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Walter E. Dean

Journal of Sedimentary Research (1974) 44 (1): 242-248.

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<https://doi.org/10.1306/74D729D2-2B21-11D7-8648000102C1865D>

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Abstract

A modified ignition loss method is described for determining organic and carbonate carbon in calcareous sedimentary materials using equipment found in most laboratories. The method has been found to equal or excel the accuracy and precision of other methods tested and has the advantage of being considerably faster if large numbers of samples are to be analyzed.

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DETERMINATION OF CARBONATE AND ORGANIC MATTER IN CALCAREOUS SEDIMENTS AND SEDIMENTARY ROCKS BY LOSS ON IGNITION: COMPARISON WITH OTHER METHODS¹

WALTER E. DEAN, JR.

Department of Geology, Syracuse University, Syracuse, New York 13210

ABSTRACT: A modified ignition loss method is described for determining organic and carbonate carbon in calcareous sedimentary materials using equipment found in most laboratories. The method has been found to equal or excel the accuracy and precision of other methods tested and has the advantage of being considerably faster if large numbers of samples are to be analyzed.

INTRODUCTION

Determination of organic and carbonate carbon in geologic materials by loss on ignition is certainly not a new technique. It appears in most standard texts on quantitative inorganic analysis (e.g., Kolthoff and Sandell, 1946), and has been used to determine the amount of CO₂ in carbonate rocks (e.g., Galle and Runnels, 1960; Waugh and Hill, 1960) and in recent sediments (e.g., Konrad, Chesters, and Keeney, 1970). The purpose of this paper is to present a modification of the ignition method which is rapid, precise, accurate and requires equipment normally found in most laboratories.

THE IGNITION LOSS METHOD

Theory

Differential thermal analysis (DTA) thermograms show that when a dried, powdered sample containing organic material and calcium carbonate is heated in a muffle furnace, the organic material begins to ignite at about 200° C and is completely ignited by the time the furnace temperature has reached approximately 550° C. Evolution of CO₂ from the calcium carbonate will begin at about 800° C and proceed rapidly so that most of the CO₂ has been evolved by the time the furnace has reached 850° C. If any dolomite is present in the sample, it will evolve CO₂ at a lower temperature than calcite (ca. 700-750° C).

Based on this theory, several different techniques have been used to measure organic and carbonate carbon evolved on ignition. The

weight loss of the sample can be determined by weighing the sample before and after ignition and determining the weight percent loss by difference. The ignition loss procedure described below is based on this weight loss principle. Other techniques involve collecting the gas evolved and measuring the amount either volumetrically or chromatographically.

Procedure

The ignition loss procedure described below is a modification of the procedure described by Galle and Runnels, 1960:

(a) A powdered sample is dried in an oven at 90-100° C in a preweighed ceramic crucible for one hour. After cooling to room temperature in a desiccator, the sample and crucible are weighed. This gives the dry weight of the sample which is the basis for all weight loss calculations.

(b) The sample and crucible are then placed in a muffle furnace and heated to 550° C for one hour. After cooling to room temperature, the sample is again weighed. The difference between this weight and the dry weight is the amount of organic carbon ignited.

(c) The sample is returned to the muffle furnace and heated to 1,000° C for one hour. The weight loss between 550-1,000° is the amount of CO₂ evolved from carbonate minerals.

Although this technique takes several hours to complete, only a fraction of this time is actual operator time since most of the time is involved in heating and cooling. The procedure is most efficient if a number of samples are ignited at the same time. I have found that the best way to cut operator time is to construct a

¹ Manuscript received April 19, 1972; revised March 3, 1973.

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