A five-parameter least-squares refinement was made. The variables were the scale factor, two isotropic temperature factors (one for uranium and one for the light atoms), the x parameter of the fourfold carbonate oxygens, and the y parameter of the uranyl oxygens (see Christ *et al.*, 1955). The McWeeny (1951) form factors were used for carbon and oxygen. For uranium, the form factor given in the *Internationale Tabellen* was used after subtracting 5·6 electrons to correct for anomalous dispersion. The observations were given a weight proportional to $1/\sigma_I^{\frac{1}{2}}$, where σ_I is the standard deviation of the intensity measurements. The 020 reflection was given zero weight because of obvious extinction effects. The calculations were made on an IBM 701.

The results of the least-squares refinement are given below:

$$x = 0{\cdot}011 \pm 0{\cdot}018,\ y = 0{\cdot}932 \pm 0{\cdot}010;$$
$$B_U = 0{\cdot}51 \pm 0{\cdot}20,\ B_{O,C} = 1{\cdot}65 \pm 0{\cdot}86\ \text{Å}^2\,.$$

For this projection $R = 7{\cdot}3\%$ with 020 omitted.

The significant result is the value of the y coordinate of the uranyl oxygen. Assuming that the uranyl ion is linear, the U–O distance is $1{\cdot}67 \pm 0{\cdot}09$ Å. This distance is shorter than any previously observed. Zachariasen (1954) has published a survey of the uranyl and similar type compounds and has given an empirical curve relating bond strength to bond length. In the plane of the carbonate groups (see Christ *et al.*, 1955) there are four U–O distances of 2·52 Å, each with a bond strength of 0·38, and two distances of 2·44 Å having a bond strength of 0·46. The remaining two bonds must have a strength of 1·78 in order to achieve a total of 6·0 valence electrons. A distance of 1·68 Å is empirically predicted. This distance compares very favorably with the observed value of $1{\cdot}67 \pm 0{\cdot}09$ Å.

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Crystallographic data for rhodanine, $C_3H_3S_2NO$.*

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(*Received* 8 *August* 1955)

Rhodanine was first prepared by Nencki (1877). Feigl & Pollack (1926) reported the water-insoluble silver and cuprous salts. Dubsky, Sindelar & Cernak (1938) and Feigl (1949) have studied the salt-forming properties of the molecule but these studies have not succeeded in clarifying the stereochemistry of rhodanine and its metallic salts. An attempt to prove the structures of these compounds by X-ray methods has been initiated in this laboratory.

Rhodanine crystals grown from a solution in hot ethanol were hexagonal plates with well formed {010} and {101} faces. Oscillation and Weissenberg photographs were taken about the three crystallographic axes with nickel-filtered Cu $K\alpha$ radiation and gave the monoclinic unit cell dimensions:

$$a = 10{\cdot}02 \pm 0{\cdot}10,\ b = 7{\cdot}67 \pm 0{\cdot}08,\ c = 7{\cdot}28 \pm 0{\cdot}07\ \text{Å}\,,\quad \beta = 102^\circ\ 38'\,.$$

There are 4 molecules per unit cell. Density: calculated 1·61 g.cm.$^{-3}$; observed (flotation) 1·55 g.cm.$^{-3}$.

Reflections of type ($h0l$) were observed only when $h+l = 2n$ and reflections of the type ($0k0$) only when $k = 2n$. These data fix the space group as C_{2h}^5–$P2_1/n$.

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

$$n_\alpha = 1{\cdot}585 \pm 0{\cdot}003,\ n_\beta = 1{\cdot}78 \pm 0{\cdot}01,\ n_\gamma = 1{\cdot}81 \pm 0{\cdot}01\,.$$

The powder pattern was run using nickel-filtered Cu $K\alpha$ radiation and Eastman Kodak No-Screen X-ray film. The intensities of the most intense lines, estimated visually by comparison with standard intensity strips prepared by timed exposures of one reflection from a single crystal, are summarized in Table 1.

Table 1. *Powder diffraction data*

d (Å)	I/I_0	d (Å)	I/I_0
3·25	0·23	1·82	0·09
3·04	0·09	1·70	1·00
2·48	0·26	1·30	0·34
2·20	0·34	1·25	0·26
2·00	0·44	1·20	0·23
1·95	0·13		

The intensities of 926 unique (hkl) reflections have been estimated visually by comparison with the standard intensity strips and a three-dimensional Patterson synthesis has been prepared. Attempts to solve this structure have thus far proven unsuccessful. However, work is continuing towards the solution of this problem.

The authors wish to acknowledge support of the Office of Ordnance Research under contract No. DA-008 ord-706.

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