

Table 19 Typical Results Showing hR_f Values of Amino Acids on Plain and Impregnated Plates

Amino acid	Solvent I		Solvent II	
	Plain	SO ₄ ²⁻	Plain	SO ₄ ²⁻
Ala	39	47	15	15
Val	65	68	35	67
Leu	63	73	34	73
Ile	63	67	35	64
Pro	30	35	11	10
Ser	25T	32	11T	02
Thr	33	42	15T	07
Cys	16	50	08T	17ST
Met	78	62	25	58
Glu	47	69	39	54
Asp	16	38	02	06
Gln	22	31	04	04
Asn	15	28	07	03
Trp	62	69	40	70
Phe	64	67	40	66
Tyr	45	71	37	64

Solvent I: *n*-Butanol–methanol–acetic acid (8:1:3), solvent front 10 cm in 80 min.

Solvent II: *n*-Butanol–carbon tetrachloride–acetic acid (8:3:1), solvent front 10 cm in 80 min.

T = Tailing; ST = slight tailing.

Detection: Ninhydrin 0.2% in acetone.

Source: Ref. 30f.

procedure (154): L-Alanine (17.8 g) is dissolved in ethanol (200 mL) and 10% palladium on activated charcoal catalyst (3 g), and propionaldehyde (43 mL) is added. The mixture is hydrogenated for 48 h at 40–50°C at an initial hydrogen pressure of 50 psi. The catalyst is removed using a sintered glass filter, and the filtrate is evaporated to dryness. The reaction product (*N,N*-di-*n*-propyl-L-alanine) is crystallized from chloroform, and the purity may be confirmed by TLC, NMR, and C, H, N analysis.

A. Resolution of Enantiomers of Dansyl DL-Amino Acids

Grinberg and Weinstein (155) reported a two-dimensional RP-TLC technique for the resolution of complex mixtures of dansyl amino acids. The Dns derivatives were first separated in a nonchiral mode using 0.3 M sodium acetate in H₂O–acetonitrile (80:20, pH 6.3) to which 0.3 M sodium acetate in H₂O–acetonitrile (70:30) was added to give a final acetonitrile concentration of 38% or 47%. For the second dimension the mobile phase was 8 mM *N,N*-di-*n*-propyl-L-alanine and 4 mM copper(II) acetate dissolved in 0.3 M sodium acetate in H₂O–acetonitrile (70:30, pH 7). The plates were developed in the second dimension using a temperature gradient. The method is reported to be applicable for the resolution of amino acids in a protein hydrolysate and quantification by densitometry.

Alak and Armstrong (156) resolved enantiomers of dansyl amino acids and β -naphthylamide amino acids using β -cyclodextrin (β -CD) plates. The plates were prepared by mixing 1.5 g of β -CD bonded silica gel in 15 mL of 50% methanol (aq.) with 0.002 g of binder (ASTE—all-solvent binder) and acetate in 50/50 MeOH–1% aqueous triethylammonium acetate (pH 4.1). Some of these results are shown in Table 27.