data reports

Crystal structure of [(2S)-1-[(3S)-3-carboxy-6,7-dimethoxy-1,2,3,4-tetra-hydroisoquinolin-2-yl]-1-oxopropan-2-yl][(2S)-1-ethoxy-1-oxo-4-phenylbutan-2-yl]azanium chloride acetonitrile mono-solvate

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The title compound (trivial name moexipril hydrochloride) crystallizes as the acetonitrile monosolvate, $C_{27}H_{35}N_2O_7{}^+$·Cl^-·C_2H_3N, with the salt comprising a U-shaped cation and a chloride anion. The conformation of the cation is stabilized by a weak intramolecular N^+—H···O hydrogen bond and the tetrahydropyridine ring adopts a *boat* conformation. The dihedral angle between the planes of the benzene rings is 85.6 (1)°. In the crystal, the cations and anions form tight ionic pairs by strong intermolecular O—H···Cl hydrogen bonds. C—H···Cl and C—H···N hydrogen bonds link these ionic pairs and the acetonitrile solvate molecules into puckered layers parallel to (100).

1. Related literature

For the synthesis of the title compound, see: Klutchko *et al.* (1986); Yamazaki & Suzuki (1998). For the structure and applications of related compounds, see: Suzuki *et al.* (2000, 2010).
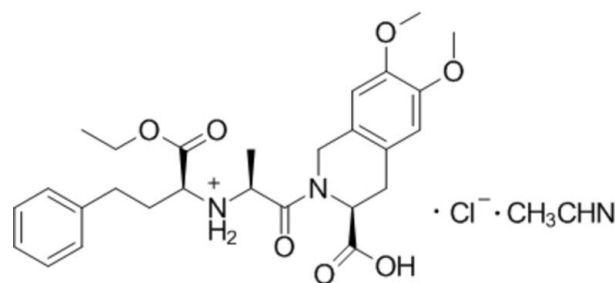
2. Experimental

2.1. Crystal data

$C_{27}H_{35}N_2O_7{}^+$·Cl^-·C_2H_3N
$M_r = 576.07$
Monoclinic, $P2_1$
$a = 10.9391$ (6) Å
$b = 10.4655$ (4) Å
$c = 13.3159$ (5) Å
$\beta = 97.419$ (4)°

$V = 1511.68$ (12) Å^3
$Z = 2$
Cu $K\alpha$ radiation
$\mu = 1.52$ mm^{-1}
$T = 103$ K
$0.50 \times 0.20 \times 0.20$ mm

2.2. Data collection

Agilent Xcalibur, Eos, Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
$T_{min} = 0.516$, $T_{max} = 0.750$

11078 measured reflections
5728 independent reflections
5570 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.087$
$S = 1.03$
5728 reflections
367 parameters
1 restraint
H-atom parameters constrained

$\Delta\rho_{max} = 0.55$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}
Absolute structure: Flack (1983), 2620 Friedel pairs
Absolute structure parameter: 0.000 (10)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5···Cl1[i]	0.84	2.09	2.924 (3)	171
N1—H1A···O3	0.92	2.04	2.587 (2)	117
N1—H1B···O4[ii]	0.92	1.87	2.724 (3)	154
C13—H13···Cl1	1.00	2.52	3.459 (2)	157
C14—H14B···Cl1[iii]	0.98	2.71	3.593 (2)	150
C18—H18A···N3[iv]	0.99	2.39	3.367 (4)	168
C29—H29A···Cl1[iv]	0.98	2.74	3.698 (2)	165

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

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Acknowledgements

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supporting information

Crystal structure of [(2*S*)-1-[(3*S*)-3-carboxy-6,7-dimethoxy-1,2,3,4-tetrahydro-isoquinolin-2-yl]-1-oxopropan-2-yl][(2*S*)-1-ethoxy-1-oxo-4-phenylbutan-2-yl]azanium chloride acetonitrile monosolvate

Ai-zhen Li and Wen-jie Xu

S1. Comment

Moexipril is a nonsulfhydryl containing precursor of the active angiotensin-converting enzyme. It is well known that the polymorphic and *pseudo*-polymorphic crystals of moexipril show different physico-chemical properties (Klutchko *et al.*, 1986; Suzuki *et al.*, 2010). The crystal structure of the *β*-form, the monohydrate and the sesquihydrate have been already reported previously (Yamazaki & Suzuki, 1998; Suzuki *et al.*, 2000). In this paper, we report the *X*-ray crystal structure and stereochemistry of moexipril hydrochloride acetonitrile monosolvate.

S2. Experimental

A solution of 2-[2-[(1-Ethoxycarbonyl-3-phenylpropyl)amino]propanoyl]-6,7-dimethoxy–3,4-dihydro-1*H*-isoquinoline-3-carboxylic acid in absolute ethanol was treated with hydrochloride in the presence of 5% Pd/C. The resulting mixture was concentrated and stood for 3 h at reduced pressure. The residue obtained was recrystallized from acetonitrile at room temperature to give colourless crystals of **I** suitable for X-ray diffraction analysis.

S3. Refinement

The absolute structure of **I** was objectively determined by the refinement of Flack parameter (2620 (94%) Friedel pairs measured), which has become equal to 0.00 (1). The calculated Hooft parameter is equal to -0.003 (6).

  The hydroxyl and amino hydrogen atoms were objectively localized in the difference-Fourier map and include into refinement with fixed positional and isotropic displacement parameters [U_{iso}(H) = 1.5U_{eq}(O) and U_{iso}(H) = 1.2U_{eq}(N)]. The other hydrogen atoms were placed in the calculated positions with C—H distances = 0.95 (aryl-H), 0.98 (methyl-H), 0.99 (methylene-H) and 1.00 (methine-H) Å and refined in the riding model with fixed isotropic displacement parameters [U_{iso}(H) = 1.5U_{eq}(C) for the methyl groups and 1.2U_{eq}(C) for the other groups].
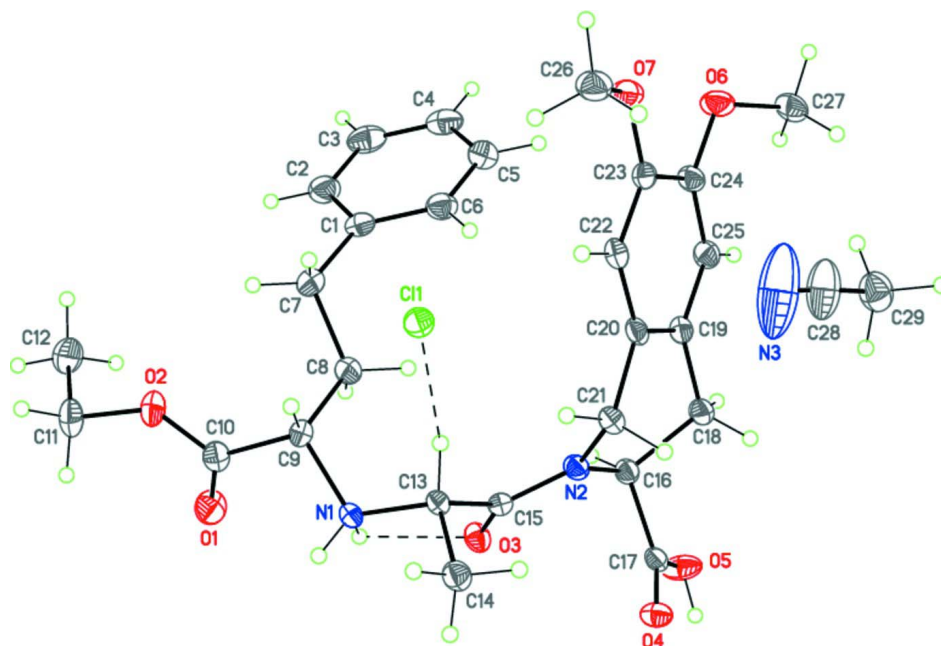
Figure 1

Molecular structure of the title compound. Displacement ellipsoids are presented at the 50% probability level. H atoms are depicted as small spheres of arbitrary radius. Dashed lines indicate the intramolecular N—H···O and the intermolecular C—H···Cl hydrogen bonds.
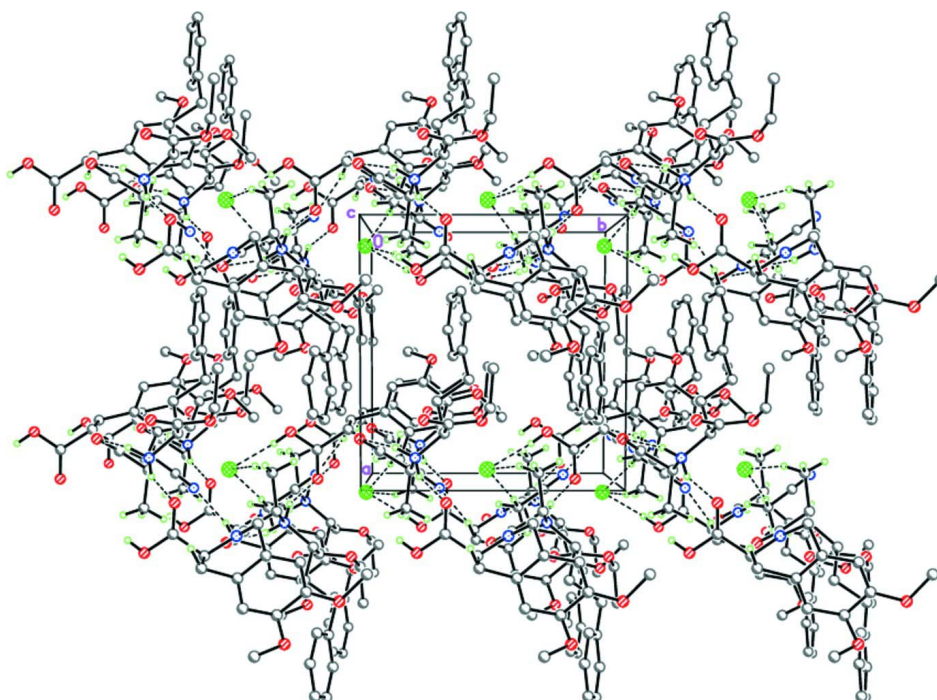
Figure 2

Crystal packing showing the puckered layers parallel to (100). Dashed lines indicate the intra- and intermolecular hydrogen bonds.

[(2S)-1-[(3S)-3-carboxy-6,7-dimethoxy-1,2,3,4-tetrahydroisoquinolin-2-yl]-1-oxopropan-2-yl][(2S)-1-ethoxy-1-oxo-4-phenylbutan-2-yl]azanium chloride acetonitrile monosolvate

Crystal data

$C_{27}H_{35}N_2O_7^+\cdot Cl^-\cdot C_2H_3N$	$F(000) = 612$
$M_r = 576.07$	$D_x = 1.266$ Mg m^{-3}
Monoclinic, $P2_1$	Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Hall symbol: P 2yb	Cell parameters from 6099 reflections
$a = 10.9391$ (6) Å	$\theta = 3.3$–71.7°
$b = 10.4655$ (4) Å	$\mu = 1.52$ mm^{-1}
$c = 13.3159$ (5) Å	$T = 103$ K
$\beta = 97.419$ (4)°	Block, colourless
$V = 1511.68$ (12) Å^3	$0.50 \times 0.20 \times 0.20$ mm
$Z = 2$	

Data collection

Agilent Xcalibur, Eos, Gemini diffractometer	11078 measured reflections
Radiation source: fine-focus sealed tube	5728 independent reflections
Graphite monochromator	5570 reflections with $I > 2\sigma(I)$
Detector resolution: 16.0971 pixels mm^{-1}	$R_{int} = 0.021$
ω scans	$\theta_{max} = 71.8°$, $\theta_{min} = 3.4°$
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)	$h = -13 \rightarrow 12$
	$k = -12 \rightarrow 12$
$T_{min} = 0.516$, $T_{max} = 0.750$	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.033$	Hydrogen site location: difference Fourier map
$wR(F^2) = 0.087$	H-atom parameters constrained
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2 + 0.3525P]$
5728 reflections	where $P = (F_o^2 + 2F_c^2)/3$
367 parameters	$(\Delta/\sigma)_{max} = 0.001$
1 restraint	$\Delta\rho_{max} = 0.55$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{min} = -0.34$ e Å^{-3}
	Absolute structure: Flack (1983), 2620 Friedel pairs
	Absolute structure parameter: 0.000 (10)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
Cl1	0.93335 (4)	0.48179 (4)	0.82049 (3)	0.02184 (10)

O6	0.52273 (13)	0.26128 (14)	0.40381 (11)	0.0274 (3)
O5	0.79427 (13)	−0.28384 (13)	0.76776 (12)	0.0286 (3)
H5	0.8374	−0.3472	0.7887	0.043*
O3	0.81278 (12)	−0.00568 (13)	0.94514 (9)	0.0204 (3)
O4	0.96943 (12)	−0.17651 (13)	0.81353 (10)	0.0214 (3)
O2	0.73833 (13)	0.45383 (13)	1.15813 (10)	0.0240 (3)
C22	0.77885 (16)	0.30103 (17)	0.60435 (13)	0.0174 (3)
H22	0.8293	0.3680	0.6349	0.021*
O1	0.73521 (14)	0.24231 (14)	1.19090 (11)	0.0285 (3)
O7	0.65710 (12)	0.44801 (12)	0.48979 (10)	0.0229 (3)
N2	0.85280 (14)	0.05279 (14)	0.78967 (11)	0.0173 (3)
C25	0.62888 (17)	0.10402 (18)	0.51823 (14)	0.0204 (4)
H25	0.5763	0.0373	0.4902	0.024*
C17	0.86058 (16)	−0.17996 (17)	0.78125 (12)	0.0170 (3)
C23	0.68475 (16)	0.32815 (18)	0.52690 (13)	0.0185 (3)
C15	0.86015 (16)	0.06883 (17)	0.88977 (13)	0.0168 (3)
C26	0.7471 (2)	0.54471 (19)	0.51712 (15)	0.0278 (4)
H26A	0.7196	0.6252	0.4840	0.042*
H26B	0.8257	0.5190	0.4954	0.042*
H26C	0.7578	0.5564	0.5908	0.042*
N1	0.87847 (13)	0.20668 (14)	1.03592 (10)	0.0154 (3)
H1A	0.8400	0.1330	1.0528	0.018*
H1B	0.9429	0.2227	1.0859	0.018*
C20	0.79909 (16)	0.17574 (18)	0.63698 (12)	0.0172 (3)
C6	0.50803 (18)	0.34869 (19)	0.75184 (15)	0.0242 (4)
H6	0.5901	0.3387	0.7369	0.029*
C8	0.67413 (17)	0.28867 (19)	0.95447 (14)	0.0209 (4)
H8A	0.7007	0.2557	0.8911	0.025*
H8B	0.6235	0.2219	0.9818	0.025*
C24	0.61083 (17)	0.22814 (19)	0.48234 (14)	0.0202 (4)
C13	0.92855 (16)	0.18674 (17)	0.93702 (12)	0.0160 (3)
H13	0.9074	0.2624	0.8923	0.019*
C21	0.90294 (16)	0.13908 (17)	0.71794 (13)	0.0174 (3)
H21A	0.9693	0.0957	0.6870	0.021*
H21B	0.9379	0.2164	0.7536	0.021*
C14	1.06858 (16)	0.17006 (19)	0.95410 (13)	0.0207 (4)
H14A	1.0983	0.1501	0.8896	0.031*
H14B	1.0900	0.1001	1.0020	0.031*
H14C	1.1071	0.2493	0.9817	0.031*
C9	0.78851 (16)	0.31546 (17)	1.03144 (13)	0.0171 (3)
H9	0.8295	0.3954	1.0118	0.021*
C18	0.75488 (17)	−0.05633 (18)	0.63247 (13)	0.0202 (4)
H18A	0.8264	−0.0883	0.6013	0.024*
H18B	0.6840	−0.1132	0.6111	0.024*
C16	0.78556 (16)	−0.06097 (17)	0.74921 (13)	0.0170 (3)
H16	0.7063	−0.0656	0.7790	0.020*
C12	0.5522 (2)	0.4949 (2)	1.23442 (16)	0.0331 (5)
H12A	0.5295	0.5680	1.1899	0.050*

H12B	0.5200	0.5075	1.2990	0.050*
H12C	0.5170	0.4166	1.2021	0.050*
C2	0.36654 (19)	0.3986 (2)	0.86908 (16)	0.0288 (4)
H2	0.3510	0.4224	0.9351	0.035*
C10	0.75218 (16)	0.33115 (19)	1.13720 (14)	0.0203 (4)
C19	0.72420 (17)	0.07729 (18)	0.59540 (13)	0.0185 (4)
C28	0.9733 (2)	0.2135 (3)	0.4377 (2)	0.0452 (6)
C29	0.9077 (2)	0.1088 (2)	0.38715 (17)	0.0317 (5)
H29A	0.9422	0.0895	0.3245	0.048*
H29B	0.8205	0.1315	0.3709	0.048*
H29C	0.9155	0.0335	0.4313	0.048*
C27	0.49224 (18)	0.1638 (2)	0.32940 (14)	0.0260 (4)
H27A	0.4375	0.1993	0.2721	0.039*
H27B	0.4506	0.0933	0.3595	0.039*
H27C	0.5678	0.1322	0.3056	0.039*
C1	0.48732 (18)	0.38403 (19)	0.84900 (15)	0.0235 (4)
C11	0.69039 (18)	0.4832 (2)	1.25307 (14)	0.0283 (4)
H11A	0.7266	0.5643	1.2812	0.034*
H11B	0.7138	0.4145	1.3030	0.034*
C3	0.26836 (19)	0.3788 (2)	0.79368 (18)	0.0326 (5)
H3	0.1862	0.3895	0.8082	0.039*
C5	0.41007 (19)	0.3281 (2)	0.67694 (15)	0.0276 (4)
H5A	0.4253	0.3031	0.6111	0.033*
C4	0.28987 (19)	0.3435 (2)	0.69729 (17)	0.0303 (4)
H4	0.2228	0.3300	0.6455	0.036*
N3	1.0276 (3)	0.2938 (3)	0.4810 (3)	0.0866 (12)
C7	0.59528 (19)	0.40899 (19)	0.93067 (15)	0.0268 (4)
H7A	0.5636	0.4384	0.9931	0.032*
H7B	0.6473	0.4778	0.9079	0.032*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0299 (2)	0.01598 (18)	0.01952 (19)	0.00160 (16)	0.00259 (15)	0.00102 (15)
O6	0.0268 (7)	0.0263 (7)	0.0257 (7)	0.0052 (6)	−0.0092 (6)	0.0006 (6)
O5	0.0232 (7)	0.0160 (7)	0.0432 (9)	−0.0014 (5)	−0.0095 (6)	0.0032 (6)
O3	0.0274 (6)	0.0178 (6)	0.0161 (5)	−0.0040 (5)	0.0039 (5)	0.0010 (5)
O4	0.0188 (6)	0.0201 (6)	0.0235 (7)	0.0006 (5)	−0.0033 (5)	0.0023 (5)
O2	0.0287 (7)	0.0232 (7)	0.0213 (6)	−0.0008 (5)	0.0084 (5)	−0.0059 (5)
C22	0.0185 (8)	0.0208 (9)	0.0135 (8)	−0.0032 (7)	0.0040 (6)	−0.0023 (6)
O1	0.0358 (8)	0.0269 (7)	0.0250 (7)	0.0003 (6)	0.0121 (6)	0.0033 (6)
O7	0.0258 (7)	0.0203 (7)	0.0211 (6)	0.0029 (5)	−0.0020 (5)	0.0027 (5)
N2	0.0198 (7)	0.0162 (7)	0.0150 (7)	−0.0004 (6)	−0.0006 (6)	−0.0003 (6)
C25	0.0200 (9)	0.0220 (9)	0.0184 (8)	−0.0002 (7)	−0.0005 (7)	−0.0030 (7)
C17	0.0204 (8)	0.0181 (9)	0.0117 (8)	0.0003 (7)	−0.0007 (6)	−0.0003 (6)
C23	0.0204 (8)	0.0206 (9)	0.0156 (8)	0.0040 (7)	0.0068 (6)	0.0016 (7)
C15	0.0179 (8)	0.0163 (8)	0.0157 (8)	0.0040 (6)	0.0009 (6)	0.0003 (6)
C26	0.0367 (11)	0.0193 (10)	0.0260 (10)	0.0000 (8)	−0.0019 (8)	0.0028 (8)

N1	0.0168 (7)	0.0175 (7)	0.0116 (6)	0.0005 (5)	0.0006 (5)	−0.0003 (5)
C20	0.0172 (8)	0.0235 (9)	0.0112 (7)	0.0007 (7)	0.0037 (6)	0.0000 (7)
C6	0.0213 (9)	0.0263 (10)	0.0253 (10)	0.0046 (7)	0.0037 (7)	0.0044 (8)
C8	0.0204 (9)	0.0230 (9)	0.0187 (9)	0.0022 (7)	−0.0002 (7)	−0.0019 (7)
C24	0.0169 (8)	0.0270 (10)	0.0162 (8)	0.0036 (7)	−0.0003 (7)	0.0006 (7)
C13	0.0197 (8)	0.0167 (8)	0.0116 (7)	0.0018 (7)	0.0017 (6)	0.0007 (6)
C21	0.0173 (8)	0.0206 (9)	0.0142 (8)	−0.0013 (6)	0.0015 (6)	0.0003 (6)
C14	0.0190 (9)	0.0254 (10)	0.0179 (8)	0.0000 (7)	0.0026 (7)	−0.0005 (7)
C9	0.0189 (8)	0.0155 (8)	0.0172 (8)	0.0010 (7)	0.0030 (6)	−0.0025 (6)
C18	0.0243 (9)	0.0191 (9)	0.0160 (8)	0.0011 (7)	−0.0015 (7)	−0.0027 (7)
C16	0.0188 (8)	0.0170 (8)	0.0151 (8)	−0.0006 (7)	0.0010 (6)	−0.0008 (6)
C12	0.0349 (11)	0.0371 (12)	0.0291 (10)	0.0074 (10)	0.0111 (8)	−0.0039 (9)
C2	0.0284 (10)	0.0317 (11)	0.0274 (10)	0.0094 (8)	0.0079 (8)	0.0054 (8)
C10	0.0158 (8)	0.0251 (9)	0.0203 (9)	−0.0016 (7)	0.0032 (7)	−0.0035 (7)
C19	0.0200 (9)	0.0210 (9)	0.0146 (8)	0.0016 (7)	0.0029 (7)	−0.0018 (7)
C28	0.0409 (13)	0.0531 (16)	0.0477 (14)	−0.0144 (12)	0.0293 (12)	−0.0160 (12)
C29	0.0277 (10)	0.0404 (12)	0.0278 (10)	−0.0020 (9)	0.0064 (8)	−0.0019 (9)
C27	0.0229 (9)	0.0316 (10)	0.0207 (9)	−0.0006 (8)	−0.0071 (7)	0.0004 (8)
C1	0.0251 (9)	0.0203 (9)	0.0248 (9)	0.0070 (7)	0.0020 (8)	0.0032 (7)
C11	0.0311 (10)	0.0326 (10)	0.0222 (9)	−0.0015 (9)	0.0077 (7)	−0.0106 (9)
C3	0.0211 (10)	0.0326 (11)	0.0443 (13)	0.0066 (8)	0.0054 (9)	0.0093 (9)
C5	0.0323 (10)	0.0259 (10)	0.0239 (10)	0.0029 (8)	0.0013 (8)	0.0012 (8)
C4	0.0261 (10)	0.0230 (10)	0.0388 (12)	0.0017 (8)	−0.0069 (8)	0.0042 (8)
N3	0.081 (2)	0.088 (2)	0.105 (2)	−0.0498 (18)	0.0661 (19)	−0.059 (2)
C7	0.0289 (10)	0.0260 (11)	0.0243 (9)	0.0101 (8)	−0.0004 (8)	−0.0035 (7)

Geometric parameters (Å, º)

O6—C24	1.372 (2)	C13—C14	1.529 (2)
O6—C27	1.431 (2)	C13—H13	1.0000
O5—C17	1.306 (2)	C21—H21A	0.9900
O5—H5	0.8400	C21—H21B	0.9900
O3—C15	1.232 (2)	C14—H14A	0.9800
O4—C17	1.213 (2)	C14—H14B	0.9800
O2—C10	1.327 (2)	C14—H14C	0.9800
O2—C11	1.462 (2)	C9—C10	1.521 (2)
C22—C23	1.389 (2)	C9—H9	1.0000
C22—C20	1.390 (3)	C18—C19	1.506 (3)
C22—H22	0.9500	C18—C16	1.548 (2)
O1—C10	1.202 (2)	C18—H18A	0.9900
O7—C23	1.368 (2)	C18—H18B	0.9900
O7—C26	1.426 (2)	C16—H16	1.0000
N2—C15	1.336 (2)	C12—C11	1.505 (3)
N2—C16	1.465 (2)	C12—H12A	0.9800
N2—C21	1.471 (2)	C12—H12B	0.9800
C25—C24	1.389 (3)	C12—H12C	0.9800
C25—C19	1.395 (3)	C2—C3	1.388 (3)
C25—H25	0.9500	C2—C1	1.390 (3)

C17—C16	1.522 (2)	C2—H2	0.9500
C23—C24	1.406 (3)	C28—N3	1.141 (4)
C15—C13	1.535 (2)	C28—C29	1.430 (3)
C26—H26A	0.9800	C29—H29A	0.9800
C26—H26B	0.9800	C29—H29B	0.9800
C26—H26C	0.9800	C29—H29C	0.9800
N1—C9	1.501 (2)	C27—H27A	0.9800
N1—C13	1.505 (2)	C27—H27B	0.9800
N1—H1A	0.9200	C27—H27C	0.9800
N1—H1B	0.9200	C1—C7	1.521 (3)
C20—C19	1.386 (3)	C11—H11A	0.9900
C20—C21	1.511 (2)	C11—H11B	0.9900
C6—C5	1.383 (3)	C3—C4	1.385 (3)
C6—C1	1.392 (3)	C3—H3	0.9500
C6—H6	0.9500	C5—C4	1.386 (3)
C8—C7	1.536 (2)	C5—H5A	0.9500
C8—C9	1.537 (2)	C4—H4	0.9500
C8—H8A	0.9900	C7—H7A	0.9900
C8—H8B	0.9900	C7—H7B	0.9900
C24—O6—C27	115.19 (15)	C10—C9—C8	110.46 (14)
C17—O5—H5	109.5	N1—C9—H9	109.4
C10—O2—C11	116.64 (15)	C10—C9—H9	109.4
C23—C22—C20	119.84 (16)	C8—C9—H9	109.4
C23—C22—H22	120.1	C19—C18—C16	111.62 (14)
C20—C22—H22	120.1	C19—C18—H18A	109.3
C23—O7—C26	116.47 (14)	C16—C18—H18A	109.3
C15—N2—C16	115.59 (14)	C19—C18—H18B	109.3
C15—N2—C21	126.25 (15)	C16—C18—H18B	109.3
C16—N2—C21	118.14 (14)	H18A—C18—H18B	108.0
C24—C25—C19	120.04 (17)	N2—C16—C17	109.56 (14)
C24—C25—H25	120.0	N2—C16—C18	112.16 (14)
C19—C25—H25	120.0	C17—C16—C18	110.26 (14)
O4—C17—O5	125.15 (16)	N2—C16—H16	108.2
O4—C17—C16	122.96 (16)	C17—C16—H16	108.2
O5—C17—C16	111.88 (14)	C18—C16—H16	108.2
O7—C23—C22	124.31 (17)	C11—C12—H12A	109.5
O7—C23—C24	116.23 (16)	C11—C12—H12B	109.5
C22—C23—C24	119.46 (17)	H12A—C12—H12B	109.5
O3—C15—N2	122.80 (17)	C11—C12—H12C	109.5
O3—C15—C13	118.93 (15)	H12A—C12—H12C	109.5
N2—C15—C13	118.26 (15)	H12B—C12—H12C	109.5
O7—C26—H26A	109.5	C3—C2—C1	120.7 (2)
O7—C26—H26B	109.5	C3—C2—H2	119.7
H26A—C26—H26B	109.5	C1—C2—H2	119.7
O7—C26—H26C	109.5	O1—C10—O2	126.37 (17)
H26A—C26—H26C	109.5	O1—C10—C9	123.12 (18)
H26B—C26—H26C	109.5	O2—C10—C9	110.47 (16)

C9—N1—C13	112.41 (13)	C20—C19—C25	119.49 (17)
C9—N1—H1A	109.1	C20—C19—C18	117.64 (16)
C13—N1—H1A	109.1	C25—C19—C18	122.78 (16)
C9—N1—H1B	109.1	N3—C28—C29	177.4 (4)
C13—N1—H1B	109.1	C28—C29—H29A	109.5
H1A—N1—H1B	107.9	C28—C29—H29B	109.5
C19—C20—C22	120.94 (16)	H29A—C29—H29B	109.5
C19—C20—C21	116.57 (16)	C28—C29—H29C	109.5
C22—C20—C21	122.49 (16)	H29A—C29—H29C	109.5
C5—C6—C1	120.51 (18)	H29B—C29—H29C	109.5
C5—C6—H6	119.7	O6—C27—H27A	109.5
C1—C6—H6	119.7	O6—C27—H27B	109.5
C7—C8—C9	112.03 (15)	H27A—C27—H27B	109.5
C7—C8—H8A	109.2	O6—C27—H27C	109.5
C9—C8—H8A	109.2	H27A—C27—H27C	109.5
C7—C8—H8B	109.2	H27B—C27—H27C	109.5
C9—C8—H8B	109.2	C2—C1—C6	118.74 (18)
H8A—C8—H8B	107.9	C2—C1—C7	120.87 (18)
O6—C24—C25	123.58 (17)	C6—C1—C7	120.37 (17)
O6—C24—C23	116.26 (16)	O2—C11—C12	109.82 (15)
C25—C24—C23	120.15 (16)	O2—C11—H11A	109.7
N1—C13—C14	110.89 (13)	C12—C11—H11A	109.7
N1—C13—C15	104.89 (14)	O2—C11—H11B	109.7
C14—C13—C15	113.26 (15)	C12—C11—H11B	109.7
N1—C13—H13	109.2	H11A—C11—H11B	108.2
C14—C13—H13	109.2	C4—C3—C2	120.15 (19)
C15—C13—H13	109.2	C4—C3—H3	119.9
N2—C21—C20	108.06 (14)	C2—C3—H3	119.9
N2—C21—H21A	110.1	C6—C5—C4	120.46 (19)
C20—C21—H21A	110.1	C6—C5—H5A	119.8
N2—C21—H21B	110.1	C4—C5—H5A	119.8
C20—C21—H21B	110.1	C3—C4—C5	119.44 (19)
H21A—C21—H21B	108.4	C3—C4—H4	120.3
C13—C14—H14A	109.5	C5—C4—H4	120.3
C13—C14—H14B	109.5	C1—C7—C8	111.73 (16)
H14A—C14—H14B	109.5	C1—C7—H7A	109.3
C13—C14—H14C	109.5	C8—C7—H7A	109.3
H14A—C14—H14C	109.5	C1—C7—H7B	109.3
H14B—C14—H14C	109.5	C8—C7—H7B	109.3
N1—C9—C10	107.06 (14)	H7A—C7—H7B	107.9
N1—C9—C8	111.17 (14)		
C26—O7—C23—C22	14.3 (2)	C15—N2—C16—C18	166.61 (15)
C26—O7—C23—C24	−166.07 (16)	C21—N2—C16—C18	−12.1 (2)
C20—C22—C23—O7	179.68 (16)	O4—C17—C16—N2	−16.0 (2)
C20—C22—C23—C24	0.0 (3)	O5—C17—C16—N2	165.23 (15)
C16—N2—C15—O3	−0.8 (2)	O4—C17—C16—C18	107.90 (19)
C21—N2—C15—O3	177.83 (16)	O5—C17—C16—C18	−70.85 (19)

C16—N2—C15—C13	−179.54 (14)	C19—C18—C16—N2	−36.4 (2)
C21—N2—C15—C13	−0.9 (3)	C19—C18—C16—C17	−158.82 (15)
C23—C22—C20—C19	−2.0 (3)	C11—O2—C10—O1	−4.1 (3)
C23—C22—C20—C21	177.53 (15)	C11—O2—C10—C9	173.57 (14)
C27—O6—C24—C25	−29.9 (3)	N1—C9—C10—O1	−39.3 (2)
C27—O6—C24—C23	150.20 (17)	C8—C9—C10—O1	81.9 (2)
C19—C25—C24—O6	177.08 (17)	N1—C9—C10—O2	142.99 (15)
C19—C25—C24—C23	−3.0 (3)	C8—C9—C10—O2	−95.82 (17)
O7—C23—C24—O6	2.7 (2)	C22—C20—C19—C25	1.4 (3)
C22—C23—C24—O6	−177.65 (16)	C21—C20—C19—C25	−178.13 (16)
O7—C23—C24—C25	−177.23 (16)	C22—C20—C19—C18	178.01 (16)
C22—C23—C24—C25	2.5 (3)	C21—C20—C19—C18	−1.5 (2)
C9—N1—C13—C14	133.60 (15)	C24—C25—C19—C20	1.1 (3)
C9—N1—C13—C15	−103.80 (15)	C24—C25—C19—C18	−175.32 (17)
O3—C15—C13—N1	−21.2 (2)	C16—C18—C19—C20	44.4 (2)
N2—C15—C13—N1	157.68 (15)	C16—C18—C19—C25	−139.15 (18)
O3—C15—C13—C14	99.90 (19)	C3—C2—C1—C6	−0.2 (3)
N2—C15—C13—C14	−81.27 (19)	C3—C2—C1—C7	178.4 (2)
C15—N2—C21—C20	−124.98 (18)	C5—C6—C1—C2	−0.3 (3)
C16—N2—C21—C20	53.6 (2)	C5—C6—C1—C7	−178.89 (19)
C19—C20—C21—N2	−46.4 (2)	C10—O2—C11—C12	−92.1 (2)
C22—C20—C21—N2	134.08 (17)	C1—C2—C3—C4	0.3 (3)
C13—N1—C9—C10	−176.13 (14)	C1—C6—C5—C4	0.7 (3)
C13—N1—C9—C8	63.13 (18)	C2—C3—C4—C5	0.1 (3)
C7—C8—C9—N1	−167.02 (15)	C6—C5—C4—C3	−0.6 (3)
C7—C8—C9—C10	74.3 (2)	C2—C1—C7—C8	118.7 (2)
C15—N2—C16—C17	−70.60 (18)	C6—C1—C7—C8	−62.7 (2)
C21—N2—C16—C17	110.70 (16)	C9—C8—C7—C1	175.96 (16)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5···Cl1[i]	0.84	2.09	2.924 (3)	171
N1—H1*A*···O3	0.92	2.04	2.587 (2)	117
N1—H1*B*···O4[ii]	0.92	1.87	2.724 (3)	154
C13—H13···Cl1	1.00	2.52	3.459 (2)	157
C14—H14*B*···Cl1[iii]	0.98	2.71	3.593 (2)	150
C18—H18*A*···N3[iv]	0.99	2.39	3.367 (4)	168
C29—H29*A*···Cl1[iv]	0.98	2.74	3.698 (2)	165

Symmetry codes: (i) *x*, *y*−1, *z*; (ii) −*x*+2, *y*+1/2, −*z*+2; (iii) −*x*+2, *y*−1/2, −*z*+2; (iv) −*x*+2, *y*−1/2, −*z*+1.