

## Indanylation of *o*-Cresol with Indene in the presence of Benzenesulphonic Acid

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### Abstract

Indanyl *o*-cresol has been obtained in high yield by the reaction of *o*-cresol with indene in the presence of benzenesulphonic acid. The effect of variation of temperature, molar ratio of *o*-cresol to indene, time of reaction and amount of benzenesulphonic acid on the reaction has been studied.

**Keywords:** Indanylation, *o*-Cresol, Indene, Benzenesulphonic acid, Antioxidant.

### Introduction

Alkylated phenols and their derivatives are the outstanding antioxidants and multifunctional stabilizers in fuels, lubricating oils and polymeric materials<sup>1-3</sup>. Some of their derivatives are strong herbicides and bactericides<sup>4-5</sup>. Alkylated cresols with long alkyl group are intermediates for surfactants and detergents<sup>6</sup>. Isomeric cresols have been alkylated with olefins by several authors<sup>7-28</sup>. But studies on the reactions of cresols with indene in the presence of benzenesulphonic acid are absent.

In the present work, reaction of *o*-cresol with indene in the presence of benzenesulphonic acid has been investigated.

### Experimental

The reactions were carried out in a three necked round bottomed flask fitted with a condenser, a thermometer, a dropping funnel and a stirrer. *o*-Cresol-catalyst mixture was charged into the flask, heated to the temperature of the experiment, then indene was introduced into the mixture gradually over a certain period of time (time of addition) with constant stirring. The reaction mixture was stirred for another period of time (time of stirring) at the same temperature after the addition of the total amount of indene.

The reaction mass was then cooled to room temperature, dissolved in petroleum ether and neutralized. The reaction mixture was then washed with distilled water several times and unreacted reactants and solvent were distilled off at atmospheric pressure. The residual product was finally distilled and characterized by spectral means.

### Results and Discussion

The reaction of *o*-cresol with indene was investigated in the presence of benzenesulphonic acid over the temperature range of 100 to 140°C. Molar ratio of *o*-cresol to indene was varied from 4:1 to 8:1, amount of benzenesulphonic acid was varied from 2 to 8% by wt. of *o*-cresol and time of reaction from 2 to 5h.

The reaction gave the isomeric mixture of indanyl *o*-cresol (indanyl group substituted the aromatic ring to the ortho- or para- position with respect to the -OH group). The effect of the variation of temperature, time of reaction and molar ratio of *o*-cresol to indene on the reaction has been shown in the Table 1. Under the conditions studied, the yield of the product increased with the increase of temperature (Expt. No. 2, 5 & 9), molar ratio of *o*-cresol to indene (Expt. No. 2, 4 & 7); amount of benzenesulphonic acid (Expt. No. 1, 2 & 3) and time of reaction (Expt. No. 2, 6 & 8).

**Table 1:** Alkylation of *o*-cresol with indene in presence of benzenesulphonic acid.

Expt. No.	Reaction conditions					% yield of indanyl <i>o</i> -cresol
	Temp., °C	Molar ratio of <i>o</i> -cresol to indene	Time of addition, h	Time of stirring, h	Amount of benzenesulphonic acid, % by wt. of <i>o</i> -cresol	
1	140	8:1	2	1	8	83.1
2	140	8:1	2	1	5	79.2
3	140	8:1	2	1	2	72.7
4	140	5:1	2	1	5	73.8
5	120	8:1	2	1	5	69.9
6	140	8:1	2	3	5	83.1
7	140	4:1	2	1	5	56.5
8	140	8:1	2	0	5	67.7
9	100	8:1	2	1	5	60.7

84

Thus, the maximum yield of the product was obtained under the following conditions: temperature = 140°C, molar ratio

of *o*-cresol to indene = 8:1, amount of benzenesulphonic acid = 8% by wt. of *o*-cresol, time of addition = 2h and time

of stirring = 1h. The UV-spectrum of the product showed strong absorption at  $\lambda_{\max} = 298.5$  nm in 0.01M methanol solution.

IR spectrum of the product showed absorption bands at 710-750  $\text{cm}^{-1}$  for 1, 2, 3-trisubstituted aromatic ring. Bands near 800-900  $\text{cm}^{-1}$  indicated the presence of 1,2,4- trisubstituted

aromatic ring. Band at 3400  $\text{cm}^{-1}$  accounted for the -OH group. Bands at 2910 $\text{cm}^{-1}$  and 1590 $\text{cm}^{-1}$  showed the C-H stretch and C=C stretch respectively.

**Table 2:**  $^1\text{H}$ NMR- spectrum of indanyl o-cresol.

Observed signals of the protons	Chemical shift in the $\delta$ ppm
Three protons on the $-\text{CH}_3$ group	2.3
All the protons on the indanyl group except four on the aromatic ring	3.1-3.6
Aromatic ring protons	6.9-7.6
-OH group proton	6.9

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