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1,3-Bis(carboxymethyl)imidazolium triiodide 1-carboxylatomethyl-3-carboxymethylimidazolium

Jinling Miao,[a]* Chunhua Hu,[b] Haiyan Chen,[a] Guizhen Yuan[a] and Yong Nie[a]

[a]School of Chemistry and Chemical Engineering, University of Jinan, Jinan 250022, People's Republic of China, and [b]Department of Chemistry, New York University, 100 Washington Square East, New York, NY 10003-6688, USA
Correspondence e-mail: chm_miaojl@ujn.edu.cn

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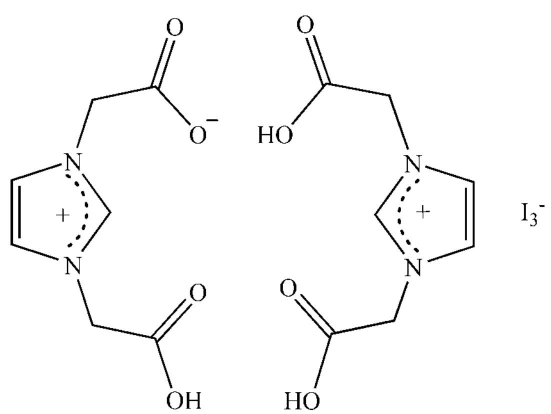
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.031; wR factor = 0.112; data-to-parameter ratio = 16.1.

In the title compound, $C_7H_9N_2O_4^+ \cdot I_3^- \cdot C_7H_8N_2O_4$, the two imidazolium units are hydrogen bonded through the carboxyl groups. The units are further linked *via* intermolecular O—H···O hydrogen bonding, resulting in a one-dimensional ladder-type structure. As a result, the two carboxy groups of each imidazolium unit adopt a *cis* configuration with respect to the imidazolium ring.

Related literature

For the preparation of 1,3-bis(carboxymethyl)imidazole, see: Kratochvíl *et al.* (1988); Fei *et al.* (2004); Barczynski *et al.* (2008). For its structure, see: Kratochvíl *et al.* (1988).



Experimental

Crystal data

$C_7H_9N_2O_4^+ \cdot I_3^- \cdot C_7H_8N_2O_4$
M_r = 750.02
Monoclinic, $C2/c$
a = 22.260 (3) Å
b = 10.1973 (17) Å
c = 10.1077 (17) Å
β = 92.209 (2)°
V = 2292.7 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.14 mm^{-1}
T = 298 K
0.49 × 0.44 × 0.40 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.237, T_{max} = 0.289 (expected range = 0.157–0.191)
6257 measured reflections
2248 independent reflections
1702 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.112
S = 1.03
2248 reflections
140 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.84 e Å^{-3}
$\Delta\rho_{min}$ = −0.99 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3O···O1^{i}	0.81 (8)	1.80 (8)	2.591 (6)	166 (9)
O2—H2O···O2^{ii}	1.224 (4)	1.224 (4)	2.449 (6)	179 (9)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); 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: BT2982).

References

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supplementary materials

Acta Cryst. (2009). E**65**, o1780 [doi:10.1107/S1600536809025239]

1,3-Bis(carboxymethyl)imidazolium triiodide 1-carboxylatomethyl-3-carboxymethylimidazolium

J. Miao, C. Hu, H. Chen, G. Yuan and Y. Nie

Comment

1,3-bis(carboxymethyl)imidazole was first prepared by the condensation reaction of formaldehyde, glyoxal and glycine (KratochvÍ *et al.*, 1988). Recently its synthesis by the reaction of alkyl haloacetate with imidazole has been reported (Fei *et al.*, 2004; Barczynski *et al.*, 2008). We have found that the reaction of imidazole with chloroacetic acid in the presence of NaOH as a base produces colorless 1,3-bis(carboxymethyl)imidazole, while the same reaction with iodoacetic acid affords the red title compound.

As shown in Fig. 1, two imidazolium units are hydrogen bonded through the carboxy groups. The presence of an I_3^- anion accounts for the neutral nature of the whole structure. The bond lengths of C4—O1 and C4—O2 are 1.231 (6), 1.259 (6) Å (table 1), respectively, which are between those for a C—O single bond and a C═O double bond. The C—N bond lengths on the rings are found to be within 1.316 (6)–1.384 (6) Å (Table 1), which are between those for a C—N single bond and a C═N double bond, suggesting charge delocalization on the planar imidazolium rings. The two imidazolium units are extended by intermolecular hydrogen bonding (O3-H3O—O1i, [i = x, y+1, z], 2.591 (6) Å) to generate a one-dimensional ladder-type structure along the *c* axis (Fig. 2). As a result of the hydrogen bonding, the two carboxy groups of each imidazolium unit adopt a *cis* configuration, while in the structure of 1,3-bis(carboxymethyl)imidazole (KratochvÍ *et al.*, 1988) a *trans* configuration has been found.

Experimental

To a solution of iodoacetic acid (9.314 g, 0.05 mol) in distilled water (25 ml), an aqueous solution (25 ml) of NaOH (2.020 g, 0.05 mol) was added, and followed by the addition of imidazole (2.020 g, 0.03 mol). The resulting colorless solution was heated to reflux during which the color gradually changed to yellow. The pH was adjusted using saturated NaOH solution once per 20 min., keeping in the range of 8–9, till no obvious change observed. The mixture was further refluxed for 30 min. and cooled, acidified with hydrochloric acid till pH 2–3, to give an orange-red solution. After 5 days, deep red crystals (yield 11.5% based on iodoacetic acid) were formed over evaporation. IR (KBr): ν = 3437, 3117, 1720, 1665, 1350, 1239, 890 cm $^{-1}$.

Refinement

H2O and H3O were located on the difference Fourier map. All other H-atoms were positioned geometrically and refined using a riding model with d(C—H) = 0.93 Å, U_{iso}=1.2U_{eq} (C) for aromatic, 0.97 Å, U_{iso} = 1.2U_{eq} (C) for CH_2 atoms.

Figures

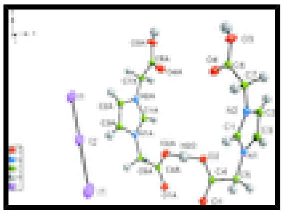

Fig. 1. The molecular structure, with atom labels and 25% probability thermal ellipsoids.

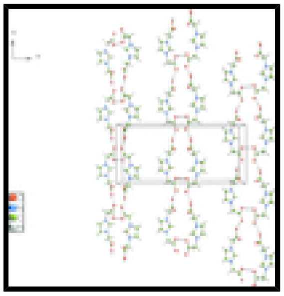

Fig. 2. The crystal packing diagram viewed along the *c* axis (only one layer shown), showing the hydrogen bonds as dotted lines; iodine atoms have been omitted for clarity

1,3-Bis(carboxymethyl)imidazolium triiodide 1-carboxylatomethyl-3-carboxymethylimidazolium

Crystal data

$C_7H_9N_2O_4^+ \cdot I_3^- \cdot C_7H_8N_2O_4$	F_{000} = 1408
M_r = 750.02	D_x = 2.173 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation, λ = 0.71073 Å
a = 22.260 (3) Å	Cell parameters from 2974 reflections
b = 10.1973 (17) Å	θ = 3.0–27.2°
c = 10.1077 (17) Å	μ = 4.14 mm^{-1}
β = 92.209 (2)°	*T* = 298 K
V = 2292.7 (6) Å^3	Plate, red
Z = 4	0.49 × 0.44 × 0.40 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	2248 independent reflections
Radiation source: fine-focus sealed tube	1702 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.031
T = 298 K	θ_{max} = 26.0°
φ and ω scans	θ_{min} = 1.8°
Absorption correction: multi-scan (SADABS; Bruker, 2001)	*h* = −18→27
T_{min} = 0.237, T_{max} = 0.289	*k* = −11→12
6257 measured reflections	*l* = −9→12

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.031$	$w = 1/[\sigma^2(F_o^2) + (0.0655P)^2 + 3.4411P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.112$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.03$	$\Delta\rho_{max} = 0.84$ e Å^{-3}
2248 reflections	$\Delta\rho_{min} = -0.98$ e Å^{-3}
140 parameters	Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0032 (2)
Secondary atom site location: difference Fourier map	

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

	x	y	z	$U_{iso}*/U_{eq}$
O1	0.46232 (19)	−0.1010 (4)	0.1047 (4)	0.0471 (10)
O2	0.45813 (18)	0.1069 (4)	0.1678 (4)	0.0467 (10)
O3	0.4311 (2)	0.7258 (4)	−0.0724 (5)	0.0608 (13)
O4	0.4461 (2)	0.5756 (4)	0.0836 (4)	0.0533 (11)
N1	0.38543 (17)	0.1836 (4)	−0.0301 (4)	0.0331 (9)
N2	0.38902 (19)	0.3887 (4)	−0.0756 (4)	0.0337 (9)
C1	0.4130 (2)	0.2736 (5)	−0.0999 (5)	0.0349 (11)
H1	0.4441	0.2581	−0.1566	0.042*
C2	0.3446 (2)	0.3728 (5)	0.0145 (6)	0.0415 (12)
H2	0.3207	0.4385	0.0487	0.050*
C3	0.3423 (3)	0.2448 (5)	0.0434 (6)	0.0431 (13)
H3	0.3167	0.2047	0.1016	0.052*
C4	0.4439 (2)	0.0124 (5)	0.0929 (5)	0.0357 (11)
C5	0.4008 (2)	0.0437 (5)	−0.0226 (5)	0.0362 (11)
H5A	0.4189	0.0175	−0.1043	0.043*
H5B	0.3643	−0.0069	−0.0140	0.043*
C6	0.4295 (2)	0.6072 (5)	−0.0261 (5)	0.0365 (11)
C7	0.4056 (3)	0.5151 (5)	−0.1311 (5)	0.0381 (12)
H7A	0.3706	0.5541	−0.1757	0.046*
H7B	0.4359	0.5019	−0.1962	0.046*
I1	0.228024 (19)	0.42974 (5)	0.27861 (4)	0.0609 (2)
I2	0.2500	0.2500	0.5000	0.0519 (2)
H2O	0.5000	0.106 (9)	0.2500	0.08 (3)*
H3O	0.435 (4)	0.780 (7)	−0.014 (9)	0.07 (2)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.054 (2)	0.037 (2)	0.049 (2)	0.0015 (17)	−0.0163 (19)	−0.0009 (17)
O2	0.052 (2)	0.044 (2)	0.042 (2)	0.0067 (17)	−0.0205 (18)	−0.0129 (17)
O3	0.094 (4)	0.038 (2)	0.048 (3)	−0.016 (2)	−0.026 (2)	0.009 (2)
O4	0.078 (3)	0.051 (2)	0.030 (2)	−0.0123 (19)	−0.014 (2)	0.0074 (17)
N1	0.030 (2)	0.035 (2)	0.034 (2)	0.0008 (17)	−0.0065 (17)	−0.0041 (18)
N2	0.038 (2)	0.040 (2)	0.023 (2)	−0.0028 (18)	−0.0028 (17)	−0.0004 (17)
C1	0.034 (3)	0.040 (3)	0.031 (3)	0.002 (2)	0.001 (2)	−0.005 (2)
C2	0.036 (3)	0.046 (3)	0.043 (3)	0.003 (2)	0.010 (2)	0.000 (2)
C3	0.038 (3)	0.049 (3)	0.044 (3)	−0.002 (2)	0.012 (2)	0.000 (2)
C4	0.032 (3)	0.042 (3)	0.033 (3)	−0.005 (2)	−0.004 (2)	−0.001 (2)
C5	0.035 (3)	0.035 (3)	0.038 (3)	0.000 (2)	−0.010 (2)	−0.005 (2)
C6	0.036 (3)	0.044 (3)	0.030 (3)	−0.001 (2)	−0.001 (2)	0.006 (2)
C7	0.050 (3)	0.038 (3)	0.025 (2)	−0.003 (2)	−0.004 (2)	0.002 (2)
I1	0.0478 (3)	0.0875 (4)	0.0468 (3)	0.0116 (2)	−0.00472 (19)	−0.0100 (2)
I2	0.0379 (3)	0.0668 (4)	0.0508 (4)	0.0012 (2)	−0.0021 (2)	−0.0218 (3)

Geometric parameters (Å, °)

O1—C4	1.231 (6)	C1—H1	0.9300
O2—C4	1.259 (6)	C2—C3	1.339 (7)
O2—H2O	1.224 (4)	C2—H2	0.9300
O3—C6	1.297 (6)	C3—H3	0.9300
O3—H3O	0.81 (8)	C4—C5	1.516 (7)
O4—C6	1.199 (7)	C5—H5A	0.9700
N1—C1	1.323 (6)	C5—H5B	0.9700
N1—C3	1.384 (6)	C6—C7	1.500 (7)
N1—C5	1.467 (6)	C7—H7A	0.9700
N2—C1	1.316 (6)	C7—H7B	0.9700
N2—C2	1.380 (6)	I1—I2	2.9192 (6)
N2—C7	1.459 (6)	I2—I1^{i}	2.9192 (6)
C4—O2—H2O	125 (4)	O1—C4—C5	118.1 (5)
C6—O3—H3O	112 (6)	O2—C4—C5	116.0 (5)
C1—N1—C3	108.5 (4)	N1—C5—C4	112.6 (4)
C1—N1—C5	126.2 (4)	N1—C5—H5A	109.1
C3—N1—C5	125.1 (4)	C4—C5—H5A	109.1
C1—N2—C2	108.9 (4)	N1—C5—H5B	109.1
C1—N2—C7	127.3 (4)	C4—C5—H5B	109.1
C2—N2—C7	123.7 (4)	H5A—C5—H5B	107.8
N2—C1—N1	108.7 (4)	O4—C6—O3	124.9 (5)
N2—C1—H1	125.7	O4—C6—C7	125.0 (5)
N1—C1—H1	125.7	O3—C6—C7	110.0 (5)
C3—C2—N2	107.0 (4)	N2—C7—C6	111.7 (4)
C3—C2—H2	126.5	N2—C7—H7A	109.3
N2—C2—H2	126.5	C6—C7—H7A	109.3

C2—C3—N1	106.9 (5)	N2—C7—H7B	109.3
C2—C3—H3	126.6	C6—C7—H7B	109.3
N1—C3—H3	126.6	H7A—C7—H7B	107.9
O1—C4—O2	125.8 (5)	I1^{i}—I2—I1	180.0

Symmetry codes: (i) $-x+1/2$, $-y+1/2$, $-z+1$.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3O···O1^{ii}	0.81 (8)	1.80 (8)	2.591 (6)	166 (9)
O2—H2O···O2^{iii}	1.224 (4)	1.224 (4)	2.449 (6)	179 (9)

Symmetry codes: (ii) x, $y+1$, z; (iii) $-x+1$, y, $-z+1/2$.

Fig. 1

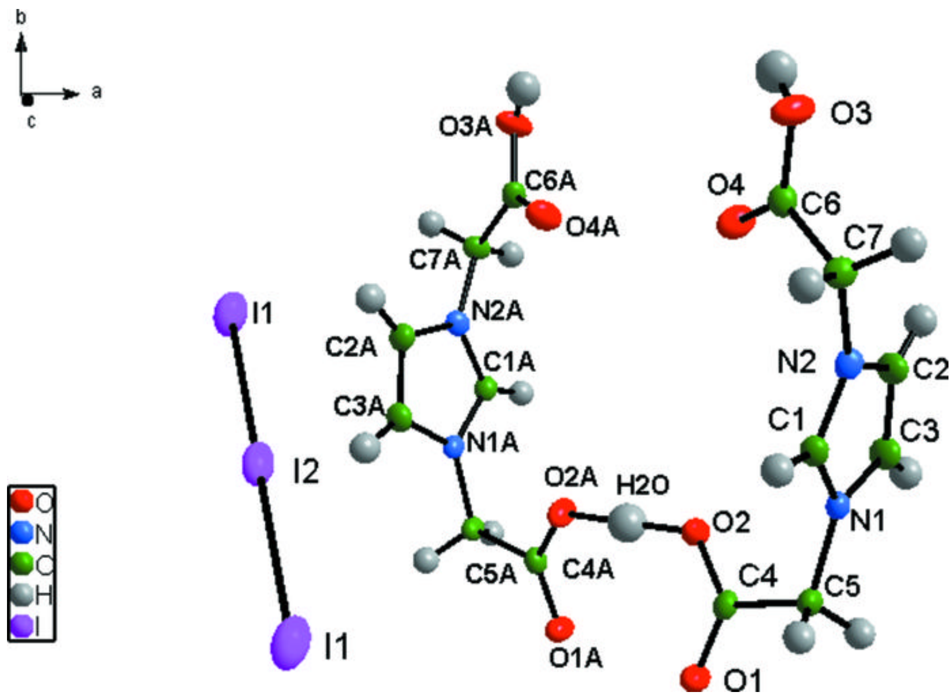
b
a
c
O3A
C6A
O4A
C7A
N2A
C2A
C1A
C3A
N1A
O2A
H2O
O2
C4A
C5A
O1A
C4
C5
O1
O3
O4
C6
C7
N2
C2
C1
C3
N1
I1
I2
I1
O
N
C
H
I

Fig. 2