

silyl group is not a good acceptor and the oxygen atom in disiloxane is a poor donor towards such acceptors as trimethylboron. If we equate the $O \cdots Si(2)$ interaction with the incipient S_N2 reaction situation, as has been suggested for other crystalline state intermolecular interactions (Bürgi, 1975), then we expect distortions to the intramolecular geometry at Si(2), but not at Si(1). Specifically, the Si(2)—O bond is expected to lengthen and the O—Si(2)—H angles to narrow. We do not observe these distortions: the Si(2)—O and Si(1)—O bonds are of equal length, and although $\angle O-Si(2)-H$, 102° (average), is less than $\angle O-Si(1)-H$, 108° (average), the difference is within experimental error. The situation here is in contrast to that for crystalline silyl isocyanate (Barrow, Cradock, Ebsworth & Harding, 1977) where a lengthening of the Si—N bond compared with the gas-phase ED value apparently correlates well with the presence of a weak axial $N \cdots Si$ interaction in the solid state. We assume that in crystalline disiloxane the $O \cdots Si(2)$ interaction is strong enough to effect an alignment of the molecules but too weak to affect the intramolecular geometry by any significant extent.

In crystalline hexamethyldisiloxane the molecules exist as discrete entities and there are no specific intermolecular contacts involving Si or O atoms. This is in keeping with the general observation that methylation at silicon sharply reduces the σ -acceptor character of the silicon atom. Here also the six methyl groups appear to shield the Si—O—Si core from neighbouring molecules.

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The Crystal and Molecular Structure of 2,4-Di-*tert*-butyl-6-methyl-*N*-thiosulphinylaniline

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Abstract

The crystal and molecular structure of 2,4-di-*tert*-butyl-6-methyl-*N*-thiosulphinylaniline has been determined by X-ray diffraction. Crystals are monoclinic, the space group is $P2_1/a$ with cell dimensions: $a = 19.050$ (6), $b = 9.614$ (2), $c = 10.084$ (3) Å, $\beta = 0567-7408/79/092099-05\01.00

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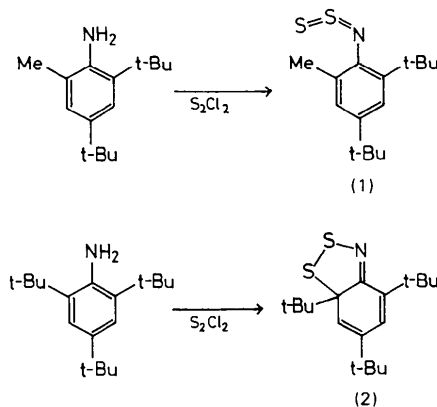
118.25 (3) $^\circ$, and $Z = 4$. The structure was solved by the heavy-atom method and refined by the block-diagonal least-squares method. The final R value was 0.057. The molecule has a $-N=S=S$ group. The $S=S$ and $N=S$ double-bond distances are 1.898 (2) and 1.548 (3) Å, respectively, and $C-N=S$ and $N=S=S$ bond angles are 126.4 (3) and 119.7 (1) $^\circ$, respectively.

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tively. The terminal S atom is *cis* to C across the N=S bond with the torsion angle of $4.7(4)^\circ$. The dihedral angle between the planes of the benzene ring and *N*-thiosulphinyl group is $68(3)^\circ$.

Introduction

Reaction of 2,4-di-*tert*-butyl-6-methylaniline with sulphur monochloride gave the corresponding *N*-thiosulphinylaniline (1). However, similar reactions with 2,4,6-tri-*tert*-butylaniline gave a cyclic 5*H*-1,2,3-dithiazole compound (2) (Inagaki, Okazaki & Inamoto, 1975). The determination of the structures of these compounds is of interest in order to reveal the nature of the bonding of the S atom and to facilitate the understanding of their reactions. In the present paper, the result of an X-ray study of 2,4-di-*tert*-butyl-6-methyl-*N*-thiosulphinylaniline is reported.



Experimental

The crystals were grown from an ethanol solution. They are reddish-black flat plates. Since the crystals decomposed very slowly, X-ray data were obtained with the specimen coated with a thin film of Araldite.

Crystal data

C₁₅H₂₃NS₂, $M_r = 281.47$; monoclinic, $P2_1/a$, $a = 19.050(6)$, $b = 9.614(2)$, $c = 10.084(3)$ Å, $\beta = 118.25(3)^\circ$, $U = 1629.9$ Å³, $Z = 4$, $D_x = 1.149$ Mg m⁻³.

Intensity data were collected on a Rigaku automatic diffractometer using graphite-monochromatized Mo $K\alpha$ radiation. A crystal with approximate dimensions $0.3 \times 0.1 \times 0.3$ mm was used. The reflexions within the range $2\theta \leq 55^\circ$ were measured by the ω - 2θ scan technique with a scan width of $\Delta\omega = 1.1^\circ + 0.5^\circ \tan \theta$ and with a scanning speed of 4° min^{-1} in 2θ . At both ends of the scan range 10 s background counts were taken for each reflexion. Three standard reflexions,

measured after every 50 reflexions, showed no decrease in intensity during the data collection. 2049 reflexions had $|F_o| \geq 3\sigma(F_o)$ and were considered as observed. No absorption corrections were applied.

Structure determination

A sharpened three-dimensional Patterson synthesis was calculated, from which the coordinates of the S atoms could be determined. A Fourier synthesis then made it possible to locate all the non-hydrogen atoms. The structure was refined using the block-diagonal least-

Table 1. Atomic parameters with their estimated standard deviations

Atomic coordinates are $\times 10^4$ for the non-hydrogen atoms and $\times 10^3$ for H. The B_{eq} values are the equivalent isotropic temperature factors.

	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq} (Å ²)
S(1)	3886 (0)	746 (1)	3670 (1)	5.2
S(2)	4079 (1)	917 (2)	1997 (2)	7.5
N	3022 (1)	838 (3)	3430 (3)	4.2
C(1)	2318 (1)	948 (4)	2026 (3)	3.3
C(2)	1826 (2)	2130 (4)	1726 (4)	3.0
C(3)	1134 (2)	2160 (4)	349 (4)	3.3
C(4)	899 (2)	1062 (4)	-672 (4)	3.3
C(5)	1379 (2)	-102 (4)	-306 (4)	3.9
C(6)	2093 (2)	-181 (4)	1039 (4)	3.8
C(21)	2031 (2)	3352 (4)	2839 (4)	3.9
C(22)	2795 (2)	4094 (5)	3033 (5)	5.4
C(23)	2155 (2)	2837 (5)	4380 (4)	5.5
C(24)	1361 (2)	4451 (5)	2296 (5)	5.8
C(41)	130 (2)	1149 (4)	-2207 (4)	3.8
C(42)	-452 (2)	2239 (6)	-2167 (5)	8.4
C(43)	-301 (2)	-258 (5)	-2604 (5)	7.2
C(44)	359 (2)	1560 (5)	-3412 (4)	6.2
C(61)	2569 (2)	-1531 (5)	1415 (5)	5.3

	<i>x</i>	<i>y</i>	<i>z</i>	B (Å ²)
H(3)	78 (2)	299 (3)	12 (3)	4.5 (7)
H(5)	128 (2)	-97 (4)	-100 (4)	6.0 (9)
H(21)	261 (2)	448 (4)	203 (4)	7.2 (10)
H(22)	332 (2)	352 (4)	348 (4)	6.3 (9)
H(23)	298 (2)	485 (4)	382 (4)	6.2 (9)
H(24)	262 (2)	222 (4)	486 (4)	7.1 (9)
H(25)	222 (2)	366 (4)	498 (4)	6.7 (9)
H(26)	169 (2)	236 (4)	426 (4)	6.2 (9)
H(27)	155 (2)	521 (4)	310 (4)	7.8 (10)
H(28)	80 (2)	406 (4)	188 (4)	7.2 (10)
H(29)	137 (2)	486 (4)	134 (4)	6.6 (9)
H(41)	-60 (2)	171 (4)	-139 (4)	7.2 (10)
H(42)	-95 (2)	222 (4)	-324 (4)	7.3 (10)
H(43)	-19 (2)	312 (4)	-223 (4)	8.3 (10)
H(44)	-33 (2)	-29 (4)	-166 (4)	8.8 (11)
H(45)	-81 (2)	-15 (4)	-365 (4)	6.6 (9)
H(46)	4 (1)	-80 (3)	-295 (3)	4.6 (7)
H(47)	-9 (2)	148 (4)	-463 (4)	7.6 (10)
H(48)	68 (2)	78 (4)	-344 (4)	7.9 (10)
H(49)	44 (2)	251 (4)	-310 (4)	8.7 (11)
H(61)	222 (2)	-223 (4)	83 (4)	6.8 (9)
H(62)	294 (2)	-128 (4)	114 (4)	7.2 (10)
H(63)	281 (2)	-178 (4)	252 (4)	7.0 (9)

squares method. When the R value was 0.11, a difference synthesis was calculated, which revealed the positions of all H atoms. All atoms were refined with the block-diagonal least-squares method using anisotropic temperature factors for non-hydrogen atoms and isotropic for H. The quantity minimized was $\sum w(|F_o| - k^{-1}|F_c|)^2$ where $w = 1/\sigma(F_o)$, as derived from counting statistics. The final R value was 0.057 for all the observed reflexions. The weighted R value $\{[\sum w(|F_o| - k^{-1}|F_c|)^2]/\sum wF_o^2\}^{1/2}$ was 0.044. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The final atomic parameters and their estimated standard deviations are given in Table 1.*

Results and discussion

Fig. 1 shows a stereoscopic view of the molecule. The bond lengths and angles with their estimated standard deviations are listed in Table 2.

The structure has been determined unequivocally as *N*-thiosulphonylaniline. The S(1)–S(2) length is 1.898 Å, which is significantly shorter than the normal S–S single-bond length (2.08 Å). Very few S=S double-bond distances have been determined; S=S is reported to be 1.89 Å in S₂ (Abrahams, 1956). The N–S(1) length is 1.548 Å. The length of the N=S double bond estimated from the sum of the covalent radii is 1.56 Å (Pauling, 1960). A similar –N=S=S group has recently been found in 7,7,9,9-tetramethyl-8-thio-sulphinyliminothio-1,4-dioxo-8-azaspiro[4.5]decane (compound 3 in Table 3), in which S=S and S=N are 1.912 and 1.569 Å, respectively (Tamura, Aiba, Sato, Hata, Morimura & Yoshioka, 1977). However, the lengths of the S–N and S–S bonds in (2) are 1.656 and 2.084 Å, respectively (Iwasaki, 1979*a*). Some short S^{IV}=N distances have been reported in structures

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34153 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

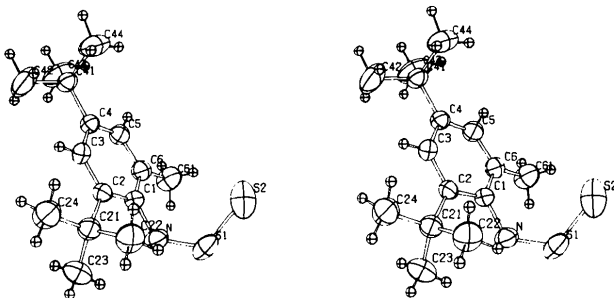


Fig. 1. Stereoscopic view of the molecule. Thermal ellipsoids of non-hydrogen atoms are at a level of 50% probability (ORTEP; Johnson, 1965).

Table 2. Bond lengths (Å) and angles (°) with their estimated standard deviations

S(1)–S(2)	1.898 (2)	C(3)–H(3)	0.99 (3)
S(1)–N	1.548 (3)	C(5)–H(5)	1.05 (4)
N–C(1)	1.420 (5)	C(22)–H(21)	0.97 (4)
C(1)–C(2)	1.411 (5)	C(22)–H(22)	1.05 (4)
C(2)–C(3)	1.391 (5)	C(22)–H(23)	1.01 (4)
C(3)–C(4)	1.393 (5)	C(23)–H(24)	0.98 (4)
C(4)–C(5)	1.380 (5)	C(23)–H(25)	0.97 (4)
C(5)–C(6)	1.396 (5)	C(23)–H(26)	0.95 (4)
C(1)–C(6)	1.396 (5)	C(24)–H(27)	1.02 (4)
C(2)–C(21)	1.541 (5)	C(24)–H(28)	1.01 (4)
C(21)–C(22)	1.548 (6)	C(24)–H(29)	1.05 (4)
C(21)–C(23)	1.543 (6)	C(42)–H(41)	1.08 (4)
C(21)–C(24)	1.545 (6)	C(42)–H(42)	1.05 (4)
C(4)–C(41)	1.551 (5)	C(42)–H(43)	1.00 (4)
C(41)–C(42)	1.539 (7)	C(43)–H(44)	0.98 (4)
C(41)–C(43)	1.533 (6)	C(43)–H(45)	1.05 (4)
C(41)–C(44)	1.525 (6)	C(43)–H(46)	1.01 (4)
C(6)–C(61)	1.525 (6)	C(44)–H(47)	1.12 (4)
		C(44)–H(48)	0.97 (4)
		C(44)–H(49)	0.96 (4)
		C(61)–H(61)	0.93 (4)
		C(61)–H(62)	0.91 (4)
		C(61)–H(63)	1.01 (4)
S(2)S(1)N	119.7 (1)	C(2)C(3)H(3)	117 (2)
S(1)NC(1)	126.4 (3)	C(4)C(3)H(3)	120 (2)
NC(1)C(2)	119.5 (3)	C(4)C(5)H(5)	125 (2)
NC(1)C(6)	118.6 (3)	C(6)C(5)H(5)	113 (2)
C(2)C(1)C(6)	121.6 (4)	C(6)C(61)H(61)	107 (3)
C(1)C(2)C(3)	116.8 (3)	C(6)C(61)H(62)	99 (3)
C(2)C(3)C(4)	122.9 (3)	C(6)C(61)H(63)	112 (2)
C(3)C(4)C(5)	118.5 (3)	H(61)C(61)H(62)	114 (4)
C(4)C(5)C(6)	121.4 (3)	H(61)C(61)H(63)	110 (3)
C(1)C(6)C(5)	118.7 (4)	H(62)C(61)H(63)	113 (3)
C(1)C(2)C(21)	122.9 (3)	C(21)C(22)H(21)	102 (3)
C(2)C(2)C(21)	120.3 (3)	C(21)C(22)H(22)	118 (2)
C(3)C(4)C(41)	121.2 (3)	C(21)C(22)H(23)	113 (2)
C(5)C(4)C(41)	120.3 (3)	C(21)C(23)H(24)	111 (3)
C(1)C(6)C(61)	122.6 (4)	C(21)C(23)H(25)	106 (2)
C(5)C(6)C(61)	118.6 (4)	C(21)C(23)H(26)	110 (2)
C(2)C(21)C(22)	110.0 (3)	C(21)C(24)H(27)	107 (2)
C(2)C(21)C(23)	110.8 (3)	C(21)C(24)H(28)	115 (2)
C(2)C(21)C(24)	112.4 (3)	C(21)C(24)H(29)	102 (2)
C(22)C(21)C(23)	109.6 (3)	C(41)C(42)H(41)	98 (2)
C(22)C(21)C(24)	107.5 (3)	C(41)C(42)H(42)	106 (2)
C(23)C(21)C(24)	106.5 (3)	C(41)C(42)H(43)	101 (2)
C(4)C(41)C(42)	111.3 (3)	C(41)C(43)H(44)	92 (2)
C(4)C(41)C(43)	110.4 (3)	C(41)C(43)H(45)	108 (2)
C(4)C(41)C(44)	108.5 (3)	C(41)C(43)H(46)	102 (2)
C(42)C(41)C(43)	107.7 (4)	C(41)C(44)H(47)	120 (2)
C(42)C(41)C(44)	109.1 (4)	C(41)C(44)H(48)	104 (2)
C(43)C(41)C(44)	109.9 (4)	C(41)C(44)H(49)	93 (3)
		Mean value	
		HC _{tert} -Bu-H	113 (3)








involving N=S=N and S=N=S groups, as shown in Table 3. The bond angles C(1)NS(1) and NS(1)S(2) are 126.4 and 119.7°, respectively. The angle of NS(1)S(2) is larger than that in (3) (114.9°). It can be seen from Table 3 that short N=S bond lengths are related to large N=S=X (X = N, S, O) angles. The value of the angle C(1)NS(1) is similar to the corresponding angles in bis(tolylimino)sulphur (6) (130.8°;

Leandri, Buseti, Valle & Mammi, 1970) and 2,4,6-*tert*-butylsulphinylaniline (4) (131.1°; Iwasaki, 1979b).

The torsion angles of some bonds in the molecule are shown in Fig. 2. S(2) is *cis* to C(1) across the S(1)—N bond with a torsion angle of 4.7 (4)°. The dihedral angle between the plane of C(1)—N—S(1)—S(2) and that of the benzene ring is 68 (3)°, and the S=S group is inclined towards the methyl group in spite of the bulky *tert*-butyl group. The short intramolecular non-bonding distances are S(1)⋯C(61) 3.295 (5), S(2)⋯N 2.985 (4), N⋯C(61) 2.898 (6), S(1)⋯H(22) 2.84 (4), S(2)⋯H(62) 2.85 (4) and N⋯H(24) 2.34 (4) Å.

The C(1)—N distance is 1.420 Å, which is shorter than a C—N single bond but is similar to a normal C(*sp*²)—N(*sp*²) bond. The average C—C length in the benzene ring is 1.395 Å. C(1)—C(2) (1.411 Å) is, however, significantly longer than the other bonds on account of the bulky *ortho* substituents. Deviations from the least-squares planes are listed in Table 4. The planarity of the benzene ring is not perfect. Maximum deviations occur at C(1) and C(2), owing to the

Table 3. Comparison of S^{IV}=N bonds

Bond type	S=N lengths (Å)		α (°)	Compound	Reference
	1.548		119.9	1	Present work
	1.569		114.9	3	Tamura <i>et al.</i> (1977)
	1.494		121.9	4	Iwasaki (1979b)
	1.560		113.6	5	Gieren & Dederer (1977)
	1.53	1.56	117.2	6	Leandri <i>et al.</i> (1970)
	1.533	1.532	119.3	7	Gieren, Dederer, Roesky & Janssen (1976)
	1.535	1.534	118.5	8	Gieren & Pertlik (1976)
	1.567	1.564	115.0	9	Weiss (1977)
	1.57	1.53	113	10	Kops, van Aken & Schenk (1973)
	1.532	1.537	149.2	11	Glemser, Krebs, Wegener & Kindler (1969)

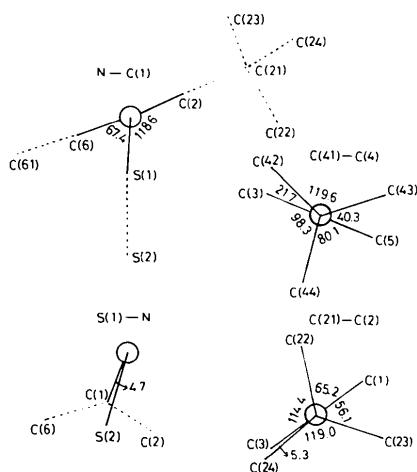
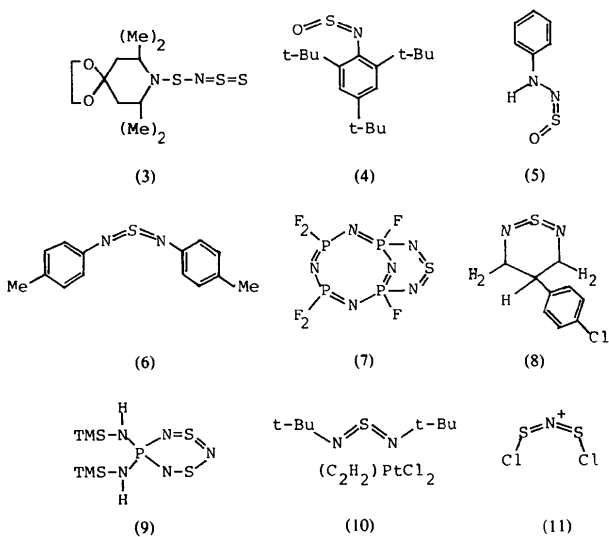


Fig. 2. Torsion angles (°) in the molecule. The e.s.d.'s are within the range 0.3–0.5°.

Table 4. Least-squares planes

Atom deviations (Å) are given from the least-squares planes expressed in the form $AX + BY + CZ + D = 0$, where X , Y and Z are the coordinates in Å referred to the a , b and c^* axes respectively. The e.s.d.'s of deviations are 0.020 Å for plane 1 and 0.033 Å for plane 2.

(1) Benzene ring $-0.795X - 0.413Y + 0.445Z + 2.295 = 0$
 (2) C(1)NS(1)S(2) $-0.016X - 0.994Y - 0.105Z + 1.165 = 0$

	C(1)	C(2)	C(3)	C(4)	C(5)	C(6)
Plane 1	-0.020	0.022	-0.008	-0.008	0.011	0.004
Plane 2	0.013	-1.076	-0.965	0.179	1.246	1.184
	N	S(1)	S(2)	C(21)	C(41)	C(61)
Plane 1	0.045	-1.040	-2.698	0.088	-0.067	0.110
Plane 2	-0.022	0.018	-0.008			

Angles (°) between interatomic vectors and plane 1; e.s.d.'s are 1.0–1.4°.

C(1)—N	2.6	C(2)—C(21)	2.5	C(4)—C(41)	2.2
C(6)—C(61)	4.0	N—S(1)	44.5	S(1)—S(2)	60.9

repulsion between the two bulky substituents. The deviations of the exocyclic atoms N, C(21), C(41) and C(61) from the ring plane are within 0.11 Å.

The lengths of C(2)—C(21) and C(4)—C(41) are 1.541 and 1.551 Å, respectively. These values are normal for $C_{ar}-C_{tert-Bu}$ lengths but slightly longer than the normal C(*sp*²)—C(*sp*³) length. In 3,5-di-*tert*-butylbenzoic acid the $C_{ar}-C_{tert-Bu}$ lengths are 1.53 Å (Florenco, Garcia-Blanco & Smith-Verdier, 1976). The length of C(6)—C(61) ($C_{ar}-C_{Me}$) is 1.525 Å. The endocyclic bond angles opposite the *tert*-butyl groups are 116.8 and 118.5° and that opposite the methyl group is 118.7°. Similar values have been found in analogous compounds bearing alkyl substituents (Domenicano, Vaciago & Coulson, 1975). In the *tert*-butyl groups the C—CH₃ distances are within the range

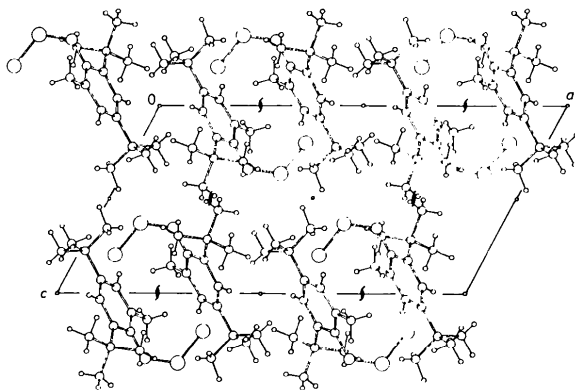


Fig. 3. Projection of the structure along the *b* axis.

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).