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AEC-NASA TECH BRIEF

Isothermal Drop Calorimeter Provides Measurements for Alpha Active, Pyrophoric Materials

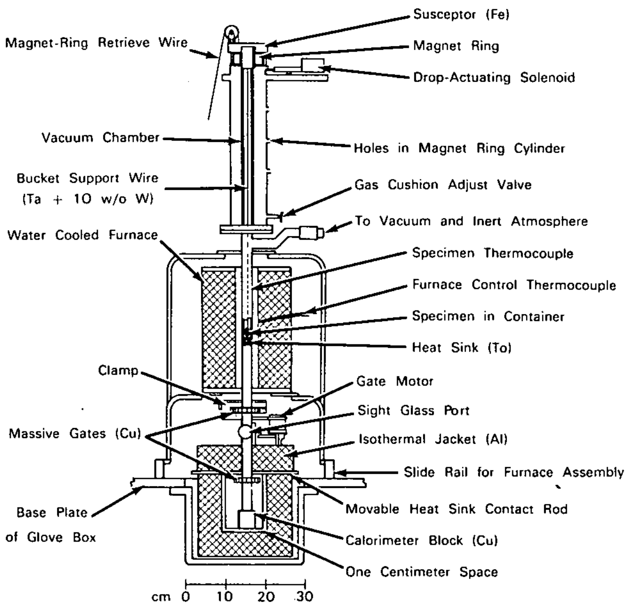

FIGURE 1

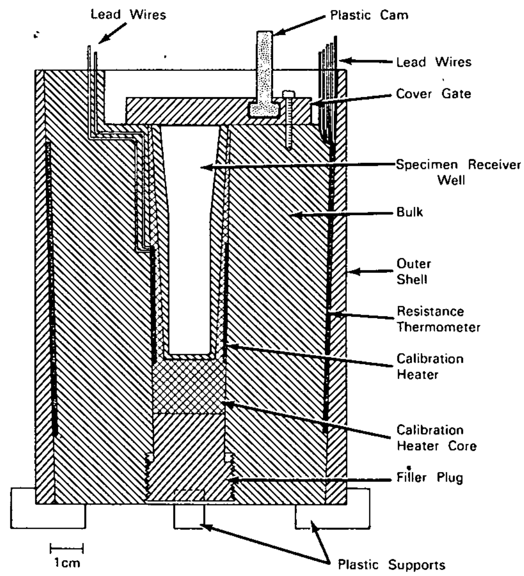

FIGURE 2

The problem:

To provide heat-content measurements for very toxic materials in inert atmospheres. The measurement of the calorimetric properties of plutonium-containing materials and other intense alpha emitters has been difficult because of the toxicity and pyrophoricity of the samples. Those measurements of plutonium-bearing materials are needed to aid in reactor kinetics calculations.

The solution:

A sophisticated copper block drop calorimeter to measure the heat content of intensely alpha active materials in inert atmospheres. The instrument designed to measure heat contents, heats of transition, etc. of potentially hazardous materials to an accuracy of 1% at 1008°K, is capable of operation from 306° to 1450°K.

How it's done:

The isothermal drop calorimeter, shown in Figure 1, consists of a furnace, drop mechanism, calorimeter, jacket, and accessories.

The furnace, calorimeter, and aluminum isothermal jacket are contained within an inert-atmosphere glovebox, which permits the use of unencapsulated materials without exposing personnel to alpha contamination. The inert atmosphere also permits measurements on materials which are pyrophoric in air.

The apparatus is equipped with a suppressed range recorder to monitor continuously the resistance

(continued overleaf)

thermometer signal with a resolution of ±0.4 cal/μv. Electrical calibration of the calorimeter yielded a precision of ±0.08% in the thermometer and recording system.

The copper block used in the isothermal drop calorimeter, shown in Figure 2, includes a resistance thermometer circuit to detect small changes in its temperature. The copper block is an assembly of the resistance thermometer, calibration heater, double junction copper–constantan thermocouple, and six structural subsections (the outer shell, bulk, cover gate, filler plug, calibration heater core, and specimen receiver well).

The calorimeter attains internal thermal equilibrium rapidly because it is compact and the subsections are joined by large contact surfaces. Heat exchange between the calorimeter and external masses is minimized by a 1-cm gap, low-conductive plastic supports, and a cover gate linkage between the silver-plated copper block and the aluminum isothermal jacket. Argon gas at 1 atmosphere in the sealed chamber maximizes heat transfer from the specimen to the copper block.

A resistance furnace, rated for 1200°C, is used to heat the specimens. A tantalum specimen container fits within the furnace heat sink.

The method of measuring the output of the calorimeter thermometer employs a suppressed range recorder, which has a full span of 1mV for each of the 12 ranges and an absolute accuracy of 3μV.

Notes:

1. This information may be of interest to metallurgists, the National Bureau of Standards, the Bureau of Mines, and the aircraft industry.
2. Reference: Additional details may be found in the *Review of Scientific Instruments*, vol. 37, no. 8, p. 1062–1064 (August 1966)
3. Inquiries concerning this innovation may be directed to:

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Patent status:

Inquiries about obtaining rights for commercial use of this innovation may be made to:

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