

1924

## Conclusions in Regard to the Methylation of Phenol

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### Recommended Citation

Lewis, Harry F. and Trieschmann, Wesley (1924) "Conclusions in Regard to the Methylation of Phenol," *Proceedings of the Iowa Academy of Science*, 31(1), 282-282.

Available at: <https://scholarworks.uni.edu/pias/vol31/iss1/69>

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of chlorine. It has been found that by passing chlorine through lime covered with water in the presence of iron or manganese as a catalyst that practically quantitative yields of oxygen are evolved. The method is believed to be commercially applicable for producing oxygen. It is also a superior method for laboratory demonstration and use.

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## CONCLUSIONS IN REGARD TO THE METHYLATION OF PHENOL

HARRY F. LEWIS AND WESLEY TRIESCHMANN

As the result of a large number of methylations carried out with phenol with dimethyl sulfate as the methylating agent, the following conclusions have been drawn:

1. Anisol is not methylated by the action of methyl sulfate on an aqueous solution of phenol in either neutral or acid condition.

2. The presence of either sodium or potassium hydroxide results in the methylation of phenol.

3. The amount of methylation is in the same order as the ratio of alkali to phenol, a tenth mol of alkali resulting in about a 10% methylation. An excess of alkali has no influence, at least in the direction of greater action.

4. The second methyl group can be used in methylation to a considerable extent; this is based upon the use of a half mol of dimethyl sulfate and one mol of phenol, in the presence of two to four mols of water and a mol and a half of sodium hydroxide, where yields as high as 70% of anisol have been obtained.

5. Considerable methylation takes place even at low temperatures with the first group entering into the reaction. Practically all of the first group may be used in this way.

6. Other things being equal, an increase in the amount of water present causes a decrease in the amount of methylation, two mols of water to one mol of phenol and one and a half mols of sodium hydroxide being about the lowest practical amount which can be used.

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## NOTE ON THE HYDROLYSIS OF NITROBENZENE

H. F. LEWIS AND G. W. THIESSEN

Wohl<sup>1</sup> describes the oxidation of nitrobenzene to nitrophenols by the action of powdered potassium hydroxide at temperatures

between 60° and 90°C. This reaction does not take place under similar conditions with solid sodium hydroxide.

Although Wohl found no organic reduction products and was at a loss to account for the remainder of the nitrobenzene which was lost, it was suggested by R. R. Renshaw that possibly he might have missed any inorganic products of hydrolysis which might have been present.

Consequently the authors have made a number of preparations under various conditions, the standard conditions comprising 5cc of nitrobenzene and 25 grams of powdered potassium hydroxide. These were mixed and heated at 90° C for varying times, water was then added, the unchanged nitrobenzene separated and the aqueous layer extracted with ether to remove the nitrophenols. This aqueous layer was then tested for nitrites and nitrates by the usual standard methods of water analysis for nitrites and nitrates. Nitrophenol interfered with the accuracy of the tests but this factor was eliminated through the use of blanks.

Traces of nitrite and nitrate were observed but not in sufficient amounts to be significant. This would indicate that there had been no appreciable hydrolysis of the nitrobenzene by the alkali. It is of interest to note that about half of the nitrobenzene can be recovered as nitrophenol.

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### THE STABILITY OF IRON CARBIDE AND CALCULATIONS OF ITS HEAT OF FORMATION FROM EQUILIBRIUM STUDIES

HAROLD L. MAXWELL AND ANSON HAYES

Values of the equilibrium constants for the system:



have been determined at temperatures of 650°C and 700°C. The value of  $K$  for this reaction at 650°C is 1.66 and at 700°C is 2.21.

Values of the equilibria constants for the system:



as calculated by Juptner and determined experimentally by Boudouard; Thead and Wheeler; and checked in part in this laboratory, are smaller than the values of  $K$  for reaction (1) at the same temperature. This indicates that iron carbide is metastable with respect to carbon and alpha iron at these temperatures. From the values of the equilibrium constants for reaction (1) its heat effect was calculated and corrected to 20°C. This gave the heat of formation of iron carbide as -18,331 and -16,131, assuming