

XRD Quartz Reference Standard		Procedure#: LAB WI 633
Issue Date: Nov 9, 2012	Issue#: 001	E-P Reference#: n/a
Reference Method(s): n/a		Page 1 of 2

1) REVISION HISTORY

Issue Number	Issue Date	Reviewed By	Approved By	Description of Changes
001	Nov 9, 2012	Corporate Quality Manager Sr. Research Scientist	Corporate Quality Manager Sr. Research Scientist	Documenting previously defined procedure

- 2) **PURPOSE** - This purpose of this procedure is to guide individuals with previous experience in operating the D-5000 x-ray diffractometer in using the quartz reference standard to determine changes in x-ray tube intensity.
- 3) **SCOPE** - This procedure should be used once per month or after every 100 samples, whichever comes last. It applies to all x-ray diffraction work performed using the Siemens D-5000. The quartz intensity ratio shall be used to adjust subsequent peak intensities (by dividing said intensities by the ratio) when quantifying crystalline silica phases.
- 4) **RESPONSIBILITY/ OWNER** - Sr. Research Scientist
- 5) **EQUIPMENT AND MATERIALS REQUIRED** -
 - a) Siemens D-5000 X-ray Diffractometer plus associated equipment and controlling software
 - b) Quartz reference standard (mounted on aluminum sample holder)
- 6) **SAFETY/REQUIRED PPE** – Safety glasses must be worn in the lab. Observe radiation safety precautions.
- 7) **ENVIRONMENTAL** – There are no environmental concerns.
- 8) **RECORDS / FORMS** – No set format required. Records must be kept in accordance with each site’s ISO 9001 records procedure relating to calibration or verification records. Results from quartz calibration runs should be recorded within the first 10 pages of the XRD laboratory notebook. Results should include the date of testing, the primary quartz peak net intensity, and the ratio (%) of current peak intensity to that recorded when quartz and cristobalite control standards were analyzed.
- 9) **PROCEDURE OR WORK INSTRUCTION**
 - a) Locate the quartz reference standard and energize the diffractometer to 36mA and 50KV.
 - b) Place the quartz reference sample (and holder) into the sample spinner, and set the testing parameters (using DataScan 4) to the following:
 - i) 2-theta range = 25.0 to 28.0
 - ii) Step-size = 0.04^o
 - iii) Step Time = 4.0 seconds per step
 - iv) Sample spinner at 15 rpm
 - c) Run the sample.
 - d) Using Jade software, determine the peak net intensity of the primary quartz peak, and record this value.
 - e) Check the secondary quartz peak intensity, along with any interfering phases, and adjust the primary quartz peak net intensity accordingly.
 - f) Calculate the intensity ratio and record this value.

XRD Quartz Reference Standard		Procedure#: LAB WI 633
Issue Date: Nov 9, 2012	Issue#: 001	E-P Reference#: n/a
Reference Method(s): n/a		Page 2 of 2

10) CALCULATIONS –

- (1) **Quartz Intensity Ratio** = I_b / I_a where I_b = current quartz standard net intensity and I_a = quartz standard net intensity at the time of analysis of quartz and cristobalite control standards. This ratio can be expressed as a percentage by multiplying by 100.

11) HARD COPY LOCATIONS - R&D Department and Corporate Quality Manager.