

Acids converted to chlorides, amides or esters

Acid	RNH ₂ /ArOH/ROH	Product ¹⁰	Yield
Acetic	Aniline	Anilide	84%
	p-Aminophenol	p-Hydroxyacetanilide	55%
		p-acetoxyacetanilide	35%
Trifluoroacetic	Aniline	Anilide ¹¹	64%
	L-Valine	N-Trifluoroacetyl-L-valine ¹²	50%
Oxalic	Aniline	Anilide	52%
Malonic	- / Aniline	Chloride or Anilide	0%
Succinic	Aniline	Anilide	55%
Z-glycine	2,4,5-Trichlorophenol	Trichlorophenyl ester ¹³	41%
Z-glycine	Gly-OEt	Z-Gly-Gly-OEt ¹⁴	40%
Z-L-valine	Gly-OEt	Z-L-Val -Gly-OEt ¹⁵	45%
Boc-L-valine	Gly-OEt	Boc-L-Val-Gly-OEt ¹⁶	38%
Phenylacetic	Aniline	Anilide	86%
Benzoic	-	Chloride	81%
	Aniline	Anilide	87%
	o-Phenylene-diamine	N,N-Dibenzoyl-o-phenylenediamine	68%
	o-Aminophenol	N,O-Dibenzoyl-o-aminophenol	73%
	Methanol	Methyl benzoate	82%
p-Nitrobenzoic	-	Chloride	58%
Cinnamic	Aniline	Anilide	93%
Aspirin	Methanol	Methyl ester	45%
3-Hydroxy-2-naphthoic	Aniline	Anilide ¹⁷	61%

solution after the removal of unconverted acid and amine, is then crystallized¹⁴⁻¹⁶. The three peptides and Z-glycine trichlorophenyl ester were characterized by their NMR and mass spectra in addition to their melting points.

The yields recorded in the Table are of the isolated products after purification by acid and base washing, when they were chromatographically homogenous and had mp's about 5°C lower than the literature values, but before the crystallization; the recovery uncovered acid was not taken into account. A simple routine procedure using the regenerated TEA was followed for the preparation of esters and amides, and no attempt was made to optimize conditions for maximum yields.

REFERENCES AND FOOTNOTES

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11. E. J. Bourne *et al.*, **J. Chem. Soc.** **4014** (1952), prepared this compound by treatment of aniline with trifluoroacetic anhydride.
12. C. A. Panetta, **Org. Synth.** **56**, **122** (1977), has described the "trifluoroacetylation of amines and amino acids under neutral, mild conditions: N-trifluoroacetanilide and N-trifluoroacetyl-L-tyrosine" by the action of 1,1,1-trichloro-3,3,3-trifluoroacetone in DMSO at 25-35°C. Panetta has also discussed the disadvantages of the usual procedure using trifluoroacetic anhydride. In the present work a minor modification in our general procedure was to add L-valine in DMSO (10 mL); the product was worked up as described by Panetta.
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17. Separation through carbon tetrachloride before adding aniline showed that the reaction mixture contained the acid, chloride and deposite, cf. E. N. Abrahart, **J. Chem. Soc.** **424** (1938) R. V. Bhat, R. B. Forster and K. Venkataraman, **J. Soc. Dyers Col.**, **56**, **166** (1940). The anilide, which is sparingly soluble in acetone, was taken up in 2 N sodium hydroxide.