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An Apparatus for the Wet Combustion of Organic Compounds for C 14 Assay

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In the last two years direct mounting of organic compounds for the assay of C 14 has been used in several instances (Entenman, Lerner, Chaikoff and Dauben [1], Wilk, Barnet and Ackerman [2], Popjak [3]). The direct assay, however, requires relatively large amounts of material, *i.e.* about 25 mg/sq. cm. area, if it is to be assayed in infinite thickness. When only small amounts of material are available, the conversion to $BaCO_3$ is the method of choice, unless the direct assay of CO_2 in windowless counter is used. Besides the direct mounting is troublesome when working with oily substances.

In connection with work on the metabolism of C 14 labelled fatty acids we needed a method enabling us to run many combustions simultaneously. Many different methods have been described but a search through the literature revealed the need of a more easily handled apparatus satisfying the above demands.

A modification of the combustion tubes described by Baxter (Calvin, Heidelberger, Reid, Tolbert and Yankwich [4]) has been developed. The samples are combusted in van Slyke-Folch fluid in vacuum at 230° in an oilbath for 1 ½ hour and the CO_2 formed is collected in barium hydroxide. The amount of bariumcarbonate formed is estimated by titrating the excess of barium-hydroxide. A simple shaking device is described that enables us to run a number of combustions simultaneously. A device similar to that described here has recently been published by Claycomb, Hutchans and van Bruggen [5].

METHOD OF OPERATION

The sample to be combusted, containing 5—20 mg of carbon, is weighed in a glass cup 12 × 12 mm, which is then placed in the combustion flask A (see Fig. 1). 10 ml of van Slyke-Folch combustion fluid (without potassium

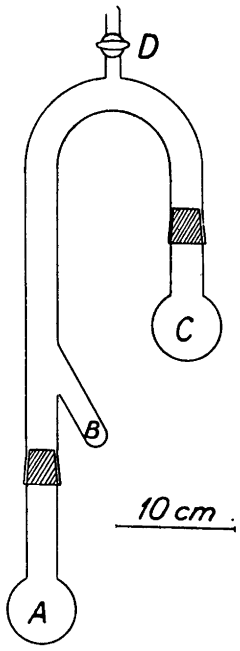

Fig. 1. Wet combustion apparatus (for description see text).

iodate) is pipetted into side tube B, while the apparatus is held in an upright position. C is filled with 12 ml of 0.25 *N* barium hydroxide. The apparatus is assembled and evacuated through D with an oil pump while carefully shaking to avoid splashing of the barium hydroxide solution in C. The combustion fluid is used as a joint lubricant at the lower joint and high-vacuum stopcock grease at the upper one. After the evacuation the combustion fluid in B is transferred to A by gently tipping the tube, and the tube is placed in an oil-bath at 230° for one hour and a half. The oil-bath here described (see Fig. 2) can hold six combustion tubes and is provided with a thermoregulator and an arrangement for automatic shaking of flasks C, in order to facilitate rapid adsorption of the CO_2. A fan is used to cool the tubes over A, thus preventing the SO_3 of the combustion fluid from reaching C.

For the samples containing no nitrogen or halogen the amount of carbon dioxide formed is determined by titrating the excess of barium hydroxide with 0.25 *N* HCl, using phenolphtalein as indicator. The $BaCO_3$ is then collected and washed by centrifugation.

ACCURACY OF THE METHOD

The method has hitherto been used for combustion of fatty acids and cholesterol.

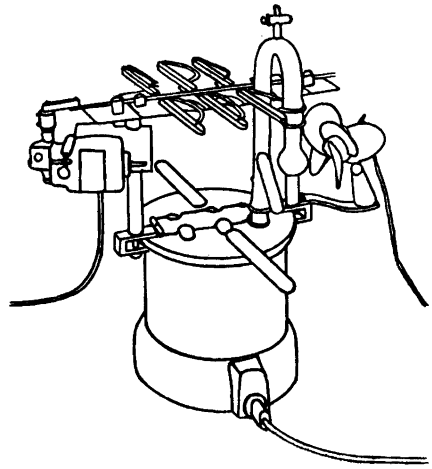

Fig. 2. Oil bath with shaking device and fan for six combustion tubes.

Ten determinations on 14.0 mg of stearic acid gave a mean value of 13.4 (95.6 per cent) S.D. $\pm$ 0.2.

SUMMARY

An apparatus is described for the wet combustion of organic compounds to be used in C-14 assay, convenient for serial analyses.

I wish to express my indeptedness to professor E. Hammarsten, Stockholm, for the time spent in his laboratory learning the isotope technic used there.

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REFERENCES

1. Entenman, C., Lerner, S. R., Chaikoff, I. L., and Dauben, W. G. *Proc. Soc. Exptl. Med.* **70** (1949) 364.
2. Wick, A. N., Barnet, H. N., and Ackerman, N. *Anal. Chemi.* **21** (1949) 1511.
3. Popjak, G. *Biochem. J.* **46** (1950) 559.
4. Calvin, M., Heidelberger, C., Reid, J. C., Tolbert, B. M., and Yankwich, P. F. *Isotopic carbon*. New York (1949).
5. Claycomb, C. K., Hutchens, T. T., and van Bruggen, J. T. *Nucleonics*. **7** (1950) 38.

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