

TABLE 53

$R_F$  VALUES (RELATIVE) OF SOME HEXITOLS AND THEIR ACETALS  
(T. G. BONNER AND N. M. SAVILLE, *J. Chem. Soc.*, (1960) 2851)

Solvents:  $S_1$  = Butan-1-ol satd. with  $H_2O$  (at  $0^\circ$ ).

$S_2$  = Acetone-water (4:1; v/v).

$S_3$  = Butan-1-ol-pyridine-water-satd. aq. boric acid (6:4:2:1; by vol.).

Paper: Whatman No. 1.

Detection: Reagents as in literature (W. E. TREVELYAN, D. P. PROCTER AND J. S. HARRISON, *Nature*, 166 (1950) 444; M. L. WOLFROM AND J. B. MILLER, *Anal. Chem.*, 28 (1956) 1037; L. HOUGH, J. K. N. JONES AND W. H. WADMAN, *J. Chem. Soc.*, (1950) 1702; K. WALLENFELS, *Naturwiss.*, 37 (1950) 491).

Compound	$R_G^*$ $S_2$	$R_M^{**}$		
		$S_1$	$S_2$	$S_3$
D-Glucitol	1.00			
2,4-O-Methylene-D-glucitol	1.25, 1.36			
D-Mannitol		1.00	1.00	1.00
2,5-O-Methylene-D-mannitol			1.28	2.00
1,3-O-Methylene-D-mannitol		2.66	1.40	
2-O-Methyl-D-mannitol		2.60		1.4

\*  $R_G = R_F$  of compound /  $R_F$  of glucitol.

\*\*  $R_M = R_F$  of compound /  $R_F$  of mannitol.

TABLE 54

$R_F$  VALUES OF SOME PENTAERYTHRITOL ESTERS AND RELATED COMPOUNDS  
(T. G. BONNER, E. J. BOURNE AND N. M. SAVILLE, *J. Chem. Soc.*, (1960) 2917)

Solvents:  $S_1$  = Water satd. butan-1-ol (at  $0^\circ$ ).

$S_2$  = Light petroleum (b.p.  $60-80^\circ$ ) satd. with dimethyl sulphoxide (B. WICKBERG, *Acta Chem. Scand.*, 12 (1958) 615).

Paper: Whatman No. 1.

Impregnation:  $I_1$  = Dip paper in 20% dimethyl sulphoxide in benzene, press between absorbent paper, dry 1 min at  $60^\circ$ . Repeat treatment and place between glass plates to prevent water absorption. Dry at  $120^\circ$  for 20 min before spraying.

Detection:  $D_1$  = Hydroxylamine reagent (M. ABDEL-AKHER AND F. SMITH, *J. Am. Chem. Soc.*, 73 (1951) 5859) [freshly prepared 1:1 v/v mixture of methanolic hydroxylamine hydrochloride (6.95 g/100 ml) and KOH (6.17 g/100 ml)].

Time and temperature of drying depending on ester reactivity, e.g. pentaerythritol tetraacetate is detected only by heating to  $110^\circ$  for 10 min.

Final spraying: 1:1 v/v mixture of aqueous  $FeCl_3$  and aqueous 0.5 N HCl. (Acetates and lactones give purple spots on yellow background, benzoates not detected).

$D_2$  = 2,4-Dinitrophenylhydrazine spray (S. A. BARKER, E. J. BOURNE, A. B. FOSTER AND R. M. PINKHARD, *Chem. & Ind. (London)*, (1959) 226). (Detection of benzylidene and isopropylidene derivatives.)

Compound	$R_F$	
	$S_1$	$S_2 I_1$
Tetra-O-acetylpentaerythritol	0.94	0.46, 0.51
Pentaerythritol	0.45	
Di-O-acetyl-O-benzylidenepentaerythritol		0.66, 0.68, 0.71
Mono-O-acetyl-O-benzylidenepentaerythritol*		0.05

\* Possible identification.

TABLE 55

 $R_F$  VALUES OF SOME POLYHYDRIC ALCOHOLS AND RELATED COMPOUNDS(A. B. FOSTER, D. HORTON, N. SALIM, M. STACEY AND J. M. WEBBER, *J. Chem. Soc.*, (1960) 2587)

Solvents: Butanol-ethanol-water (4:1:5), organic phase.

Paper: Whatman No. 1 (descending).

Detection: Aniline hydrogen phthalate; ninhydrin and  $\text{AgNO}_3$  reagents.

Compound	$R_F$
2-Acetamido-2-deoxy-3-O-methyl-D-glucitol	0.31
Glycerol	0.43
Erythritol	0.315
D-Arabitol	0.225
D-Glucitol	0.145
2-Amino-2-deoxyglycerol·HCl	0.17
2-Amino-2-deoxy-L-threitol·HCl	0.14
2-Amino-2-deoxy-L-xylitol·HCl	0.12
2-Amino-2-deoxy-D-glucitol·HCl	0.097

TABLE 56

 $R_F$  VALUES (RELATIVE) OF SOME ACYCLIC POLYHYDROXY COMPOUNDS AND THEIR PRE-FORMED MOLYBDATE COMPLEXES(E. J. BOURNE, D. H. HUTSON AND H. WEIGEL, *J. Chem. Soc.*, (1961) 35)

Solvent: Butan-1-ol-acetic acid-water (4:1:5), organic phase.

Paper: Whatman No. 1.

Detection:  $D_1$  = Acetone-silver nitrate-alcoholic sodium hydroxide (W. E. TREVELYAN, D. P. PROCTER AND J. S. HARRISON, *Nature*, 166 (1950) 444); for polyhydroxy compounds. $D_2$  = 5% aqueous catechol solution (J. B. PRIDHAM, *J. Chromatog.*, 2 (1959) 605); for molybdate-containing compounds.Preparation of pre-formed complexes: aqueous polyhydroxy compound (1 mol.) mixed with aqueous sodium molybdate (2 mol.), pH adjusted to 2 with IR-120 ( $\text{H}^+$ ).

Original compound	$R_G$ Untreated compound	$R_G$			
		Pre-formed complexes			
Sorbitol	1.0	0.9*	0.6*†	0.5*†	0†
2-Deoxysorbitol	1.2	1.2*	0.7*†	0.4*†	0†
D-Mannitol	1.1	0.9*	0.7*†	0.5*†	0†
Galactitol	1.1	1.1*	0.7*†	0.5*†	0†
D-Arabitol	1.1	1.1*	0.7*†	0.4*†	0†
Erythritol	1.7	1.7*	1.2*†	—	0†
Glycerol	2.2	2.2*	—	—	0†
Molybdic acid	—	—	—	—	0†

 $R_G = R_F$  compound/ $R_F$  glucose.\*  $D_1$ .†  $D_2$ .

TABLE 57

## ELECTROPHORETIC MOBILITIES OF ACYCLIC POLYHYDROXY COMPOUNDS

(E. J. BOURNE, D. H. HUTSON AND H. WEIGEL, *J. Chem. Soc.*, (1961) 35)Electrolyte: Hydrated sodium molybdate in water (25 g in 1200 ml) adjusted to pH 5 with H<sub>2</sub>SO<sub>4</sub>.  
Paper: Whatman No. 3 MM (10 cm wide).Apparatus: High voltage; horizontal (D. GROSS, *Chem. & Ind. (London)*, (1959) 1219).

Potential: 20–80 V/cm (not more closely defined).

Time of run: 1–2 h (not more closely defined).

Units:  $M_S = \frac{\text{True distance migrated by compound}}{\text{True distance migrated by sorbitol}}$ Detection: Acetone–silver nitrate–alcoholic sodium hydroxide (W. E. TREVELYAN, D. P. PROCTER AND J. S. HARRISON, *Nature*, 166 (1950) 444); 0.1 N H<sub>2</sub>SO<sub>4</sub> spray followed by heating (120°; 10 min) for primary hydroxyl-containing compounds (bluish green spots).

Compound	MS	Compound	MS
Ethane-1,2-diol	<0.1	2,3-Di-O-methylsorbitol	<0.1
Propane-1,2-diol	<0.1	4-O- $\alpha$ -D-Glucopyranosylsorbitol	0.4
Propane-1,3-diol	<0.1	4-O- $\beta$ -D-Glucopyranosylsorbitol	0.4
Butane-2,3-diol	<0.1	4-O- $\beta$ -D-Galactopyranosylsorbitol	0.4
Butane-1,3-diol	<0.1	4-O- $\alpha$ -Isomaltosylsorbitol	0.4
Butane-1,4-diol	<0.1	4-O- $\alpha$ -Nigerosylsorbitol	0.4
Pentane-1,5-diol	<0.1	5-O- $\alpha$ -D-Glucopyranosylsorbitol	0.8
Hexane-1,6-diol	<0.1	6-O- $\alpha$ -D-Glucopyranosylsorbitol	0.8
2-Methylpentane-2,4-diol	<0.1	6-O- $\beta$ -D-Glucopyranosylsorbitol	0.8
2-Methylhexane-1,3-diol	<0.1	6-O- $\alpha$ -D-Galactopyranosylsorbitol	0.8
Pentaerythritol	<0.1	6-O- $\alpha$ -Isomaltosylsorbitol	0.7
Glycerol	<0.1	6-O- $\alpha$ -Isomaltotriosylsorbitol	0.6
Erythritol	1.0	6-O- $\alpha$ -Isomaltotetraosylsorbitol	0.5
D-Threitol	0.5	6-O- $\alpha$ -Isomaltopentaosylsorbitol	0.4
Ribitol	1.1	6-O- $\alpha$ -Isomaltohexaosylsorbitol	0.3
D-Arabitol	1.1	6-O- $\alpha$ -Isomaltoheptaosylsorbitol	0.25
3-O- $\alpha$ -D-Galactopyranosyl-D-arabitol	<0.1	D-Mannitol	1.0
Xylitol	1.1	1-Deoxy-D-mannitol	1.0
Sorbitol	1.0	2-O-Methyl-D-mannitol	1.0
2-Deoxysorbitol	1.0	2-O- $\alpha$ -D-Glucopyranosyl-D-mannitol	0.8
2-O- $\beta$ -D-Glucopyranosylsorbitol	0.9	2-O- $\alpha$ -D-Mannopyranosyl-D-mannitol	0.8
2-O-Methylsorbitol	<0.1	3-O- $\alpha$ -Mannopyranosyl-D-mannitol	<0.1
3-O- $\alpha$ -D-Glucopyranosylsorbitol	<0.1	1,2-Di-O-methyl-D-mannitol	1.0
3-O- $\beta$ -D-Glucopyranosylsorbitol	<0.1	Galactitol	1.0
3-O- $\alpha$ -Maltosylsorbitol	<0.1	6-Deoxy-D-galactitol	1.0

TABLE 58

## ELECTROPHORETIC MOBILITIES OF POLYHYDROXY COMPOUNDS

(E. J. BOURNE, D. H. HUTSON AND H. WEIGEL, *J. Chem. Soc.*, (1960) 4252)Electrolyte: Aq. sodium molybdate dihydrate (25 g/1200 ml), adjusted to pH 5 with conc. H<sub>2</sub>SO<sub>4</sub>.  
Paper: Whatman No. 3 MM (10 cm wide).

Apparatus: Gross type (max. 5000 V at 100 mA); horizontal, cooled.

Potential: 30–60 V/cm.

Time: 1–2 h.

Units:  $M_S$  = Migration relative to sorbitol (ca. 25 cm in 2 h at 60 V/cm).

Marker: Glycerol (comparison with 2,3,4,6-tetra-O-methyl-D-glucose showed that it did not form a complex; used to correct for electroendosmosis).

Detection: Acetone–silver nitrate–ethanolic sodium hydroxide (W. E. TREVELYAN, D. P. PROCTER AND J. S. HARRISON, *Nature*, 166 (1950) 444); *p*-anisidine hydrochloride in butan-1-ol (L. HOUGH, J. K. N. JONES AND W. H. WADMAN, *J. Chem. Soc.*, (1950) 1702).

<i>Compound</i>	<i>M<sub>S</sub></i>	<i>Compound</i>	<i>M<sub>S</sub></i>
<i>Complex forming</i>			
<i>Aldoses and their derivatives</i>		<i>Cyclitols</i>	
D-Erythrose	0.9	Myoinositol	0.2
L-Threose	0.6	Alloinositol	0.4
D-Ribose	0.4	Epi-inositol	1.1
Methyl α-D-ribofuranoside	0.1		
D-Lyxose	1.1	<i>1,6-Anhydro-β-D-aldopyranoses</i>	
D-Mannose	0 → 0.9	1,6-Anhydro-β-D-mannopyranose	0.5
D-Gulose	1.1		
D-Talose	0.7	<i>Ketoses</i>	
L-Rhamnose	0 → 0.6	D-Fructose	0.5
D-Glycero-D-alloheptose	0.9	L-Sorbose	0.3
D-Glycero-L-glucoheptose	0.2	D-Glucosone	0.9
D-Glycero-L-mannoheptose	0.8	D-Glucoheptulose	1.0
D-Glycero-D-guloheptose	1.1	D-Mannoheptulose	0.4
D-Glycero-D-idoheptose	1.0	Leucrose	0.4
D-Glycero-D-galaheptose	0.4	Turanose	0.1
D-Glycero-L-galaheptose	0.4		
<i>Not complex forming</i>			
<i>Aldoses and their derivatives</i>		<i>Aldoses and their derivatives</i>	
Glyceraldehyde	< 0.1	Methyl α-D-glucopyranoside	< 0.1
2-Deoxy-D-ribose	< 0.1	Phenyl β-D-glucopyranoside	< 0.1
D-Arabinose	< 0.1	Catechol β-D-glucopyranoside	< 0.1
Methyl α-D-arabofuranoside	< 0.1	3,4-Di-O-methyl-D-mannose	< 0.1
Methyl β-D-arabofuranoside	< 0.1	Methyl α-D-mannopyranoside	< 0.1
1,2-Dideoxy-D-arabinose	< 0.1	Methyl β-D-mannopyranoside	< 0.1
D-Xylose	< 0.1	D-Galactose	< 0.1
Methyl α-D-xylofuranoside	< 0.1	Sophorose	< 0.1
Methyl α-D-lyxopyranoside	< 0.1	Nigerose	< 0.1
Methyl β-D-lyxopyranoside	< 0.1	Laminariabiose	< 0.1
D-Altrose	< 0.1	Maltose	< 0.1
D-Glucose	< 0.1	Cellobiose	< 0.1
3-O-Methyl-D-glucose	< 0.1	Lactose	< 0.1
2,3,4-Tri-O-methyl-D-glucose	< 0.1	Isomaltose	< 0.1
2,3,6-Tri-O-methyl-D-glucose	< 0.1	Gentiobiose	< 0.1
2,3,4,6-Tetra-O-methyl-D-glucose	< 0.1	Melibiose	< 0.1
2-Deoxy-D-glucose	< 0.1		

(Continued on p. D37)

TABLE 58 (continued)

Compound	$M_S$	Compound	$M_S$
<i>Not complex forming</i>			
<i>Cyclitols</i>		<i>1,6-Anhydro-<math>\beta</math>-D-aldopyranoses</i>	
Mucoinositol	< 0.1	1,6-Anhydro- $\beta$ -D-glucopyranose	< 0.1
Scylloinositol	< 0.1	1,6-Anhydro- $\beta$ -D-gulopyranose	< 0.1
Mytilitol	< 0.1	1,6-Anhydro- $\beta$ -D-galactopyranose	< 0.1
Pinitol	< 0.1		
Quebrachitol	< 0.1	<i>Ketose</i>	
(-)-Viboquercitol	< 0.1	Sucrose	< 0.1
Scylloquercitol	< 0.1		
(-)-Protoquercitol	< 0.1		

TABLE 59

## ELECTROPHORETIC MOBILITIES OF SOME MONOMETHYLGLUCOSES

(T. G. BONNER, E. J. BOURNE AND S. McNALLY, *J. Chem. Soc.*, (1960) 2929)Electrolyte: 0.2 *M* borate buffer (pH 10.0).

Paper: Whatman No. 3.

Apparatus: Not given\*.

Potential: Not given\*.

Units:  $M_G^*$ .

Detection:  $\text{AgNO}_3$  and ethanolic NaOH; *p*-anisidine·HCl; aniline hydrogen phthalate; urea hydrochloride; diphenylamine, aniline and phosphoric acid; 2,4-dinitrophenylhydrazine and HCl;  $\alpha$ -naphthol and phosphoric acid; phloroglucinol and trichloroacetic acid; potassium periodatocuprate (T. G. BONNER, *Chem. & Ind. (London)*, (1960) 345).

Compound	$M_G^*$
2-O-Methylglucose	0.23
3-O-Methylglucose	0.82
4-O-Methylglucose	0.24
6-O-Methylglucose	0.82

\* Cf. A. B. FOSTER, *J. Chem. Soc.*, (1953) 982.

TABLE 60

 $R_F$  VALUES OF SOME METHYLATED SUGARS(P. D. BRAGG AND L. HOUGH, *Biochem. J.*, 78 (1961) 11)

Solvent: Butan-1-ol-ethanol-water (40:11:19, by vol.).

Paper: Whatman No. 1 (descending).

Detection: *p*-Anisidine hydrochloride (L. HOUGH, J. K. N. JONES AND W. H. WADMAN, *J. Chem. Soc.*, (1950) 1702).

Compound	$R_F$
2,6-Di-O-methyl-D-mannose*	0.54-0.57
Mono-O-methylmannose**	0.36
2,3,4,6-Tetra-O-methyl-D-galactose	0.70-0.74
Tri-O-methylhexose**	0.61-0.63
2,3,4,6-Tetra-O-methyl-D-mannose	0.81-0.82
Tri-O-methylmannose**	0.71

\* Tentative identification.

\*\* Not identified closer.

TABLE 61

 $R_G$  VALUES (RELATIVE) OF SUGARS AND METHYLATED SUGARS(G. O. ASPINALL, M. J. JOHNSTON AND A. M. STEPHEN, *J. Chem. Soc.*, (1960) 4918)Solvents:  $S_1$  = Butan-1-ol-ethanol-water (4:1:5, upper layer). $S_2$  = Benzene-ethanol-water (169:47:15, upper layer). $S_3$  = Ethyl acetate-acetic acid-formic acid-water (18:3:1:4).

Paper: Whatman Nos. 1, 4 and 20 (not specified).

Detection: Spray of *p*-anisidine hydrochloride in moist butan-1-ol (L. HOUGH, J. K. N. JONES AND W. H. WADMAN, *J. Chem. Soc.*, (1950) 1702).

Compound	$R_G^*$		
	$S_1$	$S_2$	$S_3$
Mono-O-methylrhamnose	0.55, 0.58, 0.57		
2,3,4,6-Tetra-O-methyl-D-galactose	0.86, 0.89	0.87	
3,4-Di-O-methyl-L-rhamnose	0.86	0.25	
2,3,6-Tri-O-methyl-D-galactose	0.69, 0.70, 0.72	0.71	0.25
2,3,6-Tri-O-methyl-D-galactonolactone	1.00		
2,3,4-Tri-O-methyl-D-galactose	0.68, 0.65		
2,3,4-Tri-O-methyl-L-lyxose**	0.82		
2,3,4-Tri-O-methyl-L-rhamnose	1.00		
2,3,5-Tri-O-methyl-L-arabinose	0.98	1.02	
2,3-Di-O-methyl-L-threose**	0.92	0.90	
3-O-Methyl-L-rhamnose	0.58, 0.57, 0.55		
2,6-Di-O-methyl-D-galactose	0.49, 0.50, 0.48		
2,3-Di-O-methyl-D-galactose	0.45, 0.46, 0.48		
2,4-Di-O-methyl-D-galactose	0.43, 0.49, 0.44		
2,3,4-Tri-O-methyl-D-glucose	0.87, 0.88, 0.81	0.20	
2,3,4-Tri-O-methyl-L-xylose***	0.92	0.86	
3,4-Di-O-methyl-D-mannose		0.07	
Rhamnose	0.30		
2,3,4,6-Tetra-O-methyl-D-glucose	1.00		
2-O-Methyl-D-galactose	0.29		

\*  $R_G = R_F$  compound/ $R_F$  of 2,3,4,6-tetra-O-methyl-D-glucose.

\*\* Tentative identification.

\*\*\* Presumed.

TABLE 62

 $R_F$  VALUES (RELATIVE) OF SOME OLIGOSACCHARIDES AND METHYL SUGARS(G. O. ASPINALL, I. M. CAIRNCROSS, R. J. STURGEON AND K. C. B. WILKIE, *J. Chem. Soc.*, (1960) 3881)Solvents:  $S_1$  = Ethyl acetate-pyridine-water (10:4:3). $S_2$  = Butan-1-ol-ethanol-water (4:1:5, upper layer).

Paper: Whatman No. 1 or 3 MM (not specified).

Detection: Not given.

Compound	$R_{Xylose}$ *	
	$S_1$	$S_2$
Xylobiose	0.60	
Xylotriose	0.30	
Xyloetraose	0.14	
O-L-Arabinofuranosyl-(1 → 3)-O- $\beta$ - D-xylopyranosyl-(1 → 4)-D-xylose	0.40	
2,3,5-Tri-O-methyl-L-arabinose		0.96
2,4-Di-O-methyl-D-xylose		0.70

\*  $R_{Xylose}$  =  $R_F$  compound/ $R_F$  xylose.\*\*  $R_G$ : Not defined.

TABLE 63

 $R_F$  VALUES OF SOME METHYL GLYCOSIDE PHOSPHATES(P. SZABÓ AND L. SZABÓ, *J. Chem. Soc.*, (1960) 3762)Solvents:  $S_1$  = Propan-2-ol-ammonia-water (7:1:2). $S_2$  = Propan-1-ol-ammonia-water (7:1:2). $S_3$  = Butan-1-ol-acetic acid-water (4:1:5). $S_4$  = Propan-2-ol-conc. HCl-water (65:17.2:17.8). $S_5$  = Propan-1-ol-ammonia-water (6:1:3).

Paper: Whatman No. 1 (ascending).

Detection: Not specified.

Compound	$R_F$				
	$S_1$	$S_2$	$S_3$	$S_4$	$S_5$
Me $\alpha$ -D-glucoside 2-(dihydrogen phosphate)	0.29	0.18	0.15	—	0.48
Me $\alpha$ -D-glucoside 3-(dihydrogen phosphate)	0.34	0.20	0.19	—	0.50
Me $\alpha$ -D-glucoside 4-(dihydrogen phosphate)	0.32	0.18	0.15	—	0.45
Me $\alpha$ -D-glucoside 6-(dihydrogen phosphate)	0.26	0.13	—	—	0.41
Me $\beta$ -D-galactoside 4-(dihydrogen phosphate)	—	0.21	0.17	—	0.45
Me $\beta$ -D-galactoside 6-(dihydrogen phosphate)	—	0.11	0.09	—	0.36
Me $\alpha$ -D-glucoside 4,6-(hydrogen phosphate)	0.64	0.47	0.16	0.90	—
Me $\alpha$ -D-glucoside 4,6-(hydrogen phosphate)	—	0.47	0.16	—	—
Me $\beta$ -D-galactoside 4,6-(hydrogen phosphate)	0.57	0.38	0.12	0.86	—

TABLE 64

$R_F$  VALUES OF 1,2-O-ISOPROPYLIDENE-D-GLUCOFURANOSE PHOSPHATES  
(P. SZABÓ AND L. SZABÓ, *J. Chem. Soc.*, (1961) 448)

Solvents:  $S_1$  = Propan-2-ol-ammonia-water (7:1:2).

$S_2$  = Propan-2-ol-ammonia-water (8:1:1).

Paper: Whatman No. 1 (ascending).

Detection: Not given.

Phosphate of 1,2-O-isopropylidene D-glucofuranose	$R_F$	
	$S_1$	$S_2$
3-Phosphate	0.42	0.16
3,5-Phosphate	0.77	0.67, 0.60
3,6-Phosphate	0.72	0.60, 0.53
5-Phosphate	0.49	0.20
6-Phosphate	0.38	0.11

TABLE 65

$R_F$  VALUES (RELATIVE) OF TRIPHENYLMETHYL ETHERS OF CERTAIN CARBOHYDRATES  
(D. A. APPELGARTH AND J. G. BUCHANAN, *J. Chem. Soc.*, (1960) 4706).

Solvent: Di-isopropyl ether.

Paper: Whatman No. 1 (descending).

Impregnation: Paper dipped twice in 20% v/v solution of dimethyl sulphoxide in benzene and drying at 60° for 90 sec each time. Dimethyl sulphoxide removed after run by heating at 75° for 25 min (B. WICKBERG, *Acta. Chem. Scand.*, 12 (1958) 615).

Detection:  $D_1$  = Spray of approx. *N* perchloric acid; heat at 75° for 5 min (10<sup>-5</sup>g triphenylmethanol). Colour (yellow) fades on cooling, restored on heating (for triphenylmethyl group).

$D_2$  = Removal of triphenylmethyl group. Dip paper in ethereal formic acid solution (25% v/v of 98% formic acid). Heat at 100° for 10 min. Place in forced draught at room temp. for 1 h. Alkaline silver nitrate (W. E. TREVELYAN, D. P. PROCTER AND J. S. HARRISON, *Nature*, 166 (1950) 444) or periodate-Schiff's reagent (J. BADDILEY, J. G. BUCHANAN AND B. CARSS, *J. Chem. Soc.*, (1957) 4138) (for polyols and similar compounds).

Compound	$R_T^*$
2,3,4-Tri-O-acetyl-1,5-di-O-triphenylmethylribitol	1.7
Methyl 3,4-anhydro-6-O-triphenylmethyl- $\alpha$ -D-galactoside	0.50
Methyl 2,3-anhydro-6-O-triphenylmethyl- $\alpha$ -D-guloside	0.31
1,5-Di-O-triphenylmethylribitol	0.28
1-O-Triphenylmethylribitol	0.00

\*  $R_T = R_F$  compound/ $R_F$  triphenylmethanol.

$R_F$  of triphenylmethanol varied from 0.50 to 0.75 but rates of movement relative to triphenylmethanol were fairly constant.



TABLE 66

 $R_F$  VALUES OF PIPECOLIC ACIDS AND RELATED COMPOUNDS(J. W. CLARK-LEWIS AND P. I. MORTIMER, *J. Chem. Soc.*, (1961) 189)Solvents:  $S_1$  = Butan-1-ol-acetic acid-water (4:1:5; S. M. PARTRIDGE, *Biochem. J.*, 42 (1948) 238). $S_2$  = Butan-1-ol-benzyl alcohol (1:1). $S_3$  = Phenol-water; ammonia and HCN atmosphere (R. CONSDEN, A. H. GORDON AND A. J. P. MARTIN, *Biochem. J.*, 38 (1944) 224).

Paper: Not specified.

Time of run:  $T_1$  = 12 h;  $T_2$  = 16 h;  $T_3$  = 15 h;  $T_4$  = 40 h;  $T_5$  = 24 h;  $T_6$  = 19 h;  $T_7$  = 40 h;  $T_8$  = 44 h;  $T_9$  = 20 h;  $T_{10}$  = 28 h;  $T_{11}$  = 48 h.Length of run:  $L_1$  = 24 cm;  $L_2$  = 37 cm;  $L_3$  = 39 cm;  $L_4$  = 27.4 cm;  $L_5$  = 45 cm;  $L_6$  = 48 cm.Impregnation:  $I_1$  = *M*/15 phosphate buffer (pH 7.5; E. F. MCFARREN, *Anal. Chem.*, 23 (1951) 168; L. F. BURROUGHS, *J. Sci. Food Agric.*, 8 (1957) 122).Detection:  $D_1$  = Ninhydrin at 100°. $D_2$  = Isatin at 100°. $D_3$  = Cold neutral silver nitrate solution. $D_4$  = U.V. light fluorescence.

Compound	$R_F$		$R_{\pi}^*$		Colour**			
	$S_1$	$S_3$	$S_1$	$S_2I_1$	$D_1$	$D_2$	$D_3$	$D_4$
Pipecolic acid	0.36 <sup>a,1</sup> , 0.37 <sup>b</sup>				brblv	gn		
	0.33 <sup>e</sup> , 0.35 <sup>d</sup>	0.33 <sup>d</sup> , 0.27 <sup>e</sup>	1.00 <sup>g</sup>	1.00 <sup>f</sup>	( $S_2$ )	( $S_2$ )		br
<i>cis</i> -4-Hydroxy-pipecolic acid	0.16 <sup>a</sup> , 0.20 <sup>a,1</sup>	0.55 <sup>d</sup> , 0.47 <sup>e</sup>	0.56 <sup>g</sup>	0.47 <sup>g</sup>	grgn			
<i>trans</i> -4-Hydroxy-pipecolic acid					-blp			
4-Iodopipecolic acids	0.52 <sup>a</sup> , 0.58 <sup>a</sup>	—	—	—	grbn ( $S_2$ )	gn		
2-Aminopent-4-enoic acid	0.37 <sup>e</sup>	—	0.99 <sup>h</sup>	0.59 <sup>f</sup>	p	blgn,	bn	
Baikiaian	0.28 <sup>e</sup> , 0.31 <sup>e</sup>	—	0.86 <sup>h</sup>	0.82 <sup>f</sup>	bn ( $S_2$ )	fapi		
Proline	—	—	—	0.46 <sup>f</sup>	p <sup>1</sup>	w ( $S_2$ )		
5-Hydroxypipicolinic acid	—	0.53 <sup>d</sup> , 0.45 <sup>e</sup>	0.58 <sup>k</sup>	0.27 <sup>f</sup>	ygn <sup>2</sup>	p-pi		
2-Aminopentanoic acid (norvaline)	0.45 <sup>1</sup> , 0.49 <sup>e</sup>	—	—	—	bry ( $S_2$ )	grgn,	pi	
Valine	0.39 <sup>1</sup>	—	—	—	bkr	( $S_2$ )		
$\alpha$ -Aminobutyric acid	0.28 <sup>d</sup>	—	—	—	bl	( $S_2$ )		
3-Hydroxypyrrolidin-2-yl-acetic acid	0.27 <sup>n</sup>	—	—	—	yo	gn		na
Leucine	0.54 <sup>d</sup>	—	—	—				
Isoleucine	0.54 <sup>d</sup>	—	—	—				
$\beta$ -Alanine	0.24 <sup>d</sup>	—	—	—				
<i>cis</i> -3-Hydroxypipicolinic acid	—	0.24 <sup>e</sup>	0.42 <sup>k</sup>	0.52 <sup>m</sup>				

\*  $R_{\pi}$  = Distance moved by compound/distance moved by pipecolic acid.

\*\* bkr = brick red; bl = blue; bn = brown; br = bright; du = dull; egr = edges grey; fa = faint; gn = green; gr = grey; na = no absorption; o = orange; p = purple; pi = pink; r = red; v = violet; w = white; y = yellow.

a =  $L_2T_2$ ; b =  $L_1T_1$ ; c =  $L_3T_3$ ; d =  $L_4T_1$ ; e =  $L_6T_5$ ; f =  $T_7$ ; g =  $T_4$ ; h =  $T_8$ ; i =  $T_9$ ; j =  $L_1T_6$ ; k =  $T_{10}$ ; m =  $T_{11}$ ; n =  $L_6T_6$ ; l = 110-115°; 2 = 80-90°.

TABLE 67

$R_F$  VALUES OF AMINO ACIDS AND RELATED COMPOUNDS FROM OSTROEGRYCIN  
(F. W. EASTWOOD, B. K. SNELL AND A. TODD, *J. Chem. Soc.*, (1960) 2286)

Solvents:  $S_1$  = Phenol-water (4:1) in presence of HCN.  
 $S_2$  = 2,6-Lutidine-collidine-water (1:1:1) containing 1% diethylamine.  
 $S_3, S_4, S_5$  = described by T. L. HARDY, D. O. HOLLAND AND J. H. C. NAYLER (*Anal. Chem.*, 27 (1955) 971) as I, L, O.  
 $S_6$  = Butanol-acetic acid-water (4:1:5).  
 $S_7$  = Decalin-acetic acid-isopentanol (15:10:2) (W. GRASSMANN, H. HÖRMANN AND H. ENDRES, *Chem. Ber.*, 86 (1953) 1477).  
 $S_8$  = Decalin-10% acetic acid-isopentanol (15:10:4) (as for  $S_7$ ).  
 $S_9$  = *tert.*-Pentyl alcohol-pH 6 phthalate buffer (S. BLACKBURN AND A. G. LOWTHER, *Biochem. J.*, 48 (1951) 126).  
 Paper:  $P_1$  = Whatman No. 1.  
 $P_2$  = Whatman No. 4.  
 Detection:  $D_1$  = Ninhydrin reagent.  
 $D_2$  = *p*-Nitrobenzoyl chloride-pyridine reagent (J. C. SHEEHAN, H. G. ZACHAU AND W. B. LAWSON, *J. Am. Chem. Soc.*, 80 (1958) 3349).  
 $D_3$  = U.V. light.  
 $D_4$  = Periodate-anisidine reagent (P. D. BRAGG AND L. HOUGH, *J. Chem. Soc.*, (1958) 4050).

Compound	$R_F$										Colour*			
	$S_1P_1$	$S_2P_1$	$S_3P_1$	$S_4P_1$	$S_5P_1$	$S_6P_1$	$S_7P_1$	$S_8P_1$	$S_9P_1$	$S_9P_2$	$D_1$	$D_2$	$D_3$	$D_4$
Threonine	0.45	0.24									p			
$\alpha$ -Aminobutyric acid	0.62	0.31									p			
4-Oxopipicolinic acid	0.52	0.43									o-r			
Proline	0.81	0.30									y			
Phenylglycine	0.73	0.52									p	r		
DL- <i>p</i> -Dimethylamino-N-methyl-phenylalanine	0.93	0.75	0.76	0.81	0.77	0.30					bn**	r		
3-Hydroxypicolinic acid	0.79	0.91									bn→p			+
DL-2-Amino-2-phenyl-ethanol														
2-(2,4-Dinitrophenylamino)-2-phenyl-ethanol										0.27	0.86			
Tetrapeptide***											0.34			fe
Pentapeptide†											0.77			fe

\* p = purple; o = orange; r = red; y = yellow; bn = brown; fe = fluorescence; + = positive result; --- = negative result.  
 \*\* Development above 80°.

\*\*\* Containing 3-hydroxypicolinic acid,  $\alpha$ -aminobutyric acid, proline and threonine.

† Containing 3-hydroxypicolinic acid,  $\alpha$ -aminobutyric acid, proline, threonine and *p*-dimethylamino-N-methylphenylalanine.

TABLE 68

$R_F$  VALUES OF GLUCOSAMINE-1-PHOSPHATE AND N-ACETYLGLUCOSAMINE-1-PHOSPHATE  
(G. BALUJA, B. H. CHASE, G. W. KENNER AND A. TODD, *J. Chem. Soc.*, (1960) 4678)

Solvents:  $S_1$  = Propan-2-ol-1%  $(NH_4)_2SO_4$ .  
 $S_2$  = Propan-1-ol-ammonia-water (6:3:1, v/v).  
 $S_3$  = 95% ethanol-*M* ammonium acetate (75:30).  
 Paper: Not given (ascending).  
 Impregnation:  $I_1$  = 1%  $(NH_4)_2SO_4$ , then dried.  
 Detection: Not given.

Compound	$R_F$		
	$S_1I_1$	$S_2$	$S_3$
Glucosamine-1-phosphate	0.26	0.15	0.09
N-Acetylglucosamine-1-phosphate	0.49	0.22	0.19

TABLE 69

$R_F$  VALUES OF SOME LANTHIONINE DERIVATIVES AND RELATED COMPOUNDS  
(M. FRANKEL AND D. GERTNER, *J. Chem. Soc.*, (1961) 459, 463)

Solvent: 80% aqueous phenol.  
 Paper: Not given.  
 Detection: Not given.

Compound	$R_F$
N,N'-Dibenzoyloxycarbonyl-meso-lanthionine diethyl ester	0.95
N-Benzoyloxycarbonyl-lanthionine monobenzyl ester	0.90
N-Acetyl-lanthionine monomethyl ester	0.84-0.86
S-Benzoyloxycarbonyl-DL-homocysteine	0.92

TABLE 70

$R_F$  VALUES OF SOME L-LYSINE PEPTIDES  
(B. BEZAS AND L. ZERVAS, *J. Am. Chem. Soc.*, 83 (1961) 719)

Solvent: Butan-1-ol-acetic acid-water-pyridine (Y. LEVIN, A. BERGER AND E. KATCHALSKI,  
*Biochem. J.*, 63 (1956) 308).  
 Paper: Whatman No. 1 (ascending).  
 Detection: Ninhydrin.

Compound	$R_F$
N <sup>ε</sup> -Glycyl-L-lysine	0.18
N <sup>ε</sup> -L-Valyl-L-lysine	0.36
N <sup>ε</sup> -L-Phenylalanyl-L-lysine	0.46
N <sup>ε</sup> -L-Phenylalanyl-L-lysyl-L-tyrosine	0.82
N <sup>α</sup> -L-Phenylalanyl-L-lysine	0.46
N <sup>α</sup> -L-Phenylalanyl-N <sup>ε</sup> -L-valyl-L-lysine	0.62

TABLE 71

$R_F$  VALUES OF SOME ACETYL-SERINE PEPTIDES  
(L. BENOITON AND H. N. RYDON, *J. Chem. Soc.*, (1960) 3328)

Solvents:  $S_1$  = *tert.*-Butanol-formic acid-water (70:15:15).

$S_2$  = *n*-Butanol-acetic acid-water (4:1:5).

$S_3$  = Phenol saturated with 10% sodium citrate.

Paper: Whatman No. 1.

Detection:  $D_1$  = Ninhydrin reagent.

$D_2$  = Chlorine-starch-iodide procedure (H. N. RYDON AND P. W. G. SMITH, *Nature*, 169 (1952) 922).

Compound	$R_F$		
	$S_1$	$S_2$	$S_3$
DL-Seryl-glycyl-glycine	0.28	0.22	0.43
O-Acetyl-DL-seryl-glycyl-glycine	0.40	0.32	0.65
N-Acetyl-DL-seryl-glycyl-glycine	0.51	0.40	0.73
$\alpha$ -L-Aspartyl-O-acetyl-L-seryl-glycyl-glycine	0.32	0.25	0.49
O- $\alpha$ -L-Aspartyl-N-acetyl-L-seryl-glycyl-glycine	0.32	0.24	0.49

TABLE 72

$R_F$  VALUES OF SOME PEPTIDES  
(D. T. GISH, *J. Am. Chem. Soc.*, 82 (1960) 6329)

Solvent: Butan-1-ol-acetic acid-water-pyridine (30:6:24:20) (S. G. WALEY AND J. WATSON, *Biochem. J.*, 55 (1953) 328).

Paper: Whatman No. 3 MM.

Detection:  $D_1$  = Ninhydrin reagent.

$D_2$  = *p*-Dimethylaminobenzaldehyde reagent (I. SMITH, *Nature*, 171 (1953) 43).

$D_3$  = Sakaguchi reagent (R. ACHER AND C. CROCKER, *Biochim. Biophys. Acta.*, 9 (1952) 704).

$D_4$  = Hypochlorite-starch-KI test (S. C. PAN AND J. D. DUTCHER, *Anal. Chem.*, 38 (1956) 836).

$D_5$  = Chlorine-starch-KI test (H. N. RYDON AND P. W. G. SMITH, *Nature*, 169 (1952) 922).

Peptide	$R_F$	Colour*				
		$D_1$	$D_2$	$D_3$	$D_4$	$D_5$
Lys·Pro·Ser·Pro·GluNH <sub>2</sub>	0.18	+	—	—		
Lys·Pro·Ser·Pro·GluNH <sub>2</sub> ·Val·Thr	0.27	+	—	—		
Val·Arg	0.40	+	—	+		
Val·Thr·Val·Arg	0.51	+	—	+		
Ser·GluNH <sub>2</sub> ·Val·Try	0.65	y	+	—		
Pyroglu·Phe	0.65	y	+	—	+	
Pyroglu·Phe·Ser·GluNH <sub>2</sub> ·Val·Try	0.90	—	+	—		
Phe	0.61	+				—
Pyroglu	0.43	—				+

\* y = yellow; + = positive; — = negative.

TABLE 73

 $R_F$  VALUES (RELATIVE) OF SOME PEPTIDES(A. WITTER AND H. TUPPY, *Biochim. Biophys. Acta*, 45 (1960) 429)Solvent: *n*-Butanol-pyridine-acetic acid-water (4:1:1:5, by vol.).

Paper: Schleicher &amp; Schüll 2045 BM.

Detection: Ninhydrin.

Compound*	$R_{Phe}$ **	Colour***
Cys-Pro	0.31	pu
Pro-Phe	1.21	y
Glu-Cys-Pro	0.08	v
Glu-Glu-Cys-Pro	0.13	v
Glu-Cys-Pro-Phe	0.22	v
Cys-Pro-Phe	0.37	p
Glu-Glu-Cys	0.05	v
Glu-Cys	0.08	pu
Glu-Glu	0.13	pu
Phe	1.00	v
Glu	0.19	v
Pro	0.43*	y
2-Amino-2-carboxyethyl- mercaptosuccinic acid	0.08	

\* Inferred sequence where peptide.

\*\*  $R_F$  relative to that of phenylalanine.

\*\*\* pu = purple; y = yellow; v = violet; p = pink.

TABLE 74

 $R_F$  VALUES OF SOME PEPTIDES AND PEPTIDE DERIVATIVES(D. THEODOROPOULOS AND J. GAZOPOULOS, *J. Chem. Soc.*, (1960) 3861)Solvents:  $S_1$  = Butan-1-ol-acetic acid-water (4:1:5). $S_2$  = Butan-2-ol-formic acid-water (1:3:2). $S_3$  = Butan-1-ol-acetic acid-pyridine-water (15:3:10:12).

Paper: Whatman No. 1.

Detection: 0.1% ninhydrin in ethanol.

Compound	$R_F$		
	$S_1$	$S_2$	$S_3$
L-Isoleucyl-1(or 3)-benzyl-L-histidyl-L-prolyl-L-phenylalanine methyl ester dihydrobromide	0.85	0.77	
L-Valyl-L-tyrosine methyl ester hydrochloride			0.91
L-Isoleucyl-1(or 3)-benzyl-L-histidyl-L-prolyl-L-phenylalanine		0.76	
L-Isoleucyl-1(or 3)-benzyl-L-histidyl-L-prolyl-L-phenylalanine benzyl ester ditoluene- <i>p</i> -sulphonate			0.95