



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.

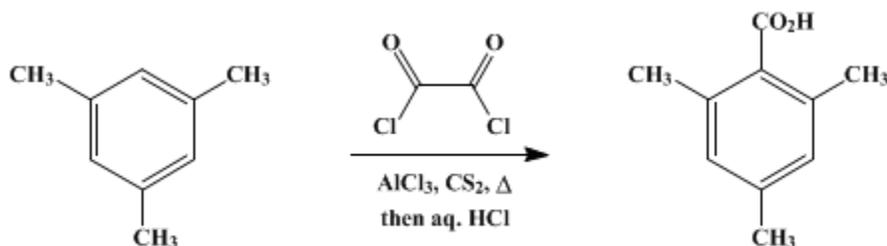
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*These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.*

*Organic Syntheses, Coll. Vol. 5, p.706 (1973); Vol. 44, p.69 (1964).*

## MESITOIC ACID

### [Benzoic acid, 2,4,6-trimethyl-]



Submitted by Phillip E. Sokol<sup>1</sup>

Checked by Melvin S. Newman and Vern G. DeVries.

### 1. Procedure

*Caution! The reaction should be carried out in a hood because carbon monoxide is evolved.*

The apparatus consists of a 2-l. three-necked flask fitted with a sealed stirrer, a 500-ml. addition funnel, and a condenser protected by a drying tube connected to an alkaline trap. In it are placed 146 g. (1.10 moles) of anhydrous aluminum chloride (Note 1) and 700 ml. of dry carbon disulfide. The suspension is cooled to 10–15° in an ice bath, and 139 g. (1.10 moles) of oxalyl chloride (Note 2) is added dropwise with stirring over a 30-minute period. After this addition the reaction mixture is stirred for 15 minutes. A solution of 120 g. (1.00 mole) of mesitylene (Note 3) in 200 ml. of dry carbon disulfide is added dropwise with stirring over a 1-hour period to the mixture, the temperature being maintained at 10–15°. Hydrogen chloride evolution is observed after about 5 minutes, and a red complex soon forms.

After the addition is completed, the reaction mixture is refluxed for 1 hour and is then poured very cautiously with manual stirring onto a mixture of 2 kg. of crushed ice and 300 ml. of 12*N* hydrochloric acid in a 4-l. beaker. The mixture thus formed is extracted with three 250-ml. portions of carbon tetrachloride. The combined organic extracts are washed with two 500-ml. portions of water, and the acid is then extracted with 500 ml. of ice-cold 10% sodium hydroxide solution. The aqueous extract is then slowly added to 250 ml. of 6*N* hydrochloric acid. The suspension is cooled, and the mesitoic acid is separated by filtration, washed thoroughly with water, and dried. The colorless crude acid (m.p. 149–150°) weighs 106–124 g. (65–76%) (Note 4) and is sufficiently pure for most purposes (Note 5).

### 2. Notes

1. Good results have been obtained with several different varieties of anhydrous aluminum chloride.
2. High-purity commercial oxalyl chloride was used without further purification.
3. Commercial mesitylene of high purity (99+%) was used.
4. Similar yields were obtained when experiments were run on a 0.10-mole scale.
5. For recrystallization, 10 g. of crude acid is dissolved in 20 ml. of 45% methanol at reflux. About 9.5 g. of mesitoic acid, m.p. 153–154°. (uncor.),<sup>2</sup> is obtained.

### 3. Discussion

Mesitoic acid has been prepared by carbonation of mesitylmagnesium bromide;<sup>2,3,4</sup> by hydrolysis of its amide prepared by condensation of mesitylene with carbamyl chloride under the influence of aluminum chloride;<sup>5</sup> by oxidation of isodurene with dilute nitric acid;<sup>6,7</sup> by distillation of 2,4,6-trimethylmandelic acid (low yield);<sup>8</sup> by dry distillation of 2,4,6-trimethylphenylglyoxylic acid;<sup>9</sup> by

oxidation of the latter with potassium permanganate;<sup>10</sup> and by treating 2,4,6-trimethylphenylglyoxylic acid with concentrated sulfuric acid in the cold<sup>11</sup> or with heating.<sup>12</sup>

#### 4. Merits of the Preparation

The method described in this preparation of mesitoic acid avoids the preparation of bromomesitylene,<sup>13</sup> and the yield of acid is essentially the same as that from the two-step synthesis.<sup>2,13</sup> This procedure appears to be general and can be used to prepare such acids as  $\alpha$ - and  $\beta$ -naphthoic acids,<sup>14</sup> cumenecarboxylic acid, 2,5-dimethylbenzoic acid, and durenecarboxylic acid. Carboxylic acids could not be obtained from benzothiophene, veratrole, *p*-dimethoxybenzene, and ferrocene under the conditions of this reaction. Although there has been no exhaustive study, this procedure is probably applicable to a variety of aromatic compounds, especially alkylated aromatics. Aromatic compounds which readily undergo oxidation, *e.g.*, ferrocene, catechol, and hydroquinone, do not lend themselves to this method.

This preparation is referenced from:

- *Org. Syn. Coll. Vol. 7*, 420

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#### References and Notes

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#### Appendix

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

Ferrocene

sulfuric acid (7664-93-9)

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

methanol (67-56-1)

sodium hydroxide (1310-73-2)

carbon monoxide (630-08-0)  
hydroquinone (123-31-9)  
nitric acid (7697-37-2)  
potassium permanganate (7722-64-7)  
carbon tetrachloride (56-23-5)  
aluminum chloride (3495-54-3)  
carbon disulfide (75-15-0)  
Catechol (120-80-9)  
Mesitylene (108-67-8)  
Bromomesitylene (27129-86-8)  
Isodurene (527-53-7)  
carbonyl chloride  
veratrole (91-16-7)  
oxalyl chloride (79-37-8)  
2,4,6-trimethylphenylglyoxylic acid (3112-46-7)  
mesitylmagnesium bromide  
Mesitylic acid,  
Benzoic acid, 2,4,6-trimethyl- (480-63-7)  
2,4,6-trimethylmandelic acid  
p-dimethoxybenzene (150-78-7)  
durene-carboxylic acid  
benzothiophene (95-15-8)  
cumene-carboxylic acid  
2,5-dimethylbenzoic acid (610-72-0)