

## COMMENTS

### Explosion during the Preparation of Ethylphenyl Thallic Acetate Perchlorate

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We have reported a method of preparing arylthallic acetate perchlorate monohydrates<sup>1)</sup> ( $\text{RC}_6\text{H}_4\text{TlOAc}\cdot\text{ClO}_4\cdot\text{H}_2\text{O}$ ), with the syntheses of phenyl, tolyl, three isomeric xylyl, and anisyl derivatives. On applying this method to the preparation of ethylphenyl derivative, a violent explosion occurred in the last stage of concentration of the reaction mixture carried out with a rotary vacuum evaporator. The procedure was as follows. Into acetic acid (180 ml) containing  $\text{Tl}(\text{OAc})_3$  (45 g), 70%  $\text{HClO}_4$  (18 g), and then ethylbenzene (25 g) were added at 65°C under stirring. After 5 hr the precipitated thallium(I) salts (*ca.* 5 g) were filtered off and the solvent in the filtrate was evaporated slowly at *ca.* 60°C in a vacuum with a rotary

vacuum evaporator to give *ca.* 30 ml of white pasty residue. Soon after evaporation was stopped, a violent explosion occurred.

Before this experiment, two runs of the same reaction were carried out safely to give the expected product in 14.7% yield and a trace amount. About 150 runs of preparative experiments were carried out without any trouble. However, care should be taken during the process of evaporating the solvent and too much evaporation should be avoided.

We would like to add the following remark with respect to the handling of perchloric acid. When one drop of 70% perchloric acid was added to 10 ml of dimethylsulfoxide (DMSO) at room temperature, a violent explosion occurred. Reactions in DMSO with perchloric acid should be avoided.

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1) K. Ichikawa, S. Uemura, E. Uegaki, and T. Nakano, *This Bulletin*, **44**, 545 (1971).