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New open-framework germanates with different types of inorganic molecular clusters

Kirsten E. Christensen,^a Lei Shi,^a Tiezhen Ren^a, Xiaodong Zou^a

^aStructural Chemistry, Stockholm University, SE-106 91 Stockholm, Sweden. E-mail: kec@struc.su.se

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A series of open-framework germanates synthesized at Stockholm University, denoted as SU-n, will be presented. They include pure germanates as SU-44, the recently published crystalline SU-M [1] and an aluminogermanate SU-46.

All these germanates were prepared by hydrothermal synthesis from a solution consisting of germanium dioxide and an organic amine as template, using water as solvent. In most cases hydrofluoric acid was added as a mineralizer. The resulting solution was transferred to a teflon-lined autoclave and heated to temperatures between 160-170°C for one or two weeks.

Single crystal X-ray diffraction data for both SU-M and SU-46 were collected at 100 K using graphite-monochromatized Mo K_α radiation. For needle-like crystals of SU-44, single crystal X-ray diffraction data were collected at 293 K using a synchrotron radiation at the beamline I711, Max-lab, Lund, Sweden. The structure solution and refinement were carried out using the interface WinGX [2] with the software SHELX-97. SU-46 is a three-dimensional structure with a new zeolite topology. It contains 2D intersecting 8-ring channels. Due to a tendency for twinning it has been difficult to determine the space group, apply absorption correction and find template positions.

SU-M is built from a single type of cluster Ge₁₀O₂₄(F,OH)₃ (denoted Ge₁₀). The clusters are connected in such a way that they lie on a gyroidal minimal surface with fully ordered crystalline walls [1].

When the temperature for the synthesis of SU-M is raised, SU-44 was then synthesized. Instead of the Ge₁₀-clusters in SU-M, SU-44 contains two different clusters, Ge₇O₁₇(F,OH)₂ (denoted Ge₇) and Ge₉O₂₂(F,OH)₄ (denoted Ge₉). Two different clusters in the same germanate structure has not previously been reported in the literature.

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[2] L. J. Farrugia, *J. Appl. Cryst.* 32 (1999), 837-838.

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Novel propeller-like phosphorus containing compounds

Alexey B. Dobrynin^a, Igor A. Litvinov^a, Aidar T. Gubaidullin^a, Yuliya Yu Borisova^b, Liliya M. Burnaeva^b, Vladimir F. Mironov^a

^aA.E. Arbusov Institute of Organic and Physical Chemistry, Russian Academy of Sciences, Kazan, Russia. ^bKazan State University, Kazan, Russia. E-mail: aldo@iopc.knc.ru.

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Molecular and crystal structures of novel propeller-like phosphorus containing compounds have been studied by X-Ray analysis. The molecule of 1-phenyl-3,3,10,10-tetrakis(triphenyl)-6,7-benzo-1-phospha-2,3,8,9-tetraoxa-tricyclo[3,3,0,2]-dec-6-ene is a first molecule of such type. The angles between the vanes of propeller are approximately 60 degrees. Benzene ring, bounded to phosphorus atom, is deviated from axis of propeller on 8 degrees. It is perpendicular to unsaturated cycle. In crystal of this compound there are no classical hydrogen bonds. But short contacts of F..F-type and O..F-type result in formation of cylindrical supramolecular ensembles with "fluorine channels" along *0b* axe. The crystal packing is stabilized by π - π -interactions between aromatic ring of tricyclic system. The packing coefficient and solvent accessible potential area in crystal were also analyzed.

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