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MS39-03 SU-66: Combining X-ray powder diffraction, electron diffraction and IR spectroscopy. A. Ken Inge,^a Tom Willhammar,^a Lynne B. McCusker^b and Xiaodong Zou^{*a} ^a*Berzelii Center EXSELENT on Porous Materials and Department of Materials and Environmental Chemistry, Stockholm University, SE-106 91 Stockholm, Sweden.* ^b*Laboratory of Crystallography, ETH Zurich, CH-8093 Zurich, Switzerland.*
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Crystalline open-framework materials such as zeolites and related oxides are of interest for their ability to size- and shape-selectively accommodate guest species in their pores and channels. Their properties relating to ion-exchange, sorption and catalysis allow for applications such as cracking of crude oil, water softeners, heating/cooling devices, catalysts for organic synthesis, and CO₂ capture. Open-framework germanates are of interest for their ability to form architectures with extra-large pores (≥14-rings), even if small organic guest species are used as structure directing agents. The majority of open-framework germanates are an assembly of large cluster building units, which in turn promotes the formation of structures with large pores. Thus, germanates are an excellent example of scale chemistry, i.e. frameworks with large pores can be constructed by using large clusters compared to a structure with the same topology but with smaller building units.¹ Many elegant germanate frameworks are composed of clusters built of multiple coordination polyhedra types. The Ge₁₀(O,OH)₂₇ (Ge₁₀) cluster for example is built of GeO₄ tetrahedra and GeO₆ octahedra, and is found in structures such as mesoporous SU-M with 30-ring channels.² Structure determination of germanates is often impeded by a number of factors. These include the difficulty in synthesizing sufficiently large crystals for single crystal X-ray diffraction, the tendency of having large unit cells that enhance peak overlap in X-ray powder diffraction (XRPD) patterns, and beam sensitivity under a transmission electron microscope.

SU-66 is a new open-framework germanate solved by a novel combination of techniques to overcome the mentioned issues. Prior to structure determination the cluster type was identified by comparing its infrared spectrum (IR) with spectra of germanates built of various cluster types. The region in the IR spectrum of SU-66 corresponding to Ge-O vibrations had strong resemblance of structures built of Ge₁₀ clusters. Once the cluster was identified, the unit cell and space group were determined by XRPD. However due to the poor figure of merit, the unit cell and reflection conditions were reconfirmed by selected area electron diffraction. Structure determination was performed by inserting two Ge₁₀ clusters, the unit cell parameters, space group, and the XRPD pattern into a simulated annealing parallel tempering algorithm in the program FOX.³ SU-66 has extra-large 26-ring channels and is one of the more complicated framework structures solved by XRPD. Additional studies show that other complicated framework structures can be solved in a similar way, even with rather poor quality XRPD data.

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