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5-(Benzo[1,3]dioxol-5-yl)-7-(4-chlorophenyl)-3-methyl-1-phenyl-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
R factor = 0.056
wR factor = 0.150
Data-to-parameter ratio = 16.3

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

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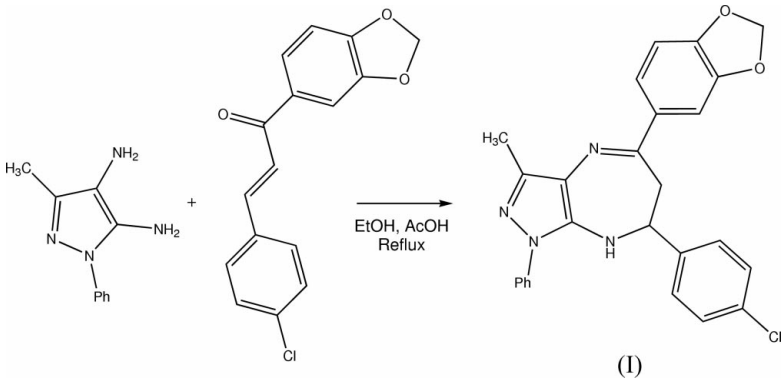
The title compound, $C_{26}H_{21}ClN_4O_2$, crystallizes in space group $P2_1/c$ with two molecules in the asymmetric unit. There are no significant differences between the bonds and angles of the two molecules; however, there are significant differences in the magnitudes of some of the equivlent torsion angles involving the chiral C^7 atoms (located at the points of attachment of the 4-chlorophenyl substituents).

Comment

Benzodiazepines are an important class of psychotherapeutic compounds. In recent years, some examples of fused heterocyclic rings with a seven-member diazepine ring system have appeared in the literature (Fray *et al.*, 1995; Kelly *et al.*, 1997). The pharmacological application, CNS activity for various pyrazolodiazepines (DeWald *et al.*, 1981), and general application as psychotropic compounds (Sharp, 1984; Chimirri *et al.*, 1993) have been reported.



(I)

The compound is chiral at C^7 (the site of the 4-chlorophenyl substituent), and the asymmetric unit has been chosen to have the *R* enantiomer at C17, while the *S* enantiomer is at C27 (Fig. 1, views (*a*) and (*b*), respectively). The unit cell contains both enantiomers of both molecules, of course.

Table 1 lists selected parameters for the fused heterocyclic ring system containing the diazepine ring.

The title compound contains no conventional hydrogen bonds, since the potential donors N18—H18 and N28—H28 lie in a pocket bounded by the chlorophenyl and phenyl groups within each molecule; this prevents the close approach of any potential acceptors. Table 2 lists C—H···O short contacts.

Experimental

A solution of 4,5-diamino-3-methyl-1-phenylpyrazole (3.2 mmol) and 1-(1,3-benzodioxol-5-yl)-3-(4-chlorophenyl)-2-propen-1-one in 1 ml

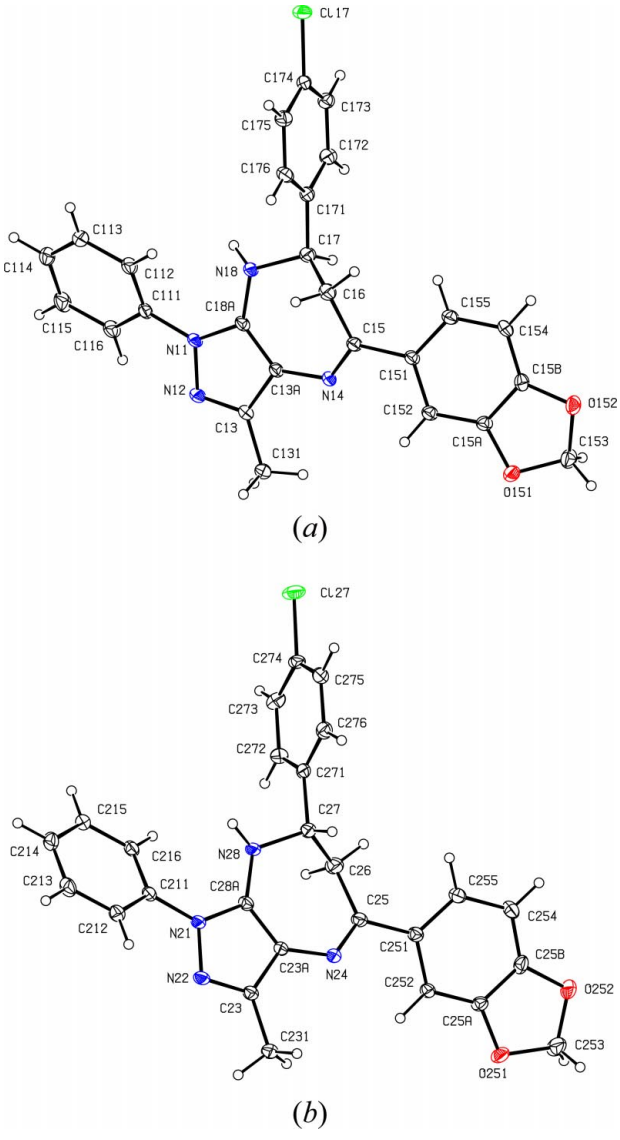


Figure 1
Views of the two molecules of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

of absolute ethanol with 1 ml of glacial acetic acid was heated to reflux for 7 h (reaction monitorized by TLC). The resulting precipitate was filtered off, washed with fresh ethanol, dried and recrystallized from ethanol. Yield 75%, m.p. 459 K. MS (EI 70 eV): 458/456 (*M*+, 37/100), 443 (20), 441 (54), 335 (10), 318 (14), 241 (14), 148 (17), 147 (13), 103 (10), 89 (17), 77 (43), 51 (13).

Crystal data

$C_{26}H_{21}ClN_4O_2$
M_r = 456.92
Monoclinic, $P2_1/c$
a = 11.3422 (2) Å
b = 15.6889 (3) Å
c = 24.1716 (5) Å
β = 92.437 (1)°
V = 4297.37 (14) Å^3
Z = 8
D_x = 1.412 Mg m^{-3}
Mo Kα radiation
Cell parameters from 9748 reflections
θ = 3.1–27.5°
μ = 0.21 mm^{-1}
T = 120 (2) K
Plate, yellow
0.25 × 0.20 × 0.02 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} = 0.949, T_{max} = 0.996
34725 measured reflections
9748 independent reflections
6221 reflections with $I > 2\sigma(I)$
R_{int} = 0.067
θ_{max} = 27.5°
$h = -12 \rightarrow 14$
$k = -20 \rightarrow 20$
$l = -30 \rightarrow 31$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.150
S = 1.02
9748 reflections
597 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0779P)^2 + 0.4063P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.71 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N11—C18*A*	1.371 (3)	N21—C28*A*	1.371 (3)
N11—N12	1.386 (2)	N21—N22	1.393 (2)
N12—C13	1.318 (3)	N22—C23	1.312 (3)
C13—C13*A*	1.423 (3)	C23—C23*A*	1.427 (3)
C13—C131	1.495 (3)	C23—C231	1.497 (3)
C13*A*—N14	1.394 (3)	C23*A*—N24	1.386 (3)
C13*A*—C18*A*	1.396 (3)	C23*A*—C28*A*	1.396 (3)
N14—C15	1.287 (3)	N24—C25	1.283 (3)
C15—C16	1.519 (3)	C25—C26	1.522 (3)
C16—C17	1.513 (3)	C26—C27	1.504 (3)
C17—N18	1.475 (3)	C27—N28	1.464 (3)
C18*A*—N18	1.384 (3)	C28*A*—N28	1.377 (3)
C18*A*—N11—N12	111.33 (17)	C28*A*—N21—N22	111.41 (17)
C13—N12—N11	105.10 (16)	C23—N22—N21	104.91 (16)
N12—C13—C13*A*	112.35 (18)	N22—C23—C23*A*	112.58 (18)
N14—C13*A*—C18*A*	133.66 (18)	N24—C23*A*—C28*A*	133.63 (18)
N14—C13*A*—C13	121.82 (18)	N24—C23*A*—C23	121.91 (18)
C18*A*—C13*A*—C13	104.42 (18)	C28*A*—C23*A*—C23	104.45 (18)
C15—N14—C13*A*	123.34 (18)	C25—N24—C23*A*	124.21 (18)
N14—C15—C16	122.81 (19)	N24—C25—C26	124.4 (2)
C17—C16—C15	112.7 (2)	C27—C26—C25	116.3 (2)
N18—C17—C16	110.07 (19)	N28—C27—C26	112.5 (2)
N11—C18*A*—N18	122.00 (19)	N21—C28*A*—N28	122.42 (19)
N11—C18*A*—C13*A*	106.76 (17)	N21—C28*A*—C23*A*	106.63 (17)
N18—C18*A*—C13*A*	131.22 (19)	N28—C28*A*—C23*A*	130.95 (19)
C18*A*—N18—C17	116.24 (17)	C28*A*—N28—C27	118.84 (17)
C18*A*—N11—N12—C13	1.6 (2)	C28*A*—N21—N22—C23	−0.5 (2)
N11—N12—C13—C13*A*	−0.2 (2)	N21—N22—C23—C23*A*	−0.5 (2)
N12—C13—C13*A*—N14	−177.83 (19)	N22—C23—C23*A*—N24	−179.68 (19)
N12—C13—C13*A*—C18*A*	−1.1 (2)	N22—C23—C23*A*—C28*A*	1.2 (2)
C18*A*—C13*A*—N14—C15	15.3 (4)	C28*A*—C23*A*—N24—C25	−12.3 (4)
C13—C13*A*—N14—C15	−169.1 (2)	C23—C23*A*—N24—C25	169.0 (2)
C13*A*—N14—C15—C16	4.7 (3)	C23*A*—N24—C25—C26	−4.7 (4)
N14—C15—C16—C17	−58.6 (3)	N24—C25—C26—C27	52.7 (3)
C15—C16—C17—N18	88.4 (2)	C25—C26—C27—N28	−79.3 (3)
N12—N11—C18*A*—N18	176.13 (19)	N22—N21—C28*A*—N28	−179.48 (19)
N12—N11—C18*A*—C13*A*	−2.3 (2)	N22—N21—C28*A*—C23*A*	1.3 (2)
N14—C13*A*—C18*A*—N11	178.1 (2)	N24—C23*A*—C28*A*—N21	179.6 (2)
C13—C13*A*—C18*A*—N11	2.0 (2)	C23—C23*A*—C28*A*—N21	−1.4 (2)
N14—C13*A*—C18*A*—N18	−0.1 (4)	N24—C23*A*—C28*A*—N28	0.4 (4)
C13—C13*A*—C18*A*—N18	−176.2 (2)	C23—C23*A*—C28*A*—N28	179.4 (2)
N11—C18*A*—N18—C17	−166.9 (2)	N21—C28*A*—N28—C27	168.4 (2)
C13*A*—C18*A*—N18—C17	11.1 (3)	C23*A*—C28*A*—N28—C27	−12.5 (4)
C16—C17—N18—C18*A*	−56.0 (3)	C26—C27—N28—C28*A*	52.7 (3)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C153—H15A···O252^{i}	0.99	2.48	3.327 (3)	144
C16—H16A···N12^{ii}	0.99	2.54	3.429 (3)	150
C172—H172···N24	0.95	2.56	3.426 (3)	152
C275—H275···O251^{iii}	0.95	2.45	3.367 (3)	163

Symmetry codes: (i) $x-1, \frac{1}{2}-y, z-\frac{1}{2}$; (ii) $2-x, 1-y, -z$; (iii) $x, \frac{1}{2}-y, z-\frac{1}{2}$.

H atoms were treated as riding atoms, with C—H = 0.95–1.00 Å and N—H = 0.95 Å.

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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