



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](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.

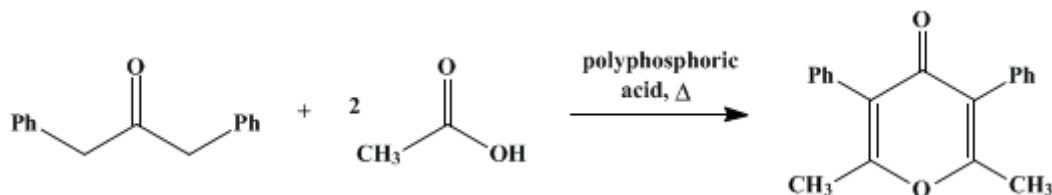
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*These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.*

*Organic Syntheses, Coll. Vol. 5, p.450 (1973); Vol. 47, p.54 (1967).*

## 2,6-DIMETHYL-3,5-DIPHENYL-4H-PYRAN-4-ONE

[4*H*-Pyran-4-one, 2,6-dimethyl-3,5-diphenyl-]



Submitted by Thomas L. Emmick and Robert L. Letsinger<sup>1</sup>.

Checked by Donald J. MacGregor and Peter Yates.

### 1. Procedure

A mixture of 400 g. of polyphosphoric acid and 250 ml. of glacial acetic acid is heated to reflux in a 2-l. round-bottomed flask equipped with a stirrer, reflux condenser, and thermometer. Dibenzyl ketone (42.0 g., 0.200 mole) (Note 1) is then added, and the reaction mixture is heated at reflux (130–135°) for 1.5 hours. The solution is cooled to 30° in an ice water bath, and 1 l. of water is added slowly with stirring. The brown precipitate which forms is collected by filtration, washed with 1 l. of water, and dissolved in 1 l. of hot benzene. The hot benzene solution is treated with 2 g. of activated carbon, filtered hot through a pad of diatomaceous earth, dried with 10 g. of magnesium sulfate, decanted from the magnesium sulfate, and concentrated to 500 ml. On addition of 450 ml. of hexane and cooling to 5–10°, tan crystals of the crude pyranone separate. Filtration affords 25–27 g. (45–49%) of product melting at 202–206°. For purification this material is dissolved in 500 ml. of hot benzene, treated with 1 g. of activated carbon as before, and precipitated from solution by the addition of 250 ml. of hexane and cooling of the mixture to 5–10°. On filtration 19–21 g. (34–38%) of 2,6-dimethyl-3,5-diphenyl-4*H*-pyran-4-one is obtained. This material melts sharply at 207–209° (Note 2). An additional quantity (3–4 g., 5–7%) of somewhat less pure product (m.p. 204–206°) may be recovered by evaporation of the filtrate and recrystallization of the residue from 200 ml. of benzene-hexane (50% benzene by volume).

### 2. Notes

1. For this preparation Matheson, Coleman and Bell practical grade dibenzyl ketone was recrystallized once from anhydrous ether at –70°. It melted at 33–34°. Practical grade dibenzyl ketone may be used directly; however, the yield of the pyranone is somewhat lower.
2. The corrected melting point is 209.5–210.0°. Melting points were obtained with a Fisher-Johns apparatus. The recrystallized sample retained a pale tan shade.

### 3. Discussion

This procedure is a modification of that of Letsinger and Jamison.<sup>2</sup> The pyranone has also been prepared by treatment of dibenzyl ketone with acetic anhydride-perchloric acid or acetyl chloride-aluminum chloride.<sup>3</sup>

### 4. Merits of the Preparation

This procedure represents a simple and unique route to certain pyran-4-ones. The reaction can be applied also to benzyl methyl ketone and diethyl ketone; the corresponding pyran-4-ones are obtained in yields of 48% and 26%, respectively.

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### References and Notes

1. Northwestern University, Evanston, Illinois.
  2. R. L. Letsinger and J. D. Jamison, *J. Am. Chem. Soc.*, **83**, 193 (1961).
  3. A. T. Balaban, G. D. Mateescu, and C. D. Nenitzescu, *Acad. Rep. Populare Romine, Studii Cercetari Chim.*, **9**, 211 (1961) [*C.A.*, **57**, 15065 (1962)].
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**Appendix**  
**Chemical Abstracts Nomenclature (Collective Index Number);**  
**(Registry Number)**

polyphosphoric acid

acetic acid (64-19-7)

Benzene (71-43-2)

ether (60-29-7)

carbon (7782-42-5)

benzyl methyl ketone (103-79-7)

magnesium sulfate (7487-88-9)

dibenzyl ketone (102-04-5)

diethyl ketone (96-22-0)

benzene-hexane (1077-16-3)

hexane (110-54-3)

2,6-Dimethyl-3,5-diphenyl-4H-pyran-4-one,  
4H-Pyran-4-one, 2,6-dimethyl-3,5-diphenyl- (33731-54-3)

acetic anhydride-perchloric acid

acetyl chloride-aluminum chloride