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Syntheses and structures of three complexes obtained from lithium halides and dimethyl sulfoxide (DMSO)

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Keywords: crystal structure; lithium bromide; lithium iodide; DMSO complexes; homoleptic complexes.

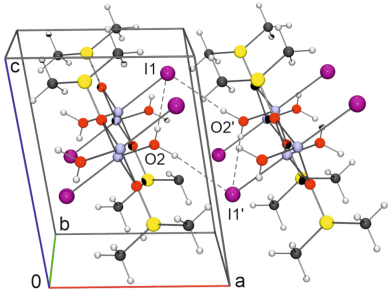
CCDC references: 2601817; 2601816; 2601815

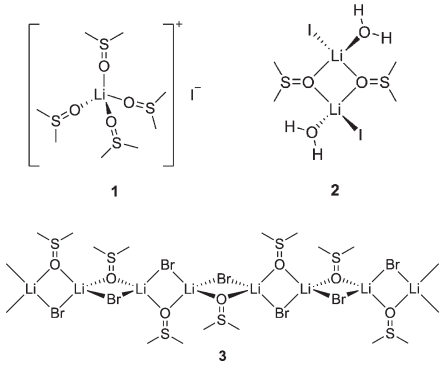
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The structures of three new lithium–DMSO complexes containing bromide and iodide anions are reported. One of the latter is the homoleptic tetrakis(DMSO) lithium cation with an isolated iodide counter-anion, tetrakis(dimethyl sulfoxide-κO)lithium(I) iodide, $[Li\{(CH_3)_2SO\}_4]I$. However, in the presence of a water molecule within the coordination sphere, a dimeric DMSO-bridged dinuclear lithium complex, bis(μ-dimethyl sulfoxide-κ^2O:O)bis[aquaiodidolithium(I)], $[Li_2I_2\{(CH_3)_2SO\}_2(H_2O)_2]$ or $[I\{\mu\text{-}(CH_3)_2SO\}(H_2O)Li]_2$, is formed, featuring both iodido and aqua ligands at the lithium centers. In contrast, the lithium bromide–DMSO complex *catena*-poly[lithium(I)-μ-bromido-μ-(dimethyl sulfoxide)-κ^2O:O], $\{LiBr\{(CH_3)_2SO\}\}_n$, is isotypic with the previously reported polymeric $[LiCl\{(CH_3)_2SO\}]_n$ structure described by Valdez *et al.* [(2023). *Acta Cryst.* E**79**, 33–37].

1. Chemical context

Many homoleptic DMSO complexes have been described, particularly for transition-metal centers such as copper, zinc, and nickel (Persson *et al.*, 1982; Bobicz *et al.*, 2002; Liu *et al.*, 2016). In contrast, homoleptic DMSO complexes with alkali metal centers remain rare; reported examples are limited predominantly to sodium-centered complexes (Sharutin, 2016; Wu *et al.*, 2019; Duarte-Ruiz *et al.*, 2011; Plata *et al.*, 2017; Takemoto *et al.*, 2018; Liu *et al.*, 2008; Yang *et al.*, 2023). A single homoleptic lithium–DMSO complex cation $[\mu\text{-}(DMSO)(DMSO)_2Li]_2^{2+}$ has been reported so far, and it features a dinuclear lithium complex with bridging and terminal DMSO ligands (Liu *et al.*, 2008). Given the extensive investigation of lithium salts and solvents in the context of battery applications, this scarcity is somewhat surprising (Bushkova *et al.*, 2017; Mauger *et al.*, 2018; Younesi *et al.*, 2015).





Here, we report the crystal structure of the first mononuclear homoleptic lithium DMSO complex. In this

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compound, the lithium cation is coordinated by four DMSO ligands in a distorted tetrahedral fashion. The unit cell contains an iodide counter-anion, which is not coordinated. Upon addition of water, a dimeric structure is formed in which two lithium ions are bridged by two DMSO ligands. Each lithium center is further coordinated by one aqua and one iodido ligand. In contrast, using LiBr instead of LiI, leads to the formation of a polymeric structure. This structure is isotypic with the $\{LiCl{\cdot}DMSO\}_n$ structure reported recently by Valdez *et al.* (2023).

2. Structural commentary

$[\{(CH_3)_2SO\}_4Li]I$ (**1**)

The obtained crystals are colorless. The product crystallizes in the orthorhombic space group *Pnc*2. The asymmetric unit contains half of the formula unit and is completed by symmetry (Fig. 1). The lithium atom is tetrahedrally coordinated by four DMSO ligands *via* the oxygen atoms. The Li—O bond lengths are 1.885 (7) and 2.050 (5) Å. The O—Li—O angles deviate from the ideal tetrahedral values with angles in the range from 77.5 (4) to 127.2 (5)°. The homoleptic lithium–DMSO complex reported by Liu *et al.* (2008) exhibits O—Li bond lengths in the range of 1.890 (9) to 1.96 (1) Å, which are therefore comparable. The iodide acts as a non-coordinating anion. The data obtained here are compromised by a disorder of one of the DMSO ligands.

$[I(\mu\text{-}(CH_3)_2SO)(H_2O)Li]_2$ (**2**)

The crystals are colorless. The product crystallizes in the triclinic space group $P\overline{1}$. The asymmetric unit consists of one lithium atom, the iodido ligand, the DMSO ligand, and one aqua ligand (Fig. 2). Symmetry-imposed inversion leads to the dimeric molecular structure revealing a square of two lithium centers and two oxygen atoms of the two bridging DMSO ligands. Furthermore, each lithium atom is coordinated by one iodido ligand and one aqua ligand. Each Li center exhibits a tetrahedral coordination. The four-membered ring formed by the two lithium ions and two oxygen atoms is constrained to be planar by symmetry with O—Li—O angles of 90.09 (16)°. The Li—O bond lengths 1.928 (4) and 1.921 (4) Å are equal within the esd.

$\{LiBr{\cdot}(CH_3)_2SO\}_n$ (**3**)

The crystals are colorless. The product crystallizes in the tetragonal space group $I4_1/a$. Each lithium atom is coordinated by two DMSO ligands via the oxygen atoms and by two bromido ligands (Fig. 3). Both the DMSO and bromido ligands act as bridging ligands, resulting in a polymeric structure. The Li—O bond lengths are 1.936 (4) and 1.923 (4) Å. The lithium atoms are in a distorted tetrahedral coordination environment with O—Li—Br angles in the range from 97.89 (14) to 119.47 (17)°. This compound is comparable to $\{LiCl{\cdot}(CH_3)_2SO\}_n$, as reported by Valdez *et al.* (2023), in terms of Li–O bond lengths and O–Li–Cl angles; both compounds also crystallize in the same space group.

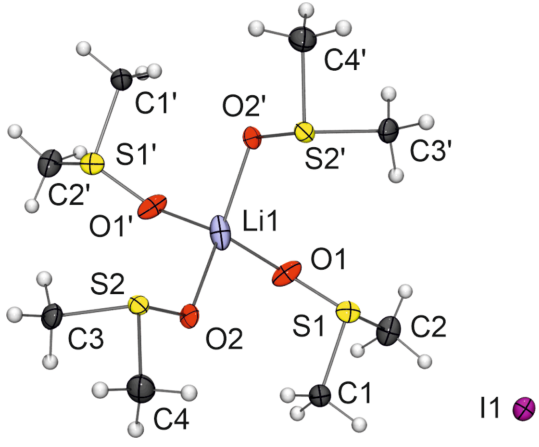


Figure 1
Coordination environment of the Li atom in $[\{(CH_3)_2SO\}_4Li]I$ (**1**). Atoms are shown as ellipsoids at the 30% probability level. Hydrogen atoms are displayed as spheres of arbitrary radius. Only one split position of O2 and S2 is shown. Symmetry code; (′) $-x, 1-y, z$.

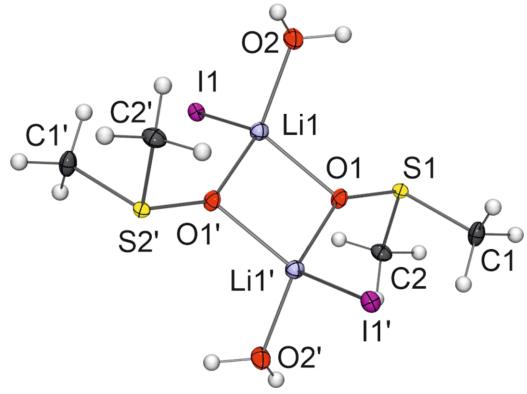


Figure 2
Coordination environment of the Li atom in $[I(\mu\text{-}(CH_3)_2SO)(H_2O)Li]_2$ (**2**). The second half of the molecule is generated by symmetry. Atoms are shown as ellipsoids at the 30% probability level. Hydrogen atoms are displayed as spheres of arbitrary radius. Symmetry code; (′) $1-x, 1-y, 1-z$.

3. Supramolecular features

$[\{(CH_3)_2SO\}_4Li]I$ (**1**) crystallizes with separate complex cations and iodide anions (Fig. 4).

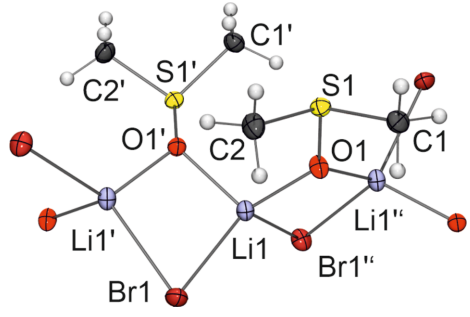


Figure 3
Detail of the polymeric crystal structure of $\{LiBr{\cdot}(CH_3)_2SO\}_n$ (**3**). Atoms are shown as ellipsoids at the 30% probability level. Hydrogen atoms are displayed as spheres of arbitrary radius.

Table 1
Hydrogen-bond geometry (Å, °) for **2**.

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···I1^{i}	0.72 (3)	2.8 (3)	3.533 (2)	173 (3)
O2—H2B···I1^{ii}	0.89 (3)	2.64 (3)	3.522 (2)	170 (3)

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

Table 2
Hydrogen-bond geometry (Å, °) for **3**.

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···Br1^{i}	0.98	2.91	3.852 (3)	162
C1—H1B···Br1^{i}	0.98	2.96	3.852 (3)	152

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

$[I(\mu\text{-}(CH_3)_2SO)(H_2O)Li]_2$ (**2**) exhibits an O—H···I hydrogen-bonding network in which the iodido ligand acts as a double acceptor (Table 1, Fig. 5), interacting with both hydrogen atoms of the coordinated water molecule (O2—H2A···I = 3.533 (2) Å; O2—H2B···I = 3.522 (2) Å) to form a zigzag chain along the [001] direction.

In $\{LiBr\cdot(CH_3)_2SO\}_n$ (**3**), hydrogen atoms were added in calculated positions. The resulting distances Br1···H1A—C1 3.852 (3) Å and Br1···H1B—C1 3.855 (3) Å (Table 2, Fig. 6), suggest that the polymeric units may be held together by hydrogen bonding, analogous to the interactions described for $\{LiCl\cdot(CH_3)_2SO\}_n$ by Valdez *et al.* (2023).

4. Database survey

A search for different homoleptic DMSO complexes was performed in the Cambridge Structural Database (CSD, version 2026.1.0, February 2026; Groom *et al.*, 2016). The ambidentate nature of DMSO was taken into account. A variety of homoleptic DMSO complexes was identified, but only a few contained alkali metals as central atoms. Only one homoleptic lithium DMSO complex is known (Liu *et al.*, 2008; CSD refcode TOQFIP). This complex is binuclear, with two DMSO molecules acting as bridging ligands. No homoleptic, homonuclear lithium DMSO complex was found.

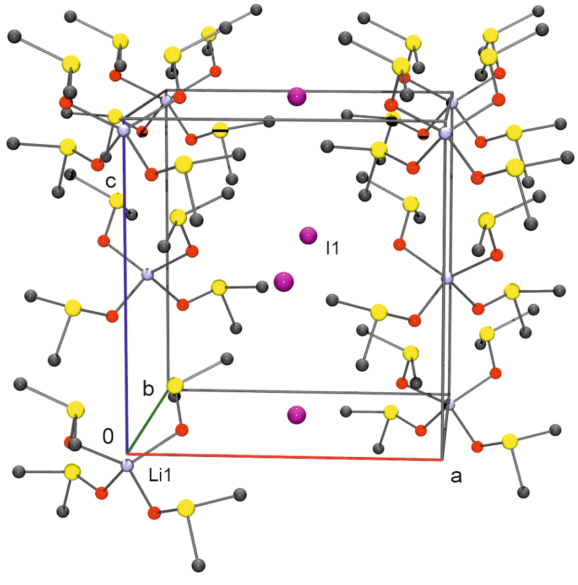


Figure 4
Packing diagram of $[\{(CH_3)_2SO\}_4Li]I$ (**1**). The complex cations and iodide anions are separated.

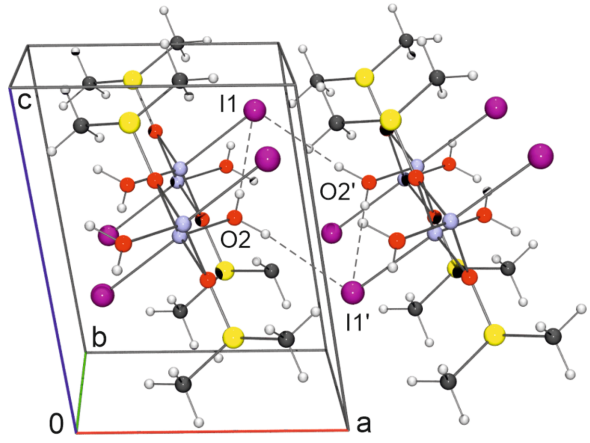


Figure 5
Packing diagram of $[I(\mu\text{-}(CH_3)_2SO)(H_2O)Li]_2$ (**2**). The structure is held together by hydrogen bonding. Hydrogen bonds are shown as dashed lines.

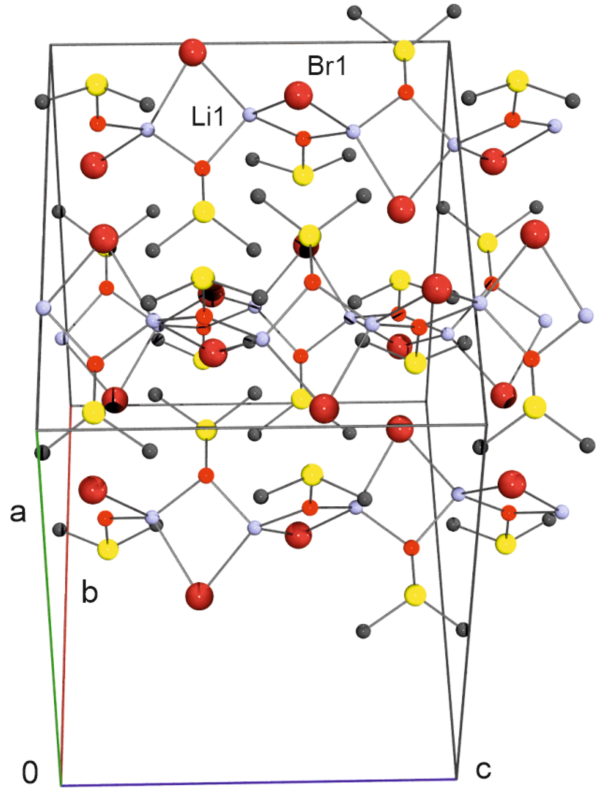


Figure 6
Packing diagram of $\{LiBr\cdot(CH_3)_2SO\}_n$ (**3**). The polymeric units may be held together by hydrogen bonding, analogous to the interactions described for $\{LiCl\cdot(CH_3)_2SO\}_n$ by Valdez *et al.* (2023).

Table 3
Experimental details.

	1	**2**	**3**
Crystal data			
Chemical formula	$Li(C_2H_6OS)_4]I$	$[Li_2I_2(C_2H_6OS)_2(H_2O)_2]$	$LiBr(C_2H_6OS)]$
M_r	446.35	459.97	164.98
Crystal system, space group	Orthorhombic, *Pnc*2	Triclinic, $P\overline{1}$	Tetragonal, $I4_1/a$
Temperature (K)	100	100	200
a, *b*, *c* (Å)	9.4961 (7), 9.7157 (7), 10.1935 (6)	6.2824 (6), 7.4147 (8), 8.3722 (9)	14.6281 (5), 14.6281 (5), 11.0592 (4)
α, β, γ (°)	90, 90, 90	96.563 (9), 97.701 (8), 93.656 (8)	90, 90, 90
V (Å^3)	940.47 (11)	382.70 (7)	2366.46 (18)
Z	2	1	16
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Cu $K\alpha$
μ (mm^{-1})	2.15	4.37	11.67
Crystal size (mm)	0.23 × 0.16 × 0.07	0.29 × 0.22 × 0.09	0.14 × 0.11 × 0.1
Data collection			
Diffractometer	Stoe STADIVARI	Stoe STADIVARI	Stoe STADIVARI
Absorption correction	Multi-scan [*LANA* (Stoe & Cie, 2016), absorption correction by scaling of reflection intensities (Koziskova *et al.*, 2016)]	Multi-scan [*LANA* (Stoe & Cie, 2016), absorption correction by scaling of reflection intensities (Koziskova *et al.*, 2016)]	Multi-scan [*LANA* (Stoe & Cie, 2016), absorption correction by scaling of reflection intensities (Koziskova *et al.*, 2016)]
T_{min}, T_{max}	0.442, 0.780	0.282, 0.675	0.195, 0.311
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9875, 2726, 2331	4662, 2173, 2043	3825, 1134, 1105
R_{int}	0.021	0.020	0.018
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.731	0.703	0.615
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.020, 0.039, 0.96	0.020, 0.051, 1.04	0.024, 0.067, 1.08
No. of reflections	2726	2173	1134
No. of parameters	125	74	58
No. of restraints	15	0	0
H-atom treatment	H-atom parameters constrained	H atoms treated by a mixture of independent and constrained refinement	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.31, −0.49	1.10, −1.08	0.58, −0.34
Absolute structure	Flack *x* determined using 943 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)	–	–
Absolute structure parameter	0.001 (16)	–	–

Computer programs: *X-AREA Pilatus3_SV*, *X-AREA Recipe*, *X-AREA Integrate* and *X-AREA LANA* (Stoe & Cie, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2025/1* (Sheldrick, 2015*b*), *OLEX2* (Dolomanov *et al.*, 2009) and *ORTEP-3 for Windows* (Farrugia, 2012).

5. Synthesis and crystallization

The reactions were carried out by using LiBr (Sigma-Aldrich, ≥99%), LiI (TCI, 99.9%), dimethylsulfoxide-d_6 (deutero, 99.8%) and *o*-difluorobenzene (abcr, 98%). Dimethylsulfoxide-d_6 (DMSO-d_6) and *o*-difluorobenzene (*o*-DFB) were stored over molecular sieve (3 Å) before use. All operations involving LiI were carried out with all laboratory lights switched off. The reaction flask was wrapped in aluminum foil.

$[\{(CH_3)_2SO\}_4Li]I$ (**1**)

LiI (425 mg, 3.18 mmol) was dissolved in DMSO-d_6 (3 mL, 41.5 mmol). After stirring for 30 min, volatile components were removed *in vacuo*. A few single crystals suitable for X-ray diffraction analysis were obtained from the colorless solid, which was isolated in quantitative yield.

^{7}Li NMR (194 MHz, 298 K, DMSO-d_6) δ: −1.1 ppm.

$[I(\mu\text{-}(CH_3)_2SO)(H_2O)Li]_2$ (**2**)

LiI (500 mg, 3.74 mmol) and DMSO-d_6 (0.27 mL, 3.74 mmol) were dissolved in H_2O (10 mL). The resulting solution was stirred for 30 min before volatile components were removed *in vacuo*. A few single crystals suitable for X-ray diffraction analysis were obtained from the colorless solid, which was isolated in quantitative yield.

^{7}Li NMR (194 MHz, 298 K, DMSO-d_6) δ: −1.1 ppm.

$\{LiBr\cdot(CH_3)_2SO\}_n$ (**3**)

LiBr (1000 mg, 11.5 mmol) was suspended in *o*-DFB (5 mL). DMSO-d_6 (0.76 mL, 11.1 mmol) was added, and the suspension was stirred for 20 h. Volatile components were removed *in vacuo*. Single crystals suitable for X-ray diffraction analysis were obtained from the colorless solid, which was isolated in quantitative yield.

^{7}Li NMR (194 MHz, 298 K, DMSO-d_6) δ: −1.1 ppm.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The disorder of one DMSO ligand in **1** was modeled by refinement of two split positions with a fixed occupancy of 0.5 for each position.

Acknowledgements

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Syntheses and structures of three complexes obtained from lithium halides and dimethyl sulfoxide (DMSO)

Jan Cramer and Clemens Bruhn

Computing details

Tetrakis(dimethyl sulfoxide-κO)lithium(I) iodide (1)

Crystal data

$Li(C_2H_6OS)_4]I$
M_r = 446.35
Orthorhombic, *Pnc*2
a = 9.4961 (7) Å
b = 9.7157 (7) Å
c = 10.1935 (6) Å
V = 940.47 (11) Å^3
Z = 2
$F(000)$ = 448
D_x = 1.576 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 12512 reflections
θ = 2.1–31.5°
μ = 2.15 mm^{-1}
T = 100 K
Plate, colorless
0.23 × 0.16 × 0.07 mm

Data collection

Stoe STADIVARI diffractometer
Radiation source: Mo Genix
Graded multilayer mirror monochromator
Detector resolution: 5.81 pixels mm^{-1}
rotation method, ω scans
Absorption correction: multi-scan [*LANA* (Stoe & Cie, 2016), absorption correction by scaling of reflection intensities (Koziskova *et al.*, 2016)]
T_{min} = 0.442, T_{max} = 0.780
9875 measured reflections
2726 independent reflections
2331 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 31.3°, θ_{min} = 2.2°
h = −13→12
k = −14→10
l = −12→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.039
S = 0.96
2726 reflections
125 parameters
15 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0212P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}
Absolute structure: Flack x determined using 943 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.001 (16)

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}	Occ. (<1)
I1	0.500000	1.000000	0.52473 (4)	0.01896 (6)	
S1	0.21522 (8)	0.72030 (7)	0.39164 (7)	0.02024 (14)	
S3	0.19760 (17)	0.30411 (16)	0.59470 (14)	0.0227 (3)	0.5
S2	0.12958 (15)	0.30539 (14)	0.67128 (14)	0.0171 (3)	0.5
O1	0.1025 (2)	0.6197 (2)	0.35067 (19)	0.0288 (5)	
C1	0.3730 (3)	0.6217 (3)	0.4114 (3)	0.0215 (6)	
H1A	0.359794	0.554159	0.481763	0.032*	
H1B	0.451106	0.683248	0.434088	0.032*	
H1C	0.394627	0.573723	0.329294	0.032*	
C2	0.2638 (4)	0.8084 (3)	0.2449 (3)	0.0277 (7)	
H2A	0.285100	0.741156	0.176079	0.042*	
H2B	0.347265	0.865176	0.261641	0.042*	
H2C	0.185893	0.867396	0.216435	0.042*	
Li1	0.000000	0.500000	0.4651 (8)	0.0277 (16)	
O3	0.0883 (5)	0.4146 (4)	0.6093 (4)	0.0265 (10)	0.5
O2	0.1628 (5)	0.3981 (4)	0.5546 (4)	0.0205 (10)	0.5
C5	0.104 (3)	0.148 (2)	0.5652 (14)	0.026 (4)	0.5
H5A	0.033998	0.133971	0.634697	0.039*	0.5
H5B	0.170010	0.070576	0.564397	0.039*	0.5
H5C	0.055929	0.153570	0.480161	0.039*	0.5
C3	0.105 (3)	0.137 (2)	0.6056 (15)	0.023 (3)	0.5
H3A	0.020824	0.136477	0.549850	0.035*	0.5
H3B	0.092891	0.071439	0.677512	0.035*	0.5
H3C	0.187379	0.111623	0.553285	0.035*	0.5
C4	0.2957 (17)	0.274 (3)	0.751 (3)	0.029 (4)	0.5
H4A	0.365561	0.246442	0.685042	0.043*	0.5
H4B	0.284753	0.201174	0.815881	0.043*	0.5
H4C	0.327451	0.358875	0.794144	0.043*	0.5
C6	0.2568 (18)	0.261 (3)	0.756 (2)	0.028 (4)	0.5
H6A	0.318226	0.334128	0.789262	0.042*	0.5
H6B	0.309246	0.174060	0.753185	0.042*	0.5
H6C	0.175404	0.250783	0.814450	0.042*	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.01963 (10)	0.01998 (10)	0.01727 (10)	−0.00264 (12)	0.000	0.000
S1	0.0202 (3)	0.0247 (3)	0.0158 (3)	0.0017 (3)	0.0002 (3)	−0.0022 (3)
S3	0.0243 (8)	0.0226 (8)	0.0213 (8)	−0.0029 (7)	−0.0030 (7)	0.0007 (6)

S2	0.0162 (7)	0.0161 (7)	0.0189 (7)	0.0020 (6)	0.0051 (6)	0.0040 (5)
O1	0.0206 (11)	0.0424 (14)	0.0234 (10)	−0.0105 (11)	−0.0029 (9)	0.0039 (9)
C1	0.0199 (15)	0.0191 (14)	0.0254 (15)	−0.0001 (12)	0.0008 (12)	−0.0003 (12)
C2	0.035 (2)	0.0281 (17)	0.0198 (14)	−0.0065 (15)	−0.0042 (13)	0.0022 (13)
Li1	0.047 (5)	0.017 (3)	0.019 (3)	−0.001 (4)	0.000	0.000
O3	0.030 (3)	0.023 (2)	0.026 (2)	0.005 (2)	−0.007 (2)	0.0023 (18)
O2	0.021 (2)	0.015 (2)	0.026 (3)	−0.0019 (17)	−0.0016 (18)	0.0123 (17)
C5	0.020 (4)	0.018 (5)	0.039 (10)	−0.002 (4)	0.003 (8)	−0.003 (6)
C3	0.023 (4)	0.019 (5)	0.027 (8)	−0.004 (4)	−0.001 (7)	0.006 (5)
C4	0.028 (9)	0.027 (5)	0.031 (6)	0.001 (6)	−0.009 (6)	0.003 (4)
C6	0.034 (9)	0.032 (7)	0.018 (5)	0.007 (8)	−0.006 (6)	−0.001 (5)

Geometric parameters (Å, º)

S1—O1	1.509 (2)	Li1—O3^{i}	1.885 (7)
S1—C1	1.790 (3)	Li1—O3	1.885 (7)
S1—C2	1.784 (3)	Li1—O2^{i}	2.050 (5)
S3—Li1	2.981 (4)	Li1—O2	2.050 (5)
S3—O3	1.500 (5)	C5—H5A	0.9800
S3—C5	1.78 (2)	C5—H5B	0.9800
S3—C6	1.79 (2)	C5—H5C	0.9800
S2—O2	1.525 (4)	C3—H3A	0.9800
S2—C3	1.78 (2)	C3—H3B	0.9800
S2—C4	1.80 (2)	C3—H3C	0.9800
O1—Li1	1.913 (5)	C4—H4A	0.9800
C1—H1A	0.9800	C4—H4B	0.9800
C1—H1B	0.9800	C4—H4C	0.9800
C1—H1C	0.9800	C6—H6A	0.9800
C2—H2A	0.9800	C6—H6B	0.9800
C2—H2B	0.9800	C6—H6C	0.9800
C2—H2C	0.9800		

O1—S1—C1	106.18 (13)	O3^{i}—Li1—S3	102.5 (3)
O1—S1—C2	105.22 (13)	O3—Li1—O1	121.12 (17)
C2—S1—C1	97.74 (15)	O3^{i}—Li1—O1	115.74 (16)
O3—S3—Li1	32.0 (2)	O3^{i}—Li1—O3	77.5 (4)
O3—S3—C5	106.2 (8)	O3^{i}—Li1—O2^{i}	26.46 (17)
O3—S3—C6	107.1 (8)	O3—Li1—O2^{i}	101.6 (4)
C5—S3—Li1	98.9 (6)	O2—Li1—S3^{i}	125.9 (3)
C5—S3—C6	96.5 (9)	O2^{i}—Li1—S3^{i}	11.43 (12)
C6—S3—Li1	139.0 (8)	O2^{i}—Li1—O2	127.2 (5)
O2—S2—C3	106.0 (5)	S3—O3—Li1	123.0 (3)
O2—S2—C4	105.5 (8)	S2—O2—Li1	118.4 (3)
C3—S2—C4	97.5 (12)	S3—C5—H5A	109.5
S1—O1—Li1	125.9 (2)	S3—C5—H5B	109.5
S1—C1—H1A	109.5	S3—C5—H5C	109.5
S1—C1—H1B	109.5	H5A—C5—H5B	109.5
S1—C1—H1C	109.5	H5A—C5—H5C	109.5

H1A—C1—H1B	109.5	H5B—C5—H5C	109.5
H1A—C1—H1C	109.5	S2—C3—H3A	109.5
H1B—C1—H1C	109.5	S2—C3—H3B	109.5
S1—C2—H2A	109.5	S2—C3—H3C	109.5
S1—C2—H2B	109.5	H3A—C3—H3B	109.5
S1—C2—H2C	109.5	H3A—C3—H3C	109.5
H2A—C2—H2B	109.5	H3B—C3—H3C	109.5
H2A—C2—H2C	109.5	S2—C4—H4A	109.5
H2B—C2—H2C	109.5	S2—C4—H4B	109.5
S3^{i}—Li1—S3	127.4 (3)	S2—C4—H4C	109.5
O1—Li1—S3	109.76 (8)	H4A—C4—H4B	109.5
O1—Li1—S3^{i}	101.67 (8)	H4A—C4—H4C	109.5
O1^{i}—Li1—S3	101.67 (8)	H4B—C4—H4C	109.5
O1^{i}—Li1—S3^{i}	109.76 (8)	S3—C6—H6A	109.5
O1^{i}—Li1—O1	104.9 (4)	S3—C6—H6B	109.5
O1—Li1—O2^{i}	111.17 (15)	S3—C6—H6C	109.5
O1^{i}—Li1—O2^{i}	100.45 (14)	H6A—C6—H6B	109.5
O1—Li1—O2	100.45 (14)	H6A—C6—H6C	109.5
O1^{i}—Li1—O2	111.17 (15)	H6B—C6—H6C	109.5
O3—Li1—S3	24.95 (15)		

S3^{i}—Li1—O3—S3	179.1 (3)	O3^{i}—Li1—O3—S3	178.5 (5)
O1—Li1—O3—S3	−68.8 (4)	C5—S3—O3—Li1	−80.6 (5)
O1^{i}—Li1—O3—S3	59.7 (4)	C3—S2—O2—Li1	93.1 (9)
C1—S1—O1—Li1	80.3 (2)	C4—S2—O2—Li1	−164.1 (10)
C2—S1—O1—Li1	−176.76 (17)	C6—S3—O3—Li1	177.2 (7)

Symmetry code: (i) −*x*, −*y*+1, *z*.

Bis(*μ*-dimethyl sulfoxide-*κ*^{2}*O*:*O*)bis[aquaiodidolithium(I)] (2)

Crystal data

$[Li_2I_2(C_2H_6OS)_2(H_2O)_2]$
M_r = 459.97
Triclinic, $P\overline{1}$
a = 6.2824 (6) Å
b = 7.4147 (8) Å
c = 8.3722 (9) Å
α = 96.563 (9)°
β = 97.701 (8)°
γ = 93.656 (8)°
V = 382.70 (7) Å^{3}
Z = 1
F(000) = 216
D_x = 1.996 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 12597 reflections
θ = 2.5–32.5°
μ = 4.37 mm^{-1}
T = 100 K
Plate, colorless
0.29 × 0.22 × 0.09 mm

Data collection

Stoe STADIVARI diffractometer
Radiation source: Mo Genix
Graded multilayer mirror monochromator
Detector resolution: 5.81 pixels mm^{-1}
rotation method, *ω* scans
Absorption correction: multi-scan [*LANA* (Stoe & Cie, 2016), absorption correction by scaling of reflection intensities (Koziskova *et al.*, 2016)]
T_{min} = 0.282, T_{max} = 0.675
4662 measured reflections
2173 independent reflections
2043 reflections with $I > 2\sigma(I)$

R_{int} = 0.020
θ_{max} = 30.0°, θ_{min} = 2.5°
h = −8→8
k = −10→10
l = −9→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.051
S = 1.04
2173 reflections
74 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0356P)^2 + 0.0323P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.10 e Å^{-3}
$\Delta\rho_{min}$ = −1.08 e Å^{-3}

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}
I1	0.13264 (2)	0.75536 (2)	0.24545 (2)	0.01794 (6)
S1	0.63060 (8)	0.38667 (6)	0.18966 (6)	0.01450 (10)
O1	0.5700 (3)	0.4373 (2)	0.35839 (18)	0.0197 (3)
O2	0.7103 (3)	0.8526 (3)	0.4711 (3)	0.0287 (4)
H2A	0.746 (5)	0.927 (4)	0.534 (4)	0.024 (8)*
H2B	0.823 (5)	0.819 (4)	0.424 (4)	0.029 (8)*
C1	0.8294 (4)	0.2296 (3)	0.2201 (3)	0.0243 (4)
H1A	0.957584	0.291679	0.289030	0.036*
H1B	0.868221	0.180956	0.114980	0.036*
H1C	0.772281	0.129574	0.273113	0.036*
C2	0.4138 (4)	0.2340 (3)	0.0840 (3)	0.0228 (4)
H2C	0.390296	0.132616	0.146814	0.034*
H2D	0.449144	0.186752	−0.022589	0.034*
H2E	0.282604	0.298657	0.069706	0.034*
Li1	0.4847 (6)	0.6675 (5)	0.4501 (5)	0.0198 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.01576 (8)	0.02273 (8)	0.01467 (8)	0.00095 (5)	0.00006 (5)	0.00234 (5)
S1	0.0161 (2)	0.0155 (2)	0.0124 (2)	0.00190 (17)	0.00215 (17)	0.00344 (16)
O1	0.0263 (8)	0.0205 (7)	0.0137 (7)	0.0064 (6)	0.0062 (6)	0.0012 (5)
O2	0.0219 (8)	0.0261 (8)	0.0355 (10)	−0.0059 (7)	0.0090 (7)	−0.0088 (8)
C1	0.0251 (10)	0.0311 (11)	0.0189 (10)	0.0135 (9)	0.0044 (8)	0.0049 (8)
C2	0.0214 (10)	0.0240 (10)	0.0208 (11)	−0.0059 (8)	−0.0001 (8)	0.0017 (8)
Li1	0.0200 (17)	0.0193 (16)	0.0203 (18)	0.0022 (13)	0.0020 (14)	0.0036 (13)

Geometric parameters (Å, º)

I1—Li1	2.770 (4)	O2—Li1	1.885 (4)
S1—O1	1.5240 (15)	C1—H1A	0.9800
S1—C1	1.774 (2)	C1—H1B	0.9800
S1—C2	1.781 (2)	C1—H1C	0.9800
O1—Li1	1.928 (4)	C2—H2C	0.9800
O1—Li1^{i}	1.921 (4)	C2—H2D	0.9800
O2—H2A	0.72 (3)	C2—H2E	0.9800
O2—H2B	0.89 (3)	Li1—Li1^{i}	2.720 (7)
O1—S1—C1	104.06 (10)	S1—C2—H2D	109.5
O1—S1—C2	105.32 (10)	S1—C2—H2E	109.5
C1—S1—C2	99.29 (12)	H2C—C2—H2D	109.5
S1—O1—Li1	128.17 (15)	H2C—C2—H2E	109.5
S1—O1—Li1^{i}	141.67 (15)	H2D—C2—H2E	109.5
Li1^{i}—O1—Li1	89.91 (16)	O1—Li1—I1	107.72 (17)
H2A—O2—H2B	108 (3)	O1^{i}—Li1—I1	117.58 (17)
Li1—O2—H2A	132 (2)	O1^{i}—Li1—O1	90.09 (16)
Li1—O2—H2B	114 (2)	O1^{i}—Li1—Li1^{i}	45.15 (12)
S1—C1—H1A	109.5	O1—Li1—Li1^{i}	44.93 (12)
S1—C1—H1B	109.5	O2—Li1—I1	109.86 (17)
S1—C1—H1C	109.5	O2—Li1—O1	111.3 (2)
H1A—C1—H1B	109.5	O2—Li1—O1^{i}	118.1 (2)
H1A—C1—H1C	109.5	O2—Li1—Li1^{i}	126.2 (3)
H1B—C1—H1C	109.5	Li1^{i}—Li1—I1	122.9 (2)
S1—C2—H2C	109.5		
C1—S1—O1—Li1^{i}	−40.9 (3)	C2—S1—O1—Li1	−109.4 (2)
C1—S1—O1—Li1	146.7 (2)	C2—S1—O1—Li1^{i}	63.0 (3)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···I1^{ii}	0.72 (3)	2.8 (3)	3.533 (2)	173 (3)
O2—H2*B*···I1^{iii}	0.89 (3)	2.64 (3)	3.522 (2)	170 (3)

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

catena-Poly[lithium(I)-μ-bromido-μ-(dimethyl sulfoxide)-κ^2O:O] (3)

Crystal data

LiBr(C_2H_6OS)]
M_r = 164.98
Tetragonal, $I4_1/a$
a = 14.6281 (5) Å
c = 11.0592 (4) Å
V = 2366.46 (18) Å^3
Z = 16
F(000) = 1280
D_x = 1.852 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54186 Å
Cell parameters from 8396 reflections
θ = 5.0–71.7°

μ = 11.67 mm^{-1}
T = 200 K

Block, yellow
0.14 × 0.11 × 0.1 mm

Data collection

Stoe STADIVARI diffractometer
Radiation source: Cu-Genix
Graded multilayer mirror monochromator
Detector resolution: 5.81 pixels mm^{-1}
rotation method, ω scans
Absorption correction: multi-scan [*LANA* (Stoe & Cie, 2016), absorption correction by scaling of reflection intensities (Koziskova *et al.*, 2016)]

T_{min} = 0.195, T_{max} = 0.311
3825 measured reflections
1134 independent reflections
1105 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 71.5°, θ_{min} = 5.0°
h = −11→17
k = −17→10
l = −5→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.067
S = 1.08
1134 reflections
58 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0448P)^2 + 2.6713P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.58 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}
Extinction correction: SHELXL-2025/1 (Sheldrick 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00014 (3)

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Br1	0.10586 (2)	0.43230 (2)	0.35529 (2)	0.03321 (15)
S1	0.19696 (4)	0.66089 (4)	0.62759 (5)	0.02597 (16)
O1	0.20946 (11)	0.55714 (11)	0.62543 (13)	0.0298 (4)
C1	0.11884 (16)	0.68087 (15)	0.7477 (2)	0.0324 (5)
H1A	0.064721	0.642234	0.736820	0.049*
H1B	0.100494	0.745303	0.747540	0.049*
H1C	0.148187	0.666091	0.824886	0.049*
Li1	0.2330 (2)	0.4758 (3)	0.4924 (3)	0.0293 (7)
C2	0.12427 (17)	0.68524 (16)	0.5030 (2)	0.0365 (5)
H2A	0.157618	0.674927	0.427333	0.055*
H2B	0.104400	0.749146	0.507005	0.055*
H2C	0.070695	0.645070	0.505827	0.055*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.03019 (18)	0.03499 (19)	0.0344 (2)	−0.00953 (9)	−0.00390 (8)	0.00083 (9)
S1	0.0238 (3)	0.0260 (3)	0.0281 (3)	−0.0009 (2)	−0.00096 (18)	0.00102 (19)
O1	0.0366 (8)	0.0282 (8)	0.0245 (7)	0.0088 (7)	−0.0021 (6)	−0.0014 (6)
C1	0.0355 (11)	0.0283 (10)	0.0335 (12)	0.0026 (8)	0.0028 (9)	−0.0039 (8)
Li1	0.0345 (18)	0.0312 (18)	0.0224 (16)	−0.0024 (15)	−0.0009 (14)	0.0001 (13)
C2	0.0440 (12)	0.0334 (12)	0.0323 (12)	0.0075 (10)	−0.0061 (10)	0.0034 (9)

Geometric parameters (Å, º)

Br1—Li1	2.482 (4)	C1—H1B	0.9800
Br1—Li1^{i}	2.485 (4)	C1—H1C	0.9800
S1—O1	1.5288 (16)	Li1—Li1^{ii}	2.8318 (12)
S1—C1	1.776 (2)	Li1—Li1^{i}	2.8318 (11)
S1—C2	1.777 (2)	C2—H2A	0.9800
O1—Li1^{ii}	1.936 (4)	C2—H2B	0.9800
O1—Li1	1.923 (4)	C2—H2C	0.9800
C1—H1A	0.9800		
Li1—Br1—Li1^{i}	69.52 (9)	Br1—Li1—Li1^{i}	55.28 (11)
O1—S1—C1	104.60 (10)	O1—Li1—Br1	119.47 (17)
O1—S1—C2	104.98 (10)	O1^{i}—Li1—Br1	97.89 (14)
C1—S1—C2	99.32 (12)	O1^{i}—Li1—Br1^{ii}	113.68 (17)
S1—O1—Li1^{ii}	127.19 (14)	O1—Li1—Br1^{ii}	98.14 (14)
S1—O1—Li1	130.35 (14)	O1—Li1—O1^{i}	107.98 (18)
Li1—O1—Li1^{ii}	94.41 (12)	O1—Li1—Li1^{i}	127.48 (19)
S1—C1—H1A	109.5	O1^{i}—Li1—Li1^{ii}	120.98 (19)
S1—C1—H1B	109.5	O1^{i}—Li1—Li1^{i}	42.63 (10)
S1—C1—H1C	109.5	O1—Li1—Li1^{ii}	42.96 (10)
H1A—C1—H1B	109.5	Li1^{ii}—Li1—Li1^{i}	162.41 (15)
H1A—C1—H1C	109.5	S1—C2—H2A	109.5
H1B—C1—H1C	109.5	S1—C2—H2B	109.5
Br1—Li1—Br1^{ii}	119.98 (15)	S1—C2—H2C	109.5
Br1^{ii}—Li1—Li1^{i}	131.27 (15)	H2A—C2—H2B	109.5
Br1^{ii}—Li1—Li1^{ii}	55.19 (11)	H2A—C2—H2C	109.5
Br1—Li1—Li1^{ii}	139.95 (16)	H2B—C2—H2C	109.5
C1—S1—O1—Li1	148.03 (19)	C2—S1—O1—Li1^{ii}	−175.54 (19)
C1—S1—O1—Li1^{ii}	−71.5 (2)	C2—S1—O1—Li1	44.0 (2)

Symmetry codes: (i) $y-1/4, -x+3/4, z-1/4$; (ii) $-y+3/4, x+1/4, z+1/4$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···Br1^{iii}	0.98	2.91	3.852 (3)	162

C1—H1*B*···Br1^{iii}	0.98	2.96	3.852 (3)	152

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