

the nitrous oxide formed, is $k^* = 387 \times 10^{-5}$ min.⁻¹ (decadic logs) at 35°, in excellent agreement with Baughan and Bell's³ $k^* = 386 \times 10^{-5}$ min.⁻¹ obtained for proto-nitramide in H₂O.

Partially deuterized nitramide prepared from 48% D₂O and sulfur trioxide yielded $k^* = 125.5 \times 10^{-5}$ min.⁻¹ at 24.84° in 0.01 *N* hydrochloric acid in H₂O. La Mer and Greenspan¹ obtained $k^* = 126.6 \times 10^{-5}$ min.⁻¹ under the same conditions for proto-nitramide in H₂O.

II. Direct Exchange.—Proto-nitramide was dissolved in D₂O (temp., approx. 5°). The nitramide produced by exchange was extracted with ether (dried over sodium) by the freezing method of Marlies and La Mer.^{4,5} The exchange and extraction required less than one hour.

To ensure the complete removal of possible traces of deuterio-solvent, the nitramide was kept over phosphorus pentoxide for one week. It was then decomposed by gentle heating in the presence of a trace of anhydrous sodium carbonate. The liberated water was redistilled and the density determined by the falling drop method.⁶

The following results show that the deuterium content of the nitramide approximated the deuterium content of the water from which it had been extracted.

Mole fraction of D in the solvent water in which exchange takes place	0.28	0.87	0.97
Mole fraction of D in the water from the decompn. of the extracted nitramide	.25	.65	.94

There is no point in calculating an exchange constant because, due to the method of extraction, the equilibrium temperature and the equilibrium concentration of D₂O cannot be held constant. Since deuterio-nitramide decomposes in water at the same rate as proto-nitramide and since deuterio-nitramide containing more than one atom of D per molecule may be extracted from a solution of proto-nitramide in D₂O, it is evident that both atoms of hydrogen in nitramide exchange with D₂O before decomposition occurs.

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(3) Baughan and Bell, *Proc. Roy. Soc. (London)*, **A894**, 158, 464-478 (1937).

(4) Marlies and La Mer, *THIS JOURNAL*, **57**, 2008 (1935).

(5) Marlies, La Mer, and Greenspan, "Inorganic Syntheses," McGraw-Hill Book Co., Inc., New York, N. Y., Vol. I, p. 72.

(6) Hochberg and La Mer, *Ind. Eng. Chem., Anal. Ed.*, **9**, 291 (1937).

Ferric Chloride as a Condensing Agent

BY W. M. POTTS AND R. J. DOBSON

A further investigation¹ of anhydrous ferric chloride as a condensing agent in the alkylation of benzene has been carried out using butyl alcohols.

1. Condensation products were not formed with *n*-butyl alcohol and benzene. This agrees with the results reported by Huston and Hsieh² using aluminum chloride and primary alcohols up to and including *n*-hexyl alcohol.

2. *s*-Butyl alcohol gave evidence of a reaction but no product was identified. With aluminum chloride under the same conditions, a 69% yield of *s*-butylbenzene was obtained. Huston and Hsieh² reported a 25-28% yield, while Tzukervanik and Tokareva³ reported a 70% yield. The *s*-butylbenzene was identified by its physical constants and the diacetamino derivative, m. p. 193°.⁴

3. At room temperature, one mole of *t*-butyl alcohol, one mole of ferric chloride and five moles of benzene gave an 82% yield of *t*-butylbenzene. With aluminum chloride under the same conditions a 50% yield of *t*-butylbenzene was obtained. Using two moles of *t*-butyl alcohol, one mole of benzene and one mole of ferric chloride, a 24% yield of mono-*t*-butylbenzene and 64% of 1,4-di-*t*-butylbenzene was obtained. The acetamino derivative of the former melted at 168°.⁴ The latter was identified by the method reported by Potts and Carpenter.¹

Better yields of *t*-butylbenzene were obtained with ferric chloride than with aluminum chloride, but ferric chloride does not effect the condensation of primary and secondary alcohols with benzene. Higher temperatures and larger quantities of ferric chloride lower the yields of *t*-butylbenzene.

(1) Potts and Carpenter, *THIS JOURNAL*, **61**, 663 (1939).

(2) Huston and Hsieh, *ibid.*, **58**, 439 (1936).

(3) Tzukervanik and Tokareva, *J. Gen. Chem. (U. S. S. R.)*, **5**, 764 (1935); *C. A.*, 442 (1936).

(4) Ipatieff and Schmerling, *THIS JOURNAL*, **59**, 1056 (1937).

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α -Hydrindone

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Although ring closures of many β - and γ -aryl butyric acids to the corresponding cyclic ketones have been accomplished successfully by the ac-