

Detection of Quartz in Talc by X-ray Diffraction

Principle

X-ray diffraction is a convenient method for determining the presence of crystalline impurities, such as quartz, in talc. It is necessary that at least the three strongest diffraction lines of quartz be present in the x-ray pattern to confirm the presence of quartz in the talc: 3.34_{x} , 4.26_4 , 1.82_2dA .

It has been experimentally determined that compression of a talc sample containing quartz into a pellet results in an x-ray diffraction pattern having more intense quartz peaks than the pattern obtained from a packed-powder sample of the same talc. Using this pressed pellet technique, it is possible to detect the three strongest quartz peaks at a minimum level of 2% w/w.

Apparatus

1. X-ray diffractometer with nickel-filtered $\text{CuK}\alpha$ radiation and a suitable 1-1/4" circular sample holder for the pelletized sample.
2. Hydraulic press capable of maintaining from 15,000-24,000 lb. (as calculated on a 3" ram).
3. 1-1/4" pellet press.

Reagents

1. Boric acid

Procedure

1. Sample preparation

Approximately 2 gm of sample talc is transferred to the die-holder and evenly distributed on a polished, scratch free die; approximately 4 gm of boric acid is then distributed on top of the talc layer. The two layers are then compressed into a pellet whose talc face is free of flaws¹.

¹The amount of pressure necessary for a good talc face may vary from talc to talc and can only be experimentally determined.

2. Instrumentation

The sample pellet is x-ray scanned through three critical quartz regions under instrumental conditions which have previously been determined sensitive to the 2% w/w quartz level.

As an example, the following conditions have been employed on the Siemens Diffractometer (Model No. M386-X-A4) and Siemens Counter and Recorder (Type T):

Radiation: Cu with K_{β} filter at 40 KV and
24 ma
Divergence slit: $1/2^{\circ}$ Receiving slit: 0.2 mm
Goniometer speed: $1/2^{\circ}$ 20/minute
Recorder speed: 600 mm/hour
Attenuation = 1×10^3 impulses/second
Time constant, T [S] = 1
Rise time = 0.18 Attenuator = 20

3. X-ray Diffraction Scans

The sample pellet is first scanned from $25.5-27.5^{\circ} 2\theta$ for the presence of a peak at $26.7(\pm 0.1)^{\circ} 2\theta$. If no peak is detected, quartz is absent at the lower limit of detection.

If a peak is observed in the above scan, the pellet is then scanned through the $19.5-21.5^{\circ} 2\theta$ region for a quartz reflection at $20.8(\pm 0.1)^{\circ} 2\theta$. The absence of a peak confirms the absence of quartz at or above 2% w/w.

If both peaks are observed, the sample is then scanned from $49.0-51.0^{\circ} 2\theta$ for the presence of a peak at $50.1(\pm 0.2)^{\circ} 2\theta$.

The presence of all three peaks confirms that the talc contains quartz at a level equal to or greater than 2% w/w.