As is shown in the condensation between piperonal and β-veratryl-γ-butyrolactone this method is not restricted to a few aldehydes but is obviously a general one for the synthesis of α,β-disubstituted γ-butyrolactones.

We are presently engaged in applying this route in the preparation of other lignans of the α,β-benzylbutyrolactone class.

EXPERIMENTAL

Melting points are uncorrected. Microanalyses by A. Bernhardt, Mikroanalytisches Laboratorium im Max-Planck-Institut, Mülheim/Ruhr, Germany.

The infrared spectra were taken in nujol mulls on a Baird double-beam spectrophotometer.

dl-α-(3,4-Dimethoxybenzylidene)-β-(3,4-dimethoxybenzyl)-butyrolactone. Two grams (8.5 millimoles) of β-(3,4-dimethoxybenzyl)butyrolactone,[3] 1.4 g. (8.5 millimoles) of veratraldehyde, 0.5 g. (9 millimoles) of sodium methoxide, and 16 ml. of benzene were kept in a stoppered bottle for 6 days with occasional shaking. Sulfuric acid, 25 ml. of 2*N*, was added; after some agitating, the layers were separated. The aqueous layer was once more extracted with benzene and the combined organic phase was washed successively with 10% sodium bisulfite and 2*N* sodium carbonate solutions, then with water. After removal of the solvent a brownish oil remained which was taken up in methanol-ether. The compound crystallized after standing in the refrigerator for about 10 days; in later experiments, the process could be accelerated by seeding. The crude first fraction (0.5 g.; m.p. 130–131°) was brownish but could easily be washed colorless with a small amount of methanol. By working up the filtrates, an additional 0.4 g. (m.p. 127–128°) of the compound was obtained; total yield 0.9 g. (28%). The analytical sample melted at 131–131.5°; microcrystalline powder from methanol. The infrared spectrum was as follows: 5.77 μ and 6.08 μ (lactonic C=O, C=C double bond).

Anal. Calcd. for $C_{22}H_{24}O_6$: C, 68.73; H, 6.29. Found: C, 68.57; H, 6.19.

dl-α,β-Bis(3,4-dimethoxybenzyl)butyrolactone (dl-Matairesinol dimethyl ether) (II). A. One gram of α,β-bis(3,4-dimethoxybenzyl)succinic anhydride (m.p. 109–111°; lit., m.p. 110–112°) was reduced with amalgamated aluminum according to Haworth and Woodcock.[6] The work-up procedure was changed as follows: After filtration, the alumina was extracted (Soxhlet, 24 hr.) with chloroform; evaporation of the combined extract and filtrate yielded an oil which was refluxed with 5% methanolic potassium hydroxide solution (10 ml.) for 30 min. The methanol was removed, the residue taken up in water (10 ml.), the aqueous solution washed twice with methylene chloride, acidified with hydrochloric acid, and heated on a water bath for 1 hr. The cooled mixture was extracted with chloroform, the extract washed with sodium bicarbonate solution and water, dried (sodium sulfate), and the solvent removed. The residue was taken up in methanol to give 420 mg. (43%) of the lactone, m.p. 111–113°. One recrystallization from methanol raised the melting point to 112–114° (lit., 113–115°).

B. The preceding unsaturated lactone (2.60 g.) was hydrogenated in methanol with platinum oxide (50 p.s.i.). The crude product (2.52 g.) was an almost colorless oil; its methanolic solution did not deposit any crystals, even after standing in the refrigerator for 1 year. Crystallization could easily be induced, however, by seeding with a sample prepared by procedure A; 1.56 g. (60%) of short white prisms, m.p. 102–107°, were obtained. Two recrystallizations from methanol gave material melting at 112–114°, unchanged when mixed with a sample prepared by procedure A. The infrared spectra (5.67 μ) were identical.

The *dinitro derivative* was prepared according to Haworth and Woodcock[6]: (1) from the reduction product of the anhydride (yellow needles from chloroform-methanol, m.p. 193–194°; lit.: 191–192°), and (2) from the crude hydrogenation product (yellow needles from dioxane-methanol, m.p. 190.5–191.5°). Mixed melting point of the two specimens was 191–193°. The infrared spectra (5.69μ) were identical.

Anal. Calcd. for $C_{22}H_{24}N_2O_{10}$: C, 55.46; H, 5.08; N, 5.88. Found: C, 55.45; H, 5.14; N, 5.89.

dl-α-(p-Dimethylaminobenzylidene)-β-(3,4-dimethoxybenzyl)-butyrolactone was obtained from β-(3,4-dimethoxybenzyl)-butyrolactone and *p*-dimethylaminobenzaldehyde as above, with a yield of 43% as pale-yellow short needles from dioxane-methanol, m.p. 193–193.5°; infrared spectrum: 5.74 β and 6.12 β.

Anal. Calcd. for $C_{22}H_{25}NO_4$: C, 71.91; H, 6.86; N, 3.81. Found: C, 72.00; H, 6.73; N, 3.99.

dl-α-(p-Dimethylaminobenzyl)-β-(3,4-dimethoxybenzyl)-butyrolactone was prepared by hydrogenation of the preceding compound in 60% yield, as colorless blocks from methanol, m.p. 115–115.5°. The infrared spectrum was as follows: 5.64 μ (no peak at ~6.1 β, no C=C-unsaturation).

Anal. Calcd. for $C_{22}H_{27}NO_4$: C, 71.52; H, 7.37; N, 3.79. Found: C, 71.76; H, 7.47; N, 3.84.

dl-α-(3,4-Methylenedioxybenzylidene)-β-(3,4-dimethoxybenzyl)butyrolactone was prepared from β-(3,4-dimethoxybenzyl)butyrolactone and piperonal as above, yield 12% as pale-yellowish short needles from methanol, m.p. 97–97.5°. The infrared spectrum showed the following bands: 5.75 and 6.10 μ.

Anal. Calcd. for $C_{21}H_{20}O_6$: C, 68.47; H, 5.47; Found: C, 68.40; H, 5.44.

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Esters and Ketones Related to Diphenylacetic Acid

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In order to study the pharmacological properties of ketones and basic esters related to diphenylacetic acid, the preparation of a number of such compounds (Table I) was required. These molecules are of interest since they are structurally related to substances possessing antispasmodic, local anesthetic, anti-adrenal, and analgetic activities.

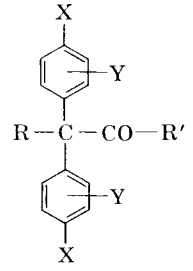
Most of the final compounds prepared in the present study possess nuclear amino groups. 2,2-Bis(*p*-nitrophenyl)propionic acid (I) and 2,2-bis(*p*-nitrophenyl)acetic acid (II)[1,2] were prepared by acid hydrolysis of the respective methyl esters.[3] The reported[1] facile decarboxylation of II

(1) I. M. Hunsberger and E. D. Amstutz, *J. Am. Chem. Soc.*, 71, 2635 (1949).

(2) L. Haskelberg and D. Lavie, *J. Am. Chem. Soc.*, 71, 2580 (1949).

TABLE I

COMPOUNDS RELATED TO DIPHENYLACETIC ACID



No.	R	R'	X	Y	Yield,[a] %	Solvent	Salt	M.P. or B.P. (mm.)	Formula	Analyses: Carbon Calcd.	Carbon Found	Hydrogen Calcd.	Hydrogen Found
1	CH_3	OH	NO_2	H	71	80% HOAc	...	175–177[b]	$C_{15}H_{12}N_2O_6$	56.96	57.07	3.82	3.78
2	CH_3	Cl	NO_2	H	63	C_6H_6-PE	...	125–126	$C_{15}H_{11}ClN_2O_5$	53.82	53.85	3.31	3.33
3[c]	CH_3	Cl	CH_3	NO_2	57	C_6H_{14}	...	89–90	$C_{17}H_{15}ClN_2O_5$	56.28	56.59	4.17	4.18
4	H	$(CH_3)_2NCH_2CH_2O$	NO_2	H	67	C_6H_6-PE	...	117–119[b]	$C_{19}H_{19}N_3O_6$	57.90	58.17	5.13	5.29
5	H	$(C_2H_5)_2NCH_2CH_2O$	NO_2	H	64	$(CH_3)_2CO(C_2H_5)_2O$	HCl	164–166	$C_{20}H_{24}ClN_3O_6$	54.86	54.98	5.52	5.73
6	H	C_2H_5O	NO_2	H	...	C_2H_5OH	...	126–128	$C_{11}H_{14}N_2O_6$	58.18	57.99	4.27	4.45
7	CH_3	$(CH_3)_2NCH_2CH_2O$	NO_2	H	...	CH_3OH-$(C_2H_5)_2O$	HCl	222–223[b]	$C_{19}H_{22}ClN_3O_6$	53.84	53.89	5.23	5.29
8	CH_3	$(C_2H_5)_2NCH_2CH_2O$	NO_2	H	45	2-C_3H_7OH	HCl	171–172	$C_{21}H_{26}ClN_3O_6$	55.81	55.82	5.80	5.88
9	CH_3	$(C_2H_5)_2NCH_2CH_2O$	CH_3	NO_2	...	C_2H_5OH	Pier.	105–107	$C_{29}H_{32}N_6O_{13}$	51.78	51.78	4.80	5.00
10	CH_3	$(C_2H_5)_2NCH_2CH(CH_3)O$	NO_2	H	...	CH_3OH-$(C_2H_5)_2O$	HCl	185–187	$C_{22}H_{28}ClN_3O_6$	56.71	56.43	6.06	6.23
11[d]	CH_3	CH_3	NO_2	H	77	95% C_2H_5OH	...	164–166	$C_{16}H_{14}N_2O_5$	61.14	61.31	4.49	4.56
12	CH_3	CH_3	CH_3	NO_2	81	CH_3OH	...	100–101[e]	$C_{18}H_{18}N_2O_5$	63.15	62.17	5.30	5.55
13	CH_3	CH_3	CH_3	NH_2	26	C_6H_6-PE	...	117–118	$C_{18}H_{22}N_2O$	76.56	76.89	7.85	7.98
14[c]	CH_3	$(C_2H_5)_2NCH_2CH_2O$	CH_3	NH_2	35	C_2H_5OH-$(C_2H_5)_2O$	HCl	195–196	$C_{23}H_{34}ClN_3O_2$	65.77	65.54	8.16	8.38
15	H	$(CH_3)_2NCH_2CH_2O$	NH_2	H	67	C_6H_6-PE	...	130–132	$C_{18}H_{23}N_3O_2$	68.98	68.72	7.39	7.07
16	H	C_2H_5O	NH_2	H	...	C_6H_6-PE	...	88–89	$C_{16}H_{18}N_2O_2$	71.09	71.25	6.71	6.84
17	CH_3	$(C_2H_5)_2NCH_2CH_2O$	NH_2	H	70	[f]	...	[g]	$C_{21}H_{29}N_3O_2$	70.95	70.84	8.22	8.30
18	H	OH	$C_6H_5CH_2OCONH$	H	51	...	...	159–162	$C_{30}H_{26}N_2O_6$	70.57	70.34	5.13	5.13
19	CH_3	CH_3	$TsNCH_3$	H	59	EtOH	...	147–149[h]	$C_{32}H_{34}N_2O_5S_2$	65.06	64.97	5.80	6.01
					...	EtOH		170–171[h]			65.13		6.00
20	CH_3	CH_3	$TsNC_2H_5$	H	44	95% EtOH	...	166–167	$C_{34}H_{38}N_2O_5S_2$	65.99	65.89	6.19	6.23
21	CH_3	CH_3	CH_3NH	H	73	EtOH	HCl	203–204	$C_{18}H_{24}Cl_2N_2O\cdot 1/2\ H_2O$	59.34	59.54	6.92	7.03
22	CH_3	CH_3	C_2H_5NH	H	40	EtOH	...	96–97	$C_{20}H_{26}N_2O\cdot 1/2\ H_2O$	75.20	75.23	8.52	8.34
23	CH_3	CH_3	F	H	27	...	...	133 (0.7)	$C_{16}H_{14}F_2O$	73.83	73.81	5.42	5.55

[a] Yields refer to purified products. [b] Melted with decomposition. [c] Ref. 9. [d] M.p. 165.5–167.5° when prepared by a different method: J. Korman and E. C. Olsen, *J. Org. Chem.*, 22, 870 (1957). [e] Further recrystallization failed to improve the analysis, but the impure product could be reduced to No. 13. [f] Purified by chromatography on alumina in benzene. [g] Occurred as an oil. [h] Occurred in interconvertible dimorphic forms.

during acid hydrolysis of its methyl ester and subsequent recrystallization apparently arose from the use of alkali in isolating the product, since we observed no such degradation when using a similar procedure[2] which avoided alkaline conditions. Conversely, I, which lacks an acidic hydrogen atom on the α carbon atom, was stable under basic conditions.

The acids were converted to the respective chlorides and then to the esters. For the preparation of methyl ketones, the acid chlorides were treated with diazomethane and the resulting diazoketones were reduced with hydriodic acid. The use of dimethylcadmium for the conversion to methyl ketones gave no isolable product, although it is reported[4] that dimethylcadmium does not react with aromatic nitro groups.

The aromatic nitro compounds were reduced catalytically to the desired amino derivatives. In some cases, basic esters of I and II could not be reduced in or recrystallized from methanol or ethanol, since transesterified products were obtained; the reductions proceeded normally in dioxane.

In an attempt to avoid the low yield nitration required for the preparation of II, 2,2-bis(*p*-aminophenyl)acetic acid monohydrochloride was prepared directly[5] from aniline and dichloroacetic acid and converted to the carbobenzyloxy derivative. Treatment of the latter with β-diethylaminoethyl chloride[6] did not, however, result in an isolable product.

3,3 - Bis(*p* - *N* - methylaminophenyl) - 2 - butanone and the corresponding *N*-ethyl compound were prepared by a different sequence. 3,3-Bis(*p*-aminophenyl)-2-butanone (III)[7] was tosylated and the resulting sulfonamide was alkylated. Acid hydrolysis gave the desired secondary amines. 3,3-Bis(*p*-fluorophenyl)-2-butanone was obtained by tetrazotization of III, conversion to the fluoborate salt, and pyrolysis.

EXPERIMENTAL[8]

2,2-Bis(p-nitrophenylpropionic acid) (No. 1). A stirred mixture of 302 ml. of sulfuric acid, 71 ml. of water, 163 ml. of acetic acid, and 90.6 g. (0.28 mole) of methyl 2,2-bis(*p*-nitrophenyl)propionate[3] was heated at 95° for 18 hr. and then poured into water. The product was extracted into chloroform and the filtered chloroform solution was washed with 10% sodium hydroxide solution. Acidification of the alkaline wash with concd. hydrochloric acid precipitated the crystalline product, which was collected and recrystallized.

The crystalline *acid chloride (No. 2)* was obtained when a solution of 1.7 g. of the acid in 25 g. of thionyl chloride was refluxed for 1 hr. and the excess thionyl chloride was removed *in vacuo* with the aid of dry benzene.

2,2-Bis(p-methyl-x-nitrophenyl)propionyl chloride (No. 3).[9] A solution of 15 g. (0.04 mole) of 2,2-bis(*p*-methyl-*x*-nitrophenyl)propionic acid[10] in 300 g. of thionyl chloride was heated at 80° until the cessation of hydrogen chloride evolution and then evaporated *in vacuo*. The residue was recrystallized (Darco).

Nitro esters (Nos. 4, 5, 6, 7, 8, 9, and 10). A 10% solution of the appropriate acid chloride (1 mole equivalent) in benzene was treated with the requisite alcohol (2 mole equivalents) and the mixture was refluxed for 1 hr.[11] The cooled solution was washed, dried (sodium sulfate), filtered, and evaporated. The residue was recrystallized or converted to a salt. When 2,2-bis(*p*-nitrophenyl)acetyl chloride was esterified with 2-dibutylaminoethanol and the product was recrystallized from ethanol, transesterification took place and only the ethyl ester (No. 6) was obtained.

Methyl ketones by diazoketone synthesis (Nos. 11, 12). A 10% solution of the requisite acid chloride (1 mole equivalent) in methylene chloride was added dropwise to diazomethane (2 mole equivalents) in methylene chloride. The resulting solution was allowed to stand for 18 hr. and then stirred with excess 58% hydriodic acid. The layers were separated and the methylene chloride was washed successively with water, 10% sodium thiosulfate solution and water. The dried (sodium sulfate) solution was evaporated and the product was recrystallized.

3,3-Bis(p-methyl-x-aminophenyl)-2-butanone (No. 13). A warm solution of 1 g. (0.003 mole) of 3,3-bis(*p*-methyl-*x*-nitrophenyl)-2-butanone and 0.6 g. (0.018 mole) of hydrazine in 10 ml. of absolute alcohol was treated with portions of Raney Nickel from the tip of a microspatula until frothing ceased. The cooled solution was filtered and evaporated and the residue was recrystallized.

Reduction of nitro esters (Nos. 15, 16, and 17). A 10% solution of the nitro ester in pure dioxane was shaken with Adams' catalyst under hydrogen until the theoretical quantity of hydrogen was absorbed. The mixture was filtered and evaporated and the product was recrystallized.

2,2-Bis(p-carbobenzyloxyamidophenyl)acetic acid (No. 18). To a stirred solution of 14.0 g (0.5 mole) of 2,2-bis(*p*-aminophenyl)acetic acid monohydrochloride[5] in 30 ml. (0.12 mole) of 4*N* sodium hydroxide solution there was added, alternately and dropwise, 21.0 g. (0.12 mole) of benzyl chloroformate and 30 ml. (0.12 mole) of 4*N* sodium hydroxide at 5° during 30 min. After 15 min. a gum separated and stirring was continued for 2 hr. Then 150 ml. of ethyl acetate and 150 ml. of 10% hydrochloric acid were added and the layers were separated. The organic phase was washed with water, dried (sodium sulfate), filtered, and evaporated to leave an oil which crystallized on trituration with ether. The product could not be recrystallized but was obtained in pure condition on washing with chloroform.

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(5) G. Heller, *Ann.*, **375**, 261 (1910).

(6) H. Horenstein and H. Pahlicke, *Ber.*, **71**, 1644 (1938).

(7) M. J. Allen and A. H. Corwin, *J. Am. Chem. Soc.*, **72**, 117 (1950); W. L. Bencze and M. J. Allen, *J. Org. Chem.*, **22**, 352 (1957). We thank Dr. D. W. Blackburn and Mr. R. Lange for preparing this material.

(8) All melting points are corrected. Microanalyses were carried out by the Analytical Section.

(9) R. B. Holmes and A. J. Hill, U. S. Patent **2,423,025**, June 24, 1947, disclosed this compound without analytical results.

(10) A. Haiss, *Ber.*, **15**, 1474 (1882).

(11) In later runs it was found that the use of equimolar amounts of the aminoalcohol and 2,2-bis(*p*-nitrophenyl)-acetyl chloride gave cleaner products. It was also found advantageous to add the amino-alcohol slowly to a refluxing solution of the acid chloride. Both of these modifications serve to reduce the possibility of alkaline degradation of the nitro compound.

N-Alkyl derivatives of 3,3-bis(p-toluenesulfonamidophenyl)-2-butanone (Nos. 19 and 20). A mixture of 300 ml. of pyridine, 38.7 g. (0.12 mole) of 3,3-bis(*p*-aminophenyl)-2-butanone dihydrochloride[7] and 57.2 g. (0.3 mole) of *p*-toluenesulfony chloride was stirred for 45 min. at 27° and poured into water. The product was extracted into chloroform and the chloroform was washed successively with 20% sulfuric acid, 5% sodium carbonate solution, and water. The dried (magnesium sulfate), filtered chloroform solution was evaporated and the residue was dissolved in 10% sodium hydroxide solution and filtered. The filtrate was acidified with acetic acid to give a pink solid which was collected and dried. There was obtained 86.6 g. (95%) of product, m.p. 75–80°, which could not be recrystallized but which was suitable for use in subsequent reactions.

A 25% solution of the sulfonamide (1 mole equivalent) in ethanol was treated with 1*N* sodium hydroxide (3 mole equivalents) and the requisite alkyl iodide (3 mole equivalents) and stirred at 75° for 3 hr. It was partially evaporated and then extracted with benzene. The benzene layer was washed with water, dried (magnesium sulfate) and evaporated. The residue was recrystallized.

3,3-Bis(p-N-alkylaminophenyl)-2-butanones (Nos. 21 and 22). The appropriate sulfonamide derivative in two volumes of 80% sulfuric acid was heated at 155–160° for 5 min. The cooled solution was poured into water, made alkaline with 20% sodium hydroxide solution, and the product was extracted into ether. The washed, dried (potassium carbonate) ether extract was evaporated and the residue was recrystallized or converted to a salt.

3,3-Bis(p-fluorophenyl)-2-butanone (No. 23). A stirred solution of 16.4 g. (0.05 mole) of 2,2-bis(*p*-aminophenyl)-3-butanone dihydrochloride[7] in 25 ml. of 37% hydrochloric acid and 25 ml. of water was treated dropwise with a solution of 7.2 g. (0.105 mole) of sodium nitrite in 15 ml. of water at 0°. The excess nitrite was neutralized with urea and to the solution there was added 15.2 g. (0.14 mole) of sodium fluoborate in 30 ml. of water. The resulting precipitate was filtered, washed successively with 6 ml. of water, 3 ml. of methanol, and 10 ml. of ether and dried to give 17.0 g. (76%) of salt, m.p. 139° dec.

The diazonium fluoborate was decomposed by heating with a free yellow flame, and the residue was dissolved in chloroform, washed with dilute hydrochloric acid, 10% sodium hydroxide and water, and then dried (sodium sulfate). The filtered chloroform solution was fractionally distilled.

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γ-(*p*-Aminophenyl)butyric Acid

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The preparation of γ-(*p*-aminophenyl)butyric acid was reported by van der Scheer[1] in 1934 through reduction of γ-(*p*-nitrophenyl)butyric acid. The nitrated derivative was obtained from γ-phenylbutyric acid which in turn had been prepared from phenylethyl bromide by the malonic ester synthesis described by Fischer.[2]

Starting with γ-phenylbutyric acid which had been prepared through Clemmensen reduction[3] of β-benzoylpropionic acid, the procedure of van der Scheer gave the amino acid in an over-all yield of 6%. The nitration of γ-phenylbutyric acid to γ-(*p*-nitrophenyl)butyric acid was accomplished in only 20% yield, the formation of the *ortho* isomer predominating. Although van der Scheer reported a yield of 70% for the preparation of the γ-(*p*-aminophenyl)butyric acid by zinc dust–hydrochloric acid reduction of the nitro compound, the yield obtained in several reactions was not over 40%.

The need for considerable quantities of γ-(*p*-aminophenyl)butyric acid led to the development of the two-step synthesis described herein which affords the amino acid in an over-all yield of 43%. Acetanilide is succinoylated by the procedure described in the literature[4] to give β-(*p*-acetylaminobenzoyl)propionic acid in 60% yield. Treatment of this keto acid by the Huang-Minlon[5] modification of the Wolff-Kishner reaction effected both reduction of the carbonyl group and hydrolysis of the acetamido group in one step to form the γ-(*p*-aminophenyl)butyric acid.

Previous attempts to effect this combined hydrolysis-reduction through the Clemmensen reaction were not successful.

EXPERIMENTAL[6]

A mixture of β-(*p*-acetylaminobenzoyl)propionic acid[4] (77 g., 0.33 mole), 76 g. of potassium hydroxide, 55 ml. of hydrazine hydrate (85%), and 400 ml. of triethylene glycol were heated under reflux for 1.5 hr. The condenser was then removed and the temperature of the solution raised to 195° during which time excess hydrazine hydrate was expelled. (*Caution*—Hood). Refluxing was then continued for an additional 4 hr. at this temperature. The cooled solution was diluted with 400 ml. of water and made weakly acidic (to Alkacid paper) by the addition of 6*N* hydrochloric acid, about 200 ml. being required. The acid which precipitated was removed by filtration, washed with cold water, and dried in a vacuum desiccator over anhydrous calcium chloride; yield 42.8 g., 73%, m.p. 115–120°. Recrystallization of the analytical sample from water gave white plates, m.p. 130–132°. The melting point was not depressed when mixed with an authentic sample of γ-(*p*-aminophenyl)-butyric acid.[7]

Anal. Calcd. for $C_{10}H_{13}O_2N$: C, 67.02; H, 7.31; N, 7.82. Found: C, 66.96; H, 7.55; N, 7.76.

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(1) J. van der Scheer, *J. Am. Chem. Soc.*, **56**, 744 (1934).

(2) E. Fischer, *Ber.*, **39**, 2211 (1906).

(3) E. L. Martin, *Org. Syntheses*, **Coll. Vol. II**, John Wiley and Sons, Inc., New York, N. Y., 1943, p. 81.

(4) J. P. English, *et al.*, *J. Am. Chem. Soc.*, **67**, 2263 (1945).

(5) Huang-Minlon, *J. Am. Chem. Soc.*, **68**, 2487 (1946).

(6) All melting points are uncorrected.

(7) van der Scheer reported a melting point of 130–131°.