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7-Chloro-5-methyl-2-phenylpyrazolo-[1,5-*a*]pyrimidine

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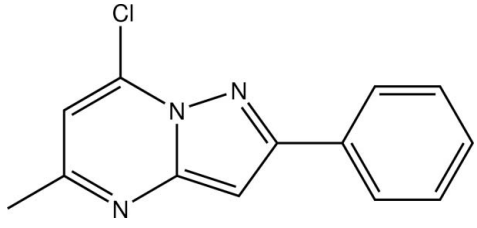
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.035; wR factor = 0.103; data-to-parameter ratio = 19.0.

The fused pyrazole and pyrimidine rings in the title compound, $C_{13}H_{10}ClN_3$, are almost coplanar, their planes being inclined to one another by 0.8 (2)°. The mean plane of the fused ring system is nearly coplanar with the phenyl ring, as indicated by the dihedral angle between their planes of 9.06 (7)°.

Related literature

For pharmacological and biochemical properties of pyrazolo[1,5-*a*]pyrimidine derivatives, see: Selleri *et al.* (2005); Almansa *et al.* (2001); Suzuki *et al.* (2001), Chen *et al.* (2004). For related structures, see: Senga *et al.* (1981).



Experimental

Crystal data

$C_{13}H_{10}ClN_3$
M_r = 243.69
Monoclinic, $P2_1/n$
a = 6.5993 (2) Å
b = 12.6166 (4) Å
c = 13.8702 (5) Å
β = 100.131 (2)°
V = 1136.84 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 296 K
0.41 × 0.32 × 0.21 mm

Data collection

Bruker X8 APEXII area-detector diffractometer
16957 measured reflections
2925 independent reflections
2521 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.103
S = 1.06
2925 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ5056).

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supporting information

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7-Chloro-5-methyl-2-phenylpyrazolo[1,5-*a*]pyrimidine

Ibtissam Bassoude, Sabine Berteina-Raboin, El Mokhtar Essassi, Gérald Guillaumet and Lahcen El Ammari

S1. Comment

Pyrazolo[1,5-*a*]pyrimidines have attracted considerable interest because of their biological activity. For instance, they are known for their potent utility as selective peripheral benzodiazepine receptor ligands (Selleri *et al.*, 2005), COX-2 selective inhibitors (Almansa *et al.*, 2001), HMG-CoA reductase inhibitors (Suzuki *et al.*, 2001) and CRF1 antagonists (Chen *et al.*, 2004). Our research group targeted at the synthesis of heterocycles with a bridgehead nitrogen atom such as the title compound (Senga *et al.* 1981).

The crystal structure of the title compound is built up from two fused five and six-membered rings (N1/N2/C4–C6 and N1/N3/C1–C4) linked to a methyl group and to a phenyl ring (C7–C12) as shown in Fig. 1. The pyrazole and pyrimidine rings are essentially planar with maximum deviations of 0.0010 (13) Å and 0.0052 (13) Å for C6 and C1, respectively, and form a dihedral angle of 0.8 (2)°. The mean plane through the fused ring system makes a dihedral angle of 9.06 (7)° with the phenyl ring.

S2. Experimental

Under argon, a mixture of 7-hydroxy-5-methyl-2-phenylpyrazolo[1,5-*a*]pyrimidine (0.8 g, 3.5 mmol), phosphorus oxychloride (0.8 ml, 8.89 mmol) and triethylamine (1 mL, 7 mmol) in 1,4-dioxane (2 ml) was heated to reflux for 3 h. The reaction mixture was allowed to cool to room temperature. After evaporation of the solvent under reduced pressure and addition of a $NaHCO_3$ saturated solution at 273 K (pH = 8), the residue was extracted with CH_2Cl_2. The combined organic layers were dried with $MgSO_4$, concentrated under vacuum and the residue was purified on silica gel by column chromatography using a 9:1 (*v*/*v*) mixture of petroleum ether and ethyl acetate as eluent. The compound was recrystallized from a mixture of cyclohexane/CH_2Cl_2 (1:1 *v*/*v*) to give colourless crystals.

S3. Refinement

All H atoms could be located in a difference Fourier map and treated as riding with C—H = 0.93 Å (aromatic), and C—H = 0.96 Å (methyl) and with U_{iso}(H) = 1.2 U_{eq}(aromatic) or U_{iso}(H) = 1.5 U_{eq} (methyl).

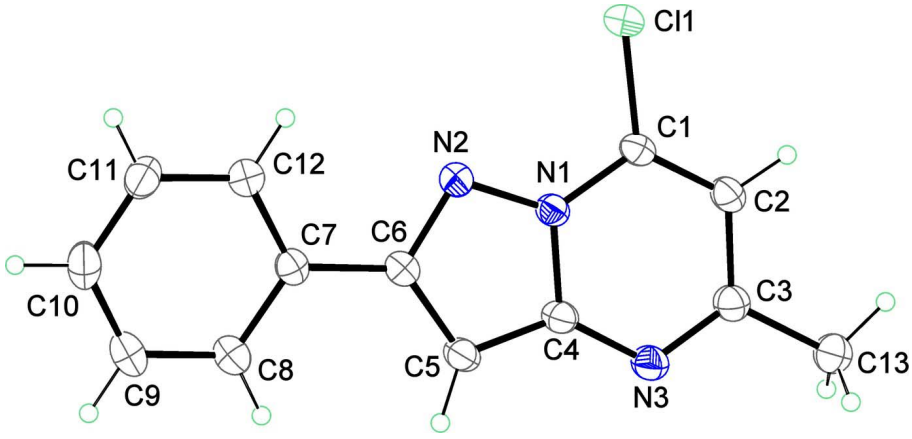

Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are represented as small circles of arbitrary radius.

7-Chloro-5-methyl-2-phenylpyrazolo[1,5-*a*]pyrimidine

Crystal data

$C_{13}H_{10}ClN_3$
$M_r = 243.69$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 6.5993$ (2) Å
$b = 12.6166$ (4) Å
$c = 13.8702$ (5) Å
$\beta = 100.131$ (2)°
$V = 1136.84$ (6) Å^3
$Z = 4$
$F(000) = 504$
$D_x = 1.418$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2925 reflections
$\theta = 3.0$–$28.7°$
$\mu = 0.31$ mm^{-1}
$T = 296$ K
Block, colourless
0.41 × 0.32 × 0.21 mm

Data collection

Bruker X8 APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
16957 measured reflections
2925 independent reflections
2521 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 28.7°$, $\theta_{min} = 3.0°$
$h = -6 \rightarrow 8$
$k = -17 \rightarrow 17$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.103$
$S = 1.06$
2925 reflections
154 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
$w = 1/[\sigma^2(F_o^2) + (0.0523P)^2 + 0.2738P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.07651 (19)	0.69856 (10)	0.25074 (9)	0.0344 (3)
C2	−0.0283 (2)	0.72024 (10)	0.32394 (10)	0.0393 (3)
H2	−0.1459	0.7622	0.3121	0.047*
C3	0.0426 (2)	0.67836 (11)	0.41889 (10)	0.0420 (3)
C4	0.3143 (2)	0.59912 (10)	0.36620 (9)	0.0369 (3)
C5	0.4894 (2)	0.54075 (11)	0.36126 (9)	0.0398 (3)
H5	0.5708	0.5041	0.4120	0.048*
C6	0.51851 (19)	0.54836 (10)	0.26417 (9)	0.0349 (3)
C7	0.68242 (19)	0.49912 (10)	0.21982 (9)	0.0353 (3)
C8	0.8475 (2)	0.44870 (11)	0.27861 (11)	0.0423 (3)
H8	0.8546	0.4468	0.3462	0.051*
C9	1.0010 (2)	0.40136 (12)	0.23699 (13)	0.0502 (4)
H9	1.1112	0.3684	0.2768	0.060*
C10	0.9911 (2)	0.40282 (13)	0.13685 (13)	0.0522 (4)
H10	1.0940	0.3706	0.1092	0.063*
C11	0.8287 (2)	0.45210 (13)	0.07785 (12)	0.0521 (4)
H11	0.8215	0.4528	0.0103	0.063*
C12	0.6755 (2)	0.50072 (12)	0.11910 (11)	0.0448 (3)
H12	0.5671	0.5347	0.0789	0.054*
C13	−0.0778 (3)	0.69939 (16)	0.49944 (12)	0.0599 (4)
H13A	−0.1946	0.7430	0.4748	0.090*
H13B	0.0084	0.7352	0.5525	0.090*
H13C	−0.1239	0.6334	0.5224	0.090*
N1	0.24981 (16)	0.63830 (8)	0.27118 (7)	0.0332 (2)
N2	0.37353 (16)	0.60790 (9)	0.20808 (8)	0.0357 (2)
N3	0.20986 (19)	0.61941 (10)	0.43971 (8)	0.0432 (3)
Cl1	0.00409 (5)	0.74223 (3)	0.13336 (2)	0.04662 (13)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0348 (6)	0.0302 (5)	0.0367 (6)	0.0020 (5)	0.0021 (5)	0.0041 (5)
C2	0.0389 (7)	0.0361 (6)	0.0427 (7)	0.0089 (5)	0.0061 (5)	0.0022 (5)
C3	0.0462 (7)	0.0413 (7)	0.0385 (6)	0.0091 (6)	0.0078 (5)	−0.0005 (5)
C4	0.0393 (6)	0.0370 (6)	0.0326 (6)	0.0047 (5)	0.0014 (5)	0.0011 (5)
C5	0.0401 (7)	0.0423 (7)	0.0351 (6)	0.0103 (5)	0.0012 (5)	0.0014 (5)

C6	0.0331 (6)	0.0323 (6)	0.0382 (6)	0.0011 (5)	0.0029 (5)	−0.0004 (5)
C7	0.0318 (6)	0.0319 (6)	0.0421 (6)	−0.0016 (5)	0.0061 (5)	−0.0006 (5)
C8	0.0379 (7)	0.0412 (7)	0.0459 (7)	0.0033 (5)	0.0021 (5)	−0.0029 (5)
C9	0.0359 (7)	0.0483 (8)	0.0647 (10)	0.0074 (6)	0.0044 (6)	−0.0021 (7)
C10	0.0412 (8)	0.0508 (8)	0.0691 (10)	0.0031 (6)	0.0219 (7)	−0.0026 (7)
C11	0.0527 (8)	0.0576 (9)	0.0508 (8)	0.0019 (7)	0.0224 (7)	0.0033 (7)
C12	0.0414 (7)	0.0502 (8)	0.0436 (7)	0.0053 (6)	0.0095 (6)	0.0043 (6)
C13	0.0659 (10)	0.0735 (11)	0.0433 (8)	0.0281 (9)	0.0176 (7)	0.0038 (7)
N1	0.0334 (5)	0.0318 (5)	0.0338 (5)	0.0036 (4)	0.0042 (4)	0.0023 (4)
N2	0.0344 (5)	0.0360 (5)	0.0368 (5)	0.0026 (4)	0.0068 (4)	0.0024 (4)
N3	0.0479 (6)	0.0474 (6)	0.0335 (5)	0.0130 (5)	0.0053 (5)	0.0004 (5)
Cl1	0.0463 (2)	0.0521 (2)	0.04017 (19)	0.00949 (14)	0.00399 (14)	0.01307 (13)

Geometric parameters (Å, º)

C1—C2	1.3533 (18)	C7—C8	1.3940 (18)
C1—N1	1.3610 (16)	C8—C9	1.386 (2)
C1—Cl1	1.7055 (13)	C8—H8	0.9300
C2—C3	1.4197 (19)	C9—C10	1.379 (2)
C2—H2	0.9300	C9—H9	0.9300
C3—N3	1.3201 (18)	C10—C11	1.377 (2)
C3—C13	1.504 (2)	C10—H10	0.9300
C4—N3	1.3519 (17)	C11—C12	1.389 (2)
C4—C5	1.3818 (18)	C11—H11	0.9300
C4—N1	1.4019 (16)	C12—H12	0.9300
C5—C6	1.3966 (18)	C13—H13A	0.9600
C5—H5	0.9300	C13—H13B	0.9600
C6—N2	1.3503 (16)	C13—H13C	0.9600
C6—C7	1.4727 (17)	N1—N2	1.3532 (15)
C7—C12	1.3897 (19)		
C2—C1—N1	118.58 (11)	C10—C9—C8	120.34 (14)
C2—C1—Cl1	123.81 (10)	C10—C9—H9	119.8
N1—C1—Cl1	117.61 (9)	C8—C9—H9	119.8
C1—C2—C3	119.48 (12)	C11—C10—C9	119.88 (14)
C1—C2—H2	120.3	C11—C10—H10	120.1
C3—C2—H2	120.3	C9—C10—H10	120.1
N3—C3—C2	122.62 (12)	C10—C11—C12	120.10 (15)
N3—C3—C13	117.87 (12)	C10—C11—H11	119.9
C2—C3—C13	119.50 (13)	C12—C11—H11	119.9
N3—C4—C5	132.85 (12)	C11—C12—C7	120.65 (14)
N3—C4—N1	122.10 (11)	C11—C12—H12	119.7
C5—C4—N1	105.05 (11)	C7—C12—H12	119.7
C4—C5—C6	105.62 (11)	C3—C13—H13A	109.5
C4—C5—H5	127.2	C3—C13—H13B	109.5
C6—C5—H5	127.2	H13A—C13—H13B	109.5
N2—C6—C5	112.96 (11)	C3—C13—H13C	109.5
N2—C6—C7	119.48 (11)	H13A—C13—H13C	109.5

C5—C6—C7	127.56 (11)	H13B—C13—H13C	109.5
C12—C7—C8	118.62 (12)	N2—N1—C1	127.17 (10)
C12—C7—C6	121.13 (12)	N2—N1—C4	112.96 (10)
C8—C7—C6	120.25 (12)	C1—N1—C4	119.86 (11)
C9—C8—C7	120.39 (14)	C6—N2—N1	103.41 (10)
C9—C8—H8	119.8	C3—N3—C4	117.35 (11)
C7—C8—H8	119.8		