there is a fairly sharp end-point of titration just when one equivalent of oxidizing agent is used up. The drawn-out curve is the one calculated for a univalent oxidation curve (index potential, 28.6 mv.), the circles are experimental points. The potentials corresponding to more than 100% oxidation drift much more than those within the titration curve proper. The normal potential under the conditions prevailing in this experiment was +0.900 volt.

The absorption spectrum of the radical (Fig. 2) was determined with a König–Martens spectrophotometer for a solution of the amine in 80% acetic acid containing 1.5 × $10^{-4}$ mole per liter and oxidized with 25% of the equivalent amount of lead tetraacetate. The color is not perfectly stable in time, but the fading is negligible within the period necessary for the readings.

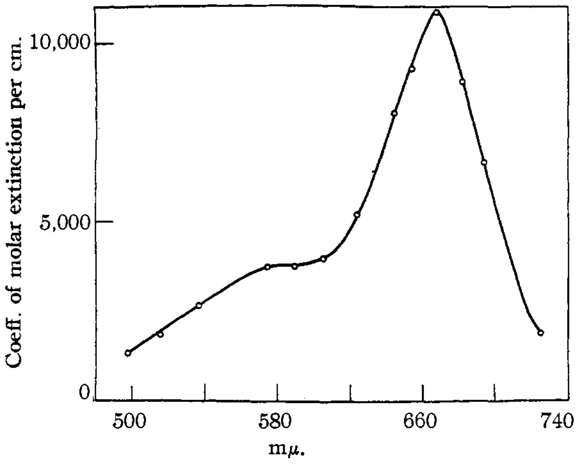

Fig. 2.

Tri-*p*-tolylamine was prepared according to Wieland[1] as a crystalline white powder, m. p. 117°. Lead tetraacetate was prepared according to J. C. Bailar.[4]

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(4) "Inorganic Syntheses," Vol. I, 1939.

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NEW YORK, N. Y.

RECEIVED MAY 29, 1940

## Note on the Grignard Reagent

BY MARTIN KILPATRICK AND EUGENE A. BARR, JR.

In connection with kinetic studies of the reaction of magnesium with alkyl and aryl halides it was noted that the reaction of magnesium[1,2] with bromobenzene gave a black precipitate. This precipitate has been noted in other reactions with magnesium but has not been identified. We assumed the precipitate to be colloidal magnesium, and we have confirmed this assumption by analysis. The black precipitate was washed with ether, weighed, dissolved in hydrochloric acid, precipitated as magnesium ammonium phosphate, and weighed as magnesium pyrophosphate.

| Black ppt., mg. | Magnesium found, mg. |
|---|---|
| 4.45 | 4.35 |
| 7.35 | 7.07 |

The black precipitate has been obtained with magnesium from various sources including the analyzed samples used by Kilpatrick and Rushton.[3]

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(1) Kilpatrick and Simons, *J. Org. Chem.*, **2**, 459 (1937).

(2) Gzemski and Kilpatrick, *ibid.*, **5**, 264 (1940).

(3) Kilpatrick and Rushton, *J. Phys. Chem.*, **38**, 269 (1934).

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RECEIVED MAY 16, 1940

## The Presence of Indole in "Practical" α-Methylnaphthalene

BY M. S. KHARASCH, STEPHEN S. KANE AND HERBERT C. BROWN

The presence of indole in the coal tar fraction of b. p. 220–260° has been demonstrated by Weissgerber.[1] However, to our knowledge no mention is made anywhere that "Practical" α-methylnaphthalene contains about 1–2% of indole. This product was found by us in a study of the action of oxalyl chloride on commercial α-methylnaphthalene.

### Experimental

The practical α-methylnaphthalene obtained from Eastman Kodak Co. is vacuum distilled. The original liquid is dark red with a beautiful blue fluorescence, while the distillate (b. p. 118° (17 mm.)) is light straw yellow but still shows the blue fluorescence. The red color returns on exposure to air. On addition of 2–3 cc. of oxalyl chloride to about 15 cc. of the distilled methylnaphthalene, 0.25 g. of gold yellow precipitate separates and is collected on a filter. The filtrate is distilled and a non-fluorescent, practically colorless methylnaphthalene (b. p. 118° (17 mm.)) is obtained. Exposure to light and air has no effect on the color of pure α-methylnaphthalene, but addition of a trace of indole produced a red color after a short exposure to air.

The yellow precipitate was shown to be the acid chloride of 3-indole glyoxalic acid by comparing its properties and those of its derivatives with the compounds obtained by treatment of pure indole dissolved in pure α-methylnaphthalene or benzene with oxalyl chloride. The same

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(1) Weissgerber, *Ber.*, **43**, 3520 (1910).