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Key indicators

Single-crystal X-ray study
 T = 298 K
 Mean $\sigma(C-C) = 0.003 \text{ \AA}$
 Disorder in main residue
 R factor = 0.048
 wR factor = 0.128
 Data-to-parameter ratio = 34.0

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Amlodipine mesylate monohydrate: a twinned and disordered structure

In the title compound, (*R,S*)-3-ethyl 5-methyl 2-(2-ammonioethoxymethyl)-4-(2-chlorophenyl)-6-methyl-1,4-dihydropyridine-3,5-dicarboxylate methanesulfonate monohydrate, $C_{20}H_{26}ClN_2O_5^+ \cdot CH_3O_3S^- \cdot H_2O$, the cation, anion and water molecule are linked by extensive hydrogen bonding which results in the formation of ribbons in the *c* direction.

Received 27 October 2006
 Accepted 2 November 2006

Comment

The title compound, (I), $C_{21}H_{31}N_2O_9ClS$, is a derivative of amlodipine, the latter being anti-anginal and antihypertensive. Amlodipine itself is a calcium channel blocker used for heart medication in the treatment of hypertension (Murdoch & Heel, 1991). The asymmetric unit contains an amlodipine cation, a mesylate anion and a water molecule all linked by extensive hydrogen bonding (Fig. 1). The geometric parameters of the amlodipine cation are similar to those found for phthaloyl amlodipine (Yathirajan *et al.*, 2005) and other structurally characterized amlodipine derivatives (Goldmann *et al.*, 1992; Mereiter & Rollinger, 2002).

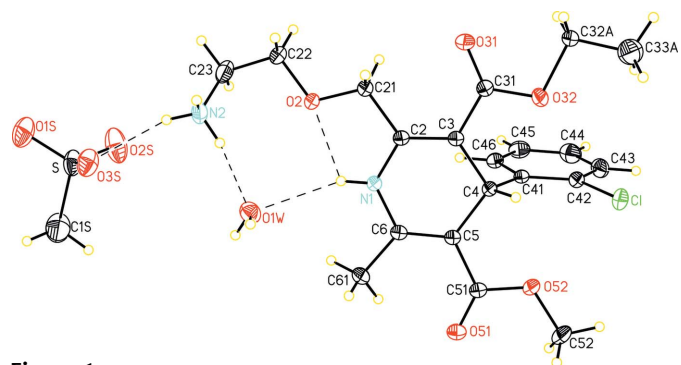
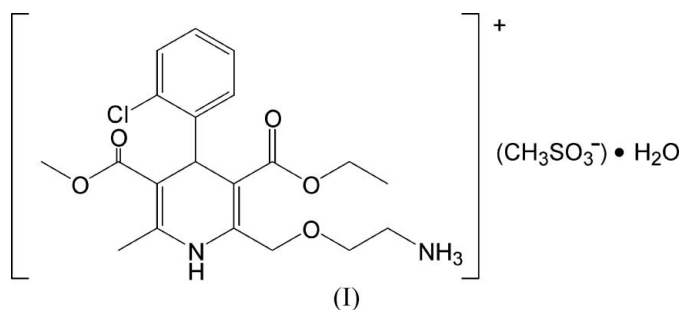


Figure 1
 View of the asymmetric unit of (I), showing the atom-numbering scheme with displacement ellipsoids drawn at the 20% probability level. Hydrogen bonds are drawn as dashed lines. Only the major component of the disordered ethyl group is shown.

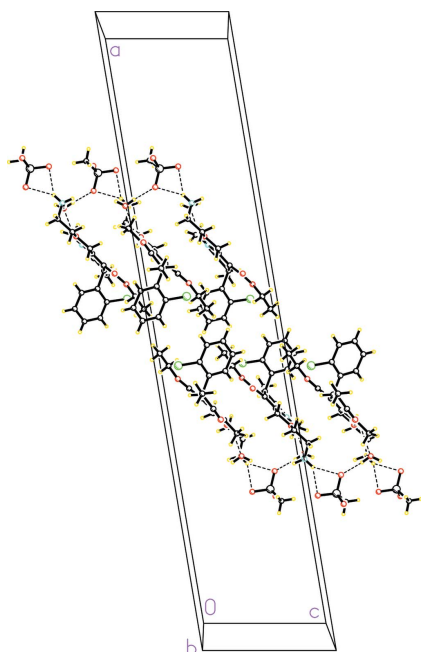


Figure 2

The molecular packing of (I), viewed down the *b* axis, with hydrogen bonds drawn as dashed lines. Only the major disorder component is shown.

There is an extensive hydrogen-bonding network involving the amine H atoms, the mesylate anion and the water H atoms (Table 2), which form ribbons in the *c* direction.

Experimental

The title compound was obtained as a gift sample from CIPLA, Mumbai, India. The compound was used without further purification. Recrystallization from dichloromethane yielded X-ray diffraction quality crystals (m.p. 405 K).

Crystal data

$C_{20}H_{26}ClN_2O_5^+ \cdot CH_3O_3S^- \cdot H_2O$
 $M_r = 522.99$
 Monoclinic, *C2/c*
 $a = 47.656$ (11) Å
 $b = 11.069$ (2) Å
 $c = 9.848$ (2) Å
 $\beta = 99.560$ (6)°
 $V = 5122.3$ (19) Å³

$Z = 8$
 $D_x = 1.356$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.28$ mm⁻¹
 $T = 298$ (2) K
 Needle, colorless
 $0.54 \times 0.21 \times 0.16$ mm

Data collection

Bruker APEX-II CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.802$, $T_{\max} = 1.000$

10949 measured reflections
 10949 independent reflections
 6706 reflections with $I > 2\sigma(I)$
 $\theta_{\max} = 26.7^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.128$
 $S = 0.95$
 10949 reflections
 322 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0667P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.28$ e Å⁻³
 $\Delta\rho_{\min} = -0.28$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Cl—C42	1.743 (2)	O52—C51	1.319 (2)
O2—C22	1.413 (2)	O52—C52	1.436 (2)
O2—C21	1.4163 (19)	N1—C2	1.367 (2)
O31—C31	1.2126 (19)	N1—C6	1.379 (2)
O32—C31	1.342 (2)	N2—C23	1.460 (3)
O51—C51	1.186 (2)		
C22—O2—C21	111.66 (12)	O31—C31—O32	121.78 (15)
C31—O32—C32A	107.1 (5)	O51—C51—O52	120.59 (17)
C2—N1—C6	123.19 (14)		

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O1W—H1W2···O1S ⁱ	0.830 (15)	1.939 (15)	2.767 (2)	176 (3)
O1W—H1W1···O2S ⁱⁱ	0.842 (15)	1.875 (15)	2.704 (3)	168 (2)
N1—H1A···O2	0.86	2.14	2.5670 (17)	111
N1—H1A···O1W	0.86	2.42	3.174 (2)	146
N2—H2A···O2S	0.89	2.23	3.032 (3)	150
N2—H2A···O3S	0.89	2.24	2.971 (2)	140
N2—H2A···S	0.89	2.70	3.5744 (18)	167
N2—H2B···O1S ⁱⁱⁱ	0.89	2.07	2.949 (3)	168
N2—H2B···O3S ⁱⁱⁱ	0.89	2.52	2.985 (2)	113
N2—H2B···S ⁱⁱⁱ	0.89	2.77	3.5299 (19)	144
N2—H2C···O1W	0.89	1.89	2.770 (2)	169

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

The crystal was twinned (non-merohedral) and the two domains were determined with the program *CELL-NOW* (Sheldrick, 2003). A data file was generated with separate scale factors for the two components. During final refinement this refined to 0.2947 (6). The ethyl group attached to O32 is disordered over two conformations [occupancy factor of 0.641 (3) for the major component]. All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry with C—H distances of 0.96 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$, but each group was allowed to rotate freely about its C—C bond. All other H atoms bound to C were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances in the range 0.93–0.98 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The H attached to N1 was idealized with an N—H distance of 0.86 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$. The H atoms attached to N2 were constrained to an ideal geometry with N—H distances of 0.89 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{N})$, but each group was allowed to rotate freely about its C—N bond. The H atoms of the water molecule were located in a difference Fourier map and their positional parameters were refined freely, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$.

Data collection: *APEX2* (Bruker, 2006); cell refinement: *CELL-NOW* (Sheldrick, 2003); data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2003); software used to prepare material for publication: *SHELXTL*.

One of the authors (RSN) thanks the CIPLA Company, Mumbai, for a gift sample of amlodipine mesylate monohydrate.

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