

This article was downloaded by:

On: 23 January 2011

Access details: *Access Details: Free Access*

Publisher *Taylor & Francis*

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



Journal of Coordination Chemistry

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713455674>

Syntheses and crystal structures of two lead complexes containing tricyanomethanide

Yu-Jun Shi^a; Yi-Zhi Li^a; Jian-Hao Zhou^a; Xue-Tai Chen^a; Bin Huang^a; Xiao-Zeng You^a

^a State Key Laboratory of Coordination Chemistry, Coordination Chemistry Institute, Nanjing University, Nanjing 210093, P.R. China

Online publication date: 12 May 2010

To cite this Article Shi, Yu-Jun , Li, Yi-Zhi , Zhou, Jian-Hao , Chen, Xue-Tai , Huang, Bin and You, Xiao-Zeng(2003) 'Syntheses and crystal structures of two lead complexes containing tricyanomethanide', *Journal of Coordination Chemistry*, 56: 17, 1481 – 1488

To link to this Article: DOI: 10.1080/00958970310001617085

URL: <http://dx.doi.org/10.1080/00958970310001617085>

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.informaworld.com/terms-and-conditions-of-access.pdf>

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

SYNTHESES AND CRYSTAL STRUCTURES OF TWO LEAD COMPLEXES CONTAINING TRICYANOMETHANIDE

YU-JUN SHI*, YI-ZHI LI, JIAN-HAO ZHOU, XUE-TAI CHEN[†],
BIN HUANG and XIAO-ZENG YOU[†]

*State Key Laboratory of Coordination Chemistry, Coordination Chemistry Institute,
Nanjing University, Nanjing 210093, P.R. China*

(Received 29 April 2003; In final form 11 August 2003)

Two lead complexes $[\text{Pb}(2,2'\text{-bipy})_2(\text{H}_2\text{O})_2(\text{tcm})]_2 \cdot (\text{tcm})_2 \cdot 2\text{H}_2\text{O}$, **1**, and $[\text{Pb}(\text{phen})_2(\text{tcm})_2]$, **2**, have been prepared by reacting $\text{Pb}(\text{NO}_3)_2$, potassium tricyanomethanide (Ktcm) and 2,2-bipyridine or 10-phenanthroline, respectively. X-ray crystallography studies show that **1** is a dimer formed by two eight-coordinate $[\text{Pb}(\text{bipy})_2(\text{H}_2\text{O})_2]$ units bridged by tricyanomethanide while **2** is a mononuclear six-coordinate compound with the lead atom surrounded by two phen ligands and two tricyanomethanide ions.

Keywords: Lead; Tricyanomethanide; Crystal structure; Mononuclear; Dimer

INTRODUCTION

Coordination chemistry of tricyanomethanide (tcm^{-1}) initiated research interest fifty years ago [1]. Subsequently, various metal tricyanomethanide complexes were studied by Kohler and co-workers [2] and a considerable number of spectroscopic studies have been undertaken. More recently, transition metal complexes, especially the binary compounds $\text{M}(\text{tcm})_2$, have aroused renewed interest due to their interesting supramolecular structures and magnetic properties [3–8]. Similarly, analogous metal dicyanamide complexes have been extensively studied [8–10]. In contrast, main group metal compounds with tricyanomethanide have been less studied and published structures are few in number. Crystal structure determinations have been carried out for $\text{Na}(\text{tcm})$ [11] and $\text{K}(\text{tcm})$ [12] where the metal ion is coordinated to six nitrogen atoms. In $\text{MeTl}(\text{tcm})$, tcm is coordinated to four metal ions [13].

Recently our research has focused on PbX_2 -based organic–inorganic hybrids with polymeric network structures by taking advantage of the versatility of lead in coordination chemistry [14–18]. We have prepared three lead dicyanamide complexes with very

*Permanent address: Department of Chemistry, Nantong Teacher's College, Nantong 226007, P.R. China.

[†]Corresponding authors. E-mail: xtchen@netra.nju.edu.cn

particular supramolecular structures and new coordination modes of dicyanamide [17,18]. As a continuation of our research on lead-pseudohalides we report the syntheses and structure of two new lead tricyanomethanide complexes $[\text{Pb}(2,2'\text{-bipy})_2(\text{H}_2\text{O})_2(\text{tcm})_2] \cdot (\text{tcm})_2 \cdot 2\text{H}_2\text{O}$, **1**, and $[\text{Pb}(\text{phen})_2(\text{tcm})_2]$, **2**.

EXPERIMENTAL

All reagents were commercially available and were used as received without further purification. Elemental analyses were carried on a Perkin-Elmer 240C instrument. IR spectra were recorded on a Bruker Optik spectrophotometer (range: 400–4000 cm^{-1}) as KBr pellets.

Synthesis of **1** and **2**

$[\text{Pb}(2,2'\text{-bipy})_2(\text{H}_2\text{O})_2(\text{tcm})_2] \cdot (\text{tcm})_2 \cdot 2\text{H}_2\text{O}$, **1** 5 cm^3 of an ethanol solution containing 0.065 g (0.5 mmol) of Ktcm and 0.078 g (0.5 mmol) of 2,2'-bipy was added dropwise to 5 cm^3 of an aqueous solution of 0.083 g $\text{Pb}(\text{NO}_3)_2$ (0.25 mmol) without stirring. The resulting mixture was left at room temperature and single crystals suitable for single-crystal X-ray diffraction appeared after several weeks. Yield: 0.12 g (63% based on $\text{Pb}(\text{NO}_3)_2$). Anal. Calcd. for $\text{C}_{56}\text{H}_{44}\text{N}_{20}\text{O}_6\text{Pb}_2$ (%): C, 44.61; H, 2.94; N, 18.57. Found C, 44.86; H, 2.82; N, 18.38. IR (KBr pellets, cm^{-1}): 564(s), 625(w), 644(m), 735(m), 810(w), 971(m), 1009(vs), 1061(m), 1099(m), 1156(s), 1172(s), 1244(s), 1437(vs), 1473(s), 1491(s), 1566(m), 1592(vs), 2179(vs), 2219(m), 3413(s).

$[\text{Pb}(1,10\text{-phen})_2(\text{tcm})_2]$, **2** The synthesis for **2** was similar to that for **1** except the ligand 1,10-phen was used instead of 2,2'-bipy. Single crystals suitable for single-crystal X-ray diffraction appeared after several weeks. Yield: 0.14 g (72% based on $\text{Pb}(\text{NO}_3)_2$). Anal. Calcd. for $\text{C}_{32}\text{H}_{16}\text{N}_{10}\text{Pb}$ (%): C, 51.40; H, 2.16; N, 18.72. Found: C, 51.66; H, 2.02; N, 18.48. IR (KBr pellets, cm^{-1}): 563(s), 637(m), 726(vs), 781(m), 819(w) 851(vs), 896(w), 960(w), 1006(w), 1034(m), 1099(s), 1144(s), 1223(s), 1294(s), 1342(s), 1426(vs), 1494(s), 1517(vs), 1573(m), 1589(s), 1623(s), 2172(vs).

X-ray Crystallography

Data were collected on a Bruker P4 area detector diffractometer in the range $3.98 < 2\theta < 50.00^\circ$ for **1** at 293 K and on a Bruker SMART CCD area detector diffractometer in the range $4.08 < 2\theta < 48.98^\circ$ for **2** at 293 K with graphite-monochromated Mo K α radiation ($\lambda = 0.71073 \text{ \AA}$). Of the total 7525 reflections for **1** and 6007 reflections for **2** that were collected, 5110 and 4884 were unique, respectively. The structures were solved by direct methods (SHELXS-97) [19] and refined by full-matrix least-squares calculations on F^2 (SHELXL-97) [20]. The non-hydrogen atoms were refined anisotropically, while hydrogen atoms adjacent to carbon atoms were placed on calculated positions ($\text{C-H} = 0.93 \text{ \AA}$) riding on their parent atoms and assigned isotropic thermal parameters; hydrogen atoms from water molecules were located in a different map and refined with isotropic thermal parameters. The crystal data and details of crystal structure determinations are summarized in Table I and atomic coordinates and equivalent isotropic displacement parameters for **1** and **2** are listed in Tables II and III. Selected bond distances and angles for **1** and **2** are listed in Table IV.

TABLE I Crystal data and structure refinement details for **1** and **2**

Compound	1	2
Empirical formula	C ₅₆ H ₄₄ N ₂₀ O ₆ Pb ₂	C ₃₂ H ₁₆ N ₁₀ Pb
Formula weight	1507.52	747.75
Crystal size (mm)	0.20 × 0.16 × 0.13	0.30 × 0.20 × 0.20
Crystal system	Triclinic	Monoclinic
Space group	<i>P1</i>	<i>P2₁/c</i>
<i>a</i> (Å)	9.352(4)	13.863(2)
<i>b</i> (Å)	10.307(5)	14.531(2)
<i>c</i> (Å)	16.026(8),	14.859(2)
α (°)	84.50(1)	
β (°)	75.78(1)	112.15(1)
γ (°)	86.92(1)	
Volume (Å ³)	1489.8(12)	2772.3(7)
<i>Z</i>	1	4
<i>D</i> calc. (g cm ⁻³)	1.680	1.791
μ (mm ⁻¹)	5.709	6.128
<i>F</i> (000)	732	1440
Temperature (K)	293	293
θ range (°)	2.0, 25.0	2.0, 25.0
Limiting indices	$-10 \leq h \leq 11, -9 \leq k \leq 12,$ $-17 \leq l \leq 19$	$-16 \leq h \leq 15,$ $0 \leq k \leq 17, 0 \leq l \leq 17$
Reflections collected	7525	6007
Independent reflections	5110 ($R_{\text{int}} = 0.028$)	4884 ($R_{\text{int}} = 0.0436$)
Observed reflections ($I > 2\sigma$)	4555	3013
Absorption correction	Empirical	Empirical
Max. and min. transmission	0.317, 0.263	0.294, 0.239
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/parameters	5110/379	4884/388
Goodness-of-fit on F^2	1.030	0.969
Final <i>R</i> indices [$I > 2\sigma(I)$]	$R1 = 0.0359$ $wR = 0.0945$	$R1 = 0.0467$ $wR = 0.0621$
Max./min. $\Delta\rho$ (e Å ⁻³)	0.983/−0.902	0.845/−0.525

TABLE II Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters (Å² × 10³) for **1**

	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	<i>U</i> (eq)
Pb(1)	1669(1)	10 663(1)	7300(1)	41(1)
O(1)	1426(6)	12 837(5)	8174(4)	71(2)
O(2)	4298(6)	10 741(5)	7873(4)	72(2)
N(1)	1656(6)	8636(5)	8392(4)	49(2)
N(2)	−686(5)	10 371(5)	8539(3)	45(2)
N(3)	246(7)	9026(6)	6639(4)	56(2)
N(4)	−323(6)	11 636(6)	6494(4)	51(2)
N(5)	3701(9)	9472(7)	5939(5)	84(3)
N(6)	6887(8)	7161(8)	4034(5)	78(3)
N(7)	5229(13)	5582(9)	6745(6)	118(4)
C(1)	2744(7)	7773(7)	8286(5)	54(2)
C(2)	2750(8)	6652(7)	8814(6)	64(3)
C(3)	1609(9)	6404(8)	9476(6)	71(3)
C(4)	408(10)	7278(7)	9615(5)	68(3)
C(5)	456(7)	8395(6)	9059(4)	44(2)
C(6)	−806(6)	9393(6)	9167(4)	42(2)
C(7)	−1954(7)	9328(7)	9883(5)	52(2)
C(8)	−3053(8)	10 274(8)	9963(5)	61(3)
C(9)	−2968(8)	11 263(8)	9327(5)	63(3)
C(10)	−1715(7)	11 290(8)	8637(5)	59(3)
C(11)	659(11)	7769(8)	6616(6)	76(3)

(continued)

TABLE II Continued

	x/a	y/b	z/c	$U(\text{eq})$
C(12)	102(11)	6907(9)	6170(6)	76(3)
C(13)	-778(11)	7355(10)	5713(6)	81(4)
C(14)	-1290(10)	8656(9)	5739(5)	69(3)
C(15)	-718(7)	9468(7)	6207(5)	52(2)
C(16)	-1182(8)	10 855(8)	6256(4)	55(3)
C(17)	-2573(8)	11 373(9)	6062(5)	69(3)
C(18)	-2898(10)	12 688(10)	6103(7)	85(4)
C(19)	-1902(11)	13 465(9)	6318(6)	79(3)
C(20)	-638(7)	12 909(8)	6504(5)	56(3)
C(21)	4397(10)	8548(8)	5754(6)	76(3)
C(22)	5275(8)	7396(7)	5538(5)	55(2)
C(23)	6161(8)	7266(7)	4717(5)	57(3)
C(24)	5232(11)	6380(8)	6202(6)	77(3)
N(8)	6458(7)	8582(8)	7785(5)	84(3)
N(9)	9125(9)	4861(8)	8119(5)	95(3)
N(10)	5786(9)	6321(7)	10 283(5)	85(3)
C(25)	6767(8)	7683(7)	8185(5)	61(3)
C(26)	7153(8)	6582(7)	8685(5)	53(2)
C(27)	8287(10)	5646(8)	8361(6)	67(3)
C(28)	6391(10)	6439(7)	9574(6)	64(3)
O(3)	4395(8)	3384(5)	7953(4)	82(2)

TABLE III Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **2**

	x/a	y/b	z/c	$U(\text{eq})$
Pb(1)	7415(1)	1758(1)	8293(1)	046(1)
N(1)	7212(5)	1460(4)	6609(4)	49(2)
N(2)	5581(4)	1319(4)	7190(5)	50(3)
N(3)	9292(5)	1261(5)	8618(5)	55(3)
N(4)	7700(5)	52(4)	8398(4)	53(3)
N(5)	8174(5)	3376(6)	7750(6)	78(3)
N(6)	9208(6)	6134(6)	9056(6)	90(4)
N(7)	11 422(5)	4193(5)	8695(5)	76(3)
N(8)	6745(5)	1167(5)	9785(5)	67(3)
N(9)	3551(6)	1409(6)	9173(5)	86(4)
N(10)	5659(5)	1500(6)	12 229(5)	84(4)
C(1)	4786(6)	1247(5)	7460(6)	52(3)
C(2)	3760(6)	1149(5)	6850(6)	52(3)
C(3)	3519(6)	1127(5)	5878(6)	52(3)
C(4)	4366(6)	1176(5)	5540(6)	49(3)
C(5)	4187(6)	1118(5)	4515(6)	51(3)
C(6)	5021(6)	1164(5)	4273(6)	51(3)
C(7)	6071(6)	1259(5)	4948(6)	50(3)
C(8)	6897(6)	1263(5)	4650(6)	51(3)
C(9)	7877(6)	1399(5)	5364(5)	51(3)
C(10)	7979(6)	1512(5)	6291(6)	51(3)
C(11)	6252(6)	1345(5)	5915(6)	48(3)
C(12)	5385(6)	1270(5)	6231(6)	48(3)
C(13)	6877(6)	-497(6)	8288(5)	55(3)
C(14)	7034(6)	-1440(6)	8307(5)	57(3)
C(15)	8007(6)	-1783(7)	8489(5)	60(3)
C(16)	8795(6)	-1226(6)	8613(6)	57(3)
C(17)	9847(6)	-1550(6)	8769(5)	58(3)
C(18)	10 597(6)	-962(6)	8903(6)	56(3)
C(19)	10 493(6)	-22(6)	8843(6)	55(3)

(continued)

TABLE III Continued

	x/a	y/b	z/c	$U(\text{eq})$
C(20)	11 282(6)	574(6)	8964(5)	58(3)
C(21)	11 098(6)	1486(6)	8931(6)	58(3)
C(22)	10 083(5)	1810(7)	8762(5)	58(3)
C(23)	9450(6)	310(6)	8680(5)	53(3)
C(24)	8650(6)	-264(6)	8552(6)	53(3)
C(25)	8819(7)	3929(7)	8106(7)	70(4)
C(26)	9593(7)	4580(8)	8487(6)	69(4)
C(27)	10 598(7)	4394(7)	8592(6)	68(4)
C(28)	9372(7)	5446(8)	8758(7)	72(4)
C(29)	6148(6)	1193(7)	10 123(6)	67(4)
C(30)	5330(6)	1329(7)	10 436(6)	69(4)
C(31)	4332(7)	1373(7)	9794(7)	74(4)
C(32)	5542(6)	1401(7)	11 424(7)	71(4)

TABLE IV Bond lengths [\AA] and angles [$^\circ$] for **1** and **2**

1			
Pb(1)–N(2)	2.587(5)	Pb(1)–N(1)	2.592(5)
Pb(1)–N(4)	2.623(5)	Pb(1)–N(3)	2.648(6)
Pb(1)–O(1)	2.726(6)	Pb(1)–O(2)	2.836(5)
Pb(1)–N(5)	2.845(7)	Pb(1)–N(6a)	3.068(8)
N(2)–Pb(1)–N(1)	63.65(16)	N(2)–Pb(1)–N(4)	80.12(17)
N(1)–Pb(1)–N(4)	131.41(17)	N(2)–Pb(1)–N(3)	80.01(18)
N(1)–Pb(1)–N(3)	79.96(19)	N(4)–Pb(1)–N(3)	61.96(19)
N(2)–Pb(1)–O(1)	74.64(17)	N(1)–Pb(1)–O(1)	108.20(18)
N(4)–Pb(1)–O(1)	90.69(18)	N(3)–Pb(1)–O(1)	145.57(19)
N(2)–Pb(1)–O(2)	113.89(18)	N(1)–Pb(1)–O(2)	74.06(16)
N(4)–Pb(1)–O(2)	153.99(18)	N(3)–Pb(1)–O(2)	139.46(18)
O(1)–Pb(1)–O(2)	73.40(17)	N(2)–Pb(1)–N(5)	145.9(2)
N(1)–Pb(1)–N(5)	93.4(2)	N(4)–Pb(1)–N(5)	100.7(2)
N(3)–Pb(1)–N(5)	71.0(2)	O(1)–Pb(1)–N(5)	139.0(2)
O(2)–Pb(1)–N(5)	80.0(2)	N(2)–Pb(1)–N(6a)	137.75(18)
N(1)–Pb(1)–N(6a)	154.97(18)	N(4)–Pb(1)–N(6a)	72.56(19)
N(3)–Pb(1)–N(6a)	112.7(2)	O(1)–Pb(1)–N(6a)	74.1(2)
O(2)–Pb(1)–N(6a)	83.18(18)	N(5)–Pb(1)–N(6a)	72.2(2)
Pb(1)–N(1)	2.449(6)	Pb(1)–N(4)	2.506(6)
Pb(1)–N(2)	2.532(6)	Pb(1)–N(3)	2.564(6)
Pb(1)–N(5)	2.816(9)	Pb(1)–N(8)	2.841(7)
N(1)–Pb(1)–N(4)	81.01(19)	N(1)–Pb(1)–N(2)	66.0(2)
N(4)–Pb(1)–N(2)	83.56(19)	N(1)–Pb(1)–N(3)	82.0(2)
N(4)–Pb(1)–N(3)	65.5(2)	N(2)–Pb(1)–N(3)	138.7(2)
N(1)–Pb(1)–N(5)	76.5(2)	N(4)–Pb(1)–N(5)	141.0(2)
N(2)–Pb(1)–N(5)	114.6(2)	N(3)–Pb(1)–N(5)	80.0(2)
N(1)–Pb(1)–N(8)	143.0(2)	N(4)–Pb(1)–N(8)	75.1(2)
N(2)–Pb(1)–N(8)	83.4(2)	N(3)–Pb(1)–N(8)	112.0(2)
N(5)–Pb(1)–N(8)	138.0(2)		

Symmetry code a: $1-x$; $2-y$; $1-z$.

Crystallographic data for the structures of **1** and **2** have been deposited with the Cambridge Crystallographic Data Centre as supplementary publications CCDC-208266 (**1**) and CCDC-208265 (**2**). Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

The crystal structure of **1** consists of dimeric cations, uncoordinated tricyanomethanide anion and solvate water (see Fig. 1). The cation can be considered as a dimer constructed of two eight-coordinated lead units bridged by tcm^{-1} . The cation is centrosymmetric, leading to a 12-membered metallomacrocycle with a chair-like configuration. Lead is coordinated by two bipy ligands, two H_2O molecules and two nitrogen atoms from tcm^{-1} . The coordination geometry around lead is not regular. Pb–N(bipy) bonds are in the range 2.587(5)–2.648(6) Å with an average value of 2.617 Å, comparable to literature values [21]. Two water molecules are coordinated to lead with different distances, 2.726(6) Å for Pb(1)–O(1) and 2.836(5) Å for Pb(1)–O(2). Pb(1)–N(tcm) bonds are much longer than Pb(1)–N(bipy) bonds. Pb(1)–N(5) is 2.845(7) Å. The Pb(1)–N(6a) distance is rather long (3.068(8) Å), and could be considered as a “weak bond”. Similar long Pb–N bond distances have been observed in $\text{Pb}(\text{dca})_2$ (3.138(17) Å) [17] and $\{\text{[HC(3,5-Me}_2\text{Pz)}_3\text{]Pb\{HB(3,5-Me}_2\text{Pz)}_3\}\text{BF}_4$ (2.827(7) Å) [22]. The bond angles around the lead atom are in the range 61.96(16) to 154.97(18)°. The bite angles of chelated 2,2-bipyridine are 63.65(16)° for N(1)–Pb(1)–N(2) and 61.96(19)° for N(3)–Pb(1)–N(4), respectively. In the crystal several hydrogen bonds can be defined. There are six traditional hydrogen bonds O–H...O(N) and one C–H...O hydrogen bond in the crystal structure (see Table V).

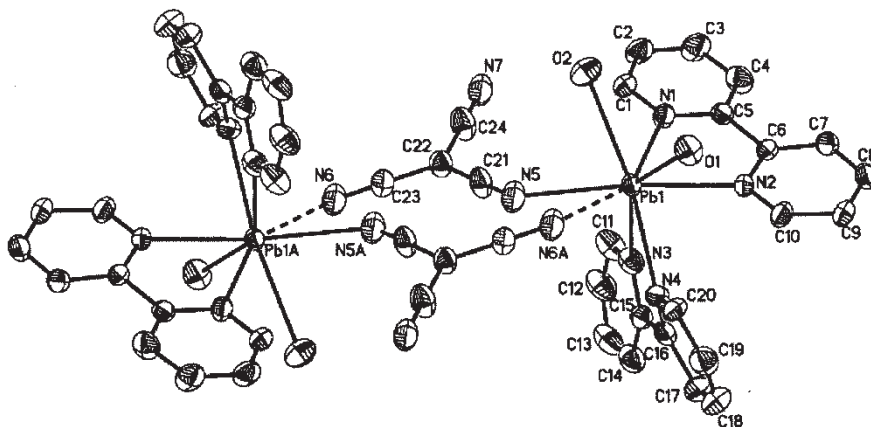


FIGURE 1 ORTEP drawing of the cation of **1** with the atom labelling scheme (30% probability thermal ellipsoids).

TABLE V Hydrogen bonds in the crystal structure of **1**

$D-H\cdots A$	$D\cdots H$ (Å)	$H\cdots A$ (Å)	$D\cdots A$ (Å)	$\angle DHA$ (°)	<i>Sym. trans.</i>
O(1)–H(1A)···O(3)	0.8498	1.9813	2.793(10)	159.38	$x, 1+y, z$
O(1)–H(1B)···N(9)	0.8498	2.2576	2.927(10)	135.72	$-1+x, 1+y, z$
O(2)–H(2A)···N(8)	0.8493	2.3996	2.914(10)	119.55	
O(2)–H(2D)···O(3)	0.8500	2.0604	2.747(7)	137.37	$x, 1+y, z$
O(3)–H(3A)···N(10)	0.8511	2.3561	2.835(10)	116.05	$1-x, 1-y, 2-z$
O(3)–H(3D)···N(7)	0.8497	1.9957	2.845(11)	179.53	
C(9)–H(9)···N(10)	0.9303	2.5918	3.510(11)	169.28	$-x, 2-y, 2-z$

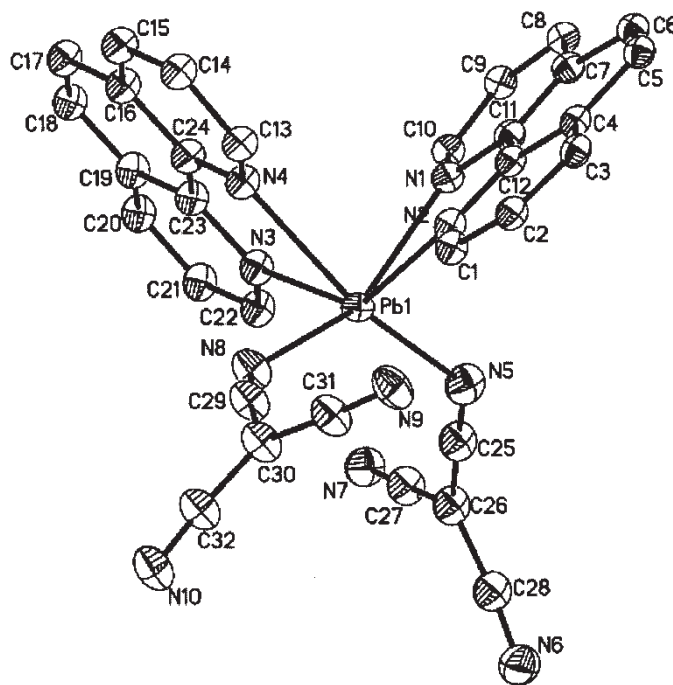


FIGURE 2 ORTEP drawing of **2** with the atom labelling scheme (30% probability thermal ellipsoids).

Complex **2** is a six-coordinate mononuclear lead complex with lead coordinated by two phen ligands and two tcm^{-1} ions as shown in Fig. 2. The coordination geometry is irregular, Pb(1)–N(phen) distances lie in the range 2.449(6)–2.564(6) Å with an average value of 2.512 Å, comparable to those reported for Pb(phen)X₂ (X=Cl, Br, I) [23]. Two Pb–N(tcm) bonds are 2.816 and 2.841 Å, respectively, similar to the Pb(1)–N(5) bond (2.845(7) Å) in **1**. Bond angles around lead range from 65.5(2) to 143.0(2)°. Bite angles of coordinated phen are 60.0(2)° for N(1)–Pb(1)–N(2) and 65.5° for N(3)–Pb(1)–N(4). There are no significant intermolecular interactions in the crystal structure of **2**.

Acknowledgements

This work was supported by the Major State Basic Research Development Program (Grant No. G2000077500), the Overseas Outstanding Young Scholar Fund from the National Natural Science Foundation of China (No. 20028101) and the Analytical Centre of Nanjing University.

References

- [1] E. Cox and A. Fontaine, *Bull. Soc. Chim. Fr.* 948 (1954).
- [2] H. Kohler, *Chemistry of Pseudohalides* (Elsevier, Amsterdam, 1987).
- [3] S.R. Batten, B.F. Hoskins and R. Robson, *J. Chem. Soc., Chem. Commun.* 445 (1991).
- [4] S.R. Batten, B.F. Hoskins, B. Moubaraki, K.S. Murray and R. Robson, *J. Chem. Soc., Dalton Trans.* 2977 (1999).

- [5] J.L. Manson, C. Campana and J.S. Miller, *J. Chem. Soc., Chem. Commun.* 251 (1998).
- [6] J.L. Manson, E. Ressouche and J.S. Miller, *Inorg. Chem.* **39**, 1135 (2000).
- [7] H. Hoshino, K. Iida, T. Kawamoto and T. Mori, *Inorg. Chem.* **38**, 4229 (1999).
- [8] J. Kohout, L. Jäger, M. Hvastijová and J. Kozisek, *J. Coord. Chem.* **51**, 169 (2000).
- [9] S.R. Batten, P. Jensen, B. Moubaraki, K.S. Murray and R. Robson, *J. Chem. Soc., Chem. Commun.* 439 (1998).
- [10] M. Kurmoo and C.J. Kepert, *New J. Chem.* **22**, 1515 (1998).
- [11] P. Andersen, B. Klewe and E. Thom, *Acta Chem. Scand.* **21**, 1530 (1967).
- [12] J.R. Witt and D. Britton, *Acta Crystallor.* **B27**, 1835 (1971).
- [13] Y.M. Chow and D. Britton, *Acta Crystallor.* **B31**, 1934 (1975).
- [14] Y.J. Shi, Y. Xu, Y. Zhang, B. Huang, D.R. Zhu, C.M. Jin, H.G. Zhu, Z. Yu, X.T. Chen and X.Z. You, *Chem. Lett.* 678 (2001).
- [15] Y.J. Shi, X.T. Chen, C.X. Cai, Y. Zhang, Z.L. Xue, X.Z. You, S.M. Peng and G.H. Lee, *Inorg. Chem. Commun.* **5**, 621 (2002).
- [16] Y.J. Shi, L.H. Li, Y.Z. Li, Y. Xu, X.T. Chen, Z.L. Xue and X.Z. You, *Inorg. Chem. Commun.* **5**, 1090 (2002).
- [17] Y.J. Shi, X.T. Chen, Y.Z. Li, Z.L. Xue and X.Z. You, *New J. Chem.* **26**, 1711 (2002).
- [18] Y.J. Shi, L.H. Li, Y.Z. Li, X.T. Chen, Z.L. Xue and X.Z. You, *Polyhedron* **22**, 917 (2003).
- [19] G.M. Sheldrick, SHELXS 97, Program for Crystal Structure Determinations (University of Göttingen, Germany, 1997).
- [20] G.M. Sheldrick, SHELXL 97, Program for Crystal Structure Refinements (University of Göttingen, Germany, 1997).
- [21] L. Shimon-Livny, J.P. Glusker and C.W. Bock, *Inorg. Chem.* **37**, 1853 (1998).
- [22] D.L. Reger, J.E. Collins, A.L. Rheingold, L.M. Liable-Sands and G.P.A. Yap, *Inorg. Chem.* **36**, 345 (1997).
- [23] G.A. Bowmaker, J.M. Harrowfield, H. Miyamae, T.M. Shand, B.W. Skelton, A.A. Soudi and A.H. White, *Aust. J. Chem.* **49**, 1089 (1996).