Table 2. *Selected geometric parameters* (Å, °)

As—O1	1.560 (9)	N4—C8	1.36 (1)
As—O2	1.705 (8)	N4—C9	1.35 (1)
As—O3	1.708 (8)	N5—C8	1.32 (1)
As—C1	1.901 (8)	N6—C9	1.31 (1)
N1—C4	1.42 (1)	C1—C2	1.38 (1)
N1—C7	1.37 (1)	C1—C6	1.38 (1)
N2—C7	1.36 (1)	C2—C3	1.38 (1)
N2—C8	1.32 (1)	C3—C4	1.39 (1)
N3—C7	1.33 (1)	C4—C5	1.39 (1)
N3—C9	1.34 (1)	C5—C6	1.41 (1)
O1—As—O2	115.4 (5)	N1—C4—C3	114.4 (7)
O1—As—O3	112.6 (5)	N1—C4—C5	125.4 (8)
O1—As—C1	114.8 (4)	C3—C4—C5	120.3 (8)
O2—As—O3	104.4 (5)	C4—C5—C6	118.3 (8)
O2—As—C1	104.4 (4)	C1—C6—C5	121.8 (8)
O3—As—C1	103.9 (4)	N1—C7—N2	113.7 (7)
C4—N1—C7	129.8 (7)	N1—C7—N3	118.8 (7)
C7—N2—C8	114.2 (7)	N2—C7—N3	127.4 (8)
C7—N3—C9	114.9 (7)	N2—C8—N4	122.6 (8)
C8—N4—C9	118.9 (7)	N2—C8—N5	119.8 (8)
AS—C1—C2	119.6 (7)	N4—C8—N5	117.6 (8)
AS—C1—C6	122.1 (7)	N3—C9—N4	121.9 (8)
C2—C1—C6	118.2 (8)	N3—C9—N6	119.7 (8)
C1—C2—C3	121.7 (9)	N4—C9—N6	118.4 (8)
C2—C3—C4	119.7 (9)		

Although the waters are quite ordered, we were dissatisfied with the refinement of the H atoms associated with them and these H atoms were thus excluded from the final stage of the analysis. The protons required for electroneutrality have not been located. Anomalous-dispersion effects were derived from Ibers & Hamilton (1964).

Computer programs used: *TEXSAN* (Molecular Structure Corporation, 1992), *DIRDIF* (Beurskens, 1984), *MITHRIL* (Gilmore, 1984), *SHELXS86* (Sheldrick, 1985) and *ORTEP* (Johnson, 1965).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1147). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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8-(Noradamantan-3-yl)-1,3-dipropylxanthine

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Abstract

The title compound, KW-3902 {1,3-dipropyl-8-(3-tricyclo[3.3.1.0^{3,7}]nonyl)-3,7-dihydro-1*H*-purine-2,6-dione, $C_{20}H_{28}N_4O_2$}, is a selective adenosine A1-receptor antagonist. In the crystal of KW-3902, the mirror plane of the 3-noradamantyl group is nearly in the plane of the xanthine moiety. The two propyl side chains have fully extended conformations and are on the same side of the xanthine plane.

Comment

Xanthine derivatives block adenosine receptors and exhibit varied pharmacological activities. Because these compounds act as antagonists, it is obvious that their three-dimensional structures are very important in the process of binding to the receptors. To elucidate the structure–activity relationships of xanthine derivatives, we have undertaken the X-ray analysis of a series of these derivatives (Hirayama, Nagahara, Shimada & Suzuki, 1993). The title compound, KW-3902, displays

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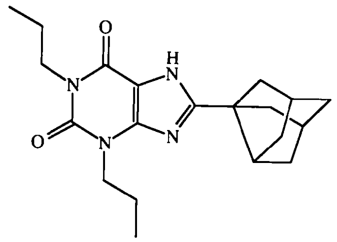
a high affinity for the adenosine A1-receptor and exhibits high potency against acute renal failure (Suzuki *et al.*, 1992).



(KW-3902)

The exocyclic bond angles around C6 are remarkably asymmetric. Similar asymmetry is observed in 3-isobutyl-1-methylxanthine (Srikrishnan & Parthasarathy, 1988) and 8-(dicyclopropylmethyl)-1,3-dipropylxanthine (KF15372; Hirayama, Nagahara, Shimada & Suzuki, 1993). The two propyl groups have all-*trans* conformations, as in KF15372. However, in KW-3902 the propyl groups are extended on the same side of the xanthine plane, in contrast to those of KF15372. The C7′ atom of the noradamantyl group is almost eclipsed by N9. The mirror plane of the noradamantyl group, which is defined by C3′, C7′ and C9′, is almost on the xanthine plane; therefore, the KW-3902 molecule (excluding propyl groups) has almost C_s symmetry.

Two KW-3902 molecules related by inversion form a dimer through an O6x—H···N7$(-x+1, -y, -z)$ hydrogen bond of 2.772 (4) Å. The dimers and their inversion images are alternately stacked along the [101] axis.

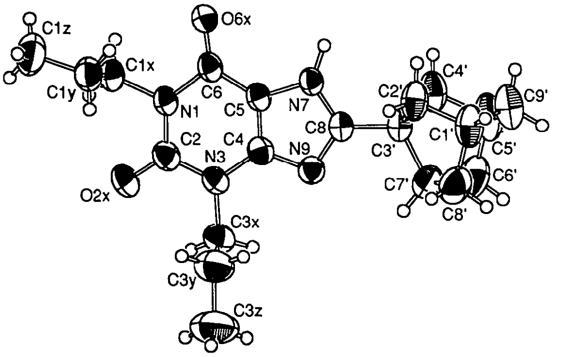


Fig. 1. *ORTEPII* (Johnson, 1976) drawing of KW-3902 with C, N and O atoms represented as 50% probability ellipsoids, and H atoms as spheres of arbitrary radii.

Experimental

Crystals of the title compound suitable for X-ray analysis were obtained by recrystallization from an ethanol solution. The density D_m was measured by flotation in aqueous $NaNO_3$.

Crystal data

$C_{20}H_{28}N_4O_2$
$M_r = 356.47$
Triclinic
$P\overline{1}$
$a = 9.557\,(1)$ Å
$b = 13.580\,(1)$ Å
$c = 8.290\,(1)$ Å
$\alpha = 90.819\,(7)°$
$\beta = 112.97\,(1)°$
$\gamma = 97.737\,(7)°$
$V = 978.9\,(4)$ Å^3
$Z = 2$
$D_x = 1.21$ Mg m^{-3}
$D_m = 1.20$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 25 reflections
$\theta = 35–55°$
$\mu = 0.603$ mm^{-1}
$T = 295$ K
Block
0.60 × 0.50 × 0.20 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4092 measured reflections
4060 independent reflections
3603 observed reflections [$I > 3\sigma(I)$]

$R_{\rm int} = 0.012$
$\theta_{\rm max} = 75°$
$h = -11 \rightarrow 11$
$k = 0 \rightarrow 16$
$l = -10 \rightarrow 10$
3 standard reflections
frequency: 83.3 min
intensity decay: 0.6%

Refinement

Refinement on F
$R = 0.0760$
$wR = 0.0815$
$S = 0.801$
3603 reflections
236 parameters
Unit weights applied
$(\Delta/\sigma)_{\rm max} = 0.005$
$\Delta\rho_{\rm max} = 0.28\,(6)$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.30\,(6)$ e Å^{-3}

Extinction correction: Zachariasen (1963)
Extinction coefficient: 7.83×10^{-6}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{\rm eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
N1	0.4945 (3)	0.2811 (2)	0.0634 (3)	3.93 (6)
C2	0.4213 (4)	0.3325 (3)	0.1468 (5)	4.49 (8)
N3	0.3305 (3)	0.2760 (2)	0.2163 (4)	4.35 (6)
C4	0.3205 (3)	0.1736 (2)	0.2062 (4)	3.67 (7)
C5	0.3928 (3)	0.1264 (2)	0.1230 (4)	3.47 (7)
C6	0.4869 (4)	0.1773 (2)	0.0444 (4)	3.77 (7)
N7	0.3556 (3)	0.0257 (2)	0.1401 (3)	3.56 (6)
C8	0.2654 (3)	0.0200 (2)	0.2318 (4)	3.51 (7)
N9	0.2400 (3)	0.1095 (2)	0.2752 (3)	3.92 (6)
C1′	0.2548 (5)	−0.2007 (3)	0.4866 (5)	6.0 (1)
C2′	0.3371 (4)	−0.1342 (3)	0.3948 (5)	5.27 (9)
C3′	0.2059 (4)	−0.0765 (3)	0.2817 (4)	3.84 (7)
C4′	0.1074 (5)	−0.1527 (3)	0.1224 (5)	5.7 (1)
C5′	0.0165 (5)	−0.2195 (4)	0.2064 (6)	7.1 (1)
C6′	−0.0431 (5)	−0.1387 (4)	0.2895 (7)	7.3 (1)
C7′	0.1064 (4)	−0.0685 (3)	0.3920 (5)	5.8 (1)
C8′	0.1862 (5)	−0.1206 (4)	0.5580 (5)	6.4 (1)
C9′	0.1250 (5)	−0.2734 (3)	0.3516 (6)	6.8 (1)
C1*x*	0.5994 (4)	0.3436 (3)	0.0004 (4)	4.50 (8)
C1*y*	0.7609 (4)	0.3688 (3)	0.1447 (5)	5.26 (9)
C1*z*	0.8634 (5)	0.4429 (4)	0.0880 (7)	7.4 (1)
O2*x*	0.4366 (3)	0.4230 (2)	0.1572 (4)	6.26 (7)
C3*x*	0.2477 (4)	0.3252 (3)	0.3019 (5)	5.31 (9)
C3*y*	0.3443 (5)	0.3483 (4)	0.4961 (6)	7.0 (1)
C3*z*	0.2575 (6)	0.4060 (5)	0.5785 (7)	10.3 (2)
O6*x*	0.5573 (3)	0.1410 (2)	−0.0304 (3)	4.95 (6)

Table 2. *Selected geometric parameters* (Å, °)

N1—C2	1.396 (5)	C8—N9	1.342 (5)
N1—C6	1.405 (4)	C8—C3′	1.486 (5)
N1—C1*x*	1.480 (5)	C1′—C2′	1.519 (7)
C2—N3	1.378 (5)	C1′—C8′	1.562 (7)
C2—O2*x*	1.217 (4)	C1′—C9′	1.523 (5)
N3—C4	1.379 (4)	C2′—C3′	1.560 (5)
N3—C3*x*	1.464 (6)	C3′—C4′	1.559 (5)
C4—C5	1.358 (5)	C3′—C7′	1.567 (6)
C4—N9	1.361 (5)	C4′—C5′	1.530 (7)
C5—C6	1.419 (5)	C5′—C6′	1.567 (8)
C5—N7	1.388 (4)	C5′—C9′	1.522 (6)
C6—O6*x*	1.220 (5)	C6′—C7′	1.529 (5)
N7—C8	1.351 (5)	C7′—C8′	1.526 (6)
C2—N1—C6	126.8 (3)	C4—C5—C6	123.3 (3)
C2—N1—C1*x*	115.8 (3)	C4—C5—N7	104.9 (3)
C6—N1—C1*x*	117.3 (3)	N1—C6—C5	111.7 (3)
N1—C2—N3	117.0 (3)	N1—C6—O6*x*	120.7 (3)
N1—C2—O2*x*	121.6 (4)	C5—C6—O6*x*	127.6 (3)
N3—C2—O2*x*	121.4 (4)	C5—N7—C8	106.3 (3)
C2—N3—C4	119.4 (3)	N7—C8—N9	112.9 (3)
C2—N3—C3*x*	119.7 (3)	N7—C8—C3′	122.3 (3)
C4—N3—C3*x*	120.9 (3)	N9—C8—C3′	124.8 (3)
N3—C4—C5	121.8 (3)	C4—N9—C8	103.0 (3)
C5—C4—N9	112.8 (3)		
N7—C8—C3′—C2′	58.3 (4)	N7—C8—C3′—C7′	178.5 (3)
N7—C8—C3′—C4′	−58.4 (4)	N9—C8—C3′—C7′	0.3 (5)

The atomic coordinates of H atoms were geometrically recalculated after each refinement cycle and included in the structure-factor calculations. The B_{iso} of each H atom was reset to 1.30 × $B_{eq}(X)$ after each refinement cycle, where $B_{eq}(X)$ is B_{eq} of the atom to which the H atom is connected.

Data collection: *CAD*-4 *SDP-Plus* (B. A. Frenz & Associates, Inc., 1985). Cell refinement: *CAD*-4 *SDP-Plus*. Data reduction: *CAD*-4 *SDP-Plus*. Structure solution: *MULTAN*11/82 (Main *et al.*, 1982). Structure refinement: *CAD*-4 *SDP-Plus*. Molecular graphics: *ORTEP*II (Johnson, 1976). Preparation of material for publication: *CAD*-4 *SDP-Plus*.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: AS1162). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). C**51**, 1957–1958

On the Structure of (13-Methyl-1,4,7,8,13,-13b-hexahydro[1′,2′]oxazepino[2′,3′:1,2]-pyrido[3,4-*b*]indol-1-yl)methanol

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Abstract

The crystal structure of $C_{17}H_{20}N_2O_2$, originally described in space group *Pc* [Ohishi, Sakamoto, Harusawa, Yoneda & Kurihara (1995). *Acta Cryst.* C**51**, 135–139], is better described and refined in *P*2₁/*c*. Revised coordinates and bond lengths are given. Whereas it was originally reported that the structure is based on four independent molecules in the asymmetric unit, three of one chirality and one of the opposite chirality, both the original space group (*Pc*) and the revised space group (*P*2₁/*c*) are enantiomorphic and the structure must contain an equal number of both configurations.

Comment

My attention – and, I suspect, that of many other readers – was first drawn to this article (Ohishi, Sakamoto, Harusawa, Yoneda & Kurihara, 1995; hereafter, OSHYK) by the authors' claim that 'four molecules exist as an enantiomeric mixture with a ratio of 3:1 in a single crystal' whereas the reported space group, *Pc*, is achiral and hence the structure must contain equal numbers of both enantiomers. Further examination suggested that the structure might be better described in space group $P2_1/c$, with but two molecules in the asymmetric unit; the coordinates reported by OSHYK indicate that corresponding atoms in the molecules *A* and *B*, and in molecules *C* and *D*, are related to one another by an approximate center of symmetry at (0.485, 0.250, 0.492). Another strong indication that the structure was described incorrectly as non-centrosymmetric lies in the reported bond lengths (OSHYK, Table 2); corresponding bonds in the four molecules differ by as much as 0.22 Å in the face of e.s.d.'s of 0.01 Å or less.

Refinement in $P2_1/c$ was based on the 4021 F_o values in the supplementary material (OH1042). Included were two reflections, 010 and 050, which should be extinguished in $P2_1/c$; both had F_o and F_c values among the smallest in the list. H atoms were first placed in assumed positions, and then included in the refinement. Convergence was reached quickly at R = 0.0629 for

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[†] Contribution No. 9067.