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(Methoxycarbonyl)hydrazinium chloride monohydrate

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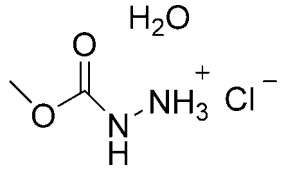
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(O–C) = 0.002 Å; R factor = 0.030; wR factor = 0.080; data-to-parameter ratio = 14.9.

In the title compound, $C_2H_7N_2O_2^+\cdot Cl^-\cdot H_2O$, the non-H atoms of the cation are approximately coplanar. The organic cations, chloride ions and water molecules are linked into a two-dimensional network parallel to the bc plane by N—H···O, N—H···Cl and O—H···Cl hydrogen bonds.

Related literature

For applications of benzaldehydehydrazone derivatives, see: Parashar *et al.* (1988); Hadjoudis *et al.* (1987). For the crystal structure of a nickel methylcarbazate complex, see: Song *et al.* (2003).



Experimental

Crystal data

$C_2H_7N_2O_2^+\cdot Cl^-\cdot H_2O$
M_r = 144.56
Monoclinic, $P2_1/c$
a = 12.6621 (13) Å
b = 7.6444 (7) Å
c = 6.6948 (7) Å
β = 97.199 (4)°
V = 642.91 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.53 mm^{-1}
T = 123 (2) K
0.28 × 0.24 × 0.23 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.861, T_{max} = 0.881
7105 measured reflections
1445 independent reflections
1360 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.080
S = 1.04
1445 reflections
97 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.59 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1W	0.92 (2)	1.84 (2)	2.743 (2)	167 (2)
N1—H1B···Cl1^{i}	0.93 (2)	2.20 (2)	3.1152 (14)	168 (2)
N1—H1C···O1^{ii}	0.89 (2)	2.00 (2)	2.8443 (17)	158 (2)
O1W—H1W···Cl1^{iii}	0.85 (2)	2.41 (3)	3.2172 (16)	161 (3)
N2—H2···Cl1^{iv}	0.86 (1)	2.33 (1)	3.1833 (13)	171 (2)
O1W—H2W···Cl1	0.82 (2)	2.58 (3)	3.1959 (14)	133 (3)

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2680).

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supplementary materials

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(Methoxycarbonyl)hydrazinium chloride monohydrate

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Comment

Benzaldehydehydrazone derivatives have received considerable attention for a long time due to their pharmacological activity (Parashar *et al.*, 1988) and their photochromic properties (Hadjoudis *et al.*, 1987). The title compound is an important intermediate in the synthesis of benzaldehydehydrazone derivatives. We report here the crystal structure of the title compound (Fig. 1).

In the cation, atoms O1, O2, N2, C1 and C2 are coplanar (r.m.s. deviation 0.029 Å) and atom N1 deviates by 0.260 (2) Å from the C1/C2/O1/O2/N2 plane. The bond lengths and angles in the organic cation are comparable to those in a related structure (Song *et al.*, 2003).

The molecules are linked into a two-dimensional network parallel to the *bc* plane by N–H···O, N—H···Cl and O—H···Cl hydrogen bonds involving the water molecule and chloride ions (Table 1 and Fig.2).

Experimental

Methyl hydrazinecarboxylate (0.90 g, 0.01 mol) was dissolved in ethanol- dilute HCl and single crystals suitable for X-ray analysis were obtained by slow evaporation at room temperature (m.p. 463–465 K).

Refinement

O- and N-bound H atoms were located in a difference map and were refined with O-H and N2-H2 distances restrained to 0.85 (2) Å and 0.87 (1) Å, respectively. The methyl H atoms were disordered over two orientations and their occupancies were initially refined and later fixed at 0.75 and 0.25, with C-H = 0.96 å and U_{iso}(H) = 1.5U_{eq}(C).

Figures

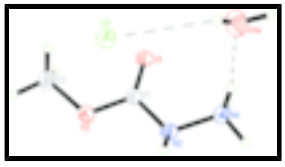

Fig. 1. The molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atomic numbering. Hydrogen bonds are shown as dashed lines.

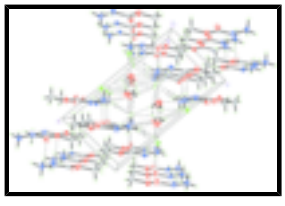

Fig. 2. The crystal packing of the title compound, viewed approximately along the *c* axis. Hydrogen bonds are shown as dashed lines.

(Methoxycarbonyl)hydrazinium chloride monohydrate

Crystal data

$C_2H_7N_2O_2^+ \cdot Cl^- \cdot H_2O$	$F_{000} = 304$
$M_r = 144.56$	$D_x = 1.494$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 1122 reflections
$a = 12.6621$ (13) Å	$\theta = 1.6$–25.0º
$b = 7.6444$ (7) Å	$\mu = 0.53$ mm^{-1}
$c = 6.6948$ (7) Å	$T = 123$ (2) K
$\beta = 97.199$ (4)º	Block, colourless
$V = 642.91$ (11) Å^3	0.28 × 0.24 × 0.23 mm
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	1445 independent reflections
Radiation source: fine-focus sealed tube	1360 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.021$
$T = 273$(2) K	$\theta_{max} = 27.5$º
φ and ω scans	$\theta_{min} = 1.6$º
Absorption correction: multi-scan (SADABS; Bruker, 2002)	$h = -14 \rightarrow 15$
$T_{min} = 0.861$, $T_{max} = 0.881$	$k = -9 \rightarrow 9$
7105 measured reflections	$l = -8 \rightarrow 8$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.030$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.080$	$w = 1/[\sigma^2(F_o^2) + (0.0389P)^2 + 0.2916P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} = 0.001$
1445 reflections	$\Delta\rho_{max} = 0.59$ e Å^{-3}
97 parameters	$\Delta\rho_{min} = -0.30$ e Å^{-3}
3 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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}	Occ. (<1)
O1	0.83051 (8)	0.17986 (13)	0.89504 (15)	0.0351 (2)	
O2	0.88232 (9)	−0.10131 (13)	0.86201 (17)	0.0380 (3)	
N1	0.69905 (11)	0.07910 (18)	1.1544 (2)	0.0343 (3)	
H1A	0.6428 (16)	0.110 (3)	1.061 (3)	0.047 (6)*	
H1B	0.6731 (14)	0.031 (3)	1.267 (3)	0.042 (5)*	
H1C	0.7342 (16)	0.174 (3)	1.203 (3)	0.047 (5)*	
N2	0.77164 (10)	−0.04049 (16)	1.08177 (18)	0.0337 (3)	
H2	0.7430 (14)	−0.1422 (16)	1.064 (3)	0.045 (5)*	
C1	0.82736 (11)	0.02547 (18)	0.93676 (19)	0.0287 (3)	
C2	0.93930 (14)	−0.0505 (2)	0.6966 (3)	0.0441 (4)	
H2A	0.9769	−0.1498	0.6530	0.066*	0.75
H2B	0.9892	0.0405	0.7402	0.066*	0.75
H2C	0.8896	−0.0086	0.5869	0.066*	0.75
H2D	0.9269	0.0712	0.6670	0.066*	0.25
H2E	0.9146	−0.1191	0.5799	0.066*	0.25
H2F	1.0141	−0.0700	0.7332	0.066*	0.25
O1W	0.55461 (12)	0.1831 (2)	0.8367 (2)	0.0561 (4)	
H1W	0.4948 (17)	0.216 (4)	0.867 (4)	0.095 (10)*	
H2W	0.537 (3)	0.109 (4)	0.751 (4)	0.115 (12)*	
Cl1	0.64630 (3)	−0.09761 (5)	0.54932 (5)	0.03710 (14)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0401 (6)	0.0279 (5)	0.0388 (5)	−0.0011 (4)	0.0106 (4)	0.0032 (4)
O2	0.0416 (6)	0.0320 (5)	0.0426 (6)	0.0058 (4)	0.0136 (5)	0.0020 (4)
N1	0.0351 (7)	0.0369 (7)	0.0323 (6)	−0.0037 (5)	0.0101 (5)	−0.0023 (5)
N2	0.0381 (7)	0.0275 (6)	0.0371 (6)	−0.0032 (5)	0.0113 (5)	0.0010 (5)
C1	0.0276 (6)	0.0293 (6)	0.0285 (6)	−0.0009 (5)	0.0013 (5)	0.0005 (5)
C2	0.0430 (9)	0.0471 (9)	0.0455 (8)	0.0020 (7)	0.0182 (7)	−0.0039 (7)
O1W	0.0544 (8)	0.0635 (9)	0.0482 (7)	0.0110 (7)	−0.0022 (6)	−0.0172 (6)
Cl1	0.0391 (2)	0.0390 (2)	0.0344 (2)	0.00628 (14)	0.00935 (14)	0.00276 (13)

Geometric parameters (Å, °)

O1—C1	1.2146 (17)	C2—H2A	0.96
O2—C1	1.3272 (17)	C2—H2B	0.96
O2—C2	1.4486 (19)	C2—H2C	0.96
N1—N2	1.4243 (17)	C2—H2D	0.96
N1—H1A	0.92 (2)	C2—H2E	0.96
N1—H1B	0.93 (2)	C2—H2F	0.96
N1—H1C	0.89 (2)	O1W—H1W	0.847 (17)
N2—C1	1.3661 (17)	O1W—H2W	0.819 (18)
N2—H2	0.860 (9)		
C1—O2—C2	115.31 (12)	H2B—C2—H2C	109.5
N2—N1—H1A	114.1 (12)	O2—C2—H2D	109.5
N2—N1—H1B	109.4 (12)	H2A—C2—H2D	141.1
H1A—N1—H1B	109.2 (16)	H2B—C2—H2D	56.3
N2—N1—H1C	109.5 (13)	H2C—C2—H2D	56.3
H1A—N1—H1C	110.5 (18)	O2—C2—H2E	109.5
H1B—N1—H1C	103.5 (17)	H2A—C2—H2E	56.3
C1—N2—N1	114.74 (12)	H2B—C2—H2E	141.1
C1—N2—H2	118.7 (13)	H2C—C2—H2E	56.3
N1—N2—H2	110.5 (13)	H2D—C2—H2E	109.5
O1—C1—O2	126.08 (13)	O2—C2—H2F	109.5
O1—C1—N2	123.84 (13)	H2A—C2—H2F	56.3
O2—C1—N2	109.90 (12)	H2B—C2—H2F	56.3
O2—C2—H2A	109.5	H2C—C2—H2F	141.1
O2—C2—H2B	109.5	H2D—C2—H2F	109.5
H2A—C2—H2B	109.5	H2E—C2—H2F	109.5
O2—C2—H2C	109.5	H1W—O1W—H2W	102 (3)
H2A—C2—H2C	109.5		
C2—O2—C1—O1	−8.8 (2)	N1—N2—C1—O1	12.3 (2)
C2—O2—C1—N2	175.92 (13)	N1—N2—C1—O2	−172.27 (12)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O1W	0.92 (2)	1.84 (2)	2.743 (2)	167 (2)
N1—H1B···Cl1^{i}	0.93 (2)	2.20 (2)	3.1152 (14)	168 (2)
N1—H1C···O1^{ii}	0.89 (2)	2.00 (2)	2.8443 (17)	158 (2)
O1W—H1W···Cl1^{iii}	0.85 (2)	2.41 (3)	3.2172 (16)	161 (3)
N2—H2···Cl1^{iv}	0.86 (1)	2.33 (1)	3.1833 (13)	171 (2)
O1W—H2W···Cl1	0.82 (2)	2.58 (3)	3.1959 (14)	133 (3)

Symmetry codes: (i) *x*, *y*, *z*+1; (ii) *x*, −*y*+1/2, *z*+1/2; (iii) −*x*+1, *y*+1/2, −*z*+3/2; (iv) *x*, −*y*−1/2, *z*+1/2.

Fig. 1

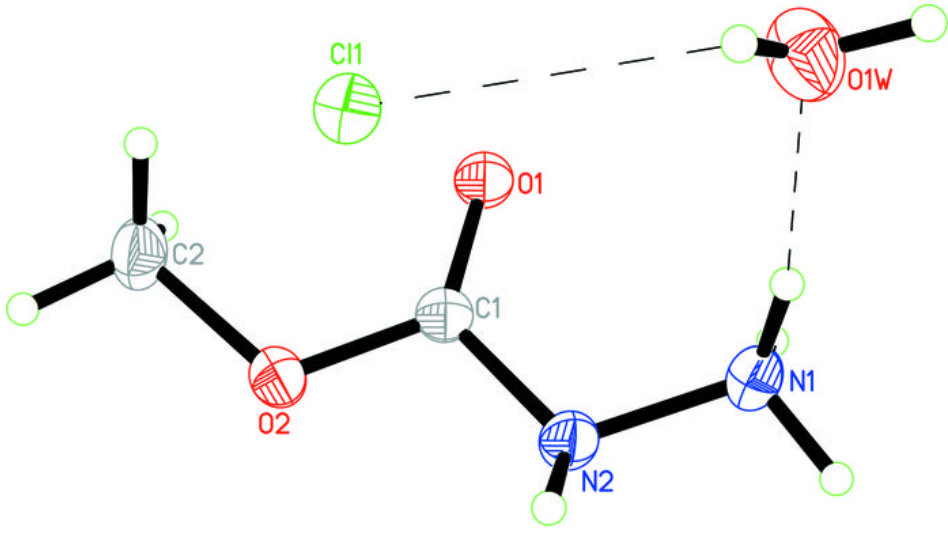

Fig. 2