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Synthesis and characterization of lead(II)–semicarbazone complexes. Crystal structures of dinitrato di-2-pyridylketone semicarbazone lead(II) and dichloro cyclohexane-1,2-dione bis(semicarbazone) dimethylformamide lead(II)

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Abstract

Two new heptacoordinated semicarbazone lead complexes $[Pb(Hdips)(NO_3)_2]$ (**1**) and $[Pb(H_2chds)Cl_2(dmf)]$ (**2**) (Hdips = di-2-pyridylketone semicarbazone, H_2chds = cyclohexane-1,2-dione bis(semicarbazone)) were synthesized and characterized by conventional methods and X-ray crystal analysis. Compound **1** is triclinic, $a = 7.856(1)$, $b = 8.148(1)$, $c = 13.788(1)$ Å, $\alpha = 74.39(1)$, $\beta = 82.77(1)$, $\gamma = 77.21(1)°$, space group $P\bar{1}$, $Z = 2$, $R = 0.0241$. Three sites of the heptacoordinated metal are occupied by the hydrazone ligand: Pb–O(1) 2.548(4), Pb–N(3) 2.547(4), Pb–N(5) 2.598(4) Å; the two asymmetric nitrate groups complete the coordination with Pb–O(2) 2.651(5), Pb–O(3) 2.801(5) Å and Pb–O(5) 2.676(6), Pb–O(7) 2.755(4) Å. The coordination polyhedron shows a space characterized by large angles which is suggestive of lone pair stereochemical activity. Compound **2** is monoclinic: $a = 12.926(4)$, $b = 12.376(4)$, $c = 12.023(3)$ Å, $\beta = 109.70(6)°$, space group $C2/c$, $Z = 4$, $R = 0.0319$. The molecule is symmetrically related by a twofold axis passing through Pb and the midpoints of the C(2)–C(2′) and C(4)–C(4′) bonds, the ligand is planarly four-coordinated with Pb–O 2.577(6), Pb–N(3) 2.585(6) Å and lies on the equatorial plane of a distorted pentagonal bipyramid, the two chlorines are at the apical sites (Pb–Cl 2.870(3) Å), the oxygen of a dmf statistically distributed molecule affords the seventh site with Pb–O(f) 2.805(17) Å.

Key words: Crystal structures; Lead complexes; Semicarbazone complexes

Introduction

The coordinative properties of lead are of great chemical interest either for the coordination numbers and stereochemistries this heavy metal can assume or in relation to the stereochemical activity of the lone pair. Nevertheless only few structural studies of lead complexes have been undertaken in spite of the relevance of this element in the industrial field and in environmental pollution. To extend our knowledge in this research field and following our interest in the chelating behaviour of semicarbazones we report the synthesis, spectroscopic and structural properties of $[Pb(Hdips)(NO_3)_2]$ (**1**) and $[Pb(H_2chds)Cl_2(dmf)]$ (**2**) (Hdips = di-2-pyridylketone semicarbazone; H_2chds = cyclohexane-1,2-dione bis(semicarbazone)).

Experimental

All reactants were reagent grade. Cyclohexane-1,2-dione and di-2-pyridylketone were from Aldrich Chemical, semicarbazide hydrochloride was from Carlo Erba and used as received. Elemental C, H and N analyses were carried out on a Perkin-Elmer model 240 automatic equipment. IR spectra were recorded in the range 4000–200 cm^{-1} using a Nicolet 5PC FT-IR spectrophotometer.

Preparations

Di-2-pyridylketone semicarbazone

The semicarbazide hydrochloride 0.605 g (2.34×10^{-2} mol) was dissolved in 20 cm^3 of water, pH was adjusted to 6–7 by NaOH diluted solution and then refluxed with 1 g (2.34×10^{-2} mol) of di-2-pyridylketone in 50 cm^3 of methanol for 3 h. After slow evaporation a microcrystalline pale brown product was obtained (yield *c.* 75%), m.p. 214–216 °C. The product was characterized

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by mass and IR spectra: 3436w, 3288w, 3213w ν(NH), 3060w ν(CH), 1720w, 1660s ν(CO) amide I, 1588ms ν(C=C), ν(C=N), 1481ms δ(NH) + ν(C–N) cm^{-1}.

Cyclohexane-1,2-dione bis(semicarbazone)

This was prepared as previously described [1].

Complex 1

0.33 g (10^{-3} mol) of $Pb(NO_3)_2$ and 0.23 g (10^{-3} mol) of Hdips were dissolved in 50 cm^3 of methanol and then refluxed for 2 h. After slow evaporation elongated yellow prisms were formed (yield *c.* 60%). *Anal.* Found: C, 24.96; H, 1.78; N, 17.01. Calc. for $C_{12}H_{11}N_7O_7Pb$: C, 25.18; H, 1.94; N, 17.13%.

IR frequencies: 3418ms, 3330m, 3280m ν(NH) and ν(NH_2), 3230mw, 1651vs ν(CO) amide I, 1592m, 1532vs ring ν(C–N), 1450ms amide II, 1384vs ν(NO_3) cm^{-1}. Of note is the negative shift of ν(CO), ν(NH) and ν(C–N) upon coordination.

Complex 2

0.28 g (10^{-3} mol) of $PbCl_2$ and 0.23 g (10^{-3} mol) of H_2chds were dissolved in 50 cm^3 of dmf and heated to 65 °C for 15 min. After two weeks colourless elongated prisms separated (yield *c.* 40%), m.p. 245–247 °C. *Anal.* Found: C, 22.50; H, 3.51; N, 16.77. Calc for $C_{11}H_{21}Cl_2N_7O_3Pb$: C, 22.88; H, 3.66; N, 16.98%.

IR frequencies: 3388s, 3290m, 3228m, 3180sh ν(NH) and ν(NH_2), 1658vs ν(CO) amide I, 1621m, 1552m ring ν(C–N), 1511ms cm^{-1}, in this case the ν(CO) negative shift in comparison to the free ligand affords only one value in agreement to the equivalence of the carbonyls; the variations of ν(NH), ν(NH_2) and ν(C–N) can be interpreted as due to the coordination.

Crystallography

Crystal and intensity data were obtained by mounting the crystals in a random orientation on a single crystal four-circle automated diffractometer; the resulting crys-

TABLE 1 Experimental data for the crystallographic analyses

Formula	$C_{12}H_{11}N_7O_7Pb$	$C_{11}H_{21}N_7O_3Cl_2Pb$
M_r	572.46	577.44
Space group	$P\bar{1}$	$C2/c$
a (Å)	7.856(1)	12.926(4)
b (Å)	8.148(1)	12.376(4)
c (Å)	13.788(1)	12.023(3)
α (°)	74.39(1)	90
β (°)	82.77(1)	109.70(6)
γ (°)	77.21(1)	90
U ($Å^3$)	826.9(2)	1811(1)
Z	2	4
D_c	2.299	2.118
D_m	2.33	2.05
Reflections for lattice parameters		
No.	25	25
θ Range	11.1–15.6	9.8–17.4
$F(000)$	540	1104
Temperature (K)	294	294
Diffractometer	CAD4	Philips PW 1100
Crystal size (mm)	0.26 × 0.33 × 0.70	0.19 × 0.19 × 0.33
μ (cm^{-1})	103.30	97.09
Radiation, λ (Å)	Mo Kα, 0.7107	Mo Kα, 0.7107
Scan speed (° min^{-1})	0.91–3.30	3–12
Scan width (°)	$1.28 + 0.35\mathrm{tg}\theta$	$1.4 + 0.35\mathrm{tg}\theta$
θ Range (°)	3–25	3–25
h Range	15 to −15	17 to −17
k Range	15 to −15	0 to 17
l Range	0 to 20	0 to 16
Standard reflection	0 −4 1	−6 0 4
Max. intensity variation (%)	3.3	4.0
Scan mode	ω–2θ	ω–2θ
No. reflections measured	3037	1777
No. reflections used in the refinement	2584	1388
No. refined parameters	244	104
$R = \Sigma\lvert\Delta F\rvert/\Sigma\lvert F_o\rvert$	0.0241	0.0319
$R' = [\Sigma w(\Delta F^2)/\Sigma w F_o^2]^{1/2}$	0.0289	0.0331
g in $w = [\sigma^2(F_o) + gF_o^2]^{-1}$	2.92×10^{-4}	5×10^{-3}
Max., min. height in final ΔF map (e $Å^{-3}$)	2.1, −1.2	0.33, −0.35

TABLE 2 Fractional atomic coordinates (×10⁵ for Pb and ×10⁴ for the others) with e.s d.s in parentheses for [Pb(Hdips)(NO_3)$_2$]

Atom	*x/a*	*y/b*	*z/c*
Pb	−21819(2)	56877(2)	90877(1)
O(1)	540(5)	6766(5)	9268(3)
O(2)	−270(6)	5337(5)	7401(3)
O(3)	−662(7)	2840(6)	8288(3)
O(4)	1337(9)	3013(8)	7094(4)
O(5)	−2813(6)	7605(7)	10441(4)
O(6)	−5299(8)	8771(8)	11037(4)
O(7)	−5160(6)	6870(6)	10186(4)
N(1)	1668(6)	9148(6)	9188(4)
N(2)	−582(5)	9386(5)	8238(3)
N(3)	−1863(5)	8704(5)	8027(3)
N(4)	−1206(6)	11057(6)	5911(4)
N(5)	−4469(5)	7279(6)	7765(3)
N(6)	143(7)	3717(7)	7593(4)
N(7)	−4429(7)	7779(7)	10566(4)
C(1)	563(6)	8346(7)	8931(4)
C(2)	−2854(6)	9585(6)	7308(3)
C(3)	−2533(7)	11233(6)	6596(4)
C(4)	−3619(9)	12825(8)	6617(5)
C(5)	−3299(12)	14276(8)	5880(5)
C(6)	−1887(10)	14107(8)	5179(5)
C(7)	−900(9)	12472(8)	5225(4)
C(8)	−4315(6)	8827(6)	7151(4)
C(9)	−5477(8)	9677(8)	6429(4)
C(10)	−6843(8)	8931(9)	6337(5)
C(11)	−7052(8)	7383(9)	6958(5)
C(12)	−5775(8)	6549(8)	7667(4)

tal data and details concerning data collection and refinements are given in Table 1. For both compounds after the usual data reduction an empirical correction was applied following the method of Walker and Stuart [2]. The structures were solved by Patterson and Fourier methods and refined by full-matrix least-squares with anisotropic thermal parameters; hydrogens were not located. The final atomic coordinates are given in Tables 2 and 3 for **1** and **2**, respectively; the atomic scattering factors were taken from the International Tables [3]. All the calculations were performed on a GOULD 33/77 computer with SHELX [4], ORTEP [5] and PARST [6] programs.

TABLE 3. Fractional atomic coordinates (×10⁴) with e.s.d s in parentheses for [Pb(H_2chds)Cl_2(dmf)]

Atom	*x/a*	*y/b*	*z/c*
Pb	5000	3090 1(3)	2500
Cl	3015(2)	3500(2)	2998(2)
O	3832(5)	2994(4)	292(5)
N(1)	2871(6)	4835(5)	218(5)
N(3)	4409(5)	4889(5)	1371(5)
C(1)	3509(6)	3826(6)	−276(7)
C(2)	4652(6)	5821(6)	1874(6)
C(3)	4229(8)	6869(7)	1258(8)
C(4)	4892(8)	7840(7)	1850(8)
N(f)	0000	5000	5000
O(f)	476(13)	3946(13)	6768(14)
C(1f)	−253(17)	4322(19)	5833(18)
C(2f)	1146(21)	5263(20)	5238(22)
C(3f)	−859(18)	5488(19)	4118(20)

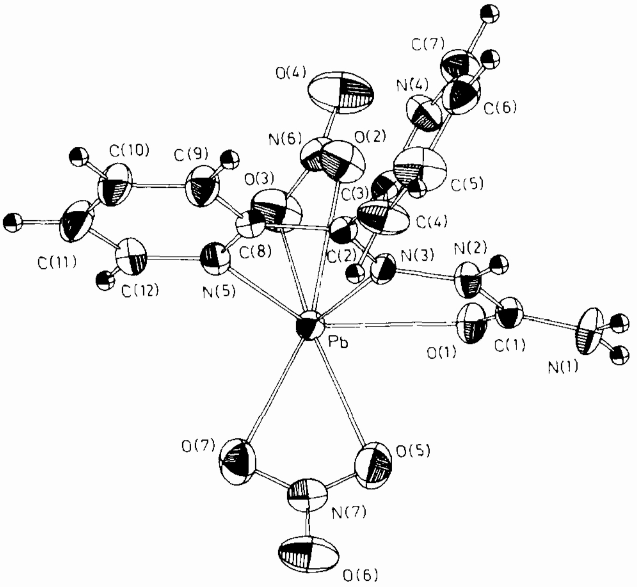

Fig 1 Perspective view of the structure of **1**.

Discussion

In complex **1** the metal is heptacoordinated with two bidentate asymmetric nitrate groups and one O,N,N′ ligand chelate molecule; the resulting coordination geometry cannot be described as a regular polyhedron. The neighbourhood of the lead (Fig. 1) is occupied by the carbonyl oxygen, and the N(3) hydrazonic and N(5) pyridine atoms of the Hdips terdentate moiety, while the other pyridyl group is uncoordinated These atoms, having shorter distances 2.548(4), 2.547(4), 2.598(4) Å (Table 4(a)), belong to two five-term chelate rings in which the light atoms are planar; their mean planes form a dihedral angle of 13.4(2)°. The chelate rings show typical angles at the metal of 62.7(1) and 62.5(1)°; the internal angles at N(2), C(2) and C(8) smaller than 120° seem to be influenced by the constraints of the chelation. The metal is out of the mean planes O(1)C(1)N(2)N(3) and N(3)C(2)C(8)N(5) by 0.4655(2) and 0.1369(2) Å, respectively; the coordination distances are comparable to those found, for example, in dilead bis(ethylene-diaminetetraacetate) trihydrate Pb–O 2.540–2.645, Pb–N 2.572–2.624 Å [7]. The two bidentate nitrate groups form a dihedral angle of 61.9(2)° and show different bond distances: Pb–O(2) (2.651(5) Å) and Pb–O(5) (2.676(6) Å) are shorter than Pb–O(3) (2.801(5) Å) and Pb–O(7) (2 755(4) Å) so that asym-

TABLE 4 (a) Bond distances (Å) and angles (°), (b) hydrogen bonds (Å and °) for [Pb(Hdips)(NO$_3$)$_2$]

(a)

Pb–O(1)	2.548(4)	Pb–O(2)	2.651(5)
Pb–O(3)	2.801(5)	Pb–O(5)	2.676(6)
Pb–O(7)	2.755(4)	Pb–N(3)	2.547(4)
Pb–N(5)	2.598(4)		
O(1)–C(1)	1.249(6)		
N(1)–C(1)	1.327(8)	N(2)–N(3)	1.347(7)
N(2)–C(1)	1.372(6)	N(3)–C(2)	1.291(6)
N(4)–C(3)	1.326(7)	N(4)–C(7)	1.326(7)
N(5)–C(12)	1.332(8)	C(2)–C(3)	1.486(6)
C(2)–C(8)	1.481(8)	C(3)–C(4)	1.391(8)
C(4)–C(5)	1.384(9)	C(5)–C(6)	1.385(10)
C(6)–C(7)	1.375(9)	N(5)–C(8)	1.337(6)
C(8)–C(9)	1.372(7)	C(9)–C(10)	1.380(10)
C(11)–C(12)	1.418(9)	C(10)–C(11)	1.351(9)
O(2)–N(6)	1.249(7)	O(3)–N(6)	1.228(7)
O(4)–N(6)	1.222(8)	O(5)–N(7)	1.241(7)
O(6)–N(7)	1.212(8)	O(7)–N(7)	1.275(8)

N(3)–Pb–N(5)	62.5(1)	O(1)–Pb–O(5)	68.9(1)
O(7)–Pb–N(5)	74.7(1)	O(1)–Pb–O(3)	100.5(1)
O(7)–Pb–N(3)	94.5(1)	O(1)–Pb–O(2)	78.1(1)
O(5)–Pb–N(5)	103.9(1)	Pb–O(1)–C(1)	118.5(3)
O(5)–Pb–N(3)	76.7(1)	Pb–O(2)–N(6)	99.6(3)
O(5)–Pb–O(7)	46.7(1)	Pb–O(2)–O(3)	70.6(2)
O(3)–Pb–N(5)	99.1(1)	Pb–O(3)–O(2)	63.3(2)
O(3)–Pb–N(3)	117.9(1)	Pb–O(3)–N(6)	92.9(4)
O(3)–Pb–O(7)	140.1(1)	Pb–O(3)–O(4)	121.5(3)
O(3)–Pb–O(5)	156.7(1)	Pb–O(5)–N(7)	99.6(4)
O(2)–Pb–N(5)	79.7(1)	Pb–O(7)–N(7)	94.8(3)
O(2)–Pb–N(3)	71.9(1)	Pb–N(3)–N(2)	117.2(3)
O(2)–Pb–O(7)	154.4(1)	Pb–N(3)–C(2)	123.3(3)
O(2)–Pb–O(5)	142.1(1)	Pb–N(5)–C(12)	120.8(4)
O(2)–Pb–O(3)	46.1(1)	Pb–N(5)–C(8)	120.1(3)
O(1)–Pb–N(5)	124.8(1)	O(1)–Pb–O(7)	115.5(1)
O(1)–Pb–N(3)	62.7(1)	C(3)–C(2)–C(8)	118.8(4)
N(3)–N(2)–C(1)	117.9(4)	N(4)–C(3)–C(2)	115.1(4)
N(2)–N(3)–C(2)	119.5(4)	C(2)–C(3)–C(4)	121.9(5)
C(3)–N(4)–C(7)	118.1(5)	N(4)–C(3)–C(4)	122.9(5)
C(8)–N(5)–C(12)	119.0(5)	C(3)–C(4)–C(5)	118.0(6)
O(3)–N(6)–O(4)	120.4(6)	C(4)–C(5)–C(6)	119.3(7)
O(2)–N(6)–O(4)	120.3(6)	C(5)–C(6)–C(7)	117.9(6)
O(2)–N(6)–O(3)	119.3(5)	N(4)–C(7)–C(6)	123.8(6)
O(6)–N(7)–O(7)	120.4(6)	N(5)–C(8)–C(2)	116.5(4)
O(5)–N(7)–O(7)	117.9(5)	C(2)–C(8)–C(9)	122.0(5)
O(5)–N(7)–O(6)	121.7(6)	N(5)–C(8)–C(9)	121.4(5)
N(1)–C(1)–N(2)	114.9(5)	C(8)–C(9)–C(10)	119.5(6)
O(1)–C(1)–N(2)	120.9(5)	C(9)–C(10)–C(11)	120.5(6)
O(1)–C(1)–N(1)	124.2(5)	C(10)–C(11)–C(12)	117.1(6)
N(3)–C(2)–C(8)	117.3(4)	N(5)–C(12)–C(11)	122.4(6)
N(3)–C(2)–C(3)	123.7(4)		

(b) Possible hydrogen bonds

Donor–H	Donor···Acceptor	H···Acceptor	Donor–H···Acceptor
N(2)–H(3)	N(2)···O(3')	H(3)···O(3')	N(2)–H(3)···O(3')
1.000	2.820(7)	1.996	138.1
N(1)–H(2)	N(1)···O(7")	H(2)···O(7")	N(1)–H(2)···O(7")
1.000	2.998(6)	2.054	156.4

Equivalent positions (i) x, $y+1$, z, (ii) $x+1$, y, z

metrical coordination results. Similar values are found in other lead bidentate nitrate complexes such as bis(thiourea)lead(II) nitrate (2.68(1) and 2.85(1) Å) [8] and hexakis(1-methylimidazolidine-2(3*H*)-thione) tetrakis(nitrato)-dilead(II) (2.524(5) and 2.748(6) Å) [9]. The coordination sphere of the lead exhibits a large space characterized by a number of angles at the metal greater than 120° on the opposite side of the free pyridyl group suggestive of lone pair stereochemical activity. Bond distances and angles in the ligand show no abnormal features; the fragment O(1)C(1)N(2)-N(3)C(2) is a π-delocalized system with the shortest bond N(3)–C(2) 1.291(6) Å. Packing is granted by van der Waals contacts and by the hydrogen bonds involving NH groups and nitrate oxygens of adjacent molecules (Table 4(b)).

The molecule of **2** is symmetrically related: the metal is located on a twofold axis passing through the midpoints of the C(2)–C(2′) and C(4)–C(4′) bonds (Fig. 2). The lead is seven-coordinated with two Pb–O (2.577(6) Å) and two Pb–N(3) (2.585(6) Å) shorter bond distances involving the hydrazone molecule, two Pb–Cl (2.870(8) Å) bonds and one Pb–O(f) (2.805(17) Å) bond of the dmf statistically distributed molecule (Table 5(a)). In fact the nitrogen atom of this moiety lies on a symmetry centre so that its remaining atoms and, in particular the coordinated O(f) oxygen, are statistically distributed in two centrosymmetric positions (only alternatively present) and belong to two adjacent coordination polyhedra The presence of the twofold axis means that when the unprimed dmf is coordinated, the primed one, related by this symmetry element, exists in the centrosymmetrical position. The ligand is coordinated in a way similar (excepting for the bridging carbonyl) to that found in $[Co_2(H_2chds)_2(OH_2)(NO_3)_2](NO_3)$ [1] forming three five-term chelate rings with characteristic angles at the metal of 62.1(2) and 61.1(2)°; the bond distances and angles agree well with those of the cobalt and nickel complexes and also of the free ligand reported in ref. 1. The girdle formed by the atoms of the chelate rings is planar with lead out only by 0.0880(6) Å; the two chlorines occupy approximately apical sites with

TABLE 5 (a) Bond distances (Å) and angles (°) with e.s.d s in parentheses, (b) hydrogen bonds (Å and °) for $[Pb(H_2chds)Cl_2(dmf)]$

(a)

Pb–Cl	2 870(3)	C(2)–C(2′)	1.471(9)
Pb–O	2 577(6)	C(2)–C(3)	1 50(1)
Pb–N(3)	2 585(6)	C(3)–C(4)	1 51(1)
Pb–O(f)	2 805(17)	C(4)–C(4′)	1.49(1)
O–C(1)	1.228(9)	N(f)–C(1f)	1 43(2)
N(1)–C(1)	1.347(9)	N(f)–C(2f)	1 45(3)
N(2)–N(3)	1 362(8)	N(f)–C(3f)	1 39(2)
N(2)–C(1)	1.37(1)	O(f)–C(1f)	1.29(2)
N(3)–C(2)	1.29(1)		
N(3)–Pb–N(3′)	61 1(2)	C(1f)–N(f)–C(2f)	117(1)
O–Pb–N(3′)	123 2(2)	N(f)–C(1f)–O(f)	123(2)
O–Pb–N(3)	62.1(2)	N(f)–C(1f)–O(f)	123(2)
Cl–Pb–N(3′)	82 3(2)	N(2)–N(3)–C(2)	119.4(6)
Cl–Pb–N(3)	80 2(2)	N(1)–C(1)–N(2)	114.4(7)
Cl–Pb–O	88 2(2)	O–C(1)–N(2)	122.5(7)
O(f)–Pb–N(3′)	131 8(4)	O–C(1)–N(1)	123.1(7)
O(f)–Pb–N(3)	151.8(4)	N(3)–C(2)–C(3)	123.3(7)
O(f)–Pb–O	99.6(4)	N(3)–C(2)–C(2′)	116 6(7)
O(f)–Pb–Cl	77 7(4)	C(2′)–C(2)–C(3)	120 0(7)
Pb–O(f)–C(1f)	123(2)	N(3′)–C(2′)–C(2)	116 6(7)
Pb–O–C(1)	120.4(5)	C(2)–C(3)–C(4)	113 6(7)
N(3)–N(2)–C(1)	117.3(6)	C(3)–C(4)–C(4′)	111.7(8)
Pb–N(3)–N(2)	117.7(4)	C(2f)–N(f)–C(3f)	124(1)
Pb–N(3)–C(2)	122.8(5)	C(1f)–N(f)–C(3f)	119(1)

(b) Hydrogen bonds

Donor–H	Donor · Acceptor	H Acceptor	Donor–H · · Acceptor
N(2)–H(3)	N(2) · Cl^{ii}	H(3) · · Cl^{ii}	N(2)–H(3) · · Cl^{ii}
0.974	3.251(8)	2.298	165.8
N(1)–H(1)	N(1) Cl^{ii}	H(1) · · Cl^{ii}	N(1)–H(1) Cl^{ii}
0 875	3 397(9)	2 640	145.4
N(1)–H(2)	N(1) Cl^{iii}	H(2) · Cl^{iii}	N(1)–H(2) Cl^{iii}
0 951	3.426(9)	2.478	174 6

Equivalent positions: (i) $1-x$, y, $\frac{1}{2}-z$, (ii) x, $1-y$, $z-\frac{1}{2}$; (iii) $\frac{1}{2}-x$, $\frac{1}{2}-y$, $-z$

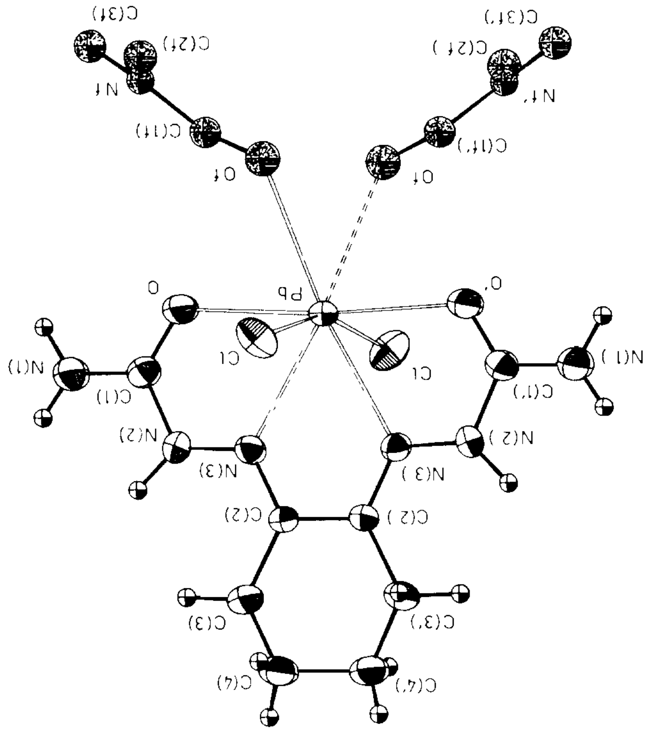

Fig 2 Perspective view of the structure of **2**

respect to this plane Cl–Pb–O 88.2(2)° and Cl–Pb–N(3) 80.2(2)°; the O(f) atom lies 1.25(2) Å out so that coordination can be described as very distorted bipyramidal pentagonal. It is interesting to compare the present complex with chloro[2,6-diacetylpyridine-bis(semicarbazone)] lead(II) nitrate [10]. the quinquedentate ligand, approximately planar, occupies the equatorial sites of a pentagonal bipyramid with Pb–N 2.523(8), 2.556(8) and 2 668(7) Å, Pb–O 2.607(6) and 2.872(7) Å. In this case the ligand is asymmetrically chelated and only the shorter values are in agreement with the present ones The Pb–Cl value of 2.707(4) Å is shorter if compared with our compound, 2 870(3) Å, and this difference could be attributed to the *trans* influence; comparable values 2.830(7), 2.878(7), 2.879(7), 2 911(7) Å are found, for example, in bis(n-propylammonium)tetrachloroplumbate [11] in which all the chlorines are bridging. In this structure hydrogen bonds play a basic role in the packing (Table 5(b)) connecting all NH groups with chlorine atoms of adjacent molecules.

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