

W0238

Electron Crystallography of Zeolites - Direct 3-D Determination of the MWW Framework. D.L. Dorset, CSR, ExxonMobil Research & Engineering Co., Annandale, NJ 08848, C.J. Gilmore, Dept. of Chemistry, Univ. of Glasgow, Glasgow, G12 8QQ Scotland.

Recently, 3-D measurement of electron diffraction intensities at 300 kV from three calcined members of the MWW family of zeolites (MCM-22, MCM-49, ITQ-1) revealed their frameworks to be identical, aside from differing $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratios. A composite data set comprising 155 reflections was collected in space group P6/mmm, where $a = 14.21$, $c = 24.94$ Å. The samples are thin plates so that the amplitudes of strongest reflections are well-matched by the framework model ($R = 0.23$). *Ab initio* direct phase determination was carried out using maximum entropy and likelihood methods in the program MICE. The best solution yielded a good match to the actual hk0 phases (rms error 45°) and the [001] map revealed positions of all T-atoms and oxygen connectors. The 3-D solution was less optimal, phase error 53° for the 27 most intense reflections, but the map densities served as an adequate envelope to the structural framework. Attempts to overcome the sampling problem caused by the missing cone of data (beyond 60° tilt) were made by a Sayre extension into the unknown region of reciprocal space, yielding a somewhat distorted structure that could be adjusted easily to the actual MWW framework.