses reported are attributed to gaseous loss of nitrogen, since adequate precautions were used to restrict leaching and to eliminate errors in sampling and analysis. The techniques and procedures used have been tested in both laboratory and in field studies and have been shown to recover essentially 100% of the N present in the soil at the time of analysis. Since all possible losses have been eliminated except those to the atmosphere, loss must be in the gaseous form.

The pH levels were established only in the top 15 cm of soil in this experiment. There is a possibility that conditions in the lower soil layers are as important in these losses as the upper 15 cm. Also, by addition of these fertilizers to the upper 15 cm, the pH of the soil would be affected by the form and amounts of the fertilizer applied. These factors could be partially responsible for the small differences in total N recovery at different pH levels.

The mechanism of the consistent losses that occurred, regardless of the form of N added, cannot be explained with certainty. The most probable explanation is that slow denitri-

fication was taking place, since the losses increased with movement of nitrate into the lower horizons where anaerobic conditions in the micropores of the soil are more likely to occur

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