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

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

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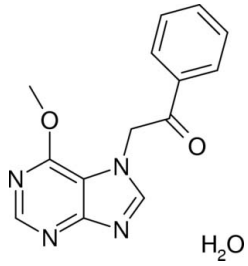
Key indicators: single-crystal X-ray study; T = 193 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.127; data-to-parameter ratio = 12.5.

The crystal structure of the title compound, $C_{14}H_{12}N_4O_2\cdot H_2O$, was determined in the course of our studies of the synthesis and optimization of 7-aryl-7*H*-purines as inhibitors of the vascular endothelial growth factor receptor (VEGF-R), c-Jun NH_2-terminal protein kinase 3 (JNK3) and the p38α mitogen-activated protein kinase (MAPK). In the title compound, two molecules are associated with each other through O—H···N hydrogen bonds to different N atoms in the purine ring system. The compound was prepared *via* a regioselective synthesis using the methyl(aqua)cobaloxime complex, $CH_3Co(DH)_2OH_2$, as a temporary auxiliary. The X-ray crystallographic results confirmed the regioselective N-7 alkylation of this molecule.

Related literature

Background: Hopkins & Groon (2002); Laufer *et al.* (2005); Meijer & Raymond (2003). Synthesis: Dalby *et al.* (1993); Marzilli *et al.* (1975); Bader & Chiang (1983); Schrauzer (1968). Related purine derivatives: 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

$C_{14}H_{12}N_4O_2\cdot H_2O$
M_r = 286.29
Monoclinic, $P2_1/c$
a = 12.2729 (6) Å
b = 8.2830 (7) Å
c = 14.3335 (10) Å
β = 112.851 (3)°
V = 1342.74 (16) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.86 mm^{-1}
T = 193 (2) K
0.40 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
2645 measured reflections
2534 independent reflections
2089 reflections with $I > 2\sigma(I)$
R_{int} = 0.083
3 standard reflections
frequency: 60 min
intensity decay: 5%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.127
S = 1.08
2534 reflections
202 parameters
Only H-atom displacement parameters refined
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1W···N8	0.96	2.00	2.923 (2)	159
O1W—H2W···N6^{i}	0.92	2.03	2.9066 (19)	159

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

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) and *ORTEP* (Johnson, 1968); 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: FL2156).

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supplementary materials

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

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

Comment

Compound **4** was prepared as an inhibitor of the Vascular Endothelial Growth Factor Receptor (VEGF-*R*). In **4** the purine system from the cosubstrate ATP of these protein kinases (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.

In 4 a water links two symmetry related (2-fold screw axis) purine molecules by H-bonding to N6 and N8, respectively. In turn, each purine molecule hydrogen bonds to two water molecules such that the phenyl rings protrude from opposite sides of an infinite sheet of purine moieties. Thus, a layer-like structure is formed perpendicular to the *a* axis. The phenyl ring is oriented approximately perpendicular (67.33 (6)°) to the purine ring system.

Experimental

The synthetis of 4 (scheme 1) 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 CH3Co(DH)2OH2 was used as an auxiliary. The complex 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. A s a consequence, the coordination of the cobalt- atom to the N-7 atom of the purine is not possible because of steric hindrance from the neighbouring C-6 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%) and the adenine derivative 5 (34,9%).

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 addded 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-methoxy-7*H*-purine-7-yl)-1-phenylethanone **4**, 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 **4** for X-ray analysis precipitated as colourless sheets 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 O1W were located in diff. fourier maps. H atoms attached to carbon atoms were refined with isotropic displacement parameters using a riding motion model with fixed C—H distance. The O1W—H distance was free to refine with fixed O1W—H vector direction.

Figures

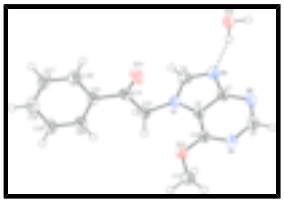


Fig. 1. *ORTEPII* (Johnson, 1968) view of one molecule of **4**. Displacement ellipsoids are drawn at the 50% probability level. H atoms are depicted as circles of arbitrary size.

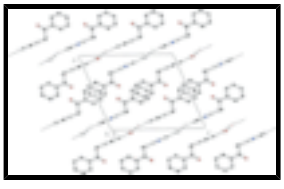


Fig. 2. Crystal packing of compound **4** viewed along the *b* axis. Only important H atoms are shown.

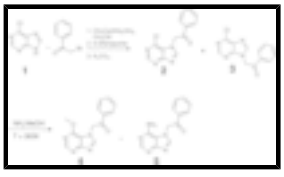


Fig. 3. Synthesis of compounds **4** and **5**.

2-(6-Methoxy-7*H*-purine-7-yl)-1-phenylethanone

Crystal data

$C_{14}H_{12}N_4O_2·H_2O$	F_{000} = 600
M_r = 286.29	D_x = 1.416 Mg m^{-3}
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation λ = 1.54178 Å
Hall symbol: -P 2ybc	Cell parameters from 25 reflections
a = 12.2729 (6) Å	θ = 61–70º
b = 8.2830 (7) Å	μ = 0.86 mm^{-1}
c = 14.3335 (10) Å	T = 193 (2) K
β = 112.851 (3)º	Plate, colourless

V = 1342.74 (16) Å^3	0.40 × 0.20 × 0.10 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	θ_{max} = 70.2º
Monochromator: graphite	θ_{min} = 3.9º
T = 193(2) K	h = −13→14
ω/2θ scans	k = 0→10
Absorption correction: none	l = −17→0
2645 measured reflections	3 standard reflections
2534 independent reflections	every 60 min
2089 reflections with $I > 2\sigma(I)$	intensity decay: 5%
R_{int} = 0.083	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.044	Only H-atom displacement parameters refined
$wR(F^2)$ = 0.127	$w = 1/[\sigma^2(F_o^2) + (0.0712P)^2 + 0.325P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.08	$(\Delta/\sigma)_{max} < 0.001$
2534 reflections	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
202 parameters	$\Delta\rho_{min}$ = −0.27 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 $F^2 > 2$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	0.18404 (12)	0.47672 (17)	0.62611 (10)	0.0285 (3)
C2	0.16775 (14)	0.32546 (19)	0.58263 (12)	0.0257 (4)
C3	0.18454 (14)	0.1669 (2)	0.61821 (12)	0.0262 (4)

N4	0.15423 (12)	0.04636 (17)	0.55245 (11)	0.0307 (3)
C5	0.10739 (15)	0.0839 (2)	0.45274 (13)	0.0330 (4)
H5	0.0840	−0.0050	0.4073	0.037 (5)*
N6	0.09001 (13)	0.22894 (19)	0.41046 (10)	0.0329 (4)
C7	0.12163 (14)	0.3509 (2)	0.47865 (12)	0.0282 (4)
N8	0.11165 (13)	0.51367 (19)	0.45755 (11)	0.0339 (4)
C9	0.14978 (15)	0.5824 (2)	0.54765 (13)	0.0331 (4)
H9	0.1509	0.6954	0.5624	0.031 (5)*
O10	0.23226 (11)	0.14118 (14)	0.71795 (9)	0.0325 (3)
C11	0.2425 (2)	−0.0240 (2)	0.75123 (15)	0.0488 (6)
H11A	0.1661	−0.0783	0.7185	0.068 (5)*
H11B	0.2664	−0.0271	0.8249	0.068 (5)*
H11C	0.3021	−0.0792	0.7329	0.068 (5)*
C12	0.23133 (15)	0.5177 (2)	0.73339 (12)	0.0294 (4)
H12A	0.2233	0.4235	0.7728	0.044 (4)*
H12B	0.1849	0.6077	0.7448	0.044 (4)*
C13	0.36068 (15)	0.5666 (2)	0.77075 (12)	0.0294 (4)
O14	0.41546 (12)	0.5476 (2)	0.71708 (10)	0.0485 (4)
C15	0.41426 (14)	0.6416 (2)	0.87259 (12)	0.0278 (4)
C16	0.52074 (16)	0.7238 (3)	0.89910 (15)	0.0397 (5)
H16	0.5591	0.7292	0.8530	0.049 (6)*
C17	0.57072 (18)	0.7978 (3)	0.99248 (17)	0.0478 (5)
H17	0.6427	0.8558	1.0099	0.077 (9)*
C18	0.51658 (17)	0.7878 (3)	1.06072 (15)	0.0435 (5)
H18	0.5517	0.8378	1.1252	0.051 (6)*
C19	0.41193 (17)	0.7056 (3)	1.03499 (14)	0.0425 (5)
H19	0.3749	0.6983	1.0820	0.069 (8)*
C20	0.36001 (16)	0.6333 (2)	0.94135 (14)	0.0367 (4)
H20	0.2870	0.5778	0.9238	0.048 (6)*
O1W	0.04215 (13)	0.77041 (17)	0.30574 (9)	0.0418 (4)
H1W	0.0523 (2)	0.670 (2)	0.3418 (7)	0.068 (6)*
H2W	−0.0121 (12)	0.7424 (6)	0.2431 (13)	0.068 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0327 (7)	0.0265 (7)	0.0236 (7)	0.0001 (6)	0.0079 (6)	−0.0012 (5)
C2	0.0244 (8)	0.0289 (8)	0.0228 (8)	0.0008 (6)	0.0081 (6)	−0.0006 (6)
C3	0.0233 (7)	0.0295 (9)	0.0242 (8)	0.0001 (6)	0.0075 (6)	−0.0013 (6)
N4	0.0296 (7)	0.0298 (7)	0.0300 (8)	0.0000 (6)	0.0087 (6)	−0.0044 (6)
C5	0.0313 (9)	0.0377 (10)	0.0286 (9)	−0.0013 (7)	0.0100 (7)	−0.0091 (8)
N6	0.0332 (8)	0.0404 (9)	0.0229 (7)	−0.0004 (6)	0.0085 (6)	−0.0041 (6)
C7	0.0259 (8)	0.0350 (9)	0.0219 (8)	0.0001 (7)	0.0074 (6)	0.0002 (7)
N8	0.0368 (8)	0.0342 (8)	0.0270 (7)	−0.0006 (6)	0.0081 (6)	0.0040 (6)
C9	0.0364 (9)	0.0286 (9)	0.0314 (9)	0.0020 (7)	0.0101 (7)	0.0041 (7)
O10	0.0399 (7)	0.0281 (6)	0.0231 (6)	−0.0008 (5)	0.0055 (5)	0.0030 (5)
C11	0.0731 (15)	0.0312 (10)	0.0331 (10)	−0.0064 (10)	0.0109 (10)	0.0067 (8)
C12	0.0342 (9)	0.0300 (9)	0.0239 (8)	−0.0013 (7)	0.0112 (7)	−0.0038 (7)

C13	0.0325 (9)	0.0295 (8)	0.0264 (8)	0.0029 (7)	0.0116 (7)	0.0036 (7)
O14	0.0405 (7)	0.0767 (11)	0.0337 (7)	−0.0043 (7)	0.0203 (6)	−0.0095 (7)
C15	0.0285 (8)	0.0275 (8)	0.0259 (8)	0.0038 (6)	0.0088 (7)	0.0028 (7)
C16	0.0321 (9)	0.0485 (12)	0.0407 (10)	−0.0040 (8)	0.0164 (8)	−0.0058 (9)
C17	0.0323 (10)	0.0568 (13)	0.0518 (12)	−0.0133 (9)	0.0136 (9)	−0.0167 (11)
C18	0.0375 (10)	0.0517 (12)	0.0356 (10)	−0.0015 (9)	0.0080 (8)	−0.0142 (9)
C19	0.0398 (10)	0.0588 (13)	0.0307 (10)	−0.0053 (9)	0.0158 (8)	−0.0084 (9)
C20	0.0338 (9)	0.0459 (11)	0.0312 (9)	−0.0086 (8)	0.0136 (7)	−0.0047 (8)
O1W	0.0568 (9)	0.0334 (7)	0.0272 (6)	−0.0036 (6)	0.0075 (6)	−0.0010 (5)

Geometric parameters (Å, °)

N1—C9	1.357 (2)	C12—C13	1.520 (2)
N1—C2	1.379 (2)	C12—H12A	0.9900
N1—C12	1.457 (2)	C12—H12B	0.9900
C2—C7	1.389 (2)	C13—O14	1.213 (2)
C2—C3	1.395 (2)	C13—C15	1.484 (2)
C3—N4	1.323 (2)	C15—C20	1.389 (2)
C3—O10	1.335 (2)	C15—C16	1.389 (3)
N4—C5	1.353 (2)	C16—C17	1.380 (3)
C5—N6	1.325 (2)	C16—H16	0.9500
C5—H5	0.9500	C17—C18	1.382 (3)
N6—C7	1.353 (2)	C17—H17	0.9500
C7—N8	1.377 (2)	C18—C19	1.372 (3)
N8—C9	1.320 (2)	C18—H18	0.9500
C9—H9	0.9588	C19—C20	1.379 (3)
O10—C11	1.438 (2)	C19—H19	0.9500
C11—H11A	0.9800	C20—H20	0.9500
C11—H11B	0.9800	O1W—H1W	0.9625
C11—H11C	0.9800	O1W—H2W	0.9160
C9—N1—C2	105.53 (14)	N1—C12—C13	111.55 (13)
C9—N1—C12	126.35 (15)	N1—C12—H12A	109.3
C2—N1—C12	128.10 (14)	C13—C12—H12A	109.3
N1—C2—C7	105.92 (14)	N1—C12—H12B	109.3
N1—C2—C3	135.67 (15)	C13—C12—H12B	109.3
C7—C2—C3	118.40 (15)	H12A—C12—H12B	108.0
N4—C3—O10	121.82 (15)	O14—C13—C15	122.37 (16)
N4—C3—C2	119.29 (15)	O14—C13—C12	120.15 (15)
O10—C3—C2	118.88 (15)	C15—C13—C12	117.46 (14)
C3—N4—C5	117.73 (15)	C20—C15—C16	119.30 (16)
N6—C5—N4	128.18 (16)	C20—C15—C13	121.72 (16)
N6—C5—H5	115.9	C16—C15—C13	118.97 (16)
N4—C5—H5	115.9	C17—C16—C15	119.96 (18)
C5—N6—C7	113.35 (14)	C17—C16—H16	120.0
N6—C7—N8	126.59 (15)	C15—C16—H16	120.0
N6—C7—C2	123.01 (16)	C16—C17—C18	120.34 (18)
N8—C7—C2	110.39 (15)	C16—C17—H17	119.8
C9—N8—C7	103.88 (14)	C18—C17—H17	119.8
N8—C9—N1	114.27 (16)	C19—C18—C17	119.77 (18)

N8—C9—H9	127.3	C19—C18—H18	120.1
N1—C9—H9	118.3	C17—C18—H18	120.1
C3—O10—C11	116.91 (14)	C18—C19—C20	120.52 (18)
O10—C11—H11A	109.5	C18—C19—H19	119.7
O10—C11—H11B	109.5	C20—C19—H19	119.7
H11A—C11—H11B	109.5	C19—C20—C15	120.10 (17)
O10—C11—H11C	109.5	C19—C20—H20	120.0
H11A—C11—H11C	109.5	C15—C20—H20	120.0
H11B—C11—H11C	109.5	H1W—O1W—H2W	101.6
C9—N1—C2—C7	−1.15 (18)	C2—N1—C9—N8	0.7 (2)
C12—N1—C2—C7	−179.42 (15)	C12—N1—C9—N8	179.03 (15)
C9—N1—C2—C3	−179.88 (19)	N4—C3—O10—C11	−3.8 (2)
C12—N1—C2—C3	1.9 (3)	C2—C3—O10—C11	177.03 (16)
N1—C2—C3—N4	177.35 (17)	C9—N1—C12—C13	−78.3 (2)
C7—C2—C3—N4	−1.3 (2)	C2—N1—C12—C13	99.68 (19)
N1—C2—C3—O10	−3.5 (3)	N1—C12—C13—O14	−9.8 (2)
C7—C2—C3—O10	177.92 (14)	N1—C12—C13—C15	168.46 (14)
O10—C3—N4—C5	−179.43 (15)	O14—C13—C15—C20	−166.61 (18)
C2—C3—N4—C5	−0.3 (2)	C12—C13—C15—C20	15.2 (2)
C3—N4—C5—N6	2.0 (3)	O14—C13—C15—C16	14.2 (3)
N4—C5—N6—C7	−1.8 (3)	C12—C13—C15—C16	−164.03 (16)
C5—N6—C7—N8	−178.59 (16)	C20—C15—C16—C17	−0.8 (3)
C5—N6—C7—C2	0.0 (2)	C13—C15—C16—C17	178.45 (19)
N1—C2—C7—N6	−177.56 (15)	C15—C16—C17—C18	1.3 (3)
C3—C2—C7—N6	1.4 (2)	C16—C17—C18—C19	−0.7 (3)
N1—C2—C7—N8	1.25 (19)	C17—C18—C19—C20	−0.3 (3)
C3—C2—C7—N8	−179.76 (14)	C18—C19—C20—C15	0.8 (3)
N6—C7—N8—C9	177.95 (17)	C16—C15—C20—C19	−0.3 (3)
C2—C7—N8—C9	−0.81 (19)	C13—C15—C20—C19	−179.46 (18)
C7—N8—C9—N1	0.1 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1W—H1W···N8	0.96	2.00	2.923 (2)	159
O1W—H2W···N6^{i}	0.92	2.03	2.9066 (19)	159

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

Fig. 1

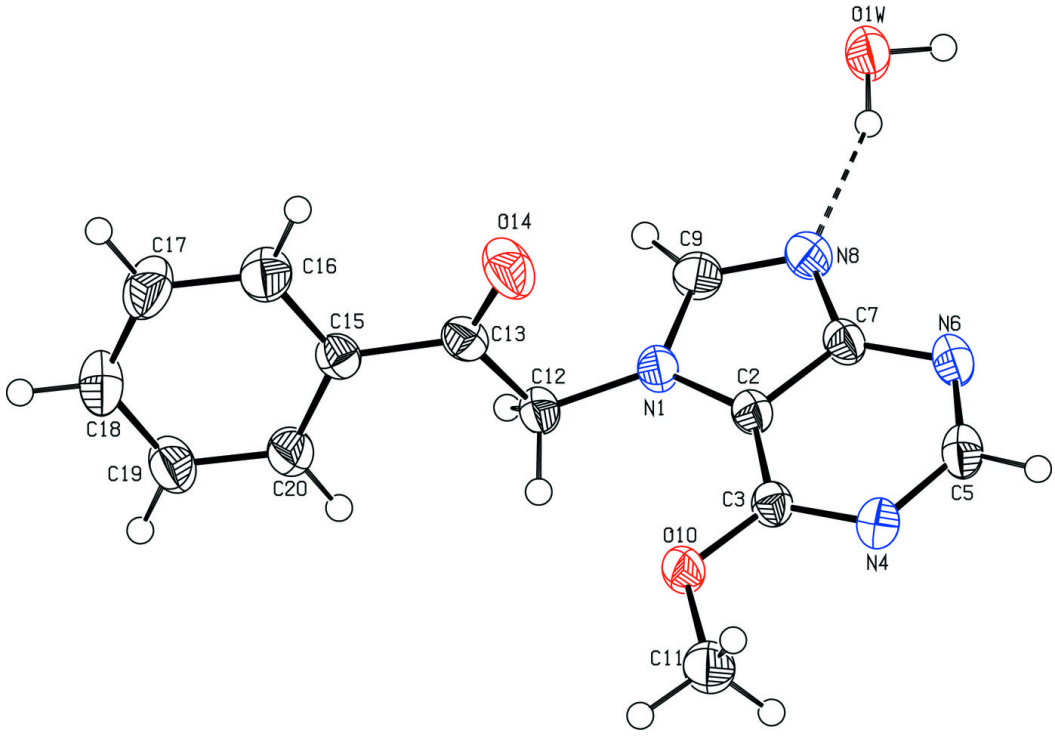

O1W
O14
C17
C16
C15
C13
C9
N8
C7
N6
C12
N1
C2
C18
C19
C20
C3
C5
O10
N4
C11

Fig. 2

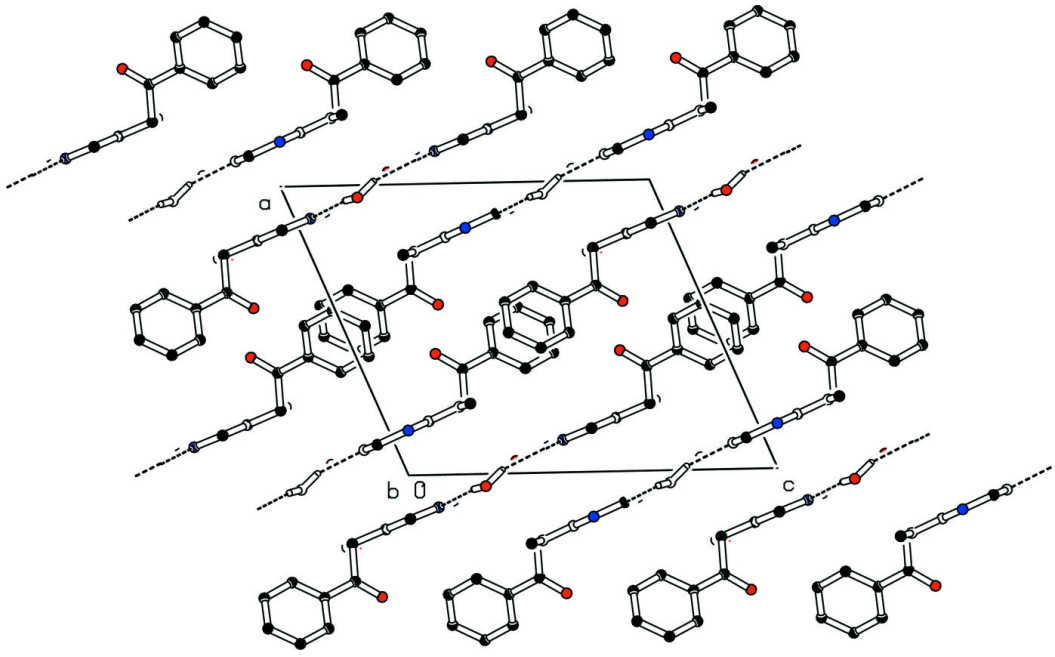
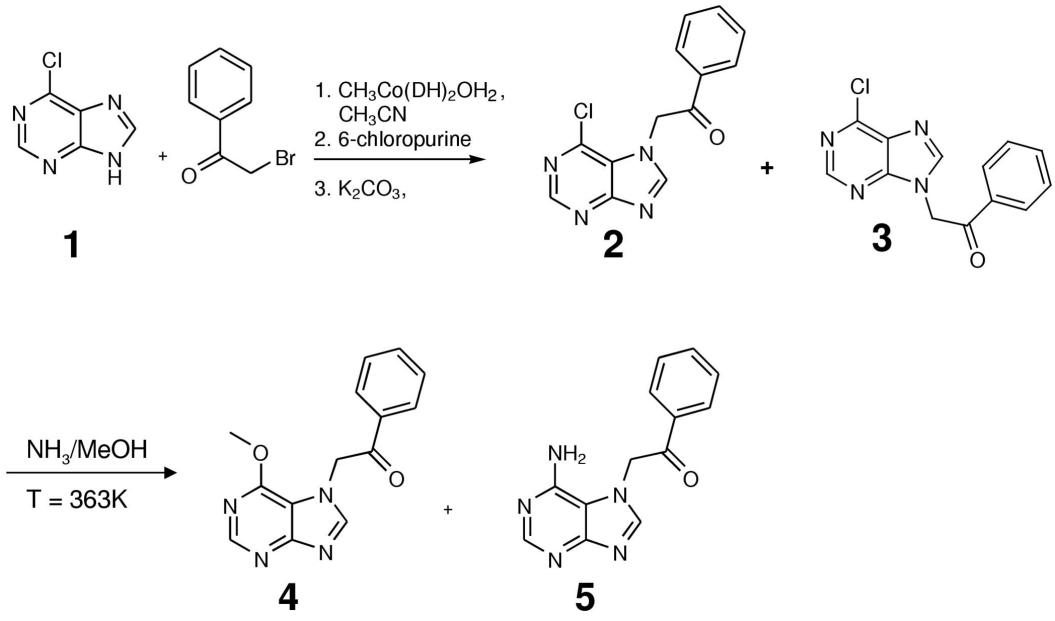


Fig. 3


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