Acta Cryst. (1973). B**29**, 2608

Salicylaldoxime

BY C. E. PFLUGER
Department of Chemistry, Syracuse University, Syracuse, New York 13210, *U.S.A.*

AND R. L. HARLOW
Department of Chemistry, University of Zambia, Box 2379, *Lusaka, Zambia*

(*Received* 28 *March* 1973; *accepted* 14 *May* 1973)

Abstract. Monoclinic, $P2_1/c$, $a=10{\cdot}41$ (1), $b=5{\cdot}078$ (5), $c=13{\cdot}60$ (1) Å, $\beta=112{\cdot}9$ (2)°, 22 °C, $C_7H_7NO_2$, $Z=4$, $D_x=1{\cdot}375$ g cm^{-3}. The structure consists of hydrogen bonded dimers of essentially planar molecules of salicylaldoxime.

Introduction. A preliminary study by Merritt & Schroeder (1956) reported a hexagonal unit cell for salicylaldoxime. While it is possible that two modifications exist, the powder pattern reported is that of the monoclinic form. The monoclinic crystals used in this study belong to space group $P2_1/c$ and were grown by slow cooling from an ethanol solution.

Intensity data were collected for 1049 reflections on a Stoe 2-circle automatic diffractometer using LiF-monochromated Cu $K\alpha$ radiation (max. $k=4$, max. $2\theta=130°$). The intensities were measured by means of ω scans of 1° per min over a width of 1·5°; background counts for 30 sec were taken at both ends of the scan range. The crystal, which was sealed in a 0·3 mm Lindemann glass capillary, showed no signs of instability as was evidenced by the absence of systematic changes in the intensity of the reference reflections. The intensities were corrected for Lorentz and polarization effects. Corrections for absorption were deemed unnecessary ($\mu=8{\cdot}62$ cm^{-1}, Cu $K\alpha$) and extinction did not prove to be a problem.

The structure was solved using a symbolic addition program supplied by Ahmed, Hall, Pippy & Saunderson (1968) and refined with Shiono's (1971) block-diagonal least-squares program using unit weights for all reflections. The heavy atoms were refined anisotropically, the hydrogens isotropically. Only the hydrogen attached to the phenolic oxygen, H(17), failed to refine properly with the O(10)–H(17) distance decreasing to 0·69 Å. H(17) was then fixed at the position found on

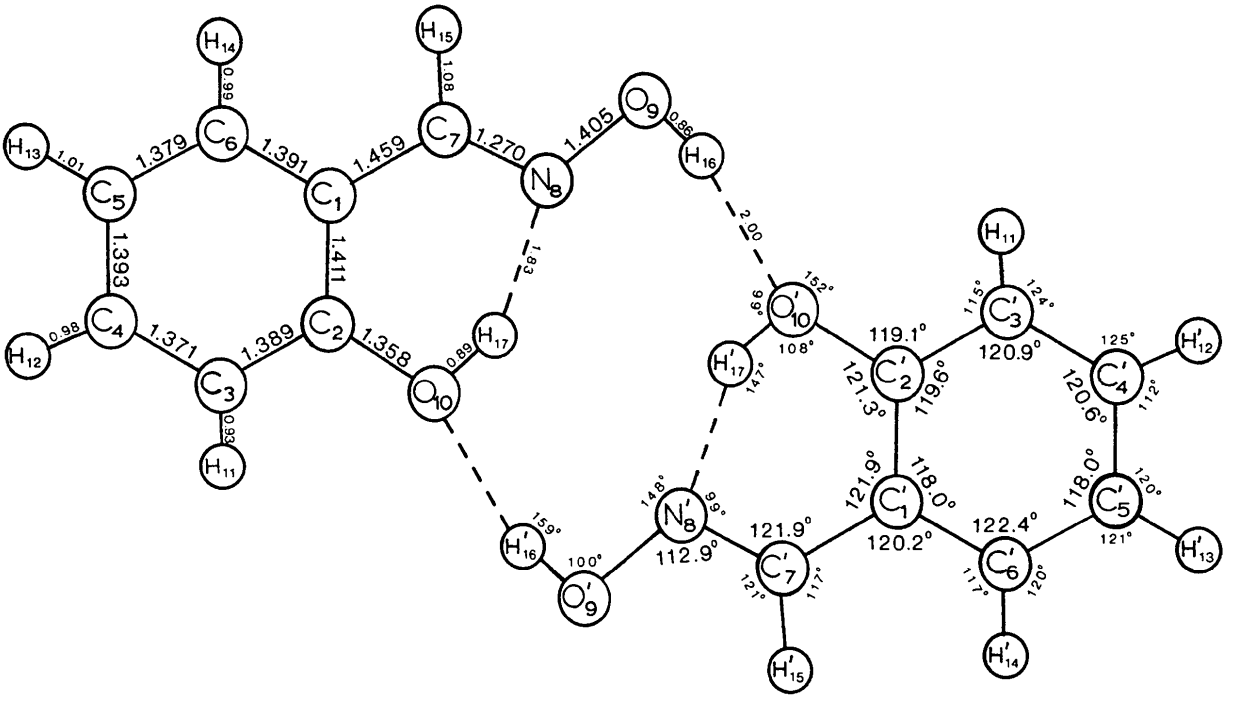

Fig. 1. Bond distances and angles for salicylaldoxime.

Table 1. *Final fractional coordinates and thermal parameters (with standard deviations in parentheses)*

The anisotropic thermal parameters are in the form:

$$\exp[-(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+2hk\beta_{12}+2hl\beta_{13}+2kl\beta_{23})].$$

(*a*) Heavy atoms (anisotropic therma parameters)

	x	*y*	*z*	$10^4\beta_{11}$	$10^3\beta_{22}$	$10^4\beta_{33}$	$10^4\beta_{12}$	$10^4\beta_{12}$	$10^4\beta_{23}$
C(1)	0·2503 (4)	0·4807 (8)	0·0093 (3)	80 (4)	40 (2)	51 (3)	15 (7)	23 (3)	21 (5)
C(2)	0·2049 (4)	0·4774 (8)	0·0945 (3)	82 (4)	42 (2)	50 (3)	3 (7)	23 (3)	8 (6)
C(3)	0·2614 (4)	0·6550 (9)	0·1781 (3)	106 (5)	46 (2)	60 (3)	− 9 (8)	33 (3)	−13 (6)
C(4)	0·3641 (4)	0·8273 (9)	0·1808 (3)	102 (5)	47 (2)	68 (3)	−16 (8)	13 (3)	− 8 (7)
C(5)	0·4122 (4)	0·8316 (10)	0·0985 (4)	89 (5)	58 (3)	81 (3)	−45 (9)	22 (3)	24 (8)
C(6)	0·3536 (4)	0·6600 (9)	0·0141 (3)	91 (4)	50 (2)	67 (3)	2 (8)	34 (3)	40 (6)
C(7)	0·1917 (4)	0·3037 (9)	−0·0819 (3)	98 (4)	44 (2)	53 (3)	7 (8)	34 (3)	13 (6)
N(8)	0·1006 (3)	0·1317 (7)	−0·0873 (2)	109 (4)	40 (2)	43 (2)	− 3 (6)	30 (2)	− 9 (4)
O(9)	0·0592 (3)	−0·0202 (7)	−0·1809 (2)	152 (4)	60 (2)	48 (2)	−80 (7)	46 (2)	−45 (5)
O(10)	0·1045 (3)	0·3076 (6)	0·0954 (2)	125 (4)	54 (2)	58 (2)	−72 (6)	50 (2)	−38 (4)

Table 1 (*cont.*)

(*b*) Hydrogen atoms (isotropic thermal parameters)

	x	*y*	*z*	10*B*
H(11)	0·220 (4)	0·655 (9)	0·228 (3)	55 (10)
H(12)	0·393 (5)	0·984 (11)	0·227 (4)	85 (14)
H(13)	0·490 (4)	0·995 (8)	0·102 (3)	44 (9)
H(14)	0·396 (4)	0·639 (9)	−0·039 (3)	62 (11)
H(15)	0·238 (5)	0·308 (10)	−0·141 (4)	85 (13)
H(16)	0·009 (4)	−0·137 (8)	−0·166 (3)	47 (9)
H(17)	0·085 (5)	0·200 (10)	0·040 (3)	93 (15)

the difference map and the refinement of all other parameters was continued until the *R* index reached 0·077 for all reflections and 0·069 omitting 101 reflections considered to be unobserved. The final positional and thermal parameters are given in Table 1.*

Discussion. The bond distances and angles are given in Fig. 1, which further shows the hydrogen bonding between two molecules of salicylaldoxime across a crystallographic center of symmetry. The e.s.d.'s for heavy-atom distances vary from 0·004 to 0·007 Å; for bonds to hydrogen atoms, the e.s.d.'s are about 0·05 Å. Angular e.s.d.'s average 0·4° for heavy-atom angles and 3° for the angles involving hydrogens. All bond distances and angles are those expected; a table of N–O and C=N bond lengths for various oximes has recently been published (Table 5, Bachechi & Zambonelli, 1972). No comparison between this structure and that of 5-chlorosalicylaldoxime (Simonsen, Pfluger & Thompson, 1961) will be made at this time; the estimated error of the latter structure is relatively large and hence is being redetermined.

The molecule as a whole is planar; a least-squares mean-plane calculation including all of the heavy atoms yields the equation $-0{\cdot}560X+0{\cdot}673Y-0{\cdot}228Z=0{\cdot}150$, where *X*, *Y*, and *Z* are the coordinates in Å with axes parallel to the cell edges. Of the heavy atoms, C(7) shows the largest deviation from the plane, 0·024 Å; the average deviation is 0·009 Å. The planes of the two molecules which are hydrogen-bonded miss being coplanar by only 0·30 Å.

The authors would like to thank Professor E. Wölfel, Lehrstuhl für Strukturforschung, Eduard Zintl Institut der Technischen Hochschule, Darmstadt, Germany, in whose laboratory the intensity data were taken. The assistance of the computer-center staff at the University of Zambia is also gratefully acknowledged.

* A list of structure factors has been deposited with the National Lending Library, England, as Supplementary Publication No. SUP 30140. Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

References

AHMED, F. R., HALL, S. R., PIPPY, M. E. & SAUNDERSON, C. P. (1968). *N.R.C. Crystallographic Programs for the IBM/360 System.* National Research Council, Ottawa, Canada.

BACHECHI, F. & ZAMBONELLI, L. (1972). *Acta Cryst.* B**28**, 2489–2494.

MERRITT, L. L. JR & SCHROEDER, E. (1956). *Acta Cryst.* **9**, 194.

SHIONO, R. (1971). *Crystallographic Computing Programs for IBM* 1130 *System.* Dept. of Crystallography, Univ. of Pittsburgh, Pittsburgh, Pa. 15213.

SIMONSEN, S. H., PFLUGER, C. E. & THOMPSON, C. M. (1961). *Acta Cryst.* **14**, 269–272.