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# 5-Amino-4-(benzotriazol-1-ylmethyl)-1-phenyl-3-*tert*-butylpyrazole

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**Key indicators**

Single-crystal X-ray study
$T$ = 100 K
Mean $\sigma$(C–C) = 0.003 Å
$R$ factor = 0.061
$wR$ factor = 0.150
Data-to-parameter ratio = 16.4

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



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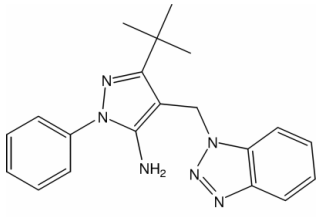
The molecules of the title compound, $C_{20}H_{22}N_6$, are linked together forming a $C(8)$ chain extending along the $c$ axis by an N—H···N hydrogen bond.

## Comment

The title compound, (I), was obtained by acid-catalysed rearrangement from 5–N-(benzotriazol-1-ylmethyl)amino-1-phenyl-3-*tert*-butylpyrazole.




(I)

The main supramolecular stuctural feature is a hydrogen bond, N51—H51···N43, 3.190 Å, which links the molecules by unit translations along the $c$ axis forming $C(8)$ chains (Bernstein *et al.*, 1995). H52 does not form a hydrogen bond. Details of the hydrogen bonding are given in Table 1. There are no weak C—H hydrogen bonds nor is there any $\pi$–$\pi$ base-stacking. Examination of the structure with *PLATON* (Spek, 2001) showed that there were no solvent-accessible voids in the crystal lattice.

## Experimental

To a solution of 0.3 g (0.867 mmol) of 5-*N*-(benzotriazol-1-ylmethyl)amino-1-phenyl-3-*tert*-butylpyrazole in 15 ml of absolute methanol were added 20–30 mg of *p*-toluensulfonic acid, and the mixture was stirred at room temperature for 1 h (TLC control). The solvent was removed under reduced pressure, the residue was redissolved in ethyl acetate, washed with a 10% NaOH solution and dried with anhydrous sodium sulfate. The solution was concentrated and purified by column chromatography on silica gel with a mixture 80:20 (hexane–ethyl acetate) as eluent, yielding 0.27 g (90%) of the title compound as white crystals (m.p. 397 K) suitable for X-ray diffraction.

*Crystal data*

$C_{20}H_{22}N_6$
$M_r$ = 346.44
Monoclinic, $P2_1/c$
$a$ = 13.465 (3) Å
$b$ = 15.135 (3) Å
$c$ = 9.1558 (18) Å
$\beta$ = 108.86 (3)°
$V$ = 1765.8 (6) Å$^3$
$Z$ = 4
$D_x$ = 1.303 Mg m$^{-3}$
Mo K$\alpha$ radiation
Cell parameters from 4002 reflections
$\theta$ = 3.1–27.5°
$\mu$ = 0.08 mm$^{-1}$
$T$ = 100 (1) K
Block, colourless
0.15 × 0.15 × 0.10 mm

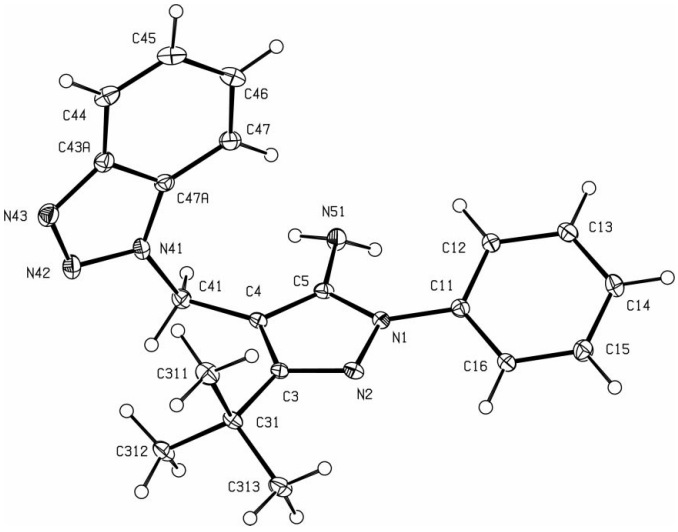



**Figure 1**
A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

*Data collection*

| | |
|---|---|
| KappaCCD diffractometer | 4002 independent reflections |
| $\varphi$ scans, and $\omega$ scans with $\kappa$ offsets | 2136 reflections with $I > 2\sigma(I)$ |
| Absorption correction: multi-scan (*DENZO-SMN*; Otwinowski & Minor, 1997) | $R_{int} = 0.077$ |
| | $\theta_{max} = 27.5^\circ$ |
| $T_{min} = 0.988$, $T_{max} = 0.992$ | $h = -17 \rightarrow 17$ |
| 10 902 measured reflections | $k = -19 \rightarrow 19$ |
| | $l = -11 \rightarrow 11$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)] = 0.061$ | $w = 1/[\sigma^2(F_o^2) + (0.0599P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.150$ | $(\Delta/\sigma)_{max} < 0.001$ |
| $S = 0.95$ | $\Delta\rho_{max} = 0.27$ e Å$^{-3}$ |
| 4002 reflections | $\Delta\rho_{min} = -0.39$ e Å$^{-3}$ |
| 244 parameters | |

**Table 1**
Hydrogen-bonding geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N51—H51···N43$^{i}$ | 0.87 | 2.37 | 3.190 (3) | 158 |

Symmetry code: (i) $x, y, 1 + z$.

H atoms attached to C atoms were included at calculated positions and treated as riding atoms with C—H 0.95–0.99 Å., The H atoms attached to the N atoms were included at positions based on those found in a difference map; they were initially restrained to a value of 0.87 Å and then made to ride on the parent N atom.

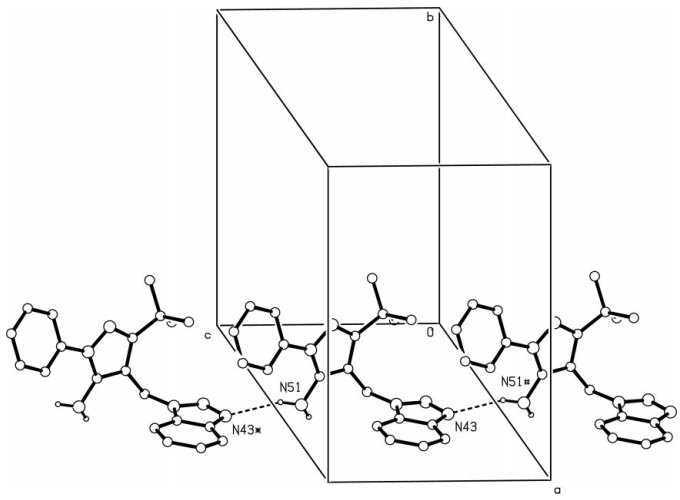



**Figure 2**
A view of the crystal structure showing the $C(8)$ chain running along the $c$ axis. The molecule labelled with an asterisk (*) is at $(x, y, 1+z)$ and that labelled with a hash (#) is at $(x, y, -1+z)$.

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, 2001); software used to prepare material for publication: *SHELXL*97 and *WordPerfect* macro *PRPKAPPA* (Ferguson, 1999).

X-ray data were collected at the EPSRC X-ray Crystallographic Service, University of Southampton, using a Nonius KappaCCD diffractometer. The authors thank the staff for all their help and advice. JNL thanks NCR Self Service Dundee for grants which have provided computing facilities for this work.

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