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# 1-(Benzotriazol-1-ylmethyl)-5-(4-dimethylaminophenyl)-3-phenyl-4,5-dihydro-1*H*-pyrazole

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**Key indicators**

Single-crystal X-ray study
*T* = 120 K
Mean σ(C–C) = 0.003 Å
*R* factor = 0.055
*wR* factor = 0.138
Data-to-parameter ratio = 17.5

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



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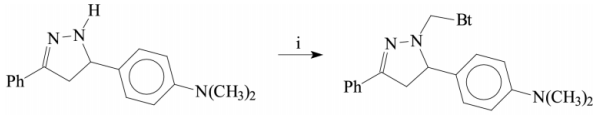
The title compound, $C_{24}H_{24}N_6$, shows no unusual features. There is no hydrogen bonding or $\pi$–$\pi$ stacking.

## Comment

The title compound, (I), was prepared to be used as an intermediate in the preparation of new heterocycles containing pyrazoline and benzazepine rings, following the benzotriazole methodology (see Scheme below) (Abonia *et al.*, 2001; Colotta *et al.*, 1996; Burckhalter *et al.*, 1952).




i = $BtCH_2OH$/EtOH/reflux
Bt = Benzotriazol-1-yl

(I)

There are no unusual bond distances or angles in (I), nor are there any intermolecular contacts less than 3.5 Å. The molecule has the *R* conformation at the chiral centre C13 (Fig. 1).

## Experimental

A mixture of 5-(4-dimethylaminophenyl)-3-phenyl-4,5-dihydro-1*H*-pyrazole (1.00 g, 3.77 mmol), 1-hydroxymethylbenzotriazole (0.57 g, 3.83 mmol) and ethanol (5 ml) was heated to reflux for 30 min. After cooling, the white solid which formed was filtered off and washed with ethanol (90% yield; m.p. 449 K). $^1$H NMR (300 MHz, DMSO-$d_6$, p.p.m.): 2.93 (1H, *dd*, *J* = 16.4, *J* = 14.4 Hz), 2.95 (3H, *s*), 3.30 (1H, *dd*, *J* = 16.4, *J* = 10.1 Hz), 4.14 (1H, *dd*, *J* = 14.5, *J* = 10.1 Hz), 5.58 (1H, *d*, *J* = 15.0 Hz), 6.33 (1H, *d*, *J* = 15.0 Hz), 6.87 (2H, *d*, 8.5 Hz), 7.31–7.54 (6H, *m*), 7.56–7.60 (3H, *m*), 8.03 (2H, *d*, *J* = 8.3 Hz); $^{13}$C NMR (75 MHz, DMSO-$d_6$, p.p.m.): 40.5, 41.4, 62.3, 65.1, 112.1, 112.9, 118.9, 124.0, 125.7, 127.2, 128.6, 128.8, 129.1, 132.1, 133.4, 145.2, 150.0, 151.1; MS (70 eV): *m*/*e* (%) 396 (12), 278 (74, *M* − Bt), 277 (100, *M* − BtH), 174 (58), 146 (41), 104 (32), 77 (57). Crystals suitable for single-crystal X-ray diffraction were grown from a solution in ethanol (96%).

### *Crystal data*

$C_{24}H_{24}N_6$
$M_r$ = 396.49
Monoclinic, $P2_1/c$
*a* = 10.8485 (2) Å
*b* = 14.4584 (4) Å
*c* = 14.0433 (4) Å
β = 106.840 (1)°
*V* = 2108.26 (9) Å$^3$
*Z* = 4
$D_x$ = 1.249 Mg m$^{-3}$
Mo Kα radiation
Cell parameters from 4800 reflections
θ = 3.0–27.5°
μ = 0.08 mm$^{-1}$
*T* = 120.0 (2) K
Needle, colourless
0.56 × 0.20 × 0.12 mm

*Data collection*

Nonius KappaCCD diffractometer
$\varphi$ scans and $\omega$ scans with $\kappa$ offsets
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)
$T_{min}$ = 0.958, $T_{max}$ = 0.991
24082 measured reflections
4800 independent reflections
3236 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.093
$\theta_{max}$ = 27.5°
$h = -13 \rightarrow 14$
$k = -18 \rightarrow 18$
$l = -15 \rightarrow 18$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.138
$S$ = 1.03
4800 reflections
274 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0573P)^2 + 0.4464P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.24 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$
Extinction correction: *SHELXL*97
Extinction coefficient: 0.033 (2)

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

| | | | |
|---|---|---|---|
| N1—C7*A* | 1.360 (2) | C6—C7 | 1.371 (3) |
| N1—N2 | 1.3639 (19) | C7—C7*A* | 1.396 (3) |
| N1—C1 | 1.457 (2) | C1—N12 | 1.443 (2) |
| N2—N3 | 1.306 (2) | N11—C15 | 1.292 (2) |
| N3—C3*A* | 1.379 (2) | N11—N12 | 1.4098 (18) |
| C3*A*—C7*A* | 1.394 (2) | N12—C13 | 1.483 (2) |
| C3*A*—C4 | 1.399 (3) | C13—C14 | 1.528 (2) |
| C4—C5 | 1.360 (3) | C14—C15 | 1.508 (2) |
| C5—C6 | 1.408 (3) | | |
| C7*A*—N1—N2 | 110.24 (14) | N1—C7*A*—C3*A* | 104.08 (15) |
| C7*A*—N1—C1 | 129.93 (14) | N1—C7*A*—C7 | 133.41 (16) |
| N2—N1—C1 | 119.81 (14) | C3*A*—C7*A*—C7 | 122.52 (17) |
| N3—N2—N1 | 108.79 (15) | N12—C1—N1 | 114.69 (13) |
| N2—N3—C3*A* | 108.01 (15) | C15—N11—N12 | 108.51 (13) |
| N3—C3*A*—C7*A* | 108.88 (16) | N11—N12—C1 | 113.35 (12) |
| N3—C3*A*—C4 | 130.58 (18) | N11—N12—C13 | 108.34 (12) |
| C7*A*—C3*A*—C4 | 120.54 (18) | C1—N12—C13 | 116.73 (13) |
| C5—C4—C3*A* | 117.3 (2) | N12—C13—C14 | 100.44 (12) |
| C4—C5—C6 | 121.6 (2) | C15—C14—C13 | 101.03 (13) |
| C7—C6—C5 | 122.4 (2) | N11—C15—C14 | 112.38 (14) |
| C6—C7—C7*A* | 115.65 (19) | | |
| C7*A*—N1—C1—N12 | 80.6 (2) | N1—C1—N12—N11 | −71.05 (17) |
| N2—N1—C1—N12 | −97.47 (17) | N1—C1—N12—C13 | 55.92 (19) |

H atoms were treated as riding atoms with C—H distances in the range 0.95–1.00 Å.

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN* (Otwinowski & Minor, 1997); 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).

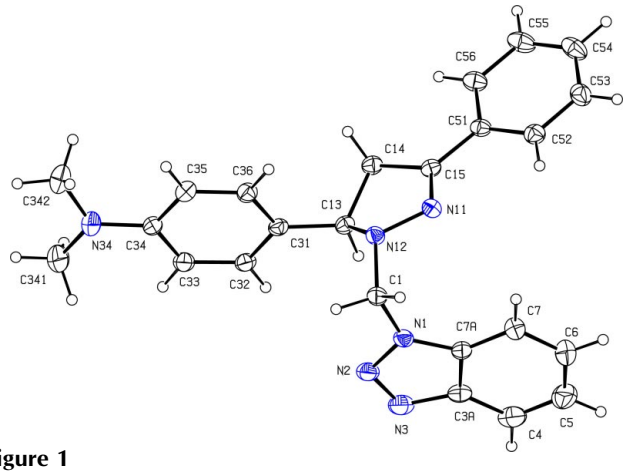



**Figure 1**
A view of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

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