

# Poly[di- $\mu_3$ -acetato- $\mu_8$ -(naphthalene-1,5-disulfonato)-dilead(II)]

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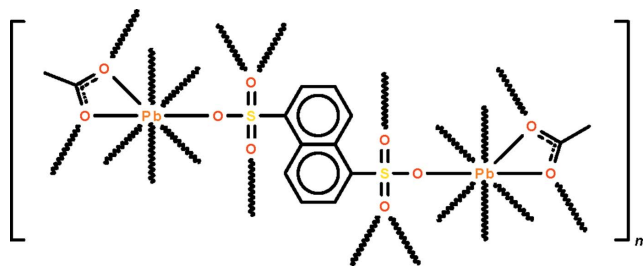
Received 23 April 2012; accepted 24 April 2012

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.008$  Å;  $R$  factor = 0.030;  $wR$  factor = 0.076; data-to-parameter ratio = 15.8.

In the polymeric title complex,  $[\text{Pb}_2(\text{CH}_3\text{CO}_2)_2(\text{C}_{10}\text{H}_6\text{O}_6\text{S}_2)]_n$ , the acetate anion functions in a chelating mode and both O atoms also coordinate to adjacent  $\text{Pb}^{\text{II}}$  atoms. The naphthalene-1,5-disulfonate dianion, which lies on a center of inversion, is connected to four  $\text{Pb}^{\text{II}}$  atoms. The bridging modes of the monoanion and dianion give rise to a three-dimensional coordination polymer. The  $\text{Pb}^{\text{II}}$  atom is eight-coordinate in the form of an undefined coordination polyhedron.

## Related literature

For a review of metal arenesulfonates, see: Cai (2004). For an example of a  $\mu_3$ -chelating acetate in a lead(II) system, see: Morsali & Mahjoub (2004).



## Experimental

### Crystal data

$[\text{Pb}_2(\text{C}_2\text{H}_3\text{O}_2)_2(\text{C}_{10}\text{H}_6\text{O}_6\text{S}_2)]$	$V = 893.3$ (7) Å <sup>3</sup>
$M_r = 818.74$	$Z = 2$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 10.442$ (4) Å	$\mu = 19.11$ mm <sup>-1</sup>
$b = 12.666$ (6) Å	$T = 293$ K
$c = 6.803$ (4) Å	$0.26 \times 0.21 \times 0.16$ mm
$\beta = 96.897$ (19)°	

### Data collection

Rigaku R-Axis RAPID IP diffractometer	8599 measured reflections
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	2041 independent reflections
$T_{\text{min}} = 0.083$ , $T_{\text{max}} = 0.150$	1922 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.060$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$	129 parameters
$wR(F^2) = 0.076$	H-atom parameters constrained
$S = 1.05$	$\Delta\rho_{\text{max}} = 2.09$ e Å <sup>-3</sup>
2041 reflections	$\Delta\rho_{\text{min}} = -2.53$ e Å <sup>-3</sup>

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalClear* (Rigaku/MSK, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

This work was supported by the Key Project of the Natural Science Foundation of Heilongjiang Province (No. ZD200903), the Key Project of the Education Bureau of Heilongjiang Province (Nos. 12511z023, 2011CJHB006), the Innovation Team of the Education Bureau of Heilongjiang Province (No. 2010 t d03), Heilongjiang University (Hdtd2010-04), and the Ministry of Higher Education of Malaysia (grant No. UM.C/HIR/MOHE/SC/12).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5522).

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## supporting information

*Acta Cryst.* (2012). E68, m707 [doi:10.1107/S160053681201834X]

**Poly[di- $\mu_3$ -acetato- $\mu_8$ -(naphthalene-1,5-disulfonato)-dilead(II)]****Shan Gao and Seik Weng Ng****S1. Comment**

Metal arenesulfonates are generally crystalline compounds; in some, the metal is connected to the arenesulfonate by a covalent bond whereas in others, the arenesulfonate interacts indirectly with the metal center in an outer-sphere type of coordination (Cai, 2004). A lead(II) derivative of naphthalene-1,5-disulfonate is known in which the coordination number is high. This is attributed to the diverse binding modes of the sulfonate entity. In this example (Morsali & Mahjoub, 2004), the acetate ion engages in  $\mu_3$ -bridging. Both features are observed in polymeric  $\text{Pb}_2(\text{C}_2\text{H}_3\text{O}_2)_2(\text{C}_{10}\text{H}_6\text{O}_6\text{S}_2)$  (Scheme I, Fig. 1). (The acetate anion is a part of the lead(II) acetate reactant.) The acetate ion functions in a chelating mode; both O atoms are further involved in coordinating to adjacent  $\text{Pb}^{\text{II}}$  atoms. The naphthalene-1,5-disulfonate ion lies on a center-of-inversion; the sulfonate  $-\text{SO}_3$  group is connected to four  $\text{Pb}^{\text{II}}$  atoms. The bridging modes of the monoanion and dianion give rise to a three-dimensional coordination polymer (Fig. 1). The  $\text{Pb}^{\text{II}}$  atom is eight-coordinate in an undefined coordination geometry (Fig. 2).

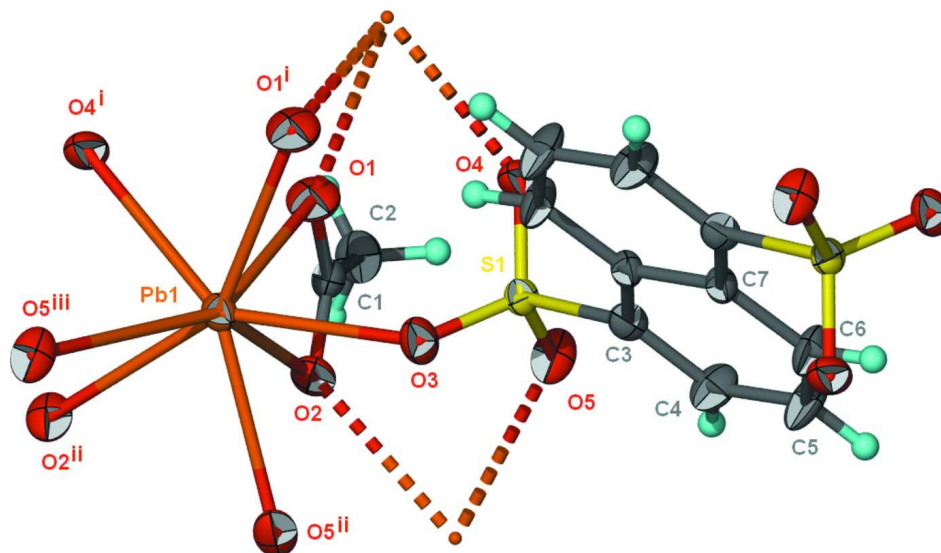
**S2. Experimental**

Equal molar amounts of lead(II) acetate (1 mmol, 327 mg) and naphthalene-1,5-disulfonic acid tetrahydrate (1 mmol, 360 mg) were dissolved in water (10 ml). The solution was filtered and then set aside for the growth of crystals. Colorless crystals separated from solution after several days.

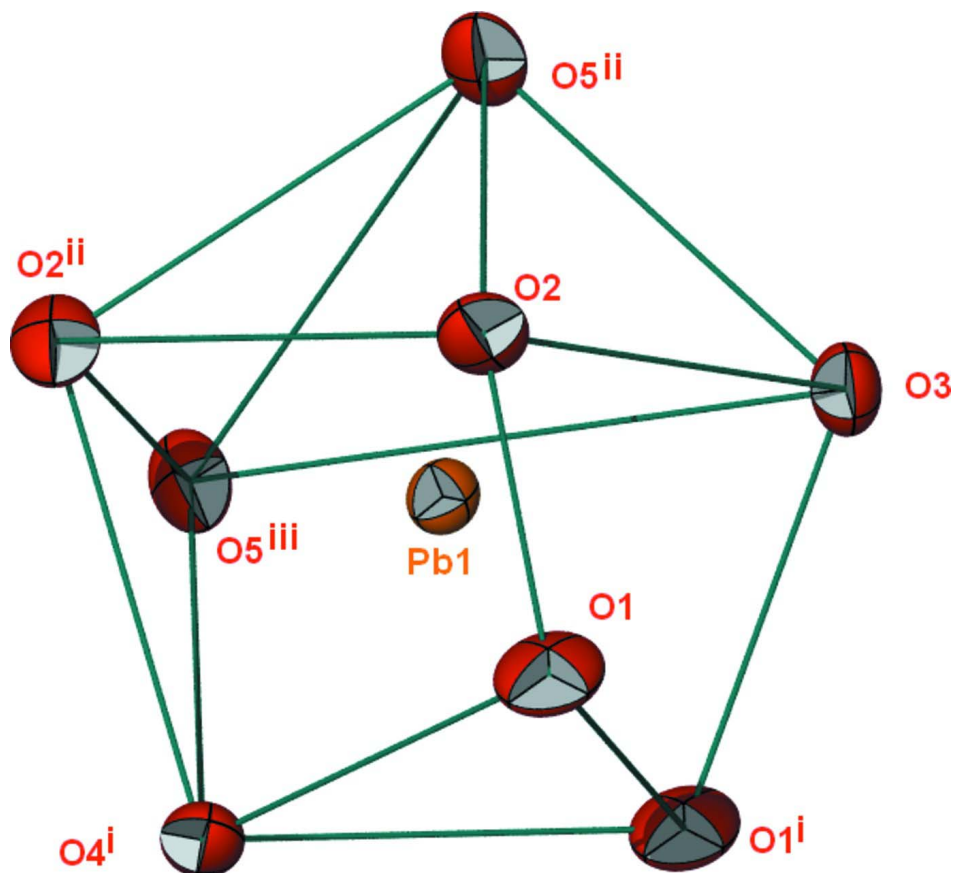
**S3. Refinement**

Carbon- and nitrogen-bound H-atoms were placed in calculated positions (C–H 0.93–0.98 Å) and were included in the refinement in the riding model approximation, with  $U(\text{H})$  set to 1.2–1.5 $U(\text{C})$ .

The final difference Fourier map had a peak in the vicinity of Pb1 as well as a hole in the vicinity of this atom.

**Figure 1**

Thermal ellipsoid plot (Barbour, 2001) illustrating the geometry of Pb<sup>II</sup> of polymeric Pb<sub>2</sub>(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)<sub>2</sub>(C<sub>10</sub>H<sub>6</sub>O<sub>6</sub>S<sub>2</sub>) at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

**Figure 2**

Eight-coordinate geometry of Pb<sup>II</sup>.

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## Crystal data

[Pb<sub>2</sub>(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)<sub>2</sub>(C<sub>10</sub>H<sub>6</sub>O<sub>6</sub>S<sub>2</sub>)] $M_r = 818.74$ Monoclinic,  $P2_1/c$ 

Hall symbol: -P 2ybc

 $a = 10.442$  (4) Å $b = 12.666$  (6) Å $c = 6.803$  (4) Å $\beta = 96.897$  (19)° $V = 893.3$  (7) Å<sup>3</sup> $Z = 2$  $F(000) = 744$  $D_x = 3.044$  Mg m<sup>-3</sup>Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 7836 reflections

 $\theta = 3.2$ – $27.5$ ° $\mu = 19.11$  mm<sup>-1</sup> $T = 293$  K

Prism, brown

 $0.26 \times 0.21 \times 0.16$  mm

## Data collection

Rigaku R-AXIS RAPID IP

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 $\omega$  scan

Absorption correction: multi-scan

(ABSCOR; Higashi, 1995)

 $T_{\min} = 0.083$ ,  $T_{\max} = 0.150$ 

8599 measured reflections

2041 independent reflections

1922 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.060$  $\theta_{\max} = 27.5$ °,  $\theta_{\min} = 3.2$ ° $h = -11 \rightarrow 13$  $k = -15 \rightarrow 16$  $l = -8 \rightarrow 8$ 

## Refinement

Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.030$  $wR(F^2) = 0.076$  $S = 1.05$ 

2041 reflections

129 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

 $w = 1/[\sigma^2(F_o^2) + (0.0383P)^2 + 2.2119P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} = 0.001$  $\Delta\rho_{\max} = 2.09$  e Å<sup>-3</sup> $\Delta\rho_{\min} = -2.53$  e Å<sup>-3</sup>Extinction correction: *SHELXL97* (Sheldrick,2008),  $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$ 

Extinction coefficient: 0.0153 (7)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Pb1	0.465191 (17)	0.638470 (13)	0.64777 (3)	0.01655 (12)
S1	0.30857 (11)	0.58790 (9)	0.14316 (19)	0.0152 (3)
O1	0.6125 (5)	0.5588 (3)	0.4215 (7)	0.0293 (10)
O2	0.5932 (4)	0.7294 (3)	0.3948 (6)	0.0241 (9)
O3	0.2901 (4)	0.6274 (3)	0.3396 (7)	0.0229 (9)
O4	0.3610 (4)	0.4829 (3)	0.1445 (6)	0.0233 (8)
O5	0.3804 (4)	0.6622 (3)	0.0349 (7)	0.0249 (9)
C1	0.6511 (6)	0.6459 (4)	0.3576 (9)	0.0176 (11)
C2	0.7677 (7)	0.6495 (5)	0.2497 (10)	0.0262 (13)
H2A	0.8085	0.7172	0.2692	0.039*
H2B	0.7424	0.6383	0.1110	0.039*
H2C	0.8271	0.5953	0.2999	0.039*

C3	0.1528 (5)	0.5777 (4)	0.0066 (8)	0.0171 (10)
C4	0.1285 (6)	0.6372 (4)	-0.1604 (10)	0.0237 (13)
H4	0.1892	0.6860	-0.1925	0.028*
C5	0.0110 (6)	0.6250 (5)	-0.2852 (10)	0.0286 (14)
H5	-0.0047	0.6654	-0.3997	0.034*
C6	-0.0786 (5)	0.5550 (4)	-0.2391 (9)	0.0232 (12)
H6	-0.1544	0.5466	-0.3245	0.028*
C7	-0.0591 (5)	0.4946 (4)	-0.0642 (7)	0.0149 (10)

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Pb1	0.01896 (16)	0.01700 (16)	0.01406 (16)	-0.00039 (6)	0.00350 (9)	-0.00111 (6)
S1	0.0139 (5)	0.0152 (5)	0.0158 (6)	-0.0021 (4)	-0.0013 (4)	-0.0009 (4)
O1	0.042 (2)	0.0187 (18)	0.029 (3)	-0.0062 (18)	0.0096 (19)	0.0069 (17)
O2	0.027 (2)	0.0229 (18)	0.023 (2)	0.0090 (16)	0.0041 (17)	-0.0050 (16)
O3	0.019 (2)	0.0274 (19)	0.021 (2)	0.0020 (15)	-0.0027 (17)	-0.0056 (15)
O4	0.0222 (18)	0.0194 (17)	0.027 (2)	0.0057 (15)	-0.0022 (16)	-0.0030 (16)
O5	0.0185 (19)	0.0312 (19)	0.025 (2)	-0.0104 (17)	0.0004 (17)	0.0063 (18)
C1	0.020 (3)	0.018 (2)	0.014 (3)	0.0008 (18)	0.000 (2)	0.0008 (18)
C2	0.029 (3)	0.032 (3)	0.020 (3)	0.000 (2)	0.012 (3)	0.001 (2)
C3	0.013 (2)	0.017 (2)	0.021 (3)	0.0008 (19)	0.0000 (19)	-0.003 (2)
C4	0.023 (3)	0.025 (3)	0.024 (3)	-0.0028 (19)	0.004 (2)	0.010 (2)
C5	0.027 (3)	0.034 (3)	0.023 (3)	-0.007 (2)	-0.005 (3)	0.017 (2)
C6	0.020 (3)	0.029 (3)	0.018 (3)	-0.002 (2)	-0.007 (2)	0.006 (2)
C7	0.016 (2)	0.016 (2)	0.012 (3)	0.0002 (18)	0.0012 (19)	-0.0014 (18)

*Geometric parameters (Å, °)*

Pb1—O1	2.515 (5)	O4—Pb1 <sup>i</sup>	2.653 (4)
Pb1—O1 <sup>i</sup>	2.652 (4)	O5—Pb1 <sup>iv</sup>	2.754 (4)
Pb1—O2	2.574 (5)	C1—C2	1.496 (9)
Pb1—O2 <sup>ii</sup>	2.622 (4)	C2—H2A	0.9600
Pb1—O3	2.615 (4)	C2—H2B	0.9600
Pb1—O4 <sup>i</sup>	2.653 (4)	C2—H2C	0.9600
Pb1—O5 <sup>iii</sup>	2.893 (5)	C3—C4	1.361 (8)
Pb1—O5 <sup>ii</sup>	2.754 (4)	C3—C7 <sup>v</sup>	1.429 (7)
S1—O4	1.438 (4)	C4—C5	1.414 (9)
S1—O5	1.457 (4)	C4—H4	0.9300
S1—O3	1.461 (5)	C5—C6	1.353 (8)
S1—C3	1.778 (5)	C5—H5	0.9300
O1—C1	1.269 (6)	C6—C7	1.409 (7)
O1—Pb1 <sup>i</sup>	2.652 (4)	C6—H6	0.9300
O2—C1	1.259 (6)	C7—C3 <sup>v</sup>	1.429 (7)
O2—Pb1 <sup>iv</sup>	2.622 (4)	C7—C7 <sup>v</sup>	1.431 (9)
O1—Pb1—O2	50.61 (14)	C1—O1—Pb1	95.9 (4)
O1—Pb1—O3	84.72 (15)	C1—O1—Pb1 <sup>i</sup>	149.4 (4)

O2—Pb1—O3	81.78 (14)	Pb1—O1—Pb1 <sup>i</sup>	106.87 (16)
O1—Pb1—O2 <sup>ii</sup>	110.26 (14)	C1—O2—Pb1	93.4 (4)
O2—Pb1—O2 <sup>ii</sup>	82.95 (9)	C1—O2—Pb1 <sup>iv</sup>	128.9 (4)
O3—Pb1—O2 <sup>ii</sup>	143.29 (12)	Pb1—O2—Pb1 <sup>iv</sup>	116.65 (15)
O1—Pb1—O1 <sup>i</sup>	73.13 (16)	S1—O3—Pb1	126.5 (2)
O2—Pb1—O1 <sup>i</sup>	118.36 (13)	S1—O4—Pb1 <sup>i</sup>	140.8 (2)
O3—Pb1—O1 <sup>i</sup>	68.75 (13)	S1—O5—Pb1 <sup>iv</sup>	128.3 (3)
O2 <sup>ii</sup> —Pb1—O1 <sup>i</sup>	146.84 (12)	O2—C1—O1	118.8 (6)
O1—Pb1—O4 <sup>i</sup>	70.34 (14)	O2—C1—C2	120.8 (5)
O2—Pb1—O4 <sup>i</sup>	103.77 (13)	O1—C1—C2	120.4 (5)
O3—Pb1—O4 <sup>i</sup>	139.81 (11)	C1—C2—H2A	109.5
O2 <sup>ii</sup> —Pb1—O4 <sup>i</sup>	76.41 (13)	C1—C2—H2B	109.5
O1 <sup>i</sup> —Pb1—O4 <sup>i</sup>	73.99 (13)	H2A—C2—H2B	109.5
O1—Pb1—O5 <sup>ii</sup>	113.41 (13)	C1—C2—H2C	109.5
O2—Pb1—O5 <sup>ii</sup>	65.11 (12)	H2A—C2—H2C	109.5
O3—Pb1—O5 <sup>ii</sup>	69.81 (12)	H2B—C2—H2C	109.5
O2 <sup>ii</sup> —Pb1—O5 <sup>ii</sup>	73.47 (12)	C4—C3—C7 <sup>v</sup>	121.2 (5)
O1 <sup>i</sup> —Pb1—O5 <sup>ii</sup>	137.12 (15)	C4—C3—S1	117.6 (4)
O4 <sup>i</sup> —Pb1—O5 <sup>ii</sup>	148.89 (12)	C7 <sup>v</sup> —C3—S1	121.1 (4)
O1—Pb1—O5 <sup>iii</sup>	151.11 (12)	C3—C4—C5	120.2 (5)
O2—Pb1—O5 <sup>iii</sup>	143.35 (12)	C3—C4—H4	119.9
O3—Pb1—O5 <sup>iii</sup>	118.32 (14)	C5—C4—H4	119.9
O2 <sup>ii</sup> —Pb1—O5 <sup>iii</sup>	62.50 (12)	C6—C5—C4	120.5 (6)
O1 <sup>i</sup> —Pb1—O5 <sup>iii</sup>	98.00 (13)	C6—C5—H5	119.8
O4 <sup>i</sup> —Pb1—O5 <sup>iii</sup>	80.81 (13)	C4—C5—H5	119.8
O5 <sup>ii</sup> —Pb1—O5 <sup>iii</sup>	92.01 (12)	C5—C6—C7	121.2 (5)
O4—S1—O5	112.5 (3)	C5—C6—H6	119.4
O4—S1—O3	113.8 (2)	C7—C6—H6	119.4
O5—S1—O3	111.9 (3)	C6—C7—C3 <sup>v</sup>	123.1 (4)
O4—S1—C3	105.1 (2)	C6—C7—C7 <sup>v</sup>	119.3 (6)
O5—S1—C3	106.0 (2)	C3 <sup>v</sup> —C7—C7 <sup>v</sup>	117.6 (6)
O3—S1—C3	106.9 (3)		
O2—Pb1—O1—C1	6.4 (3)	O2—Pb1—O3—S1	50.2 (3)
O3—Pb1—O1—C1	90.1 (4)	O2 <sup>ii</sup> —Pb1—O3—S1	116.6 (3)
O2 <sup>ii</sup> —Pb1—O1—C1	-55.4 (4)	O1 <sup>i</sup> —Pb1—O3—S1	-74.6 (3)
O1 <sup>i</sup> —Pb1—O1—C1	159.4 (4)	O4 <sup>i</sup> —Pb1—O3—S1	-51.4 (4)
O4 <sup>i</sup> —Pb1—O1—C1	-122.0 (4)	O5 <sup>ii</sup> —Pb1—O3—S1	116.6 (3)
O5 <sup>ii</sup> —Pb1—O1—C1	24.7 (4)	O5 <sup>iii</sup> —Pb1—O3—S1	-162.3 (2)
O5 <sup>iii</sup> —Pb1—O1—C1	-125.0 (3)	O5—S1—O4—Pb1 <sup>i</sup>	88.6 (4)
O2—Pb1—O1—Pb1 <sup>i</sup>	-153.0 (2)	O3—S1—O4—Pb1 <sup>i</sup>	-39.9 (5)
O3—Pb1—O1—Pb1 <sup>i</sup>	-69.36 (16)	C3—S1—O4—Pb1 <sup>i</sup>	-156.4 (4)
O2 <sup>ii</sup> —Pb1—O1—Pb1 <sup>i</sup>	145.14 (13)	O4—S1—O5—Pb1 <sup>iv</sup>	-133.6 (3)
O1 <sup>i</sup> —Pb1—O1—Pb1 <sup>i</sup>	0.0	O3—S1—O5—Pb1 <sup>iv</sup>	-4.0 (4)
O4 <sup>i</sup> —Pb1—O1—Pb1 <sup>i</sup>	78.59 (16)	C3—S1—O5—Pb1 <sup>iv</sup>	112.1 (3)
O5 <sup>ii</sup> —Pb1—O1—Pb1 <sup>i</sup>	-134.68 (15)	Pb1—O2—C1—O1	11.2 (6)
O5 <sup>iii</sup> —Pb1—O1—Pb1 <sup>i</sup>	75.6 (3)	Pb1 <sup>iv</sup> —O2—C1—O1	-117.4 (5)
O1—Pb1—O2—C1	-6.4 (3)	Pb1—O2—C1—C2	-165.4 (5)

O3—Pb1—O2—C1	-96.3 (3)	Pb1 <sup>iv</sup> —O2—C1—C2	65.9 (7)
O2 <sup>ii</sup> —Pb1—O2—C1	117.2 (3)	Pb1—O1—C1—O2	-11.5 (6)
O1 <sup>i</sup> —Pb1—O2—C1	-35.9 (4)	Pb1 <sup>i</sup> —O1—C1—O2	127.1 (7)
O4 <sup>i</sup> —Pb1—O2—C1	43.1 (3)	Pb1—O1—C1—C2	165.1 (5)
O5 <sup>ii</sup> —Pb1—O2—C1	-167.8 (4)	Pb1 <sup>i</sup> —O1—C1—C2	-56.3 (10)
O5 <sup>iii</sup> —Pb1—O2—C1	136.2 (3)	O4—S1—C3—C4	-120.1 (5)
O1—Pb1—O2—Pb1 <sup>iv</sup>	130.8 (2)	O5—S1—C3—C4	-0.8 (5)
O3—Pb1—O2—Pb1 <sup>iv</sup>	40.84 (16)	O3—S1—C3—C4	118.7 (5)
O2 <sup>ii</sup> —Pb1—O2—Pb1 <sup>iv</sup>	-105.7 (2)	O4—S1—C3—C7 <sup>v</sup>	56.5 (5)
O1 <sup>i</sup> —Pb1—O2—Pb1 <sup>iv</sup>	101.22 (19)	O5—S1—C3—C7 <sup>v</sup>	175.8 (4)
O4 <sup>i</sup> —Pb1—O2—Pb1 <sup>iv</sup>	-179.76 (14)	O3—S1—C3—C7 <sup>v</sup>	-64.7 (5)
O5 <sup>ii</sup> —Pb1—O2—Pb1 <sup>iv</sup>	-30.66 (15)	C7 <sup>v</sup> —C3—C4—C5	-2.7 (9)
O5 <sup>iii</sup> —Pb1—O2—Pb1 <sup>iv</sup>	-86.7 (2)	S1—C3—C4—C5	173.9 (5)
O4—S1—O3—Pb1	54.8 (3)	C3—C4—C5—C6	0.6 (10)
O5—S1—O3—Pb1	-74.1 (3)	C4—C5—C6—C7	1.7 (10)
C3—S1—O3—Pb1	170.2 (2)	C5—C6—C7—C3 <sup>v</sup>	177.9 (6)
O1—Pb1—O3—S1	-0.7 (3)	C5—C6—C7—C7 <sup>v</sup>	-1.8 (10)

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