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## Comparison of Quartz and Pyrex Tubes for Combustion of Organic Samples for Stable Carbon Isotope Analysis

Thomas W. Boutton,\* William W. Wong, David L. Hachey, Lucinda S. Lee, Mercedes P. Cabrera, and Peter D. Klein

*Stable Isotope Program, Children's Nutrition Research Center, USDA/ARS, Department of Pediatrics, Baylor College of Medicine, Texas Children's Hospital, Houston, Texas 77030*

Most laboratories that perform routine stable carbon isotope determinations on organic samples now employ a sealed-tube combustion method similar to that described by Buchanan and Corcoran (1). According to this method, the sample is sealed into an evacuated length of quartz tubing containing copper oxide as an oxidant and baked at 800–900 °C. This method is more efficient in processing large numbers of samples when compared to dynamic methods, where gases are repeatedly cycled through a furnace by means of a Toepler pump (2).

Sofer (3) recently has demonstrated that petroleum samples could be combusted to CO<sub>2</sub> in borosilicate (Pyrex) glass at 550 °C without loss of precision or accuracy of  $\delta^{13}\text{C}$  values compared with combustion in quartz tubing 850 °C. Sofer (3) also suggested that other organic samples could be combusted at 550 °C in Pyrex as well. Because Pyrex tubing is approximately 14% of the cost of quartz tubing, we have compared  $\delta^{13}\text{C}$  values and percent carbon obtained on a variety of organic substances using both methods, e.g., combustion in quartz tubing at 850 °C and in Pyrex tubing at 550 °C.

### EXPERIMENTAL SECTION

Combustion tubes (9 mm o.d.) were cut to 30 cm lengths and sealed at one end. Approximately 2 g of copper oxide wire and

9 mm<sup>2</sup> of silver foil were placed in each of the quartz tubes, which were subsequently baked at 850 °C for 1 h to remove potential organic contaminants. Pyrex combustion tubes were baked at 550 °C for 1 h, while the copper oxide and silver foil for the Pyrex tubes were purified separately at 850 °C for 1 h. Sample handling cuvettes, approximately 5 cm long, were made from 6 mm o.d. quartz or Pyrex tubing, sealed at one end, and baked for 1 h at the appropriate temperature.

Approximately 3–10 mg of dry sample was weighed into pre-baked cuvettes which then were slid into the larger 9 mm o.d. combustion tubes. Loaded combustion tubes were attached to a vacuum manifold with  $\frac{3}{8}$  in. Ultra-Torr unions, evacuated to  $7 \times 10^{-3}$  torr, and sealed with a torch. Each sealed tube was inserted into a  $\frac{1}{2}$  in i.d. ceramic pipe to minimize the effects of potential explosions and placed inside a muffle furnace. Quartz tubes were baked at 850 °C for 1 h, and Pyrex tubes were baked at 550 °C for 1 h. The temperature of the muffle furnace was increased slowly so that approximately 1.5 h were required to reach 550 °C, and 2 h to reach 850 °C. After combustion, the tubes were allowed to cool to room temperature inside the furnace for 12 h.

Gases from the combusted sealed-tubes were released into a vacuum line by use of a tube cracker (4). Gases were passed through a methanol-dry ice trap to remove water vapor before being frozen into a liquid nitrogen trap. Noncondensable gases were pumped away. The volume of purified CO<sub>2</sub> was measured

Table I.  $\delta^{13}\text{C}$  Values, Percent Carbon, and Results of Paired  $t$  Tests<sup>a</sup>

sample	percent carbon			$\delta^{13}\text{C}$ values		
	quartz	Pyrex	$t$ value	quartz	Pyrex	$t$ value
sucrose (Fisher Scientific)	47.1 $\pm$ 0.7	26.7 $\pm$ 2.2	17.8 <sup>b</sup>	-12.5 $\pm$ 0.1	-12.0 $\pm$ 0.3	2.5
Mazola corn oil	84.4 $\pm$ 5.0	74.0 $\pm$ 7.1	3.1 <sup>c</sup>	-16.0 $\pm$ 0.6	-16.1 $\pm$ 0.3	0.1
NBS-22 oil	93.2 $\pm$ 3.8	79.9 $\pm$ 1.8	7.3 <sup>c</sup>	-29.8 $\pm$ 0.2	-29.9 $\pm$ 0.4	1.2
taro root starch	45.1 $\pm$ 0.6	24.3 $\pm$ 2.9	18.1 <sup>b</sup>	-25.7 $\pm$ 0.8	-25.7 $\pm$ 0.6	0.0
freeze-dried banana fruit	44.8 $\pm$ 0.8	23.6 $\pm$ 1.1	47.0 <sup>b</sup>	-23.6 $\pm$ 0.3	-23.8 $\pm$ 1.0	0.5
infant stool	52.5 $\pm$ 1.0	48.2 $\pm$ 5.4	1.7	-24.1 $\pm$ 0.1	-24.4 $\pm$ 0.4	1.6
human bone collagen	45.1 $\pm$ 0.4	32.9 $\pm$ 2.9	8.8 <sup>b</sup>	-21.0 $\pm$ 0.1	-20.9 $\pm$ 0.6	0.5
infant formula	57.6 $\pm$ 0.8	42.7 $\pm$ 5.8	4.9 <sup>c</sup>	-25.4 $\pm$ 0.2	-25.4 $\pm$ 0.6	0.3

<sup>a</sup>  $\delta^{13}\text{C}$  values are expressed as the parts-per-thousand deviation from the international PDB standard.  $N = 5$  for each determination. <sup>b</sup>  $P < 0.001$ . <sup>c</sup>  $P < 0.05$ .

manometrically for determination of percent carbon and the sample transferred to a 20-mL Vacutainer (Becton-Dickinson, Rutherford, NJ) for  $\delta^{13}\text{C}$  determination on an automated Nuclide 3-60 SecTorr isotope ratio mass spectrometer (5).

The qualitative composition of gases present in the quartz and Pyrex tubes was determined for the mass range 15–200 on a Finnigan MAT 212 mass spectrometer. Resolution was 4000, and electron ionization potential was 70 eV.

## RESULTS AND DISCUSSION

Pyrex tubes combusted at 550 °C showed considerable charring, with a thin black film coating the insides of the sample tubes. This suggests that the organic samples were not completely combusted. Sofer (3) has suggested that less charring occurs when Pyrex tubes are placed into an oven preheated to 550 °C rather than into an oven at ambient temperature in which the temperature is increased slowly. In addition, 25% of the Pyrex tubes developed small cracks, possibly due to rapid temperature changes while the tubes were being sealed. Consequently, these samples had to be repeated. Slower heating and prolonged cooling of the Pyrex tube during the sealing process should reduce the incidence of tube cracking. Quartz tubes, by contrast, showed no charring and only two tubes cracked during the combustion procedure.

Measurements of percent carbon confirmed the qualitative suggestion that samples were not completely combusted by the Pyrex 550 °C method (Table I). For all sample materials except infant stool, percent carbon was significantly lower for the Pyrex method (Table I). On the average, yields were 28% lower for the Pyrex method. Whereas the quartz 850 °C method has been demonstrated to give theoretical yields of percent carbon (1), the Pyrex method cannot be used for quantitative percent carbon determinations.

Mass spectrometric analysis of gases produced by combustion of the NBS-22 carbon isotope reference material in both quartz and Pyrex tubes revealed no qualitative differences over the mass range 15–200.  $\text{CO}_2$  and  $\text{H}_2\text{O}$  were the most common gases produced by both combustion methods. No volatile organic compounds or carbon monoxide were found in the mass range examined, which suggest that the carbon not recovered from the Pyrex tubes was present in the black residue that coated the insides of the combustion tubes.

Despite incomplete combustion by the Pyrex method, mean  $\delta^{13}\text{C}$  values were virtually identical for a given organic sample

whether it was combusted by the quartz or Pyrex method (Table I). Pyrex samples averaged +0.01% relative to the quartz samples, an amount approximately 0.1 the magnitude of instrument error. Paired  $t$  tests showed that  $\delta^{13}\text{C}$  values were not significantly different between the two methods (Table I). Thus, incomplete combustion does not appear to influence  $\delta^{13}\text{C}$  values, as previously reported by Sofer (3) for petroleum samples.

It should be noted that the standard deviations of the replicate  $\delta^{13}\text{C}$  determinations were greater for those samples combusted by the Pyrex method for six out of the eight materials tested (Table I). Thus, confidence limits around mean  $\delta^{13}\text{C}$  values obtained by the Pyrex method may be larger than those for samples combusted by the quartz method.

Combustion of organic samples in Pyrex as opposed to quartz tubing can result in a substantial reduction in operating costs where large numbers of samples are being prepared, and satisfactory  $\delta^{13}\text{C}$  values can be obtained. However, several potential drawbacks preclude total acceptance of the method: (1) Pyrex is more susceptible to cracking during periods of rapid temperature change, such as when tubes are being sealed; (2) combustion is not complete, which prevents quantitative measurements of percent carbon in the sample; and (3) replicate  $\delta^{13}\text{C}$  determinations are more variable. The needs of the individual laboratory should be considered with respect to these limitations before adopting the Pyrex method for combusting organic samples.

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