Structure Determination of Zeolites: Making all the Pieces Fit

Karl G. Strohmaier, Mobae Afeworki, Gordon J. Kennedy, Douglas L. Dorset, *ExxonMobil Research & Engineering Co. Rt. 22 East, Annandale, NJ USA*. E-mail: karl.g.strohmaier@exxonmobil.com

In recent years new powder diffraction programs such as FOCUS [1] and EXPO [2] and simulated annealing techniques [3] have been developed to help solve zeolite structures, since these microporous materials are typically synthesized with crystal sizes too small for conventional single crystal structure determinations. Auxiliary techniques are usually employed to assist the researcher in solving a new framework structure. A correct unit cell and space group determination can be aided by electron diffraction, and the number of unique T-atoms and their connectivity can be determined by MQMAS and MQ-HETCOR NMR techniques. Gas absorption measurements indicate the size of the micropores and the dimensionality of its channels, while crystal density gives information about the total number of tetrahedral atoms in the unit cell.

A successful structure determination should not only ensure that the calculated diffraction pattern closely matches the experimental pattern, but also that *all the pieces fit,* i.e., all the characterization data support the proposed model. The above-mentioned and other techniques were used to elucidate the structures of zeolites ECR-34 and SUZ-4 and the aluminophosphates ECR-40 and EMM-3. The supporting characterization data were found to be essential for determining these new structures from powder diffraction data.

[1] Grosse-Kunstleve R., McCusker L.B., Baerlocher Ch., *J. Appl. Cryst.*, 1997, **30**, 985. [2] Altomar A. et al., *J. Appl. Cryst.*, 1999, **32**, 339. [3] Falcioni M., Deem M.W., *J. Chem. Physics*, 1999, **110**, 1754.

Keywords: x-ray powder diffraction, zeolites, NMR spectroscopy