Zinc Dust Distillation of Dehydro-W-III.—A small distillation apparatus (ice-cooled receiver) was charged with an intimate mixture of 1.16 g. of dehydro-W-III and 5 g. of zinc dust. The mixture was heated cautiously over a free bunsen flame. As reaction proceeded, the heat supplied to the mixture was increased. The distillate was a mixture of a mobile oil and a bright yellow solid (presumably starting material, since this sublimes readily). After 10–15 minutes, distillation ceased. A small portion of the oil was treated with ethanolic picric acid. The yellow precipitate was recrystallized from water to give α-picoline picrate, m.p. 161–162°, alone or mixed with an authentic sample of α-picoline picrate (m.p. 162–163°). The infrared spectra of the two picrate samples were identical. Another sample of the oil was converted to the methiodide, m.p. 224–226° (reported[35] for α-picoline methiodide, m.p. 226–228°).

Hydrolysis of W-I to W-II (XXI).—A suspension of 1.00 g. of W-I in 25 cc. of 5% sodium hydroxide was warmed on the steam-bath until solution was completed (about 1 minute). The mixture was cooled to room temperature, acidified with 5% hydrochloric acid, and the resulting yellow precipitate collected, washed with water and dried in air to yield 0.83 g. of W-II, m.p. 125–127°. Recrystallization from acetone–ligroin afforded light yellow, micaceous platelets, m.p. 126–127°, reported[5] m.p. 127°; ultraviolet (MeOH): $\lambda\lambda_{max}$ 264 (4.06), 332 (3.98); (in 2×10^{-3} *N* methanolic sulfuric acid): $\lambda\lambda_{max}$ 242 (3.86) (shoulder), 330 (3.46), 392 (3.13) (shoulder); (in 2×10^{-3} *M* sodium hydroxide): $\lambda\lambda_{max}$ 259 (4.06), 342 (3.96); infrared (chf.): $\lambda\lambda_{max}$ 5.80 (17), 6.08 (20), 6.45 (20), 6.84 (19), 7.08 (17).

Anal. Calcd. for $C_{13}H_{13}O_4N$: C, 63.15; H, 5.30; N, 5.67. Found: C, 63.33; H, 5.71; N, 5.65.

W-II was soluble in sodium hydroxide. It was partially soluble in sodium bicarbonate, but solution was not accompanied by release of carbon dioxide. Attempts to determine pK_a in 50% methanol (warming was necessary to effect solution) gave results for neutral equivalent varying between 290 and 500, presumably because of some recrystalization. The infrared spectrum in chloroform, Nujol or hexachlorobutadiene mull showed no evidence of free or bonded O–H absorption between 3 and 4 μ.

When a sample of W-II was heated, it melted at 127° and vigorously evolved steam at 135°. As heating at 135° was continued, the mass solidified. After 10 minutes, the product was washed with acetone and dried to give W-I, m.p. 194–197° dec., identical in infrared spectrum with an authentic sample.

Deacetylation of W-II to W-III.—A suspension of 0.40 g. of W-II in 10 cc. of 10% barium hydroxide was warmed on the steam-bath for three hours. The mixture was cooled to room temperature, filtered from a small quantity of insoluble material and then neutralized with 5% hydrochloric acid, whereupon an oil separated. Upon standing overnight at 5°, this material solidified to give 0.15 g. of W-III, m.p. 45–54°. Several recrystallizations from ligroin gave pure W-III as a light yellow solid, m.p. 56–57°; ultraviolet (MeOH): $\lambda\lambda_{max}$ 264 (3.99), 331 (3.83); infrared (chf.): $\lambda\lambda_{max}$ 6.01 (15), 6.06 (15), 6.21 (14), 6.45 (16), 6.66 (14), 6.84 (15), 7.04 (13).

Anal. Calcd. for $C_{11}H_{11}O_3N$: C, 64.38; H, 5.40; N, 6.83. Found: C, 64.10; H, 5.42; N, 6.82.

Hydrogenation of W-I to Dihydro-W-I.—A mixture of 1.96 g. of W-I and 0.10 g. of palladium black in 150 cc. of 95% ethanol was stirred in an atmosphere of hydrogen. One molar equivalent of hydrogen was absorbed in 2 hours, at which time the rate of consumption of gas had decreased appreciably. The hydrogenation was interrupted, the solution filtered and concentrated to a small volume. When cooled, the solution deposited 1.85 g. of a light yellow solid, m.p. 234–238° dec. Recrystallization from 95% ethanol gave colorless needles, m.p. 240–242° dec., reported[5] m.p. 242–243°. The pure substance showed no fluorescence in solution; ultraviolet (MeOH): $\lambda\lambda_{max}$ 243 (4.05), 295 (very broad shoulder) (3.62); infrared (chf.): $\lambda\lambda_{max}$ 5.94 (20), 6.06 (11), 6.18 (5), 6.98 (19), 7.05 (14).

Anal. Calcd. for $C_{13}H_{13}O_3N$: C, 67.52; H, 5.66; N, 6.06. Found: C, 67.42; H, 5.48; N, 5.68.

Hydrolysis of Dihydro-W-I to Dihydro-W-II.—A sample of 0.10 g. of dihydro-W-I dissolved rapidly in 5 cc. of 5% sodium hydroxide. The solution was cooled to 5° in an ice-bath and acidified with 5% hydrochloric acid, whereupon 0.09 g. of a precipitate, m.p. 118–120°, separated. Recrystallization from acetone gave almost colorless crystals, m.p. 120–120.5°; ultraviolet (MeOH): $\lambda\lambda_{max}$ 268 (4.12), 275 (4.12), 291 (4.14); infrared (chf.): $\lambda\lambda_{max}$ 5.81 (12), 6.05 (14), 6.44 (17), 6.80 (15), 7.05 (11).

Anal. Calcd. for $C_{13}H_{15}O_4N$: C, 62.64; H, 6.07; N, 5.62. Found: C, 62.50; H, 5.79; N, 5.49.

Dihydro-W-II was re-cyclized to dihydro-W-I by heating at 140°. The product had m.p. 234–237° and its infrared spectrum was identical with that of authentic dihydro-W-I.

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(35) H. O. Jones, *J. Chem. Soc.*, **83**, 1415 (1903).

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, UNIVERSITY OF MISSOURI]

Some Reactions of N-Acetylphthalimides

BY NORMAN RABJOHN, M. F. DRUMM[1,2] AND R. L. ELLIOTT

RECEIVED JULY 18, 1955

It has been shown that N-acetylphthalimide and substituted N-acetylphthalimides can acetylate butyl alcohol, in the presence of sodium butoxide, with the formation of a phthalimide or the sodium salt of a phthalimide. N-Acetylphthalimide acetylates secondary amines, but reacts with primary amines to afford N-alkyl- or arylphthalimides and acetamide. Phenylmagnesium bromide has been found to attack N-acetylphthalimide at the three carbonyl groups to yield 3-hydroxy-3-phenylphthalimidine, *o*-dibenzoylbenzene and diphenylmethylcarbinol. The reduction of N-acetylphthalimide, by means of lithium aluminum hydride, has been shown to give 3-ethylisoindoline.

The chemistry of the N-acylimides has been studied only to a limited extent. Hurd and co-workers[3] have shown that certain N-acylphthalimides of the general formula $C_6H_4(CO)_2N{-}COCH_2R$ give rise on pyrolysis to phthalimide, the acid RCH_2CO_2H, or its anhydride and the ketone $(RCH_2)_2CO$. Isobutyryl- and diphenylacetylphthalimide, in contrast, pyrolyze satisfactorily to ketenes. It has been reported also,[4] that the molecular weights of polymers which contain hydroxyl groups may be increased by heating the polymers with an N-acylpolyimide of a polycarboxylic acid. The purpose of the present work was to extend the study of the reactions of N-acetyl- and substituted N-acetylphthalimides.

It appeared to be of interest, first, to examine the reaction of N-acetylphthalimide with a representative alcohol. It was found that N-acetylphthal-

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[1] Abstracted in part from the Ph.D. thesis of M. F. Drumm.

[2] E. I. du Pont Fellow, 1949–1950.

[3] C. D. Hurd, M. F. Dull and J. W. Williams, THIS JOURNAL, **57**, 774 (1935).

[4] P. J. Flory, U. S. Patent 2,594,145; *C. A.*, **46**, 6431 (1952).

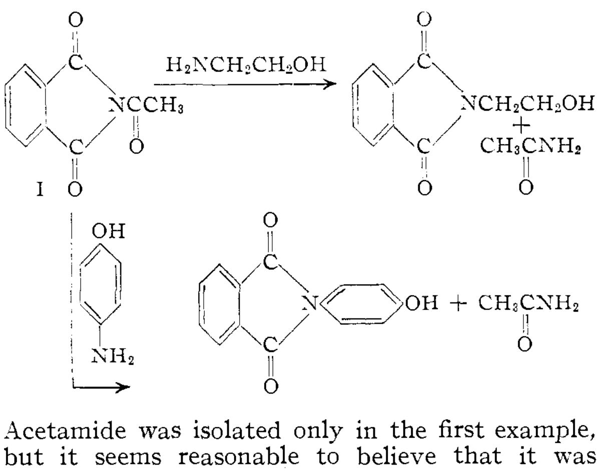
imide acetylates butyl alcohol readily in the presence of sodium butoxide, to give butyl acetate and phthalimide. The study was extended then to observe any effects that various nuclear substituents in N-acetylphthalimides might have upon the acetylation reaction.

When the substituted N-acetylphthalimides listed in Table I were allowed to react at room temperature with a sodium butoxide solution, butyl acetate resulted in conversions which varied from over 90% with N-acetylphthalimide and the 3- and 4-methyl derivatives to about 30% for the 3- and 4-bromo- and the 3- and 4-nitro-N-acetylphthalimides. Since this order appeared to be the inverse of that expected, the reaction mixtures from the condensation of 4-methyl-N-acetylphthalimide and 4-nitro-N-acetylphthalimide with butyl alcohol were examined in more detail. In addition to the ester, 4-methylphthalimide was isolated in the first case, while in the latter instance there was obtained the sodium salt of 4-nitrophthalimide. It was observed also that the amount of sodium salt produced was related directly to the quantity of sodium butoxide employed in the reaction.

This observation suggested that the sodium salts of the imides may be intermediates in the acetylation reaction. That this apparently is true was shown by experiments in which sodium phthalimide and sodium 4-methylphthalimide were found to be effective agents for the acetylation of butyl alcohol by the corresponding N-acetylphthalimides. The sodium salt of 4-nitrophthalimide, however, failed to cause any appreciable reaction between 4-nitro-N-acetylphthalimide and butyl alcohol.

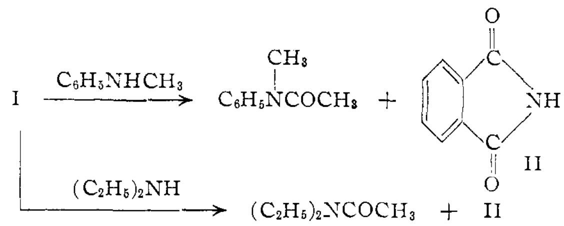
N-Acetylphthalimide appeared to have no action on *t*-butyl alcohol which contained a small amount of the corresponding sodium alkoxide.

When N-acetylphthalimide was caused to react with compounds which contain both an hydroxyl and an amino group, the results were somewhat unexpected.



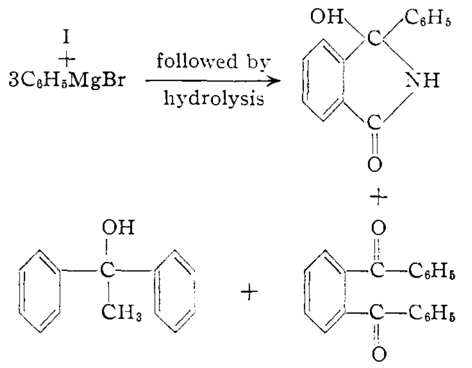
Acetamide was isolated only in the first example, but it seems reasonable to believe that it was formed also in the second case because of the similar nature of the other products.

Aniline was found to condense in the same fashion as *p*-aminophenol to produce the expected N-phenylphthalimide. However, the secondary amines, N-methylaniline and diethylamine underwent simple acetylation.



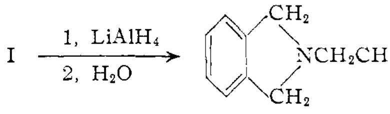
The results of the reactions of the N-acetylphthalimides with alcohols and amines suggest that in these cases there is an initial attack by a nucleophilic agent on a ring carbonyl group of the N-acetylphthalimide.

A brief investigation of N-acetylphthalimide in the Grignard "machine" indicated the presence of some active hydrogen, depending upon the time of heating, and showed that the molecule can add up to three moles of methylmagnesium iodide. Accordingly, the acylimide was treated with a little over three moles of phenylmagnesium bromide and a mixture of products was obtained.



The 3-hydroxy-3-phenylphthalimidine represented the major portion of the reaction product and the *o*-dibenzoylbenzene was obtained in the least amount. The formation of each of these compounds can be rationalized on the basis of the addition of the Grignard reagent to one or both of the ring carbonyls, as well as the acetyl group, followed by cleavage and hydrolysis of the adducts. Although acetophenone was not isolated, it apparently was an intermediate which combined with phenylmagnesium bromide to produce the diphenylmethylcarbinol.

The reaction of N-acetylphthalimide with lithium aluminum hydride involved again all three carbonyl groups, and the acylimide was reduced to 2-ethylisoindoline in quite good yields.



Experimental[5]

Preparation of N-Acetylphthalimides.—The N-acetylphthalimides were obtained from the corresponding phthalimides by acetylation with acetic anhydride according to the method of Aschan.[6] The phthalimides were obtained from the properly substituted phthalic acids or anhydrides.

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[5] All melting points are uncorrected. The semi-micro carbon and hydrogen analyses were carried out by J. S. Finney and R. L. E.

[6] O. Aschan, *Ber.*, **19**, 1398 (1886).

TABLE I

N-ACETYLPHTHALIMIDES AND RELATED MATERIALS

R	Melting points, °C. Anhydride or acid	Melting points, °C. Imide	Melting points, °C. N-Acetylimide	Formula	Analysis for N-acetylphthalimides, % Carbon Calcd.	Carbon Found	Hydrogen Calcd.	Hydrogen Found
Unsubstd.			134–135[i]					
3-CH_3	110–113[a]	181–184[d]	127–128	$C_{11}H_9O_3N$	65.02	64.87	4.46	4.73
4-CH_3	88–94[a]	192–194[e]	150–152	$C_{11}H_9O_3N$	65.02	64.91	4.46	4.62
3-NO_2	214–216 (acid)[b]	214–215[f]	138–140	$C_{10}H_6O_5N_2$	51.29	51.41	2.58	2.81
4-NO_2		197–199[g]	136–138	$C_{10}H_6O_5N_2$	51.29	50.95	2.58	2.71
3-Br		258–260[h]	129–131	$C_{10}H_6O_3NBr$	44.80	44.58	2.26	2.20
4-Br	160–163 (acid)[c]	227–229[c]	139–141	$C_{10}H_6O_3NBr$	44.80	45.01	2.26	2.45

Methods of preparation: [a] Refs. 7, 9. [b] P. J. Culhane and G. E. Woodward, "Organic Syntheses," Coll. Vol. I, John Wiley and Sons, Inc., New York, N. Y., p. 408. [c] H. Waldman, *J. prakt. Chem.*, **126**, 65 (1930). [d] By thermal decomposition of the half ammonia salt; Young, *Ber.*, **25**, 2102 (1892), reported m.p. 183–184°. [e] From half ammonium salt; M. Hayashi, S. Tsuruoka, I. Morikawa and H. Namikwaw, *Bull. Chem. Soc., Japan*, 11, 184 (1936), reported m.p. 194–195°. [f] P. P. T. Sah and T. S. Ma, *Ber.*, **65**, 1630 (1932). [g] E. H. Huntress and R. L. Shriner, "Organic Syntheses," Coll. Vol. II, John Wiley and Sons, Inc., New York, N. Y., p. 459. [h] H. D. K. Drew and R. F. Garwood, *J. Chem. Soc.*, 836 (1939). [i] Ref. 6.

The procedures employed for the synthesis of these compounds, with the exception of 4-methylphthalic anhydride, were the same as those recorded in the literature. The pertinent information in regard to the compounds is summarized in Table I.

Preparation of 4-Methylphthalic Anhydride.—The conditions employed for the Diels–Alder reaction were similar to those used by Frank, Emmick and Johnson[7] to prepare 3-methyl-1,2,3,6-tetrahydrophthalic anhydride. From 37.5 (0.382 mole) of maleic anhydride, 50 mg. of picric acid in 125 ml. of benzene and 26 g. (0.383 mole) of isoprene 50.9 g. (80%) of 4-methyl-1,2,3,6-tetrahydrophthalic anhydride was obtained, m.p. 64–65°.[8] The latter material was dehydrogenated to 4-methylphthalic anhydride in 72% yield by means of the procedure published by Newman and McCleary[9] for the synthesis of 3-methylphthalic anhydride.

The Reaction of N-Acetylphthalimide with Butyl Alcohol.—N-Acetylphthalimide was heated with butyl alcohol, (a) in the presence of sodium butoxide, (b) in the presence of *p*-toluenesulfonic acid and (c) without any added agent. A perceptible amount of butyl acetate was formed only when the basic reagent was used. In a trial experiment, a mixture of 20 g. (0.106 mole) of N-acetylphthalimide, 7.78 g. (0.105 mole) of butyl alcohol, in which had been dissolved 0.05 g. (0.002 g. atom) of clean sodium, and 300 ml. of decalin was heated on a steam-bath for 1 hour. Fractionation of the reaction mixture gave *n*-butyl acetate in about 30% yield; phthalimide was recovered from the residue in a yield of 75%. In another experiment in which excess butyl alcohol was used as the solvent and the reaction mixture was allowed to stand for 24 hours, phthalimide was isolated almost quantitatively.

The Reaction of Substituted N-Acetylphthalimides with Butyl Alcohol.—The reaction mixtures consisted of 0.0171 mole of the acetylating agent (the N-acetylphthalimides listed in Table I) and 10 ml. of a sodium butoxide solution which had been prepared by dissolving 1.00 g. (0.0435 g. atom) of clean sodium in 100 ml. of anhydrous butyl alcohol. They were allowed to stand for 2 hours at room temperature with occasional shaking, and then were distilled to dryness. The amounts of butyl acetate in the distillates were determined by saponification with standard alkali. The following results were obtained: 4-methyl- (96%), 3-methyl- (92%), 4-bromo- (35%), 3-bromo- (33%), 4-nitro- (30%), 3-nitro- (28%) and N-acetylphthalimide (86%). The analytical procedure employed was shown to account for 95–98% of the ester in blank mixtures of butyl acetate, butyl alcohol and phthalimide.

Reaction of 4-Methyl-N-acetylphthalimide with Butyl Alcohol.—A solution of 3.27 g. (0.015 mole) of 4-methyl-N-acetylphthalimide in 25 ml. of butyl alcohol, in which 0.037 g. (0.0016 g. atom) of sodium had been dissolved, was allowed to stand at room temperature for 24 hours. Crystal formation was noticed after about 8 hours. The mixture was cooled in an ice-bath and filtered. There was obtained 1.5 g. of 3-methylphthalimide which melted at 191–193° after one crystallization from benzene. The original filtrate yielded a further 1.0 g. of material, m.p. 188–192°. A mixture of 10.2 g. (0.05 mole) of 4-methyl-N-acetylphthalimide, 10 ml. of butyl alcohol, 0.92 g. (0.005 mole) of sodium 4-methylphthalimide and 50 ml. of decalin was allowed to stand at room temperature for 24 hours. The precipitate was collected by filtration and recrystallized from benzene to give 3.8 g. of 4-methylphthalimide, m.p. 190–193°. The mother liquor was distilled on a Podbielniak high temperature analyzer column to yield 4.6 g. (79%) of butyl acetate, n^{20}_{D} 1.3958.

Reaction of 4-Nitro-N-acetylphthalimide with Butyl Alcohol.—To a solution of 0.46 g. (0.02 g. atom) of sodium in 50 ml. of butyl alcohol was added 4.68 g. (0.02 mole) of 4-nitro-N-acetylphthalimide. The mixture became warm and within a few minutes a white solid started to separate from solution. It was cooled to room temperature and allowed to stand for 24 hours. The solid was removed by filtration, washed with ether and dried. It weighed 4.1 g. and did not melt up to 280°. A portion of the salt was dissolved in water and acidified with dilute hydrochloric acid. The resulting precipitate was dried and found to melt at 196–198°, a mixed melting point with a known sample of 4-nitrophthalimide gave a value of 196–198°.

A mixture of 4.68 g. (0.02 mole) of 4-nitro-N-acetylphthalimide, 2.14 g. (0.01 mole) of sodium 4-nitrophthalimide and 50 ml. of butyl alcohol was allowed to stand at room temperature for 24 hours. There were no indications that any reaction had occurred. The liquid phase was removed by distillation at room temperature by means of a vacuum pump and the distillate was collected in two traps which were cooled in Dry Ice–acetone. Saponification of aliquots of the distillate gave values which corresponded to about an 8% yield of butyl acetate. The residue in the flask was washed repeatedly with dry benzene and filtered. There was obtained 2.4 g. of material which did not melt. Evaporation of the benzene extract left 4.3 g. of material which contained about 80% of 4-nitro-N-acetylphthalimide on the basis of acetyl determinations.

Attempted Acetylation of *t*-Butyl Alcohol.—A mixture of 29.3 g. (0.155 mole) of N-acetylphthalimide, 11.5 g. (0.155 mole) of *t*-butyl alcohol to which 0.18 g. (0.0078 g. atom) of clean sodium had been added, and 75 ml. of anhydrous benzene was heated under reflux for 8 hours. There was recovered 28.9 g. (98%) of N-acetylphthalimide, m.p. 126–134°.

Reaction of N-Acetylphthalimide with 2-Aminoethanol.—To a solution of 10 g. (0.053 mole) of N-acetylphthalimide in 50 ml. of dioxane was added 3.23 g. (0.053 mole) of 2-aminoethanol. The mixture became warm, and was allowed to stand at room temperature for 24 hours. The solvent was evaporated at room temperature and the residue was taken up in hot water. The solid which separated upon cooling was recrystallized from ethanol to give 8.3 g. (82%) of N-(2-hydroxyethyl)-phthalimide, m.p. 125–127°. A mixed melting point of the material with a sample of N-

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[7] R. L. Frank, R. D. Emmick and R. S. Johnson, THIS JOURNAL, **69**, 2313 (1947).

[8] E. H. Farmer and F. L. Warren, *J. Chem. Soc.*, 3221 (1931), have prepared this compound by the condensation of isoprene with maleic anhydride in a sealed tube; m.p. 63–64°.

[9] M. S. Newman and C. D. McCleary, THIS JOURNAL, **63**, 1542 (1941).

(2-hydroxyethyl)-phthalimide, prepared from phthalic anhydride and 2-aminoethanol,[10] was not depressed.

The aqueous filtrate, from the mixture from which the hydroxyethylphthalimide had been removed, was concentrated by distillation and the residue was crystallized from benzene–ethyl acetate (3:1). There was obtained 0.55 g. of acetamide which melted at 77–81°, and which did not depress the melting point of an authentic sample of acetamide.

Reaction of N-Acetylphthalimide with *p*-Aminophenol.—A mixture of 10 g. (0.053 mole) of N-acetylphthalimide, 7.4 g. (0.069 mole) of *p*-aminophenol and 50 ml. of dioxane was refluxed for 4 hours. The solvent was evaporated to a low volume and the resulting solid was removed by filtration. It was recrystallized from ethanol and there resulted 9.1 g. (72%) of a product which melted at 297–302°. A mixed melting point of this product with N-(*p*-hydroxyphenyl)-phthalimide, obtained from the reaction between phthalic anhydride and *p*-aminophenol,[11] was not depressed.

Reaction of N-Acetylphthalimide with Aniline.—A mixture of 10 g. (0.053 mole) of N-acetylphthalimide, 4.93 g. (0.053 mole) of aniline and 50 ml. of benzene was refluxed for 2 hours. The solid which separated on cooling was removed by filtration and recrystallized from alcohol. There was obtained 9.5 g. (80%) of a white crystalline solid which melted at 204–207°. It did not depress the melting point of an authentic sample of N-phenylphthalimide which was prepared from phthalic anhydride and aniline.[12]

Reaction of N-Acetylphthalimide with N-Methylaniline.—A solution of 4.37 g. (0.023 mole) of N-acetylphthalimide and 2.46 g. (0.023 mole) of N-methylaniline in 25 ml. of benzene was heated under reflux for 8 hours. The reaction mixture was cooled and the solid which formed was collected by filtration. It was recrystallized from benzene to give 1.8 g. (53%) of phthalimide which melted at 234–237°.

The mother liquor from the original filtration was evaporated to dryness, taken up in ether and shaken with dilute hydrochloric acid. From the ether layer there was obtained 1.2 g. of material which melted at 95–99°. The melting point of N-methylacetanilide is recorded as 102°.[13]

Reaction of N-Acetylphthalimide with Diethylamine.—A solution of 5 g. (0.026 mole) of N-acetylphthalimide and 1.94 g. (0.027 mole) of diethylamine in 25 ml. of benzene was refluxed for 4 hours. Upon cooling the reaction mixture, there precipitated 3.56 g. (91%) of phthalimide, m.p. 230–234°. The mother liquor was distilled under reduced pressure and 1.5 g. (50%) of material was obtained which boiled at 74–75° (20 mm.), $n^{20}\mathrm{D}$ 1.4377, b.p. 182–184°. In the literature[14] are listed the following physical properties for N,N-diethylacetamide: b.p. 185–186°, b.p. 93–100° (40 mm.), $n^{20}\mathrm{D}$ 1.4310.

Reaction of N-Acetylphthalimide with Phenylmagnesium Bromide.—A Grignard reagent was prepared from 12 g. (0.5 g. atom) of magnesium, 80 g. (0.5 mole) of bromobenzene and 200 ml. of anhydrous ether. It was cooled to 0° and 28.5 g. (0.15 mole) of N-acetylphthalimide was added slowly with stirring. The reaction mixture was allowed to come to room temperature, was stirred for an additional hour and was treated with an ice-cold solution of 50 g. of ammonium chloride in 150 ml. of water. The ether layer was separated and the water solution was extracted twice with 50-ml. portions of ether. The ethereal solutions were combined, dried over anhydrous sodium sulfate and concentrated to a volume of about 25 ml. The white solid which precipitated (16 g.) was removed by filtration and was recrystallized from benzene; m.p. 162–163°. A sample was hydrolyzed by means of 20% sodium hydroxide and *o*-benzoylbenzoic acid resulted, m.p. 125–127°. Analytical data were in agreement with those of 3-hydroxy-3-phenylphthalimidine which has been reported to melt at 165°.[15]

The mother liquor from the above filtration was concentrated to produce an oil which solidified when cooled. The resulting solid was washed with methanol and recrystallized several times from ethanol to give 2.0 g. of *o*-dibenzoylbenzene, m.p. 145–146°, lit.[16] m.p. 145–146°. A diphenylhydrazone was prepared in the usual manner; m.p. 165–166°, lit.[17] m.p. 165°.

The filtrate obtained from the isolation of the *o*-dibenzoylbenzene was concentrated to leave a white solid which upon repeated recrystallizations from a mixture of benzene–petroleum ether (b.p. 60–70°) gave 5 g. of diphenylmethylcarbinol, m.p. 79–80°. A mixed melting point with an authentic sample of diphenylmethylcarbinol was not depressed.

Reduction of N-Acetylphthalimide.—A solution of 7.6 g. (0.2 mole) of lithium aluminum hydride in 300 ml. of anhydrous tetrahydrofuran was cooled to 10° and 18.9 g. (0.1 mole) of N-acetylphthalimide was added slowly. The reaction mixture was allowed to warm to room temperature and then was heated at reflux for 1 hour. It was cooled in an ice-bath and 250 ml. of 30% sulfuric acid was added slowly with stirring. The mixture was made strongly basic with solid sodium hydroxide, and was steam distilled. The distillate, about 1.5 l., was made strongly basic and extracted with ether. The extract was dried, the ether was removed and the residue was distilled. There was obtained 9.5 g. (64%) of 2-ethylisoindoline which boiled at 219–220°; lit.[18] b.p. 219–221°. The platinum double salt of the amine was prepared, m.p. 187–190°, lit.[18] m.p. 192°.

COLUMBIA, MISSOURI

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(10) H. Wenker, THIS JOURNAL, **59**, 422 (1937).

(11) G. Wanag and A. Veinbergs, *Ber.*, **75**, 1558 (1942).

(12) B. A. Poraĭ-Koshits, *J. Gen. Chem.* (*U.S.S.R.*), **7**, 604 (1937); *C. A.*, **31**, 5787 (1937).

(13) R. L. Shriner and R. F. Fuson, "The Systematic Identification of Organic Compounds," John Wiley and Sons, Inc., New York, N. Y., 3rd ed., p. 235.

(14) S. M. McElvain and B. E. Tate, THIS JOURNAL, **67**, 202 (1945); O. Wallach, *Ann.*, **214**, 235 (1882).

(15) C. Graebe and F. Ullmann, *Ann.*, **291**, 8 (1896).

(16) E. P. Kohler, *Am. Chem. J.*, **40**, 217 (1908).

(17) A. Guyot and J. Catel, *Bull. soc. chim.*, **35**, 1124 (1906).

(18) M. Scholtz, *Ber.*, **31**, 1700 (1898).