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

SALTS OF NITRO COMPOUNDS. III. THE REACTION OF THE SILVER SALT OF PHENYLNITROACETONITRILE WITH DIPHENYLBROMOMETHANE

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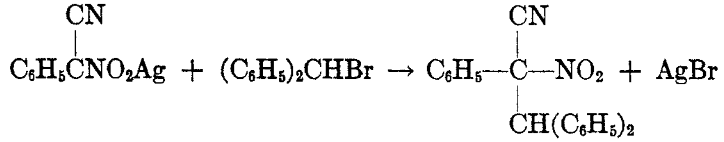
Received April 7, 1938

The alkylation of the silver salt of phenylnitroacetonitrile (I) with methyl iodide or benzyl chloride was found[1] to produce unstable nitronic esters, which showed that oxygen-alkylation had occurred. However, Wieland and Höchtlen[2] treated this same silver salt (I) with diphenylbromomethane and obtained α-nitro-α,β,β-triphenylpropionitrile (II), which involved carbon-alkylation. An examination of the literature on the alkylation of the silver salts of other nitro compounds showed that both carbon-[3] and oxygen-[4] alkylation have been reported, but that oxygen-alkylation seems to be more common. Since the course of the alkylation involving diphenylbromomethane seemed somewhat unusual, a careful study was made of this reaction in order to determine whether carbon-alkylation was the only reaction which occurred.



$$C_6H_5\overset{\overset{\displaystyle CN}{|}}{C}NO_2Ag + (C_6H_5)_2CHBr \rightarrow C_6H_5{-}\underset{\underset{\displaystyle CH(C_6H_5)_2}{|}}{\overset{\overset{\displaystyle CN}{|}}{C}}{-}NO_2 + AgBr$$

I II

Treatment of the silver salt of phenylnitroacetonitrile with diphenylbromomethane did produce the carbon-alkylated compound (II) in agreement with the observations of Wieland and Höchtlen[2] but the yield of this product amounted to only 10 to 18 per cent. The major part of the reac-

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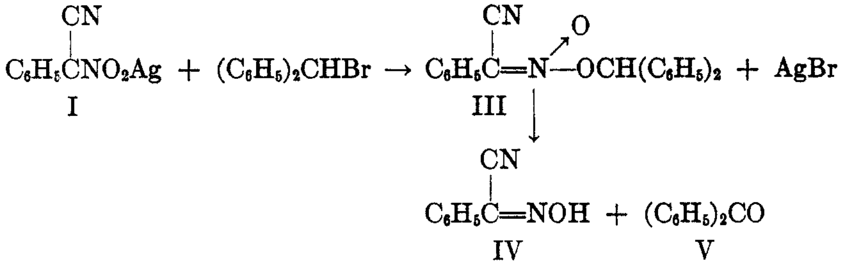
[1] THURSTON AND SHRINER, *J. Org. Chem.*, **2**, 183 (1937).

[2] WIELAND AND HÖCHTLEN, *Ann.* **505**, 237 (1933).

[3] NEF, *Ann.*, **280**, 263 (1894); DUDEN, *Ber.*, **26**, 3008 (1893); ULPIANI, *Atti. acad. Lincei*, [5], **12**, I, 439 (1903): *Chem. Zentr.*, **74**, II, 346 (1903); HANTZSCH, *Ber.*,**39**, 2472 (1906); STEINKOPF, *Ann.*, **434**, 21 (1923).

[4] RATZ, *Monatsh.*, **25**, 55 (1904); KOHLER AND STONE, *J. Am. Chem. Soc.*, **52**, 761 (1930); NENITZESCU AND ISACESCU, *Ber.*, **63**, 2484 (1930), *Bull. soc. chim. Rom.*, **14**, 53 (1932); THURSTON AND SHRINER, *J. Am. Chem. Soc.*, **57**, 2163 (1935); WISLICENUS AND ELVERT, *Ber.*, **41**, 4121 (1908).

tion mixture was composed of benzoylcyanide oxime (IV) and benzophenone (V) which would be the decomposition products expected if the nitronic ester (III) had been produced and then disproportionated.



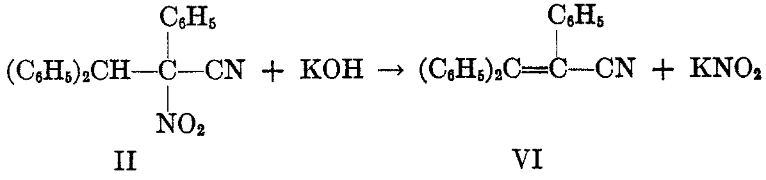
$$\underset{\text{I}}{C_6H_5\overset{CN}{\overset{|}{C}}NO_2Ag} + (C_6H_5)_2CHBr \rightarrow \underset{\text{III}}{C_6H_5\overset{CN}{\overset{|}{C}}{=}\overset{O}{\overset{\nearrow}{N}}{-}OCH(C_6H_5)_2} + AgBr$$

$$\downarrow$$

$$\underset{\text{IV}}{C_6H_5\overset{CN}{\overset{|}{C}}{=}NOH} + \underset{\text{V}}{(C_6H_5)_2CO}$$

This decomposition of a nitronic ester into an oxime and a ketone or aldehyde has been shown to be characteristic of such structures.[5] The quantities of these two products which were isolated amounted to about 50 per cent. of the theoretical yield, and indicated that oxygen-alkylation predominates over carbon-alkylation. The balance of the reaction mixture was composed of a mixture of the reactants and their decomposition products.

The second part of the present study was concerned with the proof of structure of the carbon-alkylation product, α-nitro-α,β,β-triphenylpropionitrile (II). No independent synthesis of this compound has ever been carried out. Its structure was based upon an analysis and the fact that alcoholic potassium hydroxide converted it into triphenylacrylonitrile (VI).[2]



$$\underset{\text{II}}{(C_6H_5)_2CH{-}\overset{C_6H_5}{\overset{|}{\underset{NO_2}{\underset{|}{C}}}}{-}CN} + KOH \rightarrow \underset{\text{VI}}{(C_6H_5)_2C{=}\overset{C_6H_5}{\overset{|}{C}}{-}CN} + KNO_2$$

In the course of the present work, triphenylacrylonitrile was obtained, not only by the action of alkali, but also by the action of phosphorus and hydriodic acid, on II. The triphenylacrylonitrile (VI) was characterized

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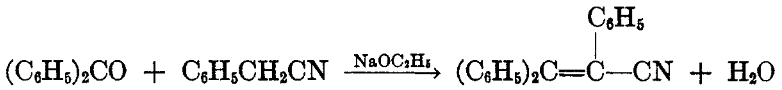
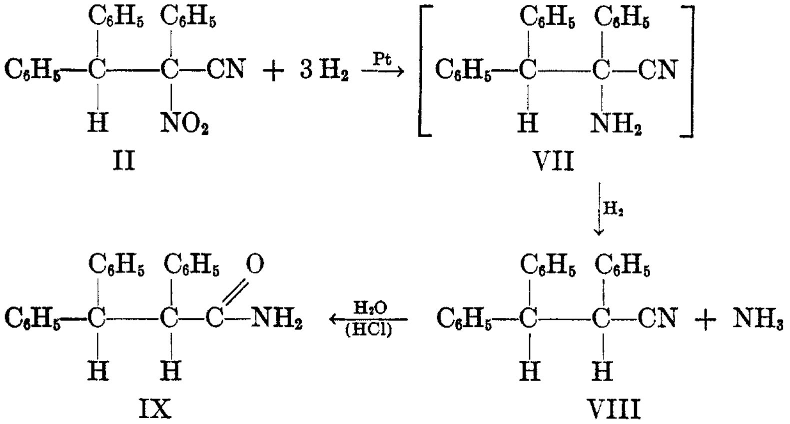
[5] Ratz, *Monatsh.*, **26**, 1487 (1905); Kohler and Stone, *J. Am. Chem. Soc.*, **52**, 761 (1930); Nenitzescu and Isacescu, *Ber.*, **63**, 2484 (1930); *Bull. soc. chim. Rom.*, **14**, 53 (1932); *ibid.*, **18**, 63 (1936); Arndt and Rose, *J. Chem. Soc.*, **1935**, 1; Thurston and Shriner, *J. Am. Chem. Soc.*, **57**, 2163 (1935); *J. Org. Chem.*, **2**, 183 (1937); Arndt, Loewe, and Isik, *Rev. fac. sci. Univ. Istanbol*, **2**, 1 (1937).

by comparison with an authentic sample synthesized by the following reaction according to the method of Bodroux.[6]



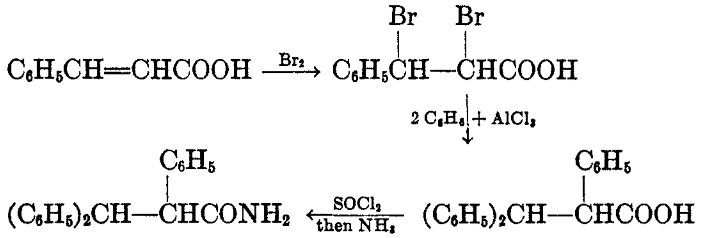
$$(C_6H_5)_2CO + C_6H_5CH_2CN \xrightarrow{NaOC_2H_5} (C_6H_5)_2C{=}\overset{C_6H_5}{\overset{|}{C}}{-}CN + H_2O$$

The most conclusive evidence supporting the structure assigned to the carbon-alkylation product (II) was obtained by characterizing the products obtained by catalytic reduction. Treatment of an alcohol solution of the compound II with platinum and hydrogen at room temperature produced ammonia and a 93 per cent. yield of α,β,β-triphenylpropionitrile (VIII).



II, VII, VIII, IX

The reduction of the nitro group in II would produce the substituted α-amino nitrile (VII) which readily undergoes hydrogenolysis[7] to the nitrile (VIII). The latter was characterized by heating it in a sealed tube with concentrated hydrochloric acid at 170°; under these conditions it was hydrolyzed to the amide (IX) which was identical with a sample synthesized by the following reactions:



IX

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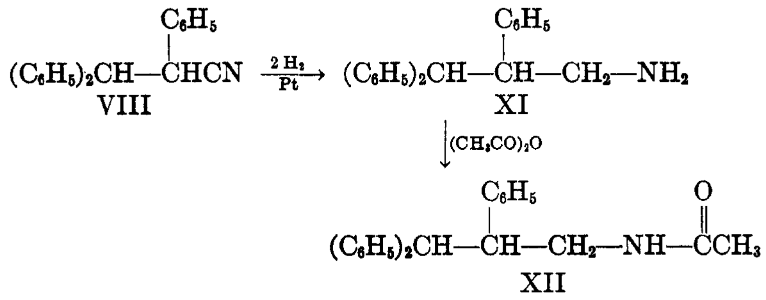
[6] Bodroux, *Compt. rend.*, **152**, 1596 (1911).

[7] See Adkins, "Reactions of Hydrogen," University of Wisconsin Press, Madison, **1937**, pp. 88, 139, for a discussion of hydrogenolysis of this type of molecule.

If the reduction of II was carried out in acetic anhydride solution, two products were obtained. One of these was the triphenylpropionitrile (VIII) and the other proved to be *N*-(2,3,3-triphenyl-*n*-propyl)acetamide (XII). The latter was evidently formed by further reduction of the nitrile (VIII).



$$\underset{\text{VIII}}{(C_6H_5)_2CH-\overset{C_6H_5}{\overset{|}{C}}HCN} \xrightarrow[Pt]{2\,H_2} \underset{\text{XI}}{(C_6H_5)_2CH-\overset{C_6H_5}{\overset{|}{C}}H-CH_2-NH_2}$$

$$\downarrow (CH_3CO)_2O$$

$$\underset{\text{XII}}{(C_6H_5)_2CH-\overset{C_6H_5}{\overset{|}{C}}H-CH_2-NH-\overset{O}{\overset{\|}{C}}CH_3}$$

In order to prepare a sample of the amide (XII), it was found more convenient to reduce the nitrile (VIII) by means of Raney nickel catalyst at a temperature of 120° and a hydrogen pressure of 2500 pounds. Subsequent acetylation of the amine (XI) gave the amide (XII), which was identical with the amide obtained from II by reduction with platinum and hydrogen.

The formation of these compounds (VIII and XII) leaves no doubt that the carbon-alkylation product is α-nitro-α,β,β-triphenylpropionitrile.

It is of interest to point out that this α-nitro-α,β,β-triphenylpropionitrile (II) is an example of a highly substituted ethane which might conceivably undergo dissociation in a manner analogous to that exhibited by pentaphenylethane.[8] However, the above-mentioned action of hydriodic acid produced no diphenylmethane and a boiling benzene solution of the compound was not affected by air. Pyrolysis did not yield tetraphenylethane, which might be expected if a diphenylmethyl radical had been produced by dissociation. The thermal decomposition products were triphenylacrylonitrile, tetraphenylethylene, benzoic acid, benzophenone and oxides of nitrogen.

EXPERIMENTAL

Silver aci-*phenylnitroacetonitrile.*—A solution of 46.0 g. of silver nitrate in 200 cc. of water was added, with vigorous stirring, to 300 cc. of water containing 50.0 g. of sodium *aci*-phenylnitroacetonitrile, prepared according to the method of Wislicenus and Enders.[9] The silver salt separated immediately as a yellow precipitate. It was collected on a filter, washed by suspending it in 500 cc. of absolute alcohol,

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[8] BACHMAN, *J. Am. Chem. Soc.*, **55**, 3005 (1933).

[9] WISLICENUS AND ENDERS, *Ber.*, **35**, 1755 (1902).

and again collected on a filter. After removing most of the liquid it was dried in a vacuum desiccator over calcium chloride, or used immediately. The yield was almost theoretical, and the salt is stable.

Anal. Calc'd for $C_8H_5N_2O_2Ag$: Ag, 40.10. Found: Ag, 40.18, 39.80.

Reaction of diphenylbromomethane with silver aci-*phenylnitroacetonitrile.*—Seventy grams of the silver salt (0.260 mole) was suspended in 200 cc. of benzene, and 67.0 g. of diphenylbromomethane (0.271 mole), prepared according to the method of Courtot,[10] was added, with stirring, over a period of one-half hour, the temperature being kept below 20° by means of a water bath. The stirring was continued for twenty-one hours at room temperature. At the end of this time the insoluble salts were removed by filtration, washed with benzene and dried in a vacuum desiccator; their total weight was 50.1 g. An analysis of this precipitate showed that it contained 5.3 per cent. of the original silver salt and 94.7 per cent. silver bromide.

The red benzene solution and the wash benzene from the silver salts was evaporated under reduced pressure, 100 cc. of 70 per cent. aqueous alcohol was added, and the solution was placed in the refrigerator overnight. Seven and one-half grams of crude α-nitro-α,β,β-triphenylpropionitrile, melting at 141–142°, crystallized. Recrystallization from alcohol, with the use of Norite to decolorize the solution, followed by two recrystallizations from acetic acid and two more from alcohol, raised the melting point to 144.5 to 145° (decomp.) which is the melting point previously recorded.[2] An analysis was made on the purified material in order to check its composition since the previously reported analyses differed somewhat from the calculated.

Anal. Calc'd for $C_{21}H_{16}N_2O_2$: C, 76.82; H, 4.88; N, 8.53; mol. wt., 328.3.
Found: C, 76.68; H, 4.77; N, 8.60; mol. wt., 316.

This represented a yield of 8.78 per cent. In other runs in which the time of stirring varied from two to forty-eight hours the yields were 9.9, 7.2, 9.7 and 10.6 per cent. When the benzene was removed at reduced pressure without heating, and the recrystallizations carried out at temperatures not exceeding 40–50° the yields were 15.0 and 18.5 per cent.

The solution from the above crystallization was evaporated in a current of air, the residue was dissolved in 150 cc. of chloroform and extracted with five 60-cc. portions of 10 per cent. aqueous sodium hydroxide. The sodium hydroxide solution was washed twice with chloroform and acidified with dilute acetic acid. The dark oil which separated was dissolved in a large volume of water, and the solution was cooled. Benzoyl cyanide oxime (10.3 g.) melting at 126–127° crystallized. This represented 28.1 per cent. of the original silver salt.

The chloroform solution from the above extraction was dried over magnesium sulfate, and the solvent was removed under reduced pressure. The residue of 67 g. was dissolved in 150 cc. of alcohol and to the solution were added 17.5 g. of hydroxylamine hydrochloride in 60 cc. of water and 25 g. of potassium hydroxide. The solution was refluxed for seven and one-half hours, during which time the evolution of ammonia was noted. The solution was cooled, 50 cc. of 25 per cent, sodium hydroxide was added, and the whole was extracted five times with ether. The alkaline solution was then poured into 500 cc. of water and acidified with acetic acid. Seven and six-tenths grams of light yellow leaflets separated, and was dried in a vacuum desiccator over calcium chloride. Without further recrystallization this benzophenone oxime melted at 139–140°. This represented 14.8 per cent. of the theoretical yield.

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[10] Courtot, *Ann. chim.*, [9], **5**, 80 (1916).

The acidified solution was extracted with ether, and the ether was removed under reduced pressure, leaving approximately 5 g. of oily residue. It was recrystallized from alcohol and a compound was obtained which charred at 238° and melted at 245–249°, with decomposition. This is probably a polymerization product of benzoylcyanide similar to that reported by Diels and Stein.[11]

The ether extract from the benzophenone oxime preparation was concentrated, and the 52 g. of red-brown oil which remained was taken up in methyl alcohol; no crystallization occurred. The solvent was removed, and the oil was distilled at 105–138°/3 mm. All fractions of the distillate showed the presence of a ketone and were combined and heated with 200 cc. of an alcoholic solution of 2,4-dinitrophenylhydrazine. A total of 31.0 g. of benzophenone 2,4-dinitrophenylhydrazone, melting at 234.5°, was obtained. This represents 16 g. of benzophenone or 33.8 per cent. of the theoretical yield. The total quantity of benzophenone recovered as the oxime and 2,4-dinitrophenylhydrazone was 48.6 per cent.

The filtrates from this hydrazone preparation, which were ketone-free, were concentrated under reduced pressure and washed with 5 per cent. hydrochloric acid, to remove any unchanged 2,4-dinitrophenylhydrazine. Twenty grams of a yellow-orange oil remaining deposited crystals after standing several weeks. These crystals were identified as benzohydryl ether melting at 107–108°. The remainder of the oil was a mixture of benzohydrol, benzohydryl ether, and diphenylbromomethane.

The 10 cc. of residue left from the distillation was sublimed at 3 mm. against a tube cooled by acetone-dry ice. The fractions up to 160° were liquid at room temperature and those from 160–240° were semi-solid. The higher fractions were crystallized from alcohol, yielding 1.0 g. of a product melting at 107–108°. A mixture with a sample of benzohydryl ether, synthesized by the method of Schenk,[12] showed no depression of the melting point. The benzohydrol and benzohydryl ether have been shown to be produced by the hydrolysis of diphenylbromomethane.[12]

Action of alcoholic potassium hydroxide on α-nitro-α,β,β-triphenylpropionitrile.—One gram of the nitrile (0.00342 mole) was suspended in 17 cc. of 1.0 per cent. methyl alcoholic potassium hydroxide (0.0034 mole) and refluxed for fourteen hours. Concentration and cooling of the solution yielded 0.7 g. of crystals which melted at 165–166° after recrystallization from methanol. A mixture of this product with synthetic triphenylacrylonitrile prepared as described below showed no depression of the melting point. It gave a violet color with concentrated sulfuric acid.

Anal. Calc'd for $C_{21}H_{15}N$: N, 4.99. Found: N, 5.08.

The filtrate was evaporated to dryness and extracted with ether. The presence of nitrites in the ether-insoluble residue was demonstrated by treatment with potassium iodide solution and hydrochloric acid.

Action of hydrogen iodide on α-nitro-α,β,β-triphenylpropionitrile.—Under an atmosphere of nitrogen in an all glass apparatus 2.4 g. of red phosphorus and 0.2 g. of iodine in 60 cc. of glacial acetic acid were allowed to react for twenty minutes. Four grams of the nitrile and 2.0 cc. of water were added, and the solution was refluxed for fifty minutes. The acetic acid solution was filtered and concentrated, yielding 1.0 g. of a substance, which, after two recrystallizations from alcohol, melted at 164.5–165.5°. A mixture with synthetic triphenylacrylonitrile showed no depression of the melting point.

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[11] Diels and Stein, *Ber.*, **40**, 1655 (1907).

[12] Schenk, *Pharm. Ztg.*, **54**, 725 (1909).

Synthesis of triphenylacrylonitrile.—The method of Bodroux[6] was used in the preparation of this compound. To 125 cc. of dry ether in a three-necked flask fitted with a mercury-sealed stirrer and a condenser were added 11 g. of sodamide and 30 g. of benzyl cyanide. The mixture was refluxed for three hours to insure complete formation of the sodium salt. At the end of this time 44 g. of benzophenone, dissolved in 60 cc. of dry ether, was added. A reaction took place immediately, and the mass became quite thick. Stirring was continued for two hours. Fifty cubic centimeters of water, followed by 50 cc. of concentrated hydrochloric acid, was added. The insoluble product was filtered and recrystallized from 400 cc. of alcohol. The yield of the product, melting at 166–166.5°, was 17.2 g. or 25 per cent. of the theoretical.

Reduction of α-nitro-α,β,β-triphenylpropionitrile.—

1. To 50 cc. of acetic anhydride was added 5 g. of the nitrile (0.0152 mole) and 0.2 g. of platinum oxide. In fourteen hours, with the addition of another 0.2 g. of platinum oxide after the sixth hour, 0.057 mole of hydrogen was absorbed. The platinum was removed by filtration, and the acetic anhydride was decomposed with water and evaporated at reduced pressure. The sticky substance which remained was repeatedly crystallized from aqueous alcohol, yielding a small quantity of a white compound melting at 100–101°, a large amount of a mixture melting between 95 and 105°, and a small amount of a compound melting at 143–144°.

2. The above procedure was repeated but the solution was poured into 40 per cent. sodium hydroxide to decompose the acetic anhydride. After dilution this was extracted three times with chloroform; the chloroform solution was dried over magnesium sulfate and evaporated at reduced pressure. The viscous oil remaining was dissolved in 10 cc. of hot ethyl acetate, and 50 cc. of high-boiling petroleum ether was added. After standing twelve hours crystals separated from the light-brown solution. After two recrystallizations from ethyl acetate and high-boiling petroleum ether 1.7 g. of product melting at 140–142° was obtained. Further recrystallizations from 70 per cent. aqueous alcohol yielded fine needles melting at 143.5–144.0°. A mixture with *N*-(2,3,3-triphenyl-*n*-propyl)acetamide, prepared as described below, showed no depression of the melting point.

Anal. Calc'd for $C_{23}H_{23}NO$: C, 83.97; H, 6.99; N, 4.25; mol. wt., 329.
Found: C, 84.14; H, 7.07; N, 4.25; mol. wt., 304.

3. To 140 cc. of absolute alcohol was added 5.0 g. of the nitrile (0.0152 mole) and 0.24 g. of platinum oxide. After twenty-five minutes 0.0529 mole of hydrogen was absorbed, and no further absorption took place during the next hour. The solution, which was strongly ammoniacal, was concentrated under reduced pressure, yielding a total of 4.0 g. of α,β,β-triphenylpropionitrile which melted at 101.5–102° after two recrystallizations from alcohol. A mixture with the low-melting compound obtained in the first reduction above showed no depression of the melting point. The yield was 93 per cent. of the theoretical.

Anal. Calc'd for $C_{21}H_{17}N$: N, 4.96. Found: N, 5.17.

Hydrolysis of α,β,β-triphenylpropionitrile.—Five-tenths of a gram of the compound melting at 101.5–102° in 12 cc. of concentrated hydrochloric acid was heated in a sealed tube at 170° for four hours.[13] The long needles which were present after the tube cooled were removed by filtration, washed by heating with sodium bicarbonate solution, and twice recrystallized from alcohol. They melted at 209°. If placed in the melting point bath at 200° the crystals melted at 210.3°. A mixture

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[13] Kohler, *Am. Chem. J.*, **35**, 401 (1906).

of this compound with a synthetic sample prepared as described below showed no depression of the melting point.

α,β,β-Triphenylpropionamide.—The necessary α,β,β-triphenylpropionic acid was synthesized by the following method.[14] In a 1-l. three-necked flask fitted with a dropping funnel, mercury-sealed stirrer, and a thermometer, were placed 50 g. of cinnamic acid and 100 cc. of dry ether. The solution was cooled to 10–15° with an ice-water bath, and 54 g. of bromine was added with stirring at such a rate that the temperature did not rise above 20°. The flask was then fitted for downward distillation, and the ether was removed, care being taken to remove the last traces of ether by heating on a water bath at reduced pressure. The cake was broken into small lumps and covered with 400 cc. of thiophene-free benzene. Stirring was started, and 70 g. of aluminum chloride was added, in small portions, to the suspension heated to 60°. After all the aluminum chloride was added the content of the flask was refluxed for about fifteen minutes. The mixture was cooled and poured on 1 kg. of cracked ice and 25 cc. of hydrochloric acid. The whole was transferred to a flask and distilled until water-clear. The solid residue was filtered from the hot aqueous solution and washed with two 1-l. portions of warm water. The dried solid was then boiled with 500 cc. of 5 per cent. sodium carbonate until most of the organic matter dissolved. The solution was filtered, and, while still hot, was decomposed with dilute hydrochloric acid. The precipitated acid was washed well with warm water and dried. It was recrystallized from hot alcohol with the aid of Norite. Colorless crystals of the α,β,β-triphenylpropionic acid which melted at 208° were obtained. Eighty grams of this acid was refluxed with 270 g. of thionyl chloride for one hour. The excess thionyl chloride was removed by distillation under reduced pressure.

To insure complete removal of the thionyl chloride 200 cc. of benzene was added to the residue and distilled. The residue was dissolved in 300 cc. of high-boiling petroleum ether and saturated with ammonia. After removal of the precipitated amide by filtration, the solution was again saturated with ammonia, and a second crop of crystals was obtained. The combined portions of the amide were dried on a suction funnel, and then boiled with two 500-cc. portions of 5 per cent. sodium carbonate solution, and then with two 300-cc. portions of water. After drying, the crude product was recrystallized three times from 500-cc. portions of alcohol. Further recrystallization did not change the melting point. A 62 per cent. yield of colorless crystals was obtained. The amide melted at 208–209°. If placed in a hot bath it melted at 210° which is 3° lower than the melting point reported by Kohler.[13]

Anal. Calc'd for $C_{21}H_{19}NO$: N, 4.65. Found: N, 4.71.

Reduction of α,β,β-triphenylpropionitrile.—To 1.3 g. of the nitrile in 100 cc. of dioxane was added 0.5 g. of Raney nickel, and this was treated with hydrogen for thirty minutes at 120° and 2500 pounds pressure.[15] The Raney nickel was removed by filtration, and the dioxane was distilled at reduced pressure. To the sticky residue was added 10 cc. of acetic anhydride and the solution refluxed for ten minutes. The acetic anhydride was decomposed by the addition of water and removed by allowing it to stand overnight in a vacuum desiccator with soda-lime. The solid residue was recrystallized once from a mixture of ethyl acetate and high-boiling petroleum ether and three times from aqueous alcohol, yielding 0.3 g. of fine needles melting at 143–144°. A mixture with *N*-(2,3,3-triphenyl-*n*-propyl)acetamide, described above, showed no depression of the melting point.

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[14] Private communication, C. F. KOELSCH, University of Minnesota.

[15] Ref. 7, page 53.

Pyrolysis of α-nitro-α,β,β-triphenylpropionitrile.—One gram of the nitrile was heated to 160°, the compound melting and decomposition taking place with evolution of nitrogen dioxide and darkening of the liquid. Crystallization of the product from alcohol yielded approximately 0.25 g. of a solid melting at 210–213°, the separation of which was accomplished by sublimation against a tube cooled by acetone-dry ice. The first fraction, about 15 mg., was collected to 205°/18 mm. After recrystallization from alcohol this melted at 165°, and gave a violet color when dissolved in sulfuric acid; a mixture with triphenylacrylonitrile showed no depression of the melting point. The second fraction of the sublimate was collected from 205–270°/18 mm. and melted at 200–205°.

The unsublimed residue, after two recrystallizations from 100-cc. portions of alcohol, melted at 222–223° and a mixture with a sample of tetraphenyl ethylene, prepared according to the method of Lohse,[16] showed no depression of the melting point.

The original alcohol filtrate was treated with 2,4-dinitrophenylhydrazone, yielding benzophenone 2,4-dinitrophenylhydrazone melting at 237–238°.

Anal. Calc'd for $C_{19}H_{14}N_4O_4$: N, 15.46. Found: N, 15.27.

The remaining solution was concentrated and added to 50 cc. of 5 per cent. sodium hydroxide, the alkaline solution was extracted with ether, acidified and again extracted with ether. The ether extract of the acidified solution was evaporated, and the residue was recrystallized from water, melting at 120°. This acid, upon treatment with thionyl chloride and then with aniline yielded benzanilide melting at 160°.

ACKNOWLEDGMENT

The authors are indebted to J. T. Thurston for the syntheses of triphenylacrylonitrile and α,β,β-triphenylpropionamide.

SUMMARY

The mixture of compounds formed in the reaction between silver phenylnitroacetonitrile and diphenylbromomethane has been examined and shown to consist of:

(*a*) α-nitro-α,β,β-triphenylpropionitrile,
(*b*) benzophenone,
(*c*) benzoylcyanide oxime,
(*d*) unchanged silver salt,
(*e*) decomposition products of the above; a *tris*-benzoylcyanide, benzohydrol and benzohydryl ether

The amounts of these products isolated indicated that carbon alkylation had taken place to the extent of 10 to 18 per cent., and that oxygen alkylation amounted to about 48 per cent.

Further proof of the structure assigned to α-nitro-α,β,β-triphenylpropionitrile has been obtained by showing that catalytic reduction produces α,β,β-triphenylpropionitrile and 2,3,3-triphenyl-*n*-propylamine which was isolated as its acetyl derivative.

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[16] Lohse, *Ber.*, **29**, 1789 (1896).