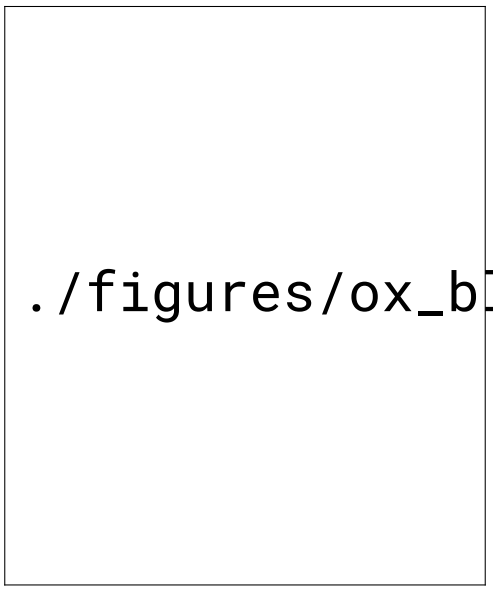
Optimising NMR Spectroscopy through Method and Software Development



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Appendix A

Peak assignments for some samples

chpt:assignments

In this appendix, I provide assignments of ^{1}H and ^{13}C chemical shifts for some of the more frequently-used samples in this thesis, occasionally with some additional data. I have made no attempt to distinguish the shifts of diastereotopic protons or methyl groups.

A.1 Andrographolide (A)

amples_andrographolide

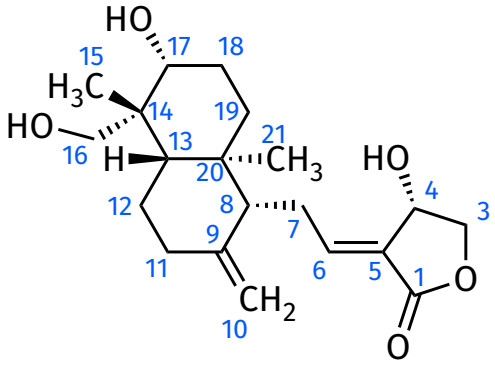


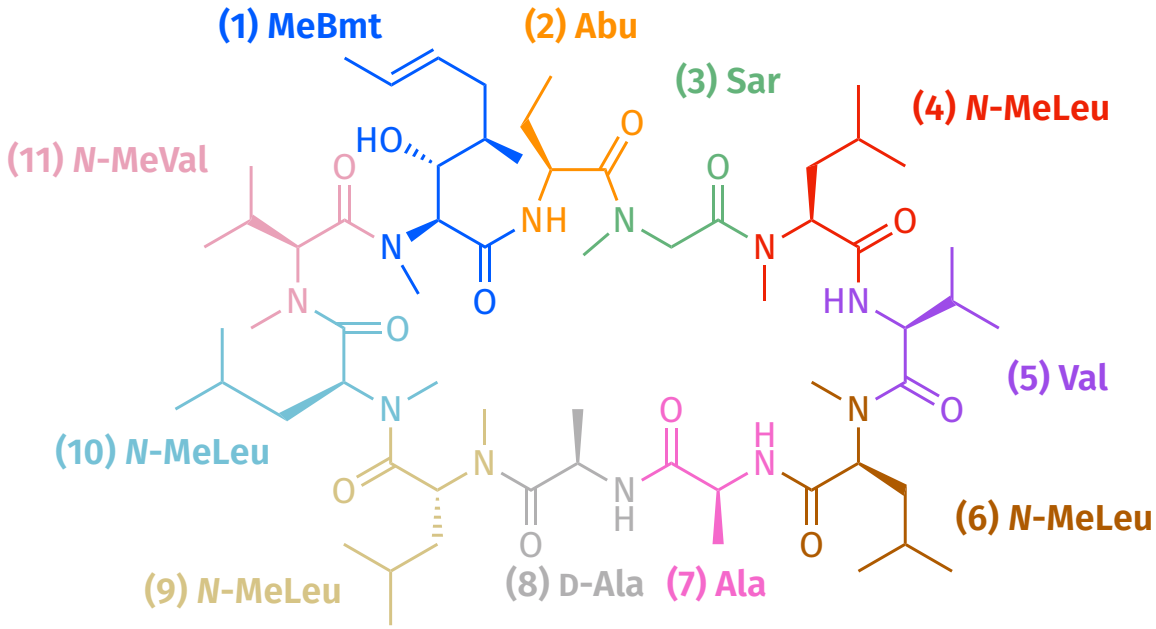
Figure A.1: Structure of andrographolide.

grapholide_assignments

Label in fig. A.1	Label in Claridge (2016)	$\delta(^1\text{H})$ (ppm)	$\delta(^{13}\text{C})$ (ppm)
1			170.42
3	g, i	4.40, 4.04	74.80
4	d	4.92	64.99
4-OH	b	5.71	
5			129.46
6	a	6.63	146.76
7	m, n	2.52, 2.47	24.43*
8	q	1.87	55.96
9			148.07
10	e, f	4.82, 4.63	108.71
11	o, p	2.33, 1.94	37.98
12	r, v	1.75, 1.36	24.42*
13	w	1.21	54.84
14			42.75
15	y	1.09	23.54
16	j, k	3.85, 3.27	63.11
16-OH	h	4.13	
17	l	3.24	78.91
17-OH	c	5.05	
18	t, u	1.65, 1.65	28.36
19	s, x	1.70, 1.21	36.99
20			39.06
21	z	0.67	15.22

Table A.1: Peak assignments for andrographolide; these can also be found in Figure 9.26 (page 340) of: Claridge, T. D. W., *High-Resolution NMR Techniques in Organic Chemistry*, 3rd ed.; Elsevier: Amsterdam, 2016. Asterisks indicate ^{13}C chemical shifts which could not be disambiguated.

A.2 Cyclosporin (C)



ig:samples_cyclosporin

Figure A.2: Structure of cyclosporin.

Yada...

A.3 Ferulic acid (F)

fig:samples_ferulic

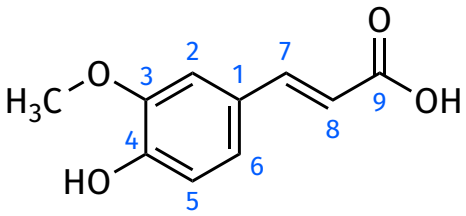


Figure A.3: Structure of ferulic acid.

bl:ferulic_assignments

Label	$\delta(^1\mathrm{H})$ (ppm)	$\delta(^{13}\mathrm{C})$ (ppm)	$^1J_{\mathrm{CH}}$ (Hz)	$T_1(^1\mathrm{H})$ at 600 MHz (s)
1		126.25		
2	7.28	111.62	157.6	1.15
3		148.38		
3-OMe	3.82	56.16	147.8	1.08
4		149.54		
5	6.79	115.98	159.5	1.59
6	7.08	123.27	158.6	2.00
7	7.49	144.97	154.3	1.96
8	6.36	116.08	160.6	1.75
9		168.45		

Table A.2: Peak assignments and other physical data for ferulic acid.

A.4 Gramicidin (G)

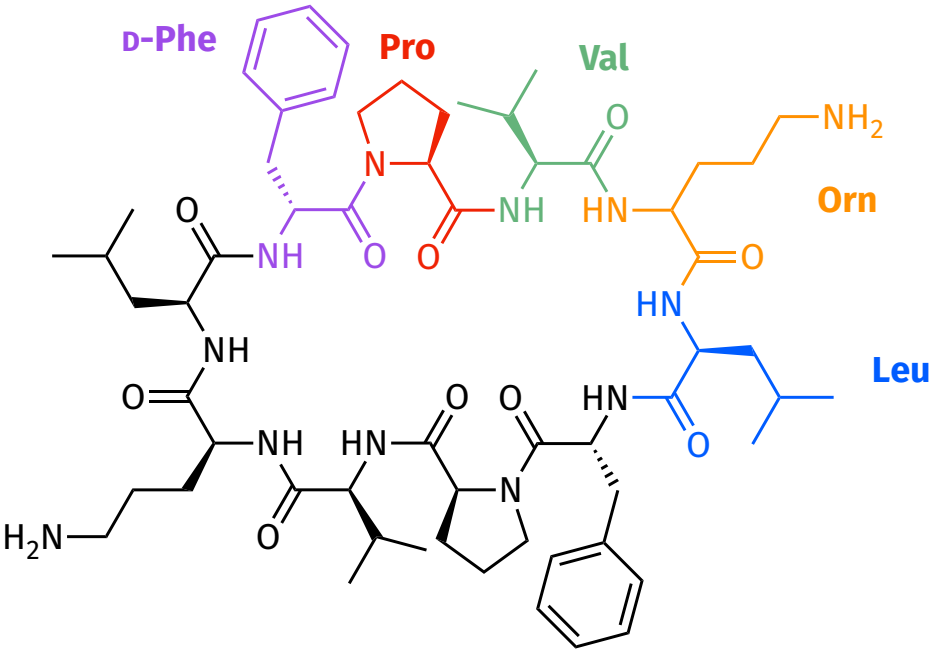


fig:samples_gramicidin

Figure A.4: Structure of gramicidin.

Residue	Label	$\delta(^1H)$ (ppm)	$\delta(^{13}C)$ (ppm)	$\delta(^{15}N)$ (ppm)
Leu	NH	8.33		123.3
	α-CH	4.57	50.09	
	β-CH_2	1.35, 1.29	41.38	
	γ-CH	1.41	24.45	
	δ-CH_3	1.41	23.20, 23.02	
Orn	NH	8.66		125.4
	α-CH	4.76	51.43	
	β-CH_2	1.75, 1.60	30.12	
	γ-CH_2	1.65	23.52	
	δ-CH_2	2.84, 2.78	39.02	
	ε-NH_2	8.04		36.0
Val	NH	7.22		113.2
	α-CH	4.41	57.31	
	β-CH	2.07	31.49	
	γ-CH_3	0.80, 0.77	19.44, 18.50	
Pro	α-CH	4.31	60.36	
	β-CH_2	1.95, 1.48	29.50	
	γ-CH_2	1.52	23.58	
	δ-CH_2	3.59, 2.50	46.48	
D-Phe	NH	9.09		128.0
	α-CH	4.36	54.38	
	β-CH_2	3.59, 2.50	46.48	
	ipso-C		136.77	
	ortho-CH	7.26	129.80	
	meta-CH	7.29	128.71	
	para-CH	7.25	127.34	

gramicidin_assignments

Table A.3: Peak assignments for gramicidin.

A.5 Brucine (X)

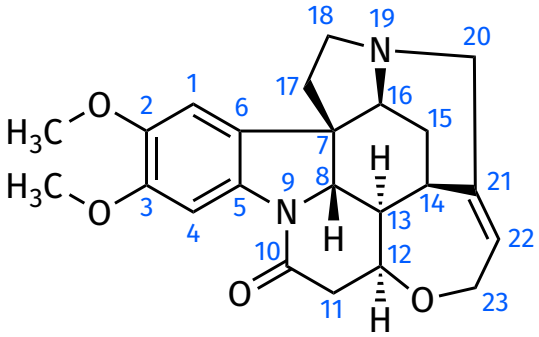


fig:samples_brucine

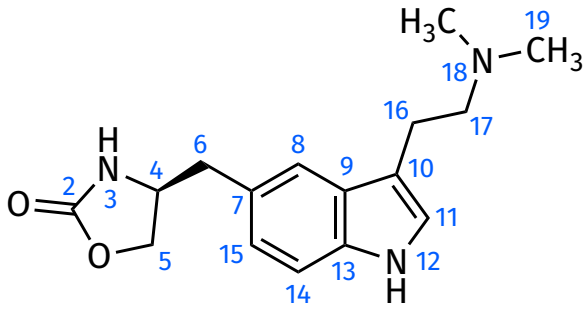
Figure A.5: Structure of brucine.

Label	$\delta(^1H)$ (ppm)	$\delta(^{13}C)$ (ppm)	$\delta(^{15}N)$ (ppm)
1	6.69	105.52	
2		146.24	
2-OMe	3.87	56.46	
3		149.25	
3-OMe	3.92	56.21	
4	7.83	101.00	
5		135.98	
6		123.33	
7		51.94	
8	3.85	60.35	
9			152.38
10		168.94	
11	3.12, 2.67	42.39	
12	4.30	77.79	
13	1.28	48.26	
14	3.16	31.55	
15	2.37, 1.49	26.79	
16	3.90	60.00	
17	1.89	42.41	
18	3.22, 2.87	50.21	
19			40.55
20	3.73, 2.75	52.71	
21		140.32	
22	5.92	127.54	
23	4.16, 4.07	64.60	

bl:brucine_assignments

Table A.4: Peak assignments for brucine.

A.6 Zolmitriptan (Z)



g:samples_zolmitriptan

Figure A.6: Structure of zolmitriptan.

Label	$\delta(^1H)$ (ppm)	$\delta(^{13}C)$ (ppm)	$\delta(^{15}N)$ (ppm)	$^1J_{CH}$ (Hz)
2		159.16		
3	7.77		89.2	
4	4.05	53.64		146.7
5	4.23, 4.03	68.54		153.0, 151.7
6	2.90, 2.79	41.11		127.7, 127.1
7		126.32		
8	7.37	119.24		155.4
9		127.99		
10		112.92		
11	7.12	123.18		179.7
12	10.71		129.6	
13		135.64		
14	7.26	111.71		158.9
15	6.93	122.97		155.7
16	2.81	23.55		125.8
17	2.53	60.46		132.2
18			106.6	
19	2.26	45.65		132.5

lmitriptan_assignments

Table A.5: Peak assignments and other physical data for zolmitriptan.