High resolution synchrotron x-ray powder data have been collected from a well-crystallized and highly de-aluminated sample of the zeolite ZSM-11 (space-group \( \text{Ia}3d \), \( a = 20.065 \text{\AA}, c = 13.408 \text{\AA} \)) on the dedicated triple-axis powder diffractometer X13A at the Brookhaven National Synchrotron Light Source (Cox, Hastings, Cardoso, and Finger, Materials Science Forum (1986), Vol. 9, edited by C.R.A. Catlow, I-20 Trans Tech Publications, Switzerland). A perfect Ge(111) crystal scattering in the horizontal plane at a wavelength of 1.54155 \( \text{\AA} \) was used as monochromator, with a flat-plate sample and a perfect Ge(220) analyzer scattering in the vertical plane. The peak shapes were symmetric and well-described by the convolution of Gaussian and Lorentzian functions, with a peak-width of about 0.04° at low angles. This is about double the instrumental resolution in this region, consistent with a mean particle size of about 0.4 \( \mu \text{m} \). The high resolution of the data is illustrated in Fig. 1.

Structure analysis was accomplished by Rietveld refinement with three Gaussian and two Lorentzian half-width parameters in the following form:

\[ T_\theta = \frac{1}{T_{\text{max}} + T_{\text{min}} + W/2} \]

\[ T_\theta = \frac{1}{T_{\text{max}} + T_{\text{min}} + W/2} \]

The data set contained contributions from 679 reflections. The framework topology of ZSM-11 was previously derived by distance least-squares modelling (Kokotailo, Chu, Lawton and Meier, Nature (1978) 275, 119) and contains seven inequivalent Si and 15 inequivalent O atoms. In the final stages of refinement, 86 parameters were varied including 54 positional coordinates and 22 individual isotropic temperature factors. Refinement converged to the following R-factors: \( R_1 = 0.11 \), \( R_w = 0.22 \), \( R_e = 0.15 \) (goodness-of-fit \( \chi^2 = 2.0 \)). With three exceptions the Si-O bond lengths fall in the range 1.57 - 1.67\( \text{\AA} \). Difference Fourier plots showed no residual features greater than about 5% of the oxygen peaks, indicating the absence of significant amounts of extra-framework species.

Rather unexpectedly, high resolution magic-angle spinning NMR spectra show more than seven resonances at room temperature, indicative of deviations from the long-range crystallographic symmetry. These disappear at 100°C, where only the expected seven resonances are observed.

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