DIFFERENTIAL MICROCALORIMETER

This invention relates to calorimetric measurements wherein the heat involved is of such a small quantity that techniques of microcalorimetry are required, and more particularly to the construction and use of microcalorimeters of the general type known as differential Tian-Calvet calorimeters.

This type of a microcalorimeter uses a solid body of aluminum, forming a heat sink. Two wells are formed within the heat sink body, wherein thermopiles are fitted, and test cells are carried within the thermopiles. The energy which is released as heat within a test cell, as during a chemical or physical reaction, is detected by a thermopile which feeds a signal into a millimicrovoltmeter or like instrument which, in turn, actuates a recorder to produce a curve indicating thermopile and cell structures and the reaction cell may be in one well while the other cell remains empty and serves as a control. Instruments are provided for insertion into the cells to initiate reactions and to calibrate the microcalorimeter, and other features include an oven to retain the heat sink and a temperature control means on the oven to regulate, as precisely as possible, the basic temperature of the heat sink body for any given test.

The present invention is concerned with improvements in the manner in which the heat sink wells, the thermopile structure, and the cells within the thermopile and integrated to produce more positive and reliable results. At the same time, the present invention is concerned with improved and simplified techniques of building a microcalorimeter having a high degree of sensitivity. Accordingly, the objects of the present invention are to provide a novel and improved microcalorimeter which is highly sensitive, which can be quickly and easily calibrated, which produces accurate, reliable measurements which can be used at a wide range of base temperatures as from below zero to as much as 300° to 400° F., and which is a structurally simple, economical, rugged and durable unit.

With the foregoing and other objects in view, my invention comprises certain constructions, combinations and arrangements of parts and elements as hereinafter described, defined in the appended claims and illustrated in the accompanying drawings, in which:

FIG. **1** is a perspective view, with portions broken away, showing the heat sink covered by a cap and housed in a constant temperature oven, indicative of the manner in which the unit is used.

FIG. **2** is a transverse section through the heat sink of the microcalorimeter illustrating the two wells within the body, with one well being empty and the other well having a thermopile mounted therein.

FIG. **3** is a perspective view of the cell holder and cell, as shown in FIG. **2**, but on an enlarged scale.

FIG. **4** is an elevational view of a sample holder which includes a resistance coil extending into the reaction cell for calibrating the apparatus.

FIG. **5** is a somewhat-diagrammatic, fragmentary sectional detail as taken from the indicated lines **5—5** at FIG. **4**, but on an enlarged scale.

FIG. **6** is a fragmentary sectional portion of the thermopile, the central core and the heat sink about it as shown in FIG. **2**, but on a greatly enlarged scale to indicate the arrangement of components therein.

FIG. **7** is a sectional plan view taken along line **7—7** of FIG. **6**, but on a reduced scale.

FIG. **8** is a sectional plan view taken along line **8—8** of FIG. **6**, but on a reduced scale.

FIG. **9** is an exploded perspective view of the liner sleeve of a heat sink well and of one of each of the components making up the thermopile of the calorimeter unit.

FIG. **10** is a fragmentary, somewhat diagrammatic, sectional detail taken along line **10—10** of FIG. **9**, but on an enlarged scale.

In the present invention, the overall organization is essentially conventional. As set forth at FIG. **1**, the body of the unit H functions as, and is commonly referred to as, the heat sink. This body is a block of aluminum, preferably in the form of a square-ended, right cylinder, and is proportioned to accommodate two wells W wherein thermopiles T are placed, as hereinafter described. When the unit is ready for testing, the heat sink H is placed in an oven O which may be a suitable open-top container. Next, a cover C is placed over the heat sink to enclose it, and the oven is closed by a lid L. Various controls and heating means, not shown, are associated with this oven to hold its interior and the heat sink H therein to a selected basic temperature, i.e., the temperature at which a test is to be conducted. To record data obtained from the microcalorimeter, electrical lead wires **20** will extend from the heat sink and from the oven in any suitable manner, not shown. A power input lead **21** is associated with the oven O to regulate the temperature within it, and other passageways such as **22** may extend through the cover C. Similar passageways, not shown, extend through the lid L to permit communication with the test cells so it is possible to physically initiate the desired reaction when the equipment is ready.

The general arrangement of the wells W in the heat sink H is shown at FIG. **2**. While the proportions of this entire organization are not critical, a suitable well size for many tests will be approximately 5 inches in depth by 3¼ inches in upper diameter and 3 inches in lower diameter, to receive thermopiles T of a comparable size, as will be described. Two such wells are provided side by side in the heat sink, and a heat sink of such size has a diameter of 10 to 11 inches and a height of 8 to 9 inches. This size provides adequate material to effectively absorb the heat generated in the wells without measurable fluctuations of temperature.

A thermopile T is formed as a cylindrical laminate of spacing and holding washers and disc-shaped thermocouples as will be hereinafter described, and a first improvement over conventional types of microcalorimeters is to provide a means for more effectively gripping a cylindrical thermopile when it is lowered into a well. Such is essential to provide an efficient heat transfer to the heat sink.

The well W is formed with a tapered, circular wall **25,** extending from a minimum diameter at the bottom **26** and to a greater diameter at a shoulder **27** near the top of the heat sink, where an enlarged cylindrical mouth **28** is provided to receive a ring-shaped holding washer **29.** A square-ended, tapered circular sleeve **30** is fitted into the well W. The outer wall **31** of the sleeve is tapered to fit the taper of the well, and the inner wall **32** of the sleeve is cylindrical and slightly greater in diameter than the outside diameter of the thermopile T. This sleeve is longitudinally sectioned to provide slots **33.**