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5-(1,3-Benzodioxol-5-yl)-3-methyl-1,7-diphenyl-1,6,7,8-tetrahydropyrazolo[3,4-*b*][1,4]diazepine

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.056
wR factor = 0.151
Data-to-parameter ratio = 15.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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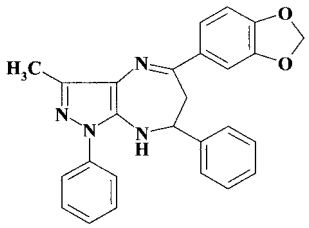
The title compound, $C_{26}H_{22}N_4O_2$, contains neither hydrogen bonds nor $\pi\cdots\pi$ short intermolecular contacts.

Comment

Benzodiazepines are an important class of psychotherapeutic compounds. In recent years, some examples of heterocyclic rings fused to the seven-membered diazepine ring system have been reported (Kelly *et al.*, 1997, and references therein). In particular, good CNS activity was reported for various pyrazolodiazepines (DeWald *et al.*, 1981). Some of these compounds are known to have psychotropic activity (Chimirri *et al.*, 1993, and references therein).



(I)

The diazepine ring in the title compound, (I), is disordered, with atom C6 taking up two possible sites. The major component has a site occupancy of 0.895 (7). There are no unusual bonds or angles, nor are there any intermolecular contacts less than the sum of the van der Waals radii.

Experimental

Glacial acetic acid (1 ml) was added to a solution of 4,5-diamino-3-methyl-1-phenylpyrazole (3.2 mmol) and 1-(1,3-benzodioxol-5-yl)-3-phenyl-2-propen-1-one (3.2 mmol) in 10 ml absolute ethanol and then refluxed for 6 h (reaction monitored by thin-layer chromatography). The resulting precipitate was filtered off, washed with ethanol, dried and recrystallized from ethanol to afford crystals suitable for X-ray diffraction. Yield: 90%; m.p. 450 K; analysis calculated for $C_{26}H_{22}N_4O_2$: C, 73.92; H, 5.25; N, 13.26%; found: C, 73.96; H, 5.19; N, 13.15.

Crystal data

$C_{26}H_{22}N_4O_2$
M_r = 422.48
Triclinic, $P\overline{1}$
a = 10.2608 (8) Å
b = 10.9910 (6) Å
c = 11.2485 (8) Å
α = 63.656 (4)°
β = 63.573 (3)°
γ = 77.080 (5)°
V = 1017.49 (12) Å^3
Z = 2
D_x = 1.379 Mg m^{-3}
Mo Kα radiation
Cell parameters from 4524 reflections
θ = 3.0–27.4°
μ = 0.09 mm^{-1}
T = 120.0 (2) K
Plate, orange
0.20 × 0.20 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)
T_{min} = 0.982, T_{max} = 0.996
13 871 measured reflections
4524 independent reflections
2909 reflections with $I > 2\sigma(I)$
R_{int} = 0.094
θ_{max} = 27.4°
$h = -13 \rightarrow 13$
$k = -13 \rightarrow 14$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.151
S = 1.04
4524 reflections
294 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0677P)^2 + 0.1359P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C8*A*	1.359 (2)	N4—C5	1.280 (2)
N1—N2	1.381 (2)	C5—C6	1.521 (3)
N2—C3	1.316 (2)	C6—C7	1.511 (3)
C3—C3*A*	1.410 (3)	C7—N8	1.458 (2)
C3*A*—N4	1.389 (2)	N8—C8*A*	1.370 (2)
C8*A*—N1—N2	111.69 (14)	N4—C5—C6	122.20 (18)
C3—N2—N1	104.67 (15)	C7—C6—C5	114.19 (19)
N2—C3—C3*A*	112.27 (16)	N8—C7—C6	111.23 (18)
C8*A*—C3A—N4	133.61 (17)	C8*A*—N8—C7	118.18 (15)
C8*A*—C3*A*—C3	104.84 (17)	N1—C8*A*—N8	122.35 (16)
N4—C3*A*—C3	121.55 (16)	N1—C8*A*—C3*A*	106.52 (16)
C5—N4—C3*A*	124.19 (16)	N8—C8*A*—C3*A*	131.12 (18)
C8*A*—N1—C11—C12	−35.1 (3)	C6—C5—C51—C55	−1.6 (3)
N2—N1—C11—C12	146.04 (18)	N4—C5—C51—C52	2.3 (3)
C8*A*—N1—C11—C16	147.4 (2)	N8—C7—C71—C76	−109.6 (2)
N2—N1—C11—C16	−31.5 (3)	C6—C7—C71—C76	126.7 (2)
N4—C5—C51—C55	−175.53 (19)	N8—C7—C71—C72	69.9 (2)
C6*A*—C5—C51—C55	50.1 (8)	C6—C7—C71—C72	−53.8 (3)

H atoms were treated as riding atoms, with C—H = 0.95–1.00 Å and N—H = 0.88 Å. Atom C6*A* of the minor component was restrained to lie 1.520 (2) Å from atoms C5 and C7. Atoms C6 and C6*A* were constrained to have the same anisotropic displacement parameters.

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *WordPerfect* macro *PRPKAPPA* (Ferguson, 1999).

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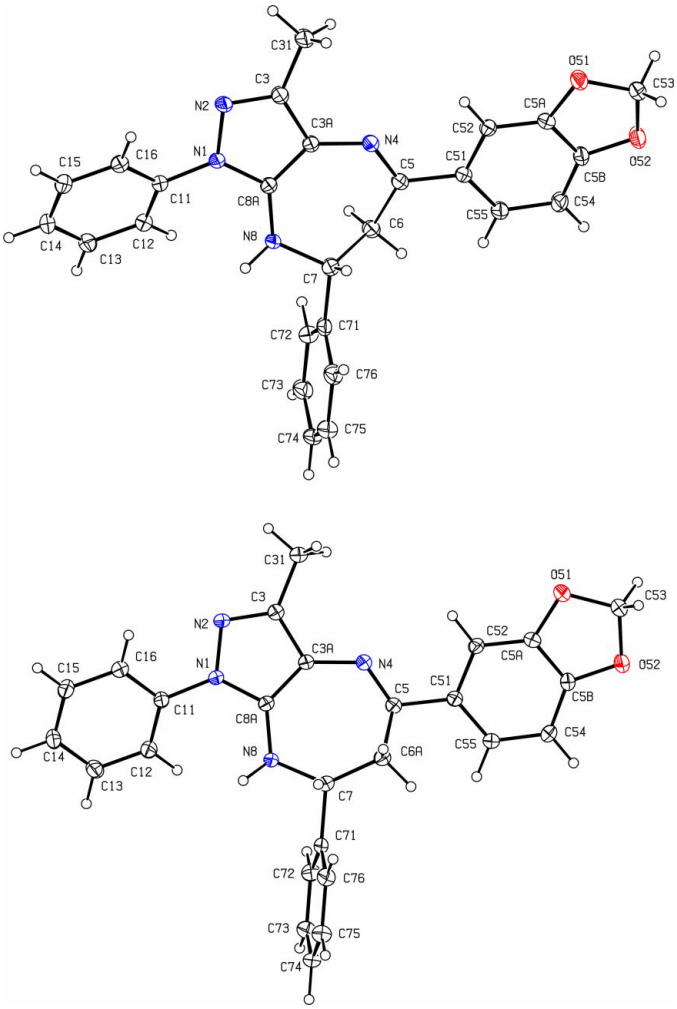

Figure 1
A view of the major (top) and minor (bottom) components of (I), with the numbering scheme. Displacement ellipsoids are drawn at the 30% probability level, and the view directions are slightly different.

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In the paper by Low *et al.* [*Acta Cryst.* (2003), E**59**, o614–o615], the title compound is better described as a racemate, *viz*. (7*RS*)-5-(1,3-benzodioxol-5-yl)-3-methyl-1,7-diphenyl-1,6,7,8-tetrahydropyrazolo[3,4-*b*][1,4]diazepine.