

Crystal structure of tetrakis[μ_2 -2-(dimethylamino)-ethanolato- $\kappa^3 N, O:O$]di- μ_3 -hydroxido-dithiocyanato- $\kappa^2 N$ -dichromium(III)dilead(II) dithiocyanate acetonitrile monosolvate

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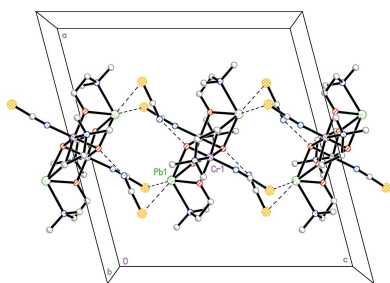
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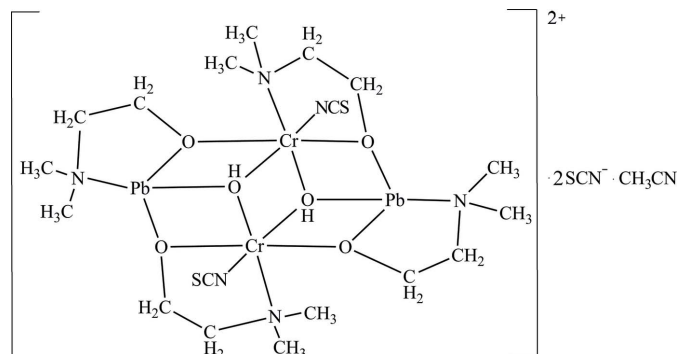
The tetranuclear complex cation of the title compound, [Cr₂Pb₂(NCS)₂(OH)₂(C₄H₁₀NO)₄](SCN)₂·CH₃CN, lies on an inversion centre. The main structural feature of the cation is a distorted seco-norcupane Pb₂Cr₂O₆ cage with a central four-membered Cr₂O₂ ring. The Cr^{III} ion is coordinated in a distorted octahedron, which involves two N atoms of one bidentate ligand and one thiocyanate anion, two μ_2 -O atoms of 2-(dimethylamino)ethanolate ligands and two μ_3 -O atoms of hydroxide ions. The coordination geometry of the Pb^{II} ion is a distorted disphenoid, which involves one N atom, two μ_2 -O atoms and one μ_3 -O atom. In addition, weak Pb \cdots S interactions involving the coordinating and non-coordinating thiocyanate anions are observed. In the crystal, the complex cations are linked through the thiocyanate anions *via* the Pb \cdots S interactions and O—H \cdots N hydrogen bonds into chains along the *c* axis. The chains are further linked together *via* S \cdots S contacts. The contribution of the disordered solvent acetonitrile molecule was removed with the SQUEEZE [Spek (2015). *Acta Cryst. C* **71**, 9–18] procedure in PLATON. The solvent is included in the reported molecular formula, weight and density.

1. Chemical context

There is considerable interest in polynuclear heterometallic complexes as a result of their potential for interesting physicochemical properties such as magnetic (Gheorghe *et al.*, 2010), catalytic (Trettenhahn *et al.*, 2006) and useful light- and/or redox-induced functions (Balzani *et al.*, 2009). The interest currently paid to the synthesis of polynuclear transition metal complexes is stimulated, in particular, by attempts to design and construct multicomponent systems. Despite of a lot of work already done in this field, a limited number of synthetic strategies have been developed to date. Spontaneous self-assembly of Schiff base ligands or rigid building blocks appears to be an extremely powerful tool for the construction of novel polynuclear assemblies incorporating metal atoms by utilizing the various coordination modes of small and flexible ligands (Buvaylo *et al.*, 2005; Kirillov *et al.*, 2005). Metal powders have been successfully applied in direct synthesis of coordination compounds to yield a number of novel monometallic (Babich *et al.*, 1996) and heterometallic complexes (Buvaylo *et al.*, 2005) of various composition, nuclearities and dimensionalities. This work is a continuation of our investigations in the field of direct synthesis of heterometallic coordination compounds based on spontaneous self-assembly,



in which one of the metals is introduced as a powder (zero-valent state) and oxidized during the synthesis (Nesterov *et al.*, 2011), in particular the application of Reinecke's salt in direct synthesis of heterometallic complexes (Nikitina *et al.*, 2008).



2. Structural commentary

The complex cation with a distorted seco-norcupane $\text{Pb}_2\text{Cr}_2\text{O}_6$ framework is centrosymmetric, as shown in Fig. 1. The two crystallographically independent dimethylaminoethanol ligands form five-membered chelate rings with the Cr^{III} and Pb^{II} ions. The Cr^{III} ion adopts a distorted octahedral coordination environment with one N atom and two $\mu_2\text{-O}$ atoms from the dimethylaminoethanol ligands and one $\mu_3\text{-O}$ atom from the hydroxide ion in the equatorial plane, and one N atom of the thiocyanate ion and one $\mu_3\text{-O}$ atom of the second hydroxide ion in the axial positions. The Cr–O and Cr–N bond lengths are 1.950 (3)–1.993 (3) Å and 2.008 (4)–2.158 (4) Å, respectively, and the N–Cr–O and O–Cr–O angles are 79.10 (11)–93.48 (12)° for *cis*-positions and 168.63 (13)–173.46 (12)° for *trans*-positions. The Pb^{II} ion is

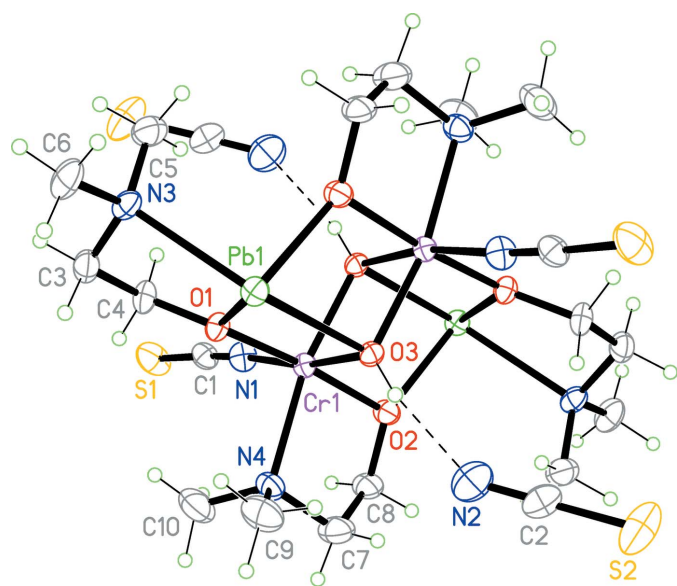


Figure 1
The molecular structure of the title compound, shown with 30% probability displacement ellipsoids. O–H···N hydrogen bonds are shown as dashed lines.

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

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
O3–H3···N2	0.82	1.95	2.757 (6)	169

tetracoordinated by the one $\mu_3\text{-O}$ atom of the hydroxide ion, one N atom and two $\mu_2\text{-O}$ atoms of the dimethylaminoethanol ligands and adopts a distorted disphenoidal coordination. There are additional weak $\text{Pb}\cdots\text{S}$ interactions [$\text{Pb}1\cdots\text{S}1$ 3.2749 (14) Å and $\text{Pb}1\cdots\text{S}2$ 3.4056 (16) Å], and thus the coordination geometry of the Pb^{II} ion can be considered as a strongly distorted trigonal prism, if these interactions are included. The Pb–O bond lengths [2.308 (3)–2.686 (3) Å] as well as the Pb–N distance [2.547 (4) Å] are in a good agreement with literature values. In general, all geometric parameters of the title complex cation are in good agreement with those in related aminoalcohol complexes (Shahid *et al.*, 2011).

3. Supramolecular features

In the crystal, the tetranuclear complex cations are linked through thiocyanate anions with the above-mentioned intermolecular $\text{Pb}\cdots\text{S}$ interactions and by an O–H···N hydrogen bond (Table 1) into chains along the *c* axis (Fig. 2). The chains are further linked together by an S···S sigma-hole bond [$\text{S}1\cdots\text{S}2$ 3.585 (2) Å], where atom S2 acts as a lone-pair donor.

4. Database survey

A search of the Cambridge Structural Database (Version 5.36; last update February 2015; Groom & Allen, 2014) for related complexes with 2-dimethylaminoethanol gave 260 hits, including some closely related structures with a distorted seco-norcupane cage with Ti (Hollingsworth *et al.*, 2008), Ge(Sn)–

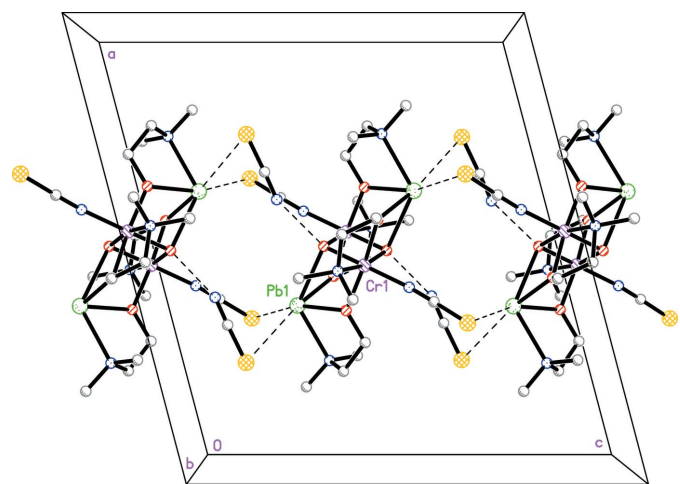


Figure 2
Crystal packing diagram of the title compound, viewed along the *b* axis. $\text{Pb}\cdots\text{S}$ contacts and O–H···N hydrogen bonds are shown as dashed lines.

Table 2
Experimental details.

Crystal data	
Chemical formula	[Cr ₂ Pb ₂ (NCS) ₂ (OH) ₂ ·(C ₄ H ₁₀ NO) ₄](NCS) ₂ ·C ₂ H ₅ N
<i>M_r</i>	1178.29
Crystal system, space group	Monoclinic, <i>C2/c</i>
Temperature (K)	298
<i>a</i> , <i>b</i> , <i>c</i> (Å)	17.533 (1), 13.8815 (7), 16.6179 (8)
β (°)	104.771 (6)
<i>V</i> (Å ³)	3910.9 (4)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	9.39
Crystal size (mm)	0.4 × 0.1 × 0.1
Data collection	
Diffraction	Agilent Xcalibur Sapphire 3
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Agilent, 2011)
<i>T_{min}</i> , <i>T_{max}</i>	0.382, 1.000
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	20193, 5680, 4133
<i>R_{int}</i>	0.064
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.703
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.035, 0.058, 0.93
No. of reflections	5680
No. of parameters	190
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	1.00, -0.69

Computer programs: *CrysAlis PRO* (Agilent, 2011), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *OLEX2* (Dolomanov *et al.*, 2009) and *PLATON* (Spek, 2009).

Li (Khrustalev *et al.*, 2004, 2008) and Na(Li)–Al (Nöth *et al.*, 2001).

5. Synthesis and crystallization

Lead monoxide (0.279 g, 1.25 mmol), NH₄[Cr(NCS)₄(NH₃)₂]·H₂O (0.443 g, 1.25 mmol), NH₄SCN (0.095 g, 1.25 mmol), 2-dimethylaminoethanol (0.5 ml, 5 mmol) and acetonitrile (20 ml) were heated in air at 323–333 K and stirred for 110 min until complete PbO dissolution occurred. Dark-grey crystals suitable for the crystallographic study were formed by slow evaporation of the resulting solution in air. The crystals were filtered off, washed with dry isopropyl alcohol and finally dried *in vacuo* at room temperature. Yield: 0.11 g, 10.3%.

The IR spectrum of the title compound (as KBr pellets) exhibited absorbance at 2250 cm⁻¹ assigned to ν (CN) of the solvent acetonitrile molecule, as well two additional bands at 2080 cm⁻¹ and 1610 cm⁻¹, which were assigned, respectively, to stretch and vibrational ν (CN) modes of the SCN anion. Analysis calculated for C₂₂H₄₅Cr₂N₉S₄Pb₂: C 22.43, H 3.85, N 10.69, S 10.88%; found: C 22.21, H 3.78, N 10.45, S 10.64%.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All hydrogen atoms were placed in

idealized positions and refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{C},\text{O})$ for methyl and hydroxyl groups.

During the refinement, several isolated electron density peaks were located, which were assignable to a solvent acetonitrile molecule(s) from the IR data and elementary analysis. Satisfactory results ($R_1 = 0.045$) were obtained modeling the disordered C and N atoms, but very large displacement parameters for them were observed. The SQUEEZE (Spek, 2015) procedure in *PLATON* (Spek, 2009) indicated solvent cavities of volume 118 Å³ centered at (0.5, 0, 0.25), (0.5, 0, 0.75), (0, 0.5, 0.75) and (0, 0.5, 0.25), each containing approximately 18 electrons. In the final refinement, this contribution was removed from the intensity data, producing better refinement results. We assumed full occupancy of the solvent molecule for each cavity, although the estimated 18 electrons are fewer than the 22 electrons expected for full occupancy. The solvent molecule is included in the reported molecular formula, weight and density.

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supporting information

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Crystal structure of tetrakis[μ_2 -2-(dimethylamino)ethanolato- $\kappa^3 N, O:O$]di- μ_3 -hydroxido-dithiocyanato- $\kappa^2 N$ -dichromium(III)dilead(II) dithiocyanate acetonitrile monosolvate

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Computing details

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO* (Agilent, 2011); data reduction: *CrysAlis PRO* (Agilent, 2011); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* and *PLATON* (Spek, 2009).

Tetrakis[μ_2 -2-(dimethylamino)ethanolato- $\kappa^3 N, O:O$]di- μ_3 -hydroxido-dithiocyanato- $\kappa^2 N$ -dichromium(III)dilead(II) dithiocyanate acetonitrile monosolvate

Crystal data

$[\text{Cr}_2\text{Pb}_2(\text{NCS})_2(\text{OH})_2(\text{C}_4\text{H}_{10}\text{NO})_4](\text{NCS})_2 \cdot \text{C}_2\text{H}_3\text{N}$
 $M_r = 1178.29$
 Monoclinic, $C2/c$
 $a = 17.533$ (1) Å
 $b = 13.8815$ (7) Å
 $c = 16.6179$ (8) Å
 $\beta = 104.771$ (6)°
 $V = 3910.9$ (4) Å³
 $Z = 4$

$F(000) = 2256$
 $D_x = 2.001$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 4335 reflections
 $\theta = 2.9\text{--}32.5^\circ$
 $\mu = 9.39$ mm⁻¹
 $T = 298$ K
 Block, metallic dark gray
 $0.4 \times 0.1 \times 0.1$ mm

Data collection

Agilent Xcalibur Sapphire 3
 diffractometer
 Radiation source: Enhance (Mo) X-ray Source
 Graphite monochromator
 Detector resolution: 16.1827 pixels mm⁻¹
 ω scans
 Absorption correction: multi-scan
 (CrysAlis PRO; Agilent, 2011)
 $T_{\min} = 0.382$, $T_{\max} = 1.000$

20193 measured reflections
 5680 independent reflections
 4133 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.064$
 $\theta_{\max} = 30.0^\circ$, $\theta_{\min} = 3.0^\circ$
 $h = -24 \rightarrow 24$
 $k = -19 \rightarrow 18$
 $l = -23 \rightarrow 23$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.058$
 $S = 0.93$
 5680 reflections

190 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

$$w = 1/[\sigma^2(F_o^2) + (0.0118P)^2]$$

where $P = (F_o^2 + 2F_c^2)/3$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 1.00 \text{ e } \text{Å}^{-3}$$

$$\Delta\rho_{\min} = -0.69 \text{ e } \text{Å}^{-3}$$

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.

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 $F^2 > 2\text{sigma}(F^2)$ 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	$U_{\text{iso}}^*/U_{\text{eq}}$
Pb1	0.369714 (9)	0.456817 (12)	0.330204 (9)	0.03296 (5)
Cr1	0.46177 (4)	0.59683 (5)	0.51003 (4)	0.02815 (15)
S1	0.33608 (10)	0.73222 (11)	0.70835 (8)	0.0651 (4)
S2	0.75622 (10)	0.58870 (14)	0.31923 (11)	0.0865 (6)
O1	0.35991 (16)	0.5544 (2)	0.44114 (16)	0.0327 (7)
O2	0.56441 (17)	0.6487 (2)	0.56837 (17)	0.0365 (7)
O3	0.50477 (15)	0.52564 (19)	0.42849 (15)	0.0294 (6)
H3	0.5314	0.5554	0.4027	0.044*
N1	0.4136 (2)	0.6465 (3)	0.5993 (2)	0.0412 (9)
N2	0.6095 (3)	0.6076 (4)	0.3499 (3)	0.0735 (14)
N3	0.2425 (2)	0.4171 (3)	0.3704 (2)	0.0385 (9)
N4	0.4503 (2)	0.7343 (3)	0.4474 (2)	0.0410 (9)
C1	0.3816 (3)	0.6803 (3)	0.6461 (3)	0.0395 (11)
C2	0.6716 (3)	0.6005 (4)	0.3384 (3)	0.0513 (13)
C3	0.2214 (3)	0.5127 (3)	0.3976 (3)	0.0441 (12)
H3A	0.1746	0.5067	0.4181	0.053*
H3B	0.2094	0.5561	0.3504	0.053*
C4	0.2877 (2)	0.5549 (3)	0.4655 (3)	0.0402 (11)
H4A	0.2746	0.6205	0.4770	0.048*
H4B	0.2939	0.5176	0.5161	0.048*
C5	0.2516 (3)	0.3465 (4)	0.4382 (3)	0.0536 (13)
H5A	0.2014	0.3357	0.4499	0.080*
H5B	0.2882	0.3708	0.4871	0.080*
H5C	0.2710	0.2870	0.4217	0.080*
C6	0.1806 (3)	0.3839 (4)	0.2987 (3)	0.0568 (14)
H6A	0.1336	0.3700	0.3160	0.085*
H6B	0.1980	0.3267	0.2762	0.085*
H6C	0.1697	0.4332	0.2568	0.085*
C7	0.5302 (3)	0.7792 (4)	0.4780 (3)	0.0604 (15)
H7A	0.5637	0.7591	0.4429	0.072*

H7B	0.5253	0.8488	0.4747	0.072*
C8	0.5662 (3)	0.7508 (3)	0.5642 (3)	0.0510 (13)
H8A	0.6202	0.7736	0.5813	0.061*
H8B	0.5371	0.7786	0.6010	0.061*
C9	0.4340 (4)	0.7257 (4)	0.3548 (3)	0.0702 (17)
H9A	0.4295	0.7889	0.3305	0.105*
H9B	0.3855	0.6913	0.3338	0.105*
H9C	0.4764	0.6916	0.3407	0.105*
C10	0.3883 (3)	0.7963 (4)	0.4640 (4)	0.0733 (18)
H10A	0.3869	0.8559	0.4344	0.110*
H10B	0.3992	0.8090	0.5226	0.110*
H10C	0.3382	0.7645	0.4459	0.110*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.03289 (9)	0.03895 (10)	0.02546 (8)	−0.00140 (8)	0.00458 (6)	−0.00003 (8)
Cr1	0.0277 (4)	0.0296 (4)	0.0261 (3)	0.0001 (3)	0.0050 (3)	−0.0017 (3)
S1	0.0878 (12)	0.0615 (10)	0.0580 (8)	0.0261 (8)	0.0407 (8)	0.0061 (7)
S2	0.0671 (11)	0.1125 (15)	0.0917 (12)	−0.0349 (10)	0.0421 (10)	−0.0433 (11)
O1	0.0271 (15)	0.0382 (17)	0.0324 (14)	−0.0022 (12)	0.0070 (12)	−0.0052 (13)
O2	0.0353 (17)	0.0310 (17)	0.0376 (15)	−0.0028 (13)	−0.0009 (13)	0.0001 (13)
O3	0.0298 (15)	0.0320 (17)	0.0285 (13)	−0.0004 (12)	0.0111 (12)	0.0022 (12)
N1	0.044 (2)	0.044 (2)	0.0354 (19)	0.0010 (18)	0.0085 (18)	−0.0085 (18)
N2	0.075 (4)	0.085 (4)	0.069 (3)	−0.007 (3)	0.033 (3)	0.004 (3)
N3	0.032 (2)	0.044 (2)	0.0366 (19)	−0.0103 (17)	0.0048 (17)	−0.0009 (18)
N4	0.042 (2)	0.036 (2)	0.040 (2)	0.0025 (17)	0.0008 (18)	0.0030 (18)
C1	0.044 (3)	0.035 (3)	0.037 (2)	0.004 (2)	0.007 (2)	−0.004 (2)
C2	0.060 (4)	0.059 (4)	0.037 (2)	−0.017 (3)	0.015 (3)	−0.004 (2)
C3	0.031 (2)	0.052 (3)	0.047 (3)	0.002 (2)	0.007 (2)	0.002 (2)
C4	0.026 (2)	0.052 (3)	0.044 (2)	0.002 (2)	0.011 (2)	−0.008 (2)
C5	0.056 (3)	0.049 (3)	0.057 (3)	−0.007 (2)	0.018 (3)	0.007 (3)
C6	0.035 (3)	0.077 (4)	0.052 (3)	−0.017 (3)	−0.001 (2)	−0.006 (3)
C7	0.061 (4)	0.046 (3)	0.066 (3)	−0.015 (3)	0.001 (3)	0.011 (3)
C8	0.045 (3)	0.034 (3)	0.065 (3)	−0.011 (2)	−0.002 (3)	0.001 (2)
C9	0.086 (5)	0.068 (4)	0.048 (3)	0.009 (3)	0.003 (3)	0.019 (3)
C10	0.081 (4)	0.051 (4)	0.089 (4)	0.025 (3)	0.025 (4)	0.015 (3)

Geometric parameters (Å, °)

Pb1—O2 ⁱ	2.308 (3)	C3—H3A	0.9700
Pb1—O1	2.329 (3)	C3—H3B	0.9700
Pb1—N3	2.547 (4)	C4—H4A	0.9700
Pb1—O3	2.686 (3)	C4—H4B	0.9700
Cr1—O1	1.950 (3)	C5—H5A	0.9600
Cr1—O2	1.951 (3)	C5—H5B	0.9600
Cr1—O3	1.975 (3)	C5—H5C	0.9600
Cr1—O3 ⁱ	1.993 (3)	C6—H6A	0.9600

Cr1—N1	2.008 (4)	C6—H6B	0.9600
Cr1—N4	2.158 (4)	C6—H6C	0.9600
S1—C1	1.627 (5)	C7—C8	1.465 (6)
S2—C2	1.603 (6)	C7—H7A	0.9700
O1—C4	1.424 (5)	C7—H7B	0.9700
O3—Cr1 ⁱ	1.993 (3)	C8—O2	1.419 (5)
O3—H3	0.8211	C8—H8A	0.9700
N1—C1	1.167 (5)	C8—H8B	0.9700
N2—C2	1.157 (6)	C9—H9A	0.9600
N3—C6	1.467 (5)	C9—H9B	0.9600
N3—C5	1.471 (5)	C9—H9C	0.9600
N3—C3	1.479 (6)	C10—H10A	0.9600
N4—C10	1.466 (6)	C10—H10B	0.9600
N4—C9	1.497 (6)	C10—H10C	0.9600
N4—C7	1.498 (6)	O2—Pb1 ⁱ	2.308 (3)
C3—C4	1.515 (6)		
O2 ⁱ —Pb1—O1	85.15 (10)	N3—C3—H3B	109.3
O2 ⁱ —Pb1—N3	88.84 (11)	C4—C3—H3B	109.3
O1—Pb1—N3	70.85 (10)	H3A—C3—H3B	107.9
O2 ⁱ —Pb1—O3	65.32 (9)	O1—C4—C3	110.8 (3)
O1—Pb1—O3	62.83 (8)	O1—C4—H4A	109.5
N3—Pb1—O3	127.79 (9)	C3—C4—H4A	109.5
O1—Cr1—O2	173.46 (12)	O1—C4—H4B	109.5
O1—Cr1—O3	84.20 (11)	C3—C4—H4B	109.5
O2—Cr1—O3	93.48 (12)	H4A—C4—H4B	108.1
O1—Cr1—O3 ⁱ	98.58 (11)	N3—C5—H5A	109.5
O2—Cr1—O3 ⁱ	86.94 (11)	N3—C5—H5B	109.5
O3—Cr1—O3 ⁱ	79.10 (11)	H5A—C5—H5B	109.5
O1—Cr1—N1	92.45 (13)	N3—C5—H5C	109.5
O2—Cr1—N1	90.82 (14)	H5A—C5—H5C	109.5
O3—Cr1—N1	170.07 (13)	H5B—C5—H5C	109.5
O3 ⁱ —Cr1—N1	92.21 (13)	N3—C6—H6A	109.5
O1—Cr1—N4	91.44 (13)	N3—C6—H6B	109.5
O2—Cr1—N4	82.75 (13)	H6A—C6—H6B	109.5
O3—Cr1—N4	96.70 (13)	N3—C6—H6C	109.5
O3 ⁱ —Cr1—N4	168.63 (13)	H6A—C6—H6C	109.5
N1—Cr1—N4	92.71 (15)	H6B—C6—H6C	109.5
C4—O1—Cr1	125.4 (2)	C8—C7—N4	110.6 (4)
C4—O1—Pb1	118.6 (2)	C8—C7—H7A	109.5
Cr1—O1—Pb1	113.50 (12)	N4—C7—H7A	109.5
Cr1—O3—Cr1 ⁱ	100.90 (11)	C8—C7—H7B	109.5
Cr1—O3—Pb1	99.42 (10)	N4—C7—H7B	109.5
Cr1 ⁱ —O3—Pb1	96.14 (10)	H7A—C7—H7B	108.1
Cr1—O3—H3	118.3	O2—C8—C7	107.9 (4)
Cr1 ⁱ —O3—H3	124.4	O2—C8—H8A	110.1
Pb1—O3—H3	113.0	C7—C8—H8A	110.1
C1—N1—Cr1	174.3 (4)	O2—C8—H8B	110.1

C6—N3—C5	109.0 (4)	C7—C8—H8B	110.1
C6—N3—C3	109.9 (4)	H8A—C8—H8B	108.4
C5—N3—C3	110.6 (3)	N4—C9—H9A	109.5
C6—N3—Pb1	111.8 (3)	N4—C9—H9B	109.5
C5—N3—Pb1	114.4 (3)	H9A—C9—H9B	109.5
C3—N3—Pb1	100.9 (2)	N4—C9—H9C	109.5
C10—N4—C9	106.5 (4)	H9A—C9—H9C	109.5
C10—N4—C7	111.4 (4)	H9B—C9—H9C	109.5
C9—N4—C7	107.4 (4)	N4—C10—H10A	109.5
C10—N4—Cr1	114.3 (3)	N4—C10—H10B	109.5
C9—N4—Cr1	113.3 (3)	H10A—C10—H10B	109.5
C7—N4—Cr1	103.9 (3)	N4—C10—H10C	109.5
N1—C1—S1	177.1 (4)	H10A—C10—H10C	109.5
N2—C2—S2	177.9 (5)	H10B—C10—H10C	109.5
N3—C3—C4	111.8 (4)	C8—O2—Cr1	111.9 (3)
N3—C3—H3A	109.3	C8—O2—Pb1 ⁱ	131.0 (3)
C4—C3—H3A	109.3	Cr1—O2—Pb1 ⁱ	110.78 (13)
O2—Cr1—O1—C4	-126.9 (10)	O1—Pb1—N3—C3	33.2 (2)
O3—Cr1—O1—C4	163.7 (3)	O3—Pb1—N3—C3	61.4 (3)
O3 ⁱ —Cr1—O1—C4	85.6 (3)	O1—Cr1—N4—C10	68.5 (4)
N1—Cr1—O1—C4	-7.0 (3)	O2—Cr1—N4—C10	-114.5 (4)
N4—Cr1—O1—C4	-99.7 (3)	O3—Cr1—N4—C10	152.8 (3)
O2—Cr1—O1—Pb1	71.6 (11)	O3 ⁱ —Cr1—N4—C10	-139.6 (6)
O3—Cr1—O1—Pb1	2.16 (13)	N1—Cr1—N4—C10	-24.0 (4)
O3 ⁱ —Cr1—O1—Pb1	-75.86 (13)	O1—Cr1—N4—C9	-53.7 (3)
N1—Cr1—O1—Pb1	-168.47 (15)	O2—Cr1—N4—C9	123.3 (4)
N4—Cr1—O1—Pb1	98.76 (15)	O3—Cr1—N4—C9	30.6 (3)
O2 ⁱ —Pb1—O1—C4	-100.0 (3)	O3 ⁱ —Cr1—N4—C9	98.2 (7)
N3—Pb1—O1—C4	-9.5 (3)	N1—Cr1—N4—C9	-146.2 (3)
O3—Pb1—O1—C4	-164.7 (3)	O1—Cr1—N4—C7	-169.9 (3)
O2 ⁱ —Pb1—O1—Cr1	62.91 (14)	O2—Cr1—N4—C7	7.1 (3)
N3—Pb1—O1—Cr1	153.37 (16)	O3—Cr1—N4—C7	-85.6 (3)
O3—Pb1—O1—Cr1	-1.78 (10)	O3 ⁱ —Cr1—N4—C7	-18.0 (8)
O1—Cr1—O3—Cr1 ⁱ	-99.92 (12)	N1—Cr1—N4—C7	97.6 (3)
O2—Cr1—O3—Cr1 ⁱ	86.21 (12)	C6—N3—C3—C4	-173.7 (4)
O3 ⁱ —Cr1—O3—Cr1 ⁱ	0.0	C5—N3—C3—C4	65.9 (5)
N1—Cr1—O3—Cr1 ⁱ	-29.3 (8)	Pb1—N3—C3—C4	-55.6 (4)
N4—Cr1—O3—Cr1 ⁱ	169.31 (13)	Cr1—O1—C4—C3	-177.4 (3)
O1—Cr1—O3—Pb1	-1.74 (10)	Pb1—O1—C4—C3	-16.7 (5)
O2—Cr1—O3—Pb1	-175.61 (11)	N3—C3—C4—O1	52.2 (5)
O3 ⁱ —Cr1—O3—Pb1	98.18 (12)	C10—N4—C7—C8	90.1 (5)
N1—Cr1—O3—Pb1	68.9 (8)	C9—N4—C7—C8	-153.7 (4)
N4—Cr1—O3—Pb1	-92.51 (12)	Cr1—N4—C7—C8	-33.4 (5)
O2 ⁱ —Pb1—O3—Cr1	-95.92 (12)	N4—C7—C8—O2	52.8 (6)
O1—Pb1—O3—Cr1	1.63 (10)	C7—C8—O2—Cr1	-45.9 (5)
N3—Pb1—O3—Cr1	-28.53 (17)	C7—C8—O2—Pb1 ⁱ	165.2 (3)
O2 ⁱ —Pb1—O3—Cr1 ⁱ	6.24 (9)	O1—Cr1—O2—C8	48.5 (12)

O1—Pb1—O3—Cr1 ⁱ	103.79 (11)	O3—Cr1—O2—C8	117.4 (3)
N3—Pb1—O3—Cr1 ⁱ	73.63 (15)	O3 ⁱ —Cr1—O2—C8	-163.7 (3)
O2 ⁱ —Pb1—N3—C6	-124.8 (3)	N1—Cr1—O2—C8	-71.6 (3)
O1—Pb1—N3—C6	149.9 (3)	N4—Cr1—O2—C8	21.1 (3)
O3—Pb1—N3—C6	178.2 (3)	O1—Cr1—O2—Pb1 ⁱ	-156.1 (10)
O2 ⁱ —Pb1—N3—C5	-0.3 (3)	O3—Cr1—O2—Pb1 ⁱ	-87.20 (13)
O1—Pb1—N3—C5	-85.5 (3)	O3 ⁱ —Cr1—O2—Pb1 ⁱ	-8.32 (13)
O3—Pb1—N3—C5	-57.3 (3)	N1—Cr1—O2—Pb1 ⁱ	83.85 (15)
O2 ⁱ —Pb1—N3—C3	118.4 (2)	N4—Cr1—O2—Pb1 ⁱ	176.47 (16)

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

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O3—H3 \cdots N2	0.82	1.95	2.757 (6)	169