

Detection of aryllead(IV) carboxylates and their solvent adducts by ESI-mass spectrometry

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Abstract

The successful detection for the first time of some aryllead(IV) carboxylates by electrospray ionisation mass spectrometry (ESI-MS) is reported; in the presence of excess carboxylate ligand, these compounds gave a reliable $[M + Na]^+$ signal. These conditions appear to be generally suitable for the observation of organolead derivatives, and may find wider application for the reliable detection of other heavy main group and transition organometallic compounds. Furthermore, under these conditions, direct evidence for the formation of solvent (MeCN) and pyridine adducts for several different aryllead(IV) carboxylates was obtained, confirming the existence of facile ligand equilibration in lead(IV) carboxylates. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Aryllead; ESI; Mass spectrometry; Carboxylates

1. Introduction

The use of lead tetraacetate for oxidative processes is widely established [1–5]. More recently, the arylation of nucleophiles with aryllead triacetates has been extensively developed, and these reagents provide an important source of aryl cation synthetic equivalents [6]. We have for some time been interested in the further development of lead(IV) reagents in organic synthesis, and since the effect of ligands on the reactivity of diverse metal complexes is well known, have recently concentrated our efforts on studying the effect of ligands on the structure and reactivity of lead(IV) compounds. We have reported the X-ray crystal structure for tetrakis(*o*-benzoylbenzoato-*O,O'*)lead(IV) and shown that this complex is monomeric with bidentate carboxylate ligands and a trigonal dodecahedron lead(IV) coordination sphere [7]. We have also recently shown that *o*-methoxyphenyllead(IV) acetates are monomeric with bidentate chelating acetate ligands, and that internal coordination of the methoxy oxygen gives an eight coordinate lead atom; clearly, the interaction of this

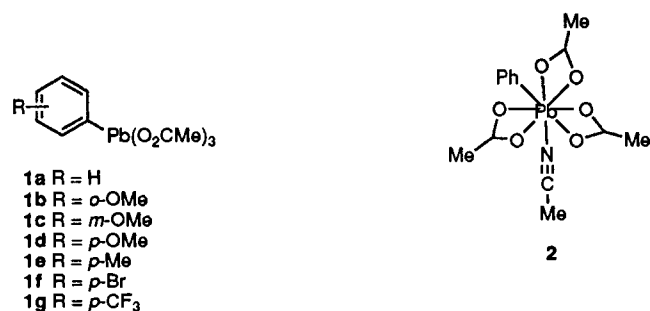
lone pair donor achieves coordination saturation for the metal cation. Such bidentate chelating behaviour is common for organolead(IV) carboxylate species, but lead(IV) complexes are also often oligomeric with bridging carboxylates [8]. The existence of rapid ligand equilibration in lead(IV) systems in solution has been shown by ^1H - and ^{207}Pb -NMR techniques [9–11].

The fact that the ligand can exert significant electronic and steric effects on the reactivity of this class of compound is evident from the variation in reactivity of lead(IV) tetracarboxylates (e.g. acetate, benzoate, trifluoroacetate) [12]. More recently the effect of chiral ligands in arylation [13–17] and in oxidation [18] reactions has been examined. Chiral induction of the arylation reactions was low (e.e. not greater than 16%), and ^{207}Pb -NMR spectroscopic investigations of these systems suggested that a facile ligand exchange was in operation, with the less hindered (and achiral) intermediates of higher reactivity being responsible for the observed poor enantioselectivity [17]. We have shown, however, that modification of the reactivity by ligands can be useful synthetically, and mild conditions for the selective oxidation with lead(IV) carboxylates of alcohols to aldehydes have been established [19]. The importance of σ -donor ligands such as pyridine for

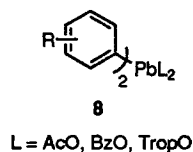
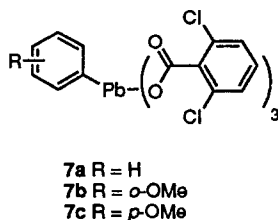
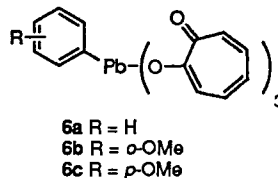
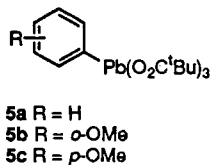
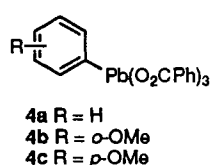
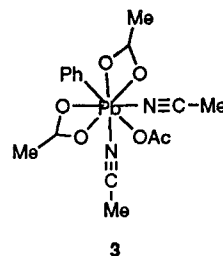
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optimal lead(IV) reactivity is well known [1–5] and internal σ -ligands can catalyse disproportionation reactions [20].



lead(IV) compounds. We report here that application of electrospray ionisation techniques¹ were found to be suitable for the reliable generation of the [M + Na]⁺



The structural characterisation of lead(IV) compounds by X-ray analysis is generally problematic due to their tendency to exist as amorphous solids, and although we have had considerable success with ²⁰⁷Pb-NMR spectroscopy to probe the solution and solid phase coordination phenomena of lead(IV) as noted above [9–11] we recently turned our attention to mass spectrometry both as an analytical tool and as a means of obtaining additional information about the nature of organolead species in solution. However, we found that a range of mass spectrometry techniques such as electron impact and chemical and FAB ionisation techniques were not suitable for organolead(IV) carboxylates. The application of electrospray ionisation mass spectrometry (ESIMS) for the detection of trimethyllead and triethyllead compounds has been reported [21] and significantly, their fragmentation patterns were found to be dependent on the applied cone voltage; this parameter reflects the additional activation occurring in the intermediate pressure region of the spectrometer, and the higher the cone voltage, the more energetic are the collisions and therefore more fragmentation is observed. The application of these conditions therefore offered considerable potential for organo-

and sometimes the [M + K]⁺ and [M – O₂CR]⁺ signals for various aryllead tricarboxylates, provided that a suitable counter anion carboxylate was added;² furthermore, this approach provided evidence for the existence of discrete solvated lead(IV) species. However, although lead(IV) tetracarboxylates were not detectable under these conditions, lead(II) dicarboxylates were observable. To our knowledge, this is the first mass spectrometric observation of these labile systems, which has permitted not only molecular mass determinations but also clearly demonstrated significant solvation phenomena for lead(IV) species.

¹ Electrospray ionisation mass spectra were recorded in a Micro-mass BioIQ triple quadrupole atmospheric pressure mass spectrometer equipped with an electrospray interface. Samples were introduced into the electrospray source via a Harvard model II syringe pump at a flow rate of 5 $\mu\text{l min}^{-1}$ as a solution with a final concentration of typically 20 $\text{pmol } \mu\text{l}^{-1}$ in acetonitrile containing the appropriate free acid or ammonium salt, at a series of cone voltages (10–70 V) and a source temperature of 40 °C. Mass spectra typically consisted of 20 10-s scans over the range 200–1000 Da. The instrument was calibrated with PEG. Peaks containing lead species could be readily identified by their characteristic isotopic distribution patterns.

² The presence of [M + Na]⁺ and [M + K]⁺ signals is due to the ubiquitous presence of alkali metal cations in the solvents, which have been stored in soda glass bottles.

Table 1
Selected ESI-MS data for aryllead triacetates **1a–g**

Conditions	[M+Na] ⁺ Relative intensity ^a						
	1a , R' = H	1b , R' = <i>o</i> -OMe	1c , R' = <i>m</i> -OMe	1d , R' = <i>p</i> -OMe	1e , R' = <i>p</i> -Me	1f , R' = <i>p</i> -Br	1g , R' = <i>p</i> -CF ₃
MeCN	–	38(20)	35(20)	–	0(20)	0(20)	0(20)
	–	100(40)	100(40)	–	0(40)	0(40)	0(30)
MeCN+AcOH (0.2%)	100(20)	69(20)	70(20)	100(20)	100(20)	29(20)	29(20)
	100(30)	100(30)	100(30)	100(30)	100(30)	58(30)	97(30)
MeCN+NH ₄ OAc	100(20)	100(20)	100(20)	22(20)	100(20)	38(20)	15(20)
	100(40)	100(40)	100(40)	–	100(40)	59(40)	74(40)

^a Numbers in brackets refer to cone voltages.

2. Aryllead(IV) triacetates

We found that phenyllead triacetate (**1a**) (20 pmol μl⁻¹) in acetonitrile containing saturated NH₄OAc gave no observable [M⁺] or [M+NH₄]⁺ signals, but reliably gave an intense [M+Na]⁺ signal over a range of cone voltages (15–50 V), which declined at higher voltages (60–70 V). Further investigations (Table 1) demonstrated that similar conditions were suitable for the detection of the [M+Na]⁺ signal for a variety of aromatic substituents **1b–g** (R = *o*-OMe, *m*-OMe, *p*-OMe, *p*-Me, *p*-Br, *p*-CF₃).³ Thus, the application of MeCN as solvent to which 0.1–0.2% HOAc or saturated NH₄OAc has been added, typically gave an intense [M+Na]⁺ signal, commonly known as the base peak; without the addition of acetate ligand, unreliable results were generally obtained. For example, the observed and calculated [M+K]⁺, [M+Na]⁺ and [M–OAc]⁺ peaks for *o*-methoxyphenyllead triacetate (**1b**) are shown in Fig. 1a–d. Interestingly, under these ESI conditions, signals corresponding to [M+Na+2MeCN]⁺ and [M+Na+MeCN]⁺ were also often observable, although the monosolvated peaks were invariably of low (<15%) intensity. In the case of aryllead triacetates **1c** and **g**, the signals for [M+Na+2MeCN]⁺ were particularly intense (70% at 20 V in MeCN and 100% at 20 V in MeCN–0.2% HOAc, respectively). These solvated peaks diminished at higher cone voltages, demonstrating their labile character. Additionally, [M–OAc]⁺ peaks were frequently observed, although unreliably and with variable intensity, but never exceeding 40% of the base peak intensity. It would seem that the added acetate ligand is important for the maintenance of coordination saturation of the organolead(IV) species in solution, and confirms earlier evidence for facile ligand equilibration

³ Aryllead triacetates were prepared by standard literature methods [28]. Phenyllead tricarboxylates were prepared from phenyllead triacetate by ligand exchange using a modification of a literature procedure [29]. All compounds showed spectroscopic data (¹H-, ¹³C- and ²⁰⁷Pb-NMR and IR) consistent with their structure.

based on NMR spectroscopic investigations [9–11]. We assume that the solvated species are of structure **2** or **3**, in which one or two acetonitrile molecules take up coordination sites which would otherwise be occupied by bidentate acetate ligands.

Aryllead triacetates **1b** and **d** in the presence of one equivalent of pyridine and 10 mM NH₄OAc or 0.2% HOAc, respectively, were also found to give an intense lead cluster centred at 512, corresponding to [M–OAc+pyridine]⁺, in addition to the expected [M+Na]⁺ cluster at 515 (Fig. 2). This provides direct mass spectroscopic evidence for the formation of organolead(IV)–pyridine adducts in solution, species postulated in many reactions involving lead(IV) but never previously detected or isolated [6,22]. Further evidence for the formation of a σ-complex has been established from a recent NMR spectroscopic study [23] and complements earlier work which clearly indicates the lability of many lead(IV) complexes, especially in

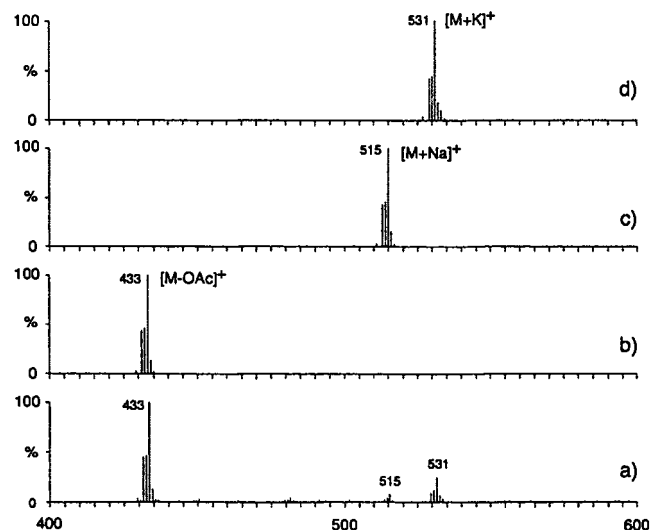


Fig. 1. (a) Observed spectrum of *o*-MeOC₆H₄Pb(OAc)₃ at CV = 20 V in MeCN/10 mM NH₄OAc. (b) Calculated isotropic distribution for [M–OAc]⁺. (c) Calculated isotropic distribution for [M+Na]⁺. (d) Calculated isotropic distribution for [M+K]⁺.

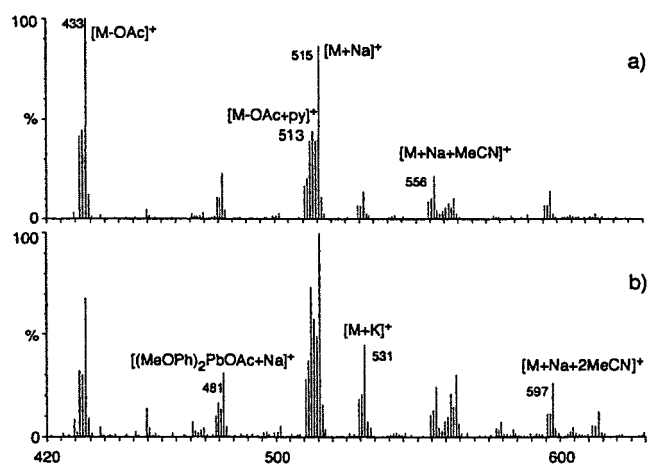


Fig. 2. (a) Observed spectrum of *o*-MeOC₆H₄Pb(OAc)₃ at CV = 20 V in MeCN/10 mM NH₄OAc containing pyridine (one equivalent). (b) Observed spectrum of *p*-MeOC₆H₄Pb(OAc)₃ at CV = 20 V in MeCN/10 mM NH₄OAc containing pyridine (one equivalent).

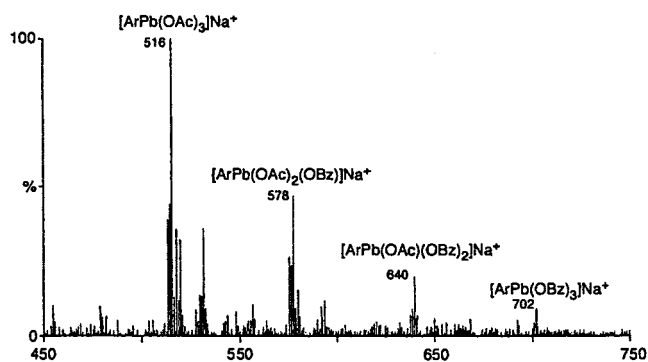


Fig. 3. Observed spectrum of *p*-MeOC₆H₄Pb(OCOPh)₃ at CV = 20 V in MeCN/0.2% HOAc.

solution [9–11,20]. The importance of pyridine for the mediation of the reactivity of lead(IV) compounds is well known; in arylation reactions, for example, it is reported to be essential for high-yielding aryl transfer reactions leading to the formation of carbon–carbon bonds [6]. The existence of additional solvent chelation in **1b** and **d** under these same conditions was again indicated by the presence of $[M + Na + MeCN]^+$ and $[M + Na + 2MeCN]^+$ clusters.

3. Aryllead(IV) tricarboxylates

Since much of our earlier work has been concerned with the effect of ligands on the reactivity of organolead(IV), of interest to us was the behaviour of other aryllead tricarboxylates in acetonitrile with added carboxylate ligand. When the spectrum of *p*-MeOC₆H₄Pb(O₂CPh)₃ was recorded in MeCN with added acetic acid (0.2%) at a cone voltage of 30 V, signals at 702 (25%), 640 (45%), 578 (80%) and 516

(100%) corresponding to the expected $[M + Na]^+$, along with the $[M + Na]^+$ peaks for *p*-MeOC₆H₄-Pb(OAc)(O₂CPh)₂, *p*-MeOC₆H₄Pb(OAc)₂(O₂CPh), and *p*-MeOC₆H₄Pb(OAc)₃ (Fig. 3) were observed. These results clearly demonstrated that facile acetate–benzoate ligand exchange occurs in solution. Although the existence of ligand exchange has been demonstrated earlier using isotope exchange reactions [24] and later with NMR techniques [9] this is the first *direct* observation of mixed ligand species in solution. The synthetic importance of ligand exchange has been discussed above, and suggested to be especially important in the context of asymmetric arylation reactions, where it appears to be responsible for poor levels of asymmetric induction; low e.e. values are thought to be due to facile ligand exchange which permits loss of chiral ligands and fast reaction via less-hindered organometallic species [17].

Given the ease of observation in this series of $[M + Na]^+$ signals, it was of interest to determine if the desired $[M + Na]^+$ peak for *any* given aryllead(IV) carboxylate may be observable in the presence of added carboxylate ligand. Spectra were therefore acquired for the aryllead(IV) tribenzoates **4a–c** in the presence of benzoic acid or ammonium benzoate, in equimolar concentrations with the aryllead compound (Table 2). Under these conditions, $[M + Na]^+$ were easily detected in all cases, and for the aryllead tribenzoates **4b** and **c**, significant $[M - O_2CR]^+$ signals were also observed. This protocol for the detection of lead(IV) species, in which the analyte solution was doped with the respective carboxylate, proved to be general, and applicable to a range of aryllead tripivaloates **5a–c**, tropolonates **6a–c**, and dichlorobenzoates **7a–c** (Table 2). Of interest was the observation that the observed stability of each of the phenyllead derivatives **4–7** increased qualitatively in the same order as the ease of detection of the $[M + Na]^+$ signal; thus, increasingly stable complexes are achieved with ligands in the order benzoate, pivaloate and tropolonate. This correlates with the solution reactivity of aryllead compounds, since the tropolonates have been found to be unreactive in Pinhey arylation [25]. This approach may find much wider application for the analysis and structural investigation of lead and other heavy metal compounds which exhibit facile ligand exchange phenomena in solution.

Noteworthy is the observation under these mass spectrometric conditions of a facile disproportionation of *o*-methoxyphenyllead tribenzoate (**4b**) to bis(*o*-methoxyphenyl)lead dibenzoate (**8**) (R' = *o*-OMe, L = O₂CPh), as shown by a strong $[Ar_2Pb(O_2CR)Na]^+$ signal in the mass spectrum of the former compound; this reaction has recently been investigated in detail by NMR spectroscopy, and appears to be due to the capacity of the *o*-OMe group to coordinate to the lead(IV) atom [20]. A similar disproportionation, which was particularly facile under basic conditions, was also

Table 2
Selected ESI-MS for aryllead tricarboxylates **4a–c**, **5a–c**, **6a–c**, and **7a–c**

L ^a	Conditions	Relative intensity ^b					
		a, R' = H		b, R' = <i>o</i> -OMe		c, R' = <i>p</i> -OMe	
		[M + Na] ⁺	[M – L] ⁺	[M + Na] ⁺	[M – L] ⁺	[M + Na] ⁺	[M – L] ⁺
4 ·BzO	MeCN	21(30)	–	93(25) ^c	20(25) ^c	100(20)	20(20)
		13(40)	–	100(35) ^c	84(35) ^c	100(30)	15(30)
	MeCN + PhCO ₂ H	100(30)	–	100(35) ^c	24(35) ^c	100(30)	5(30)
		13(40)	–	82(40) ^c	100(40) ^c	100(40)	5(40)
MeCN + PhCO ₂ NH ₄	100(25)	43(25)	100(35) ^c	21(35) ^c	100(25)	43(25)	
	72(45)	54(45)	33(45) ^c	100(40) ^c	100(35)	44(35)	
5 ·PivO	MeCN	58(5)	–	0(5)	–	100(20)	2(20)
		100(30)	–	3(20)	–	100(30)	2(30)
	MeCN + Me ₃ CCO ₂ H	47(5)	–	11(25)	–	100(25)	5(25)
		100(30)	–	8(35)	–	100(45)	5(45)
6 ·TropO	MeCN	65(10)	2(10)	100(10)	10(10)	100(10)	0(10)
		16(30)	2(30)	100(30)	95(30)	54(30)	100(30)
	MeCN + TropOH	100(10)	0(10)	100(10)	5(10)	100(10)	0(10)
		19(30)	15(30)	100(30)	95(30)	48(30)	70(30)
7 ·DCBzO	MeCN	0(20)	25(20)	20(30)	0(30)	20(30)	0(30)
		–	–	5(50)	0(50)	10(50)	0(50)
	MeCN + DCBzOH	–	–	30(30)	0(30)	–	–
		–	–	10(50)	0(50)	–	–

^a BzO = PhCO₂, PivO = Me₃CCO₂, TropO = C₇H₅O₂, DCBzO = 2,6-Cl₂C₆H₃CO₂.

^b Numbers in brackets refer to cone voltages.

^c Here M = Ar₂Pb(O₂CPh)₂.

observed for *o*-methoxyphenyllead triacetate, phenyllead tris(dichlorobenzoate) and *o*-methoxyphenyllead tris(dichlorobenzoate), all of which gave strong [Ar₂Pb(O₂CR)Na]⁺ signals (Figs. 4–6).

The value of this ionisation protocol was further demonstrated by its application to bisdiaryllead dicarboxylates **8**, prepared independently by literature methods; [20] thus, bis(*o*-methoxyphenyl)lead diacetate, and bis(*p*-methoxyphenyl)lead diacetate, dibenzoate, and ditropolonate all gave significant [M + Na]⁺ and [M – L]⁺ peaks (Table 3). For *p*-methoxyphenyllead derivatives **8**, the intensity of the [M + Na]⁺ signals was not

so dependent on the nature of the carboxylate ligand nor upon the conditions under which the mass spectrum was run, and this probably reflects the higher intrinsic stability of diaryllead compounds **8** over monoarylleads of type **4**.

4. Lead(II) diacetates

Under these ESIMS conditions, even spectra of lead(II) carboxylates could be obtained. Thus, lead diacetate gave intense [M + Na + 4MeCN]⁺, [M +

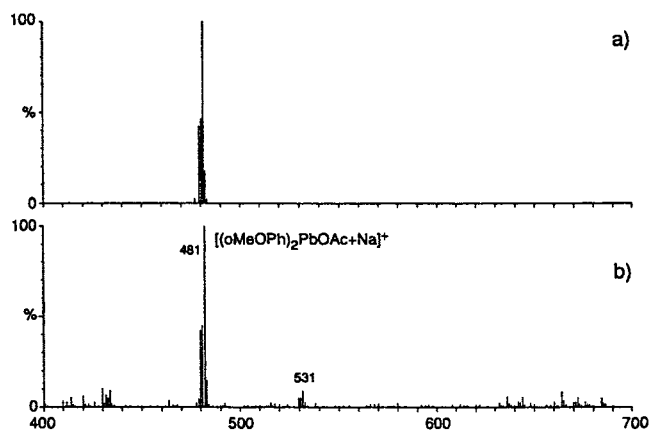


Fig. 4. (a) Calculated isotropic distribution of [M – OAc + Na]⁺ for *o*-MeOC₆H₄Pb(OAc)₃. (b) Observed spectrum of *o*-MeOC₆H₄Pb(OAc)₃ at CV = 20 V in MeCN/5 pmol⁻¹ NaHCO₃.

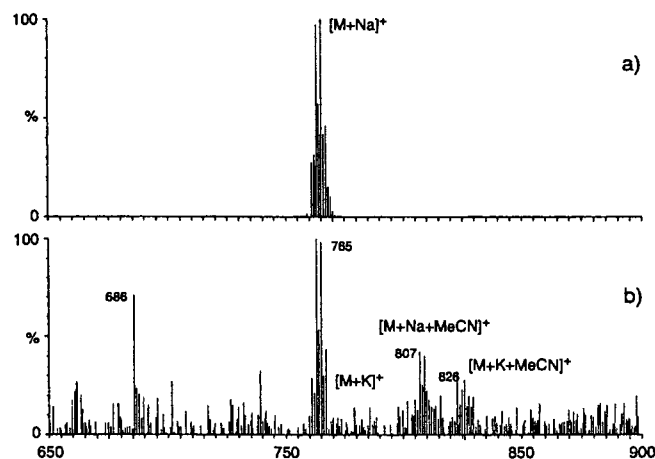


Fig. 5. (a) Calculated isotropic distribution for [Ph₂Pb(DCBzO)Na]⁺. (b) Observed spectrum for Ph₂Pb(DCBzO)₃ in MeCN.

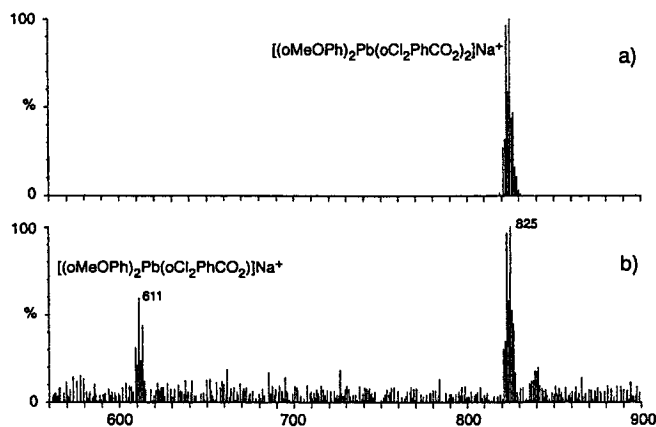


Fig. 6. (a) Calculated isotropic distribution for $[(o\text{-MeOC}_6\text{H}_4)_2\text{Pb}(\text{DCbZO})_2\text{Na}]^+$ in MeCN. (b) Observed spectrum of $o\text{-MeOC}_6\text{H}_4\text{Pb}(\text{DCbZO})_3$ in MeCN.

$[\text{Na} + 2\text{MeCN}]^+$ and a weak $[\text{M}]^+$ signal at 15–30 V in MeCN. Addition of 0.2% HOAc to the solvent all but eliminated the 4MeCN adduct, and significantly enhanced the $[\text{M} + \text{Na} + 2\text{MeCN}]^+$ and $[\text{M}]^+$ signals. The need for additional aqueous ligands in crystalline lead(II) acetate to achieve coordination saturation has already been demonstrated [26]. Lead dibenzoate in acetonitrile at 30 V also gave moderately intense $[\text{M} + \text{Na} + 4\text{MeCN}]^+$ and weak $[\text{M}]^+$ signals. Noteworthy, however, is that lead(IV) tetraacetate did not give $[\text{M} + \text{Na}]^+$ signals under these conditions.

5. Conclusions

$[\text{M} + \text{Na}]^+$ and $[\text{M} - \text{L}]^+$ signals of aryllead tricarbonylates are observable by ESIMS using cone voltages of up to 50 V provided the sample is doped with an equimolar solution ($20 \text{ pmol } \mu\text{l}^{-1}$) of the corresponding carboxylic acid or a soluble salt thereof. This technique has also been successfully applied to lead(II) carboxylates but not their lead(IV) counterparts. Application of this strategy may permit the analysis by ESIMS of a wide range of metal carboxylates and other species, particularly for heavy metal cations, with exchangeable ligands. Significantly, the method has allowed the detection of transient solvated organolead(IV) species, and further demonstrates the importance of coordination phenomena for lead(IV) species in solution. We expect this data to have a key role in the design of new ligand systems for lead(IV) which permits the development of novel reaction sequences, and will report our work in this area in due course.

Acknowledgements

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Table 3
Selected ESI-MS for diaryllead compounds **8**

L	Conditions	Relative intensity ^a			
		R' = <i>o</i> -OMe		R' = <i>p</i> -OMe	
		$[\text{M} + \text{Na}]^+$	$[\text{M} - \text{L}]^+$	$[\text{M} + \text{Na}]^+$	$[\text{M} - \text{L}]^+$
AcO	MeCN	25(20) 5(40)	100(20) 100(40)	100(20) 25(40)	65(20) 100(40)
	MeCN + AcOH	100(20)	90(20)	75(30) 40(40)	90(30) 90(40)
BzO ^b	MeCN	–	–	100(40) 40(50)	100(40) 40(50)
	MeCN + PhCO ₂ H	–	–	83(40)	100(40)
	MeCN + PhCO ₂ NH ₄	–	–	31(50) 100(40)	100(50) 65(40)
	–	–	–	86(50)	100(50)
TropO ^b	MeCN	–	–	100(10) 61(30)	10(10) 100(30)
	MeCN + TropOH	–	–	100(20)	10(20)
	–	–	–	53(30)	55(30)

^a Numbers in brackets refer to cone voltages.

^b BzO = PhCO₂, TropO = C₇H₅O₂.

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