

Soil Nitrate-nitrogen Determined by Coring and Solution Extraction Techniques¹

E. E. ALBERTS, R. E. BURWELL, AND G. E. SCHUMAN²

ABSTRACT

Soil-coring and solution-extraction sampling techniques were compared for determining the content of NO₃-N in the soil profile of a Monona silt loam (Typic Hapludoll) in southwestern Iowa. The NO₃-N content of the 3.05-m profile, determined by solution extraction, was 28% lower in 1972, 8% higher in 1973, and 13% lower in 1974 than that determined by soil coring. The profile difference in NO₃-N content between the two sampling techniques was insignificant in 1973 and 1974 but was highly significant (1%) in 1972.

Additional Index Words: inorganic N, ceramic cups, leaching.

AGRICULTURAL RESEARCH in recent years has focused on the fate of fertilizer nitrogen (N) in the environment. The movement of the NO₃⁻ ion in soils has been investigated extensively because accumulations in ground water supplies can be hazardous to human health (Gruener and Schuval, 1970). Soil core sampling has been the standard sampling technique ordinarily used to follow N movement in the soil. However, soil coring is time consuming, and each set of samples represents a different physical location in the soil. Recently, porous ceramic cups have been used to study the behavior and movement of N in the soil (Wagner, 1965; Elliott et al., 1972). Once installed, ceramic cups are versatile and easily usable and provide for in situ sampling of the soil profile. England (1974) discussed some of the basic problems in interpreting data collected from ceramic cups. Hansen and Harris (1975) found, under rapid drainage conditions, considerable variation in NO₃⁻ concentrations among samples collected from ceramic cups. The suitability of ceramic cups for collecting NO₃⁻ data needs to be evaluated under stable soil moisture conditions. The objective of our study was to compare the soil core sampling technique with the solution extraction sampling technique for determining NO₃⁻ in the soil profile. These comparisons were made between significant infiltration events when soil moisture was near field capacity.

MATERIALS AND METHODS

Six plots were established on a well-drained, deep loess Monona silt loam soil (Typic Hapludoll fine, silty mixed mesic) near Treynor, Iowa. This soil is quite uniform and varies little in textural composition with depth. The plots, each 4.1-m square with a 2.5-m square sample area centered inside, were leveled to insure near uniform infiltration. Aluminum strips were inserted around the plots to prevent surface water movement across plot borders. The plots were maintained in a continuous fallow condition.

The ceramic cups used in the study were 15-mm outside diameter by 155 mm long, with a pore diameter of 1.5 to 2.5 μm (purchased from the Coors Porcelain Company, Golden, Colorado).

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²Soil Scientist, USDA—ARS, West Lafayette, Indiana (formerly Council Bluffs, Iowa); Soil Scientist, USDA—ARS, Columbia, Mo.; and Soil Scientist, USDA—ARS Cheyenne, Wyo., respectively.

³Trade names are included for the benefit of the reader and do not imply endorsement or preferential treatment of the product named by the USDA.

do³). Soil solution moves into the ceramic cup in response to a suction gradient across the ceramic wall. Therefore, a ceramic cup samples a cylindrical volume in the soil, draining water held at suctions of < 1 bar. Wagner (1962) and Reeve and Doering (1965) described the operating procedures of similar extraction apparatus. New cups can yield several milligrams of Ca and Mg after they are flushed with a dilute acid. To remove these cations, all cups were successively flushed with 0.1N HCl and distilled water before field installation. The ceramic cups were installed at depths of 8, 22, 38, 54, 68, 84, 100, 114, 136, 168, 214, and 274 cm on 50.8-cm horizontal spacings in the sample area of each plot.

The initial comparison of the two sampling techniques was made late in the summer of 1972 during the installation of the ceramic cups. A 2.5-cm diameter vertical hole was hand augered to a point directly above the placement depth of the ceramic cup. The soil augered from the hole was placed in plastic sacks and labeled as to removal depth. To facilitate placement of the ceramic cup, we extracted a soil core from this hole using a 15-mm by 155-mm cutaway tube. This sample was placed in a plastic vial and frozen for chemical analysis. The ceramic cup was inserted into position and the hole backfilled using the original soil material. A 0.8-bar vacuum was immediately applied to the ceramic cup. Because the soil was near field capacity, a 15- to 20-ml sample of the soil solution was usually obtained within 48 hours after the vacuum was applied. The soil solution samples were refrigerated in the field with ice. After collection, the samples were stored at a temperature of 4°C until NO₃⁻ could be analyzed.

Second and third comparisons of the sampling techniques were made in August 1973 and June 1974. Nitrogen fertilizer, as granular KNO₃ (13.5% N), was applied to the plots at rates of 168 and 448 kg/ha in May 1973 and 1974. The experiment was a completely randomized design with three replications. To insure nearly uniform fertilizer application, each plot was subdivided into smaller areas where fertilizer was applied on each subdivided area. The fertilizer was then incorporated to a 5-cm depth by rotary tilling. The soil core samples collected in 1973 and 1974 were about 8 cm long and were obtained at the described sample depths in a continuous vertical profile near the center of the plots. Soil solution samples were obtained from the ceramic cups installed in 1972.

The NO₃-N content of the soil solution was determined by continuous-flow colorimetric procedures (Henriksen and Selmer-Olsen, 1970). Nitrate-nitrogen was determined on a 1:10 1N KCl extract of the thawed soil, using the same colorimetric procedure. The soil samples were dried at 65°C to determine moisture percentage. Nitrate-nitrogen concentrations determined by these two sampling techniques are reported on a dry-weight soil basis. The soil core sampling technique and the solution extraction sampling technique will be referred to hereafter as soil sampling and solution sampling, respectively.

RESULTS AND DISCUSSION

The 1972 comparison between the two sampling techniques is shown in Fig. 1a. The concentration of NO₃-N determined by solution sampling was lower than that for soil sampling at all sample depths and ranged from 6 to 57% lower for the 68- and 168-cm depths, respectively. Also shown is the total NO₃-N content in the 305-cm profile determined by solution and soil sampling. Analysis of variance showed the difference in total profile content to be highly significant ($p < 0.01$). Because a vacuum was applied to the ceramic cups immediately after installation, insufficient time was allowed for the distilled water in the cup

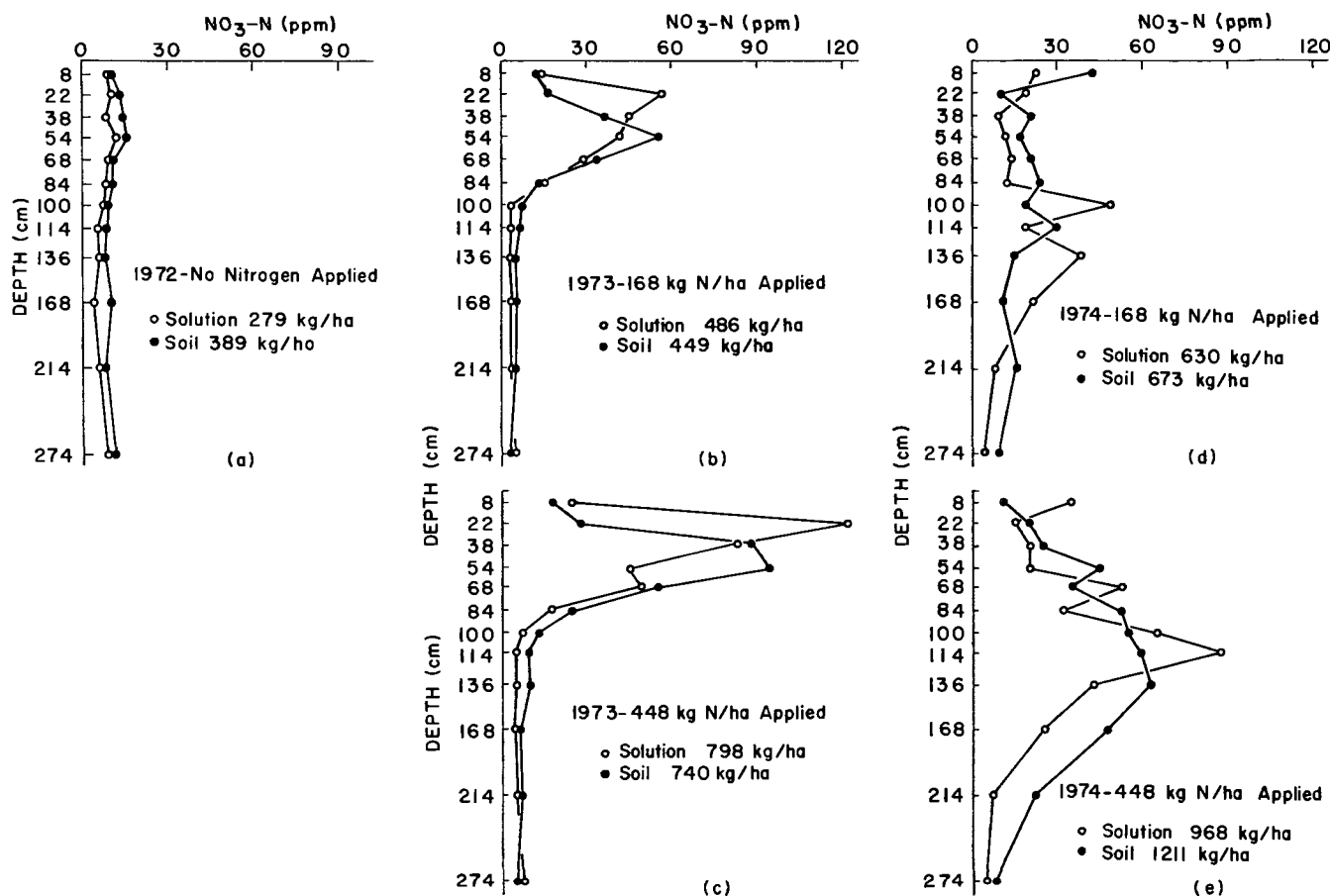


Fig. 1—Nitrate-nitrogen concentrations determined by soil coring and solution extraction sampling techniques.

walls to equilibrate with the surrounding soil solution. The distilled water should have been removed after installation by flushing with several pore volumes of soil solution. However, since it was not removed, there was some dilution of the incoming soil solution. Later we found that the ceramic wall retained about 7 ml of distilled water immediately after laboratory flushing. However, we could not determine the amount of dilution because the initial sample volumes were not recorded.

The 1973 comparisons for the 168- and 448-kg/ha N treatments are shown in Fig. 1b and 1c, respectively. For both treatments, the $\text{NO}_3\text{-N}$ bulge determined by solution sampling peaked 32 cm shallower in the soil profile than that for soil sampling. The volume of soil sampled during the collection of a 20-ml sample of the soil solution could be estimated readily if soil moisture-tension relationships were known. We determined moisture-tension relationships on undisturbed soil core samples using pressure plate apparatus. The moisture values determined for 0.33- and 0.8-bar suctions were 31 and 26% by volume, respectively. Assuming 0.33-bar as field capacity, 1.0 cm^3 of soil at field capacity contains 0.31 cm^3 of water. At a suction of 0.8-bar, then, 0.05 cm^3 of water would be available for extraction. Therefore, apparently about 400 cm^3 of soil would be drained during the collection of a 20-ml solution sample. The moisture content of the soil profile in 1973 was near field capacity. Seemingly, the soil solution would not have been extracted from depths of more than about 3 cm below

the bottom of the ceramic cup. We cannot explain why the two $\text{NO}_3\text{-N}$ bulges peaked 32 cm shallower in the profile for solution sampling.

Below 54 cm, $\text{NO}_3\text{-N}$ concentrations were similar for the two sampling techniques. However, the concentrations determined by solution sampling were generally lower at most sample depths. de Haan and Bolt (1963) showed that anions could be excluded from a portion of the soil solution due to electrical repulsion of negatively charged clay surfaces. Smith and Davis (1974) found exclusion volumes ranging from 5 to 39% of the soil solution for a wide variety of soils. The net result is that anion concentration of the soil solution held at lower suctions is usually higher than the average of the entire soil solution. Because ceramic cups sample the soil solution held at suctions of < 1 bar, the literature suggests that the solution samples should have had a higher $\text{NO}_3\text{-N}$ concentration. However, we ascribed no fundamental significance to the fact that they did not because, under field conditions, many factors affect the concentration of anions in the soil solution.

The distribution of $\text{NO}_3\text{-N}$ within the soil profile was quite different for the two sampling techniques in 1973. However, the difference in the total $\text{NO}_3\text{-N}$ content in the 305-cm profile was not statistically significant ($p < 0.05$) for these techniques.

Horizontal spatial variation in $\text{NO}_3\text{-N}$ is often high in field soils (Prince, 1923; Schuman et al., 1975). Table 1 gives the mean, standard deviation, and coefficient of varia-

Table 1—1974 comparison of soil NO₃-N determined by coring and solution extraction sampling techniques.

Depth cm	Soil sampling			Solution sampling			Significance level
	Mean	Standard deviation	CV	Mean	Standard deviation	CV	
	ppm		%	ppm		%	
168 kg/ha							
8	52.6	24.7	47	22.8	3.5	15	n.s.
22	10.7	0.6	6	19.0	3.3	17	5
38	20.7	5.8	28	9.1	1.5	16	5
54	17.0	6.8	40	12.6	3.9	31	n.s.
68	21.0	9.8	47	13.7	5.5	40	n.s.
84	23.2	12.0	52	15.2	17.7	116	n.s.
100	19.2	4.9	25	48.6	10.1	21	5
114	29.0	7.5	26	18.9	13.4	71	n.s.
136	15.0	11.1	74	38.0	12.1	32	10
168	11.1	12.0	108	30.0	21.2	71	n.s.
214	15.9	4.5	28	7.8	3.7	47	10
274	9.4	2.4	26	4.5	0.6	13	5
448 kg/ha							
8	11.4	7.1	62	34.5	14.0	41	10
22	18.8	0.1	0.5	15.8	4.8	30	n.s.
38	24.5	4.4	18	21.9	16.5	75	n.s.
54	43.9	36.2	83	21.3	15.4	72	n.s.
68	33.6	13.9	41	53.1	55.4	104	n.s.
84	51.5	32.3	63	31.0	32.7	105	n.s.
100	54.0	32.0	59	64.8	54.1	83	n.s.
114	59.9	37.4	62	88.1	23.2	26	n.s.
136	63.6	21.5	34	40.3	33.5	83	n.s.
168	47.0	22.3	47	24.0	28.2	118	n.s.
214	21.7	13.9	64	7.6	2.4	32	n.s.
274	7.6	6.5	86	5.2	0.3	6	n.s.

tion (C. V.) for the 1974 comparisons. Spatial variation in NO₃-N concentrations was found to be high for both sampling techniques. Due to the limited number of replications, the precision with which the population means and standard deviations were estimated is undoubtedly low. However, additional samples from increased replication would have quickly exceeded our manpower and laboratory capabilities. We were interested in determining the sample sizes necessary for different precision requirements. Wide fluctuations occurred in the estimates for the standard deviations, and any single estimate of the standard deviation would have been inappropriate. However, a correlation between the means and standard deviations was observed, and this relationship was used to determine the number of replications necessary. A linear relationship through the origin fitted the data rather well with $s = 0.4X$, where s was the sample standard deviation and X was the sample mean. The 1974 results for both sampling techniques (168 kg/ha) were used in determining this relationship. Snedecor and Cochran's (1967) equation for determining sample size is $\alpha = t\sigma/\sqrt{n}$, where α is the precision requirement (and here a proportion of the mean), and t is the ordinary t -value ($p = 0.05$). We simply specified the precision requirements, substituted 0.4μ for σ , and solved for n in the equation $\alpha = t(0.4\mu)/\sqrt{n}$. Because α is a proportion of the mean (e.g. $\alpha = 0.05\mu$), the values for μ cancel out. Some examples follow:

Requirement (α)	Number of Replications
0.05 μ	246
0.10 μ	64
0.20 μ	18
0.25 μ	12
0.30 μ	10

Thus, we are confident ($p < 0.05$) that our estimate of the

mean would be within 10% of the true mean if we used 64 replications. Sample size rapidly decreased as the requirements were relaxed. However, 10 replications would still be required to obtain an estimate within 30% of the true mean.

The 1974 comparisons for the 168- and 448-kg/ha N treatments are shown in Fig. 1d and 1e, respectively. The difference in the total NO₃-N content in the 305-cm profile was not statistically significant ($p < 0.05$). However, differences in NO₃-N concentrations were quite large at some sample depths. Table 1 shows the results of t -tests that determined the significance of these differences. The results suggest that, at some sample depths, the NO₃-N concentration of the soil solution held at a suction ≤ 0.8 bar was not representative of the NO₃-N concentration in the overall soil solution. The differences in NO₃-N concentration between the two sampling techniques probably would have been smaller if the soil core samples could have been obtained a few cm from each ceramic cup. However, the resulting soil disturbance would not have been compatible with other objectives of the study.

A comparison of the soil coring and solution extraction sampling techniques for determining the total NO₃-N content in the 305-cm profile was very similar in 1973 and 1974. However, large differences in NO₃-N were found at some sample depths. Spatial variation makes it difficult to interpret NO₃-N data collected by either sampling technique, especially when the sample depths can only be replicated a few times. The sampling technique to use depends upon the objectives and other circumstances surrounding the study. Ceramic cups are about the only practical way to obtain in situ samples of the soil solution under field conditions.

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