APPENDIX

A.M. Lawson and K.D.R. Setchell

List of Retention Indices[a] for Methyl Ester-Trimethylsilyl (Me-TMS) Ether Derivatives of Bile Acids

Bile acid structure[b]	Iida *et al.*[c] 1987 t_r	Iida *et al.*[c] 1987 MU	Iida *et al.*[d] 1983 t_r	Elliott[e] 1972 t_r	Almé *et al.*[f] 1977 t_r[h]	Setchell[g] and Lawson t_r	Setchell[g] and Lawson MU
None							
5α	0.53	2941				0.57	
5β	0.48	2906	0.42			0.52	
Monohydroxy							
3α-ol-5α	0.86	3141		0.72	0.82(0.76)		
3α-ol-5β	0.86	3141	0.85	0.85	0.80(0.74)	0.87	
3β-ol-5α	1.07	3232		0.82		1.06	
3β-ol-5β	0.85	3133	0.81	0.81		0.86	
3β-ol-Δ^5					0.99(0.91)	0.99	
3α-ol-Δ^6						0.81	
7α-ol-5α	0.60	2992					
7α-ol-5β	0.63	3012	0.59				
7β-ol-5α	0.82	3118					
7β-ol-5β	0.74	3079	0.70			0.75	
11β-ol-5β						0.78	
12α-ol-5α	0.64	3022					
12α-ol-5β	0.61	3000	0.56				
12β-ol-5α	0.66	3030					
12β-ol-5β	0.59	2981	0.53				
3β-ol-7α-OMe-Δ^5							3251
3β-ol-7β-OMe-Δ^5							3319
Dihydroxy							
3α,6α-ol-5α							
3α,6α-ol-5β	1.09	3238	1.14	1.12	1.11(1.02)	1.07	
3α,6β-ol-5β				1.10	1.05(0.97)	1.01	
3β,6β-ol-5α				1.33			
3α,7α-ol-5α	1.02	3210		0.84	1.00(0.92)		

(*continued*)

APPENDIX (*Continued*)

Bile acid structure[b]	Iida *et al.*[c] 1987 t_r	MU	Iida *et al.*[d] 1983 t_r	Elliott[e] 1972 t_r	Almé *et al.*[f] 1977 t_r[h]	Setchell[g] and Lawson t_r	MU
3α,7α-ol-5β	1.05	3225	1.07	1.04	1.03(0.95)	1.04	3216
3α,7β-ol-5α	1.14	3259					
3α,7β-ol-5β	1.17	3268	1.23	1.21	1.15(1.06)		3252
3β,7α-ol-5α	1.04	3223		1.08			
3β,7α-ol-5β	0.96	3190	0.96	0.96	0.93(0.86)	0.96	
3β,7α-ol-Δ^4							3200
3β,7α-ol-Δ^5							3204
3β,7β-ol-5α	1.46	3360					
3β,7β-ol-5β	1.17	3268	1.23			1.14	
3α,12α-ol-5α	0.92	3173		1.18	0.91(0.84)	0.93	
3α,12α-ol-5β	1.00	3200	1.00	1.00	1.00(0.92)	1.00	
3α,12β-ol-5α	0.94	3181				0.95	
3α,12β-ol-5β	0.95	3186	0.96		0.92(0.85)	0.95	
3β,12α-ol-5α	1.16	3267		1.23	1.18(1.09)	1.19	
3β,12α-ol-5β	0.98	3198	0.99	1.00	1.05(0.88)	0.99	
3β,12β-ol-5α	1.27	3298				1.28	
3β,12β-ol-5β	1.00	3200	0.99			0.99	
3β,12α-ol-Δ^5					1.14(1.05)		
7α,12α-ol-5α	0.65	3028					
7α,12α-ol-5β	0.90	3162	0.68			0.73	
7α,12β-ol-5α	0.67	3043					
7α,12β-ol-5β	0.71	3061	0.68				
7α,12α-ol-5α	0.89	3157					
7β,12α-ol-5β	0.86	3141	0.85				
7β,12β-ol-5α	0.97	3195					
7β,12β-ol-5β	0.88	3151	0.87				
Trihydroxy							
1,3,12-ol-5β					1.31(1.21)*		
3α,6α,7α-ol-5α							
3α,6α,7α-ol-5β	1.28	3302	1.39	1.15	1.38(1.27)		3320
3α,6β,7α-ol-5β				1.12			
3α,6β,7β-ol-5α				1.44			
3α,6β,7β-ol-5β				1.45			
3α,6α,7β-ol-5β				1.92			
3α,6β,12α-ol-5β					1.18(1.09)*		
3α,7α,12α-ol-5α	1.03	3211	1.09	1.05	1.03(0.95)		
3α,7α,12α-ol-5β	1.06	3228		1.09	1.09(1.00)	1.07	3227
3α,7α,12β-ol-5α	1.03	3211					
3α,7α,12β-ol-5β	1.02	3210	1.09			1.02	
3α,7β,12α-ol-5α	1.15	3262			1.18(1.09)		
3α,7β,12α-ol-5β	1.53	3375	1.31		1.25(1.15)		
3α,7β,12β-ol-5α	1.25	3289					

APPENDIX (*Continued*)

Bile acid structure[b]	Iida *et al.*[c] 1987 t_r	Iida *et al.*[c] 1987 MU	Iida *et al.*[d] 1983 t_r	Elliott[e] 1972 t_r	Almé *et al.*[f] 1977 t_r[h]	Setchell[g] and Lawson t_r	Setchell[g] and Lawson MU
3α,7β,12β-ol-5β	1.20	3274	1.34			1.17	
3β,7α,12α-ol-5α	1.07	3232		1.01			
3β,7α,12α-ol-5β	0.98	3198	1.00	0.96			
3β,7α,12α-ol-Δ^4							3214
3β,7α,12β-ol-5α	1.18	3269					
3β,7α,12β-ol-5β	1.00	3200	1.06				
3β,7β,12α-ol-5α	1.50	3340			1.18(1.09)*		
3β,7β,12α-ol-5β	1.18	3269	1.31		1.22(1.13)		
3β,7β,12β-ol-5α	1.75	3429					
3β,7β,12β-ol-5β	1.29	3305	1.43				
3α,12α,16α-ol-5β						1.81	
3α,12α,23-ol-5β						1.33	
Tetrahydroxy							
1β,3α,7α,12α-ol-5β					1.36(1.25)		3325
1,3,7,12-ol-5χ					1.40(1.29)		
3,6,7,12-ol-5χ					1.42(1.31)		
3α,6α,7α,12α-ol-5β					1.36(1.25)		3313
3α,7α,12α,23-ol-5β					1.56(1.44)		
Oxo							
3-oxo-Δ^4						1.27	
3-oxo-6α-ol-5β				1.32			
3-oxo-7α-ol-5α				1.08			
3-oxo-7α-ol-5β				1.17			
3-oxo-7α-ol-Δ^4							3298
3-oxo-12α-ol-5α				1.17			
3-oxo-12α-ol-5β				1.09			
3-oxo-7α,12α-ol-5α				1.09			
3-oxo-7α,12α-ol-5β				1.35			
3-oxo-7α,12α-ol-Δ^4							3360
6-oxo-3α-ol-5α							
7-oxo-3α-ol-5α				1.11			
7-oxo-3α-ol-5β				1.34		1.35	3315
7-oxo-3α,6α-diol-5β						1.42	
7-oxo-3α,12α-ol-5α				1.62			
7-oxo-3α,12α-ol-5β				1.59			
7-oxo-3β,12α-ol-5α				1.96			
12-oxo-3α-ol-5a						1.62	
12-oxo-3α-ol-5β				1.33	1.36(1.25)	1.65	
12-oxo-3β-ol-5β						1.31	
12-oxo-3α,7α-ol-5β				1.72			
12-oxo-3β-ol-5β-$\Delta^{9(11)}$						1.32	

APPENDIX (*Continued*)

Bile acid structure[b]	Iida *et al.*[c] 1987 t_r	Iida *et al.*[c] 1987 MU	Iida *et al.*[d] 1983 t_r	Elliott[e] 1972 t_r	Almé *et al.*[f] 1977 t_r[h]	Setchell[g] and Lawson t_r	Setchell[g] and Lawson MU
Dioxo							
3,7-dioxo-12α-ol-5α				1.78			
3,7-dioxo-12α-ol-5β				1.56			
3,12-dioxo-5β						1.43	
7,12-dioxo-3α-ol-5β				1.88			
Nor-bile acids							
Nor-5β-cholan-23-oic						0.39	
Norlithocholic						0.66	
Nordeoxycholic						0.77	
Norchenodeoxycholic						0.79	
Norhyodeoxycholic						0.79	
Norcholic					0.76(0.70)	0.84	
Norursodeoxycholic						0.85	
C_{27} bile acids							
3α,7α-ol-5β-C_{27}							3452
3α,7α,12α-ol-5β-C_{27}							3455
1β,3α,7α,12α-ol-5β-C_{27}							3550
3α,6α,7α12α-ol-5β							3540
3α,7α,12α,24-ol-5β[i]							3600
3α,7α,12α,24-ol-5β[i]							3612
3α,7α,12α,25-ol-5β							3628
3α,7α,12α,26-ol-5β							3715
C_{29} bile acids							
3α,7α,12α-ol-5β-C_{29}-dioic							3930
C_{20} bile acids							
3α-ol-5β						0.40	
3β-ol-5β						0.32	
3β-ol-Δ^5					0.38		

[a] Retention data are expressed for the Me-TMS ethers relative to the Me-TMS ether derivative of deoxycholic acid and/or as the methylene unit (MU) values relative to a homologous series of n-alkanes.

[b] Bile acids are listed according to the number of functional groups. For clarity unless indicated all bile acids have the cholanoic acid (C_{24}) nucleus and the presence of hydroxy groups is indicated by the term -ol (i.e., 3α-ol indicates 3α-hydroxy structure). The position of the hydrogen at C-5 is indicated as 5β or 5α and Δ signifies unsaturation at the position shown by the superscript. Nor-bile acids are indicated by their trivial names. C_{27} indicates a cholestanoic acid nucleus. The C_{20} bile acids have the androstane-17β-oic nucleus.

[c] Bile acid derivatives were chromatographed on an 18 meter OV-1 chemically bonded fused silica capillary column (HiCap-CBP-1; 0.2 mm internal diameter, 0.25 μm film thickness) at 270°C with helium as carrier gas (linear velocity 32 cm/sec)-[T. Iida *et al.*, *J. Chromatogr.* (1987)].

[d] Chromatography was performed on a 30 meter glass capillary column coated with OV-1 (Nihon Chromato Works Ltd, Tokyo, Japan) at 245°C using nitrogen (flow rate 1.7 ml/min) as carrier gas [Iida *et al.*, *J. Lipid Res.* **24**:211 (1983)].

[e] Chromatography was performed on conventional GLC column packed with 3% OV-1 liquid phase (see Ref. 56, Chapter 5).

[f] Chromatography was performed on 2.5 meter × 3.4 mm conventional GLC column packed with 1.5% SE-30 on Gas Chrom Q (80–100 ml) at 210–240°C (see ref. 13, Chapter 5).

[g] Chromatography was performed on a 30 meter DB-1 chemically bonded fused silica capillary column at 275°C using helium (flow rate 2ml/min) as carrier gas.

[h] Values were cited in the original article relative to the Me-TMS ether of cholic acid and these values are given in parentheses. Values have been recalculated and expressed relative to the Me-TMS ether of deoxycholic acid.

[i] These may be two C_{24} (*R* and *S*) or C_{25} isomers.

* This indicates a tentative identification.

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