

and between bromide and phenol [N(2)···Br 3.252 (7), H(2)···Br 2.168 (2) Å, N(2)—H(2)···Br 172.7 (5)°, O(2')···Brⁱ 3.281 (6), H(2')···Brⁱ 2.187 (2) Å, O(2')—H(2')···Brⁱ 172.0 (3)°; (i) $x + 1, y, z$].

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Structure and Conformation of a Tightly Bound Inhibitor of Adenosine Transport, 2-Amino-6-[(4-nitrobenzyl)thio]-9-β-D-ribofuranosylpurine

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Abstract. C₁₇H₁₈N₆O₆S, $M_r = 434.4$, monoclinic, $P2_1$, $a = 14.577$ (4), $b = 8.820$ (2), $c = 7.304$ (2) Å, $\beta = 94.76$ (1)°, $V = 935.9$ Å³, $Z = 2$, $D_m = 1.55$, $D_x = 1.542$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 19.66$ cm⁻¹, $F(000) = 452$, $T = 287$ K, $R = 0.029$ ($wR = 0.038$) for 1995 observed reflections. The glycosidic torsion angle, $\chi[\text{C}(8)\text{—N}(9)\text{—C}(1')\text{—O}(4')]$, is -120.0 (4)°, which corresponds to the *syn* nucleoside conformation with an intramolecular O(5')—H(O5')···N(3) hydrogen bond; the ribose has the C(2')-*endo* (²*E*) pucker conformation with pseudorotational parameters: $P = 158.6$ (3)° and $\tau_m = 36.7$ (3)°; C(2') is displaced 0.567 Å from the plane of C(3'), C(4'), O(4') and C(1'). The conformation of the exocyclic C(4')—C(5') bond is g^+ .

Introduction. The passage of nucleoside molecules across the plasma membrane of animal cells is mediated by nucleoside-specific transport mechanisms of several types (for reviews, see Plagemann & Wohlueter, 1980;

Young & Jarvis, 1983; Paterson & Cass, 1985; Paterson, Jakobs, Ng, Odegard & Adjei, 1987), including (a) facilitative diffusion (equilibrative) systems of high or low sensitivity to 6-[(4-nitrobenzyl)thio]-9-β-D-ribofuranosylpurine (NBMPR) (Soriano-Garcia, Parthasarathy, Paul & Paterson, 1984; Paterson *et al.*, 1987), the potent nucleoside transport (NT) inhibitor, and (b) concentrative, 'secondary active' NT systems linked to fluxes of sodium ions (Paterson *et al.*, 1987; Jakobs & Paterson, 1986). Of these, the best characterized is the equilibrative NBMPR-sensitive NT system of human erythrocytes (Young & Jarvis, 1983; Jarvis, Hammond, Paterson & Clanachan, 1983; Plagemann & Wohlueter, 1984*a, b*). A component of that system, a membrane glycoprotein of M_r 45 000–66 000 (Young, Jarvis, Robins & Paterson, 1983), binds NBMPR at sites with high affinity [K_D 0.3 nM (Jarvis, Hammond, Paterson & Clanachan, 1982)] for that ligand. NBMPR-sensitive equilibrative NT systems

have been recognized in a variety of cell types, including cultured mouse lymphoma S49 cells (Paterson, Jakobs, Harley, Fu, Robins & Cass, 1983). As very potent NT inhibitors, NBMPR and its cogener, the title compound 2-amino-6-[(4-nitrobenzyl)thio]-9- β -D-ribofuranosylpurine (NBTGR), have been important tools in exploration of nucleoside transporter biology and function. For example, both agents have been valuable as flux stoppers in kinetic studies of NT (Paterson, Harley & Cass, 1985), and ^3H -NBMPR has enabled (a) enumeration of NT elements (of the NBMPR-sensitive type) in cells (Paterson *et al.*, 1985; Cass, Gaudette & Paterson, 1974), and (b) identification on gel electropherograms of the membrane glycoprotein on which the high affinity NT-inhibitory sites are located (Young *et al.*, 1983; Jarvis & Young, 1987). NBMPR and NBTGR are competitive ligands at the high affinity NT-inhibitory membrane sites in S49 cells; K_D values for dissociation of NBMPR and NBTGR from the ligand-site complex in these cells are 0.096 and 0.047 nM (Paterson *et al.*, 1983). The 4-nitrobenzyl substituent of these ligands contributes importantly to their tight binding at NT inhibitory sites and to that of related NT inhibitors (Paterson, Naik & Cass, 1977).

Experimental. The molecule NBTGR was synthesized and kindly supplied by Dr M. J. Robins of the University of Alberta. Well formed brick-shaped crystals were grown by vapor diffusion of diethyl ether into a solution of the compound in methanol. The crystals of NBTGR were clear yellow and have space group $P2_1$. The crystal used was $0.047 \times 0.225 \times 0.2375$ mm in size and exhibited the forms $\{100\}$, $\{010\}$ and $\{001\}$. Unit-cell dimensions from 25 reflections, $12.24 < \theta < 26.08^\circ$; systematic absences $0k0$, $k = 2n + 1$. A CAD-4F diffractometer was used to collect three-dimensional $\text{Cu K}\alpha$ (Ni-filtered) intensity data on a unique set of 2051 ($1995 > 2\sigma$) reflections [$(\sin\theta)/\lambda < 0.609 \text{ \AA}^{-1}$] by the ω - 2θ scan mode, with index range $h -18/+18$, $k 0/+11$, $l 0/+9$. Lorentz and polarization corrections were applied; no significant change occurred in the intensities of three standard reflections which were monitored throughout the data collection; an analytical absorption correction was applied; max./min. transmission factors 0.9107 and 0.6557. Merging R based on intensities 0.015 for 544 reflections measured more than once.

The data were processed and the structure was solved by direct methods using the *XTAL* system (Stewart & Hall, 1983). Four solutions with AMOS value of 75% were found by the programs. The solution with the best CFOM (0.64) was used and all 30 non-hydrogen atoms were found on the first E map. Least-squares isotropic refinement gave an R value of 0.106. Anisotropic refinement was then carried out and gave an R value of 0.057. The positions of the H atoms

were located by using difference Fourier maps. Finally, full-matrix least-squares refinement of positions and anisotropic temperature factors for the non-hydrogen atoms and positions and isotropic temperature factors for the hydrogens, minimizing the function $\sum w(|F_o| - |F_c|)^2$ where $w = 1/\sigma^2(F_o)$, gave at convergence $R = 0.029$, $wR = 0.038$ and $S = 2.386$. A final difference Fourier map revealed residuals between -0.23 and 0.223 e \AA^{-3} . The largest and average shift/e.s.d. ratios were 0.015 and 0.002, respectively. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). A stereo *ORTEP* (Johnson, 1976) representation of the molecule with atomic labelling is shown in Fig. 1. The atomic parameters are given in Table 1.* A VAX8600 computer at the University of Saskatchewan was used to carry out all crystallographic computations.

Discussion. Bond distances and angles of NBTGR are given in Table 2. The bond distances and angles in the 4-nitrophenyl and adenine rings of NBTGR are similar to those found for these groups in other structure determinations. The structure of NBTGR is isomorphous with the structure of the closely-related compound, NBMPR (Soriano-Garcia *et al.*, 1984). Both compounds crystallize in the same space group, with very similar molecular conformations. The glycosidic torsion angle, $\chi[\text{C}(8)-\text{N}(9)-\text{C}(1')-\text{O}(4')]$, is $-120.0(4)^\circ$, which corresponds to the *syn* nucleoside conformation with an intramolecular $\text{O}(5')-\text{H}(\text{O}5') \cdots \text{N}(3)$ hydrogen bond; the ribose has the $\text{C}(2')$ -*endo*

* Lists of anisotropic thermal parameters, fractional coordinates and isotropic thermal parameters for hydrogen atoms, and observed and calculated structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44998 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

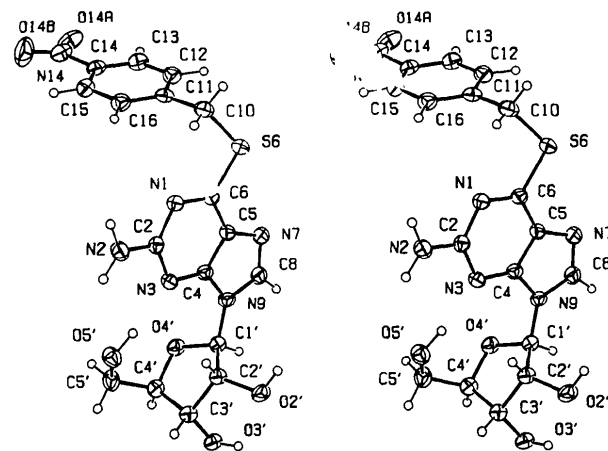


Fig. 1. Stereoscopic *ORTEP* view (Johnson, 1976) of NBTGR with atomic numbering.

Table 1. Fractional coordinates and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^3$)

$U_{eq} = (\text{trace of the orthogonalized } U_{ij} \text{ tensor})/3.$

	x	y	z	U_{eq}
S(6)	0.52571 (3)	0.23150	0.84623 (8)	49
N(1)	0.6385 (1)	0.2650 (3)	0.5785 (2)	37
N(2)	0.7371 (2)	0.2577 (3)	0.3492 (3)	48
N(3)	0.7778 (1)	0.4157 (3)	0.5946 (2)	36
N(7)	0.6759 (1)	0.4759 (3)	1.0178 (3)	45
N(9)	0.8025 (1)	0.5507 (3)	0.8851 (2)	37
C(2)	0.7171 (2)	0.3169 (3)	0.5119 (3)	35
C(4)	0.7554 (1)	0.4562 (3)	0.7604 (3)	34
C(5)	0.6777 (1)	0.4105 (3)	0.8452 (3)	37
C(6)	0.6199 (1)	0.3085 (3)	0.7444 (3)	37
C(8)	0.7505 (2)	0.5571 (4)	1.0339 (3)	44
C(1')	0.8889 (1)	0.6277 (3)	0.8680 (3)	34
C(2')	0.9688 (1)	0.5237 (3)	0.8314 (3)	35
C(3')	1.0319 (1)	0.6313 (3)	0.7380 (3)	36
C(4')	0.9641 (2)	0.7368 (3)	0.6287 (3)	39
C(5')	0.9452 (2)	0.6981 (3)	0.4266 (3)	49
O(2')	1.0147 (1)	0.4607 (3)	0.9894 (3)	52
O(3')	1.0854 (1)	0.7207 (2)	0.8712 (2)	40
O(4')	0.8785 (1)	0.7255 (2)	0.7149 (2)	40
O(5')	0.9138 (1)	0.5494 (3)	0.3922 (3)	51
C(10)	0.4670 (2)	0.1269 (3)	0.6574 (4)	46
C(11)	0.4115 (1)	0.2275 (3)	0.5239 (3)	39
C(12)	0.3418 (2)	0.3190 (3)	0.5828 (3)	46
C(13)	0.2882 (2)	0.4080 (3)	0.4602 (4)	45
C(14)	0.3053 (2)	0.4035 (3)	0.2763 (3)	43
C(15)	0.3738 (2)	0.3160 (4)	0.2149 (3)	47
C(16)	0.4272 (1)	0.2275 (4)	0.3397 (3)	45
N(14)	0.2469 (2)	0.4926 (3)	0.1437 (4)	55
O(14A)	0.1944 (2)	0.5846 (3)	0.2004 (4)	69
O(14B)	0.2532 (2)	0.4694 (5)	-0.0196 (3)	92

(2E) pucker conformation, with pseudorotational parameters: $P = 158.6 (3)^\circ$ and $\tau_m = 36.7 (3)^\circ$; C(2') is displaced 0.567 Å from the plane of C(3'), C(4'), O(4') and C(1'). The conformation of the exocyclic C(4')-C(5') bond is g^+ .

The phenyl ring is planar within 0.0056 Å. The atoms C(10) and N(14) are out of the plane of the phenyl group by 0.058 and 0.054 Å respectively, on the same side. The plane of the $-\text{NO}_2$ group is twisted relative to the plane of the phenyl group by 10.6° .

The adenine group is planar within 0.014 Å. The atoms S(6) and N(2) are out of the plane of the adenine group by 0.152 and 0.123 Å, respectively, on the same side. This may indicate that the hybridization of the attached atoms of the adenine group is not pure sp^2 .

The mean plane of the 4-nitrobenzyl group, an important substituent for potent nucleoside transport inhibition in a series of S^6 -substituted 6-thioinosine derivatives, is inclined at an angle of 102.4° to the plane of the adenine ring. The torsion angles around the methylene C atom of this benzyl group are C(6)-S(6)-C(10)-C(11) -78.0 and S(6)-C(10)-C(11)-C(12) -58.3° .

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Table 2. Bond lengths (Å) and bond angles ($^\circ$)

S(6)-C(6)	1.752 (2)	C(2')-C(3')	1.522 (3)
S(6)-C(10)	1.814 (3)	C(2')-O(2')	1.400 (3)
N(1)-C(2)	1.361 (3)	C(3')-C(4')	1.532 (3)
N(1)-C(6)	1.320 (3)	C(3')-O(3')	1.432 (3)
N(2)-C(2)	1.352 (3)	C(4')-C(5')	1.518 (3)
N(3)-C(2)	1.348 (3)	C(4')-O(4')	1.447 (3)
N(3)-C(4)	1.329 (3)	C(5')-O(5')	1.405 (4)
N(7)-C(5)	1.389 (3)	C(10)-C(11)	1.504 (4)
N(7)-C(8)	1.299 (4)	C(11)-C(12)	1.394 (4)
N(9)-C(4)	1.376 (3)	C(11)-C(16)	1.383 (3)
N(9)-C(8)	1.377 (3)	C(12)-C(13)	1.383 (4)
N(9)-C(1')	1.445 (3)	C(13)-C(14)	1.387 (4)
C(4)-C(5)	1.395 (3)	C(14)-C(15)	1.367 (4)
C(5)-C(6)	1.399 (3)	C(14)-N(14)	1.464 (4)
C(1')-C(2')	1.524 (3)	C(15)-C(16)	1.389 (4)
C(1')-O(4')	1.411 (3)	N(14)-O(14A)	1.211 (4)
		N(14)-O(14B)	1.221 (4)
C(6)-S(6)-C(10)	102.2 (1)	C(1')-C(2')-O(2')	114.5 (2)
C(2)-N(1)-C(6)	118.2 (3)	C(3')-C(2')-O(2')	110.6 (2)
C(2)-N(3)-C(4)	112.3 (2)	C(2')-C(3')-C(4')	103.0 (2)
C(5)-N(7)-C(8)	103.4 (2)	C(2')-C(3')-O(3')	110.7 (2)
C(4)-N(9)-C(8)	105.7 (2)	C(4')-C(3')-O(3')	108.1 (3)
C(4)-N(9)-C(1')	128.2 (2)	C(3')-C(4')-C(5')	115.3 (3)
C(8)-N(9)-C(1')	126.1 (3)	C(3')-C(4')-O(4')	105.9 (2)
N(1)-C(2)-N(2)	115.7 (3)	C(5')-C(4')-O(4')	108.3 (2)
N(1)-C(2)-N(3)	126.7 (2)	C(4')-C(5')-O(5')	114.5 (3)
N(2)-C(2)-N(3)	117.5 (2)	C(1')-O(4')-C(4')	110.3 (2)
N(3)-C(4)-N(9)	128.3 (2)	S(6)-C(10)-C(11)	112.8 (3)
N(3)-C(4)-C(5)	126.7 (3)	C(10)-C(11)-C(12)	120.6 (2)
N(9)-C(4)-C(5)	105.1 (2)	C(10)-C(11)-C(16)	120.5 (3)
N(7)-C(5)-C(4)	111.1 (3)	C(12)-C(11)-C(16)	118.9 (3)
N(7)-C(5)-C(6)	133.6 (2)	C(11)-C(12)-C(13)	121.1 (2)
C(4)-C(5)-C(6)	115.3 (2)	C(12)-C(13)-C(14)	118.3 (3)
S(6)-C(6)-N(1)	120.3 (3)	C(13)-C(14)-C(15)	121.9 (3)
S(6)-C(6)-C(5)	119.0 (2)	C(13)-C(14)-N(14)	119.0 (3)
N(1)-C(6)-C(5)	120.6 (2)	C(15)-C(14)-N(14)	119.1 (2)
N(7)-C(8)-N(9)	114.7 (3)	C(14)-C(15)-C(16)	119.1 (2)
N(9)-C(1')-C(2')	114.6 (3)	C(11)-C(16)-C(15)	120.6 (3)
N(9)-C(1')-O(4')	108.6 (2)	C(14)-N(14)-O(14A)	118.9 (3)
C(2')-C(1')-O(4')	105.2 (2)	C(14)-N(14)-O(14B)	118.0 (4)
C(1')-C(2')-C(3')	101.7 (3)	O(14A)-N(14)-O(14B)	123.2 (4)

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Structure of a Novel C₂₂H₂₄ Cage Dimer

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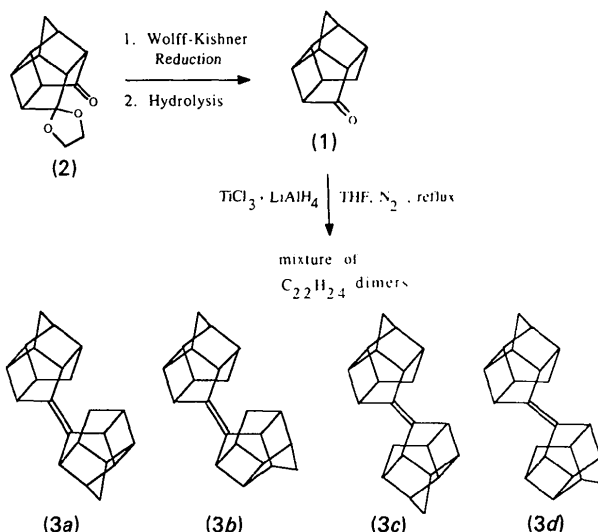
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Abstract. 8,11'-Bipentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]-undecanylidene, C₂₂H₂₄, *M_r* = 288.43, triclinic, *P* $\bar{1}$, *a* = 6.613 (2), *b* = 10.809 (3), *c* = 10.883 (2) Å, α = 97.52 (2), β = 99.85 (1), γ = 99.32 (2)°, *V* = 746.2 (2) Å³, *Z* = 2, *D_x* = 1.284 Mg m⁻³, $\lambda(\text{Mo } K\alpha)$ = 0.71073 Å, μ = 0.07 mm⁻¹, *F*(000) = 312, *T* = 295 K, final *R* = 0.048, *wR* = 0.048 for 1118 observed reflections. The molecule, which has an unusually high density for a hydrocarbon, consists of two cage moieties which are related by an approximate twofold axis along the C(11)–C(11') double bond. There are no intermolecular approaches less than van der Waals separations; the high density is probably due to compression of the cage C atoms ensuing from the small bond angles. For example, the internal ring angles at C(11) and C(11'), both *sp*² C atoms, are only 102.6 (2) and 102.2 (2)°, respectively.

Introduction. As part of a program that is involved in the synthesis and chemistry of energetic polycyclic 'cage' compounds (Marchand, 1987), the titanium-induced dicarbonyl-coupling reaction of pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecan-8-one (1) has been studied. Compound (1) was synthesized *via* Wolff–Kishner reduction of the pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane-8,11-dione monoethylene ketal (2) (Eaton, Cassar, Hudson & Hwang, 1976) followed by hydrolysis of the resulting product. Reaction of (1) with 'McMurry's reagent' (*i.e.* TiCl₃–LiAlH₄) (McMurry, 1983) potentially could result in the formation of four dimers [*i.e.* (3a)–(3d)], each of which possesses a twofold symmetry element. The product that was

obtained by refluxing a mixture of (1) and TiCl₃–LiAlH₄ in tetrahydrofuran under nitrogen for 72 h was purified *via* column chromatography (silica-gel stationary phase, hexane eluent). Careful fractional recrystallization of the purified product from hexane afforded a single isomer, m.p. 473–474 K. The results of a single-crystal X-ray structural analysis on the isomer thereby obtained revealed that this material possesses structure (3a) (see below).



Experimental. Clear colorless crystal, 0.18 × 0.35 × 0.42 mm, automated Nicolet R3m diffractometer with incident-beam graphite monochromator, 25 centered