

MICRO-SAMPLE X-RAY POWDER DIFFRACTION. APPLICATIONS IN THE PHARMACEUTICAL SCIENCES

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A micro-gram X-ray Powder diffraction method is described that employs nylon loops and/or PET (heat shrink) capillaries as sample mounts. The nylon loops and/or PET capillaries are used to position the sample in a three-circle X-ray diffractometer in such a way as to allow for Gandolfi-like scans. These scans maximize mechanical tumbling of the sample, which in turn results in higher quality data. The diffractometer utilizes monochromatic radiation, a pinhole source and a two-dimensional area detector for rapid data collection. The method can also be readily automated by employing single-crystal screening procedures. Applications to the pharmaceutical sciences include: High-throughput polymorph and salt screening, quality control, by-product identification, process analysis and structure solution from powder data. The micro-gram methodology allows for more "routine" use of x-ray powder diffraction in pharmaceutical applications by providing a procedure to quickly mount and collect sample data.