Supporting Information

An Enantioselective Assembly of Naphthopyran via NHC-Catalyzed [3+3] Annulation of Bromoenal with β-Tetralone

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†Electronic Supplementary Information (ESI) available: ^{1}H and ^{13}C NMR spectra. See DOI: 10.1039/x0xx00000x

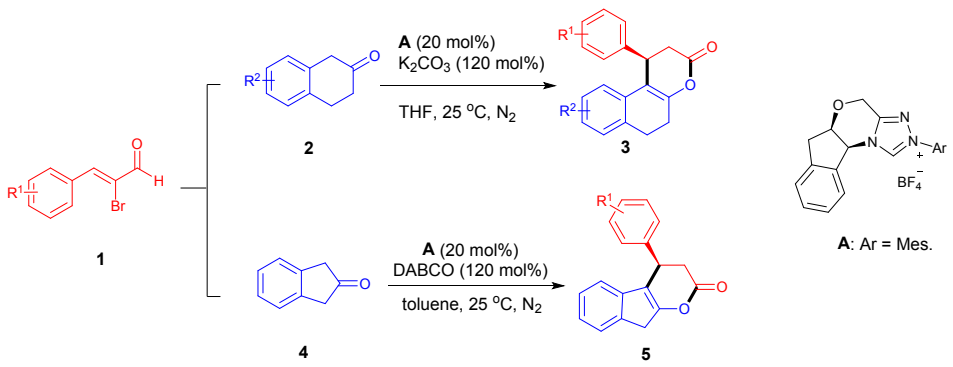
‡These authors contributed equally to the work.

Content

General methods

Common reagents and materials were purchased from commercial sources and purified by recrystallization or distillation. Melting points were determined in open capillaries and were uncorrected. IR spectra were taken on a FT-IR-Tensor 27 spectrometer in KBr pellets and reported in cm^{-1}. ^1H NMR spectra were measured on a Bruker DPX 400 MHz spectrometer in $CDCl_3$ with chemical shift (δ) given in ppm relative to TMS as internal standard. High resolution mass spectra (HRMS) were obtained on a micrOTOF-Q Ⅱ HRMS/MS instrument (Bruker) with the technique of electrospray ionization. Optical rotation values were measured with instruments operating at λ = 589 nm, corresponding to the sodium D line at the temperatures indicated.

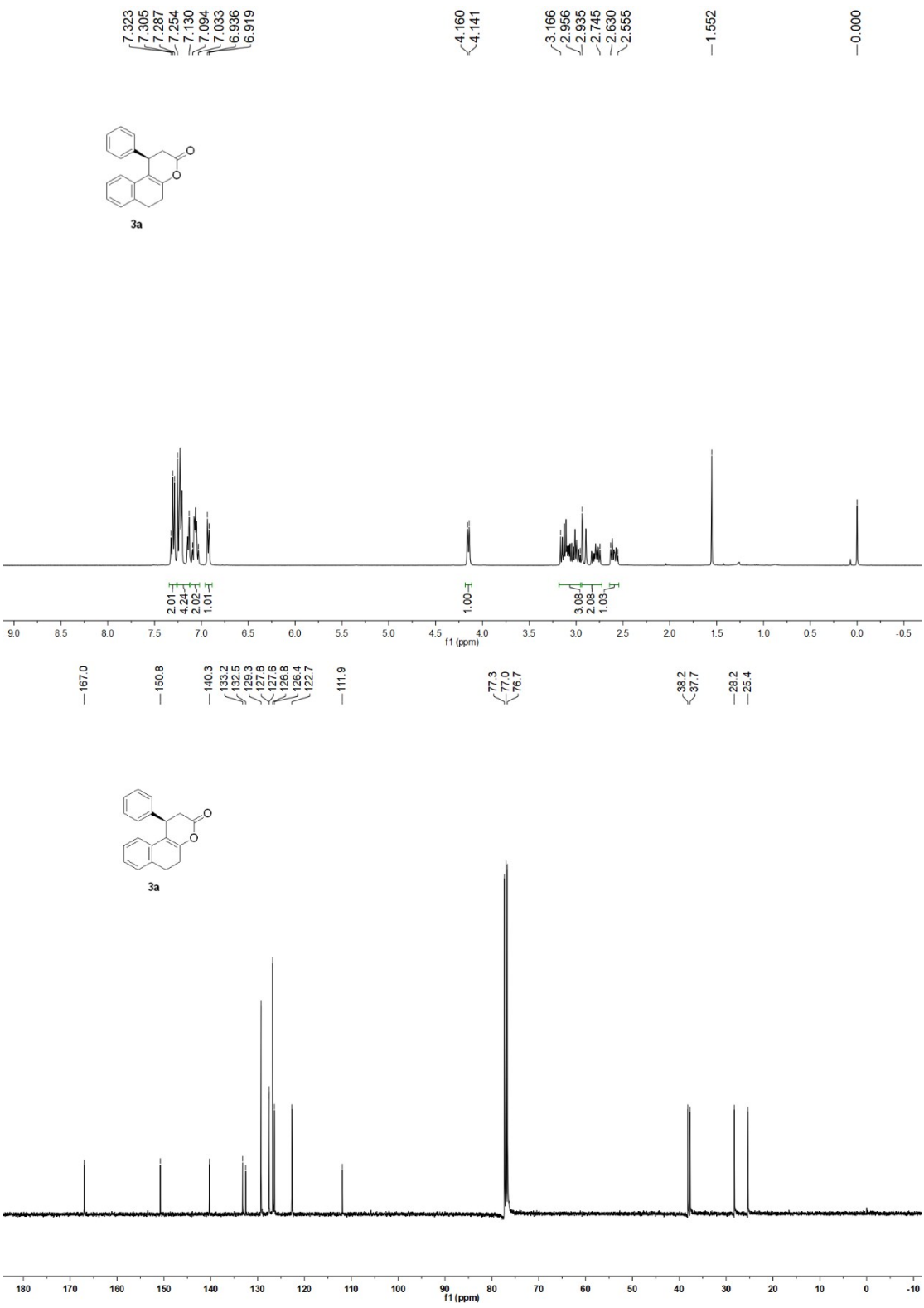
Experimental section

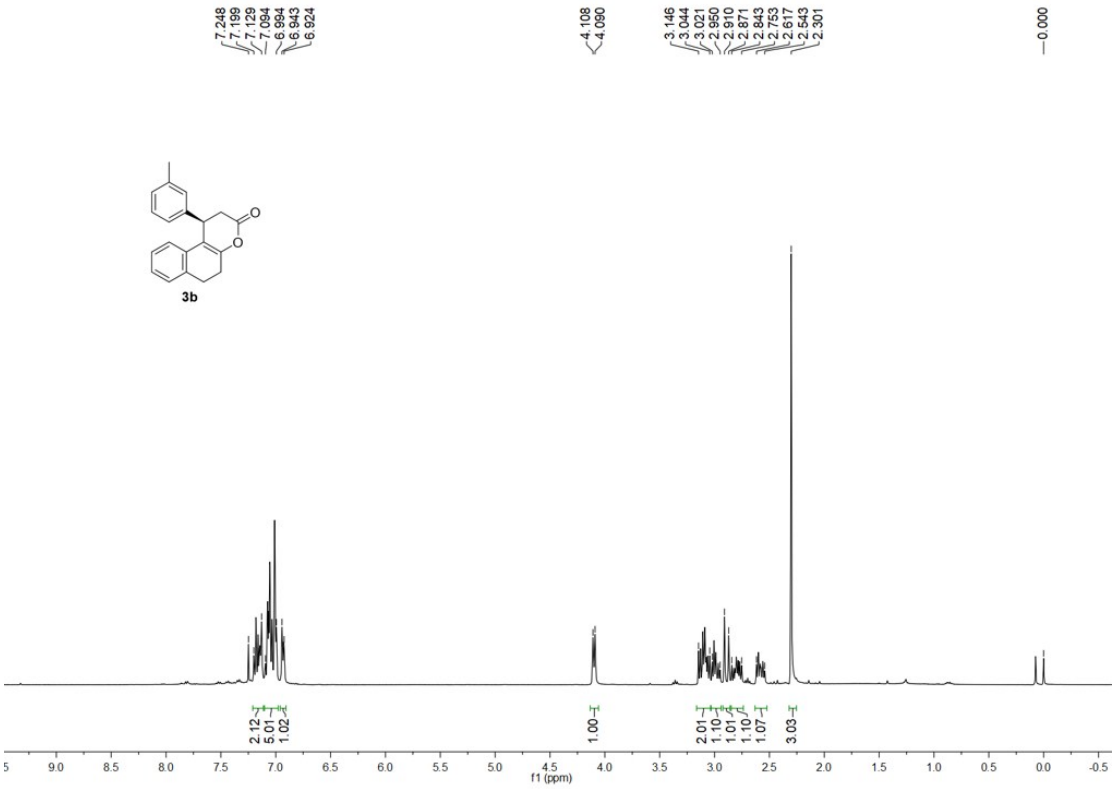


An oven-dried 10 mL Schlenk tube equipped with a magnetic stir bar was charged with triazolium salt **A** (16.8 mg, 0.04 mmol), K_2CO_3 (33.1 mg, 0.24 mmol) or DABCO (26.9 mg, 0.24 mmol), bromoenal **1** (0.30 mmol) and β-tetralone **2** (0.2 mmol) or β-Indanone **4** (0.2 mmol). Freshly distilled THF (3 mL) or toluene (3 mL) was added into the mixture with a syringe. Then tube was closed with a septum, evacuated, and refilled with nitrogen. The mixture was stirred at 25 °C until completion (monitored by TLC). After removal of the solvent under reduced pressure, the resulted crude residue was

purified by column chromatography (silicagel, mixtures of petroleum ether/ethyl acetate, 9:1-19:1, v/v) to afford the desired product **3** or **5**.

Copies of ^{1}H and ^{13}C NMR spectra of products

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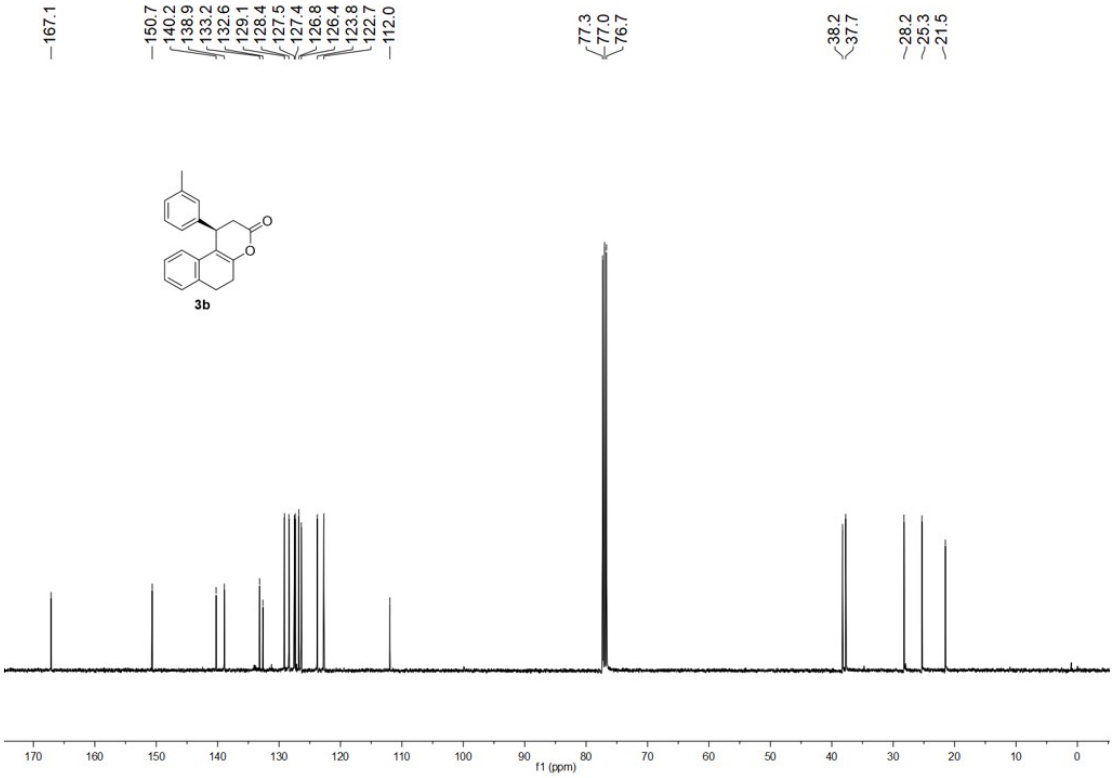
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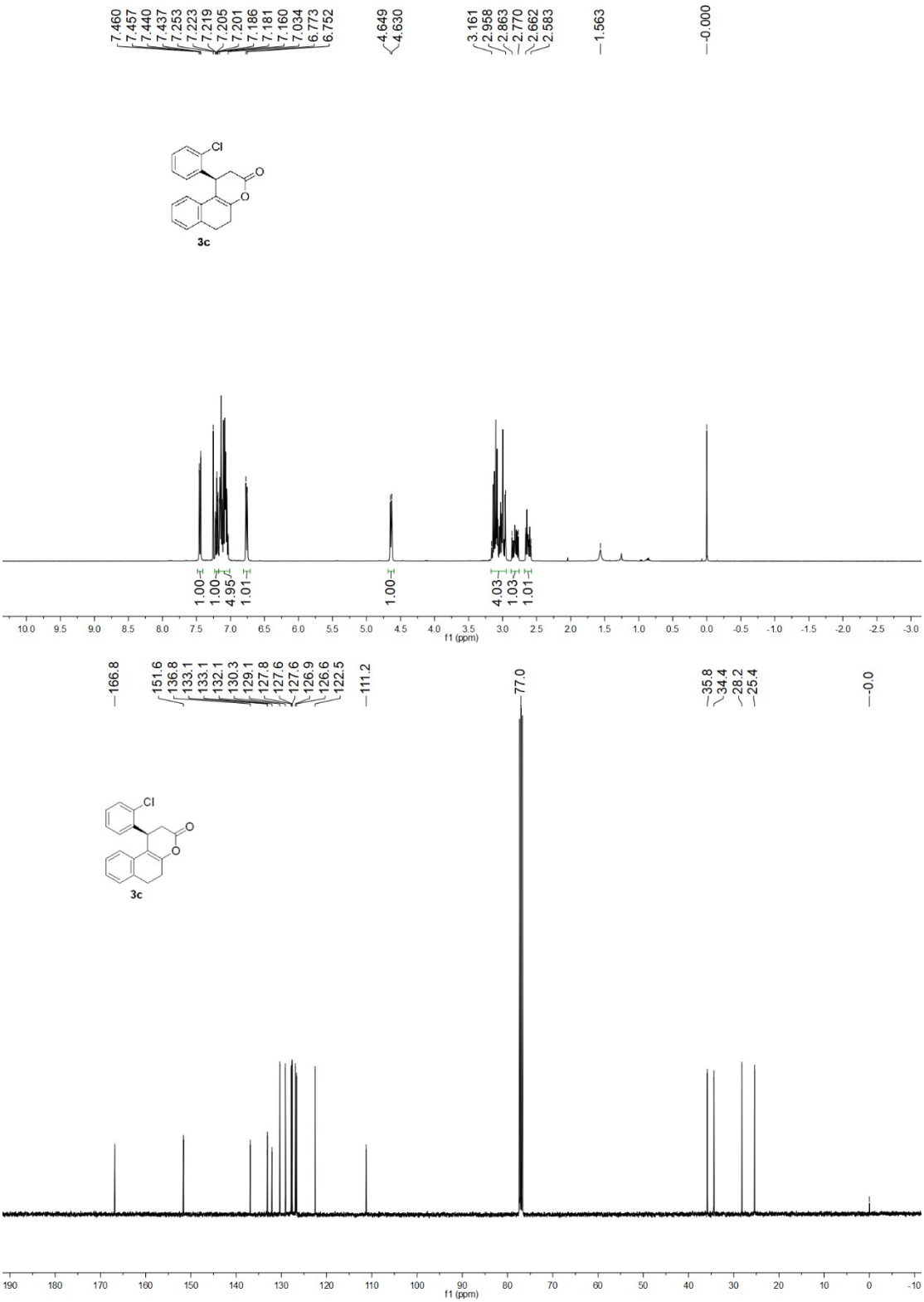
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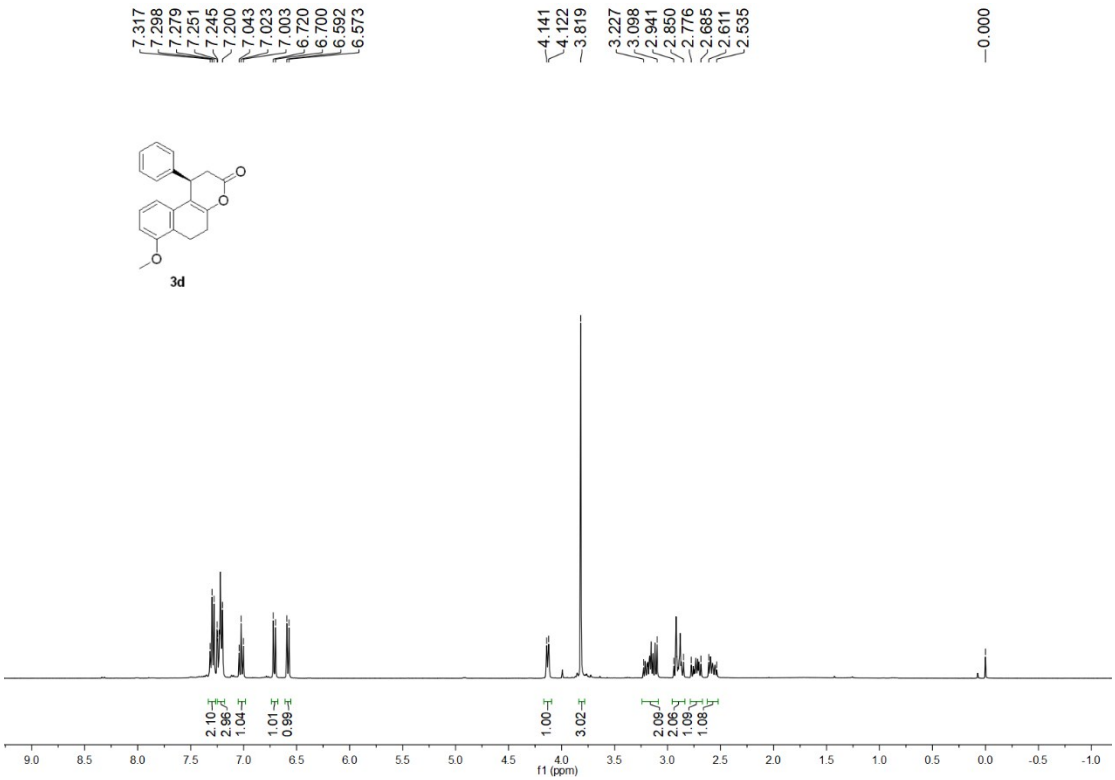
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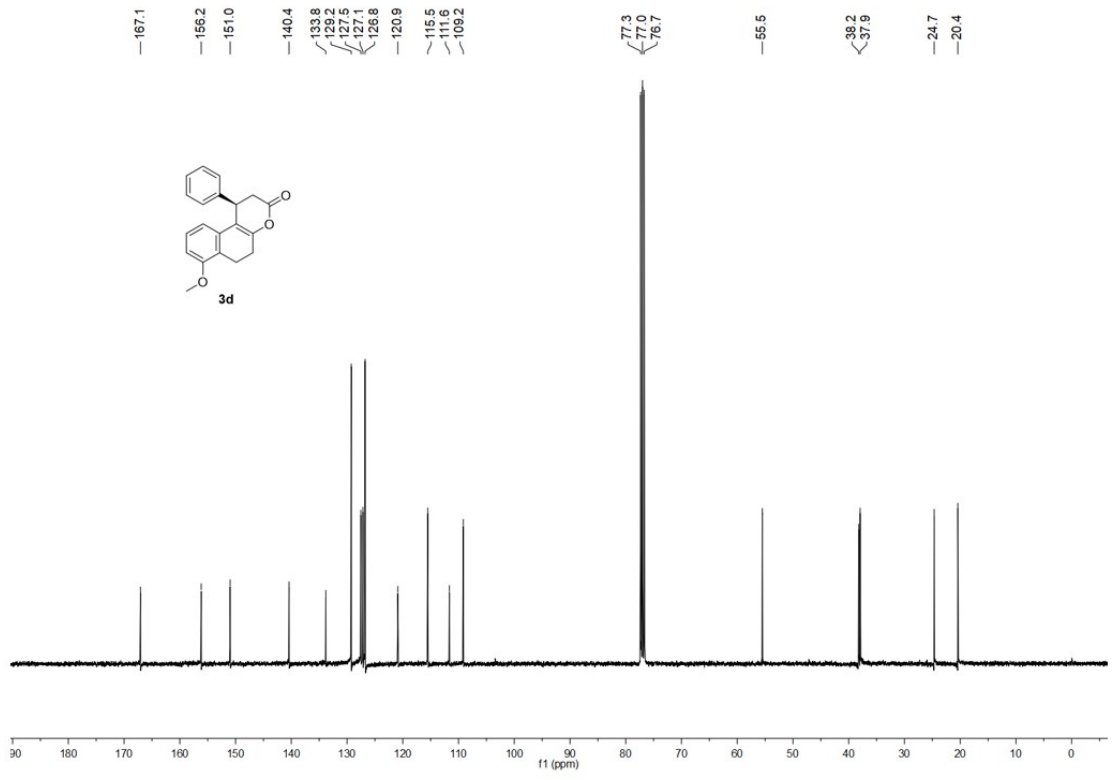
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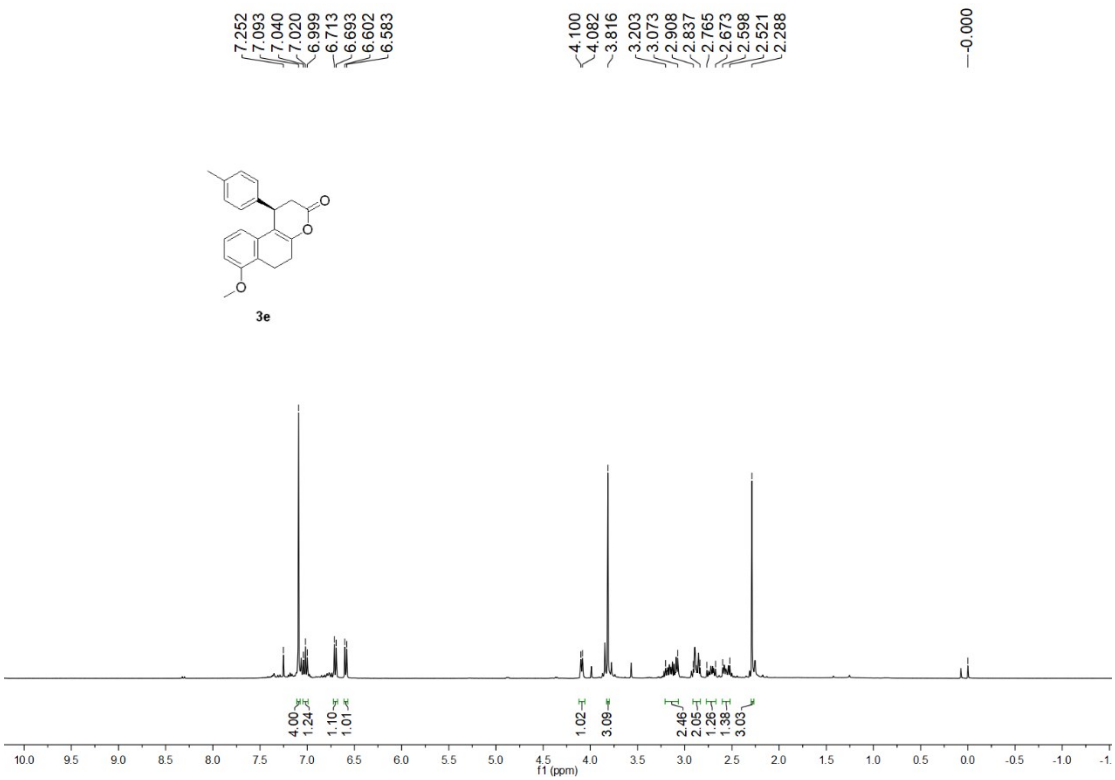
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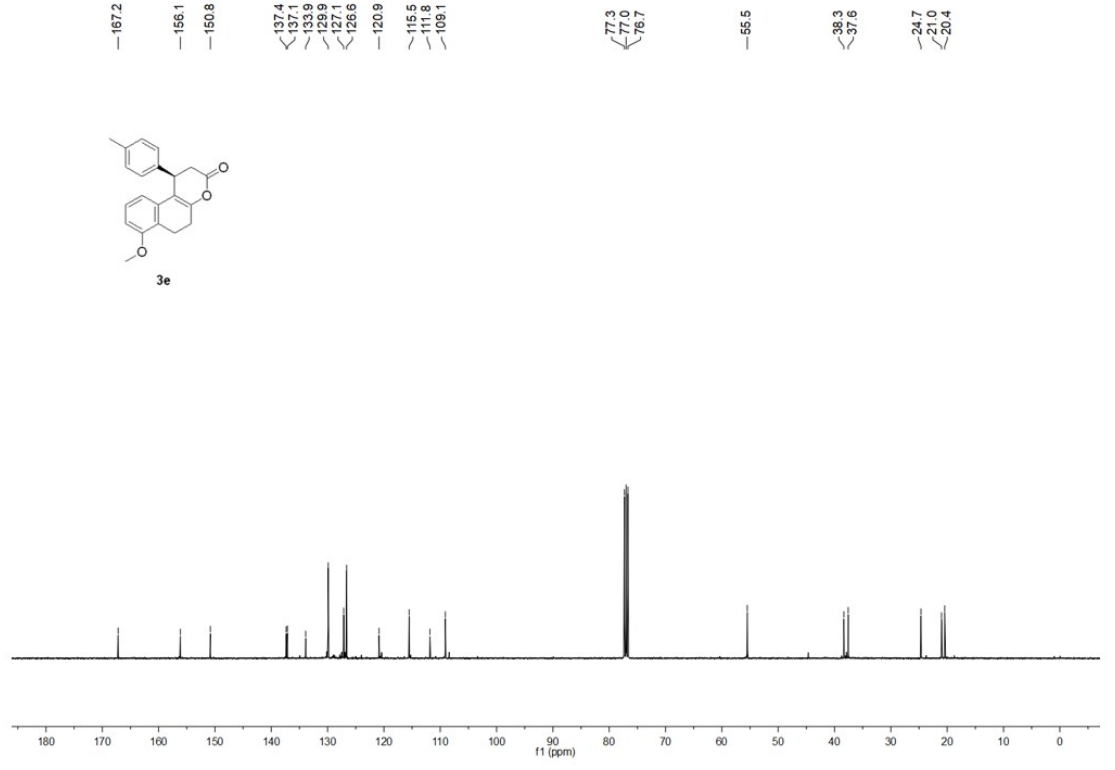
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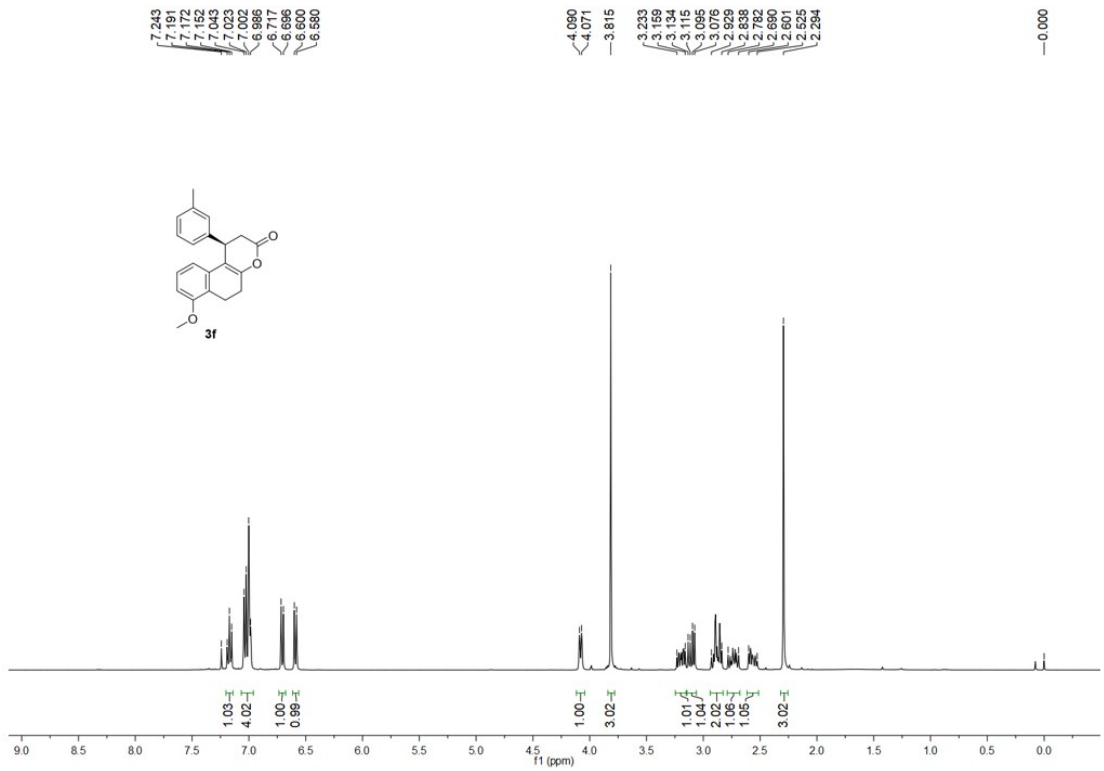
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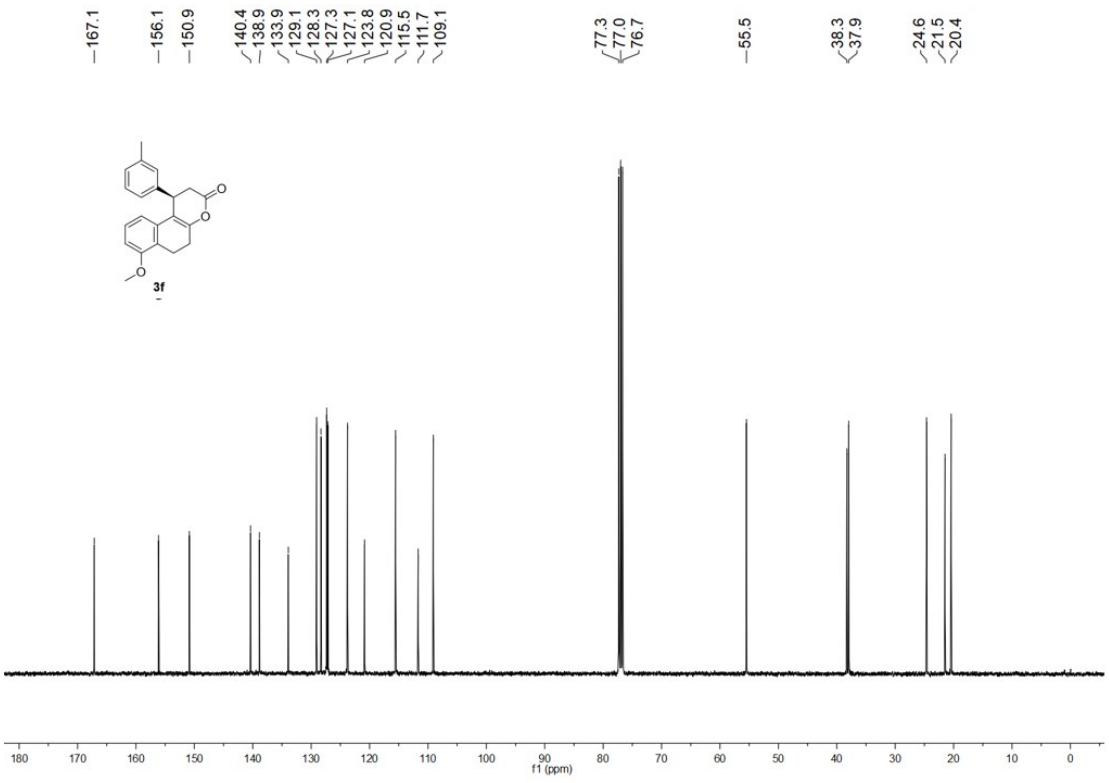
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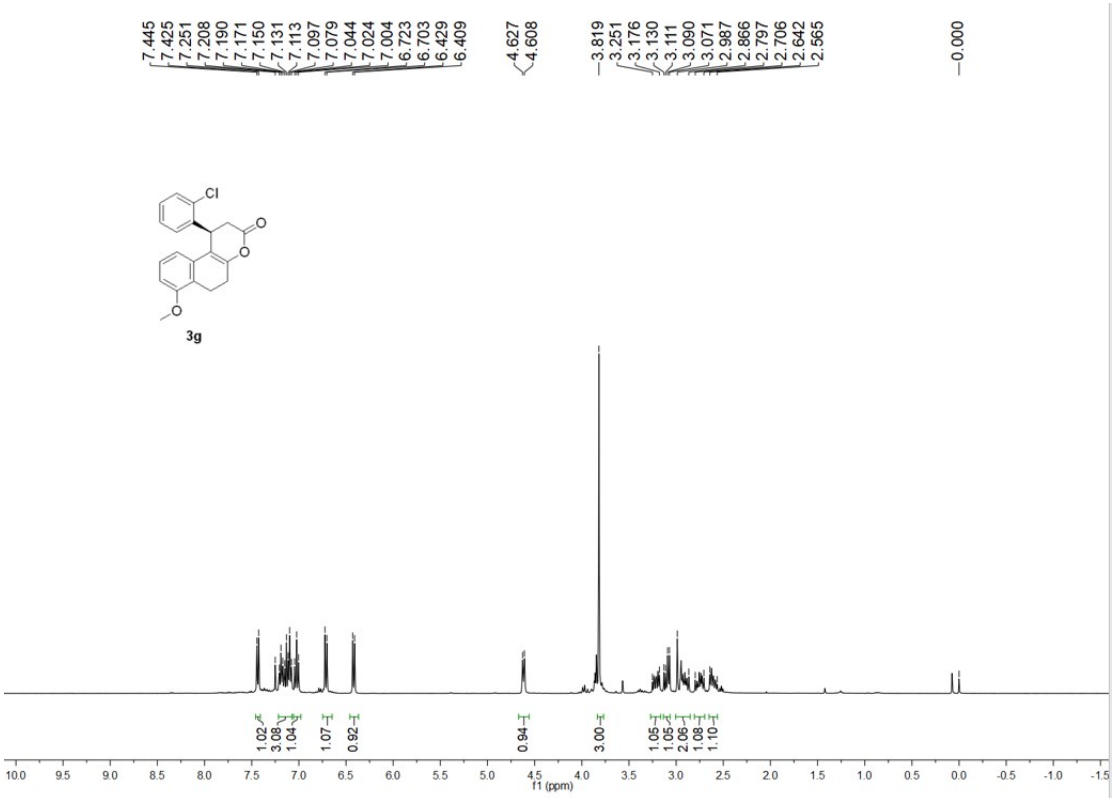
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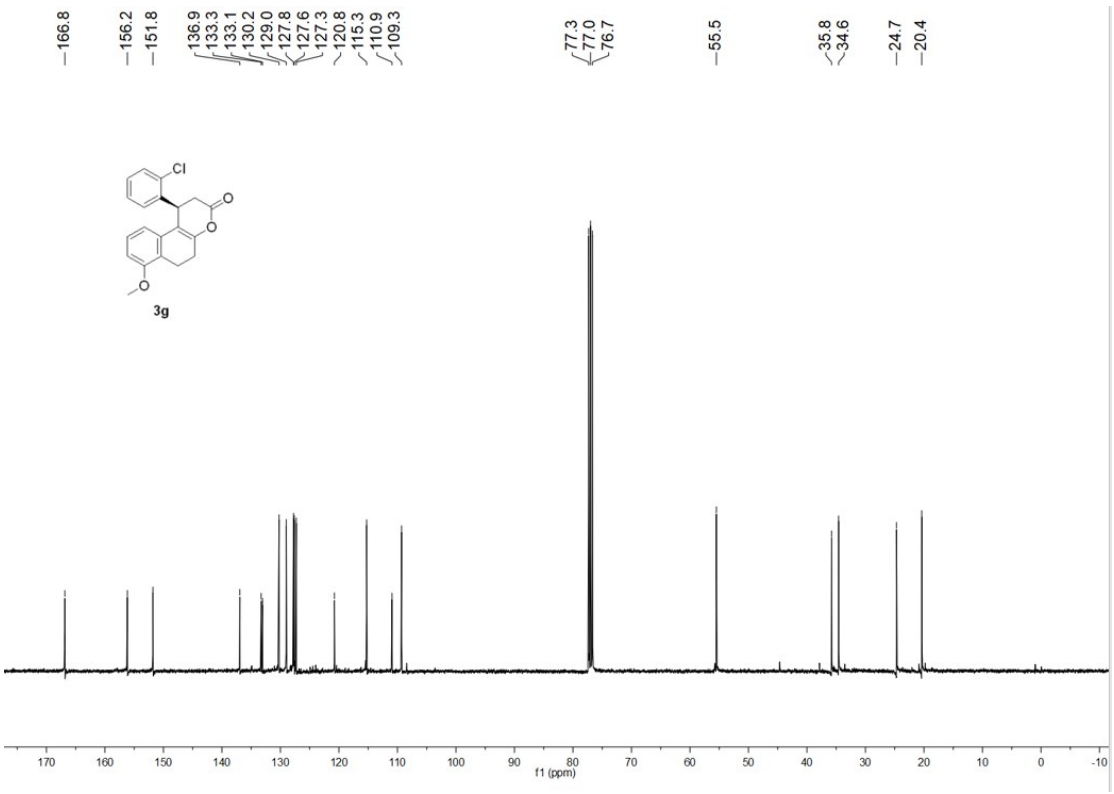
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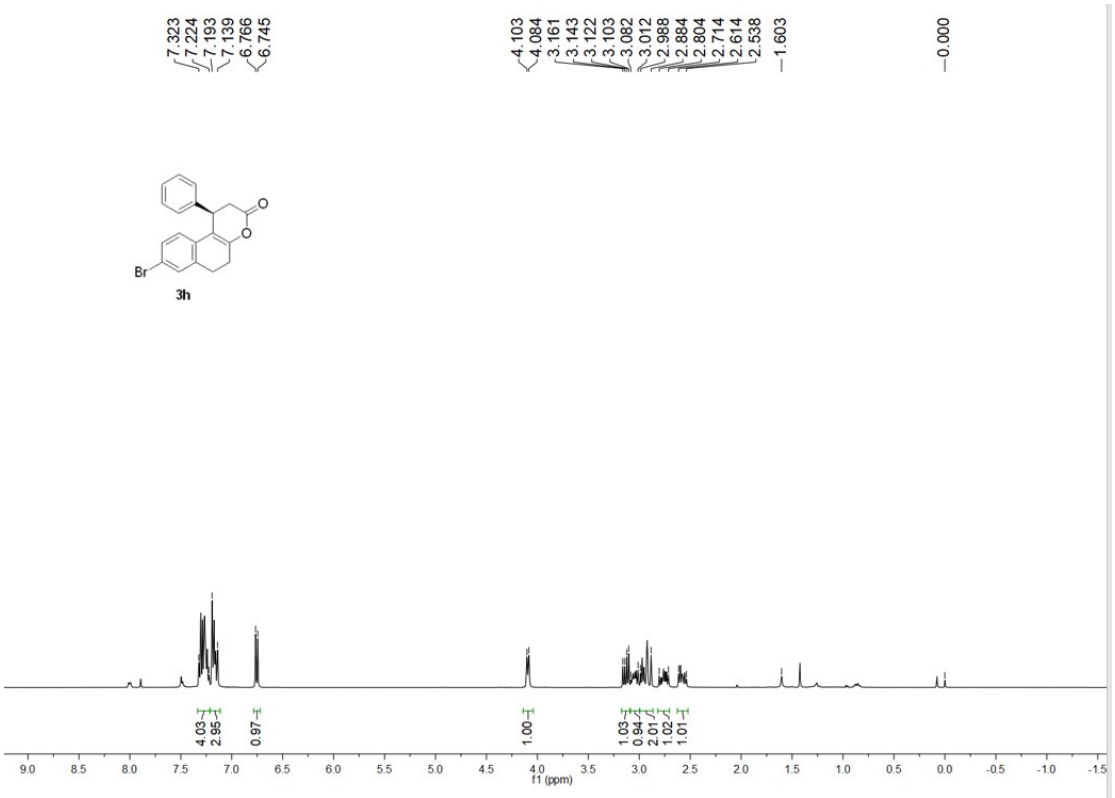
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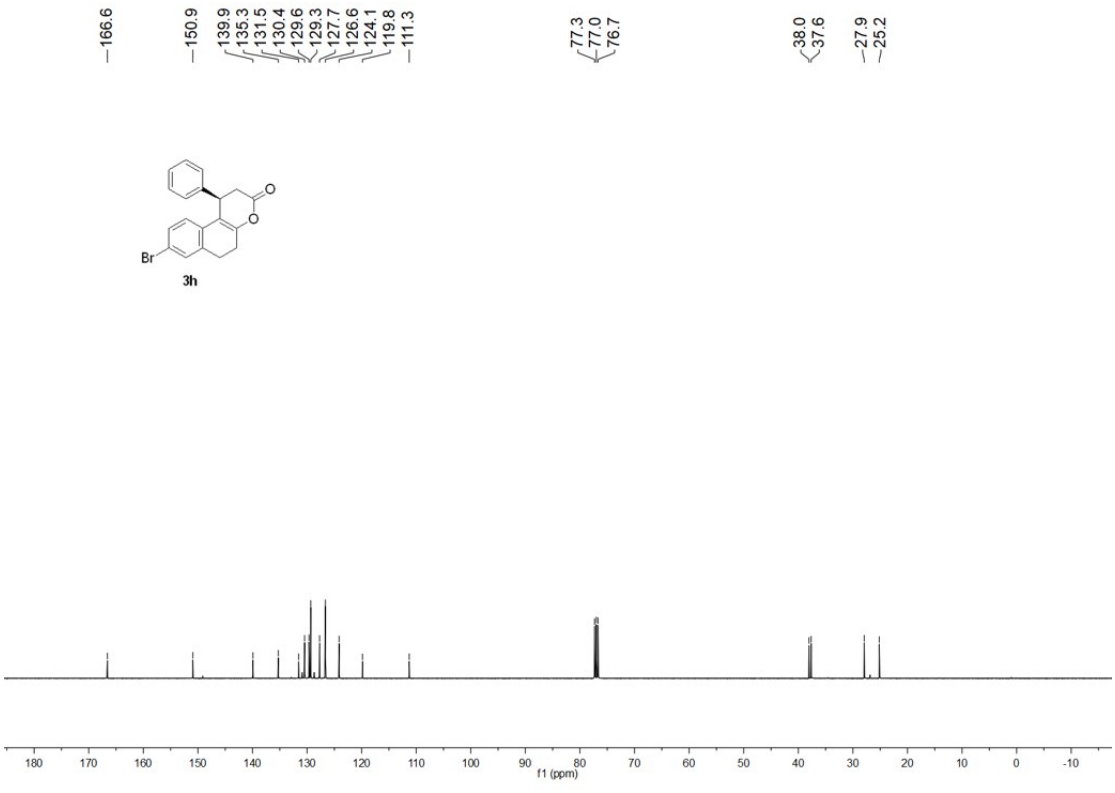
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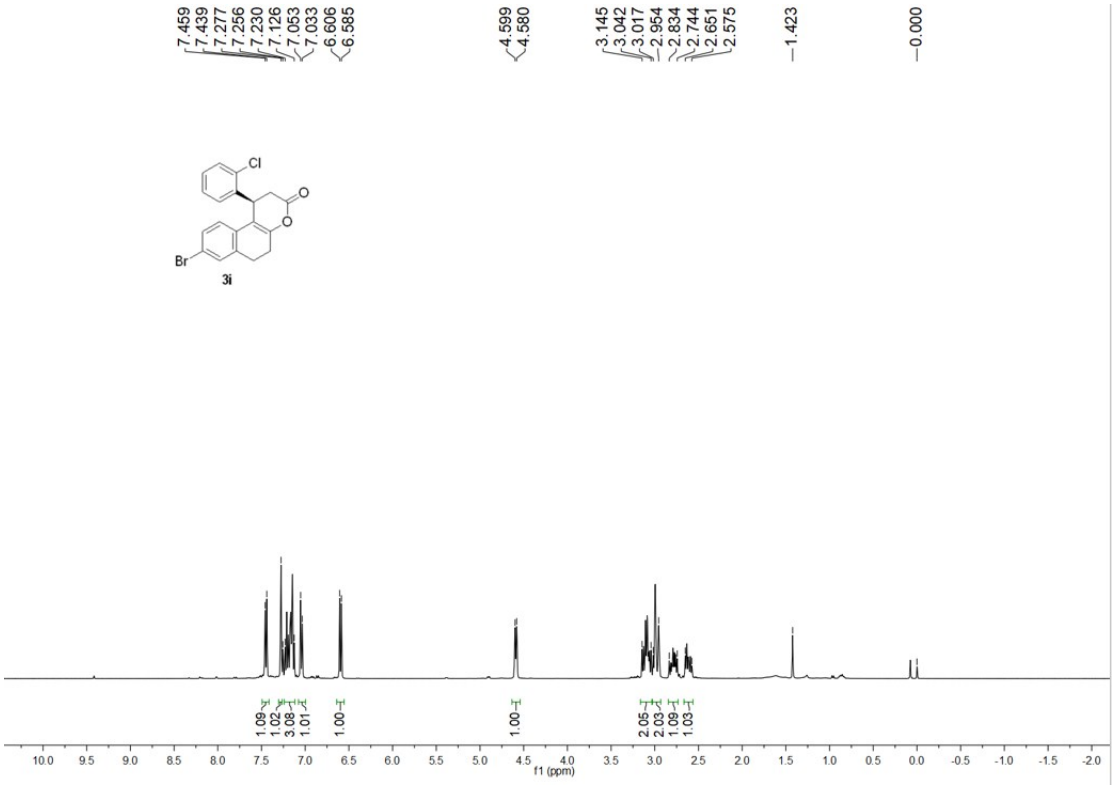
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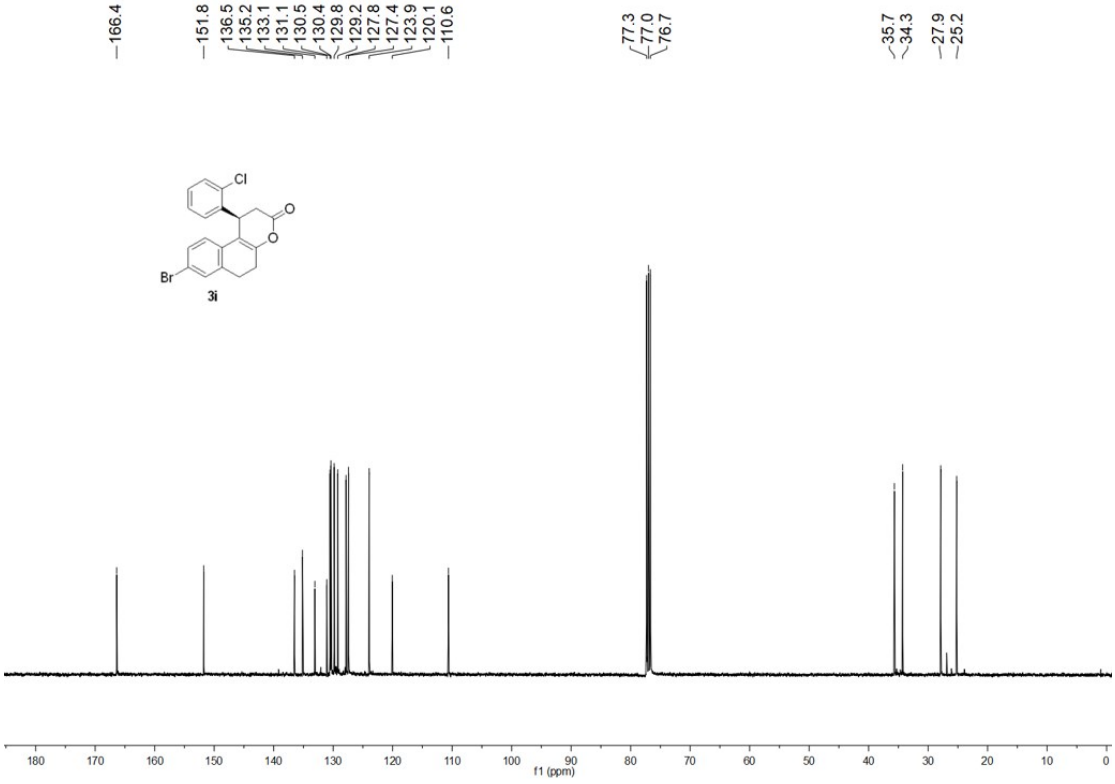
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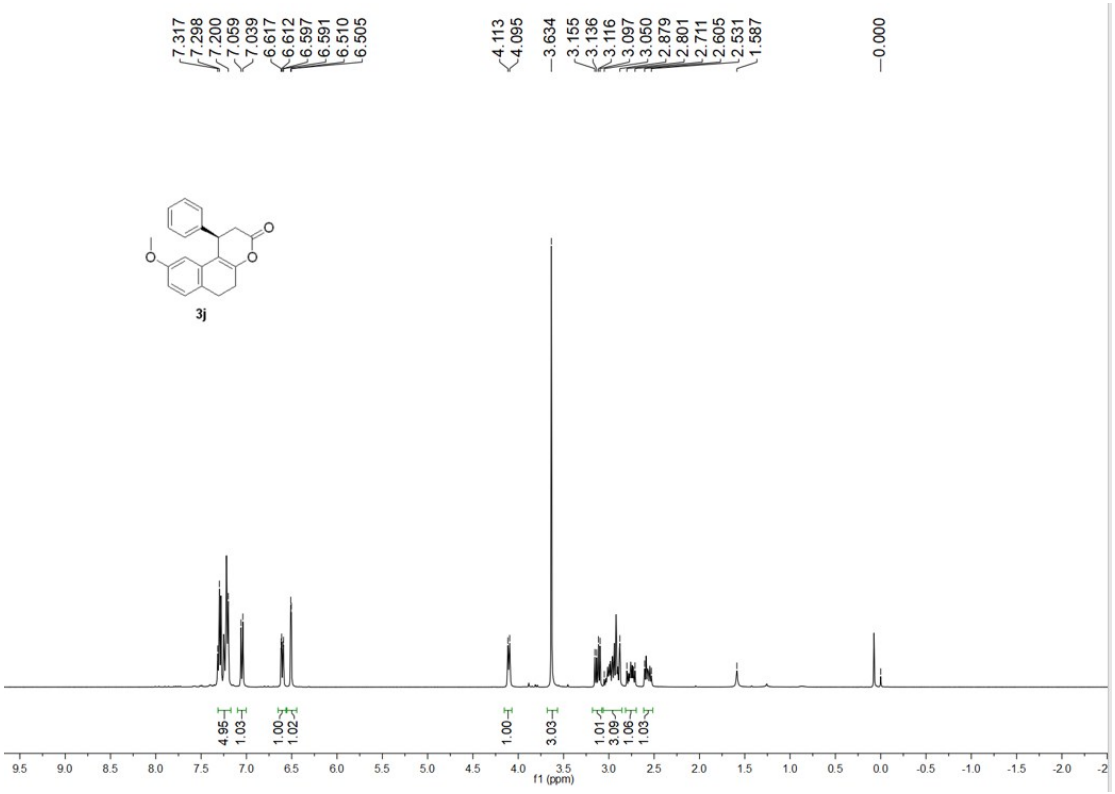
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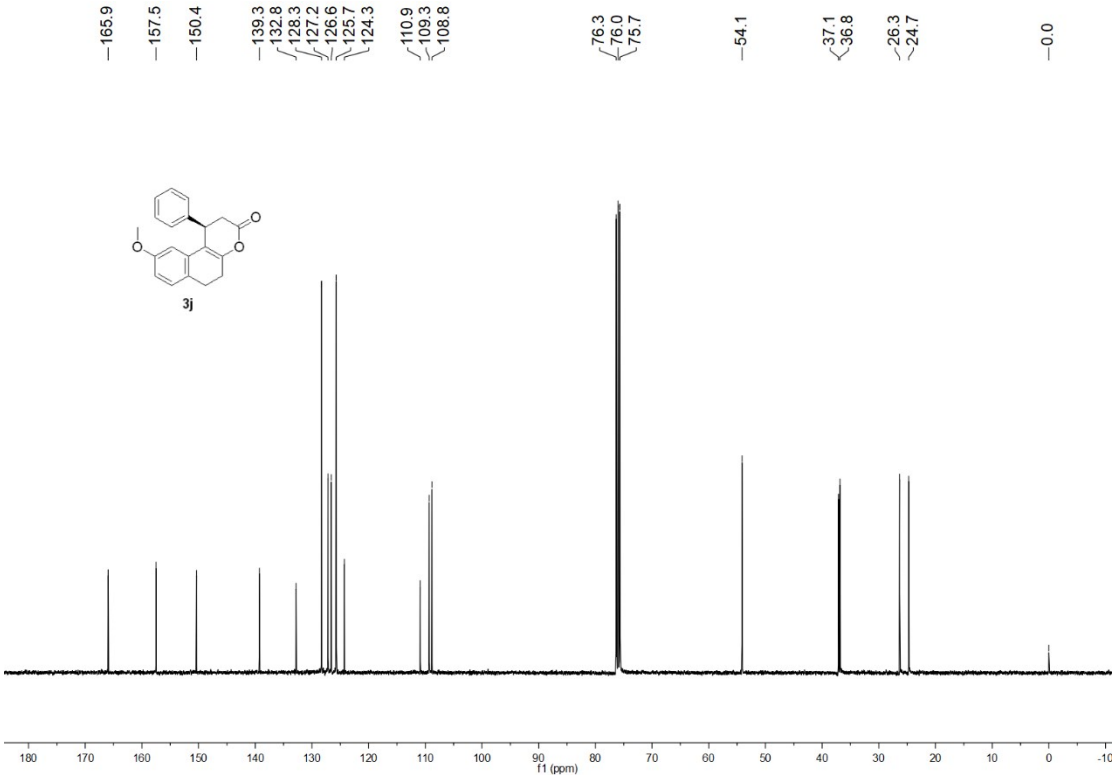
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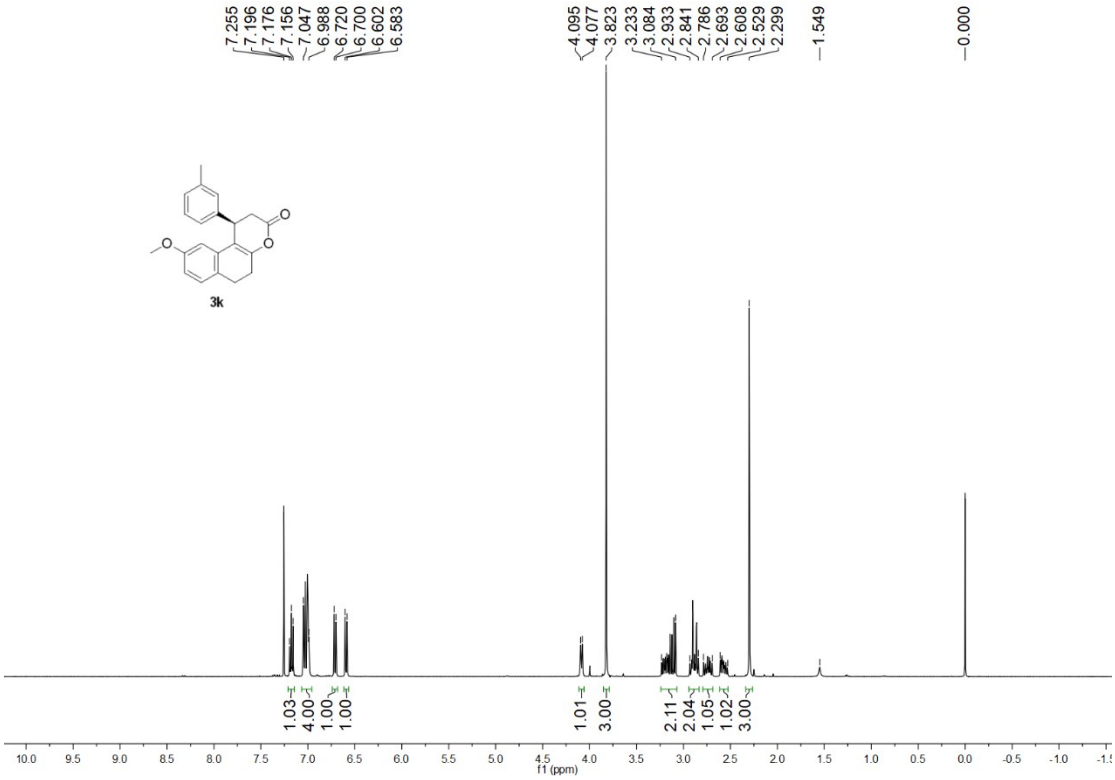
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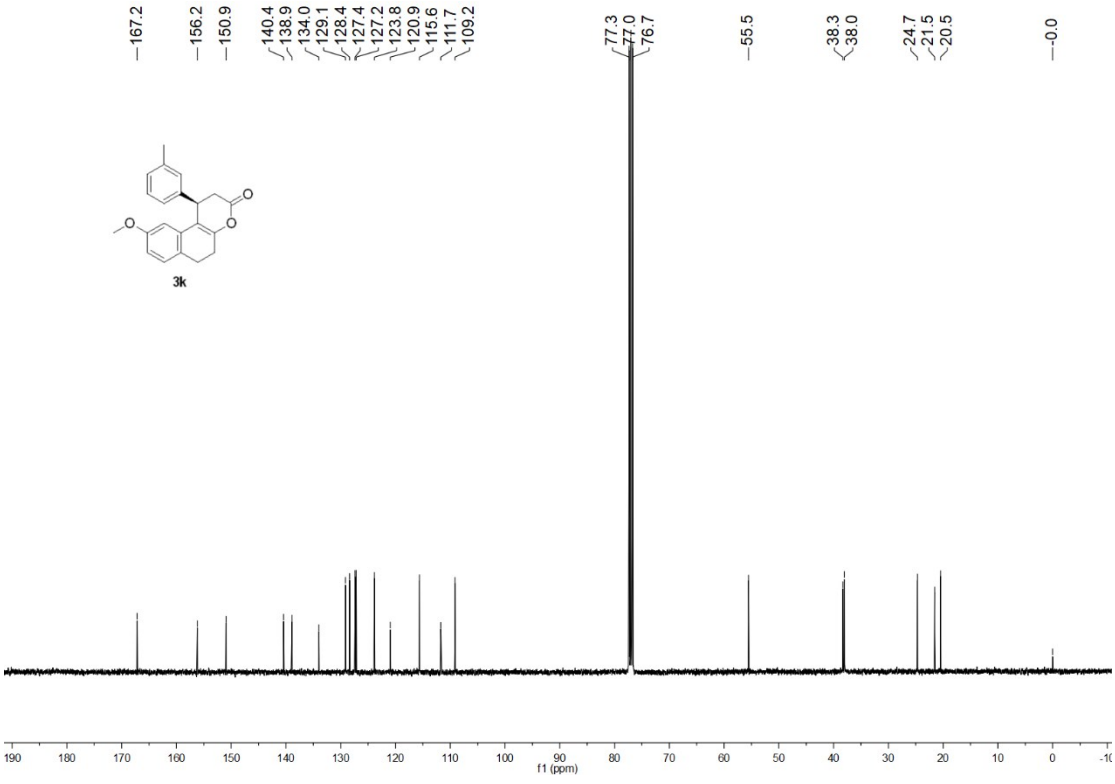
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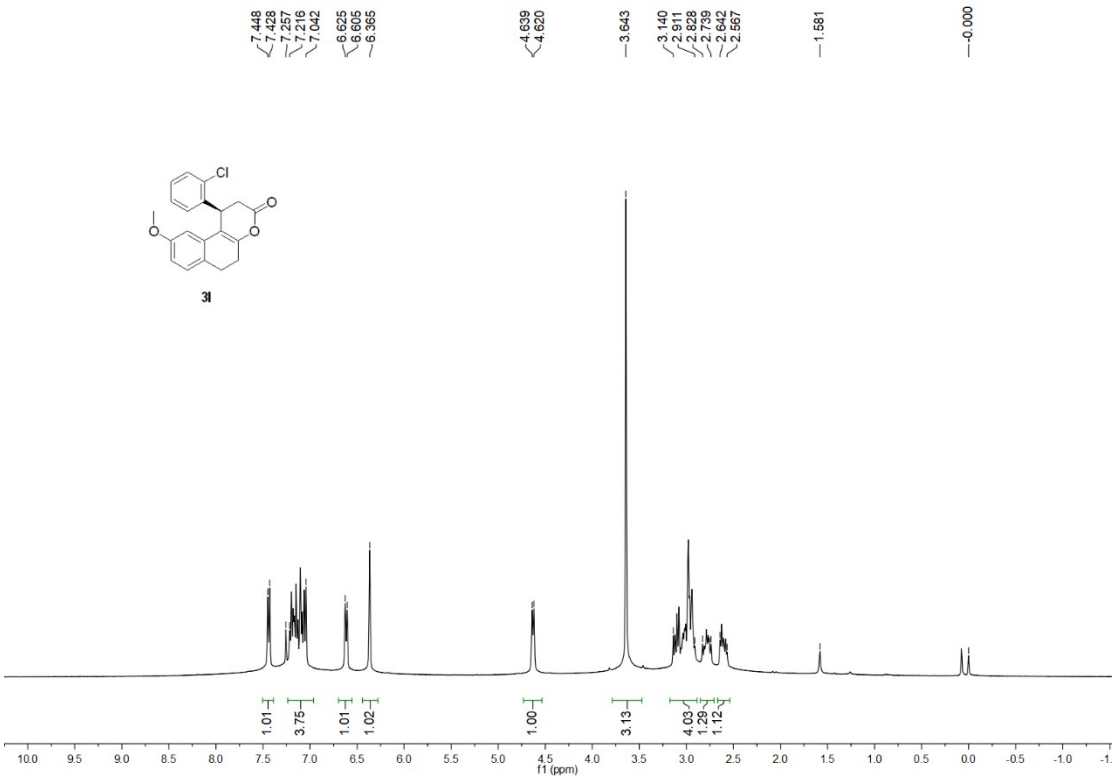
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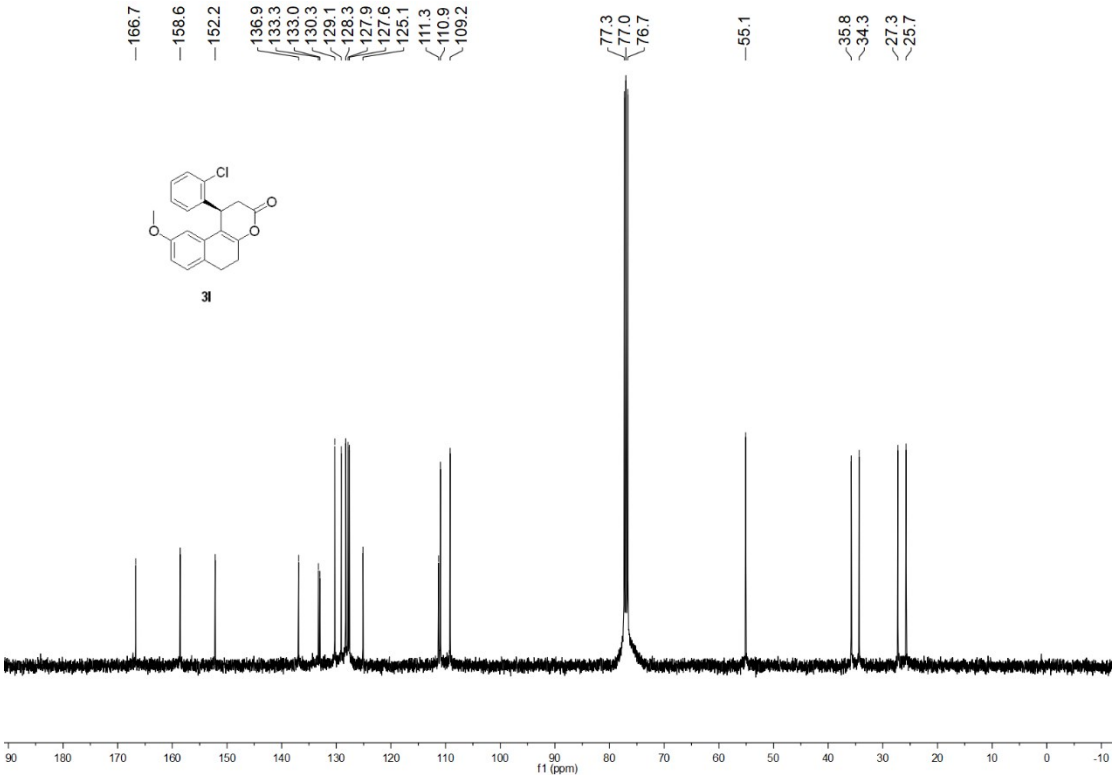
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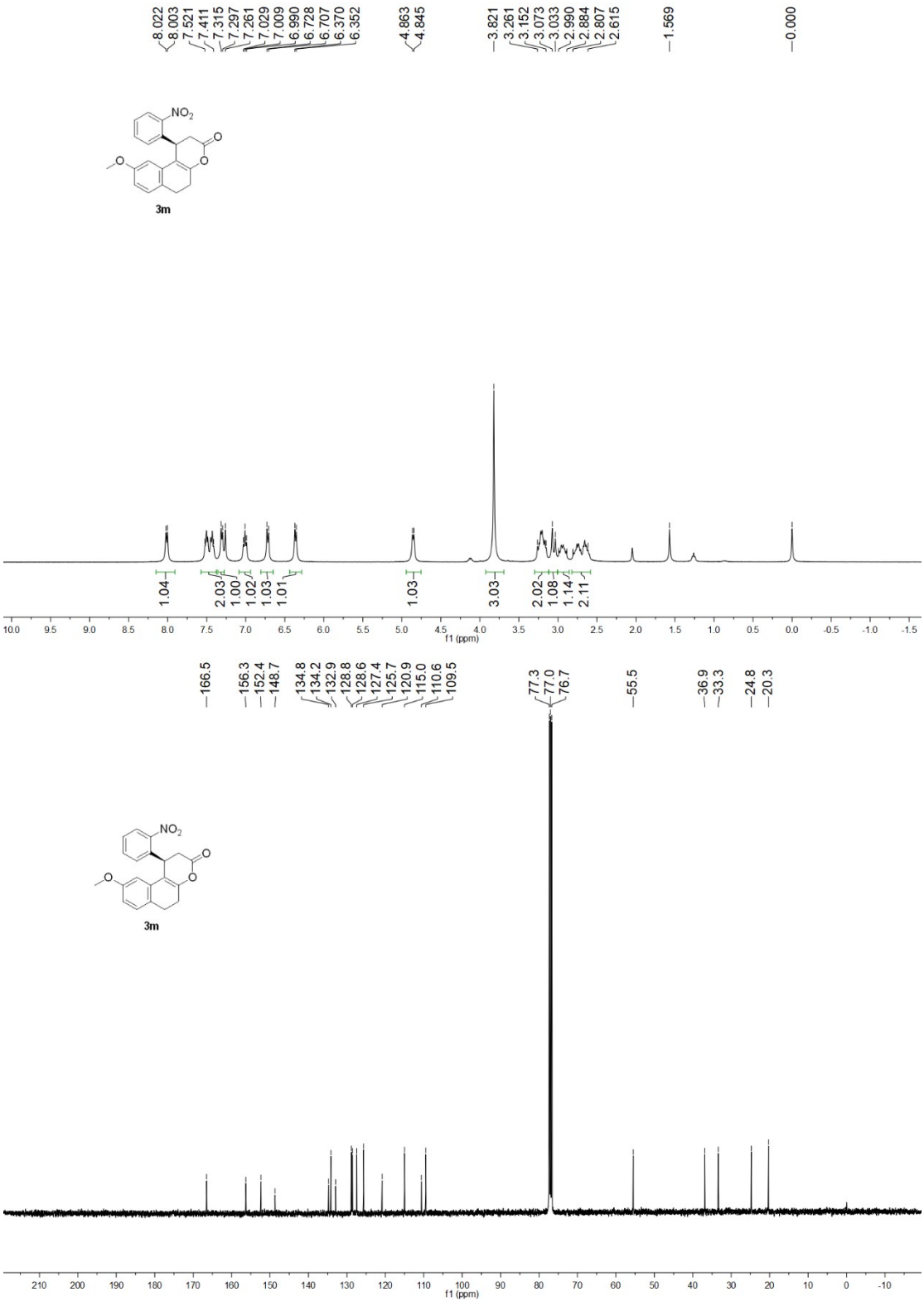
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O
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f1 (ppm)


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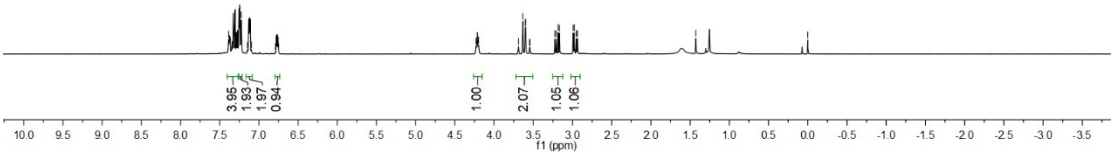
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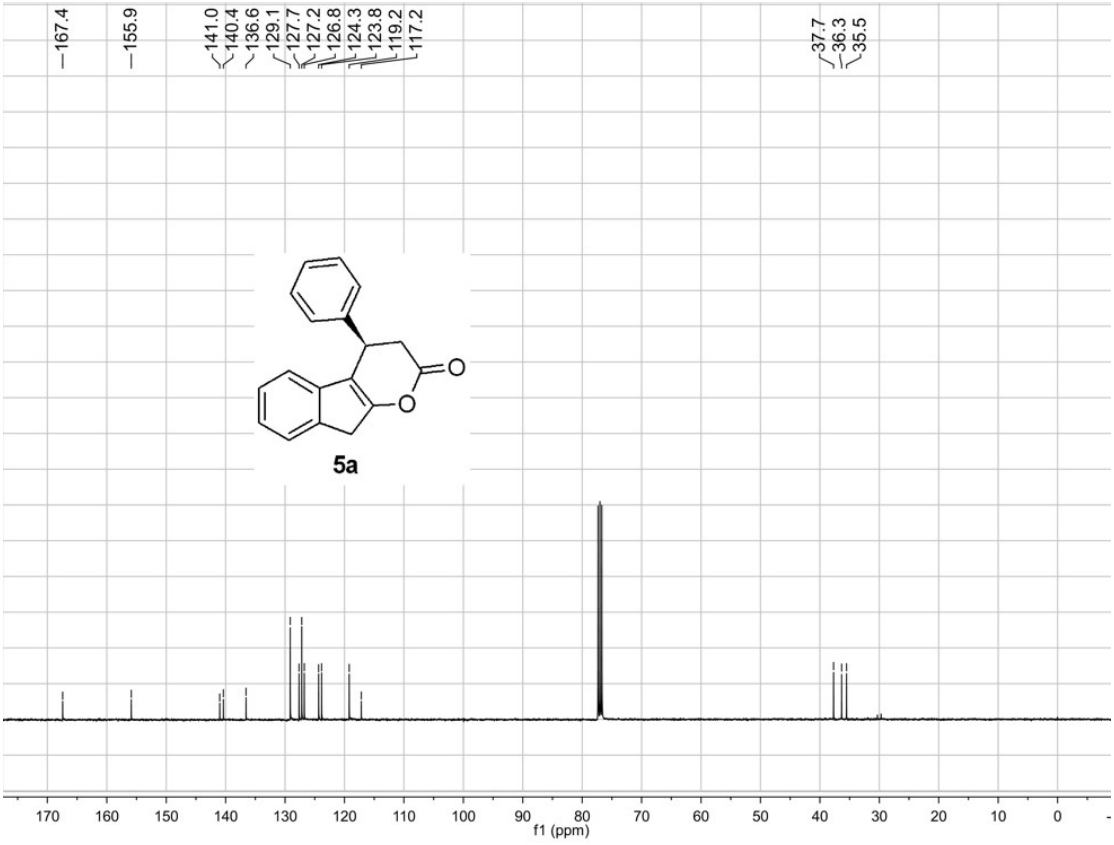
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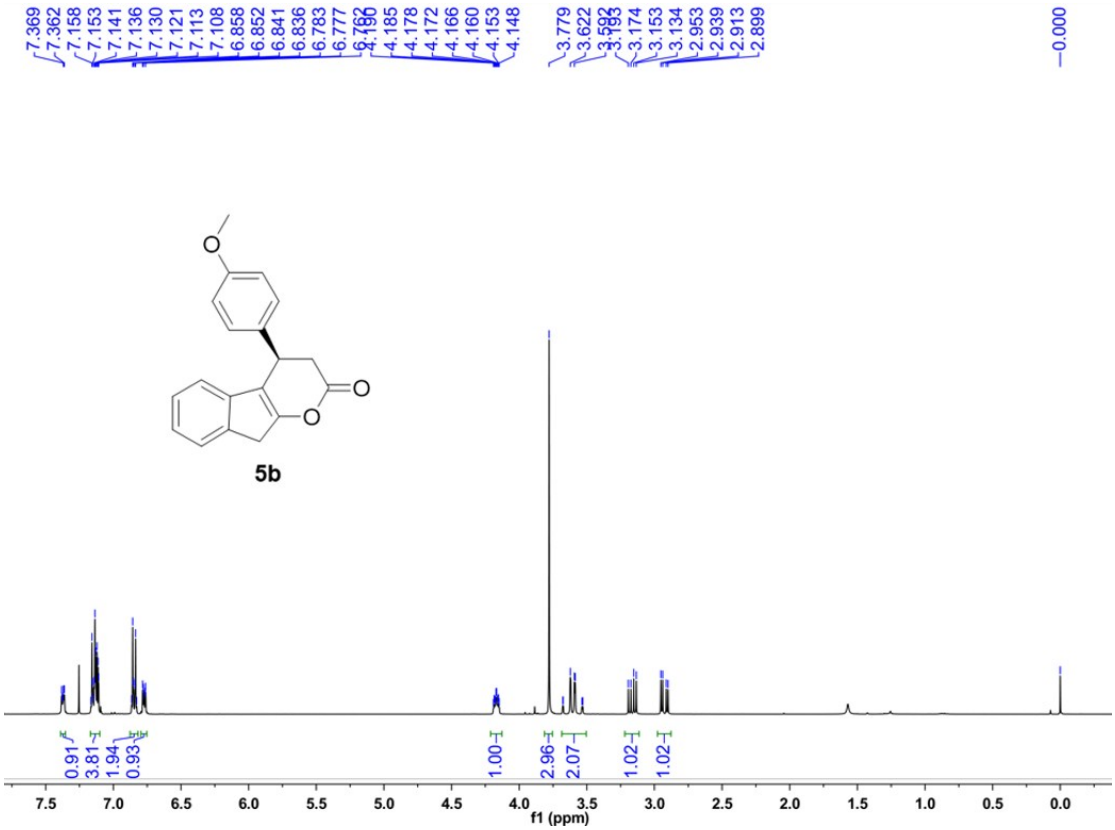
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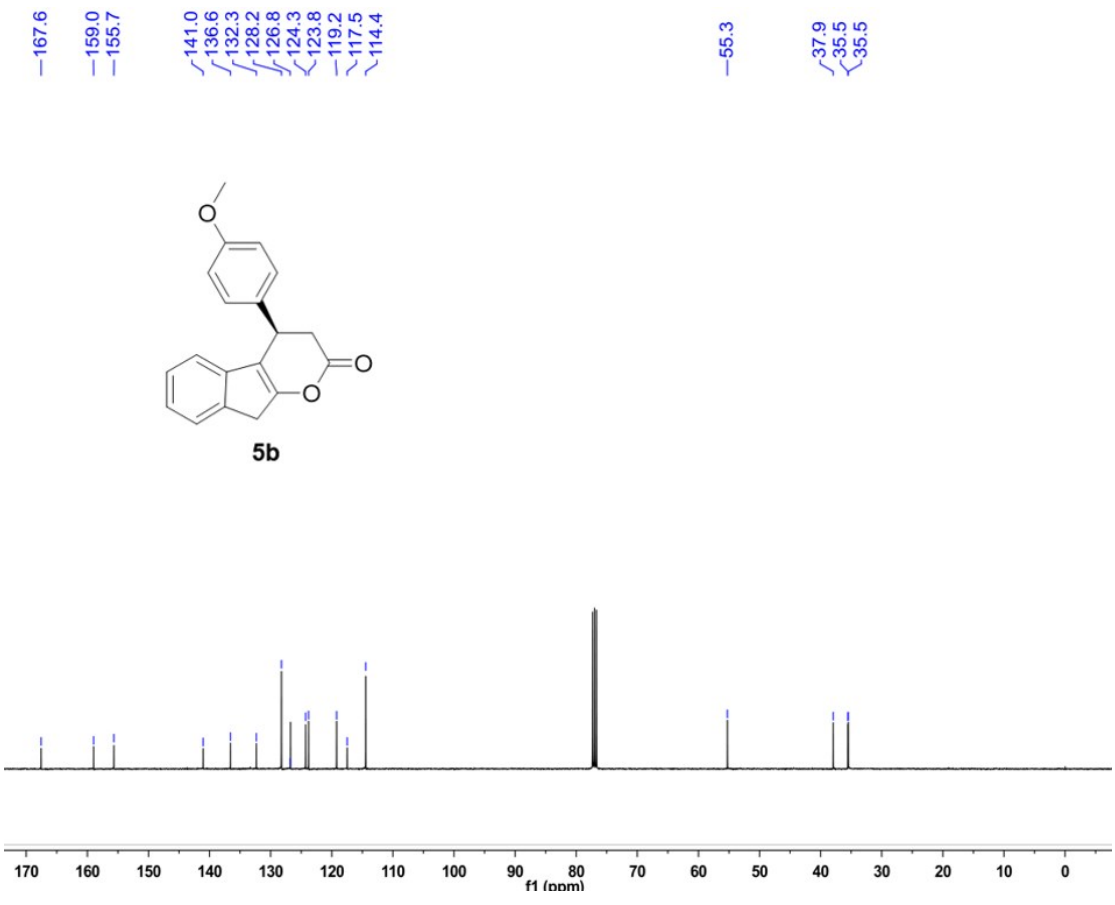
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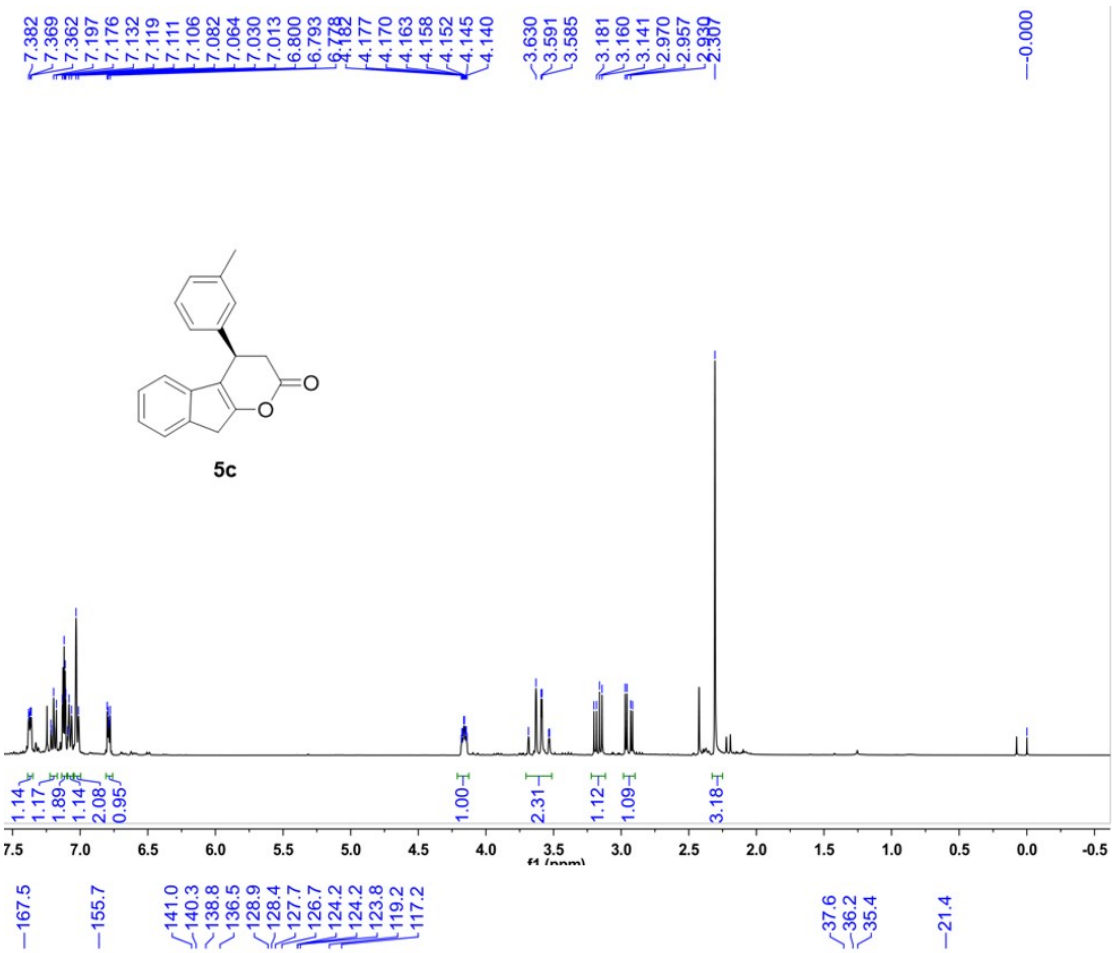
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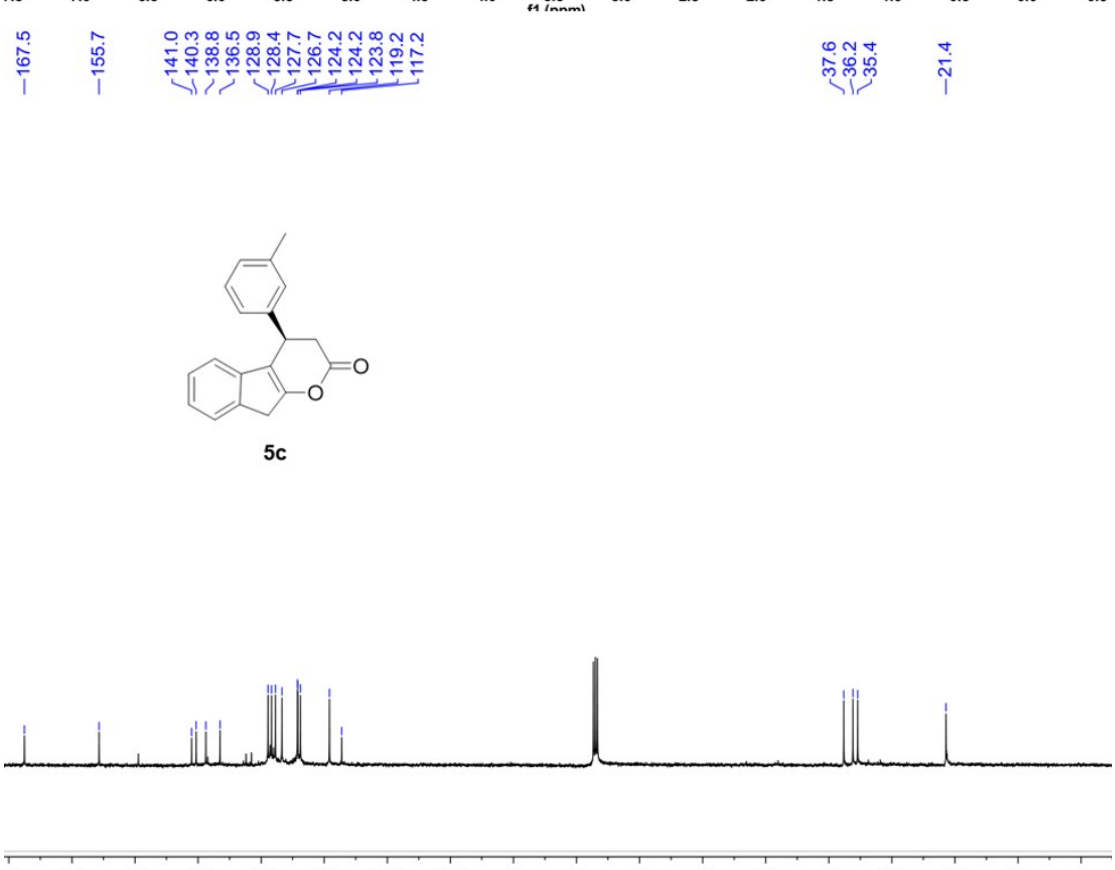
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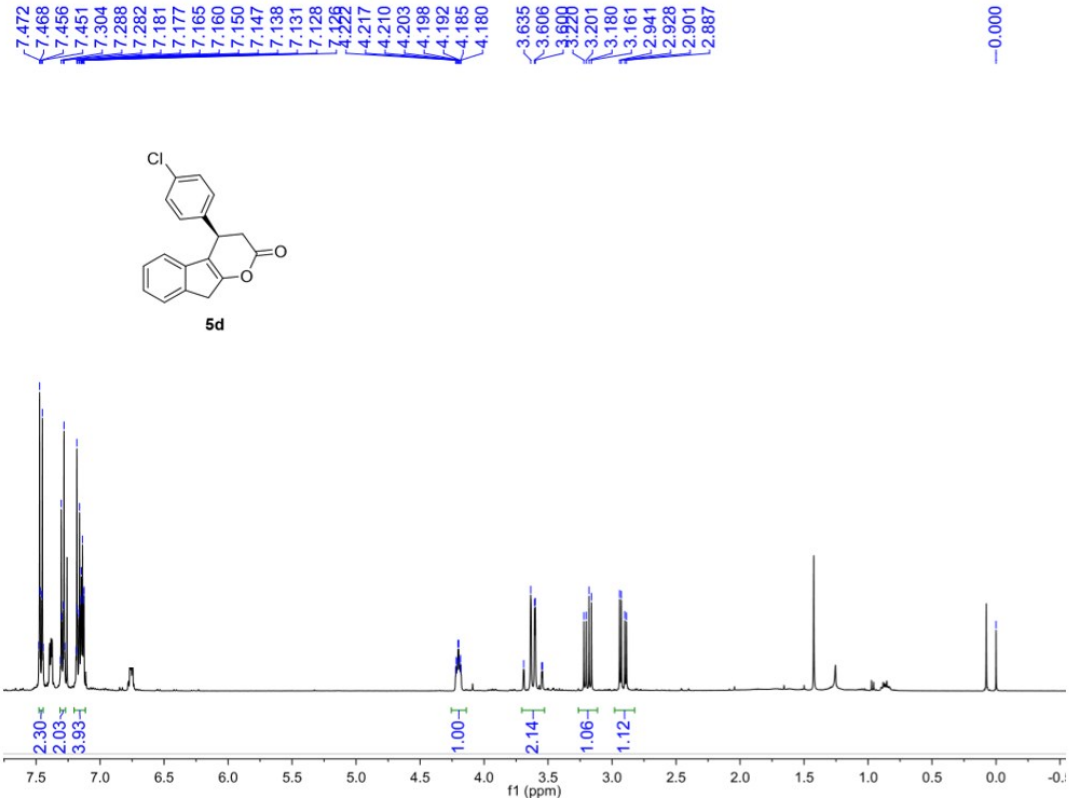
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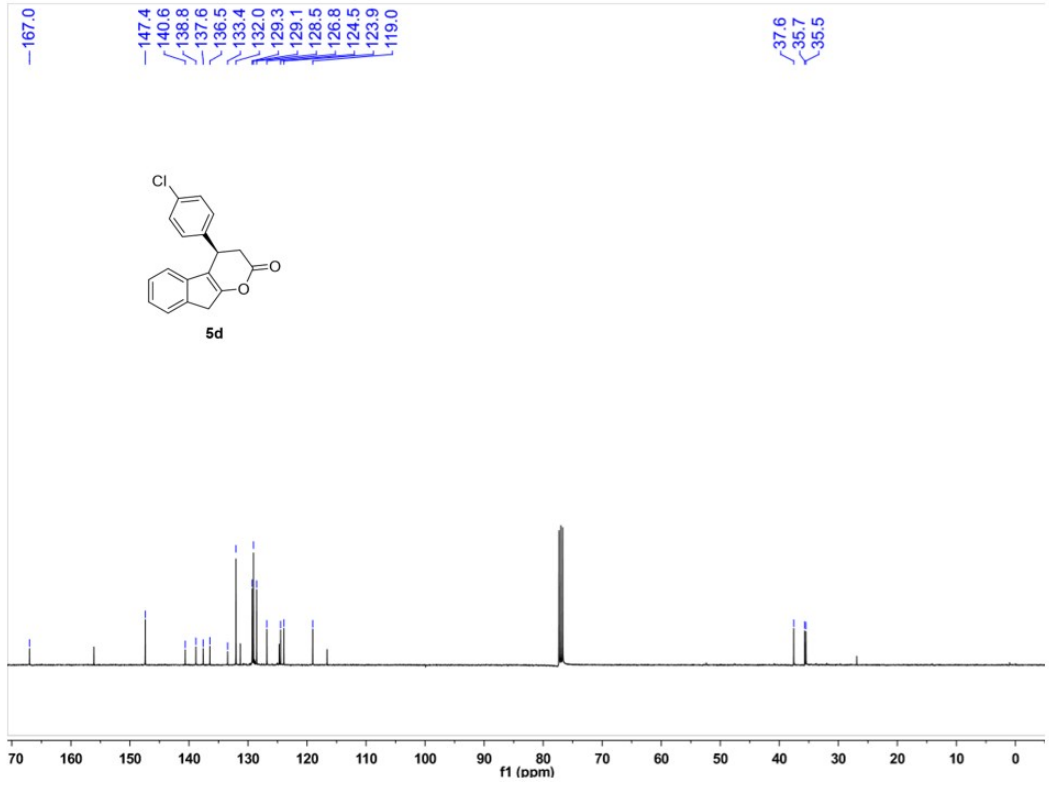
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O
5d
f1 (ppm)

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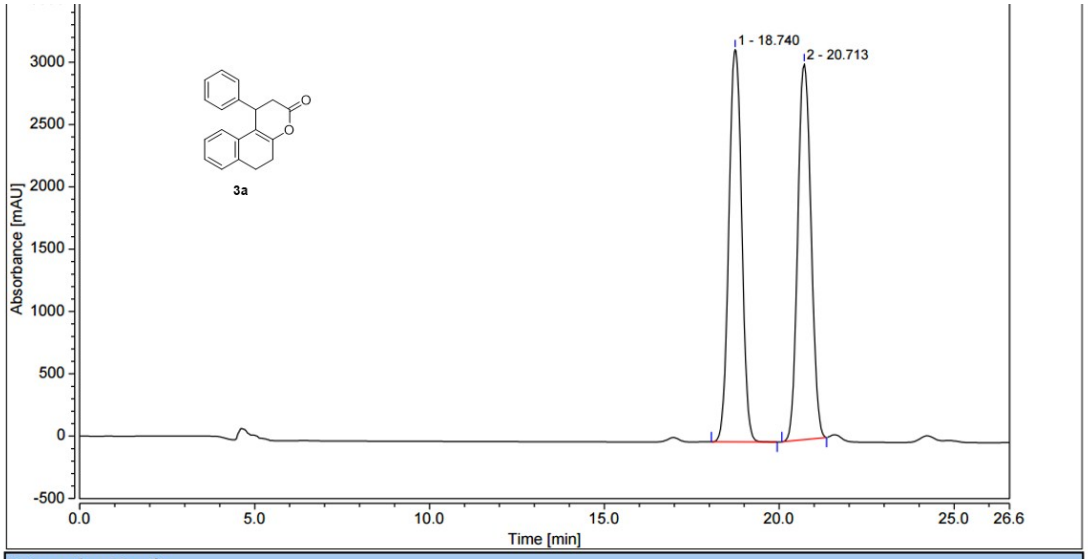
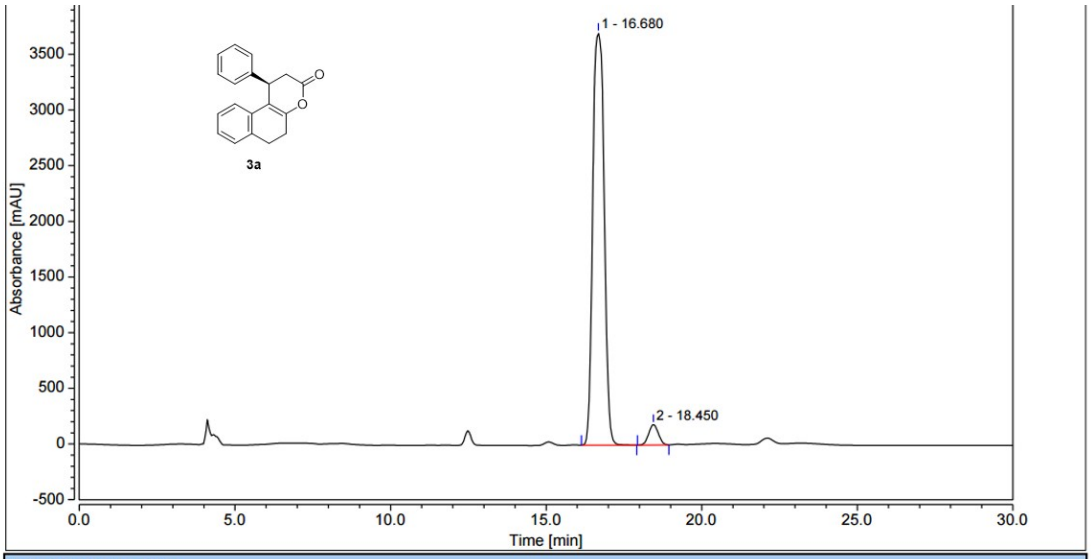


Integration Results

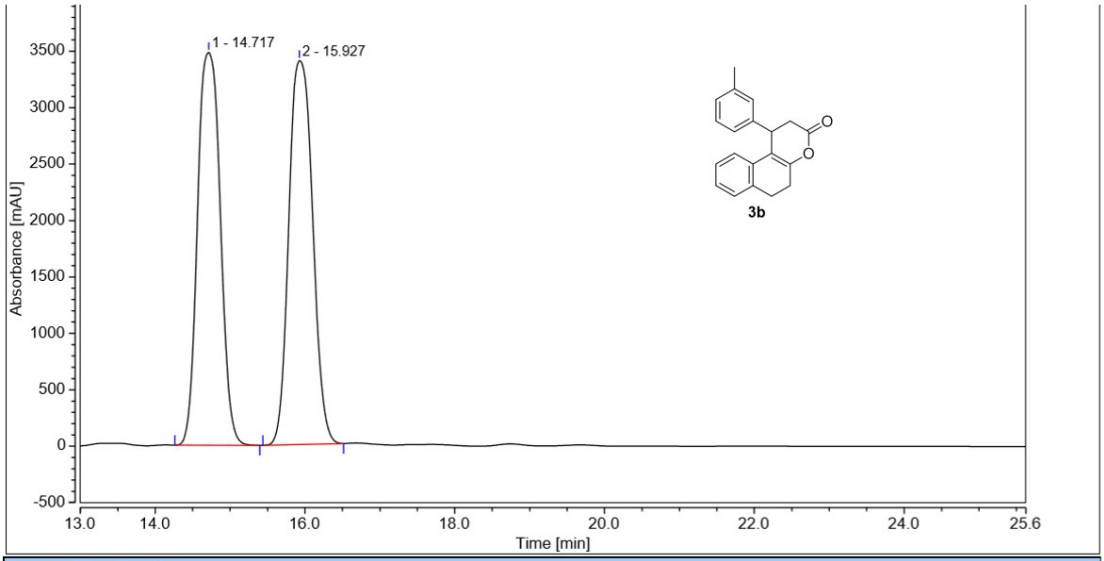
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		18.740	1336.960	3146.691	50.17	51.07	n.a.
2		20.713	1328.108	3015.376	49.83	48.93	n.a.
Total:			**2665.067**	**6162.068**	**100.00**	**100.00**	



Integration Results

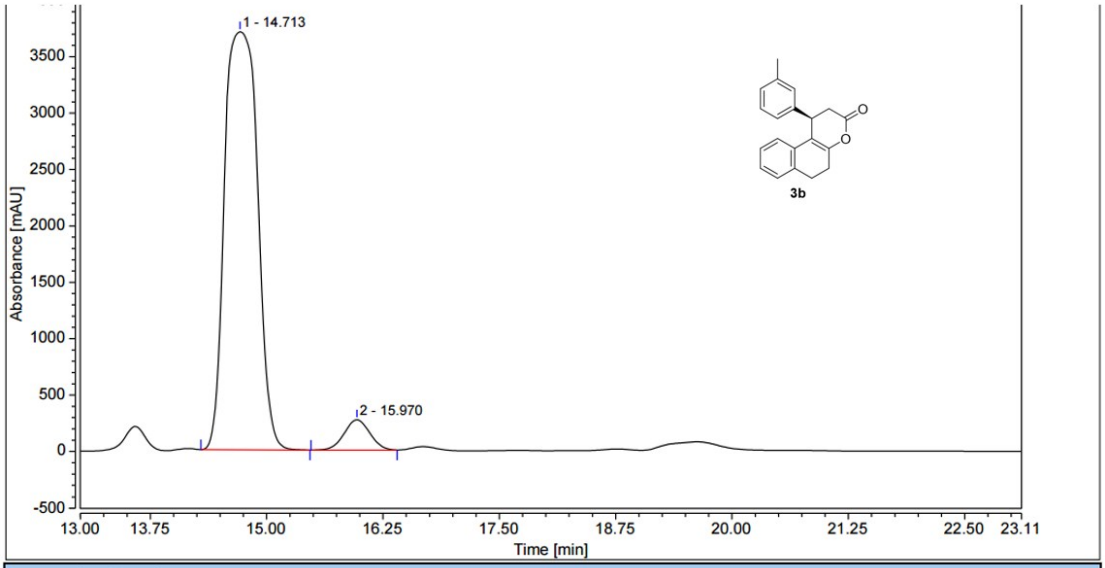
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		16.680	1570.203	3701.319	95.96	95.27	n.a.
2		18.450	66.046	183.880	4.04	4.73	n.a.
Total:			**1636.248**	**3885.199**	**100.00**	**100.00**	

HPLC analysis: 92% ee, [Daicel Chiralpak IC, n-hexane/2-propanol = 90/10, v = 0.8 mL/min, λ = 254 nm, t (major) = 16.6 min, t (minor) = 18.4 min].



Integration Results

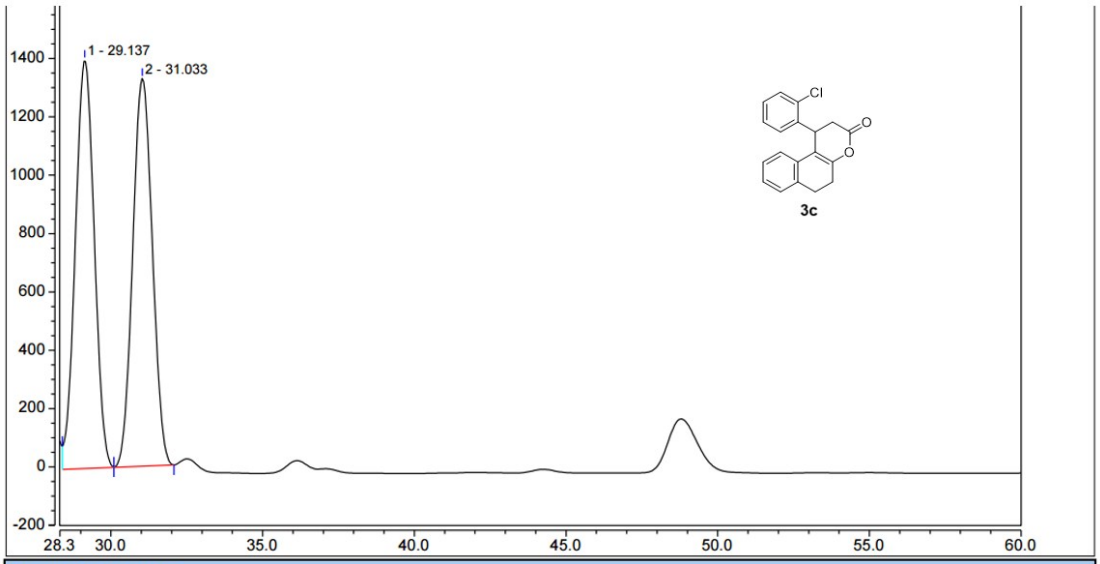
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		14.717	1225.798	3482.891	49.36	50.57	n.a.
2		15.927	1257.362	3404.522	50.64	49.43	n.a.
Total:			**2483.160**	**6887.412**	**100.00**	**100.00**	



Integration Results

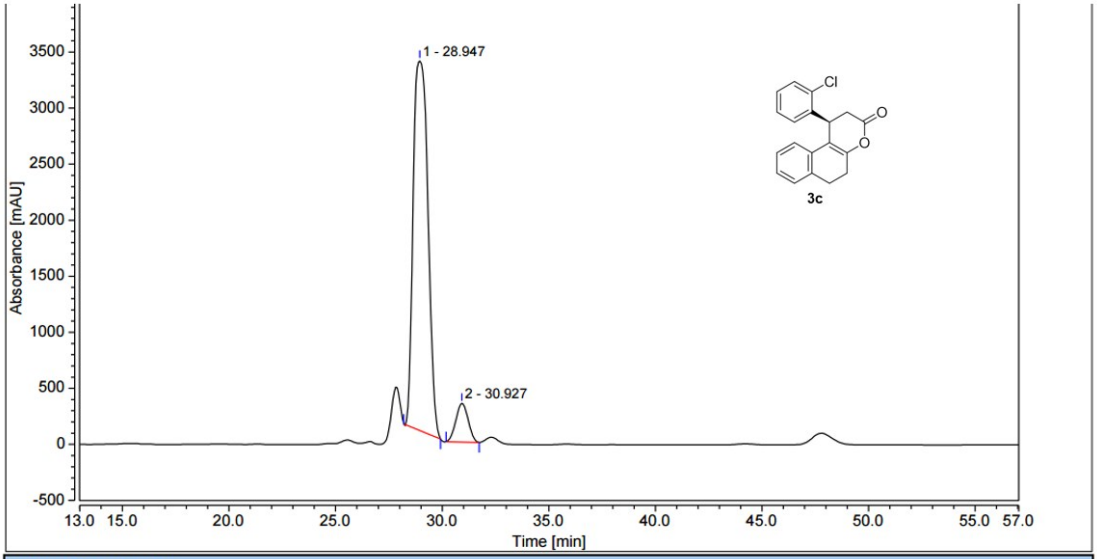
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		14.713	1522.953	3708.094	94.66	93.24	n.a.
2		15.970	85.945	268.633	5.34	6.76	n.a.
Total:			**1608.898**	**3976.727**	**100.00**	**100.00**	

HPLC analysis: 89% ee, [Daicel Chiralpak IC, n-hexane/2-propanol = 90/10, v = 0.8 mL/min, λ = 254 nm, t (major) = 14.7 min, t (minor) =15.9 min].



Integration Results

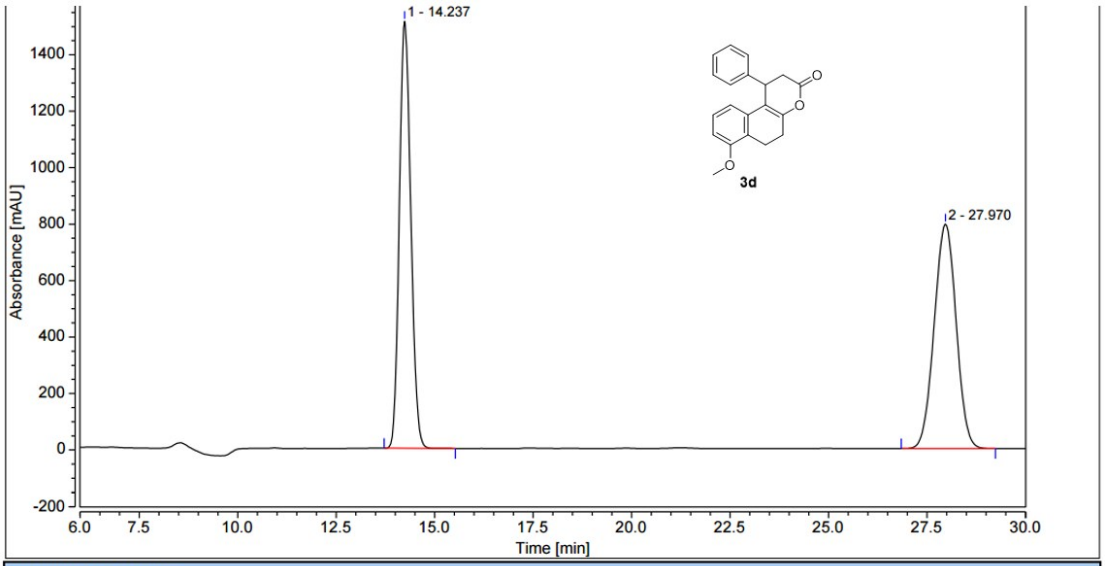
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		29.137	1011.991	1400.173	50.47	51.29	n.a.
2		31.033	992.983	1329.842	49.53	48.71	n.a.
Total:			**2004.975**	**2730.014**	**100.00**	**100.00**	



Integration Results

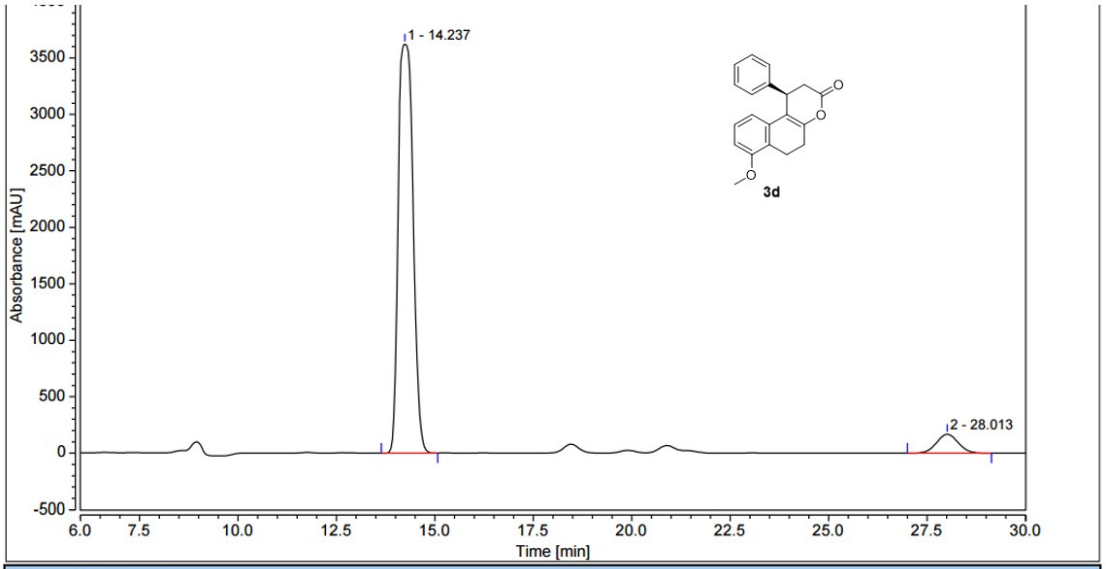
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		28.947	2646.214	3303.743	91.98	90.51	n.a.
2		30.927	230.841	346.337	8.02	9.49	n.a.
Total:			**2877.055**	**3650.080**	**100.00**	**100.00**	

HPLC analysis: 84% ee, [Daicel Chiralpak IC, n-hexane/2-propanol = 98/2, v = 0.6 mL/min, λ = 254 nm, t (major) = 28.9 min, t (minor) = 30.9 min].



Integration Results

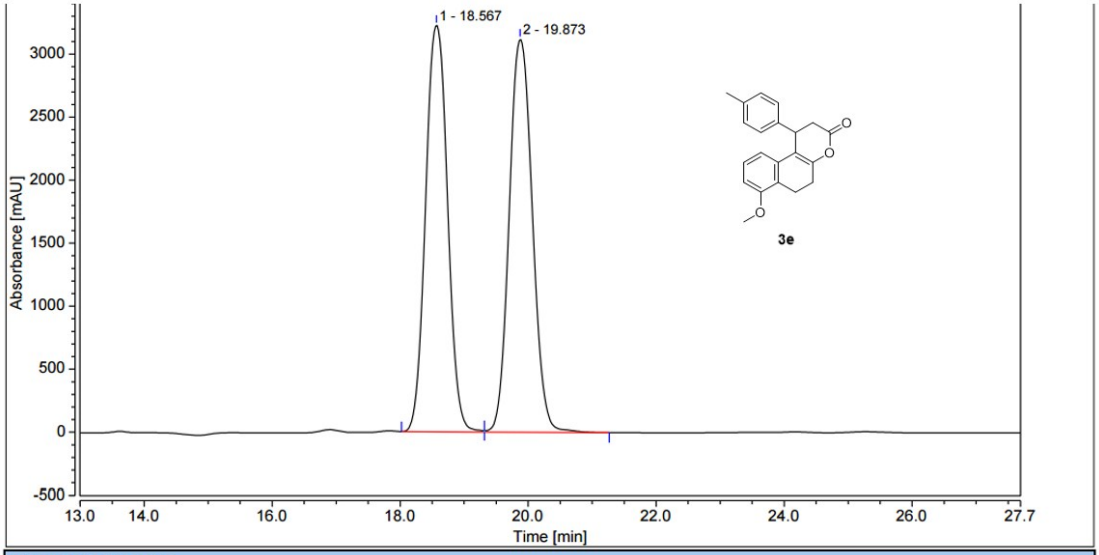
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		14.237	512.568	1512.788	49.91	65.53	n.a.
2		27.970	514.388	795.917	50.09	34.47	n.a.
Total:			**1026.956**	**2308.705**	**100.00**	**100.00**	



Integration Results

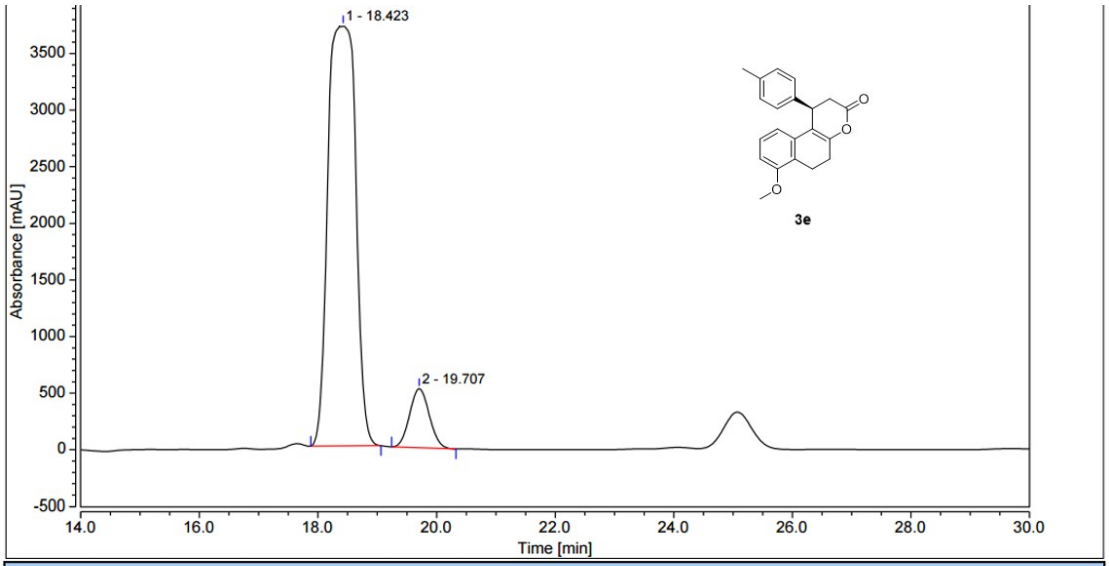
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		14.237	1589.460	3622.916	93.78	95.62	n.a.
2		28.013	105.431	165.993	6.22	4.38	n.a.
Total:			**1694.891**	**3788.909**	**100.00**	**100.00**	

HPLC analysis: 88% ee, [Daicel Chiralpak OZ-H, n-hexane/2-propanol = 90/10, v = 0.8 mL/min, λ = 254 nm, t (major) = 14.2 min, t (minor) = 28.0 min].



Integration Results

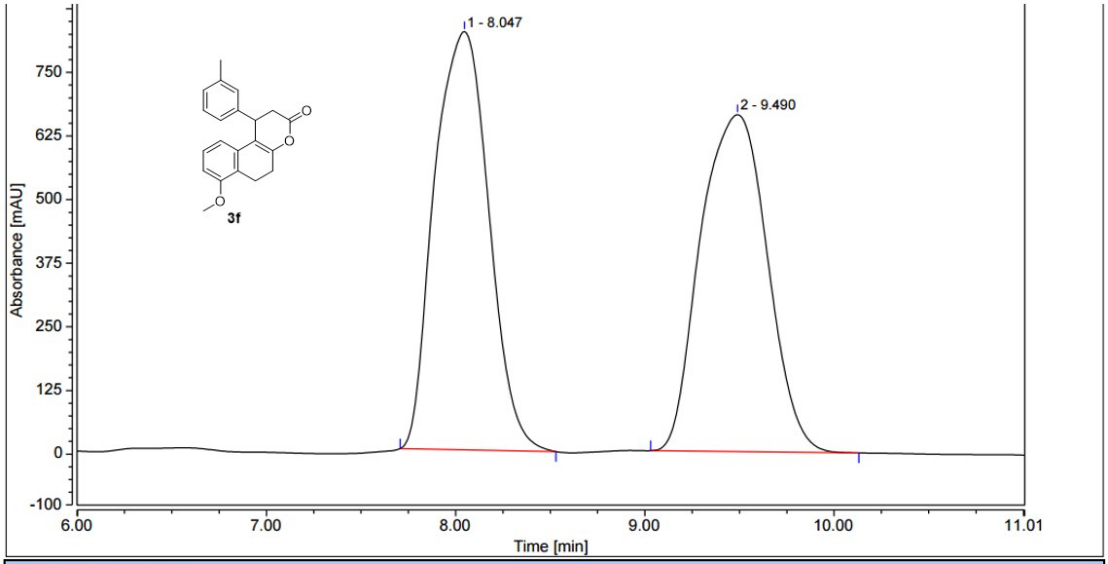
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		18.567	1281.129	3226.361	49.48	50.85	n.a.
2		19.873	1307.928	3118.904	50.52	49.15	n.a.
Total:			**2589.057**	**6345.265**	**100.00**	**100.00**	



Integration Results

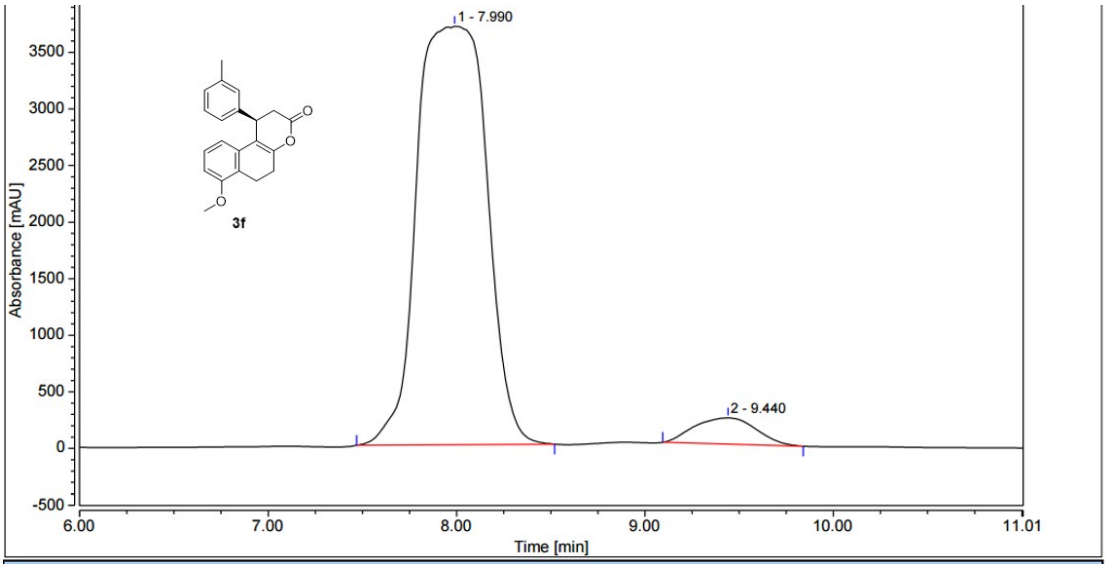
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		18.423	2010.596	3712.980	90.93	87.66	n.a.
2		19.707	200.583	522.561	9.07	12.34	n.a.
Total:			**2211.179**	**4235.542**	**100.00**	**100.00**	

HPLC analysis: 82% ee, [Daicel Chiralpak IC, n-hexane/2-propanol = 90/10, v = 0.8 mL/min, λ = 254 nm, t (major) = 18.4 min, t (minor) = 19.7 min].



Integration Results

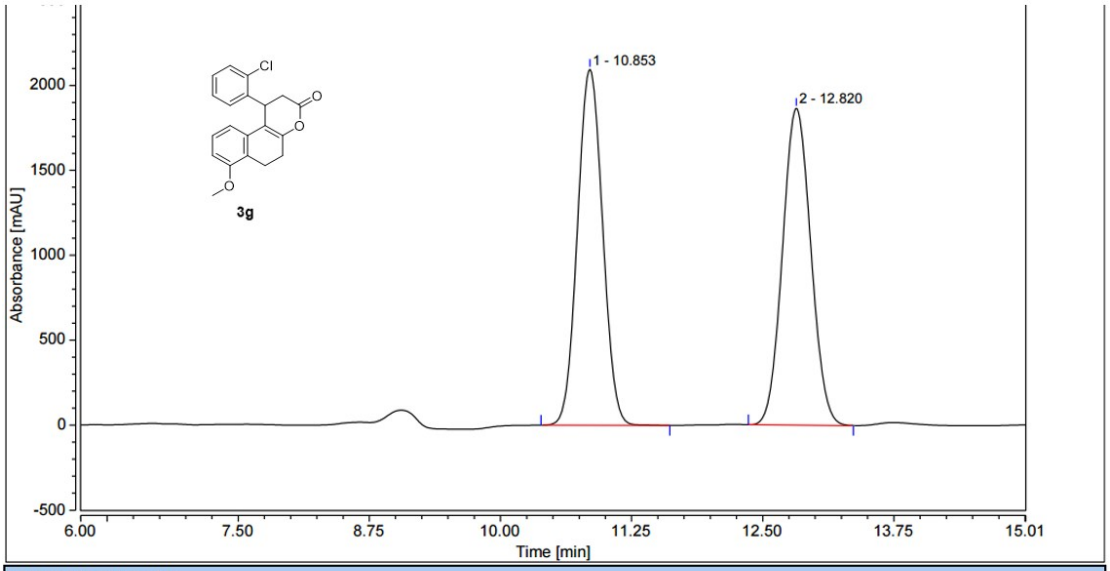
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		8.047	274.601	821.964	50.31	55.38	n.a.
2		9.490	271.188	662.317	49.69	44.62	n.a.
Total:			**545.788**	**1484.280**	**100.00**	**100.00**	



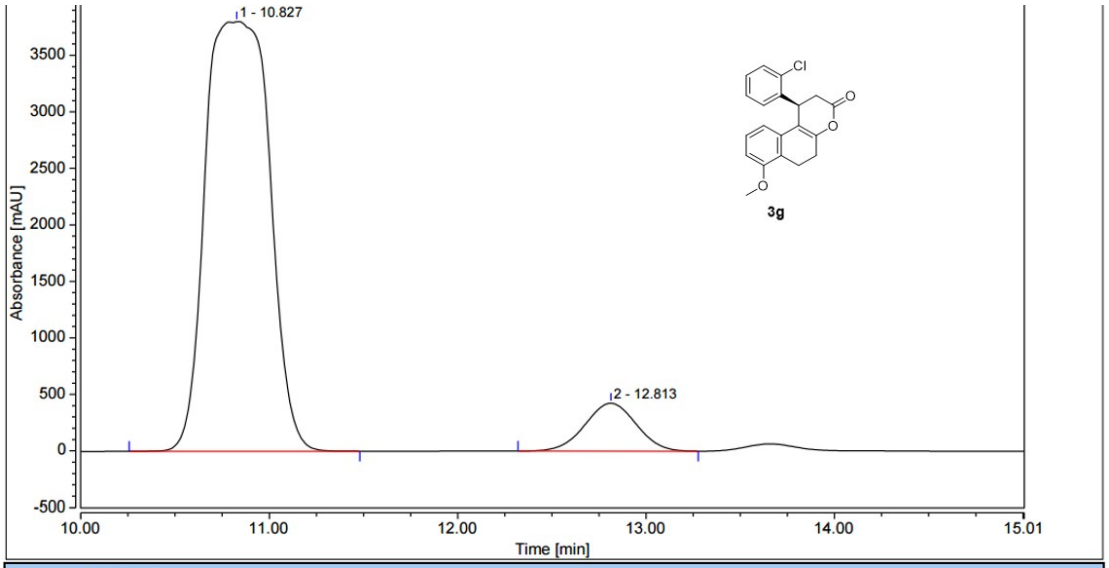
Integration Results

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		7.990	1589.607	3699.594	94.75	94.10	n.a.
2		9.440	87.991	231.789	5.25	5.90	n.a.
Total:			**1677.597**	**3931.383**	**100.00**	**100.00**	

HPLC analysis: 90% ee, [Daicel Chiralpak AD-H, n-hexane/2-propanol = 90/10, v = 0.8 mL/min, λ = 254 nm, t (major) = 7.9 min, t (minor) = 9.4 min].

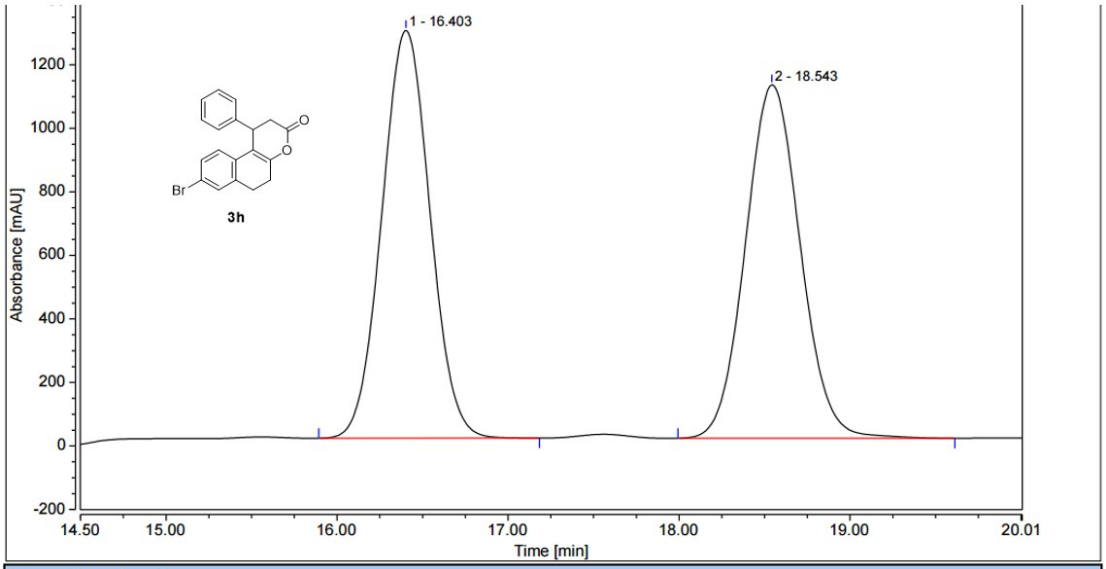


Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		10.853	583.189	2095.448	49.93	52.90	n.a.
2		12.820	584.751	1866.031	50.07	47.10	n.a.
Total:			**1167.940**	**3961.479**	**100.00**	**100.00**	

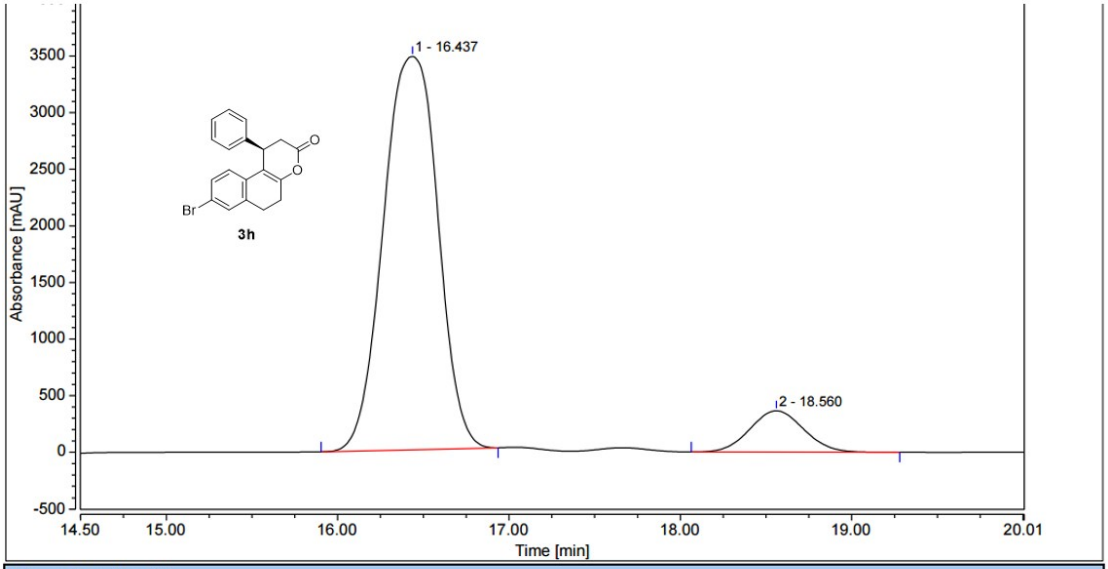


Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		10.827	1501.803	3802.561	91.80	89.97	n.a.
2		12.813	134.150	423.965	8.20	10.03	n.a.
Total:			**1635.953**	**4226.526**	**100.00**	**100.00**	

HPLC analysis: 84% ee, [Daicel Chiralpak OZ-H, n-hexane/2-propanol = 90/10, v = 0.8 mL/min, λ = 254 nm, t (major) = 10.8 min, t (minor) = 12.8 min].

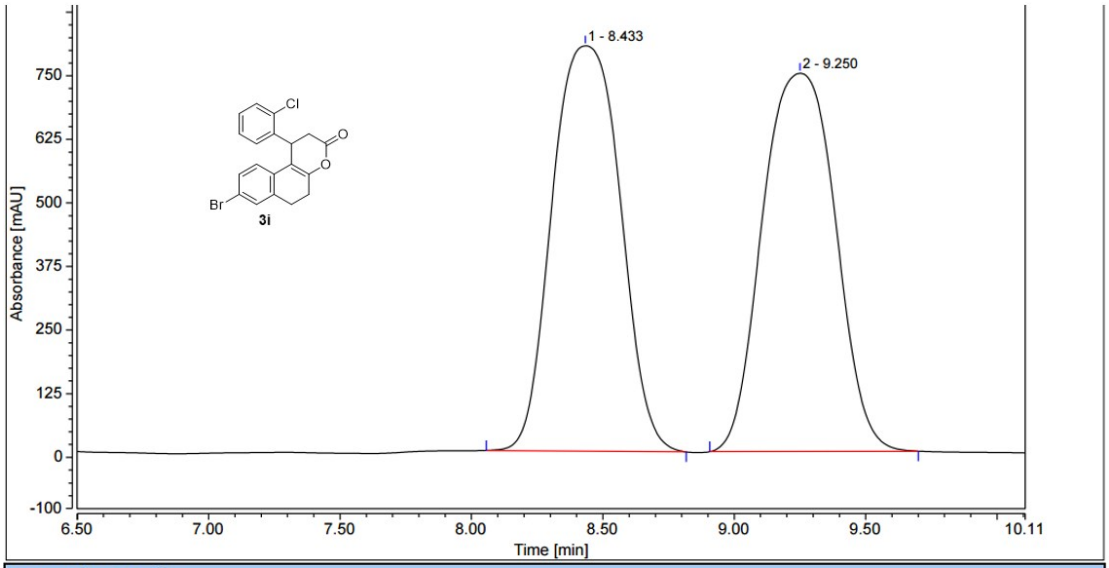


Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		16.403	419.349	1283.815	50.34	53.57	n.a.
2		18.543	413.759	1112.727	49.66	46.43	n.a.
Total:			**833.109**	**2396.542**	**100.00**	**100.00**	



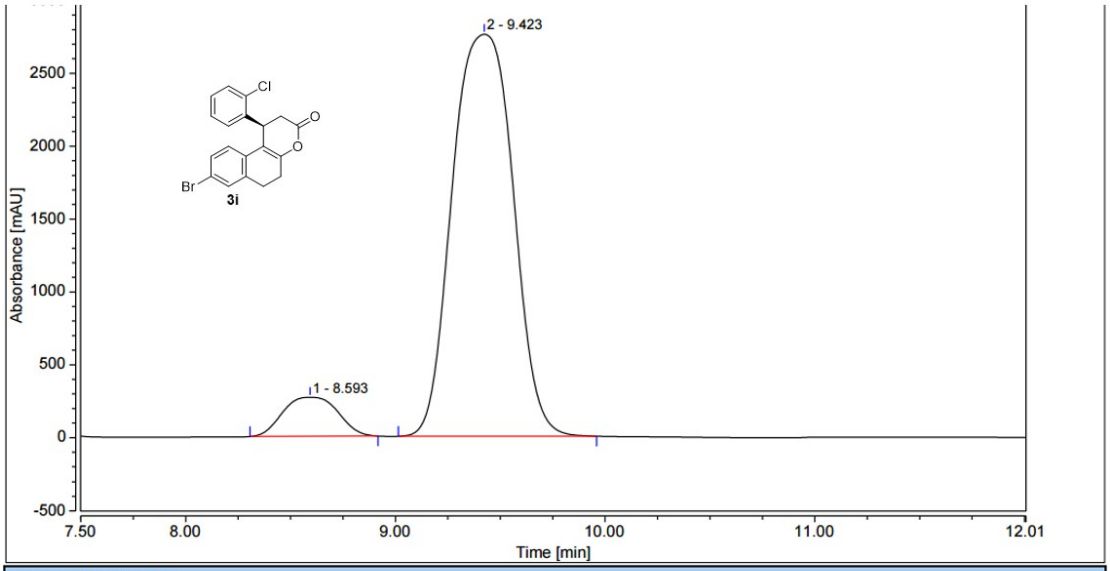
Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		16.437	1283.173	3474.418	90.61	90.48	n.a.
2		18.560	133.038	365.706	9.39	9.52	n.a.
Total:			**1416.211**	**3840.124**	**100.00**	**100.00**	

HPLC analysis: 81% ee, [Daicel Chiralpak IC, n-hexane/2-propanol = 90/0, v = 0.8 mL/min, λ = 254 nm, t (major) = 16.4 min, t (minor) = 18.5 min].



Integration Results

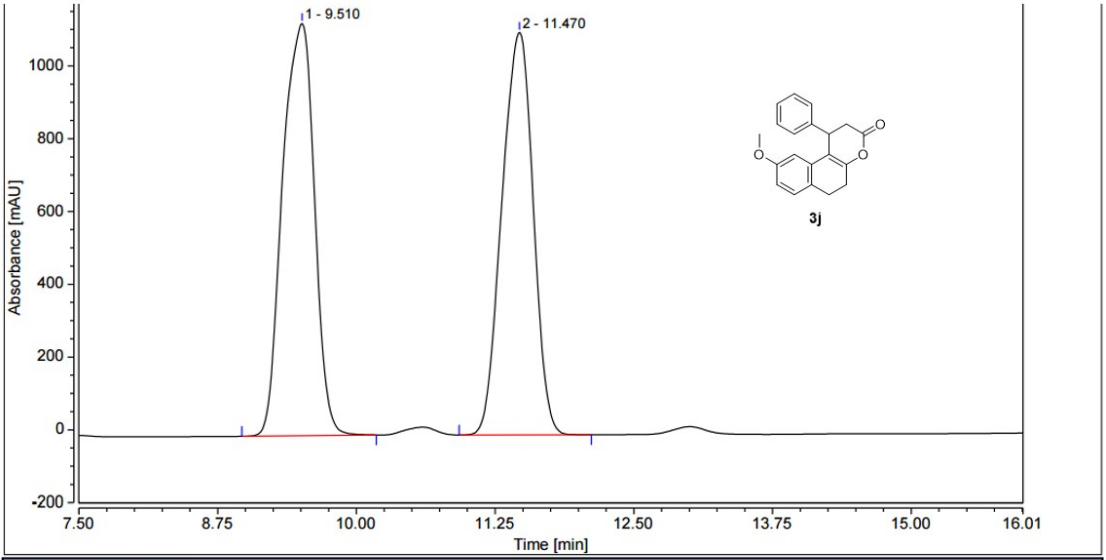
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		8.433	240.481	796.890	50.07	51.74	n.a.
2		9.250	239.834	743.435	49.93	48.26	n.a.
Total:			**480.314**	**1540.326**	**100.00**	**100.00**	



Integration Results

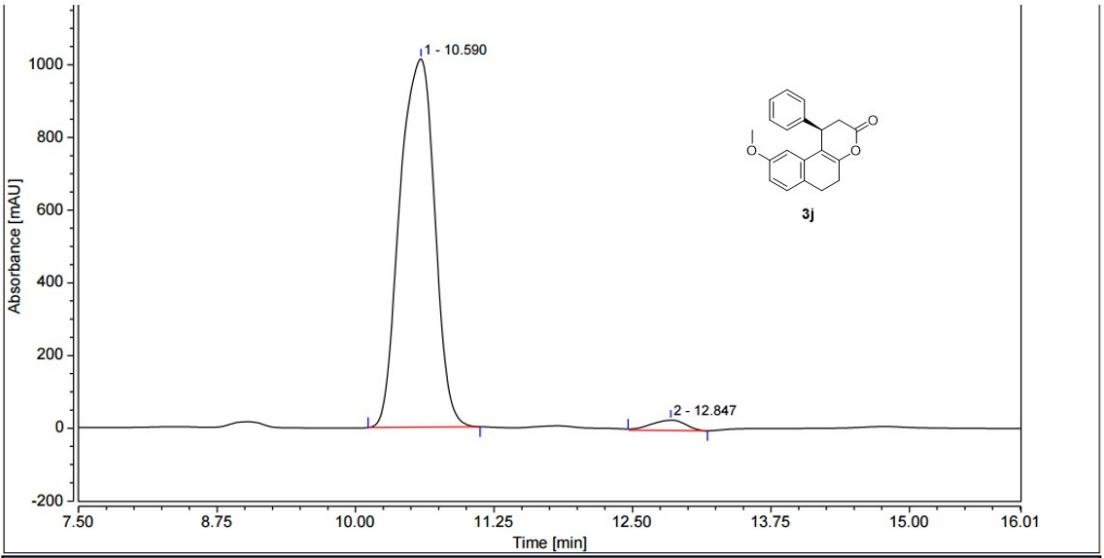
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		8.593	81.279	267.953	8.03	8.85	n.a.
2		9.423	930.589	2759.336	91.97	91.15	n.a.
Total:			**1011.868**	**3027.289**	**100.00**	**100.00**	

HPLC analysis: 84% ee, [Daicel Chiralpak IA, n-hexane/2-propanol = 90/10, v = 0.8 mL/min, λ = 254 nm, t (major) = 9.4 min, t (minor) = 8.5 min].



Integration Results

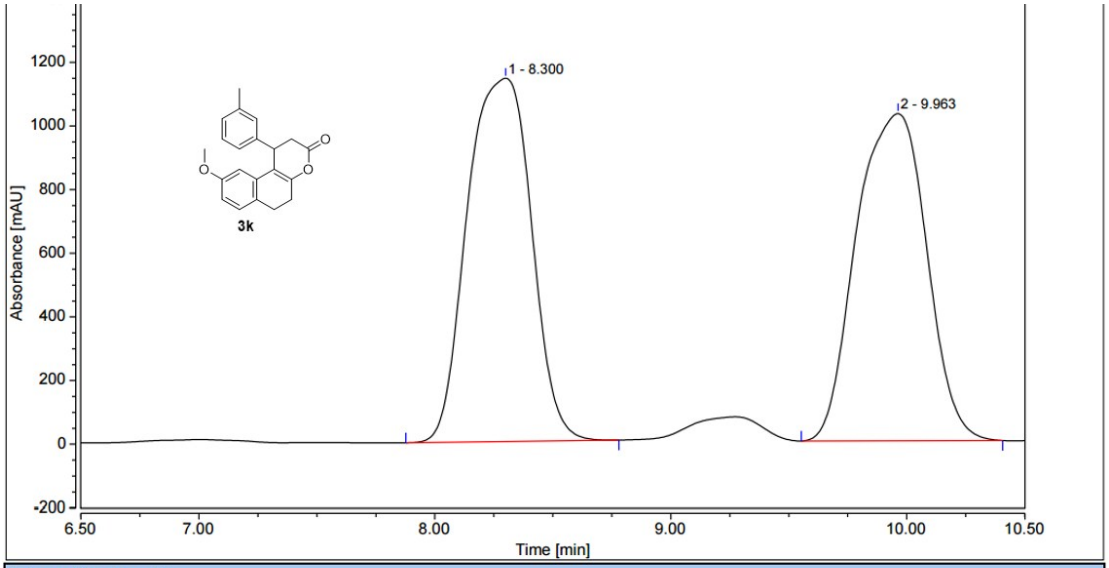
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		9.510	376.811	1133.256	51.05	50.60	n.a.
2		11.470	361.324	1106.299	48.95	49.40	n.a.
Total:			**738.135**	**2239.556**	**100.00**	**100.00**	



Integration Results

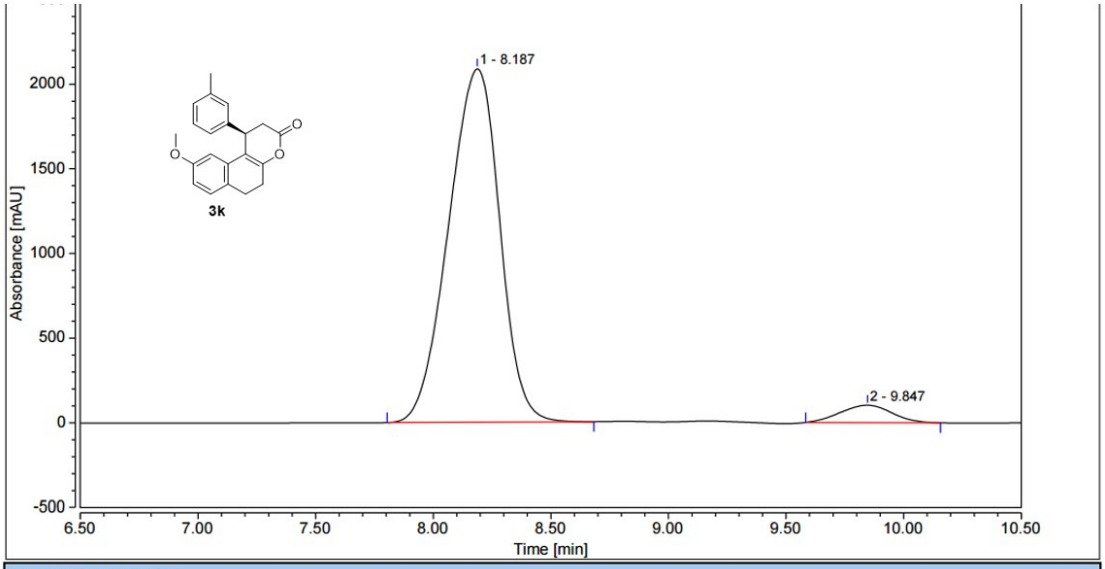
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		10.590	369.370	1013.354	97.31	97.27	n.a.
2		12.847	10.226	28.398	2.69	2.73	n.a.
Total:			**379.596**	**1041.752**	**100.00**	**100.00**	

HPLC analysis: 95% ee, [Daicel Chiralpak IA, n-hexane/2-propanol = 90/10, v = 0.8 mL/min, λ = 254 nm, t (major) = 10.5 min, t (minor) = 12.8 min].



Integration Results

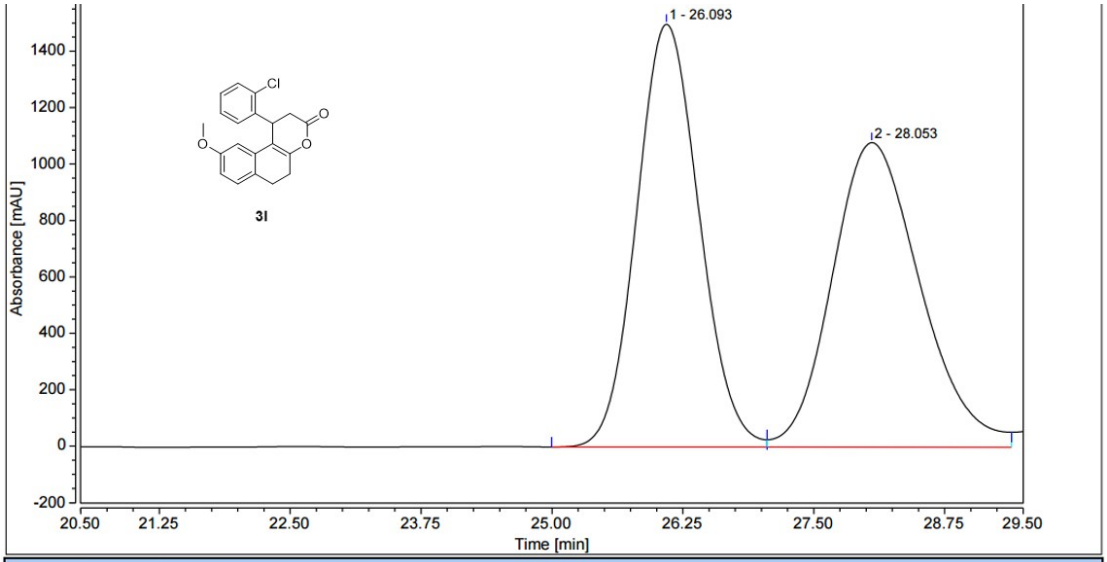
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		8.300	360.480	1141.424	50.37	52.61	n.a.
2		9.963	355.169	1028.024	49.63	47.39	n.a.
Total:			**715.649**	**2169.448**	**100.00**	**100.00**	



Integration Results

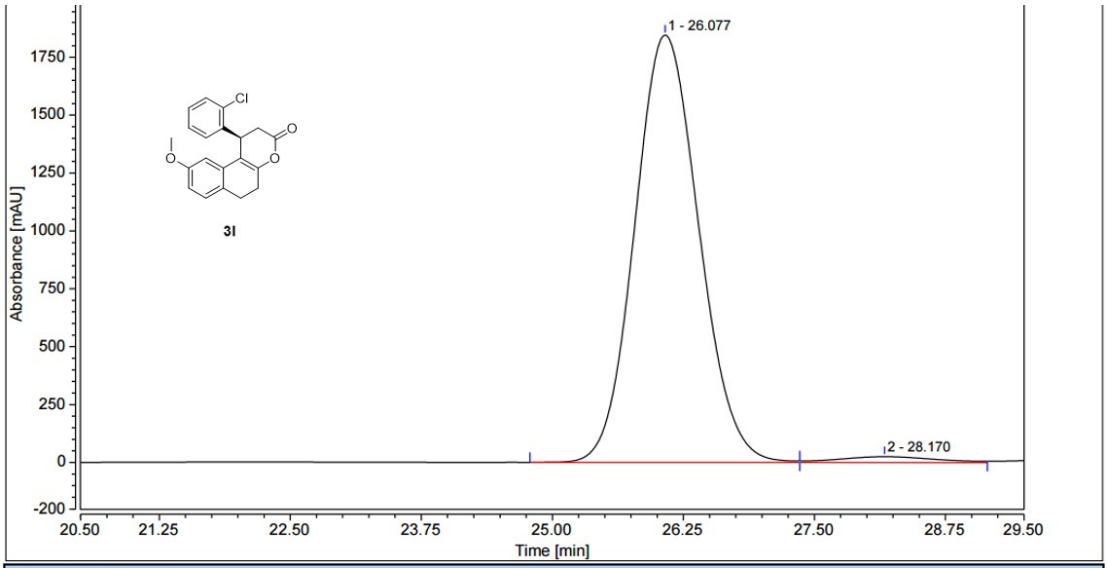
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		8.187	526.025	2087.254	95.27	95.29	n.a.
2		9.847	26.136	103.253	4.73	4.71	n.a.
Total:			**552.162**	**2190.507**	**100.00**	**100.00**	

HPLC analysis: 91% ee, [Daicel Chiralpak IA, n-hexane/2-propanol = 90/10, v = 0.8 mL/min, λ = 254 nm, t (major) = 8.1 min, t (minor) = 9.8 min].



Integration Results

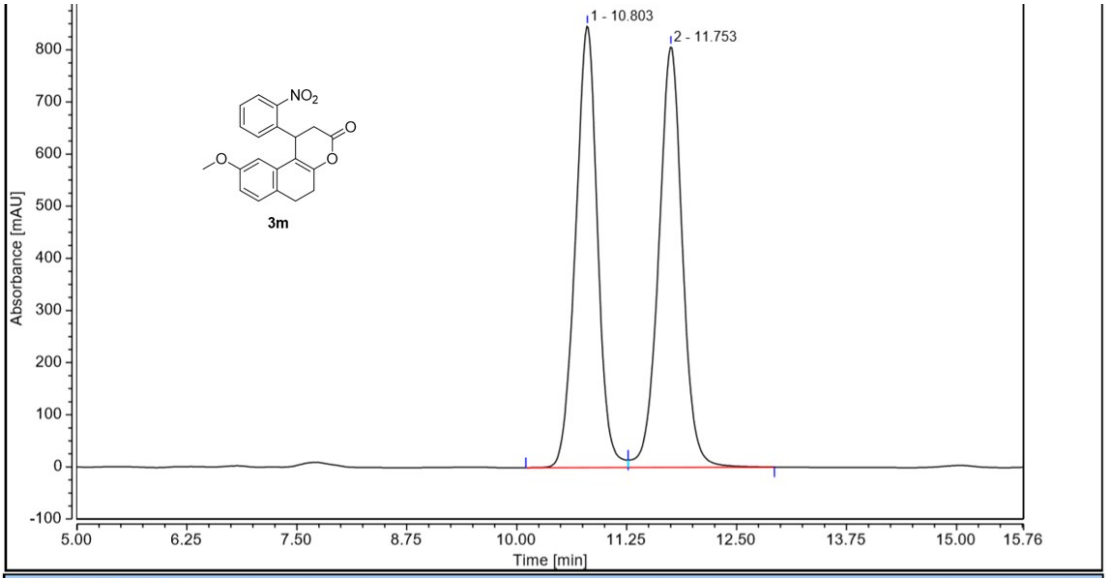
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		26.093	1038.040	1498.947	49.66	58.13	n.a.
2		28.053	1052.291	1079.832	50.34	41.87	n.a.
Total:			**2090.330**	**2578.779**	**100.00**	**100.00**	



Integration Results

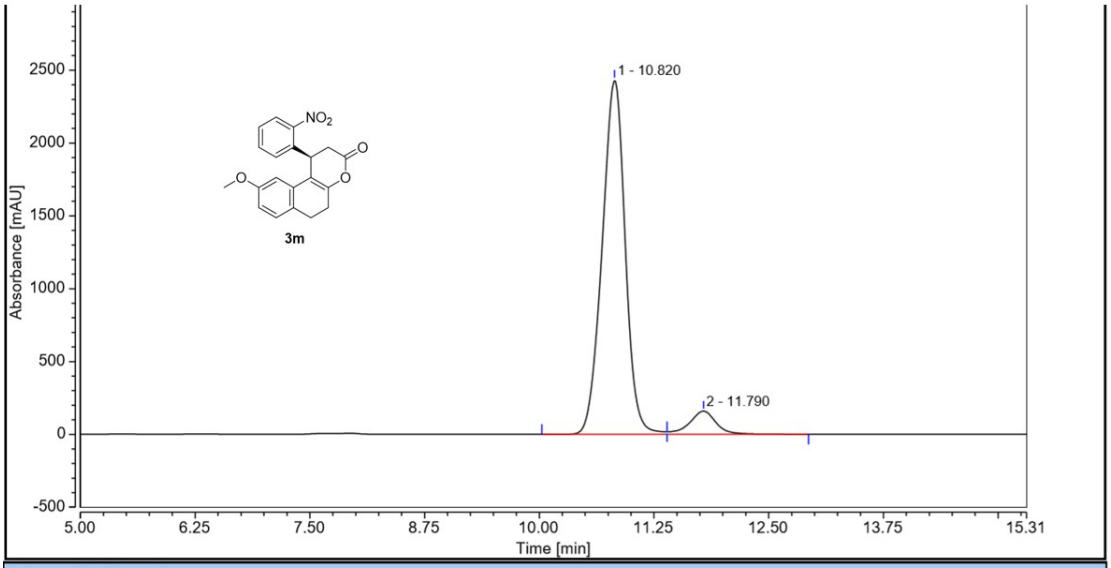
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		26.077	1308.654	1845.928	97.91	98.62	n.a.
2		28.170	27.887	25.895	2.09	1.38	n.a.
Total:			**1336.541**	**1871.823**	**100.00**	**100.00**	

HPLC analysis: 96% ee, [Daicel Chiralpak IC, n-hexane/2-propanol = 95/5, v = 0.6 mL/min, λ = 254 nm, t (major) = 26.0 min, t (minor) = 28.1 min].



Integration Results

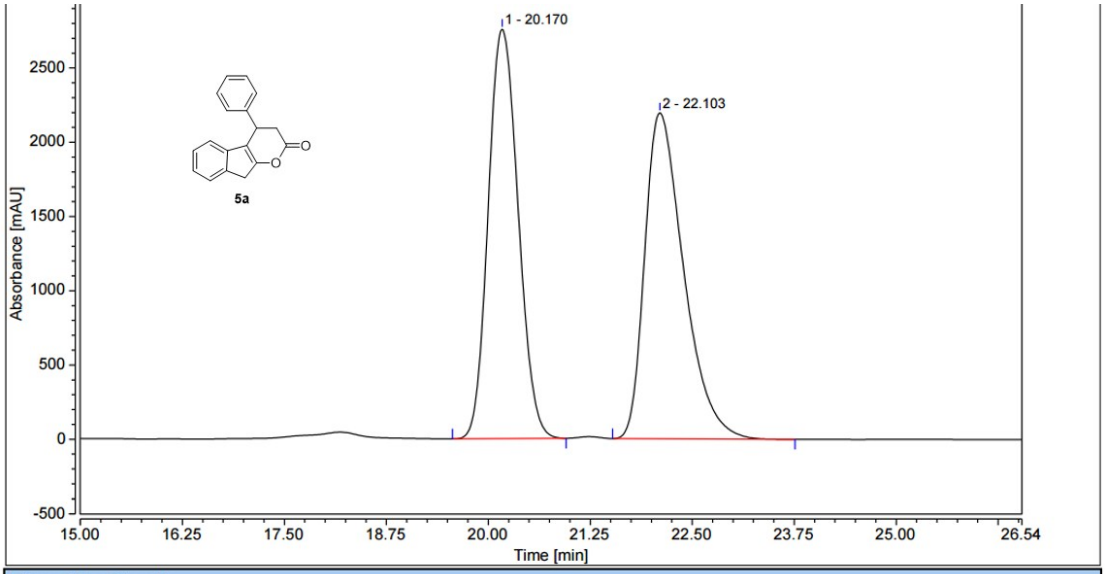
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		10.803	244.089	847.672	49.26	51.21	n.a.
2		11.753	251.386	807.509	50.74	48.79	n.a.
Total:			**495.476**	**1655.181**	**100.00**	**100.00**	



Integration Results

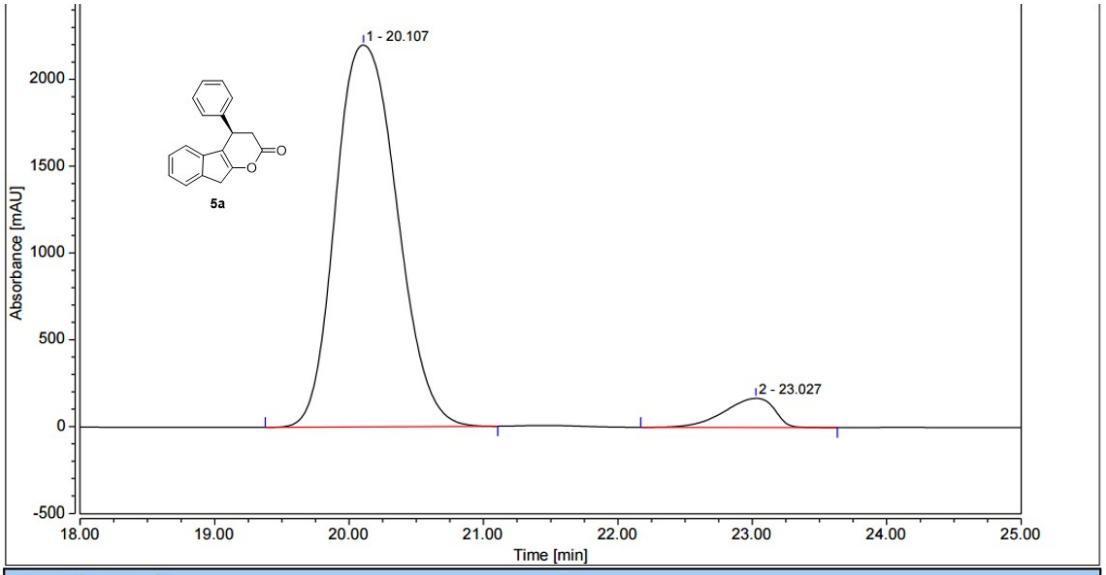
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		10.820	703.981	2431.577	92.88	93.86	n.a.
2		11.790	53.958	159.199	7.12	6.14	n.a.
Total:			**757.940**	**2590.777**	**100.00**	**100.00**	

HPLC analysis: 86% ee, [Daicel Chiralpak IA, n-hexane/2-propanol = 90/10, v = 0.8 mL/min, λ = 254 nm, t (major) = 10.8 min, t (minor) = 11.7 min].



Integration Results

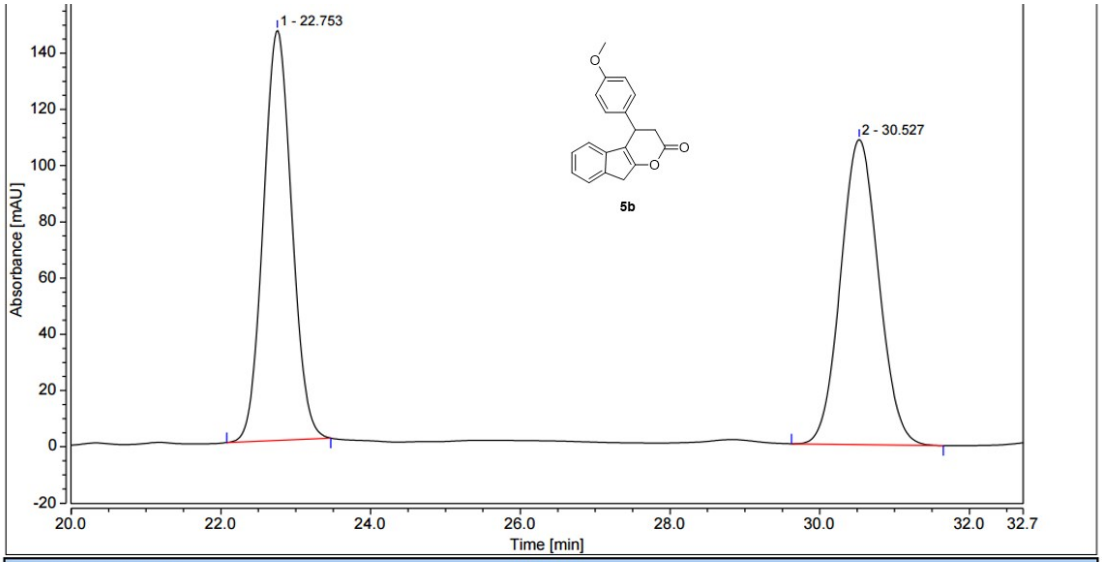
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		20.170	1173.611	2757.602	49.15	55.68	n.a.
2		22.103	1214.353	2195.305	50.85	44.32	n.a.
Total:			**2387.964**	**4952.907**	**100.00**	**100.00**	



Integration Results

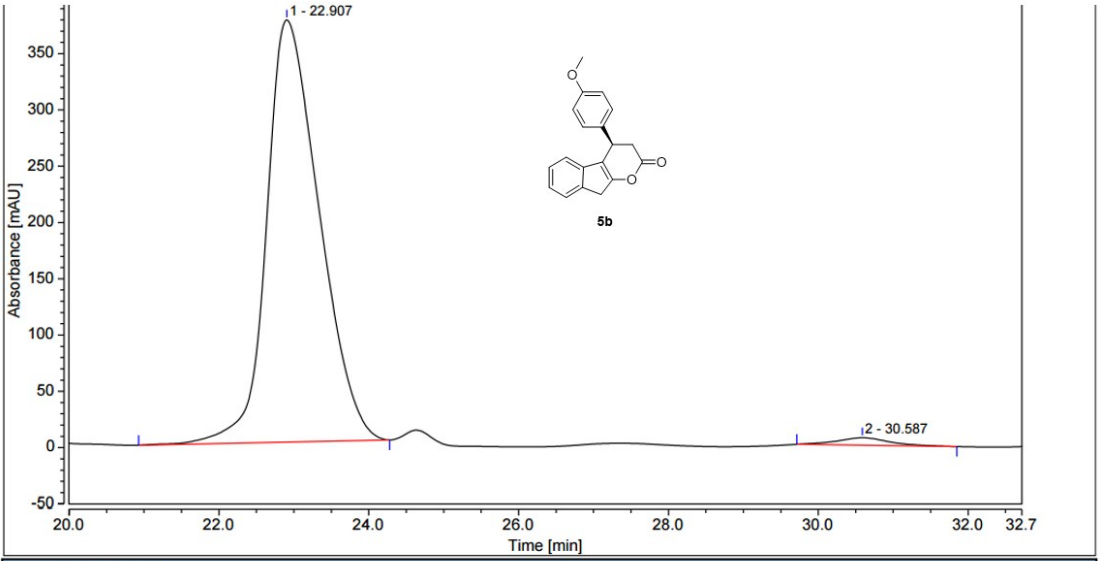
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		20.107	1170.767	2200.045	94.17	92.92	n.a.
2		23.027	72.433	167.671	5.83	7.08	n.a.
Total:			**1243.200**	**2367.716**	**100.00**	**100.00**	

HPLC analysis: 88% ee, [Daicel Chiralpak IC, n-hexane/2-propanol = 95/5, v = 0.7 mL/min, λ = 254 nm, t (major) = 20.5 min, t (minor) = 23.3 min].



Integration Results

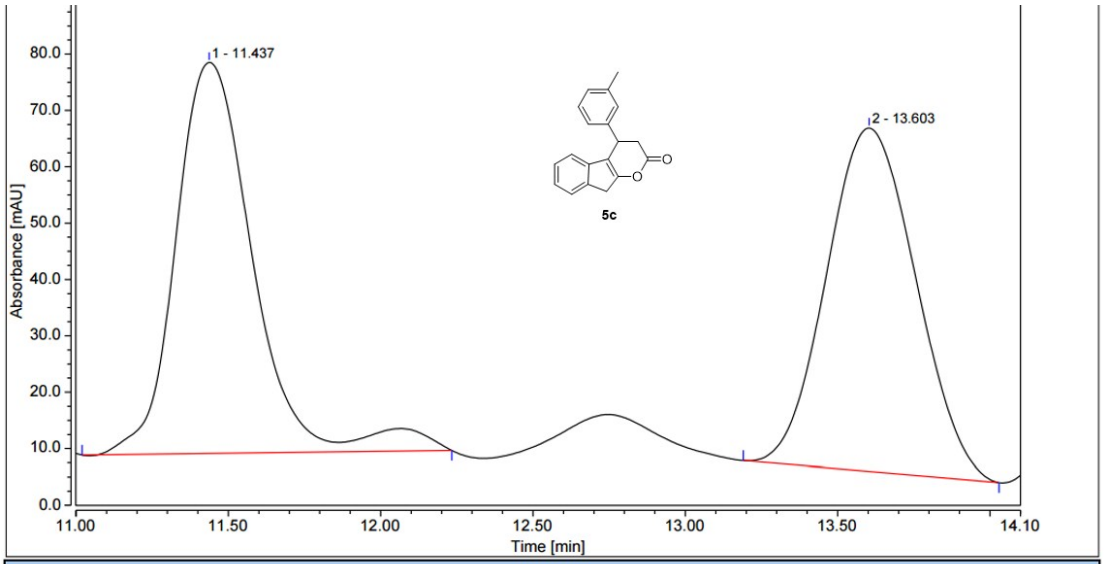
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		22.753	64.143	145.884	50.12	57.33	n.a.
2		30.527	63.838	108.574	49.88	42.67	n.a.
Total:			**127.981**	**254.458**	**100.00**	**100.00**	



Integration Results

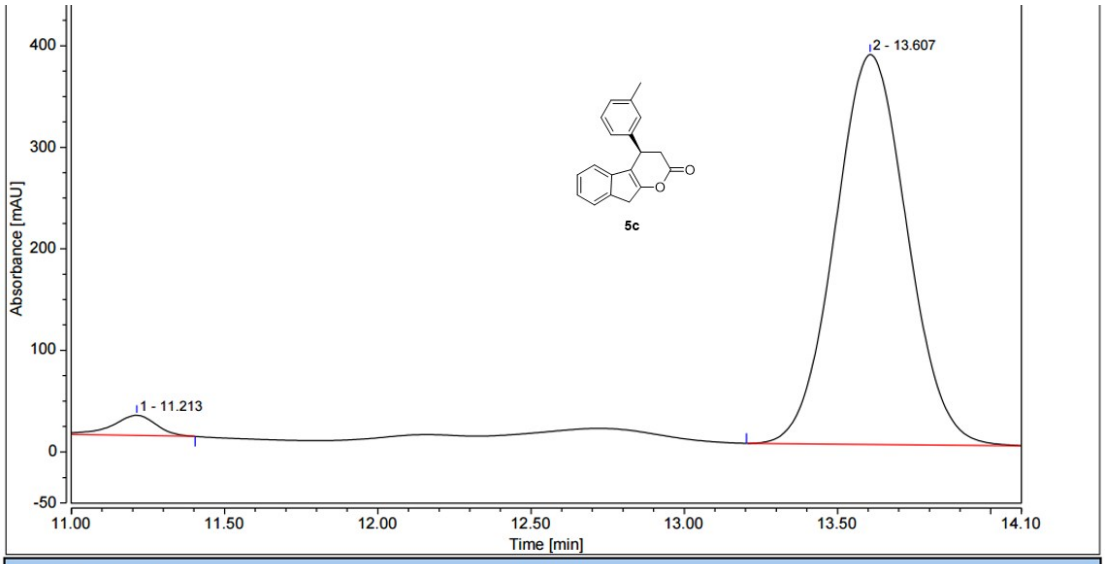
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		22.907	305.537	375.179	98.25	98.25	n.a.
2		30.587	5.446	6.671	1.75	1.75	n.a.
Total:			**310.983**	**381.850**	**100.00**	**100.00**	

HPLC analysis: 97% ee, [Daicel Chiralpak IC, n-hexane/2-propanol = 90/10, v = 0.8 mL/min, λ = 254 nm, t (major) = 22.9 min, t (minor) = 30.5 min].



Integration Results

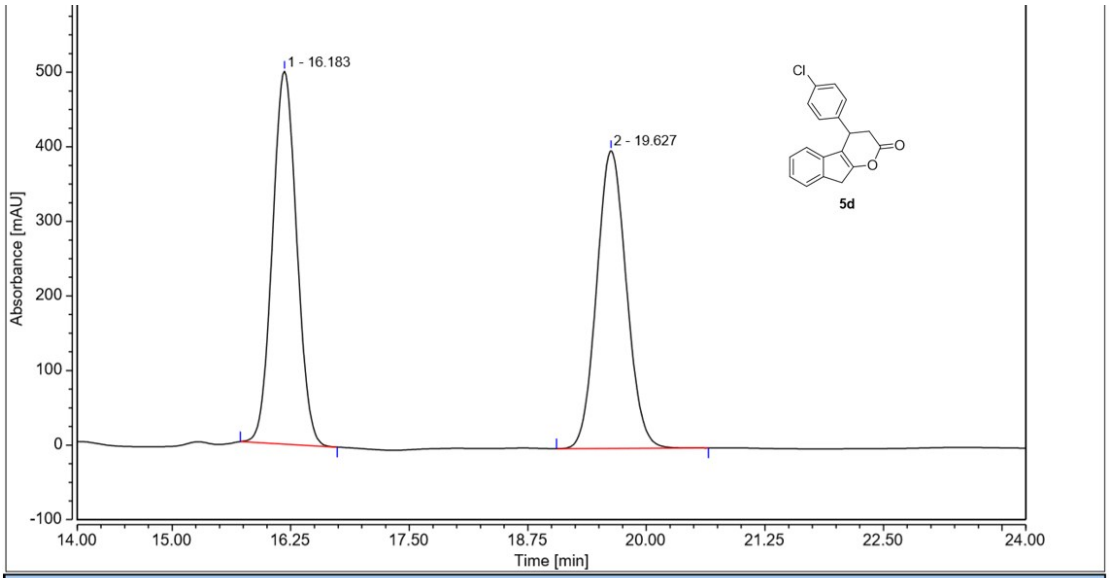
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		11.437	20.541	69.414	49.86	53.26	n.a.
2		13.603	20.656	60.905	50.14	46.74	n.a.
Total:			**41.197**	**130.319**	**100.00**	**100.00**	



Integration Results

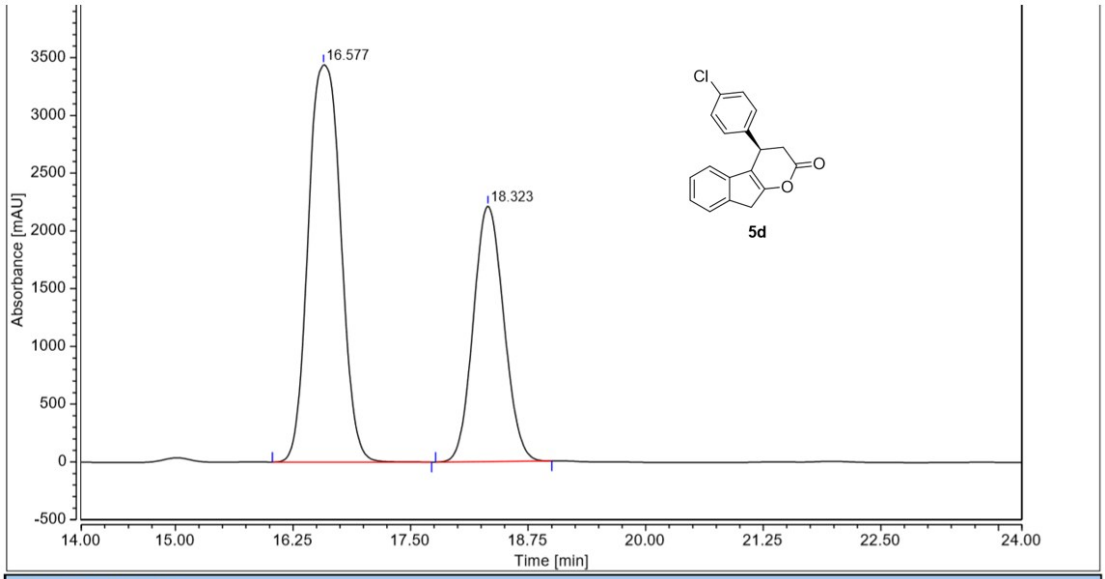
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		11.213	3.331	19.809	3.08	4.91	n.a.
2		13.607	104.689	384.003	96.92	95.09	n.a.
Total:			**108.020**	**403.812**	**100.00**	**100.00**	

HPLC analysis: 94% ee, [Daicel Chiralpak IC, n-hexane/2-propanol = 90/10, v = 0.8 mL/min, λ = 254 nm, t (major) = 13.6 min, t (minor) = 11.2 min].



Integration Results

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		16.183	148.269	500.310	50.52	55.59	n.a.
2		19.627	145.228	399.673	49.48	44.41	n.a.
Total:			**293.497**	**899.983**	**100.00**	**100.00**	



Integration Results

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		16.577	1379.490	3441.388	62.46	60.87	n.a.
2		18.323	829.200	2212.570	37.54	39.13	n.a.
Total:			**2208.690**	**5653.958**	**100.00**	**100.00**	

HPLC analysis: 25% ee, [Daicel Chiralpak IC, n-hexane/2-propanol = 90/10, v = 0.8 mL/min, λ = 254 nm, t (major) = 16.5 min, t (minor) = 18.3 min].