

2-Arylacetic Anhydrides As Ammonium Enolate Precursors

*Louis C. Morrill, Lyndsay A. Ledingham, Jean-Philippe Couturier, Jasmine Bickel,
Andrew D. Harper, Charlene Fallan and Andrew D. Smith **

*EaStCHEM, School of Chemistry, University of St Andrews, North Haugh, St
Andrews, KY16 9ST, UK.*

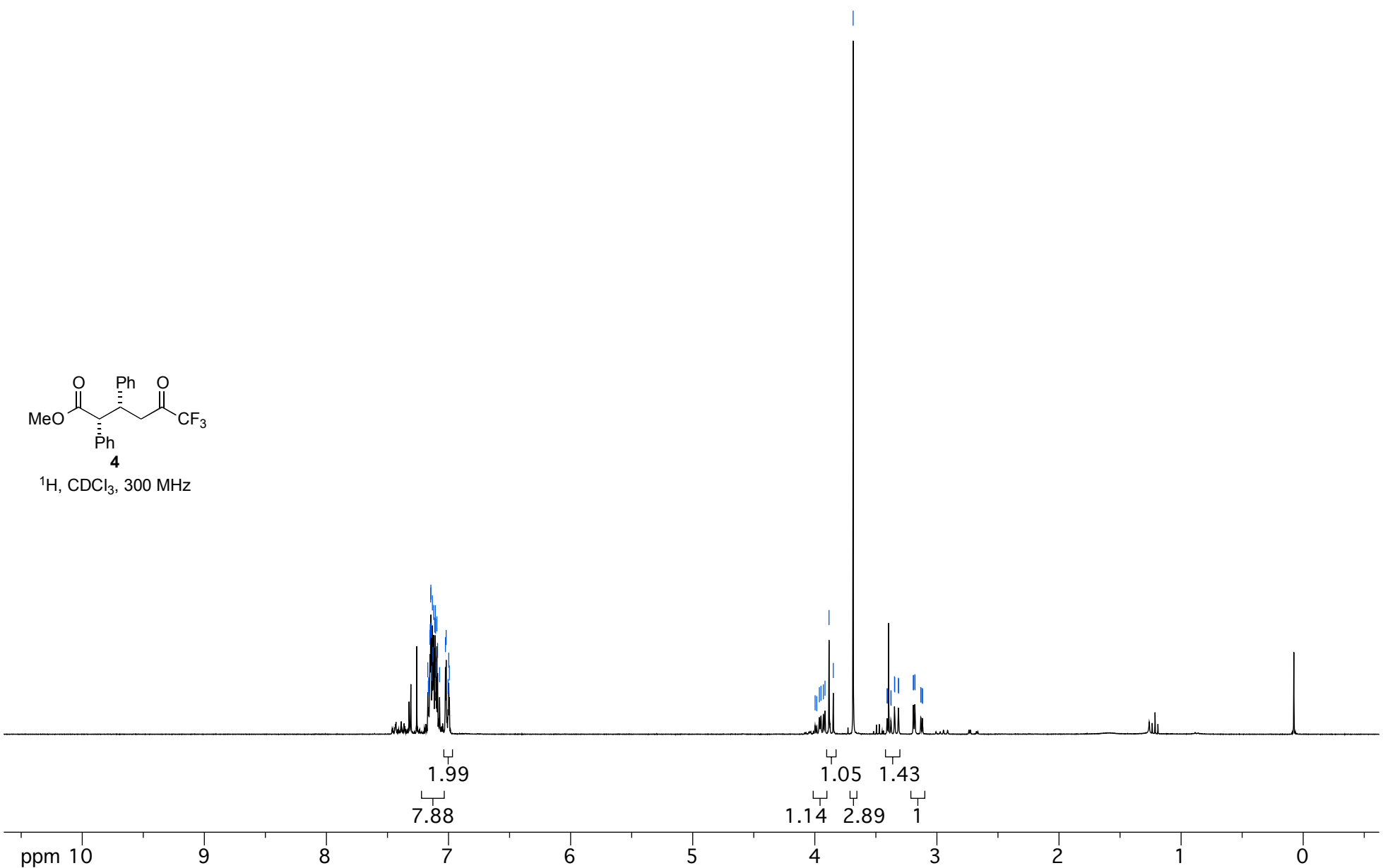
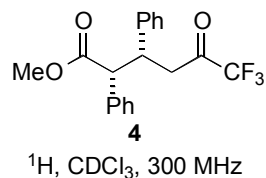
e-mail: ads10@st-andrews.ac.uk

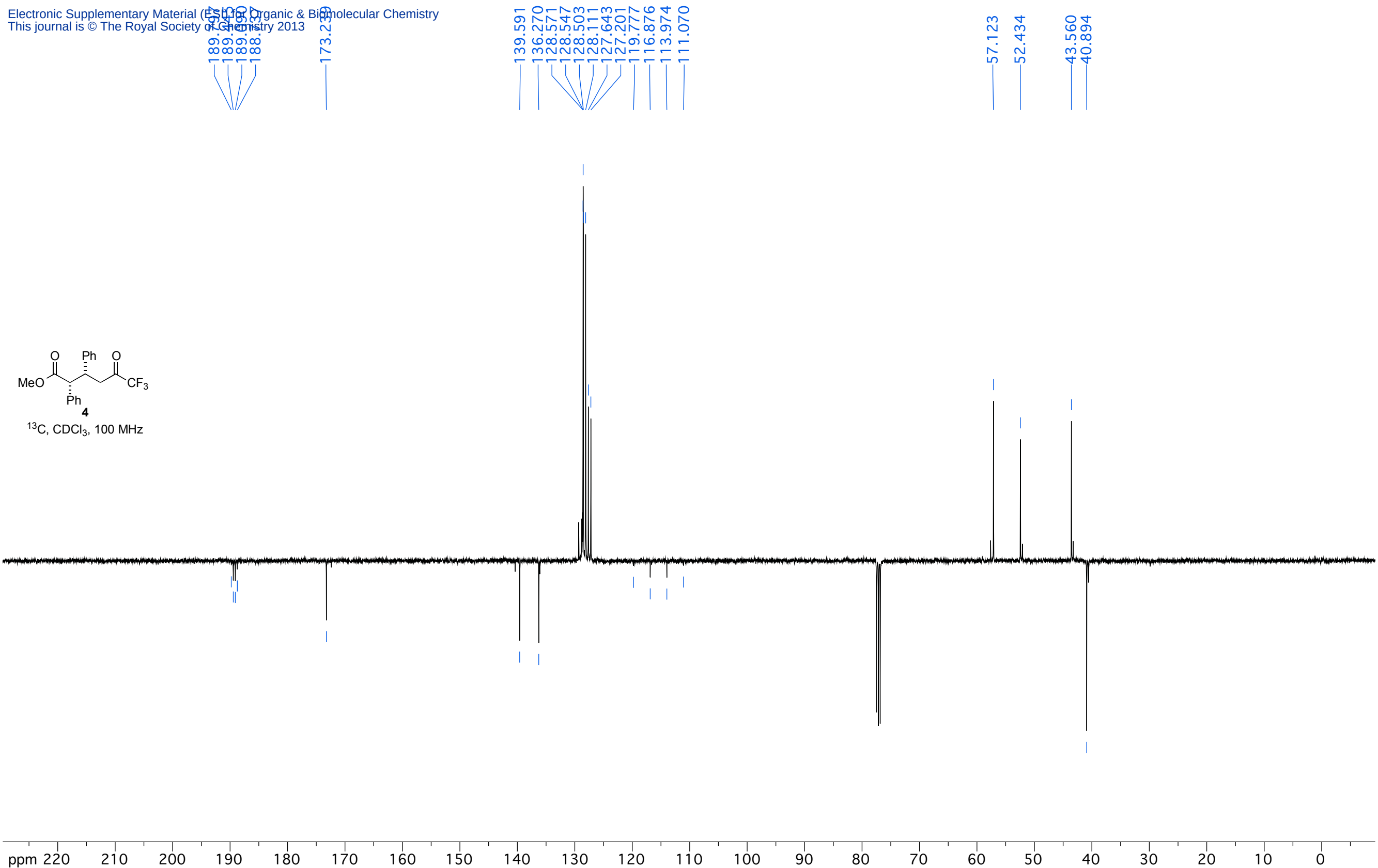
SUPPORTING INFORMATION

Contents

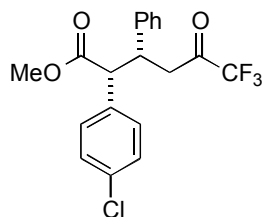
¹H and ¹³C NMR Spectra for Novel Compounds and HPLC data

7.109
7.108
7.106
7.101
7.095
7.090
7.073
7.025
7.019
7.004
6.998
6.993
6.997
3.984
3.963
3.950
3.930
3.917
3.884
3.849
3.686
3.408
3.406
3.377
3.375
3.347
3.346
3.316
3.314
3.193
3.181
3.179
3.132
3.131
3.119
3.118



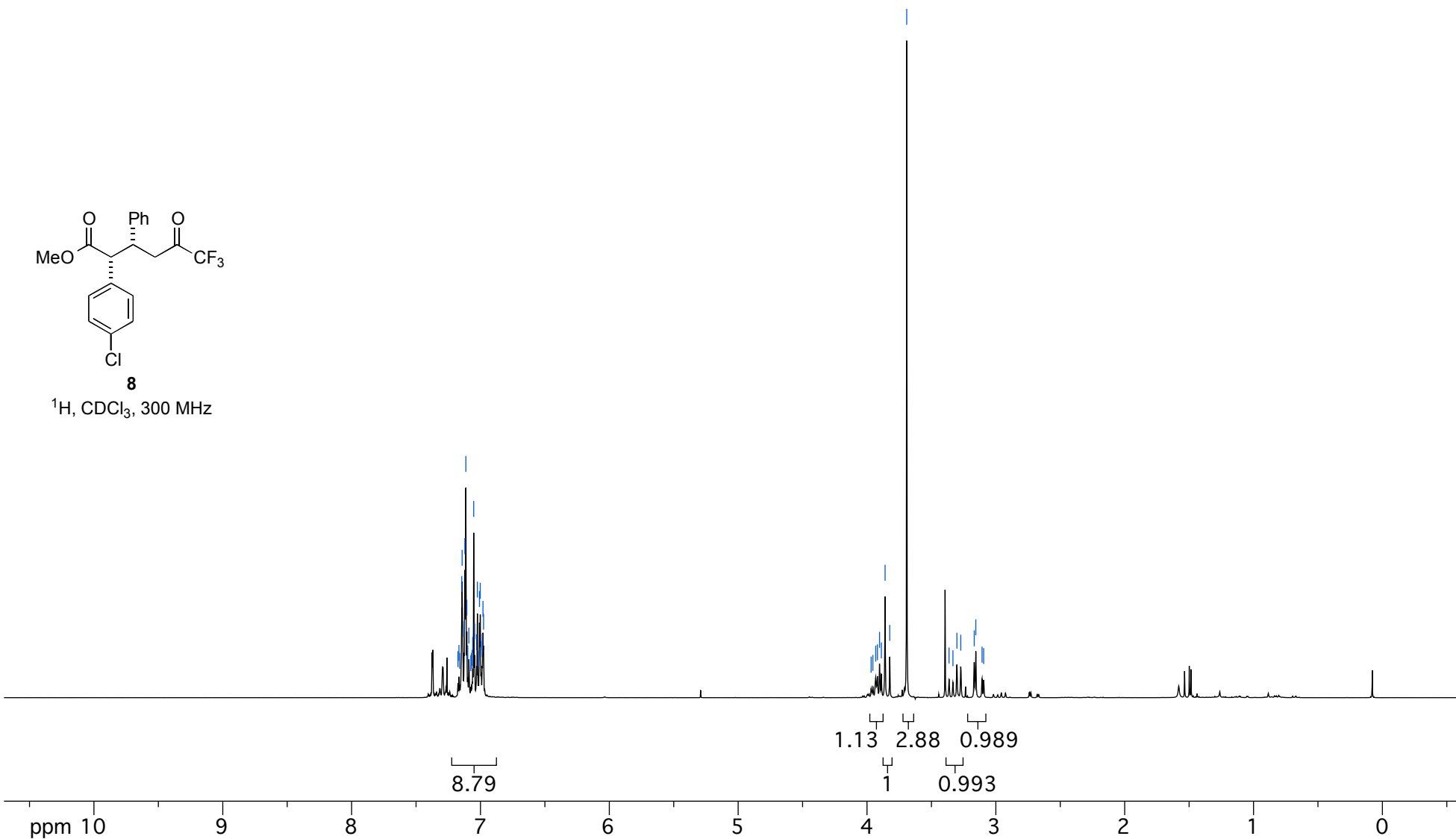


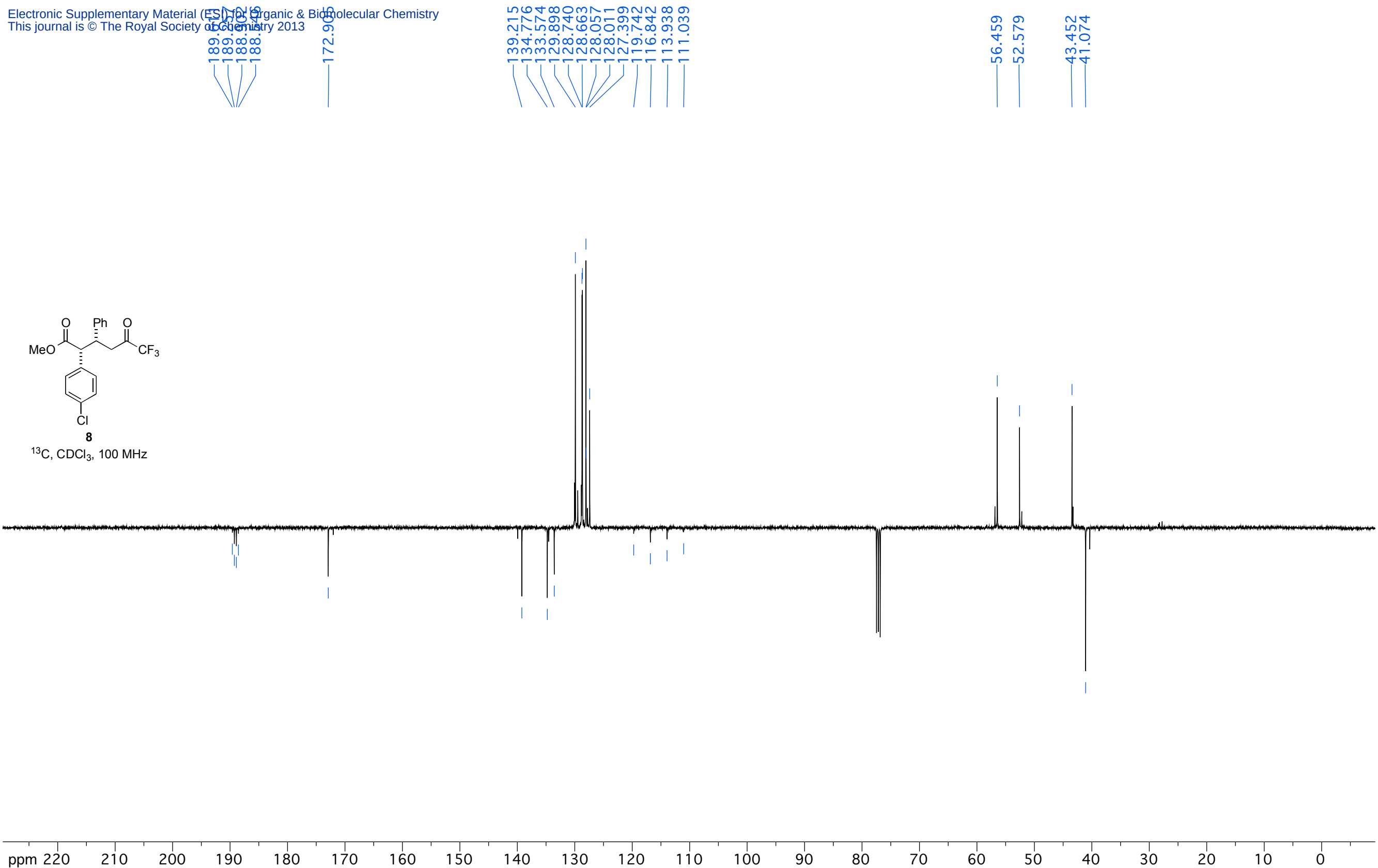
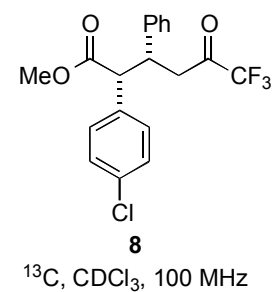
7.109
7.108
7.107
7.106
7.105
7.104
7.103
7.102
7.101
7.100
7.099
7.098
7.097
7.096
7.095
7.094
7.093
7.092
7.091
7.090
7.089
7.088
7.087
7.086
7.085
7.084
7.083
7.082
7.081
7.080
7.079
7.078
7.077
7.076
7.075
7.074
7.073
7.072
7.071
7.070
7.069
7.068
7.067
7.066
7.065
7.064
7.063
7.062
7.061
7.060
7.059
7.058
7.057
7.056
7.055
7.054
7.053
7.052
7.051
7.050
7.049
7.048
7.047
7.046
7.045
7.044
7.043
7.042
7.041
7.040
7.039
7.038
7.037
7.036
7.035
7.034
7.033
7.032
7.031
7.030
7.029
7.028
7.027
7.026
7.025
7.024
7.023
7.022
7.021
7.020
7.019
7.018
7.017
7.016
7.015
7.014
7.013
7.012
7.011
7.010
7.009
7.008
7.007
7.006
7.005
7.004
7.003
7.002
7.001
6.999
6.998
6.997
6.996
6.995
6.994
6.993
6.992
6.991
6.990
6.989
6.988
6.987
6.986
6.985
6.984
6.983
6.982
6.981
6.980
6.979
6.978
6.977
6.976
3.968
3.954
3.932
3.919
3.903
3.889
3.860
3.824
3.692
3.363
3.333
3.302
3.272
3.168
3.155
3.107
3.095

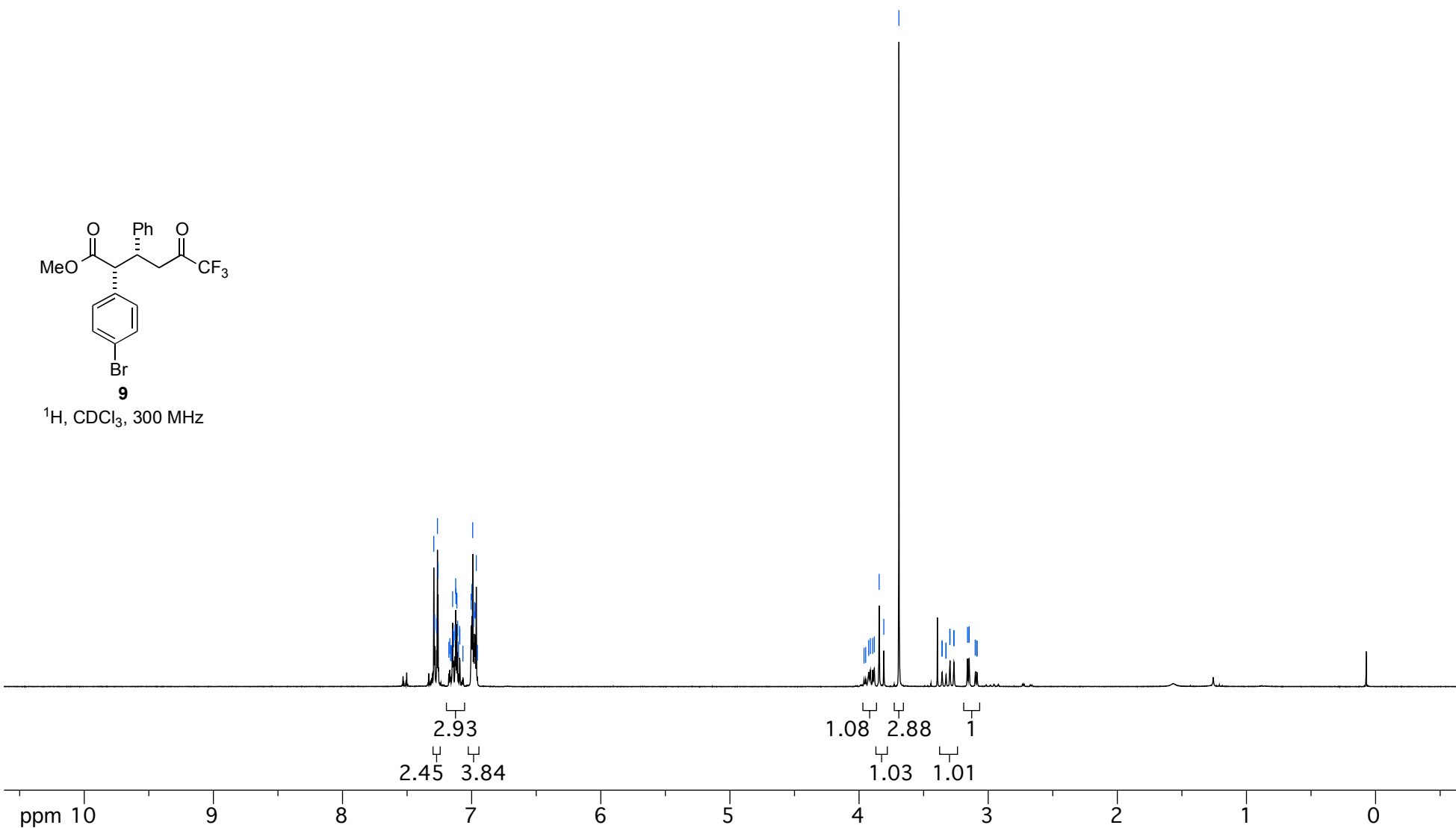
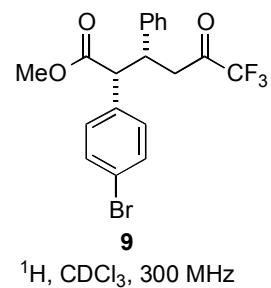


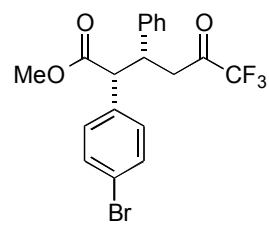
8

^1H , CDCl_3 , 300 MHz

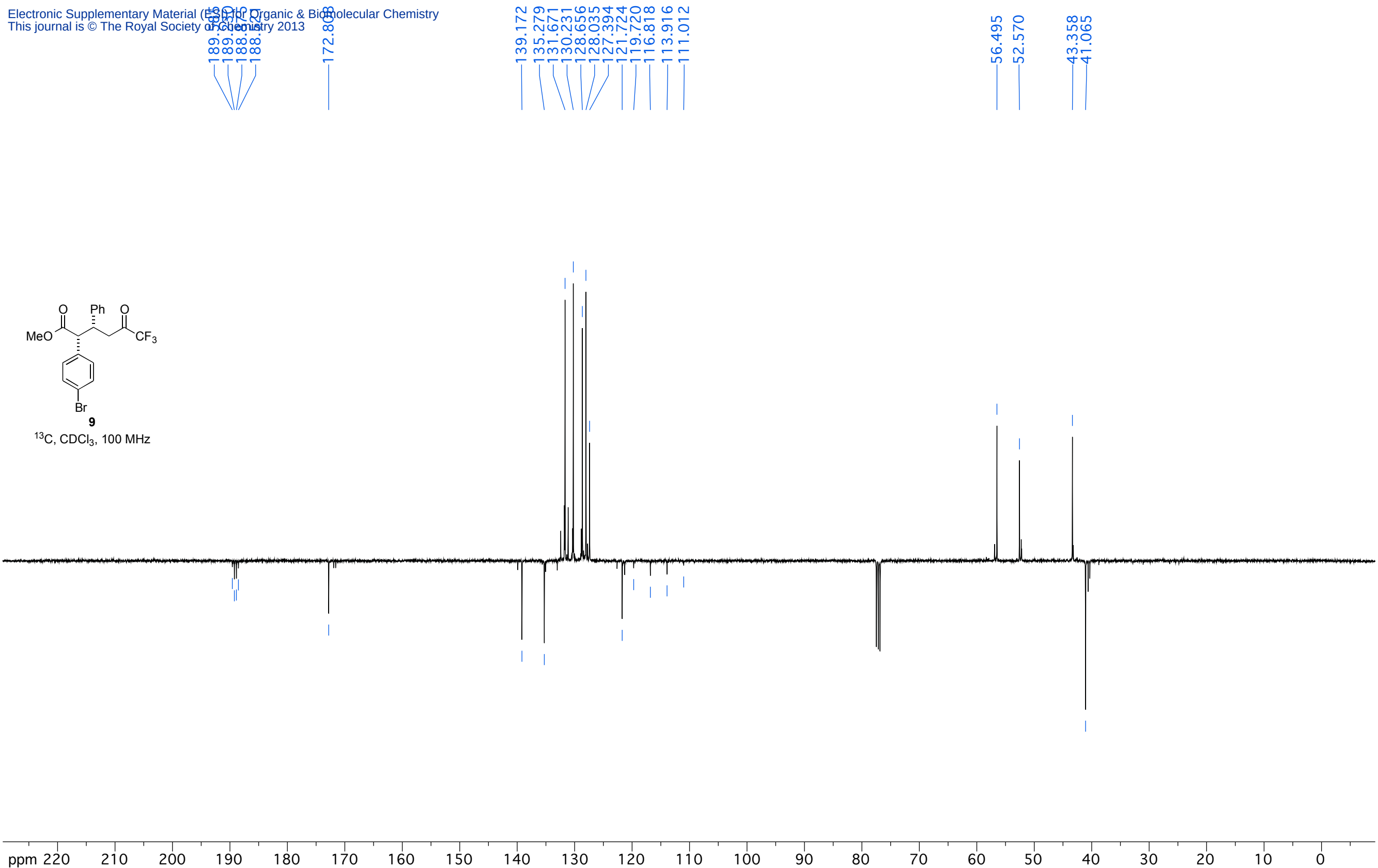


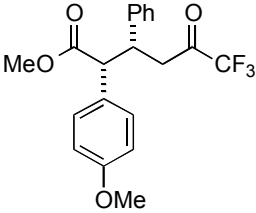




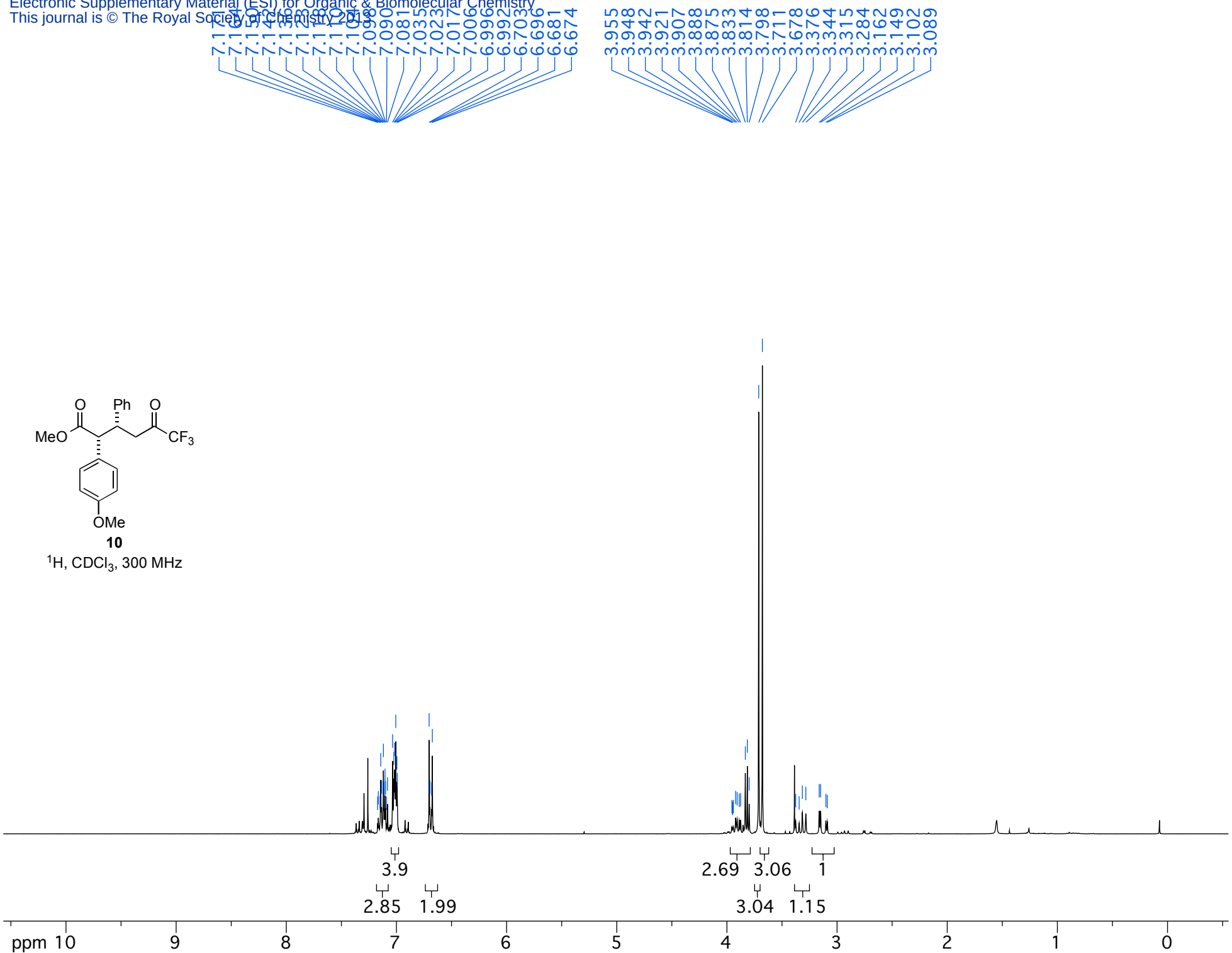


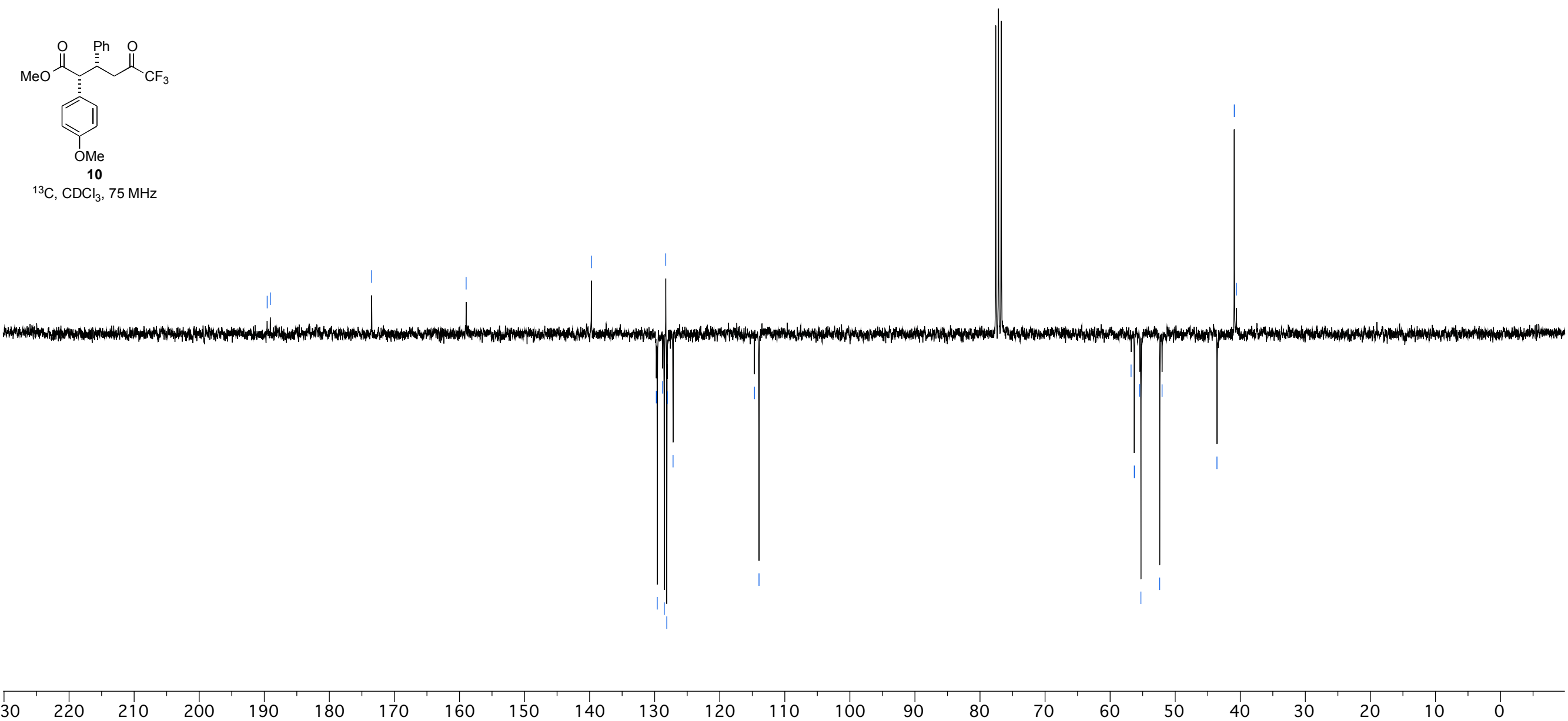
^{13}C , CDCl_3 , 100 MHz

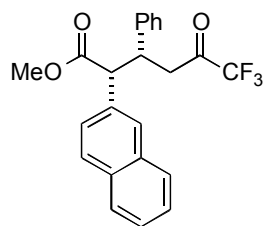




10
¹H, CDCl₃, 300 MHz

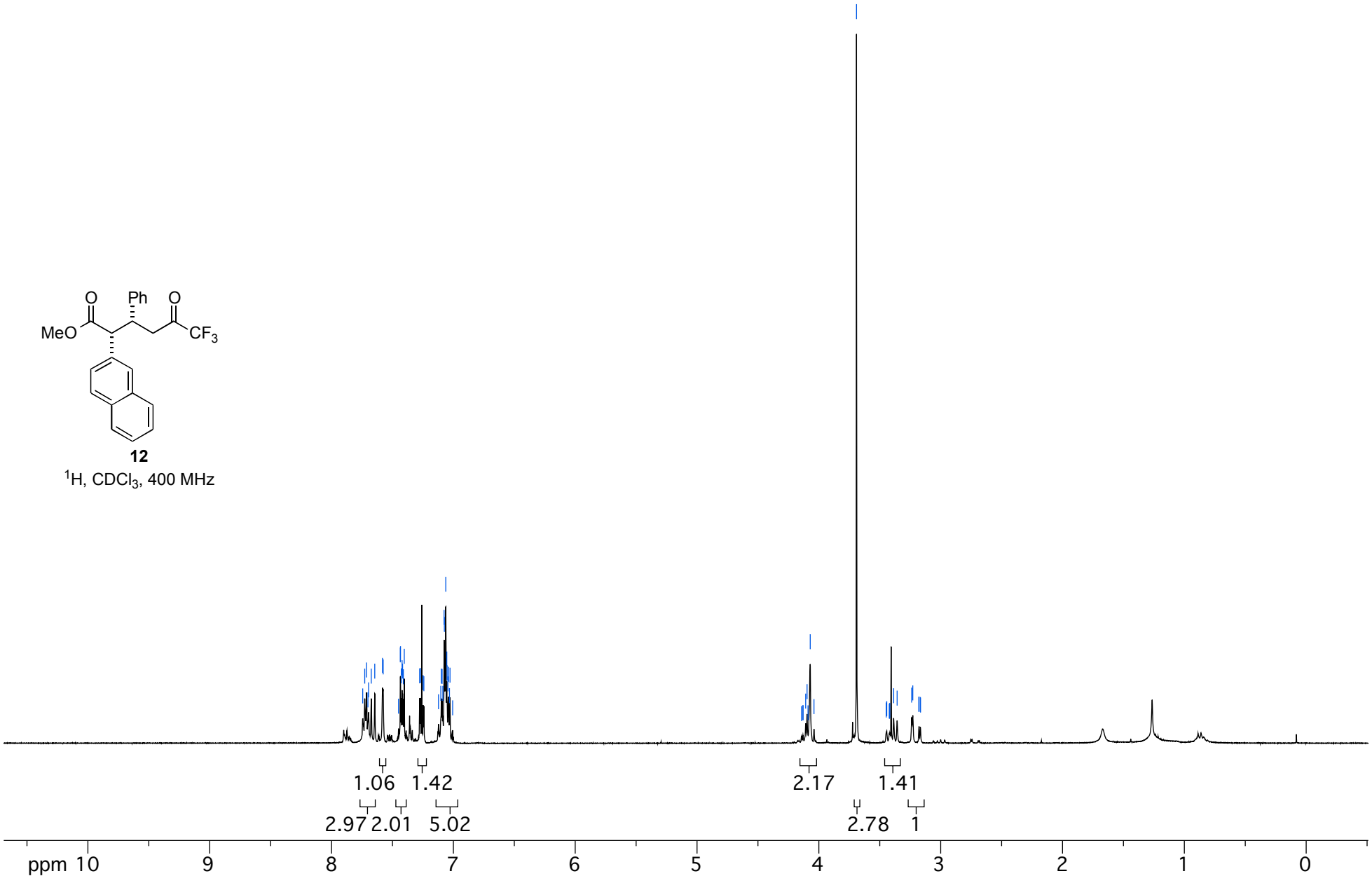


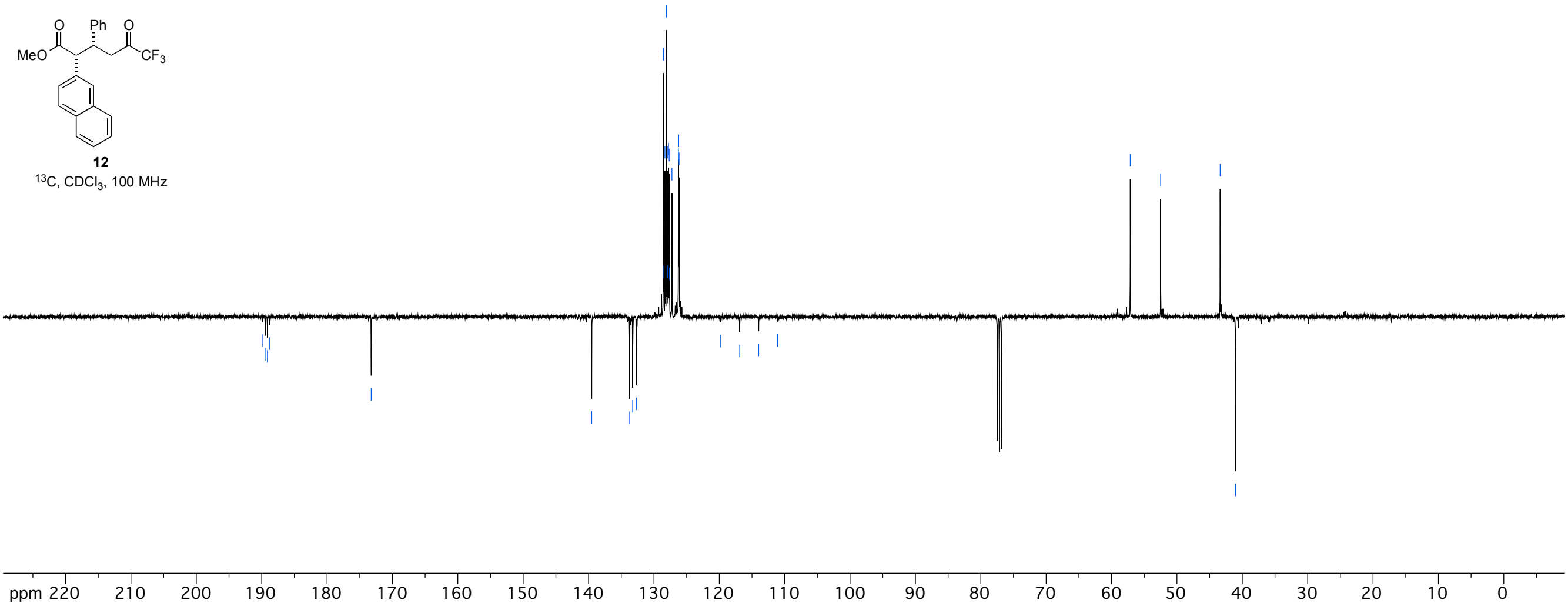


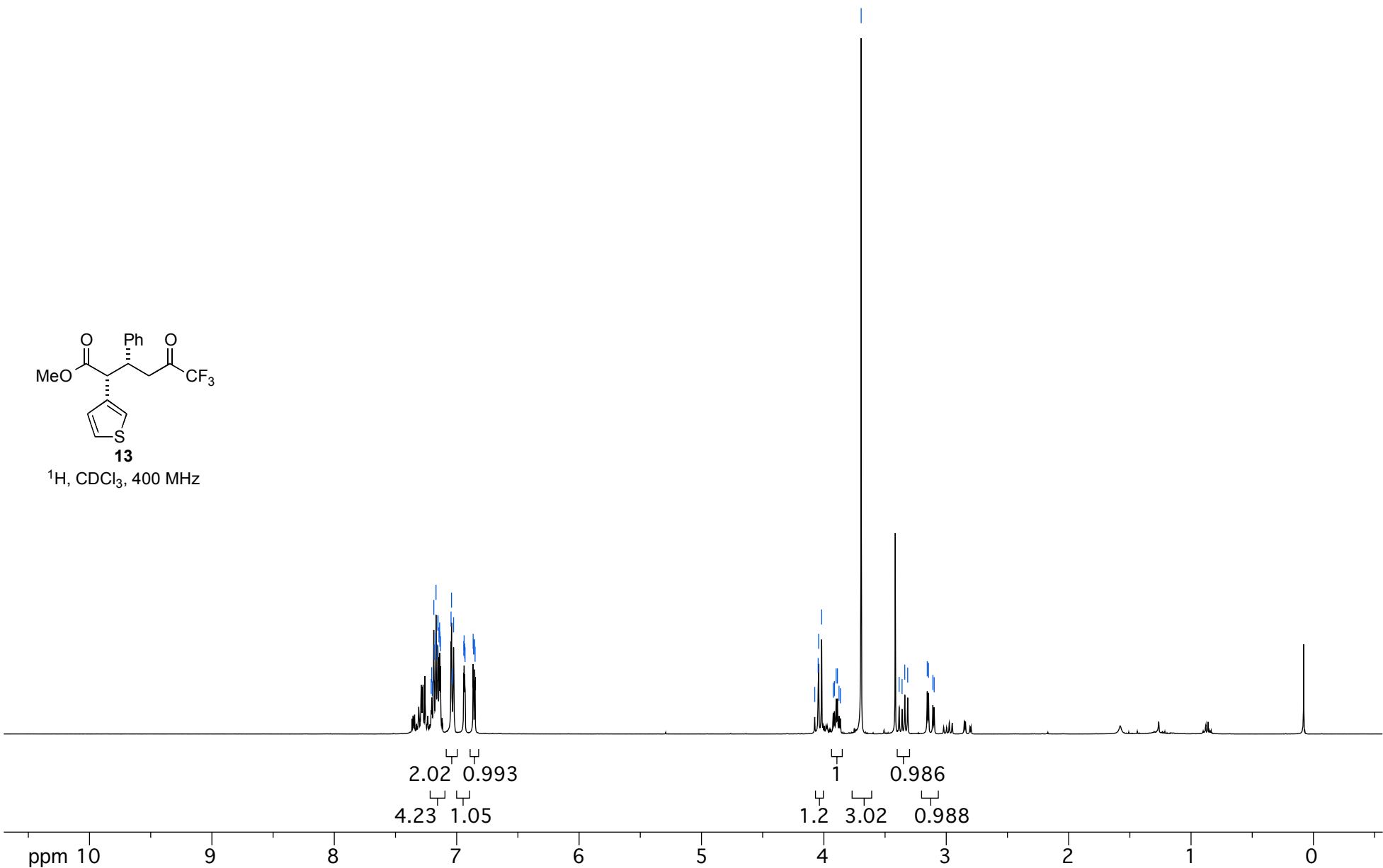
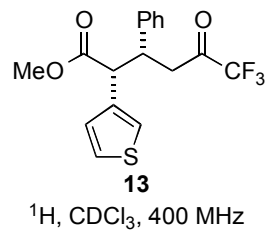


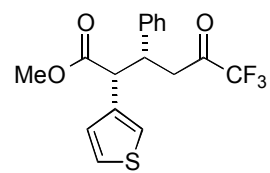
12

^1H , CDCl_3 , 400 MHz

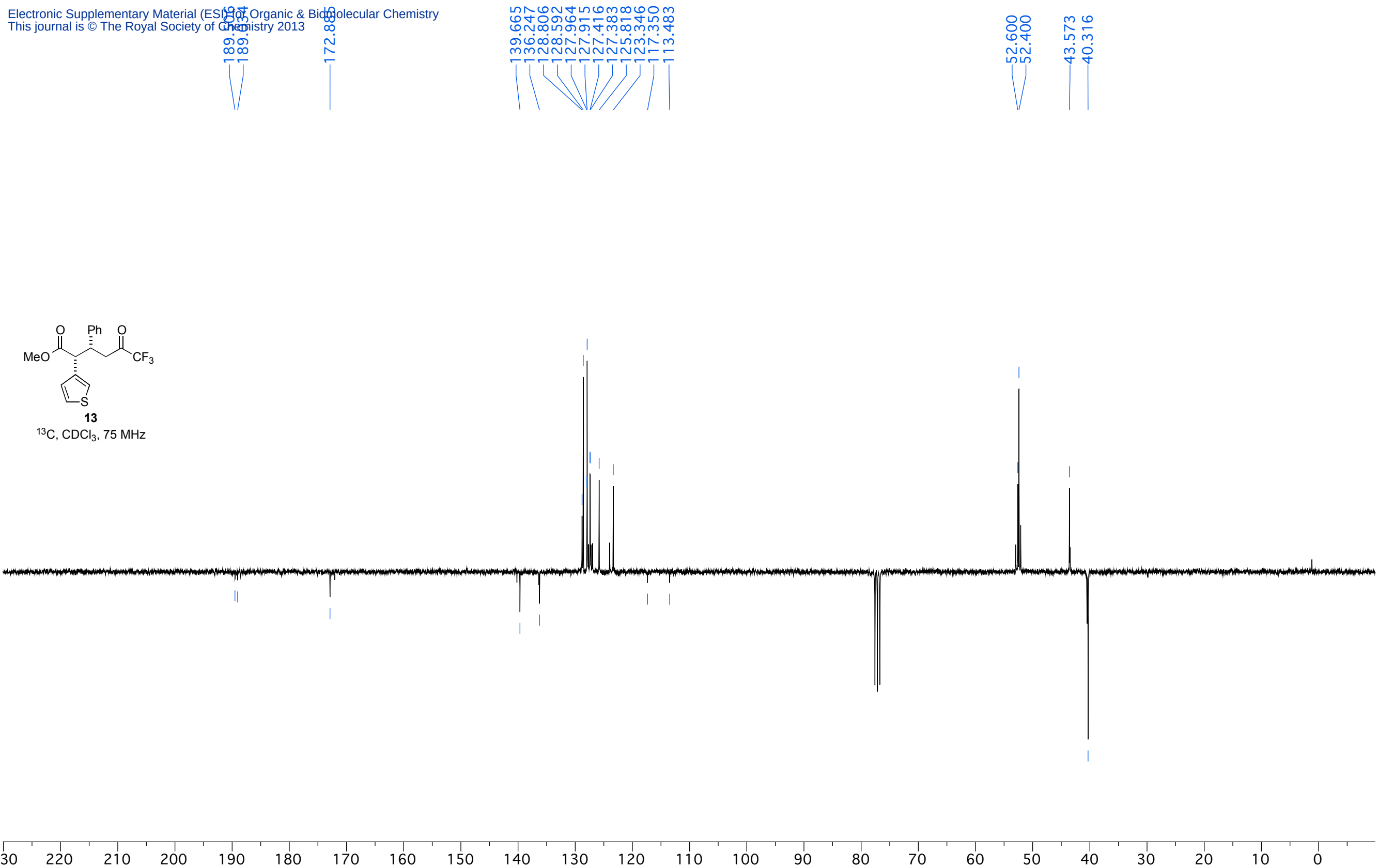


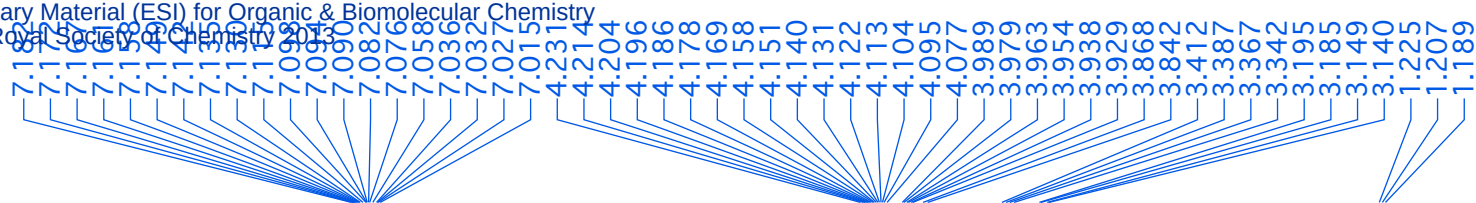
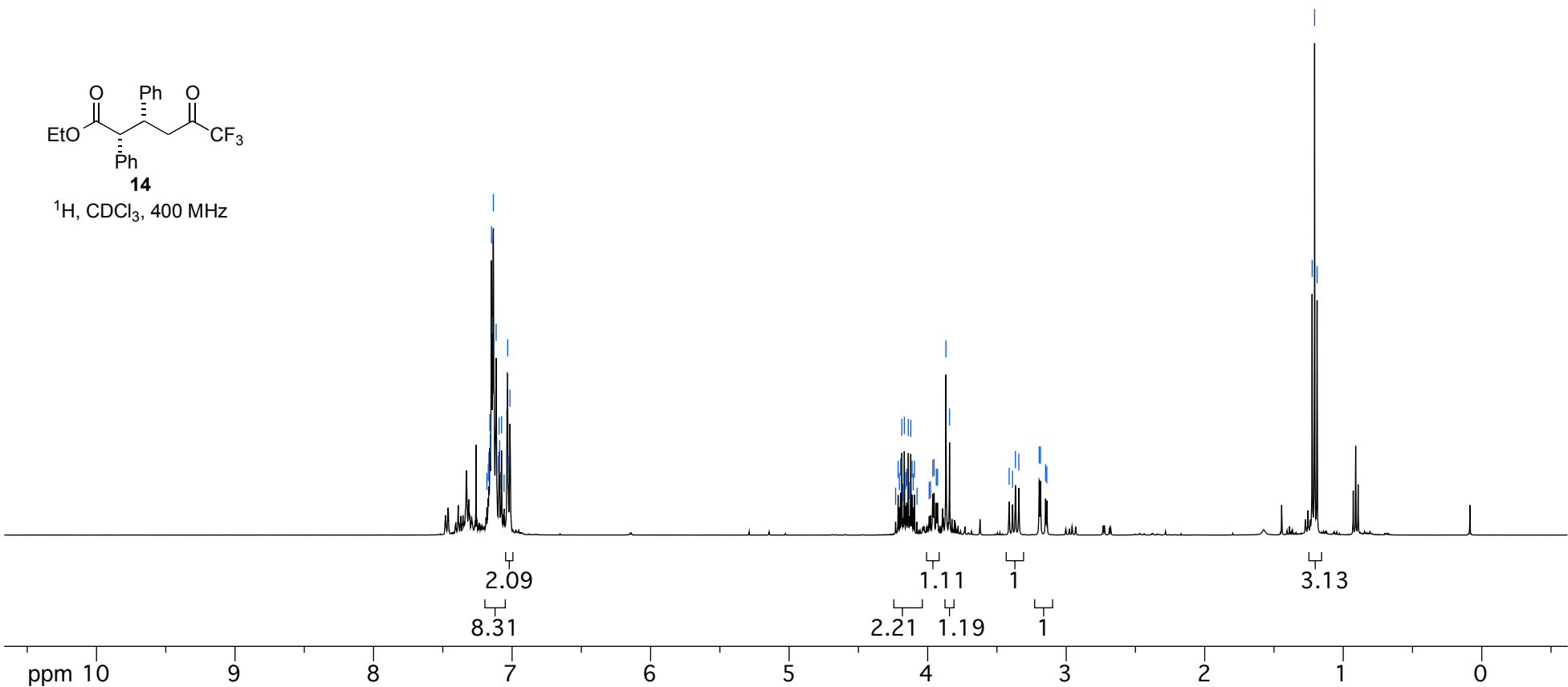
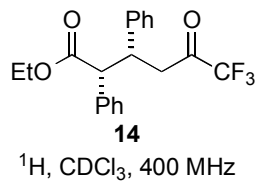


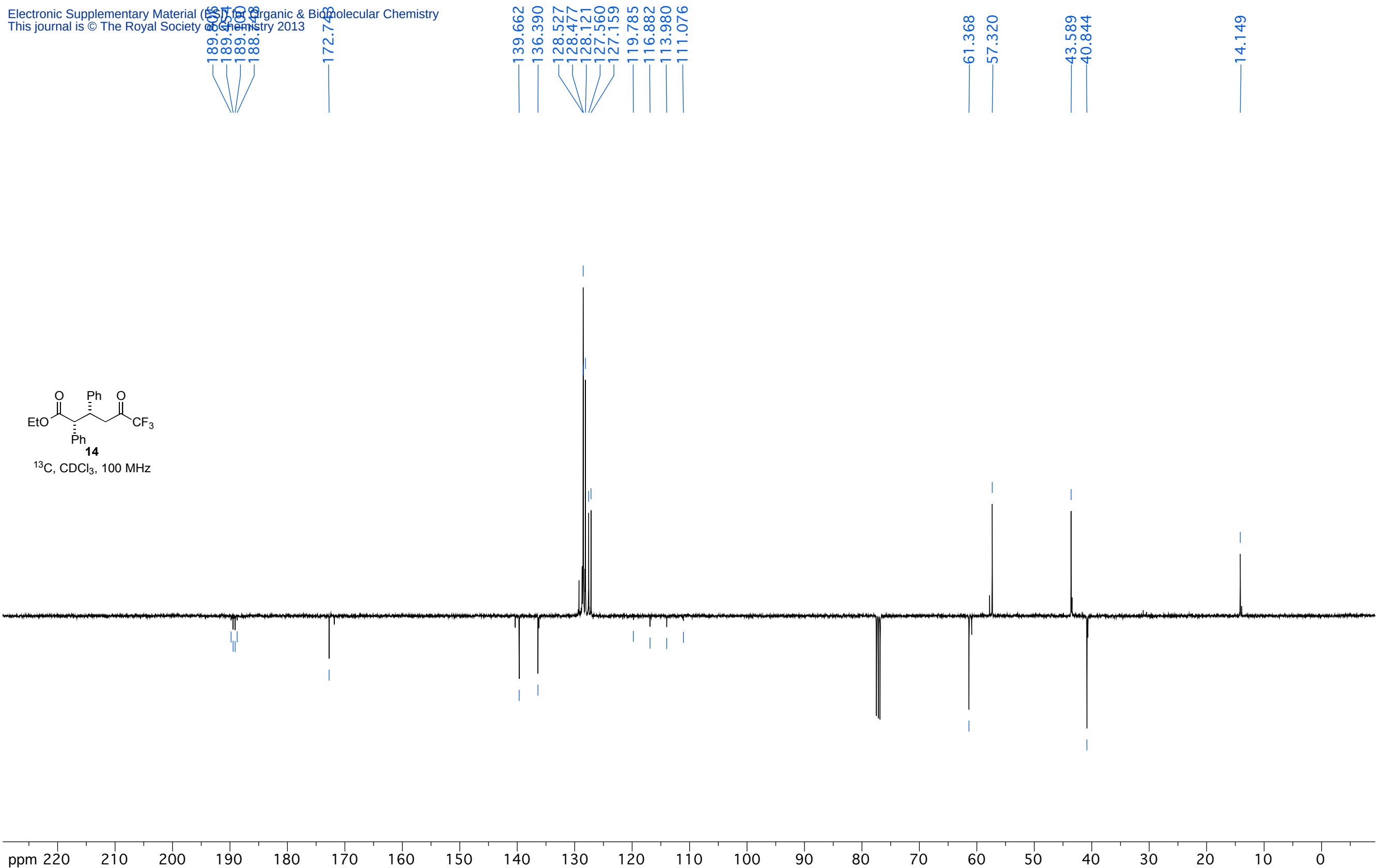
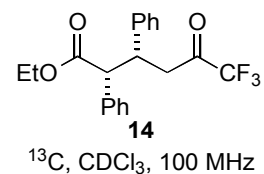


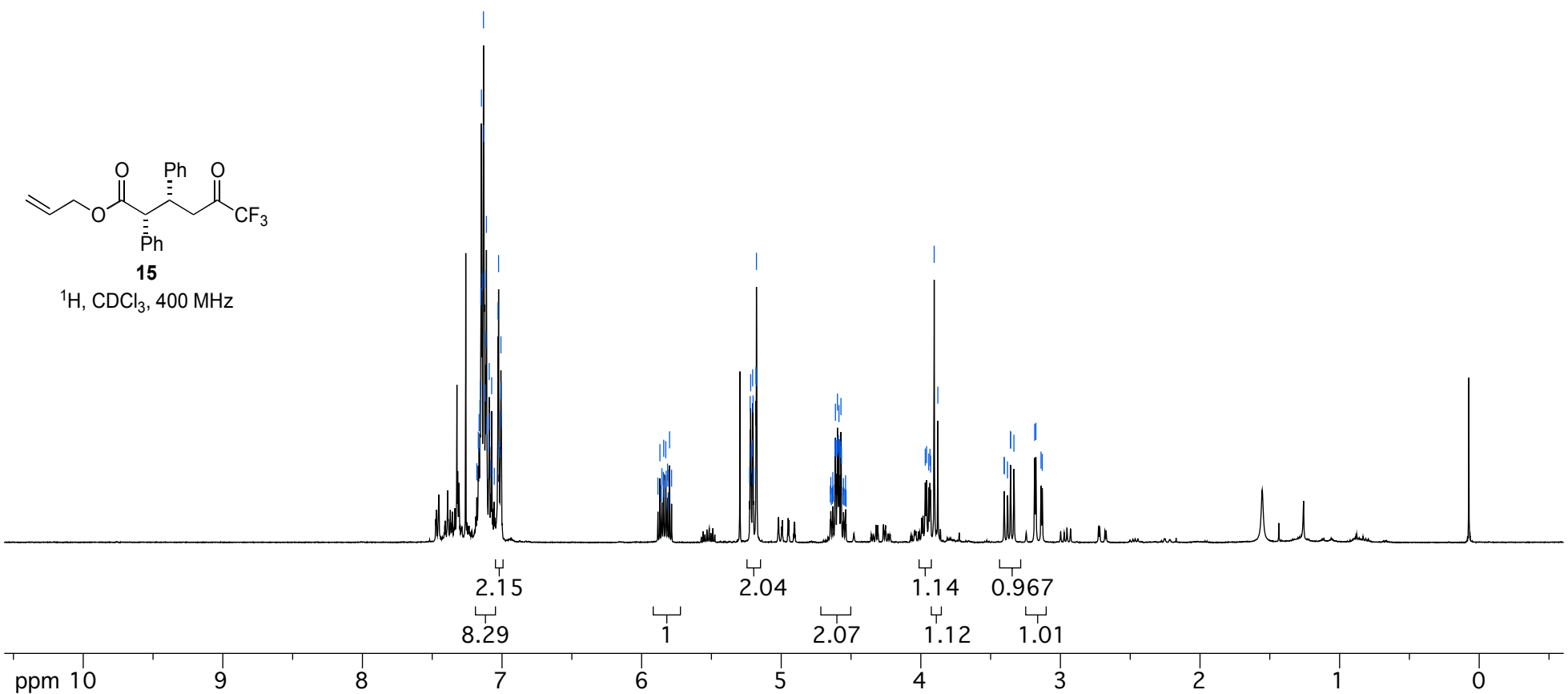


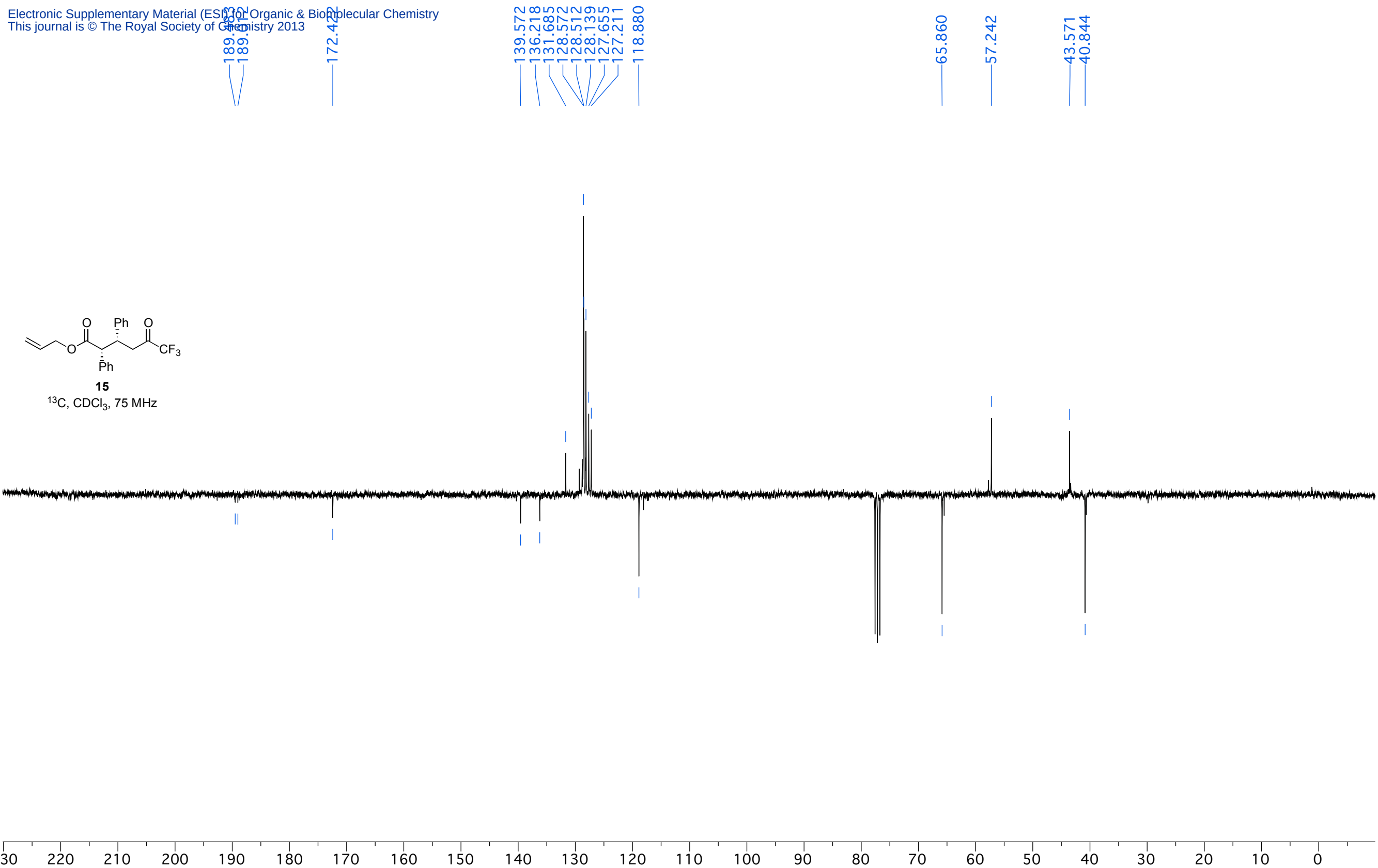
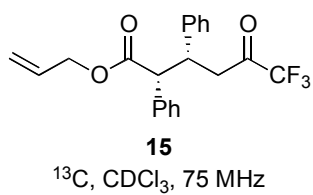
^{13}C , CDCl_3 , 75 MHz

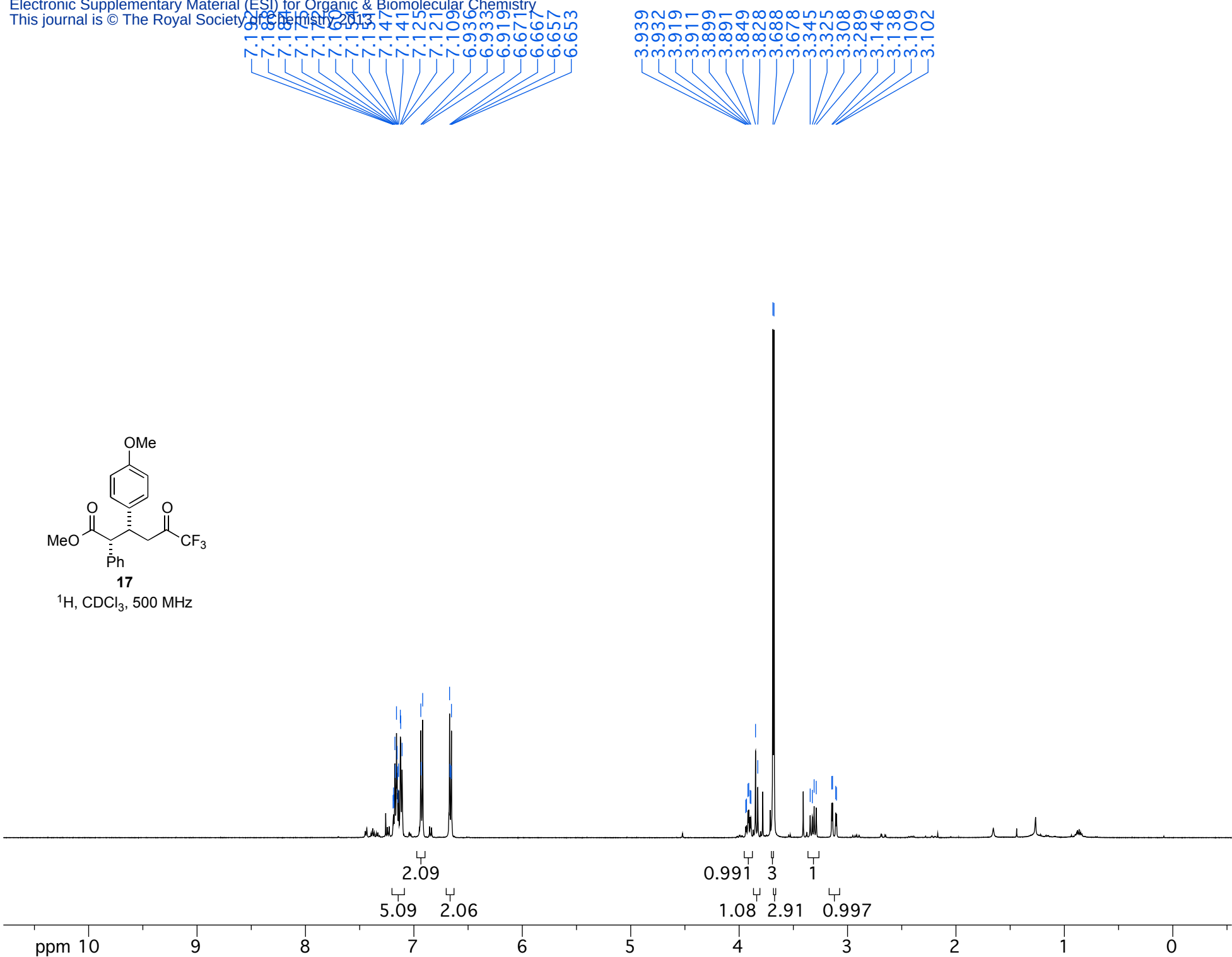
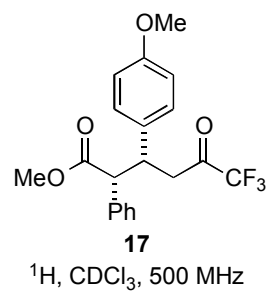


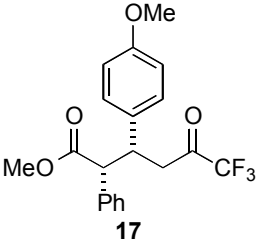




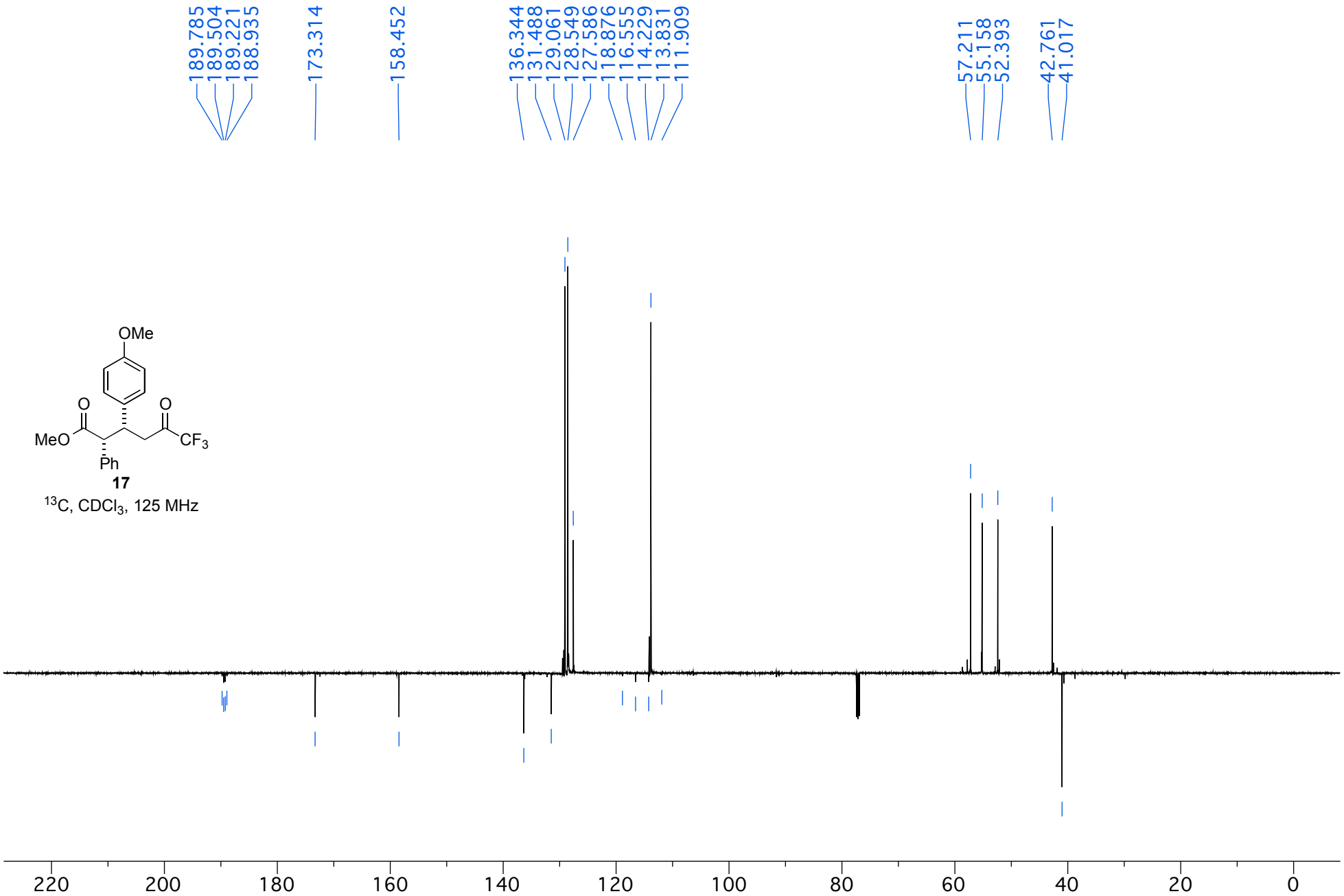


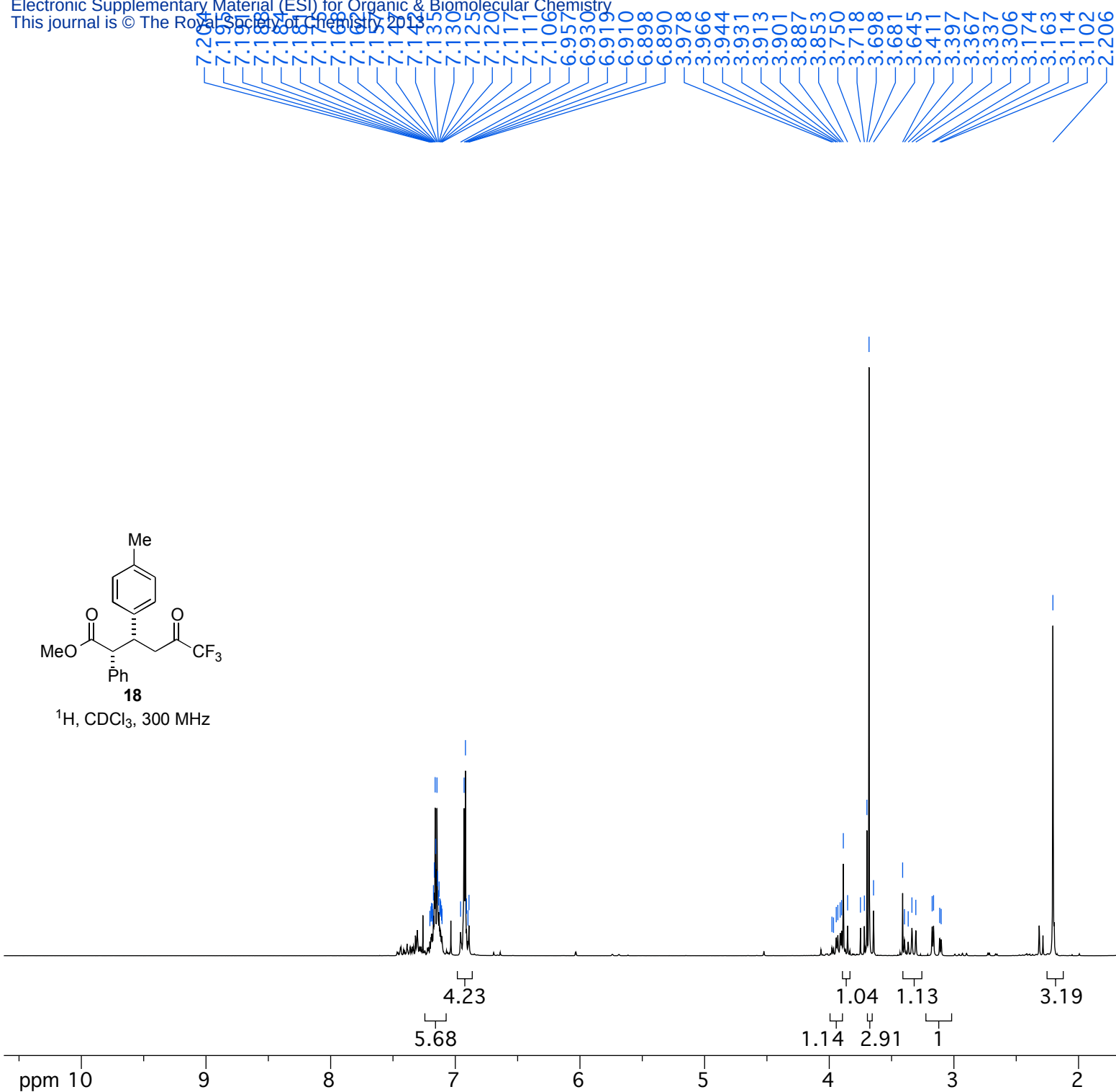
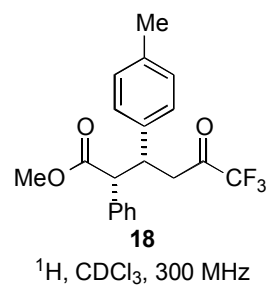


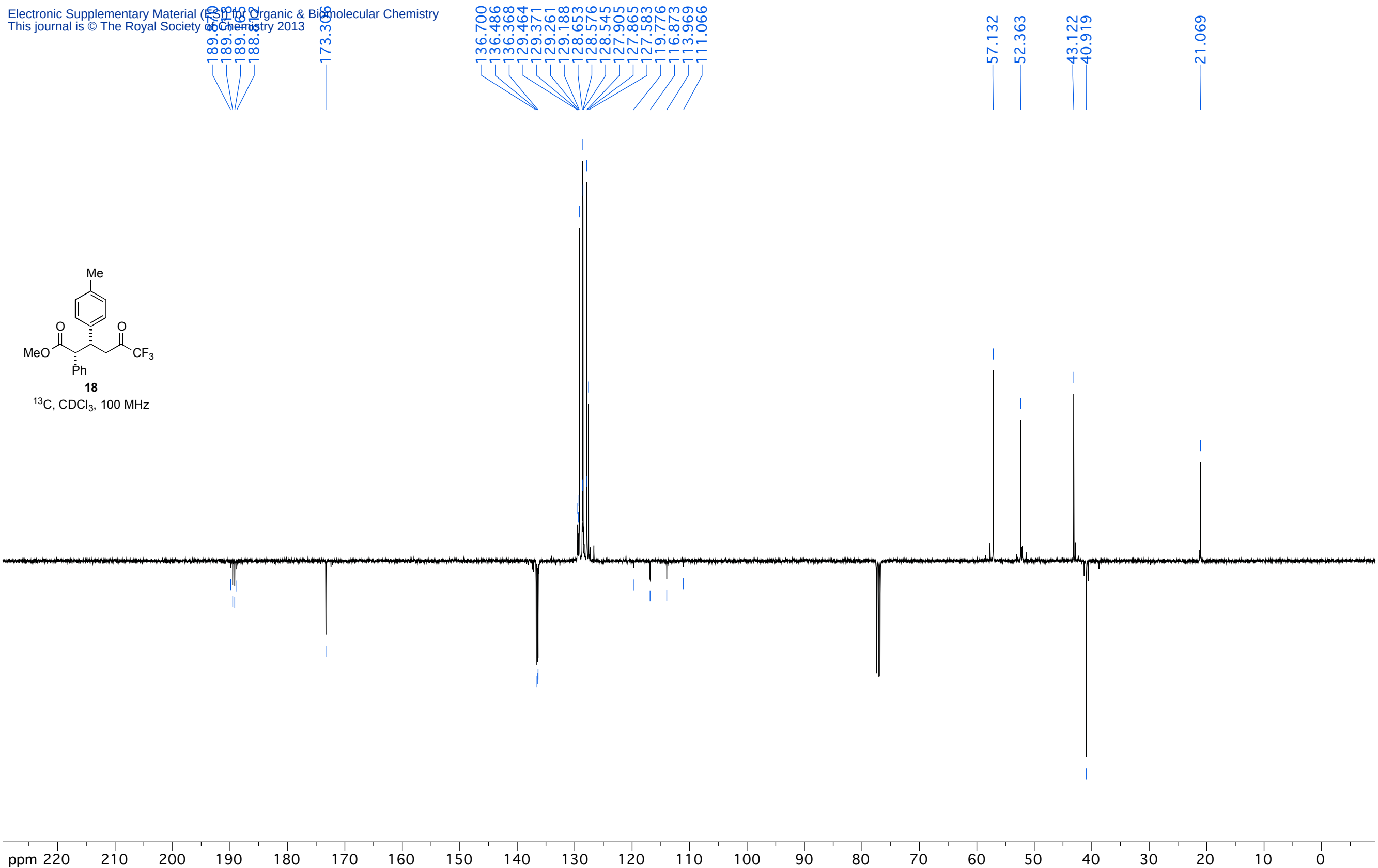
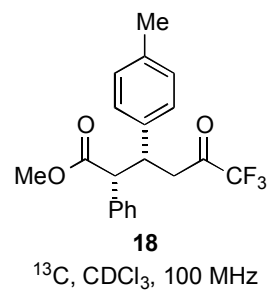


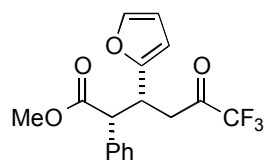


¹³C, CDCl₃, 125 MHz

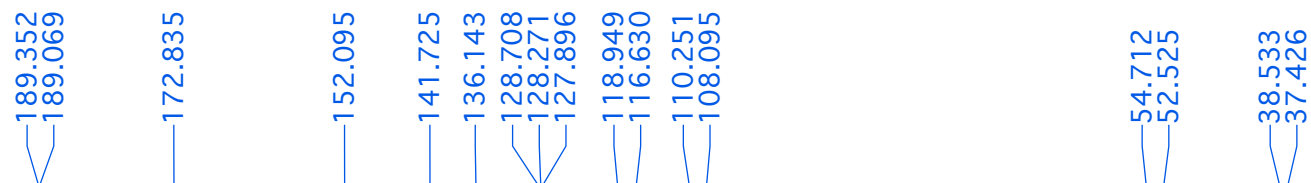


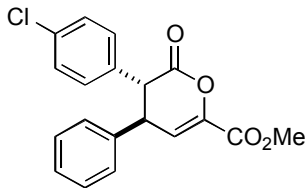




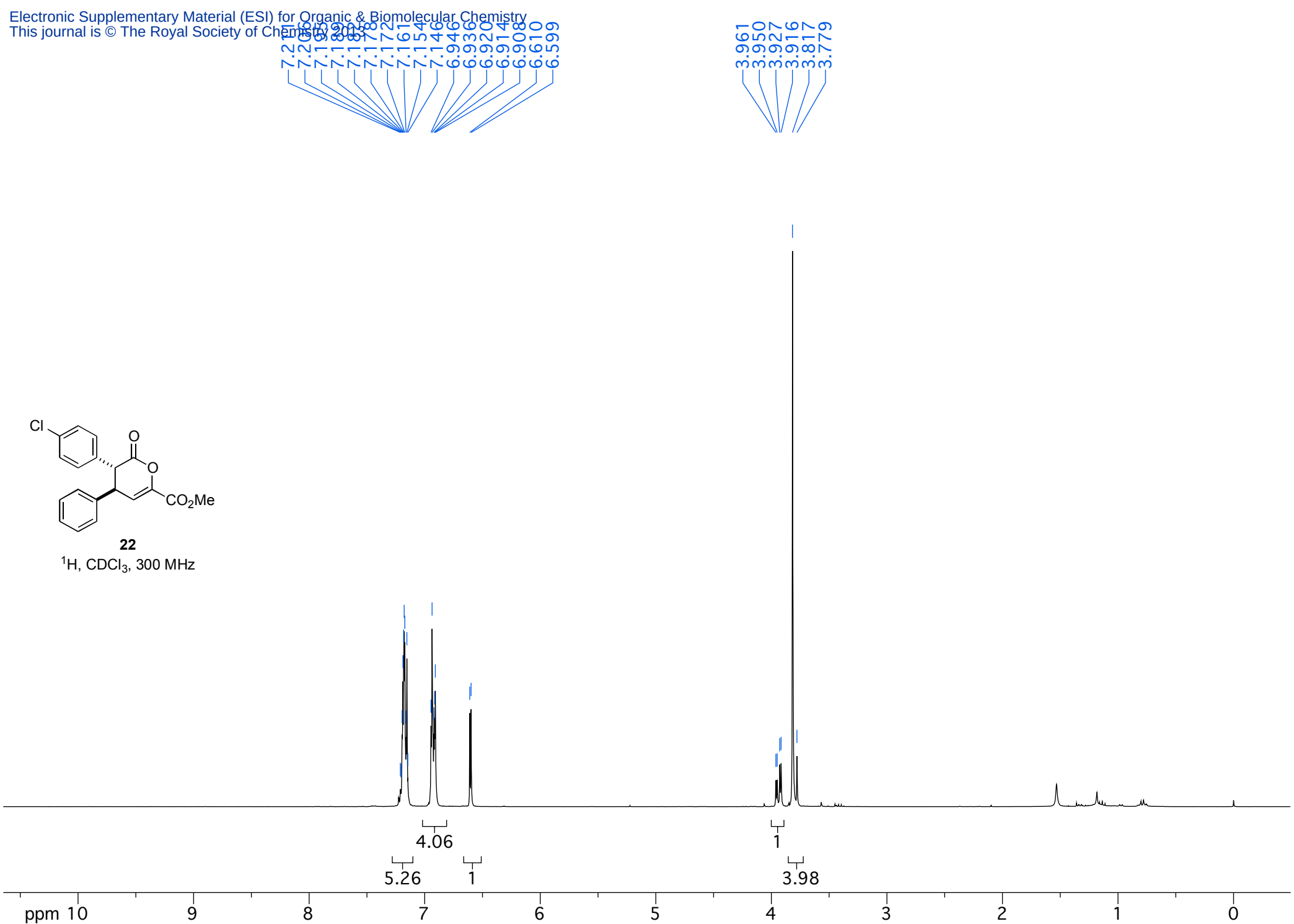


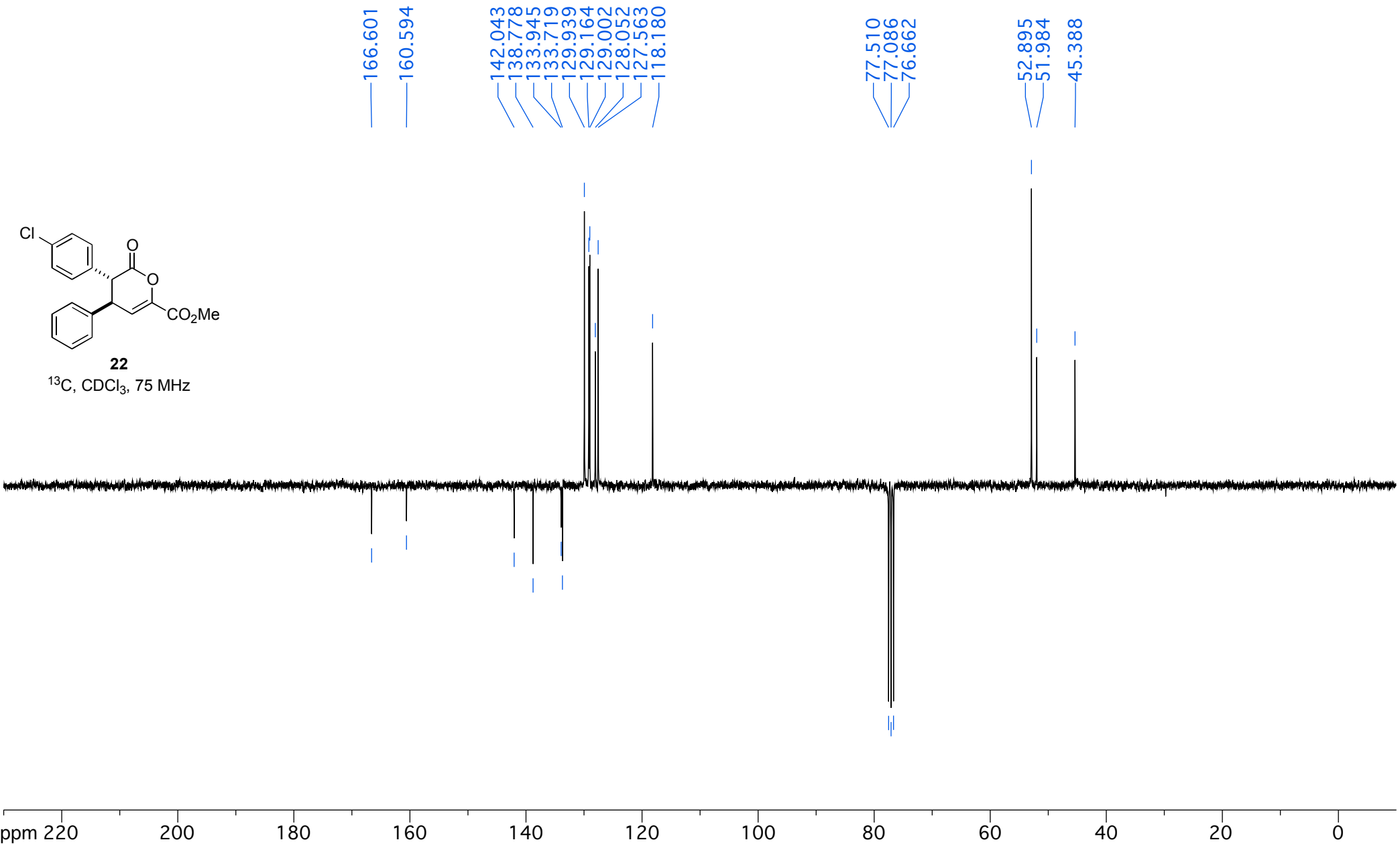
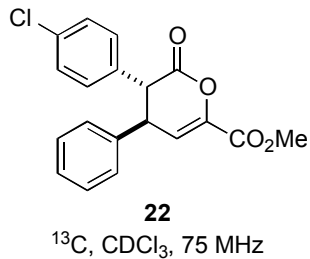
20
¹³C, CDCl₃, 125 MHz

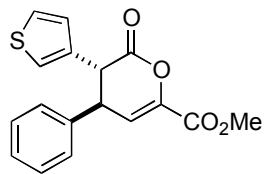




22
¹H, CDCl₃, 300 MHz

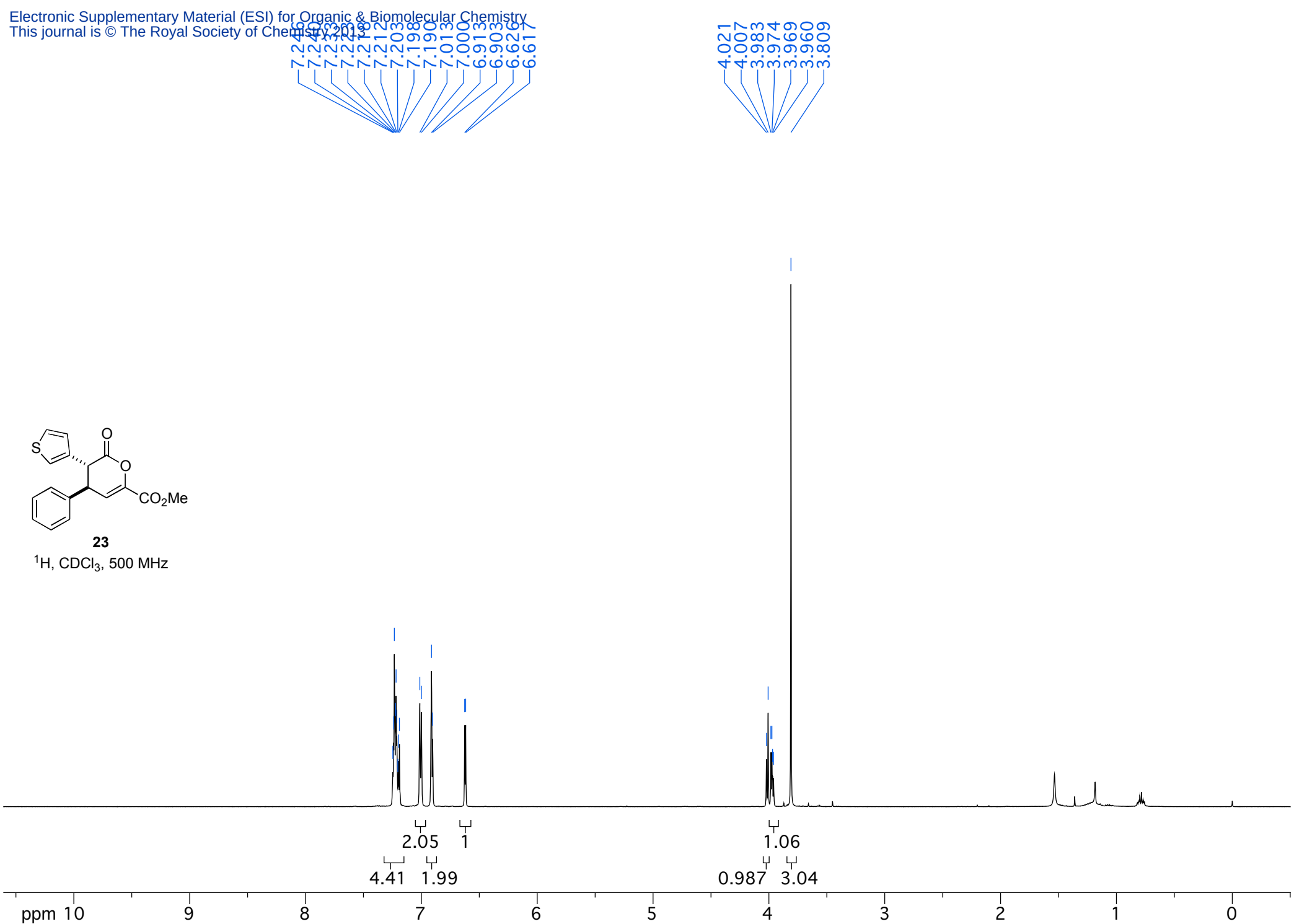


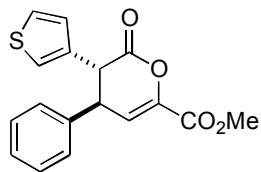




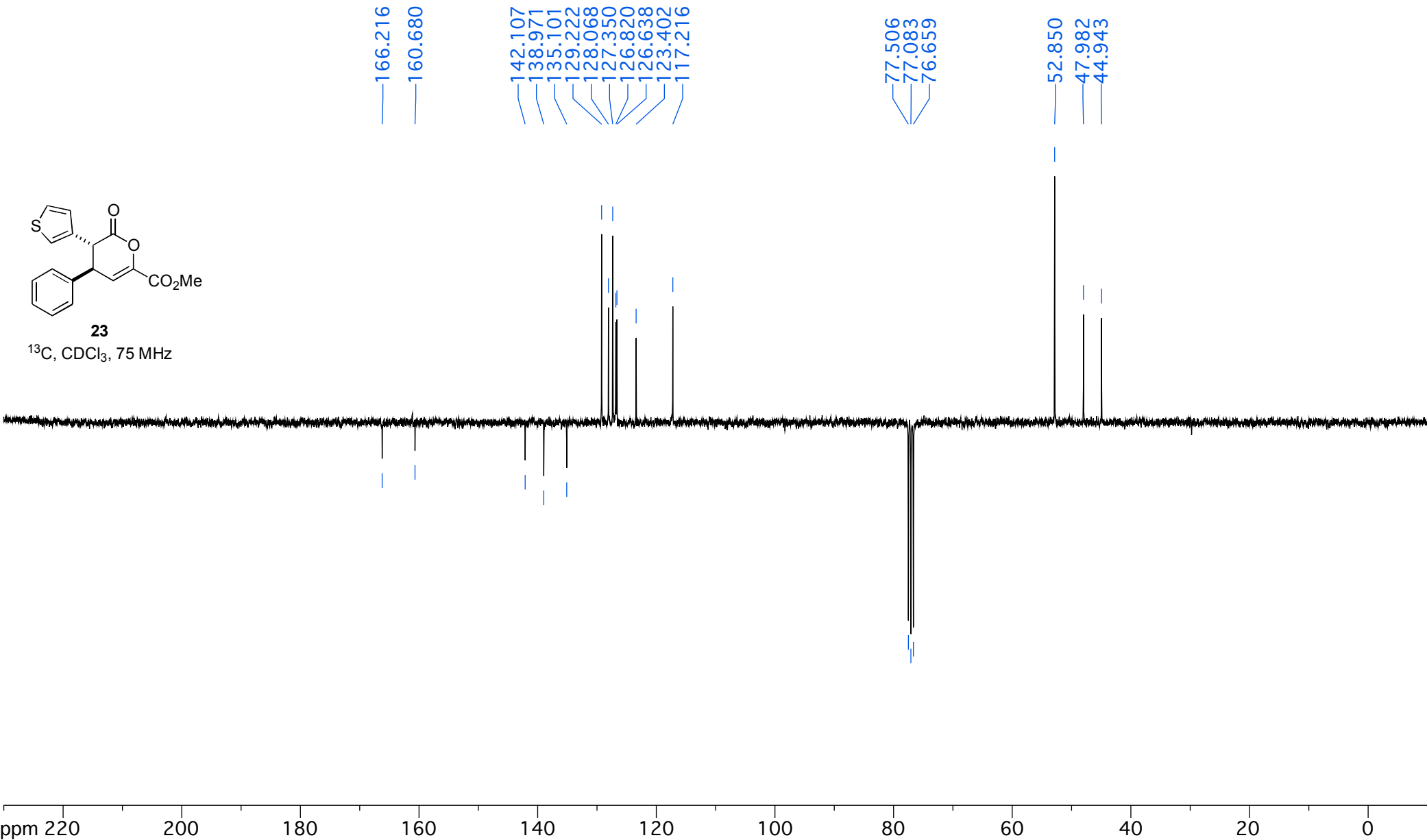
23

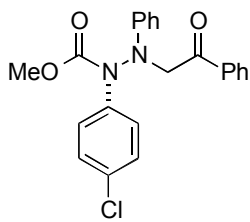
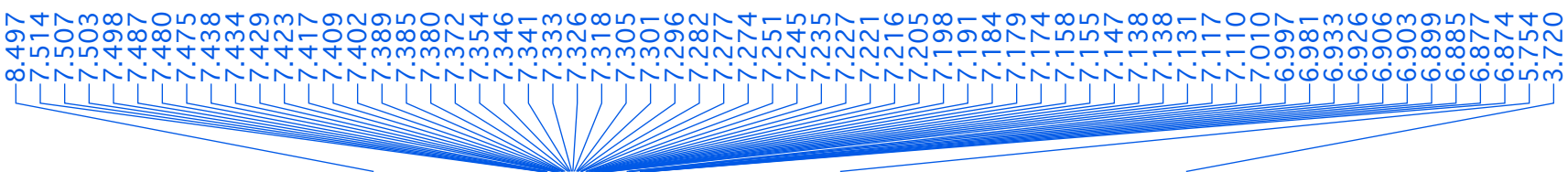
¹H, CDCl₃, 500 MHz



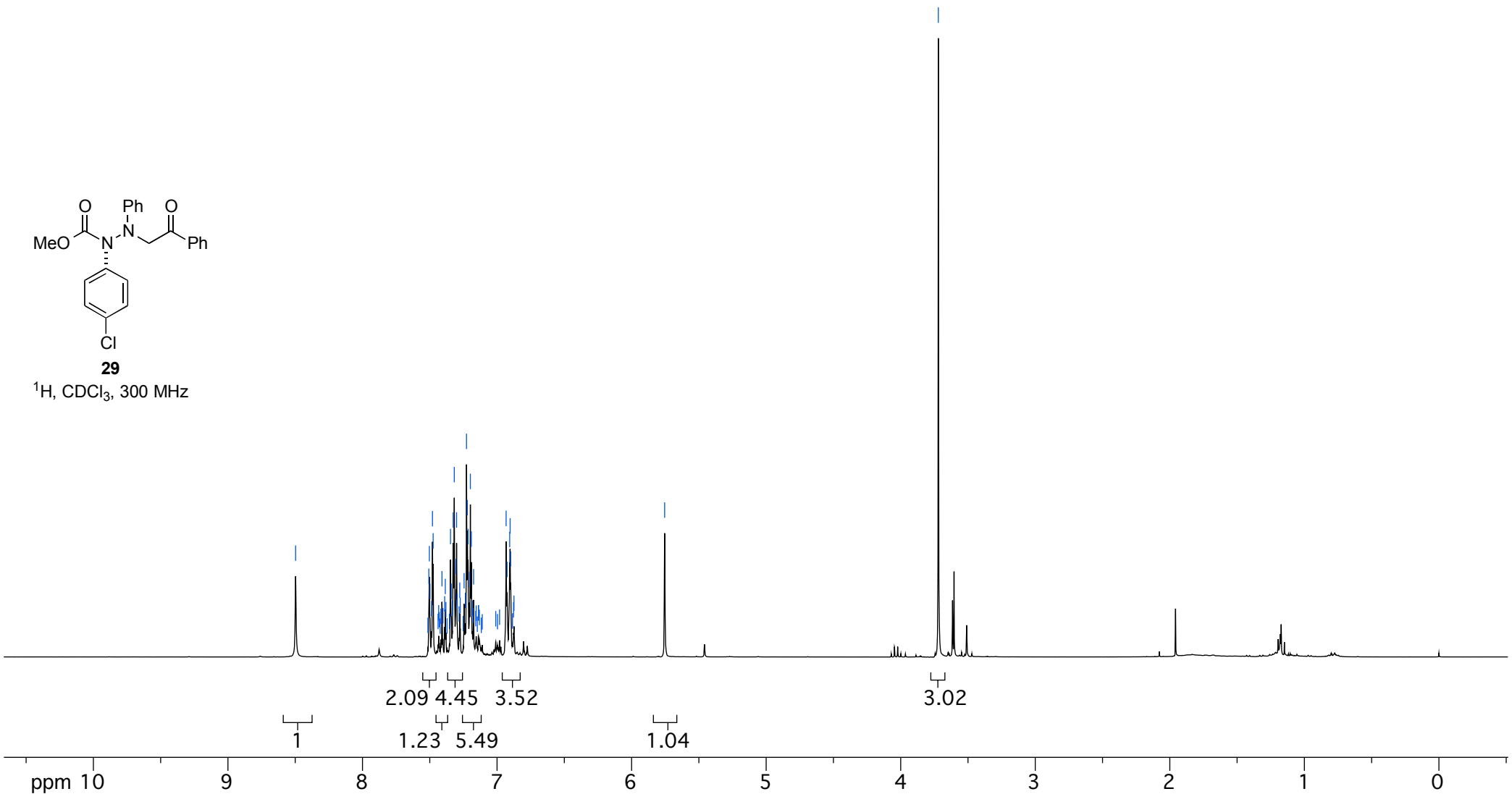


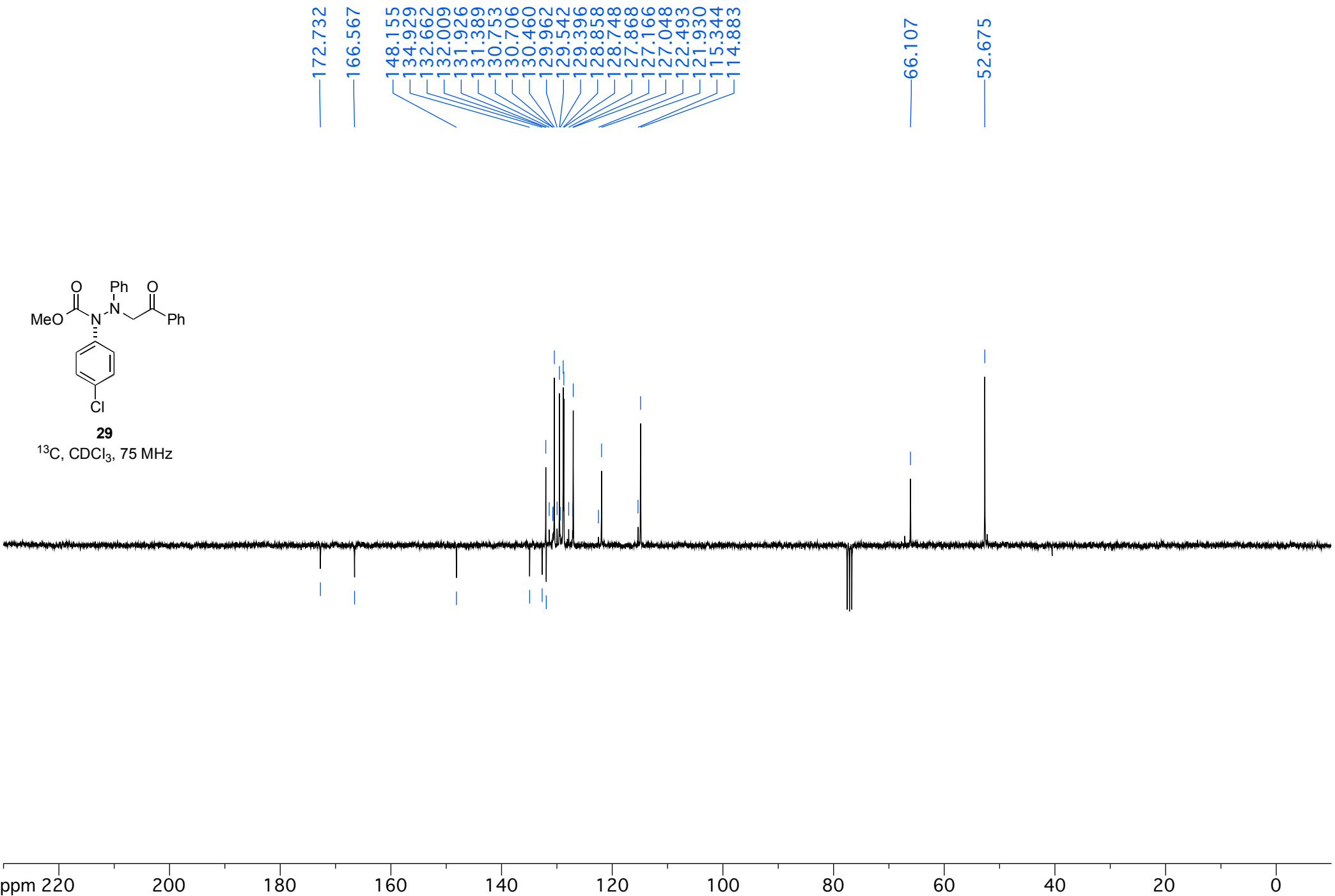
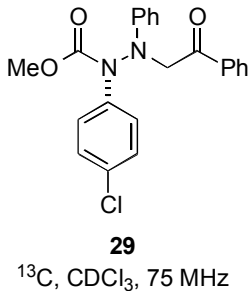
23
¹³C, CDCl₃, 75 MHz

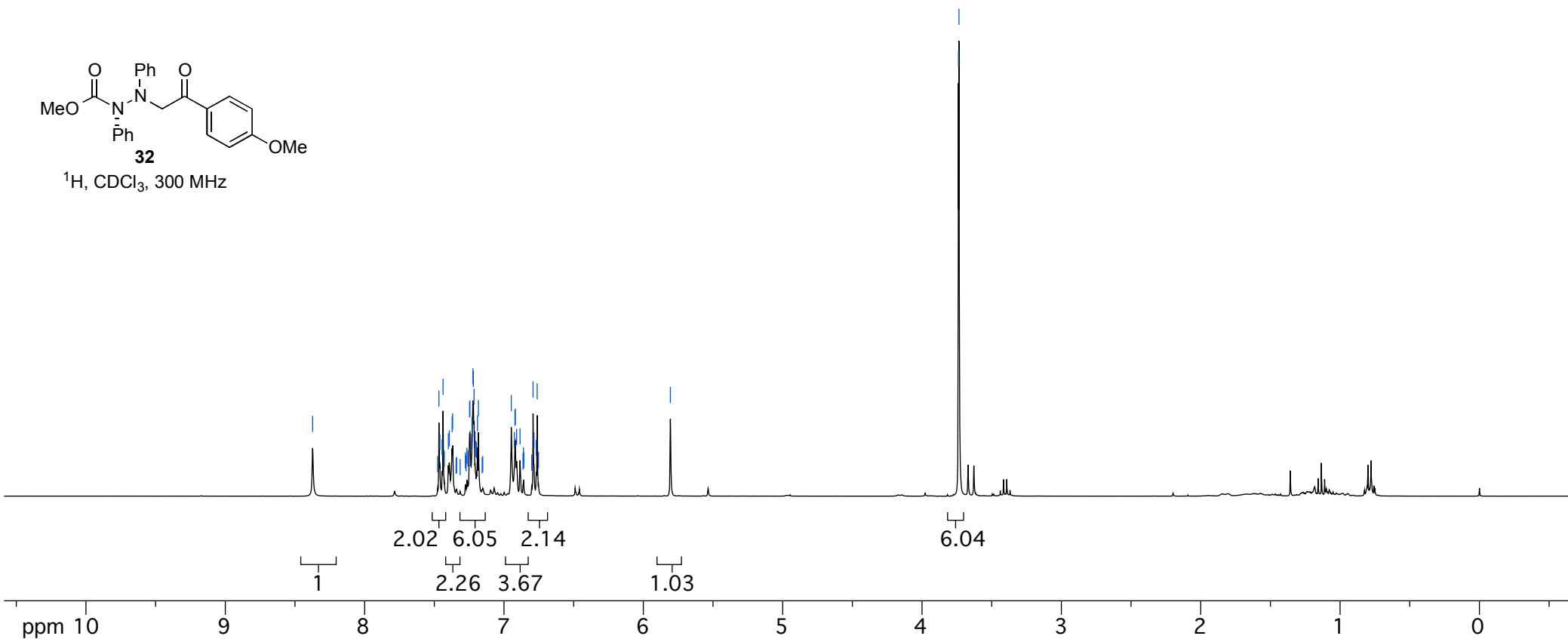


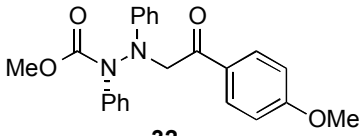


¹H, CDCl₃, 300 MHz

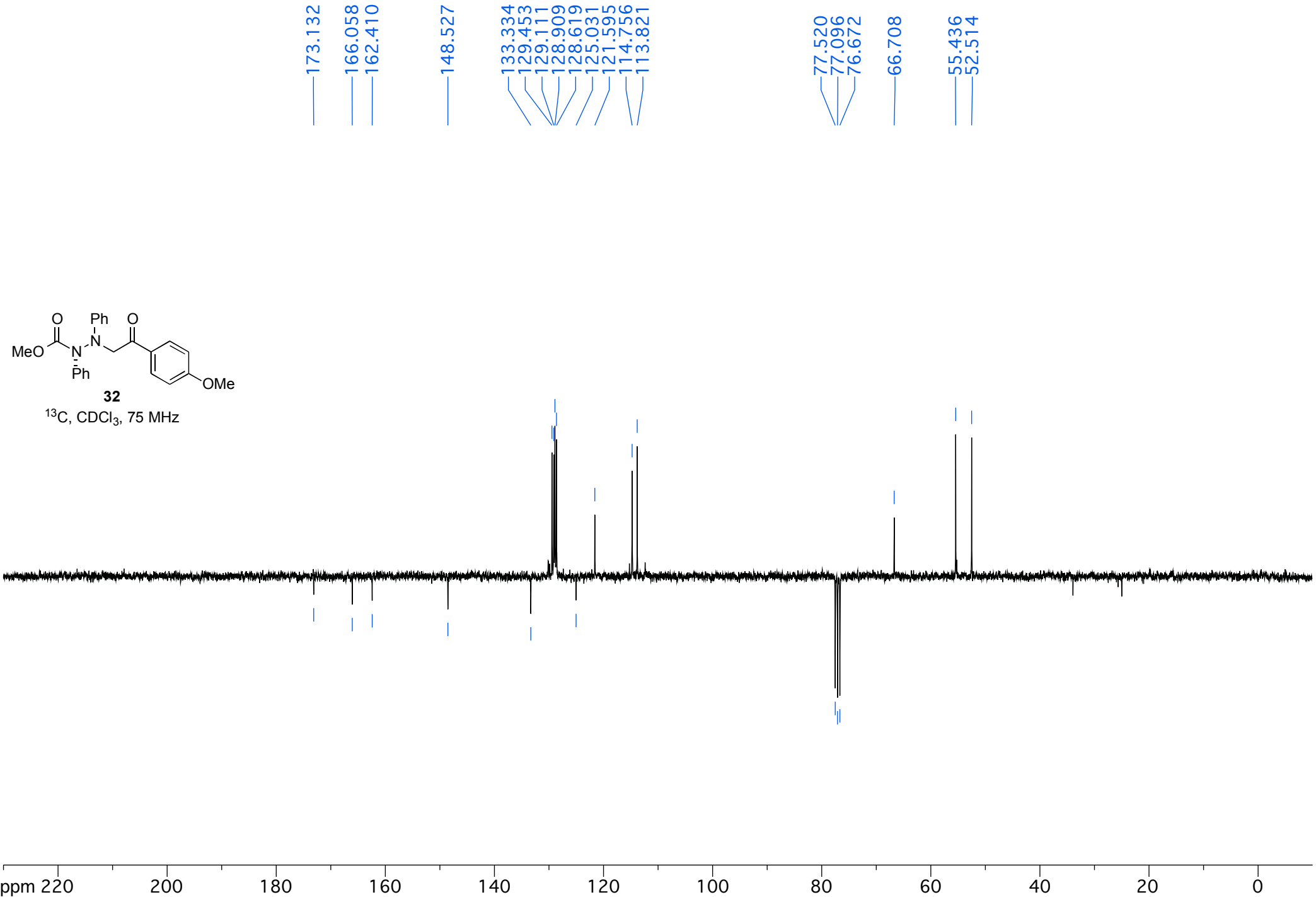


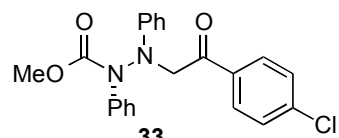
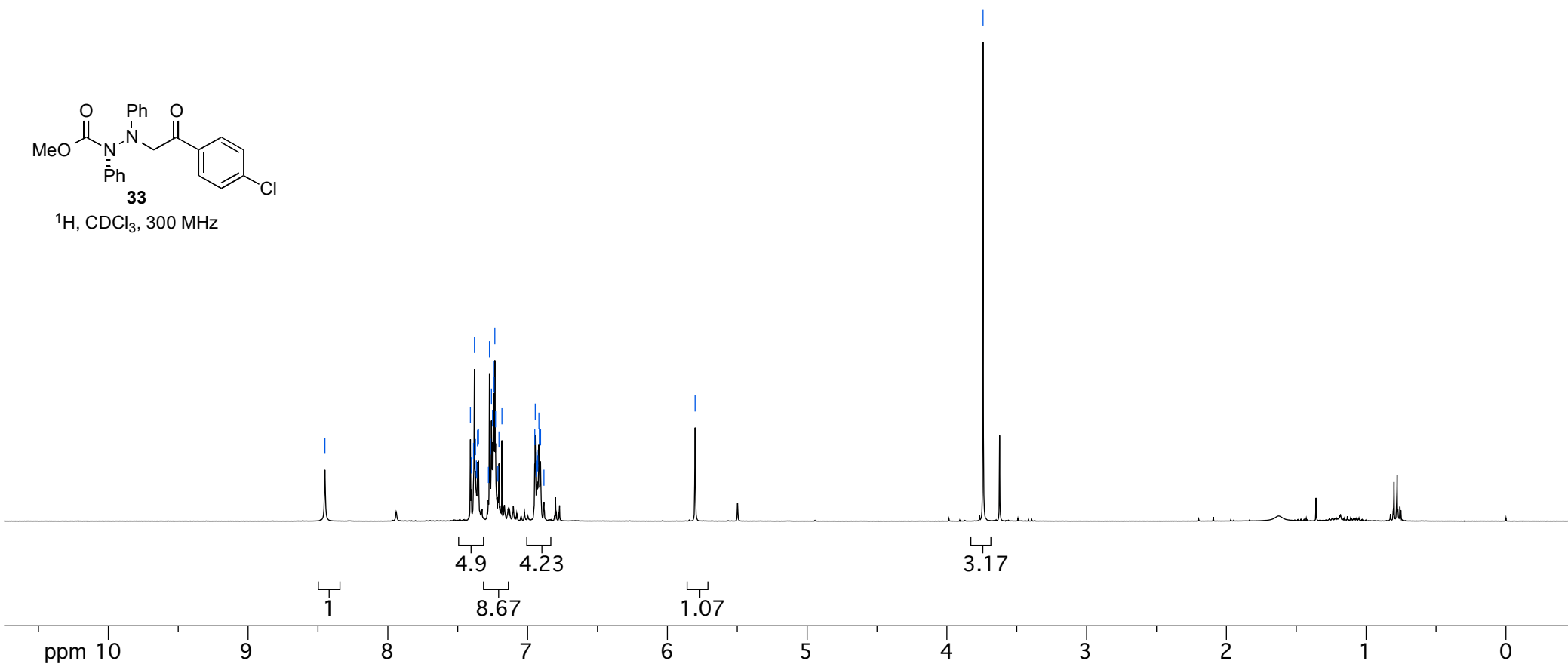


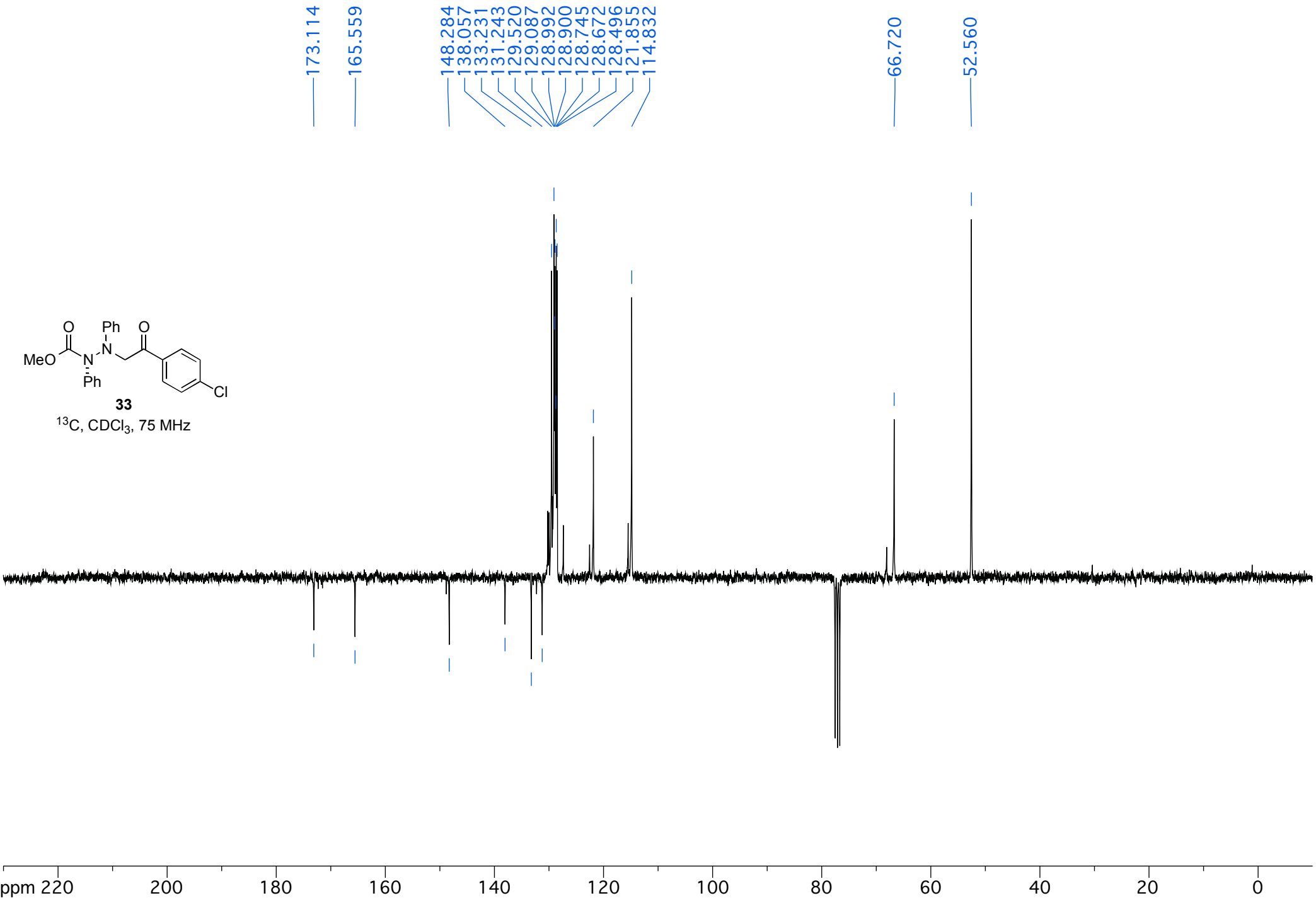
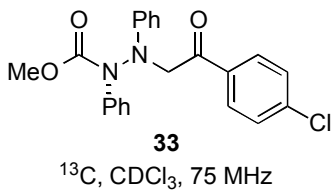


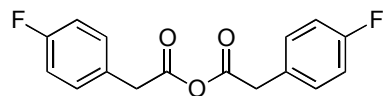


^{13}C , CDCl_3 , 75 MHz



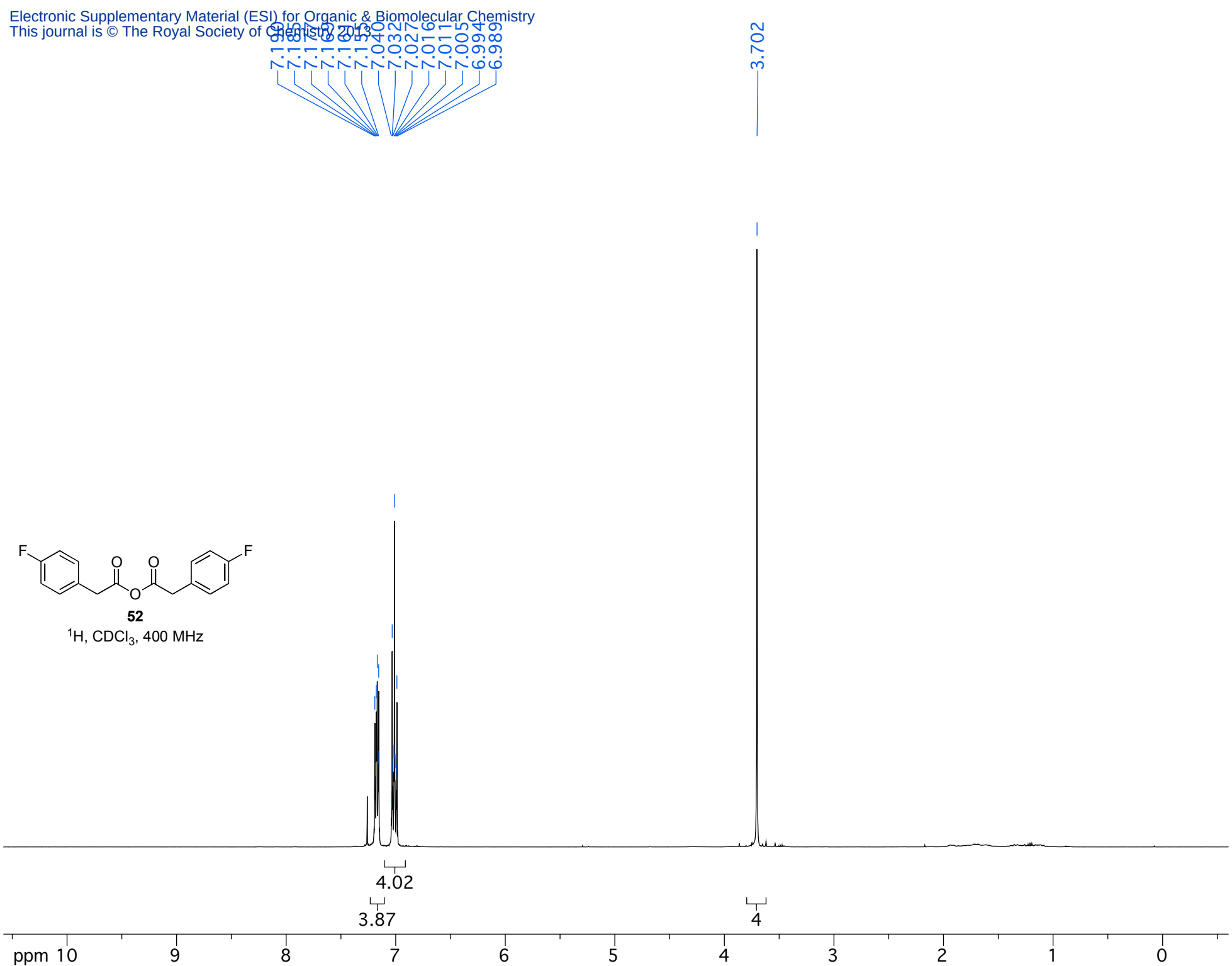
¹H, CDCl₃, 300 MHz

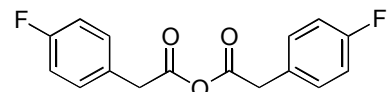




52

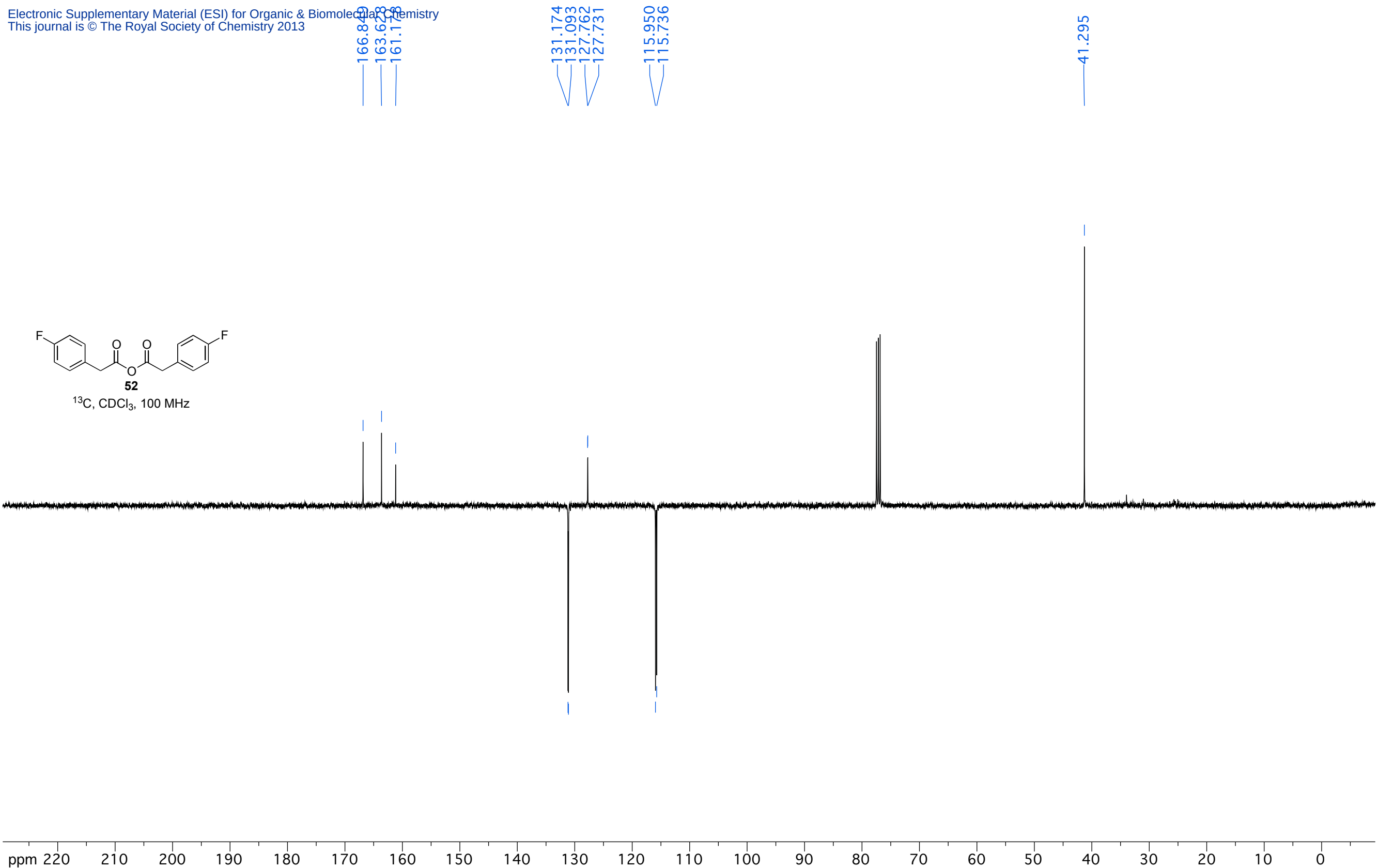
¹H, CDCl₃, 400 MHz





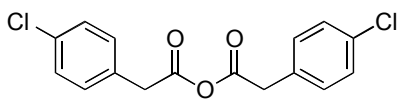
52

^{13}C , CDCl_3 , 100 MHz



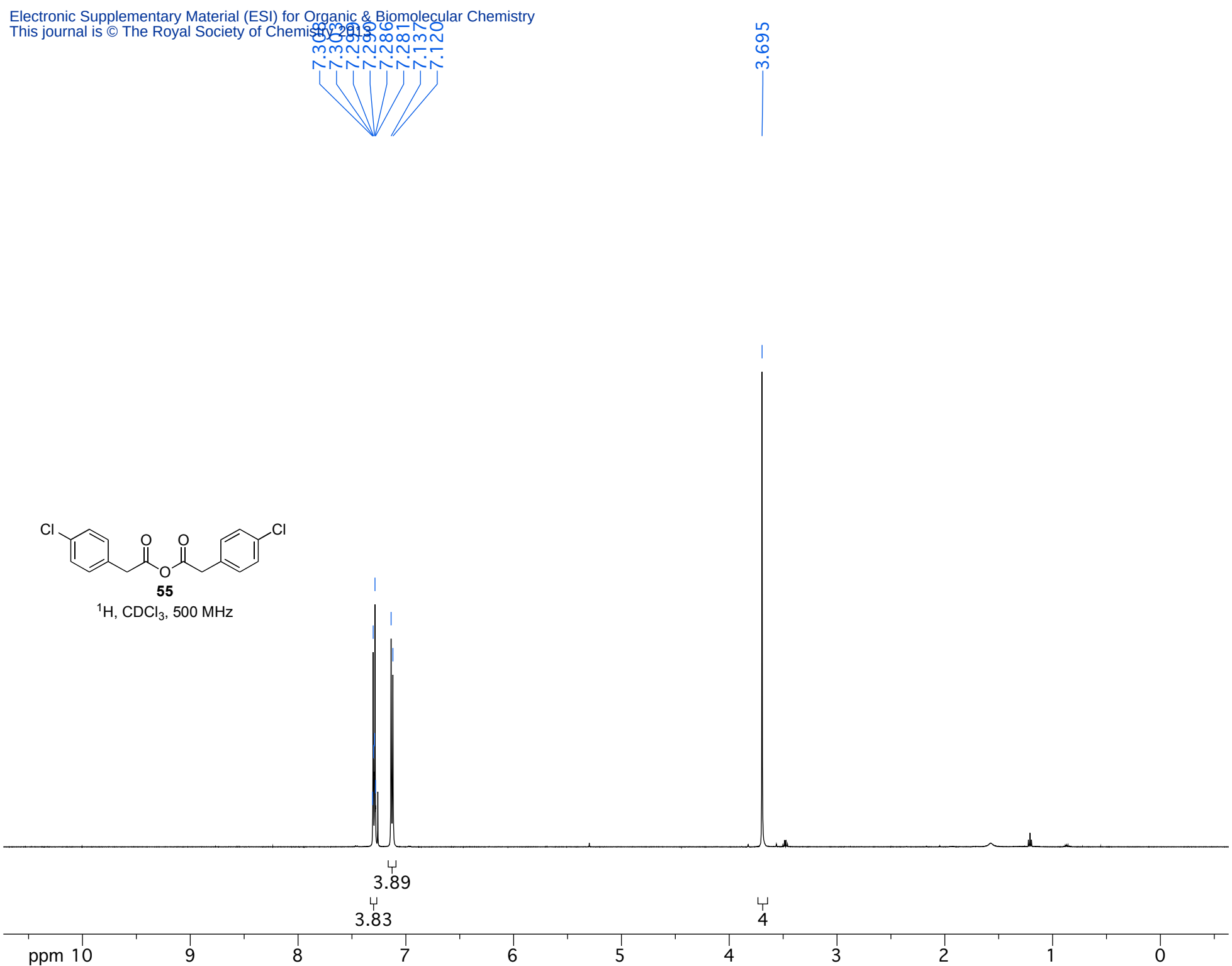
7.308
7.303
7.299
7.296
7.286
7.281
7.137
7.120

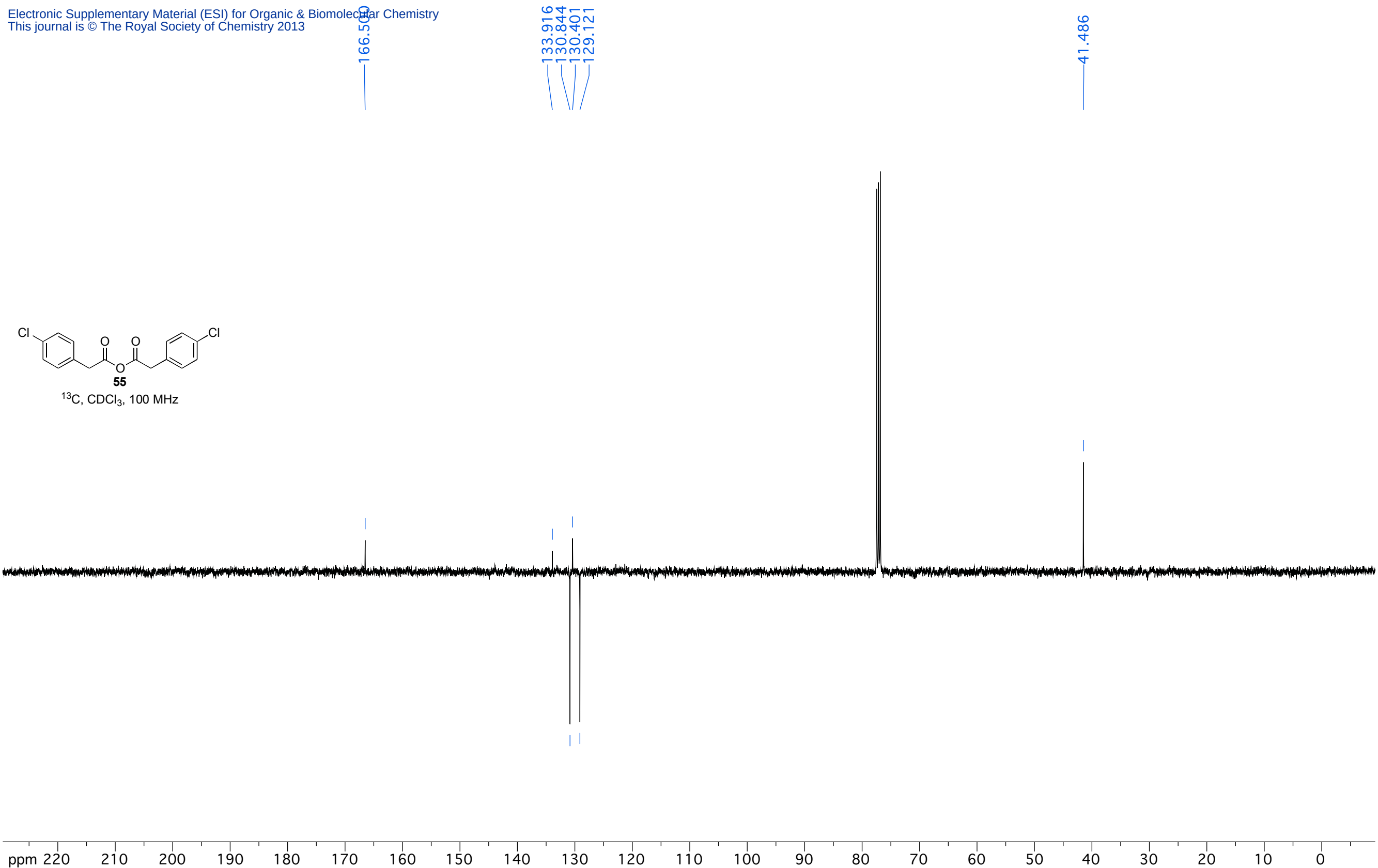
3.695

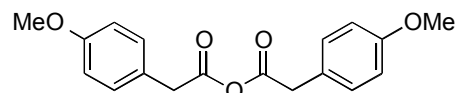


55

¹H, CDCl₃, 500 MHz

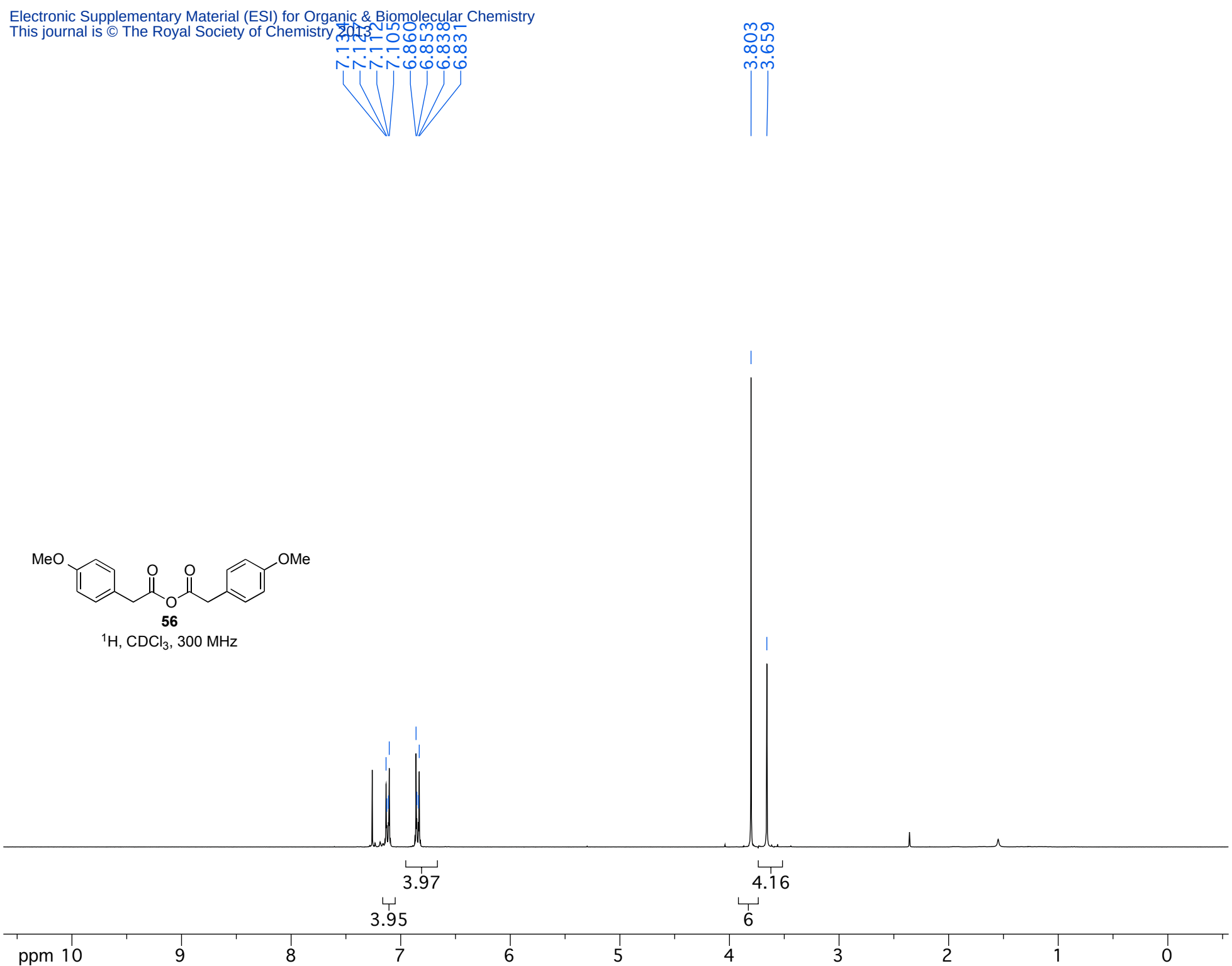


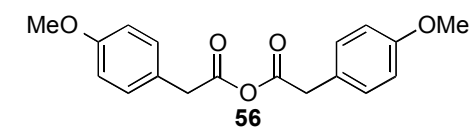




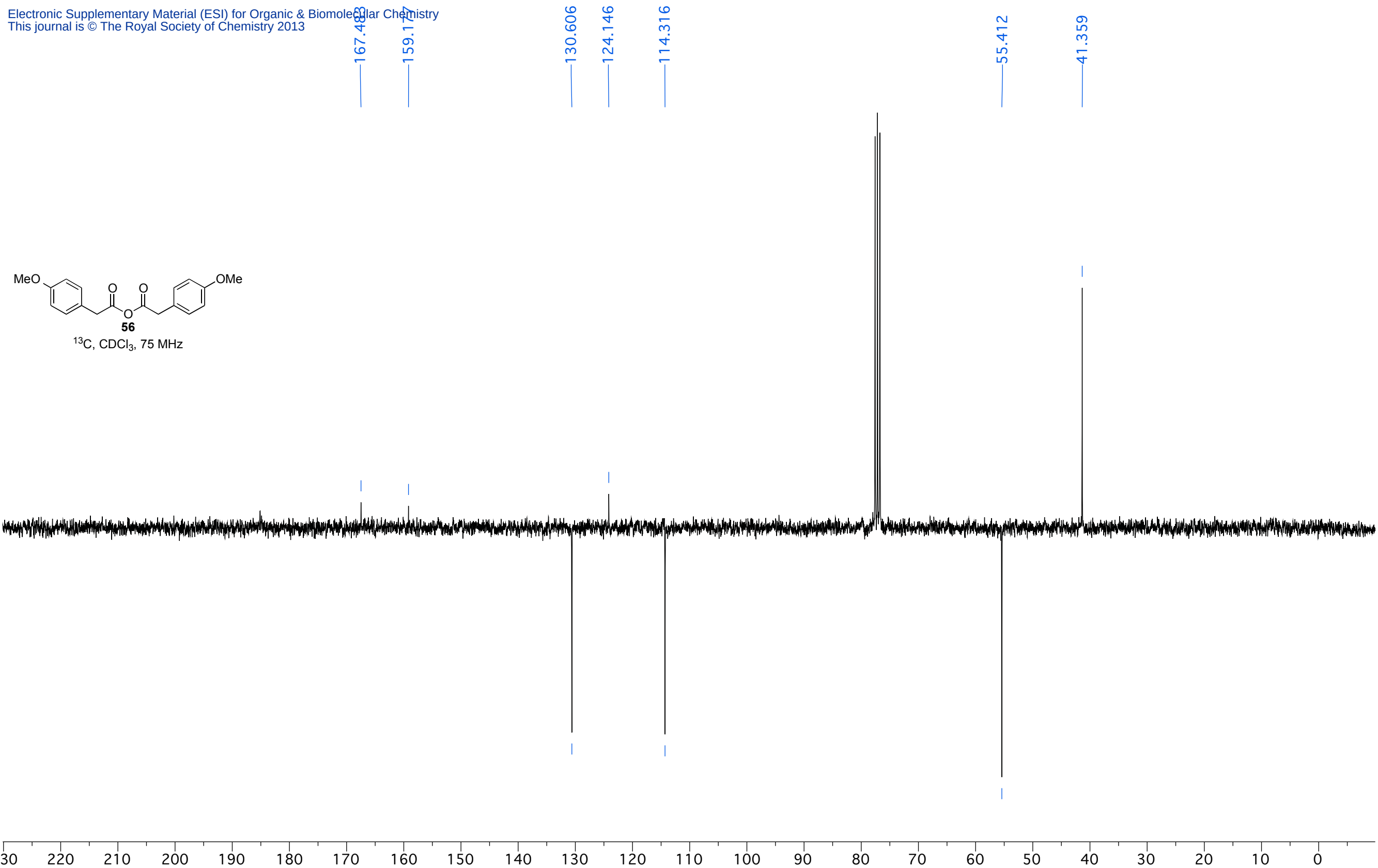
56

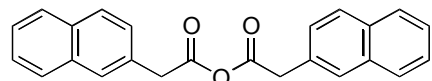
¹H, CDCl₃, 300 MHz





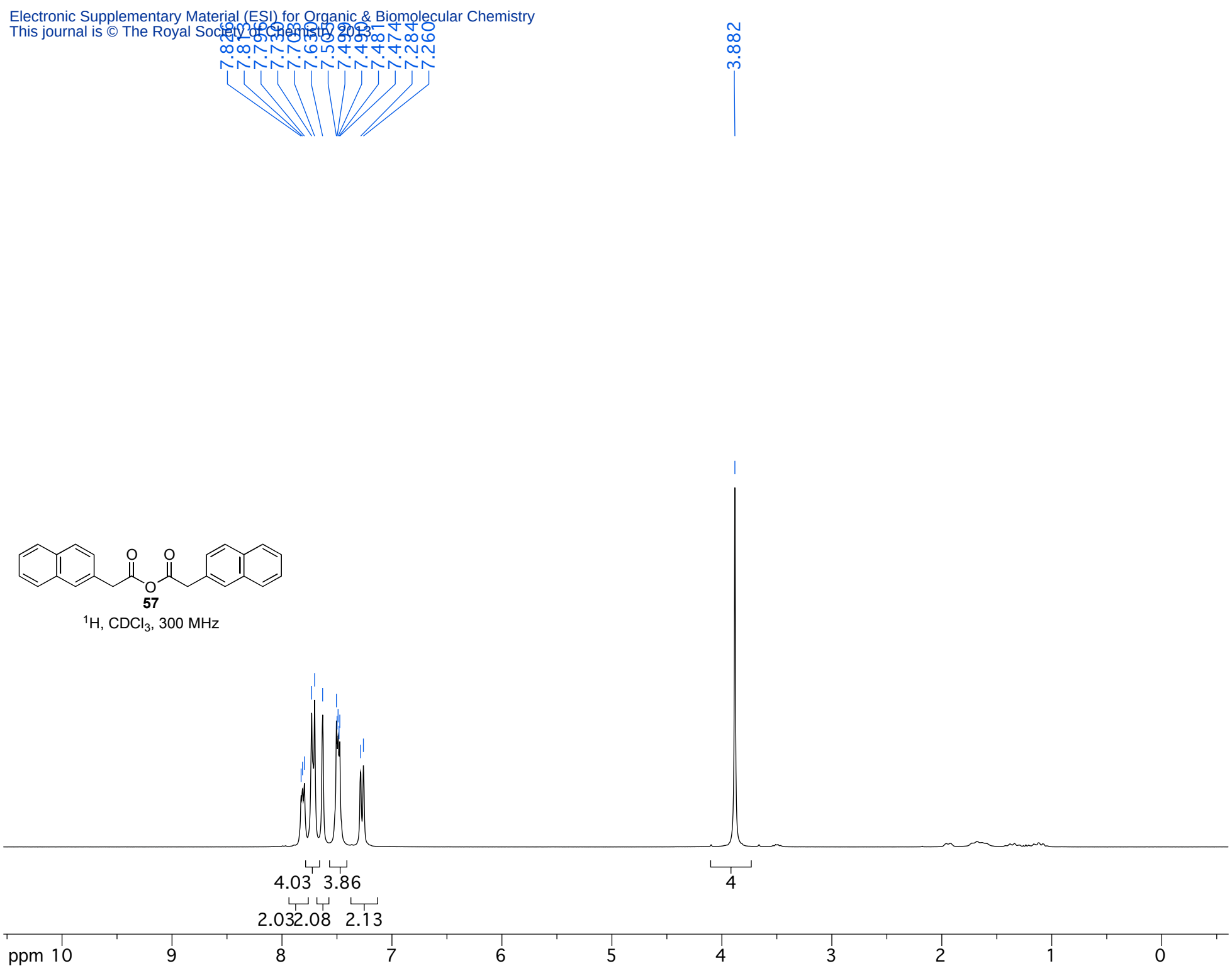
^{13}C , CDCl_3 , 75 MHz

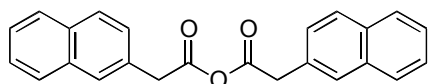




57

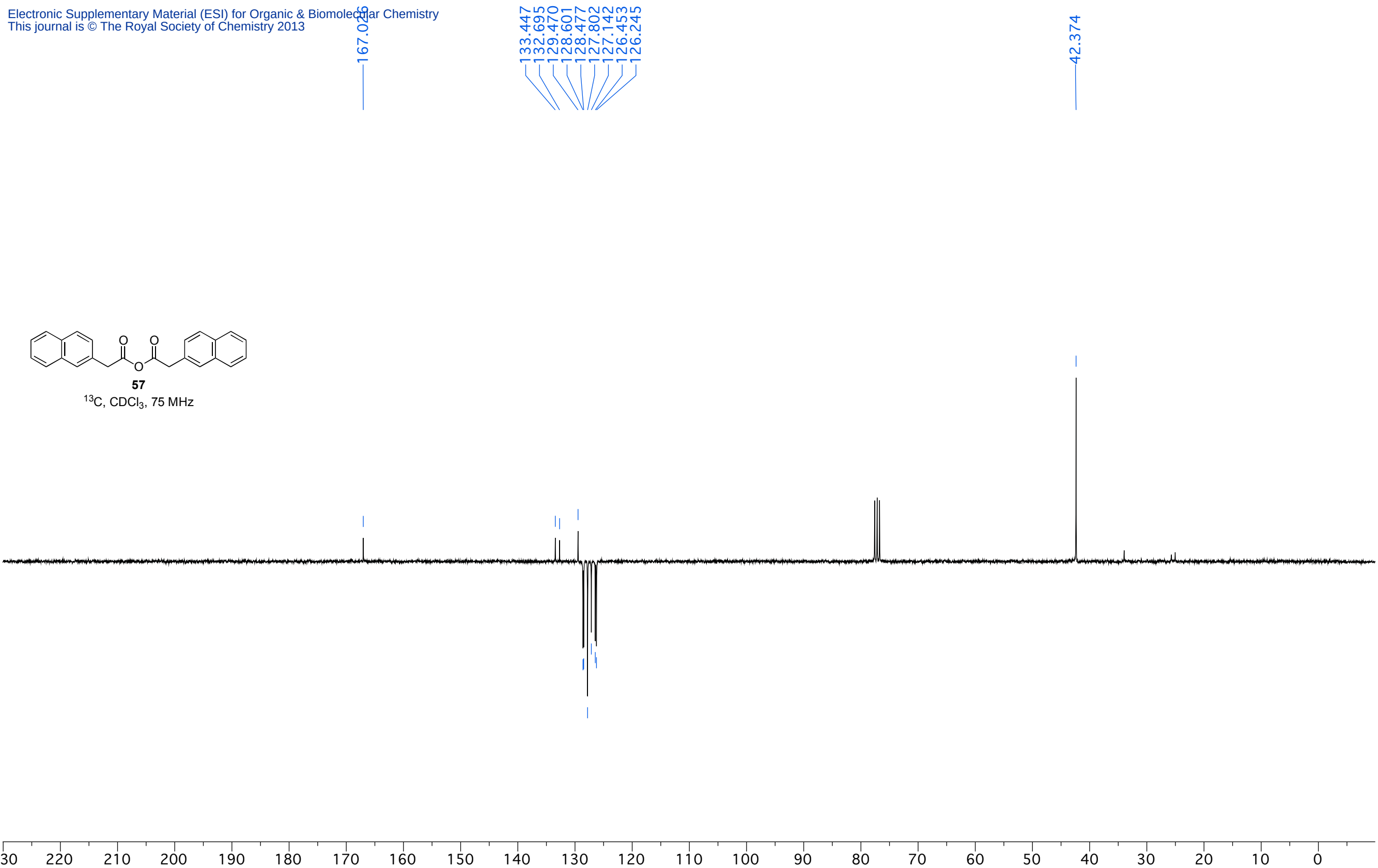
¹H, CDCl₃, 300 MHz

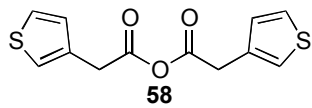




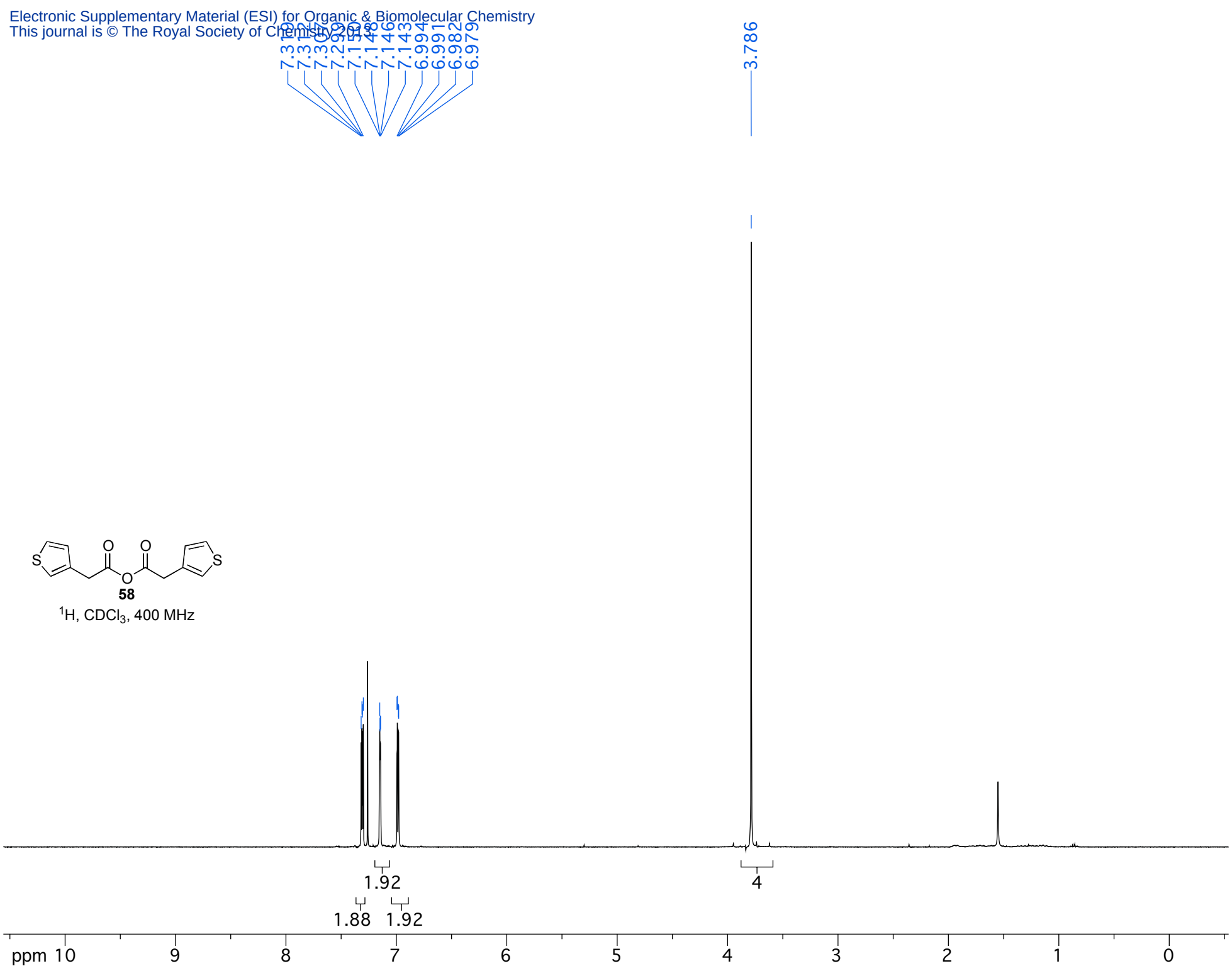
57

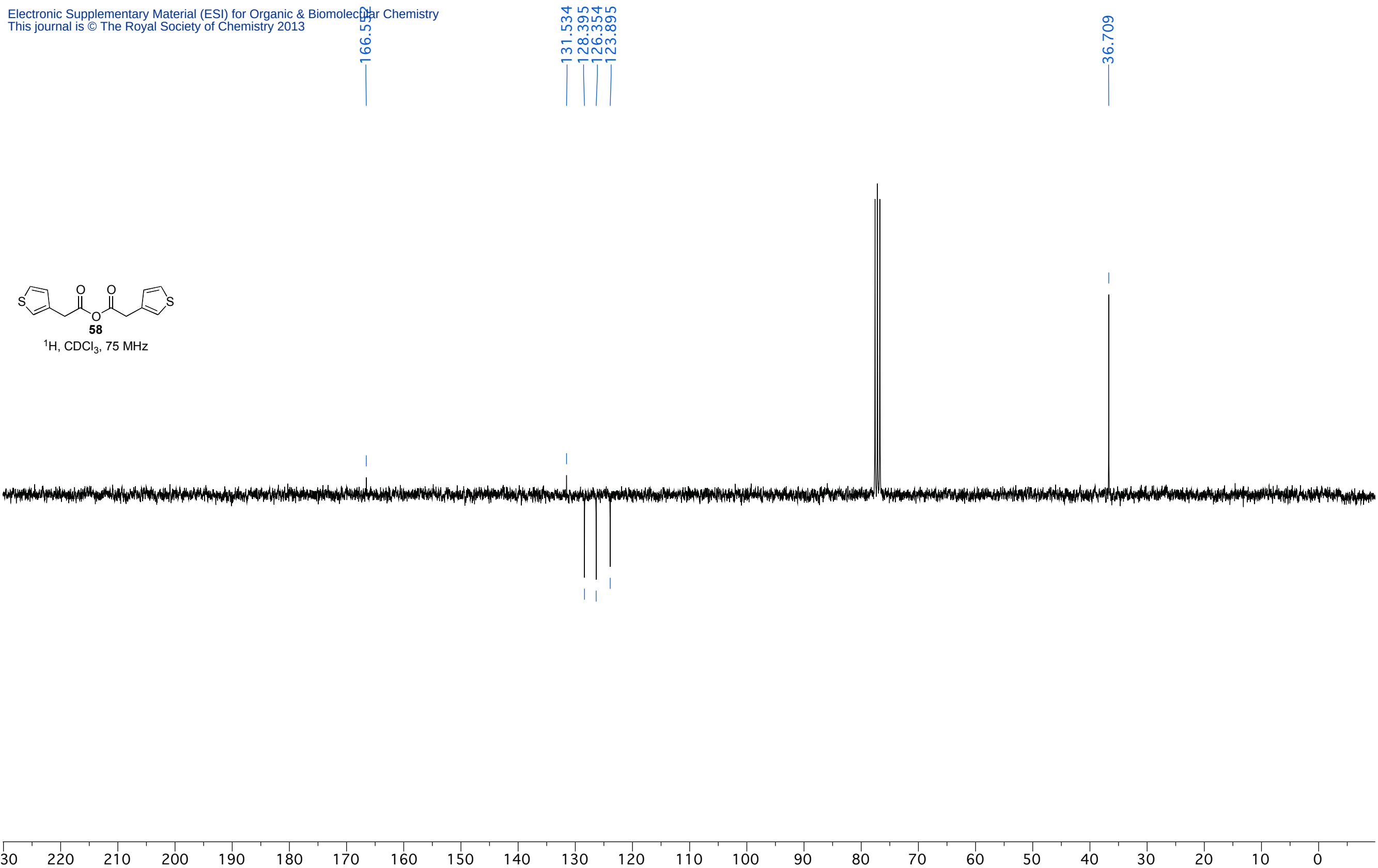
^{13}C , CDCl_3 , 75 MHz





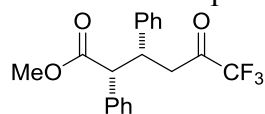
¹H, CDCl₃, 400 MHz



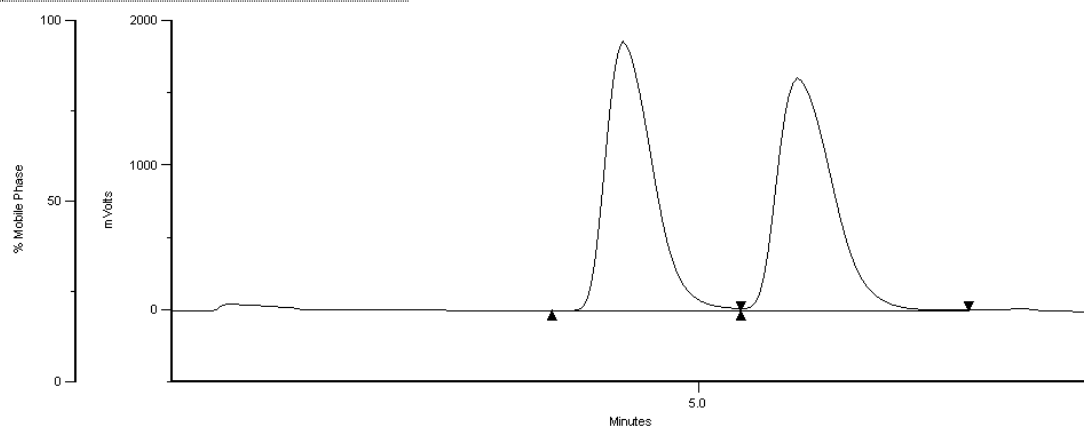


HPLC Data

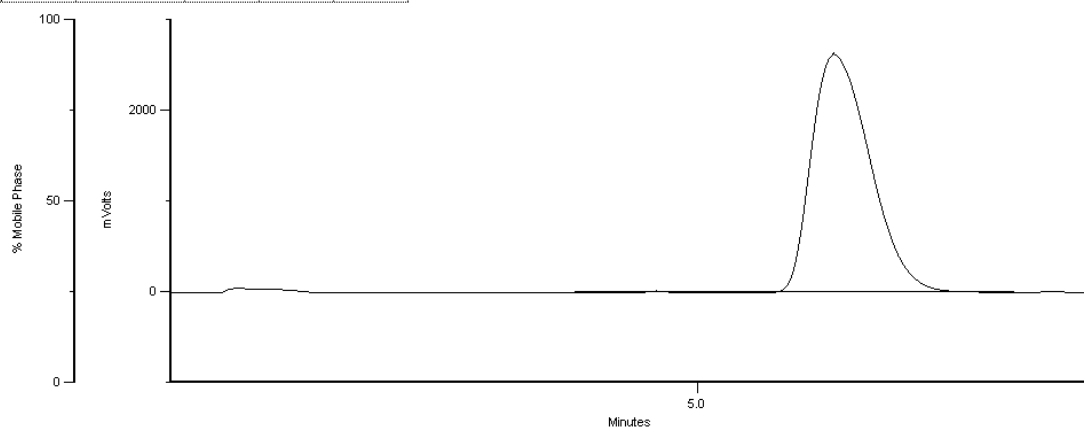
HPLC data compound 4: Chiralpak AD-H 5% IPA:hexane, 1 mL min⁻¹, 211 nm, 20 °C, 98% ee



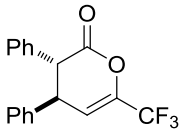
	Inj. Number	Peak Name	R. Time	Area	Area %
1	1.00	*1	4.72	38585176.00	49.56
2	1.00	2	5.38	39277184.00	50.44



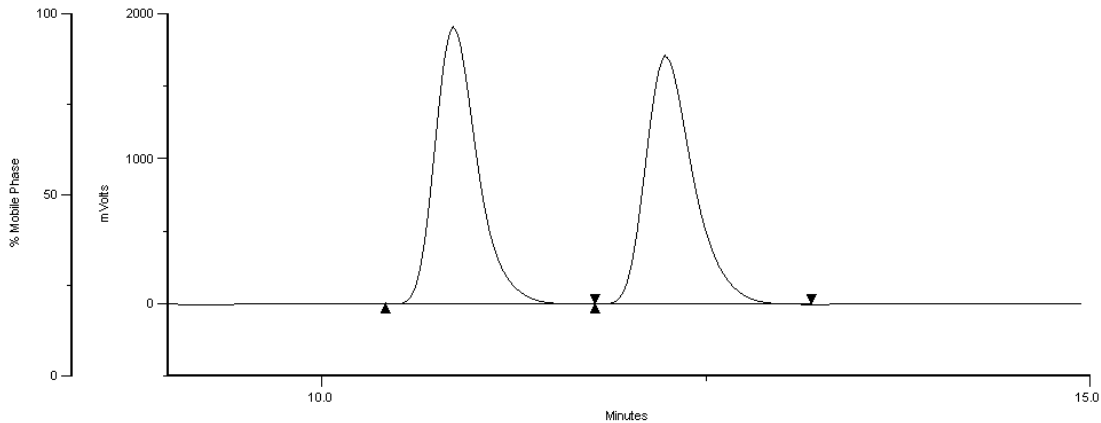
	Inj. Number	Peak Name	R. Time	Area	Area %
1	1.00	*1	4.84	254977.89	0.37
2	1.00	2	5.52	37862024.00	99.63



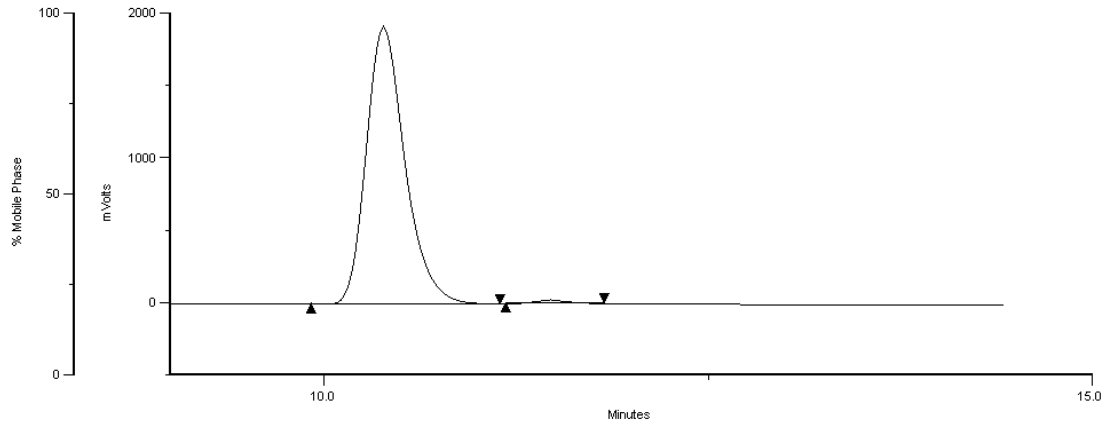
HPLC data compound 5: Chiralpak AD-H 5% IPA:hexane, 1 mL min⁻¹, 211 nm, 20 °C, 99% ee



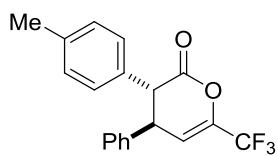
	Inj. Number	Peak Name	R. Time	Area	Area %
1	1.00	*1	10.86	51810188.00	49.99
2	1.00	2	12.24	51832800.00	50.01



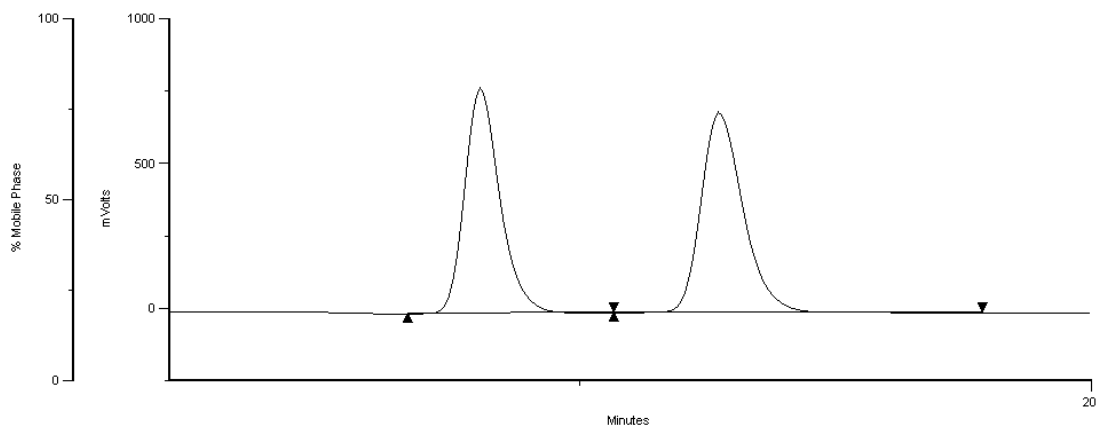
	Inj. Number	Peak Name	R. Time	Area	Area %
1	1.00	*1	10.39	56351876.00	98.80
2	1.00	*2	11.47	681560.88	1.20



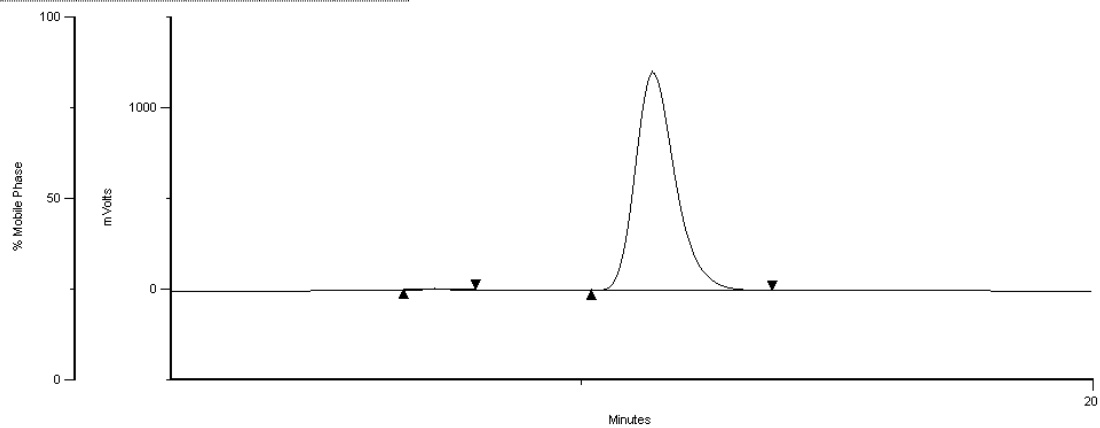
HPLC data compound 6: Chiralpak AD-H 2% IPA:hexane, 1 mL min⁻¹, 211 nm, 20 °C, 99% ee



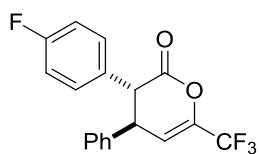
	Inj. Number	Peak Name	R. Time	Area	Area %
1	1.00	*1	14.03	31287448.00	48.24
2	1.00	2	16.37	33573960.00	51.76



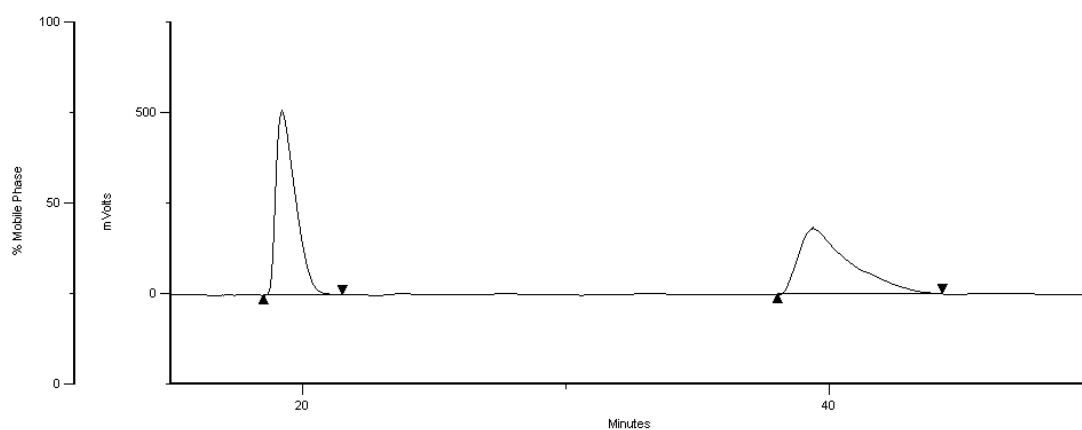
	Inj. Number	Peak Name	R. Time	Area	Area %
1	1.00	*1	13.58	276789.66	0.50
2	1.00	*2	15.70	35177844.00	99.50



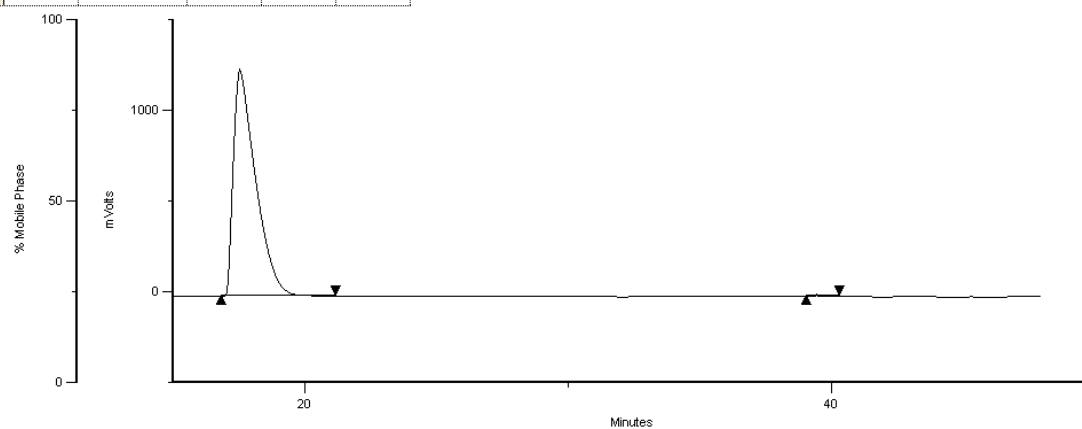
HPLC data compound 7: Chiralcel OD-H 10% IPA:hexane, 1 mL min⁻¹, 211 nm, 20 °C, >99% ee



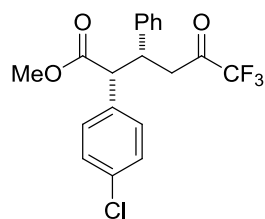
	Inj. Number	Peak Name	R. Time	Area	Area %
1	1.00	*1	19.22	13853832.00	51.16
2	1.00	*2	39.38	11858872.00	48.84



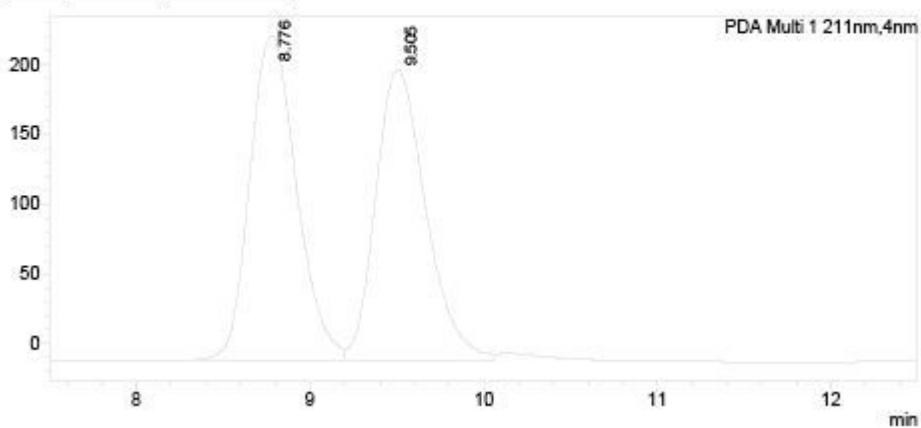
	Inj. Number	Peak Name	R. Time	Area	Area %
1	1.00	*1	17.54	22020256.00	99.85
2	1.00	*2	39.42	182349.50	0.15



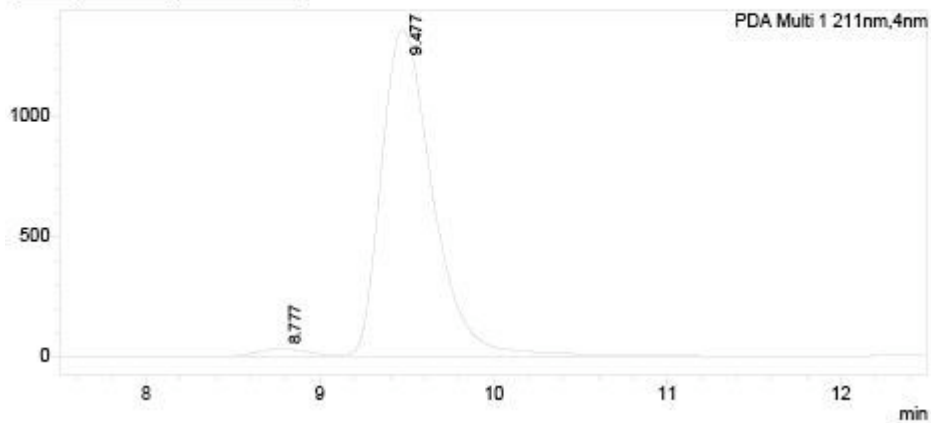
HPLC data compound 8: Chiralpak IA 10% IPA:hexane, 0.5 mL min⁻¹, 211 nm, 30 °C, 95% ee



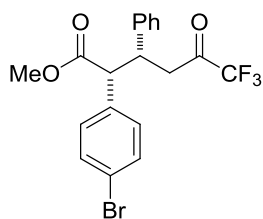
PDA Ch1 211nm		
Peak#	Ret. Time	Area%
1	8.776	51.018
2	9.505	48.982
Total		100.000



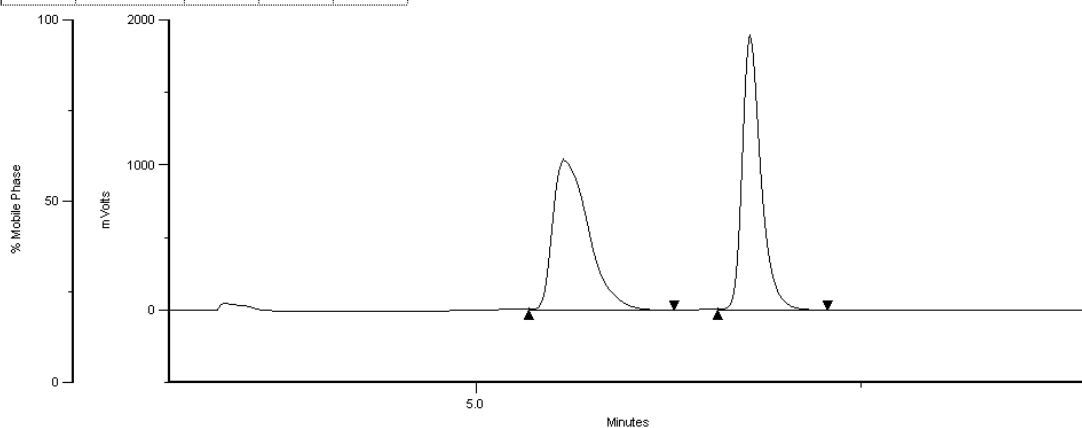
PDA Ch1 211nm		
Peak#	Ret. Time	Area%
1	8.777	2.267
2	9.477	97.733
Total		100.000



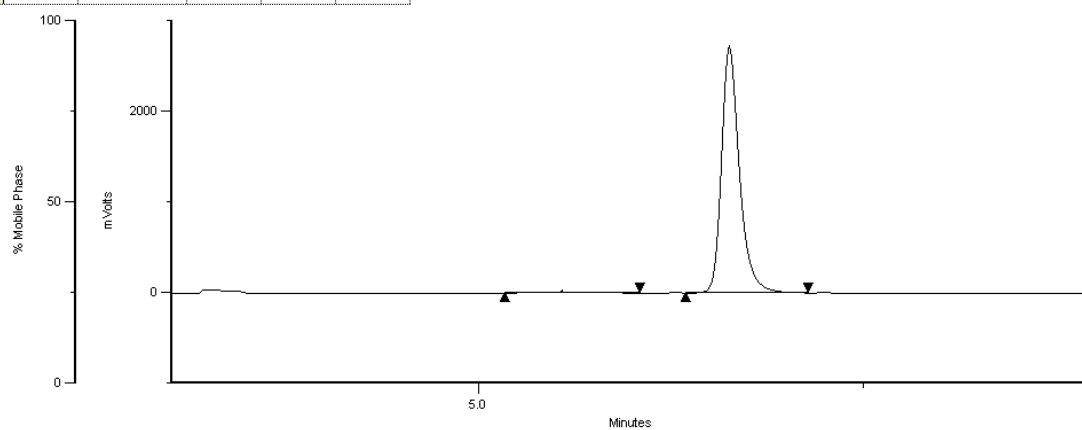
HPLC data compound 9: Chiralpak AD-H 5% IPA:hexane, 1 mL min⁻¹, 211 nm, 20 °C, 96% ee



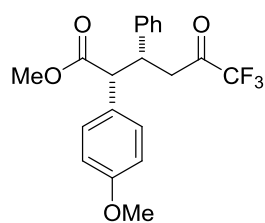
	Inj. Number	Peak Name	R. Time	Area	Area %
1	1.00	*1	5.57	28408224.00	51.06
2	1.00	*2	6.78	27229998.00	48.94



	Inj. Number	Peak Name	R. Time	Area	Area %
1	1.00	*1	5.54	841502.56	2.20
2	1.00	*2	6.63	37337720.00	97.80

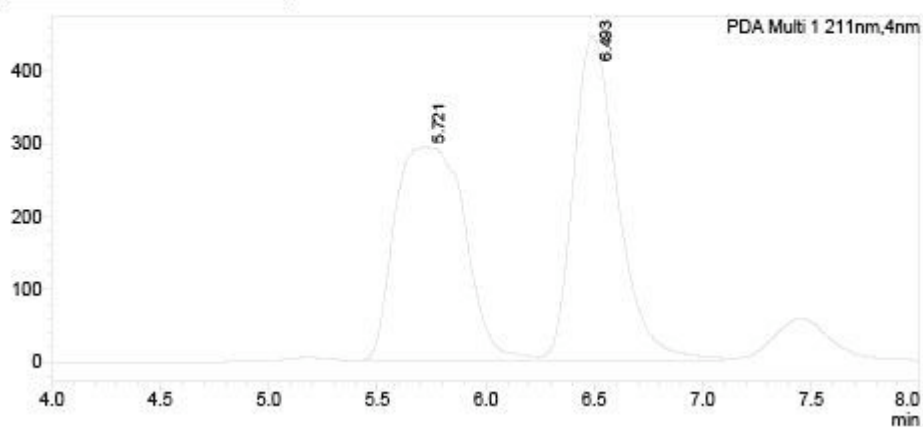


HPLC data compound 10: Chiralpak IA 5% IPA:hexane, 1 mL min⁻¹, 211 nm, 30 °C, 99% ee



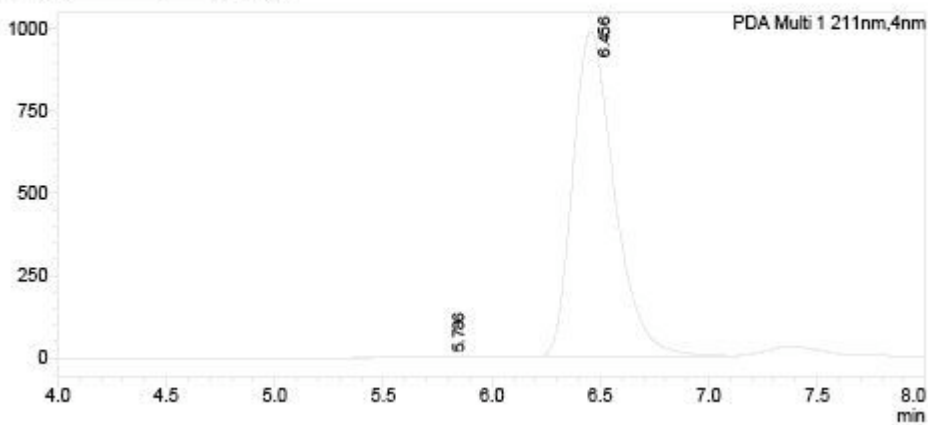
PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	5.721	50.915
2	6.493	49.085
Total		100.000

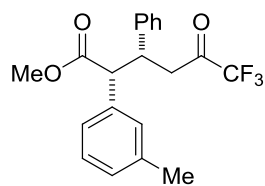


PDA Ch1 211nm

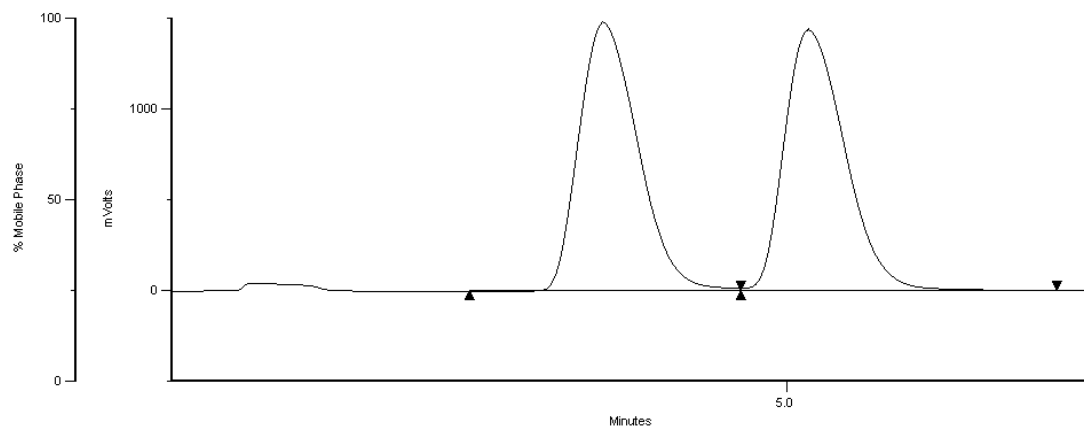
Peak#	Ret. Time	Area%
1	5.786	0.698
2	6.456	99.302
Total		100.000



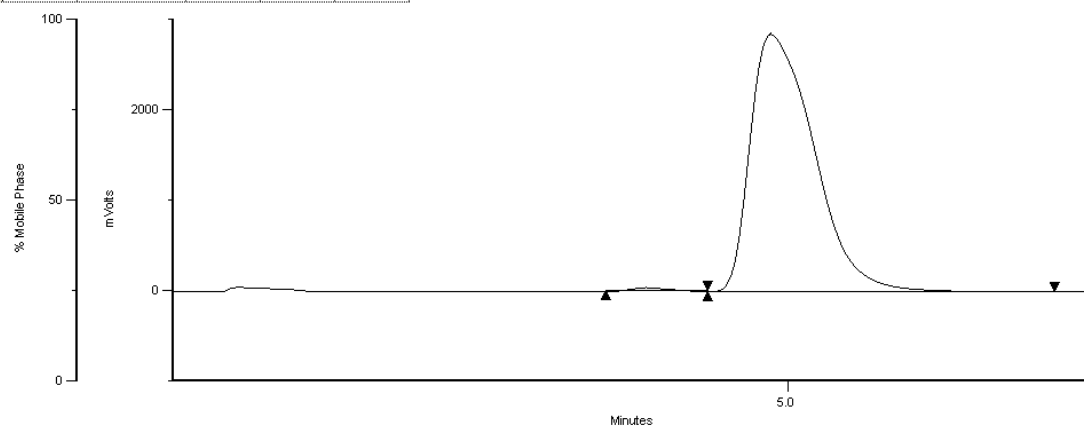
HPLC data compound 11: Chiralpak AD-H 5% IPA:hexane, 1 mL min⁻¹, 211 nm, 20 °C, 98% ee



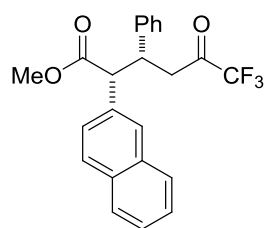
	inj. Number	Peak Name	R. Time	Area	Area %
1	1.00	*1	4.41	32904676.00	49.97
2	1.00	2	5.07	32949682.00	50.03



	inj. Number	Peak Name	R. Time	Area	Area %
1	1.00	*1	4.54	609404.75	0.88
2	1.00	*2	4.95	68998216.00	99.12

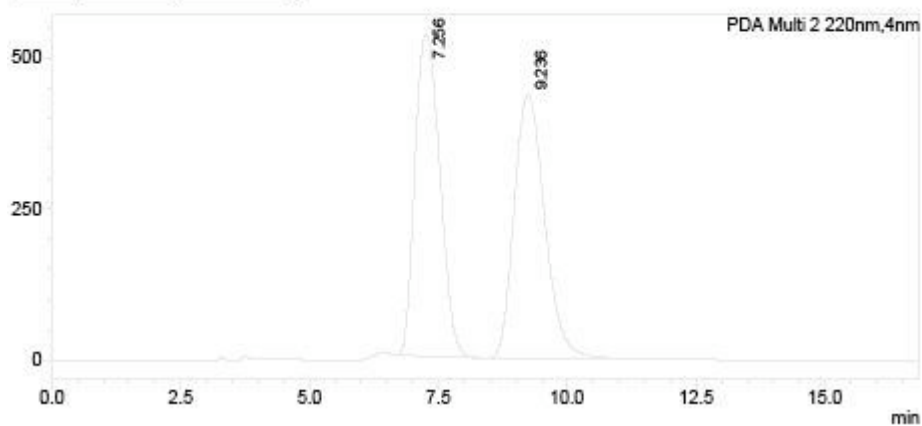


HPLC data compound 12: Chiralpak AS-H 1% IPA:hexane, 1 mL min⁻¹, 220 nm, 30 °C, >99% ee



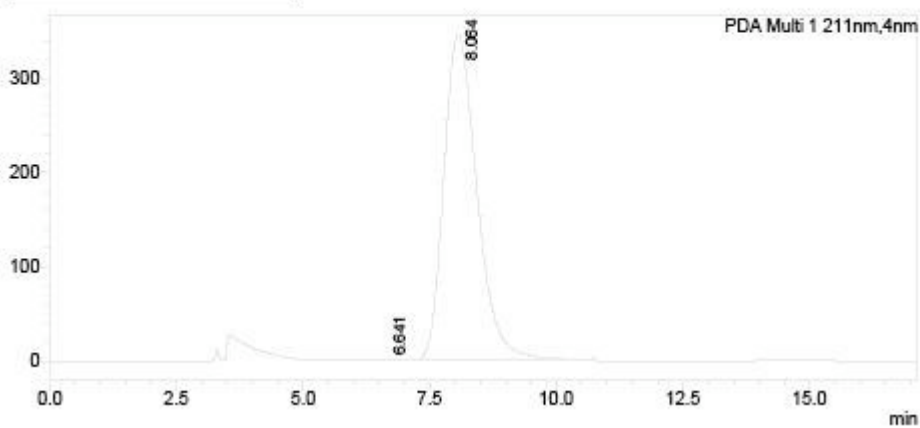
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	7.256	49.987
2	9.236	50.013
Total		100.000

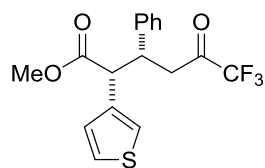


PDA Ch1 211nm

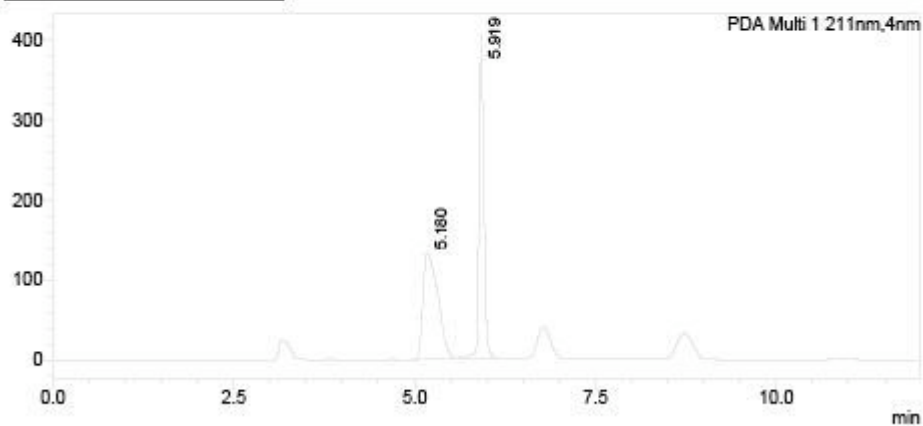
Peak#	Ret. Time	Area%
1	6.641	0.040
2	8.064	99.960
Total		100.000



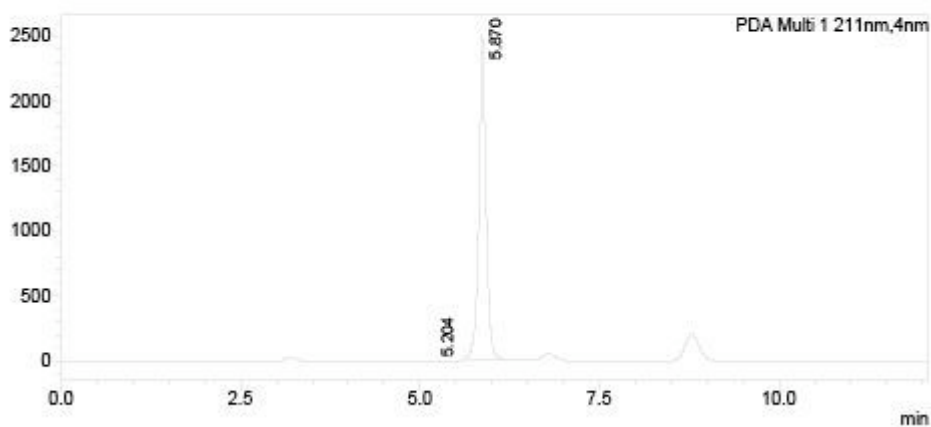
HPLC data compound 13: Chiralpak AD-H 5% IPA:hexane, 1 mL min⁻¹, 211 nm, 30 °C, >99% ee



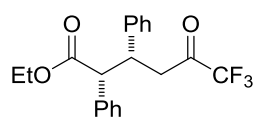
PDA Ch1 211nm		
Peak#	Ret. Time	Area%
1	5.180	50.272
2	5.919	49.728
Total		100.000



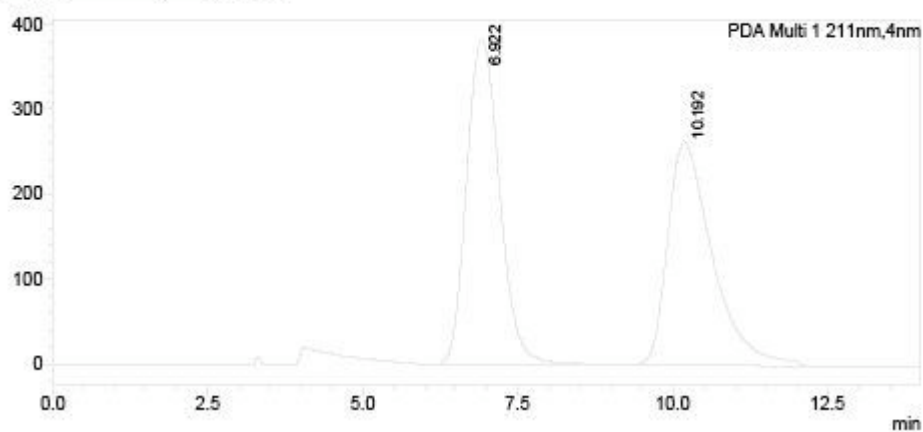
PDA Ch1 211nm		
Peak#	Ret. Time	Area%
1	5.204	0.041
2	5.870	99.959
Total		100.000



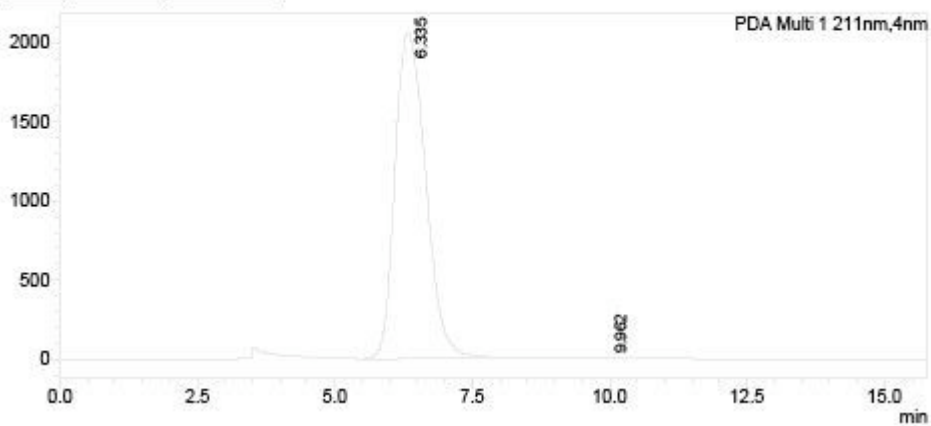
HPLC data compound 14: Chiralpak OJ-H 1% IPA:hexane, 1 mL min⁻¹, 211 nm, 30 °C, 99% ee



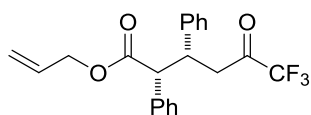
PDA Ch1 211nm		
Peak#	Ret. Time	Area%
1	6.922	51.745
2	10.192	48.255
Total		100.000



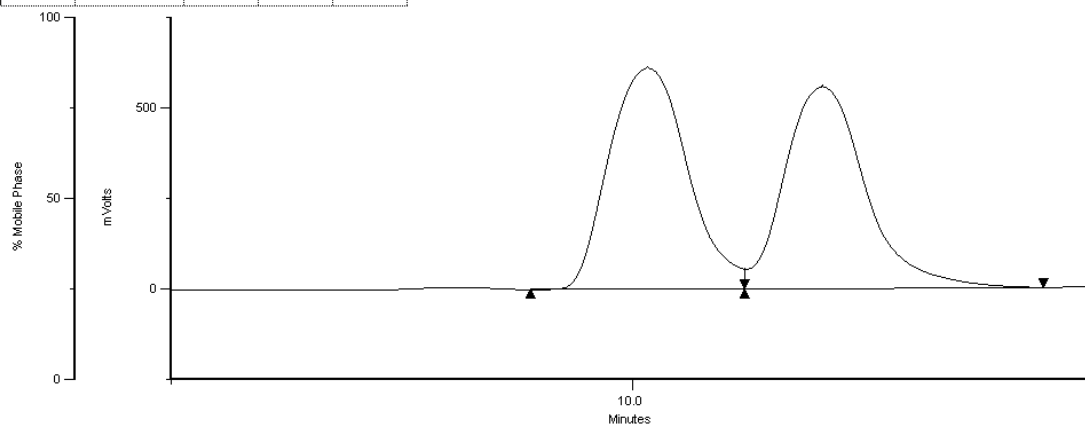
PDA Ch1 211nm		
Peak#	Ret. Time	Area%
1	6.335	99.604
2	9.962	0.396
Total		100.000



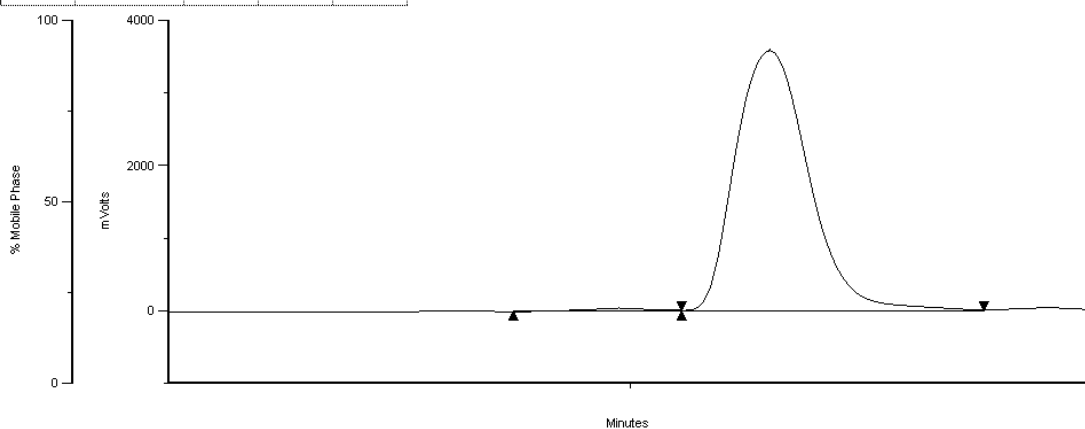
HPLC data compound 15: Chiralpak AD-H 5% IPA:hexane, 0.5 mL min⁻¹, 211 nm, 20 °C, 97% ee



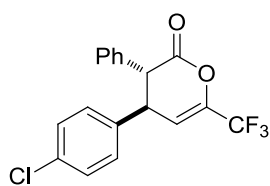
	Inj. Number	Peak Name	R. Time	Area	Area %
1	1.00	*1	10.09	30176282.00	51.24
2	1.00	2	11.03	28711072.00	48.76



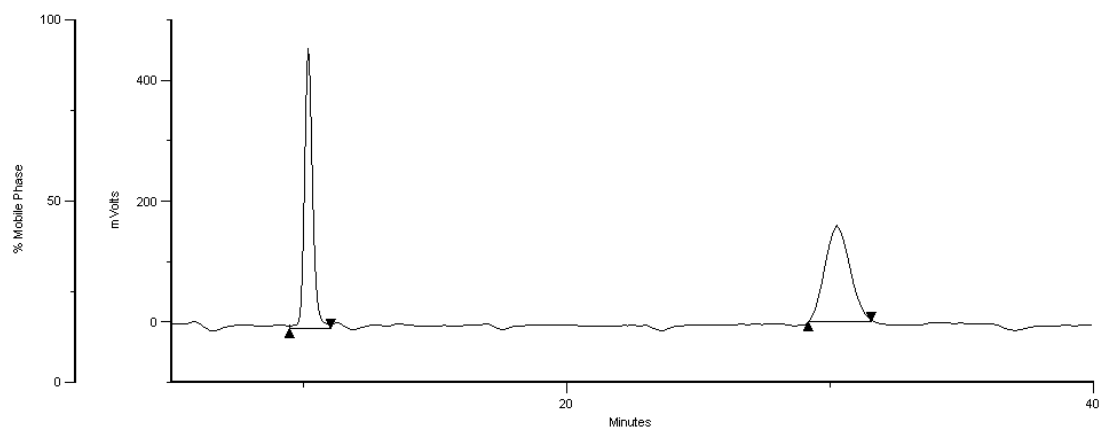
	Inj. Number	Peak Name	R. Time	Area	Area %
1	1.00	*1	9.94	2392376.00	1.41
2	1.00	2	10.76	66785264.00	98.59



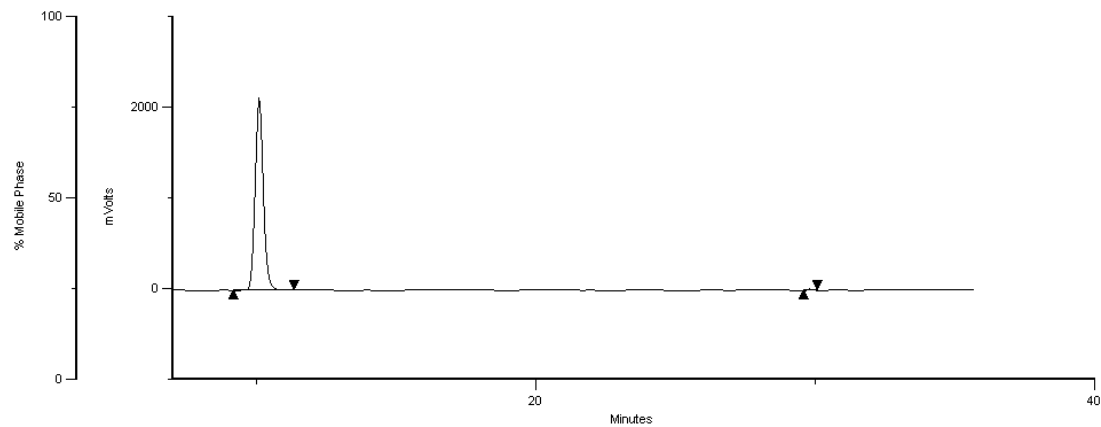
HPLC data compound 16: Chiralpak AD-H 2% IPA:hexane, 2 mL min⁻¹, 211 nm, 20 °C, >99% ee



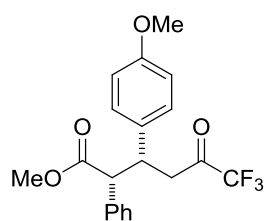
	Inj. Number	Peak Name	R. Time	Area	Area %
1	1.00	*1	10.18	16768070.00	48.26
2	1.00	*2	30.26	17978774.00	51.74



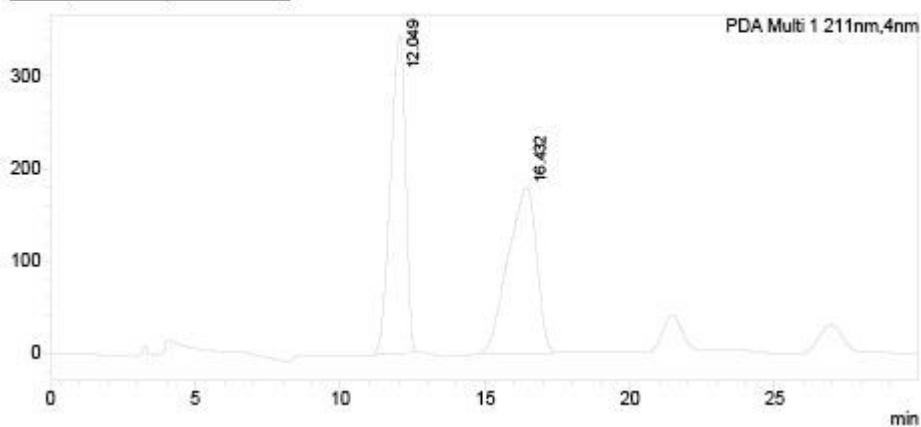
	Inj. Number	Peak Name	R. Time	Area	Area %
1	1.00	*1	10.09	73788792.00	99.88
2	1.00	*2	29.82	92164.31	0.12



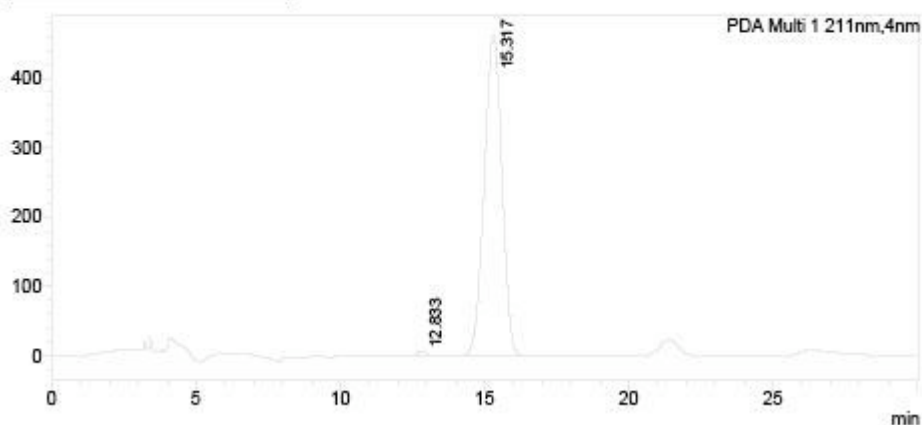
HPLC data compound 17: Chiralpak AD-H 1% IPA:hexane, 1 mL min⁻¹, 211 nm, 30 °C, 98% ee



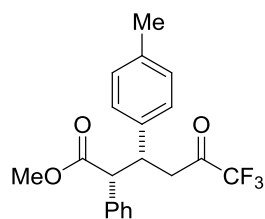
PDA Ch1 211nm		
Peak#	Ret. Time	Area%
1	12.049	49.937
2	16.432	50.063
Total		100.000



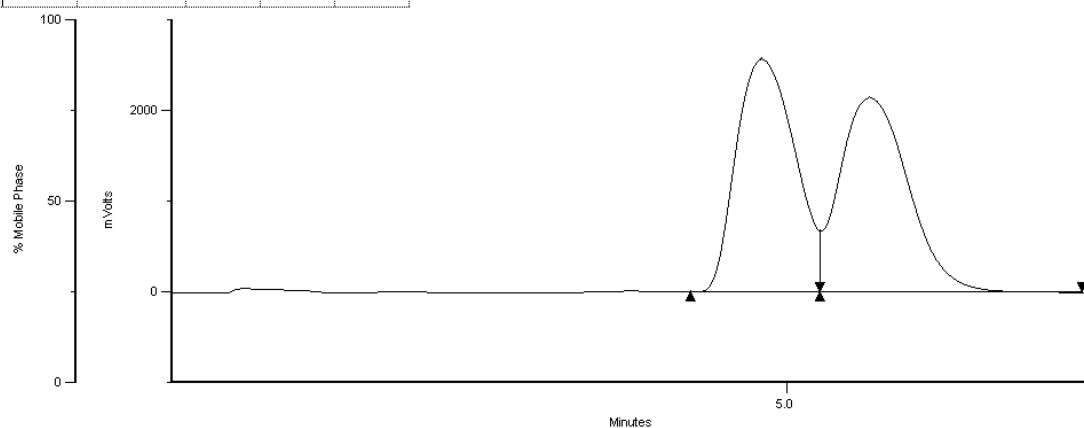
PDA Ch1 211nm		
Peak#	Ret. Time	Area%
1	12.833	0.789
2	15.317	99.211
Total		100.000



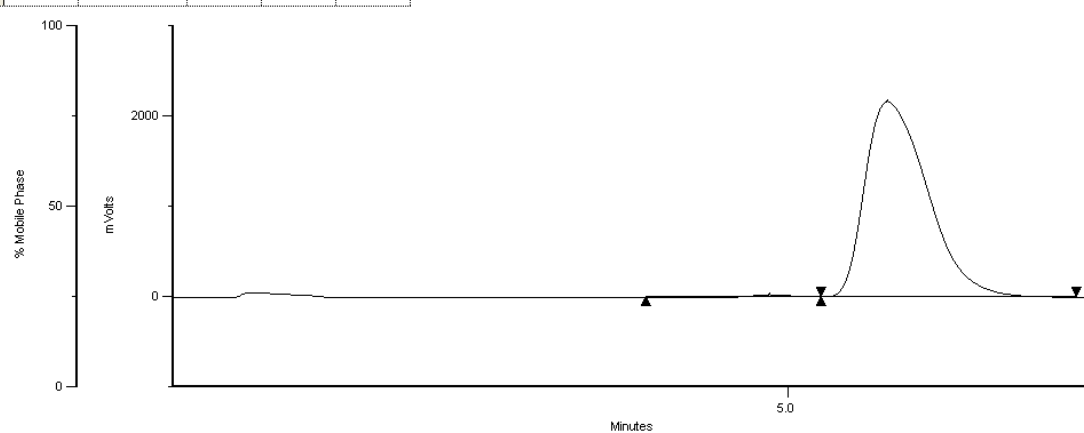
HPLC data compound **18**: Chiralpak AD-H 5% IPA:hexane, 1 mL min⁻¹, 211 nm, 20 °C, 98% ee



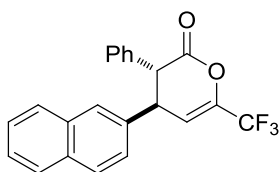
	inj. Number	Peak Name	R. Time	Area	Area %
1	1.00	*1	4.92	57374644.00	50.60
2	1.00	2	5.27	56020836.00	49.40



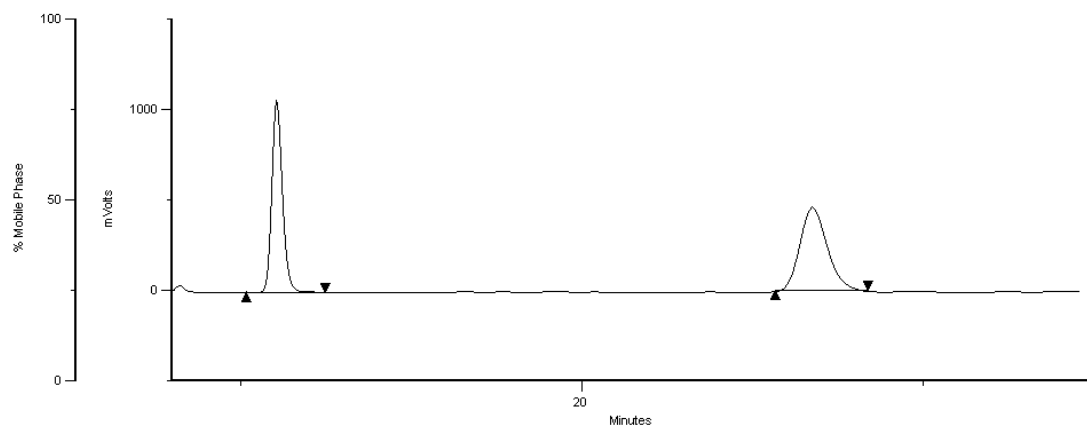
	inj. Number	Peak Name	R. Time	Area	Area %
1	1.00	*1	4.94	495181.47	0.96
2	1.00	2	5.33	51054392.00	99.04



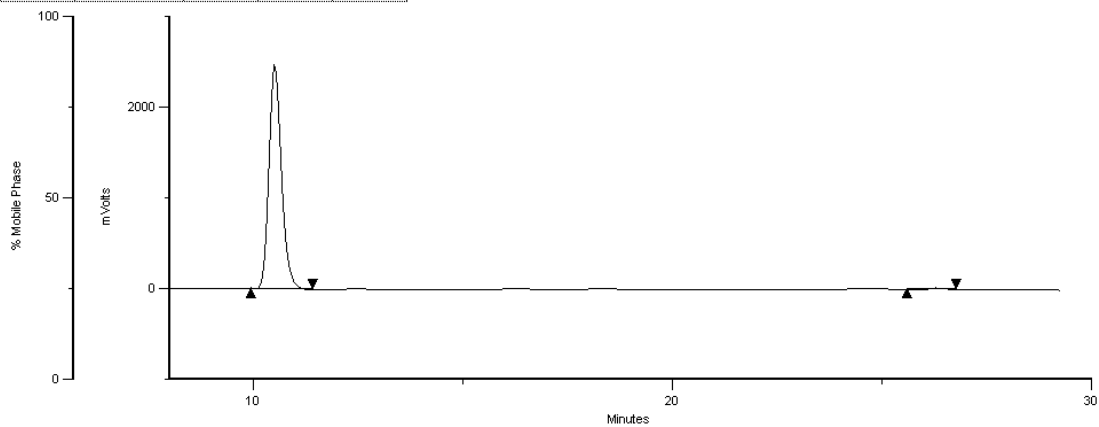
HPLC data compound **19**: Chiralpak AD-H 2% IPA:hexane, 2 mL min⁻¹, 211 nm, 20 °C, 99% ee



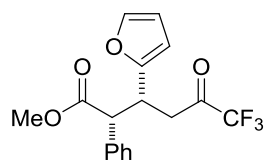
	Inj. Number	Peak Name	R. Time	Area	Area %
1	1.00	*1	11.06	39357916.00	47.11
2	1.00	*2	26.77	14185632.00	52.89



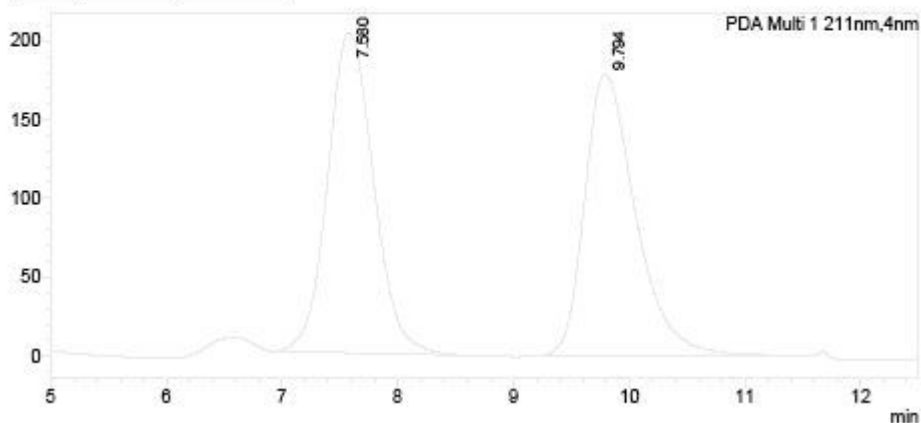
	Inj. Number	Peak Name	R. Time	Area	Area %
1	1.00	*1	10.51	36006936.00	99.61
2	1.00	*2	26.23	336162.00	0.39



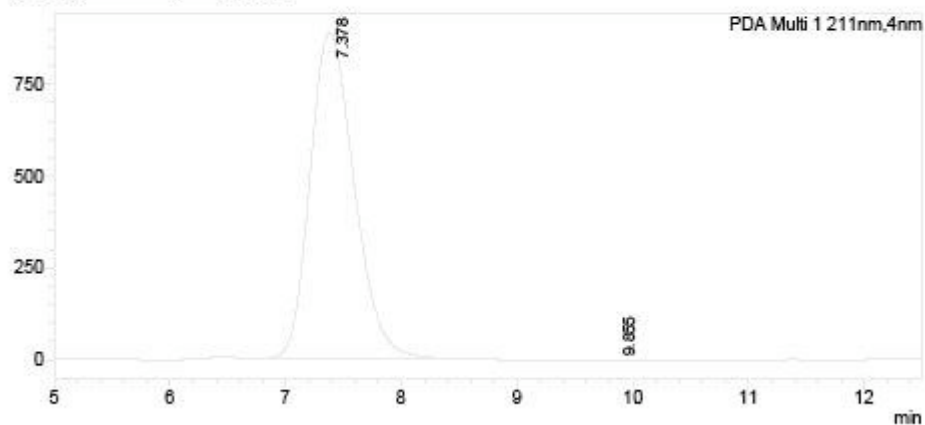
HPLC data compound 20: Chiralpak OJ-H 1% IPA:hexane, 1 mL min⁻¹, 211 nm, 30 °C, >99% ee



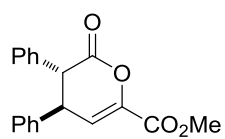
PDA Ch1 211nm		
Peak#	Ret. Time	Area%
1	7.580	50.610
2	9.794	49.390
Total		100.000



PDA Ch1 211nm		
Peak#	Ret. Time	Area%
1	7.378	99.989
2	9.855	0.011
Total		100.000

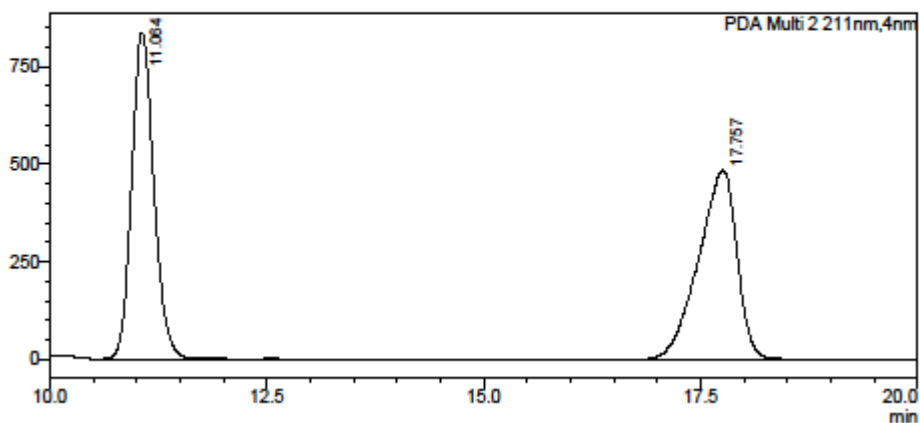


HPLC data compound 21: Chiralpak AD-H 40% IPA:hexane, 1 mL min⁻¹, 211 nm, 30 °C, >99% ee



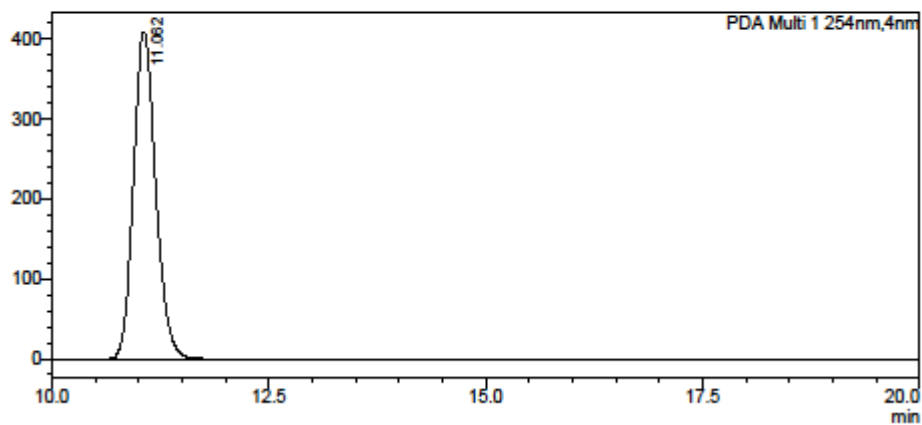
PDA Ch2 211nm

Peak#	Ret. Time	Area%
1	11.064	50.142
2	17.757	49.858
Total		100.000

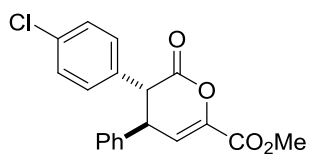


PDA Ch2 211nm

Peak#	Ret. Time	Area%
1	11.062	99.953
2	17.430	0.047
Total		100.000

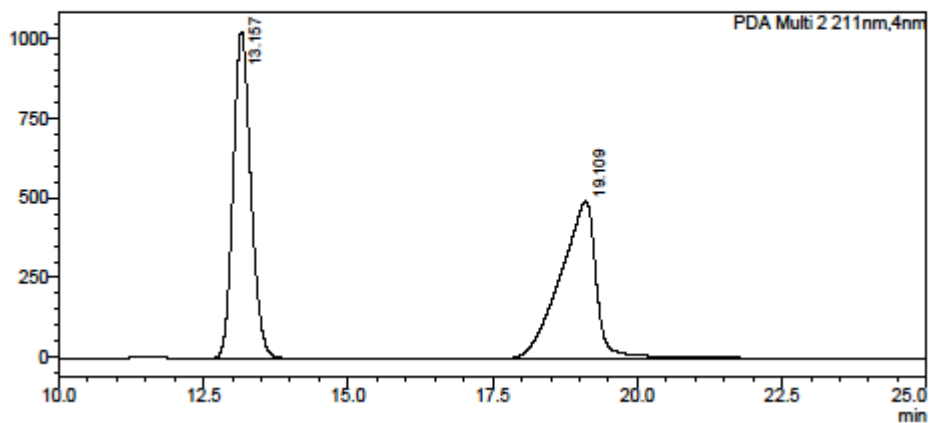


HPLC data compound 22: Chiralpak AD-H 40% IPA:hexane, 1 mL min⁻¹, 211 nm, 30 °C, >99% ee



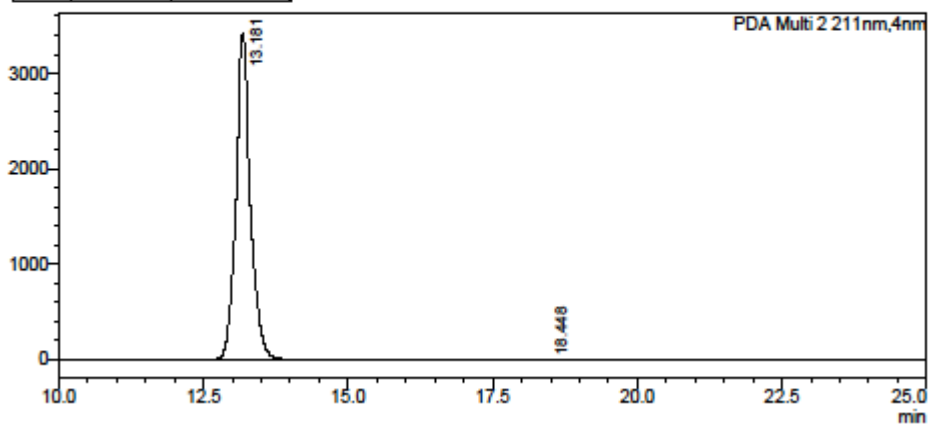
PDA Ch2 211nm

Peak#	Ret. Time	Area%
1	13.157	50.546
2	19.109	49.454
Total		100.000

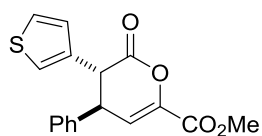


PDA Ch2 211nm

Peak#	Ret. Time	Area%
1	13.181	99.868
2	18.448	0.132
Total		100.000

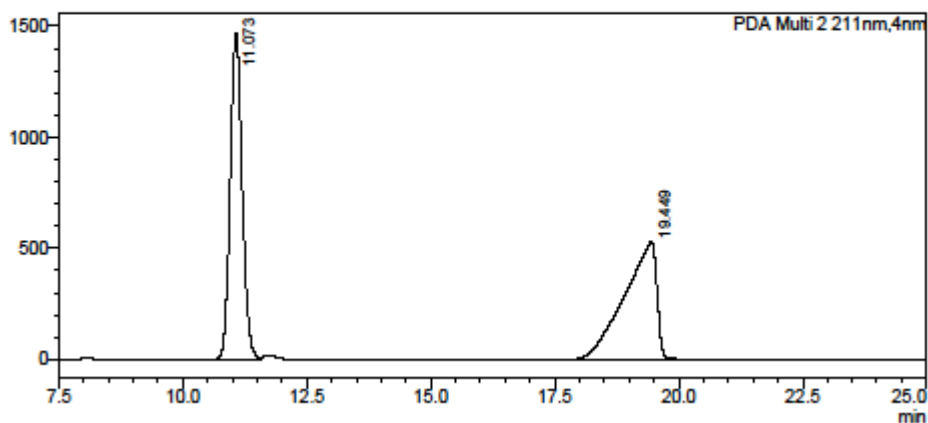


HPLC data compound 23: Chiralpak AD-H 40% IPA:hexane, 1 mL min⁻¹, 211 nm, 30 °C, >99% ee



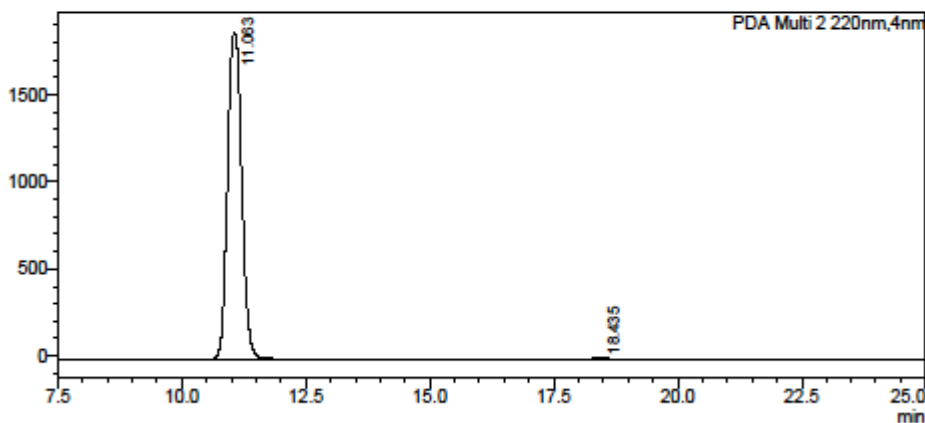
PDA Ch2 211nm

Peak#	Ret. Time	Area%
1	11.073	50.878
2	19.449	49.322
Total		100.000

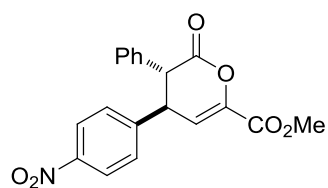


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	11.063	99.875
2	18.435	0.125
Total		100.000

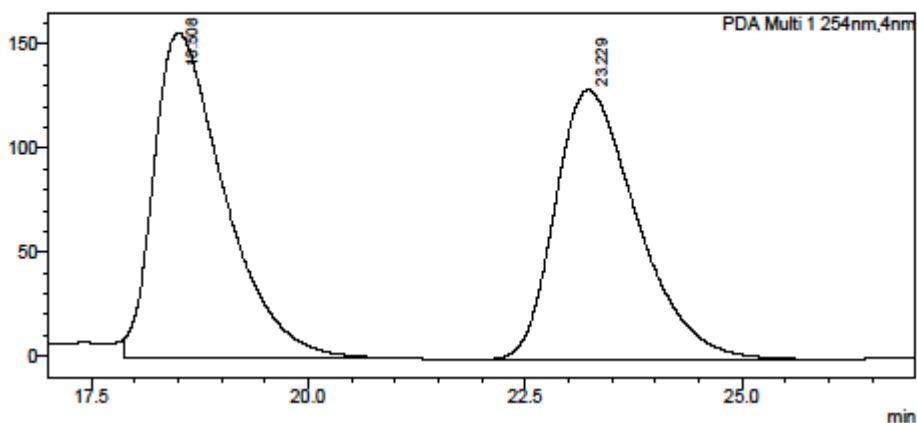


HPLC data compound 24: Chiralcel OD-H 50% IPA:hexane, 1 mL min⁻¹, 254 nm, 30 °C, >99% ee



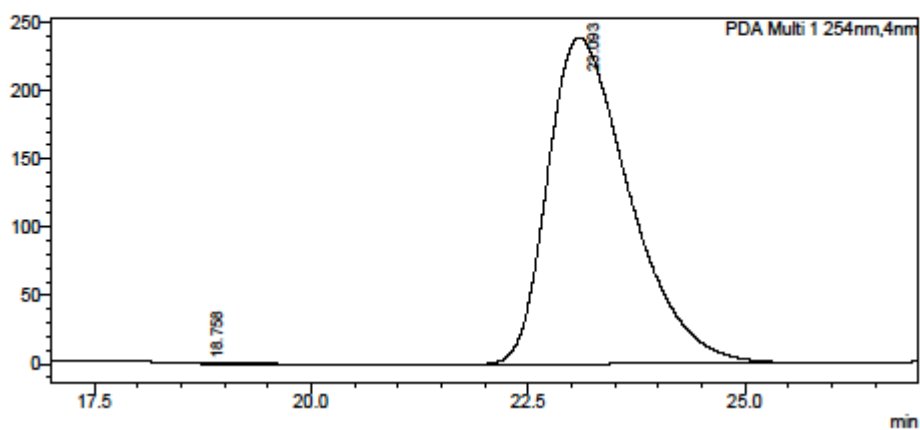
PDA Ch1 254nm

Peak#	Ret. Time	Area%
1	18.508	50.790
2	23.229	49.210
Total		100.000

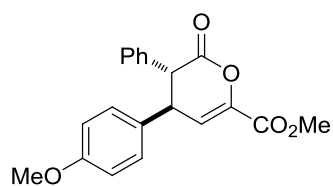


PDA Ch1 254nm

Peak#	Ret. Time	Area%
1	18.758	0.212
2	23.093	99.788
Total		100.000

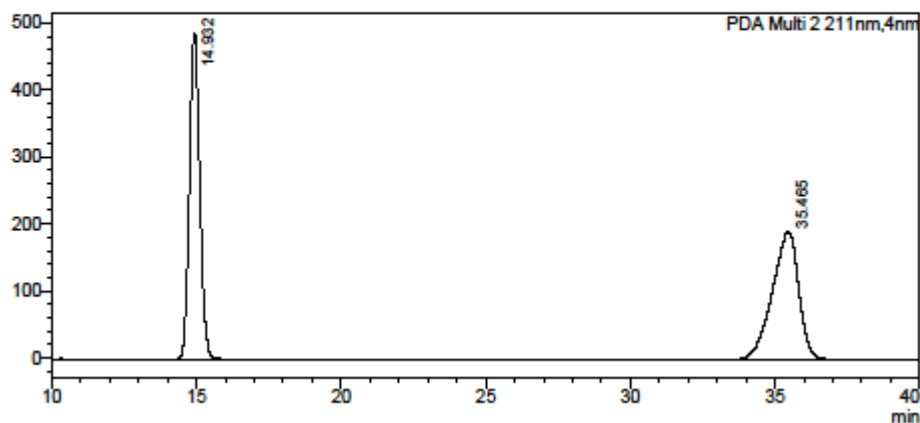


HPLC data compound 25: Chiralpak AD-H 40% IPA:hexane, 1 mL min⁻¹, 211 nm, 30 °C, >99% ee



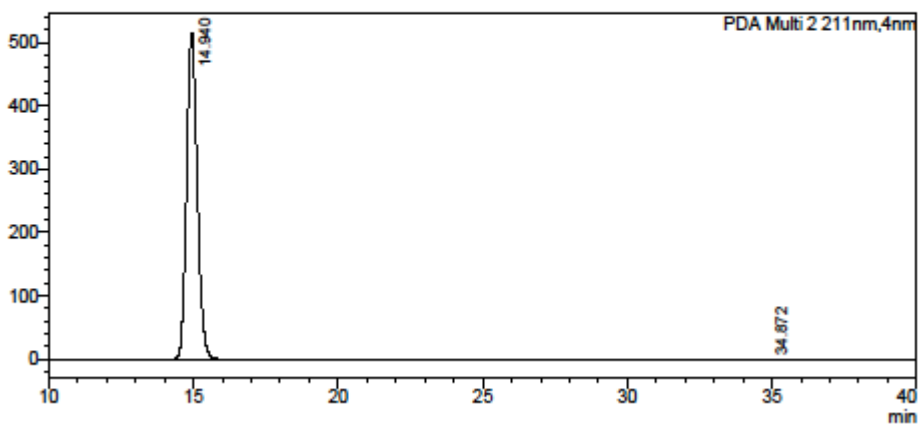
PDA Ch2 211nm

Peak#	Ret. Time	Area%
1	14.932	50.215
2	35.465	49.785
Total		100.000

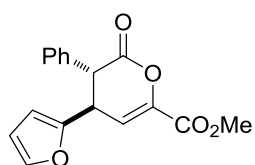


PDA Ch2 211nm

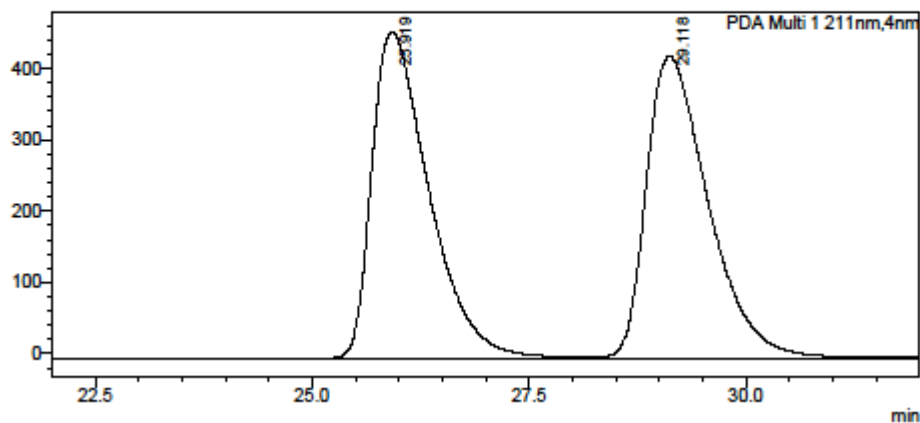
Peak#	Ret. Time	Area%
1	14.940	99.844
2	34.872	0.156
Total		100.000



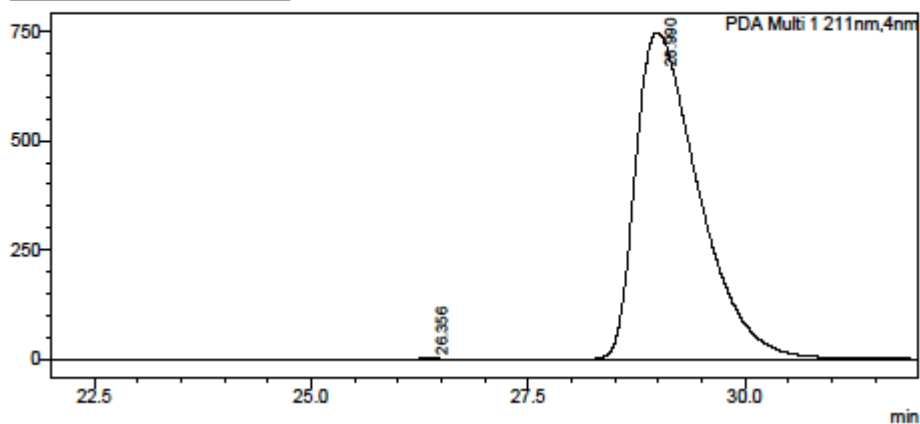
HPLC data compound 26: Chiralcel OJ-H 20% IPA:hexane, 1 mL min⁻¹, 211 nm, 30 °C, >99% ee



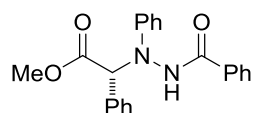
PDA Ch1 211nm		
Peak#	Ret. Time	Area%
1	25.919	50.007
2	29.118	49.993
Total		100.000



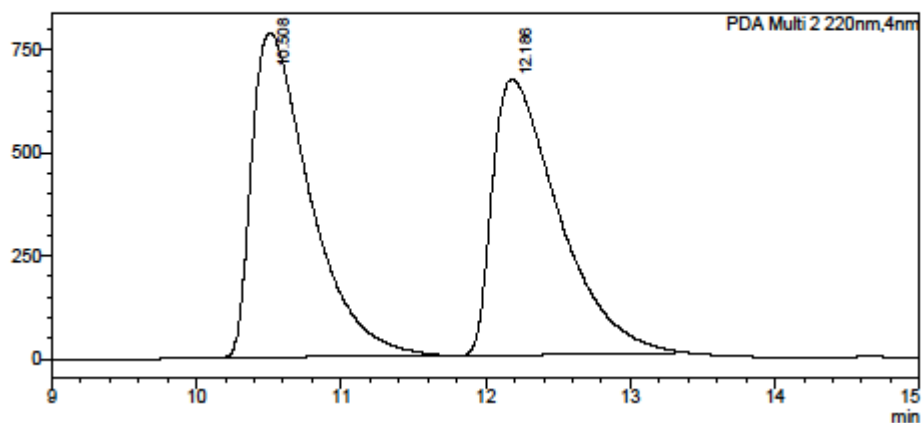
PDA Ch1 211nm		
Peak#	Ret. Time	Area%
1	26.356	0.161
2	28.990	99.839
Total		100.000



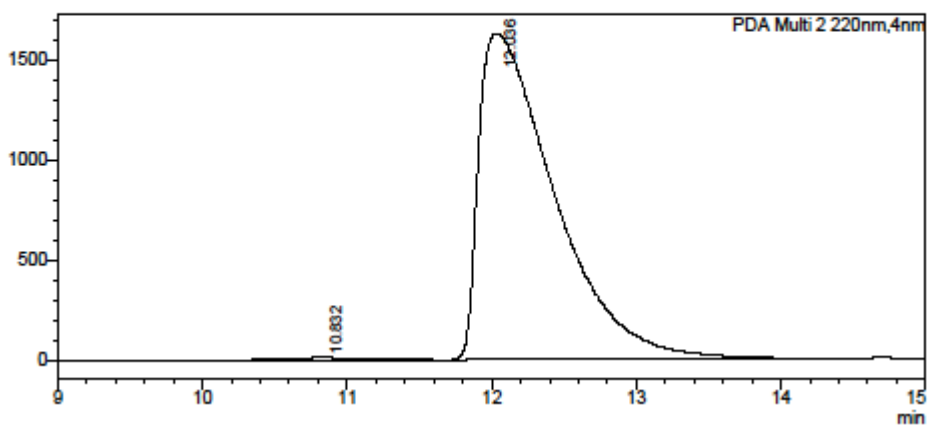
HPLC data compound 27: Chiralpak IB 10% IPA:hexane, 1 mL min⁻¹, 220 nm, 30 °C, 98% ee



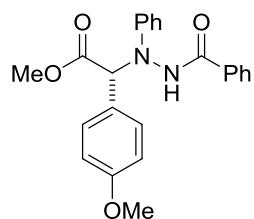
PDA Ch2 220nm			
Peak#	Ret. Time	Area	Area%
1	10.508	22000132	50.657
2	12.186	21429609	49.343
Total		43429741	100.000



PDA Ch2 220nm			
Peak#	Ret. Time	Area	Area%
1	10.832	457898	0.771
2	12.036	58948438	99.229
Total		59406336	100.000

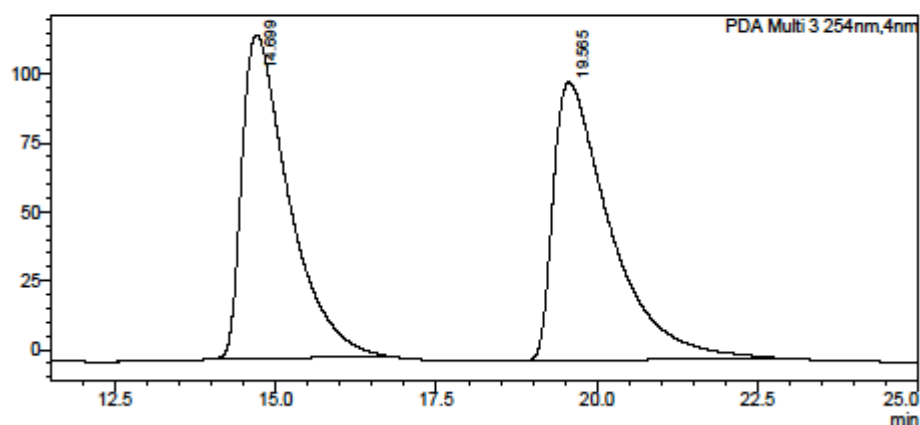


HPLC data compound 28: Chiralpak IB 10% IPA:hexane, 1 mL min⁻¹, 254 nm, 30 °C, 99% ee



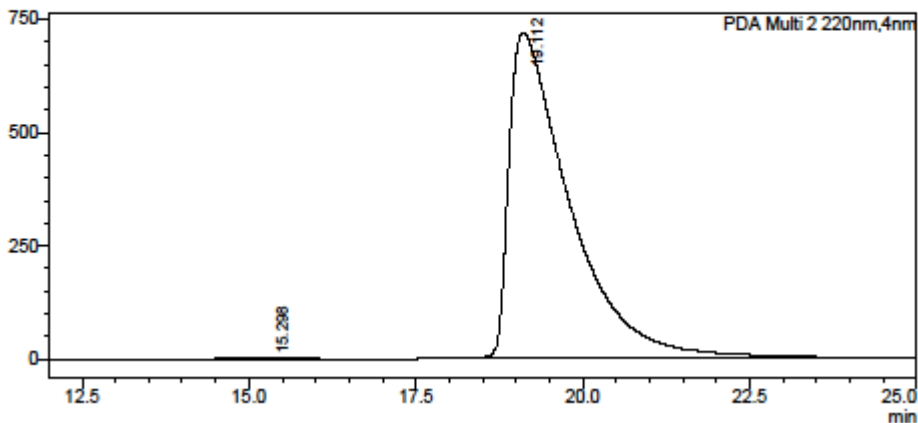
PDA Ch3 254nm

Peak#	Ret. Time	Area	Area%
1	14.899	5959544	48.792
2	19.565	6254724	51.208
Total		12214268	100.000

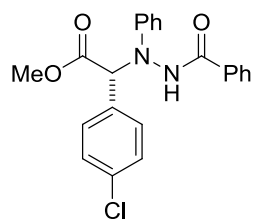


PDA Ch2 220nm

Peak#	Ret. Time	Area	Area%
1	15.298	278908	0.619
2	19.112	44796309	99.381
Total		45075217	100.000

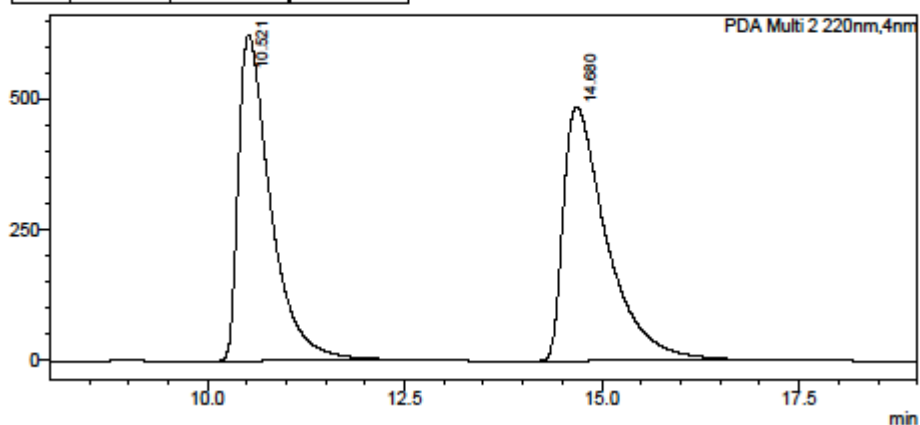


HPLC data compound 29: Chiralpak IB 10% IPA:hexane, 1 mL min⁻¹, 220 nm, 30 °C, 97% ee



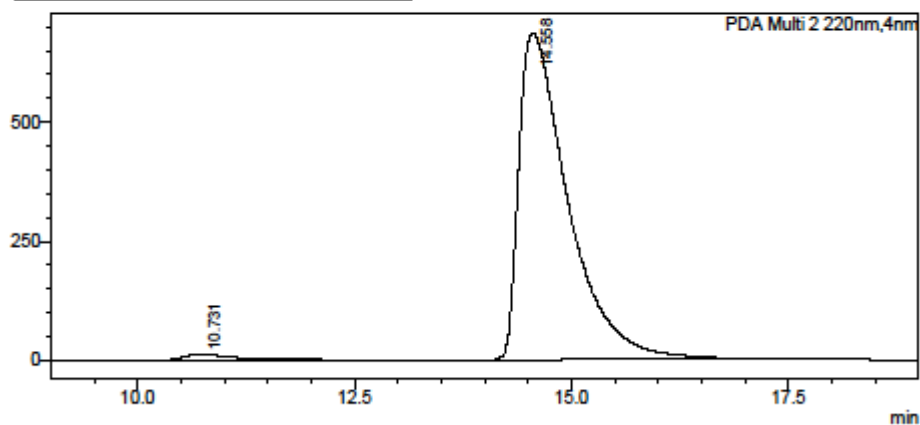
PDA Ch2 220nm

Peak#	Ret. Time	Area	Area%
1	10.521	17817237	48.757
2	14.680	18725369	51.243
Total		36542607	100.000

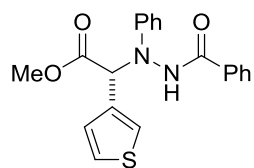


PDA Ch2 220nm

Peak#	Ret. Time	Area	Area%
1	10.731	474816	1.707
2	14.558	27337360	98.293
Total		27812175	100.000

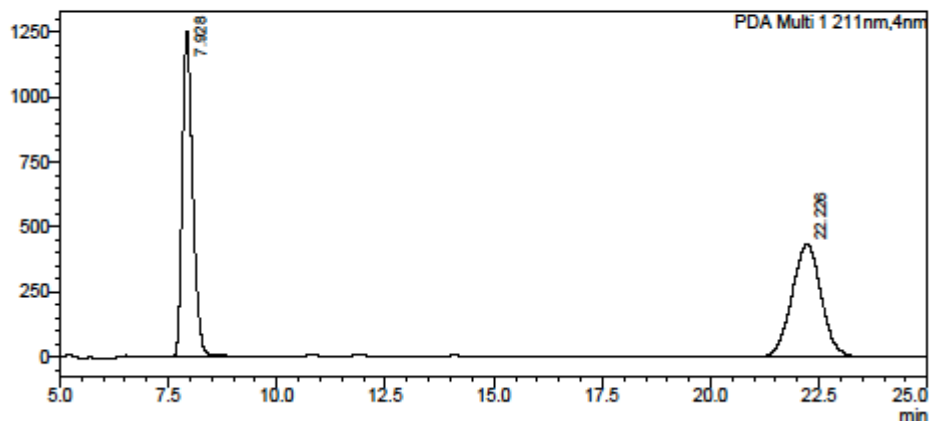


HPLC data compound 30: Chiralpak IA 40% IPA:hexane, 1 mL min⁻¹, 211 nm, 30 °C, 95% ee



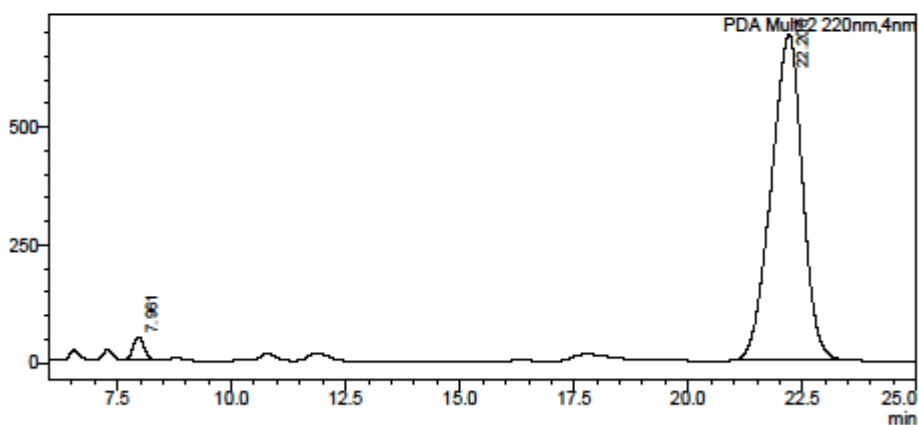
PDA Ch1 211nm

Peak#	Ret. Time	Area	Area%
1	7.928	21001870	50.904
2	22.226	20255659	49.096
Total		41257529	100.000

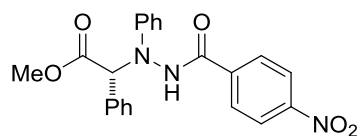


PDA Ch2 220nm

Peak#	Ret. Time	Area	Area%
1	7.961	819274	2.442
2	22.208	32724054	97.558
Total		33543328	100.000

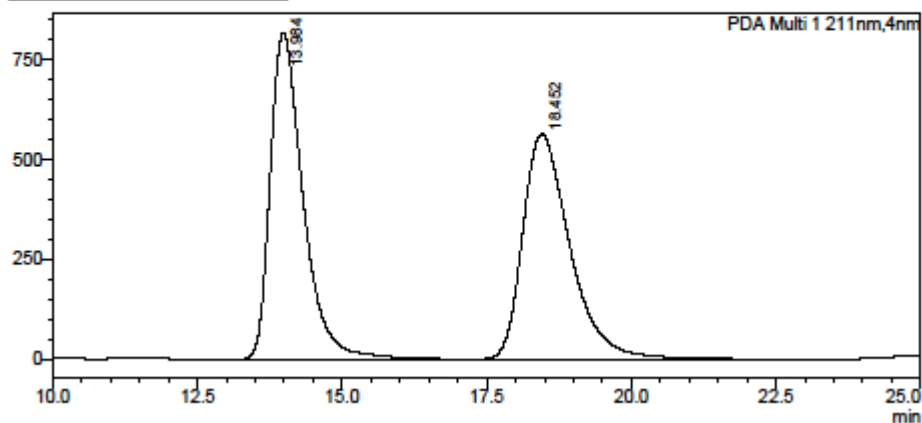


HPLC data compound 31: Chiralcel OJ-H 30% IPA:hexane, 1 mL min⁻¹, 211 nm, 40 °C, 99% ee



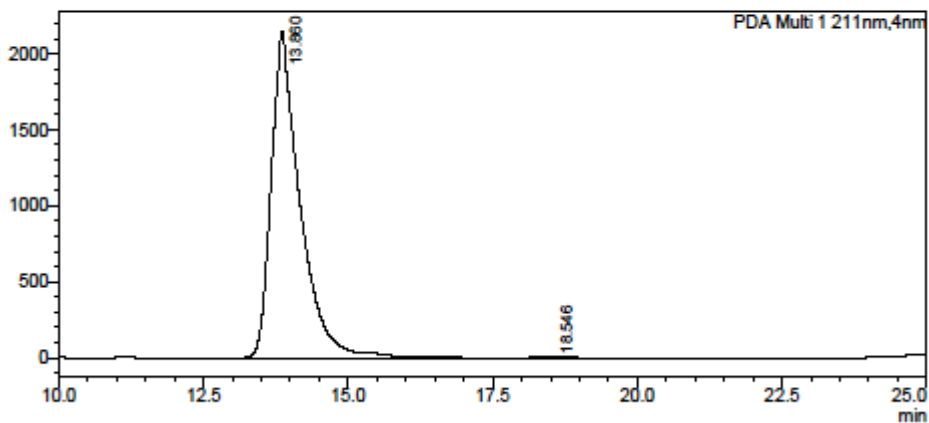
PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	13.984	50.179
2	18.452	49.821
Total		100.000

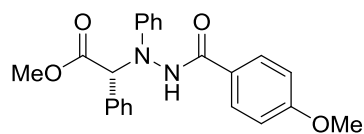


PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	13.860	99.719
2	18.546	0.281
Total		100.000

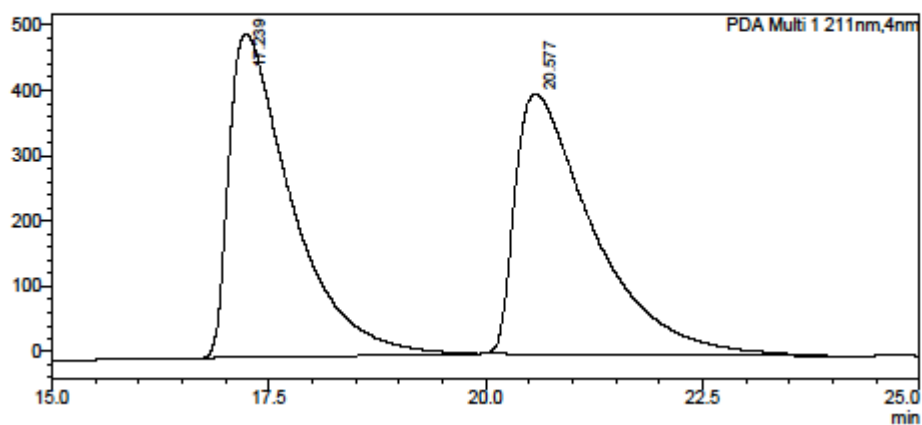


HPLC data compound 32: Chiralpak IB 10% IPA:hexane, 1 mL min⁻¹, 211 nm, 30 °C, 99% ee



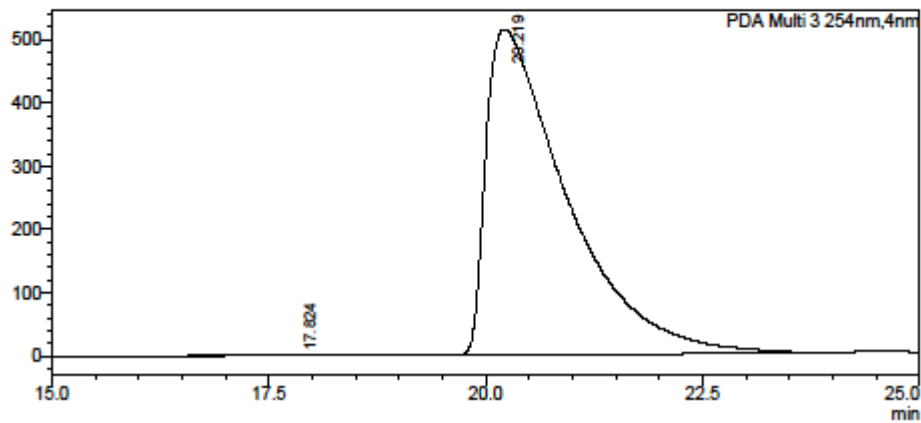
PDA Ch1 211nm

Peak#	Ret. Time	Area	Area%
1	17.239	24876539	49.874
2	20.577	25002128	50.126
Total		49878665	100.000

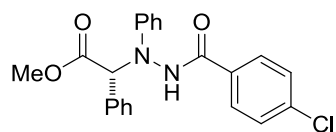


PDA Ch3 254nm

Peak#	Ret. Time	Area	Area%
1	17.824	239117	0.714
2	20.219	33244748	99.286
Total		33483862	100.000

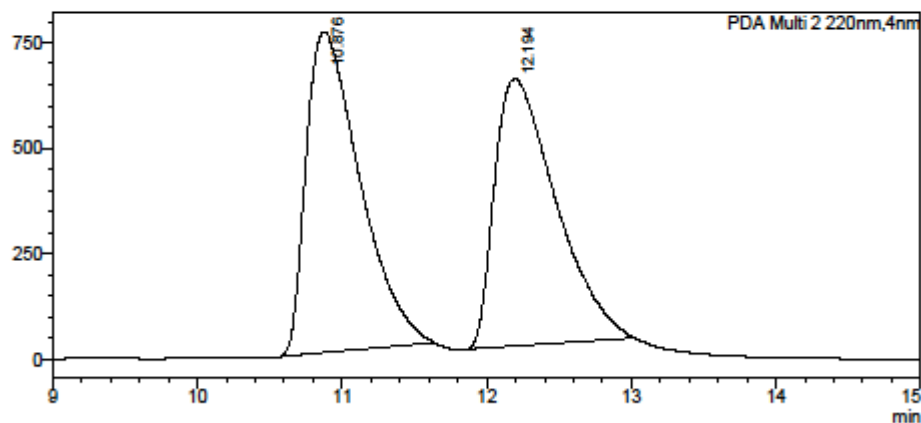


HPLC data compound 33: Chiralpak IB 10% IPA:hexane, 1 mL min⁻¹, 220 nm, 30 °C, 98% ee



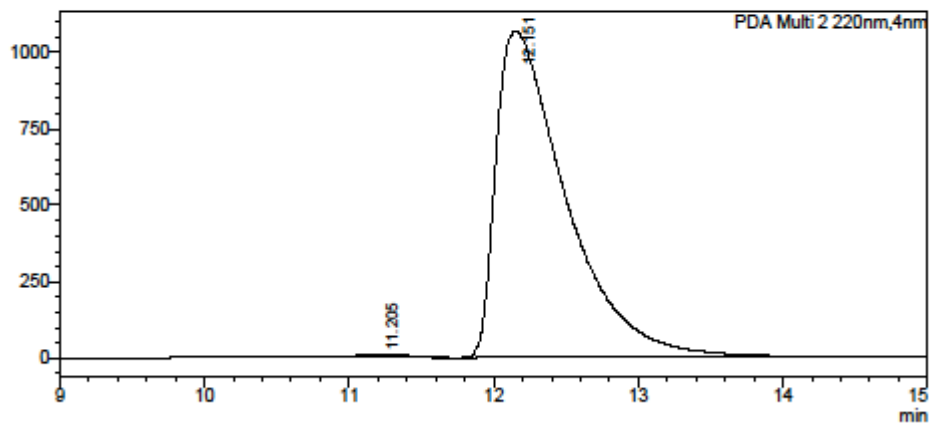
PDA Ch2 220nm

Peak#	Ret. Time	Area	Area%
1	10.876	19594699	51.219
2	12.194	18662011	48.781
Total		38256711	100.000

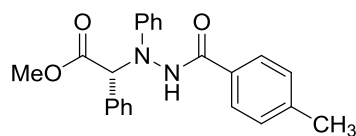


PDA Ch2 220nm

Peak#	Ret. Time	Area	Area%
1	11.205	277050	0.771
2	12.151	35640717	99.229
Total		35917767	100.000

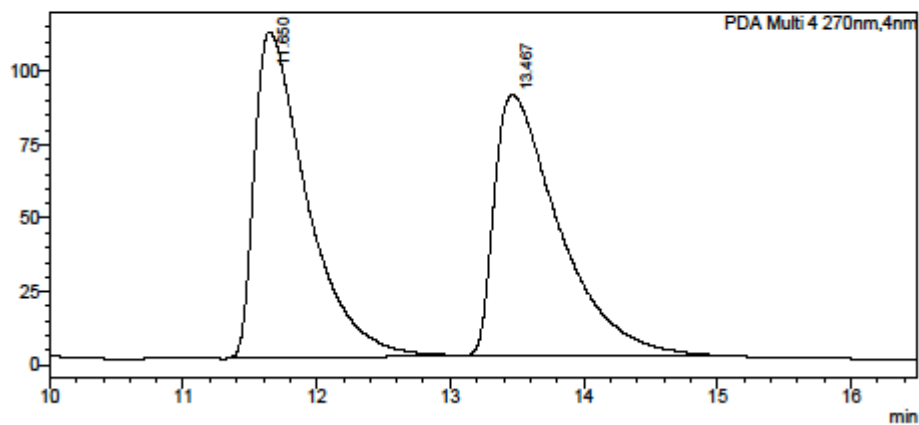


HPLC data compound 34: Chiralpak IB 10% IPA:hexane, 1 mL min⁻¹, 220 nm, 30 °C, 99% ee



PDA Ch4 270nm

Peak#	Ret. Time	Area	Area%
1	11.650	3038190	50.410
2	13.467	2988798	49.590
Total		6026987	100.000



PDA Ch2 220nm

Peak#	Ret. Time	Area	Area%
1	11.103	166456	0.451
2	12.475	36764955	99.549
Total		36931412	100.000

