

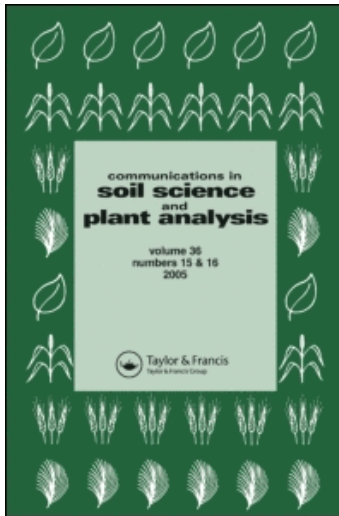
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Sodium Hydroxide Direct Distillation: A Method for Estimating Total Nitrogen in Soil

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Abstract: Soil total nitrogen (N) concentration is commonly measured by Kjeldahl and combustion methods. In this study, a method based on determination of ammonium (NH₄)-N produced by steam distillation of the soil sample with sodium hydroxide solution (NaOH-DD) is developed and evaluated for estimating soil total N concentration. The method was evaluated using two sample sets: (A) 44 samples collected from contrasting land uses in central Iran and (B) 344 samples collected from contrasting arable soils across Canada and Maine, USA. Based on this experiment, 10-min distillation of 5.0 g soil with 20 mL 12.5 M NaOH were selected as the optimum conditions. The NH₄-N released by the NaOH-DD method was highly correlated with total N concentration in the sample sets A and B ($r = 0.79$ and 0.94 , respectively). The NaOH-DD method provides a simple, rapid means of analysis with reduced measurement cost and better health and safety precautions than the traditional Kjeldahl method.

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Keywords: Combustion method, Dumas method, Kjeldahl method, NaOH distillation, soil total N

INTRODUCTION

Nitrogen (N) is the nutrient most often limiting in crop production and often added in the greatest amount (Havlin et al. 2005). Total N concentration ranges from $<0.02\%$ in subsoil to $>2.5\%$ in organic soils. In surface soils, 95 to 98% of the soil total N is present in organic compounds (Bremner 1996). Soil total N includes proteinaceous materials (proteins, peptides, and amino acids; about 40%), amino sugars (5–6%), heterocyclic N compounds (including purines and pyrimidines; about 35%), and mineral N (19%) (Stevenson 1996; Schulten and Schnitzer 1998; Olk 2007). As a result of its stability over time and importance in N supply, soil total N is commonly used for soil characterization, for modeling of soil N dynamics, and as an index of soil N availability, soil N mineralization potential, and soil quality (Angers et al. 1997; Stenberg 1998; Wander and Bollero 1999; Sharifi et al. 2007).

Soil total N concentration is one of the most frequently measured nutrients in soil-testing laboratories. The methods that are commonly used for analyses of soil total N may be divided into two major categories: wet oxidation (e.g., Kjeldahl method) and dry combustion (e.g., Dumas method). The Kjeldahl method (Kjeldahl 1883) has gained worldwide acceptance as a standard method of measuring N concentration in a variety of samples (i.e., food and beverages, meat, feed, grain, wastewater, soil). The regular Kjeldahl method includes three main steps: digestion, distillation, and titration (Bremner 1960). In general, the organic forms of N in the sample are converted to $\text{NH}_4\text{-N}$ by digestion with concentrated acid, and the $\text{NH}_4\text{-N}$ is determined from the amount of $\text{NH}_4\text{-N}$ liberated by distillation of the digest with alkali (commonly sodium hydroxide, NaOH). The Kjeldahl method currently used has been refined by numerous modifications (Bremner 1996) and permits inclusion of almost all combined forms of N. Although it is the worldwide standard, the regular Kjeldahl method has some disadvantages, including long period of the digestion, foaming or bumping during digestion, uneven digestion, precipitation of salt or caking of sample during the digestion, ammonia (NH_3) loss during the digestion, production of noxious fumes and hazardous chemical waste, and health and safety concerns about the handling of concentrated acid and heavy metals such as selenium (Se), mercury (Hg), and copper (Cu) (Bremner 1996; Rutherford et al. 2007).

The Dumas method (Dumas 1831) has also gained general acceptance for determination of soil total N in modern soil laboratories (Bremner 1996). This method normally involves an initial oxidation step

followed by passage of gases through a reduction furnace to reduce NO_x to N_2 . The quantity of N_2 is usually determined using a thermal conductivity detector (Rutherford et al. 2007). Dumas techniques have the advantages of requiring less laboratory space, providing rapid analysis, requiring less chemical reactants, producing less noxious fumes and hazardous chemical waste, and including all forms of N without lengthy pretreatments as compared with the Kjeldahl method (Bellomonte, Constantini, and Giammarioli 1987; Vittori Antisari and Sequi 1988). Combustion methods are commonly used in modern soil-testing laboratories; however, the equipment is costly to purchase and operate and requires considerable skill and experience for satisfactory use and maintenance. Sample variability may also be a concern with combustion techniques because of the small sample size commonly used for analysis (Kowalenko 2001; Rutherford et al. 2007).

Many studies have compared the performance of Kjeldahl and combustion methods for measuring soil total N. Some studies reported comparable soil total N results for Kjeldahl and dry combustion methods (Artiola 1990; Yeomans and Bremner 1991). Other studies found proportional but slightly lower (McGeehan and Naylor 1988; Vittori Antisari and Sequi 1988) or higher (Kowalenko 2001) soil total N values for the Kjeldahl method compared with the dry combustion method. High concentrations of nitrate (NO_3)-N in soil samples, and lack of pretreatment for including NO_3 -N in the Kjeldahl method, could contribute to lower measured total N concentrations by the Kjeldahl method compared with the combustion method (Rutherford et al. 2007). Kowalenko (2001) reported that the CNS-2000 instrument apparently does not measure all of the N in certain soil samples, but values that are produced are highly correlated with Kjeldahl measurements. Both methods fail to recover some forms of N, particularly N in certain heterocyclic compounds and compounds containing N-N and N-O linkages (Bremner 1996).

Several methods have been developed to assess potentially available N based on determination of the NH_4 -N liberated by distillation of soil with alkaline reagents such as NaOH or potassium permanganate plus sodium hydroxide (KMnO_4 -NaOH) (Prasad 1965; Stanford 1978; Hussain and Malik 1985; Sharifi et al. 2007) and phosphate-borate buffer (pH 11.2; Gianello and Bremner 1988; Stevenson 1996) solutions. None of these methods, however, have been tested as an estimator of soil total N. Greenfield (2001) reported that alkali hydrolysis of soils for 24 h at 110 °C with 5 M NaOH solution can hydrolyze between 40 and 99% of soil total N. The total alkali hydrolyzable N includes NH_4 -N, α -amino-N, and hydrolyzable unidentified N fractions. Alkali-insoluble residues were shown to be derived largely from fixed NH_4 -N and/or chitin (Greenfield 2001). Given that the amount of chitin and chitinous material is negligible in most soils, total alkali hydrolyzable N may be a good

estimator of soil total N concentration, particularly in soils with small amounts of fixed $\text{NH}_4\text{-N}$.

In this study, the sodium hydroxide direct distillation (NaOH-DD) method was developed and evaluated as a method for estimating soil total N concentration over a wide range of soil properties and environmental conditions. This approach may provide a simple, rapid, and cost-effective alternative to regular Kjeldahl or combustion methods, particularly in soil-test laboratories where the cost and expertise required for combustion methods is prohibitive or the safety and environmental issues associated with use and handling of concentrated acid and disposal of chemical wastes in the Kjeldahl method are problematic.

MATERIALS AND METHODS

Soils

Two sample sets were used. The samples were taken to represent different climatic zones (arid, semi-arid, subhumid, and humid), soil types, and land uses and from a range of management practices including organic amendment applications, crop rotations, and tillage practices.

Sample set A ($n = 44$) included soils collected in central Iran in 2004 from protected rangeland and forests and their adjacent rain-fed cultivated lands. Three replicates of samples were collected from the 0- to 15-cm soil depth. Soils were derived from calcareous parent materials and classified as Inceptisols (Typic Haploxerepts, Typic Calcixerepts), Mollisols (Typic Calcixerolls), Alfisols (Typic Haploxeralfs), and Aridisols (Typic Haplocalcids).

Sample set B ($n = 344$) included soils collected from existing experimental trials on cultivated land in nine provinces in Canada and in Maine, USA, in 2004–2006. Replicate (three to five) soil samples were collected from the 0- to 15-cm soil depth prior to seeding in each case. Soils were collected from humid, subhumid and semi-arid environments and are classified as Entisols (Typic Udorthents), Inceptisols (Humic Dystrochrepts, Typic Humaquepts, Typic Haplaquepts, Typic Haplaquept, Eutrochrepts), Spodosols (Typic Haplorthods, Typic Haplohumods), Mollisols (Aridic Haploborolls, Udic Haploborolls, Typic Haploboroll, Typic Argiaquoll), and Alfisols (Typic Cryoboralfs).

Soils were air-dried, ground, and sieved (<2 mm) before analysis. Soil pH was determined in a 1:1 soil–water suspension. Soil electrical conductivity was measured in a saturation paste extract (EC_e). Particle-size distribution was determined by the pipette method following organic-matter removal (Gee and Or 2002). For sample set A, soil organic carbon (C) was measured on air-dried soils using a titrimetric dichromate method

(Walkley and Black 1934), and soil total N concentration was determined by a regular micro-Kjeldahl method without pretreatment (Rutherford et al. 2007) using a 2300 Kjeltac Analyzer unit (Foss Tecator AB, Höganäs, Sweden). Pretreatment for including NO_2^- and NO_3^- was not performed because of the negligible concentrations of these anions in the soil samples (data not presented). For sample set B, soil organic C and soil total N concentrations were measured by the dry combustion method using a LECO CNS-1000 (LECO Corp., St. Joseph, Mich.) following carbonate removal. Carbonates were removed from soil by treating 1.0 g of ground soil with 3 mL of 2 M hydrochloric acid (HCl), drying at 60 °C, treatment with an additional 1 mL of 2 M HCl, drying, and grinding (Skjemstad and Baldock 2007). Soil gravimetric moisture content was measured by drying a 5-g subsample from each soil in oven at 80 °C for 48 h (O'Kelly 2004). All chemical measurement results were corrected for soil moisture and reported on an oven-dried weight basis. The physicochemical properties of the soils in both sample sets are summarized in Table 1.

Steam Distillation Method

The steam distillation apparatus (Fisher DU100 distillation unit, Fisher Scientific, Philadelphia, Penn.) described by Bremner (1996) was used. Prior to the distillation procedure, the unit was steamed for 10 min to remove traces of NH_3 , and the rate of steam generation was adjusted so that 5 to 6 mL of distillate was collected per min (Gianello and Bremner 1988). The reagents used were 50% NaOH solution, boric acid 4% solution, and sulfuric acid solution 0.01 M standard. The titrations were

Table 1. Selected chemical and physical properties of soils in two sample sets

Parameter	Sample set A (n = 44)			Sample set B (n = 344)		
	Mean.	Min.	Max.	Mean	Min.	Max.
pH	7.4	7.0	7.9	6.3	4.5	8.1
EC_e (dS m^{-1})	0.73	0.39	1.62	0.95	0.08	9.43
Sand (g kg^{-1})	222	10	443	355	55	649
Silt (g kg^{-1})	397	150	667	431	157	651
Clay (g kg^{-1})	384	90	590	214	62	636
Soil total organic C (g kg^{-1})	4.68	0.40	22.17	26.54	8.50	164
Soil total N (g kg^{-1})	2.03	0.60	4.56	2.47	0.61	11.30
NaOH-DD ^a (mg N kg^{-1})	175	11	397	365	99	1473

^aNaOH-DD, sodium hydroxide direct distillation method.

performed using a Mettler Toledo DL50 autotitrator with pH electrode (Mettler Toledo, Greifensee, Switzerland).

The NaOH-DD method was a modification of the method used by Stanford (1978). A 20-mL boric acid (H_3BO_3) 4% solution was added to a 250-mL Erlenmeyer flask and placed under the condenser of the steam distillation apparatus so that the end of the condenser was above the surface of the H_3BO_3 solution. A subsample of air-dried soil was added to a 200-mL distillation flask, connected to the steam distillation apparatus (Fisher DU100 distillation unit). NaOH solution was pumped into the distillation flask, and steam distillation started immediately. After the desired period of distillation, the $\text{NH}_4\text{-N}$ released in the distillate was back-titrated with standard 0.01 M sulfuric acid (H_2SO_4) using a Mettler Toledo DL50 autotitrator with pH electrode.

Method Optimization

The method described here originated from studies to identify a rapid chemical method to assess potentially available organic N in soil based on determination of $\text{NH}_4\text{-N}$ liberated by steam distillation of soil with an alkaline reagent for several minutes (Keeney and Bremner 1966; Stanford 1978; Hussain and Malik 1985; Gianello and Bremner 1988). The method was optimized with respect to sample size, duration of distillation, and concentration of NaOH solution.

Effect of Soil Sample Size and Distillation Duration

Three soil samples with contrasting soil characteristics (Table 2) were selected from sample set B. For each soil sample, two subsample weights (5 or 10 g air-dried) were each distilled with 20 mL 12.5 M NaOH for three durations of distillation (5, 10, and 15 min). Each measurement was replicated three times.

Effect of Sodium Hydroxide Concentration

Three soil samples with contrasting soil characteristics (Table 3) were selected from sample set B. A 5-g subsample of each soil was distilled with 20 mL of NaOH solution with concentrations of 3.125, 6.25, 12.5, or 25 M. Samples were distilled for 10 min, and each measurement was replicated three times.

Statistical Analyses

A two-way analysis of variance (SAS Institute Inc., Cary, N.C., version 8.2) was performed to test the effect of sample size, distillation duration,

Table 2. Effect of soil sample size and distillation duration on NH₄-N released by the sodium hydroxide direct distillation method

Soil	pH	Organic C (g kg ⁻¹)	Soil total N (g kg ⁻¹)	Sand (g kg ⁻¹)	Silt (g kg ⁻¹)	Clay (g kg ⁻¹)	Distillation time (min)	Soil weight (g)	NH ₄ -N (mg kg ⁻¹)
1	5.2	16.8	1.90	381	456	163	5	5.0	246b
								10.0	241b
							10	5.0	258a
								10.0	263a
							15	5.0	251a
								10.0	266a
2	8.0	15.0	1.56	307	398	295	5	5.0	230b
								10.0	224b
							10	5.0	263a
								10.0	258a
							15	5.0	265a
								10.0	272a
3	6.1	38.1	4.61	75	543	382	5	5.0	459b
								10.0	468b
							10	5.0	571a
								10.0	560a
							15	5.0	566a
								10.0	569a

Note. Means for each soil followed by the same letter are not significantly different based on LSD_{0.05}.

Table 3. Effects of sodium hydroxide (NaOH) concentration on $\text{NH}_4\text{-N}$ released by sodium hydroxide direct distillation method from 5 g soil during 10 min distillation

Soil	pH	Organic C (g kg^{-1})	Soil total N (g kg^{-1})	Sand (g kg^{-1})	Silt (g kg^{-1})	Clay (g kg^{-1})	Soil weight (g)	NaOH concentration (M)	$\text{NH}_4\text{-N}$ (mg kg^{-1})
4	5.5	12.0	0.82	467	405	128	5.0	3.125	176c
								6.25	217b
								12.5	238a
								25	242a
5	8.0	16.3	1.85	337	362	301	5.0	3.125	158c
								6.25	209b
								12.5	287a
								25	280a
6	6.3	22.2	1.81	449	305	246	5.0	3.125	317c
								6.25	377b
								12.5	479a
								25	485a

Note. Means for each soil followed by the same letter are not significantly different based on $\text{LSD}_{0.05}$.

and NaOH concentration in a completely randomized design with three replications. Treatment means were compared using a protected least significant difference ($P < 0.05$) test. The association between different variables was evaluated using correlation and regression analyses (SAS Institute Inc., Cary, N.C., version 8.2). Each variable that had a significant correlation with soil total N concentration measured by the NaOH-DD method was examined to see if its addition to the regression model significantly improved the estimation of soil total N. The coefficient of variation (CV) of each method was calculated as an index of precision and reproducibility.

RESULTS AND DISCUSSION

Inclusion of samples from a wide variety of parent materials, land use (forest, rangeland, and cultivated), management practices and history (tillage system, crop rotation, and organic amendment), and soil moisture regime (arid, semi-arid, subhumid, and humid) resulted in a wide range in the concentrations of soil total N (0.60 to 11.3 g kg⁻¹), soil total organic C (0.40 to 164 g kg⁻¹), pH (4.8 to 8.1), and clay (62 to 636 g kg⁻¹) (Table 1). This allows a robust and meaningful evaluation of the method.

To develop the optimum conditions for the method, the major factors affecting the quantity of NH₄-N produced during the direct distillation with NaOH solution were studied. Selected soils for method optimization had large differences in soil pH, soil total N, soil organic C, and soil texture (Tables 2 and 3). Greater values of NH₄-N (average of 16%) were measured after 10 min of distillation compared with 5 min of distillation for all three tested soils (Table 2). Increasing distillation duration over 10 min did not have any influence on measured NH₄-N concentration (data not presented). There was no significant effect of soil weight (5 and 10 g) on liberated NH₃. The measured values of NH₄-N were significantly increased by increasing the NaOH solution concentration from 3.125 to 12.5 M (Table 3). There was no significant difference between extracted NH₄-N concentration by 12.5 and 25 M NaOH solutions after 10 min of distillation. Therefore, the NH₄-N released by steam distillation of a 5.0-g soil sample with 20 mL of NaOH 12.5 M for 10 min was considered the optimal conditions for this method in subsequent analyses.

The concentration of NH₄-N extracted by the NaOH-DD method ranged from 11 to 397 mg N kg⁻¹ soil for sample set A and from 99 to 1473 mg kg⁻¹ for sample set B (Table 1). These values represented an average of 9 and 17% of soil total N in sample sets A and B, respectively. The NH₄-N extracted by the NaOH-DD method is probably derived mainly from exchangeable NH₄-N, amino sugars, amides, certain amino

acids, and alkali hydrolyzable unidentified N (Stanford 1978; Greenfield 2001; Sharifi et al. 2007). The broader range of NaOH-DD $\text{NH}_4\text{-N}$ in sample set B compared with set A reflects the wider range of soil organic C and soil total N concentrations in sample set B, due to inclusion of sites with a long-term history of organic amendment application.

The NaOH-DD $\text{NH}_4\text{-N}$ was highly positively correlated with soil total N measured by the regular Kjeldahl method in sample set A ($r = 0.79^{***}$) and by the combustion method in sample set B ($r = 0.94^{***}$) (Table 4). There were significant correlations between NaOH-DD $\text{NH}_4\text{-N}$ and soil properties including soil total organic C, clay concentration, and pH in sample set B. In sample set A, soil total organic C was the only soil property that positively correlated with NaOH-DD; however, the correlation coefficient was less than that for sample set B ($r = 0.47$ vs. 0.94). Soil clay concentration was negatively correlated with NaOH-DD $\text{NH}_4\text{-N}$ ($r = -0.37$), whereas there was no significant correlation between NaOH-DD $\text{NH}_4\text{-N}$ and pH in sample set A.

Regression of NaOH-DD $\text{NH}_4\text{-N}$ against the Kjeldahl method in sample set A indicated that there was a linear relationship between these two parameters; however, there is a degree of scattering of the data around the regression line (Figure 1). In sample set B, regression of NaOH-DD $\text{NH}_4\text{-N}$ against the combustion method was performed using both linear and quadratic models (Figure 2). The quadratic model explained a greater proportion of the variation in NaOH-DD $\text{NH}_4\text{-N}$ compared with the linear model ($r^2 = 0.89$ vs. 0.87); however, the improvement associated with use of the quadratic model was small. The somewhat better fit of the quadratic model suggests that the NaOH-DD method may be less effective in extracting soil N at high values (i.e., $> 5 \text{ g kg}^{-1}$) of soil total N. The NaOH-DD $\text{NH}_4\text{-N}$, expressed as a percentage of soil total N, was negatively correlated with soil organic C, clay concentration, and pH ($r = -0.57$, -0.45 , and -0.55 , respectively).

Table 4. Pearson correlation coefficients between sodium hydroxide direct distillation (NaOH-DD) $\text{NH}_4\text{-N}$ or NaOH-DD $\text{NH}_4\text{-N}$ to soil total N ratio, and selected soil characteristics for two sample sets

Parameter	NaOH-DD		NaOH-DD/soil total N
	Sample set A	Sample set B	Sample set B
Soil total N	0.79***	0.94***	-0.67***
Soil organic C	0.47***	0.92***	-0.57***
Clay	-0.37**	0.49***	-0.45**
pH	-0.16	0.55***	-0.55***

** and ***, significant at 0.01 and 0.001 probability level, respectively.

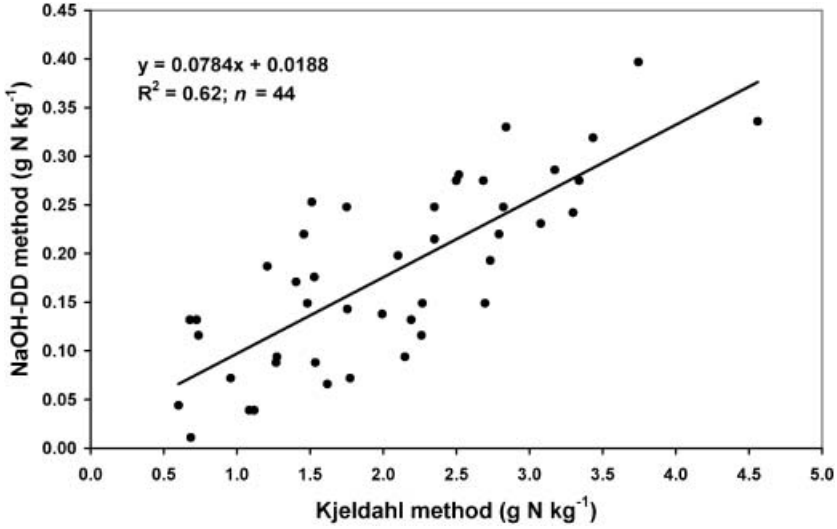


Figure 1. Regression of sodium hydroxide direct distillation (NaOH-DD) NH₄-N against soil total N concentration measured by the regular Kjeldahl method in sample set A.

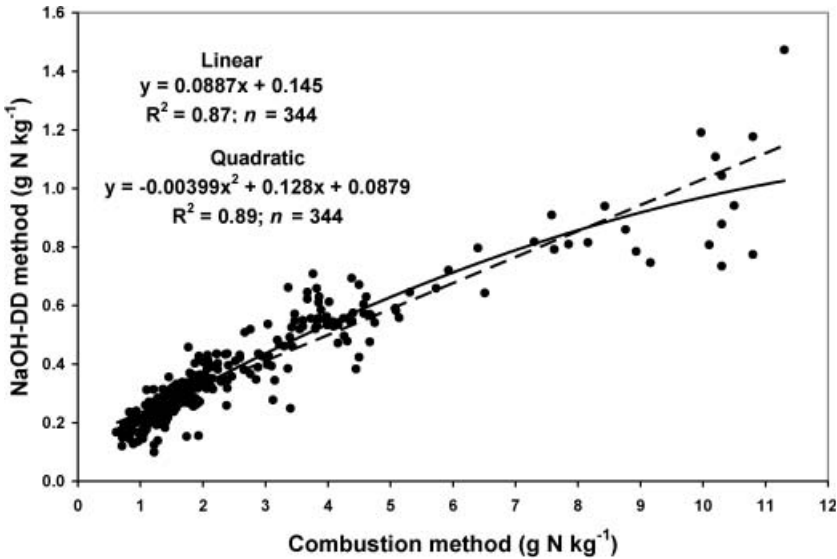


Figure 2. Regression of sodium hydroxide direct distillation (NaOH-DD) NH₄-N against soil total N concentration measured by the combustion method in sample set B.

Therefore, with increases in each of these parameters, a lower proportion of soil total N was extracted by the NaOH-DD method.

Samples in sample set B were categorized into unamended and historically organically amended soils. Regression of NaOH-DD $\text{NH}_4\text{-N}$ against the combustion method within each category resulted in a linear relationships that explained 85 and 90% of the variation in NaOH-DD $\text{NH}_4\text{-N}$ for the unamended and historically amended samples, respectively (Figure 3). The regression for the amended soil samples had a greater intercept and a lower slope compared with the regression for the unamended soils. The NaOH-DD $\text{NH}_4\text{-N}$ as a percentage of soil total N was significantly less in amended than in unamended soils (12 vs. 17%). There was a stronger negative relationship between NaOH-extractable N as a proportion of soil total N and organic carbon (C), clay concentration, and pH in organically amended soils ($r = -0.67, -0.81, -0.76$, respectively) compared with unamended soils ($r = -0.36, -0.39$, and -0.31 , respectively). These findings suggest that differences exist in the organic N compounds in these two subsets and their alkali hydrolyzability.

The lower accuracy in estimation of soil total N in sample set A compared with set B can be attributed to greater clay contents (Table 1) and the presence of clays (i.e., Illite) with a greater capacity for $\text{NH}_4\text{-N}$ fixation in sample set A (Sharifi and Kalbasi 2001). Greenfield (2001) reported that only a small proportion of fixed ammonium (range 0–31%; mean 11%) becomes soluble during alkali hydrolysis. In sample set B, the quadratic relationship between NaOH-DD and soil total N may reflect the possibility of alkali-insoluble N-containing residues present in the organic amendments. Greenfield (2001) tested alkali-insoluble N residues in a wide variety of soils and reported that most of N in these residues is either in form of fixed ammonium or polymeric hexosamine N. There is evidence that alkali-insoluble N residues could also be in the form of polymeric hexosamines derived largely from fungi and insects (Greenfield 2001).

The differences in the slope and intercept of the regression of NaOH-DD $\text{NH}_4\text{-N}$ against soil total N between the two sample sets (Figures 1 and 3) may result from differences in soil clay concentration, clay mineralogy, and/or origin of organic N compounds in these two sample sets. However, NaOH-DD $\text{NH}_4\text{-N}$ was a good estimator of soil total N within each sample set. This suggests that local calibration of the NaOH-DD method will improve estimation of soil total N concentration.

Comparable values of the CV based on three to five field replicates were measured for the NaOH-DD and Kjeldahl methods in sample set A (24 vs. 31%) and for the NaOH-DD and combustion methods in sample set B (17 vs. 19%). This suggests that the NaOH-DD method has a level of precision and repeatability comparable with the Kjeldahl and combustion methods. The high precision of the NaOH-DD method

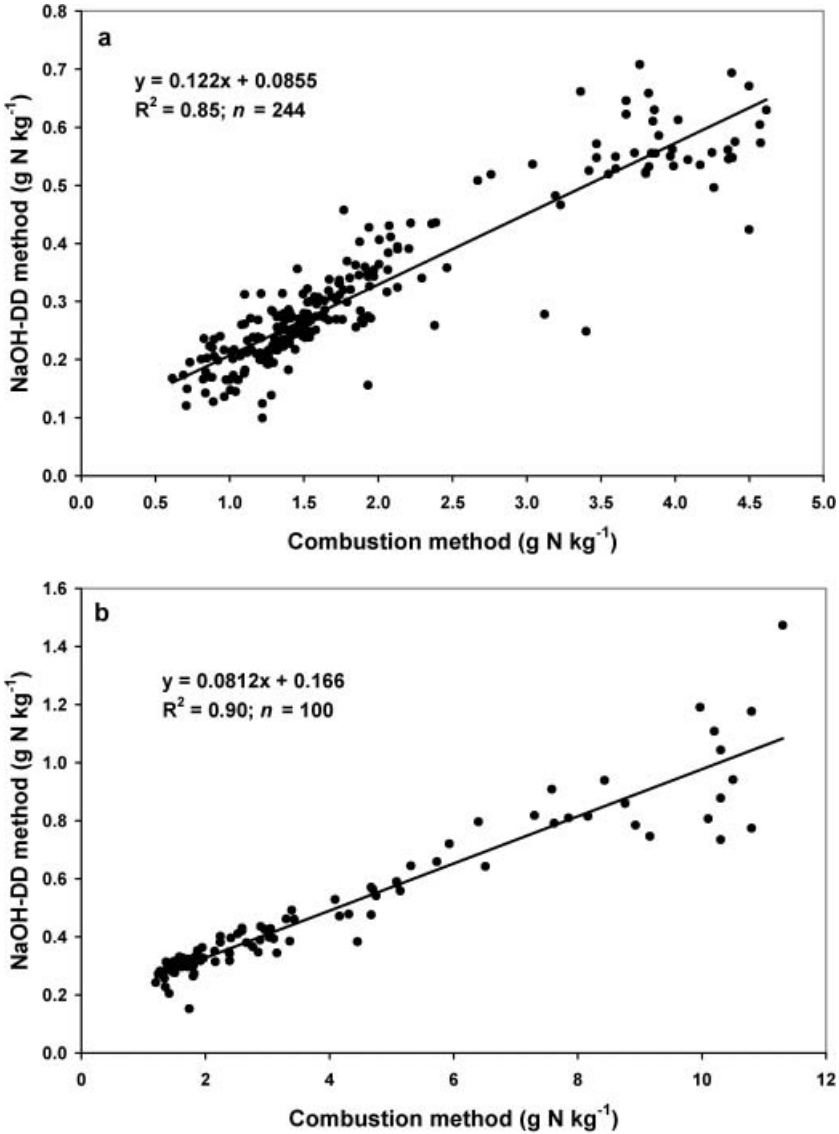


Figure 3. Regression of sodium hydroxide direct distillation (NaOH-DD) NH₄-N against soil total N concentration measured by the combustion method in (a) unamended soils (b) and organically amended soils in sample set B.

may be due in part to the use of a large (5.0 g) soil sample and the exclusion of extraction, filtration, and transfer steps.

A comparison between the regular Kjeldahl and NaOH-DD methods is presented in Table 5. It must be noted that the regular Kjeldahl and

Table 5. Simple comparison between the regular Kjeldahl and sodium hydroxide direct distillation (NaOH-DD) methods for digestion and distillation stages (calculations are based on measuring soil total N in 100 samples)

Parameter	Kjeldahl ^b	NaOH-DD
Sample size (g)	0.25 to 2.0	5
Chemical reagents	Concentrated H ₂ SO ₄ , NaOH, K ₂ SO ₄ , CuSO ₄	NaOH
Digestion temperature	220 °C for 1.5 h; 360 °C for 3.5 h	Not applicable
Digestion plus distillation time (min) ^a	36 h and 37 min	16 h and 40 min
Fume hood required	Yes	No

^aCalculations were based on running the samples using one digestion system (Fisher digestion system, basic model for 20 tubes) and one distillation unit (Fisher DU100 distillation unit) and cooling time of 30 min.

^bRegular Kjeldahl method without pretreatment as described by Rutherford et al. (2007).

NaOH-DD methods differ mainly in the digestion and distillation steps and are the same in the titration step. Elimination of the digestion step in the NaOH-DD method reduces the time for measurement to less than half of that of the regular Kjeldahl method. Replacement of the regular Kjeldahl method with the NaOH-DD method will substantially reduce the amount of chemical wastes and overcome the health and safety concerns over the handling and use of concentrated acid in the regular Kjeldahl method. An additional advantage of the NaOH-DD method is that expensive fumehood facilities are not required.

Caution should be used when analyzing soil samples with high concentrations of NO₃-N. This method, similar to the regular Kjeldahl method, will not completely recover NO₃-N and NO₂-N. The pretreatments suggested for Kjeldahl to include NO₃-N and NO₂-N (Bremner 1996) are also applicable to the NaOH-DD method.

The high correlation of the NaOH-DD method with soil total N measured by either Kjeldahl or combustion methods suggests that the NaOH-DD method can be used to rapidly and accurately estimate soil total N concentration over a wide range of soil properties, land uses, and climate conditions. However, an initial regional validation of the regression equation parameters should be performed before routine use of the NaOH-DD method for this purpose. The NaOH-DD method substantially improved the simplicity, speed, and cost-effectiveness of the soil total N measurements compared with the regular Kjeldahl method, and it can easily be performed in soil-testing laboratories. Additional work is needed to further investigate the nature of the compounds

extracted by this method and the factors that may affect the efficiency of the extraction of organic N by the NaOH-DD method.

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