Nitrophthaloyl and Aminophthaloyl Derivatives of Amino Acids

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> Seventeen 3-nitrophthalimido acids, 18 4-nitrophthalimido acids, 14 3-aminophthalimido acids, and 14 4-aminophthalimido acids have been prepared.

A RECENT paper (3) described the preparation and some of the properties of the nitrophthaloyl and aminophthaloyl derivatives of glycine. This work has been extended to the preparation of the nitrophthaloyl and aminophthaloyl derivatives of a number of other amino acids. Although it gives excellent results with glycine, the use of nitrobenzene as a solvent for nitrophthaloylations was unsatisfactory with other amino acids. The phthaloylation method of Billman and Harting (2) gave satisfactory nitrophthaloylations in most cases. The nitrophthalic acids were as effective as their anhydrides in achieving nitrophthaloylation by this method. The aminophthaloyl deriva-

tives were prepared by hydrogenation of the nitrophthaloyl derivatives.

EXPERIMENTAL

Nitrophthaloyl Derivatives of Amino Acids. Intimate mixtures of 0.01 mole of an amino acid with 0.01 mole of a nitrophthalic acid or anhydride were fused according to the method of Billman and Harting (2). The products were recrystallized until no further improvement in melting points resulted. The experimental data are summarized in Table I. The products are all ivory to cream-colored powders.

Table I. Nitrophthaloyl Derivatives of Amino Acids^a

pL-Alanine37550% Ethanol165-7pL-Alanine47670% Ethanol161.5-3.5s-Alanine38370% Ethanol145-6's-Alanine465Water153-5'pL-a-Amino-n-butyric acid350Water153-5'pL-a-Amino-n-butyric acid454Benzene157-8'a-Aminoisobutyric acid38095% Ethanol203-5'a-Aminoisobutyric acid45975% Ethanol187-9 γ -Amino-n-butyric acid38295% Ethanol165-6'pL-Norvaline3703 to 1 C_H=C_4H_1140-2pL-Norvaline3703 to 1 C_H=C_4H_1140-2pL-Norvaline3841 to 2 CHCL=CCL130-2pL-Norvaline482Benzene104-6pL-Valine356Benzene159-60.5pL-Isovaline46220% Ethanol153-5pL-Isovaline3591 to 1 C_4H=C_4H_4113-15pL-Leucine3802 to 3 CHCL=CCL132-3pL-Leucine3671 to 1 CL=CCL152-3.5pL-Leucine3601 to 1 C_4H=C_4H_4113-15pL-a-Amino-n-caprylic acid3961 to 3 CCL=CCH_4143-5pL-a-Amino-n-caprylic acid3601 to 3 CCL=CCH_4143-5pL-a-Amino-n-caprylic acid3661 to 1 C_4H=C_4H_4114-16-2pL-a-Amino-n-caprylic acid <th>Amino Acid</th> <th>Position of Nitro Group</th> <th>$\mathop{\rm Yield}_{\%}$</th> <th>Recrystallized from</th> <th>M.P., ° C^{<i>i</i>}</th>	Amino Acid	Position of Nitro Group	$\mathop{\rm Yield}_{\%}$	Recrystallized from	M.P. , ° C ^{<i>i</i>}
bL-Alanine 4 76 70% Ethanol 161.5-3.5 β -Alanine 3 83 70% Ethanol 145-6' β -Alanine 4 65 Water 209-10' DL-ar-Amino-n-butyric acid 3 50 Water 153-5' DL-ar-Amino-n-butyric acid 3 60 95% Ethanol 203-5' ar-Aminoisobutyric acid 4 59 75% Ethanol 187-9 γ -Amino-n-butyric acid 3 82 95% Ethanol 132-3' γ -Amino-n-butyric acid 3 82 95% Ethanol 132-3' γ -Amino-n-butyric acid 4 40 95% Ethanol 132-3' γ -Amino-n-butyric acid 3 82 95% Ethanol 132-3' γ -Amino-n-butyric acid 4 42 Benzene 104-6 DL-Norvaline 3 56 Benzene 159-6'. DL-Norvaline 4 82 3 to 1 C_4HC_4H_4 141-3 DL-Sovaline 4 62 20% Ethanol 153-5 DL-Norvaline 3 59 1 to 1 C_4HC_4H_4 <td< td=""><td>DL-Alanine</td><td>3</td><td>75</td><td>50% Ethanol</td><td>165 - 7</td></td<>	DL-Alanine	3	75	50% Ethanol	165 - 7
β -Alanine 3 83 70% Ethanol $145-6'$ β -Alanine 4 65 Water $209-10^4$ $DL-a-Amino-n-butyric acid 3 50 Water 153-5' DL-a-Amino-n-butyric acid 4 54 Benzene 157-8' a-Aminoisobutyric acid 3 60 9\% Ethanol 203-5' a-Aminoisobutyric acid 4 59 7\% Ethanol 187-9 \gamma-Amino-n-butyric acid 4 40 95\% Ethanol 182-3' p-Amino-n-butyric acid 4 40 95\% Ethanol 165-6' DL-Norvaline 3 70 3 10 \ cl_H-Ccl_H 140-2 DL-Norvaline 4 82 10 \ cl_H-Ccl_H 140-2 DL-Valine 3 84 10 \ cl_H-Ccl_H 141-3 DL-Isovaline 3 56 Benzene 159-60.5 DL-Norvaline 4 62 20\% Ethanol 153-5 DL-Soleucine 3 59 1 t \ Cl_L-Ccl_H 132-3 DL-Norleucine 4 $					
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	β-Alanine		83		
DL- α -Amino- n -butyric acid350Water153-5'DL- α -Amino- n -butyric acid454Benzene157-8' α -Aminoisobutyric acid36095% Ethanol203-5' α -Aminoisobutyric acid45975% Ethanol187-9 γ -Amino- n -butyric acid38295% Ethanol182-3' γ -Amino- n -butyric acid44095% Ethanol182-3' γ -Amino- n -butyric acid44095% Ethanol166-6'DL-Norvaline3703 to 1 CeHe-CeH1,140-2DL-Norvaline482Benzene104-6DL-Valine3841 to 2 CHCl_3-CCL,130-2DL-Valine356Benzene159-60.5DL-Isovaline3591 to 1 CeHe-CeH1,113-15DL-Norleucine4823 to 1 CCL_4-CcH,132-3'DL-Leucine3802 to 3 CHCL-CCL,136-7'DL-Leucine3671 to 1 CHCl_5-CCL,136-7'DL-Leucine3671 to 1 CHCl_5-CCH,160-2DL-APDenyler4601 to 3 CCL_4-CeH,118-19.5'DL- α -Phenyler4601 to 3 Acetone-CeH,203-5'DL- α -Phenyler496Benzene188-7'DL- α -Phenyler496Benzene188-7'DL- α -Phenyler366'1 to 3 Acetone-CeHs203-5'DL- α -Phenyler496Benzene188-7'	8-Alanine	4	65		
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$DL-\alpha$ -Amino- <i>n</i> -butyric acid	3			
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α -Amino- <i>n</i> -butyric acid459 75% Ethanol $187-9$ γ -Amino- <i>n</i> -butyric acid382 95% Ethanol $132-3^n$ γ -Amino- <i>n</i> -butyric acid440 95% Ethanol $132-3^n$ γ -Amino- <i>n</i> -butyric acid $132-3^n$ γ -Amino- <i>n</i> -butyric acid $140-2$ p_1 -Norvaline3703 to 1 C_sH_c-C_sH_{14} $140-2$ p_1 -Norvaline482Benzene $104-6$ p_1 -Valine3841 to 2 CHCl_n-CCl_4 $130-2$ p_1 -Valine4821 to 2 CHCl_n-CCl_4 $141-3$ p_1 -Isovaline462 20% Ethanol $153-5$ p_1 -Isovaline462 20% Ethanol $153-5$ p_1 -Norleucine3591 to 1 C_nH_n-C_sH_{14} $113-15$ p_1 -Norleucine4823 to 1 CCl_n-CCl_4 $160-2$ p_1 -Norleucine4823 to 1 CCl_n-Ccl_4 $160-2$ p_1 -Leucine3671 to 1 CH_n-Cl_4 $160-2$ p_1 -Leucine3671 to 1 CCl_n-Ccl_4 $160-2$ p_1 -Asimo- <i>n</i> -caprylic acid3961 to 2 C_cH_n-CsH_{14} $118-19.5$ $p_1-\alpha$ -Amino- <i>n</i> -caprylic acid4501 to 3 Acetone-C_sH_s $203-5$ $p_1-\alpha$ -Amino- <i>n</i> -caprylic acid3621 to 3 Acetone-C_sH_s $203-5$ $p_1-\alpha$ -Amino-1-cyclopentanecarboxylic acid366Benzene $208-5-10$ 1 -Amino-1-cyclopentanecarboxylic acid366Benzene $208-5-10$ 1 -Amino-1-cycl		3	60	95%Ethanol	
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$\begin{array}{cccccccccccccccccccccccccccccccccccc$			40		
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		3			
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	DL-Norvaline	4	82		
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	DL-Valine			1 to 2 CHCl ₃ -CCl ₄	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	DL-Valine	4	82		
DL-Norleucine3591 to 1 $C_6H_8-C_6H_{14}$ 113-15DL-Norleucine4823 to 1 $CCl_4-C_6H_{14}$ 132-3DL-Leucine3802 to 3 $CHCl_4-Ccl_1$ 152-3.5DL-Leucine465 CCl_4 186-7DL-Isoleucine3671 to 1 $C_8H_8-C_6H_{14}$ 118-19.5DL-acAmino-n-caprylic acid3961 to 2 $C_8H_8-C_6H_{14}$ 118-19.5DL-ac-Amino-n-caprylic acid4501 to 3 $CCl_4-C_8H_{14}$ 118-15-16.5DL-ac-Amino-n-caprylic acid4501 to 3 Acetone-C_8H_8203-5DL-Phenylalanine3621 to 3 Acetone-C_8H_8203-5DL-Phenylalanine496Benzene189-911-Amino-1-cyclopentanecarboxylic acid3642 to 1 $C_8H_8-C_8H_{14}$ 114-16DL-Methionine'3642 to 1 $C_8H_8-C_8H_{14}$ 114-16DL-Methionine'3642 to 1 $C_8H_8-C_8H_{14}$ 114-16DL-Methionine'3642 to 1 $C_8H_8-C_8H_{14}$ 114-16DL-Aspartic acid330Water230-1DL-Aspartic acid330Water204-5DL-Glutamic acid'''313^nWater190-2	DL-Isovaline	3	56	Benzene	159 - 60.5
DL-Norleucine4823 to 1 CCl ₄ -C ₆ H ₁₄ 132-3DL-Leucine3802 to 3 CHCl ₄ -CCl ₄ 136-7DL-Leucine465CCl ₄ 136-7DL-Isoleucine3671 to 1 CHCl ₃ -CCl ₄ 160-2DL-Isoleucine4601 to 1 CsH ₆ -C ₆ H ₁₄ 118-19.5DL- α -Amino-n-caprylic acid3961 to 2 CsH ₆ -C ₆ H ₁₄ 94.5-5.5DL- α -Amino-n-caprylic acid4501 to 3 CCl ₄ -CsH ₁₄ 115-16.5DL- α -Amino-n-caprylic acid496Benzene186-7DL-Phenylalanine3621 to 3 Acetone-CsH ₆ 203-5DL-Phenylalanine496Benzene189-911-Amino-1-cyclopentanecarboxylic acid356'Benzene189-911-Amino-1-cyclopentanecarboxylic acid3642 to 1 CsH ₆ -CcH ₁₄ 114-16DL-Methionine'4771 to 1 CHCl ₃ -CCl ₄ 138-9DL-Aspartic acid330Water200-1	DL-Isovaline	4	62	20% Ethanol	153 - 5
DL-Leucine3802 to 3 CHCl ₃ -CCl ₄ 152-3.5DL-Leucine465CCl ₄ 136-7DL-Isoleucine3671 to 1 CHCl ₃ -CCl ₄ 160-2DL-Isoleucine4601 to 1 CsH ₆ -C ₆ H ₁₄ 118-19.5DL- α -Amino-n-caprylic acid3961 to 2 CsH ₆ -C ₆ H ₁₄ 94.5-5.5DL- α -Amino-n-caprylic acid4501 to 3 CCl ₄ -CsH ₁₄ 115-16.5DL- α -Amino-n-caprylic acid499Benzene186-7DL- α -Phenylglycine419Benzene186-7DL-Phenylalanine3621 to 3 Acetone-CsH ₆ 203-5DL-Phenylalanine496Benzene189-911-Amino-1-cyclopentanecarboxylic acid356'Benzene189-911-Amino-1-cyclopentanecarboxylic acid3642 to 1 CsH ₆ -CcH ₁₄ 114-16DL-Methionine'3642 to 1 CsH ₆ -CcH ₁₄ 114-16DL-Aspartic acid330Water200-1DL-Aspartic acid330Water200-1	DL-Norleucine	3	59	1 to 1 $C_6H_6-C_6H_{14}$	113 - 15
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	DL-Norleucine	4	82	3 to 1 $CCl_4-C_6H_{14}$	132 - 3
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	DL-Leucine	3	80	2 to 3 CHCl ₃ – CCl_3	152 - 3.5
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	DL-Leucine	4	65	CCl_4	136 - 7
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	DL-Isoleucine	3	67	1 to 1 CHCl ₃ -CCl ₄	160 - 2
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	DL-Isoleucine	4	60	1 to 1 $C_6H_6-C_6H_{14}$	118 - 19.5
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		3	96	1 to 2 $C_6H_6-C_6H_{14}$	94.5 - 5.5
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	DL- α -Amino- <i>n</i> -caprylic acid	4	50	1 to 3 $CCl_4-C_6H_{14}$	115 - 16.5
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$DL-\alpha$ -Phenylglycine	4	19	Benzene	186 - 7
	DL-Phenylalanine	3	62	1 to 3 Acetone– C_6H_6	203 - 5
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	DL-Phenylalanine	4	96	Benzene	208.5 - 10
DL-Methionine3642 to 1 $C_6H_6-C_6H_{14}$ 114-16DL-Methionine4771 to 1 CHCl ₃ -CCl ₄ 138-9DL-Aspartic acid330Water230-1DL-Aspartic acid462Water204-5DL-Glutamic acid ^m 313 ⁿ Water190-2	1-Amino-1-cyclopentanecarboxylic acid	3	56'	Benzene	189-91
DL-Methionine 4 77 1 to 1 $CHCl_3-CCl_4$ 138-9 DL-Aspartic acid 3 30 Water 230-1 DL-Aspartic acid 4 62 Water 204-5 DL-Glutamic acid ^m 3 13 ⁿ Water 190-2	1-Amino-1-cyclopentanecarboxylic acid	4	62^{i}	2 to 1 EtOH– C_6H_6	158.5 - 60
DL-Aspartic acid 3 30 Water 230-1 DL-Aspartic acid 4 62 Water 204-5 DL-Glutamic acid ^m 3 13 ⁿ Water 190-2					
DL-Aspartic acid 4 62 Water 204-5 DL-Glutamic acid ^m 3 13 ⁿ Water 190-2	DL-Methionine				138 - 9
DL-Glutamic acid" 3 13" Water 190–2		3		Water	230 - 1
				Water	204-5
DL-Glutamic acid ^m 4 31 Water 227-8					190 - 2
	DL-Glutamic acid ^m	4	31	Water	227 - 8

^a Analysis done by Midwest Microlab, Inc., Indianapolis, Ind., and deposited with ADI. ^bElectrothermal apparatus. ^cReported (4) m.p. 145-8° C. ^dReported (4) m.p. 207-9° C. ^eReported (1) m.p. 154-5° C. ^fReported (1) m.p. 163-4° C. ^eReported (4) m.p. 205-7° C. ^hReported (4) m.p. 134.5-6° C. ⁱReported (4) m.p. 165-7° C. ^jPlus 5 to 10% recovery of the amino acid. ^kAnal. Calcd.: S, 9.89; Found: S, 9.84. ⁱAnal. Calcd.: S, 9.85. ^mAlthough prepared from L(+)-glutamic acid, the derivative was optically inactive. ^aPlus 28% recovery of 3-nitrophthalic acid.

Table II. Aminophthaloyl Derivatives of Amino Acids^a

	Position			
	of Amino	Yield,	Recrystallized	
Amino Acid	Group	%	from	M.P., ° C.
DL-Alanine	3	85	80% Ethanol	204-5
DL-Alanine	4	80	Water	226 - 7
β -Alanine	3	90	Water	157 - 8
β -Alanine	4	68	Water	202 - 3
$DL-\alpha$ -Amino- <i>n</i> -butyric acid	3	75	Benzene	153 - 4.5
$DL-\alpha$ -Amino- <i>n</i> -butyric acid	4	74	75% Ethanol	$183 - 4.5^{\circ}$
α -Aminoisobutyric acid	3	72	Water	225 - 7
α -Aminoisobutyric acid	4	77	50% Ethanol	254-5
γ -Amino- <i>n</i> -butyric acid	3	87	Water	160 - 2
γ -Amino- <i>n</i> -butyric acid	4	86	Water	185.5 - 6.5
DL-Norvaline	3	88	Benzene	156.5 - 8
DL-Norvaline	4	72	40% Methanol	74-6
DL-Valine	3	60	10% Ethanol	137 - 8
DL-Valine	4	93	50% Methanol	95 - 7
DL-Isovaline	3	88	75% Ethanol	199.5 - 200.5
DL-Isovaline	4	80	50% Ethanol	199 - 200
DL-Norleucine	4	64	20% Methanol	57-9
DL-Isoleucine	3	92	Benzene	138 - 40
$DL-\alpha$ -Amino- <i>n</i> -caprylic acid	3	77	1 to 1 $C_6H_6-C_6H_{14}$	113 - 14.5
DL-α-Amino- <i>n</i> -caprylic acid	4	46	1 to 1 $CHCl_3$ - CCl_4	142.5 - 3.5
DL-Phenylalanine	3	68	75% Ethanol	177 - 8.5
DL-Phenylalanine	4	86	50% Methanol	216 - 18
1-Amino-1-cyclopentanecarboxylic acid	3	83	Benzene	169.5 - 70.5
1-Amino-1-cyclopentanecarboxylic acid	4	87	Benzene	223-6
DL-Aspartic acid	3	95	Water	$235-7^{d}$
DL-Aspartic acid	4	81	Water	$230-2^{d}$
DL-Glutamic acid	3	91	Water	214 - 15
DL-Glutamic acid	4	99	1 to 1 CH ₃ OH–C ₆ H ₆	205-6

^a Analysis done by Midwest Microlab, Inc., and deposited with ADI. ^b Electrothermal apparatus. ^c Reported (1) m.p. 182-4^o C. ^d Decomposition with bubbling.

Aminophthaloyl Derivatives of Amino Acids. Mixtures of 1 to 2 grams of a nitrophthaloyl derivative, 0.3 to 0.5 gram of 10% palladium-charcoal catalyst, and 25 ml. of 95% ethanol were hydrogenated for 1 hour at room temperature in a Parr Model 3910 hydrogenation apparatus, with initial pressures of 65 p.s.i.g. The outgassed mixtures were filtered, and the filtrates were evaporated to dryness on a steam bath. The residues were recrystallized three to five times. The results are summarized in Table II. All of the aminophthalimido acids are bright-yellow, crystalline powders.

ACKNOWLEDGMENT

Thanks are due to Judy Howell for her assistance in the synthesis of some of the nitrophthalimido acids.

LITERATURE CITED

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CORRECTION

In the article "Metal Complexes of Cyanoformamidines, Oxamidines, and Oxalimidates" by H.M. Woodburn, R.H. Salvesen, J.R. Fisher, W.E. Hoffman, E.L. Graminski, and R.L. VanDeusen [J. CHEM. ENG. DATA 12, 615 (1967)], the last line in Table II on page 616 should read:

R	n	М	x	Appearance	M.P., ° C.
2-Hydroxyethyl	2	Ni	2	Yellow orange powder	224–5 dec.