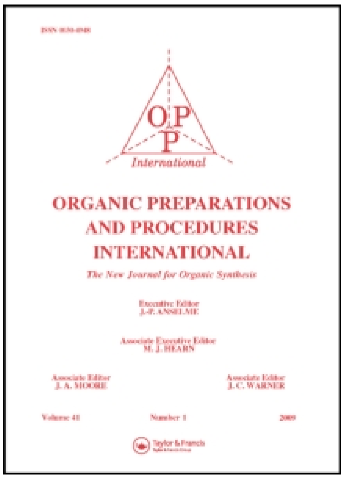

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A CONVENIENT PREPARATION OF 4-CYCLOPROPYLPHENOL

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A CONVENIENT PREPARATION OF 4-CYCLOPROPYLPHENOL

Submitted by (05/26/92)
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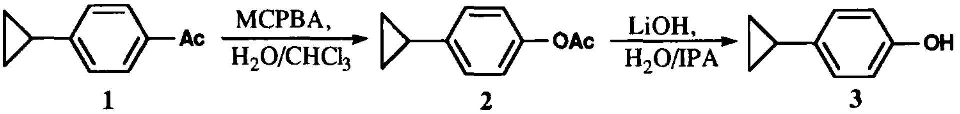
4-Cyclopropylphenol, an important intermediate[1] for the synthesis of agricultural and pharmaceutical products, has been obtained by a variety of reported methods[2,3] which however, are inefficient and low yielding. We now describe a variant of the Boissier[3] procedure which provides a facile and direct route to the title compound in an overall yield of 86% from 4-cyclopropylacetophenone with

3-chloroperoxybenzoic acid as the oxidant in the Baeyer-Villiger reaction; this procedure provided for easy isolation and purification of acetate **2**. Moreover, lithium hydroxide allows hydrolysis of the acetate under milder conditions than with standard saponification procedures.



EXPERIMENTAL SECTION

Unless otherwise noted, materials were obtained from commercial suppliers and were used without further purification. ^{1}H NMR spectra were obtained on a General Electric QE-300 NMR instrument at 300 MHz. Chemical shifts are expressed in ppm downfield from internal tetramethylsilane. Merck TLC plates were used for analytical TLC. Microanalyses were preformed by the Abbott Analytical Department. Melting points (uncorrected) were determined on a Thomas Hoover apparatus.

4-Cyclopropylphenyl Acetate (2).- A 2-L, one-neck, round-bottom flask equipped with a magnetic stirbar was charged with 76 g (0.475 mol) of 4-cyclopropylacetophenone,[4] 750 mL of chloroform and 250 mL of water. With rapid stirring, 205 g (0.594 mol) of 50-60% 3-chloroperoxybenzoic acid was added all at once. The suspension was stirred at room temperature overnight. The solid was filtered and discarded, and the liquid layers separated. The organic phase was washed with 1L of 5% potassium hydroxide solution, and then dried over anhydrous potassium carbonate. Removal of the solvent under reduced pressure furnished a yellow liquid, which was distilled to give 75 g (90%) of **2**, bp. 69-70°/0.3 mm as a colorless liquid, $n_D^{23°}$ 1.526. ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.65 (dq, 2H); 0.95 (dq, 2H); 1.85-1.95 (m, 1H); 2.28 (s, 3H); 6.95 (dt, 2H); 7.07 (dt, 2H).

Anal. Calcd. for $C_{11}H_{12}O_2$: C, 74.98; H, 6.86. Found: C, 74.93; H, 6.95

4-Cyclopropylphenol (3).- A 2-L, one-neck, round-bottom flask, equipped with a 500 mL pressure-equalizing funnel and a magnetic stirbar was charged with 70 g (0.39 mol) of 4-cyclopropylphenyl acetate (**2**) and 700 mL of isopropanol. With rapid stirring, 67 g (1.59 mol) of lithium hydroxide monohydrate in 350 mL water was added over 15 min. After addition was complete, removal of the solvent under reduced pressure furnished a white solid. Water (500 mL) was added and the suspension was cooled in an ice-water bath. With rapid stirring, 6N hydrochloric acid was added to pH 5. The precipitate was collected and dried to afford 51 g (96%) of **3** as a colorless solid, mp. 67-70°, lit.[3] bp. 110-112°/3-8 mm; mp. 65-68°. ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.6 (dq, 2H); 0.88 (dq, 2H); 1.8-1.9 (m, 1H); 4.73 (s, 1H); 6.73 (dt, 2H); 6.95 (dt, 2H).

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A NEW SYNTHESIS OF 3-AMINO-1-IMIDAZOLIN-4-ONES BY THE HYDRAZINOLYSIS OF 5-OXAZOLONES

Submitted by
(04/24/92)

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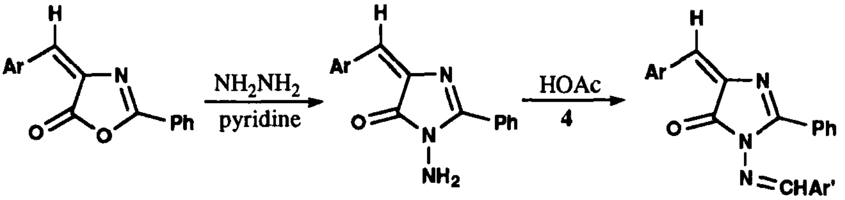
The reaction of hydrazine has been reported to give triazinones by heating with 5-phenyl-4-oxazolin-2-ones in dilute alkali,[1,2] isomeric pyrazolidin-3-ones,[3] with benzylidene oxazolones in



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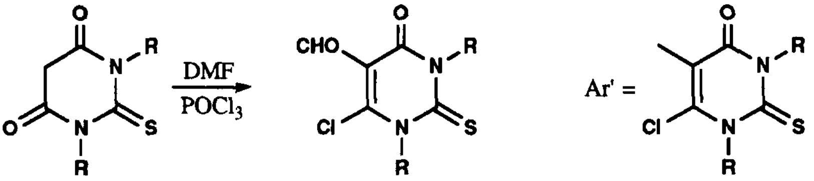

4 5

a) $R = 2\text{-MeOC}_6H_4$, $Ar = 4\text{-MeOC}_6H_4$
b) $R = C_6H_5$, $Ar = 4\text{-MeOC}_6H_4$
c) $R = 3\text{-MeC}_6H_4$, $Ar = 4\text{-MeOC}_6H_4$
d) $R = 4\text{-ClC}_6H_4$, $Ar = 4\text{-MeOC}_6H_4$
e) $R = 2\text{-MeOC}_6H_4$, $Ar = 4\text{-MeC}_6H_4$
f) $R = 3\text{-MeOC}_6H_4$, $Ar = 4\text{-MeC}_6H_4$