(Trus & Marsh, 1973), and 19° in 2-amino-4-phenylthiazole hydrobromide monohydrate (Form, Raper & Downie, 1974)]. In the present molecule, however, a larger twist is required (up to 36°) to prevent steric interference with the CH_2 group at C(5) [C(17)···C(18) = 3·264; H(9)···H(11) = 2·31 Å]. This hindrance is reflected also in the large values of the exocyclic angles C(4)–C(5)–C(18) and C(5)–C(4)–C(12), 130·9 (2) and 126·9 (1)° respectively.

Appreciable double-bond character for the S(1)–C(2) and S(1)–C(5) bonds is indicated by the bond lengths of 1·732 (2) and 1·718 (2) Å respectively. The effect of conjugation of the phenyl groups with the thiazolyl ring is shown by the bond orders, as calculated by the formula of Jenkins (1955), of 1·32 for C(2)–C(6) and 1·27 for C(4)–C(12).

The conformation of the $-CH_2-COOH$ group can be described by the torsion angles S(1)–C(5)–C(18)–C(19), 76·8 (1)°, and C(5)–C(18)–C(19)–O(20), 32·1 (1)°. The observed lengths of the C–O bonds, as well as the relatively high temperature factors of the two O atoms, may be indicative of partial disorder of the carboxyl group (Leiserowitz, 1976).

As shown in Fig. 2, the molecules are hydrogen-bonded across centres of symmetry, thus forming cyclic centrosymmetric dimers. The O–H···O angle is close to linear (174°) with an O···O distance of 2·677 Å. The carboxyl groups forming the dimers are not coplanar; the displacement between them is 0·26 Å. The shortest contact between heavy atoms in different dimers is C(12)···C(16) (at $1-x$, $1-y$, $1-z$), 3·327 Å; all the other contacts appear to be in the normal range.

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Salicylohydroxamic Acid

BY INGRID KJØLLER LARSEN

Department of Chemistry BC, Royal Danish School of Pharmacy, Universitetsparken 2, DK-2100 Copenhagen Ø, Denmark

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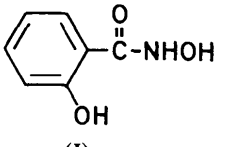
Abstract. $C_7H_7NO_3$, $M_r = 153·14$, m.p. 168–170°C, monoclinic, $C2/c$, $a = 23·783$ (5), $b = 4·608$ (1), $c = 12·575$ (3) Å, $\beta = 96·45$ (2)°, $Z = 8$, $D_m = 1·49$, $D_c = 1·485$ g cm^{-3}, μ(Mo $K\alpha$) = 1·27 cm^{-1}. The molecules are not quite planar. The conformation of O=C–N–OH is synperiplanar, and this conformation is stabilized by an intramolecular NH···O bond. The molecules in the crystal are connected by hydrogen bonds and van der Waals interactions.

Introduction. Salicylohydroxamic acid (I) was prepared as described for benzohydroxamic acid (Hauser & Renfrow, 1943). Single crystals were obtained by recrystallization from 50% aqueous ethanol. The crystal chosen for data collection (0·10 × 0·20 × 0·40 mm) was mounted in a glass capillary and oriented with **b** parallel to the φ axis of the goniostat. The X-ray intensities in the θ range 2·5–25·0° were collected on a Nonius three-circle automatic diffractometer by the moving-crystal stationary-detector technique, with graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0·7107$ Å), scan-range 1·2°, and scan speed 1·2° min^{-1}. 1154 independent reflexions were measured; 699 with $I \geq 3·0\sigma(I)$ were used in the structure refinements.



(I)

Table 1. *Fractional atomic coordinates* ($\times 10^4$, *for* H $\times 10^3$) *for salicylohydroxamic acid*

	x	*y*	*z*
C(1)	6157 (1)	243 (8)	6416 (2)
C(2)	6532 (2)	1566 (9)	7203 (3)
C(3)	6930 (2)	3519 (9)	6959 (3)
C(4)	6964 (2)	4256 (10)	5897 (3)
C(5)	6598 (2)	3002 (10)	5101 (3)
C(6)	6196 (1)	1016 (8)	5349 (2)
O(6)	5839 (1)	−282 (7)	4576 (2)
C(7)	5745 (1)	−1889 (8)	6766 (2)
O(7)	5727 (1)	−2452 (5)	7741 (2)
N(8)	5400 (1)	−3196 (7)	6027 (2)
O(9)	5045 (1)	−5422 (6)	6288 (2)
H(2)	650 (1)	107 (7)	793 (2)
H(3)	718 (1)	442 (8)	749 (3)
H(4)	725 (2)	571 (9)	569 (3)
H(5)	662 (1)	341 (8)	438 (3)
H(6)	584 (2)	46 (9)	405 (3)
H(8)	538 (1)	−285 (8)	534 (3)
H(9)	479 (1)	−457 (8)	664 (3)

The structure was solved by direct methods using the program *MULTAN* (Germain, Main & Woolfson, 1971, updated 1974). Full-matrix least-squares refinement of positional parameters for all atoms and anisotropic temperature parameters for the non-hydrogen atoms led to a final R of 0·037. The seven H atoms were located in a difference map. The quantity minimized was $\Sigma w(|F_o| - |F_c|)^2$ with unit weights for all observed reflexions. The weight analysis indicated that this choice was satisfactory. The scattering factors for H were those of Stewart, Davidson & Simpson (1965), and for O, N and C those of Cromer & Mann (1968). The final values of the atomic positions are listed in Table 1.* The programs used in the refinement were from the XRAY system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972) and the figures were drawn with *ORTEP* II (Johnson, 1971).

Discussion. Salicylohydroxamic acid has been reported to be a selective inhibitor of DNA synthesis in Ehrlich ascites tumour cells, with an effect similar to that of hydroxyurea (Gale & Hynes, 1968). Hydroxyurea has been shown to inhibit the enzyme system ribonucleotide reductase of *E. coli* by destroying the organic free radical of protein B2, a subunit of the enzyme system (Atkin, Thelander, Reichard & Lang, 1973). Salicylohydroxamic acid has recently been shown to be a less potent inhibitor of the same enzyme system (L. Thelander, personal communication). An X-ray analysis of the compound was undertaken as part of a series of structure determinations of hydroxyurea analogues.

Bond lengths and angles of the molecule are given in Table 2. These molecular dimensions are quite similar to those of salicylic acid (Sundaralingam & Jensen, 1965), and of benzohydroxamic acid as found in its Fe^{III} complex (Lindner & Göttlicher, 1969). The molecule is not quite planar (*cf.* Table 3). The planar part of the hydroxamic acid group is inclined at 1·6° to the plane of the benzene ring. The torsional angle C(2)–C(1)–C(7)–O(7) is ±1·2 (5)°. Similar small inclinations are also observed in salicylic acid and its

Table 2. *Bond distances* (Å) *and angles* (°) *for salicylohydroxamic acid*

C(1)–C(2)	1·395 (5)	C(2)–H(2)	0·96 (3)
C(2)–C(3)	1·366 (6)	C(3)–H(3)	0·95 (3)
C(3)–C(4)	1·389 (6)	C(4)–H(4)	1·01 (4)
C(4)–C(5)	1·377 (6)	C(5)–H(5)	0·93 (3)
C(5)–C(6)	1·385 (6)	O(6)–H(6)	0·75 (4)
C(6)–C(1)	1·401 (5)	N(8)–H(8)	0·87 (3)
C(6)–O(6)	1·355 (4)	O(9)–H(9)	0·88 (4)
C(1)–C(7)	1·489 (5)		
C(7)–O(7)	1·258 (4)		
C(7)–N(8)	1·316 (4)		
N(8)–O(9)	1·390 (4)		
C(1)–C(2)–C(3)	122·1 (3)	C(1)–C(2)–H(2)	118 (2)
C(2)–C(3)–C(4)	119·6 (3)	C(3)–C(2)–H(2)	120 (2)
C(3)–C(4)–C(5)	119·7 (4)	C(2)–C(3)–H(3)	122 (2)
C(4)–C(5)–C(6)	120·6 (4)	C(4)–C(3)–H(3)	118 (2)
C(5)–C(6)–C(1)	120·3 (3)	C(3)–C(4)–H(4)	121 (2)
C(6)–C(1)–C(2)	117·6 (3)	C(5)–C(4)–H(4)	119 (2)
C(6)–C(1)–C(7)	124·6 (3)	C(4)–C(5)–H(5)	121 (2)
C(2)–C(1)–C(7)	117·8 (3)	C(6)–C(5)–H(5)	118 (2)
C(1)–C(7)–N(8)	118·2 (3)	C(6)–O(6)–H(6)	112 (3)
C(1)–C(7)–O(7)	121·4 (3)	C(7)–N(8)–H(8)	125 (2)
O(7)–C(7)–N(8)	120·4 (3)	O(9)–N(8)–H(8)	113 (2)
C(7)–N(8)–O(9)	121·2 (3)	N(8)–O(9)–H(9)	105 (2)
C(5)–C(6)–O(6)	121·5 (3)		
C(1)–C(6)–O(6)	118·1 (3)		

Table 3. *Deviations* (Å) *of atoms from the least-squares planes through the benzene ring* (*A*) *and the hydroxamic acid group* (*B*)

The equations of the planes are in direct space, and atoms defining the planes are indicated by an asterisk.

(*A*) $15{\cdot}807x - 3{\cdot}426y - 1{\cdot}858z - 8{\cdot}454 = 0$
(*B*) $16{\cdot}145x - 3{\cdot}375y - 1{\cdot}621z - 8{\cdot}819 = 0$

	(*A*)		(*B*)
C(1)	0·004*	C(1)	0·001*
C(2)	−0·004*	C(7)	−0·002*
C(3)	0·001*	O(7)	0·001*
C(4)	0·000*	N(8)	0·001*
C(5)	0·000*	O(9)	0·138
C(6)	−0·001*		
O(6)	0·023		
C(7)	0·018	Angle (*A*):(*B*) = 1·6°	

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33123 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 4. *Distances and angles concerning the hydrogen-bonding system*

Symmetry code: (I) $x, -y, z-\frac{1}{2}$; (II) $-x+1, y, -z+1\frac{1}{2}$; (III) $-x+1, -y-1, -z$.

X–H···Y	X···Y	H···Y	∠X–H···Y
O(6)–H(6)···O(7)^{I}	2·616 (3) Å	1·87 (4) Å	172 (4)°
O(9)–H(9)···O(7)^{II}	2·691 (4)	1·82 (4)	173 (3)
N(8)–H(8)···O(9)^{III}	3·051 (4)	2·32 (3)	141 (3)
N(8)–H(8)···O(6)	2·580 (4)	1·94 (3)	129 (3)

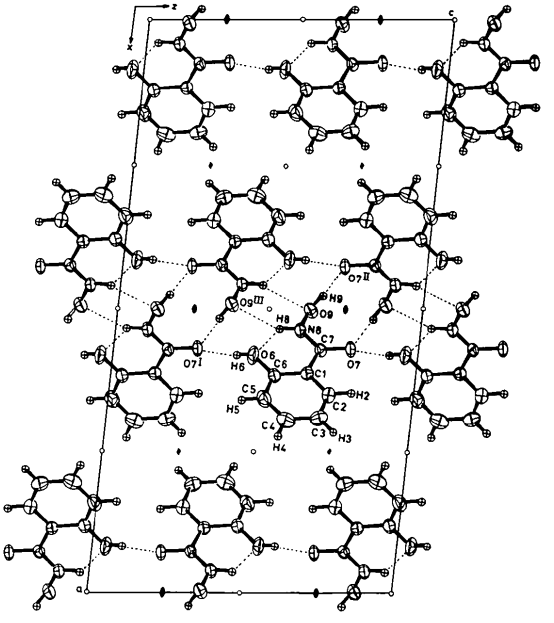


Fig. 1. The structure of salicylohydroxamic acid viewed along **b**.

derivatives (Singh & Vijayan, 1974), whereas the corresponding inclinations of the three benzohydroxamate ions in the Fe^{III} complex were found to be 9·4, 20·7 and 39·1° respectively (Lindner & Göttlicher, 1969).

In salicylic acid and its derivatives and hydrogen-bonded complexes, the *ortho* hydroxyl group is found to be intramolecularly hydrogen bonded to the carbonyl O atom of the carboxyl group. In salicylohydroxamic acid this hydroxyl group is not hydrogen bonded to the carbonyl O atom, but is the acceptor for an intramolecular NH···O bond (*cf.* Table 4 and Fig. 1). Consequently, the conformation of the O=C–N–OH moiety is synperiplanar, the torsional angle O(7)–C(7)–N(8)–O(9) being ±6·3 (5)°.

The molecular packing is stabilized by hydrogen bonds and van der Waals interactions. In the z direction the molecules are linked into chains by the hydrogen bond O(6)–H(6)···O(7)^{I}, and these chains are interconnected into double chains by the O(9)–H(9)···O(7)^{II} bond. In addition, H(8) is probably involved in bifurcated hydrogen bonding, as the distance to O(9)^{III} is rather small and the N(8)–H(8)···O(9)^{III} angle rather favourable (*cf.* Table 4). The double chains are connected solely by van der Waals interactions, as are the layers of molecules in the y direction.

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