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When coupled with the ability to oscillate while rotating the sample, these benefits are achieved:

- Sample oscillation and rotation expose a larger area of the sample surface to the X-ray beam. This is important if the crystallites are non-uniform in size or non-uniformly distributed in the sample.
- The area detector observes a large section of the  $\chi$  ring which covers signals from oriented crystallites.
- The adjustable detector distance enables the resolution to be optimized for a particular application, depending on interfering peaks in the matrix.

Although the sample was rotated and oscillated during collection of this frame, the quartz ring is not smooth and contiguous. Each bright spot on the quartz is due

to exposure of a large oriented crystallite. When this  $\chi$  ring is integrated, the average intensity of all exposed crystallites is obtained. In comparison, a scintillation (point) detector would cover only a narrow strip along the  $2\theta$  direction, potentially missing important data.

Crystallite size analysis can be coupled with this quantitative technique for applications in which the size distribution of the crystallites is also of importance. In this case, peak shape and width are evaluated to calculate the crystallite size distribution.

#### Experimental Conditions

System	Bruker AXS GADDS System with HI-STAR Area Dector
Sample Stage	Two-position $\chi$ stage with sample rotation/oscillation attachment
Source	Sealed tube, Cu anode, 40 kV/50 mA

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# GADDS: IMPROVED SAMPLING STATISTICS WITH ROTATION/OSCILLATION STAGE

## Quantitative Analysis – Preferred Orientation – Area Detector – GADDS

X-ray diffraction is currently being applied to a wide range of quantitative applications. In addition to traditional applications, such as determination of free silica in respirator dust and quartz and tremolite in talc, X-ray diffraction is now employed in the medical field for analysis such as the degree of drug crystallization in epidermal patches. In all quantitative applications, sampling techniques are critical to achieving accurate results.

In addition to sample preparation, data collection strategies and instrument configuration play an important role in assuring that a random sampling of crystallites are exposed to the X-ray beam and that the resulting diffraction signal is detected. The most critical of these issues is that of oriented crystallites. Most conventional quantitative X-ray diffraction instruments have the ability to rotate the sample in  $\phi$ , which decreases but does not eliminate the effects of preferred orientation in the sample. A two-dimensional detector, capable of collecting a large area of the diffraction cone, greatly reduces poor statistics due to preferred orientation effects.

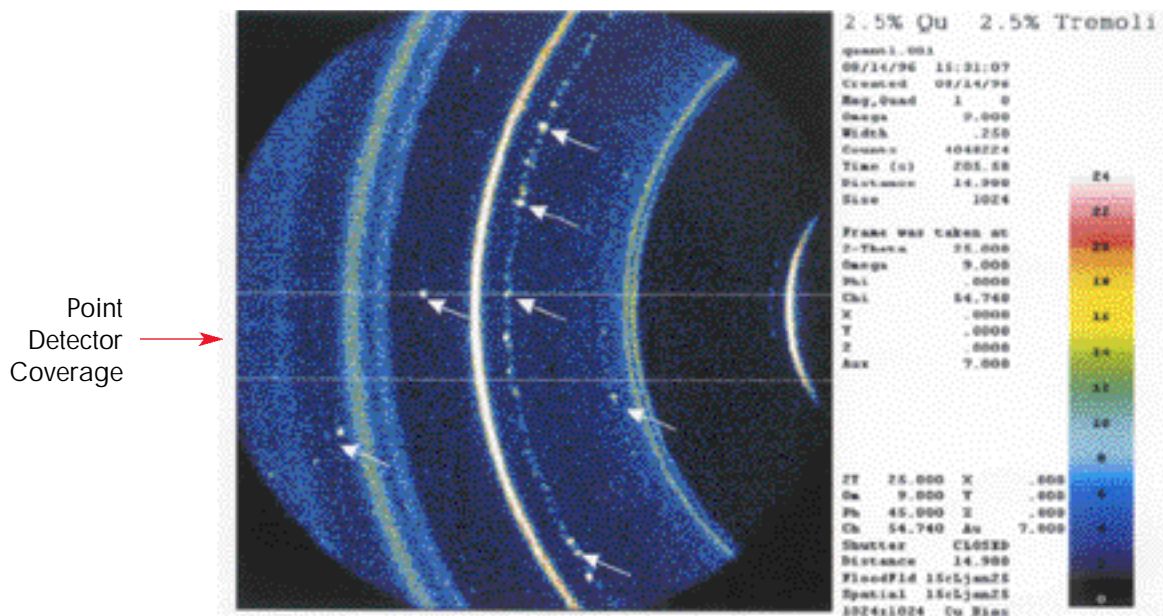


Figure 1 – Area detector frame for quartz detection in talc

