

TABLE I

Ar	Yield, %	°C.	B.p.,		n_D^{20}	Formula	Carbon, %		Hydrogen, %	
			Mm.	"			Calcd.	Found	Calcd.	Found
Phenyl	78	105	17		1.4809	C ₁₁ H ₁₄ O ₂	74.16	73.90	7.86	7.70
2-Methoxyphenyl	70	133	15		1.4913	C ₁₂ H ₁₆ O ₃	69.23	69.00	7.69	7.45
3-Methoxyphenyl	75	142	15		1.4918	C ₁₂ H ₁₆ O ₃	69.23	69.46	7.69	7.80
4-Methoxyphenyl	70	135	11		1.4905	C ₁₂ H ₁₆ O ₃	69.23	69.27	7.69	7.68
3-Methylphenyl	62	120	15		1.4828	C ₁₂ H ₁₆ O ₂	75.00	74.60	8.33	8.12

^a Boiling points are uncorrected. Analyses by Mrs. G. L. Sauvage.

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6-Chloro-9-[2-(2'-hydroxyethylamino)-ethylamino]-2-methoxyacridine

The reaction of 6,9-dichloro-2-methoxyacridine with N-(2-hydroxyethyl)-ethylenediamine was carried out in a phenol melt after the method employed for quinacrine.¹ An 83.5% yield of 6-chloro-9-[2-(2'-hydroxyethylamino)-ethylamino]-2-methoxyacridine dihydrochloride was isolated; after crystallization from water the yellow microcrystalline solid melted 289–292° dec. This compound ("Neo-acranil") has been tested for antibacterial activity.²

Anal. Calcd. for C₁₈H₂₀ClN₃O·2HCl: base, 82.6; HCl, 17.4; Cl (total), 25.4. Found^{3,4}: base,⁵ 82.3; HCl, 17.7; Cl (total), 25.2.

The dihydrochloride was converted to the base with aqueous ammonia; it separated from aqueous ethanol as golden needles, m.p. 184–184.5° (cor.).

Anal. Calcd. for C₁₈H₂₀ClN₃O: Cl, 10.75; N, 12.74. Found³: Cl, 10.30; N, 12.92.

(1) F. Mietzsch and H. Mauss, U. S. Patent 2,113,357.

(2) G. R. Goetchius and C. A. Lawrence, *J. Lab. Clin. Med.*, **29**, 134 (1944).

(3) Analyses by Mr. M. E. Auerbach.

(4) Dry basis: sample contained 6.2% moisture.

(5) Method of M. E. Auerbach, *J. Amer. Pharm. Assoc., Sci. Ed.*, **26**, 231 (1937).

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Derivatives of 3-Methoxy-4-nitrobenzoic Acid and 3-Carbomethoxy-4-nitrobenzoic Acid

3-Methoxy-4-nitrobenzoyl chloride was prepared by treating the acid¹ with phosphorus pentachloride on a steam-bath for about 30 minutes. After removing the phosphorus oxychloride by vacuum distillation, the acid chloride was recovered by extracting the crude product with hot petroleum ether, from which the acid chloride crystallized, on cooling, in light yellow needles; m.p. 63–63.5°.

Anal. Calcd. for C₈H₆O₄Cl: Cl, 16.47; Found: Cl, 16.45.

The anilide was prepared by treating a benzene solution of the acid chloride with an excess of aniline. After several recrystallizations from ethanol and water, the flat, white plates melted at 162–163°.

Anal. Calcd. for C₁₄H₁₂O₄N₂: N, 10.30. Found: N, 10.43.

The amide was prepared by adding an excess of ammonia to a benzene solution of the acid chloride. The product was recrystallized several times from ethanol, forming large crystals which softened at 189° and melted at 193–196°.

Anal. Calcd. for C₈H₈O₄N₂: N, 14.30. Found: N, 14.42.

(1) Prepared by oxidation of 3-methoxy-4-nitrotoluene by method adapted from "Organic Syntheses," Coll. Vol. II, John Wiley and Sons, Inc., New York, N. Y., 1943, p. 135.

3-Nitro-4-carbomethoxybenzoyl chloride, formed as before from the acid² and phosphorus pentachloride, crystallized in small, white crystals on cooling the petroleum ether extraction; m.p. 91–94°.

Anal. Calcd. for C₉H₆O₅NCl: N, 5.75. Found: N, 5.92.

The anilide crystallized from methanol and water in fine, white needles which melted at 140–142° after several recrystallizations.

Anal. Calcd. for C₁₅H₁₅O₅N₂: N, 9.34. Found: N, 9.54.

The amide crystallized from methanol as flat, diamond-shaped crystals melting at 196–198.5°.

Anal. Calcd. for C₉H₈O₅N₂: N, 12.50. Found: N, 12.58.

(2) Prepared from dimethyl 4-nitroisophthalate according to method of P. Axer, *Monatsh.*, **41**, 153 (1920).

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4-Methylglutamic Acid

A new amino acid amide, isolated from peanut plants, is believed to be the amide of either an unsaturated 2-amino adipic acid or of an unsaturated 4-methylglutamic acid.¹ Done and Fowden mention that 4-methylglutamic acid was not available for comparison. We prepared this amino acid in 1945 from acetamidomalonic and acetamidocyanooacetic esters. Loss of a carbethoxy group in the preparation of ethyl 2-acetamido-4-carbomethoxyvalerate was to be expected.²

Ethyl 2-Acetamido-4-carbomethoxyvalerate.—To a solution of 24 ml. of methyl methacrylate and 43.5 g. of acetamidomalonic ester in 100 ml. of dry ethanol was added a solution of 0.4 g. of sodium in 30 ml. of ethanol. The resulting solution was refluxed for five hours and concentrated to dryness. The residue was recrystallized from benzene, filtering from sodium ethylate. The solution was cooled to room temperature, filtered and washed with Skellysolve A to remove the yellow color; yield 33.8 g. melting at 105–108°. Cooling the filtrate gave 5.8 g. melting at 100–104°. Concentration of the filtrate to small volume gave 5.2 g. of lower melting material. A sample for analysis, recrystallized twice from benzene, melted at 108°.

Anal. Calcd. for C₁₁H₁₉NO₅: N, 5.71. Found: N, 5.73.

4-Methylglutamic Acid.—In the above manner, methyl methacrylate (12 ml.) was condensed with acetamidocyanooacetic ester (17 g.) to give 19.3 g. of viscous liquid. This was refluxed for four hours with 80 ml. of concentrated hydrochloric acid and then concentrated to dryness *in vacuo*. The residue was dissolved in a minimum amount of warm water, brought to pH 3 with ammonium hydroxide, treated with charcoal, filtered and chilled overnight to give about 8 g. of crude product. Recrystallization from 21 ml. of water gave 2 g. of amino acid. Recrystallization from 6 ml. of water then gave 1.1 g. melting at 168–169°.

Anal. Calcd. for C₈H₁₁NO₄: N, 8.69. Found: N, 8.69.

The amino acid was obtained in better yield by hydrolyzing the intermediate cyano compound with sulfuric acid

(1) J. Done and L. Fowden, *Biochem. J.*, **49**, XX (1951).

(2) Cf. F. H. McMillan and N. F. Albertson, *THIS JOURNAL*, **70**, 3778 (1948).