organic compounds

2,6-Dichloro-7-isopropyl-7H-purine

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In the title molecule, $C_8H_8Cl_2N_4$, the essentially planar imidazole and pyrimidine rings [maximum deviations of 0.0030 (15) and 0.0111 (15) Å, respectively] make a dihedral angle of 1.32 (8)°. In the crystal, the fused-ring systems are stacked approximately parallel to the bc plane, with a centroid–centroid distance between inversion-related pyrimidine rings of 3.5189 (9) Å.

Related literature

For the synthesis, see: Oumata *et al.* (2008). For biological activity of some purine derivatives, see: Legraverend & Grierson (2006). For the selective synthesis of N7-substituted purines, see: Kotek *et al.* (2010). For related structures, see: Rouchal *et al.* (2009*a*,*b*, 2010).
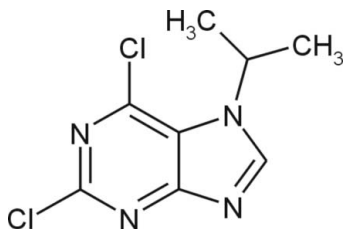
Experimental

Crystal data

$C_8H_8Cl_2N_4$
M_r = 231.08

Triclinic, $P\bar{1}$
a = 7.0146 (5) Å
b = 8.2862 (6) Å
c = 8.9686 (7) Å
α = 70.499 (7)°
β = 83.820 (6)°
γ = 74.204 (6)°

V = 472.75 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.65 mm^{-1}
T = 120 K
$0.40 \times 0.40 \times 0.20$ mm

Data collection

Oxford Diffraction Xcalibur Sapphire2 diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.933, T_{max} = 1.000

2825 measured reflections
1656 independent reflections
1419 reflections with $I > 2\sigma(I)$
R_{int} = 0.011

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.061
S = 1.05
1656 reflections

129 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = -0.24 e Å^{-3}

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97*.

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supplementary materials

2,6-Dichloro-7-isopropyl-7*H*-purine

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Comment

Purines represent a class of compounds with wide range of biological activities. The most of biologically active purines are di-, tri- or tetrasubstituted, usually on the C(2), C(6), C(8) and/or N(9) centers. Moreover, interesting biological properties were also described for some N7-substituted purines (Legraverend & Grierson, 2006). Owing to the relatively small portions of N7 isomer originating within the direct alkylation of purine bases, the selective synthesis of N7-substituted purines was recently described (Kotek *et al.*, 2010). The title molecule was isolated as a side product forming during the synthesis of novel 2,6,9-trisubstituted purine series.

The asymmetric unit of the title compound consists of a single purine molecule (Fig. 1). Both imidazole and pyrimidine rings are essentially planar with maximum deviations from the best plane being 0.0030 (15) Å for C4 (imidazole ring) and 0.0111 (15) Å for C3 (pyrimidine ring). The dihedral angle between the two rings is 1.32 (8)°. In the crystal packing (Fig .2), molecules are stacked parallel to the *bc*-plane. The distance between purine ring atoms (-*x*, 1 - *y*, -*z*) and best plane of adjacent purine ring (*x*, *y*, *z*) varies from -3.4111 (15) Å for N4 to -3.3728 (15) Å for C3. We have already published the structures of some related compounds (Rouchal *et al.*, 2009*a*,*b*; Rouchal *et al.*, 2010).

Experimental

The title compound was prepared following modified literature procedure (Oumata *et al.*, 2008). To a well stirred solution of 2,6-dichloro-9*H*-purine (4.5 g, 23.8 mmol) in DMSO (50 cm^3), potassium carbonate (9.9 g, 71.4 mmol) and 2-iodopropane (11.9 cm^3, 119.0 mmol) were added. The reaction mixture was stirred at 288–291K for 8 h. After that, the mixture was diluted with water (50 cm^3) and extracted with ether (7 × 15 cm^3). Collected organic layers were washed with brine (2 × 10 cm^3), dried over sodium sulfate and evaporated *in vacuo*. Both N7 and N9 isomers were separated from the crude material using column chromatography (silicagel; petroleum ether/ethyl acetate, 1/1, *v*/*v*). 2,6-Dichloro-7-isopropyl-7*H*-purine was obtained as a pale yellow crystalline powder (mp 425–427 K) in minor fraction. The crystal used for data collection was grown by spontaneous evaporation from deuterochloroform at room temperature.

Refinement

All carbon bound H atoms were placed at calculated positions and were refined as riding with their U_{iso} set to either 1.2U_{eq} or 1.5U_{eq} (methyl) of the respective carrier atoms; in addition, the methyl H atoms were allowed to rotate about the C—C bond.

Computing details

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis CCD* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED* (Oxford Diffraction, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and Mercury (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).
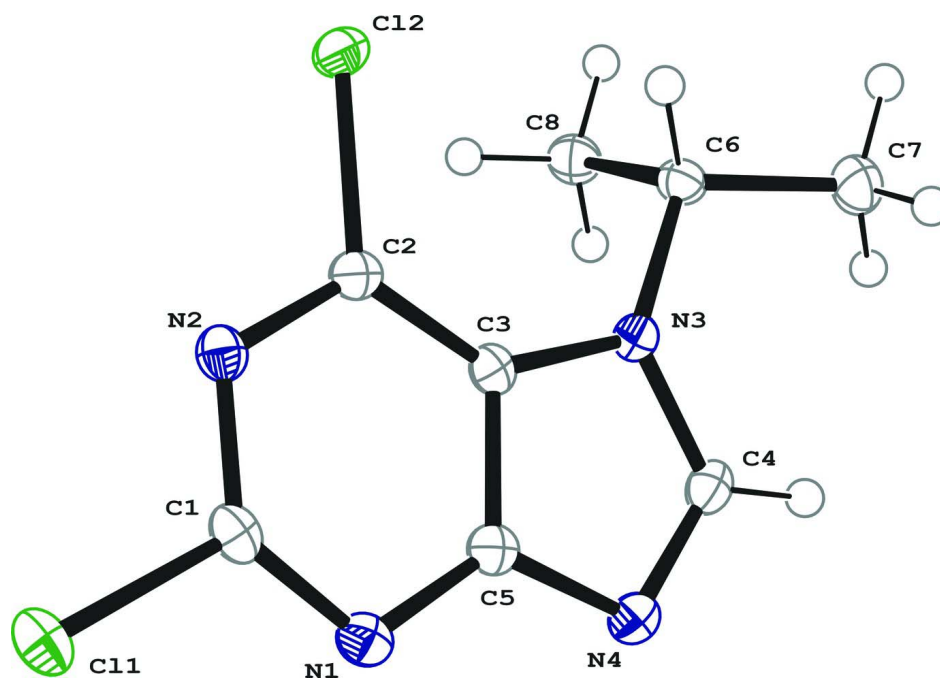
Figure 1

The molecular structure with 50% probability ellipsoids. H-atoms are shown as small spheres at arbitrary radii.
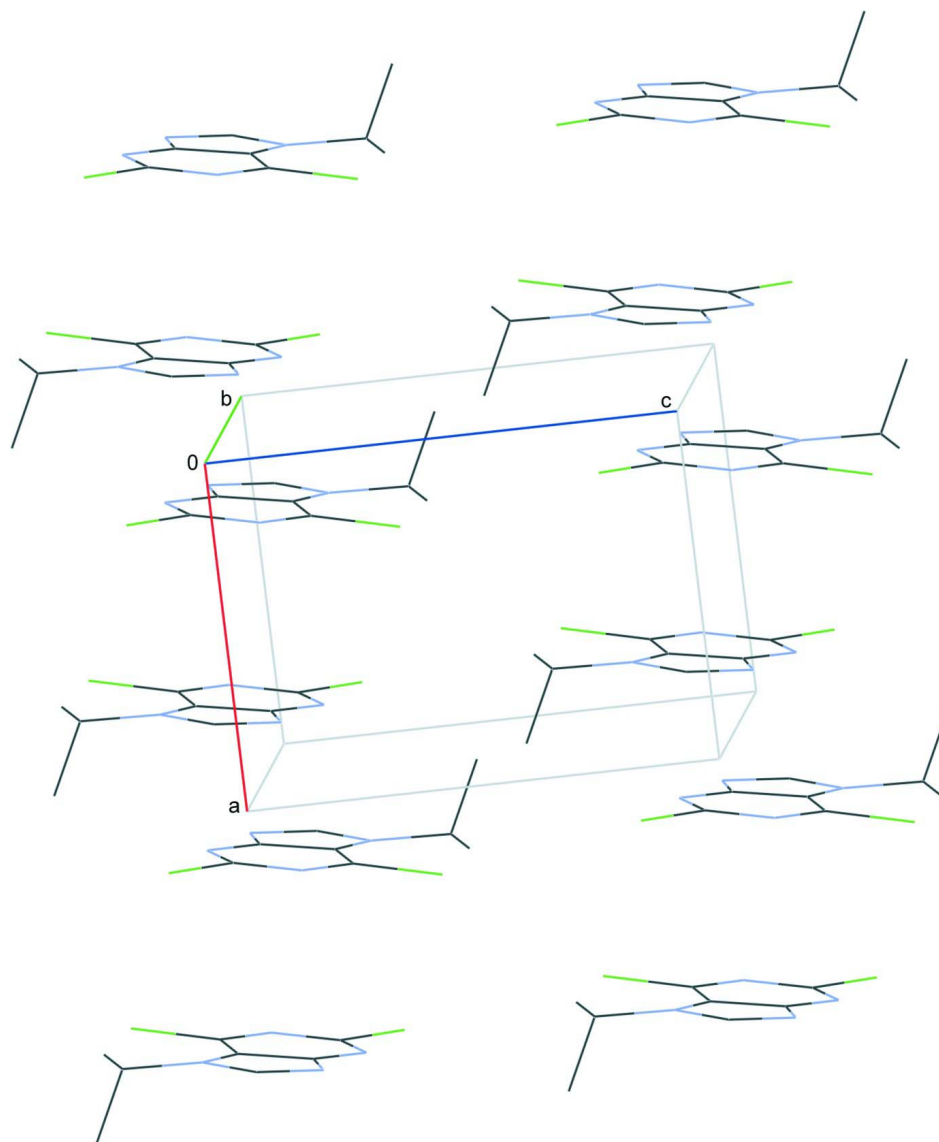
Figure 2

Molecules of title compound stacked along the *a*-axis. H-atoms have been omitted for clarity.

2,6-Dichloro-7-isopropyl-7*H*-purine

Crystal data

$C_8H_8Cl_2N_4$	$Z = 2$
$M_r = 231.08$	$F(000) = 236$
Triclinic, $P\overline{1}$	$D_x = 1.623$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 426 K
$a = 7.0146$ (5) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 8.2862$ (6) Å	Cell parameters from 2034 reflections
$c = 8.9686$ (7) Å	$\theta = 3.0–27.7°$
$\alpha = 70.499$ (7)°	$\mu = 0.65$ mm^{-1}
$\beta = 83.820$ (6)°	$T = 120$ K
$\gamma = 74.204$ (6)°	Block, colourless
$V = 472.75$ (7) Å^3	$0.40 \times 0.40 \times 0.20$ mm

Data collection

Oxford Diffraction Xcalibur Sapphire2 diffractometer	2825 measured reflections
Radiation source: Enhance (Mo) X-ray Source	1656 independent reflections
Graphite monochromator	1419 reflections with $I > 2\sigma(I)$
Detector resolution: 8.4353 pixels mm^{-1}	$R_{int} = 0.011$
ω scan	$\theta_{max} = 25.0°$, $\theta_{min} = 3.5°$
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)	$h = -8 \rightarrow 8$
	$k = -9 \rightarrow 9$
$T_{min} = 0.933$, $T_{max} = 1.000$	$l = -10 \rightarrow 10$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.023$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.061$	
$S = 1.05$	H-atom parameters constrained
1656 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0323P)^2 + 0.0416P]$ where $P = (F_o^2 + 2F_c^2)/3$
129 parameters	
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.26$ e Å^{-3}
	$\Delta\rho_{min} = -0.24$ 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 $F^2 > 2\sigma(F^2)$ 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	0.22677 (6)	0.42378 (6)	−0.21906 (5)	0.02126 (13)
Cl2	0.31193 (6)	0.39565 (5)	0.35468 (5)	0.02020 (13)
N1	0.2261 (2)	0.71202 (18)	−0.16073 (15)	0.0172 (3)
N2	0.26395 (19)	0.44137 (17)	0.05831 (15)	0.0160 (3)
N3	0.27132 (19)	0.83054 (17)	0.17303 (15)	0.0150 (3)
N4	0.2361 (2)	0.96725 (18)	−0.09060 (15)	0.0175 (3)
C1	0.2390 (2)	0.5421 (2)	−0.09346 (19)	0.0160 (4)
C2	0.2758 (2)	0.5246 (2)	0.15927 (18)	0.0153 (4)
C3	0.2616 (2)	0.7041 (2)	0.10815 (18)	0.0139 (3)
C4	0.2541 (2)	0.9822 (2)	0.04869 (19)	0.0177 (4)
H4	0.2549	1.0914	0.0613	0.021*
C5	0.2397 (2)	0.7924 (2)	−0.05616 (18)	0.0153 (4)
C6	0.2741 (2)	0.8107 (2)	0.34377 (18)	0.0165 (4)
H6	0.3683	0.6952	0.3972	0.020*
C7	0.3474 (3)	0.9569 (2)	0.3651 (2)	0.0245 (4)
H7A	0.4765	0.9589	0.3115	0.037*

H7B	0.3608	0.9358	0.4782	0.037*
H7C	0.2525	1.0708	0.3195	0.037*
C8	0.0694 (2)	0.8061 (2)	0.41748 (19)	0.0203 (4)
H8A	−0.0253	0.9186	0.3670	0.030*
H8B	0.0733	0.7879	0.5310	0.030*
H8C	0.0282	0.7090	0.4020	0.030*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0217 (2)	0.0247 (2)	0.0217 (2)	−0.00501 (18)	−0.00236 (17)	−0.01289 (18)
Cl2	0.0284 (3)	0.0158 (2)	0.0155 (2)	−0.00685 (18)	−0.00177 (17)	−0.00235 (17)
N1	0.0152 (7)	0.0196 (8)	0.0164 (7)	−0.0033 (6)	−0.0005 (6)	−0.0062 (6)
N2	0.0136 (7)	0.0170 (8)	0.0182 (7)	−0.0040 (6)	−0.0006 (6)	−0.0064 (6)
N3	0.0158 (7)	0.0140 (7)	0.0155 (7)	−0.0047 (6)	−0.0004 (6)	−0.0041 (6)
N4	0.0188 (8)	0.0152 (8)	0.0177 (7)	−0.0045 (6)	−0.0003 (6)	−0.0038 (6)
C1	0.0109 (8)	0.0211 (9)	0.0188 (8)	−0.0034 (7)	0.0003 (7)	−0.0105 (7)
C2	0.0109 (8)	0.0182 (9)	0.0152 (8)	−0.0035 (7)	0.0004 (6)	−0.0036 (7)
C3	0.0094 (8)	0.0166 (9)	0.0162 (8)	−0.0030 (7)	0.0005 (6)	−0.0062 (7)
C4	0.0161 (9)	0.0137 (8)	0.0220 (9)	−0.0045 (7)	−0.0003 (7)	−0.0038 (7)
C5	0.0103 (8)	0.0184 (9)	0.0162 (8)	−0.0030 (7)	0.0005 (6)	−0.0050 (7)
C6	0.0185 (9)	0.0166 (9)	0.0145 (8)	−0.0036 (7)	−0.0017 (7)	−0.0053 (7)
C7	0.0301 (11)	0.0255 (10)	0.0230 (9)	−0.0119 (8)	−0.0007 (8)	−0.0104 (8)
C8	0.0227 (10)	0.0233 (10)	0.0159 (8)	−0.0066 (8)	0.0016 (7)	−0.0077 (7)

Geometric parameters (Å, º)

Cl1—C1	1.7437 (16)	C3—C5	1.414 (2)
Cl2—C2	1.7249 (16)	C4—H4	0.9500
N1—C1	1.315 (2)	C6—C7	1.511 (2)
N1—C5	1.343 (2)	C6—C8	1.519 (2)
N2—C2	1.3289 (19)	C6—H6	1.0000
N2—C1	1.339 (2)	C7—H7A	0.9800
N3—C4	1.360 (2)	C7—H7B	0.9800
N3—C3	1.3770 (19)	C7—H7C	0.9800
N3—C6	1.486 (2)	C8—H8A	0.9800
N4—C4	1.318 (2)	C8—H8B	0.9800
N4—C5	1.371 (2)	C8—H8C	0.9800
C2—C3	1.381 (2)		
C1—N1—C5	112.46 (14)	N4—C5—C3	110.38 (14)
C2—N2—C1	115.85 (14)	N3—C6—C7	110.35 (13)
C4—N3—C3	105.08 (13)	N3—C6—C8	109.77 (12)
C4—N3—C6	127.26 (14)	C7—C6—C8	112.12 (14)
C3—N3—C6	127.25 (13)	N3—C6—H6	108.2
C4—N4—C5	103.55 (13)	C7—C6—H6	108.2
N1—C1—N2	130.38 (15)	C8—C6—H6	108.2
N1—C1—Cl1	116.32 (12)	C6—C7—H7A	109.5
N2—C1—Cl1	113.29 (12)	C6—C7—H7B	109.5
N2—C2—C3	121.08 (14)	H7A—C7—H7B	109.5

N2—C2—Cl2	116.30 (12)	C6—C7—H7C	109.5
C3—C2—Cl2	122.62 (13)	H7A—C7—H7C	109.5
N3—C3—C2	137.57 (15)	H7B—C7—H7C	109.5
N3—C3—C5	105.65 (13)	C6—C8—H8A	109.5
C2—C3—C5	116.70 (15)	C6—C8—H8B	109.5
N4—C4—N3	115.34 (15)	H8A—C8—H8B	109.5
N4—C4—H4	122.3	C6—C8—H8C	109.5
N3—C4—H4	122.3	H8A—C8—H8C	109.5
N1—C5—N4	126.13 (14)	H8B—C8—H8C	109.5
N1—C5—C3	123.49 (15)		