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# 24 Hour Addiction Helpline is Available



# Preparation of Lead(IV)Acetate, Pb(OAc)4

by Cherrie Baby

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#### **Procedure**

Pb3O4 + 8 CH3COOH ==> Pb(CH3COO)4 + 2 Pb(CH3COO)2 (\*3 H2O) + 4 H2O 4 H2O + 4 (CH3CO)2O -> 8 CH3COOH 2 Pb(CH3COO)2 (\*3 H2O) + Cl2 -> Pb(CH3COO)4 + PbCl2

The reaction of Pb3O4 with CH3COOH is carried out in a one-litre, three-neck flask fitted with a sealed stirrer and a thermometer; the third opening may be closed (not too tightly). The required amount of pure Pb3O4 is finely pulverised in advance, dried at 200°C and left in a desiccator over P2O5 until needed. The flask is charged with 550 ml. of glacial acetic acid and 170 ml. of pure acetic anhydride; the mixture is heated to 40°C, and then 300 g. of Pb3O4 is slowly added with vigorous stirring without further external heating. During this procedure, the loosely fitting stopper is removed, but only for brief periods. The reaction is exothermic and the rate at which the Pb3O4 is introduced is regulated so that the temperature in the flask remains under 65°C. Otherwise, the freshly formed lead tetraacetate is partially reduced by the acetic anhydride. Running water may also be used to prevent overheating of the flask. After most of the Pb3O4 has been added, the temperature gradually falls, and toward the end of the reaction it may be necessary to heat the flask (but never over 65°C).

The clear solution is left to cool protected from atmospheric moisture, and large quantities of colorless Pb(CH3COO)4 precipitate out. The supernatant liquid is decanted, and the precipitate is poured into a large Buchner funnel. The funnel is covered with a cardboard square or a tile to minimize the effects of atmospheric moisture during the very slow suction filtration. The filter cake is washed several times with glacial acetic acid and dried on a clay tile in a desiccator. The resulting product, usually tinted rose or brownish because of the presence of small amounts of PbO2, can be further purified by recrystallization from hot glacial acetic acid. Even after prolonged desiccation a small amount of glacial acetic acid will be retained by the salt. The yield is about 150 g.

Another, less pure quantity of the salt may be recovered from the mother liquor, which is treated in the original reaction flask with dry CI2 at 80°C until no further PbCI2 precipitates. Good stirring must be used. The precipitate is filtered hot and washed with glacial acetic acid, and the solution is left to crystallize. During cooling, about 100 g. of Pb(CH3COO)4 crystallizes out. However, it is contaminated with PbCI2. Pure lead tetraacetate can be obtained by repeated further recrystallization from glacial acetic acid.

The procedure may be varied by omitting the acetic anhydride (Dimroth and Schweizer). In this case, however, the maximum temperature must be held below 60°C since the water formed in the reaction is not bound and may hydrolyze

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the Pb(CH3COO)4 at higher temperatures.

Lead (IV) acetate may be stored only if absolutely dry and when kept in well-closed ground glass bottles. It is used as a selective oxidant in organic syntheses. Lead tetraacetate can also be made through electrolysis [5-8]

## **Synonyms**

Lead(IV)acetate, Lead tetraacetate, plumbic acetate, Pb(OAc)4.

## **Properties**

Colorless prismatic crystals, very sensitive to moisture. In the presence of moisture, decomposes hydrolytically to form brown PbO2. M.p. 175-180°C (some decomposition); d (17X°C) 2.23. Hydrolyzed by water, forming PbO2 and acetic acid. Dissolves in hot acetic acid without decomposition; slightly soluble in dry CHCl3, CCl4 and C6H6.

## References

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- [3] H. S. Booth, Inorg. Syn., Vol. I, New York-London 1939, p. 47.
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- [7] Oxidations in Org. Chem. part A, Wiberg, 1965, p277-366 Academic Press.
- [8] Pure Appl. Chem. 13, 567-81 (1966)

Important Message for Those With a Substance Abuse Problem: If you or someone you know is suffering from an addiction they need to find a <u>Drug Rehab</u> or <u>Alcohol Rehab</u> so they can recover from their substance abuse addiction. Relapse prevention is important in the <u>addiction treatment</u> process and is critical for long term sobreity. For more information on how to get help with an addiction please call 800-559-9503 to speak directly with a counselor.

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