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Note

A pretreatment method to determine differences in adsorbed water by differential thermal analysis

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During a recent study of the surface area of tris(ethylenediamine) chromium(III) substituted montmorillonite it became axiomatic that we attempt to determine if a difference between the water held by the clay surface and that by the complex cation could be noted. There are two ways in which water can be held in the interlamellar region of clay aggregates[1]. First, there is the water that is in contact with the surface of the clay plates and second, there is the water that is held to the exchangeable cation by coordination or hydration forces. In the case of $Cr(en)_3^{3+}$-montmorillonite both types of water are presumed to be present. Therefore, in a DTA, if the separation in free energy of the waters is sufficient, one should be able to determine the two types. Initial DTA* runs at heating rates of 5.9°C min^{-1} and 2.3°C min^{-1} did not yield two separate water endotherms for 75% exchanged Na^+ montmorillonite as shown in Fig. 1.

By predrying the sample over $CaCl_2$ for 24 h prior to running DTA it was noted that two dehydration peaks occurred at approximately 63 and 81 °C. These peaks are probably surface water and water of hydration, respectively (Fig. 1).

A pretreatment of 75% $Cr(en)_3^{3+}$-montmorillonite over a saturated $Ca(NO_3)_2$ solution (relative humidity at 25°C equals 51%) gave such a large increase in the water endotherm that no differences in water types could be detected. This curve is also shown in Fig. 1. A sample of the same clay stored over P_2O_5 for 13 h again gave the two dehydration peaks thus indicated in the two water types (see Fig. 2). The lower temperature peak is very rapid to rehydrate and if delays in handling occur after removal from the P_2O_5 dessicator the sample will rapidly return to a single undivided peak as shown in Fig. 2.

REFERENCE

1 S. B. Hendricks, R. A. Nelson and L. T. Alexander, *J. Amer. Chem. Soc.*, 62 (1940) 1457.

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*The DTA apparatus used here was built in the Baylor laboratories. It utilizes a Model SH-11BR 2 Stone sample holder and a Model HX-2A TemPres temperature controller. The heating rate was controlled by a motor driven potentiometer. For low temperature work, aluminum sample pans were used. Al_2O_3 was used as the reference material. Samples were run under static air conditions.

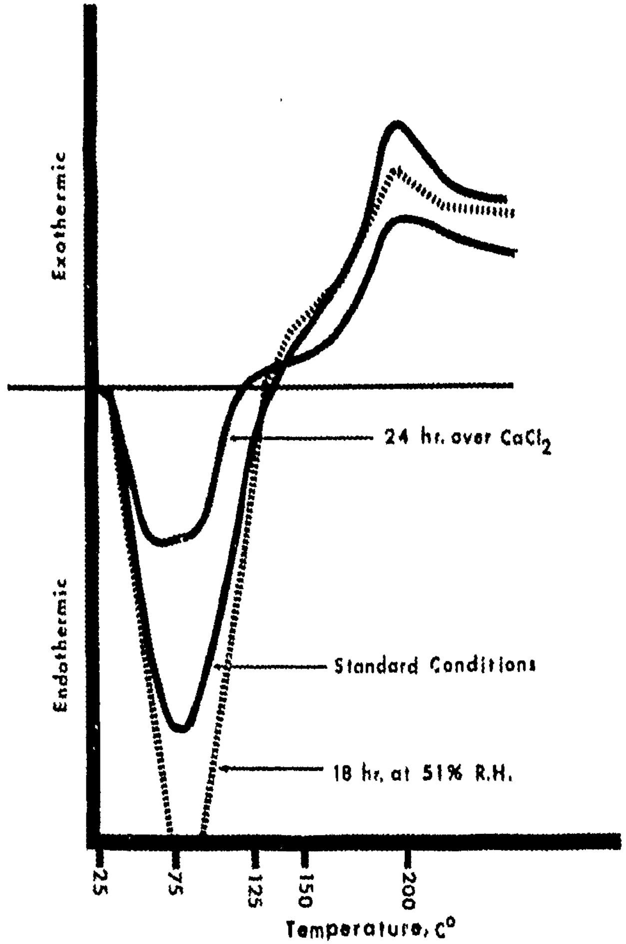

Fig. 1. DTA of tris(ethylenediamine) chromium(III) montmorillonite.

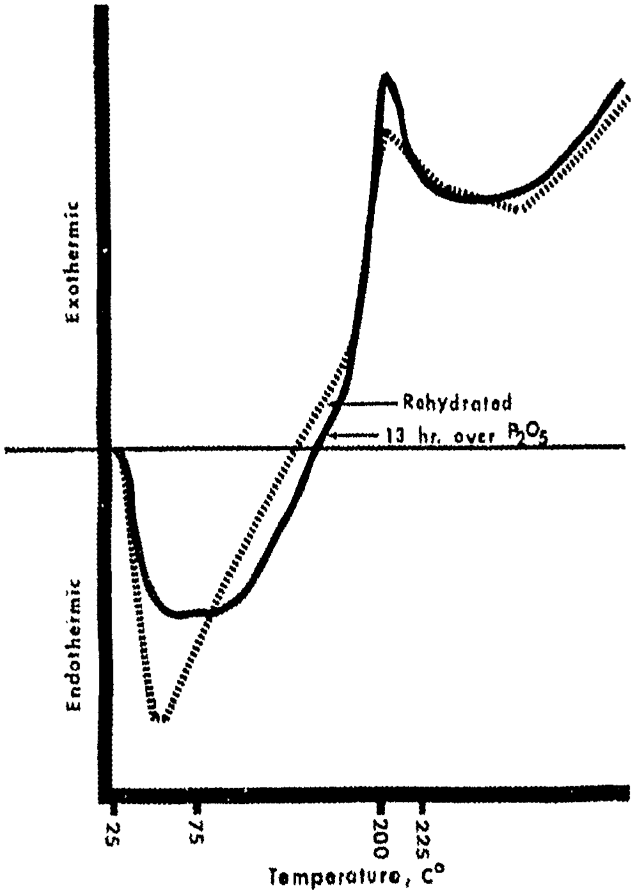

Fig. 2. DTA of tris(ethylenediamine) chromium(III) montmorillonite.