

Table 4. *Puckering and asymmetry parameters of the phenyl moiety*

Puckering parameters*			
q_2 (Å)	0.041 (4)	Q_T (Å)	0.041 (4)
q_3 (Å)	-0.001 (4)	θ_2 (°)	90.9 (6.0)
Asymmetry parameters*			
$\Delta C_1(C1)$	0.015 (2)	$\Delta C_2(C1)$	0.015 (1)
$\Delta C_1(C2)$	0.015 (2)	$\Delta C_2(C2)$	0.015 (1)
$\Delta C_1(C3)$	0.029 (2)	$\Delta C_2(C3)$	0.000 (2)
$\Delta C_1(C1-C2)$	0.024 (2)	$\Delta C_2(C1-C2)$	0.000 (2)
$\Delta C_1(C1-C6)$	0.012 (2)	$\Delta C_2(C1-C6)$	0.021 (2)
$\Delta C_1(C2-C3)$	0.012 (2)	$\Delta C_2(C2-C3)$	0.021 (2)

* Puckering and asymmetry parameters were calculated by use of the general program *PARST* (Nardelli, 1983).

O=S=O angle (see Fig. 2) and the analysis of the planarity reported in Table 3.

The phenyl C atoms deviate significantly from coplanarity: the conformational analysis reported in Table 4 indicates that the benzene ring adopts a deformation very close to the twist-boat conformation (Cremer & Pople, 1975). A quantitative evaluation of how the phenyl ring deviates from ideal symmetry can be derived from the asymmetry parameters (Duax, Weeks & Rohrer, 1976) reported in Table 4. They show that a high degree of symmetry is due to the presence of two twofold rotational axes, one through the C(3) atom and the other intersecting the C(1)—C(2) bond.

Molecular packing is determined by hydrogen bonds of the type O—H...O involving the hydroxylic O(4) atom and the sulfonic O(42) atom in the

equivalent position $1-x, 1-y, 2-z$ [O(4)...O(42) 2.706(3), H(40)...O(42) 1.74 (8) Å, O(4)—H(40)...O(42) 165 (8)°]. Other contacts are consistent with van der Waals interactions.

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Structure of the Antiviral Drug Ethyl 4-{2-[1-(6-Methyl-3-pyridazinyl)-4-piperidinyl]ethoxy}benzoate*

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Abstract. $C_{21}H_{27}N_3O_3$, $M_r = 369.5$, triclinic, $P\bar{1}$, $a = 6.801$ (7), $b = 11.08$ (1), $c = 26.82$ (4) Å, $\alpha = 79.2$ (1), $\beta = 86.5$ (1), $\gamma = 82.52$ (9)°, $V = 1967$ (4) Å³, $Z = 4$, $D_m = 1.25$, $D_x = 1.247$ Mg m⁻³, graphite-monochromated Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu =$

0.079 mm⁻¹, $F(000) = 792$, $T = 293$ K, final $R = 0.079$ for 3724 unique reflections with $|F_o| \geq 4|\sigma F_o|$. The asymmetric unit contains two molecules, *A* and *B*. They show pseudo-symmetry at $(\frac{1}{4}, \frac{1}{4}, \frac{1}{2})$, which is only broken by a 180° rotation of the ethyl-ethoxy moiety connected to the benzoate ring. The crystal structure is stabilized by hydrogen bonds between an N atom of the pyridazinyl ring of molecule *A* and an

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H atom of the pyridazinyl ring of molecule *B*. The chains are directed along the diagonal **a-b**.

Introduction. The title compound (Andries, Stokbroekx, Dewindt, Snoeks, Willebrords & Janssen, 1989) is a capsid-binding antiviral agent, approximately 1000 times more active than its predecessor 3-methoxy-6-[4-(3-methylphenyl)-1-piperazinyl]pyridazine (Andries, Dewindt, De Brabander, Stokbroekx & Janssen, 1988) which was the first antiviral agent shown effective in the prevention of the common cold in double-blind, placebo-controlled clinical trials.

Experimental. Colourless crystals obtained by slow evaporation from an acetone-toluene (10:1) solution. Size of the sample used for collecting intensity data, 0.90 × 0.36 × 0.08 mm. Density measured by flotation in *n*-heptane/CCl₄. All reflections were only indexable on the basis of a triclinic cell and no systematically absent reflections were observed. Stoe STADI-4 diffractometer, cell constants by least-squares refinement of the setting angles of 32 reflections with 15 < 2θ < 25°, ω/2θ scan, [(sin θ)/λ]_{max} = 0.8071 Å⁻¹, -11 ≤ h ≤ 11, -18 ≤ k ≤ 18, -43 ≤ l ≤ 43. Intensities of three standard reflections (31̄4, 246, 1̄15) were monitored every 180 min and showed only small statistical fluctuations. No linear scaling was applied. 34 576 reflections measured, 6534 were considered observed with |F_o| ≥ 4|σF_o| (3729 unique, R_{int} = 0.020). Data reduction with program REDU4 (Stoe & Co., 1985), Lorentz and polarization corrections, absorption corrections by the method of North, Phillips & Mathews (1968) based on four reflections (1̄32, 15̄1, 1̄53, 264). Min. and max. transmission factors 0.94 and 1.00. Scattering factors for the non-H atoms from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B) and, for H atoms from Stewart, Davidson & Simpson (1965). Since MULTAN11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) in *SDP-Plus* (Frenz, 1985) failed to solve the structure, MULTAN80 (Main *et al.*, 1980) with weighted tangent refinement (not available in *SDP-Plus*) was used. The *E* map calculated from the solution with the best figure of merit showed 46 of the 54 non-H atoms. The remaining O8A, O9A, C10A, C11A, O8B, O9B, C10B and C11B were obtained from a subsequent Fourier synthesis. Refinement on *F* by full-matrix least squares, first with isotropic temperature factors and finally anisotropically. All but the six H atoms attached at C11A and C11B, were found in a difference synthesis and they were included in the refinement with a fixed isotropic temperature factor of B = 4.0 Å². Five reflections (106, 105̄, 115̄, 014, 012) badly affected by extinction were excluded from the refinement. Final

Table 1. Atomic coordinates and equivalent isotropic displacement parameters (Å² × 10⁴) with e.s.d.'s in parentheses

$$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}a_i\cdot a_j$$

	x	y	z	B _{eq}
C1A	-0.9012 (6)	-0.5505 (4)	0.1131 (2)	4.29 (9)
C2A	-0.7895 (7)	-0.4589 (4)	0.1187 (2)	4.9 (1)
C3A	-0.6351 (6)	-0.4809 (4)	0.1515 (2)	4.5 (1)
C4A	-0.5858 (6)	-0.5977 (4)	0.1786 (1)	3.93 (9)
C5A	-0.6987 (7)	-0.6901 (4)	0.1732 (2)	5.4 (1)
C6A	-0.8527 (6)	-0.6681 (5)	0.1414 (2)	4.8 (1)
C7A	-1.0698 (7)	-0.5194 (4)	0.0782 (2)	5.4 (1)
O8A	-1.1212 (6)	-0.4175 (3)	0.0562 (2)	9.6 (1)
O9A	-1.1524 (4)	-0.6175 (3)	0.0736 (1)	6.35 (8)
C10A	-1.3212 (9)	-0.6004 (6)	0.0391 (3)	11.2 (2)
C11A	-1.369 (1)	-0.7100 (5)	0.0291 (3)	11.9 (2)
O12A	-0.4346 (4)	-0.6320 (2)	0.2114 (1)	5.23 (7)
C13A	-0.3191 (6)	-0.5413 (4)	0.2203 (2)	4.4 (1)
C14A	-0.1733 (6)	-0.6029 (4)	0.2602 (2)	4.4 (1)
C15A	-0.0497 (5)	-0.5168 (3)	0.2774 (2)	3.59 (9)
C16A	-0.1659 (5)	-0.4333 (3)	0.3113 (2)	4.10 (9)
C17A	-0.0404 (6)	-0.3518 (4)	0.3305 (2)	4.4 (1)
N18A	0.1334 (4)	-0.4225 (3)	0.3568 (1)	3.62 (7)
C19A	0.2518 (6)	-0.5038 (4)	0.3265 (2)	4.4 (1)
C20A	0.1261 (6)	-0.5881 (4)	0.3079 (2)	4.4 (1)
C21A	0.2315 (5)	-0.3616 (3)	0.3862 (1)	3.49 (8)
N22A	0.1497 (5)	-0.2488 (3)	0.3919 (1)	4.26 (8)
N23A	0.2368 (5)	-0.1848 (3)	0.4209 (1)	4.79 (9)
C24A	0.4064 (6)	-0.2307 (4)	0.4425 (2)	4.5 (1)
C25A	0.4935 (6)	-0.3490 (4)	0.4381 (2)	4.6 (1)
C26A	0.4078 (6)	-0.4152 (4)	0.4107 (2)	4.3 (1)
C27A	0.4941 (7)	-0.1485 (4)	0.4708 (2)	6.1 (1)
C1B	1.3993 (6)	0.0811 (4)	0.8737 (2)	4.3 (1)
C2B	1.3391 (6)	-0.0175 (4)	0.8564 (2)	4.6 (1)
C3B	1.1700 (6)	-0.0019 (4)	0.8289 (2)	4.3 (1)
C4B	1.0581 (6)	0.1122 (3)	0.8189 (2)	3.86 (9)
C5B	1.1201 (7)	0.2092 (4)	0.8370 (2)	5.3 (1)
C6B	1.2881 (7)	0.1946 (4)	0.8638 (2)	5.1 (1)
C7B	1.5827 (7)	0.0684 (4)	0.9031 (2)	5.5 (1)
O8B	1.6457 (5)	0.1539 (3)	0.9160 (1)	7.31 (9)
O9B	1.6699 (5)	-0.0475 (3)	0.9128 (1)	6.78 (9)
C10B	1.8505 (8)	-0.0686 (5)	0.9422 (2)	7.7 (2)
C11B	1.7879 (9)	-0.0981 (6)	0.9976 (2)	9.4 (2)
O12B	0.8918 (4)	0.1391 (2)	0.7915 (1)	4.82 (7)
C13B	0.8155 (6)	0.0446 (4)	0.7718 (2)	4.02 (9)
C14B	0.6469 (6)	0.1005 (4)	0.7396 (2)	4.3 (1)
C15B	0.5376 (5)	0.0117 (3)	0.7197 (1)	3.47 (8)
C16B	0.6624 (5)	-0.0598 (3)	0.6825 (2)	3.63 (9)
C17B	0.5450 (5)	-0.1451 (3)	0.6621 (2)	3.75 (9)
N18B	0.3638 (4)	-0.0784 (3)	0.6379 (1)	3.46 (7)
C19B	0.2394 (5)	-0.0064 (4)	0.6712 (2)	4.08 (9)
C20B	0.3548 (6)	0.0795 (3)	0.6916 (2)	3.83 (9)
C21B	0.2656 (5)	-0.1422 (3)	0.6095 (1)	3.54 (8)
N22B	0.3491 (5)	-0.2563 (3)	0.6043 (1)	4.07 (8)
N23B	0.2623 (5)	-0.3226 (3)	0.5762 (1)	4.58 (8)
C24B	0.0921 (6)	-0.2774 (3)	0.5545 (2)	4.1 (1)
C25B	0.0063 (6)	-0.1582 (4)	0.5580 (2)	4.9 (1)
C26B	0.0876 (6)	-0.0882 (4)	0.5851 (2)	4.3 (1)
C27B	0.0044 (7)	-0.3579 (4)	0.5267 (2)	6.0 (1)

R = 0.079, wR = 0.090, with $w = 4F^2/[\sigma^2(I) + 0.07F^2]$, S = 1.71. The relatively high R value is due to partially disordered ethyl group atoms. Largest parameter shift/e.s.d = 0.39. Min. and max. residual electron density -0.30 and 0.33 e Å⁻³. The number of reflections per refined variable was 3724/631 = 5.9. All calculations, except MULTAN80, were performed on a Digital PDP-11/73 microcomputer and a MicroVAX 2000 microcomputer using *SDP-Plus* (Frenz, 1985) and *PARST* (Nardelli, 1983). MULTAN80 calculations were performed on an IBM 3090.

Discussion. Final atomic coordinates and equivalent isotropic displacement parameters are listed in Table

Table 2. Bond lengths (Å), bond angles (°) and selected torsion angles (°) for molecules *A* and *B*, with *e.s.d.*'s in parentheses

			<i>A</i>	<i>B</i>	
C1	C2		1.379 (6)	1.382 (7)	
C1	C6		1.390 (5)	1.369 (5)	
C1	C7		1.490 (6)	1.494 (6)	
C2	C3		1.379 (6)	1.377 (6)	
C3	C4		1.370 (5)	1.377 (5)	
C4	C5		1.390 (6)	1.382 (6)	
C4	O12		1.365 (5)	1.357 (5)	
C5	C6		1.360 (7)	1.362 (7)	
C7	O8		1.191 (5)	1.203 (6)	
C7	O9		1.314 (6)	1.329 (5)	
O9	C10		1.490 (7)	1.471 (6)	
C10	C11		1.37 (1)	1.506 (8)	
O12	C13		1.416 (5)	1.425 (5)	
C13	C14		1.509 (6)	1.476 (6)	
C14	C15		1.501 (6)	1.497 (6)	
C15	C16		1.531 (6)	1.537 (5)	
C15	C20		1.534 (5)	1.529 (5)	
C16	C17		1.497 (6)	1.508 (6)	
C17	N18		1.471 (5)	1.472 (4)	
N18	C19		1.460 (5)	1.467 (5)	
N18	C21		1.380 (5)	1.383 (5)	
C19	C20		1.512 (6)	1.505 (6)	
C21	N22		1.333 (5)	1.345 (5)	
C21	C26		1.409 (5)	1.418 (5)	
N22	N23		1.352 (5)	1.354 (5)	
N23	C24		1.321 (5)	1.324 (5)	
C24	C25		1.390 (6)	1.390 (6)	
C24	C27		1.489 (7)	1.466 (7)	
C25	C26		1.336 (7)	1.346 (7)	
C2	C1	C6	117.9 (4)	119.4 (4)	
C2	C1	C7	119.3 (3)	122.2 (4)	
C6	C1	C7	122.8 (4)	118.5 (4)	
C1	C2	C3	122.0 (4)	120.5 (4)	
C2	C3	C4	119.7 (4)	120.2 (4)	
C3	C4	C5	118.5 (4)	118.3 (4)	
C3	C4	O12	125.4 (4)	125.4 (4)	
C5	C4	O12	116.1 (3)	116.3 (3)	
C4	C5	C6	121.9 (4)	121.8 (4)	
C1	C6	C5	120.0 (4)	119.9 (4)	
C1	C7	O8	123.6 (5)	123.8 (4)	
C1	C7	O9	112.4 (3)	112.4 (4)	
O8	C7	O9	124.0 (4)	123.8 (4)	
C7	O9	C10	118.2 (4)	116.0 (4)	
O9	C10	C11	113.1 (5)	107.5 (4)	
C4	O12	C13	119.1 (3)	120.2 (3)	
O12	C13	C14	107.6 (3)	109.2 (3)	
C13	C14	C15	114.5 (3)	115.9 (3)	
C14	C15	C16	113.3 (3)	113.8 (3)	
C14	C15	C20	111.5 (3)	110.8 (3)	
C16	C15	C20	106.7 (3)	106.8 (3)	
C15	C16	C17	113.3 (3)	112.2 (3)	
C16	C17	N18	112.3 (3)	111.9 (3)	
C17	N18	C19	113.0 (3)	112.7 (3)	
C17	N18	C21	116.7 (3)	116.5 (3)	
C19	N18	C21	118.2 (3)	116.4 (3)	
N18	C19	C20	111.4 (3)	111.8 (3)	
C15	C20	C19	112.8 (3)	112.6 (3)	
N18	C21	N22	117.1 (3)	117.4 (3)	
N18	C21	C26	122.7 (3)	121.6 (3)	
N22	C21	C26	120.2 (4)	121.0 (4)	
C21	N22	N23	120.1 (3)	120.3 (3)	
N22	N23	C24	121.0 (3)	120.6 (3)	
N23	C24	C25	120.3 (4)	120.1 (4)	
N23	C24	C27	116.1 (4)	116.8 (3)	
C25	C24	C27	123.6 (4)	123.0 (4)	
C24	C25	C26	119.7 (4)	121.5 (4)	
C24	C25	C25	118.6 (4)	116.4 (4)	
C5	C4	O12	C13	-177.4 (0.4)	-179.3 (0.4)
C7	O9	C10	C11	167.3 (0.5)	90.2 (0.5)
O12	C13	C14	C15	-175.1 (0.3)	-174.5 (0.3)
C14	C15	C16	C17	177.2 (0.3)	-178.1 (0.3)
C14	C15	C20	C19	-179.2 (0.3)	-179.7 (0.3)
C15	C16	C17	N18	-54.4 (0.4)	55.8 (0.4)
C17	N18	C21	N22	6.1 (0.5)	-3.3 (0.5)
C27	C24	C25	C26	-178.0 (0.4)	177.9 (0.4)

1.* Main bond lengths, bond angles and selected torsion angles are given in Table 2. A *PLUTO* (Motherwell & Clegg, 1978) plot of molecule *B*, with the adopted numbering scheme, is shown in Fig. 1. A stereoscopic view of the molecular packing is given in Fig. 2.

The asymmetric unit contains two molecules, *A* and *B*. These molecules show pseudosymmetry at ($\frac{1}{4}, \frac{1}{4}, \frac{1}{2}$), which is only broken by a 180° rotation of the ethyl-ethoxy moiety connected to the benzoate ring. Given the torsional flexibility of the ethyl group, atoms C10*A*, C10*B*, C11*A*, C11*B* show increasingly higher B_{eq} values (Table 1).

The pyridazinyl rings and aromatic benzoate rings are planar [max. out-of-plane deviations of 0.02 (1) Å for C24*A* and C24*B*]. Atoms C13*A*, C13*B*, O12*A* and O12*B* are coplanar with the aromatic benzoate ring [max. out-of-plane deviation 0.04 (1) Å]. The planes defined by atoms C7*A*—O8*A*—O9*A* and C7*B*—O8*B*—O9*B* make a dihedral angle with the aromatic benzoate ring of 4.9 (7)° and of 4.5 (6)° for molecules *A* and *B*, respectively. For the piperidiny ring we calculated, following the method of Cremer & Pople (1975), a phase angle $\theta_2 = 176 (1)^\circ$ and $\varphi_2 = 21 (19)^\circ$, indicating a chair conformation and a puckering amplitude $Q = 0.547 (11)$

* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles involving H atoms, H-atom parameters and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53792 (34 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

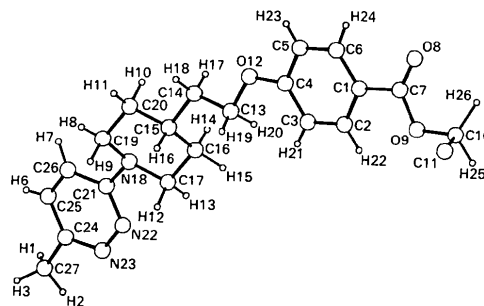


Fig. 1. A *PLUTO* (Motherwell & Clegg, 1978) plot of molecule *B* showing the numbering scheme.

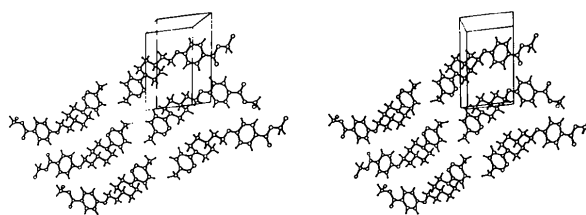


Fig. 2. A stereoscopic view of the molecular packing; *a* is horizontal, *b* is vertical and *c* is in the plane of the paper.

for the sequence N18A—C17A—C16A—C15A—C20A—C19A. For the sequence N18B—C17B—C16B—C15B—C20B—C19B a phase angle $\theta_2 = 4.2(14)^\circ$, $\varphi = 179(22)^\circ$ and $Q = 0.556(14)$ are obtained.

Intermolecular hydrogen bonds (Taylor & Kennard, 1982) are formed between the pyridazinyl hydrogen H7A of molecule A and the pyridazinyl nitrogen N23B of molecule B: $H7A \cdots N23B^i = 2.58(3) \text{ \AA}$. Hydrogen bonds are also found between the pyridazinyl hydrogen H7B of molecule B and the pyridazinyl nitrogen N23A of molecule A: $H7B \cdots N23A^{ii} = 2.62(3) \text{ \AA}$ [symmetry codes: (i) $-x, -y-1, -z+1$; (ii) $-x-1, -y, -z+1$]. No other contacts shorter than the sums of the van der Waals radii are observed.

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Structure of β -D-Lyxopyrano[1,2-d]oxazolidin-2-one

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Abstract. C₆H₉NO₅, $M_r = 175.14$, monoclinic, $P2_1$, $a = 5.526(1)$, $b = 9.543(1)$, $c = 13.769(2) \text{ \AA}$, $\beta = 93.74(1)^\circ$, $V = 724.6(2) \text{ \AA}^3$, $Z = 4$, $D_x = 1.60 \text{ g cm}^{-3}$, $\lambda(\text{Cu } K\alpha_1) = 1.54051 \text{ \AA}$, $\mu = 11.8 \text{ cm}^{-1}$, $F(000) = 368$, $T = 293 \text{ K}$, $R = 0.031$ for 1575 observed reflexions. There are two independent

molecules of the title compound in the asymmetric unit. The puckering parameters show that the pyranoid ring is significantly flattened at C(11) and C(21), respectively, in both molecules.

Introduction. Treatment of aldoses with potassium cyanate in the presence of sodium dihydrogenphosphate or ammonium chloride gives 1,2-*cis*-cyclic car-

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