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CALORIMETRY: MODIFICATION OF A STANDARD BOMB FOR SMALL HEAT OUTPUTS

Standard sizes of bomb calorimeter require about 0·7–1·0 g of dry organic material to be combusted, in order that the total heat release should comply with the standard conditions for calorimetry (see STURTEVANT, 1959). However, in experimental work, and in ecology, such large quantities of material may not be available for combustion. Miniature bombs with small capacity have been described, some only requiring 5–100 mg (MCEWAN and ANDERSEN, 1955; PHILLIPSON, 1964). Very small amounts of energy, of the order of 1–10 gramme-calories, may be determined by the wet-combustion method of IVLEV (1935) or that of KARZINKIN and TARKOVSKAIA (1962), but these methods are apt to be tedious, and moreover IVLEV's requires a correction to be made for the partial oxidation of nitrogenous organic matter.

Having available a standard ballistic bomb calorimeter (MILLER and PAYNE, 1959; manufactured by Gallenkamp, London), and being faced with the problem of analysing small samples of material (faecal matter and foods from feeding experiments with small fishes), I thought it worth investigating the

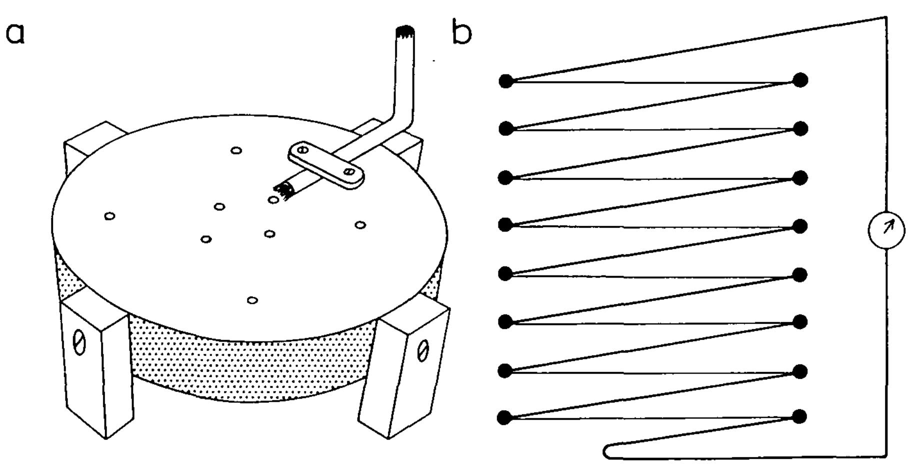

Figure 1. A brass disc holding the 8 hot thermojunctions is placed on top of the bomb (a); the series of hot and cold (reference) junctions are arranged as in (b).

possibility of increasing the sensitivity of the heat-sensing system of the bomb to enable small samples to be determined. According to the calorimeter manual, the response of the spot galvanometer supplied with the calorimeter is non-linear if the total heat release is not of the order of 4 kcal; for this amount of heat the single thermojunction produces a thermal EMF of approximately 730 μV, or 0·18 μV per gcal. A larger thermopile, comprising hot and cold (reference) junctions in series, would increase the thermal EMF in direct proportion to the number of pairs of junctions.

To hold the hot thermojunctions, a brass disc, 12·5 mm thick, with four lugs at the sides to keep it in place, and a machined undersurface, is placed on top of the bomb body (Figure 1). It has eight holes, 2 mm in diameter, passing vertically down to within 2 mm of the undersurface. A similar disc is bolted to the bomb-case. Sixteen soldered thermocouples, of 36 SWG copper and constantan wire, are connected in series, alternate junctions being embedded in each disc and held in place with potting resin; the junctions are insulated with plastic wire-cover. The junctions in the disc on the bomb serve as the hot junctions, the others are reference junctions. The thermocouple leads, of copper wire, are passed to a recording potentiometer with a full-scale deflection of 1 mV. Otherwise, the normal firing circuit, oxygen valves, etc. of the calori-

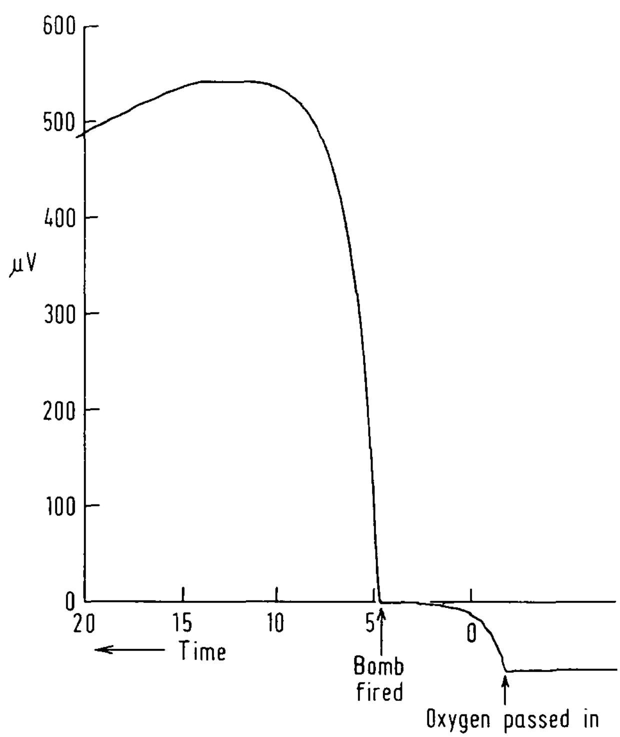

Figure 2. The trace produced by combusting 164 mg thermochemical grade benzoic acid, 6234 gcal/g. The total heat released by the sample was 1084 gcal, producing a thermal EMF of 542 μV. The time scale moves from right to left.

meter are employed. Before the bomb is fired, a box made of aluminium sheet is placed over it, enclosing the reference disc as well, in order to cut down air-currents which might affect the thermocouples and produce stray EMF.

A sample, weighing 50–300 mg, of the dry material is pelleted and placed in the crucible. It is connected to the platinum firing coil by a 5 cm length of dry cotton in the usual way; the bomb is closed, the thermocouples placed in position and the aluminium cover replaced. The potentiometer is switched on, zeroed, and the oxygen passed in to the bomb to an internal pressure of 25 atmospheres. The pressurization produces a small thermal EMF, which is recorded on the chart (see Figure 2). This soon levels off, and the bomb is ready for firing. When this is done, a heating-cooling curve is traced out; the height between the two level portions (see Figure 2) is recorded against the weight of the sample; this EMF is a measure of the total heat released by the combustion.

Thermochemical grade benzoic acid is the usual substance used for calibrating bomb calorimeters, since its heat of combustion is very accurately known; other substances may also be used. Figure 3 shows the results of three calibrating runs; with benzoic acid, 6234 gcal/g, palmitic acid, 9369 gcal/g, and d-glucose, 3739 gcal/g. The scale of the abscissa is in gcal total heat release, that of the ordinate, the thermal EMF, in μV. The regression slope was equivalent to 1 μV per 2 gcal. The standard deviation from the regression was ±45 gcal, so that obviously samples in the range of 500 + gcal would be more accurately determined than those in the lower part of the range.

When a sample is combusted, the thermal EMF is converted to gcal according to the calibration, i.e. 1 μV = 2 gcal. Figure 4 shows a typical series of results, obtained by combusting small samples of plaice (*Pleuronectes platessa*) faeces.

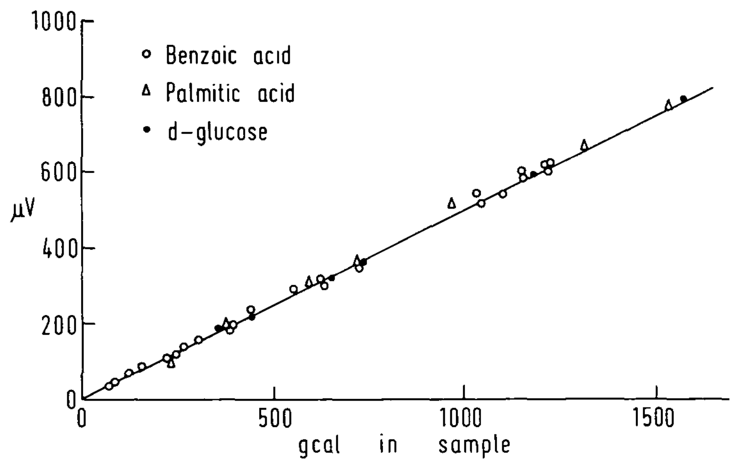

Figure 3. Calibration curve obtained with samples of benzoic acid (6234 gcal/g), palmitic acid (9369 gcal/g) and d-glucose (3739 gcal/g). The slope is equivalent to 1 μV per 2 gcal.

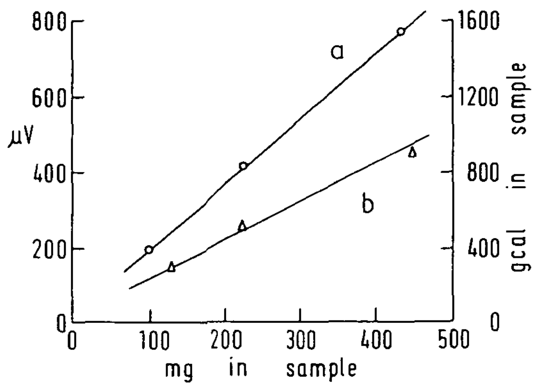

Figure 4. Samples of plaice faeces from two experiments were combusted. The mean calorific values were 3·50 gcal/mg (in a) and 2·37 gcal/mg (in b) (dry weight basis).

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