NOTES AND NEWS

DIFFERENTIAL THERMAL ANALYSIS OF SIDERITE

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Submicroscopic or minutely crystallized siderite is common in a number of important petroleum-bearing sediments occurring in aggregates particularly amenable to the methods of differential thermal analysis. It is a matter of more than ordinary importance, therefore, to clarify the uncertainty which seems to exist concerning the thermal curve of this mineral.

A differential thermal curve of siderite has been published by Cuthbert and Rowland (*Am. Mineral.*, 1947, p. 114). This curve (Fig. 2) shows a broad exothermic peak with a crest at about 560° C. In this laboratory, however, siderite has been observed to yield a strong endothermic peak ranging from 520° C. to 650° C. followed by an oxidation dome. These features are illustrated in the accompanying figure. The curves are representative of those obtained by thermal analysis of 40 siderite samples from the Columbia University collection.

Other laboratories also report endothermic peaks for the thermal curve of siderite as Cuthbert and Rowland have acknowledged in Notes and News (*Am. Mineral.*, 1947, p. 591). However, in explanation these authors state, "Perhaps a fortuitous combination of pure sample, dilution and heating rate has suppressed the endothermic reaction which other workers obtain when these conditions are *different.*"

The implication that purity, dilution or heating rate may account for the lack of agreement merits careful consideration. The purity of the Columbia University specimen from the well known Roxbury locality is at least comparable to the anomalous siderite from the same locality described by Cuthbert and Rowland as shown in Table 1. It should be added that all of the samples examined in this laboratory, shown in Fig. 1, have been confirmed by x-ray diffraction.

Dilution with other constituents ordinarily depresses both endothermic and exothermic peaks approximately alike. Grim and Rowland (*Am. Mineral.*, 1942, p. 806) recorded this effect on diluting kaolinite with illite.

Heating rate changes alter the shape or amplitude of a peak (Speil, *U. S. Bureau of Mines, Tech. Paper*, **664**, 1945), but do not eliminate an endothermic peak while leaving an exothermic peak unaffected.

Cuthbert and Rowland also explain the curve on chemical grounds as follows: "The exothermic peak is a result of the heat balance between
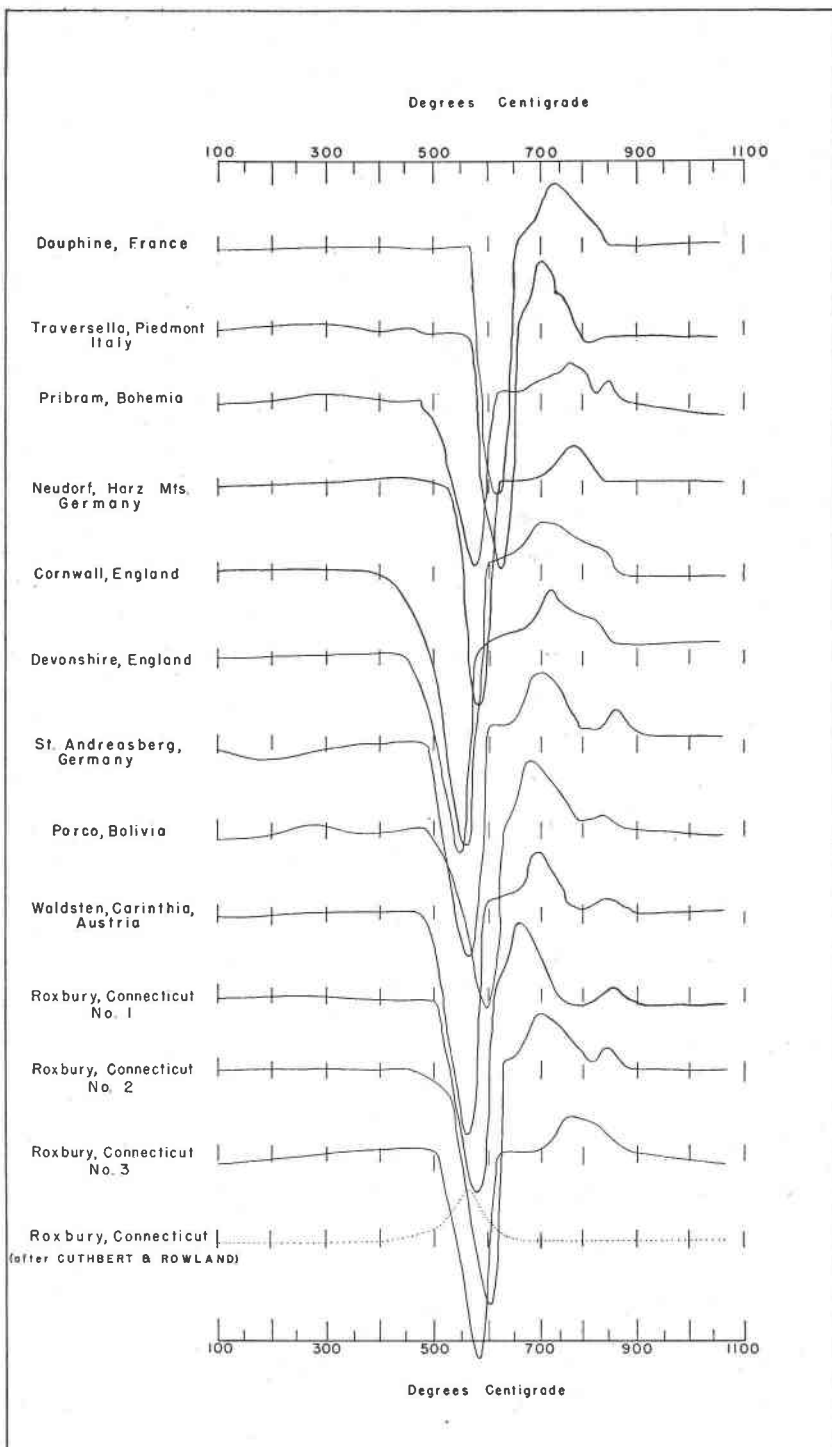
FIG. 1. Differential thermal curves of siderite.

the decomposition of the $FeCO_3$ and the immediate oxidation of the resulting FeO to Fe_2O_3." This assumption of "immediate oxidation" is open to question because the decomposition of the tightly packed powder in the thermal well is relatively sudden and the large evolution of CO_2 in the first few minutes of reaction would prevent the ready access of oxygen to the powder. After the major part of the CO_2 evolution has ceased, oxidation may proceed as observable in the accompanying curves.

TABLE 1: CHEMICAL ANALYSES OF SIDERITE FROM ROXBURY, CONN.

	Cuthbert and Rowland *Anal. by L. D. McVicker*	Kerr and Kulp *Anal. by Ledoux & Co.*
CaO	—	0.86
MgO	—	4.10
FeO	53.80	56.80
CO_2	38.21	37.25
Total	92.01	99.01

The cause of the reported exothermic peak is not clear. Neither unusual purity of sample, dilution or change in heating rate appear to furnish an adequate explanation. Until the exothermic peak is confirmed, the primary reaction for siderite should be regarded as endothermic as Cuthbert and Rowland report for other rhombohedral carbonates.

SYNTHESIS OF TOURMALINE

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The writers recently have synthesized tourmaline by a hydrothermal method at temperatures in the range from 400° to 500° C. The method employed was based on the recrystallization of powdered tourmaline glass heated in contact with water solutions of magnesium and alkali borates. The largest crystals so far obtained are slender prisms about 0.5 micron in length. Spherulitic aggregates of microcrystals also have been obtained by direct devitrification of the glass under these conditions. Alloy steel bombs basically of the Geophysical Laboratory type were employed.

Tourmaline melts incongruently at temperatures varying roughly from 1050° to 1200° C. for the iron-rich types, the melting temperature decreasing with increasing content of iron or magnesium. Homogeneous glass can be prepared from high-iron tourmaline by quenching from about 1550°. The point of complete melting of lithia-tourmaline, however, is over 1725° C. When the melts approach fluidity they boil slightly

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