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## Crystallographic data for salicylaldoxime, $C_7H_7O_2N$.*

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Salicylaldoxime is an organic reagent which forms complexes with a variety of metal ions, and which can, in some cases, be made specific by control of pH conditions of precipitation. This laboratory has been investigating the structures of chelating agents and of chelate complexes of analytical importance for the past few years. Recently the structure of nickel salicylaldoxime was reported by workers in this group (Merritt, Guare & Lessor, 1956). A study of the structure of salicylaldoxime has now been undertaken, and this paper constitutes a report on the preliminary work.

Using hexagonal crystals of salicylaldoxime, obtained from an alcohol solution of the compound, a preliminary X-ray survey has determined the unit cell and space group. From rotation, Weissenberg and precession photographs taken about the $a$ and the $c$ axes, using nickel-filtered Cu $K\alpha$ radiation, the orthorhombic unit-cell dimensions were found to be

$$a = 12{\cdot}69,\ b = 13{\cdot}51,\ c = 7{\cdot}98\ \text{Å}\,.$$

The only systematic absences of reflections occurred for ($h00$) when $h$ is odd, ($0k0$) when $k$ is odd, and ($00l$) when $l$ is odd. Accordingly, the space group is determined as $P2_12_12_1$–$D_2^4$.

There are 8 molecules per unit cell. Density: calculated 1·33 g.cm.$^{-3}$; observed (flotation) 1·34 g.cm.$^{-3}$.

The refractive indices were measured by means of standard refraction liquids:

$$n_\alpha = 1{\cdot}480,\ n_\beta = 1{\cdot}76,\ 1{\cdot}79 < n_\gamma < 1{\cdot}80\,.$$

The powder pattern of salicylaldoxime was recorded with nickel-filtered Cu $K\alpha$ radiation in a Debye–Scherrer camera of 57·3 mm. radius. The intensities were estimated visually by comparison with standard intensity strips prepared by timed exposures of one reflection from a single crystal. The data are summarized in Table 1.

Table 1. *Powder diffraction data*

| $d$ (Å) | $I/I_0$ | $d$ (Å) | $I/I_0$ |
|---|---|---|---|
| 6·76 | 0·42 | 3·08 | 0·21 |
| 6·32 | 0·15 | 2·96 | 0·07 |
| 5·99 | 0·13 | 2·89 | 0·15 |
| 5·68 | 0·28 | 2·78 | 0·10 |
| 5·10 | 0·23 | 2·64 | 0·09 |
| 4·58 | 0·36 | 2·36 | 0·12 |
| 3·97 | 0·21 | 2·20 | 0·09 |
| 3·65 | 1·00 | 1·98 | 0·08 |
| 3·38 | 0·51 | 1·79 | 0·08 |
| 3·26 | 0·67 | 1·67 | 0·06 |

Further work is in progress with the hope of completing a structure determination of this compound.

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Merritt, L. L., Jr., Guare, C. & Lessor, A. E., Jr. (1956). *Acta Cryst.* In the Press.

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* Contribution No. 687, Indiana University.

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## Partial structure of bis-benzonitrile palladium chloride.

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In connexion with an investigation of some complexes of palladium chloride with ethylenic-type compounds (Holden & Baenziger, 1955) a partial structure of bis-benzonitrile palladium chloride (an intermediate in the preparation of the ethylenic complexes) was determined.

Long blade-shaped crystals of bis-benzonitrile palladium chloride showing no external symmetry were formed on a 'cold-finger' placed in a warm solution of palladium chloride in benzonitrile. Weissenberg and precession diagrams were taken with the X-ray beam normal to the needle axis of the crystal. The unit cell was found to be triclinic with the reduced primitive cell dimensions of

$$a = 5{\cdot}79,\ b = 8{\cdot}45,\ c = 8{\cdot}71\ \text{Å}\,,$$
$$\alpha = 117{\cdot}4,\ \beta = 92{\cdot}9,\ \gamma = 95{\cdot}0^\circ\,.$$

The needle axis of the crystal is the short body diagonal

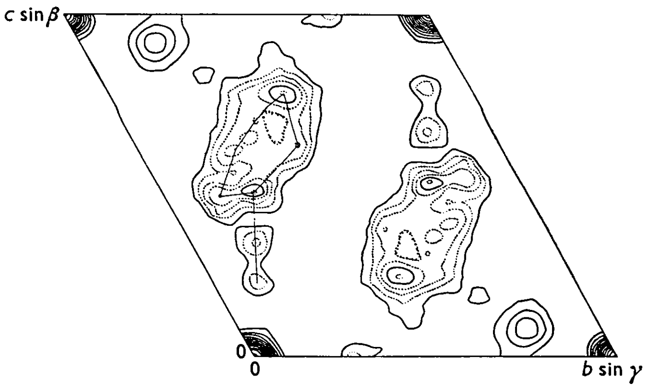

Fig. 1. Electron density projected along [100].

of the cell. All the crystals examined were twinned on the ($\bar{1}01$) plane. From the volume of the cell, 375 Å$^3$, and the formula weight of 383·7, the only reasonable calculated density is 1·70 g.cm.$^{-3}$ with one molecule per unit cell.

Since there is only one molecule per cell, the palladium atom can be placed at the origin. The signs of all but the very weakest structure factors may be assumed to be positive. Assuming space group $P\bar{1}$ and positive signs for the structure factors, the electron density was projected along [100] and [111]. The clearest resolution of atoms was obtained on the [100] projection shown in Fig. 1. The [111] projection served only to indicate the third parameter for the chlorine atoms. The chlorine parameters were found to be $x = 0{\cdot}124$, $y = 0{\cdot}792$, $z = 0{\cdot}084$. The agreement between calculated and observed $F$ values at this stage was $R = \Sigma||F_o|-|F_c|| \div \Sigma|F_o| = 0{\cdot}24$ for the zero-layer Weissenberg data and 0·26 for the zero-layer precession-camera data.

The results may be interpreted as the formation of a planar square complex with the benzonitrile groups attached end-on through the nitrogen atoms at positions *trans* to each other. The approximate Pd–Cl bond distance is 2·35 Å. Further refinement of this structure, which would require full three-dimensional intensity data and three-dimensional electron density refinement techniques, is not contemplated.

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## The crystal structure of *α*-*trans*-cinnamic acid.

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In the course of a comprehensive study of solid-state reactions we have determined the crystal structure of the stable ($\alpha$) modification of *trans*-cinnamic acid.

According to Groth (1917), the crystals are monoclinic prismatic showing {010};

$$a:b:c = 0{\cdot}8627:1:0{\cdot}3138,\ \beta = 96°\ 49\tfrac{1}{2}';$$
$$d = 1{\cdot}245-1{\cdot}247 \text{ g.cm.}^{-3}.$$

For crystals at room temperature we find:

$$a = 7{\cdot}79\pm0{\cdot}02,\ b = 18{\cdot}07\pm0{\cdot}05,\ c = 5{\cdot}67\pm0{\cdot}02 \text{ Å},$$
$$\beta = 97°\pm1°;$$

$$\text{space group } P2_1/n,\ Z = 4\ ,$$

giving

$$a:b:c = 0{\cdot}431:1:0{\cdot}314\ ,$$

in good agreement with the previous values.

The structure was determined from a three-dimensional Patterson synthesis. Coordinates were refined from zonal data; $R(hk0) = 17{\cdot}6\%$ and $R(0kl) = 18{\cdot}0\%$ at the present stage of refinement. Concurrently with the room-temperature refinement, the $x$ and $y$ coordinates were determined from ($hk0$) intensities (Cu and Mo) measured at the temperature of boiling nitrogen; for these data $R(hk0) = 13{\cdot}9\%$. The values of the relevant lattice constants at this temperature are $a \sin\beta = 7{\cdot}64$ and $b = 17{\cdot}51$ Å. Electron-density projections down [001] (Fig. 1) illustrate the increased resolution obtainable with low-temperature data; for carbon, the average peak density $\varrho(0)$ is increased from 7 to 10 e.Å$^{-2}$, and for oxygen, from 9 to 14 e.Å$^{-2}$.

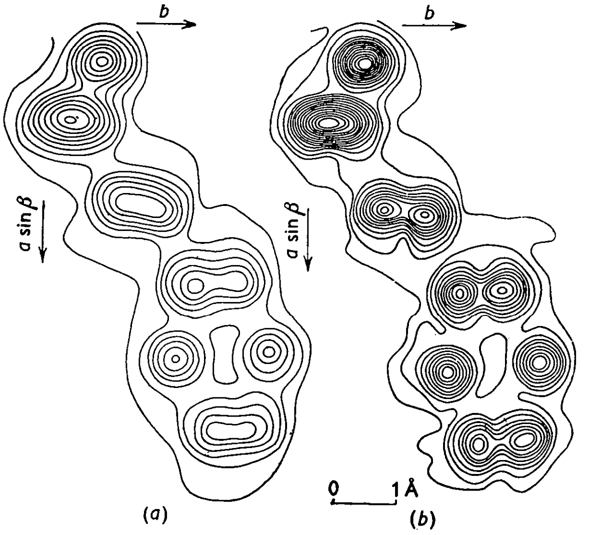

Fig. 1. The $c$-axis projection of electron density ($a$) at room temperature, ($b$) at 90° K. Contour interval 1 e.Å$^{-2}$ beginning at 1 e.Å$^{-2}$.

The observed structure confirms Lonsdale's predictions (1939) based on magnetic measurements. The structural units are dimers formed by the hydrogen bonding of carboxyl groups across centres of symmetry. Full details of the structure analysis and the relation of molecular packing to chemical behaviour will be discussed in a later paper.

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