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Isopropylammonium (isopropylamino)-oxoacetate monohydrate

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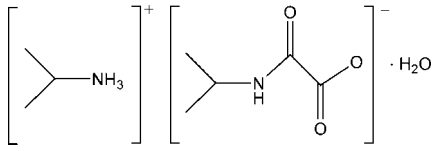
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.106; data-to-parameter ratio = 16.1.

The title compound, $C_3H_{10}N^+\cdot C_5H_8NO_3^-\cdot H_2O$, crystallizes as a salt from water. Intermolecular N—H···O and O—H···O hydrogen bonds lead to two-dimensional layers. The layers are stacked in the c-axis direction with hydrophobic interactions between the methyl groups.

Related literature

For related literature, see: Chena *et al.* (2005); Bellouard *et al.* (1999); Dumonateil (1999).



Experimental

Crystal data

$C_3H_{10}N^+\cdot C_5H_8NO_3^-\cdot H_2O$
M_r = 208.26
Triclinic, $P\overline{1}$
a = 7.172 (4) Å
b = 9.154 (5) Å
c = 9.472 (5) Å
α = 104.372 (8)°
β = 105.944 (8)°
γ = 94.731 (8)°
V = 571.6 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100 (2) K
0.38 × 0.19 × 0.08 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.962, T_{max} = 0.990
6062 measured reflections
2324 independent reflections
2126 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.106
S = 1.16
2324 reflections
144 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H9···O2^{i}	0.82 (3)	2.06 (3)	2.864 (2)	170 (3)
O4—H10···O3	0.88 (2)	1.96 (2)	2.8404 (19)	173.5 (19)
N1—H1···O4^{ii}	0.84 (2)	2.14 (2)	2.918 (2)	153.7 (17)
N2—H2A···O2	0.91	1.89	2.7957 (19)	175
N2—H2B···O3^{ii}	0.91	2.02	2.8808 (19)	157
N2—H2C···O1^{iii}	0.91	1.91	2.8182 (18)	172

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $x-1, y, z$; (iii) $-x, -y, -z$.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001; Atwood & Barbour, 2003); software used to prepare material for publication: *X-SEED*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2088).

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supplementary materials

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Isopropylammonium (isopropylamino)oxoacetate monohydrate

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Comment

Chagas disease, which is caused by Trypanosoma cruzi, is an endemic parasitic disease in Latin America, specially in the Southern part of Mexico (Dumonateil, 1999). The Trypanosoma cruzi enzyme desigenated α-hydroxyacid dehydrogenase (HADH) exhibits two molecular forms (I and II). The trypanocidal activity of *N*-isopropyloxamate (NIPOx) on cultured epimastigotes (*in vitro*) and marine trypanosomiasis (*in vitro*) is used in different Trypanosoma cruzi strains. It is an effective and selective inhibitor of HADH-isozyme II (Chena *et al.*, 2005).

It was serendipitously synthesized during a chromatographic partition of cyclenoxamide. The cyclen and diethyl oxalate were added in an equal molar ratio as starting materials to form the cyclenoxamide at room temperature with stirring for 48 h (Bellouard *et al.*, 1999). The reaction stoichiometry should be one to one, so the excess diethyl oxalate was separated by chromatography on silca ($CHCl_3$:isopropylamine, 5:1). The diethyl oxalate reacted with isopropylamine to produce the title compound at room temperature.

The title compound is a salt, consisting of an *N*-isopropyloxalamate anion, an isopropylaminium cation and a water molecule (Fig. 1). The C1—C2 bond was 1.549 (2) Å which is longer than the typical C(sp^2)—C(sp^2) bond length. This is most likely due to the stretching by hydrogen bonds at the two sides of the bond. There is a partial double bond between the C2 and N1 [1.326 (2) Å] in the amide group. The cation, anion and water molecule are connected by intermolecular N—H···O and O—H···O hydrogen bonds, as detailed in Table 1 and Figs. 1 and 2. This hydrogen-bonding network is extended in the *ab* plane to form a layer. The molecular packing consists of the hydrogen-bonding layers alternating with hydrophobic domains along the *c* direction.

Experimental

The title compound was synthesized by reacting excess diethyl oxalate with isopropylamine in chloroform at room temperature. Crystals were obtained by the slow evaporation method. After two weeks colourless plate crystals were deposited. The compound was characterized by proton NMR in deuterated chloroform as follows: 1.12 p.p.m. (6*H*, d, CH_3 for anion); 1.13 p.p.m. (6*H*, m, CH_3 for cation); 3.40 p.p.m. (1*H*, m, CH for cation); 4.18 p.p.m. (1*H*, m, CH for anion); 7.35 p.p.m. (1*H*, d, NH for anion); 7.97(1*H*, br, NH for cation).

Refinement

H atoms of CH, CH_3 and NH_3 groups were positiond geometrically and refined as riding, with C—H = 1.00Å and U_{iso}(H) = 1.2U_{eq}(C) for CH groups, and with C—H = 0.98 and 0.91Å and U_{iso}(H) = 1.5U_{eq}(C,*N*) for CH_3 and NH_3 groups. H atoms on N1 and on the water molecule were refined isotropically.

Figures

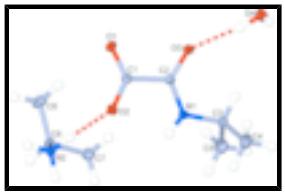

Fig. 1. Molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. The red dashed lines represent hydrogen bonds.

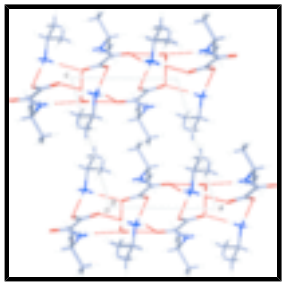

Fig. 2. The molecular packing in the title compound with hydrogen bonds shown as red dashed lines.

Isopropylammonium (isopropylaamino)oxoacetate monohydrate

Crystal data

$C_3H_{10}N^+·C_5H_8NO_3^-·H_2O$	$Z = 2$
$M_r = 208.26$	$F_{000} = 228$
Triclinic, $P\overline{1}$	$D_x = 1.210$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 56 K
$a = 7.172$ (4) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 9.154$ (5) Å	Cell parameters from 3071 reflections
$c = 9.472$ (5) Å	$\theta = 2.3$–26.5º
$\alpha = 104.372$ (8)º	$\mu = 0.10$ mm^{-1}
$\beta = 105.944$ (8)º	$T = 100$ (2) K
$\gamma = 94.731$ (8)º	Plate, colourless
$V = 571.6$ (5) Å^3	0.38 × 0.19 × 0.08 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2324 independent reflections
Radiation source: fine-focus sealed tube	2126 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.023$
$T = 100$(2) K	$\theta_{max} = 26.4$º
φ and ω scans	$\theta_{min} = 2.3$º
Absorption correction: multi-scan (SADABS; Bruker, 2002)	$h = -9 \rightarrow 8$
$T_{min} = 0.962$, $T_{max} = 0.990$	$k = -11 \rightarrow 11$
6062 measured reflections	$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.045$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.106$	$w = 1/[\sigma^2(F_o^2) + (0.0443P)^2 + 0.2432P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.16$	$(\Delta/\sigma)_{max} < 0.001$
2324 reflections	$\Delta\rho_{max} = 0.40$ e Å^{-3}
144 parameters	$\Delta\rho_{min} = -0.21$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.38400 (15)	0.13612 (11)	0.08003 (12)	0.0186 (2)
N1	0.36897 (19)	0.52457 (13)	0.22589 (14)	0.0147 (3)
C1	0.3063 (2)	0.25345 (16)	0.09391 (15)	0.0131 (3)
H1	0.246 (3)	0.512 (2)	0.205 (2)	0.021 (5)*
O2	0.12699 (14)	0.26313 (11)	0.05100 (11)	0.0167 (2)
N2	−0.18564 (17)	0.14413 (13)	0.13379 (13)	0.0140 (3)
H2A	−0.0868	0.1780	0.1010	0.021*
H2B	−0.2743	0.2098	0.1311	0.021*
H2C	−0.2463	0.0496	0.0717	0.021*
C2	0.4503 (2)	0.40621 (16)	0.17327 (15)	0.0131 (3)
O3	0.62792 (14)	0.41075 (11)	0.18756 (12)	0.0175 (2)
C3	0.4797 (2)	0.67704 (16)	0.31163 (17)	0.0169 (3)
H3	0.6232	0.6711	0.3369	0.020*
O4	0.97028 (16)	0.59125 (13)	0.18861 (14)	0.0211 (3)
C4	0.4348 (3)	0.78700 (18)	0.21497 (19)	0.0257 (4)
H4A	0.4721	0.7511	0.1217	0.039*
H4B	0.5093	0.8888	0.2735	0.039*
H4C	0.2938	0.7921	0.1874	0.039*
C5	0.4297 (3)	0.73017 (19)	0.45995 (18)	0.0254 (4)
H5A	0.2892	0.7361	0.4367	0.038*
H5B	0.5061	0.8313	0.5188	0.038*
H5C	0.4616	0.6574	0.5200	0.038*
C6	−0.1027 (2)	0.13576 (16)	0.29428 (16)	0.0161 (3)
H6	−0.2136	0.1022	0.3300	0.019*
C7	−0.0006 (2)	0.29326 (17)	0.39773 (17)	0.0201 (3)
H7A	0.1131	0.3254	0.3678	0.030*
H7B	0.0436	0.2897	0.5042	0.030*
H7C	−0.0924	0.3664	0.3880	0.030*
C8	0.0341 (2)	0.01765 (18)	0.29648 (18)	0.0234 (4)

H8A	−0.0399	−0.0829	0.2328	0.035*
H8B	0.0909	0.0136	0.4018	0.035*
H8C	0.1397	0.0460	0.2563	0.035*
H9	0.939 (3)	0.622 (3)	0.113 (3)	0.041 (6)*
H10	0.859 (3)	0.539 (2)	0.184 (2)	0.033 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0177 (5)	0.0116 (5)	0.0241 (6)	0.0033 (4)	0.0055 (4)	0.0013 (4)
N1	0.0106 (6)	0.0132 (6)	0.0187 (6)	0.0023 (5)	0.0038 (5)	0.0022 (5)
C1	0.0147 (7)	0.0148 (7)	0.0106 (6)	0.0025 (5)	0.0053 (5)	0.0032 (5)
O2	0.0127 (5)	0.0166 (5)	0.0203 (5)	0.0014 (4)	0.0049 (4)	0.0046 (4)
N2	0.0122 (6)	0.0107 (6)	0.0170 (6)	0.0016 (4)	0.0041 (5)	0.0007 (5)
C2	0.0140 (7)	0.0142 (7)	0.0122 (6)	0.0033 (5)	0.0040 (5)	0.0050 (5)
O3	0.0128 (5)	0.0138 (5)	0.0248 (6)	0.0026 (4)	0.0067 (4)	0.0021 (4)
C3	0.0131 (7)	0.0129 (7)	0.0201 (7)	0.0006 (5)	0.0040 (6)	−0.0019 (6)
O4	0.0152 (6)	0.0239 (6)	0.0258 (6)	0.0026 (5)	0.0048 (5)	0.0116 (5)
C4	0.0331 (9)	0.0181 (8)	0.0276 (9)	0.0010 (7)	0.0137 (7)	0.0056 (7)
C5	0.0281 (9)	0.0239 (8)	0.0186 (8)	0.0001 (7)	0.0055 (7)	−0.0009 (6)
C6	0.0167 (7)	0.0168 (7)	0.0148 (7)	0.0029 (6)	0.0060 (6)	0.0031 (5)
C7	0.0199 (8)	0.0185 (8)	0.0178 (7)	0.0034 (6)	0.0037 (6)	0.0001 (6)
C8	0.0288 (9)	0.0191 (8)	0.0214 (8)	0.0092 (6)	0.0046 (7)	0.0056 (6)

Geometric parameters (Å, °)

O1—C1	1.2451 (18)	C4—H4A	0.9800
N1—C2	1.3262 (19)	C4—H4B	0.9800
N1—C3	1.4623 (19)	C4—H4C	0.9800
N1—H1	0.84 (2)	C5—H5A	0.9800
C1—O2	1.2554 (18)	C5—H5B	0.9800
C1—C2	1.549 (2)	C5—H5C	0.9800
N2—C6	1.4963 (19)	C6—C7	1.518 (2)
N2—H2A	0.9100	C6—C8	1.519 (2)
N2—H2B	0.9100	C6—H6	1.0000
N2—H2C	0.9100	C7—H7A	0.9800
C2—O3	1.2394 (18)	C7—H7B	0.9800
C3—C4	1.518 (2)	C7—H7C	0.9800
C3—C5	1.518 (2)	C8—H8A	0.9800
C3—H3	1.0000	C8—H8B	0.9800
O4—H9	0.81 (2)	C8—H8C	0.9800
O4—H10	0.88 (2)		
C2—N1—C3	123.84 (13)	H4A—C4—H4C	109.5
C2—N1—H1	118.0 (13)	H4B—C4—H4C	109.5
C3—N1—H1	118.1 (13)	C3—C5—H5A	109.5
O1—C1—O2	128.18 (13)	C3—C5—H5B	109.5
O1—C1—C2	115.46 (12)	H5A—C5—H5B	109.5
O2—C1—C2	116.36 (12)	C3—C5—H5C	109.5

C6—N2—H2A	109.5	H5A—C5—H5C	109.5
C6—N2—H2B	109.5	H5B—C5—H5C	109.5
H2A—N2—H2B	109.5	N2—C6—C7	109.47 (12)
C6—N2—H2C	109.5	N2—C6—C8	109.33 (12)
H2A—N2—H2C	109.5	C7—C6—C8	112.50 (13)
H2B—N2—H2C	109.5	N2—C6—H6	108.5
O3—C2—N1	124.74 (13)	C7—C6—H6	108.5
O3—C2—C1	120.31 (12)	C8—C6—H6	108.5
N1—C2—C1	114.92 (12)	C6—C7—H7A	109.5
N1—C3—C4	110.08 (13)	C6—C7—H7B	109.5
N1—C3—C5	109.27 (12)	H7A—C7—H7B	109.5
C4—C3—C5	111.50 (13)	C6—C7—H7C	109.5
N1—C3—H3	108.6	H7A—C7—H7C	109.5
C4—C3—H3	108.6	H7B—C7—H7C	109.5
C5—C3—H3	108.6	C6—C8—H8A	109.5
H9—O4—H10	102 (2)	C6—C8—H8B	109.5
C3—C4—H4A	109.5	H8A—C8—H8B	109.5
C3—C4—H4B	109.5	C6—C8—H8C	109.5
H4A—C4—H4B	109.5	H8A—C8—H8C	109.5
C3—C4—H4C	109.5	H8B—C8—H8C	109.5
C3—N1—C2—O3	−1.4 (2)	O1—C1—C2—N1	−162.00 (12)
C3—N1—C2—C1	176.57 (12)	O2—C1—C2—N1	18.40 (17)
O1—C1—C2—O3	16.08 (19)	C2—N1—C3—C4	109.88 (15)
O2—C1—C2—O3	−163.51 (12)	C2—N1—C3—C5	−127.36 (14)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H9···O2^{i}	0.82 (3)	2.06 (3)	2.864 (2)	170 (3)
O4—H10···O3	0.88 (2)	1.96 (2)	2.8404 (19)	173.5 (19)
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Fig. 1

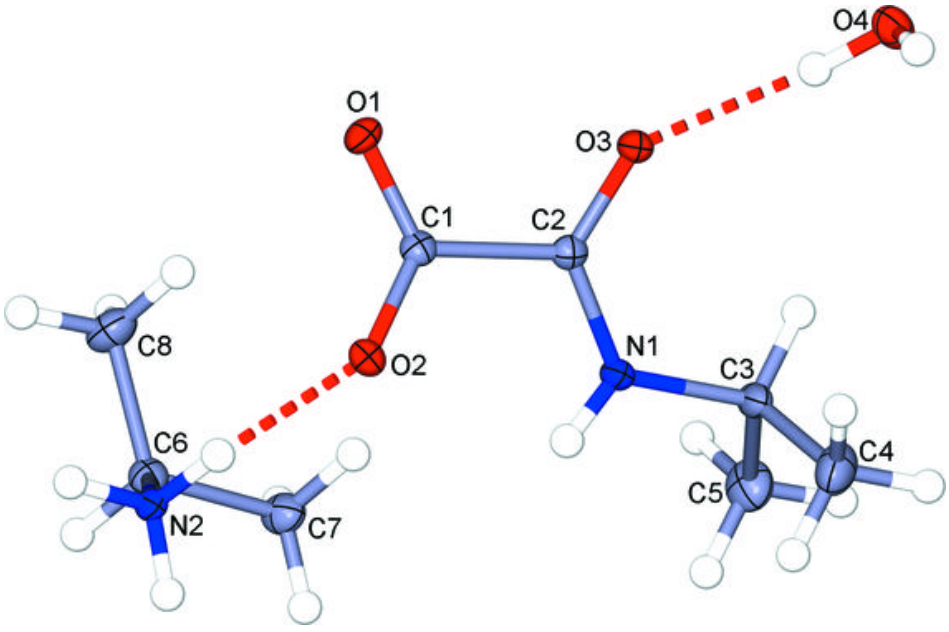

Fig. 2