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Explosion hazard

PERCHLORYL FLUORIDE: A FURTHER WARNING

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A recent note [1] about an explosion involving perchloryl fluoride prompts us to report our experience with the same reagent. Following the procedure outlined by Schlosser and Heinz [2], we attempted the preparation of 3-fluorobenzocyclobutene by treating 3-lithiobenzocyclobutene [3] with perchloryl fluoride. The experiment was not successful and a second attempt was made using a slightly different approach.

Schlosser and Hartmann [4] reported successful kaliation of some hydrocarbons with a mixture of n-butyllithium, and potassium t-butoxide. When benzocyclobutene (4.16 g, 0.04 mole) was treated, as described [4], with potassium t-butoxide and n-butyllithium, a dark red precipitate was formed. The reaction mixture was stirred for one hour and cooled to −70 °C with dry ice/acetone. A two-fold excess of perchloryl fluoride was passed in. After stirring for 1 hour, the flask was lifted from the cooling bath to allow it to warm up to room temperature. At this very moment, a violent explosion occurred, causing injury to the worker. Fortunately, the extent of injury was minimised due to safety precautions taken. We are unable to offer an explanation for the apparent detonation of the final reaction mixture.

The desired compound, 3-fluorobenzocyclobutene, has now been synthesized by a more tedious but safer route [5].

References

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