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[(1-Carboxycyclohexyl)(carboxymethyl)-ammonio]acetate

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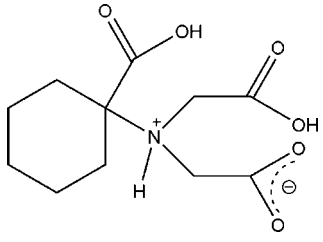
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.128; data-to-parameter ratio = 16.6.

The title compound, $C_{11}H_{17}NO_6$, was prepared by the reaction of 1-aminocyclohexanecarboxylic acid with monochloroacetic acid. In the molecule one of three carboxy groups is deprotonated while the N atom is protonated, thus the molecule exists as an inner salt in the crystal structure. The crystal packing is stabilized by intermolecular O—H···O hydrogen bonding; an intramolecular N—H···O hydrogen bond is also present.

Related literature

For general background, see: Walton & Snurr (2007); Yong *et al.* (2004); Gao *et al.* (2005).



Experimental

Crystal data

$C_{11}H_{17}NO_6$
M_r = 259.26
Orthorhombic, *Pbca*
a = 11.752 (4) Å
b = 8.172 (5) Å
c = 23.524 (3) Å
V = 2259.2 (16) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 293 (2) K
0.33 × 0.31 × 0.28 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
12989 measured reflections
2749 independent reflections
1925 reflections with $I > 2\sigma(I)$
R_{int} = 0.059

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.128
S = 1.05
2749 reflections
166 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O3^{i}	0.82	1.78	2.575 (2)	165
O6—H6···O4^{ii}	0.82	1.80	2.5823 (19)	158
N1—H1*N*···O5	0.891 (13)	2.103 (17)	2.6895 (19)	122.5 (14)

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990); software used to prepare material for publication: *SHELXTL-Plus*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2345).

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supplementary materials

Acta Cryst. (2007). E**63**, o4794 [**doi:10.1107/S1600536807057807**]

[(1-Carboxycyclohexyl)(carboxymethyl)ammonio]acetate

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Comment

Multidentate ligands such as multicarboxylate or amino acid compounds are old but important building blocks for the design and synthesis of metal-organic frameworks (Walton & Snurr, 2007), which have many potential properties in the field of material, catalysis, magnetism and enzyme mimicing (Yong *et al.*, 2004). Multicarboxylate amino acid ligands can be regarded not only as hydrogen-bond accepters, but also as hydrogen-bond donors, depending upon the number of deprotonated carboxylic acid groups present (Gao *et al.*, 2005). Here we report the structure of the title compound, as part of our ongoing studies on self-assemble supramolecular chemistry.

The asymmetric unit of the title compound is illustrated in Fig. 1. Just like most amino acids, one of the three carboxy groups is deprotonated, C10—O3 = 1.259 Å and C10—O4 = 1.258 Å, while the N atom is protonated. In the crystal the molecules link to each other by inter-molecular O—H···O and N—H···O hydrogen bonding. (Table 1)

Experimental

All solvents and chemicals were of analytical grade, commercially available and used as received without further purification. The title compound was synthesized according to the procedure of Yong *et al.* (2004), using 1-amidocycolhexanecarboxylic acid (7.16 g, 50 mmol), monochloroaceticacid (14.18 g, 150 mmol) and sodium hydroxide (1.20 g, 300 mmol) in water (100 ml) as starting materials at room temperature for two days. Single crystals were grown by slow evaporation of its aqueous solution at pH = 5.

Refinement

The amino H atom was located in a difference map and positional parameters were refined with U_{iso}(H) = 1.5U_{eq}(N). Hydroxy H atoms were placed in idealized locations and refined as riding with U_{iso}(H) = 1.5U_{eq}(O). Other H atoms were poisitioned geometrically and refined as riding atoms, with C—H = 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C).

Figures

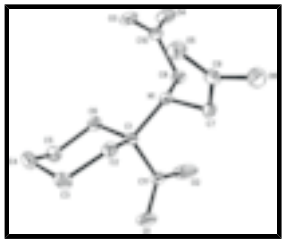

Fig. 1. A view of the molecule of (I). Displacement ellipsoids are drawn at the 30% probability level.

[(1-Carboxycyclohexyl)(carboxymethyl)ammonio]acetate

Crystal data

$C_{11}H_{17}NO_6$	F_{000} = 1104
M_r = 259.26	D_x = 1.524 Mg m^{-3}
Orthorhombic, *Pbca*	Mo $K\alpha$ radiation λ = 0.71069 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 12989 reflections
a = 11.752 (4) Å	θ = 1.7–28.4º
b = 8.172 (5) Å	μ = 0.13 mm^{-1}
c = 23.524 (3) Å	*T* = 293 (2) K
V = 2259.2 (16) Å^3	Block, colorless
Z = 8	0.33 × 0.31 × 0.28 mm

Data collection

Bruker SMART CCD area-detector diffractometer	1925 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.059
Monochromator: graphite	θ_{max} = 28.4º
T = 293(2) K	θ_{min} = 1.7º
φ and ω scans	*h* = −15→11
Absorption correction: none	*k* = −10→8
12989 measured reflections	*l* = −30→31
2749 independent reflections	

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
$wR(F^2)$ = 0.128	$w = 1/[\sigma^2(F_o^2) + (0.0665P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max} < 0.001$
2749 reflections	$\Delta\rho_{max}$ = 0.37 e Å^{-3}
166 parameters	$\Delta\rho_{min}$ = −0.35 e Å^{-3}
1 restraint	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 $F^2 > 2\text{sigma}(F^2)$ 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}
C1	0.95310 (13)	0.08729 (17)	0.13706 (6)	0.0235 (3)
C2	1.03046 (14)	0.08146 (19)	0.18937 (6)	0.0292 (4)
H2A	1.0585	0.1909	0.1970	0.035*
H2B	1.0956	0.0128	0.1809	0.035*
C3	0.97149 (16)	0.0158 (2)	0.24294 (7)	0.0380 (4)
H3A	0.9572	−0.1003	0.2382	0.046*
H3B	1.0221	0.0291	0.2752	0.046*
C4	0.86068 (16)	0.1014 (2)	0.25536 (7)	0.0434 (5)
H4A	0.8244	0.0511	0.2880	0.052*
H4B	0.8751	0.2154	0.2643	0.052*
C5	0.78311 (15)	0.0902 (2)	0.20458 (8)	0.0386 (4)
H5A	0.7125	0.1470	0.2128	0.046*
H5B	0.7651	−0.0238	0.1974	0.046*
C6	0.83729 (14)	0.1643 (2)	0.15166 (7)	0.0308 (4)
H6A	0.8474	0.2809	0.1575	0.037*
H6B	0.7861	0.1499	0.1197	0.037*
C7	1.12467 (13)	0.1299 (2)	0.07254 (7)	0.0308 (4)
H7A	1.1356	0.0210	0.0880	0.037*
H7B	1.1244	0.1217	0.0314	0.037*
C8	0.93952 (15)	0.25861 (19)	0.04386 (6)	0.0319 (4)
H8A	0.8717	0.1915	0.0411	0.038*
H8B	0.9817	0.2474	0.0086	0.038*
C9	1.22038 (15)	0.2396 (2)	0.09137 (7)	0.0323 (4)
C10	0.90488 (14)	0.4370 (2)	0.05165 (7)	0.0319 (4)
C11	0.93530 (13)	−0.07878 (18)	0.10762 (7)	0.0272 (4)
N1	1.01217 (11)	0.19803 (14)	0.09273 (5)	0.0248 (3)
H1N	1.0331 (15)	0.2880 (17)	0.1114 (7)	0.037*
O1	0.94777 (14)	−0.20441 (14)	0.14028 (5)	0.0517 (4)
H1	0.9369	−0.2883	0.1219	0.078*
O2	0.91378 (17)	−0.08999 (16)	0.05870 (6)	0.0711 (6)
O3	0.93773 (11)	0.50724 (13)	0.09639 (5)	0.0398 (3)
O4	0.84846 (11)	0.49857 (14)	0.01155 (6)	0.0449 (4)
O5	1.20469 (11)	0.34997 (15)	0.12491 (5)	0.0446 (3)
O6	1.32020 (10)	0.20820 (15)	0.07025 (6)	0.0471 (4)
H6	1.3158	0.1293	0.0488	0.071*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0276 (8)	0.0173 (7)	0.0255 (8)	−0.0011 (6)	−0.0007 (6)	−0.0003 (6)
C2	0.0314 (9)	0.0262 (8)	0.0299 (8)	−0.0016 (6)	−0.0038 (7)	−0.0029 (6)
C3	0.0489 (11)	0.0372 (10)	0.0280 (8)	−0.0058 (8)	−0.0062 (8)	0.0042 (7)
C4	0.0530 (12)	0.0449 (11)	0.0322 (9)	−0.0116 (9)	0.0122 (8)	0.0011 (8)
C5	0.0331 (10)	0.0347 (9)	0.0480 (11)	−0.0050 (7)	0.0109 (8)	−0.0004 (8)
C6	0.0286 (9)	0.0280 (8)	0.0357 (8)	0.0043 (7)	0.0025 (7)	−0.0013 (7)
C7	0.0302 (9)	0.0301 (8)	0.0322 (8)	0.0006 (7)	0.0058 (7)	−0.0049 (7)
C8	0.0450 (10)	0.0245 (8)	0.0263 (8)	0.0052 (7)	−0.0016 (7)	0.0013 (7)
C9	0.0342 (9)	0.0295 (9)	0.0333 (8)	−0.0038 (7)	0.0018 (7)	0.0037 (7)
C10	0.0316 (9)	0.0230 (8)	0.0410 (10)	−0.0010 (7)	0.0036 (7)	0.0014 (7)
C11	0.0313 (8)	0.0210 (8)	0.0292 (8)	−0.0005 (6)	−0.0016 (6)	−0.0021 (6)
N1	0.0304 (7)	0.0176 (6)	0.0265 (6)	0.0002 (5)	0.0006 (5)	−0.0028 (5)
O1	0.0967 (12)	0.0187 (6)	0.0397 (7)	−0.0012 (6)	−0.0094 (7)	−0.0021 (5)
O2	0.1445 (16)	0.0265 (7)	0.0423 (8)	−0.0036 (8)	−0.0356 (9)	−0.0043 (6)
O3	0.0547 (8)	0.0194 (6)	0.0454 (7)	0.0027 (5)	−0.0017 (6)	−0.0047 (5)
O4	0.0480 (8)	0.0300 (7)	0.0566 (8)	0.0078 (6)	−0.0163 (6)	0.0023 (6)
O5	0.0460 (8)	0.0381 (7)	0.0496 (8)	−0.0109 (6)	0.0039 (6)	−0.0131 (6)
O6	0.0340 (7)	0.0422 (8)	0.0649 (9)	−0.0070 (5)	0.0128 (6)	−0.0068 (6)

Geometric parameters (Å, °)

C1—C2	1.531 (2)	C7—C9	1.505 (2)
C1—C11	1.538 (2)	C7—N1	1.511 (2)
C1—C6	1.538 (2)	C7—H7A	0.9700
C1—N1	1.5455 (19)	C7—H7B	0.9700
C2—C3	1.535 (2)	C8—N1	1.515 (2)
C2—H2A	0.9700	C8—C10	1.525 (2)
C2—H2B	0.9700	C8—H8A	0.9700
C3—C4	1.507 (3)	C8—H8B	0.9700
C3—H3A	0.9700	C9—O5	1.213 (2)
C3—H3B	0.9700	C9—O6	1.300 (2)
C4—C5	1.505 (3)	C10—O4	1.258 (2)
C4—H4A	0.9700	C10—O3	1.2592 (19)
C4—H4B	0.9700	C11—O2	1.1818 (19)
C5—C6	1.524 (2)	C11—O1	1.2907 (19)
C5—H5A	0.9700	N1—H1N	0.891 (13)
C5—H5B	0.9700	O1—H1	0.8200
C6—H6A	0.9700	O6—H6	0.8200
C6—H6B	0.9700		
C2—C1—C11	114.54 (12)	C5—C6—H6B	109.0
C2—C1—C6	111.03 (12)	C1—C6—H6B	109.0
C11—C1—C6	109.96 (12)	H6A—C6—H6B	107.8
C2—C1—N1	107.09 (12)	C9—C7—N1	109.99 (13)
C11—C1—N1	105.89 (11)	C9—C7—H7A	109.7

C6—C1—N1	107.96 (12)	N1—C7—H7A	109.7
C1—C2—C3	113.75 (13)	C9—C7—H7B	109.7
C1—C2—H2A	108.8	N1—C7—H7B	109.7
C3—C2—H2A	108.8	H7A—C7—H7B	108.2
C1—C2—H2B	108.8	N1—C8—C10	111.82 (13)
C3—C2—H2B	108.8	N1—C8—H8A	109.3
H2A—C2—H2B	107.7	C10—C8—H8A	109.3
C4—C3—C2	112.78 (14)	N1—C8—H8B	109.3
C4—C3—H3A	109.0	C10—C8—H8B	109.3
C2—C3—H3A	109.0	H8A—C8—H8B	107.9
C4—C3—H3B	109.0	O5—C9—O6	122.20 (16)
C2—C3—H3B	109.0	O5—C9—C7	121.40 (15)
H3A—C3—H3B	107.8	O6—C9—C7	116.40 (15)
C5—C4—C3	109.95 (15)	O4—C10—O3	127.31 (15)
C5—C4—H4A	109.7	O4—C10—C8	115.64 (14)
C3—C4—H4A	109.7	O3—C10—C8	117.02 (14)
C5—C4—H4B	109.7	O2—C11—O1	122.84 (15)
C3—C4—H4B	109.7	O2—C11—C1	122.41 (14)
H4A—C4—H4B	108.2	O1—C11—C1	114.73 (13)
C4—C5—C6	111.77 (14)	C7—N1—C8	112.02 (12)
C4—C5—H5A	109.3	C7—N1—C1	112.91 (11)
C6—C5—H5A	109.3	C8—N1—C1	116.76 (12)
C4—C5—H5B	109.3	C7—N1—H1N	102.6 (12)
C6—C5—H5B	109.3	C8—N1—H1N	105.1 (11)
H5A—C5—H5B	107.9	C1—N1—H1N	105.9 (11)
C5—C6—C1	112.90 (14)	C11—O1—H1	109.5
C5—C6—H6A	109.0	C9—O6—H6	109.5
C1—C6—H6A	109.0		
C11—C1—C2—C3	−78.41 (17)	C6—C1—C11—O2	81.1 (2)
C6—C1—C2—C3	46.87 (18)	N1—C1—C11—O2	−35.3 (2)
N1—C1—C2—C3	164.50 (13)	C2—C1—C11—O1	25.70 (19)
C1—C2—C3—C4	−51.28 (19)	C6—C1—C11—O1	−100.14 (16)
C2—C3—C4—C5	55.67 (19)	N1—C1—C11—O1	143.46 (14)
C3—C4—C5—C6	−58.09 (19)	C9—C7—N1—C8	111.67 (15)
C4—C5—C6—C1	56.03 (19)	C9—C7—N1—C1	−114.05 (14)
C2—C1—C6—C5	−49.32 (18)	C10—C8—N1—C7	−124.25 (14)
C11—C1—C6—C5	78.49 (17)	C10—C8—N1—C1	103.36 (15)
N1—C1—C6—C5	−166.42 (12)	C2—C1—N1—C7	63.50 (15)
N1—C7—C9—O5	10.0 (2)	C11—C1—N1—C7	−59.14 (15)
N1—C7—C9—O6	−170.68 (14)	C6—C1—N1—C7	−176.88 (12)
N1—C8—C10—O4	177.01 (14)	C2—C1—N1—C8	−164.51 (12)
N1—C8—C10—O3	−1.4 (2)	C11—C1—N1—C8	72.85 (15)
C2—C1—C11—O2	−153.10 (18)	C6—C1—N1—C8	−44.89 (16)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O3^{i}	0.82	1.78	2.575 (2)	165

O6—H6···O4^{ii}	0.82	1.80	2.5823 (19)	158
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Symmetry codes: (i) *x*, *y*−1, *z*; (ii) *x*+1/2, −*y*+1/2, −*z*.

Fig. 1