

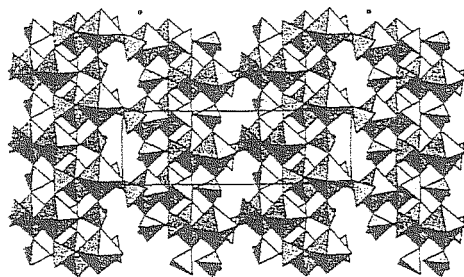
and their dependence on such synthetic parameters as pH, sulfur content, temperature and aging, the transformations from starting materials. We have done this under real-time hydrothermal conditions using powder diffraction and an IP at beamline X7B. From these results, information on the kinetics of formation, useful to optimize future syntheses, has been obtained.

**PS10.10.13 THE SYNTHESIS AND CRYSTAL STRUCTURES OF ALKALINE EARTH METAL INDIUM PHOSPHATES.** Xuejiao Tang and Abdessadek Lachgar, Chemistry Department, Wake Forest University, Winston-Salem, NC 27109-7486.

Metal phosphates with open framework structures are well known for their adsorptive, catalytic and ion-exchange properties. Al and Ga phosphates have been widely studied and exhibit great structural variations. In contrast, research of In phosphates has been very limited in scope. As a part of our search for open framework metal phosphates, we have recently been investigating ternary alkali or alkaline earth metal indium phosphates using hydrothermal synthesis techniques.

We report the syntheses and crystal structures of alkaline earth metal indium phosphates  $A[\text{In}_2(\text{PO}_4)_2(\text{HPO}_4)]$  ( $A=\text{Ca}$ ,  $\text{Sr}$  or  $\text{Ba}$ ).  $\text{Ca}[\text{In}_2(\text{PO}_4)_2(\text{HPO}_4)]$  was synthesized hydrothermally from stoichiometric amounts of  $\text{CaO}$  and  $\text{InCl}_3$  in excess  $\text{H}_3\text{PO}_4$  and  $\text{H}_2\text{O}$ . The compound crystallizes in monoclinic symmetry, space group  $P2_1/n$ ,  $a=6.5708(6)$ ,  $b=20.237(2)$ ,  $c=6.6572(7)\text{\AA}$ ,  $\beta=91.20(1)^\circ$ . The structure contains  $\text{In}_2\text{O}_{10}$  dimers built up of two edge-sharing ( $\text{InO}_6$ ) octahedra. The dimers connect to each other through ( $\text{PO}_4$  or  $\text{HPO}_4$ ) tetrahedra by sharing all of their oxo ligands. Cations are located in tunnels of 8-member ring opening running along [001] direction. To our knowledge, this is the first reported indium phosphate containing  $\text{In}_2\text{O}_{10}$  dimers.

$\text{Sr}[\text{In}_2(\text{PO}_4)_2(\text{HPO}_4)]$  was obtained hydrothermally from  $\text{Sr}(\text{OH})_2$  and  $\text{InCl}_3$  (1:1) in excess  $\text{H}_3\text{PO}_4$  and  $\text{H}_2\text{O}$ . Space group  $P2_1/n$ ,  $a=6.615(1)$ ,  $b=20.351(3)$ ,  $c=6.752(1)\text{\AA}$ ,  $\beta=91.00(1)^\circ$ . Its structure is analogue to  $\text{Ca}[\text{In}_2(\text{PO}_4)_2(\text{HPO}_4)]$



**PS10.10.14 ORIENTATION OF NAPHTHALENE IN H-ZSM-5 AS DETERMINED FROM POWDER AND SINGLE CRYSTAL XRAY DATA.** H. van Koningsveld and J. C. Jansen, Labs. of Applied Physics and Organic Chemistry and Catalysis, Delft University of Technology, Lorentzweg 1, 2628 CJ Delft, The Netherlands

The adsorption properties of frameworks with the MFI topology (H-ZSM-5 and its Al-free analogue silicalite-1) have received much attention. There is a general agreement that in low-loaded MFI/adsorbent systems the preferred adsorption site is at the intersection of channels.

Recently published papers on the localization of naphthalene (nph) in H-ZSM-5 determined by X-ray Powder Diffraction (hereafter referred to as XPD1 [Mentzen et al.; Zeolites, 13 (1993) 485] and XPD2 [Klein et al.; Microporous Materials, 3 (1994) 291]) show inconsistent results.

In both papers the structure is described in the orthorhombic space group  $Pnma$ . In XPD1 as well as in XPD2 the nph molecules (3.8 and 3.0

mols/u.c., respectively) are at the intersection of channels. However, the inversion of the unit cell axes  $a$  and  $b$  ( $a/b < 1$ ; the empty orthorhombic HZSM-5 framework has  $a/b > 1$ ), as observed in XPD1, is not reported in XPD2. In addition, the orientation of the nph molecules at the intersection of channels in XPD1 and XPD2 is quite different.

We succeeded in preparing a single crystal of the H-ZSM-5 zeolite loaded with 3.68(2) molecules nph per u.c., large enough to allow a single crystal X-ray diffraction study of the material.

The paper describes the structure of the H-ZSM-5/nph complex, gives the ensuing deformation of the channel pores and compares the orientation of nph as determined from powder and single crystal X-ray diffraction. The inversion of the  $a$  and  $b$  axes is confirmed and the orientation of nph is yet different from the orientations reported in the XPD-papers.

## Materials XI Fullerenes

**MS10.11.01 DETERMINATION OF THE ENDOHEDRAL NATURE OF THE METALLOFULLERENE  $\text{Y}@\text{C}_{82}$  BY MEM.** M. Takata, E. Nishibori, B. Umeda, M. Sakata, M. Ohno, H. Shinohara & Y. Saito\*, Nagoya University, Nagoya 464-01, Japan, \*Mie University, Tsu 514 Japan

The first conclusive evidence of endohedral nature of the Metallofullerene  $\text{Y}@\text{C}_{82}$  has been obtained via a Synchrotron X-ray powder diffraction study using the Maximum Entropy Method (MEM). Recently, the synthesis of fullerenes encapsulating various metal atoms within the carbon cage (endohedral metallofullerenes) has stimulated wide interest because of their unusual structural and electronic properties. Observations using STM, EXAFS, HRTEM and ESR have strongly suggested that the metal atoms are indeed inside the fullerene cage. Theoretical calculations also indicate that this is the case. But until now, no structural model has been derived experimentally to confirm the endohedral nature of the metallofullerenes. The  $\text{Y}@\text{C}_{82}$  fullerene was separated and isolated by the two-stage high performance liquid chromatography (HPLC) method. The purity of the  $\text{Y}@\text{C}_{82}$  fullerene was more than 99.9%. An X-ray powder pattern of  $\text{Y}@\text{C}_{82}$  was measured by using Imaging Plate at Photon Factory BL-6A2. The wavelength of incident X-rays is  $1.0\text{\AA}$ . The space group is assigned to  $P2_1$ , monoclinic. The experimental data were analyzed in an iterative way of combination of Rietveld analysis and the MEM. The reliable factor of the obtained MEM charge density is 1.4%. In the MEM charge density, there exist remarkably high densities just inside the  $\text{C}_{82}$  cage. The number of electrons around the maxima is about 38 which is very close to the atomic number of a yttrium atom. Evidently, the density maxima at the interior of the  $\text{C}_{82}$  cage is the yttrium atom. The present study revealed the fact that the yttrium atom is displaced from the centre of the  $\text{C}_{82}$  molecule and is strongly bound to the carbon cage.

**MS10.11.02 X-RAY DIFFUSE SCATTERING AND INTER-MOLECULAR INTERACTIONS IN SOLID  $\text{C}_{60}$ .** R. Moret, P. Launois and S. Ravy, Laboratoire de Physique des Solides, URA CNRS 02, Université Paris-Sud, 91405 Orsay, France

Oriental ordering phenomena control the temperature-pressure phase diagram of solid  $\text{C}_{60}$ . At present, their understanding is imperfect because microscopic models fail to describe the interactions between the fullerene molecules in details.

Above the  $T_0=259\text{K}$  orientational ordering transition the molecules do not rotate freely in the cubic crystal-field and significant short-range orientational correlations are present. They produce radial and azimuthal modulations of the diffuse scattering intensity which have been measured carefully in the first halo ( $Q \approx 3.3\text{\AA}^{-1}$ ) by single crystal X-ray diffraction. Intensity maxima at the special points  $X(100)$ ,  $L(1/2, 1/2, 1/2)$  and  $\Gamma(000)$  of the Brillouin zone together with some "extra" scattering have been