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Bis(2-methyllactato)borate tetrahydrate

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Keywords: crystal structure; organic material; borate; O—H···O hydrogen bonds.

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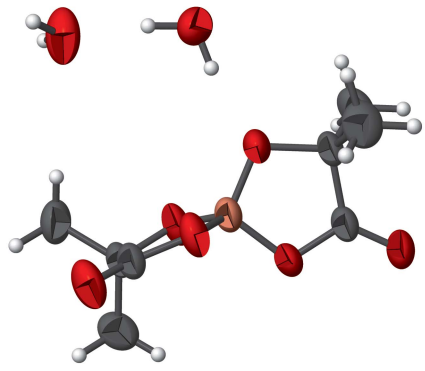
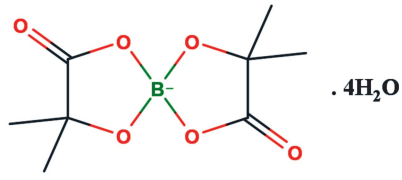
Structural data: full structural data are available from iucrdata.iucr.org

The asymmetric unit of the title compound (systematic name: 3,3,8,8-tetramethyl-1,4,6,9-tetraoxa-λ^4-boraspiro[4.4]nonane-2,7-dione tetrahydrate), $C_8H_{12}BO_6{\cdot}4H_2O$, consists of half a bis(2-methyllactato)borate molecule and two water molecules of solvation. In the crystal, O—H···O hydrogen bonds link the components into a three-dimensional network.

3D view



Chemical scheme



Structure description

Allen *et al.* (2012) have reported the structure of lithium bis(2-methyllactato)borate monohydrate. We report here the growth and structural analysis of bis(2-methyllactato)borate tetrahydrate, prepared by the slow evaporation method. Whereas the lithium salt crystallizes in the space group *Pbca* with $Z = 8$, the title compound crystallizes in the space group $P2_12_12$ with $Z = 2$.

The asymmetric unit of the title compound consists of a (2-methyllactato)borate molecule and two water molecules (Fig. 1). The five-membered ring O1/C1/C2/O3/B1 adopts an envelope form on O3 atom [puckering parameters Q_2 = 0.104 (2) Å, φ_2 = 288.5 (11)°] and B1/O1^i/C1^i/C2^i/O3^i adopts an envelope form on O3^i atom [puckering parameters Q_2 = 0.104 (2) Å, φ_2 = 144.5 (11)°]. The dihedral angle between the above two five-membered rings is 89.83 (12)°. In the crystal, O—H···O hydrogen bonds (Table 1) link the components into a three-dimensional network, as shown in Fig. 2.

Synthesis and crystallization

The title compound was synthesized by reacting 2-methyllactic acid and boric acid (molar ratio 2:1) in double-distilled water. Slow evaporation of the solvent yielded good quality crystals in a period of about four months.

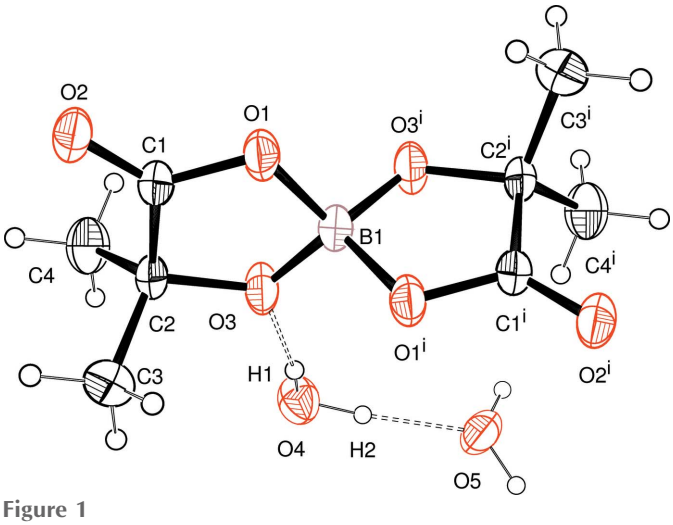


Figure 1
A view of the asymmetric unit of the title compound showing the atom numbering with displacement ellipsoids drawn at the 30% probability level. Symmetry code: (i) $-x+1, -y+1, z$.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors thank the Sophisticated Analytical Instrument Facility (SAIF), Indian Institute of Technology Madras (IITM), Chennai 600 036, Tamilnadu, India, for the single-crystal X-ray diffraction data.

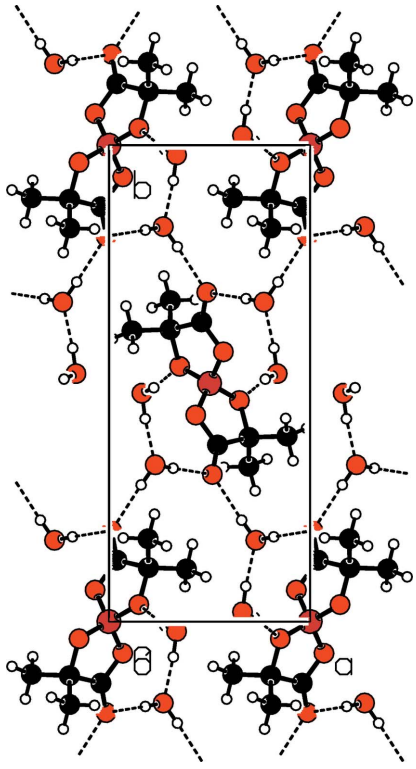


Figure 2
Packing diagram of the title compound viewed along the *c* axis.

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H1···O3	0.90 (2)	1.77 (2)	2.645 (2)	165 (4)
O5—H3···O2^{i}	0.88 (2)	1.92 (2)	2.805 (3)	179 (3)
O5—H4···O2^{ii}	0.87 (2)	1.92 (2)	2.795 (2)	175 (4)
O4—H2···O5	0.92 (2)	1.67 (2)	2.591 (2)	173 (4)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_8H_{12}BO_6\cdot4H_2O$
M_r	287.05
Crystal system, space group	Orthorhombic, $P2_12_12$
Temperature (K)	296
a, b, c (Å)	7.0809 (1), 16.7912 (3), 6.5001 (1)
V (Å^3)	772.84 (2)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.11
Crystal size (mm)	0.15 × 0.15 × 0.10
Data collection	
Diffractometer	Bruker Kappa APEX3 CMOS
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.568, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	18957, 1680, 1585
R_{int}	0.052
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.639
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.036, 0.094, 1.08
No. of reflections	1680
No. of parameters	104
No. of restraints	6
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.21, −0.12
Absolute structure	Flack x determined using 587 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons et al., 2013)
Absolute structure parameter	0.4 (4)

Computer programs: *APEX3*, *SAINT* and *XPREP* (Bruker, 2016), *SHELXT2018* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

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full crystallographic data

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3,3,8,8-Tetramethyl-1,4,6,9-tetraoxa-λ^4-boraspiro[4.4]nonane-2,7-dione tetrahydrate

Crystal data

$C_8H_{12}BO_6{\cdot}4H_2O$
M_r = 287.05
Orthorhombic, $P2_12_12$
a = 7.0809 (1) Å
b = 16.7912 (3) Å
c = 6.5001 (1) Å
V = 772.84 (2) Å^3
Z = 2
F(000) = 306
D_x = 1.234 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6304 reflections
θ = 3.1–30.4°
μ = 0.11 mm^{-1}
T = 296 K
Block, colourless
0.15 × 0.15 × 0.10 mm

Data collection

Bruker Kappa APEX3 CMOS diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scan
Absorption correction: multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min} = 0.568, T_{max} = 0.746
18957 measured reflections
1680 independent reflections
1585 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 27.0°, θ_{min} = 3.8°
h = −9→9
k = −21→21
l = −8→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.094
S = 1.08
1680 reflections
104 parameters
6 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0444P)^2 + 0.1071P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Extinction correction: SHELXL2018 (Sheldrick 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.26 (3)
Absolute structure: Flack x determined using 587 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons et al., 2013)
Absolute structure parameter: 0.4 (4)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
B1	0.500000	0.500000	0.6216 (5)	0.0446 (7)
C1	0.4604 (3)	0.62908 (10)	0.7335 (3)	0.0450 (5)
C2	0.3039 (3)	0.61335 (11)	0.5802 (3)	0.0451 (5)
C3	0.1148 (3)	0.61058 (17)	0.6908 (5)	0.0718 (7)
H3A	0.085017	0.662541	0.743128	0.108*
H3B	0.018307	0.594026	0.596294	0.108*
H3C	0.121754	0.573415	0.802770	0.108*
C4	0.3052 (5)	0.67396 (15)	0.4066 (4)	0.0705 (7)
H4A	0.273454	0.725527	0.460141	0.106*
H4B	0.428708	0.675832	0.345758	0.106*
H4C	0.214397	0.658807	0.304184	0.106*
O1	0.5687 (2)	0.56635 (8)	0.7558 (3)	0.0529 (4)
O2	0.4861 (3)	0.69107 (8)	0.8287 (3)	0.0627 (5)
O3	0.3516 (2)	0.53588 (8)	0.5003 (3)	0.0550 (4)
O4	0.1638 (3)	0.47942 (11)	0.1802 (3)	0.0664 (5)
O5	0.2299 (3)	0.32920 (10)	0.1250 (4)	0.0780 (6)
H1	0.223 (6)	0.491 (2)	0.299 (5)	0.124 (14)*
H2	0.180 (6)	0.4259 (15)	0.153 (7)	0.132 (16)*
H3	0.319 (4)	0.322 (2)	0.032 (5)	0.103 (12)*
H4	0.160 (5)	0.2871 (18)	0.146 (6)	0.109 (12)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
B1	0.0511 (17)	0.0316 (13)	0.0511 (16)	0.0068 (12)	0.000	0.000
C1	0.0458 (10)	0.0308 (8)	0.0584 (10)	0.0052 (7)	0.0008 (8)	0.0009 (7)
C2	0.0465 (10)	0.0298 (8)	0.0589 (11)	0.0086 (7)	−0.0018 (9)	0.0000 (8)
C3	0.0498 (13)	0.0664 (15)	0.0994 (19)	0.0000 (11)	0.0100 (13)	−0.0073 (14)
C4	0.0858 (18)	0.0534 (13)	0.0721 (15)	0.0146 (12)	−0.0064 (14)	0.0165 (11)
O1	0.0551 (8)	0.0353 (7)	0.0683 (9)	0.0125 (6)	−0.0147 (7)	−0.0036 (6)
O2	0.0651 (10)	0.0360 (7)	0.0870 (11)	0.0082 (7)	−0.0124 (9)	−0.0131 (7)
O3	0.0640 (9)	0.0370 (7)	0.0640 (9)	0.0160 (6)	−0.0158 (7)	−0.0091 (7)
O4	0.0687 (11)	0.0631 (11)	0.0673 (10)	0.0098 (8)	−0.0077 (8)	−0.0156 (8)
O5	0.0820 (13)	0.0421 (9)	0.1098 (16)	−0.0100 (8)	0.0335 (12)	−0.0042 (9)

Geometric parameters (Å, º)

B1—O3^{i}	1.445 (2)	C3—H3A	0.9600
B1—O3	1.445 (2)	C3—H3B	0.9600
B1—O1	1.496 (2)	C3—H3C	0.9600
B1—O1^{i}	1.496 (2)	C4—H4A	0.9600
C1—O2	1.225 (2)	C4—H4B	0.9600
C1—O1	1.311 (2)	C4—H4C	0.9600
C1—C2	1.513 (3)	O4—H1	0.90 (2)
C2—O3	1.441 (2)	O4—H2	0.92 (2)

C2—C4	1.520 (3)	O5—H3	0.88 (2)
C2—C3	1.521 (3)	O5—H4	0.87 (2)
O3^{i}—B1—O3	113.9 (2)	C2—C3—H3B	109.5
O3^{i}—B1—O1	113.09 (9)	H3A—C3—H3B	109.5
O3—B1—O1	104.13 (7)	C2—C3—H3C	109.5
O3^{i}—B1—O1^{i}	104.13 (7)	H3A—C3—H3C	109.5
O3—B1—O1^{i}	113.09 (9)	H3B—C3—H3C	109.5
O1—B1—O1^{i}	108.7 (2)	C2—C4—H4A	109.5
O2—C1—O1	122.67 (19)	C2—C4—H4B	109.5
O2—C1—C2	126.17 (17)	H4A—C4—H4B	109.5
O1—C1—C2	111.17 (16)	C2—C4—H4C	109.5
O3—C2—C1	102.92 (15)	H4A—C4—H4C	109.5
O3—C2—C4	109.59 (18)	H4B—C4—H4C	109.5
C1—C2—C4	111.57 (19)	C1—O1—B1	110.08 (14)
O3—C2—C3	110.44 (18)	C2—O3—B1	110.48 (14)
C1—C2—C3	109.80 (18)	H1—O4—H2	108 (3)
C4—C2—C3	112.1 (2)	H3—O5—H4	114 (3)
C2—C3—H3A	109.5		
O2—C1—C2—O3	174.2 (2)	O3—B1—O1—C1	6.8 (2)
O1—C1—C2—O3	−6.5 (2)	O1^{i}—B1—O1—C1	−114.04 (16)
O2—C1—C2—C4	56.7 (3)	C1—C2—O3—B1	10.8 (2)
O1—C1—C2—C4	−123.9 (2)	C4—C2—O3—B1	129.6 (2)
O2—C1—C2—C3	−68.2 (3)	C3—C2—O3—B1	−106.4 (2)
O1—C1—C2—C3	111.1 (2)	O3^{i}—B1—O3—C2	−134.64 (16)
O2—C1—O1—B1	179.2 (2)	O1—B1—O3—C2	−11.0 (2)
C2—C1—O1—B1	−0.2 (2)	O1^{i}—B1—O3—C2	106.76 (18)
O3^{i}—B1—O1—C1	130.86 (18)		

Symmetry code: (i) −*x*+1, −*y*+1, *z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H1···O3	0.90 (2)	1.77 (2)	2.645 (2)	165 (4)
O5—H3···O2^{ii}	0.88 (2)	1.92 (2)	2.805 (3)	179 (3)
O5—H4···O2^{iii}	0.87 (2)	1.92 (2)	2.795 (2)	175 (4)
O4—H2···O5	0.92 (2)	1.67 (2)	2.591 (2)	173 (4)

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