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1-[4-(4-Methylpyrimidin-2-yl)piperazin-1-ylmethyl]-1*H*-benzotriazole

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.048
wR factor = 0.135
Data-to-parameter ratio = 14.9

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

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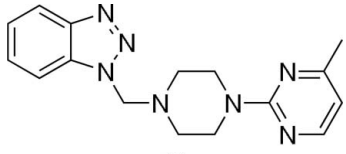
The title compound, $C_{16}H_{19}N_7$, is a potent new herbicide. X-ray analysis reveals that the piperazine ring adopts a chair conformation and weak C—H···N hydrogen bonds link the molecules into a chain along the *a* axis.

Comment

Based on the reported 1.65 Å high-resolution crystal structure of spinach KARI (ketol-acid reductoisomerase) complex (Biou *et al.*, 1997), we obtained 279 molecules with low binding energy toward KARI from MDL/ACD three-dimensional database searching, using the program DOCK 4.0 (Wang *et al.*, 2004). These potential structures provide information for further design of new targeted KARI herbicidal molecules. According to the structural information and bioactivity data of benzotriazole, one of the 279 molecules provided by MDL/ACD three-dimensional database searching, a series of benzotriazole derivatives has been designed and synthesized. The X-ray crystal structure determination of the title compound, (I), was undertaken to investigate the relationship between structure and herbicidal activity.



(I)

The molecular structure of (I) is shown in Fig. 1. The X-ray analysis reveals that the piperazine ring is in a chair conformation. The C12—N5 distance of 1.370 (2) Å is shorter than the normal C—N single-bond distance of 1.47 Å (Carey, 2000), which shows that C12—N5 is conjugated with the pyrimidine ring. The molecules translated one unit cell along the *a*-axis direction are linked by weak C—H···N hydrogen-bonding interactions to form a chain (Fig. 2 and Table 2).

Experimental

Compound (I) was prepared according to the reported procedure of Bachman & Heisey (1946), using benzotriazole (0.01 mol), 4-methyl-2-(piperazin-1-yl)pyrimidine (0.11 mol) (Xu *et al.*, 1993), 40% formalin (0.012 mol) and methanol (15 ml) (2.47 g, 80% yield). Colourless single crystals suitable for X-ray diffraction analysis were obtained by recrystallization from ethanol.

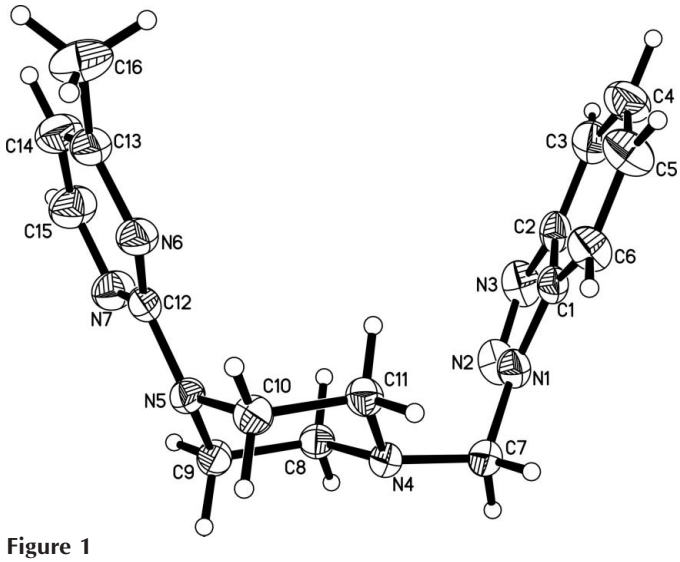


Figure 1
The molecular structure of (I), showing displacement ellipsoids drawn at the 30% probability level.

Crystal data

$C_{16}H_{19}N_7$	$Z = 2$
$M_r = 309.38$	$D_x = 1.334$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 6.5207$ (17) Å	Cell parameters from 1635 reflections
$b = 9.2488$ (16) Å	$\theta = 2.3$–$26.2°$
$c = 13.429$ (4) Å	$\mu = 0.09$ mm^{-1}
$\alpha = 102.108$ (8)°	$T = 294$ (2) K
$\beta = 94.112$ (7)°	Prism, colourless
$\gamma = 101.504$ (8)°	0.26 × 0.24 × 0.20 mm
$V = 770.5$ (3) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	3104 independent reflections
	2102 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.018$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 26.5°$
	$h = -8 \rightarrow 8$
$T_{min} = 0.972$, $T_{max} = 0.983$	$k = -11 \rightarrow 11$
4365 measured reflections	$l = -16 \rightarrow 7$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0614P)^2 + 0.1903P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.048$	
$wR(F^2) = 0.135$	
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.001$
3104 reflections	$\Delta\rho_{max} = 0.34$ e Å^{-3}
209 parameters	$\Delta\rho_{min} = -0.18$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

N1—N2	1.352 (2)	N5—C12	1.370 (2)
N1—C1	1.360 (2)	N5—C9	1.451 (2)
N1—C7	1.473 (2)	N5—C10	1.452 (2)
N2—N3	1.298 (3)	N6—C12	1.337 (2)
N3—C2	1.365 (3)	N6—C13	1.352 (3)
N4—C7	1.430 (2)	N7—C15	1.330 (3)
N4—C8	1.457 (2)	N7—C12	1.336 (3)
N4—C11	1.457 (2)		
N4—C7—N1	115.55 (15)	N6—C13—C16	115.8 (2)
N7—C12—N6	126.34 (19)	C14—C13—C16	123.0 (2)
N7—C12—N5	117.49 (17)		

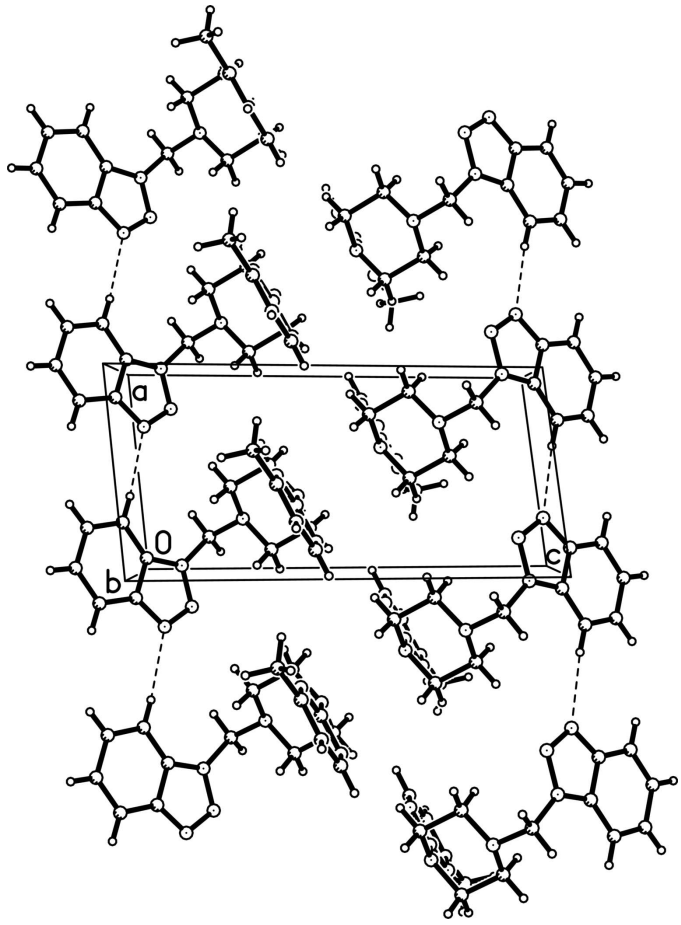


Figure 2
The crystal packing of (I), showing the C—H···N hydrogen-bonded chains (dashed lines).

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···N3^{i}	0.93	2.50	3.430 (3)	176
C9—H9A···N7	0.97	2.34	2.746 (3)	105
C10—H10B···N6	0.97	2.33	2.744 (3)	105

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

H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.93 (aromatic), 0.97 (methylene) or 0.97 Å (methyl), and with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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