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Nearly Centrosymmetric (*S*)-7-(2,6-Dichlorobenzyl)-8-(3-oxocyclopentyl)-1,3-dipropyl-7*H*-purine-2,6-dione

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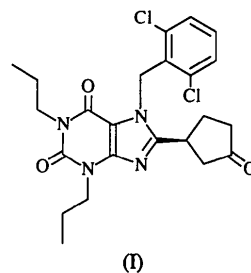
(Received 19 December 1995; accepted 20 February 1996)

Abstract

The structure of the title compound, $C_{23}H_{26}Cl_2N_4O_3$, with two molecules in the asymmetric unit, is essentially centrosymmetric apart from the cyclopentanone rings. It was possible, nevertheless, to determine the absolute configuration unambiguously.

Comment

The title compound, (I), is the slightly higher affine (*S*)-(-) enantiomer of KFM 19 (Schingnitz, Küfner-Mühl, Ensinger, Lehr & Kuhn, 1991). It belongs to a group of selective adenosine A_1 -antagonists with therapeutic potential for the treatment of dementia and related cognitive deficiencies. The structure analysis was undertaken in order to determine the absolute configuration of the stereogenic centre.



The compound turned out to be the *S* stereoisomer (Fig. 1). Apart from the cyclopentanone rings, the two molecules in the asymmetric unit show a nearly perfect centrosymmetric arrangement (Fig. 2). The cyclopentan-

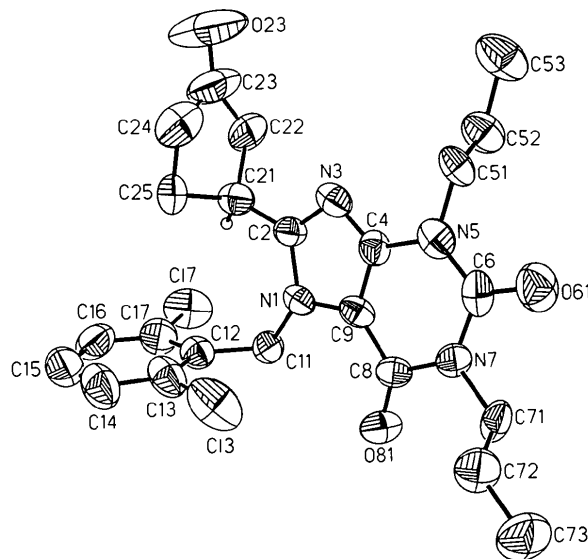


Fig. 1. View of one molecule of the title compound showing the atomic labelling. Only the tertiary H atom is shown and ellipsoids are plotted at the 50% probability level.

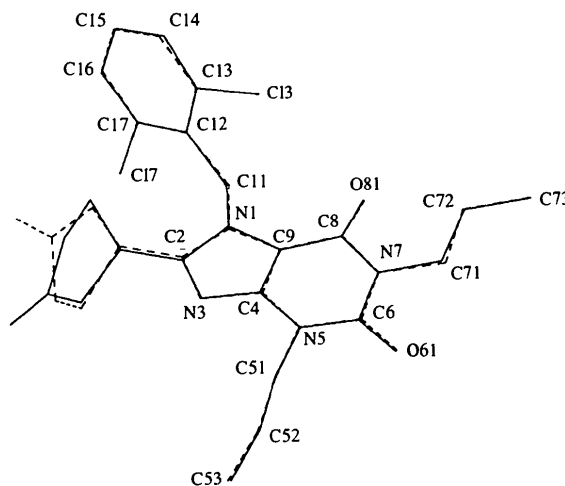


Fig. 2. A least-squares fit of the two molecules in the asymmetric unit with one molecule inverted. Fitted atoms are labelled.

one ring of the first molecule has an envelope conformation, with atom C25 deviating by 0.54 (1) Å from the plane composed of the remaining four ring atoms, while the cyclopentanone ring of the second molecule has a twist conformation, with atom C24A 0.19 (3) Å above and atom C25A 0.21 (3) Å below the plane of the remaining three atoms. The appearance of quasi-racemate crystal structures has been found previously (for example, Noguchi, Sankawa & Itaka, 1978; Giordano & Lanzetta, 1989; Kim *et al.*, 1989; Nozaki, 1979; Moon *et al.*, 1992).

Experimental

The title compound was recrystallized from ethanol/diethyl ether solution.

Crystal data

C₂₃H₂₆Cl₂N₄O₃

$M_r = 477.38$

Triclinic

*P*1

$a = 8.8140 (10) \text{ \AA}$

$b = 10.329 (2) \text{ \AA}$

$c = 13.248 (5) \text{ \AA}$

$\alpha = 74.19 (3)^\circ$

$\beta = 88.31 (2)^\circ$

$\gamma = 85.65 (2)^\circ$

$V = 1157.1 (5) \text{ \AA}^3$

$Z = 2$

$D_x = 1.370 \text{ Mg m}^{-3}$

D_m not measured

Cu $K\alpha$ radiation

$\lambda = 1.54180 \text{ \AA}$

Cell parameters from 25

reflections

$\theta = 30\text{--}35^\circ$

$\mu = 2.795 \text{ mm}^{-1}$

$T = 293 (2) \text{ K}$

Block

$0.60 \times 0.40 \times 0.20 \text{ mm}$

Colourless

Data collection

Enraf–Nonius CAD-4 four-circle diffractometer

ω scans

Absorption correction:

empirical *via* ψ scans

(North, Phillips & Mathews, 1968)

$T_{\min} = 0.45$, $T_{\max} = 0.57$

5057 measured reflections

5057 independent reflections

4930 observed reflections

$[I > 2\sigma(I)]$

$\theta_{\max} = 59.95^\circ$

$h = -9 \rightarrow 9$

$k = -11 \rightarrow 4$

$l = -14 \rightarrow 14$

3 standard reflections

frequency: 90 min

intensity decay: 2.5%

Refinement

Refinement on F^2

$R(F) = 0.0627$

$wR(F^2) = 0.2054$

$S = 1.128$

5055 reflections

577 parameters

H atoms riding

$w = 1/[\sigma^2(F_o^2) + (0.1391P)^2 + 0.4212P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.062$

$\Delta\rho_{\max} = 0.351 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.391 \text{ e \AA}^{-3}$

Extinction correction: none

Atomic scattering factors

from *International Tables for Crystallography* (1992), Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Absolute configuration:

Flack (1983) parameter = 0.06 (2)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	x	y	z	U_{eq}
N1	0.3121 (5)	0.1047 (5)	0.2686 (3)	0.0437 (10)
C11	0.4286 (6)	0.0847 (7)	0.1906 (4)	0.0496 (13)
C12	0.3635 (6)	0.0799 (7)	0.0876 (4)	0.0498 (13)
C13	0.2708 (6)	-0.0209 (9)	0.0815 (5)	0.068 (2)
C13	0.2311 (3)	-0.1457 (2)	0.1924 (2)	0.0991 (7)
C14	0.2171 (8)	-0.0337 (10)	-0.0112 (7)	0.081 (2)
C15	0.2470 (10)	0.0622 (12)	-0.1020 (8)	0.095 (3)
C16	0.3405 (9)	0.1696 (9)	-0.1005 (6)	0.076 (2)
C17	0.3959 (7)	0.1736 (8)	-0.0062 (5)	0.065 (2)
C17	0.5095 (2)	0.3048 (2)	-0.0098 (2)	0.0877 (6)
C2	0.2117 (6)	0.2170 (6)	0.2613 (4)	0.0493 (13)
C21	0.1767 (7)	0.3212 (7)	0.1631 (5)	0.059 (2)
C22	0.1200 (9)	0.4539 (8)	0.1758 (6)	0.077 (2)
C23	-0.0504 (11)	0.4673 (10)	0.1619 (8)	0.102 (3)
O23	-0.1348 (10)	0.5605 (9)	0.1598 (10)	0.190 (4)
C24	-0.0944 (10)	0.3501 (10)	0.1376 (7)	0.093 (2)
C25	0.0473 (7)	0.2838 (8)	0.0976 (5)	0.069 (2)
N3	0.1370 (5)	0.2093 (6)	0.3523 (4)	0.0521 (12)
C4	0.1995 (6)	0.1020 (7)	0.4175 (4)	0.0451 (13)
N5	0.1643 (5)	0.0563 (6)	0.5266 (4)	0.0538 (12)
C51	0.0554 (7)	0.1408 (7)	0.5702 (4)	0.0541 (15)
C52	0.1281 (8)	0.2636 (8)	0.5849 (6)	0.070 (2)
C53	0.0147 (11)	0.3551 (8)	0.6260 (7)	0.093 (2)
C6	0.2427 (7)	-0.0501 (7)	0.5863 (4)	0.053 (2)
O61	0.2188 (6)	-0.0895 (6)	0.6831 (4)	0.0783 (14)
N7	0.3465 (5)	-0.1253 (5)	0.5377 (3)	0.0476 (11)
C71	0.4252 (7)	-0.2462 (7)	0.6004 (5)	0.0560 (15)
C72	0.3644 (9)	-0.3748 (7)	0.5955 (6)	0.072 (2)
C73	0.4424 (13)	-0.4987 (10)	0.6611 (8)	0.102 (3)
C8	0.3939 (6)	-0.0862 (6)	0.4300 (5)	0.0454 (12)
O81	0.4914 (5)	-0.1529 (4)	0.3968 (3)	0.0593 (11)
C9	0.3079 (6)	0.0316 (6)	0.3744 (4)	0.0446 (12)
N1A	-0.3148 (5)	-0.1084 (5)	-0.2708 (3)	0.0498 (11)
C11A	-0.4246 (6)	-0.0792 (7)	-0.1909 (5)	0.0547 (15)
C12A	-0.3559 (6)	-0.0799 (7)	-0.0881 (4)	0.0502 (13)
C13A	-0.2680 (7)	0.0199 (7)	-0.0799 (5)	0.059 (2)
C13A	-0.2291 (2)	0.1461 (2)	-0.1941 (2)	0.0923 (7)
C14A	-0.2067 (8)	0.0254 (10)	0.0165 (7)	0.084 (3)
C15A	-0.2412 (10)	-0.0665 (11)	0.1040 (5)	0.088 (3)
C16A	-0.3264 (10)	-0.1643 (10)	0.1030 (5)	0.078 (2)
C17A	-0.3854 (7)	-0.1733 (7)	0.0086 (4)	0.0556 (15)
C17A	-0.4993 (2)	-0.3044 (2)	0.01269 (15)	0.0887 (6)
C2A	-0.2068 (6)	-0.2111 (6)	-0.2599 (4)	0.0521 (14)
C21A	-0.1723 (7)	-0.3176 (7)	-0.1594 (4)	0.061 (2)
C22A	-0.0342 (14)	-0.2852 (11)	-0.1145 (8)	0.133 (5)
C23A	0.0904 (12)	-0.3750 (17)	-0.1197 (7)	0.129 (5)
O23A	0.2099 (8)	-0.3915 (15)	-0.0785 (7)	0.219 (6)
C24A	0.0457 (11)	-0.4610 (12)	-0.1819 (9)	0.113 (4)
C25A	-0.1231 (10)	-0.4613 (8)	-0.1822 (6)	0.081 (2)
N3A	-0.1412 (5)	-0.2121 (5)	-0.3516 (4)	0.0535 (12)
C4A	-0.2063 (6)	-0.0970 (6)	-0.4229 (4)	0.0443 (12)
N5A	-0.1744 (5)	-0.0590 (5)	-0.5250 (3)	0.0461 (10)
C51A	-0.0661 (6)	-0.1426 (8)	-0.5744 (6)	0.061 (2)
C52A	-0.1363 (8)	-0.2691 (10)	-0.5851 (7)	0.085 (3)
C53A	-0.0240 (12)	-0.3603 (13)	-0.6252 (11)	0.152 (6)
C6A	-0.2530 (6)	0.0561 (6)	-0.5902 (4)	0.0506 (14)
O61A	-0.2349 (5)	0.0915 (5)	-0.6828 (3)	0.0632 (11)
N7A	-0.3584 (5)	0.1242 (6)	-0.5364 (4)	0.0547 (12)
C71A	-0.4402 (8)	0.2499 (7)	-0.6055 (5)	0.062 (2)
C72A	-0.3655 (9)	0.3736 (8)	-0.5949 (6)	0.071 (2)
C73A	-0.4641 (15)	0.5014 (9)	-0.6604 (8)	0.104 (3)
C8A	-0.3964 (6)	0.0874 (7)	-0.4292 (5)	0.0521 (14)
O81A	-0.4981 (5)	0.1521 (5)	-0.3953 (4)	0.0709 (13)
C9A	-0.3124 (6)	-0.0329 (6)	-0.3721 (4)	0.0446 (12)

Table 2. Selected geometric parameters (\AA , $^\circ$)

N1—C2	1.388 (7)	N1A—C2A	1.351 (8)
N1—C9	1.401 (7)	N1A—C9A	1.357 (7)
C2—N3	1.343 (7)	C2A—N3A	1.332 (8)
N3—C4	1.300 (8)	N3A—C4A	1.396 (8)

C4—C9	1.362 (8)	C4A—N5A	1.330 (7)
C4—N5	1.425 (7)	C4A—C9A	1.371 (8)
N5—C6	1.324 (8)	N5A—C6A	1.411 (8)
C6—O61	1.252 (7)	C6A—O61A	1.191 (7)
C6—N7	1.409 (8)	C6A—N7A	1.415 (8)
N7—C8	1.432 (7)	N7A—C8A	1.403 (8)
C8—O81	1.209 (7)	C8A—O81A	1.225 (7)
C8—C9	1.415 (8)	C8A—C9A	1.430 (9)

C25—C21—C22—C23	22.1 (8)
C21—C22—C23—O23	−174.9 (12)
C21—C22—C23—C24	−2.2 (9)
O23—C23—C24—C25	153.4 (11)
C22—C23—C24—C25	−19.8 (9)
C23—C24—C25—C21	32.3 (8)
C22—C21—C25—C24	−32.5 (7)
C25A—C21A—C22A—C23A	7.7 (11)
C21A—C22A—C23A—O23A	−167.4 (13)
C21A—C22A—C23A—C24A	7.8 (13)
O23A—C23A—C24A—C25A	153.9 (11)
C22A—C23A—C24A—C25A	−21.8 (13)
C23A—C24A—C25A—C21A	25.0 (10)
C22A—C21A—C25A—C24A	−19.2 (8)

H atoms were refined using a riding model, with isotropic displacement parameters $U(\text{H}) = 1.5U_{\text{eq}}(\text{methyl C})$ and $U(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, and aromatic C—H = 0.93, tertiary C—H = 0.98, methylene C—H = 0.97 and methyl C—H = 0.96 Å.

Data collection: *SDP* (Enraf–Nonius, 1985). Cell refinement: *SDP*. Data reduction: *SDP*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991).

The author thanks Dr H. Koeppen (Boehringer Ingelheim) for providing the sample.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: CF1077). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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An Arylsulfonyloxazolidine

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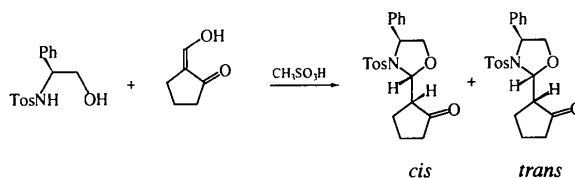
Abstract

The geometric parameters of the title compound, (2*S*)-2-[(2*R*,4*S*)-4-phenyl-3-tosyl-1,3-oxazolidin-2-yl]cyclopentanone, $\text{C}_{21}\text{H}_{23}\text{NO}_4\text{S}$, are in good agreement with other arylsulfonyl-oxazolidines. While the oxazolidine ring displays an envelope conformation, the cyclopentanone ring has a twist conformation.

Comment

The acid-catalysed condensation of 2-(*N*-tosyl)-1-alkanols with aldehydes usually produces the thermodynamically more stable 2,4-*cis*-substituted 1,3-oxazolidines (Hoppe, Hoffmann, Gärtner, Krettek & Hoppe, 1991).

From the reaction of (*S*)-*N*-tosyl-phenylglycinol and 2-(hydroxymethylene)cyclopentanone, we isolated traces of a side product which was subjected to X-ray structure analysis and turned out to have the *trans*-configuration (Fig. 1).



The oxazolidine ring in the *trans* configuration adopts an envelope conformation [$q_2 = 0.375(2)\text{Å}$, $\varphi_2 = 328.6(3)^\circ$ (Cremer & Pople, 1975)] with C5 deviating by 0.570(4) Å from the plane of the remaining four atoms, the cyclopentanone ring exhibits a twist configuration [$q_2 = 0.349(3)\text{Å}$, $\varphi_2 = 126.3(4)^\circ$] with C24 and C25 deviating by $-0.284(6)$ and $0.288(6)$ Å, respectively, from the plane of the remaining three atoms. The geometric parameters of the sulfonamide moiety agree well with those found by Herbst-Irmer (1990). The nitrogen N3 is nearly planar (sum of the bond angles: 356.5°). While one of the sulfonyl O atoms, O32, is nearly coplanar with the tolyl substituent [O32—S3—C31—C32 $30.0(2)^\circ$], the other, O31, forms a torsion angle of $-178.6(1)^\circ$ with C2. The two aromatic substituents enclose an angle of $19.8(1)^\circ$.