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Processing Extremely Large Powder Diffraction Datasets Using The Rietveld Method

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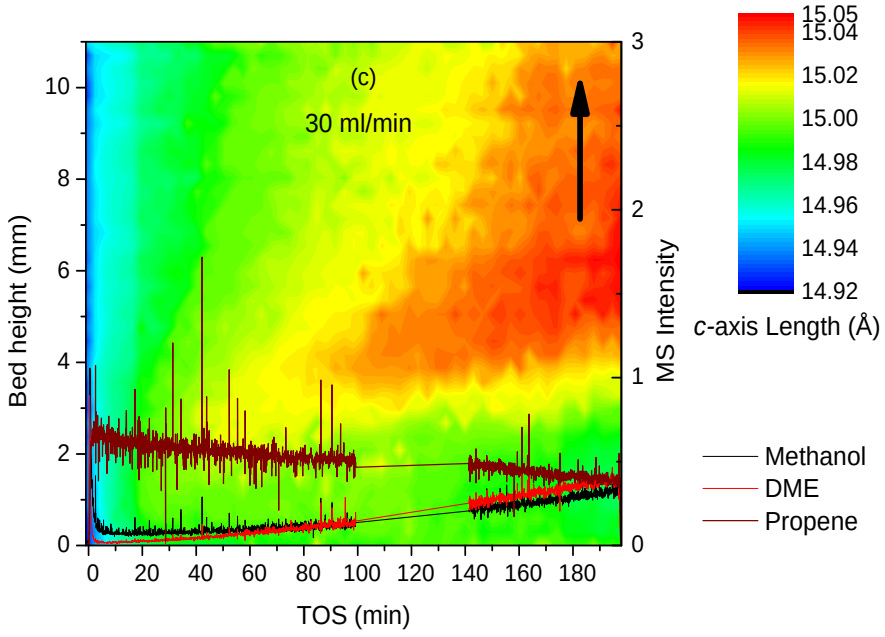
Modern in situ synchrotron powder diffraction experiments can produce massive volumes of data which are of suitable quality for real structural information to be extracted. Parametric Rietveld refinement (Stinton, 2007) is an ideal method for dealing with such datasets as a huge number of diffraction patterns can be processed in parallel while the number of refined parameters is reduced by linking between scans. The time saving compared to earlier sequential methods of refinement using batch files is very significant with processing times being reduced from weeks to a few hours. The stability of the parametric method allows not only extraction of information from data with very weak trends but also refinement of entire slices of a tomographic map including the regions with zero diffraction. The power of the technique will be illustrated by examples from reactor scanning experiments with high time resolution (Wragg, 2012, 2013) as well as more conventional in situ powder diffraction and operando experiments combining diffraction with mass spectrometry. The extraction of structural information from complete tomographic datasets and reconstructions with real structural parameters will also be demonstrated. The figure shows time and space resolved c-axis data for the SAPO-34 catalyst during methanol to olefin conversion, together with mass spectrometry data collected during the experiment.

[1] *G. W. Stinton, J.S.O. Evans, 2007, J. Appl. Cryst., 40, 87-95,* ***[2]*** *D.S. Wragg, M.G. O'Brien, F.L. Bleken, M. Di Michiel, U. Olsbye, H. Fjellvåg, 2012, Angew. Chem. Int. Ed. Engl., 51, 7956-7959,* ***[3]*** *D.S. Wragg, F. L. Bleken, M.G. O'Brien, M. Di Michiel, H. Fjellvag, U. Olsbye, 2013, Phys. Chem. Chem. Phys., 15, 8662-8671*



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