[CONTRIBUTION FROM THE NOYES CHEMICAL LABORATORY, UNIVERSITY OF ILLINOIS]

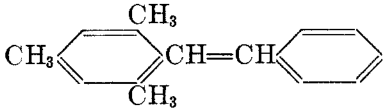
HIGHLY HINDERED STILBENES

REYNOLD C. FUSON, J. J. DENTON, AND CHRIS E. BEST

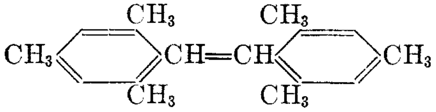
Received October 5, 1942

The hindering influence of *ortho* substituents is generally associated with reactions that are additive in type and has been noted frequently in studies of aldehydes, ketones, esters, nitriles, and other compounds in which the functional group contains a multiple linkage between carbon and a negative element. Very little attention, however, has been given to the influence of *ortho* substituents on the addition reactions of the carbon-to-carbon multiple linkages. To this end a study has been made of the effect of *ortho* substituents on the reactions of stilbene.

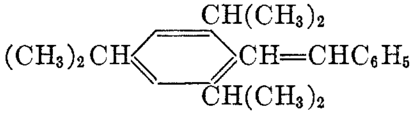
Three new stilbenes containing *ortho* substituents have been prepared and treated with reagents known to react with the central linkage of stilbene. The compounds selected for the work were 1-mesityl-2-phenylethylene (I), 1,2-dimesitylethylene (II), and 1-phenyl-2-(2,4,6-triisopropylphenyl)ethylene (III).



I



II



III

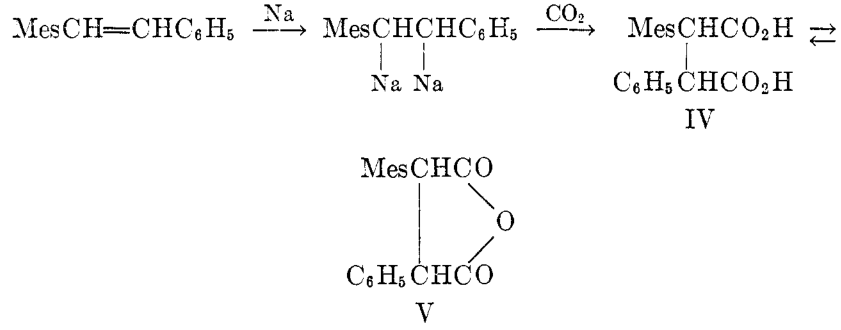
REACTIONS OF THE STILBENES

The three new stilbenes were found to undergo hydrogenation to the corresponding 1,2-diarylethanes when treated with hydrogen in the presence of Raney nickel.

Ozone cleaved the hindered stilbenes to produce the corresponding acids. From mesitylphenylethylene and dimesitylethylene small amounts of mesitol were also obtained. This unexpected result is reminiscent of the production of phenols by the autoxidation of certain vinyl alcohols (1).

Perbenzoic acid converted 1-mesityl-2-phenylethylene to the epoxide, which could be reconverted to the olefin by treatment with hydriodic acid in glacial acetic acid. A similar result could not be obtained with dimesitylethylene. The action of perbenzoic acid on the mesityl compounds appeared to be complex, and it was suspected that the mesityl radical was attacked. As a test of this suggestion, mesitylene itself was treated with perbenzoic acid. A large part of the mesitylene was acted upon by the reagent and from the products mesitol could be isolated.

Perhaps the most interesting reaction of the stilbenes was with metallic sodium. It had been shown that *trans*-stilbene, when treated successively with sodium, carbon dioxide, and water, yielded α,β-diphenylsuccinic acid (2). 1-Mesityl-2-phenylethylene behaved in a similar manner, yielding α-mesityl-β-phenylsuccinic acid (IV). The hexamethyl and the triisopropyl stilbenes failed to react under these conditions. The corresponding succinic acids were prepared from these olefins, however, by the use of a liquid sodium-potassium alloy, followed by carbonation. The anhydride (V) of α-mesityl-β-phenylsuccinic acid was prepared by heating with acetyl chloride. The acid was regenerated by treating the anhydride with water.



$$\text{MesCH}{=}\text{CHC}_6\text{H}_5 \xrightarrow{\text{Na}} \underset{\text{Na}\ \ \text{Na}}{\text{MesCHCHC}_6\text{H}_5} \xrightarrow{\text{CO}_2} \begin{array}{c}\text{MesCHCO}_2\text{H}\\ |\\ \text{C}_6\text{H}_5\text{CHCO}_2\text{H}\end{array} \rightleftarrows$$

IV

V

Solutions of potassium permanganate were decolorized slowly by the hindered olefins. The triisopropyl stilbene reacted less rapidly than the trimethyl and the hexamethyl was most sluggish of all. Stilbene itself was more reactive than any of the hindered olefins. These results are in harmony with the observation that 1,1-dineopentylethylene is not oxidized by permanganate solutions (3).

Treatment of 1,2-dimesitylethylene with silver benzoate and iodine by the method of Prévost (4) followed by hydrolysis yielded a mixture of the corresponding glycols. 1-Mesityl-2-phenylethylene appeared to react in a similar manner.

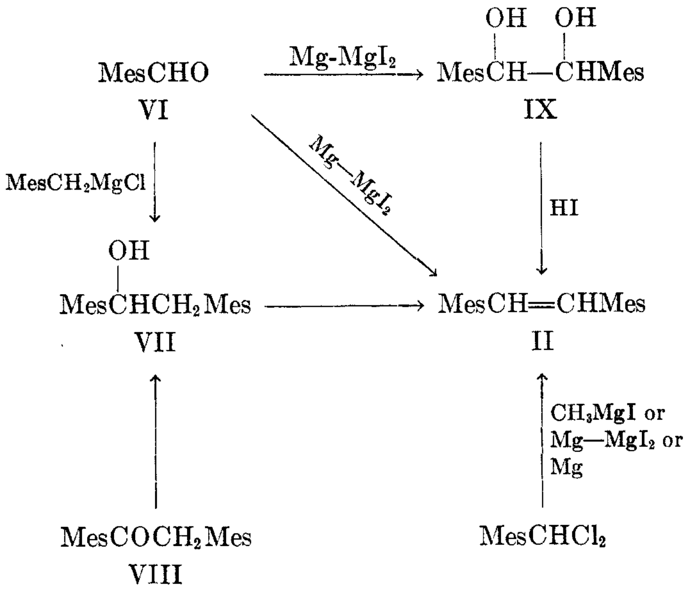
SYNTHESIS OF THE STILBENES

1,2-Dimesitylethylene. The hexamethylstilbene (I) was first made by the coupling action of methylmagnesium iodide on mesital chloride. It was to be expected by analogy with the coupling of benzal chloride (5) that the stilbene dichloride would be formed. However, the only product that could be isolated was 1,2-dimesitylethylene. The yield was about 30%.

The new stilbene was assumed to be the *trans* form because of its close resemblance to *trans*-stilbene. Moreover, other methods of synthesis yielded this form, suggesting that it is the more stable modification. One of the methods consisted in effecting the coupling of mesital chloride with the binary mixture, Mg-MgI_2 (6). Magnesium alone was found to produce the stilbene also, but the product of this reaction was difficult to purify.

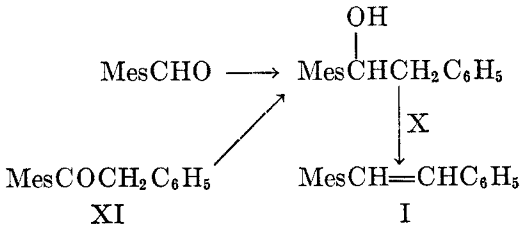
The most satisfactory method for preparing the hexamethylstilbene was by the dehydration of 1,2-dimesitylethanol (VII). The dehydration was effected in nearly quantitative yields by treating the alcohol with acetic anhydride in the presence of a small amount of hydrogen chloride. When the dehydration was carried out with sulfuric acid or phosphorus pentoxide the product was contaminated with a high-melting solid which appeared to be the di(α,β-dimesitylethyl) ether. Acetic anhydride alone produced the acetate of the alcohol.

1,2-Dimesitylethanol (VII) was prepared by high-pressure hydrogenation of desoxymesitoin (VIII) and by the condensation of mesitaldehyde (VI) with 2,4,6-trimethylbenzylmagnesium chloride.



Another method of preparing the stilbene (II) consisted in treating mesitaldehyde with the binary mixture, Mg-MgI$_2$ (7). The principal product of the reduction, dimesitylethylene glycol (IX), could also be converted to the stilbene by treatment with hydrogen iodide in glacial acetic acid.

1-Mesityl-2-phenylethylene. The trimethylstilbene (I) was prepared by dehydration of 1-mesityl-2-phenylethanol (X). This alcohol in turn could be produced either by high-pressure hydrogenation of benzyl mesityl ketone (XI) or by the condensation of mesitaldehyde with benzylmagnesium chloride.



The dehydration was effected in high yield by treatment with sulfuric acid. By analogy with stilbene, this hydrocarbon was assigned the *trans* configuration.

1-Phenyl-2-(triisopropylphenyl)ethylene (III). The triisopropylstilbene was prepared by dehydration of the corresponding alcohol. It was assumed to have the *trans* configuration. Experience has shown that the 2,4,6-triisopropylphenyl group offers much more hindrance than the mesityl radical. 1-Phenyl-2-(triisopropylphenyl)ethylene would accordingly be expected to be much less reactive than the mesityl analog.

EXPERIMENTAL

1,2-Dimesitylethylene (from mesital chloride). This hydrocarbon was made by coupling mesital chloride, prepared by the method of Asinger and Lock (8). The coupling was carried out by three different reagents—methylmagnesium iodide, the binary mixture (Mg-MgI_2), and magnesium alone. The yields are nearly the same with the three reagents but the use of the Grignard reagent is to be preferred because it gives a product that is easily purified.

A. *Methylmagnesium iodide*. The method was essentially that used with benzal chloride (5). From 30 g. of mesital chloride was obtained 5.8 g. of the ethylene. It was purified by recrystallization from ethanol; m.p. 132.5–133.5°.

Anal[1]. Calc'd for $C_{20}H_{24}$: C, 90.84; H, 9.16.
Found: C, 90.74; H, 9.21.

Removal of the solid olefin left 12.79 g. of a red oil; it was not investigated further.

B. *The binary mixture, Mg-MgI_2*. The reagent was prepared from 2.6 g. of magnesium and 12.79 g. of iodine in the usual manner (6). A solution of 10 g. of mesital chloride in 10 cc. of ether was added dropwise over a period of thirty minutes. The heat liberated caused the solution to boil. The mixture was stirred throughout the addition of the chloride and for two hours longer. During the last hour, refluxing was maintained by heating. An iodine color developed, then faded; a solid separated from the solution.

The reaction mixture was poured on a mixture of ice and hydrochloric acid, the ether-benzene layer was removed, and the water layer was extracted once with ether. The ether-benzene was united with the ether extract and washed once with sodium bicarbonate solution, twice with sodium bisulfite solution, and finally with water. The solution was dried over magnesium sulfate and concentrated to a small volume by heating on a steam-cone. The concentrate was mixed with ethanol and cooled; 2.1 g. of the ethylene was deposited. Concentration of the mother liquors produced 5 g. of a dark viscous oil.

C. *Magnesium*. A solution of 10 g. of mesital chloride in 50 cc. of dry ether was added dropwise over a period of one hour to a mixture of 2.5 g. of magnesium turnings and 75 cc. of dry ether. The reaction was initiated by the addition of a few drops of methyl iodide along with the first few drops of the mesital chloride solution. The reaction liberated heat and caused a precipitate to form. Stirring was continued during the mixing and for two hours longer. An additional 50 cc. of ether was added to facilitate stirring. After the addition was completed, the mixture was maintained at room temperature for an hour, then heated under reflux for an hour. It was poured on an ice-hydrochloric acid mixture, and the product isolated in a manner similar to that already described. The ethylene was induced to crystallize by dissolving it in high-boiling petroleum ether and cooling the solution with solid carbon dioxide; m.p. 114–121°; yield 2 g. It was purified by recrystallization alternately from methanol and from high-boiling petroleum ether; m.p. 131–132°. Six recrystallizations were necessary.

1,2-Dimesitylethanol. A. *From desoxymesitoin*. A mixture of 28 g. of desoxymesitoin, 140 cc. of absolute ethanol, and 5 g. of copper chromite was subjected to a hydrogen pres-

---

[1] Microanalyses by Miss Margaret McCarthy, Miss Theta Spoor, and Miss Dorothy Schneider.

sure of 2000 lbs. at 125°.[2] A mole of the gas was absorbed in thirty minutes. The catalyst was removed by filtration and the filtrate treated with Norit. When concentrated to 80 cc. and allowed to stand in the ice-chest overnight, the solution deposited 26 g. of 1,2-dimesitylethanol; m.p. 128–129°.

Anal. Calc'd for $C_{20}H_{26}$: C, 85.06; H, 9.29.
Found: C, 84.92; H, 9.17.

B. *From mesitaldehyde and 2,4,6-trimethylbenzylmagnesium chloride.* The Grignard reagent was prepared by adding, over a period of two and one-half hours, a solution of 13 g. of 2,4,6-trimethylbenzyl chloride in 400 cc. of dry ether to a mixture of 18.8 g. of magnesium turnings, and 170 cc. of dry ether. The reaction mixture was stirred and kept at ice-bath temperature during the mixing and for two hours longer. It was then allowed to come to room temperature. A solution of 7.4 g. of mesitaldehyde in 200 cc. of dry ether was added, with stirring, over a period of one and one-fourth hours. Stirring was discontinued and the mixture allowed to stand overnight. It was poured on a mixture of ice and acetic acid. The ether layer was removed and the water layer extracted with ether. The two ether solutions were united, dried over magnesium sulfate, and concentrated to a small volume. When cooled, this solution deposited 15.4 g. of colorless solid material melting at 109–114°. Tedious fractional crystallization from methanol separated it into 1,2-dimesitylethanol and 1,2-dimesitylethane.

1,2-Dimesitylethyl acetate. A mixture of 2 g. of 1,2-dimesitylethanol and 20 cc. of acetic anhydride was heated overnight under reflux, cooled, and poured into water. The acetate was isolated by filtration and purified by recrystallization from methanol; m.p. 117.5–118°; yield 1.6 g.

Anal. Calc'd for $C_{22}H_{28}O_2$: C, 81.43; H, 8.70.
Found: C, 81.28; H, 8.82.

The acetate also was made by acetylation with acetyl chloride.

Dimesitylethylene (from 1,2-dimesitylethanol). The dehydration of the ethanol could not be effected by heat alone. The alcohol was unchanged after two hours at 220°.

A. *Sulfuric acid.* A mixture of 2 g. of the ethanol, 10 cc. of concentrated sulfuric acid, and 10 cc. of water was heated on the steam-cone, with occasional shaking, for eighteen hours. It was cooled and poured on 200 g. of ice and water. The solid product was removed by filtration and recrystallized from 500 cc. of methanol. It was then recrystallized successively from high-boiling petroleum ether, ethanol, and a mixture of dioxane and ethanol; m.p. 177–180°. It was believed to be di(α,β-dimesityl)ethyl ether.

Anal. Calc'd for $C_{40}H_{50}O$: C, 87.91; H, 9.15.
Found: C, 87.83; H, 8.94.

The original methanolic filtrate was concentrated to a volume of 50 cc. and cooled; 1.0 g. of impure 1,2-dimesitylethylene (m.p. 127–128°) separated.

B. *Phosphorus pentoxide.* A mixture of 5 g. of the ethanol, 15 g. of phosphorus pentoxide, and 150 cc. of benzene was heated under reflux for three and one-half hours. The pentoxide was removed by filtration and the solvent evaporated. The residue was extracted with 1 liter of boiling ethanol. One gram of solid remained undissolved and, from its melting point (155–165°), was assumed to be the ether. When the ethanol solution was concentrated to a volume of 300 cc. and cooled, 2.5 g. of impure 1,2-dimesitylethylene (m.p. 122–128°) was obtained.

C. *Acetic anhydride and hydrochloric acid.* A mixture of 10 g. of the ethanol, 2 cc. of concentrated hydrochloric acid, and 50 cc. of acetic anhydride was heated under reflux for seven hours and poured into water. The ethylene was isolated by filtration and recrystallized from ethanol; yield 8 g.; m.p. 128–130°.

1-Mesityl-2-phenylethanol. A. *From mesitaldehyde and benzylmagnesium chloride.* The Grignard reagent was prepared by adding a solution of 49.2 g. of benzyl chloride in 50 cc.

---

[2] The high-pressure hydrogenations were carried out by Mr. J. C. Robinson, Jr. and Mr. John M. Stewart.

of dry ether to a mixture of 9.3 g. of magnesium turnings and 50 cc. of dry ether. A solution of 44.4 g. of mesitaldehyde in 50 cc. of dry ether was added to this, with stirring. In order to facilitate stirring, 50 cc. more of dry ether was added. The mixture was then heated under reflux for thirty minutes. It was cooled and 100 cc. of ice-cold dilute hydrochloric acid was added to it dropwise, with stirring. The layers were separated and the aqueous layer extracted with ether. The combined ether extracts were washed successively with dilute hydrochloric acid, water, and 10% sodium hydroxide solution. The ether solution was dried over magnesium sulfate and the solvent removed by heating on a steam-cone. The oily residue was diluted with an equal volume of high-boiling petroleum ether and cooled. Scratching the beaker induced the precipitation of 52.1 g. of a white solid; m.p. 62–66°. This melted at 65–66° after two recrystallizations from dilute methanol.

Anal. Calc'd for $C_{17}H_{20}O$: C, 85.00; H, 8.33.
Found: C, 85.10; H, 8.30.

B. *By catalytic reduction of benzyl mesityl ketone.* To a solution of 47.6 g. of the ketone in methanol made up to 150 cc. was added 6 g. of copper chromite. The mixture was subjected to a hydrogen pressure of 1,550 lbs. at 150° for three hours. The yield of the ethanol (m.p. 65–66°) was 27.3 g.

1-Mesityl-2-phenylethylene. A mixture of 75 g. of the ethanol, 375 cc. of sulfuric acid, and 375 cc. of water was heated, with occasional shaking, on a steam-cone for twenty-four hours. The olefin was isolated and purified by the method described for the hexamethylstilbene. The product melted at 55–56°; yield 86%.

Anal. Calc'd for $C_{17}H_{18}$: C, 91.81; H, 8.11.
Found: C, 91.69; H, 8.17.

1-Phenyl-2-(triisopropylphenyl)ethanol. This compound was prepared by the method outlined for 1-mesityl-2-phenylethanol. From 69.6 g. of 2,4,6-triisopropylbenzaldehyde was obtained 75 g. of product melting at 108–111°. After repeated recrystallization from high-boiling petroleum ether the alcohol melted at 117.5–118.5°.

Anal. Calc'd for $C_{23}H_{32}O$: C, 85.19; H, 9.88.
Found: C, 85.09; H, 9.68.

2-Phenyl-1-(triisopropylphenyl)ethylene. This hydrocarbon was prepared by dehydrating the alcohol with sulfuric acid as described for 1-mesityl-2-phenylethylene. Four grams of the ethanol yielded 2.5 g. of the olefin, melting at 79–80°. Repeated recrystallization produced a pure sample; m.p. 82.5–83.5°.

Hydrogenation. A mixture of 1 g. of 1,2-dimesitylethylene, 10 cc. of methylcyclohexane, and 1 g. of a Raney nickel catalyst was heated at 100° for one hour under a hydrogen pressure of 2000 lbs. The temperature was then raised to 150° and maintained at that point for one-half hour. The product was purified by repeated recrystallization from ethanol; m.p. 114–117°. It was shown by the mixed melting point method to be *1,2-dimesitylethane* (9).

Hydrogenation of 1-mesityl-2-phenylethylene at 100° by a similar procedure yielded *1-mesityl-1-phenylethane.* It crystallized from methanol in beautiful, white needles; m.p. 38–39°.

Anal. Calc'd for $C_{17}H_{20}$: C, 91.01; H, 8.98.
Found: C, 90.90; H, 9.22.

1-Phenyl-2-(2,4,6-triisopropylphenyl)ethylene was hydrogenated by the foregoing procedure. The *1-phenyl-2-(2,4,6-triisopropylphenyl)ethane* was purified by distillation followed by recrystallization from methanol; b.p. 155–161° (4 mm.); m.p. 33–34°.

Anal. Calc'd for $C_{23}H_{32}$: C, 89.54; H, 10.46.
Found: C, 89.55; H, 10.29.

Ozonolysis. A stream of oxygen containing approximately 1% ozone was passed for three hours into a solution of 2 g. of *1,2-dimesitylethylene* in 50 cc. of chloroform. The solvent was evaporated under reduced pressure and the residue treated with 100 cc. of 3% hydrogen peroxide solution. The mixture was boiled for five minutes; 10 cc. of 20% sodium hydroxide was then added slowly. The resulting solution was filtered and treated with solid carbon dioxide. The solid which formed was extracted with ether and purified by low-pressure sublimation; m.p. 69–71°; yield 0.04 g. This compound was shown by the mixed melting point method to be *mesitol.*

The aqueous solution remaining after removal of the mesitol was freed from dissolved ether and acidified. The precipitated mesitoic acid weighed 0.7 g. It was recrystallized from high-boiling petroleum ether, then from water; m.p. 150–151°. The melting point was not depressed by admixture of the sample with a known specimen of mesitoic acid.

1-Mesityl-2-phenylethylene was ozonized by a similar procedure. By suitable methods the product was separated into three components: mesitol, mesitoic acid, and benzoic acid.

The mesitoic acid was obtained in pure form by extracting the mixture with hot water and recrystallizing the residue from high-boiling petroleum ether. The extract, when cooled, yielded a mixture of the two acids; m.p. 88–91°. Recrystallization of this material from water or from high-boiling petroleum ether did not raise the melting point. Benzoic acid was obtained pure by partially subliming the material at low pressure and recrystallizing the sublimate from high-boiling petroleum ether.

In a similar manner *1-phenyl-2-(2,4,6-triisopropylphenyl)ethylene* was converted by ozonolysis to a mixture of benzoic and 2,4,6-triisopropylbenzoic acids. The benzoic acid was extracted with hot water and the residual triisopropylbenzoic acid was recrystallized from high-boiling petroleum ether; m.p. 180–182°.

1-Mesityl-2-phenylethylene oxide. To a chloroform solution of perbenzoic acid, containing 0.06 mole of the reagent, was added 6.7 g. (0.03 mole) of the ethylene. After being allowed to stand in the ice-box for twenty-four hours, the solution was washed with water solutions of sodium thiosulfate and potassium bicarbonate. Removal of the solvent left the crude oxide as an oil, which solidified on cooling. One recrystallization from methanol gave 4.6 g. of yellow crystals melting at 64–66°. The color was removed with Norit. Recrystallization from dilute methanol yielded white plates; m.p. 67–68°.

Anal. Calc'd for $C_{17}H_{18}O$: C, 85.71; H, 7.56.
Found: C, 86.00; H, 7.75.

The oxide could be reduced to the olefin with hydrogen iodide. A solution of 1 g. of the oxide, 10 cc. of hydriodic acid (sp. gr. 1.5), and 30 cc. of glacial acetic acid was heated for ninety minutes on a steam-cone and poured into water. The product was the olefin; m.p. 53–54°.

Action of perbenzoic acid on mesitylene. Thirty-six grams (0.3 mole) of mesitylene was added to a chloroform solution of perbenzoic acid containing 0.15 mole of the reagent. After being allowed to stand 112 hours, the solution was washed with solutions of sodium thiosulfate and potassium carbonate and then with water. Repeated extraction with 10% sodium hydroxide solution gave 3.8 g. of *mesitol* melting at 67–70°. This was a yield of 18.6%.

α-Mesityl-β-phenylsuccinic acid. A mixture of 10 g. of powdered sodium, 4 g. of 1-mesityl-2-phenylethylene, and 200 cc. of ether was shaken for five hours. It developed a dark red color and finally deposited a finely divided red-brown precipitate. The introduction of solid carbon dioxide discharged the color and left a yellow precipitate. The solution and precipitate were decanted from the sodium into 300 cc. of water. Acidification of the water layer gave 1.9 g. of product melting at 210–215°. The α-mesityl-β-phenylsuccinic acid was recrystallized from dilute acetic acid; m.p. 217–219°.

Anal. Calc'd for $C_{19}H_{20}O_4$: C, 73.08; H, 6.41; neut. equiv., 156.
Found: C, 72.89; H, 6.69; neut. equiv., 153.

The *anhydride* was made by heating 0.5 g. of the acid for two and one-half hours with 10 cc. of acetyl chloride. After the volatile portion of the mixture had been distilled under reduced pressure the residue was induced to crystallize by allowing it to stand overnight with high-boiling petroleum ether. The anhydride was recrystallized from this solvent; m.p. 129–130°.

Anal. Calc'd for $C_{19}H_{18}O_3$: C, 77.55; H, 6.17.
Found: C, 77.42; H, 6.14.

Treatment with hot 2% sodium hydroxide solution, followed by acidification, regenerated the parent acid.

α,β-Dimesitylsuccinic acid. A solution of 4 g. of 1,2-dimesitylethylene in 250 cc. of dry ether was added to an alloy of 4 g. of sodium and 6 g. of potassium. The mixture was shaken for six hours; it developed a black color. A stream of carbon dioxide was passed in until the black color had been completely discharged. The ether and suspended white solid were decanted from the excess alloy and washed twice with water. The aqueous solution was heated to drive off dissolved ether and acidified with hydrochloric acid. The crude acid melted at 250–270° and weighed 4.6 g. It was recrystallized from ethyl benzoate; m.p. 283–285°.

Anal. Calc'd for $C_{22}H_{26}O_4$: C, 74.58; H, 7.40; neut. equiv., 177.
Found: C, 74.65; H, 7.71; neut. equiv., 177.5.

α-Phenyl-β-(2,4,6-triisopropylphenyl)succinic acid. By the procedure outlined for the dimesitylsuccinic acid, 4 g. of 1-phenyl-2-(2,4,6-triisopropylphenyl)ethylene was converted to 3.6 g. of crude α-phenyl-β-(2,4,6-triisopropylphenyl)succinic acid. It was purified by recrystallization from glacial acetic acid; m.p. 195–198°.

Anal. Calc'd for $C_{25}H_{32}O_4$: C, 75.76; H, 8.08; neut. equiv., 196.
Found: C, 75.61; H, 8.22; neut. equiv., 197.

Action of potassium permanganate solutions on the stilbenes. The tests were made by adding 0.1 cc. of a 2% solution of potassium permanganate in water to a solution of 0.05 g. of the stilbene in 4 cc. of acetone and allowing to stand. The time required for discharging the permanganate color was less than one minute for stilbene, four and one-half hours for 1-mesityl-2-phenylethylene, 30 hours for 1-phenyl-2-(2,4,6-triisopropylphenyl)ethylene, and 60 hours for 1,2-dimesitylethylene. 1,2-Dimesitylethane was unaffected by the permanganate in 72 hours.

Action of the silver benzoate-iodine complex on 1,2-dimesitylethylene. The method of Prévost (4) was used. A solution of 3 g. of iodine in 50 cc. of dry benzene was added, with stirring, to a suspension of 5.7 g. of silver benzoate in 50 cc. of dry benzene. To the resulting mixture 2.64 g. of the ethylene in 50 cc. of dry benzene was added. The mixture was heated under reflux for 24 hours and cooled. The silver salts were removed by filtration and the solvent by evaporation. The residue was treated for four hours with a solution of 3 g. of potassium hydroxide in 30 cc. of ethanol and 10 cc. of water. The mixture was extracted twice with ether, and the ether solution dried over sodium sulfate. After evaporation of the solvent, the residue was dissolved in 50 cc. of hot high-boiling petroleum ether. When cooled, the solution deposited 1.1 g. of white crystals melting at 147–170°. Two recrystallizations from methanol yielded the pure high-melting hydromesitoin; m.p. 212–213°.

The solvent was evaporated from the methanol filtrates and the residue recrystallized from high-boiling petroleum ether. The low-melting hydrobenzoin melted at 158–159°. Both glycols were identified by mixed melting point determinations with known samples (7).

Action of the complex on 1-mesityl-2-phenylethylene. By use of the foregoing procedure 2.22 g. of the olefin was partially converted to a solid melting at 120–124°. Recrystallization of the solid from aqueous ethanol raised the melting point to 124–132°. The material was assumed to be a mixture of the expected glycols.

Anal. Calc'd for $C_{17}H_{20}O_2$: C, 79.64; H, 7.87.
Found: C, 79.58; H, 8.03.

SUMMARY

Three hindered stilbenes have been prepared and treated with reagents known to react with the olefin linkage. Reactions with hydrogen, permanganate, ozone, perbenzoic acid, sodium, sodium-potassium alloy, and a silver benzoate-

iodine complex were found to occur normally but often at much slower rates than with the unhindered stilbene.

URBANA, ILL.

REFERENCES

(1) FUSON, BYERS, RACHLIN, AND SOUTHWICK, *J. Am. Chem. Soc.*, **64,** 2886 (1942).
(2) SCHLENK, APPEROD, MICHAEL, AND THAL, *Ber.*, **47,** 475 (1914).
(3) BARTLETT, FRASER, AND WOODWARD, *J. Am. Chem. Soc.*, **63,** 495 (1941).
(4) PRÉVOST, *Compt. rend.*, **196,** 1129 (1933); **197,** 1661 (1933).
(5) FUSON AND ROSS, *J. Am. Chem. Soc.*, **55,** 720 (1933).
(6) GOMBERG AND BACHMANN, *J. Am. Chem. Soc.*, **49,** 237 (1927).
(7) FUSON, HORNING, WARD, ROWLAND, AND MARSH, *J. Am. Chem. Soc.*, **64,** 30 (1942).
(8) ASINGER AND LOCK, *Monatsh.*, **62,** 326 (1933).
(9) WENZEL, *Monatsh.*, **35,** 954 (1914).