mann[5] using 1-bromonaphthalene; (c) utilizing the Grignard reaction[6] with 1-bromonaphthalene. The melting points of the samples of 1,1′-binaphthyl[7] prepared by these procedures were 157.5, 157.2 and 157.5°, respectively; a mixture of the three preparations melted at 157.5°.

In Fig. 1 are presented the ultraviolet absorption spectra for these three samples, which were determined with a Beckman quartz spectrophotometer using 95% ethyl alcohol as solvent, together with the data[8] taken from the Fig. 2 published by Adams and Kirkpatrick.[2] In our Fig. 1 are included also data recorded still earlier by Pestemer and Cecelsky[9] using a hexane solution of 1,1′-binaphthyl. The low wave length absorption band which we report is at 220–224 millimicrons, which is in satisfactory agreement with the band at 226 millimicrons reported by Friedel, *et al.*,[1] but is in disagreement with the location of the absorption band at 238 millimicrons according to Adams and Kirkpatrick.

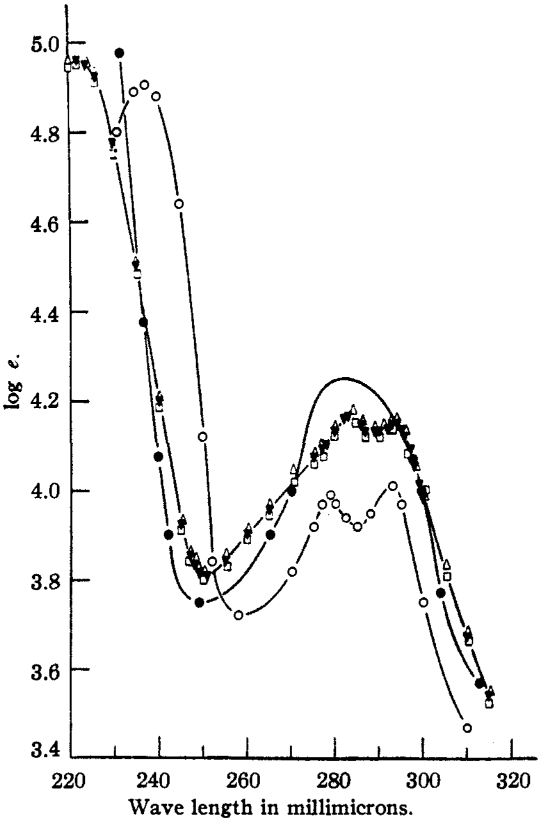

Fig. 1.—Ultraviolet absorption spectra for 1,1′-binaphthyl: ▼, 1,1′-binaphthyl prepared by the Wurtz–Fittig synthesis; △, Ullmann synthesis; □, Grignard synthesis; ○, data of Adams and Kirkpatrick; ●, data of Pestemer and Cecelsky.

2,2′-Binaphthyl,[10] melting at 187°, was synthesized through the Grignard reaction with 2-iodonaphthalene. The absorption spectrum for 2,2′-binaphthyl is presented in Fig. 2. In agreement with the findings of Friedel, *et al.*, a thorough investigation throughout the region 260–300 millimicrons did not yield evidence for the existence of an absorption maximum at 297 millimicrons, as reported by Adams and Kirkpatrick. Otherwise, the absorption spectra for 2,2′-binaphthyl are in reasonable agreement.

Fig. 2.—Ultraviolet absorption spectrum for 2,2′-binaphthyl.

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[5] Ullmann and Bielecki, *Ber.*, **34**, 2184 (1901).

[6] Sakellarios and Kyrimis, *ibid.*, **57**, 324 (1924).

[7] The melting point behavior is considerably influenced by the rate of heating. Samples can be shown to exhibit sintering at temperatures lower than the m. p. of 157.5° by very rapid heating. *Cf.* Orchin and Friedel, THIS JOURNAL, **68**, 573 (1946).

[8] These data were obtained using dioxane as solvent; however, identical data were reported utilizing ethyl alcohol as solvent.

[9] Pestemer and Cecelsky, *Monatsh.*, **59**, 119 (1932).

[10] Vesely and Stursa, *Coll. Czechoslov. Chem. Commun.*, **4**, 139 (1932), reported m. p. of 187° for a sample of 2,2′-binaphthyl obtained using lithium as the coupling agent.

THE COTTON RESEARCH COMMITTEE OF TEXAS
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The Purification of Neopentane by Mercury Photosensitization[1]

BY B. DEB. DARWENT AND E. W. R. STEACIE

In a study of the mercury photosensitized reactions of neopentane it was found necessary to prepare extremely pure samples of that hydrocarbon. The Grignard reaction of tertiary butyl chloride with methylmagnesium chloride was used to produce the neopentane. The product was washed with 85 and 95% sulfuric acid and then photobrominated to ensure the complete removal of unsaturates, since isobutene is the most likely impurity. The residual bromine and any hydrogen bromide formed were removed by distilling the mixture through 40% potassium hydroxide (aqueous) and over solid potassium hydroxide. The resulting neopentane was finally purified by low temperature distillation in a column of conventional design. The distillate was assumed to be pure when the temperature remained constant at 9.6° for thirty to sixty minutes under total reflux. This "pure" neopentane was taken off in four consecutive fractions, the purity of which was checked by determining the vapor pressure–temperature relations of large samples over a wide range of temperature. No indications of impurity could be detected.

Preliminary experiments showed that the rates of the mercury photosensitized reaction of different samples, though surprisingly low, were very variable. Samples of the unreacted and of the "partially decomposed" neopentane were analyzed by the mass spectrometer with the following results: in which Sample A was the "pure" unreacted neopentane and B to F were "partially decomposed" samples. It was stated by the analyst that samples D, E and F were "apparently of the same purity as the neopentane used for calibrating as obtained from Dr. F. D. Rossini, National Bureau of Standards, Standard Sample No. 299–5s. It is certified to be 99.96% pure; impurity 0.04 ± 0.02%."

	Percentages					
Sample	A	B	C	D	E	F
Neopentane	96.4	99.6	99.9	100	100	100
n-Pentane	1.1	..	...	...	...	...
Dimethylcyclopropane	1.6	0.4	trace	...	...	...
C_6 hydrocarbons	1	..	...	...	...	...

Accordingly a large sample of crude (undistilled) neopentane (about 30 liters of gas) was subjected to prolonged mercury sensitization by circulating a mixture of gaseous neopentane and mercury through a quartz vessel illuminated by a low pressure mercury lamp, the radiation from which consisted largely of the unreversed 2537 Å. line, for seventy to eighty hours, roughly distilled into 5 fractions and analyzed on the mass spectrometer. The results were:

	Percentages				
Fraction no.	1	2	3	4	5
Neopentane	99 (8)[a]	99 (7)[a]	99 (6)[a]	99 (4)[a]	99 (0)[a]
Hexane	0.2	0.3	0.4	0.6	1.0

[a] The figures in parentheses are uncertain.

Hence, in the absence of very efficient fractionation, mercury photosensitization is an efficient method of obtaining neopentane of high purity. This method of purification is possible only because neopentane is remarkably resistant to attack by $Hg(^3P_1)$ atoms whereas the impurities react readily to give, ultimately, heavy polymers and non-condensable gases (hydrogen and methane) which are easily separated from the unreacted neopentane.

The Grignard reaction was carried out by Dr. A. Cambron and Mr. R. A. B. Bannard of these laboratories, and the mass spectrometer analyses by Dr. Fred L. Mohler of the National Bureau of Standards, Washington, D. C., to whom our thanks are due. The laboratory assistance of Mr. J. R. Pilon is gratefully acknowledged.

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The Reduction of *p*-Hydroxyformanilide by Lithium Aluminum Hydride to N-Methyl-*p*-aminophenol

BY JACOB EHRLICH

This reduction of an anilide to a secondary amine proceeds smoothly in tetrahydrofuran as solvent. The method is applicable to various amides using diethyl ether or tetrahydrofuran as solvent, to be reported in a later communication.

Experimental

A solution of 1.9 g. (0.05 mole)[1] of lithium aluminum hydride in 36 cc. of anhydrous tetrahydrofuran[2] was prepared in a 3-neck conical flask provided with sealed stirrer, thermometer, reflux condenser, hopper[3] and calcium chloride guard-tube.[4] The solution was brought to 20–25° and 2.75 g. (0.02 mole) of *p*-hydroxyformanilide was added in small portions during thirty minutes, cooling externally with ice-water to maintain this temperature. The hopper was flushed with 4 cc. of tetrahydrofuran and the charge stirred twenty minutes more at 20–25°. Dur-

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[1] Assuming only one labile hydrogen (phenolic) in 1 mole of *p*-hydroxyformamilide, then 0.25 mole of $LiAlH_4$ would be destroyed prior to any reduction. The reduction of the formyl radical would consume a further 0.5 mole of $LiAlH_4$, making a 4:3 molar ratio of anilide:$LiAlH_4$. If the amido hydrogen is also labile, the ratio becomes 1:1. If the formyl hydrogen is labile, the ratio becomes 4:5. However, these calculations are only of academic interest, for the 0.02:0.05 molar ratio was arrived at empirically. If less $LiAlH_4$ is employed, the yield drops proportionally.

[2] The tetrahydrofuran was obtained from E. I. du Pont de Nemours and Co. This was rendered water and peroxide free by storing 500 cc. in an amber bottle over 75 g. of flake sodium hydroxide for two weeks, occasionally shaking. The clear, colorless supernatant liquor was decanted (from now discolored solids) for use in the reductions without further purification. On dissolving the $LiAlH_4$ in the tetrahydrofuran the heat of solution will raise the temperature to about 50°.

[3] Similar in construction (but smaller capacity) to that described by Swift and Billman, *Ind. Eng. Chem., Anal. Ed.*, **17**, 600 (1945). However, this may be omitted and the same results obtained by using a removable rubber stopper.

[4] In various reductions no special precautions, except the exclusion of atmospheric moisture, were employed, and no uncontrolled or explosive reaction was observed. However, as an added precaution, it would be desirable to conduct the reaction under an atmosphere of dry nitrogen.