

place as Rollefson and Lindquist suggest, but it would be unwise to infer from any of these experiments that the chlorine atom is unexcited.

Rollefson and Lindquist give only a few details of their experimental conditions, gas pressures, etc., but they mention one significant fact, namely, that their chlorine pressure was always large compared with the pressures of hydrogen and iodine monochloride. In these circumstances the iodine compound in the vapor phase may have been mainly iodine trichloride. This substance is of a lighter color than iodine monochloride, and must have an altogether different absorption spectrum. We have found that the vapors of iodine monochloride and chlorine react very readily, and, indeed, Rollefson and Lindquist themselves admit that they froze out iodine trichloride in the solid phase.

We do not wish to make any further statement until we have completed the study of the reaction kinetics.

PHYSICO-CHEMICAL LABORATORY  
UNIVERSITY OF SYDNEY  
N. S. W., AUSTRALIA

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T. IREDALE  
D. P. MELLOR

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#### CATALYTIC REDUCTION OF MIXTURES OF PARA-NITRO- AND NITROSO-PHENOLS WITH KETONES

*Sir:*

It has recently been shown by the writer that *p-sec.*-alkylaminophenols are formed by the catalytic reduction of mixtures of *p*-nitro- or nitrosophenol and ketones at room temperatures [Major, THIS JOURNAL, 53, 1901 (1931)]. The suggestion was made that the reaction probably involved formation of *p*-hydroxy-phenylhydroxylamine as an active intermediate product of the reduction and that this compound condensed with the ketone and finally that the condensation product was reduced to the alkylaminophenol. Vavon and Krajićinović have reported that they have been able to make the condensation product between benzaldehyde and phenylhydroxylamine,  $C_6H_5CH=N(O)C_6H_5$ , by the catalytic reduction of a mixture of nitrobenzene and benzaldehyde [Vavon and Krajićinović, *Compt. rend.*, 187, 420 (1928)].

However, it was noted that heat was formed during the catalytic reduction of mixtures of *p*-nitro- and nitrosophenol and acetone. There remained, then, the possibility that the heat of the reaction between *p*-nitro- or nitrosophenol and hydrogen was sufficient to cause the *p*-aminophenol which formed to condense with the ketone. This condensation product would then be readily reduced to *p-sec.*-alkylaminophenol.

Evidence which apparently favored this point of view was obtained when a solution of 0.1 mole of *p*-aminophenol in acetone was heated to

the boiling point and then shaken with hydrogen in the usual way in the presence of a platinum oxide catalyst. Hydrogen was absorbed and 50% of the theoretical yield of *p*-isopropylaminophenol was recovered from the reaction mixture.

On the other hand, it was found that a mixture of 0.05 mole of *p*-aminophenol, 3.5 moles of acetone and 0.1 g. of Adams' platinum oxide catalyst did not absorb any more hydrogen than was necessary to reduce the platinum oxide when the temperature of the reaction mixture was kept at 4–5° or at 26–32.5°. The mixture was shaken at 4–5° for one hour and at 26–32.5° for two and three-quarter hours.

It was also found that in one and one-half hours very little *p*-isopropylaminophenol was formed during the reduction of a solution of 0.05 mole of *p*-nitrosophenol in 3.5 moles of acetone in the presence of the platinum oxide catalyst at 4–5°. On the other hand, in one hour at 16–32.5° the catalytic reduction of 0.05 mole of *p*-nitrosophenol in 3.5 moles of acetone gave a 60% yield of *p*-isopropylaminophenol.

It seems fairly evident, then, that while catalytic reduction of a mixture of *p*-aminophenol and acetone at elevated temperatures will produce *p*-isopropylaminophenol, this reduction does not occur to any appreciable extent during a few hours at room temperature. However, catalytic reduction of a mixture of *p*-nitrosophenol and acetone at room temperature does give *p*-isopropylaminophenol. Accordingly, it seems most likely that *p*-hydroxyphenylhydroxylamine is formed as an intermediate during the reduction and that due to its somewhat greater activity than *p*-aminophenol it condenses with the ketone and the condensation product is in turn reduced to *p*-*sec.*-alkylaminophenol.

LABORATORY FOR PURE RESEARCH  
MERCER AND CO., INC.  
RAHWAY, N. J.

RANDOLPH T. MAJOR

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## AN ADSORPTION METHOD FOR THE DETERMINATION OF THE AREA OF A POWDER

*Sir:*

The writers have devised a simple procedure which seems to give information that is more definite with respect to the area of a fine powder than that given by any earlier method.

The powder, titanitic oxide or silicon dioxide, for example, is dried in a high vacuum at as high a temperature as can be used without affecting the area of the crystals. The cool, dry powder is immersed in a solution of oleic acid, butyric acid or some other suitable acid, in very dry benzene, and the suspension is shaken until equilibrium is attained. After the