

Abstracts of Forthcoming Articles

Solid State Chemistry of Organic Polyvalent Iodine Compounds. V. The Crystal Structures of 3-oxo-3H-2, 1-benzoxiodol-1-yl o-bromobenzoate (Two Polymorphs) and 3-oxo-3H-2, 1-benzoxiodol-1-yl o-chlorobenzoate. J. ZANOS GOUGOUTAS AND D. G. NAAE. Department of Chemistry, University of Minnesota, Minneapolis, Minnesota 55455. Single crystal transformations of 2-iodo-2'-bromo-, and 2-iodo-2'-chlorodibenzoyl peroxides (*Ila* and *Ilb*) yield topotactically aligned crystal structures of their respective isomers: 3-oxo-3H-2, 1-benzoxiodol-1-yl *o*-bromobenzoate (*Ia*) and its *o*-chlorobenzoate analog (*Ib*). Two of the polymorphic forms of *Ia* ($C_{14}H_8IBrO_4$) which are formed in crystalline *Ila* can also be obtained by crystallizing *Ia* from various solvents: (*Ia*- β), $a = 7.474$, $b = 13.994$, $c = 13.152$ Å, $\beta = 102.4^\circ$, $Z = 4$, $P2_1/a$, and a disordered acicular form (*Ia*- α) which appears to be orthorhombic, $a = 3.99$, $b = 26.553$, $c = 26.008$, $Z = 8$ ($Pbc2_1$?). The only form of *Ib* ($C_{14}H_8IClO_4$) obtained by crystallizations from solvents is also formed in crystalline *Ib*. The crystal structures of the isostructural *Ia*- β and *Ib* have been determined but only the latter has been refined. ($R = 0.059$ for 2085 "observed" intensities.) The derivative crystal structure of *Ia*- α has been determined only in (100) projection ($R = 0.08$ for 156 observed intensities). The three structures contain similar chains of bimolecular units or "dimers." The "dimers" are analogous to those found in crystal structures of the benzoate, *m*-chlorobenzoate, *o*-iodobenzoate, and *o*-fluorobenzoate analogs of I. Novel polymorphs (X_a and X_b) of *Ia* and *Ib* are formed during the solid state peroxide isomerizations. The isostructural X_a and X_b consist of chains of "dimers" which are similar to those in *Ia* and *Ib*.

Solid State Chemistry of Organic Polyvalent Iodine Compounds. VI. The Crystal Structure and Topotactic Hydrolysis of 3-oxo-3H-2, 1-benzoxiodol-1-yl o-fluorobenzoate. J. ZANOS GOUGOUTAS, KUO H. CHANG, AND M. C. ETTER. Department of Chemistry, University of Minnesota, Minneapolis, Minnesota 55455. The compound 3-oxo-3H-2, 1-benzoxiodol-1-yl *o*-fluorobenzoate (*IIIc*) is formed as a topotactically aligned crystal structure when single crystals of its dibenzoyl peroxide isomer thermally decompose at room temperature. Independently it has been found that single crystals of *IIIc* grown from solvents are hydrolyzed to *o*-iodosobenzoic acid, *II*, and *o*-fluorobenzoic acid after ~4 weeks in a moist atmosphere at ~45°C. *II* is formed topotactically in a conservatively twinned arrangement in which the (100) planes of the equally present twin members are parallel to (001) of *IIIc*. In addition, the 4 Å *b*-axes of the twin members align with the 4 Å *a*-axis of *IIIc*. Other examples of topotactic hydrolyses resulting in twinned arrangements of *II* are cited in reference to the novel possibility that the favored mode of alignment (the specific topotaxy between the reactant and one twin member) is one which, when augmented by conservative twinning, results in a favorable interphase between the conservatively twinned members. The transformation *IIIc* \rightarrow *II* and more than 20 other corroborating examples of topotaxy in our studies lead us to expect the alignment of 4 Å axes whenever they are present in both the reactant and product of topotactic transformations involving comparable aromatic molecules. The crystal structure of *IIIc* ($C_{14}H_8IFO_4$), $a = 4.08$, $b = 12.00$, $c = 26.25$ Å, $\beta = 90.5^\circ$, $Z = 4$, $P2_1/c$ (refined to $R = 0.10$ for 453 photographically recorded intensities) consists of chains of *transoid* "dimers" similar to those previously observed in structures of closely related derivatives.

Ba₁₅Fe₇S₂₅ and Ba₃FeS₅: Crystal Structures, Mössbauer, Magnetic and Electrical Behavior. J. T. LEMLEY, J. M. JENKS, J. T. HOGGINS, Z. ELIEZER, AND H. STEINFINK. Department of Chemical Engineering, The University of Texas, Austin, Texas 78712. Ba₁₅Fe₇S₂₅ is prepared by reacting stoichiometric amounts of BaS, Fe, and S in evacuated quartz ampoules while Ba₃FeS₅ is obtained in a tetrahedral press at 50 kbar and 1000°C. The crystal structure of Ba₁₅Fe₇S₂₅ has been determined using 4053 independent, nonzero X-ray reflections measured by a counter technique. The compound crystallizes in the orthorhombic system, *Pnma*, $a = 41.91(1)$ Å, $b = 9.572(3)$ Å, $c = 12.654(3)$ Å, $\rho_{calc} = 4.14$ g/cc, $z = 4$. In both compounds, Fe is tetrahedrally coordinated to S, and Ba is in either