(−)-**4-Methylpulegone** (III).—The procedure of Conia[3a] was followed. From (+)-pulegone (154 g., 1.0 mole), methyl iodide (200 g., 1.4 moles), 1.9 *N* sodium *t*-amylate (550 ml.), and diethyl ether (750 ml.), there was obtained 120.5 g. (71.5%) of a methylisopulegone fraction of b.p. 72–74° (2.0 mm.), $n^{27}\mathrm{D}$ 1.4738–1.4741, $\lambda_{\max}^{\mathrm{EtOH}}$ 253 mμ, log ε 3.44. A GLC analysis on the SAIB column showed three bands, A (12.3%), B (51.4%), and C (36.3%) in order of elution.

Treatment of this material with semicarbazide reagent[12] gave a semicarbazone of m.p. 201–205°, which after one recrystallization from ethanol melted at 204–205°. The semicarbazone of (−)-4-methylisopulegone is reported[3a] to have m.p. 203–205°. The steam distillation of the semicarbazone in the presence of 5% aqueous hydrochloric acid gave (−)-4-methylisopulegone (III) of b.p. 60° (1.0 mm.), $n^{20}\mathrm{D}$ 1.4698, $[\alpha]^{23}\mathrm{D}$ −116.5°, lit.[3a] reports b.p. 86–88° (8 mm.) $n^{19}\mathrm{D}$ 1.4711, $[\alpha]^{20}$ 578 −118.88°.

The retention time of III was found to be identical with component B in the original ketone mixture.

Component C matched the retention time of (+)-pulegone. The % pulegone calculated from the ultraviolet value (log ε 3.44) was 37.5%. This is in excellent agreement with the 36.3% calculated from the GLC data.

Component A failed to match the retention time of isopulegone.

4-Methylisopulegol (IV).—A solution of 4-methylisopulegone (7.0 g., 0.042 mole) in diethyl ether (50 ml.) was added dropwise to a slurry of lithium aluminum hydride (4.0 g., 0.105 mole) in diethyl ether (100 ml.). After hydrolysis with 10% sodium hydroxide there was obtained 6.3 g. (88%) of 4-methylisopulegols, b.p. 58–61° (1 mm.), $n^{20}\mathrm{D}$ 1.4790, infrared spectrum, 2.96, 6.11, and 11.27 μ. A gas-liquid chromatograph on Hyprose gave two bands, 43 and 57%, in order of elution. These isomers presumably correspond to the axial (43%) and equatorial (57%) hydroxyl isomers.

Anal. Calcd. for $C_{11}H_{20}O$: C, 78.51; H, 11.98. Found: C, 78.39; H, 11.92.

(+)-**3-Methyl-β-citronellal** (V).—An 18-in. Pyrex tube (0.75 in. i.d.) was packed loosely to a depth of 12 in. with 0.25-in. o.d. Pyrex-helices. The tube was placed in a vertically mounted electric muffle furnace so that all the packed area was in the heating zone. The system was heated so that the outer surface of the glass tube was maintained at 490–500°. 4-Methylisopulegol (28 g.) in the presence of a slow stream of nitrogen was dropped onto the heated glass helices. The pale yellow pyrolysate (24.5 g., 87.5% recovery) was collected in an ice-cooled receiver. Infrared analysis of this material showed the presence of some alcohol. To remove this the mixture was treated with excess isobutyl borate and a mixture of isobutanyl alcohol, isobutyl borate, and V was distilled. The distillate was treated with 10% sodium bicarbonate and extracted with hexane. The hexane layer was dried and distilled. There was obtained 18.5 g. (65%) of (+)-3-methyl-β-citronellal, b.p. 64–66° (2 mm.), $n^{20}\mathrm{D}$ 1.4564, $[\alpha]^{22}\mathrm{D}$ +10.62° (neat. 1 dm.), infrared 3.69, 5.81, and 7.25 μ. A GLC spectrum on SAIB showed the material to be better than 98.5% pure. The literature[1] reports for the DL-isomer, b.p. 63–64° (1 mm.), d^{20}_{4} 0.8261, $n^{20}\mathrm{D}$ 1.4549.

Anal. Calcd. for $C_{11}H_{20}O$: C, 78.51; H, 11.98. Found: C, 78.47; H, 11.87.

Acknowledgment.—The author wishes to thank Mr. Aaron Kossoy and his associates for GLC determinations.

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[12] R. L. Shriner and R. C. Fuson, "Identification of Organic Compounds," 3rd ed., J. Wiley & Sons, Inc., New York, N. Y., 1900.

Preparation of Some Bis(nitrophenyl) Ethers, Bis(aminophenyl) Ethers, and Some Derived Azo Compounds[1]

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Contribution from the American Cyanamid Co., Organic Chemicals Division, Bound Brook Laboratories, Bound Brook, New Jersey

Received June 18, 1962

As part of a program to prepare unusual azo compounds, we decided to investigate the chemistry of bis(2-aminophenyl) ether (III). The reaction between 2-nitrochlorobenzene and potassium 2-nitrophenoxide to produce bis(2-nitrophenyl) ether (I), a compound which may be easily reduced to the diamine, has been known for many years.[2–4] However, yields were either very low (<6%)[2,3], or the procedure unspecified[4] (23% yield).

We found that a 72% yield of I was obtained when equimolar quantities of reactants were heated at reflux overnight in dimethylformamide. Dimethylacetamide and dimethyl sulfoxide were similarly effective as reaction solvents. The use of dimethylformamide as solvent for preparing the other bis(nitrophenyl) ethers was investigated. Table I shows the results of allowing the potassium salt of the nitrophenol in the first column to react with the nitrochlorobenzene of the second column to give the bis(nitrophenyl) ether in the yield shown. The last column gives reported yields.

TABLE I

PREPARATION OF BIS(NITROPHENYL) ETHERS

Nitrophenoxide	Nitrochlorobenzene	Reaction time (hrs.)	Yields, % Found[a]	Yields, % Reported
o	*o*	18	72,[b] 33,[c] 65[d]	23[f]
p	*o*	4.5	61	30[g]
p	*p*	16	79	85[g]
m	*p*	1	73	87[h]
m	*o*	5	60	..
m	*m*	18	0[e]	23[i]

[a] After one recrystallization. [b] Dimethylformamide. [c] Dimethyl sulfoxide. [d] Dimethylacetamide. [e] A reduction product, 3,3′-dichloroazobenzene, was isolated. [f] See ref. 4. [g] M. J. Rarick, R. Q. Brewster, and F. B. Dains, *J. Am. Chem. Soc.*, **55**, 1289 (1933). [h] R. Q. Brewster and F. Strain, *Ibid.*, **56**, 117 (1934). [i] M. Julia and G. Tchernoff, *Bull. soc. chim. (France)*, 546 (1952).

When potassium 3-nitrophenoxide was allowed to react with 3-nitrochlorobenzene, none of the dinitrodiphenyl ether was obtained. A small quantity of an orange material was isolated which proved to

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[1] Presented in part at the Metropolitan Regional Meeting, New York and North Jersey Section, American Chemical Society, New York, N. Y., January 22, 1962.

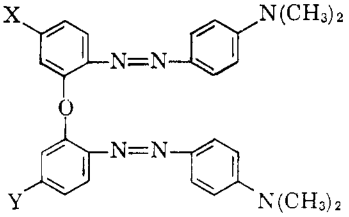
[2] C. Haeussermann and E. Bauer, *Ber.*, **29**, 2083 (1896).

[3] N. M. Cullinane, H. G. Davey, and H. J. H. Padfield, *J. Chem. Soc.*, 719 (1934).

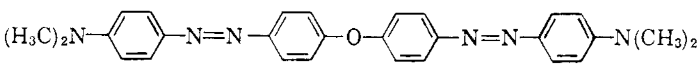
[4] N. L. Allinger and G. A. Youngdale, *J. Am. Chem. Soc.*, **84**, 1020 (1962).

be 3,3'-dichloroazobenzene.[5] This compound could have come only from reduction of the 3-nitrochlorobenzene, and the only possible reducing agent is dimethylformamide. There is no precedent for such a reduction in the literature. We hope to do further work on this reaction.

The reaction to give dinitrophenyl ethers does have certain limitations. The sodium phenoxide may not be substituted for the potassium salt and the potassium salt may not be prepared *in situ* from potassium hydroxide and the phenol, but must be prepared and dried before use.



VII. X = Y = H
IX. X = Y = NO_2
X. X = *p*-Nitrophenylaze
Y = H

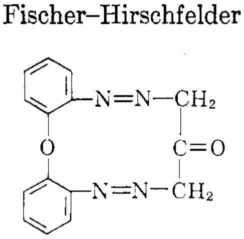


VIII

The reduction of I to III and of bis(4-nitro phenyl) ether to bis(4-aminophenyl) ether (V) in methanol with 10% palladium-on-charcoal catalyst goes in high yield. When acetic acid–acetic anhydride solvent is substituted for methanol in the reduction of I, a good yield of bis(2-acetamidophenyl) ether is obtained.

A standard method for nitrating acetanilide[9] applied to the bis(2-acetamidophenyl) ether gave bis(2-acetamido-5-nitrophenyl) ether which, when treated with refluxing concentrated hydrochloric acid, gave bis(2-amino-5-nitrophenyl) ether (VI).

It is known that two equivalents of diazonium salt react with acetonedicarboxylic acid to give a bisarylazo acetone.[7] The bis(2-aminophenyl) ether could be easily tetrazotized in dilute hydrochloric acid solution, but when allowed to react with acetone dicarboxylic acid it gave only polymeric material, no IV. Fischer–Hirschfelder models show IV to be free of strain, indicating that it could have formed. High dilution techniques were not attempted. Other reactions of III with various difunctional compounds are being investigated.



IV

VII, VIII, IX, and X are typical disazo compounds derived from the diamines III, V, and VI. Other derivatives, differing only in that other coupling components were substituted for dimethylaniline, are described in the experimental section. X was prepared by allowing *p*-nitrobenzene diazonium chloride to couple into III, then tetrazotizing the monoarylazo diamine, and allowing it to couple with dimethylaniline.

Table II gives the absorption maxima and molar extinction coefficients for the azo derivatives having a dimethylaniline moiety and for the monoazo compounds which were prepared for comparison purposes.

TABLE II

VISUAL SPECTRAL DATA FOR DIMETHYLANILINE AZOS

Compound	λ_{max} (mμ)	ε
p-(*p*-Methoxyphenylazo)-*N*,*N*-dimethylaniline[a] (XIII)	408	27,400
p-(*p*-Phenoxyphenylazo)-*N*,*N*-dimethylaniline[b]	415	29,200
4,4'-[Oxybis(*p*-phenyleneazo)]bis(*N*,*N*-dimethylaniline) (VIII)	420	39,700
p-(*o*-Methoxyphenylazo)-*N*,*N*-dimethylaniline (XI)	418	25,100
p-(*o*-Phenoxyphenylazo)-*N*,*N*-dimethylaniline (XIV)	425	26,800
4,4'-[Oxybis(*o*-phenyleneazo)]bis(*N*,*N*-dimethylaniline) (VII)	422	47,900
p-(2-Methoxy-4-nitrophenylazo)-*N*,*N*-dimethylaniline[c] (XII)	493	30,000
4,4'-[Oxybis(4-nitro-*o*-phenyleneazo)]bis-(*N*,*N*-dimethylaniline) (IX)	468	54,300
p-{2-[*o*-(*p*-Dimethylamino)phenylazophenoxy]-4-(*p*-nitrophenylazo)phenylazo}-*N*,*N*-dimethylaniline (X)	472	60,900

[a] E. Sawicki and D. Gerber, *J. Org. Chem.*, **21**, 410 (1956). [b] A. Mailhe, *Comp. rend.*, **154**, 1241 (1912). [c] J. T. Hewitt and W. Thomas, *J. Chem. Soc.*, 1297 (1909).

The curves for these compounds show only a single absorption peak in the visible region and have molar extinction coefficients normal for such compounds.

Experimental

Intermediates were obtained commercially or were prepared by standard literature methods. Melting points are corrected. Microanalyses, given only for new compounds, were by the Research Services Section of these laboratories.

Bis(2-nitrophenyl) Ether (I).—A solution of 9 g. (0.05 mole) of potassium *o*-nitrophenoxide and 8 g. (0.05 mole) of *o*-nitrochlorobenzene in 30 ml. of dimethylformamide was heated at reflux for 18 hr., cooled, and poured over ice. Filtration, air drying, and recrystallization from ethanol gave 9.3 g., 71.5%, of yellow crystals melting at 114.5–116° (lit. m.p. 114.5°,[2] 116°[3]). The reactions in dimethyl sulfoxide and dimethylacetamide were carried out similarly.

The same procedure yielded the 2,3'-isomer as orange

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(5) A. Laubenheimer, *Ber.*, **8**, 1625 (1875).

(6) R. Adams and J. R. Johnson, "Laboratory Experiments in Organic Chemistry," The Macmillan Company, New York, N. Y., 1940, p. 335.

(7) H. v. Pechmann and K. Jenisch, *Ber.*, **24**, 3257 (1891).

needles of m.p. 78–80° after recrystallization from ethanol.

Anal. Calcd. for $C_{12}H_8N_2O_5$: C, 55.4; H, 3.1; N, 10.8. Found: C, 55.8; H, 3.1; N, 10.4.

3,3′-Dichloroazobenzene.—A solution of 42 g. (0.24 mole) of potassium *m*-nitrophenoxide and 47 g. (0.30 mole) of *m*-nitrochlorobenzene in 170 ml. of dimethylformamide was heated at reflux overnight. The reaction mixture was poured on ice, and the solid filtered off, dissolved in ethanol, treated with Darco, and partially evaporated. Cooling of this concentrated solution yielded 9.5 g. of brown crystals melting at 95.5–99°. Three recrystallizations from ethanol gave orange crystals of m.p. 99–102° (lit.,[5] 101°) and infrared spectrum and microanalysis consistent with the assigned structure.

Bis(2-aminophenyl) Ether (III).—A suspension of 52 g. (0.20 mole) bis(2-nitrophenyl) ether and 0.1 g. of palladium-on-charcoal catalyst in 125 ml. of methanol was hydrogenated in the Parr apparatus until hydrogen uptake ceased. The catalyst was removed by filtration and the filtrate drowned in ice–water to recover tan-yellow needles which, when dried, melted at 62.5–63.5° (lit.[8] m.p. 60° from water). The yield was 38 g., 95%.

Bis(2-acetamidophenyl) Ether.—Bis(2-nitrophenyl) ether (I) (50 g., 0.19 mole), 90 ml. of acetic anhydride, 60 ml. of acetic acid, and 0.1 g. of 10% palladium-on-charcoal catalyst were hydrogenated in the Parr apparatus until hydrogen uptake ceased. After removal of the catalyst by filtration, 150 ml. of methanol was added and the excess acetic anhydride destroyed by heating at reflux for a few minutes. Water (400 ml.) was added and the product slowly crystallized. The yield was 41 g., 75%, m.p. 182.5–185° after one recrystallization from methanol.

Anal. Calcd. for $C_{16}H_{16}N_2O_3$: C, 67.6; H, 5.7; N, 9.9. Found: C, 67.7; H, 5.6; N, 9.6.

Bis(2-acetamido-5-nitrophenyl) Ether.—Bis(2-acetamidophenyl) ether (26 g., 0.092 mole) was dissolved in 39 ml. of hot acetic acid and poured slowly into 60 ml. of cold concentrated sulfuric acid. A nitrating mixture of 18 ml. of concentrated nitric acid and 24 ml. of concentrated sulfuric acid was slowly added while the temperature was maintained below 10°. The reaction solution was then allowed to warm to room temperature and was poured over ice. The product was filtered, washed with water, and recrystallized from Methyl Cellosolve to give yellow crystals, m.p. 258–259°.

Anal. Calcd. for $C_{16}H_{14}N_4O_7$: C, 51.4; H, 3.8; N, 15.0. Found: C, 51.4; H, 3.8; N, 15.2.

Bis(2-amino-5-nitrophenyl) Ether (VI).—Bis(2-acetamido-5-nitrophenyl) ether (9.2 g., 0.025 mole), 30 ml. of concentrated hydrochloric acid, and 30 ml. of water were heated to reflux and sufficient Methyl Cellosolve was added to give an homogeneous solution. After 1 hr. at reflux, the solution was poured over ice and neutralized with ammonia; the product was filtered and air-dried. The yield was 7.1 g., 91%, of brown material decomposing at 221–231°. Three recrystallizations from Methyl Cellosolve yielded golden crystals melting at 248–250° with decomposition.

Anal. Calcd. for $C_{12}H_{10}N_4O_5$: C, 49.7; H, 3.5; N, 19.3. Found: C, 50.0; H, 3.6; N, 19.3.

4,4′-[Oxybis(4-nitro-*o*-phenyleneazo)]bis(*N*,*N*-dimethylaniline) (IX).—Bis(2-amino-5-nitrophenyl) ether (1.1 g., 0.0038 mole) was dissolved in 10 ml. of 5 *N* hydrochloric acid and cooled to 0–5° by adding ice. Sodium nitrite solution (0.1 *N*, 8 ml., 0.008 mole) was added slowly at 0–5° and stirring was continued for 1 hr. Dimethylaniline (1 g., 0.0083 mole) was dissolved in dilute hydrochloric acid and added in one portion, the pH was adjusted to about 5 with sodium acetate, and the product was filtered after an "R salt" test showed the solution to be free of diazonium salt. The nearly quantitative yield of product was air dried, and recrystallized from toluene, giving red crystals, m.p. 217–219° (dec.).

Anal. Calcd. for $C_{28}H_{26}N_8O_5$: C, 60.7; H, 4.7; N, 20.2. Found: C, 60.3; H, 4.4; N, 20.2.

When *N*-(2-cyanoethyl)-*N*-methylaniline was substituted for dimethylaniline, the product was 4,4′-[oxybis(4-nitro-*o*-phenyleneazo)]bis[*N*-(2-cyanoethyl)-*N*-methylaniline], giving red crystals from ethyl acetate, m.p. 208–209.5°.

Anal. Calcd. for $C_{32}H_{28}N_{10}O_5$: C, 60.7; H, 4.5; O, 22.3. Found: C, 61.2; H, 4.9; O, 2.0.

4,4′-[Oxybis(*p*-phenyleneazo)]bis(*N*,*N*-dimethylaniline) (VIII).—Bis(4-aminophenyl) ether (V) (2 g., 0.01 mole) was dissolved in 20 ml. 5 *N* hydrochloric acid and the solution diluted to 100 ml. with ice and water. Sodium nitrite solution (0.1 *N*, 200 ml., 0.02 mole) was added rapidly and after a few minutes a negative starch–iodide test was obtained. Dimethylaniline (2.5 g. 0.02 mole) in 30 ml. ethanol was added and the pH brought up to 5 with sodium acetate. After the coupling reaction was complete, the product was filtered, air dried, and recrystallized from *n*-propyl alcohol. The yield was nearly quantitative of yellow crystals which sintered at 183°, and had m.p. 186–188.5°.

Anal. Calcd. for $C_{28}H_{28}N_6O$: C, 72.4; H, 6.1; N, 18.1. Found: C, 70.8; H, 6.1; N, 17.2.

When the coupling component was 2-naphthol, the product was 1,1′-[oxybis(*p*-phenyleneazo)]bis(2-naphthol), a red solid which, after two recrystallizations from toluene, melted at 223.5–224.5° (dec.).

Anal. Calcd. for $C_{32}H_{23}N_4O_3$: C, 75.3; H, 4.3; N, 11.0. Found: C, 75.5; H, 3.9; N. 11.1.

When the coupling component was *N*-(2-cyanoethyl)-*N*-methylaniline, the product was **4,4′-[oxybis(*p*-phenyleneazo)bis[*N*-(2-cyanoethyl)-*N*-methylaniline]**, an orange solid which melted at 166–168° after two recrystallizations from *n*-propyl alcohol.

Anal. Calcd. for $C_{32}H_{30}N_8O$: C, 70.8; H, 5.57; N, 20.7. Found: C, 71.0; H, 5.68; N, 20.4.

Tetrazotization of Bis(2-aminophenyl) Ether (III) was carried out in the same manner as the tetrazotization of V.

Reaction with Acetonedicarboxylic Acid.—Acetonedicarboxylic acid (1.46 g., 0.1 mole) in 20 ml. of ethanol was added and the pH adjusted to 5 with sodium acetate. The red precipitate which formed was filtered off and washed with warm ethanol. No solvent for recrystallization could be found. When heated, the material began darkening at 240° and decomposed at 260–270°. There was no evidence even of microcrystallinity, and some absorption in the infrared at 5.9 and 7.9 μ indicated that carboxylic acid end groups, which would be expected if the material were polymeric, were present.

Anal. Calcd. for cyclic structure $C_{15}H_{12}N_4O_2$: C, 64.3; H, 4.3; N, 20.0. Found: C, 64.9; H, 4.9; N, 19.5.

When the coupling component was dimethylaniline, the product was **4,4′-[oxybis(*o*-phenyleneazo)]bis(*N*,*N*-dimethylaniline) (VII)**, a dark red material which, after two recrystallizations from toluene, melted at 206–210° with decomposition.

Anal. Calcd. for $C_{28}H_{28}N_6O$: C, 72.4; H, 6.1; N, 18.1. Found: C, 72.5; H, 6.0; N, 17.9.

When the coupling component was 2-naphthol, the product was **1,1′-[oxybis(*o*-phenyleneazo)]bis(2-naphthol)**, an orange material which melted at 164–266°, after two recrystallizations from toluene.

Anal. Calcd. for $C_{32}H_{22}N_4O_3$: C, 75.3; H, 4.3; N, 11.0. Found: C, 75.6; H, 4.0; N, 10.9.

***p*-{2-[α-(*p*-Dimethylamino)phenylazophenoxy]-4-[(*p*-nitrophenylazo)phenylazo}-*N*,*N*-dimethylaniline (X).**—To a solution of *p*-nitrobenzenediazonium chloride prepared from 3.5 g. (0.025 mole) of *p*-nitroaniline was added a solution of 5 g. (0.025 mole) of III in 30 ml. of acetic acid. When the coupling was completed, the product was filtered and air dried to yield 4.5 g., 50%, of material melting at 95–110°. This diamine (3.5 g., 0.01 mole) was tetrazotized in the

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(8) C. Haeussermann and E. Bauer, *Ber.*, **30**, 738 (1897).

same manner as was III. Dimethylaniline (2.54 g., 0.02 mole) in 30 ml. of acetic acid was added. When the solution had come to room temperature, the product was filtered off, dried, and recrystallized three times from toluene. Only a few hundred milligrams of yellow-brown product melting at 196–197.5° remained.

Anal. Calcd. for $C_{34}H_{31}N_9O_3$: C, 66.5; H, 5.1; N, 20.5. Found: C, 65.2; H, 6.3; N, 19.3.

p-(2-Methoxy-4-nitrophenylazo)-*N*,*N*-dimethylaniline (XII).—2-Methoxy-4-nitroaniline (3.3 g., 0.02 mole) was diazotized in a standard way and allowed to couple with 2.4 g. (0.02 mole) of dimethylaniline to give a nearly quantitative crude yield. Recrystallization from ethanol gave violet crystals, m.p. 173.5–175°.

Anal. Calcd. for $C_{15}H_{16}N_4O_3$: C, 60.0; H, 5.4; N, 18.7; O. 16.0. Found: C, 59.7; H, 6.0; N, 18.4; O, 15.7.

p-(o-Phenoxyphenylazo)-*N*,*N*-dimethylaniline (XIV) was prepared in the usual way.

Anal. Calcd. for $C_{20}H_{19}N_3O$: C, 75.7; H, 6.0; N, 13.2; O, 5.0. Found: C, 75.5; H, 5.9; N, 13.2; O, 5.0.

The Tetraphenylphenols

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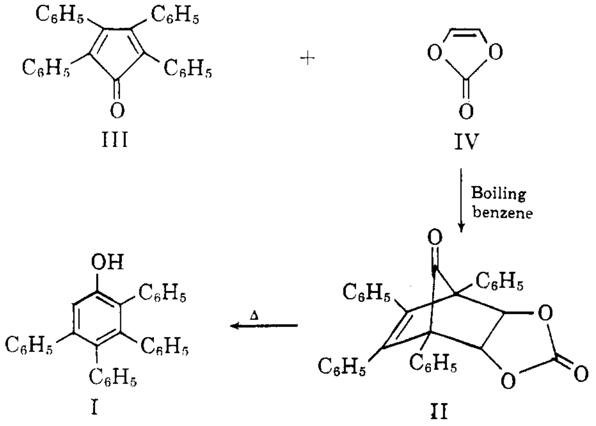
Received June 18, 1962

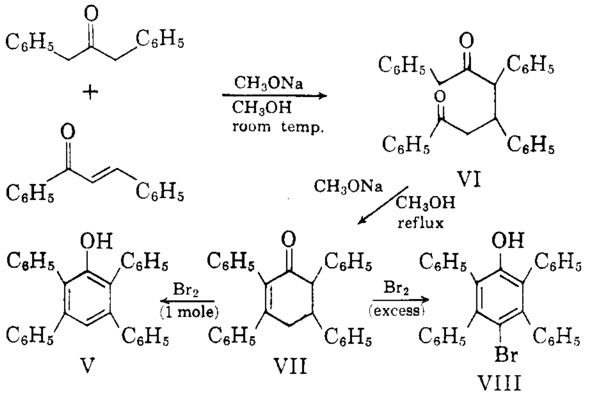
In connection with another investigation we have had occasion to synthesize the three tetraphenylphenols.

2,3,4,5-Tetraphenylphenol (I) was prepared by the following route:



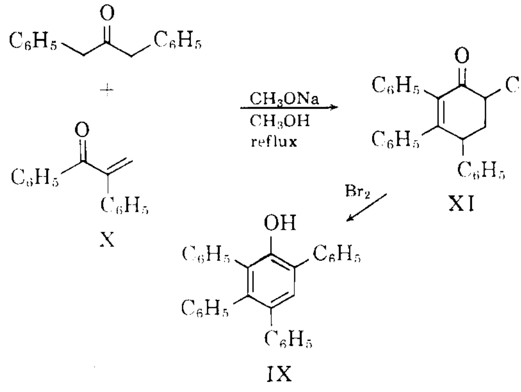
A Diels–Alder adduct II, of undetermined stereochemistry, was obtained when tetracyclone (III) and vinylene carbonate (IV)[2] were heated together in boiling benzene. The adduct has bands in its infrared spectrum at 5.50 and 5.59 μ; these can be assigned to the stretching vibrations of the carbonate[3] and ketonic[4] carbonyl groups, respectively. When II was heated at its melting point, gas evolution occurred and it was converted to a compound, $C_{30}H_{22}O$, which in its infrared spectrum shows no carbonyl band but a sharp hydroxyl band at 2.81 μ. On the basis of its genesis and spectrum this compound is assigned structure I. It was also obtained together with the adduct II when the Diels–Alder reaction was carried out in refluxing xylene.

2,3,5,6-Tetraphenylphenol (V) was prepared by the following route:



Michael addition of dibenzyl ketone to benzalacetophenone gave the δ-diketone VI (infrared bands at 5.84 and 5.93 μ) when carried out with methanolic sodium methoxide at room temperature. The same reagent at reflux effected the aldolization and dehydration of VI to give the cyclohexenone VII. This shows in its infrared spectrum a band at 6.00 μ and in its ultraviolet spectrum a maximum at 295 mμ (log ε 3.86) in accord with expectation.[5] Oxidation of VII with one molar equivalent of bromine gave 2,3,5 6-tetraphenylphenol (V) (infrared band at 2.83 μ). When excess of bromine was used for the oxidation, a brominated product, $C_{30}H_{21}OBr$, was obtained which is considered to have structure VIII on the basis of its ultraviolet spectrum (*vide infra*).

The remaining tetraphenylphenol, IX, was prepared by the following route, analogous to that used for the synthesis of V, although in this case the intermediates were not characterized (X → XI → IX).



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[1] Present address: Department of Chemistry, University of Toronto.

[2] M. S. Newman and R. W. Addor, *J. Am. Chem. Soc.*, **77**, 3789 (1955).

[3] *Cf.* C. L. Angell, *Trans. Faraday Soc.*, **52**, 1178 (1956).

[4] *Cf.* C. F. H. Allen, T. Davis, D. W. Stewart, and J. A. VanAllan, *J. Org. Chem.*, **20**, 306 (1955).