Keywords: metal-organic framwork, fluorescence, hydrothermal synthesis

P11.15.108

Acta Cryst. (2008). A**64**, C540

Hydrothermal synthesis and charaterization of transition metal gallophosphates

Li-Hsun Huang, Sue-Lein Wang
National Tsing Hua University, Chemistry, Department of Chemistry, National Tsing Hua UniVersity Hsinchu 30013, Taiwan,, Hsinchu, Hsinchu, 30013, Taiwan, E-mail：d947424@oz.nthu.edu.tw

Open-framework metal phosphates have been widely studied for their applications in ion exchange, separation, and heterogeneous catalysis. It is well known that the inclusion of heteroatoms in open-framework phosphates can lead to the modification of the physical and chemical properties of the parent compounds. Many transition metals have recently been incorporated into gallium phosphate frameworks to give MGaPOs (M = Mn, Fe, Co, Zn, Ni, Ti, U,...) with both zeolitic and new structure types. In this research we explored transition metal gallophosphates which have been synthesized under hydrothermal conditions and structurally characterized by single crystal X-ray diffraction. The characterization of MnGaPO phase is described: the 3D open-framework consists of discrete bioctahedra of $Ga_2O_{10}(H_2O)$ and GaO_5 trigonal bipyramids connected via MnO_4 and PO4 tetrahedra to generate intersecting tunnels within which piperazinium cations reside.

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Keywords: hydrothermal, gallophosphates, transition metal

P11.15.109

Acta Cryst. (2008). A**64**, C540

Elastic layered zincophosphate intercalated with ice-like water cluster or superamolecular chains

Yu-Ting Huang, Yueh-Chun Liao, Sue-Lein Wang
National Tsing Hua University, Chemistry department, Department of Chemistry, National Tsing Hua University,101, Sec 2, Kuang Fu Rd., Hsinchu 300, Taiwan, R.O.C., Hsinchu, Taiwan, 30013, Taiwan, E-mail：juliatin10@gmail.com

Remarkable discrete water hexamers $(H_2O)_6$ have been observed in pseudo neutral layers of amine-anchored zincophosphate host.1 Molecular structures of (H_2O)62-6 have been studied in several reports in which the cyclic topology as well as hydrogen bonding were clearly illustrated. However, no previous work showed that water hexamers could exist in solid-state under ambient conditions. We report here a kind of discrete water hexamers confine in-between zincophosphate strata with a chair conformation. The confined hexamers could sustain heating up to 150℃ and then get off the host at higher temperatures. We observed that the host would become amorphous upon prolonged heating. Interestingly, after exposing to moisture, the dehydrated host could restore to its originally crystalline state with absorbed water molecules being self-assembled into cyclic water hexamers again. The amorphous -to-crystalline transition was able to proceed reversibly via a repeated dehydration / rehydration process. Without heating, we found that water hexamers could still be driven out of host by reacting with terephthalic acid (TA), which could self assemble into infinite supramolecular chains within the host layers. The interchange between $(H_2O)_6$ clusters and organic supramolecular entities involved very interesting and complicated guest-to-host and guest-to-guest hydrogen bonding interactions.

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Keywords: superamolecular chains, zincophosphate, supramolecular chains

P11.15.110

Acta Cryst. (2008). A**64**, C540-541

Gas adsorption process on silica mesoporous crystals obtained by *in-situ* SR powder diffraction

Yoshiki Kubota[1], Norihiro Muroyama[2], Arisa Yoshimura[1], Osamu Terasaki[2], Ryong Ryoo[3], Masaki Takata[4]
[1]Osaka Prefecture University, Physical Science, 1-1 Gakuen-cho, Naka-ku, Sakai, Osaka, 599-8531, Japan, [2]Structural Chemistry, Arrhenius Laboratory, Stockholm University, Sweden, [3]Center for Functional Nanomaterials and Department of Chemistry, Korea Advanced Institute of Science and Technology, Korea, [4]Structural Science Laboratory, Harima Institute SPring-8 Center, RIKEN, Japan, E-mail：kubotay@p.s.osakafu-u.ac.jp

Structure of silica mesoporous crystal MCM-48 and argon gas adsorption process were studied by synchrotron powder diffraction. The diffraction intensity data were analyzed by the combination of maximum entropy method (MEM) and electron crystallographic approach[1] to solve three-dimensional structure of mesoporous crystals. In the analysis, the total electron number in the unit cell of mesoporous crystal, which is used for one of the constraint conditions in MEM calculation, was obtained from the pore volume estimated from gas adsorption isotherm. The silica wall structure of MCM-48 was confirmed to follow a Gyroid surface, which is one of the periodic minimum surfaces. The resultant MEM charge density was less affected by termination effect and has clearer distribution than that obtained by the conventional Fourier synthesis. The *in-situ* synchrotron powder diffraction experiment of gas adsorption was carried out at BL02B2 in SPring-8[2]. The changes of diffraction pattern by the gas adsorption, corresponding to the gas adsorption isotherm, were clearly observed. The argon gas adsorption process was directly observed as the change of charge density distribution inside the mesopore of MCM-48. It was clearly observed that argon gradually adsorbs on the surface of pore walls at low gas pressures, and suddenly filled mesopore by a capillary condensation as argon gas pressure increased. Comparing gas adsorption behavior obtained from the observed data and the isotherm calculated by the quenched solid density functional theory (QSDFT) model is now in progress.

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