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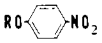
Some *p*-Nitrophenyl Esters of *N*-Acylated Amino Acids

A. ASZALOS, P. LEMANSKI, and B. BERK
The Squibb Institute for Medical Research, New Brunswick, N. J.

p-Nitrophenyl esters of some N-acylated glycines and N-acylated dl-leucines were prepared. An example of the method of preparation is cited and melting points and yields are listed.

DURING the preparation of analogs of the antibiotic septacidin, *p*-nitrophenyl esters of *N*-acylated amino acids were prepared (*1*, *3*). Dicyclohexyl carbodiimide was used as the condensing agent in the synthesis of these compounds (*2*). As an example, the preparation of the *p*-nitrophenyl ester of *N*-capryl glycine is described.

Table I. Analysis of Acyl Glycine and Leucine Esters

		Yield,		Calculated, %			Found, %		
R	M.P.,[a] ° C.	%	Formula	C	H	N	C	H	N
Acetyl glycyl	125–126 (lit. 125–126) (*6*)	60	$C_{10}H_{10}N_2O_5$	50.42	4.23	11.76	49.81	4.63	11.55
Caproyl glycyl	102–102.5	70	$C_{14}H_{18}N_2O_5$	57.14	6.12	9.49	57.34	5.87	9.79
Capryl glycyl	95–95	66	$C_{18}H_{26}N_2O_5$	61.70	7.48	8.00	61.50	7.55	7.80
Lauroyl glycyl	96–97.5	67	$C_{20}H_{30}N_2O_5$	63.30	8.24	7.38	62.93	8.22	7.48
Myristoyl glycyl	101–103	65	$C_{22}H_{34}N_2O_5$	65.00	8.43	6.89	65.10	8.53	7.00
Palmitoyl glycyl	108–109	58	$C_{24}H_{38}N_2O_5$	66.35	8.75	6.45	66.57	8.96	6.44
Acetyl *dl*-leucyl	118–119 (Lit.125–126) (*5*)	50	$C_{14}H_{18}N_2O_5$	57.13	6.12	9.49	57.56	6.41	9.43
Caproyl *dl*-leucyl	83–85	85	$C_{18}H_{26}N_2O_5$	61.70	7.48	8.00	61.52	7.51	8.13
Lauroyl *dl*-leucyl	78–79	75	$C_{24}H_{38}N_2O_5$	66.35	8.75	6.45	66.55	8.93	6.42
Palmitoyl *dl*-leucyl	83–84	75	$C_{28}H_{46}N_2O_5$	68.59	9.38	5.91	68.69	9.58	6.19

[a] All melting points were taken on a Fisher-Johns apparatus.

To a solution of 7 grams *N*-capryl glycine (*4*) (0.031 mole) in 210 ml. of anhydrous ethyl acetate at 5° C., 4.25 grams of *p*-nitrophenol (0.031 mole) and 6.30 grams of dicyclohexyl carbodiimide (0.031 mole) were added. The solution was kept at 5° C. for 30 minutes, then at room temperature for 2 hours, and finally at 35° C. for an additional 2 hours. Several drops of acetic acid were added to destroy any excess dicyclohexyl carbodiimide, and after cooling the solution to 5° C., the urea that formed was filtered off. The ethyl acetate was evaporated, and the ester was crystallized from ether-hexane mixtures. Usually a second recrystallization was necessary to obtain a pure product. The yield was 7.2 grams (66%).

All acyl glycine esters showed absorption in the IR at 5.73, 6.10, 6.20, 6.30 microns and all acyl leucine esters absorbed at 5.72, 6.16, 6.26 and 6.36 microns. IR spectra were determined in nujol with a Perkin Elmer No. 21 Spectrophotometer. Table I lists the new compounds prepared, along with the melting points and elemental analyses.

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