

means for calibrating the apparatus based upon a measured addition of heat to the asphalt, of the same order as the reaction. These two requirements are met by a combined sample holder and calibration resistance coil S, shown at FIG. 4. This holder unit is fitted onto the top of the reaction cell at the commencement of a test.

The sample holder S is formed as an inverted cup 55 having an internal diameter sufficiently large as to telescopically fit over the end of the test cell 41. The cup is preferably formed of a stable plastic material such as Teflon. A plastic disc 56, also of Teflon, is snugly fitted in this cup-shaped sample holder, about one-third the distance from the bottom, to hold a charge of aggregate 57 within the upper portion of the cup. The aggregate will be of a carefully selected size, such as that between 30-mesh and 40-mesh, so that the total surface area of the sample can be estimated with a reasonable degree of precision, and one sample will be comparable with another.

Release of the aggregate from the sample holder is effected by tipping the disc 56. A trip wire 58 is connected to one side of the disc 56, and this wire extends upwardly through a passageway 59 in the top of the holder S and thence upwardly and through the passageway 22 in the cover C of the unit. To better heat insulate the wire 58, it is fitted within a plastic tubing 60 of a type which is commonly referred to as spaghetti tubing. The wire is simply pulled to tip the disc and drop the aggregate.

A calibration coil is associated with the sample holder S, for which lead wires 61 depend from each side of the interior of the cup 55 and extend downwardly therefrom to carry a vertically extended, flatly wound resistance coil 62 having a length such that it will be immersed into the test cell 41. This resistance coil is of small diameter Nichrome wire or of a similar high-resistance wire, and it is wound about a flat mica core 63 characterized by having small projections or shelves 64 of mica outstanding from each side thereof at each coil wrap, as illustrated at FIG. 5. The purpose of these shelves is to facilitate tumbling and mixing of the aggregate in the sample holder thereabove as it is dropped into the charge of liquid asphalt within the test cell 41.

In using this apparatus, the thermopile in one of the cells will serve as a base unit while the heat reaction test will be conducted in the other. The test cell 41 is filled with a selected charge of liquid asphalt, the sample holder S is charged with aggregate 57, and the unit is placed upon the test cell 41 with the resistance coil immersed in the asphalt. The remainder of the microcalorimeter has been in the oven to attain a desired heat level when the test cell and sample holder are placed therein. When the asphalt and the aggregate reach the same temperature, which may require 24 to 72 hours, i.e., whenever the temperature of the unit has stabilized, the test may be initiated by pulling the wire 58 to tip the disc 56 and drop the aggregate charge into the test cell. As this aggregate is dispersed within the asphalt in the cell, with the help of the mica shelves 64, a small rise of temperature will occur. This temperature increase will be transmitted to the thermopile to produce a voltage which can be recorded for a time period sufficient to obtain a suitable curve showing the relation between the temperature and time.

A calibration of the apparatus may be made before and/or after each test is made by passing an electric current through the resistance coil 62. This measured current flow will produce a small but equivalent amount of heat in the asphalt, and the heat will be sensed by the thermopile and recorded in the same manner as above described, for comparison with the heat produced by the immersion of the aggregate in the asphalt. In this manner, the heat generated by the adhesion action of the aggregate in the asphalt can be precisely measured.

It will be understood that small amounts of heat generated by other physical, or by chemical reaction or electrical phenomenon, may be measured in the same way.

I have now described my invention in considerable detail. However, it is obvious that others skilled in the art can build and devise alternate and equivalent constructions which are nevertheless within the spirit and scope of my invention. Hence, I desire that my protection be limited not by the constructions illustrated and described, but only by the proper scope of the appended claims.

What is claimed is:

1. In a differential microcalorimeter of the type which is fitted into the well of a heat sink body, said well being circular in section and tapering from a minimum diameter at the bottom to a larger diameter at the mouth, and said microcalorimeter includes:

- a. a reaction cell formed as an elongated, cup-like open top member within a cylindrical core;
- b. a cylindrical thermopile having an axially centered passageway to receive the reaction cell, and which is formed as an array of flat, washer-shaped thermocouple members spaced between an array of inner spacer rings and outer spacer rings with the inner spacer rings tightly fitting about the reaction cell core wall to hold the array together;
- c. a tube-like segmented sleeve with at least one longitudinal segment space therein and having an outer surface corresponding with the tapered well to fit therein and a cylindrical inner surface having a diameter which snugly receives the aforesaid outer spacer rings, whereby with the thermopile within the sleeve and the sleeve within the well, a downward pressure against the sleeve therein effects a reduction of diameter to compress the sleeve against the outer spacer rings of the thermopile;

and wherein:

each of the outer spacer rings is split to provide a small gap to individually yield when the sleeve is compressed against the rings whereby the sleeve will tightly grip each and all of the outer rings, with a substantially uniform pressure.

2. In the microcalorimeter defined in claim 1, wherein:

the inner rings are split so that each may bear against the core wall with a substantially uniform pressure.

3. In the microcalorimeter defined in claim 2, wherein the thermocouple members comprise:

washers of insulating material having an inside diameter slightly greater than the inside diameter of the inner rings and an outside diameter slightly less than the outside diameter of the outer rings,