

Fig. 3. Projection of the structure along the *b* axis.

1.525–1.548 Å, with a mean value of 1.539 Å. The conformation of the *tert*-butyl group is shown in Fig. 2. One of the three methyl groups of each *tert*-butyl group is *cis* to the C–C bond in the benzene ring.

A projection of the crystal structure is shown in Fig. 3. There are no intermolecular contacts shorter than the sums of the van der Waals radii.

All crystallographic computations were performed on the HITAC 8800/8700 computer of the Computer Center of the University of Tokyo, using the local version of the program system UNICS (Crystallographic Society of Japan, 1967; Sakurai, Ito, Iwasaki, Watanabe & Fukuhara, 1967). The author is grateful to Professor Naoki Inamoto and Dr Renji Okazaki of the University of Tokyo for supplying the sample and to Dr Ei-ichi Asada and Mrs Midori Goto of the National Chemical Laboratory for Industry for the use of the X-ray diffractometer.

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Flukicides.

II.* Crystal Structures of the Triclinic and Monoclinic Polymorphs of 3,3',5,5',6-Pentachloro-2'-hydroxysalicylanilide

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Abstract

The structures of two polymorphs of $C_{13}H_6Cl_5NO_3$ have been determined. Crystals of the α polymorph are triclinic, space group $P1$, with $a = 8.382$ (4), $b =$

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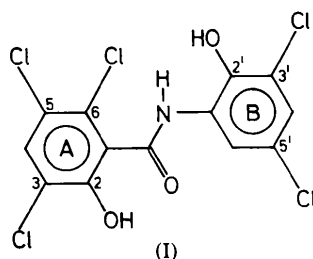
9.392 (6), $c = 10.183$ (3) Å, $\alpha = 93.06$ (5), $\beta = 95.38$ (3), $\gamma = 112.61$ (4)°, $Z = 2$. Crystals of the β polymorph are monoclinic, space group $P2_1/n$, with $a = 7.078$ (6), $b = 23.533$ (3), $c = 9.359$ (3) Å, $\beta = 110.02$ (4)°, $Z = 4$. The structures were refined to $R = 0.053$ (1746 terms) and $R = 0.060$ (1464 terms) for the α and β forms respectively. In the former the

* Part I: Sindt & Mackay (1978).

molecules are virtually planar, whereas in the latter they assume a slight butterfly conformation. The hydrogen bonding in the two crystals is similar, but the packing in the less-soluble β polymorph results in greater molecular interlocking.

Introduction

3,3',5,5',6-Pentachloro-2'-hydroxysalicylanilide (I), otherwise known as oxyclozanide, is an effective drug in the treatment of chronic liver-fluke disease (Walley, 1966). When crystallized from methanol, two unsolvated polymorphs were isolated (Pearson & Varney, 1969), one forming discrete plate-like crystals (α form), and the other long interlocking needles (β form). Both forms are relatively insoluble in water, and in aqueous suspension the α form undergoes a polymorphic phase transition to the less-soluble β form.



The structures of the two polymorphs have been determined to define the molecular conformations and packing within the crystals. These features are of interest in view of the different solubilities reported by Pearson & Varney (1969) for the α and β forms (4.8 and 2.4 mg per 100 ml). Oxyclozanide is the second compound in a series of structurally related flukicides

Table 1. *Crystal data*

	α Form	β Form
Molecular formula	$C_{13}H_6Cl_5NO_3$	$C_{13}H_6Cl_5NO_3$
M_r	401.5	401.5
Crystal system	Triclinic	Monoclinic
Space group	$P\bar{1}$	$P2_1/n$
	(confirmed by refinement)	
a	8.382 (4) Å	7.078 (6) Å
b	9.392 (6)	23.533 (3)
c	10.183 (3)	9.359 (3)
α	93.06 (5)°	
β	95.38 (3)	110.02 (4)°
γ	112.61 (4)	
U	733.3 (7) Å ³	1464 (1) Å ³
D_m	1.81 Mg m ⁻³	1.82 Mg m ⁻³
D_c	1.82	1.82
Z	2	4
$F(000)$	400	800
λ	1.5418 Å	0.7107 Å
μ	8.86 mm ⁻¹ (Cu $K\alpha$)	0.91 mm ⁻¹ (Mo $K\alpha$)

Table 2. *Final atomic coordinates* ($\times 10^4$, for H $\times 10^3$) with *e.s.d.*'s in parentheses

	α Polymorph			β Polymorph		
	x	y	z	x	y	z
Cl(1)	234 (3)	-1503 (2)	2840 (2)	1093 (4)	968 (1)	4176 (2)
Cl(2)	-1110 (3)	2978 (2)	568 (2)	2271 (4)	-118 (1)	9409 (3)
Cl(3)	1283 (3)	5401 (2)	2799 (2)	3159 (4)	1015 (1)	11195 (2)
Cl(4)	4802 (3)	10974 (2)	7456 (2)	2683 (3)	3251 (1)	15285 (2)
Cl(5)	6746 (3)	6952 (2)	10031 (2)	1446 (4)	4428 (1)	10142 (3)
O(1)	2340 (7)	896 (5)	4861 (4)	2043 (9)	1971 (2)	5995 (5)
O(2)	3854 (7)	3568 (5)	5885 (5)	2911 (10)	2546 (2)	8311 (6)
O(3)	2801 (7)	8138 (5)	5471 (4)	2704 (9)	2231 (2)	13260 (5)
N	2736 (8)	5359 (5)	5564 (5)	2354 (9)	2238 (2)	10409 (6)
C(1)	1763 (9)	3024 (6)	3995 (6)	2423 (11)	1549 (3)	8434 (8)
C(2)	1537 (9)	1450 (6)	3933 (6)	2073 (11)	1513 (3)	6839 (8)
C(3)	478 (9)	413 (7)	2880 (6)	1675 (12)	990 (3)	6116 (8)
C(4)	-351 (9)	882 (7)	1877 (6)	1704 (13)	500 (3)	6916 (9)
C(5)	-104 (9)	2421 (7)	1887 (6)	2164 (13)	518 (3)	8478 (9)
C(6)	929 (9)	3483 (6)	2924 (6)	2522 (12)	1034 (3)	9225 (8)
C(7)	2862 (9)	4023 (6)	5203 (6)	2626 (12)	2145 (3)	9062 (8)
C(1')	3743 (9)	6469 (6)	6637 (6)	2316 (11)	2778 (3)	11087 (8)
C(2')	3774 (10)	7964 (7)	6551 (6)	2459 (11)	2751 (3)	12625 (8)
C(3')	4737 (9)	9119 (6)	7555 (6)	2373 (12)	3263 (3)	13363 (8)
C(4')	5657 (9)	8820 (7)	8621 (6)	2057 (12)	3774 (3)	12622 (9)
C(5')	5590 (9)	7347 (7)	8683 (6)	1864 (12)	3783 (3)	11092 (8)
C(6')	4640 (9)	6149 (7)	7709 (6)	1996 (11)	3290 (3)	10317 (8)
H(O1)	322	172	554	275	219	671
H(O3)	309	916	551	264	224	1438
H(N)	205	560	498	308	196	1111
H(4)	-118	22	115	195	19	661
H(4')	643	960	932	244	417	1321
H(6')	460	499	779	201	333	922

to be studied by X-ray diffraction. The structure of 3,5-diiodo-3',4'-dichlorosalicylanilide (DDS) has been reported (Sindt & Mackay, 1978).

Experimental

Yellow crystals of both polymorphs were obtained by recrystallization of the amorphous material from methanol. The α and β forms were identified by their growth as discrete plates and interlocking needles respectively. Weissenberg photographs showed crystals of the α form to be triclinic; the β form was found to be monoclinic and to belong to the space group $P2_1/n$. Cell parameters were determined on a four-circle diffractometer by least squares from 2θ values measured for 25 strong reflections for the α form, and 15 for the β form. Densities were determined by flotation in a mixture of xylene and bromoform. The crystal data are given in Table 1.

Intensities were recorded for both crystals on a Rigaku AFC four-circle diffractometer (graphite crystal monochromator). Cu $K\alpha$ radiation was used for the triclinic, and Mo $K\alpha$ for the monoclinic structure. The crystal dimensions were $0.17 \times 0.14 \times 0.09$ and $0.32 \times 0.05 \times 0.17$ mm respectively. Each crystal was aligned with **a** approximately parallel to the ϕ axis of the diffractometer. The intensities were recorded by the $\omega-2\theta$ scan technique with a scan rate of 2° min^{-1} and 10 s stationary background counts. Three reference reflections were monitored every 50 reflections and showed no variation in intensity. Intensities were recorded to a 2θ limit of 125° for the triclinic crystal; of the 1932 non-equivalent terms measured, 1746 had values significantly greater than background and were considered observed. For the monoclinic crystal, of the 2587 non-equivalent terms recorded to a 2θ limit of 50° , 1701 were considered observed. The intensities were corrected for Lorentz and polarization factors, but absorption corrections were made only for the triclinic crystal. Scattering factors were from Cromer & Mann (1968) for C, N, O and Cl and from Stewart, Davidson & Simpson (1965) for H. Anomalous-dispersion corrections, f' and f'' , were made for all atoms (Cromer & Liberman, 1970).

Structure determination and refinement

Both structures were solved by direct methods with *SHELX 76* (Sheldrick, 1976). The $|E|$ values were calculated by the *K*-curve method of Karle, Hauptman & Christ (1958). For the triclinic structure phases for 415 $|E|$ terms >1.1 were derived, and from the resulting *E* map the sites of the C, N, O and Cl atoms were located. Least-squares refinement with isotropic temperature factors yielded $R = \sum (|F_o| -$

Table 3. Bond lengths (Å) and angles ($^\circ$) involving the non-hydrogen atoms, with *e.s.d.*'s in parentheses

	α Polymorph	β Polymorph
Cl(1)—C(3)	1.729 (7)	1.720 (7)
Cl(2)—C(5)	1.729 (7)	1.720 (8)
Cl(3)—C(6)	1.723 (6)	1.742 (7)
Cl(4)—C(3')	1.731 (6)	1.736 (7)
Cl(5)—C(5')	1.744 (7)	1.733 (7)
O(1)—C(2)	1.349 (8)	1.332 (9)
O(2)—C(7)	1.246 (9)	1.234 (9)
O(3)—C(2')	1.362 (9)	1.345 (8)
N—C(7)	1.334 (8)	1.357 (9)
N—C(1')	1.422 (8)	1.425 (9)
C(1)—C(2)	1.413 (8)	1.430 (10)
C(1)—C(6)	1.415 (9)	1.409 (10)
C(1)—C(7)	1.497 (9)	1.508 (10)
C(2)—C(3)	1.390 (9)	1.386 (10)
C(3)—C(4)	1.368 (10)	1.371 (10)
C(4)—C(5)	1.378 (9)	1.385 (11)
C(5)—C(6)	1.383 (9)	1.381 (10)
C(1')—C(2')	1.402 (9)	1.409 (10)
C(1')—C(6')	1.379 (9)	1.382 (10)
C(2')—C(3')	1.388 (9)	1.400 (10)
C(3')—C(4')	1.376 (9)	1.368 (10)
C(4')—C(5')	1.368 (9)	1.391 (11)
C(5')—C(6')	1.388 (9)	1.388 (10)
C(7)—N—C(1')	127.1 (6)	126.0 (6)
C(2)—C(1)—C(6)	117.1 (6)	117.2 (7)
C(2)—C(1)—C(7)	115.8 (6)	114.9 (6)
C(6)—C(1)—C(7)	127.2 (6)	127.9 (7)
O(1)—C(2)—C(1)	122.4 (6)	122.2 (7)
O(1)—C(2)—C(3)	117.4 (6)	118.0 (7)
C(1)—C(2)—C(3)	120.2 (6)	119.7 (7)
Cl(1)—C(3)—C(2)	118.8 (5)	118.5 (6)
Cl(1)—C(3)—C(4)	119.8 (5)	120.3 (6)
C(2)—C(3)—C(4)	121.4 (6)	121.2 (7)
C(3)—C(4)—C(5)	119.7 (6)	120.3 (8)
Cl(2)—C(5)—C(4)	118.2 (5)	117.5 (6)
Cl(2)—C(5)—C(6)	121.3 (5)	122.7 (6)
C(4)—C(5)—C(6)	120.5 (6)	119.8 (8)
Cl(3)—C(6)—C(1)	121.5 (5)	121.8 (6)
Cl(3)—C(6)—C(5)	117.3 (5)	116.7 (6)
C(1)—C(6)—C(5)	121.1 (6)	121.5 (7)
O(2)—C(7)—N	120.6 (6)	120.6 (7)
O(2)—C(7)—C(1)	119.1 (6)	120.1 (7)
N—C(7)—C(1)	120.2 (6)	119.2 (7)
N—C(1')—C(2')	115.3 (6)	114.1 (6)
N—C(1')—C(6')	124.2 (6)	124.8 (7)
C(2')—C(1')—C(6')	120.5 (6)	120.9 (7)
O(3)—C(2')—C(1')	115.5 (6)	116.5 (7)
O(3)—C(2')—C(3')	125.6 (6)	125.8 (7)
C(1')—C(2')—C(3')	118.9 (6)	117.7 (7)
Cl(4)—C(3')—C(2')	119.4 (5)	119.1 (6)
Cl(4)—C(3')—C(4')	119.4 (5)	118.7 (6)
C(2')—C(3')—C(4')	121.2 (6)	122.2 (7)
C(3')—C(4')—C(5')	118.6 (6)	118.5 (7)
Cl(5)—C(5')—C(6')	118.5 (5)	119.6 (6)
Cl(5)—C(5')—C(4')	119.0 (5)	118.7 (6)
C(4')—C(5')—C(6')	122.5 (6)	121.6 (7)
C(1')—C(6')—C(5')	118.3 (6)	119.0 (7)

$|F_c|/\sum |F_o| = 0.16$. Further full-matrix refinement with anisotropic temperature factors for the non-hydrogen and isotropic for the H atoms reduced *R* to

0.053 for the 1746 observed terms.* The coordinates for five H atoms were located by difference, but those for H(6') were calculated. For the monoclinic structure, phases for 235 $|E|$ terms > 1.2 were derived, and the resulting E map revealed the sites of all the non-hydrogen atoms. Subsequent refinement with isotropic temperature factors yielded $R = 0.16$. In the final least-squares refinement, anisotropic temperature factors were given to the C, N, O and Cl and isotropic to the H atoms, which had been located by difference. The atomic parameters were refined in three blocks, corresponding to positional and thermal parameters for nine, seven and eight atoms respectively. A final R of 0.060 was attained for the 1464 observed terms for which $|F_o| > 3\sigma|F_c|$.* In both analyses, the H atoms were given the same isotropic thermal parameters as the atoms to which they were bonded, and their positional coordinates were not refined.

The refinements were made with *SHELX 76* (Sheldrick, 1976), the function minimized being $\sum w|F_o| - |F_c||^2$, with $w = 1.0$. Final atomic coordinates are given in Table 2, bond lengths and angles in Tables 3 and 4. The atom numbering is illustrated in Fig. 1, prepared with *ORTEP* (Johnson, 1965). Fig. 2 shows projections of the structures down **a**. Some short intermolecular distances are listed in Table 5.

Description of the structure

Apart from the dihedral angle between the aromatic rings *A* and *B*, the molecules of the α and β forms have

* Lists of structure factors and anisotropic thermal parameters for both forms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34473 (36 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 4. Bond lengths (\AA) and angles ($^\circ$) involving the hydrogen atoms

	α Polymorph	β Polymorph
H(O1)—O(1)	1.02	0.86
H(O3)—O(3)	0.90	1.07
H(N)—N	0.89	0.95
H(4)—C(4)	0.97	0.83
H(4')—C(4')	0.98	1.06
H(6')—C(6')	1.08	1.03
C(2)—O(1)—H(O1)	115	98
C(2')—O(3)—H(O3)	104	112
C(7)—N—H(N)	113	109
C(1')—N—H(N)	119	114
C(3)—C(4)—H(4)	126	121
C(5)—C(4)—H(4)	114	113
C(3')—C(4')—H(4')	125	122
C(5')—C(4')—H(4')	116	116
C(1')—C(6')—H(6')	121	123
C(5')—C(6')—H(6')	121	118

similar conformations. In the α form the molecules are virtually planar, the angle between the two rings being only 2.5° , cf. the value 4.1° in DDS (Sindt & Mackay, 1978). However, in the β form the molecules are significantly distorted from planarity and assume a slight butterfly shape with the analogous dihedral angle 14.0° . The angles between the plane of the amide group and rings *A* and *B* in each structure are similar, 19.1 and 16.8° in the α form, and 20.1 and 19.0° in the β form. These values are much smaller than the 29.4 and 33.5° in DDS. Plane equations and perpendicular distances from the planes are given in Table 6.

The values for the bond lengths and angles in the two structures are in close agreement (Table 3), so that discussion of numerical values will refer to their mean

Table 5. Intermolecular approach distances $< 3.5 \text{ \AA}$

E.s.d.'s are 0.01 \AA .

(a) α Polymorph

C(7) ... C(7) ^I	3.42 \AA	O(2) ... O(2) ^I	3.37 \AA
C(2') ... C(5) ^{II}	3.50	O(3) ... O(1) ^{IV}	2.85
C(6') ... C(1) ^I	3.47	O(3) ... Cl(1) ^{IV}	3.39
O(1) ... C(3) ^{III}	3.39	Cl(1) ... O(1) ^{III}	3.49
O(1) ... C(2') ^I	3.49	Cl(3) ... Cl(1) ^{IV}	3.35
O(2) ... C(7) ^I	3.16	Cl(4) ... O(1) ^{IV}	3.18
O(2) ... C(1') ^I	3.42	Cl(4) ... O(2) ^{IV}	3.29
O(2) ... N ^I	3.12	Cl(5) ... C(5) ^I	3.46

Symmetry code

(I)	$1 - x, 1 - y, 1 - z$	(III)	$-x, -y, 1 - z$
(II)	$-x, 1 - y, 1 - z$	(IV)	$x, 1 + y, z$

(b) β Polymorph

C(1') ... O(1) ^I	3.43	O(3) ... N ^I	3.45
C(2') ... C(7) ^I	3.45	O(3) ... O(1) ^{III}	2.82
C(4') ... C(2) ^{II}	3.42	O(3) ... O(2) ^{III}	3.45
C(5') ... C(3) ^I	3.44	O(3) ... Cl(1) ^{III}	3.40
C(6') ... C(2) ^I	3.41	Cl(4) ... C(7) ^{II}	3.49
C(6') ... O(1) ^I	3.46	Cl(4) ... N ^I	3.46
O(2) ... O(1) ^I	3.34	Cl(4) ... O(1) ^{III}	3.15
O(3) ... C(1') ^I	3.43	Cl(4) ... O(2) ^{III}	3.24
O(3) ... C(6') ^I	3.23		

Symmetry code

(I)	$\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$	(III)	$x, y, 1 + z$
(II)	$-\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$		

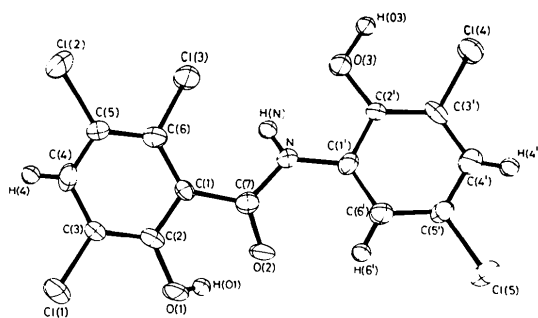


Fig. 1. A perspective view of a molecule of the β polymorph with thermal ellipsoids scaled to 50% probability.

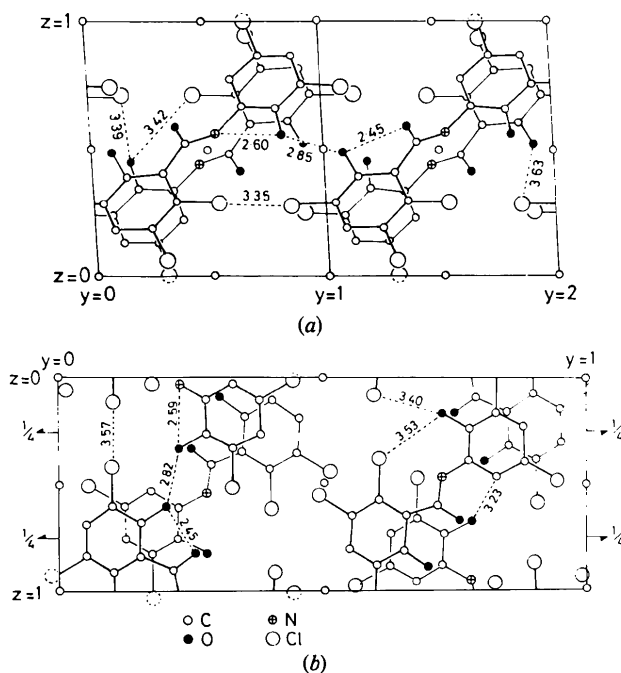


Fig. 2. Projections of (a) the α polymorph and (b) the β polymorph down a .

values. The dimensions of the amide group do not differ significantly from those in other structures (Koch & Germain, 1970; Sindt & Mackay, 1977). The aromatic C—C bonds have a mean length of 1.39 Å (mean deviation 0.01 Å) and the mean C—Cl length 1.73 Å (mean deviation 0.01 Å) agrees with other reported values (Rudman, 1971). The internal angles at positions of substitution by the Cl atoms, other than the internal angle where Cl(2) is substituted, are significantly greater than 120° and have a mean value of 121.6° (mean deviation 0.4°). Similar values have been noted in comparable structures for angles involving aromatic substitution by electron-withdrawing groups (Bellows, Stevens & Prasad, 1978; Hovmöller, Smith & Kennard, 1978). The internal angle at Cl(2) of 120.3° is close to the standard trigonal value, *cf.* 2,4,5-chloro-substituted compounds (Smith, Kennard & White, 1976; Smith, Kennard, White & Hodgson, 1977).

The hydrogen-bonding scheme in both crystals is similar (Table 7). There is an intramolecular hydrogen bond in which the hydroxyl O(1) of ring *A* acts as a donor to the carbonyl O(2), the O(1)···O(2) distance being 2.45 Å in each crystal, *cf.* the value 2.65 Å in DDS (Sindt & Mackay, 1978). There is also an intramolecular hydrogen bond in which the N atom acts as a donor to the hydroxyl O(3) attached to ring *B*, the N···O(3) distances being 2.59 and 2.60 Å. In addition, O(3) forms an intermolecular hydrogen bond with O(1) of an adjacent molecule. This interaction

Table 6. Equations of least-squares planes in which X , Y and Z are expressed in Å referred to orthogonal axes, and distances of the atoms from the planes (the *e.s.d.*'s are 0.01 Å)

Conversion from triclinic to orthogonal coordinates is given by

$$\begin{bmatrix} X \\ Y \\ Z \end{bmatrix} = \begin{bmatrix} a & b \cos \gamma & c \cos \beta \\ 0 & b \sin \gamma & cP \\ 0 & 0 & cQ \end{bmatrix} \begin{bmatrix} x/a \\ y/b \\ z/c \end{bmatrix},$$

where $P = (\cos \alpha - \cos \beta \cos \gamma) / \sin \gamma$ and $Q = (1 - \cos^2 \beta - P^2)^{1/2}$.

For the monoclinic structure, the orthogonal axes are parallel to a , b and c^* .

(a) α Polymorph

$$\text{Plane A: } 0.8343X + 0.2397Y - 0.4965Z + 1.4481 = 0$$

$$\text{Plane B: } 0.8237X + 0.2813Y - 0.4923Z + 1.7704 = 0$$

$$\text{Plane C: } 0.6536X + 0.5105Y - 0.5588Z + 1.1256 = 0$$

Plane A		Plane B		Plane C	
C(1)	-0.02 Å	C(1')	0.01 Å	C(1)	0.01 Å
C(2)	0.01	C(2')	0.00	C(7)	0.01
C(3)	0.00	C(3')	0.00	C(1')	0.02
C(4)	-0.01	C(4')	0.01	N	-0.03
C(5)	0.01	C(5')	0.00	O(2)	-0.01
C(6)	0.01	C(6')	-0.01		
†C(7)	-0.07	†N	0.02		
†O(1)	0.06	†O(3)	-0.03		
†Cl(1)	0.04	†Cl(4)	0.00		
†Cl(2)	0.05	†Cl(5)	0.01		
†Cl(3)	0.15				

(b) β Polymorph

$$\text{Plane A: } -0.9839X + 0.0953Y - 0.1511Z - 0.1691 = 0$$

$$\text{Plane B: } -0.9578X - 0.1212Y - 0.2606Z + 1.4881 = 0$$

$$\text{Plane C: } -0.8704X + 0.1091Y - 0.4801Z + 2.3333 = 0$$

Plane A		Plane B		Plane C	
C(1)	0.03 Å	C(1')	-0.01 Å	C(1)	0.03 Å
C(2)	-0.03	C(2')	0.02	C(7)	-0.03
C(3)	0.00	C(3')	-0.01	C(1')	0.03
C(4)	0.02	C(4')	0.00	N	-0.03
C(5)	-0.01	C(5')	0.01	O(2)	0.00
C(6)	-0.01	C(6')	0.00		
†C(7)	0.14	†N	0.06		
†O(1)	-0.06	†O(3)	0.05		
†Cl(1)	0.05	†Cl(4)	-0.07		
†Cl(2)	-0.06	†Cl(5)	0.03		
†Cl(3)	-0.10				

† These atoms were not included in the plane calculation.

Table 7. Hydrogen-bond dimensions

$X-H \cdots O$		$X \cdots O$	$H \cdots O$	$\angle X-H \cdots O$
N—H(N)···O(3)	(α)	2.60 Å	2.24 Å	104°
	(β)	2.59	2.21	103
O(1)—H(O1)···O(2)	(α)	2.45	1.62	136
	(β)	2.45	1.69	147
O(3)—H(O3)···O(1)	(α)	2.85	2.07	145
	(β)	2.82	1.82	156

links the molecules into ribbons along **b** for the α and along **c** for the β polymorph, the O(3)···O(1) distances being 2.85 and 2.82 Å respectively. The hydrogen-bond dimensions are consistent with expected values (Hamilton & Ibers, 1968).

The molecular packing is shown in Fig. 2. In the triclinic crystal, the molecules are oriented with their long molecular axes approximately parallel to the (011) planes, and rings *A* and *B* are inclined to the (100) planes at 33.5 and 34.5° respectively. In the monoclinic crystal, the molecules are oriented with their long molecular axes approximately parallel to the (021) planes, and rings *A* and *B* are inclined to the (100) planes at 10.3 and 16.7°.

As a consequence of the crystallographic symmetry, the molecules in both crystals are present as mirror-image conformers. This contrasts with DDS in which only one conformation is present. The stacking of the molecules in oxyclozanide also differs from that in DDS in which rings *A* and *B* of one molecule lie directly above rings *A* and *B* of an adjacent molecule. In both oxyclozanide structures, rings *A* and *B* are alternately stacked. In the triclinic structure, the benzene rings are stacked directly over one another, thus allowing interaction between overlapping π -electron systems. In the monoclinic structure, rings *A* and *B* of each molecule are stacked above rings *B* and *A* of two adjacent molecules to create a two-dimensional interlocking effect. However, in this case, the overlap of the benzene rings is only slight.

The respective stacking of molecules in the α and β forms does not allow a phase transformation *via* a shear process in the solid state. Thus, the crystallographic results support the hypothesis of Pearson & Varney (1969) that a phase change is only made possible by rearrangement of the molecules through selective dissolution in the presence of a suitable solvent. The greater interlocking of molecules in the β relative to that in the α polymorph could account for the lower solubility of the former.

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