All the H atoms were experimentally positioned and their positional parameters refined. The displacement parameters of the acidic H atoms were refined isotropically. Unit weights kept $\Sigma w(\Delta F)^2$ uniform over ranges of $\sin\theta/\lambda$ and $|F_o|$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CELDIM* in *CAD-4 Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *MULTAN*80 (Main *et al.*, 1980). Program(s) used to refine structure: *MolEN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: SK1047). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Absolute Configuration of the Double Salt of *cis*-4-Amino-5-chloro-*N*-{1-[3-(4-fluorophenoxy)propyl]-3-methoxypiperidin-1-ium-4-yl}-2-methoxybenzamide Tartrate (Cisapride Tartrate)†

OSWALD M. PEETERS, NORBERT M. BLATON AND CAMIEL J. DE RANTER

Laboratorium voor Analytische Chemie en Medicinale Fysicochemie, Faculteit Farmaceutische Wetenschappen, Katholieke Universiteit Leuven, Van Evenstraat 4, B-3000 Leuven, Belgium. E-mail: maurice.peeters@farm.kuleuven.ac.be

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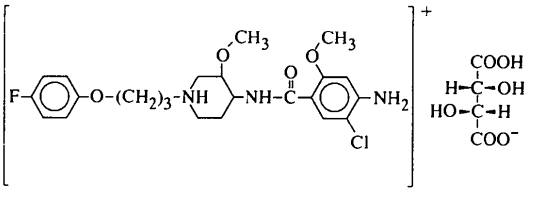
Abstract

The structure determination of cisapride (+)-tartrate, $C_{23}H_{30}ClFN_3O_4^+.C_4H_5O_6^-$, from X-ray diffraction data of crystals obtained from an ethanol solution, showed that the diastereomers [(3*R*,4*S*)(2*R*,3*R*)] and [(3*S*,4*R*)-(2*R*,3*R*)] crystallized as a double salt in a 1:1 ratio.

Comment

The crystal structure of the gastrokinetic drug cisapride (R51619) was determined by Collin *et al.* (1989). It is a 3,4-*cis* racemate, wherein the piperidine ring adopts a chair conformation with the nitrogen substituent and the benzamide function in equatorial positions and the methoxy group in an axial position. To separate cisapride into its enantiomeric forms, it is treated with (+)-tartaric acid and crystallized from ethanol, (I). As the diastereomers could not be resolved, the formation of a double salt in the crystal was supposed.



(I)

To check this hypothesis, the crystal structure and absolute configuration of R53929 has been determined. The asymmetric unit contains the two diastereomers [(3*R*,4*S*)(2*R*,3*R*)] and [(3*S*,4*R*)(2*R*,3*R*)]. The corresponding bond lengths, angles and absolute values of the torsion angles do not differ significantly between the two

† Internal code of the Janssen Research Foundation: R53929.

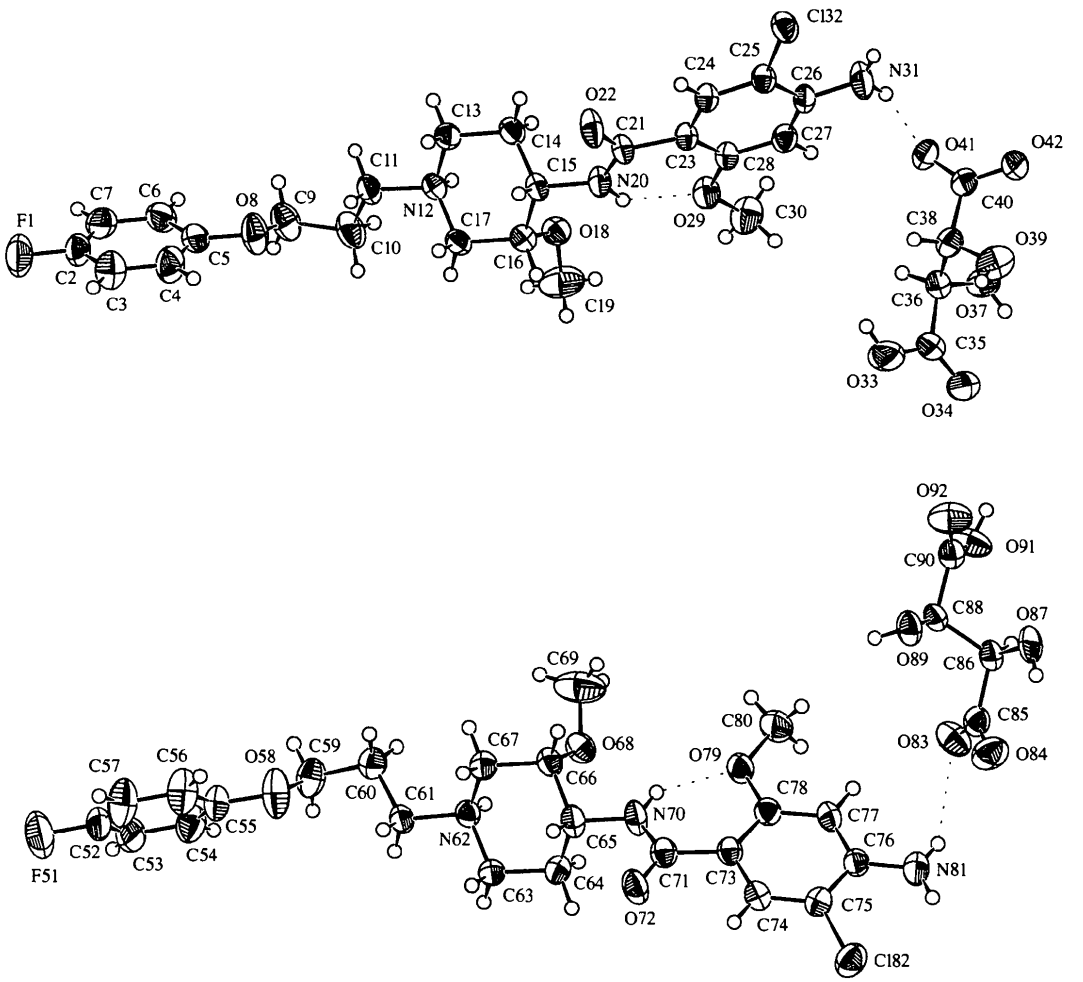


Fig. 1. Perspective views of the molecules of the title compound with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

enantiomers and the structure of cisapride itself (Collin *et al.*, 1989). Both molecules show an intramolecular hydrogen bond between the amidic N atom and the *ortho*-methoxy substituent (Table 2). The C-atom chains of the hydrogen tartrate ions are in an extended conformation, with the hydroxyl O atoms in the *–sc* position with respect to one another. The packing of the molecules is stabilized by an extensive network of hydrogen bonds which are summarized in Table 2.

Experimental

Crystals of the title compound were obtained by slow evaporation at room temperature from an ethanol solution.

Crystal data

$C_{23}H_{30}ClFN_3O_4^+.C_4H_5O_6^-$
M_r = 616.03
Cu $K\alpha$ radiation
λ = 1.54184 Å
Monoclinic
$P2_1$
a = 8.7641 (4) Å
b = 26.785 (2) Å
c = 12.5316 (7) Å
β = 104.412 (4)°
V = 2849.2 (3) Å^3
Z = 4
D_x = 1.436 Mg m^{-3}
D_m = 1.428 Mg m^{-3}
D_m measured by flotation in *n*-heptane/CCl_4
Cell parameters from 38 reflections
θ = 10.92–27.73°
μ = 1.790 mm^{-1}
T = 293 K
Prism
0.40 × 0.12 × 0.08 mm
Colourless

Data collection

Siemens *P*4 four-circle diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical (*XEMP*; Siemens, 1989)
T_{min} = 0.620, T_{max} = 0.867
6793 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.0276
θ_{max} = 57.22°
h = −9 → 9
k = −29 → 29
l = −13 → 13

8566 measured reflections
7766 independent reflections

3 standard reflections
every 100 reflections
intensity decay: <2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0415$
$wR(F^2) = 0.1092$
$S = 1.032$
7766 reflections
768 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o^2) + (0.0564P)^2 + 0.9086P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.007$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0014 (1)
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)
Flack parameter for absolute configuration determination = −0.003 (15)

Table 1. *Selected torsion angles (°)*

C6—C5—O8—C9	−17.8 (6)	C55—O58—C59—C60	173.8 (4)
C5—O8—C9—C10	−170.2 (4)	O58—C59—C60—C61	67.9 (4)
O8—C9—C10—C11	−60.1 (5)	C59—C60—C61—N62	165.1 (3)
C9—C10—C11—N12	−169.2 (3)	C64—C65—N70—C71	−84.8 (4)
C14—C15—N20—C21	74.7 (4)	C64—C65—C66—O68	−64.4 (4)
N20—C15—C16—O18	−53.8 (4)	N70—C65—C66—O68	59.2 (4)
N20—C15—C16—C17	−177.2 (3)	N70—C65—C66—C67	−179.5 (3)
C15—C16—O18—C19	163.0 (3)	C65—C66—O68—C69	−159.7 (4)
N20—C21—C23—C28	−1.7 (6)	N70—C71—C73—C78	8.0 (6)
C23—C28—O29—C30	−178.3 (3)	C73—C78—O79—C80	178.2 (3)
C35—C36—C38—O39	52.1 (4)	C85—C86—C88—O89	68.4 (4)
C35—C36—C38—C40	173.3 (3)	C85—C86—C88—C90	−170.0 (3)
O37—C36—C38—O39	−71.3 (4)	O87—C86—C88—O89	−55.7 (4)
C54—C55—O58—C59	28.7 (6)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	H···*A*	*D*···*A*	*D*—H···*A*
N20—H20···O29	1.98	2.647 (4)	134
N31—H31*A*···O87	2.21	3.041 (5)	162
N31—H31*B*···O41	2.37	3.195 (5)	160
N70—H70···O79	1.96	2.646 (4)	136
N81—H81*B*···O42	2.29	3.109 (5)	158
O91—H91···O42	1.81	2.624 (4)	171
O33—H33···O72^{i}	1.89	2.628 (5)	149
N12—H12···O83^{ii}	1.82	2.726 (5)	172
N81—H81*A*···O83^{iii}	2.66	3.435 (5)	151
N81—H81*A*···O84^{iii}	2.50	3.106 (5)	128
O37—H37···O84^{iii}	2.08	2.846 (4)	157
N62—H62···O41^{iv}	1.87	2.749 (4)	163
O89—H89···O22^{v}	1.95	2.757 (4)	166

Symmetry codes: (i) $-x, \frac{1}{2}+y, -z$; (ii) $1-x, \frac{1}{2}+y, 1-z$; (iii) $x, y, z-1$; (iv) $1-x, y-\frac{1}{2}, -z$; (v) $-x, y-\frac{1}{2}, 1-z$.

The data were collected using a variable scan speed with a scan range of 0.4° below $K\alpha_1$ to 0.4° above $K\alpha_2$. The ratio of total background time to scan time was 0.5. The structure was solved by direct methods and refined by anisotropic full-matrix least squares on F^2. All H atoms were found from difference Fourier syntheses but placed at geometrical positions and allowed to ride on their parent atom. The absolute configuration was determined according to Flack (1983).

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *DIAMOND* (Bergerhoff, 1996). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: NA1275). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Catemeric Hydrogen Bonding in a γ-Keto Acid: (±)-2a,3,4,5-Tetrahydro-1(2*H*)-oxo-acenaphthylene-2a-carboxylic Acid

ANDREW P. J. BRUNSKILL, HUGH W. THOMPSON AND ROGER A. LALANCETTE*

Carl A. Olson Memorial Laboratories, Department of Chemistry, Rutgers University, Newark, NJ 07102, USA. E-mail: lalancette@hades.rutgers.edu

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Abstract

The title keto-carboxylic acid, $C_{13}H_{12}O_3$, forms spiral carboxyl-to-ketone hydrogen-bonding catemers, whose helical axes are parallel to the *b* cell axis. Two single-strand hydrogen-bonding chains pass through the cell, each composed of molecules of a single chirality.

Comment

Keto-carboxylic acids offer opportunities for variation on the standard pattern of dimeric hydrogen bonding observed in functionally unelaborated acids. In most cases,