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PREMIXED DIGESTION SALTS FOR KJELDAHL DETERMINATION OF TOTAL NITROGEN IN SELECTED FOREST SOILS

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ABSTRACT

Estimates of total soil nitrogen by a standard Kjeldahl procedure and a modified procedure employing packets of premixed digestion salts were closely correlated ($r^2 = 0.983$). The modified procedure appears to be as reliable as the standard method for determining total nitrogen in southern alluvial forest soils.

INTRODUCTION

The Kjeldahl method for determining total soil nitrogen requires separate weighing and adding of several digestion salts and catalysts. This paper describes a test of commercially available premixed salts[2] that simplify the Kjeldahl procedure.

MATERIALS AND PROCEDURES

Samples from the top 12 inches of 10 forested alluvial soils from four geologic regions of the Midsouth and known to vary widely in nitrogen concentration were selected for comparing the standard and modified procedures. In the standard Kjeldahl method, salicylic acid was added to include nitrates in the total as described by Jackson[5]. In the simplified procedure, one package of premixed

salts (9.9 g K_2SO_4, 0.4 g HgO, and 0.08 g $CuSO_4$) was substituted for the Na_2SO_4 plus catalyst. Five-gram sieved samples (<2 mm) of air-dry soil were placed in 800-ml Kjeldahl flasks. One packet of salts was placed in each flask, 50 ml of distilled water were added, and the samples were allowed to stand. After 1 hour, 35 ml of concentrated H_2SO_4 were added, the flasks placed on the Kjeldahl digestion unit, and the samples digested for approximately 1 hour after the solution in the flasks cleared. Total digestion period did not exceed 2 hours.

The flasks were cooled for 30-45 minutes and diluted with 250 ml of distilled water. The mixture was swirled and allowed to cool for another 30 minutes. Three to five pieces of mossy zinc plus two glass beads were placed in each flask. Then 100 ml of 50-percent NaOH were slowly added. After gentle mixing, the samples were distilled into 25 ml of 4-percent H_3BO_3 containing bromcresol green-methyl red indicator. Titration was done with 0.02 N HCl.

The two techniques were statistically compared by regression and correlation analyses. Differences between values obtained by the two methods for duplicate samples were tested by analysis of variance. Coefficients of variation were also determined for each technique.

In both methods, organic-matter content was determined by wet combustion, reaction by glass electrode pH meter in a 1:1 soil-water mixture, and particle-size distribution by the hydrometer method (Table 1).

RESULTS AND DISCUSSION

The amounts of nitrogen detected by the two procedures were closely correlated ($r^2 = 0.983$). The high correlation (Fig. 1) indicates

TABLE 1

Selected Properties of Soils Tested

Soil series	Geologic region	Sand (%)	Silt (%)	Clay (%)	pH	Organic matter (%)
Alligator	Miss. River Floodplain	14	29	57	5.1	2.46
Amagon	" " "	36	33	31	5.2	2.00
Commerce	" " "	57	35	8	7.5	0.80
Dundee	" " "	14	45	41	5.8	3.72
Newellton	" " "	23	28	49	5.5	2.03
Sharkey	" " "	8	34	58	6.2	4.50
Tunica	" " "	24	35	41	5.5	1.00
Perry	Red River Floodplain	13	25	62	6.0	2.87
Adler	Silty Uplands	30	60	8	6.5	2.40
Bibb	Coastal Plain	68	26	6	5.0	0.50

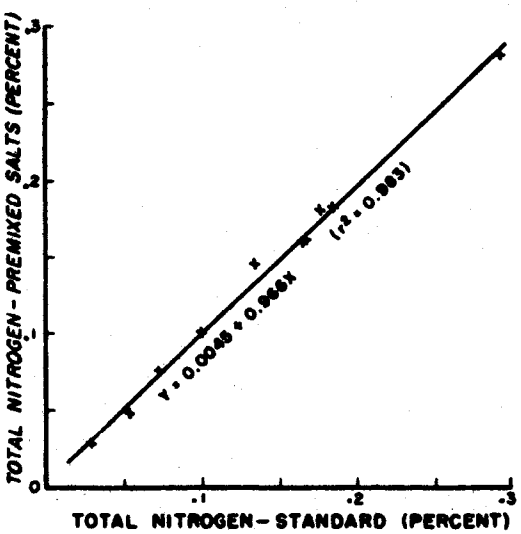

FIG. 1

Relation between values for total N by the two procedures.

that the simplified procedure was as reliable as the standard Kjeldahl technique for assessing total nitrogen. Analysis of variance of differences between duplicate samples revealed no significant variation between values obtained by the two procedures. Coefficients of variation were 54.6 percent for the standard method and 53.2 percent for the modified procedure.

There are several practical advantages to the premixed digestion salts. Since the K_2SO_4 and catalysts are sealed in small, preweighed polyethylene packets, weighing, mixing, and separate storage are eliminated. As the polyethylene bag melts, frothing, which is a common problem in early minutes of digestion, seems to be inhibited.

The differences between the two techniques are not in convenience only. The catalysts in the packets are mercury and copper. Bremner[4] states that catalytic efficiency in producing digest clearing generally decreases in the order selenium > mercury > copper; mercury is probably the most effective single catalyst when both clearing and conversion of organic nitrogen to ammonium are considered. Murneek and Heinze[6] suggest a combination of Se, Hg, and Cu. Bremner[4] points out that when mercury-containing digests are made alkaline, some of the ammonium is bound in mercuric oxide precipitate and not liberated by digestion. To overcome this problem, he recommends addition of sodium thiosulfate to precipitate mercuric sulfide or zinc dust to reduce mercuric oxide to metallic mercury. The premixed powder used here does not include sodium thiosulfate or zinc dust, yet the values obtained are correlated with those from the standard method in which sodium thiosulfate was used (Fig. 1). Perhaps the copper ion served as a reductant for part of the mercuric oxide.

Another possibility is that the mossy zinc added just prior to distillation to liberate hydrogen and promote smooth boiling (Bremner[3]) caused reduction.

In the modified procedure, salicylic acid was not added to include nitrates as described by Jackson[5]. That this omission had little effect seems to indicate that the nitrate fraction of the total nitrogen content was negligible in the soils studied. Bremner[4] also could not detect higher values with the use of reagents recommended for the inclusion of compounds with N-O linkages.

The procedure with premixed salts is safer than the standard procedure since there is no direct exposure to toxic elements such as selenium and mercury. It is also much less time consuming than the standard method and appears to be equally reliable for total nitrogen determinations in alluvial forest soils of the Midsouth.

REFERENCES

1. Research done in cooperation with the Mississippi Agricultural and Forestry Experiment Station and the Southern Hardwood Forest Research Group.

2. No. 1 Kel-Pak Powders, manufactured by Matheson, Coleman, and Bell. Mention of the trade name is solely to identify the product used and does not constitute endorsement by the USDA Forest Service.

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