C1—C7—C7^{i}	111.5 (5)	C1—C6—S6	119.4 (4)
C2—C1—C6	119.1 (5)	C5—C6—S6	120.7 (4)
C2—C1—C7	121.5 (5)	C2—S2—C12	102.2 (3)
C6—C1—C7	119.3 (5)	C62—C12—S2	117.5 (5)
C1—C2—C3	120.5 (5)	C22—C12—S2	122.8 (5)
C1—C2—S2	119.5 (4)	C13—S3—C3	105.0 (3)
C3—C2—S2	119.9 (4)	C63—C13—S3	115.2 (6)
C4—C3—C2	119.7 (5)	C23—C13—S3	124.5 (5)
C4—C3—S3	122.0 (5)	C14—S4—C4	101.3 (3)
C2—C3—S3	118.2 (4)	C64—C14—S4	115.3 (5)
C3—C4—C5	120.2 (5)	C24—C14—S4	124.5 (5)
C3—C4—S4	119.9 (5)	C5—S5—C15	'103.4 (3)
C5—C4—S4	119.9 (5)	C65—C15—S5	122.5 (5)
C4—C5—C6	119.8 (5)	C25—C15—S5	116.0 (5)
C4—C5—S5	122.1 (5)	C16—S6—C6	105.5 (3)
C6—C5—S5	117.9 (5)	C26—C16—S6	124.5 (6)
C1—C6—C5	119.9 (5)	C66—C16—S6	114.8 (7)

C7^{i}—C7—C1—C6	−83.3 (7)	C5—C4—S4—C14	84.6 (5)
C7—C1—C2—S2	4.1 (7)	C4—S4—C14—C24	9.8 (6)
C7—C1—C6—S6	−7.9 (6)	C4—C5—S5—C15	61.2 (5)
C3—C2—S2—C12	62.0 (5)	C5—S5—C15—C65	32.4 (7)
C2—S2—C12—C22	33.2 (5)	C5—C6—S6—C16	−65.4 (5)
C4—C3—S3—C13	−60.2 (5)	C6—S6—C16—C26	−19.0 (6)
C3—S3—C13—C23	−11.7 (6)		

Symmetry codes: (i) $-x, -y, 2-z$.

H atoms were placed geometrically and refined with riding model constraints and $U(\mathrm{H}) = 1.5U_{\mathrm{eq}}(\mathrm{C})$.

Data collection: *CAD-4 EXPRESS Software* (Enraf–Nonius, 1992). Cell refinement: Enraf–Nonius *SET*4 (de Boer & Duisenberg, 1984). Data reduction: *GX* (Mallinson & Muir, 1985). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1971).

We thank the Loudon Bequest for support (SJR) and Ethyl S. A. for a generous gift of 1,2-bis(pentabromophenyl)ethane.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: CF1010). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). **C52**, 212–215

(22*S*)-21-Acetoxy-11*β*-hydroxy-16*α*,17*α*-methylorthoacetoxypregna-1,4-diene-3,20-dione

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(*Received 22 March 1995; accepted 14 July 1995*)

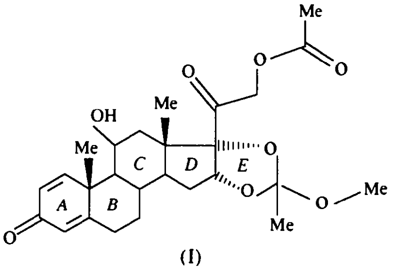
Abstract

In the title compound, methyl (22*S*)-11*β*-hydroxy-16*α*,17*α*-[(methoxy)(methyl)methylenedioxy]pregna-1,4-diene-3,20-dione-21-acetate, $C_{26}H_{34}O_8$, part of the orthoacetate group forms the dioxolane ring (*E*) fused with the steroid skeleton at the C16 and C17 atoms. Ring *A* is planar, rings *B* and *C* adopt chair conformations, while rings *D* and *E* have conformations midway between half-chair and envelope. Intermolecular O11—H···O3 hydrogen bonds are formed. The new chiral center at atom C22 of the dioxolane ring has an *S* configuration and the conformation of the ring, closed at C22, is comparable with the conformations found for the (22*R*) diastereoisomers of similar compounds.

Comment

This investigation is the fourth in a series of structure determinations of pregnadiene derivatives having a dioxolane ring attached to the C16 and C17 atoms. The compound was prepared by Infarm Co. Ltd as part of a study of new glucocorticosteroid drugs. The crystal structure analysis of (22*S*)-21-acetoxy-11*β*-hydroxy-16*α*,17*α*-methylorthoacetoxypregna-1,4-diene-3,20-dione, (I), was undertaken in order to confirm the structural formula and to establish the configuration at the C22 atom, *i.e.* the new chiral center of the molecule. Compounds having a (22*S*) configuration are either biologically less active than their (22*R*) diastereoisomers or inactive.

A view of the molecule is shown in Fig. 1. The bond lengths and valency angles (Table 2) are within the range



(I)

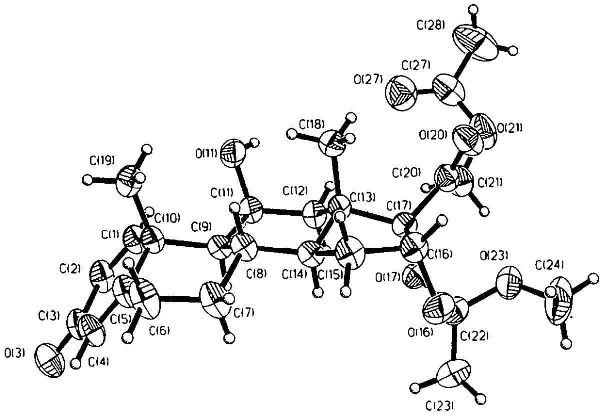


Fig. 1. Perspective drawing of the title molecule. Non-H atoms are shown as 50% probability displacement ellipsoids.

of mean values for steroid structures (Duax & Norton, 1975).

The O3 atom deviates from the plane of ring *A* [planar within 0.007 (2) Å] by 0.045 (5) Å. The 1,4-diene system in ring *A* produces a strong bend towards the α face of the steroid molecule (Duax & Norton, 1975). The plane of ring *A* and the mean plane through atoms C5–C17 form a dihedral angle of 40.1 (1)° . The distance of atom O3 from the C5–C17 plane is 2.384 (4) Å.

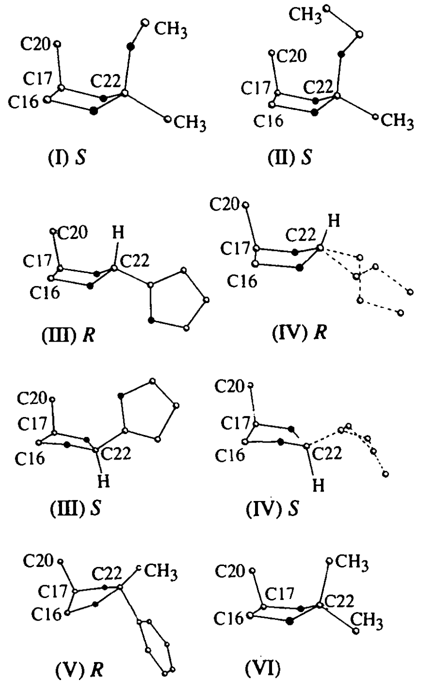
Rings *B* and *C* have chair conformations, ring *C* being slightly flatter, as shown by the smaller mean value of the torsion angles (56.3 and 52.8° for rings *B* and *C*, respectively). The asymmetry parameters (Duax & Norton, 1975) for ring *B* are $\Delta C_2(9,10) = 1.0\,(6)$, $\Delta C_s(9) = 2.8\,(9)$ and $\Delta C_s(5) = 5.9\,(9)^\circ$, and for ring *C* are $\Delta C_2(9,11) = 1.2\,(6)$, $\Delta C_s(9) = 6.2\,(7)$ and $\Delta C_s(12) = 12.5\,(7)^\circ$.

Ring *D* adopts a conformation midway between 14α-envelope and 13β,14α-half-chair. The C13 and C14 atoms lie on opposite sides of the C15–C17 plane at distances of 0.264 (8) and 0.488 (8) Å, respectively. The asymmetry parameter $\Delta C_2(16)$ is 7.9 (6)° and the pseudo-rotation parameters Δ and φ (Altona, Geise & Romers, 1968) are −12.24 (1) and 47.6 (3)°, respectively.

Ring *E* adopts a conformation midway between O17,C22-half-chair and C22-envelope. Atoms O17 and C22 lie on opposite sides of the C16, C17, O16 plane at distances of 0.217 (7) and 0.335 (7) Å, respectively. The asymmetry parameter $\Delta C_2(16)$ is 5.3 (6)° and the pseudo-rotation parameters Δ and φ are 9.87 (1) and 39.6 (3)°, respectively. The ring-junction configurations are as follows: planar for rings *A*/*B*, *trans* for rings *B*/*C* and *C*/*D*, and *cis* for rings *D*/*E*.

In the crystal, the molecules are hydrogen bonded by short O11—H···O3 intermolecular contacts; $\text{H}\cdots\text{O3}^{\text{i}}$ 2.06 (6), $\text{O11}\cdots\text{O3}^{\text{i}}$ 2.809 (4) Å and $\text{O11—H}\cdots\text{O3}^{\text{i}}$ 150 (6)° [symmetry code: (i) $-x, -\frac{1}{2}+y, -z$].

The crystal and molecular structure of the title compound (I) is quite similar to that found for the corresponding (22*S*)-ethyl orthoacetate [(II); Pniewska, Anulewicz & Uszycka-Horawa, 1995]. Although the configuration at the C22 atom was found to be *S*, the conformation of the dioxolane ring *E* in both (22*S*)-orthoacetates agrees better with those found for (22*R*)-21-acetoxy-16α,17α-furfurylidenedioxy-11β-hydroxy-1,4-pregnadiene-3,20-dione [(III); Pniewska, Anulewicz, Uszycka-Horawa & Kroszczyński, 1995] and (22*R*)-11β,21-dihydroxy-16α,17α-propylmethylenedioxy-1,4-pregnadiene-3,20-dione [(IV); Albertsson, Oskarsson & Svensson, 1978], than with those of their less active (22*S*) diastereoisomers. A comparison of torsion angles is listed in Table 3 and corresponding fragments are shown in Fig. 2.


(I) *S* (II) *S*

(III) *R* (IV) *R*

(III) *S* (IV) *S*

(V) *R* (VI)

Fig. 2. Dioxolane ring *E* fragments with O atoms represented as filled circles. The C22 configuration is indicated as *R* or *S*. (See Table 3 for torsion-angle values.)

This observation can be explained from the point of view of steric interaction. More bulky substituents [methyl in (I) and (II), furyl in (III), propyl in (IV)] distort ring *E* towards the C22-envelope conformation. In the structure of 9α-fluoro-11β,21-dihydroxy-16α,17α-isopropylidenedioxy-1,4-prenadiene-3,20 dione [(VI); Surcouf, 1979], with two methyl groups at atom C22, ring *E* has a nearly ideal envelope conformation with the mirror plane passing through the middle of the C16—C17 bond and atom C22. The bend of ring *E* is as for compounds (I) and (II) (Table 3, Fig. 2) and this may confirm the steric effect of the methyl group. The type and arrangement of the substituents at atom C22 and the type of substituents at C17 deform the C22-envelope conformation in the other compounds. In the structure of (22*R*)-21-bromo-9α-fluoro-11β-hydroxy-16α,17α-methylphenylmethylenedioxy-4-pregnene-3,20-dione [(V); Krakower, Keeler & Gougoutas, 1971], the C17, O17, C22, O16 fragment is nearly planar (Table 3, Fig. 2) as two bulky groups are substituted at C22, with ring *E* adopting a C16-envelope conformation.

Experimental

The title compound was obtained from Infarm Co. Ltd and crystals were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{26}H_{34}O_8$
$M_r = 474.53$
Monoclinic
$P2_1$
$a = 7.524\ (2)$ Å
$b = 14.819\ (3)$ Å
$c = 11.763\ (2)$ Å
$\beta = 107.26\ (3)^\circ$
$V = 1252.5\ (5)$ Å^3
$Z = 2$
$D_x = 1.258$ Mg m^{-3}

Cu *K*α radiation
$\lambda = 1.54178$ Å
Cell parameters from 40 reflections
$\theta = 17\text{–}26^\circ$
$\mu = 0.765$ mm^{-1}
$T = 293\ (2)$ K
Prism
0.52 × 0.46 × 0.30 mm
Colorless

Data collection

Kuma KM-4 computer-controlled four-circle diffractometer
ω/θ scans
Absorption correction: none
2529 measured reflections
2474 independent reflections
2304 observed reflections [$I > 2\sigma(I)$]

$R_{\text{int}} = 0.1068$
$\theta_{\max} = 80.32^\circ$
$h = -8 \rightarrow 8$
$k = -16 \rightarrow 0$
$l = 0 \rightarrow 14$
3 standard reflections monitored every 100 reflections intensity decay: <1%

Refinement

Refinement on F^2
$R(F) = 0.0475$
$wR(F^2) = 0.1355$
$S = 1.052$
2474 reflections
345 parameters
$w = 1/[\sigma^2(F_o^2) + (0.1130P)^2 + 0.0314P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.022$
$\Delta\rho_{\max} = 0.275$ e Å^{-3}
$\Delta\rho_{\min} = -0.203$ e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0094 (15)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983) parameter = −0.14 (26)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\text{eq}} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}
O3	0.2169 (4)	0.9211 (2)	−0.1004 (2)	0.0557 (6)
O11	0.1108 (4)	0.5050 (2)	0.0865 (2)	0.0499 (6)
O16	0.7627 (3)	0.5356 (2)	0.5937 (2)	0.0515 (6)
O17	0.4532 (3)	0.5491 (1)	0.5432 (2)	0.0377 (4)
O20	0.4650 (4)	0.3147 (2)	0.5235 (2)	0.0544 (6)
O21	0.1256 (4)	0.3269 (2)	0.5666 (3)	0.0631 (8)
O23	0.5968 (4)	0.4886 (2)	0.7203 (2)	0.0590 (7)
O27	0.0254 (5)	0.3249 (2)	0.3691 (3)	0.0713 (8)
C1	0.1175 (4)	0.6962 (2)	−0.0346 (3)	0.0421 (6)
C2	0.0870 (4)	0.7815 (2)	−0.0727 (3)	0.0462 (7)
C3	0.2418 (4)	0.8411 (2)	−0.0697 (3)	0.0412 (7)
C4	0.4284 (4)	0.8036 (2)	−0.0265 (3)	0.0440 (7)
C5	0.4607 (4)	0.7185 (2)	0.0107 (3)	0.0395 (6)
C6	0.6549 (5)	0.6814 (3)	0.0610 (3)	0.0510 (8)
C7	0.6838 (4)	0.6448 (2)	0.1861 (3)	0.0461 (7)
C8	0.5314 (4)	0.5790 (2)	0.1947 (3)	0.0341 (5)
C9	0.3405 (3)	0.6248 (2)	0.1450 (2)	0.0303 (5)
C10	0.3057 (4)	0.6536 (2)	0.0111 (3)	0.0373 (6)
C11	0.1750 (4)	0.5756 (2)	0.1702 (2)	0.0340 (5)
C12	0.2208 (3)	0.5414 (2)	0.2995 (2)	0.0331 (5)
C13	0.4044 (4)	0.4899 (2)	0.3406 (2)	0.0308 (5)
C14	0.5587 (3)	0.5524 (2)	0.3231 (2)	0.0319 (5)
C15	0.7385 (4)	0.5023 (2)	0.3894 (3)	0.0412 (6)
C16	0.6966 (4)	0.4691 (2)	0.5018 (3)	0.0401 (6)
C17	0.4817 (4)	0.4716 (2)	0.4762 (2)	0.0331 (5)
C18	0.3947 (5)	0.3996 (2)	0.2747 (3)	0.0425 (6)
C19	0.3096 (6)	0.5741 (3)	−0.0741 (3)	0.0531 (8)
C20	0.3984 (4)	0.3891 (2)	0.5207 (2)	0.0394 (6)
C21	0.2255 (5)	0.4071 (3)	0.5587 (3)	0.0527 (8)
C22	0.6165 (5)	0.5560 (2)	0.6399 (3)	0.0458 (7)
C23	0.6361 (7)	0.6496 (3)	0.6917 (4)	0.068 (1)
C24	0.7626 (9)	0.4651 (5)	0.8112 (5)	0.098 (2)
C27	0.0270 (5)	0.2914 (3)	0.4624 (4)	0.0579 (9)
C28	−0.0789 (9)	0.2101 (4)	0.4787 (9)	0.102 (2)

Table 2. *Selected geometric parameters (Å, °)*

O3—C3	1.237 (4)	C6—C7	1.523 (5)
O11—C11	1.420 (4)	C7—C8	1.531 (4)
O16—C22	1.396 (5)	C8—C14	1.514 (4)
O16—C16	1.438 (4)	C8—C9	1.538 (3)
O17—C22	1.409 (3)	C9—C11	1.546 (3)
O17—C17	1.444 (3)	C9—C10	1.577 (4)
O23—C22	1.414 (4)	C11—C12	1.543 (4)
O23—C24	1.424 (5)	C12—C13	1.525 (3)
O27—C27	1.201 (5)	C13—C14	1.546 (3)
C1—C2	1.338 (5)	C13—C17	1.551 (3)
C1—C10	1.497 (4)	C14—C15	1.538 (3)
C2—C3	1.454 (4)	C15—C16	1.528 (4)
C3—C4	1.454 (4)	C16—C17	1.555 (4)
C4—C5	1.335 (4)	C17—C20	1.535 (4)
C5—C6	1.507 (4)	C22—C23	1.505 (5)
C5—C10	1.512 (4)		
C22—O16—C16	107.7 (2)	C12—C11—C9	112.8 (2)
C22—O17—C17	105.2 (2)	C13—C12—C11	113.2 (2)
C22—O23—C24	115.6 (4)	C12—C13—C14	107.6 (2)
C2—C1—C10	124.6 (3)	C12—C13—C17	116.2 (2)
C1—C2—C3	120.6 (3)	C14—C13—C17	100.0 (2)
O3—C3—C2	121.6 (3)	C8—C14—C15	118.1 (2)
O3—C3—C4	121.0 (3)	C8—C14—C13	113.8 (2)
C2—C3—C4	117.4 (3)	C15—C14—C13	103.0 (2)
C5—C4—C3	122.6 (3)	C16—C15—C14	102.6 (2)
C4—C5—C6	122.3 (3)	O16—C16—C15	108.4 (3)
C4—C5—C10	122.4 (3)	O16—C16—C17	103.7 (2)
C6—C5—C10	115.2 (3)	C15—C16—C17	107.1 (2)
C5—C6—C7	110.7 (2)	O17—C17—C20	107.8 (2)
C6—C7—C8	113.3 (3)	O17—C17—C13	110.6 (2)
C14—C8—C7	110.4 (2)	C20—C17—C13	115.0 (2)
C14—C8—C9	109.2 (2)	O17—C17—C16	103.2 (2)
C7—C8—C9	109.1 (2)	C20—C17—C16	114.9 (2)
C8—C9—C11	115.7 (2)	C13—C17—C16	104.7 (2)
C8—C9—C10	111.0 (2)	O16—C22—O17	105.6 (2)
C11—C9—C10	114.5 (2)	O16—C22—O23	111.0 (3)
C1—C10—C5	112.3 (2)	O17—C22—O23	104.5 (2)
C1—C10—C9	109.4 (2)	O16—C22—C23	111.3 (3)
C5—C10—C9	106.0 (2)	O17—C22—C23	110.5 (3)
O11—C11—C12	111.8 (2)	O23—C22—C23	113.4 (3)
O11—C11—C9	110.0 (2)		

Table 3. *Torsion angles (°) in ring E of the title compound compared with corresponding angles in related compounds*

C22 configuration	(I) S^a	(II) S^b	(III) R^c	(IV) R^d	(III) S^c	(IV) S^d	(V) S^e	(VI)^f
C22—O16—C16—C17	14.6	13.9	18.7	12.0	−24.8	−33.2	−30.8	15.2
C22—O17—C17—C16	−28.9	−29.1	−27.3	−28.3	−5.5	−2.8	−15.6	−18.8
O16—C16—C17—O17	8.9	9.6	5.4	10.4	18.2	21.9	26.9	2.2
C16—O16—C22—O17	−33.7	−33.3	−36.8	−30.3	21.9	32.7	23.3	−26.9
C17—O17—C22—O16	39.5	39.5	40.3	36.9	−9.2	−17.8	−3.6	28.0
Mean e.s.d.'s	0.3	0.5	0.4	0.5	0.5	0.8	3.6	0.6

References: (*a*) present work; (*b*) Pniewska, Anulewicz & Uszycka-Horawa (1995); (*c*) Pniewska, Anulewicz, Uszycka-Horawa & Kroszczyński (1995); (*d*) Albertsson, Oskarsson & Svensson (1978); (*e*) Krakower, Keeler & Gougoutas (1971); (*f*) Surcouf (1979), C22 is not chiral.

The absolute configuration was assigned to agree with the known configuration of the main steroid skeleton. The hydroxyl H110 atom was found in a difference map and refined isotropically. All other H atoms were refined with riding models.

Data collection: *Kuma KM*-4 *Software* (Kuma, 1991). Cell refinement: *Kuma KM*-4 *Software*. Data reduction: *Kuma KM*-4 *Software*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL*93.

The authors wish to express their thanks to Professor Z. Gałdecki for his kind consent to use the *SHELXTL* program for creating drafts. The crystallographic studies were supported by Grant No. 2 P303 077 07 from the Polish State Committee for Scientific Research.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1177). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5-*tert*-Butylthiazolidine-2-thione and 5-Phenylthiazolidine-2-thione

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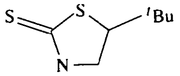
(*Received 22 May 1995; accepted 31 July 1995*)

Abstract

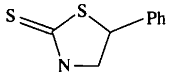
In the title compounds, $C_7H_{13}NS_2$, (I), and $C_9H_9NS_2$, (II), the heterocycle adopts a half-chair conformation with a torsion angle τ = S1—C5—C4—N3 equal to 27.5 (4)° in (I) and 30.0 (3)° in (II). In both crystals, a hydrogen bond between the thione group and the N atom is observed, the corresponding S2···N3 distance being equal to 3.367 (8) Å in (I) and 3.376 (9) Å in (II).

Comment

This report completes a series of papers devoted to a conformational study of the substituted thiazolidine-2-thione family, hereafter TH-2 TH (Laknifli, Pierrot, Chanon & Chanon, 1995, and references therein). The present paper is concerned with two derivatives in position 5: 5-*tert*-butyl-TH-2 TH, compound (I) and 5-phenyl-TH-2 TH, compound (II).



(I)



(II)

An *ORTEP*II (Johnson, 1976) view of the molecules and the atom-numbering schemes is shown in Fig. 1. Bond distances and angles are comparable with