

The Detection of Quartz in Talc by Differential Thermal Analysis

Principle

Differential thermal analysis (DTA) involves the measurement of thermal reactions which are induced in a sample as it is being heated at a constant rate. First-order thermal transitions are denoted as endothermic or exothermic depending upon whether the process is accompanied by the absorption or release of energy in the form of heat. The sample holder includes a reference thermocouple and a differential thermocouple, which detects thermal reactions by continuously monitoring any difference in temperature between the sample material and a thermally inert reference substance (calcined alumina) contained in another cavity or sample dish of the holder.

The DTA method permits the unequivocal detection of quartz (free crystalline silica) in talc at a 0.5 - 1.0% (w/w) minimum detectable level. The method utilizes the thermal transition representing the reversible alpha to beta crystal inversion of quartz at 573°C. On heating, the latent heat of inversion gives rise to an endothermic reaction; on cooling, an exothermic transition is obtained. The talc is first calcined at approximately 800°C for the purpose of inducing irreversible thermal transitions attributable to mineral impurities. The cooling curve then shows a flat baseline, thus improving detectability for quartz. Studies have shown that the intensity of this thermal peak is affected by the particle size distribution of the quartz. Therefore, DTA is not recommended for the quantitative determination of quartz.

Apparatus

1. Differential Thermal Analyzer, including either a high temperature powder sample holder of nickel or stainless steel construction with exposed-loop differential thermocouple (such as Platinel II) or sample holder employing a ring type differential thermocouple with platinum dishes, with equipment to heat to at least 800°C.
2. Spex Mixer/Mill*, or equivalent mechanical mixer.
3. Sieve, 325-mesh
4. Mortar and pestle, or grinding mill.

Reagents

1. Standard talc sample, containing no detectable quartz.
2. Standard quartz sample, at least 95% pure.

Procedure

Instrumental parameters must be determined for a particular differential thermal analyzer such that 0.5 - 1.0% (w/w) quartz in talc may be detected in the cooling curve subsequent to calcining the talc at 800°C. Once these experimental parameters have been determined, they must not be altered during the analysis of talc samples in order to assure instrument sensitivity.

1. Standard Preparation

- a. Grind standard talc and quartz samples to pass a 325-mesh sieve. This will give a particle size distribution of 0 - 44 μm .
- b. Weigh appropriate amounts of the ground standard talc and quartz into a plastic vial to prepare 1.0% (w/w) quartz-talc standard. Mix with a plastic ball approximately 10 minutes in Spex Mixer/Mill.

2. Instrumental Parameters

Experimental conditions for a typical DTA unit (Stone Model RC-202C or LA-XYH) are as follows:

Sample holder: high temperature powder type, of nickel or stainless steel construction (Model SH-8BE2), with exposed-loop differential thermocouple (Platinel II), imbedded reference thermocouple (Platinel II or Chromel/Alumel).

Temperature range: ambient to 800°C.

Sample: 130 - 150 mg. talc, using a loose, consistent packing technique.

Reference material: alumina, ground to pass 325-mesh sieve.

Atmosphere: static air

Heating rate: 10°C/min.

Sensitivity: 40 μvolts full scale

Furnace: LTF, water-cooled to 1200°C

It is emphasized that reproducibility of the method is based on standardizing the experimental conditions.

3. The prepared quartz-talc standard is run to optimize sensitivity of the instrument. If the quartz inversion endotherm (573°C) is masked by thermal transitions of mineral impurities in the talc, it is necessary to rely on a cooling curve for quartz detection. If there is no provision on the DTA instrument for programmed cooling, heat the talc to 800°C , cool to room temperature, and record the thermogram on reheating for detection of quartz.
4. Grind talc or talc ore samples to pass 325-mesh sieve to give particle size distribution comparable to standard sample. Quartz is detected by means of the crystal inversion (573°C) on heating or cooling.
5. If semi-quantitative estimation of the quartz content is desired, this may be achieved under fixed experimental parameters by comparison to quartz peak intensities obtained for additional quartz-talc standards. The particle size distribution of the quartz in talc samples and standards must be as consistent as possible.

Conclusion

The value of the DTA method lies in the specificity of the determination over that attainable by x-ray diffraction in the silicate mineral matrix. No other mineral has been reported in the literature to have a thermal transition peak at 573°C . Semi-quantitative DTA is feasible only under fixed experimental conditions and with some knowledge of quartz particle size.

Detection of Quartz in Talc by DTA

