[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, DUKE UNIVERSITY]

Conjugate Additions of the Disodio Salts of 2-Phenylacetamide and Its *N*-Phenyl and *N*-Methyl Derivatives to Ethyl Cinnamate in Liquid Ammonia[1]

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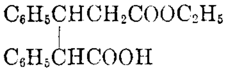
The disodio salts of 2-phenylacetanilide and *N*-methyl-2-phenylacetamide, prepared by means of two molecular equivalents of sodium amide in liquid ammonia, underwent conjugate addition to ethyl cinnamate to form the corresponding amide esters. Two diastereoisomers of the former product and the *erythro* isomer of the latter product were isolated, and their structures were established by independent syntheses. The disodio salt of 2-phenylacetamide underwent conjugate addition to ethyl cinnamate to produce exclusively the *threo*-2,3-diphenylglutarimide. Two possible courses of reaction are suggested for the formation of this cyclic imide, the yield of which was increased by the use of an extra equivalent of sodium amide.

Recently[2] disodio phenylacetate (I) was shown to undergo conjugate addition to ethyl cinnamate to give a mixture of the diastereoisomers of ester-acid II.

$$C_6H_5\overset{Na}{C}HCOONa$$

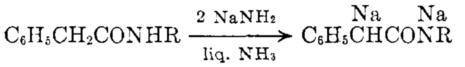
I



$$\begin{array}{l} C_6H_5CHCH_2COOC_2H_5 \\ \quad | \\ C_6H_5CHCOOH \end{array}$$

II

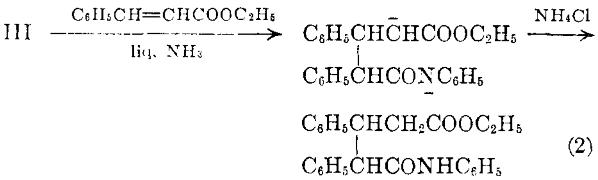
In the present investigation a similar study was made with the disodio salts of 2-phenylacetanilide, *N*-methyl-2-phenylacetamide, and 2-phenylacetamide, which were prepared by means of two molecular equivalents of sodium amide in liquid ammonia (Equation 1).



$$C_6H_5CH_2CONHR \xrightarrow[\text{liq. } NH_3]{2\ NaNH_2} C_6H_5\overset{Na}{C}HCO\overset{Na}{N}R \qquad (1)$$

III. R = C_6H_5
IV. R = CH_3
V. R = H

Analogous to disodio phenylacetate (I), disodio salt III underwent conjugate addition to ethyl cinnamate to give a mixture of the two diastereoisomers of the amide-ester VI (Equation 2).



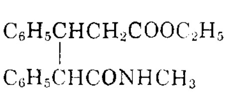
$$\text{III} \xrightarrow[\text{liq. } NH_3]{C_6H_5CH=CHCOOC_2H_5} \begin{array}{l} C_6H_5CH\overset{-}{C}HCOOC_2H_5 \\ \quad | \\ C_6H_5CHCO\underset{-}{N}C_6H_5 \end{array} \xrightarrow{NH_4Cl} \begin{array}{l} C_6H_5CHCH_2COOC_2H_5 \\ \quad | \\ C_6H_5CHCONHC_6H_5 \end{array} \qquad (2)$$

VI

The *erythro* and *threo* isomers of VI were isolated in yields of 48 and 4%, respectively. Much more of the latter isomer was evidently obtained in the impure state. Interestingly the two isomers had essentially the same melting point; however, a mixture of the two isomers melted much lower and over a range. The infrared spectra of the two isomers were not identical.

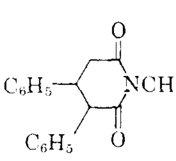
The more readily isolated isomer was shown to have the *erythro* configuration and the less easily isolated isomer the *threo* configuration by independent syntheses from the corresponding diastereoisomers of ester-acid II through the intermediate acid chlorides.

Similarly, disodio salt IV underwent conjugate addition to ethyl cinnamate to form, apparently, a mixture of the two diastereoisomers of amide-ester VII, although only the *erythro* isomer was isolated (24%). The configuration of this isomer was established by independent synthesis from the *erythro* isomer of ester-acid II. Incidently the corresponding *threo* amide-ester, prepared from the *threo* ester-acid II, melted about 50° higher than the *erythro* amide-ester. The crude conjugate addition product was indicated by infrared absorption not to contain the possible cyclic imide VIII.



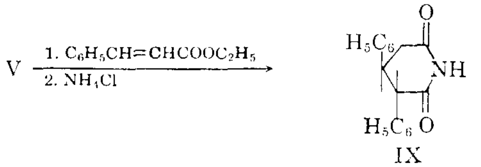
$$\begin{array}{l} C_6H_5CHCH_2COOC_2H_5 \\ \quad | \\ C_6H_5CHCONHCH_3 \end{array}$$

VII



VIII

In contrast to disodio salts III and IV, as well as I, disodio salt V underwent conjugate addition to ethyl cinnamate under similar conditions to form exclusively (53%) *threo*-2,3-diphenylglutarimide (IX) (Equation 3), and some 2-phenylacetamide was recovered. When an extra equivalent of sodium amide was employed the yield of the cyclic imide was increased to 73%.



(3)

IX

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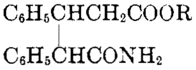
[1] Supported by the National Science Foundation.

[2] R. B. Meyer and C. R. Hauser, *J. Org. Chem.*, **26**, 3183 (1961).

Attempts to isolate amide-ester X, which would correspond to amide-esters VI and VII and to ester-acid II, were unsuccessful. Thus acidification of the reaction mixture after only one and one-half minutes produced the cyclic imide IX, and none of X was detected. The corresponding *threo* amide-acid XI was obtained in an earlier experiment with disodio salt V and ethyl cinnamate[3] but, at that time, it was assumed to have arisen from the hydrolysis of amide-ester X. It is now shown to have arisen from imide IX.

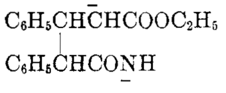


$$\begin{array}{l} C_6H_5CHCH_2COOR \\ \quad\;\;| \\ C_6H_5CHCONH_2 \end{array}$$

X. $R = C_2H_5$
XI. $R = H$

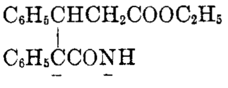
That the product was the cyclic imide IX and not X was shown by analysis and by methylation to form the *N*-methyl derivative VIII. Whereas the product from the conjugate addition exhibited infrared absorption at 3.12 μ for the N—H group as in IX, its *N*-methyl derivative VIII did not. Had the *N*-methyl derivative of X been obtained, it should still have shown infrared absorption for an N—H group. Moreover the melting points of the conjugate addition product and of its hydrolysis product agreed satisfactorily with those reported[4] for the cyclic imide IX and amide-acid X, respectively. These compounds were prepared by other methods and their configurations established.[4,5]

The exclusive formation of *threo* cyclic imide IX suggests that the conjugate addition of disodio salt V is stereospecific to give *threo* dianion X″, which cyclizes rapidly. However, as the analogous conjugate additions of disodio salts I, III, and IV are nonstereospecific it seems more likely that the *threo* and *erythro* isomers of dianion X″ are formed and the latter isomer then undergoes isomerization. The cyclization, as well as the isomerization, might be accounted for by the conversion of X″ to X″a, as the resulting neutral ester group could then be attacked by the amide ion end of the molecule to produce the anion of IX and ethoxide ion.



$$\begin{array}{l} C_6H_5CH\overset{-}{C}HCOOC_2H_5 \\ \quad\;\;| \\ C_6H_5CHCO\underset{-}{N}H \end{array}$$

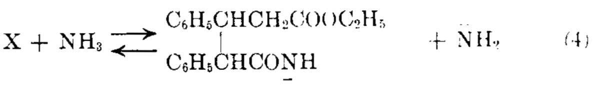
X″



$$\begin{array}{l} C_6H_5CHCH_2COOC_2H_5 \\ \quad\;\;| \\ C_6H_5\underset{-}{C}CO\underset{-}{N}H \end{array}$$

X″a

Another possible mechanism for the conversion of *threo* and *erythro* dianion X″ to the *threo* anion of IX would involve the intermediate formation of monoanion X′, a low concentration of which should be present in equilibrium (Equation 4). The *threo* monoanion X′ should readily cyclize to form the



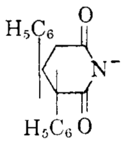
$$X + NH_3 \rightleftarrows \begin{array}{l} C_6H_5CHCH_2COOC_2H_5 \\ \quad\;\;| \\ C_6H_5CHCO\underset{-}{N}H \end{array} + \overset{-}{N}H_2 \qquad (4)$$

X′

anion of *threo* IX. Although the *erythro* isomer of the cyclic imide appears to be somewhat strained because of interaction between the *cis*-phenyl groups, it might be formed through cyclization of the *erythro* monoanion X′ and then be isomerized immediately. Actually monoanion X′, prepared from molecular equivalents of *erythro* amide-ester X and sodium amide in liquid ammonia, was found to be converted readily to the *threo* cyclic imide IX. The *erythro* amide-ester used in this experiment was obtained from the *erythro* ester-acid II through its acid chloride.

The *erythro* amide-ester X was also added to two molecular equivalents of sodium triphenylmethide in liquid ammonia. As the characteristic red color of this reagent was discharged immediately, *erythro* dianion X″ or dianion X″a was presumably an intermediate. There is a possibility, however, that the secondary ionization of *erythro* monoanion X was slower than the cyclization; if so, the resulting imide would then have neutralized the second equivalent of the reagent.

It is of interest that dianion X″ undergoes further reaction resulting in the formation of cyclic imide IX, whereas the corresponding dianions from the conjugate additions of disodio salts I, III, and IV do not. The main free energy decrease in the further reaction of dianion X″ is presumably the formation of the more weakly basic anion IX′. Apparently this result cannot be ascribed merely to the nucleophilicity of the amide end of the dianion X″, as the presumably more nucleophilic *N*-methyl amide end of the corresponding dianion from disodio salt IV does not cyclize under similar conditions.



IX′

EXPERIMENTAL[6]

Conjugate addition of 2-phenylacetanilide to ethyl cinnamate. To a stirred suspension of 0.050 mole of sodium amide in 300 ml. of commercial anhydrous liquid ammonia[7] was added 5.3 g. (0.025 mole) of solid 2-phenylacetanilide, m.p. 117–118° (lit.,[8] m.p. 117–118°), prepared from phenylacetyl chloride and aniline. After stirring for 30 min., the green solution was assumed to contain 0.025 mole of disodio salt III. To this

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(3) C. R. Hauser and M. T. Tetenbaum, *J. Org. Chem.*, **23,** 1146 (1958).

(4) S. Avery and W. D. Maclay, *J. Am. Chem. Soc.*, **51,** 2833 (1929).

(5) D. Lednicer and C. R. Hauser, *J. Am. Chem. Soc.*, **80,** 6364 (1958).

(6) Melting points were taken on a Fisher-Johns melting point apparatus and are uncorrected. Microanalyses were by Spang Microanalytical Laboratory, Ann Arbor, Mich. Infrared spectra were taken on a Perkin-Elmer Infracord by the potassium bromide method.

(7) See C. R. Hauser, F. W. Swamer, and J. T. Adams, *Org. Reactions*, **8,** 122 (1954).

(8) T. B. Johnson, *J. Am. Chem. Soc.*, **28,** 1457 (1906).

solution there was added during 3.5 min. a solution of 4.4 g. (0.025 mole) of ethyl cinnamate in 15 ml. of anhydrous ether. After stirring for 30 min., solid ammonium chloride (3 g.) was added. The ammonia was evaporated and an equal volume of ether was added. The resulting suspension was poured into 50 ml. of 3*N* hydrochloric acid, and sufficient benzene was added to dissolve the product. The layers were separated, and the organic solution was combined with two ethereal washings of the aqueous solution. After drying over magnesium sulfate and filtering, the solvent was distilled under reduced pressure. The residual solid was crystallized from 95% ethanol to give three crops: (A) 7.0 g., m.p. 155–180°; (B) 1.15 g., m.p. 160–180°; (C) 1.10 g.

Crystallization of (A) from 50 ml. of benzene afforded 4.65 g. (48%) of *erythro*-4-carbethoxy-*N*,2,3-triphenylbutyramide (VI), m.p. 181.5–182.5°. Two recrystallizations from benzene raised the m.p. to 182.5–183°. No depression in melting point was observed on admixture with an authentic sample of the *erythro* isomer prepared as described below.

Anal. Calcd. for $C_{25}H_{25}NO_3$: C, 77.49; H, 6.50; N, 3.62. Found: C, 77.56; H, 6.57; N, 3.63.

Two crystallizations of (B) from benzene-hexane afforded 0.4 g. (4%) of *threo*-4-carbethoxy-*N*,2,3-triphenylbutyramide (VI), m.p. 182–182.5°. No depression in melting point was observed on admixture with an authentic sample of the *threo* isomer prepared as described below. On admixture with the *erythro* isomer the melting point was depressed to 155–172°.

Although further purification of the remaining material was not realized, its infrared spectrum indicated it contained both isomers.

The infrared spectrum of the *erythro* isomer showed N-H absorption[9] at 3.05 μ and carbonyl absorption[9,10] at 5.75 and 6.03 μ whereas the *threo* isomer showed N-H absorption at 2.95 μ and carbonyl absorption at 5.80 and 5.96 μ.

Independent syntheses of erythro and threo VI. A mixture of 0. 94 g. (3.0 mole) of *erythro*-4-carbethoxy-2,3-diphenylbutyric acid (II)[2] and 0.40 g. of thionyl chloride in 10 ml. of benzene was heated on a steam bath for 15 min. The mixture was evaporated to dryness and redissolved in benzene. Aniline (0.60 g.) was added, and the mixture was heated to boiling. After cooling, ether was added and the suspension was washed successively with 5 ml. of water, two 5 ml. portions of 10% sodium bicarbonate solution, and two 5-ml. portions of 3*N* hydrochloric acid. On evaporation of the ether a white solid remained which was crystallized from ethanol to give *erythro* VI, m.p. 182–183°.

The *threo* isomer was prepared similarly from *threo*-4-carbethoxy-2,3-diphenylbutyric acid (II).[2] Crystallization of the crude anilide from benzene-hexane gave *threo* VI, m.p. 175–178°. Two recrystallizations from benzene-hexane raised the melting point to 181°.

Anal. Calcd. for $C_{25}H_{25}NO_3$: C, 77.49; H, 6.50; N, 3.63. Found: C, 77.54; H, 6.49; N, 3.75.

Conjugate addition of N-methyl-2-phenylacetamide to ethyl cinnamate. To a stirred suspension of 0.10 mole of sodium amide in 300 ml. of liquid ammonia[7] was added 7.5 g. (0.050 mole) of *N*-methyl-2-phenylacetamide, m.p. 54–56° (lit.,[11] m.p. 58°) prepared from phenylacetyl chloride and methylamine. After stirring for 30 min., the green solution was assumed to contain 0.050 mole of disodio salt IV. To this solution there was added during 5 min. a solution of 8.8 g. (0.050 mole) of ethyl cinnamate in 25 ml. of anhydrous ether. A yellow precipitate formed which slowly changed to a gray gum. After 15 min., solid ammonium chloride (6 g.) was added. The ammonia was evaporated and an equal volume of ether was added. The resulting suspension was poured into 50 ml. of 3*N* hydrochloric acid, and the layers were separated. After being combined with three ethereal washings of the aqueous solution, the ethereal solution was dried over Drierite, filtered, and the solvent was distilled under reduced pressure. The oily residue crystallized very slowly from benzene-hexane to give 3.9 g. (24%) of *erythro*-*N*-methyl-4-carbethoxy-2,3-diphenylbutyramide (VII), m.p. 110–112°. Repeated crystallization from benzene-hexane gave crystals m.p. 110–110.5°, which was not depressed on admixture with an authentic sample prepared as described below.

Attempts to isolate additional pure material from the filtrates were unsuccessful.

Independent syntheses of erythro and threo VII. A mixture of 0.94 g. (3.0 moles) of *erythro*-4-carbethoxy-2,3-diphenylbutyric acid (II)[2] and 0.40 g. of thionyl chloride in 10 ml. of benzene was heated on a steam bath for 15 min. The mixture was cooled and saturated with methylamine. After being warmed to boiling, the suspension was cooled and diluted with ether. The ethereal suspension was washed successively with 5 ml. of water, two 5-ml. portions of 5% sodium bicarbonate solution, and two 5-ml. portions of 3*N* hydrochloric acid. After drying over Drierite, the ethereal solution was filtered and the solvent was distilled under reduced pressure. Crystallization of the residue from benzene-hexane gave *erythro* VII, m.p. 104–106°. Two recrystallizations from benzene-hexane raised the melting point to 107.5–108°.

Anal. Calcd, for $C_{20}H_{23}NO_3$: C, 73.82; H, 7.12; N, 4.30. Found: C, 73.81; H, 7.16; N, 4.01, 4.04, 3.96.

The *threo* isomer was prepared similarly from *threo*-4-carbethoxy-2,3-diphenylbutyric acid (II).[2] Several crystallizations of the crude material from benzene-hexane gave *threo* VI, m.p. 155–155.5°.

Anal. Calcd. for $C_{20}H_{23}NO_3$: C, 73.82; H, 7.12; N, 4.30. Found: C, 73.78; H, 7.13; N, 4.34.

The infrared spectrum of the *erythro* isomer showed N—H absorption[9] at 2.92 μ and carbonyl absorption[9,10] at 5.78 and 6.05 μ whereas the *threo* isomer showed N—H absorption at 2.97 μ and carbonyl absorption at 5.78 and 6.08 μ.

Conjugate addition of 2-phenylacetamide to ethyl cinnamate. (A). *Neutralization with ammonium chloride.* To a stirred suspension of 0.050 mole of sodium amide in 300 ml. of liquid ammonia[7] was added 3.4 g. (0.025 mole) of solid 2-phenylacetamide. After stirring for 15 min., the green solution was assumed to contain 0.025 mole of disodio salt V. To this solution there was added during 5 min. a solution of 4.4 g. (0.025 mole) of ethyl cinnamate in 10 ml. of anhydrous ether. A white precipitate formed immediately, and then rapidly dissolved when most of the ester solution had been added. After stirring for 30 min., solid ammonium chloride (3.0 g.) was added. The ammonia was evaporated and an equal volume of ether was added. The resulting ethereal suspension was poured into 100 ml. of 3*N* hydrochloric acid, and ether and ethyl acetate were added to dissolve the solid. The layers were separated and the organic solution was combined with two ethereal washings of the aqueous solution. The organic solution was washed twice with 100-ml. portions of 10% sodium bicarbonate solution and dried over Drierite. After filtering, the solvent was distilled under reduced pressure. Crystallization of the residue from benzene afforded 3.6 g. (53%) of 2,3-diphenylglutarimide (IX), m.p. 231–232° (lit,[4] m.p. 225–229°). Repeated crystallization from ethanol raised the melting point to 232–233°.

Anal. Calcd. for $C_{17}H_{15}NO_2$: C, 76.96; H, 5.70; N, 5.28. Found: C, 76.90; H, 5.70; N, 5.22.

The infrared spectrum of this imide showed N—H absorption[12] at 3.12 μ and carbonyl absorption[12] at 5.83 and 5.90 μ.

Concentration of the filtrates afforded 2-phenylacetamide (9%) m.p. 158–159°, which was not depressed on admixture with an authentic sample.

Similar results were observed when the ethereal solution of the ester was added during 1.5 min. and the mixture immediately neutralized by pouring it into a solution of ammonium chloride in liquid ammonia.

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(9) See L. J. Bellamy, *The Infrared Spectra of Complex Molecules*, 2nd ed., Wiley, New York, 1958, p. 203.

(10) See ref. 9, p. 178.

(11) H. J. Taverne, *Rec. trav. chim.*, **16**, 34 (1897).

(12) See ref. 9, p. 221.

When the condensation was carried out using 0.075 mole of sodium amide, 0.025 mole of 2-phenylacetamide, and 0.025 mole of ethyl cinnamate (reaction time 30 min.), imide IX was obtained in 73% yield.

When the disodio salt V was added to the ethyl cinnamate in liquid ammonia (inverse addition) and the mixture neutralized after 30 min. by the addition of ammonium chloride, imide IX was obtained in 46% yield and 26% of the 2-phenylacetamide was recovered.

(B) *Neutralization of ethereal suspension with water.* The condensation was performed as described above using 0.050 mole of disodio salt V and 0.050 mole of ethyl cinnamate. After the ester had been added, the solution was stirred for 30 min. and the ammonia was evaporated as an equal volume of anhydrous ether was added. The resulting suspension was stirred for 30 min., and ice water was carefully added. The layers were separated and the ethereal solution was washed with two portions of 10% sodium hydroxide solution. The combined aqueous solutions were cooled, acidified with 3*N* hydrochloric acid, and filtered. Fractional crystallization from ethanol and extractions with 5% sodium bicarbonate solution afforded the following compounds: 2,3-diphenylglutarimide (IX) (16%), m.p. 227–230°; *threo*-4-carboxy-2,3-diphenylbutyramide (XI) (27%), m.p. 205.5–208° (lit.,[4] m.p. 200–205° and[3] 204–205°); cinnamic acid (2%); 2-phenylacetamide (2%).

The infrared spectrum of *threo* XI showed carbonyl absorption[9,13] at 5.86 and 6.11 μ.

N-Methyl-2,3-diphenylglutarimide (VIII). To a stirred solution of 0.020 mole of potassium amide in 150 ml. of liquid ammonia[14] was added 5.30 g. (0.020 mole) of 2,3-diphenylglutarimide. The resulting gray solution was stirred for 3 min., and a solution of 2.8 g. (0.020 mole) of methyl iodide in 20 ml. of anhydrous ether was rapidly added. After stirring for 2 hr., solid ammonium chloride (1.5 g.) was added. The ammonia was evaporated to dryness and 50 ml. of 3*N* hydrochloric acid was added to the residue. Ether and ethyl acetate were added to dissolve the solid, and the layers were separated. After being combined with two ethereal washings of the aqueous solution, the organic solution was washed with two 50-ml. portions of 5% sodium bicarbonate solution followed by 50 ml. of water. After drying over Drierite and filtering, the solvent was distilled under reduced pressure. The residue was crystallized from ethanol to give imide IX (19%), m.p. and mixed m.p. 228–230°. Concentration of the filtrates gave two crops of the *N*-methylimide VIII which were recrystallized from ethanol to give 1.4 g. (24%) of *N*-methyl-2,3-diphenylglutarimide (VIII), m.p. 140–141.5°. Recrystallization from ethanol raised the melting point to 142.5–143°.

Anal. Calcd. for $C_{18}H_{17}NO_2$: C, 77.39; H, 6.13; N, 5.01. Found C, 77.26; H, 6.06; N, 5.00.

The infrared spectrum of the *N*-methylimide showed carbonyl absorption[12] at 5.80 and 5.96 μ.

erythro-4-Carbethoxy-2,3-diphenylbutyramide (X). A solution of 3.1 g. (0.010 mole) of *erythro*-4-carbethoxy-2,3-diphenylbutyric acid (II)[2] and 1.2 g. of thionyl chloride in 110 ml. of benzene was refluxed for 15 min. The solution was cooled in ice and saturated with ammonia. The resulting suspension was poured into 50 ml. of water, and 500 ml. of ethyl acetate was added. The layers were separated, and the organic solution was washed successively with two 50-ml. portions of 5% sodium bicarbonate solution and two 50-ml. portions of 3 *N* hydrochloric acid. After drying over Drierite and filtering, the solvent was distilled under reduced pressure. The residue was crystallized from dilute ethanol to give *erythro* amide-ester X (1.9 g., 61%), m.p. 208–211.5°. Several recrystallizations from ethanol gave m.p. 208–208.5°.

Anal. Calcd. for $C_{19}H_{21}NO_3$: C, 73.29; H, 6.80. Found: C, 73.27; H, 6.70.

The infrared spectrum of *erythro* X showed carbonyl absorption[9,10] at 5.80 and 6.04 μ.

Acidification of the alkaline washings afforded 0.65 g. (21%) of starting ester-acid, m.p. and mixed m.p. 188–189°.

Cyclization of X. (A) *With sodium amide.* To a stirred suspension of 3.11 g. (0.010 mole) of *erythro* amide-ester X in 100 ml. of liquid ammonia was added a stirred suspension of 0.010 mole of sodium amide in 150 ml. of liquid ammonia.[7] The resulting gray solution was stirred for 30 min., and neutralization by the addition of solid ammonium chloride (0.6 g.). The ammonia was evaporated and an equal volume of ether was added. The ethereal suspension was poured into 50 ml. of 3*N* hydrochloric acid, and ethyl acetate (250 ml.) was added. The layers were separated and the organic solution was combined with two ethyl acetate washings of the aqueous solution. After drying over Drierite and filtering, the solvent was distilled under reduced pressure. Crystallization of the residue from ethanol afforded 1.4 g. (51%) of imide IX, m.p. 228–229°, and 229–230° on admixture with an authentic sample of the imide.

Similar results were obtained when the amide-ester X was added to the sodium amide and the solution neutralized directly with ammonium chloride after 30 min., or inversely with ammonium chloride after 0.5 min. In the former case the imide IX was obtained in 57% yield and in the latter case in 64% yield.

(B) *With sodium triphenylmethide.* To a stirred suspension of 0. 012 mole of sodium amide in 300 ml. of liquid ammonia[7] was added 3.0 g. (0.012 mole) of triphenylmethane. After stirring for 15 min., 2.0 g. (0.0064 mole) of *erythro* amide-ester X was added. The color faded immediately and a light green precipitate formed. After stirring for 30 min., solid ammonium chloride (1 g.) was added. The ammonia was evaporated and an equal volume of ether was added. The ethereal suspension was processed as in (A). There was isolated 1.1 g. (65%) of imide IX, m.p. and mixed m.p. 230–231°.

DURHAM, N. C.

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(13) See ref. 9, p. 161.

(14) See C. R. Hauser and T. M. Harris, *J. Am. Chem. Soc.*, **80**, 6360 (1958).