

## SHORT COMMUNICATION

**A rapid method for determination of nitrate, nitrite, and ammoniacal nitrogen in soils\****Summary*

A rapid procedure is proposed for simultaneous determination of  $\text{NH}_4^-$ ,  $\text{NO}_2^-$ , and  $\text{NO}_3^-$ -N in field moist samples. The procedure involves extraction, with Morgan's solution followed by determination of  $\text{NH}_4^-$ -N by Nessler's reagent,  $\text{NO}_2^-$ -N by  $\alpha$ -naphthylamine and sulphanilic acid and  $\text{NO}_3^-$ -N by phenol-disulphoric acid. The method will suit small laboratories and 50 to 75 samples can be analysed in a day.

Due to low recoveries of nitrogen in tropical and sub-tropical areas of the world <sup>9</sup> the need for intensive field studies on nitrogen dynamics have been widely felt. A large number of field experiments are being conducted to study the efficiency of nitrogen applied to field crops in these countries and this permits an opportunity to study the fate of applied nitrogen. It is very well recognised that there would be considerable variability in soil samples even within the same plot and many a times estimates considerably differ between replicates. In studies involving nitrogen dynamics, speed of analysis of the samples is also very important because the nitrogen transformations are dynamic in nature. Very few laboratories will have required soil sample storage facilities and invariably the samples must be analysed while still moist. This point has also been emphasised by Bremner <sup>3</sup>. Many workers <sup>1 2 4</sup>; have shown that storage and handling of soils samples could largely influence the proportions of ammonium, nitrite, and nitrate nitrogen. Even simple field experiments would involve the handling of a minimum 40 to 50 samples at a time. Prasad <sup>8</sup> has proposed a procedure based upon the extraction methods used in the Netherlands by small laboratories <sup>11</sup> for quick simultaneous determination of nitrate, nitrite and ammonium nitrogen. This paper reports the data on recovery of added nitrate, nitrite and ammonium nitrogen to soil samples when estimated by the procedure proposed.

*Material and methods*

The laboratory experiments from which the data are reported in this paper were conducted on a sandy clay loam soil belonging to old alluvium series

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TABLE 1  
Characteristics of the soil used

Characteristics	Value
Mechanical analysis (%)	
a) Sand	61.0
b) Silt	15.0
c) Clay	24.0
pH (1:2:5 soil to solution)	7.7
Total nitrogen (%)	0.07
Available nitrogen (Prasad <sup>7</sup> kg/ha)	24.4
Available (Olsen's) P <sub>2</sub> O <sub>5</sub> (kg/ha)	62.3
Available K <sub>2</sub> O (kg/ha)	353.0
Ammoniacal nitrogen (ppm)	4.4
Nitrate nitrogen (ppm)	9.0
Nitrite Nitrogen (ppm)	Traces
Cation exchange capacity (me/100 g soil)	11.6
Organic carbon (%)	0.6

and having important physical and chemical characteristics as given in Table 1.

Extraction of nitrate, nitrite and ammonium nitrogen from soil samples. The procedure adopted was same as reported by Schuffelen *et al.*<sup>11</sup> and consists of mechanically shaking 10 g of soil with 50 ml of Morgan's Reagent (100 g of sodium acetate + 30 ml of glacial acetic acid, diluted to one litre with distilled water and adjusted to pH 4.8) and about  $\frac{1}{4}$  g of activated charcoal. The solution was filtered through Whatmann No. 1 filter paper. If some turbidity is seen, the filtrate could be transferred to funnel and re-filtered. Although in these laboratory experiments moisture estimation was not necessary, in field samples it is possible and advisable to use moist samples immediately after taking them from the field. In such cases about 50-g samples should be simultaneously kept for moisture determination for adjusting the results obtained to dry weight basis.

Determination of NO<sub>3</sub>-N. This was done using a 10 ml aliquot of the above extract and following the procedure as described by Jackson<sup>5</sup>. The colour developing reagent used was phenol-disulphonic acid. The standard curve was prepared using 0, 1, 2, 3, 4, and 5-ml aliquots of 20 ppm NO<sub>3</sub>-N solution (prepared with potassium nitrate).

Determination of NO<sub>2</sub>-N. Nitrites were estimated by using 1- to 5-ml aliquots of the above extract and following the procedure described by Prince<sup>10</sup>. The reagents used for developing pink colour were:  $\alpha$ -naphthylamine and sulphanilic acid. (Prasad has later obtained better results with (*N*-1-Naphthyl) ethylenediamine hydrochloride in place of  $\alpha$ -naphthylamine

as proposed by Montgomery and Dymock <sup>6</sup>). The standard curve was prepared using a 1-ppm NO<sub>2</sub>-N solution (prepared with potassium nitrite).

Determination of NH<sub>4</sub>-N. Ammonium nitrogen was determined using a 2 ml aliquot of the above extract and following the procedure described by Schuffelen *et al.* <sup>11</sup>. A turbidity may be encountered with some soils but using an appropriate dilution one can easily overcome this problem.

#### *Results and discussion*

The data in Table 2 show the recovery of 87 to 90 per cent of the added NO<sub>3</sub>-N, while in case of NO<sub>2</sub>-N the recovery was 85 to 90 per cent. The recovery of added NH<sub>4</sub>-N was about 80 per cent for the soil used in the studies. Estimation of NH<sub>4</sub>-N in the extracts by Nesslerization as per procedure described above and by distillation method were also compared. Both the procedures gave same results and these are given in Table 3.

The data presented in Table 2 clearly show that the proposed procedure is fairly reliable for simultaneous estimation of NO<sub>3</sub>-N, NO<sub>2</sub>-N and NH<sub>4</sub>-N in soil samples. Only in case of NH<sub>4</sub>-N, the recovery was slightly low and this would generally be influenced by the ammonium fixing capacity of the soils.

TABLE 2  
Recovery of NO<sub>3</sub>-N, NO<sub>2</sub>-N and NH<sub>4</sub>-N added to soil

N added (mg)	N recovered (mg)	Recovery (%)
	NO <sub>3</sub> -N	
5.0	4.5	90.0
10.0	9.0	90.0
20.0	17.5	87.5
30.0	27.0	90.0
40.0	35.2	88.0
50.0	45.0	90.0
100.0	88.5	88.5
	NO <sub>2</sub> -N	
2.5	2.2	88.0
5.0	4.5	90.0
10.0	9.0	90.0
15.0	12.8	85.3
	NH <sub>4</sub> -N	
5.0	4.1	82.0
10.0	8.1	81.0
15.0	12.4	82.7
20.0	15.6	78.0
25.0	20.5	82.0
50.0	41.0	82.0
100.0	81.5	81.5

TABLE 3

Estimation of  $\text{NH}_4\text{-N}$  (ppm) by Nesslerisation (as described above) and by distillation after extraction with Morgan's reagent

By Nesslerisation	By distillation
12.6	12.1
31.8	30.9
45.0	44.5

The authors' experience is that a laboratory technician can easily handle 50 to 75 samples in a day and completely determine the  $\text{NO}_3^-$ ,  $\text{NO}_2^-$  and  $\text{NH}_4\text{-N}$ . Considering the variability encountered in the soil samples and the errors which creep in due to the poor storage and care of the samples, the recovery shown above even of added  $\text{NH}_4\text{-N}$  should be considered satisfactory. The procedure will also permit the collection of a large volume of data from various tropical and sub-tropical countries, which happen to be underdeveloped or developing nations with very little laboratory facilities.

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