Spectra Images for:

Dinuclear Zinc-Prophenol-Catalyzed Enantioselective α-Hydroxyacetate Aldol Reaction with Activated Ester Equivalents

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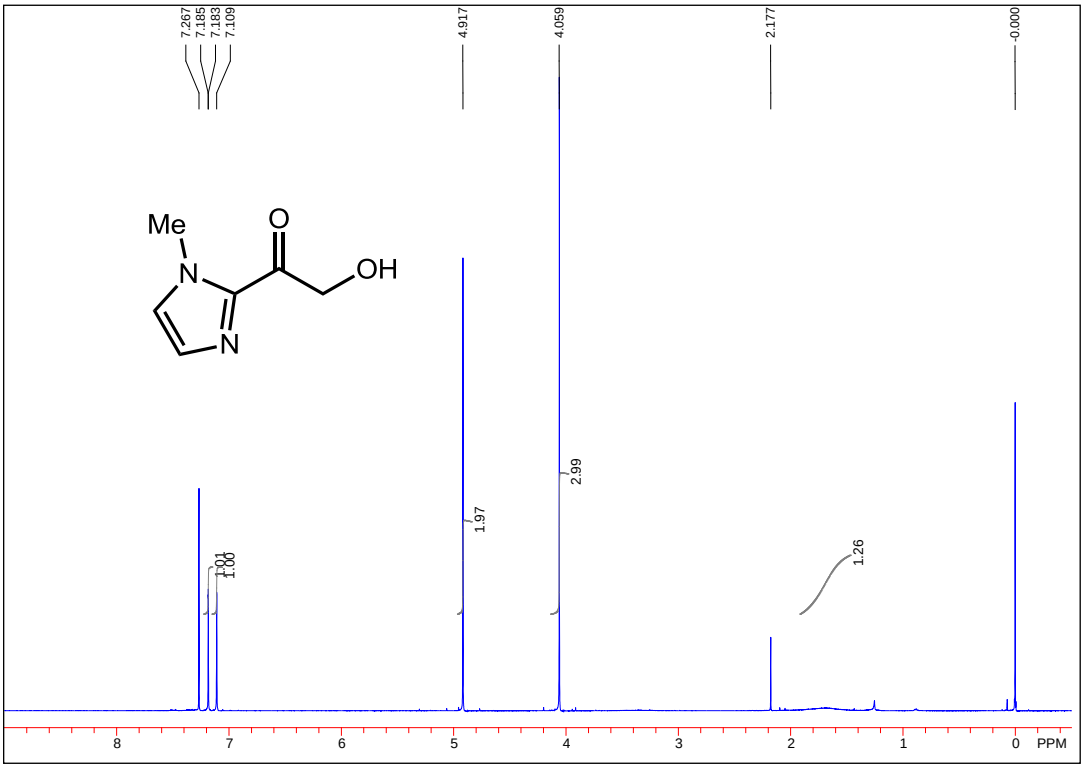
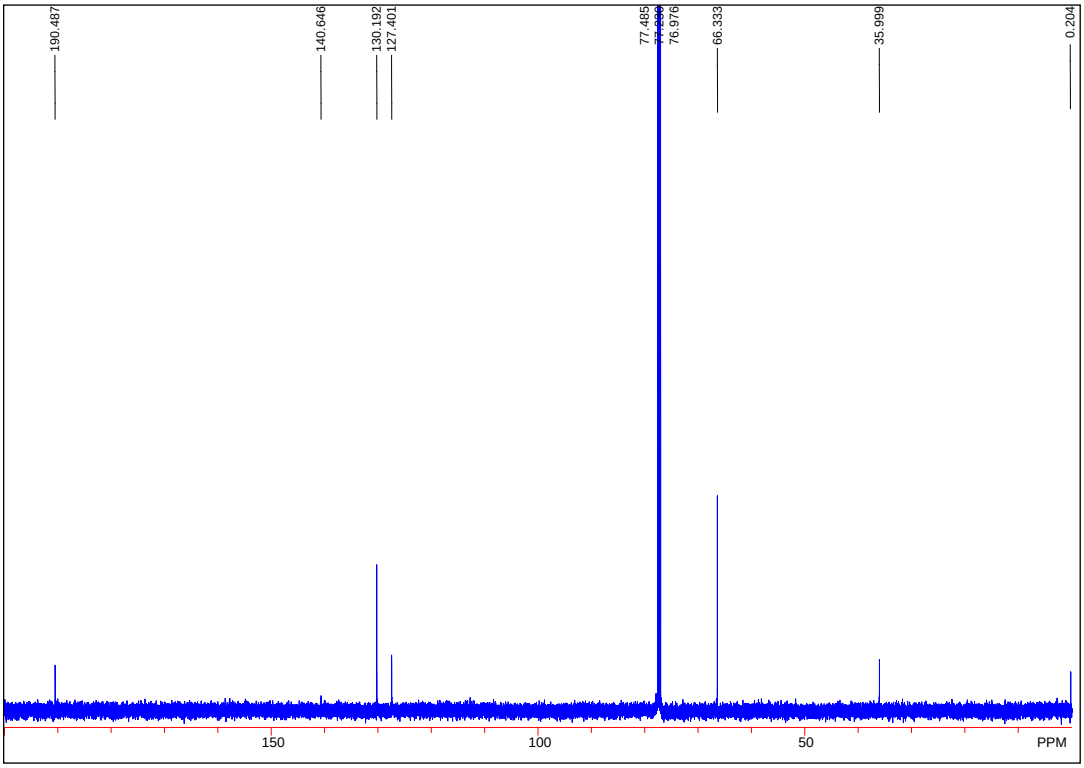
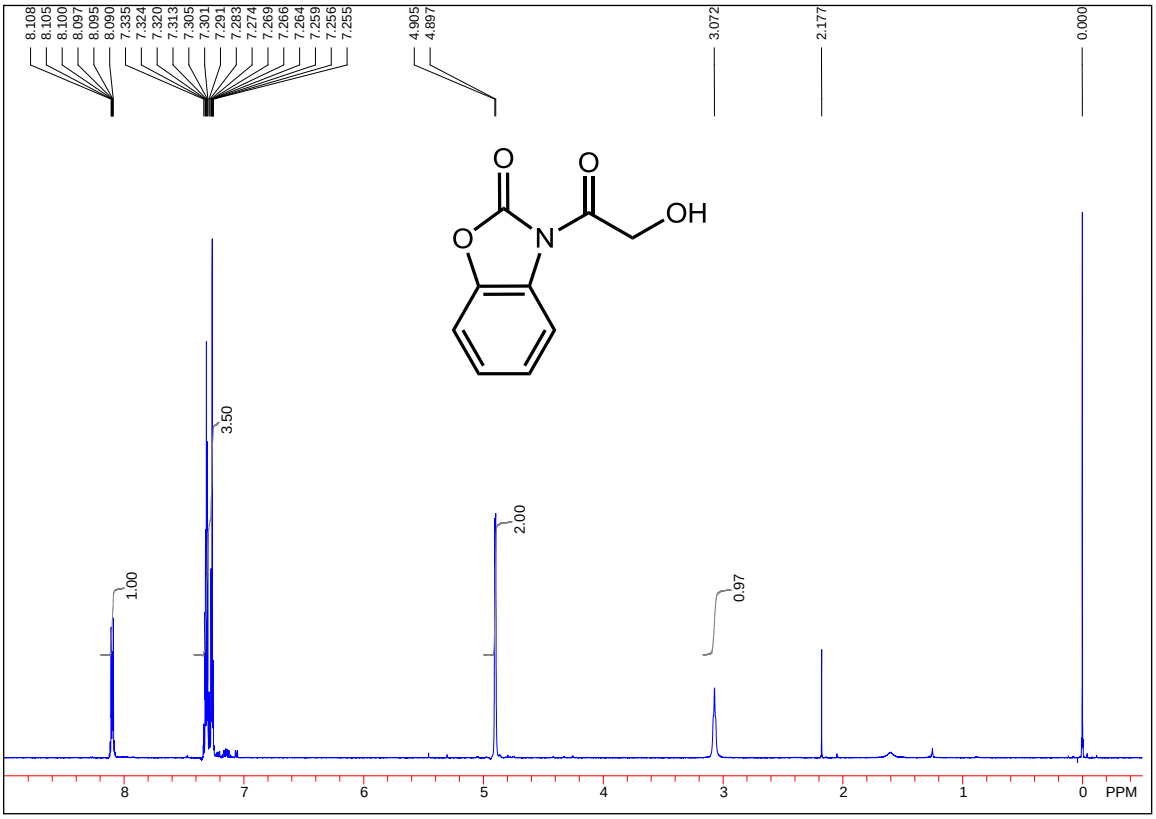
Department of Chemistry
Stanford University
Stanford, CA 94305-5080

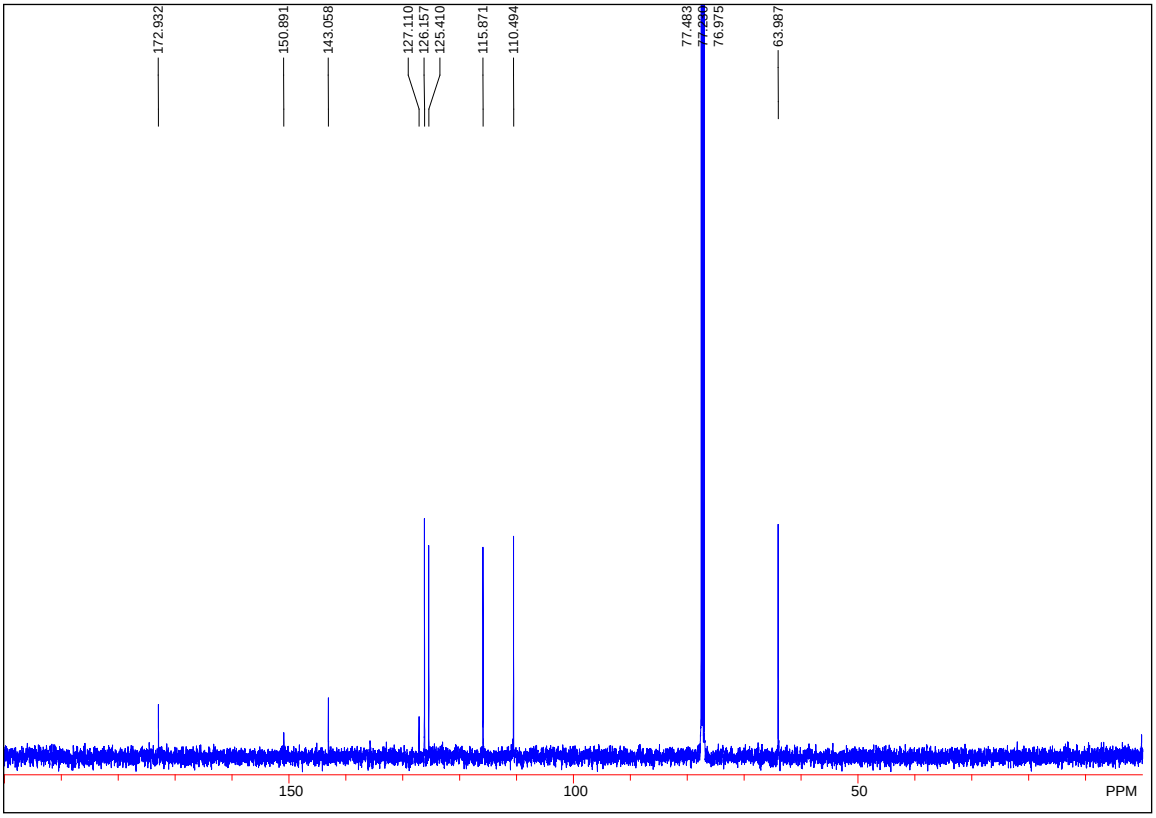
***N*-Methyl-2-hydroxyacetylimidazole (1d):**



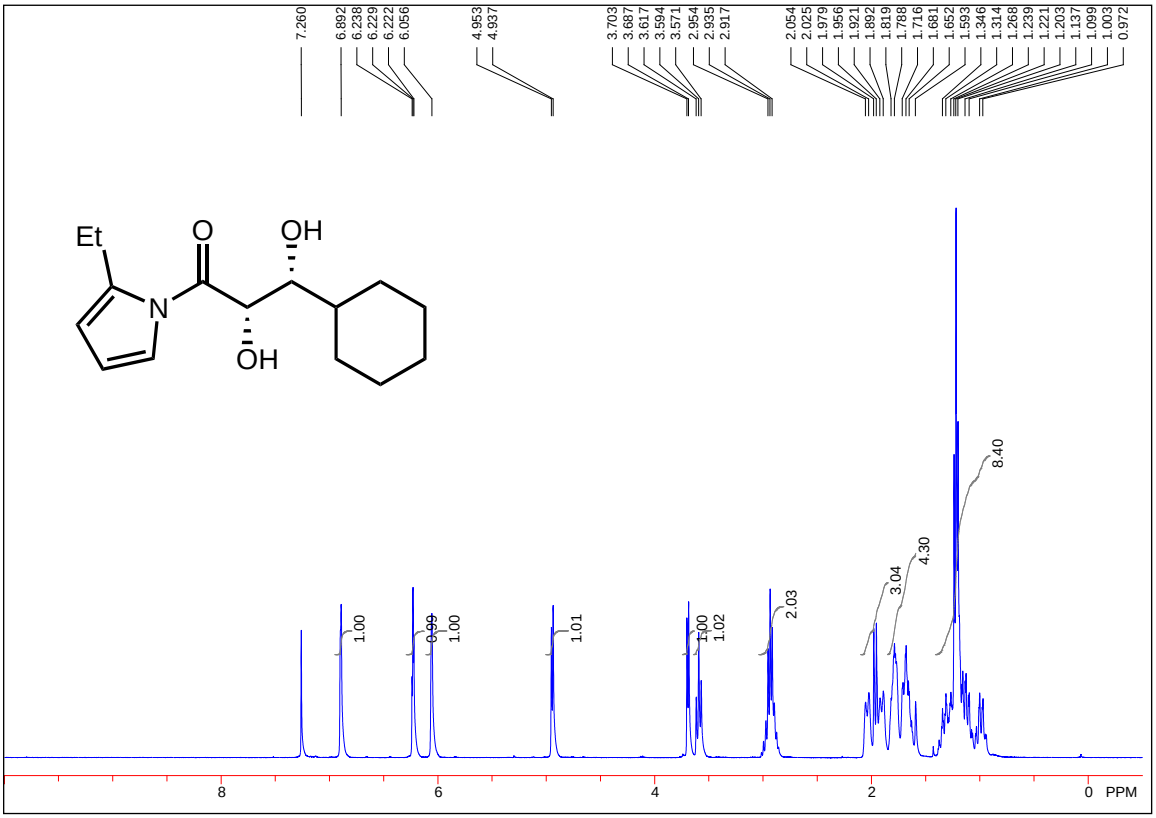


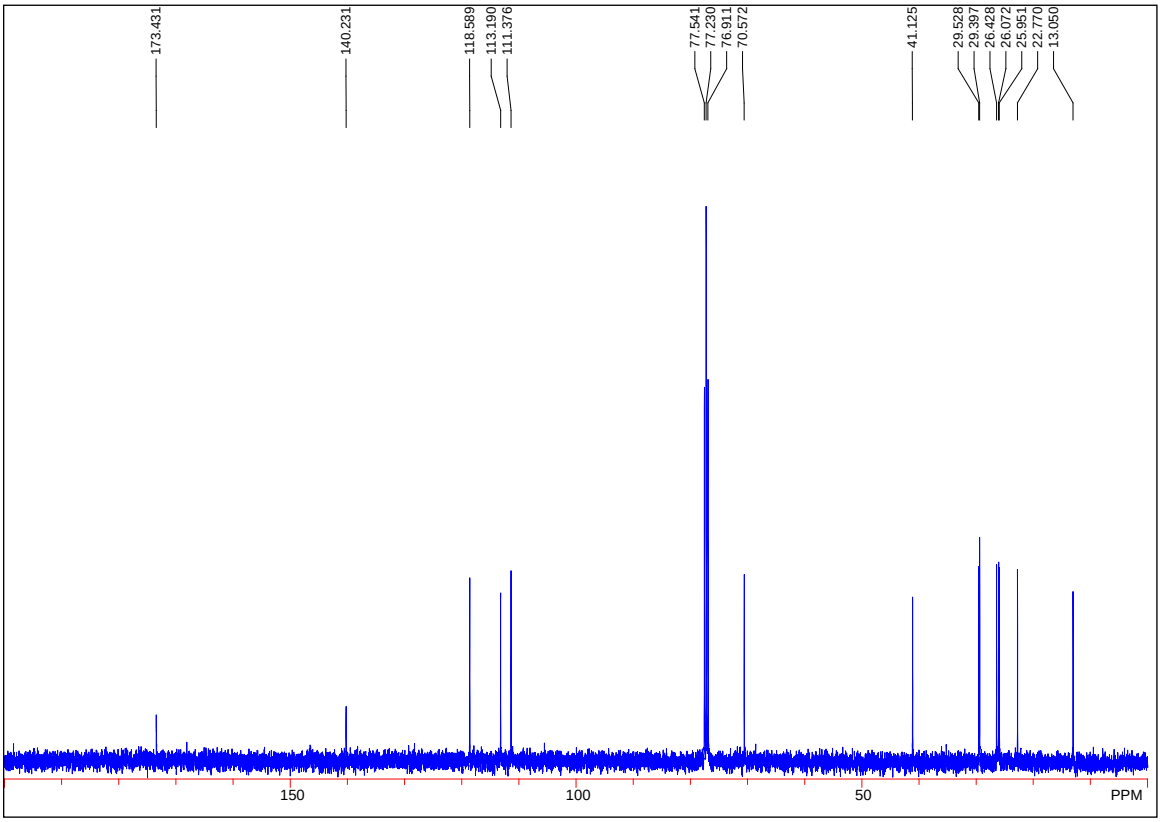
3-(2-hydroxyacetyl)benzo[d]oxazol-2(3H)-one (1f):



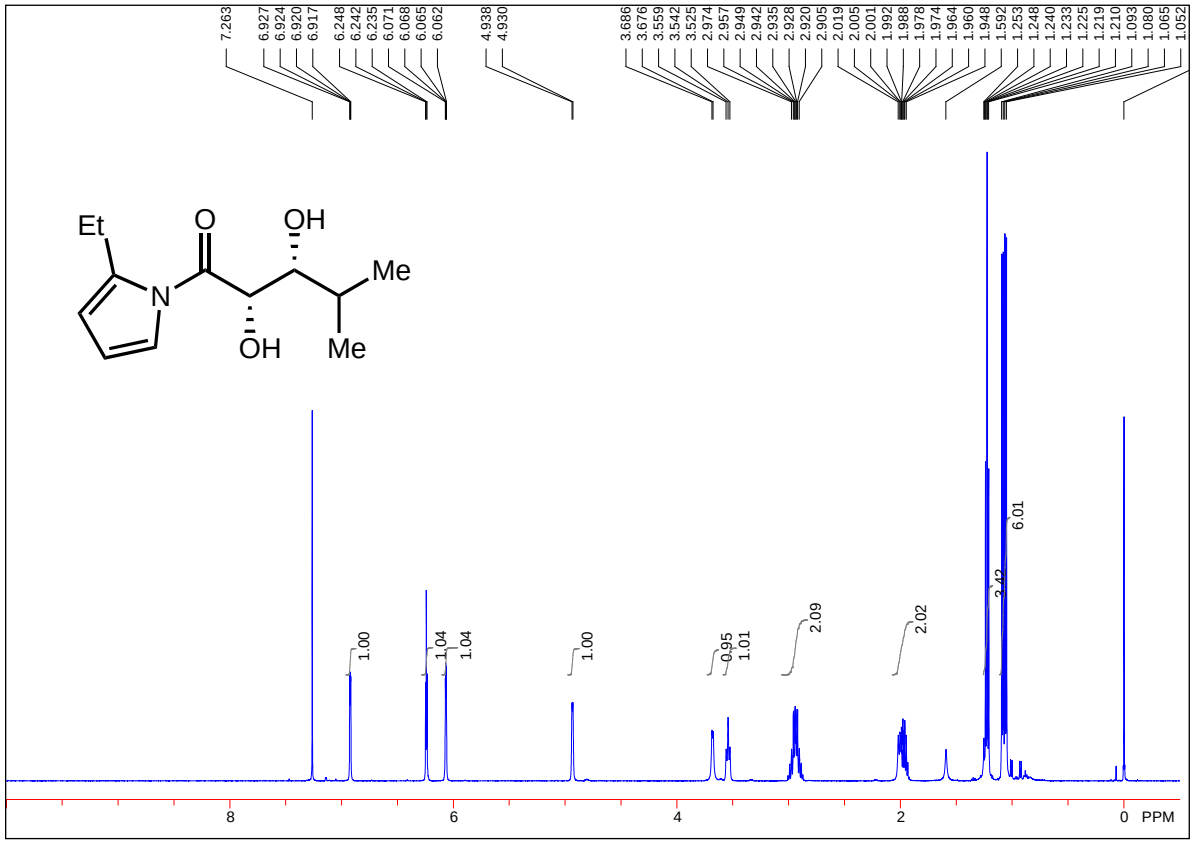


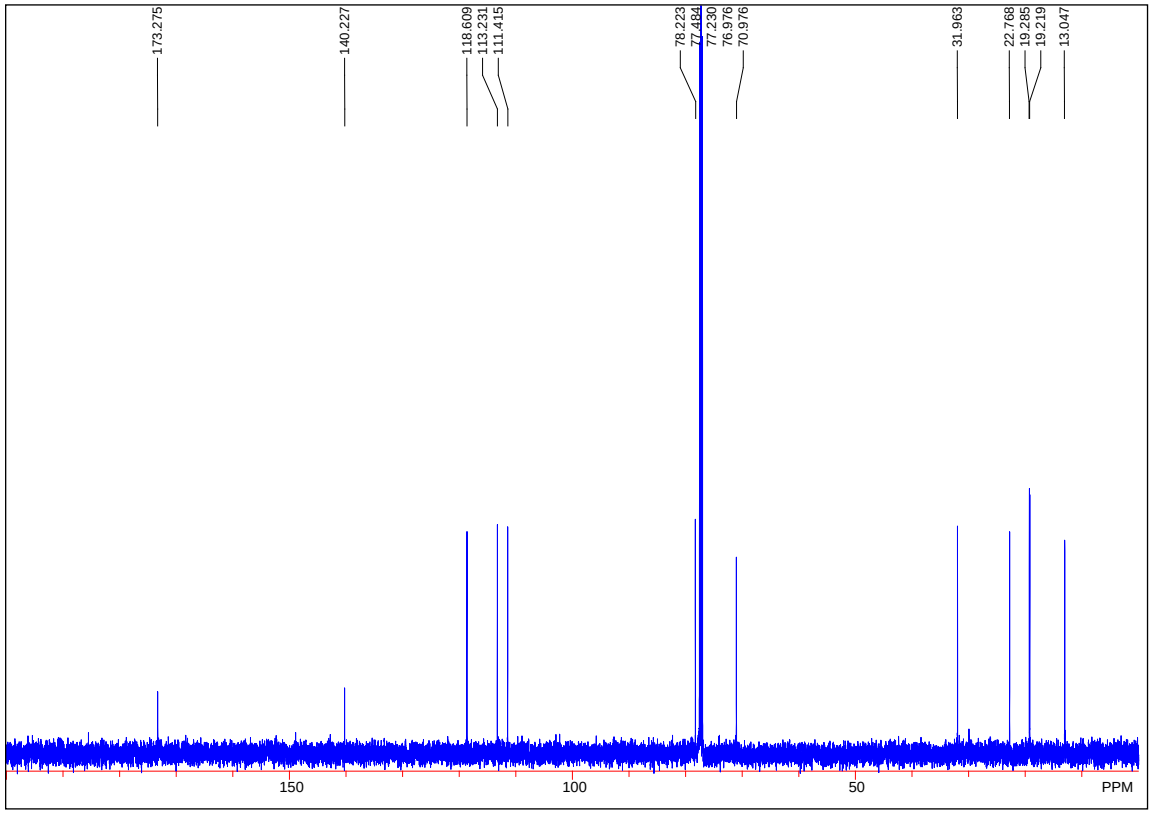
(2R,3S)-3-cyclohexyl-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxypropan-1-one (3c):



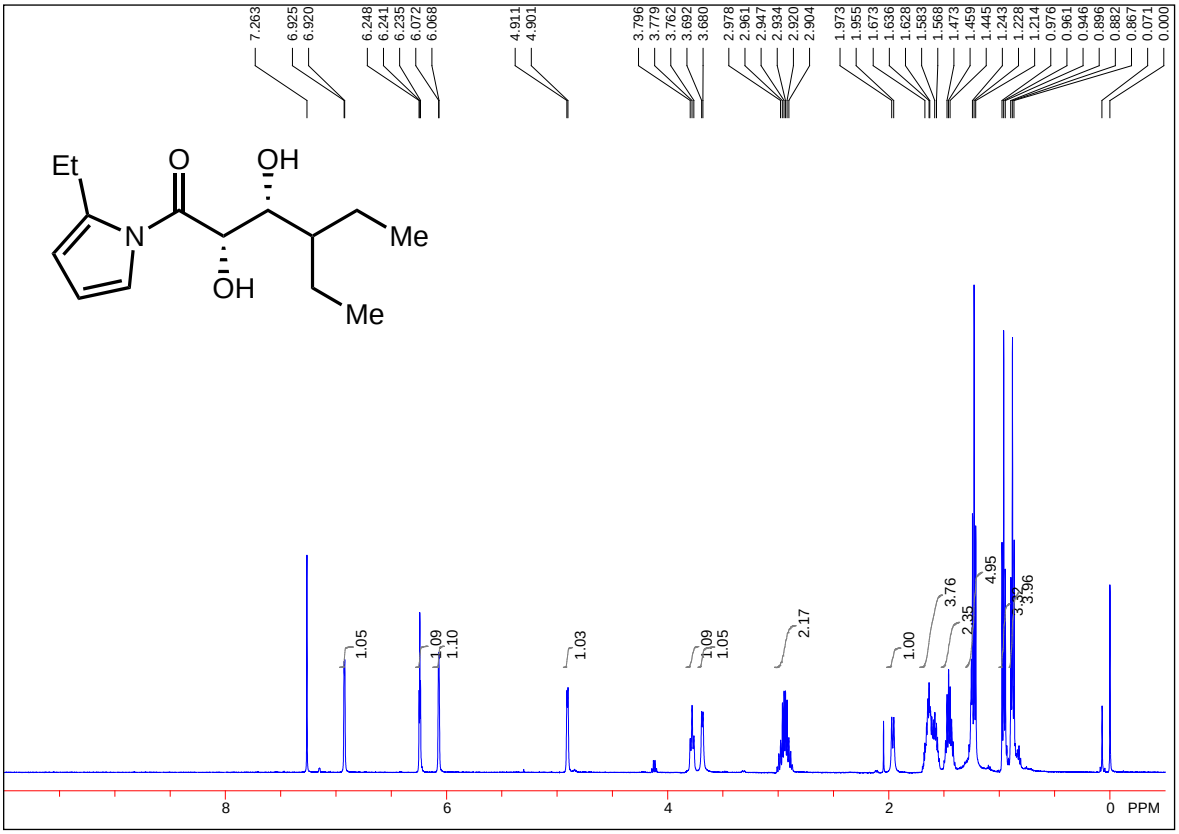


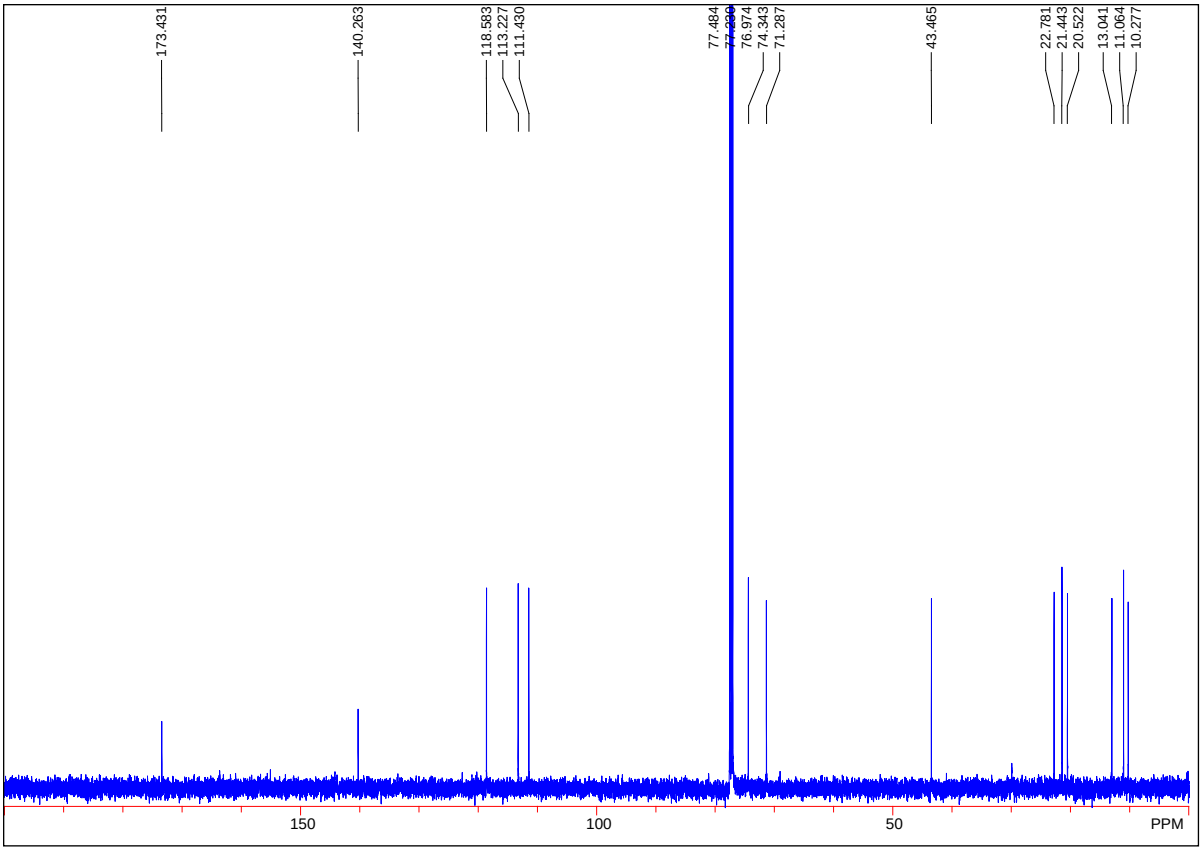
(2R,3S)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxy-4-methylpentan-1-one (3d):



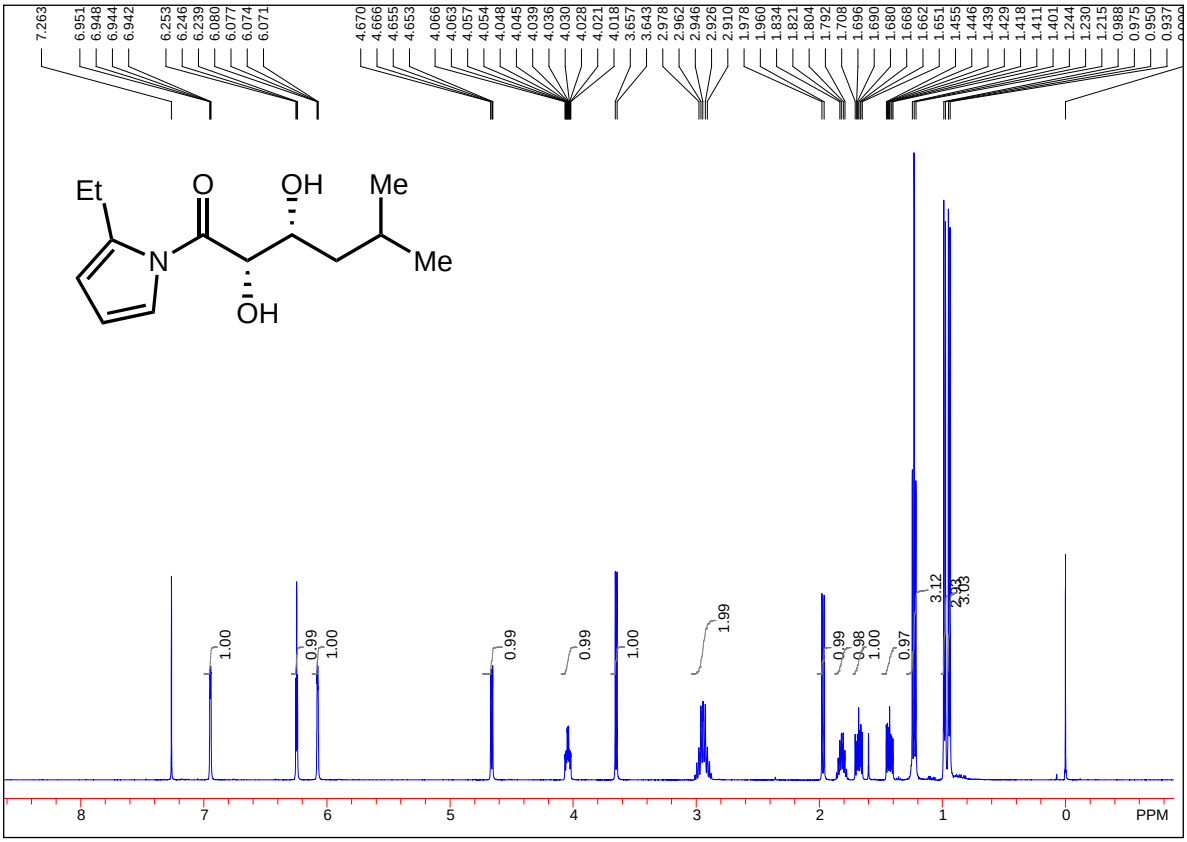


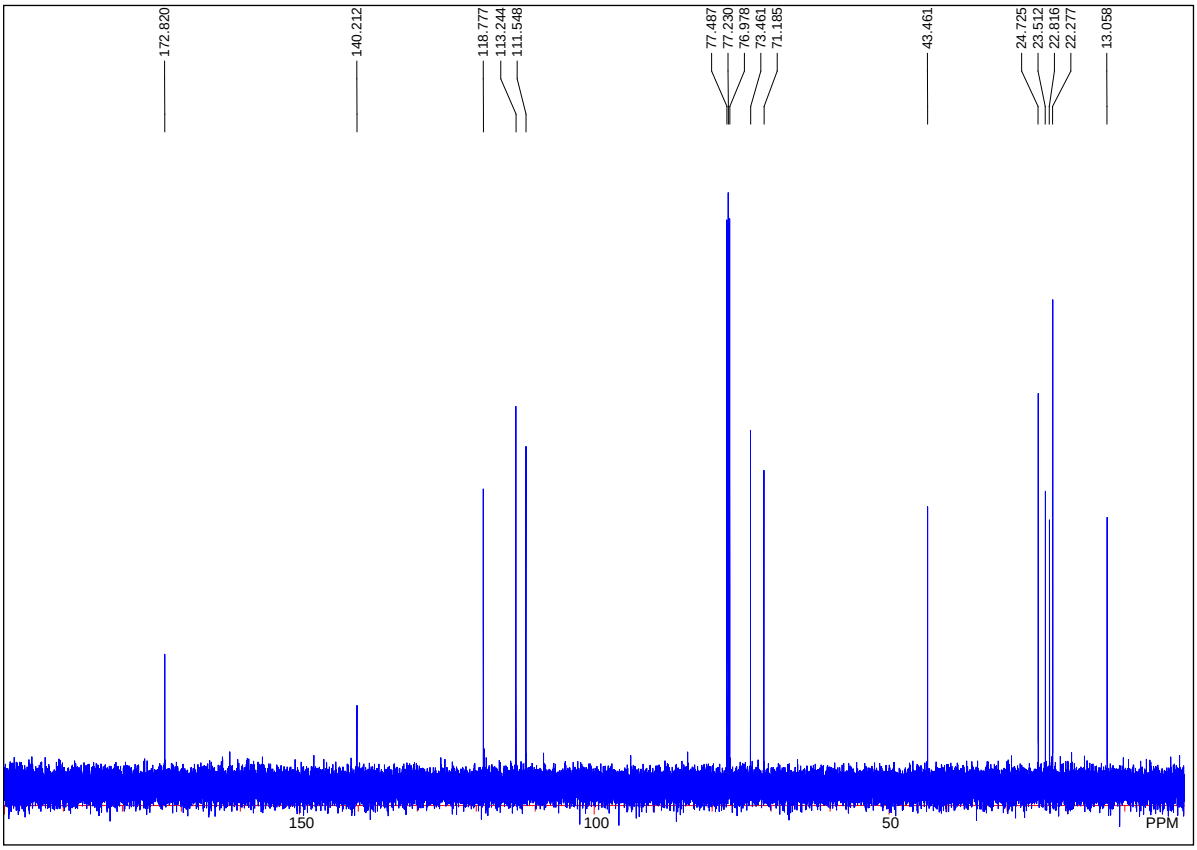
(2R,3S)-4-ethyl-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxyhexan-1-one (3e):



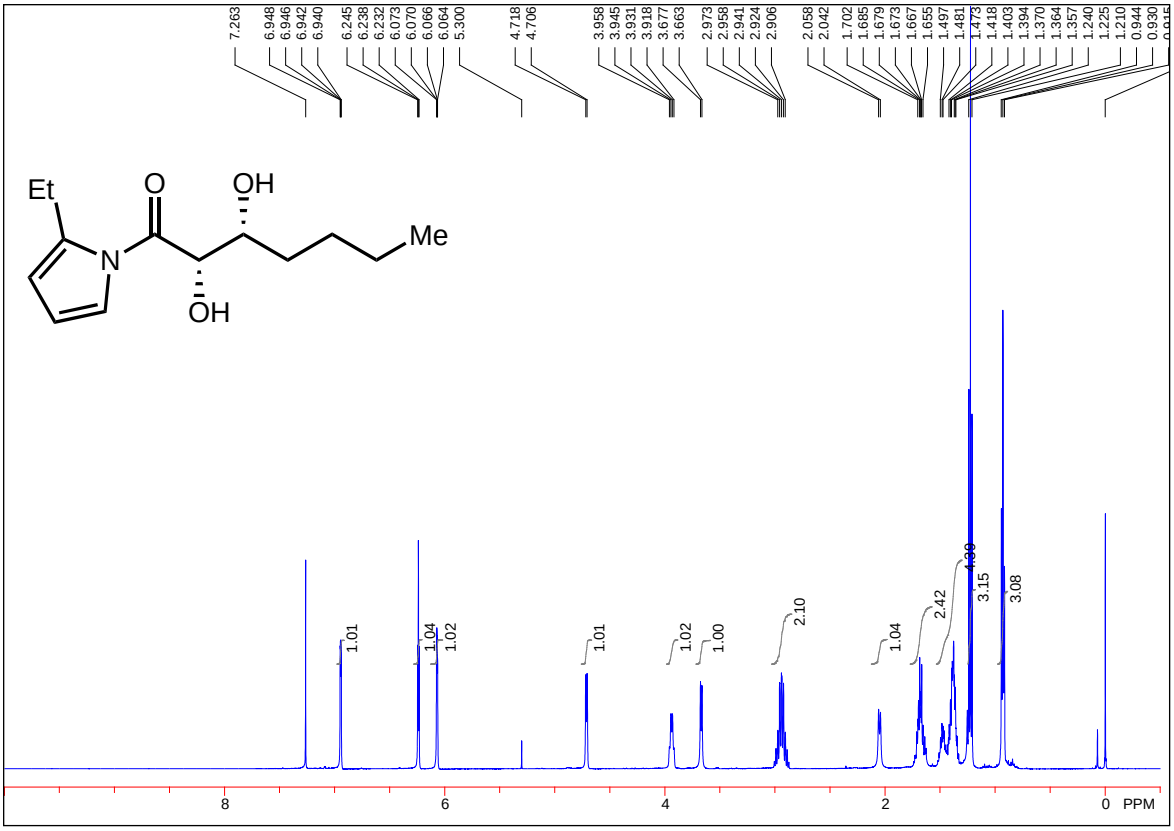


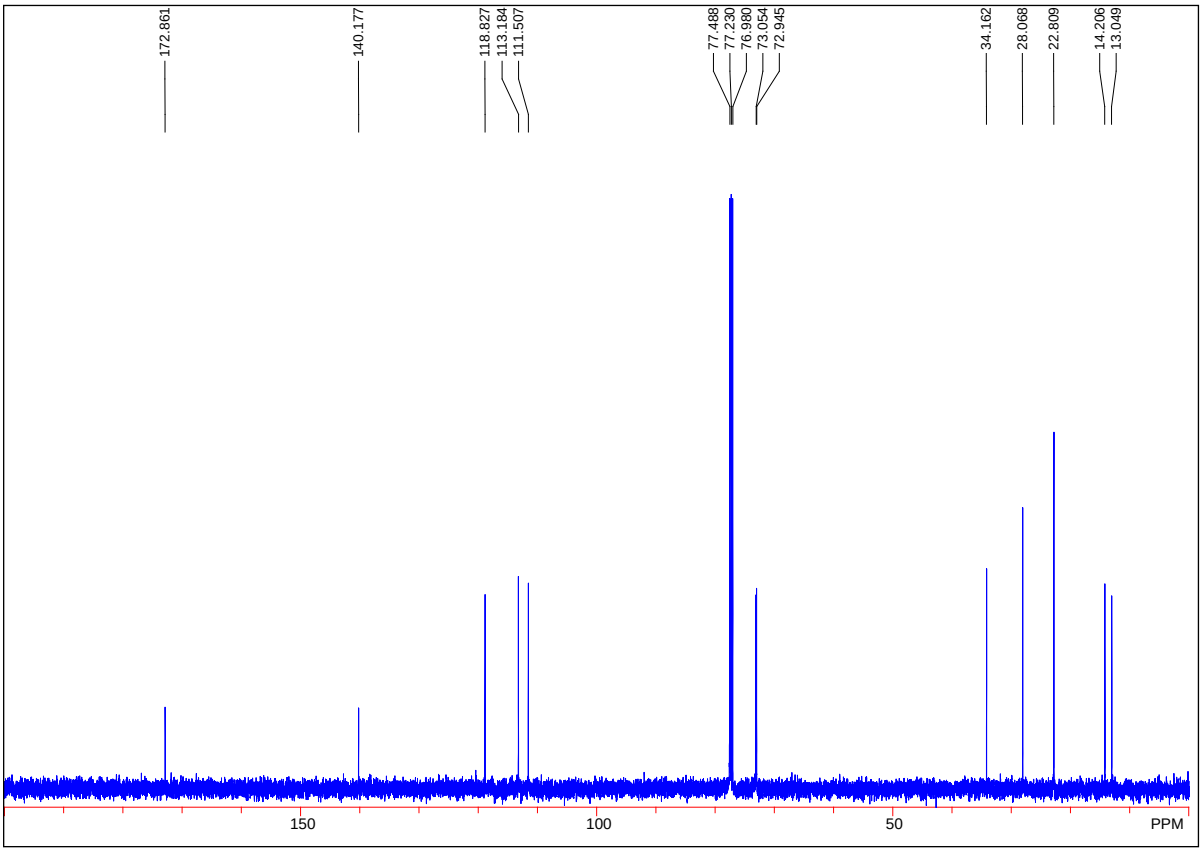
(2R,3S)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxy-5-methylhexan-1-one (3f):



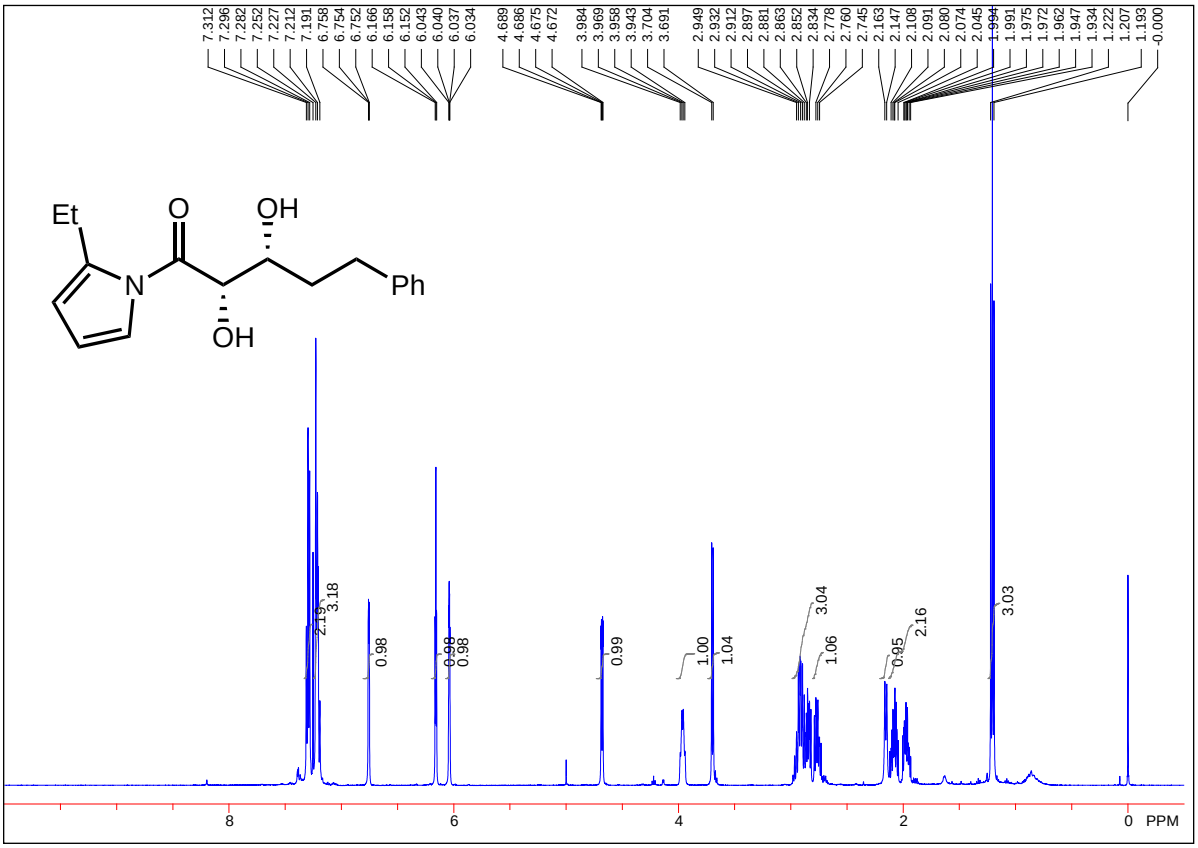


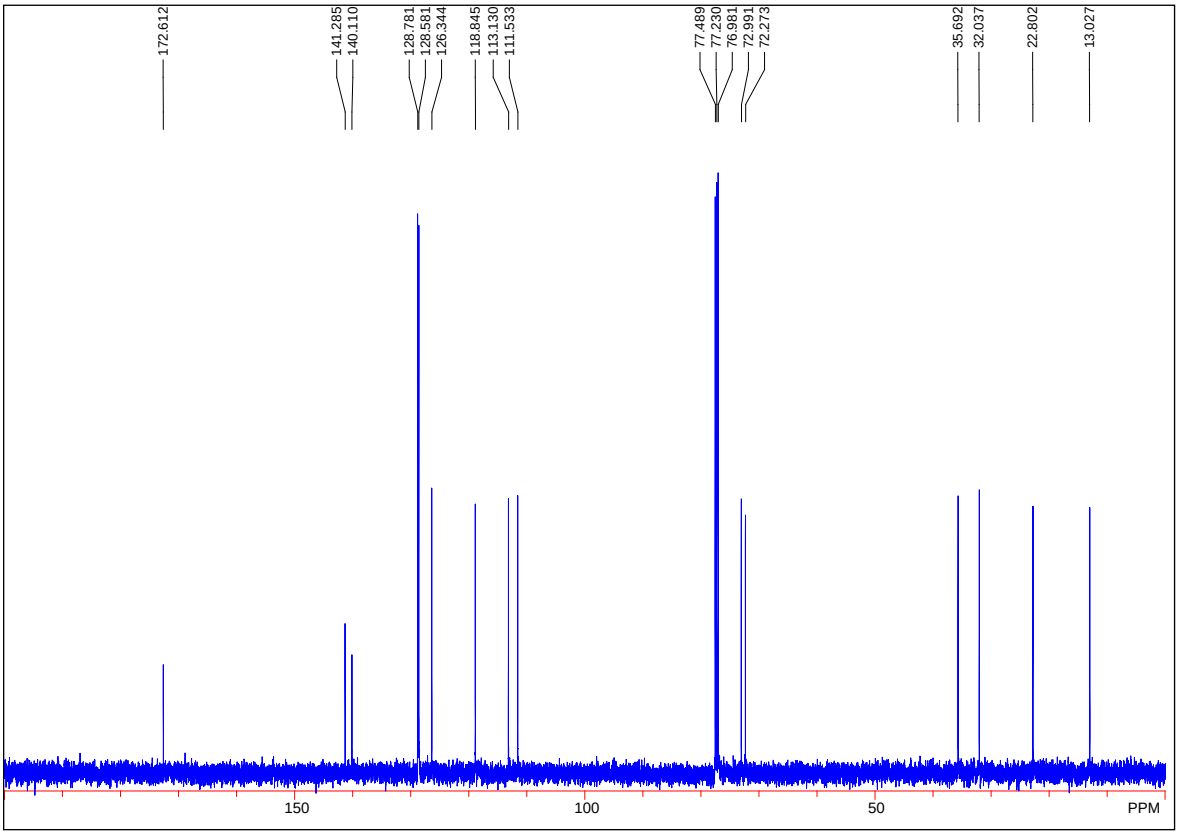
(2R,3S)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxyheptan-1-one (3g):


Et
O
OH
N
Me
OH
7.263
6.948
6.946
6.942
6.940
6.245
6.238
6.232
6.073
6.070
6.066
6.064
5.300
4.718
4.706
3.958
3.945
3.931
3.918
3.677
3.663
2.973
2.958
2.941
2.924
2.906
2.058
2.042
1.702
1.685
1.679
1.673
1.667
1.655
1.497
1.481
1.473
1.418
1.403
1.394
1.370
1.364
1.357
1.240
1.225
1.210
0.944
0.930
1.01
1.04
1.02
1.01
1.02
1.00
2.10
1.04
2.42
4.00
3.15
3.08
8
6
4
2
0
PPM

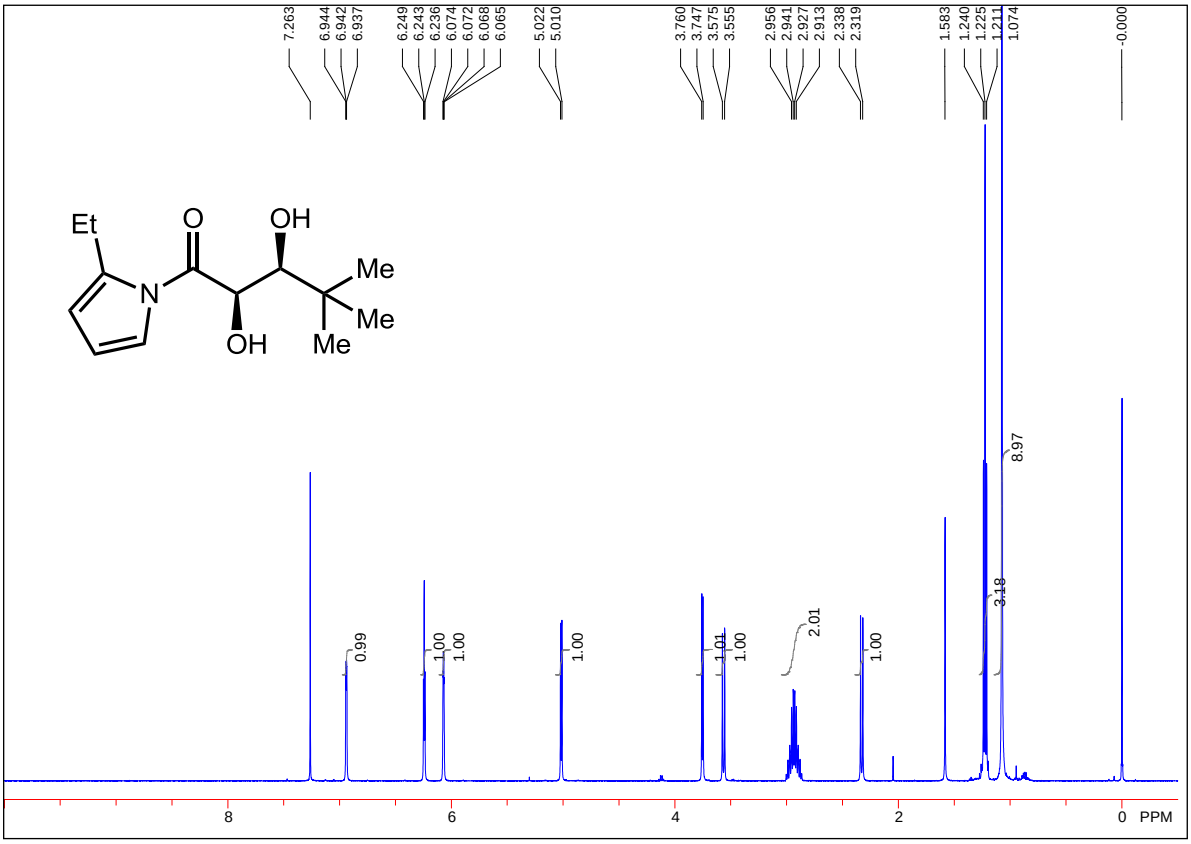

172.861
140.177
118.827
113.184
111.507
77.488
77.230
76.980
73.054
72.945
34.162
28.068
22.809
14.206
13.049
150
100
50
PPM

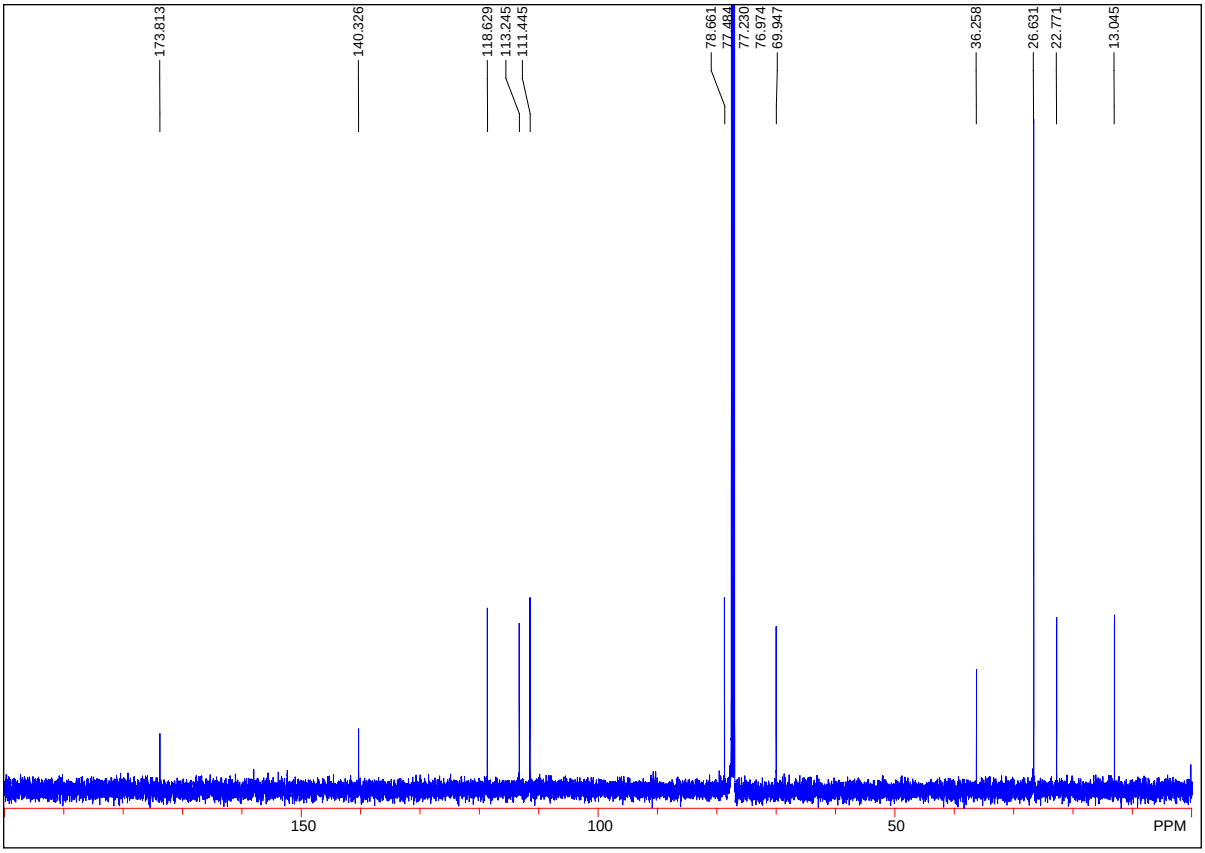
(2R,3S)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxy-5-phenylpentan-1-one (3h):



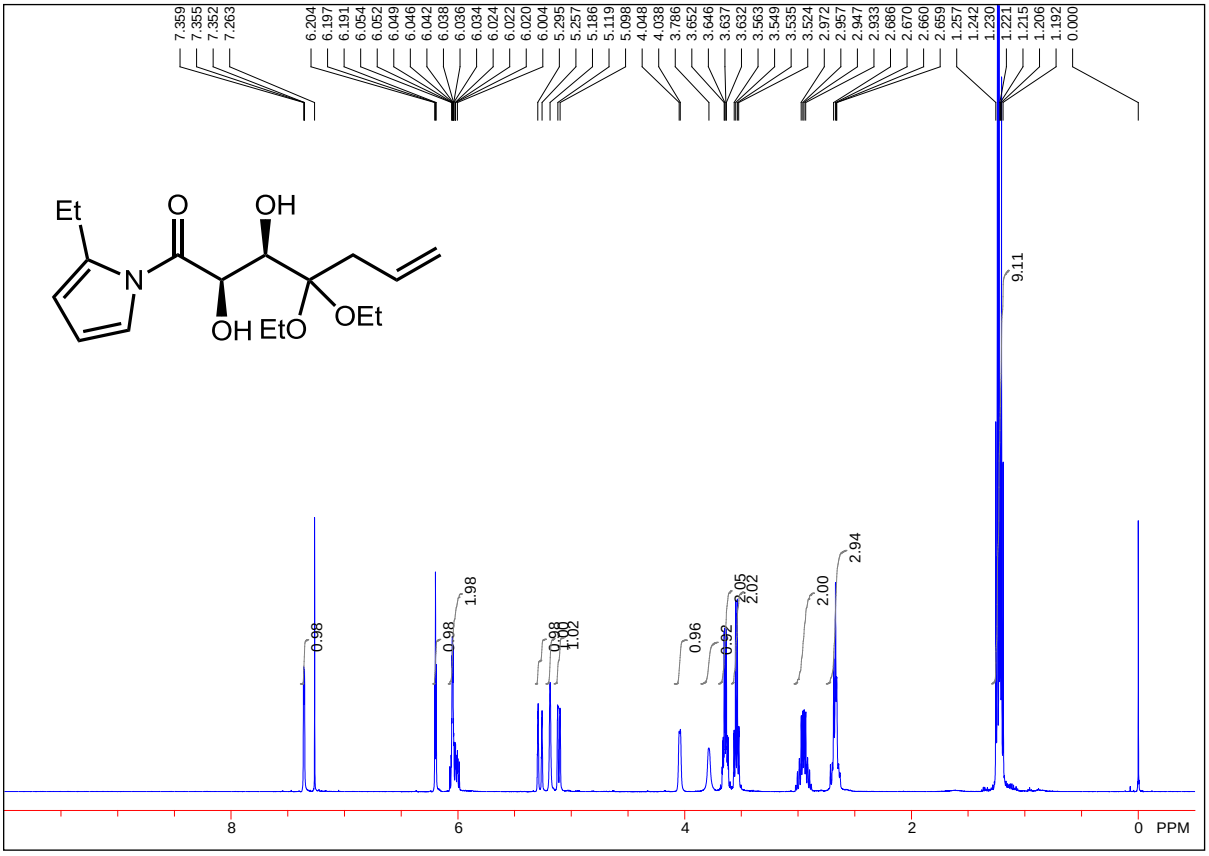


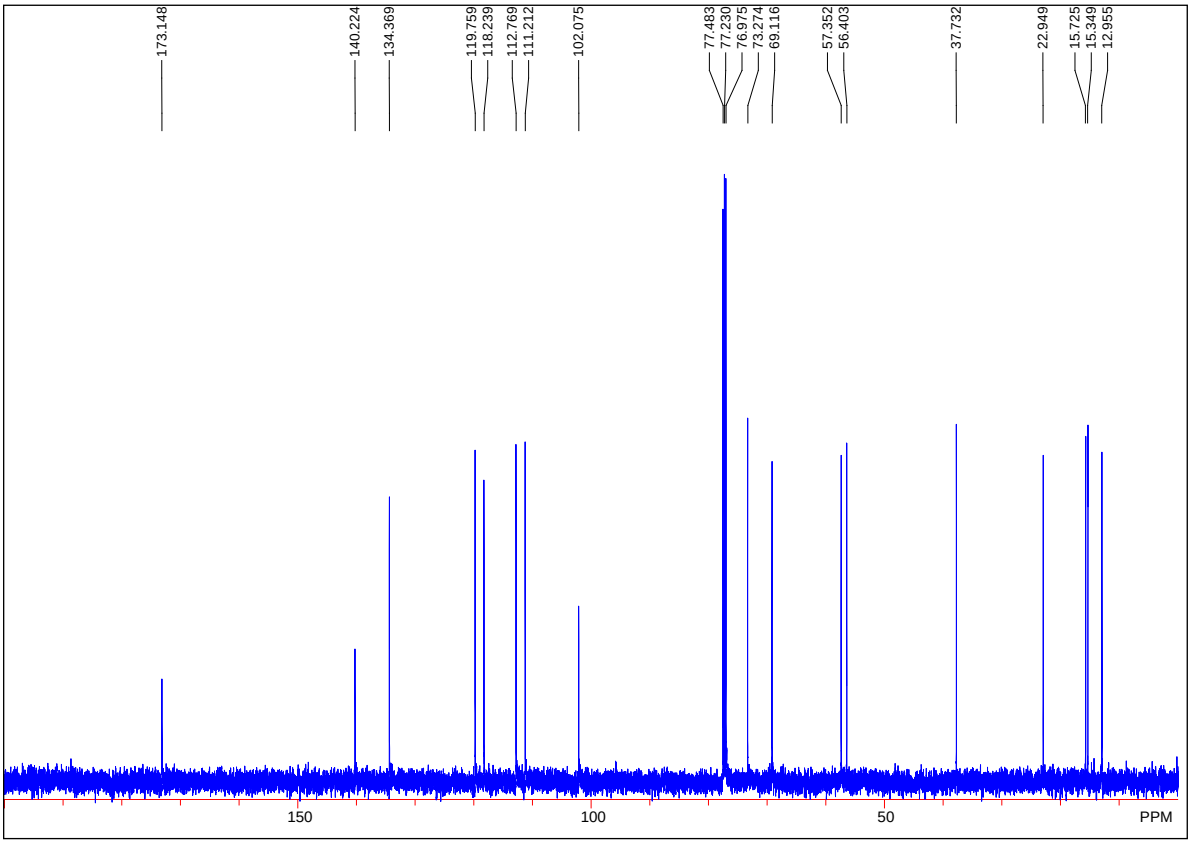
(2R,3S)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxy-4,4-dimethylpentan-1-one (3j):



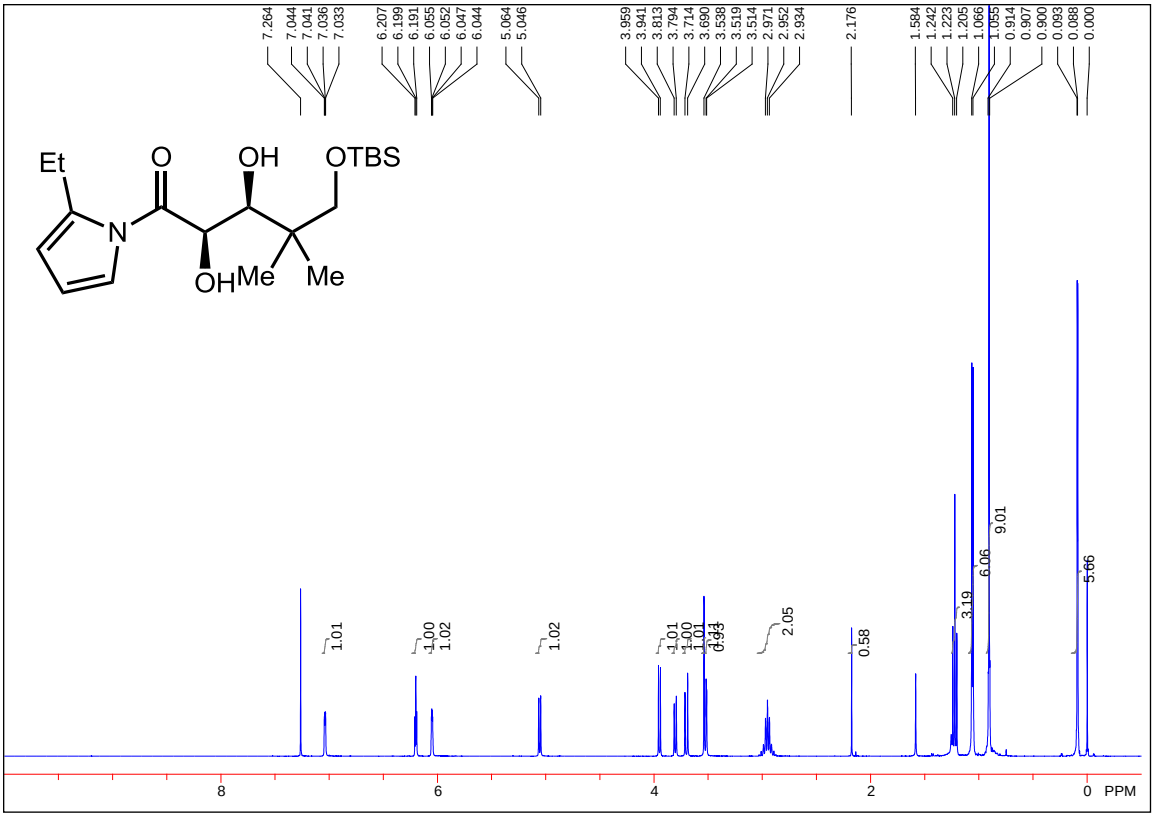


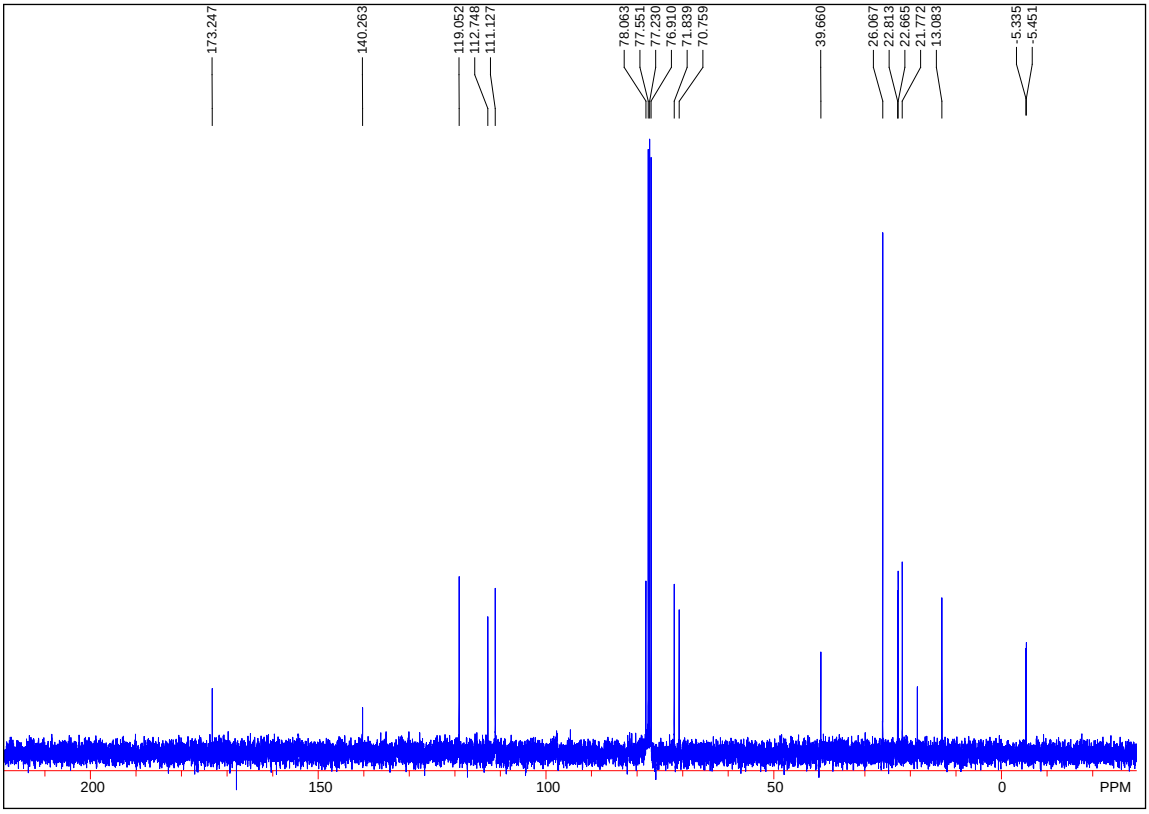
(2R,3R)-4,4-diethoxy-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxyhept-6-en-1-one (3k):



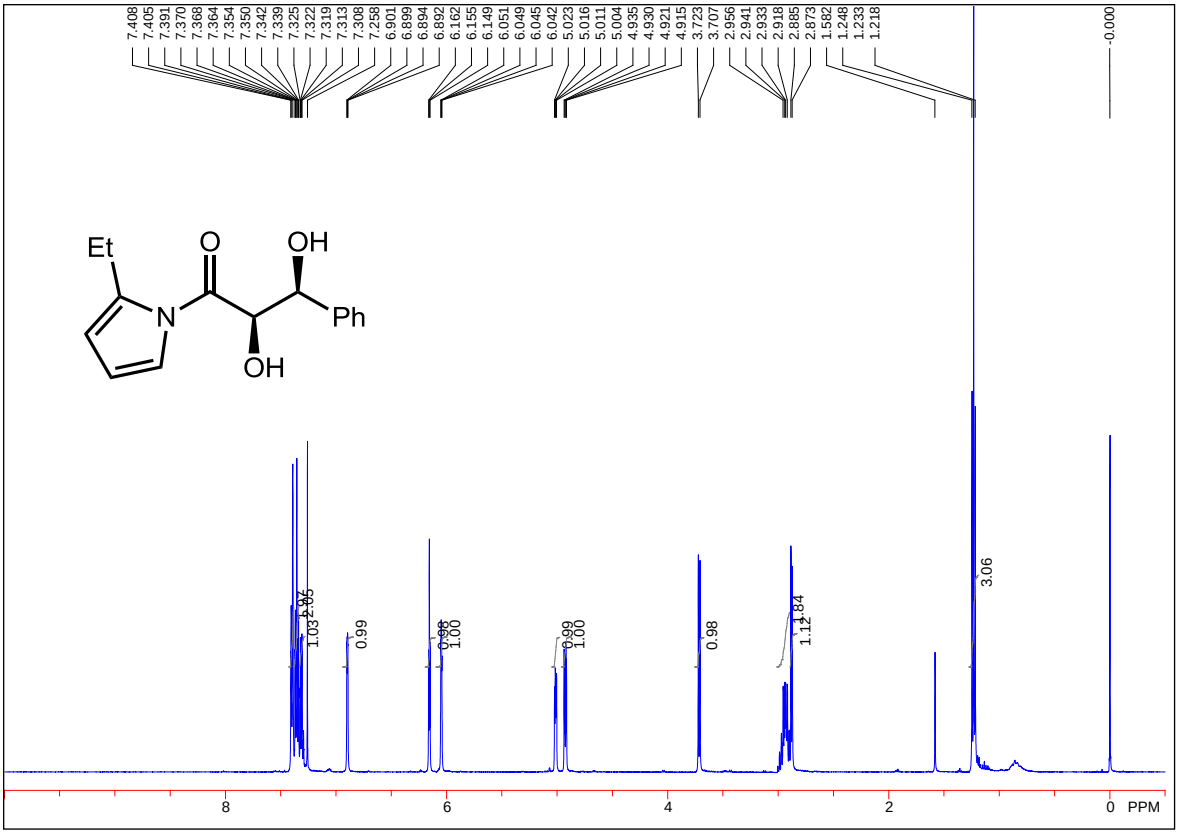


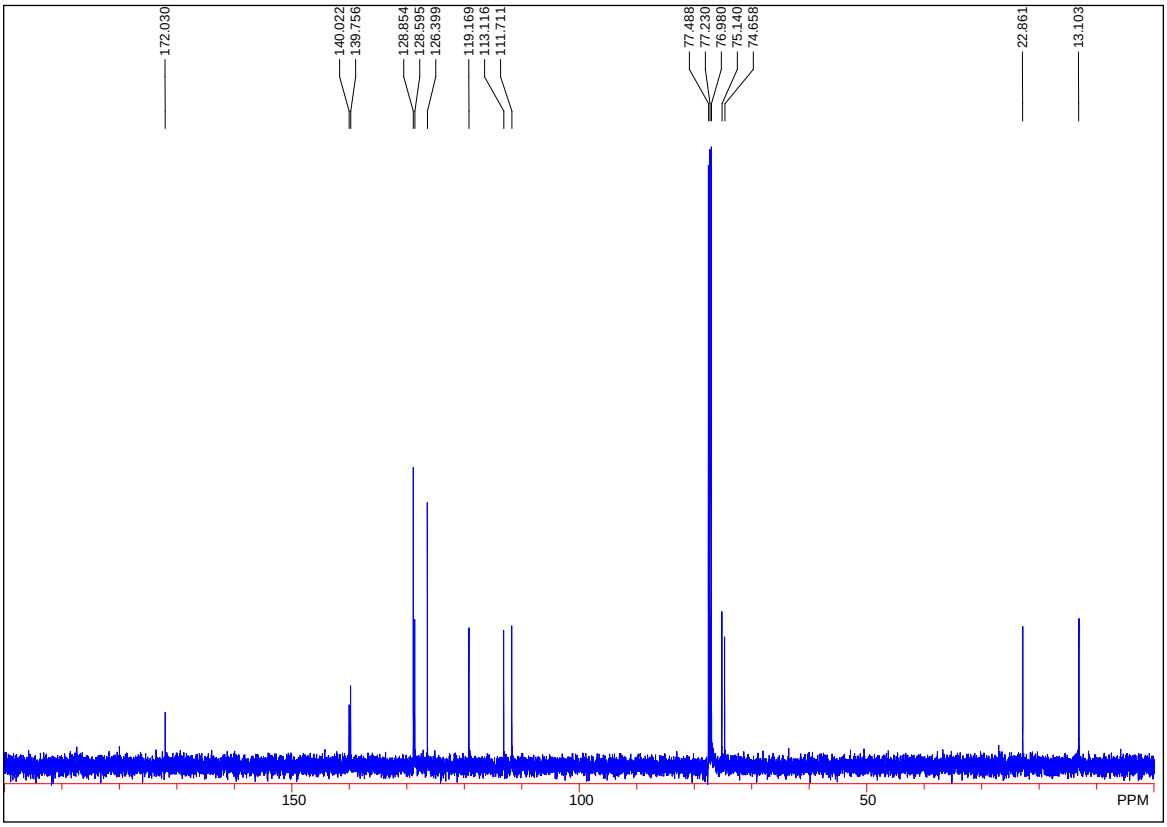
(2R,3S)-5-((tert-butyldimethylsilyl)oxy)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxy-4,4-dimethylpentan-1-one (3l):



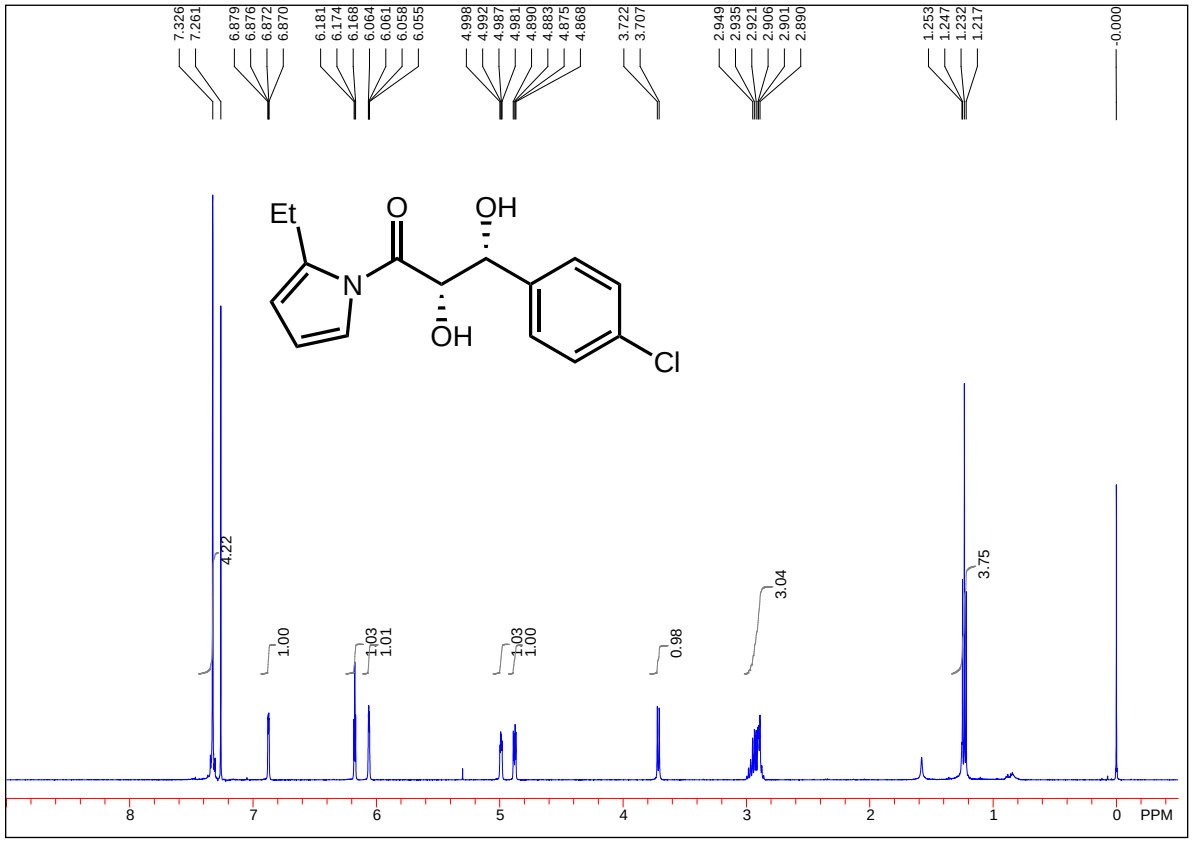


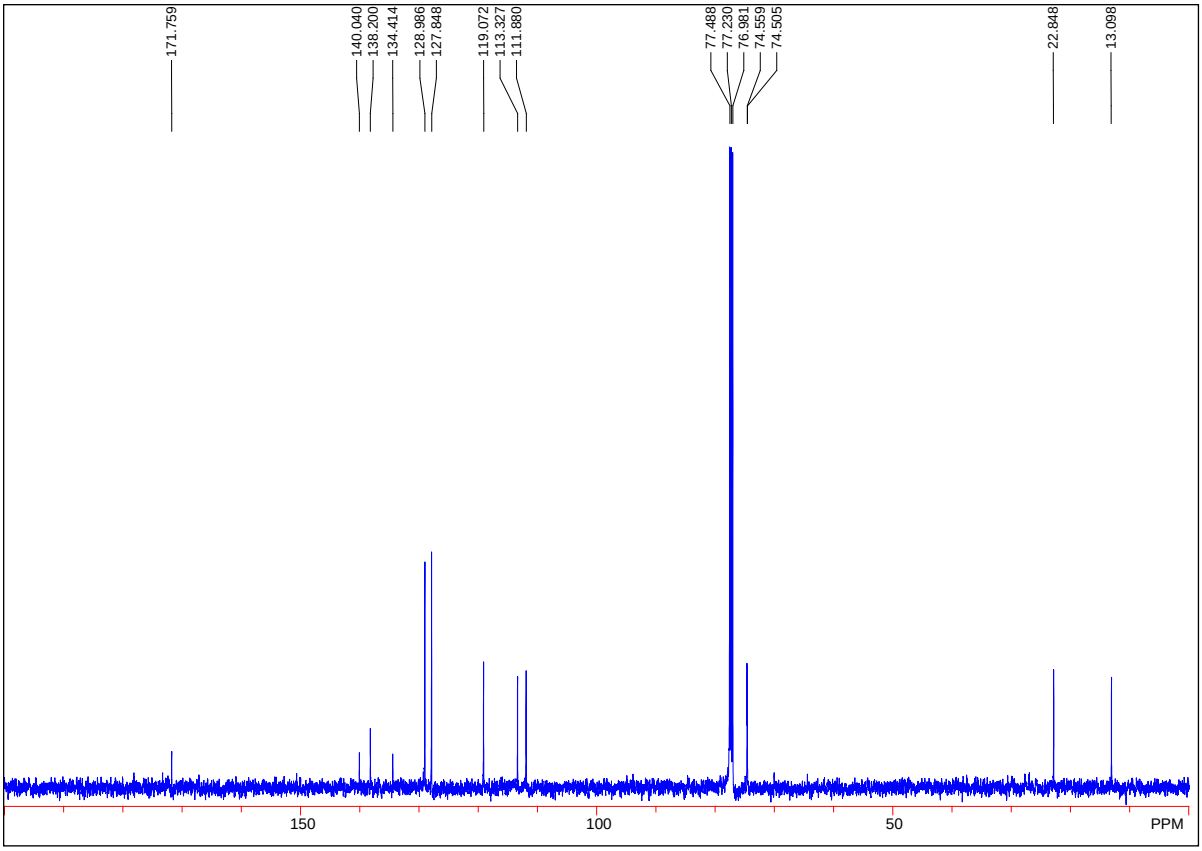
(2R,3S)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxy-3-phenylpropan-1-one (3m):


7.408
7.405
7.391
7.370
7.368
7.364
7.354
7.350
7.342
7.339
7.325
7.322
7.319
7.313
7.308
7.258
6.901
6.899
6.894
6.892
6.162
6.155
6.149
6.051
6.049
6.045
6.042
5.023
5.016
5.011
5.004
4.935
4.930
4.921
4.915
3.723
3.707
2.956
2.941
2.933
2.918
2.885
2.873
1.582
1.248
1.233
1.218
-0.000
Et
O
OH
N
Ph
OH
1.03
2.05
0.99
0.98
1.00
0.99
1.00
0.98
1.12
1.84
3.06
8
6
4
2
0
PPM

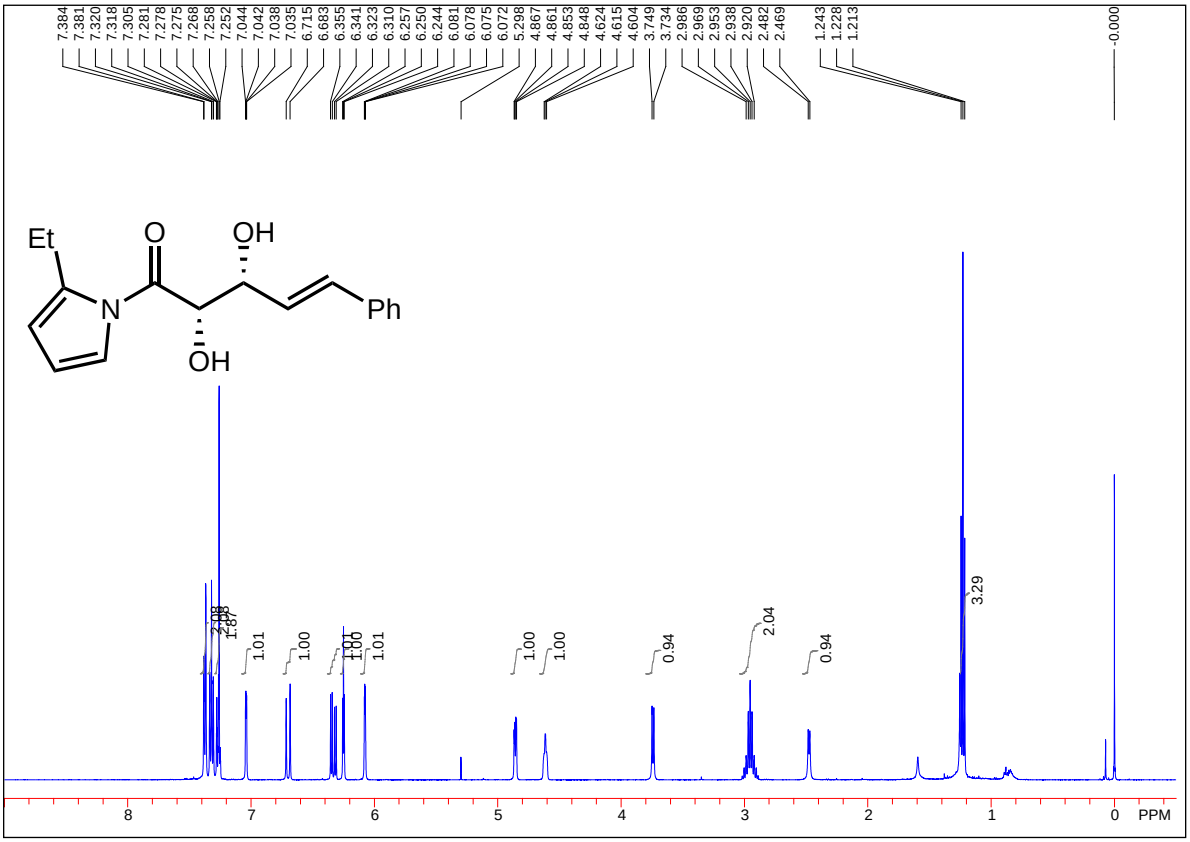

172.030
140.022
139.756
128.854
128.595
126.399
119.169
113.116
111.711
77.488
77.230
76.980
75.140
74.658
22.861
13.103
150
100
50
PPM

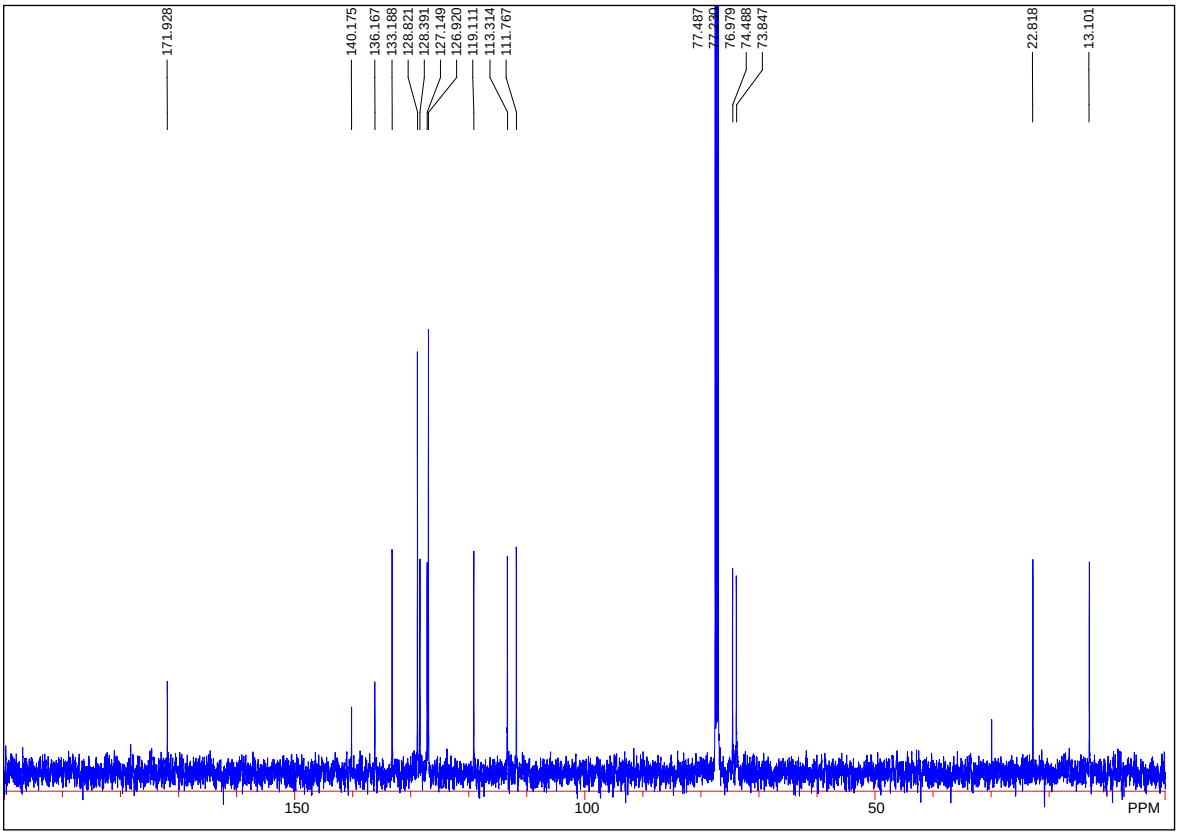
(2R,3S)-3-(4-chlorophenyl)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxypropan-1-one (3n):



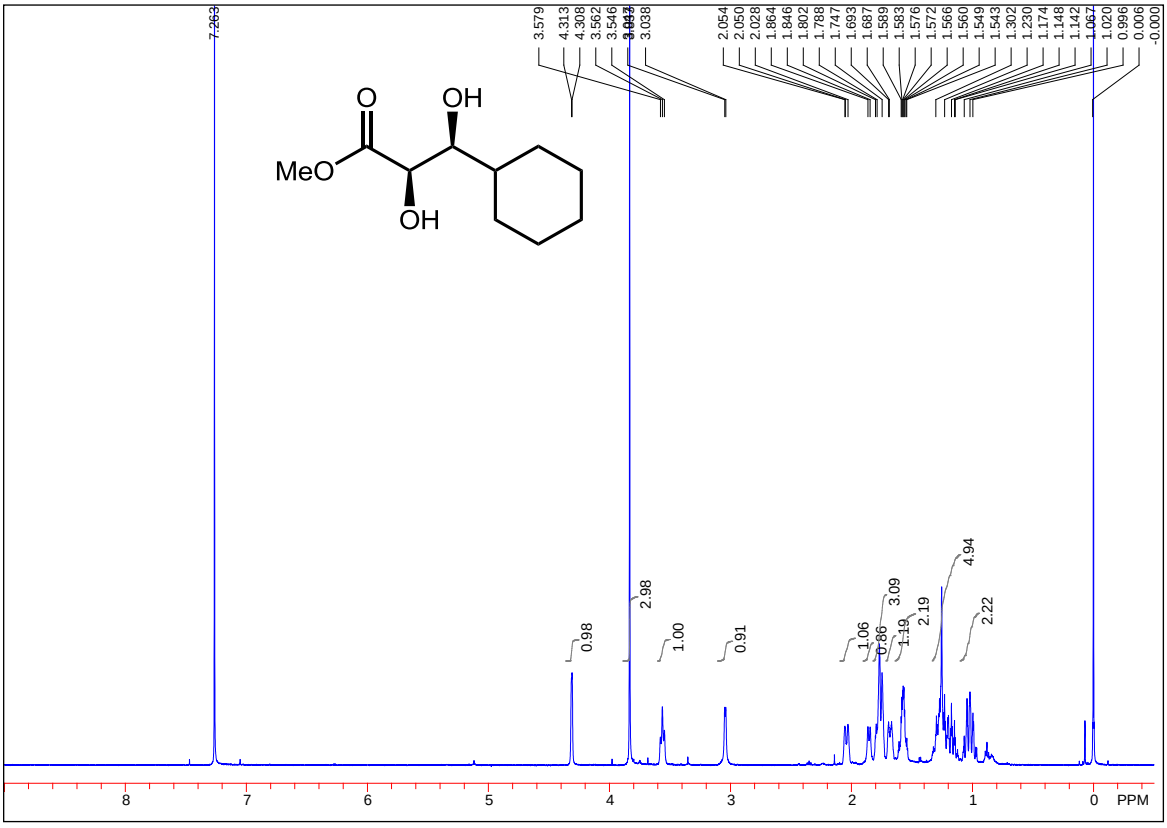


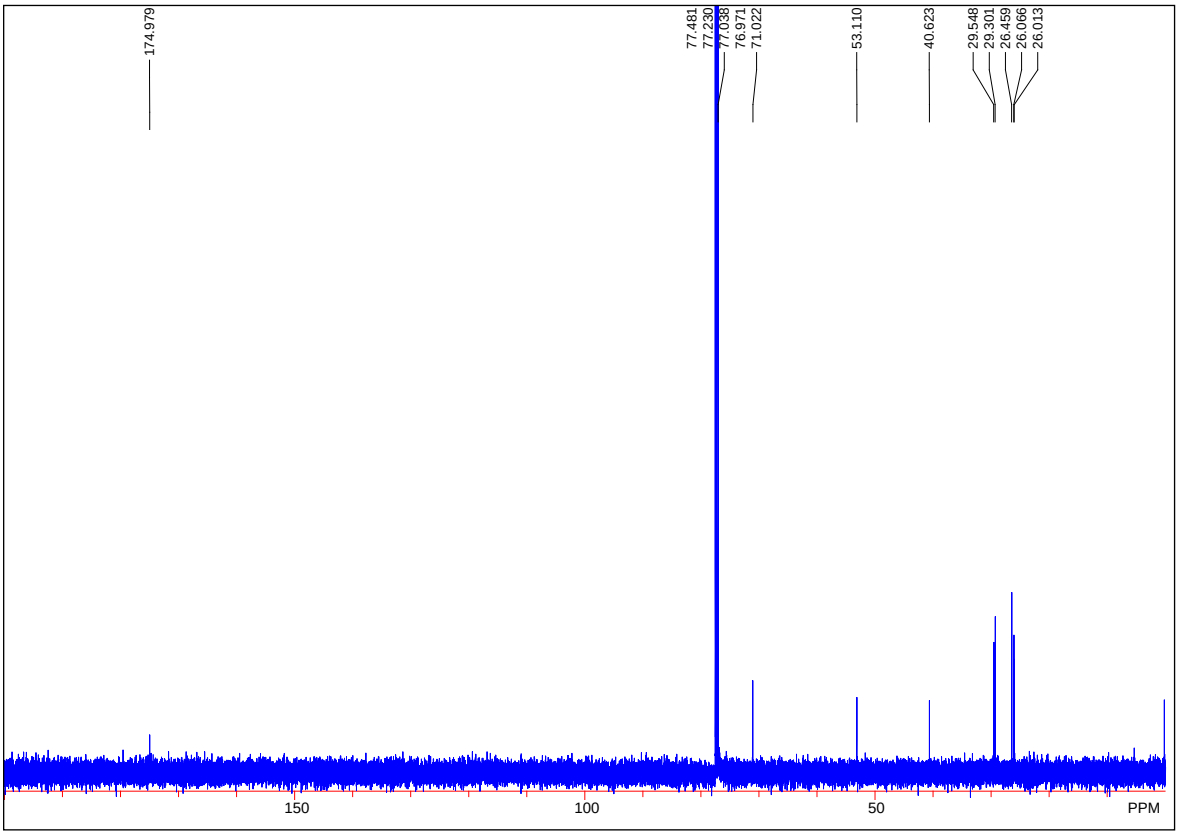
(2R,3S,E)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxy-5-phenylpent-4-en-1-one (3o):



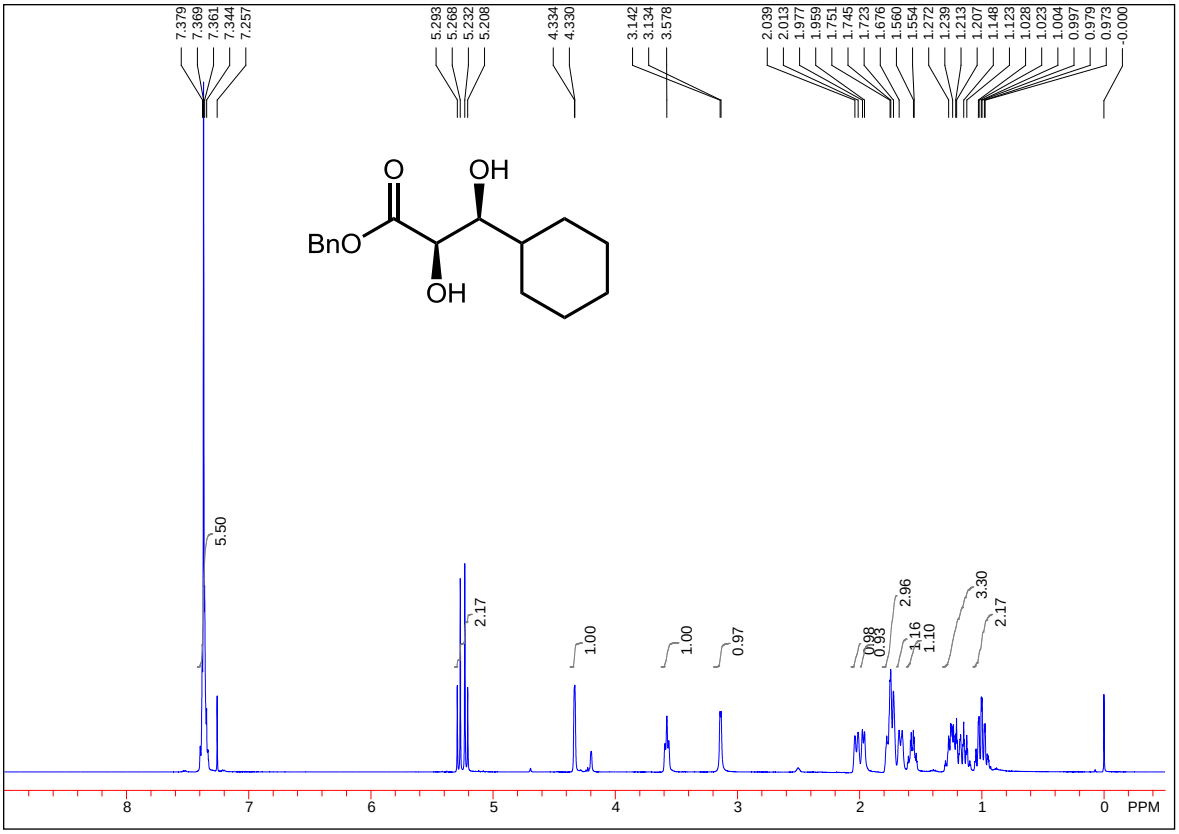


(2R,3S)-methyl 3-cyclohexyl-2,3-dihydroxypropanoate (5a):





(2R,3S)-benzyl 3-cyclohexyl-2,3-dihydroxypropanoate (5b):


7.379
7.369
7.361
7.344
7.257
5.293
5.268
5.232
5.208
4.334
4.330
3.142
3.134
3.578
2.039
2.013
1.977
1.959
1.751
1.745
1.723
1.676
1.560
1.554
1.272
1.239
1.213
1.207
1.148
1.123
1.028
1.023
1.004
0.997
0.979
0.973
-0.000
O
OH
BnO
OH
5.50
2.17
1.00
1.00
0.97
0.98
0.93
2.96
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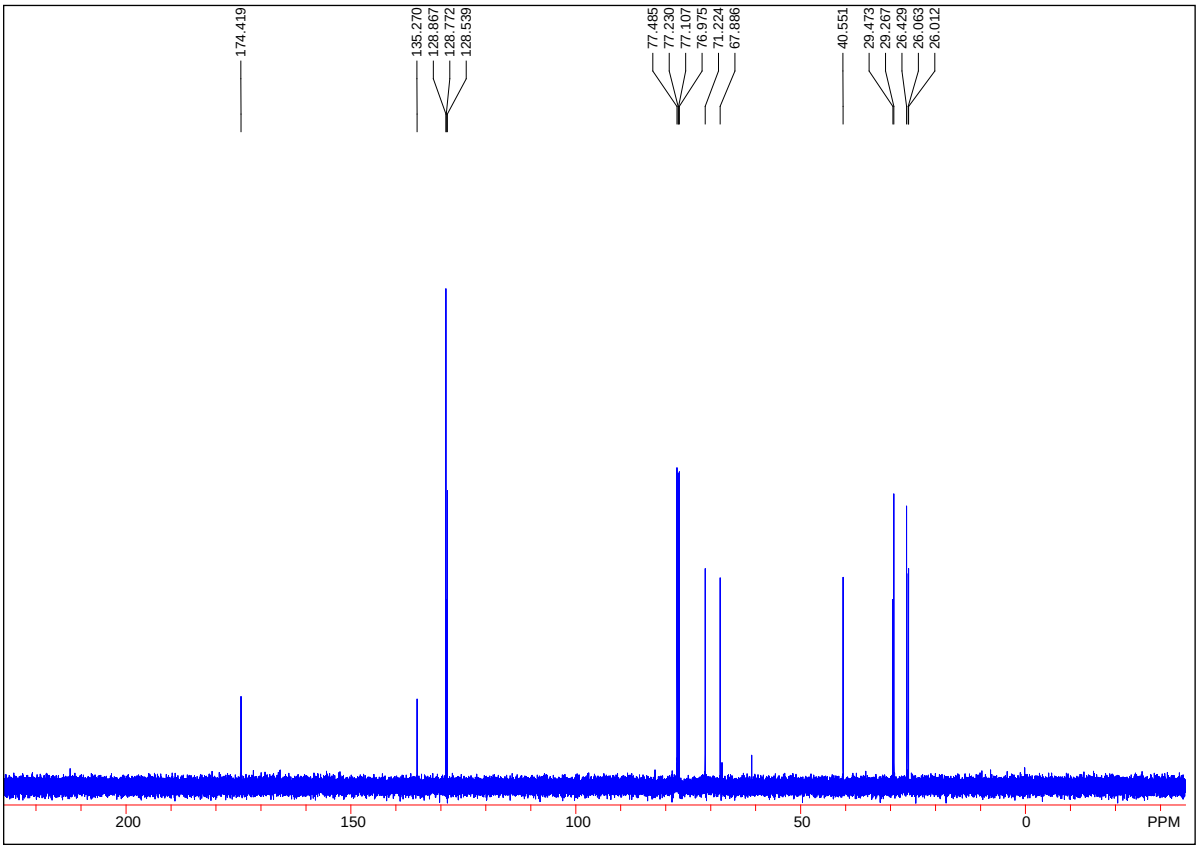
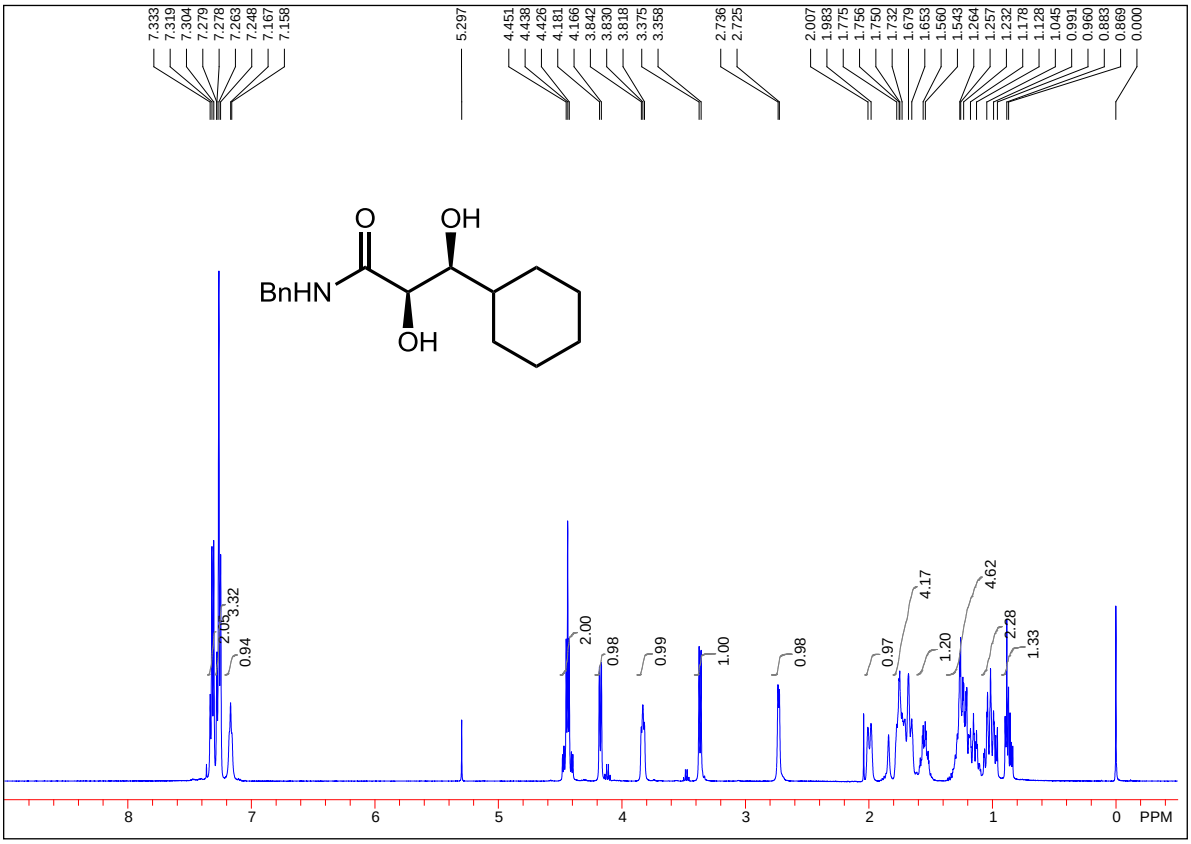
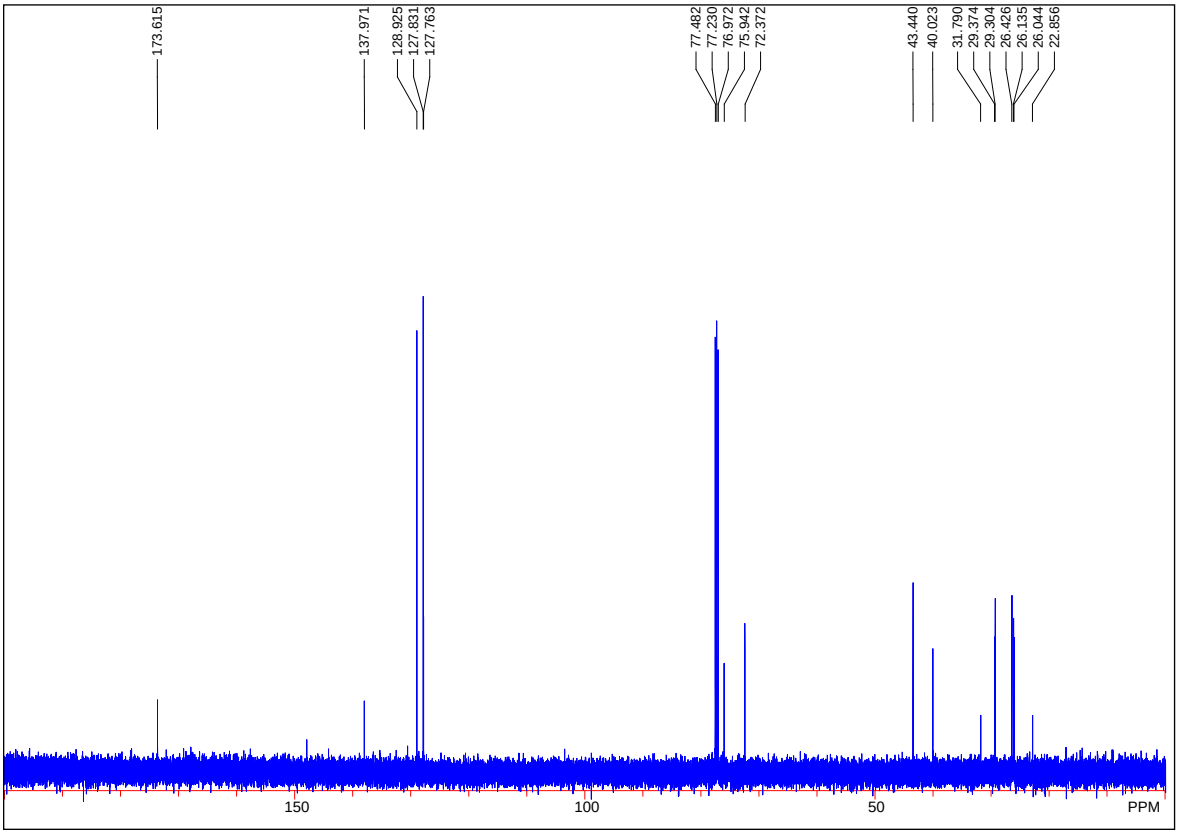
1.10
3.30
2.17
8
7
6
5
4
3
2
1
0
PPM

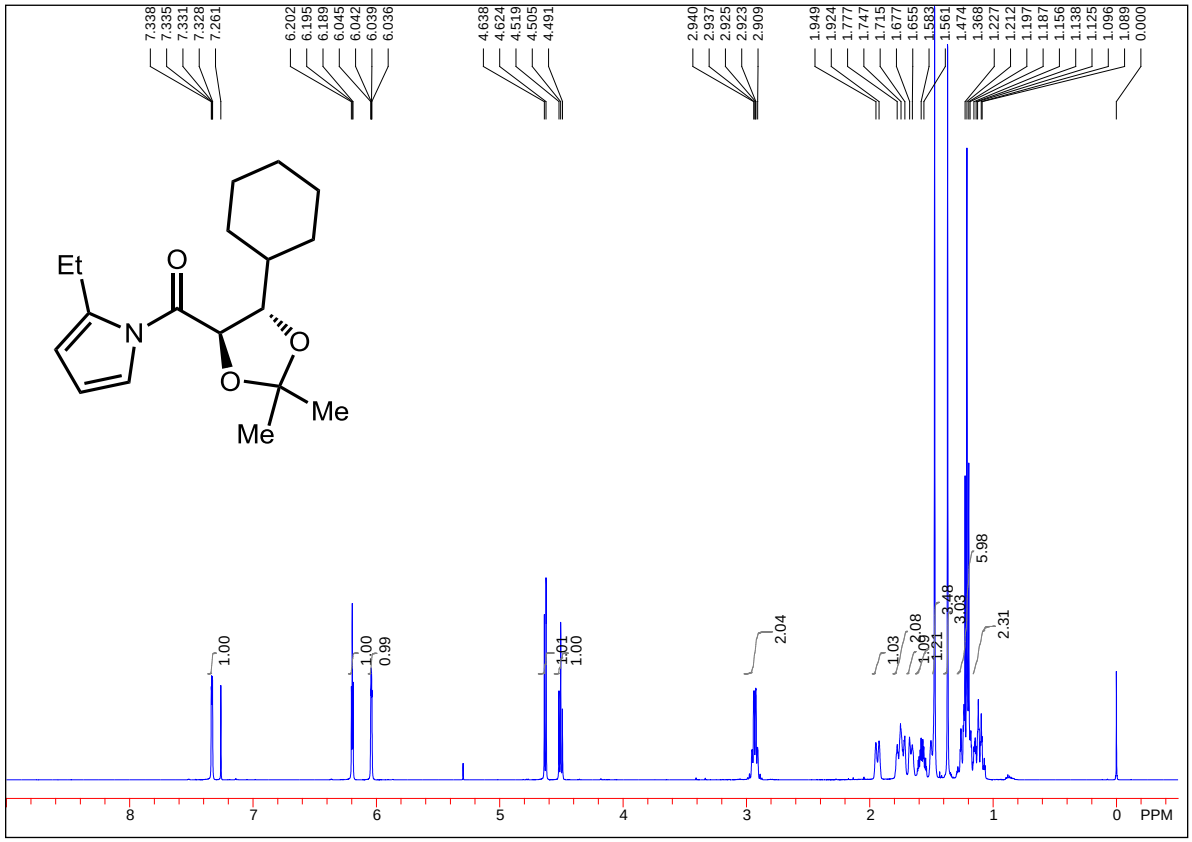

174.419
135.270
128.867
128.772
128.539
77.485
77.230
77.107
76.975
71.224
67.886
40.551
29.473
29.267
26.429
26.063
26.012
200
150
100
50
0
PPM

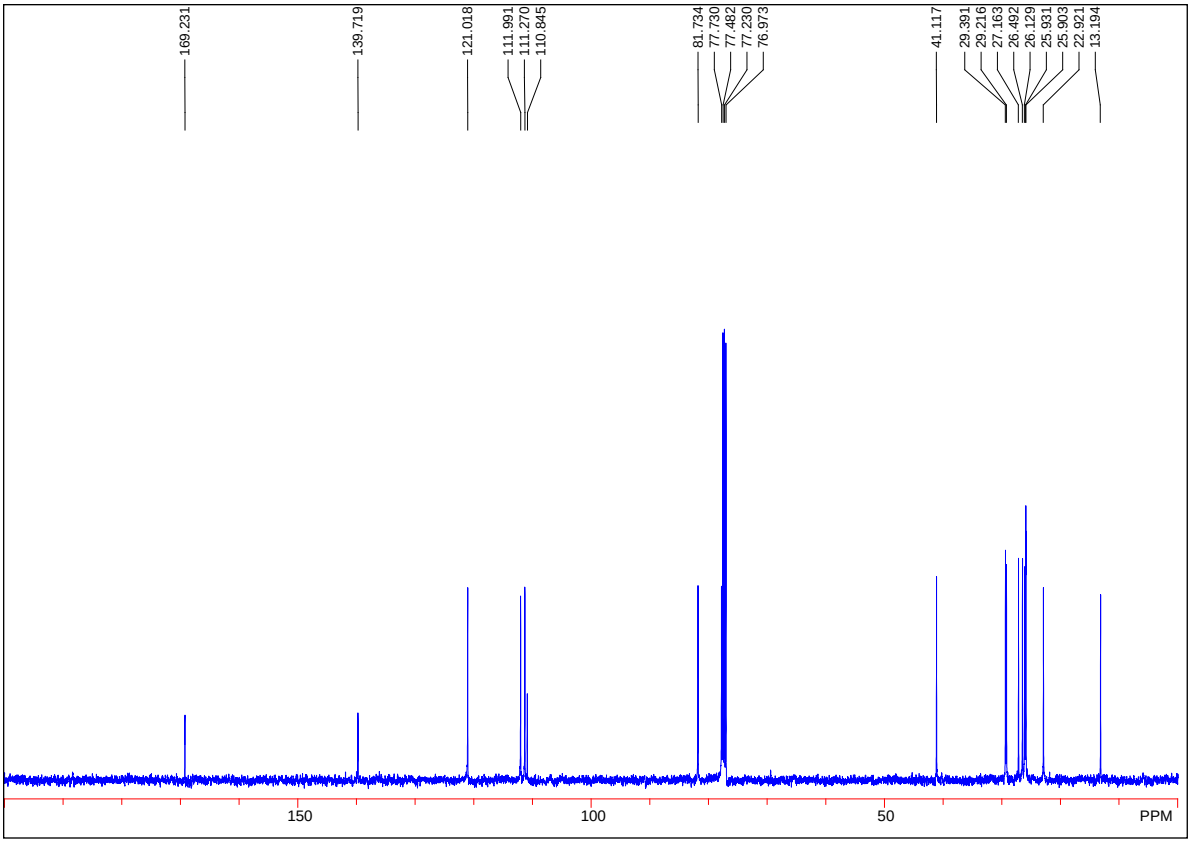
(2R,3S)-N-benzyl-3-cyclohexyl-2,3-dihydroxypropanamide (5c):



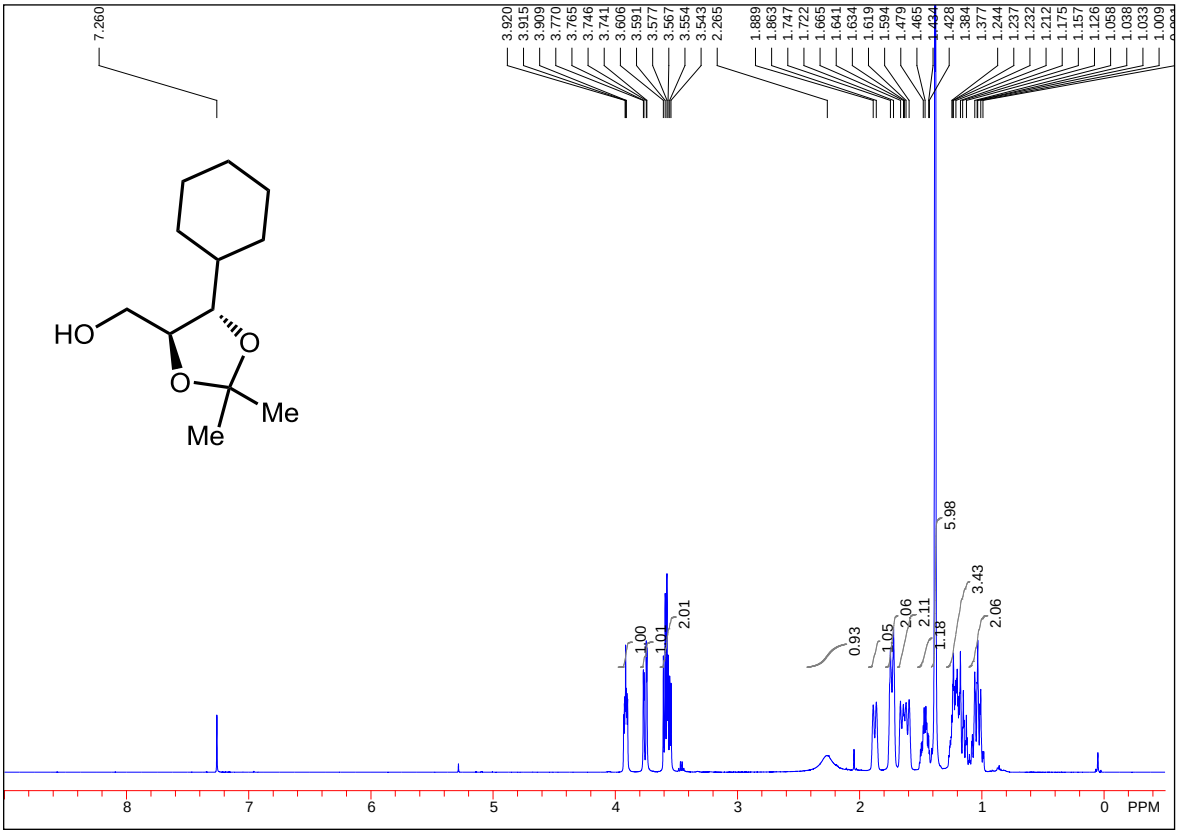


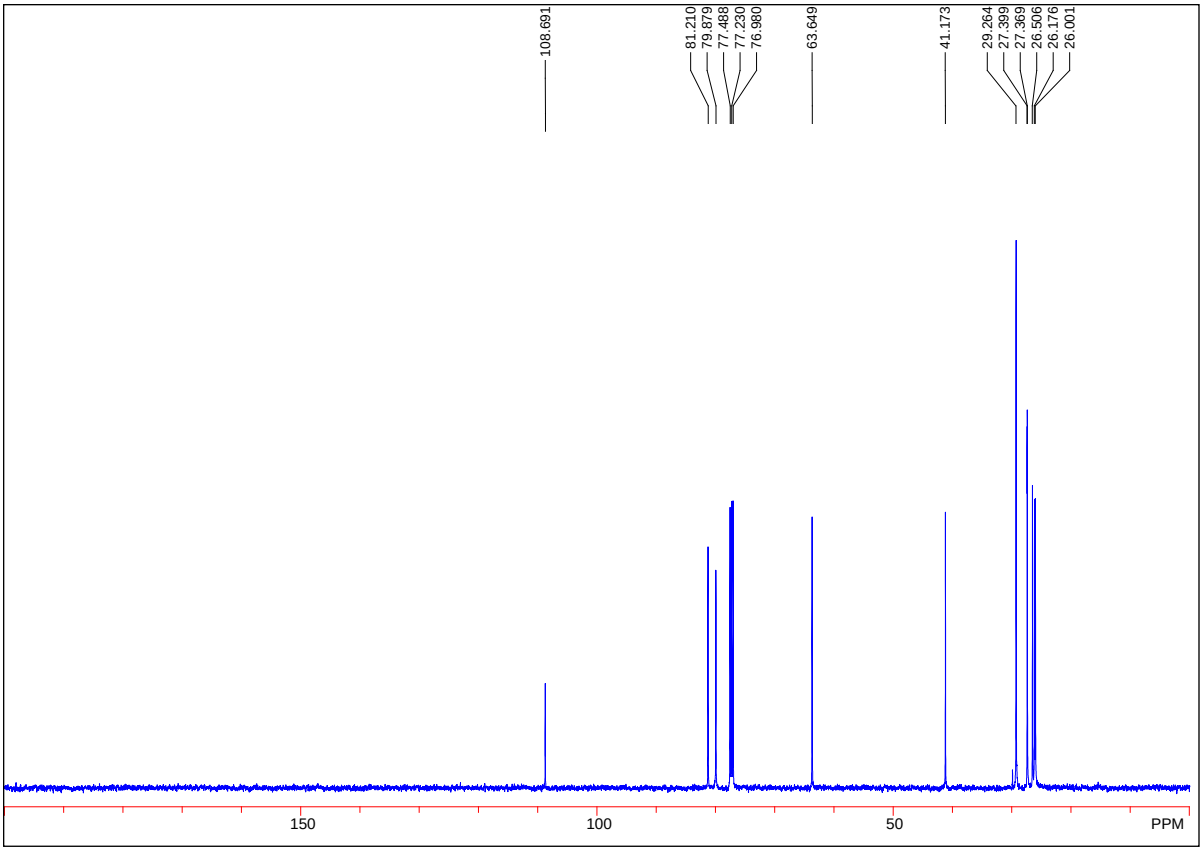
((4R,5S)-5-cyclohexyl-2,2-dimethyl-1,3-dioxolan-4-yl)(2-ethyl-1H-pyrrol-1-yl)methanone (6):



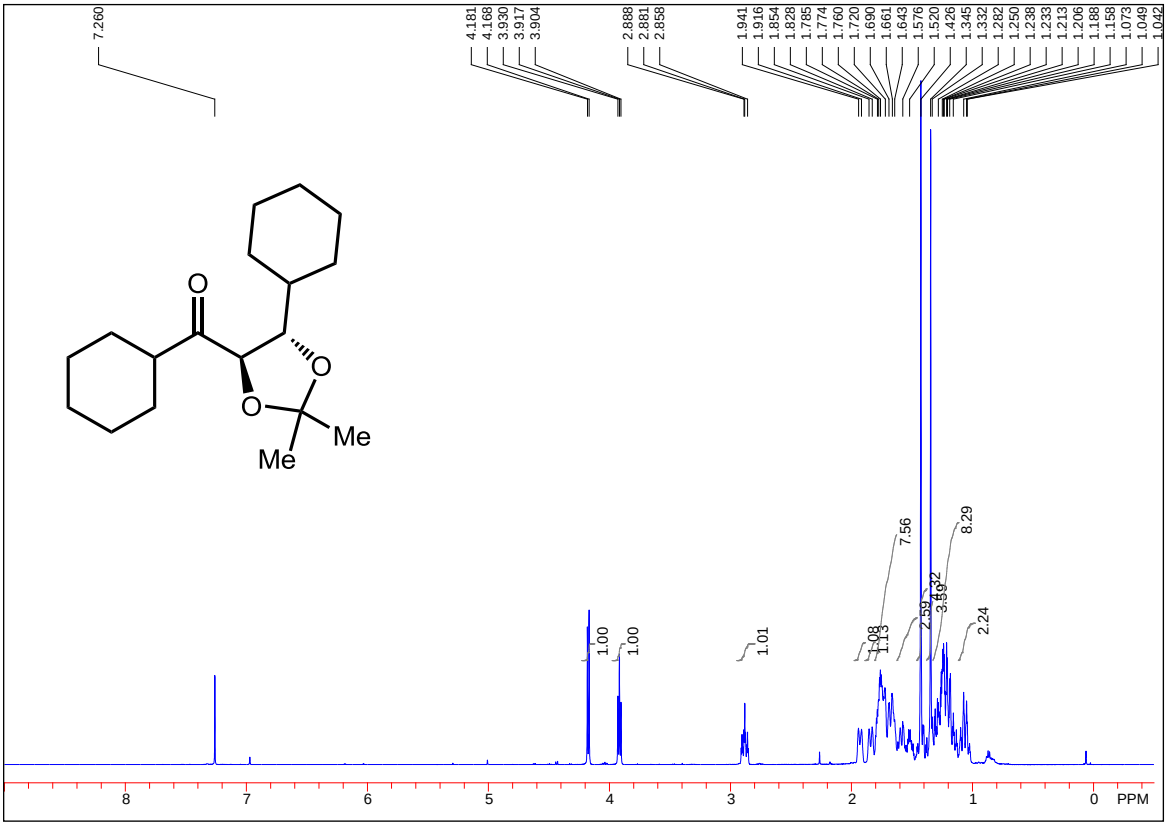


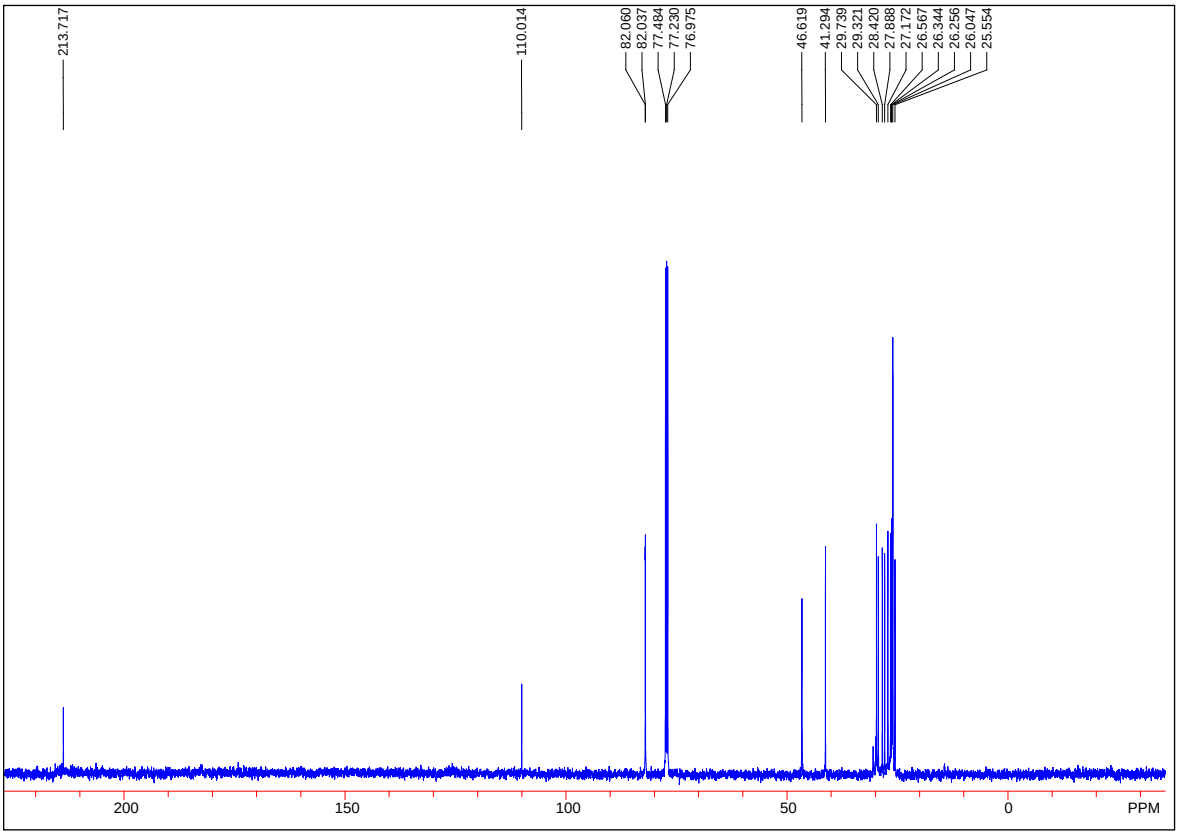
((4S,5S)-5-cyclohexyl-2,2-dimethyl-1,3-dioxolan-4-yl)methanol (7):



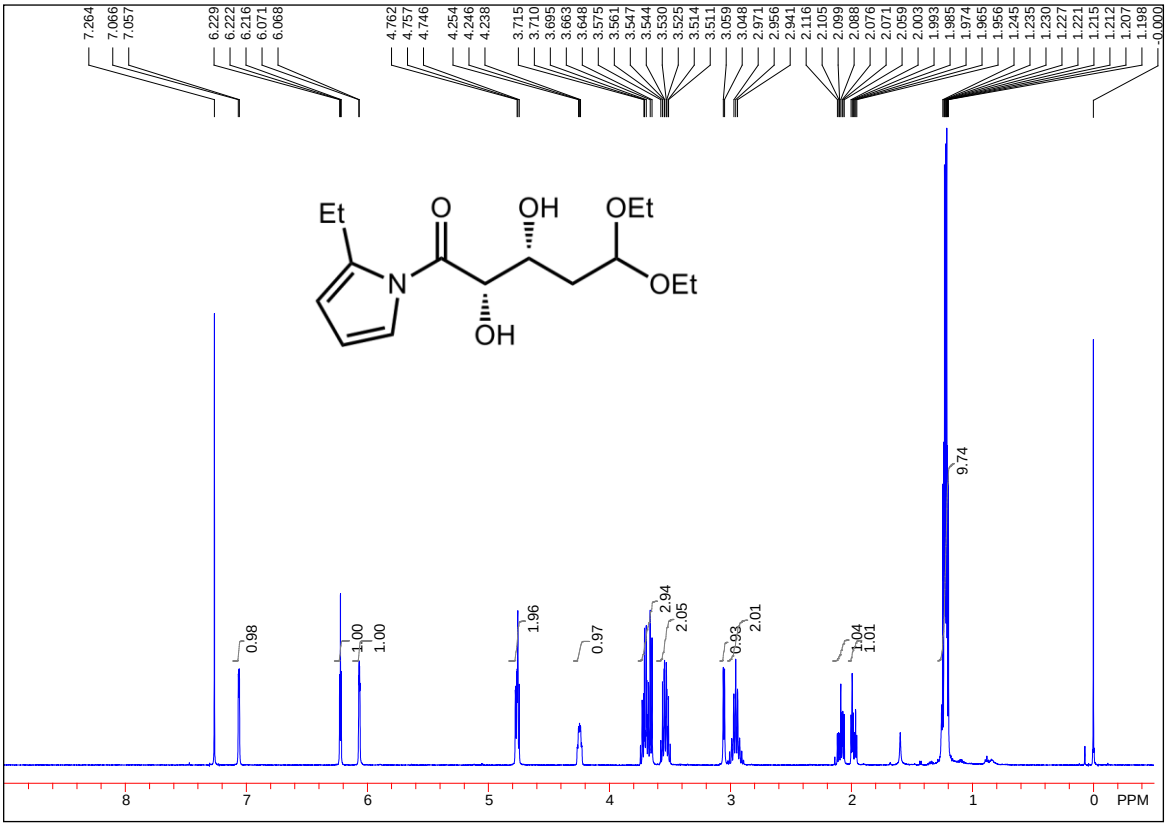


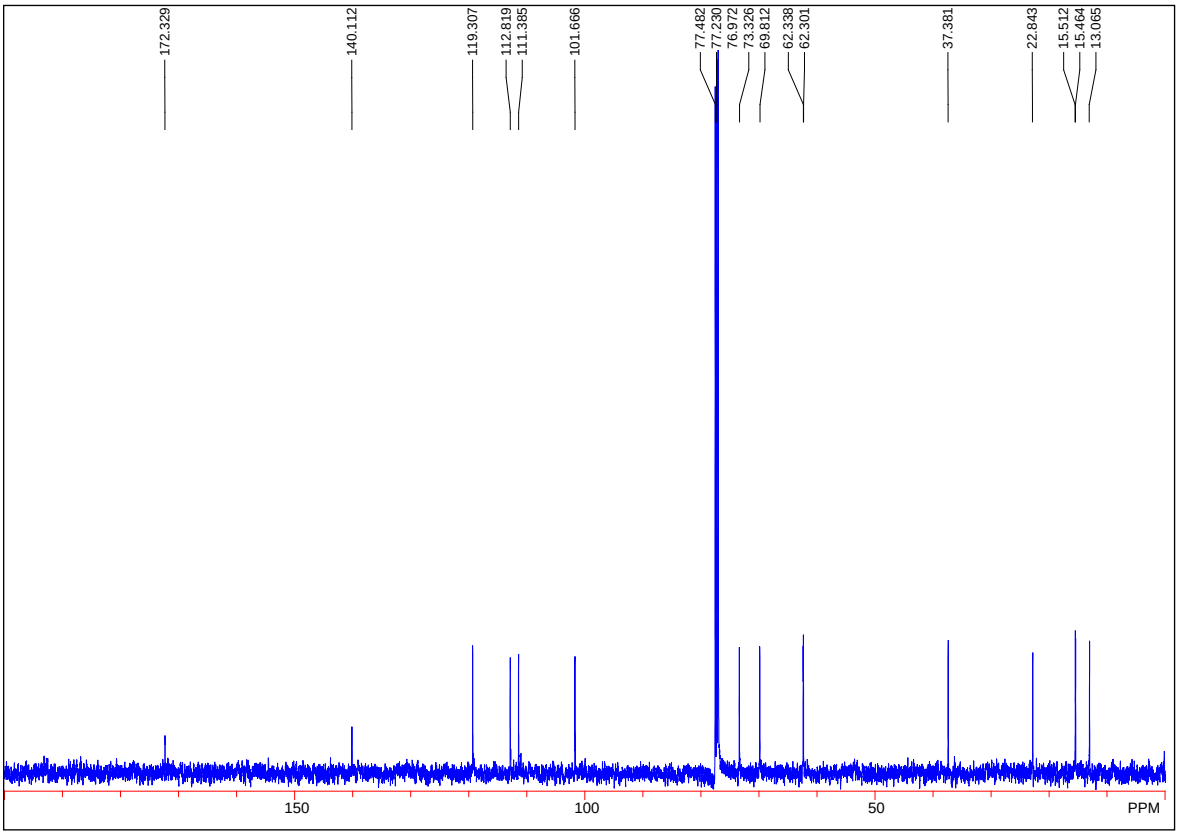
Cyclohexyl((4R,5S)-5-cyclohexyl-2,2-dimethyl-1,3-dioxolan-4-yl)methanone (8):



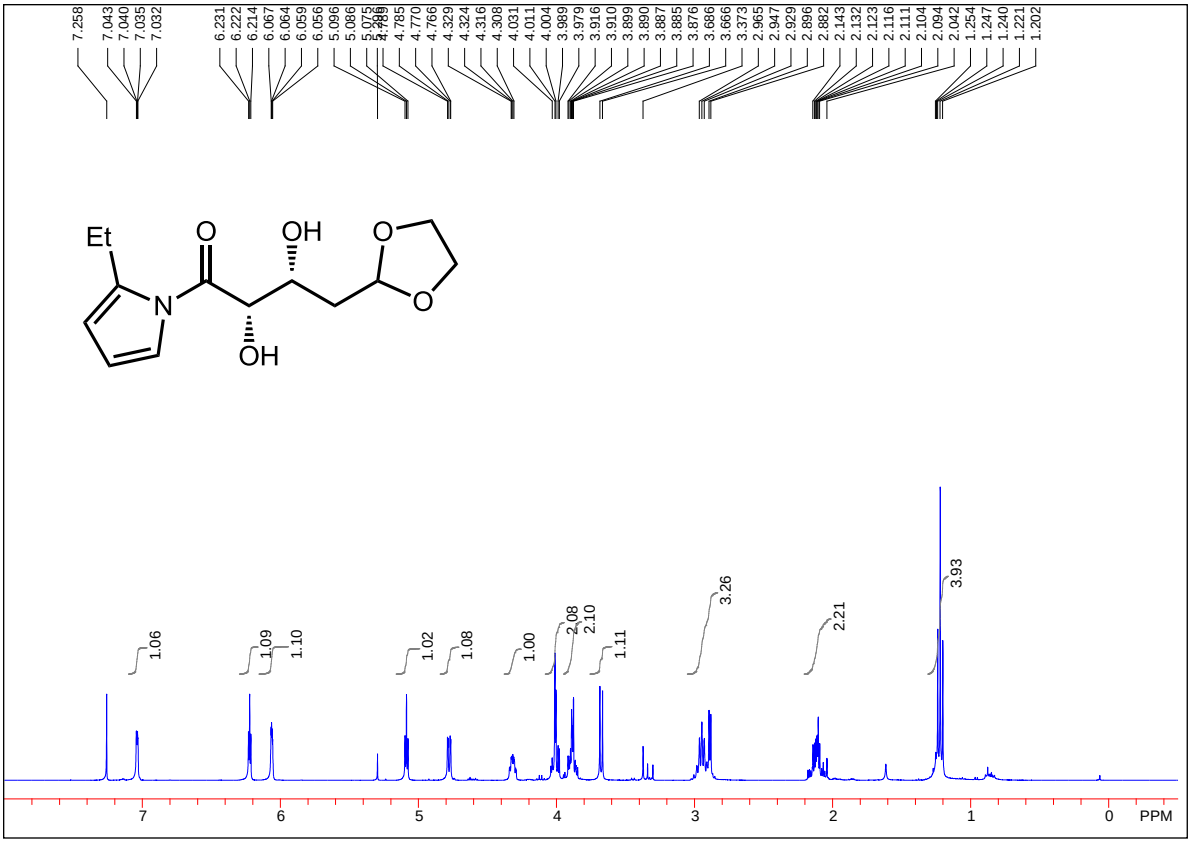


(2R,3S)-5,5-diethoxy-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxypentan-1-one (3p):





(2S,3R)-4-(1,3-dioxolan-2-yl)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxybutan-1-one (3q):


7.258
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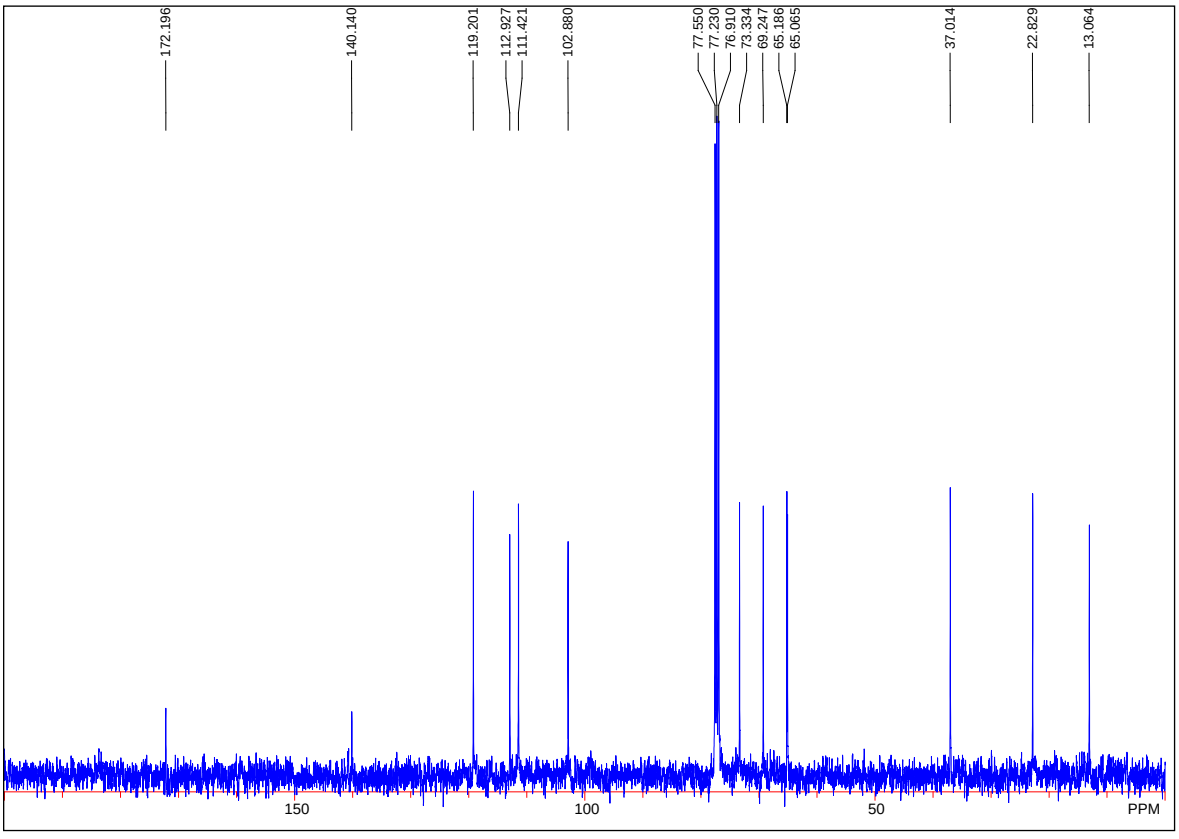
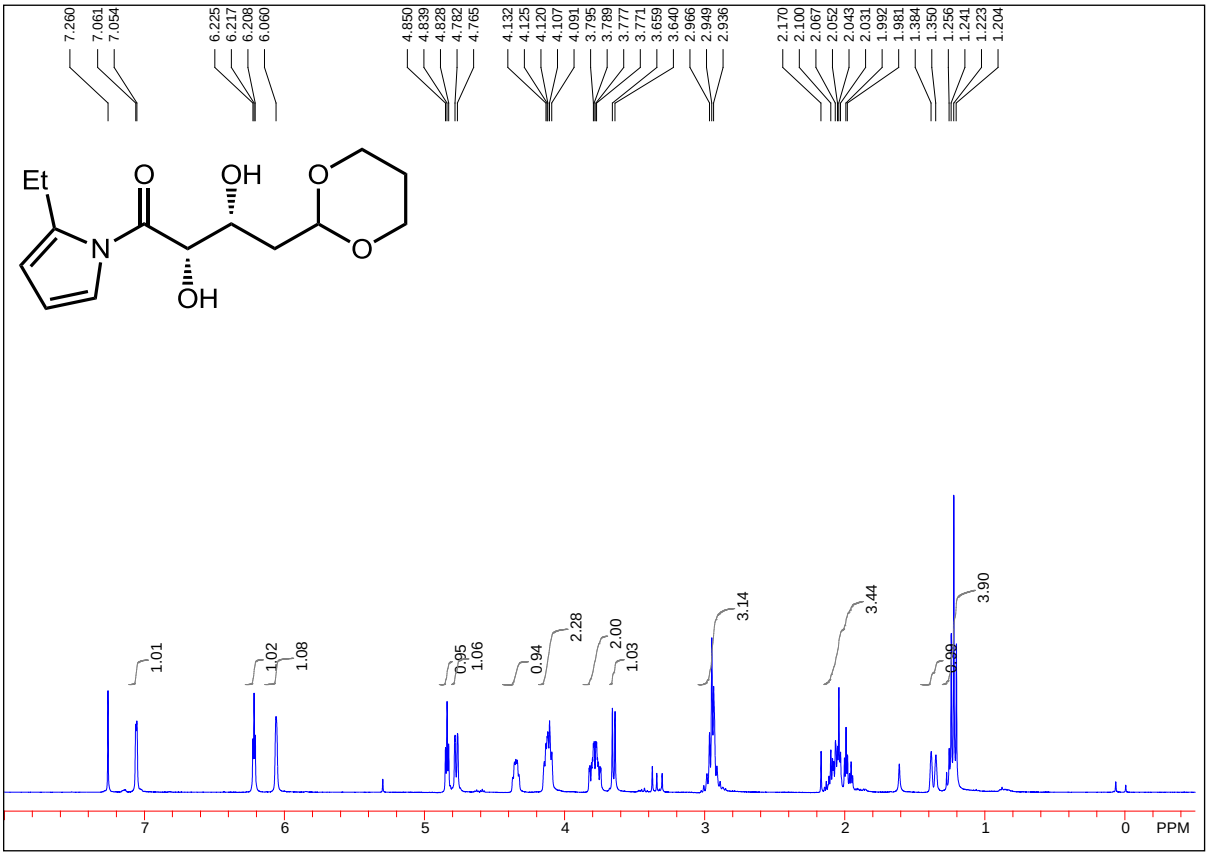
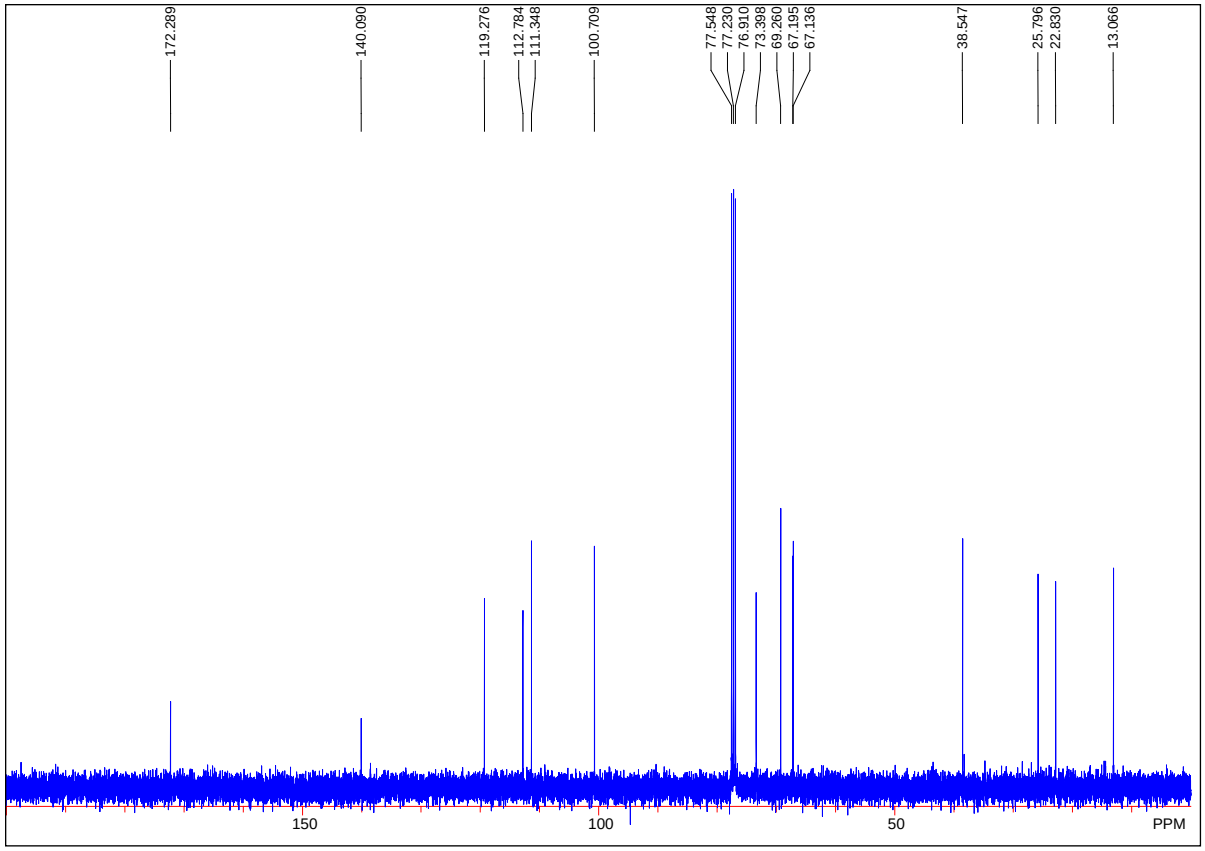
7.035
7.032
6.231
6.222
6.214
6.067
6.064
6.059
6.056
5.096
5.086
4.785
4.770
4.766
4.329
4.324
4.316
4.308
4.031
4.011
4.004
3.989
3.979
3.916
3.910
3.899
3.890
3.887
3.885
3.876
3.686
3.666
3.373
2.965
2.947
2.929
2.896
2.882
2.143
2.132
2.123
2.116
2.111
2.104
2.094
2.042
1.254
1.247
1.240
1.221
1.202
Et
O
OH
O
N
O
OH
1.06
1.09
1.10
1.02
1.08
1.00
2.08
2.10
1.11
3.26
2.21
3.93
7
6
5
4
3
2
1
0
PPM


172.196
140.140
119.201
112.927
111.421
102.880
77.550
77.230
76.910
73.334
69.247
65.186
65.065
37.014
22.829
13.064
150
100
50
PPM

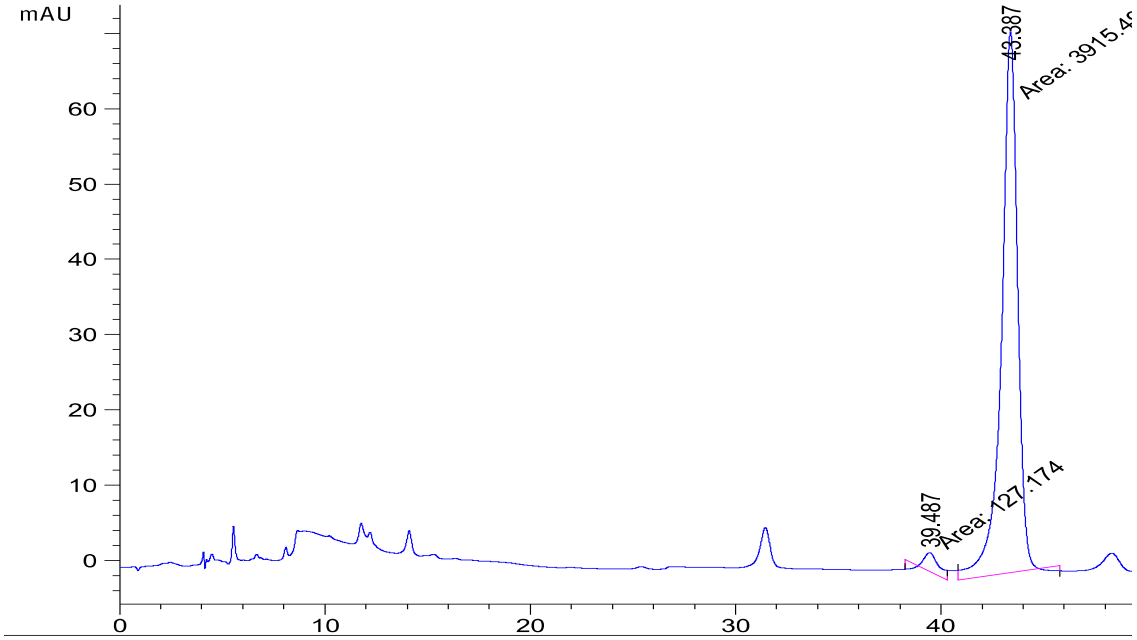
(2S,3R)-4-(1,3-dioxan-2-yl)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxybutan-1-one (3r):





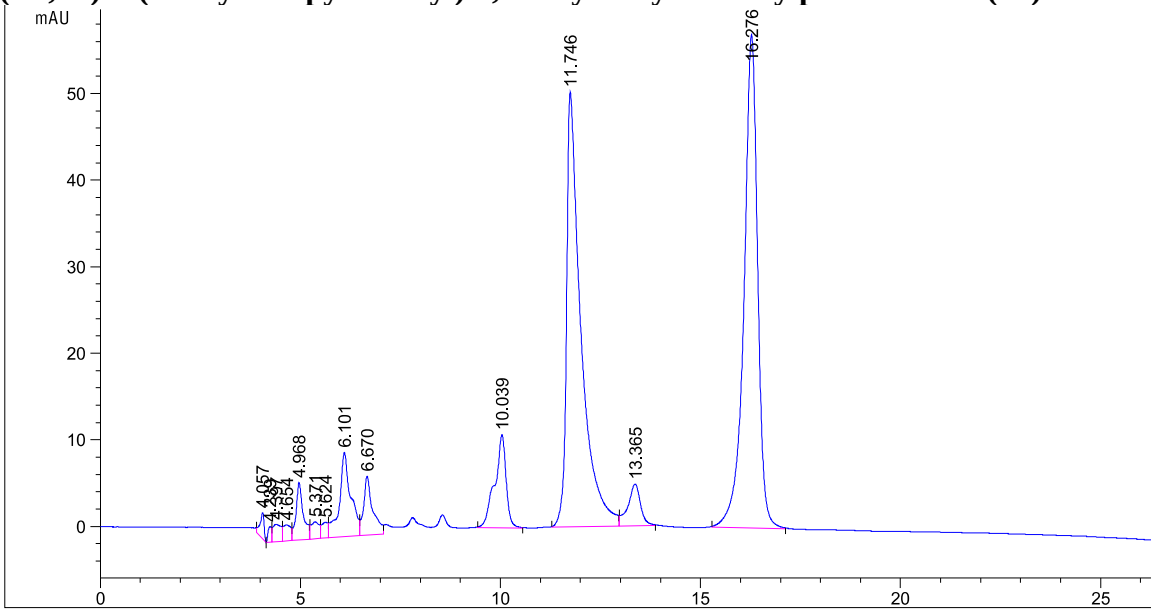
HPLC Traces for Table 2 and Scheme 4.

(2R,3S)-3-cyclohexyl-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxypropan-1-one (3c):



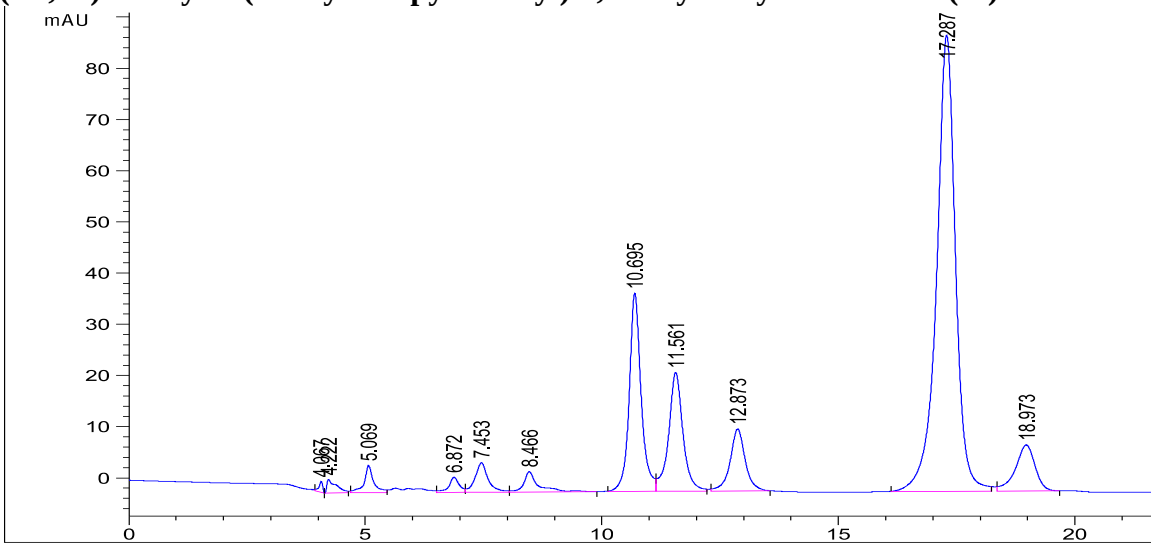
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	39.487	MM	0.8374	127.17352	2.53100	3.1458
2	43.387	MM	0.9091	3915.49316	71.78629	96.8542

(2R,3S)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxy-4-methylpentan-1-one (3d):



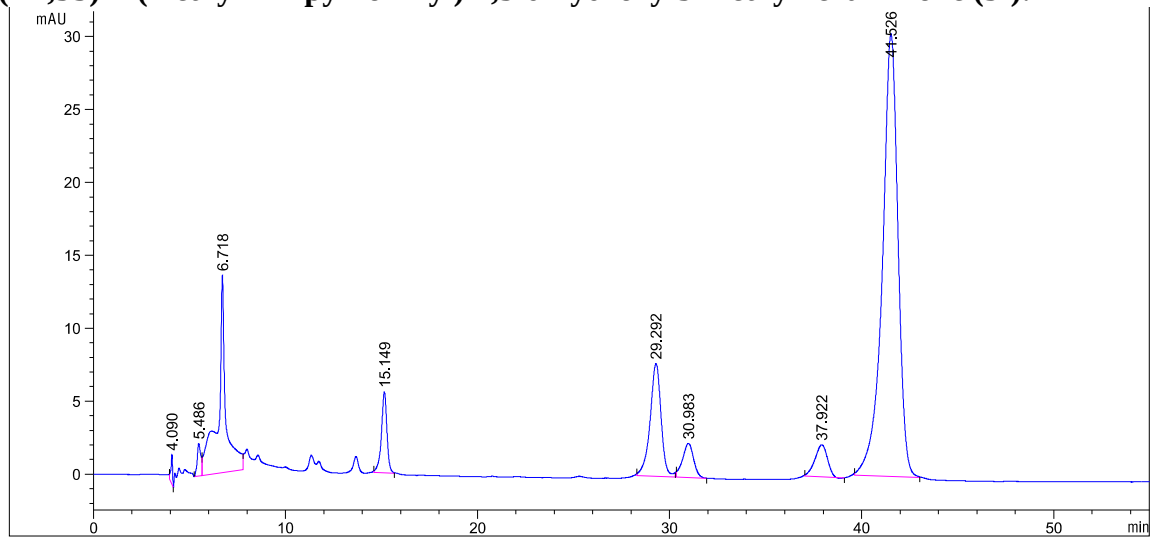
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	4.057	BV	0.1001	20.47013	2.99541	0.5920
2	4.239	VV	0.0940	10.87306	1.79491	0.3145
3	4.397	VV	0.1964	29.42323	2.01713	0.8510
4	4.654	VV	0.1985	24.57318	1.85130	0.7107
5	4.968	VB	0.1745	82.82141	6.64795	2.3953
6	5.371	BV	0.1980	28.74104	1.96869	0.8312
7	5.624	VV	0.1548	20.60103	1.82781	0.5958
8	6.101	VV	0.2703	194.64032	9.77045	5.6292
9	6.670	VV	0.2156	107.38747	6.82430	3.1058
10	10.039	BB	0.2776	213.15219	10.76470	6.1646
11	11.746	BB	0.3458	1257.36609	50.25687	36.3643
12	13.365	BB	0.3207	106.89573	4.85967	3.0915
13	16.276	BB	0.3543	1360.74622	56.99484	39.3542

(2R,3S)-4-ethyl-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxyhexan-1-one (3e):



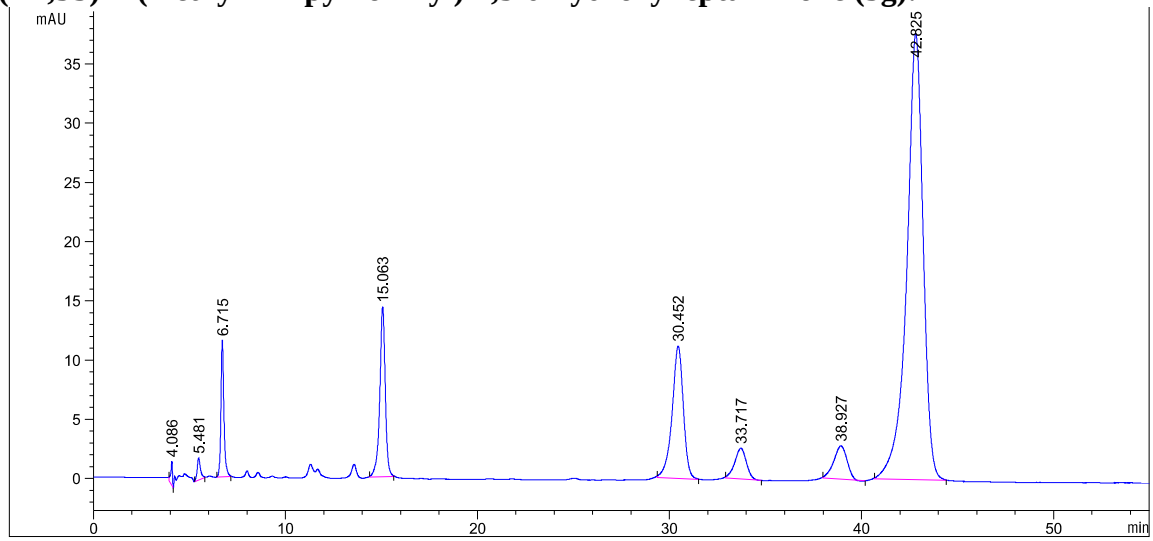
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	4.067	BV	0.0807	11.23573	2.07181	0.1998
2	4.222	VB	0.1848	36.83178	2.61298	0.6551
3	5.069	BB	0.1970	75.18270	5.31616	1.3372
4	6.872	VV	0.2195	45.23331	2.95206	0.8045
5	7.453	VV	0.2686	109.99516	5.77910	1.9563
6	8.466	VB	0.2954	85.69457	3.97224	1.5241
7	10.695	BV	0.2591	680.90759	38.71127	12.1104
8	11.561	VB	0.3113	491.92499	23.21737	8.7492
9	12.873	BB	0.3306	270.96432	12.12619	4.8193
10	17.287	BB	0.4134	2489.56445	89.07103	44.2785
11	18.973	BB	0.4312	259.24609	8.98709	4.6109
12	22.890	BB	0.8213	1065.73108	16.93998	18.9547

(2R,3S)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxy-5-methylhexan-1-one (3f):



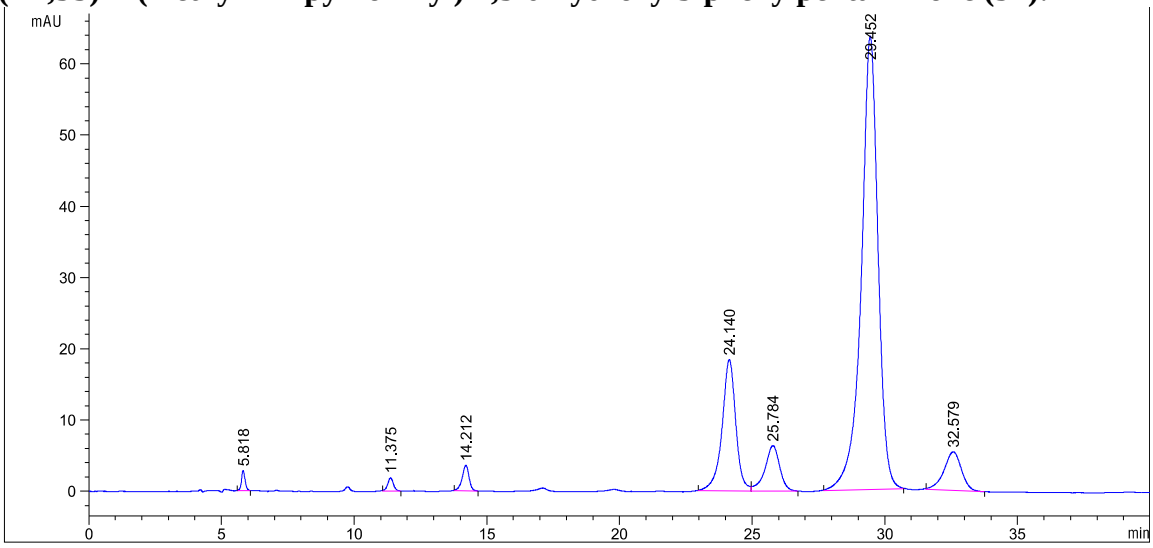
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
3	6.718	VB	0.3675	385.98544	13.54246	13.9925
4	15.149	BB	0.2984	110.50639	5.53534	4.0060
5	29.292	BB	0.5943	306.26480	7.73644	11.1025
6	30.983	BB	0.5953	94.06754	2.34918	3.4101
7	37.922	BB	0.6487	98.17845	2.19416	3.5591
8	41.526	BB	0.8499	1722.27637	30.34835	62.4350

(2R,3S)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxyheptan-1-one (3g):



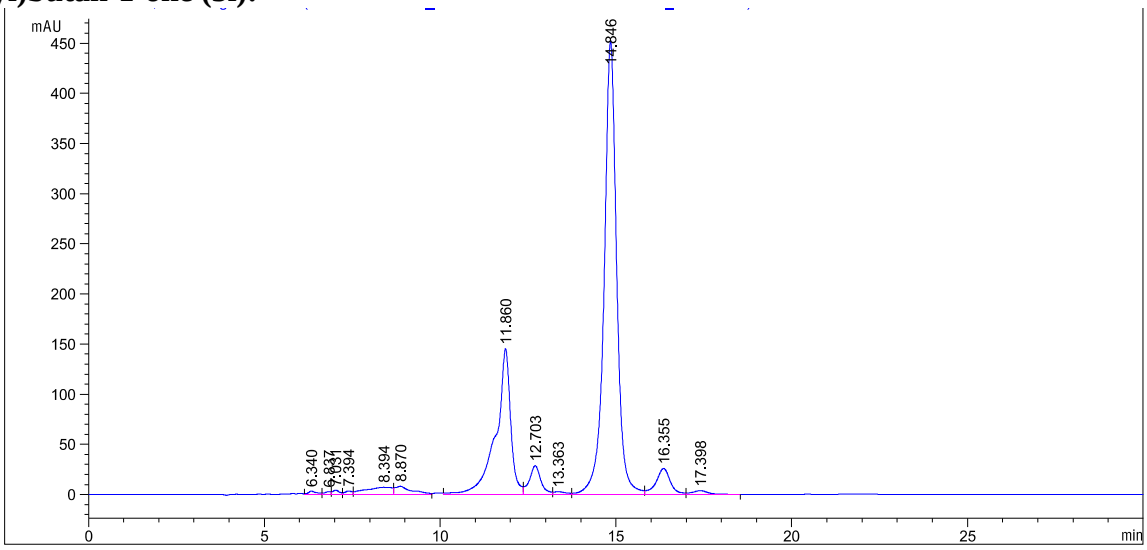
Peak #	Time [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
3	6.715	BB	0.1618	125.66795	11.55608	3.7079
4	15.063	BB	0.2980	288.07629	14.36572	8.4997
5	30.452	BB	0.6139	453.68573	11.16448	13.3861
6	33.717	BB	0.6171	109.38528	2.59426	3.2274
7	38.927	BB	0.6854	135.51190	2.81489	3.9983
8	42.825	BB	0.8841	2242.33472	37.65204	66.1605

(2R,3S)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxy-5-phenylpentan-1-one (3h):



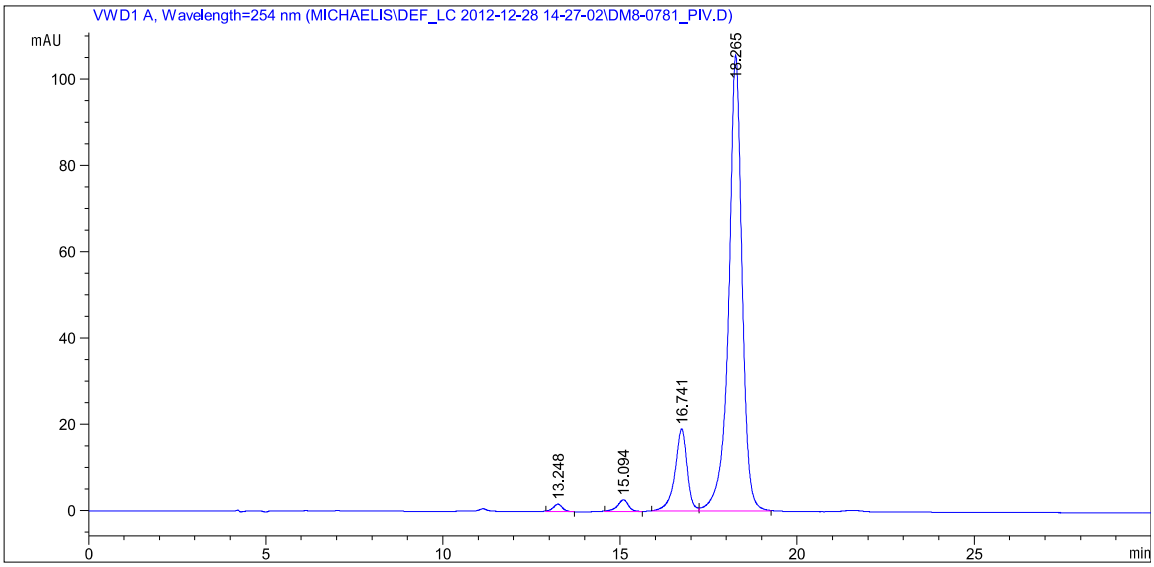
Peak #	Time [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.818	BB	0.1445	27.44228	2.84593	0.6726
2	11.375	BB	0.2256	28.23247	1.87077	0.6920
3	14.212	BB	0.2739	65.86465	3.61240	1.6143
4	24.140	BV	0.5372	665.49609	18.45604	16.3111
5	25.784	VB	0.5805	249.15974	6.40584	6.1068
6	29.452	BB	0.6539	2797.13184	63.65399	68.5566
7	32.579	BB	0.6733	246.70294	5.40946	6.0466
Totals :				4080.03001	102.25444	

(2R,3S)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxy-4-(2-methyl-1,3-dioxan-2-yl)butan-1-one (3i):



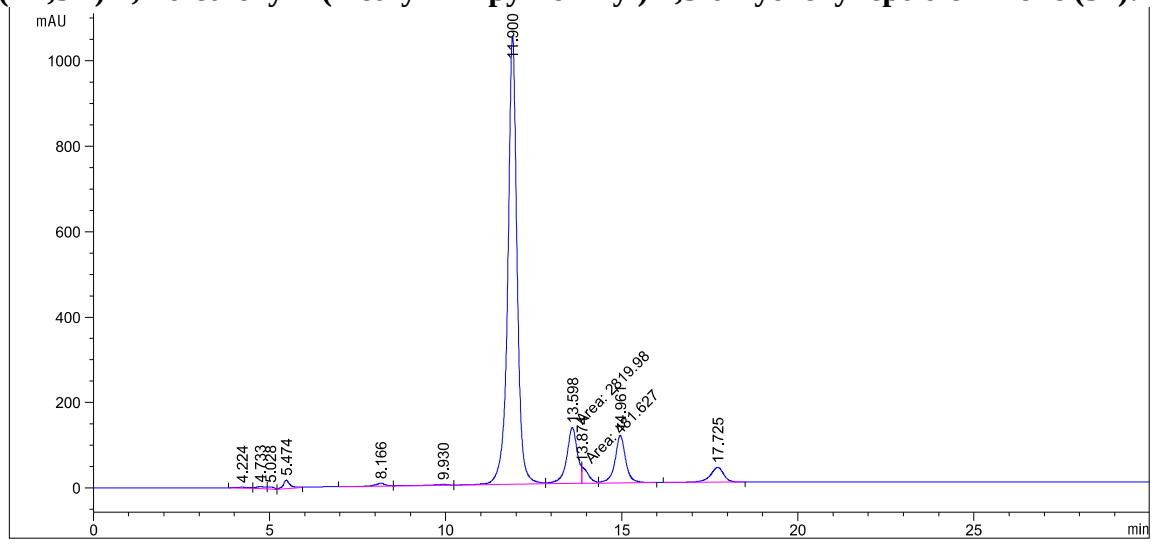
Peak #	Time [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.340	VV	0.2302	51.66825	3.13445	0.2896
2	6.837	VV	0.1783	32.30010	2.71125	0.1810
3	7.031	VV	0.1915	54.23491	4.08024	0.3040
4	7.394	VV	0.2042	47.46297	3.32793	0.2660
5	8.394	VV	0.7057	366.96234	7.05646	2.0568
6	8.870	VV	0.4388	266.91818	8.02849	1.4961
7	11.860	VV	0.4036	4256.51025	145.02238	23.8575
8	12.703	VV	0.3316	648.47833	28.59179	3.6347
9	13.363	VV	0.3472	63.80077	2.65619	0.3576
10	14.846	VV	0.3680	1.12065e4	451.83545	62.8120
11	16.355	VV	0.4166	728.69110	25.81748	4.0843
12	17.398	VB	0.4642	117.83560	3.69130	0.6605

(2R,3S)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxy-4,4-dimethylpentan-1-one (3j):



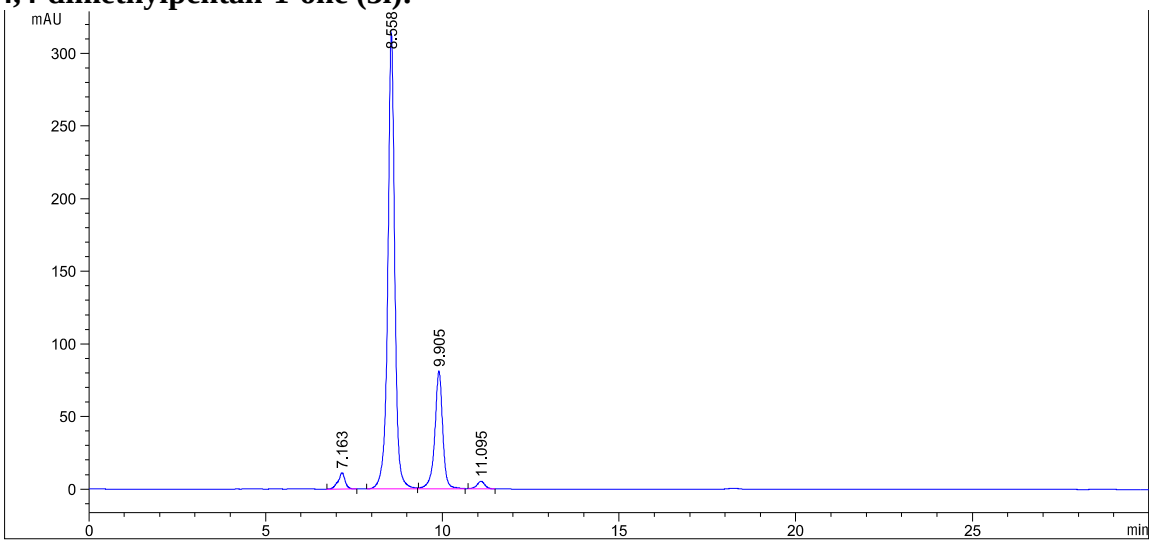
Peak #	Time [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.248	BB	0.2776	31.21518	1.71676	0.9394
2	15.094	BB	0.3164	56.98517	2.69713	1.7150
3	16.741	BV	0.3570	455.92886	19.00949	13.7211
4	18.265	VB	0.3872	2778.70386	105.55822	83.6245

(2R,3R)-4,4-diethoxy-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxyhept-6-en-1-one (3k):



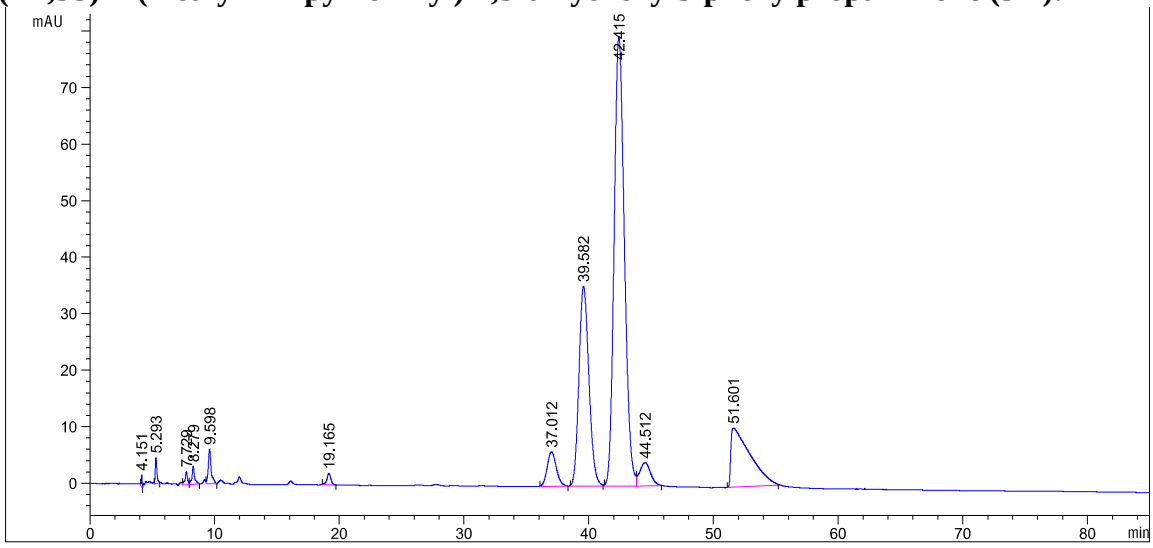
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	4.224	BB	0.3276	73.76086	2.85504	0.2774
2	4.733	BV	0.2705	102.83092	5.12410	0.3867
3	5.028	VV	0.1901	58.40385	4.84774	0.2197
4	5.474	VB	0.1940	262.21960	19.40849	0.9862
5	8.166	VV	0.2610	131.20596	7.09195	0.4935
6	9.930	VV	0.4271	82.55071	2.56028	0.3105
7	11.900	VV	0.2725	1.92784e4	1049.63708	72.5043
8	13.598	MF	0.3579	2819.97900	131.30621	10.6057
9	13.874	FM	0.2050	481.62720	39.16420	1.8114
10	14.961	VB	0.3140	2370.58252	111.29093	8.9155
11	17.725	BB	0.3957	927.76965	34.93492	3.4893

(2R,3S)-5-((tert-butyldimethylsilyl)oxy)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxy-4,4-dimethylpentan-1-one (3l):



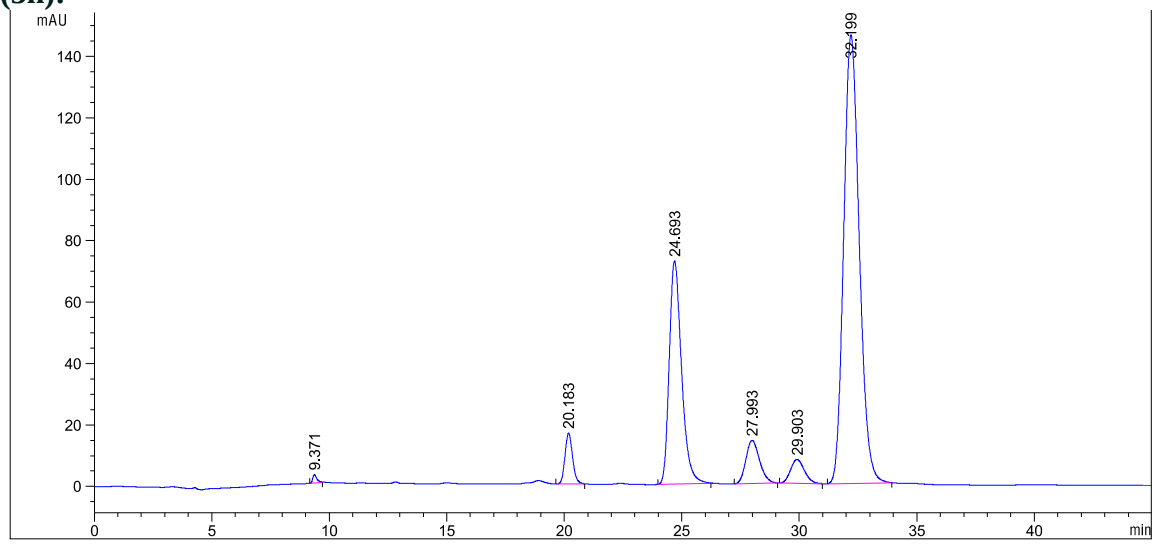
Peak #	RT [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.163	BB	0.1979	155.64168	11.24250	2.6801
2	8.558	BB	0.2037	4298.59814	313.36346	74.0202
3	9.905	BB	0.2335	1269.55762	81.12251	21.8613
4	11.095	BB	0.2331	83.53390	5.35122	1.4384

(2R,3S)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxy-3-phenylpropan-1-one (3m):



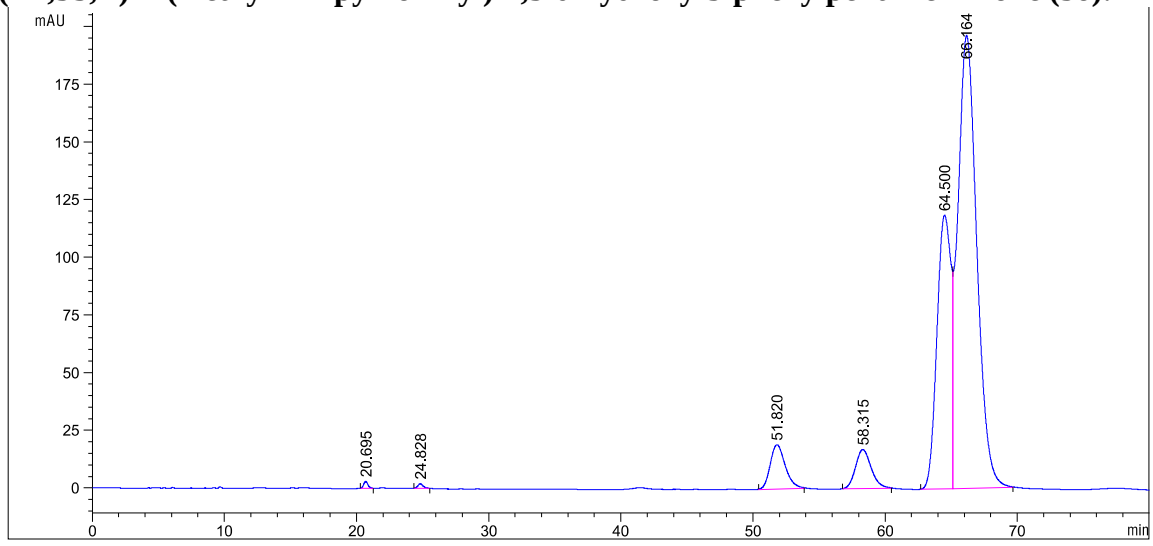
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	4.151	BV	0.0746	9.84968	2.06284	0.1161
2	5.293	VB	0.1351	42.37485	4.66566	0.4995
3	7.729	VV	0.1989	32.13319	2.28680	0.3788
4	8.279	VB	0.2140	47.19418	3.12528	0.5563
5	9.598	VB	0.2351	98.83949	6.11808	1.1651
6	19.165	BB	0.3452	46.47079	2.03400	0.5478
7	37.012	BB	0.7809	312.35718	6.11461	3.6820
8	39.582	BB	0.8639	1969.80493	35.36532	23.2194
9	42.415	BV	0.8909	4545.24316	79.54462	53.5777
10	44.512	VB	0.8978	251.19789	4.12801	2.9610
11	51.601	BB	1.3574	1127.99268	10.51877	13.2964

(2R,3S)-3-(4-chlorophenyl)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxypropan-1-one (3n):



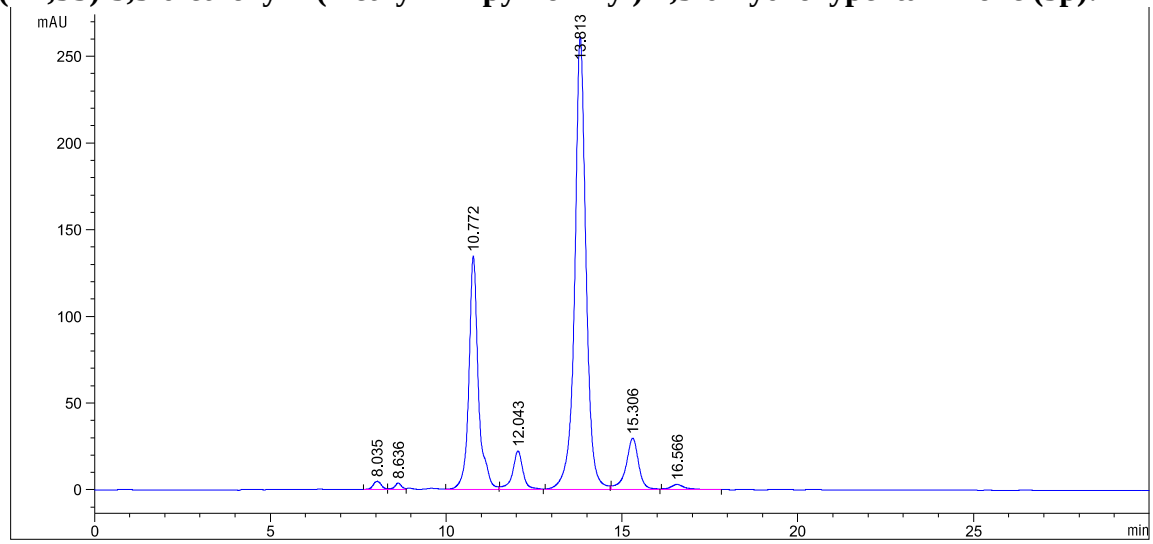
Peak #	Time [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.371	BB	0.1808	32.89509	2.71235	0.3083
2	20.183	VB	0.3453	370.69180	16.57926	3.4743
3	24.693	BB	0.5575	2645.32031	72.60931	24.7935
4	27.993	BB	0.6253	572.14655	14.08710	5.3625
5	29.903	BB	0.6333	319.52985	7.78387	2.9948
6	32.199	BB	0.7167	6728.83447	146.02054	63.0666

(2R,3S,E)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxy-5-phenylpent-4-en-1-one (3o):



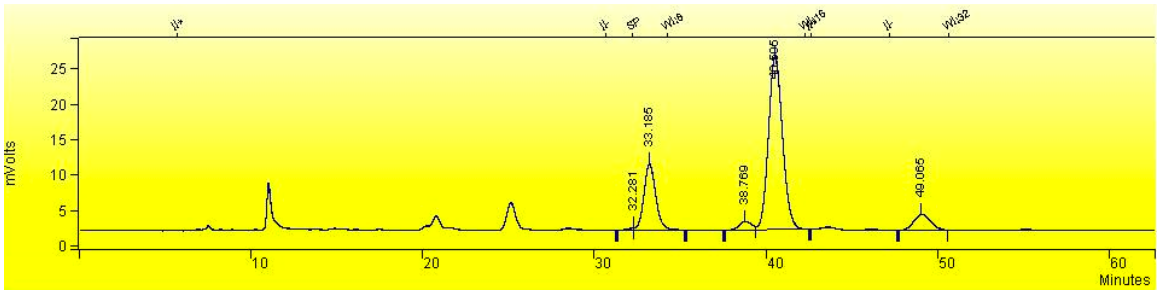
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	20.695	BB	0.3445	66.93222	2.98639	0.2128
2	24.828	BB	0.4333	54.87017	1.95775	0.1745
3	51.820	BB	1.1708	1502.99292	19.14305	4.7792
4	58.315	BB	1.2412	1398.14746	16.93659	4.4458
5	64.500	BV	1.1295	8814.61523	118.56046	28.0285
6	66.164	VB	1.4993	1.96112e4	196.24577	62.3592

(2R,3S)-5,5-diethoxy-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxypentan-1-one (3p):



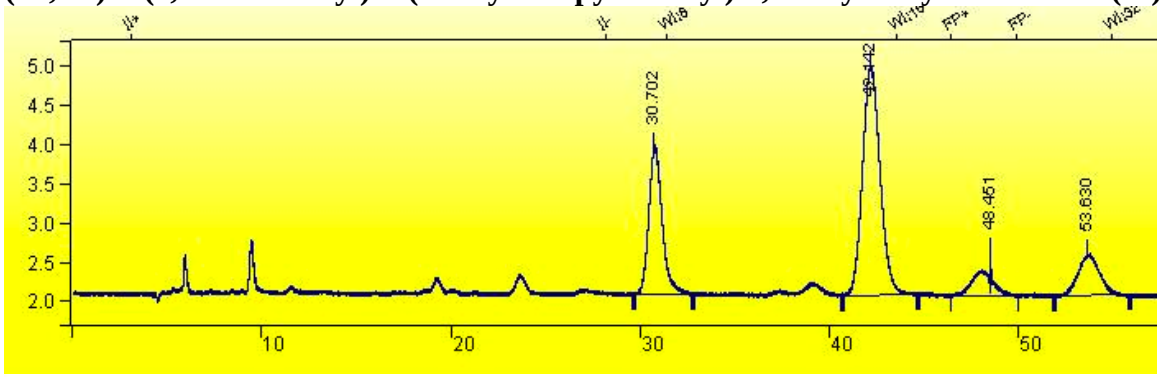
Peak #	RT [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.035	BV	0.2595	78.17309	4.70113	0.7945
2	8.636	VV	0.2010	51.98550	3.78526	0.5283
3	10.772	VB	0.2772	2543.34668	134.62369	25.8477
4	12.043	BB	0.3050	463.34738	22.30186	4.7089
5	13.813	BB	0.3319	5866.29639	261.20474	59.6184
6	15.306	BB	0.3718	746.79132	29.72198	7.5895
7	16.566	BB	0.4307	89.80212	3.02552	0.9126

(2S,3R)-4-(1,3-dioxolan-2-yl)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxybutan-1-one (3q):



Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		0.3509	32.281	0.000	7302	BV	0.0	U
2		21.6157	33.185	0.000	449853	VB	42.8	U
3		2.9933	38.769	0.000	62295	BV	46.8	U
4		67.7206	40.505	0.000	1409362	VB	53.1	U
5		7.3195	49.065	0.000	152329	BB	61.2	U
	Totals:	100.0000		0.000	2081141			

(2S,3R)-4-(1,3-dioxan-2-yl)-1-(2-ethyl-1H-pyrrol-1-yl)-2,3-dihydroxybutan-1-one (3r):



Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		26.4705	30.702	0.000	92636	BB	35.4	U
2		54.0458	42.142	0.000	189138	BB	57.8	U
3		7.4938	48.451	0.000	26225	BB	0.1	
4		11.9899	53.630	0.000	41960	BB	51.3	U
	Totals:	100.0000		0.000	349959			