The anolyte, prepared by combining 40 ml. of denatured 3 A ethanol, 5 ml. of 1*M* tetrabutylammonium chloride, 3 ml. of 1*M* tetrabutylammonium hydroxide, and 2 ml. of water, was added to the porous alundum cup and allowed to stand in air until the cup became damp on the outside. The catholyte, 19 g. of tetrabutylammonium bromide in 200 ml. of denatured 3 A ethanol and 60 ml. of water, was added to the electrolysis cell together with sufficient mercury to cover the bottom to a depth of 1 cm. After deaerating for 30 min. with nitrogen the alundum cup with a carbon rod anode and the calomel cell were inserted and the stirrer started. The controlled potential was set at −2.6 v. (SCE) for all reductions except for a run at −2.1 v. (SCE) with androstane-3β,16α-diol-17-one diacetate in an attempt to reduce selectively the 16α-acetoxy group. The solution was electrolyzed at this potential until the current decreased to a constant value. The sample, from 0.5 to 1.0 g., was then added in 40 ml. of denatured 3 A ethanol and the electrolysis continued until the current returned to that of the blank.

The electrolyzed material was siphoned off, filtered through Celite, acidified with a little acetic acid, and diluted with water. The ethanol was removed and the volume reduced to about 100 ml. The solids were filtered, washed thoroughly with water, and dried under vacuum to constant weight.

Chemical transformations. All acetylations were carried out at room temperature for 20–24 hr., using a 40 to 50-fold excess of a solution containing one part acetic anhydride and two parts of dry pyridine. Cholestane-3β,6α-diol was oxidized to cholestane-3,6-dione with a 50% excess of chromic acid in 95% acetic acid at room temperature for 18 hr. The mixtures of allopregnane-3β-20-diols were treated in the same manner except for a reaction time of 6 hr. Reductions of ketosteroids with sodium in propanol were carried out as described by Antonucci, *et al.*[14]

Pregnane-3α,12β,20β-triol was prepared in this manner from pregnane-3α,20β-diol-12-one in 95% yield after one recrystallization from ethyl acetate, m.p. 227–229°. A second recrystallization did not change the melting point. The infrared spectrum and combustion analysis indicated that the triol was solvated with 0.5 mole of ethyl acetate.

Anal. Calcd. for $C_{21}H_{36}O_3 \cdot {}^1/_2C_4H_8O_2$: C, 72.59; H, 10.59. Found C, 72.69; H, 10.65.

Paper chromatography. Papergrams were run in toluene-propylene glycol and ligroin (b.p. 60–90°)–propylene glycol. Precut sheets of Whatman No. 1 paper were dipped in 50% propylene glycol–methanol and blotted to dryness. The solvent was allowed to descend 45 cm. (3–5 hr.) or the paper was serrated and run overnight (16 hr.). A saturated solution of antimony trichloride in chloroform was used for the detection of Δ^5,3β-alcohols and 10% phosphomolybdic acid in methanol for saturated alcohols. Quantitative measurements were made with a Photovolt densitometer, model 525, and areas were measured with an Ott compensating planimeter.

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[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, DUQUESNE UNIVERSITY]

The Lithium Aluminum Hydride Reduction of Some *N*-Substituted Succinimides[1]

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The reduction of a number of *N*-substituted succinimides with lithium aluminum hydride has been studied. In all but one case reduction to the *N*-substituted pyrrolidines occurs smoothly. *N*-Benzhydryl, *N*-*t*-butyl and *N*-phenylsuccinimides undergo ring opening to yield the respective amino alcohols in addition to the normal reduction product. *N*-Tritylsuccinimide gives only *N*-trityl-4-hydroxybutyramide. Several succininamic acids have been reduced to the respective amino alcohols.

It has been reported in the literature[2-5] that *N*-substituted succinimides and *N*-substituted glutarimides, when treated with lithium aluminum hydride (I) undergo reduction solely to the *N*-substituted pyrrolidines and *N*-substituted piperidines respectively. In the course of some other work, it became necessary to reduce *N*-tritylsuccinimide with I. This compound did not undergo reduction in the manner mentioned above. It was therefore of interest to investigate the reduction of a number of *N*-substituted succinimides.

EXPERIMENTAL[6]

N-Substituted succinimides. All the succinimides, except *N*-tritylsuccinimide, were prepared by heating mixtures of succinic anhydride and the corresponding amine for a period of 24 hr.

Isopropylsuccinimide, m.p. 64–65°, lit. m.p. 60°,[7] was obtained in 84% yield.

Anal. Calcd. for $C_7H_{11}NO_2$: C, 59.55; H, 7.86; N, 9.92. Found: C, 59.72; H, 7.75; N, 9.74.

T-Butylsuccinimide, m.p. 49–50°, was obtained in 25% yield. The major product in this reaction was a solid, m.p. 164–166°. No attempt was made to determine the structure

(1) Presented before the Organic Chemistry Division at the 138th Meeting of the American Chemical Society, New York, N. Y., September 1960.

(2) N. G. Gaylord, Reduction with Complex Metal Hydrides, Interscience Publishers Inc., New York, 1956, p. 627.

(3)(a) V. C. Barry and D. Twomey, *Proc. Roy. Irish. Acad.*, **55B**, (1), 1 (1952); (b) C. V. Barry, M. L. Conalty, and D. Twomey, *Proc. Roy. Irish. Acad.*, **55B**, (4), 137 (1953).

(4) L. M. Rice, E. E. Reid, and C. H. Grogan, *J. Org. Chem.*, **19**, 884 (1954).

(5) G. Baddeley, J. Chadwick, and H. T. Taylor, *J. Chem. Soc.*, 455 (1956).

(6) All microanalyses were performed by Dr. Alfred Bernhardt, Mikroanalytisches Laboratorium in Max-Planck Institut für Kohlenforschung, Mülheim, Germany. Boiling points and melting points reported herein are uncorrected. Infrared spectra were determined with a Perkin-Elmer Infracord Spectrophotometer Model 137.

(7) J. Tafel and M. Stern, *Ber.*, **33**, 2233 (1900).

TABLE I

N-SUBSTITUTED SUCCINAMIC ACIDS (R—NH—CO—CH_2CH_2COOH)

R	Yield, %	M.P.	Calcd.			Found		
			C	H	N	C	H	N
i-C_3H_7	86	105–107	52.82	8.23	8.80	53.01	8.23	8.63
t-C_4H_9	90	134–135	55.47	8.73	8.09	55.28	8.76	8.16
C_6H_5	85	147–148[a]						
$(C_6H_5)_2CH$	92	189–190	72.07	6.05	4.94	72.21	6.05	4.99

[a] Lit. m.p. 148°, A. Ludwig and R. I. Georgescin, *Bull. Chim. Soc. Romane*, **39**, 41–63 (1937–38); *Chem. Abstr.*, **34**, 5067 (1940).

of this compound. Separation of the lower melting succinimide was attained by fractional crystallization from acetone and ligroin. Several recrystallizations from petroleum ether (b.p. 30–60°) gave pure *t*-butylsuccinimide. An increased yield (46%) of the compound could be obtained by refluxing 40 g. (0.23 mole) of *t*-butylsuccinamic acid in 2 l. of toluene for a period of 48 hr. and collecting the water produced during the reaction in a Dean and Stark trap.

Anal. Calcd. for $C_8H_{13}NO_2$; C, 61.91; H, 8.44; N, 9.03. Found: C, 61.94; H, 8.27; N, 9.14.

Phenylsuccinimide, m.p. 153°, lit.[8] m.p. 152°, was obtained in 86% yield.

Benzylsuccinimide, m.p. 103°, lit.[9] m.p. 103°, was obtained in 89%.

Benzhydrylsuccinimide, m.p. 131–132° from ethanol was obtained in 56% yield.

Anal. Calcd. for $C_{17}H_{15}NO_2$; C, 76.96; H, 5.70; N, 5.28. Found: C, 76.99; H, 5.62; N, 5.21.

In addition to the imide there was formed some higher melting material, m.p. 248–250°, which presumably is *N,N'*-dibenzhydrylsuccindimide (13.4%).

Anal. Calcd. for $C_{30}H_{28}N_2O_2$: C, 80.33; H, 6.29; N, 6.25. Found: C, 80.19; H, 6.22; N, 6.28.

The two compounds were separated by extraction of the more soluble succinimide from the solid residue with boiling ethanol.

Tritylsuccinimide was prepared by refluxing an equimolar mixture of trityl bromide and *N*-bromosuccinimide in dry chloroform for 3–4 hr. in a dry atmosphere. The bromine that formed during the reaction was removed by washing the mixture with 10% aqueous sodium hydroxide. Evaporation of the solvent left a semisolid mass from which tritylsuccinimide could be crystallized from a mixture of acetone and ligroin, m.p. 203–204°; yield 67%.

Anal. Calcd. for $C_{23}H_{19}NO_2$; C, 80.90; H, 5.61; N, 4.10. Found: C, 80.76; H, 5.62; N, 4.10.

The infrared spectra of all the substituted succinimides showed carbonyl absorptions at 5.65 μ and 5.85 μ and no N—H stretching bands in the 3 μ region.[10]

N-Substituted succinamic acids. The method of Anschütz[11] was adopted for the preparation of the succinamic acids and compounds prepared are listed in Table I.

Tritylsuccinamic acid was obtained by the hydrolysis of tritylsuccinimide. To 9 g. of potassium hydroxide dissolved in 150 ml. of ethanol was added 3 g. (9 moles) of tritylsuccinimide and the mixture was refluxed for 48 hr. The solution was then evaporated to dryness and extracted with water until all the solid went into solution. Acidification of the basic solution with concd. hydrochloric acid gave product, m.p. 222–223°.

Anal. Calcd. for $C_{23}H_{21}NO_3$: C, 76.83; H, 5.89; N, 3.90; neut. equiv., 359.4. Found: C, 76.65; H, 5.71; N, 4.04; neut. equiv., 358.3.

N-Tritylacetamide, m.p. 214–215°, lit.[12] m.p. 214–218°, was prepared in 89% yield in the same way as tritylsuccinimide by using an equimolar ratio of trityl bromide and *N*-bromoacetamide.

Anal. Calcd. for $C_{21}H_{19}NO$: C, 83.66; H, 6.35; N, 4.65. Found: C, 83.78; H, 6.37; N, 4.85.

N-methyl-N-tritylacetamide. In an alkali resistant flask with reflux condenser and calcium chloride tube, 3 g. (0.01 mole) of tritylacetamide in 100 ml. of dry benzene and 1 g. of sodium hydride were refluxed for 48 hr. After allowing the mixture to cool to room temperature, 2 ml. of methyl iodide were added with stirring and the mixture refluxed for another 6 hr. The mixture was then filtered and the solvent removed under reduced pressure from the filtrate. Several recrystallizations from acetone gave product m.p. 147–148°, yield 2.4 g. (76%). Infrared spectrum showed no N—H stretching band in the 3 μ region.[10]

Anal. Calcd. for $C_{22}H_{21}NO$: C, 83.77; H, 6.71; N, 4.44. Found: C, 83.59; H, 6.76; N, 4.58.

Reductions with lithium aluminum hydride (I). A typical run for the reduction of the *N*-substituted succinimides was as follows: To a slurry of 9 g. of I in about 100 ml. of anhydrous tetrahydrofuran in a 1-l., three-necked flask equipped with condenser, mechanical stirrer with mercury seal, and drying tubes containing calcium chloride, a solution of 0.1 mole of the imide in tetrahydrofuran was added dropwise at such a rate that the solvent in the flask refluxed gently. After the addition of the imide was complete, the solution was heated to reflux for a period of 72 hr. The mixture was cooled and the excess of I decomposed by the addition of ethyl acetate. The mixture was then hydrolyzed by the successive addition of 9 ml. of water, 9 ml. of 15% aqueous sodium hydroxide, and 27 ml. of water. The precipitate that formed was filtered and the tetrahydrofuran evaporated. The residue was extracted with ether, dried over potassium hydroxide pellets, and distilled to remove solvent. The residue, if liquid, was finally distilled under reduced pressure to give products indicated in Table II.

With *N*-tritylsuccinimide the product which was obtained in 67% yield after 3 hr. of reflux was *N*-trityl-γ-hydroxybutyramide, m.p. 173–174°. This did not depress the melting point of compound obtained on reduction of *N*-tritylsuccinamic acid. Refluxing the mixture for the usual period of 72 hr. gave only a viscous material from which no solid could be crystallized.

Anal. Calcd. for $C_{23}H_{23}NO_2$: C, 79.97; H, 6.71; N, 4.06. Found: C, 79.42; H, 6.68; N, 4.34.

N-Substituted pyrrolidines. The method of Schlinck[13] for the preparation of *N*-benzylpyrrolidine was employed in all cases except for *N*-phenylpyrrolidine. Since isopropyl chloride did not apparently react with pyrrolidine even after 24 hr. of reflux, the alkyl iodide was employed instead. The infrared spectra of the compounds obtained on reduction of the succinimides were identical to the ones prepared by the above method.

N-Tritylpyrrolidine, m.p. 126–127°, was obtained in 49% yield.

(8) A. Arcoria, H. Lumbroso, and R. Passerini, *Bull. Soc. Chim. (France)*, 757 (1959).

(9) F. R. Goss, C. K. Ingold, and I. S. Wilson, *J. Chem. Soc.*, 2460 (1926).

(10) L. J. Bellamy, *The Infrared Spectra of Complex Molecules*, Methuen and Co., Ltd., London, 1954, p. 176.

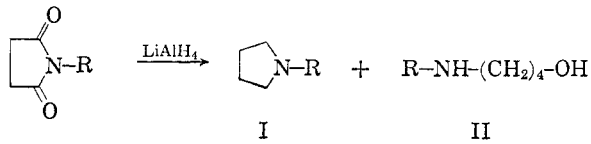
(11) R. Anschütz, *Ber.*, **20**, 3214 (1887).

(12) S. J. Cristol and J. E. Leffer, *J. Am. Chem. Soc.*, **76**, 4468 (1954).

(13) J. Schlinck, *Ber.*, **32**, 952 (1899).

TABLE II

REDUCTION OF *N*-SUBSTITUTED SUCCINIMIDES



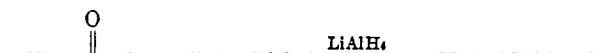
I II

	I			II	
R	Yield, %	M.P. or B.P.	M.P. of Picrate	Yield, %	B.P.
$(CH_3)_2CH$—	66	121[a]	200–201[b]	0	—
$(CH_3)_3C$—	78[c]	146[d]	Decomp. 245[e]	1	M.p. 41–42
C_6H_5—	57	81°/0.45 mm.[f]	115–116[g]	10	134°/0.7 mm.
C_6H_5—CH_2—	65	63°/0.45 mm.[h]	128[i]	0	—
$(C_6H_5)_2CH$—	48	84–85°[j]	194–195[k]	36	201–205°/1 mm.

[a] *Anal.* Calcd. for $C_7H_{15}N$: C, 74.27; H, 13.36; N, 12.37. Found: C, 73.58; H, 13.06; N, 12.35. M.p. of methiodide 290°. *Anal.* Calcd. for $C_8H_{18}IN$: C, 37.66; H, 7.11; I, 49.74; N, 5.49. Found: C, 37.36; H, 7.05; I, 49.54; N, 5.70. [b] *Anal.* Calcd. for $C_{13}H_{18}N_4O_7$: C, 45.61; H, 5.30; N, 16.37. Found: C, 45.61; H, 5.54; N, 16.19. [c] Ether was used as solvent in the reduction. [d] *Anal.* Calcd. for $C_8H_{17}N$: C, 75.52; H, 13.47. Found: C, 75.25; H, 13.21. The compound is very hydroscopic. [e] *Anal.* Calcd. for $C_{14}H_{20}N_4O_7$: C, 47.19; H, 5.66; N, 15.72. Found: C, 47.14; H, 5.75; N, 15.70. [f] Lit. b.p. 110–116°/9 mm. F. K. Signaigo and H. Adkins, *J. Am. Chem. Soc.*, **58**, 715 (1936). [g] Lit. m.p. 116°. Y. K. Yur'ev and G. A. Minkina, *J. Gen. Chem.* (U.S.S.R.), **7**, 2945 (1937). *Chem. Abstr.*, **32**, 5399 (1938). [h] Lit. b.p. 237°. Ref. 13. [i] Lit. m.p. 128°. Ref. 13. [j] *Anal.* Calcd. for $C_{17}H_{19}N$: C, 86.03; H, 8.07; N, 5.90. Found: C, 86.09; H, 8.08; N, 6.03. [k] *Anal.* Calcd. for $C_{23}H_{22}N_4O_7$: C, 59.22; H, 4.75 N, 12.01. Found: C, 58.98; H, 4.77; N, 12.19.

TABLE III

THE REDUCTION OF SUCCINAMIC ACIDS



			Calcd.			Found		
R	B.P.	Yield, %	C	H	N	C	H	N
$(CH_3)_2CH$—	85°/1 mm.	64	64.07	13.06	10.68	64.09	12.86	10.50
$(CH_3)_3C$—[a]	89–91°/1 mm. m.p. 41–42°	61	66.15	13.19	9.64	65.92	13.20	9.49
C_6H_5—	134°/0.7 mm.	50	72.69	9.15	8.48	72.68	9.08	8.28
$(C_6H_5)_2CH$—[b]	201–205°/1 mm.	50–60	79.96	8.29	5.49	80.16	8.55	5.59

[a] In addition to this product there was obtained about 5% *N*-*t*-butylpyrrolidine. Reduction was performed using ether as solvent. [b] In addition to this product there was obtained about 24% *N*-benzhydrylpyrrolidine.

Anal. Calcd. for $C_{23}H_{23}N$; C, 88.14; H, 7.39; N, 4.47. Found: C, 88.22; H, 7.52; N, 4.58.

Picrate, m.p. 133° dec.

Anal. Calcd. for $C_{29}H_{26}N_4O_7$: C, 64.20; H, 4.83; N, 10.33. Found: C, 64.35; H, 4.84; N, 10.42.

N-Substituted-γ-aminobutanols. *N*-Substituted succinamic acids were reduced in a manner similar to that employed in the reduction of the succinimides using the same molar quantities. The products obtained are given in Table III. The infrared spectra of these compounds were identical with those obtained in the reduction of the corresponding succinimides.

N-Trityl-γ-hydroxybutyramide. *N*-Tritylsuccinamic acid (5 g.; 0.014 mole) was reduced with 2 g. of I in the usual manner using tetrahydrofuran as solvent. Hydrolysis of the lithium-aluminum complex with 5% hydrochloric acid followed by evaporation of the solvent gave 4.6 g. of product, m.p. 167–174°. One recrystallization from acetone/ligroin raised the m.p. to 173–174°, yield 95%.

Anal. Calcd. for $C_{23}H_{23}NO_2$: C, 79.97; H, 6.71; N, 4.06. Found: C, 80.02; H, 6.72; N, 4.25.

N-Trityl-γ-methoxybutyramide. To a mixture of 6.2 g. (0.018 mole) of *N*-trityl-γ-hydroxybutyramide dissolved in 100 ml. of dry benzene and 1 g. of sodium refluxed for 36 hr. was added slowly with cooling 4 g. of methyl iodide dropwise. The mixture was allowed to stir for 2 hr. and then filtered, the residue washed with benzene and the combined filtrate and washings evaporated at reduced pressure. Crystallization from ethanol gave 4.2 g. (65%) of *N*-trityl-γ-methoxybutyramide, m.p. 165–165°.

Anal. Calcd. for $C_{24}H_{25}NO_2$: C, 80.19; H, 7.01; N, 3.90. Found: C, 80.01; H, 6.93; N, 4.12.

Attempted reduction of N-tritylacetamide. A solution of 3 g. (0.01 mole) of *N*-tritylacetamide dissolved in 100 ml. of tetrahydrofuran was slowly added to a well stirred mixture of 1 g. of I in 200 ml. of the same solvent. The work up of the reaction mixture after 3 hr. of reflux was as indicated for the succinimides; 2.8 g. of *N*-tritylacetamide was recovered which did not depress the melting point of pure starting material. Refluxing *N*-tritylacetamide with I for 72 hr. did not affect any reduction.

Ethylmethyltritylamine. To a slurry of 2 g. of I in 100 ml. of tetrahydrofuran, 6.5 g. (0.021 mole) of *N*-methyl-*N*-tritylacetamide in 100 ml. of tetrahydrofuran was slowly added in a dry atmosphere and the mixture refluxed for 72 hr. Decomposition of the excess of I followed by hydrolysis gave a highly viscous semisolid mass from which 1.7 g. of amine could be isolated. Recrystallization from ligroin gave solid m.p. 81–82°; yield 22%.

Anal. Calcd. for $C_{22}H_{23}N$: C, 87.66; H, 7.69; N, 4.65. Found: C, 87.62; H, 7.56; N, 4.68.

Picrate, m.p. 105–106° dec.

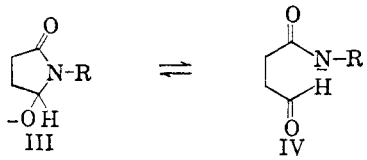
Anal. Calcd. for $C_{28}H_{26}N_4O_7$: C, 63.39; H, 4.94; N, 10.5 Found: C, 63.38; H, 4.88; N, 10.59.

RESULTS AND DISCUSSION

The reduction of *N*-tritylsuccinimide (II) with lithium aluminum hydride (I) gave *N*-trityl-γ-

hydroxybutyramide in 67% yield and none of the expected product, *N*-tritylpyrrolidine. The same compound could also be obtained in good yield by hydrolysis of II in alcoholic potassium hydroxide to *N*-tritylsuccinamic acid and reduction of this acid with I. The mixed melting point of this compound with that obtained in the reduction of II gave no depression. In none of the previously reported[2-5] *N*-substituted succinimide reductions was open chain material isolated. However, there is a report[14] in the literature showing that 2-(2,6-dimethylphenyl)phthalimide gives a 23% yield of *N*-(2,6-dimethylphenyl)-2-hydroxymethylbenzamide in addition to the expected isoindoline derivative. There are also examples[15,16] where acyl cleavage occurs. Mustafa *et al.* report that *N*-benzoylcarbazole is converted to carbazole and benzyl alcohol. Witkop and Patrick observe that *N,N*-diacetyl-*o*-anisidine and *N,N,O*-triacetyl-*o*-aminophenol are converted to *o*-*N*-ethylanisidine and *o*-ethylaminophenol respectively.

In view of the above results it was of interest to reinvestigate some of the reductions to determine whether open chain materials were formed as second products. Results obtained are given in Table I. Indeed, it was found that some of the succinimides did give open chain amino alcohols. Since these amino alcohols could result from impurity of succinamic acids present in the starting material, carefully purified succinimides were used for the reductions. It is of interest to note that *N*-phenylsuccinimide gave a 10% yield of the amino alcohol in addition to N-phenylpyrrolidine. The formation of the open chain amino alcohol can be explained as follows. The initial attack of the hydride ion on the carbonyl function would be expected to give rise to an anion of the types



III IV

III and IV (if there is no change in the position of the atoms these will be resonance structures of each other). Further reduction of III can lead to the pyrrolidine while reduction of IV would give rise to the amino alcohol. Hence the stability of IV should play an important role in the formation of the open chain product. Irrespective of the nature of R in IV there will be resonance interaction between the amide carbonyl and the negative charge on nitrogen. The R group attached to nitrogen can give further stabilization by (a) an inductive effect withdrawing electrons from the nitrogen atom and/or (b) resonance interaction of the electrons with for instance, the phenyl ring. These two factors reinforce themselves in the case where R is phenyl by acting in the same direction, thus causing considerable stability to the intermediate IV. Benzylsuccinimide in which there is little stabilization of the intermediate IV by resonance with the phenyl ring gave no open chain product, whereas benzhydrylsuccinimide and tritylsuccinimide, where the inductive effect becomes more important gave increasing amounts of open chain material. In order to evaluate the relative contribution of the inductive effect *vs.* the resonance effect, it would be interesting to determine the effect of some strongly electron withdrawing substituent on the phenyl ring in *N*-phenylsuccinimide. Unfortunately most of the common electron withdrawing groups are reduced by I, thus giving questionable results.

With II it should be noted that reduction of only one of the carbonyl groups occurs. It was thought that the formation of the anion of the γ-hydroxy group might be inhibiting further reduction. Therefore *N*-tritylacetamide was subjected to reduction. In this case almost quantitative amounts of starting amide could be recovered even after seventy-two hours of reflux in tetrahydrofuran. Since the amide linkage is sterically blocked by the trityl group, reduction may become very difficult because of this. However, reaction of the *N*-methyl-N-tritylacetamide with I at reflux temperature of tetrahydrofuran for seventy-two hours gave reduction to ethylmethyltritylamine in 22% yield with no recovery of starting material indicating that steric hindrance cannot be attributed as a major factor in the inactivity of tritylacetamide or tritylsuccinamic acid toward attack of the hydride ion. It seems therefore that the stability and/or the insolubility of the anion IV play the important role.

For the purpose of identifying the amino alcohols produced in the reduction of the succinimides the corresponding succinamic acids were reduced to the amino alcohols with I. These reactions proceeded smoothly giving rise to the expected amino alcohols in fair to good yields. Results are given in Table II. In at least two cases, namely benzhydrylsuccinamic acid and *t*-butylsuccinamic acid cyclization to the corresponding pyrrolidine derivative occured in 24 and 5% yields, respectively, during the reduction.

PITTSBURGH 19, PA.

(14) A. H. Sommers, *J. Am. Chem. Soc.*, 78, 2441 (1956).
(15) B. Witkop and J. B. Patrick, *J. Am. Chem. Soc.*, 74, 3861 (1952).
(16) A. Mustafa, W. Asker, O. H. Hishmat, A. F. A. Shalaby, and M. Kamel, *J. Am. Chem. Soc.*, 76, 5447 (1954).