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AN IMPROVED THERMAL HEAD FOR D.T.A. OF CORROSIVE MATERIALS

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Differential thermal analysis of sulfides and other minerals that corrode metal containers when heated in air was made practical through the development of specially designed receptacles described by Kopp and Kerr (1957). Research on the differential thermal analysis of sulfides supported by the National Science Foundation has recently resulted in the development of apparatus which is a further refinement of this equipment.

The thermal head, shown in Fig. 1, is machined out of 18-8, Cr-Ni stainless steel round stock. Specimen well holes are designed to contain nests of alundum insulator tubing which are supported in the head by a thin transite disc. The nests can be prepared in advance from commercially available stock and are quickly interchanged when thermocouples require replacement. Head temperature is measured by the reference thermocouple using an ordinary triple lead arrangement as described by Mackenzie (1957, p. 36, Fig. II, 4). The writers are indebted to Mr. E. M. Bollin of the mineralogy group at Columbia University for calling this procedure to their attention.

The simplification of the head design permits rapid and inexpensive machining, and facilitates the removal of used specimen wells and cement. The specimen well assemblies are constructed with the aid of brass gauges which have been made in order to insure constant well

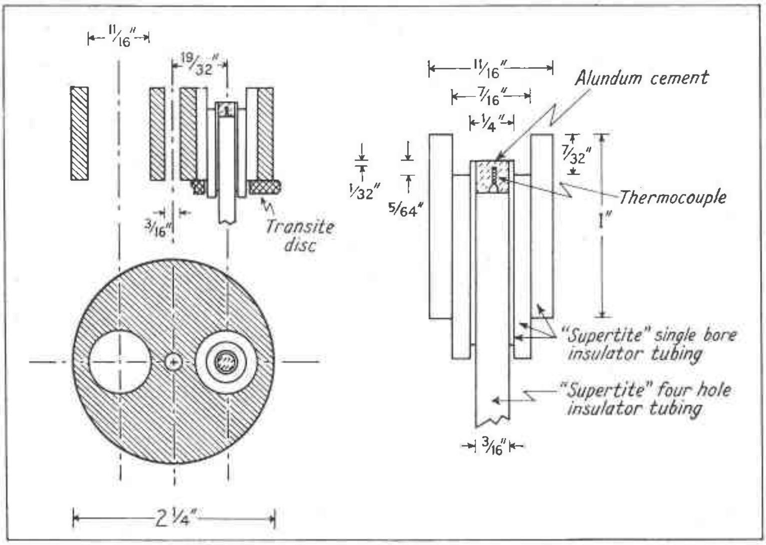

FIG. 1. A sectional view of the thermal apparatus. The metal head is shown on the left and the well nest assembly of ceramic insulator tubing on the right.

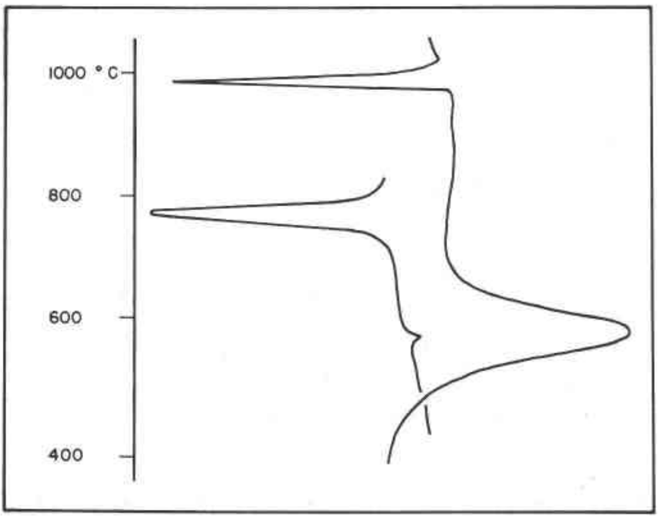

FIG. 2. D.T.A. curves of galena, Wood River, Idaho (left) and kaolinite (API No. 17; right). The latter shows the sensitivity of the apparatus for familiar materials.

dimensions and sample-to-thermocouple distances, as indicated in Fig. 1. This feature furnishes head-to-head reproducibility. Experimental data collected using the modified apparatus show reproducibility to be essentially a function of chart reading error. The 6″ chart of the Leeds and Northrup Speedomax H single point continuous recorder which is used for the majority of sulfide analyses can be read to an accuracy of $\pm 5°$ C.

The positive nature of the alundum shielding protects the thermocouples to such an extent that curves for 25 to 30 sulfide samples may be obtained without replacement. The sensitivity of the apparatus compares favorably with that of equipment normally used for clays, carbonates and other thermally active materials. The D.T.A. curve of a reference sample of kaolinite (No. 17, Am. Pet. Inst. Research Project No. 49) obtained from the modified apparatus is given in Fig. 2. The amplitude of the exothermic peak indicative of recrystallization of the sample to mullite represents a thermocouple emf of approximately 0.47 millivolts. A curve for galena from the Mayflower Mine, Wood River, Idaho is displayed to the left of the kaolinite curve in Figure 2. The exothermic peak at 790° C. represents sample oxidation. The well defined nature of the oxidation peak has been found to be characteristic of galena.

The apparatus described herein has been applied to the study of the PbS-PbSe system (Dunne and Kerr, 1960), in combination with recently developed equipment which allows D.T.A. in vacuo and furnishes differential thermal records of the reactions involved in mineral pyrosynthesis (Bollin, Dunne and Kerr, 1960).

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