Journal of Organometallic Chemistry
Elsevier Sequoia S.A., Lausanne, – Printed in The Netherlands

Preliminary communication

Aromatic lead(IV) compounds
II. PMR spectroscopy of some aryllead tricarboxylates

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(Received April 17th, 1972)

In a preceding paper[1] we reported the preparation of some aryllead triacetates. The PMR spectra of these compounds were found to be of unusual interest.

With the element lead only the isotope ^{207}Pb (natural abundance 21.6%[2*]) has a nuclear moment of ½. For aryllead tricarboxylates this means that the phenyl protons in the PMR spectra show signals in the region of ~ 7 ppm due to the species containing non-magnetic lead isotopes, and well separated signals due to the species containing the magnetic ^{207}Pb. The PMR spectra of aryllead-207 carboxylates can be considered as first order for the compounds studied, since the coupling of ^{207}Pb with the protons of the phenyl ring is large.

Tables 1, 2 and 3 show details of the spectra of 2,4,6-trimethoxy-, 2,4-dimethoxy-, and *p*-methoxy-phenyllead triacetate, respectively. In Table 4 the ^{207}Pb–H coupling constants for some *para* substituted aryllead triacetates are given[**].

TABLE 1

δ(ppm)	*Ratio peak areas*	*Protons*	
2.09 s	9	$-OOCH_3$	
3.81 s	3	$4\text{-}OCH_3$	
3.86 s	6	$2,6\text{-}OCH_3$	
6.17 s	2	$H_{3,5}$	$J(^{207}Pb\text{-}H_{3,5})$ 166 Hz

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[*] We confirmed this percentage from the peak areas of the phenyl protons of magnetic and non magnetic species.

[**] The coupling constants for phenyllead triacetate, prepared by the method of Criegee[4], are: $J(^{207}Pb\text{-}H_{ortho})$ 361, $J(^{207}Pb\text{-}H_{meta})$ 165 and $J(^{207}Pb\text{-}H_{para})$ 71 Hz.

TABLE 2

δ(ppm)	*Ratio peak areas*	*Protons*	
2.11 s	9	$-OOCCH_3$	
3.83 s	3	$4-OCH_3$	
3.89 s	3	$2-OCH_3$	
6.53 d	1	H_3	$J(H_3-H_5)$ 2.5 Hz
6.63 q	1	H_5	
7.61 d	1	H_6	$J(H_5-H_6)$ 9 Hz

TABLE 3

δ(ppm)	*Ratio peak areas*	*Protons*	
2.15 s	9	$-OOCCH_3$	
3.83 s	3	$4-OCH_3$	
7.02 d	2	$H_{3,5}$	
7.53 d	2	$H_{2,6}$	$J(H_2-H_3)$ 9 Hz

TABLE 4

Compound	*$J(^{207}Pb-H_{ortho})$ (Hz)*	*$J(^{207}Pb-H_{meta})$ (Hz)*
p-Methoxyphenyllead triacetate	327	121
p-Ethoxyphenyllead triacetate	334	125
p-n-Butoxyphenyllead triacetate	330	129
p-Phenoxyphenyllead triacetate	332	125

We studied also the influence of the carboxylate group in some 2,4-dimethoxyphenyllead tricarboxylates on $J(^{207}Pb-H)$. The results are given in Table 5. It appears that there is a positive charge on the lead atom, the magnitude of which depends on the electron-withdrawing capacity of the carboxylate group.

TABLE 5

Compound	*$J(^{207}Pb-H_3)$ (Hz)*	*$J(^{207}Pb-H_5)$ (Hz)*	*$J(^{207}Pb-H_6)$ (Hz)*
2,4-Dimethoxyphenyllead tripropionate[a]	185	96	383
2,4-Dimethoxyphenyllead triacetate	187	99	386
2,4-Dimethoxyphenyllead tris(monochloroacetate)[a]	212	113	408

[a] The synthesis of this compound will be published.

More detailed studies of the spectroscopic characteristics of aryllead tricarboxylates are in hand at this laboratory[3].

The spectra were recorded on a Jeol PS 100 NMR apparatus (in $CDCl_3$, reference TMS) by Mr. C. Erkelens.

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