NEW COMPOUNDS

Phenylpropionyl-*dl*-alanine and Phenylbutyryl-*dl*-alanine

Two new derivatives of alanine have been prepared from the amino acid and the corresponding acid chlorides in the presence of alkali.[1] After isolation, each compound was thrice recrystallized from water, dried, washed with benzene, dried and analyzed. The recrystallizations served to raise the melting points one to three degrees to constant values.

β-Phenylpropionyl-*dl*-alanine.—M. p. 139.5–140° (uncor.).

Anal. Calcd. for $C_{12}H_{15}O_3N$: C, 65.14; H, 6.83; N, 6.33; neut. eq., 221. Found: C, 65.06; H, 6.76; N, 6.36; neut. eq., 223.[2]

γ-Phenylbutyryl-*dl*-alanine.—M. p. 120–121° (uncor.).

Anal. Calcd. for $C_{13}H_{17}O_3N$: C, 66.35; H, 7.28; N, 5.96; neut. eq., 235. Found: C, 66.21; H, 7.27; N, 6.07; neut. eq., 233.

Phenylacetyl-*dl*-alanine was also prepared by the above procedure and was found to melt at 150.5–151°. Shiple and Sherwin report 150–152°.[1]

(1) Shiple and Sherwin, *J. Biol. Chem.*, **53**, 463 (1922).

(2) Analyses for C, H and N by Dr. Francine Schwarzkopf, Elmhurst, L. I., New York.

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Derivatives of 3,5-Dimethylhexanoic Acid[1]

6-Benzoylamino-3,5-dimethylhexanoic Acid.—3,5-Dimethylcyclohexanone oxime, 34.4 g., was converted to the benzoylamino acid according to the procedure of Marvel and Eck.[2] The product, m. p. 107.5–109°, weighed 45 g.

Anal. Calcd. for $C_{25}H_{21}O_3N$: C, 68.44; H, 7.99; N, 5.33. Found: C, 68.24; H, 8.28; N, 5.52.

The intermediate lactam and 6-amino acid were isolated previously.[3]

6-Benzoylamino-2-bromo-3,5-dimethylhexanoic Acid.—6-Benzoylamino-3,5-dimethylhexanoic acid, 84 g., was brominated in the usual way.[4] The yield of the 2-bromo acid, which melted at 140.5–144.5°, was 50 g.

Anal. Calcd. for $C_{15}H_{20}O_3NBr$: C, 52.63; H, 5.85. Found: C, 52.44; H, 6.02.

(1) From the Ph.D. Thesis of A. D. McLaren, University of Missouri, 1943.

(2) Eck and Marvel, *Organic Syntheses*, **19**, 20 (1939).

(3) Ungnade and McLaren, *J. Org. Chem.*, **10**, 29 (1945).

(4) Eck and Marvel, *Organic Syntheses*, **19**, 18 (1939).

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2-Decen-1-ol, 2-Undecen-1-ol, 2-Decenal, 2-Undecenal and Derivatives

2-Decen-1-ol and 2-undecen-1-ol prepared by the method of Delaby[1] had refractive indices, $n^{25}D$ 1.4482 (Delaby, $n^{16}D$ 1.4510) and 1.4490 and densities, d^{25}_4 0.8440 (Delaby, d^{16}_4 0.846) and 0.8375, respectively. The melting points and analyses of the 3,5-dinitrobenzoates and *p*-nitrophenylurethans of these alcohols are shown in Table I.

(1) Delaby, *Bull. soc. chim.*, [5] **3**, 2375 (1936).

TABLE I

3,5-DINITROBENZOATES AND *p*-NITROPHENYLURETHANS OF 2-DECEN-1-OL AND 2-UNDECEN-1-OL

Compound		2-Decen-1-ol		2-Undecen-1-ol	
Alcohol Derivative		3,5-Dinitrobenzoate	*p*-Nitrophenylurethan	3,5-Dinitrobenzoate	*p*-Nitrophenylurethan
Formula		$C_{16}H_{22}N_2O_6$	$C_{17}H_{26}N_2O_4$	$C_{17}H_{24}N_2O_6$	$C_{18}H_{28}N_2O_4$
M. p., °C.		62.5	81.5	66.0–66.5	78.0–78.5
Analyses, % Carbon	Calcd.	58.2	63.7	59.0	64.1
	Found	58.0	63.6	59.2	64.5
Hydrogen	Calcd.	6.3	7.5	6.6	7.8
	Found	6.1	7.5	6.5	7.8
Nitrogen	Calcd.	8.0	8.7	7.9	8.4
	Found	8.0	8.9	7.8	8.4

2-Decenal and 2-undecenal were prepared from their corresponding alcohols by the oxidation method of Delaby and Guillot-Allègre.[2] The melting points and analyses of the semicarbazones and 2,4-dinitrophenylhydrazones of these aldehydes are shown in Table II. The absorption spectra of the aldehydes, semicarbazones, and 2,4-dinitrophenylhydrazones (Table III, exhibit single maxima having characteristic extinction coefficients for these types of compounds.[3,4]

TABLE II

SEMICARBAZONES AND 2,4-DINITROPHENYLHYDRAZONES OF 2-DECENAL AND 2-UNDECENAL

Compound		2-Decenal		2-Undecenal	
Aldehyde Derivative		Semicarbazone	2,4-Dinitrophenylhydrazone	Semicarbazone	2,4-Dinitrophenylhydrazone
Formula		$C_{11}H_{21}N_3O$	$C_{16}H_{22}N_4O_4$	$C_{12}H_{23}N_3O$	$C_{17}H_{24}N_4O_4$
M. p., °C.		167–168[a]	128.8	161.5–162.5	122.6
Analyses, % Carbon	Calcd.	63.9	57.5	64.0	58.6
	Found	63.8	57.2	63.8	58.1
Hydrogen	Calcd.	10.3	6.6	10.3	6.9
	Found	10.1	6.4	10.2	6.8
Nitrogen	Calcd.	18.7	16.8	18.7	16.1
	Found	18.9	16.9	18.9	16.3

[a] Delaby, ref. 1, m. p. 168.5°.

TABLE III

ABSORPTION SPECTRAL DATA FOR 2-DECENAL, 2-UNDECENAL AND THEIR SEMICARBAZONES AND 2,4-DINITROPHENYLHYDRAZONES

	Characteristic of absorption spectra	Maximum, mμ	Extinction coefficient, $E^{g./l.}_{1\,cm.}$	Molar extinction, $E^{mol./l.}_{1\,cm.}$
2-Decenal	Aldehyde	221–222	109.0	16,867
	Semicarbazone	264	143.0	30,375
	2,4-Dinitrophenylhydrazone	377	86.3	28,824
2-Undecenal	Aldehyde	221–222	100.4	16,786
	Semicarbazone	263–264	135.0	30,173
	2,4-Dinitrophenylhydrazone	374–378	81.7	28,445

(2) Delaby and Guillot-Allègre, *Bull. soc. chim.*, [4] **53**, 301 (1933).

(3) Evans and Gillam, *J. Chem. Soc.*, 565 (1943).

(4) Roberts and Green, THIS JOURNAL, **68**, 214 (1946).

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