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Application News
TOC

May 2004

Total Organic Carbon In Soil

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Introduction

Soil organic carbon (SOC), which is a major component of soil organic matter, is considered perhaps the most important indicator of soil quality and productivity[1]. A common method to determine SOC is the modified Walkley-Black wet oxidation method, which involves reduction of chromic acid by organic compounds, and subsequent determination of unreduced chromic acid by titration with ferrous ammonium sulphate[2]. As there is growing interest to eliminate the use of chromic acid because of safety and disposal issues, the method that utilizes dry combustion of carbon compounds is becoming popular[3]. Also, the dry combustion method measures total carbon (TC) whereas the chromic acid method determines only oxidizable carbon. Currently, there are many instruments that utilise the dry combustion method which further simplifies the procedure and shortens the analysis time to about five minutes per measurement.

There are two types of dry combustion instrument - indirect and direct (Figure 1).

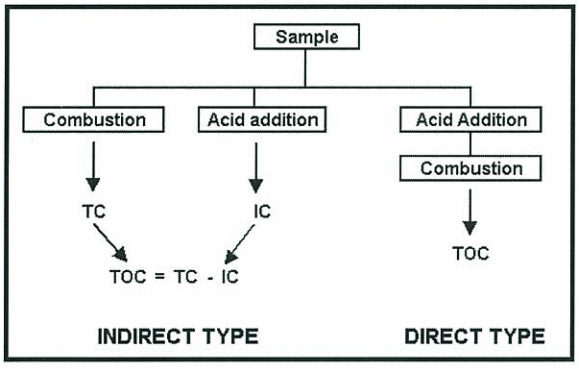


Figure1: Indirect and direct dry combustion instruments

In the indirect dry combustion instrument, there are two furnace chambers and three steps are involved in the measurement of SOC:

a) TC measurement - a sample is combusted in a high temperature furnace chamber where all forms of carbon are converted to carbon dioxide (CO_2) which is then measured, usually with an infrared detector.
b) inorganic carbon (IC) measurement - the sample is reacted with acid in a separate furnace chamber to release CO_2 which is also measured.
c) the difference between TC and IC is SOC, which is also known as total organic carbon (TOC).

The direct dry combustion instrument is equipped with only one combustion chamber. It measures TOC in two steps:

a) IC in sample is first removed by reacting the sample with acid.
b) the acid-treated sample is then combusted in a furnace chamber to release CO_2 which is then measured with an infrared detector to obtain TOC results.

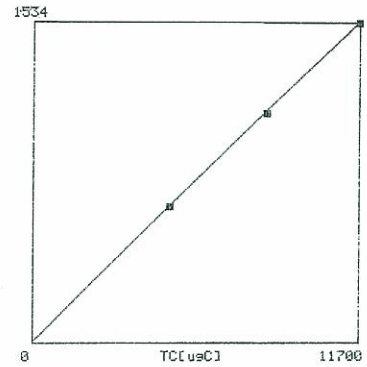
However, the accuracy of TOC results may be affected if the IC is not sufficiently removed from the sample. Hence, it is better to utilise the indirect dry combustion equipment to measure TOC. Occasionally IC result, which represents carbonate content in soil is also required. To obtain IC results using the direct dry combustion instrument, the sample, without any acid-treatment, is combusted to obtain TC. The difference between TC and TOC is IC.

Here, we described a method to measure TOC in soil using an indirect dry combustion instrument, the Shimadzu SSM-5000A.

Methods & Results

Glucose ($C_6H_{12}O_6$) and sodium carbonate (Na_2CO_3) were used as the TC and IC standard respectively. For measurement of TC, each sample (about 100 mg) was placed in a ceramic sample boat which was then transferred into a 900°C catalytic (mixture of cobalt oxide and platinum) combustion chamber inside the SSM-5000A to be oxidised into CO_2 with the aid of oxygen (O_2) gas. The CO_2 was then carried by O_2 carrier gas into the short cell of a non-dispersive infrared (NDIR) detector in the TOC-VCSH and measured. This was followed by measurement of IC - here, each sample (about 100 mg) was also placed in a ceramic sample boat. Then, 0.5 ml 85% phosphoric acid (H_3PO_4) was added to the sample using a dispenser before the sample boat was transferred into a 200°C combustion chamber, to produce CO_2 which is also measured using the NDIR detector in the TOC-VCSH. For each standard and sample, the measurements were repeated at least two times to achieve coefficient variation (CV) of 2% or standard deviation (SD) of 0.1. The average results, %CV and SD were automatically calculated by the software.

The TC and IC calibration curve results are shown in Figure 2 and 3 respectively.

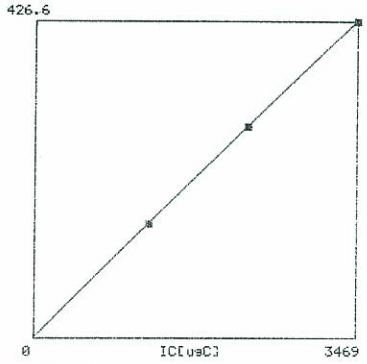


CALIBRATION CURVE : SSM-TC0 3-POINTS
[DATA]

STD	AREA	#	SD	CV %
40.00%	646.6	2	4.28	0.67
40.00%	1098	2	3.44	0.32
40.00%	1534	2	12.5	0.82

PROTECT : ON
CAL CURVE FORM : LINEAR REGRESSION
SHIFT OPTION : SHIFT TO ORIGIN
R^2 : 1.0000
DATE : 03(MAR)-02-2004

Figure 2: TC calibration curve



CALIBRATION CURVE : SSM-IC0 3-POINTS
[DATA]

STD	AREA	#	SD	CV %
11.30%	154.9	3	0.17	0.10
11.30%	285.0	2	0.14	0.05
11.30%	426.6	2	4.70	1.10

PROTECT : ON
CAL CURVE FORM : LINEAR REGRESSION
SHIFT OPTION : SHIFT TO ORIGIN
R^2 : 1.0000
DATE : 03(MAR)-03-2004

Figure 3: IC calibration curve

Overall, a good repeatability of within 2% CV or 0.1 SD was achieved for all measurements. Good linearity was also achieved with regression of more than 0.9990.

Following this, two dried and homogenised soil samples were measured with SSM-5000A and the TOC results are shown in Table 1.

Sample	TC %(w/w)	IC %(w/w)	TOC = TC – IC % (w/w)
Mineral Soil	0.96	0.00	0.96
Peat Soil	7.28	0.00	7.28

Table 1: TOC in soil

Conclusions

The SSM-5000A can be used to determine TOC in soil samples using a shorter analysis time compared to the wet oxidation method. Unlike the direct dry combustion instruments that only have one combustion chamber to measure TC (and TOC if the sample is pre-treated with acid), the Shimadzu SSM-5000A can provide both TC and IC results conveniently. Analysis is also easier as pre-treating the sample with acid to remove IC is not required prior to analysis.

References

(1) http://www.fao.org/
(2) Soil Survey Laboratory Methods Manual (1996). United States Department States of Agriculture, Natural Resources Conservation Services, National Soil Survey Centre.
(3) Recommended Soil Testing Procedures For The Northeastern United States (1995). Northeastern Regional Publication No 493. Second Edition.

Acknowledgements

The authors would like to thank Dr Khalid Haron from Malaysian Palm Oil Board for kindly providing the soil samples.

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[IPDL] Printed in Singapore.