

Fig. 2. Stereoview looking down the  $c^*$  axis to show the relation of units along the polymer chain axis.

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## [2-Phenyl-4-(*p*-chlorophenyl)-5-thiazolyl]acetic Acid

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**Abstract.**  $C_{17}H_{12}ClNO_2S$ ,  $M_r = 329.8$ , triclinic,  $P\bar{1}$ ,  $a = 7.423$  (1),  $b = 9.098$  (1),  $c = 11.549$  (1) Å,  $\alpha = 79.73$  (2),  $\beta = 79.49$  (1),  $\gamma = 88.47$  (2)°,  $Z = 2$ ,  $\mu(Mo K\alpha) = 3.95$  cm<sup>-1</sup>,  $D_x = 1.453$ ,  $D_m = 1.45$  (1) g cm<sup>-3</sup>, at  $20 \pm 2^\circ$ C. For 2850 reflexions with  $I > 0$ ,  $R = 0.039$  and  $R_w = 0.040$ . The phenyl group bonded to the thiazolyl system in the 2 position is twisted by  $15.6^\circ$  with respect to the plane of the heterocyclic ring; the twist angle of the second phenyl ring is  $35.9^\circ$ . The plane of the carboxyl group is almost exactly perpendicular to the plane of the thiazolyl ring. The structure consists of centrosymmetric dimers formed by hydrogen bonds between carboxyl groups of adjacent molecules.

**Introduction.** It has been pointed out (Shen, 1972) that a major concern in the current search for new anti-arthritis drugs is to minimize their side effects. In the field of aryl- and heteroarylalkanoic acids, a partial dissociation of activity and toxicity has been achieved by the fortuitous selection of some aryl moieties. The preferred geometry of the aryl groups has been indicated by Shen (1972) as a possible means to a rational approach to this problem. In line with this interest, the structure of the title compound, a non-steroidal antiinflammatory agent (Brown, Cater, Cavalla, Green, Newberry & Wilson, 1974), has been investigated.

Crystals in the form of colourless prisms were

prepared by evaporation of an ethyl acetate solution. Unit-cell and intensity data (no systematic absences) were measured on a computer-controlled four-circle diffractometer, with Mo  $K\alpha$  radiation (graphite monochromator) and variable-rate  $\theta$ - $2\theta$  scan technique. Background measurements were taken at both ends of the scan range, each for a time equal to one-half of the scan time. Two standard reflexions were checked after every 50 intensity measurements; they showed no appreciable trend. Of 3115 unique reflexions with  $2\theta \leq 52^\circ$ , 2850 with  $I > 0$  were classified as observed. Each reflexion was assigned a variance of  $\sigma^2(I) = \sigma^2(I)_{c.s.} + (0.03S)^2$ , where  $S$  is the scan count. The intensities and their standard deviations were corrected for Lorentz and polarization effects but not for absorption [ $\mu(\text{Mo } K\alpha) = 3.95 \text{ cm}^{-1}$ ; approximate crystal dimensions  $0.40 \times 0.35 \times 0.15 \text{ mm}$ ].

The structure was solved by direct methods; the positions of all non-hydrogen atoms were recovered from an  $E$  map and those of the H atoms from a difference synthesis. Refinement was by full-matrix least-squares minimization of the quantity  $\sum w(|F_o| - k|F_c|)^2$ , with weights  $w = 4F_o^2/\sigma^2(F_o^2)$ . Final cycles included in a single matrix 248 parameters: coordinates for all 34 atoms, anisotropic temperature coefficients for the 22 heavy atoms, isotropic  $B$ 's for the 12 H atoms, a scale factor, and a secondary extinction coefficient (Larson, 1967). The final  $R$  index  $[\sum (|F_o| - |F_c|)/\sum |F_o|]$  was 0.039, the weighted  $R$   $\{[\sum w(|F_o| - |F_c|)^2/\sum wF_o^2]^{1/2}\}$  0.040, and the goodness-of-fit  $\{[\sum w(|F_o| - |F_c|)^2/(m - s)]^{1/2}\}$  for  $m = 2850$  observations and  $s = 248$  parameters, 1.42. The largest shift in the final least-squares calculations

Table 1. *Positional parameters for heavy atoms* ( $\times 10^5$ )

	x	y	z
S(1)	67673 (6)	93721 (5)	13042 (3)
C(2)	77189 (22)	94463 (16)	25558 (13)
N(3)	67944 (17)	86977 (14)	35452 (11)
C(4)	52780 (21)	79991 (16)	33395 (13)
C(5)	50289 (22)	82487 (17)	21766 (13)
C(6)	94250 (21)	102744 (17)	24741 (14)
C(7)	103690 (25)	100265 (21)	34177 (17)
C(8)	119189 (28)	108581 (24)	33824 (20)
C(9)	125466 (29)	119304 (23)	23987 (20)
C(10)	116478 (29)	121605 (23)	14454 (20)
C(11)	100903 (27)	113399 (20)	14718 (17)
C(12)	40868 (20)	71006 (16)	43783 (13)
C(13)	38423 (23)	75539 (18)	54848 (15)
C(14)	28050 (24)	67150 (19)	64851 (16)
C(15)	19781 (21)	54117 (18)	63727 (15)
C(16)	21924 (22)	49382 (19)	52913 (16)
C(17)	32541 (22)	57760 (17)	42993 (15)
C(18)	35354 (24)	77534 (19)	16196 (15)
C(19)	41094 (22)	65165 (17)	9250 (13)
O(20)	52241 (19)	55767 (14)	12132 (12)
O(21)	33158 (20)	65308 (16)	170 (12)
Cl(22)	6857 (7)	43517 (6)	76451 (5)

Table 2. *Positional* ( $\times 10^4$ ) *and thermal* ( $\times 10$ ) *parameters for the hydrogen atoms*

The form of the temperature factor is  $\exp(-B\lambda^{-2} \sin^2 \theta)$ .

	x	y	z	$B$ ( $\text{\AA}^2$ )
H(1)	9953 (28)	9278 (22)	4070 (18)	58 (5)
H(2)	12490 (31)	10721 (24)	4033 (20)	70 (6)
H(3)	13648 (30)	12483 (23)	2407 (18)	63 (5)
H(4)	11985 (30)	12918 (24)	795 (20)	67 (5)
H(5)	9470 (27)	11548 (21)	840 (18)	53 (5)
H(6)	4391 (24)	8477 (20)	5549 (15)	44 (4)
H(7)	2684 (26)	7018 (21)	7259 (18)	56 (4)
H(8)	1745 (25)	4006 (21)	5204 (16)	50 (4)
H(9)	3464 (22)	5403 (17)	3547 (15)	40 (4)
H(10)	3110 (23)	8550 (19)	1093 (15)	42 (4)
H(11)	2490 (24)	7356 (19)	2239 (16)	43 (4)
H(12)	3715 (36)	5822 (29)	-338 (23)	84 (7)

was  $0.2\sigma$ . A final difference map showed a maximum value of  $0.27 \text{ e \AA}^{-3}$ , located near the O(20) atom.

Scattering factors were from Cromer & Waber (1965), except that for H which was from Stewart, Davidson & Simpson (1965). The S and Cl form factors were increased by 0.13 and 0.15, respectively, to take into account the real component of anomalous dispersion (Cromer, 1965). Final positional parameters and isotropic thermal parameters (for H) are listed in Tables 1 and 2.\* The final value of the extinction parameter  $g$  was  $18.1(2.0) \times 10^{-6}$ .

**Discussion.** Fig. 1 shows the molecular shape and numbering scheme. Bond distances and angles involving non-hydrogen atoms are reported in Table 3. The

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33116 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

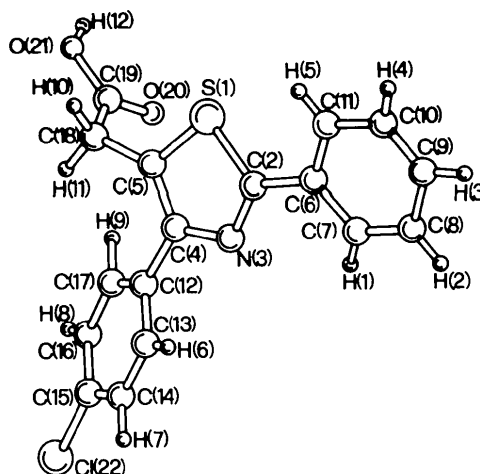


Fig. 1. Labelled perspective drawing of the molecule.

C—H bond lengths are in the range 0.92–0.98 Å (mean 0.95 Å), while the O(21)—H(12) distance is 0.84 (3) Å. No unusual values for the angles involving the H atoms are present in the molecule.

The phenyl ring at C(2) is slightly distorted (plane *B* of Table 4); the other two rings are each closely planar.

The dihedral angles  $A \wedge B$  and  $A \wedge C$  of Table 4 can be assumed as a measure of the twist angle of the phenyl groups with respect to the thiazolyl moiety. Ring systems bonded to the 4 position of thiazole are usually twisted by less than 20° [e.g. 10° in thiabendazole

Table 3. Bond lengths (Å) and angles (°)

S(1)—C(2)	1.732 (2)	C(4)—C(12)	1.480 (2)
C(2)—N(3)	1.304 (2)	C(12)—C(13)	1.391 (2)
N(3)—C(4)	1.384 (2)	C(13)—C(14)	1.379 (3)
C(4)—C(5)	1.367 (2)	C(14)—C(15)	1.386 (2)
C(5)—S(1)	1.718 (2)	C(15)—C(16)	1.373 (3)
C(2)—C(6)	1.470 (2)	C(16)—C(17)	1.380 (2)
C(6)—C(7)	1.383 (3)	C(17)—C(12)	1.393 (2)
C(7)—C(8)	1.383 (3)	C(5)—C(18)	1.495 (2)
C(8)—C(9)	1.376 (3)	C(18)—C(19)	1.504 (2)
C(9)—C(10)	1.371 (3)	C(19)—O(20)	1.220 (2)
C(10)—C(11)	1.385 (3)	C(19)—O(21)	1.291 (2)
C(11)—C(6)	1.393 (3)	C(15)—Cl(22)	1.743 (2)
C(5)—S(1)—C(2)	90.1 (1)	C(9)—C(10)—C(11)	120.5 (2)
S(1)—C(2)—N(3)	113.8 (1)	C(10)—C(11)—C(6)	119.9 (2)
S(1)—C(2)—C(6)	121.7 (1)	C(4)—C(12)—C(13)	119.3 (1)
N(3)—C(2)—C(6)	124.5 (1)	C(4)—C(12)—C(17)	122.2 (1)
C(2)—N(3)—C(4)	111.4 (1)	C(13)—C(12)—C(17)	118.5 (1)
N(3)—C(4)—C(5)	115.2 (1)	C(12)—C(13)—C(14)	121.1 (2)
N(3)—C(4)—C(12)	117.9 (1)	C(13)—C(14)—C(15)	119.0 (2)
C(5)—C(4)—C(12)	126.9 (1)	C(14)—C(15)—C(16)	121.2 (2)
C(4)—C(5)—S(1)	109.4 (1)	C(14)—C(15)—Cl(22)	118.6 (1)
C(4)—C(5)—C(18)	130.9 (2)	C(16)—C(15)—Cl(22)	120.2 (1)
S(1)—C(5)—C(18)	119.7 (1)	C(15)—C(16)—C(17)	119.3 (2)
C(2)—C(6)—C(7)	119.8 (2)	C(16)—C(17)—C(12)	120.9 (2)
C(2)—C(6)—C(11)	121.1 (2)	C(5)—C(18)—C(19)	113.2 (1)
C(7)—C(6)—C(11)	119.0 (2)	C(18)—C(19)—O(20)	122.1 (2)
C(6)—C(7)—C(8)	120.5 (2)	C(18)—C(19)—O(21)	114.5 (1)
C(7)—C(8)—C(9)	120.2 (2)	O(20)—C(19)—O(21)	123.4 (2)
C(8)—C(9)—C(10)	119.9 (2)		

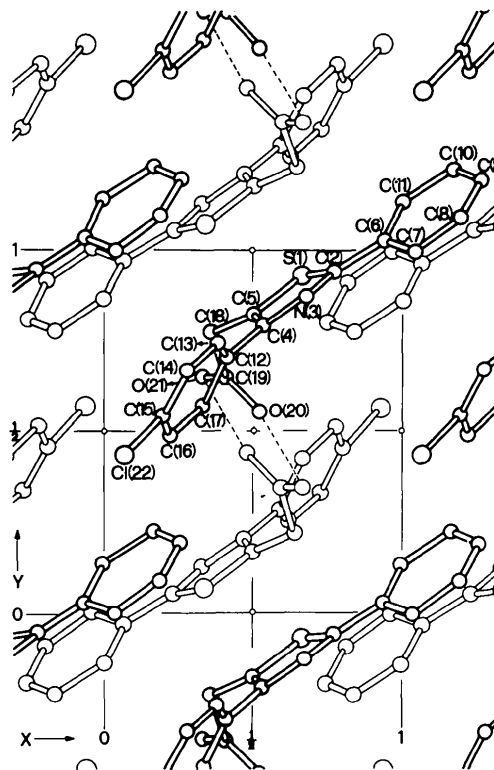


Fig. 2. A drawing of the structure as viewed down *c*. Dashed lines represent hydrogen bonds.

Table 4. Deviations (Å) from least-squares planes

The equations are referred to the triclinic axes and fractional coordinates; the distance in each equation is the origin-to-plane distance. Atoms marked with an asterisk were omitted from the least-squares calculations; the remaining atoms were given equal weights.

Plane <i>A</i>		Plane <i>B</i>		Plane <i>C</i>		Plane <i>D</i>	
	$\Delta$		$\Delta$		$\Delta$		$\Delta$
S(1)	0.000 (1)	C(6)	0.009 (2)	C(12)	−0.002 (2)	C(18)	−0.001 (2)
C(2)	−0.005 (2)	C(7)	−0.010 (2)	C(13)	−0.003 (2)	C(19)	0.003 (1)
N(3)	0.005 (1)	C(8)	−0.002 (2)	C(14)	0.006 (2)	O(20)	−0.001 (1)
C(4)	−0.006 (2)	C(9)	0.010 (2)	C(15)	−0.002 (2)	O(21)	−0.001 (1)
C(5)	0.003 (2)	C(10)	−0.005 (2)	C(16)	−0.004 (2)	*H(12)	0.02 (2)
*C(6)	−0.034 (2)	C(11)	−0.007 (2)	C(17)	0.006 (2)	*O(20)†	0.260 (1)
*C(12)	0.002 (2)	*C(2)	0.083 (2)	*C(4)	0.049 (2)		
*C(18)	0.055 (2)			*Cl(22)	0.024 (1)		

Plane *A*:  $-3.7466x + 7.6021y + 2.3564z = 4.8965$   
 Plane *B*:  $-3.5480x + 6.9287y + 5.1712z = 5.0450$   
 Plane *C*:  $6.3082x - 4.2078y + 3.1066z = 0.9524$   
 Plane *D*:  $4.8659x + 4.5463y - 4.1363z = 4.5764$

Dihedral angles (°)

$A \wedge B$	15.6	$A \wedge C$	35.9
$A \wedge D$	89.4	$B \wedge C$	45.8
$B \wedge D$	79.6	$C \wedge D$	78.2

† At  $1 - x, 1 - y, -z$ ; acceptor of hydrogen bond from O(21)—H(12).

(Trus & Marsh, 1973), and  $19^\circ$  in 2-amino-4-phenylthiazole hydrobromide monohydrate (Form, Raper & Downie, 1974)]. In the present molecule, however, a larger twist is required (up to  $36^\circ$ ) to prevent steric interference with the  $\text{CH}_2$  group at C(5) [ $\text{C}(17)\cdots\text{C}(18) = 3.264$ ;  $\text{H}(9)\cdots\text{H}(11) = 2.31$  Å]. This hindrance is reflected also in the large values of the exocyclic angles  $\text{C}(4)\text{—C}(5)\text{—C}(18)$  and  $\text{C}(5)\text{—C}(4)\text{—C}(12)$ ,  $130.9$  (2) and  $126.9$  (1) $^\circ$  respectively.

Appreciable double-bond character for the  $\text{S}(1)\text{—C}(2)$  and  $\text{S}(1)\text{—C}(5)$  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  $\text{C}(2)\text{—C}(6)$  and  $1.27$  for  $\text{C}(4)\text{—C}(12)$ .

The conformation of the  $\text{—CH}_2\text{—COOH}$  group can be described by the torsion angles  $\text{S}(1)\text{—C}(5)\text{—C}(18)\text{—C}(19)$ ,  $76.8$  (1) $^\circ$ , and  $\text{C}(5)\text{—C}(18)\text{—C}(19)\text{—O}(20)$ ,  $32.1$  (1) $^\circ$ . The observed lengths of the  $\text{C—O}$  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  $\text{O—H}\cdots\text{O}$  angle is close to linear ( $174^\circ$ ) with an  $\text{O}\cdots\text{O}$  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  $\text{C}(12)\cdots\text{C}(16)$  (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

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**Abstract.**  $\text{C}_7\text{H}_7\text{NO}_3$ ,  $M_r = 153.14$ , m.p.  $168\text{--}170^\circ\text{C}$ , monoclinic,  $C2/c$ ,  $a = 23.783$  (5),  $b = 4.608$  (1),  $c = 12.575$  (3) Å,  $\beta = 96.45$  (2) $^\circ$ ,  $Z = 8$ ,  $D_m = 1.49$ ,  $D_c = 1.485$  g cm $^{-3}$ ,  $\mu(\text{Mo } K\alpha) = 1.27$  cm $^{-1}$ . The molecules are not quite planar. The conformation of  $\text{O=C—N—OH}$  is synperiplanar, and this conformation

is stabilized by an intramolecular  $\text{NH}\cdots\text{O}$  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 ( $0.10 \times 0.20 \times 0.40$  mm) was mounted in a glass capillary and oriented with  $\mathbf{b}$  parallel to the  $\varphi$  axis of the goniostat. The X-ray intensities in the  $\theta$  range  $2.5\text{--}25.0^\circ$  were collected on a Nonius three-circle automatic diffractometer by the moving-crystal stationary-detector technique, with graphite-monochromated  $\text{Mo } K\alpha$  radiation ( $\lambda = 0.7107$  Å), scan-range  $1.2^\circ$ , and scan speed  $1.2^\circ$  min $^{-1}$ . 1154 independent reflexions were measured; 699 with  $I \geq 3.0\sigma(I)$  were used in the structure refinements.

