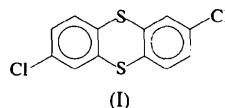


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On comparison of the dihedral angles of 2,7-dichlorothianthrene, 2,7-dimethylthianthrene (Weakley, 1982), perfluorothianthrene (Rainville *et al.*, 1980) and thianthrene itself (Larson *et al.*, 1984), it is evident that the differences are small. The observed dihedral angle is 132.55 (4)° for 2,7-dichlorothianthrene, 131.1 (1)° for 2,7-dimethylthianthrene, 132.0 (1)° for perfluorothianthrene and 127.14 (3)° for thianthrene. These differences could be the result of the 2,7-disubstitution, the inductive effects of halogens or packing considerations. Despite the small magnitude of these differences, some trends can be observed when comparing these compounds. The S···S distance increases to 3.226 (1) Å in 2,7-dichlorothianthrene compared with 3.193 (1) Å in thianthrene. The C—S—C angles in the central ring increase from 101.31 (8) and 101.05 (8)° in 2,7-disubstitution to 100.4 (1) and 99.8 (1)° in thianthrene. These effects seem to be geometrically related to the flattening (increasing dihedral angle) of the thianthrene molecule when 2,7-disubstituted.

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2,7-Dichlorothianthrene

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Abstract

The crystal structure of the title compound, C₁₂H₆Cl₂S₂, has been determined. The dihedral angle for 2,7-dichlorothianthrene is 132.55 (4)°. This value is similar to that found in other 2,7-substituted thianthrenes and also the parent thianthrene.

Comment

This structural investigation was performed as a result of our interest in the effect that substitutions in or on the aromatic rings of thianthrene have on the dihedral angle of the two planes in the title compound, (I), which are defined by one aromatic ring and the two S atoms (Larson *et al.*, 1984).

† Deceased, 29 July 1996.

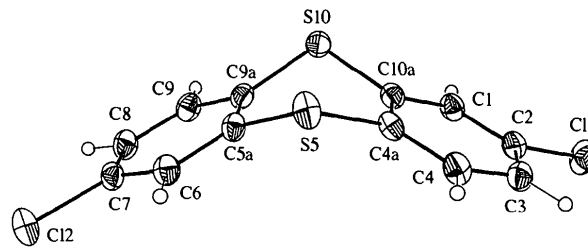


Fig. 1. View of the title compound showing the atom-labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. H atoms are drawn to an arbitrary scale.

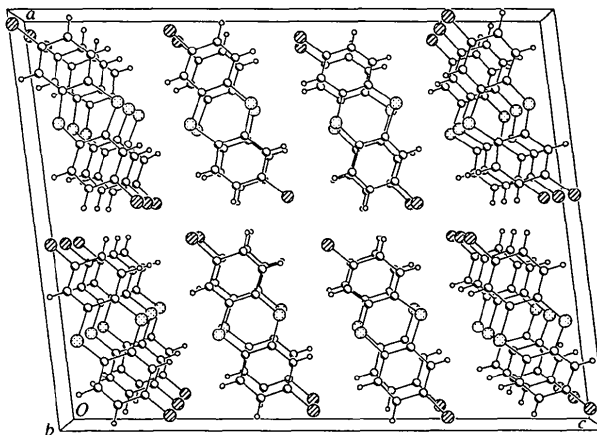


Fig. 2. Packing diagram of the title compound.

Experimental

The title compound was synthesized by Stewart N. Thomas under the supervision of Professor A. L. Ternay Jr.

Crystal data

$C_{12}H_6Cl_2S_2$
 $M_r = 285.19$
 Monoclinic
 $I2/a$
 $a = 21.311(4) \text{ \AA}$
 $b = 3.9080(8) \text{ \AA}$
 $c = 27.581(6) \text{ \AA}$
 $\beta = 97.49(3)^\circ$
 $V = 2277.4(8) \text{ \AA}^3$
 $Z = 8$
 $D_x = 1.664 \text{ Mg m}^{-3}$
 D_m not measured

Mo $K\alpha$ radiation
 $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 45 reflections
 $\theta = 21.6\text{--}29.0^\circ$
 $\mu = 0.900 \text{ mm}^{-1}$
 $T = 163(2) \text{ K}$
 Needle
 $0.25 \times 0.16 \times 0.10 \text{ mm}$
 Colorless

Data collection

Syntex $P2_1$ diffractometer
 ω scans
 Absorption correction:
 analytical from crystal shape
 $T_{\min} = 0.82$, $T_{\max} = 0.91$
 2598 measured reflections
 2598 independent reflections

2146 reflections with
 $I > 2\sigma(I)$
 $\theta_{\max} = 27.51^\circ$
 $h = -26 \rightarrow 27$
 $k = -4 \rightarrow 5$
 $l = -35 \rightarrow 7$
 4 standard reflections
 every 96 reflections
 intensity decay: $< 1\%$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.085$
 $S = 1.05$
 2597 reflections
 169 parameters
 H atoms: see below
 $w = 1/[\sigma^2(F_o^2) + (0.035P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.39 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.41 \text{ e \AA}^{-3}$
 Extinction correction: none
 Scattering factors from
International Tables for Crystallography (Vol. C)

The C—H distances range from 0.91(2) to 0.95(2) Å, while the C—C—H angles range from 118.4(1) to 121.6(1)°. The U_{iso} values of the H atoms vary from 0.0275(5) to 0.046(7) Å².

Data collection: Syntex $P2_1$ software. Cell refinement: *LSCAL* (Harlow, 1975). Data reduction: *INCON* (Riley & Davis, 1976). Program(s) used to solve structure: *SHELXL93* (Sheldrick, 1993). Program(s) used to refine structure: *SHELXL93*. Molecular graphics: *SHELXL93*. Software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1346). Services for accessing these data are described at the back of the journal.

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(S),(E)-5-Methoxycarbonyl-3-triphenylmethylaminohex-4-en-4-olide†

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Abstract

The title compound, $C_{27}H_{25}NO_4$, is the minor product of the Wittig reaction of (S)-N-triphenylmethylaspartic anhydride with the stabilized ylide $\text{Ph}_3\text{P}=\text{C}(\text{Me})\text{CO}_2\text{Me}$. The crystal structure determination unambiguously shows that this product is not the originally proposed Z isomer of the major product (S),(E)-5-methoxycarbonyl-2-triphenylmethylaminohex-4-en-4-olide, but the alternative α -adduct, also with an E configuration at the C4=C5 double bond.

Comment

We have recently shown that the readily available (S)-N-triphenylmethylaspartic anhydride, (1), can be applied in the asymmetric synthesis of amino acid and peptide derivatives through its reactions with a variety of nucleophiles (Athanasopoulos *et al.*, 1995). In particular, the Wittig reaction of anhydride (1) with the stabilized ylide $\text{Ph}_3\text{P}=\text{C}(\text{Me})\text{CO}_2\text{Me}$ produced a mixture of two isomeric enol-lactones in the ratio 6:1.7.

† Alternative name: methyl 2-[5-oxo-3-(triphenylmethylamino)tetrahydrofuran-2-ylidene]propanoate.