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.

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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.

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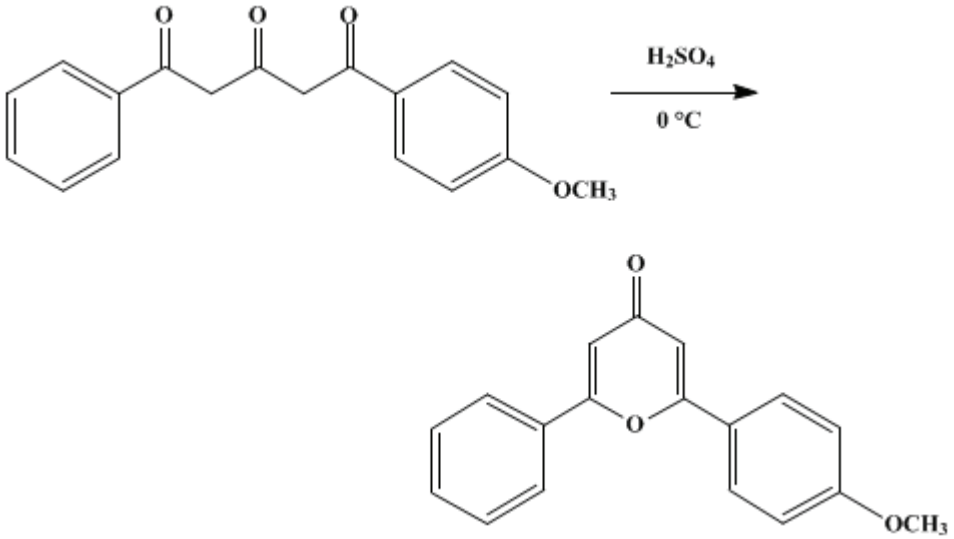
2-(*p*-METHOXYPHENYL)-6-PHENYL-4-PYRONE

[4H-Pyran-4-one, 2-(*p*-methoxyphenyl)-6-phenyl-]



Submitted by Marion L. Miles and Charles R. Hauser[1].
Checked by Victor Nelson, Wayland E. Noland, and William E. Parham.

1. Procedure

In a 50-ml. Erlenmeyer flask is placed 10 ml. of concentrated (36*N*) sulfuric acid (Note 1), and the flask is then immersed in an ice water bath. When the temperature of the acid reaches 0°, 2.96 g. (0.010 mole) of 1-(*p*-methoxyphenyl)-5-phenyl-1,3,5-pentanetrione (Note 2) is added in small portions. As each portion is added, the flask is swirled until the triketone dissolves. After the addition is completed, the solution is kept at 0° for 1 hour and then poured into 500 ml. of cold water. To the resulting slurry is added solid sodium bicarbonate until a pH of 7–8 (Note 3) is obtained. The mixture is filtered, and the filter cake is washed with cold water and then recrystallized from 15 ml. of 95% ethanol to give 2.46–2.72 g. (88–98%) of 2-(*p*-methoxyphenyl)-6-phenyl-4-pyrone, m.p. 161–163°.

2. Notes

1. Regular commercial grade of concentrated sulfuric acid (sp. gr. 1.84) obtained from the General Chemical Division of Allied Chemical Corporation was used.
2. For the preparation of this compound see this volume, p. 718.
3. This pyrone has a tendency to form a salt in aqueous sulfuric acid. The submitters used "Hydrion" paper to check the pH.

3. Discussion

The method is an adaptation of the procedure of Light and Hauser.[2] 2-(*p*-Methoxyphenyl)-6-phenyl-4-pyrone has been prepared in 50% yield by a Claisen-type acylation of *p*-methoxyacetophenone with ethyl phenylpropiolate accompanied by cyclization.[3]

4. Merits of the Preparation

This procedure offers an extremely simple and fairly general method for the preparation of 2,6-disubstituted 4-pyrones. Pyrones which have been prepared[2] by this procedure are: 2-methyl-6-phenyl-4-pyrone (60%), 2-(*p*-chlorophenyl)-6-methyl-4-pyrone (90%), 2,6-diphenyl-4-pyrone (91%), 2-(*p*-

chlorophenyl)-6-phenyl-4-pyrone (90%), 2-phenyl-6-(3-pyridyl)-4-pyrone (91%), 5,6,7,8-tetrahydroflavone (76%), 4'-methoxy-5,6,7,8-tetrahydroflavone (70%), cyclopenteno[*b*]-6-(*p*-methoxyphenyl)-4-pyrone (59%), and flavone (63%).

References and Notes

1. Chemistry Department, Duke University, Durham, North Carolina. This research was supported by the National Institutes of Health.
2. R. J. Light and C. R. Hauser, *J. Org. Chem.*, **25**, 538 (1960).
3. G. Soliman and I. E. El-Kholy, *J. Chem. Soc.*, 1755 (1954).

Appendix
Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

cyclopenteno[b]-6-(p-methoxyphenyl)-4-pyrone

ethanol (64-17-5)

sulfuric acid (7664-93-9)

sodium bicarbonate (144-55-8)

ethyl phenylpropiolate (2216-94-6)

Flavone (525-82-6)

2-methyl-6-phenyl-4-pyrone

2,6-diphenyl-4-pyrone

2-phenyl-6-(3-pyridyl)-4-pyrone

5,6,7,8-tetrahydroflavone

p-Methoxyacetophenone (100-06-1)

1-(p-Methoxyphenyl)-5-phenyl-1,3,5-pentanetrione (1678-17-7)

2-(p-Methoxyphenyl)-6-phenyl-4-pyrone,
4H-Pyran-4-one, 2-(p-methoxyphenyl)-6-phenyl- (14116-43-9)

2-(p-chlorophenyl)-6-methyl-4-pyrone

2-(p-chlorophenyl)-6-phenyl-4-pyrone

4'-methoxy-5,6,7,8-tetrahydroflavone