



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.

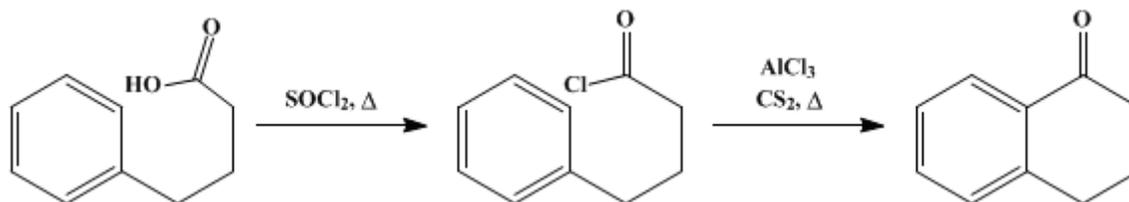
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*These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.*

*Organic Syntheses, Coll. Vol. 2, p.569 (1943); Vol. 15, p.77 (1935).*

## $\alpha$ -TETRALONE

### [1(2)-Naphthalenone, 3,4-dihydro-]



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Checked by C. R. Noller

### 1. Procedure

In a 500-cc. round-bottomed flask, fitted with a reflux condenser carrying at the top a tube leading to a gas absorption trap, are placed 32.8 g. (0.2 mole) of  $\gamma$ -phenylbutyric acid (p. 499) and 20 cc. (32 g., 0.27 mole) of thionyl chloride (Note 1). The mixture is carefully heated on a steam bath until the acid is melted, and then the reaction is allowed to proceed without the application of external heat. After twenty-five to thirty minutes hydrogen chloride is no longer evolved and the mixture is warmed on the steam bath for ten minutes. The flask is then connected to the water pump, evacuated, and heated for ten minutes on the steam bath and finally for two or three minutes over a small flame in order to remove the excess thionyl chloride. The acid chloride thus obtained is a nearly colorless liquid and needs no further purification. The flask is cooled, 175 cc. of carbon disulfide is added, and the solution cooled in an ice bath. Thirty grams (0.23 mole) of aluminum chloride is added rapidly in one lot, and the flask is immediately connected to the reflux condenser. After a few minutes, the rapid evolution of hydrogen chloride ceases and the mixture is slowly warmed to the boiling point on the steam bath. After heating and shaking the mixture for ten minutes the reaction is complete. The reaction mixture is cooled to 0°, and the aluminum chloride complex is decomposed by the careful addition, with shaking, of 100 g. of ice. Twenty-five cubic centimeters of concentrated hydrochloric acid is added and the mixture transferred to a 2-l. round-bottomed flask and steam-distilled (Note 2). The carbon disulfide distils first (Note 3), then there is a definite break in the distillation, after which the reaction product comes over completely in about 2 l. of the next distillate. The oil is separated, and the water is extracted three times with 100-cc. portions of benzene. The oil and extracts are combined, the solvent is removed, and the residue is distilled at reduced pressure. The yield of  $\alpha$ -tetralone boiling at 105–107°/2 mm. is 21.5–26.5 g. (74–91 per cent of the theoretical amount based on the  $\gamma$ -phenylbutyric acid).

### 2. Notes

1. The thionyl chloride was purified by distilling 50 g. of commercial thionyl chloride from 10 cc. of quinoline and then from 20 cc. of boiled linseed oil in a system protected from moisture.
2. It is advisable to use an efficient condenser system, such as that described in *Org. Syn. Coll. Vol. I, 1941, 479*, as  $\alpha$ -tetralone is only moderately volatile with steam.
3. On recovery of the carbon disulfide there is not more than a trace of residue.

### 3. Discussion

$\alpha$ -Tetralone has been obtained from the catalytic hydrogenation of  $\alpha$ -naphthol;<sup>1</sup> from  $\gamma$ -phenylbutyryl chloride and aluminum chloride;<sup>2</sup> from  $\gamma$ -phenylbutyric acid and concentrated sulfuric acid;<sup>3</sup> and by the oxidation of tetralin with chromic anhydride<sup>4</sup> or with atmospheric oxygen.<sup>5</sup> Detailed directions for preparing  $\alpha$ -tetralone by this last-named method are given in *Org. Syn. 20, 94*.

This preparation is referenced from:

- Org. Syn. Coll. Vol. 3, 798
- Org. Syn. Coll. Vol. 4, 898

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## References and Notes

1. Schroeter, Ger. pat. 352,720 [C. A. **17**, 1245 (1923)].
  2. Kipping and Hill, J. Chem. Soc. **75**, 147 (1899); Amagat, Bull. soc. chim. (4) **41**, 940 (1927).
  3. Krollpfeiffer and Schäfer, Ber. **56**, 624 (1923); Horne and Shriner, J. Am. Chem. Soc. **55**, 4652 (1933); Cook and Hewett, J. Chem. Soc. **1934**, 373.
  4. Schroeter, Ger. pat. 346,948 [Frdl. **14**, 491 (1921–25)].
  5. I. G. Farbenind. A.-G., Ger. pat. 539,476 [C. A. **26**, 1614 (1932)]; Cook, J. Chem. Soc. **1938**, 1778; Hock and Susemihl, Ber. **66**, 61 (1933); Brown, Widiger, and Letang, J. Am. Chem. Soc. **61**, 2601 (1939).
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## Appendix

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

1(2)-Naphthalenone, 3,4-dihydro-

sulfuric acid (7664-93-9)

hydrogen chloride,  
hydrochloric acid (7647-01-0)

Benzene (71-43-2)

thionyl chloride (7719-09-7)

$\alpha$ -naphthol (90-15-3)

aluminum chloride (3495-54-3)

carbon disulfide (75-15-0)

Quinoline (91-22-5)

chromic anhydride

Tetralin (119-64-2)

$\gamma$ -Phenylbutyric acid (1821-12-1)

$\alpha$ -Tetralone (529-34-0)

$\gamma$ -phenylbutyryl chloride