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5-Chloromethyl-1,3-dimethyl-1*H*-pyrazole

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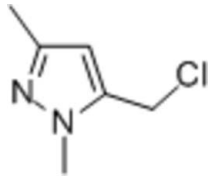
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.038; wR factor = 0.105; data-to-parameter ratio = 15.3.

The pyazole ring in the title compound, $C_6H_9ClN_2$, is almost planar (r.m.s. deviation = 0.003 Å). In the crystal, molecules are linked by C—H···N interactions, forming [100] chains.

Related literature

For a related structure, see: Baldy *et al.* (1985).



Experimental

Crystal data

$C_6H_9ClN_2$
M_r = 144.60
Triclinic, $P\overline{1}$
a = 6.5210 (7) Å
b = 7.3111 (7) Å
c = 7.9854 (8) Å
α = 88.383 (1)°
β = 77.563 (2)°
γ = 85.725 (2)°
V = 370.71 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.43 mm^{-1}
T = 296 K
0.28 × 0.22 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.890, T_{max} = 0.919
1906 measured reflections
1304 independent reflections
1135 reflections with $I > 2\sigma(I)$
R_{int} = 0.011

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.105
S = 1.05
1304 reflections
85 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1B···N2^{i}	0.97	2.50	3.446 (3)	164

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5664).

References

Baldy, A., Elguero, J., Fawe, R., Pierrot, M. & Vincent, E. J. (1985). *J. Am. Chem. Soc.* **107**, 5290-5291.
Bruker (2001). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2010). E**66**, o3006 [https://doi.org/10.1107/S1600536810042844]

5-Chloromethyl-1,3-dimethyl-1*H*-pyrazole

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S1. Experimental

N,*N*-Dimethylformamide(0.96 g, 11 mmol) was add to a 100 ml three necked-bottle, and phosphoryl trichloride(6.13 g, 40 mmol) was added slowly under ice-bath, then added (1,3-dimethyl-1*H*-pyrazole-5-yl)methanol(1.26 g, 10 mmol) at room temperature in portions. The reaction mixture was heated to reflux and reacted for 6 h. After separation through silica gel column chromatography (fluent: ethyl acetate/petroleum ether=1/5). The title product compound was obtained as a white solid (0.36 g, 22%) and recrystallised from methylene chloride to yield colourless blocks of (I).

Anal. Calcd for $C_6H_9N_2$: C, 49.84; H, 6.27; N, 19.37. Found: C, 49.81; H, 6.30; N, 19.40. ^{1}H NMR(CDCl3): 2.22(s,3*H*, CH_3), 3.84 (s,3*H*, N—CH_3), 4.53(s, 2H, CH_2), 6.04 (s, 1H, Pyrazole-H).

S2. Refinement

Although all H atoms were visible in difference maps, they werefinally placed in geometrically calculated positions, with C-Hdistances in the range 0.93–0.96Å and N—H distances of 0.86 Å, andincluded in the final refinement in the riding model approximation,with U_{iso}(H) = 1.2U_{eq}(C, N) and U_{iso}(H) = 1.5U_{eq}(C).

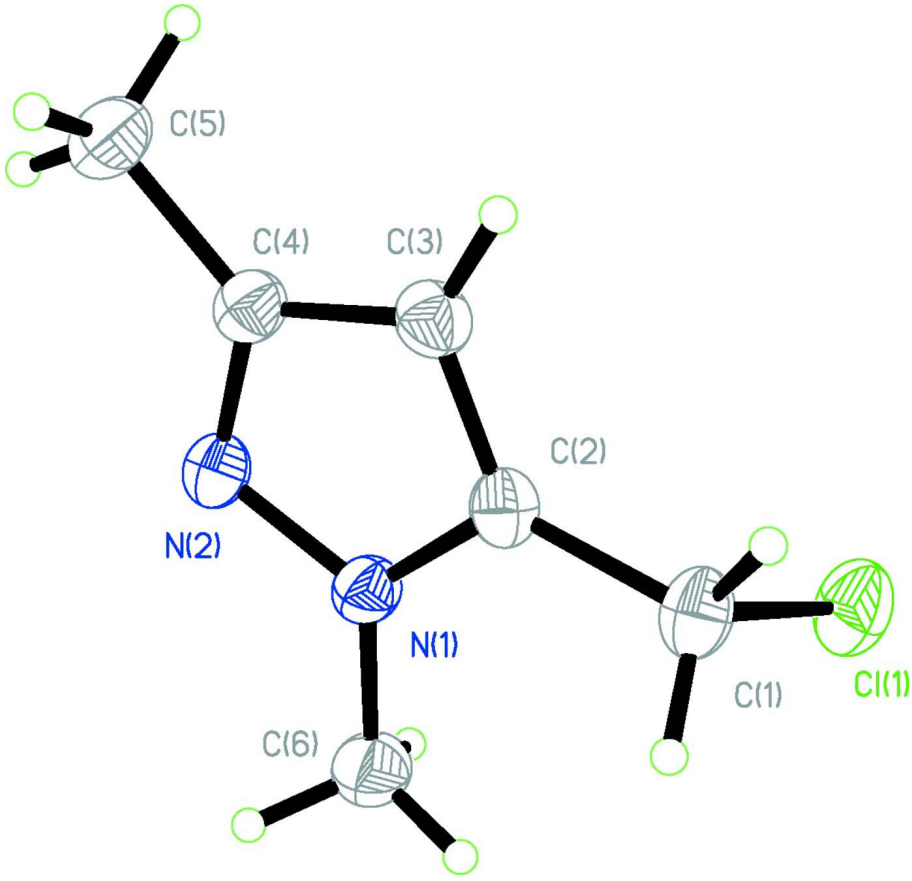

Figure 1

The molecular structure of (I), with 30% probability displacement ellipsoids.

5-Chloromethyl-1,3-dimethyl-1*H*-pyrazole

Crystal data

$C_6H_9ClN_2$
M_r = 144.60
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 6.5210 (7) Å
b = 7.3111 (7) Å
c = 7.9854 (8) Å
α = 88.383 (1)°
β = 77.563 (2)°
γ = 85.725 (2)°
V = 370.71 (6) Å^3
Z = 2
F(000) = 152
D_x = 1.295 Mg m^{-3}
Melting point = 361–364 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1109 reflections
θ = 2.6–26.7°
μ = 0.43 mm^{-1}
T = 296 K
BLOCK, colorless
0.28 × 0.22 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.890, T_{max} = 0.919
1906 measured reflections
1304 independent reflections
1135 reflections with $I > 2\sigma(I)$

R_{int} = 0.011
θ_{max} = 25.0°, θ_{min} = 2.6°
h = −7→4
k = −7→8
l = −9→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.105
S = 1.05
1304 reflections
85 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2 + 0.1537P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.43 (3)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Cl1	1.05348 (10)	0.89562 (8)	0.28229 (8)	0.0675 (3)
N1	0.7203 (2)	0.6178 (2)	0.1574 (2)	0.0414 (4)
N2	0.5716 (3)	0.4983 (2)	0.2188 (2)	0.0450 (4)
C1	1.0872 (3)	0.6828 (3)	0.1664 (3)	0.0509 (5)
H1A	1.1141	0.7093	0.0443	0.061*
H1B	1.2085	0.6102	0.1904	0.061*
C2	0.8994 (3)	0.5757 (3)	0.2144 (2)	0.0408 (5)
C3	0.8653 (3)	0.4224 (3)	0.3159 (3)	0.0453 (5)
H3	0.9593	0.3600	0.3737	0.054*
C4	0.6600 (3)	0.3784 (3)	0.3150 (2)	0.0435 (5)
C5	0.5411 (4)	0.2246 (3)	0.4043 (3)	0.0586 (6)
H5A	0.3958	0.2440	0.3976	0.088*
H5B	0.5518	0.2198	0.5224	0.088*
H5C	0.5991	0.1110	0.3504	0.088*
C6	0.6713 (4)	0.7699 (3)	0.0489 (3)	0.0579 (6)
H6A	0.5882	0.8648	0.1187	0.087*
H6B	0.5937	0.7284	−0.0304	0.087*
H6C	0.7997	0.8172	−0.0134	0.087*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0728 (5)	0.0529 (4)	0.0782 (5)	−0.0180 (3)	−0.0135 (3)	−0.0100 (3)
N1	0.0373 (9)	0.0436 (9)	0.0435 (9)	−0.0058 (7)	−0.0085 (7)	0.0046 (7)
N2	0.0381 (9)	0.0480 (10)	0.0493 (9)	−0.0092 (7)	−0.0085 (7)	0.0007 (7)
C1	0.0387 (11)	0.0513 (12)	0.0616 (13)	−0.0059 (9)	−0.0065 (9)	−0.0068 (10)
C2	0.0346 (10)	0.0425 (10)	0.0449 (10)	−0.0009 (8)	−0.0073 (8)	−0.0066 (8)
C3	0.0455 (11)	0.0434 (11)	0.0487 (11)	0.0015 (9)	−0.0157 (9)	−0.0001 (8)
C4	0.0467 (11)	0.0402 (10)	0.0417 (10)	−0.0053 (8)	−0.0044 (8)	−0.0028 (8)
C5	0.0652 (15)	0.0492 (12)	0.0589 (13)	−0.0133 (11)	−0.0054 (11)	0.0036 (10)
C6	0.0584 (14)	0.0556 (13)	0.0635 (14)	−0.0074 (11)	−0.0219 (11)	0.0140 (11)

Geometric parameters (Å, º)

Cl1—C1	1.808 (2)	C3—C4	1.401 (3)
N1—C2	1.354 (2)	C3—H3	0.9300
N1—N2	1.357 (2)	C4—C5	1.490 (3)
N1—C6	1.449 (3)	C5—H5A	0.9600
N2—C4	1.330 (3)	C5—H5B	0.9600
C1—C2	1.478 (3)	C5—H5C	0.9600
C1—H1A	0.9700	C6—H6A	0.9600
C1—H1B	0.9700	C6—H6B	0.9600
C2—C3	1.368 (3)	C6—H6C	0.9600
C2—N1—N2	111.77 (16)	N2—C4—C3	110.45 (17)
C2—N1—C6	128.92 (17)	N2—C4—C5	120.51 (19)
N2—N1—C6	119.29 (16)	C3—C4—C5	129.03 (19)
C4—N2—N1	105.36 (15)	C4—C5—H5A	109.5
C2—C1—Cl1	111.73 (14)	C4—C5—H5B	109.5
C2—C1—H1A	109.3	H5A—C5—H5B	109.5
Cl1—C1—H1A	109.3	C4—C5—H5C	109.5
C2—C1—H1B	109.3	H5A—C5—H5C	109.5
Cl1—C1—H1B	109.3	H5B—C5—H5C	109.5
H1A—C1—H1B	107.9	N1—C6—H6A	109.5
N1—C2—C3	106.40 (17)	N1—C6—H6B	109.5
N1—C2—C1	123.08 (18)	H6A—C6—H6B	109.5
C3—C2—C1	130.52 (19)	N1—C6—H6C	109.5
C2—C3—C4	106.02 (17)	H6A—C6—H6C	109.5
C2—C3—H3	127.0	H6B—C6—H6C	109.5
C4—C3—H3	127.0		
C2—N1—N2—C4	−0.5 (2)	Cl1—C1—C2—C3	−104.4 (2)
C6—N1—N2—C4	−178.81 (18)	N1—C2—C3—C4	−0.1 (2)
N2—N1—C2—C3	0.3 (2)	C1—C2—C3—C4	−179.3 (2)
C6—N1—C2—C3	178.48 (19)	N1—N2—C4—C3	0.4 (2)
N2—N1—C2—C1	179.62 (17)	N1—N2—C4—C5	−179.94 (17)
C6—N1—C2—C1	−2.2 (3)	C2—C3—C4—N2	−0.2 (2)

Cl1—C1—C2—N1	76.5 (2)	C2—C3—C4—C5	−179.8 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*B*···N2^{i}	0.97	2.50	3.446 (3)	164

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