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Mixed Diacyl Derivatives of O-Aminophenol Containing an Acyl Derived from a Sulfonic Acid

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Benzene, aluminum chloride and methyl 2-furoate give methyl α -naphthoate in 56 per cent yield.

Benzene, aluminum chloride and methyl 2-methyl-3-furoate give methyl 4-phenyl-4, 5-dihydro-2-methyl-3-furoate; and substituted benzenes and other aryl types give related products.

A very wide variety of branched and straight chained alkyl halides react with ethyl 5-bromo-2-furoate in the presence of aluminum chloride to give ethyl 4-*tert.*-butyl-5-bromo-2-furoate. However, *n*-amyl bromide (unlike *n*-C₅H₁₁Cl, *n*-C₅H₁₁I, and the other RX compounds) gives ethyl 5-*tert.*-butyl-2-furoate exclusively, unless a purer grade (resublimed) aluminum chloride is used in which case the ethyl 4-*tert.*-butyl-5-bromo-2-furoate is obtained.

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HYDROQUINONE MONOACETATE

H. S. ORCOTT

Alkyl monoesters of hydroquinone and pyrocatechol have not hitherto been prepared. Hydroquinone monoacetate has been obtained by the following series of reactions. Hydroquinone plus one molar equivalent of carbobenzoxy chloride gives hydroquinone monobenzyl carbonate. This compound is acetylated, and then hydrogenated in alcohol with Pd or Ni catalyst. Hydroquinone monoacetate, plates or needles from petroleum ether, M. P. 57-59°C., is obtained from the residue.

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MIXED DIACYL DERIVATIVES OF *O*-AMINOPHENOL CONTAINING AN ACYL DERIVED FROM A SULFONIC ACID

L. CHAS. RAIFORD AND J. REID SHELTON

In previous work in this Laboratory it was found that, in general, only one mixed diacyl derivative can be obtained from *o*-aminophenol, regardless of the order of introduction of these acyls, provided both are derived from carboxylic acids. In one

case a migration of acyl from nitrogen to oxygen must have occurred. When one of these acyls is derived from a sulfonic acid no rearrangement is observed.¹

When one of the acyls is represented by a carboaryloxy radical and this is introduced into the amino group, attempts to prepare a diacyl derivative by means of an acyl radical derived from a carboxylic acid will fail. A benzoxazolone will be formed and the required phenol will be eliminated.²

It has now been found that a mixed diacyl derivative containing the carboaryloxy radical can be obtained, provided the other radical is *p*-tolylsulfonyl, and if the latter is introduced first. The behavior of this product toward hydrolyzing reagents is now being studied.

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IODINE SUBSTITUTION PRODUCTS OF VANILLIN

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In 1872 Carles prepared a mono- and a diiodovanillin, but did not characterize them. In the current edition of Beilstein they are recorded as of unknown structures.

Carles' work has now been repeated, and the structures of his products have been shown to be 5-iodovanillin and 2, 5-diiiodovanillin, respectively. The behavior of these and similar derivatives toward typical amino compounds has been studied. No pronounced steric hindrance was noted in the latter reactions. The relation between the numerical values of the melting points and the positions of the halogen atoms in the iodine derivatives, so far as they have been obtained, is in good agreement with that observed in the cases of the chlorine and bromine compounds, viz., that the lowest value is found for position 2, the next higher for 5 and the highest for 6. Likewise, for a given position the lowest is found for the chloride, the next higher for the bromide and the highest for the iodide.

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¹ J. Am. Chem. Soc., 53, 3420 (1931).

² J. Am. Chem. Soc., 56, 1586 (1934).