

ICDD ROUND ROBIN ON QUANTITATIVE RIETVELD PHASE ANALYSIS OF PHARMACEUTICALS

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The ICDD sponsored a round robin on the quantitative Rietveld phase analysis of pharmaceuticals. Eleven participating laboratories from the pharmaceutical community submitted both raw data and processed quantitative results. The purpose of the round robin was to evaluate current practices in laboratories, so procedures and methods were not specified, but they were recorded. Cluster analysis tools were applied to all the data sets and their use helped identify the root causes of several types of errors in specimen preparation, data treatment and Rietveld analysis. The authors considered this round robin to be difficult. Sample homogeneity was an issue and molecular orientation was observed in many data sets. Each material studied has structural polymorphs so selection of starting parameters and their refinement was non-trivial. Similar to prior round robins on inorganic materials and minerals, this round robin identified operator errors as the major contributor to poor results. Four laboratories achieved excellent results on all phases in all three samples, with accuracy within relative errors of 5-10%. Participating Scientists and Laboratories:

Y. Ososkov and Z. Cherbanyk Beti, Exova, Mississauga, Canada
A. Patel, Bristol-Myer Squibb, New Brunswick, NJ, USA
J. Wright and A. Fitch, European Synchrotron Radiation Facility (ESRF), Grenoble, France
P. Varlashkin, GlaxoSmithKline, Durham, NC., USA
F. Needham, ICDD, Newtown Square, PA, USA
M. Ermrich, Roentgen Laboratory, Reinheim, Germany
X. Bokhimi, Universidad Nacional Autonoma De Mexico, Coyoacan District, Mexico
R. Suryanarayanan, University of Minnesota, College of Pharmacy, Minneapolis, MN, USA
J. Henao, Universidad Industrial de Santander, Columbia
E. Wachtel, Weizman Institute of Science, Rehovot, Israel
H. Brusova, Zentiva AS, Prague, Czech Republic

The round robin was a tremendous learning experience. The samples were challenging and the analyses and their interpretation were non-trivial. Despite challenges in all the steps of analysis including specimen preparation, data treatment and Rietveld refinement, excellent results were achieved by four out of eleven laboratories.

The purpose of the round robin was not to define best practices, but to evaluate the state of analysis and evaluate procedures used in pharmaceutical laboratories. Laboratories were asked to use their standard procedures and record the results. The diversity in the quantitative analysis results from the round robin reflects the diversity observed in specimen preparation and analysis methods.

The fact that a group of four independent laboratories achieved excellent results suggests that the technique is robust and standardized practices would be expected to yield reproducible accuracy and precision.