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The structure of 5-chlorosalicylaldoxime.

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In connection with an investigation of the structure of palladium(II) salicylaldoxime it was desirable to ascertain the distortion of the salicylaldoxime molecule upon formation of the palladium complex. 5-Chlorosalicylaldoxime was prepared because the presence of the heavy atom would facilitate the determination without appreciably affecting the structure of the salicylaldoxime molecule.

Suitable crystals of 5-chlorosalicylaldoxime were obtained by slow cooling of a saturated aqueous solution, and by evaporation of a chloroform solution. The unit cell is monoclinic with:

$$a = 13{\cdot}69,\ b = 3{\cdot}90,\ c = 14{\cdot}35\ \text{Å};\ \beta = 100{\cdot}0°.$$

Characteristic extinctions were noted which are those required by the space group $P2_1/a$, and the unit cell contains four molecules. Density: calculated, 1·510 g.cm.$^{-3}$; measured (by flotation), 1·510 g.cm.$^{-3}$.

Intensity data for the ($h0l$) zone were collected from Weissenberg photographs. The approximate structure was determined by a Patterson function projected along [010], which showed directly the outline of the molecule. Several electron-density projections were calculated, and back-shift corrections were made. At this stage of refinement, the agreement index, R, between calculated and observed structure factors was 16% (233 ($h0l$) reflections). Further refinement of the structure by least squares is in progress, and intensity data for the ($0kl$) zone are being collected.

It was evident by the shortening of the benzene bond lengths in the projection that the molecules were tilted out of the (010) plane. Using the accepted value of 1·39 Å, the angle of tilt was calculated to be 27° 46′ about an axis lying in the (010) plane and making an angle of 69° 15′ with [100]. The bond lengths, calculated using the above tilt, were in good agreement with the values reported by Jerslev (1950) for *syn-p*-chlorobenzaldoxime, and are given in Table 1.

Table 1. *Comparison of observed bond lengths of* syn-p-*chlorobenzaldoxime and 5-chlorosalicylaldoxime*

Bond	*syn-p*-Chlorobenzaldoxime	5-Chlorosalicylaldoxime
C–C in benzene ring	1·35–1·38 Å	1·38–1·40 Å
C–Cl	1·78	1·82
C(benzene)–CH	1·45	1·43
CH–N	1·31	1·25
N–O	1·36	1·40

The short CH–N and long N–O distances may be due to the N–O group being tilted slightly out of the plane of the Cl and C atoms. Full details of the structure will be reported at a later date.

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Reference

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Orientation relationships involved in the formation of α-W_2C on tungsten.

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In the study reported here, tungsten wires with a diameter of 0·025 cm. which had been previously coated with a thin film of graphite were annealed *in vacuo* at elevated temperatures and investigated using X-ray diffraction techniques. In Fig. 1(*a*) is shown the X-ray pattern from a wire sample which had been annealed at 1750° C. for 10 min. The pattern was obtained using a flat-plate front-reflection camera with a specimen–film distance of 5 cm. Molybdenum radiation was employed in conjunction with a zirconium filter. All the diffraction rings in Fig. 1(*a*) match closely with those of tungsten. From the intensity maxima on the diffraction rings, it can be deduced that the wire has a strong ⟨110⟩ texture. It has also been shown that the crystals on the surface of the wire are oriented with a {100} plane tangential to the surface and that the ⟨110⟩ texture is maintained at least up to 2050° C. (Swalin & Geisler, 1957). The size and shape of the crystals has been determined microscopically and were found to have the shape of long fibers oriented parallel to the wire axis with a diameter of about 10^{-4} cm.

In Fig. 1(*b*) is shown an X-ray pattern for a wire sample which had been annealed at 1850° C. for 10 min. The other conditions were the same as for Fig. 1(*a*). Interpretation of this pattern shows that the diffraction rings are representative of α-W_2C with a few weak tungsten lines included. A W_2C film of a few microns thickness

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