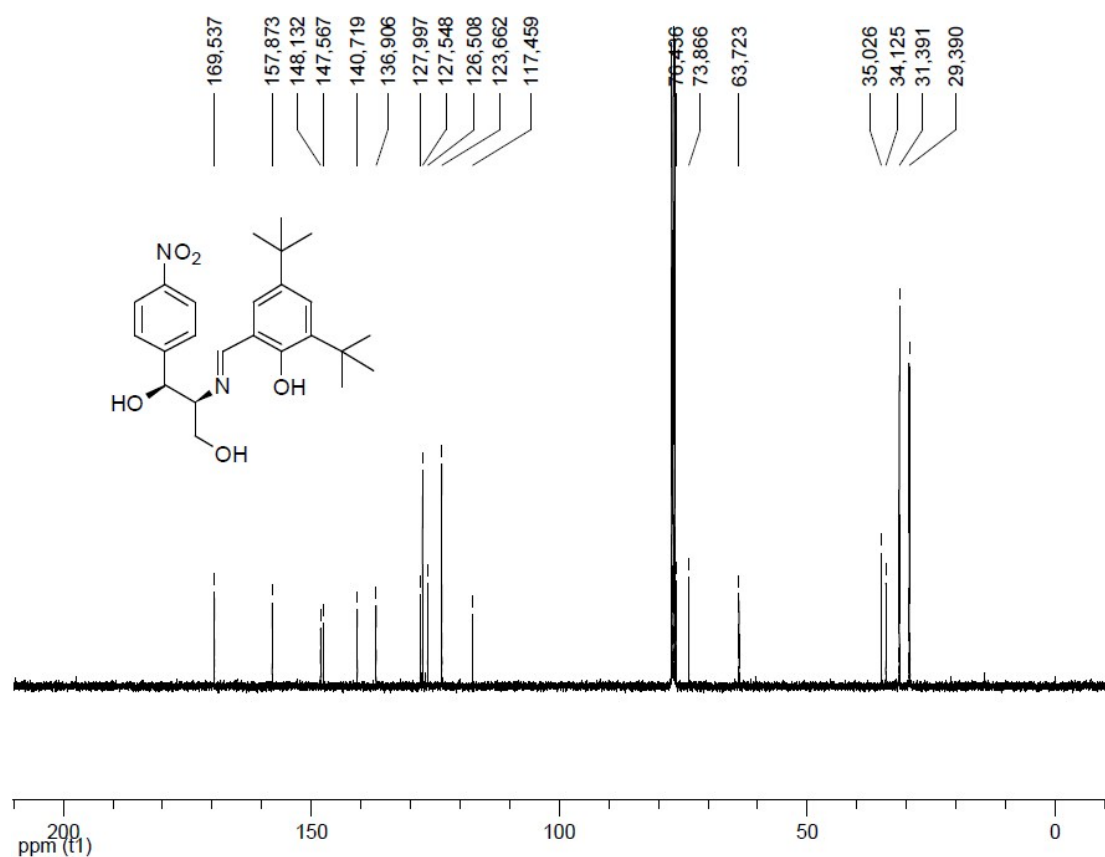
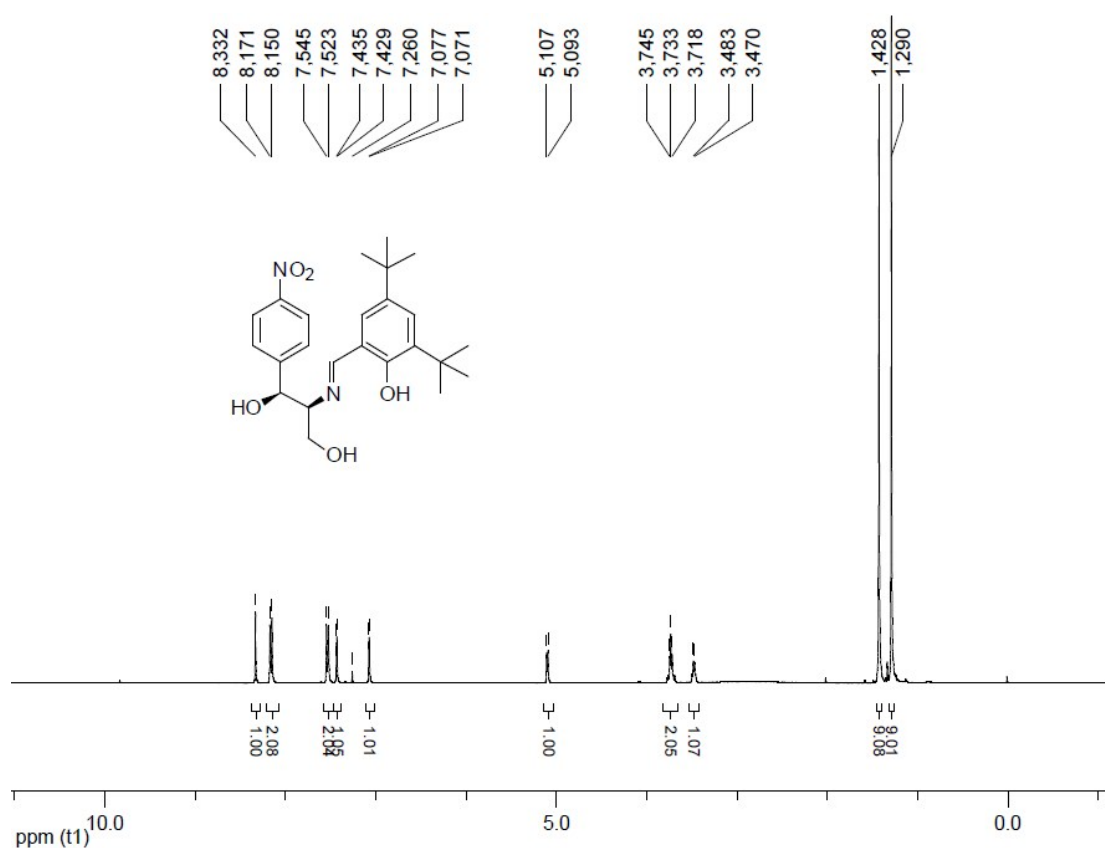
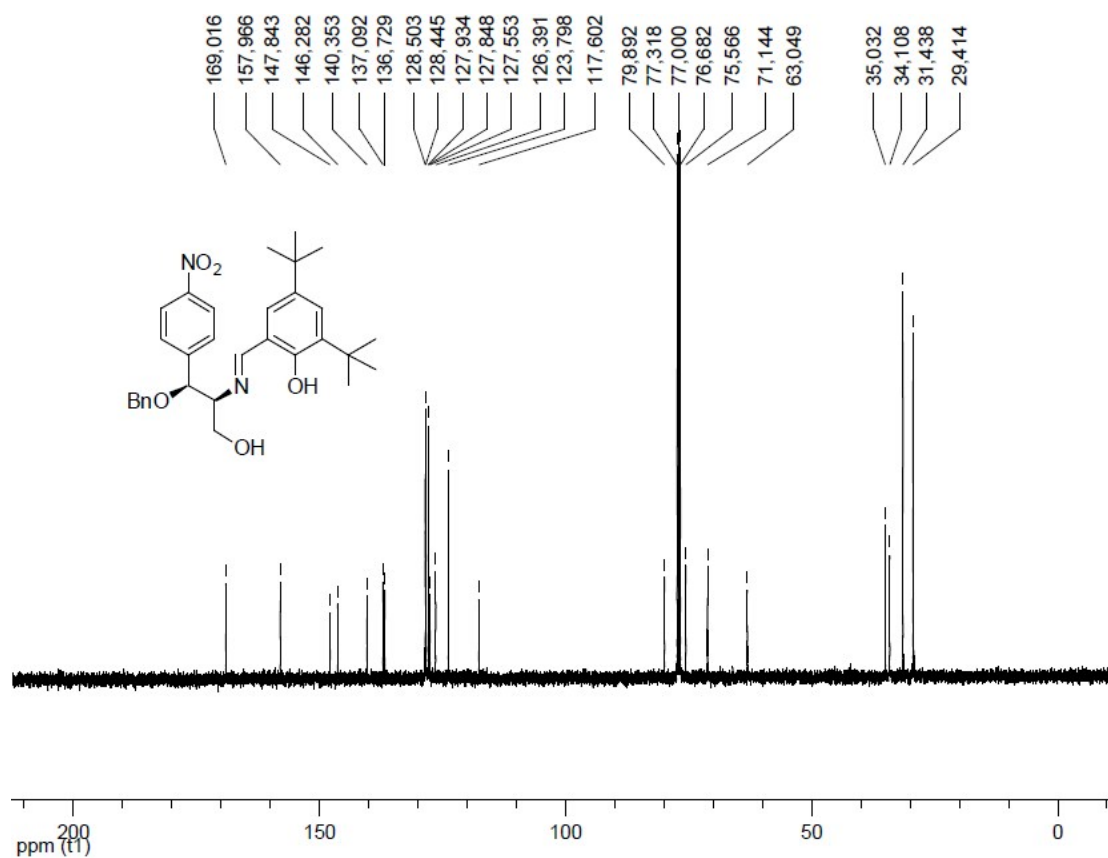
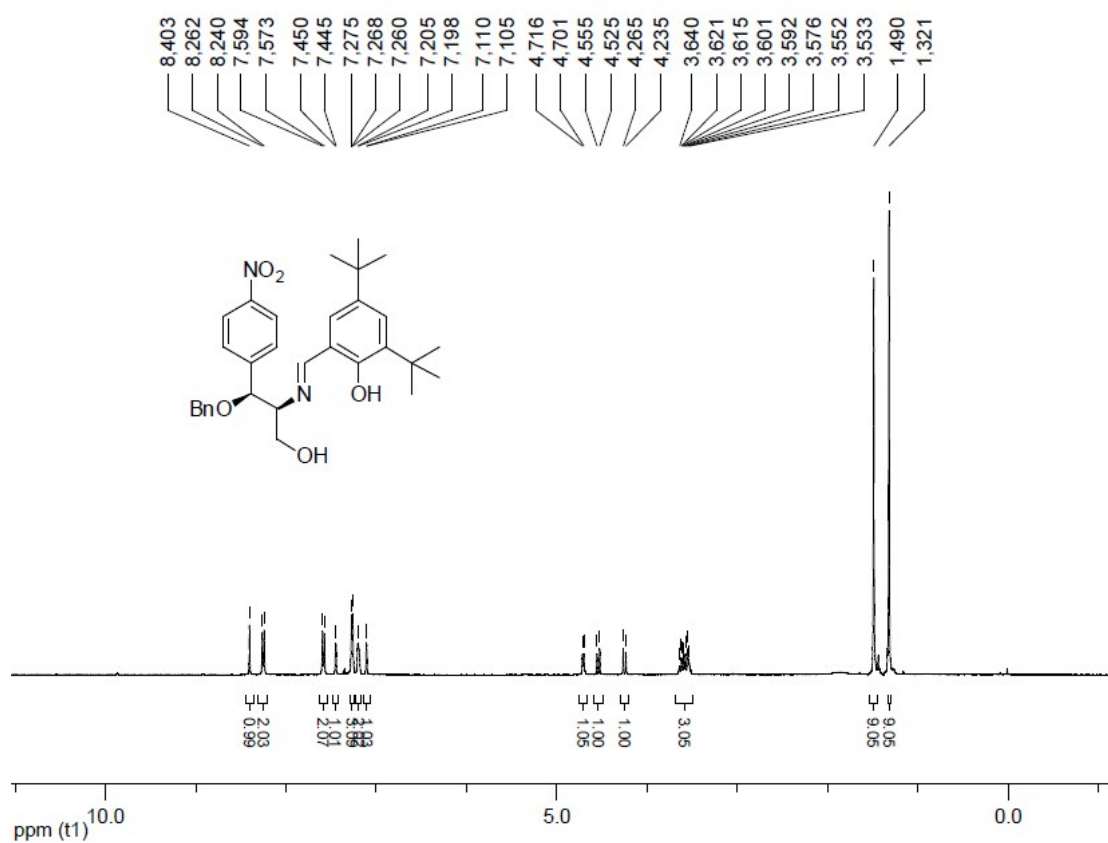


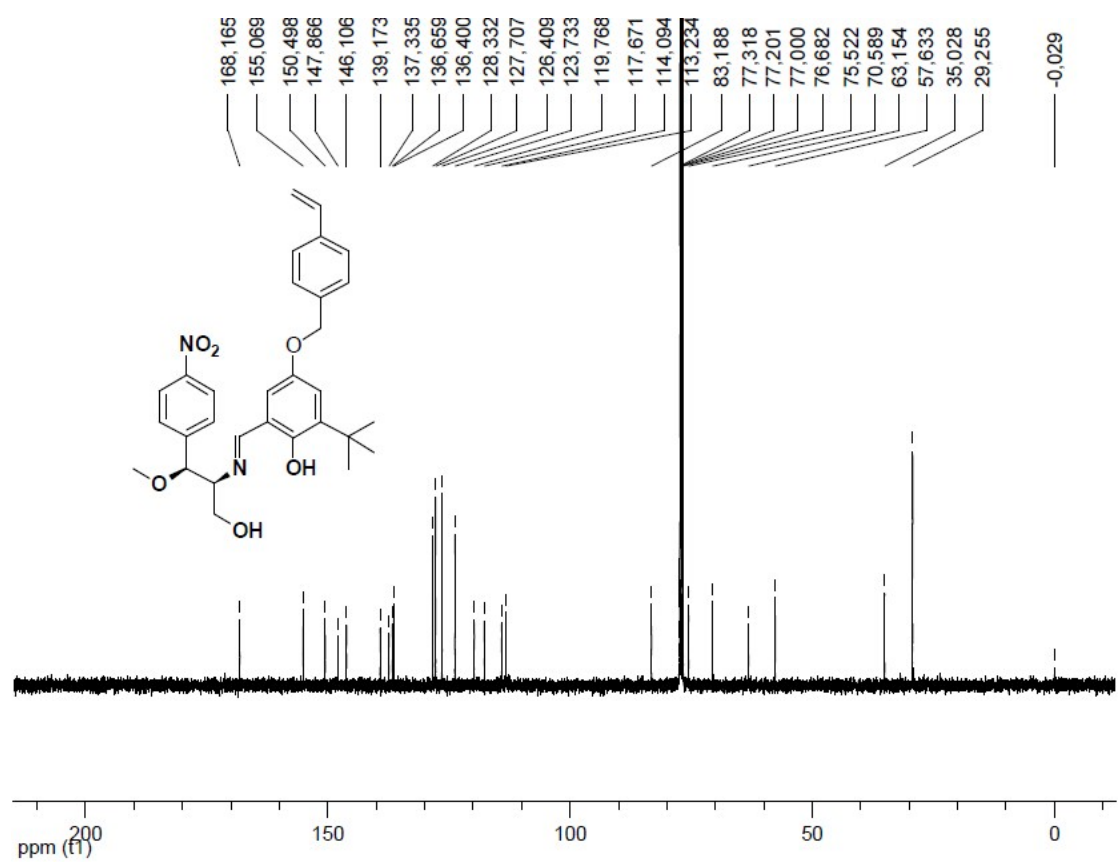
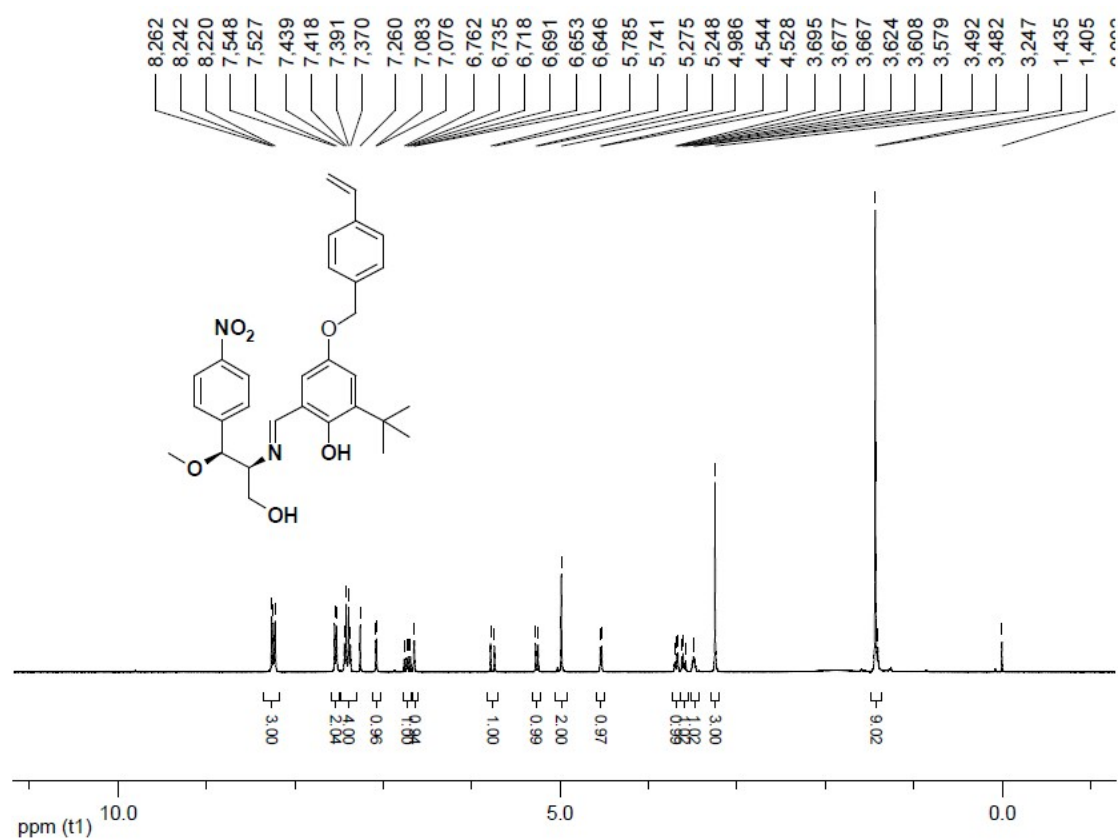
La



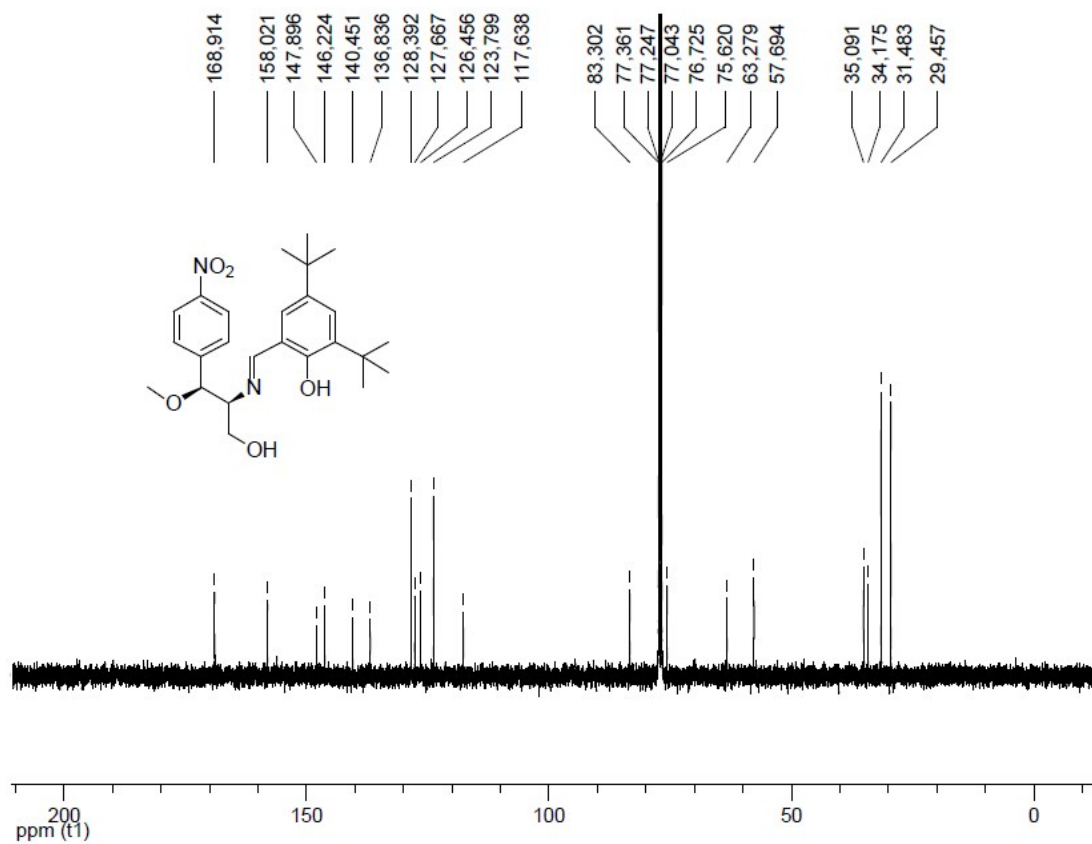
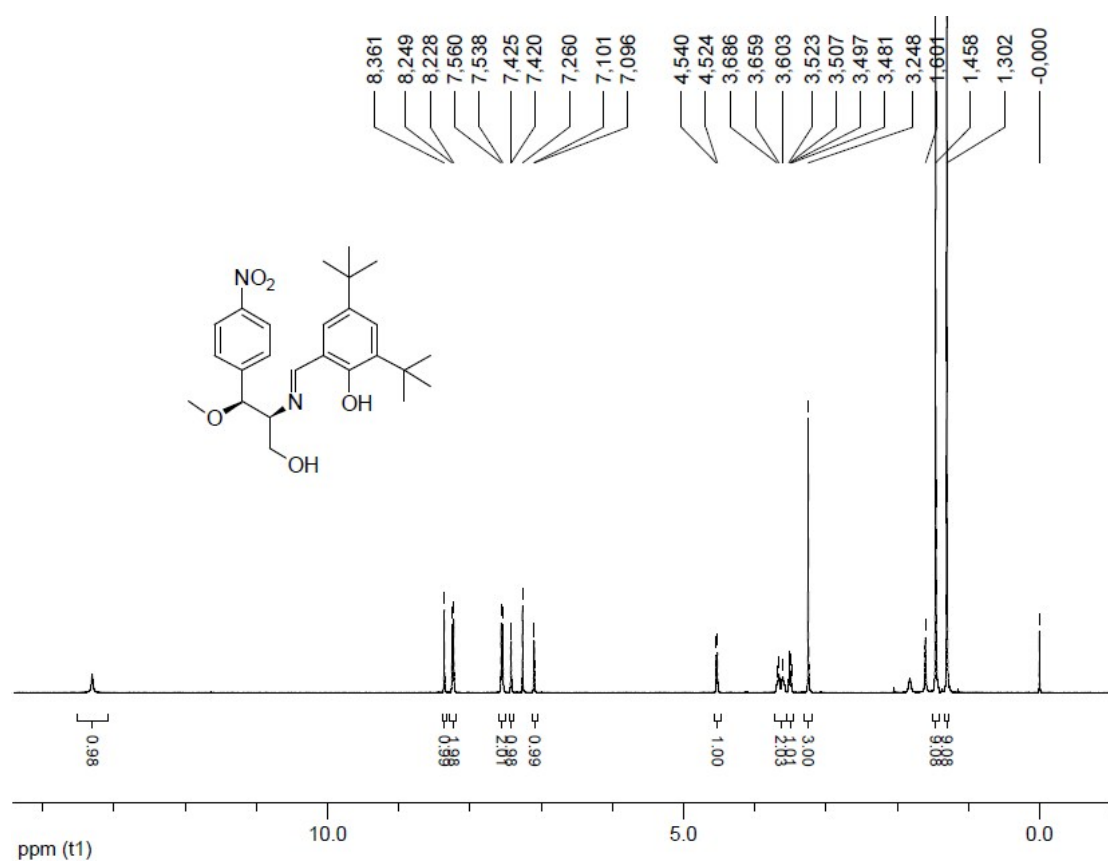
Lb



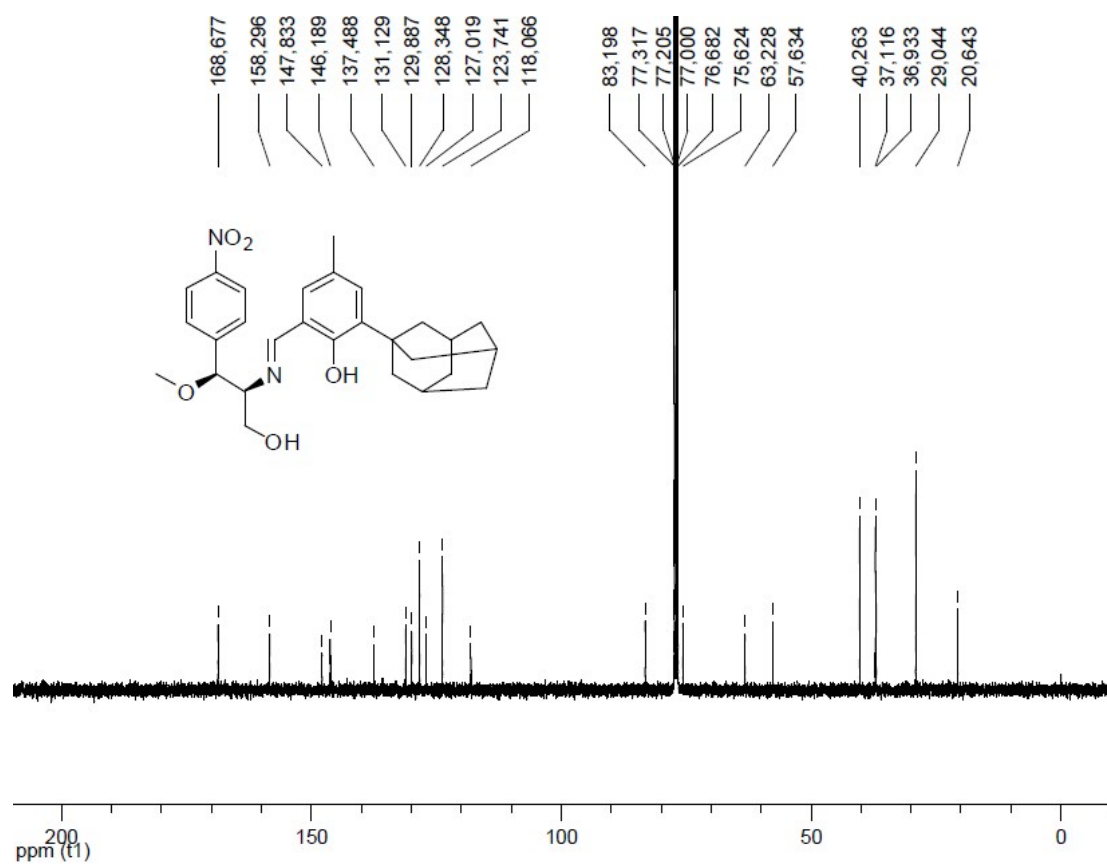
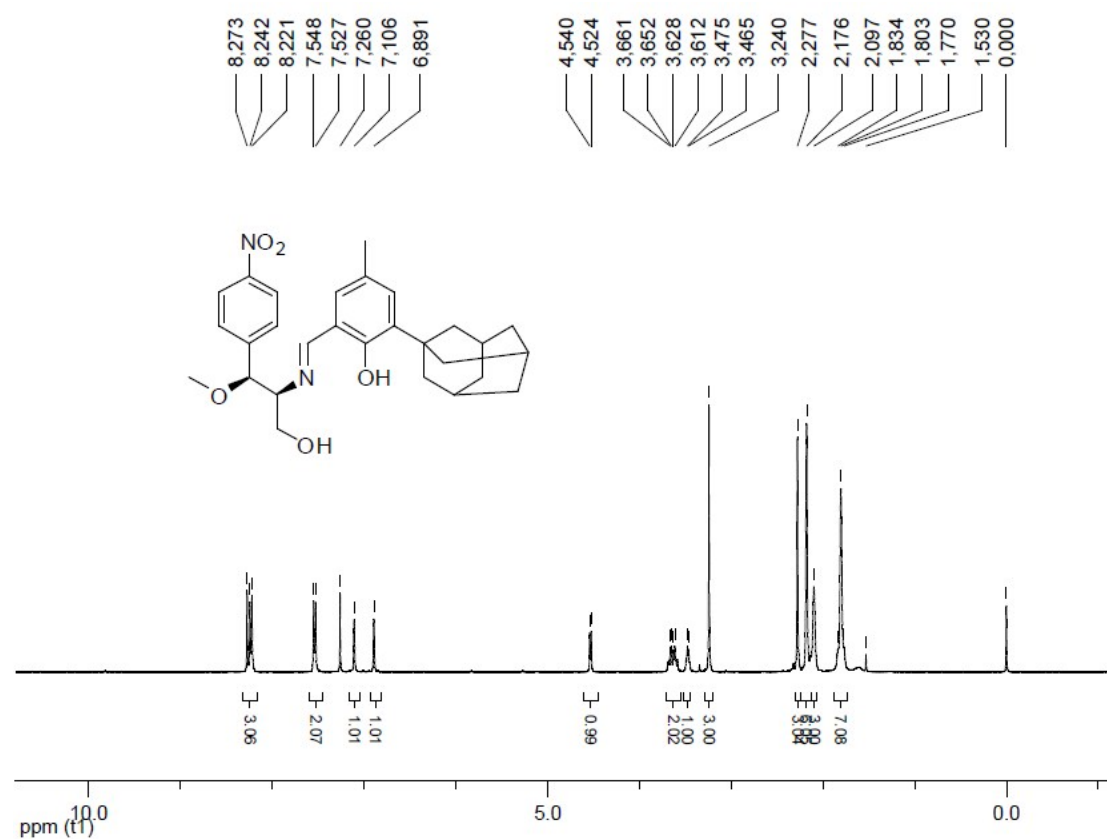
Lc



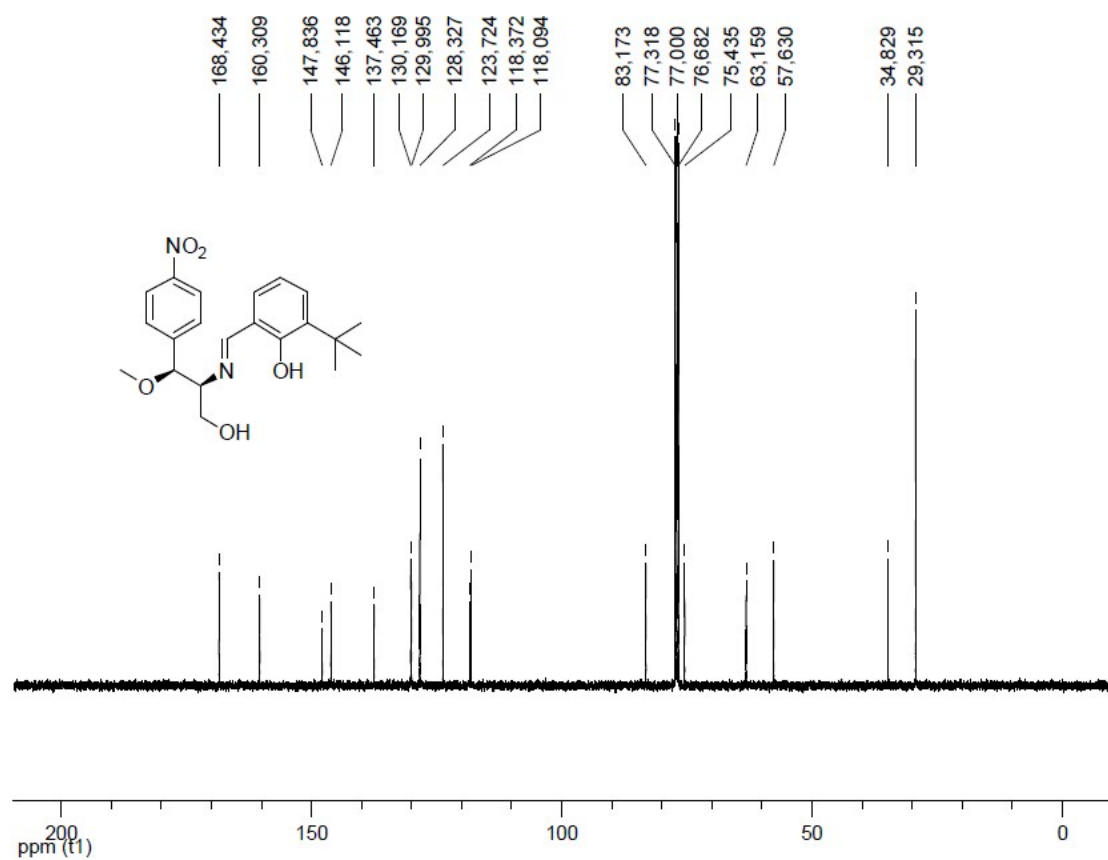
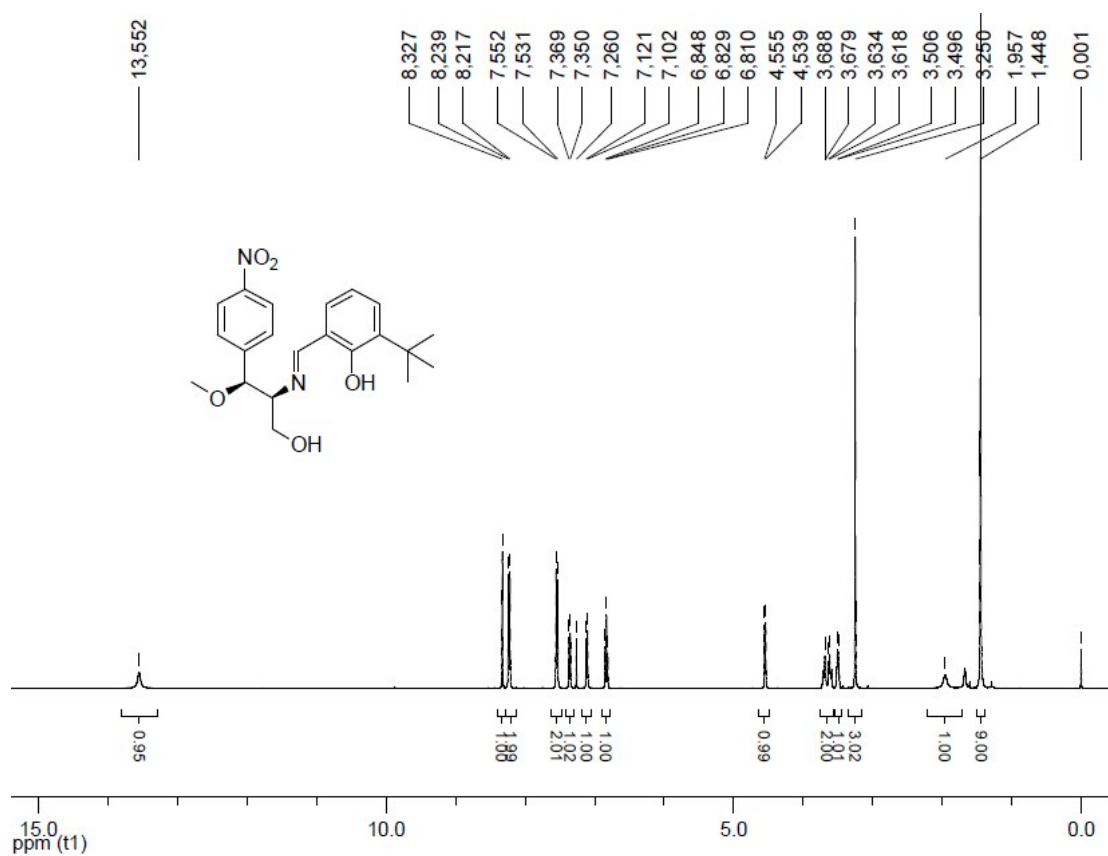
Ld



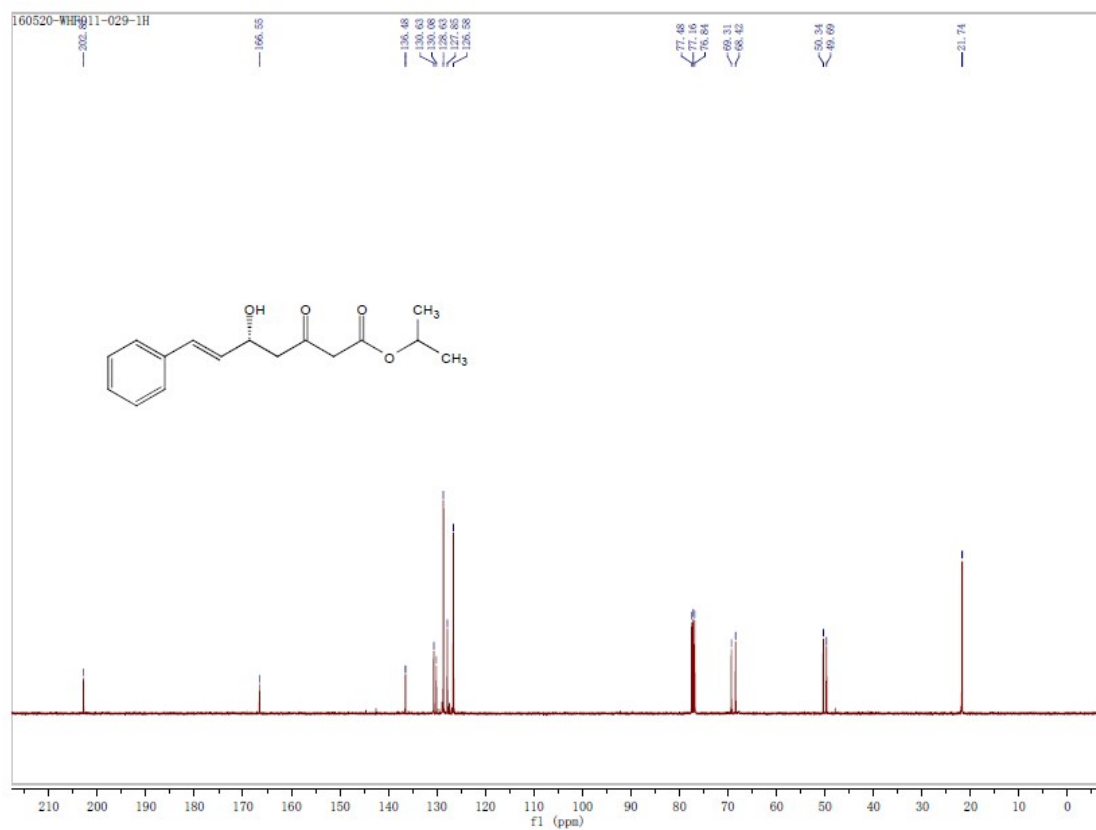
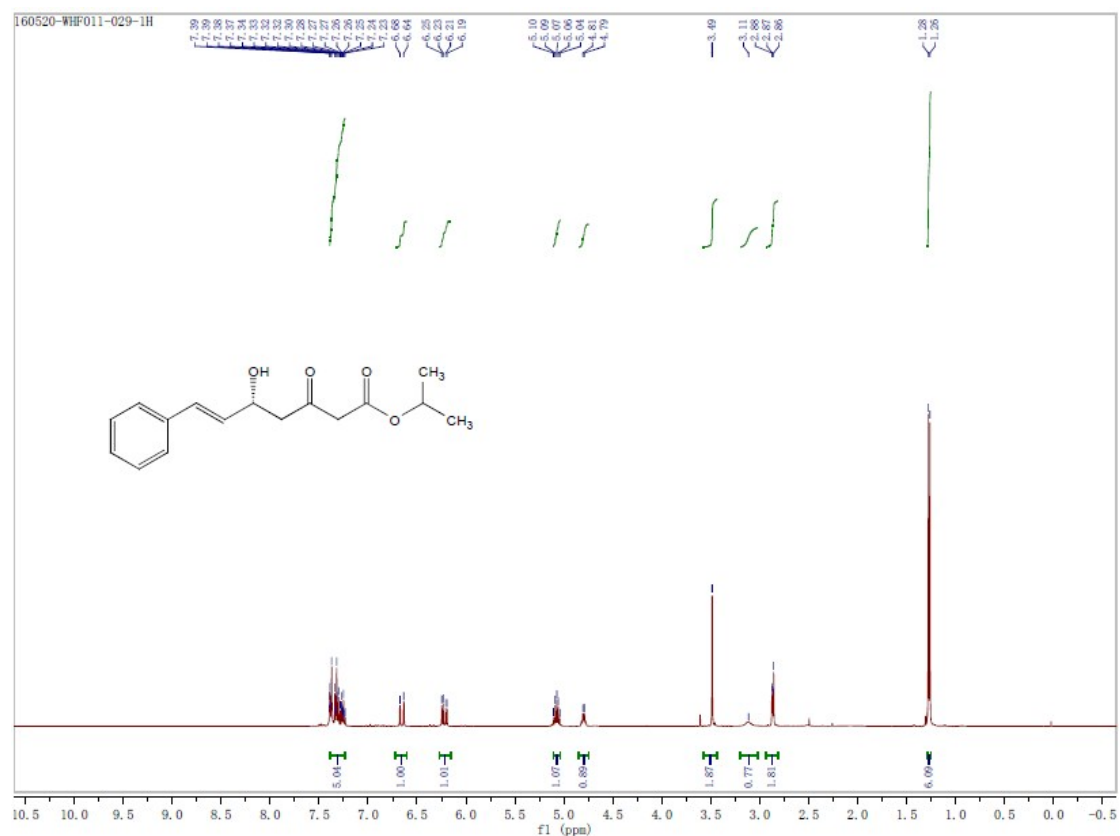
Le

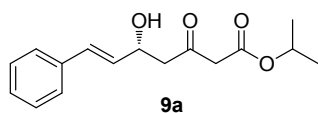


Lf

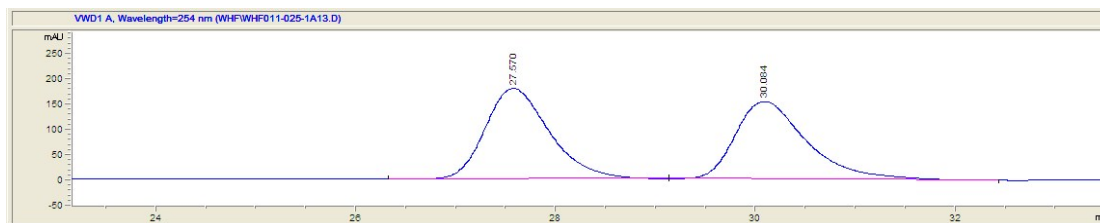


9a

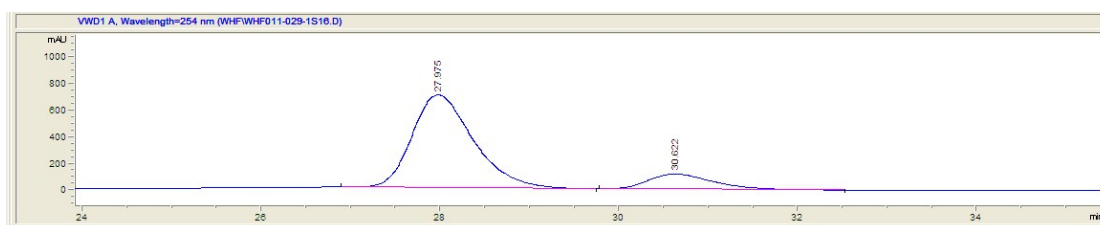




Chiralpak AD-H column (25 cm × 4.6 mm × 5 μm), n-hexane/isopropanol =95/5, 1mL/min, 254 nm, 30°C, t_R(major) = 27.975 min, t_R (minor) = 30.622 min

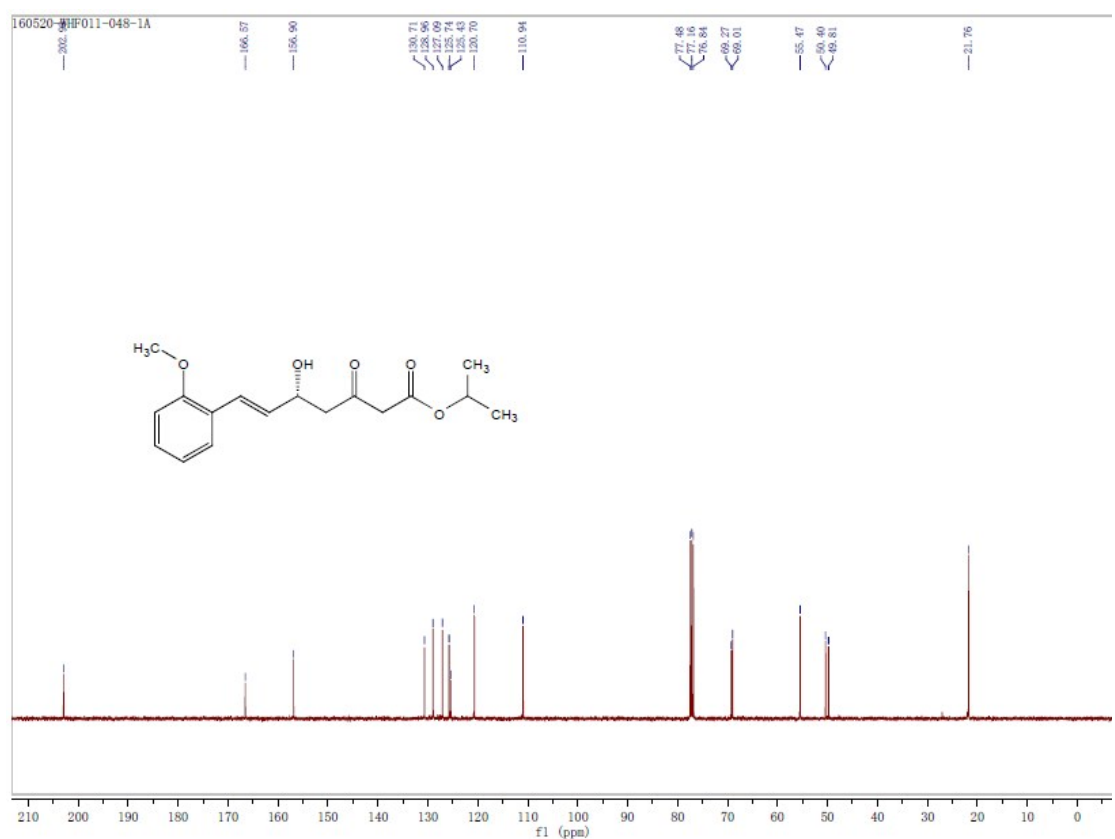
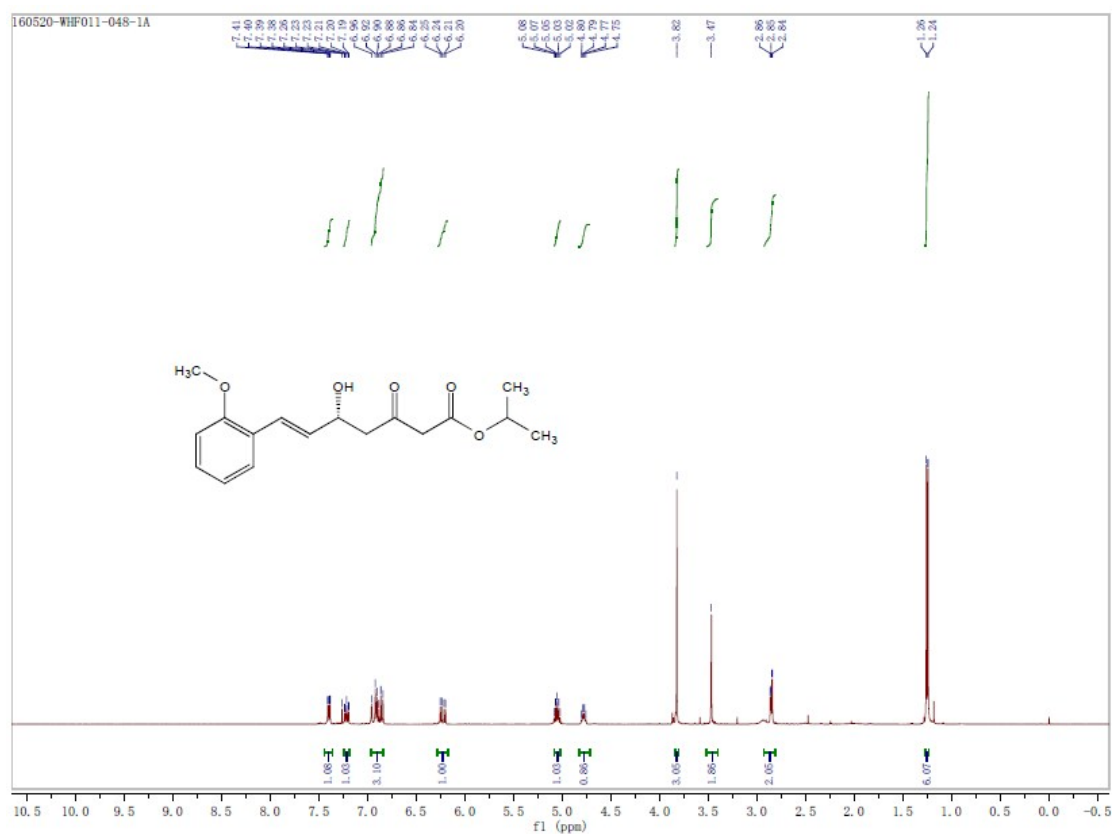


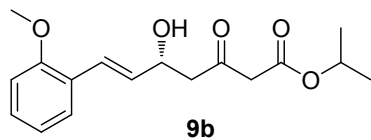
Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	27.57	8013	178.7	0.6872	0.735	51.167
2	30.084	7647.4	153.1	0.7471	0.636	48.833



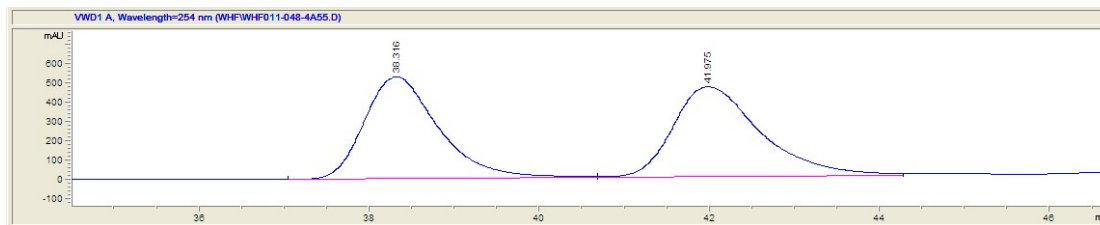
Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	27.975	33147.4	694.8	0.7276	0.701	85.216
2	30.622	5750.5	113.3	0.7645	0.67	14.784

9b

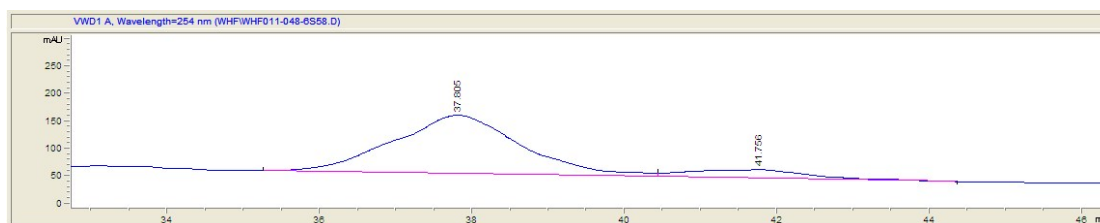




Chiralpak AD-H column (25 cm × 4.6 mm × 5 μm), n-hexane/isopropanol =95/5, 0.6mL/min, 254 nm, 30°C, tR(major) = 37.805 min, tR (minor) = 41.756 min

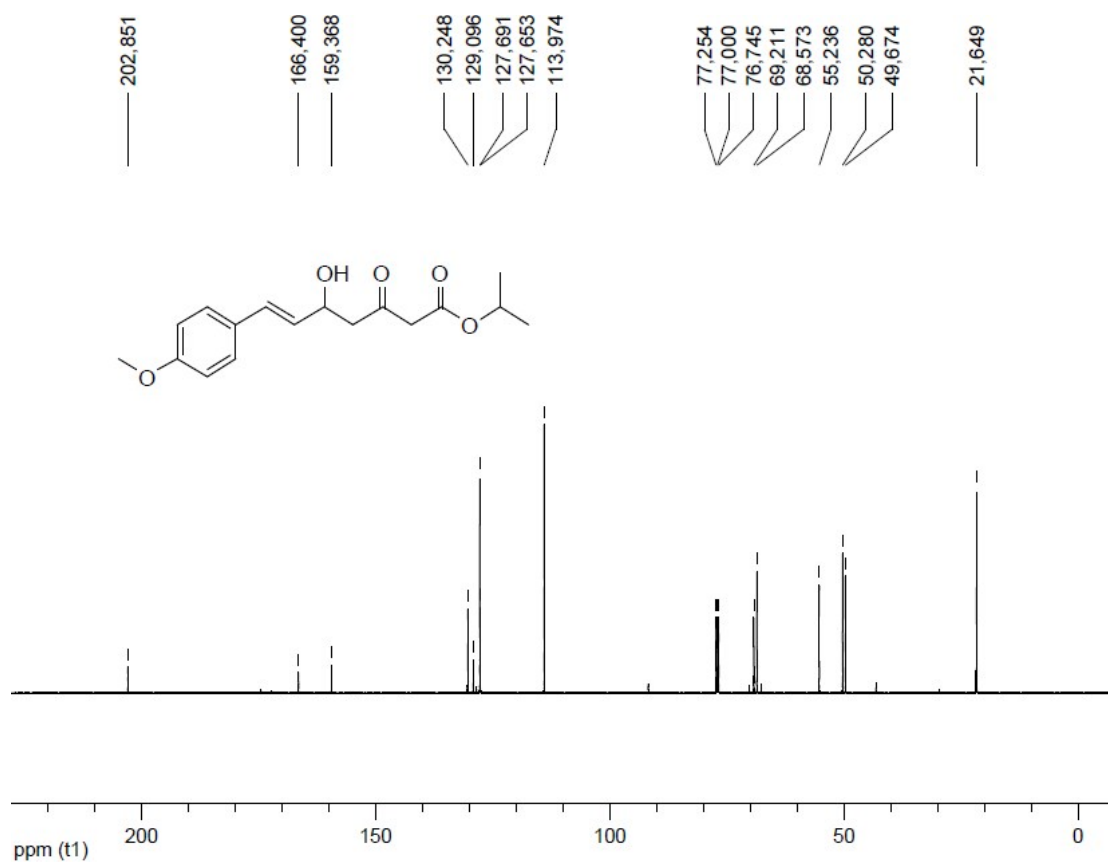
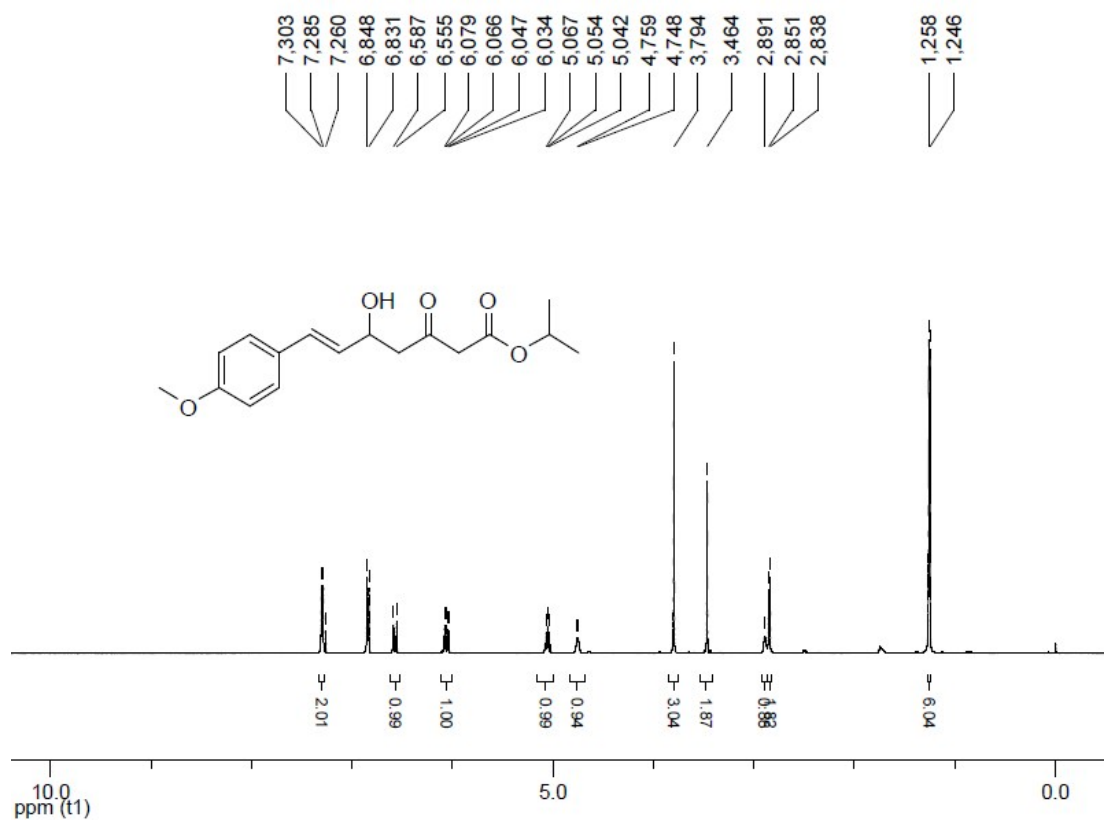


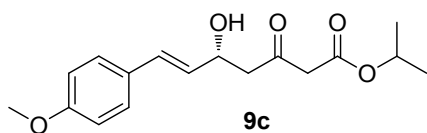
Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	38.316	32086.7	528.4	0.9165	0.712	48.339
2	41.975	33586.5	466.5	1.03	0.617	50.599



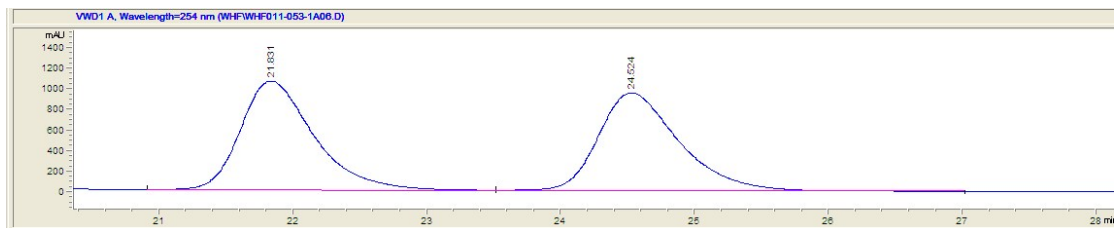
Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	37.805	12497.9	106.7	1.6866	0.969	87.695
2	41.756	1753.7	16.2	1.362	1.206	12.305

9c

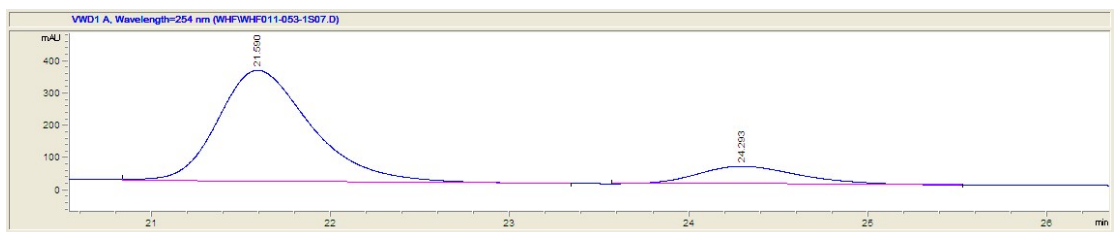




Chiralpak AD-H column (25 cm × 4.6 mm × 5 μm), n-hexane/isopropanol =90/10, 1mL/min, 254 nm, 30°C, t_R(major) = 21.59 min, t_R (minor) = 24.293 min

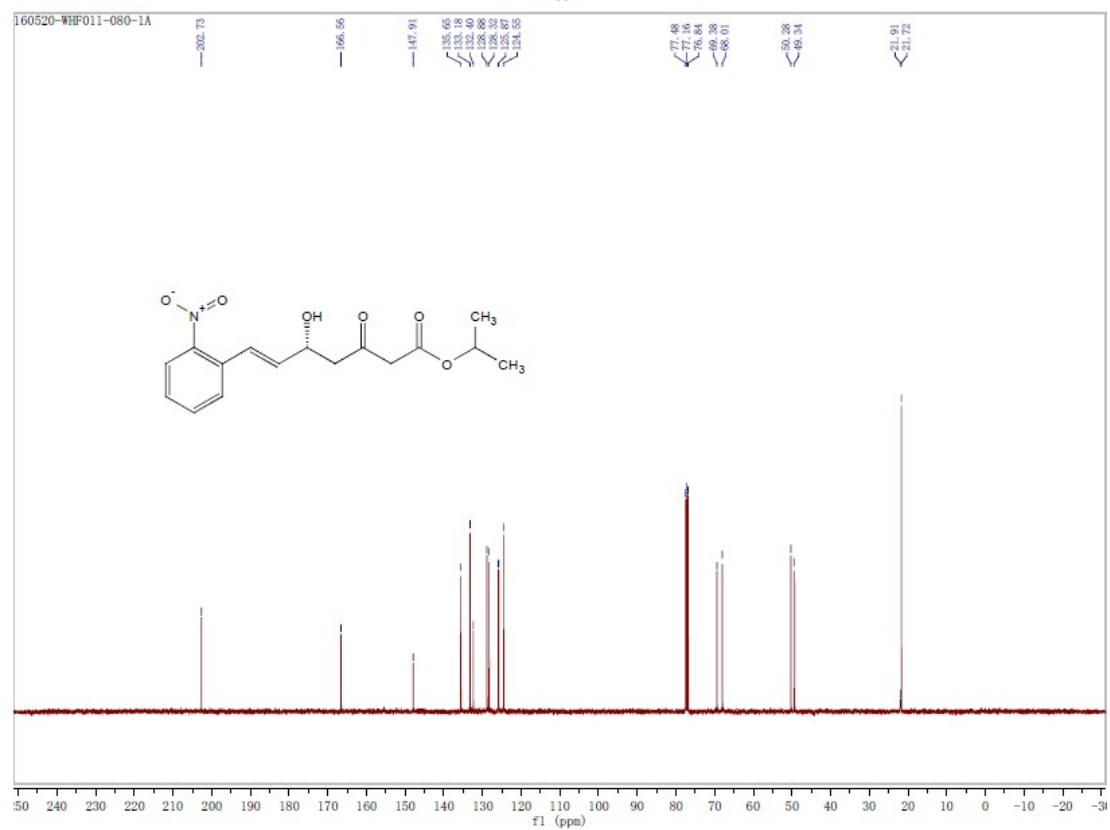
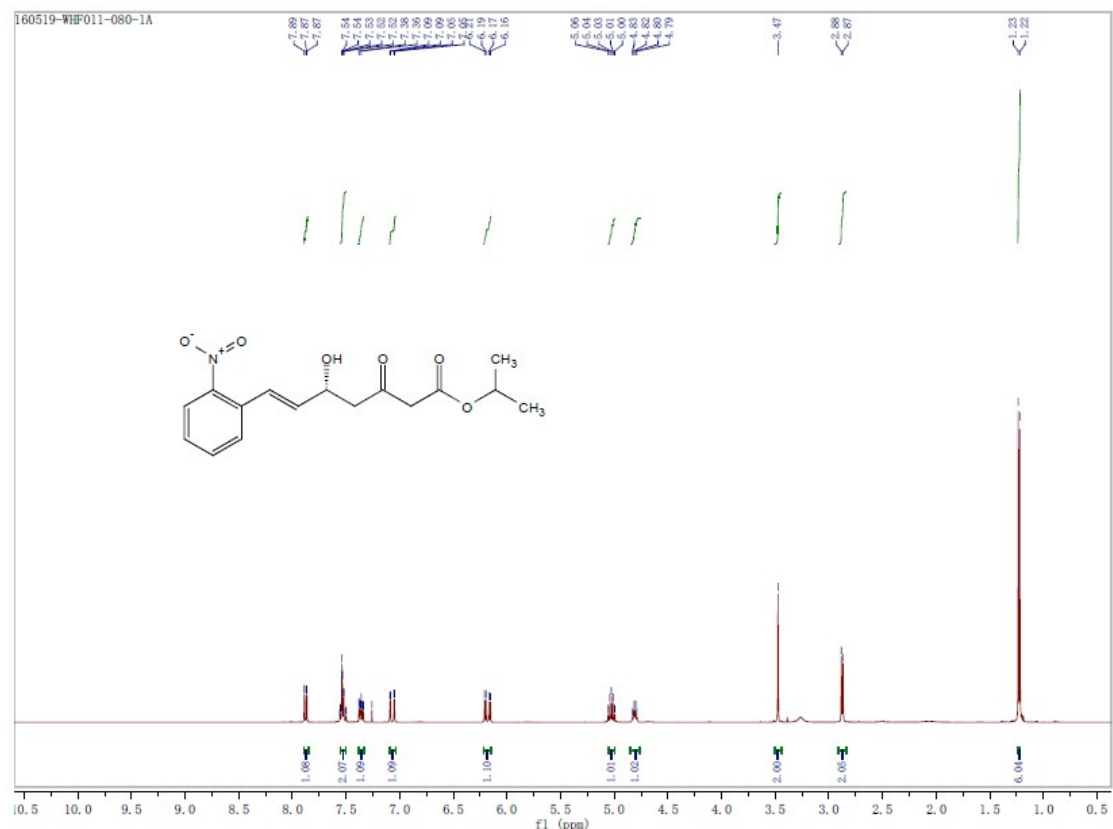


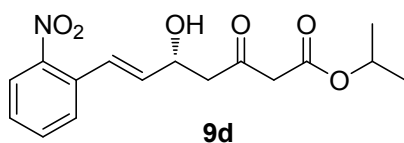
Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	21.831	41299.5	1063.3	0.5851	0.665	50.530
2	24.524	40432.9	953.4	0.6388	0.654	49.470



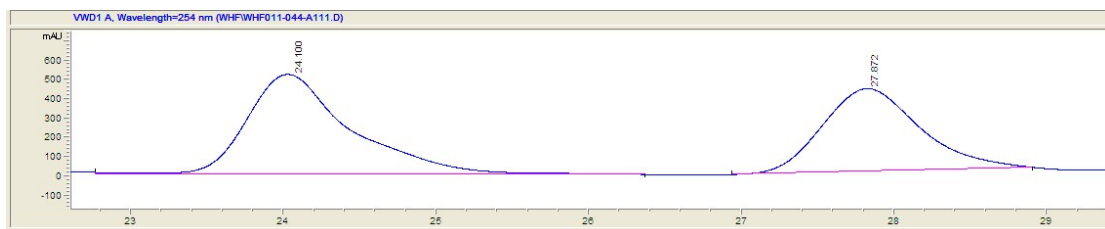
Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	21.59	12621.5	345.4	0.5543	0.71	85.070
2	24.293	2215.2	55.5	0.6173	0.717	14.930

9d

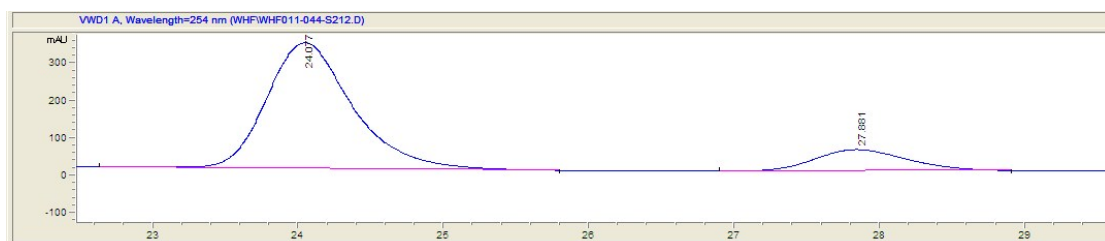




Chiralpak AD-H column (25 cm × 4.6 mm × 5 μm), n-hexane/isopropanol =90/10, 1mL/min, 254 nm, 30°C, t_R(major) = 24.077 min, t_R (minor) = 27.881 min

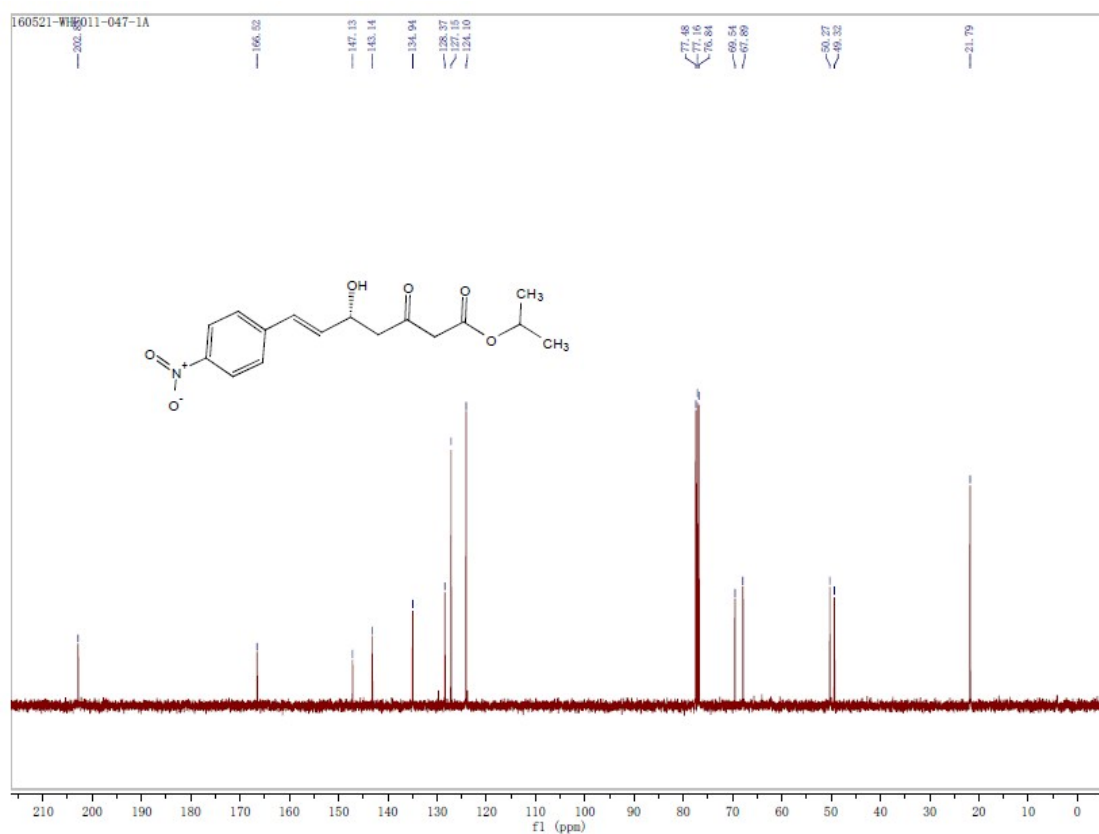
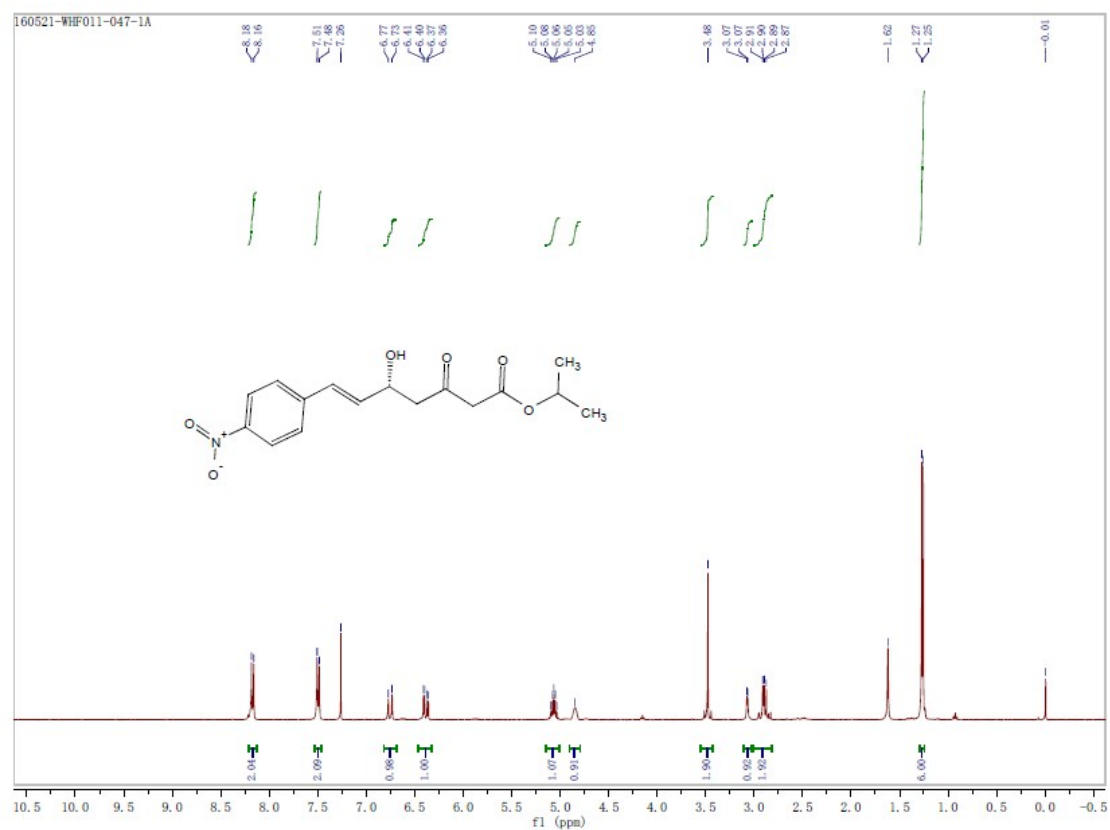


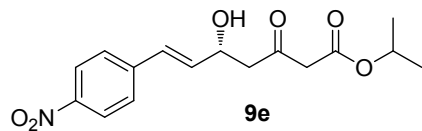
Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	24.1	24883.8	507.6	0.8279	0.792	56.784
2	27.872	18937.9	416.8	0.8501	1.025	43.216



Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	24.077	13872	324.4	0.7549	0.863	84.609
2	27.881	2523.4	55.3	0.8476	0.98	15.391

9e

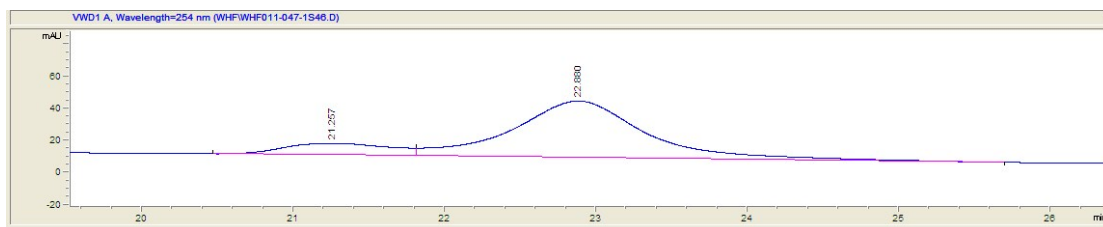




Chiralpak AD-H column (25 cm × 4.6 mm × 5 μm), n-hexane/isopropanol =90/10, 1mL/min, 254 nm, 30°C, t_R(major) = 22.88 min, t_R (minor) = 21.257 min

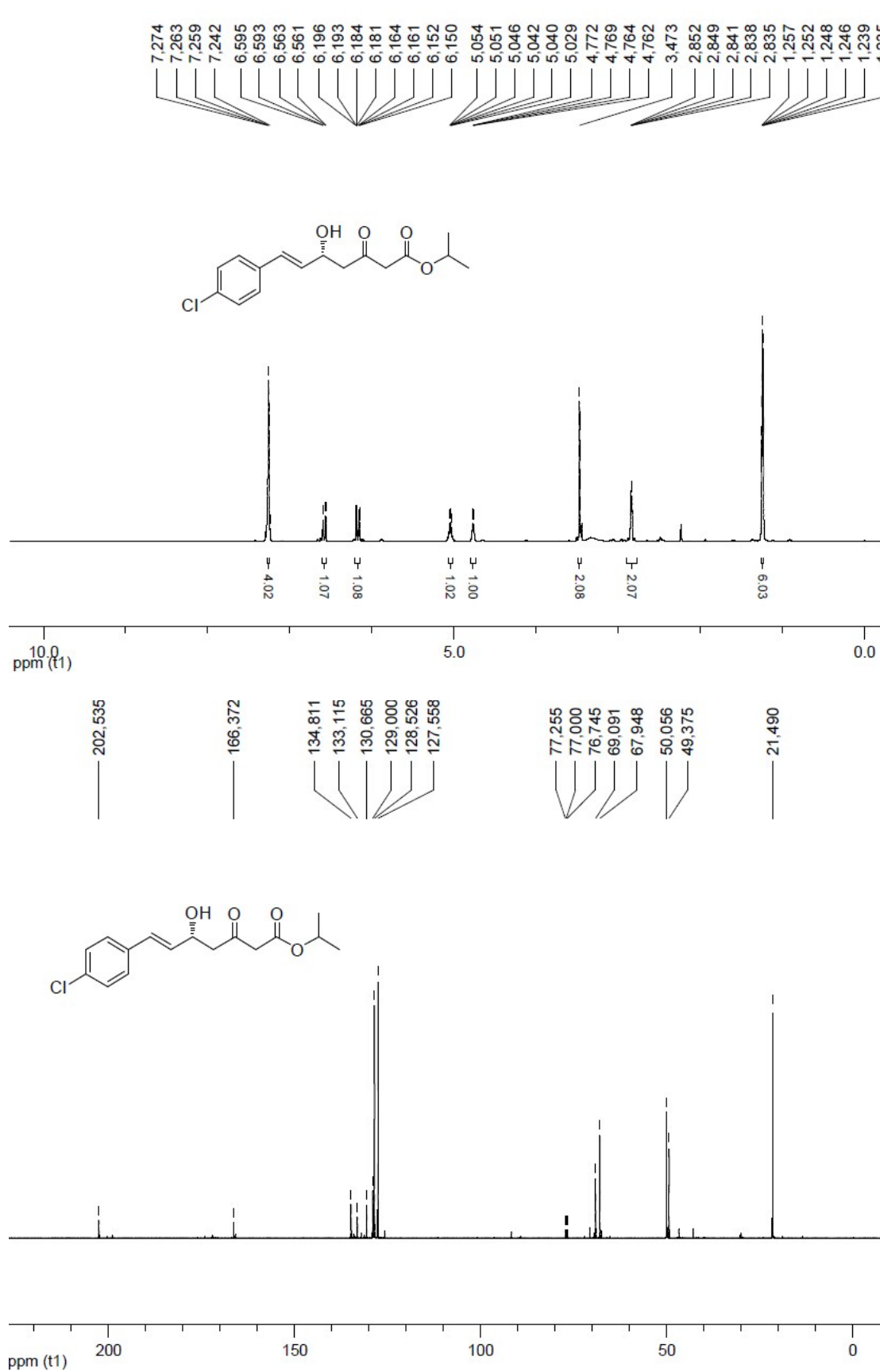


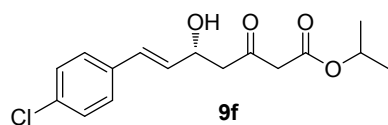
Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	20.834	845.8	20.8	0.6024	0.637	54.332
2	22.652	710.9	17.3	0.6229	0.708	45.668



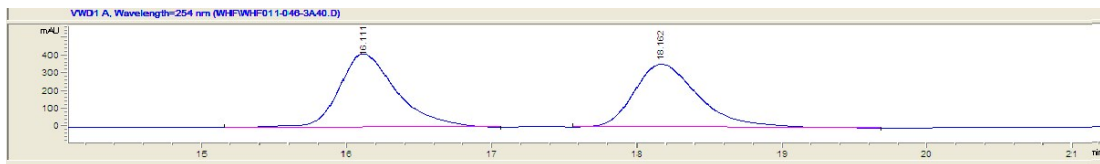
Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	21.257	362.8	7.4	0.7411	0.796	14.263
2	22.8	2181	35.3	0.8962	0.914	85.737

9f

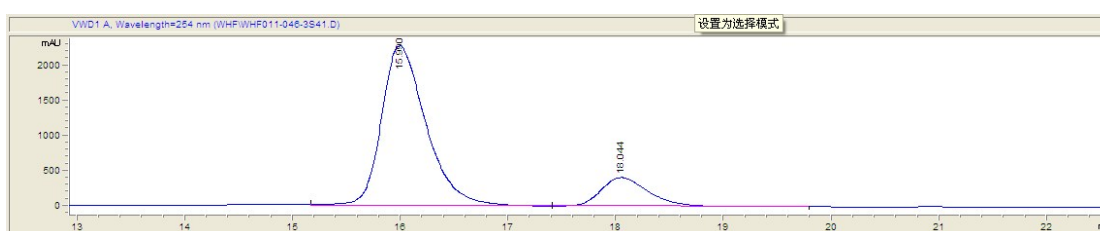




Chiralpak AD-H column (25 cm × 4.6 mm × 5 μm), n-hexane/isopropanol =90/10, 1mL/min, 254 nm, 30°C, t_R(major) = 15.99 min, t_R (minor) = 18.044 min

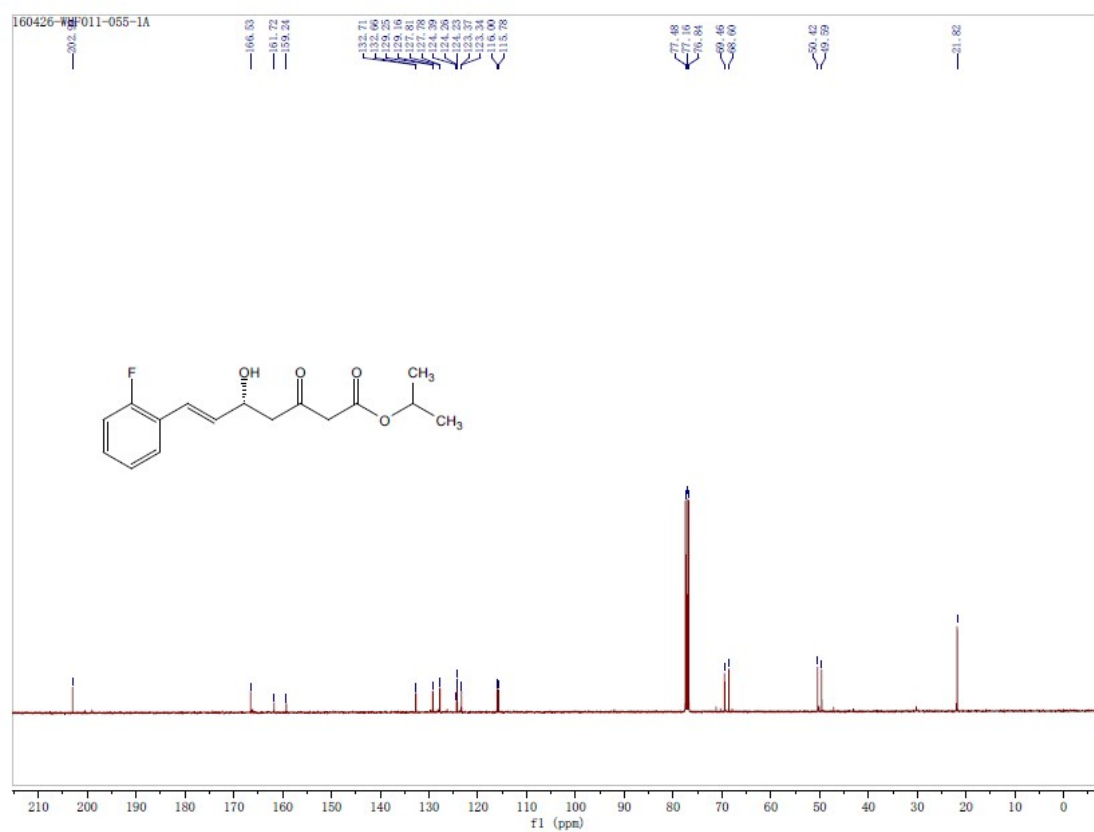
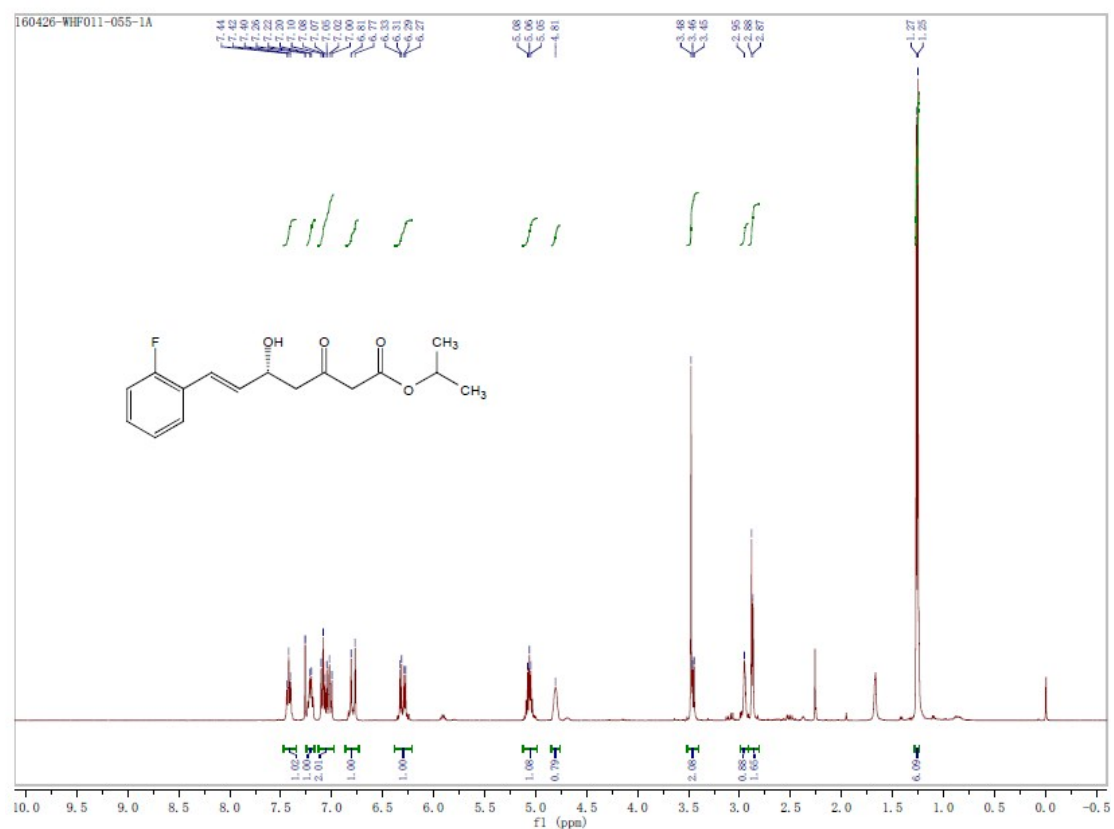


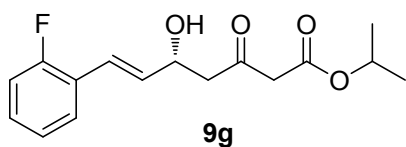
Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	16.111	11553.4	414.3	0.4193	0.717	51.086
2	18.162	11062.2	356.2	0.4723	0.682	48.914



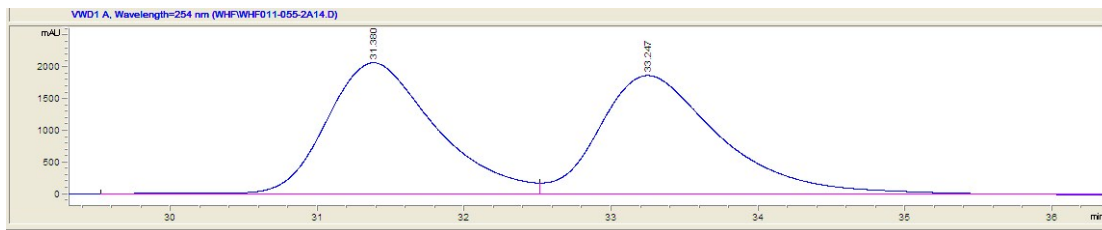
Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	15.99	65973	2278.6	0.4398	0.669	84.029
2	18.044	12539.2	410.3	0.4646	0.701	15.971

9g

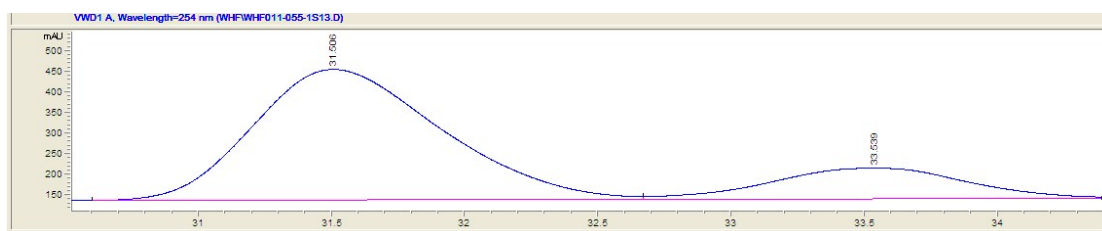




Chiralpak AD-H column (25 cm × 4.6 mm × 5 μm), n-hexane/isopropanol =95/5, 0.8mL/min, 254 nm, 30°C, t_R(major) = 31.506 min, t_R (minor) = 33.539 min



Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	31.38	107698.1	2073.2	0.7901	0.719	49.963
2	33.247	107855.7	1874	0.8634	0.618	50.037



Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	31.506	15611.7	317.1	0.7503	0.715	80.102
2	33.539	3878.2	75	0.7911	1.097	19.898

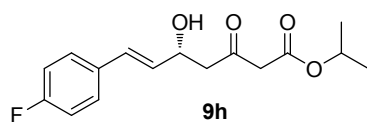
160520-WHF011-054-1A

Chemical structure: CC(C)OC(=O)CC(=O)C[C@H](O)/C=C/c1ccc(F)cc1

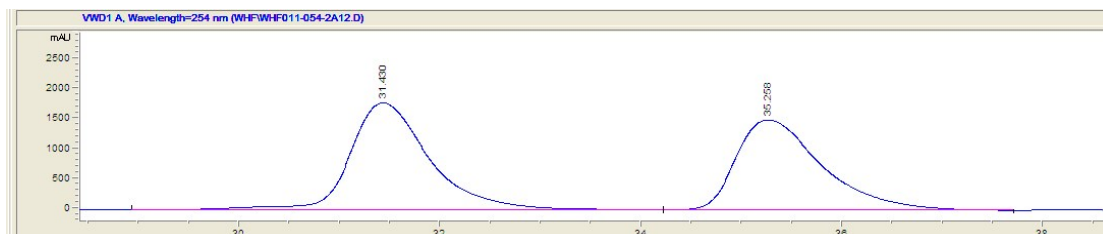
¹H NMR spectrum (ppm):

- 7.40 (d, 2H, integration 1.97)
- 7.00 (d, 2H, integration 2.00)
- 6.50 (m, 1H, integration 0.94)
- 6.00 (m, 1H, integration 0.97)
- 4.80 (d, 1H, integration 1.01)
- 4.50 (d, 1H, integration 0.85)
- 3.50 (s, 3H, integration 1.78)
- 3.00 (s, 3H, integration 0.77)
- 2.80 (s, 3H, integration 1.71)
- 1.10 (s, 6H, integration 6.00)

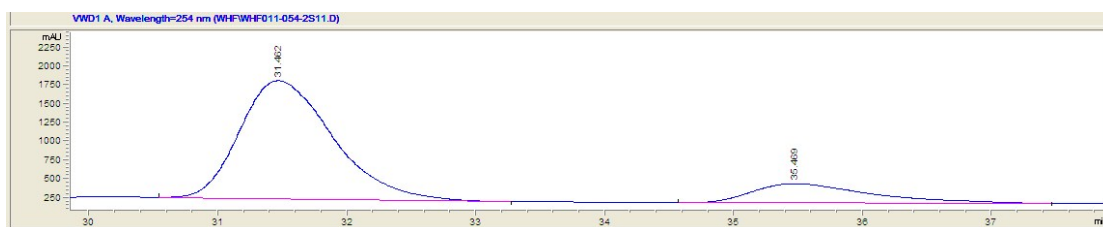




Chiralpak AD-H column (25 cm × 4.6 mm × 5 μm), n-hexane/isopropanol =95/5, 0.8mL/min, 254 nm, 30°C, t_R(major) = 31.462 min, t_R (minor) = 35.469 min



Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	31.43	100301.1	1794.9	0.8439	0.691	52.653
2	35.258	90194.5	1514.2	0.9029	0.547	47.347



Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	31.462	30541.9	1582.3	0.7939	0.672	82.184
2	35.469	17459.5	261.5	0.9469	0.497	17.816

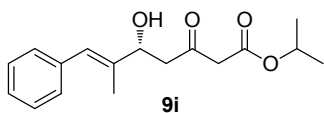
160428-WH011-070-1A

Chemical structure: CC(C)OC(=O)CC(=O)CC(O)C(=C)c1ccccc1

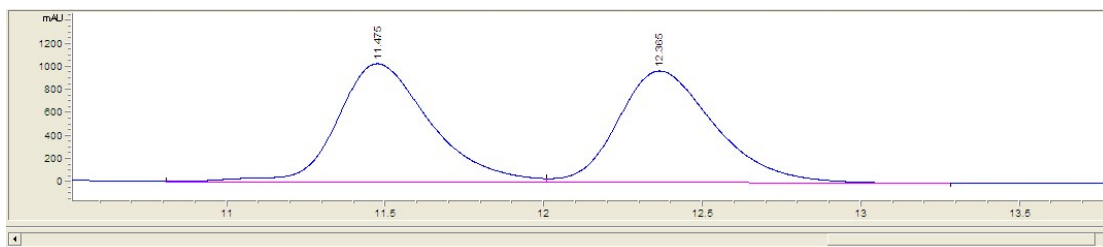
¹H NMR spectrum (CDCl₃) showing peaks and integrations:

Chemical Shift (ppm)	Integration
7.25-7.35	1.96, 3.07
6.55	0.92
4.95	1.04
4.65	0.81
3.55	1.90
2.95	2.01
2.05	2.82
1.25	6.01

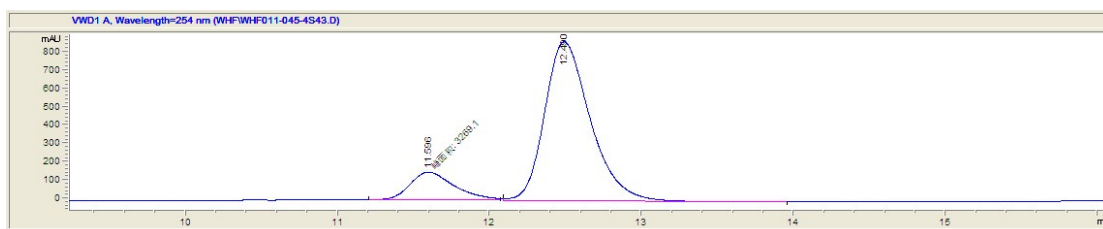




Chiralpak AD-H column (25 cm × 4.6 mm × 5 μm), n-hexane/isopropanol =90/10, 1mL/min, 254 nm, 30°C, t_R(major) = 12.49 min, t_R (minor) = 11.596 min

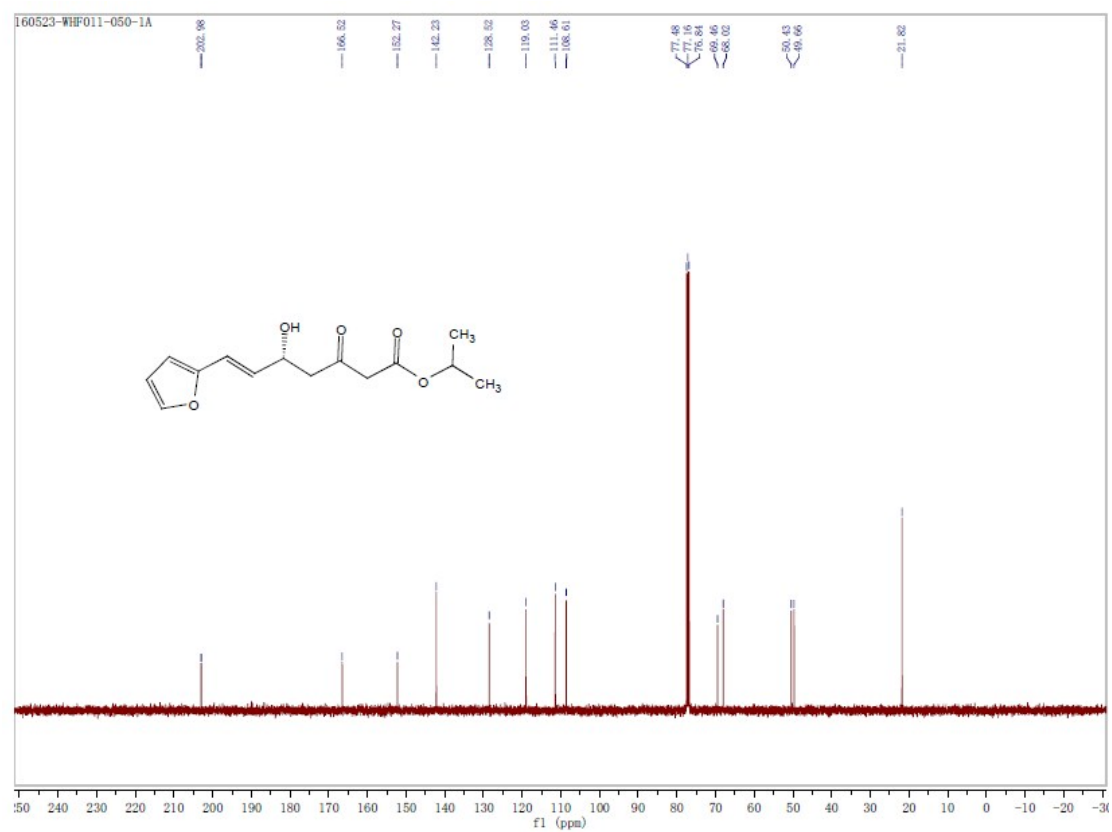
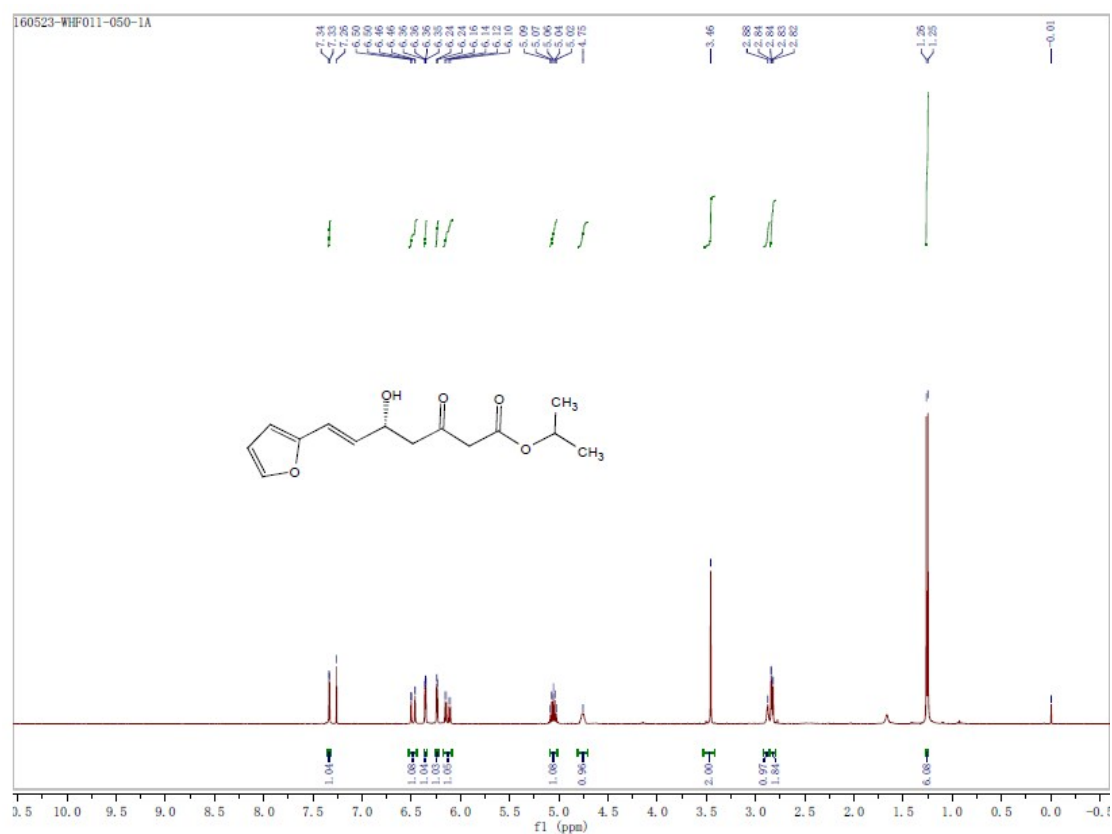


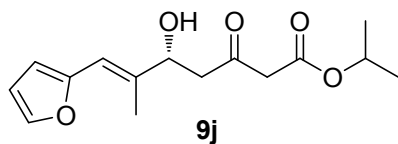
Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	11.476	21326.8	1032.7	0.3129	0.746	50.598
2	12.365	20822.6	973.1	0.3256	0.723	49.402



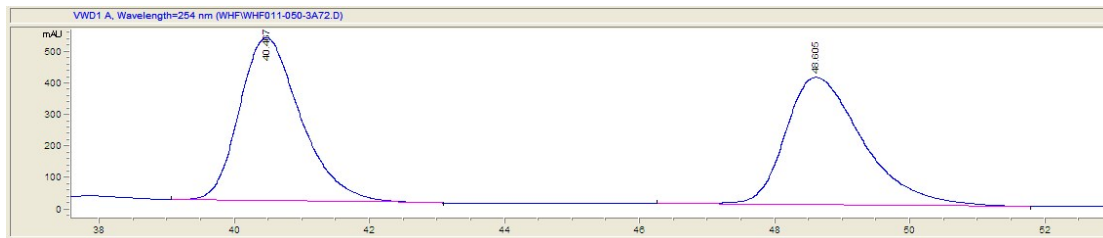
Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	11.596	3269.1	155.3	0.3509	0.681	15.223
2	12.49	18206.3	867.8	0.3208	0.714	84.777

9j

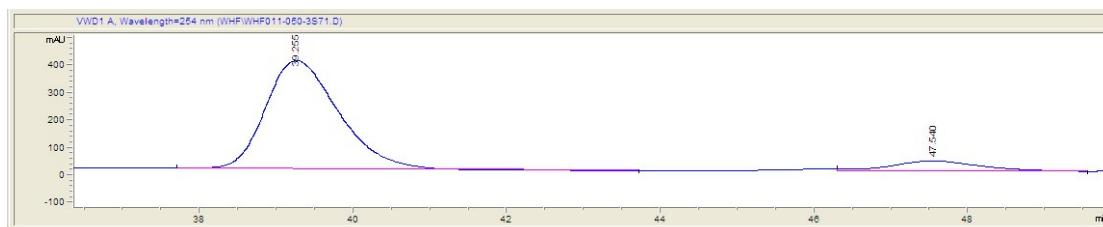




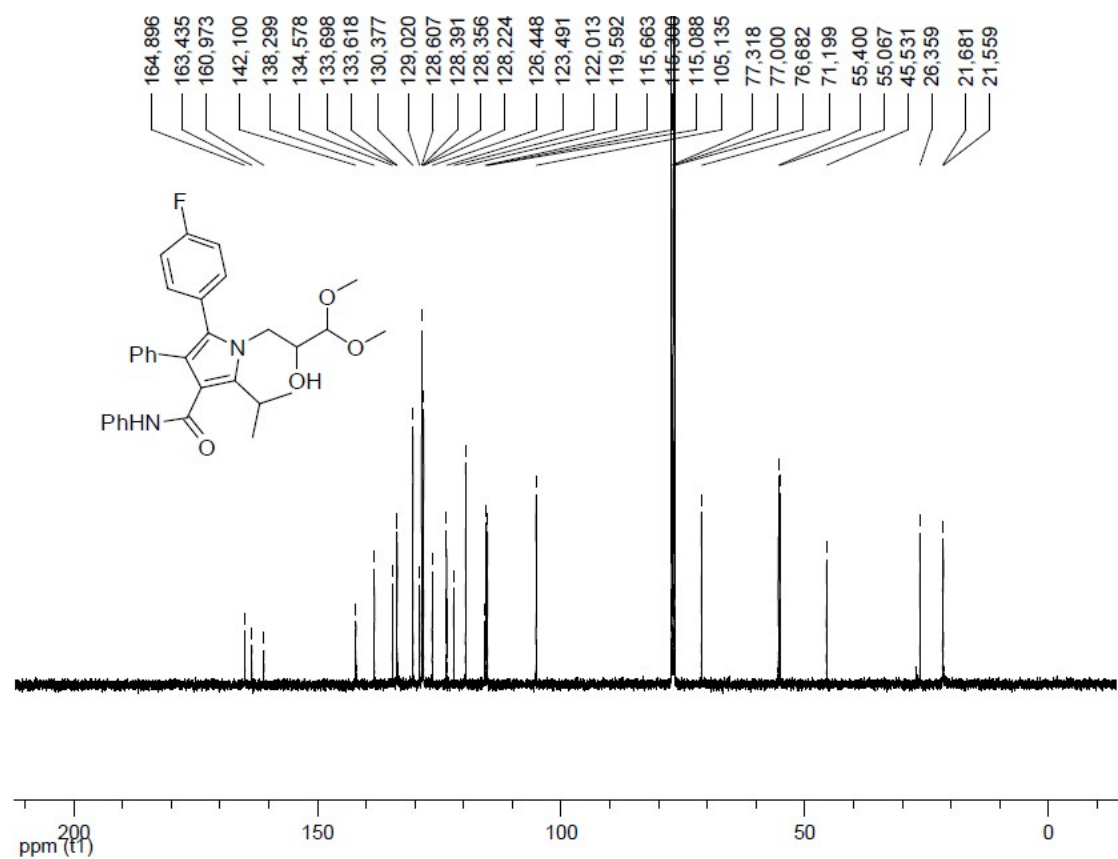
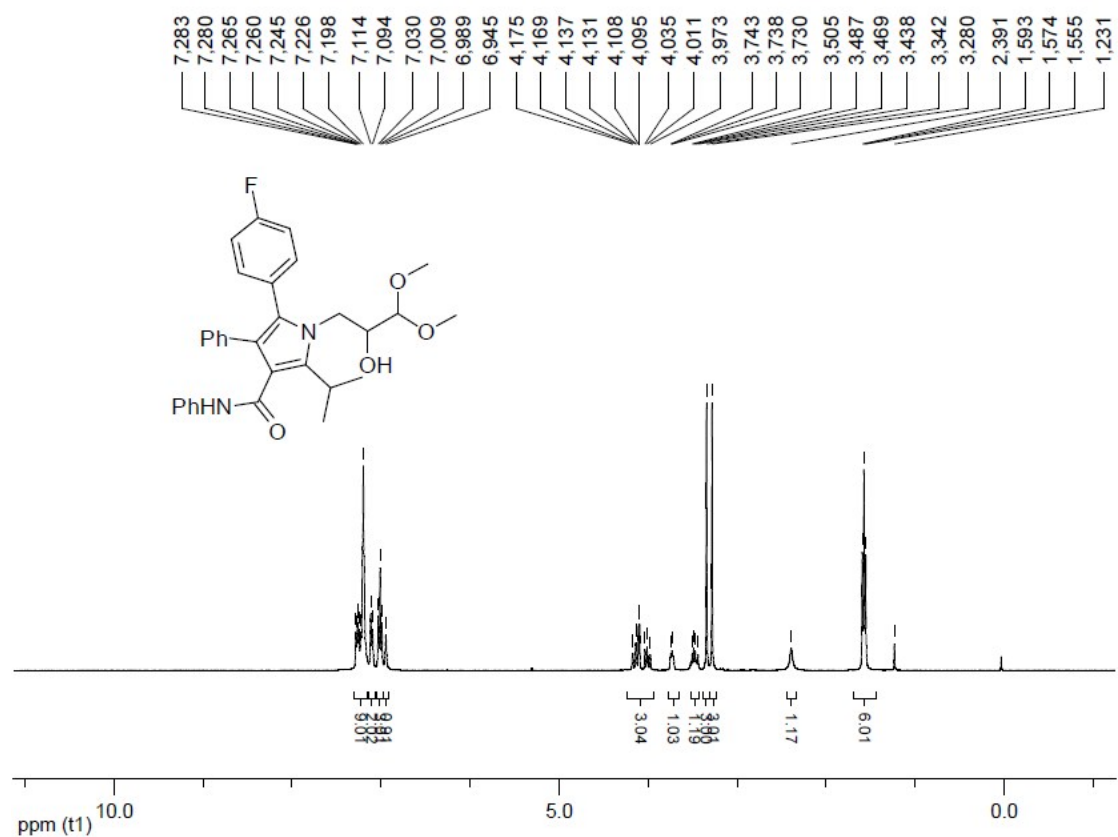
Chiralpak AD-H column (25 cm × 4.6 mm × 5 μm), n-hexane/isopropanol =90/10, 1mL/min, 254 nm, 30°C, tR(major) = 39.255 min, tR (minor) = 47.54 min

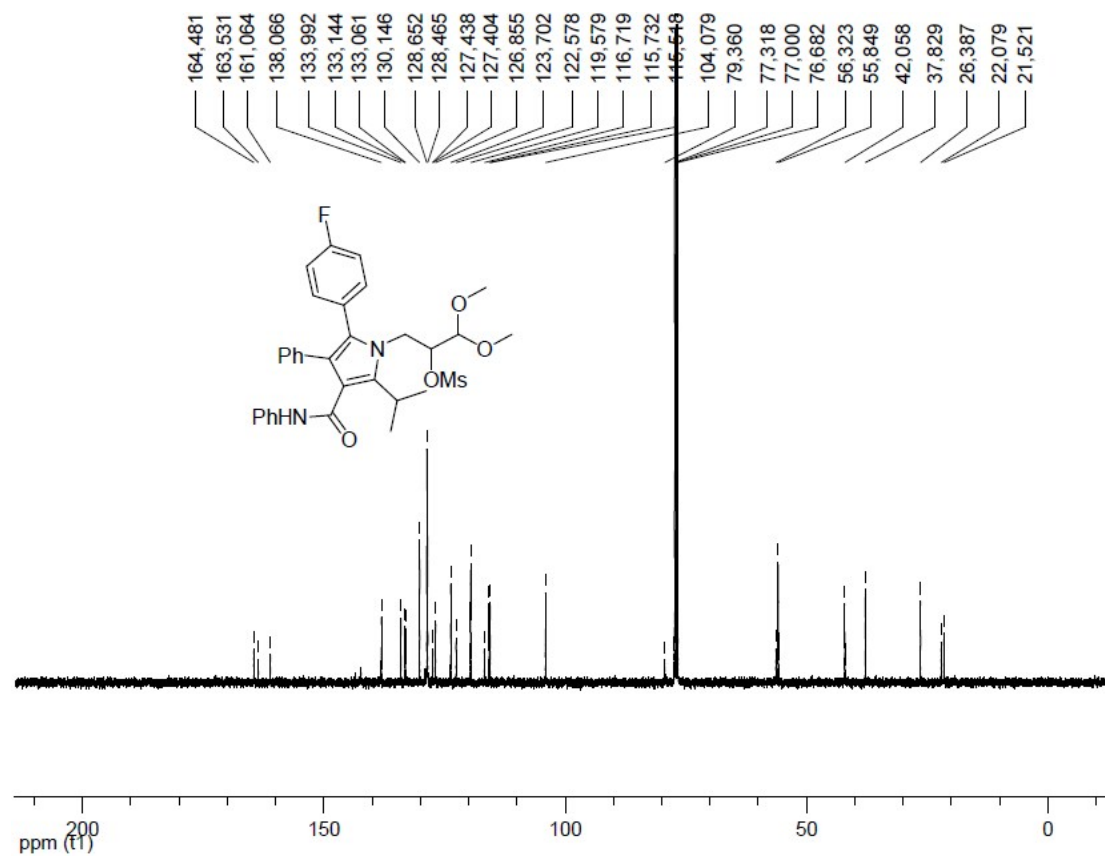
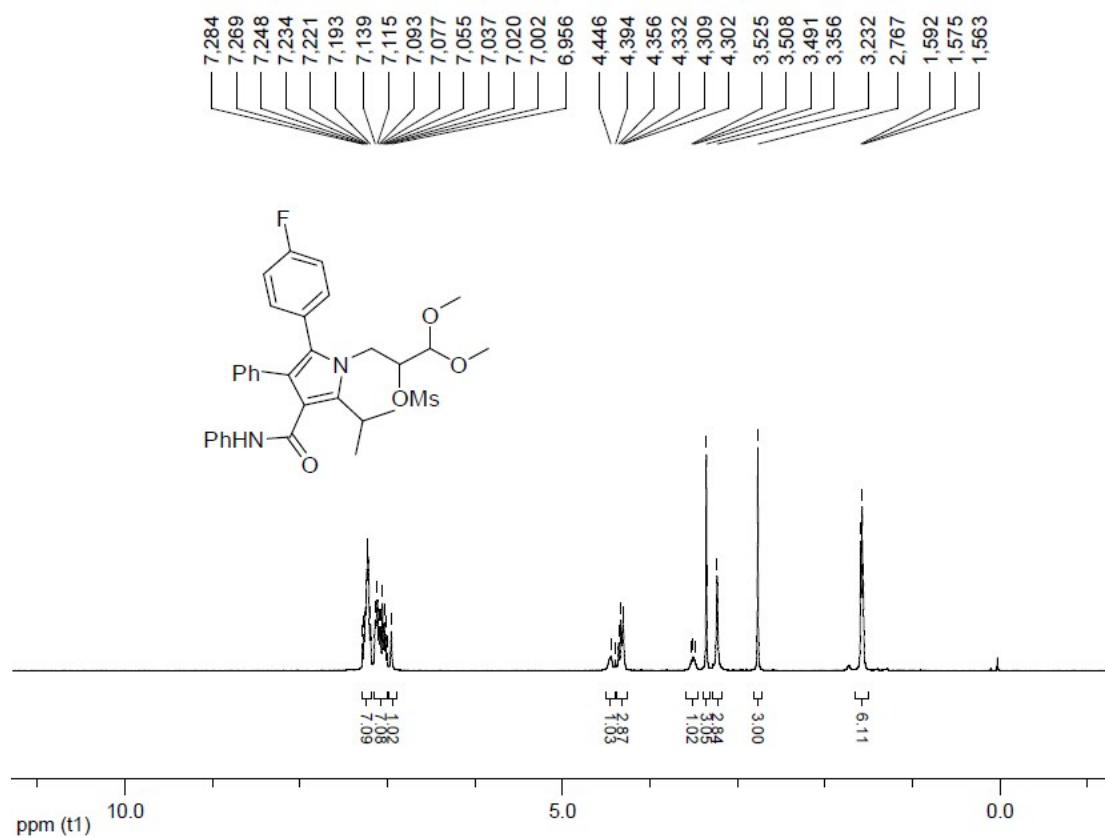


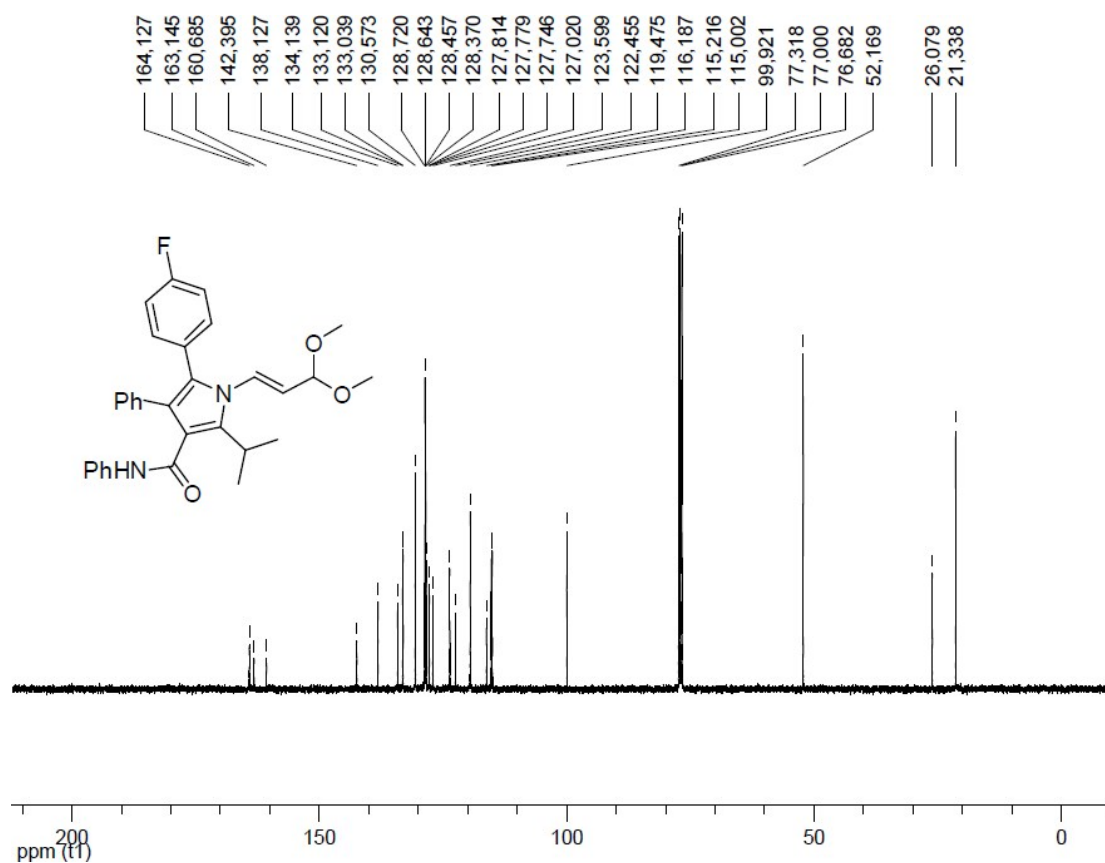
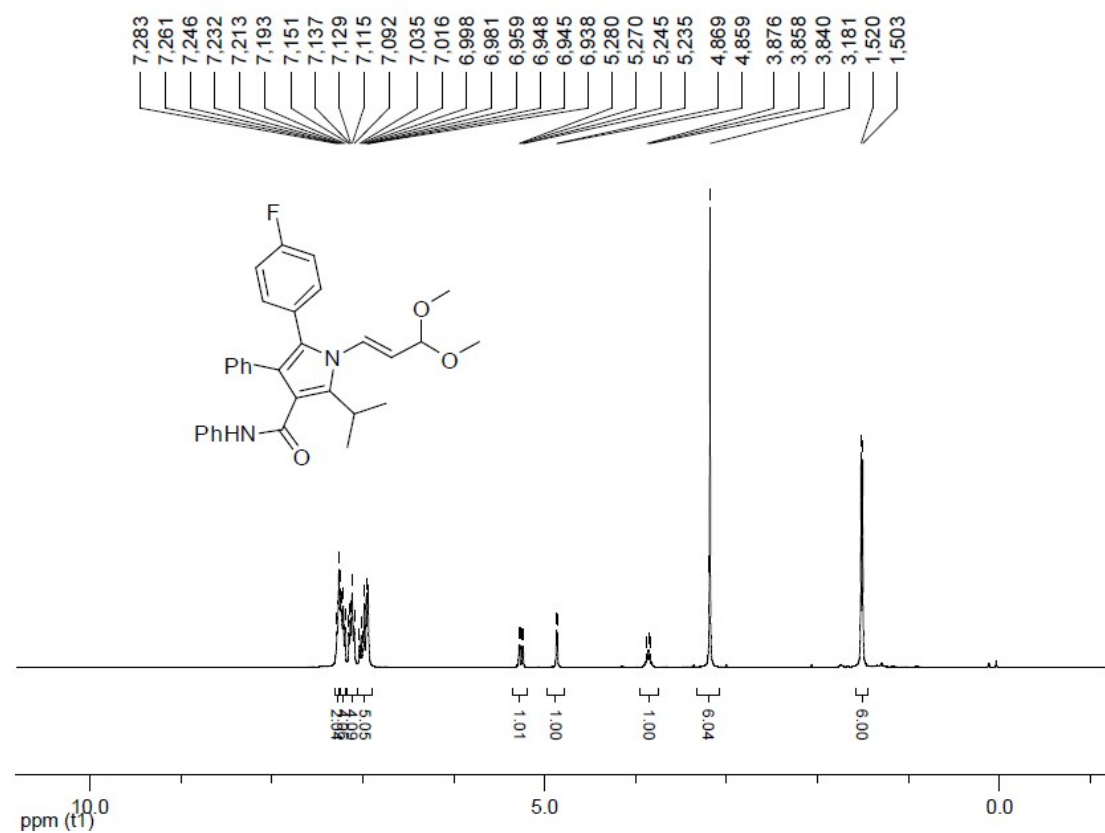
Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	40.467	32621.4	517.6	0.9673	0.74	50.137
2	48.605	32443.2	405.3	1.2378	0.629	49.863

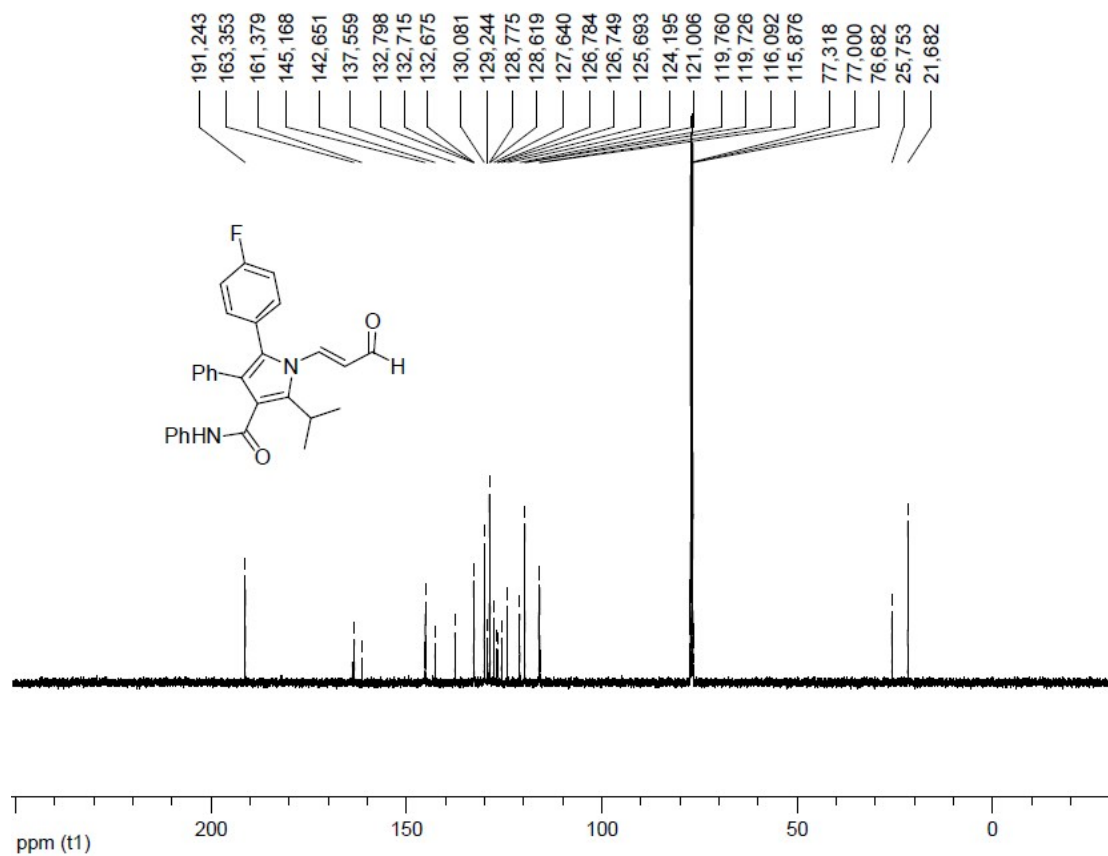
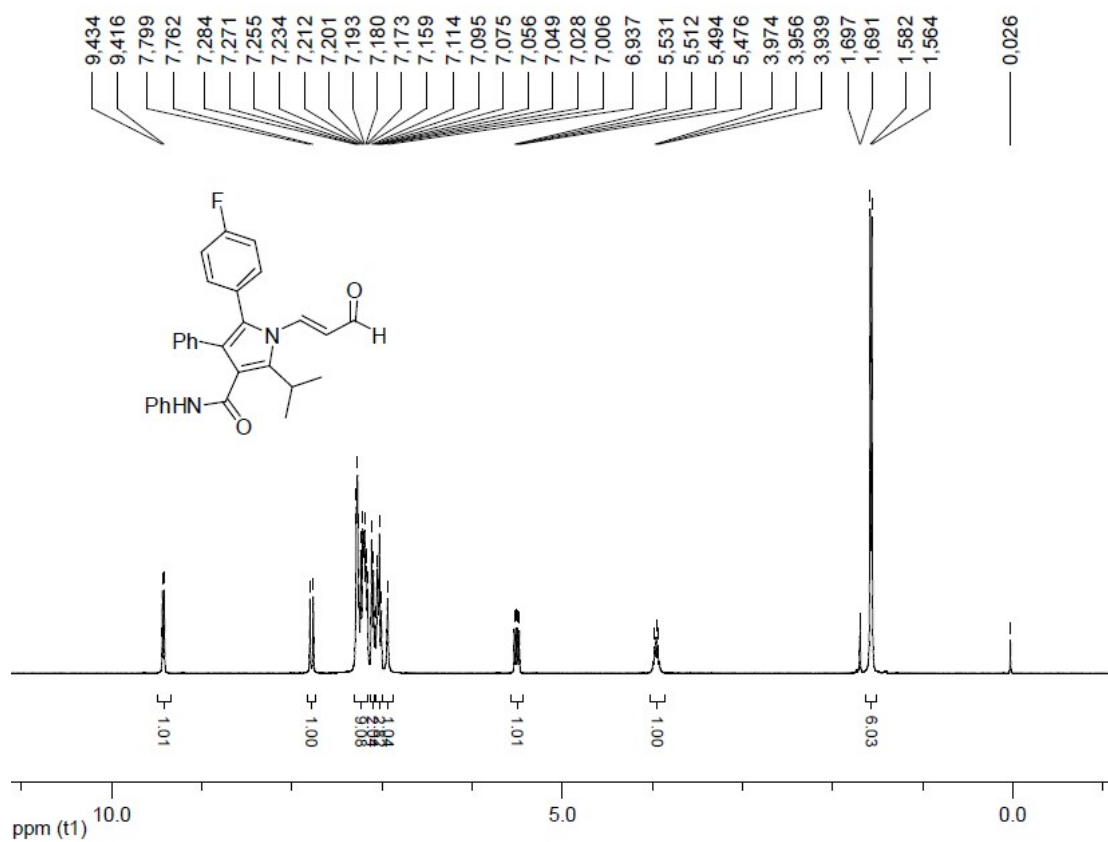


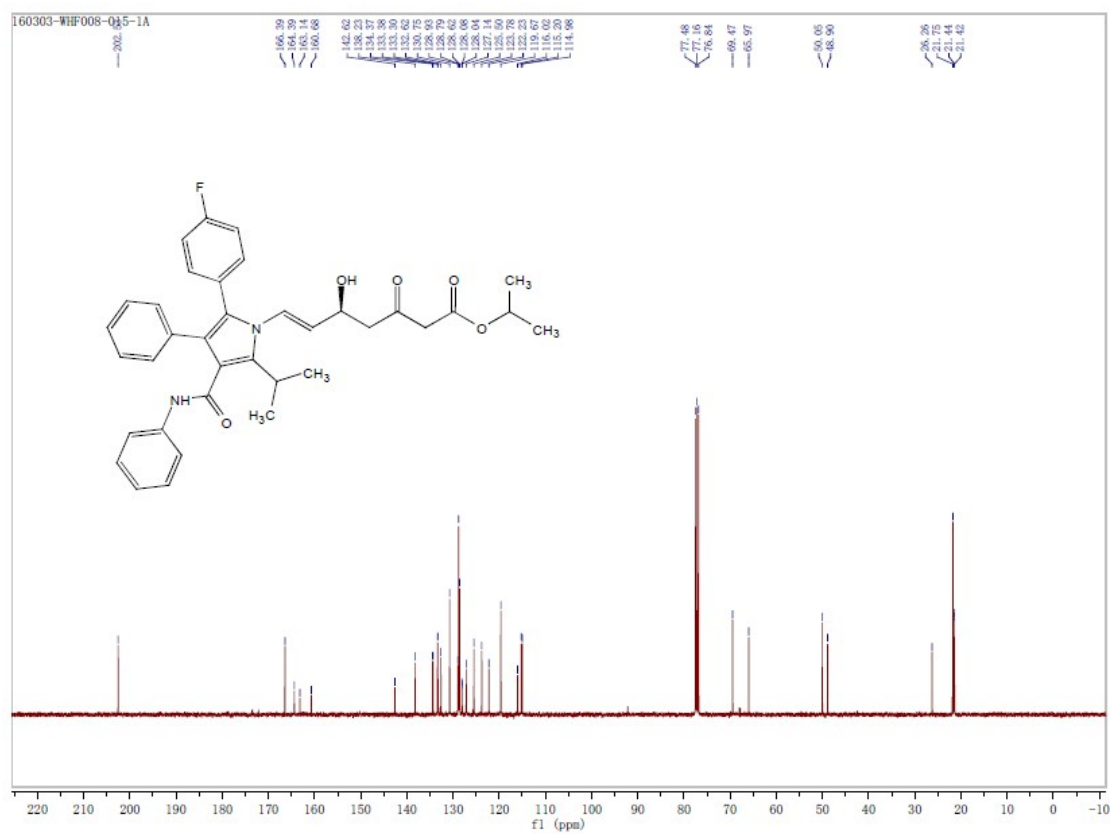
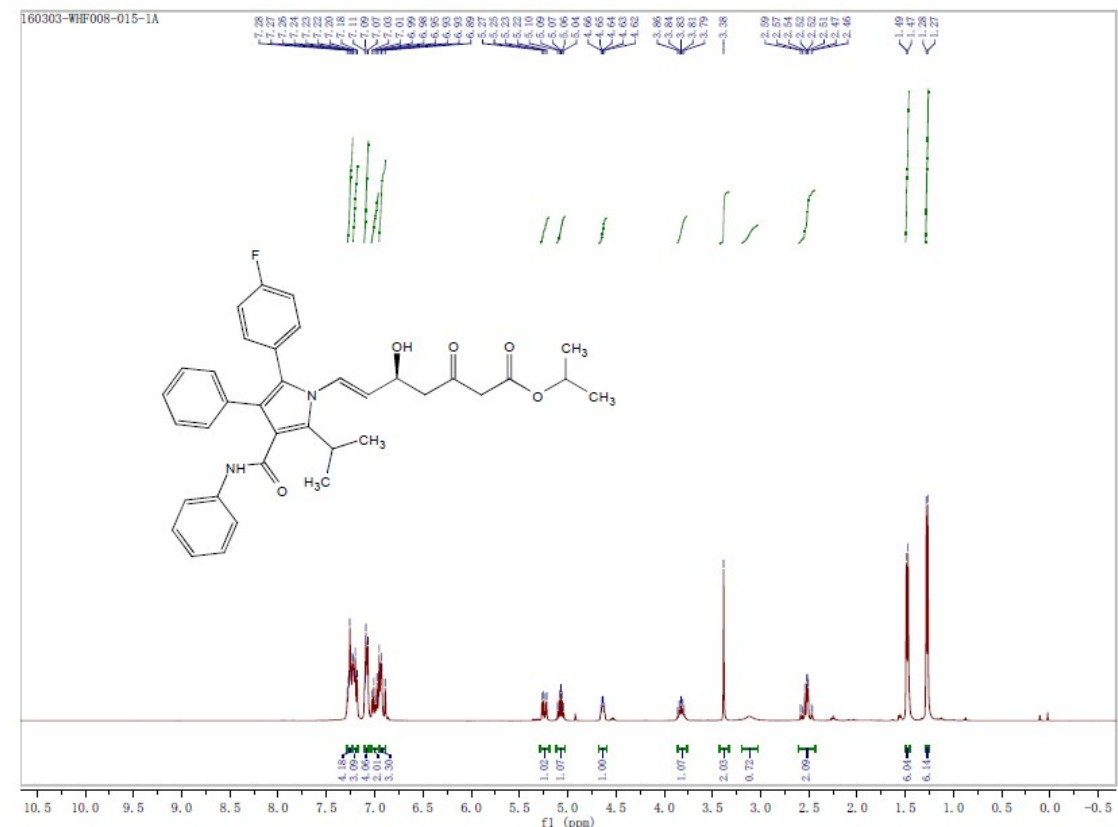
Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	39.255	26536.4	395.4	1.0229	0.691	89.757
2	47.54	3031.4	37.4	1.1942	0.878	10.243

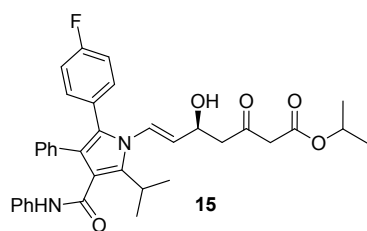




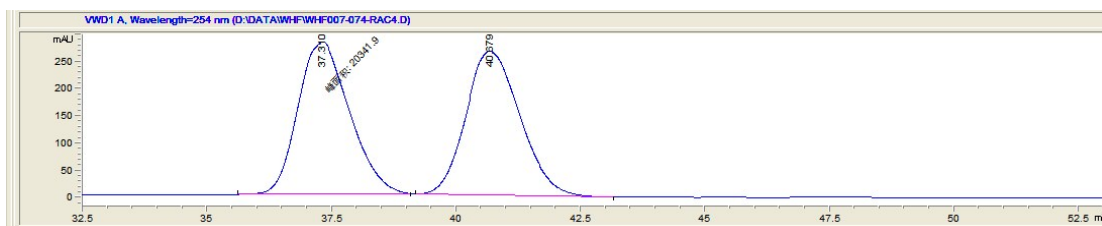




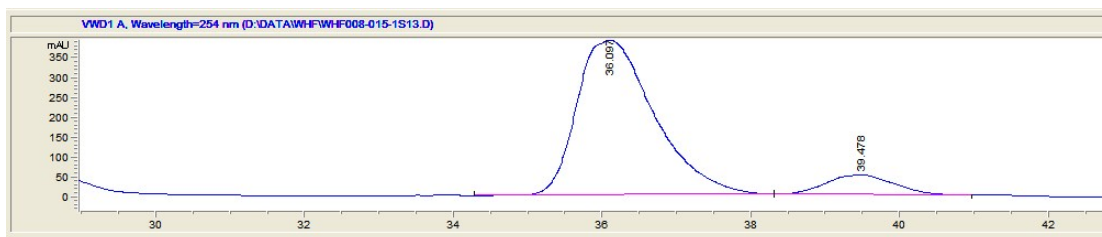




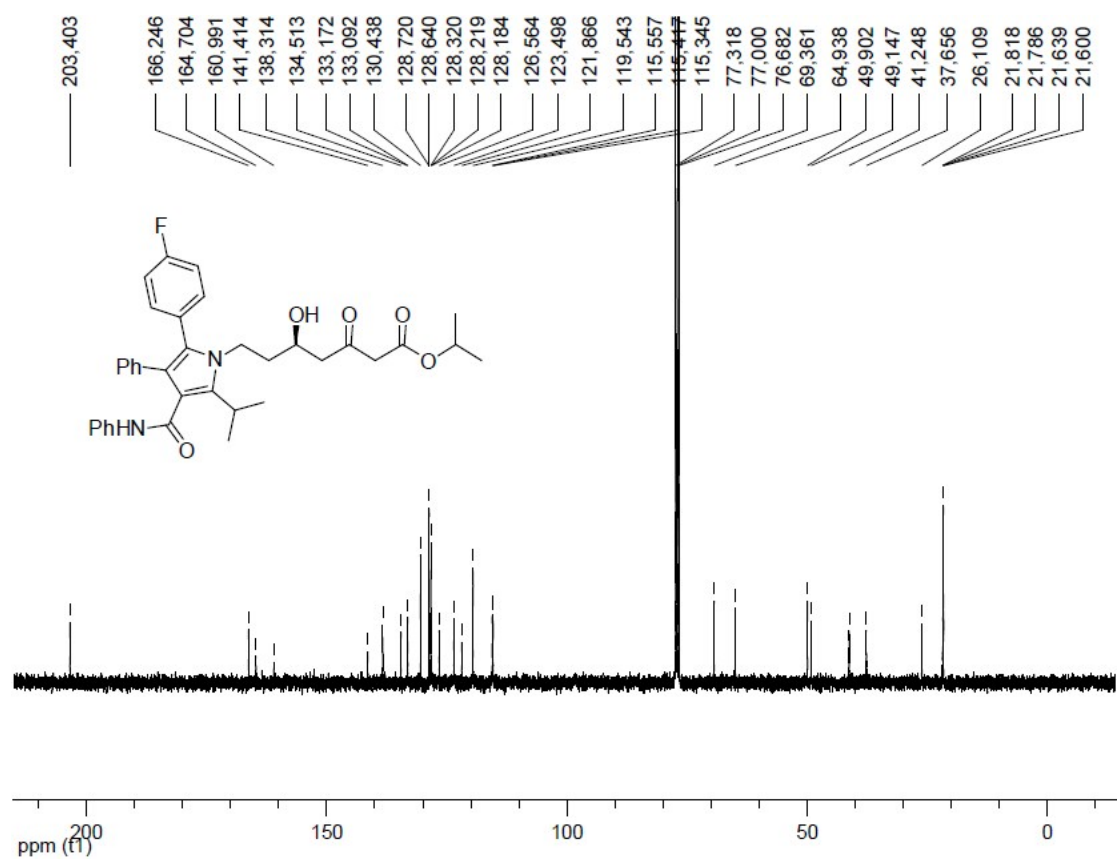
Chiralpak AD-H column (25 cm × 4.6 mm × 5 μm), n-hexane/isopropanol =95/5, 1mL/min, 254 nm, 30°C, tR(major) = 36.097 min, tR (minor) = 39.478 min

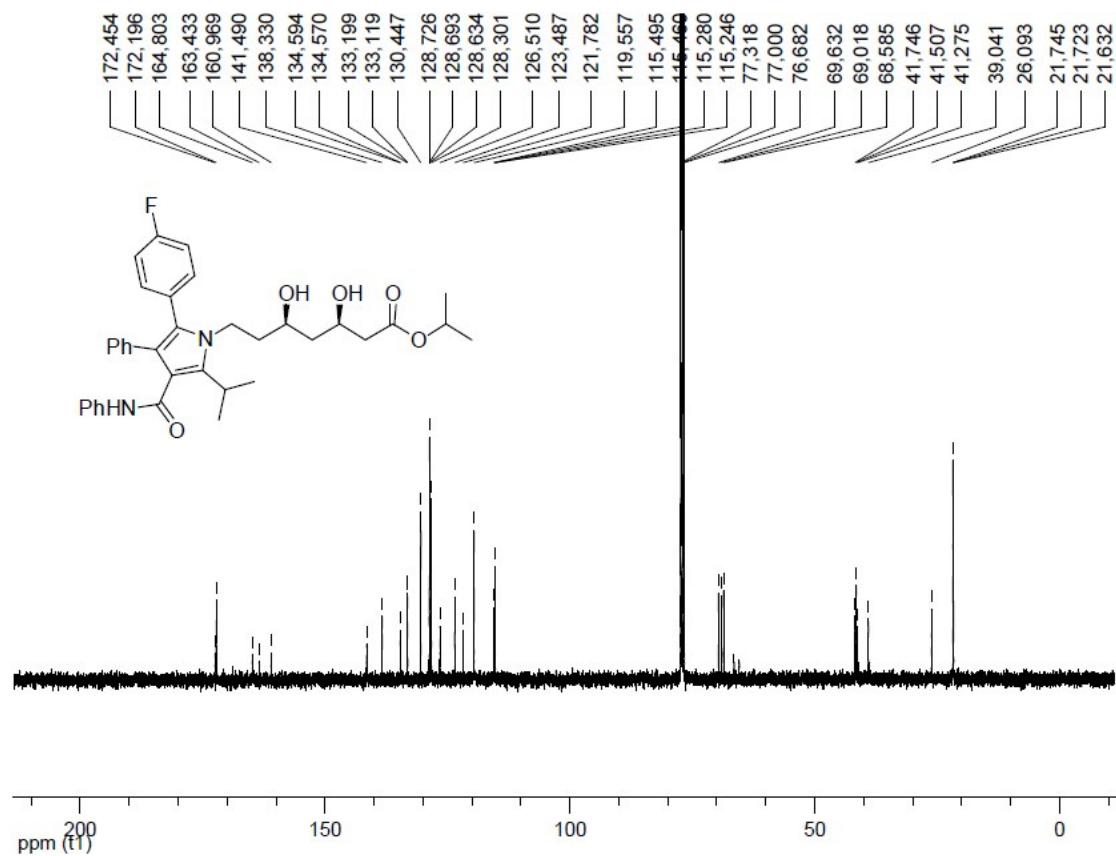


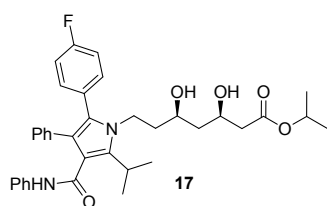
Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	37.31	20341.9	280.8	1.2075	0.872	50.514
2	40.679	19928	264.4	1.1595	0.787	49.486



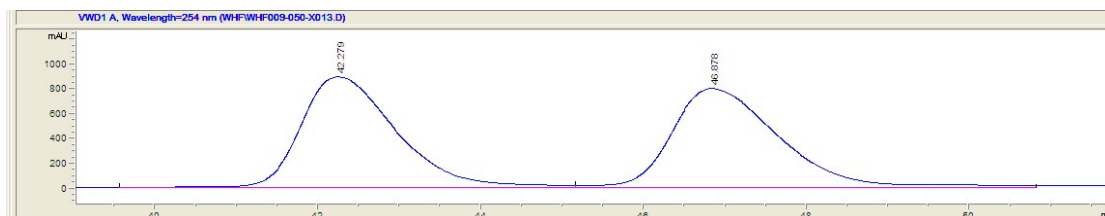
Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	36.097	28160.7	388.9	0.9929	0.704	90.011
2	39.478	3125.2	49.2	0.9116	1.028	9.989



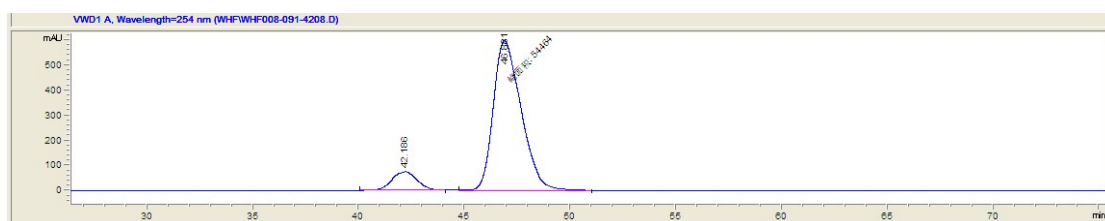
[illegible]



Chiralpak AD-H column (25 cm × 4.6 mm × 5 μm), n-hexane/isopropanol =95/5+0.05%TFA, 0.8mL/min, 254 nm, 30°C, t_R(major) = 46.881 min, t_R (minor) = 42.186 min



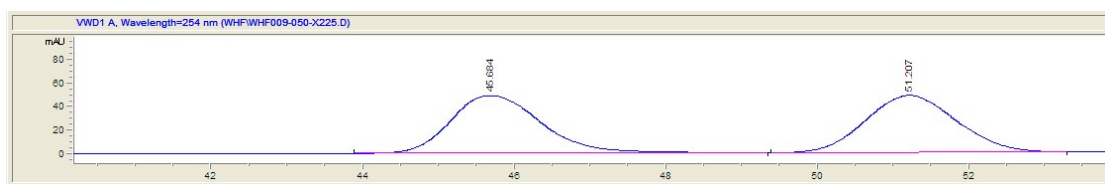
Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	42.279	72910.6	886.5	1.2475	0.664	49.753
2	46.878	73635	793.3	1.4669	0.63	50.247



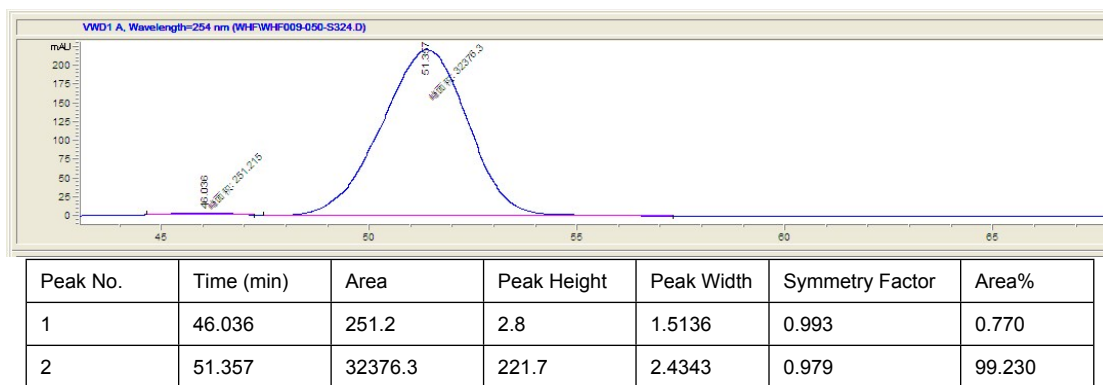
Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	42.186	6035.3	73.6	1.2757	1.03	9.976
2	46.881	54464	597.9	1.5181	0.644	90.024

The enantioselectivity of **17** (80% ee) was upgraded to 98% ee by its recrystallization from CH₃CN/H₂O (5:1, v/v).

Chiralpak AD-H column (25 cm × 4.6 mm × 5 μm), n-hexane/isopropanol =95/5, 0.8mL/min, 254 nm, 30°C, t_R(major) = 51.357 min, t_R (minor) = 46.036 min



Peak No.	Time (min)	Area	Peak Height	Peak Width	Symmetry Factor	Area%
1	46.034	4060	48.7	1.292	0.75	50.296
2	51.207	4012.1	48.4	1.304	0.893	49.704



The synconfiguration of **17** was confirmed via NOESY NMR spectroscopy of ketal **17a** obtained from **17** by treatment of 2,2-dimethoxypropane in the presence of a catalytic amount of PPTS according to a procedure from the literature.

17a

