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### New mixed-anion 2-aminomethylpyridine (AMP) lead(II) complexes, $Pb(AMP)_2(ClO_4)_n$ ( $X=CH_3COO^-$ and $n=1$ or $X=$ and $n=2$ ); crystal and molecular structure of $[Pb(AMP)_2](ClO_4)(NO_3)$

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## New mixed-anion 2-aminomethylpyridine (AMP) lead(II) complexes, $\text{Pb}(\text{AMP})_n(\text{ClO}_4)\text{X}$ , ( $\text{X} = \text{CH}_3\text{COO}^-$ and $n = 1$ or $\text{X} = \text{NO}_3^-$ and $n = 2$ ); crystal and molecular structure of $[\text{Pb}(\text{AMP})_2](\text{ClO}_4)(\text{NO}_3)$

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Lead(II) complexes with 2-aminomethylpyridine (AMP) containing two different anions,  $\text{Pb}(\text{AMP})_n(\text{ClO}_4)\text{X}$  ( $\text{X} = \text{CH}_3\text{COO}^-$  and  $n = 1$  or  $\text{X} = \text{NO}_3^-$  and  $n = 2$ ), have been synthesized using a direct synthetic method and characterized by elemental analysis, IR,  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectroscopy. The structure of  $[\text{Pb}(\text{AMP})_2](\text{ClO}_4)(\text{NO}_3)$  was confirmed by X-ray crystallography; single-crystal X-ray data show the complex to be monomeric. The Pb atom has an unsymmetrical four-coordinate geometry, being coordinated by four nitrogen atoms of the two "AMP" ligands. The arrangement of the "AMP" ligands exhibits a coordination gap around the Pb(II) ion, possibly occupied by a stereoactive lone pair of electrons on lead(II), and the coordination around the lead atoms is hemidirected.

**Keywords:** Lead(II) complexes; Crystal structure; Mixed-anion complexes; 2-Aminomethylpyridine ligand

### 1. Introduction

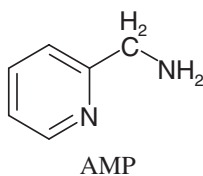
The chemistry of lead(II) has attracted interest not only because of the potential toxicity of lead but also because this is a relatively unexplored area of coordination chemistry [1, 2]. According to its electronic configuration ( $[\text{Xe}]4f^{14}5d^{10}6s^2$ ) and size, the lead(II) ion exhibits an inert electron pair and variable coordination numbers and geometries. For these various coordination numbers and coordination geometries, the role of the lone pair of electrons is important.

Lone pair activity may depend on the following factors: (1) low *versus* high coordination number, (2) hard *versus* soft ligands, (3) attractive *versus* repulsive interactions

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among ligands, (4) whether the lone pair has p character, (5) fewer or more electron donation from ligands to metal [3].

Recent reports of the crystal structures of mixed-anion adducts [Pb(phen)(O<sub>2</sub>CCH<sub>3</sub>)(O<sub>2</sub>ClO<sub>2</sub>)] [4], [Pb(phen)(O<sub>2</sub>CCH<sub>3</sub>)(O<sub>2</sub>NO)] [5], [Pb(phen)(O<sub>2</sub>CCH<sub>3</sub>)(NCS)] [6] and the 1:2 adduct of [Pb(phen)<sub>2</sub>(CH<sub>3</sub>COO)]X (X = NCS<sup>-</sup>, NO<sub>3</sub><sup>-</sup> and ClO<sub>4</sub><sup>-</sup>) [7] described the influence of different anions on the coordination stereochemistry of the lead(II) ion. In the present article, we report the syntheses and characterization of two new mixed-anion lead(II) complexes, Pb(AMP)<sub>n</sub>(ClO<sub>4</sub>)X (X = CH<sub>3</sub>COO<sup>-</sup> and n = 1 or X = NO<sub>3</sub><sup>-</sup> and n = 2), containing 2-aminomethylpyridine (AMP). This ligand is an unsymmetrical *N,N'*-bidentate ligand combining both aromatic and aliphatic groups, with no constraint on the coplanarity of the aliphatic group with the aromatic ring.



## 2. Experimental

### 2.1. Physical measurements

IR spectra were recorded as Nujol mulls using Perkin-Elmer 597 and Nicolet 510P spectrophotometers. Microanalyses were carried out using a Heraeus CHN-O-RAPID instrument.

### 2.2. Preparation of Pb(AMP)(ClO<sub>4</sub>)(CH<sub>3</sub>COO)

2-Aminomethylpyridine (0.216 g, 2 mmol) was placed in one arm of a branched tube and a mixture of lead(II) acetate (0.36 g, 1 mmol) and sodium perchlorate (0.246 g, 2 mmol) in the other. Methanol was carefully added to fill both arms, then the tube was sealed and the ligand-containing arm immersed in a bath at 60°C, while the other was kept at ambient temperature. After 6 days, a white compound (dp 250°C) had deposited in the cooler arm. Yield: 0.190 g (40%). Anal. Calcd for C<sub>8</sub>H<sub>11</sub>ClN<sub>2</sub>O<sub>6</sub>Pb (%): C, 20.23; H, 2.32; N, 5.90. Found: C, 20.80; H, 2.70; N, 5.95. IR (cm<sup>-1</sup> selected bands): 616(s), 927(w), 1075(vs), 1100(vs), 1419(s), 1584(s), 1640(s), 2895(w), 3065(w), 3245(w), 3255(w). <sup>1</sup>H NMR (DMSO; δ): 1.75 (s, 3H), 3.50 (s, 2H), 4.60 (s, 2H), 7.60 (m, 2H), 8.00 (m, 1H), 8.80 (d, 1H). <sup>13</sup>C{<sup>1</sup>H} NMR (DMSO; δ): 26.99 (<sup>13</sup>CH<sub>3</sub>-COO), 179.05 (CH<sub>3</sub>-<sup>13</sup>COO), 46.13 (Py-<sup>13</sup>CH<sub>2</sub>-NH<sub>2</sub>), 79.37, 123.26, 139.18, 148.56, 161.54.

### 2.3. Preparation of [Pb(AMP)<sub>2</sub>](ClO<sub>4</sub>)(NO<sub>3</sub>)

This complex was prepared by a method analogous to that used for [Pb(AMP)(ClO<sub>4</sub>)(CH<sub>3</sub>COO)]. Reactant materials: AMP, lead(II) nitrate, sodium perchlorate (2:1:2); colorless crystals (yield 0.214 g, 45%), m.p. 225°C. Anal. Calcd for C<sub>12</sub>H<sub>16</sub>ClN<sub>5</sub>O<sub>7</sub>Pb (%): C, 24.61; H, 2.73; N, 11.96. Found: C, 24.10; H, 2.55;

Table 1. Crystal data and structure refinement for [Pb(AMP)<sub>2</sub>](ClO<sub>4</sub>)(NO<sub>3</sub>).

Complex	[Pb(AMP) <sub>2</sub> ](ClO <sub>4</sub> )(NO <sub>3</sub> )
Empirical formula	C <sub>12</sub> H <sub>16</sub> ClN <sub>5</sub> O <sub>7</sub> Pb
Molecular weight	584.94
Temperature (K)	193(2)
Wavelength (Å)	0.71073
Crystal system	Triclinic
Space group	<i>P</i> 1
<i>a</i> (Å)	7.7830(11)
<i>b</i> (Å)	8.1810(12)
<i>c</i> (Å)	13.794(2)
$\alpha$ (°)	84.953(12)
$\beta$ (°)	83.778(12)
$\gamma$ (°)	89.314(12)
<i>V</i> (Å <sup>3</sup> )	869.7(2)
<i>Z</i>	2
<i>D</i> <sub>calcd</sub> (g cm <sup>-3</sup> )	2.226
$\mu$ (mm <sup>-1</sup> )	9.898
<i>F</i> (000)	554
Crystal size (mm <sup>3</sup> )	0.62 × 0.58 × 0.40
$\theta$ range (°)	1.49 to 25.75
Index ranges	-9 ≤ <i>h</i> ≤ 9, -9 ≤ <i>k</i> ≤ 9, -16 ≤ <i>l</i> ≤ 16
Reflections collected	11,187
Independent reflections ( <i>R</i> <sub>int</sub> )	3282 (0.0455)
Reflections observed (> 2 $\sigma$ )	3113
Absorption correction	Numerical
Max. and min. transmissions	0.077 and 0.023
Data/Restraints/Parameters	3282/0/230
Goodness-of-fit on <i>F</i> <sup>2</sup>	1.140
<i>R</i> [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )]	<i>R</i> 1 = 0.0455, <i>wR</i> 2 = 0.1055
<i>R</i> (all data)	<i>R</i> 1 = 0.0486, <i>wR</i> 2 = 0.1074
Largest diff. peak and hole (e Å <sup>-3</sup> )	1.974 and -1.557

N, 11.50. IR (cm<sup>-1</sup>) selected bonds: 620(s), 754(s), 928(m), 1082(vs), 1105(vs), 1370(vs), 1429(m), 1587(m), 2900(w), 3235(m). <sup>1</sup>H NMR (DMSO;  $\delta$ ): 3.70 (s, 2H), 4.60 (s, 2H), 7.55 (m, 2H), 8.00 (m, 1H), 8.75 (d, 1H). <sup>13</sup>C{<sup>1</sup>H} NMR (DMSO;  $\delta$ ): 45.98 (Py-<sup>13</sup>CH<sub>2</sub>-NH<sub>2</sub>), 123.45, 139.03, 148.63, 161.65, 178.12.

**2.3.1. Crystallography.** Crystallographic measurements were made at 193(2) K using an STOE IPDS II diffractometer equipped with an Oxford Cryostream low temperature unit. The intensity data were collected (STOE X-Area program package) within the range  $1.49 \leq \theta \leq 25.75^\circ$  using graphite-monochromated Mo-K $\alpha$  radiation. Intensities of 3282 unique reflections were obtained, from which 3113 with *I* > 2 $\sigma$ (*I*) were observed. The structure was solved by direct methods and refined by full-matrix least-squares techniques on *F*<sup>2</sup>. Structure solution and refinement were accomplished using SIR97, SHELXL97 and WinGX [8, 9]. A numerical absorption correction was applied. Crystal data are shown in table 1.

### 3. Discussion

#### 3.1. Synthesis

Reaction between AMP and mixtures of lead(II) acetate with sodium perchlorate and lead(II) nitrate with sodium perchlorate produced Pb(AMP)(CH<sub>3</sub>COO)(ClO<sub>4</sub>)

and  $[\text{Pb}(\text{AMP})_2](\text{NO}_3)(\text{ClO}_4)$ . IR spectra of both complexes show absorption bands resulting from the skeletal vibrations of the aromatic rings in the  $1400\text{--}1600\text{ cm}^{-1}$  range. Characteristic bands of the acetate anion in the  $\text{Pb}(\text{AMP})(\text{CH}_3\text{COO})(\text{ClO}_4)$  complex appear at  $1640\text{ cm}^{-1}$   $\nu_{\text{as}}(\text{C-O})$  and  $1419\text{ cm}^{-1}$   $\nu_{\text{sym}}(\text{C-O})$ . The  $\Delta$  value ( $\nu_{\text{as}} - \nu_{\text{sym}}$ ) indicates that the acetate anion bridges the  $\text{Pb}^{\text{II}}$  centers [10, 11]. The  $[\text{Pb}(\text{AMP})_2](\text{NO}_3)(\text{ClO}_4)$  complex shows  $\nu(\text{NO}_3)$  at  $1370\text{ cm}^{-1}$  and  $\nu(\text{ClO}_4)$  at  $1105\text{ cm}^{-1}$ . Attempts to isolate 1:2 adducts of  $\text{Pb}(\text{AMP})_2(\text{CH}_3\text{COO})(\text{ClO}_4)$ , were not successful and each time the 1:1 adduct was isolated, but we were successful in the preparation of a 1:2 adduct of  $\text{NO}_3^-$ . This is consistent with the assumption that acetate anion binding is stronger than nitrate ( $\text{CH}_3\text{COO}^- > \text{NO}_3^-$ ), resulting in the acetate anion coordinating to the lead(II) atom, preventing formation of 1:2 adducts. In lead(II) complexes containing the acetate ligands, the acetate anions are coordinated to lead(II) as bidentate in  $[\text{Pb}(\text{phen})_2(\text{CH}_3\text{COO})]\text{X}$  ( $\text{X} = \text{NCS}^-$ ,  $\text{NO}_3^-$  and  $\text{ClO}_4^-$ ) [7] and  $[\text{Pb}(\text{bpy})_2(\text{CH}_3\text{COO})(\text{ClO}_4)]$  [12], as both bidentate and bridging (tridentate) in  $[\text{Pb}(\text{phen})(\text{O}_2\text{CCH}_3)_2]_2$  [13],  $[\text{Pb}(\text{phen})(\text{O}_2\text{CCH}_3)(\text{NCS})]_2$  [6] and  $\text{Pb}(\text{H}_2\text{O})(\mu\text{-OAc})(\mu\text{-sac})_n$  [14], and also as both bidentate and bridging from two sides (tetradentate) in  $[\text{Pb}(\text{phen})(\text{O}_2\text{CCH}_3)(\text{O}_2\text{ClO}_2)]_n$  [4],  $[\text{Pb}(\text{phen})(\text{O}_2\text{CCH}_3)(\text{O}_2\text{NO})]_n$  [5] and  $[\text{Pb}(\text{O}_2\text{CCH}_3)_2]_n$  [15]. The structure of  $[\text{Pb}(\text{AMP})_2](\text{NO}_3)(\text{ClO}_4)$  was confirmed by X-ray crystallography.

### 3.2. Crystal structure of $[\text{Pb}(\text{AMP})_2](\text{NO}_3)(\text{ClO}_4)$

An ORTEP drawing of  $[\text{Pb}(\text{AMP})_2](\text{NO}_3)(\text{ClO}_4)$  is shown in figure 1, and selected bond distances and angles are listed in table 2. Single-crystal analysis reveals that  $[\text{Pb}(\text{AMP})_2](\text{NO}_3)(\text{ClO}_4)$  crystallizes in triclinic space group  $P\bar{1}$ . The structure of the complex consists of discrete  $[\text{Pb}(\text{AMP})_2]^{2+}$  cations and two different anions,  $\text{ClO}_4^-$  and  $\text{NO}_3^-$ . The Pb is coordinated by four nitrogen atoms from two ‘‘AMP’’ ligands

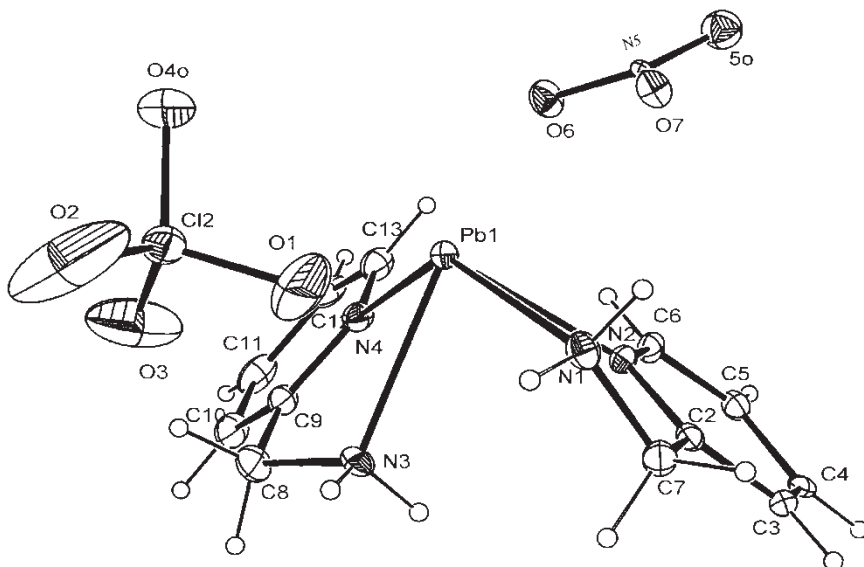


Figure 1. ORTEP diagram of  $[\text{Pb}(\text{AMP})_2](\text{ClO}_4)(\text{NO}_3)$ .

Table 2. Bond lengths (Å) and angles (°) for [Pb(AMP)<sub>2</sub>](ClO<sub>4</sub>)(NO<sub>3</sub>).

Pb1–N1	2.493(7)	N2–Pb1–N3	81.2(3)
Pb1–N2	2.462(7)	N2–Pb1–N1	68.1(3)
Pb1–N3	2.473(8)	N1–Pb1–N3	81.1(3)
Pb1–N4	2.615(7)	N2–Pb1–N4	81.2(3)
		N4–Pb1–N3	65.3(3)
		N1–Pb1–N4	137.6(2)

with Pb–N distances of 2.615(7), 2.493(7), 2.462(7) and 2.473(8) Å. There are weak interactions of lead(II) with oxygen atoms of the nitrate; each Pb atom in this structure along with four normal bonds forms two weak Pb...O bonds, 2.855(3) and 2.889(3) Å. The distance of the oxygen atom of the perchlorate anion is 3.337(3) Å.

The arrangement of the “AMP” ligands suggests a gap or hole in the coordination geometry around the metal ion [the N(1)–Pb–N(4) angle is 137.6(2)°], possibly occupied by a stereoactive lone pair of electrons on lead(II). The observed shortening of the Pb–N bonds on the side of Pb(II) opposite to the putative lone pair [2.493(7), 2.462(7) and 2.473(8) Å compared with 2.615(7) Å adjacent to the lone pair] supports the presence of this feature [16]. Hence, the geometry of the nearest coordination environment of every lead atom is also likely to be caused by the geometrical constraints of the coordinated “AMP” ligands and by the influence of a stereochemically active lone pair of electrons in a hybrid orbital on the metal atom, which occupies one equatorial coordination site of a pseudotrigonal bipyramid. For the structure described here, coordination around the lead atoms is hemidirected [3] with a significant gap *trans* to the chelating “AMP” ligands. With an unsymmetrical bidentate ligand such as “AMP”, the possibility arises of isomerism related to its orientation in the metal coordination sphere. The coordination geometry of the “AMP” ligands in this complex is distorted *cis*: N(aromatic)–Pb–N(aromatic) is 81.2(3)°, N(aliphatic)–Pb–N(aliphatic) is 81.1(3)° and N(aliphatic)–Pb–N(aromatic) is 137.6(2)°.

There are  $\pi$ – $\pi$  stacking [17] interactions between the parallel aromatic rings belonging to adjacent chains in this complex, as shown in figures 2 and 3. In the crystal reported here, the interplanar distance is 3.75 Å.

A simple model to describe the nature of  $\pi$ – $\pi$  interactions has been developed by Hunter and Sanders [18]. This model predicts that face-to-face  $\pi$ -stacked interactions will be disfavoured because of the dominance of  $\pi$ – $\pi$  repulsion. Electron-poor aromatic groups interact most strongly with electron-rich aromatic groups [19, 20]. Hence, as the molecules of this packing include equally or almost equally electron-deficient, or indeed electron-rich rings, it can be expected that within [Pb(AMP)<sub>2</sub>](NO<sub>3</sub>)(ClO<sub>4</sub>), face-to-face  $\pi$ -stacked interactions are disfavoured because of the dominance of  $\pi$ – $\pi$  repulsion.

Thus two factors, lone pair activity and  $\pi$ – $\pi$  stacking, control the coordination sphere of this complex. The obvious question then is whether the lone pair activity has stretched the coordinate bonds to result in ligand stacking or whether it is the stacking interaction that has imposed a positioning of the donor atoms to form a gap in the coordination sphere. As the model face-to-face  $\pi$ -stacked interactions must be disfavoured because of the dominance of  $\pi$ – $\pi$  repulsion, the lone pair activity may be the most important factor affecting formation of  $\pi$ – $\pi$  stacking.

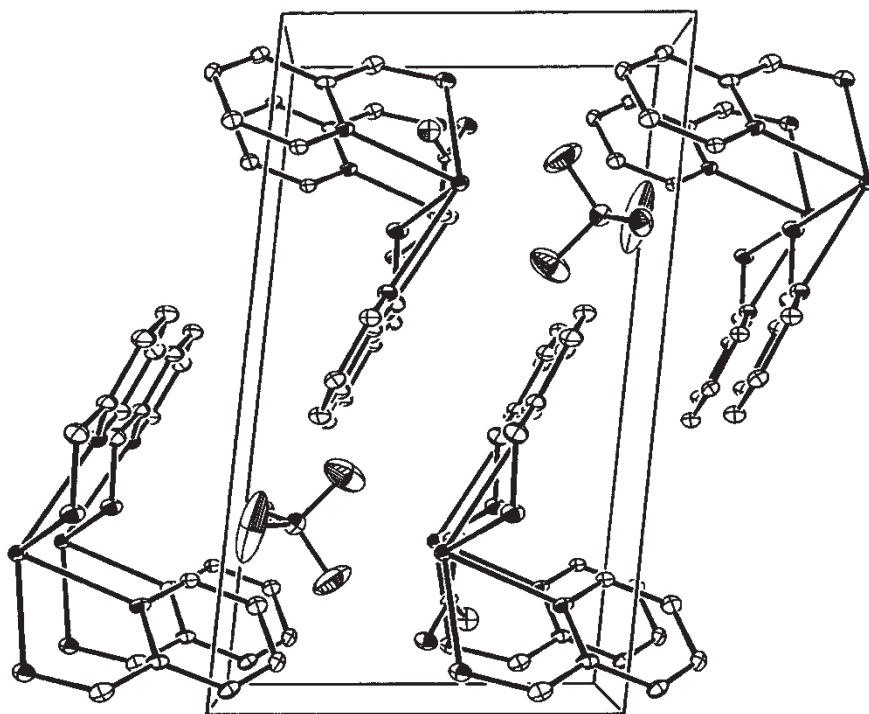


Figure 2. The unit cell of  $[\text{Pb}(\text{AMP})_2](\text{ClO}_4)(\text{NO}_3)$ , showing  $\pi$ - $\pi$  stacking interactions.

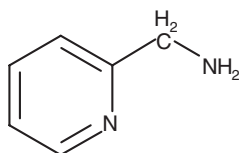


Figure 3. Projection of nearest-neighbor pairs in the  $\pi$ - $\pi$  stacks of heteroaromatic bases in  $[\text{Pb}(\text{AMP})_2](\text{NO}_3)(\text{ClO}_4)$ .

### Supplementary materials

Crystallographic data for the structure reported in the article have been deposited with the Cambridge Crystallographic Data Centre as CCDC No. 249990. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-1223-336-033; e-mail: [deposit@ccdc.cam.ac.uk](mailto:deposit@ccdc.cam.ac.uk) or <http://www.ccdc.cam.ac.uk>).

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