

TABLE 1

| Ligand | Halogen | Crystal Data |
|--|---------|---|
| 1 (Me ₂ CHCH ₂) ₂ SO | Cl | a=10.10, b=10.43, c=13.29 Å; β=106.36°; Z=2; P=2 ₁ /c |
| 2 (MePhN) ₂ CO | Cl | a=14.01, b=19.33, c=14.44 Å; β=119.28°; Z=4; P2 ₁ /n |
| 3* (MePhN) ₂ CO | Cl | a=15.87, b=13.21, c=15.76 Å; Z=4; Pna2 ₁ |
| 4 (MePhN) ₂ CO | Br | a=14.39, b=19.85, c=24.71 Å; Z=8; Fddd |
| 5** (C ₄ H ₈ N) ₃ PO | Cl | a=17.03, b=11.71, c=18.21 Å; β=109.2°; Z=4; C2/c |
| 6 Ph ₃ AsO | Br | a=10.24, b=15.79, c=12.19 Å; β=100.94°; Z=2; p2 ₁ |

*polymorph of 2

**pyrrolidyl

08.2-28 THE CRYSTAL STRUCTURE OF α-EUCRYPTITE, LiAlSiO₄. By K.-F. Hesse, Mineralogisches Institut, Universität Kiel, D-2300 Kiel, Germany.

According to Winkler (Acta Cryst. (1953) 6, 99) the low temperature polymorph of LiAlSiO₄,

α-eucryptite, is isostructural with phenakite, Be₂SiO₄, and willemite, Zn₂SiO₄, and has an ordered Al,Si distribution. However, no structure determination has been published.

Natural and synthetic crystals of α-eucryptite have now been used for structure refinement.

Crystallographic data: trigonal R $\bar{3}$, a_{hex} = 13.473(3) Å, c_{hex} = 9.001(2) Å, Z = 18. With

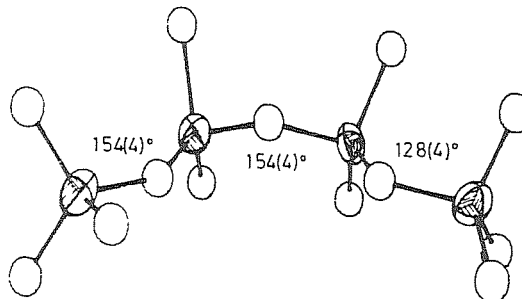
1895 (1467) non-equivalent reflections the refinement converged at residuals of R = 0.080 (0.054) and R_w = 0.040 (0.042) for the natural

(synthetic) crystal. The results of these structure determinations are: the structures consist of [(Al,Si)O₄] tetrahedra with (Al,Si)-O mean distance 1.689 Å (1.688 Å) and [Li-O₄]

tetrahedra with Li-O mean distance 1.981 Å (1.984 Å). The (Al,Si)-O distances suggest statistical Al,Si distribution or microtwinning in both natural and synthetic α-eucryptite. The Giebe-Scheibe test, high-resolution electron microscopy and diffraction strongly favour statistical Al,Si distribution.

08.2-29 Pb₈[O₂(SO₄)(Si₄O₁₃)], A NEW TETRASILICATE. By R. Fröhlich, Institut für Kristallographie, Universität, D-7500 Karlsruhe, Germany

In the system PbO-PbSO₄-PbSiO₃ several ternary phases exist; the compound 8PbO·SO₃·4SiO₂ melts congruently at 785°C and shows no phase transitions (Billhardt, thesis, Karlsruhe, 1968). Single crystals can be prepared by slow cooling from 800°C to 600°C and subsequent annealing at 600°C. Crystal data: monoclinic space group P2₁/n; a=914.0(3), b=1955.4(6), c=1131.3(4) pm, β=89.68(3)°; Z=4. The structure was solved by Patterson and Fourier methods. The present value of R=0.115 for 2456 observed reflections is poor due to a preliminary absorption correction for the irregular shape of the crystal investigated. Further refinement is in progress. The basic building blocks of the structure are two tetrahedral [Pb₄O]⁶⁺-units, a sulphate group, and the tetrasilicate anion shown below.



[Si₄O₁₃]¹⁰⁻-ion; Si drawn as thermal ellipsoids.

08.2-30 THE CRYSTAL STRUCTURE OF Zn₂Ti₁₈S₃₂

By I. Kawada, M. Onoda and M. Saeki, National Institute for Research in Inorganic Materials, Sakura-mura, Ibaraki-ken 305, Japan.

Expecting tetrahedral coordination of metal-sulfur in M-Ti-S system (M=metal), we have synthesized a new phase of Zn₂Ti₁₈S₃₂. (M. Saeki and M. Onoda, Chem. Lett. 1329, 1982).

Obtained specimen was in powder form of dark gray color. 69 independent powder diffraction data were collected by an X-ray powder diffractometer using Cu- and Mo-target. Crystal system is cubic; a=9.843 Å. Taking no account of 2 very diffuse reflections, space group is Fd3m - O_h.

Crystal structure was solved by crystal chemical considerations. The structure consists fundamentally of cubic closest packing of sulfur atoms. 16 Ti atoms occupy octahedral sites (Ti(1)) and 2 Ti atoms occupy statistically another 16 octahedral sites (Ti(2)). 2 Zn atoms occupy statistically 8 tetrahedral sites which are surrounded only by Ti(2) and S. (Table 1.). Considering very short distance between Zn and Ti(2) (2.13 Å), it is probable that Zn and Ti(2) do not simultaneously coexist at the nearest positions. If one takes account of the existence of diffuse reflections (e.g. 200 etc.), more detailed feature of the structure will be clarified.

| Table 1. | m | x | y | z | B | |
|----------|-------|-----|--------|--------|--------|------|
| Zn | 8(a) | 1/4 | 1/8 | 1/8 | 2.39 | |
| Ti(1) | 16(d) | 1 | 1/2 | 1/2 | 1.35 | |
| Ti(2) | 16(c) | 1/8 | 0 | 0 | 0.02 | |
| S | 32(e) | 1 | 0.2504 | 0.2504 | 0.2504 | 0.92 |

number of reflections: 67, R=0.0695.