Supporting information

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General information

All solvents and reagents were purchased from commercial sources and used without further purification unless noted. Cinnamaldehyde was stored under nitrogen atmosphere. 4-fluorocinnamaldehyde was purified with column chromatography prior to use. ^{1}H NMR (400 MHz), ^{13}C NMR (101 MHz) and ^{19}F NMR (376 MHz) spectra were recorded on Varian 400. The chemical shifts for the ^{1}H NMR and ^{13}C NMR spectra were reported in parts per million (ppm) relative to the peaks of $CDCl_3$ for ^{1}H NMR at δ 7.26 ppm and for ^{13}C NMR at δ 77.16 ppm. Multiplicities were indicated by: s (singlet), d (doublet), t (triplet) and m (multiplet). All coupling constants (*J*) were reported in Hertz (Hz). FT-ATR-IR spectra were recorded on a Perkin-Elmer Spectrum Frontier infrared spectrometer with pike-GladiATR™ module and are reported in wavenumber (cm^{-1}) as follows: vs (very strong), s (strong), br (broad). High resolution mass spectra (HRMS) were performed on Sciex QSTARx1 mass spectrometer. Purification of the product was performed by an automated column chromatography Biotage Isolera™ Spectra One with Biotage SNAP®-10 g KP-sil columns. Thin layer chromatography (TLC) was performed on Merck TLC aluminium sheets precoated with silica gel 60 F_{254} and visualised with UV-light (254 nm). Chiral HPLC was performed on Varian 9050 with Diacel Chiralpak with AD-H or OD-H column using eluent: *n*-hexane/2-propanol with a flow rate of 1.0 ml/min and detection wavelength at 254 nm, except for compound **24** where a detection wavelength of 211 nm were used. Optical rotations were measured on a Perkin Elmer Polarimeter 341 LC using CH_2Cl_2 with a 10 cm cell (concentration (c) given in g/100 mL). Absolute configuration of the products was determined by comparing the optical rotation with known compounds. The racemic products used to determine the ee values were synthesised according to literature procedure.[1]

Representative general procedure

To a 10 ml pear shaped round bottom flask equipped with a magnetic stir bar were added NHC catalyst **3** (4.6 mg, 0.012 mmol), oxidant **1** (10.2 mg, 0.025 mmol), lithium acetate dihydrate (12.8 mg, 0.12 mmol) and acetylacetone (38.5 µl, 0.37 mmol) that was dissolved in toluene (2 mL). The solution was stirred for 5 minutes at ambient temperature before addition of cinnamaldehyde (15.8 µl, 0.125 mmol) and iron(II)phthalocyanine (1.4 mg, 0.002 mmol). One additional portion of iron(II)phthalocyanine (1.4 mg, 0.002 mmol) was added to the reaction mixture every third hour, for the total time of 6 h (3×0.002 mmol). The reaction mixture was stirred at 40 °C open to the atmosphere. Completion of the reaction was monitored *via* ^{1}H NMR. After completion the reaction mixture was purified with column chromatography using the biotage with ethyl acetate/petroleum ether (40–60 °C) solvent mixture (25 mL/min, 100% petroleum ether →4%→6%→13%→50% ethyl acetate in petroleum ether). The product was obtained as a white solid. The enantiomeric excess was determined by HPLC analysis using a chiral AD-H column with *n*-hexane/2-propanol (99:1).

Optimisation of reaction

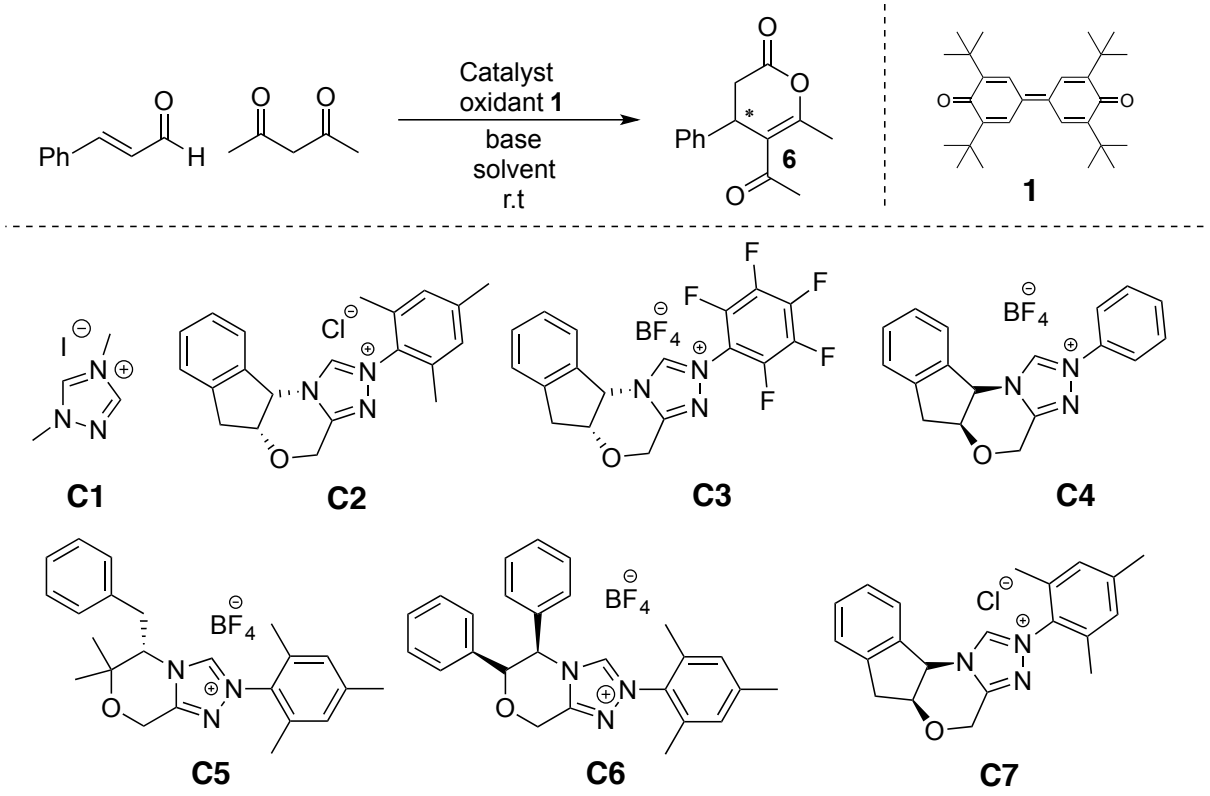


Table S1. Catalyst screen						
Entry[a]	**Cat**	**Base**	**Solvent**	**Time**	**Yield**	**ee**
1	C1	DBU	THF	3h	71%	0
2	C2	DBU	THF	28h	62%	-44.9
3	C3	DBU	THF	72h	-	-
4	C4	DBU	THF	3h	59%	18
5	C5	DBU	THF	3h	36%	-43
6		DBU	THF	22h	-	-
7	C6	DBU	THF	18h	55%	-47

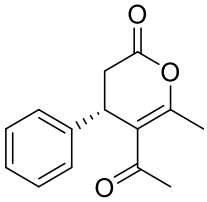
Table S2. Solvent screen						
Entry[a]	**Cat**	**Base**	**Solvent**	**Time**	**Yield**	**ee**
8	C2	DBU	Toluene	16h	56%	-62
9	C2	DBU	DCM	16h	62%	-24
10	C6	DBU	Toluene	16h	46%	-34
11	C6	DBU	DCM	16h	52%	-22
12	C2	DBU	MeCN	3h	54%	19
13	C2	DBU	Heptane	24h	-	-
14	C2	DBU	EtOAc	3h	55%	-27
15	C2	DBU	MeTHF	16h	58%	-52
16	C2	DBU	Anisole	16h	52%	-57

Table S3. Base screen						
Entry[a]	**Cat**	**Base**	**Solvent**	**Time**	**Yield**	**ee**
16	C2	DBU	Anisole	16h	52%	-57
17	C7	DABCO	Toluene	17h	60%	82
18	C7	Et_3N	Toluene	17h	60%	86
19	C7	K_2CO_3	Toluene	17h	42%	87
20	C7	DIPEA	Toluene	18h	34%	79
21	C7	Cs_2CO_3	Toluene	18h	57%	83
22	C7	Lithium acetate dihydrate	Toluene	20h	60%	94

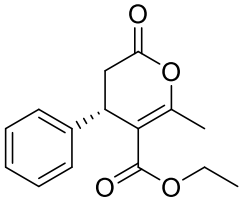
[a]To a 4 ml vial equipped with a magnetic stir bar were added NHC catalyst (0.1 eq.), solvent (3 mL) and base (0.5 eq.). The solution was stirred for 5 minutes at ambient temperature before addition of oxidant (1 eq.) and acetylacetone compound (1.5 eq.). After stirring for additional 5 minutes cinnamaldehyde (0.25 mmol) was added and the reaction was stirred at ambient temperature. The reaction was monitored *via* ^{1}H NMR and when the reaction was completed the mixture was purified with a biotage isolera system using ethyl acetate/petroleum ether (40– 60°C) solvent mixture (10 mL/min, 100% petroleum ether →4%→6%→13%→50% ethyl acetate in petroleum ether). The product was obtained as a white solid. The enantiomeric excess was determined by HPLC analysis on a chiral AD-H column (with *n*-hexane/2-propanol (99:1) as eluent).

Synthesis of methyl-(*R*)-4-acetyl-5-oxo-3-phenylhexanoate (24)

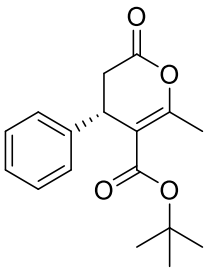
To a vial equipped with a magnetic stir bar was added (S)-5-acetyl-6-methyl-4-phenyl-3,4-dihydro-2*H*-pyran-2-one (18.2 mg, 0.08 mmol). To this were added a solution of 1,4-dimethyl-4*H*-1,2,4-triazole iodide (1.9 mg, 0.008 mmol) and 1,8-diazabicyclo[5.4.0.]undec-7-ene (1.3 mg, 0.009 mmol) in methanol (1 mL). The mixture was stirred at ambient temperature overnight. The volatiles were removed under reduced pressure yielding a yellow oil. The crude mixture was purified by column chromatography using a pipette column using petroleum ether (40–60 °C)/acetone (4:1) as eluent. The product was obtained as a white solid (19.7 mg, 0.08 mmol, 95% yield).



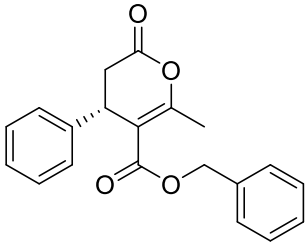
5-Acetyl-6-methyl-4-phenyl-3,4-dihydro-2*H*-pyran-2-one (6)[1]: Synthesised from cinnamaldehyde and acetylacetone according to the general procedure. Obtained as a white solid (18.2 mg, 0.079 mmol, 77% yield). The ee (94%) was measured by HPLC using chiral stationary phase [AD-H column, *n*-hexane/2-propanol (99:1), 1.0 mL/min), t_R = 32.704 min (major), 47.869 min (minor)]. $[\alpha]_D^{20}$ = +90.6 (c = 1, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36–7.31 (m, 2 H), 7.30–7.27 (m, 1 H), 7.16–7.13 (m, 2 H), 2.94 (dd, *J* = 15.6 Hz, 7.3 Hz, 1 H), 2.81 (dd, *J* = 15.8 Hz, 3.0 Hz, 1 H), 2.43 (d, *J* = 0.9 Hz, 3 H), 2.12 (s, 3 H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.7, 165.4, 160.0, 139.5, 129.2, 127.7, 126.4, 117.1, 38.6, 37.0, 29.6, 18.9.



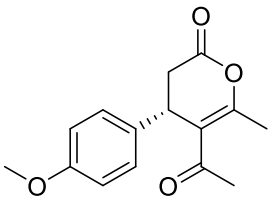
Ethyl 6-methyl-2-oxo-4-phenyl-3,4-dihydro-2*H*-pyran-5-carboxylate (15)[1]: Synthesised from cinnamaldehyde and ethyl acetoacetate according to the general procedure and obtained as a white solid (28.1 mg, 0.108 mmol, 86% yield). The ee (90%) was measured by HPLC using chiral stationary phase [OD-H column, *n*-hexane/2-propanol (90:10), 1.0 ml/min), t_R = 7.366 min (major), 13.211 min (minor)]. $[\alpha]_D^{20}$ = +120.2 (c = 0.5, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33–7.27 (m, 2 H), 7.25–7.23 (m, 1 H), 7.16–7.12 (m, 2 H), 4.28–4.23 (m, 1 H), 4.13 (q, *J* = 6.9 Hz, 2 H), 2.95 (dd, *J* = 15.9 Hz, 7.6 Hz, 1 H), 2.83 (dd, *J* = 15.9 Hz, 2.4 Hz, 1 H), 2.5 (d, *J* = 1.0 Hz, 3 H), 1.19 (t, *J* = 7.1 Hz, 3 H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.3, 166.1, 161.5, 140.8, 129.2, 127.6, 126.7, 110.2, 61.0, 38.0, 36.5, 19.0, 14.2.



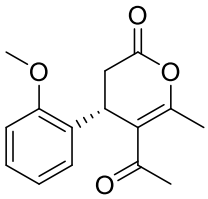
Tert-butyl 6-methyl-2-oxo-4-phenyl-3,4-dihydro-2*H*-pyran-5-carboxylate (21)[2]: Synthesised from cinnamaldehyde and *t*-butyl acetoacetate according to the general procedure and obtained as a yellow oil (26.2 mg, 0.091 mmol, 73% yield). The ee (92%) was measured by HPLC using chiral stationary phase [OD-H, *n*-hexane/2-propanol (85:5), 1.0 ml/min), t_R = 5.117 min (major), 8.129 min (minor)]. $[\alpha]_D^{20}$ = +78.2 (c = 0.5, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32–7.27 (m, 2 H), 7.25–7.22 (m, 1 H), 7.15–7.11 (m, 2 H), 7.19–7.15 (m, 1 H), 2.91 (dd, *J* = 15.7 Hz, 7.6 Hz, 1 H), 2.78 (dd, *J* = 15.9 Hz, 2.9 Hz, 1 H), 2.43 (d, *J* = 1.1 Hz, 3 H), 1.35 (9 H, s). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 165.3, 160.3, 141.1, 129.1, 127.5, 126.7, 111.5, 81.6, 38.4, 36.6, 28.2, 18.8.



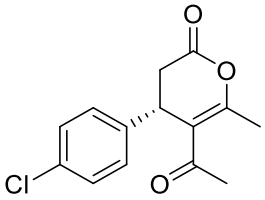
Benzyl 6-methyl-2-oxo-4-phenyl-3,4-dihydro-2H-pyran-5-carboxylate (22)[3]: Synthesised from cinnamaldehyde and benzyl acetylacetone according to the general procedure and obtained as a yellow oil (29.8 mg, 0.092 mmol, 74% yield). The ee (86%) was measured by HPLC using chiral stationary phase [OD-H, *n*-hexane/2-propanol (95:5), 1.0 ml/min), t_R = 15.089 min (major), 23.927 min (minor)]. $[\alpha]_D^{20}$ = +12.1 (c = 1, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) 7.38–7.26 (m, 6 H), 7.17–7.09 (m, 4 H), 5.19–5.06 (m, 2 H), 4.28 (d, *J* = 7.4 Hz, 1 H), 2.95 (dd, *J* = 15.9 7.7 Hz, 1H), 2.82 (dd, *J* = 15.8, 2.2 Hz, 1H), 2.49 (d, *J* = 1.0 Hz, 3H) δ ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.1, 165.9, 162.2, 140.7, 135.7, 129.2, 128.6, 128.3, 128.0, 127.7, 126.8, 109.8, 66.7, 38.1, 36.5, 19.1.



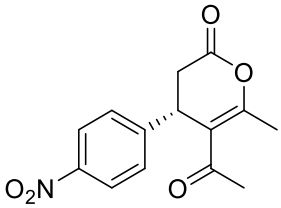
5-acetyl-4-(4-methoxyphenyl)-6-methyl-3,4-dihydro-2*H*-pyran-2-one (10)[1]**:** Synthesised from 4-methoxycinnamaldehyde and acetylacetone according to the general procedure, however, with 0.65 eq of lithium acetate dihydrate instead. Obtained as a yellow oil (25.4 mg, 0.098 mmol, 76% yield). The ee (85%) was measured by HPLC using chiral stationary phase [OD-H, *n*-hexane/2-propanol (90:10), 1.0 ml/min), t_R = 20.081 min (major), 23.806 min (minor)]. $[\alpha]_D^{20}$ = +27 (c = 0.5, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.08–7.03 (m, 2 H), 6.88–6.82 (m, 2 H), 4.11–4.07 (m, 1 H), 3.78 (s, 3 H), 2.94 (dd, *J* = 15.6 Hz, 7.2 Hz, 1 H), 2.78 (dd, *J* = 15.6 Hz, 2.7 Hz, 1 H), 2.42 (d, *J* = 1.0 Hz, 3 H), 2.12 (s, 3 H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.9, 165.6, 159.8, 159.0, 131.4, 127.6, 117.4, 114.6, 55.1, 38.0, 37.3, 29.5, 18.9.



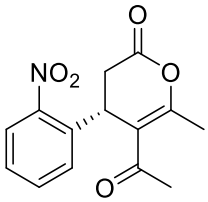
5-acetyl-4-(2-methoxyphenyl)-6-methyl-3,4-dihydro-2*H*-pyran-2-one (11)[3]**:** Synthesised from 2-methoxycinnamaldehyde and acetylacetone according to the general procedure, however, with exclusion of light using aluminium foil and obtained as a yellow solid (29.7 mg, 0.114 mmol, 91% yield). The ee (81%) was measured by HPLC using chiral stationary phase [AD-H, *n*-hexane/2-propanol (99:1), 1.0 ml/min), t_R = 26.556 min (major), 24.388 min (minor)]. $[\alpha]_D^{20}$ = +46.5 (c = 1, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29–7.23 (m, 1 H), 7.00–6.95 (m, 1 H), 6.94–6.86 (m, 2 H), 4.53–4.46 (m, 1 H), 3.86 (s, 3 H), 2.92–2.82 (m, 2 H), 2.42 (s, 3 H), 2.08 (s, 3 H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.6, 166.4, 160.6, 129.3, 127.3, 127.1, 121.1, 116.4, 110.9, 55.3, 35.0, 32.9, 29.4, 19.1.



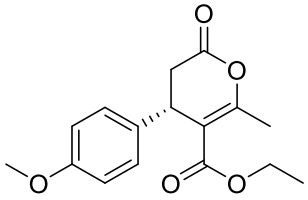
5-acetyl-4-(4-chlorophenyl)-6-methyl-3,4-dihydro-2*H*-pyran-2-one (9)[2]: Synthesised from 4-chlorocinnamaldehyde and acetylacetone according to the general procedure, however, with 0.65 eq of lithium acetate dihydrate and obtained as a white solid (26.5 mg, 0.100 mmol, 80% yield). The ee (91%) was measured by HPLC using chiral stationary phase [AD-H, *n*-hexane/2-propanol (90:10), 1.0 ml/min), t_R = 10.712 min (major), 9.989 min (minor)]. $[\alpha]_D^{20}$ = + 17.2 (c = 1, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34–7.29 (m, 2 H), 7.11–7.07 (m, 2 H), 4.16–4.13 (m, 1 H), 2.96 (dd, *J* = 15.7 Hz, 7.2 Hz, 1 H), 2.81 (dd, *J* = 15.7 Hz, 2.5 Hz, 1 H), 2.43 (d, *J* = 0.9 Hz, 3 H), 2.14 (3 H, s). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.2, 165.1, 160.4, 138.1, 133.7, 129.5, 127.9, 117.0, 38.2, 36.8, 29.7, 19.1.



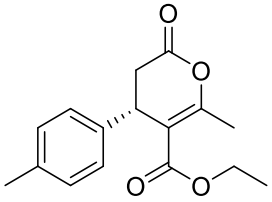
5-acetyl-6-methyl-4-(4-nitrophenyl)-3,4-dihydro-2*H*-pyran-2-one (12)[2]: Synthesized from 4-nitrocinnamaldehyde and acetylacetone according to the general procedure and obtained as a yellow solid (22.4 mg, 0.081 mmol, 65% yield). The ee (90%) was measured by HPLC using chiral stationary phase [OD-H, *n*-hexane/2-propanol (80:20), 1.0 ml/min), t_R = 25.407 min (major), 36.507 min (minor)]. $[\alpha]_D^{20}$ = + 19.3 (c = 1, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24–8.17 (m, 2 H), 7.37–7.31 (m, 2 H), 4.36–4.30 (m, 1 H), 3.02 (dd, *J* = 15.9 Hz, 7.4 Hz, 1 H), 2.85 (dd, *J* = 15.9 Hz, 2.4 Hz, 1 H), 2.48 (d, *J* = 1 Hz, 3 H), 2.21 (s, 3 H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.7, 164.9, 161.4, 147.4, 127.9, 124.8, 117.3, 38.5, 36.5, 30.4, 19.7.



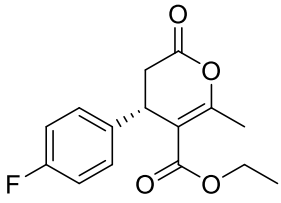
5-acetyl-6-methyl-4-(2-nitrophenyl)-3,4-dihydro-2*H*-pyran-2-one (13)[1]: Synthesised from 2-nitrocinnamaldehyde and acetylacetone according to the general procedure and obtained as a yellow solid (25.1 mg, 0.091 mmol, 73% yield). The ee (90%) was measured by HPLC using chiral stationary phase [AD-H, *n*-hexane/2-propanol (90:10), 1.0 ml/min), t_R =15.887 min (major), 14.644 min (minor)]. $[\alpha]_D^{20}$ = +316.8 (c =1, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (m, 1 H), 7.59 (m, 1 H), 7.48 (m, 1 H), 7.22 (m, 1 H), 4.88–4.84 (m, 1 H), 3.08 (dd, *J* = 16.1 Hz, 7.5 Hz, 1 H), 3.01 (dd, *J* = 16.1 Hz, 2.6 Hz, 1 H), 2.48 (d, *J* = 1.09, 3 H), 2.09 (s, 3 H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.8, 165.1, 162.1, 148.8, 134.2, 129.3, 128.3, 125.9, 116.6, 35.9, 34.3, 29.9, 19.5.



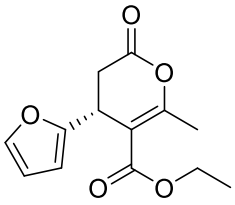
Ethyl 4-(4-methoxyphenyl)-6-methyl-2-oxo-3,4-dihydro-2*H*-pyran-5-carboxylate (16)[4]: Synthesised from 4-methoxycinnamaldehyde and ethyl acetoacetate according to the general procedure and obtained as a yellow oil (28.7 mg, 0.01 mmol, 79% yield). The ee (85%) was measured by HPLC using chiral stationary phase [OD-H, *n*-hexane/2-propanol (90:10), 1.0 ml/min), t_R = 9.677 min (major), 14.589 min (minor)]. $[\alpha]_D^{20}$ = +73.9 (c = 1, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.08–7.03 (m, 2 H), 6.85–6.79 (m, 2 H), 4.23–4.18 (m, 1 H), 4.14 (q, *J* = 7.1 Hz, 2 H), 3.77 (s, 3 H), 2.92 (dd, *J* = 15.8 Hz, 7.5 Hz, 1 H), 2.79 (dd, *J* = 15.8 Hz, 2.0 Hz, 1 H), 2.46 (s, 3 H), 1.20 (t, *J* = 7.1 Hz, 3 H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 166.2, 161.2, 159.0, 132.8, 127.8, 114.5, 110.5, 61.0, 55.4, 37.2, 36.7, 19.0, 14.2.



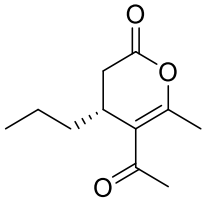
Ethyl 6-methyl-2-oxo-4-(*p*-tolyl)-3,4-dihydro-2*H*-pyran-5-carboxylate (17)[4]: Synthesised from 4-methylcinnamaldehyde and ethyl acetoacetate according to the general procedure and obtained as a yellow oil (18.6 mg, 0.068 mmol, 54% yield). The ee (87%) was measured by HPLC using chiral stationary phase [OD-H, *n*-hexane/2-propanol (90:10), 1.0 ml/min), t_R = 6.608 min (major), 10.189 min (minor)]. $[\alpha]_D^{20}$ = +113.7 (c = 1, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.12–7.07 (m, 2 H), 7.04–6.99 (m, 2 H), 4.24–4.19 (m, 1 H), 4.13 (qd, *J* = 7.1 Hz, 2.8 Hz, 2 H), 2.92 (dd, *J* = 15.8 Hz, 7.5 Hz, 1 H), 2.80 (dd, *J* = 15.8 Hz, 2.3 Hz, 1 H), 2.46 (s, 3 H), 2.30 (s, 3 H), 1.20 (t, *J* = 7.1 Hz, 3 H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.4, 166.2, 161.3, 137.7, 137.3, 129.8, 126.6, 110.4, 61.0, 37.6, 36.6, 21.2, 19.0, 14.2.



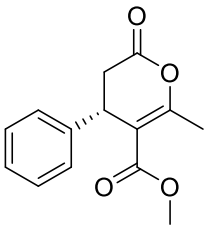
Ethyl 4-(4-fluorophenyl)-6-methyl-2-oxo-3,4-dihydro-2*H*-pyran-5-carboxylate (18)[4]: Synthesised from 4-fluorocinnamaldehyde and ethyl acetoacetate according to the general procedure and obtained as a yellow solid (20.4 mg, 0.073 mmol, 63% yield). The ee (90%) was measured by HPLC using chiral stationary phase [OD-H, *n*-hexane/2-propanol (95:5), 1.0 ml/min), t_R = 9.587 min (major), 20.219 min (minor)]. $[\alpha]_D^{20}$ = +139.3 (c = 1, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14–7.07 (m, 2 H), 7.03–6.94 (m, 2 H), 4.27–4.22 (m, 1 H), 4.14 (q, *J* = 7.1 Hz, 2 H), 2.94 (dd, *J* = 15.8 Hz, 7.6 Hz, 1 H), 2.80 (dd, *J* = 15.8 Hz, 2.2 Hz, 1 H), 2.47 (d, *J* = 1.0 Hz, 3 H), 1.20 (t, *J* = 7.1 Hz, 3 H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.0, 166.0, 162.2 (d, $^1J_{C\text{-}F}$ = 246 Hz), 161.6, 136.5 (d, $^4J_{C\text{-}F}$ = 3.4 Hz), 128.4 (d, $^3J_{C\text{-}F}$ = 8.1 Hz), 116.1 (d, $^2J_{C\text{-}F}$ = 21.5 Hz), 110.1, 61.1, 37.3, 36.6, 19.1, 14.2. ^{19}F NMR (376 MHz, $CDCl_3$) δ −115.1.



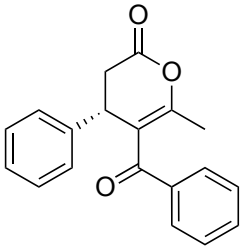
Ethyl 4-(furan-2-yl)-6-methyl-2-oxo-3,4-dihydro-2*H*-pyran-5-carboxylate (19): Synthesised from 3-(2-furyl)acrolein and ethyl acetoacetate according to the general procedure and obtained as a yellow oil (19.5 mg, 0.078 mmol, 61% yield). The ee (90%) was measured by HPLC using chiral stationary phase [OD-H, *n*-hexane/2-propanol (90:10), 1.0 ml/min), t_R = 7.622 min (major), 12.549 min (minor)]. $[\alpha]_D^{20}$ = +59 (c = 1, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33–7.27 (m, 1 H), 6.29–6.25 (m, 1 H), 6.16–5.76 (m, 1 H), 4.43–4.30 (m, 1 H), 4.31–3.97 (m, 2 H), 3.00 (dd, *J* = 16.0 Hz, 1.9 Hz, 1 H), 2.83 (dd, *J* = 16.0 Hz, 7.2 Hz, 1 H), 2.41 (s, 3 H), 1.29 (t, *J* = 7.1 Hz, 3 H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.1, 165.8, 162.1, 142.5, 110.4, 108.2, 106.2, 61.1, 33.4, 31.7, 19.1, 14.3. HRMS (ESI) calcd for $C_{13}H_{14}O_5$ $[M+H]^+$: Exact Mass: 251.0919, Found: 251.0928. FTIR-ATR (cm^{-1}): 2983.1 (br), 1787.4 (s), 1707.2 (s), 1648.7 (s), 1282 (s), 1241 (s), 1115.6 (vs), 1072.4 (vs).



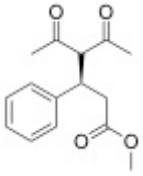
5-acetyl-6-methyl-4-propyl-3,4-dihydro-2*H*-pyran-2-one (14)[2]: Synthesised from *trans*-2-hexen-1-al and acetylacetone according to the general procedure and obtained as a yellow oil (18.7 mg, 0.095 mmol, 65% yield). The ee (83%) was measured by HPLC using chiral stationary phase [OD-H, *n*-hexane/2-propanol (90:10), 1.0 ml/min), t_R = 8.409 min (major), 9.304 min (minor)]. $[\alpha]_D^{20}$ = +16.2 (c = 0.5, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.97–2.83 (m, 1 H), 2.72 (dd, *J* = 15.9 Hz, 2.0 Hz, 1 H), 2.58 (dd, *J* = 15.9 Hz, 6.4 Hz, 1 H), 2.34 (s, 3 H), 2.27 (s, 3H). 1.49–1.19 (m, 4 H), 0.90 (t, *J* = 7.0 Hz, 3 H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.9, 167.2, 159.0, 120.5, 35.7, 33.2, 32.2, 30.4, 20.0, 19.5, 14.0.



Methyl 6-methyl-2-oxo-4-phenyl-3,4-dihydro-2*H*-pyran-5-carboxylate (20)[4]: Synthesised from cinnamaldehyde and methyl acetoacetate according to the general procedure and obtained as a off-white solid (24.3 mg, 0.099 mmol, 79% yield). The ee (95%) was measured by HPLC using chiral stationary phase [AD-H column, *n*-hexane/2-propanol (99:1), 1.0 ml/min), t_R = 12.251 min (major), 11.816 min (minor)]. $[\alpha]_D^{20}$ = +157.8 (c = 1, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33–7.22 (m, 3 H), 7.15–7.11 (m, 2 H), 4.28–4.24 (m, 1 H), 3.68 (s, 3 H), 2.94 (dd, *J* = 15. 8 Hz, 7.5 Hz, 1 H), 2.83 (dd, *J* = 15.8 Hz, 2.2 Hz, 1 H), 2.48 (d, *J* = 0.9 Hz, 3 H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.6, 166.1, 161.9, 140.5, 129.2, 127.7, 126.7, 109.8, 52.1, 37.9, 36.6, 19.1.



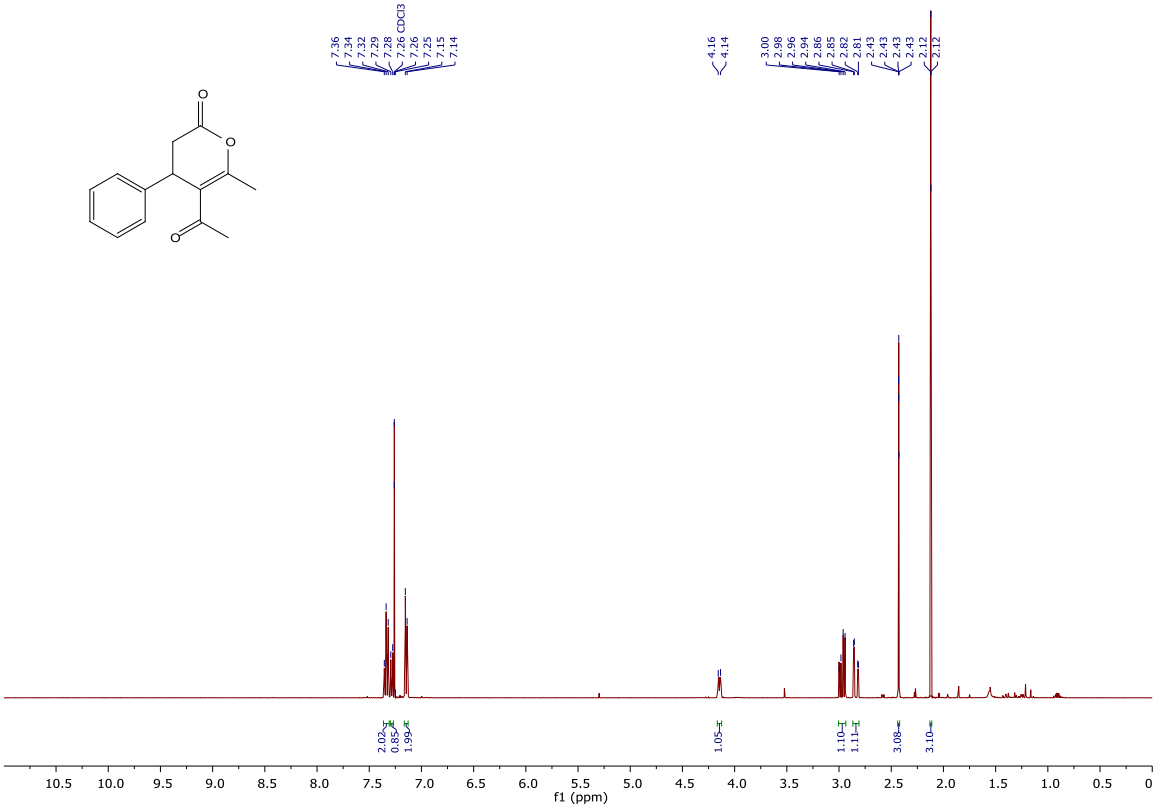
5-benzoyl-6-methyl-4-phenyl-3,4-dihydro-2*H*-pyran-2-one (23)[1]: Synthesized from cinnamaldehyde and 1-phenyl-1,3-butanedione according to the general procedure and obtained as a yellow oil (22.7 mg, 0.08 mmol, 62% yield). The ee (87%) was measured by HPLC using chiral stationary phase [OD-H column, *n*-hexane/2-propanol (85:15), 1.0 ml/min), t_R = 10.244 min (major), 12.114 min (minor)]. $[\alpha]_D^{20}$ = +30.7 (c = 1, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65-7.60 (m, 2 H), 7.55-7.49 (m, 1 H), 7.42-7.37 (m, 2 H), 7.29-7.27 (m, 1 H), 7.25-7.18 (m, 2 H) 7.16-7.12 (m, 2 H), 4.36-4.29 (m, 1 H), 3.07 (dd, *J*= 16.0, 7.5 Hz, 1 H), 2.93 (dd, *J*= 16.0, 3.5 Hz, 1 H), 1.90 (d, *J*= 1.90 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.0, 166.7, 154.9, 140.1, 138.6, 133.2, 129.3, 129.0, 128.9, 127.8, 126.9, 117.9, 39.6, 36.4, 19.2.



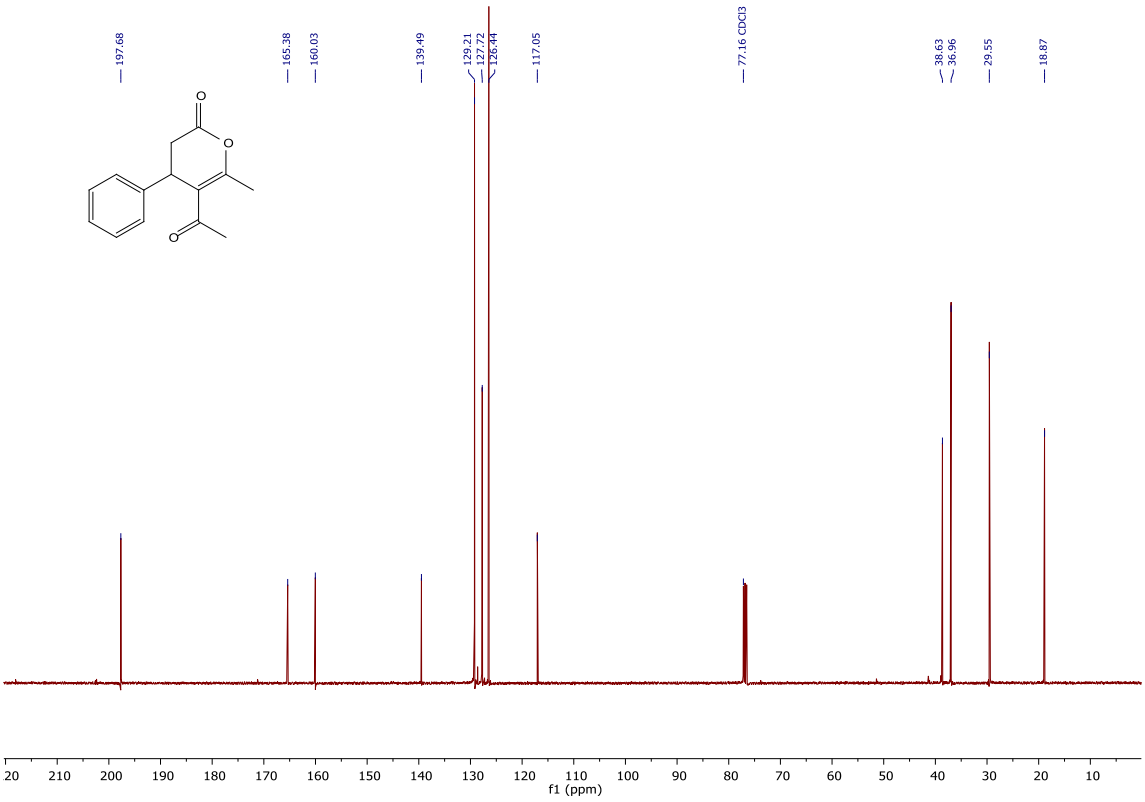
Methyl (*R*)-4-acetyl-5-oxo-3-phenylhexanoate (24)[5]: Obtained as a white solid (19.7 mg, 0.08 mmol, 95% yield). The ee (93%) was measured by HPLC using chiral stationary phase [OJ column, *n*-hexane/2-propanol (80:20), 1.0 ml/min), t_R = 18.583 min (minor), 30.938 min (major)] $[\alpha]_D^{20}$ = −96 (c = 0.28, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32–7.26 (m, 3H), 7.23–7.16 (m, 3 H), 4.28–4.21 (m, 1H), 4.01–3.93 (m, 1 H), 3.52 (s, 3 H), 2.59 (dd, *J* = 14.2, 6.9 Hz, 1H), 2.26 (s, 3 H), 1.83 (s, 3 H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 203.0, 202.8, 171.6, 139.8, 129.0, 128.1, 127.6, 74.2, 51.8, 41.7, 39.3, 30.0, 29.7.

NMR spectra of compounds

^{1}H NMR ($CDCl_3$)



^{13}C NMR ($CDCl_3$)



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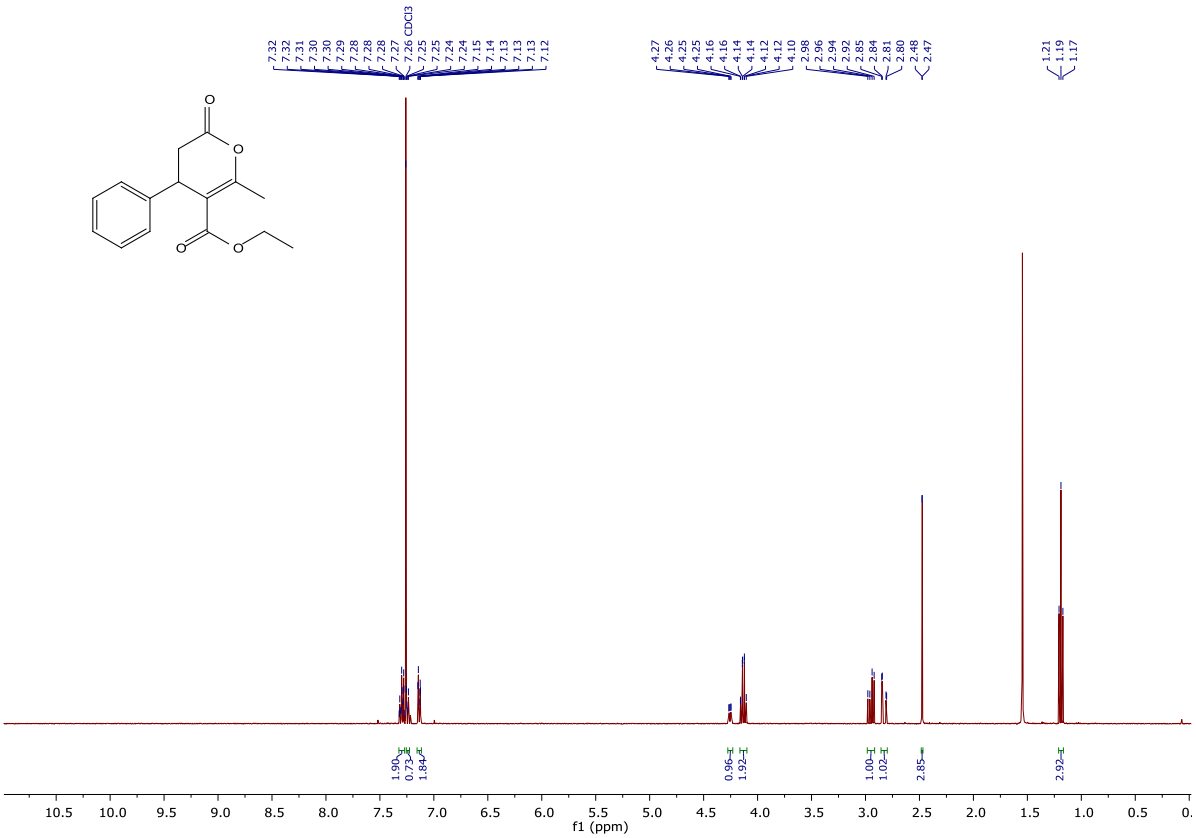
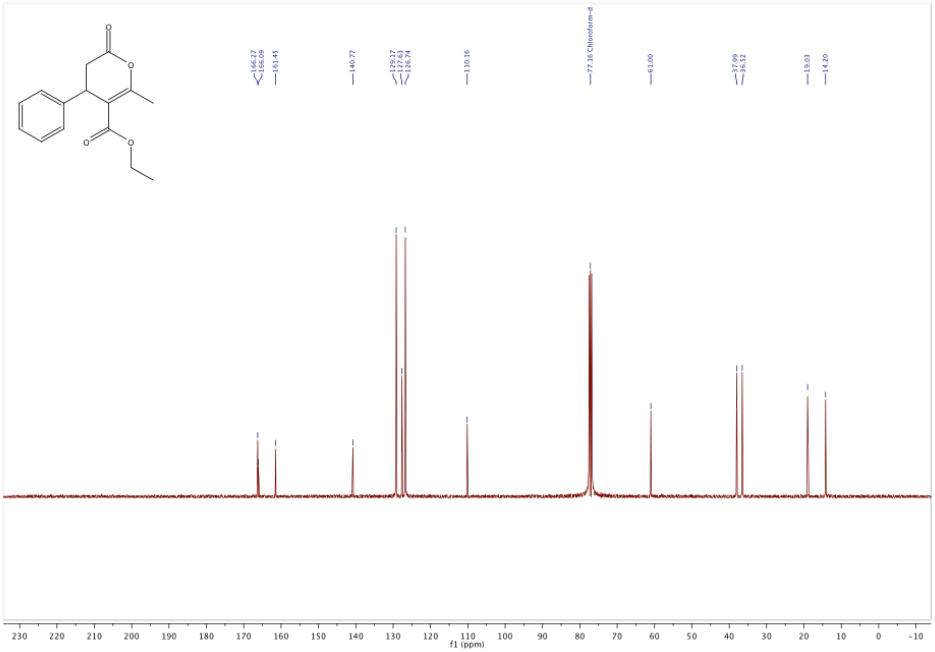


^{13}C NMR ($CDCl_3$)



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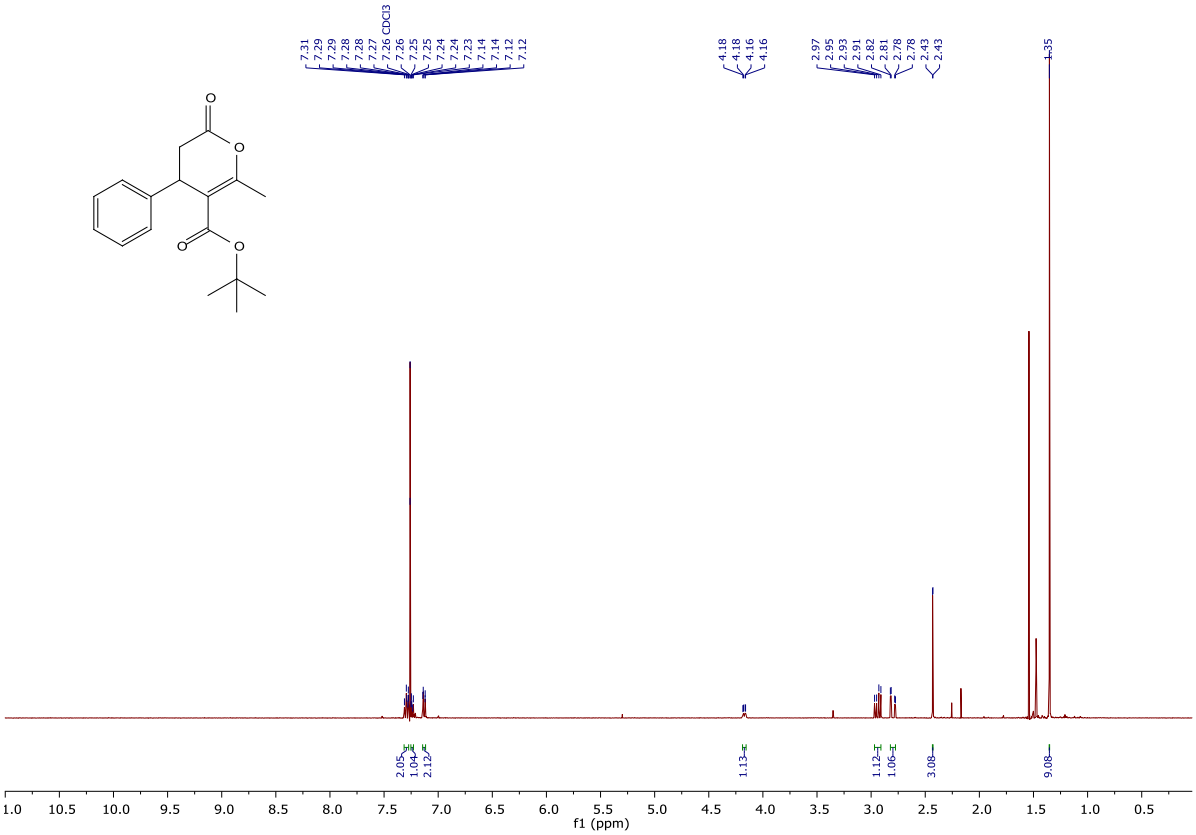

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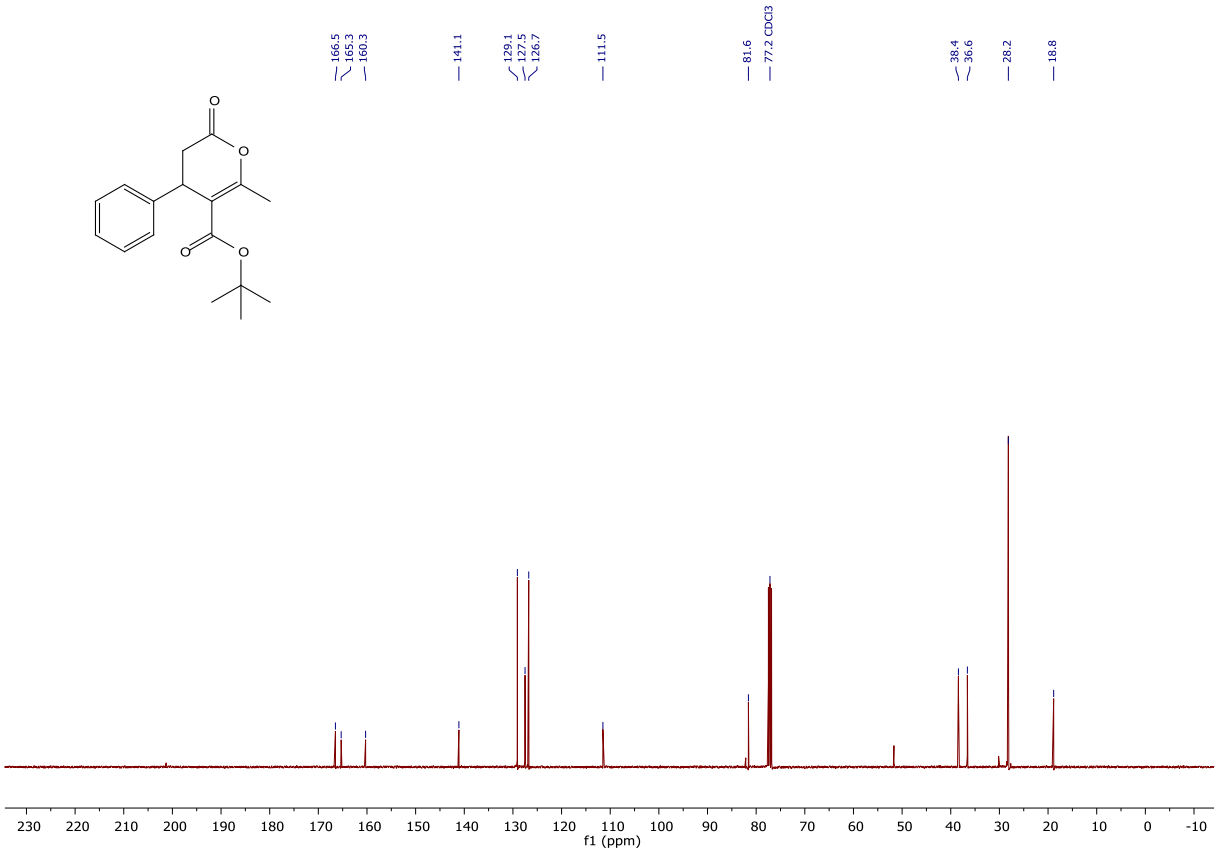

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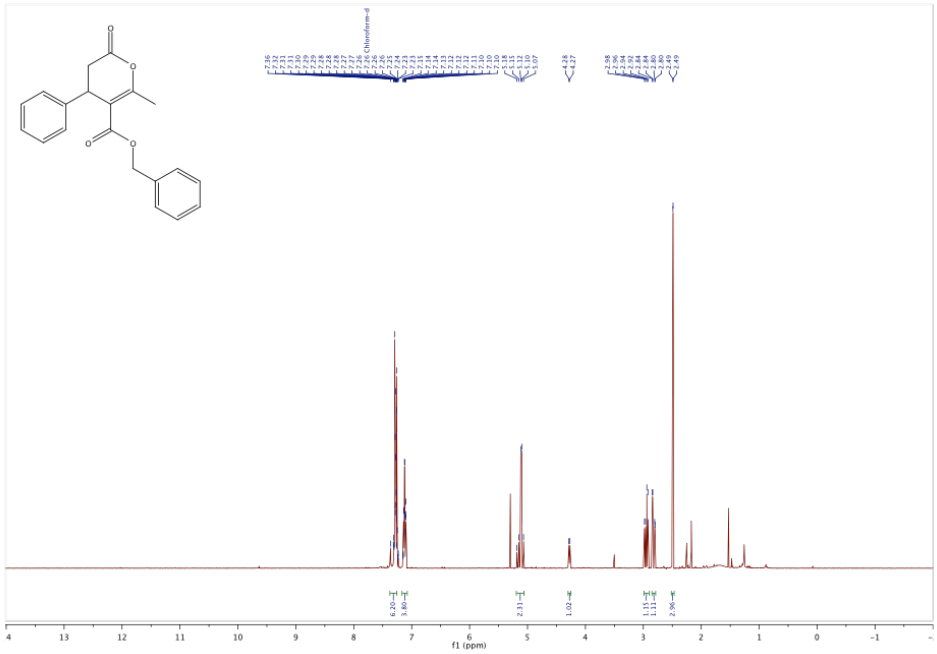

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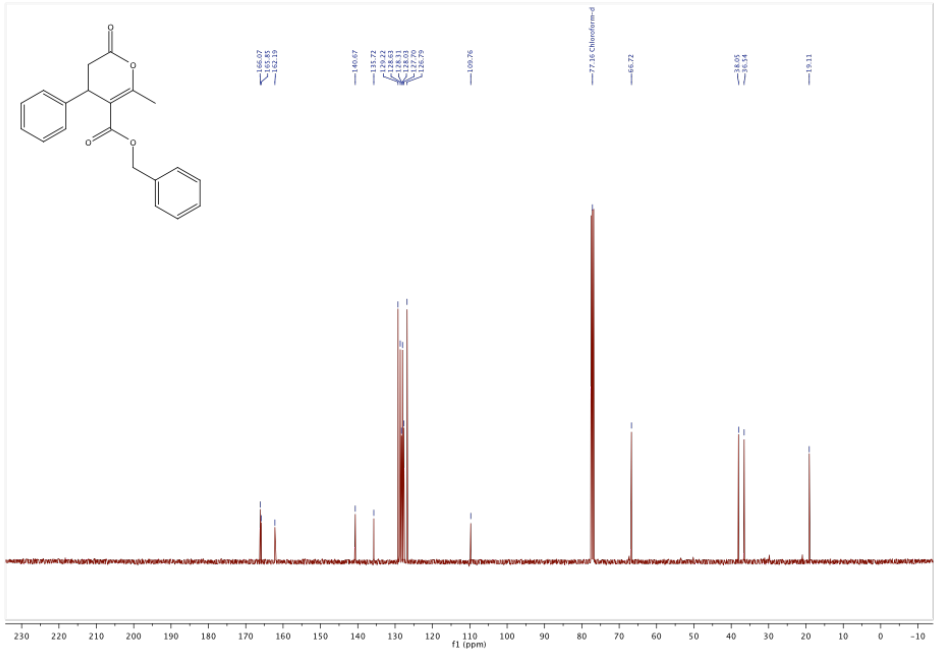

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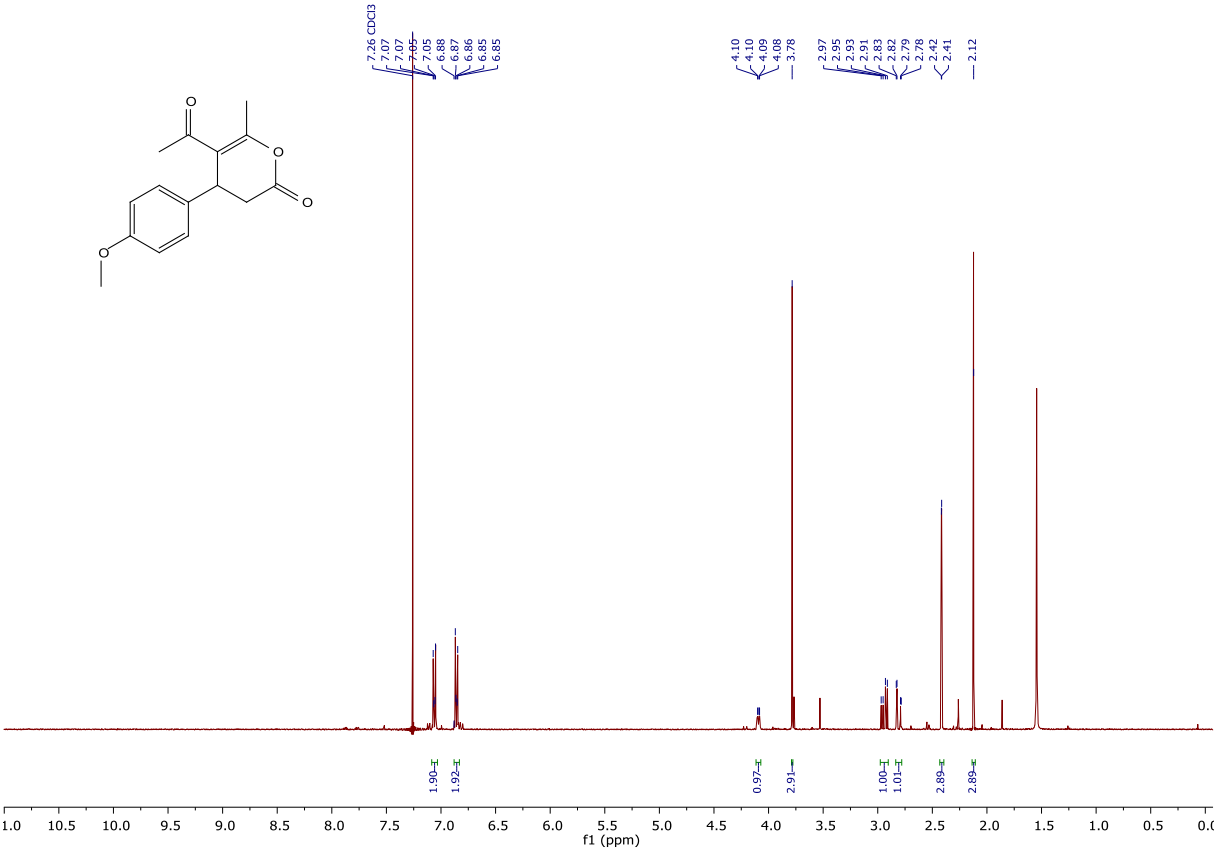
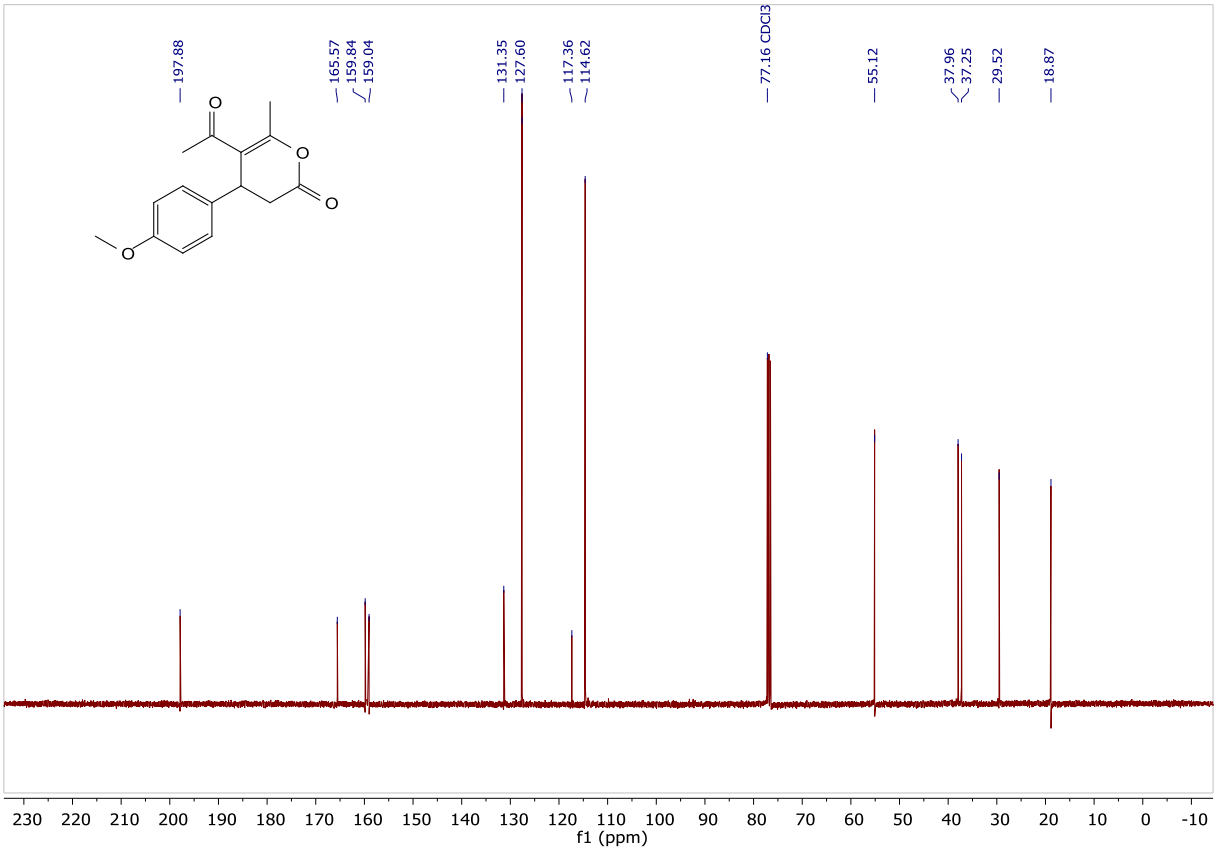
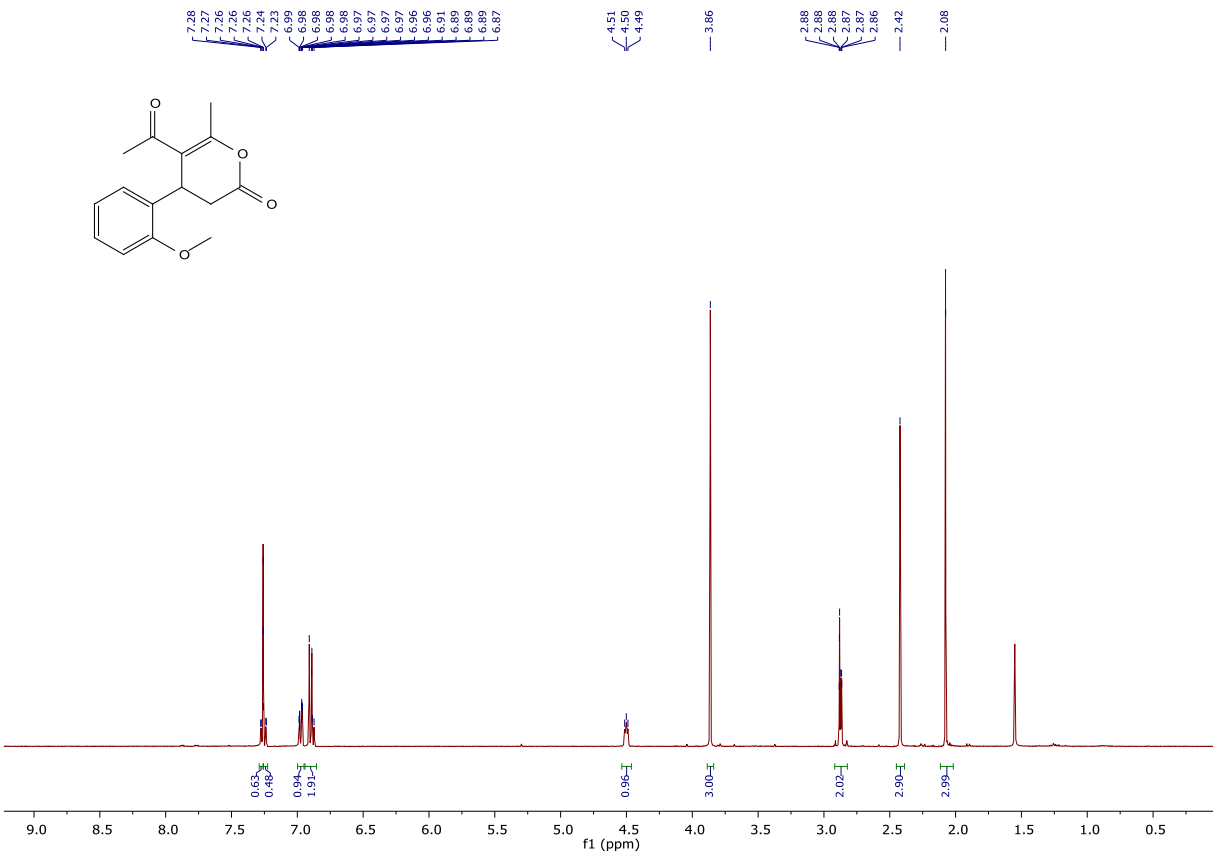


^{13}C NMR ($CDCl_3$)



^{1}H NMR ($CDCl_3$)



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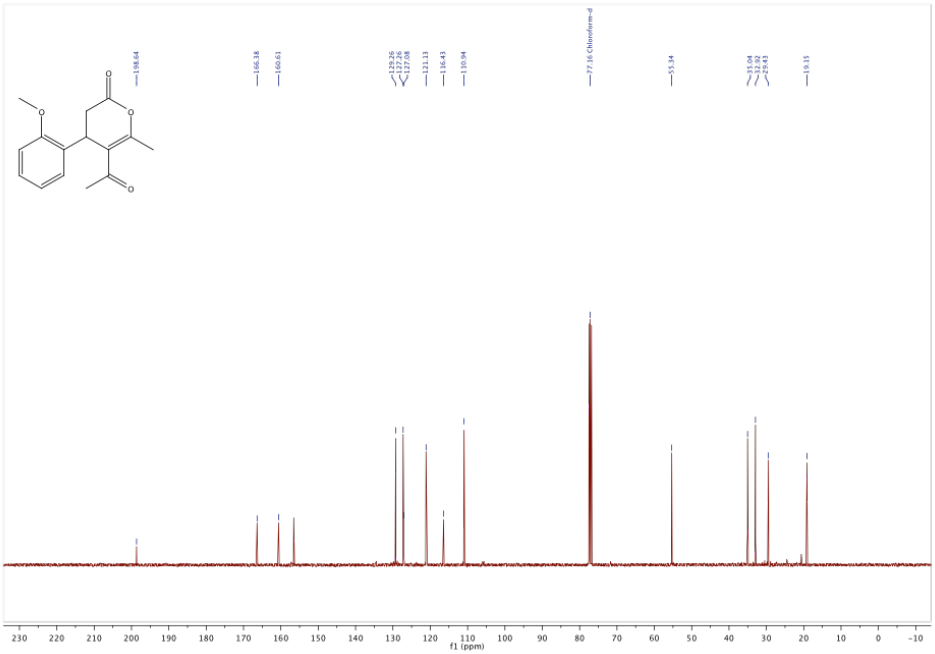

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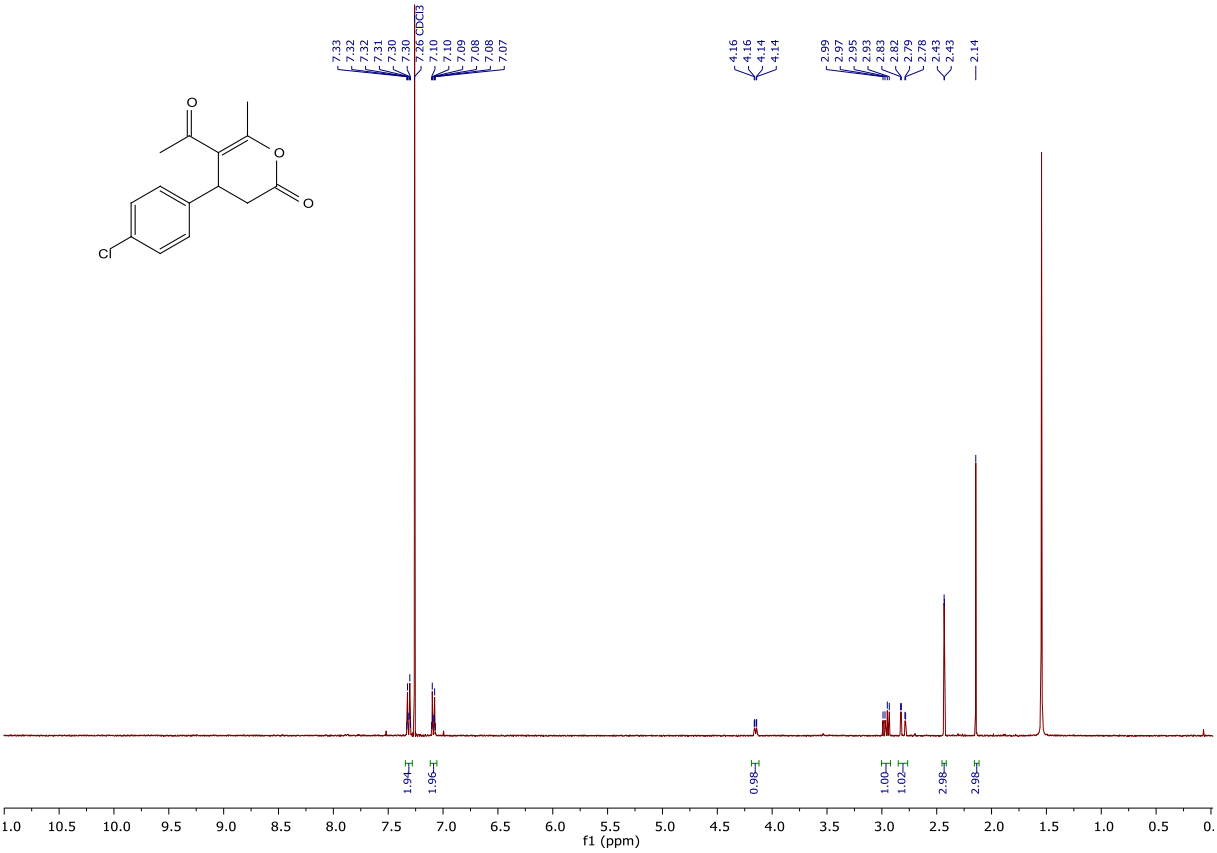

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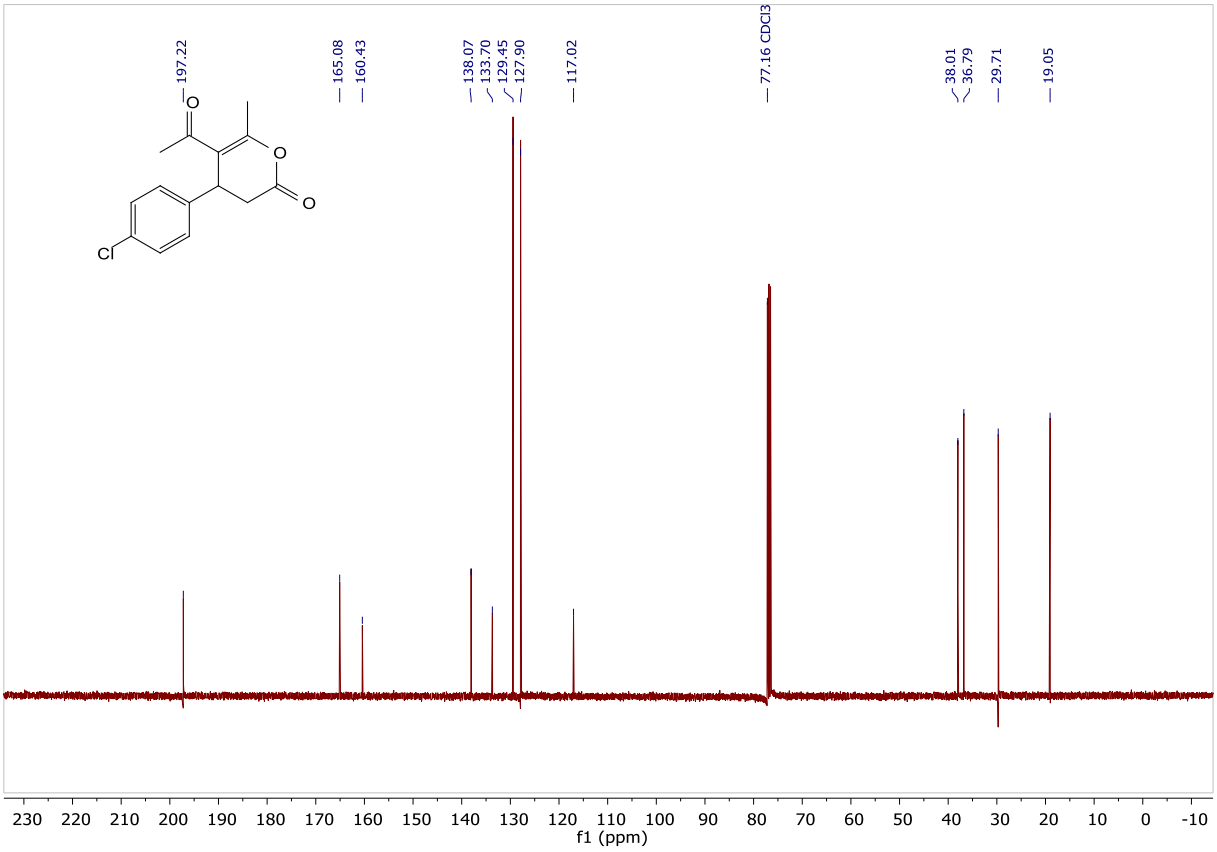

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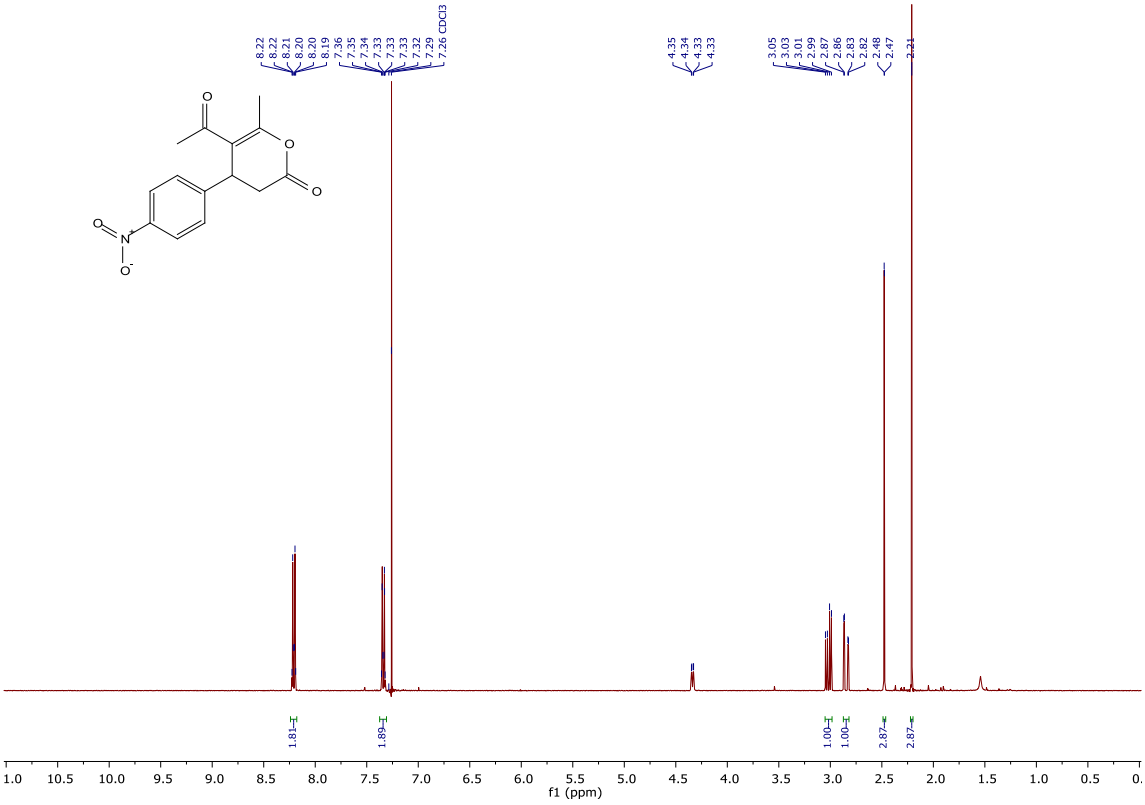

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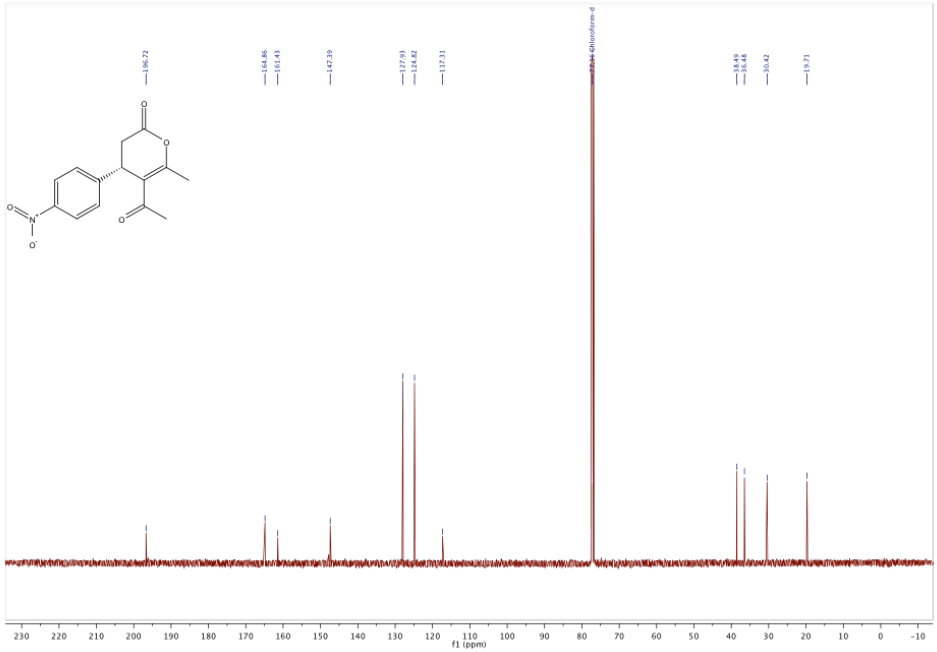

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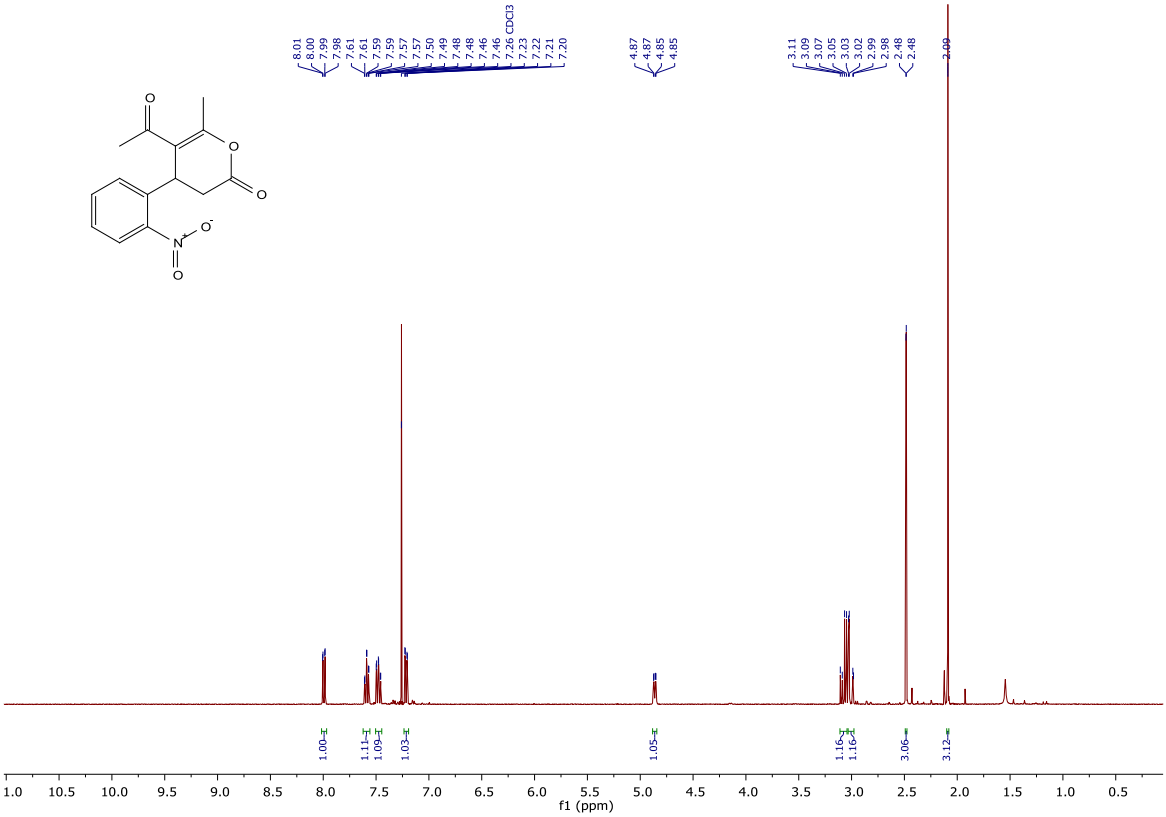
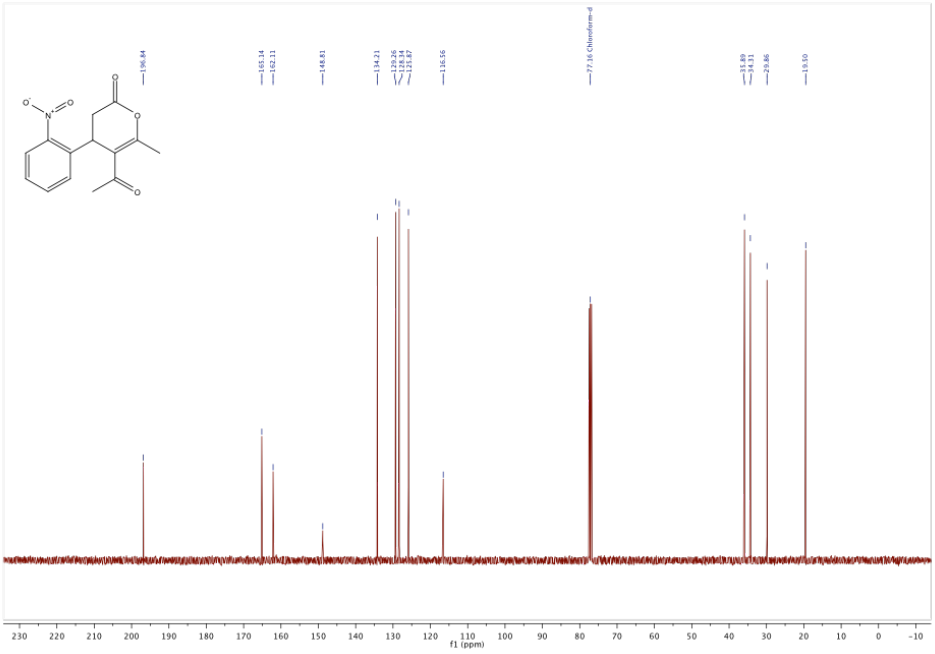


^{13}C NMR ($CDCl_3$)



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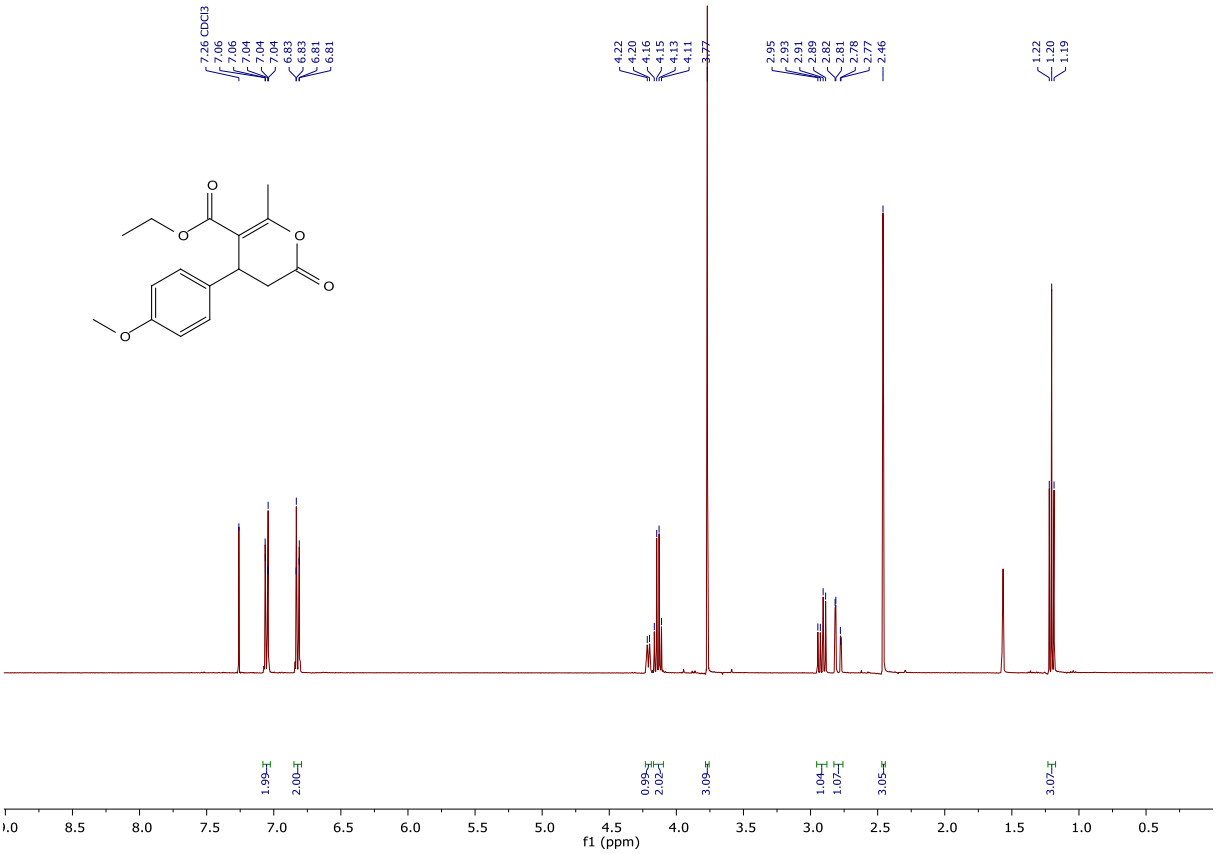

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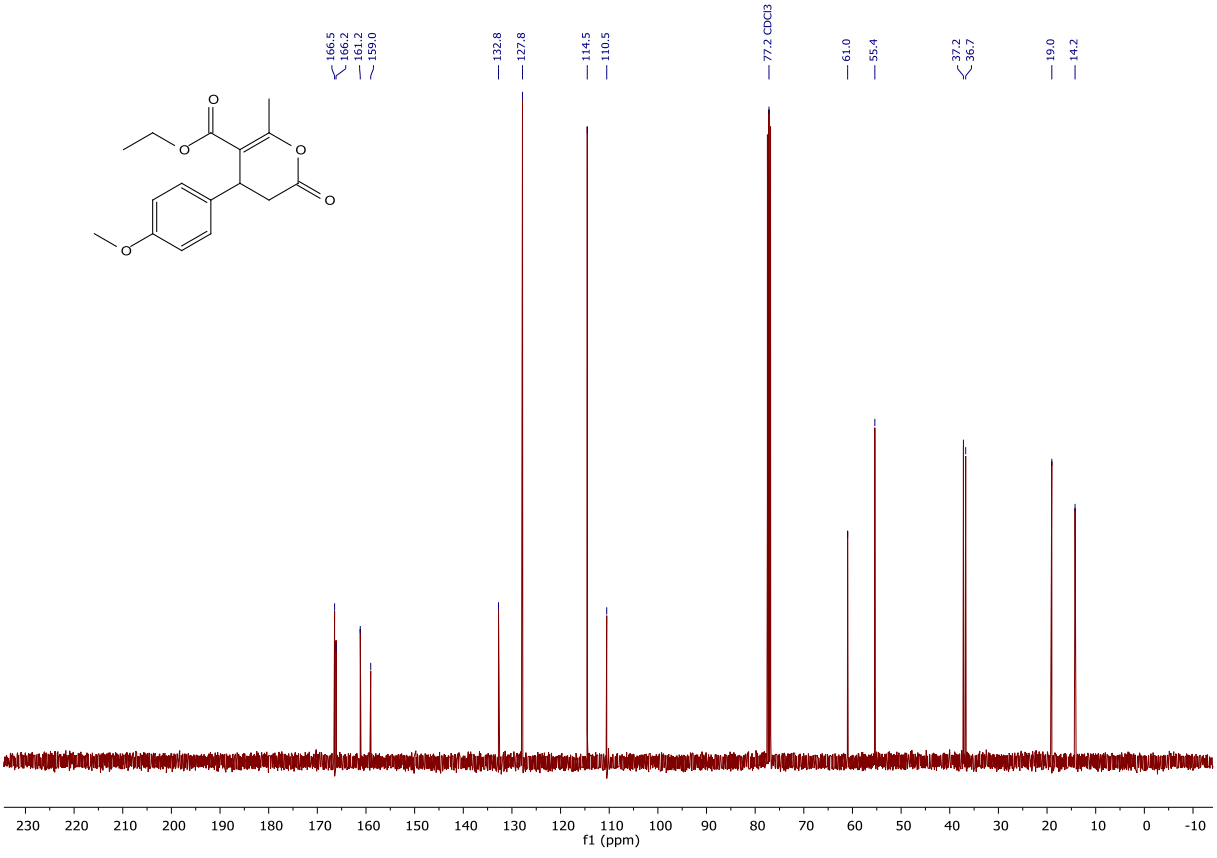

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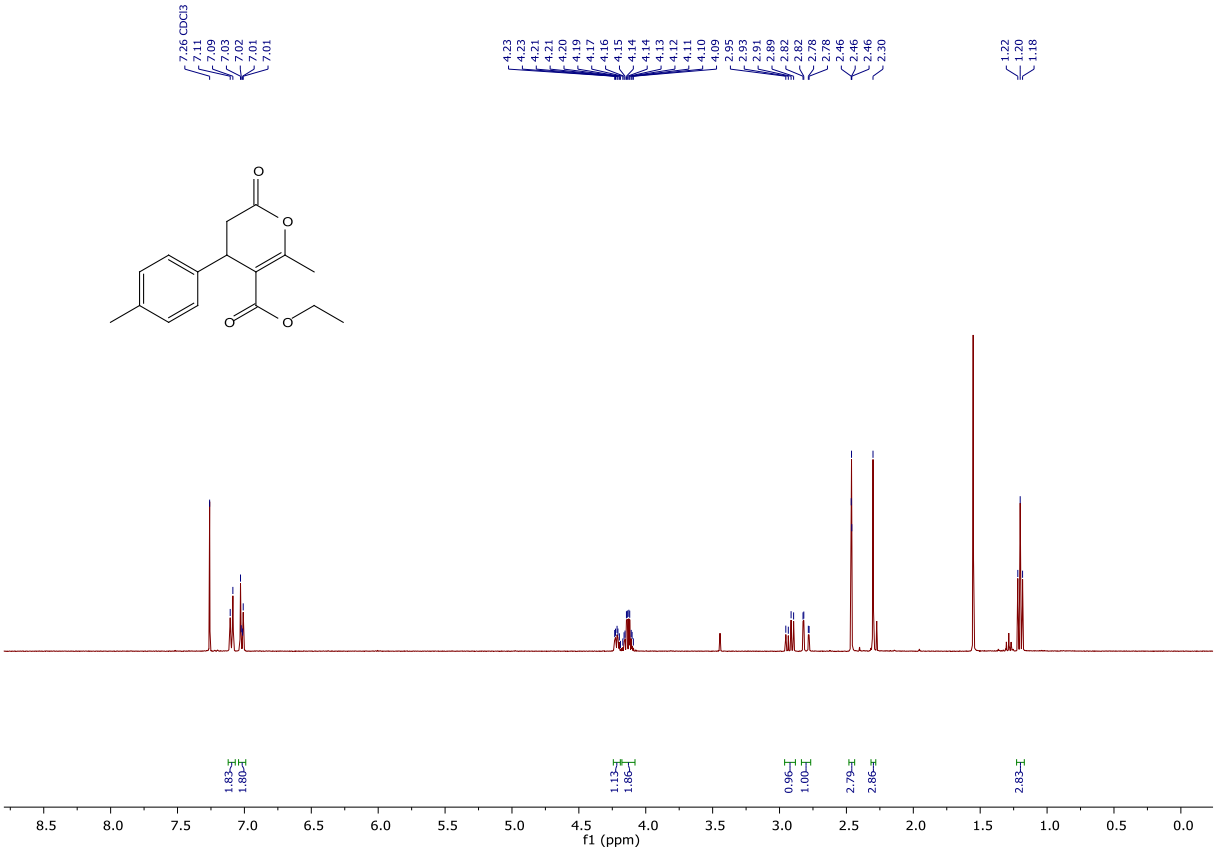

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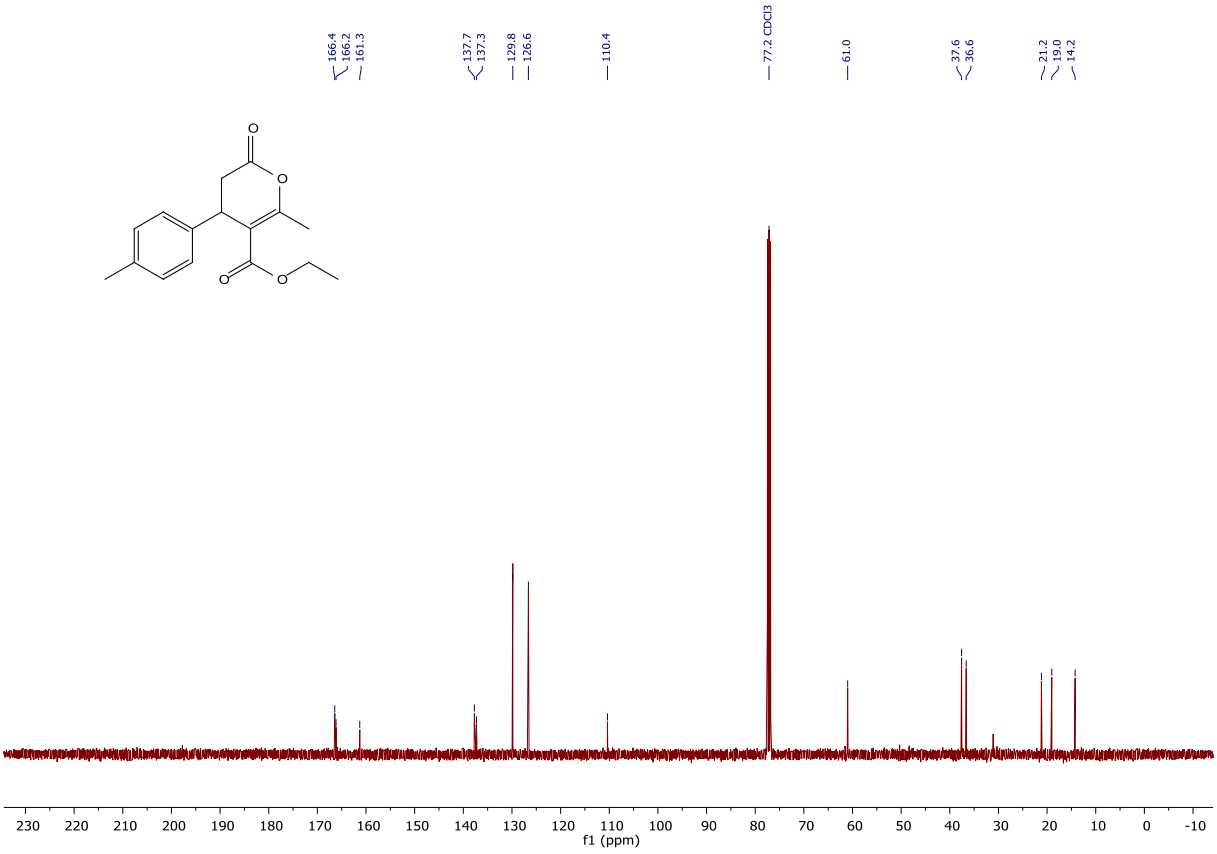

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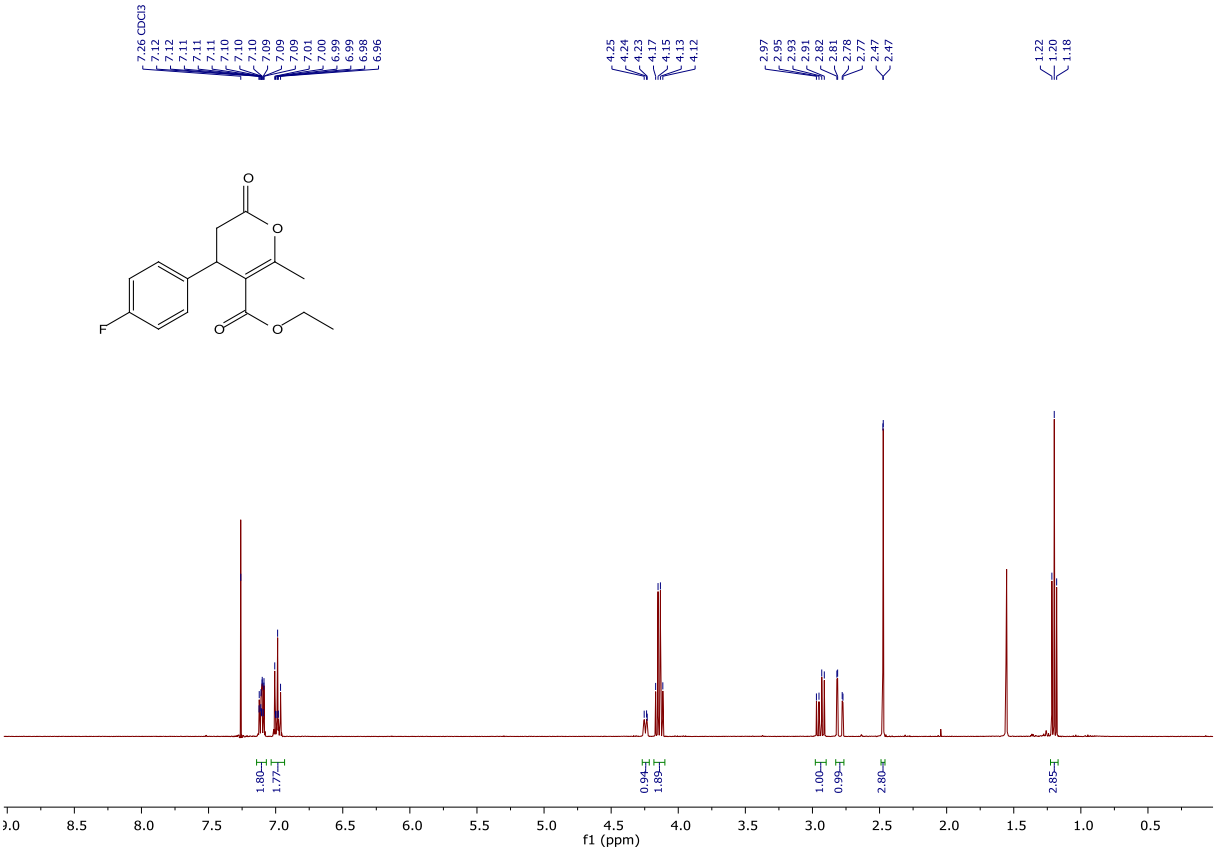

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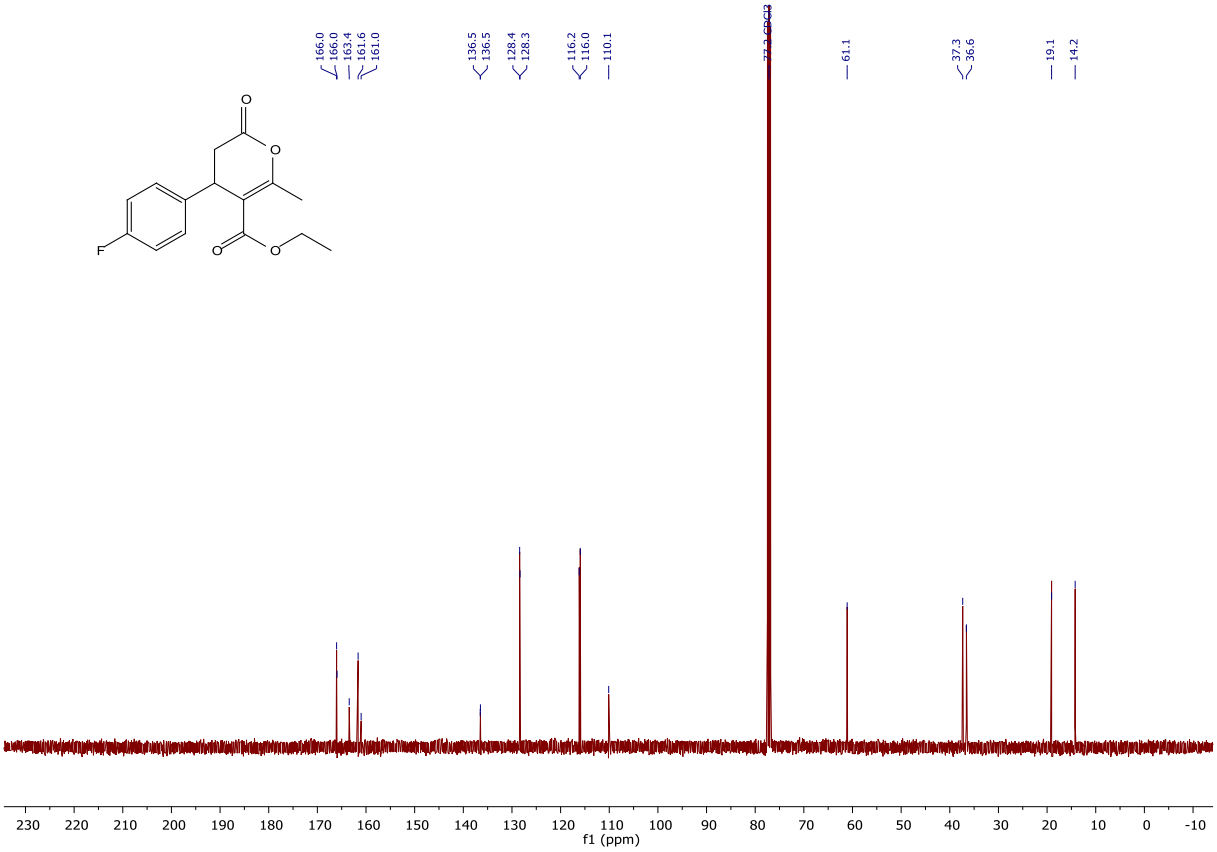
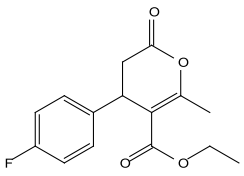
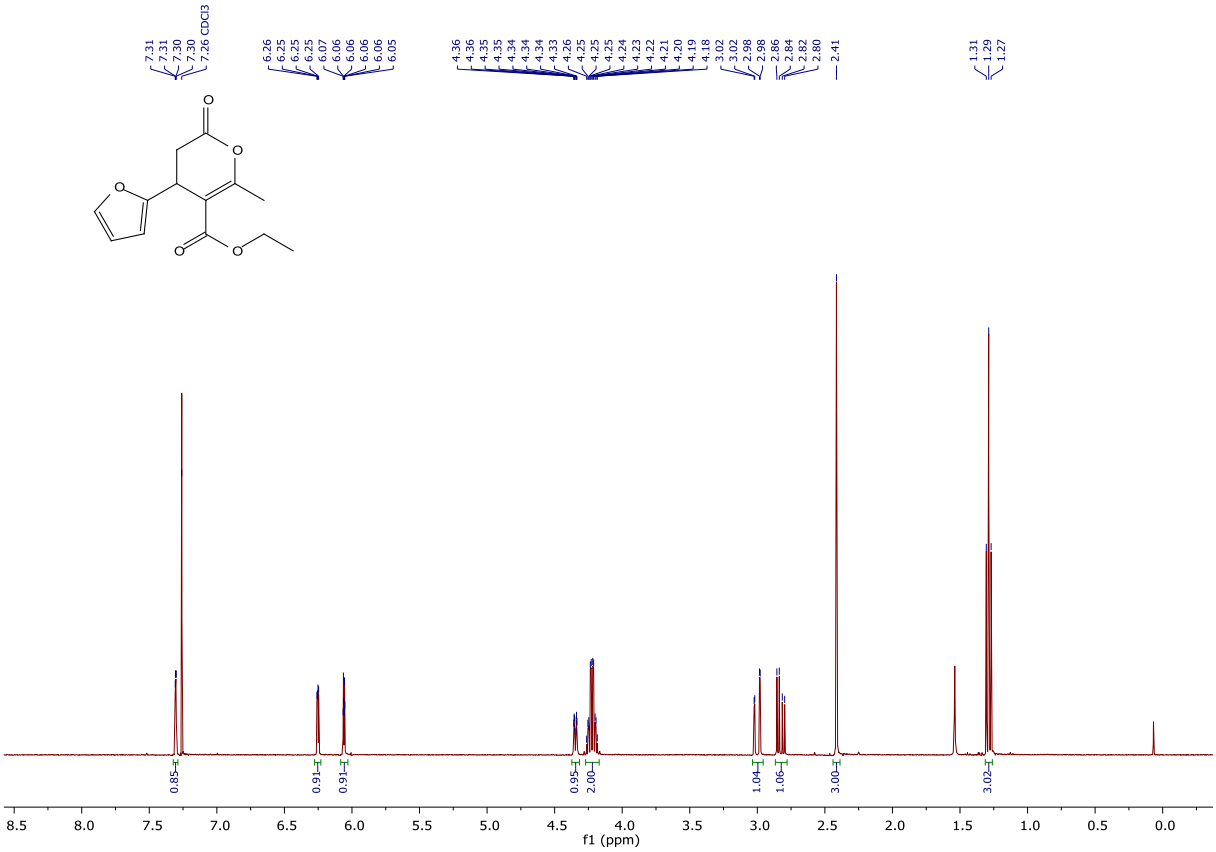


^{19}F NMR ($CDCl_3$)



^{1}H NMR ($CDCl_3$)



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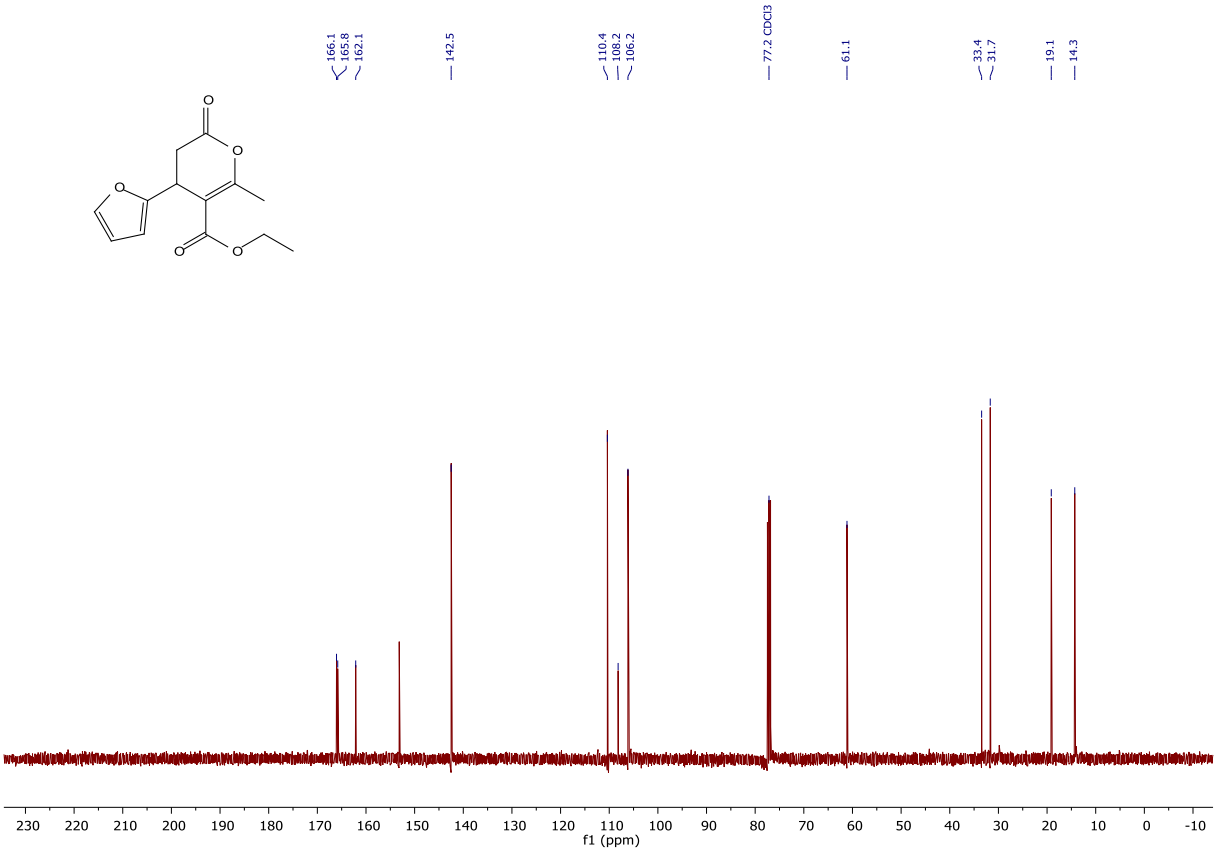
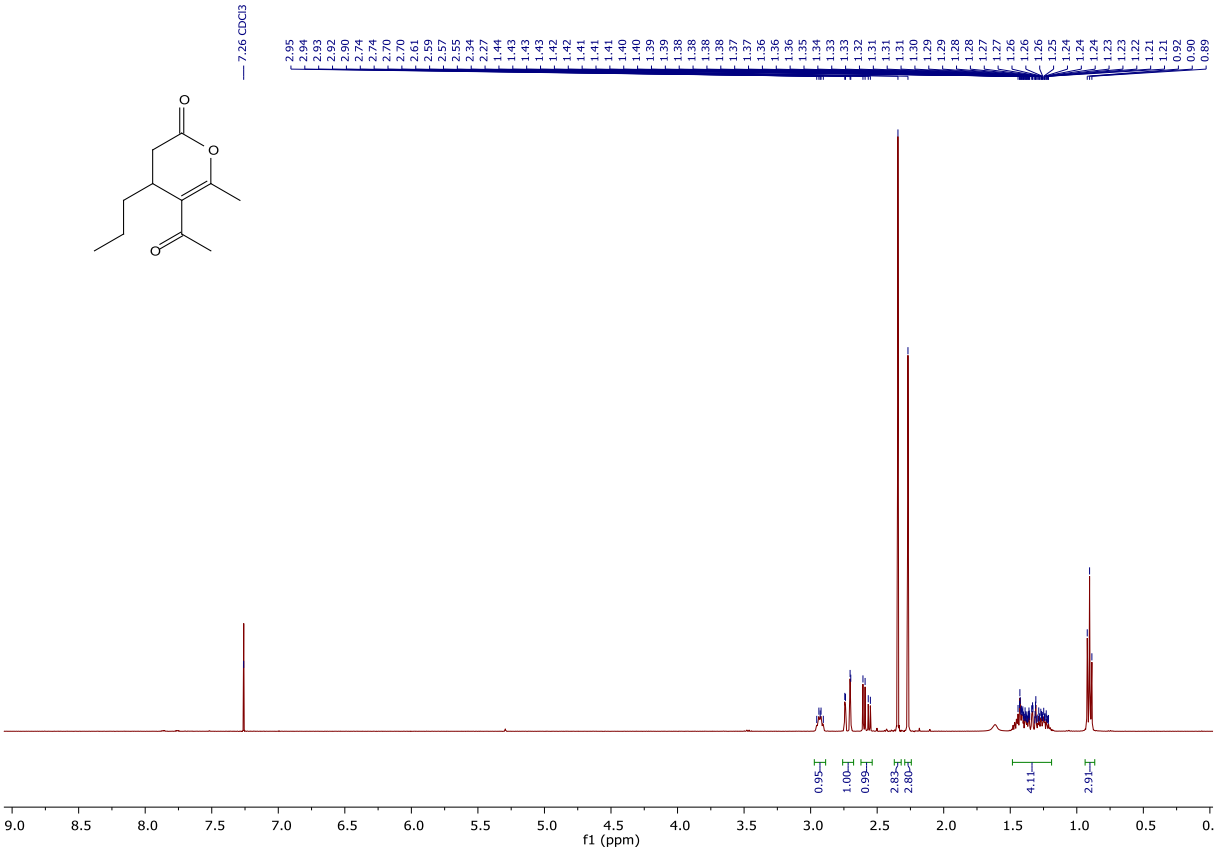


^{1}H NMR ($CDCl_3$)



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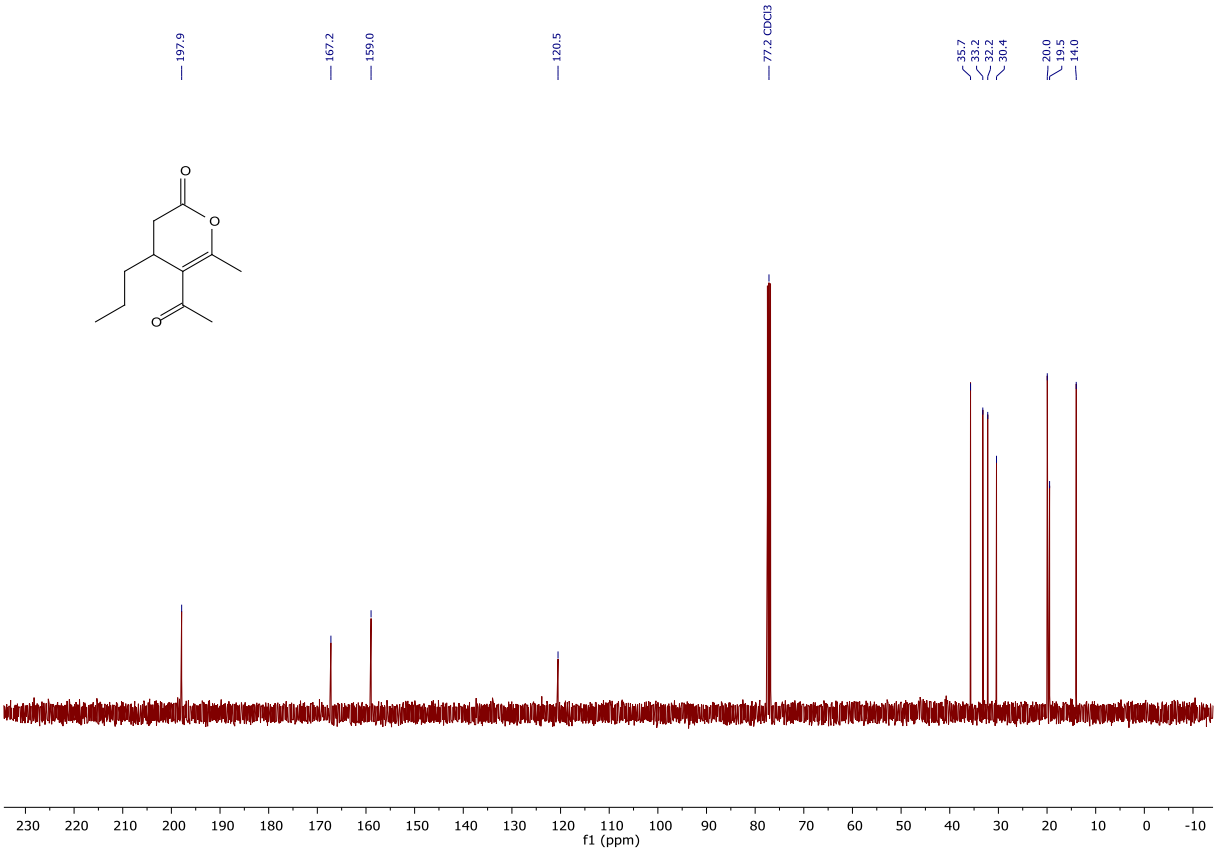

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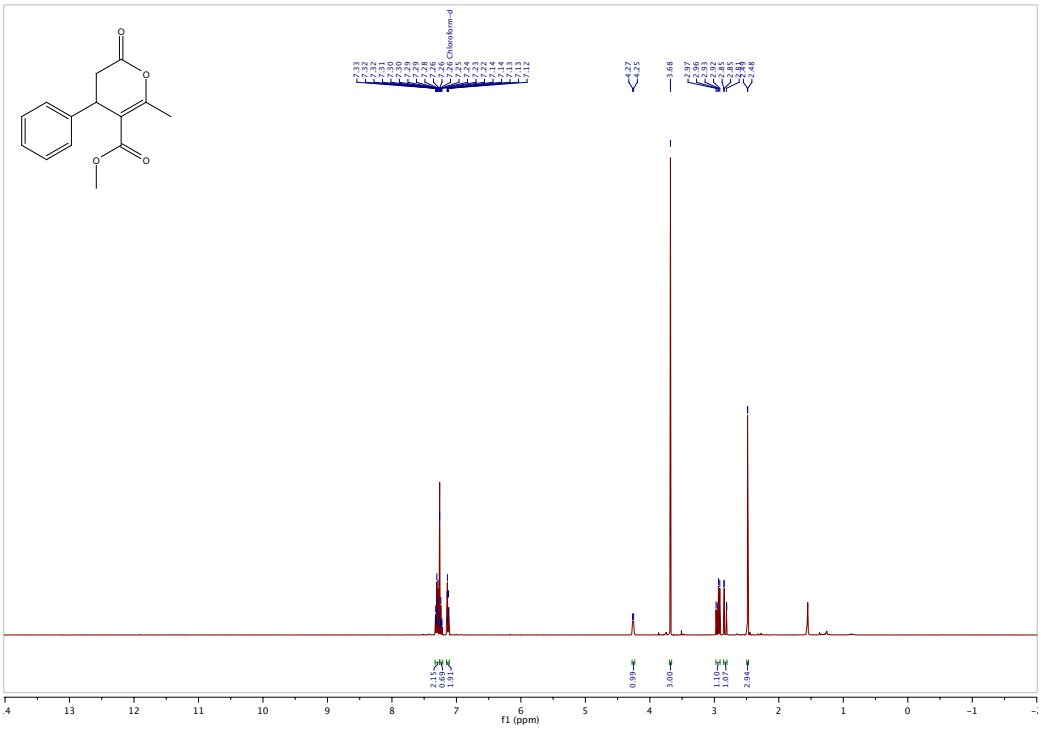

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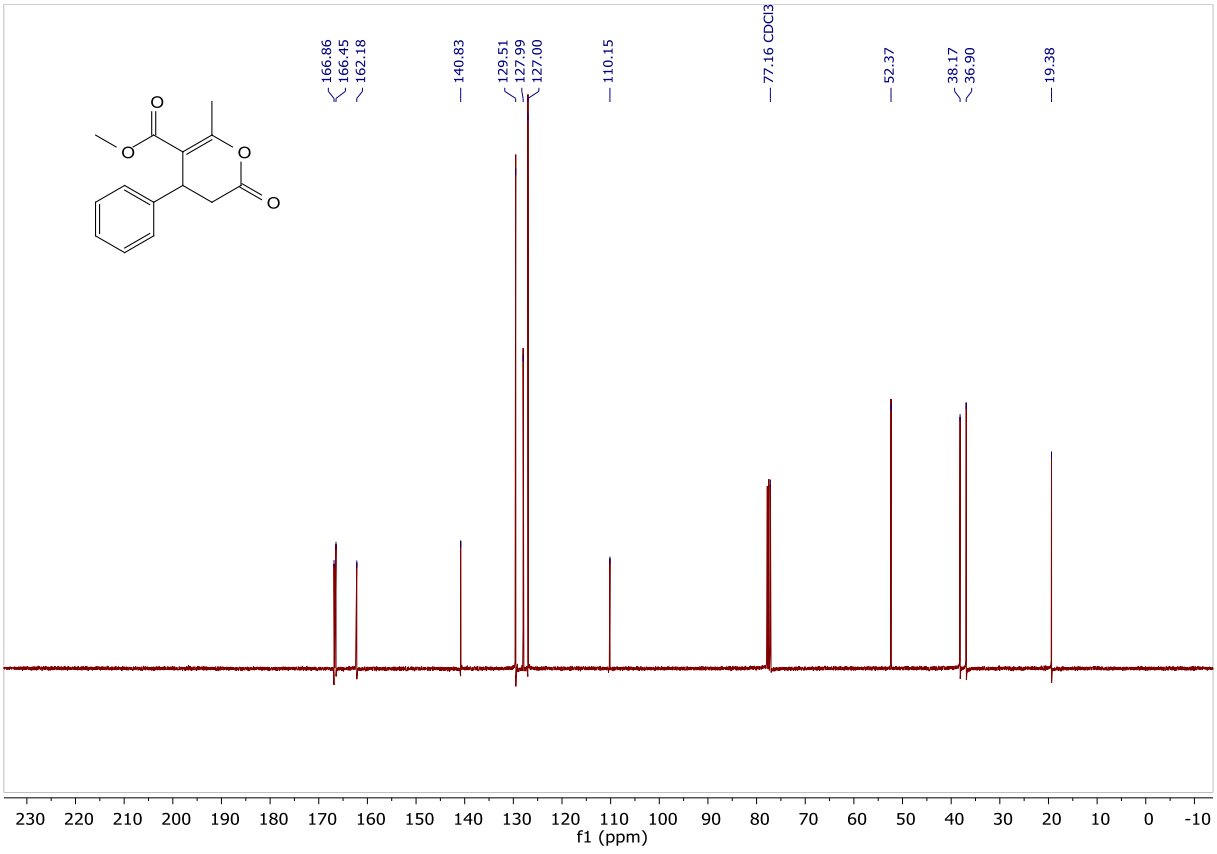
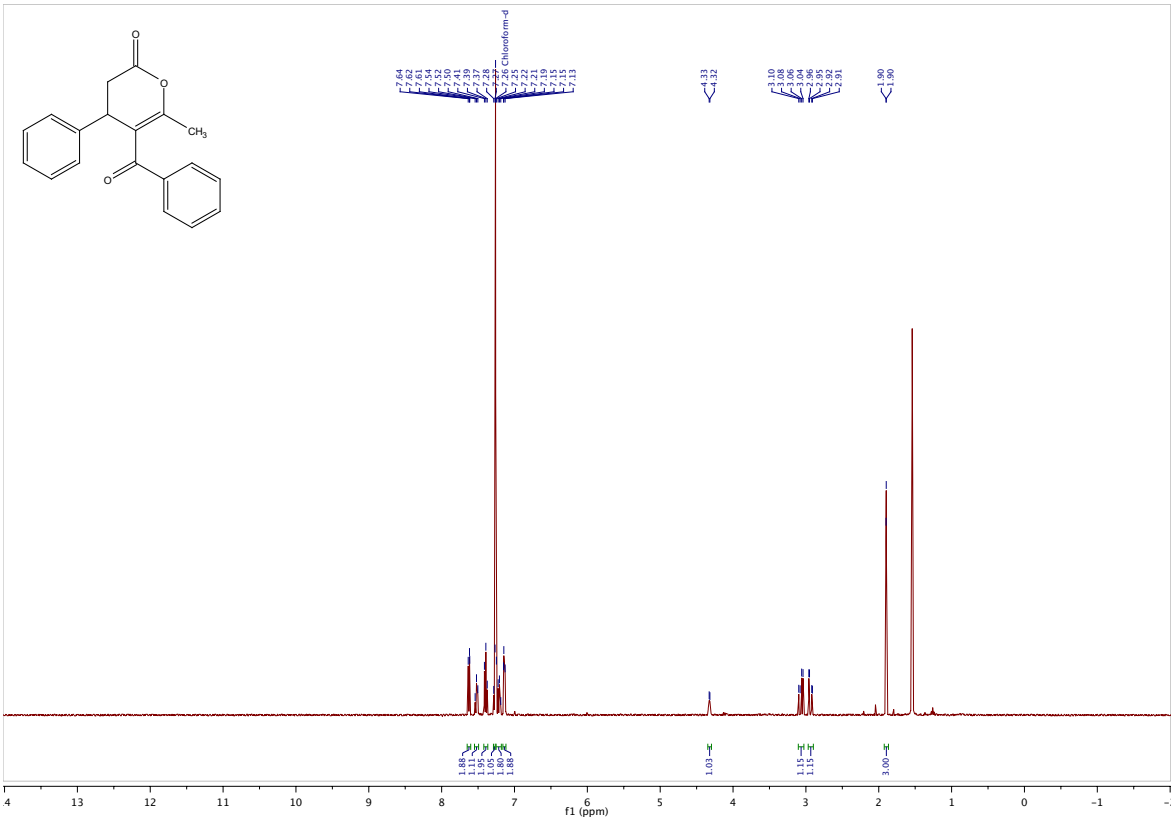
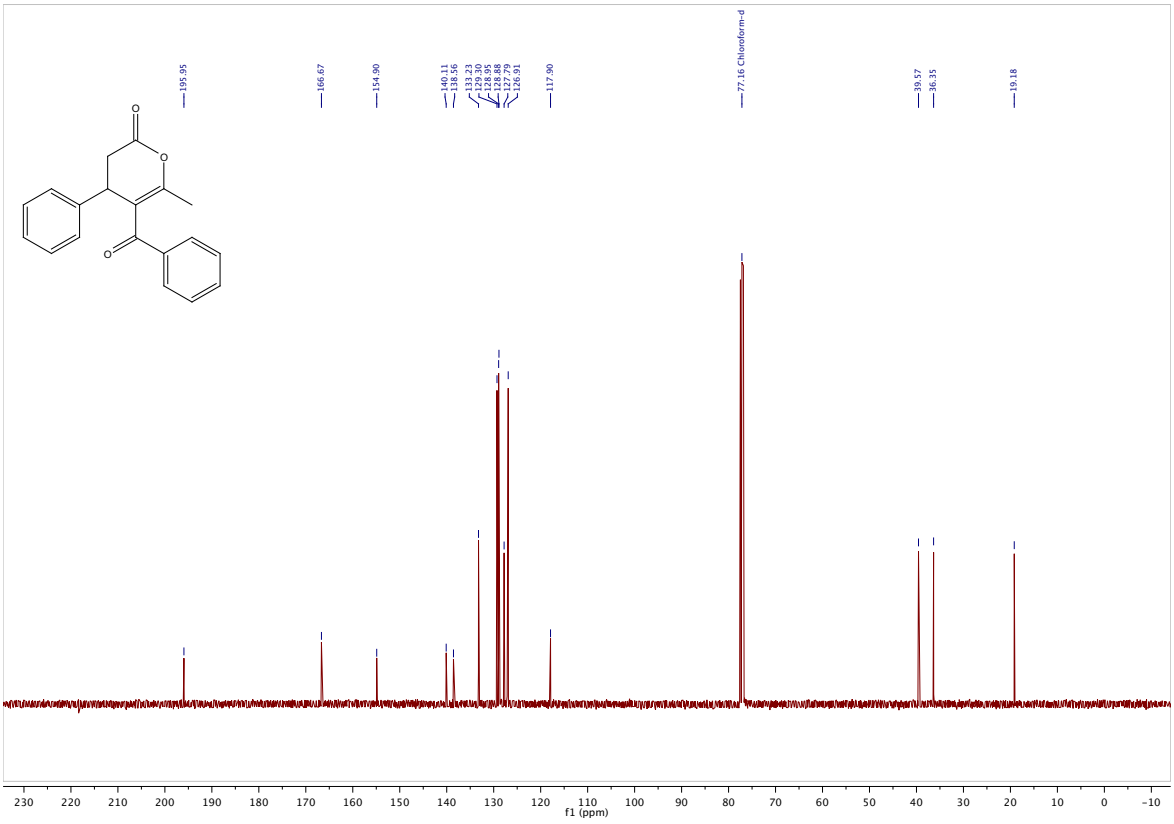


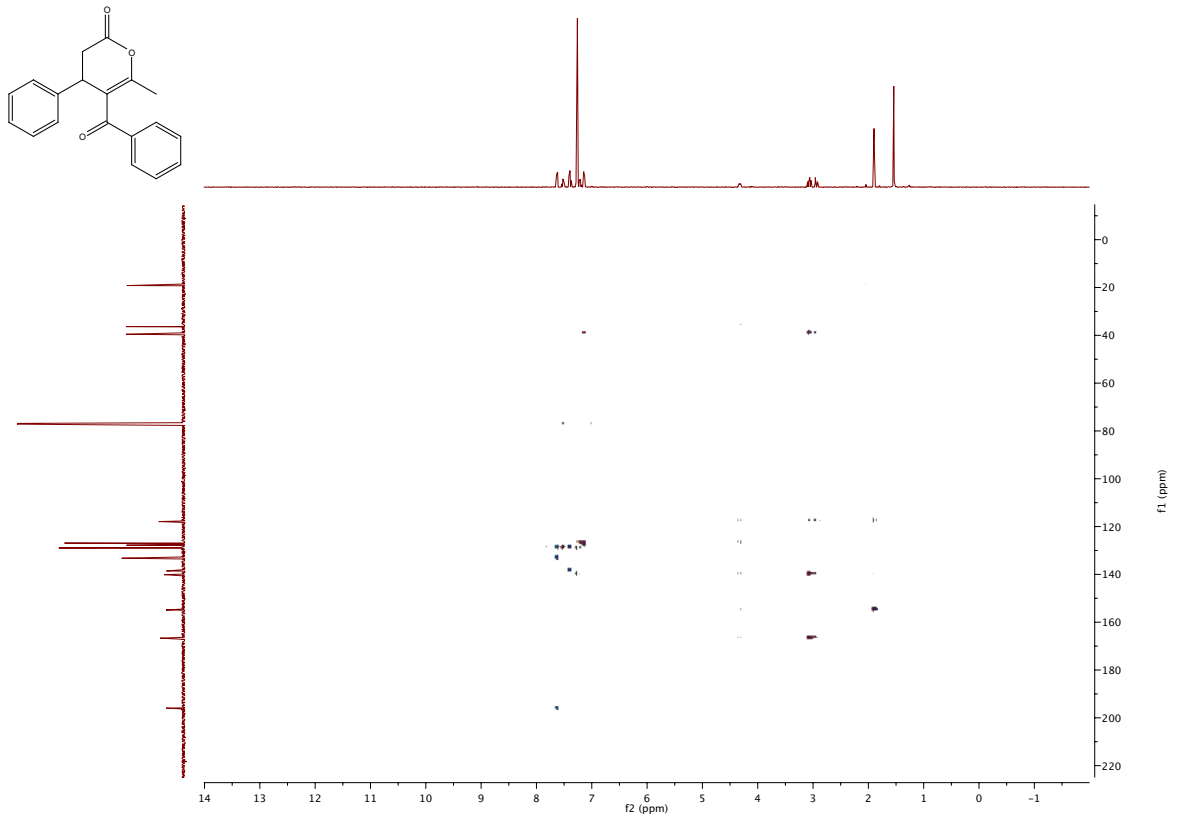
^{1}H NMR ($CDCl_3$)



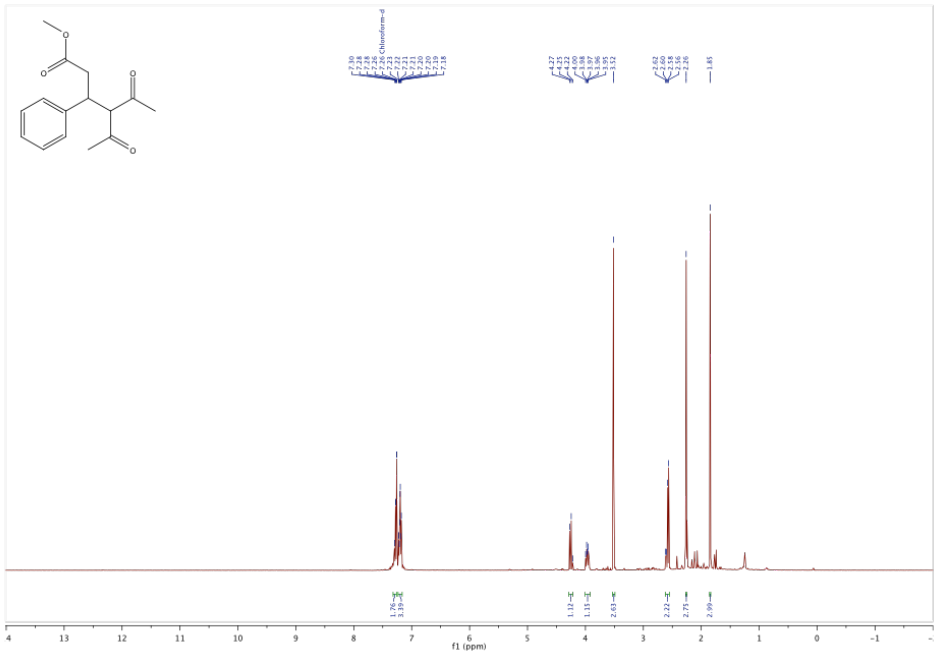
^{13}C NMR ($CDCl_3$)



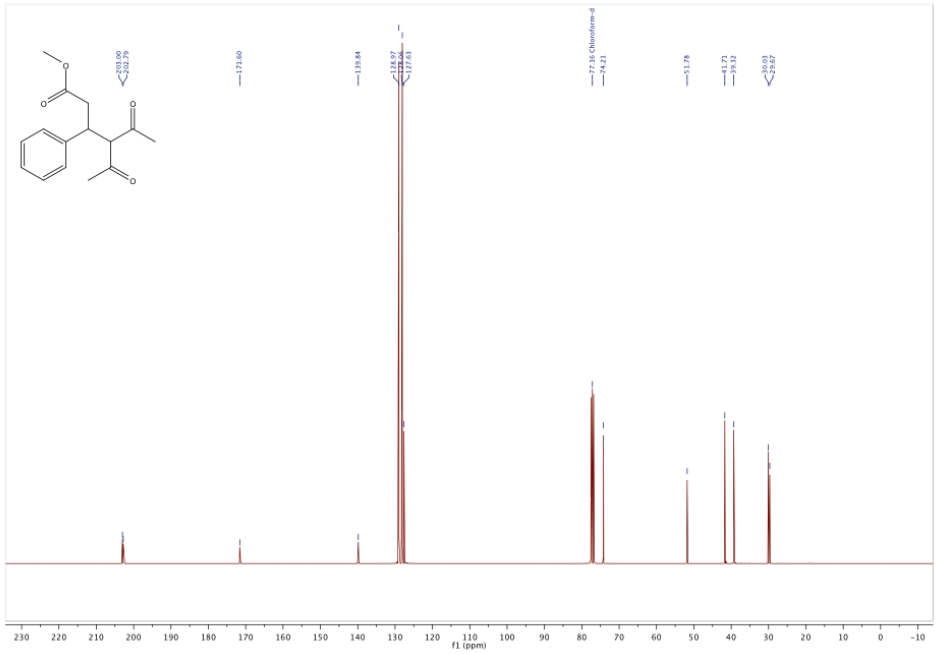
HMBC NMR ($CDCl_3$)



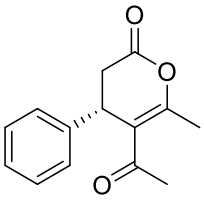
^{1}H NMR ($CDCl_3$)

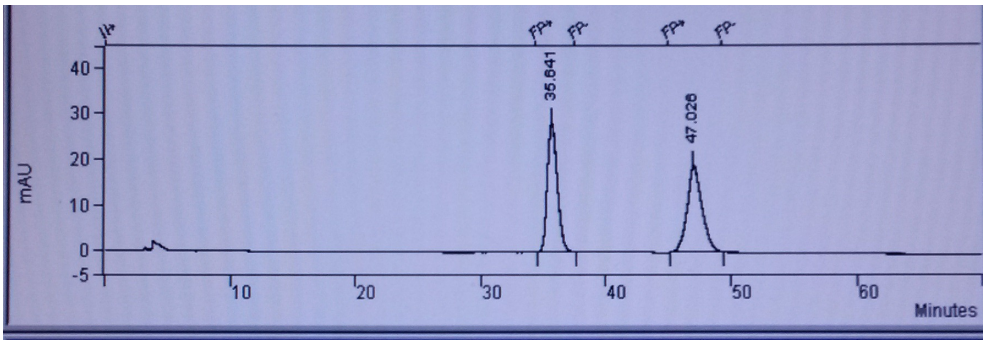


^{13}C NMR ($CDCl_3$)



HLPC data



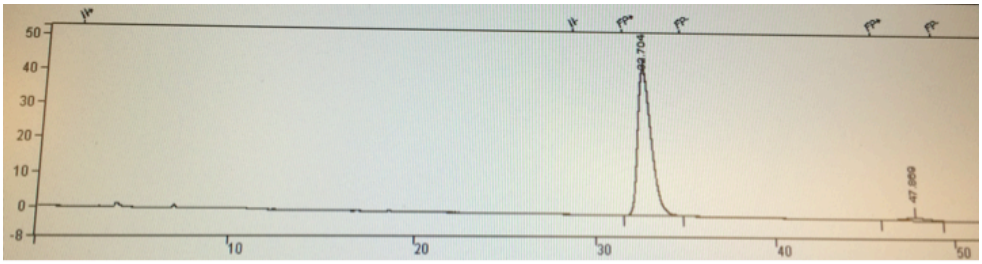


Results

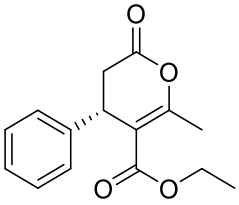
** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

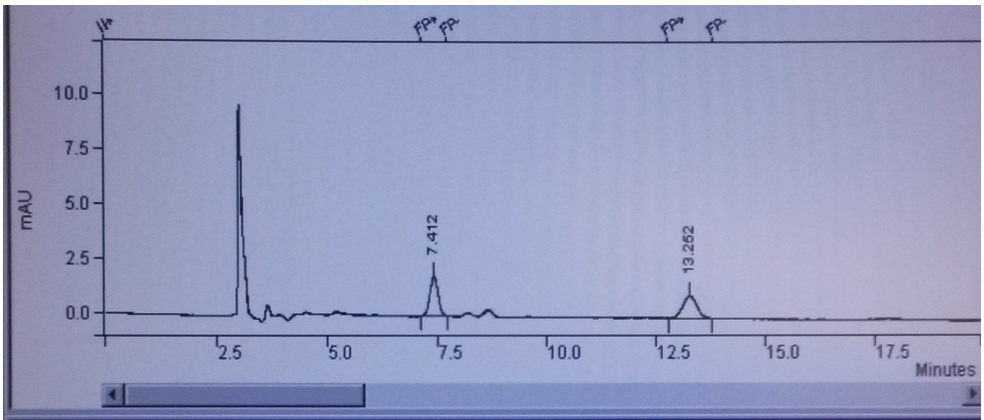
Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		50.2442	35.641	0.000	832998	BB	52.0	
2		49.7558	47.026	0.000	824899	BB	72.7	
	Totals:	100.0000		0.000	1657897			



Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		96.7539	32.704	0.000	1216452	BB	48.1	
2		3.2461	47.869	0.000	40812	BB	96.3	
	Totals:	100.0000		0.000	1257264			





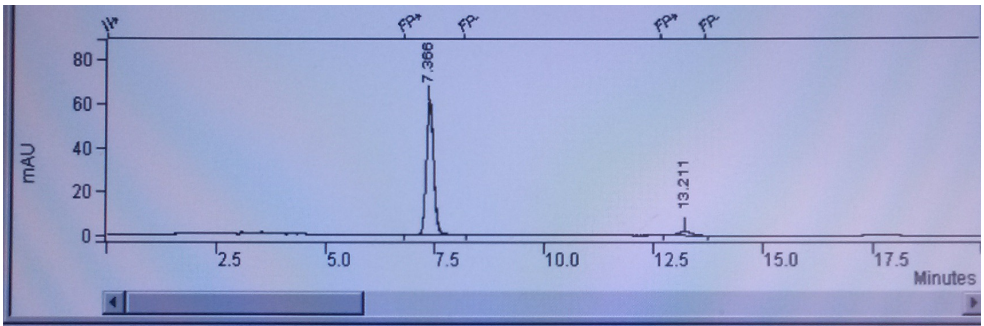
Results

Instrument : Varian Star #1 Sample Rate : 10.00 Hz
Channel : 1 = 254 nm Run Time : 69.982 min

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		50.4922	7.412	0.000	12165	BB	11.9	
2		49.5078	13.252	0.000	11928	BB	21.0	
	Totals:	100.0000		0.000	24093			

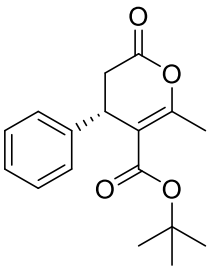


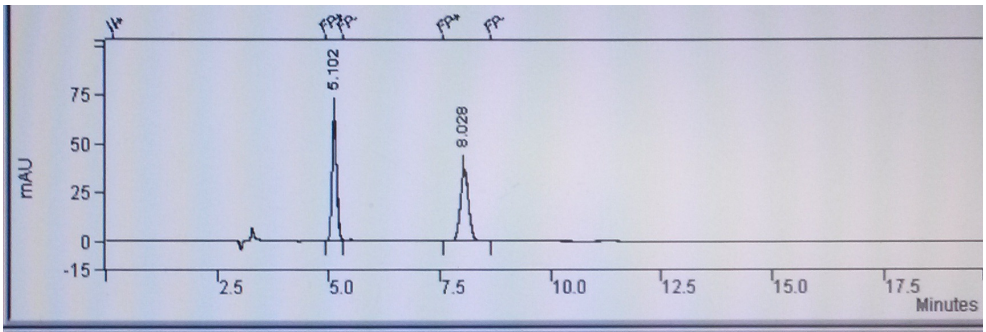
Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		94.8116	7.366	0.000	373571	BB	10.6	
2		5.1884	13.211	0.000	20443	BB	20.2	
	Totals:	100.0000		0.000	394014			



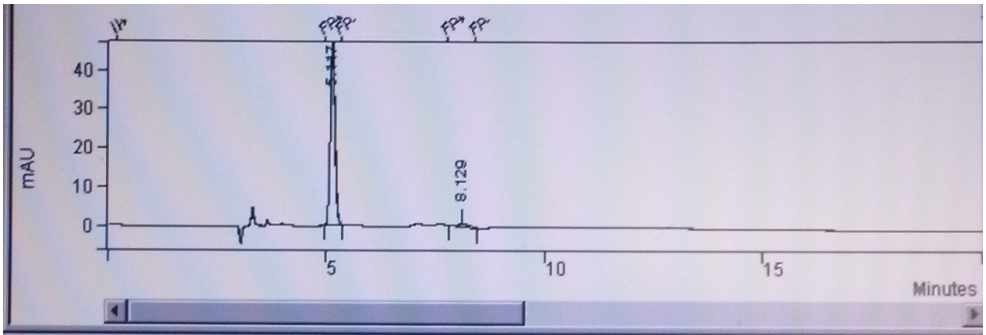


Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		49.7691	5.102	0.000	243691	BB	6.5	
2		50.2309	8.028	0.000	245952	BB	11.9	
	Totals:	100.0000		0.000	489643			

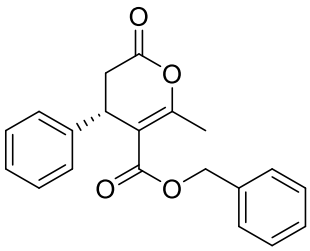


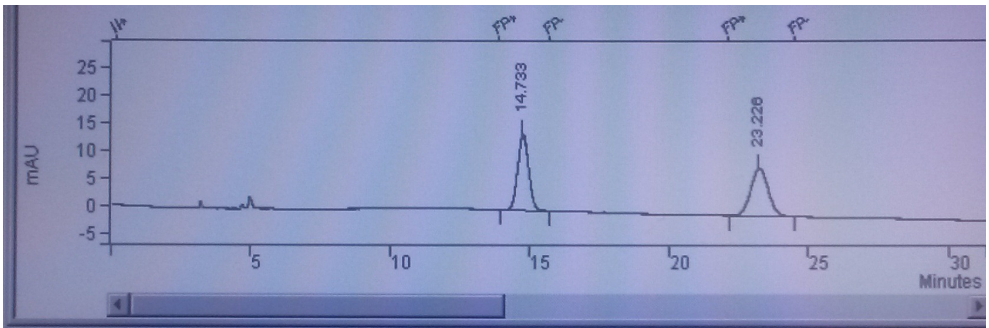
Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		96.0717	5.117	0.000	190300	BB	6.5	
2		3.9283	8.129	0.000	7781	BB	10.8	
	Totals:	100.0000		0.000	198081			



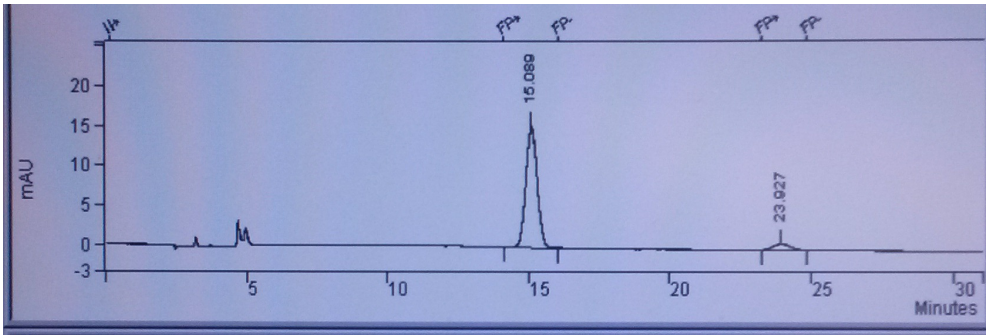


Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		49.7098	14.733	0.000	197466	BB	25.4	
2		50.2902	23.226	0.000	199771	BB	40.6	
	Totals:	100.0000		0.000	397237			

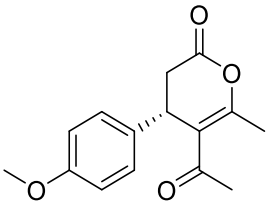


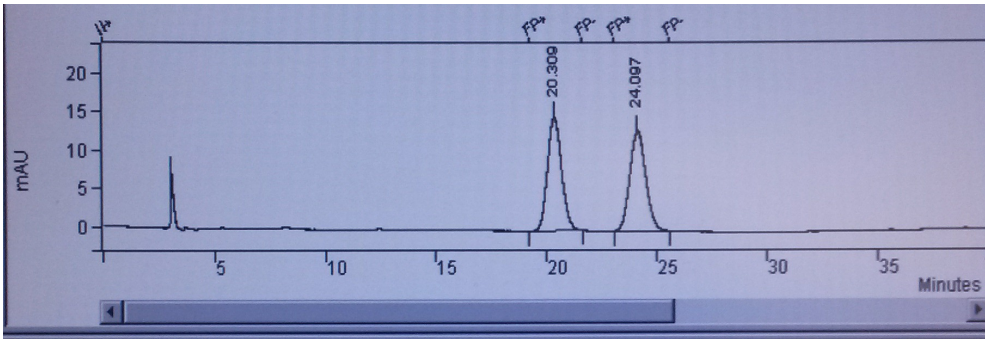
Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		93.0705	15.089	0.000	222957	BB	26.1	
2		6.9295	23.927	0.000	16600	BB	39.4	
	Totals:	100.0000		0.000	239557			



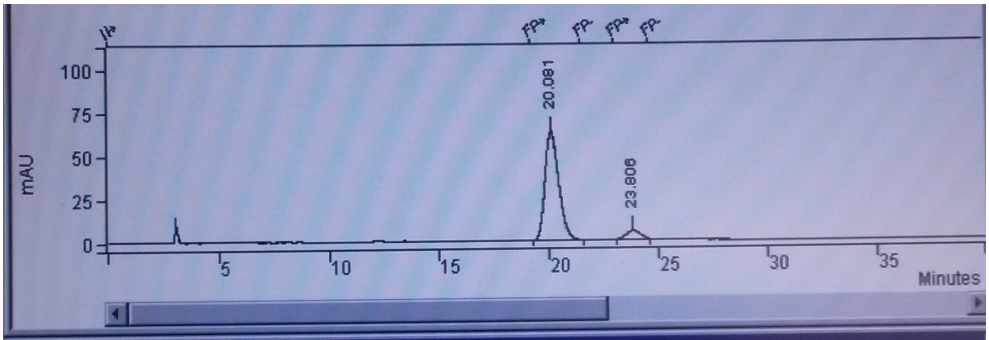


Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		50.0289	20.309	0.000	349581	BB	41.2	
2		49.9711	24.097	0.000	349178	BB	47.0	
	Totals:	100.0000		0.000	698759			

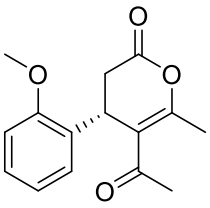


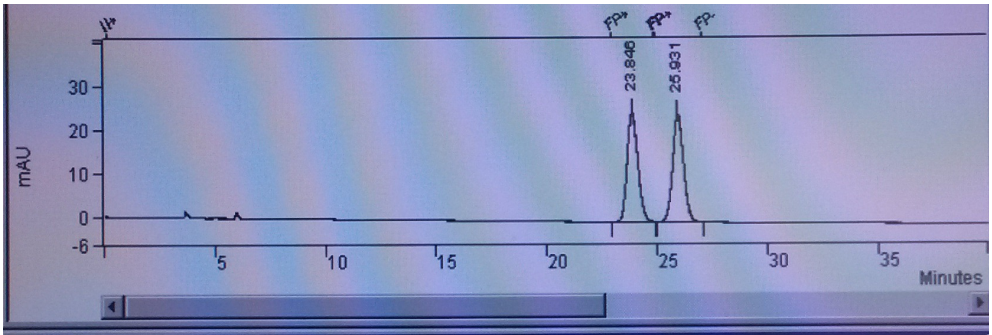
Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		92.2718	20.081	0.000	1452679	BB	40.3	
2		7.7282	23.806	0.000	121668	BB	43.5	
	Totals:	100.0000		0.000	1574347			



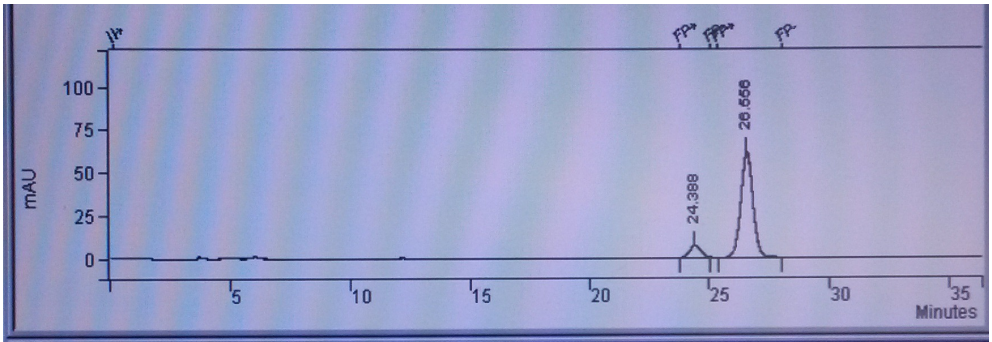


Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		50.0466	23.846	0.000	445850	BB	31.6	
2		49.9534	25.931	0.000	445020	BB	31.5	
	Totals:	100.0000		0.000	890870			

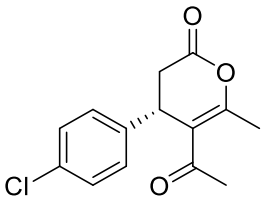


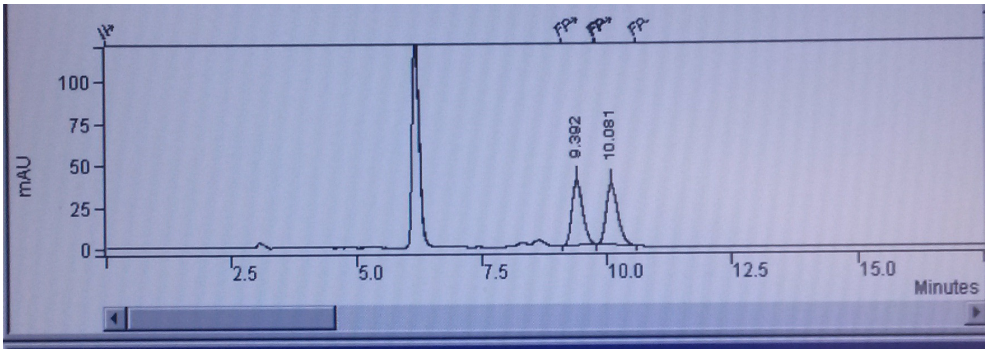
Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		9.4321	24.388	0.000	118630	BB	31.1	
2		90.5679	26.556	0.000	1139106	BB	32.0	
	Totals:	100.0000		0.000	1257736			



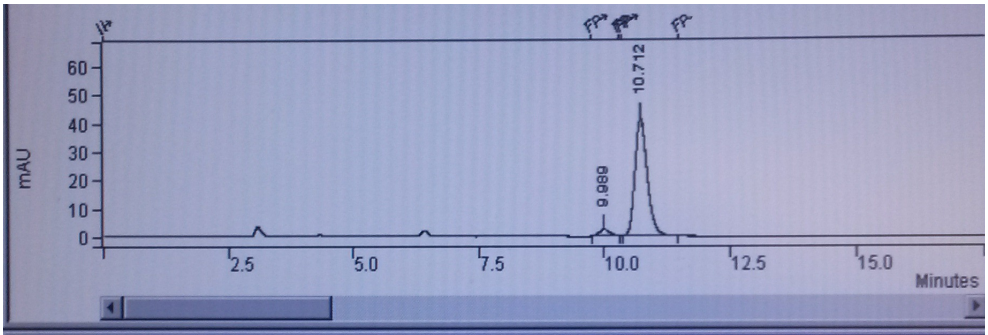


Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		49.4465	9.392	0.000	307377	BB	14.0	
2		50.5535	10.081	0.000	314259	BB	15.0	
	Totals:	100.0000		0.000	621636			

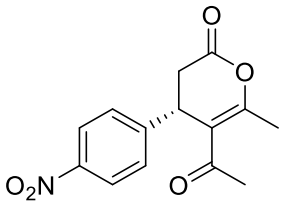


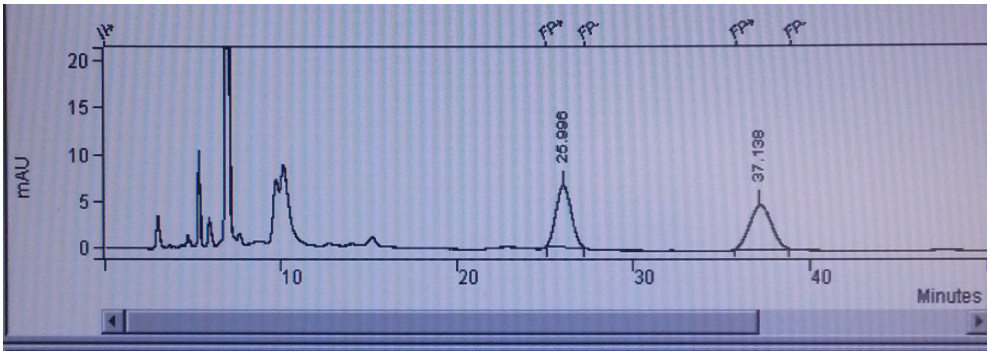
Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		4.3482	9.989	0.000	16830	BB	13.7	
2		95.6518	10.712	0.000	370222	BB	15.5	
	Totals:	100.0000		0.000	387052			



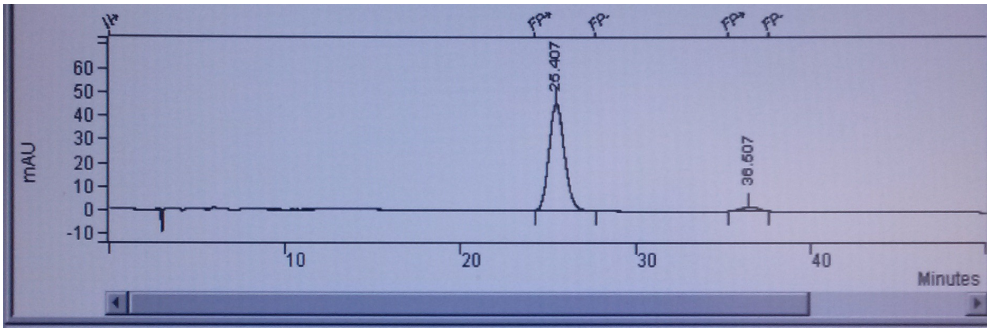


Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		49.5043	25.996	0.000	204801	BB	56.2	
2		50.4957	37.138	0.000	208902	BB	80.5	
	Totals:	100.0000		0.000	413703			

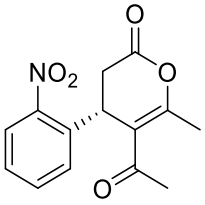


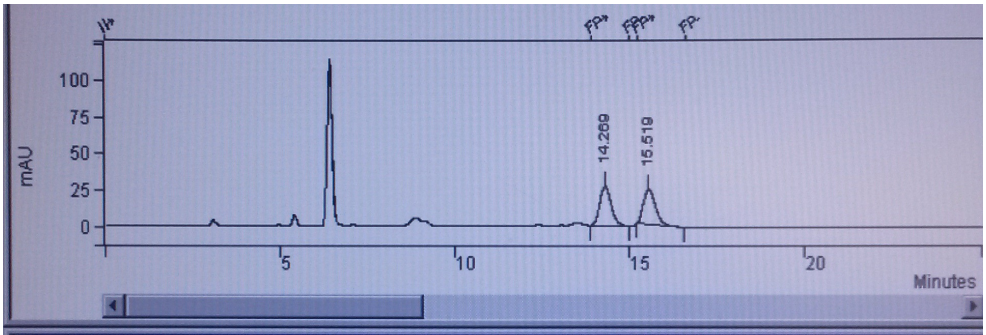
Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		95.7859	25.407	0.000	1469312	BB	56.6	
2		4.2141	36.507	0.000	64643	BB	71.8	
	Totals:	100.0000		0.000	1533955			



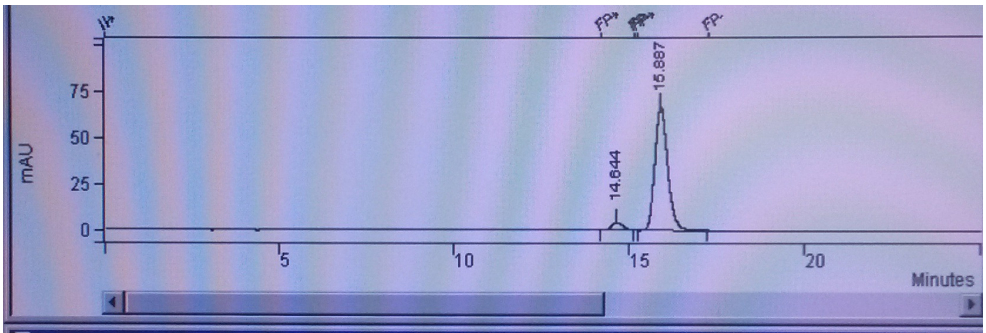


Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		52.8842	14.269	0.000	324486	BB	21.1	
2		47.1158	15.519	0.000	289093	BB	21.9	
Totals:		100.0000		0.000	613579			

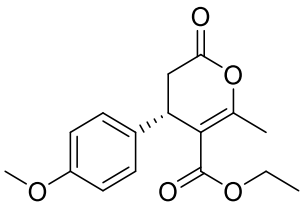


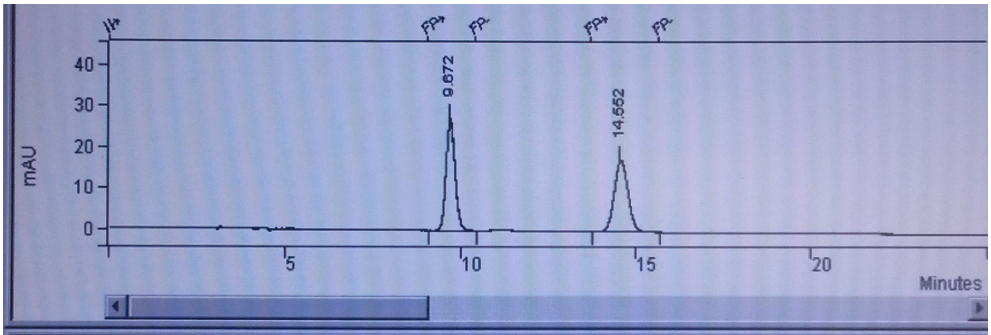
Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		4.8638	14.644	0.000	45827	BB	20.2	
2		95.1362	15.887	0.000	896365	BB	23.2	
Totals:		100.0000		0.000	942192			



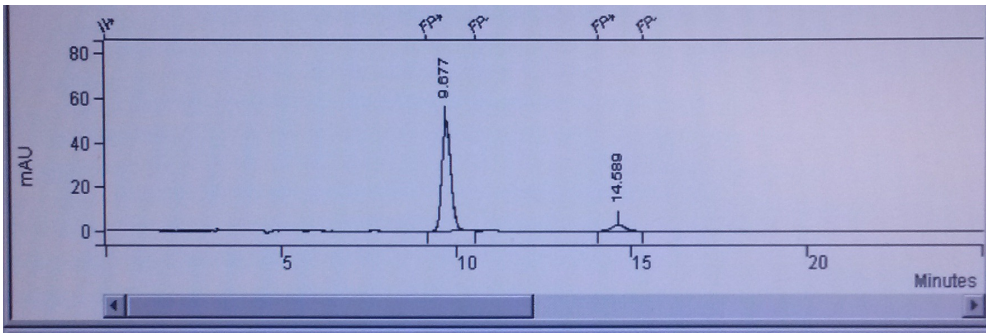


Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		49.9532	9.672	0.000	253909	BB	16.3	
2		50.0468	14.552	0.000	254384	BB	25.4	
Totals:		100.0000		0.000	508293			

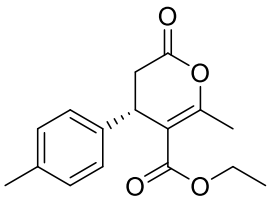


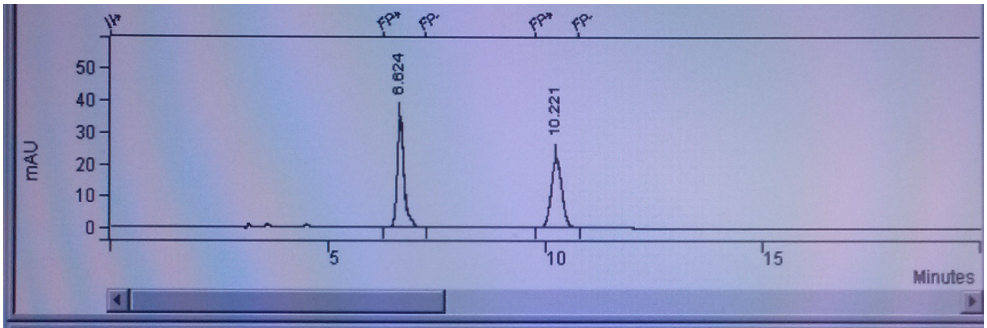
Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		92.5552	9.677	0.000	469359	BB	16.4	
2		7.4448	14.589	0.000	37753	BB	25.5	
Totals:		100.0000		0.000	507112			



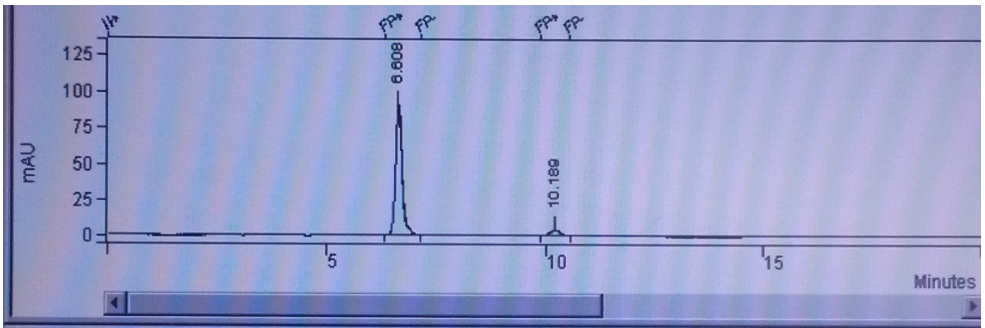


Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		50.4048	6.624	0.000	199168	BB	9.4	
2		49.5952	10.221	0.000	195969	BB	16.0	
Totals:		100.0000		0.000	395137			

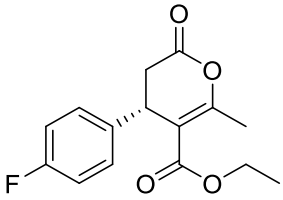


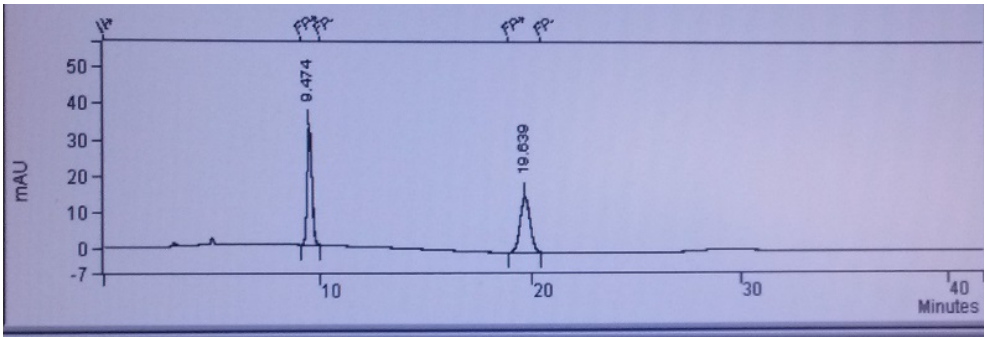
Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		93.3774	6.608	0.000	500875	BB	9.3	
2		6.6226	10.189	0.000	35523	BB	15.9	
Totals:		100.0000		0.000	536398			



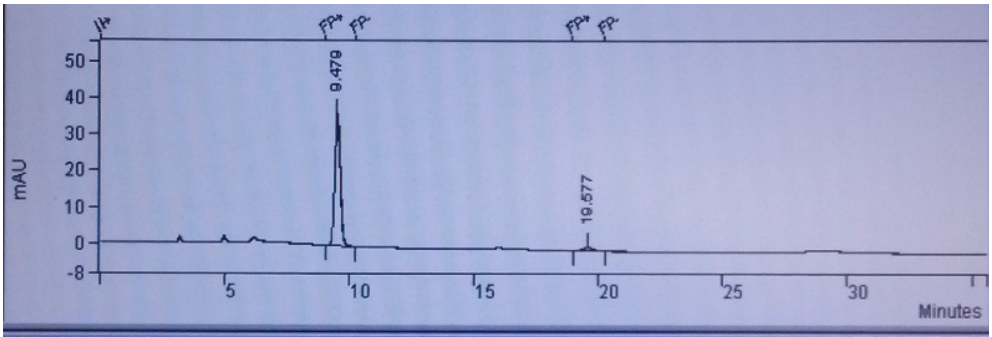


Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		50.3423	9.474	0.000	268123	BB	14.0	
2		49.6577	19.639	0.000	264477	BB	30.7	
	Totals:	100.0000		0.000	532600			

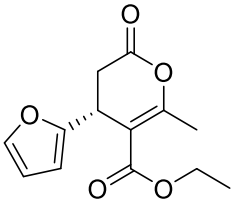


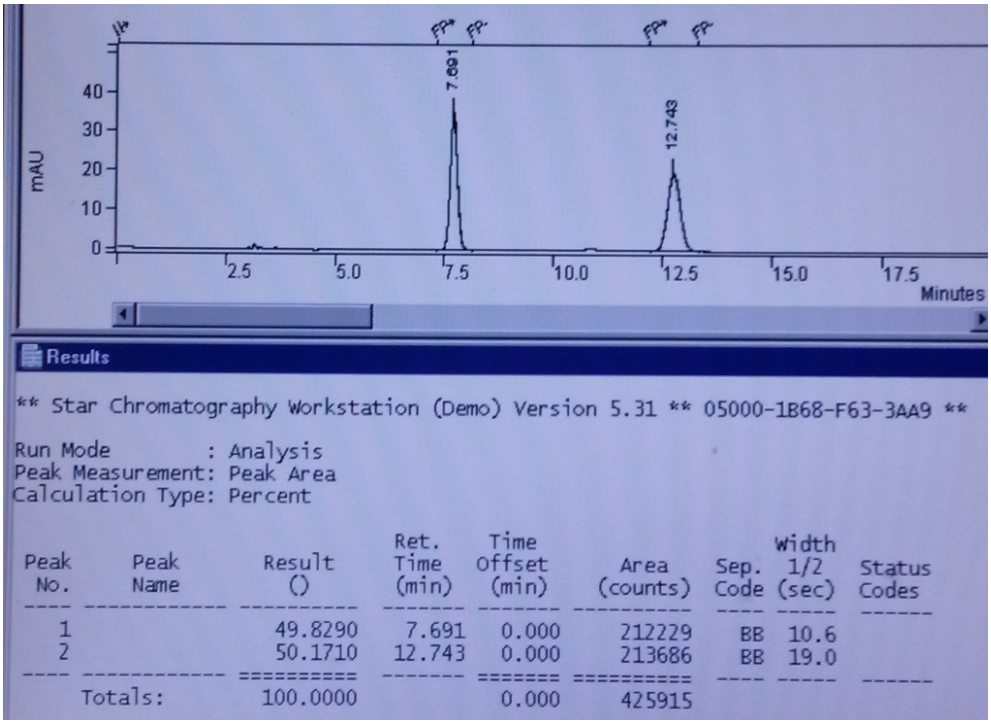
Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		94.9257	9.479	0.000	290289	BB	14.0	
2		5.0743	19.577	0.000	15518	BB	29.5	
	Totals:	100.0000		0.000	305807			



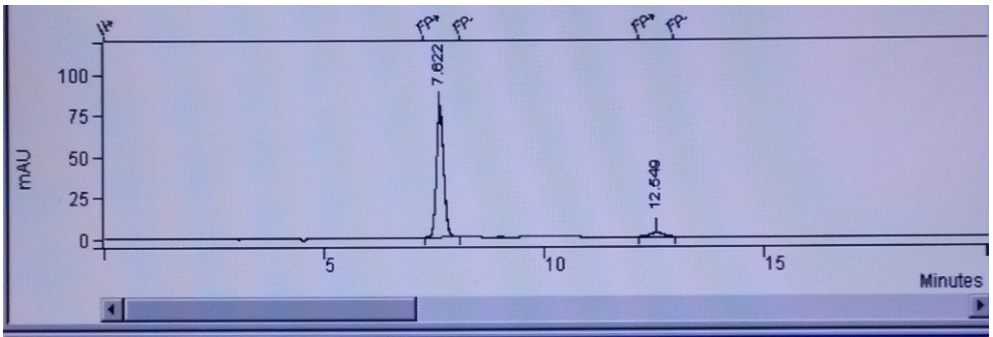


Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		49.8290	7.691	0.000	212229	BB	10.6	
2		50.1710	12.743	0.000	213686	BB	19.0	
	Totals:	100.0000		0.000	425915			

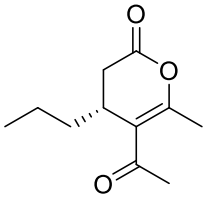


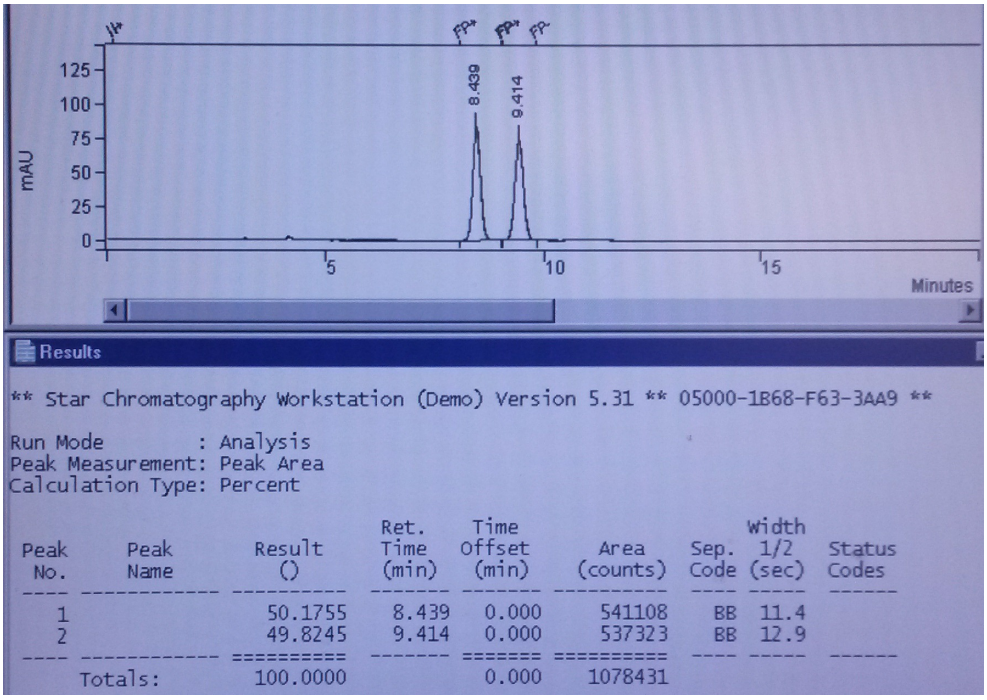
Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		95.0045	7.622	0.000	477123	BB	10.5	
2		4.9955	12.549	0.000	25088	BB	18.3	
	Totals:	100.0000		0.000	502211			



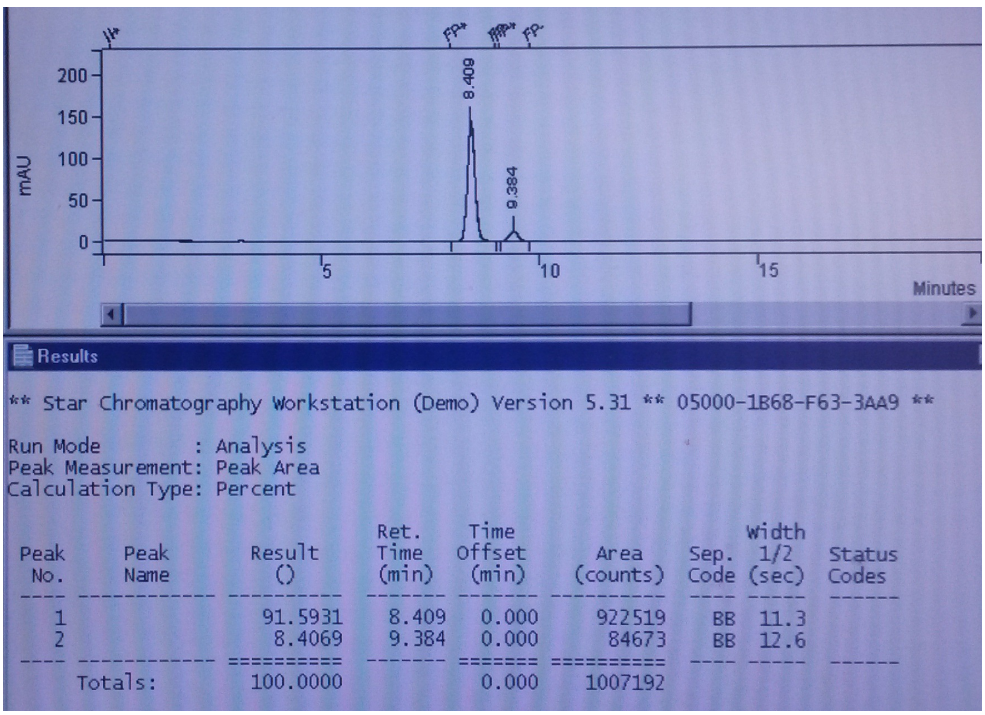


Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		50.1755	8.439	0.000	541108	BB	11.4	
2		49.8245	9.414	0.000	537323	BB	12.9	
	Totals:	100.0000		0.000	1078431			

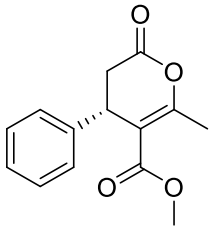


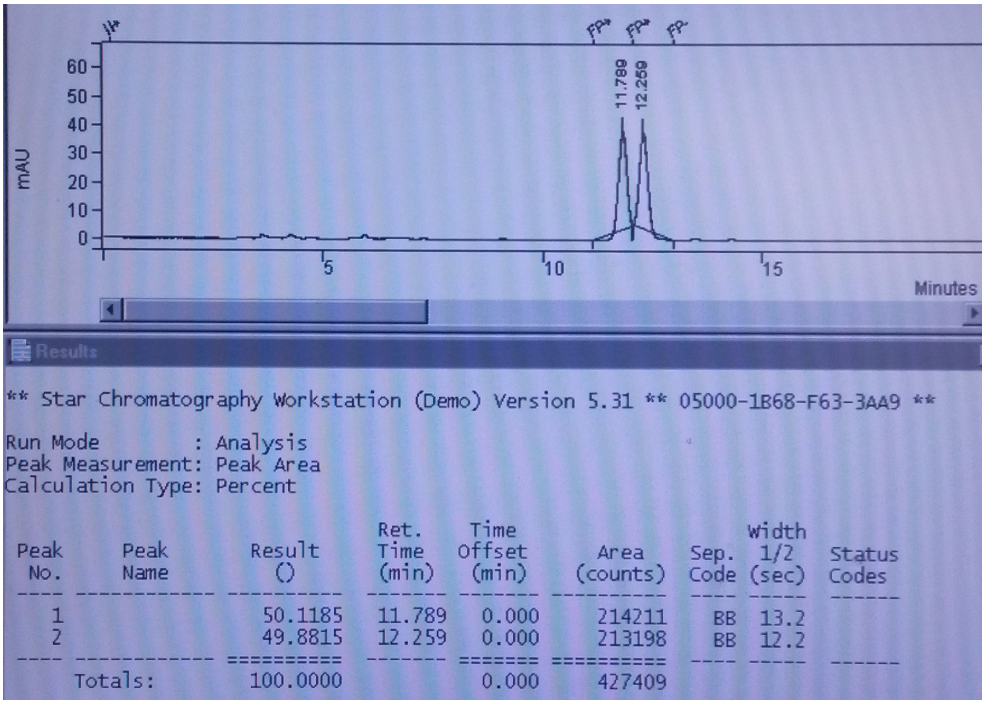
Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		91.5931	8.409	0.000	922519	BB	11.3	
2		8.4069	9.384	0.000	84673	BB	12.6	
	Totals:	100.0000		0.000	1007192			



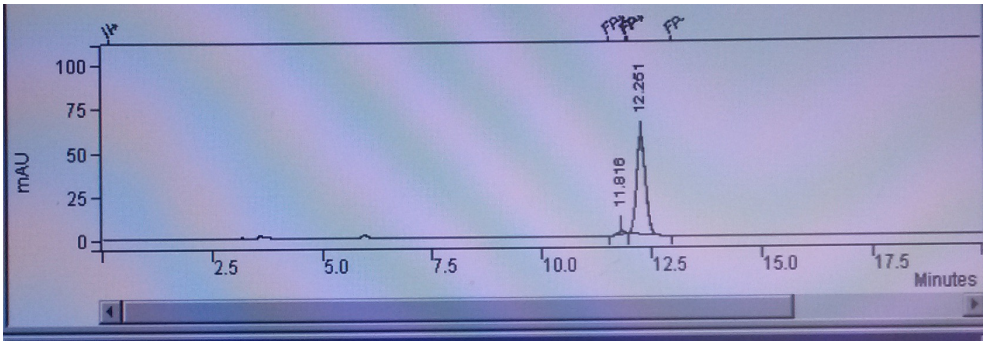


Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		50.1185	11.789	0.000	214211	BB	13.2	
2		49.8815	12.259	0.000	213198	BB	12.2	
	Totals:	100.0000		0.000	427409			

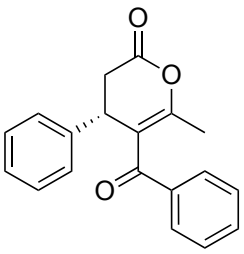


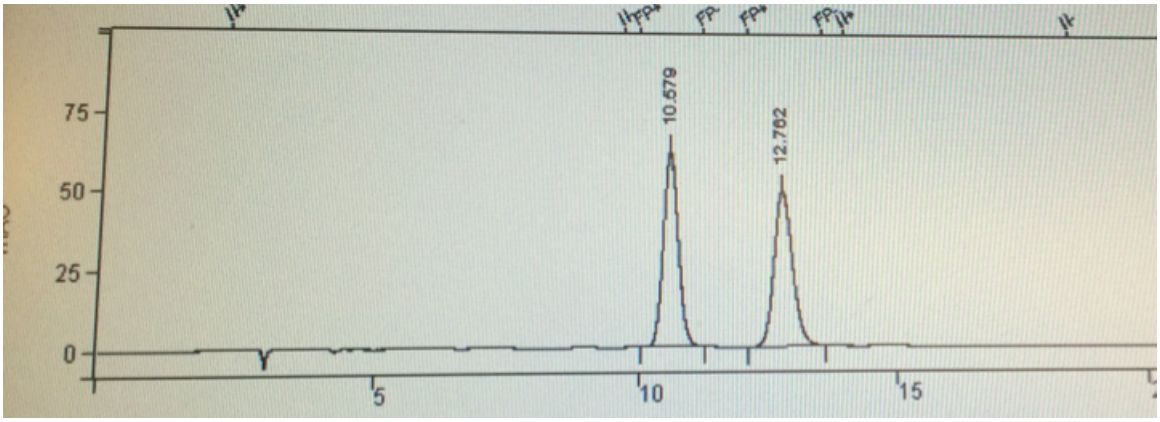
Results

** Star Chromatography Workstation (Demo) Version 5.31 ** 05000-1B68-F63-3AA9 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

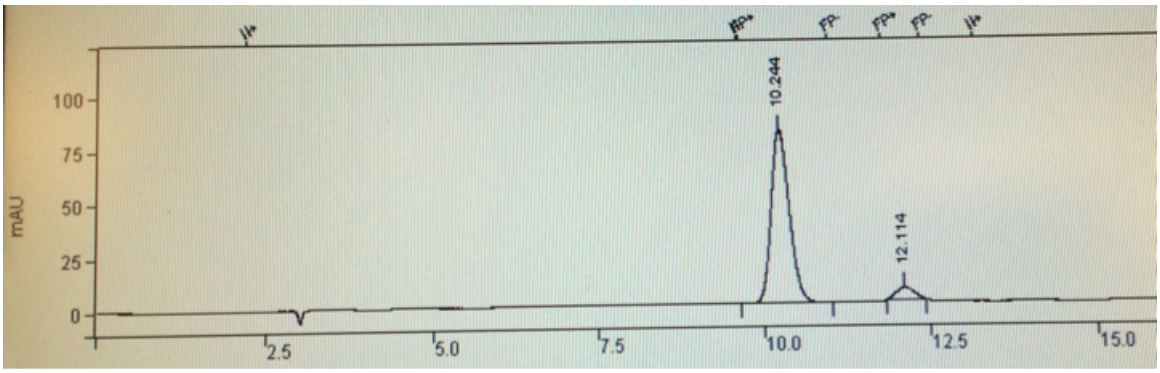
Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		2.3467	11.816	0.000	10024	BB	14.2	
2		97.6533	12.251	0.000	417116	BB	13.1	
	Totals:	100.0000		0.000	427140			





Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		50.0725	10.579	0.000	651683	BB	18.5	
2		49.9275	12.762	0.000	649796	BB	23.0	
	Totals:	100.0000		0.000	1301479			

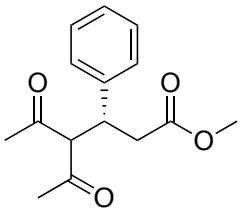
Total Unidentified Counts : 1301479 counts

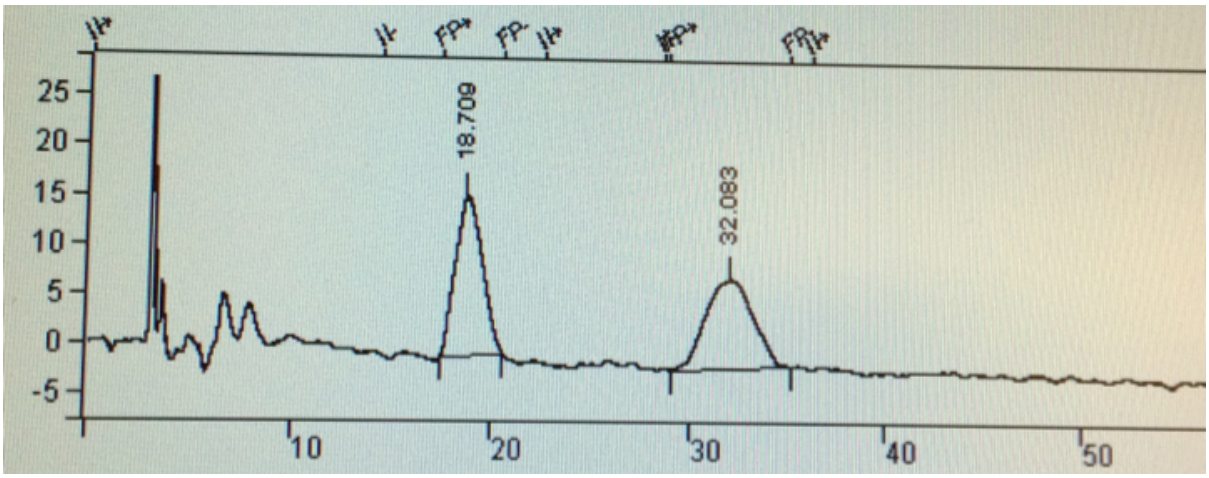


Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		93.6274	10.244	0.000	811198	BB	17.6	
2		6.3726	12.114	0.000	55213	BB	18.7	
	Totals:	100.0000		0.000	866411			

Total Unidentified Counts : 866411 counts

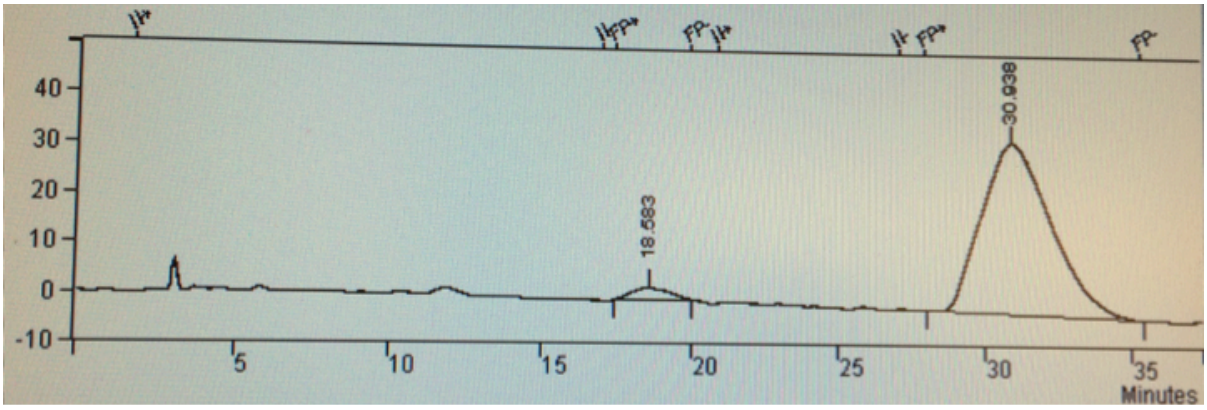
Detected Peaks: 2 Rejected Peaks: 0 Identified Peaks: 0





Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		50.0775	18.709	0.000	803856	BB	94.4	
2		49.9225	32.083	0.000	801367	BB	166.0	
	Totals:	100.0000		0.000	1605223			

Total Unidentified Counts : 1605224 counts



Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		3.4258	18.583	0.000	109622	BB	90.2	
2		96.5742	30.938	0.000	3090274	BB	154.6	
	Totals:	100.0000		0.000	3199896			

References

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(2) F.-G. Sun, L.-H. Sun and S. Ye, *Adv. Synth. Catal.*, 2011, **353**, 3134-3138

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(5) E. R. T. Robinson, C. Fallan, C. Simal, A. M. Z. Slawin, A. D. Smith, *Chem. Sci.,* 2013, **4**, 2193