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

Vinyl Alcohols. XIX.[1] Mesitylphenylacetaldehyde

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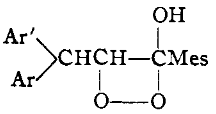
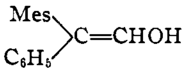
Although a number of stable 2,2-diarylvinyl alcohols are known, efforts to synthesize the corresponding diarylacetaldehydes have been unavailing. In particular, attempts to form the aldehydes by isomerization of the vinyl alcohols have failed; treatment with alcoholic hydrogen chloride, a method that had been used to convert several trisubstituted vinyl alcohols to the corresponding ketones,[2] yielded only vinyl ethers.[3] Attempts to synthesize highly hindered diarylacetaldehydes by the decomposition of peroxides of type I produced the corresponding vinyl alcohols.[4] The only evidence for the existence of such aldehydes was the fact that mesitylphenylvinyl alcohol (II) formed a semicarbazone.[4]

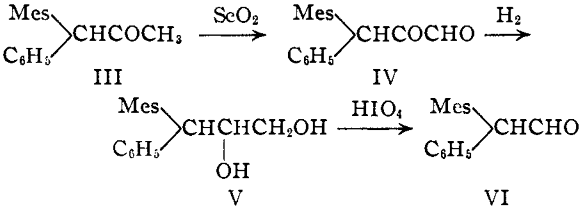

$\mathrm{Ar'(Ar)CHCH{-}C(OH)Mes}$ (with $\mathrm{O{-}O}$ bridge)

I


$\mathrm{Mes(C_6H_5)C{=}CHOH}$

II

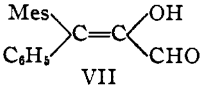
We have now tried various other methods of synthesis and have been successful in preparing an aldehyde of the desired type—mesitylphenylacetaldehyde (VI). It was made first by the oxidative cleavage of 3-mesityl-3-phenyl-1,2-propanediol (V), a hydrogenation product of mesitylphenylpyruvaldehyde (IV), which in turn was prepared by selenium dioxide oxidation of 1-mesityl-1-phenylpropanone (III).[5]


$$\mathrm{Mes(C_6H_5)CHCOCH_3} \xrightarrow{\mathrm{SeO_2}} \mathrm{Mes(C_6H_5)CHCOCHO} \xrightarrow{\mathrm{H_2}} \mathrm{Mes(C_6H_5)CHCH(OH)CH_2OH} \xrightarrow{\mathrm{HIO_4}} \mathrm{Mes(C_6H_5)CHCHO}$$

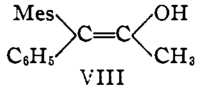
III IV V VI

The oxidation of the propanone (III) afforded a very low yield of the glyoxal and produced in addition mesityl phenyl diketone. This benzyl could be made also by oxidation of the glyoxal (IV) with nitric acid. Oxidation with hydrogen peroxide, however, cleaved the glyoxal, yielding mesitylphenylacetic acid.

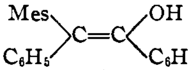
Certain reactions of the glyoxal did not correspond entirely with those to be expected of an α-keto aldehyde. It formed only a mono-oxime and a monosemicarbazone; with acetic anhydride in pyridine it gave an acetate. The glyoxal failed to yield mesitylphenyllactic acid when treated with alkali and could be regenerated by alkaline hydrolysis of the acetate. Moreover, the compound was colorless. In view of these observations it seemed probable that it was to be classed with benzyl phenyl diketone[6] and benzyl mesityl diketone[7] and to be ascribed the enol form, VII. The infrared spectrum of this compound in hexane solution showed absorption maxima at 2.94μ and 6.02μ indicating the presence of a hydroxyl and a conjugated carbonyl group, respectively.[8]


$\mathrm{Mes(C_6H_5)C{=}C(OH)CHO}$

VII


$\mathrm{Mes(C_6H_5)C{=}C(OH)CH_3}$

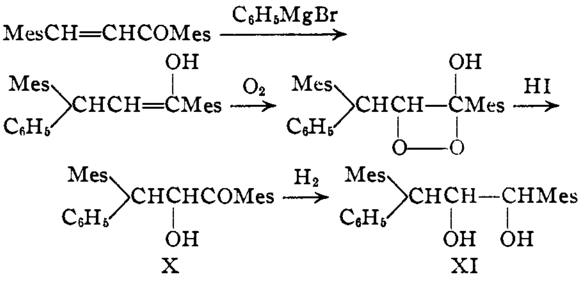
VIII


$\mathrm{Mes(C_6H_5)C{=}C(OH)C_6H}$

IX

This enol is an interesting addition to the list of stable trisubstituted vinyl alcohols, especially in view of the fact that the corresponding enols in which the aldehyde group has been replaced by a methyl (VIII) or a phenyl group (IX)[5] are known to be unstable.

Because of the unsatisfactory yields of glyoxal (IV) other methods of preparation of mesitylphenylacetaldehyde were sought. The only attempt to be brought to a successful conclusion involved the oxidative cleavage of 1,3-dimesityl-3-phenyl-1,2-propanediol (XI) with periodic acid. The glycol was prepared from mesitalacetomesitylene by the following series of changes. The hydroxy ketone (X) failed to be cleaved by periodic acid.


$$\mathrm{MesCH{=}CHCOMes} \xrightarrow{\mathrm{C_6H_5MgBr}} \mathrm{Mes(C_6H_5)CHCH{=}C(OH)Mes} \xrightarrow{\mathrm{O_2}} \mathrm{Mes(C_6H_5)CHCH{-}C(OH)Mes\ (O{-}O)} \xrightarrow{\mathrm{HI}} \mathrm{Mes(C_6H_5)CHCH(OH)COMes} \xrightarrow{\mathrm{H_2}} \mathrm{Mes(C_6H_5)CHCH(OH){-}CH(OH)Mes}$$

X XI

A by-product of the cleavage of the glycol (XI) was 2,4,6-trimethylbenzoin. Since this benzoin is known to be produced by the action of ozone on 2-mesityl-2-phenylvinyl alcohol[3] it seems probable that it was formed from mesitylphenylacetaldehyde, perhaps by way of the vinyl alcohol. Attempts were made to extend the synthetic method

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(1) For the preceding article in this series see Fuson, Chadwick and Ward, THIS JOURNAL, **68**, 389 (1946).

(2) Fuson, Byers and Rabjohn, *ibid.*, **63**, 2639 (1941).

(3) Fuson, Rabjohn and Byers, *ibid.*, **66**, 1272 (1944).

(4) Fuson, Maynert and Shenk, *ibid.*, **67**, 1939 (1945).

(5) Fuson, Armstrong, Chadwick, Kneisley, Rowland, Shenk and Soper, *ibid.*, **66**, 1464 (1944).

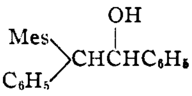
(6) Kohler and Barnes, *ibid.*, **56**, 211 (1934).

(7) Barnes, *ibid.*, **57**, 937 (1935).

(8) The authors are indebted to Dr. F. A. Miller and Mrs. J. L. Johnson for the measurement and interpretation of the infrared spectrum.

to other aldehydes but without success. When 1,3-dimesityl-3-*p*-tolyl-1,2-propanediol was treated with periodic acid, mesityl *p*-tolylacetic acid was formed.

Mesitylphenylacetaldehyde gave a positive Tollens test and yielded a semicarbazone identical with that prepared earlier from 2-mesityl-2-phenylvinyl alcohol.[4] The structure of the aldehyde was established by its reaction with phenylmagnesium bromide which converted it to 2-mesityl-1,2-diphenylethanol (XII). The carbinol yielded an acetate which proved to be identical with the known compound.[5]



$$(\mathrm{Mes})(\mathrm{C_6H_5})\mathrm{CHCH(OH)C_6H_5}$$

XII

The aldehyde structure was confirmed by the infrared absorption spectrum which showed a maximum at 5.77μ.

Mesitylphenylacetaldehyde showed a pronounced tendency to undergo enolization. The enol form could be produced by treatment with alkali or by heating at 150° for two hours. The rearrangement under the influence of heat offers an explanation of the fact that vinyl alcohols rather than aldehydes were obtained by the thermal decomposition of peroxides of Type I.[4] Heating with acetic anhydride in the presence of sodium acetate converted the aldehyde to the acetate of the enol.[3] However, the aldehyde like the vinyl alcohol did not undergo spontaneous rearrangement, *i. e.*, the tautomeric shift appeared to be inhibited. In this respect the behavior of these compounds is similar to that of the stable trisubstituted vinyl alcohols and the corresponding ketones.[5] In this connection it is interesting to recall the behavior of benzhydryl mesityl diketone which was isolated in both the enol and keto forms by Kohler and Thompson,[9] who ascribed the sluggishness of the tautomeric shift to steric hindrance.

Experimental

Oxidation of 1-Mesityl-1-phenylpropanone with Selenium Dioxide.—A number of runs were made in which the reaction conditions were varied in an attempt to improve the yield. The procedure which gave the best result was as follows:

A mixture of 20 g. of 1-mesityl-1-phenylpropanone, 10 g. of selenium dioxide, 300 ml. of dioxane and 20 ml. of water was stirred and refluxed for six hours. The solution was decanted from the free selenium formed in the oxidation and the solvent was removed by distillation. The residue was distilled *in vacuo*, the fraction boiling at 165–175° (3 mm.) being collected. Addition of hexane caused the precipitation of a brown solid which was fractionally crystallized from hexane and separated by mechanical manipulation into two components. The glyoxal, a white crystalline compound which separated preferentially from hexane, was recrystallized from the same solvent; m. p. 134–135°, yield 2 g.

Anal. Calcd. for $C_{18}H_{18}O_2$: C, 81.18; H, 6.81. Found: C, 81.31; H, 7.06.

The infrared spectrum of this compound in hexane solution showed absorption maxima at 2.94 μ and 6.02 μ indicating the presence of a hydroxyl and a carbonyl (conjugated) group, respectively.

The other component of the mixture, a yellow compound, was recrystallized from ethanol; m. p. 136–137°. A mixed melting point determination with mesityl phenyl diketone showed no depression.

Distillation of the filtrate left after the separation of the brown solid gave a red oil boiling at 149–150° (1 mm.); n^{20}_D 1.5791 (*anal.* Found: C, 82.53; H, 7.50). It was shown by a color test with codeine sulfate solution to be a selenium compound. When treated with sodium ethoxide it was converted in low yield to mesitylphenylacetic acid.

In one run in which dry dioxane was used as solvent no mesitylphenylpyruvaldehyde was formed. Mesityl phenyl diketone and the selenium compound were isolated, however.

Reactions of Mesitylphenylpyruvaldehyde. (A) With Nitric Acid.—A mixture of 0.5 g. of mesitylphenylpyruvaldehyde, 5 ml. of concentrated nitric acid and 15 ml. of water was heated under reflux for forty minutes. The acid solution was decanted and the residue dissolved in hexane. The yellow compound which had separated at the end of two weeks melted at 135–137° and was shown by a mixed melting point determination to be mesityl phenyl diketone.

(B) With Hydrogen Peroxide.—A mixture of 0.2 g. of mesitylphenylpyruvaldehyde, 20 ml. of dioxane and 10 ml. of hydrogen peroxide (30%) was heated under reflux for four hours. It was diluted with water and extracted with ether. The ether solution in turn was extracted with sodium hydroxide solution (10%). Acidification of the alkaline extract with hydrochloric acid precipitated a substance which was recrystallized from high-boiling petroleum ether; m. p. 166–168°. A mixed melting point determination with mesitylphenylacetic acid showed no depression.

(C) With Semicarbazide.—A mixture of 0.2 g. of mesitylphenylpyruvaldehyde, 1 g. of semicarbazide hydrochloride, 1.5 g. of sodium acetate, 10 ml. of ethanol and 1 ml. of water was boiled for ten minutes and allowed to stand in the cold. The semicarbazone was recrystallized from acetone; m. p. 140–145°.

Anal. Calcd. for $C_{19}H_{21}O_2N_3$: C, 70.55; H, 6.56; N, 13.00. Found: C, 70.21; H, 6.86; N, 13.25.

(D) With Hydroxylamine.—A mixture of 0.2 g. of mesitylphenylpyruvaldehyde, 0.5 g. of hydroxylamine hydrochloride, 3 ml. of water, 2 ml. of sodium hydroxide solution (10%) and 20 ml. of ethanol was heated on a steam-bath for twenty minutes. The resulting mixture was filtered to remove the sodium chloride, diluted with water and allowed to stand in the cold. The oxime which separated was recrystallized from hexane; m. p. 142–143°.

Anal. Calcd. for $C_{18}H_{19}O_2N$: C, 76.89; H, 6.82; N, 4.98. Found: C, 77.21; H, 6.96; N, 4.97.

(E) With Alkali.—A suspension of 0.2 g. of mesitylphenylpyruvaldehyde in 10 ml. of sodium hydroxide solution (10%) was heated under reflux for thirty minutes, diluted with water and extracted with ether. The aqueous layer was acidified with hydrochloric acid and the acidified solution again extracted with ether. Evaporation of the ether left an oil which could not be induced to crystallize.

(F) With Acetic Anhydride.—An acetate was formed by allowing a mixture of 1 g. of mesitylphenylpyruvaldehyde, 4 ml. of acetic anhydride and 6 ml. of pyridine to stand at room temperature overnight. It crystallized from low-boiling petroleum ether in colorless cubes; m. p. 100–101°, yield 0.9 g.

Anal. $C_{20}H_{20}O_3$: C, 77.78; H, 6.54. Found: C, 77.79; H, 6.78.

When the acetate was heated with ethanolic sodium hydroxide for twenty minutes, it was hydrolyzed to

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(9) Kohler and Thompson, This Journal, **59**, 887 (1937).

mesitylphenylpyruvaldehyde. Treatment of the acetate with semicarbazide gave the semicarbazone of the original pyruvaldehyde.

3-Mesityl-3-phenyl-1,2-propanediol.—A mixture of 1 g. of mesitylphenylpyruvaldehyde, 0.02 g. of platinum oxide and 50 ml. of ethanol was treated with hydrogen at atmospheric pressure until two molecular equivalents of hydrogen was absorbed. The catalyst was removed by filtration and the solvent evaporated. The propanediol was crystallized from benzene; yield 0.89 g., m. p. 147–148°.

Anal. Calcd. for $C_{18}H_{22}O_2$: C, 79.99; H, 8.21. Found: C, 79.97; H, 8.41.

The **diacetate** was prepared by allowing a mixture of 0.5 g. of the glycol, 4 ml. of acetic anhydride and 6 ml. of pyridine to stand overnight. It was recrystallized from hexane; m. p. 80–81°.

Anal. Calcd. for $C_{22}H_{26}O_4$: C, 74.55; H, 7.40. Found: C, 74.33; H, 7.45.

Mesitylphenylacetaldehyde.—To a solution of 1 g. of 3-mesityl-3-phenyl-1,2-propanediol in 50 ml. of ethanol was added a solution of 1 g. of potassium periodate in 50 ml. of sulfuric acid (1 *N*). The mixture was stirred for one-half hour at 18° and an additional hour at 33°. Water was then added to dilute the mixture and the precipitate which separated was collected on a filter and dried in the air. It weighed 0.89 g. and melted at 66–72°. Several crystallizations of the aldehyde from low-boiling petroleum ether raised its melting point to 73–74°.

Anal. Calcd. for $C_{17}H_{18}O$: C, 85.67; H, 7.62. Found: C, 85.92; H, 7.85.

The aldehyde gave a positive test with Tollens reagent. Its infrared spectrum showed a peak at 5.77 (presence of an aldehyde group) and no absorption in the hydroxyl and olefinic regions. It was stable under ordinary conditions. However, after standing one year in air it was found to have undergone extensive decomposition.

Reactions of Mesitylphenylacetaldehyde. (A) With Semicarbazide.—A mixture of 0.21 g. of mesitylphenylacetaldehyde, 0.15 g. of semicarbazide hydrochloride, 0.25 g. of sodium acetate, 8 ml. of ethanol (aldehyde-free) and 8 ml. of water was heated to boiling. Dilution of the solution with water caused the slow precipitation of the semicarbazone. It melted at 203–205° and did not depress the melting point of the known semicarbazone prepared from 2-mesityl-2-phenylvinyl alcohol.[4]

(B) **With Acetic Anhydride.**—A mixture of 0.1 g. of mesitylphenylacetaldehyde, 0.15 g. of fused sodium acetate and 1.5 ml. of acetic anhydride was heated on a steam-bath for two hours and poured with vigorous stirring into 20 ml. of ice water. The precipitate was collected on a filter, washed with water and recrystallized from dilute ethanol; m. p. 89–90°. A mixed melting point determination with the acetate prepared from 2-mesityl-2-phenylvinyl alcohol[3] showed no depression.

(C) **With Phenylmagnesium Bromide.**—A solution of 0.5 g. of the aldehyde in 12 ml. of absolute ether was added in fifteen minutes to a Grignard reagent prepared from 0.063 g. of magnesium turnings, 0.505 g. of bromobenzene and 10 ml. of absolute ether. Stirring was continued for two hours during which time nitrogen was passed over the reaction mixture and the solution was decomposed with cold hydrochloric acid. The ether layer was removed, washed successively with water, sodium bicarbonate solution and water and dried over magnesium sulfate. Removal of the ether left the 2-mesityl-1,2-diphenylethanol as a white solid which was recrystallized from high-boiling petroleum ether; m. p. 133–134°.

Anal. Calcd. for $C_{23}H_{24}O$: C, 87.29; H, 7.66. Found: C, 87.02; H, 7.65.

When the 2-mesityl-1,2-diphenylethanol was treated with acetic anhydride and pyridine an acetate (m. p. 125–126°) was formed. It did not depress the melting point of an authentic sample of 2-mesityl-1,2-diphenylethyl acetate prepared previously by the reduction of α-mesityldesoxybenzoin with isopropylmagnesium bromide and treatment of the product with acetyl chloride.[5]

(D) **Enolization.**—1. A sample of 0.1 g. of mesitylphenylacetaldehyde was dissolved in a solution of 0.35 g. of potassium hydroxide in 5 ml. of ethanol. The solution was allowed to stand at room temperature overnight, diluted with water and neutralized with hydrochloric acid. The precipitate was collected on a filter and recrystallized from low-boiling petroleum ether; m. p. 112–113°. It was shown by a mixed melting point determination to be 2-mesityl-2-phenylvinyl alcohol.

2. Enolization of the aldehyde was also effected by heating at 150° for two hours. The vinyl alcohol was recrystallized from low-boiling petroleum ether; m. p. 112–113°.

α-Hydroxy-β-mesityl-β-phenylpropiomesitylene.—A mixture of 4 g. of 1,3-dimesityl-3-phenyl-1-propen-1-ol peroxide (m. p. 132–134°),[4] 6.7 g. of potassium iodide and 40 ml. of glacial acetic acid was shaken at room temperature for six hours. It was diluted with 60 ml. of water and the precipitate was collected on a filter. The pure α-hydroxy-β-mesityl-β-phenylpropiomesitylene obtained by recrystallization from ethanol melted at 160–162° and weighed 2 g.

Anal. Calcd. for $C_{27}H_{30}O_2$: C, 83.88; H, 7.83. Found: C, 83.84; H, 7.82.

The **acetate** prepared by treatment with acetic anhydride and pyridine was recrystallized from ethanol; m. p. 195–196°.

Anal. Calcd. for $C_{29}H_{32}O_3$: C, 81.25; H, 7.54. Found: C, 81.14; H, 7.56.

An attempt was made to cleave α-hydroxy-β-mesityl-β-phenylpropiomesitylene with periodic acid. To a solution of 1 g. of the hydroxy ketone in 100 ml. of ethanol was added a solution of 0.6 g. of potassium periodate in 30 ml. of sulfuric acid (1 *N*). The mixture was shaken at room temperature for six hours and then diluted with water. The starting material was recovered.

1,3-Dimesityl-3-phenyl-1,2-propanediol.—A solution of 4 g. of α-hydroxy-β-mesityl-β-phenylpropiomesitylene in 20 ml. of ethanol was heated to 150° and subjected to a pressure of 2200 lb. of hydrogen for two hours in the presence of a copper chromite catalyst. After removal of the catalyst the solution was concentrated to a small volume. The white propanediol which separated when the solution was allowed to stand in the cold was recrystallized from ethanol; m. p. 134–135°, yield 2.9 g.

Anal. Calcd. for $C_{27}H_{32}O_2$: C, 83.45; H, 8.32. Found: C, 83.35; H, 8.58.

The **diacetate** prepared by the use of acetic anhydride and pyridine was recrystallized from ethanol; m. p. 187–188°.

Anal. Calcd. for $C_{31}H_{36}O_4$: C, 78.76; H, 7.69. Found: C, 78.36; H, 7.63.

Oxidative Cleavage of 1,3-Dimesityl-3-phenyl-1,2-propanediol with Periodic Acid.—To a solution of 10 g. of 1,3-dimesityl-3-phenyl-1,2-propanediol in 1000 ml. of ethanol was added a solution of 6 g. of potassium periodate in 300 ml. of sulfuric acid (1 *N*). The mixture was stirred at room temperature for seven hours and diluted with 2 l. of water. The brown oily substance which separated from the water solution was collected and dissolved in 10 ml. of ethanol. The white needles which separated when the solution was allowed to stand in the cold were recrystallized from low-boiling petroleum ether; m. p. 73–74°. A mixed melting point determination with the sample of mesitylphenylacetaldehyde prepared by cleavage of 3-mesityl-3-phenyl-1,2-propanediol with periodic acid showed no depression.

The filtrate remaining after removal of mesitylphenylacetaldehyde was evaporated under diminished pressure and the residue distilled *in vacuo*. The fractions boiling at 89–90° (5 mm.) and 186–187° (5 mm.) were collected separately.

The lower-boiling fraction was identified as mesitaldehyde. Its dinitro derivative was formed by nitration with equal volumes of nitric acid and concentrated sulfuric acid; m. p. 166–167°. The dinitromesitaldehyde formed a phenylhydrazone which melted at 208–209°.[10]

The higher-boiling fraction was dissolved in 2 ml. of low-boiling petroleum ether. The white compound which had separated at the end of two days was recrystallized from low-boiling petroleum ether; m. p. 103–105°. A mixed melting point determination with 2,4,6-trimethylbenzoin showed no depression.

1,3-Dimesityl-3-*p*-tolyl-1-propen-1-ol Peroxide.—The procedure for the preparation of this compound was based on the directions used in the preparation of 1,3-dimesityl-3-phenyl-1-propen-1-ol peroxide.[4] To a Grignard reagent prepared from 14.8 g. of magnesium turnings, 105 g. of *p*-bromotoluene and 400 ml. of absolute ether was added a solution of 116 g. of mesitylacetomesitylene in 300 ml. of dry benzene. The mixture was stirred and heated for two hours and decomposed with cold dilute hydrochloric acid. The organic layer was washed with ice water and placed in a 1-liter Erlenmeyer flask held in an ice-bath. Oxygen was bubbled rapidly through the solution for six hours. The solvent was removed by a stream of air leaving 137 g. of white powder melting at 130–131°. The enol peroxide was recrystallized from a mixture of ether, benzene and low-boiling petroleum ether; m. p. 130.5–131.5°.

Anal. Calcd. for $C_{28}H_{32}O_3$: C, 80.73; H, 7.76. Found: C, 80.92; H, 7.92.

α-Hydroxy-β-mesityl-β-*p*-tolylpropiomesitylene.—A mixture of 8 g. of 1,3-dimesityl-3-*p*-tolyl-1-propen-1-ol peroxide, 14 g. of potassium iodide and 100 ml. of glacial acetic acid was shaken at room temperature for eight hours. It was diluted with 150 ml. of water and the precipitate was collected on a filter. The yield of the crude α-hydroxy-β-mesityl-β-*p*-tolylpropiomesitylene was 7 g.; m. p. 134–136°. After recrystallization from ethanol the compound melted at 136–138°.

Anal. Calcd. for $C_{28}H_{32}O_2$: C, 83.96; H, 8.07. Found: C, 83.96; H, 8.13.

The **acetate**, prepared by treatment with acetic anhydride and pyridine, was recrystallized from ethanol; m. p. 170–171°.

Anal. Calcd. for $C_{30}H_{34}O_3$: C, 81.41; H, 7.76. Found: C, 81.48; H, 7.84.

1,3-Dimesityl-3-*p*-tolyl-1,2-propanediol.—A solution of 4 g. of α-hydroxy-β-mesityl-β-*p*-tolylpropiomesitylene in 20 ml. of ethanol was subjected for three hours to a hydrogen pressure of 2000 pounds and a temperature of 150° in the presence of a copper chromite catalyst. The catalyst was removed by filtration and the solvent evaporated. The 1,3-dimesityl-3-*p*-tolyl-1,2-propanediol crystallized from ethanol in white plates which melted at 147–148°.

Anal. Calcd. for $C_{26}H_{34}O_2$: C, 83.53; H, 8.52. Found C, 83.28; H, 8.73.

The **diacetate** of the glycol, prepared by the use of acetic anhydride and pyridine, was recrystallized from dilute ethanol; m. p. 163–165°.

Anal. Calcd. for $C_{32}H_{38}O_4$: C, 78.98; H, 7.87. Found: C, 79.17; H, 8.01.

Oxidative Cleavage of 1,3-Dimesityl-3-*p*-tolyl-1,2-propanediol with Periodic Acid.—To a solution of 5 g. of 1,3-dimesityl-3-*p*-tolyl-1,2-propanediol in 250 ml. of pure dioxane was added a solution of 3 g. of potassium periodate in 150 ml. of sulfuric acid (1 *N*). The mixture was stirred at room temperature for eight hours, diluted with 600 ml. of water and extracted twice with ether. Evaporation of the ether extract left an oil which could not be induced to crystallize.

When the oil was distilled *in vacuo* fractions boiling at 85–86° (3 mm.) and 163–165° (3 mm.) were collected. The residue that remained in the distilling flask was dissolved in ethanol and the solution allowed to stand in the cold; white crystals separated slowly. When recrystallized from ethanol they formed fine needles; m. p. 116–117°.

Anal. Calcd. for $C_{28}H_{32}O$: C, 87.45; H, 8.39. Found: C, 87.94; H, 8.59.

This compound had the composition of the glycol minus the elements of water and may be a ketone.

The lower-boiling portion of the distillate was identified as mesitaldehyde by nitration, which converted it to the known dinitromesitaldehyde; m. p. 166–167°.[10]

The higher-boiling portion was dissolved in 5 ml. of low-boiling petroleum ether and allowed to stand in the cold. White crystals which separated after several days were recrystallized from high-boiling petroleum ether; m. p. 211–212°. A mixed melting point determination with mesityl-*p*-tolylacetic acid showed no depression.

Summary

Mesitylphenylacetaldehyde has been prepared by the oxidative cleavage of 3-mesityl-3-phenyl-1,2-propanediol and of 1,3-dimesityl-3-phenyl-1,2-propanediol. It rearranged to 2-mesityl-2-phenylvinyl alcohol when heated at 150° or when treated with alkali.

Evidence is presented indicating that mesitylphenylpyruvaldehyde exists in the enol form.

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[10] Hinkel, Ayling and Morgan, *J. Chem. Soc.*, 1170 (1931).