

08.1-20 A COMPUTER DETERMINATION OF $2V$ OF ANTHRACENE FROM EXTINCTION DATA. By Maureen M. Julian, Dept. of Chemistry, Hollins College, VA 24020, U.S.A.

In biaxial crystals, measurement of the angle between the two optic axes, $2V$, is constant for a given crystal, at a given temperature, and for a given wavelength. A study of the optical properties of anthracene was done because of conflicting results in the literature and because recently developed techniques (Bloss, 1981) introduce computer and statistical methods into the practice of optical crystallography permitting greater accuracy. The work was complicated by the fact that the anthracene crystal rapidly dissolves in immersion media. Data were completed on four separate crystals, three at 540 nm and one at 900 nm. The data were analyzed by the Bloss-Riess-Roher program "excalibr" using Joel's equation to find the optic axes and refinement was done by least squares to calculate an accurate $2V$.

Bloss, F.D. "The Spindle Stage: Principles and Practice". CUP, 1981.

actions. S-S bond lengths are 2.067(2) Å. The geometry of the B_8S_{16} molecule (transannular S..S distances ca. 4.6 Å) allows the preparation of transition metal complexes with tetragonal planar coordination by the tetradentate B_8S_{16} ligand.

Two different isotypic compounds BS_2 and BSe_2 with the same 1:2 boron-to-chalcogen ratio as in B_8S_{16} are obtained, if the conditions of preparation are varied slightly. They are monoclinic, space group $P2_1/c$, with $a = 6.800(2)$, $b = 10.545(3)$, $c = 7.828(2)$ Å, $\beta = 117.27(3)^\circ$, $Z = 8$ BS_2 , $d_x = 1.995$ g.cm⁻³ for BS_2 , and $a = 7.205(2)$, $b = 11.202(3)$, $c = 8.123(2)$ Å, $\beta = 117.62(3)^\circ$, $d_x = 3.858$ g.cm⁻³ for BSe_2 . The crystal structures ($R = 6.2\%$ and 5.2%) of both show endless chains along the b axis, consisting of five-membered B_2S_3 (B_2Se_3) rings as in the porphyrin-like isomer, the rings being linked through B-S-B (B-Se-B) bridges. Rather short interannular S..S and Se..Se contacts of 3.167(3) and 3.295(3) Å appear to be essential for the stabilization of the strictly planar chains as a whole. As in B_8S_{16} , the boron-chalcogen bond lengths are quite uniform with B-S and B-Se values of 1.783(9) ... 1.824(8) Å (mean 1.803) and of 1.90(3) ... 1.98(3) Å (mean 1.93) respectively. The structural results indicate that, in contrast to some ternary phases such as $Pb_4B_4S_{10}$ and $Ag_6B_{10}S_{18}$ and in accordance to investigations on BS_2 , BSe_2 and B_2S_3 glasses, trigonal planar coordination of boron is prevalent in binary sulfides and selenides.

08.2-01 STRUCTURES, CRYSTAL CHEMISTRY AND PROPERTIES OF NOVEL BORON SULFIDES AND SELENIDES: By B. Krebs and H.-U. Hürter, Anorganisch-Chemisches Institut der Universität Münster, Gievenbecker Weg 9, D-4400 Münster, West Germany

Only very limited knowledge is available on the compositions and structural properties of binary boron sulfides and selenides. B_2S_3 , which was prepared as twinned single crystals from thermal decomposition of $Ag_6B_{10}S_{18}$, was shown to form a layer structure containing planar B_2S_2 and B_3S_3 rings (Krebs, Diercks, Acta Cryst. A31, S66 (1975); Diercks, Krebs, Angew. Chem. Int. Ed. Engl. 16, 313 (1977)).

We could now prepare three novel compounds in this class and determine their crystal structures from single crystal diffractometer data. A molecular boron sulfide B_8S_{16} is obtained from stoichiometric B_2S_3 - S_8 mixtures in graphitized reaction tubes at 300/100°C or from thiolysis of halogen-substituted trithiadiborolanes $B_2S_3Hal_2$. It crystallizes in space group $P2_1/c$ with $a = 12.091(2)$, $b = 4.063(1)$, $c = 21.870(4)$ Å, $\beta = 107.64(3)^\circ$, $Z = 2$, $d_x = 1.944$, $d_m = 1.92(2)$ g.cm⁻³ (at 20°C) and contains centrosymmetric exactly planar B_8S_{16} molecules with a porphyrin-like structure of four five-membered 1.2.4.3.5-trithiadiborolane rings linked through S bridges ($R = 5.4\%$ for 20°C data, 4.7% for -130°C data). The average B-S bond length of 1.807 Å (individual values 1.795(6) ... 1.820(6) Å) corresponds to the 1.808 Å value in B_2S_3 and indicates, in accordance to the planarity of the molecule and to CNDO calculations, strong p π -p π inter-

08.2-02 THE CRYSTAL AND MOLECULAR STRUCTURE OF TETRABORATE OF CALCIUM AND POTASSIUM, DODECAHYDRATE. By X. Solans and M. Font-Altaba.

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$(B_4O_5(OH)_4)_2 Ca K_2 \cdot 12H_2O$. Orthorhombic, $P2_12_12_1$, $a = 16.597(3)$, $b = 12.469(3)$, $c = 11.569(2)$ Å.

Intensity data were recorded on a Philips PW-1100 four circle diffractometer using MoK radiation, monochromatized by reflexion from a graphite crystal. 3175 reflections in the range $2\theta \leq 30^\circ$ were considered as observed applying the condition $I \geq 2.5 \sigma(I)$.

The structure was solved with the MULTAN 80 system of computer programs and refined by full matrix least-squares method with the SHELX 76 program.

The Ca^{2+} and K^+ ions have a distorted eight-coordination. No great differences are observed in the $B_4O_5(OH)_4$ ions.