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6-Chloro-*N*-methyl-5-nitro-*N*-phenylpyrimidin-4-amine

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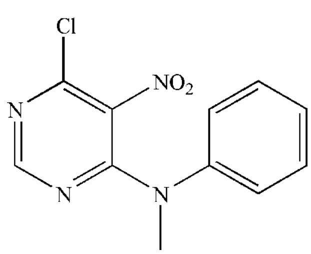
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.049; wR factor = 0.154; data-to-parameter ratio = 16.6.

In the title compound, $C_{11}H_9ClN_4O_2$, the dihedral angle between the aromatic rings is 79.67 (8)°. π–π stacking between centrosymmetrically related pairs of pyrimidine rings occurs along [100] [centroid–centroid separations = 3.4572 (8) and 3.5433 (7) Å].

Related literature

For a related structure, see: Shi *et al.* (2011).



Experimental

Crystal data

$C_{11}H_9ClN_4O_2$
M_r = 264.67
Triclinic, $P\overline{1}$
a = 6.8980 (14) Å
b = 8.9282 (18) Å
c = 11.427 (2) Å
α = 73.76 (3)°
β = 86.80 (3)°
γ = 84.21 (3)°
V = 672.0 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 293 K
0.44 × 0.38 × 0.13 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995) T_{min} = 0.885, T_{max} = 0.964
5925 measured reflections
2730 independent reflections
1742 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.154
S = 1.07
2730 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2000); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG5229).

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supplementary materials

Acta Cryst. (2011). E**67**, o2689 [**doi:10.1107/S1600536811037664**]

6-Chloro-*N*-methyl-5-nitro-*N*-phenylpyrimidin-4-amine

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Comment

Here, the crystal structure of 6-chloro-*N*-methyl-5-nitro-*N*-phenylpyrimidin-4-amine, the precursor of 6-chloro-*N*-methyl-*N*-phenylpyrimidine-4,5-diamine (Shi *et al.*, 2011) is determined by X-ray single crystal diffraction.

In the structure of (I) (Fig. 1), the dihedral angle between the aromatic rings is 79.667 (81)°. Uninterrupted aromatic π-π stacking between centrosymmetrically related pairs of pyrimidine rings occurs along with [100] direction [centroid – centroid separation = 3.4572 (8)Å or 3.5433 (7)Å].

Experimental

To a solution of 4,6-dichloro-5-nitro-pyrimidine (2.08 g, 10.8 mmol), and triethylamine (13.0 mL, 0.55 mmol) in anhydrous THF (25 mL) was added a solution of *N*-methylbenzylamine (0.85 mL, 10.8 mmol) in anhydrous THF (15 mL) slowly. The reaction mixture was stirred at room temperature overnight. The reaction mixture was concentrated in vacuo, diluted with water, and extracted with EtOAc. The organic phase was washed with 1N HCl, brine, dried over anhydrous $MgSO_4$, and concentrated in vacuo to yield the crude product as a solid. Purification by recrystallization from methanol provided the desired pure product, 6-chloro-*N*-methyl-5-nitro-*N*-phenylpyrimidin-4-amine (yellow solid, 1.85g, 64.7%, 130.3-131.4 °C). ^{1}H NMR ($CDCl_3$, 400 Hz), δ: 8.51 (s, 1H), 7.393-7.37(m, 3H), 7.17-7.15(m, 2H), 3.57 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 Hz), δ: 156.6, 153.9, 152.4, 142.2, 129.8, 128.6, 126.3, 41.7. ES-MS: 265.0 [(M + H^+)].

Refinement

All H atoms were located from difference Fourier maps. H atoms attached to C atoms were treated as riding [C—H = 0.93–0.96 Å, U_{iso}(H) = 1.2U_{eq}(aromatic carbon) and U_{iso}(H) = 1.5U_{eq}(methyl carbon)].

Figures

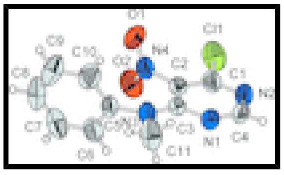

Fig. 1. The title compound, $C_{11}H_9ClN_4O_2$, with the atom-labelling scheme. Displacement ellipsoid are shown at the 50% probability level.

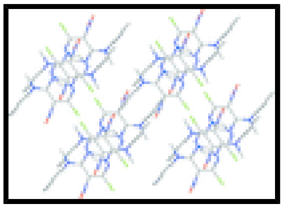

Fig. 2. Aromatic π-π stacking between centrosymmetrically related pairs of pyrimidine rings along 100].

6-Chloro-*N*-methyl-5-nitro-*N*-phenylpyrimidin-4-amine

Crystal data

$C_{11}H_9ClN_4O_2$	$Z = 2$
$M_r = 264.67$	$F(000) = 272$
Triclinic, $P\overline{1}$	$D_x = 1.308$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 6.8980 (14)$ Å	Cell parameters from 500 reflections
$b = 8.9282 (18)$ Å	$\theta = 3.4–27.5°$
$c = 11.427 (2)$ Å	$\mu = 0.28$ mm^{-1}
$\alpha = 73.76 (3)°$	$T = 293$ K
$\beta = 86.80 (3)°$	Block, colorless
$\gamma = 84.21 (3)°$	0.44 × 0.38 × 0.13 mm
$V = 672.0 (2)$ Å^3	

Data collection

Rigaku R-AXIS RAPID diffractometer	2730 independent reflections
Radiation source: fine-focus sealed tube	1742 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.030$
Detector resolution: 10.00 pixels mm^{-1}	$\theta_{max} = 27.5°$, $\theta_{min} = 3.4°$
ω scans	$h = -8 \rightarrow 7$
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	$k = -10 \rightarrow 10$
$T_{min} = 0.885$, $T_{max} = 0.964$	$l = -14 \rightarrow 14$
5925 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.049$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.154$	H-atom parameters constrained
$S = 1.07$	$w = 1/[\sigma^2(F_o^2) + (0.0827P)^2 + 0.0097P]$ where $P = (F_o^2 + 2F_c^2)/3$
2730 reflections	$(\Delta/\sigma)_{max} < 0.001$
164 parameters	$\Delta\rho_{max} = 0.25$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.19$ 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}
Cl1	0.27001 (12)	0.38397 (9)	0.55435 (6)	0.0897 (3)
C1	0.2612 (3)	0.1851 (3)	0.52481 (19)	0.0585 (5)
C2	0.2496 (3)	0.1772 (2)	0.40780 (17)	0.0483 (5)
C3	0.2386 (3)	0.0133 (2)	0.38625 (17)	0.0485 (5)
C4	0.2470 (3)	−0.1014 (3)	0.59315 (19)	0.0655 (6)
H4	0.2468	−0.1895	0.6599	0.079*
N1	0.2346 (3)	−0.1270 (2)	0.48527 (16)	0.0600 (5)
N2	0.2610 (3)	0.0465 (3)	0.62119 (16)	0.0694 (6)
C5	0.2510 (3)	0.1072 (2)	0.16239 (18)	0.0544 (5)
C6	0.4329 (4)	0.1522 (3)	0.1186 (2)	0.0716 (7)
H6	0.5428	0.1063	0.1631	0.086*
C7	0.4528 (5)	0.2692 (4)	0.0051 (3)	0.0954 (9)
H7	0.5759	0.3000	−0.0227	0.114*
C8	0.2935 (6)	0.3371 (4)	−0.0638 (2)	0.1037 (11)
H8	0.3077	0.4122	−0.1381	0.124*
C9	0.1135 (6)	0.2917 (4)	−0.0205 (3)	0.1034 (11)
H9	0.0043	0.3376	−0.0656	0.124*
C10	0.0897 (4)	0.1749 (3)	0.0930 (2)	0.0817 (8)
H10	−0.0337	0.1445	0.1202	0.098*
C11	0.2102 (5)	−0.1969 (3)	0.2719 (3)	0.0965 (10)
H11A	0.3338	−0.2570	0.2895	0.145*
H11B	0.1728	−0.1948	0.1917	0.145*
H11C	0.1136	−0.2440	0.3307	0.145*
N4	0.2395 (3)	0.3422 (2)	0.31108 (16)	0.0611 (5)
N3	0.2279 (3)	−0.0184 (2)	0.27815 (15)	0.0610 (5)
O1	0.0799 (3)	0.4051 (2)	0.27365 (17)	0.0885 (6)
O2	0.3903 (3)	0.4091 (2)	0.27659 (17)	0.0889 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1161 (6)	0.1002 (6)	0.0686 (4)	−0.0261 (4)	0.0009 (4)	−0.0440 (4)

C1	0.0513 (12)	0.0778 (15)	0.0475 (11)	−0.0073 (9)	−0.0013 (9)	−0.0187 (10)
C2	0.0444 (10)	0.0555 (12)	0.0418 (10)	−0.0054 (8)	0.0014 (8)	−0.0081 (9)
C3	0.0462 (11)	0.0536 (12)	0.0419 (10)	−0.0013 (8)	0.0020 (8)	−0.0082 (9)
C4	0.0575 (13)	0.0777 (16)	0.0451 (12)	0.0008 (10)	−0.0005 (9)	0.0073 (11)
N1	0.0608 (11)	0.0606 (11)	0.0494 (10)	−0.0012 (8)	0.0025 (8)	−0.0022 (8)
N2	0.0645 (12)	0.0960 (15)	0.0422 (10)	−0.0040 (10)	−0.0054 (8)	−0.0101 (10)
C5	0.0695 (14)	0.0575 (12)	0.0366 (10)	−0.0065 (9)	0.0008 (9)	−0.0137 (9)
C6	0.0695 (16)	0.0932 (18)	0.0524 (13)	−0.0116 (12)	0.0033 (11)	−0.0199 (12)
C7	0.103 (2)	0.123 (2)	0.0616 (16)	−0.0383 (18)	0.0260 (16)	−0.0237 (16)
C8	0.158 (3)	0.105 (2)	0.0433 (14)	−0.033 (2)	0.0002 (18)	−0.0056 (14)
C9	0.129 (3)	0.107 (2)	0.0651 (17)	−0.0076 (19)	−0.0391 (19)	−0.0022 (16)
C10	0.0740 (17)	0.102 (2)	0.0656 (15)	−0.0096 (13)	−0.0142 (13)	−0.0141 (14)
C11	0.170 (3)	0.0617 (16)	0.0635 (16)	−0.0234 (16)	0.0113 (17)	−0.0244 (12)
N4	0.0828 (14)	0.0553 (11)	0.0452 (10)	−0.0049 (9)	0.0051 (9)	−0.0154 (8)
N3	0.0842 (13)	0.0542 (11)	0.0436 (9)	−0.0103 (8)	0.0035 (8)	−0.0115 (8)
O1	0.0992 (14)	0.0837 (13)	0.0688 (11)	0.0162 (10)	−0.0197 (10)	−0.0035 (9)
O2	0.1063 (15)	0.0742 (12)	0.0829 (13)	−0.0353 (10)	0.0259 (11)	−0.0125 (9)

Geometric parameters (Å, °)

Cl1—C1	1.905 (2)	C6—H6	0.9300
C1—C2	1.366 (3)	C7—C8	1.375 (5)
C1—N2	1.408 (3)	C7—H7	0.9300
C2—C3	1.560 (3)	C8—C9	1.368 (4)
C2—N4	1.573 (3)	C8—H8	0.9300
C3—N3	1.349 (3)	C9—C10	1.431 (4)
C3—N1	1.436 (2)	C9—H9	0.9300
C4—N1	1.324 (3)	C10—H10	0.9300
C4—N2	1.456 (3)	C11—N3	1.633 (3)
C4—H4	0.9300	C11—H11A	0.9600
C5—C6	1.380 (3)	C11—H11B	0.9600
C5—C10	1.388 (3)	C11—H11C	0.9600
C5—N3	1.488 (3)	N4—O1	1.226 (2)
C6—C7	1.430 (4)	N4—O2	1.242 (3)
C2—C1—N2	119.2 (2)	C6—C7—H7	119.4
C2—C1—Cl1	119.29 (17)	C9—C8—C7	118.4 (2)
N2—C1—Cl1	121.46 (16)	C9—C8—H8	120.8
C1—C2—C3	118.29 (17)	C7—C8—H8	120.8
C1—C2—N4	113.29 (18)	C8—C9—C10	121.4 (3)
C3—C2—N4	128.35 (16)	C8—C9—H9	119.3
N3—C3—N1	110.90 (18)	C10—C9—H9	119.3
N3—C3—C2	127.02 (16)	C5—C10—C9	120.0 (3)
N1—C3—C2	122.06 (17)	C5—C10—H10	120.0
N1—C4—N2	128.60 (19)	C9—C10—H10	120.0
N1—C4—H4	115.7	N3—C11—H11A	109.5
N2—C4—H4	115.7	N3—C11—H11B	109.5
C4—N1—C3	112.86 (19)	H11A—C11—H11B	109.5
C1—N2—C4	118.93 (18)	N3—C11—H11C	109.5
C6—C5—C10	118.8 (2)	H11A—C11—H11C	109.5

C6—C5—N3	121.0 (2)	H11B—C11—H11C	109.5
C10—C5—N3	120.1 (2)	O1—N4—O2	120.9 (2)
C5—C6—C7	120.2 (2)	O1—N4—C2	118.81 (18)
C5—C6—H6	119.9	O2—N4—C2	120.25 (19)
C7—C6—H6	119.9	C3—N3—C5	120.02 (17)
C8—C7—C6	121.2 (3)	C3—N3—C11	120.80 (17)
C8—C7—H7	119.4	C5—N3—C11	118.93 (17)

Fig. 1

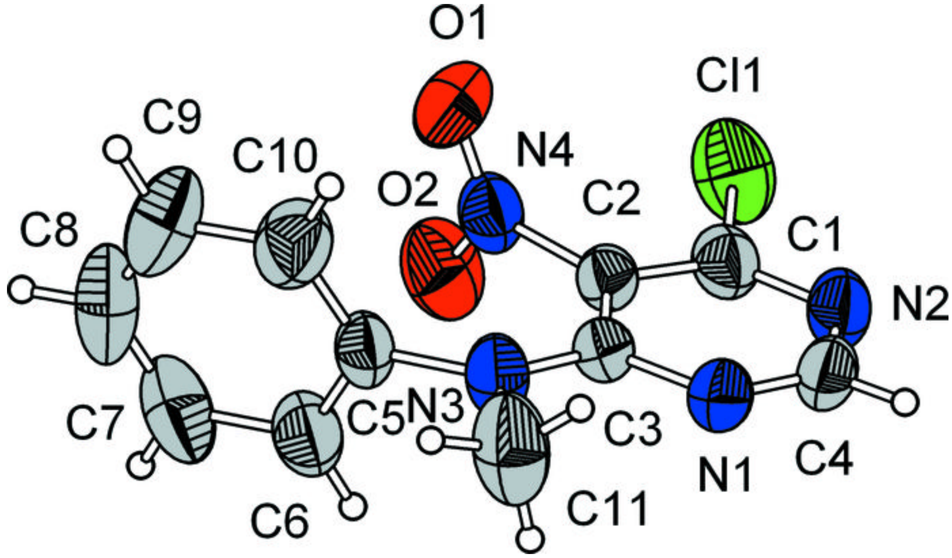

Fig. 2