Supplementary Information for:

Magnesium-catalysed Hydroboration of Isonitriles

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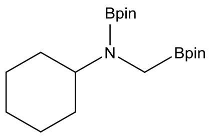
General experimental procedures

All reactions dealing with air- and moisture-sensitive compounds were carried out under an argon atmosphere using standard Schlenk line and glovebox techniques in an MBraun Labmaster glovebox at O_2, $H_2O < 0.1$ ppm. NMR experiments using air-sensitive compounds were conducted in J. Youngs tap NMR tubes prepared and sealed in a glovebox under argon. All NMR data were acquired on a Bruker 300 Ultrashield^{TM} for ^{1}H (300 MHz), ^{13}C{^{1}H} (75.48 MHz) and ^{11}B (96.3 MHz) NMR spectra at room temperature or a Bruker 400 Ultrashield^{TM} for ^{1}H (400 MHz) and ^{13}C{^{1}H} (125.76 MHz) spectra. ^{1}H/^{13}C NMR spectra were referenced using residual solvent resonances. Elemental analyses of all moisture- and air-sensitive compounds were performed by Stephen Boyer of London Metropolitan Enterprises. Solvents for air- and moisture-sensitive reactions were provided by an Innovative Technology Solvent Purification System. C_6D_6 and toluene-d_8 were purchased from Fluorochem and dried over molten potassium prior to vacuum transfer into a sealed ampoule and storage in the glovebox under argon. Pinacolborane and all isonitrile substrates were purchased from Sigma Aldrich Ltd. Compounds **1** and **8** were synthesised using a literature procedures.[1]

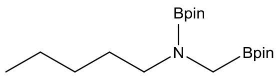
General Procedure for NMR scale catalytic reactions: 5 mg (0.01 mmol, i.e. 5 mol%) of **1** was dissolved in 0.5 ml of C_6D_6, 60.9 μL (0.42 mmol) of pinacolborane was then added followed by 0.2 mmol of isonitrile. This mixture was transferred to a sealed Youngs tap NMR tube and the reaction was heated in an oil bath at 60 °C. The reactions were regularly monitored by ^{1}H and ^{11}B NMR spectroscopy until complete conversion was observed. Due to the unbearable stench of residual isonitrile substrates (the Bath Chemistry Research building was evacuated early in this study) the hydroboration products were not isolated.

***N*,*C*-{B(OCMe$_2$)$_2$}$_2$-*N*-methylcyclohexylamine, 2. NMR scale:** 24.8 μL of cyclohexylisonitrile.



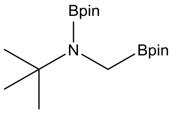
^{1}H NMR (300 MHz, C_6D_6): 3.32 (1H, m, C*H*(Cy)), 2.81 (2H, s, NC*H*$_2$B), 1.89 – 1.33 (10H, m, Cy-*H*), 1.15 (12H, s, NB(OC(C*H*$_3$)$_2$)$_2$), 1.10 (12H, s, CB(OC(C*H*$_3$)$_2$)$_2$). ^{13}C{^{1}H} NMR (75 MHz, C_6D_6): 83.4 NB(O*C*(CH$_3$)$_2$)$_2$), 83.2 (CB(O*C*(CH$_3$)$_2$)$_2$), 57.0 (N*C*H$_2$B), 33.0 (*C*H-Cy), 27.1 (Cy-*C*), 26.9 (Cy-*C*), 26.4 (Cy-*C*), 25.3 (NB(OC(*C*H$_3$)$_2$)$_2$), 25.2 CB(OC(*C*H$_3$)$_2$)$_2$). ^{11}B NMR (96 MHz, C_6D_6): 37.5 C*B*, 27.8 N*B*.

***N*,*C*-{B(OCMe$_2$)$_2$}$_2$-*N*-methyl-1-pentylamine, 3. NMR scale:** 25.1 μL of 1-pentylisonitrile.



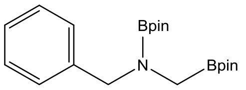
^{1}H NMR (300 MHz, C_6D_6): 3.22 (2H, t, J_{HH} = 9 Hz, NCH_2CH_2), 2.90 (2H, s, NCH_2B), 1.50 (2H, m, NCH_2CH_2), 1.27 (4H, m, $NCH_2CH_2(CH_2)_2$), 1.13 (12H, s, $NB(OC(CH_3)_2)_2$), 1.06 (12H, $CB(OC(CH_3)_2)_2$), 0.86 (3H, t, J_{HH} = 6 Hz, $N(CH_2)_4CH_3$). $^{13}C\{^1H\}$ NMR (75 MHz, C_6D_6): 83.5 ($NB(OC(CH_3)_2)_2$), 82.3 ($CB(OC(CH_3)_2)_2$), 49.2 (NCH_2B), 29.5 (NCH_2CH_2), 29.2 (NCH_2CH_2), 25.3 ($NB(OC(CH_3)_2)_2$), 25.2 ($CB(OC(CH_3)_2)_2$), 23.3 ($NCH_2CH_2(CH_2)_2$), 14.8 ($N(CH_2)_4CH_3$). ^{11}B NMR (96 MHz, C_6D_6): 37.2 C*B*, 27.9 N*B*.

***N*,*C*-{B(OCMe$_2$)$_2$}$_2$-*N*-methyl-tert-butylamine, 4. NMR scale:** 22.6 μL of *tert*-butylisonitrile.



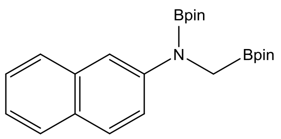
^{1}H NMR (300 MHz, C_6D_6): 2.90 (2H, s, NCH_2B), 1.39 (9H, s, $NC(CH_3)_3$), 1.14 (12H, s, $NB(OC(CH_3)_2)_2$), 1.09 (12H, s, $CB(OC(CH_3)_2)_2$). $^{13}C\{^1H\}$ NMR (75 MHz, C_6D_6): 83.5 ($NB(OC(CH_3)_2)_2$), 83.3 ($CB(OC(CH_3)_2)_2$), 81.6 ($NC(CH_3)_3$), 53.1 (NCH_2B), 30.8 ($NC(CH_3)_3$), 25.3 ($NB(OC(CH_3)_2)_2$), 25.2 ($CB(OC(CH_3)_2)_2$). ^{11}B NMR (96 MHz, C_6D_6): 36.9 C*B*, 26.9 N*B*.

***N*,*C*-{B(OCMe$_2$)$_2$}$_2$-*N*-methylbenzylamine, 5. NMR scale:** 24.3 μL of benzylisonitrile.



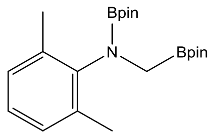
^{1}H NMR (300 MHz, C_6D_6): 7.45 (2H, d, J_{HH} = 6 Hz, *o*-C*H*), 7.27 (2H, m, *m*-C*H*), 7.17 (1H, m, *p*-C*H*), 4.53 (2H, s, $BnCH_2N$), 2.93 (NCH_2B), 1.24 (12H, s, $NB(OC(CH_3)_2)_2$), 1.11 (12H, s, $CB(OC(CH_3)_2)_2$). $^{13}C\{^1H\}$ NMR (75 MHz, C_6D_6): 141.4 (*ipso-C*), 129.3 (*o-C*), 128.9 (*p-C*), 128.7 (*m-C*), 127.2 ($BnCH_2N$), 83.5 ($NB(OC(CH_3)_2)_2$), 82.7 ($CB(OC(CH_3)_2)_2$), 53.3 (NCH_2B), 25.3 ($NB(OC(CH_3)_2)_2$), 25.2 ($CB(OC(CH_3)_2)_2$). ^{11}B NMR (96 MHz, C_6D_6): 37.4 C*B*, 28.3 N*B*.

***N*,*C*-{B(OCMe$_2$)$_2$}$_2$-*N*-methyl-2-napthylamine, 6. NMR scale:** 30.6 mg of 2-napthylisonitrile.



^{1}H NMR (300 MHz, C_6D_6): 8.26 (1H, m, *o-H*), 7.66 (2H, m, *o-H*, *m*-H), 7.27 (2H, m, Ar-*H*), 7.16 (2H, m, Ar-*H*), 3.61 (2H, s, NCH_2B), 1.12 (12H, s, $NB(OC(CH_3)_2)_2$), 0.93 (12H, s, $CB(OC(CH_3)_2)_2$). $^{13}C\{^1H\}$ NMR (75 MHz, C_6D_6): 161.9 (*ipso-C*), 147.1 (*o-C*), 143.1 (*o-C*), 135.4, 129.9, 127.5, 126.5, 123.9, 123.2, 113.6, 83.3 ($NB(OC(CH_3)_2)_2$), 82.2 ($CB(OC(CH_3)_2)_2$), 47.2 (NCH_2B), 25.1 ($NB(OC(CH_3)_2)_2$), 25.0 ($CB(OC(CH_3)_2)_2$). ^{11}B NMR (96 MHz, C_6D_6): 37.6 C*B*, 28.0 N*B*.

***N,C*-{B(OCMe$_2$)$_2$}$_2$-*N*-methyl-2,6-(methyl)phenylamine, 7. NMR scale:** 26.2 mg of 2,6-(methyl)phenylisonitrile.

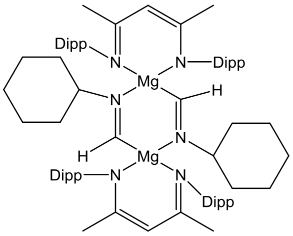


^{1}H NMR (300 MHz, C_6D_6): 6.76 (1H, m, *p*-C*H*), 6.62 (2H, m, *m*-C*H*), 3.03 (2H, s, NCH_2B), 2.46 (6H, s, *o*-CH_3), 1.03 (12H, s, NB(OC(CH_3)$_2$)$_2$), 1.00 (12H, s, CB(OC(CH_3)$_2$)$_2$). ^{13}C{^{1}H} NMR (75 MHz, C_6D_6): 146.8 (*ipso- C*), 136.9 (*o-C*), 128.9 (*p-C*), 128.2 (*m-C*), 83.6 (NB(O*C*(CH$_3$)$_2$)$_2$), 83.5 (CB(O*C*(CH$_3$)$_2$)$_2$), 47.2 (N*C*H$_2$B), 25.3 (NB(OC(*C*H$_3$)$_2$)$_2$), 25.2 (CB(OC(*C*H$_3$)$_2$)$_2$), 19.3 (*o*-*C*H$_3$). ^{11}B NMR (96 MHz, C_6D_6): 36.9 C*B*, 27.3 N*B*.

Stoichiometric reactions

Compound 9. ***NMR scale:*** Compound **8** (0.2 mmol, 100 mg) was dissolved in 0.5 ml of C_6D_6 along with CyNC (0.2 mmol, 29.8 μL). The insertion product formed cleanly at room temperature and colourless crystals of compound **9** precipitated from solution on standing.

Alternative synthesis of compound 9. ***NMR scale:*** Compound **1** (0.1 mmol, 50 mg) was dissolved in 0.5 ml of C_6D_6 along with HBpin (0.1 mmol, 14.5 μL). This reaction was left for 5 minutes to form the hydride before addition of CyNC (0.1 mmol, 12.4 μL). An intense blue solution was formed upon addition and upon leaving solution for half an hour colourless crystals of compound **9** precipitated from solution.

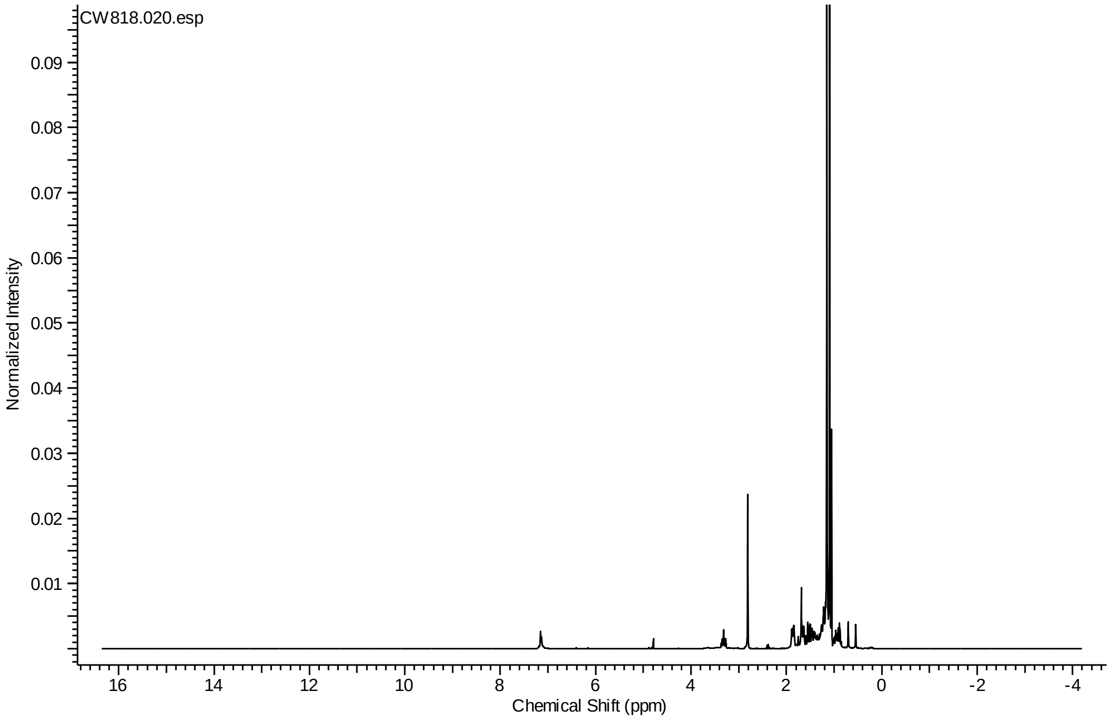


^{1}H NMR (300 MHz, C_6D_6): 9.99 (2H, s, MgC*H*), 7.14 – 7.00 (12H, m, Ar-*H*), 4.80 (2H, s, NC(CH$_3$)C*H*), 3.67 (4H, m, C*H*(CH$_3$)$_2$), 2.86 (4H, m, C*H*(CH$_3$)$_2$), 2.30 (2H, m, C*H*(Cy), 1.70 (12H, s, NC(C*H*$_3$)CH), 1.49 (12H, d, J_{HH} = 3 Hz, CH(CH_3)$_2$), 1.41 – 0.96 (20H, m, Cy-*H*), 1.24 (12H, d, J_{HH} = 3 Hz, CH(CH_3)$_2$), 0.87 (12H, d, J_{HH} = 3 Hz, CH(CH_3)$_2$), 0.22 (12H, d, J_{HH} = 3 Hz, CH(CH_3)$_2$). ^{13}C{^{1}H} NMR (75 MHz, C_6D_6): 217.0 (Mg*C*H), 169.6 (N*C*(CH$_3$)CH), 147.4 (*ipso-C*), 146.5 (*o-C*), 142.7 (*p-C*), 125.5 (*m-C*), 94.5 (NC(CH$_3$)*C*H), 35.8 (*C*HCy), 34.3 (*C*H(CH$_3$)$_2$), 32.3 (*C*H(CH$_3$)$_2$), 28.9 (NC(*C*H$_3$)$_2$), 27.2 (CH(*C*H$_3$)$_2$), 26.9 (*C*-Cy), 26.8 (*C*-Cy), 26.1 (CH(*C*H$_3$)$_2$), 25.0 (*C*-Cy), 24.7 (CH(*C*H$_3$)$_2$), 14.7 (CH(*C*H$_3$)$_2$). Despite repeated attempts, an acceptable elemental analysis could not be obtained for this highly air- and moisture-sensitive compound.

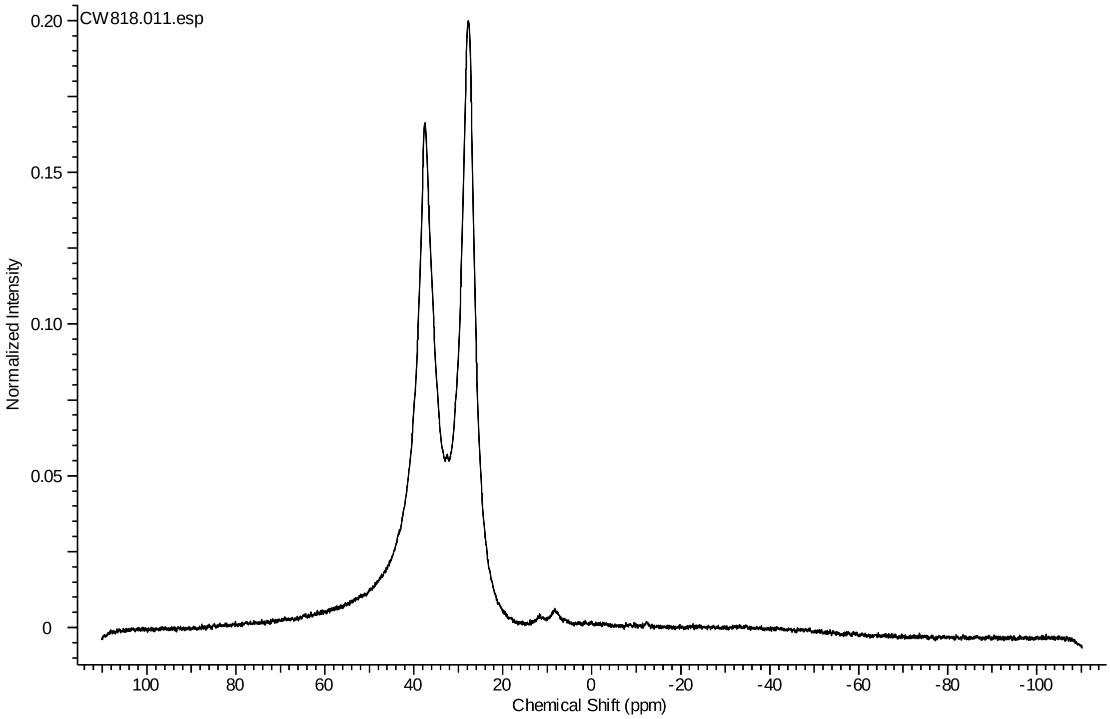
NMR Spectra

Compound 2

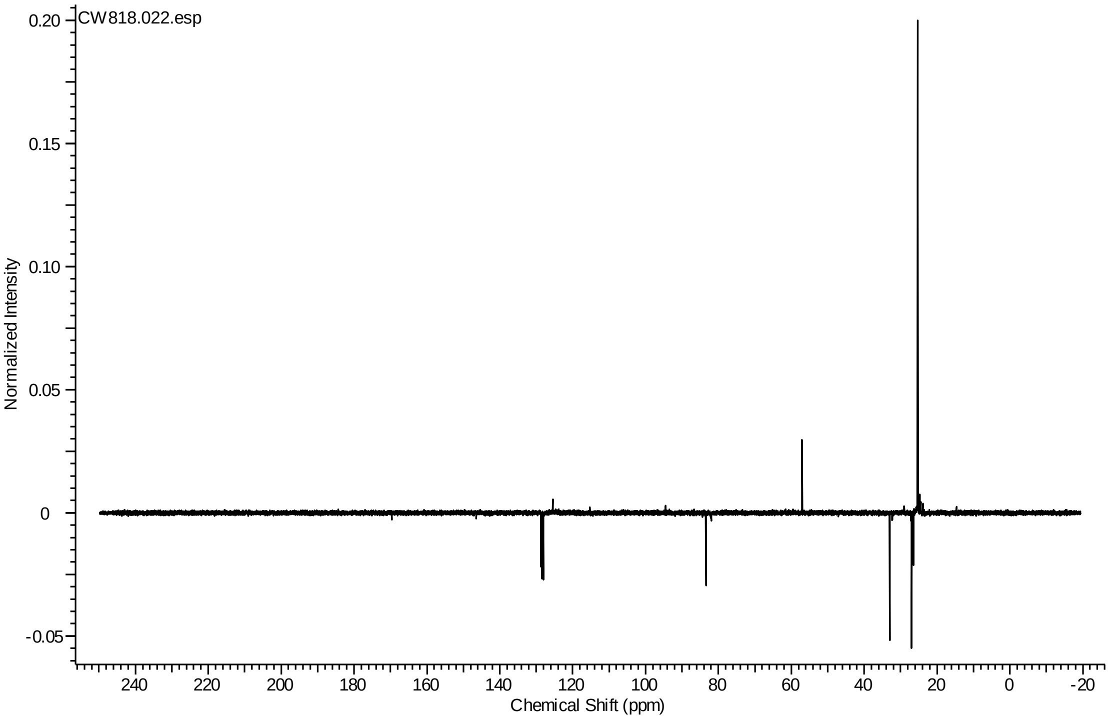
^{1}H



^{11}B

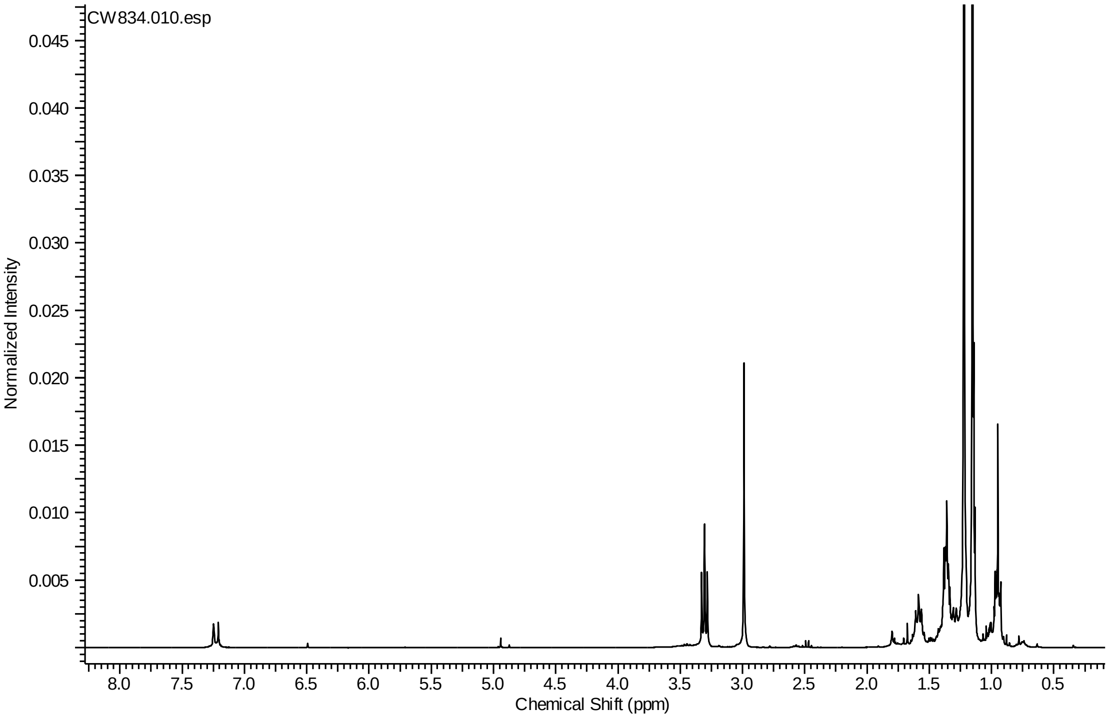


^{13}C (PENDANT)

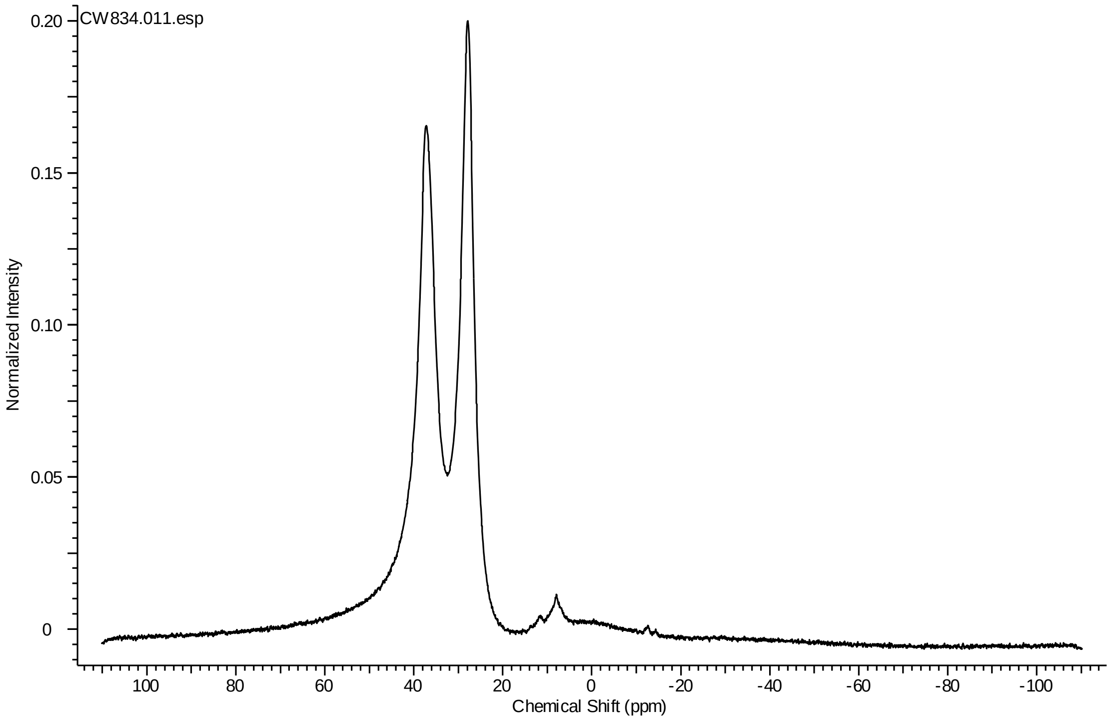


Compound 3

^{1}H



^{11}B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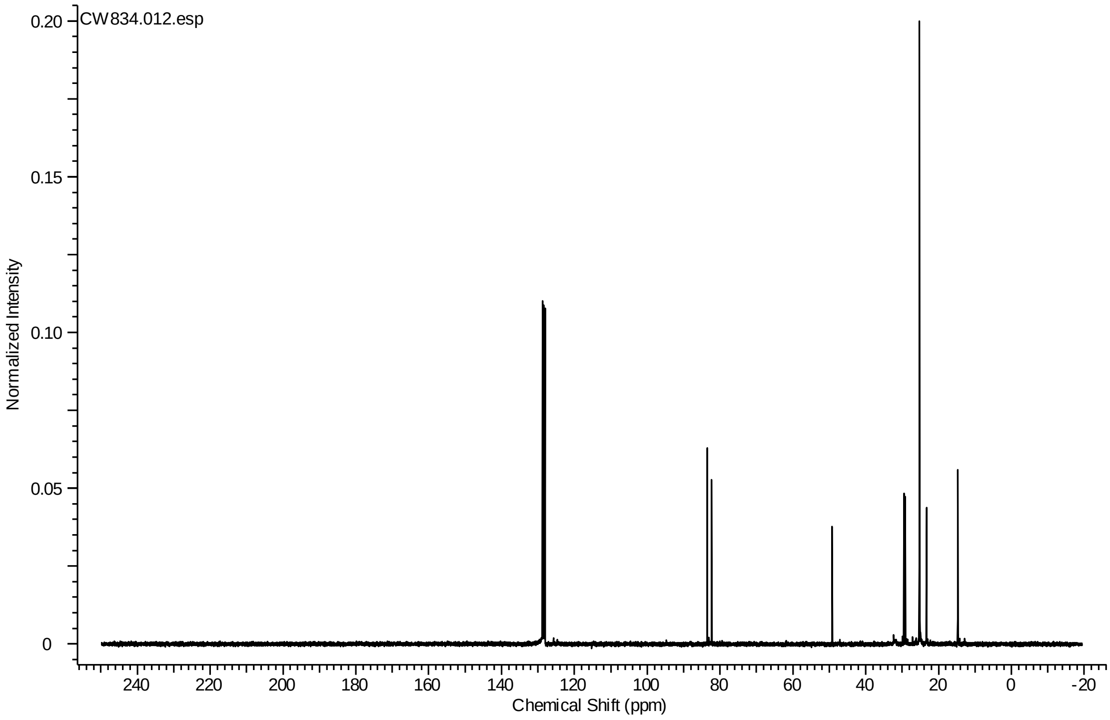
-60
-80
-100
Chemical Shift (ppm)

^{13}C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20
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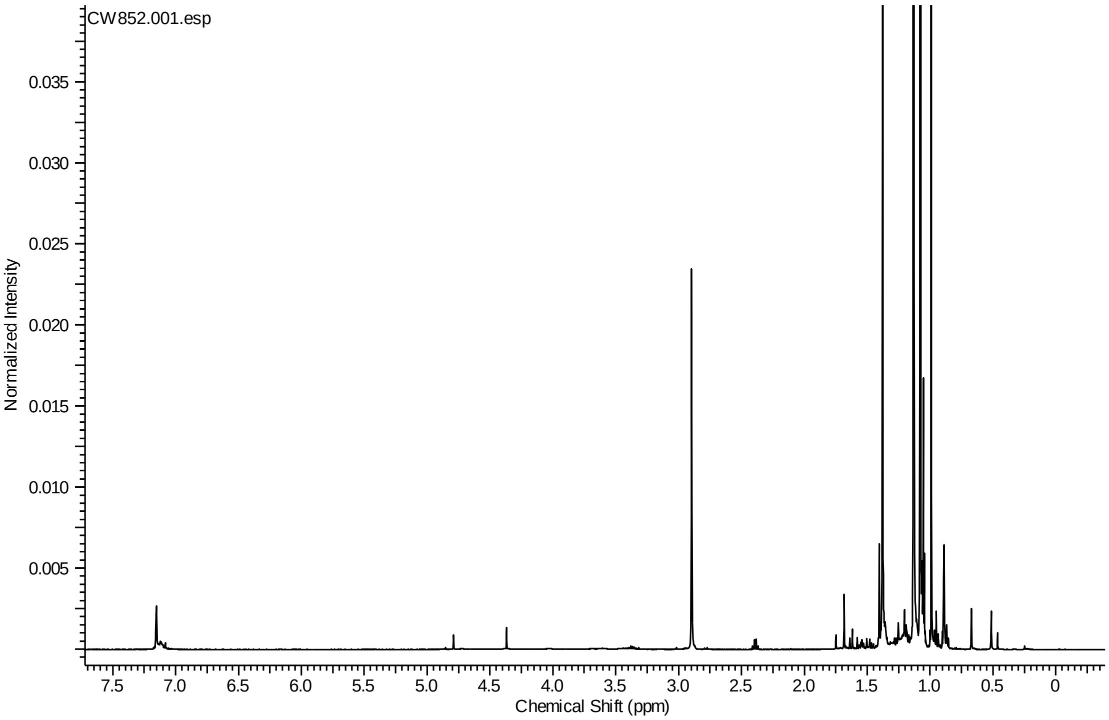
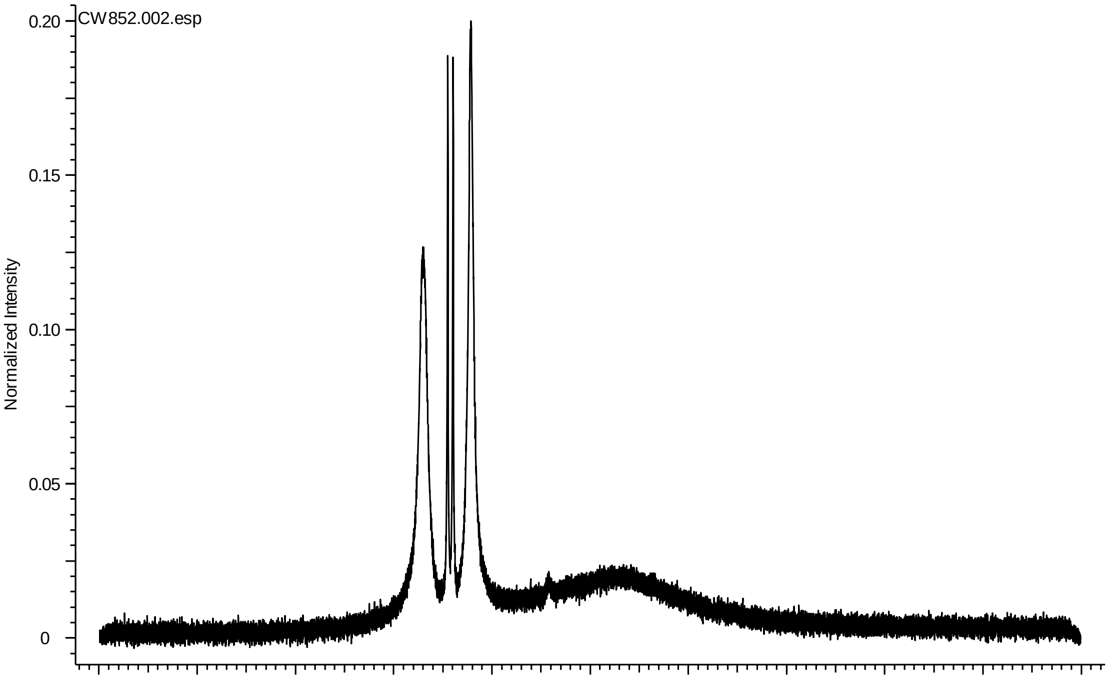
Chemical Shift (ppm)

Compound 4

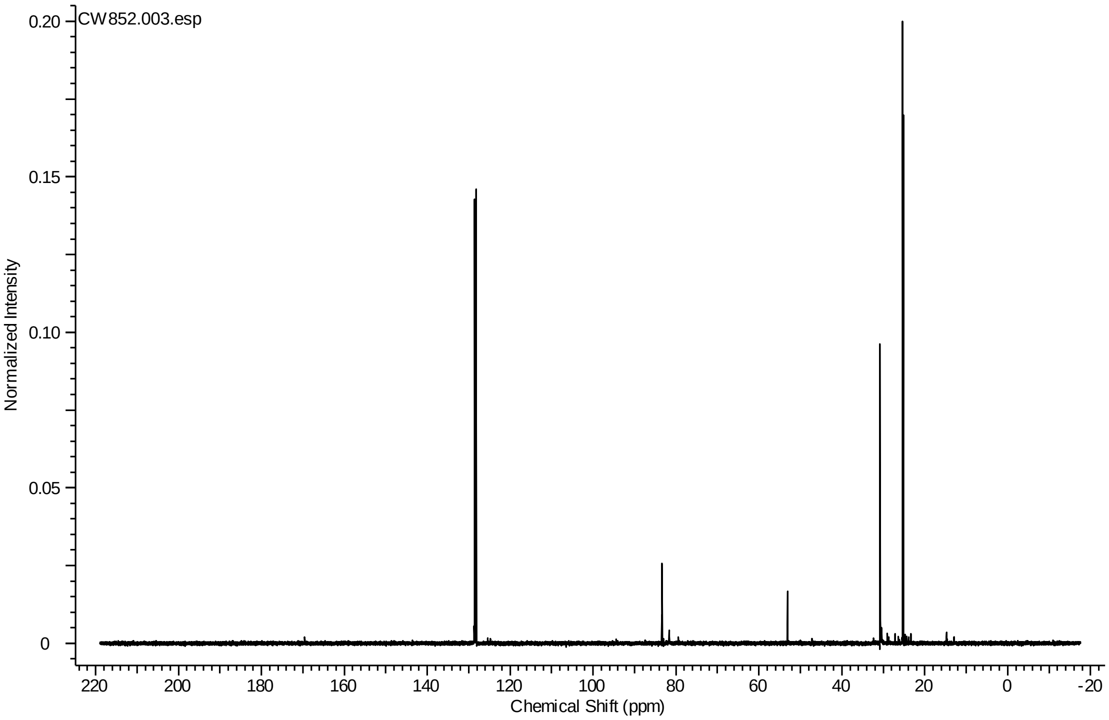
^{1}H



^{11}B

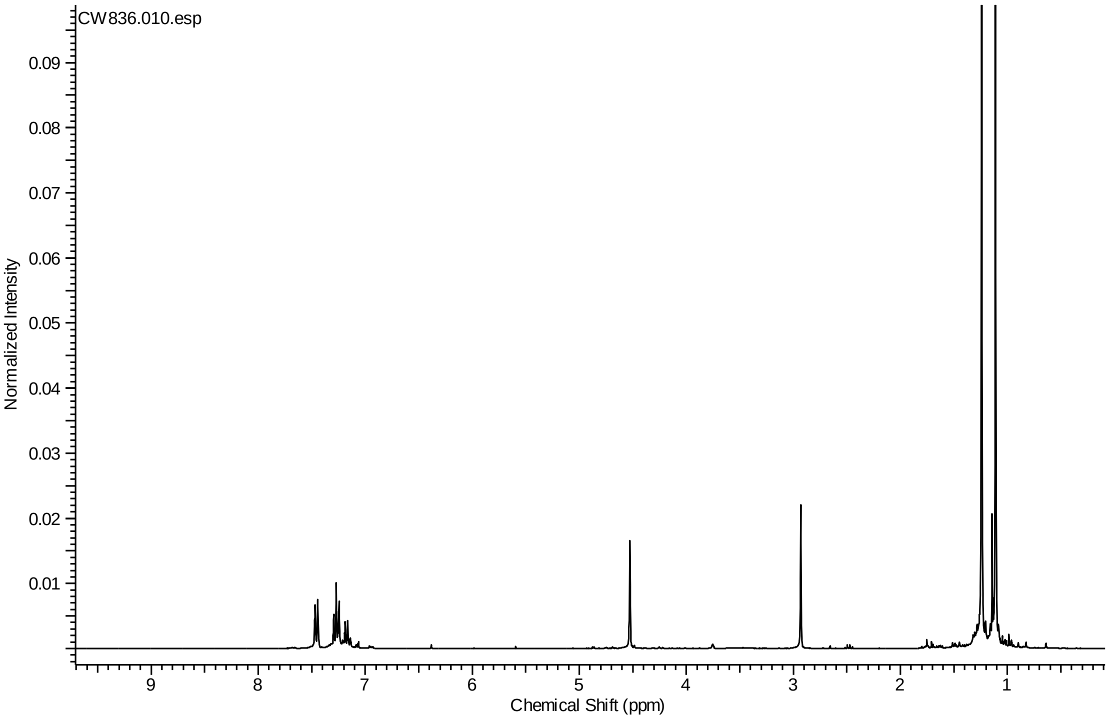


^{13}C

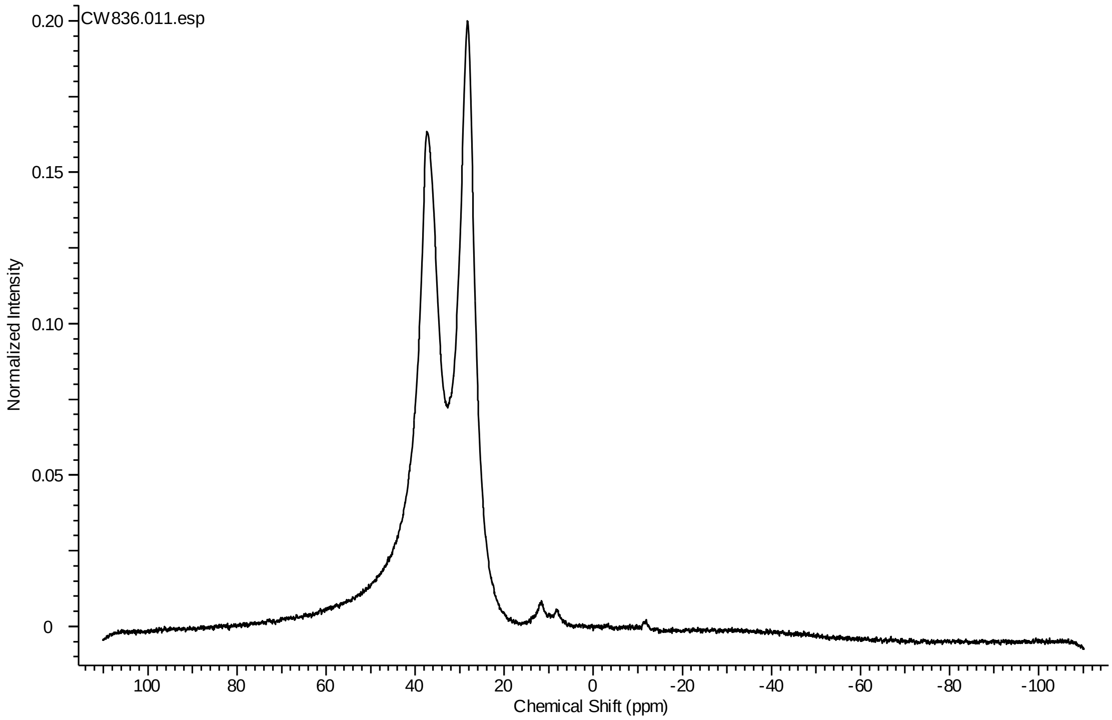


Compound 5

^{1}H



^{11}B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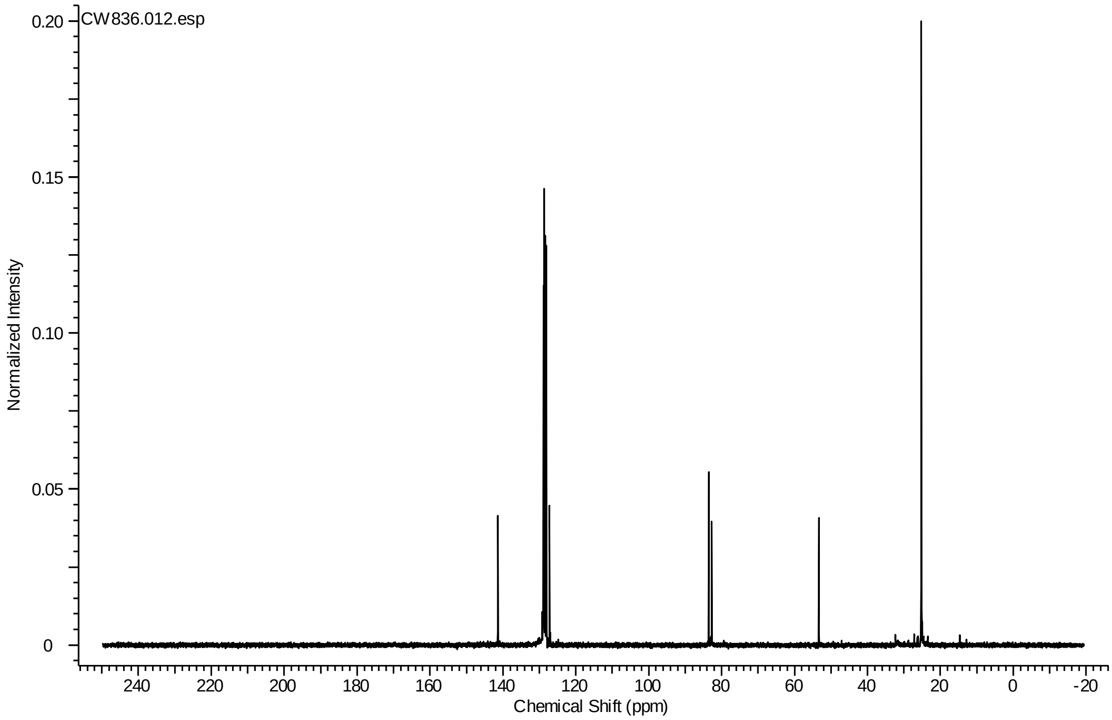
Chemical Shift (ppm)

^{13}C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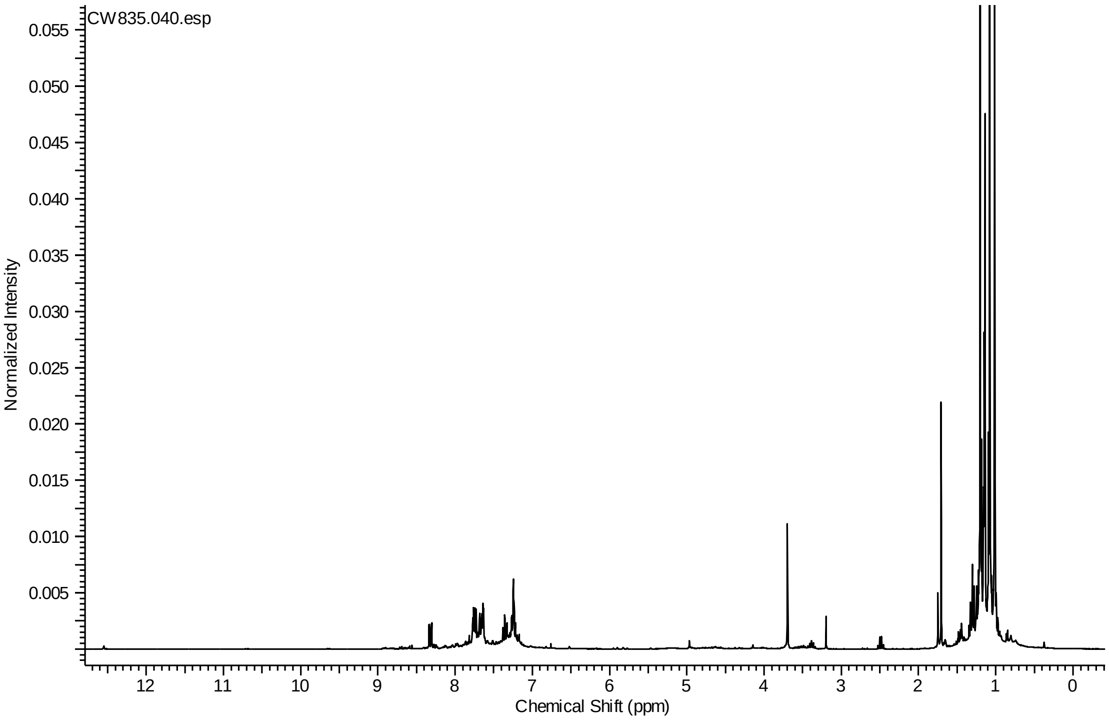
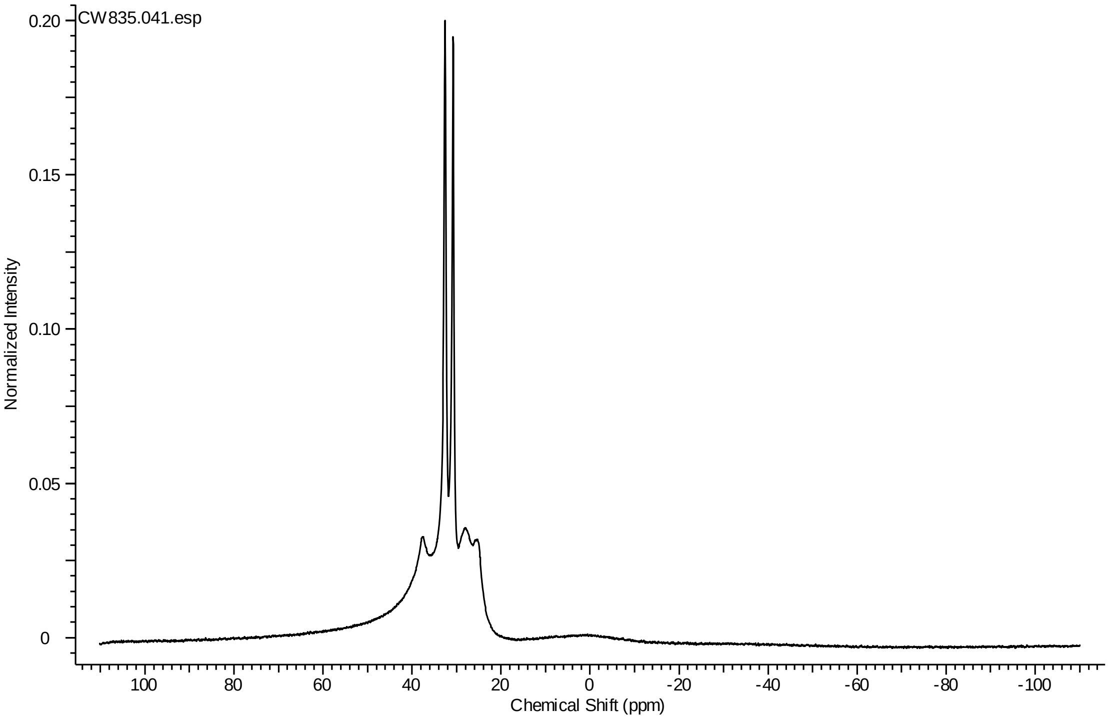
Chemical Shift (ppm)

Compound 6

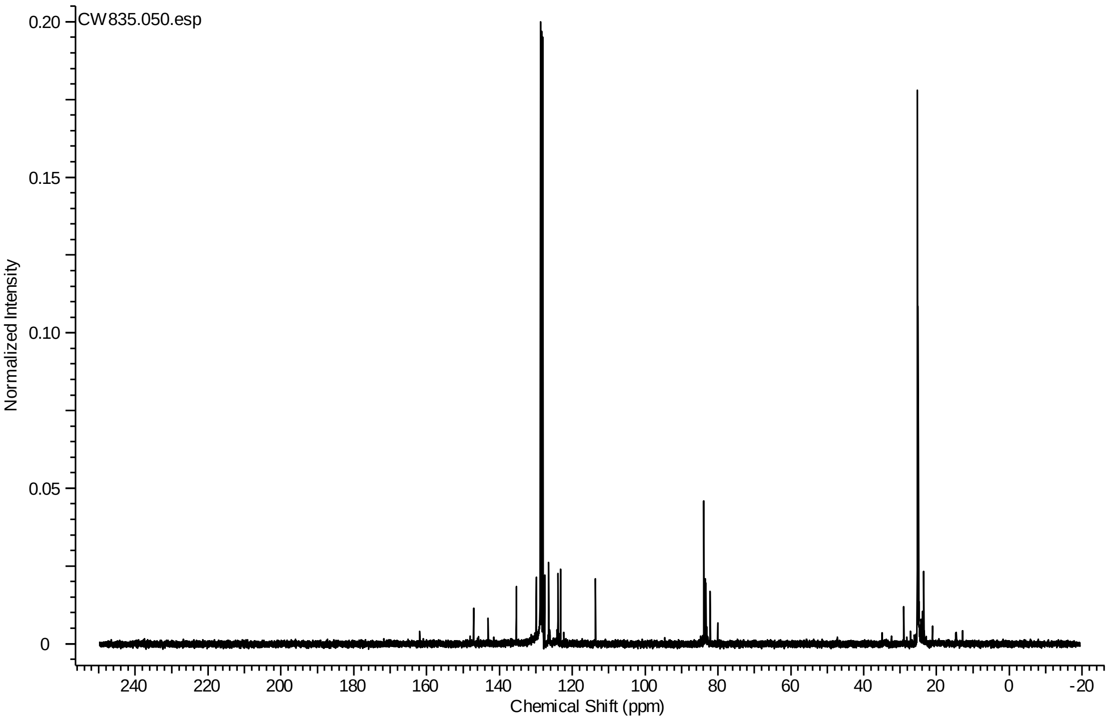
^{1}H



^{11}B

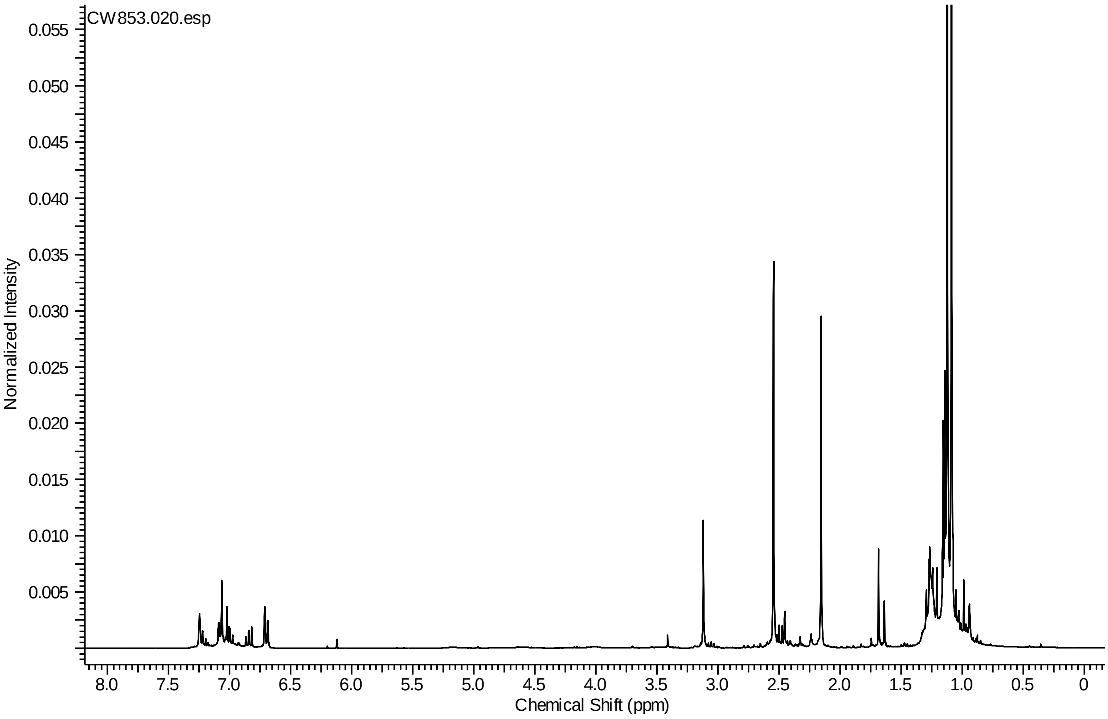


^{13}C

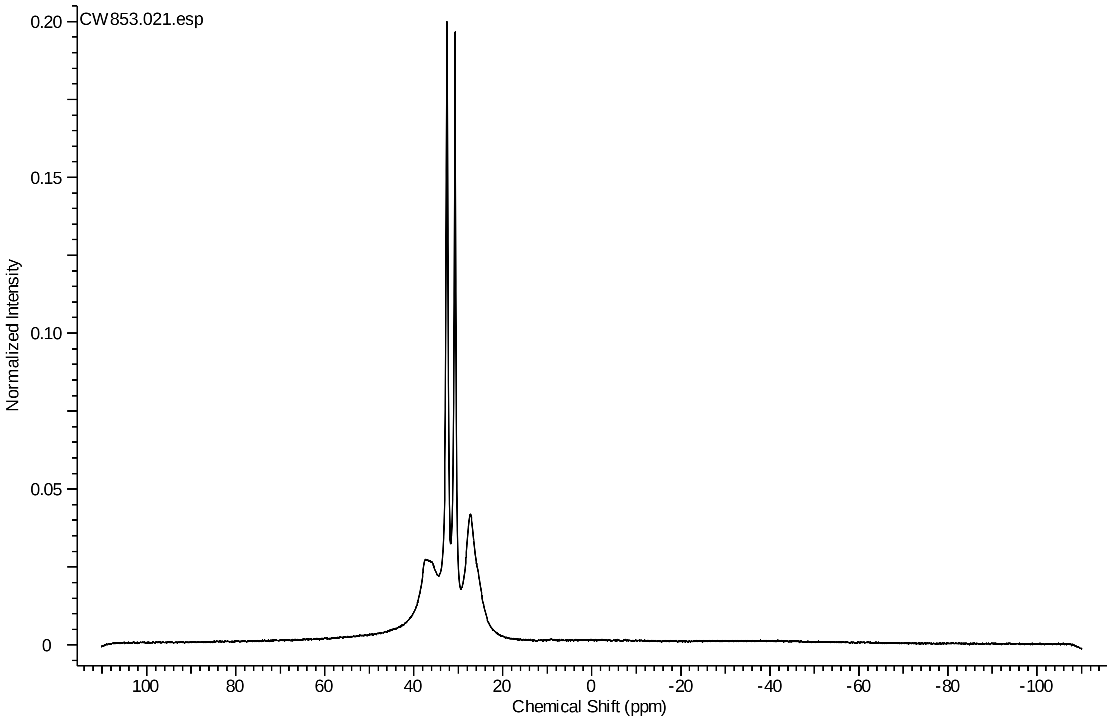


Compound 7

^{1}H



^{11}B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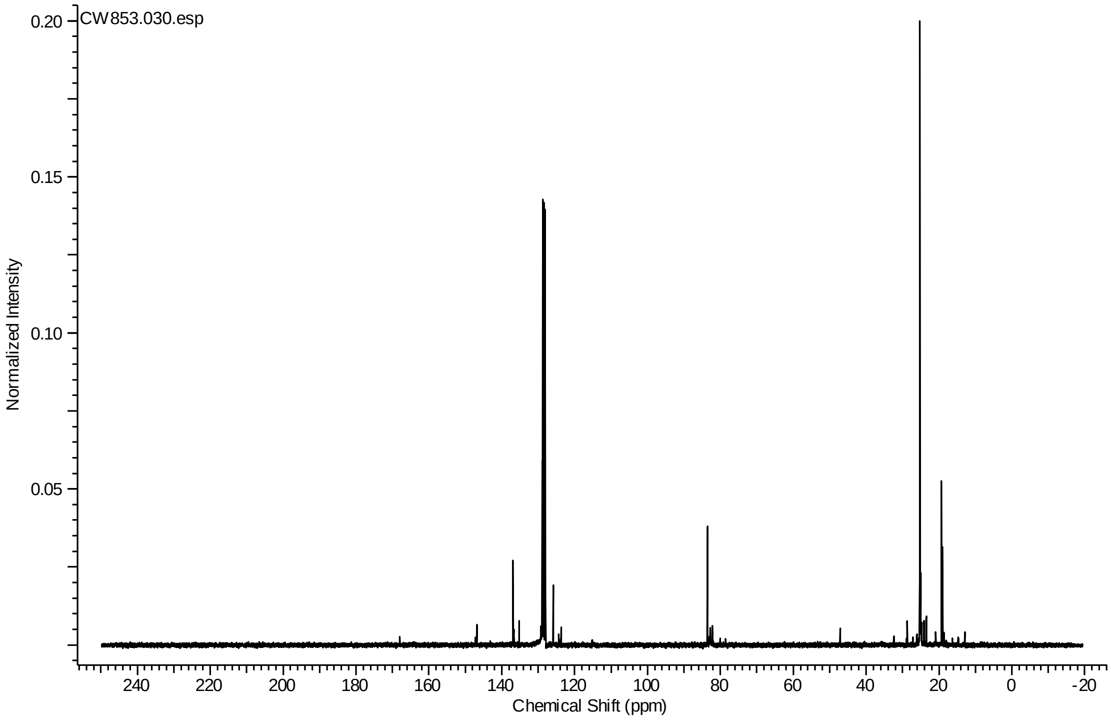
Chemical Shift (ppm)

^{13}C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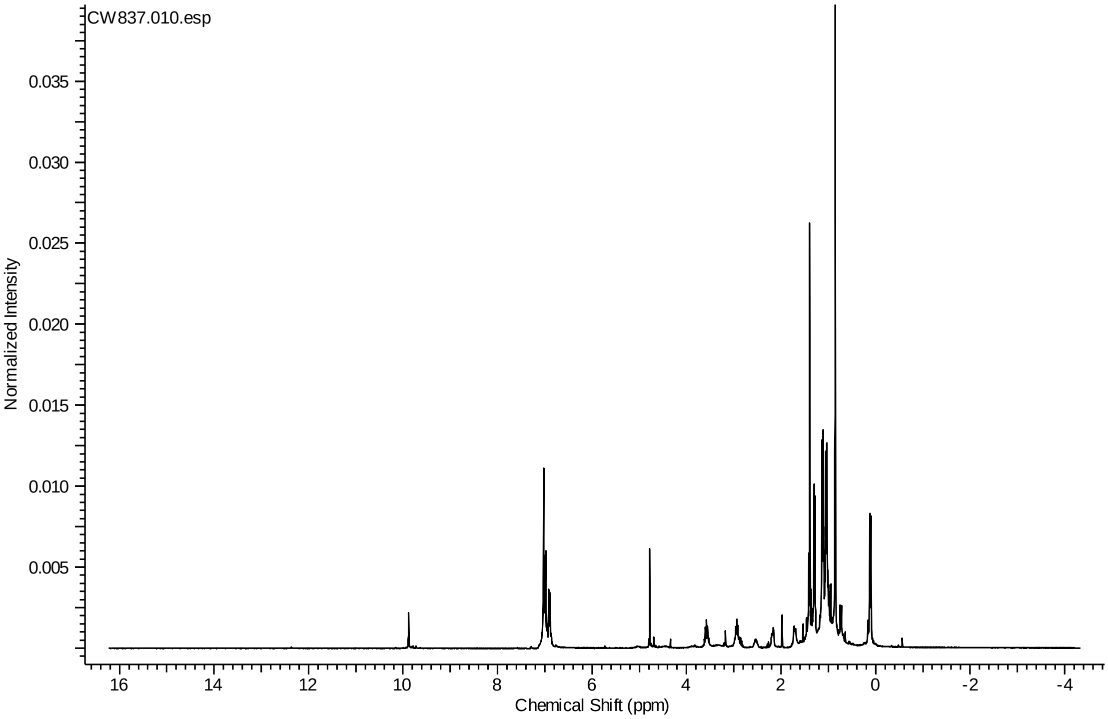
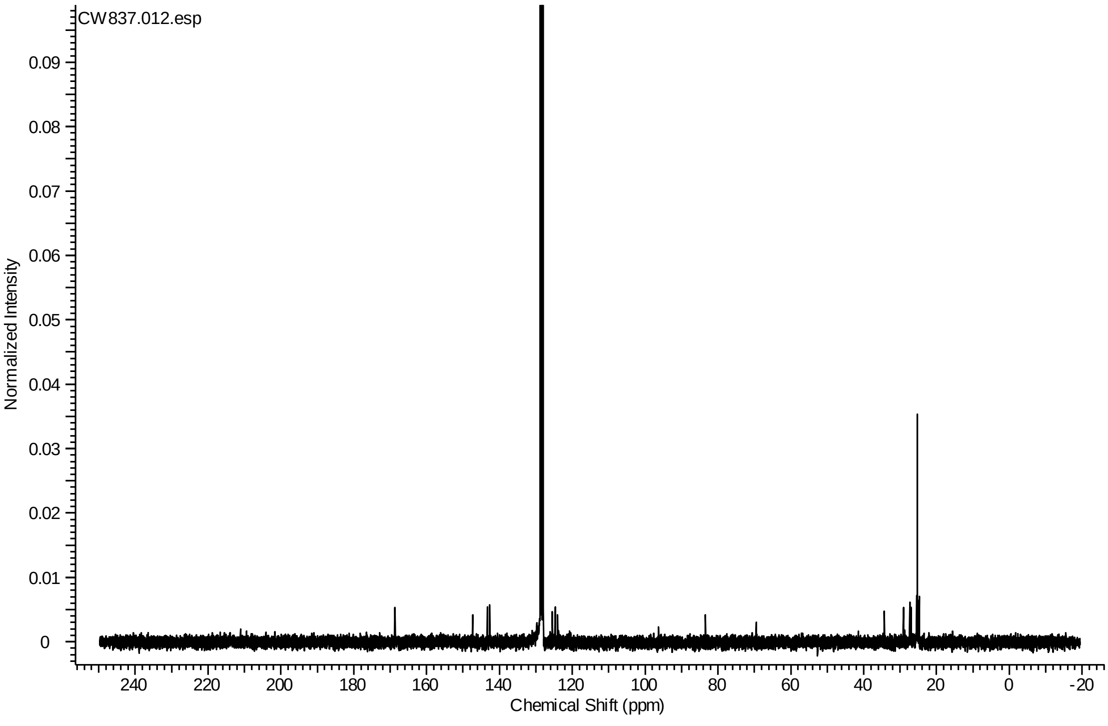
Chemical Shift (ppm)

Compound 9

^{1}H



^{13}C



Catalysis studies

Table S1: Optimisation study of CyNC hydroboration

Entry	Isocyanide	Catalyst (mol %)	Time (hrs)	Temp (°C)	NMR yield (%)
1	Cy	5	1	RT	7
2	Cy	5	1	40	15
3	Cy	5	1	50	30
4	Cy	5	1	60	>99
5	Cy	10	0.5	60	>99
6	Cy	1		60	

Table S2: Hydroboration of various isocyanides

Entry	Isocyanide	Catalyst (mol %)	Time (hrs)	Temp (°C)	NMR yield (%)	Isolated yield (%)
7	CyNC	5	1	60	>99	
8	$CH_3(CH_2)_4NC$	5	1	60	>99	
9	tBuNC	5	1	60	>99	
10	$BnCH_2NC$	5	0.5	60	>99	
11	2-NapthylNC	5	48	100	55	-
12	2,6-$(CH_3)C_6H_4NC$	5	48	100	53	-

Kinetic Studies

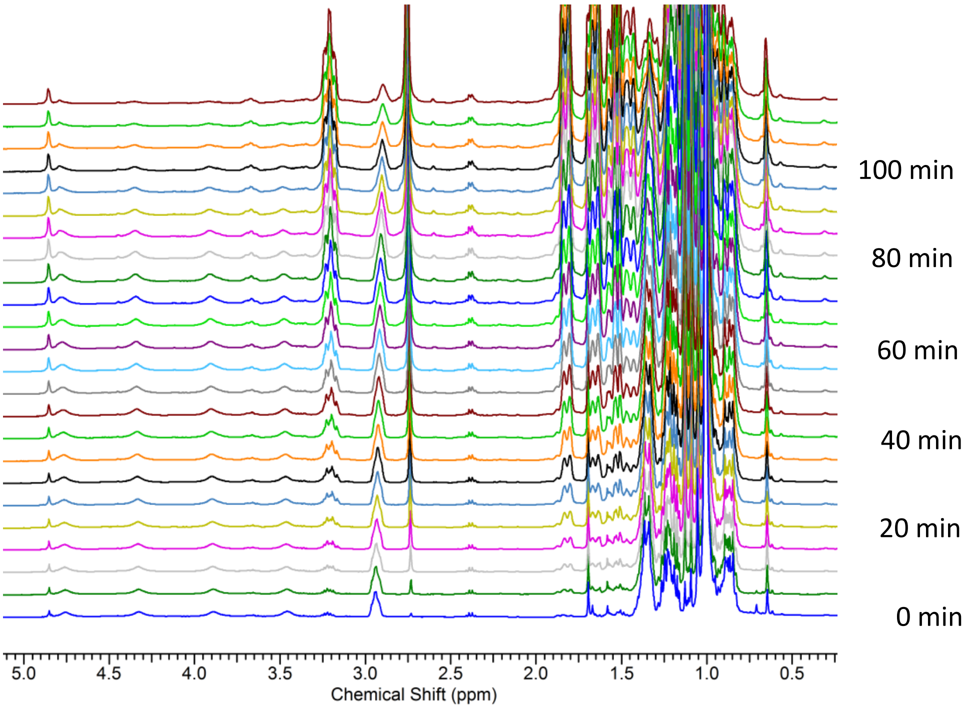
In a glovebox a stock solution of the precatalyst was made to the relevant concentration, 0.5 mL of the catalyst solution was transferred to a Youngs tap NMR tube followed by addition of the relevant quantity of HBpin, followed by the chosen substrate. The tube was sealed, removed from the glovebox, immediately frozen with liquid nitrogen and thawed just prior to loading into the NMR spectrometer which had been preheated to a chosen temperature (if required). ^{1}H NMR spectra were recorded at regular intervals. Reaction kinetics were monitored using the intensity changes in the substrate resonances over three or more half-lives on the basis of substrate consumption. Data was normalised against the initial substrate concentration $[Substrate]_{t=0}$ so that:

$$Ct = \frac{[Substrate]_{t=0}}{[Substrate]_{t=0} + [Substrate]_t}$$

Reaction rates were derived from the plot of Ct vs time (or Ln(Ct), 1/Ct) by using linear trendlines generated by Microsoft Excel software. To obtain Arrhenius and Eyring plots, kinetic analyses were conducted at 4-5 different temperatures, each separated by approximately 5 K.

Figure S1: (a) ^{1}H NMR spectra for the hydroboration of cyclohexylisonitrile, showing the consumption of the CyNC starting reagent (δ 2.90 ppm) and subsequent CyN(Bpin)CH$_2$Bpin formation (δ 3.21 ppm). Spectra were recorded every 5 minutes; (b) expansion of the region 2.5 – 3.5 ppm with spectra recorded every 15 minutes.

(a)



(b)

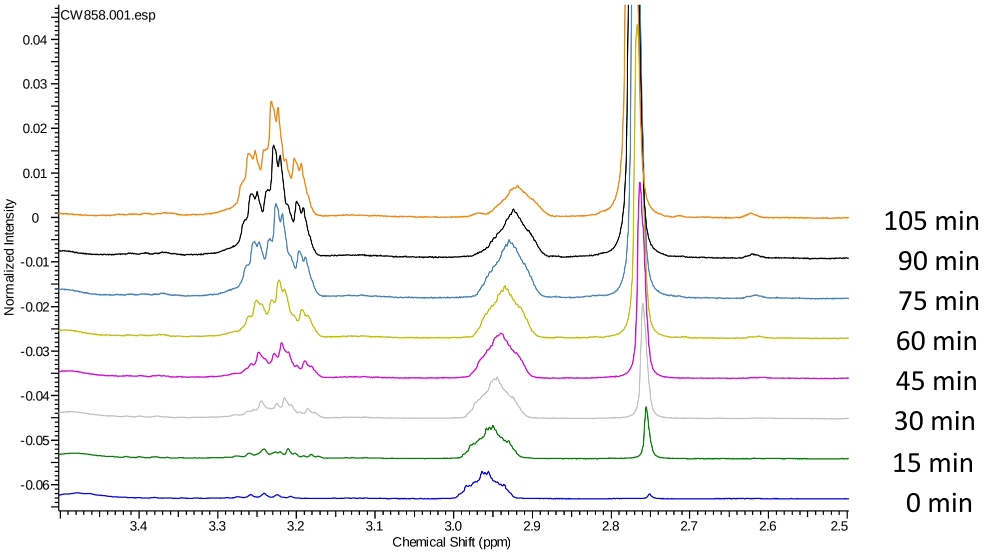
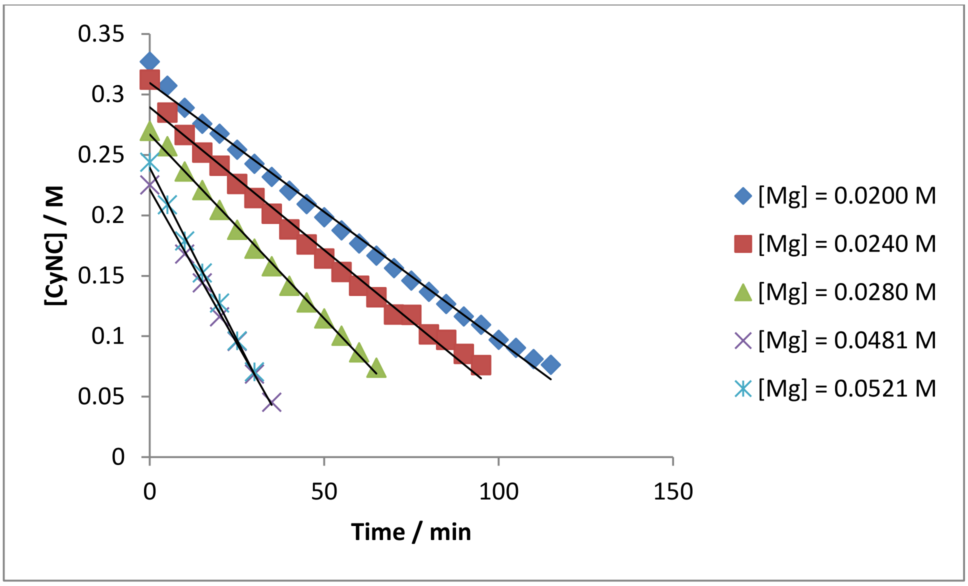


Figure S2: Zero order plots for the hydroboration of CyNC. Both CyNC and HBpin were kept constant at 0.4 M and 0.82 M respectively whilst [Mg] was varied.



	[Mg] 0.0200M	
	Value	Error
m_1	0.309566	0.002348
m_2	-0.002131	0.000035
Chisq	0.055541	n/a
R^2	0.994107	n/a

	[Mg] 0.0240M	
	Value	Error
m_1	0.289524	0.003540
m_2	-0.002361	0.000064
Chisq	0.109083	n/a
R^2	0.987061	n/a

	[Mg] 0.0280M	
	Value	Error
m_1	0.266862	0.001528
m_2	-0.003043	0.000040
Chisq	0.002119	n/a
R^2	0.997934	n/a

	[Mg] 0.0481M	
	Value	Error
m_1	0.221483	0.048019
m_2	-0.005095	0.002155
Chisq	16.95734	n/a
R^2	0.998221	n/a

	[Mg] 0.0521M	
	Value	Error
m_1	0.239246	0.002157
m_2	-0.005691	0.000120
Chisq	0.006645	n/a
R^2	0.997796	n/a

Figure S3: Non-linear first order plots for varying [Mg] for the hydroboration of CyNC by HBpin.

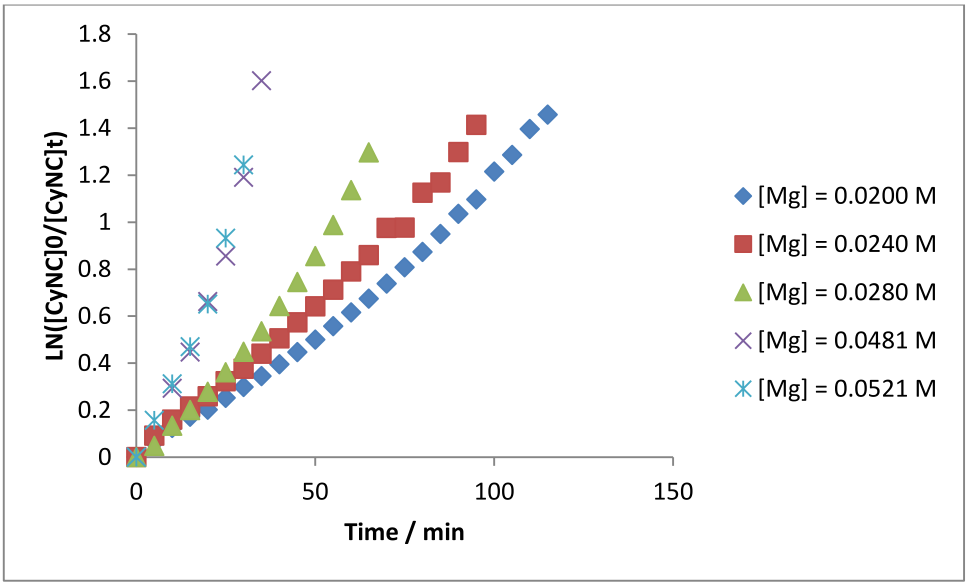


Figure S4: Non-linear second order plots for varying [Mg] for the hydroboration of CyNC by HBpin.

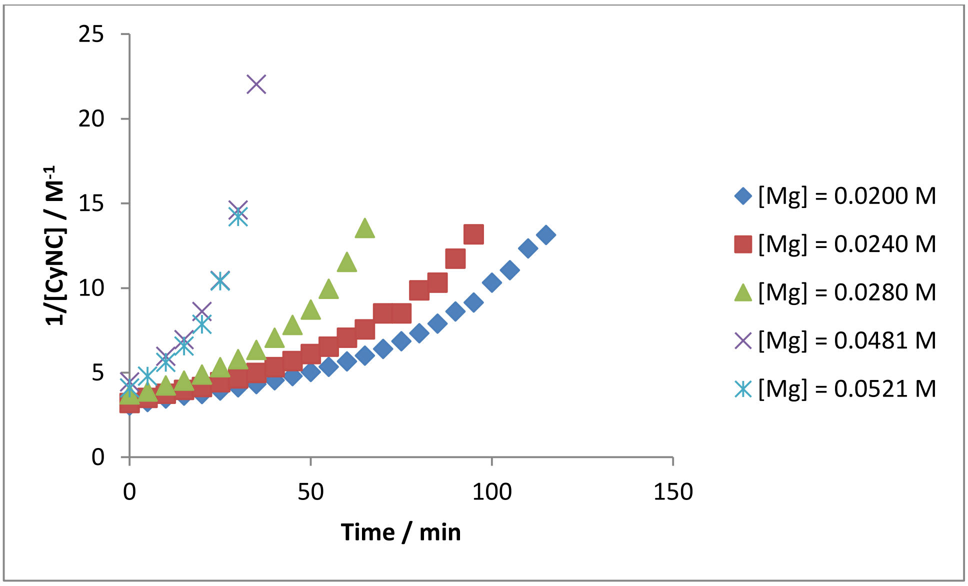
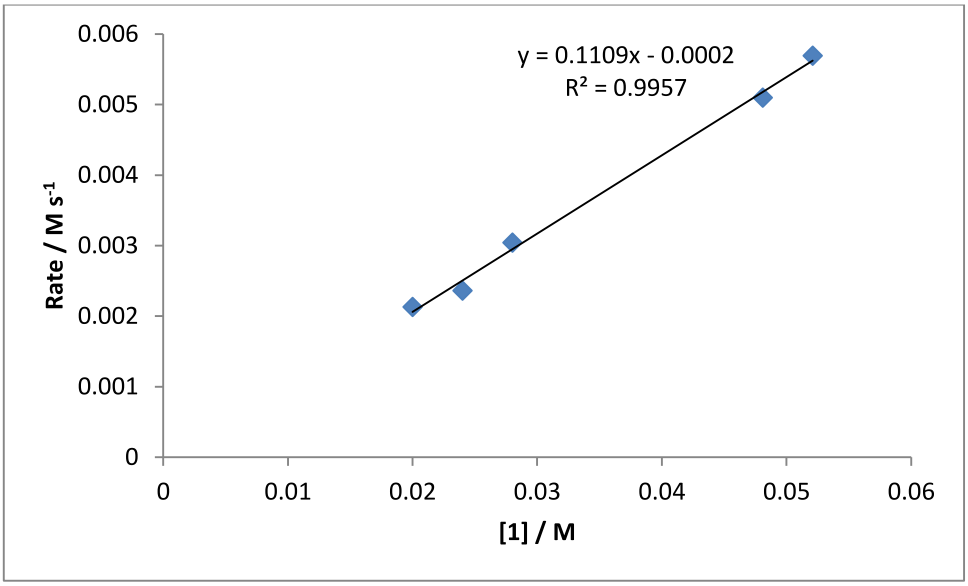
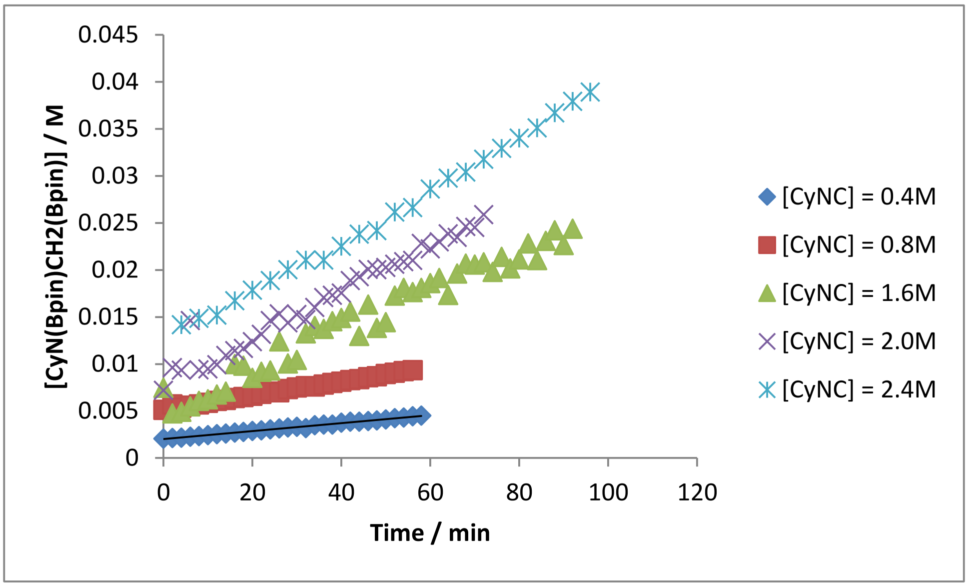


Figure S5: Plot of k_{obs} versus [Mg] for the hydroboration of CyNC by HBpin.



	Value	Error
m_1	-0.000154	0.000156
m_2	0.110870	0.004231
Chisq	0.002158	n/a
R^2	0.995651	n/a

Figure S6: Zero order plots for the hydroboration of CyNC catalysed by 0.02 M [**1**]. CyNC was varied whilst keeping [HBpin] under pseudo first order conditions, 8.0M.



	[CyNC] 0.4M	
	Value	Error
m_1	0.002007	0.000016
m_2	0.000042	0.000000
Chisq	0.001624	n/a
R^2	0.996606	n/a

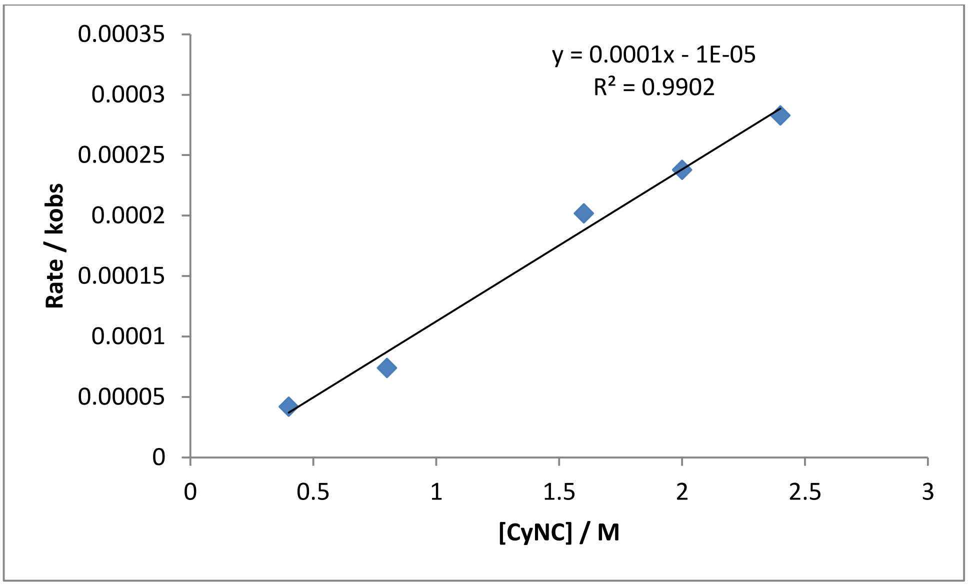
	[CyNC] 0.8M	
	Value	Error
m_1	0.005174	0.000034
m_2	0.000074	0.000001
Chisq	0.002271	n/a
R^2	0.994771	n/a

	[CyNC] = 1.6M	
	Value	Error
m_1	0.005283	0.000332
m_2	0.000209	0.000006
Chisq	0.138667	n/a
R^2	0.961244	n/a

	[CyNC] = 2.0M	
	Value	Error
m_1	0.008239	0.000337
m_2	0.000238	0.000008
Chisq	0.039415	n/a
R^2	0.961579	n/a

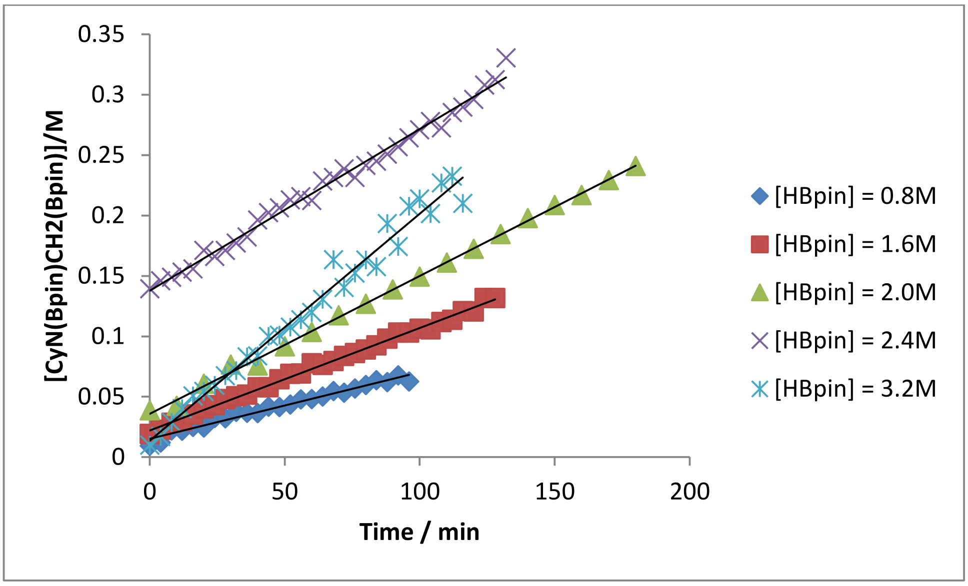
	[CyNC] = 2.4M	
	Value	Error
m_1	0.012131	0.000214
m_2	0.000273	0.000004
Chisq	0.016072	n/a
R^2	0.995899	n/a

Figure S7: Apparent first order variation in k_{obs} dependent on [CyNC] catalysed by 0.02 M [**1**] under pseudo first order conditions with respect to [HBpin].



	Value	Error
m_1	-0.000013	0.000012
m_2	0.000126	0.000007
Chisq	0.002405	n/a
R^2	0.990203	n/a

Figure S8: Zero order plots for the hydroboration of CyNC catalysed by 0.02 M [**1**]. HBpin was varied whilst keeping [CyNC] under pseudo first order conditions, 8.0M.



	[HBpin] 0.8M	
	Value	Error
m_1	0.015129	0.001054
m_2	0.000553	0.000019
Chisq	0.128281	n/a
R^2	0.974048	n/a

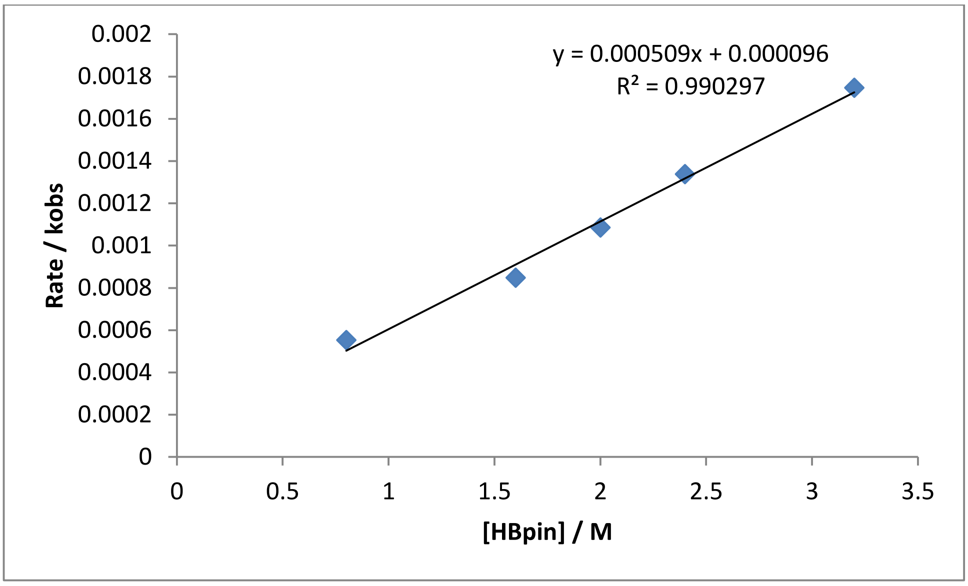
	[HBpin] 0.8M	
	Value	Error
m_1	0.021996	0.000752
m_2	0.000848	0.000010
Chisq	0.007814	n/a
R^2	0.995629	n/a

	[HBpin] = 2.0M	
	Value	Error
m_1	0.035937	0.001211
m_2	0.001141	0.000011
Chisq	0.002086	n/a
R^2	0.998276	n/a

	[HBpin] = 2.4M	
	Value	Error
m_1	0.137832	0.001753
m_2	0.001338	0.000023
Chisq	0.000823	n/a
R^2	0.990762	n/a

	[HBpin] = 3.2M	
	Value	Error
m_1	0.013615	0.003307
m_2	0.001878	0.000049
Chisq	0.003077	n/a
R^2	0.981316	n/a

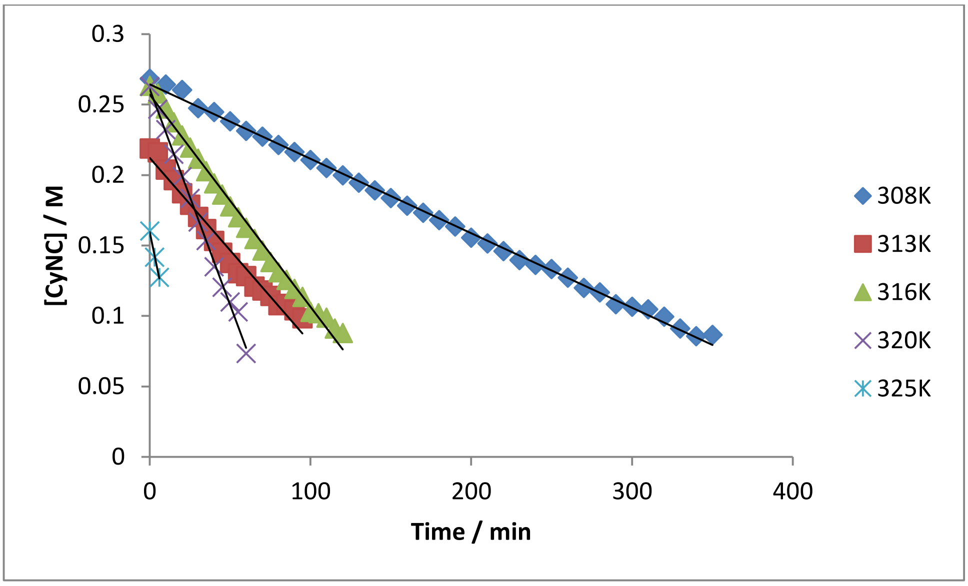
Figure S9: Apparent first order variation in k_{obs} for the hydroboration of CyNC catalysed by 0.02 M [**1**] dependent on [HBpin] under pseudo first order conditions with respect to [CyNC]



	Value	Error
m_1	0.000096	0.000063
m_2	0.000509	0.000029
Chisq	0.014812	n/a
R^2	0.990297	n/a

Variable Temperature Kinetics

Figure S10: Kinetic plots of [CyNC] hydroboration catalysed by 0.02 M [**1**] versus time for variable temperatures (308K, 313K, 316K, 320K and 325K), each shows overall zero-order dependence with the slope, $m_1 = k_{obs}$.



	308K	
	Value	Error
m_1	0.264484	0.000859
m_2	-0.000528	0.000004
Chisq	0.004748	n/a
R^2	0.997835	n/a

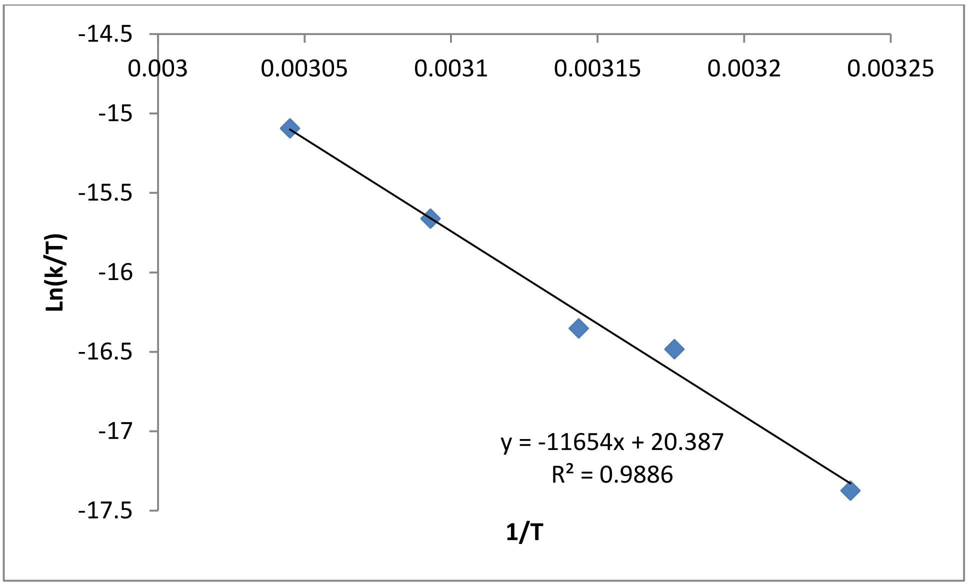
	313K	
	Value	Error
m_1	0.212165	0.002971
m_2	-0.001311	0.000053
Chisq	0.029251	n/a
R^2	0.970912	n/a

	316K	
	Value	Error
m_1	0.257173	0.001994
m_2	-0.001508	0.000028
Chisq	0.012496	n/a
R^2	0.991853	n/a

	320K	
	Value	Error
m_1	0.261047	0.002021
m_2	-0.003062	0.000057
Chisq	0.000886	n/a
R^2	0.996182	n/a

	325K	
	Value	Error
m_1	0.159550	0.001704
m_2	-0.005490	0.000440
Chisq	0.00319	n/a
R^2	0.993620	n/a

Figure S11: Eyring Plot of Ln(k_{obs}/T) versus 1/T

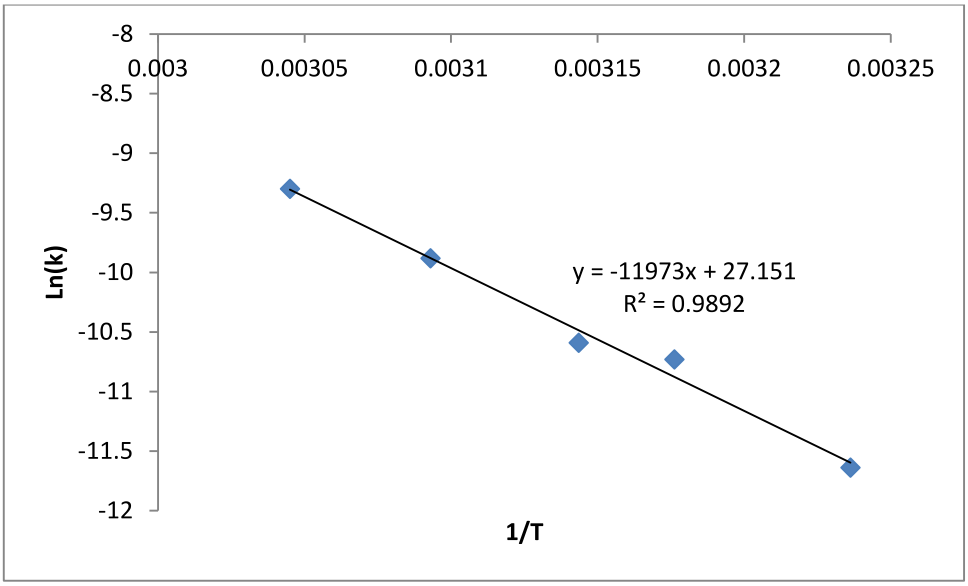


	Value	Error
m_1	22.234188	1.321007
m_2	-12248.228124	421.752914
Chisq	0.001242916	n/a
R^2	0.997634	n/a

These graphs were used to calculate the following Activation Energy Parameters, least square error analysis was also carried to provide accurate error information.

	Value	Error
Ea	104.47 kJ mol-1	± 3.52
Δ*H*	101.83 kJ mol^{-1}	± 3.51
Δ*S*	-12.7 J k^{-1} mol^{-1}	± 11.0
ΔG_{298}	105.61 kJ mol^{-1}	n/a

Figure S12: Arrhenius Plot of Ln(k_{obs}) versus 1/T



	Value	Error
m_1	28.996472	1.325458
m_2	-12566.221473	423.174090
Chisq	0.001193718	n/a
R^2	0.997737	n/a

X-ray diffraction analysis of compound 9

A suitable crystal was selected and data were collected on a SuperNova, Dual, Cu at zero, EosS2 diffractometer. The crystal was kept at 150(2) K during data collection. Using Olex2,[2] the structure was solved with the olex2.solve[3] structure solution program using Charge Flipping and refined with the ShelXL[4] refinement package using Least Squares minimization. The asymmetric unit comprises two independent dimer halves, each proximate to a crystallographic inversion centre (which is exploited to generate the molecule remainders) plus half of a toluene molecule with 60% occupancy. The latter, which also straddles an inversion centre, necessarily means that the associated methyl substituent is disordered by symmetry. Consequently, the hydrogen attached to C75 for the 50% of time when the solvent methyl group is absent from the asymmetric unit was omitted from the refinement. H30 and H66 were located and refined at a distance of 0.98Å from the parent carbons. 60:40 disorder was modelled for the cyclohexyl group based on C67. C_1-C_2 and $C_1 \ldots C_3$ distances were restrained in the minor disordered position of this ring, to assist convergence. Some ADP restraints were also included for the same reason.

Table S3:. Crystal data and structure refinement for compound **9**.

Identification code	s14msh3
Empirical formula	C74.10 H109.20 Mg2 N6
Formula weight	1132.69
Temperature	150(2) K
Wavelength	1.54184 Å
Crystal system	Monoclinic
Space group	P21/c
Unit cell dimensions	a = 18.9579(8)Å alpha = 90.0^{o}
	b = 19.7413(9)Å beta = 99.765(4)^{o}
	c = 18.7107(8)Å gamma = 90.0^{o}
Volume	6901.1(5) Å^{3}
Z	4
Density (calculated)	1.090 Mg/m^{3}
Absorption coefficient	1.563 mm^{-1}
F(000)	2479
Crystal size	0.3233 x 0.0873 x 0.0650 mm
Theta range for data collection	4.2768 to 66.60^{o}
Index ranges	-23<=h<=22; -24<=k<=24; -22<=l<=23
Reflections collected	50997
Independent reflections	12177 [R(int) = 0.0901]
Reflections observed (>2sigma)	8151
Data Completeness	0.9829
Absorption correction	Gaussian
Max. and min. transmission	0.916 and 0.743
Refinement method	Full-matrix least-squares on F^{2}
Data / restraints / parameters	12177 / 59 / 840
Goodness-of-fit on F^{2}	1.034
Final R indices [I>2sigma(I)]	*R*1 = 0.0810 *wR*2 = 0.2100
R indices (all data)	*R*1 = 0.1157 *wR*2 = 0.2414
Largest diff. peak and hole	0.402 and -0.453 eÅ^{-3}

References

1. (a) V. C. Gibson, J. A. Segal, A. J. P. White, D. J. Williams,*J. Am. Chem. Soc.* **2000**, *122*, 7120; (b) Bonyhady, S.J.; Jones, C.; Nembenna, S.; Stasch, A.; Edwards, A.J.; McIntyre, G.J. *Chem. Eur. J.* **2010**, *16*, 938-955.
2. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H. *J. Appl. Cryst.* **2009**, *42*, 339-341.
3. Bourhis, L. J.; Dolomanov, O. V.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H. *Acta Cryst. A* **2015**, *71*, 59-75.
4. Sheldrick, G.M. *Acta Cryst. A*, **2008**, *64*, 112-122.