Organic Syntheses

A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

Working with Hazardous Chemicals

The procedures in *Organic Syntheses* are intended for use only by persons with proper training in experimental organic chemistry. All hazardous materials should be handled using the standard procedures for work with chemicals described in references such as "Prudent Practices in the Laboratory" (The National Academies Press, Washington, D.C., 2011; the full text can be accessed free of charge at http://www.nap.edu/catalog.php?record_id=12654). All chemical waste should be disposed of in accordance with local regulations. For general guidelines for the management of chemical waste, see Chapter 8 of Prudent Practices.

In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior to performing a reaction, a thorough risk assessment should be carried out that includes a review of the potential hazards associated with each chemical and experimental operation on the scale that is planned for the procedure. Guidelines for carrying out a risk assessment and for analyzing the hazards associated with chemicals can be found in Chapter 4 of Prudent Practices.

The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries or damages claimed to have resulted from or related in any way to the procedures herein.

These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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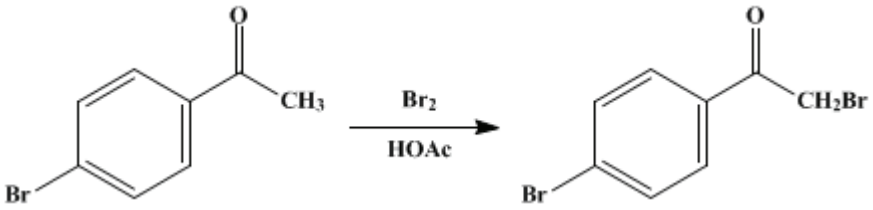
p-BROMOPHENACYL BROMIDE

[Acetophenone, α, *p*-dibromo-]



Submitted by W. D. Langley
Checked by H. T. Clarke and P. W. Boutwell.

1. Procedure

In a 500-cc. flask are placed 50 g. (0.25 mole) of *p*-bromoacetophenone (p. 109) and 100 cc. of glacial acetic acid. To the resulting solution is very slowly added 40 g. (12.5 cc., 0.25 mole) of bromine, keeping the temperature below 20°. The mixture is vigorously shaken by hand during the addition. *p*-Bromophenacyl bromide begins to separate as needles when about one-half of the bromine has been added. The addition requires about thirty minutes.

When all the bromine has been added, the flask is cooled in ice-water and the product filtered with suction. The crude crystals are washed with 50 per cent ethyl alcohol until colorless (about 100 cc. is required). The material so obtained, when air-dried, melts at 106–108° and weighs 55–60 g. It is recrystallized from 400 cc. of 95 per cent ethyl alcohol, from which it separates as colorless needles melting at 108–109°. The yield is 48–50 g. (69–72 per cent of the theoretical amount) (Note 1).

2. Notes

1. A further quantity of less pure material may be obtained from the mother liquor from the recrystallization. If the acetic acid mother liquor is treated with water until turbid and then chilled, 4–5 g. of yellow crystals may be obtained. The material recovered from both liquors (6–8 g. in all) cannot be obtained in a perfectly colorless condition but the melting point is correct after recrystallization from alcohol.
2. Judefind and Reid[1] have shown that many acids may be identified by conversion into their *p*-bromophenacyl esters by the action of *p*-bromophenacyl bromide on the sodium salts.

3. Discussion

p-Bromophenacyl bromide can be prepared by the interaction of bromobenzene and bromoacetyl chloride in the presence of aluminum chloride,[2] and by the bromination of *p*-bromoacetophenone.[1], [3]

References and Notes

1. Judefind and Reid, J. Am. Chem. Soc. **42**, 1045 (1920).
2. Collet, Compt. rend. **125**, 717 (1897).
3. Collet, Bull. soc. chim. (3) **21**, 67 (1899).

Appendix
Chemical Abstracts Nomenclature (Collective Index Number);

(Registry Number)

p-bromophenacyl esters

ethyl alcohol (64-17-5)

acetic acid (64-19-7)

bromine (7726-95-6)

Acetophenone (98-86-2)

aluminum chloride (3495-54-3)

bromobenzene (108-86-1)

bromoacetyl chloride (22118-09-8)

p-Bromophenacyl bromide (99-73-0)

p-Bromoacetophenone (99-90-1)