MS07.O03

Why was the structure of the niobium silicate AM-11 so difficult to solve?

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The synthesis of the polycrystalline niobium silicate catalyst AM-11 was first reported in 1998 [1], but its structure proved to be elusive. In 2007 we received two samples from the Aveiro group. At the time, we were looking for a material suitable for the application of the texture method of structure solution, and AM-11 seemed to be ideal for this purpose. One of the samples had needle and the other platelet morphology, and textured samples could be prepared in both cases. The conventional powder diffraction pattern could be indexed on a hexagonal, an orthorhombic or a monoclinic unit cell, so this was the first issue to be resolved. The texture measurements quickly revealed that the crystal system was orthorhombic, but the structure resisted solution. We then tried applying the precession electron diffraction technique in combination with high-resolution powder diffraction data, but beyond confirming the orthorhombic symmetry, these data did not help us to solve the structure. Another attempt was made with a new sample and an improved texture setup, but to no avail. Rotation electron diffraction data and high-resolution transmission electron microscopy images showed that some disorder was present and helped to define the space group, but the structure remained a mystery. The powder charge-flipping routine in Superflip [2], yielded tantalizingly clear electron density maps, but they could not be interpreted sensibly. The unit cell parameters were seen to be related to those of the titanium silicate zorite [3] (one axis doubled in AM-11), so the problem was taken up once again last year. By starting with a simplified zorite framework structure with Nb in place of Ti, and performing what amounts to manual Fourier recycling, the surprisingly simple structure (1Nb, 3 Si, 9 O), which is significantly different from zorite, finally revealed itself. There is some stacking disorder, but the structure is otherwise innocuous. What made it so difficult to solve?

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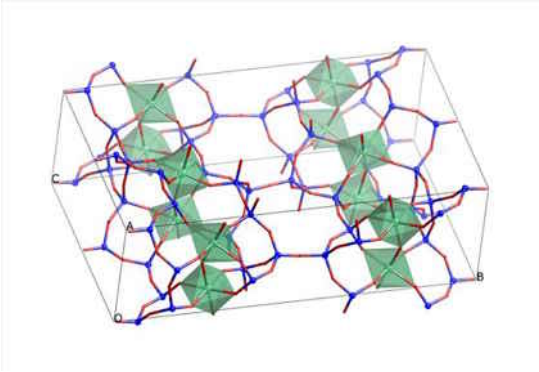

Keywords: powder diffraction, electron diffraction, structure solution