TABLE I

Aromatic nucleus	Bifunctional reactant	Catalyst	Yield, %	Indanone-1	M. p., °C.
Benzene	$ClCH_2CH_2COCl$	$AlCl_3$–H_2SO_4	54.5	Indanone-1	40–41
p-Xylene	$ClCH_2CH_2COCl$	$AlCl_3$–H_2SO_4	72.5	4,7-Dimethyl-[a,g]	76–77
α-Methylnaphthalene	$ClCH_2CH_2COCl$	$AlCl_3$–H_2SO_4	52.3	5-Methyl-(6,7-benzo)-[a]	130.5–131.5
o-Chlorotoluene	$ClCH_2CH_2COCl$	$AlCl_3$–H_2SO_4	45.0	5-Chloro-4(6)-methyl-[a]	77.5–78
p-Xylene	$CH_3CH{=}CHCOCl$	$AlCl_3$–H_2SO_4	35.4	3,4,7-Trimethyl-[b]	31–32
Benzene	$CH_3CH{=}CHCOCl$	$AlCl_3$–H_2SO_4	52.5	3-Methyl-[c,d]	Oil
p-Xylene	$CH_3CH{=}CHCOOH$	HF	81.0	3,4,7-Trimethyl-[b]	31–32
p-Xylene	$C_6H_5CH{=}CHCOOH$	HF	84.4	3-Phenyl-4,7-dimethyl-[e]	93.5–94.5
p-Xylene	$CH_2{=}C(CH_3)COOH$	HF	62.1	2,4,7-Trimethyl-[f]	Oil

[a] Mayer and Muller, *Ber.*, **60**, 2278 (1927). [b] Ref. 4. [c] Ref. 5. [d] B. p. 108–110° (4 mm.); semicarbazone, m. p. 206–207°. [e] V. Auwers and Risse, *Ann.*, **502**, 282 (1933). [f] New compound; see Experimental. [g] Plattner and Wyss, *Helv. Chim. Acta*, **24**, 483 (1941).

three hours of stirring at room temperature, the carbon disulfide was removed at the water-pump. To the residual oily complex was added 250 cc. of concentrated sulfuric acid and the mixture heated at 90° for forty-five minutes. After cooling the mixture to room temperature, it was poured onto 1 kg. of crushed ice. The flocculent yellow precipitate was extracted thoroughly with ether and benzene and the combined extracts washed successively with water, sodium carbonate solution, and water, and dried over anhydrous sodium sulfate. Replacement of the solvents by methanol yielded 23.2 g. (72.5%) of 4,7-dimethyl-indanone-1, m. p. 75.5–76.5°. Recrystallization from methanol raised the m. p. to 76–77°. This procedure is typical of that used for preparing all six indanones by method (a) as reported in Table I.

Method (b): 2,4,7-Trimethyl-indanone-1.—A mixture of 10 g. (0.094 mole) of *p*-xylene and 20.2 g. of 40% aqueous methacrylic acid (0.094 mole) was added to 115 g. of cold anhydrous liquid hydrogen fluoride in a pressure reactor and the mixture shaken to aid in solution of the reactants. The reactor was capped tightly and heated at 90° for six hours. After cooling, the mixture was poured into sodium carbonate solution and worked up in the usual manner. Vacuum distillation of the resultant oil yielded 10.2 g. (62.1%) of colorless oil, 2,4,7-trimethyl-indanone-1, b. p. 118–120° (3.5 mm.): $n^{22}D$, 1.5550. *Anal.* Calcd. for $C_{12}H_{14}O$: C, 82.72; H, 8.10. Found: C, 82.42; H, 8.67.

2,4-Dinitrophenylhydrazone, scarlet needles, m. p. 185.2–186° (cor.). *Anal.* Calcd. for $C_{18}H_{18}O_4N_4$: C, 61.01; H, 5.12; N, 15.83. Found: C, 61.52; H, 5.56; N, 15.55.

This procedure is typical of that used for preparing the three indanones by method (b) as reported in Table I.

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Preparation of Xanthene from Phenol

BY JANE HANAFIN[1]

The reaction of aluminum chloride with phenol, or with *o*-cresol–phenol mixtures, has been noted by investigators[2] as yielding diphenyl ether and xanthene. The small quantity of xanthene obtained could be attributed to cresol or other impurities present in the phenol, but no determination of the purity of the phenol used appears in the literature. The experiments reported below indicate that the impurities in the phenol here used were insufficient to account for the xanthene produced, and that the phenol itself must provide the methylene linkage between the aromatic rings in xanthene. The author is grateful to Dr. G. W. Wheland for suggesting and encouraging this work.

Experimental

Xanthene was prepared by the method of Merz and Weith,[2a] using only phenol and aluminum chloride in somewhat smaller amounts, *i. e.*, 103 g. and 60 g., respectively. Steam distillation was used to isolate the products. Xanthene was identified by its m. p., mixed m. p. with authentic samples, green fluorescence in sulfuric acid, and oxidation to xanthone; yield, 18 g. (20%) of the ether, 1.2 g. (1.3%) of the xanthene.

The amount of impurities in the phenol used was established by comparison of the cooling curves of an *o*-cresol–phenol mixture (0.49 mole % *o*-cresol) and of the phenol alone. This sensitive method is described by Schwab and Wichers.[3] The maximum impurity was found to be 0.16 mole %, insufficient to account for the 0.56 mole % xanthene recovered, even if the contaminant had been wholly *o*-cresol.

Previous experiments with *o*-cresol–phenol mixtures and aluminum chloride gave 4% xanthene. Mixed m. p.'s with other samples indicated that no methylxanthenes, reported to be a product with *o*-cresol,[4] were present.

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[2] (a) Merz and Weith, *Ber.*, **14**, 191 (1881); (b) Graebe, *ibid.*, **16**, 862 (1888).

[3] Schwab and Wichers, "Temperature, Its Measurement and Control," Am. Institute of Physics, 1941, p. 256.

[4] Postowsky and Lougowkin, *J. prakt. Chem.*, [2] **122**, 141 (1929).

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The Reaction of Amines with N-Methyl-N-nitroso-N'-nitroguanidine

BY RONALD A. HENRY

McKay and Wright[1] have shown that primary alkyl or arylamines and N-methyl-N-nitroso-N'-nitroguanidine react with the elimination of the methylnitrosamino group and formation of the corresponding alkyl or aryl substituted nitroguanidine. No mention is made, however, of the fate of the methylnitrosamino group. It has now been found that this latter group serves, either directly or indirectly, as a methylating agent since the principal by-product in the re-

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[1] McKay and Wright, THIS JOURNAL, **69**, 3028 (1947); (b) McKay, *ibid.*, **71**, 1968 (1949).