

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

# Tripotassium bis(acetato- $\kappa^2O,O'$ )-(thiocyanato- $\kappa N$ )plumbate(II) dithiocyanate

Seik Weng Ng

Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

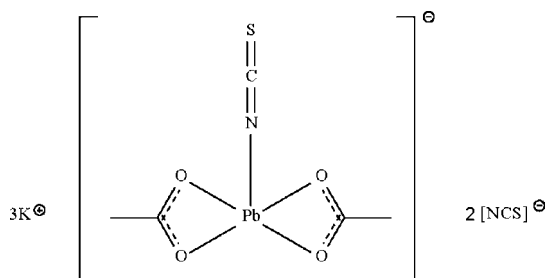
Received 2 June 2009; accepted 5 June 2009

 Key indicators: single-crystal X-ray study;  $T = 140$  K; mean  $\sigma(C-C) = 0.005$  Å;  $R$  factor = 0.022;  $wR$  factor = 0.056; data-to-parameter ratio = 19.7.

In the crystal structure of the title salt,  $K_3[Pb(CH_3COO)_2(NCS)](NCS)_2$ , the  $[Pb(CH_3COO)_2(NCS)]^-$  anion exists as a covalently bonded entity in which the acetate anions chelate in an anisobidentate manner. The Pb atom shows a distorted  $\psi$ -octahedral coordination to four acetate O atoms and one isocyanate N atom, with the stereochemically active lone pair occupying one of the six sites. When the three longer Pb $\cdots$ S interactions are considered, the eight-coordinate geometry is based on a dodecahedron. The  $[Pb(CH_3COO)_2(NCS)]^-$  anion has mirror symmetry. The potassium cations connect the other constituents into a three-dimensional network through electrostatic K $\cdots$ N and K $\cdots$ S interactions.

## Related literature

In  $[K_6Pb_6(CH_3CO_2)_{12}(NCS)_2](NCS)_4$ , the acetate groups link the lead(II) atoms into a chain; see: Morsali & Mahjoub (2004).



## Experimental

## Crystal data

 $K_3[Pb(C_2H_3O_2)_2(NCS)](NCS)_2$ 
 $M_r = 616.82$ 

 Monoclinic,  $P2_1/m$ 
 $a = 6.1968$  (1) Å

 $b = 19.2499$  (3) Å

 $c = 7.6354$  (1) Å

 $\beta = 106.982$  (1) $^\circ$ 
 $V = 871.10$  (2) Å<sup>3</sup>
 $Z = 2$ 

 Mo  $K\alpha$  radiation

 $\mu = 10.77$  mm<sup>-1</sup>
 $T = 140$  K

 $0.25 \times 0.20 \times 0.08$  mm

## Data collection

Bruker SMART APEX

diffractometer

Absorption correction: multi-scan

(SADABS; Sheldrick, 1996)

 $T_{\min} = 0.174$ ,  $T_{\max} = 0.480$ 

(expected range = 0.153–0.422)

6058 measured reflections

2045 independent reflections

 2015 reflections with  $I > 2\sigma(I)$ 
 $R_{\text{int}} = 0.023$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.022$ 
 $wR(F^2) = 0.056$ 
 $S = 1.16$ 

2045 reflections

104 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.81$  e Å<sup>-3</sup>
 $\Delta\rho_{\text{min}} = -2.00$  e Å<sup>-3</sup>

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; 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: *pubCIF* (Westrip, 2009).

I thank Dr Maryam Ranjbar of the Iranian Research Organization for Science and Technology for providing the crystal, and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2472).

## References

- Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.  
 Bruker (2008). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Morsali, A. & Mahjoub, A. R. (2004). *Helv. Chim. Acta*, **87**, 2717–2722.  
 Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.  
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
 Westrip, S. P. (2009). *pubCIF*. In preparation.

**supplementary materials**

*Acta Cryst.* (2009). E65, m755 [ doi:10.1107/S1600536809021370 ]

**Tripotassium bis(acetato- $\kappa^2O,O'$ )(thiocyanato- $\kappa N$ )plumbate(II) dithiocyanate**

S. W. Ng

**Experimental**

2,4-Diamino-6-(1-piperidinyl)-2,4-pyrimidine *N*-oxide (commercial name: minoxidil) (0.5 mmol, 0.10 g), lead(II) acetate (0.5 mmol, 0.17 g) and potassium thiocyanate (1 mmol, 0.10 g) were dissolved in methanol (100 ml). The solution was concentrated to a smaller volume and this was mixed with a similar volume of chloroform. Colorless prisms separated after several days.

**Refinement**

Hydrogen atoms were placed at calculated positions (C–H 0.98 Å) and were treated as riding on the carbon atom with  $U(H)$  set to  $1.5U_{eq}(C)$ . The final difference Fourier map had a deep hole at 1 Å from N1.

**Figures**

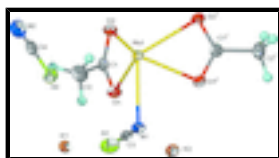


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of a portion of the polymeric structure of  $K_3Pb(NCS)_3(CH_3CO_2)_2$  at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius. Symmetry operation *i*:  $x, 1.5-y, z$ .

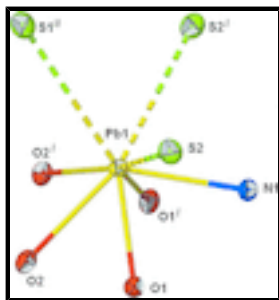


Fig. 2. Detail showing the dodecahedral geometry of the Pb(II) atom.

**Tripotassium bis(acetato- $\kappa^2O,O'$ )(thiocyanato- $\kappa N$ )plumbate(II) dithiocyanate**

*Crystal data*

$K_3[Pb(C_2H_3O_2)_2(NCS)](NCS)_2$

$M_r = 616.82$

Monoclinic,  $P2_1/m$

Hall symbol: -P 2yb

$a = 6.1968$  (1) Å

$b = 19.2499$  (3) Å

$F_{000} = 576$

$D_x = 2.352$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 4971 reflections

$\theta = 2.9$ – $28.3^\circ$

$\mu = 10.77$  mm<sup>-1</sup>

# supplementary materials

---

$c = 7.6354 (1) \text{ \AA}$   
 $\beta = 106.982 (1)^\circ$   
 $V = 871.10 (2) \text{ \AA}^3$   
 $Z = 2$

$T = 140 \text{ K}$   
Block, colorless  
 $0.25 \times 0.20 \times 0.08 \text{ mm}$

## Data collection

Bruker SMART APEX diffractometer  
Radiation source: fine-focus sealed tube  
Monochromator: graphite  
 $T = 140 \text{ K}$   
 $\omega$  scans  
Absorption correction: Multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.174, T_{\max} = 0.480$   
6058 measured reflections

2045 independent reflections  
2015 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.023$   
 $\theta_{\max} = 27.5^\circ$   
 $\theta_{\min} = 2.1^\circ$   
 $h = -7 \rightarrow 8$   
 $k = -25 \rightarrow 25$   
 $l = -9 \rightarrow 9$

## Refinement

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.022$   
 $wR(F^2) = 0.056$   
 $S = 1.16$   
2045 reflections  
104 parameters  
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  
 $w = 1/[\sigma^2(F_o^2) + (0.03P)^2 + 1.3438P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.81 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -2.00 \text{ e \AA}^{-3}$   
Extinction correction: none

## Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Pb1	0.41686 (3)	0.7500	0.47400 (2)	0.01158 (7)
K1	0.85392 (14)	0.59423 (4)	0.42518 (12)	0.01948 (17)
K2	0.74409 (19)	0.7500	0.09371 (15)	0.0160 (2)
S1	1.1475 (2)	0.7500	0.8492 (2)	0.0247 (3)
S2	0.65682 (16)	0.62677 (5)	0.76528 (14)	0.01949 (19)
N1	0.8449 (7)	0.7500	0.4972 (6)	0.0151 (9)
N2	0.2738 (6)	0.54336 (19)	0.6214 (6)	0.0289 (8)
O1	0.4696 (4)	0.66654 (13)	0.2532 (4)	0.0162 (5)
O2	0.1161 (4)	0.66405 (13)	0.2588 (4)	0.0171 (5)
C1	0.2743 (6)	0.63941 (18)	0.2063 (5)	0.0146 (7)
C2	0.2360 (7)	0.5741 (2)	0.0917 (6)	0.0236 (8)
H2A	0.2283	0.5341	0.1689	0.035*
H2B	0.3609	0.5678	0.0388	0.035*

H2C	0.0940	0.5782	-0.0069	0.035*
C3	0.9724 (8)	0.7500	0.6432 (7)	0.0142 (10)
C4	0.4309 (6)	0.57822 (19)	0.6821 (5)	0.0180 (7)

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Pb1	0.01294 (11)	0.01291 (10)	0.01004 (11)	0.000	0.00516 (7)	0.000
K1	0.0164 (4)	0.0153 (4)	0.0286 (4)	0.0018 (3)	0.0095 (3)	0.0037 (3)
K2	0.0168 (5)	0.0192 (5)	0.0129 (5)	0.000	0.0058 (4)	0.000
S1	0.0184 (6)	0.0388 (8)	0.0144 (6)	0.000	0.0011 (5)	0.000
S2	0.0194 (4)	0.0185 (4)	0.0210 (5)	-0.0041 (3)	0.0065 (4)	-0.0016 (4)
N1	0.0119 (19)	0.020 (2)	0.013 (2)	0.000	0.0035 (17)	0.000
N2	0.0217 (18)	0.0232 (17)	0.036 (2)	-0.0019 (14)	0.0000 (16)	0.0014 (15)
O1	0.0136 (12)	0.0192 (12)	0.0162 (13)	-0.0004 (9)	0.0050 (10)	-0.0031 (10)
O2	0.0142 (12)	0.0194 (12)	0.0191 (13)	0.0017 (9)	0.0071 (10)	-0.0002 (10)
C1	0.0171 (16)	0.0143 (15)	0.0107 (16)	0.0017 (12)	0.0015 (13)	0.0049 (13)
C2	0.0234 (19)	0.0199 (18)	0.025 (2)	-0.0010 (15)	0.0034 (17)	-0.0079 (16)
C3	0.012 (2)	0.012 (2)	0.020 (3)	0.000	0.008 (2)	0.000
C4	0.0175 (18)	0.0142 (16)	0.0205 (19)	0.0032 (13)	0.0031 (15)	0.0034 (14)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Pb1—O1	2.419 (3)	K2—S2 <sup>vi</sup>	3.3788 (13)
Pb1—O1 <sup>i</sup>	2.419 (3)	K2—S2 <sup>vii</sup>	3.3788 (13)
Pb1—O2	2.669 (3)	S1—C3	1.630 (5)
Pb1—O2 <sup>i</sup>	2.669 (3)	S2—C4	1.649 (4)
Pb1—N1	2.606 (4)	S2—K2 <sup>viii</sup>	3.3788 (13)
Pb1—S1 <sup>ii</sup>	3.713 (1)	N1—C3	1.163 (7)
Pb1—S2	3.294 (1)	N1—K1 <sup>i</sup>	3.0520 (12)
Pb1—S2 <sup>i</sup>	3.294 (1)	N2—C4	1.162 (5)
K1—O2 <sup>iii</sup>	2.696 (3)	N2—K1 <sup>iv</sup>	2.757 (4)
K1—O1	2.740 (3)	N2—K1 <sup>ii</sup>	2.774 (4)
K1—N2 <sup>iv</sup>	2.757 (4)	O1—C1	1.270 (4)
K1—N2 <sup>iii</sup>	2.774 (4)	O2—C1	1.255 (5)
K1—N1	3.0520 (12)	O2—K1 <sup>ii</sup>	2.696 (3)
K1—S2	3.2373 (13)	O2—K2 <sup>ii</sup>	2.818 (3)
K2—O2 <sup>v</sup>	2.818 (3)	C1—C2	1.509 (5)
K2—O2 <sup>iii</sup>	2.818 (3)	C2—H2A	0.9800
K2—O1	2.858 (3)	C2—H2B	0.9800
K2—O1 <sup>i</sup>	2.858 (3)	C2—H2C	0.9800
K2—N1	2.959 (5)	C3—K1 <sup>i</sup>	3.404 (3)
O1—Pb1—O1 <sup>i</sup>	83.23 (13)	O2 <sup>v</sup> —K2—S1 <sup>ix</sup>	143.98 (5)
O1—Pb1—N1	72.87 (9)	O2 <sup>iii</sup> —K2—S1 <sup>ix</sup>	143.98 (5)
O1 <sup>i</sup> —Pb1—N1	72.87 (9)	O1—K2—S1 <sup>ix</sup>	64.83 (6)

## supplementary materials

O1—Pb1—O2	51.04 (8)	O1 <sup>i</sup> —K2—S1 <sup>ix</sup>	64.83 (6)
O1 <sup>i</sup> —Pb1—O2	101.23 (8)	N1—K2—S1 <sup>ix</sup>	114.20 (9)
N1—Pb1—O2	123.76 (9)	S2 <sup>vi</sup> —K2—S1 <sup>ix</sup>	72.51 (3)
O1—Pb1—O2 <sup>i</sup>	101.23 (8)	S2 <sup>vii</sup> —K2—S1 <sup>ix</sup>	72.51 (3)
O1 <sup>i</sup> —Pb1—O2 <sup>i</sup>	51.04 (8)	S1 <sup>vi</sup> —K2—S1 <sup>ix</sup>	120.08 (5)
N1—Pb1—O2 <sup>i</sup>	123.76 (9)	C3—S1—K2 <sup>viii</sup>	97.79 (19)
O2—Pb1—O2 <sup>i</sup>	76.62 (11)	C3—S1—K2 <sup>x</sup>	142.1 (2)
O2 <sup>iii</sup> —K1—O1	94.59 (8)	K2 <sup>viii</sup> —S1—K2 <sup>x</sup>	120.08 (5)
O2 <sup>iii</sup> —K1—N2 <sup>iv</sup>	127.44 (11)	C4—S2—K1	93.34 (15)
O1—K1—N2 <sup>iv</sup>	104.45 (9)	C4—S2—K2 <sup>viii</sup>	128.15 (15)
O2 <sup>iii</sup> —K1—N2 <sup>iii</sup>	80.28 (10)	K1—S2—K2 <sup>viii</sup>	135.65 (4)
O1—K1—N2 <sup>iii</sup>	170.07 (10)	C3—N1—Pb1	117.3 (4)
N2 <sup>iv</sup> —K1—N2 <sup>iii</sup>	85.36 (11)	C3—N1—K2	151.1 (4)
O2 <sup>iii</sup> —K1—N1	68.92 (10)	Pb1—N1—K2	91.61 (13)
O1—K1—N1	61.85 (10)	C3—N1—K1	97.45 (9)
N2 <sup>iv</sup> —K1—N1	161.31 (12)	Pb1—N1—K1	93.42 (8)
N2 <sup>iii</sup> —K1—N1	108.25 (11)	K2—N1—K1	79.74 (8)
O2 <sup>iii</sup> —K1—S2	134.60 (6)	C3—N1—K1 <sup>i</sup>	97.45 (9)
O1—K1—S2	78.90 (6)	Pb1—N1—K1 <sup>i</sup>	93.42 (8)
N2 <sup>iv</sup> —K1—S2	97.35 (9)	K2—N1—K1 <sup>i</sup>	79.74 (8)
N2 <sup>iii</sup> —K1—S2	98.58 (9)	K1—N1—K1 <sup>i</sup>	158.52 (17)
N1—K1—S2	68.55 (9)	C4—N2—K1 <sup>iv</sup>	141.4 (3)
O2 <sup>v</sup> —K2—O2 <sup>iii</sup>	71.90 (11)	C4—N2—K1 <sup>ii</sup>	123.8 (3)
O2 <sup>v</sup> —K2—O1	130.58 (9)	K1 <sup>iv</sup> —N2—K1 <sup>ii</sup>	94.64 (11)
O2 <sup>iii</sup> —K2—O1	89.45 (8)	C1—O1—Pb1	99.1 (2)
O2 <sup>v</sup> —K2—O1 <sup>i</sup>	89.45 (8)	C1—O1—K1	123.3 (2)
O2 <sup>iii</sup> —K2—O1 <sup>i</sup>	130.58 (9)	Pb1—O1—K1	106.18 (9)
O1—K2—O1 <sup>i</sup>	68.41 (10)	C1—O1—K2	138.4 (2)
O2 <sup>v</sup> —K2—N1	68.78 (9)	Pb1—O1—K2	98.16 (8)
O2 <sup>iii</sup> —K2—N1	68.78 (9)	K1—O1—K2	86.99 (8)
O1—K2—N1	61.80 (9)	C1—O2—Pb1	87.8 (2)
O1 <sup>i</sup> —K2—N1	61.80 (9)	C1—O2—K1 <sup>ii</sup>	126.6 (2)
O2 <sup>v</sup> —K2—S2 <sup>vi</sup>	132.59 (7)	Pb1—O2—K1 <sup>ii</sup>	115.30 (10)
O2 <sup>iii</sup> —K2—S2 <sup>vi</sup>	81.51 (6)	C1—O2—K2 <sup>ii</sup>	133.8 (2)
O1—K2—S2 <sup>vi</sup>	86.29 (6)	Pb1—O2—K2 <sup>ii</sup>	104.14 (8)
O1 <sup>i</sup> —K2—S2 <sup>vi</sup>	136.45 (7)	K1 <sup>ii</sup> —O2—K2 <sup>ii</sup>	88.66 (8)
N1—K2—S2 <sup>vi</sup>	135.40 (2)	O2—C1—O1	121.6 (3)
O2 <sup>v</sup> —K2—S2 <sup>vii</sup>	81.51 (6)	O2—C1—C2	119.6 (3)
O2 <sup>iii</sup> —K2—S2 <sup>vii</sup>	132.59 (7)	O1—C1—C2	118.8 (3)
O1—K2—S2 <sup>vii</sup>	136.45 (6)	C1—C2—H2A	109.5
O1 <sup>i</sup> —K2—S2 <sup>vii</sup>	86.29 (6)	C1—C2—H2B	109.5

N1—K2—S2 <sup>vii</sup>	135.40 (2)	H2A—C2—H2B	109.5
S2 <sup>vi</sup> —K2—S2 <sup>vii</sup>	89.19 (4)	C1—C2—H2C	109.5
O2 <sup>v</sup> —K2—S1 <sup>vi</sup>	67.87 (6)	H2A—C2—H2C	109.5
O2 <sup>iii</sup> —K2—S1 <sup>vi</sup>	67.87 (6)	H2B—C2—H2C	109.5
O1—K2—S1 <sup>vi</sup>	145.78 (5)	N1—C3—S1	179.1 (5)
O1 <sup>i</sup> —K2—S1 <sup>vi</sup>	145.78 (5)	N1—C3—K1 <sup>i</sup>	62.76 (9)
N1—K2—S1 <sup>vi</sup>	125.72 (9)	S1—C3—K1 <sup>i</sup>	117.37 (8)
S2 <sup>vi</sup> —K2—S1 <sup>vi</sup>	65.92 (3)	N2—C4—S2	178.7 (4)
S2 <sup>vii</sup> —K2—S1 <sup>vi</sup>	65.92 (3)		
O2 <sup>iii</sup> —K1—S2—C4	150.92 (15)	O1 <sup>i</sup> —Pb1—O1—C1	-114.5 (2)
O1—K1—S2—C4	65.45 (14)	N1—Pb1—O1—C1	171.4 (2)
N2 <sup>iv</sup> —K1—S2—C4	-37.91 (15)	O2—Pb1—O1—C1	-4.14 (19)
N2 <sup>iii</sup> —K1—S2—C4	-124.30 (15)	O2 <sup>i</sup> —Pb1—O1—C1	-66.5 (2)
N1—K1—S2—C4	129.38 (15)	O1 <sup>i</sup> —Pb1—O1—K1	116.73 (7)
O2 <sup>iii</sup> —K1—S2—K2 <sup>viii</sup>	-9.98 (11)	N1—Pb1—O1—K1	42.61 (10)
O1—K1—S2—K2 <sup>viii</sup>	-95.45 (7)	O2—Pb1—O1—K1	-132.90 (14)
N2 <sup>iv</sup> —K1—S2—K2 <sup>viii</sup>	161.19 (9)	O2 <sup>i</sup> —Pb1—O1—K1	164.73 (9)
N2 <sup>iii</sup> —K1—S2—K2 <sup>viii</sup>	74.80 (9)	O1 <sup>i</sup> —Pb1—O1—K2	27.51 (11)
N1—K1—S2—K2 <sup>viii</sup>	-31.52 (10)	N1—Pb1—O1—K2	-46.61 (9)
O1—Pb1—N1—C3	-135.98 (7)	O2—Pb1—O1—K2	137.88 (13)
O1 <sup>i</sup> —Pb1—N1—C3	135.98 (7)	O2 <sup>i</sup> —Pb1—O1—K2	75.51 (9)
O2—Pb1—N1—C3	-131.78 (8)	O2 <sup>iii</sup> —K1—O1—C1	145.2 (3)
O2 <sup>i</sup> —Pb1—N1—C3	131.78 (8)	N2 <sup>iv</sup> —K1—O1—C1	14.7 (3)
O1—Pb1—N1—K2	44.02 (7)	N1—K1—O1—C1	-151.6 (3)
O1 <sup>i</sup> —Pb1—N1—K2	-44.02 (7)	S2—K1—O1—C1	-80.2 (3)
O2—Pb1—N1—K2	48.22 (8)	O2 <sup>iii</sup> —K1—O1—Pb1	-101.92 (10)
O2 <sup>i</sup> —Pb1—N1—K2	-48.22 (8)	N2 <sup>iv</sup> —K1—O1—Pb1	127.50 (12)
O1—Pb1—N1—K1	-35.79 (10)	N1—K1—O1—Pb1	-38.80 (11)
O1 <sup>i</sup> —Pb1—N1—K1	-123.83 (13)	S2—K1—O1—Pb1	32.68 (7)
O2—Pb1—N1—K1	-31.60 (15)	O2 <sup>iii</sup> —K1—O1—K2	-4.30 (8)
O2 <sup>i</sup> —Pb1—N1—K1	-128.03 (8)	N2 <sup>iv</sup> —K1—O1—K2	-134.88 (10)
O1—Pb1—N1—K1 <sup>i</sup>	123.83 (13)	N1—K1—O1—K2	58.82 (11)
O1 <sup>i</sup> —Pb1—N1—K1 <sup>i</sup>	35.79 (10)	S2—K1—O1—K2	130.30 (6)
O2—Pb1—N1—K1 <sup>i</sup>	128.03 (8)	O2 <sup>v</sup> —K2—O1—C1	158.4 (3)
O2 <sup>i</sup> —Pb1—N1—K1 <sup>i</sup>	31.60 (15)	O2 <sup>iii</sup> —K2—O1—C1	-136.3 (3)
O2 <sup>v</sup> —K2—N1—C3	-39.03 (6)	O1 <sup>i</sup> —K2—O1—C1	89.0 (3)
O2 <sup>iii</sup> —K2—N1—C3	39.03 (6)	N1—K2—O1—C1	157.7 (4)
O1—K2—N1—C3	140.36 (6)	S2 <sup>vi</sup> —K2—O1—C1	-54.7 (3)
O1 <sup>i</sup> —K2—N1—C3	-140.36 (7)	S2 <sup>vii</sup> —K2—O1—C1	30.2 (4)
S2 <sup>vi</sup> —K2—N1—C3	90.78 (10)	S1 <sup>vi</sup> —K2—O1—C1	-89.4 (4)
S2 <sup>vii</sup> —K2—N1—C3	-90.78 (10)	S1 <sup>ix</sup> —K2—O1—C1	17.7 (3)

## supplementary materials

---

S1 <sup>vi</sup> —K2—N1—C3	0.000 (2)	O2 <sup>v</sup> —K2—O1—Pb1	44.69 (13)
S1 <sup>ix</sup> —K2—N1—C3	180.000 (3)	O2 <sup>iii</sup> —K2—O1—Pb1	110.02 (9)
O2 <sup>v</sup> —K2—N1—Pb1	140.97 (6)	O1 <sup>i</sup> —K2—O1—Pb1	-24.68 (10)
O2 <sup>iii</sup> —K2—N1—Pb1	-140.97 (6)	N1—K2—O1—Pb1	43.94 (8)
O1—K2—N1—Pb1	-39.64 (6)	S2 <sup>vi</sup> —K2—O1—Pb1	-168.45 (8)
O1 <sup>i</sup> —K2—N1—Pb1	39.64 (6)	S2 <sup>vii</sup> —K2—O1—Pb1	-83.54 (10)
S2 <sup>vi</sup> —K2—N1—Pb1	-89.22 (10)	S1 <sup>vi</sup> —K2—O1—Pb1	156.87 (8)
S2 <sup>vii</sup> —K2—N1—Pb1	89.22 (10)	S1 <sup>ix</sup> —K2—O1—Pb1	-96.05 (8)
S1 <sup>vi</sup> —K2—N1—Pb1	180.0	O2 <sup>v</sup> —K2—O1—K1	-61.24 (11)
S1 <sup>ix</sup> —K2—N1—Pb1	0.0	O2 <sup>iii</sup> —K2—O1—K1	4.10 (8)
O2 <sup>v</sup> —K2—N1—K1	-125.85 (11)	O1 <sup>i</sup> —K2—O1—K1	-130.60 (4)
O2 <sup>iii</sup> —K2—N1—K1	-47.79 (8)	N1—K2—O1—K1	-61.98 (8)
O1—K2—N1—K1	53.55 (7)	S2 <sup>vi</sup> —K2—O1—K1	85.63 (6)
O1 <sup>i</sup> —K2—N1—K1	132.82 (11)	S2 <sup>vii</sup> —K2—O1—K1	170.54 (5)
S2 <sup>vi</sup> —K2—N1—K1	3.96 (16)	S1 <sup>vi</sup> —K2—O1—K1	50.94 (14)
S2 <sup>vii</sup> —K2—N1—K1	-177.60 (6)	S1 <sup>ix</sup> —K2—O1—K1	158.03 (8)
S1 <sup>vi</sup> —K2—N1—K1	-86.82 (7)	O1—Pb1—O2—C1	4.13 (19)
S1 <sup>ix</sup> —K2—N1—K1	93.18 (7)	O1 <sup>i</sup> —Pb1—O2—C1	75.8 (2)
O2 <sup>v</sup> —K2—N1—K1 <sup>i</sup>	47.79 (8)	N1—Pb1—O2—C1	-1.0 (2)
O2 <sup>iii</sup> —K2—N1—K1 <sup>i</sup>	125.85 (11)	O2 <sup>i</sup> —Pb1—O2—C1	120.85 (18)
O1—K2—N1—K1 <sup>i</sup>	-132.82 (11)	O1—Pb1—O2—K1 <sup>ii</sup>	134.06 (15)
O1 <sup>i</sup> —K2—N1—K1 <sup>i</sup>	-53.55 (7)	O1 <sup>i</sup> —Pb1—O2—K1 <sup>ii</sup>	-154.31 (10)
S2 <sup>vi</sup> —K2—N1—K1 <sup>i</sup>	177.60 (6)	N1—Pb1—O2—K1 <sup>ii</sup>	128.90 (11)
S2 <sup>vii</sup> —K2—N1—K1 <sup>i</sup>	-3.96 (16)	O2 <sup>i</sup> —Pb1—O2—K1 <sup>ii</sup>	-109.23 (8)
S1 <sup>vi</sup> —K2—N1—K1 <sup>i</sup>	86.82 (7)	O1—Pb1—O2—K2 <sup>ii</sup>	-130.64 (13)
S1 <sup>ix</sup> —K2—N1—K1 <sup>i</sup>	-93.18 (7)	O1 <sup>i</sup> —Pb1—O2—K2 <sup>ii</sup>	-59.01 (10)
O2 <sup>iii</sup> —K1—N1—C3	-100.2 (4)	N1—Pb1—O2—K2 <sup>ii</sup>	-135.80 (10)
O1—K1—N1—C3	152.1 (4)	O2 <sup>i</sup> —Pb1—O2—K2 <sup>ii</sup>	-13.92 (12)
N2 <sup>iv</sup> —K1—N1—C3	106.4 (5)	Pb1—O2—C1—O1	-7.2 (3)
N2 <sup>iii</sup> —K1—N1—C3	-28.7 (4)	K1 <sup>ii</sup> —O2—C1—O1	-127.5 (3)
S2—K1—N1—C3	63.5 (3)	K2 <sup>ii</sup> —O2—C1—O1	100.3 (4)
O2 <sup>iii</sup> —K1—N1—Pb1	141.70 (14)	Pb1—O2—C1—C2	170.0 (3)
O1—K1—N1—Pb1	34.03 (9)	K1 <sup>ii</sup> —O2—C1—C2	49.7 (4)
N2 <sup>iv</sup> —K1—N1—Pb1	-11.7 (4)	K2 <sup>ii</sup> —O2—C1—C2	-82.5 (4)
N2 <sup>iii</sup> —K1—N1—Pb1	-146.82 (12)	Pb1—O1—C1—O2	8.0 (4)
S2—K1—N1—Pb1	-54.57 (9)	K1—O1—C1—O2	124.4 (3)
O2 <sup>iii</sup> —K1—N1—K2	50.68 (9)	K2—O1—C1—O2	-105.3 (4)
O1—K1—N1—K2	-56.99 (9)	Pb1—O1—C1—C2	-169.2 (3)
N2 <sup>iv</sup> —K1—N1—K2	-102.7 (4)	K1—O1—C1—C2	-52.9 (4)
N2 <sup>iii</sup> —K1—N1—K2	122.16 (11)	K2—O1—C1—C2	77.4 (4)
S2—K1—N1—K2	-145.59 (10)	Pb1—N1—C3—K1 <sup>i</sup>	-97.76 (12)

O2 <sup>iii</sup> —K1—N1—K1 <sup>i</sup>	33.4 (4)	K2—N1—C3—K1 <sup>i</sup>	82.24 (12)
O1—K1—N1—K1 <sup>i</sup>	-74.3 (4)	K1—N1—C3—K1 <sup>i</sup>	164.5 (2)
N2 <sup>iv</sup> —K1—N1—K1 <sup>i</sup>	-120.0 (5)	K2 <sup>viii</sup> —S1—C3—K1 <sup>i</sup>	97.27 (19)
N2 <sup>iii</sup> —K1—N1—K1 <sup>i</sup>	104.8 (4)	K2 <sup>x</sup> —S1—C3—K1 <sup>i</sup>	-82.73 (19)
S2—K1—N1—K1 <sup>i</sup>	-162.9 (5)		

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



Fig. 2

