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Hexaaquamagnesium bis{4-[(5-bromo-2-hydroxybenzylidene)amino]benzenesulfonate} dihydrate

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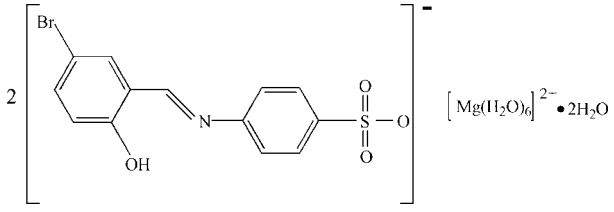
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.007 Å; R factor = 0.057; wR factor = 0.131; data-to-parameter ratio = 15.5.

In the title hydrated molecular salt, $[Mg(H_2O)_6](C_{13}H_9BrNO_4S)_2{\cdot}2H_2O$, the Mg^{2+} ion (site symmetry $\overline{1}$) adopts a near regular MgO_6 octahedral coordination geometry. In the anion, the dihedral angle between the aromatic rings is 2.5 (2)° and an intramolecular O—H···N hydrogen bond generates an S(6) ring. In the crystal, the components are linked by O—H···O and O—H···Br hydrogen bonds.

Related literature

For background to Schiff bases as ligands, see: Tai *et al.* (2003).



Experimental

Crystal data

$[Mg(H_2O)_6](C_{13}H_9BrNO_4S)_2{\cdot}2H_2O$
M_r = 878.80
Monoclinic, $P2_1/c$
a = 18.7737 (14) Å
b = 6.2837 (5) Å
c = 15.7591 (12) Å
β = 108.668 (1)°
V = 1761.3 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.51 mm^{-1}
T = 291 K
0.30 × 0.26 × 0.24 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.48, T_{max} = 0.55
9144 measured reflections
3446 independent reflections
2473 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.131
S = 1.03
3446 reflections
223 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

Table 1
Selected bond lengths (Å).

Mg1—O12	2.031 (3)	Mg1—O13	2.077 (3)
Mg1—O11	2.065 (3)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···N1	0.96	1.73	2.570 (5)	144
O5—H5D···O3^{i}	0.85	2.00	2.854 (4)	180
O5—H5A···O4^{ii}	0.85	2.08	2.892 (4)	160
O11—H11A···Br1^{iii}	0.96	2.60	3.539 (3)	166
O11—H11C···O3^{ii}	0.96	1.94	2.710 (4)	136
O12—H12A···O2^{iv}	0.96	2.04	2.747 (4)	129
O12—H12B···O5	0.96	1.90	2.729 (4)	143
O13—H13C···O4^{v}	0.96	1.88	2.763 (4)	152

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: HB5780).

References

Bruker (2000). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tai, X.-S., Yin, X.-H., Tan, M.-Y. & Li, Y.-Z. (2003). *Acta Cryst.* E**59**, o681–o682.

supporting information

Acta Cryst. (2011). E**67**, m132 [doi:10.1107/S1600536810053717]

Hexaaquamagnesium bis{4-[(5-bromo-2-hydroxybenzylidene)amino]benzenesulfonate} dihydrate

Xi-Shi Tai

S1. Experimental

1 mmol of magnesium perchlorate was added to a solution of 5-bromosalicylaldehyde-4-aminobenzene sulfonic acid (1 mmol) in 10 ml of 95% ethanol. The mixture was stirred for 3 h at refluxing temperature. Evaporating some ethanol, clear blocks of (I) were obtained after two weeks.

S2. Refinement

The H atoms were placed geometrically (C—H = 0.93–0.96 Å, O—H = 0.82 Å, N—H = 0.86 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(carrier) or 1.5U_{eq}(methyl C).

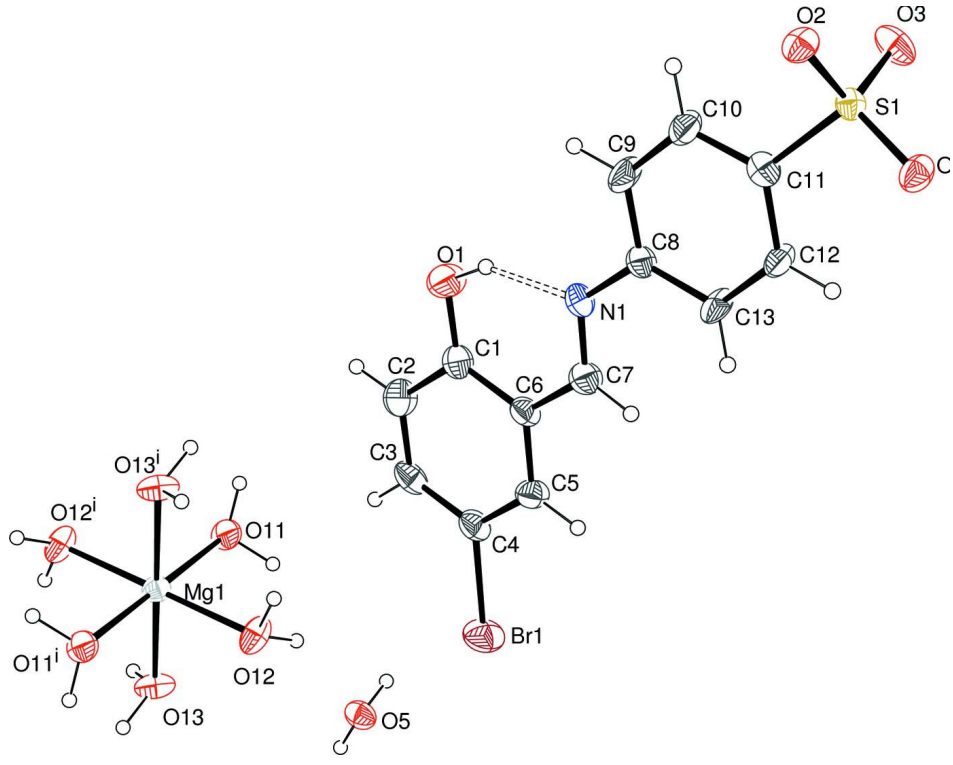

Figure 1

The molecular structure of (I) showing 50% displacement ellipsoids. Symmetry code: (i) –x, 1–y, –z.

Hexaaquamagnesium bis{4-[(5-bromo-2-hydroxybenzylidene)amino]benzenesulfonate} dihydrate

Crystal data

$[Mg(H_2O)_6](C_{13}H_9BrNO_4S)_2\cdot 2H_2O$
$M_r = 878.80$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 18.7737\ (14)$ Å
$b = 6.2837\ (5)$ Å
$c = 15.7591\ (12)$ Å
$\beta = 108.668\ (1)°$
$V = 1761.3\ (2)$ Å^3
$Z = 2$
$F(000) = 892$
$D_x = 1.657$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1852 reflections
$\theta = 2.3–22.6°$
$\mu = 2.51$ mm^{-1}
$T = 291$ K
Block, colourless
0.30 × 0.26 × 0.24 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.48$, $T_{max} = 0.55$
9144 measured reflections
3446 independent reflections
2473 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.6°$
$h = -21\rightarrow22$
$k = -7\rightarrow7$
$l = -18\rightarrow19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.131$
$S = 1.03$
3446 reflections
223 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 1.22P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.46$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.22648 (3)	1.13983 (10)	0.17778 (4)	0.0651 (2)
C1	0.3958 (3)	0.6851 (8)	0.1070 (3)	0.0477 (11)
C2	0.3228 (3)	0.6150 (9)	0.1013 (4)	0.0604 (13)
H2	0.3074	0.4775	0.0821	0.072*

C3	0.2748 (2)	0.7509 (8)	0.1242 (3)	0.0481 (11)
H3	0.2271	0.7041	0.1213	0.058*
C4	0.2961 (2)	0.9538 (8)	0.1511 (3)	0.0448 (10)
C5	0.3665 (2)	1.0246 (8)	0.1571 (3)	0.0450 (10)
H5	0.3805	1.1626	0.1767	0.054*
C6	0.4171 (2)	0.8944 (7)	0.1345 (3)	0.0397 (9)
C7	0.4892 (2)	0.9756 (8)	0.1354 (3)	0.0482 (11)
H7	0.5018	1.1160	0.1523	0.058*
C8	0.6080 (2)	0.9353 (7)	0.1133 (3)	0.0387 (9)
C9	0.6512 (3)	0.7946 (8)	0.0856 (4)	0.0563 (13)
H9	0.6325	0.6601	0.0658	0.068*
C10	0.7223 (3)	0.8507 (8)	0.0870 (3)	0.0513 (12)
H10	0.7512	0.7546	0.0671	0.062*
C11	0.7506 (2)	1.0454 (7)	0.1170 (3)	0.0403 (10)
C12	0.7072 (2)	1.1915 (7)	0.1430 (3)	0.0457 (11)
H12	0.7257	1.3271	0.1611	0.055*
C13	0.6353 (2)	1.1350 (8)	0.1421 (3)	0.0476 (12)
H13	0.6059	1.2315	0.1608	0.057*
Mg1	0.0000	0.5000	0.0000	0.0372 (4)
N1	0.53591 (18)	0.8598 (6)	0.1135 (2)	0.0436 (9)
O1	0.44163 (19)	0.5502 (6)	0.0844 (3)	0.0727 (12)
H1A	0.4853	0.6268	0.0825	0.109*
O2	0.85670 (16)	1.0353 (5)	0.04402 (18)	0.0401 (7)
O3	0.89136 (16)	0.9951 (6)	0.20297 (19)	0.0522 (8)
O4	0.85143 (17)	1.3394 (5)	0.1372 (2)	0.0450 (7)
O5	0.04046 (16)	0.9650 (5)	0.19393 (18)	0.0438 (7)
H5D	−0.0040	0.9738	0.1965	0.053*
H5A	0.0679	0.8973	0.2393	0.066*
O11	0.04471 (18)	0.3651 (5)	0.12539 (18)	0.0486 (8)
H11A	0.0911	0.2950	0.1291	0.073*
H11C	0.0542	0.4746	0.1701	0.073*
O12	0.05566 (19)	0.7759 (5)	0.0447 (2)	0.0503 (8)
H12A	0.1036	0.7722	0.0349	0.075*
H12B	0.0631	0.7904	0.1076	0.075*
O13	−0.08843 (18)	0.6110 (5)	0.0402 (2)	0.0506 (8)
H13A	−0.1230	0.6882	−0.0083	0.076*
H13C	−0.1138	0.4923	0.0560	0.076*
S1	0.84436 (5)	1.11031 (16)	0.12566 (6)	0.0322 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0464 (3)	0.0689 (4)	0.0887 (4)	0.0068 (2)	0.0338 (3)	0.0030 (3)
C1	0.040 (2)	0.047 (3)	0.055 (3)	−0.007 (2)	0.014 (2)	−0.003 (2)
C2	0.046 (3)	0.048 (3)	0.087 (4)	−0.018 (2)	0.021 (3)	−0.005 (3)
C3	0.028 (2)	0.058 (3)	0.056 (3)	−0.003 (2)	0.0105 (19)	0.013 (2)
C4	0.030 (2)	0.051 (3)	0.053 (3)	−0.0018 (19)	0.0128 (19)	0.004 (2)
C5	0.039 (2)	0.039 (3)	0.059 (3)	0.0022 (19)	0.019 (2)	0.002 (2)

C6	0.029 (2)	0.043 (3)	0.046 (2)	0.0004 (17)	0.0107 (17)	0.0030 (19)
C7	0.038 (2)	0.046 (3)	0.063 (3)	−0.003 (2)	0.018 (2)	−0.003 (2)
C8	0.0305 (19)	0.040 (2)	0.047 (2)	−0.0055 (17)	0.0137 (18)	−0.0009 (18)
C9	0.050 (3)	0.035 (2)	0.093 (4)	−0.020 (2)	0.035 (3)	−0.028 (2)
C10	0.048 (3)	0.049 (3)	0.062 (3)	−0.008 (2)	0.025 (2)	−0.025 (2)
C11	0.038 (2)	0.048 (3)	0.035 (2)	−0.0006 (19)	0.0116 (18)	0.0036 (18)
C12	0.041 (2)	0.039 (3)	0.067 (3)	−0.0133 (19)	0.031 (2)	−0.026 (2)
C13	0.047 (2)	0.049 (3)	0.063 (3)	−0.015 (2)	0.041 (2)	−0.031 (2)
Mg1	0.0380 (10)	0.0361 (11)	0.0405 (10)	0.0008 (8)	0.0166 (8)	−0.0003 (8)
N1	0.0279 (17)	0.048 (2)	0.054 (2)	−0.0080 (16)	0.0114 (15)	−0.0044 (18)
O1	0.0431 (18)	0.057 (2)	0.124 (3)	−0.0094 (17)	0.035 (2)	−0.026 (2)
O2	0.0475 (16)	0.0432 (17)	0.0386 (14)	−0.0031 (13)	0.0264 (13)	−0.0058 (13)
O3	0.0353 (16)	0.078 (2)	0.0378 (16)	0.0083 (16)	0.0042 (13)	0.0168 (16)
O4	0.0482 (17)	0.0379 (18)	0.0560 (18)	−0.0109 (14)	0.0267 (15)	−0.0102 (14)
O5	0.0436 (16)	0.0509 (19)	0.0358 (15)	0.0081 (14)	0.0111 (13)	0.0089 (13)
O11	0.063 (2)	0.0476 (19)	0.0329 (15)	0.0076 (15)	0.0119 (14)	0.0015 (13)
O12	0.065 (2)	0.0463 (19)	0.0510 (17)	−0.0202 (16)	0.0340 (16)	−0.0143 (14)
O13	0.0584 (19)	0.0361 (18)	0.072 (2)	0.0118 (15)	0.0415 (17)	0.0130 (15)
S1	0.0313 (5)	0.0350 (6)	0.0320 (5)	−0.0036 (4)	0.0123 (4)	0.0004 (4)

Geometric parameters (Å, º)

Br1—C4	1.899 (5)	C11—S1	1.770 (4)
C1—O1	1.334 (6)	C12—C13	1.390 (6)
C1—C6	1.402 (6)	C12—H12	0.9300
C1—C2	1.415 (6)	C13—H13	0.9300
C2—C3	1.370 (7)	Mg1—O12^{i}	2.031 (3)
C2—H2	0.9300	Mg1—O12	2.031 (3)
C3—C4	1.362 (7)	Mg1—O11	2.065 (3)
C3—H3	0.9300	Mg1—O11^{i}	2.065 (3)
C4—C5	1.367 (6)	Mg1—O13^{i}	2.077 (3)
C5—C6	1.385 (6)	Mg1—O13	2.077 (3)
C5—H5	0.9300	O1—H1A	0.9600
C6—C7	1.443 (6)	O2—S1	1.457 (3)
C7—N1	1.268 (6)	O3—S1	1.449 (3)
C7—H7	0.9300	O4—S1	1.452 (3)
C8—C9	1.362 (6)	O5—H5D	0.8501
C8—C13	1.376 (6)	O5—H5A	0.8500
C8—N1	1.436 (5)	O11—H11A	0.9600
C9—C10	1.373 (6)	O11—H11C	0.9600
C9—H9	0.9300	O12—H12A	0.9601
C10—C11	1.358 (6)	O12—H12B	0.9599
C10—H10	0.9300	O13—H13A	0.9599
C11—C12	1.374 (6)	O13—H13C	0.9600
O1—C1—C6	122.3 (4)	C8—C13—C12	119.6 (4)
O1—C1—C2	118.6 (4)	C8—C13—H13	120.2
C6—C1—C2	119.1 (4)	C12—C13—H13	120.2

C3—C2—C1	119.6 (5)	O12^{i}—Mg1—O12	180.0
C3—C2—H2	120.2	O12^{i}—Mg1—O11	89.32 (13)
C1—C2—H2	120.2	O12—Mg1—O11	90.68 (13)
C4—C3—C2	120.9 (4)	O12^{i}—Mg1—O11^{i}	90.68 (13)
C4—C3—H3	119.6	O12—Mg1—O11^{i}	89.32 (13)
C2—C3—H3	119.6	O11—Mg1—O11^{i}	180.0
C3—C4—C5	120.5 (4)	O12^{i}—Mg1—O13^{i}	88.86 (13)
C3—C4—Br1	119.4 (3)	O12—Mg1—O13^{i}	91.14 (13)
C5—C4—Br1	120.1 (4)	O11—Mg1—O13^{i}	91.94 (13)
C4—C5—C6	121.1 (4)	O11^{i}—Mg1—O13^{i}	88.06 (13)
C4—C5—H5	119.4	O12^{i}—Mg1—O13	91.14 (13)
C6—C5—H5	119.4	O12—Mg1—O13	88.86 (13)
C5—C6—C1	118.8 (4)	O11—Mg1—O13	88.06 (13)
C5—C6—C7	120.5 (4)	O11^{i}—Mg1—O13	91.94 (13)
C1—C6—C7	120.6 (4)	O13^{i}—Mg1—O13	180.0
N1—C7—C6	121.5 (4)	C7—N1—C8	123.0 (4)
N1—C7—H7	119.3	C1—O1—H1A	108.7
C6—C7—H7	119.3	H5D—O5—H5A	109.5
C9—C8—C13	120.0 (4)	Mg1—O11—H11A	109.3
C9—C8—N1	116.4 (4)	Mg1—O11—H11C	109.3
C13—C8—N1	123.6 (4)	H11A—O11—H11C	109.5
C8—C9—C10	120.2 (4)	Mg1—O12—H12A	109.1
C8—C9—H9	119.9	Mg1—O12—H12B	109.2
C10—C9—H9	119.9	H12A—O12—H12B	109.5
C11—C10—C9	120.4 (4)	Mg1—O13—H13A	109.3
C11—C10—H10	119.8	Mg1—O13—H13C	109.2
C9—C10—H10	119.8	H13A—O13—H13C	109.5
C10—C11—C12	120.2 (4)	O3—S1—O4	112.5 (2)
C10—C11—S1	120.2 (4)	O3—S1—O2	110.78 (19)
C12—C11—S1	119.6 (3)	O4—S1—O2	113.44 (18)
C11—C12—C13	119.5 (4)	O3—S1—C11	105.83 (19)
C11—C12—H12	120.3	O4—S1—C11	106.7 (2)
C13—C12—H12	120.3	O2—S1—C11	107.05 (19)

Symmetry code: (i) $-x$, $-y+1$, $-z$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···N1	0.96	1.73	2.570 (5)	144
O5—H5*D*···O3^{ii}	0.85	2.00	2.854 (4)	180
O5—H5*A*···O4^{iii}	0.85	2.08	2.892 (4)	160
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O12—H12*B*···O5	0.96	1.90	2.729 (4)	143
O13—H13*C*···O4^{vi}	0.96	1.88	2.763 (4)	152

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