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## NOTE

# A NEW METHOD OF PREPARING A SOLUBLE FORM OF TRIMETHYLTIN ACETATE

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The current method of preparing trimethyltin acetate involves titration of an aqueous solution of trimethyltin hydroxide with glacial acetic acid[1]. This gives a form of the acetate which is insoluble in solvents commonly employed in IR spectroscopy[2-4]. Recently Simons and Graham[4] succeeded in preparing a soluble trimethyltin acetate form by heating the insoluble form with cyclohexane in a sealed tube at 100°.

We wish to report a new and simple way of preparing a soluble form of trimethyltin acetate in high yield; this involves interaction of tetramethyltin and mercury(I) acetate in methanol at room temperature.

## EXPERIMENTAL

Tetramethyltin was purchased from Fluka and used without further purification. Methanol and mercury(I) acetate were grade reagents from C. Erba (Milan). Chloroform and Nujol were of spectrograde quality. IR spectra were run on a Perkin–Elmer Model 621 equipped with KBr optics, by using the prepared trimethyltin acetate either dissolved in chloroform or dispersed in Nujol. The melting points were taken with a Büchi apparatus (Switzerland) using sealed capillaries.

## RESULTS

### *Stoichiometry of the reaction of* $(CH_3)_4Sn$ *with* $Hg_2(OOCCH_3)_2$

Several runs were carried out to verify the stoichiometry of the reaction between tetramethyltin and mercury(I) acetate in methanol at room temperature. It was found that the products of the reaction are: trimethyltin acetate, dimethylmercury, and metallic mercury.

Table 1 shows the obtained results for reactions in which the reactant ratio $[(CH_3)_4Sn]/[Hg_2(OOCCH_3)_2]$ was 2. From the data it can be concluded that the stoichiometry of the reaction is as follows:

$$2\,(CH_3)_4Sn + Hg_2(OOCCH_3)_2 \rightarrow 2\,(CH_3)_3SnOOCCH_3 + (CH_3)_2Hg + Hg^0$$

This agrees with that previously found for the analogous reaction between tetramethyltin and mercury(I) nitrate[5].

TABLE 1

DATA ON THE REACTION OF $(CH_3)_4Sn$ AND $Hg_2(OOCCH_3)_2$ IN THE RATIO 2/1 IN METHANOL AT ROOM TEMPERATURE

| Methanol | Reactants | | Products | | |
|---|---|---|---|---|---|
| (ml) | $(CH_3)_4Sn$ (mole·$10^4$) | $Hg_2(OOCCH_3)_2$ (mole·$10^4$) | $(CH_3)_3SnOOCCH_3$ (mole·$10^4$) | $(CH_3)_2Hg^a$ (mole·$10^4$) | $Hg^0$ (mole·$10^4$) |
| 20 | 7.2 | 3.6 | 7.0 | 3.2 | 3.4 |
| 40 | 28.8 | 14.4 | 25.3 | 13.9 | 14.0 |
| 50 | 28.8 | 14.4 | 26.2 | 14.0 | 14.2 |

[a] The quantity of $(CH_3)_2Hg$ has been evaluated by weighing $CH_3HgCl$ produced by cleavage of the dimethylmercury with mercury(II) chloride.

*Preparation of trimethyltin acetate*

To a weighed amount of mercury(I) acetate (7.56 g, 14.5 mmoles) suspended in methanol (200 ml) was added tetramethyltin (5.18 g, 29.0 mmoles). Reaction starts immediately: finely powdered metallic mercury began to precipitate as soon as the reactants are mixed. The mixture was stirred at room temperature for 1 h. The black powdered mercury (recovered 2.85 g, 14.2 mmoles) was removed by filtration. The alcoholic filtrate was distilled to dryness leaving a white solid residue (6.10 g), m.p. 183° (in a sealed capillary). This residue was sublimed under vacuum to give crystalline trimethyltin acetate (5.95 g, 26.7 mmoles) in 92% yield. The product melts sharply at 193° (uncorrected) in a sealed capillary (Found: C, 26.88; H, 5.41. $C_5H_{12}O_2Sn$ calcd.: C, 26.96; H, 5.39%.)

To the collected alcoholic solution, containing dimethylmercury, mercury(II)

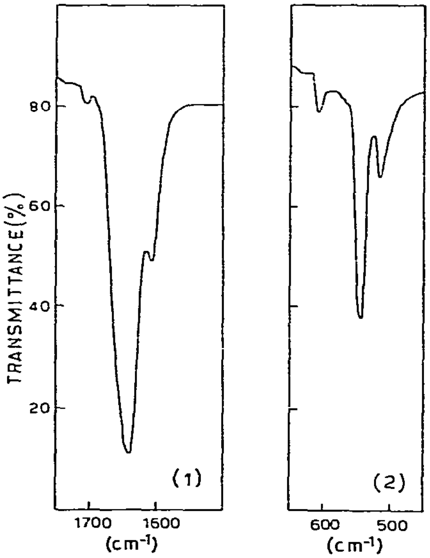

Fig. 1. IR spectra of trimethyltin acetate in chloroform; (1) carbonyl stretching vibration region, (2) $SnC_3$ stretching vibration region.

chloride (5 g) was added. A white crystalline solid was precipitated by adding water, removed by filtration, and weighed; it was shown to be $CH_3HgCl$ (6.8 g, 27.1 mmoles), m.p. 167°.

*Infrared spectra*

The IR spectra of the trimethyltin acetate in Nujol agrees completely with the data previously reported for the soluble form[4]. Particularly, absorptions were observed at 1570 $cm^{-1}$ and 1420 $cm^{-1}$ in the carbonyl vibration region and at 548 $cm^{-1}$ in the trimethyltin vibration region. Fig. 1 shows the IR spectrum of trimethyltin acetate in a chloroform solution in the two regions of interest.

## ACKNOWLEDGEMENT

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