07.9-2 SYNTHESIS AND CRYSTALLOGRAPHIC DATA ON A NEW SILICA ZEOLITE, SILICA-ZSM-48. By J.L. Schlenker, Department of Chemistry, University of Pardense, Sci Lanka & H.Gies, Mineralogy Institute, Kiel University, West Germany.

A high silica zeolite ZSM-48 has been synthesized (Schlenker, J.L. et al., Zeolites, 1985, 5, 355-8) from silica solutions containing trace amounts of aluminum. In the present study the aluminum-free end member of the zeolite ZSM-48 i.e. silica-ZSM-48, has been crystallized hydrothermally at 16°C - 225°C from a pure silicic acid solution in the presence of polysilanes as templates. Triethylammonium, tetramethylepentamethane, 1,8,12-tetrametadodecanate etc act as efficient templates in this synthesis. Microprobe analysis of the product confirmed the absence of aluminum in the framework.

The X-ray powder diffraction pattern was indexed on the basis of an orthorhombic cell and the least square refinement using X-ray powder data gave the unit cell parameters a = 19.12(1) Å, b = 20.14 Å and c = 28.0(3) Å. Powder reflections indicate C or I centering suggesting Immm, Imma, Cmm and Cmma symmetries. Rietveld studies on silica-ZSM-48 confirmed the lattice parameters obtained from X-ray powder data. All specimens investigated by TEM show weak reflections indicating the space groups Pnma or P21/m. Disregarding the white reflections the space group Imma is found to be the most plausible.

Twinned silica-ZSM-48 is a common feature and the twin plane is perpendicular to [010]. It is apparent from these data that a structure based on ferrierite sheets linking via oxygen atoms located on mirror planes and consisting of 1-D channel systems may be visualized for silica-ZSM-48. The 10-7 ring channels are running parallel to the c axis. Its framework topology appears to be closely related to that of ZSM-23 and ZSM-22. Further work is in progress to grow single crystals in order to confirm the structure by single crystal structure refinement.

07.9-3 HYPOTHETICAL FRAMEWORK STRUCTURES RELATED TO MÖBIL ZEOLITE ZSM-48. By J. L. Schlenker and W. J. Rohrbach, Mobil Research and Development Corporation, Research Department, Paulsboro, NJ 08066, USA. ZSM-48 is a high-silica zeolite whose structure has been proposed to be a disordered linking of ferrierite sheets via bridging oxygen atoms located on mirror planes. The proposed framework topology was based on agreement between observed and calculated x-ray powder diffraction patterns. The Smith plot in best agreement was obtained from a disordered intergrowth of two ideal framework structures with Cmca and Imma symmetries. Because x-ray and electron diffraction data seem to indicate an orthorhombic lattice with pseudo-I or pseudo-C-centering, attention was initially restricted to the formulation of hypothetical centered structures. With lattice parameters of a=14.24 Å and b=20.14 Å, and a C- or I-centered orthorhombic lattice, the ferrierite sheet has only four independent T-atoms, each of which may point up (U) or down (D) in order to link with other sheets. Alternating the orientation of these independent T-atoms yields twenty-eight closely related hypothetical framework structures, e.g. UUDD-Cmca, UUDD-Imma, etc.


Octahedrally shaped crystals of the zeolite up to 0.7mm in size were grown hydrothermally from a gel with a composition 3Na2O.16(TMA)O· 18(TMA)2.OAl2O3·110Si02.730OH.02H2O, for 7 days at 473 K. A spherically ground transparent crystal with r = 0.19nm was investigated on a CABASDP diffractometric system. Crystal data: (Na,TMA)4.4Al14.5Si31.5P2.972. Mr = 6337, cubic, Fd3m, a = 19.396(1) Å, V = 27927(1) Å3, Z = 1, Dc = 1.897 g.cm-3, λ(MoKα) = 0.71073 Å, μ = 6.8 cm-1, F(000) = 4180, T = 291 K, From 8938 total 65399 and 316 unique reflections with 1(x)e(1) were used in calculations. Final R(F) = 0.035 by full-matrix least-squares. Residual max. Δ(ρ) = 0.09 e.A-3.

The ZSM-39 topology, known from the powder data study (Schlenker J.L. et al., Nature, 1981, v. 294, 340-2) was confirmed. Besides, disordered Al, Na and TMA were localized in the 16-hedral cage. The Al-atom occupies a tetrahedron which shares face with another, currently vacant SiO4-tetrahedron from the framework. The apical oxygen atom of A1O4 is pointed to the centre of the cage, where both the Na and TMA are disposed. The electron density peak near the centre of the 12-hedral cage is presumably a water-oxygen.


Single crystals of zeolites were synthesized in size large enough to study their crystal structure especially ZSM-11 (MEI) and ZSM-39 (MTW) known up to now only by their powder x-ray diffraction diagrams.

One MEI single crystal measuring 35 x 25 x 14 μm with [100] and [001] faces allowed us to assign it to space group P4212 in place of I422 suggested by Kokotailo et al. (Nature, 1978, 275, 115-6). The following parameters were obtained: a = 19.93(1) Å, c = 13.35(1) Å, V = 3260(3) Å3. Independent reflections were collected using CAD4 diffractometer allowing us to index ZSM-11 Powder data without any ambiguity and confirming a non-centered space group. The Kokotailo framework hypothesis must be called into question.

A quasi-spherical single crystal of MTW, 54μm in diameter, was prepared and studied with MoKa. Faces are [100], [111] and [211], space group Fd3m and unit cell parameter a=19.43(1)Å. Data and refinements confirm the framework hypothesis given by J.L. Schlenker et al. (Nature, 1981, v. 294, 340-2) from powder x-ray diffraction data.