judiciously placed on the plural surfaces to constrain thermal gradients within the heat sink in the vicinity of the measuring cells parallel to the longitudinal axis of the heat sink. Thus, by passing a common current through each heating element and by likewise orienting the temperature sensitive surfaces of the thermopiles parallel to the axis of the heat sink, a uniform temperature scan is produced and the effect of residual thermal gradients within the heat sink is minimized.

A cylindrical adiabatic shield surrounds the heat sink and the isothermal shield and minimizes heat loss to the surroundings. The adiabatic shield and the isothermal shield are provided with cooling coils, which are used in conjunction with a microprocessor and the heating elements to generate a highly controlled negative temperature scan.

During operation, calorimetric data are obtained from a baseline scan, a solvent scan and a solute scan. The baseline and solvent effects are then subtracted from the results of the solute scan, and the absolute apparent heat capacity of the solute is derived.

When pressure is a desired calorimetric variable, pressurizable ampoules are employed. The contents of these ampoules are pressurized, the ampoules are sealed and the experiment conducted in the normal manner.

BRIEF DESCRIPTION OF THE DRAWINGS

A more complete appreciation of the invention and many of the attendant advantages thereof will be readily obtained as the same becomes better understood by reference to the following detailed description when considered in connection with the accompanying drawings, wherein:

FIGS. *1a* and *1b* are schematic section top and side views respectively of the calorimeter of this invention;

FIG. *2a* is a perspective view of an aluminum frame cell which houses a test ampoule;

FIG. *2b* is a transverse, sectional view of the aluminum cell taken along the line A—A in FIG. *2a;*

FIG. *3a* is a sectional side view of a sample or reference ampoule;

FIG. *3b* is a sectional side view of one embodiment of a pressurized ampoule;

FIG. *3c* is a sectional side view of another embodiment of a pressurized ampoule;

FIG. 4 is a schematic representation of the electrical and thermal connection of the thermopiles within the calorimeter heat sink;

FIG. 5 is a schematic view of the calorimeter heat sink and isothermal shield, showing thermopile orientation and calorimeter heater placement;

FIG. 6 is a system diagram of the calorimeter of the invention, including auxiliary components for temperature control and data collection; and

FIG. 7 is a graph of baseline data and the voltage signal obtained when a square pulse of power is applied to the calibration heater in the reference cell of the calorimeter of the invention; also shown is the signal corrected for the time response of the calorimeter and converted to an apparent heat capacity.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

Referring now to the drawings, wherein like reference numerals designate identical or corresponding parts throughout the several views, and more particularly to FIG. **1** thereof, the calorimeter is seen to consist of a reference measuring cell **8** and a sample measuring

cell **10** symmetrically seated within a copper heat sink **16**. Each measuring cell **8**, **10** includes a cylindrical stainless steel ampoule **12** which contains a test substance, an aluminum cell frame **14** which supports the ampoule **12**, and a pair of thermopiles **18** placed between the aluminum cell frame **14** and the copper heat sink **16** and which measure the temperature difference between the two. The measuring cells **8**, **10** are maintained in good thermal contact with the copper heat sink **16** by means of three copper wedges **34**, **35** and **36** which are angled so as to provide uniform lateral force to each measuring cell **8**, **10** thereby maintaining both cells rigidly in place. A fourth smaller wedge **37** perpendicular to the three larger wedges **34–36** and located at the top of these larger wedges, further maintains rigid emplacement of the cell-wedge system in the heat sink **16**. A thermal grease, consisting of a silicon base loaded with zinc oxide, is applied to the surface areas of the aluminum cell frames **14**, the thermopiles **18**, and the wedges **35–37**, thereby assuring excellent thermal coupling between the heat sink **16** and each measuring cell **8**, **10**.

The heat sink **16** is surrounded by a cylindrical copper isothermal shield **20**. The combination of the heat sink **16** and the isothermal shield **20** is enclosed within an aluminum adiabatic shield **22** and mechanically coupled thereto by means of plastic support rods **24**. The adiabatic shield **22**, which houses the heat sink **16** and the isothermal shield **20**, is placed inside an outer cylindrical can **26**. A polyurthane form **28** is provided between the adiabatic shield **22** and the outer cylindrical can **26** to serve as both an insulative layer, and to hold the adiabatic shield **22** in place within the outer cylindrical can **26**.

Also shown in FIG. *1a* are inner and outer cooling tubes **30** and **32** respectively wound around the isothermal shield **20** and the adiabatic shield **22**. As seen in FIG. *1b*, each ampoule **12** is loaded into the aluminum cell frame **14** through the outer cylindrical can **26** and the adiabatic shield **22** by means of plastic tubes **40**. Also, access openings **42** are provided within the heat sink **16** to enable loading of the ampoules **12** within the aluminum cell frames **14**. Once the ampoules **12** are seated within the aluminum cells **14**, copper plugs **44** are provided to seal the heat sink access openings **42**. Similarly, insulation plugs **46** are inserted within the plastic tubes **40**, thereby improving the thermal isolation of the heat sink **16**.

The aluminum cell frame **14** which houses ampoule **12** is seen in FIGS. *2a* and *2b* to have square aluminum front and rear plates **48** and **50**, and rectangular top and bottom plates **52** and **54**. Through the center of the frame **14** is drilled a slightly tapered cylindrical boring **56** in which the ampoule **12** is housed. The boring **56** is tapered so that the diameter of the boring exit hole **53** through the rectangular bottom plate **54** is slightly less than the diameter of the boring entry hole **55** through the rectangular top plate **52**. The tapered ampoule **12**, described hereinafter in detail, has a diameter comparable to the entry hole **55** of the cylindrical boring **56**, but slightly larger than the diameter of the exit hole **53**, such that the ampoule **12** is rigidly positioned within boring **56** of the frame **14**. A plurality of discrete honeycomb cavities **57** are drilled into the cell frame **14** at either side of the cylindrical boring **56**. This honeycomb structure reduces the thermal mass (heat capacity) of the cell frame **14** and proportionately improves the instrument sensitivity since the relative contribution of the ampoule