

Re-assessment of the titration method for determination of organic
carbon in recent sediments

by

Osman A. El-Rayis

Oceanography department, Faculty of Science, Alexandria University
Alexandria - Egypt.

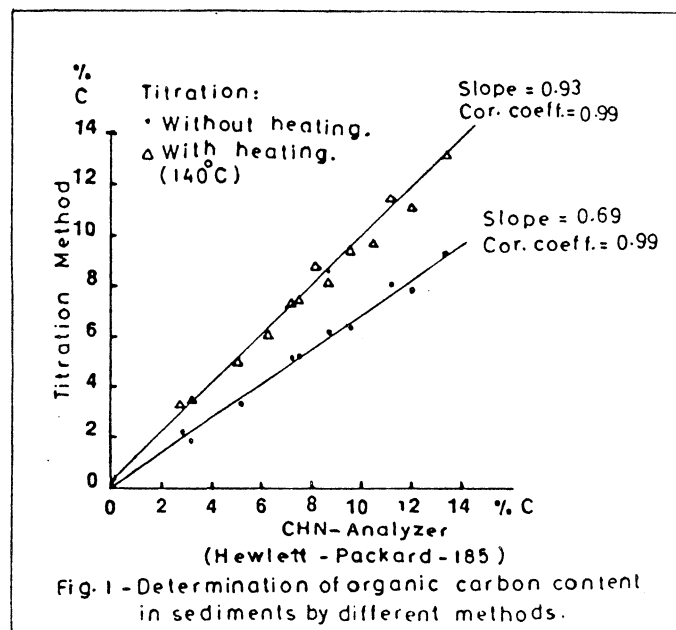
Abstract: The auto-exothermal heating produced in Gaudette et al., 1974 method of determination of organic carbon in lacustrine sediments from lake Windermere, England, is insufficient to complete the wet-oxidation reaction of the organic material. External heating was necessary to complete the reaction. The use of phosphoric or sulphuric acid for washing the sediments instead of acetic acid is recommended.

Résumé : *L'auto-réchauffement exothermique qui se produit durant la détermination du carbone organique dans les sédiments lacustres du lac Windermere (Angleterre), selon la méthode de GAUDETTE et al., 1974, est insuffisant pour l'oxydation complète, en milieu humide, des substances organiques. Un réchauffement externe est indispensable pour mener à bien la réaction.*

LECO and CHN-analyser are precise, but fairly expensive equipment used for determination of organic carbon in the sediments. Small laboratories may not be able to purchase such equipments. In expensive techniques such as that described by Gaudette et al., 1974 are therefore desirable. This technique utilizes exo-thermal heating, potassium dichromate and concentrated sulphuric acid for oxidation of the organic carbon of the sediment samples, the excess dichromate is back titrated with ferrous ammonium sulphate.

This titration method was applied on standard lacustrine, carbonate-free, sediments from lake Windermere and river Rydal, England, of organic carbon content values ranging between 2 to 13% measured by CHN-Analyser

(185 Hewlett-Packard), Liverpool University, and gave values 30% less than those of the CHN-analyser. The difference is significant, particularly in sediments of organic carbon content greater than 2%. The auto-thermal heating for the reaction seems to be insufficient to complete the oxidation process.



In the present paper, when the oxidation reaction was externally heated for 30 minutes at 140°C, the results come in good agreement with those of the CHN-Analyser. The optimum temperature for oxidation reaction in Gaudette *et al* method was 108°C, dropping to 55°C after 10 minutes. Details of the titration method, reagents, procedure and calculation, are to be found in Gaudette *et al*, 1974.

The analytical results for the CHN-analyser, Gaudette *et al* method (without and with external heating) are shown in fig 1. Penta-replicate determinations of a sediment sample from south east of Liverpool Bay, England with the modified method were made (1.1%) and gave a coefficient of variation of 9.3%.

The good agreement between the modified titration method and the CHN-combustion method, therefore allow the modified Gaudette et al method to be used for the determination of organic carbon in future sediment studies. Earlier results obtained by the unmodified method therefore should be corrected by a factor of 1.4. It is also recommended to use dilute mineral acid such as sulphuric acid instead of acetic acid (used by Gaudette et al. 1974) during measurements of organic carbon in sediments on LECO or CHN-analyser.

Reference:

Gaudette, H.E, W.R. Flight, L.Toner and D.W. Floger, 1974.

An inexpensive titration of organic carbon in recent sediments,
J. Sedimentary Petrology. Vol.44(1):249-253.

