

A palladium-catalysed enolate alkylation cascade for the formation of adjacent quaternary and tertiary stereocentres

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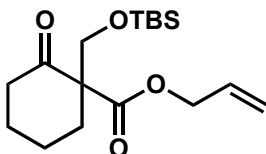
Materials and Methods

All reactions have been carried out in flame-dried Schlenk-tubes under argon atmosphere using dry solvents unless noticed otherwise. *p*-Dioxane was freshly distilled over sodium prior to use. Other solvents were dried by passage through an activated alumina column under argon. $[\text{Pd}_2(\text{dba})_3]$ and $\text{Pd}(\text{pmdba})_2$ (pmdba = di(*p*-methoxybenzylidene)acetone) were synthesised from PdCl_2 following a Literature procedure.¹ PdCl_2 was purchased from Strem and used as received. Ligands **L1–4** were prepared by the method reported in our previous work.² All other chemicals were purchased from Aldrich and used as received. Reaction temperatures were controlled by an IKA Mag temperature modulator. Thin-layer chromatography (TLC) was performed using E. Merck silica gel 60 F254 precoated plates (0.25 mm) and visualised by UV fluorescence quenching or KMnO_4 staining as described. ICN Silica gel (particle size 0.032–0.063 mm) was used for flash chromatography. Analytical chiral HPLC was performed with an Agilent 1100 Series HPLC utilizing a Chiralcel AD, OD-H or OJ columns (4.6 mm x 25 cm) obtained from Daicel Chemical Industries, Ltd with detection at $\lambda = 210$ nm (if not noted otherwise). Analytical SFC was performed with a Mettler SFC supercritical CO_2 analytical chromatography system with Chiralcel AD-H, OD-H, OJ-H, AS-H and OB-H columns. Optical rotations were measured with a Jasco P-2000 polarimeter at $\lambda = 589$ nm. ^1H and ^{13}C NMR spectra were recorded on a Varian Inova 500 spectrometer (at 500 MHz and 125 MHz respectively) and a Varian Mercury 300 spectrometer (at 300 MHz and 75 MHz respectively) and reported to CDCl_3 ($\delta = 7.26$ ppm and $\delta = 77.00$ ppm respectively). IR spectra were recorded on a Perkin Elmer Paragon 1000 spectrometer and are reported in frequency of absorption (ν). Melting points were determined using a Buechi B-545 capillary melting point apparatus and the values reported are uncorrected. High resolution mass analysis was performed on an Agilent 6200 Series Time-of-Flight LC/MS/TOF system or obtained from the Caltech Mass Spectral Facility.

Synthesis of Starting Materials.

Compounds **1a-h** have been synthesised according to our previous procedure.³ Starting materials **1e** and **1g** are new compounds. Compounds **2a-c** are literature-known but have been prepared by modified syntheses (see below).

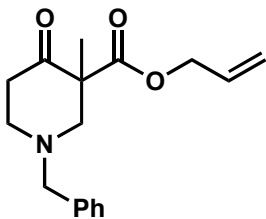
(rac)-Allyl 1-((*tert*-butyldimethylsilyloxy)methyl)-2-oxocyclohexanecarboxylate



(±)-**1e**

Prepared following a reported procedure for the corresponding TBDPS protected alcohol.³ Purified by flash chromatography (CH₂Cl₂) and isolated as the first fraction in 95% yield (colorless oil). ¹H NMR (500 MHz, CDCl₃): δ = 0.00 (s, 6H), 0.82 (s, 9H), 1.51-1.67 (m, 2H), 1.75-1.80 (m, 2H), 1.98-2.01 (m, 1H), 2.35-2.37 (m, 2H), 2.61 (d, *J* = 13.5 Hz, 1H), 3.71 (d, *J* = 9.5, 1H), 4.06 (d, *J* = 9.5 Hz, 1H), 4.55-4.63 (m, 2H), 5.20 (d, *J* = 10.5 Hz, 1H), 5.29 (d, *J* = 17.5 Hz, 1H), 5.82-5.91 (m, 1H). ¹³C NMR (125 MHz, CDCl₃): δ = -5.7, 18.07, 22.05, 27.27, 33.46, 41.23, 62.85, 65.68, 65.87, 118.63, 131.57, 169.78, 206.75. HRMS (ESI/TOF) calcd for C₁₇H₃₁O₄Si⁺: 327.1986, found: 327.2001. IR (NaCl): ν [cm⁻¹] = 3087, 2952, 2931, 2884, 2858, 1715, 1650, 1472, 1464, 1453, 1388, 1361, 1252, 1200, 1149, 1105, 1053, 981, 938, 889, 837, 778, 747, 667.

(rac)-Allyl 1-benzyl-3-methyl-4-oxopiperidine-3-carboxylate

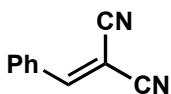


(±)-**1g**

Prepared by the following procedure. In a flame-dried 100 mL flask allyl 1-benzyl-4-oxopiperidine-3-carboxylate³ (1.75 g, 6.4 mmol, 1.0 equiv) was added to a suspension of anhydrous K₂CO₃ (3.54 g, 25.6 mmol, 4.0 equiv) in acetone (24 mL). Iodomethane (0.80

mL, 12.8 mmol, 2.0 equiv) was added and the resulting reaction mixture heated to 50°C for 10 h. The reaction was allowed to cool down to room temperature and TLC control showed the complete consumption of the starting material. The mixture was filtered and the residue washed with acetone several times. The filtrate was concentrated and the resulting oil purified by flash chromatography (hexanes/EtOAc 10:1, R_f = 0.6 in hexanes/EtOAc 5:1). 9.8% yield (180.3 mg, 0.63 mmol, colorless oil). ^1H NMR (500 MHz, CDCl_3): δ = 1.25 (s, 3H), 2.17 (d, J = 12.0 Hz, 1H), 2.37-2.43 (m, 2H), 2.91 (ddd, J = 6.5, 12.5, 15.0 Hz, 1H), 3.05 (tdd, J = 2.5, 7.0, 11.5 Hz, 1H), 3.48 (dd, J = 2.5, 11.5 Hz, 1H), 3.59 (s, 2H), 4.60 (tdd, J = 1.5, 6.0, 13.5 Hz, 1H), 4.69 (tdd, J = 1.5, 6.0, 13.5 Hz, 1H), 5.24 (ddd, J = 1.0, 2.5, 10.5 Hz, 1H), 5.32 (ddd, J = 1.5, 3.0, 17.0 Hz, 1H), 5.88 (tdd, J = 6.0, 10.5, 17.0 Hz, 1H), 7.25-7.34 (m, 5H). ^{13}C NMR (125 MHz, CDCl_3): δ = 17.76, 40.17, 53.48, 57.37, 61.74, 62.67, 65.78, 118.52, 127.28, 131.60, 137.80, 172.29, 206.19. HRMS (ESI/TOF) calcd for $\text{C}_{17}\text{H}_{22}\text{NO}_3^+$: 288.1594, found: 288.1604. IR (NaCl): ν [cm^{-1}] = 3028, 2936, 2806, 1719, 1495, 1453, 1347, 1314, 1230, 1194, 1129, 1071, 999, 932, 742, 699.

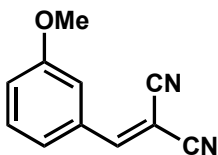
Benzylidenemalononitrile⁴



2a

This compound is commercially available (CAS 2700-22-3). A 250 mL round-bottom flask is charged with Na_2SO_4 (50 g) under air. Dichloromethane (150 mL) is added followed by malononitrile (1.98 g, 30 mmol, 1.0 equiv) and benzaldehyde (3.35 mL, 33 mmol, 1.1 equiv). Piperidine (0.3 mL, 3 mmol, 0.1 equiv) is added and the mixture is stirred for 18 h in the capped flask. The solids are filtered off and rinsed with additional dichloromethane. The combined solutions are concentrated in vacuo and the crude mixture is submitted to column chromatography (CH_2Cl_2). The first fraction contains product and a small amount of benzaldehyde. Concentration yields a brown-white powder which is washed with hexanes (4x10mL) to remove the remaining benzaldehyde. 85% yield.

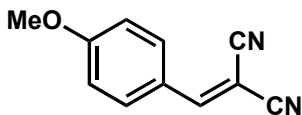
3-Methoxybenzylidenemalononitrile⁴



2b

This compound has been reported previously and was prepared following the procedure for benzylidenemalononitrile using *m*-anisaldehyde (4.0 mL, 31 mmol). The crude mixture was filtrated, the mother liquor concentrated and the product crystallised from dichloromethane. 62% yield (3.4 g, 18.5 mmol).

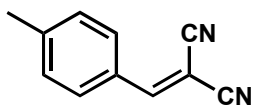
4-Methoxybenzylidenemalononitrile⁴



2c

This compound has been reported previously and is commercially available (CAS 2826-26-8). It was prepared following the procedure for **2a**.

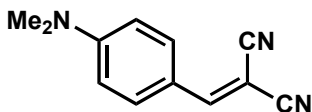
4-Methylbenzylidenemalononitrile⁵



2d

This compound has been reported previously and is commercially available (CAS 2826-25-7). It was prepared following the procedure for **2a**.

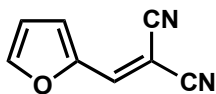
4-(Dimethylamino)benzylidenemalononitrile⁵



2e

This compound has been reported previously and is commercially available (CAS 2826-28-0). It was prepared following the procedure for **2a**.

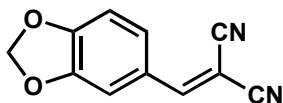
Furfurylidenemalononitrile⁴



2f

This compound is commercially available (CAS 3237-22-7). Destillation of furfural is not necessary but recommended prior to use. A 250 mL round-bottom flask is charged with Na₂SO₄ (50 g) under air. Dichloromethane (150 mL) is added followed by malononitrile (1.98 g, 30 mmol, 1.0 equiv) and furfural (2.8 mL, 33 mmol, 1.1 equiv). Piperidine (0.3 mL, 3 mmol, 0.1 equiv) is added and the mixture is stirred for 18 h in the capped flask. The reaction mixture is filtered and the solid residue thoroughly rinsed with dichloromethane and ethyl acetate. The intensely red colored organic solutions are combined and the solvent is evaporated under reduced pressure. The crude solid is washed with hot carbon tetrachloride (4-5 times) and the extract is concentrated to a volume of ~10 mL. Standing overnight at -20°C yields deep-orange crystals of pure **2d** (1.51 g, 10.5 mmol, 35%).

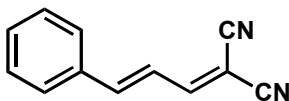
5-Benzodioxolymethylenemalononitrile⁵



2g

This compound has been reported previously (CAS 2972-82-9). It was prepared following the procedure for **2a**.

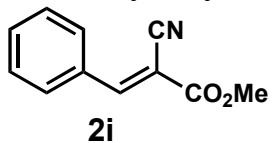
(*E*)-3-Phenylallylidenemalononitrile⁶



2h

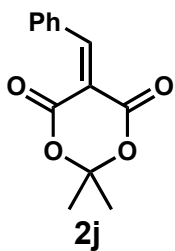
This compound has been reported previously (CAS 41109-96-0). It was prepared following the procedure for **2a**.

(E)-Methyl 2-cyano-3-phenylacrylate⁷



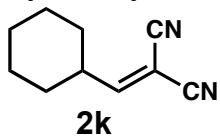
This compound has been reported previously and is commercially available (CAS 1214-54-6). It was prepared following the procedure for **2a**.

5-Benzylidene-2,2-dimethyl-1,3-dioxane-4,6-dione⁸



This compound has been reported previously (CAS 3695-84-9). It was prepared following the procedure for **2a**.

Cyclohexylmethylenemalononitrile⁹

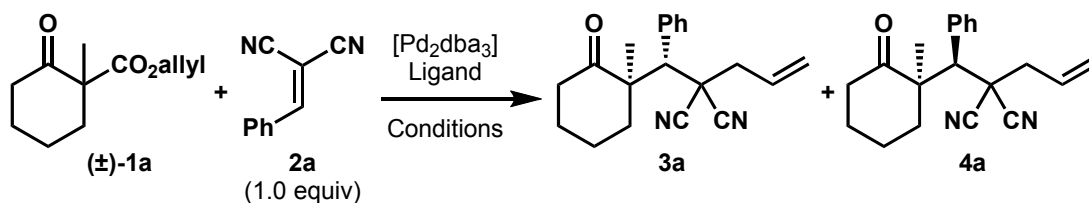


This compound has been reported previously (CAS 73776-46-2). It was prepared following the procedure for **2a**.

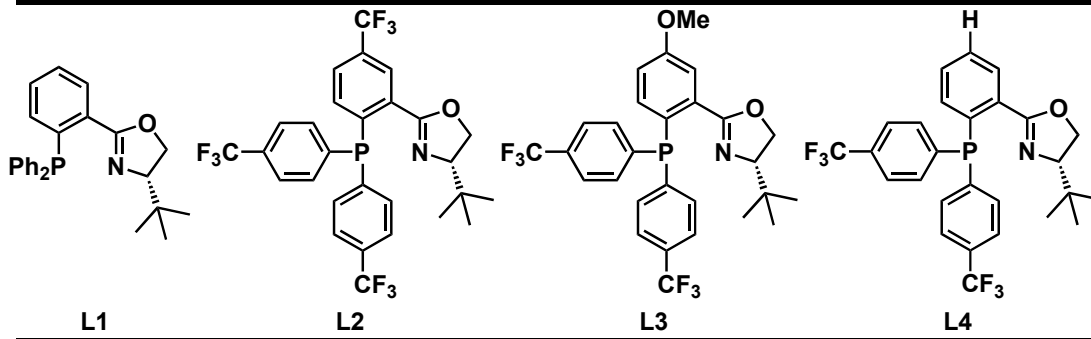
Screening Experiments

Optimisation of the Reaction Conditions. During a short ligand screen it was found that electron withdrawing substituents in the ligand backbone significantly enhance the enantiomeric excess of both diastereomers (see Table S1). Careful adjustment of solvent and reaction temperature finally led to the conditions as described in the general procedure (see below). The screening experiments were carried out following that general procedure on a 0.1 mmol scale and selected results are shown here.

Table S1. Reaction Optimisation.



entry	ligand	c	T	t	conv ^b	d.r. (3a:4a)	ee(3a) ^c	ee(4a) ^c
1	L1	0.03 M	60 °C	15 h	82%	1 : 3.1	42%	63%
2	L2	0.03 M	60 °C	15 h	87%	1 : 4.0	81%	84%
3 ^d	L2	0.03 M	60 °C	15 h	52%	1 : 3.4	66%	75%
4	L2	0.03 M	40 °C	15 h	88%	1 : 4.1	71%	83%
5	L2	0.1 M	40 °C	24 h	99%	1 : 6.1	77%	87%
6	L3	0.1 M	40 °C	15 h	15%	1 : 3.8	nd	nd
7	L4	0.1 M	40 °C	15 h	27%	1 : 2.8	nd	nd



^a Reactions carried out on a 0.1 mmol scale. ^b Conversion to product determined by crude NMR. ^c enantiomeric excess determined by chiral HPLC after filtration through a short silica column. ^d reaction carried out with 2.5 mol% $[Pd_2dba_3]$ and 6.25 mol% **L2**.

Conditions for the Determination of Enantiomeric Excess.

Table S2. Conditions for the Determination of Enantiomeric Excess.

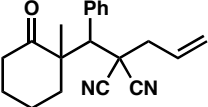

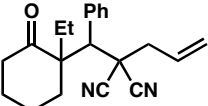
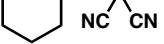
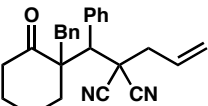

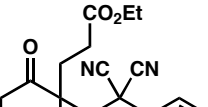
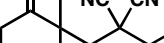
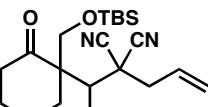
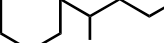
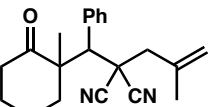
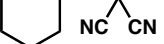
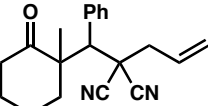

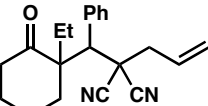

Entry	Compound	Analysis Conditions	Retention times (min)		ee ^a
			t ₁	t ₂	
1		HPLC, OD-H, 10% 2-propanol in hexane, 1 mL/min	7.9	8.4 ^b	77%
2		HPLC, OD-H, 10% 2-propanol in hexane, 1 mL/min	9.7 ^b	12.1	87%
3		HPLC, OD-H, 10% 2-propanol in hexane, 1 mL/min	6.6 ^b	8.1	95%
4		HPLC, OD-H, 10% 2-propanol in hexane, 1 mL/min	8.0	8.4 ^b	99%
5		HPLC, OJ, 4% EtOH in hexane, 1 mL/min	17.9 ^b	23.8	85% (93%)
6		HPLC, OJ, 4% EtOH in hexane, 1 mL/min	17.2 ^b	22.6	88% (94%)
7		HPLC, OD-H, 3% 2-propanol in hexane, 1 mL/min	14.9 ^b	16.0	82%
8		HPLC, OJ, 5% EtOH in hexane, 1 mL/min	24.6 ^b	29.9	89%
9		SFC, AD-H, 10% 2-propanol in CO ₂ , 2 mL/min, 40°C, 100bar	2.4 ^b	2.8	69%
10		HPLC, AD, 3% 2-propanol in hexane, 1 mL/min	6.0 ^b	6.3	70%
11		HPLC, OD-H, 10% 2-propanol in hexane, 1 mL/min	6.7	7.2 ^b	75%
12		HPLC, OD-H, 10% 2-propanol in hexane, 1 mL/min	8.1 ^b	10.6	81%
13		SFC, AD-H, 20% 2-propanol in CO ₂ , 2 mL/min, 40°C, 100bar	3.0	3.7	nd
14		SFC, AD-H, 20% 2-propanol in CO ₂ , 2 mL/min, 40°C, 100bar	3.0 ^b	3.7	89%
15		SFC, OD-H, 10% 2-propanol in CO ₂ , 2 mL/min, 40°C, 100bar	7.1 ^b	7.9	71%
16		SFC, OD-H, 20% 2-propanol in CO ₂ , 2 mL/min, 40°C, 100bar	4.4	4.9 ^b	97%

Table S2 (continued).

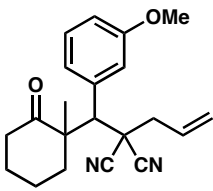
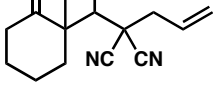
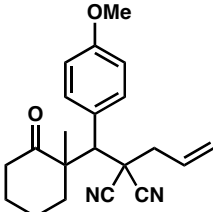
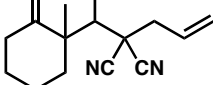
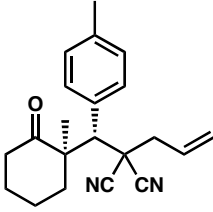
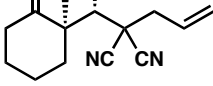
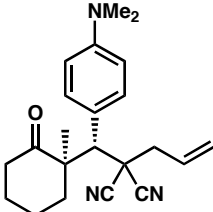
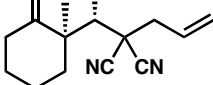
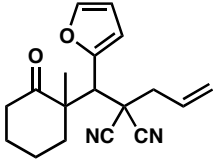
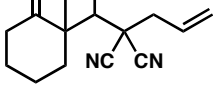
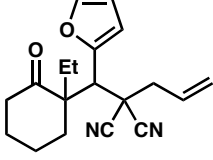
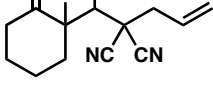
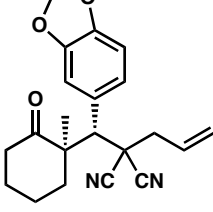
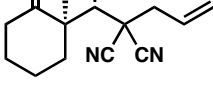
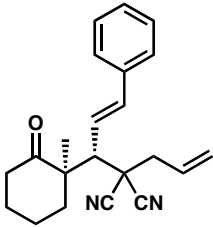
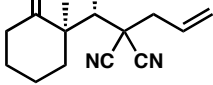
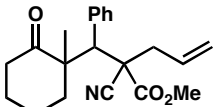
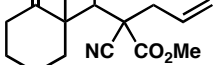
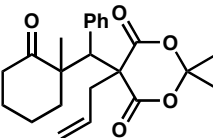
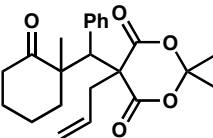
17		HPLC, OD-H, 3% 2-propanol in hexane, 1 mL/min	8.1 ^b	8.5	71%
18		SFC, AD-H, 20% 2-propanol in CO ₂ , 2 mL/min, 40°C, 100bar	2.6 ^b	3.0	86%
19		SFC, OJ-H, 10% 2-propanol in CO ₂ , 2 mL/min, 40°C, 100bar	3.3	3.8 ^b	73%
20		SFC, AD-H, 10% 2-propanol in CO ₂ , 2 mL/min, 40°C, 100bar	6.5 ^b	7.3	88%
21		SFC, OJ-H, 5% IPA, 2ml/min	3.7	4.1 ^b	75%
22		SFC, AD-H, 10% IPA, 2ml/min	4.9 ^b	5.8	87%
23		SFC, OJ-H, 10% IPA, 2ml/min	6.1 ^b	10.1	78%
24		SFC, AD-H, 10% IPA, 2ml/min	7.8	8.9 ^b	99%
25		HPLC, OD-H, 10% 2-propanol in hexane, 1 mL/min	7.3 ^b	8.0	65%
26		HPLC, AD, 10% 2-propanol in hexane, 1 mL/min	8.9 ^b	10.0	81%
27		HPLC, AD, 10% 2-propanol in hexane, 1 mL/min	6.9 ^b	8.6	89%
28		HPLC, OD-H, 10% 2-propanol in hexane, 1 mL/min	7.9	8.5 ^b	96%
29		SFC, AD-H, 20% IPA, 2ml/min	2.7 ^b	3.1	58%
30		SFC, AD-H, 20% IPA, 2ml/min	3.1 ^b	3.5	95%

Table S2 (continued).

31		SFC, 10% IPA, AD-H, 2ml/min	4.5 ^b	5.2	64%
32		SFC, OB-H, 10% IPA, 2ml/min	4.6 ^b	5.7	82%
33		SFC, OJ-H, 1% IPA, 2ml/min	9.8	13.7 ^b	64%
34		SFC, OJ-H, 1% IPA, 2ml/min	16.4	19.1 ^b	75%
35		SFC, AD-H, 20% IPA, 2ml/min	2.5 ^b	3.6	22%
36		SFC, AD-H, 10% IPA, 2ml/min	7.6 ^b	8.2	43%

^a Results shown in brackets were obtained from the reaction carried out at 23 °C. ^b major enantiomer.

General Procedures

General Procedure for Enantioselective Insertion–Allylation. A flame-dried 50 mL Schlenk-tube is charged with [Pd₂(dba)₃] (13.7 mg, 0.015 mmol, 5 mol%) and **L2** (22.2 mg, 0.0375 mmol, 12.5 mol%) under argon atmosphere and 3 mL of freshly distilled *p*-dioxane are added. After stirring for 30 min at 23 °C, **1** (0.3 mmol, 1.0 equiv) and **2** (0.3 mmol, 1.0 equiv) are added simultaneously. The resulting yellow-green solution is stirred at the reported temperature for the reported amount of time (see Table 1). The consumption of starting material can be monitored by TLC (KMnO₄ stain) or by analysis of a small NMR sample. The solvent is removed under reduced pressure and the diastereomeric ratio determined by crude ¹H NMR. Isolation and separation of products **3** and **4** was achieved by flash chromatography in Hexanes/Ethyl Acetate mixtures (see compound description) in the given combined yields (Table 1, manuscript).

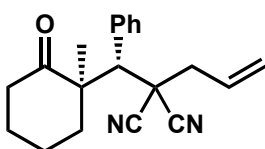
General Procedure for the Synthesis of Racemic Products. Racemic products were synthesised on a 0.3 mmol scale following the general procedure above and using dppe (1,2-bis(diphenylphosphino)ethane) as achiral ligand.

Characterisation Data for New Products.

Please note that the absolute configuration was determined only for compounds **3b** and **4b** via X-ray analysis (vide infra). The absolute configuration for all other products **3** and **4** has been assumed based on this result.

Isolated yields are reported in Table 1 (see manuscript). For respective HPLC or SFC conditions, please refer to Table S2.

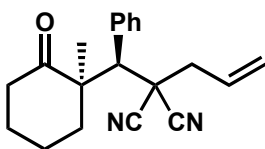
2-Allyl-2-((R)-((R)-1-methyl-2-oxocyclohexyl)(phenyl)methyl)malononitrile



3a

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 10:1 to 8:1 to 4:1, MnO₄ stain, $R_f = 0.4$ in hexanes/EtOAc 6:1) and isolated as a colorless oil. $[\alpha]_D^{25} = -44.17^\circ$ ($c = 0.21$, CH₂Cl₂, 77% ee). ¹H NMR (300 MHz, CDCl₃): $\delta = 0.80$ (s, 3H), 1.83 (dddd, $J = 3.6, 8.7, 12.3, 16.2$ Hz, 1H), 1.99-2.07 (m, 3H), 2.38 (d, $J = 13.2$ Hz, 1H), 2.47-2.64 (m, 5H), 4.33 (s, 1H), 5.30 (ddd, $J = 1.2, 2.7, 17.1$ Hz, 1H), 5.41 (ddd, $J = 1.2, 2.4, 10.2$ Hz, 1H), 5.90 (dddd, $J = 6.6, 7.8, 10.2, 17.1$ Hz, 1H), 7.17 (d, $J = 6.9$ Hz, 1H), 7.32-7.46 (m, 3H), 7.78 (d, $J = 7.5$ Hz, 1H). ¹³C NMR (75 MHz, CDCl₃): $\delta = 20.83, 24.11, 25.40, 32.91, 38.11, 39.49, 42.52, 52.04, 53.04, 115.69, 116.03, 123.07, 128.82, 128.91, 129.02, 133.69, 133.88, 212.55$. HRMS (ESI/TOF) calcd for C₂₀H₂₃N₂O⁺: 307.1805, found: 307.1802. IR (NaCl): ν [cm⁻¹] = 3084, 3032, 2945, 2872, 2244, 1702, 1493, 1454, 1417, 1229, 1129, 1088, 991, 936, 806, 771, 708.

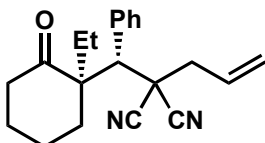
2-Allyl-2-((S)-((R)-1-methyl-2-oxocyclohexyl)(phenyl)methyl)malononitrile



4a

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 10:1 to 8:1 to 4:1, MnO₄ stain, $R_f = 0.3$ in hexanes/EtOAc 6:1) and isolated as a colorless oil. $[\alpha]_D^{25} = +38.33^\circ$ ($c = 0.63$, CH₂Cl₂, 87% ee). ¹H NMR (300 MHz, CDCl₃): $\delta = 1.33$ (ddd, $J = 4.2, 11.4, 13.8$ Hz, 1H), 1.47 (dddd, $J = 2.1, 3.9, 5.7, 13.8$ Hz, 1H), 1.58-1.77 (m, 3H), 1.79 (s, 3H), 1.95-2.03 (m, 1H), 2.47 (ddd, $J = 3.9, 4.8, 13.2$ Hz, 1H), 2.53-2.62 (m, 2H), 2.76 (ddd, $J = 6.0, 11.4, 13.2$ Hz, 1H), 3.95 (s, 1H), 5.29 (d, $J = 17.1$ Hz, 1H), 5.40 (d, $J = 10.2$ Hz, 1H), 5.93 (dddd, $J = 6.6, 7.2, 10.2, 17.1$ Hz, 1H), 7.12 (br, 1H), 7.38 (br, 3H), 7.80 (br, 1H). ¹³C NMR (75 MHz, CDCl₃): $\delta = 19.37, 20.86, 28.61, 38.32, 39.36, 40.85, 42.93, 52.59, 53.20, 115.71, 16.15, 123.16, 128.81, 128.82, 128.88, 128.94, 133.90, 214.15$. HRMS (ESI/TOF) calcd for C₂₀H₂₃N₂O⁺: 307.1805, found: 307.1793. IR (NaCl): ν [cm⁻¹] = 3084, 2944, 2868, 2245, 1703, 1494, 1454, 1385, 1307, 1262, 1227, 1112, 1088, 988, 935, 711.

2-Allyl-2-((R)-((R)-1-ethyl-2-oxocyclohexyl)(phenyl)methyl)malononitrile



3b

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 10:1 to 8:1 to 4:1, MnO₄ stain, $R_f = 0.45$ in hexanes/EtOAc 6:1) and isolated as a colorless oil. Crystallised from 2-propanol as colorless crystals. mp = 123-124°C. $[\alpha]_D^{25} = -86.18^\circ$ ($c = 0.30$, CH₂Cl₂, 95% ee). ¹H NMR (500 MHz, CDCl₃): $\delta = 0.39$ (t, $J = 7.5$ Hz, 3H), 1.11 (qd = 7.5, 15.0 Hz, 1H), 1.86 (qd, = 7.5, 15.0 Hz, 1H), 1.94 (tt, $J = 4.0, 13.0$ Hz, 1H), 1.98-2.05 (m, 2H), 2.08 (tt, $J = 3.5, 13.0$ Hz, 1H), 2.35 (ddd, $J = 7.0, 13.0, 16.0$ Hz, 1H), 2.45 (tdd, $J = 2.5, 3.0, 13.0$ Hz, 1H), 2.56 (tdd, $J = 1.0, 6.5, 14.0$ Hz, 1H), 2.57-2.65 (m, 2H), 2.66 (dd, $J = 8.0, 14.0$ Hz, 1H), 4.33 (s, 1H), 5.31 (ddd,

$J = 1.0, 2.5, 17.0$ Hz, 1H), 5.40 (ddd, $J = 1.0, 1.5, 11.0$ Hz, 1H), 5.92 (dddd, $J = 7.0, 8.0, 11.0, 17.0$ Hz, 1H), 7.19 (d, $J = 7.0$ Hz, 1H), 7.34 (t, $J = 7.0$ Hz, 1H), 7.39 (td, $J = 1.0, 7.5$ Hz, 1H), 7.44 (t, $J = 7.0$ Hz, 1H), 7.76 (d, $J = 7.5$ Hz, 1H). ^{13}C NMR (125 MHz, CDCl_3): $\delta = 7.60, 21.14, 24.29, 27.94, 33.16, 39.35, 39.99, 42.51, 50.77, 55.72, 115.83, 116.07, 122.89, 128.49, 128.81, 128.94, 129.00, 129.46, 133.45, 133.72, 211.02$. HRMS (ESI/TOF) calcd for $\text{C}_{21}\text{H}_{25}\text{N}_2\text{O}^+$: 321.1961, found: 321.1957. IR (NaCl): ν [cm^{-1}] = 3086, 3062, 3030, 2946, 2875, 2244, 1698, 1644, 1602, 1582, 1493, 1454, 1418, 1388, 1317, 1268, 1224, 1127, 1090, 1032, 991, 935, 802, 747, 710.

Determination of the Absolute Configuration of 3b

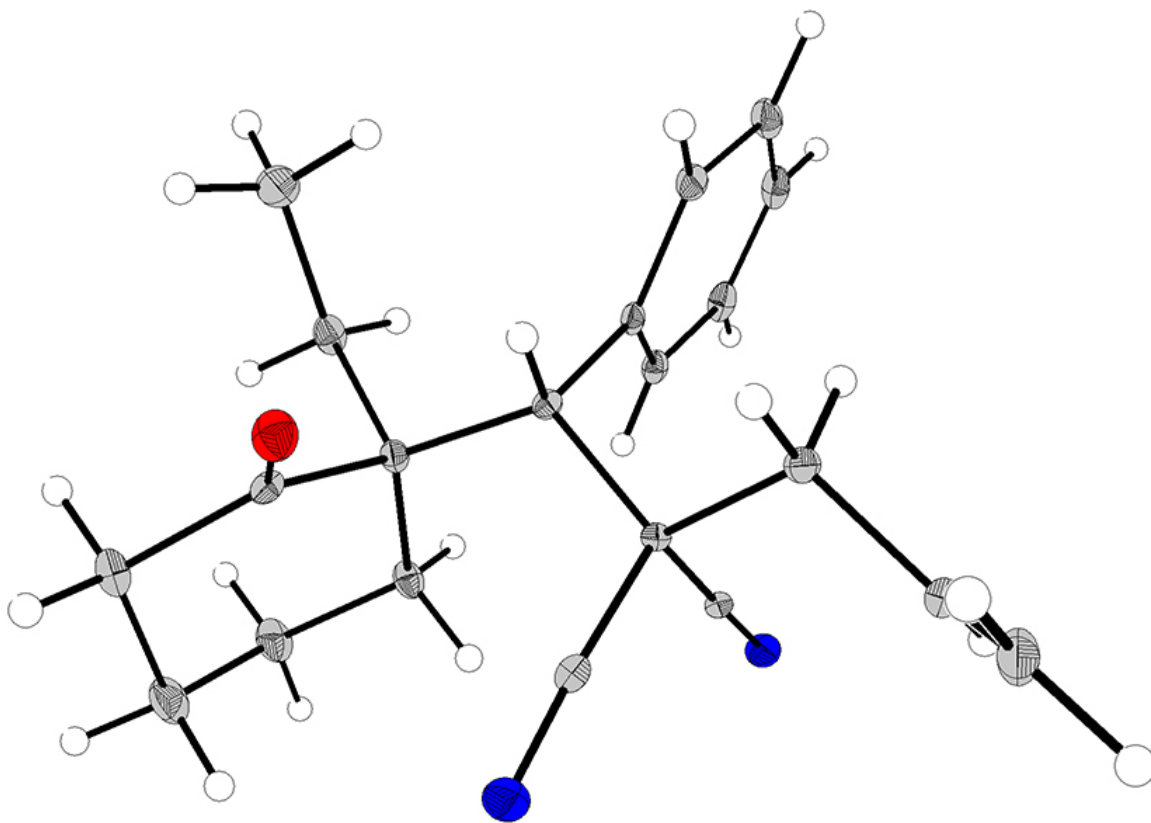


Figure S1. Crystal Structure of compound **3b**.¹⁰

Table S3. Crystal data and structure refinement for JST01 (CCDC 732239).

Empirical formula	C ₂₁ H ₂₄ N ₂ O
Formula weight	320.42
Crystallisation Solvent	2-propanol
Crystal Habit	Prism
Crystal size	0.35 x 0.18 x 0.16 mm ³
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker SMART 1000
Wavelength	1.54178 Å CuK α
Data Collection Temperature	100(2) K
θ range for 95430 reflections used in lattice determination	34.76 to 69.78°
Unit cell dimensions	a = 10.2994(3) Å b = 12.6537(4) Å c = 13.9603(4) Å
Volume	1819.38(9) Å ³
Z	4
Crystal system	Orthorhombic
Space group	P2 ₁ 2 ₁ 2 ₁
Density (calculated)	1.170 Mg/m ³
F(000)	688
Data collection program	Bruker SMART v5.630
θ range for data collection	4.72 to 69.77°
Completeness to $\theta = 69.77^\circ$	98.9 %
Index ranges	-10 \leq h \leq 12, -15 \leq k \leq 15, -16 \leq l \leq 16
Data collection scan type	ω scans at 17 settings
Data reduction program	Bruker SAINT v6.45A
Reflections collected	27441
Independent reflections	3390 [R _{int} = 0.0833]
Absorption coefficient	0.561 mm ⁻¹
Absorption correction	None
Max. and min. transmission	0.9155 and 0.8277

Table S3 (cont.)**Structure solution and Refinement**

Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F ²
Data / restraints / parameters	3390 / 0 / 313
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F ²	1.975
Final R indices [I>2σ(I), 3232 reflections]	R1 = 0.0293, wR2 = 0.0643
R indices (all data)	R1 = 0.0311, wR2 = 0.0650
Type of weighting scheme used	Sigma
Weighting scheme used	w=1/σ ² (Fo ²)
Max shift/error	0.000
Average shift/error	0.000
Absolute structure determination	Anomalous differences
Absolute structure parameter	-0.1(2)
Largest diff. peak and hole	0.150 and -0.183 e.Å ⁻³

Special Refinement Details

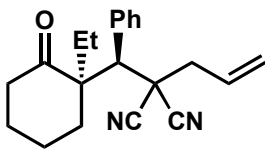
Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

The absolute structure was determined from anomalous differences and confirmed by evaluating refinement of both conformations before choosing the final model. The chirality at C(7) is R.

Refinement of F² against ALL reflections. The weighted R-factor (wR) and goodness of fit (S) are based on F², conventional R-factors (R) are based on F, with F set to zero for negative F². The threshold expression of F² > 2σ(F²) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F² are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

2-Allyl-2-((S)-((R)-1-ethyl-2-oxocyclohexyl)(phenyl)methyl)malononitrile



4b

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 10:1 to 8:1 to 4:1, MnO₄ stain, $R_f = 0.35$ in hexanes/EtOAc 6:1) and isolated as a colorless oil. Crystallised from 2-propanol as colorless crystals. $[\alpha]_D^{25} = -42.71$ ($c = 0.22$, CH₂Cl₂, 99% ee). mp = 95-96°C. ¹H NMR (500 MHz, CDCl₃): $\delta = 0.80$ (t, $J = 7.0$ Hz, 3H), 1.67-1.86 (m, 4H), 1.82 (ddd, $J = 7.5, 7.5, 15.0$ Hz, 1H), 2.10 (tdd, $J = 2.0, 3.5, 14.5$ Hz, 1H), 2.23 (dt, $J = 5.0, 9.0$ Hz, 1H), 2.27 (ddd, $J = 7.5, 7.5, 15.0$ Hz, 1H), 2.35-2.48 (m, 3H), 2.56 (tdd, $J = 1.0, 6.5, 14.0$ Hz, 1H), 3.51 (s, 1H), 5.26 (ddd, $J = 1.0, 2.5, 17.0$ Hz, 1H), 5.39 (ddd, $J = 1.0, 2.0, 10.0$ Hz, 1H), 5.87 (dddd, $J = 6.5, 8.0, 10.0, 17.0$ Hz, 1H), 7.17 (br, 1H), 7.35 (s, 3H), 7.86 (br, 1H). ¹³C NMR (125 MHz, CDCl₃): $\delta = 8.62, 21.10, 24.71, 26.88, 33.28, 38.37, 39.55, 43.80, 54.21, 56.37, 115.99, 116.00, 123.50, 128.45, 128.47, 128.48, 128.51, 128.57, 128.67, 134.33, 212.65$. HRMS (ESI/TOF) calcd for C₂₁H₂₈N₃O⁺: 338.2227, found: 338.2211. IR (NaCl): ν [cm⁻¹] = 3066, 2947, 2874, 2244, 1699, 1492, 1456, 1419, 1385, 1316, 1126, 1088, 991, 936, 763, 710.

Determination of the Absolute Configuration of 4b

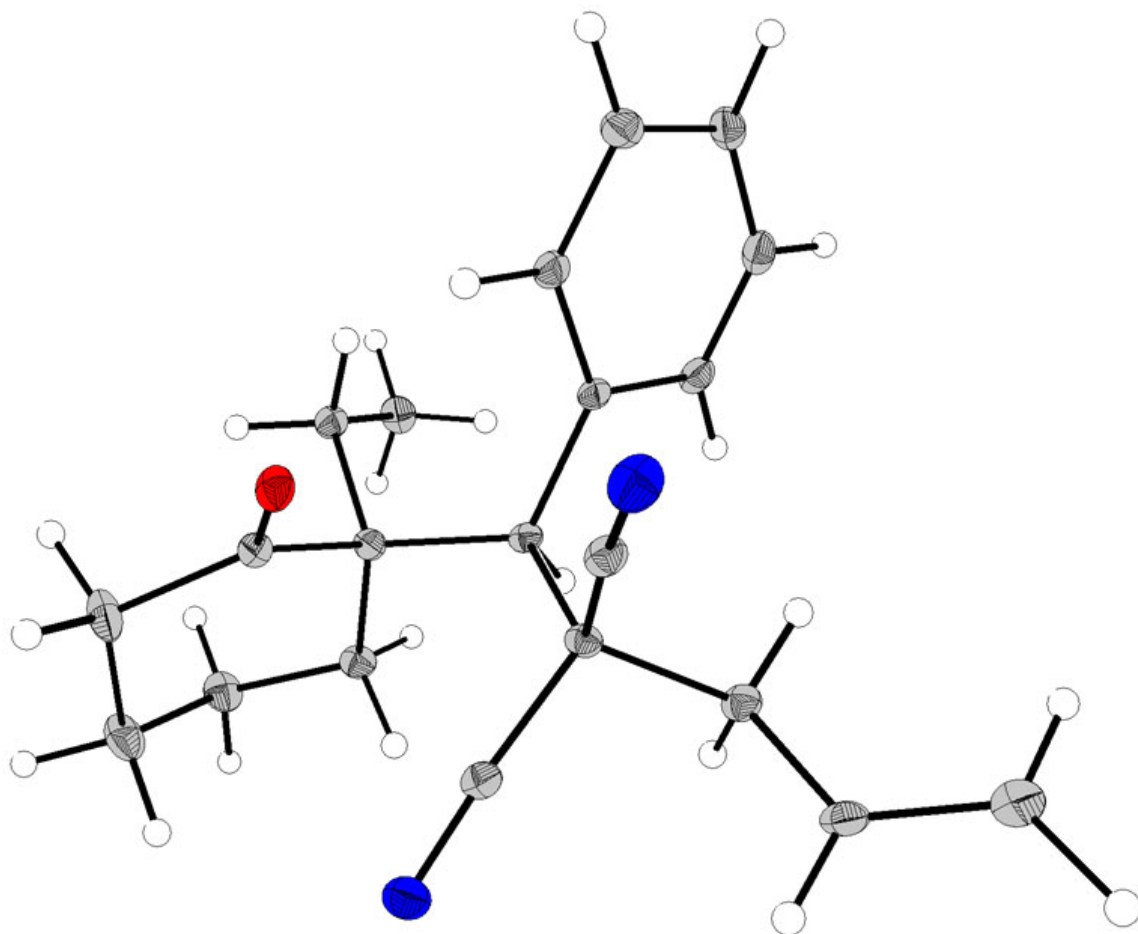


Figure S2. Crystal Structure of Compound **4b**.¹¹

Table S4. Crystal data and structure refinement for JST02 (CCDC 732240).

Empirical formula	C ₂₁ H ₂₄ N ₂ O
Formula weight	320.42
Crystallisation Solvent	2-propanol
Crystal Habit	Plate
Crystal size	0.28 x 0.28 x 0.08 mm ³
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker SMART 1000
Wavelength	1.54178 Å CuK α
Data Collection Temperature	100(2) K
θ range for 8004 reflections used in lattice determination	34.91 to 69.31°
Unit cell dimensions	a = 6.7294(2) Å b = 15.7191(5) Å c = 17.4651(6) Å
Volume	1827.29(10) Å ³
Z	4
Crystal system	Monoclinic
Space group	P2 ₁
Density (calculated)	1.165 Mg/m ³
F(000)	688
Data collection program	Bruker SMART v5.630
θ range for data collection	2.56 to 69.40°
Completeness to $\theta = 69.40^\circ$	97.7 %
Index ranges	-6 \leq h \leq 7, -19 \leq k \leq 18, -21 \leq l \leq 21
Data collection scan type	ω scans at 17 settings
Data reduction program	Bruker SAINT v6.45A
Reflections collected	27012
Independent reflections	6608 [R _{int} = 0.0824]
Absorption coefficient	0.559 mm ⁻¹
Absorption correction	None
Max. and min. transmission	0.9566 and 0.8592

Table S4 (cont.)**Structure solution and Refinement**

Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	6608 / 1 / 625
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F^2	1.512
Final R indices [$I > 2\sigma(I)$, 6151 reflections]	$R1 = 0.0355$, $wR2 = 0.0684$
R indices (all data)	$R1 = 0.0388$, $wR2 = 0.0694$
Type of weighting scheme used	Sigma
Weighting scheme used	$w = 1/\sigma^2(F_o^2)$
Max shift/error	0.001
Average shift/error	0.000
Absolute structure determination	Anomalous differences
Absolute structure parameter	0.14(17)
Largest diff. peak and hole	0.161 and -0.189 e.Å ⁻³

Special Refinement Details

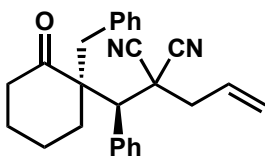
Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

The absolute structure was determined from anomalous differences and confirmed by evaluating refinement of both conformations before choosing the final model. The chirality at C(7) in both molecules is S.

Refinement of F^2 against ALL reflections. The weighted R-factor (wR) and goodness of fit (S) are based on F^2 , conventional R-factors (R) are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F , and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

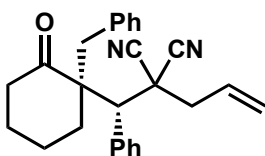
2-Allyl-2-((R)-((S)-1-benzyl-2-oxocyclohexyl)(phenyl)methyl)malononitrile



3c

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 12:1 to 4:1, MnO₄ stain, $R_f = 0.40$ in hexanes/EtOAc 12:1) and isolated as a colorless oil that solidifies on standing. $[\alpha]_D^{25} = -51.18$ ($c = 0.18$, CH₂Cl₂, 85% ee). ¹H NMR (300 MHz, CDCl₃): $\delta = 1.64$ (td, $J = 3.9, 13.5$ Hz, 1H), 1.81-2.25 (m, 4H), 2.49-2.83 (m, 7H), 4.20 (s, 1H), 5.28 (d, $J = 17.1$ Hz, 1H), 5.39 (d, $J = 10.2$ Hz, 1H), 5.87 (dddd, $J = 6.9, 7.5, 10.2, 17.1$ Hz, 1H), 6.61 (dd, $J = 2.1, 8.1$ Hz, 2H), 6.94 (d, $J = 7.8$ Hz, 1H), 7.07-7.20 (m, 3H), 7.27 (dt, $J = 1.2, 7.8$ Hz, 1H), 7.42 (tt, $J = 1.2, 7.5$, 1H), 7.49 (dt, $J = 1.5, 7.5$ Hz, 1H), 7.85 (d, $J = 8.1$ Hz, 1H). ¹³C NMR (125 MHz, CDCl₃): $\delta = 21.69, 23.78, 33.05, 39.94, 41.38, 42.63, 46.00, 54.13, 56.61, 115.41, 115.96, 123.10, 126.95, 127.96, 128.46, 128.77, 128.99, 129.06, 129.11, 130.21, 133.58, 134.59, 135.88, 212.96$. HRMS (ESI/TOF) calcd for C₂₆H₂₇N₂O⁺: 383.2118, found: 383.2102. IR (NaCl): ν [cm⁻¹] = 3087, 3062, 3030, 2947, 2875, 2244, 1697, 1602, 1497, 1454, 1418, 1320, 1267, 1206, 1194, 1128, 1081, 1031, 988, 935, 799, 768, 746, 704.

2-Allyl-2-((S)-((S)-1-benzyl-2-oxocyclohexyl)(phenyl)methyl)malononitrile

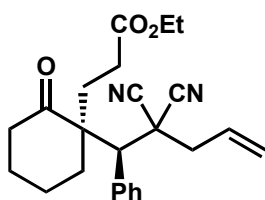


4c

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 12:1 to 4:1, MnO₄ stain, $R_f = 0.25$ in hexanes/EtOAc 12:1) and isolated as a colorless oil that solidifies on standing. $[\alpha]_D^{25} = +24.70$ ($c = 0.81$, CH₂Cl₂, 88% ee). ¹H NMR (300 MHz, CDCl₃): $\delta = 1.11$ -1.58 (m, 4H), 1.97 (ddd, $J = 4.8, 8.7, 14.4$ Hz, 1H), 2.07-2.34 (m, 3H), 2.46 (dd, $J = 7.8, 13.8$ Hz, 1H), 2.58 (dd, $J = 6.9, 13.8$ Hz, 1H), 3.09 (d, $J = 12.8$ Hz, 1H), 3.73 (s, 1H), 3.88 (d, $J = 12.8$ Hz, 1H), 5.24 (d, $J = 17.1$ Hz, 1H), 5.39 (d, $J = 10.2$ Hz, 1H), 5.87 (dddd, $J = 6.9, 7.8, 10.2, 17.1$ Hz, 1H), 7.03-7.09 (m, 3H),

7.16-7.25 (m, 3H), 7.32-7.42 (m, 2H), 7.48 (t, $J = 7.2$ Hz, 1H), 7.92 (d, $J = 7.8$ Hz, 1H). ^{13}C NMR (75 MHz, CDCl_3): $\delta = 21.25, 23.46, 31.96, 37.76, 40.97, 41.84, 43.71, 55.38, 57.38, 115.76, 115.79, 123.76, 126.68, 128.10, 128.21, 128.51, 128.83, 128.88, 128.92, 131.28, 133.51, 133.97, 136.44, 213.18$. HRMS (ESI/TOF) calcd for $\text{C}_{26}\text{H}_{26}\text{N}_2\text{ONa}^+$: 405.1937, found: 405.1947. IR (NaCl): ν [cm^{-1}] = 3086, 3062, 3029, 2945, 2872, 2245, 1702, 1602, 1495, 1455, 1265, 1125, 1086, 1032, 990, 935, 751, 736, 705.

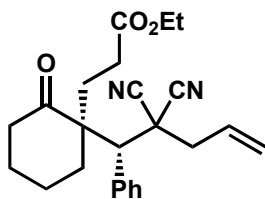
Ethyl 3-((*S*)-1-((*R*)-2,2-dicyano-1-phenylpent-4-enyl)-2-oxocyclohexyl)-propanoate



3d

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 4:1 to 2:1, MnO_4 stain, $R_f = 0.20$ in hexanes/EtOAc 10:1) and isolated as a white solid. mp = 104°C. $[\alpha]_D^{25} = -69.02$ (c = 0.15, CH_2Cl_2 , 82% ee). ^1H NMR (500 MHz, CDCl_3): $\delta = 1.13$ (t, $J = 7.0$ Hz, 3H), 1.32 (ddd, $J = 5.5, 10.0, 15.5$ Hz, 1H), 1.74 (ddd, $J = 6.0, 10.5, 16.0$ Hz, 1H), 1.85 (ddd, $J = 6.0, 10.5, 16.0$ Hz, 1H), 1.94-2.11 (m, 4H), 2.24 (ddd, $J = 5.5, 10.0, 15.0$ Hz, 1H), 2.45-2.52 (m, 2H), 2.55 (tdd, $J = 1.5, 6.5, 14.0$ Hz, 1H), 2.59-2.68 (m, 3H), 3.89-3.99 (m, 2H), 4.29 (s, 1H), 5.32 (ddd, $J = 1.5, 2.5, 17.0$ Hz, 1H), 5.41 (d, $J = 10.0$ Hz, 1H), 5.91 (dddd, $J = 6.5, 7.5, 10.0, 17.0$ Hz, 1H), 7.19 (d, $J = 1$ Hz), 7.35 (t, $J = 6.5$ Hz, 1H), 7.41 (td, $J = 1.0, 7.5$ Hz, 1H), 7.45 (t, $J = 7.5$ Hz, 1H), 7.76 (d, $J = 7.5$ Hz, 1H). ^{13}C NMR (125 MHz, CDCl_3): $\delta = 14.04, 20.96, 24.11, 27.83, 29.65, 33.08, 39.24, 39.84, 42.47, 50.88, 54.68, 60.50, 115.61, 115.84, 123.09, 128.76, 128.81, 129.19, 129.27, 129.32, 132.86, 133.44, 172.26, 211.28$. HRMS (ESI/TOF) calcd for $\text{C}_{24}\text{H}_{28}\text{N}_2\text{O}_3\text{Na}^+$: 415.1992, found: 415.1989. IR (NaCl): ν [cm^{-1}] = 3062, 2947, 2874, 2244, 1733, 1698, 1494, 1454, 1418, 1378, 1297, 1256, 1182, 1129, 1066, 1023, 990, 936, 802, 710.

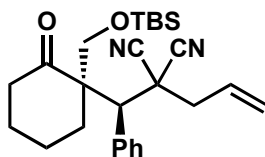
Ethyl 3-((S)-1-((S)-2,2-dicyano-1-phenylpent-4-enyl)-2-oxocyclohexyl)propanoate



4d

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 4:1 to 2:1, MnO₄ stain, $R_f = 0.15$ in hexanes/EtOAc 10:1) and isolated as a colorless oil that solidifies on standing. $[\alpha]_D^{25} = -33.99$ ($c = 0.69$, CH₂Cl₂, 89% ee). ¹H NMR (500 MHz, CDCl₃): $\delta = 1.20$ (t, $J = 7.0$ Hz, 3H), 1.69-1.88 (m, 4H), 1.95 (dddd, $J = 1.0, 3.5, 5.5, 16.0$ Hz, 1H), 2.05 (ddd, $J = 5.5, 9.5, 15.0$ Hz, 1H), 2.15 (ddd, $J = 6.0, 9.5, 15.0$ Hz, 1H), 2.18-2.23 (m, 1H), 2.36-2.61 (m, 6H), 3.48 (s, 1H), 4.06 (dq, $J = 1.0, 7.0$ Hz, 2H), 5.27 (ddd, $J = 1.0, 2.5, 17.0$ Hz, 1H), 5.40 (d, $J = 10.0$ Hz, 1H), 5.86 (dddd, $J = 6.5, 8.0, 10.0, 17.0$ Hz, 1H), 7.14 (br, 1H), 7.31-7.48 (br, m, 3H), 7.90 (br, 1H). ¹³C NMR (125 MHz, CDCl₃): $\delta = 14.13, 20.84, 24.96, 28.13, 29.11, 34.04, 38.37, 39.52, 43.82, 54.16, 55.65, 60.62, 115.65, 115.71, 123.74, 128.49, 128.90, 133.63, 172.58, 212.27$. HRMS (ESI/TOF) calcd for C₂₄H₂₈N₂O₃Na⁺: 415.1992, found: 415.1992. IR (NaCl): ν [cm⁻¹] = 3063, 2944, 2872, 2245, 1732, 1702, 1493, 1455, 1421, 1378, 1302, 1259, 1182, 1126, 1095, 1065, 1025, 992, 936, 857, 710.

2-Allyl-2-((R)-((R)-1-((tert-butyldimethylsilyloxy)methyl)-2-oxocyclohexyl)(phenyl)methyl)malononitrile

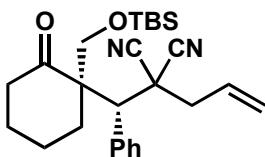


3e

Synthesised according to the general procedure using Pd(pmdba)₂ as precatalyst. Purified by flash chromatography (hexanes/EtOAc 15:1, MnO₄ stain, $R_f = 0.5$) and isolated as a colorless oil. $[\alpha]_D^{25} = -68.11^\circ$ ($c = 0.63$, CH₂Cl₂, 69% ee). ¹H NMR (500 MHz, CDCl₃): $\delta = -0.32$ (s, 3H), -0.22 (s, 3H), 0.74 (s, 9H), 1.80-1.90 (m, 1H), 1.97-2.08 (m, 3H), 2.38-2.48 (m, 2H), 2.52-2.58 (m, 2H), 2.60-2.65 (m, 1H), 2.66 (dd, $J = 8.0, 14.0$ Hz, 1H), 3.11

(d, $J = 10.0$ Hz, 1H), 3.67 (d, $J = 10.0$ Hz, 1H), 4.44 (s, 1H), 5.30 (ddd, 1.0, 2.5, 17.0 Hz, 1H), 5.39 (d, $J = 10.5$ Hz, 1H), 5.91 (dddd, $J = 6.5, 8.0, 10.5, 17.0$ Hz, 1H), 7.18 (d, $J = 7.5$ Hz, 1H), 7.30 (t, $J = 7.5$ Hz, 1H), 7.36 (tt, $J = 1.5, 7.0$ Hz, 1H), 7.40 (t, $J = 7.0$ Hz, 1H), 7.72 (d, $J = 7.5$ Hz, 1H). ^{13}C NMR (125 MHz, CDCl_3): $\delta = -6.20, -6.09, 18.01, 21.82, 24.28, 25.64, 29.98, 39.50, 39.55, 42.51, 50.33, 56.99, 66.47, 115.65, 116.21, 122.81, 128.37, 128.43, 128.76, 128.97, 129.01, 133.37, 134.42, 208.96$. HRMS (ESI/TOF) calcd for $\text{C}_{26}\text{H}_{37}\text{N}_2\text{O}_2\text{Si}^+$: 437.2619, found: 437.2623. IR (NaCl): ν [cm^{-1}] = 3089, 3063, 3033, 2953, 2931, 2882, 2857, 2244, 1713, 1644, 1499, 1472, 1455, 1420, 1390, 1362, 1318, 1258, 1223, 1117, 1096, 989, 937, 902, 840, 779, 714.

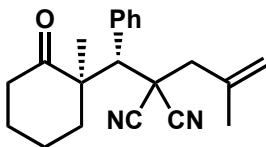
2-Allyl-2-((S)-((R)-1-((tert-butyldimethylsilyloxy)methyl)-2-oxocyclohexyl)(phenyl)methyl)malononitrile



4e

Synthesised according to the general procedure using $\text{Pd}(\text{pmdba})_2$ as precatalyst. Purified by flash chromatography (hexanes/EtOAc 15:1, MnO_4 stain, $R_f = 0.4$) and isolated as a colorless oil. $[\alpha]_D^{25} = -25.49^\circ$ ($c = 0.13, \text{CH}_2\text{Cl}_2, 70\%$ ee). ^1H NMR (500 MHz, CDCl_3): $\delta = 0.01$ (s, 3H), 0.05 (s, 3H), 0.90 (s, 9H), 1.61-1.75 (m, 3H), 1.88-1.94 (m, 2H), 2.08 (dtd, $J = 2.0, 4.0, 14.0$ Hz, 1H), 2.35 (dtd, $J = 1.5, 4.0, 15.0$ Hz, 1H), 2.45-2.51 (m, 2H), 2.59 (tdd, $J = 1.0, 6.5, 14.0$ Hz, 1H), 3.67 (s, 1H), 4.00 (d, $J = 10.5$ Hz, 1H), 4.18 (d, $J = 10.5$ Hz, 1H), 5.25 (ddd, $J = 1.0, 2.5, 17.0$ Hz, 1H), 5.38 (d, $J = 10.0$ Hz, 1H), 5.90 (dddd, $J = 7.0, 8.0, 10.0, 17.0$ Hz, 1H), 7.04 (br, 1H), 7.23-7.44 (m, 3H), 7.91 (br, 1H). ^{13}C NMR (125 MHz, CDCl_3): $\delta = -5.87, -5.77, 18.35, 21.29, 25.52, 25.89, 33.23, 38.93, 40.06, 40.83, 43.87, 53.93, 58.28, 64.33, 116.13, 123.12, 123.35, 128.43, 128.45, 128.52, 128.99, 129.73, 133.68, 134.31, 212.08$. HRMS (ESI/TOF) calcd for $\text{C}_{26}\text{H}_{37}\text{N}_2\text{O}_2\text{Si}^+$: 437.2619, found: 437.2621. IR (NaCl): ν [cm^{-1}] = 3062, 3030, 2951, 2930, 2883, 2857, 2245, 1704, 1654, 1622, 1602, 1495, 1471, 1450, 1340, 1257, 1188, 1099, 990, 937, 839, 778, 700.

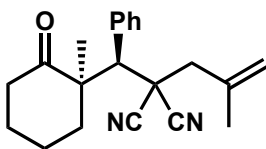
2-((R)-((R)-1-Methyl-2-oxocyclohexyl)(phenyl)methyl)-2-(2-methylallyl)malononitrile



3f

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 12:1 to 6:1, MnO₄ stain, $R_f = 0.5$ in hexanes/EtOAc 6:1) and isolated as a white solid. Contains not separable benzylidenemalononitrile as impurity. mp = 67-68°C. $[\alpha]_D^{25} = -29.40^\circ$ (c = 0.22, CH₂Cl₂, 75% ee). ¹H NMR (500 MHz, CDCl₃): δ = 0.78 (s, 3H), 1.76 (m, 1H), 1.89 (s, 3H), 1.98-2.09 (m, 3H), 2.38 (ddd, $J = 3.5, 5.0, 13.5$ Hz, 1H), 2.47 (d, $J = 14.0$ Hz, 1H), 2.53 (ddd, $J = 7.5, 11.0, 17.0$ Hz, 1H), 2.61 (d, $J = 14.0$ Hz, 1H), 2.59-2.65 (m, 2H), 4.32 (s, 1H), 4.96 (s, 1H), 5.11 (quin, $J = 1.5$ Hz, 1H), 7.17 (d, $J = 7.0$ Hz, 1H), 7.34 (t, $J = 7.5$ Hz, 1H), 7.39 (tt, $J = 1.0, 7.5$ Hz, 1H), 7.43 (t, $J = 7.5$ Hz, 1H), 7.81 (d, $J = 7.5$ Hz, 1H). ¹³C NMR (125 MHz, CDCl₃): δ = 20.80, 23.07, 24.01, 25.52, 32.75, 38.10, 38.68, 46.17, 52.14, 54.24, 112.50, 113.66, 118.99, 128.57, 128.65, 128.86, 128.94, 130.91, 133.96, 137.27, 212.67. HRMS (ESI/TOF) calcd for C₂₁H₂₅N₂O⁺: 321.1961 found: 322.1957. IR (NaCl): ν [cm⁻¹] = 3033, 2947, 2872, 2230, 1699, 1594, 1570, 1494, 1450, 1379, 1319, 1300, 1219, 1188, 1129, 1092, 1000, 955, 913, 805, 761, 710, 686, 616.

2-((S)-((R)-1-Methyl-2-oxocyclohexyl)(phenyl)methyl)-2-(2-methylallyl)malononitrile

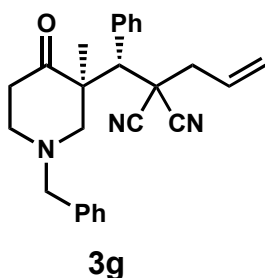


4f

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 12:1 to 6:1, MnO₄ stain, $R_f = 0.35$ in hexanes/EtOAc 6:1) and isolated as a white solid. mp = 144-146°C. $[\alpha]_D^{25} = +67.50^\circ$ (c = 0.21, CH₂Cl₂, 81% ee). ¹H NMR (500 MHz, CDCl₃): δ = 1.29 (ddd, $J = 4.0, 11.5, 14.0$ Hz, 1H), 1.44 (dtd, $J = 2.0, 4.0,$

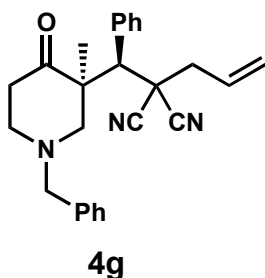
13.5 Hz, 1H), 1.58 (ddd, $J = 4.0, 8.5, 13.0$ Hz, 1H), 1.63-1.81 (m, 2H), 1.84 (s, 3H), 1.91 (s, 3H), 1.97-2.04 (m, 1H), 2.46 (d, $J = 14.0$ Hz, 1H), 2.47 (dtd, $J = 1.0, 4.5, 13.0$ Hz, 1H), 2.58 (d, $J = 14.0$ Hz, 1H), 2.80 (ddd, $J = 6.0, 11.5, 13.0$ Hz, 1H), 3.99 (s, 1H), 5.00 (s, 1H), 5.12 (quin, 1.5 Hz, 1H), 7.12 (br, 1H), 7.30-7.47 (br, 2H), 7.38 (t, $J = 7.0$ Hz, 1H), 7.84 (br, 1H). ^{13}C NMR (125 MHz, CDCl_3): $\delta = 19.15, 20.86, 23.19, 28.85, 38.26, 38.75, 41.17, 46.45, 52.77, 54.42, 116.24, 116.54, 119.05, 128.72, 128.74, 128.85, 134.08, 137.37, 214.35$. HRMS (ESI/TOF) calcd for $\text{C}_{21}\text{H}_{25}\text{N}_2\text{O}^+$: 321.1961 found: 321.1958. IR (NaCl): ν [cm^{-1}] = 3062, 2946, 2868, 2245, 1704, 1646, 1494, 1454, 1384, 1307, 1260, 1229, 1092, 909, 781, 765, 738, 712.

(rac, syn)-2-Allyl-2-(1-benzyl-3-methyl-4-oxopiperidin-3-yl)(phenyl)methyl-malononitrile



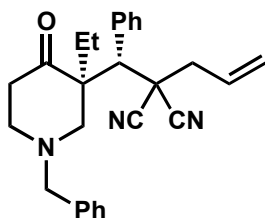
Synthesised according to the general procedure for racemic compounds. Purified by flash chromatography (hexanes/EtOAc 10:1 to 4:1, MnO_4 stain, $R_f = 0.3$ in hexanes/EtOAc 10:1) and isolated as a colorless oil that runs very close to **3f** on TLC. ^1H NMR (500 MHz, CDCl_3): $\delta = 1.10$ (s, 3H), 2.46-2.54 (m, 3H), 2.60 (ddd, $J = 8.0, 8.5, 16.0$ Hz, 1H), 2.86 (dd, $J = 4.5, 8.5$ Hz, 2H), 3.01 (d, $J = 12.0$ Hz, 1H), 3.47 (d, $J = 12.0$ Hz, 1H), 3.71 (d, $J = 13.0$ Hz, 1H), 3.76 (d, $J = 13.0$ Hz, 1H), 4.30 (s, 1H), 5.30 (ddd, $J = 1.0, 2.5, 17.0$ Hz, 1H), 5.42 (d, $J = 10.0$ Hz, 1H), 5.88 (dddd, $J = 7.0, 7.5, 10.0, 17.0$ Hz, 1H), 7.17 (d, $J = 7.0$ Hz, 1H), 7.29 (tt, $J = 1.5, 7.0$ Hz, 1H), 7.32-7.40 (m, 7H), 7.55 (br, 1H). ^{13}C NMR (125 MHz, CDCl_3): $\delta = 24.00, 38.54, 39.10, 42.77, 51.69, 52.00, 53.09, 60.21, 62.36, 115.70, 115.72, 123.25, 127.41, 128.44, 128.58, 128.65, 128.68, 128.93, 129.05, 129.23, 133.32, 133.57, 138.05, 210.09$. HRMS (ESI/TOF) calcd for $\text{C}_{26}\text{H}_{28}\text{N}_3\text{O}^+$: 398.2227, found: 398.2219. IR (NaCl): ν [cm^{-1}] = 3063, 3030, 2932, 2822, 2245, 1710, 1643, 1602, 1495, 1455, 1403, 1349, 1311, 1249, 1197, 1143, 1084, 1029, 991, 936, 739, 704.

2-Allyl-2-((S)-((S)-1-benzyl-3-methyl-4-oxopiperidin-3-yl)(phenyl)methyl)malononitrile



Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 10:1 to 4:1, MnO₄ stain, $R_f = 0.25$ in hexanes/EtOAc 10:1) and isolated as a colorless oil. Runs very close to **3f** on TLC. $[\alpha]_D^{25} = +161.51^\circ$ ($c = 0.13$, CH₂Cl₂, 89% ee). ¹H NMR (500 MHz, CDCl₃): $\delta = 1.55$ (s, 3H), 1.99 (d, $J = 12.5$ Hz, 1H), 2.34 (dd, $J = 2.0, 12.0$ Hz, 1H), 2.42 (dt, $J = 6.0, 10.5$ Hz, 1H), 2.44 (d, $J = 7.5$ Hz, 1H), 2.63-2.67 (m, 1H), 2.91-3.01 (m, 2H), 3.21 (d, $J = 13.0$ Hz, 1H), 3.45 (d, $J = 13.0$ Hz, 1H), 4.17 (s, 1H), 5.24 (ddd, $J = 1.0, 2.5, 17.0$ Hz, 1H), 5.39 (d, $J = 10.0$ Hz, 1H), 5.87 (tdd, $J = 7.5, 10.0, 17.0$ Hz, 1H), 7.23-7.39 (m, 8H), 7.42 (t, $J = 8.0$ Hz, 1H), 7.78 (d, $J = 7.5$ Hz, 1H). ¹³C NMR (125 MHz, CDCl₃): $\delta = 16.86, 38.47, 39.58, 43.42, 52.28, 53.75, 53.93, 61.59, 65.60, 114.95, 115.84, 123.52, 127.52, 127.88, 128.39, 128.47, 128.62, 128.99, 129.06, 129.11, 133.06, 133.41, 137.34, 211.53$. HRMS (ESI/TOF) calcd for C₂₆H₂₈N₃O⁺: 398.2227, found: 398.2226. IR (NaCl): ν [cm⁻¹] = 3086, 3062, 3030, 2981, 2950, 2808, 2246, 1711, 1643, 1602, 1587, 1495, 1456, 1384, 1348, 1247, 1194, 1118, 1070, 1029, 990, 936, 773, 737, 701, 620.

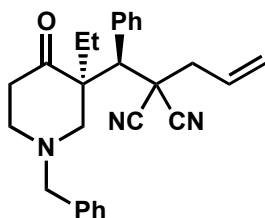
2-Allyl-2-((R)-((S)-1-benzyl-3-ethyl-4-oxopiperidin-3-yl)(phenyl)methyl)malononitrile



3h

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 10:1, MnO₄ stain, $R_f = 0.3$) and isolated as a colorless oil. $[\alpha]_D^{25} = -36.90^\circ$ ($c = 0.18$, CH₂Cl₂, 71% ee). ¹H NMR (500 MHz, CDCl₃): $\delta = 0.45$ (t, $J = 7.5$ Hz, 3H), 1.15 (qd, $J = 7.5, 15.0$ Hz, 1H), 2.25 (qd, $J = 7.5, 15.0$ Hz, 1H), 2.50-2.64 (m, 4H), 2.79 (dt, $J = 5.0, 11.0$ Hz, 1H), 3.00-3.04 (m, 1H), 3.36 (d, $J = 11.5$ Hz, 1H), 3.40 (dd, $J = 2.0, 11.5$ Hz, 1H), 3.77 (s, 2H), 4.27 (s, 1H), 5.31 (d, $J = 17.0$ Hz, 1H), 5.41 (d, $J = 10.0$ Hz, 1H), 5.90 (dddd, $J = 6.5, 7.5, 10.0, 17.0$ Hz, 1H), 7.19 (d, $J = 7.0$ Hz, 1H), 7.29 (t, $J = 7.5$ Hz, 1H), 7.32-7.40 (m, 7H), 7.52 (br, 1H). ¹³C NMR (125 MHz, CDCl₃): $\delta = 7.78, 28.91, 39.26, 39.66, 42.71, 50.71, 50.76, 56.25, 58.90, 62.56, 115.84, 123.08, 127.34, 128.41, 128.57, 128.85, 128.90, 128.96, 128.99, 129.01, 133.07, 133.58, 138.09, 209.10$. HRMS (ESI/TOF) calcd for C₂₇H₂₉CIN₃O⁻ [M+Cl]⁻: 446.2005, found: 446.2011. IR (NaCl): ν [cm⁻¹] = 3086, 3062, 3030, 2971, 2940, 2882, 2827, 2780, 2244, 1706, 1496, 1454, 1352, 1266, 1239, 1200, 1137, 1092, 1028, 990, 936, 809, 739, 709.

2-Allyl-2-((S)-((S)-1-benzyl-3-ethyl-4-oxopiperidin-3-yl)(phenyl)methyl)malononitrile

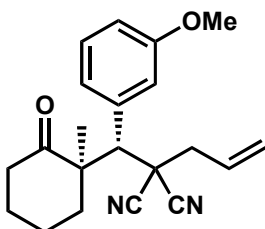


4h

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 10:1, MnO₄ stain, $R_f = 0.25$) and isolated as a colorless oil. $[\alpha]_D^{25} =$

+51.73° (c = 0.598, CH₂Cl₂, 97% ee). ¹H NMR (500 MHz, CDCl₃): δ = 0.81 (t, *J* = 7.5 Hz, 3H), 1.59 (td, *J* = 7.5, 15.0 Hz, 1H), 2.35 (dd, *J* = 7.5, 14.0 Hz, 1H), 2.40-2.46 (m, 2H), 2.51-2.70 (m, 4H), 2.85-2.96 (m, 2H), 3.39 (d, *J* = 13.0 Hz, 1H), 3.53 (d, *J* = 13.0 Hz, 1H), 3.91 (s, 1H), 5.20 (ddd, *J* = 1.0, 2.5, 17.0 Hz, 1H), 5.37 (d, *J* = 10.0 Hz, 1H), 5.83 (dddd, *J* = 7.0, 7.5, 10.0, 17.0 Hz, 1H), 7.02 (br, 1H), 7.21 (br, 1H), 7.32-7.40 (m, 7H), 7.80 (br, 1H). ¹³C NMR (125 MHz, CDCl₃): δ = 9.07, 23.93, 37.96, 40.23, 44.00, 52.34, 53.22, 56.80, 59.20, 62.22, 115.32, 115.82, 123.49, 127.53, 128.39, 128.50, 128.61, 128.70, 128.82, 129.24, 133.29, 133.61, 137.66, 210.43. HRMS (ESI/TOF) calcd for C₂₇H₂₉ClN₃O⁻ [M+Cl]⁻: 446.2005, found: 446.2026. IR (NaCl): ν [cm⁻¹] = 3086, 3062, 3030, 2965, 2886, 2824, 2245, 1707, 1495, 1455, 1350, 1243, 1191, 1137, 1109, 1078, 1029, 990, 935, 738, 707.

2-Allyl-2-((*R*)-(3-methoxyphenyl)((*R*)-1-methyl-2-oxocyclohexyl)methyl)malononitrile

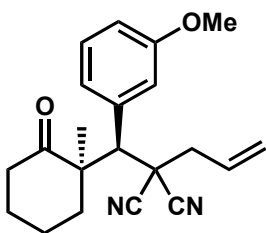


3i

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 6:1, MnO₄ stain, *R_f* = 0.40) and isolated as a colorless oil. The ¹H and ¹³C NMR spectra show a 1:1 mixture of two rotamers. [α]_D²⁵ = -134.63° (c = 0.68, CH₂Cl₂, 71% ee). ¹H NMR (500 MHz, CDCl₃): δ = 0.82 (s, 1.5H), 0.83 (s, 1.5H), 1.80-1.86 (m, 1H), 1.99-2.04 (m, 3H), 2.37 (d, *J* = 13.0 Hz, 1H), 2.49-2.64 (m, 5H), 3.81 (s, 1.5H), 3.23 (s, 1.5H), 4.25 (s, 0.5H), 4.32 (s, 0.5H), 5.31 (dd, *J* = 4.0, 17.0 Hz, 1H), 5.41 (d, *J* = 10.0 Hz, 1H), 5.91 (tdd, *J* = 7.5, 10.0, 17.0 Hz, 1H), 6.71 (s, 0.5H), 6.75 (d, *J* = 7.5 Hz, 0.5H), 6.90-6.93 (m, 1H), 7.24 (d, *J* = 8.0 Hz, 0.5H), 7.31 (s, 0.5H), 7.34 (d, *J* = 5.0 Hz, 1H). ¹³C NMR (125 MHz, CDCl₃): δ = 20.82, 24.08, 25.24, 32.82, 32.89, 38.09, 39.40, 42.52, 51.98, 52.02, 52.78, 53.17, 55.21, 55.27, 113.53, 114.17, 114.66, 115.65, 116.05, 116.24, 119.70, 120.75, 123.02, 125.86, 128.83, 128.87, 129.54, 130.11, 135.16,

135.32, 159.50, 159.85, 212.40, 212.52. HRMS (ESI/TOF) calcd for $C_{21}H_{25}N_2O_2^+$ $[M+H]^+$: 337.1911, found: 337.1918. IR (NaCl): ν [cm^{-1}] = 3084, 2945, 2872, 2838, 2229, 1702, 1600, 1583, 1494, 1456, 1436, 1322, 1296, 1264, 1227, 1166, 1128, 1049, 995, 936, 864, 729, 762, 729, 705, 684.

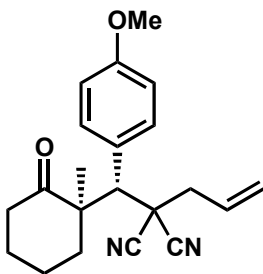
2-Allyl-2-((S)-(3-methoxyphenyl)((R)-1-methyl-2-oxocyclohexyl)methyl)malononitrile



4i

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 6:1, MnO_4 stain, $R_f = 0.35$) and isolated as a colorless oil. The 1H and ^{13}C NMR spectra show a mixture of two rotamers. $[\alpha]_D^{25} = +51.97^\circ$ ($c = 0.55$, CH_2Cl_2 , 86% ee). 1H NMR (500 MHz, $CDCl_3$): $\delta = 1.35$ -1.40 (br, 1H), 1.47 (dddd, $J = 2.0, 4.0, 6.5, 14.0$ Hz, 1H), 1.58-1.65 (m, 1H), 1.69-1.76 (m, 1H), 1.77 (s, 3H), 1.96-2.01 (m, 1H), 2.47 (td, $J = 4.5, 13.0$ Hz, 1H), 2.54 (dd, $J = 7.0, 14.0$ Hz, 1H), 2.58 (dd, $J = 8.0, 14.0$ Hz, 1H), 2.76 (ddd, 6.0, 11.0, 13.0 Hz, 1H), 3.81 (s, 3H), 3.90 (s, 1H), 5.30 (ddd, $J = 1.0, 2.5, 17.0$ Hz, 1H), 5.40 (d, $J = 10.0$ Hz, 1H), 5.93 (dddd, $J = 6.5, 7.5, 10.0, 17.0$ Hz, 1H), 6.68 (br, 1H), 6.91 (d, 6.5 Hz, 1H), 7.23 (br, 1H), 7.36 (br, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): $\delta = 19.27, 20.86, 28.65, 38.31, 39.28, 40.82, 42.92, 52.55, 53.09, 55.22, 113.25, 114.22, 114.70, 115.63, 116.22, 119.73, 120.79, 123.09, 125.61, 128.85, 129.44, 129.96, 135.23, 159.41, 159.82, 214.08$. HRMS (ESI/TOF) calcd for $C_{21}H_{25}N_2O_2^+$ $[M+H]^+$: 337.1911, found: 337.1918. IR (NaCl): ν [cm^{-1}] = 3085, 2943, 2868, 2838, 2249, 1704, 1600, 1583, 1494, 1454, 1437, 1384, 1323, 1290, 1268, 1225, 1170, 1050, 988, 936, 882, 792, 742, 704.

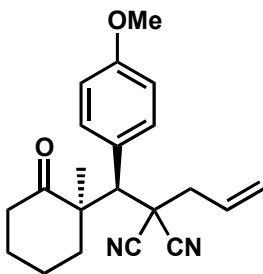
2-Allyl-2-((R)-(4-methoxyphenyl)((R)-1-methyl-2-oxocyclohexyl)methyl)malononitrile



3j

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 8:1 to 6:1 to 4:1, MnO₄ stain, $R_f = 0.40$ in hexanes/EtOAc 6:1) and isolated as a colorless oil that solidifies on standing. $[\alpha]_D^{25} = -47.87^\circ$ ($c = 0.66$, CH₂Cl₂, 73% ee). ¹H NMR (500 MHz, CDCl₃): $\delta = 0.80$ (s, 3H), 1.77-1.86 (m, 1H), 1.99-2.05 (m, 3H), 2.34 (d, $J = 13.0$ Hz, 1H), 2.48-2.63 (m, 5H), 3.82 (s, 3H), 4.26 (s, 1H), 5.30 (ddd, $J = 1.0, 2.5, 17.0$ Hz, 1H), 5.40 (d, $J = 10.0$ Hz, 1H), 5.90 (dddd, $J = 7.0, 7.5, 10.0, 17.0$ Hz, 1H), 6.84 (dd, $J = 2.5, 8.5$ Hz, 1H), 6.95 (dd, $J = 3.0, 8.5$ Hz, 1H), 7.08 (dd, $J = 1.5, 8.5$ Hz, 1H), 7.68 (dd, $J = 1.5, 8.5$ Hz, 1H). ¹³C NMR (125 MHz, CDCl₃): $\delta = 20.82, 24.10, 25.40, 32.85, 38.12, 39.62, 42.48, 52.20, 52.39, 55.22, 113.85, 114.46, 115.76, 116.09, 112.91, 125.66, 128.09, 129.54, 134.88, 159.83, 212.66$. HRMS (ESI/TOF) calcd for C₂₁H₂₄ClN₂O₂⁻ [M+Cl]⁻: 371.1532, found: 371.1493. IR (NaCl): ν [cm⁻¹] = 3084, 2944, 2872, 2840, 2244, 1701, 1610, 1580, 1515, 1461, 1417, 1295, 1256, 1185, 1130, 1084, 1033, 993, 936, 838, 813, 768, 742.

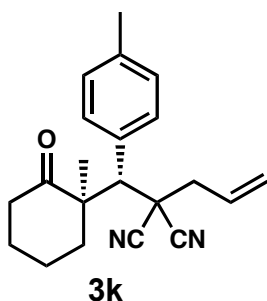
2-Allyl-2-((S)-(4-methoxyphenyl)((R)-1-methyl-2-oxocyclohexyl)methyl)malononitrile



4j

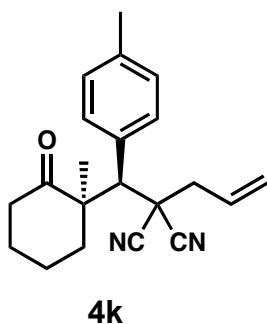
Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 8:1 to 6:1 to 4:1, MnO₄ stain, $R_f = 0.30$ in hexanes/EtOAc 6:1) and isolated as a colorless oil that solidifies on standing. $[\alpha]_D^{25} = +42.45^\circ$ ($c = 0.73$, CH₂Cl₂, 88% ee). ¹H NMR (500 MHz, CDCl₃): $\delta = 1.35$ (ddd, $J = 5.0, 11.0, 14.0$ Hz, 1H), 1.46 (ttd, $J = 1.5, 3.0, 14.0$ Hz, 1H), 1.57- 1.64 (m, 1H), 1.66-1.76 (m, 2H), 1.75 (s, 3H), 1.94- 2.00 (m, 1H), 2.47 (td, $J = 4.0, 13.0$ Hz, 1H), 2.54 (tdd, $J = 1.5, 6.5, 14.0$ Hz, 1H), 2.57 (tdd, $J = 1.0, 7.5, 14.0$ Hz, 1H), 2.74 (ddd, $J = 6.0, 11.5, 13.0$ Hz, 1H), 3.81 (s, 3H), 3.87 (s, 1H), 5.29 (ddd, $J = 1.0, 2.5, 17.0$ Hz, 1H), 5.39 (d, $J = 10.0$ Hz, 1H), 5.92 (dddd, $J = 7.0, 7.5, 10.0, 17.0$ Hz, 1H), 6.84 (br, 1H), 6.94 (br, 1H), 7.02 (br, 1H), 7.71 (br, 1H). ¹³C NMR (125 MHz, CDCl₃): $\delta = 19.34, 20.84, 28.56, 38.33, 39.51, 40.87, 42.88, 52.61, 52.74, 55.20, 113.76, 114.38, 115.74, 116.19, 123.01, 125.64, 128.88, 129.61, 134.60, 159.75, 214.27$. HRMS (ESI/TOF) calcd for C₂₁H₂₅N₂O₂⁺ [M+H]⁺: 337.1911, found: 337.1912. IR (NaCl): ν [cm⁻¹] = 3084, 2942, 2868, 2840, 2244, 1704, 1610, 1581, 1515, 1464, 1453, 1443, 1385, 1308, 1291, 1256, 1185, 1115, 1032, 989, 935, 837, 760, 740.

2-Allyl-2-((R)-((R)-1-methyl-2-oxocyclohexyl)(p-tolyl)methyl)malononitrile



Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 8:1 to 6:1 to 4:1, MnO₄ stain, $R_f = 0.45$ in hexanes/EtOAc 6:1) and isolated as a colorless oil that solidifies on standing. $[\alpha]_D^{25} = -43.98^\circ$ ($c = 0.84$, CH₂Cl₂, 75% ee). ¹H nmr (500 MHz, CDCl₃): $\delta = 0.80$ (s, 3H), 1.77-1.86 (m, 1H), 1.98-2.05 (m, 3H), 2.36 (s, 3H), 2.36 (d, $J = 12.0$ Hz, 1H), 2.50-2.63 (m, 5H), 4.28 (s, 1H), 5.30 (ddd, $J = 1.0, 2.5, 17.0$ Hz, 1H), 5.40 (ddd, $J = 1.0, 2.0, 10.0$ Hz, 1H), 5.90 (dddd, $J = 6.5, 8.0, 10.0, 17.0$ Hz, 1H), 7.04 (d, $J = 7.0$ Hz, 1H), 7.14 (d, $J = 7.0$ Hz, 1H), 7.22 (d, $J = 8.0$ Hz, 1H), 7.64 (d, $J = 8.0$ Hz, 1H). ¹³C nmr (125 MHz, CDCl₃): $\delta = 20.82, 21.04, 24.10, 25.38, 32.91, 38.12, 39.55, 42.49, 52.06, 52.73, 115.76, 116.08, 122.93, 128.31, 128.89, 129.33, 129.73, 130.72, 133.61, 138.78, 212.62$. HRMS (ESI/TOF) calcd for C₂₁H₂₅N₂O⁺: 321.1961, found: 321.1961. IR (NaCl): ν [cm⁻¹] = 3084, 2945, 2871, 1701, 1643, 1613, 1589, 1515, 1452, 1418, 1377, 1321, 1288, 1269, 1229, 1191, 1130, 1083, 991, 959, 935, 898, 828, 799, 765, 738.

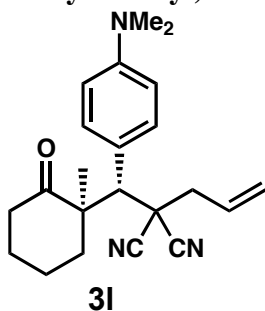
2-Allyl-2-((S)-((R)-1-methyl-2-oxocyclohexyl)(p-tolyl)methyl)malononitrile



Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 8:1 to 6:1 to 4:1, MnO₄ stain, $R_f = 0.45$ in hexanes/EtOAc 6:1) and isolated as a colorless oil that solidifies on standing. $[\alpha]_D^{25} = +44.12^\circ$ ($c = 0.695$, CH₂Cl₂,

87% ee). ¹H nmr (500 MHz, CDCl₃): δ = 1.35 (ddd, *J* = 4.0, 11.0, 14.0 Hz, 1H), 1.45 (d, *J* = 12.0 Hz, 1H), 1.56-1.63 (m, 1H), 1.68-1.75 (m, 2H), 1.76 (s, 3H), 1.94-2.01 (m, 1H), 2.35 (s, 3H), 2.47 (ddd, *J* = 4.0, 5.0, 12.0 Hz, 1H), 2.53 (tdd, *J* = 1.0, 7.0, 14.0, 1H), 2.57 (dd, *J* = 7.5, 14.0 Hz, 1H), 2.74 (ddd, *J* = 6.0, 11.5, 13.0 Hz, 1H), 3.89 (s, 1H), 5.29 (ddd, *J* = 1.5, 2.5, 17.0 Hz, 1H), 5.39 (d, *J* = 10.0 Hz, 1H), 5.92 (dddd, *J* = 7.0, 7.5, 10.0, 17.0 Hz, 1H), 7.00 (br, 1H), 7.14 (br, 1H), 7.68 (br, 1H). ¹³C nmr (125 MHz, CDCl₃): δ = 19.33, 20.83, 21.02, 28.60, 38.34, 39.42, 40.89, 42.89, 52.63, 52.91, 115.72, 116.18, 123.02, 128.37, 128.87, 129.32, 129.62, 130.71, 133.30, 138.76, 214.23. HRMS (ESI/TOF) calcd for C₂₁H₂₅N₂O⁺: 321.1961, found: 321.1963. IR (NaCl): ν [cm⁻¹] = 3084, 2944, 2867, 2245, 1705, 1643, 1613, 1516, 1452, 1419, 1384, 1307, 1266, 1227, 1126, 1113, 1084, 1065, 989, 935, 826, 798, 738, 703.

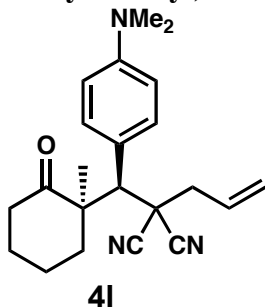
2-Allyl-2-((*R*)-(4-(dimethylamino)phenyl)((*R*)-1-methyl-2-oxocyclohexyl)methyl)malononitrile



Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 6:1, MnO₄ stain, *R_f* = 0.40 in hexanes/EtOAc 6:1) and isolated as a colorless oil that solidifies on standing. [α]_D²⁵ = -58.63° (c = 0.030, CH₂Cl₂, ee nd). ¹H nmr (500 MHz, CDCl₃): δ = 0.83 (s, 3H), 1.77-1.86 (m, 1H), 1.98-2.08 (m, 3H), 2.35 (d, *J* = 13.5 Hz, 1H), 2.47-2.63 (m, 5H), 2.97 (s, 6H), 4.18 (s, 1H), 5.31 (ddd, *J* = 1.0, 2.5, 17.0 Hz, 1H), 5.39 (d, *J* = 10.0 Hz, 1H), 5.91 (dddd, *J* = 6.5, 7.5, 10.0, 17.0 Hz, 1H), 6.62 (dd, *J* = 2.5, 8.5 Hz, 1H), 6.73 (dd, *J* = 2.0, 8.5 Hz, 1H), 6.99 (d, *J* = 8.5 Hz, 1H), 7.59 (d, *J* = 8.5 Hz, 1H). ¹³C nmr (125 MHz, CDCl₃): δ = 20.88, 24.17, 25.45, 32.97, 38.20, 39.76, 40.16, 42.50, 52.47, 52.56, 111.56, 112.49, 116.02, 116.31, 120.64, 122.72, 129.14, 129.16, 134.59, 150.37, 213.01. HRMS (ESI/TOF) calcd for C₂₂H₂₇N₃O⁺: 350.2227, found: 350.2232. IR (NaCl): ν [cm⁻¹] = 3084, 2944, 2870, 2808, 2243, 1700,

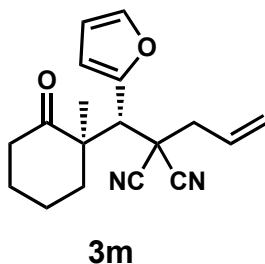
1611, 1525, 1449, 1358, 1270, 1230, 1200, 1168, 1127, 1083, 1069, 991, 948, 824, 756, 737.

2-Allyl-2-((S)-(4-(dimethylamino)phenyl)((R)-1-methyl-2-oxocyclohexyl)methyl)malononitrile



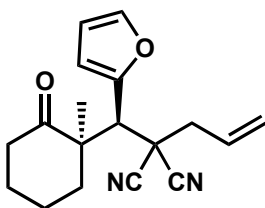
Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 6:1, MnO₄ stain, $R_f = 0.35$ in hexanes/EtOAc 6:1) and isolated as a pale yellow oil. $[\alpha]_D^{25} = +48.22^\circ$ ($c = 0.37$, CH₂Cl₂, 99% ee). ¹H nmr (500 MHz, CDCl₃): $\delta = 1.38$ (ddd, $J = 4.5, 11.0, 15.0$ Hz, 1H), 1.46 (dddd, $J = 1.5, 4.0, 5.5, 14.0$ Hz, 1H), 1.57-1.66 (m, 1H), 1.66-1.74 (m, 1H), 1.75 (s, 3H), 1.92-1.99 (m, 1H), 2.49 (ddd, $J = 4.0, 5.0, 13.0$ Hz, 1H), 2.53-2.61 (m, 2H), 2.73 (ddd, $J = 6.0, 11.0, 13.0$ Hz, 1H), 2.97 (s, 6H), 3.81 (s, 1H), 5.29 (ddd, $J = 1.5, 3.0, 17.0$ Hz, 1H), 5.38 (d, $J = 10.0$ Hz, 1H), 5.93 (dddd, $J = 7.0, 7.5, 10.0, 17.0$ Hz, 1H), 6.61 (br, 1H), 6.72 (br, 1H), 6.95 (br, 1H), 7.62 (br, 1H). ¹³C nmr (125 MHz, CDCl₃): $\delta = 19.27, 20.87, 28.74, 38.43, 39.64, 40.11, 41.03, 42.91, 52.66, 53.01, 111.41, 112.42, 115.09, 116.39, 120.48, 122.83, 129.05, 129.12, 134.25, 150.30, 214.64$. HRMS (ESI/TOF) calcd for C₂₂H₂₇N₃O⁺: 350.2227, found: 350.2231. IR (NaCl): ν [cm⁻¹] = 3083, 2981, 2943, 2867, 2808, 2244, 1704, 1612, 1525, 1447, 1359, 1230, 1199, 1166, 1128, 1064, 989, 947, 823, 736.

2-Allyl-2-((R)-furan-2-yl)((R)-1-methyl-2-oxocyclohexyl)methyl)malononitrile



Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 10:1 to 6:1 to 4:1, MnO₄ stain, $R_f = 0.45$ in hexanes/EtOAc 6:1) and isolated as a colorless oil. $[\alpha]_D^{25} = -111.77^\circ$ ($c = 0.48$, CH₂Cl₂, 65% ee). ¹H NMR (500 MHz, CDCl₃): $\delta = 0.88$ (s, 3H), 1.76-1.85 (m, 1H), 1.94-2.06 (m, 3H), 2.40-2.61 (m, 4H), 2.64 (dd, $J = 7.5, 14.0$ Hz, 1H), 2.68 (tdd, $J = 1.0, 7.0, 14.0$ Hz, 1H), 4.41 (s, 1H), 5.36 (ddd, $J = 1.0, 2.5, 17.0$ Hz, 1H), 5.43 (d, $J = 10.0$ Hz, 1H), 5.90 (dddd, $J = 6.5, 8.0, 10.0, 17.0$ Hz, 1H), 6.39 (dd, $J = 2.0, 3.0$ Hz, 1H), 6.46 (dd, $J = 0.5, 3.0$ Hz, 1H), 7.46 (d, $J = 2.0$ Hz, 1H). ¹³C NMR (125 MHz, CDCl₃): $\delta = 20.70, 24.15, 24.37, 32.62, 37.85, 38.33, 42.51, 47.53, 52.25, 110.54, 112.31, 115.21, 115.45, 123.23, 128.61, 143.34, 148.21, 212.00$. HRMS (ESI/TOF) calcd for C₁₈H₂₄N₃O₂⁺ [M+NH₄]⁺: 314.1863, found: 314.1860. IR (NaCl): ν [cm⁻¹] = 3122, 3086, 2945, 2872, 2246, 1702, 1500, 1459, 1418, 1378, 1320, 1284, 1237, 1211, 1149, 1130, 1072, 1018, 991, 932, 748.

2-Allyl-2-((S)-furan-2-yl((R)-1-methyl-2-oxocyclohexyl)methyl)malononitrile

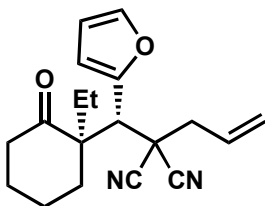


4m

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 10:1 to 6:1 to 4:1, MnO₄ stain, $R_f = 0.35$ in hexanes/EtOAc 6:1) and isolated as a colorless oil. $[\alpha]_D^{25} = +71.74^\circ$ ($c = 0.792$, CH₂Cl₂, 81% ee). ¹H NMR (500 MHz, CDCl₃): $\delta = 1.37$ (ddd, $J = 4.5, 11.0, 14.0$ Hz, 1H), 1.46 (dtd, $J = 2.5, 5.0, 14.0$ Hz, 1H), 1.60-1.81 (m, 3H), 1.79 (s, 3H), 1.98-2.05 (m, 1H), 2.43 (dtd, $J = 1.5, 5.0, 13.0$ Hz, 1H), 2.63 (d, $J = 7.0$ Hz, 2H), 2.74 (ddd, $J = 5.5, 11.0, 13.0$ Hz, 1H), 4.14 (s, 1H), 5.35 (ddd, $J = 1.0, 2.5, 17.0$ Hz, 1H), 5.43 (ddd, $J = 1.0, 2.0, 10.0$ Hz, 1H), 5.94 (tdd, $J = 7.0, 10.0, 17.0$ Hz, 1H), 6.39 (dd, $J = 1.5, 3.0$ Hz, 1H), 6.44 (dd, $J = 1.0, 3.0$ Hz, 1H), 7.46 (dd, $J = 0.5, 2.0$ Hz, 1H). ¹³C NMR (125 MHz, CDCl₃): $\delta = 18.34, 20.69, 29.08, 38.10, 38.14, 39.94, 42.69, 47.46, 53.11, 110.56, 112.02, 115.08, 115.56, 123.33, 128.60, 143.12, 148.11, 213.59$. HRMS (ESI/TOF) calcd for C₁₈H₂₀N₂O₂K⁺: 335.1156, found:

335.1158. IR (NaCl): ν [cm⁻¹] = 3123, 3086, 2945, 2868, 2247, 1706, 1500, 1450, 1390, 1307, 1238, 1209, 1149, 1016, 990, 932, 813, 752.

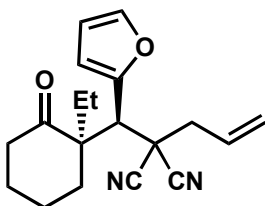
2-Allyl-2-((R)-((R)-1-ethyl-2-oxocyclohexyl)(furan-2-yl)methyl)malononitrile



3n

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 8:1, MnO₄ stain, R_f = 0.4) and isolated as a colorless oil. $[\alpha]_D^{25}$ = -385.46° (c = 0.53, CH₂Cl₂, 89% ee). ¹H NMR (500 MHz, CDCl₃): δ = 0.48 (t, J = 7.5 Hz, 3H), 1.04 (qd, J = 7.5, 15.0 Hz, 1H), 1.84-2.04 (m, 5H), 2.33-2.40 (m, 1H), 2.48 (dd, J = 3.5, 8.5 Hz, 2H), 2.56 (ddd, J = 3.5, 5.5, 16.0 Hz, 1H), 2.70 (d, J = 7.0 Hz, 2H), 4.41 (s, 1H), 5.36 (d, J = 17.0 Hz, 1H), 5.42 (d, J = 10.0 Hz, 1H), 5.92 (tdd, J = 7.5, 10.0, 17.0 Hz, 1H), 6.40 (dd, J = 2.0, 3.5 Hz, 1H), 6.44 (dd, J = 0.9, 3.3 Hz, 1H), 7.47 (d, J = 1.5 Hz, 1H). ¹³C NMR (125 MHz, CDCl₃): δ = 7.47, 20.74, 24.60, 28.16, 32.73, 38.68, 38.94, 42.62, 45.38, 55.66, 110.56, 112.43, 115.35, 115.59, 123.09, 128.80, 143.33, 148.15, 210.68. HRMS (ESI/TOF) calcd for C₁₉H₂₆N₃O₂⁺ [M+NH₄]⁺: 328.2020, found: 328.2020. IR (NaCl): ν [cm⁻¹] = 3123, 2945, 2877, 2246, 1701, 1500, 1458, 1418, 1390, 1318, 1229, 1208, 1150, 1128, 1075, 1017, 991, 931, 778, 756.

2-Allyl-2-((S)-((R)-1-ethyl-2-oxocyclohexyl)(furan-2-yl)methyl)malononitrile

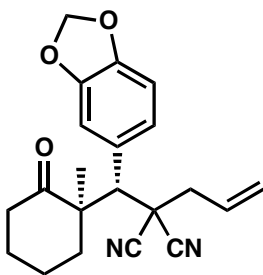


4n

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 8:1, MnO₄ stain, R_f = 0.3) and isolated as a colorless oil. $[\alpha]_D^{25}$ = -13.39° (c = 0.622, CH₂Cl₂, 96% ee). ¹H NMR (500 MHz, CDCl₃): δ = 0.91 (t, J = 7.5

Hz, 3H), 1.57-1.66 (m, 1H), 1.70-1.83 (m, 2H), 1.84-1.91 (m, 1H), 2.03 (qd, $J = 7.5, 15.0$ Hz, 1H), 2.11 (ddd, $J = 5.0, 10.0, 14.0$ Hz, 1H), 2.18 (dtd, $J = 2.0, 4.5, 14.0$ Hz, 1H), 2.30-2.51 (m, 5H), 4.08 (s, 1H), 5.33 (ddd, $J = 1.0, 2.5, 17.0$ Hz, 1H), 5.43 (d, $J = 10.0$ Hz, 1H), 5.90 (dddd, $J = 7.0, 7.5, 10.0, 17.0$ Hz, 1H), 6.37 (dd, $J = 2.0, 3.5$ Hz, 1H), 6.43 (dd, $J = 0.5, 3.5$ Hz, 1H), 7.43 (dd, $J = 1.0, 2.0$ Hz, 1H). ^{13}C NMR (125 MHz, CDCl_3): $\delta = 8.79, 20.75, 25.52, 26.47, 33.65, 36.97, 38.94, 43.18, 45.76, 56.54, 110.80, 111.79, 115.05, 115.45, 123.88, 128.25, 142.40, 149.11, 210.79$. HRMS (ESI/TOF) calcd for $\text{C}_{19}\text{H}_{26}\text{N}_3\text{O}_2^+ [\text{M}+\text{NH}_4]^+$: 328.2020, found: 328.2020. IR (NaCl): $\nu [\text{cm}^{-1}] = 3123, 2946, 2882, 2247, 1704, 1499, 1457, 1445, 1419, 1385, 1315, 1226, 1149, 1019, 992, 932, 814, 744$.

2-Allyl-2-((*R*)-benzo[*d*][1,3]dioxol-5-yl)((*R*)-1-methyl-2-oxocyclohexyl)methyl)malononitrile

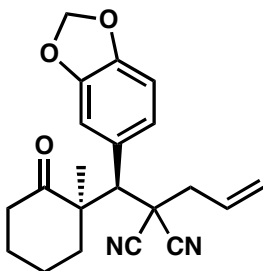


30

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 6:1, MnO_4 stain, $R_f = 0.35$) and isolated as a yellow powder. The ^1H nmr spectrum shows a mixture of two rotamers (ratio 1:1.38). $[\alpha]_{\text{D}}^{25} = -22.38^\circ$ ($c = 0.53$, CH_2Cl_2 , 56% ee). ^1H nmr (500 MHz, CDCl_3): $\delta = 0.84$ (s, 1.26H), 0.84 (s, 1.74 H), 1.77-1.87 (m, 1H), 1.97-2.05 (m, 3H), 2.32 (d, $J = 13.5$ Hz, 1H), 2.48-2.73 (m, 5H), 4.21 (s, 0.42H), 4.27 (s, 0.58H), 5.32 (d, $J = 17.0$ Hz, 1H), 5.41 (d, $J = 10.0$ Hz, 1H), 5.91 (tdd, $J = 7.5, 10.0, 17.0$ Hz, 1H), 5.99 (s, 0.84H), 6.00 (s, 1.16H), 6.65 (d, $J = 7.5$ Hz, 1H), 6.75 (d, $J = 7.5$ Hz, 0.58H), 6.86 (d, $J = 7.5$ Hz, 0.42H), 7.23 (br, 1H). ^{13}C nmr (125 MHz, CDCl_3): $\delta = 20.82, 20.84, 24.05, 25.27, 25.30, 32.73, 32.79, 38.10, 39.38, 39.77, 42.52, 42.54, 52.14, 52.30, 52.68, 52.84, 52.86, 101.34, 101.56, 108.11, 108.72, 108.85, 113.73, 115.67, 116.05, 122.14, 123.02, 123.06, 127.11, 127.18, 127.39, 128.86, 147.70, 147.94, 148.12, 148.31, 212.51, 212.53$. HRMS (ESI/TOF) calcd for $\text{C}_{21}\text{H}_{23}\text{N}_2\text{O}_3^+$: 351.1703,

found: 351,1700. IR (NaCl): ν [cm^{-1}] = 3084, 2944, 2872, 2244, 1701, 1610, 1506, 1489, 1446, 1417, 1374, 1318, 1252, 1239, 1129, 1080, 1038, 992, 930, 863, 818, 775, 740.

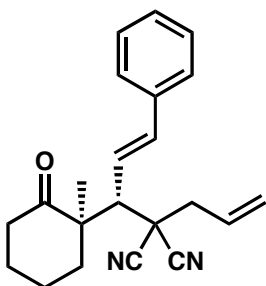
2-Allyl-2-((S)-benzo[d][1,3]dioxol-5-yl)((R)-1-methyl-2-oxocyclohexyl)methyl)malononitrile



4o

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 6:1, MnO_4 stain, $R_f = 0.30$) and isolated as a yellow powder. $[\alpha]_D^{25} = +44.08^\circ$ ($c = 0.73$, CH_2Cl_2 , 95% ee). ^1H nmr (500 MHz, CDCl_3): $\delta = 1.39$ (t, $J = 10.5$ Hz, 1H), 1.51 (d, $J = 14.0$ Hz, 1H), 1.59-1.63 (m, 1H), 1.69-1.80 (m, 2H), 1.74 (s, 3H), 1.97-2.01 (m, 1H), 2.45 (td, $J = 5.0, 13.5$ Hz, 1H), 2.58 (d, $J = 7.0$ Hz, 1H), 2.75 (ddd, $J = 6.0, 11.5, 13.5$ Hz, 1H), 3.84 (s, 1H), 5.31 (d, $J = 17.0$ Hz, 1H), 5.41 (d, $J = 10.0$ Hz, 1H), 5.93 (tdd, $J = 7.0, 10.0, 17.0$ Hz, 1H), 6.00 (s, 2H), 6.59 (br, 1H), 6.73 (d, $J = 7.5$ Hz, 0.61H), 6.87 (d, $J = 6.5$ Hz, 0.39H), 7.27 (s, 1H). ^{13}C nmr (125 MHz, CDCl_3): $\delta = 19.32, 20.89, 28.55, 38.28, 39.40, 39.63, 40.64, 40.89, 42.90, 52.65, 52.84, 101.34, 101.53, 107.94, 108.88, 113.59, 115.69, 116.14, 122.20, 123.10, 126.87, 127.16, 128.88, 147.65, 147.88, 148.06, 148.25, 214.20$. HRMS (ESI/TOF) calcd for $\text{C}_{21}\text{H}_{23}\text{N}_2\text{O}_3^+$: 351.1703, found: 351,1701. IR (NaCl): ν [cm^{-1}] = 3084, 2944, 2870, 2244, 2226, 1703, 1610, 1575, 1506, 1489, 1447, 1385, 1373, 1307, 1249, 1104, 1039, 989, 930, 868, 820, 740.

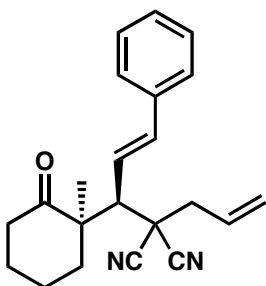
2-Allyl-2-((*R,E*)-1-((*R*)-1-methyl-2-oxocyclohexyl)-3-phenylallyl)malononitrile



3p

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 10:1, MnO₄ stain, $R_f = 0.30$) and isolated as an off-white solid that contains a small amount of unreacted Michael acceptor (unseparable mixture). $[\alpha]_D^{25} = +50.73^\circ$ ($c = 0.11$, CH₂Cl₂, 58% ee). ¹H nmr (500 MHz, CDCl₃): $\delta = 1.12$ (s, 3H), 1.78-1.86 (m, 1H), 1.97-2.06 (m, 4H), 2.33 (dt, $J = 3.5, 13.0$ Hz, 1H), 2.49-2.59 (m, 2H), 2.74 (dd, $J = 7.5, 14.0$ Hz, 1H), 2.83 (tdd, $J = 1.0, 7.0, 14.0$ Hz, 1H), 3.61 (d, $J = 11.0$ Hz, 1H), 5.39 (ddd, $J = 1.0, 2.5, 17.0$ Hz, 1H), 5.44 (d, $J = 10.0$ Hz, 1H), 5.95 (dddd, $J = 6.5, 7.5, 10.0, 17.0$ Hz, 1H), 6.16 (dd, $J = 11.0, 16.0$ Hz, 1H), 6.62 (d, $J = 16.0$ Hz, 1H), 7.32 (tt, $J = 1.0, 7.0$ Hz, 1H), 7.36 (t, $J = 7.0$ Hz, 2H), 7.43 (d, $J = 7.0$ Hz, 1H). ¹³C nmr (125 MHz, CDCl₃): $\delta = 20.63, 24.24, 25.62, 32.22, 38.12, 38.92, 41.96, 51.30, 52.00, 114.97, 115.44, 121.31, 123.02, 126.78, 128.70, 128.79, 128.83, 135.45, 138.81, 212.84$. GHMBC (500 MHz, CDCl₃): (3.61, 114.60) and (3.61, 115.06) indicating the CH of the tertiary stereocenter next to the two nitrile groups. HRMS (ESI/TOF) calcd for C₂₂H₂₅N₂O⁺: 333.1961, found: 333.1957. IR (NaCl): ν [cm⁻¹] = 3084, 3060, 3029, 2944, 2871, 2243, 1699, 1645, 1600, 1497, 1450, 1418, 1378, 1286, 1266, 1221, 1132, 1075, 978, 936, 801, 760, 737, 699.

2-Allyl-2-((*S,E*)-1-((*R*)-1-methyl-2-oxocyclohexyl)-3-phenylallyl)malononitrile

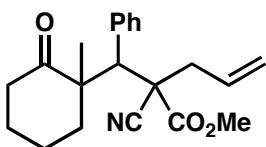


4p

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 10:1, MnO₄ stain, $R_f = 0.25$) and isolated as an off-white solid. $[\alpha]_D^{25} = +66.70^\circ$ ($c = 0.79$, CH₂Cl₂, 82% ee). ¹H nmr (500 MHz, CDCl₃): $\delta = 1.60$ -1.84 (m, 5H), 1.63 (s, 3H), 2.02-2.05 (m, 1H), 2.46 (td, $J = 4.0, 13.0$ Hz, 1H), 2.72 (td, $J = 6.0, 12.0$ Hz, 1H), 2.78 (dd, $J = 8.0, 14.0$ Hz, 1H), 2.84 (dd, $J = 7.0, 14.0$ Hz, 1H), 3.32 (d, $J = 10.5$ Hz, 1H), 5.41 (d, $J = 17.0$ Hz, 1H), 5.45 (d, $J = 10.0$ Hz, 1H), 5.98 (tdd, $J = 7.0, 10.0, 17.0$ Hz, 1H), 6.18 (dd, $J = 10.5, 16.0$ Hz, 1H), 6.59 (d, $J = 16.0$ Hz, 1H), 7.32 (t, $J = 7.0$ Hz, 1H), 7.37 (t, $J = 7.5$ Hz, 2H), 7.43 (d, $J = 7.5$ Hz, 2H). ¹³C nmr (125 MHz, CDCl₃): $\delta = 17.99, 20.66, 29.07, 38.44, 38.77, 41.40, 42.23, 51.93, 51.99, 115.18, 121.57, 123.08, 126.71, 128.68, 128.79, 128.85, 135.36, 138.21, 214.22$. GHMBC (500 MHz, CDCl₃): (3.32, 115.18) indicating the CH of the tertiary stereocenter next to the two nitrile groups (which show up as one single signal in this case). HRMS (ESI/TOF) calcd for C₂₂H₂₅N₂O⁺: 333.1961, found: 333.1961.

IR (NaCl): ν [cm⁻¹] = 3084, 3060, 3028, 2939, 2867, 2245, 1704, 1643, 1496, 1450, 1387, 1308, 1116, 978, 936, 759, 703.

Methyl 2-cyano-2-((1-methyl-2-oxocyclohexyl)(phenyl)methyl)pent-4-enoate

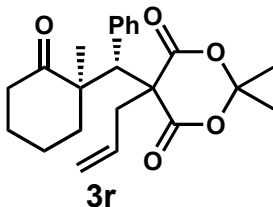


3,4q

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 8:1, MnO₄ stain, $R_f = 0.35$) and isolated as an unseparable mixture of

both diastereomers. The given nmr data was acquired from the racemic sample. The ^1H nmr spectrum of the enantioenriched sample (dr 4.1:1:0:0, 64% ee, major) is attached as well. ^1H nmr (500 MHz, CDCl_3): δ = 0.88 (s, 1.8H), 1.23-1.36 (m, 2.6H), 1.51 (s, 3H), 1.58-1.68 (m, 3.4H), 1.85-1.95 (m, 3.2H), 1.99-2.05 (m, 1.6H), 2.29-2.39 (m, 3H), 2.52-2.56 (dd, J = 7.5, 13.5 Hz, 0.6H), 2.81-2.89 (m, 1.6H), 3.71 (s, 3H), 3.73 (s, 1.8H), 3.86 (s, 1H), 3.97 (s, 0.6H), 4.98-5.11 (m, 3.2H), 5.57-5.70 (m, 1.6H), 7.12-7.14 (dt, J = 7.0, 2.0 Hz, 0.6H), 7.25-7.40 (m, 4.8H), 7.44 (t, J = 6.5 Hz, 1H), 7.84 (d, J = 8.0 Hz, 0.6H), 7.92 (d, J = 8.0 Hz, 1H). ^{13}C nmr (125 MHz, CDCl_3): δ = 20.20, 20.47, 20.50, 24.55, 25.71, 27.57, 32.57, 38.33, 38.46, 40.19, 42.71, 44.93, 51.11, 51.43, 52.32, 53.09, 53.14, 53.17, 54.19, 54.51, 119.15, 119.72, 120.88, 120.94, 127.65, 127.95, 128.00, 128.25, 128.32, 128.35, 128.86, 129.08, 129.93, 130.65, 133.33, 133.38, 134.84, 136.10, 168.39, 168.58, 212.42, 214.56. HRMS (ESI/TOF) calcd for $\text{C}_{21}\text{H}_{26}\text{NO}_3^+$: 340.1907, found: 340.1907. IR (NaCl): ν [cm^{-1}] = 3061, 3007, 2949, 2868, 2243, 1744, 1701, 1642, 1493, 1453, 1437, 1386, 1310, 1267, 1250, 1230, 1142, 1089, 991, 932, 798, 766, 712.

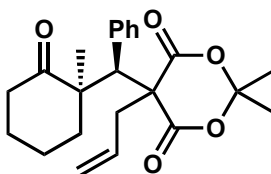
5-Allyl-2,2-dimethyl-5-((*S*)-((*R*)-1-methyl-2-oxocyclohexyl)(phenyl)methyl)-1,3-dioxane-4,6-dione



Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 6:1, MnO_4 stain, R_f = 0.4) and isolated as a white amorphous solid. $[\alpha]_D^{25}$ = +5.19° (c = 0.905, CH_2Cl_2 , 22% ee). ^1H nmr (500 MHz, CDCl_3): δ = 1.17 (ddd, J = 4.0, 12.0, 14.5 Hz, 1H), 1.24 (s, 3H), 1.29-1.37 (m, 1H), 1.48 (s, 3H), 1.54 (d, J = 14.5 Hz, 1H), 1.57 (s, 3H), 1.62-1.72 (m, 2H), 2.01-2.09 (m, 1H), 2.31 (dd, J = 7.5, 12.5 Hz, 1H), 2.45 (d, J = 12.5 Hz, 1H), 2.69 (dd, J = 7.5, 13.0 Hz, 1H), 3.07 (ddd, J = 6.5, 12.5, 13.5 Hz, 1H), 4.40 (s, 1H), 5.07 (d, J = 17.0 Hz, 1H), 5.10 (d, J = 10.0 Hz, 1H), 5.49 (ddd, J = 7.5, 10.0, 17.0 Hz, 1H), 7.24 (td, J = 2.0, 7.0 Hz, 1H), 7.30 (ddd, J = 0.5, 2.0, 7.0 Hz, 1H), 7.33 (ddd, J = 1.5, 1.5, 7.5 Hz, 1H), 7.37-7.40 (m, 1H), 7.74 (d, J = 8.0 Hz, 1H). ^{13}C nmr (125 MHz, CDCl_3): δ = 20.48, 27.82, 28.51, 30.77, 39.09, 42.32, 43.42,

53.20, 56.07, 58.67, 106.42, 122.09, 128.14, 128.26, 128.73, 130.18, 130.89, 133.59, 135.82, 167.56, 169.07, 216.64. HRMS (ESI/TOF) calcd for $C_{23}H_{32}NO_5^+$ [$M+NH_4^+$]: 402.2275, found: 402.2275. IR (NaCl): ν [cm^{-1}] = 3060, 3026, 2983, 2944, 2867, 1768, 1733, 1701, 1640, 1600, 1581, 1494, 1455, 1415, 1379, 1392, 1359, 1323, 1265, 1205, 1140, 1089, 1030, 997, 937, 897, 860, 821, 765, 737, 715.

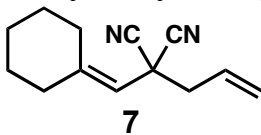
5-Allyl-2,2-dimethyl-5-((*R*)-((*R*)-1-methyl-2-oxocyclohexyl)(phenyl)methyl)-1,3-dioxane-4,6-dione



4r

Synthesised according to the general procedure. Purified by flash chromatography (hexanes/EtOAc 6:1, MnO_4 stain, $R_f = 0.3$) and isolated as a white amorphous solid. $[\alpha]_D^{25} = -6.28^\circ$ ($c = 0.79$, CH_2Cl_2 , 43% ee). 1H nmr (500 MHz, $CDCl_3$): $\delta = 0.87$ (s, 3H), 1.26 (s, 3H), 1.56 (s, 3H), 1.60-1.69 (m, 1H), 1.84-1.95 (m, 4H), 2.43 (ddd, $J = 7.0, 10.5, 16.5$ Hz, 1H), 2.50 (dddd, $J = 2.0, 4.5, 6.0, 16.5$ Hz, 1H), 2.63 (dd, $J = 7.0, 12.5$ Hz, 1H), 2.81 (dd, $J = 7.5, 12.5$ Hz, 1H), 2.91 (dt, $J = 4.0, 14.0$ Hz, 1H), 4.41 (s, 1H), 5.11 (d, $J = 10.0$ Hz, 1H), 5.14 (d, $J = 17.0$ Hz, 1H), 5.54 (ddd, $J = 7.5, 10.0, 17.0$ Hz, 1H), 7.11 (td, $J = 2.0, 7.0$ Hz, 1H), 7.24-7.33 (m, 3H), 7.49 (d, $J = 8.0$ Hz, 1H). ^{13}C nmr (125 MHz, $CDCl_3$): $\delta = 20.93, 24.33, 26.92, 27.84, 30.83, 33.48, 38.63, 41.71, 52.60, 56.28, 59.29, 106.01, 121.97, 127.80, 128.14, 128.39, 129.63, 131.49, 133.83, 136.77, 167.94, 169.05, 213.61$. HRMS (ESI/TOF) calcd for $C_{23}H_{32}NO_5^+$ [$M+NH_4^+$]: 402.2275, found: 402.2272. IR (NaCl): ν [cm^{-1}] = 3060, 2979, 2943, 2870, 1769, 1734, 1700, 1640, 1600, 1495, 1415, 1453, 1392, 1379, 1320, 1298, 1264, 1203, 1159, 1130, 1083, 1028, 998, 938, 735, 706.

2-Allyl-2-(cyclohexylidenemethyl)malononitrile



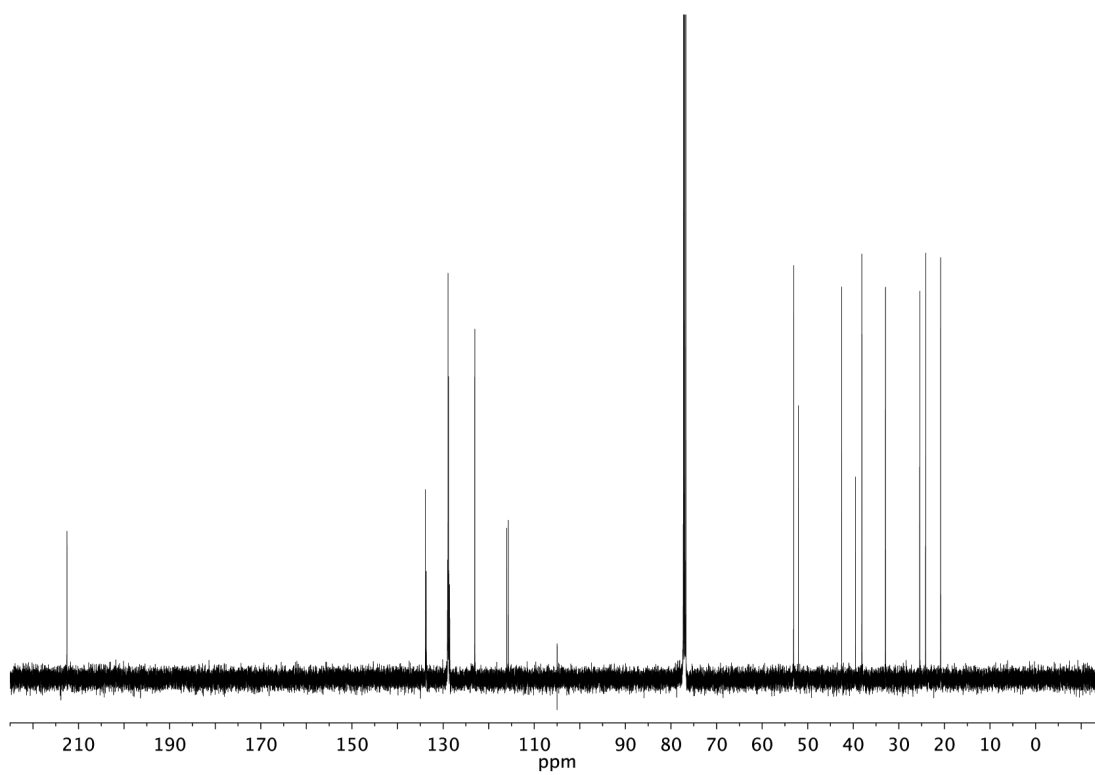
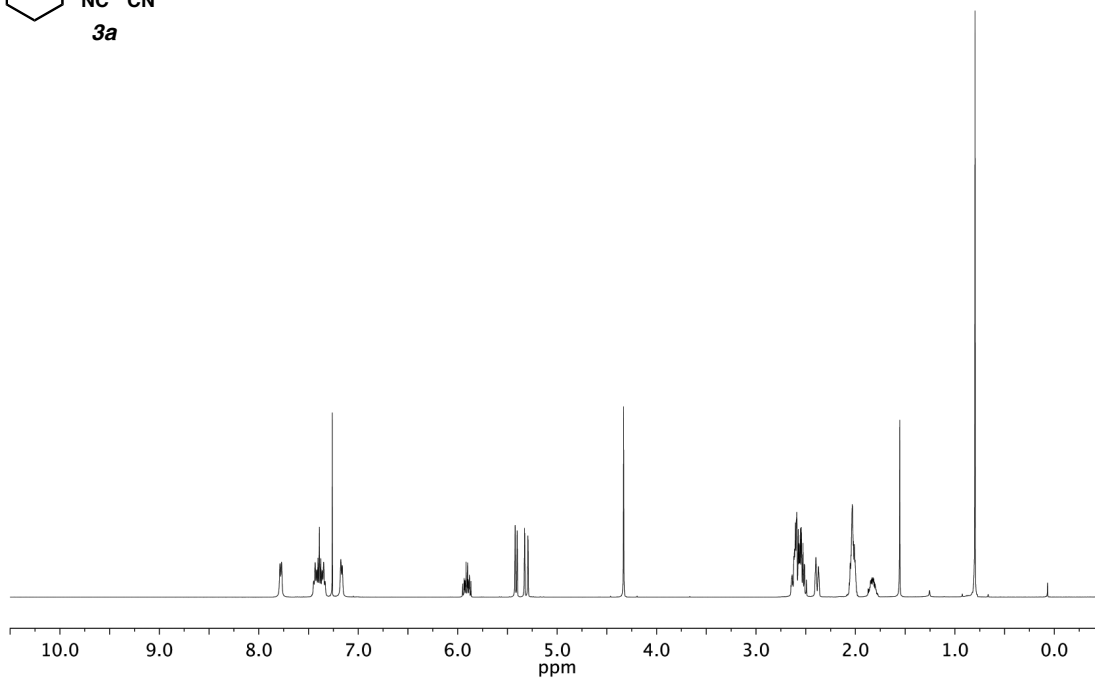
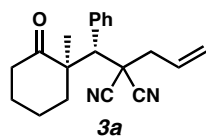
Isolated as sideproduct from the reaction of **1a** with **2k** following the conditions for racemic product synthesis (dppe as ligand). Flash chromatography (hexanes/EtOAc 15:1 yields this compound as a colorless oil. ^1H nmr (300 MHz, CDCl_3): $\delta = 1.59$ - 1.64 (m, 4H), 1.67 - 1.74 (m, 2H), 2.15 (t, $J = 6.0$ Hz, 2H), 2.48 (t, $J = 6.0$ Hz, 2H), 2.78 (td, $J = 1.5$, 12.0 Hz, 2H), 5.05 (s, 1H), 5.42 (tdd, $J = 1.2$, 1.5 , 16.2 Hz, 1H), 5.43 (tdd, $J = 0.8$, 3.0 , 10.5 Hz, 1H), 5.91 (tdd, $J = 7.2$, 10.5 , 16.2 Hz, 1H). ^{13}C nmr (75 MHz, CDCl_3): $\delta = 25.68$, 26.23 , 28.09 , 30.58 , 33.25 , 36.78 , 43.00 , 112.06 , 112.08 , 115.20 , 123.11 , 128.63 , 153.13 .

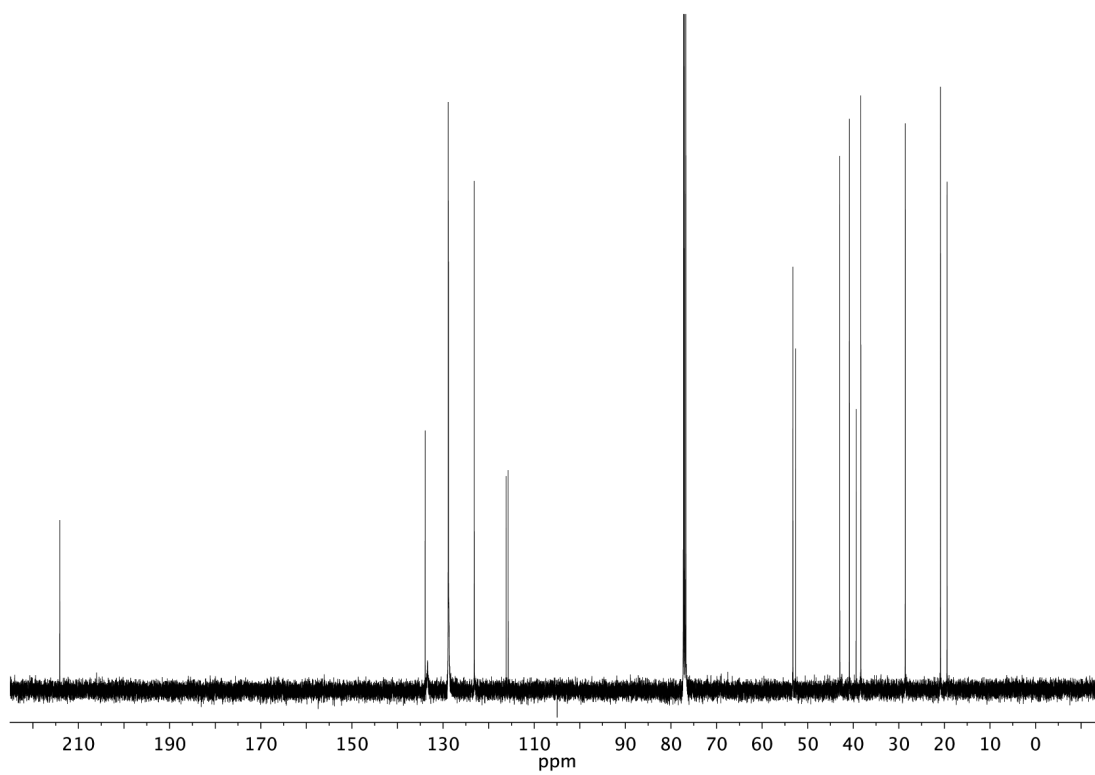
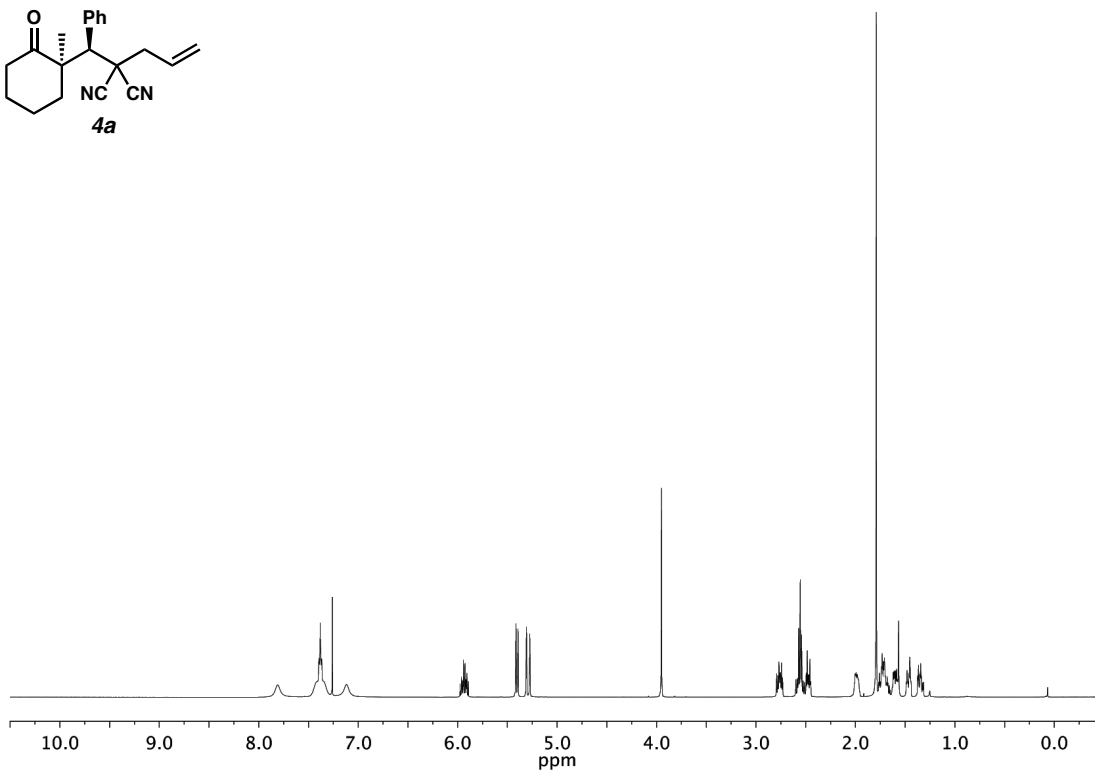
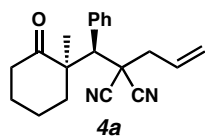
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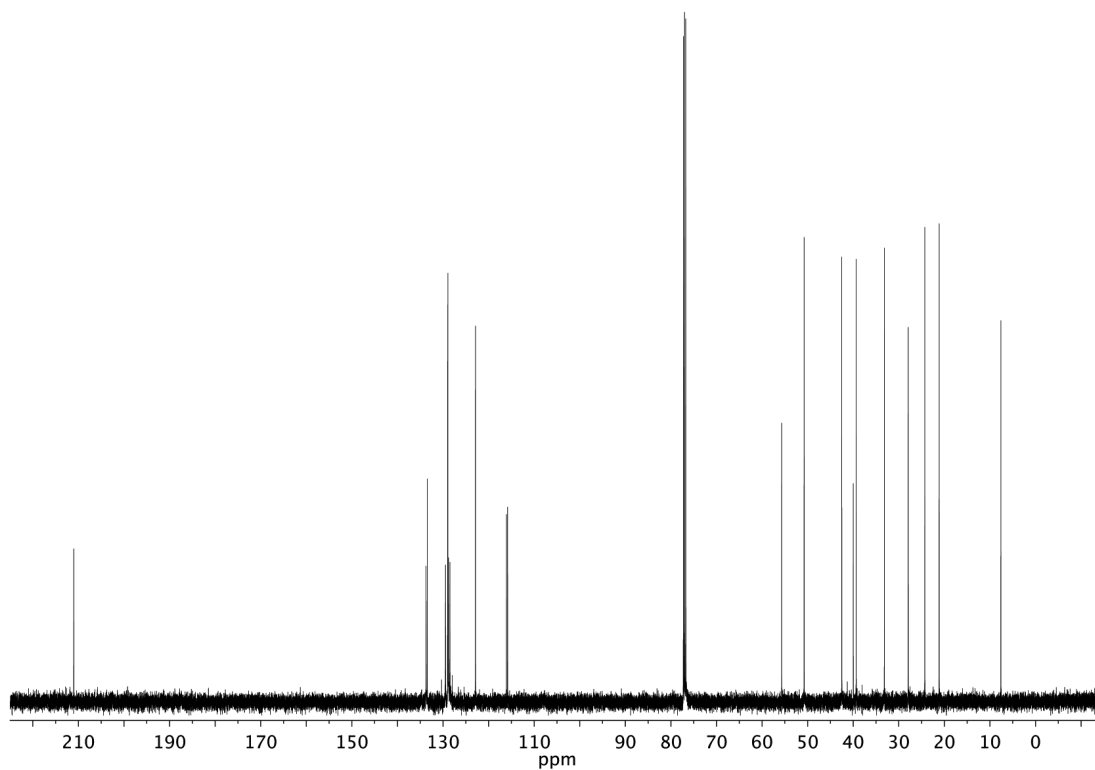
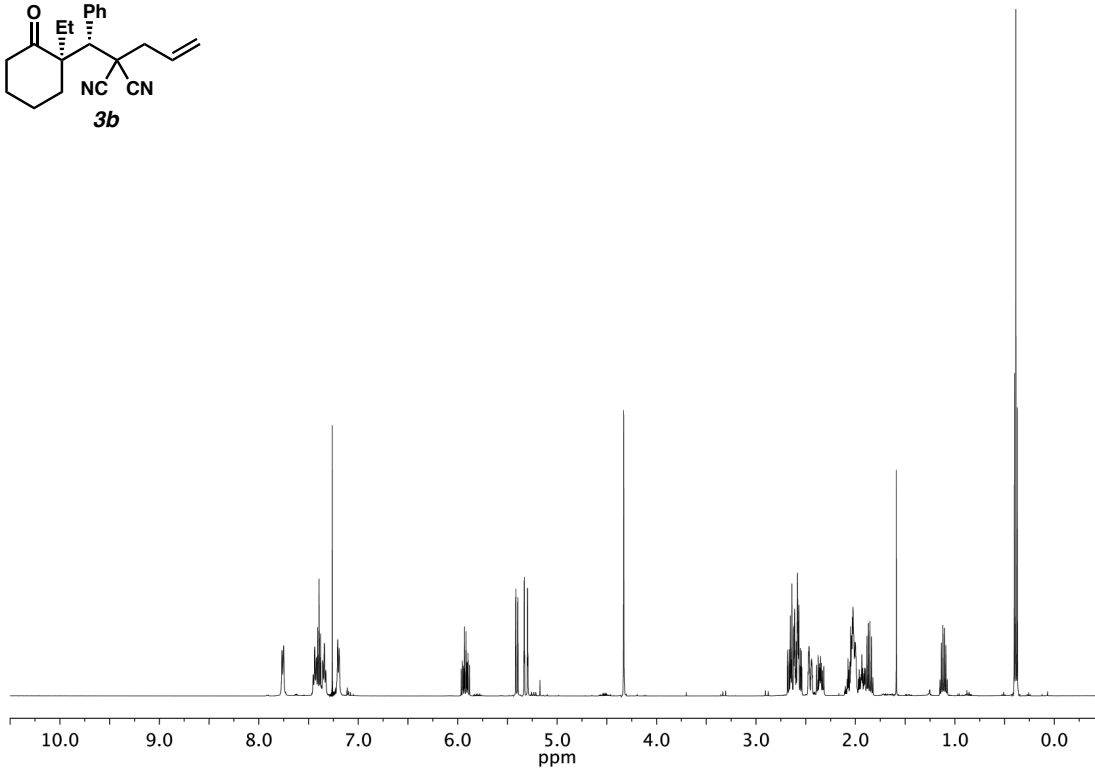
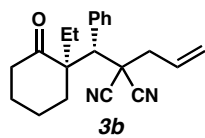
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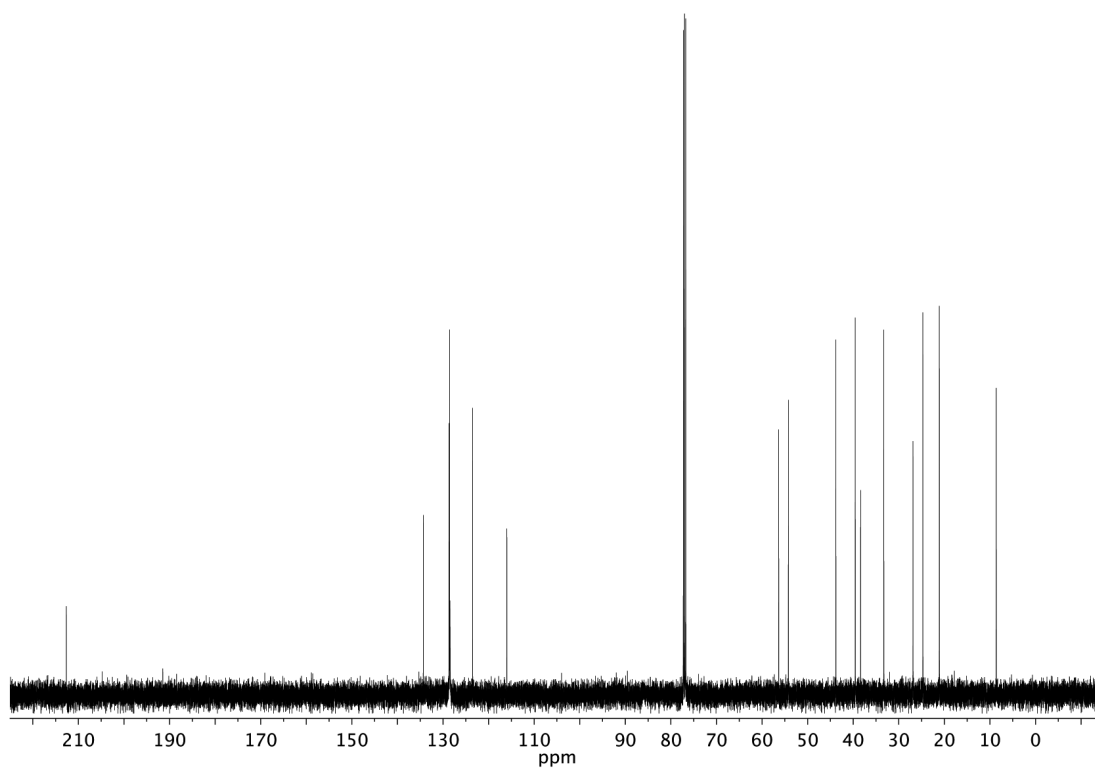
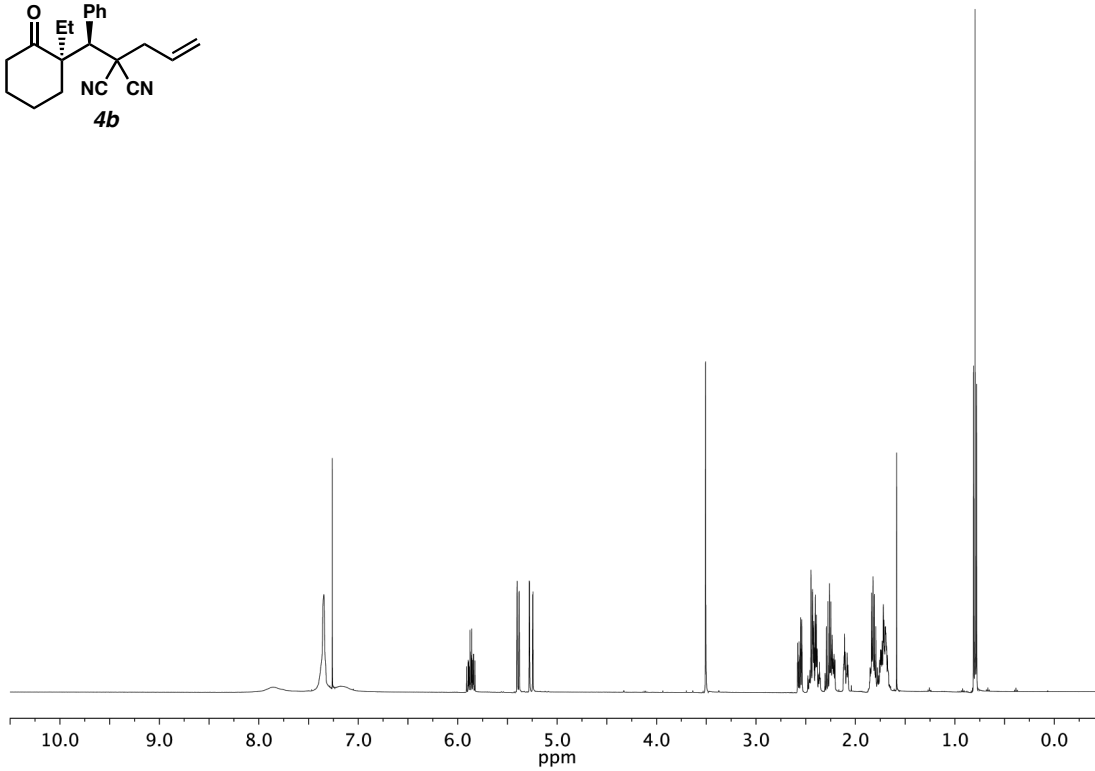
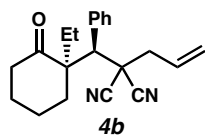
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10. Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 732239.
 11. Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 732240.

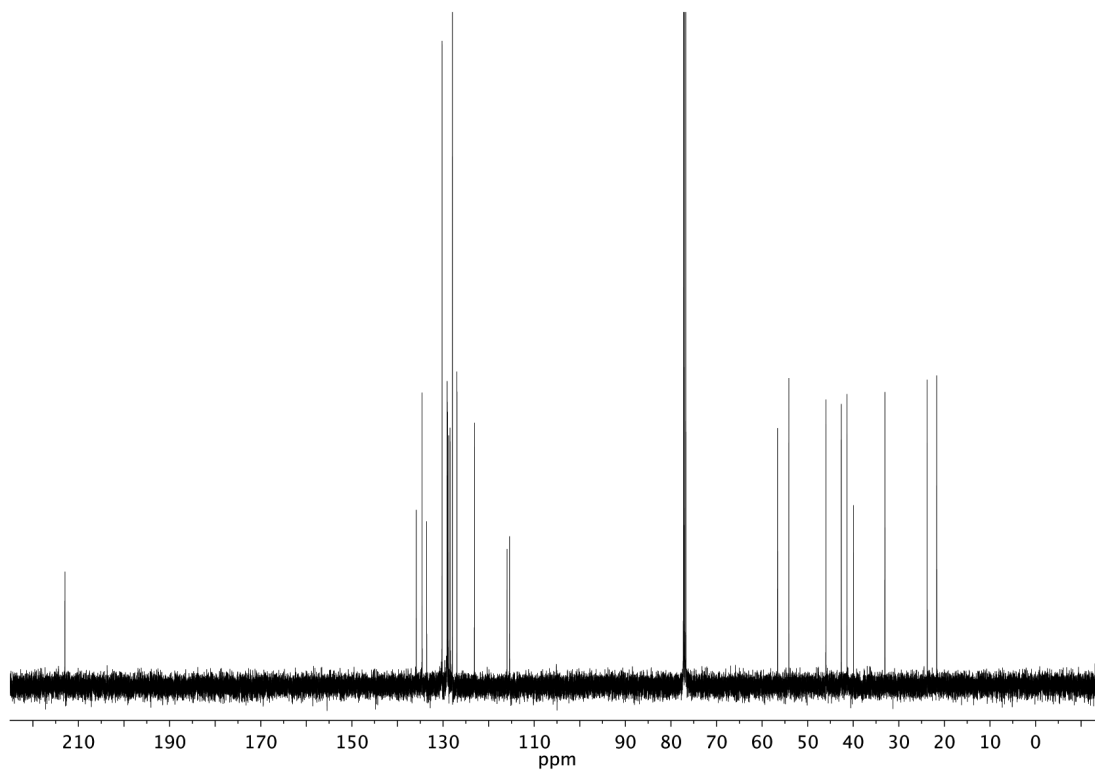
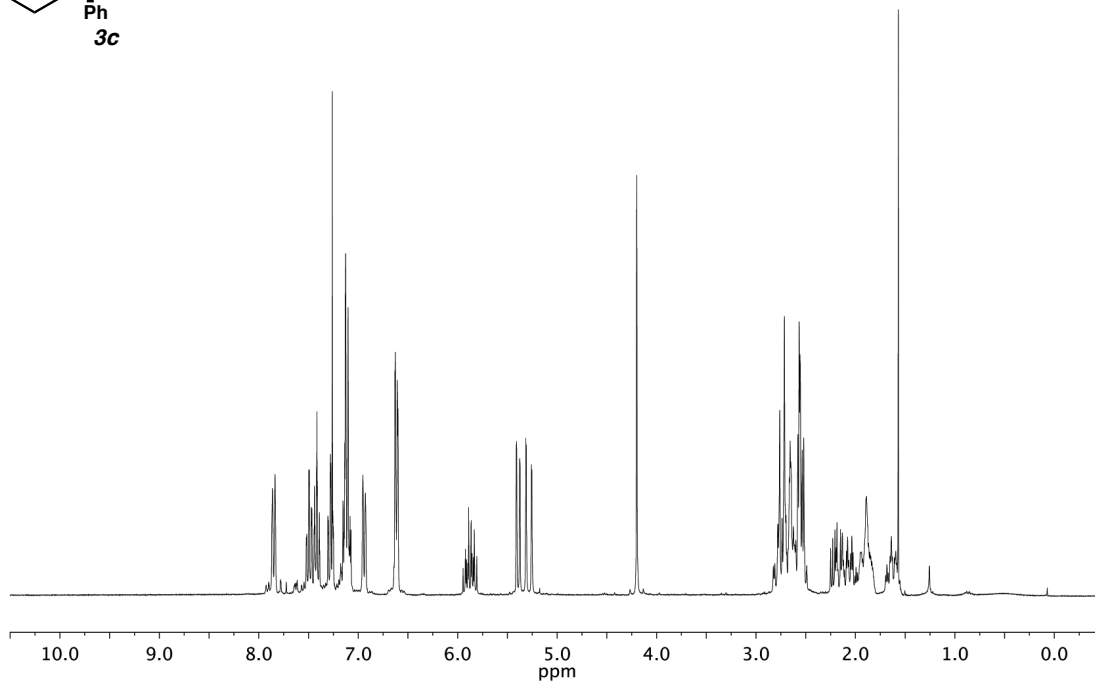
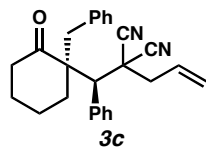
^1H and ^{13}C NMR Spectra of Products 3 and 4

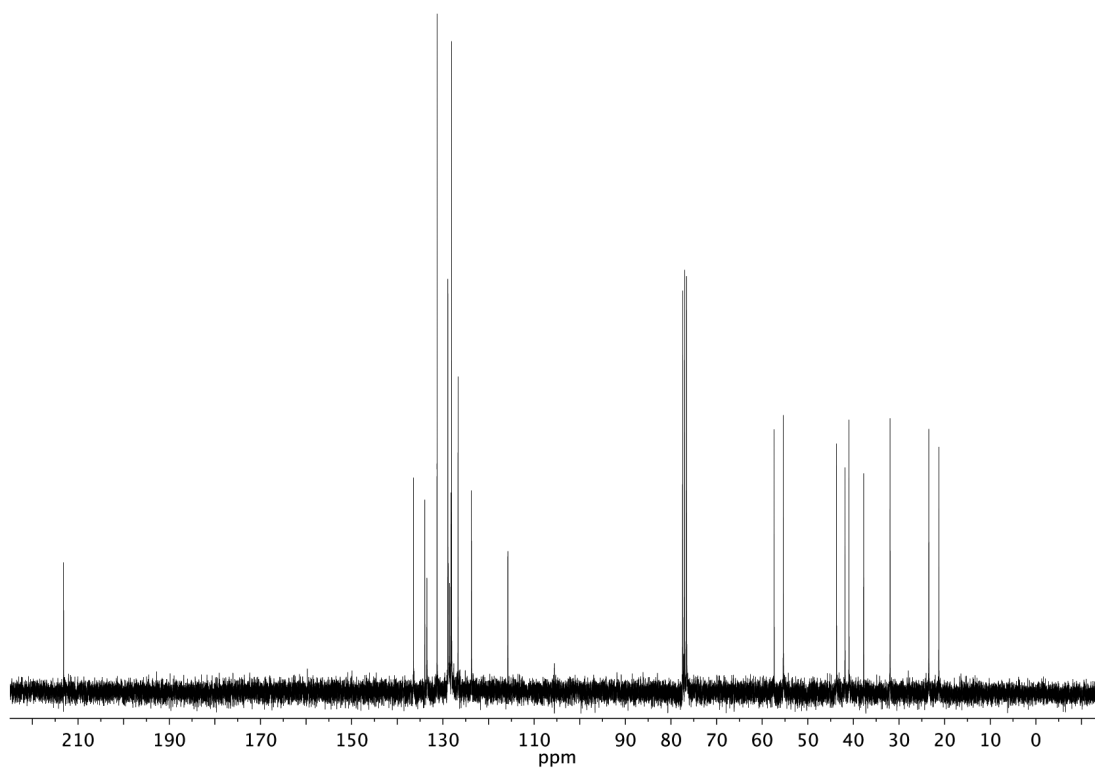
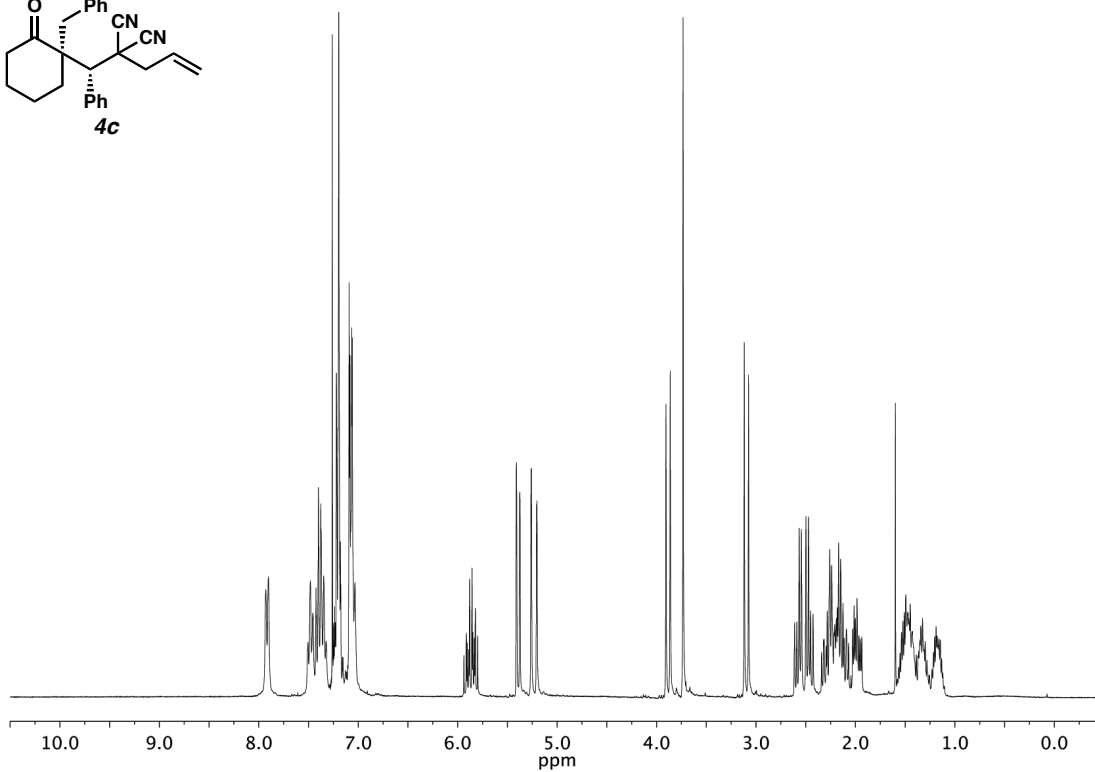
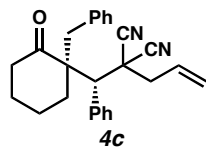


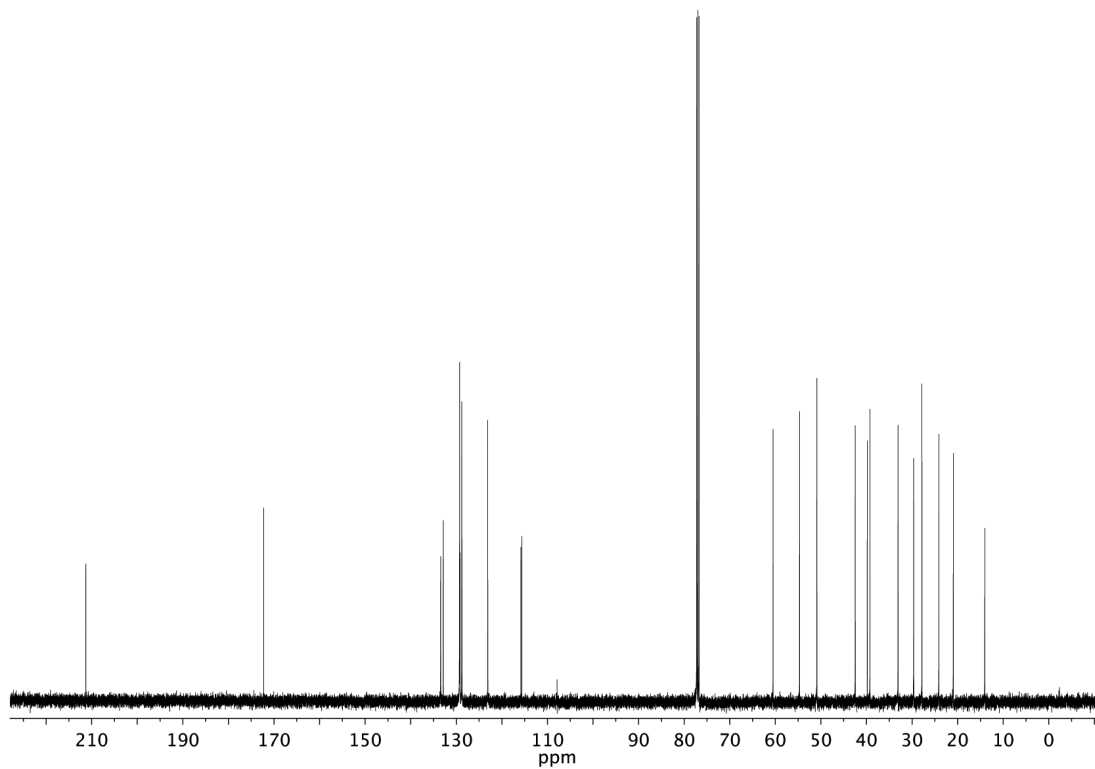
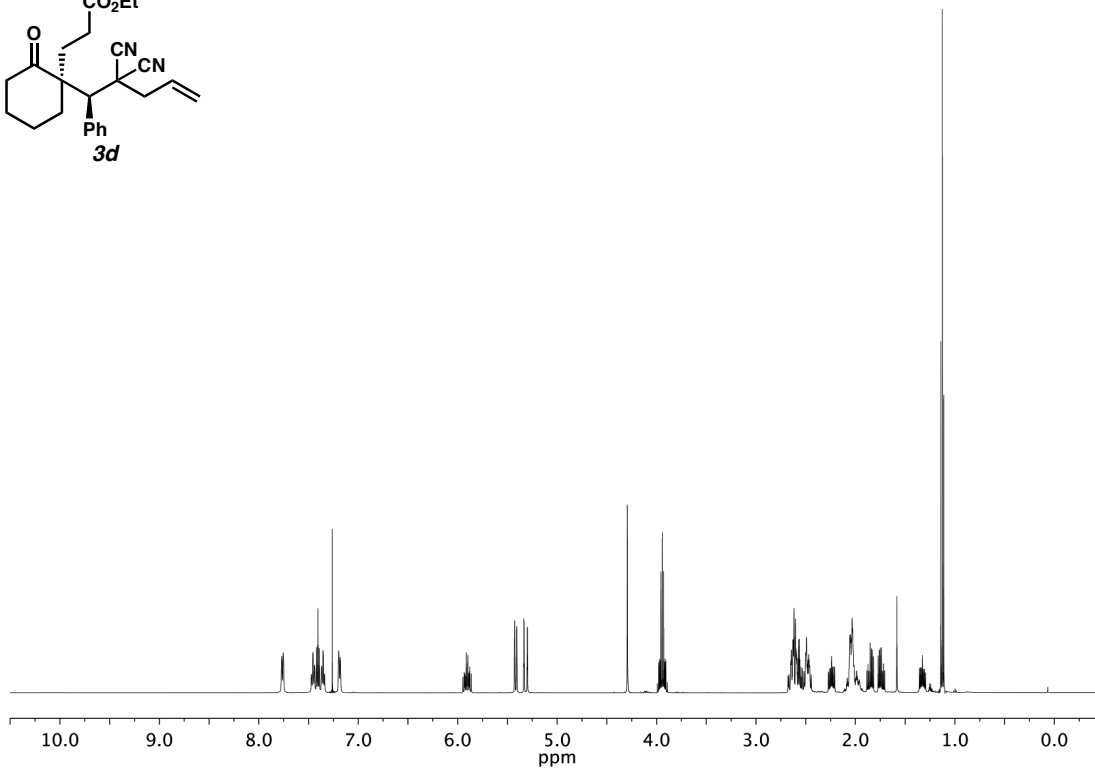
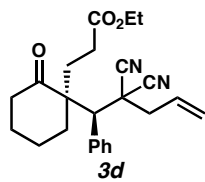


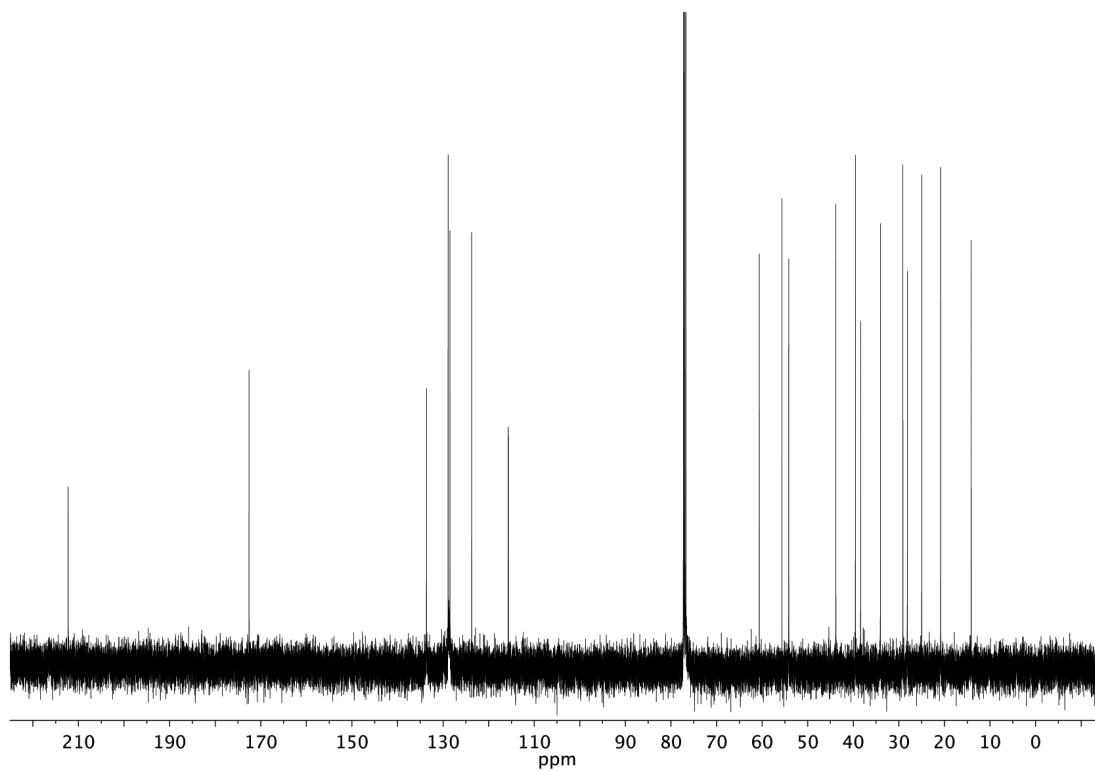
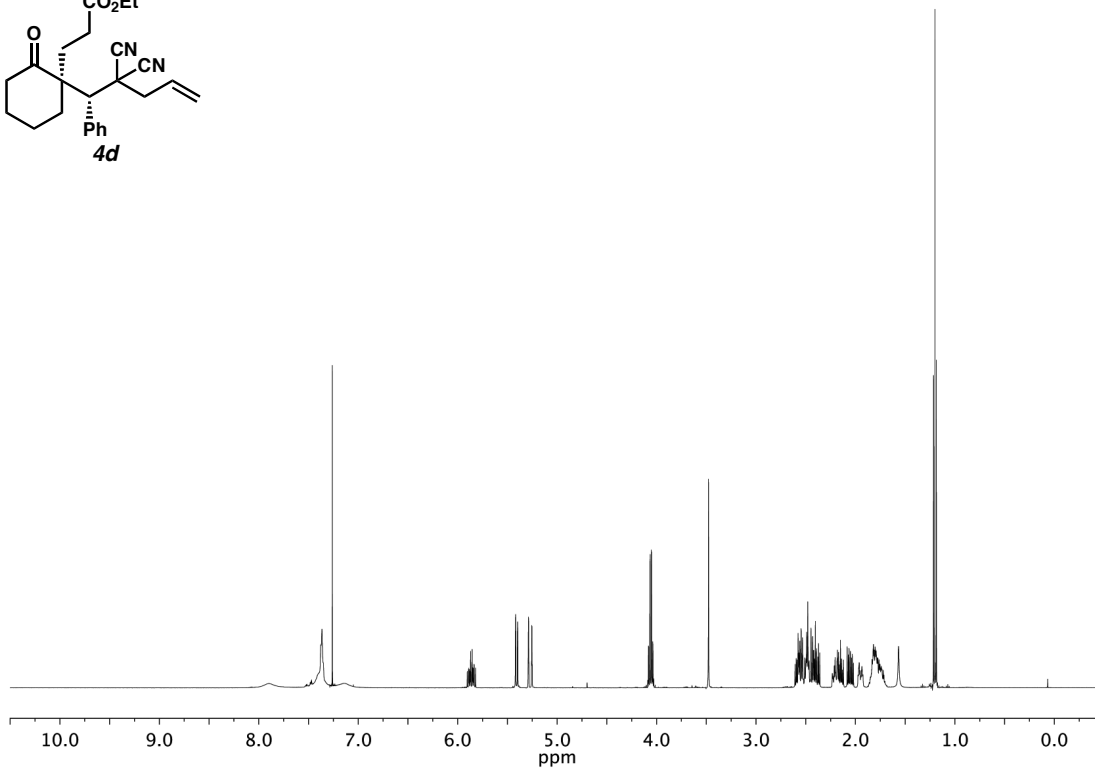
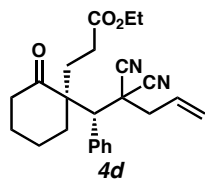


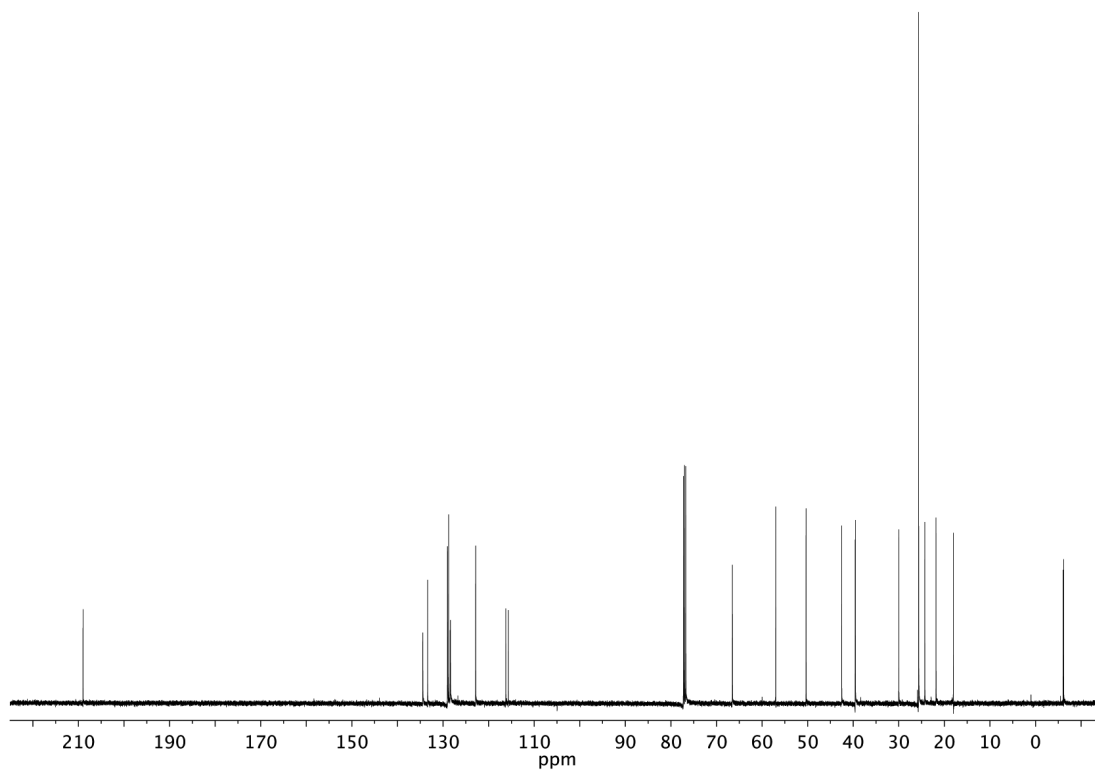
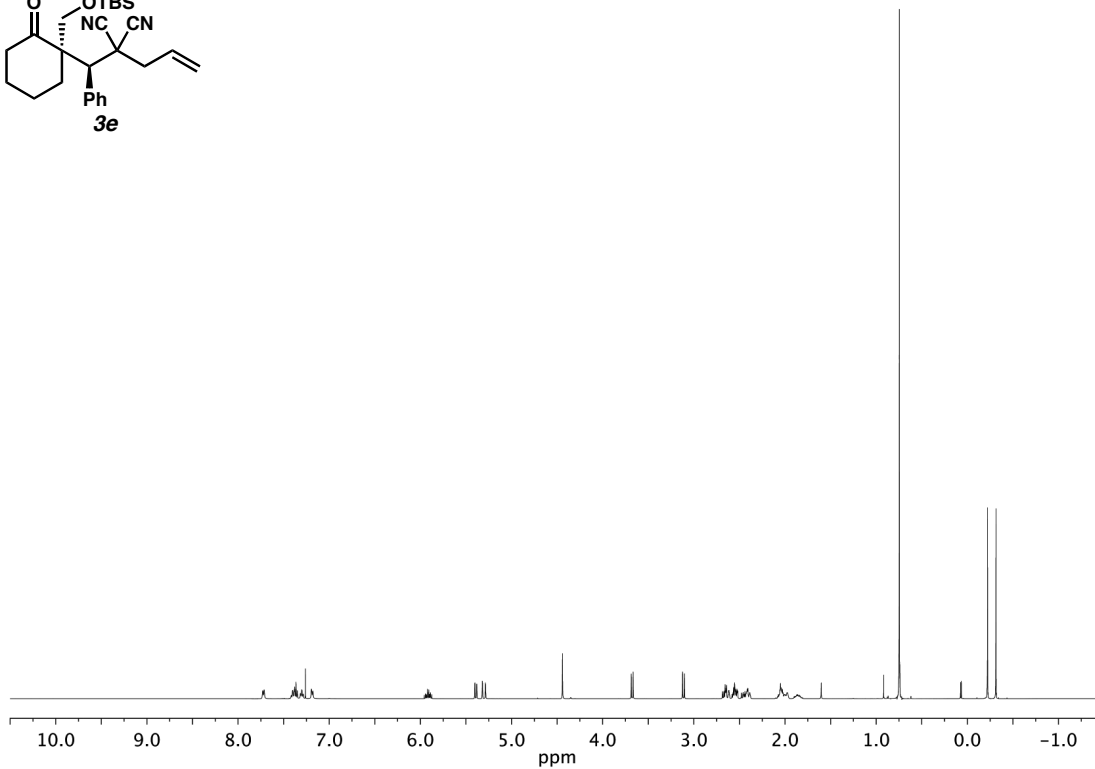
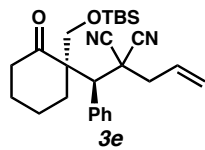


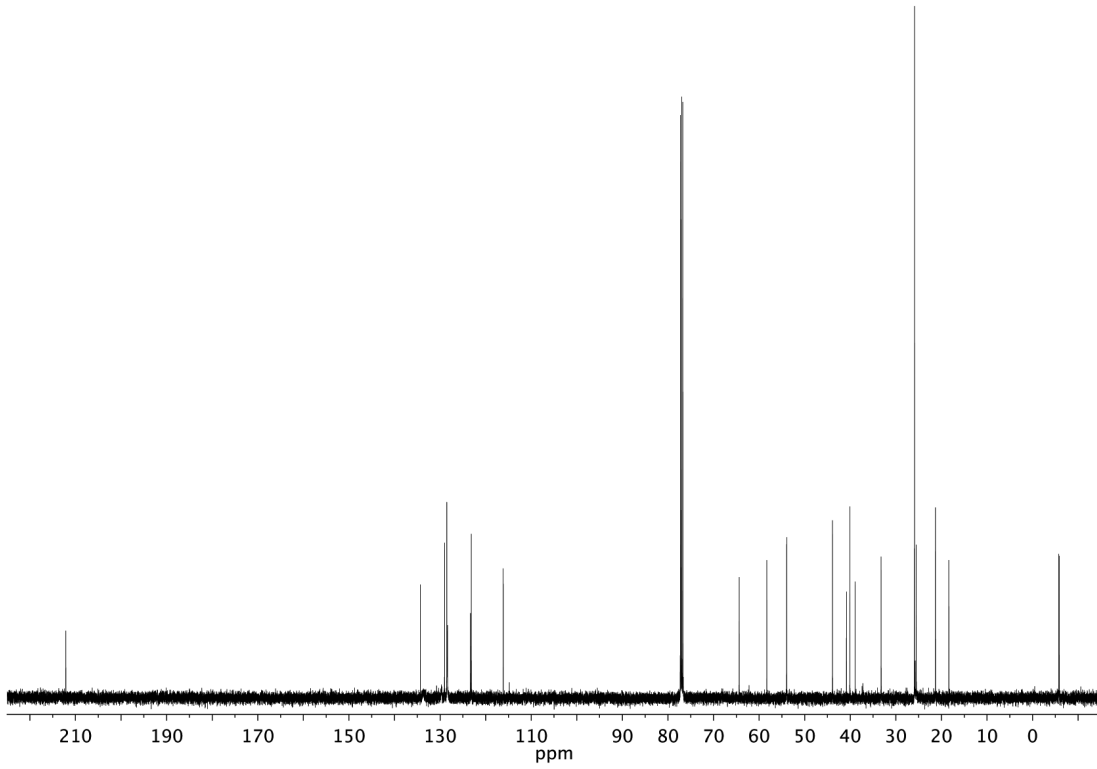
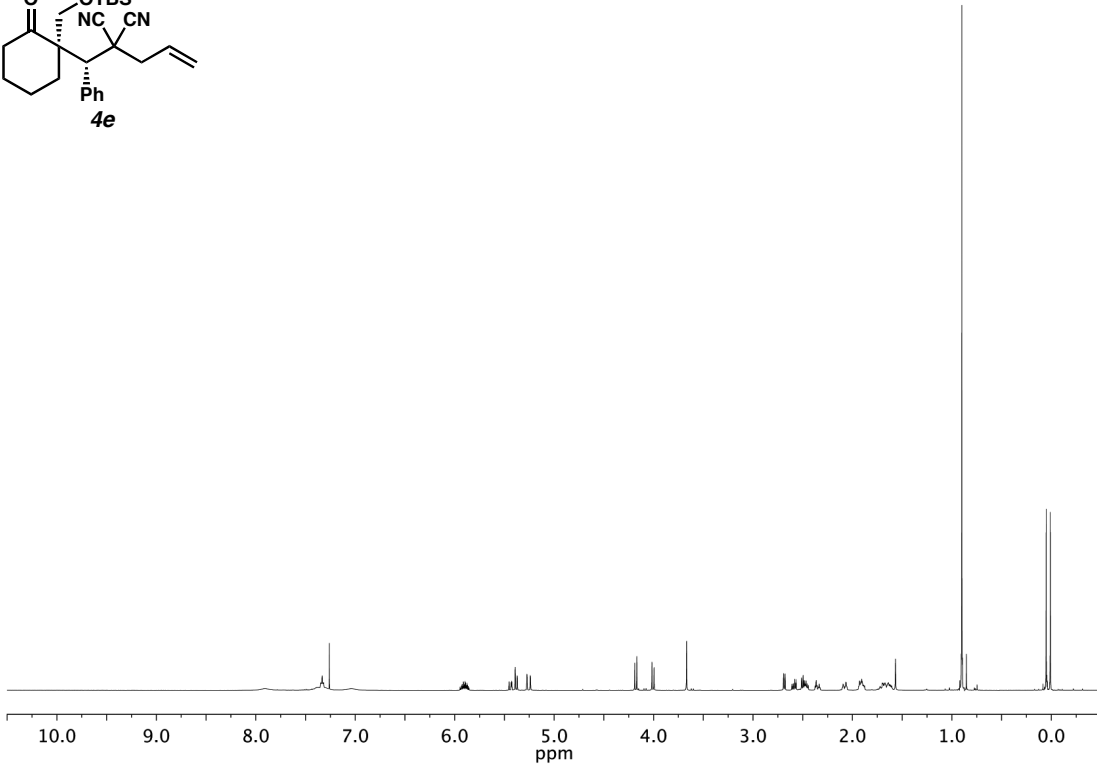
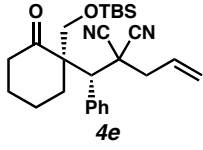


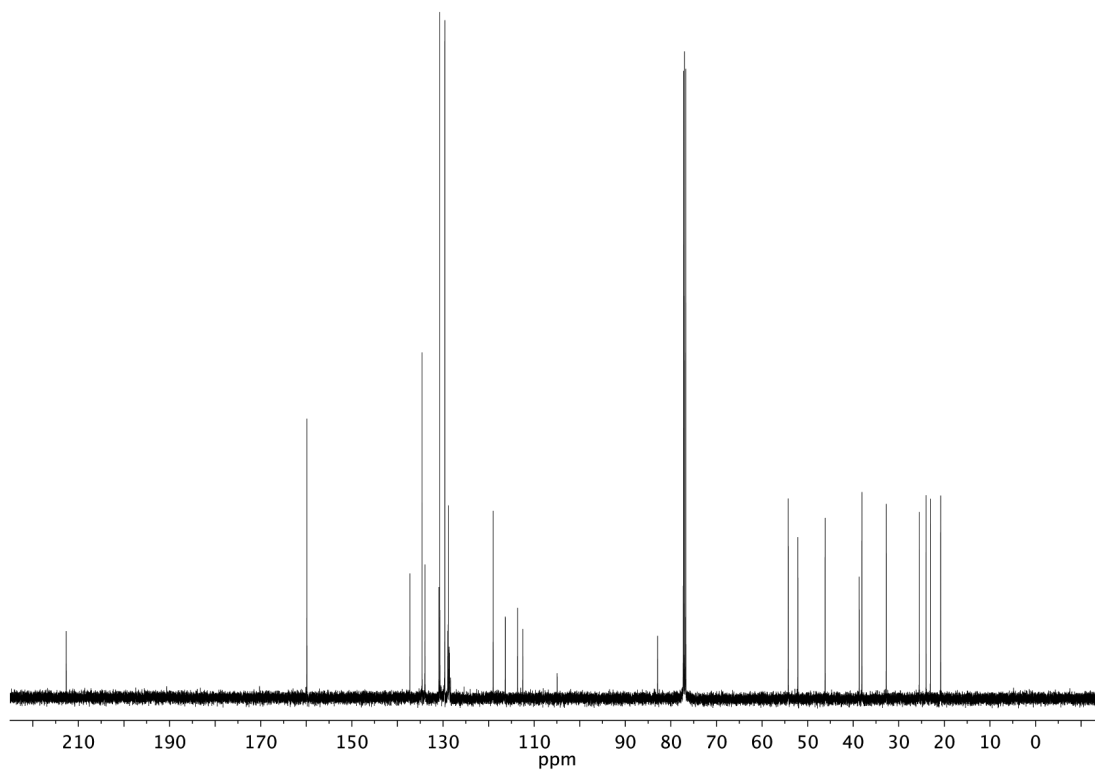
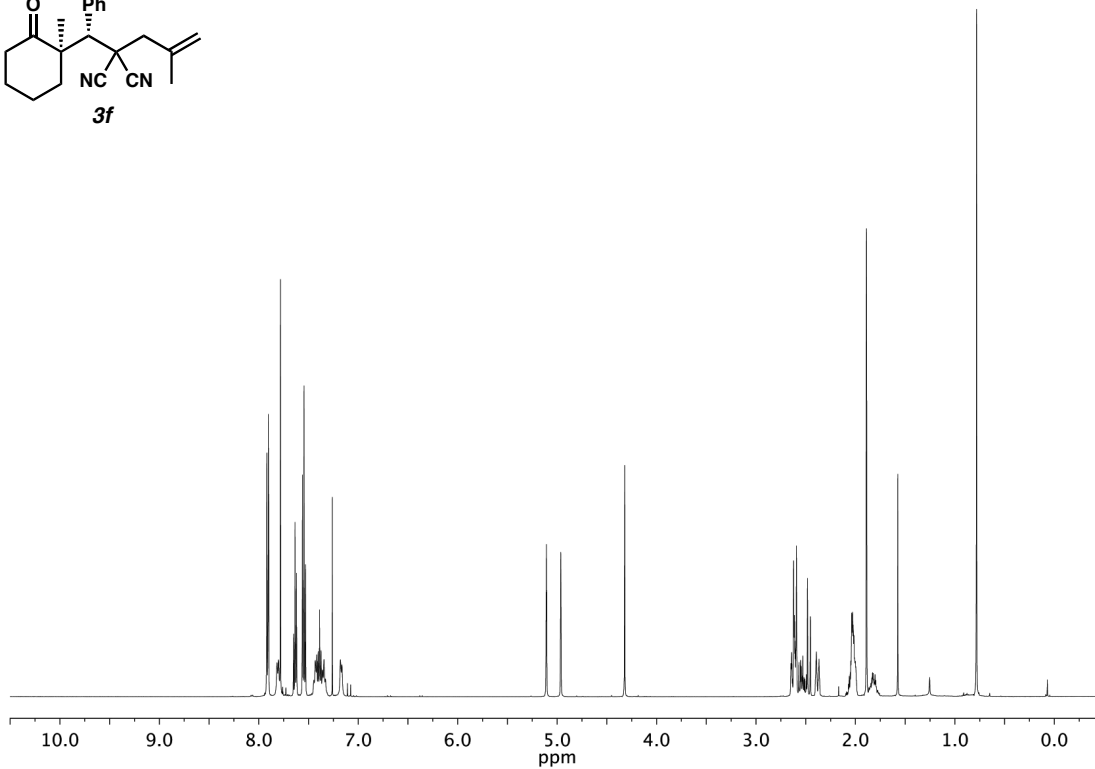
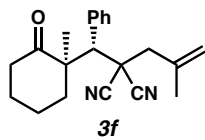


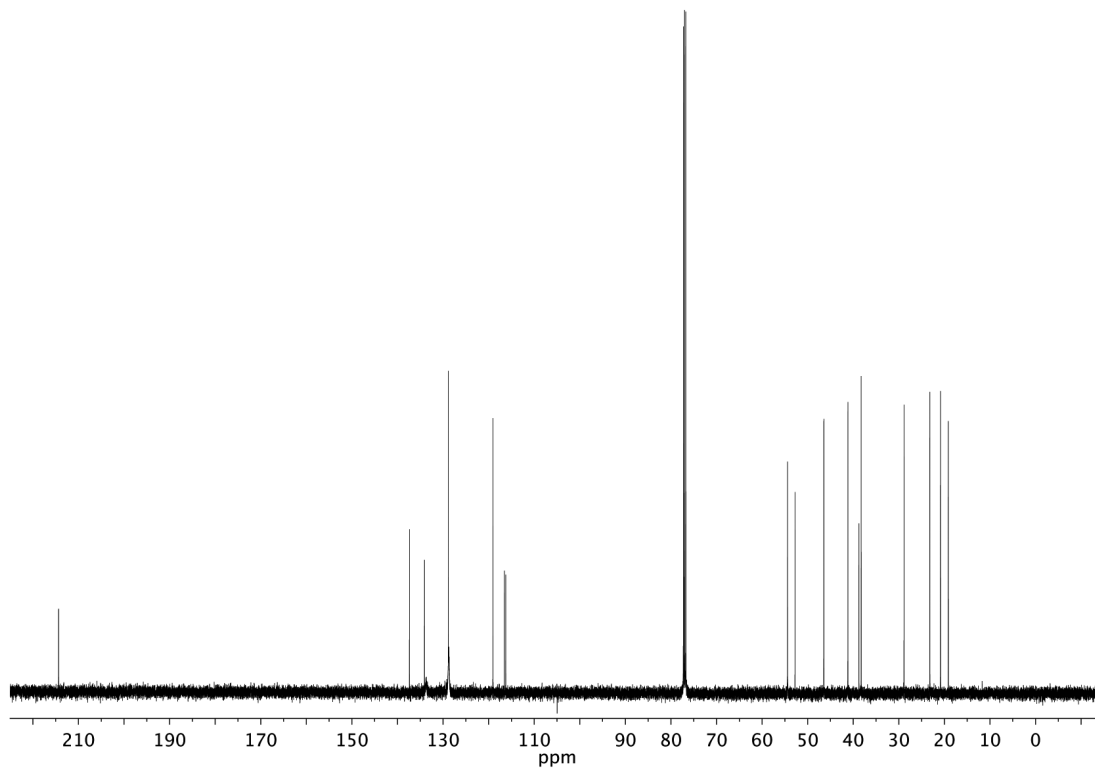
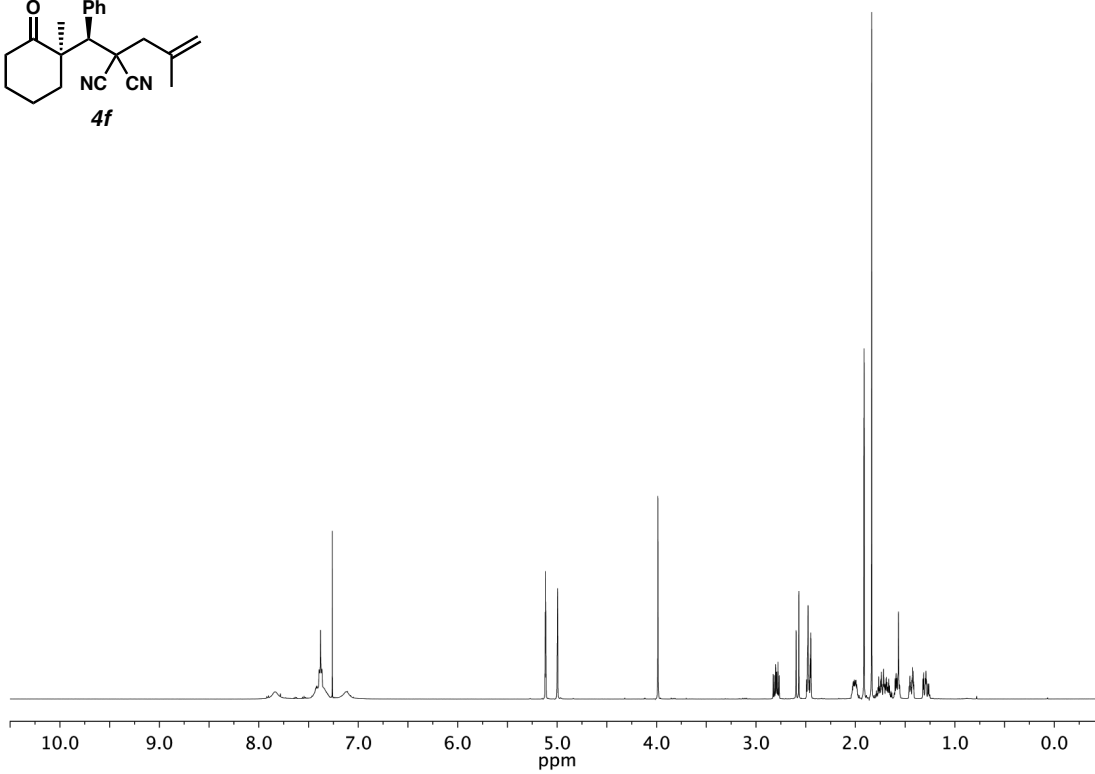
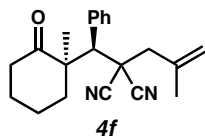


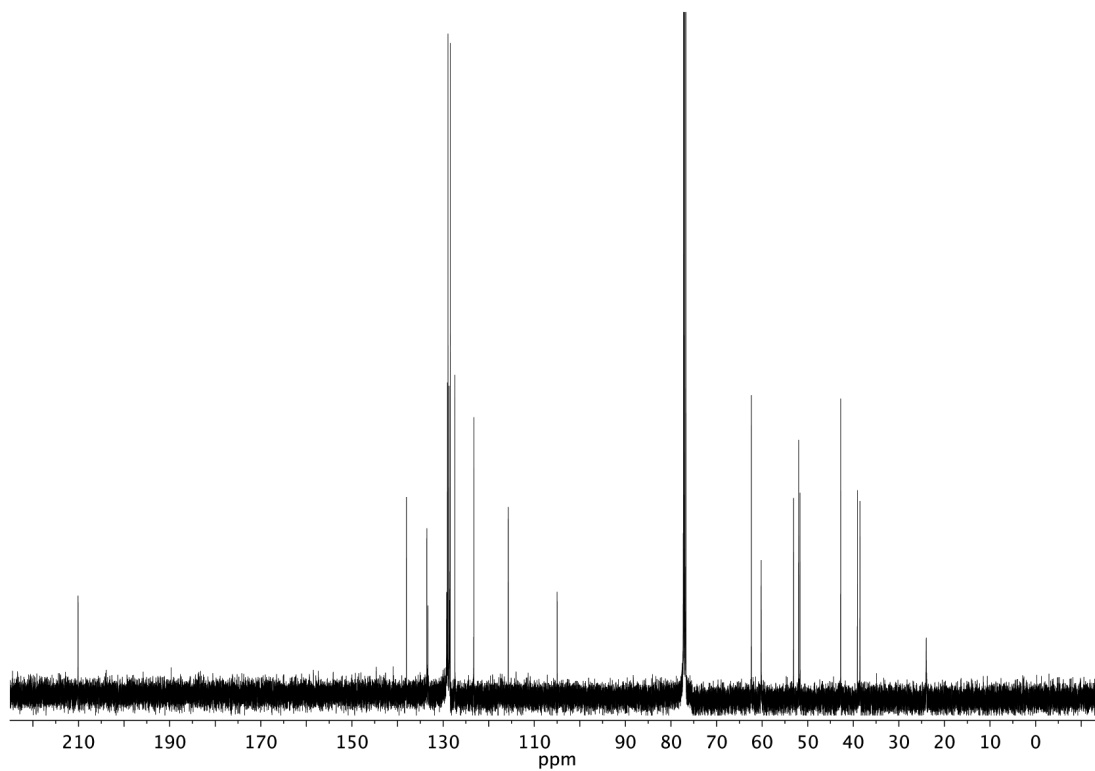
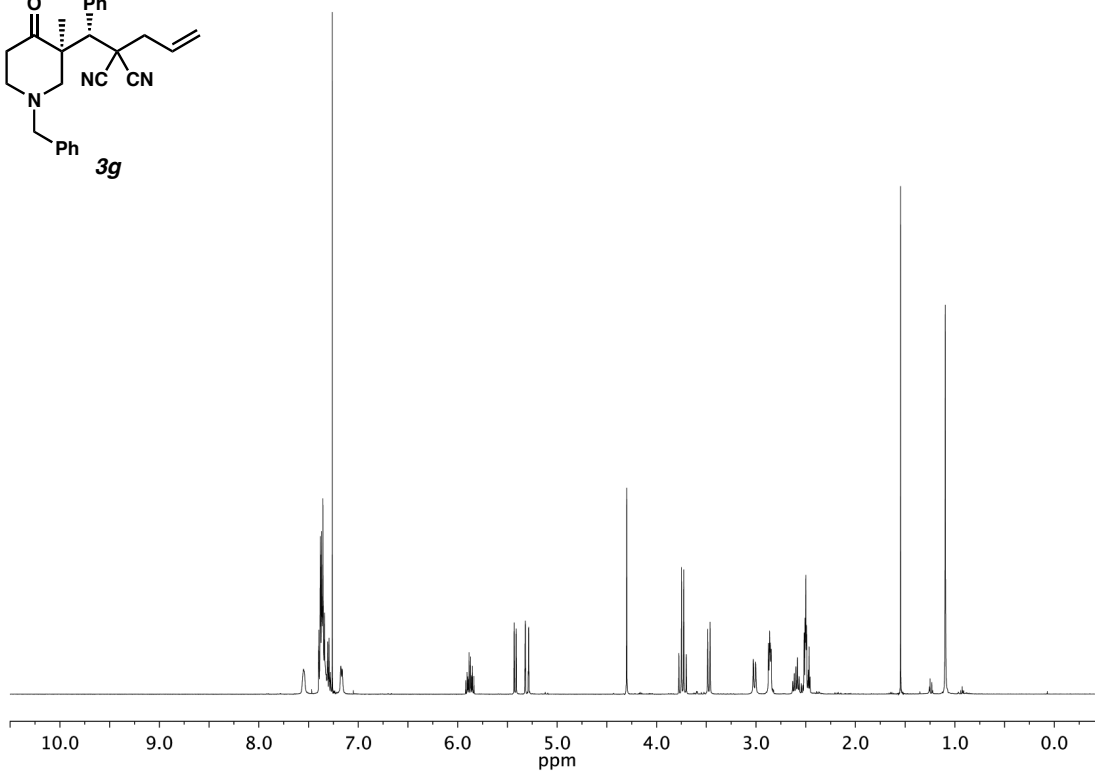
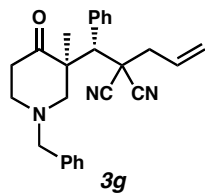


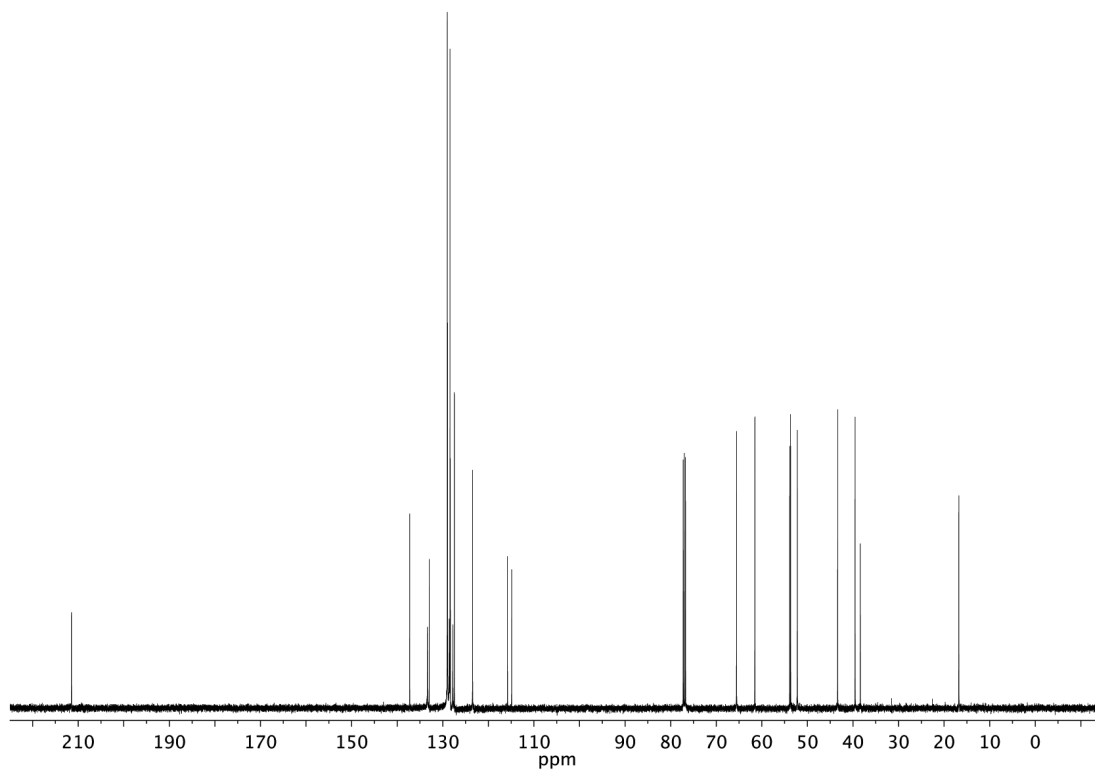
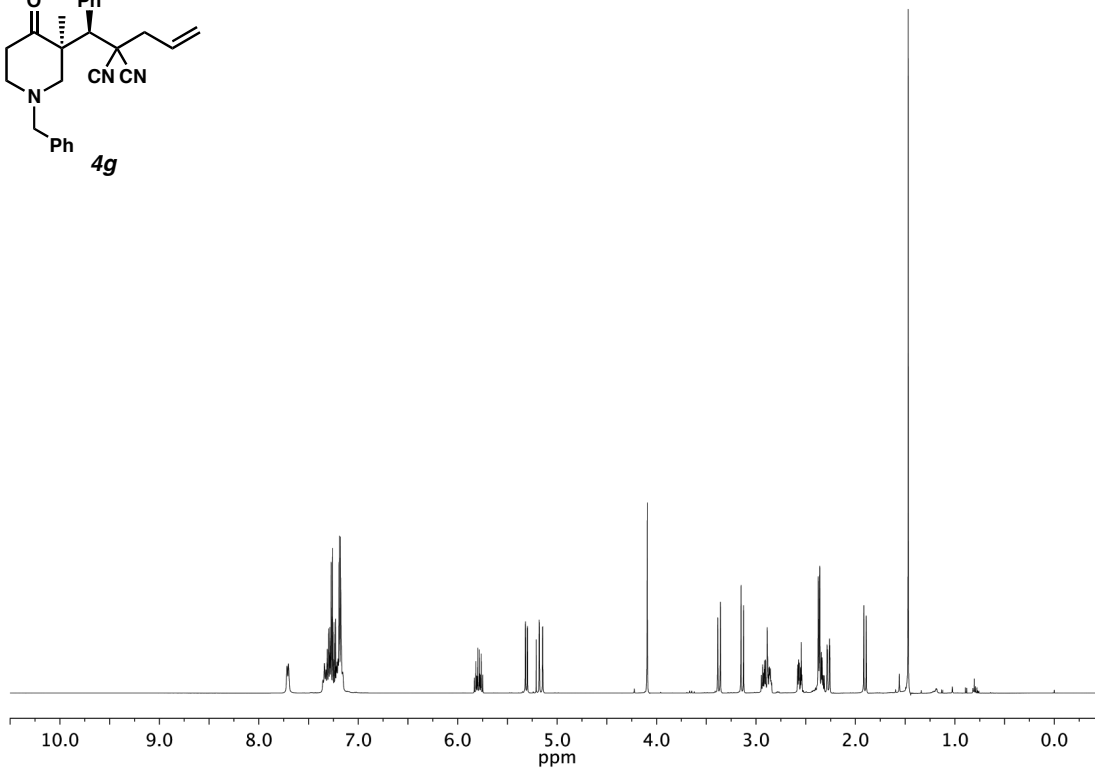
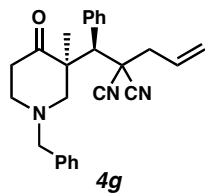


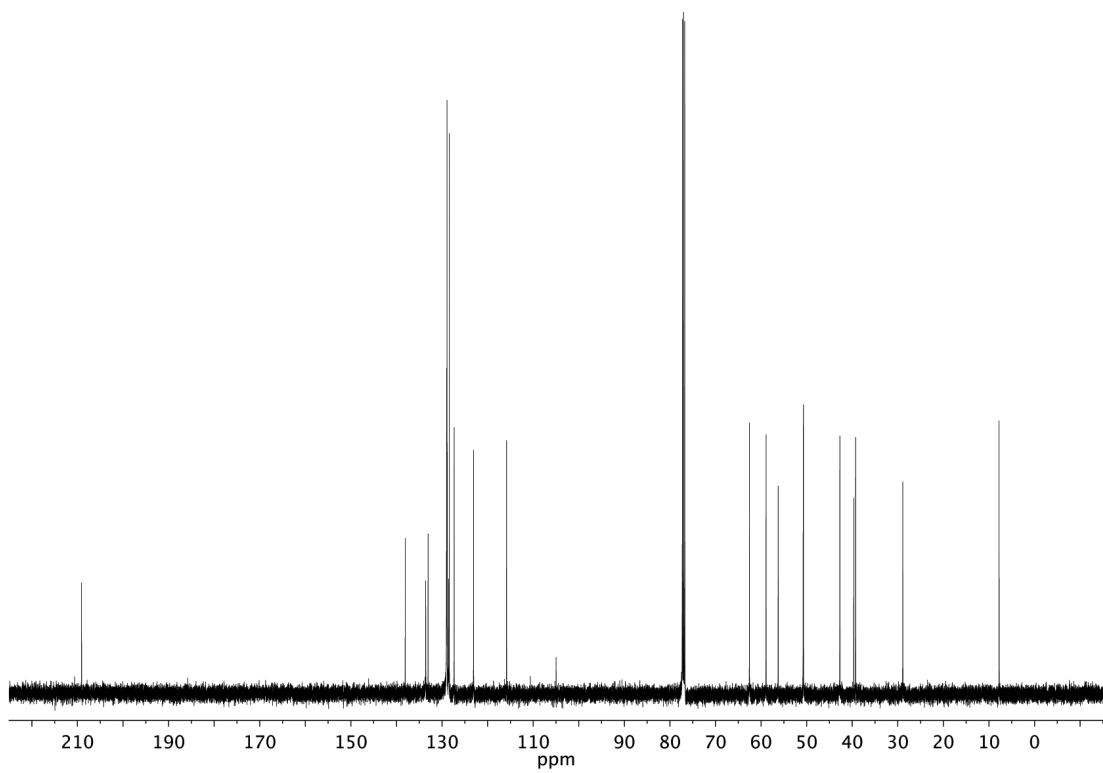
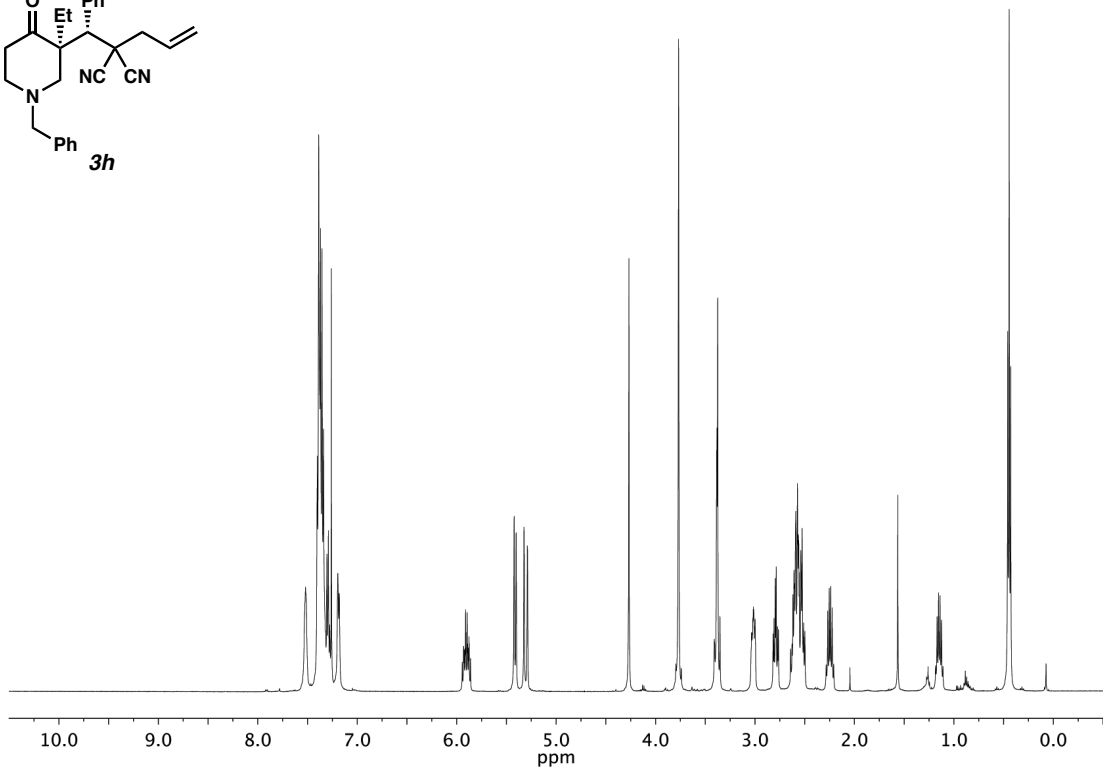
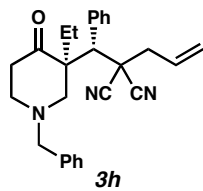


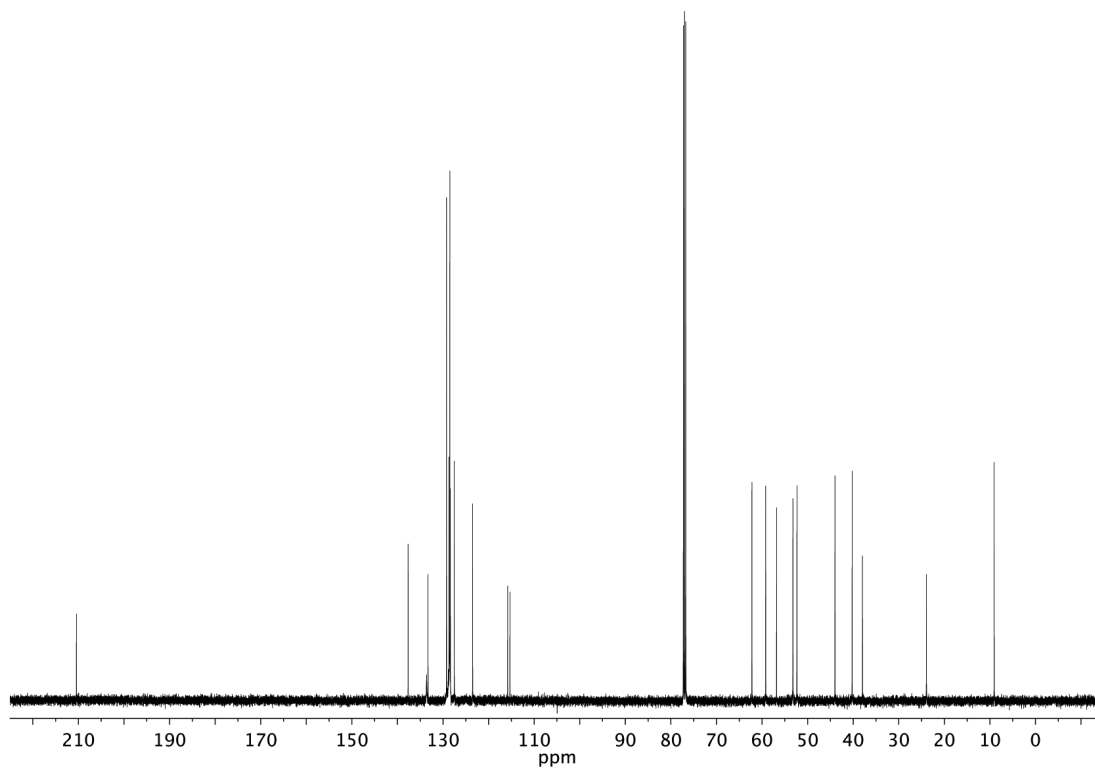
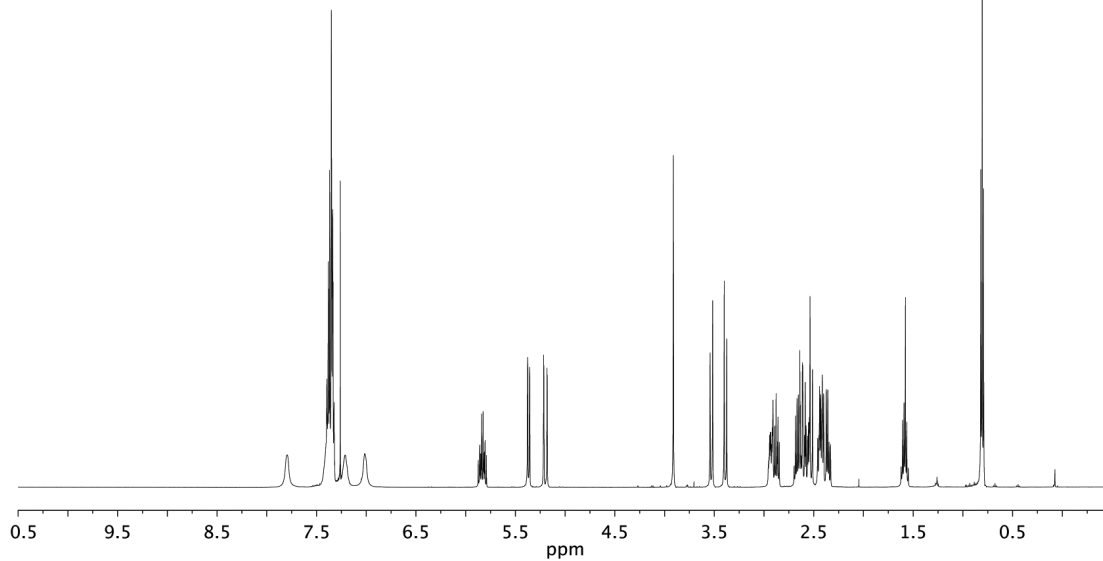
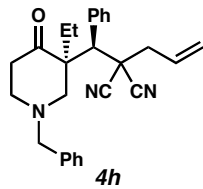


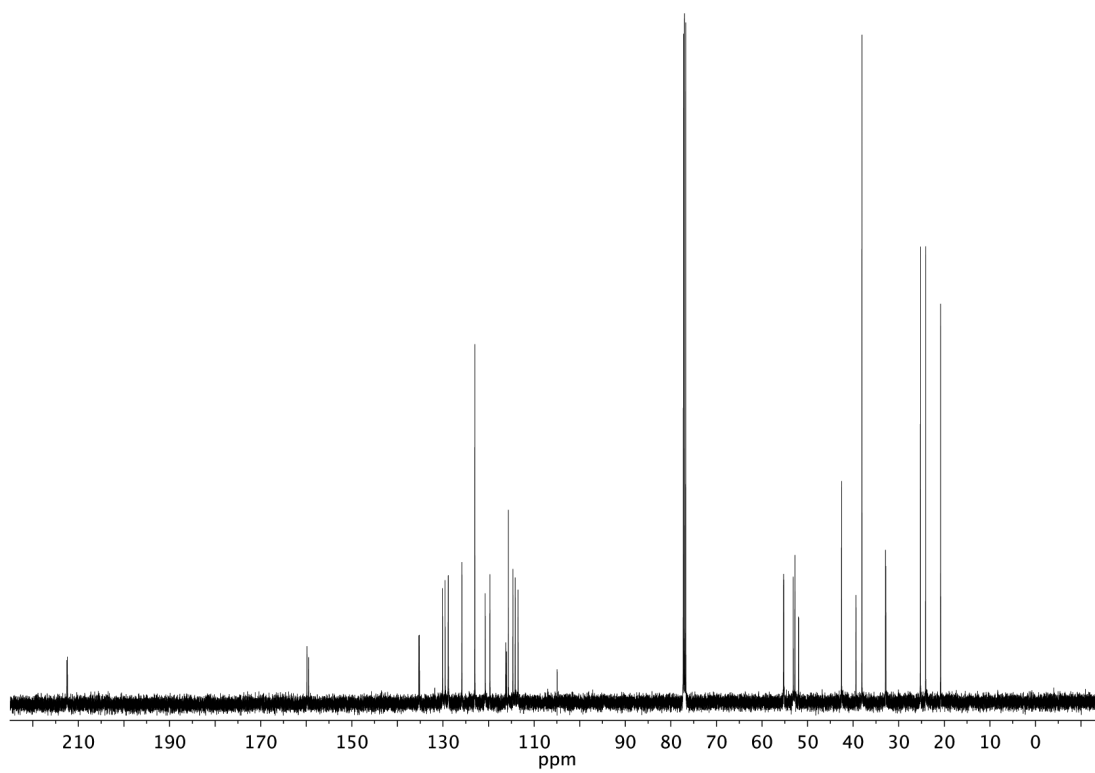
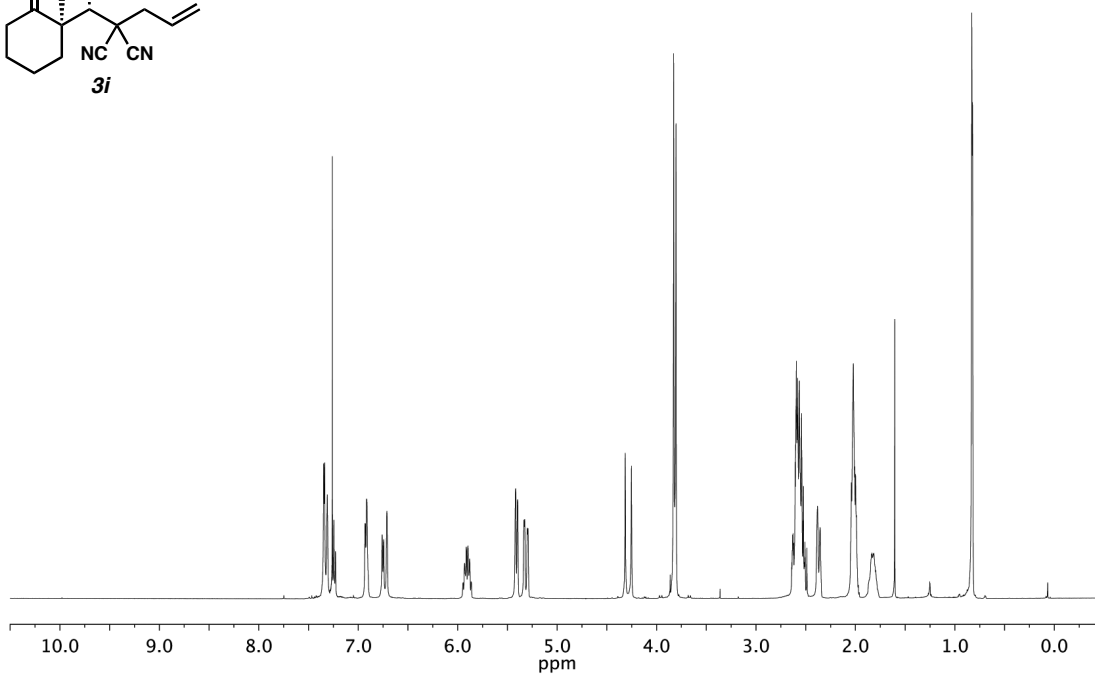
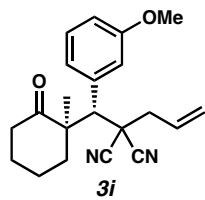


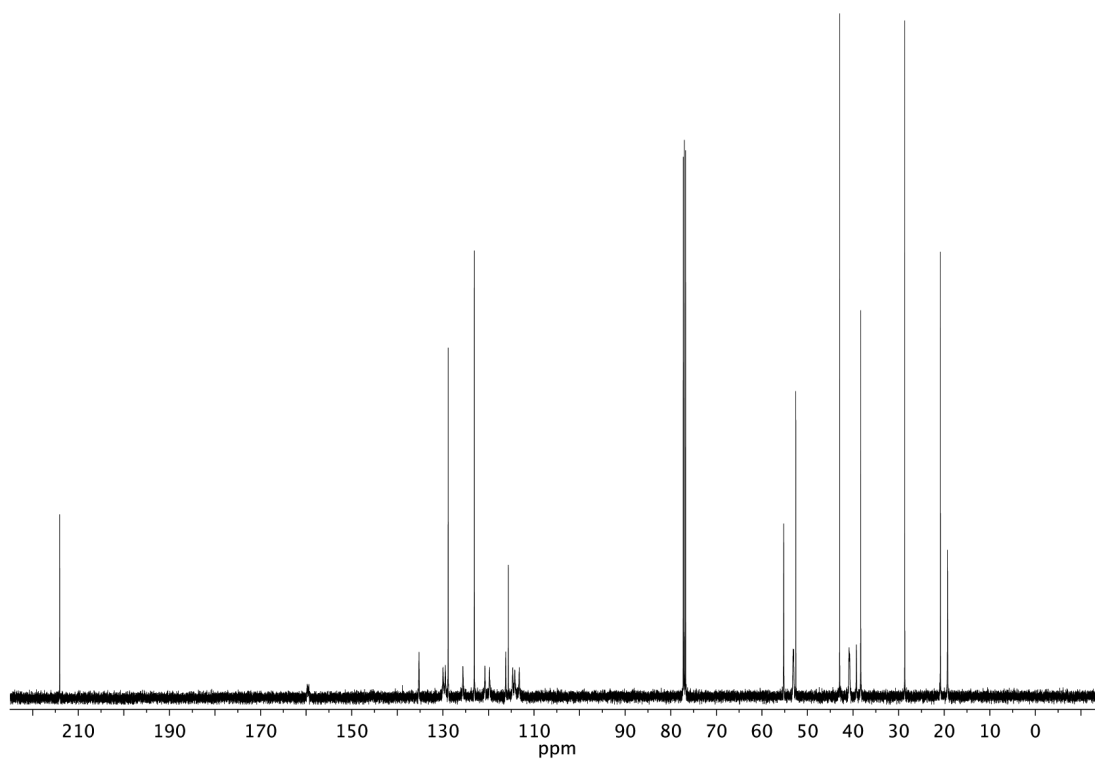
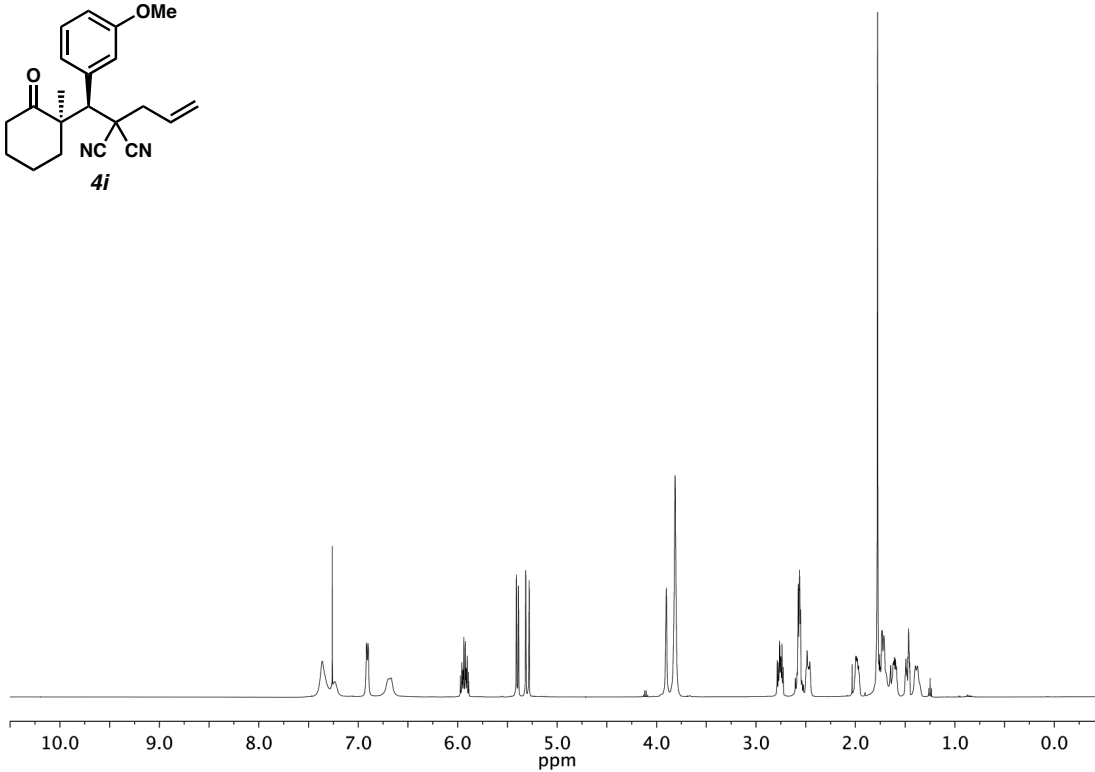
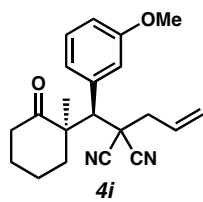


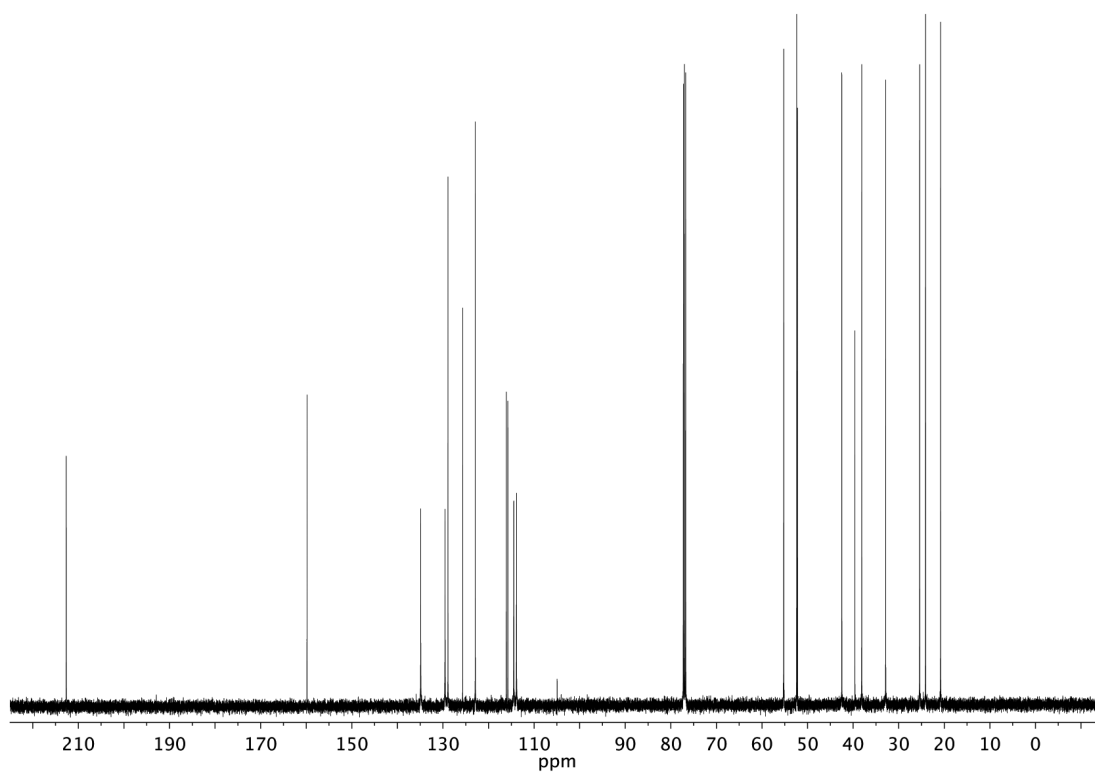
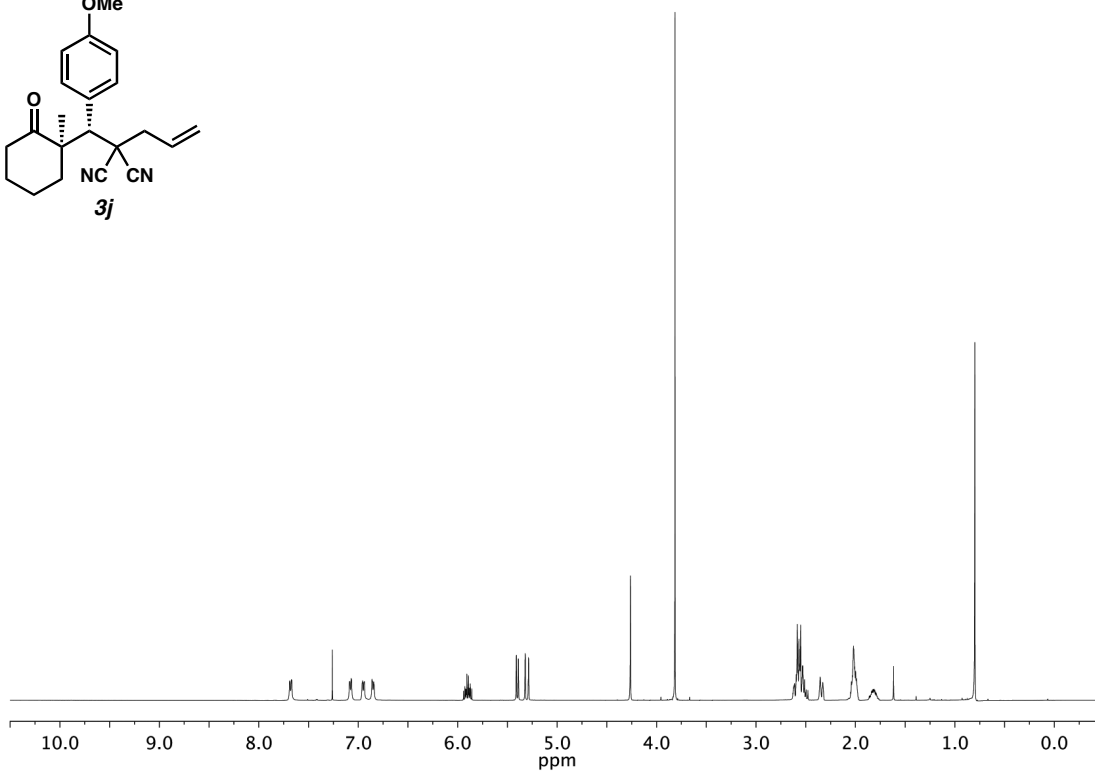
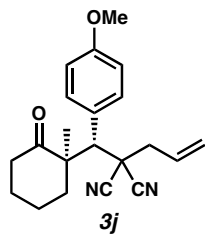


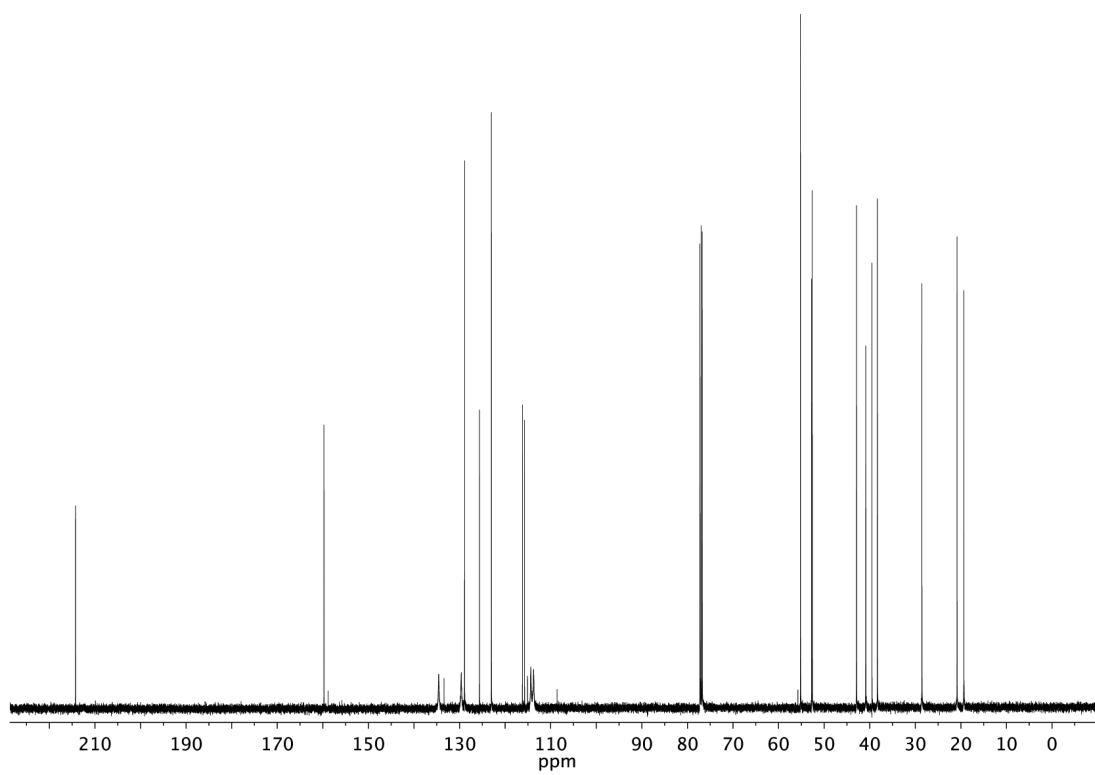
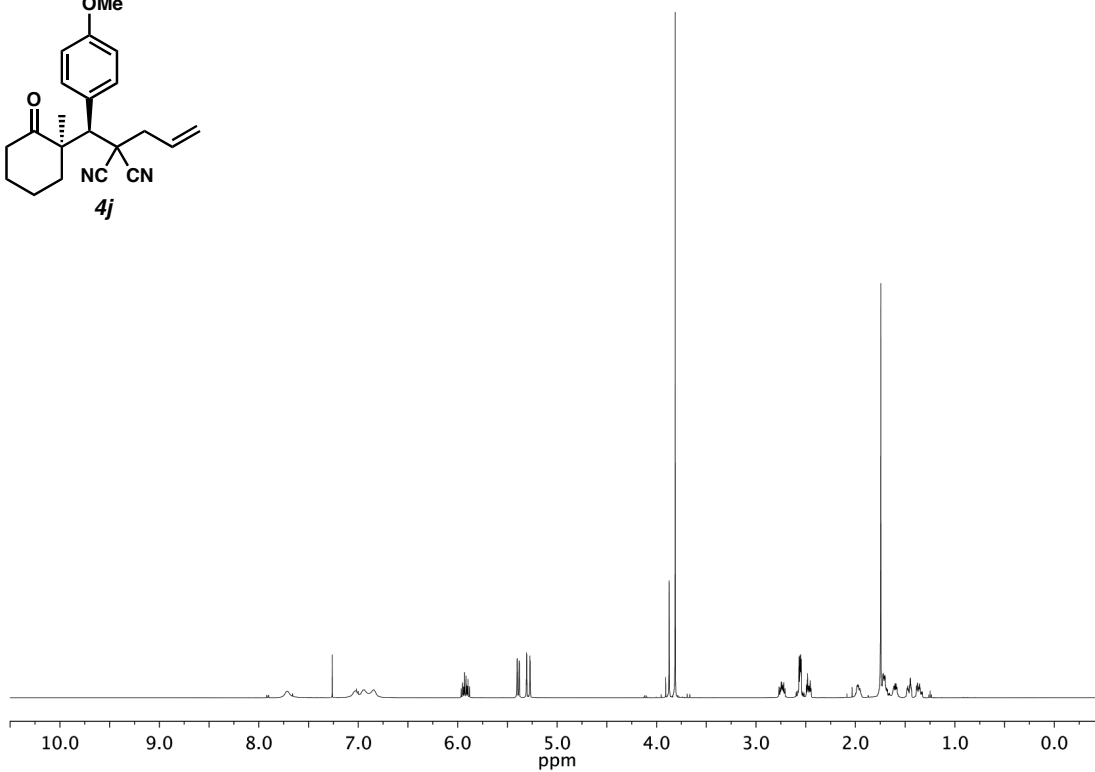
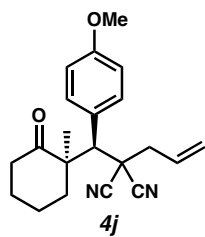


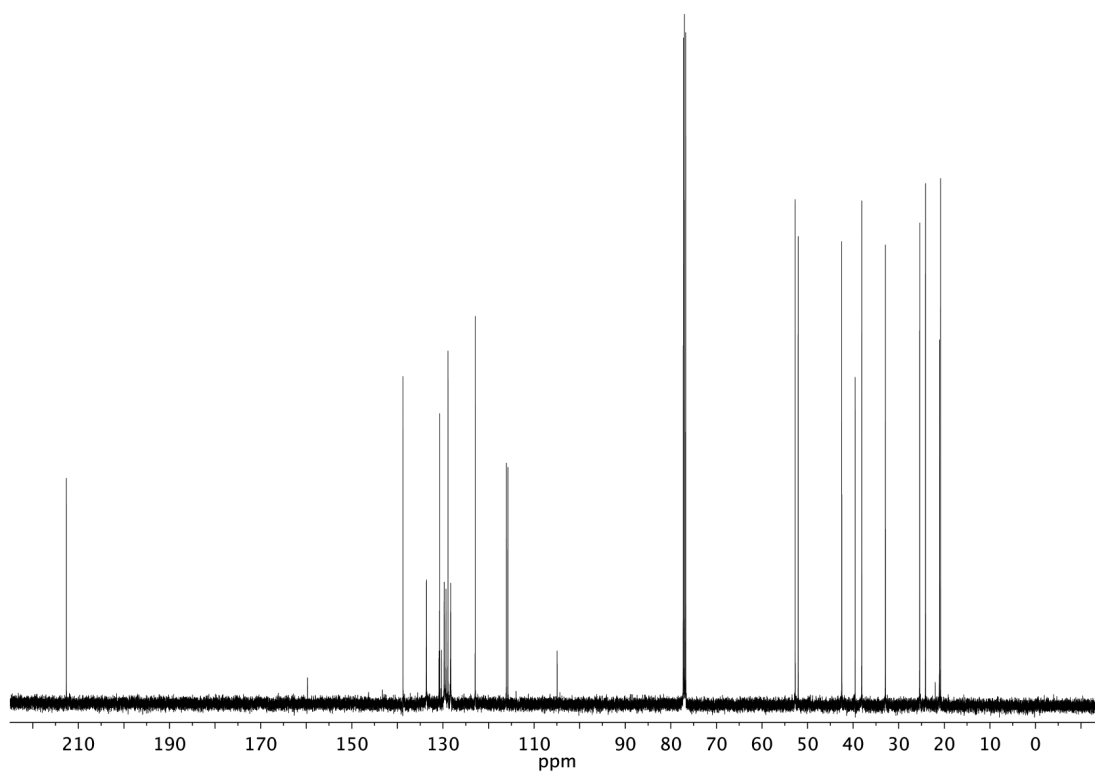
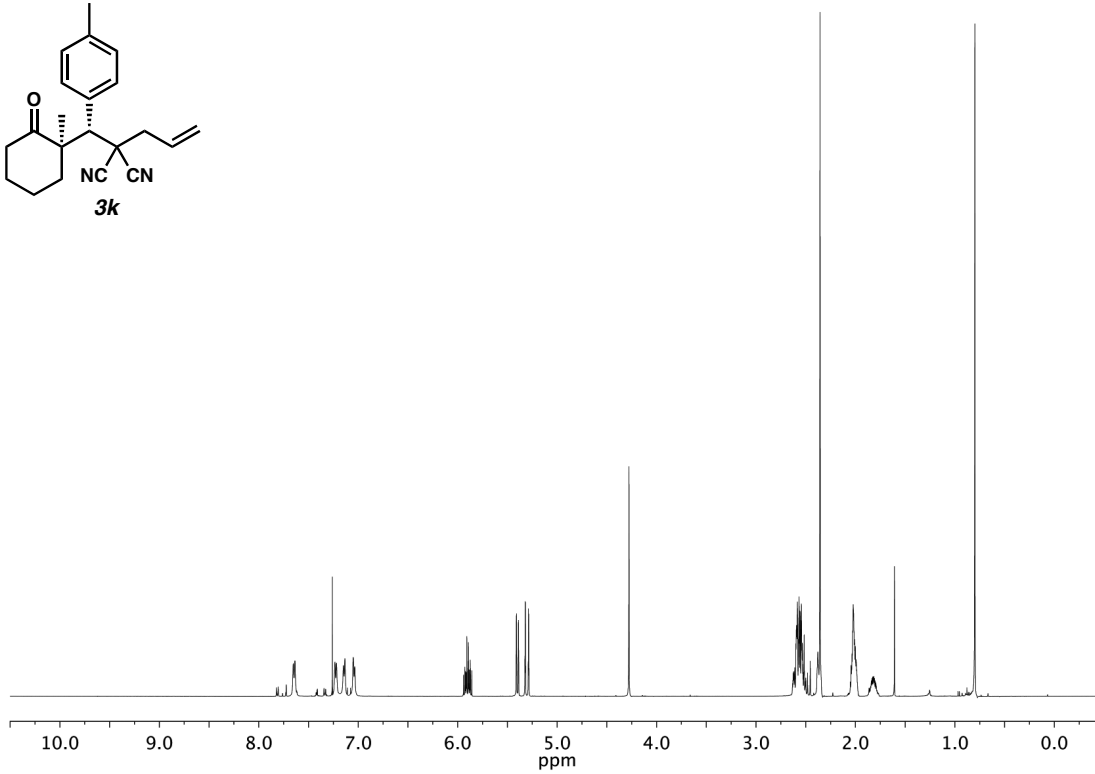
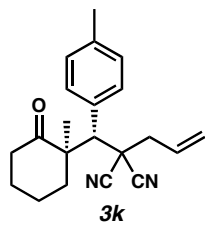


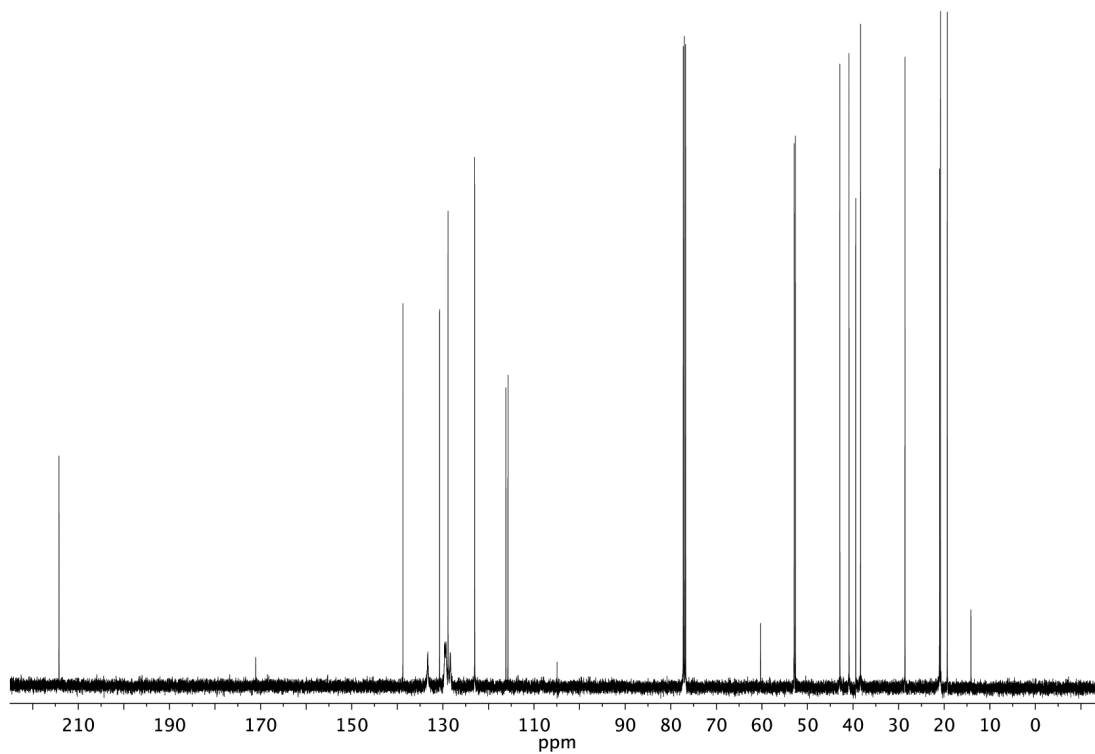
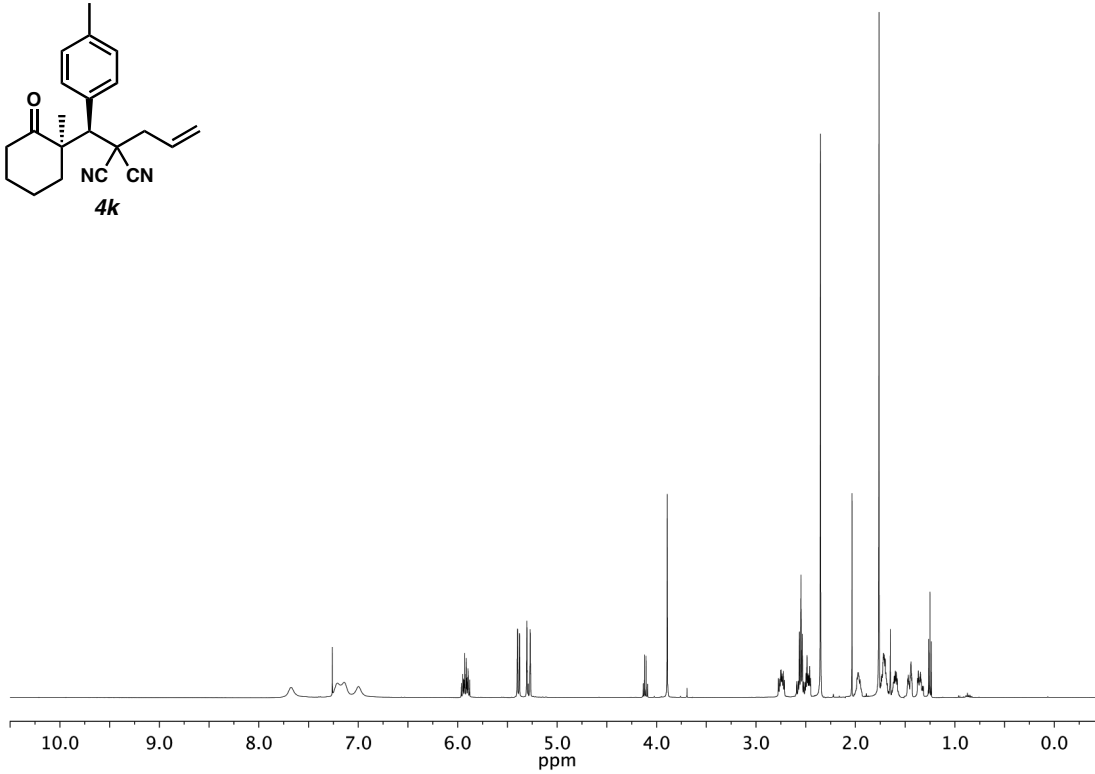
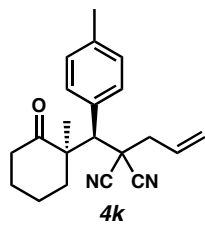


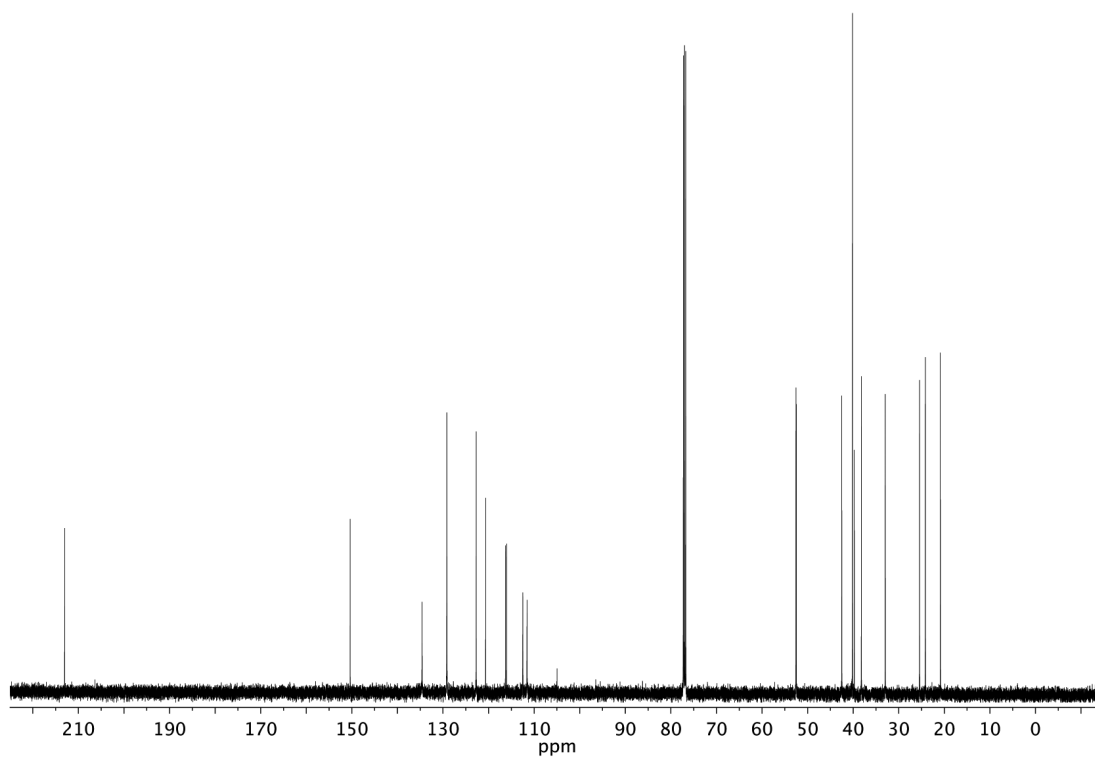
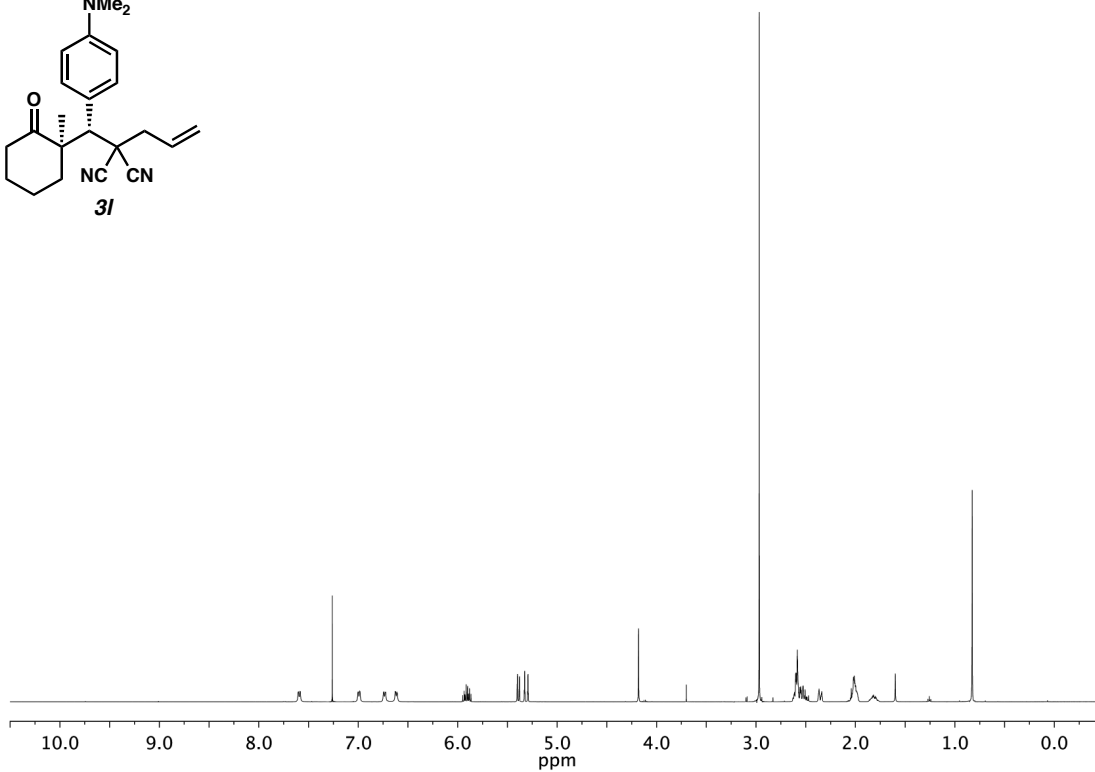
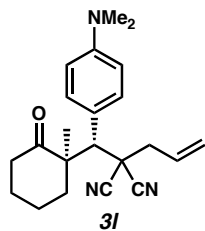


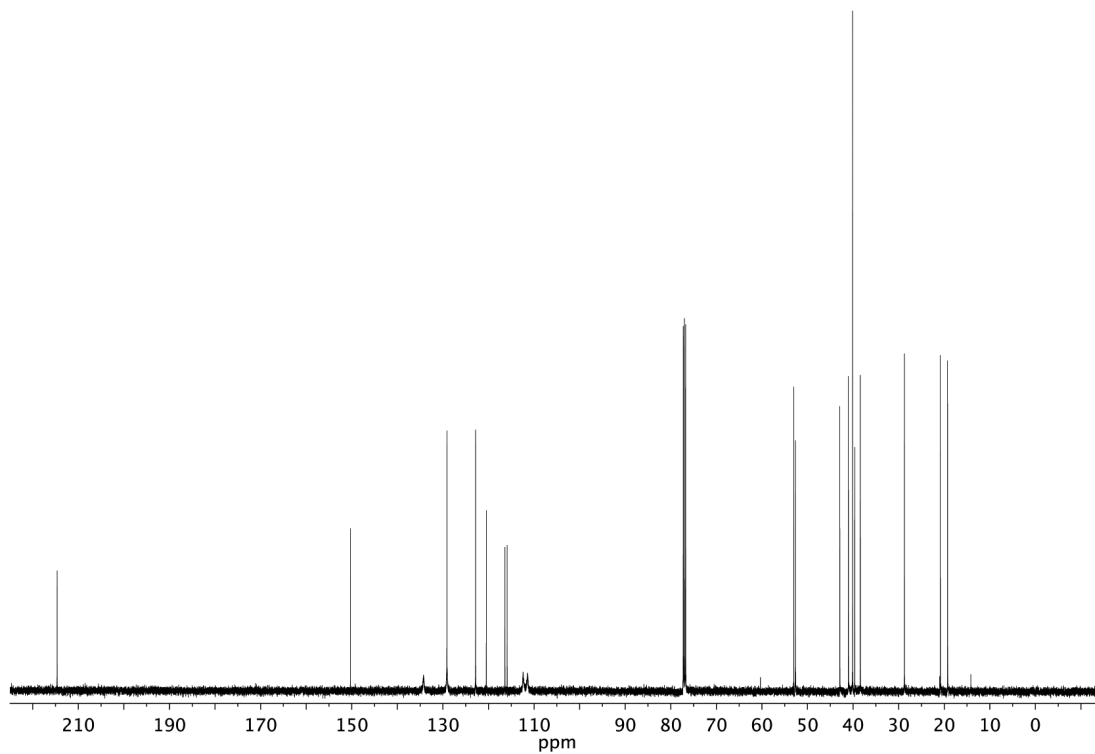
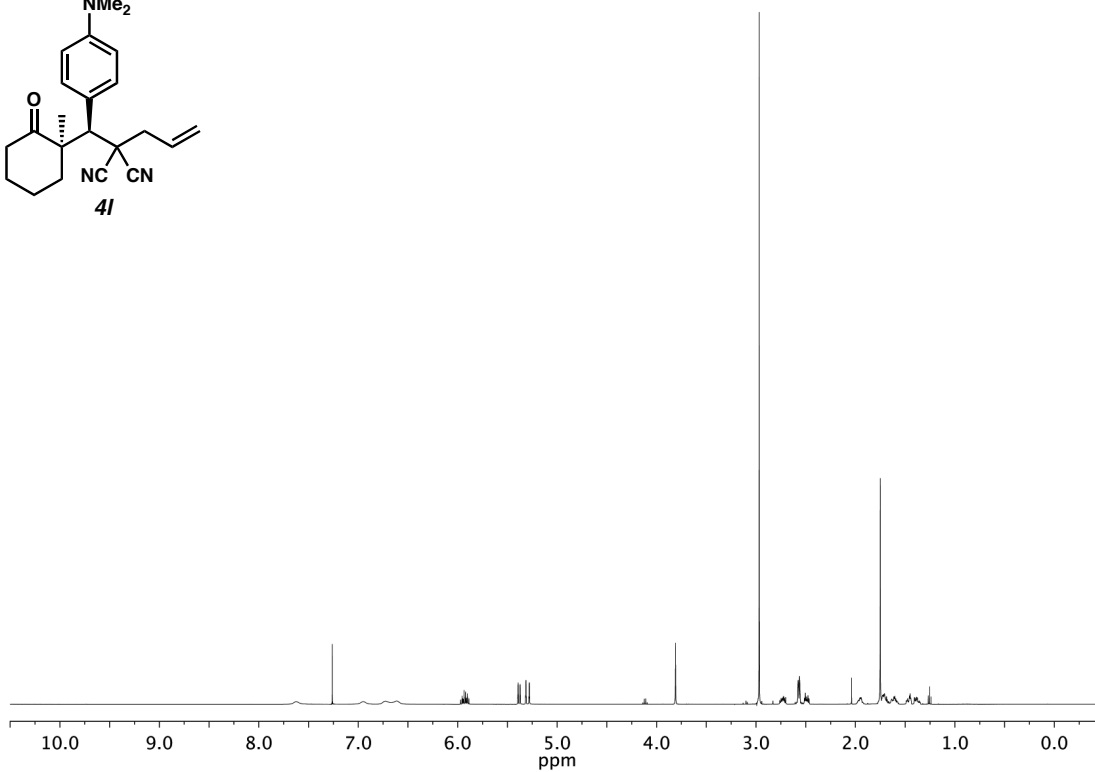
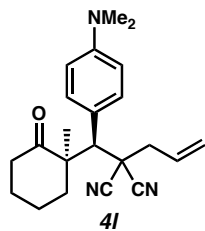


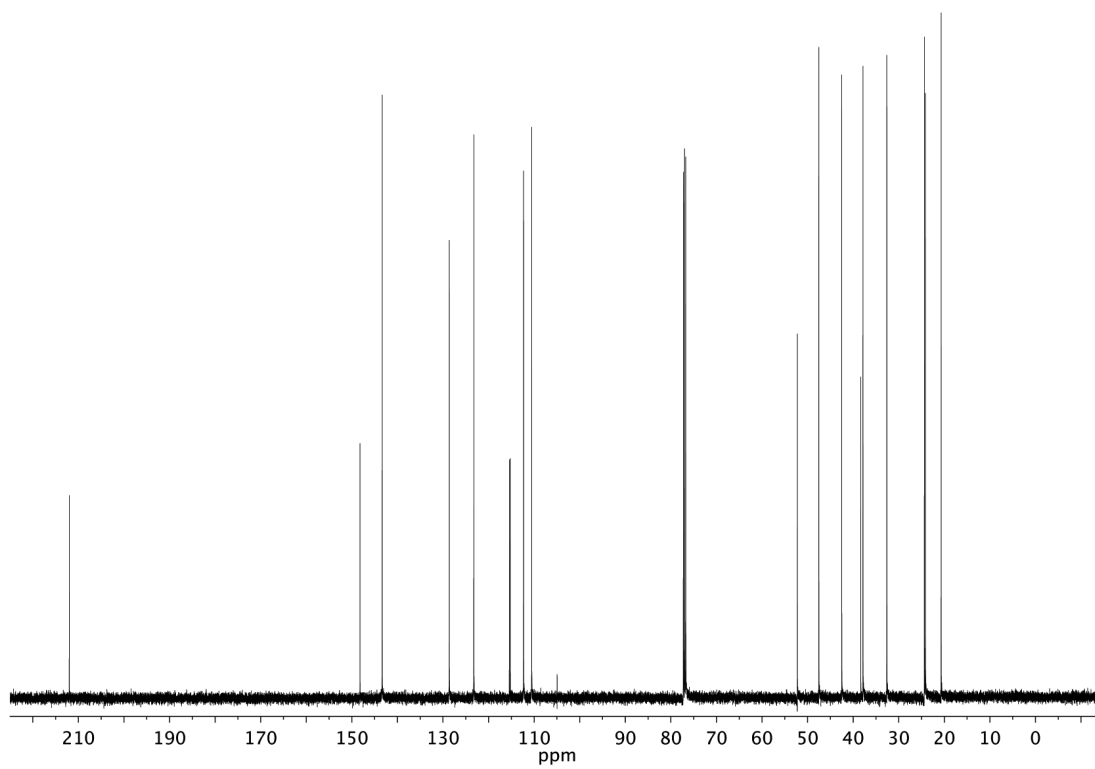
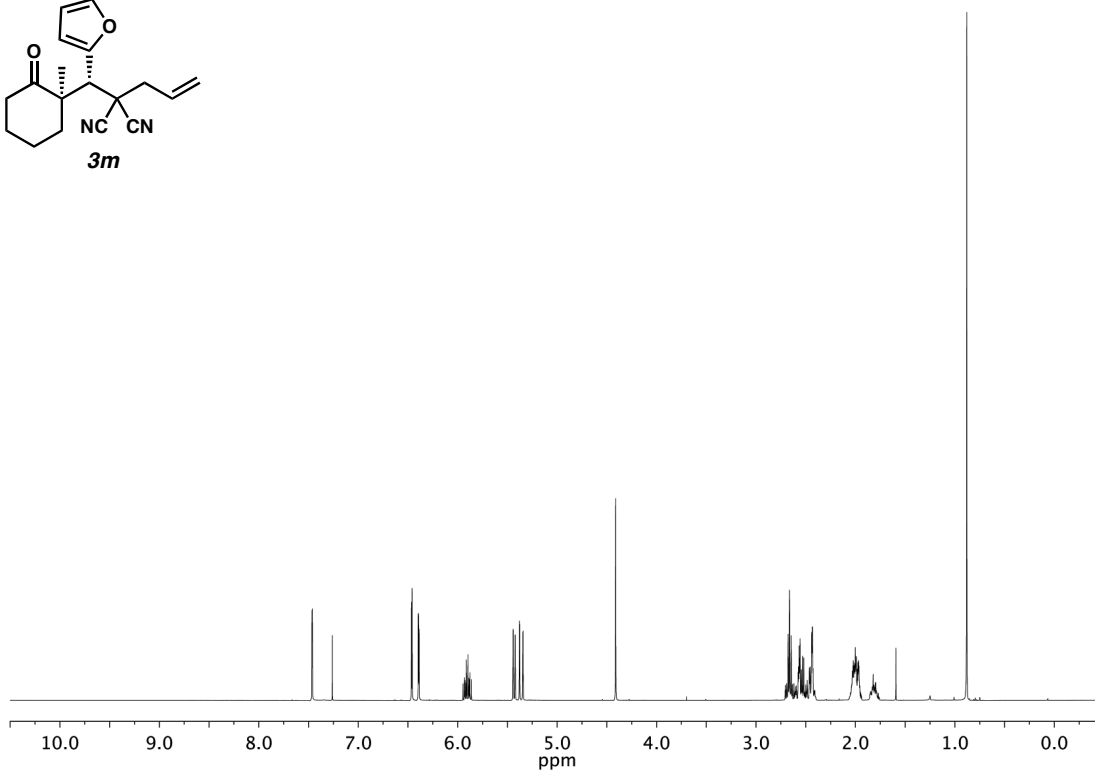
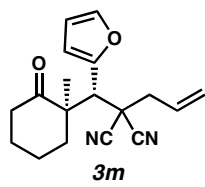


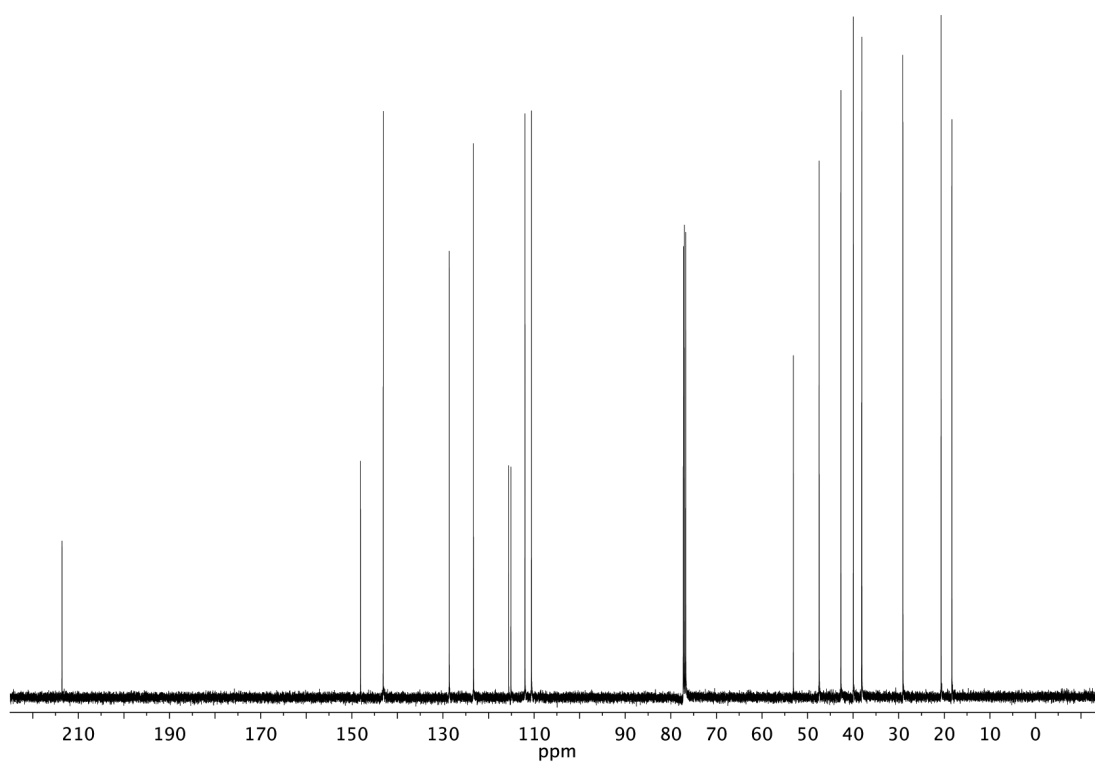
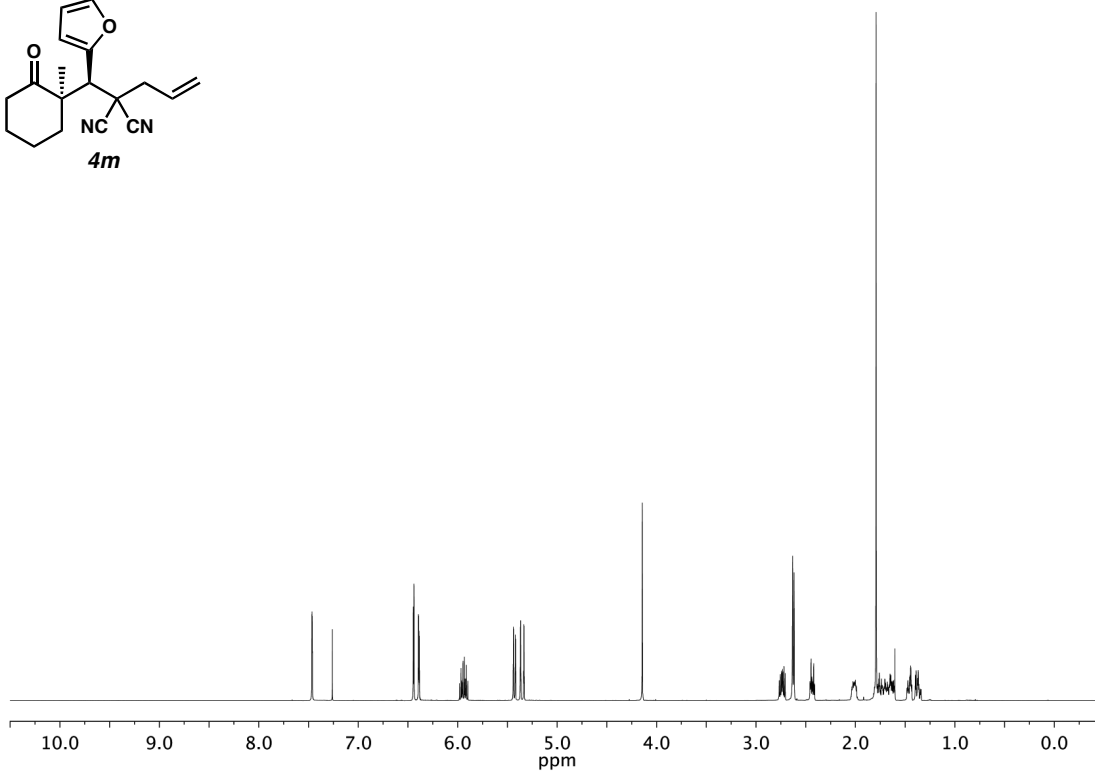
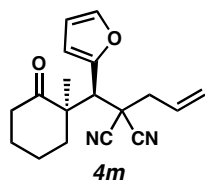


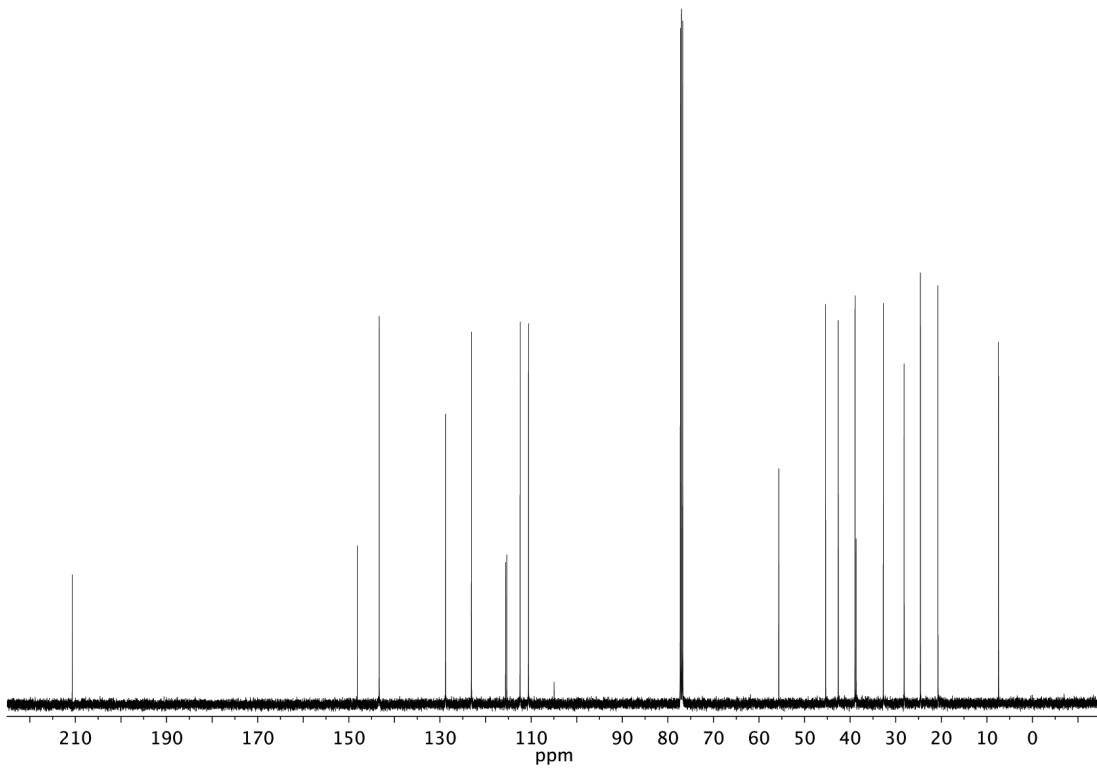
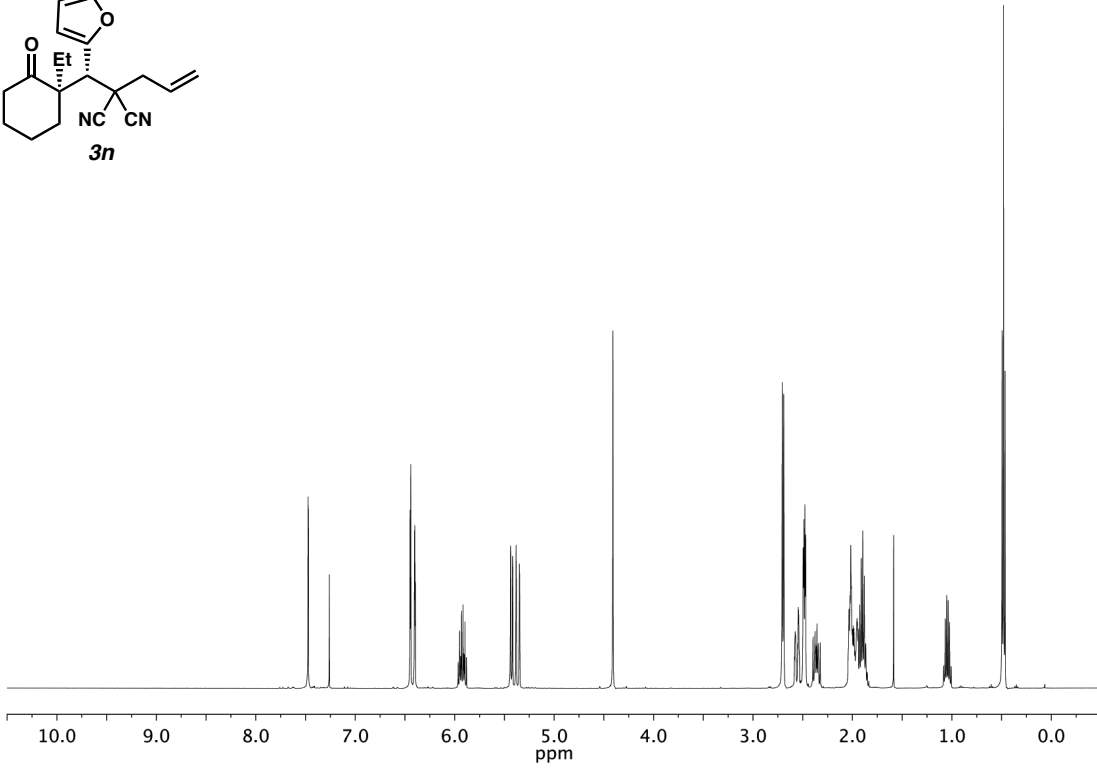
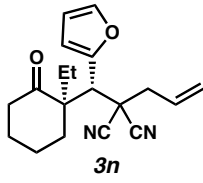


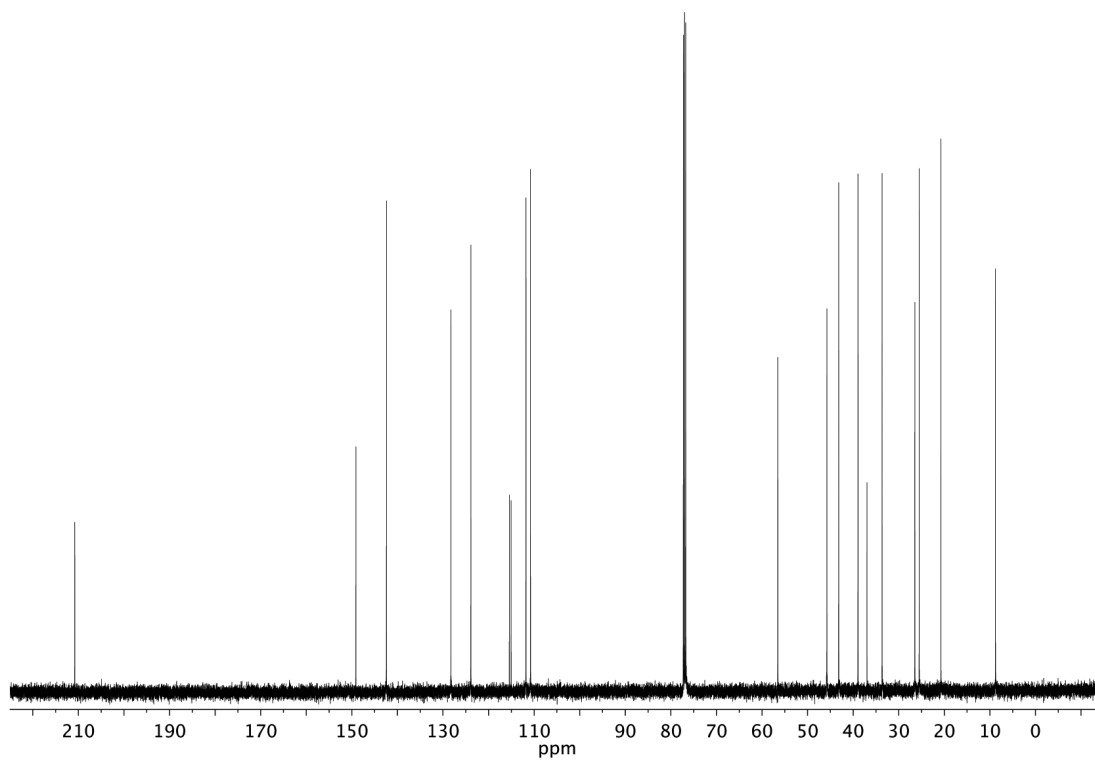
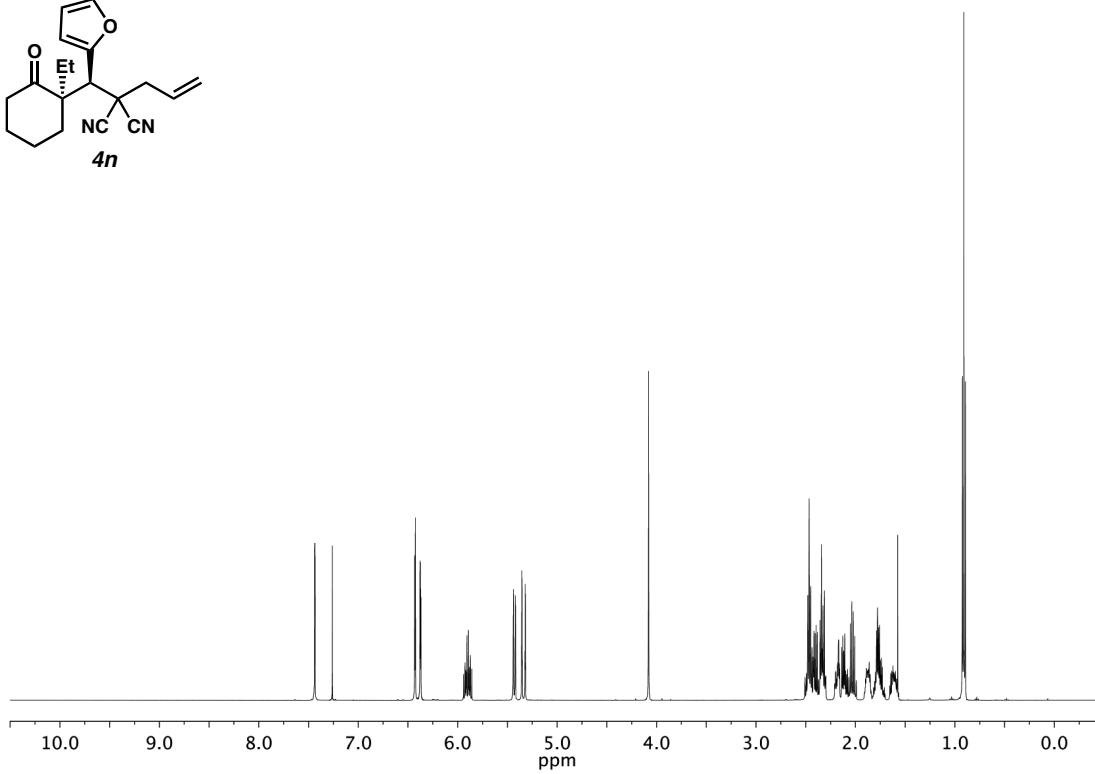
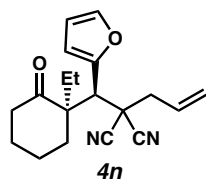


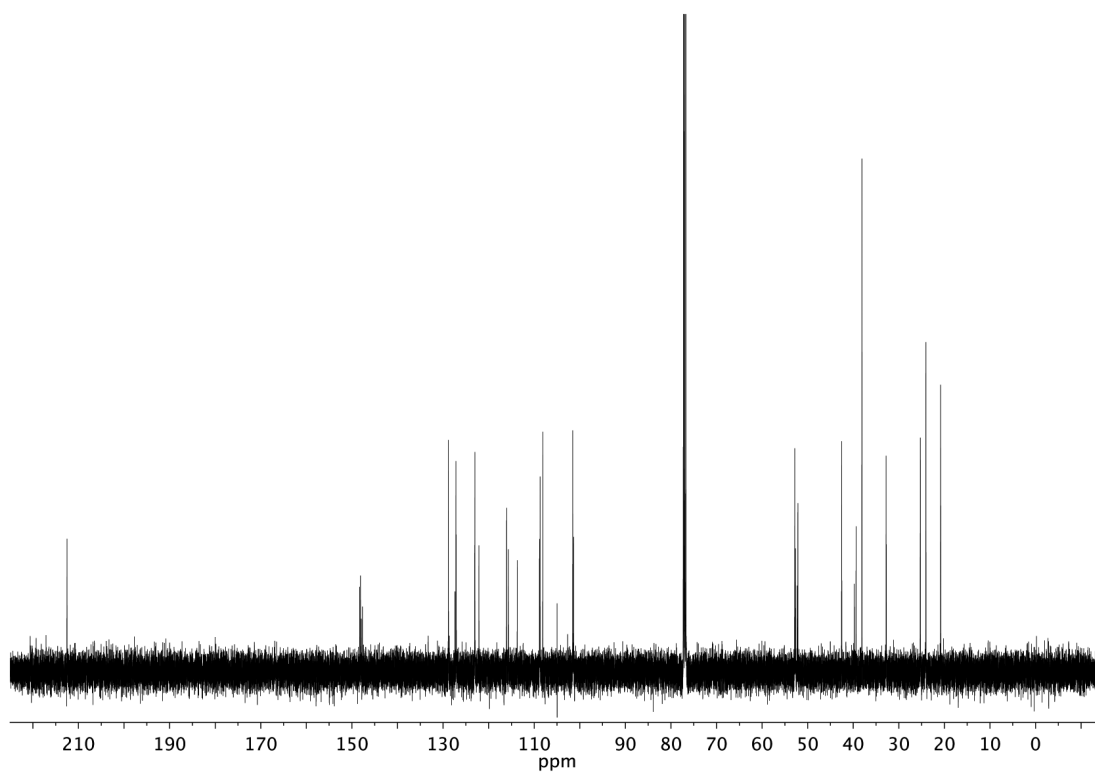
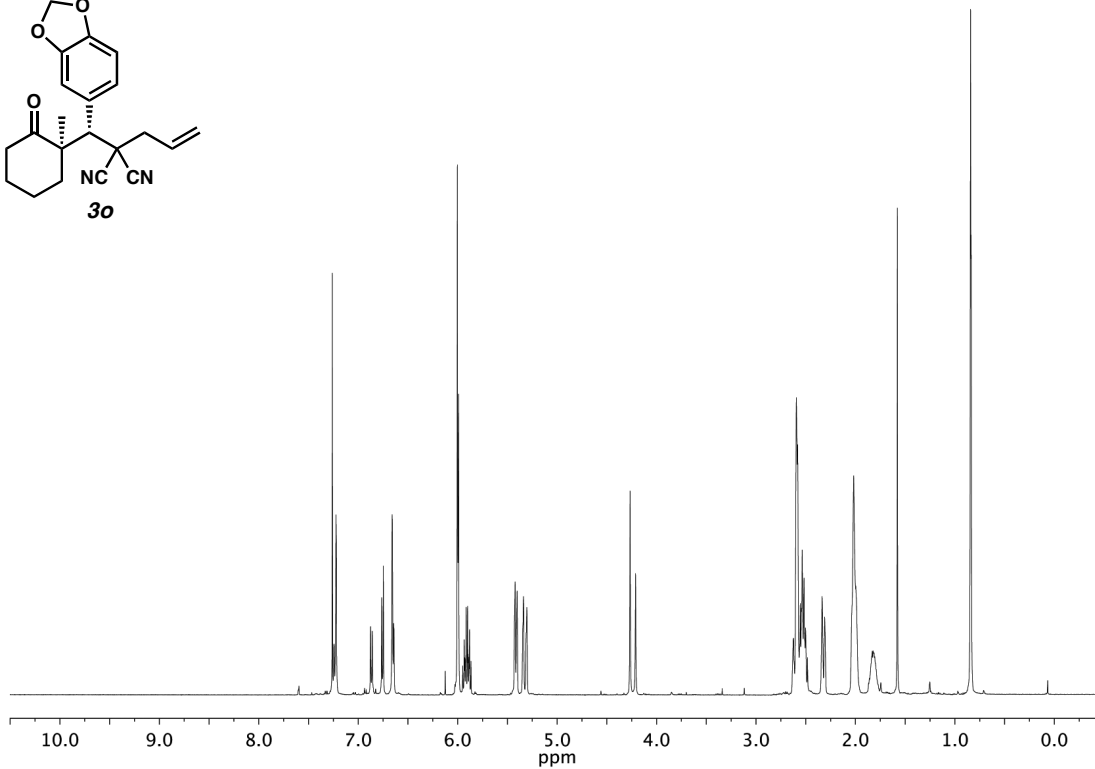
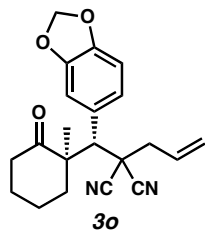


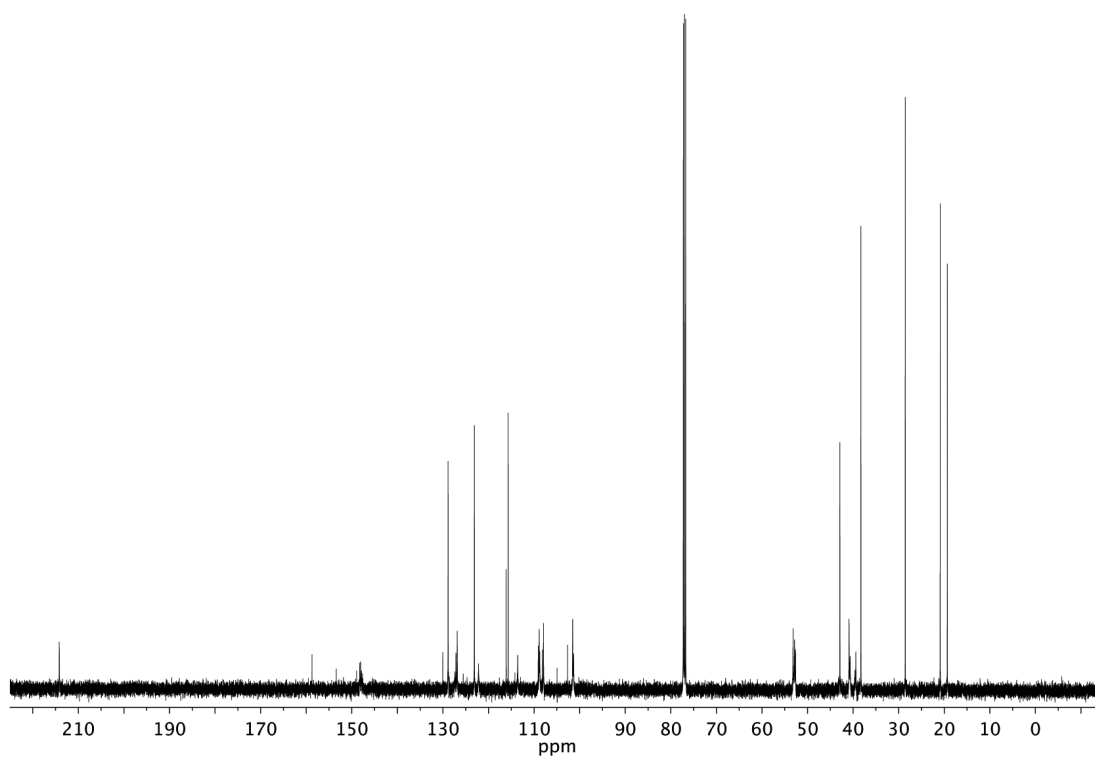
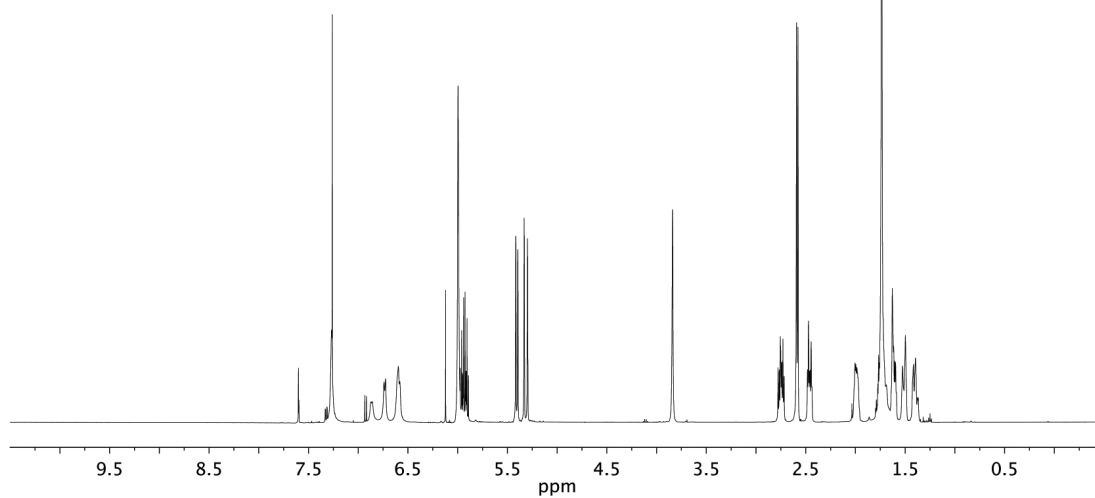
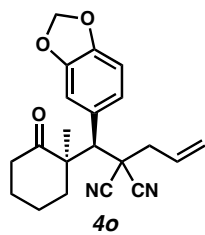


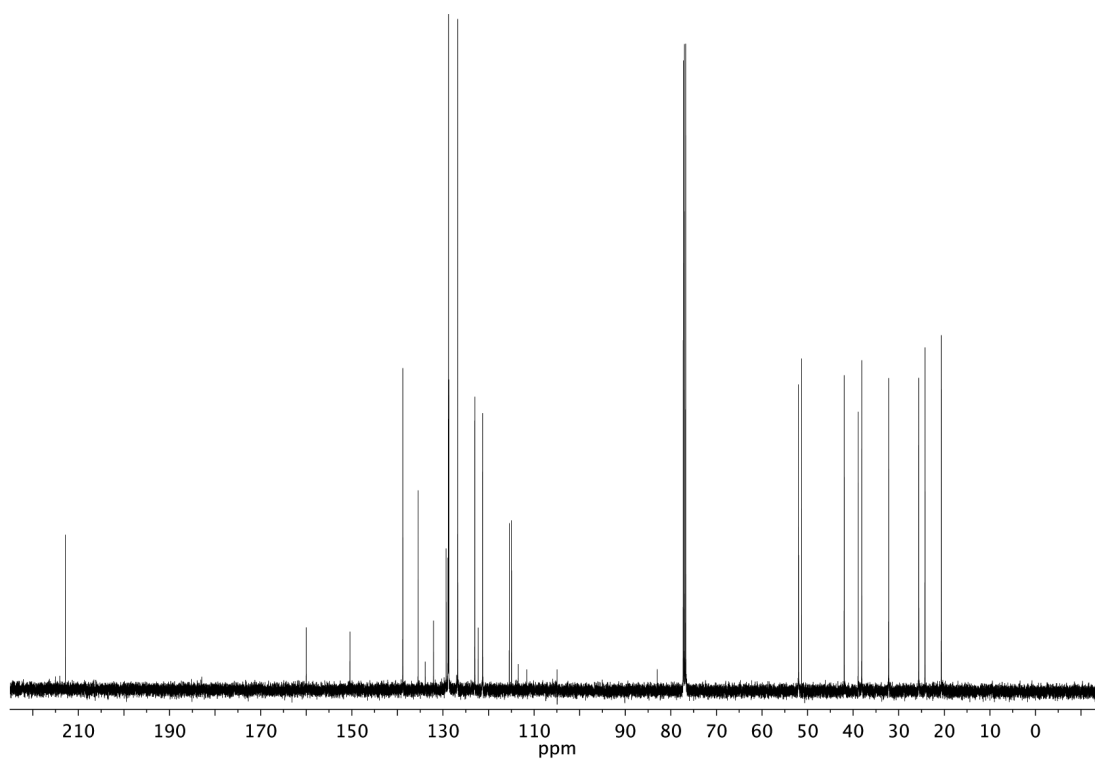
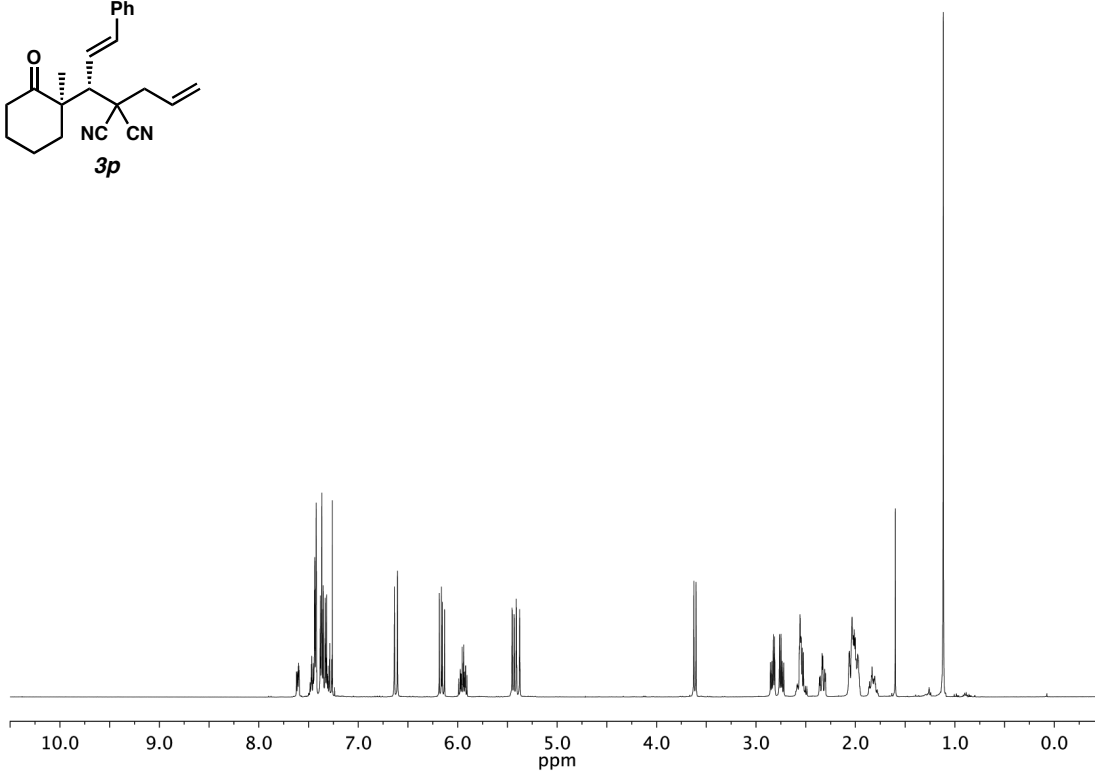
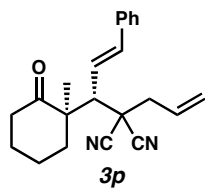


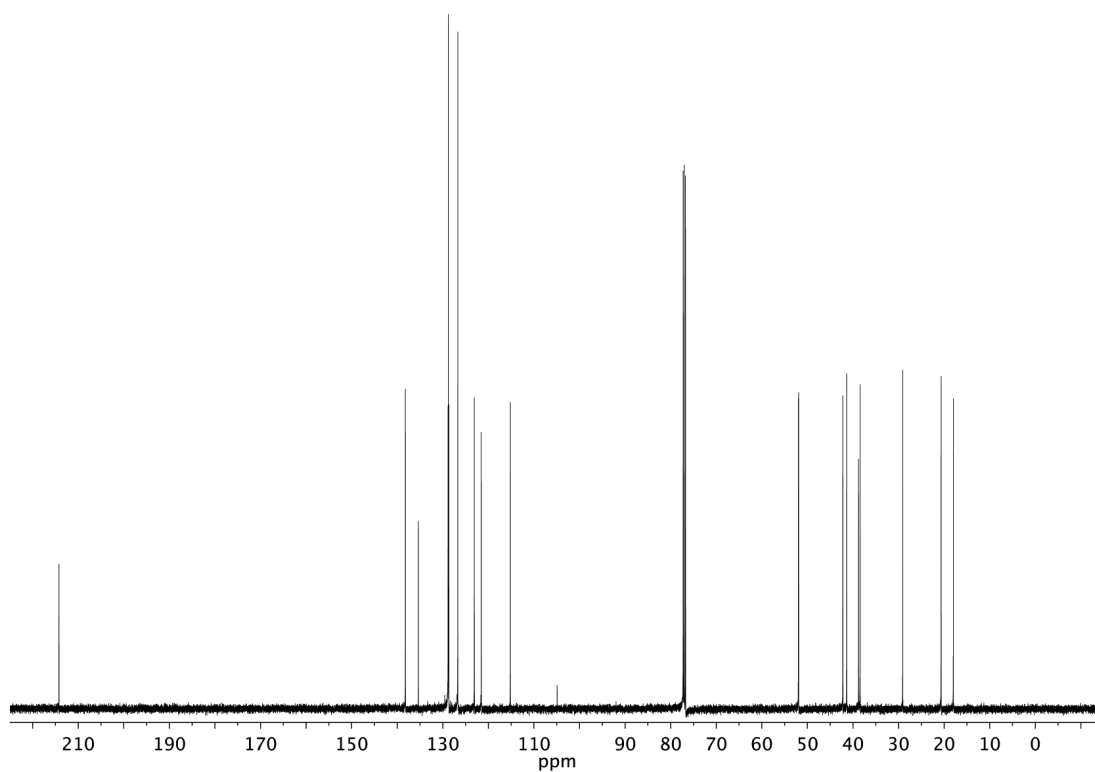
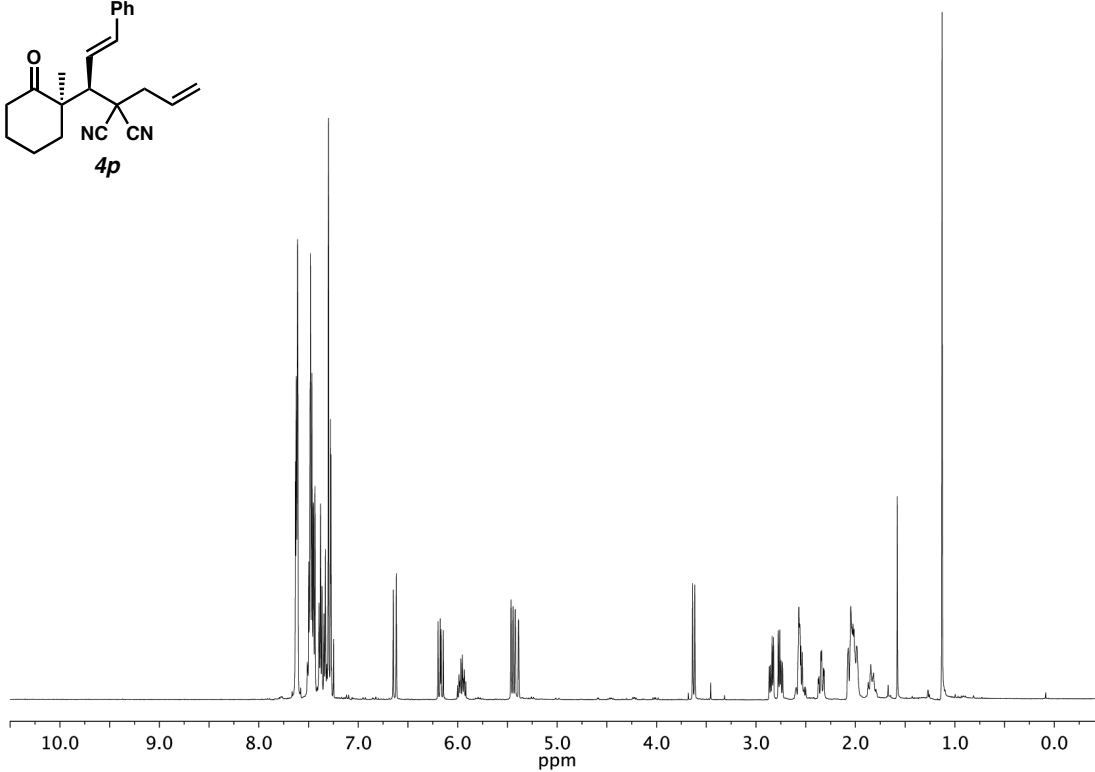
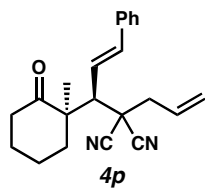


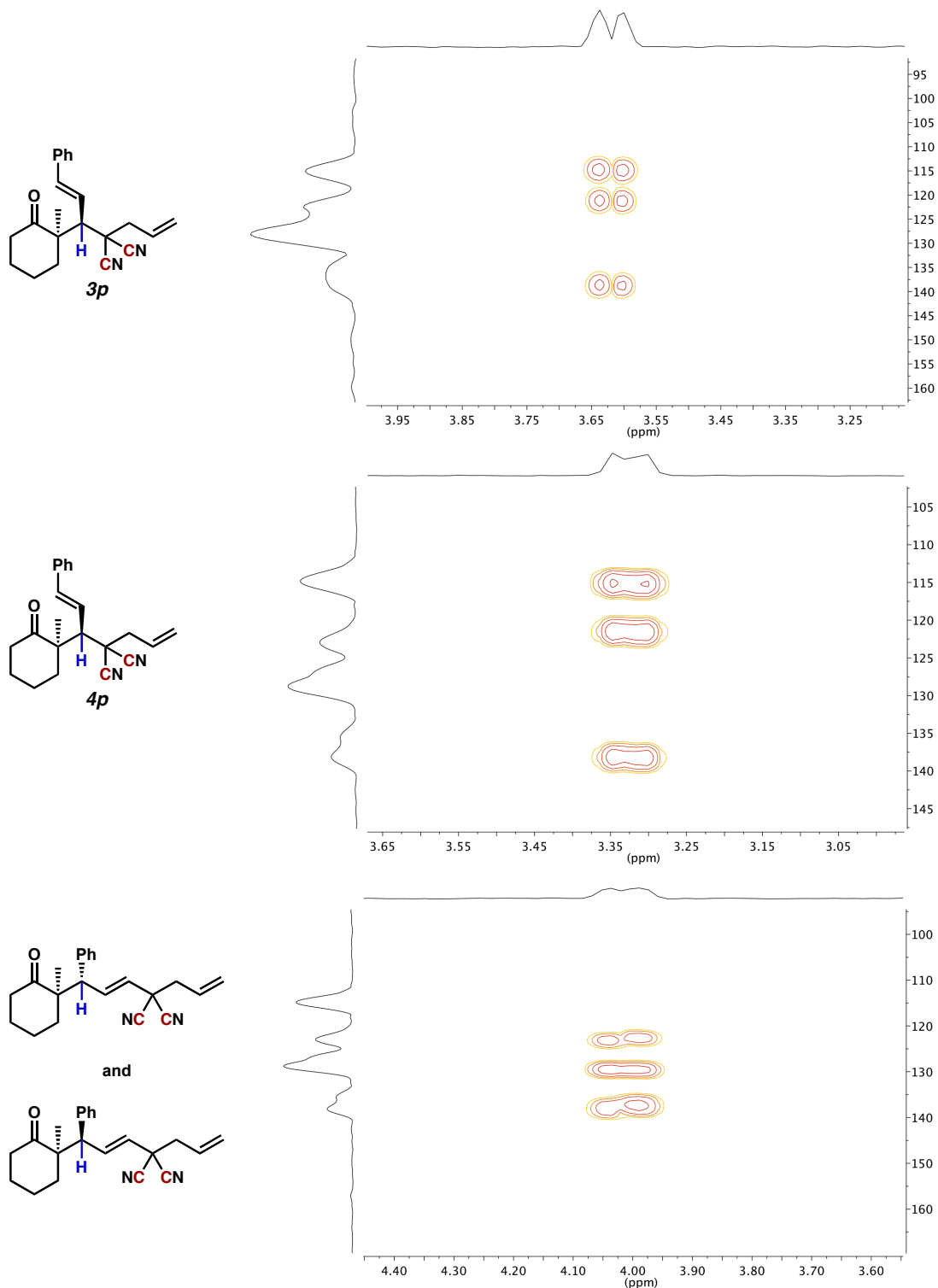






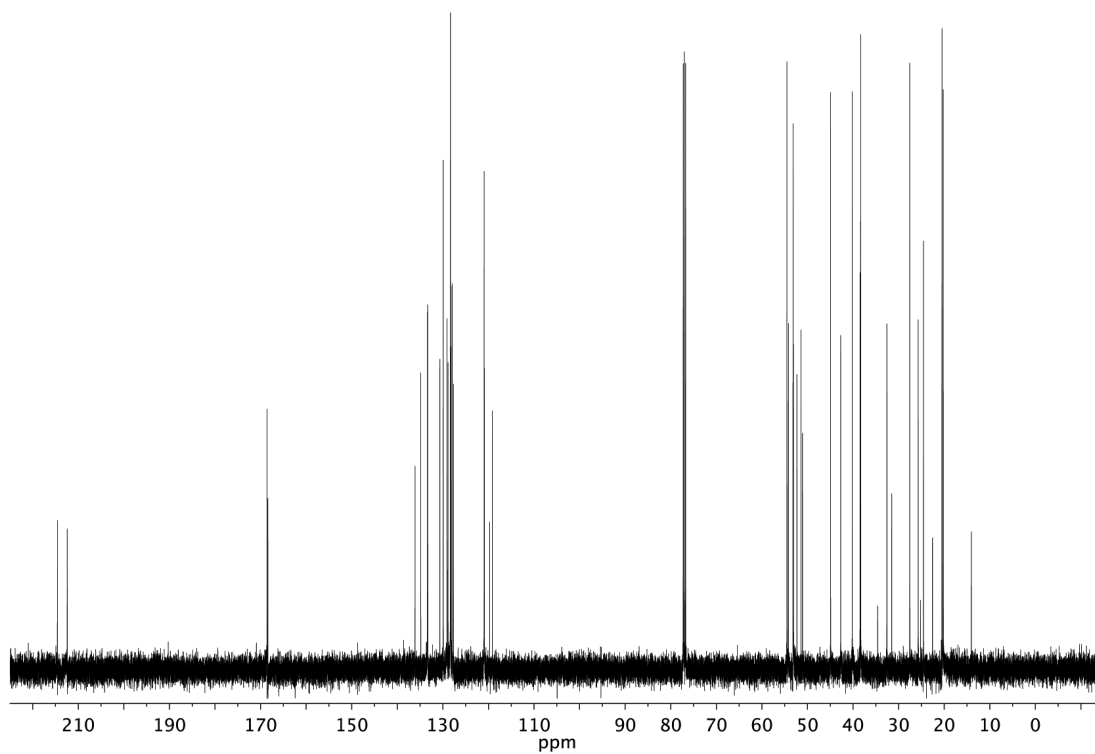
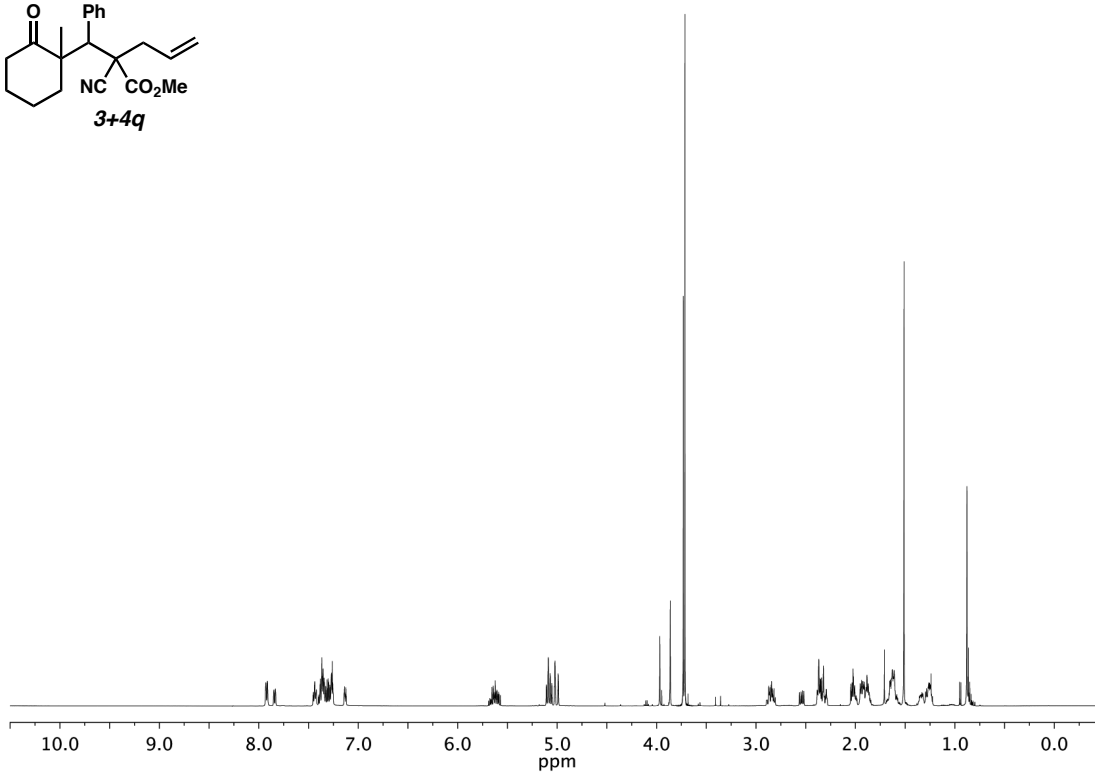
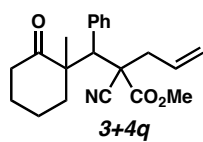


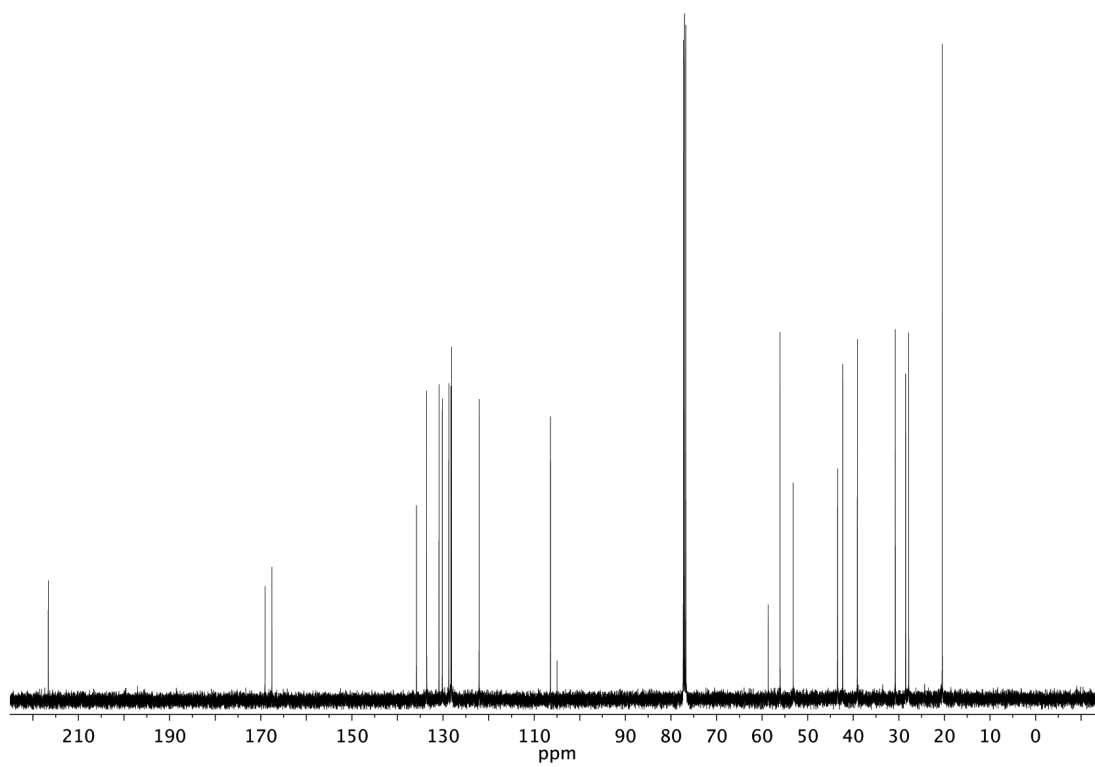
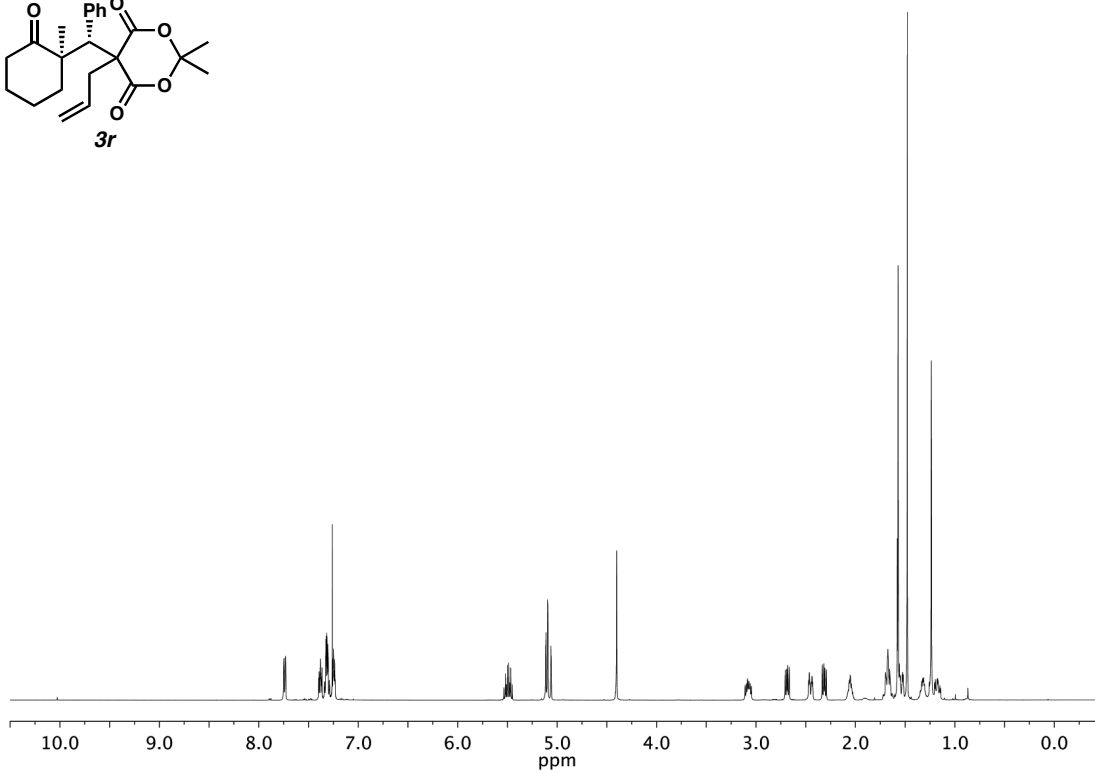
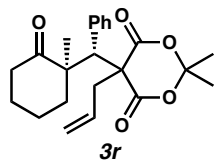


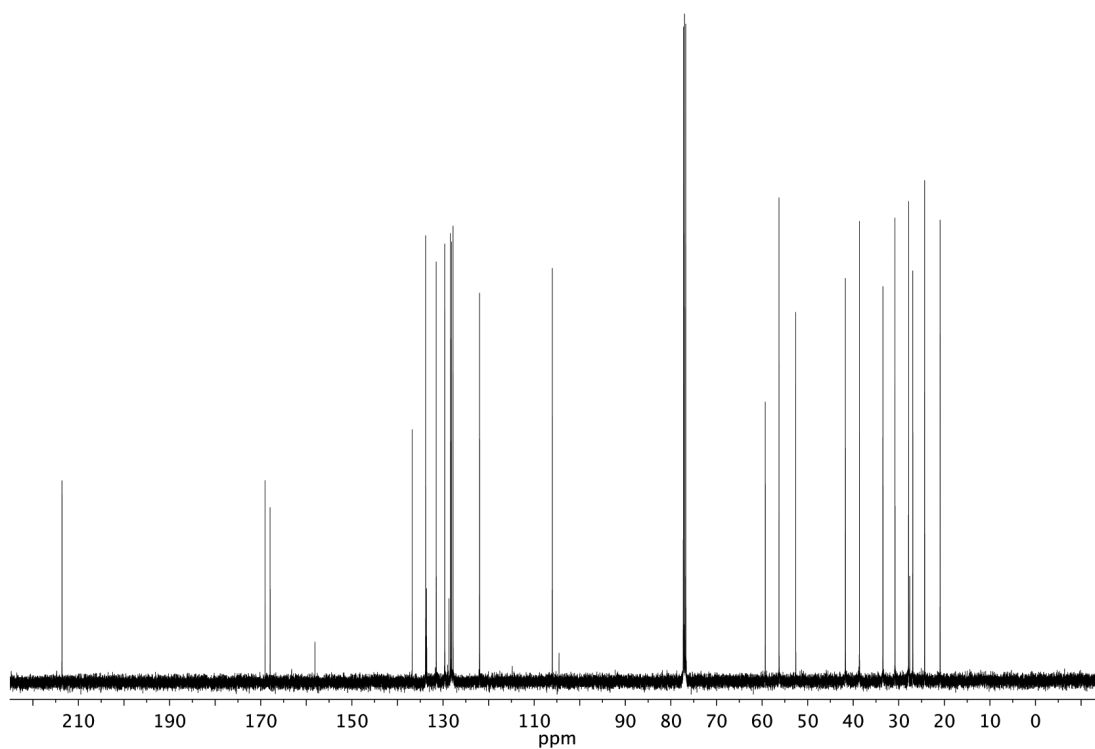
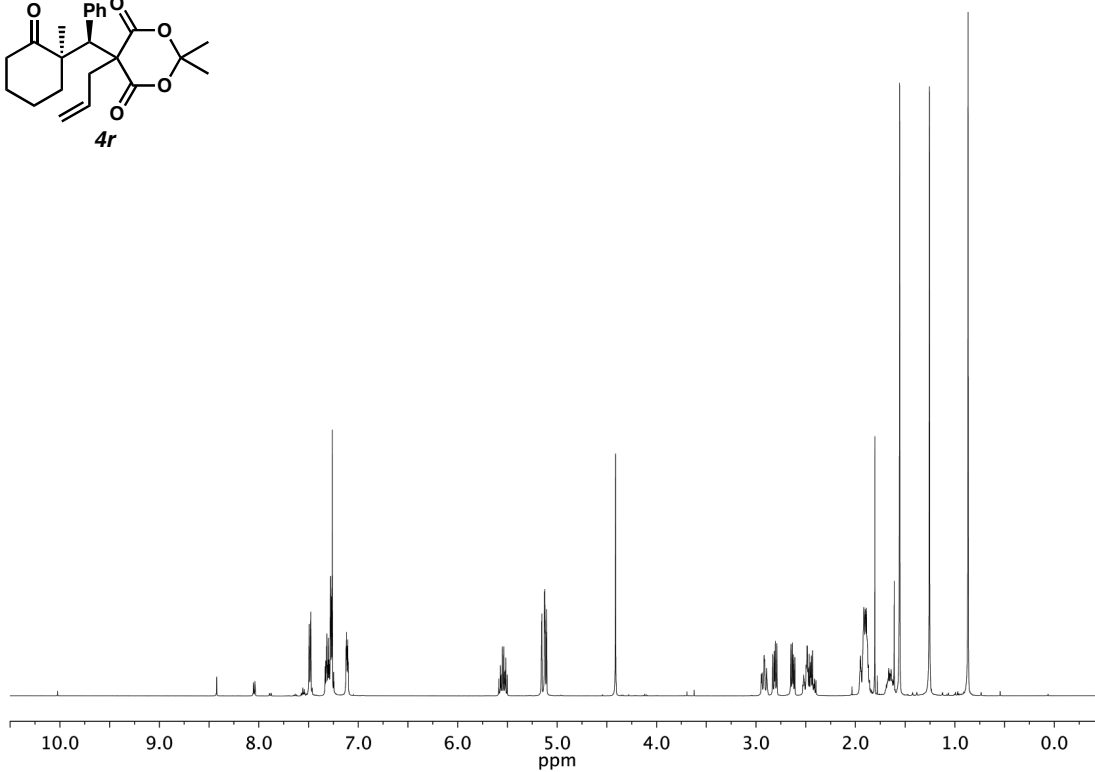
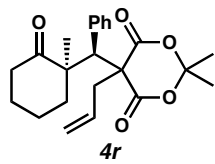


GHMBC-nmr spectra (excerpts):

In the upper two cases (**3,4p**) the proton signal at 3.6 or 3.3 ppm crosses with the nitrile carbons (at 115 ppm). In the case of the two minor isomers, which were not isolated from the asymmetric reaction (bottom), this signal is missing.





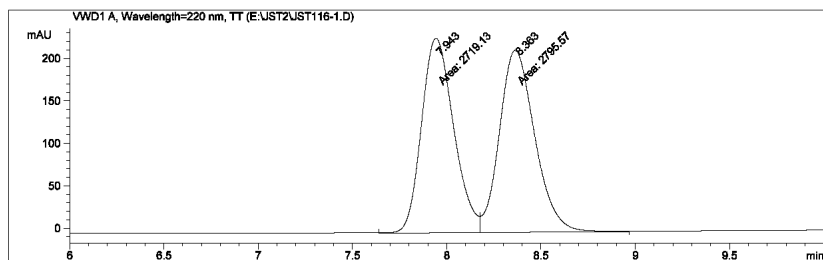


HPLC and SFC Data for Products 3 and 4

Data File E:\JST2\JST116-1.D
 Sample Name: jst-ii-116-1

```

=====
Acq. Operator   : jst                      Seq. Line : 3
Acq. Instrument : Instrument 3              Location  : Vial 31
Injection Date  : 12/17/2008 6:30:51 PM    Inj       : 1
                                           Inj Volume: 5 µl
Acq. Method     : C:\HPCHEM\3\METHODS\JST10IPA.M
Last changed    : 12/17/2008 6:10:30 PM by MSW
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
C5 10IPA.M)
Last changed    : 10/17/2009 4:28:06 PM by jst
                (modified after loading)
Sample Info     : jst-ii-116-1,10% IPA, 1mL/min, 30min, 220nm
=====
  
```



Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
  
```

Signal 1: WWD1 A, Wavelength=220 nm, TT

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.943	MF	0.1977	2719.13086	229.18980	49.3069
2	8.363	FM	0.2172	2795.57300	214.55382	50.6931

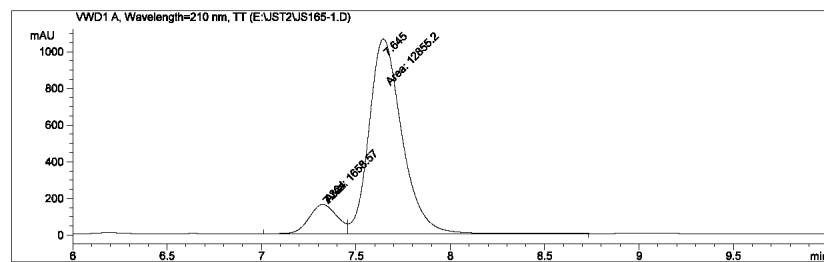
Totals : 5514.70386 443.74362

*** End of Report ***

Data File E:\JST2\JST165-1.D
 Sample Name: jst-ii-165-1

```

=====
Acq. Operator   : jst                      Seq. Line : 4
Acq. Instrument : Instrument 3              Location  : Vial 71
Injection Date   : 2/21/2009 8:08:15 PM    Inj       : 1
                                           Inj Volume: 5 µl
Acq. Method     : C:\HPCHEM\3\METHODS\JST10IPY.M
Last changed    : 2/2/2009 7:39:10 PM by ntm
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
C5 10IPA.M)
Last changed    : 10/17/2009 4:40:31 PM by jst
                (modified after loading)
Sample Info     : jst-ii-165-1, Diast 1, OD-H, 10%IPA, 1ml/min, 20min, 21
                0 nm
=====
  
```



Area Percent Report

```

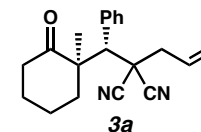
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
  
```

Signal 1: WWD1 A, Wavelength=210 nm, TT

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.324	MF	0.1732	1658.56885	159.60066	11.4275
2	7.645	FM	0.2017	1.28552e4	1062.45593	88.5725

Totals : 1.45138e4 1222.05659

*** End of Report ***

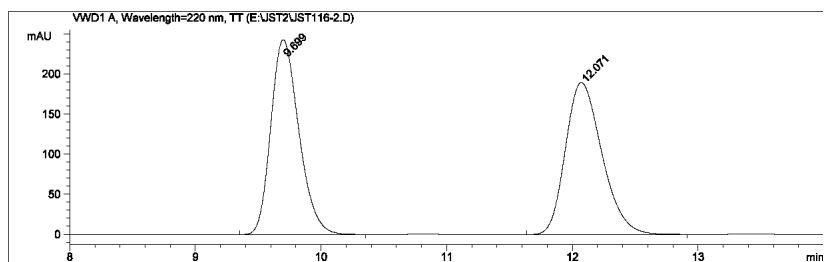


Data File E:\JST2\JST116-2.D
Sample Name: jst-ii-116-2

=====

Acq. Operator	: jst	Seq. Line	: 2
Acq. Instrument	: Instrument 3	Location	: Vial 32
Injection Date	: 12/17/2008 6:45:33 PM	Inj	: 1
		Inj Volume	: 5 µl

Acq. Method : C:\HPCHEM\3\METHODS\JST10IPA.M
Last changed : 12/17/2008 6:10:30 PM by MSW
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL C5 10IPA.M)
Last changed : 10/17/2009 4:41:43 PM by jst
(modified after loading)
Sample Info : jst-ii-116-2, 10% IPA, 1mL/min, 30min, 220nm



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: WVD1 A, Wavelength=220 nm, TT

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s [mAU]	Area %
1	9.699	BB	0.2391	3765.78906	242.89462	49.6787
2	12.071	BB	0.3122	3814.49902	189.38126	50.3213

Totals : 7580.28809 432.27588

=====
*** End of Report ***

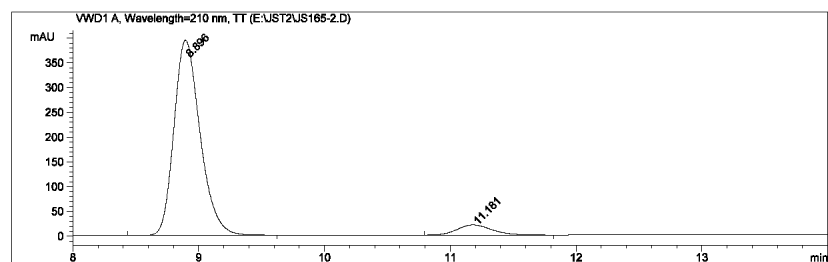
Instrument 1 10/17/2009 4:41:45 PM jst

Data File E:\JST2\JS165-2.D
Sample Name: jst-ii-165-2

=====

Acq. Operator	: jst	Seq. Line	: 6
Acq. Instrument	: Instrument 3	Location	: Vial 72
Injection Date	: 2/21/2009 8:40:05 PM	Inj	: 1
		Inj Volume	: 5 µl

Acq. Method : C:\HPCHEM\3\METHODS\JST10IPY.M
Last changed : 2/2/2009 7:39:10 PM by ntm
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL C5 10IPA.M)
Last changed : 10/17/2009 4:42:10 PM by jst
(modified after loading)
Sample Info : jst-ii-165-2, Diast 1, CD-H, 10%IPA, 1ml/min, 20min, 210 nm



=====
Area Percent Report
=====

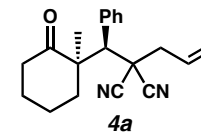
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: WVD1 A, Wavelength=210 nm, TT

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s [mAU]	Area %
1	8.896	VB	0.2218	5663.72656	393.58646	93.5253
2	11.181	BB	0.2932	392.09930	20.48903	6.4747

Totals : 6055.82587 414.07549

=====
*** End of Report ***



Page 1 of 1 Instrument 1 10/17/2009 4:42:20 PM jst

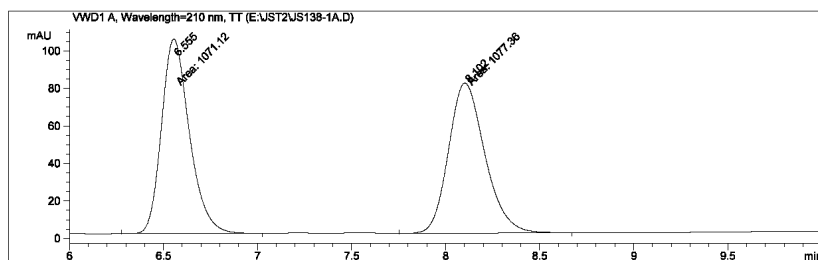
Page 1 of 1

Data File E:\JST2\JS138-1A.D
 Sample Name: jst-ii-138-1

```

=====
Acq. Operator   : jst                      Seq. Line :    4
Acq. Instrument : Instrument 3              Location  : Vial 71
Injection Date  : 1/29/2009 8:41:59 PM     Inj       :    1
                                           Inj Volume: 5 µl

Acq. Method     : C:\HPCHEM\3\METHODS\JST10IPY.M
Last changed    : 1/29/2009 8:40:59 PM by ntm
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
                  C5 10IPA.M)
Last changed    : 10/17/2009 4:42:53 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-138-1,diast1, 10% IPA, OD-H, 1ml/min 20min, 210n
                  m
                  rac
  
```



Area Percent Report

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: VWD1 A, Wavelength=210 nm, TT

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]	Area %
1	6.555	MM	0.1720	1071.11731	49.8548	103.76250	49.8548
2	8.102	MM	0.2242	1077.35596	50.1452	80.07286	50.1452

Totals : 2148.47327 183.83536

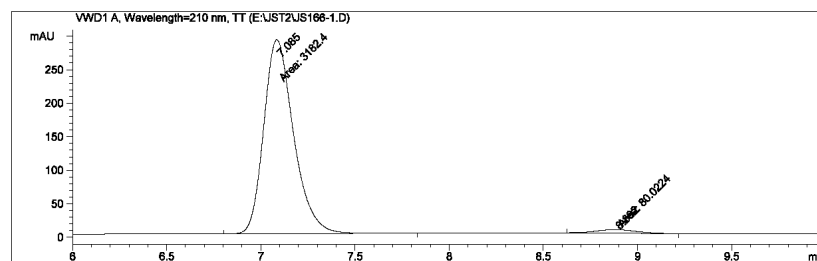
*** End of Report ***

Data File E:\JST2\JS166-1.D
 Sample Name: jst-ii-166-1

```

=====
Acq. Operator   : jst                      Seq. Line :    4
Acq. Instrument : Instrument 3              Location  : Vial 71
Injection Date   : 2/23/2009 1:19:35 AM     Inj       :    1
                                           Inj Volume: 5 µl

Acq. Method     : C:\HPCHEM\3\METHODS\JST10IPY.M
Last changed    : 2/2/2009 7:39:10 PM by ntm
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
                  C5 10IPA.M)
Last changed    : 10/17/2009 4:45:00 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-166-1, Diast 1, OD-H, 10%IPA, 1ml/min, 20min, 21
                  0 nm
  
```



Area Percent Report

```

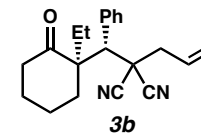
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: VWD1 A, Wavelength=210 nm, TT

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]	Area %
1	7.085	MM	0.1832	3182.40210	97.5471	289.57819	97.5471
2	8.882	MM	0.2466	80.02241	2.4529	5.40944	2.4529

Totals : 3262.42451 294.98763

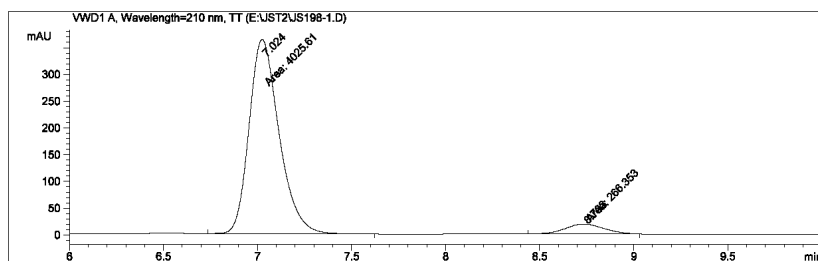
*** End of Report ***



Data File E:\JST2\JS198-1.D
 Sample Name: jst-ii-198-1

```

=====
Acq. Operator   : jst                      Seq. Line :    5
Acq. Instrument : Instrument 3              Location  : Vial 91
Injection Date  : 4/14/2009 7:53:52 PM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : C:\HPCHEM\3\METHODS\JST10IPY.M
Last changed    : 3/10/2009 3:03:02 PM by jst
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
                  C5 10IPA.M)
Last changed    : 10/17/2009 4:49:57 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-198-1, 1st diast, OD-H, 10% IPA, 1ml/min, 40min,
                  210nm
=====
  
```



Area Percent Report

```

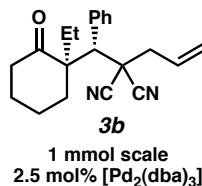
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: WVD1 A, Wavelength=210 nm, TT

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	7.024	MM	0.1848	4025.61377	363.09946	93.7942
2	8.733	MM	0.2390	266.35269	18.57378	6.2058

Totals : 4291.96646 381.67324

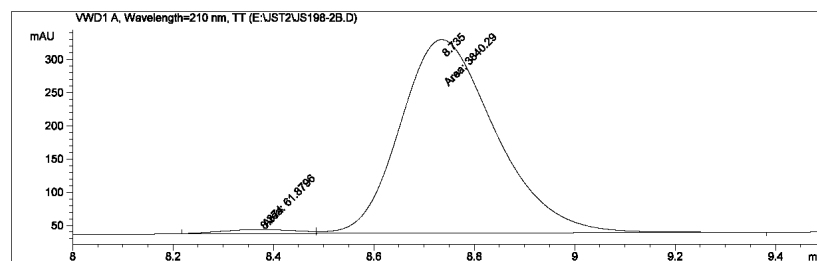
*** End of Report ***



Data File E:\JST2\JS198-2B.D
 Sample Name: jst-ii-198-2

```

=====
Acq. Operator   : jst                      Seq. Line :    3
Acq. Instrument : Instrument 3              Location  : Vial 92
Injection Date  : 4/14/2009 11:25:44 PM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : C:\HPCHEM\3\METHODS\JST51PY.M
Last changed    : 3/20/2009 7:53:11 PM by JL
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
                  C5 10IPA.M)
Last changed    : 10/17/2009 4:50:31 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-198-2, 2nd diast, OD-H, 5% IPA, 1ml/min, 40min,
                  210nm
=====
  
```



Area Percent Report

```

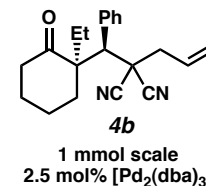
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: WVD1 A, Wavelength=210 nm, TT

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	8.374	MF	0.1691	61.87960	6.09783	1.5858
2	8.735	EM	0.2197	3840.28687	291.36536	98.4142

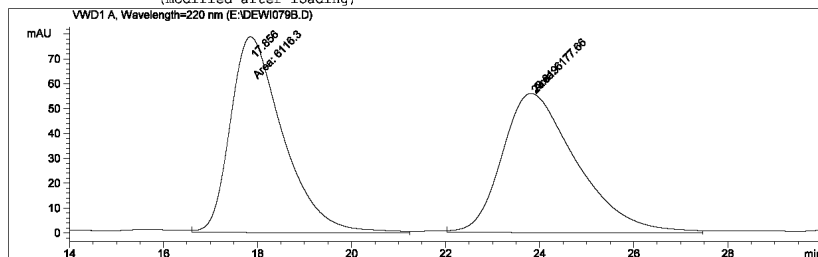
Totals : 3902.16646 297.46319

*** End of Report ***



Data File E:\DEW1079B.D
Sample Name: DEW1079B

=====
Acq. Operator : DEW Seq. Line : 3
Acq. Instrument : Instrument 3 Location : Vial 4
Injection Date : 3/7/2006 1:34:54 PM Inj : 1
 Inj Volume : 5 µl
Acq. Method : C:\HPCHEM\3\METHODS\4-E0H60W.M
Last changed : 3/7/2006 1:18:52 PM by jlr
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
 C5 10IPA.M)
Last changed : 10/17/2009 4:54:33 PM by jst
 (modified after loading)



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	17.856	MM	1.2945	6116.29688	78.74833	49.7504
2	23.819	MM	1.8397	6177.66113	55.96516	50.2496

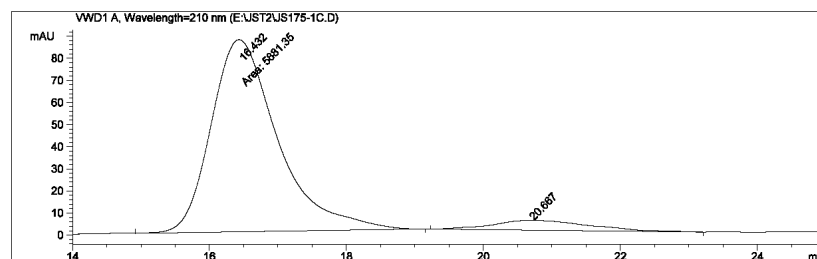
Totals : 1.22940e4 134.71349

=====
*** End of Report ***

Data File E:\JST2\JST175-1C.D
Sample Name: jst-ii-177-1

=====
Acq. Operator : jst Seq. Line : 23
Acq. Instrument : Instrument 3 Location : Vial 71
Injection Date : 3/3/2009 12:43:05 AM Inj : 1
 Inj Volume : 15 µl
Acq. Method : C:\HPCHEM\3\METHODS\JST177.M
Last changed : 3/3/2009 12:28:10 AM by jst
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
 C5 10IPA.M)
Last changed : 10/17/2009 4:55:27 PM by jst
 (modified after loading)

Sample Info : jst-ii-177-1, Diast 1, OJ, 4%EtOH, 1ml/min, 40min, 210n
 m



=====
Area Percent Report
=====

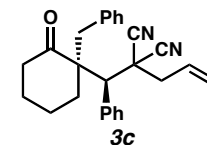
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	16.432	MM	1.1276	5881.35107	86.92758	92.9751
2	20.667	BV	1.1763	444.37845	4.41816	7.0249

Totals : 6325.72952 91.34574

=====
*** End of Report ***

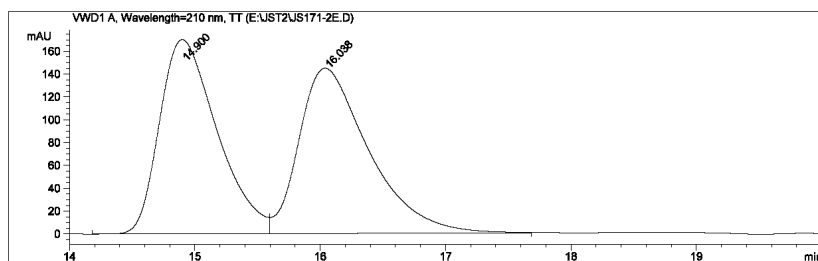


Data File E:\JST2\JS171-2E.D
 Sample Name: jst-ii-171-2

```

=====
Acq. Operator   : jst                      Seq. Line :    6
Acq. Instrument : Instrument 3              Location  : Vial 71
Injection Date  : 3/5/2009 3:24:42 PM      Inj       :    1
                                           Inj Volume: 5 µl

Acq. Method     : C:\HPCHEM\3\METHODS\JS171-2.M
Last changed    : 3/5/2009 12:49:08 PM by jst
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
                  C5 10IPA.M)
Last changed    : 10/17/2009 5:01:32 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-171-2, Diast 1, OD-H, 3%IPA, 1ml/min, 40min, 210
                  nm
=====
  
```



Area Percent Report

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: WVD1 A, Wavelength=210 nm, TT

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	14.900	VV	0.4826	5419.77686	169.98222	49.1140
2	16.038	VB	0.5787	5615.32031	144.70215	50.8860

Totals : 1.10351e4 314.68437

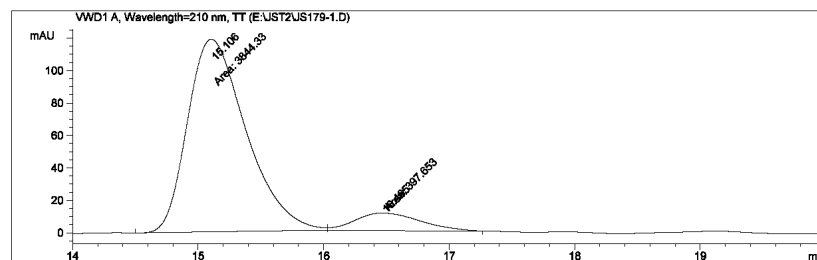
*** End of Report ***

Data File E:\JST2\JS179-1.D
 Sample Name: jst-ii-179-1

```

=====
Acq. Operator   : jst                      Seq. Line :   11
Acq. Instrument : Instrument 3              Location  : Vial 91
Injection Date   : 3/6/2009 2:54:04 AM      Inj       :    1
                                           Inj Volume: 5 µl

Acq. Method     : C:\HPCHEM\3\METHODS\JS171-2A.M
Last changed    : 3/5/2009 5:28:08 PM by jst
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
                  C5 10IPA.M)
Last changed    : 10/17/2009 5:02:51 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-179-1, Diast 1, ODH, 3%IPA, 1ml/min, 210nm
=====
  
```



Area Percent Report

```

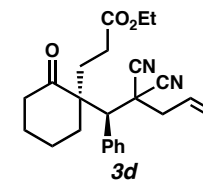
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: WVD1 A, Wavelength=210 nm, TT

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	15.106	MM	0.5395	3844.32886	118.75735	90.6258
2	16.465	MM	0.6136	397.65253	10.80195	9.3742

Totals : 4241.98138 129.55929

*** End of Report ***

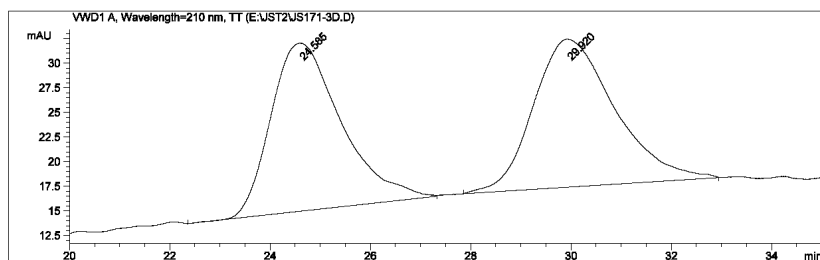


Data File E:\JST2\JS171-3D.D
 Sample Name: jst-ii-171-3

```

=====
Acq. Operator   : jst                      Seq. Line :    3
Acq. Instrument : Instrument 3              Location  : Vial 72
Injection Date  : 3/5/2009 10:43:27 AM     Inj       :    1
                                           Inj Volume: 5 µl

Acq. Method    : C:\HPCHEM\3\METHODS\JS171-2.M
Last changed   : 3/5/2009 11:20:55 AM by jst
                (modified after loading)
Analysis Method: C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
                C5 10IPA.M)
Last changed   : 10/17/2009 5:03:21 PM by jst
                (modified after loading)
Sample Info    : jst-ii-171-3, Diast 2, OJ, 5%EtOH, 1mL/min, 60min, 210n
                m
  
```



Area Percent Report

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: WVD1 A, Wavelength=210 nm, TT

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	24.585	VB	1.1437	1665.96545	17.08151	49.5903
2	29.920	BB	1.3197	1693.49585	15.03772	50.4097

Totals : 3359.46130 32.11923

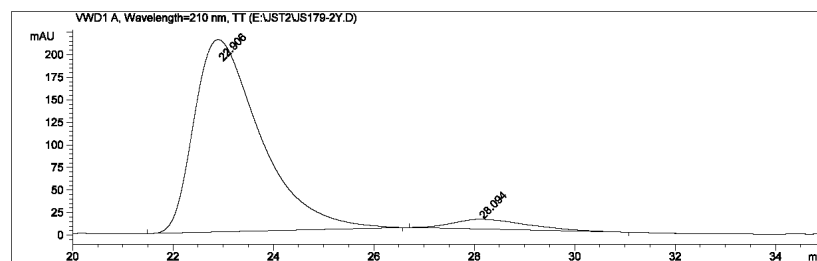
*** End of Report ***

Data File E:\JST2\JS179-2Y.D
 Sample Name: jst-ii-179-2

```

=====
Acq. Operator   : jst                      Seq. Line :    2
Acq. Instrument : Instrument 3              Location  : Vial 91
Injection Date  : 3/5/2009 2:06:51 PM     Inj       :    1
                                           Inj Volume: 25 µl

Acq. Method    : C:\HPCHEM\3\METHODS\JS171-3A.M
Last changed   : 3/5/2009 1:58:56 PM by jst
Analysis Method: C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
                C5 10IPA.M)
Last changed   : 10/17/2009 5:03:49 PM by jst
                (modified after loading)
Sample Info    : jst-ii-179-2, Diast 2, OJ, 5%EtOH, 1mL/min, 45min, 210n
                m, 25µL inject
  
```



Area Percent Report

```

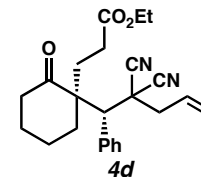
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: WVD1 A, Wavelength=210 nm, TT

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	22.906	BB	1.0934	1.95937e4	213.18834	94.5163
2	28.094	BB	1.2277	1136.78894	10.85404	5.4837

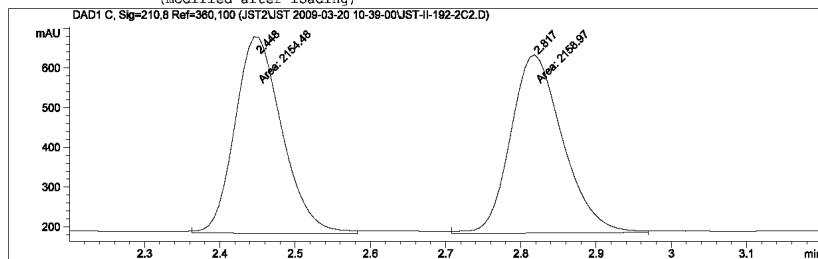
Totals : 2.07305e4 224.04238

*** End of Report ***



Data File C:\CHEM32\1\DATA\JST2\JST 2009-03-20 10-39-00\JST-II-192-2C2.D
 Sample Name: jst-ii-190-2

```
=====
Acq. Operator   : jst                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : P1-D-08
Injection Date  : 3/20/2009 10:43:06 AM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-03-20 10-39-00\JS10IPAC2.M
Last changed    : 3/20/2009 10:31:33 AM by NTM
Analysis Method  : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
                  C5 10IPA.M)
Last changed    : 10/17/2009 5:05:37 PM by jst
                  (modified after loading)
=====
```



Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

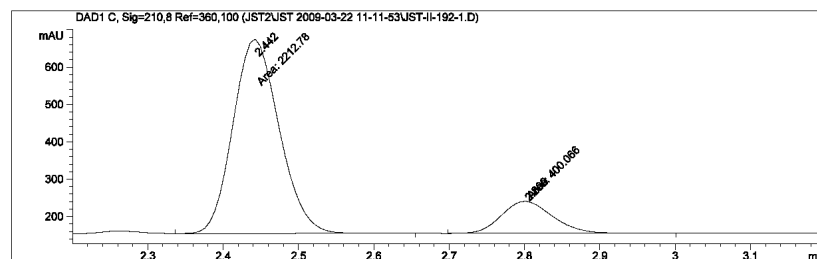
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.448	MM	0.0720	2154.47925	498.98123	49.9479
2	2.817	MM	0.0798	2158.97363	450.63431	50.0521

Totals : 4313.45288 949.61554

*** End of Report ***

Data File C:\CHEM32\1\DATA\JST2\JST 2009-03-22 11-11-53\JST-II-192-1.D
 Sample Name: jst-ii-192-1

```
=====
Acq. Operator   : jst                      Seq. Line :    4
Acq. Instrument : Instrument 1              Location  : P1-C-09
Injection Date   : 3/22/2009 11:22:58 AM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-03-22 11-11-53\JS10IPAC2.M
Last changed    : 3/20/2009 10:31:33 AM by NTM
Analysis Method  : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
                  C5 10IPA.M)
Last changed    : 10/17/2009 5:07:05 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-192-1, 10% IPA, AD-H, 10 min run, 2ml/min
=====
```



Area Percent Report

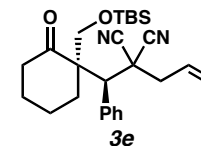
```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.442	MM	0.0707	2212.78052	521.72894	84.6885
2	2.800	MM	0.0776	400.06573	85.93865	15.3115

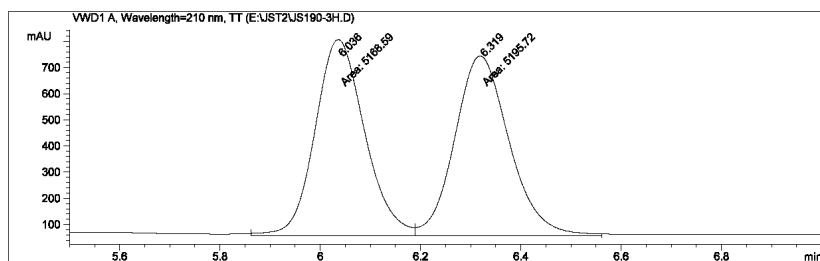
Totals : 2612.84625 607.66759

*** End of Report ***



Data File E:\JST2\JS190-3H.D
 Sample Name: js190-3

```
=====
Acq. Operator   : jst                      Seq. Line :    6
Acq. Instrument : Instrument 3              Location  : Vial 91
Injection Date  : 3/21/2009 7:36:01 PM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : C:\HPCHEM\3\METHODS\JST31PY.M
Last changed    : 3/21/2009 6:52:00 PM by JL
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
C5 10IPA.M)
Last changed    : 10/17/2009 5:07:51 PM by jst
                 (modified after loading)
Sample Info     : jst-190-3, AD, 38IPA, 1ml/min, 40min, 210nm
                 (with 5ipa equilibr)
=====
```



Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: WVD1 A, Wavelength=210 nm, TT

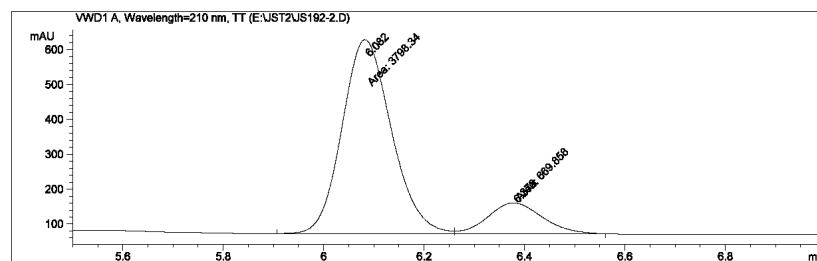
Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	6.036	MF	0.1146	5168.59082	751.73157	49.8691
2	6.319	FM	0.1257	5195.71533	688.82416	50.1309

Totals : 1.03643e4 1440.55573

*** End of Report ***

Data File E:\JST2\JS192-2.D
 Sample Name: js192-2

```
=====
Acq. Operator   : jst                      Seq. Line :    4
Acq. Instrument : Instrument 3              Location  : Vial 92
Injection Date   : 3/22/2009 12:23:58 PM   Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : C:\HPCHEM\3\METHODS\JST31PY.M
Last changed    : 3/21/2009 6:52:00 PM by JL
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
C5 10IPA.M)
Last changed    : 10/17/2009 5:08:29 PM by jst
                 (modified after loading)
Sample Info     : jst-192-2, Diast2, AD, 38IPA, 1ml/min, 40min, 210nm
                 (with 5ipa equilibr)
=====
```



Area Percent Report

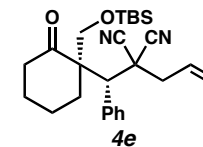
```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: WVD1 A, Wavelength=210 nm, TT

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	6.082	MF	0.1137	3798.33862	556.97052	85.0083
2	6.378	FM	0.1256	669.85809	88.86078	14.9917

Totals : 4468.19672 645.83130

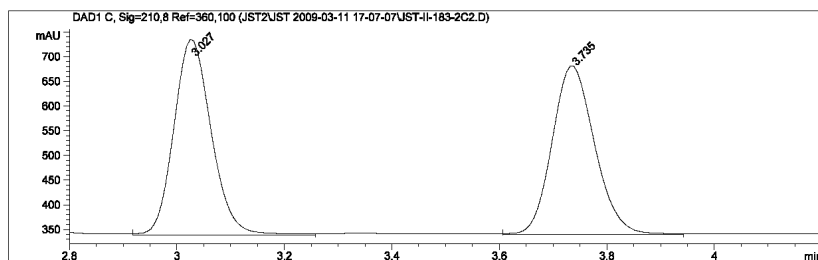
*** End of Report ***



Data File C:\CHEM32\1\DATA\JST2\JST 2009-03-11 17-07-07\JST-II-183-2C2.D
 Sample Name: jst-ii-183-2C2

```

=====
Acq. Operator   : jst                      Seq. Line :    7
Acq. Instrument : Instrument 1              Location  : P1-C-09
Injection Date  : 3/11/2009 6:31:46 PM     Inj       :    1
                                           Inj Volume: 15 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-03-11 17-07-07\JST-183-1C2.M
Last changed    : 3/11/2009 5:32:00 PM by NFM
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
C5 10IPA.M)
Last changed    : 10/17/2009 5:13:18 PM by jst
                 (modified after loading)
Sample Info     : jst-ii-183-2, AD-H, 1ml/min, 20% IPA, 8 min run
=====
  
```



Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
  
```

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.027	BB	0.0744	1919.73047	397.42151	50.4683
2	3.735	VV	0.0842	1884.10547	342.50269	49.5317

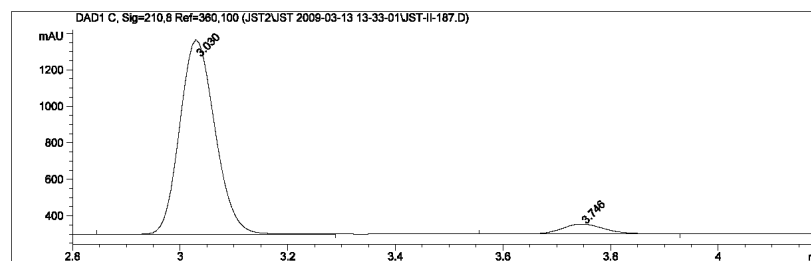
Totals : 3803.83594 739.92419

*** End of Report ***

Data File C:\CHEM32\1\DATA\JST2\JST 2009-03-13 13-33-01\JST-II-187.D
 Sample Name: jst-ii-187

```

=====
Acq. Operator   : JST                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : P1-C-09
Injection Date   : 3/13/2009 1:33:27 PM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-03-13 13-33-01\JST-II-187.M
Last changed    : 3/13/2009 1:34:39 PM by JST
                 (modified after loading)
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
C5 10IPA.M)
Last changed    : 10/17/2009 5:14:50 PM by jst
                 (modified after loading)
Sample Info     : jst-ii-183-1, OJ-H, 1ml/min, 4% IPA, 11 min run
=====
  
```



Area Percent Report

```

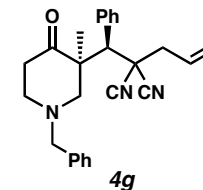
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
  
```

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.030	VB	0.0709	4835.30273	1066.86963	94.4789
2	3.746	BB	0.0812	282.56439	53.90346	5.5211

Totals : 5117.86713 1120.77309

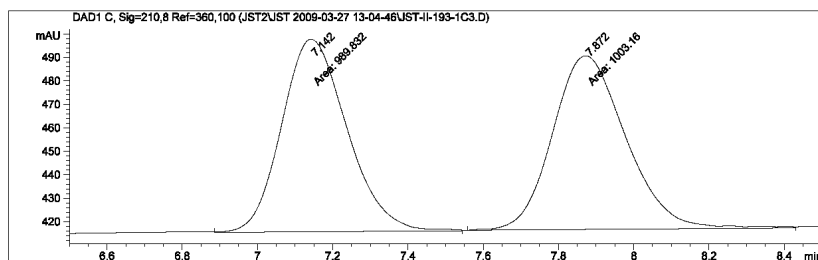
*** End of Report ***



Data File C:\CHEM32\1\DATA\JST2\JST 2009-03-27 13-04-46\JST-II-193-1C3.D
 Sample Name: jst-ii-193-1

```

=====
Acq. Operator   : jst                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : P1-D-06
Injection Date  : 3/27/2009 1:08:54 PM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-03-27 13-04-46\JS10IPAC3.M
Last changed    : 3/26/2009 3:47:08 PM by NTM
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
                  C5 10IPA.M)
Last changed    : 10/17/2009 5:16:10 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-193-1, col3, 10% IPA, 10min run
=====
  
```



Area Percent Report

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.142	MM	0.2010	989.83197	82.09347	49.6655
2	7.872	MM	0.2259	1003.16492	73.99932	50.3345

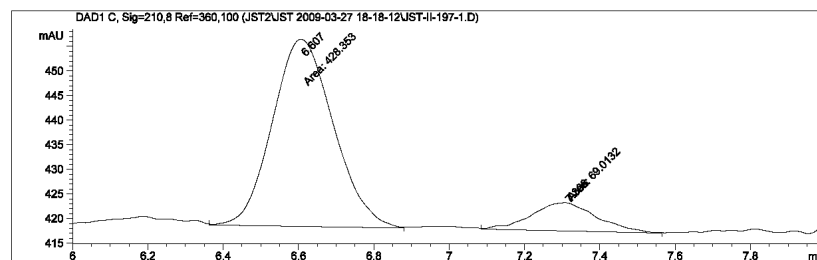
Totals : 1992.99689 156.09279

*** End of Report ***

Data File C:\CHEM32\1\DATA\JST2\JST 2009-03-27 18-18-12\JST-II-197-1.D
 Sample Name: jst-ii-197-1

```

=====
Acq. Operator   : jst                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : P1-D-06
Injection Date   : 3/27/2009 6:22:18 PM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-03-27 18-18-12\JS10IPAC3.M
Last changed    : 3/26/2009 3:47:08 PM by NTM
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
                  C5 10IPA.M)
Last changed    : 10/17/2009 5:18:26 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-197-1, OD-H, 10% IPA, 10min run
=====
  
```



Area Percent Report

```

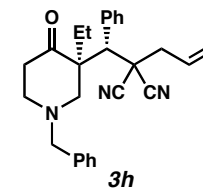
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.607	MM	0.1874	428.35297	38.09812	86.1243
2	7.308	MM	0.1997	69.01321	5.76064	13.8757

Totals : 497.36617 43.85876

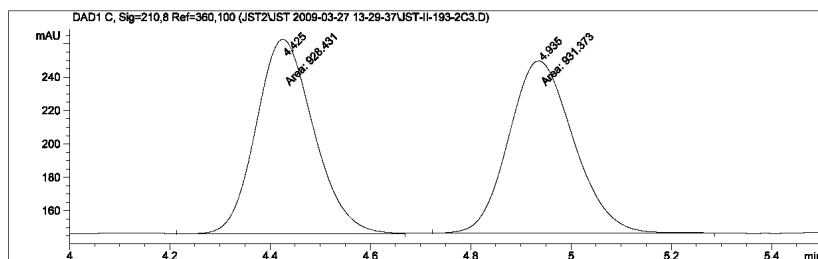
*** End of Report ***



Data File C:\CHEM32\1\DATA\JST2\JST 2009-03-27 13-29-37\JST-II-193-2C3.D
 Sample Name: jst-ii-193-2

```

=====
Acq. Operator   : jst                      Seq. Line :    4
Acq. Instrument : Instrument 1              Location  : P1-D-07
Injection Date  : 3/27/2009 1:53:12 PM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-03-27 13-29-37\JS20IPAC3.M
Last changed    : 3/27/2009 1:46:53 PM by jst
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
                  C5 10IPA.M)
Last changed    : 10/17/2009 5:19:43 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-193-2, col3, 20% IPA, 10min run
=====
  
```



Area Percent Report

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.425	MM	0.1326	928.43091	116.69833	49.9209
2	4.935	MM	0.1501	931.37262	103.40519	50.0791

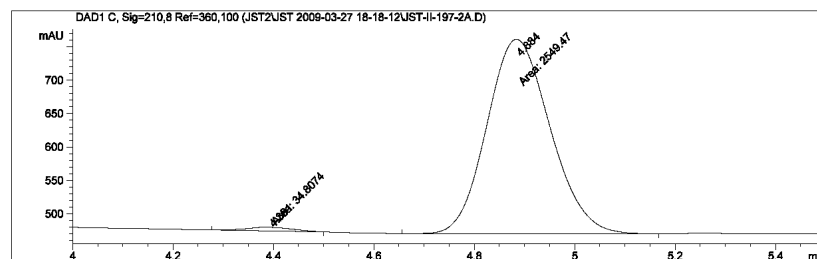
Totals : 1859.80353 220.10352

*** End of Report ***

Data File C:\CHEM32\1\DATA\JST2\JST 2009-03-27 18-18-12\JST-II-197-2A.D
 Sample Name: jst-ii-197-2

```

=====
Acq. Operator   : jst                      Seq. Line :    8
Acq. Instrument : Instrument 1              Location  : P1-D-07
Injection Date  : 3/27/2009 7:15:15 PM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-03-27 18-18-12\JS20IPAC3.M
Last changed    : 3/27/2009 1:46:53 PM by jst
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
                  C5 10IPA.M)
Last changed    : 10/17/2009 5:21:19 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-197-2, OD-H, 20% IPA, 10min run
=====
  
```



Area Percent Report

```

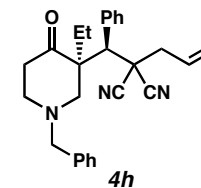
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.391	MM	0.1068	34.80737	5.43432	1.3469
2	4.884	MM	0.1457	2549.46777	291.63715	98.6531

Totals : 2584.27514 297.07146

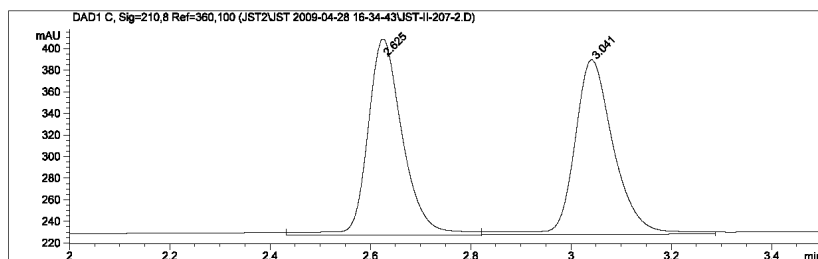
*** End of Report ***



Data File C:\CHEM32\1\DATA\JST2\JST 2009-04-28 16-34-43\JST-II-207-2.D
 Sample Name: jst-ii-207-2

```

=====
Acq. Operator   : jst                      Seq. Line :    4
Acq. Instrument : Instrument 1              Location  : P1-D-07
Injection Date  : 4/28/2009 4:55:43 PM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-04-28 16-34-43\JSTCHIRAL C2 20IPA.M
Last changed    : 4/27/2009 2:04:51 PM by JAE
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
                  C5 10IPA.M)
Last changed    : 10/17/2009 5:23:31 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-207-2, AD-H, 20% IPA, standard run
=====
  
```



Area Percent Report

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.625	VV	0.0721	876.91998	182.24883	49.9985
2	3.041	VB	0.0811	876.97119	162.16396	50.0015

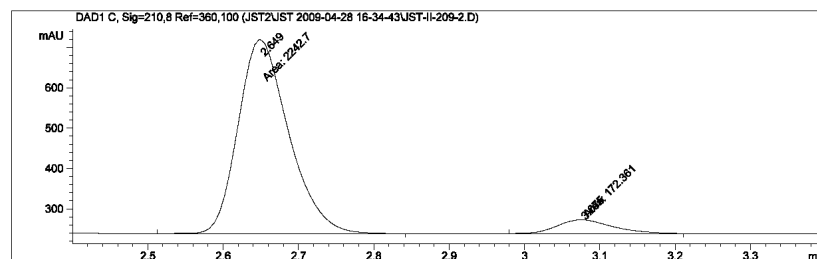
Totals : 1753.89117 344.41278

*** End of Report ***

Data File C:\CHEM32\1\DATA\JST2\JST 2009-04-28 16-34-43\JST-II-209-2.D
 Sample Name: jst-ii-209-2

```

=====
Acq. Operator   : jst                      Seq. Line :   14
Acq. Instrument : Instrument 1              Location  : P4-D-07
Injection Date   : 4/28/2009 6:20:35 PM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-04-28 16-34-43\JSTCHIRAL C2 20IPA.M
Last changed    : 4/27/2009 2:04:51 PM by JAE
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
                  C5 10IPA.M)
Last changed    : 10/17/2009 5:25:00 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-209-2, AD-H, 20% IPA, standard run
=====
  
```



Area Percent Report

```

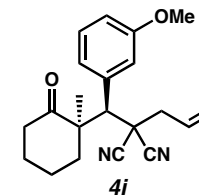
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.649	MM	0.0773	2242.70288	483.31525	92.8631
2	3.075	MM	0.0843	172.36148	34.07234	7.1369

Totals : 2415.06436 517.38759

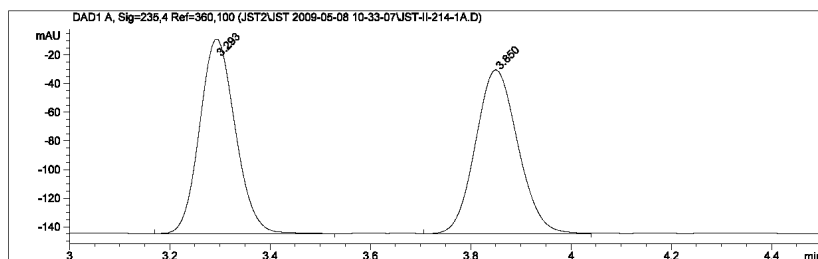
*** End of Report ***



Data File C:\CHEM32\1\DATA\JST2\JST 2009-05-08 10-33-07\JST-II-214-1A.D
 Sample Name: jst-ii-214-1

```

=====
Acq. Operator   : jst                      Seq. Line : 17
Acq. Instrument : Instrument 1              Location  : P1-D-06
Injection Date  : 5/8/2009 12:25:31 PM     Inj       : 1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-05-08 10-33-07\JSTCHIRAL C4 10IPA.M
Last changed    : 5/8/2009 10:50:52 AM by RN
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
                  C5 10IPA.M)
Last changed    : 10/17/2009 5:26:23 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-214-1, OJ-H, 10% IPA, 1ml/min
=====
  
```



Area Percent Report

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 A, Sig=235,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.293	EV	0.0774	670.38794	136.37428	49.8780
2	3.850	VV	0.0905	673.66760	114.68407	50.1220

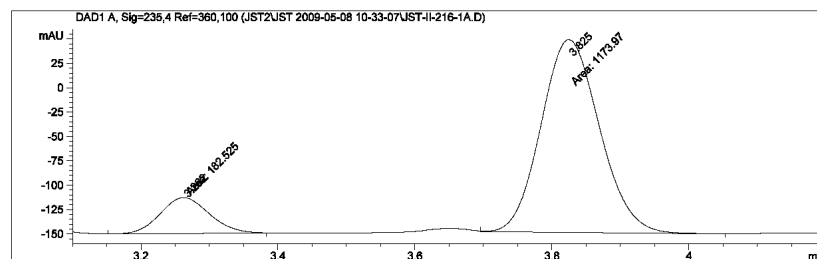
Totals : 1344.05554 251.05836

*** End of Report ***

Data File C:\CHEM32\1\DATA\JST2\JST 2009-05-08 10-33-07\JST-II-216-1A.D
 Sample Name: jst-ii-216-1

```

=====
Acq. Operator   : jst                      Seq. Line : 20
Acq. Instrument : Instrument 1              Location  : P1-D-08
Injection Date  : 5/8/2009 12:49:53 PM     Inj       : 1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-05-08 10-33-07\JSTCHIRAL C4 10IPA.M
Last changed    : 5/8/2009 10:50:52 AM by RN
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
                  C5 10IPA.M)
Last changed    : 10/17/2009 5:27:29 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-214-1, OJ-H, 10% IPA, 1ml/min
=====
  
```



Area Percent Report

```

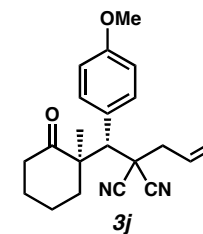
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 A, Sig=235,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.262	MM	0.0823	182.52458	36.96958	13.4556
2	3.825	MM	0.0985	1173.97095	198.64160	86.5444

Totals : 1356.49553 235.61118

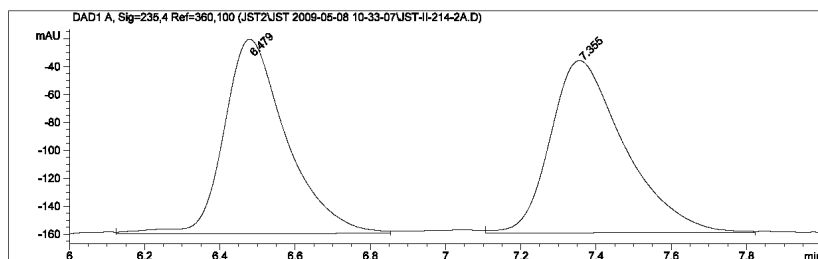
*** End of Report ***



Data File C:\CHEM32\1\DATA\JST2\JST 2009-05-08 10-33-07\JST-II-214-2A.D
 Sample Name: jst-ii-214-2

```

=====
Acq. Operator   : jst                      Seq. Line : 24
Acq. Instrument : Instrument 1              Location  : P1-D-07
Injection Date  : 5/8/2009 1:17:50 PM      Inj       : 1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-05-08 10-33-07\JSTCHIRAL C2 10IPA.M
Last changed    : 5/8/2009 10:50:08 AM by RN
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
                  C5 10IPA.M)
Last changed    : 10/17/2009 5:29:27 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-214-2, AD-H, 10% IPA, 1ml/min
=====
  
```



Area Percent Report

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 A, Sig=235,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.479	VV	0.1729	1611.40491	139.39507	48.7899
2	7.355	VV	0.2039	1691.34033	123.38937	51.2101

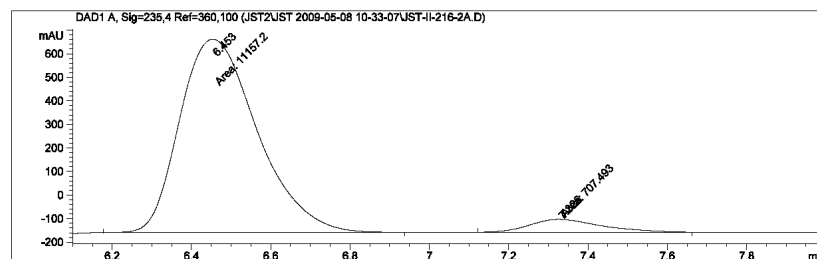
Totals : 3302.74524 262.78443

*** End of Report ***

Data File C:\CHEM32\1\DATA\JST2\JST 2009-05-08 10-33-07\JST-II-216-2A.D
 Sample Name: jst-ii-216-2

```

=====
Acq. Operator   : jst                      Seq. Line : 27
Acq. Instrument : Instrument 1              Location  : P1-D-09
Injection Date  : 5/8/2009 1:42:16 PM      Inj       : 1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-05-08 10-33-07\JSTCHIRAL C2 10IPA.M
Last changed    : 5/8/2009 10:50:08 AM by RN
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-10-15 10-44-36\JST-II-283B-1.D\DA.M (JSTCHIRAL
                  C5 10IPA.M)
Last changed    : 10/17/2009 5:30:09 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-216-2, AD-H, 10% IPA, 1ml/min
=====
  
```



Area Percent Report

```

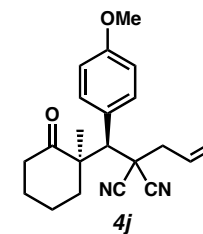
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 A, Sig=235,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.453	MM	0.2263	1.11572e4	821.80597	94.0370
2	7.326	MM	0.2121	707.49298	55.59576	5.9630

Totals : 1.18647e4 877.40173

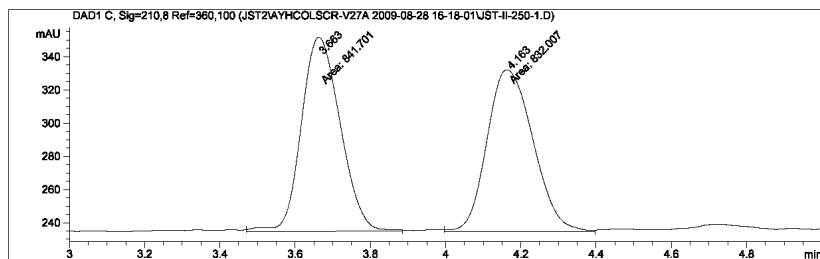
*** End of Report ***



Data File C:\CHEM32\1\DATA\JST2\AYHCOLSCR-V27A 2009-08-28 16-18-01\JST-II-250-1.D
 Sample Name: jst-ii-250-1

```

=====
Acq. Operator   : jst                      Seq. Line :    4
Acq. Instrument : Instrument 1              Location  : P1-F-01
Injection Date  : 8/28/2009 4:29:13 PM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\AYHCOLSCR-V27A 2009-08-28 16-18-01\JSTCHIRAL C4 5IPA.M
Last changed    : 8/28/2009 4:17:57 PM by jst
Analysis Method : C:\CHEM32\1\METHODS\USRJSTRE\JSTCHIRAL C2 10IPA.M
Last changed    : 10/17/2009 9:18:14 PM by jst
                                           (modified after loading)
Sample Info     : jst-ii-250-1, 5% ipa, OJ-H
=====
  
```



Area Percent Report

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.663	MM	0.1196	841.70105	117.31083	50.2896
2	4.163	MM	0.1417	832.00696	97.83093	49.7104

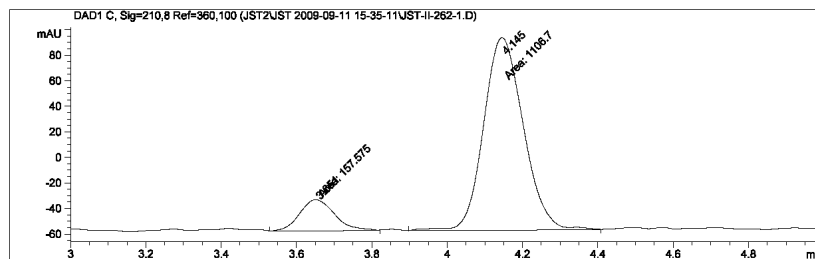
Totals : 1673.70801 215.14176

*** End of Report ***

Data File C:\CHEM32\1\DATA\JST2\JST 2009-09-11 15-35-11\JST-II-262-1.D
 Sample Name: jst-ii-262-1

```

=====
Acq. Operator   : jst                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : P1-E-01
Injection Date  : 9/11/2009 3:39:29 PM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-09-11 15-35-11\JSTCHIRAL C4 5IPA.M
Last changed    : 8/28/2009 4:17:57 PM by jst
Analysis Method : C:\CHEM32\1\METHODS\USRJSTRE\JSTCHIRAL C2 10IPA.M
Last changed    : 10/17/2009 9:14:25 PM by jst
                                           (modified after loading)
Sample Info     : jst-ii-262-1, 5% IPA, OJ-H
=====
  
```



Area Percent Report

```

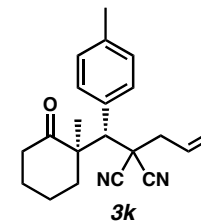
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.651	MM	0.1072	157.57541	24.50137	12.4637
2	4.145	MM	0.1220	1106.69922	151.17523	87.5363

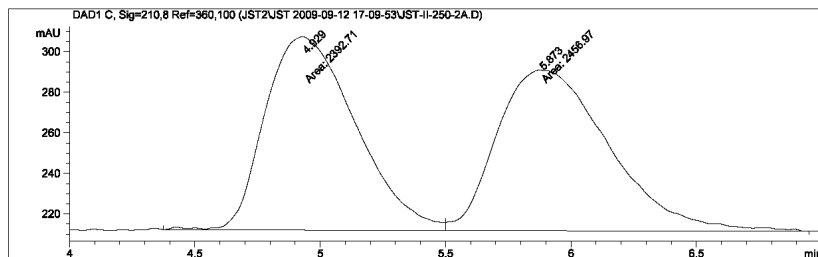
Totals : 1264.27463 175.67660

*** End of Report ***



Data File C:\CHEM32\1\DATA\JST2\JST 2009-09-12 17-09-53\JST-II-250-2A.D
 Sample Name: jst-ii-250-2

```
=====
Acq. Operator   : jst                      Seq. Line :    5
Acq. Instrument : Instrument 1              Location  : P1-F-02
Injection Date  : 9/12/2009 5:38:41 PM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-09-12 17-09-53\JSTCHIRAL C2 10IPA.M
Last changed    : 5/8/2009 10:50:08 AM by RN
Analysis Method : C:\CHEM32\1\METHODS\USRJSTRE\JSTCHIRAL C2 10IPA.M
Last changed    : 10/17/2009 9:21:09 PM by jst
                                           (modified after loading)
Sample Info     : jst-ii-250-2, 10% IPA, AD-H
=====
```



Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

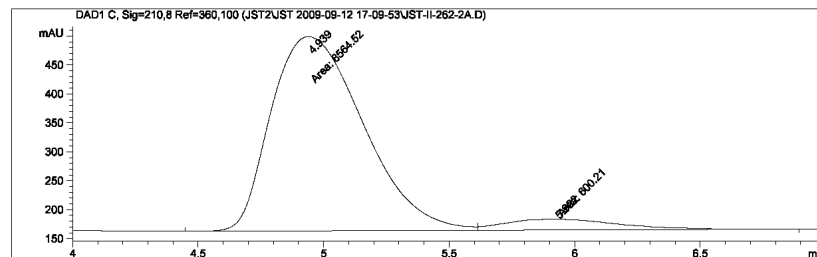
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.929	MF	0.4179	2392.71460	95.42999	49.3375
2	5.873	FM	0.5171	2456.97290	79.19283	50.6625

Totals : 4849.68750 174.62282

*** End of Report ***

Data File C:\CHEM32\1\DATA\JST2\JST 2009-09-12 17-09-53\JST-II-262-2A.D
 Sample Name: jst-ii-262-2

```
=====
Acq. Operator   : jst                      Seq. Line :    3
Acq. Instrument : Instrument 1              Location  : P1-E-02
Injection Date  : 9/12/2009 5:17:48 PM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-09-12 17-09-53\JSTCHIRAL C2 10IPA.M
Last changed    : 5/8/2009 10:50:08 AM by RN
Analysis Method : C:\CHEM32\1\METHODS\USRJSTRE\JSTCHIRAL C2 10IPA.M
Last changed    : 10/17/2009 9:19:38 PM by jst
                                           (modified after loading)
Sample Info     : jst-ii-262-2, 10% IPA, AD-H
=====
```



Area Percent Report

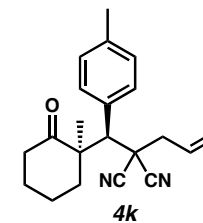
```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.939	MF	0.4252	8564.52246	335.68533	93.4509
2	5.922	FM	0.5267	600.20978	18.99205	6.5491

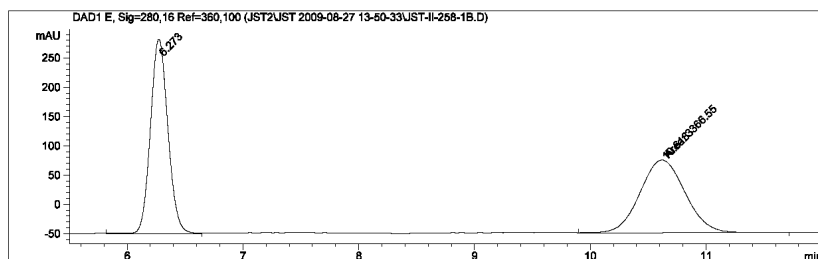
Totals : 9164.73224 354.67739

*** End of Report ***



Data File C:\CHEM32\1\DATA\JST2\JST 2009-08-27 13-50-33\JST-II-258-1B.D
 Sample Name: jst-ii-258-1

```
=====
Acq. Operator   : jst                      Seq. Line : 13
Acq. Instrument : Instrument 1              Location  : P1-F-05
Injection Date  : 8/27/2009 3:42:34 PM     Inj       : 1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-08-27 13-50-33\JSTCHIRAL C4 10IPA.M
Last changed    : 8/27/2009 1:49:43 PM by jst
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-08-27 13-50-33\JST-II-258-1B.D\DA.M (JSTCHIRAL
                  C4 10IPA.M)
Last changed    : 10/17/2009 9:30:04 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-258-1, OJ-H, 10% IPA
=====
```



Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 E, Sig=280,16 Ref=360,100

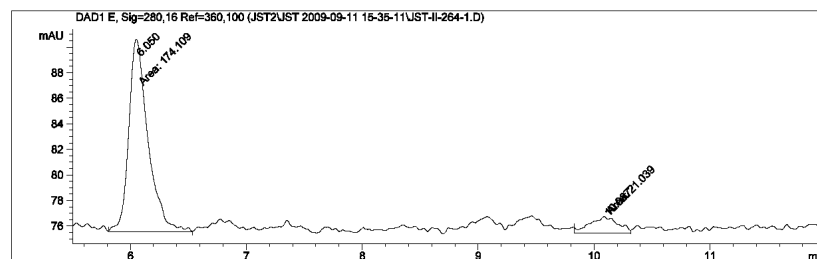
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.273	VB	0.1641	3473.59692	331.88678	50.7825
2	10.618	MM	0.4515	3366.54712	124.28202	49.2175

Totals : 6840.14404 456.16880

*** End of Report ***

Data File C:\CHEM32\1\DATA\JST2\JST 2009-09-11 15-35-11\JST-II-264-1.D
 Sample Name: jst-ii-264-1

```
=====
Acq. Operator   : jst                      Seq. Line : 10
Acq. Instrument : Instrument 1              Location  : P1-E-05
Injection Date   : 9/11/2009 4:35:17 PM     Inj       : 1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-09-11 15-35-11\JSTCHIRAL C4 10IPA.M
Last changed    : 8/27/2009 1:49:43 PM by jst
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-09-11 15-35-11\JST-II-264-1.D\DA.M (JSTCHIRAL
                  C4 10IPA.M)
Last changed    : 10/17/2009 9:30:50 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-264-1, 10% IPA, OJ-H
=====
```



Area Percent Report

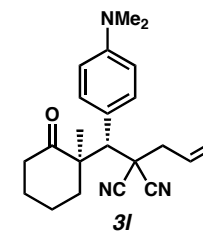
```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 E, Sig=280,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.050	MM	0.1925	174.10873	15.07142	89.2189
2	10.087	MM	0.2715	21.03904	1.29150	10.7811

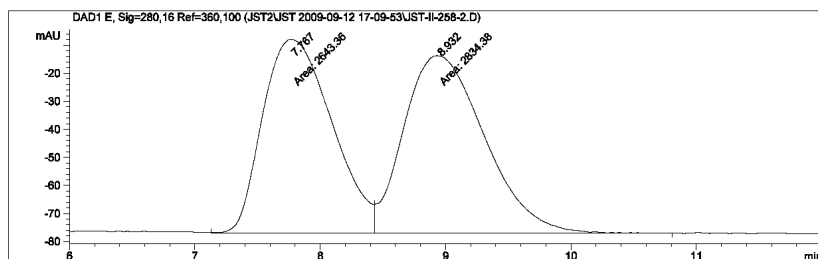
Totals : 195.14778 16.36292

*** End of Report ***



```

=====
Acq. Operator   : jst                      Seq. Line : 11
Acq. Instrument : Instrument 1              Location  : P1-F-06
Injection Date  : 9/12/2009 6:41:17 PM     Inj       : 1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-09-12 17-09-53\JSTCHIRAL C2 10IPA.M
Last changed    : 5/8/2009 10:50:08 AM by RN
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-09-11 15-35-11\JST-II-264-1.D\DA.M (JSTCHIRAL
                  C4 10IPA.M)
Last changed    : 10/17/2009 9:33:27 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-258-2, 10% IPA, AD-H
=====
  
```



Area Percent Report

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 E, Sig=280,16 Ref=360,100

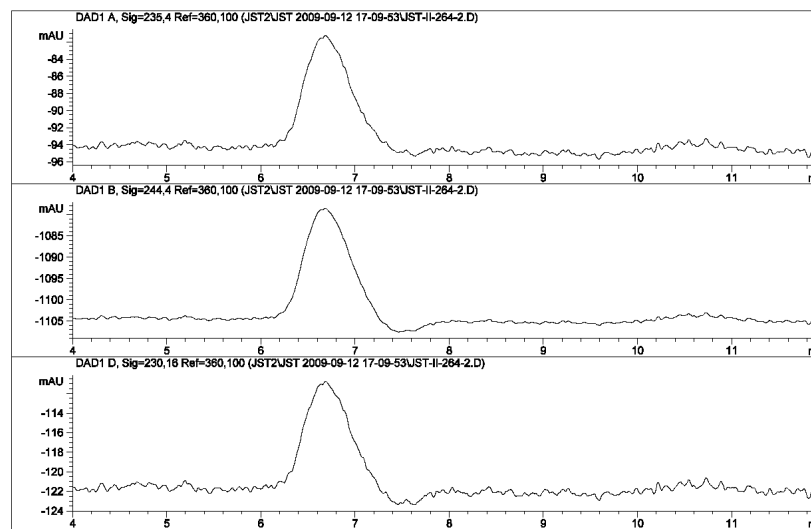
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.767	MF	0.6371	2643.36060	69.15063	48.2564
2	8.932	FM	0.7461	2834.38013	63.31586	51.7436

Totals : 5477.74072 132.46649

*** End of Report ***

```

=====
Acq. Operator   : jst                      Seq. Line : 13
Acq. Instrument : Instrument 1              Location  : P1-E-06
Injection Date  : 9/12/2009 7:02:07 PM     Inj       : 1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-09-12 17-09-53\JSTCHIRAL C2 10IPA.M
Last changed    : 5/8/2009 10:50:08 AM by RN
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-09-12 17-09-53\JST-II-264-2.D\DA.M (JSTCHIRAL
                  C2 10IPA.M)
Last changed    : 10/17/2009 9:36:32 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-264-2, 10% IPA, AD-H
=====
  
```



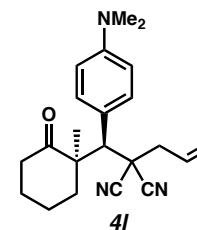
Area Percent Report

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

No peaks found

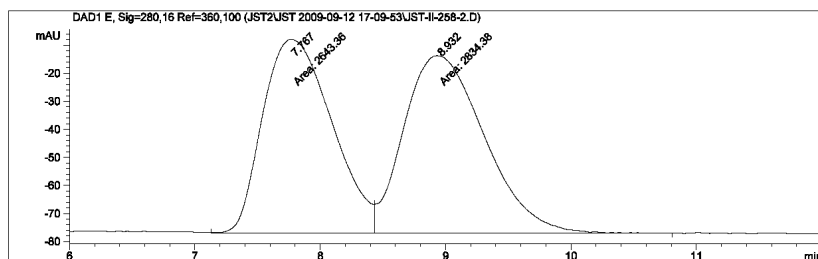
*** End of Report ***



Data File C:\CHEM32\1\DATA\JST2\JST 2009-09-12 17-09-53\JST-II-258-2.D
 Sample Name: jst-ii-258-2

```

=====
Acq. Operator   : jst                      Seq. Line : 11
Acq. Instrument : Instrument 1              Location  : P1-F-06
Injection Date  : 9/12/2009 6:41:17 PM     Inj       : 1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-09-12 17-09-53\JSTCHIRAL C2 10IPA.M
Last changed    : 5/8/2009 10:50:08 AM by RN
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-09-11 15-35-11\JST-II-264-1.D\DA.M (JSTCHIRAL
                  C4 10IPA.M)
Last changed    : 10/17/2009 9:33:27 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-258-2, 10% IPA, AD-H
=====
  
```



Area Percent Report

```

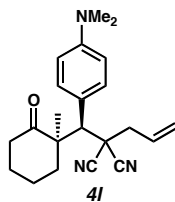
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 E, Sig=280,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.767	MF	0.6371	2643.36060	69.15063	48.2564
2	8.932	FM	0.7461	2834.38013	63.31586	51.7436

Totals : 5477.74072 132.46649

*** End of Report ***



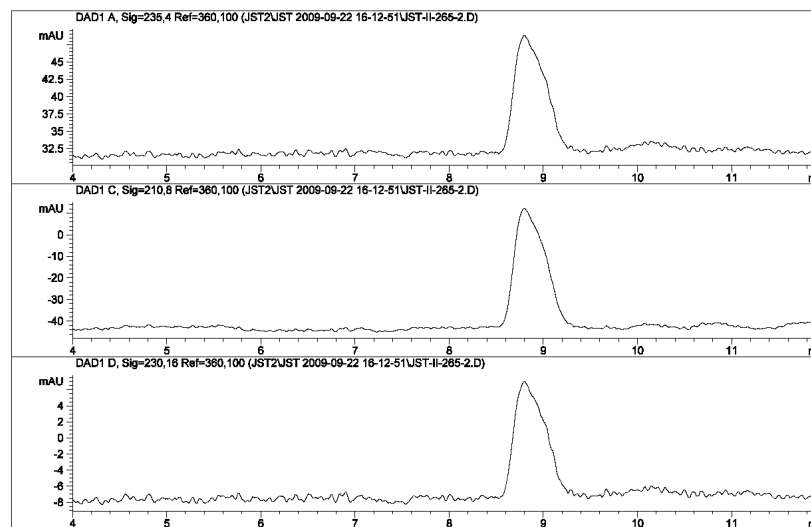
catalyst: Pd(dmdba)₂

Page 1 of 1

Data File C:\CHEM32\1\DATA\JST2\JST 2009-09-22 16-12-51\JST-II-265-2.D
 Sample Name: jst-ii-265-2

```

=====
Acq. Operator   : jst                      Seq. Line : 6
Acq. Instrument : Instrument 1              Location  : P1-F-02
Injection Date  : 9/22/2009 4:47:07 PM     Inj       : 1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-09-22 16-12-51\JSTCHIRAL C2 10IPA.M
Last changed    : 5/8/2009 10:50:08 AM by RN
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-09-12 17-09-53\JST-II-264-2.D\DA.M (JSTCHIRAL
                  C2 10IPA.M)
Last changed    : 10/17/2009 9:39:28 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-265-2, AD-H, 10% IPA
=====
  
```



Area Percent Report

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

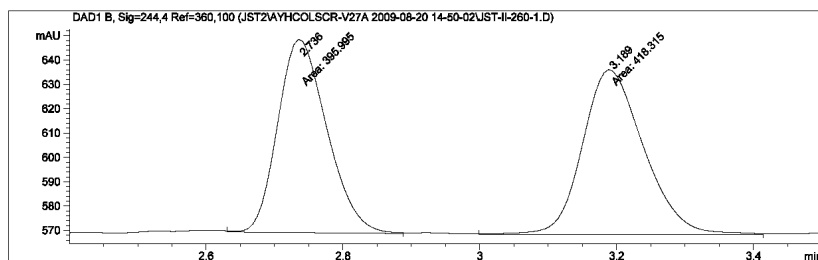
No peaks found

*** End of Report ***

Data File C:\CHEM32\1\DATA\JST2\AYHCOLSCR-V27A 2009-08-20 14-50-02\JST-II-260-1.D
 Sample Name: jst-ii-260-1

```

=====
Acq. Operator   : jst                      Seq. Line : 18
Acq. Instrument : Instrument 1              Location  : P1-E-03
Injection Date  : 8/20/2009 5:10:08 PM     Inj       : 1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\AYHCOLSCR-V27A 2009-08-20 14-50-02\JSTCHIRAL C2 20IPA.M
Last changed    : 7/10/2009 2:09:00 PM by VJS
Analysis Method : C:\CHEM32\1\DATA\JST2\AYHCOLSCR-V27A 2009-08-20 14-50-02\JST-II-260-1.D\DA.M (
                  JSTCHIRAL C2 20IPA.M)
Last changed    : 10/17/2009 9:47:30 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-260-1, 20% IPA, AD-H
=====
  
```



Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
  
```

Signal 1: DAD1 B, Sig=244,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.736	MM	0.0828	395.99509	79.69503	48.6295
2	3.189	MM	0.1029	418.31451	67.74582	51.3705

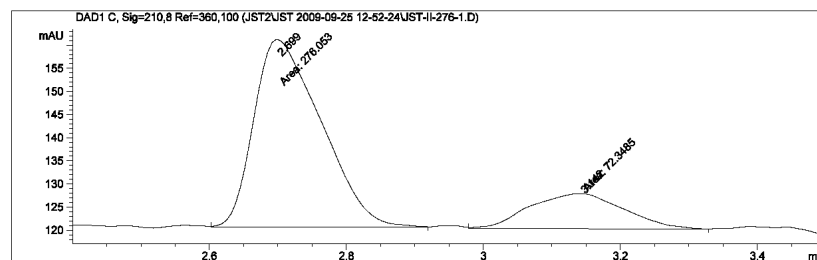
Totals : 814.30960 147.44085

*** End of Report ***

Data File C:\CHEM32\1\DATA\JST2\JST 2009-09-25 12-52-24\JST-II-276-1.D
 Sample Name: jst-ii-276-1

```

=====
Acq. Operator   : jst                      Seq. Line : 12
Acq. Instrument : Instrument 1              Location  : P1-F-05
Injection Date   : 9/25/2009 2:38:39 PM     Inj       : 1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-09-25 12-52-24\JSTCHIRAL C2 20IPA.M
Last changed    : 7/10/2009 2:09:00 PM by VJS
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-09-25 12-52-24\JST-II-276-1.D\DA.M (JSTCHIRAL
                  C2 20IPA.M)
Last changed    : 10/17/2009 9:43:46 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-276-1, 20% IPA, AD-H
=====
  
```



Area Percent Report

```

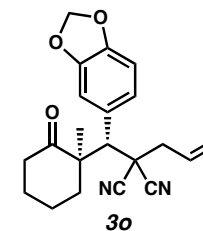
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
  
```

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.699	MM	0.1129	276.05334	40.73826	79.2342
2	3.142	MM	0.1584	72.34846	7.61422	20.7658

Totals : 348.40181 48.35248

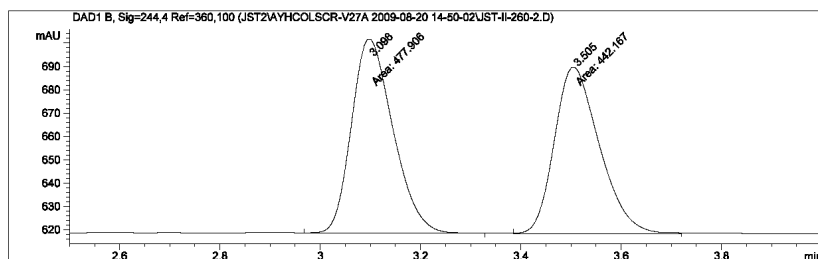
*** End of Report ***



Data File C:\CHEM32\1\DATA\JST2\AYHCOLSCR-V27A 2009-08-20 14-50-02\JST-II-260-2.D
 Sample Name: jst-ii-260-2

```

=====
Acq. Operator   : jst                      Seq. Line : 20
Acq. Instrument : Instrument 1              Location  : P1-E-04
Injection Date  : 8/20/2009 5:27:07 PM     Inj       : 1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\AYHCOLSCR-V27A 2009-08-20 14-50-02\JSTCHIRAL C2 20IPA.M
Last changed    : 7/10/2009 2:09:00 PM by VJS
Analysis Method : C:\CHEM32\1\DATA\JST2\AYHCOLSCR-V27A 2009-08-20 14-50-02\JST-II-260-1.D\DA.M (
                  JSTCHIRAL C2 20IPA.M)
Last changed    : 10/17/2009 9:49:24 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-260-2, 20% IPA, AD-H
=====
  
```



Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
  
```

Signal 1: DAD1 B, Sig=244,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.098	MM	0.0953	477.90616	83.61041	51.9422
2	3.505	MM	0.1030	442.16721	71.53754	48.0578

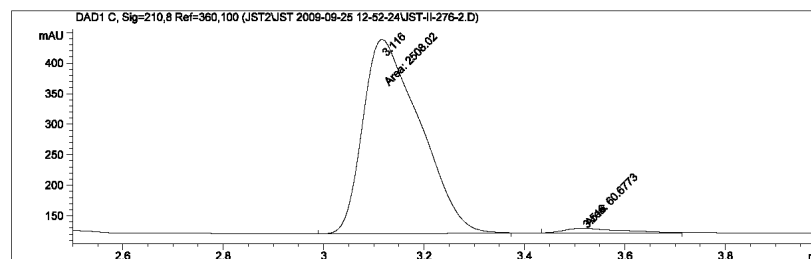
Totals : 920.07336 155.14795

*** End of Report ***

Data File C:\CHEM32\1\DATA\JST2\JST 2009-09-25 12-52-24\JST-II-276-2.D
 Sample Name: jst-ii-276-2

```

=====
Acq. Operator   : jst                      Seq. Line : 14
Acq. Instrument : Instrument 1              Location  : P1-F-06
Injection Date   : 9/25/2009 2:55:37 PM     Inj       : 1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-09-25 12-52-24\JSTCHIRAL C2 20IPA.M
Last changed    : 7/10/2009 2:09:00 PM by VJS
Analysis Method : C:\CHEM32\1\DATA\JST2\AYHCOLSCR-V27A 2009-08-20 14-50-02\JST-II-260-1.D\DA.M (
                  JSTCHIRAL C2 20IPA.M)
Last changed    : 10/17/2009 9:51:22 PM by jst
                  (modified after loading)
Sample Info     : jst-ii-276-2, 20% IPA, AD-H
=====
  
```



Area Percent Report

```

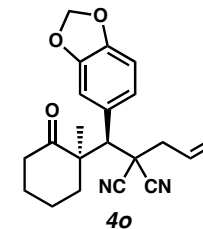
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
  
```

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.116	MF	0.1311	2508.02026	318.86459	97.6378
2	3.516	FM	0.1301	60.67731	7.77173	2.3622

Totals : 2568.69757 326.63632

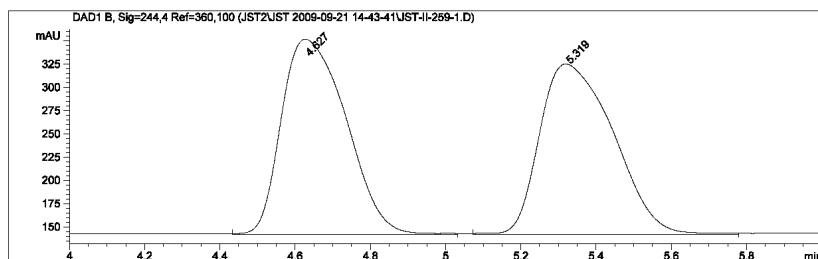
*** End of Report ***



Data File C:\CHEM32\1\DATA\JST2\JST 2009-09-21 14-43-41\JST-II-259-1.D
 Sample Name: jst-ii-259-1

```

=====
Acq. Operator   : jst                      Seq. Line :    3
Acq. Instrument : Instrument 1              Location  : P1-F-01
Injection Date  : 9/21/2009 2:51:52 PM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method    : C:\Chem32\1\DATA\JST2\JST 2009-09-21 14-43-41\JSTCHIRAL C2 10IPA.M
Last changed   : 5/8/2009 10:50:08 AM by RN
Analysis Method: C:\CHEM32\1\DATA\JST2\AYHCOLSCR-V27A 2009-08-20 14-50-02\JST-II-260-1.D\DA.M (
                JSTCHIRAL C2 20IPA.M)
Last changed   : 10/17/2009 9:52:53 PM by jst
                (modified after loading)
Sample Info    : jst-ii-259-1, AD-H 10% IPA
=====
  
```



Area Percent Report

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 B, Sig=244,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.627	BB	0.1972	2507.70142	210.16379	50.0262
2	5.319	BB	0.2272	2505.07739	183.60765	49.9738

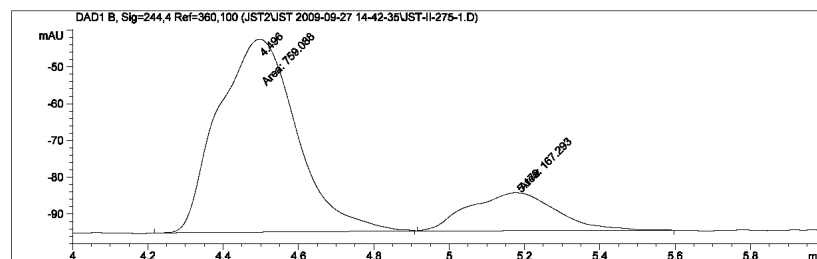
Totals : 5012.77881 393.77144

*** End of Report ***

Data File C:\CHEM32\1\DATA\JST2\JST 2009-09-27 14-42-35\JST-II-275-1.D
 Sample Name: jst-ii-275-1

```

=====
Acq. Operator   : jst                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : P1-F-03
Injection Date   : 9/27/2009 2:47:54 PM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method    : C:\Chem32\1\DATA\JST2\JST 2009-09-27 14-42-35\JSTCHIRAL C2 10IPA.M
Last changed   : 5/8/2009 10:50:08 AM by RN
Analysis Method: C:\CHEM32\1\DATA\JST2\JST 2009-09-27 14-42-35\JST-II-275-1.D\DA.M (JSTCHIRAL
                C2 10IPA.M)
Last changed   : 10/17/2009 9:55:26 PM by jst
                (modified after loading)
Sample Info    : jst-ii-275-1, 10% IPA, AD-H
=====
  
```



Area Percent Report

```

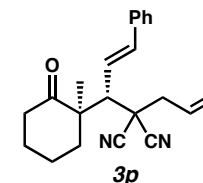
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 B, Sig=244,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.496	MM	0.2411	759.08752	52.48259	81.9412
2	5.179	MM	0.2695	167.29295	10.34753	18.0588

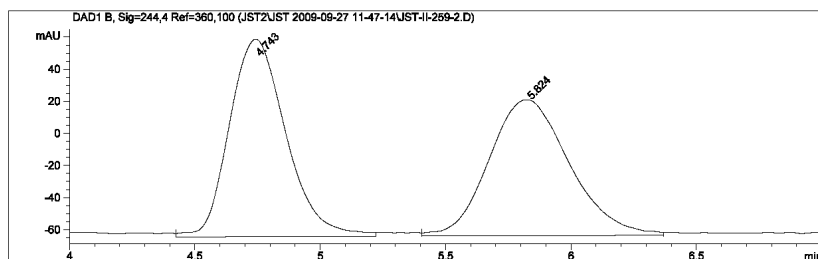
Totals : 926.38048 62.83012

*** End of Report ***



Data File C:\CHEM32\1\DATA\JST2\JST 2009-09-27 11-47-14\JST-II-259-2.D
 Sample Name: jst-ii-259-2

```
=====
Acq. Operator   : jst                      Seq. Line :    3
Acq. Instrument : Instrument 1              Location  : P1-E-01
Injection Date  : 9/27/2009 12:04:40 PM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-09-27 11-47-14\JSTCHIRAL C6 10IPA.M
Last changed    : 9/27/2009 11:44:40 AM by AYH
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-09-27 14-42-35\JST-II-275-1.D\DA.M (JSTCHIRAL
                C2 10IPA.M)
Last changed    : 10/17/2009 9:56:55 PM by jst
                (modified after loading)
Sample Info     : jst-ii-259-2, 10% IPA, OB-H
=====
```



Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 B, Sig=244,4 Ref=360,100

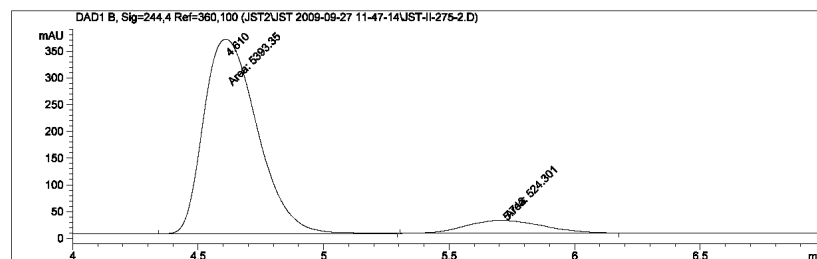
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.743	VB	0.2405	1926.25781	123.12312	50.4662
2	5.824	VV	0.3461	1890.66602	84.81303	49.5338

Totals : 3816.92383 207.93615

*** End of Report ***

Data File C:\CHEM32\1\DATA\JST2\JST 2009-09-27 11-47-14\JST-II-275-2.D
 Sample Name: jst-ii-275-2

```
=====
Acq. Operator   : jst                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : P1-F-04
Injection Date   : 9/27/2009 11:47:40 AM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-09-27 11-47-14\JSTCHIRAL C6 10IPA.M
Last changed    : 9/27/2009 11:44:40 AM by AYH
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-09-27 14-42-35\JST-II-275-1.D\DA.M (JSTCHIRAL
                C2 10IPA.M)
Last changed    : 10/17/2009 9:58:07 PM by jst
                (modified after loading)
Sample Info     : jst-ii-275-2, 10% IPA, OB-H
=====
```



Area Percent Report

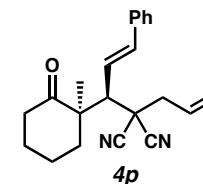
```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 B, Sig=244,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.610	MM	0.2470	5393.34912	363.96295	91.1400
2	5.713	MM	0.3646	524.30115	23.96499	8.8600

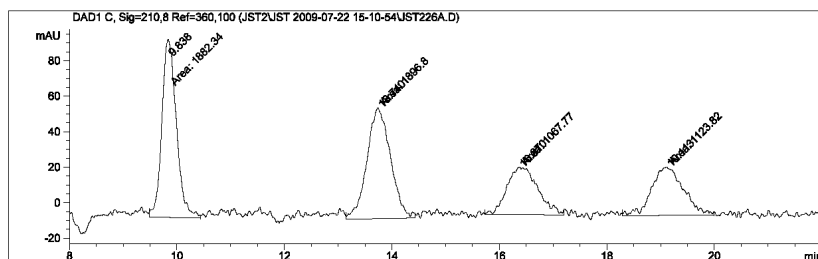
Totals : 5917.65027 387.92795

*** End of Report ***



Data File C:\CHEM32\1\DATA\JST2\JST 2009-07-22 15-10-54\JST226A.D
 Sample Name: jst226-4

```
=====
Acq. Operator   : jst                      Seq. Line :    5
Acq. Instrument : Instrument 1              Location  : P1-F-09
Injection Date  : 7/22/2009 3:25:09 PM     Inj       :    1
                                           Inj Volume: 15 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-07-22 15-10-54\JSTCHIRAL C4 I1PA.M
Last changed    : 7/22/2009 3:10:02 PM by jst
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-09-27 14-42-35\JST-II-275-1.D\DA.M (JSTCHIRAL
C2 I1PA.M)
Last changed    : 10/17/2009 10:06:22 PM by jst
                 (modified after loading)
Sample Info     : jst-ii-226-4, OJ-H, 21% IPA, 2ml/min
=====
```



Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

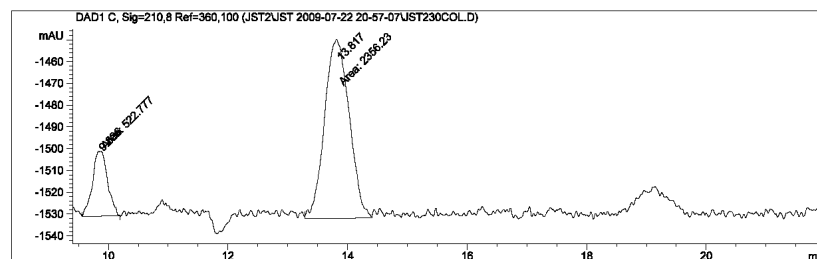
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.838	MM	0.3120	1882.33606	100.54717	31.5261
2	13.740	MM	0.5057	1896.79797	62.51189	31.7683
3	16.370	MM	0.6678	1067.76526	26.65067	17.8834
4	19.113	MM	0.6874	1123.82312	27.24824	18.8222

Totals : 5970.72241 216.95797

*** End of Report ***

Data File C:\CHEM32\1\DATA\JST2\JST 2009-07-22 20-57-07\JST230COL.D
 Sample Name: jst230col

```
=====
Acq. Operator   : jst                      Seq. Line :    5
Acq. Instrument : Instrument 1              Location  : P1-F-09
Injection Date   : 7/22/2009 9:11:23 PM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : C:\Chem32\1\DATA\JST2\JST 2009-07-22 20-57-07\JSTCHIRAL C4 I1PA2.M
Last changed    : 7/22/2009 8:46:12 PM by jst
Analysis Method : C:\CHEM32\1\DATA\JST2\JST 2009-07-22 20-57-07\JST230COL.D\DA.M (JSTCHIRAL C4
I1PA2.M)
Last changed    : 10/17/2009 10:17:40 PM by jst
                 (modified after loading)
Sample Info     : jst230col, 18%ipa, OJ-H, 30 min run
=====
```



Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.836	MM	0.2923	522.77698	29.81277	18.1583
2	13.817	MM	0.4775	2356.22510	82.24318	81.8417

Totals : 2879.00208 112.05595

*** End of Report ***

