THE AMERICAN MINERALOGIST, VOL. 44, MARCH–APRIL, 1959

A SAMPLE HOLDER FOR DIFFERENTIAL THERMAL ANALYSIS OF FUSIBLE OR REACTIVE SAMPLES

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The usual sample-thermocouple arrangement in differential thermal analysis (DTA) involves direct placement of the differential themocouple within the samples. With this arrangement, fusion or sintering of samples makes sample changes difficult. Often, thermocouples are damaged beyond repair in attempting to remove them from the samples. Furthermore, chemical attack by various materials may alter the electrical characteristics of the thermocouples, in addition to shortening their lives. The use of noble metal couples reduces these difficulties, but does not eliminate them.

The use of external thermocouples facilitates sample changes and greatly prolongs thermocouple life. However, there have been objections to all previously suggested external thermocouple arrangements, and none have gained popular acceptance. Increased base line drift and loss of sensitivity and/or resolution have been the most serious difficulties experienced with the external couples. These problems are reviewed in recent books by Smothers and Chiang (1958) and Mackenzie (1957).

The sample holder-external thermocouple arrangement described here, with amplification of the response, provides good sensitivity without serious base line drift. The holders are thin-walled platinum microcrucibles, 12 mm. deep and about 12 mm. in diameter. They have a capacity of 1.3 cc. The bottom of each crucible is fitted with a closed-end center well 3 mm. in diameter and 6 mm. deep to accommodate the external thermocouple junction. The cylindrical well is made from platinum foil and is soldered to the crucible with a platinum alloy solder which melts at 1500 C.

The differential thermocouples used are platinum-platinum, 13 per cent rhodium made from B. & S. No. 28 gauge wire. The junctions are butt-welded. The thermocouples are housed in two-hole insulating spaghetti, about 2.5 mm. in diameter, and are mounted in an alundum cylinder used to seal one end of a tubular furnace. Quartz tubing, extending through the support block and along most of the length of the spaghetti, encloses the thermocouples to add strength.

The samples are supported in the furnace by the thermocouples them selves. When the sample holders are slipped over the thermocouples, the junctions are automatically positioned in the center of the sample cruci-

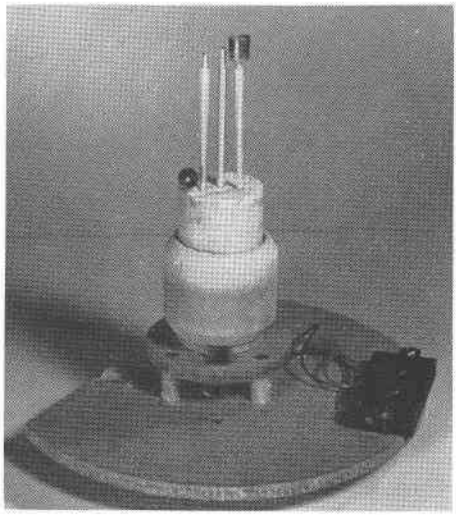

Fig. 1. DTA sample holder. External thermocouple assembly.

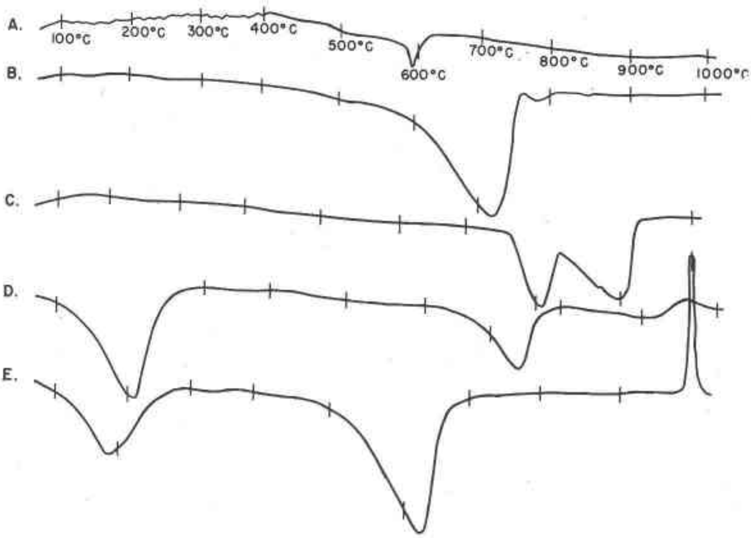

Fig. 2. DTA curves. (A) Quartz, Arkansas; amplification ×50. (B) Magnesite, Luning, Nevada; ×20. (C) Dolomite, Thornwood, New York; ×20. (D) Montmorillonite, Osage, Wyoming; ×20. (E) Halloysite, Eureka, Utah; ×20.

bles. A thin coating of "Sauereisen" cement on the thermocouple junctions electrically insulates them from the sample holders.

The assembly, with one sample holder in place, is shown in Fig. 1. The other sample container, resting on top of the alundum block, is tilted to show the center thermocouple well. The center insulating tubing houses two thermocouples used to monitor the furnace temperature. They are connected to a recorder and temperature controller. The entire assembly, which may be exchanged for a conventional nickel block assembly, is supported by a collar which moves freely on a tubular stand (not shown). Exchange of the two types of sample holders is facilitated by quick-disconnect thermocouple lead connectors. By means of a clamp and locator-pin arrangement the samples are quickly and accurately positioned in the center of the furnace.

The quality of the DTA data obtained with the external thermocouple and platinum holders is illustrated by typical DTA curves shown in Fig. 2. The authors find the curves entirely satisfactory for most DTA purposes.

Acknowledgment

Permission of the Magnolia Petroleum Company to publish this note is gratefully acknowledged.

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THE AMERICAN MINERALOGIST, VOL. 44, MARCH–APRIL, 1959

SPONTANEOUS OXIDATION OF A SAMPLE OF POWDERED SIDERITE*

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In checking over the analyzed samples left by the late Dr. Roger C. Wells, former geochemist, U. S. Geological Survey, it was noted that a powdered sample from near Linden, Texas, labeled siderite, had a marked reddish color, suggesting considerable oxidation of the ferrous

* Publication authorized by the Director, U. S. Geological Survey.