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1,4-Diformyl-*t*-5-methyl-*r*-2,*t*-3-diphenylpiperazine-1,4-dicarbaldehyde

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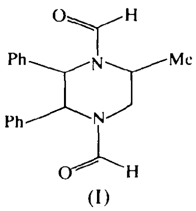
Abstract

A single-crystal X-ray diffraction study of the title compound ($C_{19}H_{20}N_2O_2$) confirms the chair conformation of the piperazine ring, with the methyl and phenyl groups in axial orientations.

Comment

The major highlight of this investigation is the presence of an alternate chair conformation of the piperazine ring of the title compound, (I), in which the phenyl and the methyl groups assume axial orientations. The normal chair conformation with equatorial substituents is not preferred due to the steric interaction between the *N*-formyl and phenyl groups. The C5—C6 and C3—C2 bond distances, 1.519 (3) and 1.537 (3) Å, respectively, are in the range 1.515–1.535 Å reported for other piperazine derivatives (Sekido *et al.*, 1985; Okamato *et al.*, 1979, 1982; Sakurai *et al.*, 1977; Davis & Hassel, 1963). The bond angles around the Csp^3 atoms in the piperazine ring [109.0 (2)–111.3 (2)°] indicate only slight distortion from the tetrahedral arrangement of groups around these atoms. The bond angles around N1 and N4 [116.6 (2)–122.2 (2)°] suggest sp^2 character for these atoms, as in the case of similar piperazine derivatives (Sekido *et al.*, 1985; Okamato *et al.*, 1979; Sakurai *et al.*, 1977). The N1 and N4 atoms are at a distance of 0.554 (2) and −0.594 (2) Å, respectively, from the C2—C3—C5—C6 plane, indicating a chair conformation in which the two N atoms are unsymmetrically disposed. The axial orientations of the methyl and the two phenyl groups are evident from the torsion angles, N4—C5—C6—C9 76.9 (2), N1—C2—C3—C16 −78.0 (2) and N4—C3—C2—C10 −78.2 (2)°.



(I)

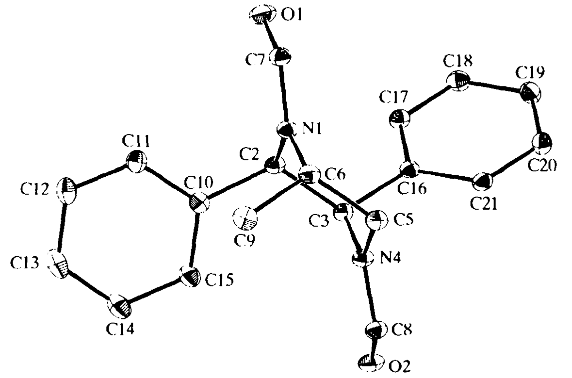


Fig. 1. *ZORTEP* (Zsolnai, 1997) plot of (I) showing 30% probability displacement ellipsoids. The H atoms are omitted for clarity.

Experimental

The title compound was obtained by the formylation of *t*-5-methyl-*r*-2,*t*-3-diphenylpiperazine by a mixture of acetic anhydride and 85% formic acid using benzene as solvent. The crude product isolated from the benzene layer was recrystallized using ethanol.

Crystal data

$C_{19}H_{20}N_2O_2$
$M_r = 308.37$
Monoclinic
$P2_1/n$
$a = 12.355$ (3) Å
$b = 11.645$ (3) Å
$c = 12.750$ (3) Å
$\beta = 117.407$ (16)°
$V = 1628.5$ (7) Å^3
$Z = 4$
$D_x = 1.258$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54180$ Å
Cell parameters from 25 reflections
$\theta = 4.12$–$70.13°$
$\mu = 0.657$ mm^{-1}
$T = 293$ (2) K
Cubic
0.1 × 0.1 × 0.1 mm
Colourless

Data collection

Rigaku AFC-7*S* diffractometer
ω–2θ scans
Absorption correction: ψ scans (North *et al.*, 1968)
$T_{min} = 0.829$, $T_{max} = 0.936$
3358 measured reflections
3021 independent reflections

2382 reflections with $I > 2\sigma(I)$
$R_{int} = 0.060$
$\theta_{max} = 70.13°$
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 14$
$l = -15 \rightarrow 13$
3 standard reflections every 150 reflections intensity decay: negligible

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.227$
$S = 1.090$
3021 reflections
212 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.1572P)^2 + 0.2382P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = <0.001$
$\Delta\rho_{\max} = 0.241$ e Å^{-3}
$\Delta\rho_{\min} = -0.266$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997*a*)
Extinction coefficient: 0.0058 (14)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

N4—C8	1.344 (3)	N1—C6	1.470 (3)
N4—C5	1.464 (3)	N1—C2	1.470 (3)
N4—C3	1.465 (2)	O1—C7	1.211 (3)
O2—C8	1.219 (3)	C2—C10	1.530 (3)
N1—C7	1.345 (3)		
C8—N4—C5	122.2 (2)	C7—N1—C6	120.5 (2)
C8—N4—C3	120.8 (2)	C7—N1—C2	120.5 (2)
C5—N4—C3	116.6 (2)	C6—N1—C2	119.0 (2)

The formyl H atoms were located from the difference map; their displacement parameters were kept fixed. The H atoms of the CH, CH_2 and CH_3 groups were fixed using geometrical considerations; their overall displacement parameters were refined.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Software Corporation, 1995*a*). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995*b*). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*b*). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*). Molecular graphics: *ZORTEP* (Zsolnai, 1997). Geometrical calculations: *PARST* (Nardelli, 1983). Software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: VJ1091). Services for accessing these data are described at the back of the journal.

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Troglitazone, an euglycemic antidiabetic drug†

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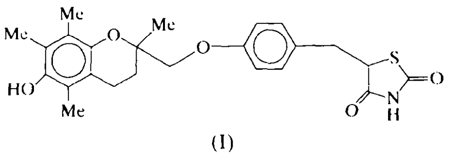
Abstract

Troglitazone (or 5-{4-[(6-hydroxy-2,5,7,8-tetramethylchroman-2-yl)methoxy]benzyl}thiazolidine-2,4-dione, $C_{24}H_{27}NO_5S$) is the first euglycemic drug. The molecules are held together in the lattice by intermolecular hydrogen bonds between the hydroxy O atom of the chroman moiety, and the ketone O and ring N atom of the thiazolidine-2,4-dione moiety.

Comment

The title compound, (I), has been prepared according to the procedure of Horikoshi *et al.* (1994) as part of our antidiabetic research. X-ray diffraction studies have been undertaken, as the structure has not previously been reported in the literature.



(I)

The molecular structure of (I) is shown in Fig. 1. All the bond parameters are normal (Allen *et al.*, 1987). The C23—O4 and C24—O5 bond lengths are 1.198 (4) and 1.206 (4) Å, respectively. This indicates that they are carbonyl groups, as found in 1,3-thiazolidine-2,4-dione (Forn *et al.*, 1975) and in 3-phenyl-1,3-thiazolidine-2,4-dione (Stankovic & Andretti, 1979).

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