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The Crystal Structure of the Monoclinic Phase of 2,2-Di-t-butyl-3,3-diphenylthiirane at –140°C

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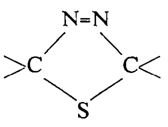
The title compound, $C_{22}H_{28}S$, is a stable intermediate in the synthesis of 1,1-di-t-butyl-2,2-diphenylethylene. The substance crystallizes at room temperature in an orthorhombic phase [space group $P2_12_12_1$, $a=9{\cdot}098$ (2), $b=12{\cdot}791$ (2), $c=16{\cdot}616$ (2) Å, $Z=4$, $D_c=1{\cdot}115$ g cm^{-3}] and in a monoclinic phase [space group $P2_1/c$, $a=8{\cdot}910$ (2), $b=16{\cdot}051$ (3), $c=13{\cdot}169$ (2) Å, $\beta=100{\cdot}05$ (2)°, $Z=4$, $D_c=1{\cdot}162$ g cm^{-3}; crystal data measured at -140°C]. The density measured at room temperature over a mixture of both forms is $D_m=1{\cdot}121$ g cm^{-3}. Intensities were collected on a four-circle diffractometer. The orthorhombic and monoclinic structures were solved by Patterson and direct methods from room- and low-temperature X-ray data respectively, and refined by full-matrix least squares. Emphasis is given to the monoclinic structure ($T=-140$°C, $R=0{\cdot}048$). The overcrowding in the molecule is reflected in long bond distances from the central ring to the t-butyl groups (average 1·59 Å) and in tight intramolecular contacts, the shortest 1,5 distances being $C_{\text{t-butyl}}\ldots C_{\text{t-butyl}}=3{\cdot}03$, $C_{\text{t-butyl}}\ldots C_{\text{phenyl}}=2{\cdot}83$, $C_{\text{phenyl}}\ldots C_{\text{phenyl}}=3{\cdot}18$ Å. In the thiirane ring the C–S lengths are 1·82 and 1·85 Å; the central C–C distance is 1·55 Å. Geometric parameters of the molecule in the orthorhombic phase (incomplete refinement, probable partial disorder, $R=0{\cdot}099$) are generally in good agreement with those of the monoclinic phase.

Introduction

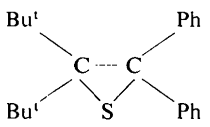
The extrusion of S and N from Δ^3-1,3,4-thiadiazolines (I), leading to unsaturated compounds (II), has been recently applied in the synthesis of some very hindered olefins (Barton, Guziek & Shahak, 1974).



(I)

$>C{=}C<$

(II)



(III)

During the synthesis of 1,1-di-t-butyl-2,2-diphenylethylene a first step of the extrusion procedure led to a thiirane derivative (III). The compound is of interest because of the molecular overcrowding and its effect on geometrical parameters.

Experimental

Crystals were obtained by slow evaporation from a solution in n-propyl alcohol. During preliminary photographic and counter work, two crystal phases were recognized, one monoclinic, space group $P2_1/c$, m.p. 102°C,* and one orthorhombic, space group $P2_12_12_1$, m.p. 114°C. The monoclinic phase was chosen for low-temperature data collection. In the Appendix a short account of the room-temperature structure of the orthorhombic phase is given.

A monoclinic crystal was ground to a sphere of diameter 0·3 mm and centred on a Syntex $P\bar{1}$ diffractometer equipped with graphite monochromator in the incident beam and LT1 low-temperature attachment, with the outlet nozzle fixed to the χ arc directly opposite the goniometer head along the φ axis. Cold nitrogen was allowed to flow at constant rate in order to maintain the temperature of the sample at -140°C. The temperature was measured by a thermocouple placed at the optical centre of the goniostat before the crystal was mounted. Besides the check reflexions, the orientation matrix and lattice parameters were periodically monitored during data collection.

Systematic extinctions ($0k0$ for k odd, $h0l$ for l odd) confirmed the space group $P2_1/c$. Lattice constants were obtained from a least-squares fit of 20 reflexions (plus some of their equivalents) centred on the diffractometer with Mo $K\alpha$ radiation ($\lambda=0{\cdot}71069$ Å). The density was measured at room temperature by flotation in a dilute Thoulet (K_2HgI_4) solution. The crystallographic data are given in the abstract.

Low-temperature intensities were collected with monochromatized Mo $K\alpha$ radiation by the ω-scan technique, total ω scan range 1°, variable scan speed between 2 and 12° min^{-1}, and dead time for coincidence correction $2{\cdot}1\times10^{-6}$ s. The background was counted for half the total scanning time 1° on each side of the $K\alpha$ position. After every group of 50 reflexions two standard reflexions were monitored; no significant change in their intensities was observed. In the range explored ($2\theta_{\max}=55°$) 4060 independent reflexions were measured, of which 3769 had intensities above background. The variance $\sigma^2(I_{\rm rel})$ was calculated as $[S+B+(0{\cdot}02\ S)^2]v^2$, with S=scan count, B=total background count, and v=scan speed. Lorentz and polarization factors were applied; absorption effects were neglected ($\mu=1{\cdot}7$ cm^{-1} for Mo $K\alpha$ radiation). The

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* By recrystallization from the melt and subsequent slow heating, the melting point rose to 114°C, indicating the orthorhombic phase as the more stable at these temperatures.

$N(z)$ test (Howells, Phillips & Rogers, 1950) was in agreement with a centric distribution.

Structure determination and refinement

The structure was solved by direct methods with the programs written by Bianchi, Destro & Gavezzotti (1972). The three reflexions with largest E values were apt to define the origin; the starting set was completed by four symbolic phases. The symbolic addition procedure led to a series of relationships among symbols and known phases with a high degree of internal consistency, and allowed the derivation of phases for the four symbols. In the following step, the 46 reflexions with largest E values and the assigned phases were used as the starting set in the tangent formula phase extension procedure. The resulting 684 phased reflexions ($|E|_{\min}=1{\cdot}40$) led to an E map which showed all the non-hydrogen atoms.

Refinement on all 3769 observed reflexions was by full-matrix least squares, minimizing $\sum w(\Delta F)^2$ with weights $w=1/\sigma^2(F)$. The program used was a modified version of *ORFLS* (Busing, Martin & Levy, 1962). Scattering factors for C and S were taken from Cromer & Waber (1965), and for H from Stewart, Davidson & Simpson (1965). Initial coordinates of the phenyl H atoms were derived from geometrical considerations and introduced in structure factor calculations with isotropic thermal factors $B_H=2$ Å^2. After anisotropic refinement of C and S atoms, a difference map showed the positions of the methyl H atoms. All the H atom parameters were then refined isotropically; because of computer-core size limitation, H and heavy atoms were refined in separate cycles. Refinement was considered complete when the parameter shifts were less than $0{\cdot}2\sigma$ (most shifts being less than $0{\cdot}1\sigma$) for heavy atoms, and less than $0{\cdot}3\sigma$ for H. The final value of R for all 3769 observed reflexions was 0·048, and for all 4060 measured reflexions 0·056.* A final difference map calculated with all the observed reflexions showed no significant features.

The final atomic parameters are given in Tables 1 and 2. The standard deviations of the coordinates, derived from the residuals and the diagonal elements of the least-squares inverse matrix, correspond to positional uncertainties of less than 0·001 Å for S, of about 0·002 Å for C and 0·02 Å for the H atoms. The corresponding standard deviations in the C–S and C–C bond distances are around 0·002 and 0·003 Å respectively, those in bond angles involving the S atom about 0·09°, and for bond and torsion angles involving only C atoms they are 0·15 and 0·30° respectively; in the C–H bonds and in the C–C–H and H–C–H angles they are around 0·02 Å and 1·15 and 1·60°, respectively. The numbering of atoms is shown on the thermal ellipsoid plot (Johnson, 1965) of Fig. 1.

Results and discussion

The molecular parameters involving heavier atoms are collected in Tables 3, 4 and 5. All these values are uncorrected for librational effects. Signs for the torsion angles follow the convention of Klyne & Prelog (1960). The C–H distances are in the range 0·94–1·02 Å.

Table 1. *The heavy-atom parameters of the monoclinic phase at* −140°C, *and their standard deviations*

All values in this table have been multiplied by 10^4. Thermal factors are of the form $\exp[-(B_{11}h^2+2B_{12}hk+\ldots)]$.

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
S	6392 (0)	2125 (0)	10016 (0)	95 (1)	17 (0)	16 (0)	6 (0)	9 (0)	1 (0)
C(1)	6212 (1)	1771 (1)	8685 (1)	62 (2)	16 (1)	16 (1)	5 (1)	9 (1)	2 (1)
C(2)	4643 (1)	1557 (1)	8095 (1)	57 (2)	15 (1)	25 (1)	8 (1)	8 (1)	1 (1)
C(3)	3588 (2)	1144 (1)	8571 (1)	67 (2)	22 (1)	28 (1)	7 (1)	10 (1)	3 (1)
C(4)	2197 (2)	895 (1)	8012 (1)	67 (2)	28 (1)	44 (1)	−3 (1)	17 (1)	2 (1)
C(5)	1848 (2)	1049 (1)	6963 (1)	56 (2)	35 (1)	44 (1)	1 (1)	−4 (1)	−2 (1)
C(6)	2899 (2)	1446 (1)	6474 (1)	82 (2)	34 (1)	27 (1)	7 (1)	−2 (1)	2 (1)
C(7)	4289 (2)	1694 (1)	7033 (1)	62 (2)	24 (1)	27 (1)	4 (1)	8 (1)	3 (1)
C(8)	7178 (1)	1002 (1)	8542 (1)	45 (2)	14 (1)	28 (1)	−3 (1)	3 (1)	−2 (1)
C(9)	7509 (2)	793 (1)	7576 (1)	56 (2)	20 (1)	31 (1)	−6 (1)	8 (1)	−4 (1)
C(10)	8283 (2)	63 (1)	7440 (1)	63 (2)	24 (1)	48 (1)	−7 (1)	18 (1)	−14 (1)
C(11)	8698 (2)	−482 (1)	8250 (1)	65 (2)	18 (1)	72 (1)	6 (1)	8 (1)	−10 (1)
C(12)	8313 (2)	−304 (1)	9195 (1)	74 (2)	17 (1)	53 (1)	5 (1)	−7 (1)	2 (1)
C(13)	7560 (2)	430 (1)	9340 (1)	68 (2)	18 (1)	32 (1)	1 (1)	2 (1)	0 (1)
C(14)	6804 (2)	2676 (1)	8864 (1)	82 (2)	14 (1)	16 (1)	4 (1)	5 (1)	0 (1)
C(15)	8554 (2)	2830 (1)	8827 (1)	78 (2)	16 (1)	28 (1)	−4 (1)	−1 (1)	−1 (1)
C(16)	8834 (2)	2745 (1)	7710 (1)	89 (2)	24 (1)	39 (1)	−12 (1)	18 (1)	0 (1)
C(17)	9171 (2)	3694 (1)	9210 (1)	125 (3)	20 (1)	46 (1)	−11 (1)	−11 (1)	0 (1)
C(18)	9634 (2)	2223 (1)	9504 (1)	74 (2)	23 (1)	50 (1)	−3 (1)	−8 (1)	5 (1)
C(19)	5648 (2)	3438 (1)	8600 (1)	104 (2)	15 (1)	27 (1)	11 (1)	12 (1)	1 (1)
C(20)	3950 (2)	3219 (1)	8545 (1)	100 (2)	23 (1)	42 (1)	23 (1)	19 (1)	3 (1)
C(21)	5937 (2)	4095 (1)	9473 (1)	168 (3)	20 (1)	39 (1)	14 (1)	22 (1)	−5 (1)
C(22)	5774 (2)	3860 (1)	7569 (1)	115 (3)	20 (1)	33 (1)	14 (1)	9 (1)	7 (1)

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* A table of observed and calculated structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31583 (31 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 2. *Parameters for the hydrogen atoms of the monoclinic phase at* −140°C

	x	y	z	B (Å^2)
H(1)	0·381	0·103	0·931	2·7
H(2)	0·146	0·062	0·836	3·5
H(3)	0·084	0·089	0·658	4·3
H(4)	0·266	0·157	0·576	3·9
H(5)	0·498	0·200	0·670	3·0
H(6)	0·713	0·116	0·699	3·1
H(7)	0·853	−0·005	0·678	3·6
H(8)	0·921	−0·098	0·815	4·5
H(9)	0·858	−0·068	0·977	3·8
H(10)	0·731	0·056	1·000	3·4
H(11)	0·992	0·279	0·770	4·7
H(12)	0·853	0·222	0·742	6·0
H(13)	0·833	0·318	0·725	5·4
H(14)	0·872	0·415	0·878	3·9
H(15)	1·027	0·371	0·913	4·6
H(16)	0·906	0·377	0·992	3·8
H(17)	1·070	0·235	0·942	3·3
H(18)	0·950	0·229	1·023	4·5
H(19)	0·947	0·163	0·931	4·8
H(20)	0·349	0·296	0·787	3·4
H(21)	0·376	0·287	0·913	3·5
H(22)	0·339	0·374	0·858	3·7
H(23)	0·698	0·437	0·955	5·5
H(24)	0·574	0·387	1·014	4·9
H(25)	0·519	0·453	0·933	5·7
H(26)	0·502	0·429	0·743	4·0
H(27)	0·679	0·412	0·758	4·0
H(28)	0·558	0·345	0·697	3·9

Table 3. *Bond distances* (Å)

S——C(1)	1·822	C(10)-C(11)	1·379
S——C(14)	1·848	C(11)-C(12)	1·377
C(1)-C(14)	1·550	C(12)-C(13)	1·386
C(1)-C(2)	1·517	C(13)-C(8)	1·392
C(2)-C(3)	1·387	C(14)-C(15)	1·587
C(3)-C(4)	1·385	C(15)-C(16)	1·541
C(4)-C(5)	1·385	C(15)-C(17)	1·544
C(5)-C(6)	1·381	C(15)-C(18)	1·540
C(6)-C(7)	1·384	C(14)-C(19)	1·598
C(7)-C(2)	1·397	C(19)-C(20)	1·543
C(1)-C(8)	1·535	C(19)-C(21)	1·549
C(8)-C(9)	1·396	C(19)-C(22)	1·539
C(9)-C(10)	1·386		

Table 4. *Bond angles* (°)

C(1)—C(2)—C(3)	120·9	C(14)-C(1)—S	65·9
C(1)—C(2)—C(7)	120·3	C(1)—S——C(14)	49·9
C(2)—C(3)—C(4)	120·8	C(1)—C(14)-S	64·2
C(3)—C(4)—C(5)	120·2	C(1)—C(14)-C(15)	116·9
C(4)—C(5)—C(6)	119·7	C(1)—C(14)-C(19)	119·7
C(5)—C(6)—C(7)	120·1	S——C(14)-C(15)	115·9
C(6)—C(7)—C(2)	120·8	S——C(14)-C(19)	109·4
C(7)—C(2)—C(3)	118·4	C(15)-C(14)-C(19)	118·3
C(1)—C(8)—C(9)	121·2	C(14)-C(15)-C(16)	109·8
C(1)—C(8)—C(13)	120·4	C(14)-C(15)-C(17)	115·2
C(8)—C(9)—C(10)	120·7	C(14)-C(15)-C(18)	113·7
C(9)—C(10)-C(11)	120·6	C(16)-C(15)-C(17)	106·5
C(10)-C(11)-C(12)	119·4	C(16)-C(15)-C(18)	107·9
C(11)-C(12)-C(13)	120·3	C(17)-C(15)-C(18)	103·2
C(12)-C(13)-C(8)	121·1	C(14)-C(19)-C(20)	115·2
C(13)-C(8)—C(9)	117·8	C(14)-C(19)-C(21)	110·0
C(2)—C(1)—C(8)	103·9	C(14)-C(19)-C(22)	112·7
C(2)—C(1)—C(14)	123·4	C(20)-C(19)-C(21)	103·0
C(2)—C(1)—S	118·8	C(20)-C(19)-C(22)	106·3
C(8)—C(1)—C(14)	126·1	C(21)-C(19)-C(22)	109·1
C(8)—C(1)—S	114·3		

Table 5. *Some torsion angles* (°)

C(14)-C(1)—C(2)—C(3)	−118·0
C(14)-C(1)—C(2)—C(7)	68·7
C(14)-C(1)—C(8)—C(9)	−84·9
C(14)-C(1)—C(8)—C(13)	104·4
C(15)-C(14)-C(1)—C(2)	−142·9
C(15)-C(14)-C(1)—C(8)	4·0
C(19)-C(14)-C(1)—C(2)	11·2
C(19)-C(14)-C(1)—C(8)	158·0
C(17)-C(15)-C(14)-C(19)	36·7
C(15)-C(14)-C(19)-C(21)	−70·0

In the thiirane ring the C–C distance (1·550 Å) is considerably longer than that found (1·468 Å) for the analogous bond in (2*S*,3*S*)-1-cyano-2-hydroxy-3,4-epithiobutane-α-naphthylurethane (Bates, Grady & Sneath, 1972); in thiirane itself (Cunningham, Boyd, Myers & Gwinn, 1951) the corresponding bond length is 1·492 Å, in agreement with recent *ab initio* MO–SCF calculations (Rohmer & Roos, 1975). The longer C–C distance in the present compound could be ascribed to the strong hindrance between t-butyl groups and phenyl rings, which could also be reflected in the long C(14)–S distance (1·848 Å), the C(1)–S bond having a more normal length (1·822 Å). In the previously quoted compound (Bates *et al.*, 1972) the two C–S distances are 1·802 and 1·815 Å; in 1,3,5,7-tetrathiocane (Frank & Degen, 1973) the C–S length, averaged over the three rings of the asymmetric unit, is 1·817 Å; in thiirane the experimental (Cunningham *et al.*, 1951) and the calculated value (Rohmer & Roos, 1975) is 1·819 Å. However, C–S distances as long as 1·84–1·85 Å have been found, *e.g.* in dibenzyl disulphide (Lee & Bryant, 1969) and in L-α-(*p*-chlorobenzenesulphonamido)-β-propionothiolactone (Milinović & Bezjak, 1973).

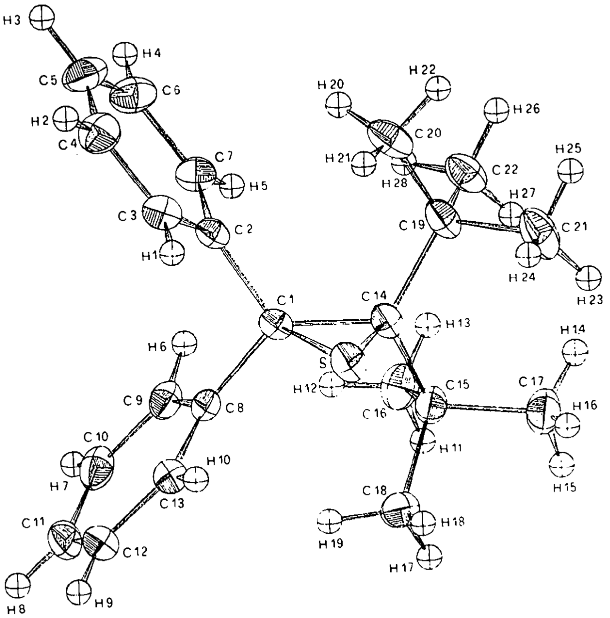

Fig. 1. Numbering of atoms and temperature ellipsoids. Heavy atoms are drawn at a probability 0·50; hydrogen atoms, treated as isotropic, are on an arbitrary scale.

The molecular overcrowding is also reflected in the long C(1)–C(8) $C(sp^3)$–$C(sp^2)$ length (1·535 Å) and in the $C(sp^3)$–$C(sp^3)$ distances connecting the t-butyl groups to the thiirane ring (1·587 and 1·598 Å, respectively). These last values are in good agreement with those (1·59–1·61 Å) found for the bonds to the t-butyl groups in tetra-t-butylacetone (Lepicard, Berthou, Delettré, Laurent & Mornon, 1973).

Evidence for steric hindrance in the present compound is given by the short intramolecular contacts reported in Table 6. These may be responsible for some deviation from planarity of phenyl rings. Least-squares planes have been calculated by the method of Schomaker, Waser, Marsh & Bergman (1959); atoms of the first phenyl ring, C(2) to C(7), are coplanar within 0·010 Å, and those of the second, C(8) to C(13), within 0·020 Å. The angles between the best plane through C(1), C(2), C(5), C(8), C(11) and the first and second phenyl ring are 89 and 72°, respectively.

Table 6. *Some* 1···5 *and* 1···6 *intramolecular contacts* (Å)

C(7)····C(9)	3·18	C(17)···C(21)	3·03
C(9)····C(16)	3·34	C(17)···C(22)	3·41
C(8)····C(18)	3·04	C(20)···C(2)	2·83
C(13)···C(18)	3·41	C(20)···C(3)	3·35
C(16)···C(22)	3·24	C(20)···C(7)	3·20

Intermolecular contacts are normal. There are only five contacts shorter by 0·1 Å or more than the sum of van der Waals radii (S 1·85, C 1·7, CH_3 2·0, H 1·2 Å; Pauling, 1960):

Atom in x,y,z	to atom	in position			Distance (Å)
S	H(28)	x	$\frac{1}{2}-y$	$\frac{1}{2}+z$	2·94
C(10)	H(22)	$1-x$	$-\frac{1}{2}+y$	$\frac{3}{2}-z$	2·80
C(16)	H(8)	$2-x$	$\frac{1}{2}+y$	$\frac{3}{2}-z$	3·03
C(18)	H(9)	$2-x$	$-y$	$2-z$	3·01
C(21)	H(25)	$1-x$	$1-y$	$2-z$	2·99

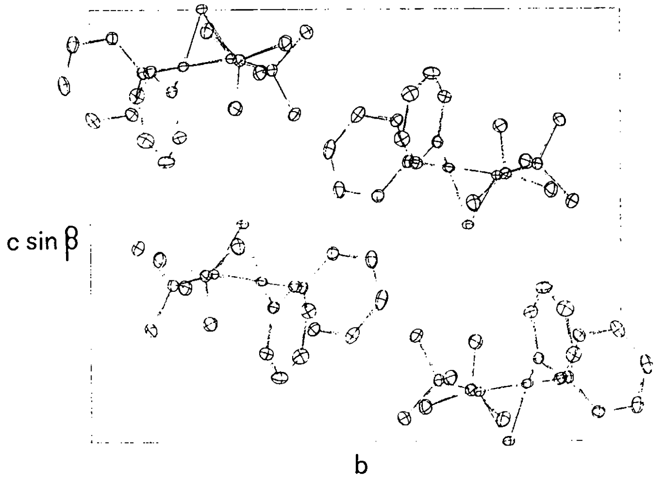

Fig. 2. Packing of molecules as seen along the x axis.

A plotter output of the packing of molecules in the crystal is shown in Fig. 2.

The authors thank Professor D. H. R. Barton for the gift of a sample of the compound from which both crystalline phases could be isolated, and Dr G. Casalone for the room-temperature data collection.

APPENDIX

The orthorhombic phase

An orthorhombic crystal was examined at room temperature with the same diffractometer and Cu $K\alpha$ radiation. Lattice parameters were derived from nine high-angle reflexions (plus their equivalents) for $\lambda = 1{\cdot}54178$ Å; the results are reported in the abstract. Systematic extinctions ($h00$ for h odd, $0k0$ for k odd, $00l$ for l odd) led to space group $P2_12_12_1$. Intensities were collected with the θ–2θ scan technique with graphite-monochromatized Cu $K\alpha$ radiation, scan width $2° + \alpha_1\alpha_2$ separation, variable scan speed between 1 and 12° $\min^{-1}$, and dead time for coincidence correction $2{\cdot}1 \times 10^{-6}$ s. The background was counted for half the total scanning time on each side of the profile. After each group of 40 reflexions, a standard reflexion was measured, which showed an intensity decrease nearly linear with time.* Accordingly, a linear decay correction was applied; data were then corrected for Lorentz and polarization factors. No absorption correction was applied [μ(Cu $K\alpha$) = 14 cm^{-1}]. In the range explored ($2\theta_{max} = 140°$) 2056 independent reflexions were measured, of which 1930 had intensities above background.

The structure was solved with the molecular model obtained from the monoclinic phase. The orientation of the molecule in the unit cell was determined by rotational search in a sharpened Patterson map calculated around the origin (Filippini & Gramaccioli, 1969), and then the position of the oriented model in the unit cell was found by means of a translational search in the Patterson map with a program written by one of us (A.M.). The result was confirmed by the minimum residual technique.

Refinement was accomplished on S and C atoms with all observed reflexions and the same program as for the monoclinic phase. After anisotropic refinement, anomalies were found in the geometry of the first phenyl ring. A difference map gave some indication of disorder in the orientation of this ring. Because of the large thermal factors and the insufficient resolution of our room-temperature data, we deemed it unfruitful to refine this structure further.

The heavy-atom parameters are given in Table 7. R for all 1930 observed reflexions is 0·099.† Aside from the first phenyl ring and from a longer C(19)–C(21) distance, the geometry of the molecule is in good agreement with that of the monoclinic phase.

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* At the end of data collection, the intensity had decreased by 17%.

† See footnote on p. 1763.

Table 7. *The heavy-atom parameters of the orthorhombic phase at room temperature, and their standard deviations*

All values in this table have been multiplied by 10^4. Thermal factors are of the form $\exp\,[-(B_{11}h^2+2B_{12}hk+\ldots)]$.

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
S	8265 (3)	2491 (1)	7727 (1)	321 (5)	55 (1)	52 (1)	35 (2)	27 (2)	3 (1)
C(1)	8357 (8)	1211 (4)	7255 (3)	166 (11)	69 (4)	37 (2)	3 (6)	4 (5)	1 (3)
C(2)	6921 (9)	700 (6)	6982 (4)	149 (11)	110 (6)	39 (2)	17 (8)	18 (5)	0 (3)
C(3)	5821 (10)	1310 (8)	6667 (5)	166 (13)	179 (9)	48 (3)	54 (10)	19 (6)	19 (5)
C(4)	4538 (11)	768 (11)	6345 (5)	174 (15)	229 (14)	54 (4)	36 (14)	7 (6)	17 (6)
C(5)	4377 (16)	−333 (11)	6404 (7)	291 (24)	183 (13)	85 (6)	54 (17)	1 (10)	1 (7)
C(6)	5504 (14)	−888 (8)	6696 (7)	280 (21)	141 (9)	89 (6)	−36 (13)	37 (10)	−13 (6)
C(7)	6873 (10)	−403 (6)	7006 (5)	193 (14)	99 (6)	77 (4)	−50 (8)	14 (7)	−11 (4)
C(8)	9388 (8)	1156 (5)	6508 (3)	147 (10)	76 (4)	36 (2)	−6 (6)	4 (4)	−1 (3)
C(9)	9923 (8)	203 (6)	6244 (4)	136 (10)	94 (5)	43 (3)	8 (6)	0 (4)	−17 (3)
C(10)	10749 (9)	148 (7)	5529 (5)	169 (12)	131 (7)	56 (3)	3 (9)	−7 (6)	−34 (4)
C(11)	10986 (11)	1078 (11)	5077 (5)	204 (16)	216 (12)	46 (3)	−36 (13)	12 (6)	−13 (6)
C(12)	10391 (11)	2027 (7)	5329 (5)	260 (18)	137 (8)	52 (3)	−25 (11)	9 (7)	−1 (4)
C(13)	9591 (10)	2049 (5)	6046 (4)	248 (15)	87 (5)	43 (3)	−3 (8)	17 (6)	11 (3)
C(14)	8878 (9)	1226 (5)	8136 (3)	242 (13)	55 (4)	34 (2)	7 (6)	9 (5)	−3 (3)
C(15)	10630 (10)	1168 (6)	8300 (5)	219 (14)	93 (5)	50 (3)	−8 (8)	−17 (6)	−18 (4)
C(16)	11155 (12)	24 (7)	8151 (6)	296 (19)	95 (6)	93 (5)	49 (9)	−38 (9)	−12 (5)
C(17)	11143 (13)	1571 (9)	9131 (5)	354 (23)	222 (12)	49 (3)	−36 (15)	−32 (8)	−37 (6)
C(18)	11539 (11)	1856 (7)	7726 (6)	228 (17)	156 (9)	85 (5)	−92 (11)	−7 (8)	21 (6)
C(19)	7803 (11)	822 (6)	8829 (4)	265 (17)	100 (6)	40 (3)	34 (8)	27 (6)	11 (3)
C(20)	6168 (12)	902 (10)	8638 (6)	235 (17)	223 (13)	66 (4)	60 (13)	57 (7)	34 (6)
C(21)	7864 (18)	1611 (11)	9569 (5)	574 (39)	275 (16)	45 (3)	21 (22)	45 (11)	−47 (7)
C(22)	8179 (14)	−287 (8)	9107 (7)	352 (24)	138 (9)	126 (7)	25 (13)	81 (12)	64 (7)

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