Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(6-Amino-7*H*-purin-7-yl)-1-phenylethanone

Stefanie Buehler,[a] Dieter Schollmeyer,[b] Dominik Hauser,[a] Stefan Laufer[a] and Christian Peifer[a]*

[a]Institute of Pharmacy, Department of Pharmaceutical and Medicinal Chemistry, Eberhard-Karls-University Tübingen, Auf der Morgenstelle 8, 72076 Tübingen, Germany, and [b]Department of Organic Chemistry, Johannes Gutenberg-University Mainz, Duesbergweg 10-14, D-55099 Mainz, Germany
Correspondence e-mail: christian.peifer@uni-tuebingen.de

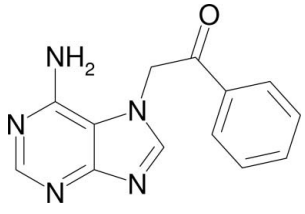
Received 6 November 2007; accepted 7 November 2007

Key indicators: single-crystal X-ray study; T = 193 K; mean σ(C–C) = 0.003 Å; R factor = 0.047; wR factor = 0.136; data-to-parameter ratio = 13.2.

In the title compound, $C_{13}H_{11}N_5O$, the exocyclic amino group of one purine molecule forms two intermolecular N—H···N hydrogen bonds to different ring N atoms of another molecule. The purine system is orientated almost perpendicular [77.96 (6)°] to the phenylethanone substituent. The preparation of the title compound occurred *via* a regioselective synthesis using the methyl(aqua)cobaloxime complex $CH_3Co(DH)_2OH_2$ as a temporary auxiliary, and its X-ray crystal structure confirmed the regioselective *N*-alkylation of this molecule.

Related literature

For background, see: Hopkins & Groon (2002) ; Laufer *et al.* (2005); Meijer & Raymond (2003); Dalby *et al.* (1993). For preparation, see: Marzilli *et al.* (1975); Bader & Chiang (1983); Schrauzer (1968). The structures of an analogous compound (Buehler *et al.*, 2007) and further purine derivatives related to the title compound have been reported (Kowalska *et al.*, 1999; Houlton *et al.*, 1999; Takimoto *et al.*, 1983; Hockova *et al.*, 1999; Sood *et al.*, 1998; Baumann *et al.*, 1994).



Experimental

Crystal data

$C_{13}H_{11}N_5O$
M_r = 253.27
Monoclinic, $P2_1/n$
a = 5.2098 (9) Å
b = 17.738 (3) Å
c = 13.046 (3) Å
β = 97.998 (19)°
V = 1193.9 (4) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.79 mm^{-1}
T = 193 (2) K
0.50 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
2521 measured reflections
2266 independent reflections
1715 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
3 standard reflections
frequency: 60 min
intensity decay: 5%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.136
S = 1.02
2266 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N19—H19A···N6^{i}	0.96	2.01	2.922 (2)	157
N19—H19B···N8^{ii}	0.94	2.17	2.984 (2)	144

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $x+\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *CORINC* (Dräger & Gattow, 1971); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

We are grateful to BERGHOF Products & Instruments GmbH, Eningen, Germany, for the high-pressure reactor BR-25 and for technical support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2589).

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**. 435–436.
Bader, H. & Chiang, Y. H. (1983). US Patent 4 405 781 A19 830 920.
Baumann, T. W., Schulthess, B. H., Linden, A. & Ruedi, P. (1994). *Phytochemistry*, **36**, 537–542.
Buehler, S., Schollmeyer, D., Hauser, D., Laufer, S. & Peifer, C. (2007). *Acta Cryst.* E**63**, o4154–o4155.
Dalby, C., Bleasdale, C., Clegg, W., Elsegood, M. R. J., Golding, B. T. & Griffin, R. J. (1993). *Angew. Chem. Int. Ed. Engl.* **105**, 1822–1823.
Dräger, M. & Gattow, G. (1971). *Acta Chem. Scand.* **25**, 761–762.
Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Hockova, D., Budesinsky, M., Marek, R., Marek, J. & Holy, A. (1999). *Eur. J. Org. Chem.* pp. 2675–2682.
Hopkins, A. L. & Groon, C. R. (2002). *Nat. Rev. Drug Discov.* **1**, 727–730.
Houlton, A., Isaac, C. J., Gibson, A. E., Horrocks, B. R., Clegg, W. & Elsegood, M. R. J. (1999). *J. Chem. Soc. Dalton Trans.* pp. 3229–3234.
Kowalska, A., Pluta, K., Maslankiewicz, R. & Luboradzki, R. (1999). *J. Chem. Crystallogr.* **29**, 103–106.
Laufer, S. A., Domeyer, D. M., Scior, T. R. F., Albrecht, W. & Hauser, D. R. J. (2005). *J. Med. Chem.* **48**, 710–722.
Marzilli, L. G., Epps, L. A., Sorrell, T. & Kistenmacher, T. J. (1975). *J. Am. Chem. Soc.* **97**, 3351–3358.
Meijer, L. & Raymond, E. (2003). *Acc. Chem. Res.* **36**, 417–425.

  doi:10.1107/S1600536807056450

Schrauzer, G. N. (1968). *Inorg. Synth.* **11**, 61–70.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Sood, G., Schwalbe, C. H. & Fraser, W. (1998). *Acta Cryst.* C**54**, 1316–1318.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Takimoto, M., Takenaka, A. & Sasada, Y. (1983). *Acta Cryst.* C**39**, 73–75.

supplementary materials

Acta Cryst. (2007). E**63**, o4646-o4647 [**doi:10.1107/S1600536807056450**]

2-(6-Amino-7*H*-purin-7-yl)-1-phenylethanone

S. Buehler, D. Schollmeyer, D. Hauser, S. Laufer and C. Peifer

Comment

Protein kinases (PK) are favoured targets for the development of new drugs (Hopkins & Groon, 2002) because the reversible protein; phosphorylation by PK is an important control mechanism in signal pathways of a cell (Laufer *et al.*, 2005). In the title compound, the purine system is combined with an acetophenone unit in order to interact with the active site of protein kinases (Laufer *et al.*, 2005). Purine derivatives have been reported as inhibitors for other PK, mainly cyclin- dependent kinases (Meijer & Raymond, 2003). The general synthetic procedure for **3** and **5** is illustrated in Figure 3 (Dalby *et al.*, 1993). The preparation of **1** and of the auxiliary methyl(aqua)cobaloxime- complex $CH_3Co(DH)_2OH_2$ (Marzilli *et al.*, 1975) was performed according to the published procedures (Bader & Chiang, 1983; Schrauzer, 1968). The analogue compound **4** (Buehler *et al.*, 2007) and further purine derivatives related to **5** have been published as crystal structures (Kowalska *et al.*, 1999; Houlton *et al.*, 1999; Takimoto *et al.*, 1983; Hockova *et al.*, 1999; Sood *et al.*, 1998; Baumann *et al.*, 1994).

Compound **5** was prepared as an inhibitor of the Vascular Endothelial Growth Factor Receptor (VEGF-*R*). In the design of compound **5** the purine system from the cosubstrat ATP of these protein kinase (PK) is combined with an acetophenone moiety in order to interact with the hydrophobic region of the PK. In general, the reversible protein - phosphorylation by PK is an important control mechanism in signal pathways of a cell.

The X-ray crystal structure of compound **5** was determined to investigate if an intramolecular 8-membered ring was formed by the interaction of the N19 amino- group to the neighbour carbonyl- oxygen-atom O-12 of the acetophenone moiety. This intramolecular H-bond may influence the conformation of **5** in the binding pocket, and thereby accounting for biological activity. However, this interaction was not detected in the crystal structure. In fact, these two functional groups are rotated in opposite directions. The analysis of the crystal structure of **5** shows that the amino- group of the one purine- molecule links another purine- ring system by the building of two intermolecular hydrogen bonds N—H···N to the nitrogen- atoms N-3 and N-9, whereas the N-3···H distance is 2.01 Å. The length of the second hydrogen bond N-9···H is 2.17 Å.

The synthetis of **5** (Figure 3) starts from 6- chloropurine **1** showing a tautomerism between the 7*H*- and the 9*H*- purine, in which the 9*H*- isomer is the favoured form. Thus, the direct alkylation of **1** results in mainly N-9- substituted purines with the N-7 substitution as the minor product. In order to obtain a regioselective N-7- alkylation $CH_3Co(DH)_2OH_2$ was used as an auxiliary. The complex of $CH_3Co(DH)_2OH_2$ and purine forms an intramolecular N—H···O hydrogen bond from purine N-9 to dimethylglyoximato-O-1 and this indirect shielding prevents the N-9- alkylation of **1**. As a consequence, the coordination of the cobalt- atom to the N-7 atom of the purine is not possible because of the sterical hindrance of the neighbour C-6 halogen substituent. Hence, due to the temporary protection of the N-3 and N-9 positions of **1**, by addition of ω- bromoacetophenone, the solid **2** was obtained as the main product and the N-9 alkylated isomer **3** as the minor product. Subsequently, treatment of compound **2** with methanolic ammonia in a high pressure reactor yielded the 6- methoxy- substituted compound **4** (49,5%) as a main product, which crystal structure has been published (Buehler *et al.*, 2007) and the adenine derivative **5** (34,9%) as a byproduct.

Experimental

Regioselective N-7- alkylation of 6- chloropurine **1** for the preparation of 2-(6-chlor-7*H*-purin-7-yl)-1-phenylethanone **2:** To a solution of methyl(aqua)cobaloxime $CH_3Co(DH)_2OH_2$ (1.55 mmol) in anhydrous acetonitrile (10 ml) was added 6-chloropurine **1** (1.55 mmol) under vigorous stirring and under light exclusion. After the orange purinecobaloxime- complex had precipitated, K_2CO_3 (1.55 mmol) and acetonitrile (5 ml) were added and the reaction mixture was stirred for another 30 min. After the addition of ω- bromoacetophenone (1.55 mmol) the progress of the reaction was monitored by thin - layer chromatography (ethyl acetate: ethanol 9:1). After the reaction was completed, acetonitrile was evaporated and aqueous NaOH (20 ml, 4 *M*) was added. The aqueous layer was extracted with dichloromethane, and the combined organic extracts were dried over Na_2SO_4 and evaporated. The residue was purified by flash column chromatography using ethyl acetate: ethanol (9:1) to give **2** (R_f = 0.49 (ethyl acetate: ethanol 9:1)) as a colourless solid (45.0%). The byproduct 2-(6-chlor-9*H*-purin-9-yl)-1-phenylethanone **3** (R_f = 0.76 (ethyl acetate: ethanol 9:1)) was isolated with a yield of 4.7% (Dalby *et al.*, 1993).

For the synthesis of 2-(6-amino-7*H*-purine-7-yl)-1-phenylethanone **5**, NH_3 (5 ml) was added to a solution of **3** (1.36 mmol) in 15 ml me thanol. The reaction mixture was heated at T = 363 K in a high pressure reactor from *BERGHOF*. The progress was again monitored by thin - layer chromatography (ethyl acetate: ethanol 9:1). After cooling to rt, water was added and the mixture extracted with ethyl acetate, dried over Na_2SO_4 and evaporated. The residue was purified by flash column chromatography using ethyl acetate: ethanol (9:1) to yield 49.5% of **4** (R_f = 0.70, ethyl acetate: ethanol 1:1) and 2-(6-amino-7*H*-purine-7-yl)-1-phenylethanone **5** (34.9%, R_f = 0.43, ethyl acetate: ethanol 1:1) as a byproduct. Crystals of **5** for X-ray analysis precipitated as colourless needles by slow evaporation of ethanol- diethylether solution.

Refinement

Hydrogen atoms attached to carbons were placed at calculated positions with C—H=0.95A% (aromatic) or 0.99–1.00 Å (sp^3 C-atom). Hydrogen atom attached to N19 were located in diff. fourier maps. All H atoms were refined with isotropic displacement parameters (set at 1.2–1.5 times of the U_{eq} of the parent atom).

Figures

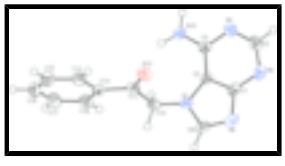


Fig. 1. *ORTEPII* (Johnson, 1968) view of one molecule of **5**. Displacement ellipsoids are drawn at the 50% probability level. H atoms are depicted as circles of arbitrary size.

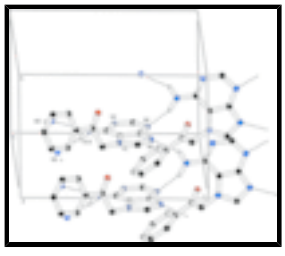


Fig. 2. Part of the crystal packing of compound **5**. Only important H atoms are shown.

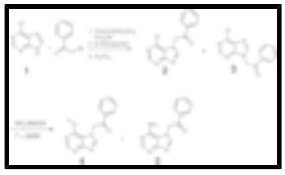


Fig. 3. Synthesis of compounds **4** and **5**.

2-(6-Amino-7*H*-purin-7-yl)-1-phenylethanone

Crystal data

$C_{13}H_{11}N_5O$	F_{000} = 528
M_r = 253.27	D_x = 1.409 Mg m^{-3}
Monoclinic, $P2_1/n$	Cu $K\alpha$ radiation λ = 1.54178 Å
Hall symbol: -P 2yn	Cell parameters from 25 reflections
a = 5.2098 (9) Å	θ = 25–39º
b = 17.738 (3) Å	μ = 0.79 mm^{-1}
c = 13.046 (3) Å	T = 193 (2) K
β = 97.998 (19)º	Needle, colourless
V = 1193.9 (4) Å^3	0.50 × 0.10 × 0.10 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	θ_{max} = 70.1º
Monochromator: graphite	θ_{min} = 4.2º
T = 193(2) K	h = −6→0
ω/2θ scans	k = 0→21
Absorption correction: none	l = −15→15
2521 measured reflections	3 standard reflections
2266 independent reflections	every 60 min
1715 reflections with $I > 2\sigma(I)$	intensity decay: 5%
R_{int} = 0.040	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.047	H-atom parameters constrained
$wR(F^2)$ = 0.136	$w = 1/[\sigma^2(F_o^2) + (0.0801P)^2 + 0.080P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max} < 0.001$
2266 reflections	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
172 parameters	$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Primary atom site location: structure-invariant direct methods

Extinction correction: none

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 $F^2 > 2\mathrm{sigma}(F^2)$ 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}
N1	0.2317 (3)	0.32674 (9)	0.53914 (12)	0.0271 (4)
C2	0.4370 (3)	0.27657 (10)	0.55492 (13)	0.0242 (4)
C3	0.5890 (3)	0.23729 (10)	0.49112 (14)	0.0251 (4)
N4	0.7799 (3)	0.19328 (10)	0.53861 (12)	0.0327 (4)
C5	0.8118 (4)	0.18891 (12)	0.64190 (16)	0.0369 (5)
H5	0.9491	0.1572	0.6717	0.044*
N6	0.6790 (4)	0.22254 (10)	0.70889 (12)	0.0346 (4)
C7	0.4892 (4)	0.26716 (10)	0.66170 (14)	0.0278 (4)
N8	0.3232 (3)	0.30991 (10)	0.71111 (13)	0.0347 (4)
C9	0.1762 (4)	0.34381 (12)	0.63502 (16)	0.0345 (5)
H9	0.0415	0.3777	0.6462	0.041*
C10	0.1080 (4)	0.36032 (11)	0.44408 (15)	0.0295 (4)
H10A	−0.0544	0.3853	0.4570	0.035*
H10B	0.0618	0.3201	0.3921	0.035*
C11	0.2806 (3)	0.41779 (11)	0.40063 (15)	0.0278 (4)
O12	0.5035 (3)	0.42658 (9)	0.44132 (11)	0.0388 (4)
C13	0.1692 (4)	0.46102 (11)	0.30769 (15)	0.0300 (4)
C14	0.3089 (4)	0.52134 (11)	0.27538 (17)	0.0365 (5)
H14	0.4693	0.5352	0.3146	0.044*
C15	0.2178 (5)	0.56100 (13)	0.18757 (18)	0.0446 (6)
H15	0.3139	0.6024	0.1668	0.054*
C16	−0.0146 (5)	0.54055 (13)	0.12922 (17)	0.0449 (6)
H16	−0.0768	0.5674	0.0677	0.054*
C17	−0.1551 (5)	0.48118 (14)	0.16068 (18)	0.0460 (6)
H17	−0.3144	0.4672	0.1206	0.055*
C18	−0.0666 (4)	0.44154 (12)	0.25024 (17)	0.0378 (5)
H18	−0.1664	0.4013	0.2722	0.045*
N19	0.5613 (3)	0.24173 (10)	0.38802 (11)	0.0302 (4)
H19A	0.4165	0.2622	0.3426	0.045*
H19B	0.6721	0.2107	0.3557	0.045*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0213 (8)	0.0339 (8)	0.0278 (8)	0.0003 (6)	0.0092 (6)	−0.0004 (6)
C2	0.0205 (8)	0.0287 (9)	0.0238 (9)	−0.0023 (7)	0.0043 (7)	0.0006 (7)
C3	0.0220 (9)	0.0298 (9)	0.0235 (9)	−0.0036 (7)	0.0034 (7)	−0.0015 (7)
N4	0.0298 (9)	0.0376 (9)	0.0299 (9)	0.0054 (7)	0.0010 (7)	−0.0022 (7)
C5	0.0349 (11)	0.0398 (11)	0.0334 (11)	0.0052 (9)	−0.0046 (9)	0.0020 (9)
N6	0.0378 (10)	0.0411 (9)	0.0233 (8)	−0.0046 (8)	−0.0016 (7)	0.0031 (7)
C7	0.0292 (10)	0.0315 (9)	0.0232 (9)	−0.0096 (8)	0.0059 (7)	−0.0002 (7)
N8	0.0392 (10)	0.0404 (9)	0.0276 (8)	−0.0044 (8)	0.0154 (7)	−0.0014 (7)
C9	0.0329 (11)	0.0390 (11)	0.0360 (11)	−0.0021 (9)	0.0196 (9)	−0.0024 (9)
C10	0.0192 (9)	0.0358 (10)	0.0343 (10)	0.0013 (8)	0.0064 (8)	0.0035 (8)
C11	0.0206 (9)	0.0299 (9)	0.0339 (10)	0.0035 (7)	0.0067 (8)	−0.0015 (8)
O12	0.0214 (7)	0.0450 (8)	0.0494 (9)	−0.0019 (6)	0.0033 (6)	0.0074 (7)
C13	0.0276 (10)	0.0320 (10)	0.0324 (10)	0.0053 (8)	0.0109 (8)	0.0001 (8)
C14	0.0342 (11)	0.0324 (10)	0.0444 (12)	0.0018 (9)	0.0110 (9)	0.0028 (9)
C15	0.0523 (14)	0.0366 (11)	0.0480 (13)	0.0032 (10)	0.0181 (11)	0.0085 (10)
C16	0.0589 (16)	0.0415 (12)	0.0355 (11)	0.0150 (11)	0.0104 (11)	0.0092 (9)
C17	0.0403 (13)	0.0553 (14)	0.0410 (13)	0.0072 (11)	0.0012 (10)	0.0062 (10)
C18	0.0285 (10)	0.0422 (11)	0.0422 (12)	0.0019 (9)	0.0037 (9)	0.0089 (9)
N19	0.0287 (9)	0.0408 (9)	0.0218 (8)	0.0040 (7)	0.0060 (7)	−0.0041 (6)

Geometric parameters (Å, °)

N1—C9	1.357 (2)	C13—C14	1.392 (3)
N1—C2	1.384 (2)	C14—C15	1.372 (3)
N1—C10	1.444 (2)	C15—C16	1.386 (4)
C2—C7	1.392 (2)	C16—C17	1.377 (3)
C2—C3	1.411 (2)	C17—C18	1.386 (3)
C3—N19	1.335 (2)	N19—H19A	0.9600
C3—N4	1.346 (2)	N19—H19B	0.9400
N4—C5	1.337 (3)	C5—H5	0.9500
C5—N6	1.329 (3)	C9—H9	0.9500
N6—C7	1.347 (3)	C10—H10A	0.9900
C7—N8	1.376 (3)	C10—H10B	0.9900
N8—C9	1.312 (3)	C14—H14	0.9500
C10—C11	1.520 (3)	C15—H15	0.9500
C11—O12	1.218 (2)	C16—H16	0.9500
C11—C13	1.483 (3)	C17—H17	0.9500
C13—C18	1.391 (3)	C18—H18	0.9500
C9—N1—C2	105.41 (16)	C17—C16—C15	119.8 (2)
C9—N1—C10	124.94 (17)	C16—C17—C18	120.7 (2)
C2—N1—C10	129.50 (15)	C17—C18—C13	119.6 (2)
N1—C2—C7	105.39 (16)	H19A—N19—H19B	115.00
N1—C2—C3	135.67 (17)	C3—N19—H19A	127.00
C7—C2—C3	118.93 (17)	C3—N19—H19B	116.00

N19—C3—N4	117.87 (17)	N4—C5—H5	115.00
N19—C3—C2	125.07 (17)	N6—C5—H5	115.00
N4—C3—C2	117.04 (17)	N1—C9—H9	123.00
C5—N4—C3	118.54 (17)	N8—C9—H9	123.00
N6—C5—N4	129.34 (19)	N1—C10—H10A	109.00
C5—N6—C7	112.29 (16)	N1—C10—H10B	109.00
N6—C7—N8	125.38 (17)	C11—C10—H10A	109.00
N6—C7—C2	123.84 (18)	C11—C10—H10B	109.00
N8—C7—C2	110.78 (17)	H10A—C10—H10B	108.00
C9—N8—C7	103.70 (15)	C13—C14—H14	120.00
N8—C9—N1	114.73 (18)	C15—C14—H14	120.00
N1—C10—C11	112.35 (16)	C14—C15—H15	120.00
O12—C11—C13	122.09 (18)	C16—C15—H15	120.00
O12—C11—C10	120.06 (18)	C15—C16—H16	120.00
C13—C11—C10	117.84 (16)	C17—C16—H16	120.00
C18—C13—C14	119.2 (2)	C16—C17—H17	120.00
C18—C13—C11	121.87 (18)	C18—C17—H17	120.00
C14—C13—C11	118.91 (19)	C13—C18—H18	120.00
C15—C14—C13	120.8 (2)	C17—C18—H18	120.00
C14—C15—C16	119.9 (2)		
C9—N1—C2—C7	0.1 (2)	C7—N8—C9—N1	0.1 (2)
C10—N1—C2—C7	175.72 (18)	C2—N1—C9—N8	−0.2 (2)
C9—N1—C2—C3	−179.6 (2)	C10—N1—C9—N8	−176.00 (17)
C10—N1—C2—C3	−4.0 (3)	C9—N1—C10—C11	104.5 (2)
N1—C2—C3—N19	0.3 (3)	C2—N1—C10—C11	−70.3 (2)
C7—C2—C3—N19	−179.34 (17)	N1—C10—C11—O12	5.6 (3)
N1—C2—C3—N4	178.9 (2)	N1—C10—C11—C13	−175.16 (16)
C7—C2—C3—N4	−0.8 (3)	O12—C11—C13—C18	167.90 (19)
N19—C3—N4—C5	179.31 (18)	C10—C11—C13—C18	−11.3 (3)
C2—C3—N4—C5	0.7 (3)	O12—C11—C13—C14	−10.1 (3)
C3—N4—C5—N6	0.1 (3)	C10—C11—C13—C14	170.72 (17)
N4—C5—N6—C7	−0.6 (3)	C18—C13—C14—C15	−0.6 (3)
C5—N6—C7—N8	−178.92 (18)	C11—C13—C14—C15	177.45 (18)
C5—N6—C7—C2	0.4 (3)	C13—C14—C15—C16	−0.8 (3)
N1—C2—C7—N6	−179.52 (17)	C14—C15—C16—C17	1.0 (3)
C3—C2—C7—N6	0.2 (3)	C15—C16—C17—C18	0.0 (4)
N1—C2—C7—N8	−0.1 (2)	C16—C17—C18—C13	−1.3 (3)
C3—C2—C7—N8	179.68 (16)	C14—C13—C18—C17	1.6 (3)
N6—C7—N8—C9	179.42 (19)	C11—C13—C18—C17	−176.35 (19)
C2—C7—N8—C9	0.0 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N19—H19A···N6^{i}	0.96	2.01	2.922 (2)	157
N19—H19B···N8^{ii}	0.94	2.17	2.984 (2)	144

Symmetry codes: (i) *x*−1/2, −*y*+1/2, *z*−1/2; (ii) *x*+1/2, −*y*+1/2, *z*−1/2.

Fig. 1

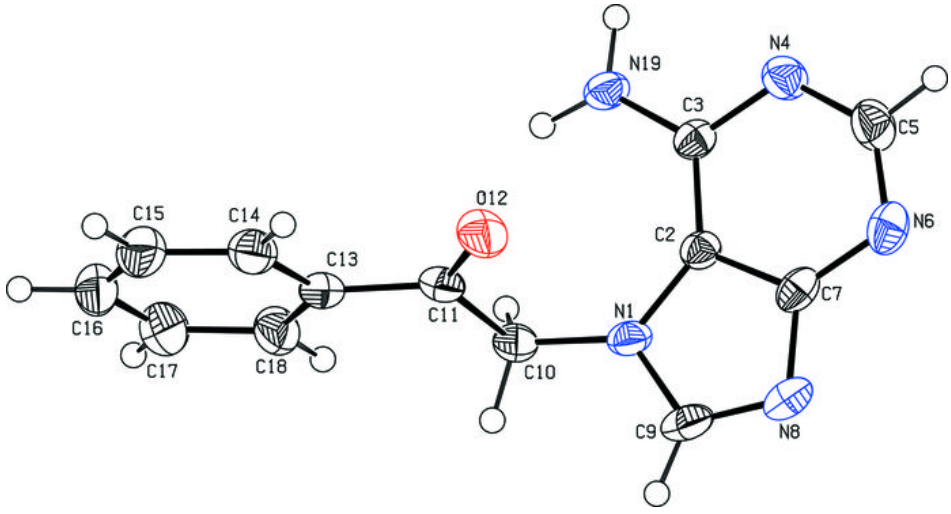


Fig. 2

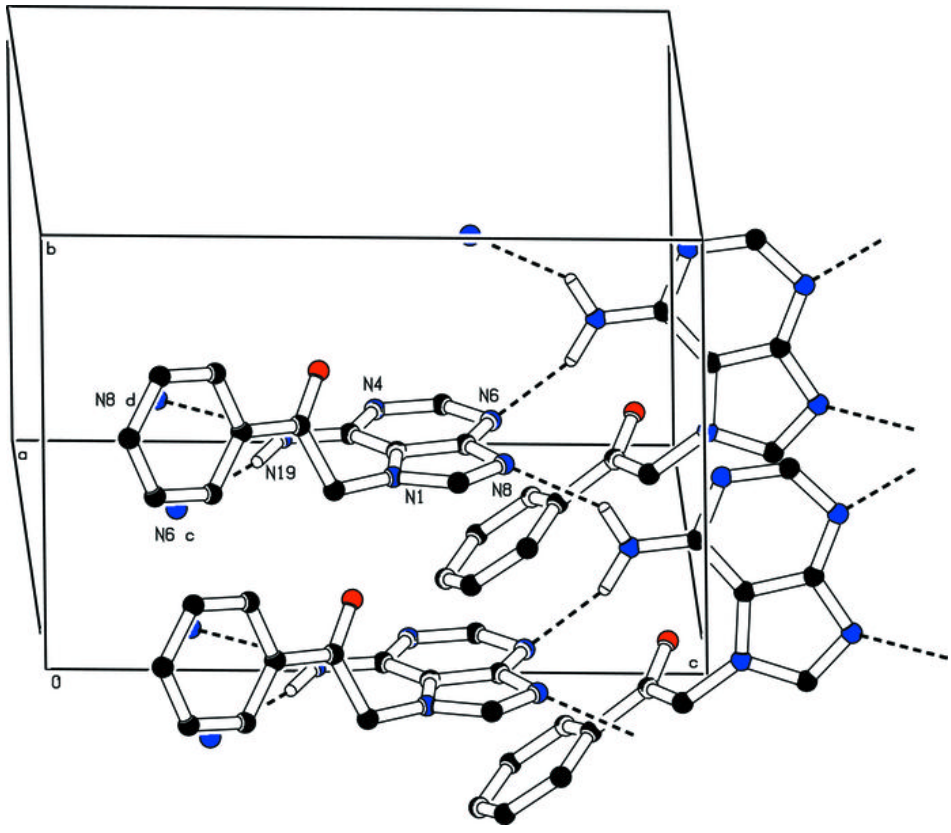
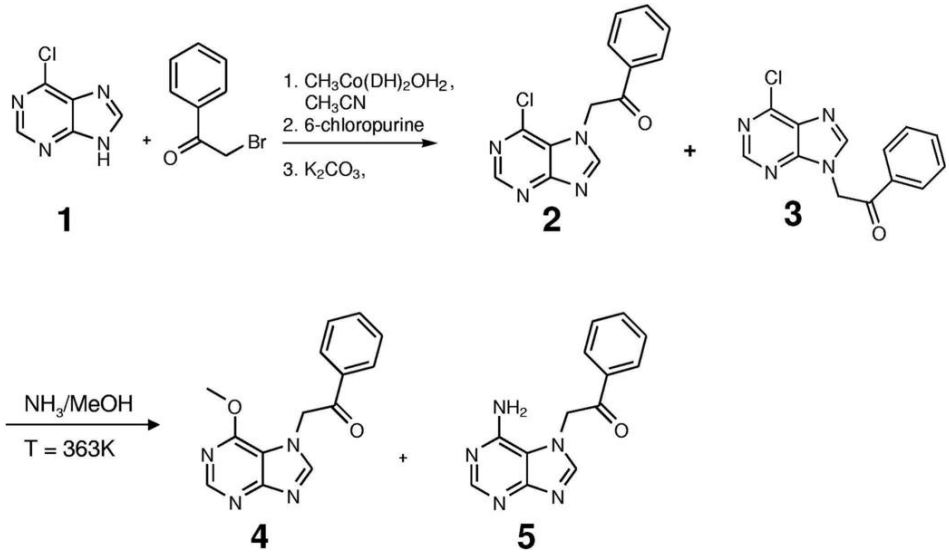


Fig. 3


Cl
N
N
N
H
1
Br
O
1. $CH_3Co(DH)_2OH_2$, CH_3CN
2. 6-chloropurine
3. K_2CO_3,
2
3
NH_3/MeOH
T = 363K
4
5
NH_2