is 75°;[20] these authors also reported its conversion to cinnamic acid by boiling hydrochloric acid, a result which we duplicated. A sample of the acid (0.6 g.) was heated to boiling with 25 cc. 6.0*N* hydrochloric acid and gave, on cooling, crystalline cinnamic acid (0.45 g.), melting point and mixed melting point with authentic cinnamic acid, 130–132°.

Oxidation of 2-(β-styryl)-4-methyl-1,3-dioxolane (VIa) *with peracetic acid catalyzed by sulfuric acid.* To 76 g. (0.4 mole) of 2-(β-styryl)-4-methyl-1,3-dioxolane containing 0.76 g. of sulfuric acid was added with stirring 157 g. of a 24.3% solution of peracetic acid in ethyl acetate (*i.e.*, containing 38.0 g. or 0.5 mole peracetic acid) over a period of 1 hr. The reaction was exothermic from the outset, and the temperature was maintained at 25–33° by ice-water cooling. After an additional 4 hr. at 30°, titration for peracetic acid indicated the theoretical amount had been consumed. Sodium acetate, 1.25 g., was added to neutralize the sulfuric acid. Distillation through a glass helix-packed column gave, after recovery of solvent and acetic acid, 23 g. (28% yield) of impure propylene glycol monocinnamate, b.p. *ca.* 195°/1.25 mm., n_D^{30} 1.5483.

Anal. Calcd. for $C_{12}H_{14}O_3$: C, 69.88; H, 6.84. Found: C, 71.92; H, 7.25.

1,1-Diacetoxy-2-ethyl-2-butene. To acetic anhydride (217 g., 2.15 moles) containing 0.34 g. of a mixed boric-oxalic acid catalyst[13] (1:1 molar ratio) was added 139 g. (1.42 moles) of 2-ethylcrotonaldehyde over a period of 35 min. The solution was stirred constantly, and slight cooling was required to maintain a temperature of 30°. After the mixture had stood for an additional 20 hr., the catalyst was neutralized with 0.93 g. of sodium acetate, and the reaction mixture was fractionated directly to give 212 g. (75% yield) of 1,1-diacetoxy-2-ethyl-2-butene, b.p. 88–90°/4 mm., n_D^{30} 1.4339.

Anal. Calcd. for $C_{10}H_{16}O_4$: C, 59.98; H, 8.05. Found: C, 60.58; H, 8.21.

1,1-Diacetoxy-2,3-epoxy-2-ethylbutane. To 200 g. (1.0 mole) of 1,1-diacetoxy-2-ethyl-2-butene was added 396 g. of a solution containing 23.0% peracetic acid in ethyl acetate (91 g. or 1.2 moles peracetic acid). The addition and subsequent stirring (both at 60°) required 4 hr., at the end of which time the theoretical quantity of peracid was consumed. The cooled reaction mixture was fed to ethylbenzene under reflux in a still at 30 mm. pressure while acetic acid and unchanged peracetic acid was removed continuously at the still head as an ethylbenzene azeotrope. Fractionation of the residue gave 119 g. (55% yield) of 1,1-diacetoxy-2,3-epoxy-2-ethylbutane, b.p. 95–96°/3.0 mm., n_D^{30} 1.4289.

Anal. Calcd. for $C_{10}H_{16}O_5$: C, 55.54; H, 7.46. Found: C, 55.46; H, 7.42.

1,1-Diacetoxy-2-ethyl-2-hexene. This preparation based on 2-ethyl-3-propyl acrolein and acetic anhydride was similar to that for 1,1-diacetoxy-2-ethyl-2-butene. Yield of 1,1-diacetoxy-2-ethyl-2-hexene was 75%, b.p. 91–95°/2.0 mm., n_D^{30} 1.4366.

Anal. Calcd. for $C_{12}H_{20}O_4$: C, 63.13; H, 8.83. Found: C, 63.48; H, 8.82.

1,1-Diacetoxy-2,3-epoxy-2-ethylhexane. The epoxidation of the above olefin was analogous to the preparation of 1,1-diacetoxy-2,3-epoxy-2-ethylbutane with the following results. Yield of 1,1-diacetoxy-2,3-epoxy-2-ethylhexane was 72%, b.p. 102–103°/1.25 mm.; n_D^{30} 1.4308.

Anal. Calcd. for $C_{12}H_{20}O_5$: C, 59.00; H, 8.25. Found: C, 59.17; H, 7.90.

South Charleston, W. Va.

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(20) W. Schreuth, W. Schoeller and R. Streunsee, *Ber.*, **44**, 1432 (1911).

[Contribution from the Summit Research Laboratories, Celanese Corp. of America]

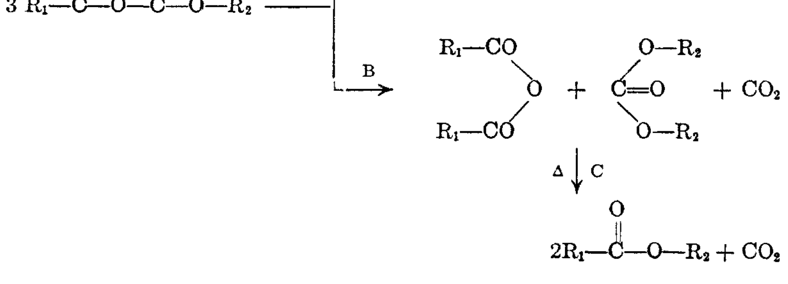
Decomposition of Mixed Carboxylic-Carbonic Anhydrides[1a]

THOMAS B. WINDHOLZ[1b]

Received June 22, 1959

The decomposition of a number of mixed carboxylic-carbonic anhydrides has been investigated and the occuring reactions established. It was found that the structures of both the carboxylic (R_1) and carbonic (R_2) components have directing influences on the possible paths of decomposition. Polar and steric effects as well as proposed reaction mechanisms are discussed. A method of preferential ester formation from mixed anhydrides is demonstrated for ethyl benzoate.

The preparation and characterization of stable carboxylic-carbonic anhydrides was first reported by Tarbell and Leister.[2] Since then it has been established[3a,3b] that mixed carboxylic-carbonic anhydrides decompose according to Equations A and B



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(1)(a) Part of this work has been presented before the Division of Organic Chemistry at the 136th Meeting of the American Chemical Society, Atlantic City, September 1959.

(1)(b) Present Address: Merck Sharp and Dohme Research Laboratories, Rahway, N. J.

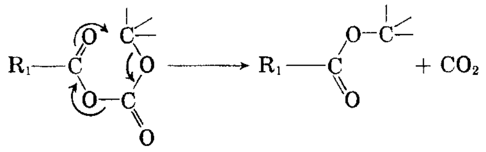
Decomposition may occur exclusively by either path A or B, or by both paths simultaneously with the formation of ester, symmetrical anhydride, carbonate, and carbon dioxide. In the present work it has been found that in some cases the ratio of products formed may vary with the temperature of decomposition. Certain mixed anhydrides which decompose equally by path A and B at 160°, yield more ester than symmetrical anhydride when the temperature of decomposition is raised to 200°. When the temperature is 250° however, ester is the only decomposition product. This fact suggests a secondary reaction (C) between symmetrical anhydrides and carbonates and it was possible to demonstrate this reaction experimentally by the preparation of certain esters in very good yields by this procedure at elevated temperatures. From these experiments it was concluded that at the decomposition temperatures reported in the table below, the isolated esters are formed only according to reaction path A, and the possibility of reaction C occurring under these conditions can be excluded.

The procedure for the preparation and quantitative decomposition of the mixed anhydrides is described in the experimental part and includes the volumetric determination of the amount of carbon dioxide formed. This amount was in good agreement with the ratio of ester to symmetrical anhydride that was obtained. Since the instability of some mixed anhydrides made it necessary to decompose them *in situ,* it was necessary to decompose all mixed anhydrides in this way without isolation and purification so that standard conditions would be maintained. To ascertain whether the possibility of impurities present in the crude carboxylic-carbonic anhydride may influence the direction of decomposition, a number of stable mixed anhydrides were prepared and purified according to Tarbell and Leister[2] and submitted to decomposition. These results were in good agreement with the values obtained with the crude mixed anhydrides. Decomposition temperatures for unstable mixed anhydrides were determined in the original tetrahydrofuran solution in order to obtain information on their relative stabilities. In the isolation and characterization of reaction products heating was avoided to prevent any secondary reactions.

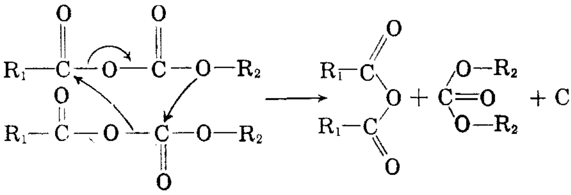
The results of the experiments with carboxylic-carbonic anhydrides are listed in Table I. Relative yields of esters and symmetrical anhydrides are considered sufficient to represent reaction paths A and B.

A number of mixed anhydrides derived from aliphatic acids decompose exclusively by path A (Experiments 1–5) while aromatic derivatives tend to decompose equally along paths A and B· The differences in reactivity are not fully understood, although steric factors are apparently significant. Directing influences of the alkyl radicals R_2 have been examined only for a few typical examples. These experiments indicate that the more electron releasing isopropyl group favors easier decomposition by path A. Phenyl as R_2 favors path B as shown by the high yields of symmetrical anhydride obtained in Experiments 10 and 15. This effect is counterbalanced only in the cases where the acyl radicals have been shown to cause ester formation exclusively (*cf.* Experiment 5).

An S_{Ni}-type reaction mechanism, involving a six membered cyclic transition state is suggested



to explain ester formation (path A) by intramolecular decarboxylation.[4] The disproportionation (path



B) can best be represented as in accordance with the disproportionation mechanism of phenyl chloroformates suggested by Wiberg and Shryne.[5]

A number of experiments were made to influence the direction of decomposition of benzoic-ethyl carbonic anhydride, which was considered to be a suitable model compound. It was noticed that heating the mixed anhydride in the presence of triethylamine hydrochloride lowered the decomposition temperature and favored ester formation. Triethylamine alone lowered the decomposition temperature but the ratio of the products formed, remained unchanged. Boron trifluoride etherate has proved to be most efficient in directing the decarboxylation of benzoic-ethyl carbonic anhydride. It lowered the decomposition temperature considerably and caused exclusively ethyl benzoate formation. Acid catalysis of this type substantiates the mechanism proposed for path A. This method of preparing esters from mixed anhydrides is being investigated further.

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[2] D. S. Tarbell and N. A. Leister, *J. Org. Chem.,* **23,** 1149 (1958).

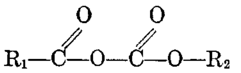
[3] (a) Preliminary communication: T. B. Windholz, *J. Org. Chem.,* **23,** 2044 (1958); (b) D. S. Tarbell and F. J. Longosz, *J. Org. Chem.,* **24,** 774 (1959).

[4] R. Boschan: *J. Am. Chem. Soc.* **81,** 3341 (1959) has postulated a four-membered cyclic transition state with acyl fission based largely on analogies with the preparation of nitrate esters from alkylchloroformates. The observation of Tarbell and Longosz, ref. 3b that ester formation proceeds with retention of configuration, supports our proposed mechanism.

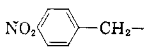
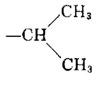
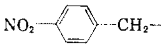
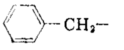
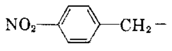
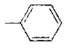
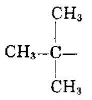
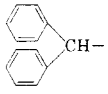
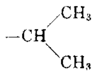
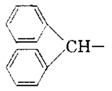
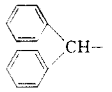
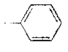
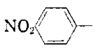
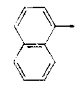
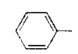
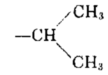
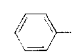
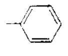
[5] K. B. Wiberg and T. M. Shryne, *J. Am. Chem. Soc.,* **77,** 2774 (1955).

TABLE I

DECOMPOSITION OF CARBOXYLIC-CARBONIC ANHYDRIDES



$R_1-C(=O)-O-C(=O)-O-R_2$

Expt. No.	R_1	R_2	Decomp. Temp. in THF	Decomp. Temp. When isolated	% of CO_2 formed[a]	% Products formed: Ester (A)	% Products formed: Symmetrical Anhydride (B)
1	$CN-CH_2-$	$-C_2H_5$	25	...	97	95[b]	...
2		 $-CH(CH_3)_2$	25	...	92	95[c]	...
3		$-C_2H_5$	25–40	...	94	90[d]	...
4		C_2H_5	35–60	...	92	90[b]	...
5			25–40	...	88	90[c]	...
6	$CH_3-CH_2-CH_2-$	$-C_2H_5$	...	120–130	85	75[e]	15
7	 $CH_3-C(CH_3)_2-$	$-C_2H_5$	...	120–130	70	50	30
8		 $-CH(CH_3)_2$	...	f	..	20[d]	60
9		$-C_2H_5$	...	c	..	10[c]	70
10			...		..	...	95[d,g]
11		$-C_2H_5$	...	150–160	77	50[c]	50
12		$-C_2H_5$	...	150–160	72	50[e]	50
13		 $-CH(CH_3)_2$	...	160–170	78	60[e]	40
14		$-C_2H_5$	...	175–185	72	50[c]	50
15			...	f	..	10	80[d,g]

[a] Theoretical yield calculated for path A. [b] Distilled from the reaction mixture, characterized by boiling point, analysis and comparison of infrared spectrum with an authentic sample. [c] See experimental part. [d] Isolated from the reaction mixture, identified by analysis and melting point. [e] Ratios of formed products were satisfactorily determined by preparing artificial mixtures of the expected products and comparing the infrared spectra. [f] Decomposed in a few hours at room temperature. [g] Diphenyl carbonate was isolated in nearly the same yield from the reaction mixture.

It should be mentioned that the decarboxylation of alkyl chloroformates is reported[6] to be catalyzed by pyridine and boron trifluoride.

EXPERIMENTAL

Melting points are uncorrected. Microanalyses are by Mr. Grant Gustin of our Analytical Department. Infrared spectra were taken on a Perkin Elmer Infracord, Model 137. Commercially available carboxylic acids, dry tetrahydrofuran, and dry triethylamine were used in all preparations. The chloroformates were obtained from the Food Machinery and Chemical Corporation and redistilled before use. All preparations and decompositions of mixed anhydrides were run in a closed system equipped for volumetric determination of the carbon dioxide formed.[7]

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(6) S. Nakanishi, T. C. Myers, and E. V. Jensen, *J. Am. Chem. Soc.*, **77**, 5033 (1955).

I. *Preparation of esters. A. From a mixed anhydride. Glycol dibenzoate.* Benzoic-ethylene carbonic anhydride was prepared from benzoic acid and ethylene glycol bischloroformate, according to the general procedure described below. The product isolated by crystallization from petroleum ether (b.p. 45–60°) melted at 70–72° and had infrared bands at 1820, 1740 cm^{-1}.

Anal: Calcd. for $C_{18}H_{14}O_8$: C, 60.40; H, 3.90. Found: C, 60.27; H, 3.81.

The crystalline mixed anhydride was gradually heated and at 130°–150° approximately 72% of the theoretical carbon dioxide was eliminated. The infrared spectrum of the reaction products was identical with that of an artificial mixture of equimolecular amounts of glycol dibenzoate, benzoic anhydride, and ethylene carbonate. When heating was continued to 250°, additional carbon dioxide was eliminated, and the mixture showed an increase in glycol dibenzoate content. In another experiment 3.58 g. (0.01 mole) of the same mixed anhydride was heated directly to 275°. A loss in weight of 0.82 g. was recorded, in good agreement with the nearly quantitative carbon dioxide elimination. The solid residue, after one recrystallization from methanol, yielded 2.20 g. (82%) glycol dibenzoate, m.p. 73–74°, undepressed by an authentic sample prepared from ethylene glycol and benzoyl chloride.

B. From symmetrical anhydrides and neutral carbonates. Glycol dibenzoate. A mixture of 2.26 g. (0.01 mole) of benzoic anhydride and 0.88 g. (0.01 mole) of ethylene carbonate was heated and at 220–275°, 92% of the theoretical amount of carbon dioxide was formed. The cooled product was recrystallized from methanol yielding 2.17 g. (80%) glycol dibenzoate, m.p. 73–74°.

Phenyl benzoate. On heating equimolecular amounts of benzoic anhydride and diphenyl carbonate at 205°–245°, 90% of the ester melting at 70°, was obtained.

Attempted preparation of ethyl benzoate. No reaction took place between benzoic anhydride and diethyl carbonate at 200°. Attempts to promote the reaction by heating the reactants in the presence of ethyl benzoate, benzoic-ethyl carbonic anhydride or boron trifluoride etherate also failed to produce the expected ester.

II. Preparation and decomposition of mixed carboxylic-carbonic anhydrides. A. Preparation. A solution of 0.02 mole of carboxylic acid and 0.02 mole of triethylamine in 35 ml. tetrahydrofuran was cooled to −1° to −5°. To this stirred solution was added a solution of 0.02 mole of the chloroformate in 15 ml. of tetrahydrofuran maintaining the above temperature during addition and for an additional 30 min. The stirred mixture was allowed to warm to room temperature and held there for 30 min.

B. Decomposition. 1. Stable compounds. When the mixed anhydrides were stable, *i.e.* no carbon dioxide evolution occurred at this point, the precipitated triethylamine hydrochloride was filtered, washed, and dried, and isolated in nearly quantitative yield. The salt was also checked for water solubility and its solution acidified to indicate any unchanged carboxylic acid. The tetrahydrofuran filtrate was concentrated *in vacuo* to constant weight at room temperature. The stable products obtained from experiments 6, 7, 11, 12, 13, and 14 were heated without further purification, the carbon dioxide formed was determined and the decomposition products identified. The emphasis was on the identification of esters and anhydrides, since the more volatile carbonates were frequently evaporated during the concentration step. Crystalline products were isolated and characterized, avoiding any heating. Ratios of oily products were satisfactorily determined by preparing artificial mixtures of the expected materials and comparing the infrared spectra. To check the results, experiments 11, 12 and 14 were also run with pure mixed anhydrides prepared as described by Tarbell and Leister.[2] These had the same infrared spectra as the crude mixed anhydrides and on heating gave the same amounts of decomposition products. A few examples are given to illustrate typical procedures.

p-Nitrobenzoic–ethyl carbonic anhydride,[2] 2.40 g. (0.01 mole) was heated at 150–180° until evolution of carbon dioxide ceased; at this point 77% of the theoretical amount was collected. Treatment with petroleum ether (b.p. 45–60°) and ether yielded 0.80 g. of insoluble product having only anhydride absorption. The filtrate on concentration gave a fluffy yellow powder which on crystallization from ethanol gave 0.96 g. (49%) of ethyl nitrobenzoate, m.p. 56–57°. The 0.80 g. insoluble product was treated with cold ethylacetate and filtered yielding 0.71 g. (46%) of *p*-nitrobenzoic anhydride, m.p. 189°.

2. Unstable compounds. Whenever the mixed anhydrides were unstable as shown by carbon dioxide evolution in their preparation, the reaction mixtures were kept for 2 hr. at 0°, cooled to −5° and the triethylamine hydrochloride filtered. Even in these experiments the salt was obtained in nearly quantitative yield. The cold filtrate was divided into two parts. One half was allowed to warm up to room temperature and heated if necessary to complete the carbon dioxide elimination. The other half was concentrated *in vacuo* without heating. The decomposition products were characterized and their ratios found to be almost identical in both cases.

p-Nitrophenylacetic–isopropyl carbonic anhydride. The tetrahydrofuran filtrate decomposed at room temperature in 30 min. After concentration, the reaction product (2.12 g.) was a semisolid material which showed only traces of anhydride absorption in the infrared. Isopropyl *p*-nitrophenylacetate,[8] m.p. 33–34° was isolated by careful addition of water to an ethanol solution of the reaction product.

Diphenylacetic–ethyl carbonic anhydride. The tetrahydrofuran filtrate was refluxed for 20 min. No carbon dioxide was eliminated. The mixture was cooled, and concentrated to give a semicrystalline residue. On treatment with petroleum ether (b.p. 45–60°) ether, 3.15 g. of solid was obtained. After washing with cold ethanol, 2.70 g. (66.5%) of diphenylacetic anhydride, m.p. 96–97° remained. The petroleum ether–ether filtrate on concentration yielded 0.42 g. (9%) of ethyl diphenylacetate, m.p. 59°.

III. *Directed decarboxylation of benzoic–ethyl carbonic anhydride.* When the mixed anhydride was prepared in tetrachloroethane instead of tetrahydrofuran, the triethylamine hydrochloride did not precipitate. Carbon dioxide was eliminated on refluxing to 145° and was completed after 45 min. The solution was cooled, washed with water, and concentrated. It contained 75% ethyl benzoate and 25% benzoic anhydride as shown by infrared analysis. Refluxing pure mixed anhydride in tetrachloroethane in the absence of triethylamine salt produced no decomposition and the mixed anhydride was recovered unchanged after 1 hr. When pure benzoic–ethyl carbonic anhydride was heated in the presence of triethylamine, decarboxylation started at 120° and was terminated at 140°. Equal amounts of ester and symmetrical anhydride formed, as shown by infrared analysis.

Addition of 25% of the molar amount of boron trifluoride etherate to the mixed anhydride caused slow elimination of carbon dioxide accompanied by the evolution of heat. However further heating was needed to complete gas evolution. After 30 min. the brown mixture was dissolved in ether, washed with dilute hydrochloric acid, bicarbonate solution, water, then dried and concentrated. It consisted of 85% ethyl benzoate and 15% benzoic anhydride. Addition of one molar amount of boron trifluoride etherate to the mixed an-

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(7) The apparatus was a modification of that described by W. M. Schubert, *J. Am. Chem. Soc.*, **71**, 2639 (1949).

(8) H. P. Ward and E. F. Jankins, *J. Org. Chem.*, **10**, 371 (1945).

hydride caused exothermic decarboxylation with elimination of the theoretical amount of carbon dioxide. The reaction mixture was worked up by hydrolizing the boron trifluoride complex that was formed. No more anhydride could be detected, the decarboxylation yielding ethyl benzoate exclusively, in excellent yield.

Acknowledgments. The author expresses his appreciation to Dr. J. B. Clements and Dr. A. B. Conciatori for reviewing this manuscript and to Mr. Harry Barnum for skilled technical assistance.

SUMMIT, N. J.

[CONTRIBUTION FROM THE CARWIN CO.]

Ketones as Catalysts in the Reduction of Aromatic Nitrogen Compounds

ADNAN A. SAYIGH

Received April 5, 1960

The use of aromatic ketones in catalytic amounts in the methanolic sodium hydroxide reduction of *o*-nitrotoluene has considerably improved the yields of the desired azoxytoluene; further reduction of the latter and of azoxybenzene to their hydrazo derivatives has been effected by the use of a catalytic amount of 9-fluorenone.

Aromatic hydrazo compounds are available *via* the reduction of azoxy or azo compounds,[1-10] which are intermediates in the properly controlled reduction of nitro compounds. The direct reduction of nitro compounds to the corresponding hydrazo derivatives can be accomplished with zinc dust and sodium hydroxide or electrolytically; both procedures have been used to give the simplest member, hydrazobenzene.[7,11]

The chemical reduction of *o*-nitrobromobenzene to dibromohydrazobenzene is accomplished with the halo groups remaining intact[12]; many electrolytic reduction procedures have been reported with yields varying from 50 to 95%.[10,13]

A magnesium-magnesium iodide system has also been employed as a reducing agent for the azobenzenes.[14]

The earliest method of reducing aromatic nitro compounds[4] to their azoxyderivatives by the use of methanolic sodium methoxide is limited in its application, mainly because the reduction of the nitro compounds does not proceed beyond the azoxy stage, and it is confined to substituted nitrobenzenes in which the substituents are not affected by the reagent. Thus, in the case of *o*-chloronitrobenzene, considerable displacement of the chloride with methoxide ion occurs, and in the case of nitrotoluenes, extensive intramolecular oxidation-reduction takes place.[15]

In order to extend the scope of this method to the reduction of azoxy and azo compounds to their hydrazo derivatives and to improve the yields of the former, a number of catalysts were investigated. In the reduction of *o*-nitrotoluene (Table I), the addition of 3–5% of a ketone as a catalyst to the reducing medium has considerably increased both the yield and the purity of azoxytoluene; also, 9-fluorenone is an effective catalyst in the reduction of azo and azoxy benzene and toluene (Table I) to their corresponding hydrazo derivatives.

Bamberger, *et al.*[16] proposed that the formation of azoxybenzene in the reduction of nitrobenzene results from condensation between the intermediates phenyl hydroxylamine and nitrosobenzene; this has been further confirmed by Ogata, *et al.*,[17] who also found that a rapid equilibrium takes place between phenyl hydroxylamines and nitrosobenzene. The poor yield of azoxytoluene encountered in the methanolic sodium hydroxide reduction of *o*-nitrotoluene could be attributed in

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(1) H. E. Bigelow and D. B. Robinson, *Org. Syntheses*, Coll. Vol. III, 103 (1955).

(2) R. F. Nystrom and W. G. Brown, *J. Am. Chem. Soc.*, **70**, 3738 (1948).

(3) M. Busch and K. Schulz, *Ber.*, **62**, 1458 (1929).

(4) H. S. Fry and J. L. Cameron, *J. Am. Chem. Soc.*, **49**, 864 (1927); C. M. Suter and F. B. Dains, *J. Am. Chem. Soc.*, **50**, 2733 (1928).

(5) H. E. Bigelow and A. Palmer, *Org. Syntheses*, Coll. Vol. II, 57 (1943).

(6) G. Bacharach and R. Weinstein, *Rec. trav. chim.*, **54**, 932 (1935).

(7) P. Ruggli and M. Kinovker, *Helv. Chim. Acta*, **17**, 396 (1934).

(8) L. Zechmeister and P. Rom, *Ann.*, **468**, 128 (1929).

(9) C. Weygand, *Organic Preparations*, Interscience Publishers, New York, 1945, p. 249.

(10) S. Swann, Jr., *Technique of Organic Chemistry*, A. Weissberger, Ed., Vol. II, Interscience Publishers, New York, 1956, p. 486.

(11) L. Gattermann and H. Wieland, *Laboratory Methods of Organic Chemistry*, The Macmillan Company, New York, 1938, p. 184.

(12) H. R. Snyder, C. Weaver, and C. D. Marshall, *J. Am. Chem. Soc.*, **71**, 289 (1949).

(13) S. Swann, Jr., *Trans. Electrochem. Soc.*, **69**, 307 (1936); **77**, 479 (1940).

(14) W. E. Bachmann, *J. Am. Chem. Soc.*, **53**, 1524 (1931).

(15) *Chem. Zentr.*, **1908**, I, 1345; O. Fischer and E. Hepp *Ber.*, **26**, 2231 (1893).

(16) E. Bamberger and E. Renauld, *Ber.*, **30**, 2278 (1897); E. Bamberger and A. Rising, *Ann.*, **316**, 257 (1901); V. O. Lukashevich, *Compt. rend. acad. sci. U.R.S.S.*, **21**, 376 (1938); *Chem. Abstr.*, **33**, 3769 (1939).

(17) Y. Ogata, M. Tsuchida, and Y. Takagi, *J. Am. Chem. Soc.*, **79**, 3397 (1957).