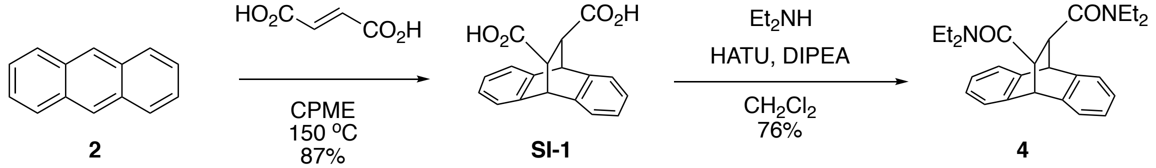
Supporting Information

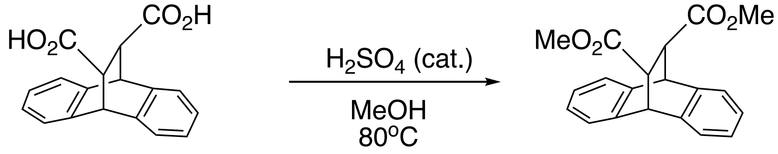
Compound Synthesis

^{1}H NMR spectra were obtained on a JEOL instrument at 400 MHz in $CDCl_3$ or DMSO-*d6*. Chemical shifts (δ) were reported in parts per million with the residual solvent peak used as an internal standard δ ^{1}H / ^{13}C (solvent): 7.26 / 77.00 ($CDCl_3$); 2.50 (DMSO-*d6*). ^{1}H NMR spectra were obtained and are tabulated as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, bs = broad singlet), number of protons, and coupling constant(s). ^{13}C NMR spectra were recorded using a proton-decoupled pulse sequence run at 100 MHz and are tabulated by observed peaks.



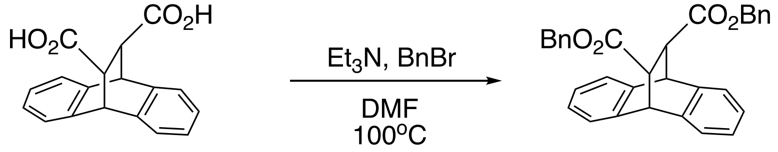
SI-1[1]: A suspension of anthracene (**2**, 6.11 g, 34.3 mmol, 1.8 equiv.) and fumaric acid (2.21 g, 19.0 mmol, 1.00 equiv.) in cyclopentyl methyl ether (19 mL) was heated at 150 °C in a heating block for 22 h. The dark brown solution was quenched with satd. $NaHCO_3$ (50 mL), and the solid was filtered off while still warm. The solid was rinsed with satd. $NaHCO_3$ (20 mL) and Et_2O (30 mL). The filtrate layers were separated, and the aqueous phase was extracted a second time with Et_2O (30 mL). The aqueous layer was then acidified with conc. HCl and extracted with Et_2O (4x30 mL). The ethereal extracts were rinsed with brine (30 mL), dried (Na_2SO_4), and concentrated giving **SI-1** (4.88 g, 16.6 mmol, 87%) as an off-white solid. ^{1}H NMR (DMSO-*d6*, 400 MHz) δ 12.58 (bs, 2 H), 7.41-7.39 (m, 2 H), 7.27-7.25 (m, 2 H), 7.13-7.06 (m, 4 H), 4.72 (s, 2 H), 3.12 (s, 2 H).

4: To a suspension of **SI-1** (2.00 g, 6.46 mmol, 1.00) in CH_2Cl_2 (31 mL) was added ^{i}Pr$_2$NEt (DIPEA, 3.40 mL, 19.3 mmol, 3.00 equiv.) and HATU (5.89 g, 15.5 mmol, 2.40 equiv.). The mixture was stirred for 20 min, then Et_2NH (1.50 mL, 14.2 mmol, 2.20 equiv.) was added, and stirring continued for 24 h. The reaction mixture was diluted with Et_2O (70 mL), rinsed with satd. $NaHCO_3$, (2x40 mL), 1 M HCl (2x40 mL), and brine (40 mL), then dried (Na_2SO_4) and concentrated. Purification by chromatography on SiO_2 (1:1 hexanes:ethyl acetate) gave **4** (2.04 g, 5.04 mmol, 78%) as a colorless solid. Mp = 170.1-170.4 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29-7.26 (m, 2 H), 7.18-7.16 (m, 2 H), 7.15-7.07 (m, 4 H), 4.27 (s, 2 H), 3.60-3.37 (m, 8 H), 3.14-3.05 (m, 2 H), 1.21 (t, 6 H, *J* = 6.0 Hz), 1.06 (t, 6 H, *J* = 6.0 Hz); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.25, 142.71, 139.95, 126.23, 126.06, 125.03, 122.46, 48.40, 45.11, 42.04, 40.35, 14.96, 13.18.



SI-1 **6**

6^2: To a solution of **SI-1** (0.400 g, 1.09 mmol, 1.00 equiv.) in methanol (3.6 mL) was added conc. sulfuric acid (1 drop). The vial was sealed, heated to 80 °C, and stirred for 25 h. The reaction was neutralized with satd. $NaHCO_3$ (8 mL) and extracted with Et_2O (3x10 mL). The combined organic layers were rinsed with water (5 mL) and brine (5 mL), then dried (Na_2SO_4) and concentrated. Purification by chromatography on SiO_2 (6:1 hexanes:ethyl acetate) gave **6** (0.353 g, 1.10 mmol, 81%) as a colorless solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34-7.32 (m, 2 H), 7.26-7.23 (m, 2 H), 7.14-7.08 (m, 4 H), 4.73 (s, 2 H), 3.63 (s, 6 H), 3.42 (s, 2 H).



SI-1 **7**

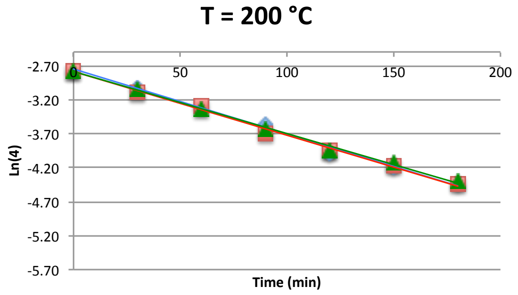
7: To a suspension of **SI-1** (0.300 g, 0.815 mmol, 1.00 equiv.) in DMF (1.9 mL) was added triethylamine (0.227 mL, 1.63 mmol, 2.00 equiv.) and BnBr (0.485 mL, 4.08 mmol, 5.00 equiv.). The vial was sealed, heated to 100 °C, and stirred for 48 h. The reaction diluted with 1:1 hexanes:ethyl acetate (10 mL). The mixture was rinsed with 1 M HCl (5 mL), water (5 mL), and brine (5 mL), then dried (Na_2SO_4) and concentrated. Purification by chromatography on SiO_2 (10:1 hexanes:EtOAc) gave the desired product with a significant amount of grease. The pale yellow oil was recrystallized from heptane giving **7** (0.099 g, 0.21 mmol, 25%) as a colorless solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37-7.36 (m, 6 H), 7.32-7.30 (m, 2 H), 7.25 (m, 4 H), 7.12-7.08 (m, 2 H), 7.03-7.00 (m, 4 H), 5.07, 5.00 (ABq, 4 H, J_{AB} = 12.0 Hz), 4.72 (s, 2 H), 3.52 (s, 2 H).

Procedures for Microwave and Conventional Heating Experiments

Microwave experiments were performed using a CEM Discover S equipped with a high temperature spill cup purchased from CEM. The microwave cavity was preheated prior to use by applying full power (300 W) to a sample of tridecane (~2 mL) in a 10-mL pyrex microwave tube for 20 min. All temperatures were monitored using the fiber optic probe purchased from CEM, with the probe inserted into the reaction mixture in a sapphire thermowell. All reactions were performed in oven-dried 10-mL pyrex reaction vessels. Aliquots (0.10-0.15 mL) were removed immediately following the dissolution of starting materials and immediately following the reaction period. These aliquots were dissolved in $CDCl_3$ (0.40-0.45 mL) for ^{1}H NMR analysis. Baselines in all NMRs were corrected using full auto (Whittaker Smoother) prior to integration to ensure consistent results. The methine peak of triphenylmethane was used as an internal standard for monitoring reaction progress.

General Procedure A: Microwave Kinetic Experiments

Cycloadduct **4** (64.5 mg, 0.159 mmol, 1.00 equiv.) and triphenylmethane (39.0 mg, 0.159 mmol, 1.00 equiv.; internal standard) under nitrogen were suspended in freshly degassed tridecane (2.5 mL) in a 10 mL pyrex microwave tube equipped with a rubber septum and nitrogen balloon. The tube was then transferred to a preheated heating block (130 °C) for ~5 min to dissolve the starting materials. An aliquot was removed from the reaction mixture and saved for later ^{1}H NMR analysis. The tube, which now contained a clear, colorless mixture, was transferred to the microwave. The fiber optic probe attachment was added, and the sample was heated to an internal temperature of 200 °C (constant temperature mode) for 0.5 h. The reaction mixture was allowed to cool to ~140 °C (requires ~30 seconds under stream of air in microwave cavity), then the microwave opened and the tube caped with a septum fitted with a nitrogen balloon immediately. (Control experiments under conventional heating showed that this ~30-sec cooling and vial transfer had no observable effect on reaction conversion.) An aliquot was removed for later ^{1}H NMR analysis. This process was repeated for reaction times of 1 h, 1.5 h, 2 h, 2.5 h, and 3 h. This process was then repeated twice more, resulting in 3 total experiments for each time point.



T = 200 °C			
Trial	*k* (x10^{-2}) (min^{-1})	σ (x10^{-2})	R^2
1	0.95	0.03	0.994
2	0.94	0.03	0.995
3	0.91	0.02	0.997
Average	0.93	0.05	

General Procedure B: Conventional Heating Kinetic Experiments

Cycloadduct **4** (64.5 mg, 0.159 mmol, 1.00 equiv.) and triphenylmethane (39.0 mg, 0.159 mmol, 1.00 equiv.; internal standard) under nitrogen were suspended in freshly degassed tridecane (2.5 mL) in a 10 mL pyrex microwave tube equipped with a rubber septum, internal fiber optic probe in a sapphire thermowell and nitrogen balloon. The tube was transferred to a preheated heating block (130°C) for ~5 min to dissolve the starting materials. An aliquot was removed from the reaction mixture and saved for later ^{1}H NMR analysis. The clear, colorless mixture was transferred to a preheated heating block and heated for 0.5 h at an internal temperature of 200 °C. An aliquot was removed for later ^{1}H NMR analysis. (The ~30-second cooling and vial removal process that was necessary for reliable sampling in the microwave heating experiments was not necessary for sampling conventional heating experiments, nor did it have any observable effect on reaction conversion, so it was not incorporated into the standard protocol here.) This process was repeated for reaction times of 1 h, 1.5 h, 2 h, 2.5 h, and 3 h. This process was then repeated twice more, resulting in 3 total experiments for each time point.

Conventional Heating Kinetic Experiments at 205 °C

Experiments were conducted according to General Procedure B, except that they were performed at 205 °C.

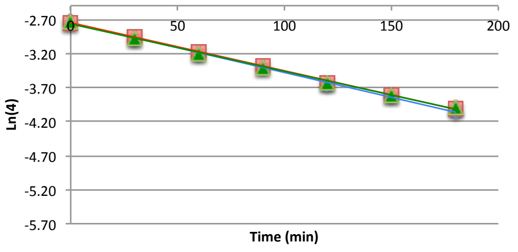
Conventional Heating Kinetic Experiments at 210 °C

Experiments were conducted according to General Procedure B, except that they were performed at 210 °C.

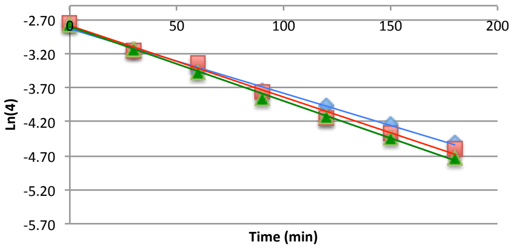
Conventional Heating Kinetic Experiments at 215 °C

Experiments were conducted according to General Procedure B, except that they were performed at 215 °C. Additionally, reactions were only performed for 0.5 h, 1 h, 1.5 h, and 2 h. No significant further conversion was observed beyond this point.

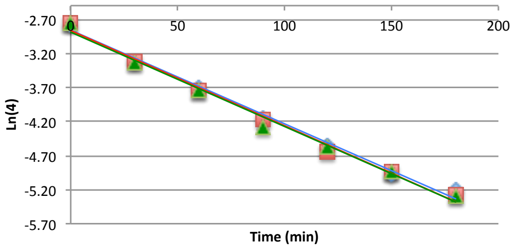
T = 200 °C



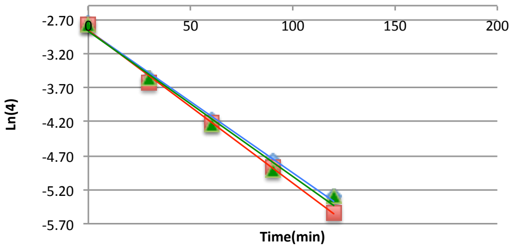
T = 205 °C



T = 210 °C



T = 215 °C



T = 200 °C			
Trial	***k*** **(x10^{-2}) (min^{-1})**	**σ (x10^{-2})**	**R^2**
1	0.73	0.02	0.998
2	0.71	0.01	0.999
3	0.70	0.01	0.998
Average	0.71	0.02	

T = 205 °C			
Trial	***k* (x10^{-2}) (min^{-1})**	**σ (x10^{-2})**	**R^2**
1	0.95	0.03	0.994
2	1.05	0.05	0.992
3	1.09	0.03	0.997
Average	1.03	0.06	

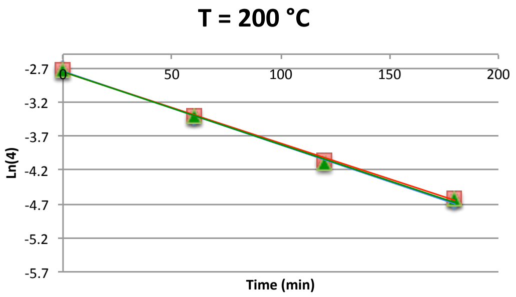
T = 210 °C			
Trial	***k* (x10^{-2}) (min^{-1})**	**σ (x10^{-2})**	**R^2**
1	1.37	0.05	0.993
2	1.40	0.05	0.992
3	1.38	0.07	0.989
Average	1.39	0.10	

T = 215 °C			
Trial	***k* (x10^{-2}) (min^{-1})**	**σ (x10^{-2})**	**R^2**
1	2.09	0.10	0.993
2	2.24	0.08	0.996
3	2.13	0.14	0.987
Average	2.15	0.19	

Experiments with SPAN 60

Microwave Kinetic Experiments with SPAN 60

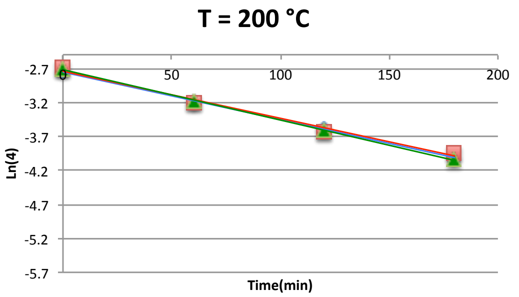
Performed according to General Procedure A with the inclusion of SPAN 60 (6.5 mg, 0.015 mmol, 0.094 equiv.). Reactions were performed for 1 h, 2 h, and 3 h time points in triplicate.



T = 200 °C			
Trial	***k* (x10^{-2}) (min^{-1})**	**σ (x10^{-2})**	**R^2**
1	1.08	0.04	0.998
2	1.05	0.04	0.998
3	1.07	0.05	0.996
Average	1.07	0.07	

Conventional Heating Kinetic Experiments with SPAN 60

Performed according to General Procedure B with the inclusion of SPAN 60 (6.5 mg, 0.015 mmol, 0.094 equiv.). Reactions were performed fro 1 h, 2 h, and 3 h time points in triplicate.



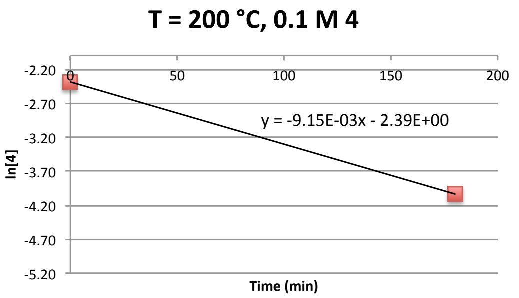
T = 200 °C

T = 200 °C			
Trial	k (x10^{-2}) (min^{-1})	σ (x10^{-2})	R^2
1	0.70	0.01	0.999
2	0.69	0.05	0.990
3	0.74	0.01	0.999
Average	0.71	0.05	

Experiments at Different Concentrations

Microwave Experiment at 0.1 M of cycloadduct 4

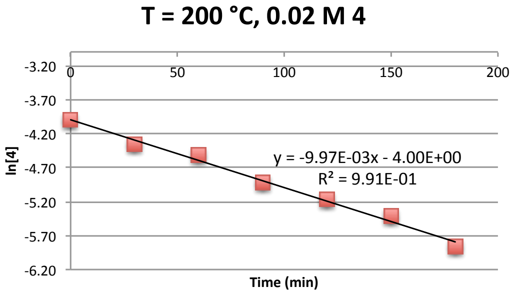
Performed according to General Procedure A, but using **4** (100 mg, 0.247 mmol, 1.00 equiv.) and triphenylmethane (60.5 mg, 0.248 mmol, 1.00 equiv.; internal standard). A single 3 h experiment was performed.



T = 200 °C, 0.1 M 4

Microwave Experiment at 0.02 M of cycloadduct 4

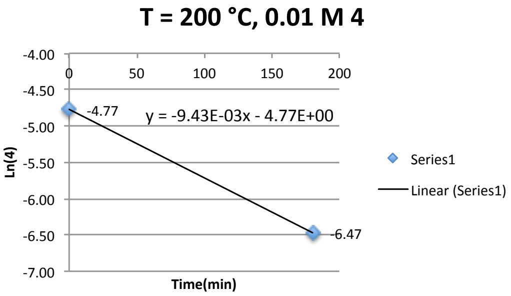
Performed according to General Procedure A, but using **4** (20.0 mg, 0.049 mmol, 1.00 equiv.) and triphenylmethane (12.1 mg, 0.049 mmol, 1.00 equiv.; internal standard). A separate experiment was performed for each time point of 0.5 h, 1 h, 1.5 h, 2 h, 2.5 h, and 3 h.



T = 200 °C, 0.02 M 4

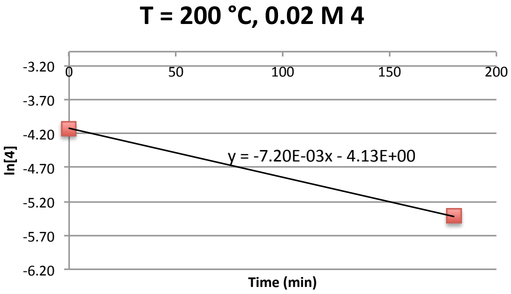
Microwave Experiment at 0.01 M of cycloadduct 4

Performed according to General Procedure A, but using **4** (10.0 mg, 0.025 mmol, 1.00 equiv.) and triphenylmethane (6.0 mg, 0.025 mmol, 1.00 equiv.; internal standard). A single 3 h experiment was performed.



Conventional Heating Experiment at 0.02 M of cycloadduct 4

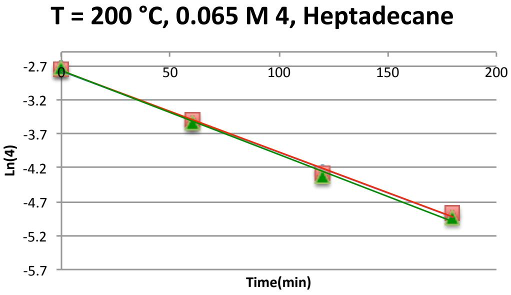
Performed according to General Procedure B, but using **4** (20.0 mg, 0.049 mmol, 1.00 equiv.) and triphenylmethane (12.1 mg, 0.049 mmol, 1.00 equiv.; internal standard). A single 3 h experiment was performed.



Experiment Using Heptadecane Solvent

Microwave Kinetic Experiments at 0.065 M of cycloadduct 4

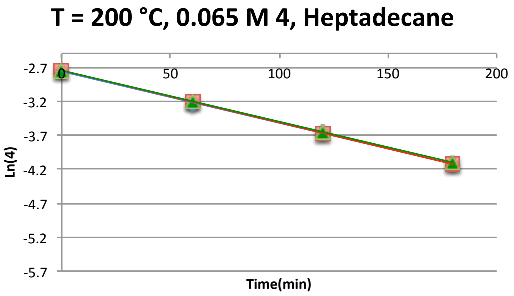
Performed according to General Procedure A, but using heptadecane (2.5 mL). Experiments were performed for 1 h, 2 h, and 3 h time points in triplicate.



T = 200 °C			
Trial	*k* (x10^{-2}) (min^{-1})	σ (x10^{-2})	R^2
1	1.19	0.04	0.997
2	1.19	0.05	0.997
3	1.23	0.06	0.996
Average	1.21	0.09	

Conventional Heating Kinetic Experiments at 0.065 M of cycloadduct 4

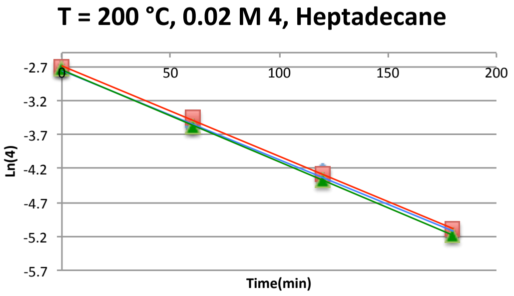
Performed according to General Procedure B, but using heptadecane (2.5 mL). Experiments were performed for 1 h, 2 h, and 3 h time points in triplicate.


T = 200 °C, 0.065 M 4, Heptadecane

T = 200 °C			
Trial	*k* (x10^{-2}) (min^{-1})	σ (x10^{-2})	R^2
1	0.76	0.02	0.999
2	0.77	0.01	1.000
3	0.75	0.003	1.000
Average	0.76	0.02	

Microwave Kinetic Experiments at 0.02 M of cycloadduct 4

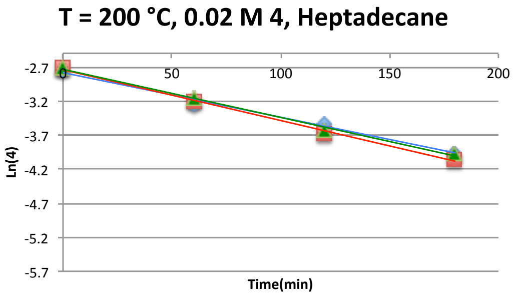
Performed according to General Procedure A, but using **4** (20.2 mg, 0.050 mmol, 1.00 equiv.), triphenylmethane (12.2 mg, 0.050 mmol, 1.00 equiv.; internal standard), and heptadecane (2.5 mL). Experiments were performed for 1 h, 2 h, and 3 h time points in triplicate.


T = 200 °C, 0.02 M 4, Heptadecane

T = 200 °C			
Trial	*k* (x10^{-2}) (min^{-1})	σ (x10^{-2})	R^2
1	1.31	0.06	0.996
2	1.33	0.02	1.000
3	1.35	0.01	1.000
Average	1.33	0.06	

Conventional Heating Kinetic Experiments at 0.02 M of cycloadduct 4

Performed according to General Procedure **A**, but using **4** (20.2 mg, 0.050 mmol, 1.00 equiv.), triphenylmethane (12.2 mg, 0.050 mmol, 1.00 equiv.; internal standard), and heptadecane (2.5 mL). Experiments were performed for 1 h, 2 h, and 3 h time points in triplicate.



T = 200 °C			
Trial	k (x10^{-2}) (min^{-1})	σ (x10^{-2})	R^2
1	0.65	0.04	0.992
2	0.75	0.03	0.996
3	0.71	0.04	0.994
Average	0.70	0.07	

Experiments Using Other Substrates

Microwave Conversion Experiment using 6

Performed according to General Procedure A but using **6** (51.4 mg, 0.159 mmol, 1.00 equiv.). A single 3 h experiment was performed.

Conventional Heating Conversion Experiment using 6

Performed according to General Procedure B but using **6** (51.4 mg, 0.159 mmol, 1.00 equiv.). A single 3 h experiment was performed.

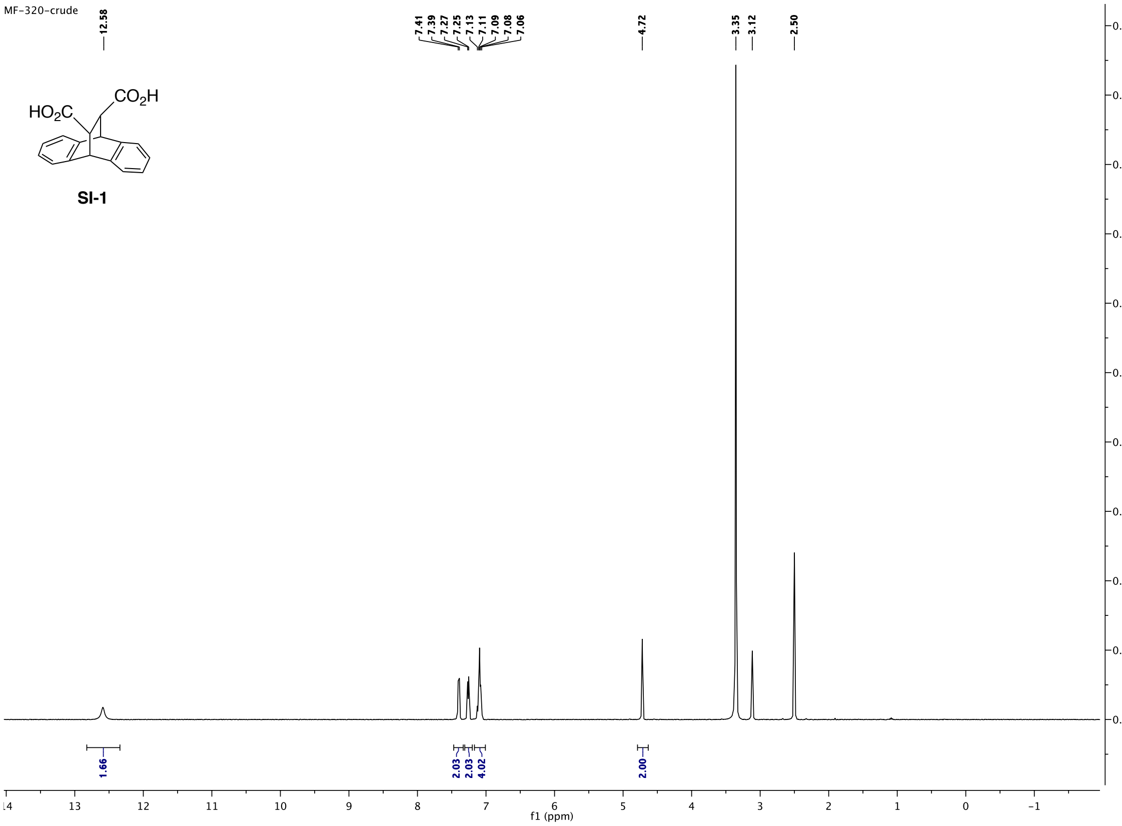
Microwave Conversion Experiment using 7

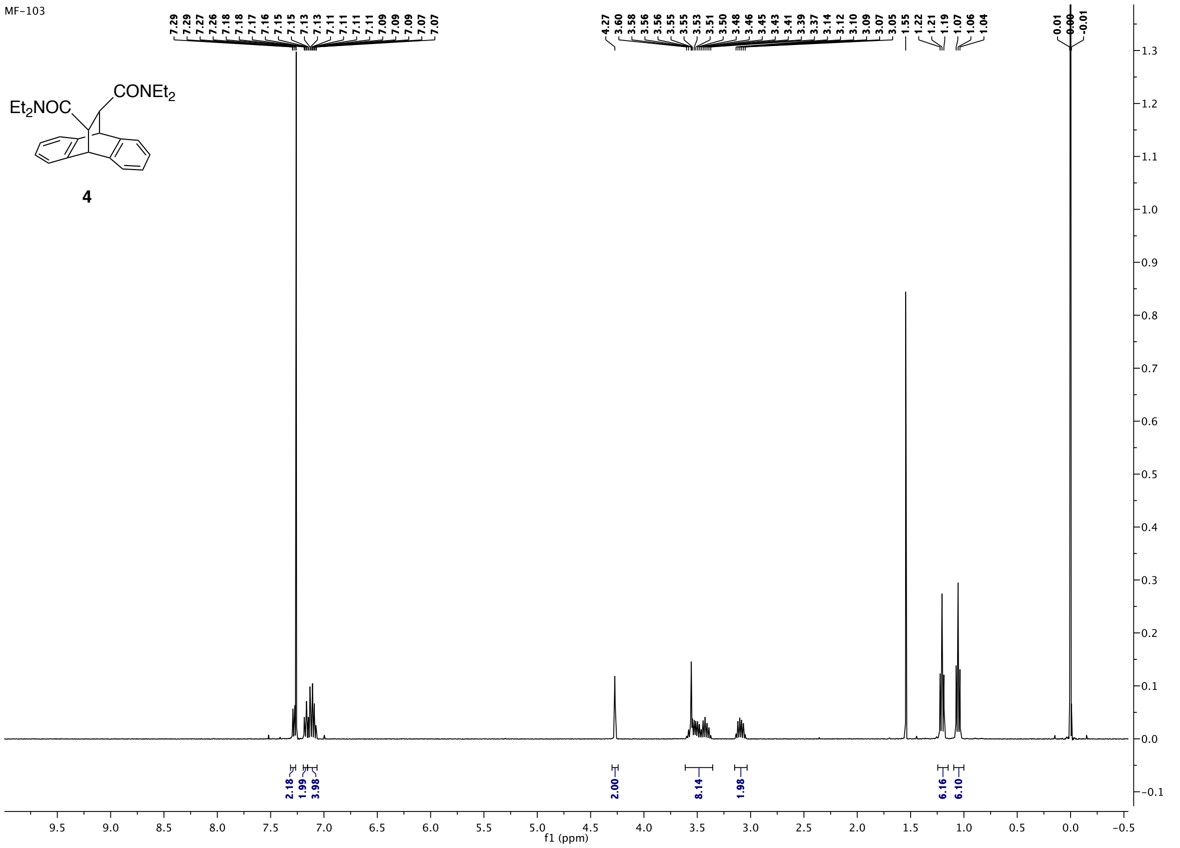
Performed according to General Procedure A but using **7** (75.7 mg, 0.159 mmol, 1.00 equiv.). A single 3 h experiment was performed.

Conventional Heating Conversion Experiment using 7

Performed according to General Procedure B but using **7** (75.7 mg, 0.159 mmol, 1.00 equiv.). A single 3 h experiment was performed.

NMR Spectra of SI-1, 4, 6, and 7

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7.09
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7.07
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3.60
3.58
3.56
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3.41
3.39
3.37
3.14
3.12
3.10
3.09
3.07
3.05
1.55
1.22
1.21
1.19
1.07
1.06
1.04
0.01
0.00
-0.01
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$CONEt_2$
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3.98
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8.14
1.98
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8.0
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5.0
4.5
4.0
3.5
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2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)
1.3
1.2
1.1
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
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0.0
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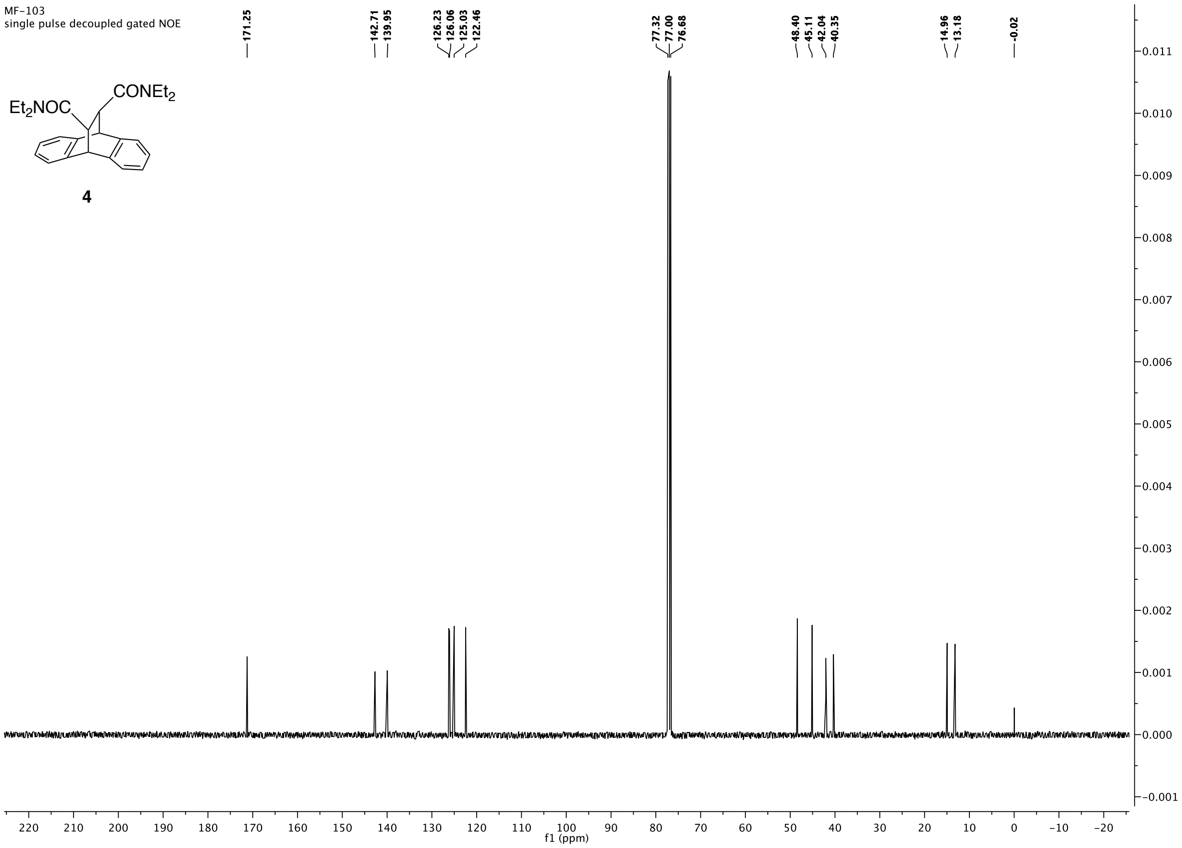
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40.35
14.96
13.18
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210
200
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180
170
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150
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130
120
110
100
90
80
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60
50
40
30
20
10
0
-10
-20
f1 (ppm)
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0.001
0.000
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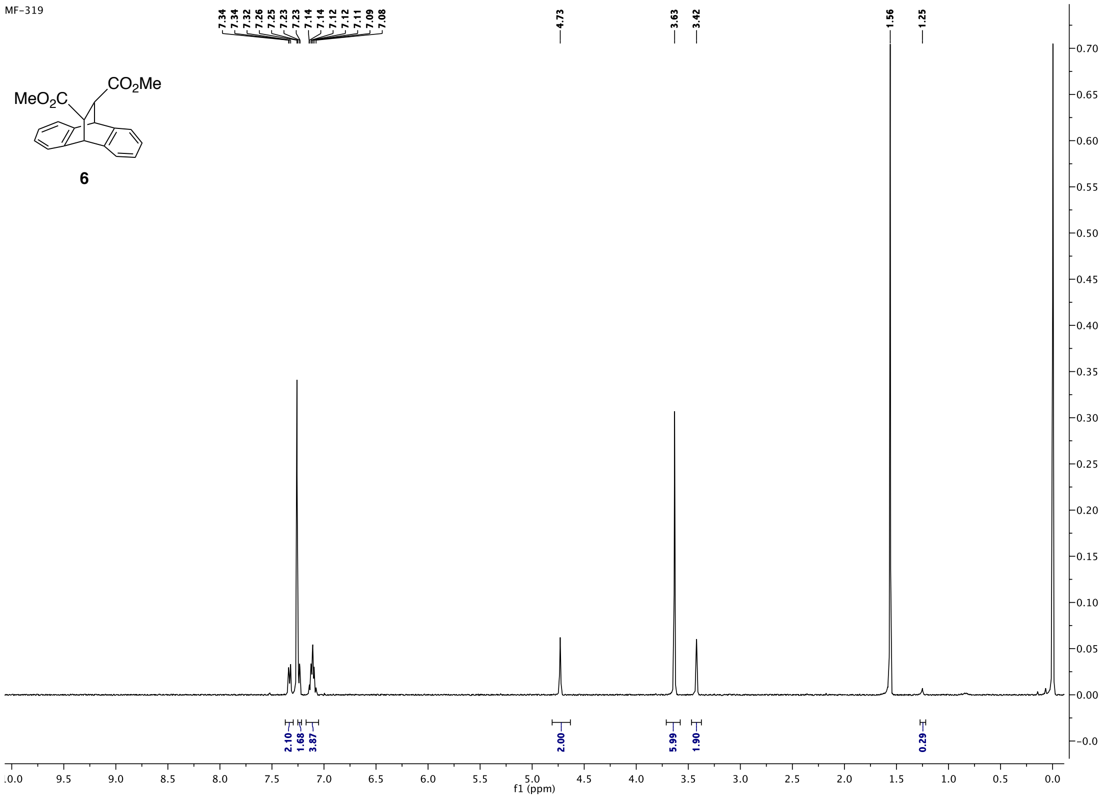
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3.42
1.56
1.25
2.10
1.68
3.87
2.00
5.99
1.90
0.29
0.70
0.65
0.60
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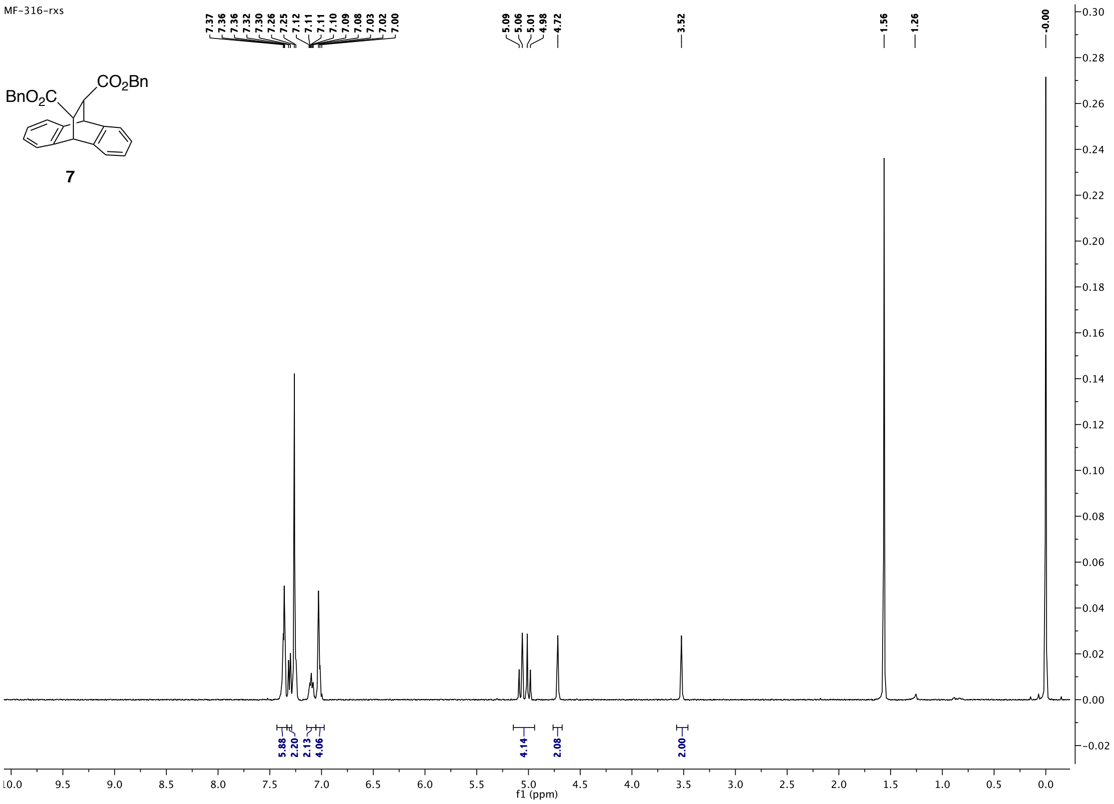
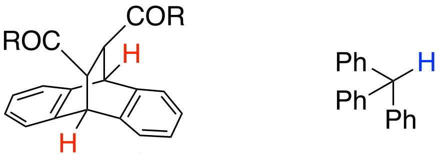
0.5
0.0
f1 (ppm)


MF-316-rxs
BnO2C
CO2Bn
7
7.37
7.36
7.36
7.32
7.30
7.26
7.25
7.12
7.11
7.11
7.10
7.09
7.08
7.03
7.02
7.00
5.09
5.06
5.01
4.98
4.72
3.52
1.56
1.26
-0.00
5.88
2.20
2.13
4.06
4.14
2.08
2.00
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
-0.02
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

Representative ^{1}H NMR Spectra for Kinetic Experiments



4, 6, 7

triphenylmethane

Figure 1: Peaks used for analysis in this section. On 4 (R = NEt_2), 6 (R = OMe), and 7 (R = OBn) the red Hs were used and are labeled as a red "A" (A). The internal standard triphenylmethane peak is labeled as a blue "B" (B).

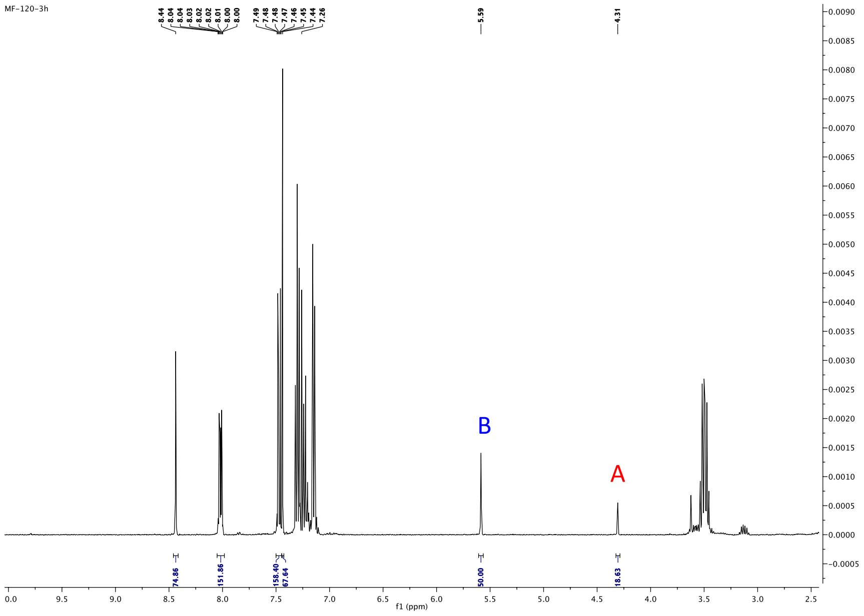


Figure 2: ^{1}H NMR spectrum of 4 + internal standard after 3 h at 200 °C in tridecane with MW heating.

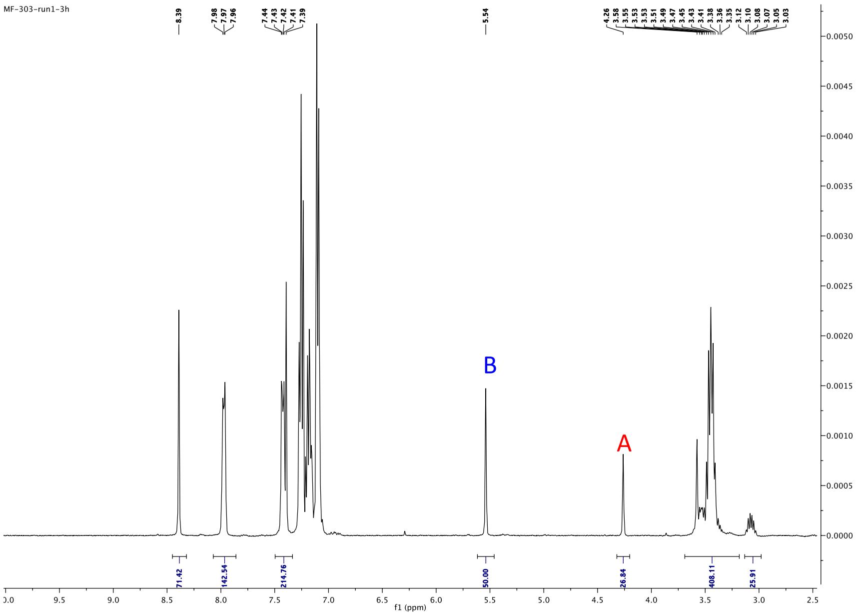


Figure 3: ^{1}H NMR spectrum of 4 + internal standard after 3 h at 200 °C in tridecane with CH heating.

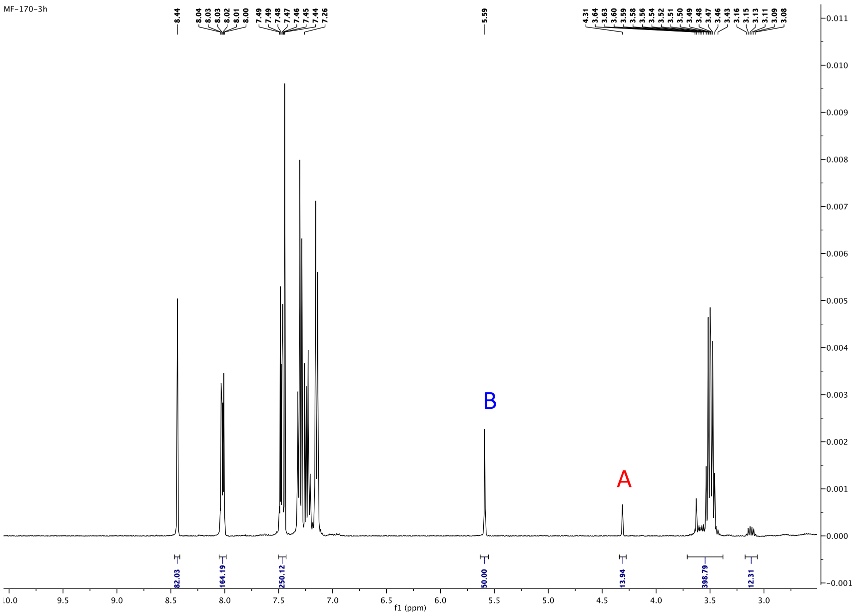


Figure 4: ^{1}H NMR spectrum of 4 + internal standard after 3 h at 205 °C in tridecane with CH heating.

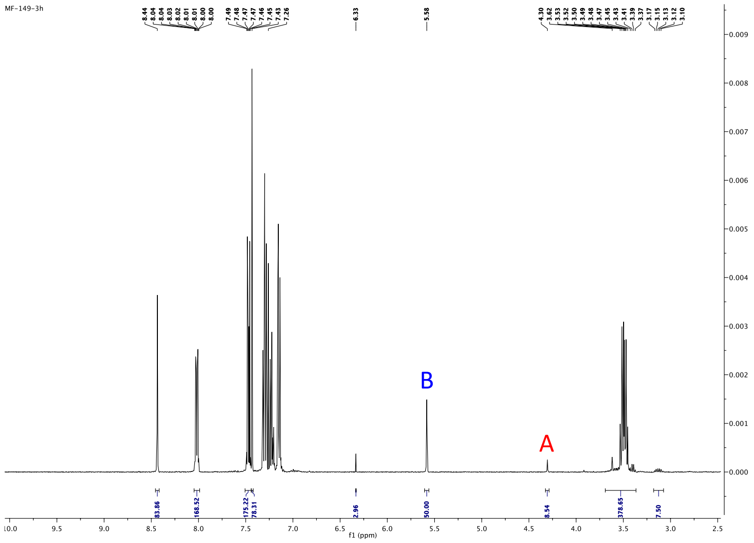


Figure 5: ^{1}H NMR spectrum of 4 + internal standard after 3 h at 210 °C in tridecane with CH heating.

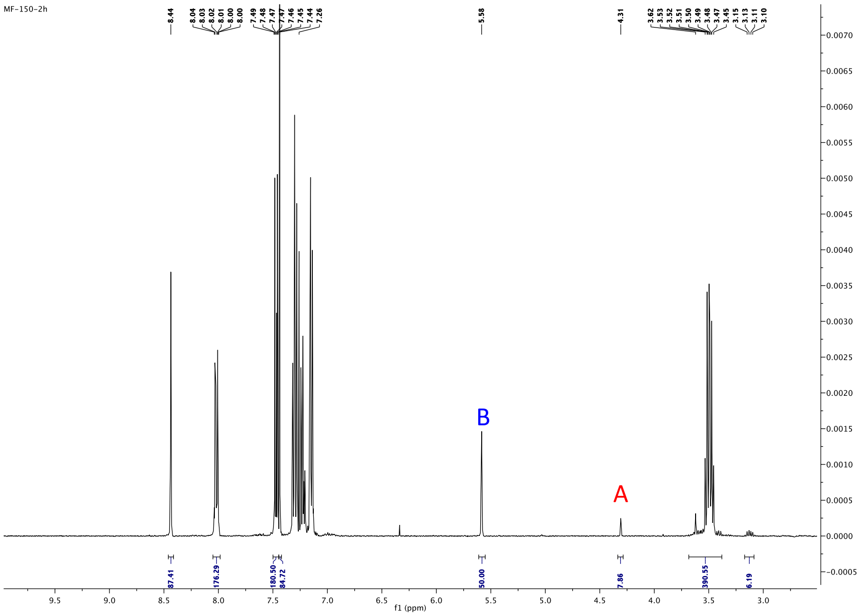


Figure 6: ^{1}H NMR spectrum of 4 + internal standard after 2 h at 215 °C in tridecane with CH heating.

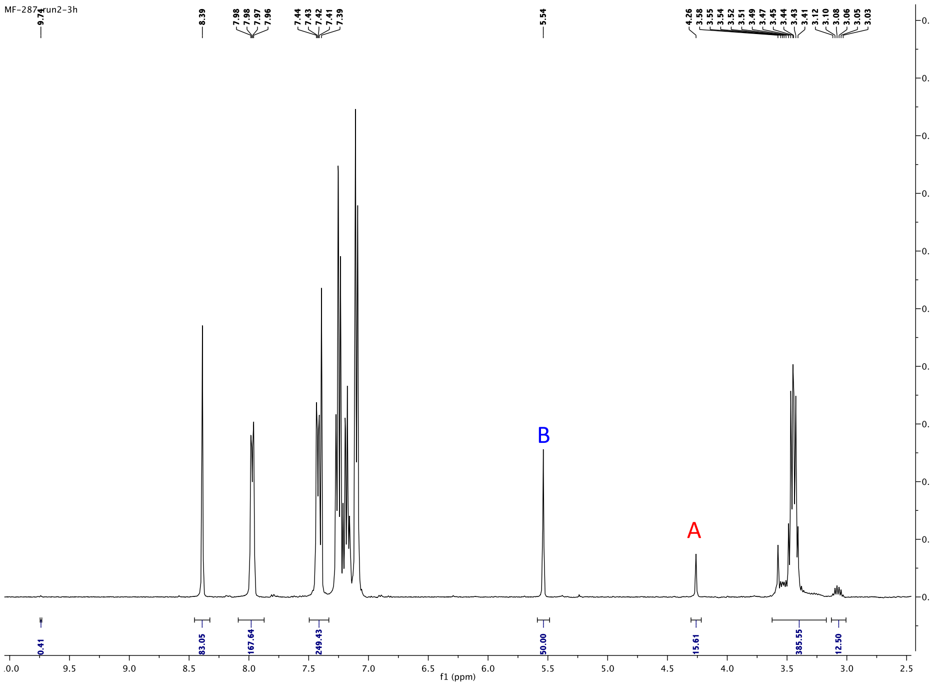


Figure 7: ^{1}H NMR spectrum of 4 + SPAN 60 + internal standard after 3 h at 200 °C in tridecane with MW heating.

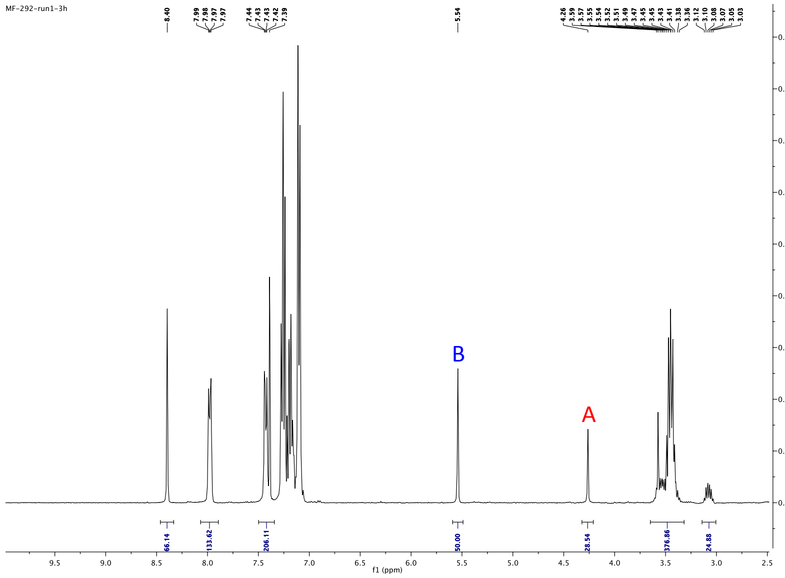


Figure 8: ^{1}H NMR spectrum of 4 + SPAN 60 + internal standard after 3 h at 200 °C in tridecane with CH heating.

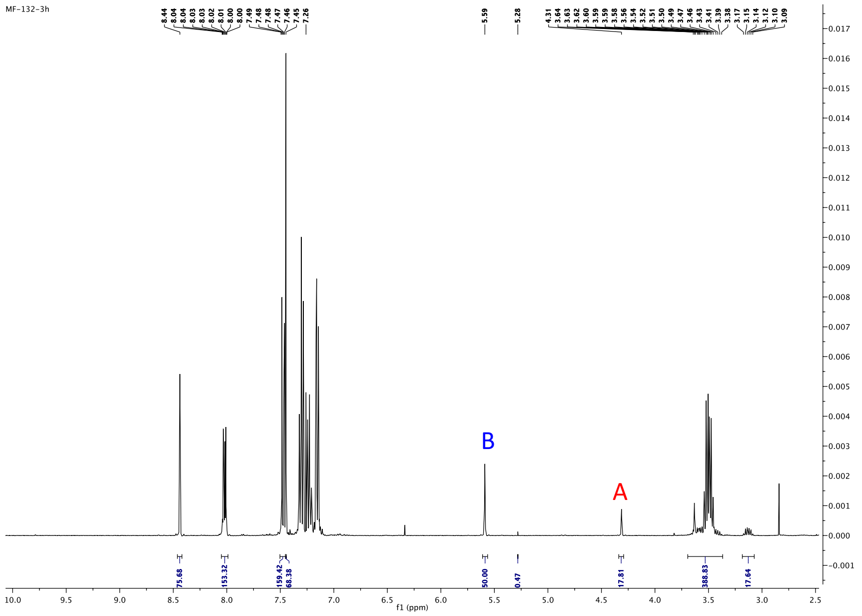


Figure 9: ^{1}H NMR spectrum of 0.1 M 4 + internal standard after 3 h at 200 °C in tridecane with MW heating.

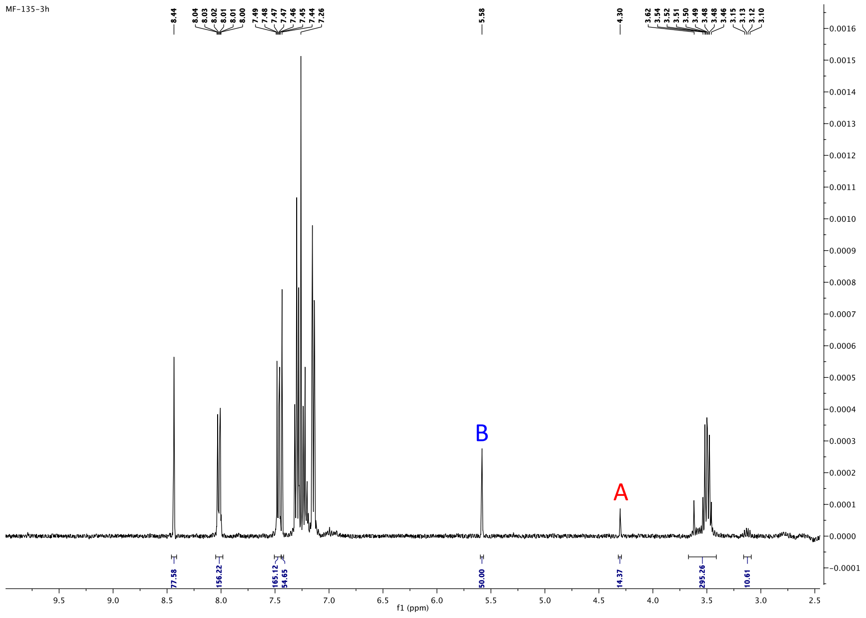


Figure 10: ^{1}H NMR spectrum of 0.02 M 4 + internal standard after 3 h at 200 °C in tridecane with MW heating.

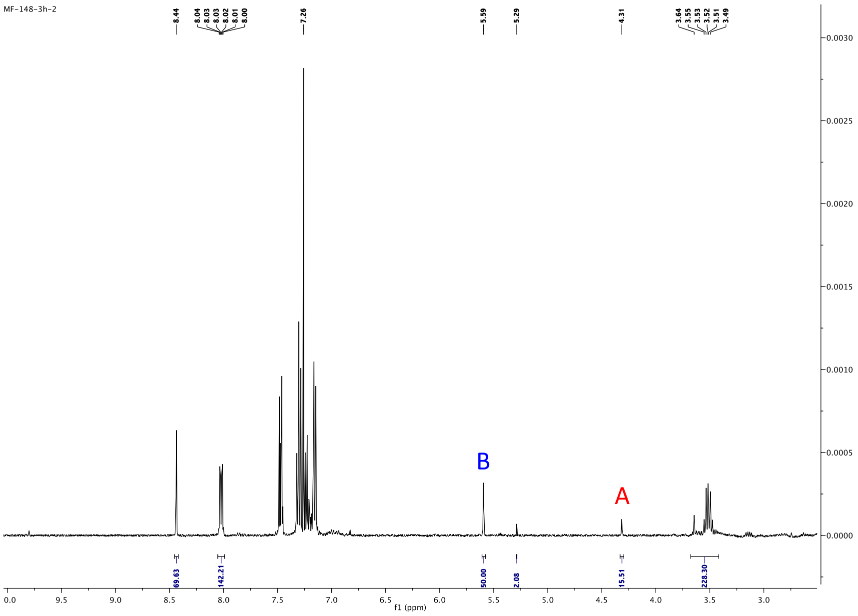


Figure 11: ^{1}H NMR spectrum of 0.01 M 4 + internal standard after 3 h at 200 °C in tridecane with MW heating.

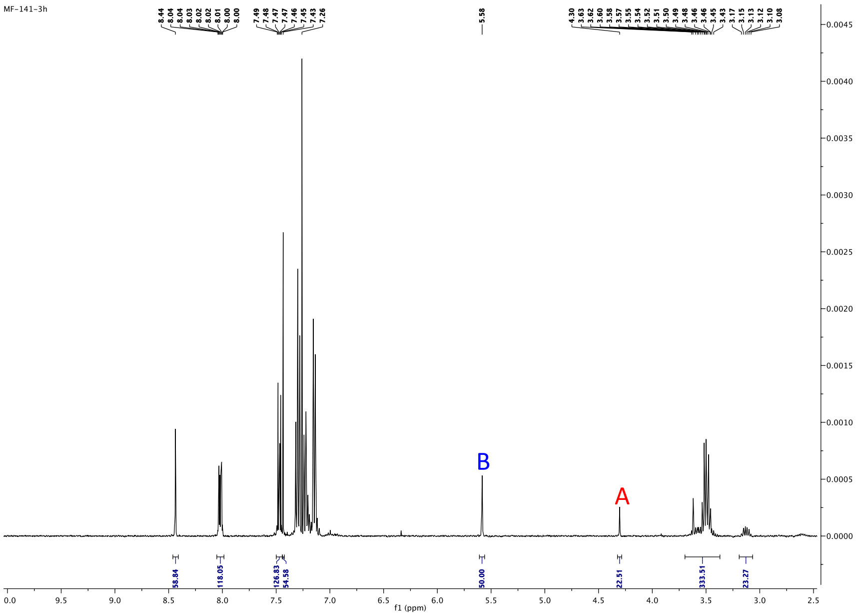


Figure 12: ^{1}H NMR spectrum of 0.02 M 4 + internal standard after 3 h at 200 °C in tridecane with CH heating.

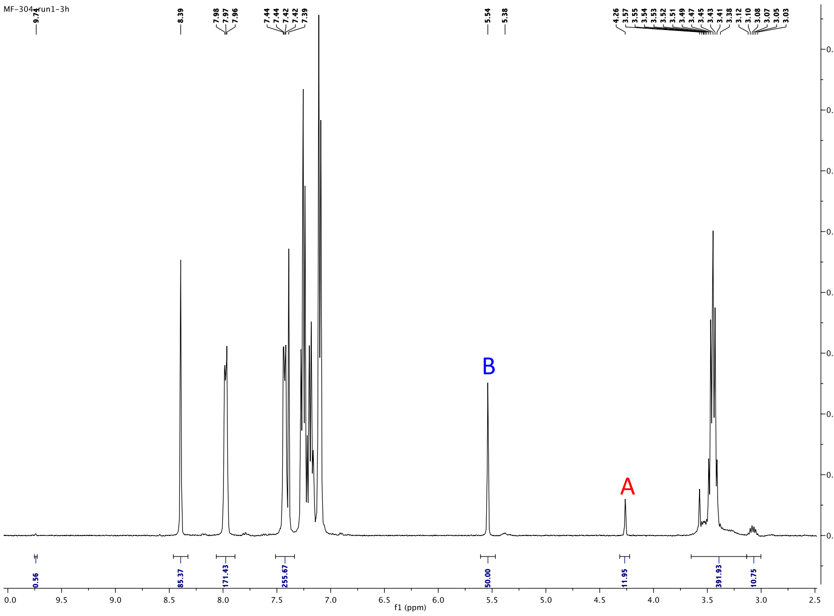


Figure 13: ^{1}H NMR spectrum of 0.065 M 4 + internal standard after 3 h at 200 °C in heptadecane with MW heating.

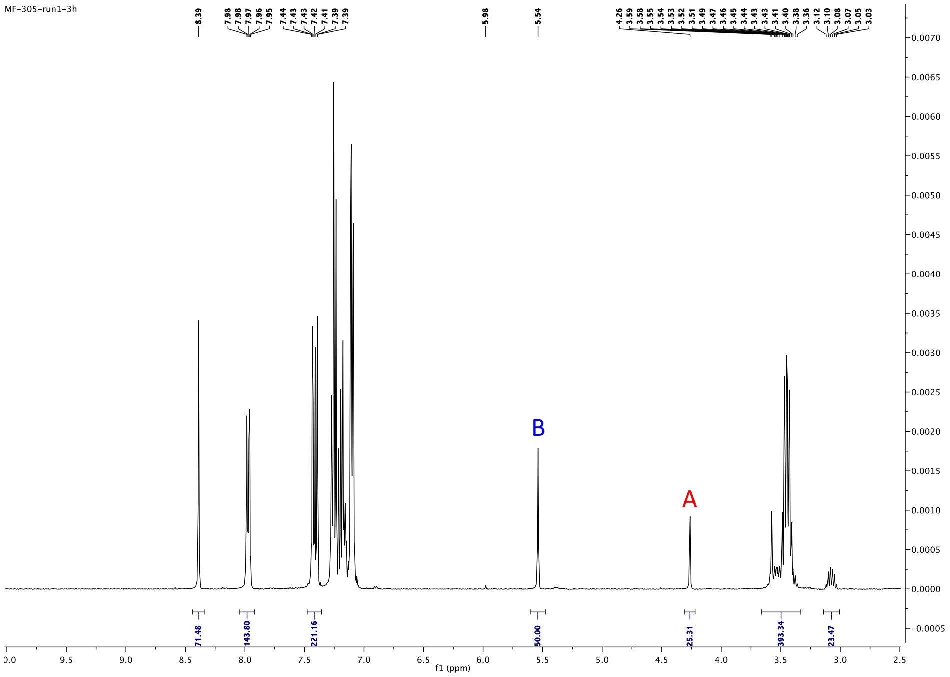


Figure 14: ^{1}H NMR spectrum of 0.065 M 4 + internal standard after 3 h at 200 °C in heptadecane with CH heating.

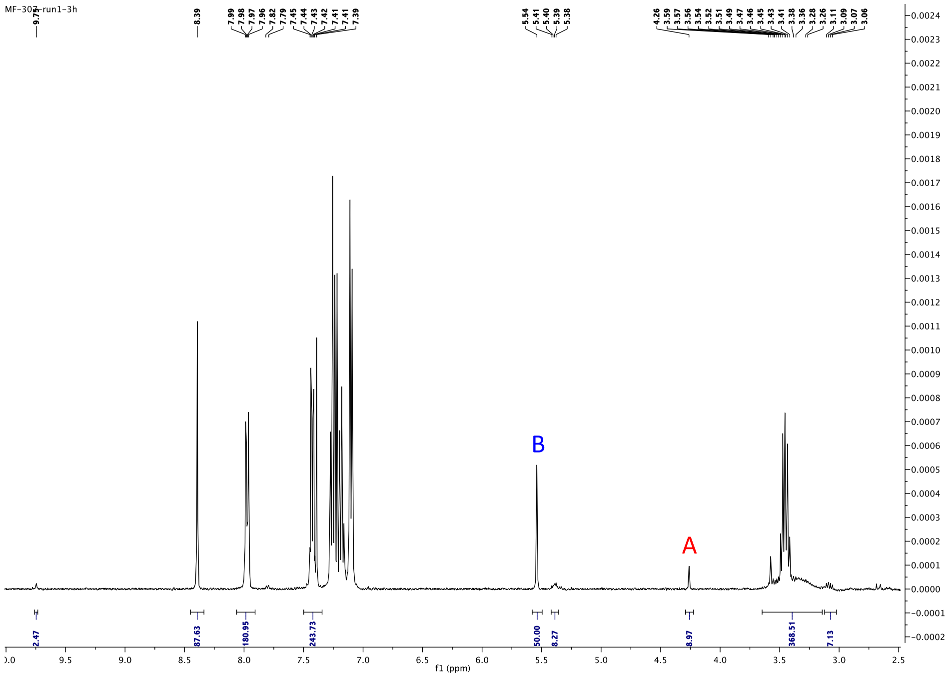


Figure 15: ^{1}H NMR spectrum of 0.065 M 4 + internal standard after 3 h at 200 °C in heptadecane with MW heating.

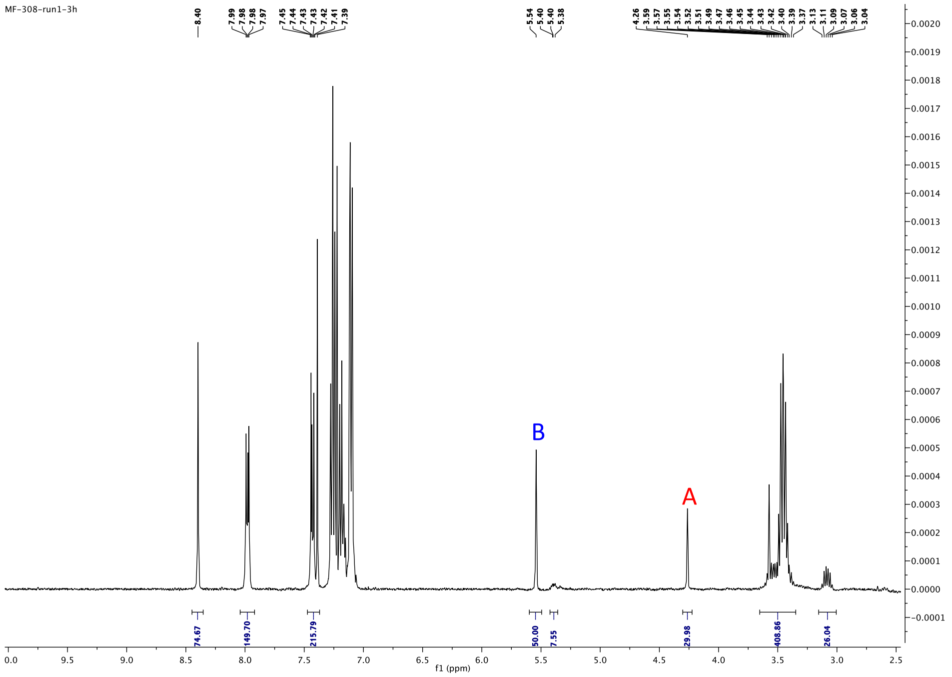


Figure 16: ^{1}H NMR spectrum of 0.065 M 4 + internal standard after 3 h at 200 °C in heptadecane with CH heating.

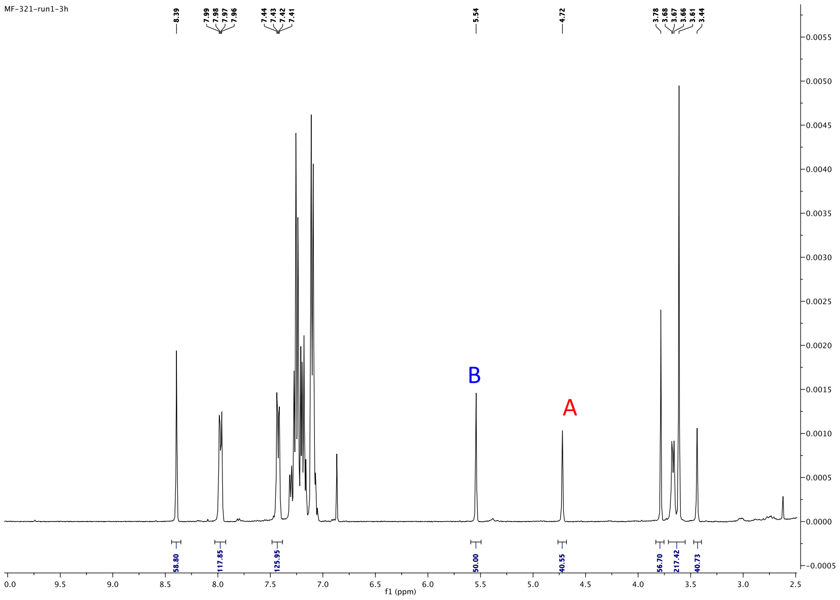


Figure 17: ^{1}H NMR spectrum of 6 + internal standard after 3 h at 200 °C in tridecane with MW heating.

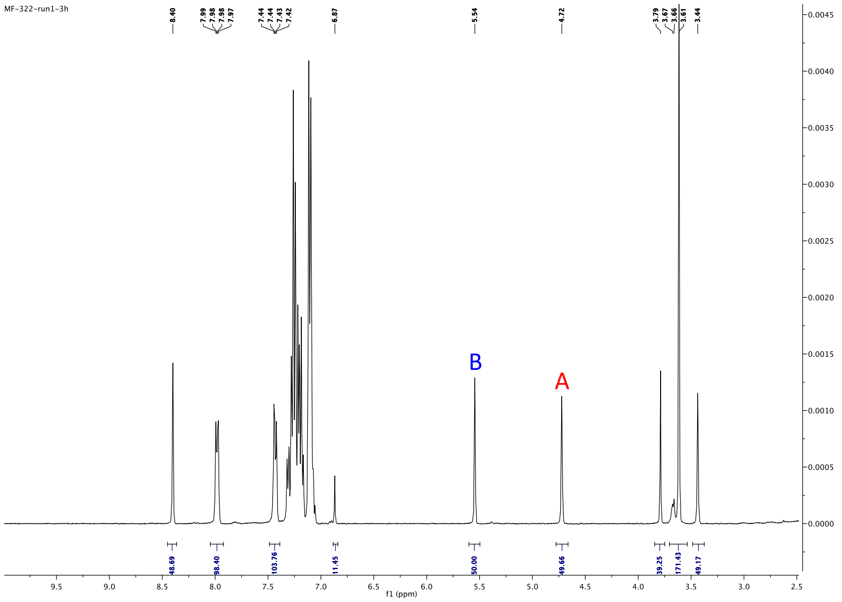


Figure 18: ^{1}H NMR spectrum of 6 + internal standard after 3 h at 200 °C in tridecane with CH heating.

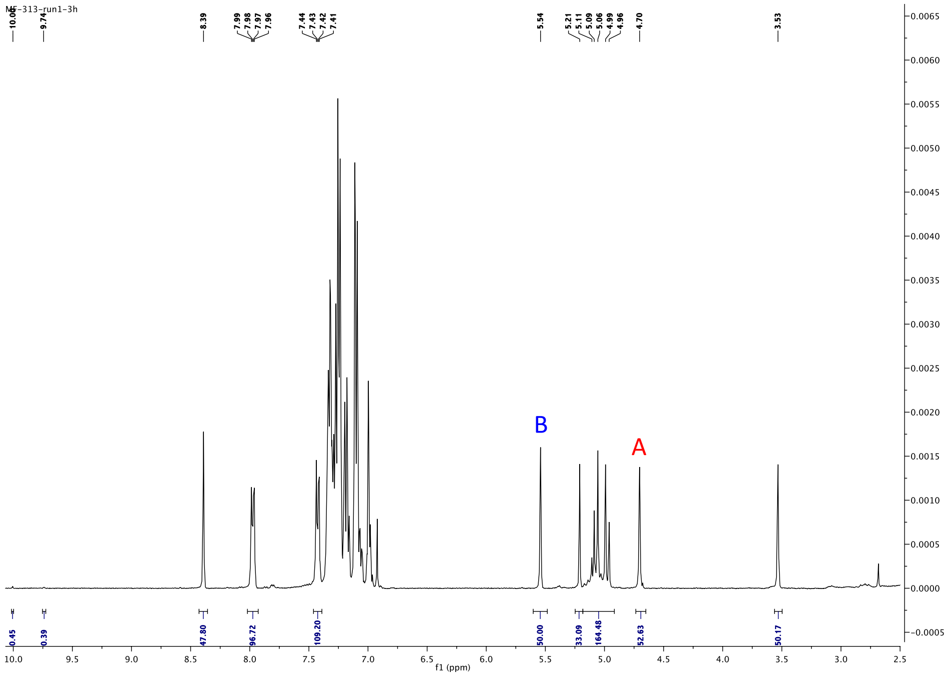


Figure 19: ^{1}H NMR spectrum of 7 + internal standard after 3 h at 200 °C in tridecane with MW heating.

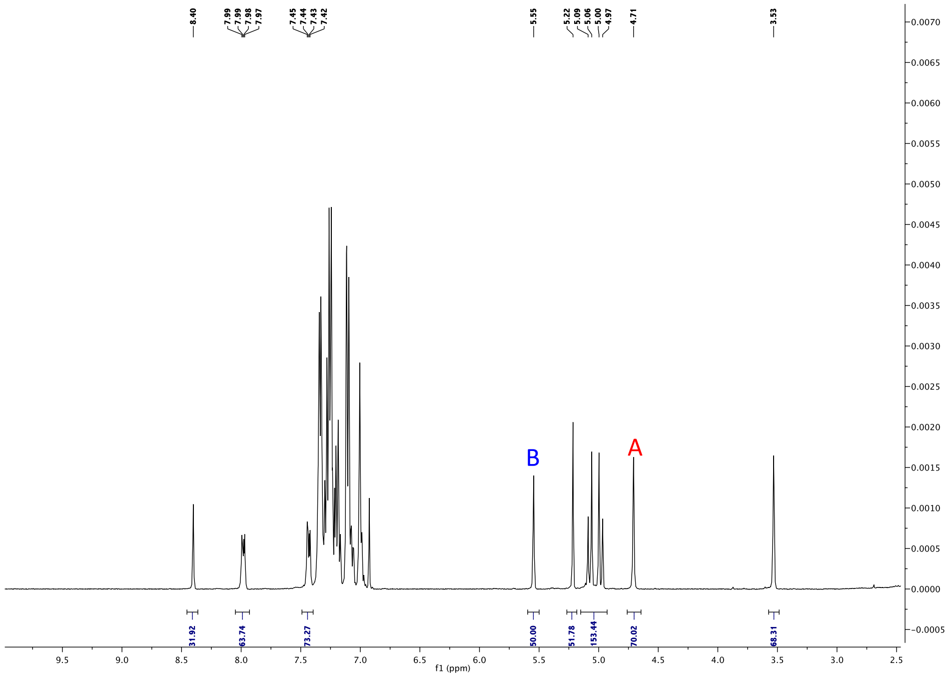
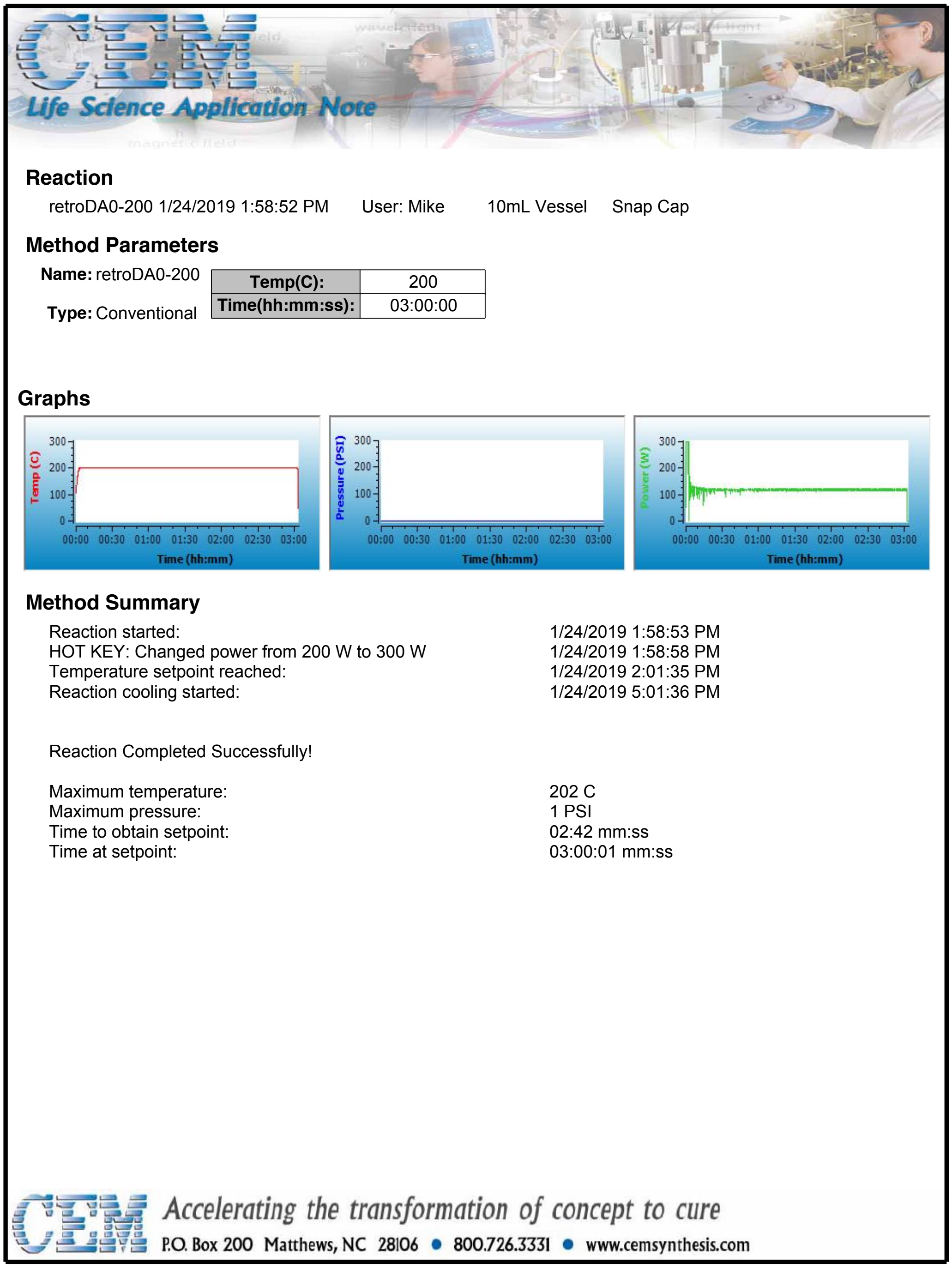


Figure 20: ^{1}H NMR spectrum of 7 + internal standard after 3 h at 200 °C in tridecane with CH heating.

Representative CEM Data for MW Reactions



Life Science Application Note

Reaction

retroDA0-200 1/24/2019 1:58:52 PM User: Mike 10mL Vessel Snap Cap

Method Parameters

Name: retroDA0-200

Type: Conventional

Temp(C):	200
Time(hh:mm:ss):	03:00:00

Graphs

Method Summary

Reaction started:	1/24/2019 1:58:53 PM
HOT KEY: Changed power from 200 W to 300 W	1/24/2019 1:58:58 PM
Temperature setpoint reached:	1/24/2019 2:01:35 PM
Reaction cooling started:	1/24/2019 5:01:36 PM

Reaction Completed Successfully!

Maximum temperature:	202 C
Maximum pressure:	1 PSI
Time to obtain setpoint:	02:42 mm:ss
Time at setpoint:	03:00:01 mm:ss

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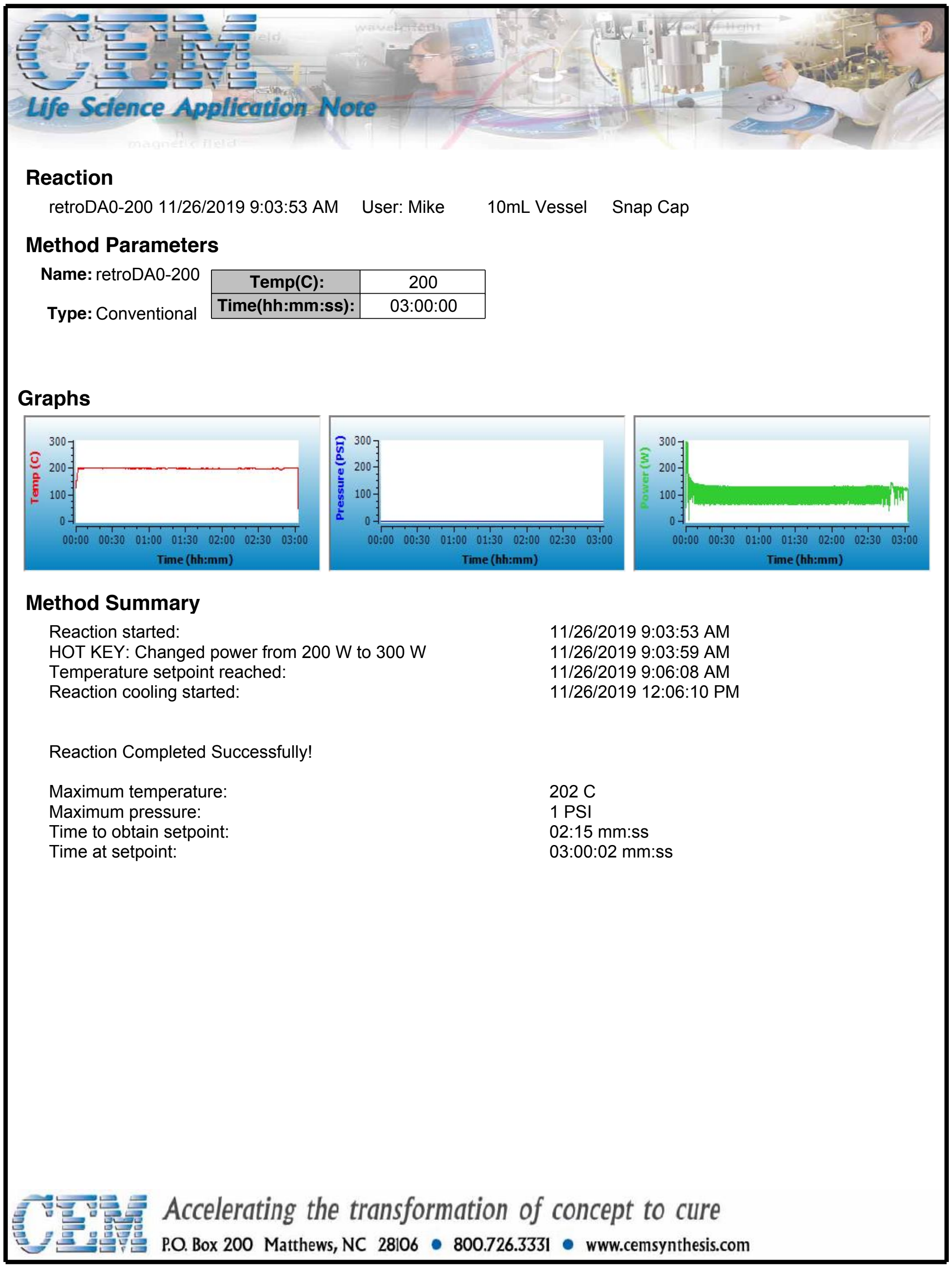
Figure 21: Representative heating profile of 0.065 M 4 in tridecane for 3 h at 200 °C in MW.



Reaction

retroDA0-200 11/26/2019 9:03:53 AM User: Mike 10mL Vessel Snap Cap

Method Parameters

Name: retroDA0-200

Type: Conventional

Temp(C):	200
Time(hh:mm:ss):	03:00:00

Graphs

Method Summary

Reaction started:	11/26/2019 9:03:53 AM
HOT KEY: Changed power from 200 W to 300 W	11/26/2019 9:03:59 AM
Temperature setpoint reached:	11/26/2019 9:06:08 AM
Reaction cooling started:	11/26/2019 12:06:10 PM

Reaction Completed Successfully!

Maximum temperature:	202 C
Maximum pressure:	1 PSI
Time to obtain setpoint:	02:15 mm:ss
Time at setpoint:	03:00:02 mm:ss

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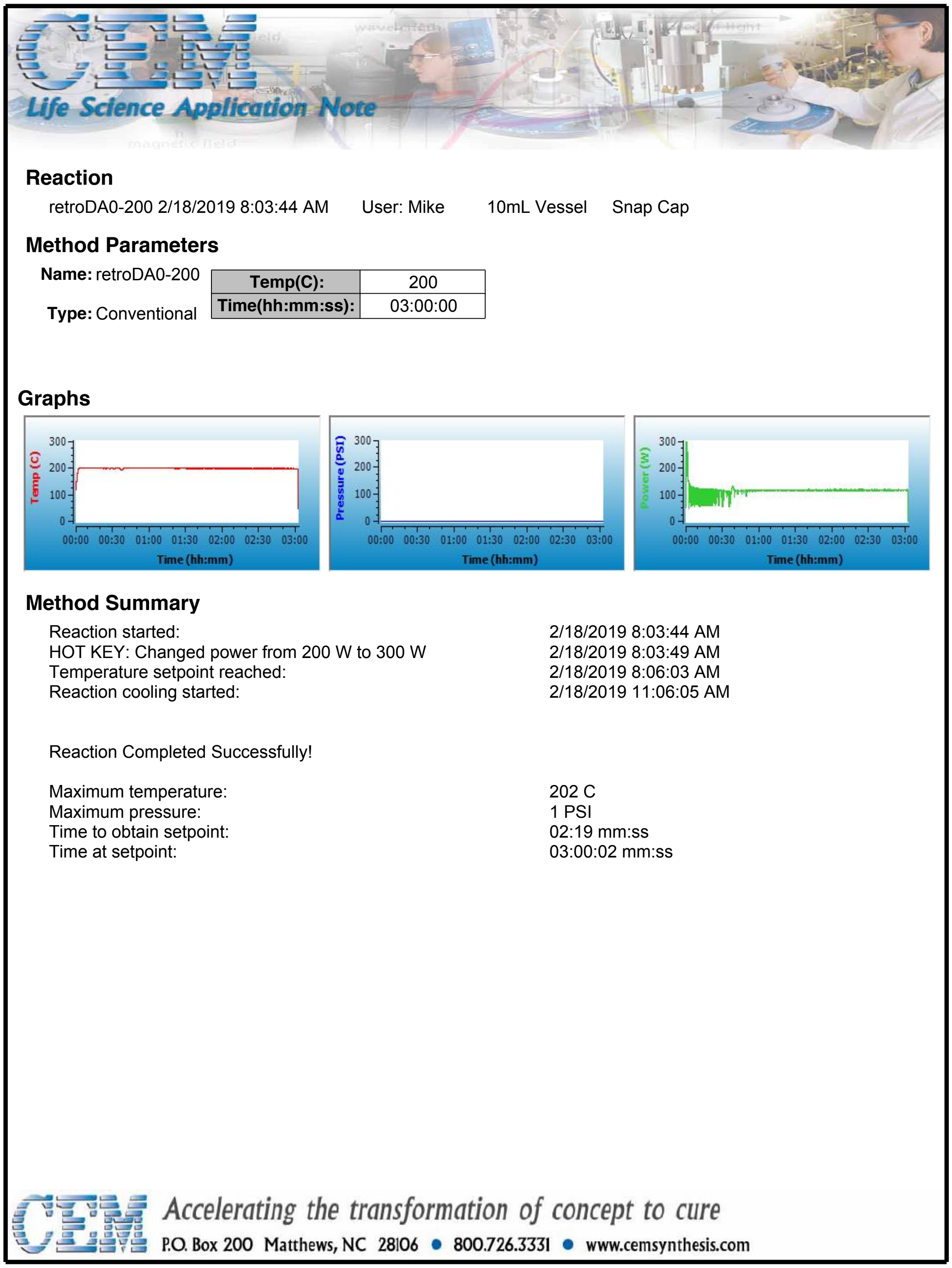
Figure 22: Representative heating profile of 4 + SPAN 60 in tridecane for 3 h at 200 °C in MW.



Life Science Application Note

Reaction

retroDA0-200 2/18/2019 8:03:44 AM User: Mike 10mL Vessel Snap Cap

Method Parameters

Name: retroDA0-200

Type: Conventional

Temp(C):	200
Time(hh:mm:ss):	03:00:00

Graphs

Method Summary

Reaction started:	2/18/2019 8:03:44 AM
HOT KEY: Changed power from 200 W to 300 W	2/18/2019 8:03:49 AM
Temperature setpoint reached:	2/18/2019 8:06:03 AM
Reaction cooling started:	2/18/2019 11:06:05 AM

Reaction Completed Successfully!

Maximum temperature:	202 C
Maximum pressure:	1 PSI
Time to obtain setpoint:	02:19 mm:ss
Time at setpoint:	03:00:02 mm:ss

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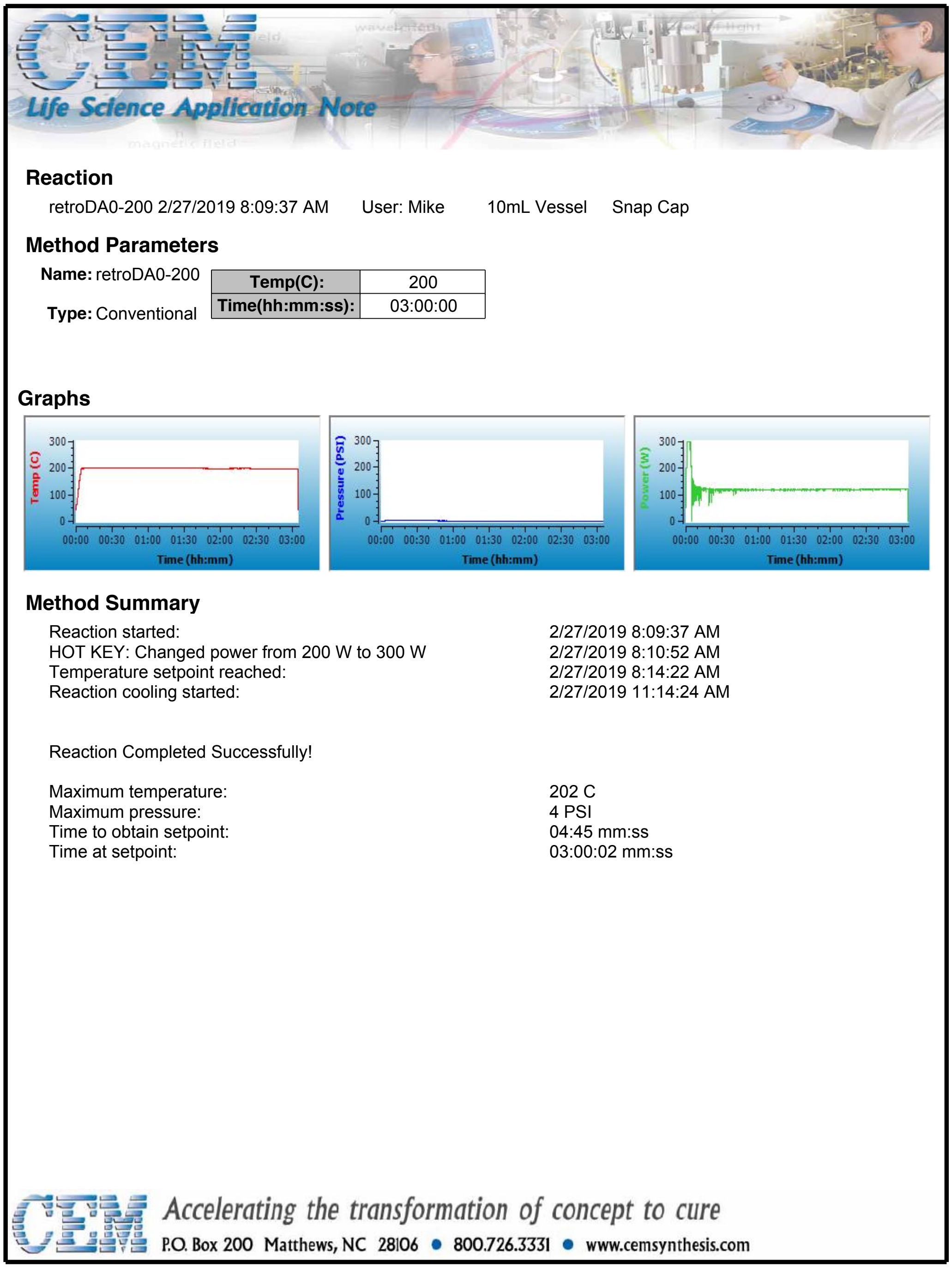
Figure 23: Representative heating profile of 0.1 M 4 in tridecane for 3 h at 200 °C in MW.



Life Science Application Note

Reaction

retroDA0-200 2/27/2019 8:09:37 AM User: Mike 10mL Vessel Snap Cap

Method Parameters

Name: retroDA0-200

Type: Conventional

Temp(C):	200
Time(hh:mm:ss):	03:00:00

Graphs

Method Summary

Reaction started:	2/27/2019 8:09:37 AM
HOT KEY: Changed power from 200 W to 300 W	2/27/2019 8:10:52 AM
Temperature setpoint reached:	2/27/2019 8:14:22 AM
Reaction cooling started:	2/27/2019 11:14:24 AM

Reaction Completed Successfully!

Maximum temperature:	202 C
Maximum pressure:	4 PSI
Time to obtain setpoint:	04:45 mm:ss
Time at setpoint:	03:00:02 mm:ss

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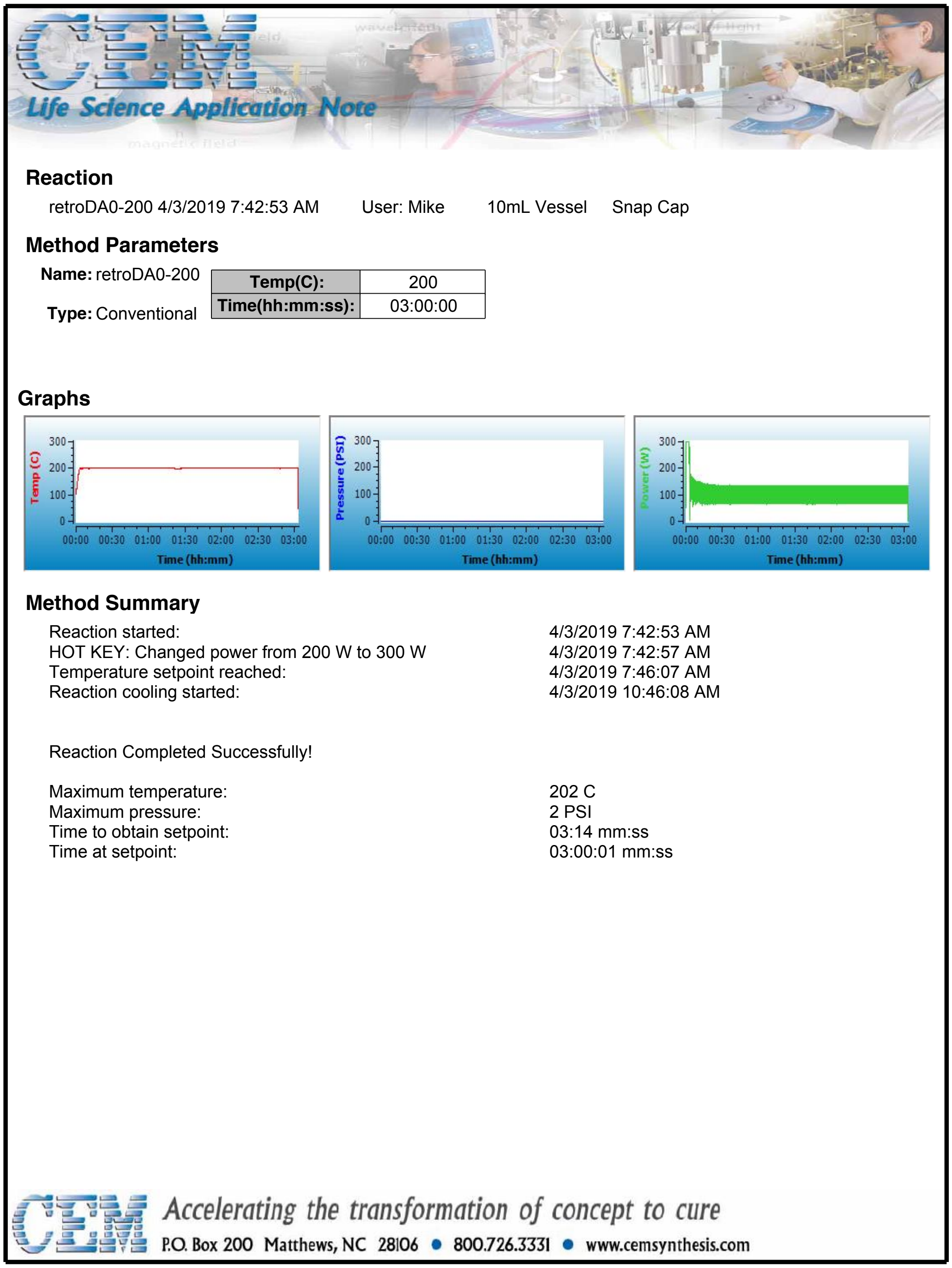
Figure 24: Representative heating profile of 0.02 M 4 in tridecane for 3 h at 200 °C in MW.



CEM

Life Science Application Note

Reaction

retroDA0-200 4/3/2019 7:42:53 AM User: Mike 10mL Vessel Snap Cap

Method Parameters

Name: retroDA0-200

Type: Conventional

Temp(C):	200
Time(hh:mm:ss):	03:00:00

Graphs

Method Summary

Reaction started:	4/3/2019 7:42:53 AM
HOT KEY: Changed power from 200 W to 300 W	4/3/2019 7:42:57 AM
Temperature setpoint reached:	4/3/2019 7:46:07 AM
Reaction cooling started:	4/3/2019 10:46:08 AM

Reaction Completed Successfully!

Maximum temperature:	202 C
Maximum pressure:	2 PSI
Time to obtain setpoint:	03:14 mm:ss
Time at setpoint:	03:00:01 mm:ss

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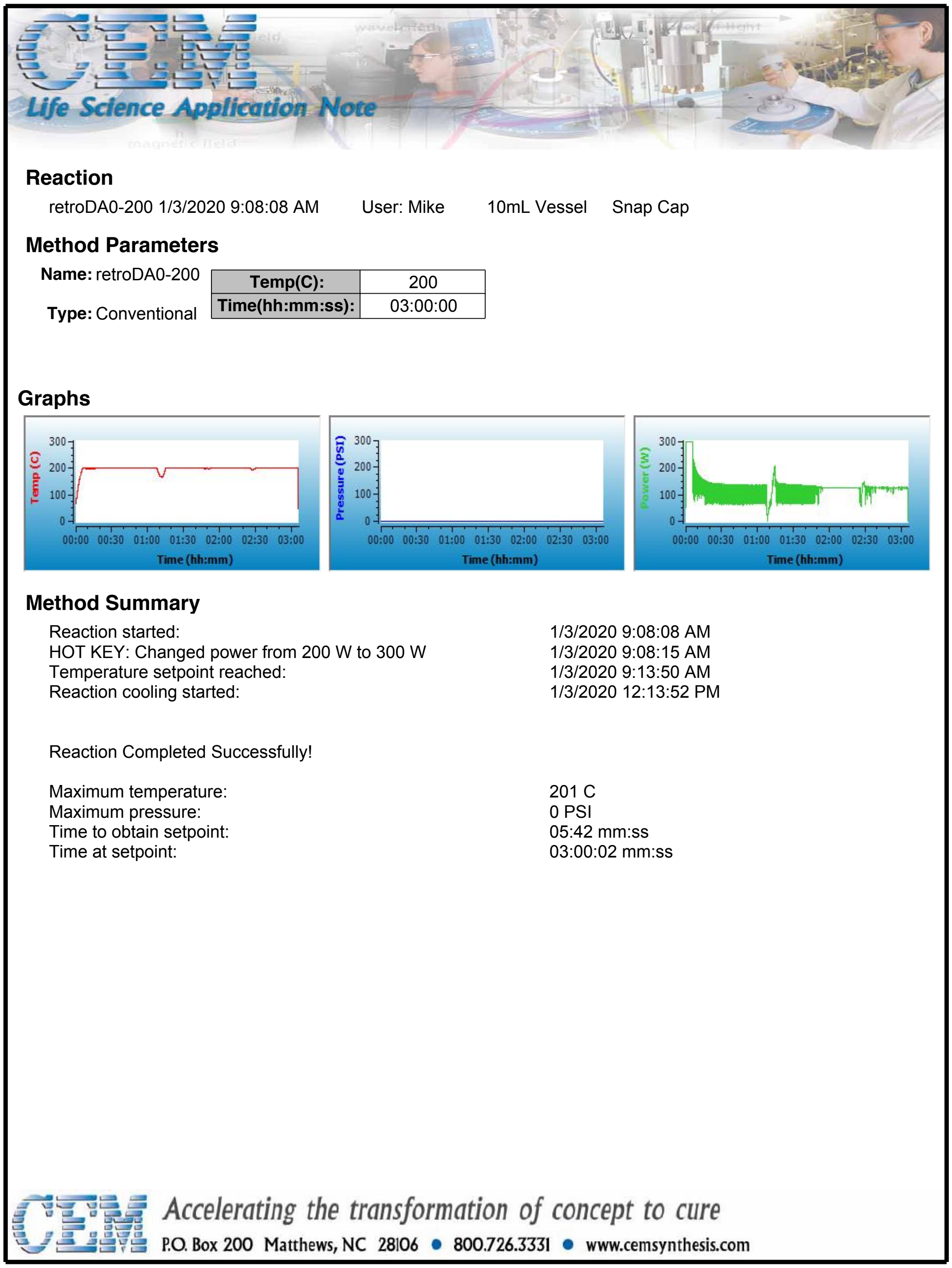
Figure 25: Representative heating profile of 0.01 M 4 in tridecane for 3 h at 200 °C in MW.



Life Science Application Note

Reaction

retroDA0-200 1/3/2020 9:08:08 AM User: Mike 10mL Vessel Snap Cap

Method Parameters

Name: retroDA0-200

Type: Conventional

Temp(C):	200
Time(hh:mm:ss):	03:00:00

Graphs

Method Summary

Reaction started:	1/3/2020 9:08:08 AM
HOT KEY: Changed power from 200 W to 300 W	1/3/2020 9:08:15 AM
Temperature setpoint reached:	1/3/2020 9:13:50 AM
Reaction cooling started:	1/3/2020 12:13:52 PM

Reaction Completed Successfully!

Maximum temperature:	201 C
Maximum pressure:	0 PSI
Time to obtain setpoint:	05:42 mm:ss
Time at setpoint:	03:00:02 mm:ss

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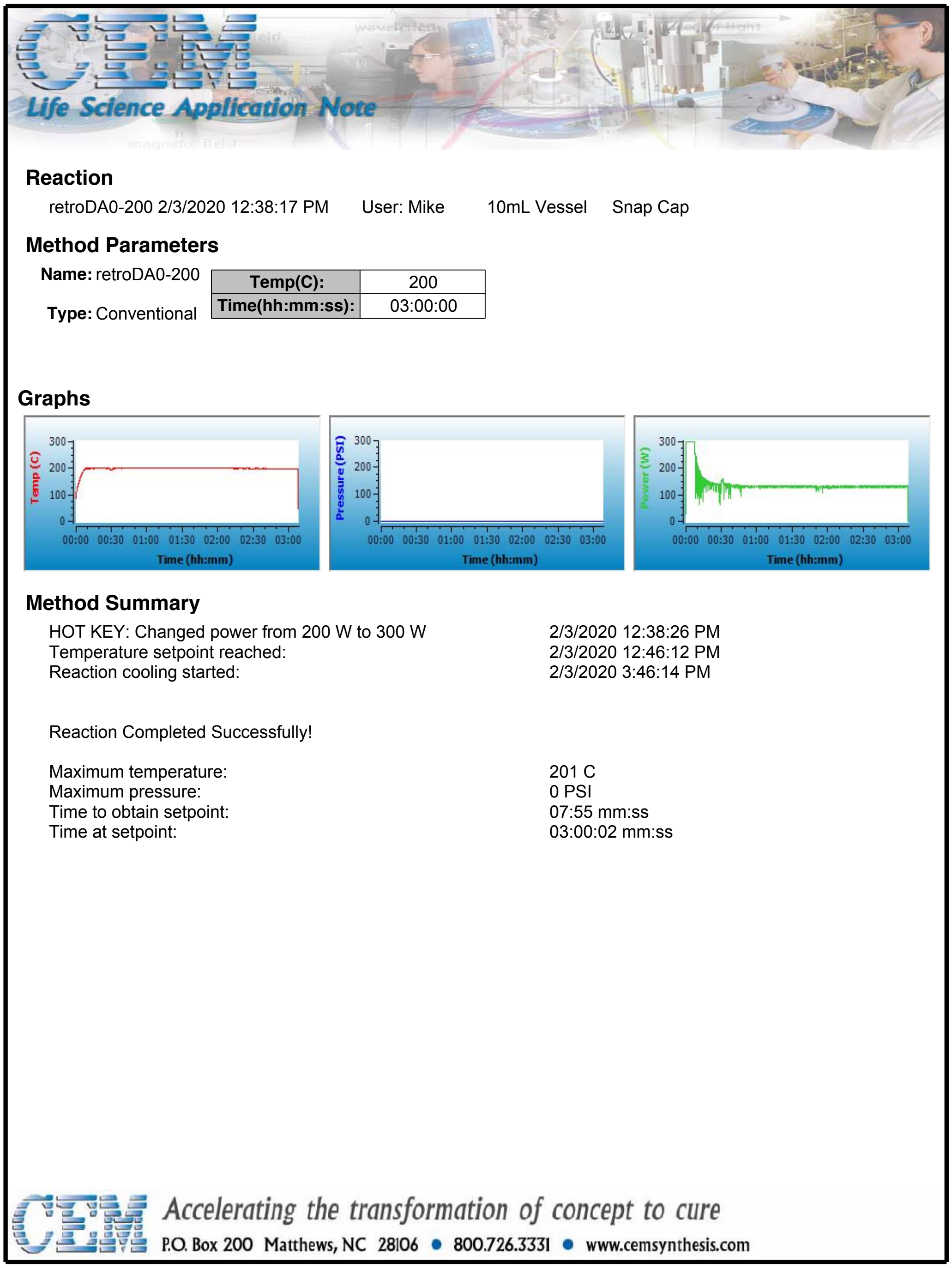
Figure 26: Representative heating profile of 0.065 M 4 in heptadecane for 3 h at 200 °C in MW.



Life Science Application Note

Reaction

retroDA0-200 2/3/2020 12:38:17 PM User: Mike 10mL Vessel Snap Cap

Method Parameters

Name: retroDA0-200

Type: Conventional

Temp(C):	200
Time(hh:mm:ss):	03:00:00

Graphs

Method Summary

HOT KEY: Changed power from 200 W to 300 W 2/3/2020 12:38:26 PM
Temperature setpoint reached: 2/3/2020 12:46:12 PM
Reaction cooling started: 2/3/2020 3:46:14 PM

Reaction Completed Successfully!

Maximum temperature: 201 C
Maximum pressure: 0 PSI
Time to obtain setpoint: 07:55 mm:ss
Time at setpoint: 03:00:02 mm:ss

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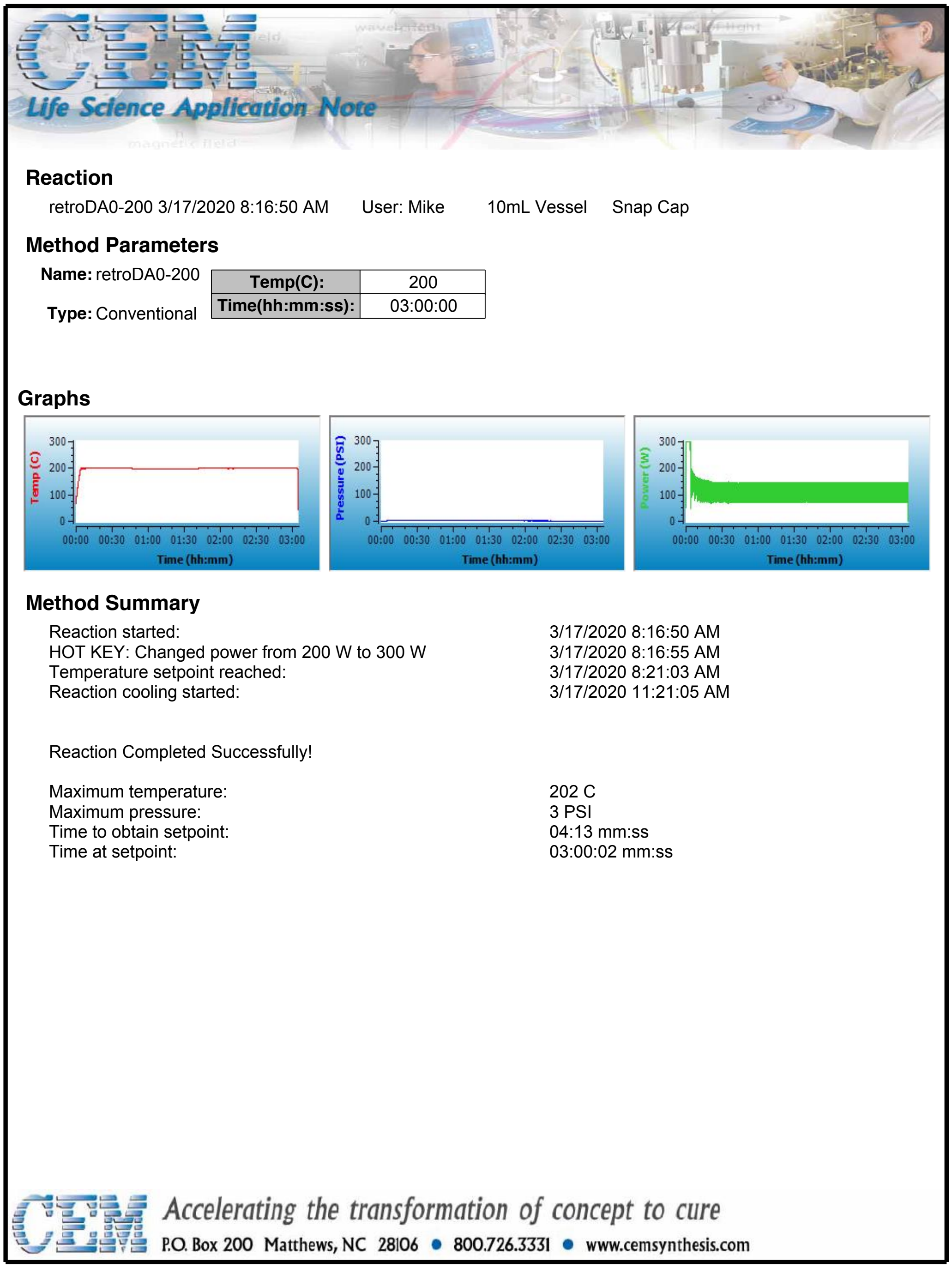
Figure 27: Representative heating profile of 0.02 M 4 in heptadecane for 3 h at 200 °C in MW.



Reaction

retroDA0-200 3/17/2020 8:16:50 AM User: Mike 10mL Vessel Snap Cap

Method Parameters

Name: retroDA0-200

Type: Conventional

Temp(C):	200
Time(hh:mm:ss):	03:00:00

Graphs

Method Summary

Reaction started:	3/17/2020 8:16:50 AM
HOT KEY: Changed power from 200 W to 300 W	3/17/2020 8:16:55 AM
Temperature setpoint reached:	3/17/2020 8:21:03 AM
Reaction cooling started:	3/17/2020 11:21:05 AM

Reaction Completed Successfully!

Maximum temperature:	202 C
Maximum pressure:	3 PSI
Time to obtain setpoint:	04:13 mm:ss
Time at setpoint:	03:00:02 mm:ss

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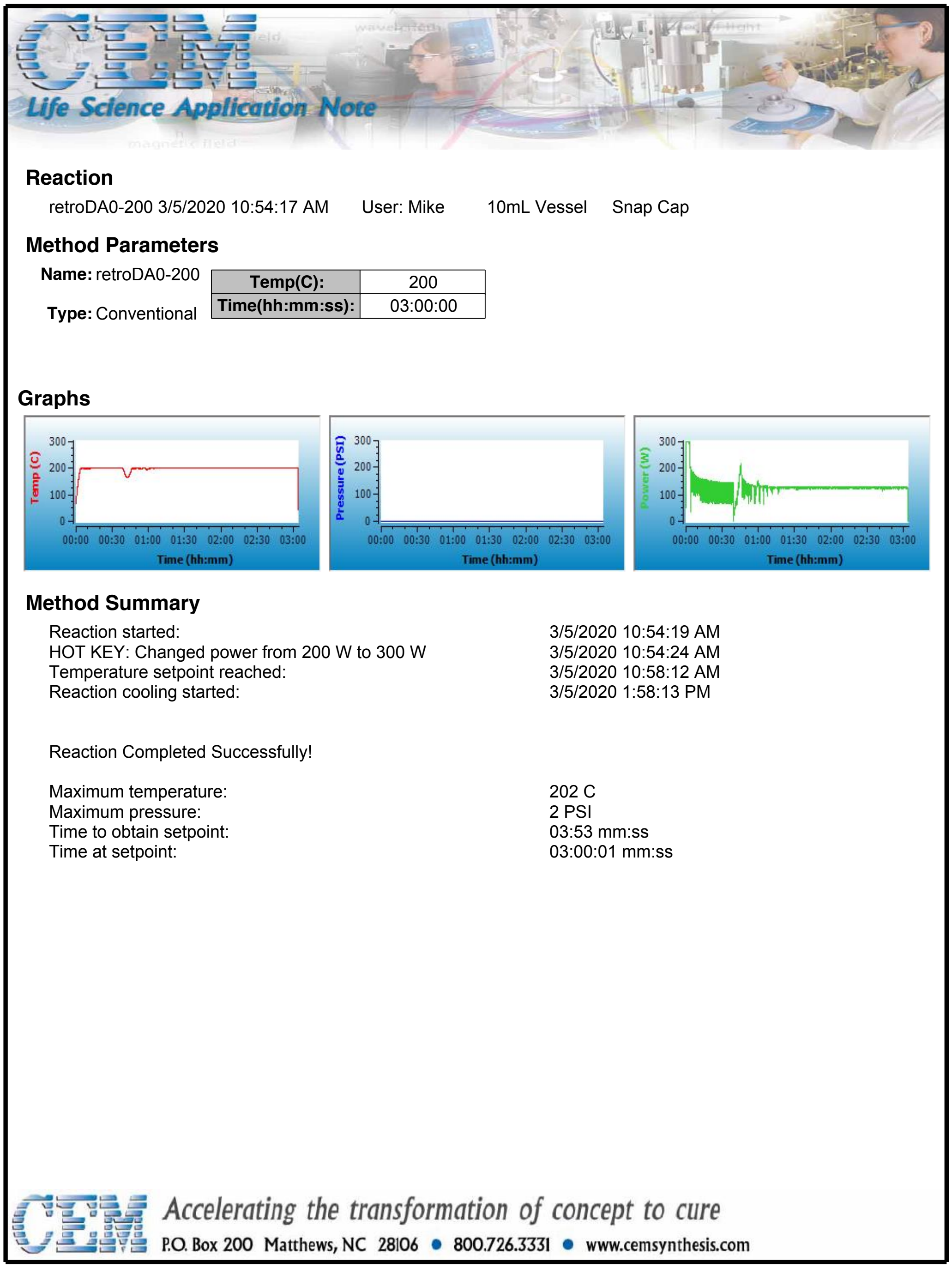
Figure 28: Representative heating profile of M 6 in tridecane for 3 h at 200 °C in MW.



Life Science Application Note

Reaction

retroDA0-200 3/5/2020 10:54:17 AM User: Mike 10mL Vessel Snap Cap

Method Parameters

Name: retroDA0-200

Type: Conventional

Temp(C):	200
Time(hh:mm:ss):	03:00:00

Graphs

Method Summary

Reaction started:	3/5/2020 10:54:19 AM
HOT KEY: Changed power from 200 W to 300 W	3/5/2020 10:54:24 AM
Temperature setpoint reached:	3/5/2020 10:58:12 AM
Reaction cooling started:	3/5/2020 1:58:13 PM

Reaction Completed Successfully!

Maximum temperature:	202 C
Maximum pressure:	2 PSI
Time to obtain setpoint:	03:53 mm:ss
Time at setpoint:	03:00:01 mm:ss

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Figure 29: Representative heating profile of M 7 in tridecane for 3 h at 200 °C in MW.

References

1. Naidu, A. B.; Jaseer, E. A.; Sekar, G., General, mild, and intermolecular Ullmann-type synthesis of diaryl and alkyl aryl ethers catalyzed by diol-copper(I) complex. *J. Org. Chem.* **2009,** *74*, 3675-3679.
2. Winkler, J. D.; Piatnitski, E. L.; Mehlmann, J.; Kasparec, J.; Axelsen, P. H., Design and Synthesis of Foldamers Based on an Anthracene Diels-Alder Adduct *Angew. Chem. Int. Ed. Engl.* **2001,** *40*, 743-745.