

DIFFERENTIAL THERMAL ANALYSIS FOR SERPENTINE (CHRYSOTILE) IN TALC

1. The Differential Thermal Analyzer (DTA) permits the detection of chrysotile and other serpentine minerals in talc for cosmetic use. The talc must not contain greater than 10% (w/w) chlorite for this method to be applicable because of the possible interference of chlorite thermal transitions. The minimum level of detection is 0.5 - 1% by weight of chrysotile asbestos.
2. The instrumental parameters must be determined for the particular differential thermal analyzer which is employed, with regard to geometry of the sample holder, sensitivity of the differential thermocouple, etc., such that 0.5 - 1% by weight chrysotile may be easily detected.
3. The successful development of the DTA method is based on the preparation of homogeneous chrysotile-talc standard samples. Standards containing 0.5, 1, 3, and 5% by weight chrysotile in talc are prepared as follows:
 - a. Five gm. of the standard talc is slurried in about 100 ml. of ethanol in a Waring Blendor[®].
 - b. The appropriate weight of chrysotile asbestos of at least 95% purity is then added to the talc slurry, and the mixture is blended for 5 minutes at low speed.
 - c. The solution is washed into a beaker with ethanol, and the solvent is evaporated on a steam bath.
 - d. The resulting caked sample is broken and mixed in a plastic vial by shaking for 5 minutes in a mixer such as the Spex Mixer/Mill[®].
 - e. The homogeneity of the chrysotile-talc standards should be verified by optical or electron microscopy.
4. The experimental conditions for the Stone DTA unit (Models LA-XYH and RC-202C), for example, are as follows:

High temperature powder sample holder of nickel or stainless steel construction (Model SH-8BE2) with exposed-loop differential thermocouple (Platinel II).

Temperature range: ambient to 1000°C.
Reference thermocouple: Platinel II or Chromel/Alumel,
exposed-loop
Sample: 130-140 mg. talc, using a loose, consistent
packing technique.
Reference material: Alumina, ground to pass a 325-mesh
sieve (maximum particle size of
44 microns)
Inert dynamic atmosphere: helium or nitrogen at 0.05 SCFH
Heating rate: 10°C./minute
Sensitivity: 40 μ volts full scale
Furnace: LTF, water cooled to 1200°C.

The purpose of the inert dynamic atmosphere is to prevent oxidative reactions and to sweep out gaseous mineral decomposition products. The Stone powder sample holder yields maximum sensitivity by permitting direct contact of thermocouple and sample. True dynamic gas flow through the sample is also attained. It may be possible to use a more rapid heating rate with some DTA instruments, if it can be ascertained that constant thermal equilibrium exists between sample holder and furnace. Reproducibility of the method is based on standardizing the experimental conditions.

Experimental conditions for the duPont DTA unit (Models 900 and 990) would be as follows:

Temperature range: ambient to 1000°C.
Furnace: 1200°C.
Sample: about 40 mg. talc
Reference Material: alumina
Atmosphere: static air
Heating rate: 30°C./minute
Sensitivity: ΔT 0.004 mv./in.
T 0.8 mv./in.

5. Initially, a thermogram of the blank standard sample is run in order to determine the thermal profile of the talc.
6. A thermogram is run on chrysotile asbestos by diluting the material approximately 1:4 in fine alumina. The thermal transitions involve a dehydroxylation endotherm at about 650°C and a recrystallization exotherm at about 820°C. These two peaks establish the presence of serpentine mineral.
7. The 5, 3, 1, and 0.5% chrysotile in talc standards are then analyzed. Both thermal transitions of the chrysotile in the 1% standard should be easily detectable. If not, the experimental conditions must be altered to improve detectability.

8. Normally, talcs for cosmetic purposes will not require additional grinding for DTA. The analysis of talc rock or the presence of lumps or gritty material will, however, necessitate grinding the sample to pass a 325-mesh sieve.
9. The presence of large amounts of carbonates or bicarbonates (10% w/w or greater) in talc will obscure the serpentine exotherm at approximately 820°C. A mild acid leaching is used to remove these carbonates while leaving any chrysotile present intact:
 - a. Weigh out 2 g. of the talc into a 100 ml. beaker. Add 25 ml. of 10% HCl (v/v) slowly (to prevent excessive evolution of gas if carbonates are present) and heat, with occasional stirring, on a steam bath for 5 minutes.
 - b. Filter on a vacuum filter, in either a porcelain cone with glass fiber filter mat or a porous glass bottom cup, and wash several times with hot water. Dry the talc.
10. When there is insufficient talc available to fill the entire sample cavity or cup, a lesser amount of the talc may be placed in the bottom of the cup and covered with fine alumina, or packed as a "sandwich" surrounding the thermocouple, between layers of fine alumina. In this case, the standard samples should be packed and run by the same technique to determine the corresponding decrease in peak intensities.
11. If serpentine mineral is indicated in a talc sample by the presence of the two characteristic thermal transitions, the thermogram should be rerun on another sample of the powder to verify these peaks. Because of the sensitivity of the powder sample holder with exposed-loop thermocouple, spurious heat effects caused, for example, by slight shifting of the sample during the run, can occasionally produce minor baseline fluctuations. Therefore, very weak thermal peaks should be checked for reproducibility.