

Comparison of Total Nitrogen Methods Applied for Histosols and Soil Horizons with High Organic Matter Content

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Abstract: This study was carried out to correlate total nitrogen analyzed by two different methods: Kjeldahl (wet oxidation) and Perkin-Elmer 2.400 Series II–CHN Mode (dry oxidation or combustion) in Histosols and soil surface horizons with high organic matter content, sampled in different regions of Brazil. A positive correlation ($r = 0.95^{**}$) was verified between the methods, showing that Kjeldahl, because of its simplicity, can be used to routinely determine total nitrogen content in the evaluated soils.

Keywords: Histic horizons, soil chemical, tropical soils

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INTRODUCTION

Nitrogen (N) in soils is found in quite heterogeneous chemical species, although it predominates in the organic form, varying from low molar mass compounds up to complex decomposition-resistant substances (Cantarella and Trivelin 2001). The organic matter mineralization process plays an important role in the nitrogen cycle; it is responsible for the transformation of organic N present in plant tissues into simple inorganic forms (Franzluebbers, Hons, and Zuberer 1994). To measure N in soils, when it occurs as ions such as NH_4^+ , NO_3^- , NO_2^- , methods based on distillation or colorimetric reactions are used. The most reactive gases such as NO and NO^2 also make determination easy, which is not the case with N_2O and N_2 (Tedesco et al. 1995).

Among the different methods of total N determination in soils, the Kjeldahl method (wet oxidation) and dry oxidation or the combustion method, also called CHN, are more frequently used. Because of its performance and simplicity, utilization of ordinary equipment, and low cost of analyses, the Kjeldahl method is the most used in routine soil and plant analysis. However, dry combustion semiautomatic devices are also available.

The objective of this study was to compare Kjeldahl (wet oxidation) and Perkin-Elmer 2.400 Series II-CHN Mode (dry oxidation or combustion) methods in the determination of total N in soil samples of Histosols and horizons with high organic matter content, from different regions of Brazil.

MATERIALS AND METHODS

Twenty-one samples of surface horizons, from Histosols and other soils with high organic matter content (with O horizon and histic H), were used. They were collected in areas with different environment, soil usage, and vegetal coverage, from eight Brazilian states.

For the determination of the total nitrogen, two methods were applied: Kjeldahl (Bremner 1996) and Perkin-Elmer 2.400 Series II-CHN. In the first method, the organic N is converted into NH_4^+ through digestion with H_2SO_4 and metals that act as catalyzers [copper (Cu) and selenium (Se)] or that further the conversion and help maintaining high temperatures during digestion (NaSO_4). NH_4^+ is separated after distillation by vapor carrying, resulting from the addition of concentrated NaOH solution to the digestion extract. The ammonia produced is carried by water vapor and collected in a boric acid solution containing the detection substance. The borate is retrotitrated with H_2SO_4 standardized solution. The amount of acid used in the titration is proportional to the N- NH_4^+ present in the sample.

In the dry oxidation or combustion method, the total carbon and nitrogen were determined through dry combustion, using an element analyzer, Perkin-Elmer 2400 CHNS (Jimenez and Ladho 1993), from 5.0 mg (± 0.1 mg) soil samples. Acetonelida was used as reference. A detailed description of the

Perkin-Elmer 2.400 CHNS device and function can be found in Jimenez and Ladho (1993).

The statistical method employed to compare the total nitrogen content obtained from the two methods was linear regression ($Y = b_0 + b_1X$), as suggested by Miller and Miller (1993). The null hypotheses formulated were that the declivity (b_1) was not different from one, and the intercept (b_0) was not different from zero. Such hypotheses were tested by means of the confidence limits calculation at 95% for both coefficients. The software adopted in the statistical analysis was the data analysis tool in Microsoft Excel 97.

RESULTS AND DISCUSSION

Total N values measured with both methods are presented in Table 1 and show only small variations for each soil horizon. For the Kjeldahl method, the

Table 1. Total nitrogen determined by Kjeldahl and Perkin-Elmer 2400 CHNS methods in Histosols and soils with histic horizon

Soil samples	N Perkin-Elmer 2400 CHNS (g/kg)	N Kjeldahl (g/kg)
DF1 Hap	5.8	4.7
DF1 Ha1	6.6	6.0
DF1 Ha2	8.0	6.6
ES1 Hai	9.7	8.9
MG1 2Hai	17.2	16.8
MG2 Hai1	7.7	4.9
MG2 Hai2	11.3	14.1
MS2 Hai	8.0	7.0
MS2 2Ha	7.6	7.3
PR2 Hap1	6.5	5.0
PR2 Hap2	6.0	6.0
PR2 Hap3	6.7	5.8
RJ3 Hai1	23.3	22.5
RJ3 Hai2	18.5	19.2
RJ3 Ha	17.8	21.5
RJ4 Hap1	5.2	4.2
RJ4Hap2	4.1	3.3
RS3 Ha1	5.1	5.2
RS3 Ha2	2.5	4.5
SP1 Hp1	9.4	9.5
SP1 Hp2	3.9	2.6

Notes: DF: Distrito Federal, ES: Espírito Santo, MG: Minas Gerais, MS: Mato Grosso Sul, PR: Paraná, RJ: Rio de Janeiro, RS: Rio Grande do Sul, and SP: São Paulo.

lowest value of N obtained was 2.6 g kg^{-1} and the highest 22.5 g kg^{-1} . For the Perkin-Elmer 2.400 CHNS method, values ranged from 2.5 g kg^{-1} to 23.3 g kg^{-1} , the highest values belonging to the RJ3 Hai1 soil horizon.

A comparison between Kjeldahl and Perkin-Elmer 2.400 CHNS methods for measuring C and N in weathered Brazilian soils showed only small differences between values of total C and N (Pérez et al. 2001). This similarity between methods was attributed to the soils' low content of 2:1 clays, such as the smectite, that may adsorb the ion NH_4^+ internally and to the low soil contents of NO_3^- and NO_2^- , which are rapidly leached in tropical environments.

A significant correlation ($R^2 = 0.95^{**}$) was observed between the two methods of total N determination (Figure 1). The relationship between the nitrogen values measured by Dumas and Kjeldahl methods is described by this equation: Perkin-Elmer 2400 CHNS = $1.0799 \times (\text{total N Kjeldahl}) - 0.9809$. The Kjeldahl method presented values statistically comparable to results obtained from the dry oxidation method. Thus, the first may be favored in routine total N analyses, even in the soils with high organic matter content such as Histosols.

A study with 250 soils, collected in estuarine marsh areas of North Carolina, showed a linear correlation (0.986^{**}) between total N content determined through dry oxidation and Kjeldahl methods (Craft, Seneca, and Broome 1991). The relationship between the two methods was described by the following equation: N Perkin-Elmer 2.400 CHNS = $1.048 \times (\text{total N Kjeldahl}) - 0.010$.

The equivalence between total N determination methods is verified through the regression analysis with the confidence interval as shown by the values of coefficient b_1 and b_0 (Table 2). The value of coefficient b_1

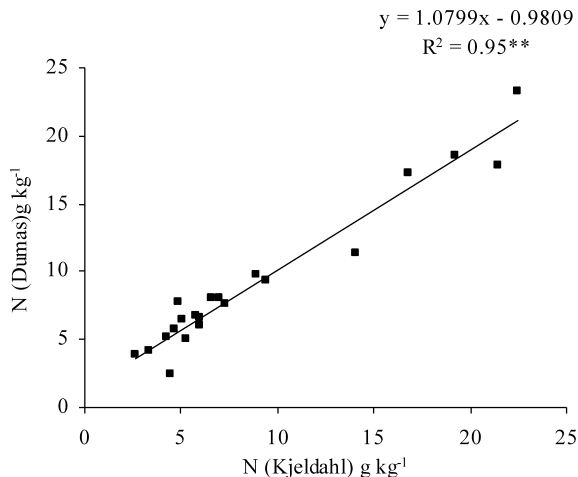


Figure 1. Correlation between soil total N measured by Perkin-Elmer 2400 CHNS and Kjeldahl methods, in Histosols and soils with histic horizon.

Table 2. Linear regression intercept and slope ($Y = b_0 + b_1X$) for the total N determinations

Coefficients	Minimum	Intermediate	Maximum
Linear	-2.29	Intercept (b_0) -0.98	0.33
Angular	0.96	Slope (b_1) 1.08	1.20

(angular coefficient) remained between 0.96 and 1.20, and the intercept average value (b_0) showed a linear coefficient varying from a negative value (-2.29) to 0.33. These variations were not significant; therefore, the methods are equivalent.

CONCLUSIONS

The results obtained through the evaluated methods showed values statistically compatible, indicating that the Kjeldahl method, because of its performance and simplicity, may be preferred for the routine analysis of total N, even for soils with high organic matter content.

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