which was precipitated at the same time was extracted from the filtrate with ether. The ethereal extract evaporated to leave a residual oil and several large crystals. The crystals dissolved when the mixture was stirred with 10% potassium carbonate solution, but the oil did not, and was removed from the suspension by ether extraction. Acidification of the bicarbonate solution precipitated an additional 0.3 g. of acid, and evaporation of the ether extract left 0.8 g. of an oil which solidified completely after standing for two days. This material melted at 55–57°, and crystallized in the form of needles or plates from mixtures of ether and low-boiling petroleum ether. After two crystallizations from these solvents it melted at 61–62°.

Anal. Calcd. for $C_{12}H_{12}O_2$: C, 76.57; H, 6.43. Found: C, 76.46; H, 6.66.

The compound dissolved slowly in cold aqueous potassium hydroxide solution and could be recovered by acidification of this solution. It dissolved more rapidly in saturated alcoholic potassium hydroxide solution and was not precipitated by dilution of the solution with water. When the diluted solution was acidified with hydrochloric acid a solid product, m. p. 155–157°, was precipitated. This compound was found by the mixed melting point method to be identical with acid B. It seems probable that the compound melting at 61–62° is the lactone of δ-hydroxy-β-methyl-δ-phenyl-α-pentenoic acid.

β-Styrylcrotonic Acid (Form A) from Benzalacetone and Ethyl Bromoacetate.—The procedure employed was essentially that of Kohler and Heritage.[8] These investigators, however, distilled the hydroxy ester which they obtained and subsequently dehydrated it by heating with hydrochloric acid, whereas in the present instance it was more convenient to dehydrate the crude ester directly by distillation in the presence of a few crystals of iodine. To the product obtained from the Reformatsky condensation of 61 g. of benzalacetone with 70 g. of ethyl bromoacetate, 0.1 g. of iodine was added and distillation was carried out under reduced pressure. After the water had been removed, three fractions were collected; a 14.5-g. fraction at 141–145° (2 mm.), a 20-g. fraction at 145–147° (2 mm.) and a 3-g. fraction at 147–149° (2 mm.).

To 1 g. of the middle fraction was added 25 cc. of saturated alcoholic potassium hydroxide solution, and the mixture, in which a precipitate quickly formed, was allowed to stand for two days. The precipitate dissolved when the solution was diluted with water, and a crude acid, m. p. 120–140°, was precipitated when the solution was acidified with hydrochloric acid. White needles melting at 158–159° were obtained after three crystallizations from benzene. No depression of the melting point was observed when this product was mixed with acid A from condensation of benzaldehyde with methyl γ-bromosenecioate.

The melting point reported by Kohler and Heritage[8] was 153°. von Auwers and Müller,[9] using the same method, obtained a product melting at 156–157°. Kuhn and Hoffer reported a melting point of 160°. It seems probable that all the samples made by this method are identical, and different from form B.

Summary

A method has been developed for the preparation of methyl γ-bromosenecioate from methallyl chloride.

Methyl γ-bromosenecioate has been condensed with benzaldehyde to yield two forms of β-styrylcrotonic acid and a third compound thought to be the lactone corresponding to the expected hydroxy acid.

URBANA, ILLINOIS RECEIVED JANUARY 20, 1944

---

[8] Kohler and Heritage. *Am. Chem. J.*, **43**, 475 (1909).

[9] von Auwers and Müller, *J. prakt. Chem.*, [2] **105**, 379 (1932).

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

1,2-Addition of Methylmagnesium Iodide to Mesityl Ketones

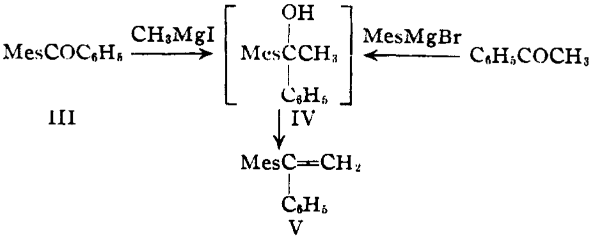
BY REYNOLD C. FUSON, M. D. ARMSTRONG, WM. E. WALLACE[1] AND J. WAYNE KNEISLEY[2]

The carbonyl group of simple mesityl ketones has been known to react additively only with hydrogen. In particular, normal or 1,2 addition of a Grignard reagent has never been observed.[3] Recently mesityl *t*-butyl diketone (I) was found to undergo 1,2 addition of methylmagnesium iodide to each of the carbonyl groups.[4] Also the conversion of certain mesitoic esters to ketones by the action of Grignard reagents[5] may be explained by assuming 1,2 addition of the reagent to the ester carbonyl group.

These results led to a reëxamination of the behavior of simple mesityl ketones toward the Grignard reagent. The most favorable structure appeared to be that in which the mesitoyl radical was joined to the smallest possible group that possessed no *alpha* hydrogen atom. Among aliphatic radicals *t*-butyl was the logical choice. However, pivalylmesitylene (II) could not be induced to react with methylmagnesium iodide.[4] In the aromatic series benzoylmesitylene (III) appeared to offer the best chance of realizing 1,2 addition.

$$\underset{\text{I}}{\mathrm{MesCOCOC(CH_3)_3}} \qquad \underset{\text{II}}{\mathrm{MesCOC(CH_3)_3}}$$

When benzoylmesitylene was treated with methylmagnesium iodide it was converted in 64% yield to 1-mesityl-1-phenylethylene (V), the dehydration product of the carbinol (IV) that would have resulted from 1,2 addition. The direct production of the olefin rather than the carbinol is similar to results obtained by others.[6]



---

[1] Present address: General Aniline and Film Corporation, Easton, Pennsylvania.

[2] Present address: Hercules Powder Company, Wilmington, Delaware.

[3] Kohler and Baltzly, THIS JOURNAL, **54**, 4015 (1932).

[4] Fuson and Robertson, *J. Org. Chem.*, **7**, 466 (1942).

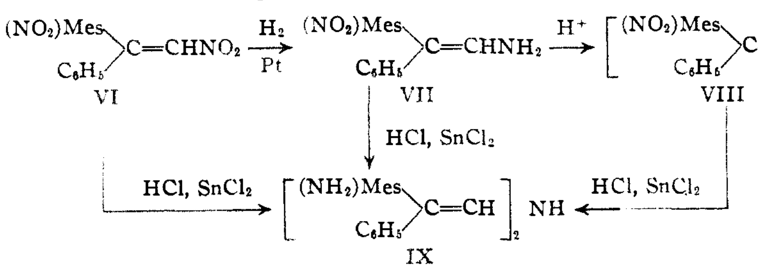
[5] Fuson, Bottorff and Speck, THIS JOURNAL, **64**, 1450 (1942).

[6] Tissier and Grignard, *Compt. rend.*, **132**, 1184 (1901).

The structure of the olefin was established by an independent synthesis. 1-Mesityl-1-phenylethylene was formed in 15% yield by the condensation of mesitylmagnesium bromide with acetophenone. The experimental conditions were similar to those which were employed with the benzoylmesitylene and the product was the ethylene rather than the carbinol.[7]

Since the olefin was a liquid, it was desirable to compare the two samples by reference to a solid derivative. Such a derivative was prepared by nitration according to the procedure used by Anschütz and Romig to nitrate 1,1-diphenylethylene.[8]

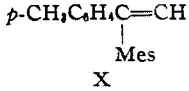
The nitro derivative, 2-(3-nitromesityl)-2-phenyl-1-nitroethylene (VI), was very much like known nitroölefins. The reduction products are shown in the following outline.



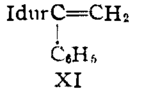
The nitroamine (VII) has been written as a vinylamine rather than as an imine because of the analogy with 2,2-dimesitylvinyl alcohol.[9] It was neutral and could not be hydrolyzed to the corresponding aldehyde and thus appeared to differ in type from the corresponding derivative of 1,1-diphenylethylene.[10]

It did resemble 2,2-diphenylvinylamine,[11] however, in the ease with which it lost ammonia in the presence of acids to yield the corresponding divinylamine (VIII). Reduction with stannous chloride in ethanolic hydrochloric acid converted VI, VII and VIII to the aminodivinylamine, IX. The divinylamines are similar to 2,2,2′,2′-tetraphenyldivinylamine, described by Lipp[12] and by Krabbe and Schmidt.[11]

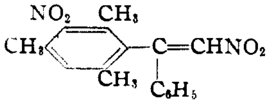
p-Toluylmesitylene and benzoylisodurene reacted with methylmagnesium iodide in a manner similar to that observed with benzoylmesitylene. The products were 1-mesityl-1-(*p*-tolyl) ethylene (X) and 1-isoduryl-1-phenylethylene (XI), respectively. The latter also was prepared by condensing acetophenone with isodurylmagnesium bromide. The 1-mesityl-1-*p*-tolylethylene (X), obtained only in impure form, yielded a dinitro derivative (XII) analogous to VI. 1-Isoduryl-1-phenylethylene, by a slightly different procedure, gave a trinitro derivative (XIII).


$$p\text{-}CH_3C_6H_4\underset{\text{Mes}}{\underset{|}{C}}=CH_2$$

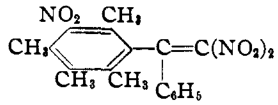
X


$$\text{Idur}\underset{C_6H_5}{\underset{|}{C}}=CH_2$$

XI



XII



XIII

The new olefins immediately decolorized a solution of permanganate and were attacked by chromic acid. They decolorized a solution of bromine in carbon tetrachloride with the evolution of a small amount of hydrogen bromide.

Experimental

Benzoylisodurene.—This ketone was made by condensing 134 g. of isodurene with 140 g. of benzoyl chloride by the Friedel–Crafts method. The ketone was purified by fractionation *in vacuo;* b. p. 159–164° (4 mm.); m. p. 60–61°; yield 78%.

Anal.[13] Calcd. for $C_{17}H_{18}O$: C, 85.67; H, 7.61. Found: C, 85.98; H, 7.57.

Benzoylmesitylene and Methylmagnesium Iodide.—A solution of 30 g. of benzoylmesitylene, prepared according to the directions of Montagne,[14] in 50 cc. of dry ether was added slowly to a solution of methylmagnesium iodide, prepared from 30 g. of methyl iodide, 5.4 g. of magnesium and 75 cc. of dry ether. After the addition was completed, a little benzene was added and ether was allowed to evaporate until the temperature of the vapors was 60°. The reaction mixture was then stirred under reflux for sixteen hours. The contents of the flask were decomposed and the organic layer was separated. After the solution had been washed with water and dried, the solvent was removed by evaporation. The resulting oil was distilled *in vacuo*. The fraction which came over at 129–134° (3 mm.) was collected; yield 19 g. (64%); $n^{20}\text{D}$ 1.5812. Redistillation yielded the pure olefin; b. p. 120° (3 mm.); $n^{20}\text{D}$ 1.5835; d_{20}^{20} 1.0003.

Anal. Calcd. for $C_{17}H_{18}$: C, 91.84; H, 8.16; *MR*, 74.2. Found: C, 91.57; H, 8.19; *MR*, 74.4.

1-Mesityl-1-phenylethylene was obtained also by the condensation of acetophenone with mesitylmagnesium bromide. To a mesitylmagnesium bromide solution, prepared from 41 g. of bromomesitylene and 6.5 g. of magnesium in 100 cc. of dry ether, a solution of 20 g. of acetophenone in 25 cc. of dry ether was added at such a rate that the ether refluxed vigorously. When the addition was completed the reaction mixture was refluxed for one hour, cooled in an ice-bath and decomposed with cold, dilute hydrochloric acid. The ether layer was treated in the usual manner and the viscous, yellow oil which was isolated was distilled *in vacuo*. The forefraction contained 12 g. of mesitylene and 12 g. of acetophenone. The main fraction was redistilled *in vacuo* until the index of refraction remained constant; this required four distillations; yield 3 g.; b. p. 117–120° (4 mm.); $n^{20}\text{D}$ 1.5831; d_{20}^{20} 1.0004. The olefin was a light yellow oil which decolorized neutral permanganate immediately and reacted with bromine with the evolution of hydrogen bromide.

Reactions of 1-Mesityl-1-phenylethylene.—A solution of the olefin rapidly decolorized a solution of potassium

---

(7) Kohler and Blanchard, THIS JOURNAL, **57**, 367 (1935).

(8) Anschütz and Romig, *Ber.*, **18**, 662 (1885); Anschütz and Hilbert, *ibid.*, **54**, 1854 (1921).

(9) Fuson and Rowland, THIS JOURNAL, **65**, 992 (1943).

(10) Kohler and Drake, *ibid.*, **45**, 1281 (1923).

(11) Krabbe and Schmidt, *Ber.*, **72B**, 381 (1939).

(12) Lipp, *Ann.*, **449**, 15 (1926).

(13) The microanalyses reported in this paper were carried out by Miss Margaret McCarthy, Miss Theta Spoor and Miss Dorothy Schneider.

(14) Montagne, *Rec. trav. chim.*, **27**, 327 (1908).

permanganate in the cold. It absorbed bromine from a bromine solution in carbon tetrachloride; the evolution of hydrogen bromide was detected.

A small amount of platinum oxide was added to a solution of 0.73 g. of the olefin in 40 cc. of 95% ethanol, and the solution was shaken for several hours with hydrogen at one atmosphere. One mole of hydrogen was absorbed.

The 1-mesityl-1-phenylethane boiled at 154–155° (4 mm.); $n^{23}\text{D}$ 1.5621.[15]

Anal. Calcd. for $C_{17}H_{20}$: C, 91.01; H, 8.99. Found: C, 90.64; H, 9.11.

The isomeric hydrocarbon 1-mesityl-2-phenylethane is a solid melting at 39–39°.[16]

2-(3-Nitromesityl)-2-phenyl-1-nitroethylene (VI).—A mixture of 6.5 g. of fuming nitric acid, 4 cc. of glacial acetic acid and 4 cc. of acetic anhydride was added dropwise, with stirring, over a period of one hour to an ice-cold solution of 8 g. of 1-mesityl-1-phenylethylene in 5.5 cc. of acetic anhydride. Yellow crystals appeared during the addition of the nitrating mixture. The crude dinitro derivative was collected on a filter; yield 7 g. It crystallized from ethanol in yellow plates melting at 144–145°.

Anal. Calcd. for $C_{17}H_{16}N_2O_4$: C, 65.37; H, 5.16; N, 8.97; mol. wt., 312. Found: C, 65.23; H, 5.36; N, 8.89; mol. wt. (cryoscopic in benzophenone), 306.

2-(3-Nitromesityl)-2-phenylvinylamine (VII).—A mixture of 5 g. of 2-(3-nitromesityl)-2-phenyl-1-nitroethylene (VI), 100 cc. of ethyl acetate and 0.1 g. of platinum oxide catalyst was shaken in an atmosphere of hydrogen. Hydrogen was absorbed rapidly. After thirty minutes the mixture was filtered to remove the catalyst and the solvent was evaporated by a stream of air. The orange residue was recrystallized from ethanol; m. p. 100–101°; yield 4 g.

•*Anal.* Calcd. for $C_{17}H_{18}N_2O_2$: C, 72.32; H, 6.43; N, 9.92; mol. wt., 280. Found: C,. 72.42; H, 6.30; N, 10.03; mol. wt. (cryoscopic in benzophenone), 290.

The **acetyl derivative** was formed by heating a mixture of the amine (VII) (1 g.) with acetic anhydride (5 cc.) under reflux for fifteen minutes. It separated from ethanol in pale yellow crystals; m. p. 199–200°.

Anal. Calcd. for $C_{19}H_{20}N_2O_3$: C, 70.35; H, 6.23; N, 8.64. Found: C, 70.45; H, 6.24; N, 8.67.

The benzoyl **derivative** was produced by shaking 0.5 g. of the amine (VII) with 1 cc. of benzoyl chloride and 5 cc. of 10% sodium hydroxide for ten minutes. The mixture was poured into water and the product was purified by recrystallization from ethanol. It formed pale yellow crystals melting at 143–144°.

Anal. Calcd. for $C_{24}H_{22}N_2O_3$: C, 74.59; H, 5.74; N, 7.25. Found: C, 74.60; H, 5.96; N, 7.19.

2,2′-Di-(3-aminomesityl)**-2,2′-diphenylvinylamine** (**IX**).—A mixture of 5 g. of 2-phenyl-2-(3-nitromesityl)-1-nitroethylene (VI), 250 cc. of absolute ethanol, 25 g. of hydrated stannous chloride and 25 cc. of concentrated hydrochloric acid was heated gradually at first and finally under reflux, for six hours. The product was the divinylamine, IX. It was recrystallized from 95% ethanol. It rapidly turned blue when allowed to stand in the air. In the pure state it had a light cream color; m. p. 184–186°.

Anal. Calcd. for $C_{34}H_{37}N_3$: C, 83.73; H, 7.65; N, 8.62; mol. wt., 488. Found: C, 83.86; H, 7.65, 7.50; N, 8.78; mol. wt. (cryoscopic in benzophenone), 489.

The divinylamine (IX) was obtained also by the reduction of the nitro amine (VII). A mixture of 2 g. of the amine, 100 cc. of absolute ethanol, 8 g. of hydrated stannous chloride and 10 cc. of concentrated hydrochloric acid was heated for eight hours under reflux. The amine was isolated in the usual manner; m. p. 184–186°.

The divinylamine was produced also by reduction of 2,2′-di-(3-nitromesityl)-2,2′-diphenyldivinylamine (VIII). The procedure was similar to that just described for reducing the mononitro amine (VII); m. p. 184–186°

2,2′-Di-(3-nitromesityl)-2,2′-diphenyldivinylamine (**XIII**).—When 5 g. of 2-(3-nitromesityl)-2-phenylvinylamine (VII) was dissolved in 95% ethanol and 10 cc. of 6 *N* hydrochloric acid was added, the divinylamine (VIII) separated from the solution as a yellow solid. It was recrystallized from glacial acetic acid; m. p. 235–236°.

Anal. Calcd. for $C_{34}H_{33}N_3O_4$: C, 74.56; H, 6.07; N, 7.67; mol. wt., 548. Found: C, 74.75; H, 6.16; N, 7.77; mol. wt. (cryoscopic in benzophenone), 582.

Treatment of this amine with a hot mixture of concentrated hydrochloric acid and glacial acetic acid produced no change.

1-Isoduryl-1-phenylethylene.—The condensation of benzoylisodurene with methylmagnesium iodide, carried out in the manner described for that of the mesityl analog, produced 1-isoduryl-1-phenylethylene as a yellow oil boiling at 152–154° (3 mm.); $n^{20}\text{D}$ 1.5872; d_4^{20} 1.000; yield 42%.

Anal. Calcd. for $C_{18}H_{20}$: C, 91.47; H, 8.53; *MR*, 77.7. Found: C, 91.31; H, 8.35; *MR*, 79.2.

The same compound was obtained, in 10% yield, by the condensation of acetophenone with isodurylmagnesium bromide; b. p. 156–157° (4 mm.); $n^{20}\text{D}$ 1.5864. This olefin decolorized an acetone solution of potassium permanganate. It was readily attacked by chromic acid in acetic acid.

A by-product of the reaction between methylmagnesium iodide and benzoylisodurene was a red-orange solid. After repeated recrystallization from absolute ethanol, it melted at 191–192.5°. The yield in grams was about the same as that of the olefin.

Anal. Calcd. for $C_{34}H_{35}O_2$: C, 85.31; H, 8.00; mol. wt., 478. Found: C, 85.80; H, 8.17; mol. wt. (ebullioscopic in chloroform), 457.

This compound did not reduce neutral potassium permanganate solution; it reacted slowly with bromine in carbon tetrachloride, with the evolution of hydrogen bromide. It was treated with acetyl chloride, but no derivative could be isolated.

Three grams of the olefin was treated with hydrogen at 50° and 2000 lb. pressure in the presence of Raney nickel. The product, 1-isoduryl-1-phenylethane, boiled at 160° (5 mm.); $n^{20}\text{D}$ 1.5695; d_4^{20} 0.990; m. p. 54.5–55°.

Anal. Calcd. for $C_{18}H_{22}$: C, 90.69; H, 9.31; *MR*. 78.4. Found: C, 90.78; H, 9.54; *MR*, 78.9.

2-(3-Nitroisoduryl)-2-phenyl-1,1-dinitroethylene (**X**).—1-Isoduryl-1-phenylethylene was nitrated in a manner similar to that described for mesitylphenylethylene except that the nitration mixture was allowed to stand for two days at room temperature. The trinitro derivative separated from ethanol in pale yellow crystals; m. p. 193–194°.

Anal. Calcd. for $C_{18}H_{17}N_3O_6$: C, 58.22; H, 4.62; N. 11.29. Found: C, 58.00; H, 4.84; N, 11.15.

1-Mesityl-1-(*p*-tolyl)-ethylene.—To prepare this olefin methylmagnesium iodide was condensed with *p*-toluylmesitylene by a procedure similar to that used to make the corresponding mesitylphenylethylene. The product was difficult to purify, being contaminated in all probability with the ketone. Nitration, however, produced a satisfactory sample of the dinitro derivative (IX).

2-(3-Nitromesityl)-2-*p*-tolyl-1-nitroethylene (**IX**).—This compound was prepared from 1-mesityl-1-*p*-tolylethylene by a procedure similar to that described for the phenyl derivative. The gummy residue solidified only after several weeks. It separated from ethanol in yellow crystals; m. p. 174–175°.

Anal. Calcd. for $C_{18}H_{18}N_2O_4$: C, 66.24; H, 5.56; N. 8.59. Found: C, 65.88; H, 5.47; N, 8.75.

Summary

It has been shown that methylmagnesium iodide condenses in the 1,2 manner with benzoylmesitylene, benzoylisodurene and *p*-toluylmesitylene.

---

[15] This compound was prepared by Dr. Norman Rabjohn.

[16] Fuson, Denton and Best, *J. Org. Chem.*, **8**, 64 (1943).

The product in each case is not the expected carbinol, but the olefin that would be formed from it by dehydration. The yields of 1-mesityl-1-phenylethylene and of 1-isoduryl-1-phenylethylene were 64 and 42%, respectively.

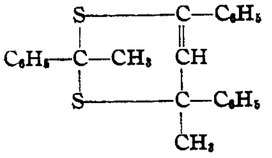
Urbana, Illinois Received February 9, 1944

[Contribution from the M. D. Anderson Hospital for Cancer Research, and the Department of Preventive Medicine and Public Health of the University of Texas Medical Branch]

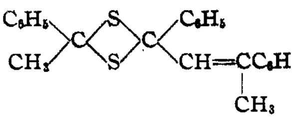
The Synthesis of 2,4-Diarylthiophenes[1]

By E. Campaigne

Baumann and Fromm,[2] while investigating thioacetophenone, reported the isolation of a compound, $C_{24}H_{22}S_2$, in good yield from the alcoholic mother liquors of the thioacetophenone preparation. Pyrolysis of this disulfide gave 2,4-diphenylthiophene but no yield was reported. On the basis of analysis and the formation of 2,4-diphenylthiophene, two possible structures were assigned to this compound, which they called "Anhydroacetophenone disulfide"



I

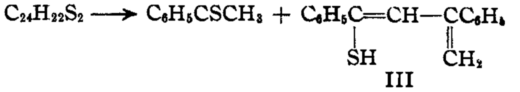


II

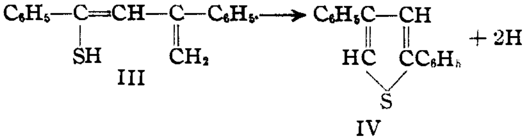
Structure I was assumed to be the most probable, since dithioacetophenone had never been isolated.

The synthesis of 2,4-diarylthiophenes has been reviewed by Bogert and Herrera.[3] These compounds are usually prepared by the reaction of phenylethylene or its derivatives with sulfur. This type of reaction gives a possibility of three different isomers, and consequently the yields have been extremely low. Bogert and Herrera[3] reported that by fusing acetophenone anil with sulfur, 2,4-diphenylthiophene was obtained in 28% yield. No 2,5-diphenylthiophene was found. These authors suggest that the formation of thioacetophenone as an intermediate accounts for the higher yield and lack of formation of 2,5-diphenylthiophene. However, Baumann and Fromm[2] reported the isolation of both 2,4- and 2,5-diphenylthiophene from the pyrolysis of thioacetophenone.

It seemed most probable that if either structure, (I) or (II), of "Anhydroacetophenone disulfide" given by Baumann and Fromm[2] were correct, highest yields of pure compounds could be obtained through this intermediate, since only 2,4-diarylthiophene, (IV), could be formed, according to the equations

(1) 

$$C_{24}H_{22}S_2 \longrightarrow C_6H_5CSCH_3 + \underset{\text{SH}}{C_6H_5C}{=}CH{-}\underset{\text{CH}_2}{\overset{}{CC_6H_5}}\quad \text{III}$$

(2) 

$$\underset{\text{SH}}{C_6H_5{-}C}{=}CH{-}\underset{\text{CH}_2}{C}{-}C_6H_5 \longrightarrow \text{IV} + 2H$$

III IV

The hypothetical compound III forms 2,4-diphenylthiophene by the loss of two atoms of hydrogen. In the pyrolysis of "Anhydroacetophenone disulfide" apparently acetophenone acts as the hydrogen acceptor, since in addition to the diarylthiophene, styrene and hydrogen sulfide were isolated.[2]

Several experiments were conducted to determine the yield of 2,4-diphenylthiophene from "Anhydroacetophenone disulfide." Pyrolysis of the material at 180° gave a tarry residue from which it was only possible to obtain sufficient pure 2,4-diphenylthiophene for identification. Refluxing the compound for several hours in boiling xylene caused a color change from colorless to deep purple. This would be expected, since the compound is unstable above its melting point, decomposing to give the highly colored thioketone monomers. No diarylthiophene could be isolated from this reaction, but the oily residue contained unsaturated carbon linkages. Apparently the reaction represented by the second equation does not take place readily. When this mixture was treated with copper chromite catalyst, IV was obtained in 83% yield. This reaction might be expected, since this catalyst has been used in the dehydrogenation of alcohols.[4]

A similar experiment with the "Anhydrodisulfide," V, prepared from *p*-methoxypropiophenone gave a 66% yield of 2,4-di-*p*-anisyl-3,5-dimethylthiophene, VI. This reaction was shown to proceed in two steps, as indicated by equations 1 and 2, as follows: the "Anhydrodisulfide" V, was refluxed in xylene, causing decomposition to an unsaturated oil. No thiophene derivative was formed by this treatment. However, when this residual oil was refluxed in alcohol with copper chromite, the dianisylthiophene, VI, was formed in good yield. "Anhydro-*p*-methoxypropiophenone disulfide," V, was not changed by re-

---

[1] Presented before the Organic Division at the 106th meeting of the American Chemical Society in Pittsburgh, Pa., September, 1943.

[2] Baumann and Fromm, *Ber.*, **28**, 895 (1895).

[3] Bogert and Herrera, This Journal, **45**, 238 (1923).

[4] Lazier, British Patent 301,806, *C. A.*, **23**, 4306 (1929).