Parallel Synthesis and Purification using Anthracene-Tagged Substrates

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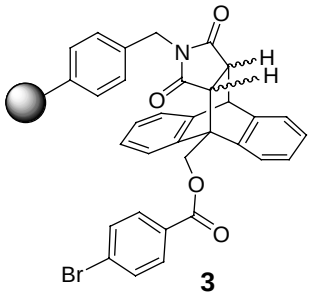
Supporting Information

General Information: ^{1}H NMR spectra were recorded on a 400 MHz spectrometer at ambient temperature with $CDCl_3$ as the solvent unless otherwise stated. ^{13}C NMR spectra were recorded on a 75.0 MHz spectrometer at ambient temperature. Chemical shifts are reported in parts per million relative to chloroform (^{1}H, δ 7.24; ^{13}C, δ 77.0). Data for ^{1}H NMR are reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, b = broad) and coupling constants. All ^{13}C NMR spectra were recorded with complete proton decoupling. Infrared spectra were recorded on either a Perkin-Elmer 1800 series IR, Nicolet Nexus 670 FT-IR, or a Nicolet Impact spectrophotometer equipped with an Inspect IR microscope. High-resolution mass spectra were obtained in the Boston University Mass Spectrometry Laboratory using a Finnegan MAT-90 spectrometer. UV absorbances were measured using a Spectronic 20^{+} (Spectronic Instruments). Unfunctionalized polystyrene resin was obtained from Argonaut Technologies (San Carlos, CA). Methylene chloride (CH_2Cl_2) and toluene were distilled from calcium hydride. Tetrahydrofuran was distilled from sodium and benzophenone. Elemental Analysis was performed by Galbraith Laboratories (Knoxville, TN). HPLC analysis was performed on a Dynamax (Model SD-200, Varian) using [CH_3CN:H_2O 10-100% (9 min), then 100% CH_3CN (6 min)]. HPLC purities were determined by area under the curve integration using UV detection at 223 nm. All parallel synthesis transformations were conducted using Quest 210 and FirstMate Synthesizers (Argonaut Technologies). Analytical thin layer chromatography was performed on 0.25 mm silica gel 60-F plates. Flash chromatography was performed using 200-400 mesh silica gel (Natland International Corporation). 3-Bromo-4-methylbenzoic acid chloride was prepared from 3-bromo-4-methylbenzoic acid using oxalyl chloride

(1.1 equiv) *cat.* DMF/CH_2Cl_2. All other reagents were used as supplied by Sigma-Aldrich, Fluka, Lancaster, and Strem Chemicals unless noted otherwise. ChemElut Plus 1 mL aqueous capacity (6 mL cartridge) were obtained from Argonaut Technologies (Part. No. 900266).

Procedure for preparation of *N*-benzylmaleimide resin (2): To a 250 mL 2-neck flask (pre-silanized with 5% TMSCl/hexanes and oven dried) were added unfunctionalized polystyrene resin (5 g) followed by 50 mL freshly distilled CH_2Cl_2 under nitrogen. A mechanical stirring apparatus was attached and the reaction mixture was cooled to 0 ^oC. $FeCl_3$ (367 mg, 2.26 mmol, 0.23 equiv) was added followed by *N*-chloromethylmaleimide **1** (1.46 g, 10 mmol, 1 equiv) dissolved in 20 mL CH_2Cl_2. The reaction was warmed to room temperature and mechanically stirred for 5 h. Using a vacuum suction tube, the resin was washed 2 x 100 mL CH_2Cl_2, 4 x 100 mL 1:1 THF/HCl(1M), 4 x 100 mL THF and dried under high vacuum overnight to yield *N*-benzylmaleimide resin **2**. IR (KBr): 1710, 826 cm^{-1}; Elemental analysis (N) = 2.27 % (1.62 mmol/g).



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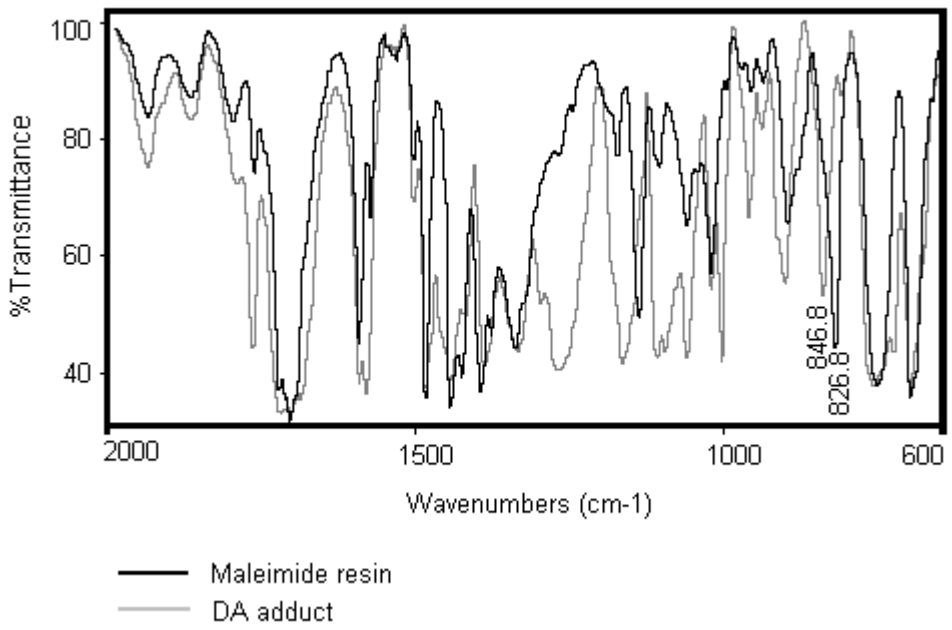
Procedure for preparation of *N*-benzylmaleimide resin-anthracene tag Diels-Alder adduct (3): To a 5 mL Quest 210 RV was added *N*-benzylmaleimide resin (1.6 mmol/g, 200 mg, 0.32 mmol, 1 equiv), followed by anthracene **4a** (250 mg, 0.64 mmol, 2 equiv) and 2 mL toluene. After heating at 90^oC for 20 h, the reaction mixture was cooled to room temperature and washed 3 x 3 mL toluene, 3 x 3 mL 1:1 THF/MeOH, and dried under high vacuum overnight to yield Diels-Alder adduct resin **3**. IR (KBr) 705, 764, 1728 cm^{-1}; Elemental analysis (N) = 1.33 % (0.95 mmol/g) (Br) = 8.32 % (1.04 mmol/g).

IR Spectra of *N*-benzylmaleimide resin (2) and Diels-Alder Adduct (3):

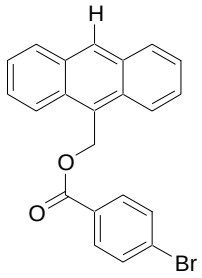


Examination of the infrared band at 826 cm^{-1} (out-of-plane hydrogen deformation of the *cis*-disubstituted double bond of the maleimide group conjugated with carbonyl group[1]) may be used to monitor conversion of the maleimide resin **2** to the Diels-Alder adduct **3** when using an excess of anthracene tag. The band at 846.8 cm^{-1} of the adduct is derived from the anthracene-tagged bromide.

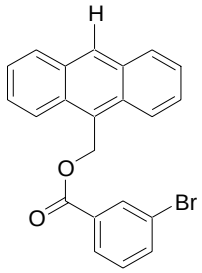
General Procedure for preparation of anthracene-tagged benzoates: To a 25 mL round-bottomed flask, 9-anthracenemethanol (1.04 g, 5 mmol, 1.2 equiv) was suspended in 10 mL of freshly distilled CH_2Cl_2 under nitrogen and cooled to 0^oC. Distilled triethylamine (611 μL, 5 mmol, 1.2 equiv) was then added followed by 3-bromobenzoic acid chloride (529 μL, 4 mmol, 1 equiv) dissolved in 5 mL CH_2Cl_2 and the reaction mixture was warmed to rt. After 6 h, the reaction mixture was diluted with 20 mL CH_2Cl_2, and subsequently washed with 5 mL distilled water, 5 mL saturated NH_4Cl, and 5 mL saturated NaCl, dried with $MgSO_4$, and concentrated under reduced pressure. The

(1) Wang, C. S.; Lin, C.H. *Polymer* **1999**, *40*, 5665-5773.

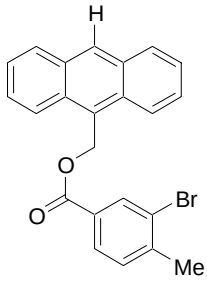
residue was purified by column chromatography (30 % CH_2Cl_2 in hexane) to yield **4b** as a yellow solid (1.47 g, 94 % yield). Anthracene-tagged 3-bromobenzoate **6** was prepared in a similar fashion by acylation of 9-(hydroxymethyl)-10-methylanthracene.[2]



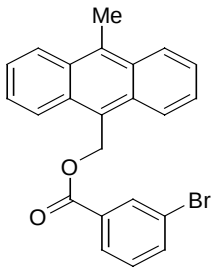
4a: m.p. 163-166 ^{o}C, IR (neat): 1713, 1267, 1101, 1012, cm^{-1}; ^{1}H NMR: δ 8.52 (1H, s), 8.40 (2H, d, J=8.8), 8.04 (2H, d, J=8.8), 7.83 (2H, d, J=6.4), 7.58 (2H, m), 7.51-7.46 (4H, m), 6.37 (3H, s) ppm; ^{13}C NMR : δ 167.1, 132.7, 132.5, 132.3, 132.2, 130.4, 130.2, 130.0, 129.2, 127.8, 127.0, 126.2, 125.0, 60.7 ppm; HRMS (EI): calcd. for $C_{22}H_{15}BrO_2$ 390.0255, found 390.0265.



4b: m.p. 124-126 ^{o}C, IR (neat): 1718, 1252, 1116 cm^{-1}; ^{1}H NMR: δ 8.53 (1H, s), 8.40 (2H, d, J=8.8), 8.10 (1H, s), 8.04 (2H, d, J=8.4), 7.90 (1H, d, J=8.0), 7.62-7.56 (3H, m), 7.50 (2H, m), 7.21 (1H, t, J=8.0) ppm; ^{13}C NMR : δ 166.5 137.0, 133.8, 133.0, 132.5, 132.3, 130.9, 130.5, 130.2, 129.4, 127.9, 126.9, 126.2, 124.9, 123.5, 60.8 ppm; HRMS (EI): calcd. for $C_{22}H_{15}BrO_2$ 390.0255, found 390.0234.



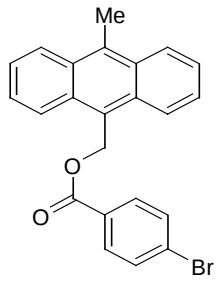
4c: m.p. 139-142 ^{o}C, IR (neat): 1714, 11280, 1248, 1107 cm^{-1}; ^{1}H NMR: δ 8.52 (1H, s), 8.40 (2H, d, J=8.8), 8.12 (1H, d, J=1.6), 8.04 (2H, d, J=8.4), 7.80 (1H, dd, J=8.0, 1.6), 7.60-7.55 (2H, m), 7.51-7.47 (2H, m), 7.18 (1H, d, J=8.0), 6.36 (3H, s) 2.37 (3H, s) ppm; ^{13}C NMR : δ 166.6, 144.5, 134.6, 132.5, 132.3, 131.7, 130.4, 130.2, 129.6, 127.8, 127.1, 126.2, 125.8, 125.0, 60.7, 24.2 ppm; HRMS (EI): calcd. for $C_{23}H_{17}BrO_2$ 404.0412, found 404.0387.



6: m.p. 126-129 ^{o}C, IR (neat): 1718, 1251, 1118 cm^{-1}; ^{1}H NMR: δ 8.42 (2H, d, J=8.4), 8.37 (2H, d, J=8.8), 8.10 (1H, s), 7.91 (1H, d, J=8.0), 7.56 (4H, m), 7.21 (1H, t, J=8.0), 6.38 (3H, s), 3.14 (3H, s) ppm; ^{13}C NMR: δ 167.0, 137.0, 134.3, 133.7, 133.2, 132.0, 130.9, 129.4, 128.6,

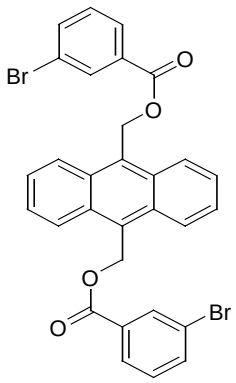
(2) Wigal, C. T.; McKinley, J. D.; Coyle, J.; Porter, D. J.; Lehman, D. E. *J. Org. Chem.* **1995**, *60*, 8421-8423.

127.3, 126.5, 126.4, 126.1, 125.5, 125.4, 123.5, 122.2, 61.1, 15.6 ppm; HRMS (EI): calcd. for $C_{23}H_{17}BrO_2$ 404.0412, found 404.0381.



8: m.p. 178-181 ^{o}C, IR (neat): 1710, 1261, 1102, 1013 cm^{-1}; ^{1}H NMR: δ 8.39 (4H, dd, J=26, 8.8), 7.83 (2H, d, J=7.2), 7.56 (4H, m), 7.46 (2H, d, J=8.8), 6.37 (3H, s), 3.13 (3H, s) ppm; ^{13}C NMR : δ 167.2, 134.2, 132.7, 132.4, 132.0, 131.0, 130.1, 129.2, 127.3, 126.5, 126.1, 125.6, 60.9, 15.6 ppm; HRMS (EI): calcd. for $C_{23}H_{17}BrO_2$ 404.0412, found 404.0446.

Procedure for preparation of 9,10 bis-functionalized anthracene (7): To a 25 mL round bottom flask 9,10-bischloromethylanthracene[3] (0.55 g, 2 mmol, 1 equiv) and 3-bromobenzoic acid (1.005 g, 5 mmol, 2.5 equiv) were suspended in 10 mL DMF under nitrogen, followed by addition of Cs_2CO_3 (1.63 g, 5 mmol, 2.5 equiv). After heating at 35^{o}C for 20 h, the reaction mixture was cooled to room temperature, washed 2 x 10 H_2O mL, 2 x 10 mL CH_2Cl_2, 2 x 10 mL EtOAc, and dried under high vacuum to afford **7** (1.003 g, 83 % yield) as a yellow solid.

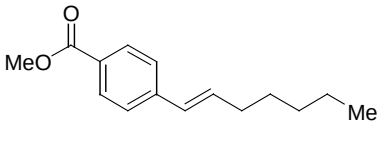


7: m.p. 206-209 ^{o}C, IR (neat): 1728, 1241 cm^{-1}; ^{1}H NMR: δ 8.48 (4H, dd, J=6.8, 3.2), 8.10 (1H, s), 7.91 (1H, d, J=7.6), 7.63 (5H, m), 7.23 (1H, t, J=8.0), 6.42 (3H, s) ppm; HRMS (EI): calcd. for $C_{30}H_{20}Br_2O_4$ 601.9728, found 601.9778.

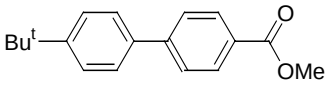
General procedure for Parallel Stille Coupling Reactions (Table 1, entry 1**)** 9-anthracenemethyl-4-bromobenzoate (**4a)** (78.2 mg, 0.2 mmol, 1 equiv) was weighed into an oven-dried 13x150 mm test tube, which was positioned into the Firstmate Synthesizer (Argonaut Technologies). Phenyltributyltin (0.130 mL, 0.4 mmol, 2 equiv) and $Pd(PPh_3)_4$

(3) Miller, M. W.; Amidon, R. W.; Tawney, P. O. *J. Am. Chem. Soc*. **1955**, *117*, 2845-2848.

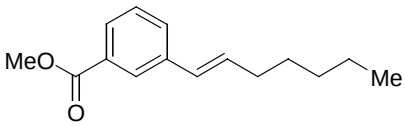
(11.6 mg, 0.01 mmol, 5 mol%) in 1 mL degassed toluene were added *via* syringe. After heating at 100 ^{o}C for 12 h under nitrogen, the reaction mixture was transferred to a 5 mL Quest 210 RV and diluted with 1 mL distilled toluene. *N*-benzylmaleimide resin **2** (1.6 mmol/g, 200 mg, 0.32 mmol, 1.6 equiv) was then added and the reaction mixture heated at 90 ^{o}C for 8 h. After cooling to rt, the resin was washed 3 x 4 mL DMF, 3 x 4 mL CH_2Cl_2, and 3 x 4 mL THF (5 min agitations each). Product cleavage was performed by addition of freshly prepared 1M NaOMe in MeOH (0.8 mL, 0.8 mmol, 4 equiv) in 2 mL THF followed by agitation for 30 minutes. Elution of the reaction mixture through a 6 mL polypropylene cartridge (Applied Separations) containing 2 mL silica gel, followed by rinsing of the cartridge 3 x 3 mL THF and concentration afforded methyl-4-phenylbenzoate[4] as a white solid (35.6 mg, 84 % yield). HPLC purity 92 % (223 nm).



5b: IR (neat): 2927, 1723, 1607, 1435, 1412, 1278, 1178, 1109 cm^{-1}; ^{1}H NMR: 7.94 (2H, d, J=8.4), 7.37 (2H, d, 8.4), 6.37 (2H, m), 3.88 (3H, s), 2.21 (2H, q, J=7.6), 1.47 (2H, m), 1.32 (4H, m), 0.89 (3H, t, J=6.4) ppm; ^{13}C NMR: δ 168.0, 143.5, 135.3, 130.9, 130.0, 129.3, 126.8, 53.0, 34.2, 32.5, 29.9, 23.6, 15.1 ppm; HRMS (EI): calcd. for $C_{15}H_{20}O_2$ 232.1463, found 232.1450.



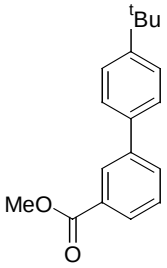
5c: m.p. 119~122 ^{o}C, IR (neat): 1717, 1280, 1105 cm^{-1}; ^{1}H NMR: δ 8.07 (2H, d, J=8.4), 7.64 (2H, d, J=8.8), 7.56 (2H, d, J=8.8), 7.47 (2H, d, J=8.0), 3.92 (3H, s), 1.35 (9H, s) ppm; ^{13}C NMR (75 MHz) δ 171.2, 155.5, 149.6, 141.2, 134.2, 132.7, 131.0, 130.0, 56.2, 38.7, 35.4 ppm; HRMS (EI): calcd. for $C_{15}H_{14}O_2$ 268.1463, found 268.1455.



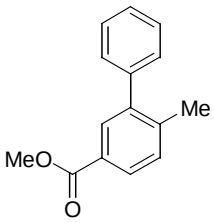
5e: IR (neat): 2927, 1725, 1439, 1289, 1203, 1107 cm^{-1}; ^{1}H NMR: δ 8.00 (1H, s), 7.83 (1H, d, J=8.0), 7.49 (1H, d, J=8.0), 7.336 (1H, t, J=8.0), 6.35 (2H, m), 3.90 (3H,

(4) Saito, S.; Oh-tani, S.; Miyaura, N. *J. Org. Chem*. **1997**, *62*, 8024-8030.

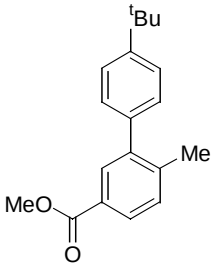
s), 2.20 (2H, q, J=7.6), 1.46 (2H, m), 1.31 (4H, m), .89 (3H, t, J=6.4) ppm; ^{13}C NMR δ 168.2, 139.4, 133.7, 131.5, 131.3, 130.0, 129.5, 128.8, 128.0, 53.0, 34.0, 32.5, 30.0, 23.6, 15.0 ppm; HRMS (EI): calcd. for $C_{15}H_{20}O_2$ 232.1463, found 232.1479.



5f: m.p. 65~67 ^{o}C, IR (neat): 1717, 1248 cm^{-1}; ^{1}H NMR: δ 8.26 (1H, s), 7.97 (1H, d, J=8.0), 7.76 (1H, d, J=7.6), 7.55 (2H, d, J=8.8), 7.50~7.46 (3H, m) ppm; ^{13}C NMR (75 MHz) δ 168.2, 151.9, 142.4, 138.2, 132.4, 131.7, 129.8, 129.1, 127.8, 126.9, 53.2, 35.6, 32.4 ppm; HRMS (EI): calcd. for $C_{18}H_{20}O_2$ 268.1463, found 268.1489.



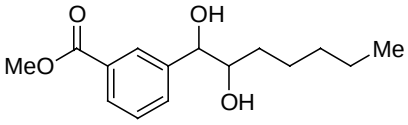
5g: IR (neat): 1719, 1308, 1240, 1108 cm^{-1}; ^{1}H NMR: δ 7.92~7.88 (2H, m), 7.41 (2H, t, J=6.8), 7.36~7.29 (4H, m), 3.88 (3H, s), 2.30 (9H, s) ppm; ^{13}C NMR (75 MHz) δ 168.1, 143.2, 142.0, 132.0, 131.5, 130.2, 129.4, 129.3, 128.9, 128.2, 53.0, 21.7 ppm; HRMS (EI): calcd. for $C_{15}H_{14}O_2$ 226.0944, found 226.1008.



5h: m.p. 83~85 ^{o}C; IR (neat): 2960, 1722, 1436, 1308, 1242, 1105 cm^{-1}; ^{1}H NMR: δ 7.91 (1H, d, J=2.0 Hz), 7.88 (1H, dd, J=8.0, 2.0 Hz), 7.42 (2H, d, J=8.4 Hz), 7.31 (1H, d, J=8.0 Hz), 7.24 (2H, d, J=8.4 Hz), 3.87 (3H, s), 2.32 (3H, s), 1.35 (9H, s) ppm; ^{13}C NMR (75 MHz) δ 168.2, 151.1, 143.0, 142.1, 139.0, 132.1, 131.4, 129.8, 129.2, 128.8, 126.1, 53.0, 35.6, 32.4, 21.9 ppm; HRMS (EI): calcd. for $C_{19}H_{22}O_2$ 282.1620, found 282.1611.

Procedure for preparation of diol 12 using multistep synthesis: 9-anthracenemethyl-3-bromobenzoate (**4b**) (78.2 mg, 0.2 mmol, 1 equiv) was weighed into an oven-dried 13 x 150 mm test tube, which was positioned into the Firstmate Synthesizer (Argonaut Technologies). (*E*)-tributyl-1-heptenyl stannane (0.123 mL, 0.32 mmol, 1.6 equiv) and $Pd(PPh_3)_4$ (11.6 mg, 0.01 mmol, 5 mol%) in 1 mL degassed toluene were added *via* syringe. After heating at 100 ^{o}C for 4 h under nitrogen, the reaction mixture was filtered through a ChemElut Plus cartridge (Argonaut Technologies) followed by rinsing of the

cartridge with 10 mL THF. Solvents were removed under reduced pressure, and the residue was then diluted with fresh THF (1.5 mL) followed by addition of NMO (37.5 mg, 0.32 mmol, 1.6 equiv) in 0.5 mL water and OsO_4 (4 wt % in water, 25 μL). After stirring at room temperature for 12 h followed by removal of volatiles, the reaction mixture was eluted through a ChemElut Plus cartridge, rinsed with 10 mL EtOAc, and the solvent concentrated *in vacuo*. The crude product was mixed with toluene (2 mL), transferred to a 5 mL Quest 210 RV, and *N*-benzylmaleimide resin **1** (1.6 mmol/g, 200 mg, 0.32 mmol, 1.6 equiv) was then added. After heating at 90^{o}C for 8 h, the resin was washed with 3 x 4 mL DMF, 3 x 4 mL CH_2Cl_2, and 3 x 4 mL THF (5 min agitations each). Cleavage was performed by addition of freshly prepared 1M NaOMe in MeOH (0.8 mL, 0.8 mmol, 4 equiv) in 2 mL THF followed by agitation for 30 minutes. Elution of the reaction mixture through a 6 mL polypropylene cartridge containing 2 mL silica gel, followed by rinsing of the cartridge 3 x 3 mL THF afforded compound diol **12** as a brown oil (34.3 mg, 64 % yield). HPLC purity 93 % (223 nm).



12: IR (neat): 3420, 1724, 1289 cm^{-1}; ^{1}H NMR: δ 7.97 (1H, s), 7.94 (1H, d, J=7.6), 7.52 (1H, d, J=7.6), 7.40 (1H, t, J=7.6), 4.49 (1H, d, J=6.4), 3.88 (3H, s), 3.67 (1H, m), 2.80 (1H, s), 2.29 (1H, s), 1.50~1.12 (8H, m), 0.82 (3H, t, J=6.4) ppm; ^{13}C NMR δ 168.0, 143.0, 132.4, 131.4, 130.2, 129.6, 129.0, 77.6, 77.0, 53.2, 33.8, 32.7, 26.4, 23.6, 15.0 ppm; HRMS (CI): calcd. for $[M+1]^{+}$ $C_{15}H_{23}O_4$ 267.1596, found 267.1635.

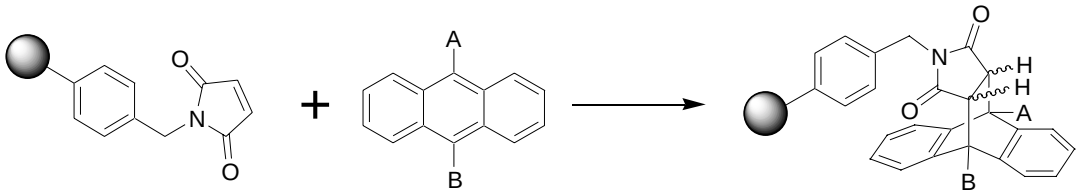
Procedure for comparison of Diels-Alder kinetics of anthracene tags: To a 10 mL Quest 210 reaction vessel were added **4b** (39.1 mg, 0.1 mmol, 1 equiv), *N*-benzylmaleimide resin (1.05 mmol/g, 120 mg, 0.126 mmol, 1.26 equiv), followed by 10 mL freshly distilled toluene. The reaction was heated at 90 ^{o}C and periodically the solution was filtered and collected into a 100 x 13 mm test tube to obtain the absorbance using a Spectronics 20^{+} UV spectrometer (wavelength=410 nm)[5] and was then readded to the reaction vessel for further heating. A solution of **4b (**19.5 mg, 0.05 mmol) in 10 mL

(5) UV wavelength = 430 nm was used for anthracene-tagged compound **7**.

toluene at 90 ^{o}C was prepared and tested for absorbance (0.21) as the standard for calculation of the concentration of **4b**. Absorbance *vs*. time data obtained, as well as calculated tag concentrations and y_2 values are shown in **Table 1**.

Table 1

T (h)	Absorbance	Concentration of tag (mmol/L)	y_2 (l/mol)
0.5	0.324	7.71	22.9
1.5	0.29	6.9	34.1
2.5	0.24	5.71	55.4
4	0.234	5.57	58.4



The Diels-Alder reaction shown above is a second-order reaction according to literature precedent.[6] The following kinetic equation (**1**) was used for calculation of rate constants (M = maleimide resin; D = anthracene tag):

$$-\frac{d[D]}{dt} = k_2 \cdot [M] \cdot [D] = k_2 \cdot ([D] + c) \cdot [D] \qquad (\mathbf{1})$$

$$k_2 t = \frac{1}{c} \cdot \ln \frac{[D] + c}{[D]} - \frac{1}{c} \cdot \ln \frac{[M]_0}{[D]_0} = y_2 \qquad (\mathbf{2})$$

k_2 ($M^{-1}h^{-1}$) : second-order reaction rate constant;

t (h) : reaction time;

$[M]_0$: initial concentration of Maleimide;

$[D]_0$: initial concentration of anthracene tag;

$c = [M]_0 - [D]_0$;

[D]: concentration of anthracene tag after "t" hours.

Equation **2** derived from equation **1** shows a linear relationship between y_2 and t. The concentration of the tag was used to calculate the y_2 value. After drawing y_2-t plots, best fit lines were drawn in order to determine the slope (rate constants k_2).

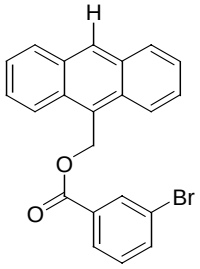
[6] (a) Mielert, A.; Braig, C.; Sauer, J.; Martelli, J.; Sustmann, R. *Liebigs Ann. Chem.* **1980**, 954-970. (b) Fleischhauer, J.; Asaad, A. N.; Schleker, W.; Scharf, H-D. *Liebigs Ann. Chem.* **1981**, 306-311.

Table 2

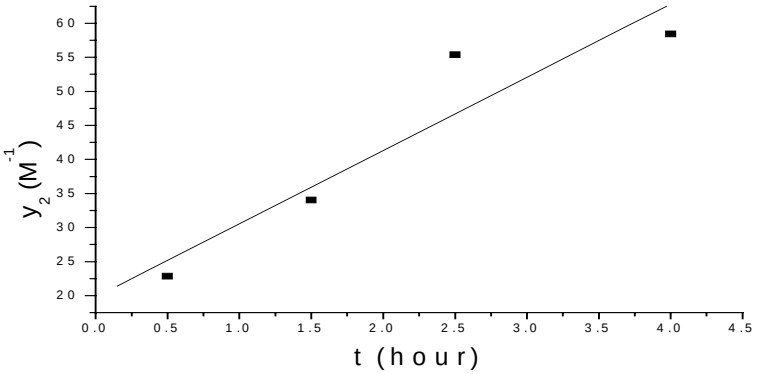
Anthracene tag	k_2 ($M^{-1}h^{-1}$)	Scavenging time using condition 1 (h)	Scavenging time using condition 2 (h)
4b	10.8	13	5.8
6	146	1	0.43
7	48	3	1.3

Condition 1: time for >99% scavenging calculated using kinetic rate equations using 1.2 equiv resin (1.05 mmol/g). 10 mL solvent was used for per gram resin.
Condition 2: time for >99% scavenging calculated using kinetic rate equations using 1.6 equiv resin (1.6 mmol/g). 10 mL solvent was used for per gram resin.

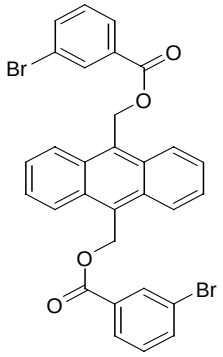
Table 2 shows k_2 ($M^{-1}h^{-1}$) determined for tagged compounds **4b**, **6** and **7** at 90 ^{o}C (toluene), as well as estimated scavenging times for these compounds using two conditions. Considering the activities will decrease with increasing concentrations of tag and maleimide resin, in experiments operationally slightly longer times were used for anthracene tag removal.



4b



$k_2 = 10.8\ M^{-1}h^{-1}$.



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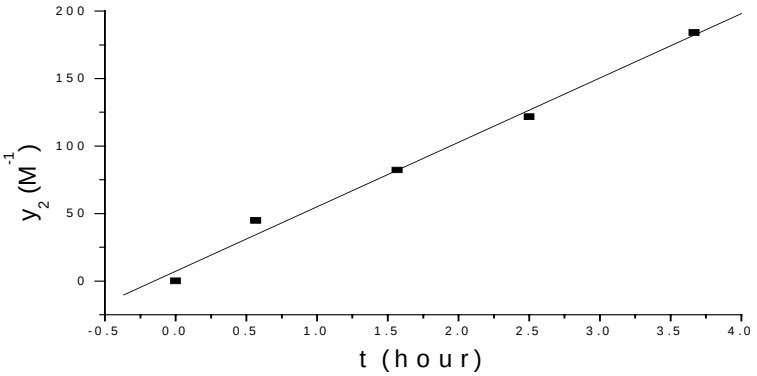
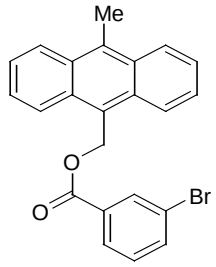


$k_2 = 48\ M^{-1}h^{-1}$.



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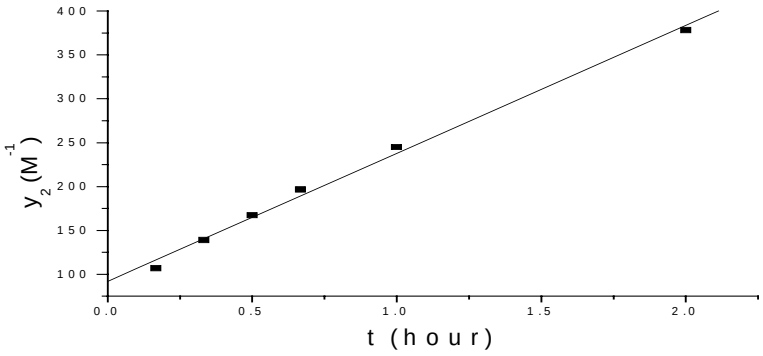
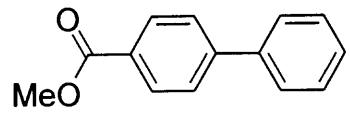


$k_2 = 146\ M^{-1}h^{-1}$.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MeO

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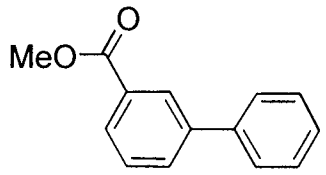
8
7
6
5
4
3
2
1
ppm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O

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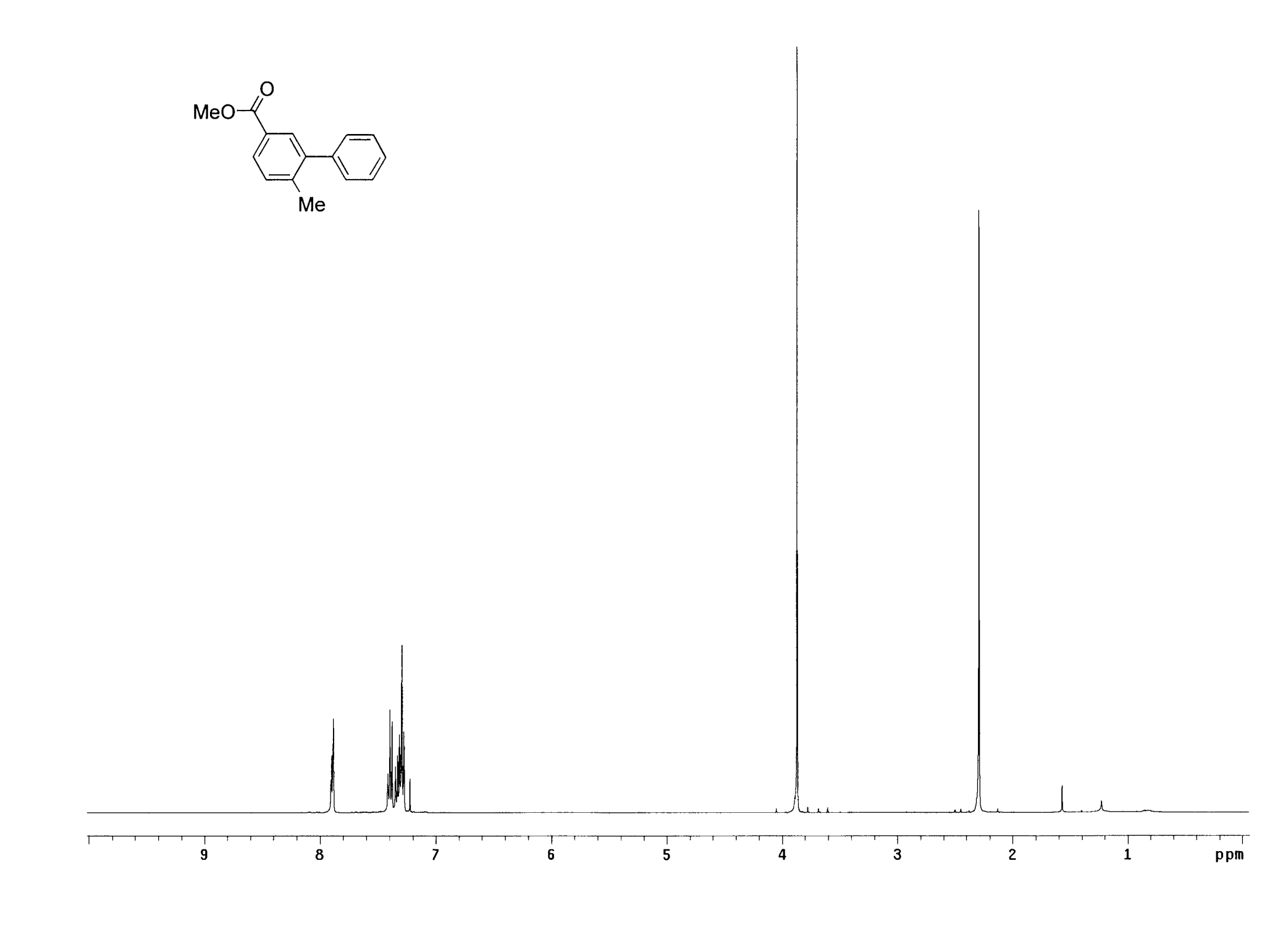
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O
Me
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8
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6
5
4
3
2
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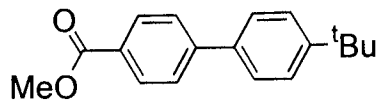
ppm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MeO
tBu

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7
6
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4
3
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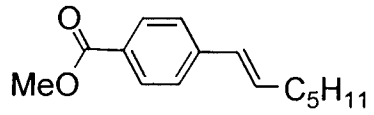
1
ppm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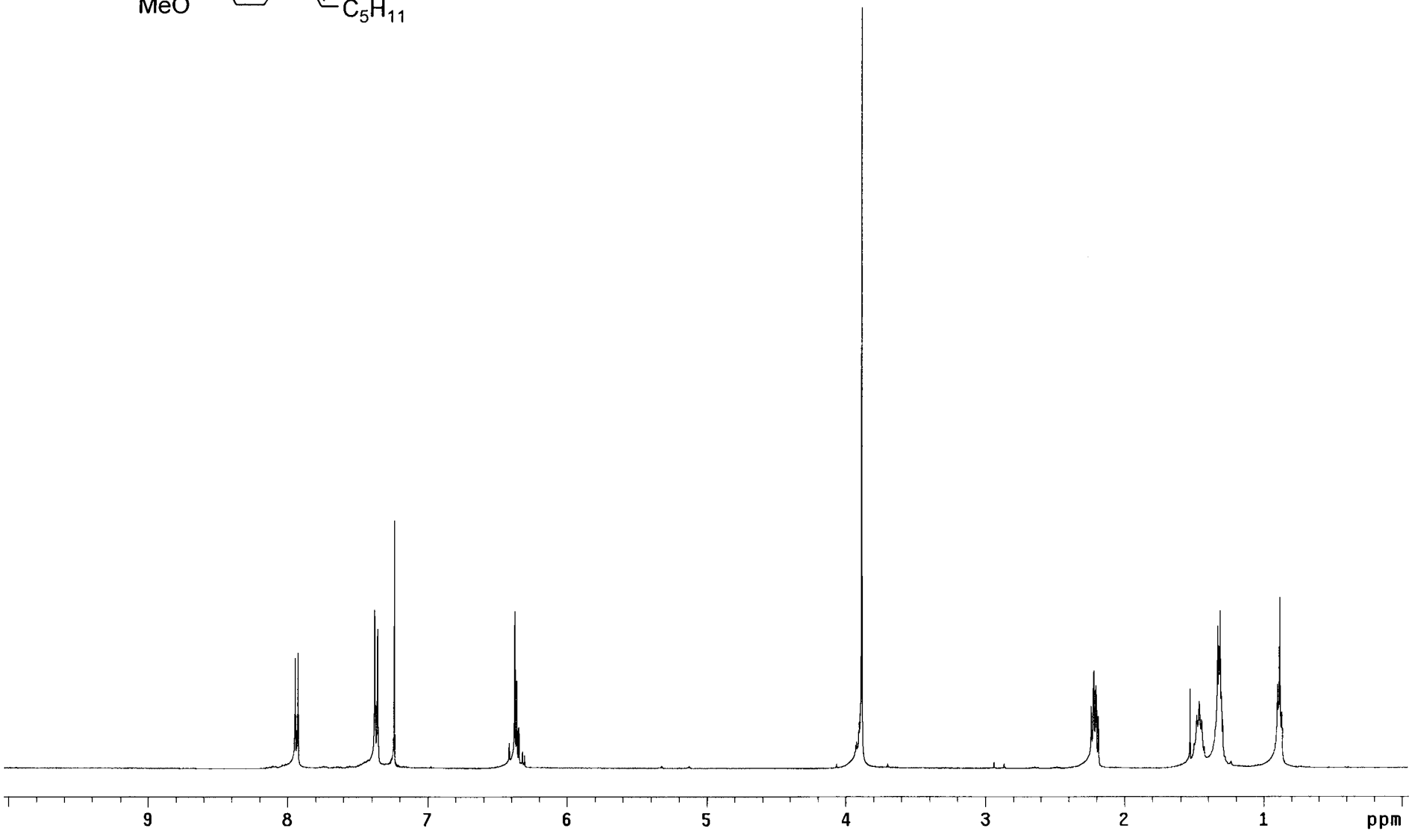
C5H11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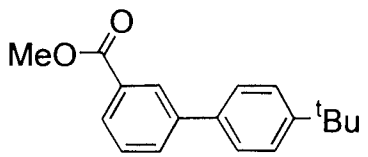
MeO
O
tBu

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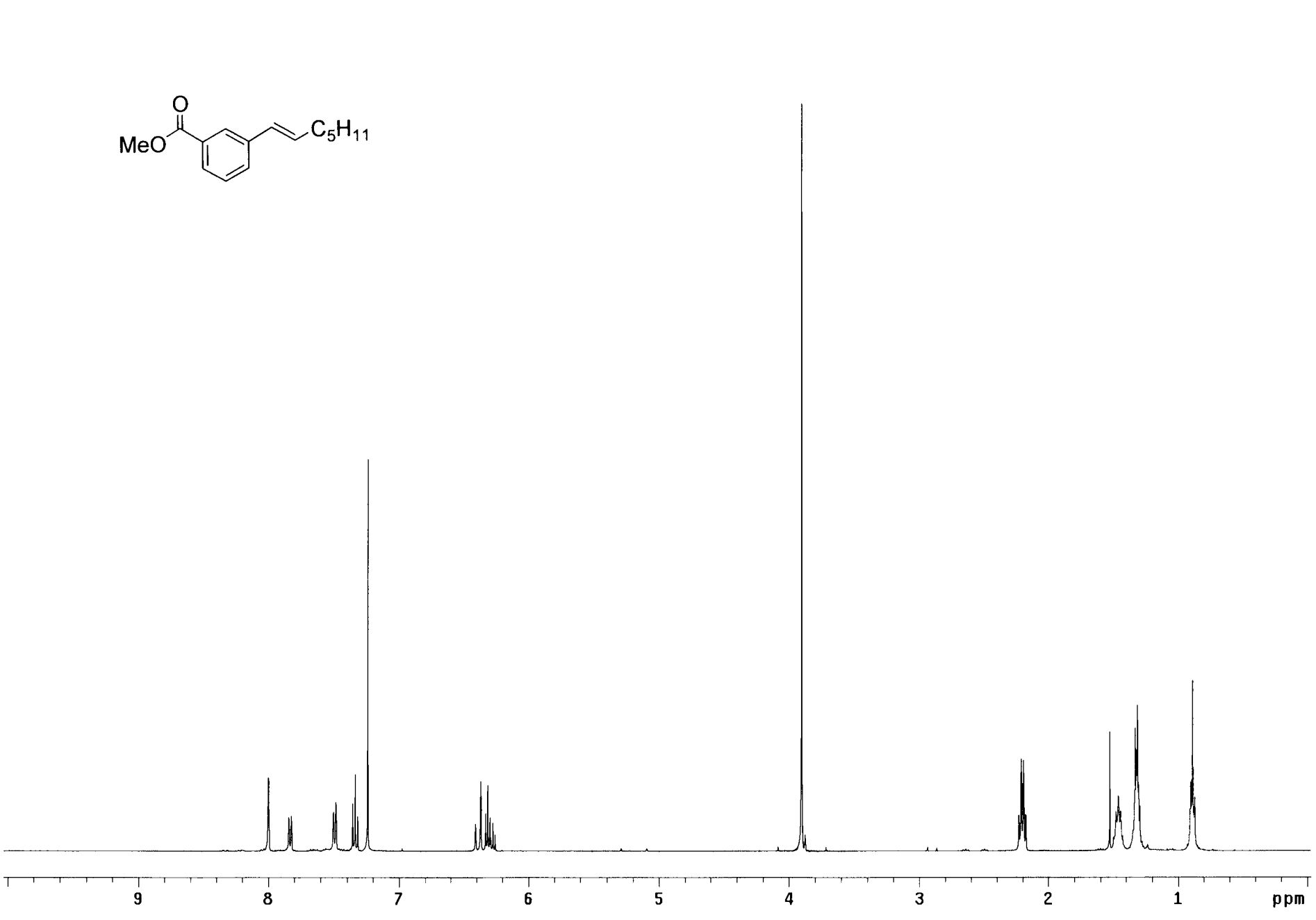
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MeO
C_5H_{11}
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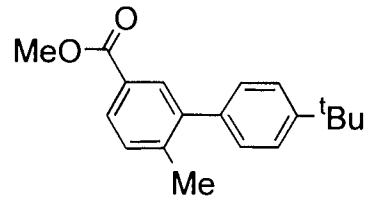
ppm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tBu
Me

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2
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ppm