



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.

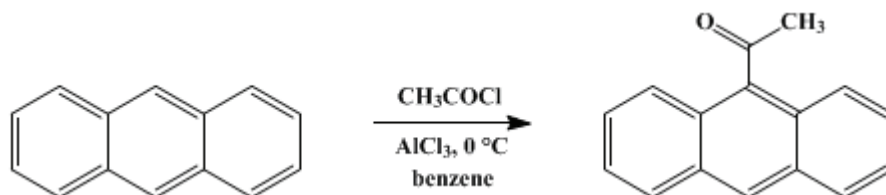
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 4, p.8 (1963); Vol. 30, p.1 (1950).

9-ACETYLANTHRACENE

[Ketone, 9-anthryl methyl]



Submitted by Charles Merritt, Jr. and Charles E. Braun¹.

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1. Procedure

Fifty grams (0.28 mole) of purified [anthracene](#) ([Note 1](#)) is suspended in 320 ml. of anhydrous [benzene](#) and 120 ml. (1.68 moles) of reagent grade [acetyl chloride](#) contained in a 1-l. three-necked flask. The flask is fitted with a thermometer which is immersed in the suspension, a calcium chloride drying tube, an efficient motor-driven sealed stirrer, and a rubber addition tube to which a 125-ml. Erlenmeyer flask containing 75 g. (0.56 mole) of anhydrous [aluminum chloride](#) is attached.²

The flask is surrounded by an ice-calcium chloride cooling mixture, and the [aluminum chloride](#) is added in small portions from the Erlenmeyer flask at such a rate that the temperature is maintained between -5° and 0° . After the addition is complete, the mixture is stirred for an additional 30 minutes, and the temperature is then allowed to rise slowly to 10° . The red complex which forms is collected with suction on a sintered-glass funnel and washed thoroughly with dry [benzene](#) ([Note 2](#)). The complex is added in small portions by means of a spatula with stirring to a 600-ml. beaker nearly filled with a mixture of ice and concentrated [hydrochloric acid](#). The mixture is then allowed to come to room temperature, and the crude ketone is collected on a suction filter.

The product is digested under reflux for about 20 minutes with 100–150 ml. of boiling 95% [ethanol](#). The suspension ([Note 3](#)) is cooled quickly almost to room temperature and filtered rapidly with suction to remove any [anthracene](#). The [9-acetylanthracene](#), which separates in the filtrate, is redissolved by heating and allowed to crystallize by slowly cooling the solution (finally to $0-5^{\circ}$ in an icebox) ([Note 4](#)). A second recrystallization from 95% [ethanol](#) yields 35–37 g. (57–60%) of light-tan granules of [9-acetylanthracene](#) melting at $75-76^{\circ}$ ([Note 5](#)).

2. Notes

1. The Eastman Kodak Company grade melting at $214-215^{\circ}$ is satisfactory. Technical grade [anthracene](#) can be purified by codistillation with [ethylene glycol](#). (See Fieser, *Experiments in Organic Chemistry*, 2nd ed., p. 345, footnote 13, D. C. Heath and Company, 1941.)
2. A regular Büchner funnel fitted with a mat of glass wool can be employed successfully. The filtration should be carried out as rapidly as possible, and the hydrolysis should be performed immediately thereafter if the humidity is high to minimize reaction on the funnel.
3. Most of the unreacted [anthracene](#) remains undissolved as a brown fluffy residue.
4. If the product has a tendency to separate as an oil, the addition of more solvent followed by heating to redissolve the material and subsequent cooling will usually yield a crystalline product.
5. Lüttringhaus and Kacer³ reported the melting point as ca. 80° , but May and Mosettig⁴ have found it to be $74-76^{\circ}$.

3. Discussion

The procedure described is essentially that of Lüttringhaus and Kacer³ except for the method of

isolation of the product, which is due to May.⁵

References and Notes

1. University of Vermont, Burlington, Vermont.
 2. Fieser, *Experiments in Organic Chemistry*, 3rd ed., p. 265, Fig. 46.4, D. C. Heath and Company, 1955.
 3. Lüttringhaus and Kacer, Ger. pat. 493,688 [*C. A.*, **24**, 2757 (1930)].
 4. May and Mosettig, *J. Am. Chem. Soc.*, **70**, 686 (1948).
 5. May, Private communication.
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Appendix Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

ethanol (64-17-5)

hydrochloric acid (7647-01-0)

Benzene (71-43-2)

acetyl chloride (75-36-5)

aluminum chloride (3495-54-3)

ethylene glycol (107-21-1)

anthracene (120-12-7)

9-Acetylanthracene,
Ketone, 9-anthryl methyl (784-04-3)