LS. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN FINISH*. Literature search: *CSSR* (1984).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71596 (36 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1059]

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Structures of *ap*-8-Bromo-1,4-dimethyl-9-(2-methylbenzyl)triptycene and *sc**(9*S**)-8-Bromo-1,4-dimethyl-9-(2-methylbenzyl)triptycene

Takashi Nemoto, Taizo Ono, Akira Uchida
and Yuji Ohashi

Department of Chemistry, Tokyo Institute of Technology, 2-12-1 O-okayama, Meguro-ku, Tokyo 152, Japan

Gaku Yamamoto

Department of Chemistry, The University of Tokyo, 7-3-1 Hongo, Bunkyo-ku, Tokyo 113, Japan

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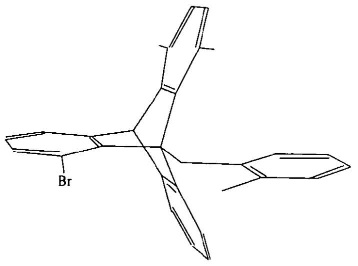
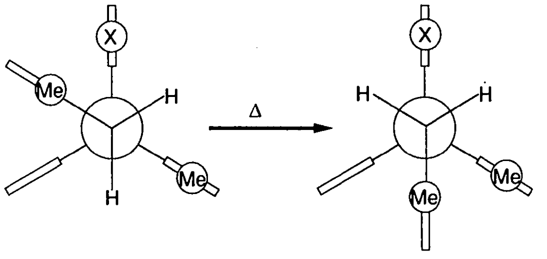
Abstract

Two atropisomers, *ap* and *sc**(9*S**), of the title compound [8-bromo-1,4-dimethyl-9-(2-methylbenzyl)-9,10-dihydro-9,10-*o*-benzenoanthracene, $C_{30}H_{25}Br$] have been isolated and their structures determined by X-ray diffraction. The molecular structures confirm those assumed by NMR study. The crystal of the *sc**(9*S**) isomer decomposed during the transition to the *ap* form at 457.3 K, probably because the two crystal structures are quite different to each other.

Comment

Atropisomerism is defined as stereoisomerism due to restricted rotation about single bonds where the isomers can actually be isolated (Eliel, 1962). Several examples of atropisomerism have been found in 9-substituted triptycene derivatives (Iwamura, 1973; Yamamoto & Oki, 1975; Otsuka, Yamamoto, Mitsuhashi & Oki, 1979; Otsuka, Mitsuhashi & Oki, 1980; Yamamoto, Suzuki & Oki, 1981; Yonemoto, Kakizaki, Yamamoto, Nakamura & Oki, 1985; Tanaka, Yonemoto, Nakai, Yamamoto & Oki, 1988; Oki, Tanuma, Tanaka & Yamamoto, 1988). Recently 8-chloro- and 8-bromo-1,4-dimethyl-9-(2-methylbenzyl)triptycene were synthesized (Yamamoto, 1990, 1991). These compounds have three rotational isomers, *ap*, *sc**(9*S**) and *sc**(9*R**), as shown below. The *sc**(9*R**) form may be less stable since the 2-methylphenyl group lies between two *peri* substituents. From the NMR study, the isolated isomers were assumed to be the *ap* and *sc**(9*S**) forms. The differential scanning calorimetry (DSC) measurements performed when the *sc**(9*S**) isomer was heated, produced a weak broad endothermic peak at 457.3 K and then a sharp exothermic peak due to melting at 533.5 K. The endothermic peak was assigned to the transition from the *sc**(9*S**) isomer to the *ap* form by NMR measurements. Since no change in appearance of the crystalline samples was observed during the transition, it was assumed that the transition may occur with retention of the crystal form (Yamamoto, Nemoto & Ohashi, 1992). The molecular structures shown in Fig. 1 confirm those assumed by NMR

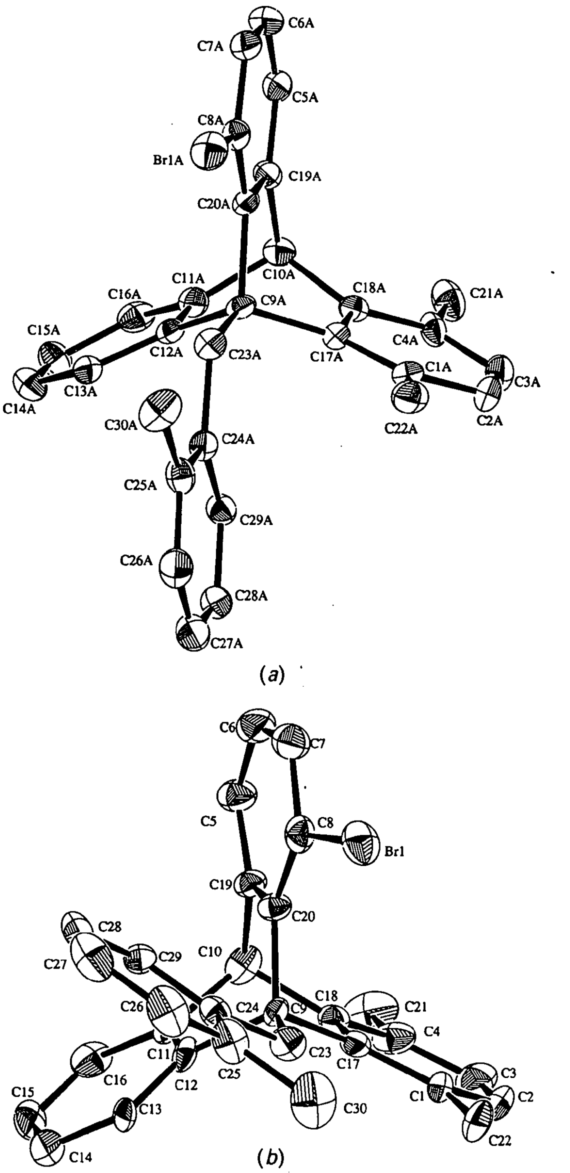




sc
Isomer (II)

ap
Isomer (I)

study. In the *ap* form, there are two crystallographically independent molecules *A* and *B*. In each molecule the 2-methylbenzyl group occupies the opposite position to the bromophenyl moiety. In the *sc**(9*S**) form, on the other hand, the 2-methylphenyl group is directly opposite the dimethylphenyl moiety. The crystal structures of the *ap* and *sc**(9*S**) forms are quite different to each other. This suggests that the crystal of the *sc**(9*S**) form is destroyed when it is transformed to the *ap* form upon heating. In order to ascertain this, the crystal was warmed to 473 K on the four-circle diffractometer using a hot-air flow method. The crystal decomposed and the diffraction peaks disappeared.



(*a*)

(*b*)

Fig. 1. (*a*) A perspective view of molecule *A* of the *ap* rotamer (30% probability) with the numbering scheme. (*b*) A perspective view of a molecule of the *sc* rotamer (30% probability) with the numbering scheme.

Experimental

Isomer (I) (*ap*)

Crystal data

$C_{30}H_{25}Br$
$M_r = 465.43$
Triclinic
$P\bar{1}$
$a = 10.070\ (1)$ Å
$b = 15.925\ (1)$ Å
$c = 15.582\ (2)$ Å
$\alpha = 115.032\ (9)^\circ$
$\beta = 102.59\ (2)^\circ$
$\gamma = 78.09\ (1)^\circ$
$V = 2191.\ (1)$ Å^3
$Z = 4$
$D_x = 1.411$ Mg m^{-3}

$D_m = 1.413$ Mg m^{-3}
Density measured by flotation in C_6H_{12}/CCl_4
Cu *Kα* radiation
$\lambda = 1.5418$ Å
Cell parameters from 21 reflections
$\theta = 20.85\text{–}30.40^\circ$
$\mu = 2.650$ mm^{-1}
$T = 296$ K
Plate like crystal
0.4 × 0.3 × 0.2 mm
Colorless

Data collection

Rigaku AFC-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
7082 measured reflections
6911 independent reflections
5098 observed reflections [$F > 4\sigma(F)$]

$\theta_{\max} = 62.5^\circ$
$h = -11 \rightarrow 11$
$k = -18 \rightarrow 17$
$l = 0 \rightarrow 17$
3 standard reflections monitored every 50 reflections
intensity variation: none

Refinement

Refinement on F
$R = 0.061$
$wR = 0.068$
$S = 0.874$
4470 reflections
673 parameters
$w = 1/[\sigma^2(F) + 0.003413F^2]$
$(\Delta/\sigma)_{\max} = 0.013$

$\Delta\rho_{\max} = 0.73$ e Å^{-3}
$\Delta\rho_{\min} = -0.59$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Only the coordinates of the H atoms were refined except for the H atoms attached to C5*A*, C21*A*, C22*A*, C27*A*, C30*A*, C21*B*, C29*B*, C30*B*, whose positions were obtained geometrically and not refined. Program(s) used to solve structure: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular graphics: *ORTEP*II (Johnson, 1976).

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

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

	x	y	z	U_{eq}
Br1*A*	0.11927 (9)	0.38203 (6)	0.95075 (6)	0.0525
C1*A*	0.2880 (7)	0.4128 (5)	0.6810 (5)	0.0378
C2*A*	0.3957 (8)	0.4106 (6)	0.6374 (5)	0.0512
C3*A*	0.4677 (8)	0.4872 (6)	0.6686 (6)	0.0504
C4*A*	0.4367 (6)	0.5673 (5)	0.7467 (5)	0.0386
C5*A*	0.3902 (7)	0.6197 (5)	1.0388 (5)	0.0448
C6*A*	0.3930 (8)	0.5724 (6)	1.0954 (6)	0.0557
C7*A*	0.3103 (8)	0.5036 (6)	1.0650 (5)	0.0473
C8*A*	0.2228 (7)	0.4811 (5)	0.9779 (5)	0.0384
C9*A*	0.1320 (7)	0.5184 (5)	0.8182 (4)	0.0323
C10*A*	0.2948 (7)	0.6464 (5)	0.8849 (5)	0.0357
C11*A*	0.1488 (7)	0.6861 (5)	0.8703 (5)	0.0379
C12*A*	0.0586 (6)	0.6189 (4)	0.8360 (4)	0.0309
C13*A*	−0.0799 (7)	0.6479 (5)	0.8298 (5)	0.0385

C14*A*	−0.1296 (8)	0.7416 (6)	0.8518 (5)	0.0482
C15*A*	−0.0413 (9)	0.8059 (6)	0.8805 (6)	0.0547
C16*A*	0.0989 (8)	0.7799 (5)	0.8919 (5)	0.0441
C17*A*	0.2480 (6)	0.4968 (4)	0.7561 (4)	0.0306
C18*A*	0.3306 (6)	0.5690 (5)	0.7917 (5)	0.0342
C19*A*	0.3032 (7)	0.5979 (5)	0.9517 (5)	0.0350
C20*A*	0.2157 (6)	0.5286 (4)	0.9185 (4)	0.0303
C21*A*	0.5210 (8)	0.6472 (6)	0.7857 (7)	0.0576
C22*A*	0.2287 (8)	0.3209 (5)	0.6411 (5)	0.0488
C23*A*	0.0242 (8)	0.4500 (5)	0.7780 (5)	0.0361
C24*A*	−0.0625 (6)	0.4354 (4)	0.6812 (4)	0.0315
C25*A*	−0.1524 (7)	0.3681 (5)	0.6470 (5)	0.0387
C26*A*	−0.2304 (8)	0.3494 (6)	0.5578 (6)	0.0510
C27*A*	−0.2233 (8)	0.3958 (6)	0.5012 (6)	0.0576
C28*A*	−0.1331 (8)	0.4617 (5)	0.5352 (5)	0.0454
C29*A*	−0.0545 (7)	0.4804 (5)	0.6244 (5)	0.0380
C30*A*	−0.1665 (9)	0.3153 (6)	0.7044 (6)	0.0582
Br1*B*	−0.42267 (9)	0.12183 (6)	0.54388 (6)	0.0552
C1*B*	−0.0565 (7)	0.0913 (5)	0.8177 (5)	0.0360
C2*B*	0.0808 (8)	0.0912 (6)	0.8581 (5)	0.0460
C3*B*	0.1774 (8)	0.0130 (6)	0.8275 (5)	0.0471
C4*B*	0.1427 (7)	−0.0681 (5)	0.7513 (5)	0.0400
C5*B*	−0.0604 (8)	−0.1195 (6)	0.4611 (5)	0.0441
C6*B*	−0.1218 (9)	−0.0722 (7)	0.4032 (6)	0.0567
C7*B*	−0.2280 (9)	−0.0031 (6)	0.4317 (5)	0.0515
C8*B*	−0.2743 (7)	0.0224 (5)	0.5197 (5)	0.0384
C9*B*	−0.2389 (6)	−0.0121 (4)	0.6802 (4)	0.0298
C10*B*	−0.0423 (7)	−0.1452 (5)	0.6140 (5)	0.0352
C11*B*	−0.1588 (6)	−0.1817 (5)	0.6288 (5)	0.0332
C12*B*	−0.2645 (6)	−0.1102 (5)	0.6637 (5)	0.0322
C13*B*	−0.3840 (7)	−0.1357 (5)	0.6712 (5)	0.0365
C14*B*	−0.3944 (8)	−0.2278 (5)	0.6485 (5)	0.0402
C15*B*	−0.2864 (8)	−0.2978 (6)	0.6179 (5)	0.0455
C16*B*	−0.1646 (8)	−0.2745 (5)	0.6085 (5)	0.0399
C17*B*	−0.0945 (7)	0.0078 (4)	0.7412 (4)	0.0319
C18*B*	0.0063 (6)	−0.0668 (4)	0.7078 (5)	0.0320
C19*B*	−0.1058 (7)	−0.0963 (5)	0.5466 (4)	0.0317
C20*B*	−0.2124 (6)	−0.0257 (5)	0.5782 (4)	0.0314
C21*B*	0.2502 (8)	−0.1510 (6)	0.7167 (6)	0.0544
C22*B*	−0.1450 (9)	0.1840 (5)	0.8575 (6)	0.0437
C23*B*	−0.3616 (7)	0.0585 (5)	0.7188 (5)	0.0353
C24*B*	−0.3955 (6)	0.0729 (5)	0.8142 (5)	0.0355
C25*B*	−0.5089 (7)	0.1383 (5)	0.8487 (5)	0.0423
C26*B*	−0.5397 (9)	0.1539 (6)	0.9371 (6)	0.0547
C27*B*	−0.4648 (10)	0.1080 (6)	0.9923 (6)	0.0607
C28*B*	−0.3535 (9)	0.0438 (6)	0.9578 (6)	0.0516
C29*B*	−0.3194 (7)	0.0263 (5)	0.8700 (5)	0.0432
C30*B*	−0.5966 (8)	0.1908 (6)	0.7894 (6)	0.0612

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

C9*A*—C12*A*	1.553 (9)	C9*B*—C12*B*	1.540 (11)
C9*A*—C17*A*	1.571 (10)	C9*B*—C17*B*	1.565 (8)
C9*A*—C20*A*	1.564 (9)	C9*B*—C20*B*	1.585 (10)
C9*A*—C23*A*	1.541 (11)	C9*B*—C23*B*	1.527 (9)
C10*A*—C11*A*	1.486 (9)	C10*B*—C11*B*	1.511 (12)
C10*A*—C18*A*	1.512 (8)	C10*B*—C18*B*	1.527 (8)
C10*A*—C19*A*	1.517 (12)	C10*B*—C19*B*	1.521 (11)
C11*A*—C12*A*	1.399 (10)	C11*B*—C12*B*	1.398 (8)
C17*A*—C18*A*	1.405 (10)	C17*B*—C18*B*	1.387 (8)
C19*A*—C20*A*	1.408 (10)	C19*B*—C20*B*	1.387 (8)
C12*A*—C9*A*—C17*A*	108.5 (6)	C12*B*—C9*B*—C17*B*	109.4 (5)
C12*A*—C9*A*—C20*A*	103.1 (4)	C12*B*—C9*B*—C20*B*	102.9 (5)
C12*A*—C9*A*—C23*A*	109.3 (5)	C12*B*—C9*B*—C23*B*	109.0 (6)
C17*A*—C9*A*—C20*A*	102.5 (5)	C17*B*—C9*B*—C20*B*	101.4 (5)
C17*A*—C9*A*—C23*A*	116.5 (5)	C17*B*—C9*B*—C23*B*	116.9 (4)
C20*A*—C9*A*—C23*A*	115.8 (7)	C20*B*—C9*B*—C23*B*	116.1 (6)
C11*A*—C10*A*—C18*A*	109.1 (5)	C11*B*—C10*B*—C18*B*	108.6 (6)
C11*A*—C10*A*—C19*A*	106.0 (7)	C11*B*—C10*B*—C19*B*	105.9 (5)
C18*A*—C10*A*—C19*A*	105.0 (6)	C18*B*—C10*B*—C19*B*	104.3 (6)
C10*A*—C11*A*—C12*A*	112.9 (6)	C10*B*—C11*B*—C12*B*	111.6 (6)
C9*A*—C12*A*—C11*A*	113.6 (5)	C9*B*—C12*B*—C11*B*	115.0 (6)
C9*A*—C17*A*—C18*A*	112.4 (5)	C9*B*—C17*B*—C18*B*	112.1 (5)
C10*A*—C18*A*—C17*A*	113.1 (7)	C10*B*—C18*B*—C17*B*	114.1 (5)
C10*A*—C19*A*—C20*A*	114.5 (5)	C10*B*—C19*B*—C20*B*	114.8 (6)
C9*A*—C20*A*—C19*A*	111.0 (7)	C9*B*—C20*B*—C19*B*	111.4 (6)

Isomer (II) (*sc*)

Crystal data

$C_{30}H_{25}Br$
M_r = 465.43
Tetragonal
$I4_1/a$
a = 27.746 (2) Å
c = 11.817 (3) Å
V = 9097 (2) Å^3
Z = 16
D_x = 1.36 Mg m^{-3}
D_m = 1.36 Mg m^{-3}
Density measured by flotation in C_6H_{12}/CCl_4

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 12.47–14.85°
μ = 1.80 mm^{-1}
T = 296 K
Cubic
0.30 × 0.30 × 0.25 mm
Colorless

Data collection

Rigaku AFC-5*R* diffractometer
ω scans
Absorption correction: none
5706 measured reflections
5482 independent reflections
2011 observed reflections [$F > 3\sigma(F)$]

θ_{max} = 27.5°
$h = 0 \rightarrow 36$
$k = 0 \rightarrow 36$
$l = 0 \rightarrow 15$
3 standard reflections monitored every 100 reflections intensity variation: none

Refinement

Refinement on F
R = 0.083
wR = 0.063
S = 1.68
2011 reflections
328 parameters
$w = 1/[\sigma^2(F) + 0.0034F^2]$
$(\Delta/\sigma)_{max}$ = 0.07

$\Delta\rho_{max}$ = 1.03 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

The H atoms were refined with fixed isotropic temperature factors except those of the Me groups, whose positions were obtained geometrically and not refined. Program(s) used to solve structure: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976).

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for isomer* (II)

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

	x	y	z	U_{eq}
Br1	0.27760 (4)	0.18533 (4)	−0.23887 (10)	0.0606
C1	0.2462 (4)	0.2719 (4)	0.0990 (8)	0.0482
C2	0.2365 (4)	0.3205 (5)	0.1341 (10)	0.0601
C3	0.2711 (5)	0.3541 (5)	0.1505 (9)	0.0604
C4	0.3183 (4)	0.3439 (4)	0.1339 (8)	0.0503
C5	0.4079 (4)	0.2824 (4)	−0.1377 (10)	0.0490
C6	0.3987 (5)	0.2685 (4)	−0.2465 (12)	0.0639
C7	0.3604 (4)	0.2386 (4)	−0.2746 (9)	0.0537
C8	0.3326 (3)	0.2200 (3)	−0.1838 (9)	0.0446
C9	0.3185 (3)	0.2130 (3)	0.0377 (7)	0.0283
C10	0.3810 (4)	0.2818 (4)	0.0647 (10)	0.0472
C11	0.3926 (3)	0.2388 (3)	0.1398 (8)	0.0361
C12	0.3592 (3)	0.2015 (3)	0.1261 (8)	0.0338
C13	0.3635 (4)	0.1609 (3)	0.1910 (9)	0.0405
C14	0.4012 (5)	0.1564 (4)	0.2667 (10)	0.0566
C15	0.4345 (4)	0.1931 (5)	0.2779 (9)	0.0586
C16	0.4301 (4)	0.2347 (5)	0.2135 (9)	0.0520

C17	0.2946 (4)	0.2609 (3)	0.0810 (7)	0.0361
C18	0.3304 (3)	0.2976 (3)	0.0974 (7)	0.0360
C19	0.3783 (3)	0.2640 (3)	−0.0552 (8)	0.0353
C20	0.3427 (3)	0.2298 (3)	−0.0733 (8)	0.0327
C21	0.3563 (5)	0.3822 (4)	0.1536 (10)	0.0787
C22	0.2021 (3)	0.2387 (4)	0.0881 (9)	0.0502
C23	0.2839 (4)	0.1696 (4)	0.0275 (8)	0.0427
C24	0.3038 (3)	0.1217 (3)	−0.0180 (8)	0.0365
C25	0.2710 (4)	0.0841 (4)	−0.0275 (9)	0.0545
C26	0.2872 (5)	0.0406 (4)	−0.0729 (12)	0.0664
C27	0.3341 (6)	0.0335 (4)	−0.1059 (10)	0.0672
C28	0.3662 (4)	0.0710 (4)	−0.0942 (9)	0.0533
C29	0.3507 (3)	0.1149 (4)	−0.0499 (8)	0.0360
C30	0.2180 (4)	0.0891 (4)	0.0044 (11)	0.0813

Table 4. *Selected geometric parameters* (Å, °) *for isomer* (II)

C9—C12	1.57 (1)	C10—C18	1.52 (1)
C9—C17	1.57 (1)	C10—C19	1.50 (2)
C9—C20	1.55 (1)	C11—C12	1.40 (1)
C9—C23	1.55 (1)	C17—C18	1.44 (1)
C10—C11	1.52 (2)	C19—C20	1.39 (1)
C12—C9—C17	104.9 (7)	C18—C10—C19	106.7 (8)
C12—C9—C20	108.3 (7)	C10—C11—C12	112.0 (8)
C12—C9—C23	109.9 (7)	C9—C12—C11	113.8 (8)
C17—C9—C20	101.8 (7)	C9—C17—C18	110.6 (8)
C17—C9—C23	115.0 (7)	C10—C18—C17	113.7 (8)
C20—C9—C23	116.1 (7)	C10—C19—C20	114.0 (8)
C11—C10—C18	105.8 (8)	C9—C20—C19	112.7 (8)
C11—C10—C19	107.6 (8)		

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, torsion angles and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71554 (47 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1064]

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5,5-Diphenyl-1*H*,2*H*,5*H*-imidazo[1,2-*a*]imidazole-3,6-dione

J. Karolak-Wojciechowska

*Institute of General and Ecological Chemistry,
Technical University, 90-924 Łódź, Poland*

K. Kieć-Kononowicz

*Institute of Pharmaceutical Chemistry,
Medical Academy, 31-065 Kraków, Poland*

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Abstract

The conformation of the title molecule, $C_{17}H_{13}N_3O_2$, is generally similar to the conformations of analogous compounds. Both five-membered rings possess envelope conformations. The dihedral angle between the mean planes of these two rings equals 5.33 (8)°. The molecules in the crystal are joined by an intermolecular hydrogen bond N1—H4···O1(x, $\frac{1}{2} + y$, z) of 2.869 (3) Å.

Comment

Hydantoins are classified as compounds that influence the central nervous system (CNS) (Vida, 1977). The crystal and molecular structures of pharmacologically active hydantoins have been the subject of many papers (Heeg, Subramanian & Dryhurst, 1986; Camerman, Mastropaolo & Camerman, 1983; Uno

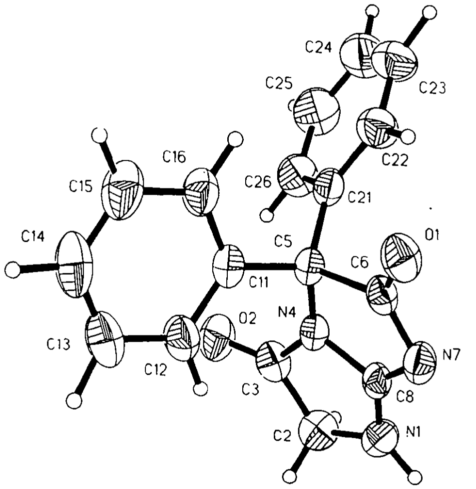

Fig. 1. Molecular structure and atomic numbering scheme for the title compound.

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