1,6-Didesoxy-L-glucitol (= 1,6-Didesoxy-D-gulitol) (VII).—Desulfurization of 10.7 g. of the mercaptal VI with 110 g. of Raney nickel in the usual manner yielded 5 g. of a sirup which failed to crystallize in the course of several days. In order to characterize the 1,6-didesoxy-L-glucitol, therefore, the sirup was dissolved in 10 ml. of concentrated hydrochloric acid, the mixture was cooled to 0°, and 5 ml. of benzaldehyde was added. Crystallization occurred immediately, and after one hour at 0° the mixture was diluted with ice water, filtered, washed and dried. The crude product weighed 7 g. Three recrystallizations from absolute ethanol furnished fine needles of a substance which was shown by analysis to be 1,6-didesoxy-2,3,4,5-dibenzylidene-L-glucitol. It melted at 171–173° when heated at the rate of 0.5° per minute and at 176–178° when heated 2° per minute. The $[\alpha]^{20}\text{D}$ value was +12.1° in chloroform (*c*, 4).

Anal. Calcd. for $C_{20}H_{22}O_4$: C, 73.59; H, 6.80. Found: C, 73.51; H, 7.00.

The mother liquors from the recrystallization of the dibenzylidene compound yielded about 0.4 g. of a levorotatory substance ($[\alpha]^{20}\text{D}$ −3.2° in chloroform) which, after four recrystallizations from a mixture of chloroform and pentane as needles, melted at 170–172°. From its analysis it is presumed to be a 1,6-didesoxymonobenzylidene-L-glucitol.

Anal. Calcd. for $C_{13}H_{18}O_4$: C, 65.53; H, 7.61. Found: C, 65.71; H, 7.68.

1,6-Didesoxy-2,3,4,5-tetraacetyl-L-glucitol.—Acetylation of 2.7 g. of sirupy 1,6-didesoxy-L-glucitol (VII) by heating with 0.7 g. of fused sodium acetate and 12 ml. of acetic anhydride for two hours on the steam-bath, followed by pouring the solution on cracked ice, produced an oil which crystallized when the mixture was left overnight in the refrigerator. The acetate weighed 3.6 g. It separated from chloroform upon the addition of ether and pentane as clusters of acicular prisms; after three such recrystallizations it melted at 80–82° and showed $[\alpha]^{20}\text{D}$ −25.5° in chloroform (*c*, 1).

Anal. Calcd. for $C_{14}H_{22}O_8$: C, 52.82; H, 6.97; CH_3CO, 54.1. Found: C, 52.96; H, 6.99; CH_3CO, 54.3.

Acknowledgment.—The authors wish to thank Mr. John T. Sipes for preparing the D-gluco-D-*gulo*-heptose, and Mr. William C. Alford, Mrs. Margaret M. Ledyard, Miss Paula M. Parisius and Mrs. Evelyn G. Peake for carrying out the microchemical analyses.

BETHESDA, MARYLAND RECEIVED MARCH 9, 1951

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY AND THE PURDUE RESEARCH FOUNDATION, PURDUE UNIVERSITY]

The Reactions of Succinic Anhydride with Hydrazine Hydrate[1,2,3]

BY HENRY FEUER, G. BRYANT BACHMAN AND EMIL H. WHITE[4]

The reaction of succinic hydrazide with hydrazine hydrate has been studied under various conditions. Previous investigators have obtained only oily products. We have found that disuccinhydrazide diacid, succinhydrazide and polysuccinhydrazide may be obtained according to the relative amounts of the reagents used and the conditions employed. The reaction to form succindihydrazide has been shown to involve disuccinhydrazide diacid and the hydrazonium salt of succinhydrazide monoacid as intermediates. Cyclic succinhydrazide, a new compound, has been prepared by the reduction of cyclic maleic hydrazide. Its properties indicate that earlier investigators who report cyclic succinhydrazide had actually prepared polysuccinhydrazide. Perhydro-1,4,6,9-tetraketopyridazo-(1,2-a)-pyridazine, representing a new bicyclic system, has been prepared by the reaction of cyclic succinhydrazide with succinyl chloride or diethyl succinate.

In an attempt to prepare succindihydrazide (III) by treating succinic anhydride with hydrazine hydrate, it was discovered that several products are formed, depending upon the reaction conditions. The literature contains only one reference to the above reaction, the authors[5] stating that an oil was obtained as the only product. We have found that this reaction is an excellent method for the preparation of disuccinhydrazide diacid (I), succindihydrazide (III) and a polymeric material to which the name polysuccinhydrazide (IV) is assigned. Compound I is produced as an immediate precipitate by the addition of one-half mole of hydrazine hydrate to one mole of succinic anhydride in ethanol. Addition of more than this quantity of hydrazine hydrate leads to dissolution of I and separation of an oil phase. The latter is probably the oil to which Alexa[5] referred. Heating converts the oil into compound III which dissolves in the ethanolic phase. The data of Table I indicate that the oil first formed is composed of compounds I and II, and excess hydrazine hydrate. As heating proceeds, the oil becomes richer in II. The acids I and II exist, no doubt, in the oil phase in the form of their hydrazonium salts. Compound II can be isolated as the hydrazonium salt if the oil is evaporated *in vacuo* for several days. However, a part of the acid is dehydrated by this operation to succindihydrazide (III). It is preferable to determine the amount of II in the oil phase by isolating its monobenzal derivative.

TABLE I

COMPOSITION OF THE OIL PHASE[a]

Initial temp., °C.	Reaction time,	Reaction temp., °C.	Hydrazine hydrate, %	Disuccinhydrazide diacid (I), %	Succinhydrazide monacid (II), %	Ratio of (I) to (II)
30	1 min.	30	29.3	60.7	14.0	4.34
70	10 min.	60	21.9	51.3	15.4	3.33
70	15 hr.	30	32.8	37.9	32.0	11.8
70	10 hr.	60	27.9	35.6	39.3	0.91

[a] In these runs, succindihydrazide was not detected in the oil phase.

Polysuccinhydrazide (IV) is obtained by the reaction of equimolar quantities of succinic anhydride and hydrazine hydrate, with or without a solvent. It is an extremely insoluble material, for which no suitable solvent has been found. Alkali decomposes it to hydrazine hydrate and a salt of succinic acid. Refluxing with hydrazine

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(1) Presented before the Division of Organic Chemistry at the Chicago Meeting of the American Chemical Society, September 7, 1950.

(2) Abstracted from a thesis by Emil H. White, submitted to the Faculty of the Graduate School of Purdue University in partial fulfillment of the requirements for the degree of Doctor of Philosophy, February, 1950.

(3) Financial support of this research was supplied by the United States Office of Naval Research.

(4) Department of Chemistry, University of Chicago, Chicago, Illinois.

(5) V. Alexa and G. Gheorghiu, *Bull. soc. chim.*, [4] 49, 1112 (1931).

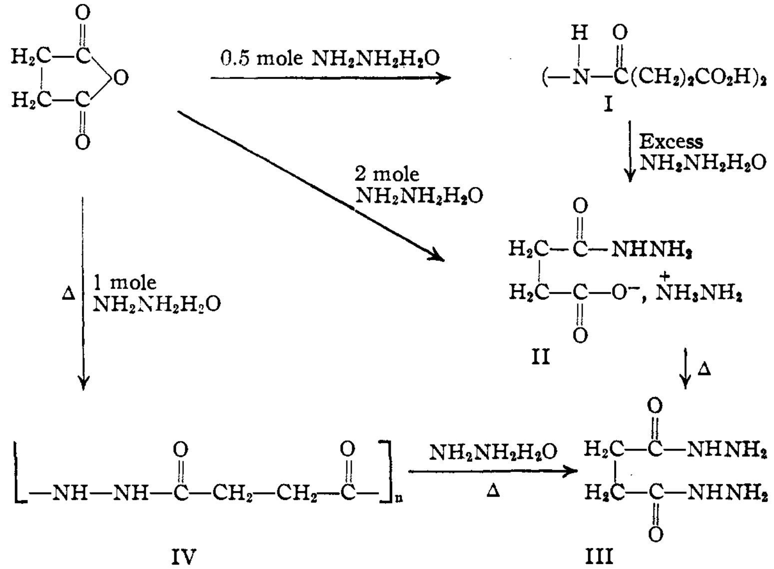

Fig. 1.—Reactions of succinic anhydride with hydrazine hydrate.

hydrate converts it to succindihydrazide (III). These reactions, and the fact that IV can also be prepared by condensation of hydrazine hydrate with ethyl hydrogen succinate, succinic acid or disuccinhydrazide diacid point to the assigned structure.

The reaction of polymer IV with benzenesulfonyl chloride yields the monobenzenesulfonyl derivative of cyclic succinhydrazide (V) and a bicyclic disuccinhydrazide, perhydro-1,4,6,9-tetraketopyridazo[1,2-a]pyridazine (VI). The latter may also be prepared by treating IV with diethyl succinate. The structure of compounds V and VI is indicated by their preparation from the cyclic succinhydrazide (VIII) as shown in Fig. 2, and also by their analyses and molecular weights as determined by the Rast method.

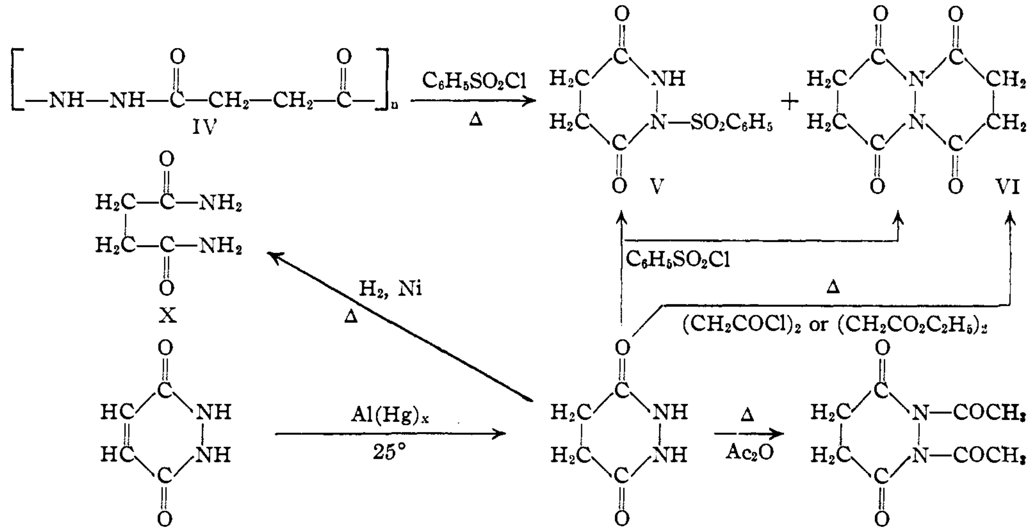

Fig. 2.—Formation and reactions of the cyclic hydrazides of succinic acid.

Sernagiotto[6] and Curtius[7] report the preparation of cyclic succinhydrazide (VIII) by the dehydration of the monohydrazonium salt of succinic acid or by the rearrangement of N-aminosuccinimide. The properties which these authors claim for VIII indicate, however, that the compound they obtained was actually the polymer IV.

We prepared authentic cyclic succinhydrazide by the reduction of maleic hydrazide, but were unable to obtain it by treating equimolar amounts of hydrazine hydrate with diethyl succinate, ethyl hydrogen sulfate or succinimide.

Polymer IV is an insoluble, amorphous powder, whereas the cyclic succinhydrazide (VIII) is a crystalline substance, soluble in water, methanol and ethanol. It forms a diacetate (IX), is readily reduced to succinamide (X), and reacts with succinyl chloride as well as diethyl succinate to give the bicyclic disuccinhydrazide (VI). Compound VIII like polymer IV gives a mixture of compounds, V and VI, when heated with benzenesulfonyl chloride. However, in contrast to IV, compound VIII may be converted to succindihydrazide (III) by treating with hydrazine hydrate at 25°. The structure of the cyclic succinhydrazide (VIII) is indicated by its synthesis, its analysis and by the fact that it titrates potentiometrically as a monobasic acid. This distinguishes it from the known N-aminosuccinimide[7] which shows basic properties, which is hygroscopic, and whose diacetate melts about 60° lower than the diacetate of VIII.

Experimental[8]

Disuccinhydrazide Diacid (I).—A 10.0-g. (0.10 mole) sample of succinic anhydride was dissolved in 150 ml. of ethanol at 60° and 2.95 g. (0.05 mole) of 85% hydrazine hydrate in 25 ml. of ethanol was slowly added. The mixture was allowed to stand for 3 hours, and then filtered. The white platelets were washed with ethanol, dried at 100°,

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[6] E. Sernagiotto and M. D. Paravagno, *Gazz. chim. ital.*, **44**, I, 538 (1914).

[7] Th. Curtius, *J. prakt. Chem.*, [2] **92**, 80 (1915).

[8] All melting points are uncorrected.

and recrystallized from water to yield 7.9 g. (68.1%) of product; m.p. 222.5°, lit. value 212°.[7]

Anal. Calcd. for $C_8H_{12}O_6N_2$: C, 41.38; N, 12.28; H, 5.18; neut. equiv., 116. Found: C, 41.55; N, 12.52; H, 5.38; neut. equiv., 119.

Succindihydrazide (III).—Succinic anhydride, 5.0 g. (0.05 mole) was dissolved in 100 ml. of refluxing ethanol in a 250-ml., two-necked flask equipped with a Hershberg stirrer and a reflux condenser. A solution of 7.1 g. (0.12 mole) of 85% hydrazine hydrate in 25 ml. of ethanol was slowly added through the condenser. The initial reaction was very vigorous, subsiding when approximately 25% of the hydrazine had been added, at which point acid I precipitated. The remaining hydrazine was added more rapidly and the resulting two-phase system stirred and refluxed for 24 hours. The mixture was cooled and the white crystals filtered, washed with ethanol, then ether, and dried at 100° to yield 6.90 g. (94.5%) of succindihydrazide, m.p. 166–167°, lit. value 167°.[8a]

Analysis of the Oil Obtained in the Preparation of Succindihydrazide.—Succinic anhydride 10.0 g. (0.10 mole) was dissolved in 200 ml. of ethanol at 70° and a solution of 11.8 g. (0.20 mole) of 85% hydrazine hydrate in 20 ml. of ethanol was slowly added over a period of 10 minutes. The ethanol was decanted from the oil, and the latter placed in a vacuum desiccator for 30 minutes to remove any residual ethanol. A sample of the oil weighing 3.9 g. was removed and dissolved in 20 ml. of water. Nine grams of benzaldehyde was added and the mixture heated near the boiling point for 15 minutes. The two-phase mixture was poured into a wet filter paper, thereby separating the oil and the aqueous phases. The aqueous filtrate was cooled and filtered, yielding 1.05 g. of disuccinhydrazide diacid (I), m.p. 218–220°. The mother liquid was evaporated to a slurry and filtered to yield an additional 0.95 g. of I. The total yield was 2.0 g. (8.62 millimoles). This compound represents 51.3% of the oil by weight.

The oily material remaining on the filter paper was triturated with 150 ml. of ether; 50 ml. of hexane was added, and the mixture filtered to yield 1.0 g. (4.55 millimoles) of the monobenzalhydrazide of succinic acid, m.p. 163–164° (lit. value 164°). This represents 4.55 millimoles of 0.60 g. of succinhydrazide monoacid. Thus the latter compound represents 15.4% of the oil by weight.

The ethereal mother liquid was placed on a steam plate and a jet of air played on the surface of the liquid until all of the ether and benzaldehyde had evaporated. The remaining dry benzalazine (m.p. 86–88°) weighed 3.55 g. (17.1 millimoles). This corresponds to 0.86 g. of hydrazine hydrate. Therefore, hydrazine hydrate represents 21.9% of the oil by weight.

Hydrazonium Salt of Succinhydrazide Monoacid (II).—Succinic anhydride, 2.5 g. (0.025 mole), dissolved in 20 ml. of ethanol was added slowly to a solution of 2.5 g. (0.05 mole) of hydrazine hydrate in 20 ml. of ethanol. The mixture was allowed to stand for 30 minutes and the solvent was removed *in vacuo*. The remaining oil was then placed under a vacuum of 0.1 mm. for several days until it solidified. The solid was then recrystallized from methanol yielding 25% of succindihydrazide and 75% of the hydrazonium salt of succinhydrazide monoacid; m.p. 105°, lit. value[7] 104°.

Polysuccinhydrazide (IV) (a) From Succinic Anhydride.—A sample of 5.0 g. (0.05 mole) of succinic anhydride was dissolved in 100 ml. of toluene. The refluxing solution was stirred, and 2.95 g. (0.05 mole) of 85% hydrazine hydrate was slowly added. The mixture was then refluxed for 15 hours, the white solid filtered, and washed with ethanol, then with copious amounts of hot water to remove soluble impurities.[10] The dried powder weighed 4.96 g. (87%) and softened over the range 255–262°, lit. value 255–258°.[7]

Anal. Calcd. for $C_4H_6O_2N_2$: C, 42.1; H, 5.26; N, 24.6. Found: C, 41.1; H, 5.15; N, 23.5.

(b) **From Disuccinhydrazide Diacid** (I).—A mixture of 2.32 g. (0.01 mole) of I and 0.59 g. (0.01 mole) of hydrazine hydrate was heated at 115° for 10 hours. The powder which was obtained after filtration and washing with ethanol and hot water melted at 258–265°. A mixed m.p. with a sample of the polymer prepared by method (a) was not depressed.

Reaction of Polysuccinhydrazide with Benzenesulfonyl Chloride.—One gram (8.77 millimoles) of polysuccinhydrazide was added to 15 ml. of benzenesulfonyl chloride and the mixture heated to 180°, at which temperature gas evolution occurred. The mixture was held at this temperature until the gas evolution ceased, the gaseous products being collected over benzene. A small amount of hydrogen sulfide was evolved; the major part of the gas, however, was composed of 6.4 millimoles of nitrogen and 13.0 millimoles of hydrogen chloride.

Cooling the black reaction mixture yielded 0.25 g. (1.3 millimoles) of perhydro-1,4,6,9-tetraketopyridazo[1,2-a]-pyridazine (VI) (29%), m.p. 308–309°. Upon prolonged standing (three days), the mother liquid deposited 0.61 g. (2.4 millimoles) of cyclic N-benzenesulfonylsuccinhydrazide (V) (27%), m.p. 188–190°. For analyses of these compounds see below.

Carrying out the reaction with equimolar quantities of the reactants in dichlorobenzene yielded small amounts of N-benzenesulfonylsuccinhydrazide only, the major portion of the polymer being recovered unreacted.

Perhydro-1,4,6,9-tetraketopyridazo[1,2-a]pyridazine (VI) (a) With Diethyl Succinate.—One gram (8.76 millimoles) of cyclic succinhydrazide and 1.52 g. (8.76 millimoles) of diethyl succinate were refluxed in 40 ml. of nitrobenzene for 12 hours. The solution was evaporated to a slurry and filtered to yield 1.03 g. (62%) of product, m.p. 299–301°. Recrystallization from water yielded white crystals melting at 308–309°.

Anal. Calcd. for $C_6H_8N_4O_2$: C, 49.0; N, 14.29; H, 4.08; mol. wt., 196. Found: C, 49.1; N, 14.38; H, 3.94; mol. wt., 203 (Rast method).

(b) **With Succinyl Chloride.**—One gram (8.76 millimoles) of cyclic succinhydrazide and 1.35 g. (8.76 millimoles) of succinyl chloride were refluxed in 20 ml. of dioxane for 15 hours. The reaction mixture was cooled, decanted from a small amount of insoluble material, and diluted with an equal volume of ether. Upon further cooling in Dry Ice, VI crystallized out and was purified by recrystallizing it twice from water, m.p. 308–309°. A mixed melting point of a sample prepared by method (a) and from the polymer (IV) was not depressed.

The Reaction of Cyclic Succinhydrazide with Benzenesulfonyl Chloride.—A mixture of 2.0 g. (0.075 mole) of cyclic succinhydrazide and 5 ml. of benzenesulfonyl chloride was heated until solution was complete. Cooling yielded a small amount of VI and prolonged standing caused V to deposit. Recrystallization of the latter from methanol gave 1.33 g. (30%) of product; m.p. 188–190°. A mixed melting point of a sample prepared from the polymer (IV) and benzenesulfonyl chloride was found to be 189°.

Anal. Calcd. for $C_{10}H_{10}O_4N_2S$: C, 47.3; H, 3.94; N, 11.02; mol. wt., 254. Found: C, 47.3; H, 4.09; N, 11.02; mol. wt., 265 (Rast method).

Reduction of Maleic Hydrazide (VII) to Cyclic Succinhydrazide (VIII). (a) **With Adams Catalyst.**[10]—Two-tenths of a gram of platinum oxide was added to a solution of 1.0 g. (8.9 millimoles) of maleic hydrazide[11] in 150 ml. of absolute ethanol, and the mixture shaken under four atmospheres of hydrogen for 22 hours at 100°. The catalyst was removed by filtration and the clear solution evaporated to a slurry. Filtration yielded 0.72 g. (71%) of cyclic succinhydrazide, m.p. 267–271°. Recrystallization from ethanol raised the melting point to 277–278°. This compound exhibits the peculiarity of melting sharply at 214°, then resolidifying, and remelting at 277–278°.

Anal. Calcd. for $C_4H_6O_2N_2$: C, 42.1; H, 5.26; N, 24.6; neut. equiv., 114. Found: C, 42.2; H, 5.15; N, 24.4; neut. equiv., 110.

(b) **With Aluminum Amalgam.**—A suspension of 2 g. (0.0178 mole) of maleic hydrazide in 50 ml. of ethanol and 15 ml. of water was added to 5 g. of aluminum amalgam and allowed to stand at 25° for 1 hour with occasional shaking. The mixture was filtered and washed with 15 ml. of ethanol.

(8a) Th. Curtius, G. Schofer and N. Schwan, *J. prakt. Chem.*, **51**, 190 (1895).

(9) Due to the high insolubility of polysuccinhydrazide, this was the only purification step employed.

(10) Excess of catalyst is necessary because it is apparently partially poisoned by the hydrazide.

(11) Th. Curtius and H. A. Foesterling, *J. prakt. Chem.*, **51**, 371 (1895).

Evaporation of the combined filtrates yielded 1.2 g. (59.1%) of cyclic succinhydrazide, m.p. 267–271°. Recrystallization from ethanol raised the melting point to 277°.

Cyclic Succinhydrazide Diacetate (IX).—A mixture of 0.5 g. (0.0044 mole) of cyclic succinhydrazide and 10 ml. of acetic anhydride was refluxed for 2 hours and the excess acetic anhydride evaporated. The resulting oil was dissolved in about 15 ml. of a 1–4 ethanol–ether mixture and cooled. The diacetate crystallized and was purified from an ethanol–ether mixture to yield 0.41 g. of material, m.p. 130–132°.

Anal. Calcd. for $C_8H_{10}O_4N_2$: C, 48.48; H, 5.05; N, 14.14. Found: C, 48.50; H, 4.92; N, 14.12.

Reduction of Cyclic Succinhydrazide (VIII) to Succinamide (X).—One gram of cyclic succinhydrazide was dissolved in 400 ml. of absolute ethanol and 0.1 g. of Raney nickel catalyst was added. The mixture was then shaken under 70 atmospheres of hydrogen at 140° for 24 hours. The catalyst was removed by filtration, and the clear solution evaporated to a slurry. Filtration yielded 0.6 g. (60%) of succinamide, m.p. 260° dec., lit. value[12] 260° dec. A mixed melting point with an authentic specimen of succinamide was also found to be 260° dec.

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[12] G. F. Morrel, *J. Chem. Soc.*, **105**, 2705 (1914).

LAFAYETTE, INDIANA RECEIVED JANUARY 25, 1951

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, COLLEGE OF ARTS AND SCIENCES, UNIVERSITY OF LOUISVILLE]

Decarboxylation of α-(Benzenesulfonamido)-carboxylic Acids

BY RICHARD H. WILEY AND N. R. SMITH[1]

A continued study of the base-catalyzed decarboxylation of carboxylic acids has shown that α-(benzenesulfonamido)-propionic acid decarboxylates on refluxing in a pyridine–acetic anhydride mixture to give 58% of the theoretical amount of carbon dioxide, 42% of diphenyl disulfide, and traces of 1,4-dibenzenesulfonyl-3,6-dimethylpiperazinedione-2,5. A possible mechanism for the reaction is discussed.

Recent studies of the decarboxylation reaction have resulted in several discussions of the mechanism of the reactions involved under the influence of acid[2a,b] and of basic catalysts[3–6] and on pyrolysis.[6–9] We wish to report in this paper the unusual formation of diphenyl disulfide in the base-catalysed decarboxylation of α-(benzenesulfonamido)-carboxylic acid and to comment on the possible mechanism of the reaction in terms of a *transitory* carbanion as an intermediate in a β-elimination type of reaction.[10]

When α-(benzenesulfonamido)-propionic acid was refluxed with excess pyridine and acetic anhydride a vigorous evolution of a gas took place. This gas consists only of carbon dioxide and was obtained in 58% of the theoretical amount. Two products have been isolated from the reaction mixture. Diphenyl disulfide was obtained in 42% yield (crude) and traces of a compound thought to be 1,4-dibenzenesulfonyl-3,6-dimethylpiperazinedione-2,5 have been isolated. Although the latter has not been previously described, compounds of this type are known to be formed by the action of acetic anhydride on *p*-toluenesulfonyl derivatives of amino acids.[11] In other experiments it has been shown that benzenesulfonamide, substituted for the acid, gave no diphenyl disulfide on refluxing with excess acetic anhydride and pyridine. Also, on refluxing α-(benzenesulfonamido)-propionic acid with pyridine alone no carbon dioxide was formed. On refluxing with acetic anhydride alone carbon dioxide and a tar, from which none of the disulfide was isolated, were formed.

This reaction may be formulated as a base-catalyzed, β-elimination corresponding to the dehydrohalogenation of β-halo acids. Initial attack by the base at the carbon adjacent to the nitrogen would provide a *transitory* carbanion in a reaction which, as in other β-elimination reactions,[10] is probably truly simultaneous and need not involve independent existence of the carbanion.

$$C_6H_5SO_2NHCH(CH_3)CO_2H \xrightarrow[-H^+]{\text{Base}}$$

$$[C_6H_5SO_2NH\overset{\curvearrowleft}{-}\ddot{C}H(CH_3)_2CO_2H]^- \xrightarrow{-[C_6H_5SO_2]^-}$$

$$[NH{=}C(CH_3)CO_2H] \xrightarrow{H^+} [CH_3CH{=}NH] + CO_2$$

Once formed, the sulfinite ion can undergo disproportionation, a recognized reaction,[12] but this reaction in which the yield is limited to 33¹/₃% is

$$3C_6H_5SO_2H \longrightarrow 2C_6H_5SO_3H + C_6H_5SH$$

insufficient to account for the 42% yield of disulfide

$$C_6H_5SO_2H \longrightarrow 2C_6H_5SO_3H + C_6H_5SH$$

obtained. The sulfinite may be reduced further by the aldimine. Under mild conditions anils[13] and

$$C_6H_5SO_2H + 2CH_3CH{=}NH \longrightarrow C_6H_5SH + 2CH_3CONH_2$$

aldehydes[14] are known to form addition compounds which dissociate at higher temperatures with oxidation–reduction and unclarified decomposition reactions, but there appears to be no exact analogy in the literature for this reaction. The fact that not over 58% of the theoretical amount of carbon diox-

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[1] The authors wish to acknowledge with thanks the grant received from the Research Committee of the College of Arts and Sciences of the University of Louisville in support of this work.

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[10] Alexander, "Ionic Organic Reactions," John Wiley and Sons, Inc., New York, N. Y., 1950, p. 106.

[11] Wallin, *Acta Universitates Lundensis*, 2 Abt. Abhandl. II, **28**, 12 (1892); Beilstein, "Handbuch der organischen Chemie," Vol. 24, p. **269**.

[12] Connor, in Gilman "Organic Chemistry," Vol. I, John Wiley and Sons, Inc., New York, N. Y., 1948, p. 906.

[13] Knoevenagel and Römer, *Ber.*, **56**, 215 (1923).

[14] Bazlen, *ibid.*, **60**, 1470 (1927).