





Table I. Sodium Tetrachloroaluminate Promoted Cyclodehydrations

reactant	product	contact time, min	yield, %	PPA yield, % <sup>a</sup>
3-phenylpropanoic acid		5	96	87
4-phenylbutanoic acid		20	96	89
2-phenylbenzoic acid		20	100	99
2-phenoxybenzoic acid		20	99	97

<sup>a</sup> G. Metz, *Synthesis*, 612, 614 (1972).

identical with an authentic sample, with a melting point of 39–40 °C (lit. mp 40–42 °C).

**1-Tetralone (2).** Reaction of 4-phenylbutanoic acid for 20 min at 180–200 °C afforded a 96% yield of 1-tetralone, spectroscopically identical with an authentic sample.

**9-Fluorenone (3).** Reaction of *o*-phenylbenzoic acid for 20 min at 180–190 °C afforded a quantitative yield of 9-fluorenone, spectroscopically identical with an authentic sample, with a melting point of 81–82 °C (lit. mp 84 °C).

**Xanthone (4).** Reaction of *o*-phenoxybenzoic acid for 25 min at 200 °C afforded a 99% yield of xanthone, spectroscopically identical with an authentic sample, with a melting point of 172–174 °C (lit. mp 174 °C).

**Registry No.** 1, 83-33-0; 2, 529-34-0; 3, 486-25-9; 4, 90-47-1; 3-phenylpropanoic acid, 501-52-0; 4-phenylbutanoic acid, 1821-12-1; 2-phenylbenzoic acid, 947-84-2; 2-phenoxybenzoic acid, 2243-42-7; sodium tetrachloroaluminate, 7784-16-9.

## A New, Simple Synthesis of Tropone

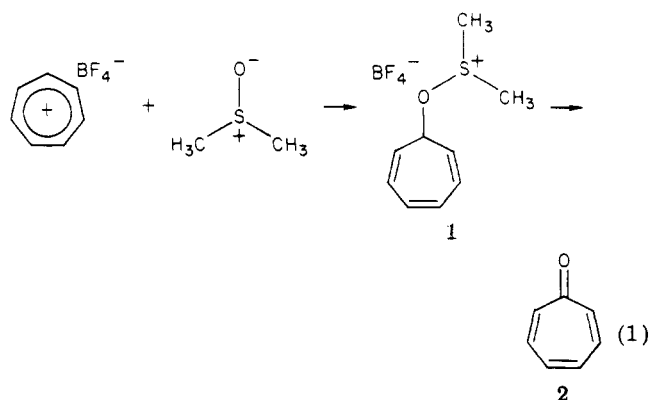
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Attempts to prepare tropone (2) by various literature methods<sup>1-3</sup> have met with only limited success in our hands: low yields and/or messy reaction conditions led us to consider an alternate route to tropone. The common intermediate of the Moffatt oxidation,<sup>4</sup> the Corey–Kim oxidation,<sup>5</sup> and the Swern oxidation,<sup>6</sup> among others, appears to be an effective species for the generation of the carbonyl group. An analogous intermediate, 1, should be produced simply on mixing a tropylium salt with dimethyl sulfoxide (Me<sub>2</sub>SO). Addition of triethylamine (TEA) should give tropone (eq 1).

At room temperature this sequence produced no product. At 55 °C, however, substantial amounts of



tropone were obtained. Surprisingly, a control reaction carried out without adding TEA proved to be just as good as those with TEA. The progress of the reaction was followed by NMR (in Me<sub>2</sub>SO-*d*<sub>6</sub>). Over the course of 4 h at 55 °C, the singlet of the tropylium salt was slowly converted to the complex pattern of a substituted cycloheptatriene. No tropone was observed, either with or without TEA, until the addition of water and chloroform, at which point the broad singlet of tropone ( $\delta$  7.05) replaced all the other peaks. Although the conditions have not been optimized, yields of 50% are routine. Our best yield to date is 58%.

## Experimental Section

**Tropone (2).** A solution of 3.0 g (0.0168 mol) of tropylium fluoborate<sup>7,8</sup> was dissolved in 15 mL of Me<sub>2</sub>SO<sup>9</sup> and heated overnight at 55 °C. The dark brown solution was cooled to room temperature and saturated aqueous sodium bicarbonate solution was added (with considerable gas evolution) until the mixture was basic to pH paper.<sup>10</sup> The aqueous mixture was extracted several times with chloroform and the combined organic layers were washed with water and stripped of solvent. Chromatography of the residue on silica gel, using ethyl acetate as eluant, afforded 1.03 g (58%) of tropone as a pale yellow oil, identical in all respects with tropone prepared by literature methods.<sup>1-3</sup>

On a sufficiently large scale, or on combination of several small-scale reactions, the chromatography could be replaced by a distillation. The major impurity was Me<sub>2</sub>SO (bp 37 °C (3 mm)); tropone distilled at 60 °C (3 mm).

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**Registry No.** 2, 539-80-0; tropylium fluoborate, 27081-10-3; Me<sub>2</sub>SO, 67-68-5.

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- (7) Tropylium fluoborate was prepared by the method of Conrow.<sup>8</sup> We have found that increasing the volume of solvent from 800 to 1200 mL and allowing the first step of the reaction to proceed overnight under nitrogen increases the yield and especially the ease of the reaction.
- (8) K. Conrow, "Organic Syntheses", Collect. Vol. V, Wiley, New York, N. Y., 1973, p 1138.
- (9) Me<sub>2</sub>SO was obtained from Eastman and used as received.
- (10) Some heat is necessary at this point. Generally the heat of neutralization is sufficient; however, reheating the mixture to 55 °C for 1 h does not hurt and may help. We thank Dr. David Ledlie of Bates College for this suggestion.

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