



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.

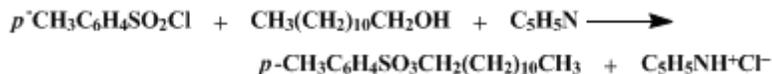
In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior 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. 3, p.366 (1955); Vol. 20, p.50 (1940).

***n*-DODECYL (LAURYL) *p*-TOLUENESULFONATE**



Submitted by C. S. Marvel and V. C. Sekera.

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1. Procedure

In a 1-l. three-necked flask fitted with a stirrer and thermometer are placed 93 g. (0.5 mole) of [dodecanol](#) ([Note 1](#)) and 158 g. (2 moles) of [pyridine](#). The flask is surrounded by a water bath sufficiently cold to lower the temperature of the mixture to 10°. At this temperature 105 g. (0.55 mole) of [p-toluenesulfonyl chloride](#) is added in portions over a 20- to 30-minute period, or at such a rate that the temperature does not exceed 20° at any time. The mixture is then stirred for 3 hours at a temperature below 20°, after which it is diluted with 300 ml. of [hydrochloric acid](#) (sp. gr. 1.19) in 1 l. of ice water. The ester which crystallizes is collected on a chilled Büchner funnel and sucked as dry as possible. The solid is transferred to a 600-ml. beaker, 250–300 ml. of [methanol](#) is added, and the mixture is warmed on the steam bath until the ester melts. It is then cooled in a freezing mixture while being stirred continuously; the ester separates in a fairly fine state. It is then collected on a chilled funnel and allowed to dry in the air, preferably at a temperature below 20°. The yield of ester is 152–156 g. (88–90% based upon the [dodecanol](#) used). It melts at 20–25° ([Note 2](#)) and is sufficiently pure for most purposes.

If a purer product is desired, it is recrystallized from petroleum ether (b.p. 30–60°), using 4 ml. per 3 g., and drying over anhydrous [sodium sulfate](#). The solution is chilled to 0° and the ester filtered on a chilled funnel; the recovery is 90%, and the melting point is 28–30°. Evaporation of the solvent to a small volume deposits an additional amount ([Note 3](#)).

2. Notes

1. [Dodecanol](#) ([lauryl alcohol](#)), m.p. 20–22°; [pyridine](#), b.p. 113–115°; and [p-toluenesulfonyl chloride](#), m.p. 66–68°, are used.
2. The ester contains traces of water, which makes the melting point unreliable; the freezing point is 24–25°.
3. The following esters have been made in essentially the same yields; [butyl p-toluenesulfonate](#) [*Org. Syntheses Coll. Vol. 1, 145 (1941)*]; [n-tetradecyl p-toluenesulfonate](#), m.p. 35°; [n-hexadecyl p-toluenesulfonate](#), m.p. 49°; [n-octadecyl p-toluenesulfonate](#), m.p. 56°; [n-decyl p-bromobenzenesulfonate](#), m.p. 43–44°; [n-dodecyl p-bromobenzenesulfonate](#), m.p. 49°; [n-tetradecyl p-bromobenzenesulfonate](#), m.p. 51.5°; [n-hexadecyl p-bromobenzenesulfonate](#), m.p. 60°; [n-octadecyl p-bromobenzenesulfonate](#), m.p. 64–65°.

3. Discussion

n-Dodecyl *p*-toluenesulfonate has been prepared only by the action of *p*-toluenesulfonyl chloride on [dodecanol-1](#) in the presence of [pyridine](#)¹ according to the general procedure developed by Patterson and Frew² for making esters of sulfonic acids.

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 6, 376](#)
- [Org. Syn. Coll. Vol. 6, 652](#)

1. Sekera and Marvel, *J. Am. Chem. Soc.*, **55**, 345 (1933).
 2. Patterson and Frew, *J. Chem. Soc.*, **89**, 332 (1906).
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Appendix
Chemical Abstracts Nomenclature (Collective Index Number);
(Registry Number)

petroleum ether

hydrochloric acid (7647-01-0)

methanol (67-56-1)

Lauryl alcohol,
dodecanol,
dodecanol-1 (112-53-8)

sodium sulfate (7757-82-6)

pyridine (110-86-1)

p-Toluenesulfonyl chloride (98-59-9)

n-Dodecyl p-toluenesulfonate (10157-76-3)

Butyl p-toluenesulfonate (778-28-9)

n-DODECYL (LAURYL) p-TOLUENESULFONATE

n-tetradecyl p-toluenesulfonate

n-hexadecyl p-toluenesulfonate

n-octadecyl p-toluenesulfonate (3386-32-1)

n-decyl p-bromobenzenesulfonate

n-dodecyl p-bromobenzenesulfonate

n-tetradecyl p-bromobenzenesulfonate

n-hexadecyl p-bromobenzenesulfonate

n-octadecyl p-bromobenzenesulfonate