sodium hydroxide solution and extracted with ether. The aqueous solution was acidified and extracted with ether. The ether solution was evaporated, and the residue was crystallized from aqueous ethanol to yield 13 mg. (15%) of benzoic identical with an authentic sample in melting point, mixture melting point, and ultraviolet spectrum.

Chromic Acid Oxidation of 1-Methyl-1-hydroxy-2-benzyl-3-oxopyrollo[3,4-*c*]quinoline.—To a solution of 210 mg. of 1-methyl-1-hydroxy-2-benzyl-3-oxopyrollo[3,4-*c*]quinoline (1) in 10 ml. of acetic acid was added dropwise a solution of 120 mg. of chromium trioxide in 0.5 ml. of water and 5 ml. of acetic acid. This was heated on steam bath for 2.5 hr. and concentrated to a volume of *ca.* 2 ml. under reduced pressure. The residue was diluted with 20 ml. of water, treated with potassium carbonate, and extracted with chloroform. Evaporation of the chloroform afforded 150 ml. of material which was chromatographed on Woelm neutral alumina, Grade I. Elution with 30 ml. of chloroform gave 81 mg. of the *N*-benzylimide of 2-methylquinoline-3,4-dicarboxylic acid, identical in all respects with an authentic sample. Continued elution with chloroform yielded 48 mg. of 1-methyl-1-hydroxy-2-benzyl-3-oxopyrollo[3,4-c]quinoline, identical with the starting material.

Acetic Anhydride Dehydration of 1-Methyl-1-hydroxy-2-benzyl-3-oxopyrollo[3,4-*c*]quinoline.—A solution of 130 mg. of 1-methyl-1-hydroxy-2-benzyl-3-oxopyrollo[3,4-*c*]quinoline and 3 ml. of acetic anhydride was heated under reflux for 3 hr. This mixture was diluted with 3 ml. of water, agitated for 30 min., and evaporated under reduced pressure. The residue was triturated with ethyl acetate and collected to afford 90 mg. (69%) of the anhydro compound **3**, m.p. 156–159°. The analytical sample melted at 163–164° after crystallization from ethanol then ether; ultraviolet absorption: $\lambda_{\max}^{\mathrm{EtOH}}$ 250 mμ (ϵ 34,800), 296 (9500), 306 (10,000), 338 (6080), 349 (6340).

Anal. Calcd. for $C_{19}H_{14}ON_2$: C, 79.70, H, 4.83; N, 9.78. Found: C, 79.58; H, 4.98, N, 9.92.

The *N*-Benzylimide of 2-Methylquinoline-3,4-dicarboxylic Acid.—A mixture of 2.0 g. of 2-methylquinoline-3,4-dicarboxylic acid, m.p. 241–243°, prepared by the method of Pfitzinger,[10] 1.0 g. of benzylamine, and 6 ml. of 2,4,6-collidine was heated under reflux for 1 hr. The cooled mixture was diluted with water, and the precipitated imide was collected. After several crystallizations from ethyl acetate, there was obtained 1.20 g. (42%) of the *N*-benzylimide of 2-methylquinoline-3,4-dicarboxylic acid, m.p. 182–183°; ultraviolet absorption: $\lambda_{\max}^{\mathrm{EtOH}}$ 255 mμ (ϵ 69,000), 350 (6080).

Anal. Calcd. for $C_{19}H_{14}O_2N_2$: C, 75.48; H, 4.66; N, 9.26. Found: C, 75.20; H, 4.69; N, 9.50.

The *N*-Benzylimide of Quinoline-3,4-dicarboxylic Acid.—To a solution of 584 mg. of the *N*-benzylimide of 2-methylquinoline-3,4-dicarboxylic acid in 17 ml. of acetic acid was added dropwise a solution of 600 mg. of chromium trioxide in 0.5 ml. of water and 10 ml. of acetic acid. The mixture was heated on a steam bath for 3 hr. then concentrated under reduced pressure to *ca.* 4 ml. The residue was diluted with water, treated with potassium carbonate, and extracted with chloroform. The chloroform was evaporated, and the residue was chromatographed on Woelm neutral alumina, Grade I. Elution with 60 ml. of chloroform gave 171 mg. (30%) of starting material, and continued elution with chloroform gave 72 mg. of the impure *N*-benzylimide of quinoline-3,4-dicarboxylic acid. The second fraction was rechromatographed on alumina. Elution with benzene gave 10 mg. of starting material, and elution with 50% benzene–chloroform gave 45 mg. (8%) of the *N*-benzylimide of quinoline-3,4-dicarboxylic acid, m.p. 154–155° after crystallization from 70% ethanol–water; ultraviolet absorption: $\lambda_{\max}^{\mathrm{EtOH}}$ 255 mμ (ϵ 67,000), 350 (5900). The proton magnetic resonance spectrum in dimethyl sulfoxide showed a singlet at τ 0.70, ascribed to the proton adjacent to the nitrogen of the quinoline ring.

Anal. Calcd. for $C_{18}H_{12}O_2N_2$: C, 74.99; H, 4.19; N, 9.94. Found: C, 75.19; H, 4.24; N, 9.72.

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[10] W. Pfitzinger, *J. prakt. Chem.*, [2] **56**, 283 (1897).

Preparation of *m*-Polyphenyls[1]

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3-Ethoxy-2-cyclohexenone has been used for the synthesis of 3-(3-bromophenyl)-2-cyclohexenone, 1,3-di(3-keto-1-cyclohexenyl)benzene, and 3,3′-di(3-keto-1-cyclohexenyl)biphenyl. These ketones were employed with the appropriate Grignard reagents for the preparation of the following linear *m*-polyphenyls: *m*-quarterphenyl, *m*-sexaphenyl, *m*-octaphenyl, 3,3′′-dibromo-*m*-terphenyl, 3,3′′′′-dibromo-*m*-quinquephenyl, and 3,3′′′′′-dibromo-*m*-sexaphenyl. Infrared and ultraviolet spectra data are reported for the preceding polyphenyls.

The use of 3-ethoxy-2-cyclohexenone (I) has been shown by earlier work to be a very valuable reagent for the synthesis of *m*-diarylbenzenes.[4,5] This paper will present the preparation of 3-(3-bromophenyl)-2-cyclohexenone (II), 1,3-di(3-keto-1-cyclohexenyl)benzene (III), and 3,3′-di(3-keto-1-cyclohexenyl)biphenyl (IV) from 3-ethoxy-2-cyclohexenone (I) and subsequent reaction of these diketones to give linear *m*-polyphenyls.

The reaction of 3-ethoxy-2-cyclohexenone (I) and 3-bromophenylmagnesium bromide produced II while reaction of two moles of I with the di-Grignard reagent of 1,3-dibromobenzene afforded III. This latter reaction was accomplished in 90% yield. Earlier, Ruskie[6] had prepared III by the use of *n*-butyllithium as the metalating agent.

The synthesis of IV was achieved by the employment of two moles of I with the di-Grignard reagent of 3,3′-dibromobiphenyl.

3,3′′-Dibromo-*m*-terphenyl (V) was produced by the sequence: (1) reaction of II and 3-bromophenylmagnesium bromide, (2) dehydration of the resulting alcohol, and (3) aromatization of the preceding diene. Compound V could not be obtained as a solid in our hands. Interestingly, 3-bromo-*m*-terphenyl,[6] which was prepared earlier in our laboratory, also was found to be a liquid at room temperature.

The diketone (III) was treated with two moles of 3-bromophenylmagnesium bromide to give 3,3′′′′-dibromo-*m*-quinquephenyl (VI). Compound VI was treated with an excess of *n*-butyllithium and, after hydrolysis, *m*-quinquephenyl was obtained. This in itself is not a proof of structure for VI, but it does aid in the proof of structure for the *m*-quinquephenyl skeleton moiety.

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[1] This work was supported in part by U. S. Air Force contract AF 33(616)-5063.

[2] Du Pont Teaching Fellow, 1960–1961.

[3] Gillette–Harris Research Fellow, 1961–1962.

[4] G. F. Woods, F. T. Reed, T. E. Arthur, and H. J. Ezekiel, *J. Am. Chem. Soc.*, **73**, 3854 (1951).

[5] G. F. Woods, D. D. Centola, H. E. Ruskie, and C. D. Miller, *ibid.* **82**, 5227 (1960).

[6] Personal communications, H. Ruskie.

CHART I

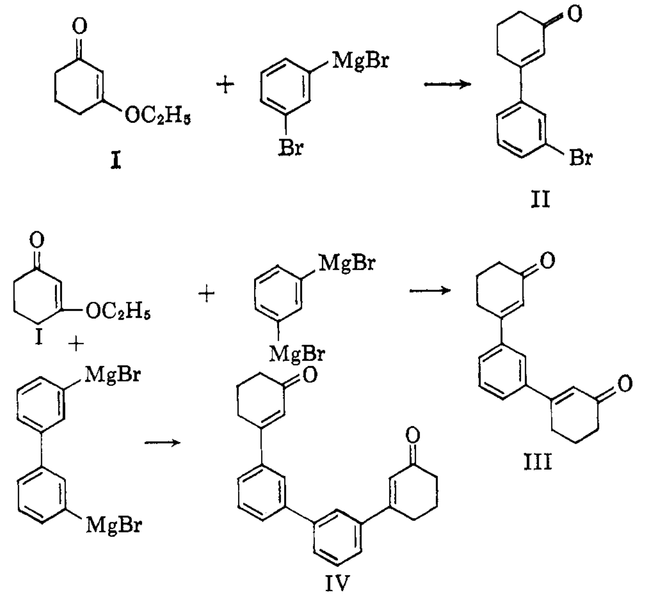
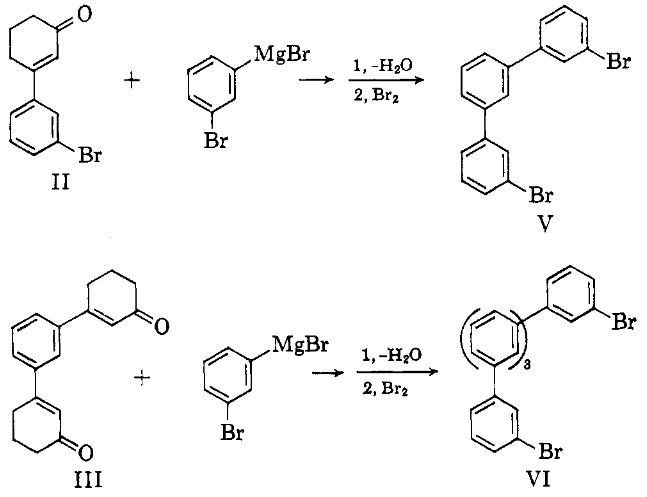


CHART II



Both compounds V and VI have been synthesized in this laboratory by a different route which will be described in a later paper.[7]

The four-ring diketone (IV) proved to be a versatile reagent as shown in the preparation of *m*-quaterphenyl (VII), *m*-sexaphenyl (VIII), *m*-octaphenyl (IX), and 3,3'''''-dibromo-*m*-sexaphenyl (X).

It will be noted that aromatization was effected in the reactions (Chart III) by use of palladium–charcoal except in the compounds containing bromine. If halogens are present during aromatization by palladium–charcoal, loss of hydrogen bromide occurs; thus the bromine addition–dehydrobromination procedure was employed in these cases.

The ultraviolet spectra of the *m*-polyphenyls that were described in this paper were observed to correlate well with earlier reported data.[4,8] Since conjugation for the *m*-polyphenyls is prohibited beyond that shown in the biphenyl molecule, λ_{max} is shown to be relatively constant in this series of compounds. However, ϵ_{max} increases with the addition of each phenyl group and the higher *m*-polyphenyls are very strong absorbers.

CHART III

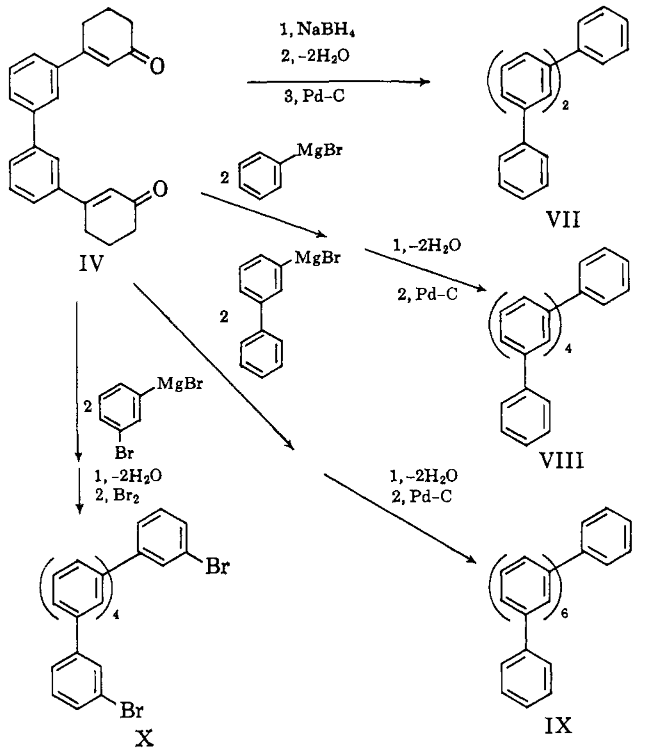


TABLE I

	λ_{max}	$\epsilon_{max} \times 10^{-4}$
m-Quaterphenyl	247	61,000
m-Sexaphenyl	248	105,000
m-Octaphenyl	248	142,000
3,3'-Dibromobiphenyl	248	20,830
3,3''-Dibromo-*m*-terphenyl	247	44,600
3,3''''-Dibromo-*m*-quinquephenyl	247	87,300
3,3'''''-Dibromo-*m*-sexaphenyl	247	113,000

The infrared spectra of *m*-polyphenyls reported in this paper were examined closely in the 700–900-cm.$^{-1}$ region. The region (700–900 cm.$^{-1}$), which is associated with the C–H out-of-plane deformations, is usually the most important for the interpretation of the polyphenyls.[9]

3,3'-Dibromobiphenyl shows absorption bands associated with one and three adjacent free hydrogen atoms, 875 and 775 cm.$^{-1}$, while biphenyl shows a band at 730 cm.$^{-1}$ corresponding to five adjacent free hydrogen atoms. Compound V shows bands at 875 and 775 cm.$^{-1}$ corresponding to one and three adjacent free hydrogen atoms and *m*-terphenyl shows these bands in addition to a band at 755 cm.$^{-1}$ which is associated with five adjacent free hydrogen atoms. A similar statement may be made concerning the comparison of VI (880 and 775 cm.$^{-1}$) with *m*-quinquephenyl (885, 790, and 755 cm.$^{-1}$) and X (855 and 775 cm.$^{-1}$) with *m*-sexaphenyl (885, 780, and 755 cm.$^{-1}$). Both VII and IX show bands corresponding to one, three, and five adjacent free hydrogen atoms. All of the *m*-polyphenyls show bands near 700 cm.$^{-1}$.

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[7] N. B. Sunshine and G. F. Woods, *J. Org. Chem.*, **28**, 2517 (1963).

[8] R. L. Alexander, Jr., *ibid.*, **21**, 1464 (1956).

[9] L. J. Bellamy, "The Infrared Spectra of Complex Molecules," John Wiley and Sons, Inc., New York, N. Y., 1958, Chap. V.

Experimental

3-Ethoxy-2-cyclohexenone was prepared according to the directions in *Organic Syntheses*.[10]

Tetrahydrofuran employed in the Grignard reactions was dried by distillation from lithium aluminum hydride while ether and benzene were dried over sodium.

The ultraviolet absorption spectra were obtained with a Beckman spectrophotometer, Model DK-1, using Spectro Grade isooctane or 95% ethanol as the solvent. All infrared absorption spectra were obtained with a Beckman IR-5 recording spectrophotometer using melts on sodium chloride plates. Molecular weights were determined by the use of a Mechrolab osmometer. All melting points and boiling points are uncorrected.

3-(3-Bromphenyl)-2-cyclohexenone (II).—3-Bromophenylmagnesium bromide was prepared from magnesium (0.85 g.-atom) and 1,3-dibromobenzene (1 mole) in 450 ml. of anhydrous ether. The flask was equipped with a condenser, magnetic stirrer, and dropping funnel. After most of the magnesium had reacted, 3-ethoxy-2-cyclohexenone (105 g., 0.75 mole) dissolved in 150 ml. of ether was added slowly. The reaction mixture was refluxed for 3 hr. after the ketone addition, cooled, and hydrolyzed with dilute sulfuric acid. The resulting mixture was extracted with ether, and the organic layer was submitted to steam distillation from an acid medium (5% sulfuric acid). After 4 l. of distillate had been collected, the residue in the steam pot was cooled and extracted with ether. The ether solution was washed with water, 10% sodium hydrogen carbonate solution, and water, and dried over magnesium sulfate. The residue was distilled using a Vigreux column after evaporation of the ether. The fraction distilling in the range of 163–166° at 1.2 mm. was collected; yield, 86.6 g. (46%). The 2,4-dinitrophenylhydrazone derivative was prepared in the usual manner, m.p. 253–255° (lit.[6] m.p. 253–257°), after recrystallization from ethyl acetate.

3,3''-Dibromo-*m*-terphenyl (V).—1,3-Dibromobenzene (0.27 mole) dissolved in 100 ml. of ether was placed in a three-neck flask which was equipped with a stirrer, reflux condenser, and dropping funnel. While a stream of dry nitrogen was being passed into the system, 196 ml. of 1.02 *N* *n*-butyllithium was added slowly. The reaction mixture was kept in an ice bath during the addition. After the addition was completed, stirring was continued for 3 hr. and then a solution of II (0.16 mole) dissolved in 70 ml. of ether was added. This reaction mixture was stirred 24 hr. more, hydrolyzed with cold dilute sulfuric acid, and submitted to steam distillation in an acid medium. The residue in the steam pot was extracted with ether and the ethereal solution was washed with water, 5% sodium hydrogen carbonate solution, and water in succession. The ether layer was dried and, after evaporation of the ether, a yellow oil was obtained. This oil was dissolved in chloroform and aromatized by addition of bromine followed by elimination of hydrogen bromide. The chloroform solution was warmed on the steam bath and after evaporation of most of the solvent a dark viscous substance was isolated and distilled using a Hickman still. No crystalline substance could be obtained from the four relatively equal fractions of the distillate. The infrared spectra of fractions of two and three were identical with the infrared spectrum of the material 3,3''-dibromo-*m*-terphenyl which had been prepared by another method to be reported.[7]

Anal. Calcd. for $C_{18}H_{12}Br_2$: C, 55.68; H, 3.11. Found: C, 56.20; H, 3.70.

1,3-Di(3-keto-1-cyclohexenyl)benzene (III).—1,3-Dibromobenzene (0.2 mole) dissolved in 200 ml. of tetrahydrofuran was added as fast as possible to magnesium turnings (4.86 g.; 0.2 g.-atom). After all of the magnesium had been consumed, more magnesium (5.23 g., 0.215 mole) was added and the mixture was refluxed for 18 hr. 3-Ethoxy-2-cyclohexenone (56 g.; 0.4 mole) was dissolved in a minimum amount of tetrahydrofuran and was added slowly to the Grignard mixture. The reaction mixture was refluxed 8 hr. more and filtered through glass wool into an ammonium chloride solution. The resulting solution was extracted with 750 ml. of benzene, and the benzene layer was steam distilled in an acid medium (5% sulfuric acid). The residue in the steam pot was dissolved in hot benzene, washed with water, twice with 100-ml. portions of 5% sodium hydroxide solution, and with water again. The benzene layer was dried and after filtration of the drying agent, the solution was concentrated to 150 ml. This solution was diluted with 100 ml. of petroleum ether (b.p. 100–120°) and allowed to stand at room temperature. A tan-white precipitate was isolated and recrystallized from isopropyl alcohol, m.p. 133–135°; yield, 48 g. (90%).

3,3''''-Dibromo-*m*-quinquephenyl (VI).—Magnesium (0.97 g., 0.04 g.-atom) and 70 ml. of ether were placed in a three-neck flask which was equipped with a magnetic stirrer, condenser, and dropping funnel. 1,3-Dibromobenzene (11.8 g., 0.05 mole) was added as fast as possible and, after most of the magnesium had been consumed, III (0.013 mole) in 100 ml. of benzene was added slowly. The reaction mixture was refluxed for 24 hr., cooled, and added to dilute sulfuric acid. The benzene layer was separated, submitted to steam distillation from an acid medium (5% sulfuric acid by weight), and the residue in the steam pot was extracted with benzene. This benzene layer was washed successively with water, 10% sodium hydrogen carbonate solution, and water again. After being dried over magnesium sulfate, the benzene was evaporated. The residue was dissolved in chloroform and aromatized by the bromine method. This resulting solution was heated to expel hydrogen bromide and then was chromatographed on alumina (80–200 mesh) and eluted with a mixture of petroleum ether (b.p. 60–80°)–benzene (4:1). A yellow-orange glass was obtained which crystallized from ethanol, m.p. 104–106°; yield, *ca.* 0.5 g. (9%). The infrared spectrum was identical with the infrared spectrum of 3,3''''-dibromo-*m*-quinquephenyl that has been prepared by another method to be reported.[7] A higher melting substance also was isolated but was not identified.

Anal. Calcd. for $C_{30}H_{20}Br_2$: C, 66.68; H, 3.74; mol. wt., 540. Found: C, 66.70; H, 4.00, mol. wt., 569.

***m*-Quinquephenyl.**—Compound VI (0.45 g.) was dissolved in 50 ml. of dry ether. This solution was kept cold while an excess of *n*-butyllithium was added slowly. The reaction mixture was stirred for 8 hr. at room temperature, cooled, and hydrolyzed by the addition of water. The ether layer was separated and filtered through a short column filled with alumina. The solvent was evaporated and the residue was crystallized from alcohol, m.p. 114–115° (lit.[8] m.p. 117–118°). A mixture melting point was taken with the starting material and a range of 99–108° was observed.

Anal. Calcd. for $C_{30}H_{22}$: C, 94.20; H, 5.80. Found: C, 94.15; H, 5.60.

3,3'-Di(3-keto-1-cyclohexenyl)biphenyl (IV).—3,3'-Dibromobiphenyl (0.1 mole) dissolved in 50 ml. of benzene was added with stirring to a mixture of magnesium (0.24 g.-atom) and 120 ml. of tetrahydrofuran, and the resulting mixture was refluxed 24 hr. 3-Ethoxy-2-cyclohexenone (0.2 mole) in 70 ml. of benzene was added slowly to the Grignard medium. Following the addition of the ketone, the mixture was refluxed gently for 12 hr., cooled, and added to a cold dilute solution of sulfuric acid. The organic layer was steam distilled under acid (5% sulfuric acid by weight) conditions, and the residue in the steam pot was extracted with hot benzene. The benzene solution was washed with water, 10% sodium hydrogen carbonate, and water and dried over magnesium sulfate. Filtration removed the drying agent and a yellow precipitate was obtained when the benzene solution was concentrated to approximately 30 ml. This solid was isolated by filtration and recrystallized from ethyl acetate; m.p. 176–178°; yield, 28.4 g. (83%).

Anal. Calcd. for $C_{24}H_{22}O_2$: C, 84.21; H, 6.43. Found: C, 84.34; H, 6.67.

The 2,4-dinitrophenylhydrazone of the diketone was prepared by the extraction of the diketone (using a Soxhlet apparatus) into a solution of the reagent, concentrated hydrochloric acid, and a solvent mixture of ethyl alcohol–petroleum ether (2:1). The red derivative melted at 268–270°.

Anal. Calcd. for $C_{36}H_{30}O_8$: C, 61.54; H, 4.27. Found: C, 61.51; H, 4.49.

3,3'-Di(3-hydroxy-1-cyclohexenyl)biphenyl.—To a solution of 0.013 mole of the diketone (IV) in 100 ml. of Cellosolve, sodium borohydride (0.078 mole) in 25 ml. of water was added, and the whole was allowed to stand at room temperature for 12 hr. The solution was made just neutral to litmus with 4 *N* sulfuric acid, and water and ice were added until the solution became cloudy. A gummy white material precipitated which was extracted with ether. The ether layer was washed with water, sodium hydrogen carbonate, and finally with water, and dried. After removal of the drying agent and the ether, a colorless oil was obtained which could not be crystallized; yield, 4.4 g.

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(10) W. F. Gannon and H. O. House, *Org. Syn.*, **40**, 41 (1960).

3,3'-Di(cyclohexa-1,3-dienyl)biphenyl.—A mixture of 4.4 g. of the above crude dihydroxyl compound and 0.026 mole of crystalline boric acid[10] was placed in a test tube and heated in a Wood's metal bath at 280–300° for 5 min. The mixture was stirred with a stirring rod while being heated. After cooling, the material in the test tube became a yellow glass. This material was dissolved in hot benzene by portionwise extraction. The benzene solution was filtered, washed with water, sodium hydrogen carbonate solution, and finally with water. The yellow organic layer was dried and after removal of the drying agent, the solution was evaporated to 15 ml. This was chromatographed on alumina (80–200 mesh) with benzene as an eluent. After complete removal of the solvent from the eluate, 2.0 g. of a yellow gum was obtained. This material could not be induced to crystallize.

m-**Quaterphenyl** (**VII**).—A solution of the prior crude diene (3.5 g.) in 10 ml. of *p*-cymene was heated under reflux with 5% palladium-charcoal (0.35 g.) to effect aromatization. The solvent was slowly and almost completely removed by distillation and at a temperature of 300–310° rapid evolution of hydrogen was observed. This temperature was maintained until no further gas evolution was seen (2 hr.). The resulting black glassy material was dissolved in hot benzene and filtered through a 20-cm. alumina column. Evaporation of the benzene from the eluate left a pale yellow glass. This material was distilled and a colorless distillate was collected at 240–250° at 1-mm. pressure. A solution of the distillate in absolute ethanol deposited white platelike crystals after standing for 2 days which were recrystallized from ethanol. The over-all yield of *m*-quaterphenyl based on the starting diketone (IV) was 30% (1.2 g.); m.p. 83–84° (lit. m.p. 85–86°).[11]

Anal. Calcd. for $C_{24}H_{18}$: C, 94.12; H, 4.88. Found: C, 93.33; H, 5.60.

m-**Sexaphenyl** (**VIII**).—A Soxhlet extractor was employed for the addition of diketone (IV) (0.01 mole) to an ethereal solution (150 ml.) of phenylmagnesium bromide which had been prepared in the usual manner from 0.12 mole of bromobenzene. The addition required approximately 4 days. The Grignard complex was decomposed with dilute sulfuric acid. The yellow ethereal layer was separated and the water layer was extracted again with benzene. The combined organic extracts were submitted to steam distillation from dilute phosphoric acid. The residue in the steam pot was extracted with benzene, and the resulting benzene solution was washed with water, saturated sodium hydrogen carbonate solution, and finally with water. The benzene volume was reduced to approximately 10 ml. by distillation, and this solution was passed through a small alumina column with the aid of a further 25 ml. of benzene. By evaporation of the solvent, 3 g. of glassy material was obtained which could not be induced to crystallized.

Direct aromatization of this material was effected with palladium-charcoal in the manner described earlier. The product was treated also as described before. *m*-Sexaphenyl (2.1 g., 44%) was obtained upon recrystallization from ethanol; m.p. 144–145° (lit.[8] m.p. 147–147.5°).

Anal. Calcd. for $C_{36}H_{26}$: C, 94.32; H, 5.68. Found: C, 94.41; H, 5.55.

m-**Octaphenyl** (**IX**).—In the manner described in the preceding preparation and employing the Soxhlet apparatus, 0.01 mole of diketone (IV) was added to the Grignard reagent prepared from 0.082 mole of 3-xenyl bromide in ether. The addition was effected in 4 days. The Grignard reaction mixture was then hydrolyzed and worked up as described before for the analogous reaction mixture. The amount of yellow glass obtained after the final evaporation of solvent was 4.4 g. This material could not be crystallized.

The prior crude tetraene (4.4 g.) in 10 ml. of *p*-cymene was aromatized with palladium-charcoal as described for *m*-sexaphenyl. The flask was cooled and the material dissolved in ether. The solution was filtered through a 10-cm. alumina column. The eluate was evaporated to dryness and the residue was crystallized from ether–petroleum ether (b.p. 30–60°) and recrystallized from ether to yield *m*-octaphenyl (1.4 g., 23%); m.p. 127–129° (lit.[8] m.p. 127–129°).

Anal. Calcd. for $C_{48}H_{34}$: C, 94.42; H, 5.38. Found: C, 94.22; H, 5.85.

3,3'''''-Dibromo-*m*-sexaphenyl (**X**).—3-Bromophenylmagnesium bromide was prepared from magnesium (0.98 g., 0.04 g.-atom) and 1,3-dibromobenzene (9.4 g., 0.04 mole) in 60 ml. of ether. Most of the magnesium had reacted when the diketone IV (0.013 mole), dissolved in 150 ml. of benzene, was added slowly. The mixture was stirred under reflux for 40 hr., cooled, hydrolyzed by cold dilute sulfuric acid, and extracted with benzene. The benzene layer was submitted to steam distillation from a dilute sulfuric acid medium, and the steam pot residue was extracted with hot benzene. The benzene layer was washed with water, dilute base, and water and finally dried. Evaporation of the solvent left behind a viscous yellow-orange glass. This residue was dissolved in chloroform and aromatized by the previously described bromine method. The resulting solution, after expulsion of hydrogen bromide, was passed through a small alumina (80–200 mesh) column. Petroleum ether (b.p. 60–80°)-benzene (4:1) was employed as the eluent. An orange viscous material was isolated after evaporation of the first 200 ml. of solvent and recrystallized from acetone (six times). It was finally recrystallized from a mixture of carbon tetrachloride and ethanol to provide X in small yield; m.p. 143–145°.

Anal. Calcd. for $C_{36}H_{24}Br$: C, 70.15; H, 3.93; mol. wt., 616. Found: C, 70.43; H, 4.21; mol. wt., 620.

Acknowledgment.—The author wishes to thank Dr. Franz Kasler for the microanalyses and Mr. Donald Glover of the Naval Ordnance Laboratory for the molecular weight determinations.

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[11] G. F. Woods and F. T. Reed, *J. Am. Chem. Soc.*, **71**, 1348 (1949).

Preparation of Linear *m*-Polyphenyls from Mono- and Dichalcones[1]

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A new method has been developed for the synthesis of linear *m*-polyphenyls. This procedure involves the following steps: (1) chalcone formation, (2) Michael condensation and ring closure, (3) decarbethoxylation, (4) reduction, (5) dehydration, and (6) aromatization. The preparation of *m*-terphenyls, *m*-quinquephenyls, certain intermediates, and the possible preparation of *m*-sexaphenyls will be discussed.

Heretofore, the only useful general method for the preparation of linear *m*-polyphenyls has been the one which employs the reaction of 3-ethoxy-2-cyclohexenone[4,5] with organolithium or Grignard reagents. The resulting ketone is treated with another organolithium or Grignard reagent and, after dehydration and aromatization steps, the desired polyphenyl is obtained.

Another general method for the synthesis of *m*-polyphenyls which is reported herein has been desired for two reasons. The first was that an alternate syn-

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[1] This work was supported in part by the U. S. Air Force contract AF 33(616)-5063.

[2] Du Pont Teaching Fellow, 1960–1961.

[3] Gillette-Harris Research Fellow, 1961–1962.

[4] G. F. Woods, F. T. Reed, T. E. Arthur, and H. J. Ezekiel, *J. Am. Chem. Soc.*, **73**, 3854 (1951).

[5] G. F. Woods, D. D. Centola, H. E. Ruskie, and C. D. Miller, *ibid.*, **82**, 5227 (1960).