

## Short Communication

# A RAPID ESTIMATION OF ORGANIC CARBON IN SILTY LAKE SEDIMENTS

J.J. LYNCH, R.G. GARRETT and I.R. JONASSON

*Geochemistry Section, Geological Survey of Canada, Ottawa, Ont. (Canada)*

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## ABSTRACT

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A rapid and convenient method is described for the estimation of active organic carbon in silty lake sediments collected in regional geochemical surveys. The method utilizes measurement of optical density at 500 nm, of a 4 M nitric acid – 0.1 M hydrochloric acid leach of a sediment sample, and has a precision of  $\pm 26\%$  as determined from separate replicate analyses. An attractive feature of the method lies in its use of the same leach solution on which atomic absorption analyses of trace metals may be made.

## INTRODUCTION

A rapid and convenient method is described for the estimation of organic carbon in silty lake sediments. It was developed specifically for a large-scale regional geochemical survey in the Canadian Arctic recently completed by the Geochemistry Section of the Geological Survey of Canada (Allan et al., 1973). Samples of silty lake sediments were collected from areas of sparse tree cover and from tundra terrain. In all, over 4000 samples were collected which were subsequently analysed for organic carbon by this method. Whole sediments, when first collected from these regions, often show little or no physical evidence of organic carbon, but when silt-clay fractions are separated for analysis, the proportion of organic carbon present as humic materials may be much higher in this fine-grained material. It is clearly of considerable importance in making a correct geochemical interpretation of trace element data derived from the sieved fraction, to have a knowledge of, at least, relative amounts of organic carbon present. The strong sequestering action of humic materials on metal cations is well known and need not be described here.

The method has not been applied to similar samples from other climatic zones of Canada where lake sediments are often considerably richer in organic materials. Further evaluation would be necessary before the technique could be directly applied to such samples.

## ANALYTICAL METHOD

It was observed that during the preparation of acid leach solutions of lake sediments sieved to minus 250 - mesh that different samples exhibited varying intensities of a brown colouration ranging from colourless to very dark brown. An examination of spectrographic data revealed that these variations could not be related to abnormal concentrations of coloured ions, e.g., titanium and iron. In addition, iron does not exhibit any colouration in a nitric acid medium. Hence, it was considered that the colour was due to organic matter present in the sample and that the measurement of this colour would provide an estimate of the organic matter. The principle of the method is the same as that used for the determination of carbon in steel (see for example: Furman, 1962, pp. 293–295).

Spectral curves of a number of coloured sample solutions revealed no peak in the visible portion of the spectrum (360–650 nm) but rather showed a continually increasing extinction coefficient towards the ultra-violet end of the spectrum. In order to make routine measurements of test solutions, 500 nm was chosen as the optimum wavelength to cover the useful concentration range of organic material in this particular suite of samples. Either lower or higher wavelengths could be chosen.

Routine measurements of colour intensity were performed as follows:

(1) A 400-mg sample of air-dried sieved (minus 250-mesh) sample was placed in a 18 × 150 mm pyrex test tube calibrated at 20 ml.

(2) 6 ml of an acid mixture (4 M HNO<sub>3</sub>–0.1 M HCl) was added from an automatic dispenser.

(3) The sample was placed in a hot water bath at 90°–95°C and heated for 1½ hours with intermittent shaking.

(4) After heating, the sample solution was cooled and diluted to 20 ml with deionized distilled water and mixed.

(5) Metal analyses were performed on this solution and then it was allowed to settle for 2–3 days until perfectly free of suspended matter. (Alternatively, the sample could be centrifuged).

(6) After the leachate was clarified its optical density was determined using a Bausch and Lomb Spectronic 20 colorimeter.

An attractive feature of the method lies in the fact that the same leach solution may be used for atomic absorption analyses of trace metals present in the sample. Thus, an extra weighing and acid extraction is not required for the estimation of organic carbon.

It was found that the 18 × 150 mm test tube could be used as the cuvette since it fits quite snugly in the ¾ " cell holder of the colorimeter. It was also found that test tubes from the same packing case were sufficiently well-matched that no cell corrections were necessary for the purpose of this work.

In order to determine the relationship between optical density and organic matter, 55 weighed silt samples were dried at 120°C for 24 h and the weight loss determined. The samples were then placed in a cold muffle furnace and brought up to 800°C over 3 h. This temperature was held for 30 min. After cooling, the samples were weighed again and the percentage weight loss at 120°C and at 120–800°C determined.

## RESULTS AND DISCUSSION

To study the interrelationships between the data, correlation coefficients and plots were calculated for both the raw variables and for the variables after logarithmic transformation. The plots show an apparently linear relationship between optical density and the percentage weight loss between 120°C and 800°C after both these variables have been logarithmically transformed (Fig. 1). Plots of these two variables untransformed, and mixed log-linear plots, showed curvilinear relationships. The correlation coefficient of 0.93 for the logarithmic plot of optical density versus percentage weight loss between 120°C and 800°C based on 55 samples is considered highly significant (Table I).

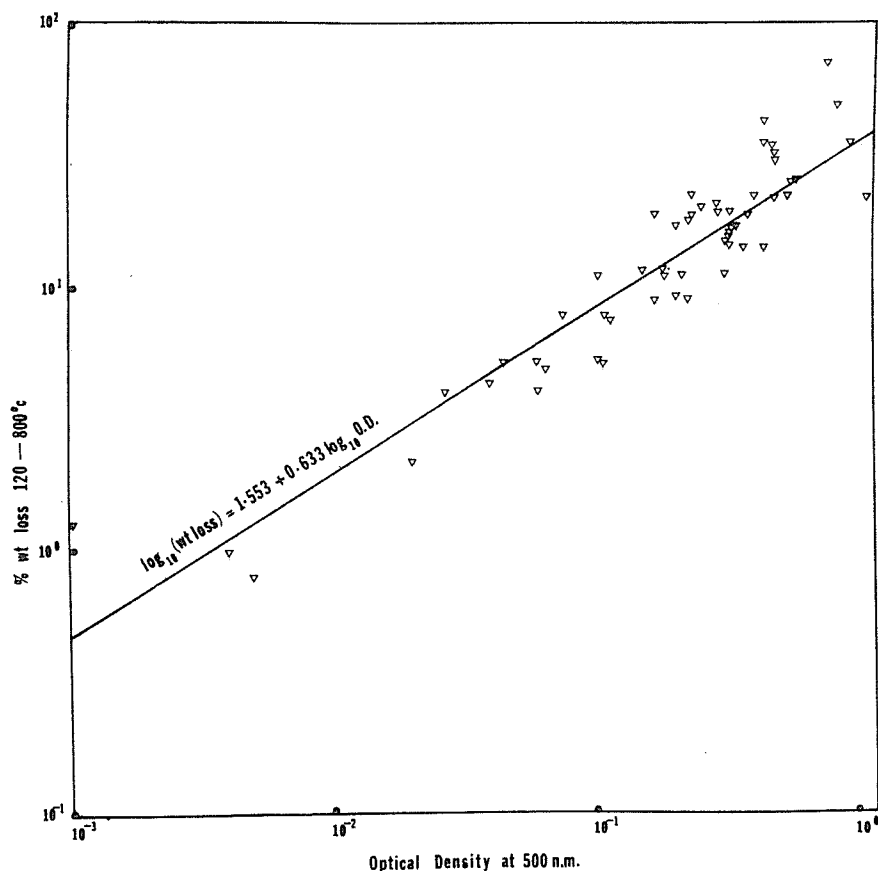


Fig. 1. Regression equation for the plot of optical density vs. percentage weight loss 120–800°C.

The percentage weight loss values over three temperature ranges all correlate well with optical density; however, those for total loss and loss between 120°C and 800°C are somewhat better than that for loss to 120°C. It is considered that the weight loss to 120°C can largely be accounted for by water, and that the correlation with optical density is dominantly due to the labile water held in the humic material.

The possibility that the percentage weight loss on heating to 800°C could be partly due to the decomposition of calcite (825°C) was investigated by determining total Ca in the

TABLE I

Correlation coefficients between  $\log_{10}$  transformed data

	1	2	3	4	5	6
1. Optical density	1.00	0.85	0.93	0.91	0.08	0.30
2. % wt. loss 120°C		1.00	0.79	0.85	-0.11	0.18
3. % wt. loss 120–800°C			1.00	0.98	0.00	0.18
4. Total % wt. loss				1.00	-0.01	0.18
5. Ca total					1.00	0.42
6. Fe total						1.00

Critical value of  $r$  (97,0.95) = 0.168.

samples. As can be seen in Table I the correlations of Ca with optical density and the various percentage weight losses are insignificant. The correlation of optical density with total Fe (Table I) is seen to be minor and is probably related to chemical associations between iron and humates rather than to a direct contribution to optical density by ferric ion complexes. In this respect iron is not different from any other transition metal cation in its affinity for strongly complexing organic ligands such as those found in natural organic materials.

The precision of the method as determined at the 95% confidence level on 97 separate, replicate analyses of a control sample of lake sediment was  $\pm 26\%$  at the 0.035 level of optical density.

## CONCLUSIONS

The method is advantageous, not only in terms of rapidity, but also because the active organic groups which bind the metals are liberated by the acid leach along with the metals on oxidative attack. It is a measure of this organic content of the samples which is relevant to the interpretation of exploration geochemical data. It is believed that the proposed method has advantages over the direct determination of total organic carbon.

## REFERENCES

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