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Poly[[μ_{10}-2,3-bis(carboxymethyl)butanedioato]disodium]

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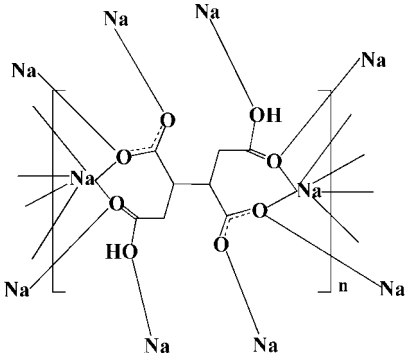
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.111; data-to-parameter ratio = 12.8.

The asymmetric unit of the title compound, $[Na_2(C_8H_8O_8)]_n$, contains one Na^+ ion and half of a 2,3-bis(carboxymethyl)butanedioate (H_2BTC^{2-}) dianion, which lies on a center of symmetry. The dianion exhibits a μ_{10}-bridging mode. Each Na atom lies in a NaO_6 octahedron defined by six O atoms from five dianions. Adjacent NaO_6 octahedra share a common O—O edge, generating a bioctahedron; adjacent bioctahedra are O—O edge-connected to one another, building up a chain along [001]. The chains are connected by adjacent H_2BTC^{2-} anions into a three-dimensional framework. The structure is further stabilized by O—H···O hydrogen bonds.

Related literature

For related structures, see: Delgado *et al.* (2007); Liu *et al.* (2008); Wang *et al.* (2005); Zheng *et al.* (2004); Zhu & Zheng (2010).



Experimental

Crystal data

$[Na_2(C_8H_8O_8)]$ M_r = 278.12
Orthorhombic, *Pbcn*
a = 8.9053 (18) Å
b = 8.6395 (17) Å
c = 12.527 (3) Å
V = 963.8 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 293 K
0.44 × 0.36 × 0.32 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.900, T_{max} = 0.925
8610 measured reflections
1097 independent reflections
1000 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.111
S = 1.10
1097 reflections
86 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2C···O4^{i}	0.85 (2)	1.67 (3)	2.5097 (18)	177 (2)

Symmetry code: (i) $-x+\frac{5}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); 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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG5044).

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supporting information

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Poly[[μ_{10}-2,3-bis(carboxymethyl)butanedioato]disodium]

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S1. Comment

Recently, the aliphatic multi-carboxylic acids have attractived considerable attention due to both its conformational flexibility and a variety of coordination fashions (Wang *et al.*, 2005; Zheng *et al.*, 2004).The butane-1,2,3,4-tetracarboxylic acid (H_4BTC) ligand possesses four ionizable protons that can be removed gradually to form a series of deprotonated anions such as H_3BTC^-, H_2BTC^{2-}, $HBTC^{3-}$, BTC^{4-}, which have allowed the preparation of a variety of complexes with differents metals (Delgado *et al.*, 2007; Liu *et al.*, 2008; Zhu *et al.*, 2010). In this contribution, we report the synthesis and crystal structure of the title compound.

The asymmetric unit of the title compound contains one Na^+ ion and half a H_2BTC^{2-} anion(Figure 1). The H_2BTC^{2-} ligand is diprotonated, which is crystallographically imposed by symmetry of center with inversion centers at the midpoints of the central C3—C3^i bond with the Wyckoff 4*b* site. Each H_2BTC^{2-} anions coordinate ten sodium ions through eight carboxyl oxygen atoms. The carboxylate group and carboxylic group all coordinates to two metal atoms in a *syn*/*anti* $\mu_2\eta^2$ bridging fashion, and two seven-membered chelating rings are concomitantly formed. Each Na atom is in a distorted octahedra NaO_6 gemetry defined by six O atoms from five H_2BTC^{2-} ligands, the Na—O contact distances are all within the normal ranges. The adjacent two NaO_6 octahedra are fused *via* common edge O1—O1 and O3—O3, generating a one-dimensional sodium-oxide chains (Figure 2), and the resulting chains are further interlinked by H_2BTC^{2-} anions into three-dimensional frameworks (Figure 3).

S2. Experimental

All chemicals were obtained from commerical sources and were used as obtained. NaOH (0.079 g, 1.98 mmol) was added to a stirred mixture solution of butane-1,2,3,4-tetracarboxylic acid (0.1173 g, 0.50 mmol) in 10 ml H_2O and 10 ml me thanol, and the resulting mixture was stirred for 5 min. Colorless crystals were obtained from the solution (pH = 7.13) after standing at room temperature for five weeks.

S3. Refinement

H atoms bonded to C atoms were palced in geometrically calculated position and were refined using a riding model, with U_{iso}(H) = 1.2 U_{eq}(C). H atoms attached to O atoms were found in a difference Fourier synthesis and refined with the O—H distance restranied to 0.83 (1) Å.

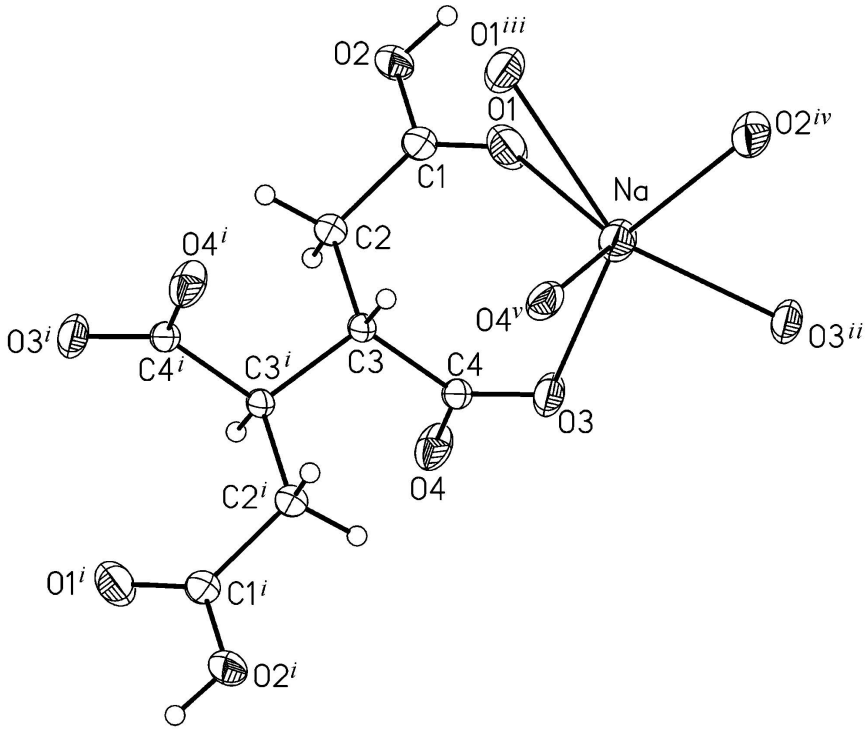

Figure 1

The content of asymmetric unit showing the atomic numbering and 45% probability dispalcement ellipsoids.[Symmetry codes: (i) -*x* + 2, -*y* + 1, -*z* + 1. (ii) -*x* + 2, -*y*, -*z* + 1. (iii) -*x* + 2, *y*, -*z* + 1.5. (iv) *x* - 1/2,*y* - 1/2, -*z* + 1.5. (v) *x* - 1/2, -*y* + 1/2, -*z* + 1.]

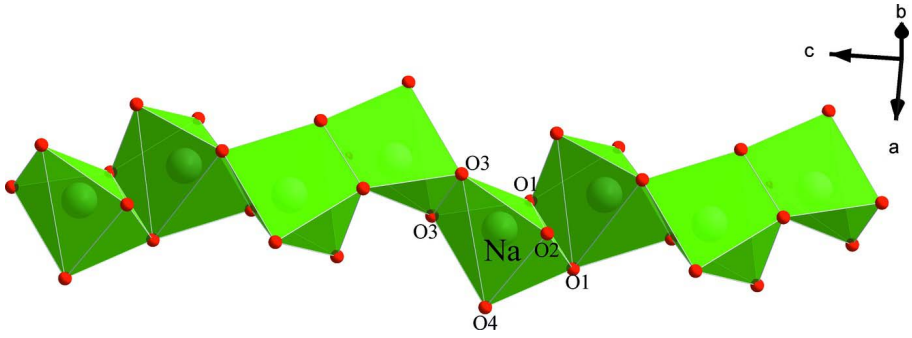

Figure 2

The one-dimensional sodium-oxide chains with the common edges O1—O1 and O3—O3.

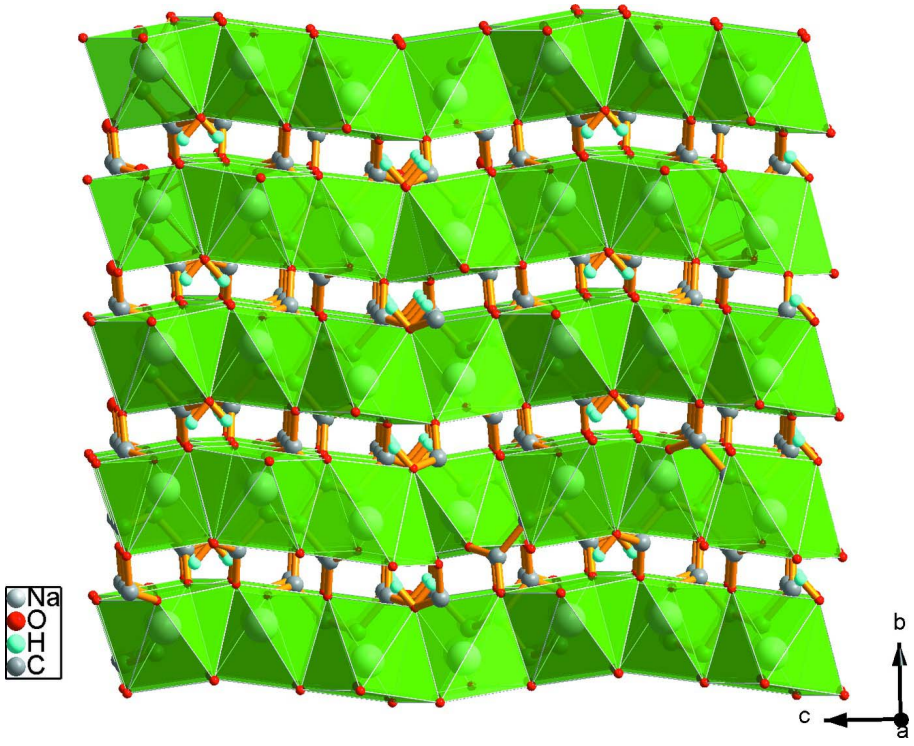

Figure 3

The three-dimensional metal-organic framework in the title compound.

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Crystal data

$[Na_2(C_8H_8O_8)]$
$M_r = 278.12$
Orthorhombic, *Pbcn*
Hall symbol: -P 2n 2ab
$a = 8.9053$ (18) Å
$b = 8.6395$ (17) Å
$c = 12.527$ (3) Å
$V = 963.8$ (3) Å^3
$Z = 4$
$F(000) = 568$
$D_x = 1.917$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7116 reflections
$\theta = 3.3$–$27.4°$
$\mu = 0.24$ mm^{-1}
$T = 293$ K
Block, colorless
0.44 × 0.36 × 0.32 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 0 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.900$, $T_{max} = 0.925$
8610 measured reflections
1097 independent reflections
1000 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 27.4°$, $\theta_{min} = 3.3°$
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.111$
$S = 1.10$
1097 reflections
86 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0658P)^2 + 0.5154P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Na	0.91916 (8)	0.06655 (8)	0.62976 (5)	0.0292 (2)
O1	1.12521 (17)	0.21959 (14)	0.69249 (10)	0.0391 (4)
O2	1.30500 (16)	0.37558 (13)	0.75029 (10)	0.0325 (3)
C1	1.19167 (19)	0.34236 (18)	0.68752 (12)	0.0243 (3)
C2	1.15019 (18)	0.46906 (17)	0.61037 (11)	0.0219 (3)
H2A	1.2386	0.4963	0.5692	0.026*
H2B	1.1207	0.5598	0.6508	0.026*
C3	1.02287 (16)	0.42768 (14)	0.53277 (10)	0.0161 (3)
H3A	0.9358	0.3927	0.5741	0.019*
C4	1.07185 (16)	0.29671 (16)	0.45770 (11)	0.0177 (3)
O3	1.01257 (15)	0.16760 (12)	0.46431 (9)	0.0300 (3)
O4	1.17325 (15)	0.33112 (14)	0.39020 (10)	0.0300 (3)
H2C	1.310 (4)	0.308 (3)	0.7989 (19)	0.088 (11)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Na	0.0336 (4)	0.0244 (4)	0.0296 (4)	−0.0038 (3)	0.0023 (3)	−0.0031 (2)
O1	0.0481 (8)	0.0278 (7)	0.0415 (7)	−0.0062 (6)	−0.0172 (6)	0.0104 (5)
O2	0.0432 (7)	0.0262 (6)	0.0282 (6)	0.0011 (5)	−0.0177 (5)	0.0041 (5)
C1	0.0305 (8)	0.0216 (7)	0.0208 (7)	0.0046 (6)	−0.0054 (6)	−0.0007 (5)
C2	0.0278 (7)	0.0185 (7)	0.0194 (7)	0.0020 (6)	−0.0054 (6)	0.0003 (5)
C3	0.0208 (7)	0.0137 (6)	0.0138 (6)	0.0032 (5)	0.0005 (5)	0.0002 (5)
C4	0.0212 (7)	0.0160 (6)	0.0159 (6)	0.0039 (5)	−0.0012 (5)	−0.0008 (5)

O3	0.0448 (7)	0.0166 (5)	0.0286 (6)	−0.0050 (5)	0.0081 (5)	−0.0041 (4)
O4	0.0340 (6)	0.0247 (6)	0.0314 (6)	−0.0026 (5)	0.0148 (5)	−0.0082 (5)

Geometric parameters (Å, º)

Na—O4^{i}	2.3748 (15)	O2—H2C	0.843 (10)
Na—O1	2.3943 (15)	C1—C2	1.506 (2)
Na—O3	2.3978 (13)	C2—C3	1.536 (2)
Na—O3^{ii}	2.4188 (13)	C2—H2A	0.9700
Na—O2^{iii}	2.4522 (14)	C2—H2B	0.9700
Na—O1^{iv}	2.6196 (15)	C3—C4	1.5346 (18)
Na—Na^{iv}	3.3388 (14)	C3—C3^{vi}	1.550 (2)
Na—Na^{ii}	3.7369 (14)	C3—H3A	0.9800
O1—C1	1.216 (2)	C4—O3	1.2368 (18)
O1—Na^{iv}	2.6196 (15)	C4—O4	1.2723 (19)
O2—C1	1.311 (2)	O3—Na^{ii}	2.4188 (13)
O2—Na^{v}	2.4522 (14)	O4—Na^{vii}	2.3748 (15)
O4^{i}—Na—O1	122.39 (5)	C1—O1—Na	146.90 (11)
O4^{i}—Na—O3	95.36 (5)	C1—O1—Na^{iv}	123.86 (11)
O1—Na—O3	79.44 (5)	Na—O1—Na^{iv}	83.38 (5)
O4^{i}—Na—O3^{ii}	119.46 (5)	C1—O2—Na^{v}	146.49 (11)
O1—Na—O3^{ii}	115.41 (6)	C1—O2—H2C	109 (2)
O3—Na—O3^{ii}	78.24 (5)	Na^{v}—O2—H2C	90 (2)
O4^{i}—Na—O2^{iii}	86.14 (5)	O1—C1—O2	122.32 (14)
O1—Na—O2^{iii}	119.22 (5)	O1—C1—C2	123.18 (14)
O3—Na—O2^{iii}	156.69 (5)	O2—C1—C2	114.49 (14)
O3^{ii}—Na—O2^{iii}	80.80 (5)	C1—C2—C3	114.71 (13)
O4^{i}—Na—O1^{iv}	76.26 (5)	C1—C2—H2A	108.6
O1—Na—O1^{iv}	63.76 (7)	C3—C2—H2A	108.6
O3—Na—O1^{iv}	127.10 (5)	C1—C2—H2B	108.6
O3^{ii}—Na—O1^{iv}	150.95 (5)	C3—C2—H2B	108.6
O2^{iii}—Na—O1^{iv}	75.89 (5)	H2A—C2—H2B	107.6
O4^{i}—Na—Na^{iv}	119.52 (4)	C4—C3—C2	110.47 (11)
O1—Na—Na^{iv}	51.20 (4)	C4—C3—C3^{vi}	110.16 (13)
O3—Na—Na^{iv}	129.07 (5)	C2—C3—C3^{vi}	109.98 (14)
O3^{ii}—Na—Na^{iv}	109.34 (4)	C4—C3—H3A	108.7
O2^{iii}—Na—Na^{iv}	68.03 (4)	C2—C3—H3A	108.7
O1^{iv}—Na—Na^{iv}	45.42 (3)	C3^{vi}—C3—H3A	108.7
O4^{i}—Na—Na^{ii}	112.22 (4)	O3—C4—O4	123.93 (13)
O1—Na—Na^{ii}	99.22 (5)	O3—C4—C3	120.17 (12)
O3—Na—Na^{ii}	39.32 (3)	O4—C4—C3	115.89 (12)
O3^{ii}—Na—Na^{ii}	38.92 (3)	C4—O3—Na	122.30 (9)
O2^{iii}—Na—Na^{ii}	119.06 (4)	C4—O3—Na^{ii}	127.90 (10)
O1^{iv}—Na—Na^{ii}	162.35 (5)	Na—O3—Na^{ii}	101.76 (5)
Na^{iv}—Na—Na^{ii}	128.23 (4)	C4—O4—Na^{vii}	144.24 (11)

Symmetry codes: (i) $x-1/2, -y+1/2, -z+1$; (ii) $-x+2, -y, -z+1$; (iii) $x-1/2, y-1/2, -z+3/2$; (iv) $-x+2, y, -z+3/2$; (v) $x+1/2, y+1/2, -z+3/2$; (vi) $-x+2, -y+1, -z+1$; (vii) $x+1/2, -y+1/2, -z+1$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*C*···O4^{viii}	0.85 (2)	1.67 (3)	2.5097 (18)	177 (2)

Symmetry code: (viii) −*x*+5/2, −*y*+1/2, *z*+1/2.