moieties are bent away from the epoxide oxygen, tilting the H atoms on C(2) and C(6) away from those on C(12) and C(16), e.g. $H(2)\cdots H(12) = 2\cdot01$ (3) Å. The planes defined by C(1)C(2)C(6) and C(11)C(12)C(16) make an angle of $35\cdot0$ (3)°. In the dioxetane derivative the same planes make an angle of $62\cdot4$°, leading to an increased separation of the $H(2)\cdots H(12)$ type atoms ($2\cdot18$ Å). Because of the greater bending in the dioxetane derivative, the $H(3)\cdots H(13)$ type interactions are reduced to $2\cdot01$ Å as compared with a value of $2\cdot48$ (3) Å in the epoxide.

Except for the epoxide functionality, there is little bond-length or valence-angle strain in adamantylideneadamantane epoxide. Bonds within the adamantane system range from $1\cdot509$ to $1\cdot541$ Å with an average value of $1\cdot524$ (8) Å. This is equivalent to the $1\cdot528$ (5) Å value reported for cyclohexane determined by gas-phase electron diffraction (Davis & Hassel, 1963). The valence angles, excluding those at C(1) and C(11), average $109\cdot4$ (5)° with a range of $108\cdot0$ to $110\cdot2$°. The strain energy for (2) is calculated to be $46\cdot5$ kJ mol^{-1} greater than that in (1) (Allinger & Yuh, 1980).

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Structure of (Z)-4-Methylthiotamoxifen

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Abstract. $C_{27}H_{31}NOS$, $M_r = 417\cdot61$, orthorhombic, $Pna2_1$, $a = 16\cdot720$ (18), $b = 13\cdot741$ (19), $c = 10\cdot527$ (14) Å, $V = 2419$ Å^3, $Z = 4$, $D_x = 1\cdot15$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot71069$ Å, $\mu = 1\cdot44$ cm^{-1}, $F(000) = 894\cdot88$, room temperature, final $R = 0\cdot0449$ for 1454 unique observed reflexions. 4-Methylthiotamoxifen (1) is a second-generation analogue of the antioestrogen tamoxifen (2). Only one of the diastereoisomers of (1) shows antioestrogenic activity whilst the other is a mild oestrogen. The unambiguous identification of the Z-geometry of one diastereoisomer of the alkene (3) by X-ray diffraction confirms a provisional assignment based on NMR and establishes that the antioestrogenic isomer of (1) has the same relative geometry as tamoxifen.

**Introduction.** Tamoxifen (2) is the drug most commonly used for the treatment of advanced disseminated breast cancer (Legha & Carter, 1976). It is a powerful antioestrogen. One of its metabolites, 4-hydroxytamoxifen (4), is 100 times more potent than tamoxifen in antitumour assays *in vitro*, but cannot be used *in vivo* because of its rapid O-glucuronidation and subsequent excretion (Kemp, Adam, Wakeling & Slater, 1983; Jordan, Collins, Rowsby & Prestwich, 1977). Tamoxifen analogues intended as second-generation antioestrogens are therefore designed to block this rapid metabolic oxidation while retaining the enhanced potency associated with a polar group at position 4 of tamoxifen.

The geometry at the central alkene is a key feature of the biological activity of the tamoxifen class of compounds. In general those compounds derived from Z-tamoxifen (2) show antioestrogenic activity whereas E-tamoxifen (5) and its derivatives are oestrogens (Harper & Walpole, 1966, 1967). The configuration of

tamoxifen (2) was originally assigned on the basis of dipole and NMR measurements (Bedford & Richardson, 1966) and was later confirmed by X-ray crystal-structure analysis (Kilbourn & Owston, 1970; Precigoux, Courseille, Geoffre & Hospital, 1979). To date the geometry of known analogues of tamoxifen with only a few exceptions (Kuroda, Cutbush, Neidle & Leung, 1985) has been assigned on the basis of NMR data (Bedford & Richardson, 1966; Allen, Clark & Jordan, 1980).
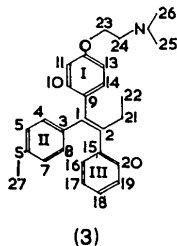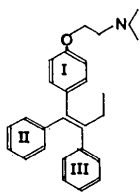
Numbering of carbon atoms in (3) and of rings in (3) and (5).

We have made (Goodman, 1986) a number of analogues of tamoxifen having a sulfur substituent at position 4 and have separated their diastereoisomers. As with other 4-substituted tamoxifen analogues, there is a significant difference between the biological activities of the pairs of isomers, which is of profound importance for further developments in this field. In the case of (1) a provisional assignment of geometry was based on the upfield shift (ca 0·1 p.p.m.) of the A_2B_2 signals of the aminoethane side chain for one of the isomers relative to the other. The isomer with the downfield chemical shifts gave good crystals from petroleum ether (60–80) which were suitable for X-ray structure analysis. We have therefore determined the crystal structure of this isomer to provide a definite assignment of configuration for this class of tamoxifen analogue.

Experimental. 1-(4-Hydroxyphenyl)-1-(4-methylthio-phenyl)-2-phenylbut-1-ene was prepared as a 1:1 mixture of Z- and E-isomers (Goodman, 1986). These were converted by the method of Jarman & McCague (1985) into the mixed heptafluorotolyl ethers, from which one diastereoisomer was purified by fractional crystallization. Removal of the heptafluorotolyl group and alkylation with 2-dimethylaminoethyl chloride provided a single diastereoisomer (3), purified by chromatography on silica (methanol elution) and crystallization from petrol to give a white solid, m.p. 365–366 K, which was characterized by elemental (found C, 77·84; H, 7·65; N, 3·11; S, 7·96%: calculated 77·69, 7·43, 3·36, 7·67%) and mass spectrometric analysis, M/z 562 (M^+). The proton NMR spectrum of this isomer showed an A_2B_2 signal at 2·69 and 4·04 p.p.m. compared with 2·61 and 3·91 for the other isomer.

Table 1. *Atom coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* ($\text{Å}^2 \times 10^3$)

	x	y	z	U_{eq}*
S(1)	4725 (1)	8582 (1)	0	97 (1)
O(1)	4118 (2)	1521 (2)	−198 (3)	84 (1)
N(1)	3026 (2)	331 (3)	−2702 (4)	85 (1)
C(1)	6098 (2)	4636 (3)	1249 (4)	56 (1)
C(2)	6791 (2)	4490 (3)	1856 (4)	57 (1)
C(3)	5783 (2)	5619 (2)	939 (4)	54 (1)
C(4)	6266 (2)	6363 (3)	519 (4)	68 (2)
C(5)	5965 (2)	7264 (3)	229 (4)	69 (2)
C(6)	5166 (2)	7462 (3)	336 (4)	66 (1)
C(7)	4675 (2)	6714 (3)	739 (5)	69 (1)
C(8)	4977 (2)	5808 (3)	1032 (4)	61 (1)
C(9)	5598 (2)	3812 (3)	826 (4)	58 (1)
C(10)	5407 (2)	3667 (3)	−430 (4)	58 (1)
C(11)	4928 (2)	2898 (3)	−819 (4)	59 (1)
C(12)	4616 (2)	2281 (3)	68 (5)	66 (1)
C(13)	4791 (3)	2406 (4)	1310 (5)	94 (2)
C(14)	5275 (3)	3167 (4)	1676 (5)	88 (2)
C(15)	7236 (2)	5288 (3)	2507 (4)	56 (1)
C(16)	6873 (2)	5805 (3)	3462 (4)	62 (1)
C(17)	7266 (3)	6518 (3)	4109 (5)	72 (2)
C(18)	8050 (3)	6741 (3)	3797 (5)	79 (2)
C(19)	8409 (2)	6232 (3)	2857 (5)	75 (2)
C(20)	8022 (2)	5512 (3)	2221 (5)	68 (1)
C(21)	7169 (3)	3507 (3)	2000 (5)	75 (2)
C(22)	7234 (4)	3176 (4)	3363 (7)	109 (2)
C(23)	3904 (2)	1348 (3)	−1477 (4)	74 (2)
C(24)	3270 (3)	587 (3)	−1450 (5)	89 (2)
C(25)	2404 (4)	969 (6)	−3086 (8)	98 (3)
C(25a)	2158 (8)	−57 (19)	−2796 (27)	227 (12)
C(26)	2784 (10)	−691 (8)	−2748 (19)	178 (8)
C(26a)	3444 (11)	−606 (12)	−2948 (20)	125 (7)
C(27)	5531 (3)	9354 (3)	−376 (7)	117 (3)

* U_{eq} defined as one third of the trace of the orthogonalized U_{ij} tensor.

X-ray reflexion data from a plate-shaped fragment broken from the end of a blade-shaped crystal (approx. 0·2 × 0·4 × 0·5 mm) were collected on a Nicolet R3M four-circle automatic diffractometer operating in the $\theta/2\theta$ scan mode and using Mo $K\alpha$ radiation (graphite monochromator, $\lambda = 0·71069$ Å). The unit-cell parameters were determined by least squares from the ω measurements of 25 well centred reflexions. 2451 reflexions were measured and gave $R(\text{merge}) = 0·0045$. An empirical absorption correction was based on azimuthal scans of nine reflexions (324 measurements) and gave maximum and minimum transmission factors of 0·915 and 0·867 respectively. No correction was made for extinction. 1454 unique reflexions in the range $3·5 < 2\theta < 47°$ and having h, k and l positive were found with $|F| > 3\sigma(F)$. Two check reflexions were monitored every 100 reflexions and showed no evidence of decay.

Direct methods yielded a ten-atom fragment and the remaining atoms were found by repeated Fourier expansion. The structure was refined by cascade blocked-diagonal least squares with weights $w_i = 1/[\sigma^2(F_i) + 0·00147F_i^2]$ to a final R of 0·0449 ($wR = 0·0529$). H atoms were inserted at calculated positions and constrained to ride on their adjacent heavy atoms with isotropic thermal factors fixed at 1·2 times the U_{eq} values for their heavy neighbours. All shifts of the 228 refined parameters in the final cycle

Table 2. *Bond lengths* (Å) *and angles* (°)

S(1)—C(6)	1·742 (4)		S(1)—C(27)	1·759 (5)
O(1)—C(12)	1·365 (5)		O(1)—C(23)	1·413 (6)
N(1)—C(24)	1·424 (7)		N(1)—C(25)	1·419 (9)
N(1)—C(25a)	1·549 (16)		N(1)—C(26)	1·463 (12)
N(1)—C(26a)	1·487 (18)		C(1)—C(2)	1·337 (5)
C(1)—C(3)	1·487 (5)		C(1)—C(9)	1·477 (5)
C(2)—C(15)	1·492 (5)		C(2)—C(21)	1·498 (5)
C(3)—C(4)	1·376 (5)		C(3)—C(8)	1·376 (5)
C(4)—C(5)	1·370 (6)		C(5)—C(6)	1·367 (5)
C(6)—C(7)	1·383 (6)		C(7)—C(8)	1·378 (6)
C(9)—C(10)	1·374 (6)		C(9)—C(14)	1·370 (6)
C(10)—C(11)	1·387 (5)		C(11)—C(12)	1·365 (6)
C(12)—C(13)	1·350 (7)		C(13)—C(14)	1·378 (7)
C(15)—C(16)	1·372 (6)		C(15)—C(20)	1·384 (5)
C(16)—C(17)	1·362 (6)		C(17)—C(18)	1·386 (6)
C(18)—C(19)	1·352 (7)		C(19)—C(20)	1·359 (6)
C(21)—C(22)	1·509 (9)		C(23)—C(24)	1·488 (6)
C(25)—C(25a)	1·499 (27)		C(25a)—C(26)	1·363 (24)
C(26)—C(26a)	1·129 (25)			

|				
C(6)—S(1)—C(27)	104·7 (2)		C(12)—O(1)—C(23)	118·6 (3)
C(24)—N(1)—C(25)	108·7 (5)		C(24)—N(1)—C(25a)	114·4 (11)
C(25)—N(1)—C(25a)	60·5 (10)		C(24)—N(1)—C(26)	110·3 (8)
C(25)—N(1)—C(26)	112·4 (8)		C(25a)—N(1)—C(26)	53·7 (11)
C(24)—N(1)—C(26a)	103·9 (8)		C(25)—N(1)—C(26a)	146·0 (9)
C(25a)—N(1)—C(26a)	97·5 (12)		C(26)—N(1)—C(26a)	45·0 (10)
C(2)—C(1)—C(3)	123·3 (3)		C(2)—C(1)—C(9)	121·3 (3)
C(3)—C(1)—C(9)	115·4 (3)		C(1)—C(2)—C(15)	122·7 (3)
C(1)—C(2)—C(21)	123·3 (3)		C(15)—C(2)—C(21)	113·9 (3)
C(1)—C(3)—C(4)	122·5 (3)		C(1)—C(3)—C(8)	120·2 (3)
C(4)—C(3)—C(8)	117·2 (3)		C(3)—C(4)—C(5)	121·8 (3)
C(4)—C(5)—C(6)	121·4 (4)		S(1)—C(6)—C(5)	124·9 (3)
S(1)—C(6)—C(7)	117·9 (3)		C(5)—C(6)—C(7)	117·2 (4)
C(6)—C(7)—C(8)	121·5 (3)		C(3)—C(8)—C(7)	120·9 (3)
C(1)—C(9)—C(10)	122·2 (3)		C(1)—C(9)—C(14)	121·5 (4)
C(10)—C(9)—C(14)	116·3 (4)		C(9)—C(10)—C(11)	121·9 (4)
C(10)—C(11)—C(12)	119·4 (4)		O(1)—C(12)—C(11)	124·6 (4)
O(1)—C(12)—C(13)	115·3 (4)		C(11)—C(12)—C(13)	120·0 (4)
C(12)—C(13)—C(14)	119·6 (5)		C(9)—C(14)—C(13)	122·7 (5)
C(2)—C(15)—C(16)	119·8 (3)		C(2)—C(15)—C(20)	122·5 (4)
C(16)—C(15)—C(20)	117·6 (4)		C(15)—C(16)—C(17)	121·7 (4)
C(16)—C(17)—C(18)	119·8 (4)		C(17)—C(18)—C(19)	118·6 (4)
C(18)—C(19)—C(20)	121·8 (4)		C(15)—C(20)—C(19)	120·5 (4)
C(2)—C(21)—C(22)	113·5 (4)		O(1)—C(23)—C(24)	106·3 (4)
N(1)—C(24)—C(23)	111·1 (4)		N(1)—C(25)—C(25a)	64·1 (7)
N(1)—C(25a)—C(25)	55·4 (8)		N(1)—C(25a)—C(26)	59·9 (8)
C(25)—C(25a)—C(26)	113·5 (12)		N(1)—C(26)—C(25a)	66·4 (11)
N(1)—C(26)—C(26a)	68·7 (11)		C(25a)—C(26)—C(26a)	132·6 (16)
N(1)—C(26a)—C(26)	66·4 (11)			
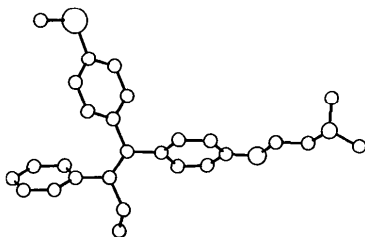
Fig. 1. The molecule of (Z)-4-methylthiotamoxifen (3).

were less than 0·1 of the corresponding e.s.d.'s. A final difference Fourier synthesis showed peaks of up to 0·12 e Å^{-3}, three of which could be interpreted as an ethanol molecule at about 3% population. There is a cavity in the structure which evidently contains some solvent but, because of the low occupancy, no satisfactory refinement could be achieved and the solvent was not included in the final calculations. The final

Table 3. *Dihedral angles* (°) *between the planes of the phenyl rings and that of the ethylenic bond* (*see text*)

Ring	(Z)-4-Methylthiotamoxifen (3)	(E)-Tamoxifen (5)	
		Type A	Type B
I	63·4 (0·5)	52	60
II	43·3 (2·2)	55	49
III	62·6 (1·2)	55	53

analysis of variance against $\sin\theta$ and against $|F|$ showed no unusual features; neither did the normal probability plot. The *SHELXTL* suite of crystallographic programs (Sheldrick, 1983) was used throughout on a NOVA3 computer. Scattering factors, anomalous-dispersion constants and atomic absorption coefficients were taken from *International Tables for X-ray Crystallography* (1974).

The atomic positional parameters are listed in Table 1 and bond lengths and angles in Table 2.* The molecule is illustrated in Fig. 1.

Discussion. There is some disorder of the N-methyl groups, the dominant (60%) conformation being represented by C(25) and C(26), and the remainder (40%) by C(25A) and C(26A). The three rings C(3) to C(8), C(9) to C(14), and C(15) to C(20), show a propeller conformation about the normal to the central plane, C(1) to C(3), C(9), C(15) and C(21), of the molecule.

The dihedral angles between the phenyl rings and the plane of the ethylenic bond are compared in Table 3 with those found for tamoxifen by Kilbourn & Owston (1970). Whilst not strictly comparable with either, the present compound is closer to the conformation of the type B molecules of tamoxifen than to the type A ones.

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Structure of Cyclooctatetraene at 129 K

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Abstract. C_8H_8, $M_r = 104 \cdot 2$, orthorhombic, $Aba2$, $a = 7 \cdot 664$ (6), $b = 7 \cdot 650$ (3), $c = 10 \cdot 688$ (6) Å, $V = 627$ (1) Å^3, $T = 129$ K, $Z = 4$, $D_x = 1 \cdot 10$ Mg m^{-3}, $F(000) = 224$, $\lambda(\text{Mo } K\alpha) = 0 \cdot 71069$ Å, $\mu = 0 \cdot 06$ mm^{-1}, final $R = 0 \cdot 036$ ($wR = 0 \cdot 043$) for 529 observed reflections $[I > 2\sigma(I)]$ [4525 ($\pm h$, $\pm k$, $+l$) measured, 597 averaged reflections (R_{int} $0 \cdot 05$) and 52 refined parameters]. The molecular structure of COT in the solid state confirms earlier electron-diffraction results for the title compound suggesting a boat conformation. This is in accordance with recent structural investigations of COT derivatives.

Introduction. Cyclooctatetraene (COT) ring systems are capable of three structural changes, which involve ring inversion, bond shifting and valence isomerization (Paquette, 1975). Whereas the first process requires a planar transition state only, all bond-shifting mechanisms involve additionally an equalization of bond lengths, and consequently slightly higher energy barriers are found for the latter. As a ligand to transition metals, COT usually adopts a boat form or a twisted boat conformation (Brauer & Krüger, 1976); frequently the ligand shows fluxionality (Mann, 1982). As a dianionic ligand the COT skeleton is planar (Streitwieser *et al.*, 1973; Rösch & Streitwieser, 1978). For free, non-substituted COT a non-planar ring shape was found by electron diffraction (Bastiansen, Hedberg & Hedberg, 1957; Trætteberg, 1966); preliminary results of a crystal structure analysis of COT have been quoted in the literature (Bordener, Parker & Stanford, 1972). A low-temperature X-ray diffraction study of COT was undertaken to reveal basic structural data for this important compound in the solid state.

Experimental. Colourless single crystals of the title compound (m.p. 277·1 K by differential scanning calorimetry) were grown directly on the diffractometer at 248 K from polycrystalline material in capillaries (0·5 mm diameter, 0·01 mm wall thickness) using focused heat radiation techniques, as previously described (Brodalla & Mootz, 1981; Brodalla, 1983; Brodalla, Mootz, Boese & Osswald, 1985). Commercially available equipment was modified to operate under computer control on an Enraf–Nonius CAD-4 diffractometer. Crystal growth on the diffractometer was monitored by photographic techniques. After several attempts at crystallization a suitable crystal chosen for data collection had dimensions 0·14 × 0·29 × 0·36 mm. The crystal was subsequently cooled to 129 K and kept at this temperature for all further measurements. Several Ω scans of low-order reflections along the three crystal axes showed that the crystal displayed acceptable mosaicity. Unit-cell parameters were obtained by a least-squares fit to the θ values of 61 automatically centred reflections ($12 \cdot 6 < \theta < 19 \cdot 7°$). 4525 intensity data (before transformation: $-13 < h < 13$, $-19 < k < 19$, $0 < l < 10$) were measured within the range $1 \cdot 9 < \theta < 39 \cdot 9°$ using graphite-monochromated Mo $K\alpha$ X-radiation by a θ–2θ scan technique in 48 steps, where the time spent measuring the background was half that taken to measure the peak. The intensity of a reflection and its e.s.d. were calculated from $I = \text{INT} - 2(\text{BGL} + \text{BGR})/\text{Lp}$ and $\sigma(I) = [\text{INT} + 4(\text{BGL} + \text{BGR})]^{1/2}/\text{Lp}$, where INT, BGL and BGR are the peak intensity, left and right background counts, respectively. The horizontal detector aperture was $(3 \cdot 0 + 1 \cdot 05\tan\theta)$ mm and the vertical 4 mm; ω-scan width $= (0 \cdot 8 + 0 \cdot 35\tan\theta)°$. The inten-