NOTES

AN ORGANIC CARBON-ORGANIC MATTER CONVERSION EQUATION FOR PENNSYLVANIA SURFACE SOILS[1]

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ABSTRACT

Low temperature ashing was used to determine the organic matter content of soils by weight loss. This technique utilizes oxygen excited by radiofrequency energy to effect oxidation at relatively low temperatures. Ashing at 375C was used for samples high in organic matter. Corrections were applied for weight loss of inorganic constituents and for incomplete oxidation of organic matter. The following regression equation was developed to convert organic carbon percent to organic matter percent in Pennsylvania surface soils:

Percent organic matter = 0.35 + 1.80 × percent organic carbon.

Additional Key Words for Indexing: low temperature ashing.

ORGANIC matter content of soils is usually determined indirectly by converting the analytically determined organic carbon percentage to percent organic matter through arithmetic conversion. Most often the percent organic carbon is multiplied by a factor of 1.724. Many investigations have indicated, however, that this factor underestimates the quantity of organic matter in soils. Summarizing some American work, Broadbent (1953) concluded that a conversion factor of 1.9 for surface soils and 2.5 for subsoils would be more appropriate. DeLeenheer, Van Hove and Van Ruymbeke (1957) presented evidence that a factor of 2 should be used for Belgian soils with more than 1% organic matter. Howard (1965) reports factors of 1.97 to 2.07 for mull layers and 1.77 to 1.95 for mor layers in British soils. Ponomareva and Plotnikova (1967) used ratios of extracted humic and fulvic acids to conclude that a factor of 2 would be generally more acceptable than 1.724. Loftus (1966) stated that factors of 1.8 for organic soils and 2.2 for surface horizons of mineral soils would be best for a few Pennsylvania soils on which he worked.

The present study was conducted to develop an organic carbon to organic matter conversion factor for Pennsylvania soils using weight loss from ashing, corrected for inorganic weight loss, to measure organic matter concentration.

METHODS

Samples were collected from Ap, 02, A1, A2 and Bh horizons of soils chiefly in the northern two-thirds of Pennsylvania. The 48 samples used represent the following soil series:

Albrights	Middlebury
Cookport	Morris
Dekalb	Natalie
Frankstown	Oquaga
Gresham	Ravenna
Klinesville	Sciotoville
Leck Kill	Vanderlip
Leetonia	Volusia
Lobdell	Wellsboro
Meckesville	Wurtsboro

These soils range from well drained to somewhat poorly drained, and forested and cultivated sampling sites were included. None of the samples were calcareous. All soil samples were ground to pass a 60-mesh screen and dried at 105C prior to the following treatments:

Muffle Furnace—850C—Samples weighing approximately 2 g were placed in porcelain crucibles, covered loosely, heated to 850C for 90 min, cooled, and reweighed.

Muffle Furnace—375C—Samples were weighed into crucibles as before and heated to 375C for 28 hours, cooled, and reweighed.

Low Temperature Ashing—Samples weighing 1 g were subjected to oxidation in a Tracerlab LTA—600 Low Temperature Asher (Tracerlab, 2030 Wright Avenue, Richmond, Calif.). This method was chosen because it is designed to remove organic material without appreciably altering inorganic materials left in the ash. In this apparatus the sample is under pressure of approximately 1 mm Hg and is in contact with activated oxygen excited by a radiofrequency electromagnetic field which facilitates the oxidation of organic matter at a relatively low temperature. The ashing temperature may vary from one sample to another, but most likely is in the range of 100–200C. Samples were removed from the asher twice a day, weighed and stirred. Ashing was continued for a total of 4 to 5 days until weight loss had essentially stopped. Five oxidation chambers are available making it possible to complete five samples every 5 days.

Subsoil samples with negligible amounts of organic matter were ashed by each of the three treatments to measure the loss due to inorganic materials. For low temperature ashing 50 mg of tartaric acid were added to each low organic sample to ensure that it would reach temperatures comparable to samples containing significant amounts of combustible materials.

The original sample and the resulting ash from all three methods were analyzed for carbon with the Fisher Induction Carbon apparatus using cupric oxide in the metal accelerator mixture (Young and Lindbeck, 1964).

RESULTS AND DISCUSSION

Weight loss on ashing of the low organic subsoil samples occurred with all three methods necessitating a correction for inorganic weight loss in samples containing organic matter. Most inorganic weight loss can be attributed to loss of structural water from clay minerals and hydrated oxides. Since these usually occur primarily in the clay-size fraction, the correction applied was based on the percentage of clay in the sample. Based on an average of 10 subsoil samples, it was calculated that a soil loses 1.5% of the weight of its clay during low temperature ashing. The same 10 soils lost an average of 3.8% of their clay weight by ashing at 375C. Seven of these same soils showed an average of 17.4% weight loss from clay by ashing at 850C. For example, a soil containing 20% clay and no organic matter

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would lose 0.30% of its weight by low temperature ashing, 0.76% by ashing at 375C, and 3.5% at 850C.

Corrections based on these figures are subject to errors. Inorganic material coarser than clay size may lose more weight in some soils than in others. Variations in the mineralogy of the clay also affect weight loss and although the subsoil samples were taken from the same group of soils as the samples containing organic matter, the mineralogy in surface horizons tends to be somewhat different from subsoil horizons. It is very important, then, to reduce the inorganic weight loss to a minimum so that this source of error will be reduced. Low temperature ashing is superior in this respect. Ashing at 850C is unacceptable for most surface soils because the correction is often more than half the total weight loss. Ashing at 375C is intermediate in inorganic weight loss, and is acceptable for soils with high organic matter percentages.

In order to calculate organic matter content from weight loss on ashing the following formula was used:

$$\text{Percent organic matter} = \frac{\text{percent weight loss on ashing} - A}{1 - A/100}$$

where A is the calculated percent weight loss of the mineral portion of ashing based on the percent clay in the sample and on the average inorganic weight loss from 10 subsoil samples. The denominator of the formula is necessary since the clay percentage is based on the mineral fraction only and not the total sample.

Completeness of combustion is also a problem which must be considered. Analysis of the ash for residual carbon indicated that 850C ashing effected essentially complete removal of organic matter. Ashing at 375C and low temperature ashing, however, left varying amounts of residual carbon usually proportional to the original carbon content of the sample. Residual carbon ranged from less than 0.1% to 1.5% of the original sample weight. Percent residual carbon was multiplied by 2 to convert to an organic matter percentage and this was added to the percent weight loss. Some degree of error is certainly involved but it is tolerable because residual organic matter is a small percentage of the total organic matter in each case.

Results from low temperature ashing were used to determine the relationship between organic carbon and organic matter. For samples above 30% organic matter results from 375C ashing were also used because this was a more economical means of obtaining data than low temperature ashing and because any inorganic loss on ashing would be small relative to the total organic matter. No inorganic weight loss correction was made for these high organic samples because clay content was not determined and is believed to be low.

Figure 1 shows organic matter plotted against organic carbon. A regression equation was calculated with the following result:

Percent organic matter = 0.35 + 1.80 × percent organic

carbon. This equation indicates that for Pennsylvania soils the customary conversion factor of 1.724 is too low par-
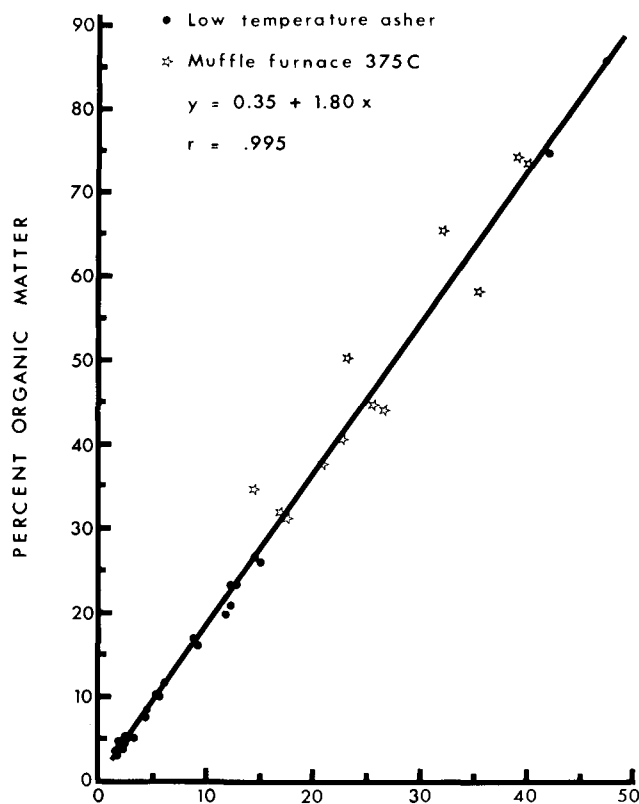

Fig. 1—Percent organic matter by loss on ashing vs. percent organic carbon by induction furnace (Young and Lindbeck, 1964). All organic matter percentages are corrected for residual organic matter and samples below 30% organic matter were corrected for inorganic weight loss.

ticularly for horizons with less than 5% organic matter such as Ap horizons. For these horizons a more suitable conversion factor would be 2.0. Use of a single conversion factor for all Pennsylvania soils would not be valid because high organic surface horizons of forest soils have lower ratios of organic matter to organic carbon on the average than do samples with less organic matter. Thus, a range of conversion factors or the regression equation should be used for the conversion of organic carbon data to percent organic matter of surface soils.

The lowest organic carbon concentration of samples shown in Fig. 1 is 1.4%. When organic carbon percentages approach zero, as they do in many subsoils, the results of the equation are obviously incorrect since it states that a sample containing no carbon has 0.35% organic matter. If the organic matter percent were known for a group of samples ranging from 0 to 1.4% carbon, it is supposed that the slope of the line would steepen at the lower values and intercept at zero. The conversion factor for subsoils, then, would be expected to be somewhat above 2 as suggested by Broadbent (1953).

ACKNOWLEDGMENT

R. P. Matelski and members of the soil characterization group at The Pennsylvania State University for assistance and suggestions.

LITERATURE CITED

1. Broadbent, F. E. 1953. The soil organic fraction. Advance. Agron. 5:153–183.
2. DeLeenheer, L., J. Van Hove, and M. Van Ruymbeke. 1957. Determination quantitative de la matiere organique du sol. Pedologie 7:324–347.
3. Howard, P. J. A. 1965. The carbon-organic matter factor in various soil types. OIKOS 15:229–236.
4. Loftus, Nelson S., Jr. 1966. The contribution of extractable humic colloids to the exchange capacity of surface soils. Ph.D. Thesis. Pennsylvania State Univ. Univ. Microfilms. Ann Arbor, Mich. (Diss. Abstr. 27:3752B. Order no. 67-5939).
5. Ponomareva, V. V., and T. A. Plotnikova. 1967. Data on the degree of intramolecular oxidation of humus in various soil groups (problem of the carbon to humus conversion factors). Soviet Soil Sci. 924–933.
6. Young, J. L., and M. R. Lindbeck. 1964. Carbon determination in soils and organic materials with a high-frequency induction furnace. Soil Sci. Soc. Amer. Proc. 28: 377–381.

A SIMPLE X-RAY FLUORESCENCE TECHNIQUE FOR THE DETERMINATION OF IRON AND MANGANESE IN SOILS AND CONCRETIONS[1]

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Abstract

A simple technique for the determination of Fe and Mn in concretions and soils was developed using X-ray fluorescence. A 25 mg sample was suspended in a 1% glue-methyl alcohol solution and transferred to a 4.25-cm circle of filter paper. The filter paper containing a thin layer of uniformly distributed sample was placed in an X-ray emission spectrograph for analysis, and highly reproducible and accurate results were obtained. Precision was achieved over a wide range of Fe and Mn oxide contents.

Additional Key Words for Indexing: X-ray emission, iron and manganese analysis.

SEVERAL TECHNIQUES have been described in the literature for X-ray fluorescence analysis of soil and rock materials. However, most of these involve either fusion with a flux such as lithium tetraborate or pressing samples into a disk with relatively high pressures, Beavers (1960), Bertin and Longobucco (1962), Welday et al. (1964), and Campbell et al. (1966). These sample preparation techniques result in "thick" samples. In the case of pressed samples, reproducible and accurate results are achieved with some difficulty because of matrix effects. The use of a flux reduces these matrix effects, but such techniques are somewhat undesirable because of the high fusion temperatures required, mixing problems, and breakage or cracking of the glass disks.

The use of so called "infinitely thin samples" for X-ray emission analysis has been discouraged from a theoretical point of view because variations in sample thickness affect the results as pointed out by Liebhafsky et al. (1960). However, there has been an increasing number of papers being published using "thin film" techniques (Campbell, 1966; Johnson, 1968). This has been largely the results of attempts to find a more sensitive method in which matrix effects and self-absorption have been reduced enough to allow measurement of trace quantities of a particular element.

In a current study to determine the total Mn and Fe content of concretions in soils with differing internal drainage characteristics, it was desirable to obtain rapid and accurate measurements of a large number of samples. Previous methods require complete dissolution of the sample and subsequent analysis by colorimetry, atomic absorption or flame-emission spectroscopy. The use of the usual orthophenanthroline method for Fe determination (Jackson, 1958) would require a series of dilutions in order to obtain a concentration within the linear portion of the standard curve. The use of atomic absorption for the determination of Mn is unattractive because of difficulties of extractions and possible interferences. The method described below provides a rapid and accurate measure of Fe and Mn.

Materials and Equipment

Fe and Mn Standards—Reagent grade Fe_2O_3 and MnO_2 were used as primary standards and were 99.6% and 99.8% pure, respectively. These oxides were oven dried at 110C and sieved through a 300-mesh screen. National Bureau of Standards sample numbers 76 and 77 were oven dried at 110C prior to use.

Sample Pretreatment—Soil and Fe-Mn concretions from Maury silt loam, A3 horizon were finely ground in a plastic cell with a "Wig-L-Bug" grinder and were oven dried at 110C.

Filter Apparatus and Suspending Solution—A Millipore Filter apparatus, modified with a straight wall top, equipped with Whatman no. 42 filter paper was used in the preparation of the thin layers of sample for X-ray fluorescence analysis. Samples were suspended in either 1% glue (Duco cement) in methyl alcohol or a 3% gelatin-H_2O solution prior to filtration.

X-Ray Instrument—A General Electric XRD-5 X-ray emission spectrograph was used in the analysis. A Pt target X-ray tube operated at 50 kv and 40 ma was employed as the primary X-ray source. A LiF analyzing crystal in conjunction with a PR gas-flow proportional counter tube was used in the detection of the emitted X-rays. A pulse-height discrimination system was used with settings of $\Delta E = 10$ volts and $EL = 2$ volts and counting rates were obtained over a 100-sec time interval. The 2θ angles used in the analysis were 57.52° 2θ for Fe Kα; 62.97° 2θ for Mn Kα; and 61.00 2θ for background radiation.