**PARTICLE STATISTICS ENHANCEMENT FOR XRD ANALYSIS USING VIBRATING SAMPLE HOLDERS**

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The X-ray powder diffraction method is based on the assumption that a very large number of randomly-oriented crystallites are presented to the X-ray beam. "Particle statistics" is often used to refer to this condition. Poor particle statistics can produce potentially large inaccuracies in peak intensities. Good particle statistics are commonly obtained by grinding specimens to a very fine grain size (< 5 µm, dependent on specimen composition and radiation). The analysis of very small specimens or non-ideally fine powders requires the use of methods to increase the number of crystallite orientations explored during the XRD measurement. The most common approach consists of moving the specimen in the beam to analyze either a larger number of crystallites (translations) or to explore more orientations of a number of crystallites (rotations). We are developing an alternate approach based on random motions generated within the specimen to increase the *effective* number of crystallites and their orientations analyzed. Random motions are induced by sonic or ultrasonic vibrations applied to a sample holder to fluidize the powder and obtain either a flow of material through the system or internal motions by granular convection. This method greatly improves particle statistics for specimens that are too coarse grained to be analyzed in a conventional manner.

Vibrating sample holders can be designed for dry powders or liquid suspensions with a variety of diffraction geometries. Instruments fitted with position-sensitive detectors are preferable because they provide a longer integration time for each angular position, allowing better cumulative particle statistics with moving specimens. Vibrations can also be used to fluidize powder for automated sample loading and removal.

Developed initially for a miniature XRD/XRF instrument designed at NASA for planetary exploration, this technique has proven to be equally useful for laboratory instruments. Dramatic improvement in data quality is observed when analyzing coarse-grained materials. For example, organic powders with grain sizes as large as several hundred micrometers can yield accurate peak intensities, allowing unequivocal phase identification within a few minutes with virtually no sample preparation.

The vibrating sample-holder technique will be helpful for powder diffraction analysis of drugs whenever fine-grained powders cannot be prepared, e.g., for high-throughput analysis, process monitoring, analysis of grinding intolerant materials and the like. Systems optimized for analyses of pharmaceuticals are under development.