Application of microcrystalline cellulose triacetate mixed with silica gel for TLC resolution of enantiomers of PTH amino acids and N- and C-substituted amino acids has been reported (179a,179b) using 2-propanol or ethanol–water mixtures as mobile phase. The experiments showed that retention of solutes increased as the percentage of alcohol was reduced, in accordance with the general behavior of reversed-phase chromatography, and the analytes with an aromatic group usually resulted in better resolution than those with a nonaromatic group. Further, the compounds with a stereogenic center on a conformationally more rigid substrate (PTH-Phe and PTH-Tyr) were found to resolve efficiently into their enantiomers. The resolution data for these compounds are shown in Table 32.

C. Resolution of DL-Amino Acids

Günther et al. (157) covered the glass plates with silicic acid that had been made hydrophobic (RP 18-TLC), immersed (1 min) in a 0.25% copper(II) acetate solution (MeOH–H_2O, 1:9), dried, and then immersed in a 0.8% methanolic solution of the chiral selector (1 min); the chiral selector was (2*S*,4*R*,2′*RS*)-4-hydroxyl-1-(2′-hydroxydodecyl)proline. Using these plates they reported the resolution of racemic α-amino acids, as shown in Table 33. Using the same approach, described as ligand-exchange TLC, Günther, Martens, and coworkers were able to develop ready-to-use Chiralplate, marketed by Macherey-Nagel (158). Martens et al. (159) and Günther et al. (160) reported the resolution of DL-methyl DOPA, and DL-DOPA on Chiralplate using methanol–H_2O–acetonitrile (50:50:30) as the mobile phase and ninhydrin as the detecting reagent. The R_f values for L-DOPA and D-DOPA were reported to be 0.47 and 0.61, respectively, and the system was capable of resolving enantiomers in trace amounts, with the lowest level of detection for the D enantiomers in L-DOPA samples being 0.25% (160). The resolution of enantiomers of α-substituted α-amino acids (161) and racemic mixtures of natural and nonnatural amino acids, *N*-methylated and *N*-formylated amino acids, and various other derivatives of amino acids (162) has also been reported by Günther et al. (161) on a ready-to-use Chiralplate; typical results are presented in Tables 34 and 35. Enantiomers of unusual aromatic amino acids were analyzed on Macherey-Nagel Chiralplates with MeCN–MeOH–water (4:1:1 or 4:1:2) or MeCN–MeOH–water–diisopropyl ethylamine (4:1:2:0.1) as mobile phase and ninhydrin as detection reagent (162a). TLC on Chiralplates with acetonitrile–methanol–water (4:1:1) mobile phase and ninhydrin detection reagent was used in the quality control of L-tryptophan (162b).

As noted above, the resolution of enantiomers of amino acids and certain of their derivatives was achieved by Martens et al. (28,159) and Günther and coworkers (157,158,160–162), by the immersion technique and ligand exchange. Bhushan (20,22,163,163a) reported resolution of enantiomers of amino acids by mixing the chiral selector with silica gel slurry, e.g., (−)-brucine for the resolution of enantiomers of amino acids (Table 36), and impregnating the silica gel plates with Cu-L-proline complex for the resolution of DL-Phe, -Tyr, -Ile, and -Trp using different solvents (163a). TLC resolution of enantiomers of amino acids and their various derivatives has been

Table 32 Resolution Data for Separation of Enantiomers of PTH Amino Acids and N- and C-Substituted Amino Acids

Compound	Mobile phase	hR_f L	hR_f D
PTH-Phe	Ethanol–water (80:20)	31	36
PTH-Tyr	2-Propanol–water (80:20)	53	64
N-CBZ-Phe-ONp	2-Propanol–water (80:20)	27	24
N-*t*-Boc-Phe-ONp	Methanol–water (80:20)	32	34

Migration distance: 15 or 13 cm; detection at 254 nm.
Source: Ref. 179d.