

Reactions of Alkali Diphenylmethides with 1,1-Dihalides and 1,1,1-Trihalides. In Table II are summarized the results obtained from the reactions with various halides. The details of typical experiments are described below.

A. Reaction with 1,1-Dichloroethane. A solution of 2.5 g (0.025 mol) of 1,1-dichloroethane in ether was added to 0.05 mol of ammoniacal potassium diphenylmethide. The ammonia was evaporated, and the residue was stirred with ethanol and filtered. The ethanolic solution was combined with an acidic solution of 5 g of 2,4-dinitrophenylhydrazine in ethanol. The yellow dinitrophenylhydrazone of acetaldehyde, mp and mmp 168° (after recrystallization from ethanol), weighed 0.62 g (11%).

B. Reaction with 1,1,1-Trichloroethane. An ethereal solution of 2.23 g (0.017 mol, 0.33 mole equiv) of 1,1,1-trichloroethane was added to 0.05 mol of potassium diphenylmethide in 200 ml of liquid ammonia. The acetaldehyde dinitrophenylhydrazone, isolated as above, weighed 0.45 g (12%); mp and mmp 168° after recrystallization from ethanol.

C. Reaction with α,α,α -Trichlorotoluene. To a solution of 0.05 mol of potassium diphenylmethide in 200 ml of liquid ammonia was added 3.29 g (0.017 mol) of α,α,α -trichlorotoluene. The benzaldehyde dinitrophenylhydrazone, isolated as above, was

recrystallized from ethanol-ethyl acetate to give 1.24 g (26%) of orange crystals, mp and mmp 235–237°.

Reaction of Potassium Diphenylmethide with N-Bromosuccinimide. To a solution of 0.02 mol of potassium diphenylmethide, prepared by addition of a solution of 3.36 g (0.02 mol) of diphenylmethane in tetrahydrofuran to 0.02 mol of potassium amide in 100 ml of liquid ammonia, was added a solution of 1.78 g (0.01 mol) of N-bromosuccinimide in tetrahydrofuran. The orange color gradually disappeared. The mixture was stirred as the ammonia evaporated, and the residue was stirred with carbon tetrachloride and filtered. Evaporation of the carbon tetrachloride gave 2.74 g (82%) of tetraphenylethane, mp and mmp 210°. The material insoluble in carbon tetrachloride was stirred with acetone and enough concentrated hydrochloric acid to make the mixture slightly acidic, and the mixture was filtered. The acetone was evaporated, the residue was stirred with acetone and filtered, and the solution was concentrated and chilled, to give 0.76 g (76%) of succinimide, mp and mmp 125–127°.

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Structure of the Diquinone Resulting from Oxidation of β -Naphthoquinone

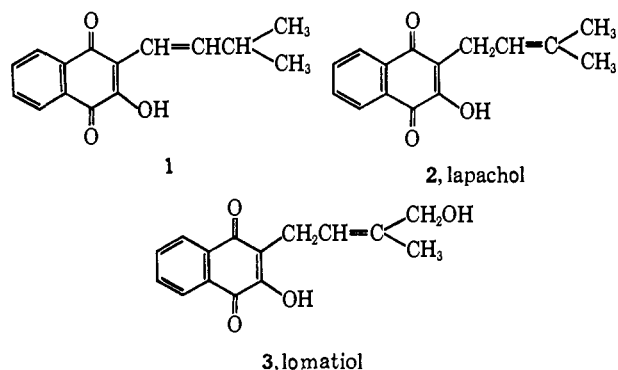
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Contribution from the Chemical Laboratory of Harvard University, Cambridge, Massachusetts 02138. Received February 13, 1968

Abstract: A yellow compound, $C_{20}H_{10}O_6$, formed in good yield on oxidation of β -naphthoquinone with ferric chloride was reported by Hooker and Fieser¹ in a paper of 1936 to yield β,β -dinaphthyl on zinc dust distillation and on this evidence was regarded as the hydroxydinaphthylidiquinone **6a** rather than **6**. Later British work, however, showed that β,β -dinaphthyl could have been formed by rearrangement of the initially formed α,α or α,β isomer. We have now found that comparison of the nuclear magnetic resonance spectrum with those of the model compounds **7** and **8** shows conclusively that the structure is **6** and not **6a**. Compound A, obtained by oxidation as one of six transformation products described by Hooker and Fieser, is inferred on infrared evidence to have the structure **10**, and this is amply confirmed by the nuclear magnetic resonance of the acetate methyl ester, as shown in Figure 1. The decarboxylation of A to the methyl compound C can be followed by continuous recording of the nmr spectrum in hot pyridine. Orange compound D and red compound F, obtained by the reaction of concentrated sulfuric acid on A and on C, are characterized by formation and by nonformation of a bisulfite addition compound as the β -naphthoquinone **11** and the α,β -unsaturated α -naphthoquinone lactone **15**.

This investigation is an extension of work reported in a paper written by L. F. F. as junior author¹ and published in 1936, a year after the death of the senior author, Samuel C. Hooker. This English-born chemist, after taking the Ph.D. degree with Bamberger at Munich after just 1 year of research, had, in 1885, become chief chemist for the Franklin Sugar Refining Co. in Philadelphia.² A Philadelphian importer of South American bethabarra wood, valued for the preparation of fine fishing rods and bows on account of its remarkable elasticity, invited young German-trained Hooker to investigate a yellow substance which occurs in the grain of the wood.³ Greene and Hooker⁴ isolated the pigment and identified it as the known but in-

completely characterized substance lapachol, then regarded by Paternò⁵ as the 2-hydroxy-3-(α -alkenyl)-1,4-naphthoquinone **1**. Hooker found the chemistry



(1) S. C. Hooker and L. F. Fieser, *J. Am. Chem. Soc.*, **58**, 1216 (1936).

(2) C. A. Browne, *J. Chem. Soc.*, 550 (1936).

(3) L. F. Fieser, "The Scientific Method," Reinhold Publishing Corp., New York, N. Y., 1964.

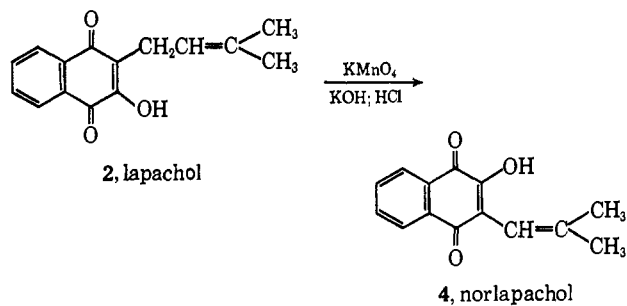
(4) W. H. Greene and S. C. Hooker, *Am. Chem. J.*, **11**, 267 (1889); S. C. Hooker and W. H. Greene, *ibid.*, **11**, 393 (1889); *Chem. Ber.*, **22**, 1723 (1889).

of lapachol a fascinating subject for spare-time research and he gained the experimental cooperation of various

(5) E. Paternò, *Gazz. Chim. Ital.*, **12**, 377 (1889).

voluntary assistants who proffered their services in return for the valuable training which the association provided. Problems attacked with successful outcome included the complete elucidation of the structure of lapachol as the β -alkenyl derivative **2**,⁶ elucidation of the structure of lomatiol (from an Australian seed) as **3**,⁷ and conversion of *o*- into *p*-, and of *p*- into *o*-quinone derivatives.⁸

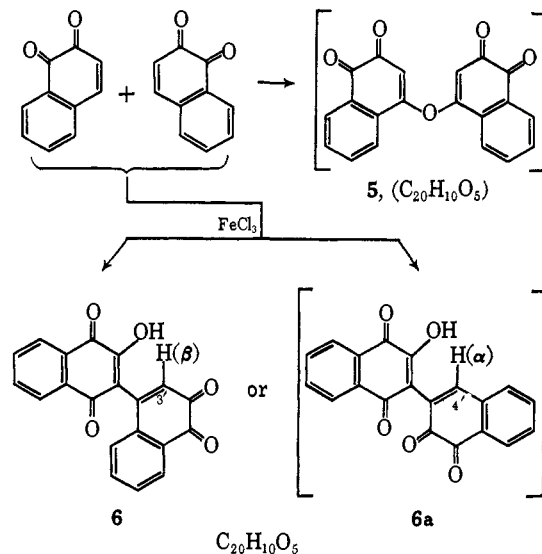
When the senior author of the present paper first met Hooker in 1927, he had retired from the sugar industry, transformed an elegant former stable to accommodate a chemical laboratory, an extensive chemical library, and a stage for the performance of magic to fellow members of the American Society of Magicians, and he had resumed the quinone research where he had left off in 1896. An intimate association with this gifted experimentalist of rare personality and ability continued until his death in 1935. Hooker's naphthoquinone samples were bequeathed to me and eventually provided the clue for an extensive investigation in chemotherapy which is still in progress.⁹ Research in the period 1915-1935 had all gone unpublished because some of them were still incomplete. However, Hooker had discussed these problems with me many times and I had, with his assent, achieved the synthesis of lapachol¹⁰ and extended his discovery of the remarkable "Hooker oxidation" reaction,¹¹ for example, of **2** into **4**, by



undertaking clarification of the mechanism.¹² Hooker's laboratory notes and those of his coworkers were in impeccable condition and on his death I edited, or wrote, or completed by experimentation, 11 papers which were published posthumously in a block in 1936.

At the outset, the last of these papers was far from complete. Wichelhaus¹³ had discovered that ferric chloride added to a suspension of β -naphthoquinone in hot water oxidizes the quinone to a much less soluble yellow product, mp 253-254° dec, which he formulated as "the di- β -naphthoquinone oxide" **5**. Hooker reexamined the compound and found the properties inconsistent with formula **5**, for it dissolves easily in dilute alkali in the cold, as well as in sodium carbonate or bicarbonate, to give red solutions, and forms a crystal-

line acetyl derivative. The condensation product thus must be either 2-hydroxy-3,4'-dinaphthyl-1,4,1',2'-diquinone (**6**) or 2-hydroxy-3,3'-dinaphthyl-1,4,1',2'-diquinone (**6a**). By such simple methods as air oxida-



tion, refluxing in acetic acid or in xylene, or treatment with sulfuric acid, Hooker succeeded in converting the supposed oxide into no less than six well-defined, crystalline, quinonoid transformation products (A-F). However, these were all new compounds and their properties cast no light on the structure of the precursor discernible to Hooker or, later, to me. I then sought to distinguish between formulas **6** and **6a** by zinc dust distillation and isolated from the reaction a hydrocarbon identified as β,β -dinaphthyl. I concluded that the oxidation product has the structure **6a**. Twenty-four years later, however, this evidence was invalidated by the finding of Copeland, Dean, and McNeil¹⁴ that the three isomeric dinaphthyls undergo rapid interconversion at 490°, the order of stability being $\beta,\beta > \alpha,\beta > \alpha,\alpha$. In the single degradation experiment carried out by one of us in 1936¹ no attempt had been made to maintain mild conditions. A later degradation is uncertain with respect to the identity of the starting material. Weygand and Frank¹⁵ treated 3 g of α -tetralone in *n*- or isopropyl alcohol with 6 g of selenium dioxide in 7 ml of water at 30° for 17-22 hr. Work-up, involving extraction with sodium carbonate solution and acidification, and then crystallization from dioxane and aqueous acetic acid, afforded 50 mg of an orange-red product melting at 258-259°, a few degrees higher than reported for the Wichelhaus product, and analyses and molecular weight determinations were in approximate agreement with the requirements for C₂₀H₁₀O₅, a hydroxydinaphthylidiquinone, that is, **6** or **6a**. Zinc dust distillation of 500 mg of the quinone with 8 g of zinc dust carried out by the German investigators by heating the mixture in four portions "bis zur beginnenden Rotglut" gave a product which crystallized nicely and melted at 73° either alone or mixed with α,β -dinaphthyl. We repeated the oxidation of α -tetralone with selenium dioxide and found the product to be identical in melting point and its spectrum with the

(6) S. C. Hooker, *J. Chem. Soc.*, **61**, 611 (1892); S. C. Hooker and A. D. Gray, *ibid.*, **63**, 424 (1893); S. C. Hooker, *ibid.*, **63**, 1376 (1893); **65**, 15 (1894); **69**, 1355 (1896).

(7) S. C. Hooker, *ibid.*, **69**, 1381 (1896).

(8) S. C. Hooker and W. C. Carvell, *ibid.*, **65**, 76 (1894); S. C. Hooker and J. G. Walsh, Jr., *ibid.*, **65**, 321 (1894); S. C. Hooker and E. Wilson, *ibid.*, **65**, 717 (1894).

(9) L. F. Fieser, M. Z. Nazer, S. Archer, D. A. Berberian, and R. G. Slighter, *J. Med. Chem.*, **10**, 517 (1967).

(10) L. F. Fieser, *J. Am. Chem. Soc.*, **49**, 857 (1927).

(11) S. C. Hooker, *ibid.*, **58**, 1168, 1174 (1936); S. C. Hooker and A. Steyermark, *ibid.*, **58**, 1179, 1198 (1936).

(12) L. F. Fieser, J. L. Hartwell, and A. M. Seligman, *ibid.*, **58**, 223 (1936); L. F. Fieser and M. Fieser, *ibid.*, **70**, 3215 (1948).

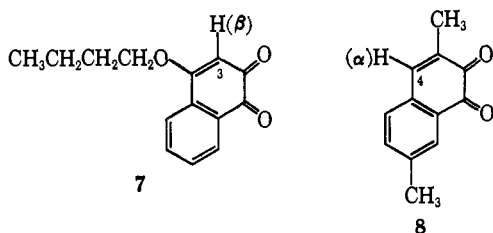
(13) H. Wichelhaus, *Ber.*, **30**, 2199 (1897).

(14) P. G. Copeland, R. E. Dean, and D. McNeil, *J. Chem. Soc.*, 1689 (1960).

(15) F. Weygand and I. Frank, *Ber.*, **84**, 591 (1951).

Wichelhaus product from β -naphthoquinone. Acetylation of the α -tetralone-SeO₂ product by the procedure used by Hooker for the β -naphthoquinone-FeCl₃ product gave an identical crystalline acetate. Combination of this new information with that of Weygand and Frank indicated that the Wichelhaus compound has structure **6** and not **6a**.

Fortunately the structure indicated by the somewhat tenuous evidence cited is neatly confirmed by nuclear magnetic resonance spectroscopy. The spectrum of the Wichelhaus compound, taken in dioxane at 50° relative to hexamethylsiloxane, showed multiplets centered at δ 8.12, 7.85, and 7.42 ppm and a sharp singlet at δ 6.30 ppm. Integration gave a ratio of 8:1 for the sum of the aromatic multiplets to the singlet. In the spectrum of the acetate in CDCl₃ the methyl hydrogen absorption appeared as a singlet at δ 2.18 ppm and a sharp singlet integrating as a single hydrogen appeared at δ 6.24 ppm, well upfield of the remainder of the aromatic absorption, as it had for the Wichelhaus product itself. Clearly the electronic environment of one particular proton has nicely singled it out from the rest. Examination of the alternative structures **6** and **6a** shows that the β -quinonoid 3'-H of **6** and the 4'-H of **6a** (and only these protons) would be expected to have a chemical shift removed from the remainder of the aromatic system and not coupled with other proton signals. To determine what positions such protons occupy, we took 4-*n*-butoxy-1,2-naphthoquinone¹⁶ (**7**) as a model of **6** and 3,7-dimethyl-1,2-naphthoquinone¹⁷ (**8**) as a model of **6a**. The spectrum of **7** showed a



shielded singlet at 5.83 ppm,¹⁸ corresponding to **6**; that of **8** showed none. The nmr evidence thus supports formula **6** for the Wichelhaus "oxide."

Structures previously suggested² for compounds A-F on the basis of formula **6a** clearly call for revision, and the structures in some instances can be evaluated with the aid of nmr data not before available. Compound A, the first transformation product, had been obtained by Hooker by allowing an alkaline solution of the hydroxydinaphthylidiquinone **6** to stand exposed to air for 2 weeks and acidifying. An improved method now reported consists in oxidation with hydrogen peroxide in acetic acid, and we postulate that the initial step is cleavage of the quinonoid ring of **6** between the two carbonyl groups to give the dicarboxylic acid **9**. Karrer and Schneider¹⁹ oxidized β -naphthoquinone with perbenzoic acid in chloroform at room temperature for 5 days, removed acidic material, and isolated the anhydride of the diacid corresponding to **9**, *o*-carboxyallocinnamic

(16) L. F. Fieser, *J. Am. Chem. Soc.*, **48**, 2922 (1926).

(17) L. F. Fieser and A. M. Seligman, *ibid.*, **56**, 2690 (1934).

(18) Data for other aromatic ethers suggests that shielding from the 4-*n*-butoxy group is negligible; see N. S. Bhacca, L. F. Johnson, and J. W. Shoolery, *NMR Spectra Catalog*, Varian Associates, Palo Alto, Calif., 1962.

(19) P. Karrer and L. Schneider, *Helv. Chim. Acta*, **30**, 859 (1947).

acid. Compound A, the yellow, soda-soluble oxidation product isolated by Hooker, has the formula C₂₀H₁₂O₇ as required for **9** but on treatment with diazomethane it afforded a dimethyl and not a trimethyl derivative. Thus the unsaturated diacid **9** must have cyclized to the lactone acid **10**. The two acidic groups of this substance both respond to treatment with diazomethane to give a dimethyl derivative, but on Fischer esterification the 3-substituted 2-hydroxy-1,4-naphthoquinone group, like others of its kind,²⁰ remains unattacked and the product is **12**, the presence in which of a free hydroxyl group is shown by conversion to the acetate methyl ester **13**.

The infrared spectrum of compound A in Nujol shows strong hydroxyl absorption at 3.05 μ , a singlet carbonyl absorption at 5.65 μ attributable to a γ -lactone carbonyl, a singlet at 5.80 μ attributable to a carboxyl carbonyl, and a multiplet at 6.05 μ attributable to the quinone carbonyl groups.

Compound A is sparingly soluble in neutral organic solvents but could be dissolved in pyridine to a concentration sufficient for nmr spectroscopy. A well-defined AB coupling was observed upfield, presumably for the two methylene hydrogens: doublet centers at δ 3.79 and 4.48 ppm and a coupling constant $J = 17$ cps. Such large coupling constants generally are observed only for nonequivalent protons on the same carbon atom; in structure **10** the two protons are nonequivalent because of the adjacent asymmetric center.²¹ Nmr spectrographic characterization of compound A acetate methyl ester (**13**) was also illuminating. The derivative is readily soluble in CDCl₃ and the nmr spectrum (Figure 1) clearly shows the same AB splitting pattern with doublets now centered at δ 2.89 and 4.18 ppm. The acetyl protons yield a singlet at δ 2.26 ppm, and the singlet at δ 3.52 ppm is attributable to the methyl ester protons. Integration yields the ratio of aromatic protons:methyl ester protons:acetyl protons:methylene protons = 8:3:3:2, precisely as expected.

Boiling a solution of compound A in pyridine, best with addition of copper powder, results in smooth decarboxylation to compound C (**14**), probably by reversal of the equilibrium to the unsaturated diacid **9**, decarboxylation, and lactonization. Spectrographic confirmation of formula **14** is as follows: an ir peak at 3.05 μ for the hydroxyl group, one at 5.67 μ for the γ -lactone carbonyl, and one at 6.00 μ for the quinone carbonyls. The nmr spectrum in pyridine shows a sharp singlet at δ 2.30 ppm (δ 2.15 ppm in CDCl₃), integrating as three protons and thus attributable to the methyl group. The decarboxylation can be followed simply by continuous recording of the nmr spectrum of compound A in pyridine as the temperature is raised gradually to 100°. At 60°, even in the absence of copper, one begins to notice a diminution of the methylene AB doublet and simultaneous appearance of the methyl singlet. At 100° (after about 10 min) the doublets have disappeared and the singlet has reached maximal height.

Compound A (**10**), its derivatives **12** and **13**, and compound C (**14**) all have the yellow color characteristic of 1,4-naphthoquinones other than those having α,β -

(20) L. F. Fieser, *J. Am. Chem. Soc.*, **48**, 2922 (1926).

(21) For a discussion of this point see F. A. Boverly, *Chem. Eng. News*, **43**, 111 (April 5, 1965).

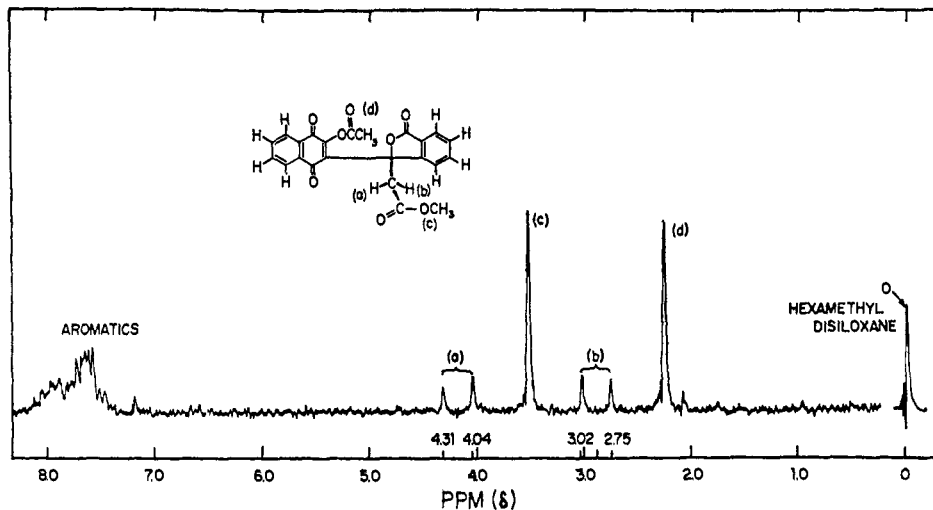
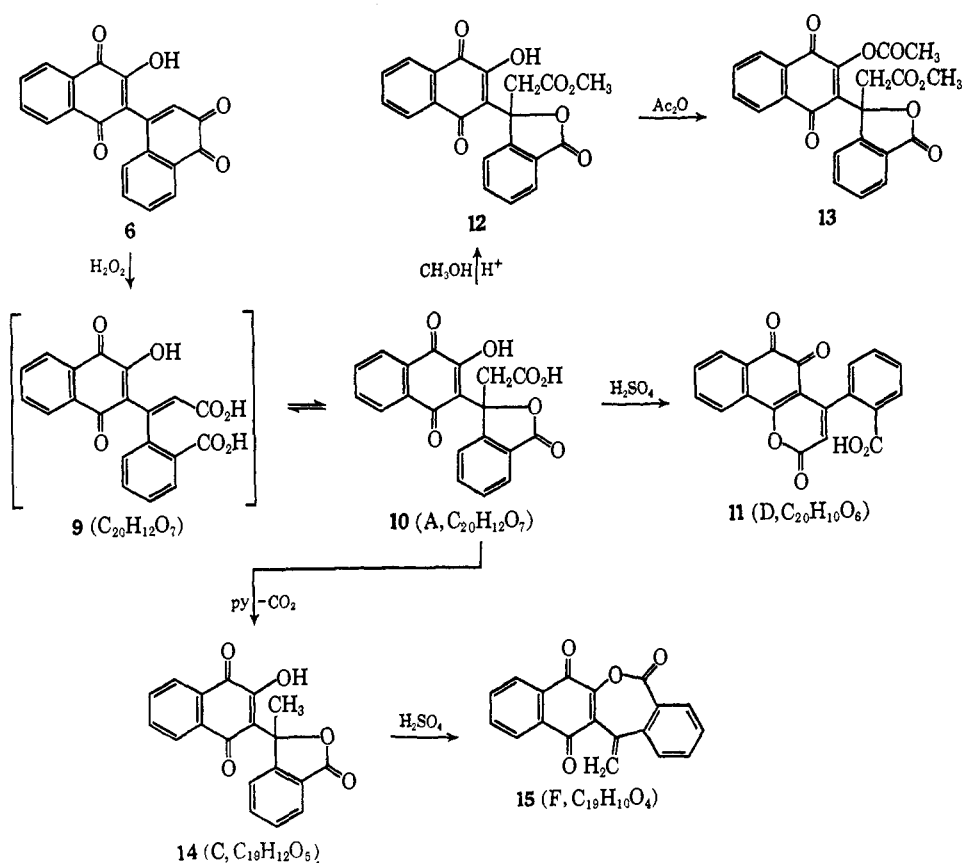


Figure 1.



unsaturated substituents (1) or amino groups, which, like *o*-quinones, are red or orange. Two transformation products of 6 belong to this second group. Compound D ($C_{20}H_{10}O_6$) results on dissolving A ($C_{20}H_{20}O_7$) in concentrated sulfuric acid and pouring the solution into water. The compound has the highly characteristic property of forming a colorless, water-soluble sodium bisulfite addition product and hence can be formulated as the *o*-quinone 11. Confirmatory evidence is that the nmr spectrum taken in trifluoroacetic acid shows a sharp singlet near δ 6 ppm, attributable to the vinylic proton. The preferential formation of the *o*-quinone, where the *para* isomer is also possible, conforms to the observation of Hooker⁸ that β -lapachone is more stable in concentrated sulfuric acid than

α -lapachone, a relationship accounted for by Ettlinger's finding that β -lapachone is a stronger base than α -lapachone by about 2 p*K*_b units.²²

The second nonyellow product, F, is a red, neutral compound of the formula $C_{19}H_{10}O_4$. It is isomeric with compound C and prepared by pouring a solution of C in concentrated sulfuric acid into water. Compound F could not be dissolved in sodium bisulfite solution and hence does not appear to be an *o*-quinone. We suggest that F is the lactone 15 and that the red color is attributable to the α,β -unsaturation of the side chain.

Two additional yellow quinones were described in the earlier work.¹ Refluxing compound A in acetic

(22) M. G. Ettlinger, *J. Am. Chem. Soc.*, 72, 3090 (1950).

acid effects isomerization to compound B and refluxing B in xylene effects elimination of a molecule of water to give compound E. Data now at hand do not suffice for unequivocal elucidation of the structures.

Experimental Section

Preparation of the "Wichelhaus Oxide" (2-Hydroxy-3,4'-dinaphthyl-1,4,1',2'-diquinone, 6). The following modifications in the preparation procedure of Hooker and Fieser¹ improve the yield and quality of the product.

a. The crude yellow product must be washed *absolutely* free of chloride. Mere washing of the filter cake was found to be insufficient, for material so obtained always decomposed to some extent on crystallization. Instead, the filter cake from 10 g of β -naphthoquinone is transferred to a large beaker, stirred mechanically with 300 ml of distilled water, and refiltered. This process is repeated four times, when the filtrate should be colorless. Material thus obtained is stable indefinitely, can be recrystallized almost quantitatively, and is analytically pure.

b. Crystallization is accomplished by dissolving 2 g of the above product in 300 ml of acetone, filtering, and heating the yellow solution of what appears to be a complex with 15 ml of concentrated hydrochloric acid in 250 ml of water. The solution turned orange and crystals soon began to separate. After 1 hr, the product, consisting in orange-red needles, was collected, washed with water, and dried at 110° yield; 1.9 g (95%), mp 251–253°.

Compound A (10). **a.** Hooker's procedure¹ for the air oxidation of the hydroxydiquinone 6 in alkaline solution can be replaced by the following simplified procedure with approximately the same yield. A solution of 10 g of 6 in 300 ml of 6 N potassium hydroxide was stirred vigorously in a stream of air until dark red (about 1 hr).

The solution was chilled and acidified with 20 ml of concentrated hydrochloric acid, and a flocculent precipitate was filtered and discarded. The filtrate on standing at room temperature for 5 days deposited a first crop of product which was collected, yield 5.5 g (50%).

b. A procedure which is faster and more efficient and which gives a purer product is as follows. A 250-ml erlenmeyer flask containing a suspension of 5 g of 6 in 75 ml of acetic acid was stirred magnetically in a water bath kept at 65° and 6 ml of 30% hydrogen peroxide was added in 1-ml portions over a period of 1 hr. The mixture was stirred for 1 hr longer and then cooled. The product, which separated as a bright yellow solid, was collected, washed with acetic acid, and dried at 100°. As judged by melting point, spectrum, and analysis, it was directly pure, yield 4.5 g.

Compound A Methyl Ester (12). A suspension of 0.5 g of compound A in 50 ml of methanol was treated with 15 drops of concentrated sulfuric acid and refluxed for 1 hr, by which time a clear yellow solution had resulted. The condenser was removed and methanol allowed to distill off until the volume had been reduced to about 5 ml. The product which separated on cooling was recrystallized from methanol and gave 0.4 g of yellow crystals, mp 197° gassing.

Anal. Calcd for C₂₁H₁₄O₇ (378.32): C, 66.67; H, 3.73. Found: C, 66.87; H, 3.89.

Compound A Methyl Ester Acetate (13). A 0.3-g sample of the methyl ester was stirred with a solution of ten drops of concentrated sulfuric acid in 19 ml of acetic anhydride until dissolved. After standing for 1 hr at room temperature, water (25 ml) was added with vigorous stirring with cooling when required. A pale yellow crystallize separated and was collected, washed well with water, and dried at 100°; yield 0.3 g, mp 213–214° (directly pure).

Anal. Calcd for C₂₃H₁₆O₈ (420.36): C, 65.71; H, 3.84. Found: C, 65.43; H, 3.37.

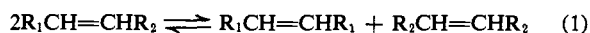
Olefin Metathesis. I. Acyclic Vinylenic Hydrocarbons¹

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Contribution No. 396 from the Research Division, The Goodyear Tire and Rubber Company, Akron, Ohio 44316. Received January 30, 1968

Abstract: Olefin metathesis is a new reaction of olefins and is effected by catalysts derived from WCl₆, C₂H₅OH, and C₂H₅AlCl₂. The major product from the metathesis of 2-butene with 2-butene-*d*₈ was C₄H₄D₄ which supports a scheme of interchange of alkylidene groups. The reaction mixture from the metathesis of 2-pentene with 6-dodecene contained all six olefins predicted by the proposed transalkylidenation scheme, and the absence of other olefins indicated that side reactions involving double-bond migration do not occur. The random statistical composition attainable in these reactions indicates that they are essentially thermoneutral, *i.e.*, entropy controlled. *cis-trans* equilibria are attained as a consequence of the transalkylidenation step itself which is relatively nonselective in the formation of geometrical isomers. A three-step sequence is proposed involving bisolefin-metal complex formation, transalkylidenation, and olefin exchange.

Recently, a new catalytic process was disclosed^{2,3} whereby vinylenic olefins, when treated with a catalyst combination of WCl₆, C₂H₅OH, and C₂H₅AlCl₂, undergo a unique metathetic transformation.



It was found that this reaction, designated olefin metathesis, proceeds rapidly under mild conditions and provides a highly effective means of interchanging alkylidene species.

(1) Presented in part before the Division of Organic Chemistry, 154th National Meeting of the American Chemical Society, Chicago, Ill., Sept 1967, Abstracts, S172.

(2) N. Calderon, H. Y. Chen, and K. W. Scott, *Tetrahedron Letters*, 3327 (1967).

(3) *Chem. Eng. News*, 45, 51 (Sept 25, 1967).

A somewhat related process, designated olefin dismutation or disproportionation, has been reported^{4,5} wherein at elevated temperatures the isomerization and disproportionation of olefins over solid bed, mixed metal oxide catalysts leads to a series of homologous products of lower and higher molecular weights. For the particular dismutation of propylene to form ethylene and 2-butene good selectivity was attainable with equilibrium apparently established among the ethylene, propylene, and butene components of the system. When the dismutation of higher olefins was attempted,

(4) R. L. Banks and G. C. Bailey, *Ind. Eng. Chem., Prod. Res. Develop.*, 3, 170 (1964).

(5) C. P. C. Bradshaw, E. J. Howman, and L. Turner, *J. Catalysis*, 7, 269 (1967).