THERMAL BEHAVIOUR OF SOME MINERALS

Differential thermal analysis and determination of PA curves for different heating rates

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Thermal behaviour of some minerals (kaolinite, halloysite, two montmorillonites, quartz and calcite) for different sample amounts and heating rates has been investigated using differential thermal analysis. On the basis of the DTA analysis carried out, the PA curves of each mineral have been obtained for different heating rates. These diagrams can be employed in the semi-quantitative evaluation of identical substances contained in uncharacterized polymineral samples.

A great deal of information on the minerals here concerned is to be found in the literature [1, 5] and, for some of them, the relative PA curves (Probenabhängigkeit), i.e. the ultimate objective of this work, are also provided. The PA curve (curve of sample amount dependence) of a substance can be determined only when the endothermic effect is due to dehydration, dehydroxylation and/or structure decomposition. In this case, the relative peak temperatures basically depend upon sample amount and heating rate [2, 4]. If standard PA curves can be obtained from pure enough monomineral samples, the interdependence between these parameters allows to determine the amount of unknown samples that undergo dehydration or decomposition during heating. Consequently the minerals contained in a polymineral unknown sample can be determined semiquantitatively.

As it will be explained later, the tests have been performed keeping some parameters constant and varying others. Finally, on the basis of the results, the PA curves of all the studied minerals have been obtained. Moreover, any discrepancies between the literature data [4] and our experimental data are discussed.
Experimental

Material and methods

The Stanton Redcroft STA 780 Series apparatus, used for the analysis, gives simultaneous thermogravimetric (TG), differential thermogravimetric (DTG) and differential thermal analysis (DTA) records, for sample amounts up to 100 mg, depending on density and packing.

The sample and reference material crucibles are in Pt-Rh with a volume of about 135 mm³ and a diameter of 6 mm. The thermocouples, in Pt-Rh too, are exterior to and in contact with the sides of the crucibles.

The analysis of the present study have been carried out in static air.

The microprocessor-based control unit of the electronic microbalance guarantees high accuracy in weight determination (around µg).

Identification of the minerals

The mineral studied by thermal analysis are the following four clay international standards:

1) kaolinite 5, Bath, South Carolina 48W0250;
2) halloysite 13, Dragon Iron Mine, Eureka, Utah 48W0130;
3) montmorillonite 22b (cream), Amory, Mississippi 48W1222;
4) montmorillonite 22a (cream), Amory, Mississippi 48W1221.

All these standards are supplied by the World’s Natural Science Establishment Inc., P.O. box 1712, Rochester, New York 14603.

In addition calcite and quartz samples, derived from large monocrystals, have also been studied.

Calcite, quartz and clay minerals have been previously subjected to chemical and XRD analysis and, only once their purity had been ascertained, they were studied by DTA.

Table 1 gives the chemical analysis for the major elements and their proportional formulas. For calcite some trace elements have also been determined, as their presence can have a marked effect on reaction temperatures [2]. However, Pb and Mn are only present in minute quantities or are absent altogether.

As mentioned in the foregoing, the temperature variation of the endothermic reactions has been calculated by differential thermal analysis for different minerals, changing the sample amount and keeping some other parameters constant. Table 2 shows the test conditions.

Calcined Al₂O₃ has been used as inert reference material in all the tests. As Table 2 shows, sample amounts ranging between 1 and 70 mg have been tested at heating rates of 10 deg/min, 15 deg/min and 20 deg/min. In the case of the montmoril-