Caesium chloride 2-(*N*-morpholinio)ethanesulfonate

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In the structure of caesium chloride 2-(*N*-morpholinio)ethanesulfonate, $Cs^+ \cdot Cl^- \cdot C_6H_{13}NO_4S$, determined by X-ray diffraction analysis at 100 K, the caesium cation is coordinated by the chloride anion and six O atoms.

Comment

Caesium chloride and 2-(*N*-morpholino)ethanesulfonic acid (MES) are components of some screens for the crystallization of biological macromolecules. MES is one of the Good buffers (Good *et al.*, 1966), while caesium chloride is mainly used as a crystallization agent. It is not rare that in some crystallization trials, instead of the expected biomacromolecular crystals, crystals of other mother liquor components are formed. In this paper, we report the results of the structural study on a crystal of caesium chloride 2-(*N*-morpholinio)ethanesulfonate, (I), which was obtained serendipitously while trying to crystallize a certain oligopeptide.

(I)
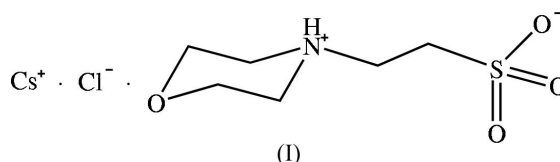
In the asymmetric unit of (I), there is one molecule of MES in the zwitterionic form, one caesium cation and one chloride anion (Fig. 1). The caesium cation is coordinated by six O atoms and the chloride anion (Fig. 2). Geometrical parameters
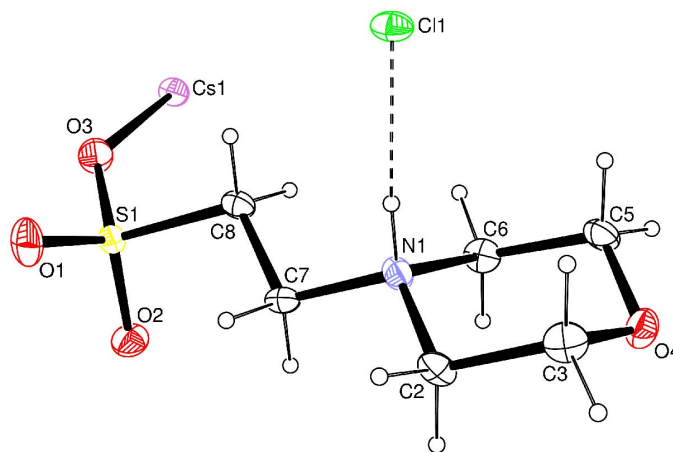
Figure 1
The asymmetric unit of (I). The N1—H1···Cl1 hydrogen bond is denoted by a dashed line. Displacement ellipsoids are drawn at the 50% probability level.

of the MES molecule are similar to those found in the structures of zwitterionic 2-(*N*-morpholino)ethanesulfonic acid monohydrate (Nørlund Christensen *et al.*, 1993) and sodium 2-(*N*-morpholino)ethanesulfonate monohydrate (Deschamps *et al.*, 2002), except for the C—N bond distances which are, as expected, significantly shorter in the structure of the sodium salt due to the unprotonated N atom. A hydrogen bond is formed between Cl1 and protonated atom N1 (Table 2).

Experimental

Crystals of (I) were grown by the sitting drop vapour diffusion method. Equal volumes of an aqueous solution of the natural octapeptide PGKPADDA (20 mg ml^{-1}) and solution No. 24 from Hampton Research Crystal Screen 2 [0.05 *M* CsCl, 0.1 *M* MES (pH 6.5) and 30% (*v*/*v*) Jeffamine M-600] were mixed and equilibrated against the reservoir solution at 291 K. Crystals suitable for data collection were obtained after seven months.

Crystal data

$Cs^{+} \cdot Cl^{-} \cdot C_6H_{13}NO_4S$	$D_x = 2.093$ Mg m^{-3}
$M_r = 363.59$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 2097
$a = 12.110\,(2)$ Å	reflections
$b = 8.1753\,(15)$ Å	$\theta = 3.0–26.5°$
$c = 12.566\,(2)$ Å	$\mu = 3.61$ mm^{-1}
$\beta = 111.963\,(17)°$	$T = 100\,(1)$ K
$V = 1153.7\,(4)$ Å^3	Needle, colourless
$Z = 4$	$0.40 \times 0.04 \times 0.04$ mm

Data collection

Oxford Diffraction Xcalibur CCD diffractometer	3341 independent reflections
	2688 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.059$
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2003)	$\theta_{max} = 30.0°$
	$h = -16 \rightarrow 17$
	$k = -11 \rightarrow 11$
$T_{min} = 0.489$, $T_{max} = 0.862$	$l = -17 \rightarrow 17$
15065 measured reflections	

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.046$	$w = 1/[\sigma^2(F_o^2) + (0.0661P)^2]$
$wR(F^2) = 0.120$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.10$	$(\Delta/\sigma)_{max} = 0.001$
3341 reflections	$\Delta\rho_{max} = 2.99$ e Å^{-3}
127 parameters	$\Delta\rho_{min} = -1.58$ e Å^{-3}

Table 1
Selected bond lengths (Å).

Cs1—O3	3.025 (3)	Cs1—O2^{iv}	3.148 (4)
Cs1—O1^{i}	3.014 (3)	Cs1—Cl1^{v}	3.7783 (14)
Cs1—O1^{ii}	3.348 (3)	N1—C2	1.487 (5)
Cs1—O3^{ii}	3.143 (3)	N1—C6	1.501 (5)
Cs1—O4^{iii}	3.069 (3)	N1—C7	1.503 (6)

Symmetry codes: (i) $x, y - 1, z$; (ii) $-\frac{3}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (iii) $-\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (iv) $-1 - x, -y, 1 - z$; (v) $-1 - x, -y, -z$.

Figure 2
Coordination of the caesium cation. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $x, y - 1, z$; (ii) $-\frac{3}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (iii) $-\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (iv) $-1 - x, -y, 1 - z$; (v) $-1 - x, -y, -z$.]
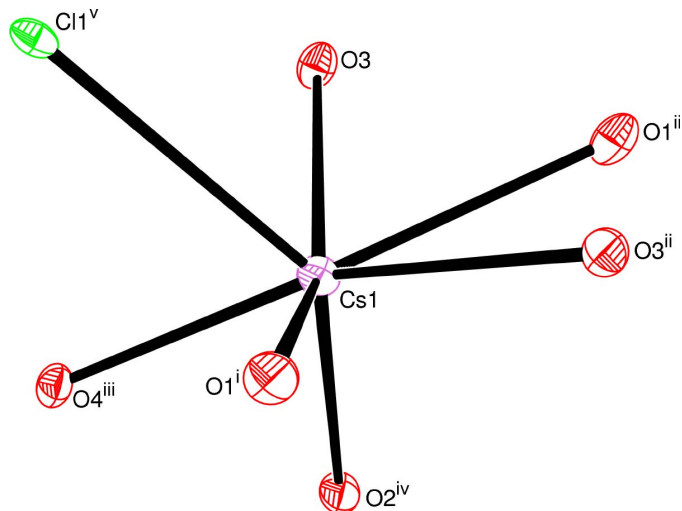
Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$\cdots$Cl1	0.93	2.11	3.039 (4)	173

All H atoms were positioned geometrically (N—H = 0.83, C—H = 0.99 Å) and refined using the riding model, with $U_{iso}(H) = 1.2U_{eq}(C,N)$. The highest peak and deepest hole in the difference map are located 0.68 and 0.71 Å, respectively, from atom Cs1.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2003); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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