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Crystal modifications of bis-salicylaldehydatocopper(II).

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Crystals of bis-salicylaldehydatocopper have been described by von Stackelberg (1947) as probably orthorhombic, with $a = 11\cdot 74$, $b = 3\cdot 99$, $c = 12\cdot 45$ Å. It was considered possible that the crystals were in fact monoclinic with $\beta = 90 \pm 1°$, and the apparent orthorhombic diffraction symmetry was due to twinning. In the course of an investigation of the crystal structure of this compound we have obtained the following crystal modifications.

A. Green prismatic crystals, displaying [101], [10$\bar{1}$] and [111] equally developed, obtained from a cold chloroform solution. Monoclinic $a = 8\cdot 72$, $b = 6\cdot 20$, $c = 11\cdot 26$ Å; $\beta = 104\cdot 8°$. $D_x = 1\cdot 725$, $D_m = 1\cdot 71$ g.cm^{-3}, $Z = 2$. Systematic absences: $h0l$ when $h + l$ is odd, $0k0$ when k is odd. Space group $P2_1/n$.

B. Thin, green, poorly formed flakes displaying [100], obtained by cooling a hot saturated solution in ethanol. Monoclinic, $a = 11\cdot 75$, $b = 4\cdot 00$, $c = 12\cdot 42$ Å; $\beta = 90\cdot 3°$. $D_x = 1\cdot 73$, $D_m = 1\cdot 71$ g.cm^{-3} (Stackelberg, 1947), $Z = 2$. Systematic absences: $h0l$ when l is odd, $0k0$ when k is odd. Space group $P2_1/c$.

C. Thin, green, rhombic plates obtained from an equivolume chloroform–methanol solution. Apparently orthorhombic, $a = 12\cdot 35$, $b = 6\cdot 20$, $c = 23\cdot 04$ Å. Systematic absences: hkl when neither $h \pm l$ equals $3n$, $h0l$ when h is odd, $0k0$ when k is odd.

It is apparent that *B* is the same modification as that described by von Stackelberg, and that these crystals are in fact monoclinic. The unusual systematic absences shown by *C*, together with the fact that the *b* axial length is identical with that of *A*, suggests that *C* may also be a twinned modification. Consideration of the lattice of *A* showed that an alternative unit cell could be chosen with $a' = 12\cdot 35$, $c = 23\cdot 04$ Å, $\beta = 90\cdot 0°$, this cell being triply primitive with additional lattice points at $\frac{1}{3}, 0, \frac{1}{3}$ and $\frac{2}{3}, 0, \frac{2}{3}$. When indexed on this cell reflexions are absent except when $h + l = 3n$. If twinning were to occur by rotation about a' or c' then, assuming the individuals to be present to equal extent, diffraction symmetry *mmm* would result, with the above observed systematic absences. Superposition of two appropriately oriented $h0l$ Weissenberg photographs of *A* does in fact reproduce that obtained from *C*. It appears that both of the genuine structural modifications *A* and *B* can simulate higher symmetry by such a twinning process.

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The unit cell and space group of $SF_3.BF_4$.

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Single-crystal X-ray data have been obtained to assist in the interpretation of electron spin resonance (ESR) spectra of radiation damaged crystals of $SF_3.BF_4$. This compound was prepared by the method of Bartlett & Robinson (1961) in a quartz tube and needle-like crystals were formed in a temperature gradient. The crystals were orthorhombic, diffraction symbol *mmmPn.a*, with possible space groups $Pnma$ (D_{2h}^{16}) and $Pn2_1a$ (C_{2v}^{9}). With a precession camera and Mo K radiation the zones $h0l$, $h1l$, $h2l$, $hk0$, $hk1$, $hk2$ and $0kl$ were recorded; two crystals of good quality were examined in detail. The axial lengths were

$$a = 9\cdot 63 \pm 0\cdot 03,\ b = 5\cdot 77 \pm 0\cdot 02,$$
$$c = 8\cdot 99 \pm 0\cdot 03\text{Å};\ U = 500\ \text{Å}^3,$$
$$Z = 4,\ D_x = 2\cdot 34\ \text{g.cm}^{-3},\ \text{F.W. } 175\cdot 9.$$

The crystals were pyramidally terminated needles, elongated along *b*, with the *c* axis approximately perpendicular to the glass surface; the forms {102} and {101} were well developed. There is strong similarity between the observed crystallographic data for $SF_3^+.BF_4^-$ and the data for NH_4IO_3 (MacGillavry, 1943). The assumption that the structures are similar is consistent with the observed ESR spectra (Morton, 1963).

No further work is planned but we understand that a structure determination will shortly be undertaken at the University of British Columbia.

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