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ELECTRONIC SUPPLEMENTARY INFORMATION

For

A chiral organocatalytic polymer-based monolithic reactor

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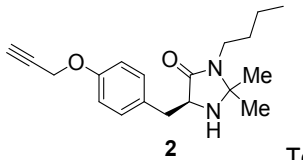
General methods: Dry solvents were purchased and stored under nitrogen over molecular sieves (bottles with crown caps). Tris[(1-benzyl-1H-1,2,3-triazol-4-yl)methyl]amine[1] and 4-azidomethylstyrene[2] were prepared as described. Divinylbenzene (Aldrich, tech. grade, 80%, mixture of isomers) was filtered through neutral alumina before use and stored at -20°C under N_2. The other chemicals were used as received. Reactions were monitored by analytical thin-layer chromatography (TLC) using silica gel 60 F254 pre-coated glass plates (0.25 mm thickness) and visualized using UV light. Flash chromatography was carried out on silica gel (230-400 mesh). ^{1}H NMR and ^{13}C NMR spectra were recorded at 25°C with a Varian Inova 600 spectrometer working at the frequency of 599.68 MHz and 150.81 MHz, respectively or with a Bruker Fourier 300 or AMX 300 at the frequency of 300 and 75 MHz respectively. The relaxation delay d_1 between pulses in ^{1}H NMR and ^{13}C NMR experiments was set to 10 s and 1 s, respectively. Chemical shifts values (δ) are reported in part per million (ppm). Peak multiplicity is summarized as br. (broad), s (singlet), d (doublet), t (triplet), m (multiplet). Electrospray ionization mass spectra [MS(ESI)] were recorded as methanol solutions by using a AB Sciex Instruments triple quadrupole LC/MS/MS API 365 mass spectrometer. The BET surface area was determined by nitrogen adsorption using a ThermoQuest Surface Area Analizer Qsurf S1. Enantiomeric excess determinations were performed under the conditions reported below with Agilent 1200 series HPLC.

Preparation of imidazolidinone (2) (Known compound): [3]



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To a stirred portion of butylamine (10 mL, 101.2 mmol) kept under nitrogen, (*S*)-tyrosine methylester (6 g, 30.8 mmol) was added and the mixture was stirred for 24 h at room temperature. After addiction of CH_2Cl_2, the reaction was then evaporated under vacuum giving 7.2g of a pale yellow solid that was used for the next reaction without any further purification step. The compound was dissolved in CH_3OH (60 mL) and acetone (60 mL), and PTSA (60 mg) was added. The mixture was refluxed for 24 h and concentrated under vacuum to afford 8.4g of the crude imidazolidinone that was used for the next reaction without any further purification step. It had m.p. 99-101°C. $[\alpha]_D^{23}$ –78.2 (c = 0.72 in CH_2Cl_2); ^{1}H NMR ($CDCl_3/D_2O$): δ 7.04 (B part of AB system, ^3J (H,H) = 8.5 Hz, 2H; aromatic protons), 6.74 (A part of AB system, 3*J* (H,H) = 8.5 Hz, 2H; aromatic protons), 3.73 (t, 3*J* (H,H) = 5.8 Hz, 1H; CHN), 3.29 (ddd, 2*J* (H,H) = 12.0 Hz, 3*J* (H,H) = 6.7 and 3.2 Hz, 1H; one H of NCH_2), 3.04 (ddd, 2*J* (H,H) = 12.0 Hz, 3*J* (H,H) = 5.8 and 5.4 Hz, 2H; $ArCH_2$), 2.89 (ddd, 2*J* (H,H) = 12.0 Hz, 3*J* (H,H) = 6.2 and 3.1 Hz, 1H; one H of NCH_2), 1.43-1.49 (m, 2H; NCH_2CH_2), 1.21-1.31 (m, 2H; CH_3CH_2), 1.27 (s, 3H; CMe), 1.17 (s, 3H; CMe), 0.90 (t, 3*J* (H,H) = 7.3 Hz, 3H; CH_2CH_3); ^{13}C NMR: δ 174.2, 155.8, 130.7, 127.2, 115.7, 76.4, 58.9, 40.4, 35.5, 31.3, 27.8, 26.3, 20.3, 13.7; IR: 3270, 1675, 1620 cm^{-1}; elemental analysis calcd for $C_{16}H_{24}N_2O_2$ (276.4): C 69.53, H 8.75, N 10.14; found: C 69.71, H 8.64, N 10.23.

To a solution of the crude imidazolidinone (5 g, 18 mmol) in dry acetonitrile (40 mL) potassium carbonate (20 g, 145 mmol). was added and the mixture was cooled to 0°C with an external ice water bath; propargyl bromide solution (80% wt. in toluene, 8 mL, 72 mmol) was added dropwise over 15 min. and the resulting mixture was allowed to warm to Room Temperature and stirred under inert

atmosphere for 24 hours. The reaction mixture was then filtered through a celite plug and the solvent was removed under vacuum; the crude product was purified by flash column chromatography (eluent: CH_2Cl_2/MeOH = 98/2) to afford compound **2** as a brownish oil (15.9 mmol, 88%).

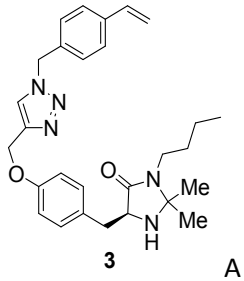
TLC R_f = 0.47 (CH_2Cl_2/MeOH = 98/2)

^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.12 (d, 2H, Ar-**H**); 6.85 (d, 2H, Ar-**H**); 4.59 (d, 2H, -O-C**H$_2$**); 3.65 (t, 1H, -NH-C**H**-); 3.23-3.20 (m, 1H, -N-C**H$_2$**-); 2.97 (dd, 2H, Ar-C**H$_2$**-); 2.96-2.84 (m, 1H, -N-C**H$_2$**-); 2.46 (t, 1H, -C≡C**H**); 1.41 (m, 2H, -N-CH_2-C**H$_2$**-); 1.29-1.24 (m, 2H, C**H$_2$**-CH_3); 1.20 (s, 3H, -C-C**H$_3$**); 1.09 (s, 3H, -C-C**H$_3$**); 0.86 (t, 3H, -CH_2-C**H$_3$**).

^{13}C-NMR (75 MHz, $CDCl_3$): δ 174.1 (1C, -**C**=O); 156.8 (1C, Ar-**C**-O-); 131.0 (2C, Ar-**C**H); 130.1 (1C, Ar-**C**-); 115.2 (2C, Ar-**C**H); 78.8 (1C, -**C**≡CH); 76.3 (1C, -N-**C**-NH-); 75.7 (1C, -C≡**C**H); 59.1 (1C, -NH-**C**H-C=O); 56.1 (1C, -O-**C**H_2-); 40.5 (1C, -N-**C**H_2-); 36.2 (1C, Ar-**C**H_2-); 31.7 (1C, -N-CH_2-**C**H_2-); 28.3 (1C, -C-**C**H_3); 26.8 (1C, -C-**C**H_3); 20.6 (1C, -**C**H_2-CH_3); 14.0 (1C, -CH_2-**C**H_3).

IR: 3302, 2961, 1685, 1610 cm^{-1}

Preparation of MacMillan monomer (3):



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A 25mL flame-dried Schlenk tube was charged under nitrogen with CuCl (5.5 mg, 0.056 mmol, 0.05 equiv.), tris[(1-benzyl-1H-1,2,3-triazol-4-yl)methyl]amine (29.5 mg, 0.0556 mmol, 0.05 equiv.) and freshly distilled CH_2Cl_2 (4 mL). After stirring for 10 min, the MacMillan-alkyne derivative **2** (350 mg, 1.13 mmol) in CH_2Cl_2 (5 mL) was added, followed by 4-azidomethylstyrene (354 mg, 2.23 mmol, 2 equiv.) in CH_2Cl_2 (4 mL). The pale-brownish suspension was degassed by three vacuum/N_2 cycles and the Schlenk tube was sealed under nitrogen and kept stirring at r.t. After complete conversion of **2** (20 h, TLC monitoring), the reaction mixture was concentrated to approx. 10 mL and directly loaded onto a silica-gel column. By eluting with EtOAc, the pure monomer **3** was obtained as pale-yellow viscous oil (458 mg, 0.967 mmol, 86% yield).

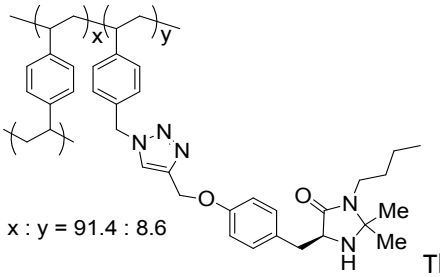
TLC, R_f= 0.20 (SiO_2, EtOAc)

^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.50 (s, 1H), 7.40 (d, *J* = 8.0 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.12 (d, *J* = 8.4 Hz, 2H), 6.87 (d, *J* = 8.6 Hz, 2H), 6.69 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.73-5.58 (m, 1H), 5.50 (s, 2H), 5.26-5.30 (m, 1H), 5.13 (s, 2H), 3.70 (t, *J* = 5.3 Hz, 1H), 3.28 (ddd, *J* = 14.3, 9.8, 6.0 Hz, 1H), 3.04 (dd, *J* = 14.3, 5.8 Hz, 1H), 2.99 (dd, *J* = 14.3, 4.8 Hz, 1H), 2.88 (ddd, *J* = 14.5, 9.8, 5.6 Hz, 1H), 1.62 (br. s, NH+H_2O), 1.54 – 1.38 (m, 2H), 1.30-1.25 (m, 2H), 1.24 (s, 3H), 1.13 (s, 3H), 0.90 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 173.8, 157.1, 144.5, 138.1, 135.9, 133.8, 130.7, 129.4, 128.3, 126.8, 122.6, 114.9, 114.7, 76.0, 62.0, 58.8, 53.9, 40.2, 35. 9, 31.4, 28.0, 26.5, 20.3, 13.7.

HRMS Mass (ESI+) m/z calc. for $C_{28}H_{35}N_5O_2H^+$: 474.2871, found: 474.2874 [M + H].

Preparation of MacMillan Monolith (Supp3):



x : y = 91.4 : 8.6

The feed solution for the monolith synthesis was prepared by mixing monomers, porogenic solvents, and the radical initiator in the following order: MacMillan-monomer **3** (0.289 g, 0.610 mmol, 25 wt% of monomers), toluene (0.400 g), 1-dodecanol (1.150 g), divinylbenzene (0.900 g, 75 wt% of monomers), and AIBN (12 mg, 1 wt %). The mixture was deoxygenated by slowly bubbling nitrogen for 10 minutes under stirring. The resulting solution was poured in a stainless steel HPLC column sealed at the bottom end (0.46 cm i.d. $\times$ 15 cm), paying attention to fill completely the available volume. The top end was sealed under nitrogen and the column was weighed in order to determine the exact amount of the feed mixture (2.16 g, 0.484 mmol of the chiral monomer **3**). The monolith formation was carried out by placing the column vertically into a preheated oil bath (70°C) and keeping for 18 h at the same temperature. After cooling to r.t., the seals were replaced by metal frits and the soluble components were removed by flushing the column with 150 mL dry THF (syringe pump, flow-rate ϕ = 300 μL min^{-1}). Evaporation of the volatiles from the eluate afforded 0.90 g of a clear oil consisting of essentially pure 1-dodecanol (NMR and TLC). UV check indicated that the concentration of the chiral derivative into the final washings was lower than $7 \cdot 10^{-5}$ M.

For comparison purposes, a portion of the feed mixture was subjected to copolymerization in a sealed vial placed in the same oil bath used for the preparation of the column monolith. The recovered colourless opaque mass was crushed and washed thoroughly with dry THF. After removal of the volatiles under reduced pressure (10^{-2} mmHg), the resulting white powder was subjected to IR characterization, determination of BET surface area by N_2 adsorption, and employed in reference catalytic runs in batch.

IR(KBr pellet) ν (cm^{-1}): 3020, 2927, 2869, 1695, 1605, 1512, 1448, 1245, 905, 830, 798, 710. BET Surface area: 485 m^2 g^{-1}.

Diels-Alder cycloaddition

Flow reaction

The monolithic reactor containing 0.93 g of imidazolidinone organocatalyst **Supp-3** (loading 0,51 mmol/g, 0,475 mmol of catalyst) was flow-treated overnight with trifluoroacetic acid (TFA) solution (0.2 M in a 95/5 CH_3CN/H_2O mixture) or a 48% solution in water of HBF_4 (0.2 M in a 95/5 CH_3CN/H_2O mixture). After this treatment, the column was washed with 5 ml of 95/5 CH_3CN/H_2O mixture to remove excess acid. The Diels-Alder cycloaddition reaction was then carried out at 25°C, by pumping a solution of the reagents (*trans*-cinnamaldehyde 0.195 M, 1eq and cyclopentadiene 1.365 M, 7eq in a 95/5 CH_3CN/H_2O mixture) through the reactor.

Batch reaction

Powder **Supp-3** (0,11mmol, 200mg, 0,3eq), trifluoroacetic acid (0,11mmol, 8 µl) and the reagents *trans*-cinnamaldehyde (0,37mmol, 46µl, 1eq) and cyclopentadiene (2,59mmol, 214µl, 7eq) were mixed at room temperature in 2ml of a 95/5 CH_3CN/H_2O mixture. After 48 hours the polymer-supported catalyst was isolated by centrifugation and filtration and the organic phase was worked-up to obtain the products. The same amount of reagents was added and the same procedure was repeated after a reaction time of 24hours.

The same procedure was followed using a 48% solution in water of HBF_4 (0,11mMol, 7µl).

The *endo*/*exo* ratio was established on the crude product by using the CHO signals at δ= 9.60 (*endo*) and 9.93 (*exo*) ppm. For ee determination the aldehyde was converted into the corresponding alcohol by reduction with an excess $NaBH_4$ in CH_3OH, 24°C, 1 h.

(1*S*, 2*S*, 3*S*, 4*R*)-3-phenylbicyclo[2.2.1]hept-5-ene-2-carbaldehyde and (1*R*, 2*S*, 3*S*, 4*S*)-3-phenylbicyclo[2.2.1]hept-5-ene-2-carbaldehyde: This product is known[4] and was purified by flash column chromatography on silica gel with a 98:2 hexane/ethyl acetate mixture as eluant affording a mixture of *endo* and *exo* Diels-Alder adducts.

Data for *endo*:

TLC, R_f= 0.42 (hex/EtOAc 9:1 stained blue with phosphomolibdic acid)

^{1}H-NMR (300 MHz, $CDCl_3$): δ 9.61 (d, J=2.2 Hz, 1H, CHO), 7.14-7.34 (m, 5H, Ph), 6.43 (dd, J=3.3, 5.6 Hz, 1H, CH=CH), 6.18 (dd, J=2.8, 5.7 Hz, 1H, CH=CH), 3.34 (brs, 1H, CHCH=CHCH), 3.14 (brs, 1H, CHCH=CHCH), 3.10 (d, J=4.8, 1H, CHPh), 2.99 (dd, J=2.7, 5.4, 1H, CHCHO), 1.82 (d, J=8.7, 1H, CHH), 1.61-1.64 (m, 1H, CHH).

Data for *exo*:

TLC, R_f= 0.42 (hex/EtOAc 9:1 stained blue with phosphomolibdic acid)

^{1}H-NMR (300 MHz, $CDCl_3$): δ 9.93 (d, J=2.0 Hz, 1H, CHO), 7.14-7.34 (m, 5H, Ph), 6.34 (dd, J=3.4, 5.5 Hz, 1H, CH=CH), 6.08 (dd, J=3.0, 5.5 Hz, 1H, CH=CH), 3.73 (t, J=3.8, 1H, CHCH=CHCH), 3.23 (m, 2H, CHCH=CHCH, CHPh), 2.60 (dd, J=1.5, 3.4, 1H, CHCHO), 1.61-1.64 (m, 2H, CHH, CHH).

((1*S*, 2*S*, 3*S*, 4*R*) 3-phenylbicyclo[2.2.1]hept-5-en-2-yl)methanol and ((1*R*, 2*S*, 3*S*, 4*S*)-3-phenylbicyclo[2.2.1]hept-5-en-2-yl)methanol:

This product is known and was injected into the HPLC without further purification.

Data of a mixture *endo*:*exo*:

^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.18-7.31 (m, 10H), 6.34-6.41 (m, 2H), 6.15-6.20 (m, 1H), 5.93-5.98 (m, 1H), 3.86-3.94 (m, 1H), 3.59-3.70 (m, 3H), 3.39 (t, J=12.8 Hz, 1H), 3.04 (brs, 2H), 2.83-2.88 (m, 3H), 2.30-2.42 (m, 2H), 2.15-2.18 (m, 2H), 1.76-1.82 (d, J=8.9, 1H), 1.65-1.68 (d, J=8.7, 1H).

The enantiomeric excess was determined by chiral HPLC with Daicel Chiralcel OJ-H column [eluant: 7:3 hex/IPA; 0.8 mL/min flow rate, detection: 225 nm; t_R 11.6 min (*endo*-minor), t_R 23.9 in (*endo*- major) t_R 30.3 min (*exo*-minor), t_R 40.3 min (*exo*-major).

1,3 Dipolar cycloaddition:

The reaction was carried out at different temperatures (see text) by pumping a solution of the reagents (*N*-benzyl-*C*-phenyl nitrone 0.195 M and crotonic aldehyde 0.975 M in wet CH_3NO_2) through the reactor.

The *endo*/*exo* ratio was established on the crude product by using the CHO signals at δ= 9.81 (*endo*) and 9.33 (*exo*) ppm. For ee determination the purified aldehyde was converted into the corresponding alcohol by reduction with an excess $NaBH_4$ in CH_3OH, 24°C, 1 h.

4-Formyl-5-methyl-3-phenyl-2-(phenylmethyl)isoxazolidine (11): This product is known[5] and was purified by flash column chromatography on silica gel with a 85:15 hexane/ethyl acetate mixture as eluant affording a mixture of *endo* and *exo* cycloadducts.

Data for *endo*:

TLC, R_f= 0.35 (hex/EtOAc 85:15 stained blue with phosphomolibdic acid)

^{1}H-NMR (300 MHz, $CDCl_3$): δ 9.81 (d, J=2.4 Hz, 1H, C**H**O), 7.28-7.49 (m, 10H, Ph), 4.58 (dq, J=6.1, 12.2 Hz, 1H, C**H**CH_3), 4.20 (d, J=7.8 Hz, 1H, C**H**Ph), 4.02 (d, J=14.4 Hz 1H, C**H$_2$**Ph), 3.83(d, J=14.3 Hz 1H, C**H$_2$**Ph), 3.15 (m, 1H, C**H**CHO), 1.55 (d, J=6.2 Hz, 3H, -C**H$_3$**).

((3S,4S,5R)-2-benzyl-5-methyl-3-phenylisoxazolidin-4-yl)methanol:

This product is known and was injected into the HPLC without further purification

Data for *endo*:

^{1}H-NMR (300 MHz, $CDCl_3$): 7.23-7.48 (m, 10H, Ph), 4.21-4.25 (dq, 1H, C**H**CH_3), 3.98 (d, 1H, C**H$_2$**Ph), 3.78(d, 1H, C**H$_2$**Ph), 3.67 (m, 2H, C**H$_2$**OH), 3.64 (d, 1H, C**H**Ph), 2.36-2.41 (m, 1H, C**H**CH_2OH), 1.45 (d, 3H, -C**H$_3$**).

The enantiomeric excess was determined by chiral HPLC with Daicel Chiralcel AD column; eluant: 95:5 hex/EtOH; 0.8 mL/min flow rate, detection: 230 nm; t_R 11.6 min (*endo*-minor), t_R 14.7 min (*endo*- major).

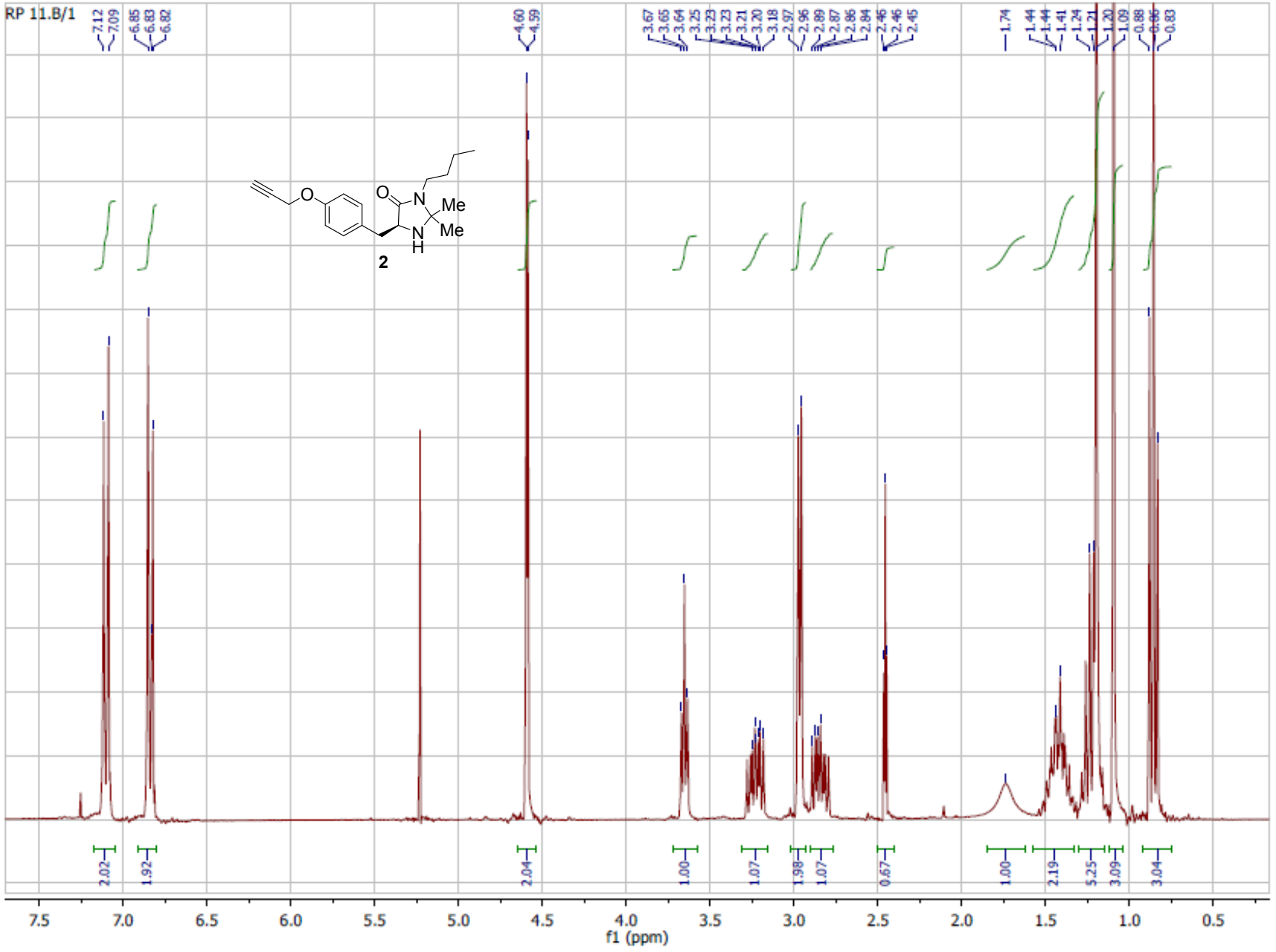
Friedel-Crafts alkylation:

The reaction was carried out at 25°C by pumping a solution of the reagents (*trans*-cinnamaldehyde 0.195 M and *N*-methyl pyrrole 0.975 M in wet THF) through the reactor.

(*S*)-3-Phenyl-3-(1-methyl-1H-pyrrol-2-yl)-propanal (13) : This product is known; [6] it was converted into the corresponding alcohol by reduction with an excess $NaBH_4$ in CH_3OH, 24°C, 1 h and purified by flash column chromatography on silica gel with a 70:30 hexane/ethyl acetate mixture as eluent.

^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.28-7.19 (m, 4H), 7.13 (m, 1H), 6.42 (d, 2H), 6.36 (t, 1H), 4.18 (t, 1H), 3.62-3.49 (m, 2H), 2.90 (s, 3H) 2.39-2.32 (m, 1H), 2.11-2.03 (m, 1H).

The enantiomeric excess was determined by chiral HPLC with Daicel Chiralcel OD-H column; eluant: 90:10 hex/IPA; 0.8 mL/min flow rate, detection: 210 nm; t_R 14.6 min (minor), t_R 46.1 min (major).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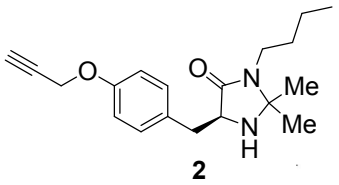

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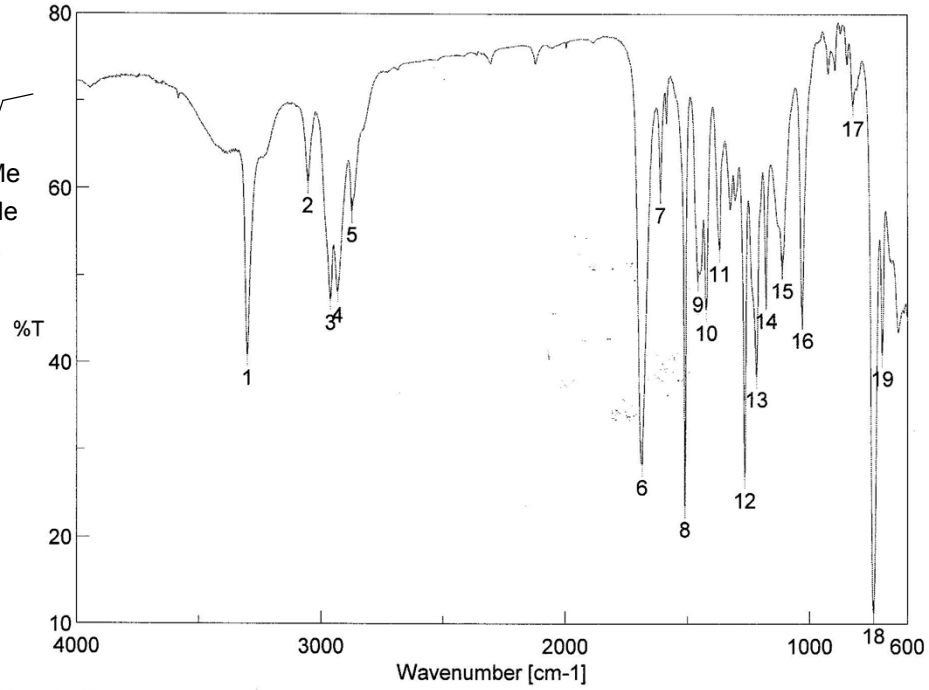

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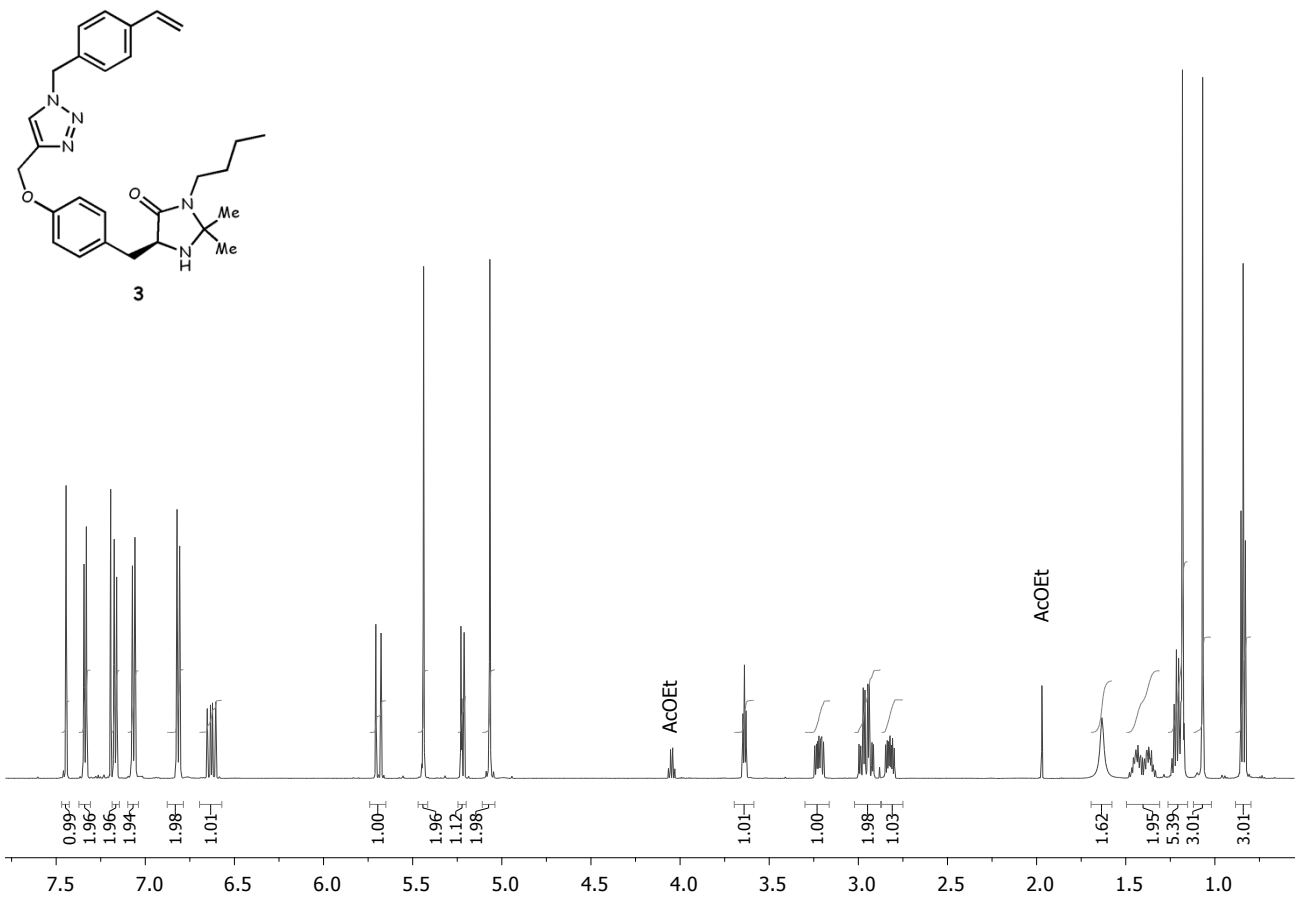
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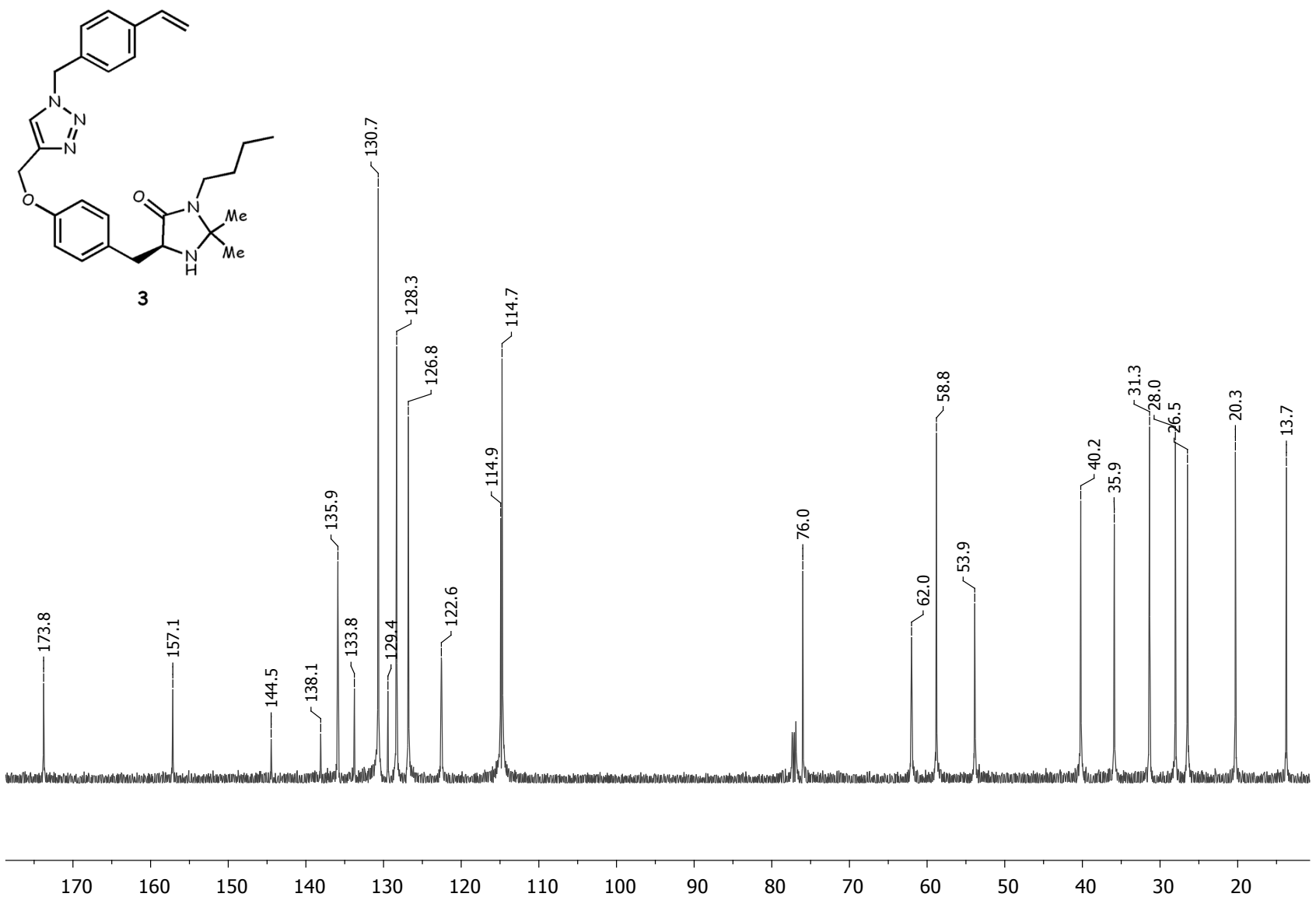
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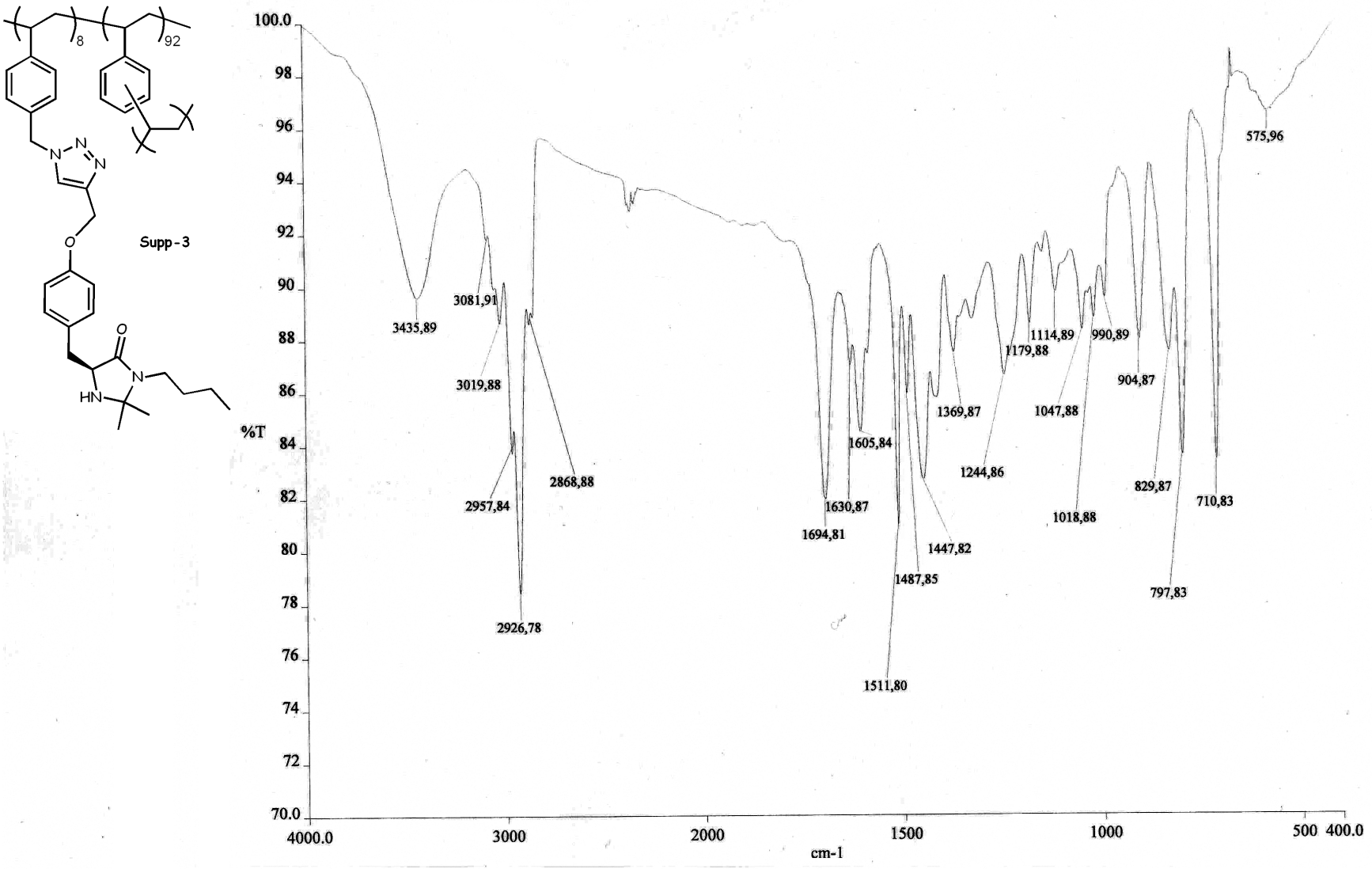
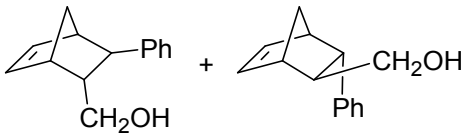
40.2
35.9
31.3
28.0
26.5
20.3
13.7
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20

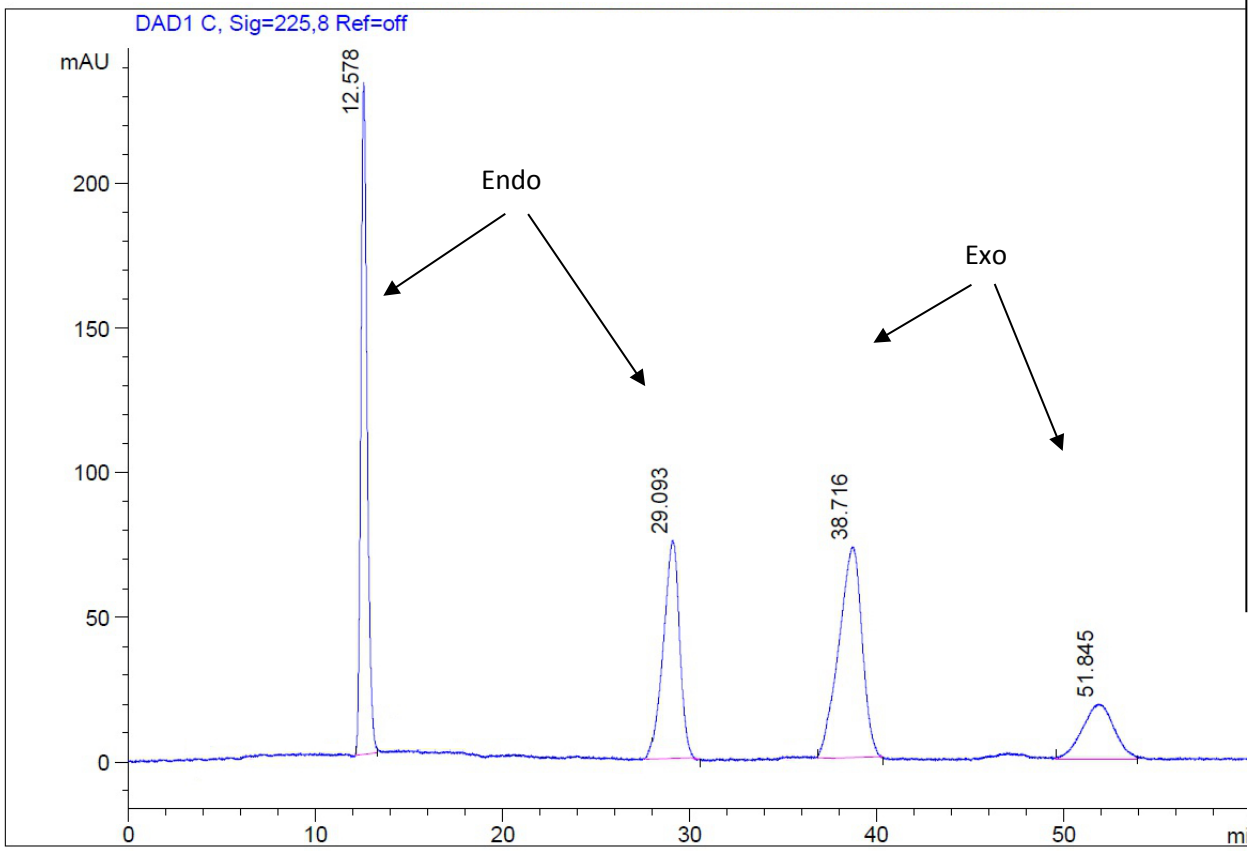

Supp-3
100.0
98
96
94
92
90
88
86
84
82
80
78
76
74
72
70.0
%T
4000.0
3000
2000
1500
1000
500 400.0
cm-1
3435,89
3081,91
3019,88
2957,84
2926,78
2868,88
1694,81
1630,87
1605,84
1511,80
1487,85
1447,82
1369,87
1244,86
1179,88
1114,89
1047,88
1018,88
990,89
904,87
829,87
797,83
710,83
575,96

HPLC CHROMATOGRAMS:

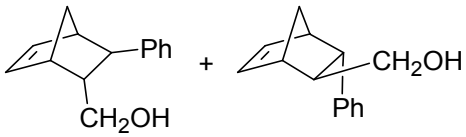
Non stereoselective Diels-Alder reaction

The product is known.[4] The stereochemistry was assigned for comparison with MacMillan[4] and Hayashi assignements.[7]



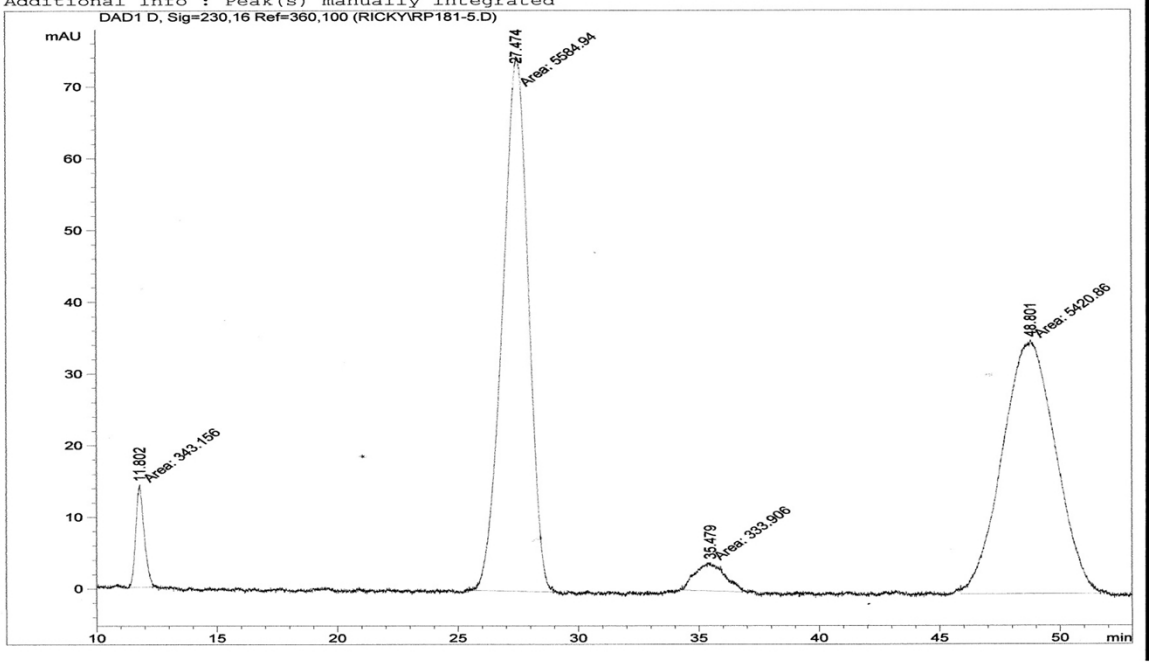


Stereoselective Diels-Alder reaction



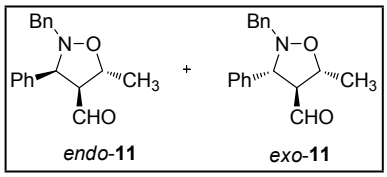
Sample Info : RP181-5
CHIRALCEL OJ-H HEX/IPA 7/3 0.8 mL/ min 48 bar

Additional Info : Peak(s) manually integrated



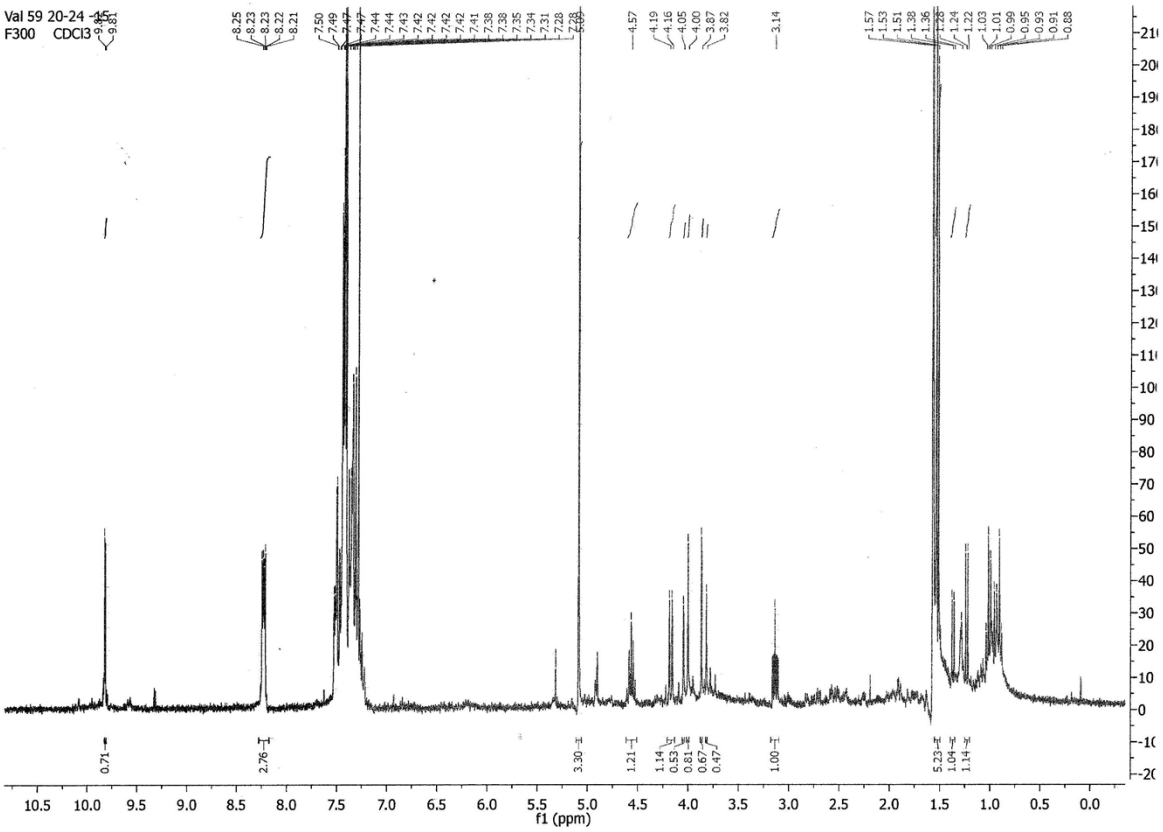
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.802	MM	0.4000	343.15558	14.29693	2.9373
2	27.474	MM	1.2486	5584.93750	74.55125	47.8045
3	35.479	MM	1.3874	333.90649	4.01128	2.8581
4	48.801	MM	2.5555	5420.85986	35.35433	46.4001
Totals :				1.16829e4	128.21379	

1,3-Dipolar Cycloaddition

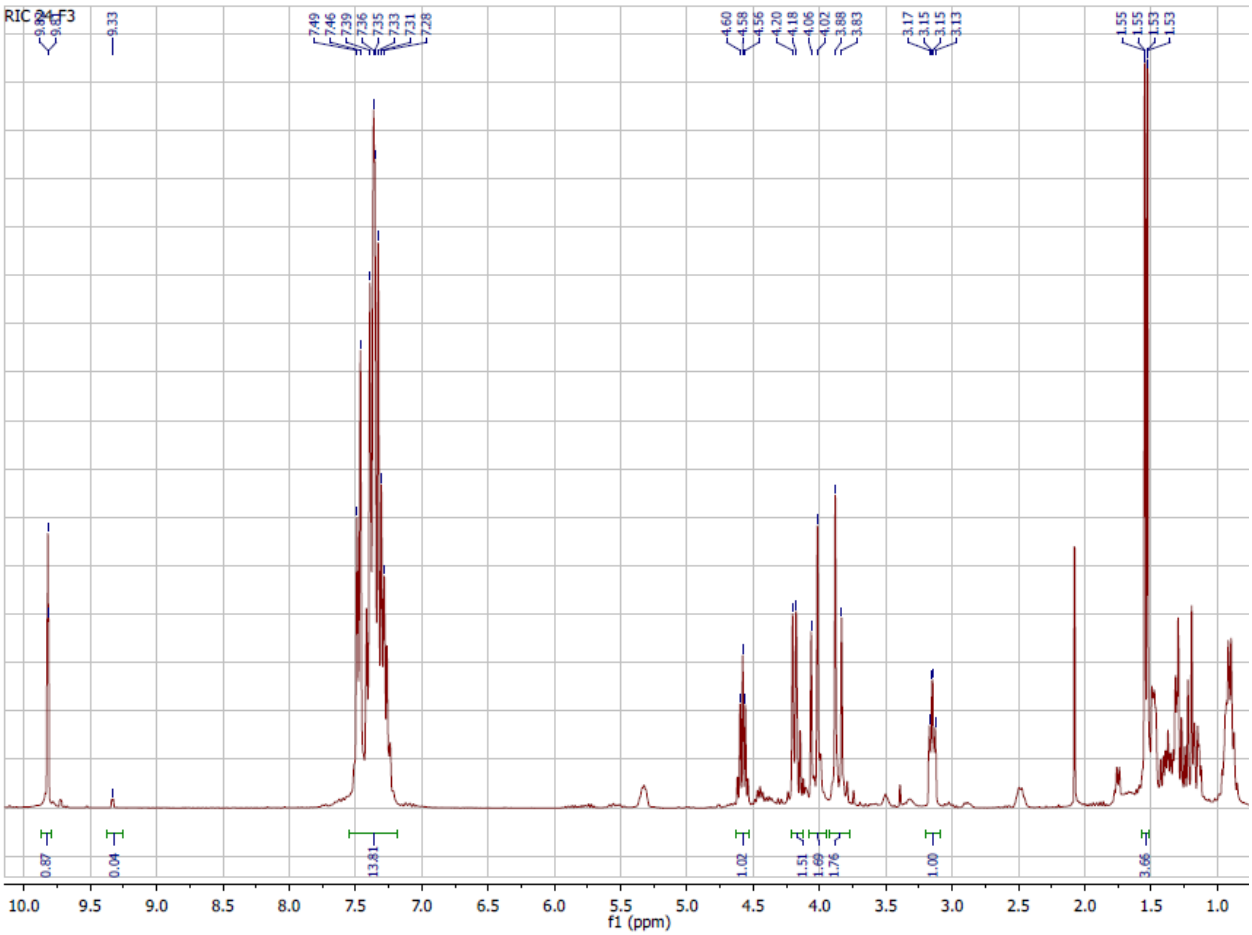


endo-**11** *exo*-**11**

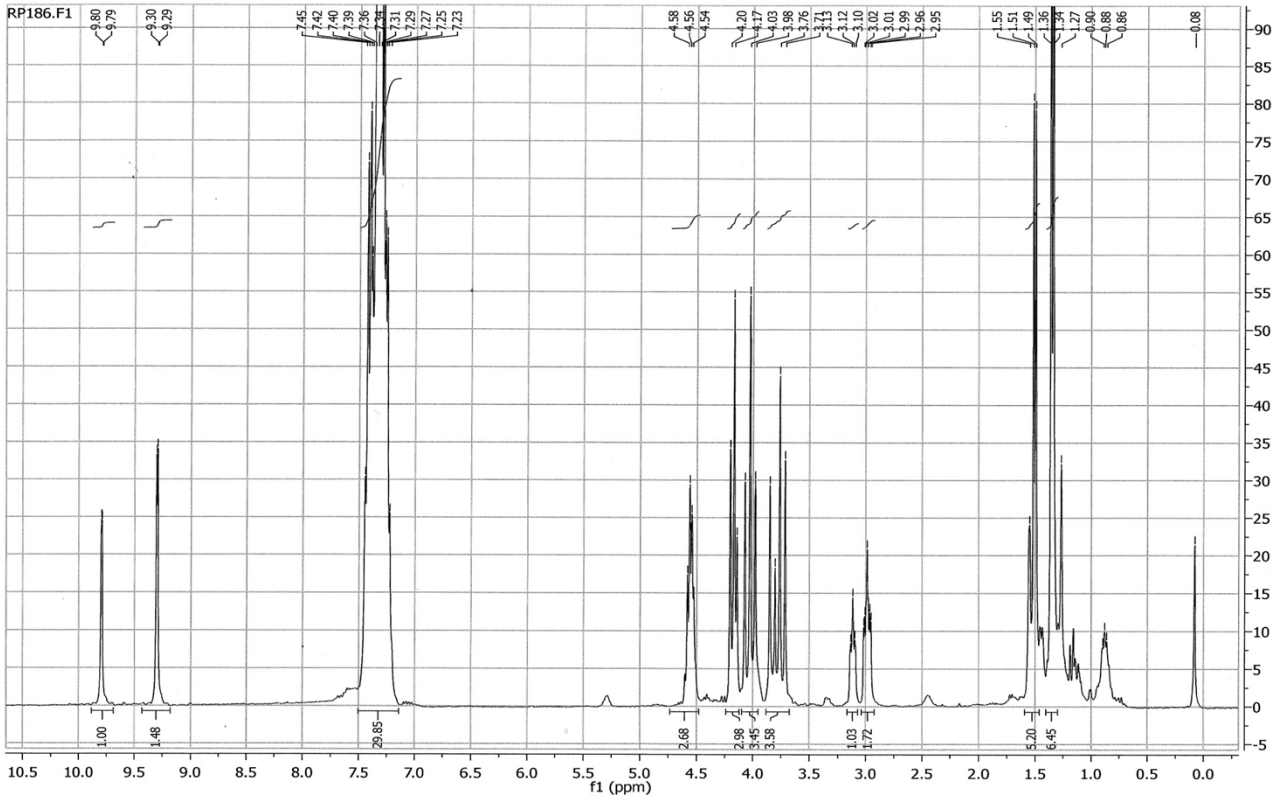
^{1}H-NMR spectrum of an aliquot of crude reaction mixture under continuous flow conditions (4h running time, about 0.05 mmol, 13 mg)



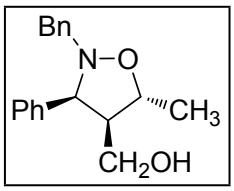
^{1}H-NMR spectrum of the isolated products **11** at the end of the reaction under continuous flow conditions (30 h running time, -15 °C; entry 6 Table 7).

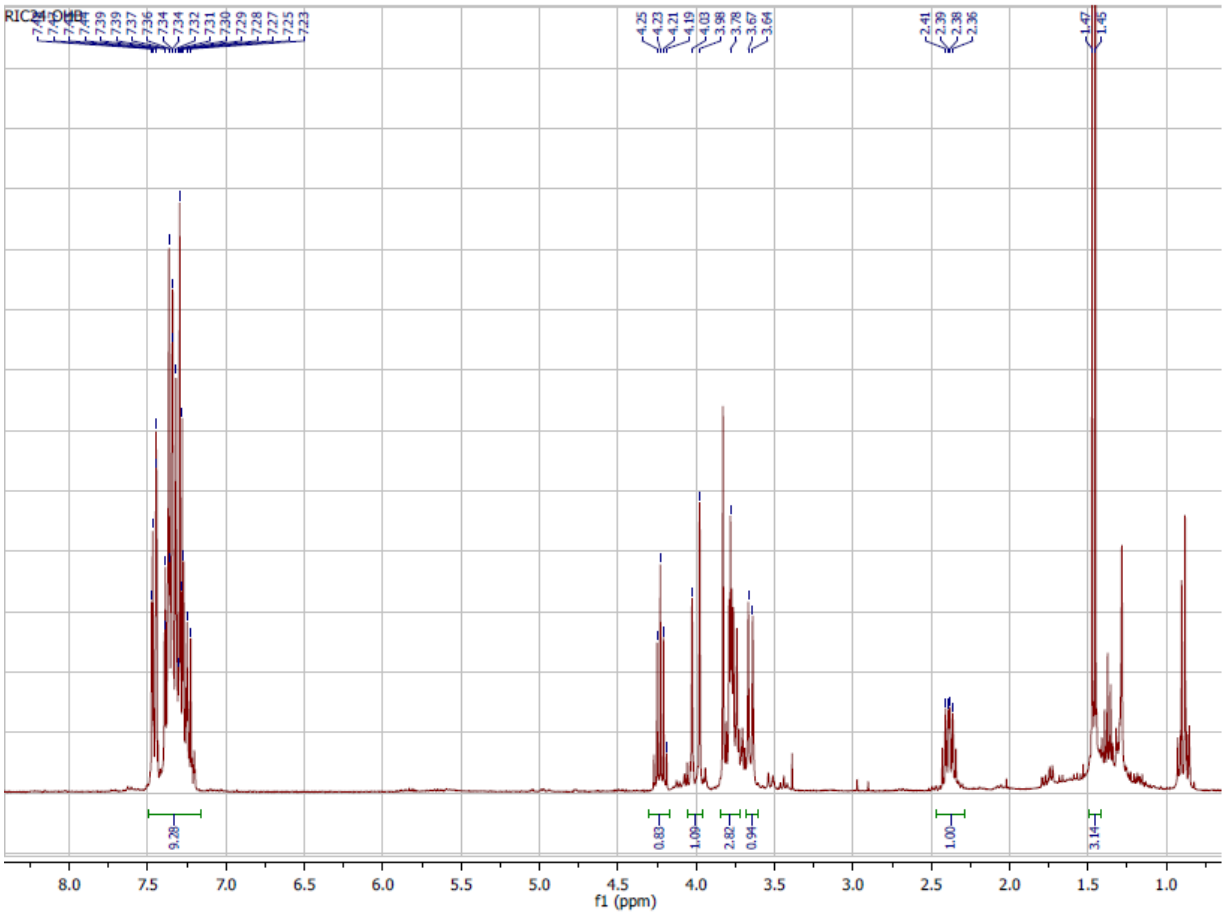


As a comparison, see the ^{1}H-NMR spectrum of the isolated products **11** obtained using commercially available MacMillan catalyst.



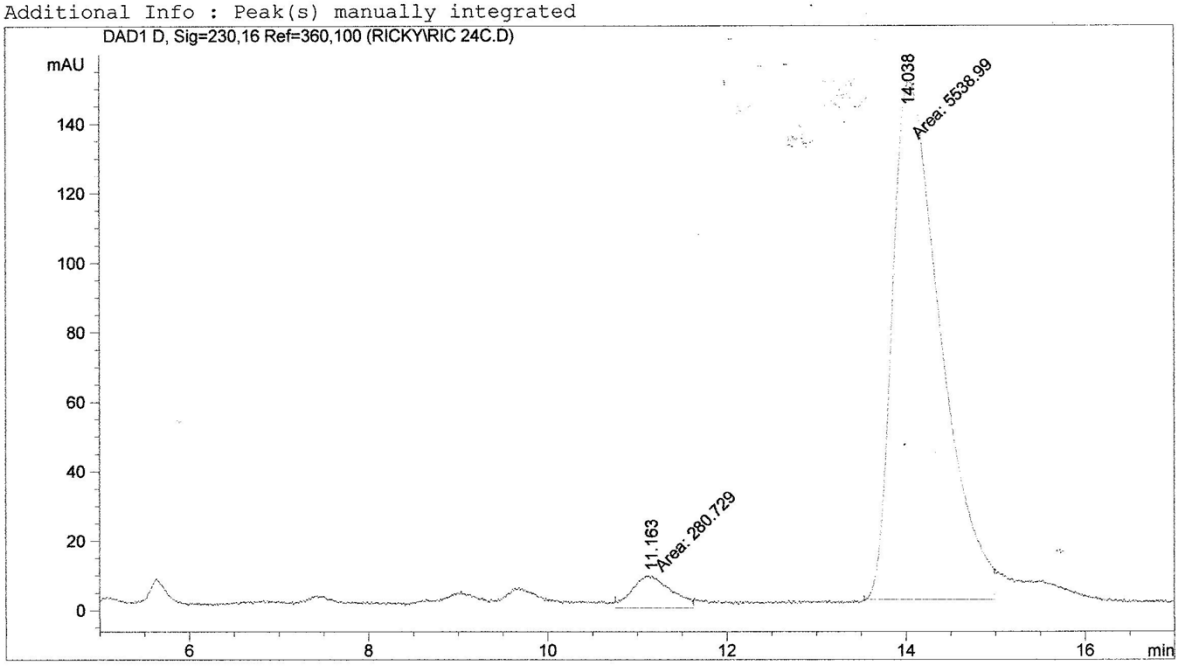
^{1}H-NMR spectrum of *endo*-**11** after reduction to the corresponding alcohol





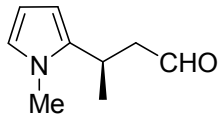
CHIRALPACK AD, HEX/EtOH 95:5 0.8mL/ min 20 BAR

Additional Info : Peak(s) manually integrated



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.163	MM	0.4997	280.72882	9.36414	4.8238
2	14.038	MM	0.6184	5538.98633	149.27679	95.1762

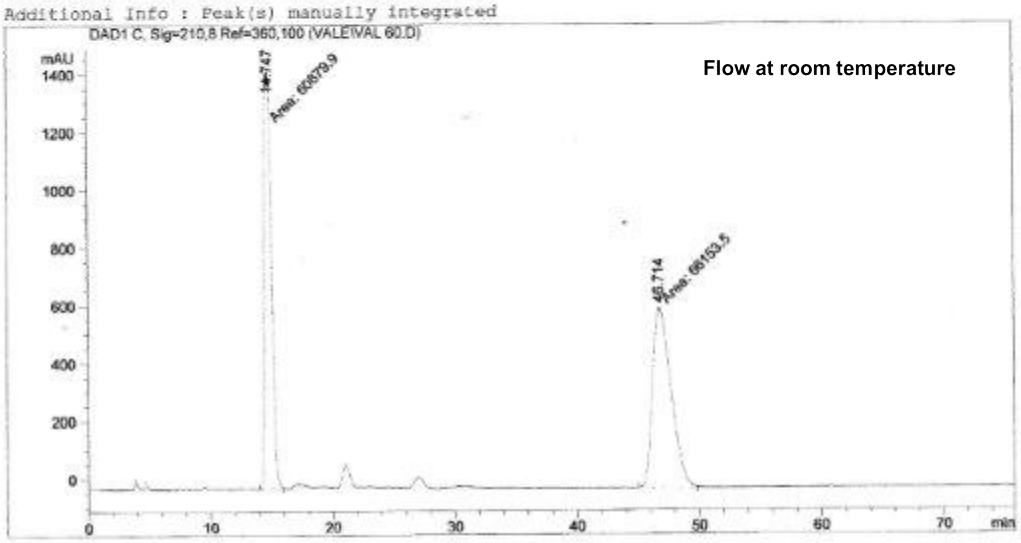
Friedel-Crafts alkylation



13

CHILALPAK ODH HEX:IPA 9:1 0.8 mL/min 35 bar

Additional Info : Peak(s) manually integrated



Flow at room temperature

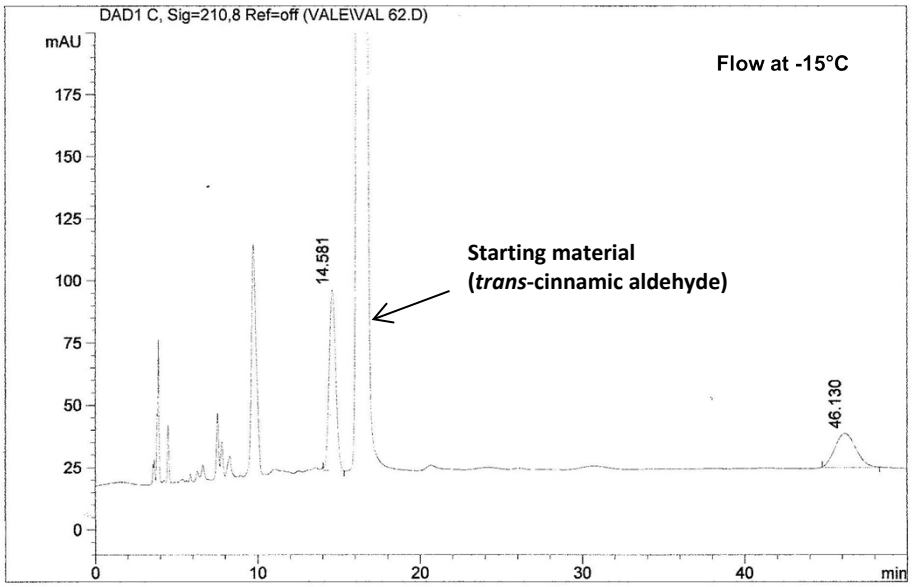
Area Percent Report

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	14.747	MM	0.7020	6.08799e4	1445.37219	47.9243
2	46.714	MM	1.7631	6.61535e4	625.36102	52.0757

CHIRALCEL OD-H; Hex:iPrOH 9:1; 0.8 mL/min; 38 bar

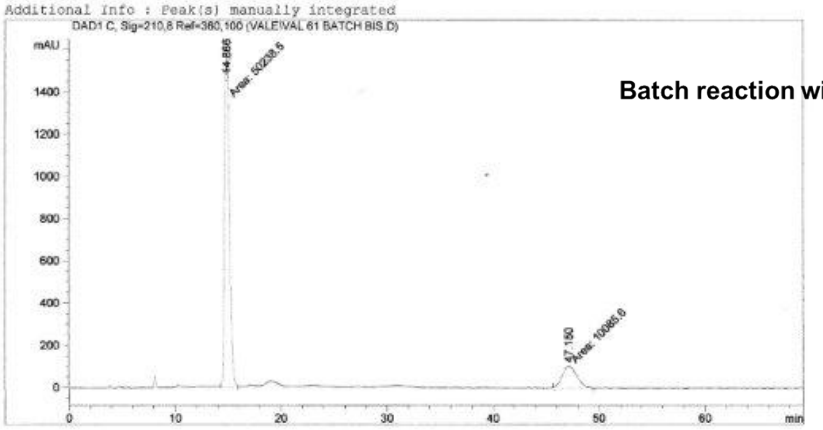


Signal 1: DAD1 C, Sig=210,8 Ref=off

Peak #	RT [min]	Type	Width [min]	Area	Area %	Name
1	14.581	BB	0.444	2063.243	62.766	
2	46.130	BB	1.046	1223.981	37.234	

CHILALPAK ODH HEX:IPA 9:1 0.8 mL/min 35 bar

Additional Info : Peak(s) manually integrated



Batch reaction with commercial MacMillan

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	14.866	MM	0.5338	5.02385e4	1569.72705	83.2810
2	47.150	MM	1.5703	1.00856e4	107.04371	16.7190

Totals : 6.03241e4 1675.77076

HPLC 7/1/2013 5:04:25 PM Paul Page 1 of 2

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[3] a) J. Y. Shi, C. A. Wang, Z. J. Li, Q. Wang, Y Zhang, W Wang, *Chem. Eur. J.* **2011**, *17*, 6206-6213; b) P. Riente, J. Yadav, M. A. Pericas, *Org. Lett*. **2012**, *14*, 3668-3671.

[4] K. A. Ahrendt, C. J. Borths, D. W. C. MacMillan, *J. Am. Chem. Soc*. **2000**, *122*,4243-4244.

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[7] H. Gotoh, Y. Hayashi, *Org. Lett.* **2007**, *15*, 2859-2862.