

Journal of Organometallic Chemistry, 153 (1978) C23—C24
 © Elsevier Sequoia S.A., Lausanne — Printed in The Netherlands

Preliminary communication

A METHOD FOR PREPARING RPbI_3 COMPOUNDS

GERALD CHOBERT et MARGUERITE DEVAUD
 INSCIR, B.P. 08, 76130 Mont Saint Aignan (France)
 (Received March 31st, 1978)

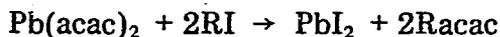
Summary

The reaction between an alkyl iodide and lead(II) iodide catalysed by trimethylstibine gives an alkyltriiodolead compound. The RPbI_3 products ($\text{R} = \text{Et}$; $n\text{-Pr}$; $i\text{-Pr}$; $n\text{-Bu}$; $t\text{-Bu}$) are yellow-orange crystalline solids with low melting-points. The carbon—lead bond is stable towards water, alcohols, acids and bases.

The one monoalkylated derivative of lead(IV) previously described was prepared by direct plumbylation of pregnenolone [1].

Two methods of synthesis were proposed some time ago [2, 3]; we have not reinvestigated the reaction of alkyl halide with sodium plumbate(II) [2] since it appears to be unsatisfactory, being more difficult than the corresponding reaction with sodium stannate(II) [4]. We have previously observed that iodides RI react to a slight extent with caesium trichloroplumbate(II) [3], and traces of ethyltriiodolead were identified during experiments under pressure at 170°C .

Alkylation of lead tetraacetate by an organomagnesium derivative always gives disubstituted organolead compounds as found also with tin(IV) salts, and we have confirmed the observation of Criegee et al. [5], that lead tetraacetate is not alkylated by dialkylmercury. Bis(2,4-pentane dionato)lead(II) [6] reacts with alkyl iodides, but in contrast with its tin parent [7], undergoes only the exchange reaction 1.



(acac = 2,4-pentane dionate)

1

Trimethylstibine is known to be a good catalyst for the reaction between a tin(II) halide and alkyl halide [8], and we found that it is also effective with lead iodide (eq. 2).



2

To avoid halide exchange we used only iodides. The reaction is carried out at ca. 140°C in a closed reactor with an excess of alkyl iodide. (Below 100°C no

TABLE 1

PRODUCTS ISOLATED FROM THE REACTION FOR 48 h BETWEEN PbI_2 AND RI AT $140^\circ C$, CATALYSED BY $(CH_3)_3Sb$. NMR SPECTRA: SOLVENT $CDCl_3$, REFERENCE TMS, t, TRIPLET; q, QUARTET.

R	NMR (ppm)		
	CH_3	$(CH_2)_n$	CH_2-Pb
C_2H_5	1.7 t		2.65 q
$n-C_3H_7$	1.05 t	1.95	2.80
$n-C_4H_9$	0.9 t	mixed	3.05

reaction occurs, while above $160^\circ C$ the organolead compound is decomposed). After reaction, the product was extracted with ethyl acetate, and evaporation of the solvent left an oil which crystallised more or less rapidly. Sublimation (in some cases in the presence of phosphoric anhydride) gave yellow-orange crystals of alkyltriiodolead which melt. The products, all of which melt between 30 and $50^\circ C$ are listed in Table 1. All compounds showed the characteristic infrared vibrations of the R group. Satisfactory elemental analyses were obtained in all cases.

We were unable to isolate the methyl compound; but methyl iodide should be the most reactive halide, as its reactions with organotin compounds show, and the failure is probably due to the low solubility of lead salt in methyl iodide. *t*-Butyl iodide gives considerable iodine because of decomposition of the organic compound, after removal of iodine with acetone, small quantities of *t*-butyltriiodolead were isolated.

The $RPbI_3$ compounds are stable and very hygroscopic. The carbon-lead bond is not broken at room temperature by water, alcohols, acids and bases.

References

- 1 M. Ephritikhine and J. Levisalles, *J. Chem. Soc. Chem. Commun.*, (1974) 429 and *Bull. Soc. Chim. Fr.*, (1975) 339.
- 2 M. Lesbre, *C. R. Acad. Sci. Paris*, 200 (1935) 559; 210 (1940) 535.
- 3 M. Lesbre, *C. R. Acad. Sci. Paris*, 204 (1937) 1822; 206 (1938) 1481.
- 4 M. Devaud and M.C. Madec-Ogez, *J. Organometal. Chem.*, 93 (1975) 85.
- 5 R. Criegee, P. Dimroth and R. Schempf, *Ber.*, 90 (1957) 1337.
- 6 G.K. Schweitzer, B.P. Pullen and Yi-Hung Fang, *Anal. Chim. Acta*, 43 (1968) 332.
- 7 M. Devaud, *J. Organometal. Chem.*, 141 (1977) 371 and ref. therein.
- 8 E.J. Bulten, *J. Organometal. Chem.*, 97 (1975) 167.