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NOTES AND COMMENTS

MICROWAVE OVEN PRETREATMENT OF CARBONATES FOR ^{14}C DATING BY ACCELERATOR MASS SPECTROMETRY

DAVID C LOWE, GORDON W BRAILSFORD
and GREGORY W DRUMMOND

Department of Scientific and Industrial Research, Institute of Nuclear Sciences, Lower Hutt, New Zealand

ABSTRACT. A microwave oven is used to pretreat carbonate samples prior to graphitization and radiocarbon dating by accelerator mass spectrometry. The method reduces the risk of contamination of small carbonate samples and provides a fast and convenient method for the acid evolution of CO_2.

INTRODUCTION

One of the major impacts of the accelerator mass spectrometry (AMS) method of radiocarbon dating has been to lower sample size requirements of 0.1 to 5mg of carbon. This has resulted in the development of various micro-chemical techniques for the pretreatment and subsequent graphitization of samples submitted for dating. In our laboratory, we have had a large demand for radiocarbon dates on various kinds of carbonates including shells, marine phosphorites, bone apatite, and starfish spicules. To liberate CO_2 from these species, we have traditionally used concentrated orthophosphoric acid with the samples in a glass apparatus heated to 100°C in a steam bath to dissolve the carbonates. During the last few months, however, we have used a conventional domestic microwave oven to quickly and conveniently dissolve the various carbonate species submitted to our laboratory for ^{14}C AMS analysis.

EXPERIMENTAL PROCEDURE

The carbonate sample to be dated is placed in the main arm of a side-arm flask and 3ml of CO_2-free concentrated orthophosphoric acid are metered into the side arm of the pyrex apparatus (Fig 1) using a Socorex dispenser fitted with a teflon needle. In order to do this, the glass piston valve, fitted with a teflon "O" ring, is removed from the apparatus. Taking care not to tip the acid onto the carbonate, the valve is replaced and the apparatus carefully evacuated to a pressure of ca 10^{-3} Torr. After evacuation, the valve is closed, the acid mixed with the carbonate, and the apparatus is placed in a conventional domestic microwave oven. Concentrated acids are effective absorbers of microwave energy in the 2.45 GHz region (Mahan *et al*, 1987). Microwaving at 650 watts radiated power for 60 secs easily dissolves individual portions of carbonates like shell weighing 30 to 40mg.

After dissolution of the carbonate, CO_2 is extracted and purified by careful vacuum distillation over alcohol/dry ice and liquid nitrogen traps. Reaction yields are measured by expanding the CO_2 into a calibrated manifold and measuring the pressure with a pressure transducer. Carbon yields

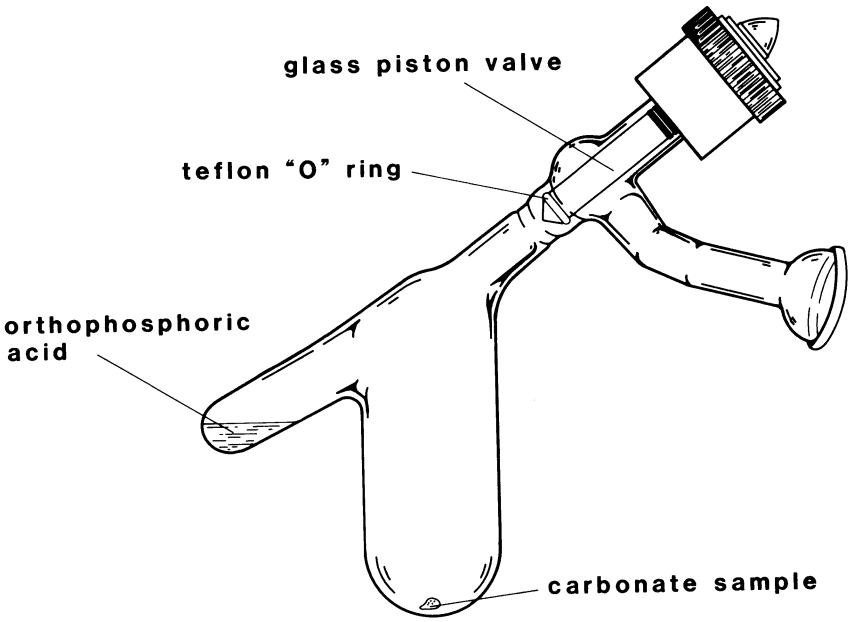

Fig 1. Side-arm flask used for microwave oven acid dissolution of carbonate samples

are always within ±2% of theoretical from calcium carbonate standards with the major error in the determination being in the pressure measurement.

When the yield has been calculated, the CO_2 is converted into a graphite target using the method of Lowe and Judd (1987) and ^{14}C AMS measurements are made using the New Zealand AMS facility (Wallace *et al,* 1987). We have used the technique for a wide variety of carbonate types including marine phosphorites, shells, bone apatite, and Crown of Thorns starfish spicules (Sparks, Wallace & Lowe, 1986).

RESULTS AND DISCUSSIONS

Microwave oven pretreatment appears to have some major advantages over the traditional steam bath technique. At 100°C 30mg solid pieces of shell form a semi-impervious layer of calcium phosphate around the bulk of the carbonate and reaction times for complete dissolution can be several hours. To speed the reaction rate, most laboratories grind carbonates into a fine powder before adding acid. For ^{14}C AMS measurements in which samples are small, this process greatly increases the risk of sample contamination. In the microwave technique reported here, the orthophosphoric acid reaches a temperature of ca 140°C and a 30mg piece of shell dissolves typically in ca 1 minute. Hence, the need for initial powdering of carbonate sample is eliminated and the risks of sample contamination during pretreatment are greatly reduced. Using a 10ml apparatus (Fig 1), we have treated

samples containing as little as 0.2mg of carbon with the technique. For bigger samples, eg, 4mg of carbon, a larger, 30ml apparatus is used.

After an extensive series of tests, we have now adopted the microwave technique as part of our standard pretreatment technique for radiocarbon AMS measurement of carbonate samples.

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