

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

(continued)

APPENDIX (Continued)

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

APPENDIX (Continued)

Bile acid structure ^b	Iida <i>et al.</i> ^c 1987		Iida <i>et al.</i> ^d 1983	Elliott ^e 1972	Almé <i>et al.</i> ^f 1977	Setchell ^g and Lawson	
	<i>t_r</i>	MU	<i>t_r</i>	<i>t_r</i>	<i>t_r</i> ^h	<i>t_r</i>	MU
<i>Dioxo</i>							
3,7-dioxo-12 α -ol-5 α				1.78			
3,7-dioxo-12 α -ol-5 β				1.56			
3,12-dioxo-5 β						1.43	
7,12-dioxo-3 α -ol-5 β				1.88			
<i>Nor-bile acids</i>							
Nor-5 β -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	
<i>C₂₇ bile acids</i>							
3 α ,7 α -ol-5 β -C ₂₇							3452
3 α ,7 α ,12 α -ol-5 β -C ₂₇							3455
1 β ,3 α ,7 α ,12 α -ol-5 β - C ₂₇							3550
3 α ,6 α ,7 α ,12 α -ol-5 β							3540
3 α ,7 α ,12 α ,24-ol-5 β ⁱ							3600
3 α ,7 α ,12 α ,24-ol-5 β ⁱ							3612
3 α ,7 α ,12 α ,25-ol-5 β							3628
3 α ,7 α ,12 α ,26-ol-5 β							3715
<i>C₂₉ bile acids</i>							
3 α ,7 α ,12 α -ol-5 β - C ₂₉ -dioic							3930
<i>C₂₀ bile acids</i>							
3 α -ol-5 β						0.40	
3 β -ol-5 β						0.32	
3 β -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 (C₂₄) nucleus and the presence of hydroxy groups is indicated by the term -ol (i.e., 3 α -ol indicates 3 α -hydroxy structure). The position of the hydrogen at C-5 is indicated as 5 β or 5 α and Δ signifies unsaturation at the position shown by the superscript. Nor-bile acids are indicated by their trivial names. C₂₇ indicates a cholestanolic acid nucleus. The C₂₀ bile acids have the androstane-17 β -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 μ m 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 C₂₄ (*R* and *S*) or C₂₅ isomers.
- * This indicates a tentative identification.

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