Table 3. *Comparison of intra-annular torsion angles (°) in the C rings of the related compounds (Fig.* 1)

	(I)[a]	(II)[b]	(III)[c]	(IV)[d]
C(1)–C(2)–C(3)–C(4)	0·8 (3)	−0·1 (2)	0·1 (2)	54·5 (3)
C(2)–C(3)–C(4)–N(13)	19·6 (2)	−6·3 (2)	24·2 (2)	−55·3 (3)
C(3)–C(4)–N(13)–N(14)	−4·1 (2)	24·6 (1)	−52·7 (2)	53·8 (3)
C(4)–N(13)–N(14)–C(1)	−32·3 (2)	−36·6 (1)	57·6 (2)	−50·8 (3)
N(13)–N(14)–C(1)–C(2)	50·7 (2)	28·9 (2)	−30·3 (2)	52·3 (3)
N(14)–C(1)–C(2)–C(3)	−34·6 (3)	−10·8 (2)	1·9 (2)	−54·2 (3)

References: (*a*) Foces-Foces, Cano & García-Blanco (1977*a*). (*b*) Foces-Foces, Cano & García-Blanco (1977*b*). (*c*) Foces-Foces, Cano & García-Blanco (1978). (*d*) This work.

The hydroxyl groups are *trans*–axial to each other. O(15) and O(18) deviate by 2·1 and 1·5° respectively from the theoretical axial position. The deviations of the equatorial methyl C atoms, C(17) and C(16), are 2·3 and 3·4° respectively.

The *C* ring has a chair conformation, while in other related compounds [(I), (II) and (III) in Fig. 1] (Foces-Foces, Cano & García-Blanco, 1977*a*,*b*, 1978) it has approximately diplanar, half-chair and envelope conformations respectively, as shown in Table 3.

We thank Professor M. Lora Tamayo and co-workers for suggesting the problem and for providing the material. We also thank the Centro de Proceso de Datos del Ministerio de Educación y Ciencia (Madrid) for allowing us the use of the 1108 Univac computer. Most of the computations were performed with the XRAY 70 system of crystallographic programs (Stewart, Kundell & Baldwin, 1970).

References

Foces-Foces, C., Cano, F. H. & García-Blanco, S. (1977*a*). *Acta Cryst.* B**33**, 3521–3524.
Foces-Foces, C., Cano, F. H. & García-Blanco, S. (1977*b*). *Acta Cryst.* B**33**, 3957–3959.
Foces-Foces, C., Cano, F. H. & García-Blanco, S. (1978). *Acta Cryst.* B**34**, 991–993.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.
Main, P., Woolfson, M. M., Lessinger, L., Germain, G. & Declercq, J. P. (1974). *MULTAN 74. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
Stewart, J. M., Kundell, F. A. & Baldwin, J. C. (1970). The XRAY 70 system. Computer Science Center, Univ. of Maryland, College Park, Maryland.
Winkler, F. K. & Dunitz, J. D. (1971). *J. Mol. Biol.* **59**, 169–182.

Acta Cryst. (1978). B**34**, 3403–3407

The Structure of 2-(2,6-Dimethylphenylimino)-3,3-dimethyl-4,4-diphenylthietane

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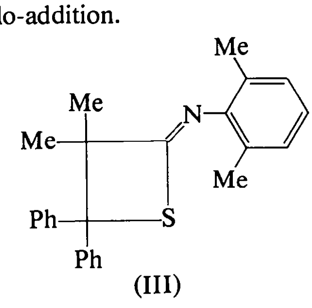
Abstract. $C_{25}H_{25}NS$, orthorhombic, $P2_12_12_1$, $Z = 4$, $a = 12{\cdot}598$ (3), $b = 17{\cdot}763$ (4), $c = 9{\cdot}376$ (3) Å, $D_c = 1{\cdot}16$ g cm^{-3}, μ(Cu $K\alpha$) = 13·2 cm^{-1}. The structure was solved by direct methods and refined by full-matrix least squares to an R value of 0·061. The thietane ring is puckered with dihedral angles of 20 and 21° and contains a C(sp^3)–C(sp^3) single bond distance of 1·591 Å.

Introduction. As a part of a study on the molecular structure of the 1:1 adducts between thiobenzophenone (I) and substituted ketenimines (II)

$$\underset{\text{(I)}}{Ph_2C{=}S} \quad \underset{\text{(II)}}{R^1_2C{=}C{=}N{-}R^2} \quad \begin{array}{l}(a)\ R^1 = Ph; R^2 = p\text{-}MeC_6H_4 \\ (b)\ R^1 = Me; R^2 = 2,6\text{-}Me_2C_6H_3\end{array}$$

we have recently determined (Dondoni, Battaglia, Giorgianni, Gilli & Sacerdoti, 1977; Sacerdoti, Bertolasi, Gilli, Dondoni & Battaglia, 1977) the crystal structure of the adduct between (I) and (II*a*). Rather unexpectedly this turned out to be a six-membered heterocycle, *i.e.* a 4*H*-3,1-benzothiazine. In the present paper the crystal structure of the product of the reaction between (I) and (II*b*) is reported, proving that such reaction leads to a 2-iminothietane derivative (III) by a 2 + 2 cyclo-addition.



(III)

Table 1. *Positional parameters* ($\times 10^4$) *with e.s.d.'s in parentheses*

	x	y	z
S	2865 (1)	1611 (1)	8784 (1)
N	4749 (4)	813 (2)	8636 (5)
C(1)	3503 (4)	2559 (3)	8881 (5)
C(2)	4582 (4)	2212 (3)	8298 (6)
C(3)	4237 (4)	1403 (3)	8584 (5)
C(4)	4721 (5)	2270 (4)	6678 (7)
C(5)	5627 (4)	2457 (3)	9023 (6)
C(6)	3017 (4)	3150 (3)	7877 (4)
C(7)	2015 (4)	3069 (3)	7307 (5)
C(8)	1596 (4)	3634 (3)	6436 (7)
C(9)	2148 (5)	4276 (3)	6154 (6)
C(10)	3160 (4)	4363 (3)	6744 (7)
C(11)	3568 (4)	3810 (3)	7603 (6)
C(12)	3537 (4)	2819 (2)	10442 (6)
C(13)	3137 (5)	3496 (3)	10866 (6)
C(14)	3147 (6)	3710 (3)	12318 (7)
C(15)	3579 (5)	3237 (4)	13314 (7)
C(16)	3957 (6)	2540 (4)	12880 (6)
C(17)	3939 (5)	2343 (3)	11473 (6)
C(18)	4212 (4)	113 (3)	8825 (7)
C(19)	3749 (5)	−245 (3)	7671 (9)
C(20)	3237 (6)	−916 (5)	7895 (12)
C(21)	3197 (7)	−1253 (4)	9242 (13)
C(22)	3744 (6)	−915 (3)	10389 (10)
C(23)	4232 (5)	−218 (3)	10171 (8)
C(24)	3821 (8)	75 (4)	6194 (10)
C(25)	4758 (7)	164 (4)	11404 (8)

Intensities were collected on an automated Siemens AED diffractometer with Ni-filtered Cu $K\bar{\alpha}$ radiation and an $\omega/2\theta$ scan. Out of the 1534 reflections collected ($\theta \leq 55°$), 1455 having $I_o \geq 3\sigma(I_o)$ were used in the refinement. Intensities were not corrected for absorption ($\mu R = 0{\cdot}20$). Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The structure was solved by *MULTAN* 74 (Main, Woolfson, Lessinger, Germain & Declercq, 1974) and the remaining calculations were carried out by the *SHELX* 76 system of programs (G. M. Sheldrick). H atoms were assigned calculated positions (with a C—H bond distance of 1·08 Å) and their isotropic temperature factors were then fixed as 20% greater than those of their bonding atoms. The structure was refined by a full-matrix least-squares method using anisotropic temperature factors. Final disagreement factors were R ($= \sum|\Delta|/\sum|F_o|$) $= 0{\cdot}061$ and R_w $[=(\sum w|\Delta|^2/\sum w|F_o|^2)^{1/2}] = 0{\cdot}069$. Weights were given as $1/w = \sigma^2(F_o) + 0{\cdot}0146|F_o|^2$. The final coordinates are reported in Table 1.*

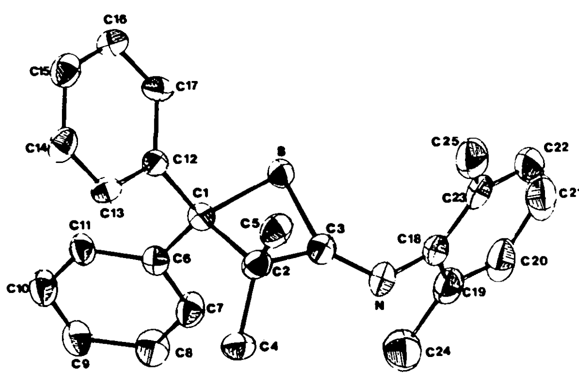


Fig. 1. An *ORTEP* view of the molecule showing the thermal ellipsoids at 40% probability (Johnson, 1965).

Discussion. A drawing of the molecule is shown in Fig. 1 and bond distances and angles are given in Tables 2 and 3.

The structure consists of discrete molecular units without significantly short intermolecular distances. The conformations of the three phenyl rings appear to be weakly affected by the crystal forces, as can be proved by the following simple arguments. The experimental torsion angles, C(3)—N—C(18)—C(19) = $T1$ = 81·5, S—C(1)—C(6)—C(7) = $T2$ = 19·5 and S—C(1)—C(12)—C(13) = $T3$ = −126·5°, can be recalculated by minimizing the non-bonded intramolecular potential energy (U) of the free molecule, using semi-empirical atom-pair potential curves (Giglio, 1969). The two sharp minima observed in the $U(T1)$ and $U(T2,T3)$ energy maps obtained in this way correspond to torsion angles $T1$ = 80·4, $T2$ = 22·3 and $T3$ = −130·5°, which are in reasonable agreement with the experimental values. This result matches the idea that, in general, molecular conformations are weakly affected by the crystal field when the potential-energy minima are sharp enough (Kitaigorodsky, 1970).

The molecule does not show any remarkable characteristics except for the puckering of the thietane ring and the lengthening of some of its bond distances.

In general the puckering of four-membered rings depends on the balancing of two opposing forces in the molecule, the ring strain (caused by the narrowing of the ring angles and by the cross-ring repulsion) and the torsional strain (caused by the non-bonded interactions among vicinal substituents). The ring strain tends to flatten the ring as the puckering further decreases the already highly-strained ring angles. The torsional strain depends on the connectivity of the atoms of the ring; in the thietane molecule it is mainly caused by the repulsion of the substituents of the two vicinal C(sp^3) atoms, which are eclipsed if the ring is planar. If the torsional force dominates, the potential function will have a double minimum with a barrier corresponding to the planar configuration. An order of magnitude for such a barrier can be derived from the

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33675 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

paper by Harris, Harrington, Luntz & Gwinn (1966) on the unsubstituted thietane. They evaluated the height of the barrier and the position of the double minimum as 274·2 cm^{-1} and $\pm32°$ respectively. That is, the barrier is rather small and smaller than in cyclobutanes (490 cm^{-1}; Cotton & Frenz, 1974).

The puckering angles found in the present and in other similar molecules are reported in Table 4. No strict correlation can be established between puckering angles and steric hindrance of the vicinal substituents. However, puckering angles $\leq5°$ have been observed only if all the substituents on C(sp^3) atoms are H, while puckering angles $\geq20°$ have been observed in all the rings with bulky substituents on vicinal carbons. This happens in the present molecule and in *cis*-2,2-diphenyl-3,4-dichlorothietane (puckering angle B = 28·9°; Kumakura & Shimozawa, 1972). These conclusions are in agreement with the results of the structural analysis on five thietane 1-dioxide (Andreetti, Bocelli & Sgarabotto, 1974) and two thietane 1-oxide derivatives (Hardgrove, Brathold & Lein, 1974; Abrahamsson & Rehnberg, 1972).

The values of the bond angles inside the ring show clearly the strain the ring is submitted to. Deviations from the sp^3 geometry for the angles C(1)–C(2)–C(3) and S–C(1)–C(2) are −16·0 and −19·4°, and the deviation from the sp^2 geometry for the angle S–C(3)–C(2) is −24·2°. The deviation of the angle C(1)–S–C(3) from the mean C–S–C angle found in diphenyl sulphides (103·7°; Sacerdoti, Bertolasi & Gilli, 1976) is −26·7°.

The torsion angles C(12)–C(1)–C(2)–C(5) and N–C(3)–C(2)–C(5) are respectively 25·2 and 41·4°. It may be remarked that these angles are differently related to the puckering of the ring, rotation around the C(1)–C(2) and C(2)–C(3) bonds tending respectively to pucker or to flatten the ring itself.

A comparison of the S–C distances in thietane rings is shown in Table 4. The mean values of the S–C(sp^3) and S–C(sp^2) bond lengths are respectively 1·82 (1) and 1·77 (1) Å, in agreement with the corresponding values 1·83 (1) and 1·76 (1) Å found as an average in thiazine derivatives (Sacerdoti, Bertolasi, Gilli, Dondoni & Battaglia, 1977). As for the S–C(sp^2) and

Table 2. *Bond distances* (Å) *with e.s.d.'s in parentheses*

S–C(1)	1·868 (5)	C(10)–C(11)	1·370 (8)
S–C(3)	1·777 (5)	C(12)–C(13)	1·364 (7)
N–C(3)	1·232 (7)	C(12)–C(17)	1·380 (8)
N–C(18)	1·427 (7)	C(13)–C(14)	1·413 (9)
C(1)–C(2)	1·591 (7)	C(14)–C(15)	1·369 (10)
C(1)–C(6)	1·537 (7)	C(15)–C(16)	1·386 (9)
C(1)–C(12)	1·535 (8)	C(16)–C(17)	1·365 (8)
C(2)–C(3)	1·524 (7)	C(18)–C(19)	1·385 (9)
C(2)–C(4)	1·532 (9)	C(18)–C(23)	1·393 (10)
C(2)–C(5)	1·544 (8)	C(19)–C(20)	1·372 (11)
C(6)–C(7)	1·378 (7)	C(19)–C(24)	1·499 (13)
C(6)–C(11)	1·387 (7)	C(20)–C(21)	1·399 (14)
C(7)–C(8)	1·397 (8)	C(21)–C(22)	1·411 (14)
C(8)–C(9)	1·361 (9)	C(22)–C(23)	1·398 (9)
C(9)–C(10)	1·398 (8)	C(23)–C(25)	1·496 (10)

Table 3. *Bond angles* (°) *with e.s.d.'s in parentheses*

C(1)–S–C(3)	77·0 (2)	C(9)–C(10)–C(11)	119·7 (5)
C(3)–N–C(18)	119·9 (4)	C(6)–C(11)–C(10)	121·8 (5)
S–C(1)–C(2)	90·1 (3)	C(1)–C(12)–C(13)	122·2 (5)
S–C(1)–C(6)	114·5 (3)	C(1)–C(12)–C(17)	119·6 (5)
S–C(1)–C(12)	109·2 (3)	C(13)–C(12)–C(17)	118·1 (5)
C(2)–C(1)–C(6)	113·2 (4)	C(12)–C(13)–C(14)	120·9 (5)
C(2)–C(1)–C(12)	114·9 (4)	C(13)–C(14)–C(15)	119·7 (5)
C(6)–C(1)–C(12)	113·0 (4)	C(14)–C(15)–C(16)	118·9 (6)
C(3)–C(2)–C(1)	93·5 (4)	C(15)–C(16)–C(17)	120·5 (6)
C(3)–C(2)–C(4)	105·7 (5)	C(12)–C(17)–C(16)	121·7 (5)
C(3)–C(2)–C(5)	115·6 (4)	N–C(18)–C(19)	120·2 (6)
C(4)–C(2)–C(1)	114·3 (5)	N–C(18)–C(23)	118·2 (5)
C(4)–C(2)–C(5)	108·7 (5)	C(19)–C(18)–C(23)	121·5 (6)
C(5)–C(2)–C(1)	117·9 (4)	C(18)–C(19)–C(20)	118·5 (8)
S–C(3)–N	133·0 (4)	C(18)–C(19)–C(24)	121·5 (7)
S–C(3)–C(2)	95·8 (3)	C(20)–C(19)–C(24)	120·0 (8)
N–C(3)–C(2)	131·2 (5)	C(19)–C(20)–C(21)	121·8 (8)
C(1)–C(6)–C(7)	122·0 (4)	C(20)–C(21)–C(22)	119·3 (7)
C(1)–C(6)–C(11)	119·4 (4)	C(21)–C(22)–C(23)	118·7 (8)
C(7)–C(6)–C(11)	118·4 (4)	C(18)–C(23)–C(22)	119·9 (7)
C(6)–C(7)–C(8)	119·8 (5)	C(18)–C(23)–C(25)	121·1 (6)
C(7)–C(8)–C(9)	121·5 (5)	C(22)–C(23)–C(25)	119·0 (7)
C(8)–C(9)–C(10)	118·8 (5)		

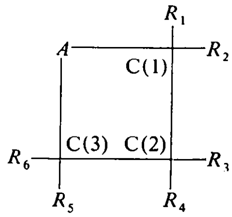
Table 4. *Geometrical data of thietane rings, distances in* Å *and angles in degrees, with e.s.d.'s in parentheses*

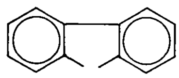
	Reference	S–C(sp^3)	S–C(sp^2)	C–S–C	Puckering angles A*	Puckering angles B†
3-(p-BrPh–SO_2–NH–)thietan-2-one	*a*	1·81 (1)	1·770 (8)	77·7 (4)	8	–
3-(p-MePh–SO_2–NH–)thietan-2-one	*a*	1·810 (6)	1·763 (6)	76·3 (3)	17	–
3-(p-CNPh–SO_2–NH–)thietan-2-one	*a*	1·81 (1)	1·78 (1)	76·2 (4)	21	–
2-(R_2C=)thietane	*b*	1·823 (8)	1·773 (6)	77·5 (3)	0	0
2-(R'_2C=)thietane	*c*	1·84 (1)	1·749 (6)	76·5 (4)	–	5
Mean values		1·82 (1)	1·77 (1)	76·8 (7)		
Present structure		1·868 (5)	1·777 (5)	77·0 (2)	21	20

References: (*a*) Matijašić, Andreetti & Sgarabotto (1976). (*b*) Nakai & Koyama (1971). (*c*) Nakai & Koyama (1972).

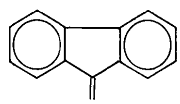
* A = dihedral angle between the S–C(1)–C(2) and S–C(3)–C(2) planes.
† B = dihedral angle between the C(1)–S–C(3) and C(1)–C(2)–C(3) planes.

Table 5. *A selection of long* C(sp^3)—C(sp^3) *bond distances* (Å) *in four-membered rings with e.s.d.'s in parentheses*



A	R_1	R_2	R_3	R_4	R_5	R_6	C(1)—C(2)	C(2)—C(3)	Reference
S	Ph	Ph	Me	Me	o,o'-Me_2Ph—N=		1·591 (7)	–	*a*
S	H	H	Me	—CN	Me—N(CO—N—Me)(CH_2—CH_2)		1·541 (4)	1·597 (4)	*b*
O=C=	=CHPh		H	Ph	—CN	—CMe_3	–	1·614 (3)	*c*
R*=C=	=CHPh				H	Ph	–	1·606 (6)	*d*
H_2C=	—COOMe	p-NO_2Ph	—COOMe	p-NO_2Ph	H	H	1·606 (3)	1·567 (3)	*e*

References: (*a*) Present work. (*b*) Guilhem (1977). (*c*) McDonald (1975). (*d*) Dreissig, Luger & Rewicki (1974). (*e*) Carr, Finney, Lindley & De Titta (1977).

*R= 

S—C(sp^3) distances found in the present structure [1·777 (5) and 1·868 (5) Å], the former agrees well with the mean values but the latter is significantly longer. Whether this lengthening of the S—C(sp^3) bond is a peculiarity of thietane rings with bulky substituents cannot be decided now, owing to the lack of experimental data. Conversely, the long bond length C(1)—C(2) of 1·591 (7) Å can be easily interpreted in terms of steric hindrance of vicinal substituents. Table 5 reports most of the long C(sp^3)—C(sp^3) bond distances so far found in non-condensed four-membered rings and shows that abnormally long bonds have never been found if the two substituents of the same C atom are H atoms.

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References

Abrahamsson, S. & Rehnberg, G. (1972). *Acta Chem. Scand.* **26**, 494–500.

Andreetti, G. D., Bocelli, G. & Sgarabotto, P. (1974). *Gazz. Chim. Ital.* **104**, 1207–1215.

Carr, P., Finney, J. L., Lindley, P. F. & De Titta, G. T. (1977). *Acta Cryst.* B**33**, 1022–1026.

Cotton, F. A. & Frenz, B. A. (1974). *Tetrahedron*, **30**, 1587–1594.

Dondoni, A., Battaglia, A., Giorgianni, P., Gilli, G. & Sacerdoti, M. (1977). *J. Chem. Soc. Chem. Commun.* pp. 43–44.

Dreissig, W., Luger, P. & Rewicki, D. (1974). *Acta Cryst.* B**30**, 2037–2042.

Giglio, G. (1969). *Nature* (*London*), **222**, 339–341.

Guilhem, J. (1977). *Cryst. Struct. Commun.* **6**, 93–96.

Hardgrove, G. L., Brathold, J. S. & Lein, M. M. (1974). *J. Org. Chem.* **39**, 246–248.

Harris, D. O., Harrington, H. W., Luntz, A. C. & Gwinn, W. D. (1966). *J. Chem. Phys.* **44**, 3467–3480.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794, revised. Oak Ridge National Laboratory. Tennessee.

Kitaigorodsky, A. I. (1970). *Advances in Structure Research by Diffraction Methods*, edited by R. Brill & R. Mason, pp. 173–247. New York: John Wiley.

Kumakura, S. & Shimozawa, T. (1972). *Acta Cryst.* A**28**, S29.

McDonald, W. S. (1975). *Acta Cryst.* B**31**, 2504–2505.

Main, P., Woolfson, M. M., Lessinger, L., Germain, G. & Declercq, J. P. (1974). *MULTAN* 74. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. York, England, and Louvain, Belgium.

MATIJAŠIĆ, I., ANDREETTI, G. D. & SGARABOTTO, P. (1976). *Izv. Jugosl. Cent. Kristallogr.* **11**, A124–A126.

NAKAI, H. & KOYAMA, H. (1971). *J. Chem. Soc. B*, pp. 1525–1529.

NAKAI, H. & KOYAMA, H. (1972). *J. Chem. Soc. Perkin Trans.* 2, pp. 248–252.

SACERDOTI, M., BERTOLASI, V. & GILLI, G. (1976). *Cryst. Struct. Commun.* **5**, 477–481.

SACERDOTI, M., BERTOLASI, V., GILLI, G., DONDONI, A. & BATTAGLIA, A. (1977). *Acta Cryst.* B**33**, 2816–2819.

Acta Cryst. (1978). B**34**, 3407–3409

3,5,3′-Triiodo-4′-methoxythyropropionic Acid Methyl Ester

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Abstract. $C_{17}H_{15}I_3O_4$, m.p. 185 °C, monoclinic, $P2_1/c$, $a = 9{\cdot}736$ (2), $b = 7{\cdot}510$ (1), $c = 28{\cdot}387$ (5) Å, $\beta = 94{\cdot}59$ (2)°, $Z = 4$, $M_r = 664{\cdot}02$, $D_c = 2{\cdot}13$ g cm^{-3}, observed data $R = 6{\cdot}8\%$. The conformation of the iodine at the 3′ position is proximal and the propionic acid side chain is fully extended. Both the methoxy and methyl ester carbons are coplanar with their respective functional groups. The torsion angles between the phenyl rings and the ether oxygen (φ and φ') are 103 and −21°, respectively, and the C—O—C angle is 117°.

Table 1. *Positional parameters for* 3,5,3′-*triiodo*-4′-*methoxythyropropionic acid methyl ester*

	x	y	z
I(3′)	0·3378 (2)	0·2316 (2)	−0·15697 (6)
I(3)	0·9483 (2)	0·2130 (2)	−0·09156 (6)
I(5)	0·6282 (2)	0·8223 (2)	−0·01414 (8)
C(1)	0·7840 (20)	0·3235 (26)	0·04296 (68)
C(2)	0·8544 (20)	0·2465 (29)	0·00645 (82)
C(3)	0·8511 (22)	0·3351 (28)	−0·03665 (76)
C(4)	0·7833 (21)	0·4975 (27)	−0·04427 (72)
C(5)	0·7234 (23)	0·5725 (26)	−0·00636 (80)
C(6)	0·7175 (21)	0·4846 (27)	0·03722 (75)
O(41)	0·7933 (15)	0·5886 (19)	−0·08601 (52)
C(1′)	0·6824 (23)	0·5803 (30)	−0·11917 (76)
C(2′)	0·5860 (23)	0·4445 (28)	−0·11908 (82)
C(3′)	0·4799 (23)	0·4368 (29)	−0·15525 (72)
C(4′)	0·4670 (25)	0·5756 (32)	−0·18782 (82)
C(5′)	0·5624 (30)	0·7087 (38)	−0·18763 (88)
C(6′)	0·6660 (27)	0·7119 (23)	−0·15350 (89)
O(4′1)	0·3582 (19)	0·5626 (23)	−0·22148 (61)
C(4′2)	0·3406 (31)	0·6959 (43)	−0·25667 (103)
C(7)	0·7810 (26)	0·2241 (31)	0·08923 (94)
C(8)	0·8939 (25)	0·3002 (29)	0·12618 (80)
C(9)	0·9310 (24)	0·1838 (30)	0·16933 (71)
O(9)	0·9067 (24)	0·0315 (23)	0·17150 (67)
O(10)	0·9939 (19)	0·2786 (23)	0·20153 (66)
C(10)	1·0377 (34)	0·1877 (35)	0·24495 (118)

Introduction. 3,5,3′-Triiodo-4′-methoxythyropropionic acid methyl ester was prepared by methylation of triiodothyropropionic acid in methanol using diazomethane. Diazomethane in ether–alcoholic solution was prepared from *N*-methyl-*N*-nitroso-*p*-toluenesulfonamide by a commercial diazomethane generator (Aldrich Diazald Kit, Aldrich Chemical Co., Milwaukee, Wisconsin). A small crystal (0·02 × 0·36 × 0·38 mm) was selected for intensity data collection. The data showed the systematic absences for the space group $P2_1/c$ and the cell constants were determined by least-squares analysis of the angular settings of 45 reflections having $2\theta > 21°$ [at 20 °C; λ(Mo $K\alpha$) = 0·7091 Å]. The intensities of 2885 (1202 reflections had $I > 2\sigma$) with $2\theta < 55°$ were measured on an Enraf–Nonius CAD-4 automated diffractometer. Reflections were measured in the θ–2θ scan mode using Zr-filtered Mo $K\alpha$ radiation and a sweep = 1·1° + 0·1 tan θ. No significant changes were observed in the intensities of the standard reflections measured daily during data collection. Intensities were corrected for Lorentz and polarization factors, but not for absorption. The structure was solved by application of heavy-atom techniques.

The positional and anisotropic thermal parameters for all non-hydrogen atoms were refined using full-matrix least-squares procedures. The weights used were the quantities ($1/\sigma_F^2$) where σ_F is defined by Stout & Jensen (1968, equation H.14), and the instability correction was 0·06. This value increases σ_F for reflections with a large $|F|$ and prevents them from controlling the refinement. The R index, defined as $\sum||F_o| - |F_c||/\sum|F_o|$, was 6·8% using 1202 data with $\sin\theta/\lambda < 0{\cdot}55$ Å^{-1}. The Fourier and least-squares programs are part of the Nonius crystallographic package for the PDP 11/45. Scattering factors are from *International Tables for X-ray Crystallography* (1974).