Supplementary Information

Catalytic asymmetric [3,3]-rearrangements of allylic acetimidates

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Table of Contents

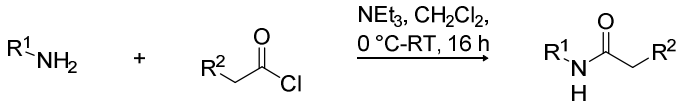
General Remarks

All reactions were performed in oven-dried glassware (oven temperature at 150 °C) and unless otherwise indicated under a positive pressure of nitrogen (about 0.2 bar). Liquids were added *via* syringe and solids were added neat against a nitrogen flow. Solvents were removed by rotary evaporation at a heating bath temperature of 40 °C and 600-10 mbar pressure. Non-volatile compounds were dried *in vacuo* at ca. 0.1 mbar. Absolution of dichloromethane and THF used for reactions was achieved by a solvent purification system. Chloroform was stored in crown-capped bottles over 4Å molecular sieves and used as purchased. For work-up procedures and column chromatography, distilled technical grade solvents were used. *n*-Hexane and *i*-Propanol (HPLC-quality) were used as purchased. **[PPFOP-Cl]$_2$**,[1] **[PPFIP-Cl]$_2$**,[2,3] **[FBIP-Cl]$_2$**[4] and **[FBIPP-Cl]$_2$**[5] were prepared according to literature procedures. PGA (*Penicillin G Amidase* from *Escherichia coli*) (ammonium sulfate suspension, ≥10 units/mg protein) was purchased from Sigma Aldrich. All other laboratory chemicals were used without purification unless otherwise indicated. Yields refer to purified compounds and are calculated in mol% of the used starting material. Except otherwise indicated, reactions were magnetically stirred and monitored by NMR-spectroscopy or thin layer chromatography (TLC) using silica gel plates (silica gel 60 F_{254}). Visualization occurred by fluorescence quenching under UV light and/or staining with $KMnO_4$/NaOH. Purification by column chromatography was performed on silica gel 0.040 – 0.063 mm using a forced flow of eluent at moderate pressure applied with a hand pump. Deactivated silica gel for column chromatography and dry column chromatography was prepared by elution with the corresponding solvent until the eluent shows basic reactivity. NMR-spectra were recorded at 21 °C at 500, 300 or 250 MHz (^{1}H) and 125 or 75 MHz (^{13}C). Chemical shifts δ are referred in terms of ppm and coupling constants *J* are given in Hz. Abbreviations for multiplicities are as follows: *s* (singulet), *d* (duplet), *t* (triplet), *q* (quartet), *m* (multiplet), *p* (pentet), *hex* (sextet), *hep* (heptet) and *b* (broad signal). IR-spectra were recorded by the analytical service of the Universität Stuttgart on a *FT-IR* spectrometer with an ATR unit and the signals are given by wavenumbers (cm^{-1}). Melting points were measured in open glass capillaries and are uncorrected. Optical rotation was measured at the sodium D line in a 100 mm path cell length and values are given in deg mL g^{-1} dm^{-1}. The *ee* values were determined by chiral stationary phase HPLC. Mass spectra were obtained from the analytical service of the Universität Stuttgart. Ionization methods are stated in parenthesis. Elemental analysis was performed by the analytical service of the Universität Stuttgart.

General Procedures

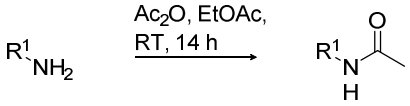
General Procedures for the Synthesis of Amides (GP1)

Synthesis *via* Acid Chlorides (GP1a)[6]



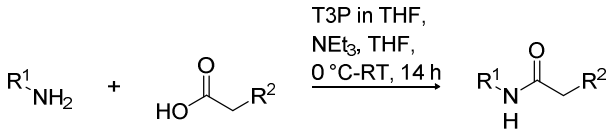
The corresponding amine (1.1 equiv) was dissolved in dry CH_2Cl_2 (8.0 mL per 1.0 mmol) and cooled to 0 °C. NEt_3 (1.1 equiv.) was added in one portion and then the corresponding acid chloride (1.0 equiv.) was added dropwise. Subsequently, the mixture was stirred at room temperature for 16 h. The pH-value of the mixture was then adjusted to pH = 12 by addition of saturated aqueous Na_2CO_3. The phases were separated and the aqueous phase was extracted with CH_2Cl_2. The combined organic phases were washed with aqueous HCl (20.0 mL per 1.0 mmol, 1 M) and dried over Na_2SO_4. Removal of solvent under reduced pressure yielded the amide, which was purified by recrystallization if necessary.

Synthesis *via* Acetic Acid Anhydride (GP1b)[7]



The corresponding amine (1.0 equiv.) was dissolved in EtOAc (2.0 mL pro 1.0 mmol) and Ac_2O (1.1 equiv.) was added dropwise. The mixture was stirred at room temperature for 14 h. Subsequently, saturated aqueous $NaHCO_3$ (20 mL per 1.0 mmol) was added and the phases were separated. The organic phase was washed with aqueous HCl (20 mL per 1.0 mmol, 1M) and dried over Na_2SO_4. Removal of solvent under reduced pressure yielded the amide, which was purified by recrystallization if necessary.

Synthesis *via* Acids (GP1c)[8]

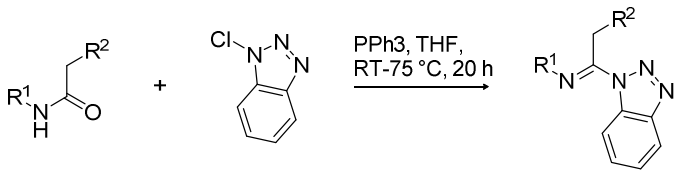


A solution of T3P (1-propanephosphonic anhydride) (1.2 equiv., 50% in THF) was diluted with dry THF (5 mL per 1.0 mmol) and cooled to 0 °C. Subsequently, NEt_3 (2.0 equiv.), the corresponding

amine (1.0 equiv.) and the corresponding acid (1.0 equiv.) were added and the mixture was stirred at room temperature for 14 h. Then H_2O (20 mL per 1.0 mmol) was added and the phases were separated. The aqueous phase was extracted with EtOAc (2 x 20 mL per 1.0 mmol) and the combined organic phases were dried over Na_2SO_4. Removal of solvent under reduced pressure yielded the amide.

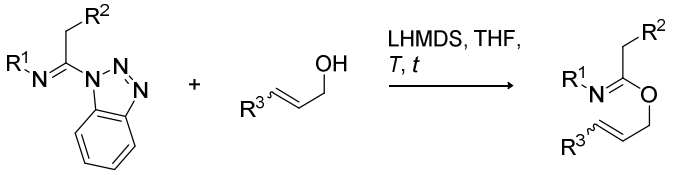
General Procedures for the Synthesis of Allylic Imidates (GP2)

Synthesis of *N*-Imidoylbenzotriazoles (GP2a)[9]



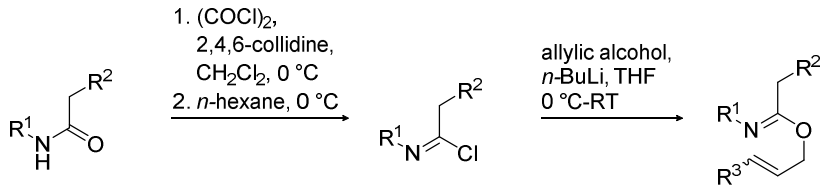
According to a literature procedure,[9] to a solution of triphenylphosphine (2.0 equiv.) in dry THF (10.0 mL per 1.0 mmol) was added 1-chloro-1H-benzo[d][1,2,3]triazole (2.0 equiv) and the mixture was stirred at room temperature for 1 h. Subsequently a solution of the corresponding amide (1.0 equiv.) in dry THF (2.0 mL per 1.0 mmol) was added and the mixture was stirred at 75 °C for 20 h. After cooling to room temperature the solvent was removed under reduced pressure and the *N*-imidoylbenzotriazolewas isolated by column chromatography on deactivated silicagel.

Synthesis of Allylic Imidates *via N*-Imidoylbenzotriazoles (GP2b)



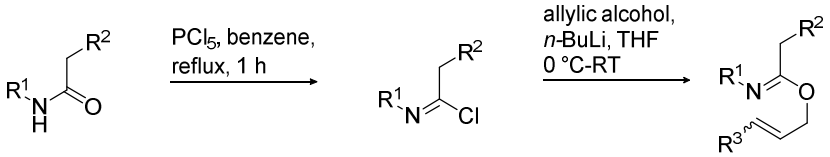
The corresponding allylic alcohol (1.0 equiv.) was dissolved in dry THF (4 mL per 1.0 mmol) and cooled to −70 °C. LHMDS (Lithium bis(trimethylsilyl)amide) (1.0 equiv., 1 M in THF) was added dropwise and the solution was stirred at this temperature for 10 min. Subsequently, a solution of the corresponding *N*-imidoylbenzotriazole (0.5-1.0 equiv.) in dry THF (4 mL per 1.0 mmol) was added and the mixture was stirred at the indicated temperature for the indicated time (monitoring *via* ^{1}H-NMR). The solvent was removed under reduced pressure and the allylic imidate was isolated by column chromatography on deactivated silicagel.

Synthesis of Allylic Imidates *via* Oxalyl Chloride (GP2c)[1,10]



According to a literature procedure,[1,10] a solution of the corresponding amide (1.5-2.0 equiv.) and 2,4,6-collidine (3.0 equiv.) in dry CH_2Cl_2 (4 mL per 1.0 mmol) was cooled to 0 °C. A solution of oxalyl chloride (1.5-2.0 equiv.) in dry CH_2Cl_2 (1 mL per 1.0 mmol) was added dropwise and the mixture was stirred at 0 °C for 30 min. Subsequently, the solvent was removed under vacuum at room temperature and *n*-hexane (4 mL per 1.0 mmol) was added to the residue. Ultrasonification yielded a suspension, which was stirred at 0 °C for 1 h and then filtered directly into a solution of the corresponding alcoholate [prepared by addition of *n*-BuLi (1.0 equiv., 15% in hexane) to a solution of the corresponding allylic alcohol (1.0 equiv.) in dry THF (1 mL per 0.5 mmol) at 0 °C and stirring of the mixture at 0 °C for 15 min]. The mixture was stirred at room temperature for 14 h, then diluted with Et_2O (2 mL per 1.0 mmol amide) and filtered over silicagel. Removal of solvent under reduced pressure and column chromatography or dry column chromatography of the residue on deactivated silicagel yielded the allylic imidate.

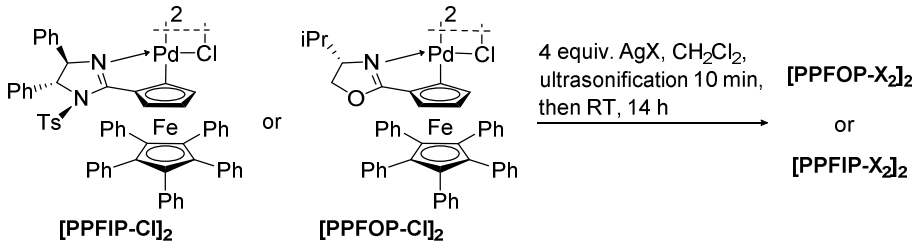
Synthesis of Allylic Imidates *via* PCl_5 (GP2d)[11]



According to a literature procedure,[11] to a solution of the corresponding amide (1.0 equiv.) in dry benzene (2 mL per 1.0 mmol) was added PCl_5 (1.0 equiv.) and the mixture was refluxed for 1 h. After cooling to room temperature the solvent was removed under reduced pressure (venting with N_2), the residue was dissolved in dry THF (1.0 mL per 1.0 mmol) and directly added to a solution of the corresponding alcoholate [prepared by addition of *n*-BuLi (1.0 equiv., 15% in hexane) to a solution of the corresponding allylic alcohol (1.0 equiv.) in dry THF (1 mL per 0.5 mmol) at 0 °C and stirring of the mixture at 0 °C for 15 min]. The mixture was stirred at room temperature for 14 h and the solvent removed under reduced pressure. The residue was dissolved in CH_2Cl_2 and washed with H_2O. The organic phase was dried over Na_2SO_4 and the solvent removed under reduced pressure. Column chromatography on deactivated silicagel yielded the allylic imidate.

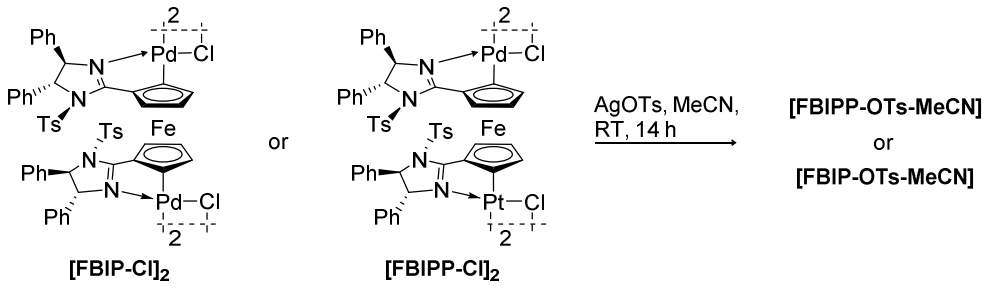
General Procedures for the Activation of Pre-Catalysts (GP3)

Activation of Mono-Palladacycles (GP3a)



The corresponding palladacycle (1.0 equiv.) and the corresponding silver salt (4.0 equiv) were suspended in dry CH_2Cl_2 (0.5 mL per 5 mg palladacycle) and ultrasonificated for 10 min. The mixture was stirred at room temperature for 14 h and then filtered through CaH_2/celite (1:1) under N_2. The solvent was removed by a stream of N_2 and then high vacuum was applied. A stock solution was prepared by the addition of a definite amount of the corresponding solvent.

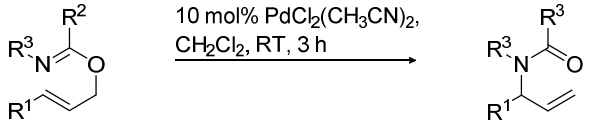
Activation of the Bis-Palladacycle and the Pallada-/Platinacycle (GP3b)



To a solution of AgOTs (4.0 equiv.) in dry MeCN (0.5 mL per 5 mg bis-Palladacyle) was added the corresponding bis-palladacycle (1.0 equiv.). The mixture was stirred at room temperature for 14 h and then filtered through CaH_2/celite (1:1) under N_2. The solvent was removed by a stream of N_2 and then high vacuum was applied. A stock solution was prepared by the addition of a definite amount of the corresponding solvent.

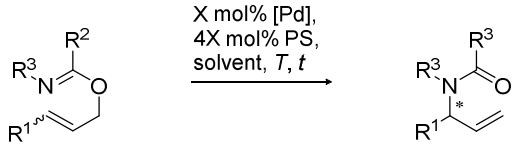
General Procedures for the Rearrangement of Allylic Imidates (GP4)

Non-Enantioselective Rearrangements (GP4a)[12]



To a solution of the corresponding allylic imidate (1.0 equiv.) in dry CH_2Cl_2 (0.5 mL per 0.1 mmol) was added $PdCl_2(CH_3CN)_2$ (10 mol%) and the mixture was stirred at room temperature for 3 h. Subsequently the solvent was removed under educed pressure and the corresponding allylic amide was isolated by column chromatography on silicagel.

Catalytic Asymmetric Rearrangements (GP4b)



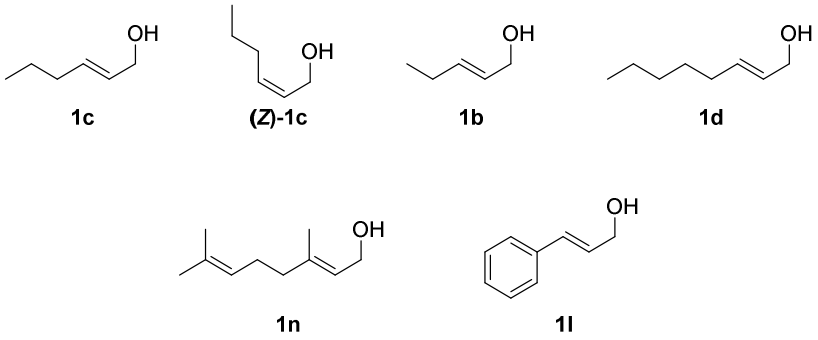
A dry screw-cap vial was charged with the corresponding allylic imidate (1.0 equiv.), then vacuum was applied and the vial was refilled with N_2 (3 times). A stock solution of proton sponge (1,8-bis-(*N*,*N*-dimethylamino)naphthalene, PS) (4X mol%) [only used in the case of mono-palladacycles] in the indicated solvent and a stock solution of the corresponding activated catalyst [prepared according to **GP3**] in the indicated solvent were added. The amount of solvent was reduced by a stream of N_2 if necessary (final concentration: around 150 µL per 100 µmol of substrate). The vial was closed by a screw-cap and the mixture was stirred at the indicated temperature for the indicated time. Subsequently, the solvent was removed under reduced pressure and mesitylene (10 µL per each 50 µmol of substrate) was added to the crude product as an internal standard followed by $CDCl_3$ (1 mL) to determine conversion and yield by ^{1}H-NMR. The crude product was afterwards directly used for silicagel chromatography to isolate the corresponding allylic amide. The purified samples were used to determine the *ee* value by HPLC.

Synthesis of Allylic Alcohols (1)

Commercially available:

The following allylic alcohols used in these investigations are commercially available in sufficiently high isomerical purity:

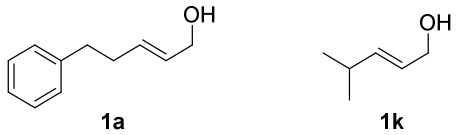
trans-hex-2-en-1-ol (**1c**), *cis*-hex-2-en-1-ol ((**Z**)-**1c**), *trans*-pent-2-en-1-ol (**1b**), *trans*-oct-2-en-1-ol (1**d**), geraniol (**1n**), cinnamyl alcohol (**1l**).



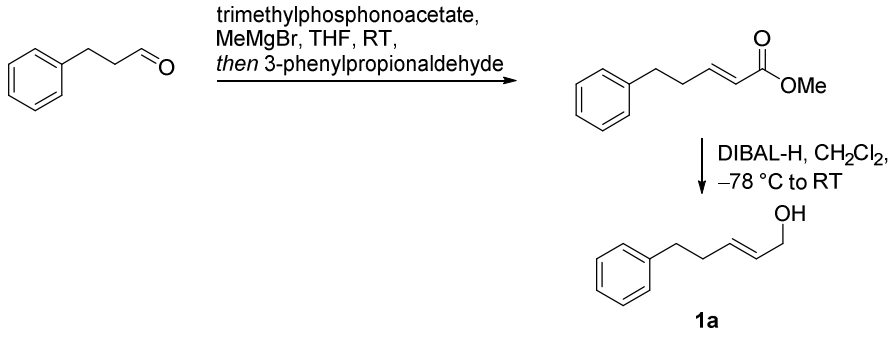
Via HWE-reaction:

The following alcohols were prepared according to a literature procedure utilizing the Horner-Wadsworth-Emmons reaction (HWE),[13] followed by reduction with DIBAL-H:

Trans-5-phenylpent-2-en-1-ol (**1a**)[14] and *trans*-4-methylpent-2-en-1-ol (**1k**).[15]



Representative Procedure for HWE and DIBAL-H-Reduction: trans-5-Phenylpent-2-en-1-ol (1a)[14]



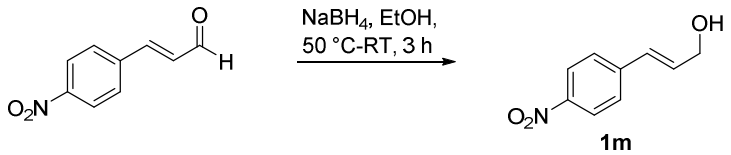
To a solution of trimethylphosphonoacetate (1.00 equiv., 1.40 mmol, 252.0 mg, 200 µL) in THF (10 mL) was added MeMgBr (0.86 equiv., 1.20 mmol, 400 µL of a 3M solution in Et_2O) dropwise at room temperature. This mixture was stirred at room temperature for additional 20 min, then 3-phenylpropionaldehyde (0.97 equiv., 1.32 mmol, 183.1 mg, 180 µL) was added in one portion and the solution was stirred at room temperature for another 1 h. Subsequently, saturated aqueous NH_4Cl (6.3 mL) was added and the mixture was extracted with Et_2O (2 times 20 mL). The combined organic phases were dried over Na_2SO_4 and the solvent was removed under reduced pressure. Purification by silica gel chromatography (petrol ether/ethyl acetate = 10/1) yielded the intermediate ester as a colorless oil (0.58 mmol, 110.0 mg, 48%).

$C_{12}H_{14}O_2$, MW: 190.24 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.34-7.25 (*m*, 2 H, Ar-*H*), 7.24-7.14 (*m*, 3 H, Ar-*H*), 7.00 (*dt*, *J* = 15.6, 6.8, 1 H, C(=O)CH=C*H*), 5.85 (*dt*, *J* = 15.6, 1.5, 1 H, C(=O)C*H*=CH), 3.72 (*s*, 3 H, OCH_3), 2.82-2.73 (*m*, 2 H, $CH_2CH_2C_6H_5$), 2.59-2.46 (*m*, 2 H, $CH_2CH_2C_6H_5$). The analytical data are in accordance with the literature.[14]

To a solution of the ester (1.00 equiv., 2.09 mmol, 398.7 mg) in CH_2Cl_2 (5 mL) at −78 °C was added DIBAL-H (2.63 equiv., 5.50 mmol, 5 mL of a 1.1M solution in cyclohexane) in one portion. After stirring for 15 min at this temperature, the solution was slowly warmed to room temperature and stirred for 1.5 h. Subsequently, the mixture was cooled again to −78 °C and aqueous hydrochloric acid (1M, 28 mL) was added until all solid had dissolved. The mixture was extracted with CH_2Cl_2 (2 times 20 mL), the combined organic phases were dried over $MgSO_4$ and the solvent removed under reduced pressure. Purification by silica gel chromatography (petrol ether/ethyl acetate = 5/1) yielded the alcohol **1a** as a colorless oil (2.03 mmol, 329.2 mg, 97%).

$C_{11}H_{14}O$, MW: 162.23 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.32-7.25 (*m*, 2 H, Ar-*H*), 7.23-7.14 (*m*, 3 H, Ar-*H*), 5.81-5.61 (*m*, 2 H, C*H*=C*H*), 4.09 (*d*, *J* = 5.0, 2 H, OCH_2), 2.75-2.67 (*m*, 2 H, $CH_2CH_2C_6H_5$), 2.42-2.33 (*m*, 2 H, $CH_2CH_2C_6H_5$), 1.28 (*bs*, 1 H, O*H*). The analytical data are in accordance with the literature.[14]

trans-3-(4-Nitrophenyl)prop-2-en-1-ol (1m)



1m

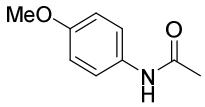
Following a literature procedure,[16] 4-nitrocinnamaldehyde (1.00 equiv., 2.77 mmol, 490.0 mg) was dissolved in EtOH (40 mL) by heating to 50 °C. A solution of $NaBH_4$ (1.00 equiv., 2.77 mmol, 105.0 mg) in EtOH (11 mL) was added in one portion and the mixture was stirred at room temperature

for 3 h. Subsequently, the mixture was filtered through celite and concentrated aqueous HCl was added until the solution turned from orange to yellow. Then the solvent was removed under reduced pressure, the residue was dissolved in $CHCl_3$ (50 mL) and washed with H_2O (50 mL). The phases were separated, the organic phase was dried over $MgSO_4$. Removal of solvent under reduced pressure and washing of the residue with Et_2O yielded the alcohol **1m** as a yellow solid (2.42 mmol, 434.2 mg, 88%).

$C_9H_7NO_3$, MW: 179.17 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 8.22-8.16 (*m*, 2 H, *m*-$C_6H_4NO_2$), 7.55-7.48 (*m*, 2 H, *o*-$C_6H_4NO_2$), 6.72 (*bd*, *J* = 16.1, 1 H, CH=C*H*$C_6H_4NO_2$), 6.54 (*dt*, *J* = 16.1, 5.0, 1 H, C*H*=CH$C_6H_4NO_2$), 4.44-4.37 (*m*, 2 H, OCH_2), 1.55 (*t*, *J* = 5.7, 1 H, O*H*). The analytical data are in accordance with the literature.[16]

Synthesis of Amides (6)

N-(4-Methoxyphenyl)acetamide (6a)

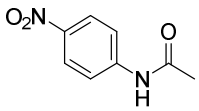


6a

According to **GP1a**, *p*-anisidine (55.0 mml, 6.77 g) was reacted with Ac_2O (57.7 mmol, 1.84 g. 1.70 mL). After recrystallization from EtOH/H_2O (1/1) the amide **6a** was obtained as a grey-white solid (38.3 mmol, 6.32 g, 70%).

$C_9H_{11}NO_2$, MW: 165.19 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.42-7.35 (*m*, 2 H, *o*-C_6H_4OMe), 7.04 (*bs*, 1 H, N*H*), 6.89-6.81 (*m*, 2 H, *m*-C_6H_4OMe), 3.79 (*s*, 3 H, OCH_3), 2.14 (*s*, 3 H, C(=O)CH_3). The analytical data are in accordance with the literature.[17]

N-(4-Nitrophenyl)acetamide (6e)

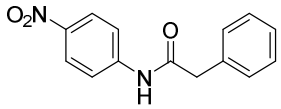


6e

According to **GP1a**, 4-nitroaniline (16.2 mmol, 2.24 g) was reacted with Ac_2O (17.1 mmol. 1.75 g, 1.62 mL). After recrystallization from EtOH/H_2O (1/1) the amide **6e** was obtained as a yellow solid (14.0 mmol, 2.53 g, 86%).

$C_8H_8N_2O_3$, MW: 180.16 g mol^{-1}. **^{1}H-NMR (300 MHz, DMSO-d_6):** δ = 10.56 (*bs*, 1 H, N*H*), 8.25-8.17 (*m*, 2 H, *m*-$C_6H_4NO_2$), 7.86-7.79 (*m*, 2 H, *o*-$C_6H_4NO_2$), 2.12 (*s*, 3 H, C(=O)CH_3). The analytical data are in accordance with the literature.[18]

N-(4-Nitrophenyl)-2-phenylacetamide (6f)

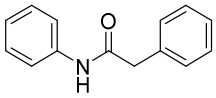


6f

According to **GP1c**, 4-nitroaniline (20.0 mmol, 2.76 g) was reacted with phenylacetic acid (20.0 mmol, 2.73 g). The amide **6f** was obtained as a yellow solid (19.9 mmol, 5.09 g, 99%).

$C_{14}H_{12}N_2O_3$, MW: 256.26 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 8.20-8.13 (*m*, 2 H, *m*-$C_6H_4NO_2$), 7.64-7.57 (*m*, 2 H, *o*-$C_6H_4NO_2$), 7.47-7.31 (*m*, 6 H, C_6H_5 & N*H*), 3.80 (*s*, 2 H, C(=O)CH_2). The analytical data are in accordance with the literature.[19]

N-Phenyl-2-phenylacetamide (6g)

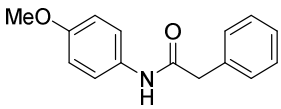


6g

According to **GP1b**, aniline (17.5 mmol, 1.63 g, 1.60 mL) was reacted with phenylacetyl chloride (17.0 mmol, 2.63 g, 2.25 mL). The amide **6g** was obtained as a yellow solid (16.2 mmol, 3.43 g, 95%).

$C_{14}H_{13}NO$, MW: 211.26 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.47-7.26 (*m*, 9 H, $CH_2C_6H_5$ & *o*- & *m*-C_6H_5), 7.19-7.06 (*m*, 2 H, *p*-C_6H_5 & N*H*), 3.77 (*s*, 2 H, C(=O)CH_2). The analytical data are in accordance with the literature.[20]

N-(4-Methoxyphenyl)-2-phenylacetamide (6h)

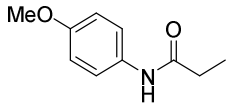


6h

According to **GP1b**, *p*-anisidine (16.2 mmol, 2.00 g) was reacted with phenylacetyl chloride (14.8 mmol, 2.28 g, 1.96 mL). The amide **6h** was obtained as brown solid (14.7 mmol, 3.57 g, 99%).

$C_{15}H_{15}NO_2$, MW: 241.29 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.43-7.28 (*m*, 7 H, *o*-C_6H_4OMe & C_6H_5), 7.09 (*bs*, 1 H, N*H*), 6.84-6.77 (*m*, 2 H, *m*-C_6H_4OMe), 3.76 (*s*, 3 H, OCH_3), 3.71 (*s*, 2 H, C(=O)CH_2). The analytical data are in accordance with the literature.[20]

N-(4-Methoxyphenyl)propionamide (6o)

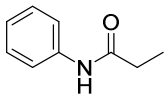


6o

According to **GP1b**, *p*-anisidine (16.2 mmol, 2.00 g) was reacted with propionyl chloride (15.5 mmol, 1.43 g, 1.35 mL). The amide **6o** was obtained as a brown solid (15.1 mmol, 2.70 g, 97%).

$C_{10}H_{13}NO_2$, MW: 179.22 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.44-7.37 (*m*, 2 H, *o*-C_6H_4OMe), 7.32 (*bs*, 1 H, N*H*), 6.87-6.80 (*m*, 2 H, *m*-C_6H_4OMe), 3.78 (*s*, 3 H, OCH_3), 2.35 (*q*, *J* = 7.5, 2 H, C(=O)CH_2), 1.23 (*t*, *J* = 7.5, 3 H, C(=O)CH_2CH_3). The analytical data are in accordance with the literature.[21]

N-Phenylpropionamide (6p)



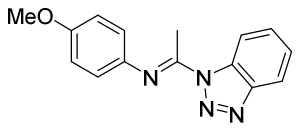
6p

According to **GP1b**, aniline (16.2 mmol, 1.51 g, 1.48 mL) was reacted with propionyl chloride (14.7 mmol, 1.36 g, 1.29 mL). The amide **6p** was obtained as a slightly yellow solid (14.5 mmol, 2.17 g, 99%).

$C_9H_{11}NO$, MW: 149.19 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.52 (*bd*, *J* = 8.2, 2 H, *o*-C_6H_5), 7.32 (*bt*, *J* = 7.7, 2 H, *m*-C_6H_5), 7.17-7.05 (*m*, 2 H, *p*-C_6H_5 & N*H*), 2.39 (*q*, *J* = 7.5, 2 H, C(=O)CH_2), 1.25 (*t*, *J* = 7.5, 3 H, CH_3). The analytical data are in accordance with the literature.[22]

Synthesis of *N*-Imidoylbenzotriazoles (8)

(*E*)-1-(1H-Benzo[d][1,2,3]triazol-1-yl)-*N*-(4-methoxyphenyl)ethan-1-imine (8a)

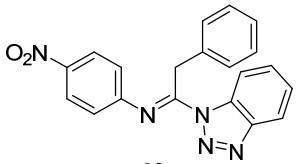


8a

According to **GP2a**, triphenylphosphine (15.02 mmol, 3.94 g) was reacted with 1-chloro-1H-benzo[d][1,2,3]triazole (15.04 mmol, 2.31 g) and *N*-(4-methoxyphenyl)acetamide **6a** (5.00 mml, 821.0 mg) to yield **8a** after purification by column chromatography on deactivated silicagel (petrol ether/ethyl acetate = 10/1 + 3% NEt_3) as a white solid (3.45 mmol, 918.2 mg, 69%).

$C_{15}H_{14}N_4O$, MW: 266.30 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 8.55 (*dt*, *J* = 8.3, 1.0, 1 H, C_6H_4), 8.12 (*dt*, *J* = 8.3, 1.0, 1 H, C_6H_4), 7.60 (*ddd*, *J* = 8.3, 7.1, 1.0, 1 H, C_6H_4), 7.48 (*ddd*, *J* = 8.3, 7.1, 1.0, 1 H, C_6H_4), 7.00-6.88 (*m*, 4 H, C_6H_4OMe), 3.85 (*s*, 3 H, OCH_3), 2.77 (*s*, 3 H, C(=N)CH_3). The analytical data are in accordance with the literature.[9]

(*E*)-1-(1H-Benzo[d][1,2,3]triazol-1-yl)-*N*-(4-nitrophenyl)-2-phenylethan-1-imine (8f)



8f

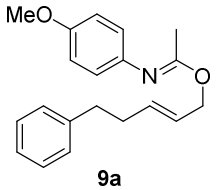
According to **GP2a**, triphenylphosphine (1.00 mmol, 262.0 mg) was reacted with 1-Chloro-1H-benzo[d][1,2,3]triazole (1.00 mmol, 154.0 mg) and *N*-(4-nitrophenyl)-2-phenylacetamide **6f** (0.50 mmol, 128.0 mg) to yield **8f** after purification by column chromatography on deactivated silicagel (petrol ether/ethyl acetate = 18/1 + 3% NEt_3) as a yellow solid (0.29 mmol, 102.0 mg, 57%).

$C_{20}H_{15}N_5O_2$, MW: 357.37 g mol^{-1}. **Mp:** 89.3-91.0 °C (Decomposition). **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 8.45 (*d*, *J* = 8.3, 1 H, C_6H_4), 8.31-8.25 (*m*, 2 H, *m*-$C_6H_4NO_2$), 8.15 (*d*, *J* = 8.3, 1 H, C_6H_4), 7.64 (*dt*, *J* = 8.3, 1.0, 1 H, C_6H_4), 7.52 (*dt*, *J* = 8.3, 1.0, 1 H, C_6H_4), 7.23-7.17 (*m*, 3 H, C_6H_5), 7.13-7.07 (*m*, 2 H, C_6H_5), 7.06-7.00 (*m*, 2 H, *o*-$C_6H_4NO_2$), 4.61 (*s*, 2 H, $C_6H_5CH_2$). **^{13}C-NMR (75 MHz, $CDCl_3$):** δ = 155.2, 152.8, 146.8, 144.5, 134.4, 131.3, 129.8, 128.9, 128.7, 127.3, 126.0, 125.2, 120.7, 120.2, 115.4, 35.4. **IR ($CDCl_3$):** $\tilde{\nu}$ = 3065, 1672, 1588, 1512, 1486, 1449, 1397, 1339, 1292, 1251, 1222, 1110, 1070, 1026, 857, 784, 769, 751, 715, 699. **MS (ESI) *m/z*:** 380.1 (100%, $[M+Na]^+$), 330.1 (35%),

279.1 (3%), 239.1 (9%), 182.1 (1%), 142.0 (2%), 118.1 (5%), 91.1 (1%). **HRMS (ESI) *m/z*:** calculated for $C_{20}H_{15}N_5O_2$ + Na: 380.1118; found: 380.1118.

Synthesis of Allylic Imidates (9)

((*E*)-5-Phenylpent-2-en-1-yl)-*N*-(4-methoxyphenyl)acetimidate (9a)



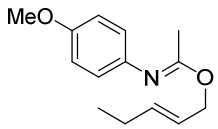
9a

According to **GP2c**, *N*-(4-methoxyphenyl)acetamide **6a** (5.45 mmol, 900.0 mg) was reacted with oxalyl chloride (5.45 mmol, 710.4 mg, 0.48 mL) and (*E*)-5-phenylpent-2-en-1-ol **1a** (1.82 mmol, 295.0 mg) to yield **9a** after purification by column chromatography on deactivated silicagel (petrol ether/ethyl acetate = 18/1 + 3% NEt_3) as a slightly yellow oil (1.18 mmol, 365.1 mg, 65%).

Alternatively, (*E*)-5-phenylpent-2-en-1-ol **1a** (2.10 mmol, 340.0 mg) was reacted with LHMDS (2.10 mml, 2.10 mL, 1M in THF) and (*E*)-1-(1H-benzo[d][1,2,3]triazol-1-yl)-*N*-(4-methoxyphenyl)ethan-1-imine **8a** (1.17 mmol, 312.0 mg) at 60 °C for 20 h. Purification by column chromatography on deactivated silicagel (petrol ether/ethyl acetate = 18/1 + 3% NEt_3) yielded **9a** as a slightly yellow oil (0.66 mmol, 203.1 mg, 56%).

$C_{20}H_{23}NO_2$, MW: 309.40 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.32-7.25 (*m*, 2 H, *m*-C_6H_5), 7.22-7.15 (*m*, 3 H, *o*-C_6H_5 & *p*-C_6H_5), 6.87-6.81 (*m*, 2 H, *o*-C_6H_4OMe), 6.72-6.65 (*m*, 2 H, *m*-C_6H_4OMe), 5.92-5.80 (*m*, 1 H, C*H*=CH), 5.79-5.58 (*m*, 1 H, CH=C*H*), 4.62 (*d*, *J* = 6.0, 2 H, OCH_2), 3.79 (*s*, 3 H, OCH_3), 2.77-2.70 (*m*, 2 H, $C_6H_5CH_2CH_2$), 2.46-2.36 (*m*, 2 H, $C_6H_5CH_2CH_2$), 1.84 (*s*, 3 H, C(=O)CH_3). **^{13}C-NMR (75 MHz, $CDCl_3$):** δ = 161.4, 155.7, 142.5, 141.9, 134.7, 128.6, 128.5, 126.0, 125.6, 122.1, 114.4, 66.5 (OCH_2), 55.6, 35.6, 34.4, 16.3. **IR ($CDCl_3$):** $\tilde{\nu}$ = 3027, 2933, 2834, 2183, 1960, 1774, 1747, 1668, 1506, 1455, 1381, 1271, 1234, 1038, 967, 840, 750, 699. **MS (EI) *m/z*:** 309.1 (11%, $[M]^+$), 218.1 (100%), 165.1 (24%), 123.0 (55%), 91.0 (43%). **HRMS (ESI) *m/z*:** calculated for $C_{20}H_{23}NO_2$ + Na: 332.1621; found: 332.1628.

((*E*)-Pent-2-en-1-yl)-*N*-(4-methoxyphenyl)acetimidate (9b)

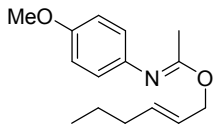


9b

According to **GP2c**, *N*-(4-methoxyphenyl)acetamide **6a** (2.00 mmol, 330.0 mg) was reacted with oxalyl chloride (2.04 mmol, 259.0 mg, 175 µL) and (*E*)-pent-2-en-1-ol **1b** (0.99 mmol, 85.0 mg, 100 µL) to yield **9b** after purification by dry column chromatography on deactivated silicagel (petrol ether + 2% NEt_3) as a colorless oil (0.43 mmol, 99.2 mg, 43%).

$C_{14}H_{19}NO_2$, MW: 233.31 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 6.87-6.80 (*m* 2 H, *o*-C_6H_4OMe), 6.72-6.66 (*m*, 2 H, *m*C_6H_4OMe), 5.92-5.80 (*m*, 1 H, C*H*=CH), 5.75-5.63 (*m*, 1 H, CH=C*H*), 4.62 (*dd*, *J* = 6.4, 1.0, 2 H, OCH_2), 3.78 (*s*, 3 H, OCH_3), 2.17-2.05 (*m*, 2 H, CH_2CH$_3$), 1.84 (*s*, 3 H, C(=N)CH_3), 1.03 (*t*, *J* = 7.4, 3 H, CH$_2$CH_3). **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 161.4, 155.5, 142.4, 137.3, 123.7, 122.0, 114.3, 66.6, 55.5, 25.4, 16.2, 13.3. **IR ($CDCl_3$):** $\tilde{\nu}$ = 2961, 2933, 2834, 1666, 1609, 1504, 1462, 1440, 1379, 1363, 1286, 1270, 1230, 1179, 1102, 1036, 965, 927, 838, 801, 753, 710, 643, 623, 538. **MS (ESI) *m/z*:** 256.1 (10%, [M+Na]$^+$), 234.2 (4%, [M+H]$^+$), 192.1 (1%), 166.1 (100%), 124.1 (25%). **HRMS (ESI) *m/z*:** calculated for $C_{14}H_{19}NO_2$ + Na: 256.1308; found: 256.1281.

((*E*)-Hex-2-en-1-yl)-*N*-(4-methoxyphenyl)acetimidate (9c)



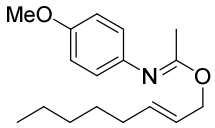
9c

According to **GP2c**, *N*-(4-methoxyphenyl)acetamide **6a** (2.00 mmol, 330.0 mg) was reacted with oxalyl chloride (2.04 mmol, 259.0 mg, 175 µL) and (*E*)-hex-2-en-1-ol **1c** (1.01 mmol, 100.8 mg, 120 µL) to yield **9c** after purification by dry column chromatography on deactivated silicagel (petrol ether + 3% NEt_3) as a colorless oil (0.68 mmol, 167.2 mg, 67%).

$C_{15}H_{21}NO_2$, MW: 247.34 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 6.79-6.73 (*m*, 2 H, *o*-C_6H_4OMe), 6.65-6.58 (*m*, 2 H, *m*-C_6H_4OMe), 5.80-5.56 (*m*, 2 H, C*H*=C*H*), 4.55 (*dd*, *J* = 6.0, 0.9, 2 H, OCH_2), 3.71 (*s*, 3 H, OCH_3), 1.99 (*bq*, *J* = 7.0, 2 H, CH_2CH$_2$CH$_3$), 1.77 (*s*, 3 H, C(=N)CH_3), 1.37 (*pseudo hex*, *J* = 7.3, 2 H, CH$_2$CH_2CH$_3$), 0.85 (*t*, *J* = 7.3, 3 H, CH$_2$CH$_2$CH_3). **^{13}C-NMR (75 MHz, $CDCl_3$):** δ = 161.3, 155.5, 142.4, 135.5, 124.9, 122.0, 114.2, 66.5, 55.5, 34.5, 22.2, 16.2, 13.7. **IR ($CDCl_3$):** $\tilde{\nu}$ = 2957, 2930, 2872, 2834, 1667, 1505, 1463, 1440, 1379, 1364, 1287, 1271, 1232, 1179, 1102,

1038, 839, 803, 753, 711, 644, 623. **MS (ESI) *m/z*:** 303.2 (1%), 270.2 (19%, $[M+Na]^+$), 248.2 (1%, $[M+H]^+$), 166.1 (100%), 124.1 (22%). **HRMS (ESI) *m/z*:** calculated for $C_{15}H_{21}NO_2$ + Na: 270.1465; found: 270.1460.

((*E*)-Oct-2-en-1-yl)-*N*-(4-methoxyphenyl)acetimidate (9d)

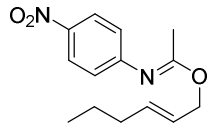


9d

According to **GP2c**, *N*-(4-methoxyphenyl)acetamide **6a** (2.00 mmol, 330.0 mg) was reacted with oxalyl chloride (2.04 mmol, 259.0 mg, 175 µL) and (*E*)-oct-2-en-1-ol **1d** (0.98 mmol, 126.0 mg, 150 µL) to yield **9d** after purification by dry column chromatography on deactivated silicagel (petrol ether + 2% NEt_3) as a colorless oil (0.36 mmol, 98.7 mg, 37%).

$C_{17}H_{25}NO_2$, MW: 275.39 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 6.87-6.80 (*m*, 2 H, *o*-C_6H_4OMe), 6.72-6.65 (*m*, 2 H, *m*-C_6H_4OMe), 5.87-5.75 (*m*, 1 H, C*H*=CH), 5.74-5.63 (*m*, 1 H, CH=C*H*), 4.62 (*dd*, *J* = 6.2, 0.9, 2 H, OCH_2), 3.78 (*s*, 3 H, OCH_3), 2.08 (*bq*, *J* = 7.0, 2 H, CH_2(CH_2)$_3$$CH_3$), 1.84 (*s*, 3 H, C(=N)CH_3), 1.47-1.24 (*m*, 6 H, CH_2(CH_2)$_3$$CH_3$), 0.89 (*t*, *J* = 6.9, 3 H, CH_2(CH_2)$_3$CH_3). **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 161.3, 155.5, 142.4, 135.8, 124.6, 122.0, 114.3, 66.5, 55.5, 32.4, 31.4, 28.7, 22.6, 16.2, 14.1. **IR ($CDCl_3$):** $\tilde{\nu}$ = 2955, 2926, 2856, 1666, 1504, 1463, 1440, 1380, 1364, 1286, 1270, 1230, 1179, 1101, 1037, 964, 837, 801, 753, 710, 643, 623, 559, 537. **MS (ESI) *m/z*:** 298.2 (28%, $[M+Na]^+$), 276.2 (1%, $[M+H]^+$), 166.1 (100%), 124.1 (23%). **HRMS (ESI) *m/z*:** calculated for $C_{17}H_{25}NO_2$ + Na: 298.1778; found: 298.1754.

((*E*)-Hex-2-en-1-yl)-*N*-(4-nitrophenyl)acetimidate (9e)



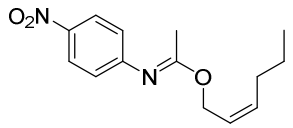
9e

According to **GP2d**, *N*-(4-nitrophenyl)acetamide **6e** (1.00 mmol, 180.0 mg) was reacted with PCl_5 (1.00 mmol, 208.2 mg) and (*E*)-Hex-2-en-1-ol **1c** (1.00 mmol, 99.1 mg, 118 µL) to yield **9e** after purification by column chromatography on deactivated silicagel (petrol ether/ethyl acetate = 35/1 +3% NEt_3) as a slightly yellow solid (0.46 mmol, 120.7 mg, 46%).

$C_{14}H_{18}N_2O_3$, MW: 262.30 g mol^{-1}. **Mp:** 69.9-70.8 °C. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 8.22-8.14 (*m*, 2 H, *m*-$C_6H_4NO_2$), 6.88-6.81 (*m*, 2 H, *o*-$C_6H_4NO_2$), 5.89-5.77 (*m*, 1 H, C*H*=CH), 5.74-5.62 (*m*,

1 H, CH=C*H*), 4.64 (*dd*, *J* = 6.2, 1.0, 2 H, OCH_2), 2.08 (*bq*, *J* = 7.1, 2 H, CH_2CH$_2$CH$_3$), 1.86 (*s*, 3 H, C(=N)CH_3), 1.45 (*pseudo hex*, *J* = 7.3 2 H, CH$_2$CH_2CH$_3$), 0.93 (*t*, *J* = 7.3, 3 H, CH$_2$CH$_2$CH_3). **^{13}C-NMR (75 MHz, CDCl$_3$):** δ = 161.1, 155.6, 143.5, 136.3, 125.1, 124.1, 121.5, 67.3, 34.4, 22.1, 16.6, 13.7. **IR (CDCl$_3$):** $\tilde{\nu}$ = 2953, 2870, 1681, 1591, 1504, 1458, 1385, 1336, 1285, 1241, 1172, 1108, 1036, 967, 918, 868, 853, 778, 753, 707, 673, 578, 561. **MS (ESI) *m/z*:** 395.2 (10%), 383.1 (13%), 317.1 (9%), 295.1 (5%), 285.1 (88%, [M+Na]$^+$), 234.2 (6%), 181.1 (100%), 152.1 (28%), 139.1 (15%), 110.1 (7%), 93.1 (18%). **HRMS (ESI) *m/z*:** calculated for $C_{14}H_{18}N_2O_3$ + Na: 285.1210; found: 285.1196.

((*Z*)-Hex-2-en-1-yl)-*N*-(4-nitrophenyl)acetimidate ((*Z*)-9e)

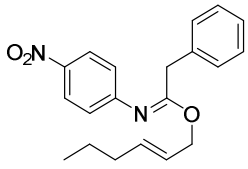


(*Z*)-9e

According to **GP2d**, *N*-(4-nitrophenyl)acetamide **6e** (1.00 mmol, 180.0 mg) was reacted with PCl_5 (1.00 mmol, 208.2 mg) and (*Z*)-hex-2-en-1-ol **(*Z*)-1c** (1.00 mmol, 99.1 mg, 118 µL) to yield **(*Z*)-9e** after purification by column chromatography on deactivated silicagel (petrol ether/ethyl acetate = 20/1 + 3% NEt_3) as a yellow oil (0.52 mmol, 136.0 mg, 52%).

$C_{14}H_{18}N_2O_3$, MW: 262.30 g mol^{-1}. **^{1}H-NMR (300 MHz, CDCl$_3$):** δ = 8.21-8.15 (*m*, 2 H, *m*-$C_6H_4NO_2$), 6.89-6.82 (*m*, 2 H, *o*-$C_6H_4NO_2$), 5.74-5.61 (*m*, 2 H, C*H*=C*H*), 4.78-4.72 (*m*, 2 H, OCH_2), 2.17-2.07 (*m*, 2 H, CH_2CH$_2$CH$_3$), 1.86 (*s*, 3 H, C(=N)CH_3), 1.42 (*pseudo hex*, *J* = 7.3, CH$_2$CH_2CH$_3$), 0.92 (*t*, *J* = 7.3, 3 H, CH$_2$CH$_2$CH_3). **^{13}C-NMR (75 MHz, CDCl$_3$):** δ = 161.2, 155.5, 143.5, 135.0, 125.1, 123.8, 121.5, 62.4, 29.7, 22.6, 16.6, 13.7. **IR (CDCl$_3$):** $\tilde{\nu}$ = 2961, 2933, 1670, 1591, 1511, 1377, 1338, 1291, 1243, 1170, 1110, 1035, 965, 904, 864, 852, 781227, 649. **MS (ESI) *m/z*:** 319.1 (1%), 285.1 (79%, [M+Na]$^+$), 181.1 (100%), 152.1 (1%), 139.1 (12%), 122.1 (3%), 93.1 (17%). **HRMS (ESI) *m/z*:** calculated for $C_{14}H_{18}N_2O_3$ + Na: 285.1210; found: 285.1199.

((*E*)-Hex-2-en-1-yl)-*N*-(4-nitrophenyl)-2-phenylacetimidate (9f)



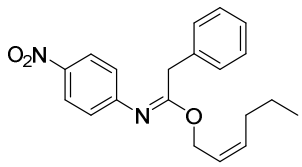
9f

According to **GP2d**, *N*-(4-nitrophenyl)-2-phenylacetamide **6f** (1.00 mmol, 256.0 mg) was reacted with PCl_5 (1.00 mmol, 208.0 mg) and (*E*)-hex-2-en-1-ol **1c** (1.00 mmol, 99.1 mg, 118 µL) to yield **9f** after

purification by column chromatography on deactivated silicagel (petrol ether/ethyl acetate = 18/1 + 3% NEt_3) as a colorless oil (0.23 mmol, 78.5 mg, 23%).

$C_{20}H_{22}N_2O_3$, MW: 338.40 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 8.19-8.12 (*m*, 2 H, *m*-$C_6H_4NO_2$), 7.31-7.21 (*m*, 3 H, *m*-C_6H_5 & *p*-C_6H_5), 7.10-7.05 (*m*, 2 H, *o*-C_6H_5), 6.83-6.76 (*m*, 2 H, *o*-$C_6H_4NO_2$), 5.85-5.73 (*m*, 1 H, C*H*=CH), 5.72-5.60 (*m*, 1 H, CH=C*H*), 4.66 (*bd*, *J* = 6.2, 2 H, OCH_2), 3.50 (*s*, 2 H, C(=N)CH_2), 2.06 (*bq*, *J* = 7.0, 2 H, $CH_2CH_2CH_3$), 1.42 (*pseudo hex*, *J* = 7.3, 2 H, $CH_2CH_2CH_3$), 0.91 (*t*, *J* = 7.3, 3 H, $CH_2CH_2CH_3$). **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 161.2, 155.0, 143.5, 136.1, 134.9, 128.8, 128.6, 127.0, 125.0, 124.1, 121.8, 67.4, 36.9, 34.4, 22.2, 13.7. **IR ($CDCl_3$):** $\tilde{\nu}$ = 2958, 2930, 1665, 1590, 1511, 1455, 1340, 1312, 1236, 1168, 1109, 972, 863, 852, 757, 734, 700, 652, 582. **MS (ESI) *m/z*:** 410.2 (2%), 394.2 (6%), 378.2 (2%), 361.2 (25%, $[M+Na]^+$), 340.2 (8%), 257.1 (100%), 241.1 (4%), 139.1 (7%), 115.0 (2%), 91.1 (3%). **HRMS (ESI) *m/z*:** calculated for $C_{20}H_{22}N_2O_3$ + Na: 361.1523; found: 361.1537.

((*Z*)-Hex-2-en-1-yl)-*N*-(4-nitrophenyl)-2-phenylacetimidate ((*Z*)-9f)

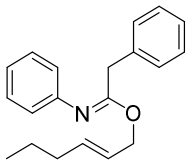


(*Z*)-9f

According to **GP2b**, (*Z*)-hex-2-en-1-ol **(*Z*)-1c** (1.21 mmol, 121.8 mg, 145 µL) was reacted with LHMDS (1.21 mmol, 1.21 mL, 1M in THF) and *(E)*-1-(1H-benzo[d][1,2,3]triazol-1-yl)-*N*-(4-nitrophenyl)-2-phenylethan-1-imine **8f** (1.20 mmol, 430.0 mg) at room temperature for 24 h. Purification by column chromatography on deactivated silicagel (petrol ether/ethyl acetate = 18/1 + 3% NEt_3) yielded **(*Z*)-9f** as a yellow oil (0.53 mmol, 180.1 mg, 44%).

$C_{20}H_{22}N_2O_3$, MW: 338.40 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 8.19-8.13 (*m*, 2 H, *m*-$C_6H_4NO_2$), 7.30-7.21 (*m*, 3 H, *m*-C_6H_5 & *p*-C_6H_5), 7.10-7.04 (*m*, 2 H, *o*-C_6H_5), 6.84-6.78 (*m*, 2 H, *o*-$C_6H_4NO_2$), 5.72-5.60 (*m*, 2 H, C*H*=C*H*), 4.80-4.74 (*m*, 2 H, OCH_2), 3.50 (*s*, 2 H, C(=N)CH_2), 2.13-2.04 (*m*, 2 H, $CH_2CH_2CH_3$), 1.40 (*pseudo hex*, *J* = 7.4, 2 H, $CH_2CH_2CH_3$), 0.90 (*t*, *J* = 7.4, 3 H, $CH_2CH_2CH_3$). **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 161.3, 154.9, 143.5, 135.0, 134.9, 128.7, 128.6, 127.0, 125.0, 123.7, 121.8, 62.7, 36.8, 29.7, 22.6, 13.7. **IR ($CDCl_3$):** $\tilde{\nu}$ = 2960, 2930, 1668, 1590, 1510, 1339, 1301, 1237, 1168, 1109, 996, 863, 852, 757, 700. **MS (ESI) *m/z*:** 455.3 (17%), 441.3 (40%), 413.3 (10%), 379.2 (2%), 361.2 (51%, $[M+Na]^+$), 340.2 (5%), 312.2 (17%), 282.3 (3%), 257.1 (100%), 241.2 (8%), 228.1 (23%), 139.1 (6%), 122.0 (2%). **HRMS (EI) *m/z*:** calculated for $C_{20}H_{22}N_2O_3$ + Na: 361.1523; found: 361.1521.

((*E*)-Hex-2-en-1-yl)-*N*-phenyl-2-phenylacetimidate (9g)

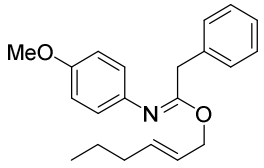


9g

According to **GP2d**, *N*-phenyl-2-phenylacetamide **6g** (5.00 mmol, 1.06 g) was reacted with PCl_5 (5.00 mmol, 1.04 g) and (*E*)-hex-2-en-1-ol **1c** (4.95 mmol, 495.6 mg, 590 µL) to yield **9g** after purification by column chromatography on deactivated silicagel (petrol ether/ethyl acetate = 20/1 + 3% NEt_3) as a colorless oil (3.40 mmol, 996.2 mg, 68%).

$C_{20}H_{23}NO$, MW: 293.40 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.32-7.19 (*m*, 5 H, *m*-$C_6H_5CH_2$ & *p*-$C_6H_5CH_2$ & *m*-C_6H_5N), 7.16-7.10 (*m*, 2 H, *o*-$C_6H_5CH_2$), 7.08-7.00 (*m*, 1 H, *p*-C_6H_5N), 6.79-6.72 (*m*, 2 H, *o*-C_6H_5N), 5.81-5.60 (*m*, 2 H, C*H*=C*H*), 4.65 (*bd*, *J* = 5.6, 2 H, OCH_2), 3.51 (*s*, 2 H, C(=N)CH_2), 2.04 (*bq*, *J* = 6.8, 2 H, $CH_2CH_2CH_3$), 1.42 (*pseudo hex*, *J* = 7.3, 2 H, $CH_2CH_2CH_3$), 0.91 (*t*, *J* = 7.3, 3 H, $CH_2CH_2CH_3$). **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 160.7, 148.6, 135.9, 135.2, 129.0, 128.9, 128.4, 126.5, 124.8, 122.9, 121.4, 66.6, 36.2, 34.4, 22.2, 13.7. **IR ($CDCl_3$):** $\tilde{\nu}$ = 3029, 2957, 2928, 2872, 1665, 1596, 1489, 1454, 1377, 1290, 1229, 1168, 1151, 1072, 971, 902, 761, 730, 697, 649. **MS (EI) *m/z*:** 293.2 (19%, $[M]^+$), 264.2 (29%), 251.2 (15%), 211.1 (20%),294.1 (21%), 175.1 (21%), 132.1 (18%), 119.0 (23%), 93.1 (100%), 83.1 (12%), 55.1 (28%). **HRMS (EI) *m/z*:** calculated for $C_{20}H_{23}NO$: 293.1780; found: 293.1782.

((*E*)-Hex-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate (9h)



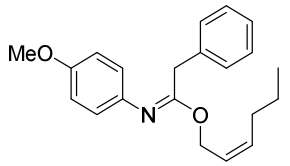
9h

According to **GP2c**, *N*-(4-methoxyphenyl)-2-phenylacetamide **6h** (1.99 mmol, 480.0 mg) was reacted with oxalyl chloride (2.04 mmol, 259.0 mg, 175 µL) and (*E*)-hex-2-en-1-ol **1c** (1.00 mmol, 99.1 mg, 118 µL) to yield **9h** after purification by dry column chromatography on deactivated silicagel (petrolether + 3% NEt_3) as a colorless oil (0.50 mmol, 161.8 mg, 50%).

$C_{21}H_{25}NO_2$, MW: 323.44 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.29-7.18 (*m*, 3 H, *m*-C_6H_5 & *p*-C_6H_5), 7.15-7.10 (*m*, 2 H, *o*-C_6H_5), 6.86-6.79 (*m*, 2 H, *o*-C_6H_4OMe), 6.71-6.64 (*m*, 2 H, *m*-C_6H_4OMe), 5.80-5.59 (*m*, 2 H, C*H*=C*H*), 4.64 (*bd*, *J* = 5.5, 2 H, OCH_2), 3.79 (*s*, 3 H, OCH_3), 3.52 (*s*, 2 H, OCH_2),

2.04 (*bq*, *J* = 6.9, 2 H, CH_2CH$_2$CH$_3$), 1.41 (*pseudo hex*, *J* = 7.4, 2 H, CH$_2$CH_2CH$_3$), 0.91 (*t*, *J* = 7.4, 3 H, CH$_2$CH$_2$CH_3). **^{13}C-NMR (125 MHz, CDCl$_3$):** δ = 161.4, 155.6, 141.9, 136.1, 135.1, 128.9, 128.4, 126.5, 124.9, 122.3, 114.3, 66.5, 55.5, 36.1., 34.3, 22.2, 13.7. **IR (CDCl$_3$):** $\tilde{\nu}$ = 2956, 2929, 2871, 1663, 1603, 1504, 1454, 1440, 1376, 1288, 1228, 1163, 1102, 1037, 971, 840, 734, 715, 697. **MS (ESI) *m/z*:** 346.2 (25%, [M+Na]$^+$), 324.2 (3%, [M+H]$^+$), 242.1 (100%), 124.1 (2%). **HRMS (ESI) *m/z*:** calculated for $C_{21}H_{25}NO_2$ + Na: 346.1778; found: 346.1768.

((*Z*)-Hex-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate ((*Z*)-9h)

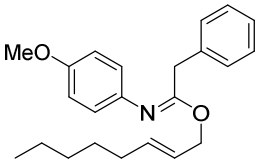


(*Z*)-9h

According to **GP2c**, *N*-(4-methoxyphenyl)-2-phenylacetamide **6h** (0.50 mmol, 120.0 mg) was reacted with oxalyl chloride (0.52 mmol, 65.7 mg, 45 µL) and (*Z*)-hex-2-en-1-ol **(*Z*)-1c** (0.40 mmol, 40.3 mg, 48 µL) to yield **(*Z*)-9h** after purification by column chromatography on deactivated silicagel (petrol ether/ethyl acetate = 30/1 + 3% NEt$_3$) as a colorless oil (0.23 mmol, 73.0 mg, 57%).

C$_{21}$H$_{25}$NO$_2$, MW: 323.44 g mol^{-1}. **^{1}H-NMR (300 MHz, CDCl$_3$):** δ = 7.30-7.18 (*m*, 3 H, *m*-C$_6H_5$ & *p*-C$_6H_5$), 7.15-7.09 (*m*, 2 H, *o*-C$_6H_5$), 6.86-6.79 (*m*, 2 H, *o*-C$_6H_4$OMe), 6.72-6.64 (*m*, 2 H, *m*-C$_6H_4$OMe), 5.71-5.55 (*m*, 2 H, C*H*=C*H*), 4.77-4.72 (*m*, 2 H, OCH_2), 3.79 (*s*, 3 H, OCH_3), 3.52 (*s*, 2 H, C(=N)CH_2), 2.07 (*bq*, *J* = 7.0, 2 H, CH_2CH$_2$CH$_3$), 1.38 (*pseudo hex*, *J* = 7.3, 2 H, CH$_2$CH_2CH$_3$), 0.89 (*t*, *J* = 7.3, 3 H, CH$_2$CH$_2$CH_3). **^{13}C-NMR (125 MHz, CDCl$_3$):** δ = 161.5, 155.6, 141.8, 136.0, 134.2, 128.9, 128.4, 126.5, 124.5, 122.2, 114.3, 62.0, 55.5, 36.1, 29.7, 22.7, 13.7. **IR (CDCl$_3$):** $\tilde{\nu}$ = 3001, 2960, 2929, 2872, 1658, 1599, 1505, 1494, 1453, 1424, 1292, 1271, 1225, 1178, 1162, 1108, 1029, 990, 970, 919, 898, 854, 815. **MS (ESI) *m/z*:** 346.2 (3%, [M+Na]$^+$), 324.2 (5%, [M+H]$^+$), 242.1 (100%), 124.1 (1%). **HRMS (ESI) *m/z*:** calculated for $C_{21}H_{25}NO_2$ + H: 324.1958; found: 324.1964.

((*E*)-Oct-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate (9i)



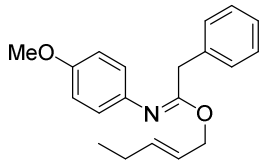
9i

According to **GP2c**, *N*-(4-methoxyphenyl)-2-phenylacetamide **6h** (1.99 mmol, 480.0 mg) was reacted with oxalyl chloride (2.04 mmol, 259.0 mg, 175 µL) and (*E*)-oct-2-en-1-ol **1d** (0.98 mmol, 126.0 mg,

150 µL) to yield **9i** after purification by dry column chromatography on deactivated silicagel (petrol ether + 1% NEt_3) as a slightly yellow oil (0.74 mmol, 260.4 mg, 76%).

$C_{23}H_{29}NO_2$, MW: 351.49 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.30-7.18 (*m*, 3 H, *m*-C_6H_5 & *p*-C_6H_5), 7.15-7.09 (*m*, 2 H, *o*-C_6H_5), 6.86-6.79 (*m*, 2 H, *o*-C_6H_4OMe), 6.70-6.64 (*m*, 2 H, *m*-C_6H_4OMe), 5.80-5.58 (*m*, 2 H, C*H*=C*H*), 4.63 (*d*, *J* = 5.7, 2 H, OCH_2), 3.78 (*s*, 3 H, OCH_3), 3.52 (*s*, 2 H, C(=N)CH_2), 2.05 (*bq*, *J* = 6.9, 2 H, CH_2(CH_2)$_3$$CH_3$), 1.43-1.23 (*m*, 6 H, CH_2(CH_2)$_3$$CH_3$), 0.89 (*t*, *J* = 6.9, 3 H, CH_2(CH_2)$_3$CH_3). **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 161.4, 155.6, 141.9, 136.1, 135.4, 128.9, 128.4, 126.5, 124.6, 122.2, 114.3, 66.6, 55.5, 36.1, 32.3, 31.4, 28.7, 22.6, 14.1. **IR ($CDCl_3$):** $\tilde{\nu}$ = 3030, 2954, 2926, 2856, 1663, 1603, 1504, 1455, 1440, 1376, 1288, 1228, 1163, 1151, 1102, 1038, 974, 840, 817, 774, 734, 715, 697, 676, 625, 544. **MS (ESI) *m/z*:** 374.2 (21%, [M+Na]$^+$), 352.2 (9%, [M+H]$^+$), 242.1 (100%), 124.1 (2%). **HRMS (ESI) *m/z*:** calculated for $C_{23}H_{29}NO_2$ + Na: 374.2091; found: 374.2073.

((*E*)-Pent-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate (9j)

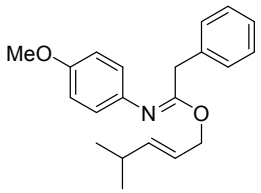


9j

According to **GP2c**, *N*-(4-methoxyphenyl)-2-phenylacetamide **6h** (1.99 mmol, 480.0 mg) was reacted with oxalyl chloride (2.04 mmol, 259.0 mg, 175 µL) and (*E*)-pent-2-en-1-ol **1b** (1.48 mmol, 127.5 mg, 150 µL) to yield **9j** after purification by dry column chromatography on deactivated silicagel (petrol ether + 1% NEt_3) as a slightly yellow oil (0.72 mmol, 224.2 mg, 49%).

$C_{20}H_{23}NO_2$, MW: 309.41 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.30-7.18 (*m*, 3 H, *m*-C_6H_5 & *p*-C_6H_5), 7.15-7.09 (*m*, 2 H, *o*-C_6H_5), 6.86-6.79 (*m*, 2 H, *o*-C_6H_4OMe), 6.70-6.63 (*m*, 2 H, *m*-C_6H_4OMe), 5.85-5.73 (*m*, 1 H, C*H*=CH), 5.70-5.58 (*m*, 1 H, CH=C*H*), 4.64 (*dd*, *J* = 6.0, 1.0, 2 H, OCH_2), 3.78 (*s*, 3 H, OCH_3), 3.52 (*s*, 2 H, C(=N)CH_2), 2.14-2.01 (*m*, 2 H, C$H_2$$CH_3$), 1.00 (*t*, *J* = 7.4, 3 H, CH_2CH_3). **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 161.4, 155.6, 141.8, 136.8, 136.0, 128.9, 128.4, 126.5, 123.7, 122.2, 114.3, 66.6, 55.5, 36.1, 25.4, 13.3. **IR ($CDCl_3$):** $\tilde{\nu}$ = 3030, 2961, 2833, 1662, 1603, 1505, 1455, 1440, 1375, 1289, 1227, 1163, 1102, 1037, 994, 969, 841, 775, 734, 715, 698, 625, 545. **MS (ESI) *m/z*:** 332.2 (56%, [M+Na]$^+$), 310.2 (2%, [M+H]$^+$), 242.1 (100%), 124.1 (2%). **HRMS (ESI) *m/z*:** calculated for $C_{20}H_{23}NO_2$ + Na: 332.1621; found: 332.1614.

((*E*)-4-Methylpent-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate (9k)

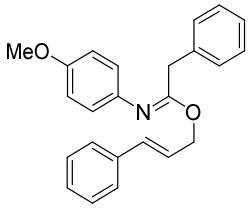


9k

According to **GP2c**, *N*-(4-methoxyphenyl)-2-phenylacetamide **6h** (0.99 mmol, 240.0 mg) was reacted with oxalyl chloride (1.05 mmol, 133.2 mg, 90 µL) und (*E*)-4-methylpent-2-en-1-ol **1k** (0.50 mmol, 55 mg of a 92% solution in CH_2Cl_2) to yield **9k** after purification by dry column chromatography on deactivated silicagel (petrol ether + 3% NEt_3) as a yellow oil (0.25 mmol, 81.7 mg, 50%).

$C_{21}H_{25}NO_2$, MW: 323.44 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.31-7.17 (*m*, 3 H, *m*-C_6H_5 & *p*-C_6H_5), 7.16-7.10 (*m*, 2 H, *o*-C_6H_5), 6.86-6.79 (*m*, 2 H, *o*-C_6H_4OMe), 6.71-6.64 (*m*, 2 H, *m*-C_6H_4OMe), 5.76-5.54 (*m*, 2 H, C*H*=C*H*), 4.64 (*bd*, *J* = 5.8, 2 H, OCH_2), 3.79 (*s*, 3 H, OCH_3), 3.52 (*s*, 2 H, C(=N)CH_2), 2.31 (*pseudo hex*, *J* = 6.7, 1 H, C*H*(CH_3)$_2$), 1.00 (*d*, *J* = 6.7, 6 H, CH(CH_3)$_2$). **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 161.4, 155.6, 141.92, 141.86, 136.1, 128.9, 128.4, 126.5, 122.2, 121.7, 114.3, 66.6, 55.5, 36.1, 30.9, 22.2. **IR ($CDCl_3$):** $\tilde{\nu}$ = 2957, 2833, 1662, 1603, 1504, 1455, 1381, 1290, 1227, 1163, 1103, 973, 840, 774, 734, 715, 698, 627, 545. **MS (ESI) *m/z*:** 346.2 (44%, [M+Na]$^+$), 324.2 (1%, [M+H]$^+$), 242.1 (100%), 124.1 (2%). **HRMS (ESI) *m/z*:** calculated for $C_{21}H_{25}NO_2$ + Na: 346.1778; found: 346.1776.

((*E*)-3-Phenylprop-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate (9l)



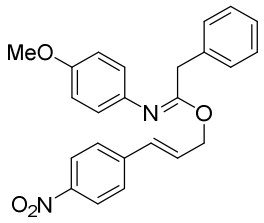
9l

According to **GP2c**, *N*-(4-methoxyphenyl)-2-phenylacetamide **6h** (0.99 mmol, 240.0 mg) was reacted with oxalyl chloride (1.05 mmol, 133.2 mg, 90 µL) and (*E*)-3-phenylprop-2-en-1-ol **1l** (0.50 mmol, 67.0 mg) to yield **9l** after purification by dry column chromatography on deactivated silicagel (petrol ether + 3% NEt_3) as a white solid (0.35 mmol, 126.8 mg, 70%).

$C_{24}H_{23}NO_2$, MW: 357.45 g mol^{-1}. **Mp:** 60.2-61.0 °C. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.40-7.20 (*m* 8 H, *m*-$C_6H_5CH_2$ & *p*-$C_6H_5CH_2$ & C_6H_5CH), 7.18-7.12 (*m*, 2 H, *o*-$C_6H_5CH_2$), 6.87-6.81 (*m*, 2 H, *o*-C_6H_4OMe), 6.73-6.67 (*m*, 2 H, *m*-C_6H_4OMe), 5.69 (*bd*, *J* = 16.0, 1 H, C_6H_5C*H*=CH), 6.37 (*dt*,

J = 16.0, 5.8, 1 H, C_6H_5CH=C*H*), 4.86 (*dd*, *J* = 5.8, 1.5, 2 H, OCH_2), 3.79 (*s*, 3 H, OCH_3), 3.55 (*s*, 2 H, C(=N)CH_2). **^{13}C-NMR (75 MHz, $CDCl_3$):** δ = 161.2, 155.7, 141.7, 136.7, 135.9, 132.7, 129.0, 128.5, 127.8, 126.6, 124.4, 122.2, 114.3, 66.2, 55.5, 36.1. **IR ($CDCl_3$):** $\tilde{\nu}$ = 3028, 2932, 2832, 1662, 1601, 1504, 1453, 1288, 1223, 1163, 1103, 1070, 1033, 995, 965, 839, 808, 774, 734, 715, 693, 606, 547. **MS (ESI) *m/z*:** 380.2 (49%, $[M+Na]^+$, 358.2 (11%, $[M+H]^+$), 339.1 (3%), 301.1 (12%), 263.1 (6%), 242.1 (11%), 203.1 (1%), 172.0 (3%), 129.1 (1%), 117.1 (100%). **HRMS (ESI) *m/z*:** calculated for $C_{24}H_{23}NO_2$ + Na: 380.1621; found: 380.1621.

((*E*)-3-(4-Nitrophenylprop-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate (9m)

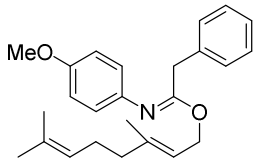


9m

According to **GP2c**, *N*-(4-methoxyphenyl)-2-phenylacetamide **6h** (0.99 mmol, 240.0 mg) was reacted with oxalyl chloride (1.05 mmol, 133.2 mg, 90 µL) und (*E*)-3-(4-nitrophenyl)prop-2-en-1-ol **1m** (0.50 mmol, 89.6 mg) to yield **9m** after purification by dry column chromatography on deactivated silicagel (petrol ether + 5% NEt_3) as a yellow solid (0.11 mmol, 43.4 mg, 22%).

$C_{24}H_{22}N_2O_4$, MW: 402.45 g mol^{-1}. **Mp:** 106.4-107.4 °C. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 8.21-8.13 (*m*, 2 H, *m*-$C_6H_4NO_2$), 7.48-7.42 (*m*, 2 H, *o*-$C_6H_4NO_2$), 7.33-7.21 (*m*, 3 H, *m*-C_6H_5 & *p*-C_6H_5), 7.19-7.13 (*m*, 2 H, *o*-C_6H_5), 6.88-6.82 (*m*, 2 H, *o*-C_6H_4OMe), 6.74-6.68 (*m*, 2 H, *m*-C_6H_4OMe), 6.62-6.46 (*m*, 2 H, C*H*=C*H*), 4.91 (*d*, *J* = 4.1, 2 H, OCH_2), 3.79 (*s*, 3 H, OCH_3), 3.58 (*s*, 2 H, C(=N)CH_2). **^{13}C-NMR (75 MHz, $CDCl_3$):** δ = 160.9, 155.8, 147.0, 143.2, 141.3, 135.8, 129.7, 129.6, 129.0, 128.5, 127.0, 126.7, 124.0, 122.1, 114.4, 65.3, 55.5, 36.0. **IR ($CDCl_3$):** $\tilde{\nu}$ = 3031, 2933, 2834, 1666, 1597, 1504, 1454, 1340, 1290, 1225, 1163, 1108, 1076, 1033, 1005, 969, 860, 842, 775, 736, 716, 699, 625, 545. **MS (ESI) *m/z*:** 425.1 (73%, $[M+Na]^+$), 403.2 (100%, $[M+H]^+$). **HRMS (ESI) *m/z*:** calculated for $C_{24}H_{22}N_2O_4$ + Na: 425.1472; found: 425.1471.

((*E*)-3,7-Dimethylocta-2,6-dien-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate (9n)

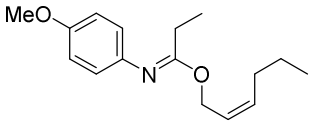


9n

According to **GP2c**, *N*-(4-methoxyphenyl)-2-phenylacetamide **6h** (0.99 mmol, 240.0 mg) was reacted with oxalyl chloride (1.05 mmol, 133.2 mg, 90 µL) and geraniol **1n** (0.50 mmol, 77.0 mg) to yield **9n** after purification by dry column chromatography on deactivated silicagel (petrol ether + 3% NEt_3) as a colorless oil (0.28 mmol, 104.3 mg, 56%).

$C_{25}H_{31}NO_2$, MW: 377.53 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.29-7.16 (*m*, 3 H, *m*-C_6H_5 & *p*-C_6H_5), 7.15-7.09 (*m* 2 H, *o*-C_6H_5), 6.85-6.79 (*m*, 2 H, *o*-C_6H_4OMe), 6.71-6.65 (*m*, 2 H, *m*-C_6H_4OMe), 5.48-5.40 (*m*, 1 H, OCH_2CH), 5.14-5.06 (*m*, 1 H, CH_2CH_2CH), 4.71 (*d*, *J* = 7.0, 2 H, OCH_2), 3.79 (*s*, 3 H, OCH_3), 3.51 (*s*, 2 H, $C(=N)CH_2$), 2.17-2.00 (*m*, 4 H, CH_2CH_2), 1.68 (*s*, 6 H, CH_3), 1.61 (*s*, 3 H, CH_3). **^{13}C-NMR (75 MHz, $CDCl_3$):** δ = 161.6, 155.6, 141.9, 141.0, 136.1, 131.7, 128.8, 128.4, 126.4, 123.9, 122.2, 119.3, 114.2, 62.9, 55.5, 39.6, 36.1, 26.4, 25.7, 17.7, 16.6. **IR ($CDCl_3$):** $\tilde{\nu}$ = 3031, 2914, 1663, 1603, 1505, 1454, 1377, 1290, 1229, 1163, 1103, 1038, 988, 839, 775, 734, 715, 698, 676, 625, 536. **MS (ESI) *m/z*:** 400.2 (20%, $[M+Na]^+$), 288.9 (1%), 264.1 (23%), 226.9 (6%), 159.0 (1%), 124.1 (6%). **HRMS (ESI) *m/z*:** calculated for $C_{25}H_{31}NO_2$ + Na: 400.2247; found: 400.2258.

((*Z*)-Hex-2-en-1-yl)-*N*-(4-methoxyphenyl)propionimidate ((*Z*)-9o)



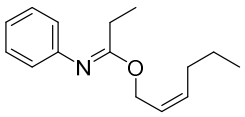
(*Z*)-9o

According to **GP2d**, *N*-(4-methoxyphenyl)propionamide **6o** (1.00 mmol, 179.2 mg) was reacted with PCl_5 (1.00 mmol, 208.0 mg) and (*Z*)-hex-2-en-1-ol **(*Z*)-1c** (0.98 mmol, 97.8 mg, 115 µL) to yield **(*Z*)-9o** after purification by column chromatography on deactivated silicagel (petrol ether/ethyl acetate = 20/1 + 3% NEt_3) as a slightly yellow oil (0.51 mmol, 132.0 mg, 52%).

$C_{16}H_{23}NO_2$, MW: 261.37 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 6.86-6.80 (*m*, 2 H, *o*-C_6H_4OMe), 6.71-6.65 (*m*¸2 H, *m*-C_6H_4OMe), 5.73-5.58 (*m*, 2 H, $CH=CH$), 4.71 (*bd*, *J* = 5.5, 2 H, OCH_2), 3.78 (*s*, 3 H, OCH_3), 2.22-2.07 (*m*, 4 H, $C(=N)CH_2CH_3$ & $CH_2CH_2CH_3$), 1.41 (*pseudo hex*, *J* = 7.3, 2 H, $CH_2CH_2CH_3$), 1.07 (*t*, *J* = 7.6, 3 H, $C(=N)CH_2CH_3$), 0.91 (*t*, *J* = 7.3, 3 H, $CH_2CH_2CH_3$). **^{13}C-NMR**

(125 MHz, $CDCl_3$): δ = 165.0, 155.4, 142.2, 134.1, 124.7, 122.0, 114.2, 61.6, 55.5, 29.7, 23.2, 22.7, 13.7, 11.2. **IR ($CDCl_3$):** $\tilde{\nu}$ = 2959, 2934, 2874, 1666, 1505, 1463, 1293, 1226, 1180, 1082, 1070, 1038, 1013, 911, 838, 734, 611. **MS (ESI) *m/z*:** 304.3 (1%), 284.2 (13%, $[M+Na]^+$), 262.2 (12%, $[M+H]^+$), 202.1 (1%), 180.1 (100%), 162.1 (1%), 124.1 (16%). **HRMS (ESI) *m/z*:** calculated for $C_{16}H_{23}NO_2$ + H: 262.1802; found: 262.1803.

((*Z*)-Hex-2-en-1-yl)-*N*-phenylpropionimidate ((*Z*)-9p)

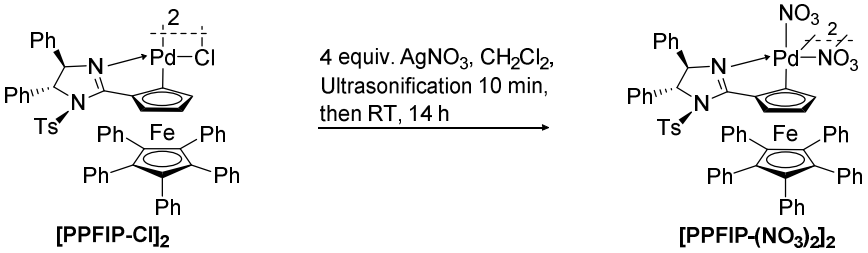


(*Z*)-9p

According to **GP2d**, *N*-phenylpropionamide **6p** (1.00 mmol, 149.2 mg) was reacted with PCl_5 (1.00 mmol, 208.0 mg) and (*Z*)-hex-2-en-1-ol **(*Z*)-1c** (0.98 mmol, 97.8 mg, 115 µL) to yield **(*Z*)-9p** after purification by column chromatography on deactivated silicagel (petrol ether/ethyl acetate = 20/1 + 3% NEt_3) as a colorless oil (0.48 mmol, 111.6 mg, 49%).

$C_{15}H_{21}NO$, MW: 231.33 g mol^{-1}. **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.31-7.23 (*m*, 2 H, *m*-C_6H_5), 7.05-6.99 (*m*, 1 H, *p*-C_6H_5), 6.78-6.73 (*m*, 2 H, *o*-C_6H_5), 5.74-5.59 (*m*, 2 H, C*H*=C*H*), 4.72 (*bd*, *J* = 5.3, 2 H, OCH_2), 2.22-2.08 (*m*, 4 H, C(=N)CH_2CH_3 & $CH_2CH_2CH_3$), 1.42 (*pseudo hex*, *J* = 7.4, 2 H, $CH_2CH_2CH_3$), 1.08 (*t*, *J* = 7.5, 3 H, C(=N)CH_2CH_3), 0.92 (*t*, *J* = 7.4, 3 H, $CH_2CH_2CH_3$). The analytical data are in accordance with the literature.[23]

Activation of [PPFIP-Cl]$_2$ by $AgNO_3$



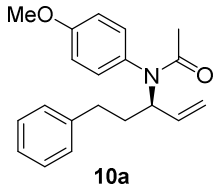
Acoording to GP3a, **[PPFIP-Cl]$_2$** (5.0 µmol ,10.8 mg) was reacted with $AgNO_3$ (20.0 µmol, 3.4 mg) to yield **[PPFIP-$(NO_3)_2$]$_2$** as a brown paramagnetic solid (5.0 µmol, 11.7 mg, quant.).

$C_{124}H_{94}Fe_2N_8O_{16}Pd_2S_2$, MW: 2340.81 g mol^{-1}. **Mp:** >200 °C (decomposition). **$[\alpha]_D^{20}$:** –908 (*c* = 0.05, CH_2Cl_2). **IR (solid):** $\tilde{\nu}$ = 3056, 1577, 1443, 1378, 1266, 1169, 1077, 1025, 986, 968, 843, 800, 781, 737, 693, 667, 619, 594, 541, 508. **MS (ESI) *m/z*:** 2154.4 (1%, $[M-(NO_3)_3]^+$), 1045.2 (100%,

[ligand+Pd]$^+$). **Microanalysis:** Calculated for $C_{124}H_{94}Fe_2N_8O_{16}Pd_2S_2$ + 2 THF: C: 63.80; H: 4.46; N: 4.51; S: 2.58; found: C: 63.72; H: 4.31; N: 4.21; S: 2.65.

Synthesis of Allylic Amides (10)

(*R*)-*N*-(4-Methoxyphenyl)-*N*-(5-phenylpent-1-en-3-yl)acetamide (10a)



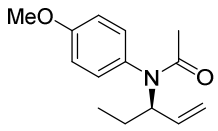
10a

According to **GP4b**, (*E*)-((*E*)-5-phenylpent-2-en-1-yl)-*N*-(4-methoxyphenyl)acetimidate **9a** (64.4 µmol, 20.0 mg) was reacted with proton sponge (4 mol%, 2.5 µmol, 50 µL of a stock solution with c = 50.0 µmol/mL in $CHCl_3$) and the activated catalyst **[PPFIP-$(NO_3)_2$]$_2$** (1 mol%, 0.6 µmol, 100 µL of a stock solution with c = 6.0 µmol/mL in $CHCl_3$, prepared according to **GP3a**) in $CHCl_3$ at 70 °C for 24 h to yield **10a** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 4/1) as a colorless oil (59.8 µmol, 18.5 mg, 93%, *ee* = 98%). The enantiomeric excess was determined by HPLC: *Chiracel AD-H*, *n*-hexane/*i*-PrOH (98/2), 1.0 mL min^{-1}, 250 nm, 25.34 min (minor enantiomer), 34.04 min (major enantiomer).

Alternatively, (*E*)-((*E*)-5-phenylpent-2-en-1-yl)-*N*-(4-methoxyphenyl)acetimidate **9a** (64.4 µmol, 20.0 mg) was reacted according to **GP4b** with the activated catalyst **[FBIP-OTs-MeCN]** (3 mol%, 1.9 µmol, 160 µL of a stock solution with c = 12.0 µmol/mL in $CHCl_3$, prepared according to **GP3b**) in $CHCl_3$ at room temperature for 72 h to yield **10a** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 4/1) as a colorless oil (59.8 µmol, 18.5 mg, 93%, *ee* = 89%).

$C_{20}H_{23}NO_2$, MW: 309.41 g mol^{-1}. **$[\alpha]_D^{20}$:** +37.1 (c = 0.41, $CHCl_3$, sample with *ee* = 98%). **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.30-7.23 (*m*, 2 H, *m*-C_6H_5), 7.20-7.13 (*m*, 3 H, *o*-C_6H_5 & *p*-C_6H_5), 7.06-7.00 (*m,* 2 H, *o*-C_6H_4OMe), 6.91-6.86 (*m*, 2 H, *m*-C_6H_4OMe), 5.64 (*ddd*, J = 17.3, J = 10.2, J = 7.9, 1 H, C*H*=CH_2), 5.32-5.14 (*m*, 3 H, C*H*CH=CH_2), 3.82 (*s*, 3 H, OCH_3), 2.73-2.56 (*m*, 2 H, $C_6H_5CH_2CH_2$), 1.91-1.81 (*m*, 1 H, $C_6H_5CH_2CH_2$), 1.78 (*s*, 3 H, C(=O)CH_3), 1.75-1.65 (*m*, 1 H, $C_6H_5CH_2CH_2$). **^{13}C-NMR (75 MHz, $CDCl_3$):** δ = 170.8, 159.3, 141.9, 137.1, 132.6, 131.1, 128.49, 128.46, 126.0, 118.1, 114.4, 57.4, 55.5, 34.2, 33.0, 23.5. **IR ($CDCl_3$):** $\tilde{\nu}$ = 3026, 2934, 2839, 2365, 2099, 1890, 1651, 1508, 1454, 1383, 1312, 1292, 1245, 1170, 1106, 1031, 997, 927, 839, 753, 700, 593. **MS (ESI) *m/z:*** 332.1 (100%, [M+Na]$^+$), 310.2 (73%, [M+H]$^+$). **HRMS (ESI) *m/z*:** calculated for $C_{20}H_{23}NO_2$ + H: 310.1802; found: 310.1804.

(*R*)-*N*-(4-Methoxyphenyl)-*N*-(pent-1-en-3-yl)acetamide (10b)

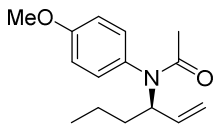


10b

According to **GP4b**, (*E*)-((*E*)-pent-2-en-1-yl)-*N*-(4-methoxyphenyl)acetimidate **9b** (85.7 µmol, 20.0 mg) was reacted with proton sponge (4 mol%, 3.5 µmol, 70 µL of a stock solution with c = 50.0 µmol/mL in $CHCl_3$) and the activated catalyst **[PPFIP-$(NO_3)_2$]$_2$** (1 mol%, 0.8 µmol, 130 of a stock solution with c = 6.0 µmol/mL in $CHCl_3$, prepared according to **GP3a**) in $CHCl_3$ at 70 °C for 24 h to yield **10b** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 4/1) as a colorless oil (78.8 µmol, 18.4 mg, 92%, *ee* = 96%). The enantiomeric excess was determined by HPLC: *Chiracel AD-H*, *n*-hexane/*i*-PrOH (99.5/0.5), 2.0 mL min^{-1}, 250 nm, 21.98 min (major enantiomer), 28.96 min (minor enantiomer).

$C_{14}H_{19}NO_2$, MW: 233.31 g mol^{-1}. **$[\alpha]_D^{20}$:** +5.5 (c = 0.33, $CHCl_3$, sample with *ee* = 96%). **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.05-6.97 (*m*, 2 H, *o*-C_6H_4OMe), 6.91-6.85 (*m*, 2 H, *m*-C_6H_4OMe), 5.65-5.50 (*m*, 1 H, CHC*H*=CH_2), 5.22-5.03 (*m*, 3 H, C*H*CH=CH_2), 3.82 (*s*, 3 H, OCH_3), 1.76 (*s*, 3 H, C(=O)CH_3), 1.62-1.34 (*m*, 2 H, CH_2CH$_3$), 0.92 (*t*, J = 7.4, 3 H, CH_2CH_3). **^{13}C-NMR (75 MHz, $CDCl_3$):** δ = 170.6, 159.1, 137.1, 132.7, 131.0, 117.6, 114.2, 59.0, 55.4, 25.1, 23.3, 11.0. **IR ($CDCl_3$):** $\tilde{\nu}$ = 2965, 2934, 1654, 1509, 1460, 1443, 1384, 1316, 1291, 1247, 1170, 1106, 1075, 1032, 998, 967, 926, 839, 810, 791, 756, 647, 588. **MS (ESI) *m/z:*** 256.1 (100%, $[M+Na]^+$), 234.2 (1%, $[M+H]^+$). **HRMS (ESI) *m/z*:** calculated for $C_{14}H_{19}NO_2$ + Na: 256.1308; found: 256.1295.

(*R*)-*N*-(Hex-1-en-3-yl)-*N*-(4-methoxyphenyl)acetamide (10c)



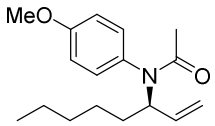
10c

According to **GP4b**, (*E*)-((*E*)-hex-2-en-1-yl)-*N*-(4-methoxyphenyl)acetimidate **9c** (80.8 µmol, 20 mg) was reacted with proton sponge (4 mol%, 3.2 µmol, 0.7 mg, 65 µL of a stock solution c = 50.0 µmol/mL in $CHCl_3$) and the activated catalyst **[PPFIP-$(NO_3)_2$]$_2$** (1 mol%, 0.8 µmol, 130 of a stock solution with c = 6.0 µmol/mL in $CHCl_3$, prepared according to **GP3a**) in $CHCl_3$ at 70 °C for 24 h to yield **10c** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 5/1) as a colorless oil (74.4 µmol, 18.4 mg, 92%, *ee* = 96%). The enantiomeric excess was

determined by HPLC: *Chiracel AD-H*, *n*-hexane/*i*-PrOH (99.2/0.8), 1.0 mL min^{-1}, 250 nm, 31.13 min (minor enantiomer), 34.44 min (major enantiomer).

$C_{15}H_{21}NO_2$, MW: 247.34 g mol^{-1}. **$[\alpha]_D^{20}$:** −12.2 (*c* = 0.39, $CHCl_3$, sample with *ee* = 96%).**^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.04-6.97 (*m*, 2 H, *o*-C_6H_4OMe), 6.92-6.85 (*m*, 2 H, *m*-C_6H_4OMe), 5.66-5.52 (*m*, 1 H, CHC*H*=CH_2), 5.24-5.06 (*m*, 3 H, C*H*CH=CH_2), 3.83 (*s*, 3 H, OCH_3), 1.75 (*s*, 3 H, C(=O)CH_3), 1.55-1.27 (*m*, 4 H, CH_2C$H_2$$CH_3$), 0.91 (*t*, *J* = 7.1, 3 H, $CH_2CH_2CH_3$). **^{13}C-NMR (63 MHz, $CDCl_3$):** δ = 170.6, 159.1, 137.4, 132.6, 131.0, 117.4, 114.2, 57.1, 55.4, 34.2, 23.4, 19.6, 14.0. **IR ($CDCl_3$):** $\tilde{\nu}$ = 2957, 2932, 1654, 1509, 1463, 1383, 1314, 1293, 1247, 1170, 1106, 1033, 925, 837, 758, 590. **MS (ESI) *m/z:*** 270.1 (100%, $[M+Na]^+$), 248.2 (1%, $[M+H]^+$). **HRMS (ESI) *m/z*:** calculated for $C_{15}H_{21}NO_2$ + Na: 270.1465; found: 270.1446.

(*R*)-*N*-(4-Methoxyphenyl)-*N*-(oct-1-en-3-yl)acetamide (10d)

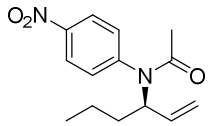


10d

According to **GP4b**, (*E*)-((*E*)-oct-2-en-1-yl)-*N*-(4-methoxyphenyl)acetimidate **9d** (72.6 µmol, 20.0 mg) was reacted with proton sponge (4 mol%, 3.0 µmol, 60 µL of a stock solution with *c* = 50.0 µmol/mL in $CHCl_3$) and the activated catalyst **[PPFIP-$(NO_3)_2$]$_2$** (1 mol%, 0.7 µmol, 120 µL of a stock solution with *c* = 6.0 µmol/mL in $CHCl_3$, prepared according to **GP3a**) in $CHCl_3$ at 70 °C for 24 h to yield **10d** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 6/1) as a colorless oil (66.1 µmol, 18.2 mg, 91%, *ee* = 92%). The enantiomeric excess was determined by HPLC: *Chiracel AD-H*, *n*-hexane/*i*-PrOH (99/1), 1.0 mL min^{-1}, 250 nm, 25.33 min (minor enantiomer), 29.71 min (major enantiomer).

$C_{17}H_{25}NO_2$, MW: 275.39 g mol^{-1}. **$[\alpha]_D^{20}$:** −20.5 (*c* = 0.41, $CHCl_3$, sample with *ee* = 92%).**^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.04-6.96 (*m*, 2 H, *o*-C_6H_4OMe), 6.92-6.85 (*m*, 2 H, *m*-C_6H_4OMe), 5.66-5.52 (*m*, 1 H, CHC*H*=CH_2), 5.22-5.07 (*m*, 3 H, C*H*CH=CH_2), 3.83 (*s*, 3 H, OCH_3), 1.76 (*s*, 3 H, C(=O)CH_3), 1.55-1.18 (*m*, 8 H, $(CH_2)_4CH_3$), 0.87 (*t*, *J* = 6.8, 3 H, $(CH_2)_4CH_3$). **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 170.6, 159.1, 137.5, 132.7, 131.1, 117.4, 114.2, 57.4, 55.4, 32.1, 31.8, 26.1, 23.4, 22.6, 14.1. **IR ($CDCl_3$):** $\tilde{\nu}$ = 2954, 2929, 2857, 1654, 1508, 1464, 1442, 1383, 1292, 1245, 1169, 1106, 1032, 997, 924, 836, 807, 756, 645, 590. **MS (ESI) *m/z:*** 298.2 (100%, $[M+Na]^+$), 276.2 (2%, $[M+H]^+$). **HRMS (ESI) *m/z*:** calculated for $C_{17}H_{25}NO_2$ + Na: 298.1778; found: 298.1766.

(*R*)-*N*-(Hex-1-en-3-yl)-*N*-(4-nitrophenyl)acetamide (10e)



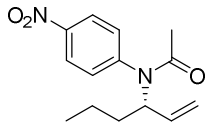
10e

According to **GP4b**, (*E*)-((*E*)-hex-2-en-1-yl)-*N*-(4-nitrophenyl)acetimidate **9e** (76.2 µmol, 20.0 mg) was reacted with proton sponge (4 mol%, 3.0 µmol, 60 µL of a stock solution with *c* = 50.0 µmol/mL in $CHCl_3$) and the activated catalyst **[PPFIP-$(NO_3)_2$]$_2$** (1 mol%, 0.7 µmol, 120 µL of a stock solution with *c* = 6.0 µmol/mL in $CHCl_3$, prepared according to **GP3a**) in $CHCl_3$ at 70 °C for 24 h to yield **10e** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 4/1) as a white solid (71.6 µmol, 18.8 mg, 94%, *ee* = 94%). The enantiomeric excess was determined by HPLC: *Chiracel AS-H*, *n*-hexane/*i*-PrOH (96/4), 1.0 mL min^{-1}, 250 nm, 43.07 min (minor enantiomer), 52.51 min (major enantiomer).

Alternatively, (*E*)-((*E*)-hex-2-en-1-yl)-*N*-(4-nitrophenyl)acetimidate **9e** (76.2 µmol, 20.0 mg) was reacted according to **GP4b** with the activated catalyst **[FBIP-OTs-MeCN]** (3 mol%, 2.3 µmol, 190 µL of a stock solution with *c* = 12.0 µmol/mL in $CHCl_3$, prepared according to **GP3b**) in $CHCl_3$ at room temperature for 72 h to yield **10e** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 4/1) as a white solid (68.6 µmol, 18.0 mg, 90%, *ee* = 90%).

$C_{14}H_{18}N_2O_3$, MW: 262.31 g mol^{-1}. **Mp:** 46.4-47.0 °C (sample with *ee* = 90%). **$[\alpha]_D^{20}$:** −32.3 (*c* = 0.32, $CHCl_3$, sample with *ee* = 90%). **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 8.30-8.23 (*m*, 2 H, *m*-$C_6H_4NO_2$), 7.34-7.27 (*m*, 2 H, *o*-$C_6H_4NO_2$), 5.70-5.55 (*m*, 1 H, CHC*H*=CH_2), 5.25-5.11 (*m*, 3 H, C*H*CH=CH_2), 1.82 (*s*, 3 H, C(=O)CH_3), 1.57-1.28 (*m*, 4 H, CH_2CH_2CH$_3$), 0.91 (*t*, *J* = 7.2, 3 H, $CH_2CH_2CH_3$). **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 169.1, 147.2, 146.3, 136.6, 130.9, 124.6, 118.4, 58.1, 34.2, 23.6, 19.6, 13.9. **IR ($CDCl_3$):** $\tilde{\nu}$ = 2959, 2932, 2873, 2362, 1666, 1591, 1522, 1493, 1380, 1345, 1310, 1109, 927, 854, 752, 706. **MS (ESI) *m/z*:** 413.2 (2%), 360.3 (13%), 285.1 (100%, $[M+Na]^+$), 263.1 (1%, $[M+H]^+$).]$^+$). **HRMS (ESI) *m/z*:** calculated for $C_{14}H_{18}N_2O_3$ + Na: 285.1210; found: 285.1210.

(*S*)-*N*-(Hex-1-en-3-yl)-*N*-(4-nitrophenyl)acetamide ((*ent*)-10e)

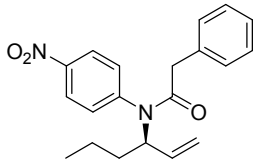


(*ent*)-10e

According to **GP4b**, (*E*)-((*Z*)-hex-2-en-1-yl)-*N*-(4-nitrophenyl)acetimidate **(*Z*)-9e** (76.2 µmol, 20.0 mg) was reacted with the activated catalyst **[FBIP-OTs-MeCN]** ((3 mol%, 2.3 µmol, 190 µL of a stock solution with c = 12.0 µmol/mL in $CHCl_3$, prepared according to **GP3b**) in $CHCl_3$ at room temperature for 72 h to yield **(*ent*)-10e** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 4/1) as a white solid (70.9 µmol, 18.6 mg, 93%, *ee* = 92%). The enantiomeric excess was determined by HPLC: *Chiracel AS-H*, *n*-hexane/*i*-PrOH (96/4), 1.0 mL min^{-1}, 250 nm, 39.92min (major enantiomer), 53.48 min (minor enantiomer).

$C_{14}H_{18}N_2O_3$, MW: 262.31 g mol^{-1}. **Mp:** 46.2-46.9 °C (sample with *ee* = 92%). **$[\alpha]_D^{20}$:** +35.3 (c = 0.31, $CHCl_3$, sample with *ee* = 92%). **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 8.30-8.23 (*m*, 2 H, *m*-$C_6H_4NO_2$), 7.34-7.27 (*m*, 2 H, *o*-$C_6H_4NO_2$), 5.70-5.55 (*m*, 1 H, CHC*H*=CH_2), 5.25-5.11 (*m*, 3 H, C*H*CH=CH_2), 1.82 (*s*, 3 H, C(=O)CH_3), 1.57-1.28 (*m*, 4 H, CH_2CH_2CH_3), 0.91 (*t*, *J* = 7.2, 3 H, $CH_2CH_2CH_3$). The other analytical data are in accordance with **10e**.

(*R*)-*N*-(Hex-1-en-3-yl)-*N*-(4-nitrophenyl)-2-phenylacetamide (10f)



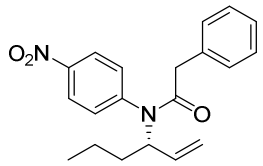
10f

According to **GP4b**, (*E*)-((*E*)-hex-2-en-1-yl)-*N*-(4-nitrophenyl)-2-phenylacetimidate **9f** (59.1 µmol, 20.0 mg) was reacted with proton sponge (4 mol%, 2.4 µmol, 50 µL of a stock solution with c = 50.0 µmol/mL in $CHCl_3$) and the activated catalyst **[PPFIP-$(NO_3)_2$]$_2$** (1 mol%, 0.6 µmol, 100 µL of a stock solution with c = 6.0 µmol/mL in $CHCl_3$, prepared according to **GP3a**) in $CHCl_3$ at 70 °C for 24 h to yield **10f** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 4/1) as a colorless oil (54.9 µmol, 18.6 mg, 93%, *ee* = 95%). The enantiomeric excess was determined by HPLC: *Chiracel AS-H*, *n*-hexane/*i*-PrOH (99/1), 0.8 mL min^{-1}, 250 nm, 69.13 min (minor enantiomer), 77.19 min (major enantiomer).

$C_{20}H_{22}N_2O_3$, MW: 338.41 g mol^{-1}. **$[\alpha]_D^{20}$:** −18.0 (c = 0.50, $CHCl_3$, sample with *ee* = 95%). **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 8.23-8.16 (*m*, 2 H, *m*-$C_6H_4NO_2$), 7.25-7.19 (*m*, 3 H, *m*-C_6H_5 & *p*-C_6H_5), 7.18-

7.12 (*m*, 2 H, *o*-$C_6H_4NO_2$), 7.02-6.92 (*m*, 2 H, *o*-C_6H_5), 5.66-5.51 (*m*, 1 H, CHC*H*=CH_2), 5.24-5.11 (*m*, 3 H, C*H*CH=CH_2), 3.40 (*s*, 2 H, C(=O)CH_2), 1.54-1.23 (*m*, 4 H, CH_2CH_2CH_3), 0.89 (*t*, *J* = 7.1, 3 H, $CH_2CH_2CH_3$). **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 169.7, 147.2, 145.5, 136.4, 134.7, 131.4, 128.8, 128.6, 126.9, 118.6, 58.4, 42.4, 34.1, 19.6, 13.8. **IR ($CDCl_3$):** $\tilde{\nu}$ = 2959, 2931, 2872, 1658, 1591, 1522, 1493, 1454, 1379, 1343, 1311, 1246, 1200, 1167, 1110, 998, 930, 856, 715, 703. **MS (ESI) *m/z*:** 377.1 (1%, $[M+K]^+$), 361.2 (97%, $[M+Na]^+$), 339.2 (97%, $[M+H]^+$), 304.3 (2%), 282.3 (1%), 257.1 (100%), 233.1 (1%), 151.1 (1%), 139.1 (3%). **HRMS (ESI) *m/z*:** calculated for $C_{20}H_{22}N_2O_3$ + Na: 361.1523; found: 361.1527.

(*S*)-*N*-(Hex-1-en-3-yl)-*N*-(4-nitrophenyl)-2-phenylacetamide ((*ent*)-10f)

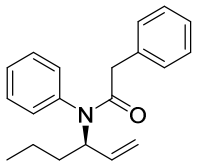


(*ent*)-10f

According to **GP4b**, (*E*)-((*Z*)-hex-2-en-1-yl)-*N*-(4-nitrophenyl)-2-phenylacetimidate (**Z**)-**9f** (59.1 µmol, 20.0 mg) was reacted with the activated catalyst **[FBIP-OTs-MeCN]** (3 mol%, 1.7 µmol, 140 µL of a stock solution with *c* = 12.0 µmol/mL in $CHCl_3$, prepared according to **GP3b**) in $CHCl_3$ at room temperature for 72 h to yield **10f** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 4/1) as a colorless oil (56.7 µmol, 19.2 mg, 96%, *ee* = 95%). The enantiomeric excess was determined by HPLC: *Chiracel AS-H*, *n*-hexane/*i*-PrOH (99/1), 0.8 mL min^{-1}, 250 nm, 60.26 min (major enantiomer), 71.24 min (minor enantiomer).

$C_{20}H_{22}N_2O_3$, MW: 338.41 g mol^{-1}. **$[\alpha]_D^{20}$:** +17.3 (*c* = 0.53, $CHCl_3$, sample with *ee* = 95%). **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 8.23-8.16 (*m*, 2 H, *m*-$C_6H_4NO_2$), 7.25-7.19 (*m*, 3 H, *m*-C_6H_5 & *p*-C_6H_5), 7.18-7.12 (*m*, 2 H, *o*-$C_6H_4NO_2$), 7.02-6.92 (*m*, 2 H, *o*-C_6H_5), 5.66-5.51 (*m*, 1 H, CHC*H*=CH_2), 5.24-5.11 (*m*, 3 H, C*H*CH=CH_2), 3.40 (*s*, 2 H, C(=O)CH_2), 1.54-1.23 (*m*, 4 H, CH_2CH_2CH_3), 0.89 (*t*, *J* = 7.1, 3 H, $CH_2CH_2CH_3$).). The other analytical data are in accordance with **10f**.

(*R*)-*N*-(Hex-1-en-3-yl)-*N*,2-diphenylacetamide (10g)

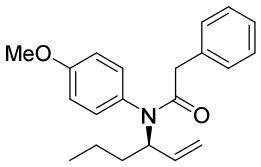


10g

According to **GP4b**, (*E*)-((*E*)-hex-2-en-1-yl)-*N*-phenyl-2-phenylacetimidate **9g** (68.2 µmol, 20.0 mg) was reacted with proton sponge (4 mol%, 2.7 µmol, 55 µL of a stock solution with c = 50.0 µmol/mL in $CHCl_3$) and the activated catalyst **[PPFIP-$(NO_3)_2$]$_2$** (1 mol%, 0.7 µmol, 120 µL of a stock solution with c = 6.0 µmol/mL in $CHCl_3$, prepared according to **GP3a**) in $CHCl_3$ at 70 °C for 24 h to yield **10g** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 5/1) as a colorless oil (61.4 µmol, 18.0 mg, 90%, *ee* = 97%). The enantiomeric excess was determined by HPLC: *Chiracel OD-H*, *n*-hexane/*i*-PrOH (99.5/0.5), 1.0 mL min^{-1}, 250 nm, 14.39 min (minor enantiomer), 15.10 min (major enantiomer).

$C_{20}H_{23}NO$, MW: 293.41 g mol^{-1}. **$[\alpha]_D^{20}$:** +9.4 (c = 0.31, $CHCl_3$, sample with *ee* = 97%). **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.40-7.33 (*m*, 3 H, Ar-*H*), 7.25-7.14 (*m*, 3 H, Ar-*H*), 7.06-6.98 (*m*, 4 H, Ar-*H*), 5.66-5.52 (*m*, 1 H, CHC*H*=CH_2), 5.23-5.06 (*m*, 3 H, C*H*CH=CH_2), 3.33 (*s*, 2 H, C(=O)CH_2), 1.60-1.24 (*m*, 4 H, CH_2C$H_2$$CH_3$), 0.89 (*t*, *J* = 7.2, $CH_2CH_2CH_3$). **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 170.1, 139.4, 137.2, 135.6, 130.5 (*b*), 129.06, 129.06, 128.3, 128.2, 126.5, 117.7, 58.0, 41.9, 34.3, 19.6, 13.9. **IR ($CDCl_3$):** $\tilde{\nu}$ = 3062, 2958, 2931, 2872, 1652, 1594, 1493, 1454, 1383, 1344, 1315, 1257, 1162, 1118, 1074, 1030, 998, 926, 771, 710. **MS (EI) *m/z:*** 293.2 (26%, $[M]^+$), 278.2 (3%), 264.1 (10%), 250.1 (24%), 211.1 (10%), 202.1 (17%), 175.1 (21%), 146.1 (6%), 132.1 (100%), 119.0 (10%), 104.0 (3%), 91.0 (59%), 83.1 (12%), 65.0 (7%), 55.0 (17%), 41.0 (10%). **HRMS (ESI) *m/z*:** calculated for $C_{20}H_{23}NO$ + H: 294.1852; found: 294.1845.

(*R*)-*N*-(Hex-1-en-3-yl)-*N*-(4-methoxyphenyl)-2-phenylacetamide (10h)



10h

According to **GP4b**, (*E*)-((*E*)-hex-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate **9h** (61.8 µmol, 20.0 mg) was reacted with proton sponge (1 mol%, 0.6 µmol, 12 µL of a stock solution with c = 50.0 µmol/mL in $CHCl_3$) and the activated catalyst **[PPFIP-$(NO_3)_2$]$_2$** (0.25 mol%, 0.15 µmol,

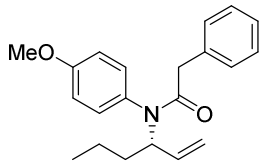
25 µL of a stock solution with c = 6.0 µmol/mL in $CHCl_3$, prepared according to **GP3a**) in $CHCl_3$ at 70 °C for 24 h to yield **10h** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 6/1) as a colorless oil (58.1 µmol, 18.8 mg, 94%, *ee* = 97%). The enantiomeric excess was determined by HPLC: *Chiracel OD-H*, *n*-hexane/*i*-PrOH (99.5/0.5), 1.0 mL min^{-1}, 250 nm, 20.83 min (minor enantiomer), 22.12 min (major enantiomer).

Upscale: According to **GP4b**, (*E*)-((*E*)-hex-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate **9h** (0.77 mmol, 250.0 mg) was reacted with proton sponge (2 mol%, 15 µmol, 3.3 mg) and the activated catalyst **[PPFIP-$(NO_3)_2$]$_2$** (0.5 mol%, 3.8 µmol, 320 µL of a stock solution with c = 12.0 µmol/mL in $CHCl_3$, prepared according to **GP3a**) in $CHCl_3$ at 70 °C for 24 h to yield **10h** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 6/1) as a colorless oil (0.75mol, 242.6 mg, 97%, *ee* = 96%).

Alternatively, (*E*)-((*E*)-hex-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate **9h** (61.8 µmol, 20.0 mg) was reacted according to **GP4b** with the activated catalyst **[FBIP-OTs-MeCN]** (3 mol%, 1.8 µmol, 150 µL of a stock solution with c = 12.0 µmol/mL in $CHCl_3$, prepared according to **GP3b**) in $CHCl_3$ at room temperature for 72 h to yield **10h** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 6/1) as a colorless oil (58.7 µmol, 19.0 mg, 95%, *ee* = 86%).

$C_{21}H_{25}NO_2$, MW: 323.44 g mol^{-1}. **$[\alpha]_D^{20}$:** +6.6 (c = 0.80, $CHCl_3$, sample with *ee* = 97%). **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.25-7.14 (*m*, 3 H, *m*-C_6H_5 & *p*-C_6H_5), 7.06-7.00 (*m*, 2 H, *o*-C_6H_5), 6.96-6.82 (*m*, 4 H, C_6H_4OMe), 5.64-5.50 (*m*, 1 H, CHC*H*=CH_2), 5.22-5.05 (*m*, 3 H, C*H*CH=CH_2), 3.84 (*s*, 3 H, OCH_3), 3.34 (*s*, 2 H, C(=O)CH_2), 1.54-1.24 (*m*, 4 H, CH_2CH_2CH_3), 0.89 (*t*, *J* = 7.2, 3 H, $CH_2CH_2CH_3$). **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 170.9, 159.2, 137.3, 135.7, 131.9, 131.8 (*b*), 129.1, 128.2, 126.4, 117.6, 114.1, 57.7, 55.5, 41.8, 34.2, 19.6, 13.9. **IR ($CDCl_3$):** $\tilde{\nu}$ = 2958, 2932, 2871, 1651, 1509, 1454, 1386, 1292, 1248, 1169, 1106, 1033, 926, 835, 725, 697, 596. **MS (ESI) *m/z*:** 346.2 (36%, $[M+Na]^+$), 324.2 (100%, $[M+H]^+$), 242.1 (3%). **HRMS (ESI) *m/z*:** calculated for $C_{21}H_{25}NO_2$ + H: 324.1958; found: 324.1963.

(*S*)-*N*-(Hex-1-en-3-yl)-*N*-(4-methoxyphenyl)-2-phenylacetamide ((*ent*)-10h)



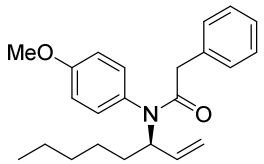
(*ent*)-10h

According to **GP4b**, (*E*)-((*Z*)-hex-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate **(*Z*)-9h** (61.8 µmol, 20.0 mg) was reacted with the activated catalyst **[FBIP-OTs-MeCN]** (3 mol%, 1.8 µmol,

150 µL of a stock solution with c = 12.0 µmol/mL in $CHCl_3$, prepared according to **GP3b**) in $CHCl_3$ at room temperature for 72 h to yield **(*ent*)-10h** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 6/1) as a colorless oil (59.9 µmol, 19.4 mg, 97%, *ee* = 89%). The enantiomeric excess was determined by HPLC: *Chiracel OD-H*, *n*-hexane/*i*-PrOH (99.5/0.5), 0.5 mL min^{-1}, 250 nm, 39.67 min (major enantiomer), 47.24 min (minor enantiomer).

$C_{21}H_{25}NO_2$, MW: 323.44 g mol^{-1}. **$[\alpha]_D^{20}$:** −5.0 (c = 0.61, $CHCl_3$, sample with *ee* = 89%). **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.25-7.14 (*m*, 3 H, *m*-C_6H_5 & *p*-C_6H_5), 7.06-7.00 (*m*, 2 H, *o*-C_6H_5), 6.96-6.82 (*m*, 4 H, C_6H_4OMe), 5.64-5.50 (*m*, 1 H, CHC*H*=CH_2), 5.22-5.05 (*m*, 3 H, C*H*CH=CH_2), 3.84 (*s*, 3 H, OCH_3), 3.34 (*s*, 2 H, C(=O)CH_2), 1.54-1.24 (*m*, 4 H, CH_2C$H_2$$CH_3$), 0.89 (*t*, J = 7.2, 3 H, CH_2CH_2CH_3). The other analytical data are in accordance with **10h**.

(*R*)-*N*-(4-Methoxyphenyl)-*N*-(oct-1-en-3-yl)-2-phenylacetamide (10i)

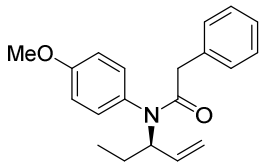


10i

According to **GP4b**, (*E*)-((*E*)-oct-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate **9i** (56.9 µmol, 20.0 mg) was reacted with proton sponge (1 mol%, 0.5 µmol, 10 µL of a stock solution with c = 50.0 µmol/mL in $CHCl_3$) and the activated catalyst **[PPFIP-$(NO_3)_2$]$_2$** (0.25 mol%, 0.14 µmol, 23 µL of a stock solution with c = 6.0 µmol/mL in $CHCl_3$, prepared according to **GP3a**) in $CHCl_3$ at 70 °C for 24 h to yield **10i** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 7/1) as a colorless oil (52.3 µmol, 18.4 mg, 92%, *ee* = 94%). The enantiomeric excess was determined by HPLC: *Chiracel AD-H*, *n*-hexane/*i*-PrOH (99.2/0.8), 1.1 mL min^{-1}, 250 nm, 23.48 min (minor enantiomer), 27.00 min (major enantiomer).

$C_{23}H_{29}NO_2$, MW: 351.49 g mol^{-1}. **$[\alpha]_D^{20}$:** +15.4 (c = 0.44, $CHCl_3$, sample with *ee* = 94%). **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.25-7.14 (*m*, 3 H, *m*-C_6H_5 & *p*-C_6H_5), 7.06-7.00 (*m*, 2 H, *o*-C_6H_5), 6.95-6.82 (*m*, 4 H, C_6H_4OMe), 5.65-5.50 (*m*, 1 H, CHC*H*=CH_2), 5.22-5.05 (*m*, 3 H, C*H*CH=CH_2), 3.84 (*s*, 3 H, OCH_3), 3.34 (*s*, 2 H, C(=O)CH_2), 1.56-1.18 (*m*, 8 H, $(CH_2)_4CH_3$), 0.86 (*t*, J = 6.8, 3 H, $(CH_2)_4$CH_3). **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 170.9, 159.2, 137.3, 135.7, 131.9, 131.2, 129.1, 128.2, 126.4, 117.6, 114.1, 57.9, 55.5, 41.9, 32.1, 31.7, 26.0, 22.6, 14.1. **IR ($CDCl_3$):** $\tilde{\nu}$ = 2955, 2930, 2858, 1651, 1509, 1454, 1386, 1343, 1248, 1169, 1031, 997, 925, 835, 724, 696, 597. **MS (ESI) *m/z*:** 374.2 (100%, $[M+Na]^+$), 352.3 (6%, $[M+H]^+$). **HRMS (ESI) *m/z*:** calculated for $C_{23}H_{29}NO_2$ + Na: 374.2091; found: 374.2097.

(*R*)-*N*-(4-Methoxyphenyl)-*N*-(pent-1-en-3-yl)-2-phenylacetamide (10j)

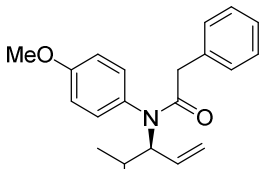


10j

According to **GP4b**, (*E*)-((*E*)-pent-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate **9j** (64.6 µmol, 20.0 mg) was reacted with proton sponge (0.4 mol%, 0.25 µmol, 10 µL of a stock solution with c = 25.0 µmol/mL in $CHCl_3$) and the activated catalyst **[PPFIP-$(NO_3)_2$]$_2$** (0.1 mol%, 0.06 µmol, 10 µL of a stock solution with c = 6.0 µmol/mL in $CHCl_3$, prepared according to **GP3a**) in $CHCl_3$ at 70 °C for 24 h to yield **10j** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 5/1) as a colorless oil (60.1 µmol, 18.6 mg, 93%, *ee* = 96%). The enantiomeric excess was determined by HPLC: *Chiracel AS-H*, *n*-hexane/*i*-PrOH (99/1), 1.0 mL min^{-1}, 250 nm, 33.14 min (major enantiomer), 44.14 min (minor enantiomer).

$C_{20}H_{23}NO_2$, MG: 309.41 g mol^{-1}. **$[\alpha]_D^{20}$:** +2.0 (c = 0.50, $CHCl_3$, sample with *ee* = 96%). **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.25-7.15 (*m*, 3 H, *m*-C_6H_5 & *p*-C_6H_5), 7.07-7.00 (*m*, 2 H, *o*-C_6H_5), 6.96-6.82 (*m*, 4 H, C_6H_4OMe), 5.63-5.49 (*m*, 1 H, CHC*H*=CH_2), 5.22-5.02 (*m*, 3 H, C*H*CH=C*H*$_2$), 3.84 (*s*, 3 H, OC*H*$_3$), 3.35 (*s*, 2 H, C(=O)C*H*$_2$), 1.61-1.33 (*m*, 2 H, C*H*$_2$CH$_3$), 0.90 (*t*, *J* = 7.5, 3 H, CH_2CH_3). **^{13}C-NMR (75 MHz, $CDCl_3$):** δ = 171.0, 159.2, 137.0, 135.7, 131.9, 131.1, 129.1, 128.2, 126.4, 117.8, 114.1, 59.5, 55.5, 41.8, 25.1, 11.0. **IR ($CDCl_3$):** $\tilde{\nu}$ = 2964, 2932, 2875, 1649, 1508, 1454, 1385, 1291, 1247, 1169, 1106, 1032, 996, 927, 838, 752, 724, 595. **MS (EI) *m/z*:** 309.2 (36%, [M]$^+$), 294.2 (7%), 280.1 (7%), 241.1 (22%), 224.1 (7%), 218.1 (29%), 191.1 (17%), 175.1 (10%), 162.1 (100%), 149.1 (15%), 134.1 (12%), 123.1 (52%), 108.0 (9%), 91.1 (65%), 77.0 (4%), 69.1 (25%), 41.0 (14%). **HRMS (EI) *m/z*:** calculated for $C_{20}H_{23}NO_2$: 309.1729; found: 309.1736.

(*R*)-*N*-(4-Methoxyphenyl)-*N*-(4-methylpent-1-en-3-yl)-2-phenylacetamide (10k)



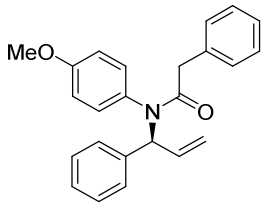
10k

According to **GP4b**, (*E*)-((*E*)-4-methylpent-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate **9k** (61.8 µmol, 20.0 mg) was reacted with proton sponge (4 mol%, 2.5 µmol, 50 µL of a stock solution with c = 50.0 µmol/mL in CH_2Cl_2) and the activated catalyst **[PPFIP-$(NO_3)_2$]$_2$** (1 mol%, 0.6 µmol,

100 µL of a stock solution with c = 6.0 µmol/mL in CH_2Cl_2, prepared according to **GP3a**) in CH_2Cl_2 at 50 °C for 48 h to yield **10k** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 7/1) as a colorless oil (59.3 µmol, 19.2 mg, 96%, *ee* = 95%). The enantiomeric excess was determined by HPLC: *Chiracel AD-H*, *n*-hexane/*i*-PrOH (99/1), 1.0 mL min^{-1}, 250 nm, 33.81 min (major enantiomer), 38.72 min (minor enantiomer).

$C_{21}H_{25}NO_2$, MW: 323.44 g mol^{-1}. **$[\alpha]_D^{20}$:** −27.8 (c = 0.50, $CHCl_3$, sample with *ee* = 95%). **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.25-7.13 (*m*, 3 H, *m*-C_6H_5 & *p*-C_6H_5), 7.06-6.80 (*m*, 6 H, *o*-C_6H_5 & C_6H_4OMe), 5.61-5.46 (*m*, 1 H, C*H*=CH_2), 5.27-5.07 (*m*, 2 H, CH=CH_2), 4.63 (*pseudo t*, J = 10.0, 1 H, NC*H*), 3.84 (*s*, 3 H, OCH_3), 3.35 (*s*, 2 H, C(=O)CH_2), 1.89-1.76 (*m*, 1 H, C*H*$(CH_3)_2$), 1.03 (*d*, J = 6.7, 3 H, CH(CH_3)$_2$), 0.79 (*d*, J = 6.7, 3 H, CH(CH_3)$_2$). **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 171.1, 159.1, 136.7, 135.7, 133.0, 132.1, 130.6, 129.1, 128.2, 126.4, 118.6, 114.2, 66.4, 55.5, 42.0, 29.6, 20.2, 20.0. **IR ($CDCl_3$):** $\tilde{\nu}$ = 2963, 1651, 1509, 1454, 1383, 1329, 1292, 1248, 1170, 1039, 927, 836, 752, 725, 595. **MS (ESI) *m/z*:** 346.2 (100%, $[M+Na]^+$), 324.2 (19%, $[M+H]^+$), 242.1 (3%). **HRMS (ESI) *m/z*:** calculated for $C_{21}H_{25}NO_2$ + Na: 346.1778; found: 346.1778.

(*S*)-*N*-(4-Methoxyphenyl)-2-phenyl-*N*-(1-phenylallyl)acetamide (10l)



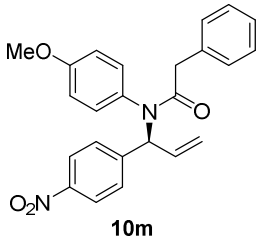
10l

According to **GP4b**, (*E*)-((*E*)-3-phenylprop-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate **9l** (55.9 µmol, 20.0 mg) was reacted with proton sponge (4 mol%, 2.2 µmol, 45 µL of a stock solution with c = 50.0 µmol/mL in CH_2Cl_2) and the activated catalyst **[PPFIP-$(NO_3)_2$]$_2$** (1 mol%, 0.5 µmol, 85 µL of a stock solution with c = 6.0 µmol/mL in CH_2Cl_2, prepared according to **GP3a**) in CH_2Cl_2 at room temperature for 72 h to yield **10l** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 4/1) as a colorless oil (52.0 µmol, 18.6 mg, 93%, *ee* = 95%). The enantiomeric excess was determined by HPLC: *Chiracel AD-H*, *n*-hexane/*i*-PrOH (95/5), 1.0 mL min^{-1}, 250 nm, 25.57 min (minor enantiomer), 38.24 min (major enantiomer).

$C_{24}H_{23}NO_2$, MW: 357.45 g mol^{-1}. **$[\alpha]_D^{20}$:** +11.7 (c = 0.50, $CHCl_3$, sample with *ee* = 95%). **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.26-6.20 (*m*, 15 H, Ar-*H* & NC*H*), 6.05-5.90 (*m*, 1 H, C*H*=CH_2), 5.41-5.24 (*m*, 2 H, CH=CH_2), 3.79 (*s*, 3 H, OCH_3), 3.39 (*s*, 2 H, C(=O)CH_2). **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 171.0, 159.2, 139.7, 135.5, 135.0, 131.9, 131.7, 129.2, 128.5, 128.3, 128.2, 127.4, 126.5, 119.0, 113.9, 61.3, 55.4, 41.7. **IR ($CDCl_3$):** $\tilde{\nu}$ = 3061, 3029, 2837, 1650, 1603, 1509, 1454, 1382, 1312,

1293, 1248, 1169, 1075, 1030, 930, 837, 811, 774, 737, 724, 701, 595, 555. **MS (ESI)** ***m/z:*** 396.1 (1%, $[M+K]^+$), 380.2 (100%, $[M+Na]^+$), 358.2 (6%, $[M+H]^+$). **HRMS (ESI)** ***m/z*****:** calculated for $C_{24}H_{23}NO_2$ + Na: 380.1621; found: 380.1614.

(*S*)-*N*-(4-Methoxyphenyl)-*N*-(1-(4-nitrophenyl)allyl)-2-phenylacetamide (10m)

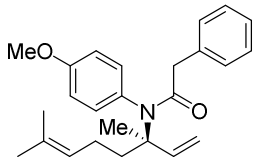


10m

According to **GP4b**, (*E*)-((*E*)-3-(4-nitrophenylprop-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate **9m** (49.7 µmol, 20.0 mg) was reacted with proton sponge (4 mol%, 2.0 µmol, 40 µL of a stock solution with c = 50.0 µmol/mL in CH_2Cl_2) and the activated catalyst **[PPFIP-$(NO_3)_2$]$_2$** (1 mol%, 0.5 µmol, 85 µL of a stock solution with c = 6.0 µmol/mL in CH_2Cl_2, prepared according to **GP3a**) in CH_2Cl_2 at room temperature for 72 h to yield **10m** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 4/1) as a colorless oil (47.7 µmol, 19.2 mg, 96%, *ee* = 90%). The enantiomeric excess was determined by HPLC: *Chiracel AD-H*, *n*-hexane/*i*-PrOH (93/7), 1.0 mL min^{-1}, 250 nm, 44.69 min (minor enantiomer), 53.77 min (major enantiomer).

$C_{24}H_{22}N_2O_4$, MW: 402.45 g mol^{-1}. **$[\alpha]_D^{20}$:** +77.7 (c = 0.13, $CHCl_3$, sample with *ee* = 90%).**^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 8.14-8.07 (*m*, 2 H, *m*-$C_6H_4NO_2$), 7.41-7.33 (*m*, 2 H, *o*-$C_6H_4NO_2$), 7.27-6.27 (*m*, 10 H, C_6H_5 & C_6H_4OMe & NC*H*), 6.02-5.87 (*m*, 1 H, C*H*=CH_2), 5.43-5.30 (*m*, 2 H, CH=CH_2), 3.81 (*s*, 3 H, OCH_3), 3.41 (*s*, 2 H, C(=O)CH_2). **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 171.3, 159.5, 147.4, 147.1, 135.0, 133.8, 131.6, 131.2, 129.1, 129.0, 128.4, 126.8, 123.5, 120.5, 114.5, 61.5, 55.5, 41.6. **IR ($CDCl_3$):** $\tilde{\nu}$ = 3029, 2935, 2838, 1653, 1605, 1509, 1378, 1344, 1313, 1294, 1248, 1170, 1108, 1029, 935, 856, 838, 808, 738, 726, 704, 594, 569. **MS (ESI)** ***m/z:*** 441.1 (1%, $[M+K]^+$), 425.2 (100%, $[M+Na]^+$), 403.2 (6%, $[M+H]^+$). **HRMS (ESI)** ***m/z*****:** calculated for $C_{24}H_{22}N_2O_4$ + Na: 425.1472; found: 425.1463.

(*R*)-*N*-(3,7-Dimethylocta-1,6-dien-3-yl)-*N*-(4-methoxyphenyl)-2-phenylacetamide (10n)

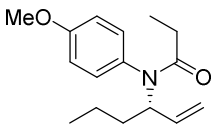


10n

According to **GP4b**, (*E*)-((*E*)-3,7-dimethylocta-2,6-dien-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate **9n** (53.0 µmol, 20.0 mg) was reacted with proton sponge (4 mol%, 2.1 µmol, 42 µL of a stock solution with c = 50.0 µmol/mL in CH_2Cl_2) and the activated catalyst **[PPFIP-$(NO_3)_2$]$_2$** (1 mol%, 0.5 µmol, 85 µL of a stock solution with c = 6.0 µmol/mL in CH_2Cl_2, prepared according to **GP3a**) in CH_2Cl_2 at 50 °C for 48 h to yield **10n** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 12/1) as a colorless oil (51.9 µmol, 19.6 mg, 98%, *ee* = 98%). The enantiomeric excess was determined by HPLC: *Chiracel AD-H*, *n*-hexane/*i*-PrOH (99/1), 1.0 mL min^{-1}, 250 nm, 22.19 min (minor enantiomer), 24.54 min (major enantiomer).

$C_{25}H_{31}NO_2$, MW: 377.53 g mol^{-1}. **$[\alpha]_D^{20}$:** −20.0 (c = 0.66, $CHCl_3$, sample with *ee* = 98%). **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.25-7.12 (*m*, 3 H, *m*-C_6H_5 & *p*-C_6H_5), 7.04-6.79 (*m*, 6 H, *o*-C_6H_5 & C_6H_4OMe), 6.22 (*dd*, J = 17.6, 10.9, 1 H, C*H*=CH_2), 5.12-4.96 (*m*, 3 H, CH=CH_2 & C*H*=C$(CH_3)_2$), 3.84 (*s*, 3 H, OCH_3), 3.26 (*s*, 2 H, C(=O)CH_2), 2.33-2.19 (*m*, 1 H, NC(CH_3)CH_2CH_2), 2.00-1.90 (*m*, 2 H, NC(CH_3)CH_2CH_2), 1.83-1.71 (*m*, 1 H, NC(CH_3)CH_2CH_2), 1.65 (*s*, 3 H, CH=C(CH_3)$_2$), 1.55 (*s*, 3 H, CH=C(CH_3)$_2$), 1.17 (*s*, 3 H, NC(CH_3)). **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 171.2, 159.0, 143.6, 135.9, 134.4, 131.7, 131.6, 131.5, 129.2, 128.1, 126.4, 124.2, 113.99, 113.95, 111.7, 64.0, 55.5, 43.9, 38.5, 25.7, 24.2, 23.4, 17.7. **IR ($CDCl_3$):** $\tilde{\nu}$ = 3029, 2968, 2915, 1654, 1508, 1454, 1359, 1289, 1247, 1167, 1105, 1032, 918, 836, 731, 696, 598, 566. **MS (ESI) *m/z*:** 400.2 (100%, $[M+Na]^+$), 378.2 (9%, $[M+H]^+$), 242.1 (4%). **HRMS (ESI) *m/z*:** calculated for $C_{25}H_{31}NO_2$ + Na: 400.2247; found: 400.2235.

(*S*)-*N*-(Hex-1-en-3-yl)-*N*-(4-methoxyphenyl)propionamide ((*ent*)-10o)



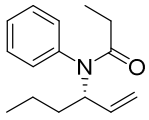
(*ent*)-10o

According to **GP4b**, (*E*)-((*Z*)-hex-2-en-1-yl)-*N*-(4-methoxyphenyl)propionimidate **(*Z*)-9o** (76.5 µmol, 20.0 mg) was reacted with the activated catalyst **[FBIP-OTs-MeCN]** ((3 mol%, 2.3 µmol, 190 µL of a

stock solution with c = 12.0 µmol/mL in $CHCl_3$, prepared according to **GP3b**) in $CHCl_3$ at room temperature for 72 h to yield **(*ent*)-10o** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 8/1) as a colorless oil (64.3 µmol, 16.8 mg, 84%, *ee* = 92%). The enantiomeric excess was determined by HPLC: *Chiracel AD-H*, *n*-hexane/*i*-PrOH (99.4/0.6), 1.0 mL min^{-1}, 250 nm, 24.91 min (major enantiomer), 30.45 min (minor enantiomer).

$C_{16}H_{23}NO_2$, MW: 261.36 g mol^{-1}. **$[\alpha]_D^{20}$:** −6.9 (c = 0.40, $CHCl_3$, sample with *ee* = 92%). **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.05-6.95 (*m*, 2 H, *o*-C_4H_4OMe), 6.93-6.84 (*m*, 2 H, *m*-C_6H_4OMe), 5.66-5.51 (*m*, 1 H, CHC*H*=CH_2), 5.24-5.05 (*m*, 3 H, C*H*CH=CH_2), 3.83 (*s*, 3 H, OCH_3), 1.94 (*qd*, *J* = 7.6, 1.5, 2 H, C(=O)C$H_2$$CH_3$), 1.56-1.27 (*m*, 4 H, CH_2C$H_2$$CH_3$), 1.00 (*t*, *J* = 7.6, 3 H, C(=O)CH_2CH_3), 0.91 (*t*, *J* = 7.0, 3 H, CH_2CH_2CH_3). **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 173.7, 159.1, 137.5, 132.1, 131.5, 130.9, 117.4, 114.2, 57.3, 55.4, 34.3, 28.3, 19.6, 14.0, 9.6, 8.1. **IR ($CDCl_3$):** $\tilde{\nu}$ = 2958, 2935, 2873, 1655, 1509, 1462, 1389, 1291, 1247, 1170, 1034, 924, 835, 805, 757, 588, 530. **MS (EI) *m/z*:** 261.2 (20%, $[M]^+$), 246.2 (5%), 232.1 (12%), 218.1 (12%), 204.1 (14%), 188.1 (3%), 179.1 (17%), 162.1 (100%), 148.1 (15%), 134.0 (8%), 123.1 (67%), 113.1 (17%), 92.0 (3%), 77.0 (5%), 65.0 (1%), 57.0 (9%), 41.0 (7%). **HRMS (EI) *m/z*:** calculated for $C_{16}H_{23}NO_2$: 261.1729; found: 261.1727.

(*S*)-*N*-(Hex-1-en-3-yl)-*N*-phenylpropionamide ((*ent*)-10p)



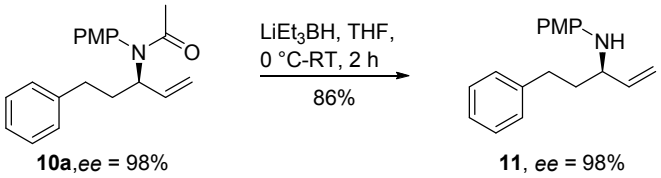
(*ent*)-10p

According to **GP4b**, (*E*)-((*Z*)-hex-2-en-1-yl)-*N*-phenylpropionimidate **(*Z*)-9p** (86.5 µmol, 20.0 mg) was reacted with the activated catalyst **[FBIP-OTs-MeCN]** ((3 mol%, 2.6 µmol, 215 µL of a stock solution with c = 12.0 µmol/mL in $CHCl_3$, prepared according to **GP3b**) in $CHCl_3$ at room temperature for 72 h to yield **(*ent*)-10p** after purification by column chromatography on silicagel (petrol ether/ethyl acetate = 8/1) as a colorless oil (73.5 µmol, 17.0 mg, 85%, *ee* = 90%). The enantiomeric excess was determined by HPLC: *Chiracel AS-H*, *n*-hexane/*i*-PrOH (99.5/0.5), 1.0 mL min^{-1}, 250 nm, 10.68 min (major enantiomer), 12.07 min (minor enantiomer).

$C_{15}H_{21}NO$, MW: 231.33 g mol^{-1}. **$[\alpha]_D^{20}$:** −3.8 (c = 0.24, $CHCl_3$, sample with *ee* = 90%). **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.43-7.32 (*m*, 3 H, Ar-*H*), 7.13-7.06 (*m*; 2 H, Ar-*H*), 5.68-5.52 (*m*, 1 H, CHC*H*=CH_2), 5.25-5.06 (*m*, 3 H, C*H*CH=CH_2), 1.93 (*qd*, *J* = 7.6, 1.5, 2 H, C(=O)C$H_2$$CH_3$), 1.60-1.28 (*m*, 4 H, CH_2C$H_2$$CH_3$), 1.01 (*t*, *J* = 7.6, 3 H, C(=O)CH_2CH_3), 0.91 (*t*, *J* = 7.2, 3 H, CH_2CH_2CH_3). The other analytical data are in accordance with the literature.[24]

Removal of Amine Protecting Groups (11, 12, 13)

(*R*)-*N*-(4-Methoxyphenyl)-5-phenylpent-1-en-2-amine (11)



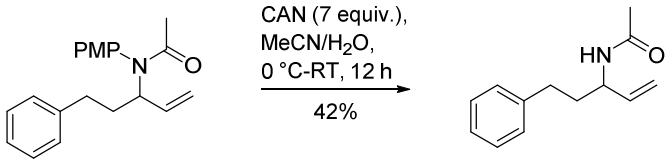
10a,*ee* = 98% **11**, *ee* = 98%

According to a literature procedure,[25] (*R*)-*N*-(4-methoxyphenyl)-*N*-(5-phenylpent-1-en-3-yl)acetamide **10a** (48.5 µmol, 15.0 mg, *ee* = 98%) was dissolved in dry THF (0.33 mL) and cooled to 0 °C. Then $LiEt_3BH$ (3.1 equiv., 0.15 mmol, 0.15 mL, 1 M in THF) was added dropwise and the mixture was stirred at room temperature for 2 h. Subsequently, saturated aqueous NH_4Cl (4.0 mL), aqueous NaOH (4.0 mL, 1 M) and Et_2O (10 mL) were added. The phases were separated and the aqueous phase was extracted with Et_2O (2 x 10 mL). The combined organic extracts were dried over $MgSO_4$ and the solvent was removed under reduced pressure to yield **11** as a slightly yellow oil (41.5 µmol, 11.1 mg, 86%). The enantiomeric excess was determined by HPLC: *Chiracel OD-H*, *n*-hexane/*i*-PrOH (98.2/1.8), 0.8 mL, 250 nm, 22.72 min (minor enantiomer), 24.27 min (major enantiomer).

$C_{18}H_{21}NO$, MW: 267.37 g mol^{-1}. **$[\alpha]_D^{20}$:** −9.2 (*c* = 0.20, $CHCl_3$, sample with *ee* = 98%). **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.31-7.25 (*m*, 2 H, Ar-*H*), 7.22-7.16 (*m*, 3 H, Ar-*H*), 6.77-6.72 (*m*, 2 H, *o*-C_4H_4OMe), 6.56-6.50 (*m*, 2 H, m-C_4H_4OMe), 5.76 (*ddd*, *J* = 17.1, 10.3, 6.3, 1 H, C*H*=CH_2), 5.24-5.12 (*m*, 2 H, CH=CH_2), 3.80-3.74 (*m*, 1 H, NC*H*), 3.74 (*s*, 3 H, OCH_3), 2.75 (*t*, *J* = 7.8, 2 H, $C_6H_5CH_2CH_2$), 1.95-1.85 (*m*, 2 H, $C_6H_5CH_2CH_2$). The analytical data are in accordance with the literature.[1]

The absolute configuration of (*R*)-*N*-(4-methoxyphenyl)-5-phenylpent-1-en-2-amine has already been previously determined.[26] HPLC data and the $[\alpha]_D^{20}$ value were compared to an authentic sample prepared by rearrangement of the corresponding allylic trifluoroacetimidate.[1]

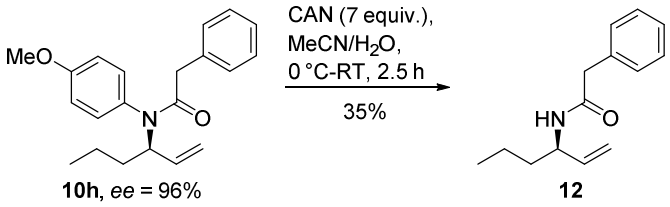
N-(5-Phenylpent-1-en-3-yl)acetamide



Following a literature procedure,[1] ceric ammonium nitrate (0.46 mmol, 250.0 mg) was dissolved in H_2O (2 mL) and cooled to 0 °C. A solution of (*rac*)-*N*-(4-methoxyphenyl)-*N*-(5-phenylpent-1-en-3-yl)acetamide **10a** (64.6 µmol, 20.0 mg, *racemic*) in MeCN (2 mL) was added dropwise and the mixture was stirred at room temperature for 12 h. Subsequently, Et_2O (5 mL) was added, the phases were separated and the aqueous phase was extracted with Et_2O (2 x 10 mL). The combined organic phases were dried over Na_2SO_4 and the solvent was removed under reduced pressure. Purification of the residue by column chromatography on deactivated silicagel (petrol ether/ethyl acetate = 3/1 + 3% NEt_3) yielded *N*-(5-phenylpent-1-en-3-yl)acetamide as a slightly yellow oil (27.1 µmol, 5.5 mg, 42%).

$C_{13}H_{17}NO$, MW: 203.28 g mol^{-1} **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.32-7.25 (*m*, 2 H, Ar-*H*), 7.22-7.15 (*m*, 3 H, Ar-*H*), 5.80 (*ddd*, *J* = 17.3, 10.4, 5.7, 1 H, C*H*=CH_2), 5.28 (*br*, 1 H, N*H*), 5.21-5.13 (*m*, 2 H, CH=CH_2), 4.59-4.48 (*m*, 1 H, NC*H*), 2.67 (*t*, *J* = 7.9, 2 H, $C_6H_5CH_2CH_2$), 1.97 (*s*, 3 H, C(=O)CH_3), 1.94-1.75 (*m*, 2 H, $C_6H_5CH_2CH_2$). **^{13}C-NMR (75 MHz, $CDCl_3$):** δ = 169.5, 141.7, 138.2, 128.6 , 128.5, 126.2, 115.4, 51.4, 36.55, 32.3, 23.6. **IR (in $CDCl_3$)**: $\tilde{\nu}$ = 3272, 2254, 1965, 1650, 1550, 1373, 903, 722, 650. **MS (ESI) *m/z:*** 226.1 (100%, $[M+Na]^+$), 204.1 (83%, $[M+H]^+$), 162.1 (10%), 145.10 (14%). **HRMS (ESI) *m/z*:** calculated for $C_{13}H_{17}NO$ + H: 204.1370; found: 204.1383.

(*R*)-*N*-(Hex-1-en-3-yl)-2-phenylacetamide (12)



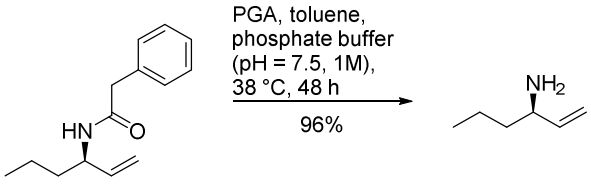
10h, *ee* = 96% **12**

Following a literature procedure,[1] ceric ammonium nitrate (3.64 mmol, 1.99 g) was dissolved in H_2O (15 mL) and cooled to 0 °C. A solution of (*R*)-*N*-(hex-1-en-3-yl)-*N*-(4-methoxyphenyl)-2-phenylacetamide **10h** (0.52 mmol, 170.0 mg, *ee* = 96%) in MeCN (15 mL) was added dropwise and the mixture was stirred at room temperature for 2.5 h. Subsequently, Et_2O (30 mL) was added, the phases were separated and the aqueous phase was extracted with Et_2O (2 x 30 mL). The combined organic phases were dried over Na_2SO_4 and the solvent was removed under reduced pressure. Purification of the residue by column chromatography on deactivated silicagel (petrol ether/ethyl acetate = 7/1 + 3% NEt_3) yielded **12** as a white solid (0.18 mmol, 39.5 mg, 35%).

$C_{14}H_{19}NO$, MW: 217.31 g mol^{-1}. **Mp:** 69.2-70.2 °C. **$[\alpha]_D^{20}$:** −9.3 (*c* = 0.42, $CHCl_3$). **^{1}H-NMR (300 MHz, $CDCl_3$):** δ = 7.40-7.25 (*m*, 5 H, C_6H_5), 5.74-5.61 (*m*, 1 H, C*H*=CH_2), 5.22-5.11 (*bs*, 1 H, N*H*), 5.04-4.94 (*m*, 2 H, CH=CH_2), 4.51-4.39 (*m*, 1 H, C*H*NH), 3.60 (*s*, 2 H, $C_6H_5CH_2$), 1.50-1.17 (*m*,

4 H, CH_2CH_2CH$_3$), 0.86 (*t*, *J* = 7.2, 3 H, CH$_2$CH$_2$CH_3). **^{13}C-NMR (125 MHz, CDCl$_3$):** δ = 170.2, 138.3, 135.0, 129.5, 129.1, 127.4, 114.4, 51.0, 44.1, 36.9, 18.8, 13.8. **IR (CDCl$_3$):** $\tilde{\nu}$ = 3286, 2957, 2931, 1647, 1542, 921, 703. **MS (ESI)** ***m/z:*** 240.1 (100%, [M+Na]$^+$), 226.9 (3%), 218.2 (9%, [M+H]$^+$), 136.1 (1%). **HRMS (ESI)** ***m/z*****:** calculated for C$_{14}$H$_{19}$NO + Na: 240.1359; found: 240.1369.

(*R*)-Hex-1-en-3-amine (13)



12 **13**, *ee* = 97%

(*R*)-N-(Hex-1-en-3-yl)-2-phenylacetamide **12** (92.0 µmol, 20.0 mg) was suspended in sodium phosphate buffer (1.0 mL, pH = 7.5, 1 M) and PGA (*Penicillin G Amidase* from *Escherichia coli*) (ammonium sulfate suspension, ≥10 units/mg protein) (30 µL) and toluene (0.1 mL) were added. The flask was closed and the mixture was stirred at 38 °C for 48 h. Subsequently, H_2O (5 mL) and CH_2Cl_2 (5 mL) were added, the phases were separated and the aqueous phase was extracted with CH_2Cl_2 (2 x 5 mL). The combined organic phases were dried over Na_2SO_4 and the solvent removed under reduced pressure. Mesitylene (10 µL) was added to the crude product as an internal standard followed by $CDCl_3$ (1 mL) to determine conversion and yield by ^{1}H-NMR. The crude product was afterwards dissolved in CH_2Cl_2 (10 mL) and extracted with aqueous HCl (3 x 5 mL, 1 M). The pH value of the combined aqueous phases was adjusted to pH = 14 by addition of aqueous NaOH (2 M) and the combined aqueous phases were extracted by CH_2Cl_2 (3 x 10 mL). The combined organic phases were dried over Na_2SO_4 and the solvent removed under reduced pressure to yield **13** as a colorless oil (88.7 µmol, 8.8 mg, 96%).

C$_6$H$_{13}$N, MW: 99.18 g mol^{-1}. **^{1}H-NMR (300 MHz, CDCl$_3$):** δ = 5.84-5.70 (*m*, 1 H, C*H*=CH$_2$), 5.13-4.95 (*m*, 2 H, CH=CH_2), 3.32-3.22 (*m*, 1 H, NH$_2$C*H*), 1.44-1.30 (*m*, 4 H, CH_2CH_2CH$_3$), 1.25 (*bs*, 2 H, NH_2), 0.91 (*t*, *J* = 7.1, 3 H, CH$_2$CH$_2$CH_3). The analytical data are in accordance with the literature.[27]

The enantiomeric excess was determinated by HPLC after tosyl protection of **13**:[28] *Chiracel OD-H*, *n*-hexane/*i*-PrOH (94/6), 1.0 mL, 250 nm, 8.42 min (major enantiomer), 10.10 min (minor enantiomer).

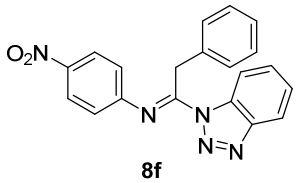
C$_{12}$H$_{17}$NO$_2$S, **MW:** 253.36 g mol^{-1}. **mp:** 55.3-56.3 °C (sample with *ee* = 97). **[α]$_D^{20}$:** −15.1 (c = 0.42, CHCl$_3$, sample with *ee* = 97%). **^{1}H-NMR (300 MHz, CDCl$_3$):** δ = 7.73 (*d*, *J* = 8.4, 2 H, *o*-C$_6$*H*$_4$CH$_3$), 7.27 (*d*, *J* = 8.4, 2 H, *m*-C$_6$*H*$_4$CH$_3$), 5.60-5.47 (*m*, 1 H, C*H*=CH$_2$), 5.02-4.92 (*m*,2 H, CH=CH_2), 4.41 (*bd*, *J* = 8.0, 1 H, N*H*), 3.82-3.70 (*m*, 1 H, NC*H*), 2.42 (*s*, 3 H, C$_6$H$_4$CH_3), 1.49-1.38 (*m*, 2 H,

C*H*$_2$CH$_2$CH$_3$), 1.38-1.17 (*m*, 2 H, CH$_2$C*H*$_2$CH$_3$), 0.83 (*t*, *J* = 7.2, 3 H, CH$_2$CH$_2$C*H*$_3$). The analytical data are in accordance with the literature.[29]

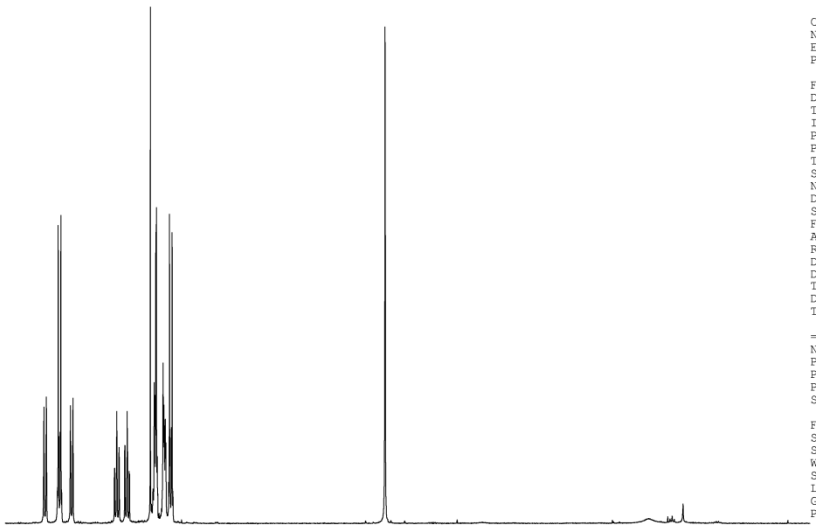
The absolute configuration of *N*-(hex-1-en-3-yl)-4-methylbenzenesulfonamide has already been previously determined.[30] HPLC data and the $[\alpha]_D^{20}$ value were compared to an authentic sample prepared by rearrangement of the corresponding allylic carbamate.[31]

NMR Spectra of New Compounds

(*E*)-1-(1H-Benzo[d][1,2,3]triazol-1-yl)-*N*-(4-nitrophenyl)-2-phenylethan-1-imine (8f)



8f



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RG                   362
DW                80.800 usec
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TE                296.0 K
D1             1.00000000 sec
TD0                    1

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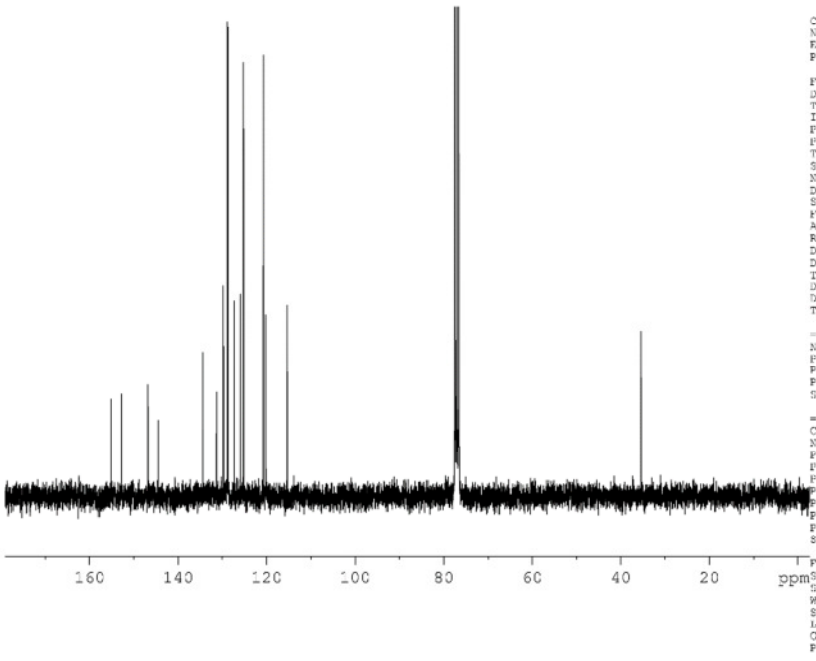

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FIDRES          0.275098 Hz
AQ              1.8175818 sec
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TE                296.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
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P1                11.75 usec
PL1       0 dB
PL1W        31.14739227 W
SFO1        75.4732953 MHz

======== CHANNEL f2 ========
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NUC2                 1H
PCPD2             100.00 usec
PL2               -2.00 dB
PL12              17.03 dB
PL13              19.00 dB
PL2W        37.02396774 W
PL12W        0.46076876 W
PL13W        0.29409182 W
SFO2        300.1312003 MHz

F2 - Processing parameters
SI                 32768
SF           75.4677490 MHz
WDW                   EM
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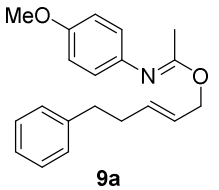
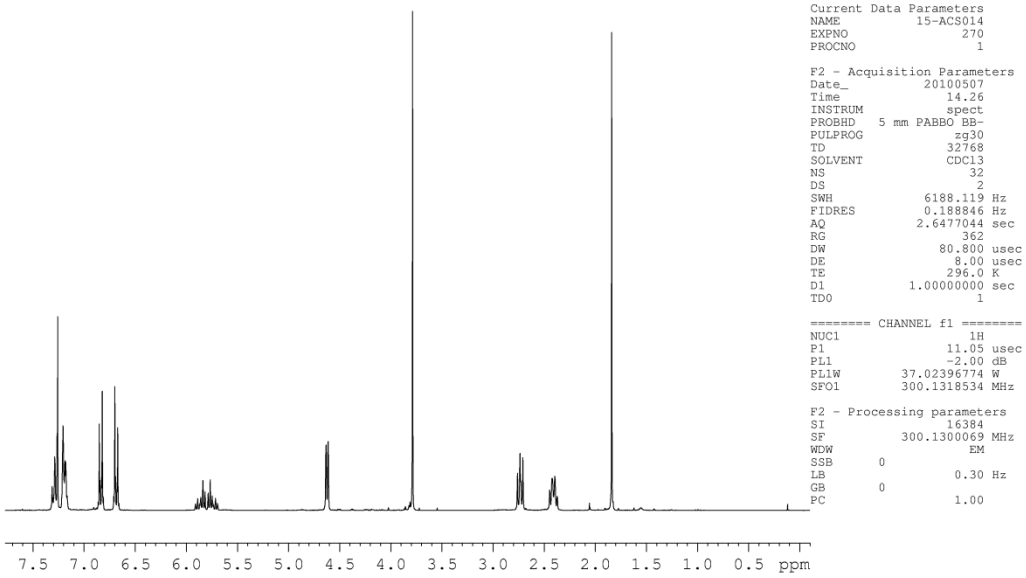
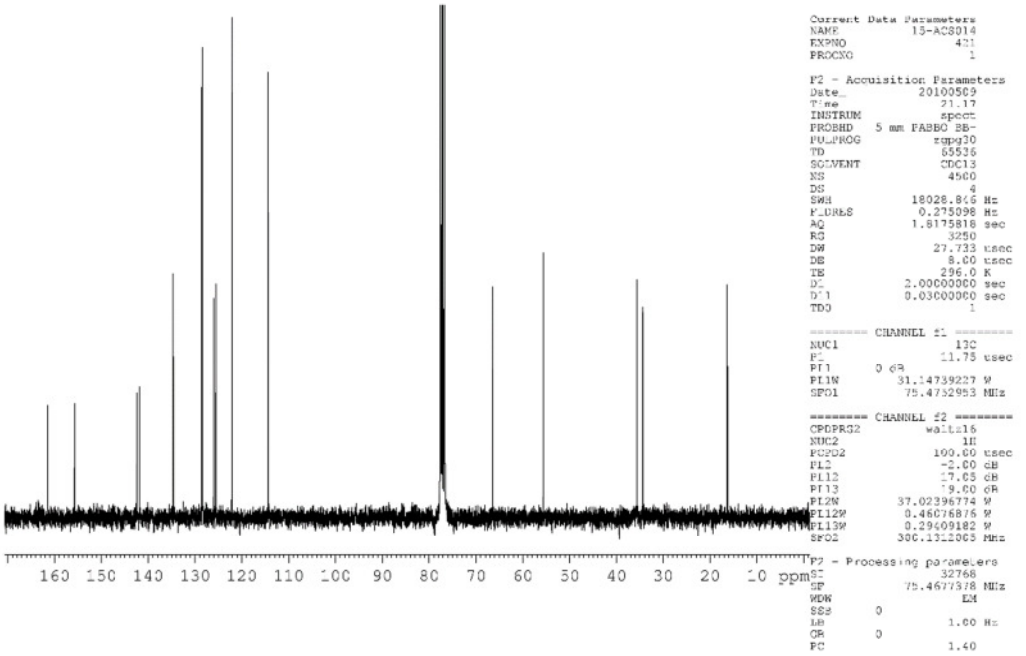
((*E*)-5-Phenylpent-2-en-1-yl)-*N*-(4-methoxyphenyl)acetimidate (9a)

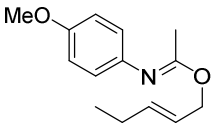


9a

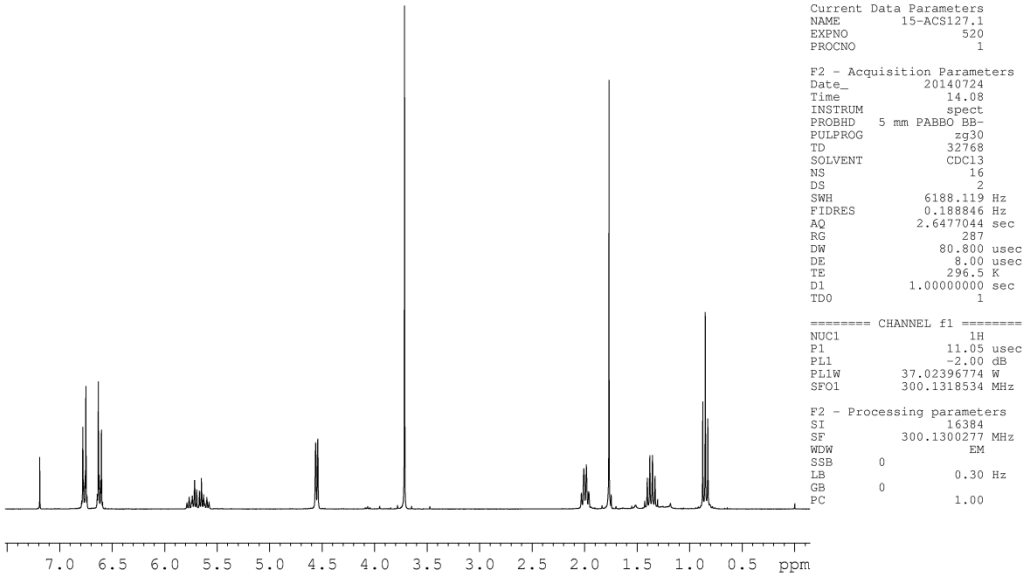


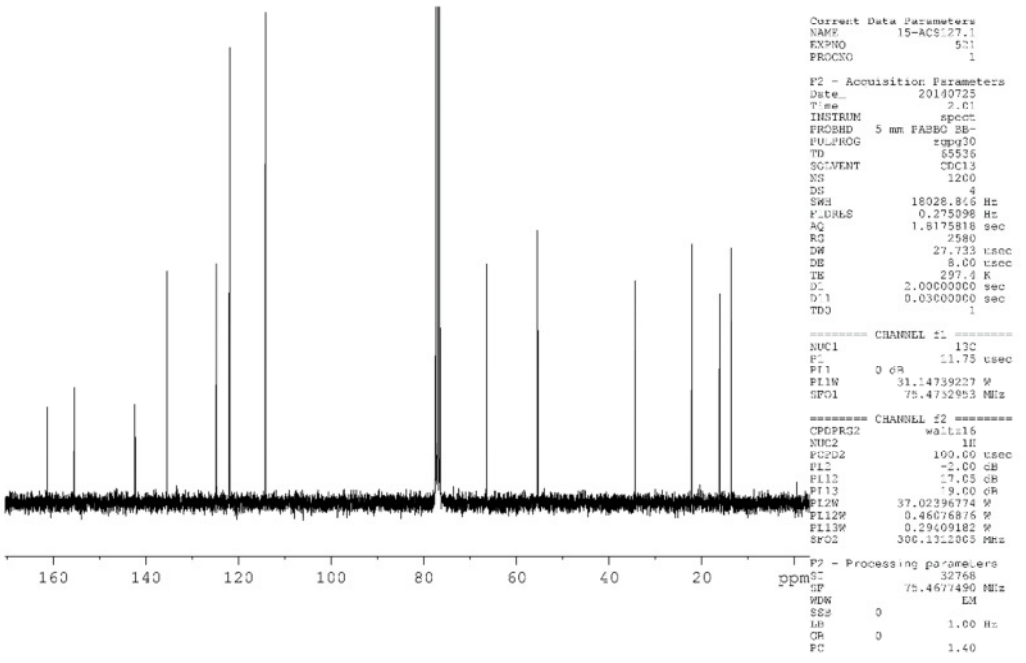


((*E*)-Pent-2-en-1-yl)-*N*-(4-methoxyphenyl)acetimidate (9b)

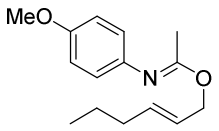


9b





((*E*)-Hex-2-en-1-yl)-*N*-(4-methoxyphenyl)acetimidate (9c)



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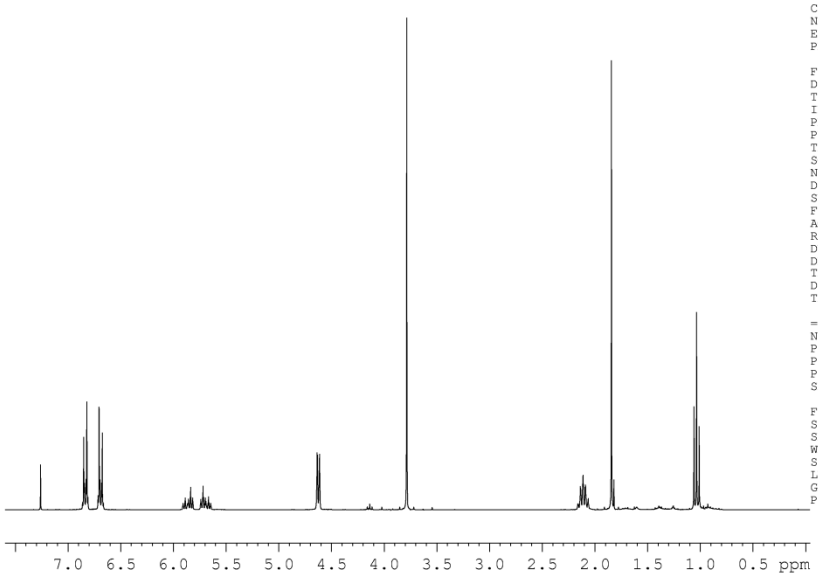


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TD                 32768
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FIDRES          0.188846 Hz
AQ              2.6477044 sec
RG                   256
DW                80.800 usec
DE                 8.00 usec
TE                296.5 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
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PL1W         37.02396774 W
SFO1         300.1318534 MHz

F2 - Processing parameters
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WDW                   EM
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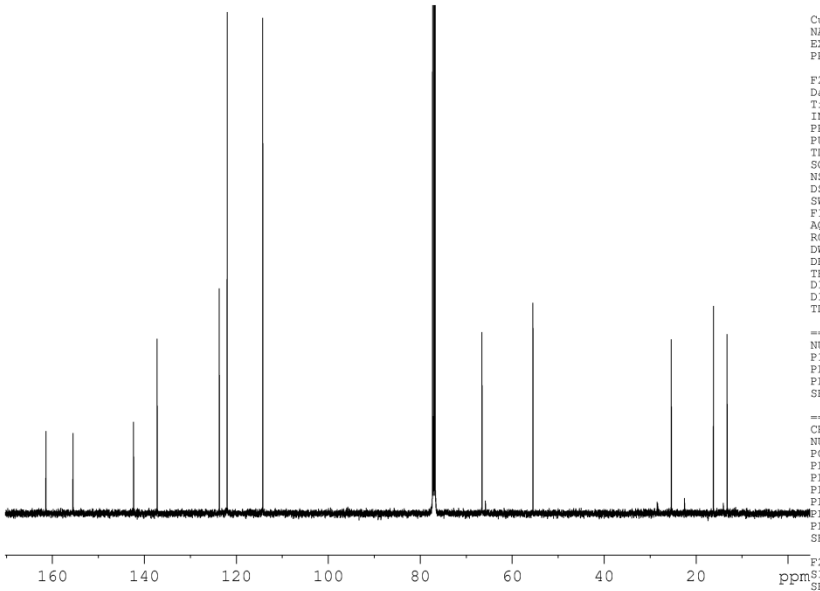


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TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            32894.738 Hz
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AQ              0.9961972 sec
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TE                300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.20 usec
PL1                1.50 dB
PL1W         51.74793243 W
SFO1         125.7761482 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                1.00 dB
PL12              19.99 dB
PL13              21.00 dB
PL2W         19.75309753 W
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F2 - Processing parameters
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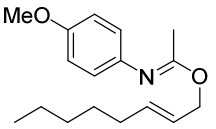
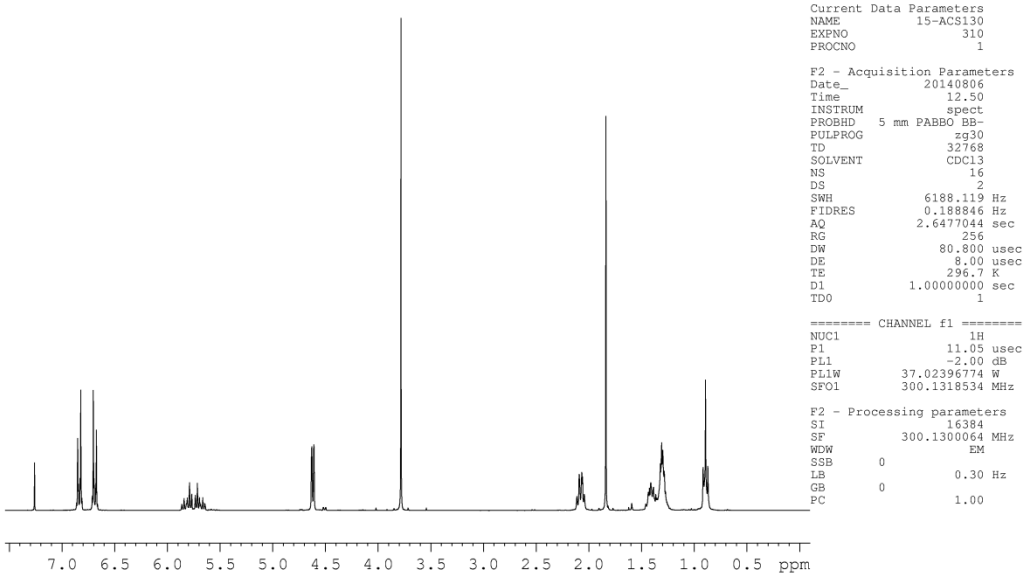
((*E*)-Oct-2-en-1-yl)-*N*-(4-methoxyphenyl)acetimidate (9d)



9d

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PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
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DS                     2
SWH             6188.119 Hz
FIDRES          0.188846 Hz
AQ              2.6477044 sec
RG                   256
DW                80.800 usec
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TE                296.7 K
D1             1.00000000 sec
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PL1W         37.02396774 W
SFO1         300.1318534 MHz

F2 - Processing parameters
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LB                 0.30 Hz
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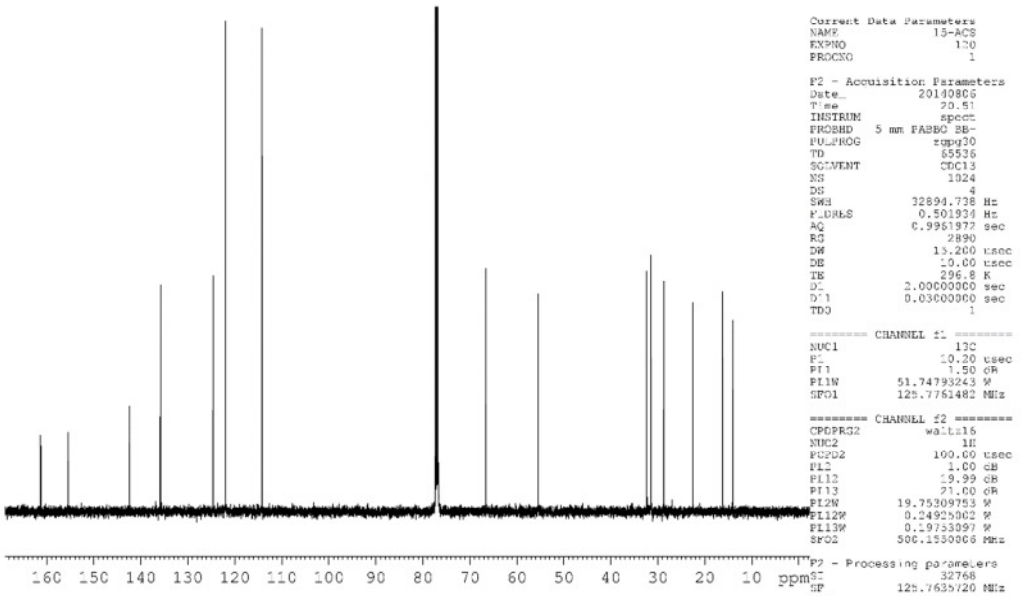


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FIDRES          0.501924 Hz
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RG                  2890
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DE                10.00 usec
TE                296.8 K
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D11            0.03000000 sec
TD0                    1

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SFO1         125.7761482 MHz

======== CHANNEL f2 ========
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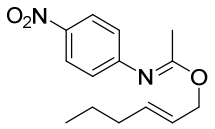
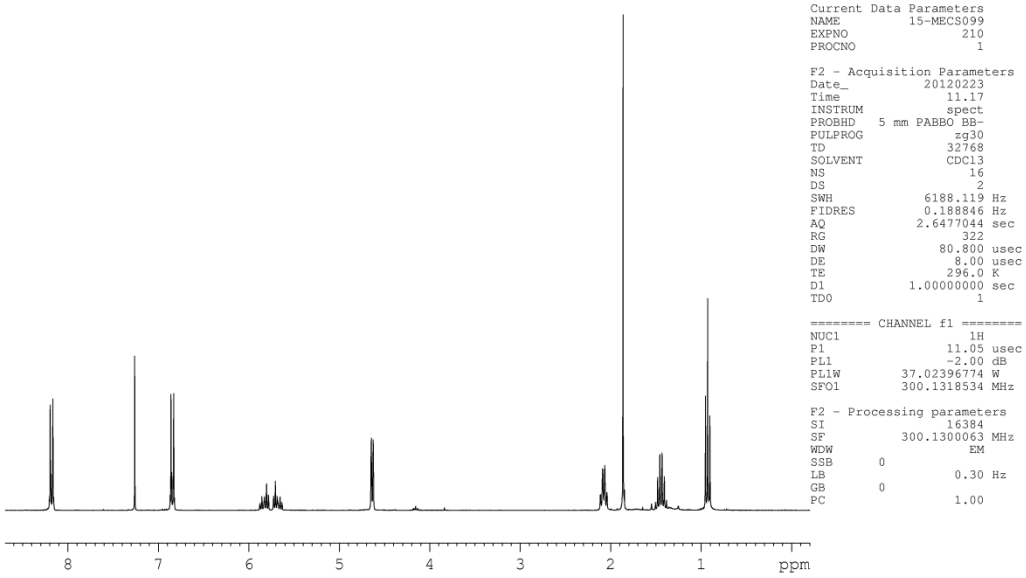
NUC2                  1H
PCPD2              100.00 usec
PL2                1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925002 W
PL13W         0.19753097 W
SFO2         500.1330006 MHz

F2 - Processing parameters
SI                 32768
SF           125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

((*E*)-Hex-2-en-1-yl)-*N*-(4-nitrophenyl)acetimidate (9e)



9e

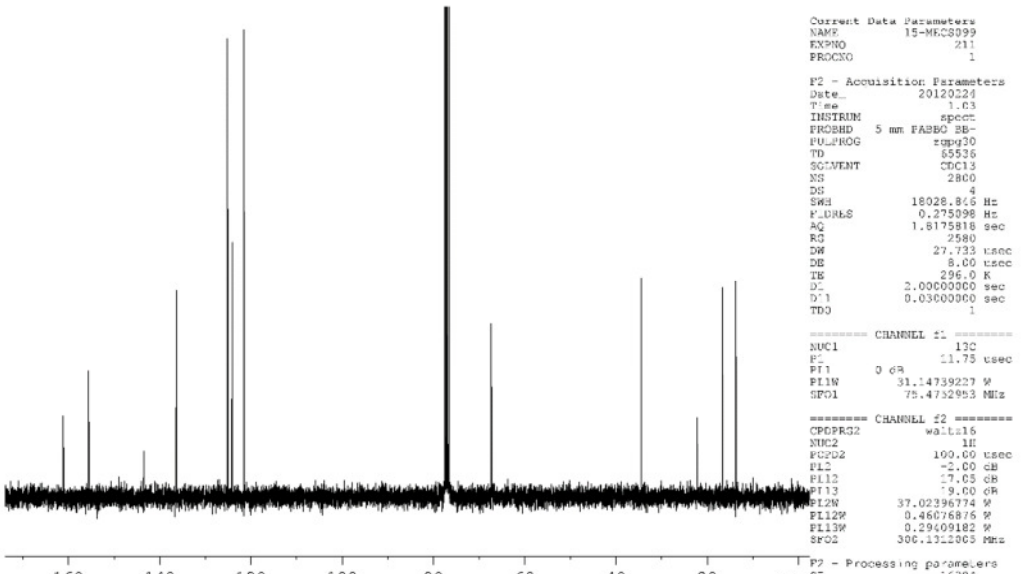



```
Current Data Parameters
NAME          15-MECS099
EXPNO                210
PROCNO                 1

F2 - Acquisition Parameters
Date_          20120223
Time              11.17
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            6188.119 Hz
FIDRES        0.188846 Hz
AQ            2.6477044 sec
RG                  322
DW               80.800 usec
DE                8.00 usec
TE               296.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.05 usec
PL1               -2.00 dB
PL1W        37.02396774 W
SFO1        300.1318534 MHz

F2 - Processing parameters
SI                 16384
SF          300.1300063 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```




```
Current Data Parameters
NAME          15-MECS099
EXPNO                211
PROCNO                 1

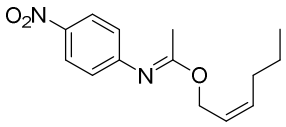
F2 - Acquisition Parameters
Date_          20120224
Time              1.03
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2800
DS                    4
SWH           18028.846 Hz
FIDRES        0.275098 Hz
AQ            1.8175818 sec
RG                 2580
DW               27.733 usec
DE                8.00 usec
TE               296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                11.75 usec
PL1       0 dB
PL1W        31.14739227 W
SFO1        75.4752953 MHz

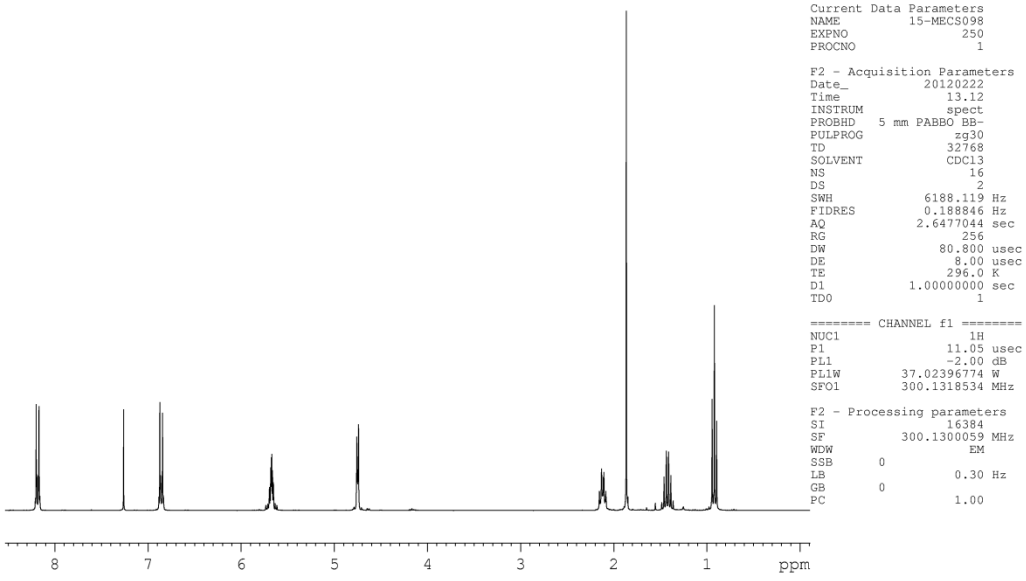
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               -2.00 dB
PL12              17.05 dB
PL13              19.00 dB
PL2W        37.02396774 W
PL12W        0.46076876 W
PL13W        0.29409182 W
SFO2        300.1312005 MHz

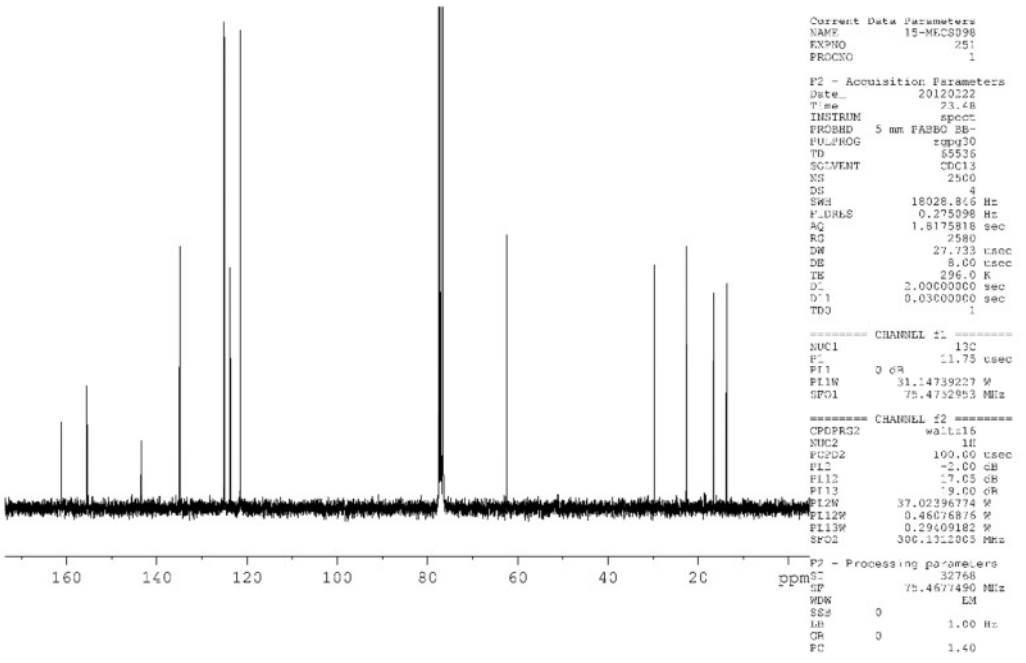
F2 - Processing parameters
SI                 16384
SF          75.4677490 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                1.40
```

((*Z*)-Hex-2-en-1-yl)-*N*-(4-nitrophenyl)acetimidate ((*Z*)-9e)

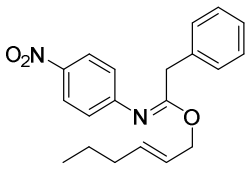


(*Z*)-9e

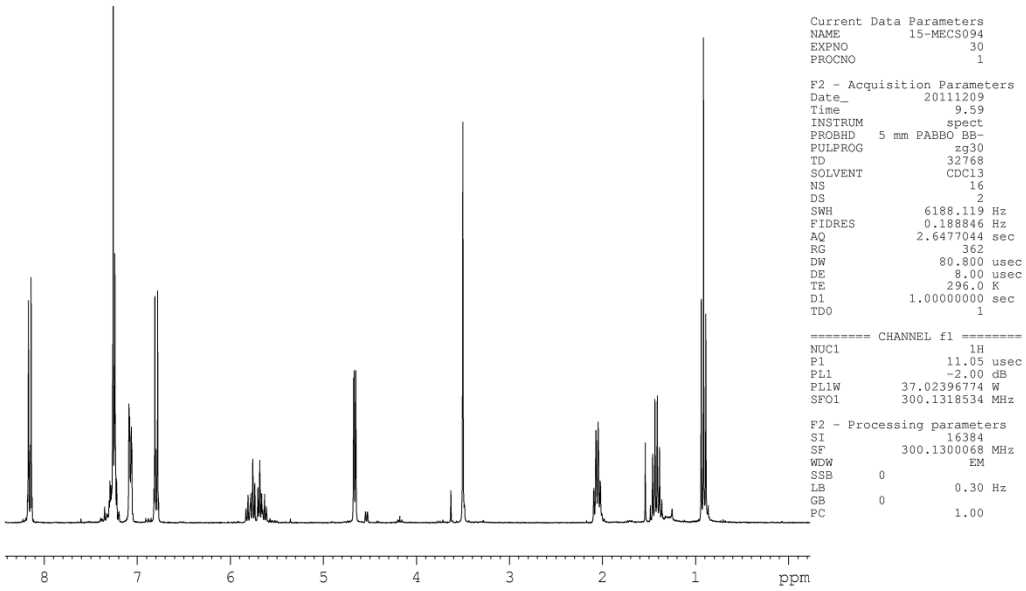


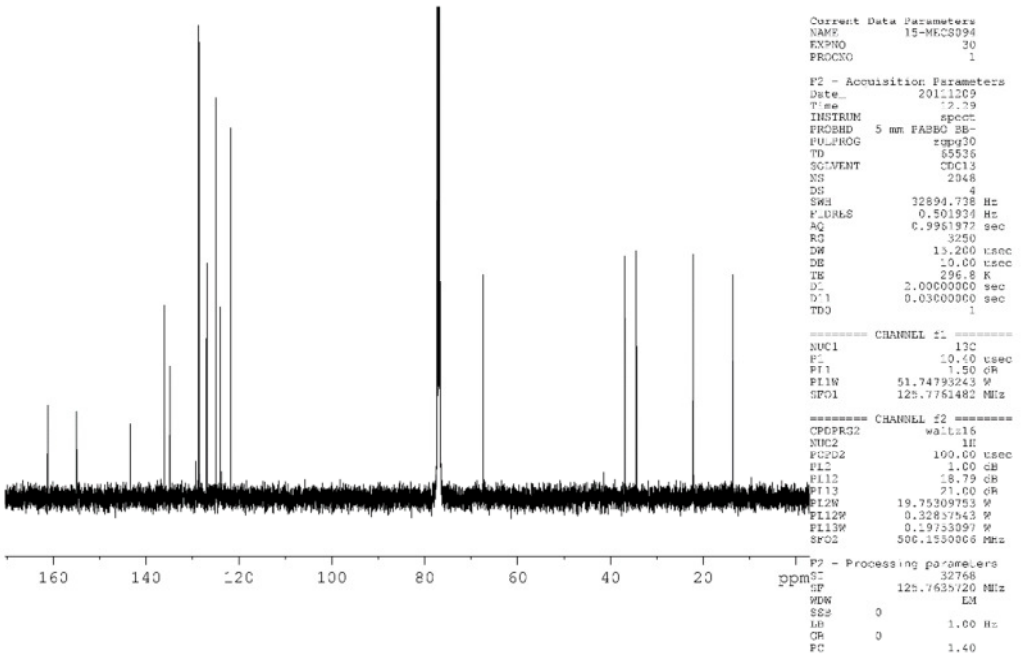


((*E*)-Hex-2-en-1-yl)-*N*-(4-nitrophenyl)-2-phenylacetimidate (9f)

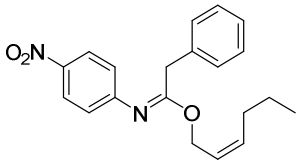


9f

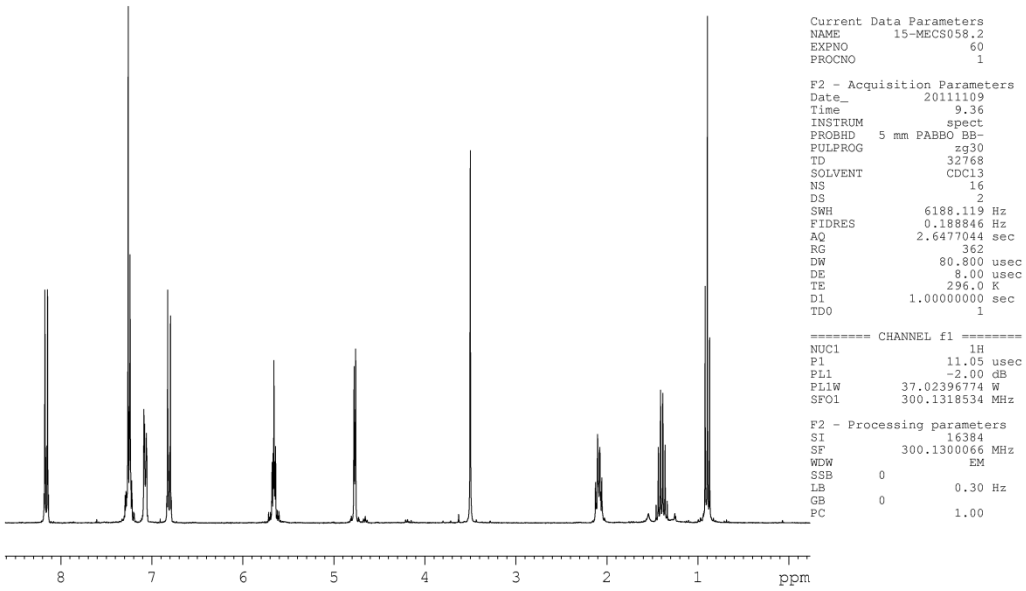


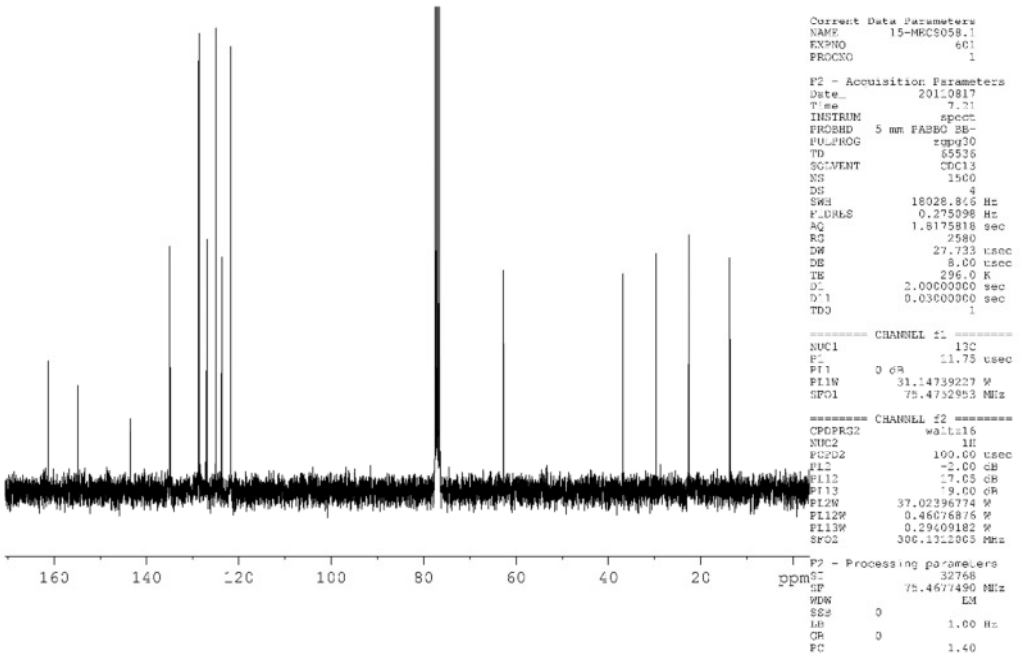


((*Z*)-Hex-2-en-1-yl)-*N*-(4-nitrophenyl)-2-phenylacetimidate ((*Z*)-9f)

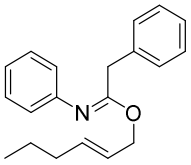


(*Z*)-9f

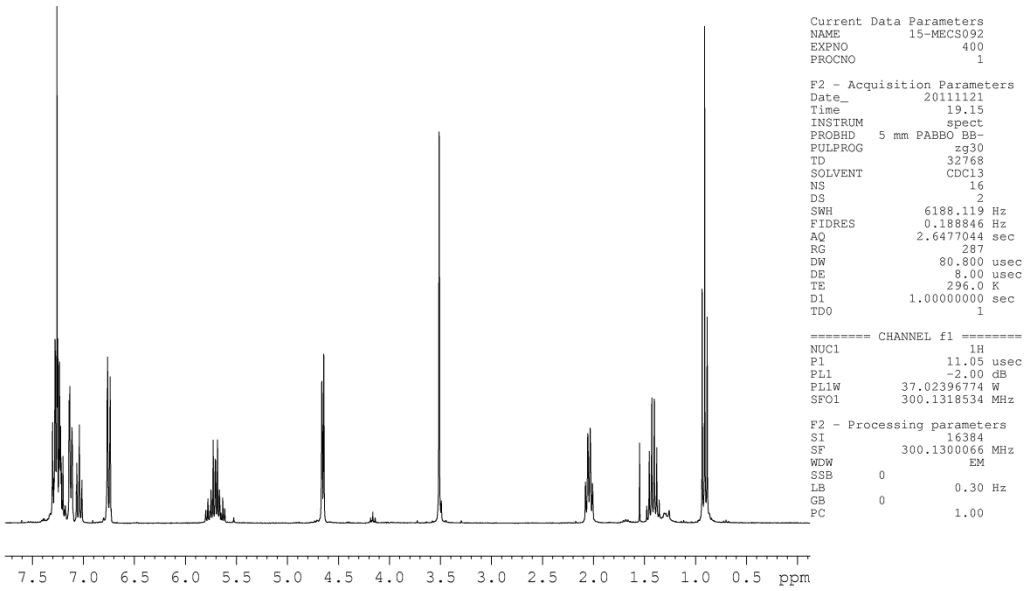




((*E*)-Hex-2-en-1-yl)-*N*-phenyl-2-phenylacetimidate (9g)



9g

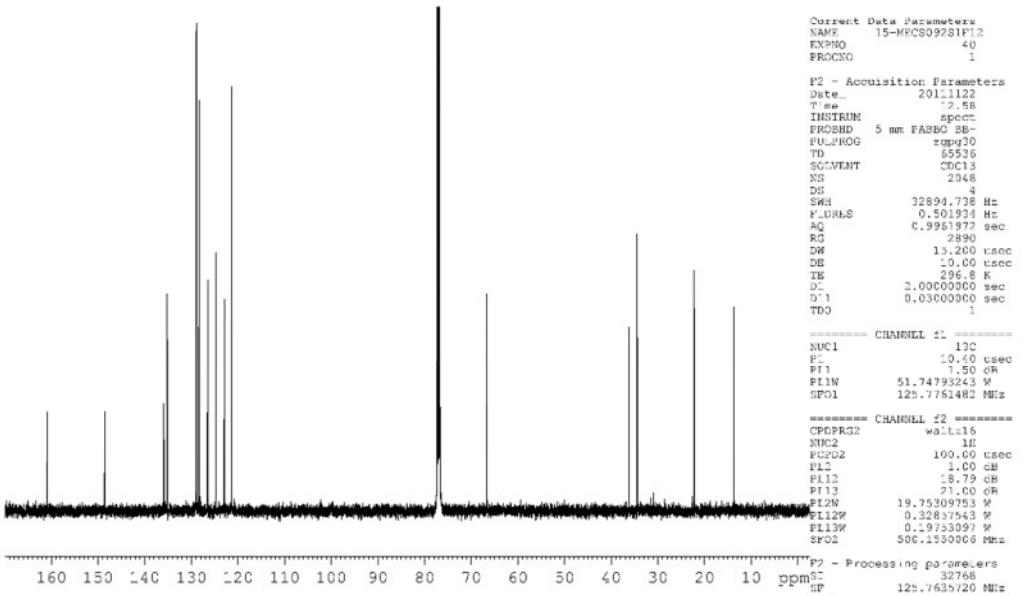



```
Current Data Parameters
NAME          15-MECS092
EXPNO              400
PROCNO               1

F2 - Acquisition Parameters
Date_          20111121
Time             19.15
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG          zg30
TD              32768
SOLVENT          CDCl3
NS                 16
DS                  2
SWH          6188.119 Hz
FIDRES       0.188846 Hz
AQ           2.6477044 sec
RG                287
DW             80.800 usec
DE              8.00 usec
TE             296.0 K
D1          1.00000000 sec
TD0                 1

======== CHANNEL f1 ========
NUC1               1H
P1              11.05 usec
PL1             -2.00 dB
PL1W       37.02396774 W
SFO1       300.1318534 MHz

F2 - Processing parameters
SI              16384
SF         300.1300066 MHz
WDW                EM
SSB       0
LB              0.30 Hz
GB        0
PC              1.00
```




```
Current Data Parameters
NAME      15-MECS092SIP12
EXPNO              40
PROCNO              1

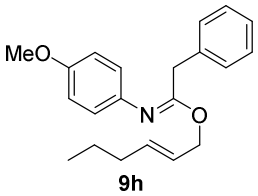
F2 - Acquisition Parameters
Date_          20111122
Time             12.58
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD              65536
SOLVENT          CDCl3
NS                2048
DS                   4
SWH          32894.738 Hz
FIDRES       0.501924 Hz
AQ           0.9951977 sec
RG                2890
DW             15.200 usec
DE              10.00 usec
TE             296.8 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                 1

======== CHANNEL f1 ========
NUC1               13C
P1              10.40 usec
PL1              1.50 dB
PL1W       51.74793243 W
SFO1       125.7761482 MHz

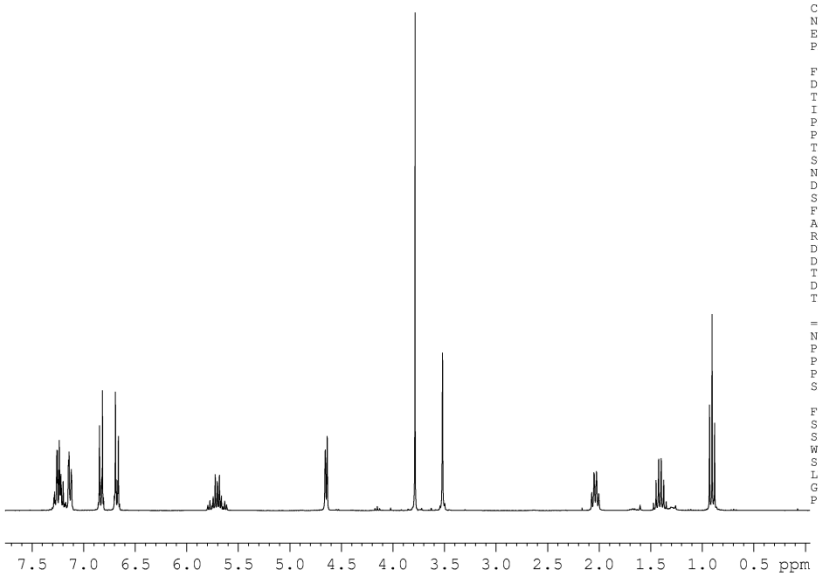
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2          100.00 usec
PL2              1.00 dB
PL12            18.79 dB
PL13            21.00 dB
PL2W       19.75309753 W
PL12W       0.32837543 W
PL13W       0.19753097 W
SFO2       500.1330006 MHz

F2 - Processing parameters
SI              32768
SF         125.7635720 MHz
WDW                EM
SSB       0
LB              1.00 Hz
GB        0
PC              1.40
```

((*E*)-Hex-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate (9h)



9h

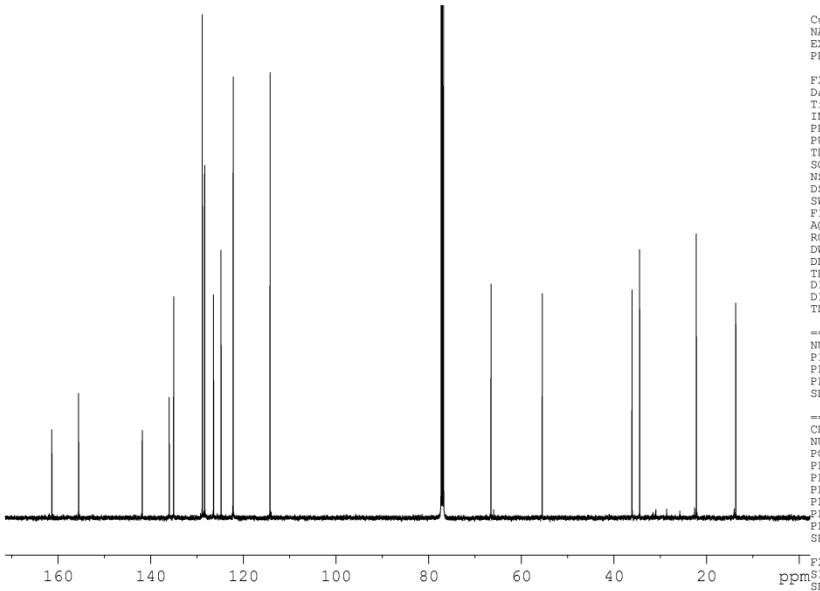



```
Current Data Parameters
NAME          15-MECS033
EXPNO                 30
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110317
Time               10.00
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6188.119 Hz
FIDRES          0.188846 Hz
AQ              2.6477044 sec
RG                   144
DW                80.800 usec
DE                 8.00 usec
TE                296.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.05 usec
PL1                -2.00 dB
PL1W        37.02396774 W
SFO1        300.1318534 MHz

F2 - Processing parameters
SI                 16384
SF          300.1300087 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME      15-MECS033S1F12-21
EXPNO                  110
PROCNO                   1

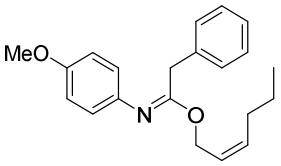
F2 - Acquisition Parameters
Date_           20110317
Time               18.53
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            32894.738 Hz
FIDRES          0.501934 Hz
AQ              0.9961972 sec
RG                  3250
DW                15.200 usec
DE                10.00 usec
TE                297.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.40 usec
PL1                1.50 dB
PL1W        51.74793243 W
SFO1        125.7761482 MHz

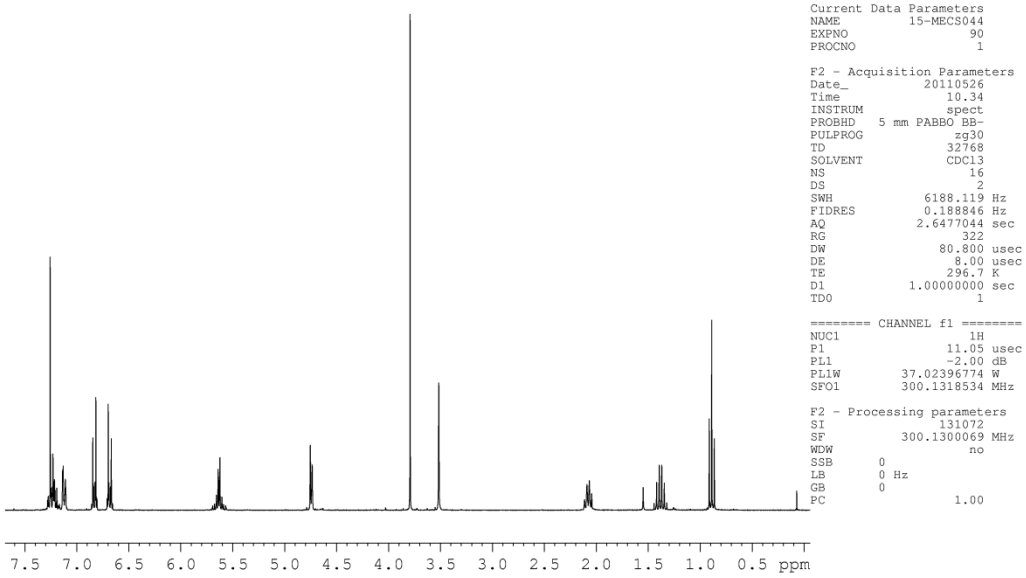
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                1.00 dB
PL12              18.79 dB
PL13              21.00 dB
PL2W        19.75309753 W
PL12W        0.32857543 W
PL13W        0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

((*Z*)-Hex-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate ((*Z*))-9h



(*Z*)-9h

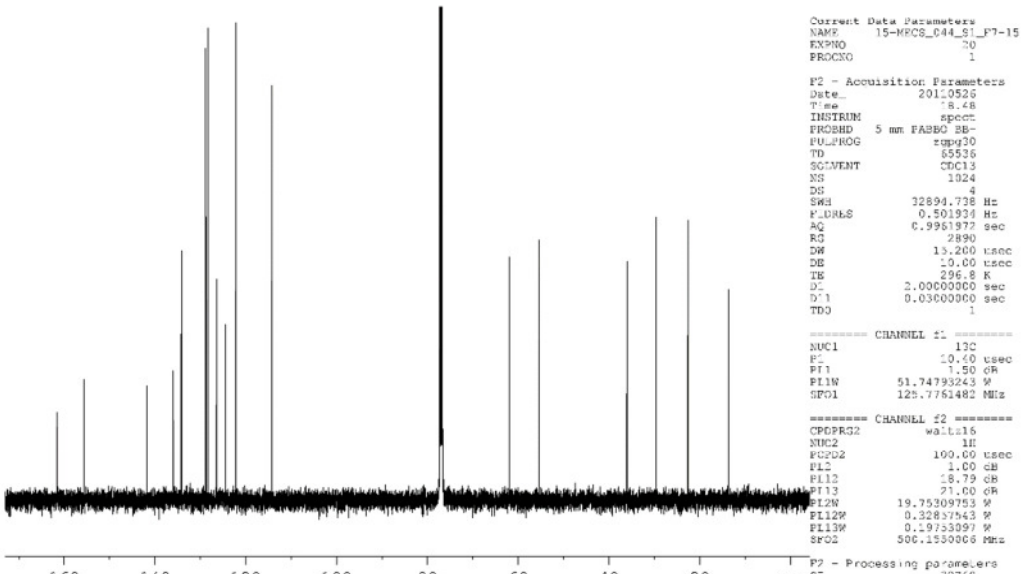



```
Current Data Parameters
NAME          15-MECS044
EXPNO                 90
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110526
Time               10.34
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6188.119 Hz
FIDRES          0.188846 Hz
AQ              2.6477044 sec
RG                   322
DW                80.800 usec
DE                 8.00 usec
TE                296.7 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.05 usec
PL1                -2.00 dB
PL1W         37.02396774 W
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                131072
SF          300.1300069 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```




```
Current Data Parameters
NAME      15-MECS_044_S1_P7-15
EXPNO                   20
PROCNO                   1

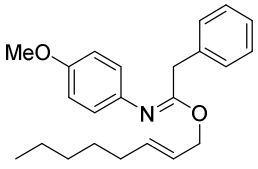
F2 - Acquisition Parameters
Date_           20110526
Time               18.48
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           32894.738 Hz
FIDRES        0.501924 Hz
AQ            0.9951972 sec
RG                 2890
DW               15.200 usec
DE                10.00 usec
TE                296.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.40 usec
PL1                 1.50 dB
PL1W         51.74793243 W
SFO1         125.7761482 MHz

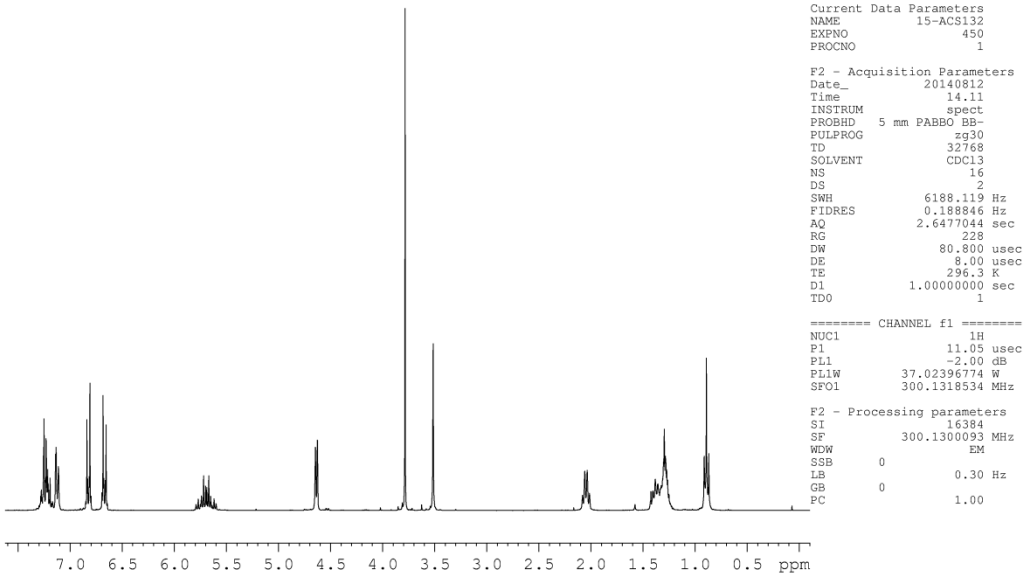
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                 1.00 dB
PL12               18.79 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.32837543 W
PL13W         0.19753097 W
SFO2         500.1330006 MHz

F2 - Processing parameters
SI                 32768
SF          125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                  1.40
```

((*E*)-Oct-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate (9i)



9i

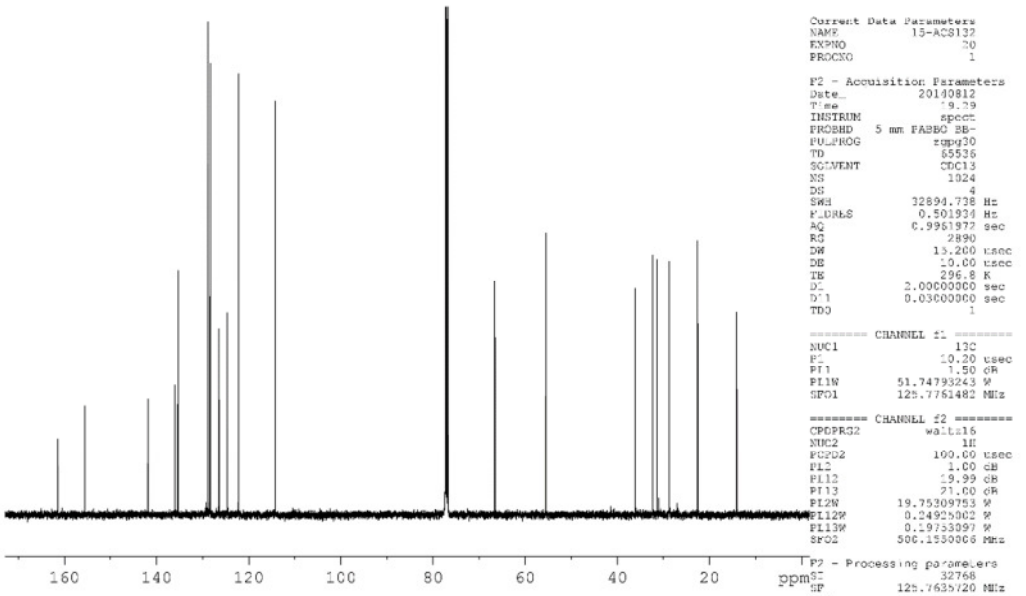



```
Current Data Parameters
NAME           15-ACS132
EXPNO                450
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140812
Time               14.11
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6188.119 Hz
FIDRES          0.188846 Hz
AQ              2.6477044 sec
RG                   228
DW                80.800 usec
DE                 8.00 usec
TE                296.3 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.05 usec
PL1                -2.00 dB
PL1W         37.02396774 W
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                 16384
SF           300.1300093 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME           15-ACS132
EXPNO                 20
PROCNO                 1

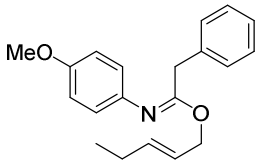
F2 - Acquisition Parameters
Date_           20140812
Time               19.39
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            32894.738 Hz
FIDRES          0.501924 Hz
AQ              0.9961977 sec
RG                  2890
DW                15.200 usec
DE                10.00 usec
TE                296.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W         51.74793243 W
SFO1         125.7761482 MHz

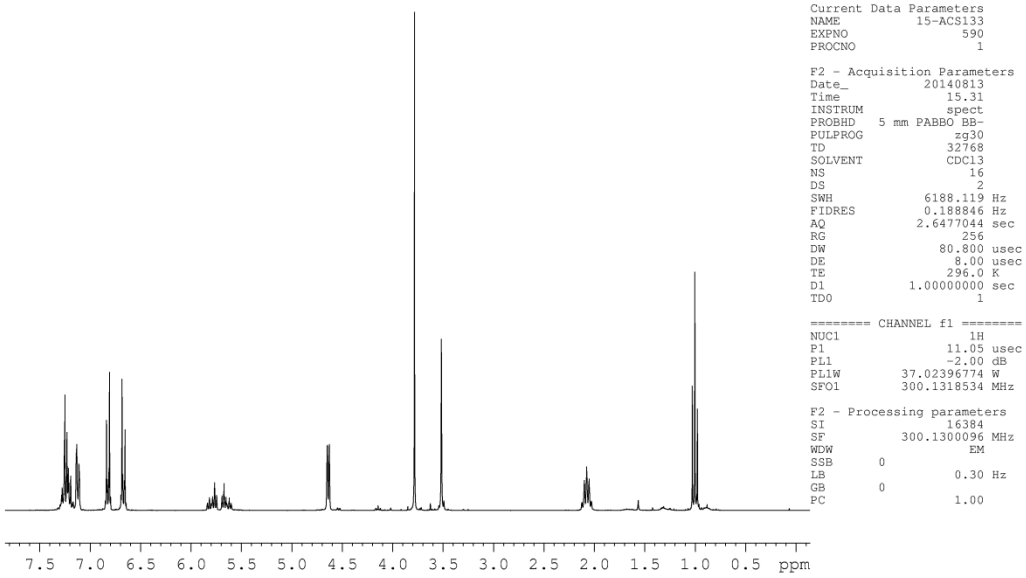
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925062 W
PL13W         0.19753097 W
SFO2         500.1330006 MHz

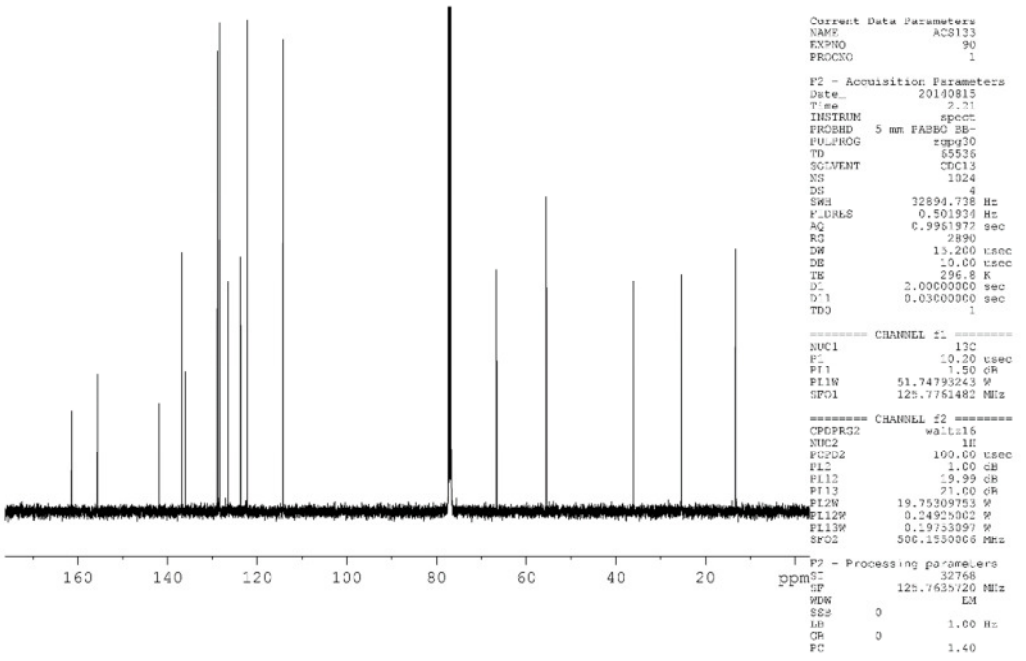
F2 - Processing parameters
SI                 32768
SF           125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

((*E*)-Pent-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate (9j)

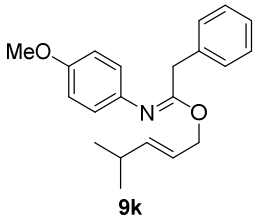


9j

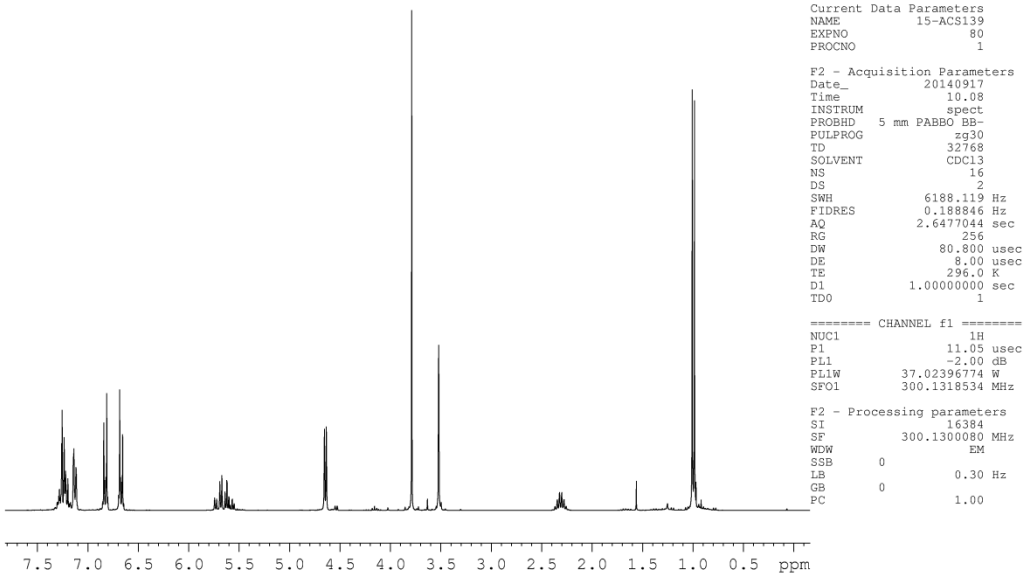


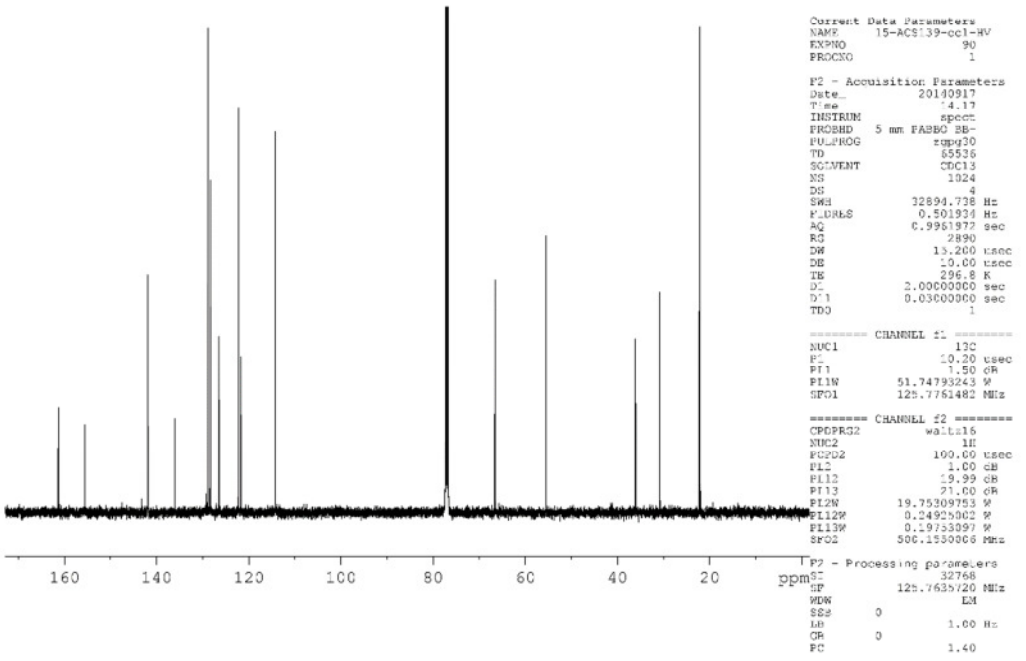


((*E*)-4-Methylpent-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate (9k)

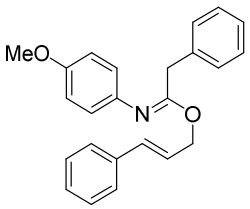


9k

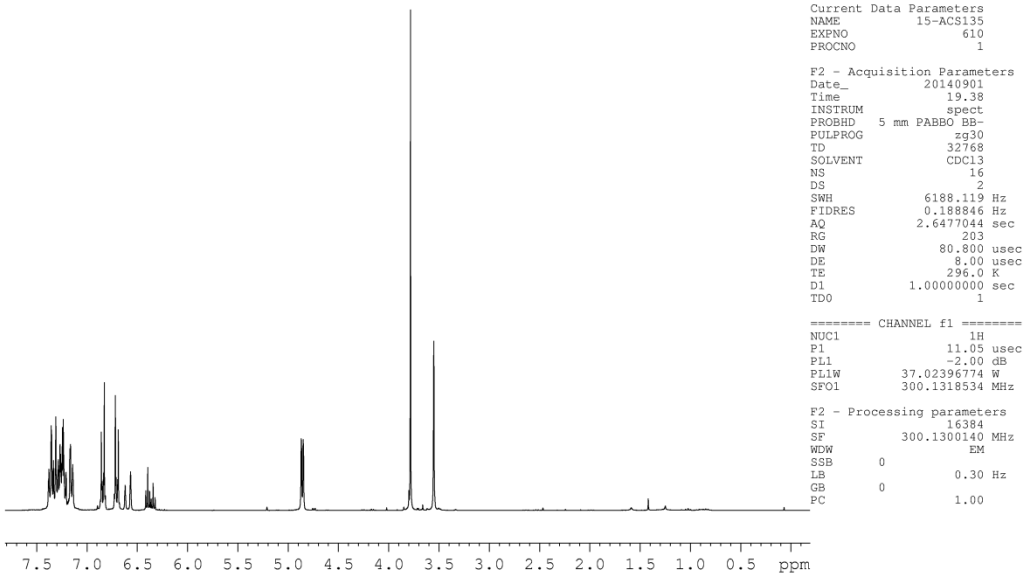




((*E*)-3-Phenylprop-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate (9l)



9l

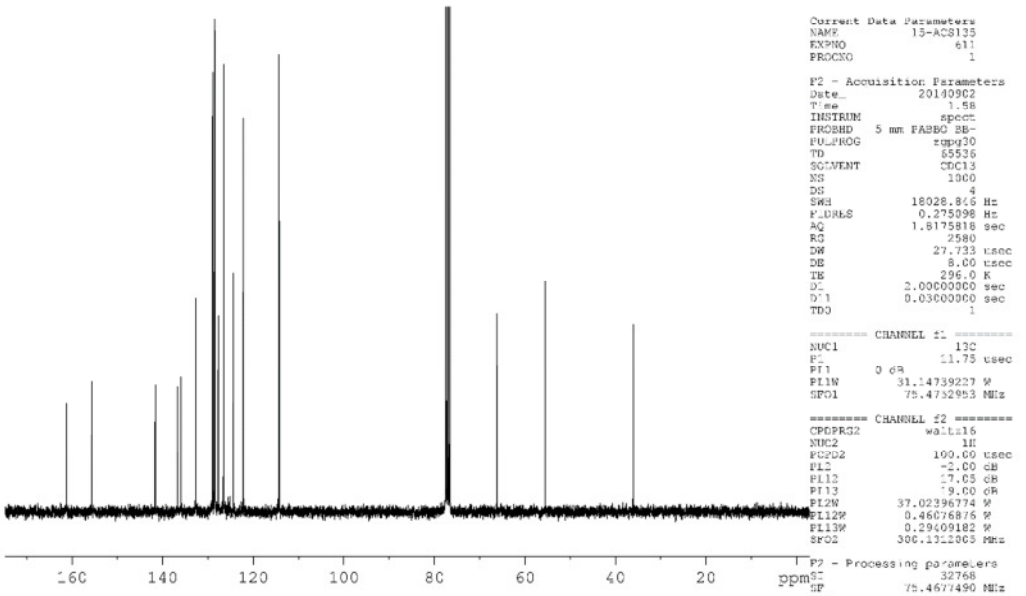



```
Current Data Parameters
NAME           15-ACS135
EXPNO                610
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140901
Time               19.38
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6188.119 Hz
FIDRES          0.188846 Hz
AQ              2.6477044 sec
RG                   203
DW                80.800 usec
DE                 8.00 usec
TE                296.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.05 usec
PL1                -2.00 dB
PL1W         37.02396774 W
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                 16384
SF           300.1300140 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME           15-ACS135
EXPNO                611
PROCNO                 1

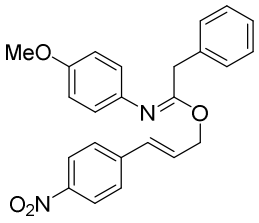
F2 - Acquisition Parameters
Date_           20140902
Time               1.58
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1000
DS                     4
SWH            18028.846 Hz
FIDRES          0.275098 Hz
AQ              1.8175818 sec
RG                  2580
DW                27.733 usec
DE                 8.00 usec
TE                296.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                11.75 usec
PL1       0 dB
PL1W         31.14739227 W
SFO1         75.4752953 MHz

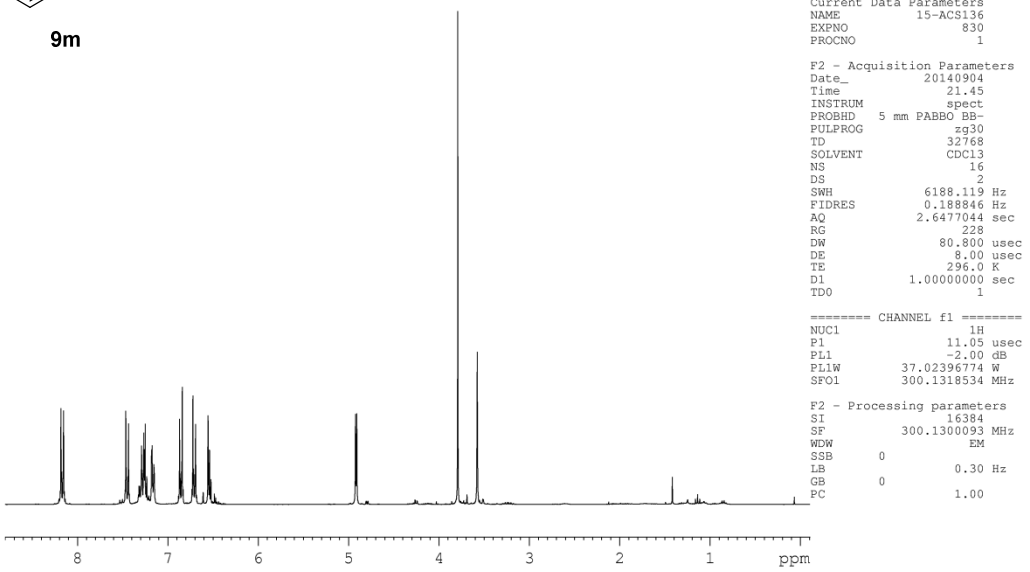
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                -2.00 dB
PL12               17.03 dB
PL13               19.00 dB
PL2W         37.02396774 W
PL12W         0.46076876 W
PL13W         0.29409182 W
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF           75.4677490 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

((*E*)-3-(4-Nitrophenylprop-2-en-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate (9m)



9m

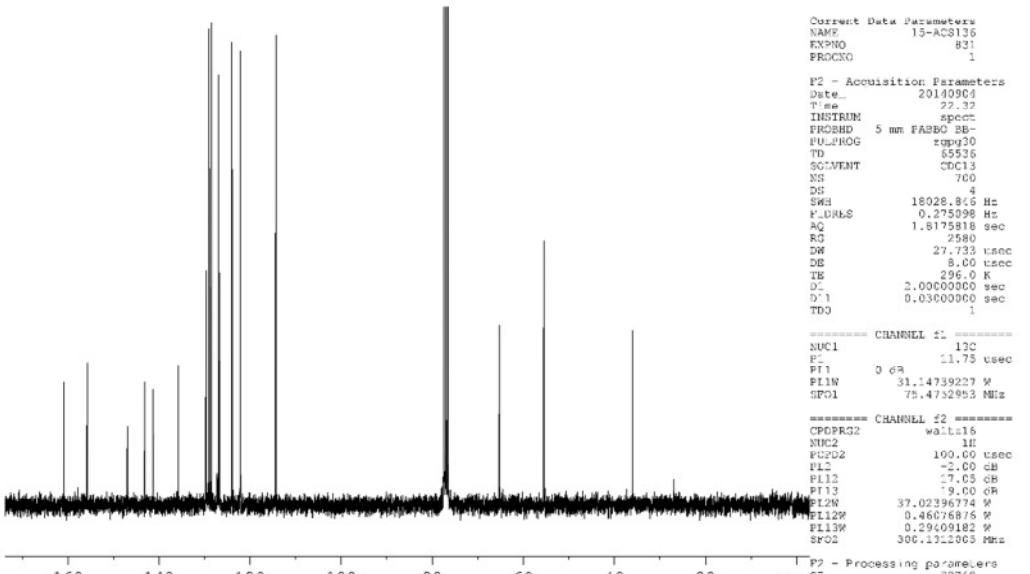



```
Current Data Parameters
NAME           15-ACS136
EXPNO                830
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140904
Time               21.45
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6188.119 Hz
FIDRES          0.188846 Hz
AQ              2.6477044 sec
RG                   228
DW                80.800 usec
DE                 8.00 usec
TE                296.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.05 usec
PL1                -2.00 dB
PL1W         37.02396774 W
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                 16384
SF           300.1300093 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME           15-ACS136
EXPNO                831
PROCNO                 1

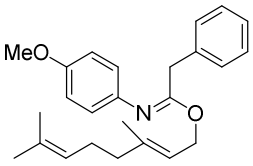
F2 - Acquisition Parameters
Date_           20140904
Time               22.32
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   700
DS                     4
SWH            18028.846 Hz
FIDRES          0.275098 Hz
AQ              1.8175818 sec
RG                  2580
DW                27.733 usec
DE                 8.00 usec
TE                296.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 11.75 usec
PL1       0 dB
PL1W         31.14739227 W
SFO1         75.4752953 MHz

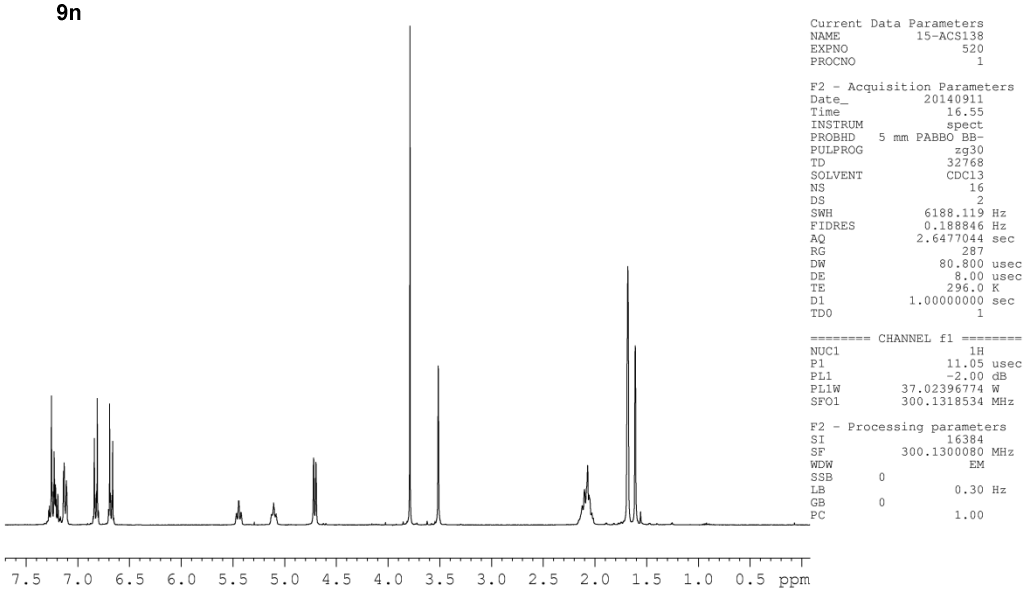
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                -2.00 dB
PL12               17.05 dB
PL13               19.00 dB
PL2W         37.02396774 W
PL12W         0.46076876 W
PL13W         0.29409182 W
SFO2         300.1312005 MHz

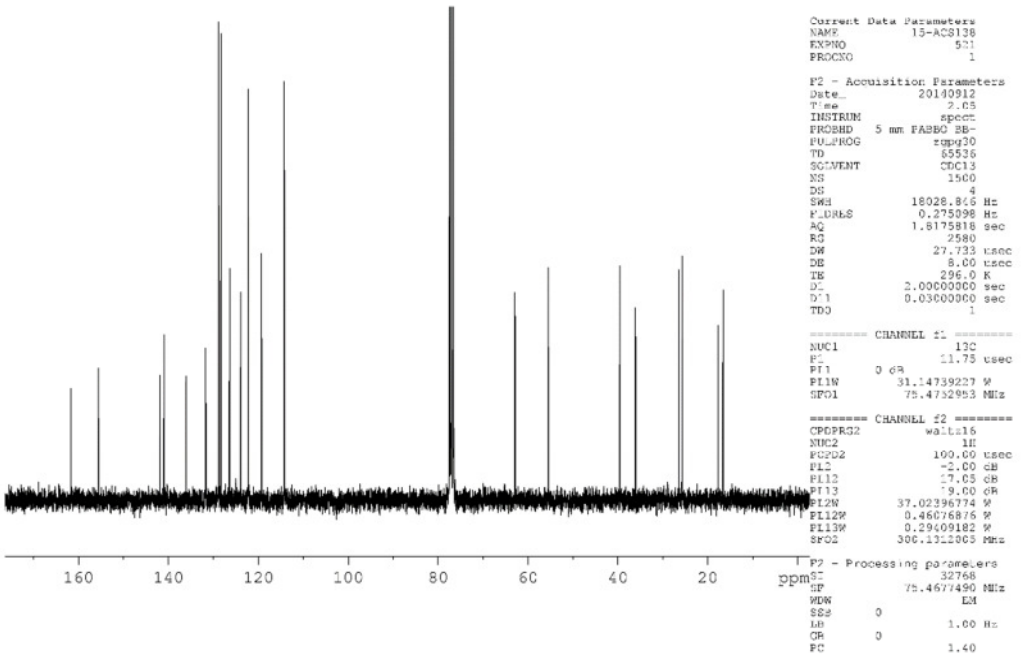
F2 - Processing parameters
SI                 32768
SF           75.4677490 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

((*E*)-3,7-Dimethylocta-2,6-dien-1-yl)-*N*-(4-methoxyphenyl)-2-phenylacetimidate (9n)

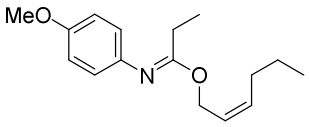


9n

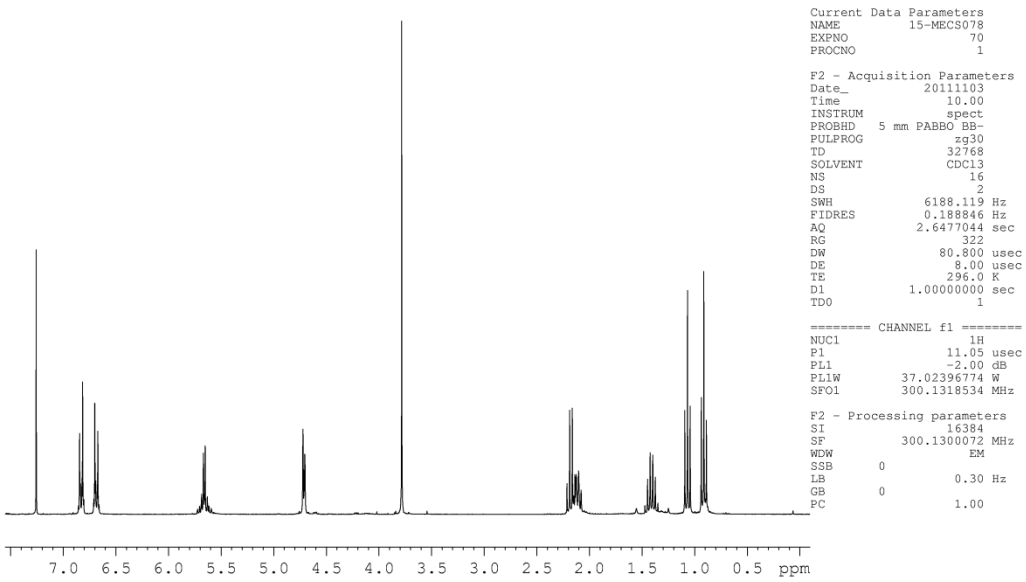


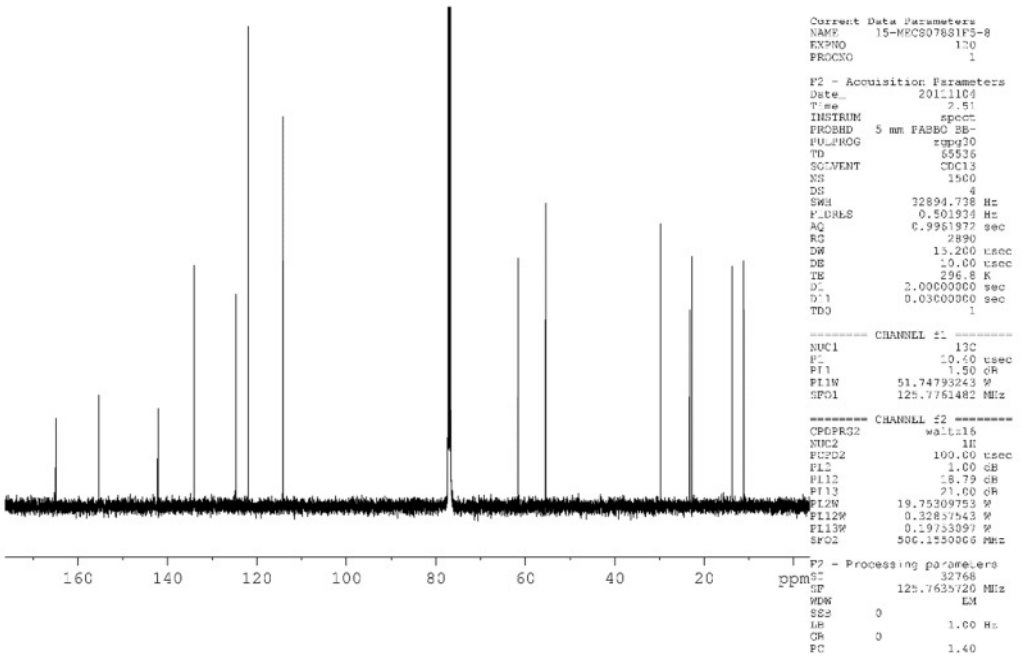


((*Z*)-Hex-2-en-1-yl)-*N*-(4-methoxyphenyl)propionimidate ((*Z*)-9o)

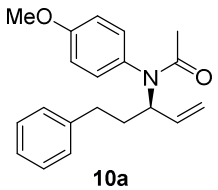


(*Z*)-9o

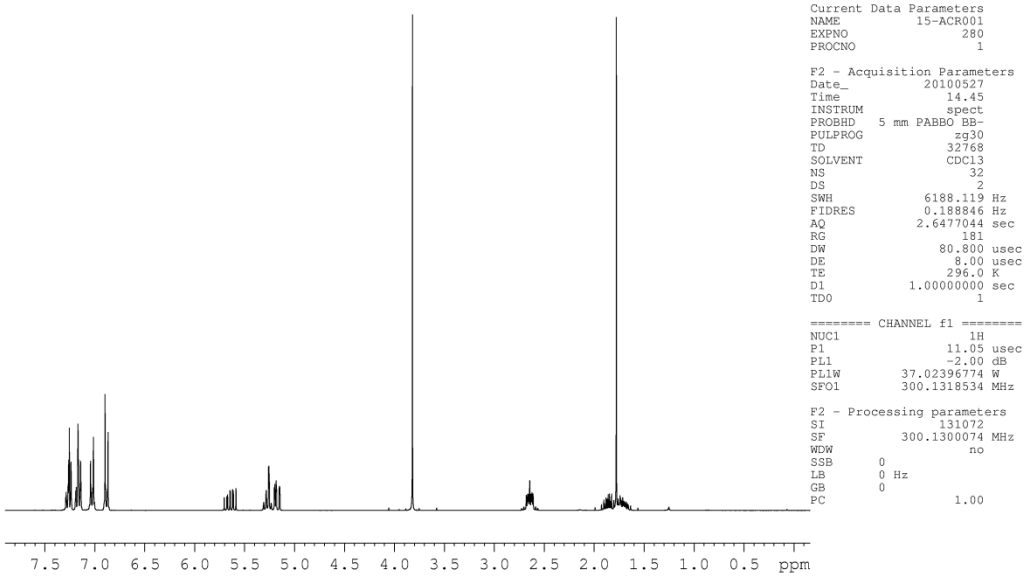




(*R*)-*N*-(4-Methoxyphenyl)-*N*-(5-phenylpent-1-en-3-yl)acetamide (10a)



10a

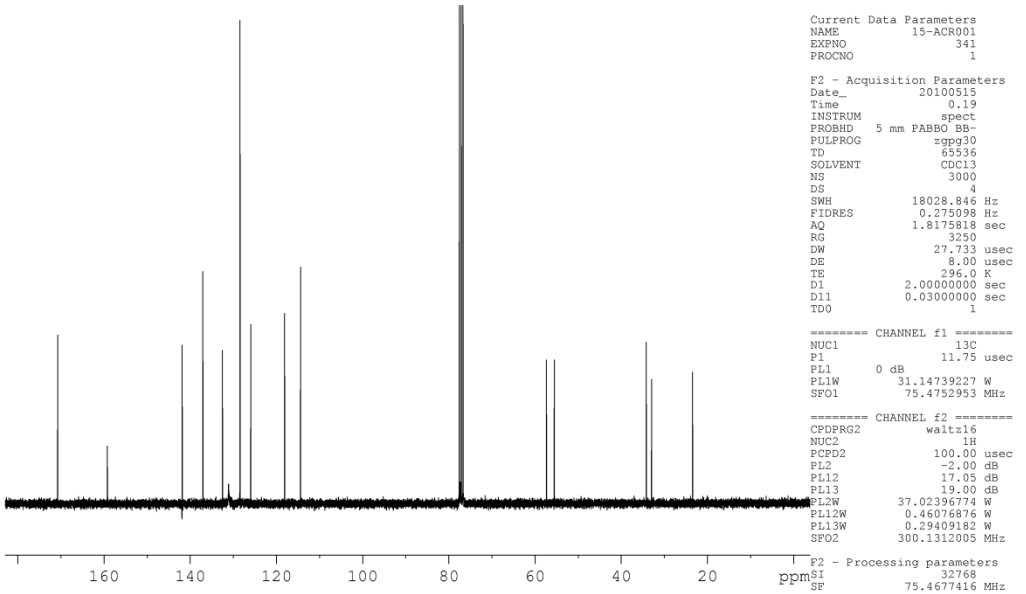



```
Current Data Parameters
NAME           15-ACR001
EXPNO                280
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100527
Time               14.45
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     2
SWH             6188.119 Hz
FIDRES          0.188846 Hz
AQ              2.6477044 sec
RG                   181
DW                80.800 usec
DE                 8.00 usec
TE                296.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.05 usec
PL1                -2.00 dB
PL1W         37.02396774 W
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                131072
SF          300.1300074 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```




```
Current Data Parameters
NAME           15-ACR001
EXPNO                341
PROCNO                 1

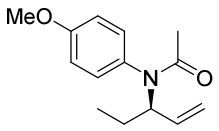
F2 - Acquisition Parameters
Date_           20100515
Time                0.19
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3000
DS                     4
SWH            18028.846 Hz
FIDRES          0.275098 Hz
AQ              1.8175818 sec
RG                  3250
DW                27.733 usec
DE                 8.00 usec
TE                296.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 11.75 usec
PL1        0 dB
PL1W         31.14739227 W
SFO1         75.4752953 MHz

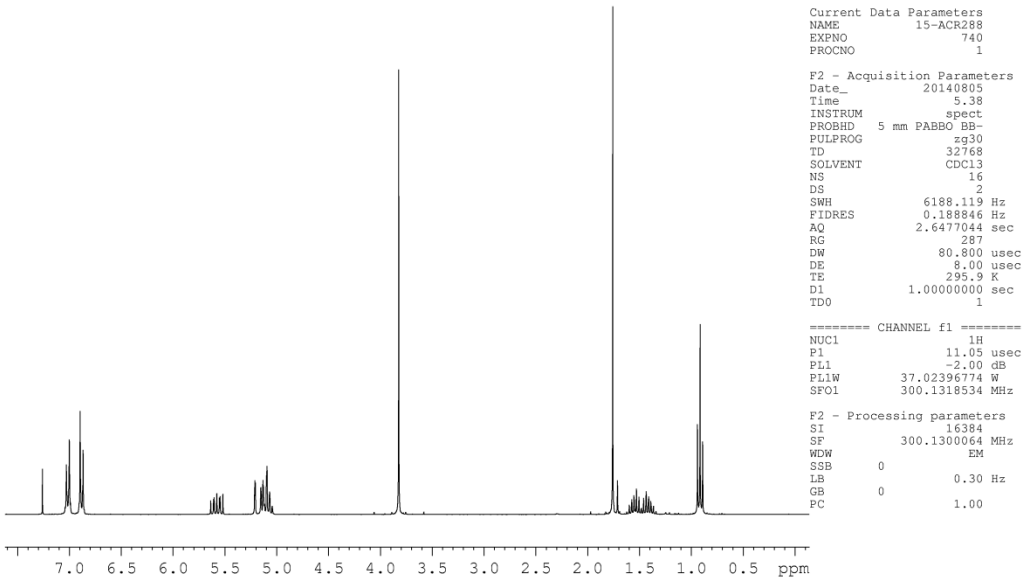
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                -2.00 dB
PL12               17.05 dB
PL13               19.00 dB
PL2W         37.02396774 W
PL12W         0.46076876 W
PL13W         0.29409182 W
SFO2         300.1312005 MHz

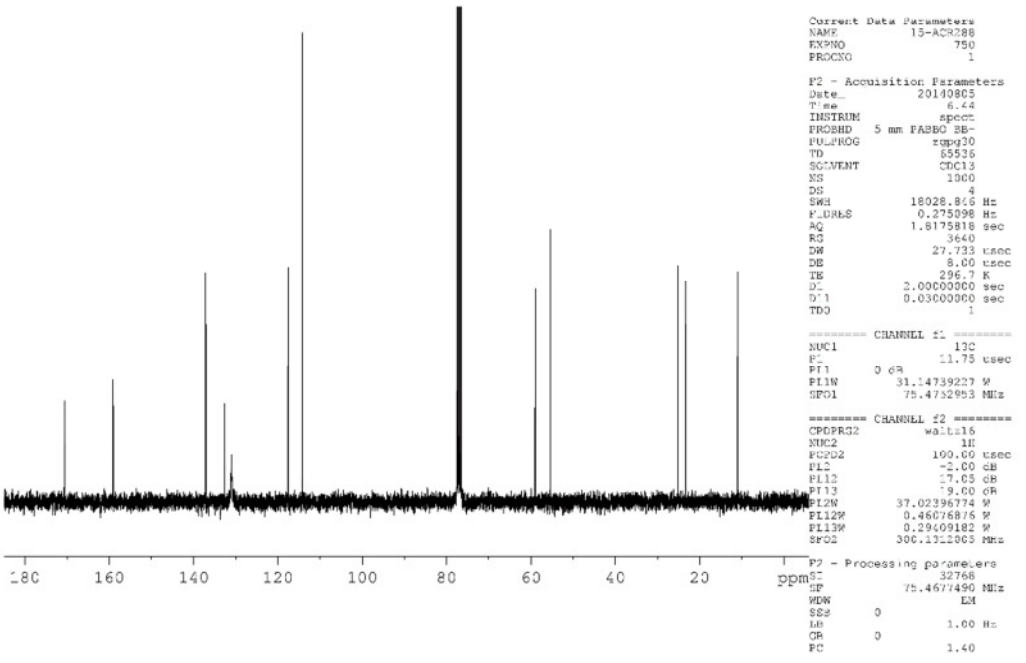
F2 - Processing parameters
SI                 32768
SF          75.4677416 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                  1.40
```

(*R*)-*N*-(4-Methoxyphenyl)-*N*-(pent-1-en-3-yl)acetamide (10b)

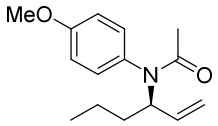


10b

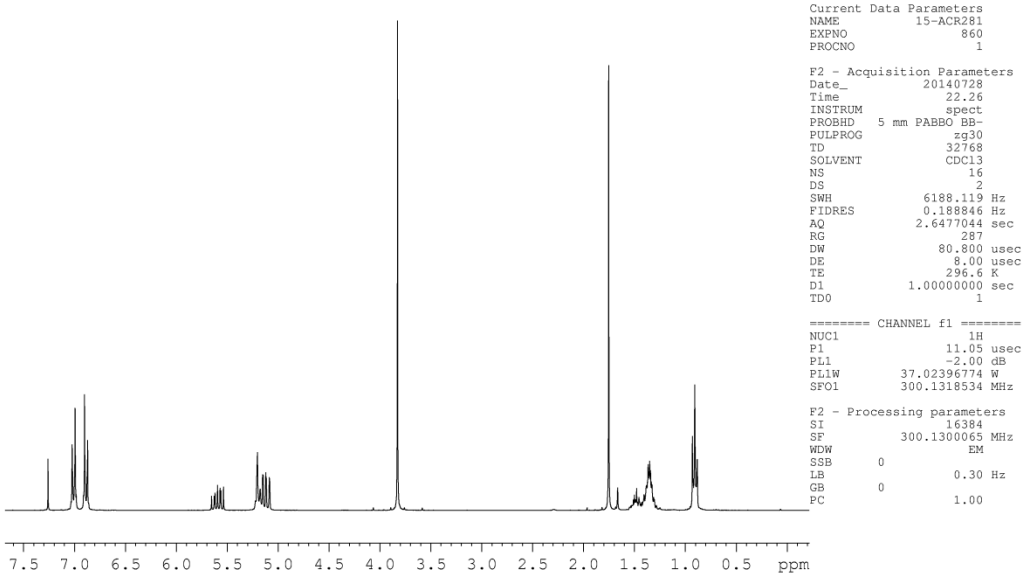


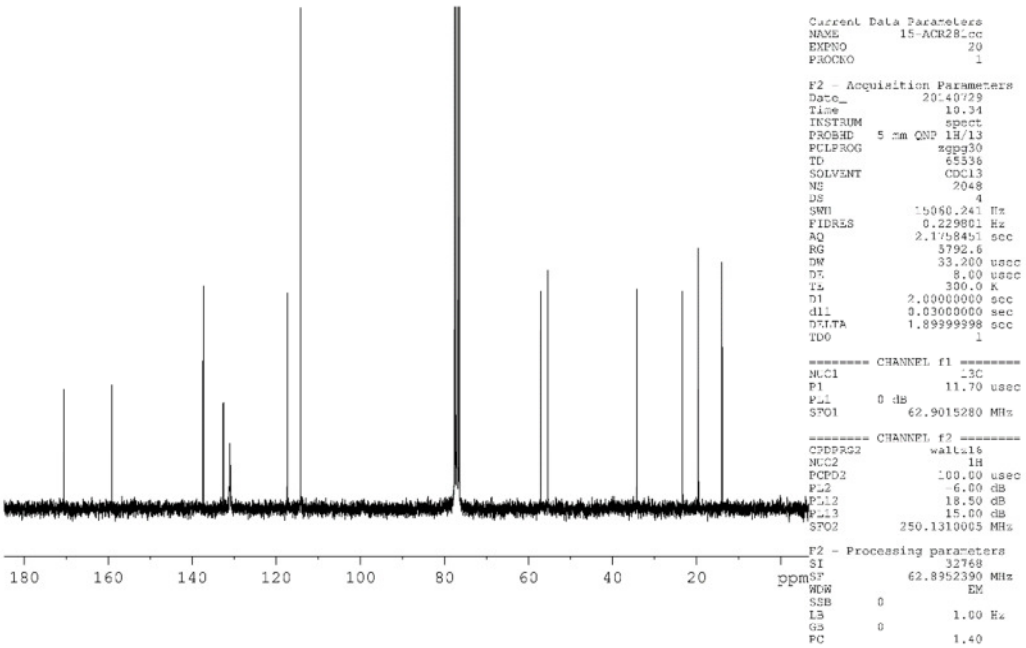


(*R*)-*N*-(Hex-1-en-3-yl)-*N*-(4-methoxyphenyl)acetamide (10c)

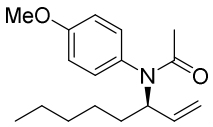


10c

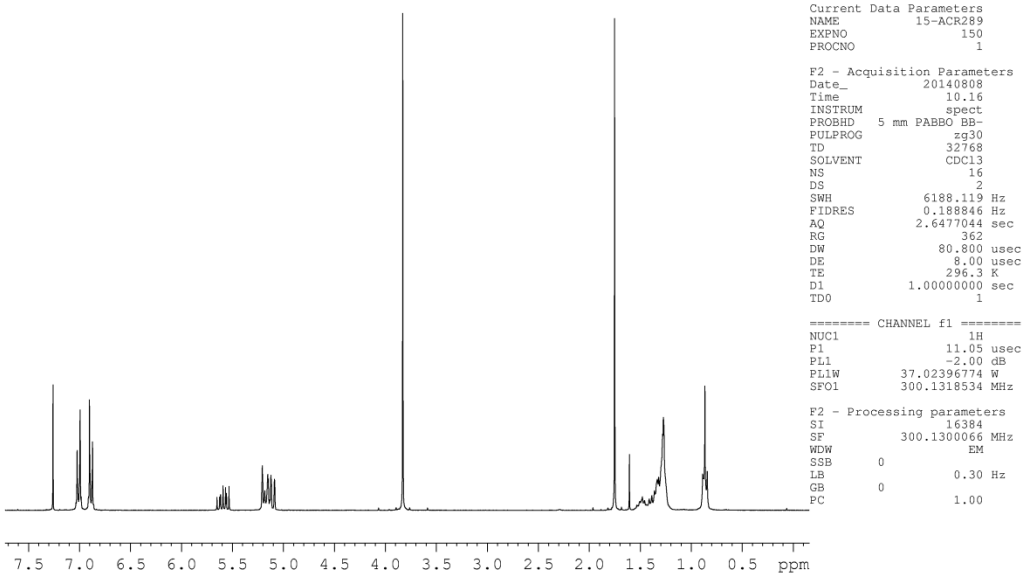


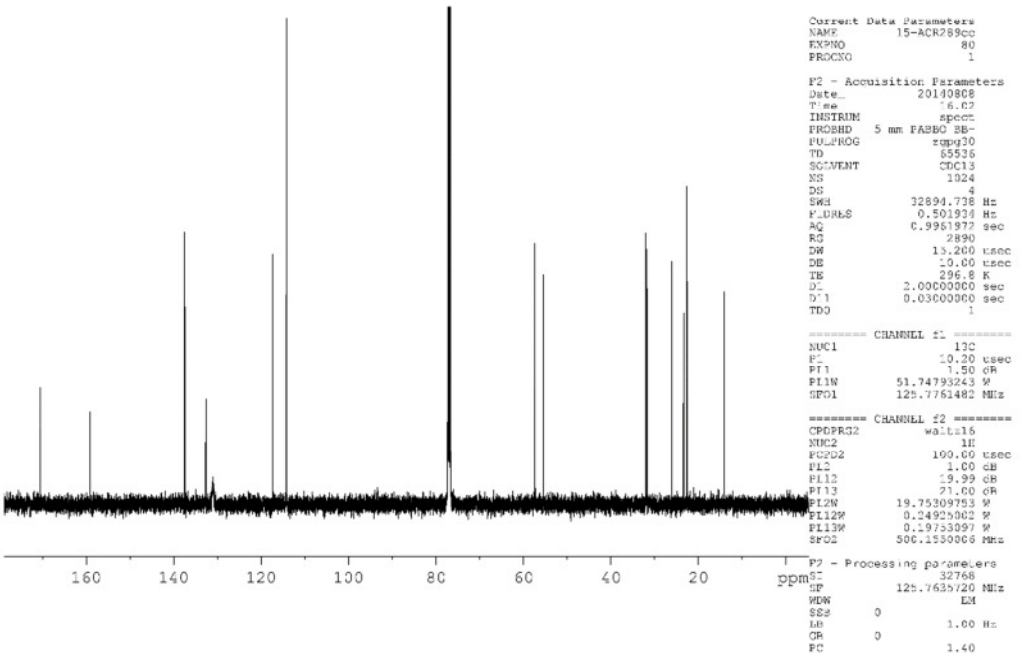


(*R*)-*N*-(4-Methoxyphenyl)-*N*-(oct-1-en-3-yl)acetamide (10d)

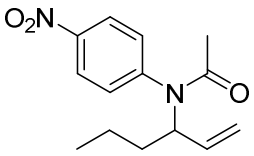


10d

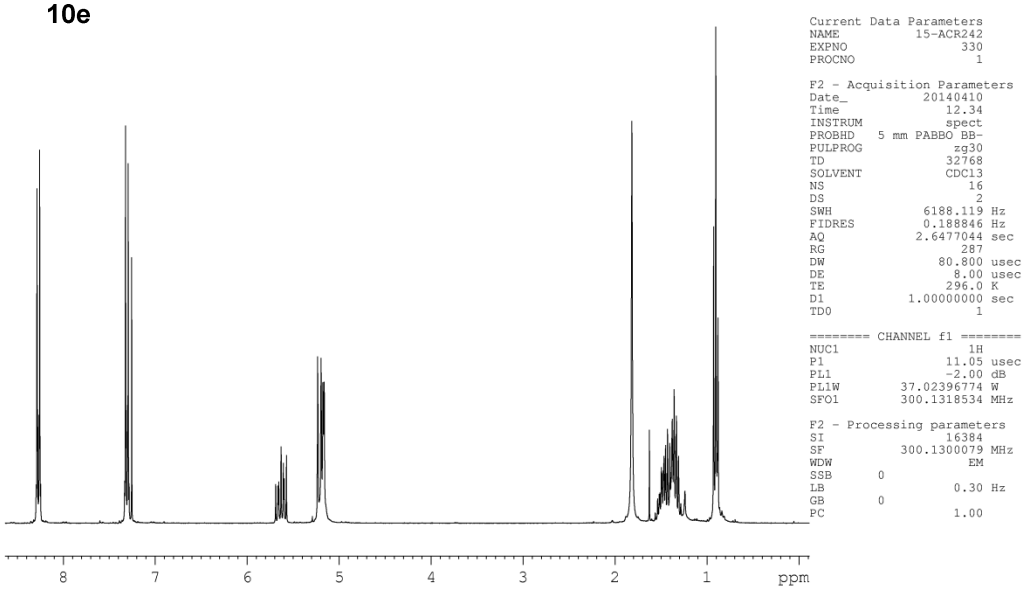




N-(Hex-1-en-3-yl)-*N*-(4-nitrophenyl)acetamide (10e)



10e

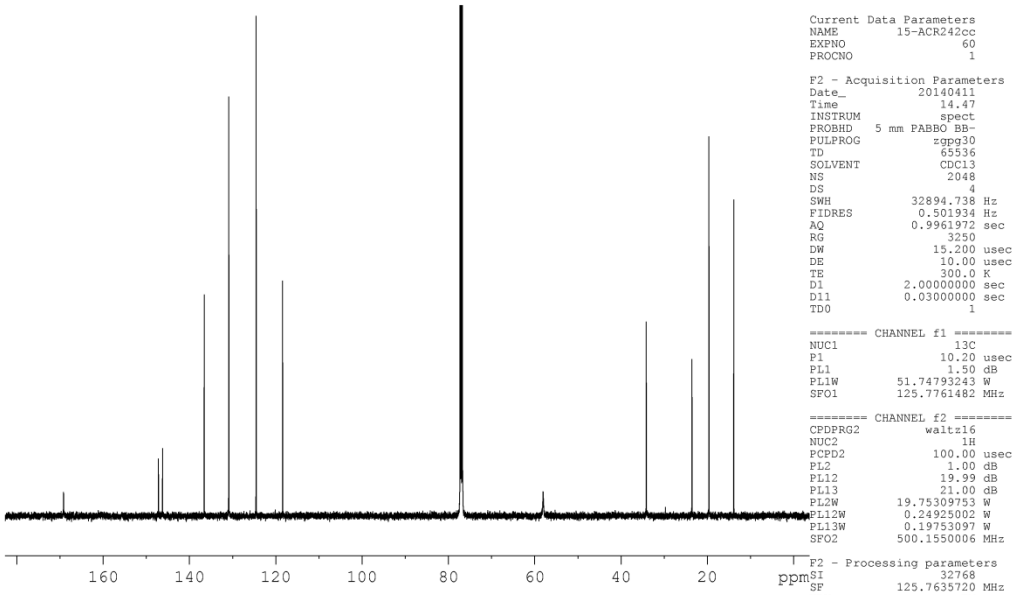



```
Current Data Parameters
NAME          15-ACR242
EXPNO               330
PROCNO                1

F2 - Acquisition Parameters
Date_          20140410
Time              12.34
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            6188.119 Hz
FIDRES        0.188846 Hz
AQ            2.6477044 sec
RG                  287
DW               80.800 usec
DE                8.00 usec
TE               296.0 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                1H
P1              11.05 usec
PL1             -2.00 dB
PL1W        37.02396774 W
SFO1        300.1318534 MHz

F2 - Processing parameters
SI                16384
SF          300.1300079 MHz
WDW                  EM
SSB       0
LB             0.30 Hz
GB        0
PC                1.00
```




```
Current Data Parameters
NAME        15-ACR242cc
EXPNO                60
PROCNO                1

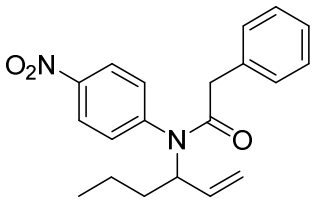
F2 - Acquisition Parameters
Date_          20140411
Time              14.47
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2048
DS                    4
SWH           32894.738 Hz
FIDRES        0.501934 Hz
AQ            0.9961972 sec
RG                 3250
DW               15.200 usec
DE                10.00 usec
TE                300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1              10.20 usec
PL1              1.50 dB
PL1W        51.74793243 W
SFO1        125.7761482 MHz

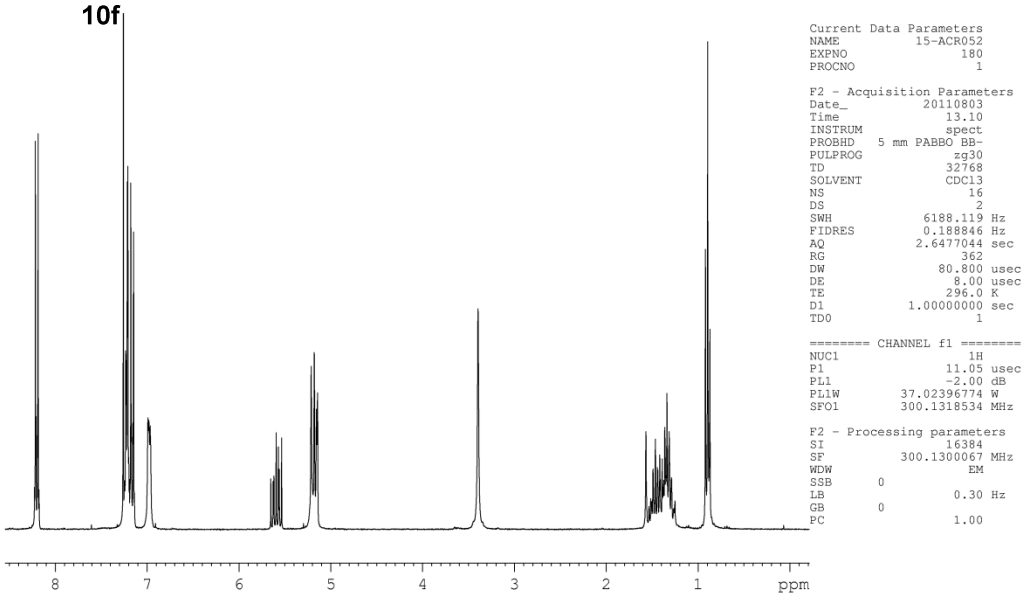
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2           100.00 usec
PL2              1.00 dB
PL12            19.99 dB
PL13            21.00 dB
PL2W        19.75309753 W
PL12W        0.24925002 W
PL13W        0.19753097 W
SFO2        500.1550006 MHz

F2 - Processing parameters
SI                32768
SF          125.7635720 MHz
WDW                  EM
SSB       0
LB             1.00 Hz
GB        0
PC                1.40
```

N-(Hex-1-en-3-yl)-*N*-(4-nitrophenyl)-2-phenylacetamide (10f)



10f

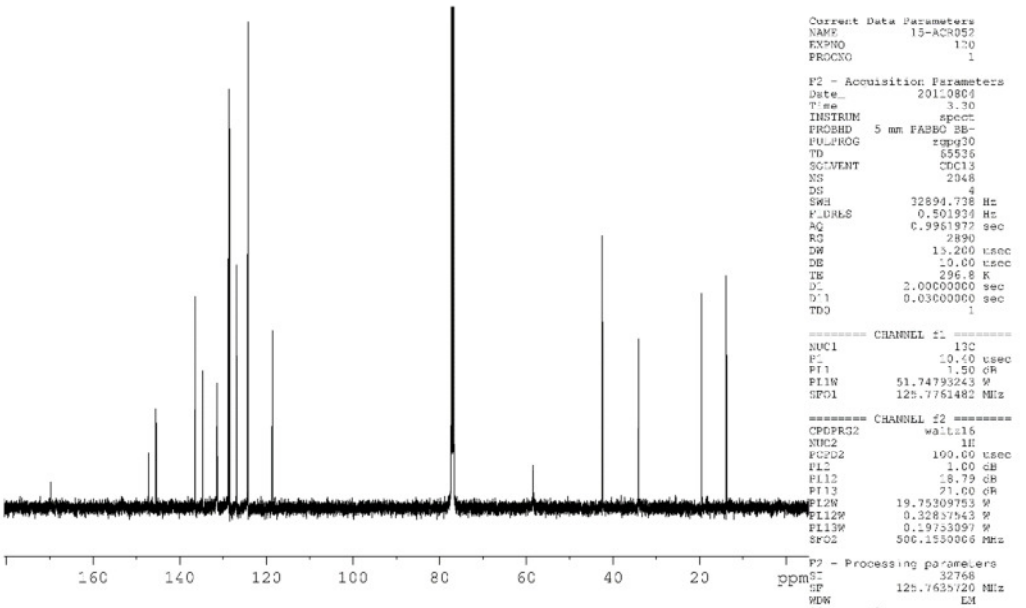



```
Current Data Parameters
NAME           15-ACR052
EXPNO                180
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110803
Time               13.10
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6188.119 Hz
FIDRES          0.188846 Hz
AQ              2.6477044 sec
RG                   362
DW                80.800 usec
DE                 8.00 usec
TE                296.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.05 usec
PL1                -2.00 dB
PL1W         37.02396774 W
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                 16384
SF           300.1300067 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME           15-ACR052
EXPNO                120
PROCNO                 1

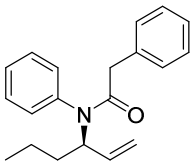
F2 - Acquisition Parameters
Date_           20110804
Time               3.30
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  2048
DS                     4
SWH            32894.738 Hz
FIDRES          0.501934 Hz
AQ              0.9961972 sec
RG                  2890
DW                15.200 usec
DE                10.00 usec
TE                296.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.40 usec
PL1                 1.50 dB
PL1W         51.74793243 W
SFO1         125.7761482 MHz

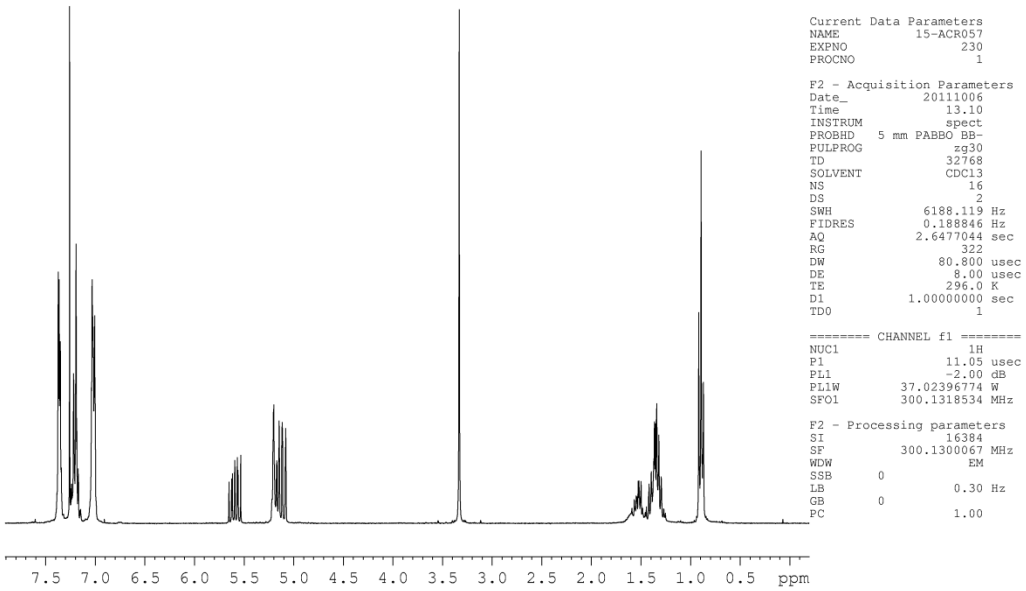
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                 1.00 dB
PL12               18.79 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.32837543 W
PL13W         0.19753097 W
SFO2         500.1330006 MHz

F2 - Processing parameters
SI                 32768
SF           125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

(*R*)-*N*-(Hex-1-en-3-yl)-*N*,2-diphenylacetamide (10g)



10g

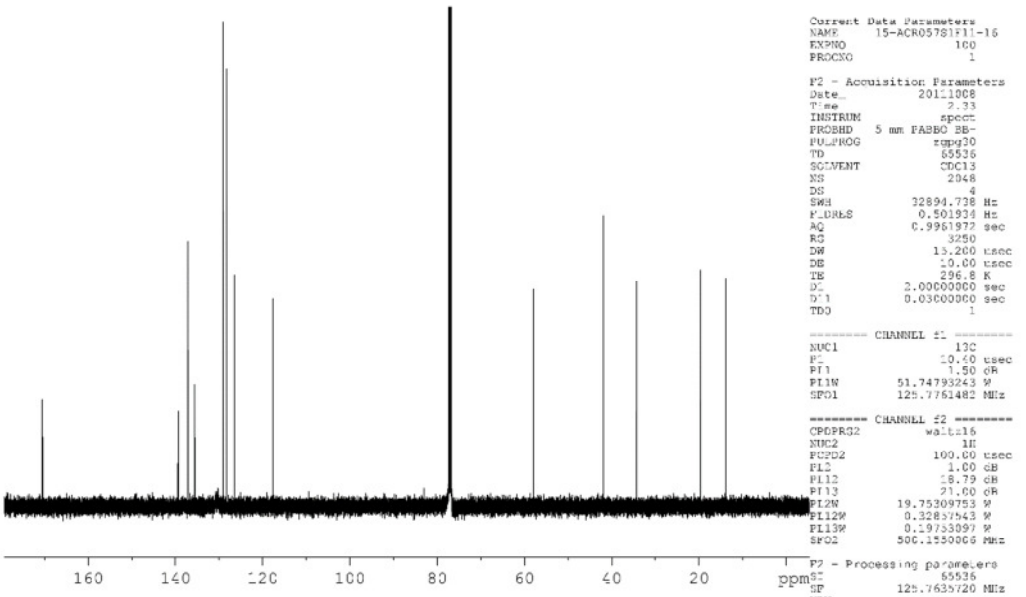



```
Current Data Parameters
NAME           15-ACR057
EXPNO                230
PROCNO                 1

F2 - Acquisition Parameters
Date_           20111006
Time               13.10
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6188.119 Hz
FIDRES          0.188846 Hz
AQ              2.6477044 sec
RG                   322
DW                80.800 usec
DE                 8.00 usec
TE                296.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.05 usec
PL1                -2.00 dB
PL1W         37.02396774 W
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                 16384
SF           300.1300067 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME      15-ACR057S1F11-16
EXPNO                  160
PROCNO                   1

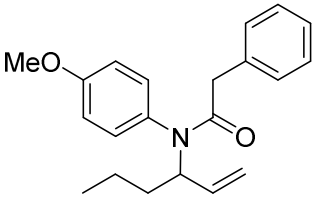
F2 - Acquisition Parameters
Date_           20111008
Time               2.33
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2048
DS                    4
SWH            32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961972 sec
RG                 3250
DW               15.200 usec
DE                10.00 usec
TE                296.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.40 usec
PL1                1.50 dB
PL1W         51.74793243 W
SFO1         125.7761482 MHz

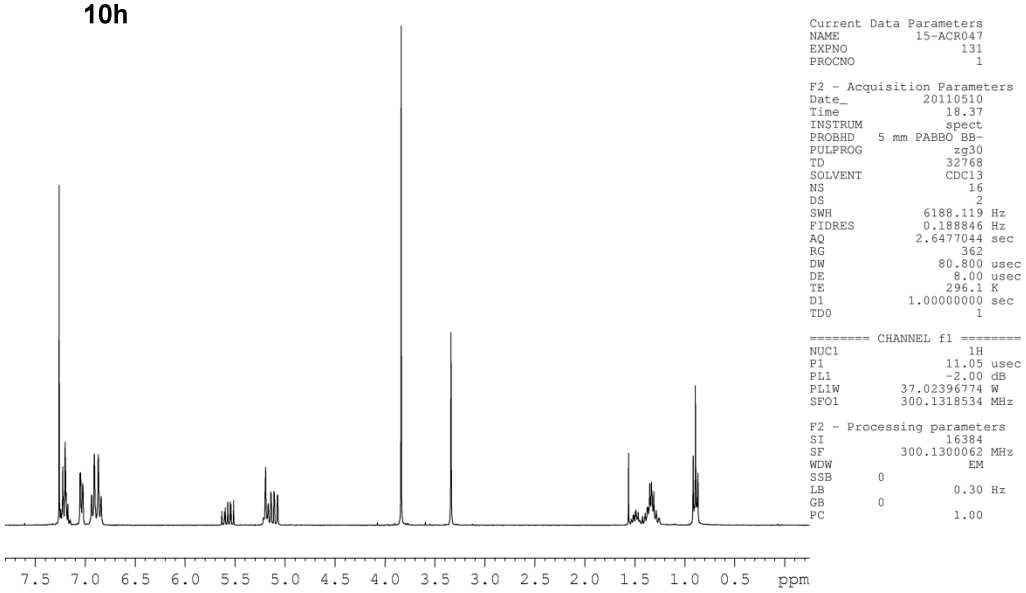
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                1.00 dB
PL12               18.79 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.32837543 W
PL13W         0.19733097 W
SFO2         500.1550066 MHz

F2 - Processing parameters
SI                 65536
SF           125.7635720 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                 1.40
```

N-(Hex-1-en-3-yl)-*N*-(4-methoxyphenyl)-2-phenylacetamide (10h)



10h

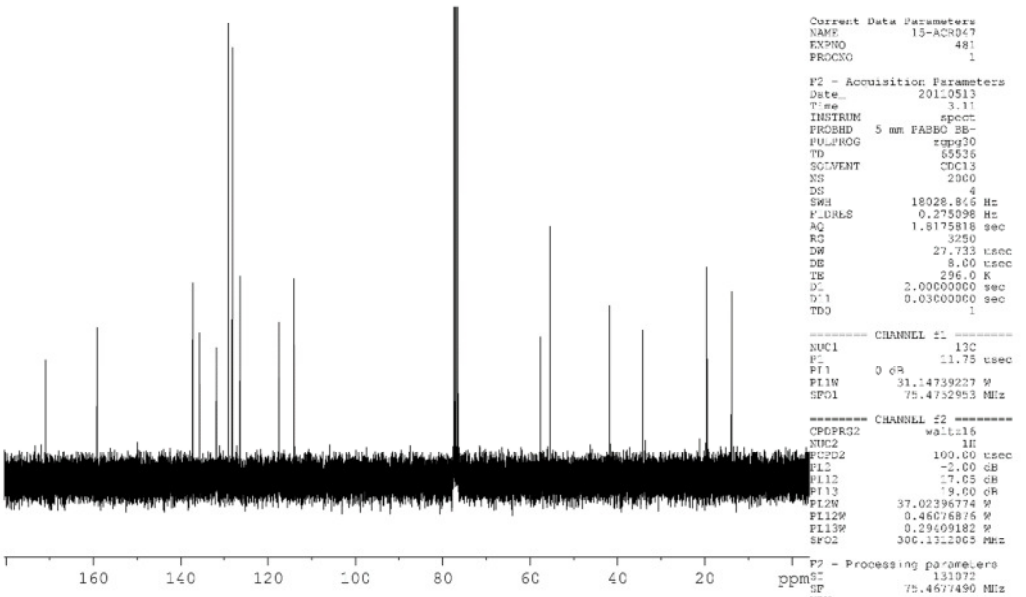



```
Current Data Parameters
NAME           15-ACR047
EXPNO                131
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110510
Time               18.37
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6188.119 Hz
FIDRES          0.188846 Hz
AQ              2.6477044 sec
RG                   362
DW                80.800 usec
DE                 8.00 usec
TE                296.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.05 usec
PL1                -2.00 dB
PL1W         37.02396774 W
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                 16384
SF           300.1300062 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME           15-ACR047
EXPNO                481
PROCNO                 1

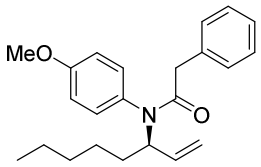
F2 - Acquisition Parameters
Date_           20110513
Time               3.11
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  2000
DS                     4
SWH            18028.846 Hz
FIDRES          0.275098 Hz
AQ              1.8175818 sec
RG                  3250
DW                27.733 usec
DE                 8.00 usec
TE                296.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 11.75 usec
PL1        0 dB
PL1W         31.14739227 W
SFO1         75.4752953 MHz

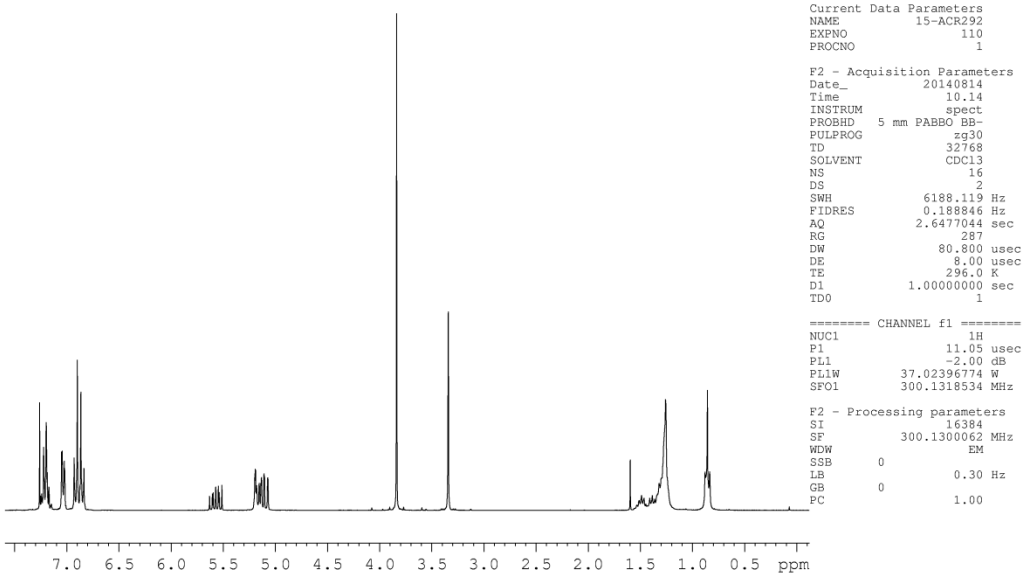
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                -2.00 dB
PL12               17.05 dB
PL13               19.00 dB
PL2W         37.02396774 W
PL12W         0.46076876 W
PL13W         0.29409182 W
SFO2         300.1312005 MHz

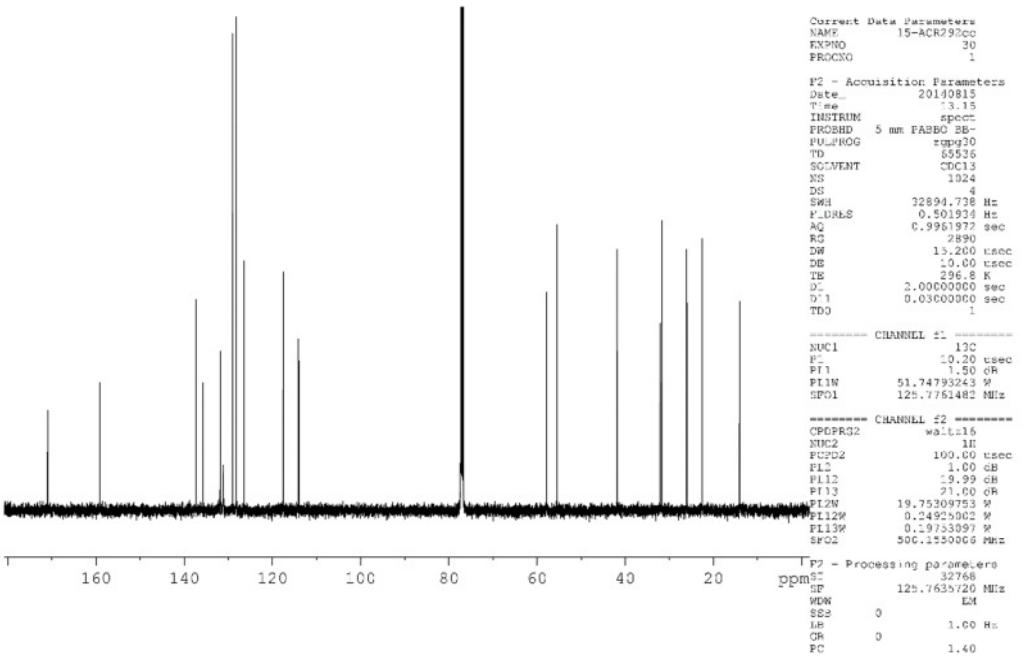
F2 - Processing parameters
SI                131072
SF           75.4677490 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                 1.40
```

(*R*)-*N*-(4-Methoxyphenyl)-*N*-(oct-1-en-3-yl)-2-phenylacetamide(10i)

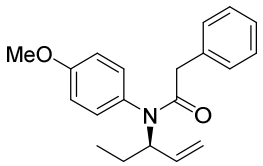


10i

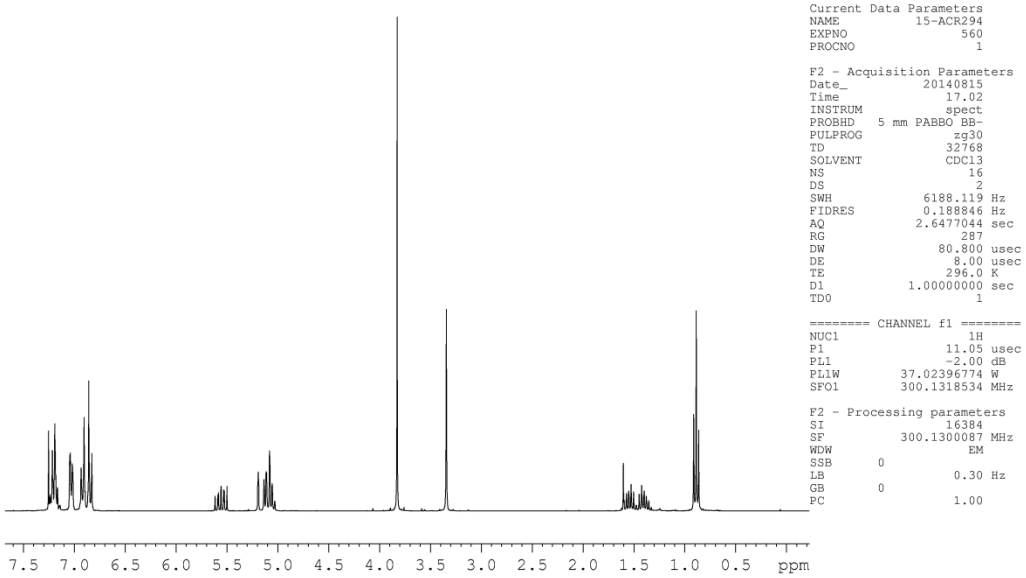


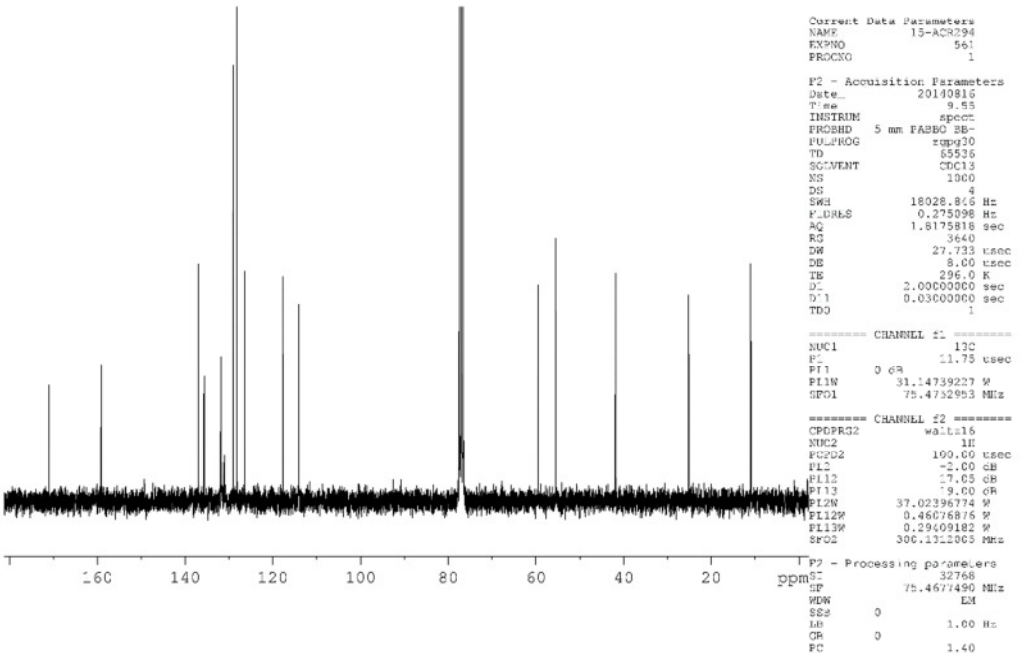


(*R*)-*N*-(4-Methoxyphenyl)-*N*-(pent-1-en-3-yl)-2-phenylacetamide (10j)

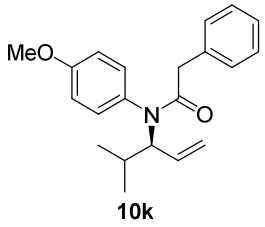


10j

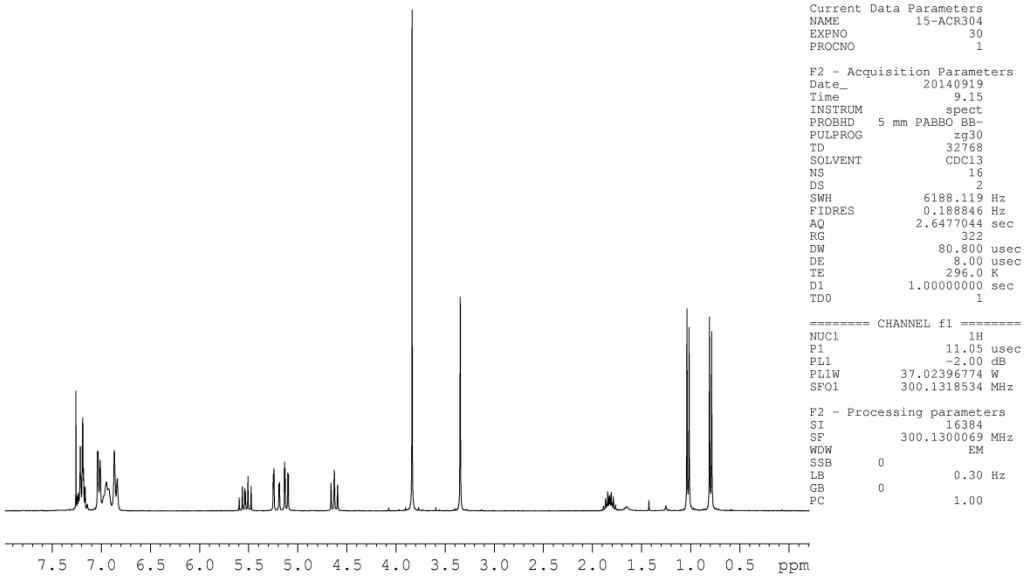




(*R*)-*N*-(4-Methoxyphenyl)-*N*-(4-methylpent-1-en-3-yl)-2-phenylacetamide (10k)



10k

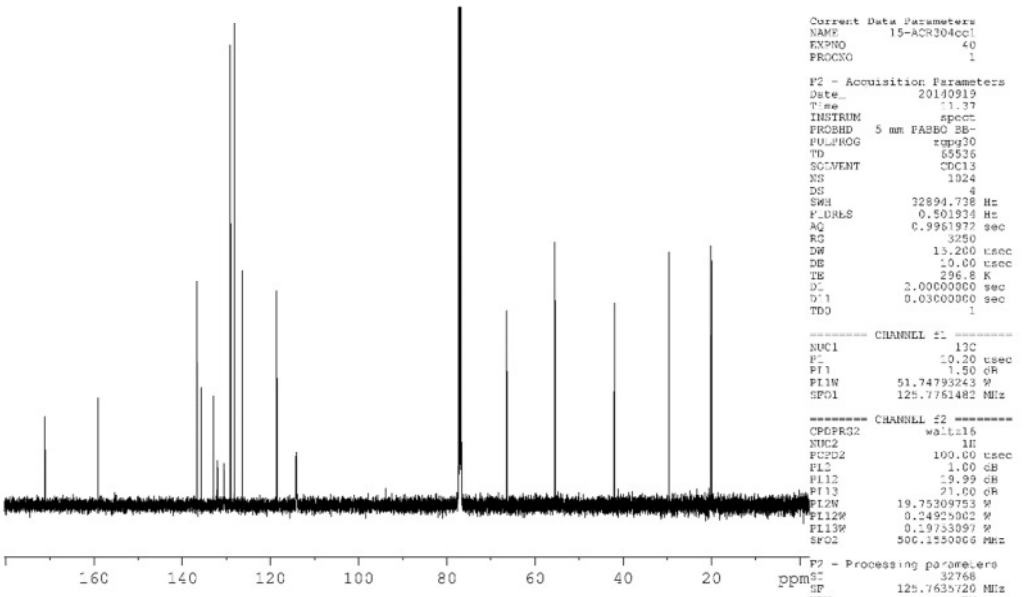



```
Current Data Parameters
NAME           15-ACR304
EXPNO                 30
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140919
Time                9.15
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6188.119 Hz
FIDRES          0.188846 Hz
AQ              2.6477044 sec
RG                   322
DW                80.800 usec
DE                 8.00 usec
TE                296.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.05 usec
PL1                -2.00 dB
PL1W         37.02396774 W
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                 16384
SF           300.1300069 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME        15-ACR304ccl
EXPNO                 40
PROCNO                 1

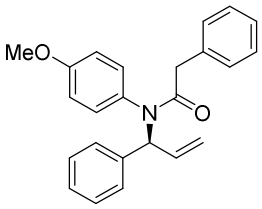
F2 - Acquisition Parameters
Date_           20140919
Time                11.37
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961972 sec
RG                  3250
DW                15.200 usec
DE                10.00 usec
TE                296.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W         51.74793243 W
SFO1         125.7761482 MHz

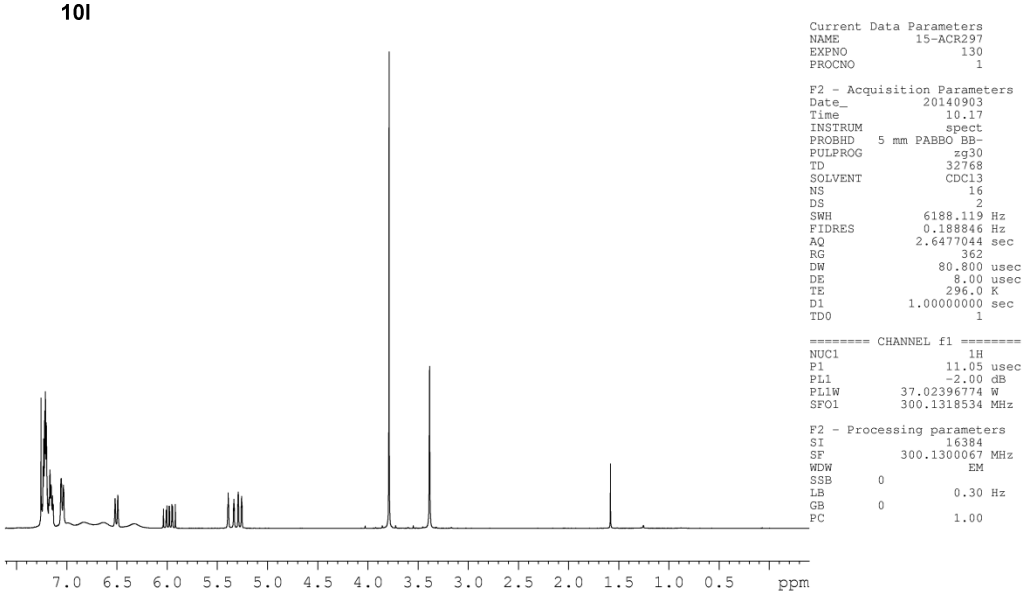
======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                   1H
PCPD2             100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925062 W
PL13W         0.19733097 W
SFO2         500.1550066 MHz

F2 - Processing parameters
SI                 32768
SF           125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

(*S*)-*N*-(4-Methoxyphenyl)-2-phenyl-*N*-(1-phenylallyl)acetamide (10l)



10l

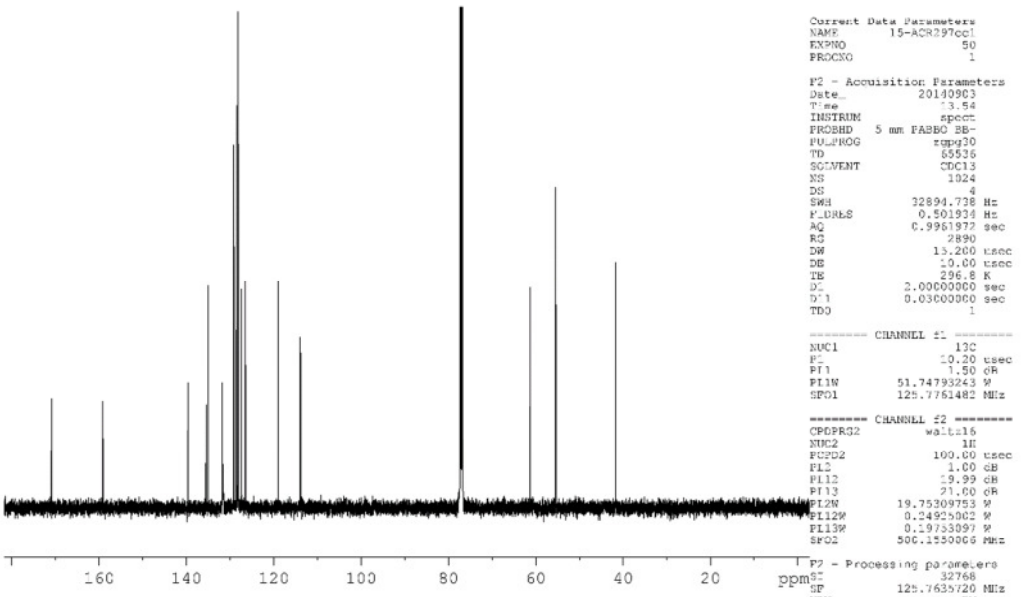



```
Current Data Parameters
NAME           15-ACR297
EXPNO                130
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140903
Time               10.17
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6188.119 Hz
FIDRES          0.188846 Hz
AQ              2.6477044 sec
RG                   362
DW                80.800 usec
DE                 8.00 usec
TE                296.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.05 usec
PL1                -2.00 dB
PL1W         37.02396774 W
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                 16384
SF           300.1300067 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME        15-ACR297cc
EXPNO                50
PROCNO                1

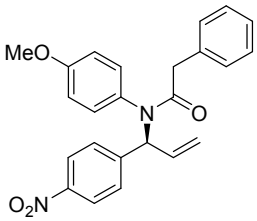
F2 - Acquisition Parameters
Date_           20140903
Time               13.54
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            32894.738 Hz
FIDRES          0.501934 Hz
AQ              0.9961972 sec
RG                  2890
DW                15.200 usec
DE                10.00 usec
TE                296.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W         51.74793243 W
SFO1         125.7761482 MHz

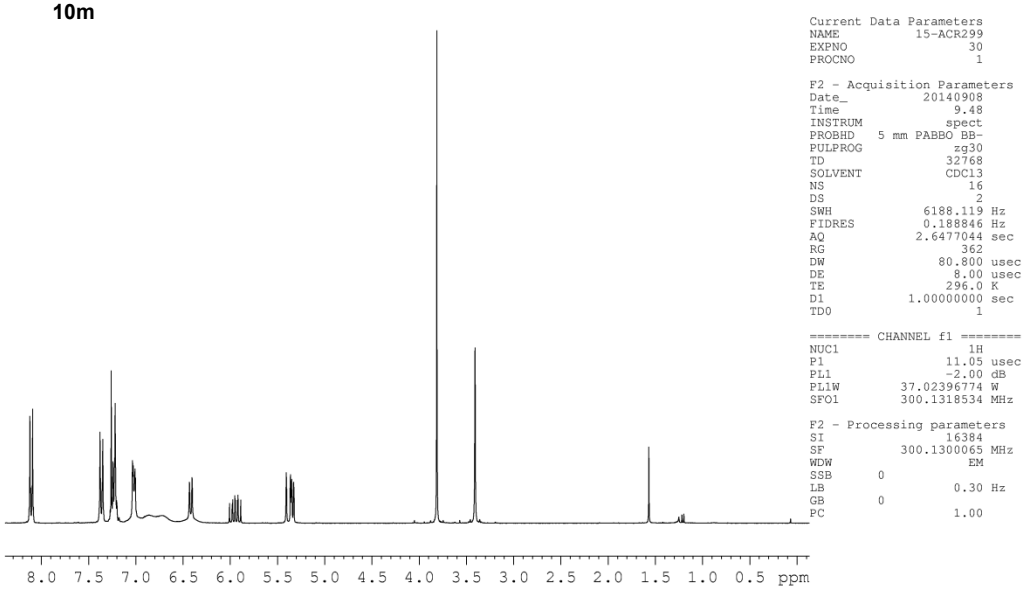
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925062 W
PL13W         0.19733097 W
SFO2         500.1550006 MHz

F2 - Processing parameters
SI                 32768
SF           125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

(*S*)-*N*-(4-Methoxyphenyl)-*N*-(1-(4-nitrophenyl)allyl)-2-phenylacetamide (10m)



10m

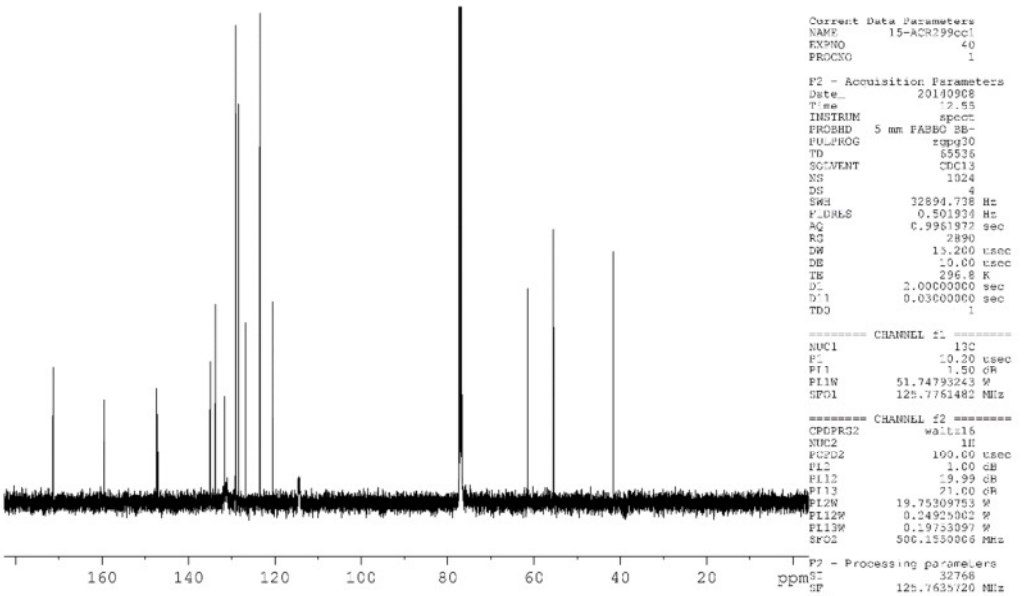



```
Current Data Parameters
NAME           15-ACR299
EXPNO                 30
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140908
Time                9.48
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6188.119 Hz
FIDRES          0.188846 Hz
AQ              2.6477044 sec
RG                   362
DW                80.800 usec
DE                 8.00 usec
TE                296.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.05 usec
PL1                -2.00 dB
PL1W         37.02396774 W
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                 16384
SF           300.1300065 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME        15-ACR299cc
EXPNO                40
PROCNO                1

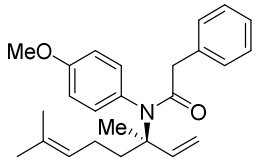
F2 - Acquisition Parameters
Date_           20140908
Time               12.55
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            32894.738 Hz
FIDRES         0.501934 Hz
AQ             0.9961972 sec
RG                  2890
DW                15.200 usec
DE                10.00 usec
TE                296.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.20 usec
PL1                1.50 dB
PL1W         51.74793243 W
SFO1         125.7761482 MHz

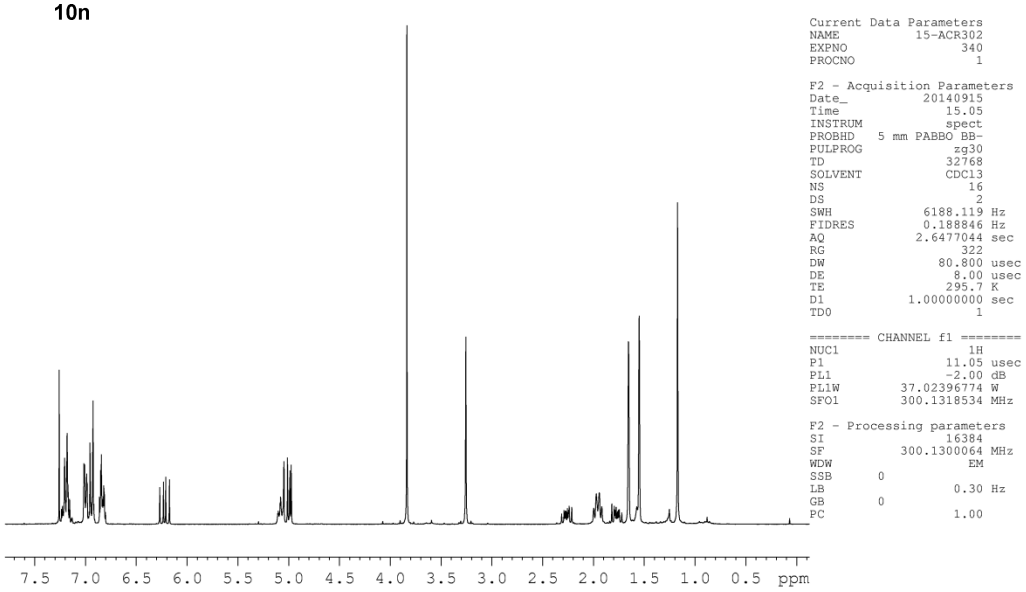
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925062 W
PL13W         0.19753097 W
SFO2         500.1330006 MHz

F2 - Processing parameters
SI                 32768
SF           125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

(*R*)-*N*-(3,7-Dimethylocta-1,6-dien-3-yl)-*N*-(4-methoxyphenyl)-2-phenylacetamide (10n)



10n

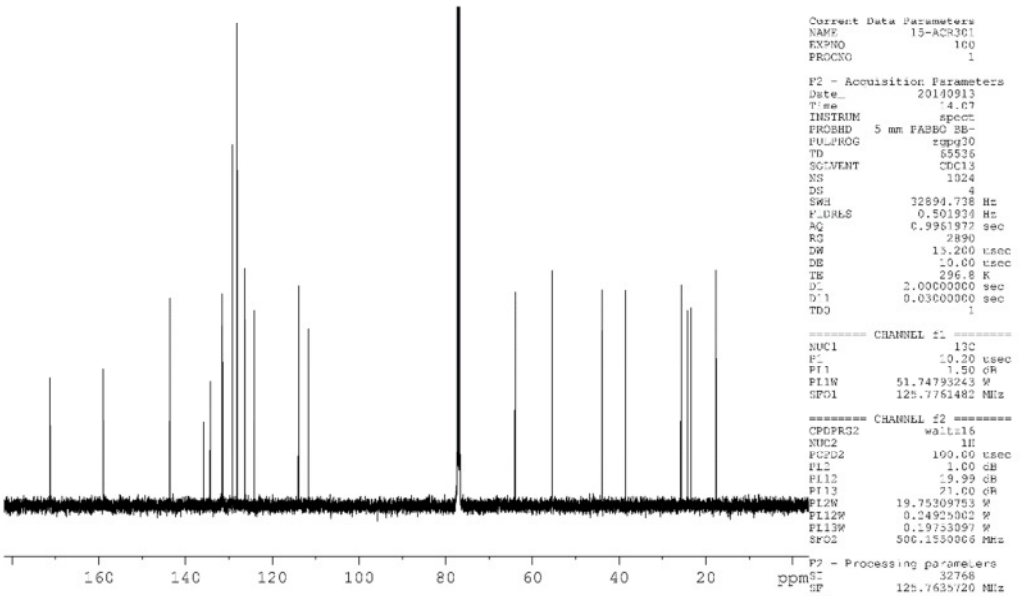



```
Current Data Parameters
NAME           15-ACR302
EXPNO                340
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140915
Time               15.05
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6188.119 Hz
FIDRES          0.188846 Hz
AQ              2.6477044 sec
RG                   322
DW                80.800 usec
DE                 8.00 usec
TE                295.7 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.05 usec
PL1                -2.00 dB
PL1W         37.02396774 W
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                 16384
SF           300.1300064 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME           15-ACR301
EXPNO                160
PROCNO                 1

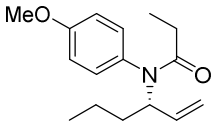
F2 - Acquisition Parameters
Date_           20140913
Time               14.07
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            32894.738 Hz
FIDRES          0.501934 Hz
AQ              0.9961977 sec
RG                  2890
DW                15.200 usec
DE                10.00 usec
TE                296.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.20 usec
PL1                -1.50 dB
PL1W         51.74793243 W
SFO1         125.7761482 MHz

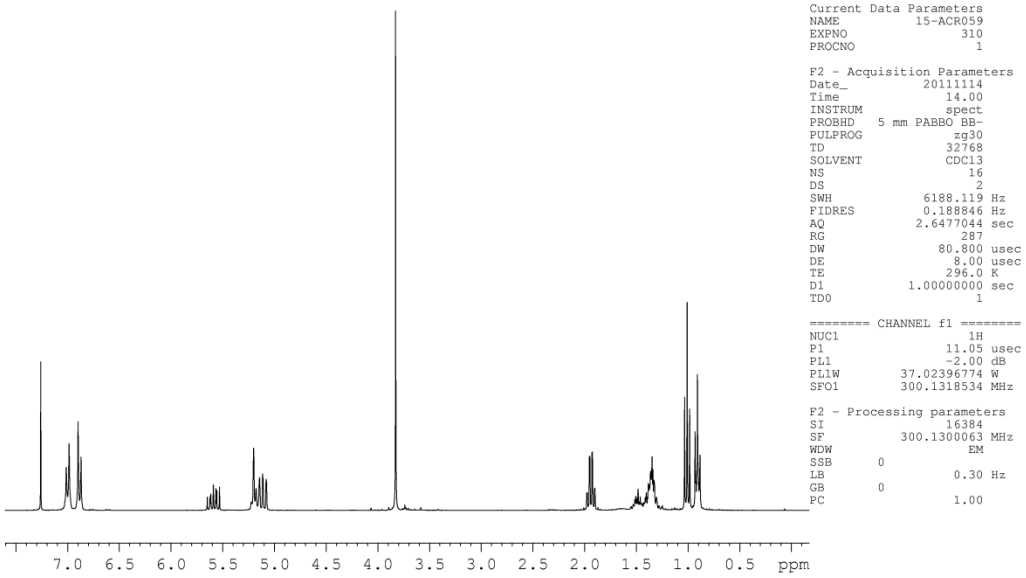
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925062 W
PL13W         0.19753097 W
SFO2         500.1330006 MHz

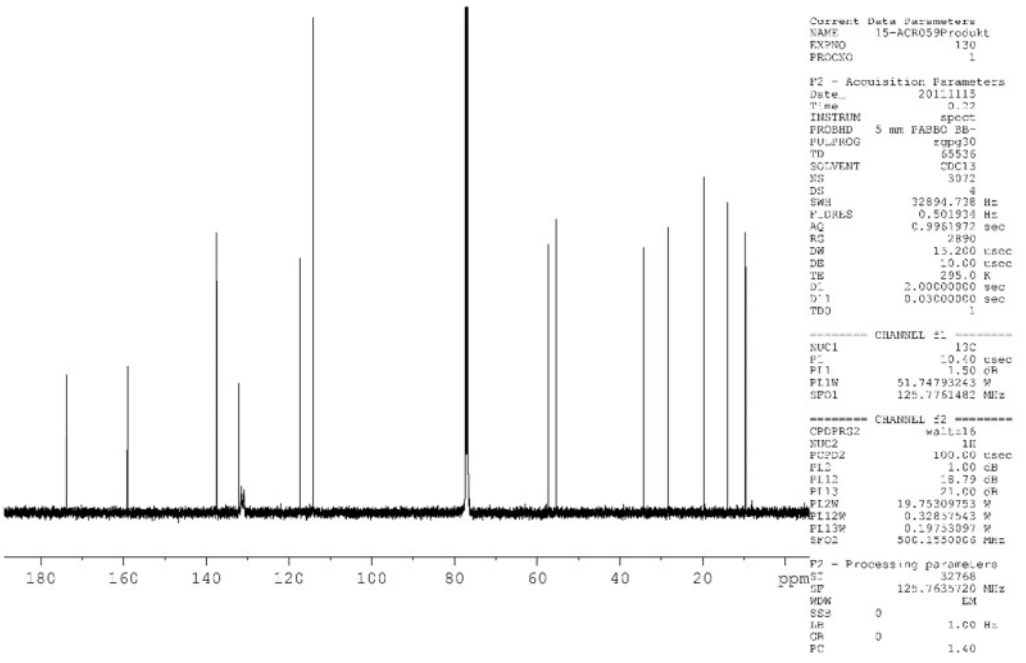
F2 - Processing parameters
SI                 32768
SF           125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

(*S*)-*N*-(Hex-1-en-3-yl)-*N*-(4-methoxyphenyl)propionamide ((*ent*)-10o)

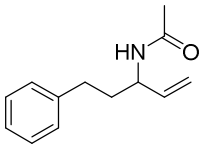


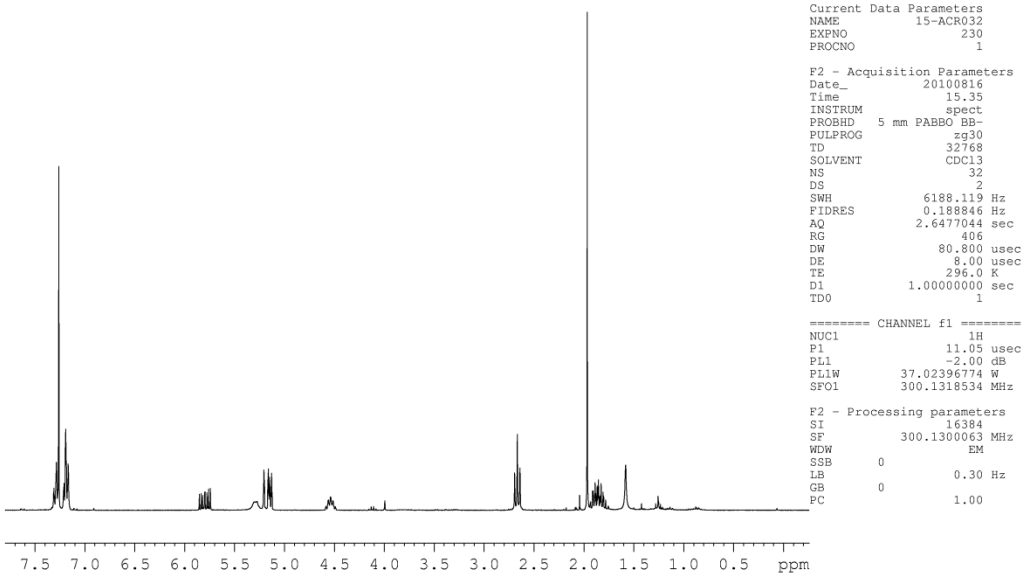
(*ent*)-10o

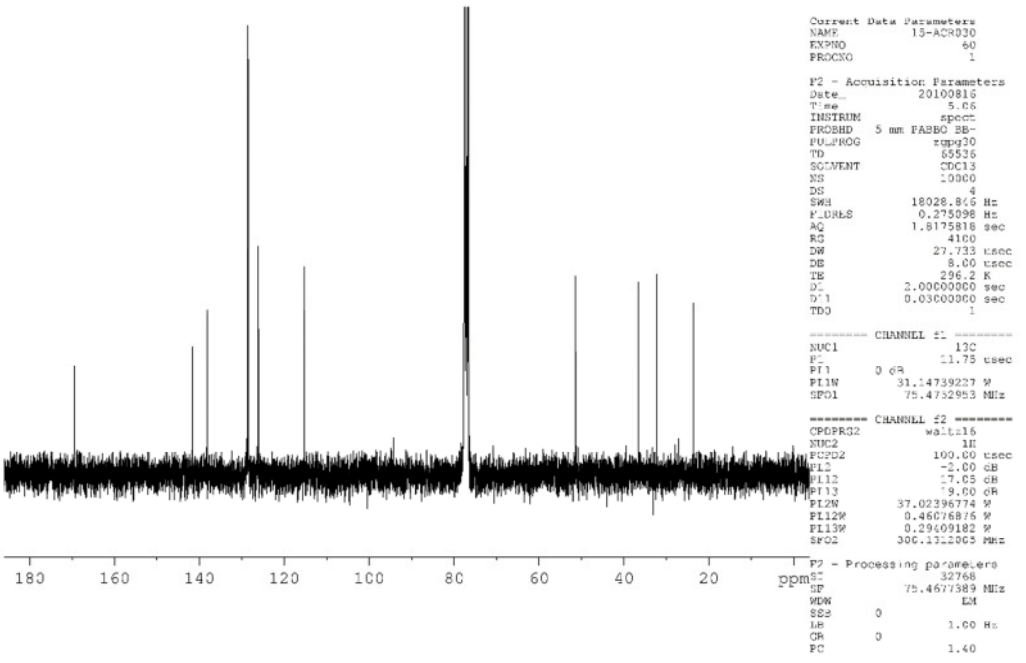




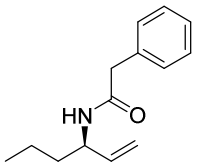
N-(5-Phenylpent-1-en-3-yl)acetamide



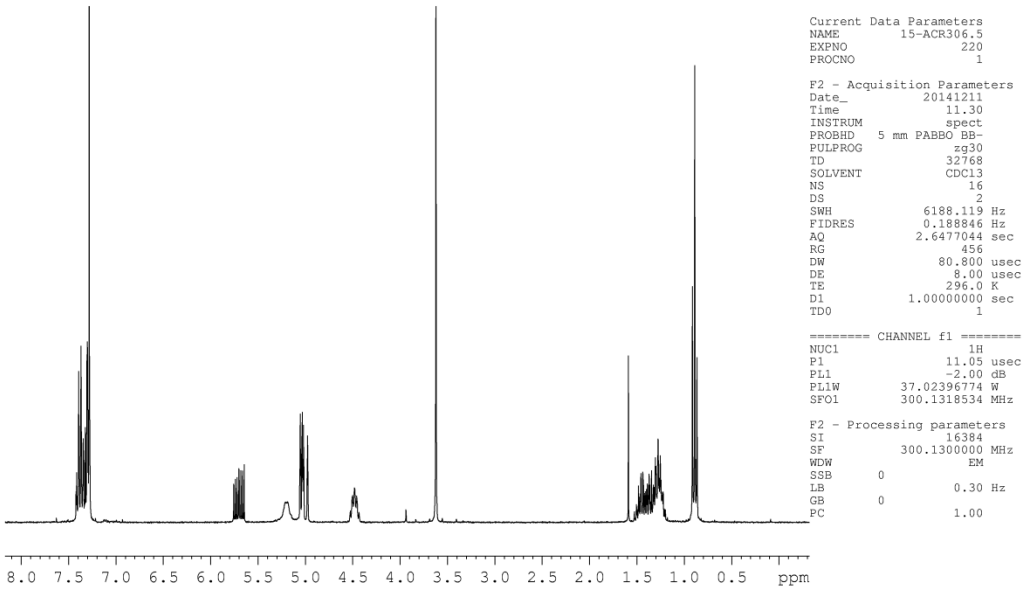




(*R*)-*N*-(Hex-1-en-3-yl)-2-phenylacetamide (12)



12

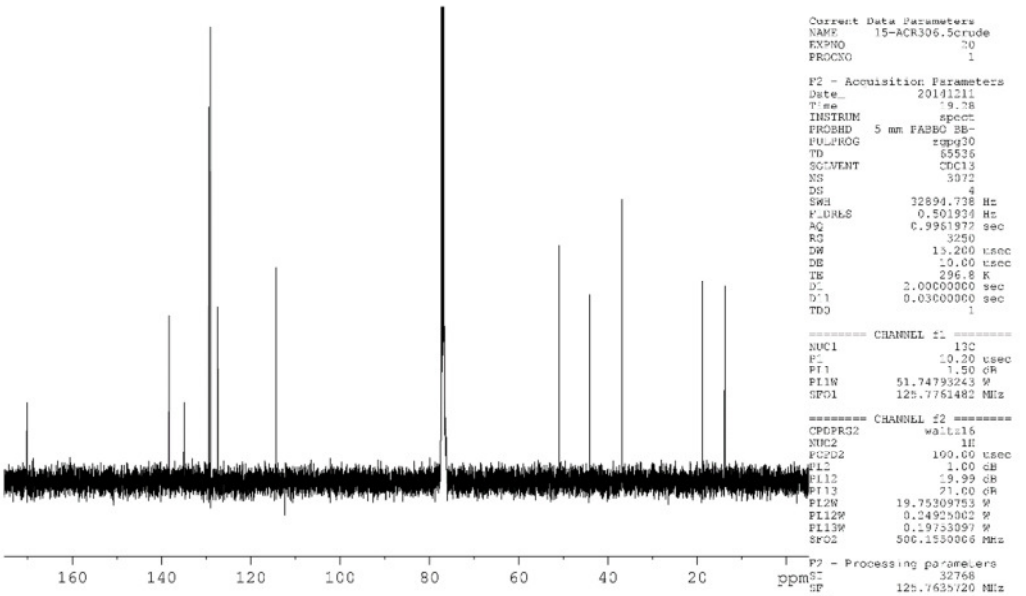



```
Current Data Parameters
NAME         15-ACR306.5
EXPNO                220
PROCNO                 1

F2 - Acquisition Parameters
Date_           20141211
Time               11.30
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             6188.119 Hz
FIDRES          0.188846 Hz
AQ              2.6477044 sec
RG                   456
DW                80.800 usec
DE                 8.00 usec
TE                296.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.05 usec
PL1                -2.00 dB
PL1W         37.02396774 W
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                 16384
SF           300.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




```
Current Data Parameters
NAME      15-ACR306.5crude
EXPNO                   20
PROCNO                   1

F2 - Acquisition Parameters
Date_           20141211
Time               19.28
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDCl3
NS                  3072
DS                     4
SWH            32894.738 Hz
FIDRES          0.501924 Hz
AQ              0.9961977 sec
RG                  3250
DW                15.200 usec
DE                10.00 usec
TE                296.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.20 usec
PL1                 1.50 dB
PL1W         51.74793243 W
SFO1         125.7761482 MHz

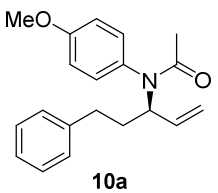
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                 1.00 dB
PL12               19.99 dB
PL13               21.00 dB
PL2W         19.75309753 W
PL12W         0.24925062 W
PL13W         0.19753097 W
SFO2         500.1330006 MHz

F2 - Processing parameters
SI                 32768
SF           125.7635720 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

HPLC Spectra of compounds 10, 11, 13

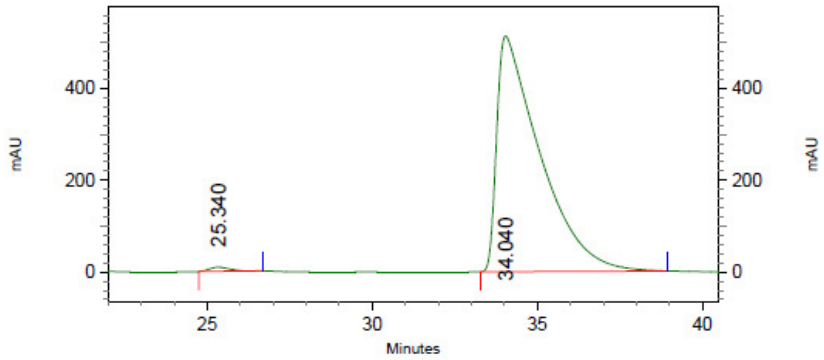
(*R*)-*N*-(4-Methoxyphenyl)-*N*-(5-phenylpent-1-en-3-yl)acetamide (10a)

Compound:



10a

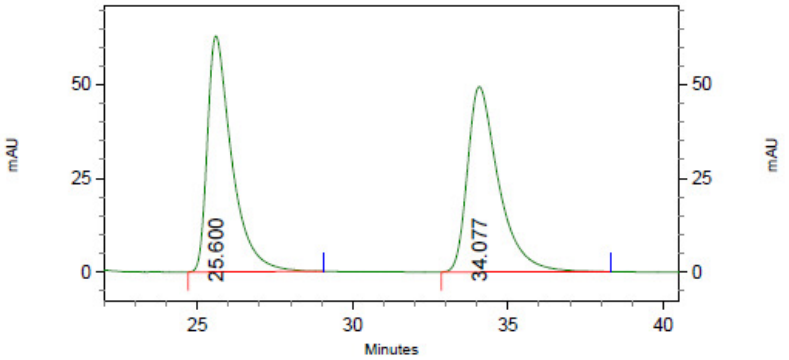
Column: *Chiralpak AD-H*
Method: *n*hexane/*i*PrOH (98/2), 1.0 ml/min, 250 nm



UV Results

Retention Time	Height	Area	Area Percent
25.34	35455	1552585	0.82
34.04	2054975	187006542	99.18
Totals	2090430	188559127	100.00

Racemic reference:

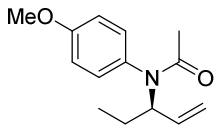


UV Results

Retention Time	Height	Area	Area %
25.60	251811	13821255	49.97
34.08	197911	13835743	50.03
Totals	449722	27656998	100.00

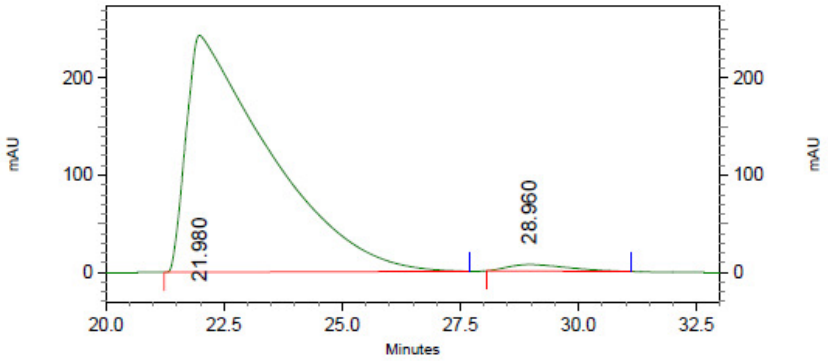
(*R*)-*N*-(4-Methoxyphenyl)-*N*-(pent-1-en-3-yl)acetamide (10b)

Compound:



10b

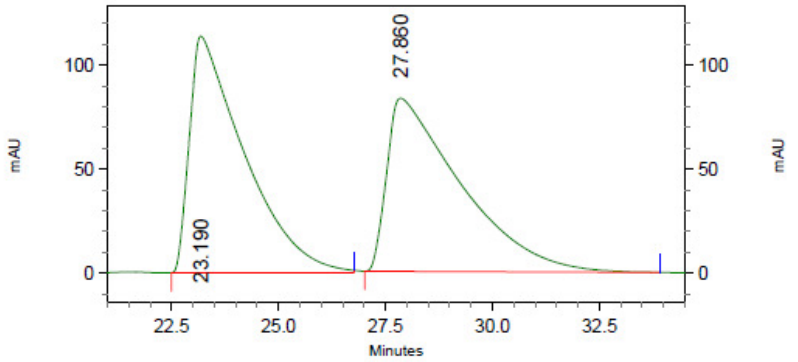
Column: *Chiralpak AD-H*
Method: *n*hexane/*i*PrOH (99.5/0.5), 2.0 ml/min, 250 nm



UV Results

Retention Time	Height	Area	Area Percent
21.98	974086	119287346	98.06
28.96	26722	2363281	1.94
Totals	1000808	121650627	100.00

Racemic reference:

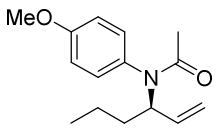


UV Results

Retention Time	Height	Area	Area %
23.19	454778	40389187	50.31
27.86	332660	39897877	49.69
Totals	787438	80287064	100.00

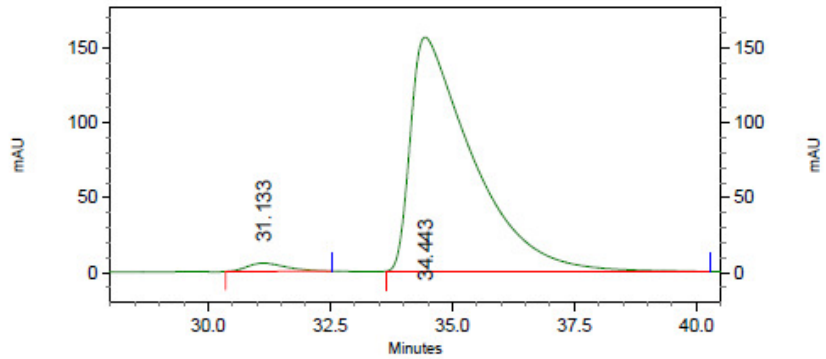
(*R*)-*N*-(Hex-1-en-3-yl)-*N*-(4-methoxyphenyl)acetamide (10c)

Compound:



10c

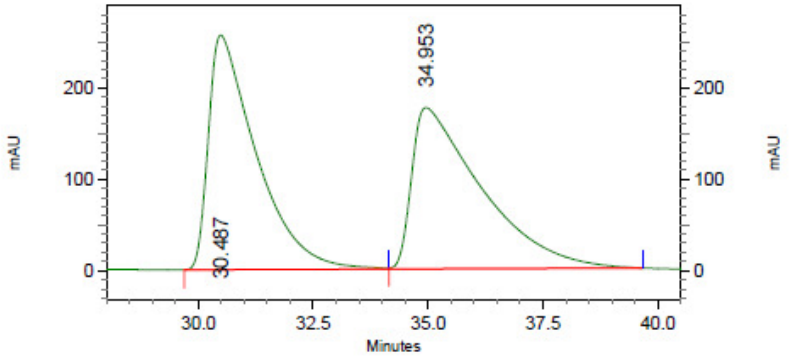
Column: *Chiralpak AD-H*
Method: *n*hexane/*i*PrOH (99.2/0.8), 1.0 ml/min, 250 nm



UV Results

Retention Time	Height	Area	Area Percent
31.13	22110	1236087	2.19
34.44	625205	55189402	97.81
Totals	647315	56425489	100.00

Racemic reference:

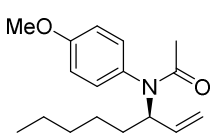


UV Results

Retention Time	Height	Area	Area %
30.49	1026674	74237332	49.98
34.95	705920	74285214	50.02
Totals	1732594	148522546	100.00

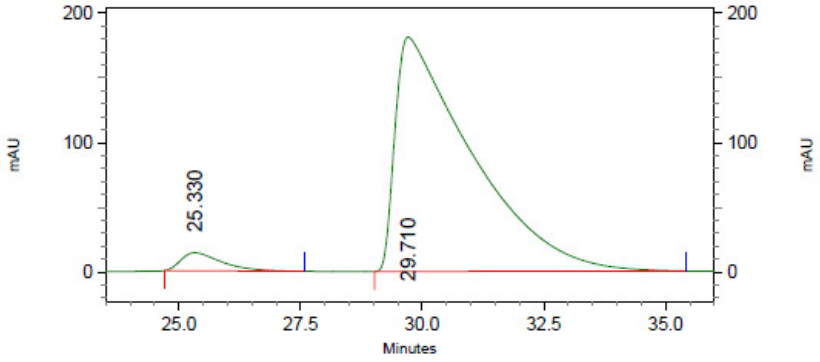
(*R*)-*N*-(4-Methoxyphenyl)-*N*-(oct-1-en-3-yl)acetamide (10d)

Compound:



10d

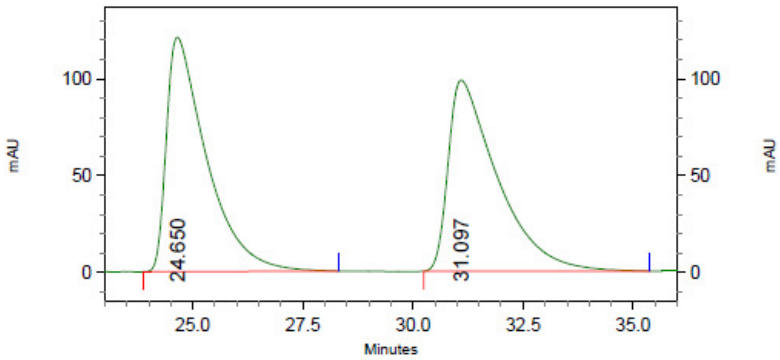
Column: *Chiralpak AD-H*
Method: *n*hexane/*i*PrOH (99/1), 1.0 ml/min, 250 nm



UV Results

Retention Time	Height	Area	Area Percent
25.33	55772	3230889	3.92
29.71	724026	79275372	96.08
Totals	779798	82506261	100.00

Racemic reference:

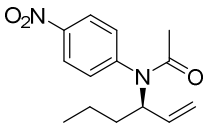


UV Results

Retention Time	Height	Area	Area %
24.65	484325	30652002	50.01
31.10	394550	30644569	49.99
Totals	878875	61296571	100.00

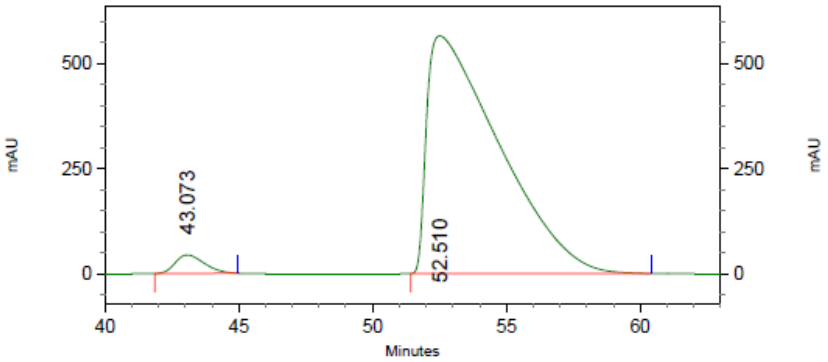
(*R*)-*N*-(Hex-1-en-3-yl)-*N*-(4-nitrophenyl)acetamide (10e)

Compound:



10e

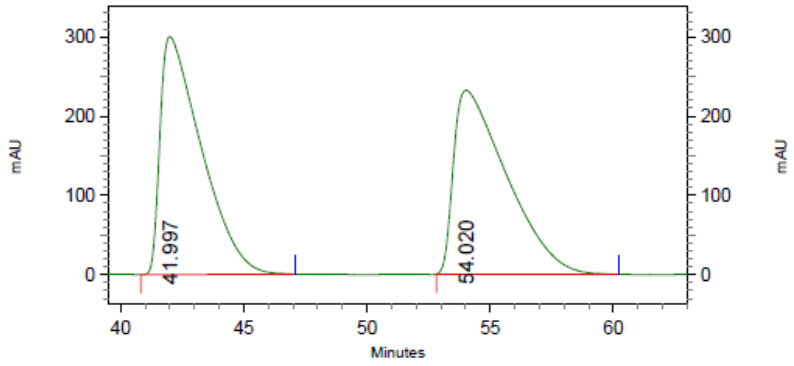
Column: *Chiralpak AS-H*
Method: *n*hexane/*i*PrOH (96/4), 1.0 ml/min, 250 nm



UV Results

Retention Time	Height	Area	Area Percent
43.07	176208	13305051	3.03
52.51	2264991	426026222	96.97
Totals	2441199	439331273	100.00

Racemic reference:

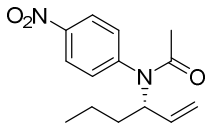


UV Results

Retention Time	Height	Area	Area %
42.00	1200391	139695121	50.13
54.02	927212	138978320	49.87
Totals	2127603	278673441	100.00

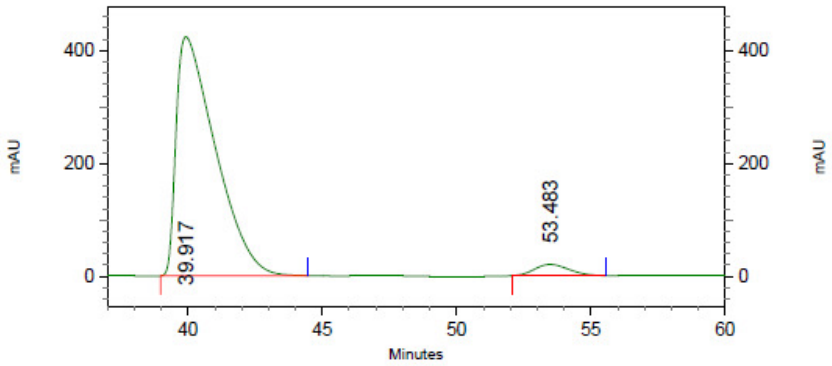
(*S*)-*N*-(Hex-1-en-3-yl)-*N*-(4-nitrophenyl)acetamide ((*ent*)-10e)

Compound:



(*ent*)-10e

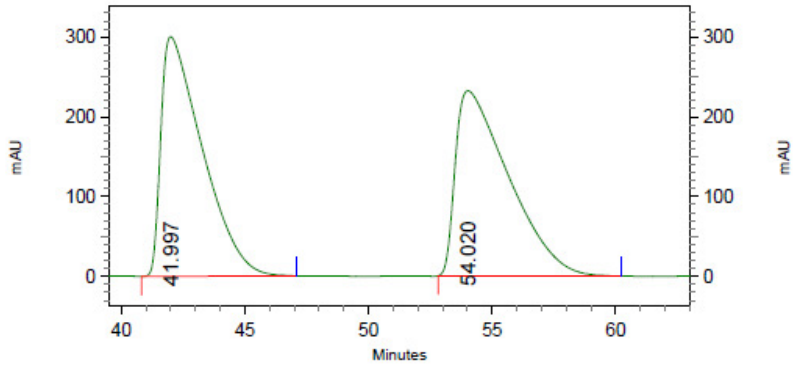
Column: *Chiralpak AS-H*
Method: *n*hexane/*i*PrOH (96/4), 1.0 ml/min, 250 nm



UV Results

Retention Time	Height	Area	Area Percent
39.92	1693275	172225072	96.03
53.48	79962	7123533	3.97
Totals	1773237	179348605	100.00

Racemic reference:

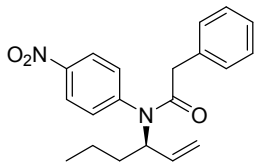


UV Results

Retention Time	Height	Area	Area %
42.00	1200391	139695121	50.13
54.02	927212	138978320	49.87
Totals	2127603	278673441	100.00

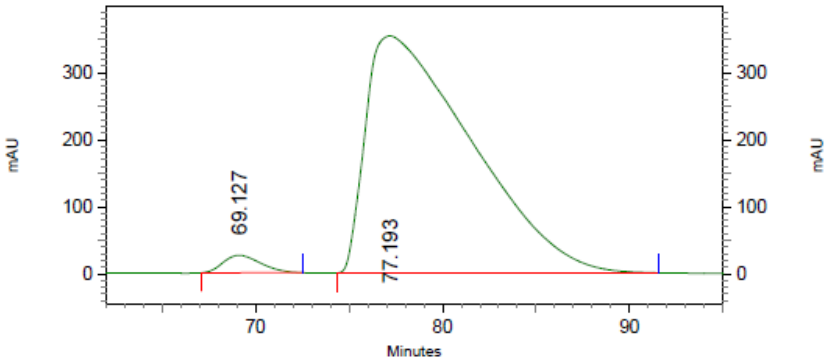
(*R*)-*N*-(Hex-1-en-3-yl)-*N*-(4-nitrophenyl)-2-phenylacetamide (10f)

Compound:



10f

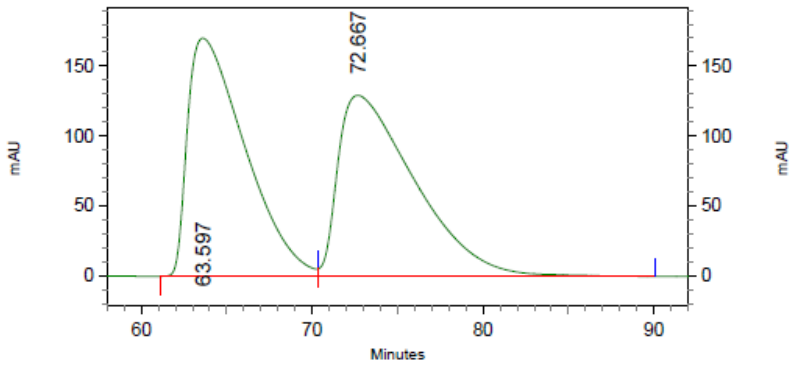
Column: *Chiralpak AS-H*
Method: *n*hexane/*i*PrOH (99/1), 0.8 ml/min, 250 nm



UV Results

Retention Time	Height	Area	Area Percent
69.13	104507	14579767	2.56
77.19	1414643	555387762	97.44
Totals	1519150	569967529	100.00

Racemic reference:

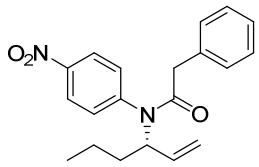


UV Results

Retention Time	Height	Area	Area %
63.60	680593	159083815	49.64
72.67	517449	161397341	50.36
Totals	1198042	320481156	100.00

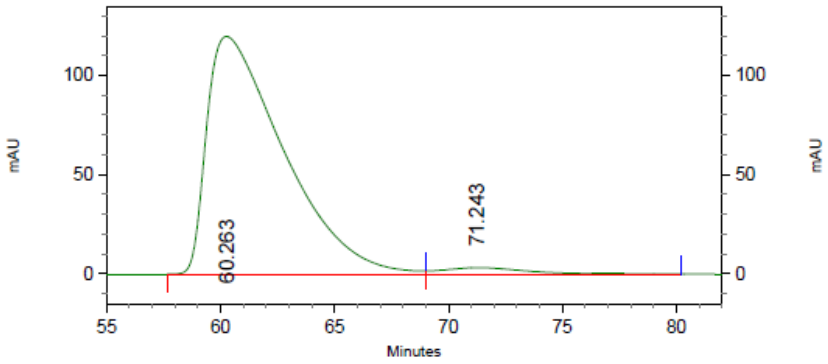
(*S*)-*N*-(Hex-1-en-3-yl)-*N*-(4-nitrophenyl)-2-phenylacetamide ((*ent*)-10f)

Compound:



(*ent*)-10f

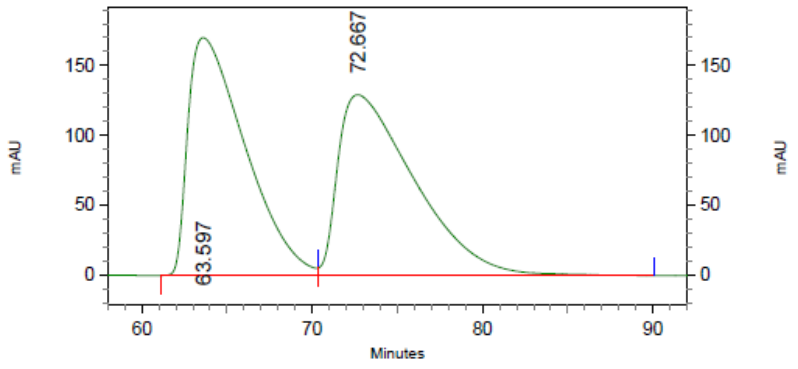
Column: *Chiralpak AS-H*
Method: *n*hexane/*i*PrOH (99/1), 0.8 ml/min, 250 nm



UV Results

Retention Time	Height	Area	Area Percent
60.26	479409	112201312	97.27
71.24	12821	3153737	2.73
Totals	492230	115355049	100.00

Racemic reference:

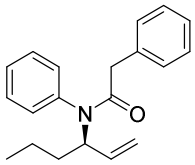


UV Results

Retention Time	Height	Area	Area %
63.60	680593	159083815	49.64
72.67	517449	161397341	50.36
Totals	1198042	320481156	100.00

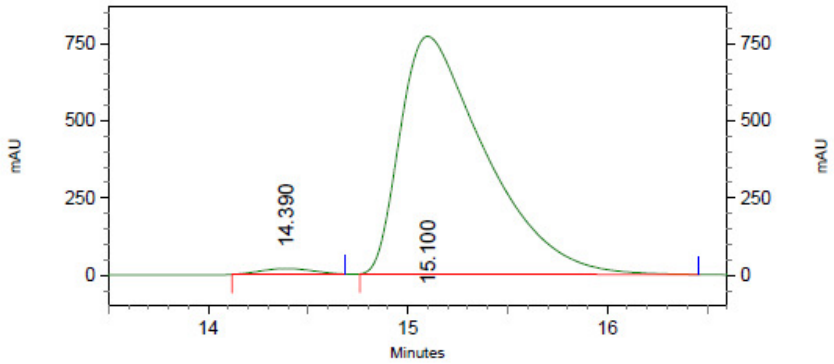
(*R*)-*N*-(Hex-1-en-3-yl)-*N*,2-diphenylacetamide (10g)

Compound:



10g

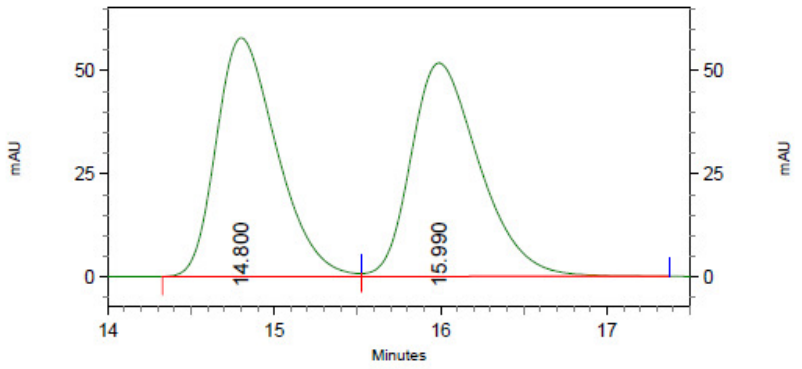
Column: *Chiralpak OD-H*
Method: *n*hexane/*i*PrOH (99.5/0.5), 1.0 ml/min, 250 nm



UV Results

Retention Time	Height	Area	Area Percent
14.39	75203	1342038	1.44
15.10	3079112	91638905	98.56
Totals	3154315	92980943	100.00

Racemic reference:

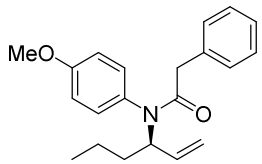


UV Results

Retention Time	Height	Area	Area %
14.80	231315	5854389	49.75
15.99	206957	5913760	50.25
Totals	438272	11768149	100.00

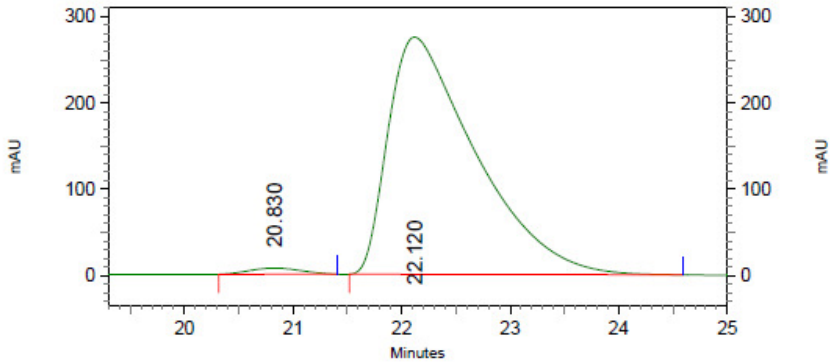
(*R*)-*N*-(Hex-1-en-3-yl)-*N*-(4-methoxyphenyl)-2-phenylacetamide (10h)

Compound:



10h

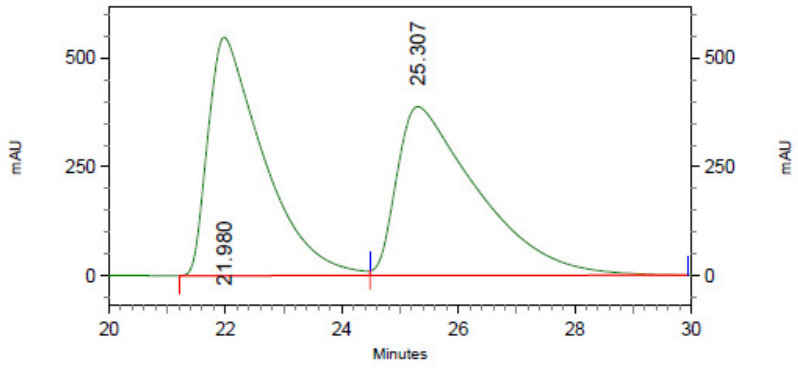
Column: *Chiralpak OD-H*
Method: *n*hexane/*i*PrOH (99.5/0.5), 1.0 ml/min, 250 nm



UV Results

Retention Time	Height	Area	Area Percent
20.83	28968	955845	1.53
22.12	1099892	61579853	98.47
Totals	1128860	62535698	100.00

Racemic reference:

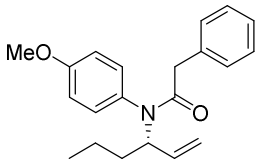


UV Results

Retention Time	Height	Area	Area %
21.98	2186734	145000785	49.24
25.31	1548836	149497607	50.76
Totals	3735570	294498392	100.00

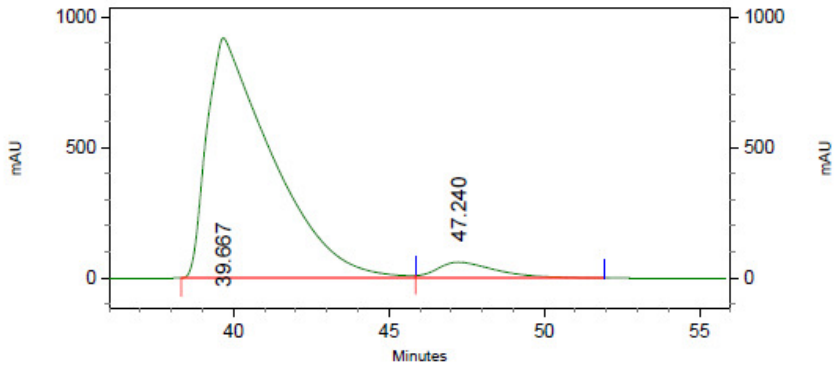
(S)-*N*-(Hex-1-en-3-yl)-*N*-(4-methoxyphenyl)-2-phenylacetamide ((*ent*)-10h)

Compound:



(*ent*)-10h

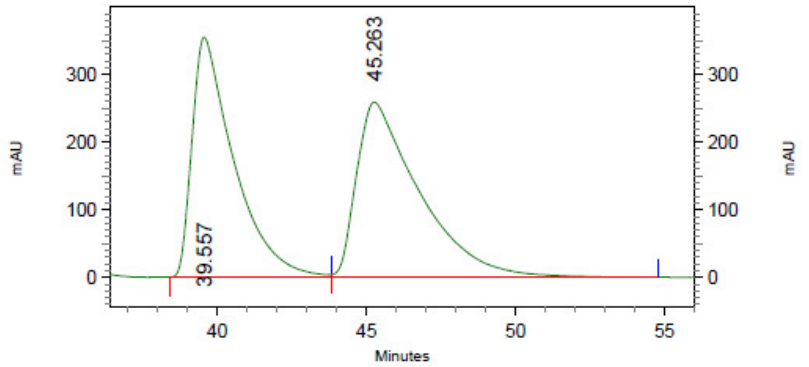
Column: *Chiralpak OD-H*
Method: *n*hexane/*i*PrOH (99.5/0.5), 0.5 ml/min, 250 nm



UV Results

Retention Time	Height	Area	Area Percent
39.67	3675864	537606759	94.52
47.24	239053	31175676	5.48
Totals	3914917	568782435	100.00

Racemic reference:

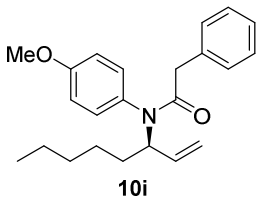


UV Results

Retention Time	Height	Area	Area %
39.56	1421333	138510923	47.73
45.26	1038226	151702304	52.27
Totals	2459559	290213227	100.00

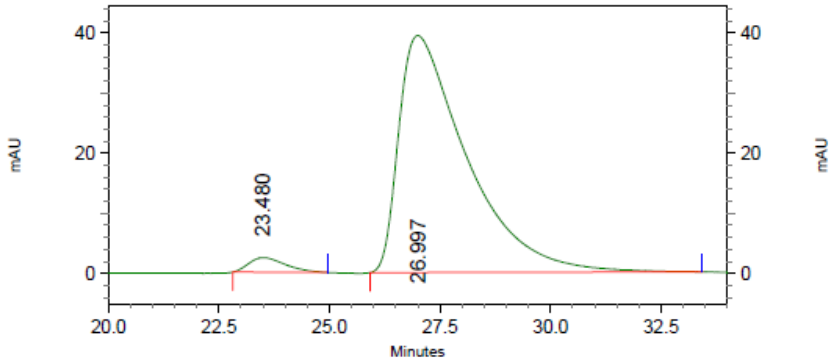
(*R*)-*N*-(4-Methoxyphenyl)-*N*-(oct-1-en-3-yl)-2-phenylacetamide(10i)

Compound:



10i

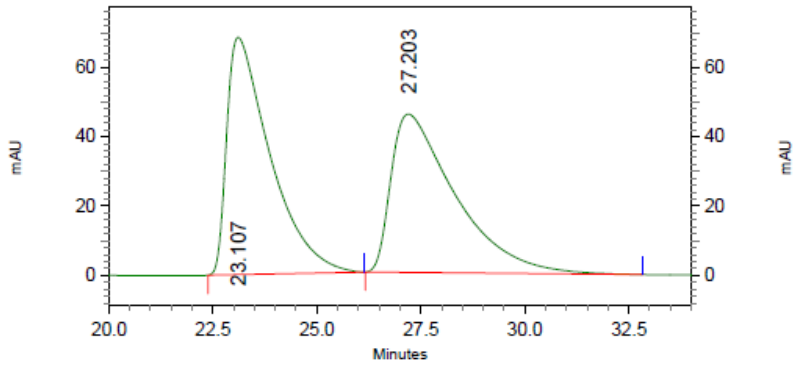
Column: *Chiralpak AD-H*
Method: *n*hexane/*i*PrOH (99.2/0.8), 1.1 ml/min, 250 nm



UV Results

Retention Time	Height	Area	Area Percent
23.48	9690	555879	3.19
27.00	157586	16887366	96.81
Totals	167276	17443245	100.00

Racemic reference:

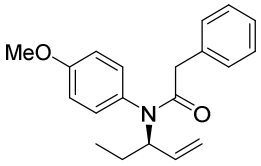


UV Results

Retention Time	Height	Area	Area %
23.11	273744	19823003	50.28
27.20	182601	19598799	49.72
Totals	456345	39421802	100.00

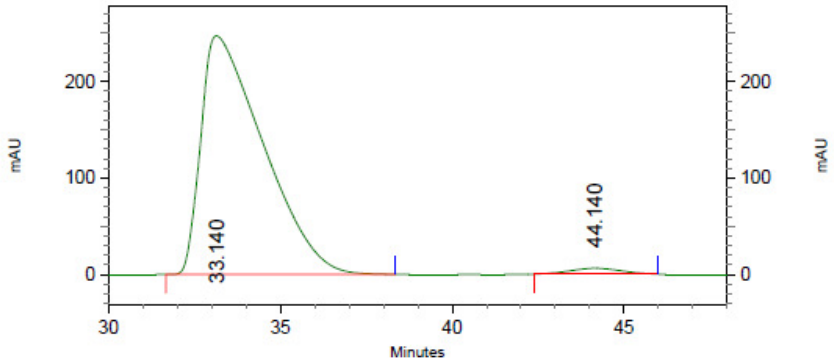
(*R*)-*N*-(4-Methoxyphenyl)-*N*-(pent-1-en-3-yl)-2-phenylacetamide (10j)

Compound:



10j

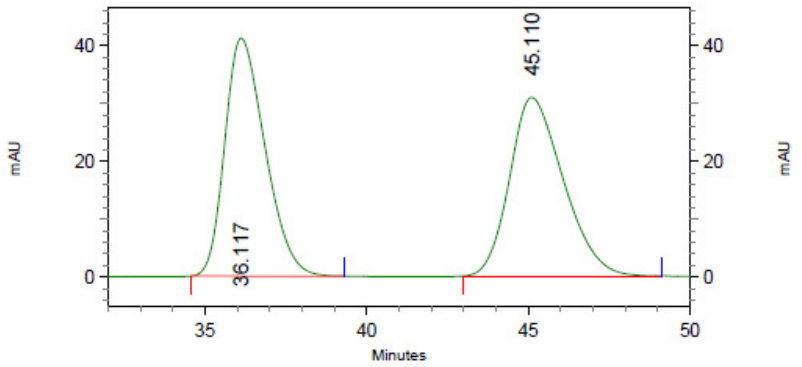
Column: *Chiralpak AS-H*
Method: *n*hexane/*i*PrOH (99/1), 1.0 ml/min, 250 nm



UV Results

Retention Time	Height	Area	Area Percent
33.14	989570	124733455	98.05
44.14	24278	2484027	1.95
Totals	1013848	127217482	100.00

Racemic reference:

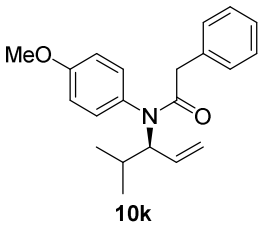


UV Results

Retention Time	Height	Area	Area %
36.12	164528	14252824	49.97
45.11	123511	14267714	50.03
Totals	288039	28520538	100.00

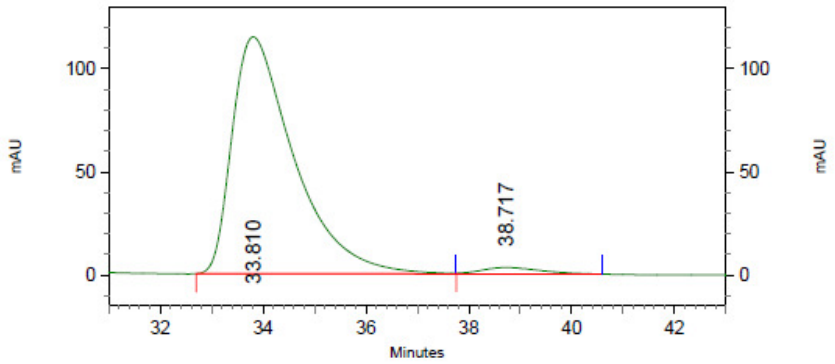
(*R*)-*N*-(4-Methoxyphenyl)-*N*-(4-methylpent-1-en-3-yl)-2-phenylacetamide (10k)

Compound:



10k

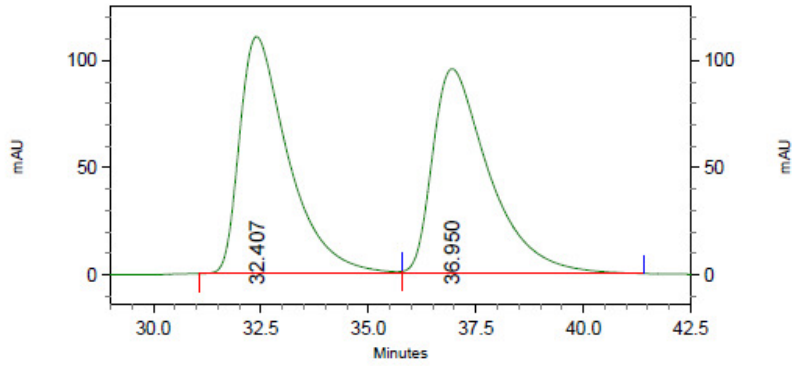
Column: *Chiralpak AD-H*
Method: *n*hexane/*i*PrOH (99/1), 1.0 ml/min, 250 nm



UV Results

Retention Time	Height	Area	Area Percent
33.81	459368	38536637	97.40
38.72	12866	1026833	2.60
Totals	472234	39563470	100.00

Racemic reference:

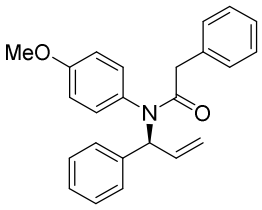


UV Results

Retention Time	Height	Area	Area %
32.41	441541	34898976	49.90
36.95	381735	35038954	50.10
Totals	823276	69937930	100.00

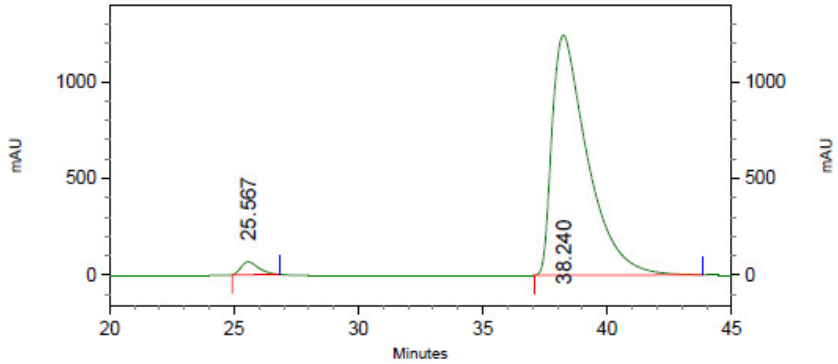
(*S*)-*N*-(4-Methoxyphenyl)-2-phenyl-*N*-(1-phenylallyl)acetamide (10l)

Compound:



10l

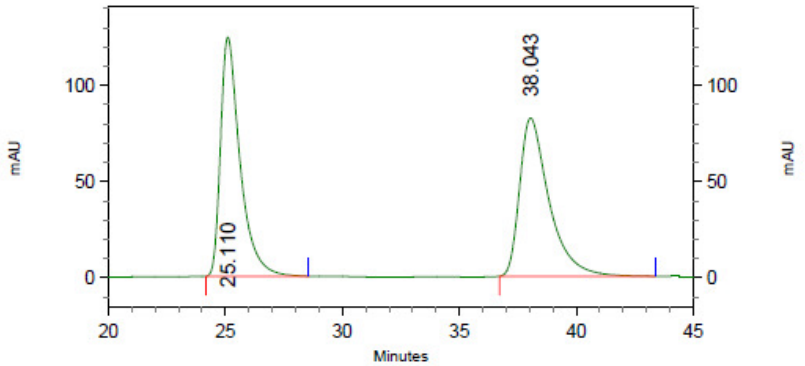
Column: *Chiralpak AD-H*
Method: *n*hexane/*i*PrOH (95/5), 1.0 ml/min, 250 nm



UV Results

Retention Time	Height	Area	Area Percent
25.57	264643	12959655	2.57
38.24	4961754	490774246	97.43
Totals	5226397	503733901	100.00

Racemic reference:

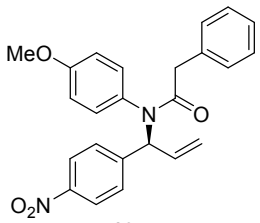


UV Results

Retention Time	Height	Area	Area %
25.11	497583	28868095	50.04
38.04	329004	28825049	49.96
Totals	826587	57693144	100.00

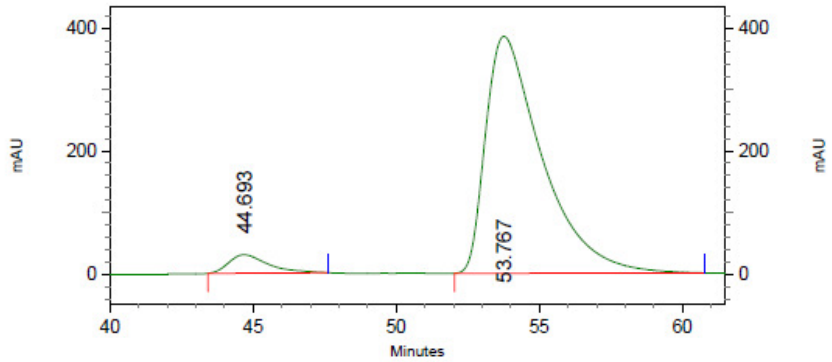
(*S*)-*N*-(4-Methoxyphenyl)-*N*-(1-(4-nitrophenyl)allyl)-2-phenylacetamide (10m)

Compound:



10m

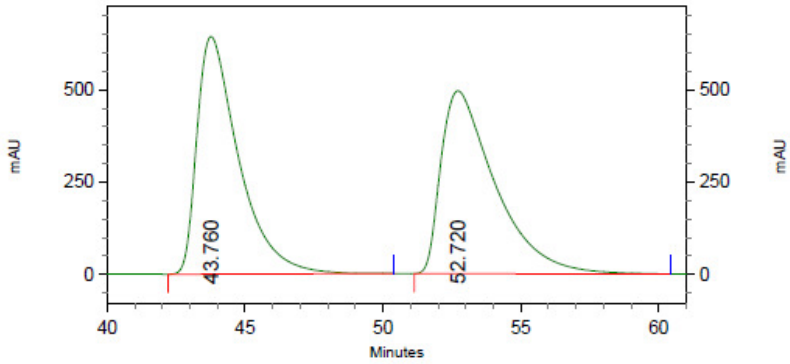
Column: *Chiralpak AD-H*
Method: *n*hexane/*i*PrOH (93/7), 1.0 ml/min, 250 nm



UV Results

Retention Time	Height	Area	Area Percent
44.69	119776	11427540	5.13
53.77	1539616	211446841	94.87
Totals	1659392	222874381	100.00

Racemic reference:

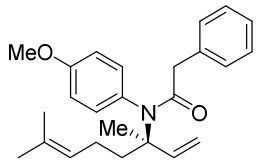


UV Results

Retention Time	Height	Area	Area %
43.76	2571144	270733009	50.00
52.72	1978847	270723678	50.00
Totals	4549991	541456687	100.00

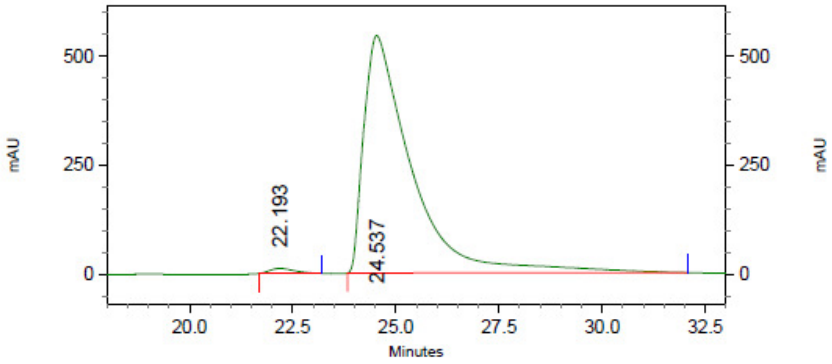
(*R*)-*N*-(3,7-Dimethylocta-1,6-dien-3-yl)-*N*-(4-methoxyphenyl)-2-phenylacetamide (10n)

Compound:



10n

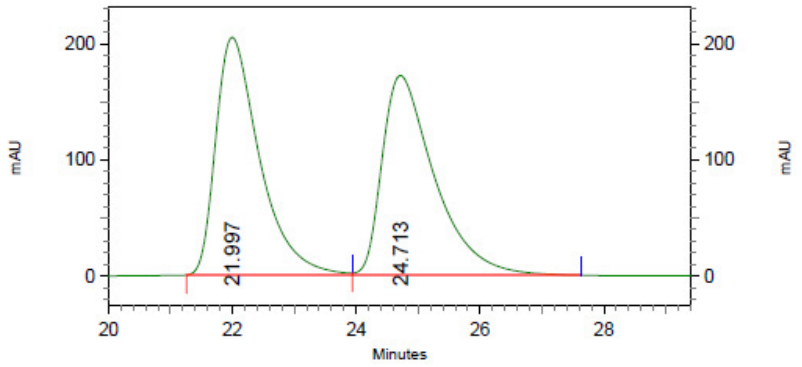
Column: *Chiralpak AD-H*
Method: *n*hexane/*i*PrOH (99/1), 1.0 ml/min, 250 nm



UV Results

Retention Time	Height	Area	Area Percent
22.19	45778	1875097	1.06
24.54	2177843	175458890	98.94
Totals	2223621	177333987	100.00

Racemic reference:

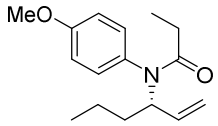


UV Results

Retention Time	Height	Area	Area %
22.00	817422	40223516	49.93
24.71	686481	40338059	50.07
Totals	1503903	80561575	100.00

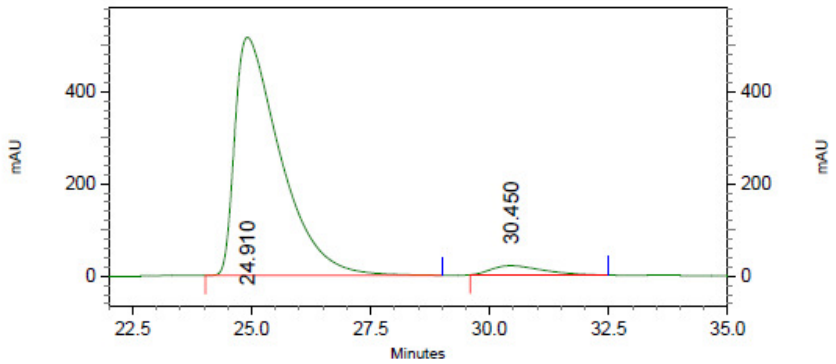
(*S*)-*N*-(Hex-1-en-3-yl)-*N*-(4-methoxyphenyl)propionamide ((*ent*)-10o)

Compound:



(*ent*)-10o

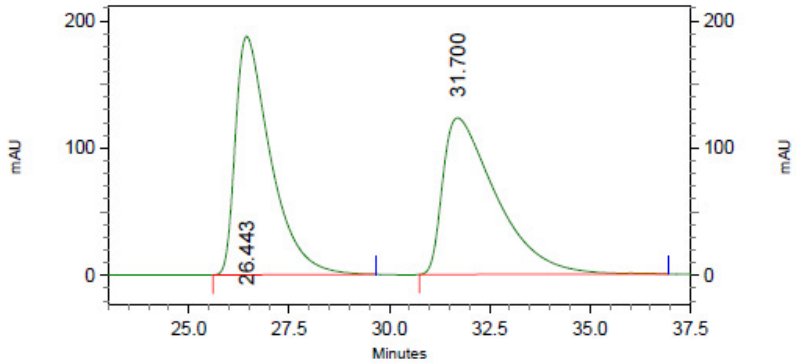
Column: *Chiralpak AD-H*
Method: *n*hexane/*i*PrOH (99.4/0.6), 1.0 ml/min, 250 nm



UV Results

Retention Time	Height	Area	Area Percent
24.91	2065812	137836072	95.83
30.45	79924	5999156	4.17
Totals	2145736	143835228	100.00

Racemic reference:

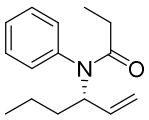


UV Results

Retention Time	Height	Area	Area %
26.44	748825	45718453	50.01
31.70	491649	45698024	49.99
Totals	1240474	91416477	100.00

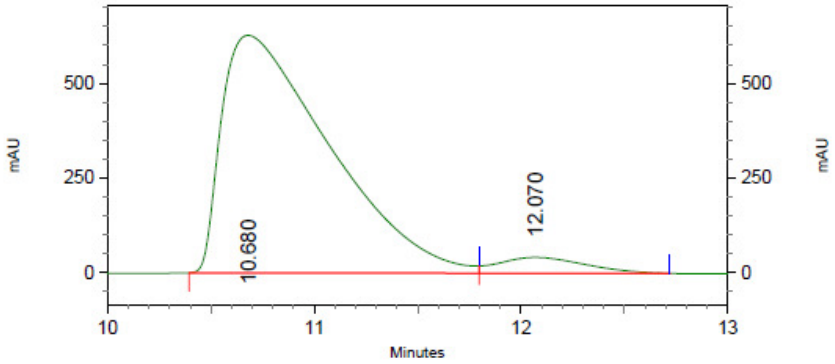
(*S*)-*N*-(Hex-1-en-3-yl)-*N*-phenylpropionamide ((*ent*)-10p)

Compound:



(*ent*)-10p

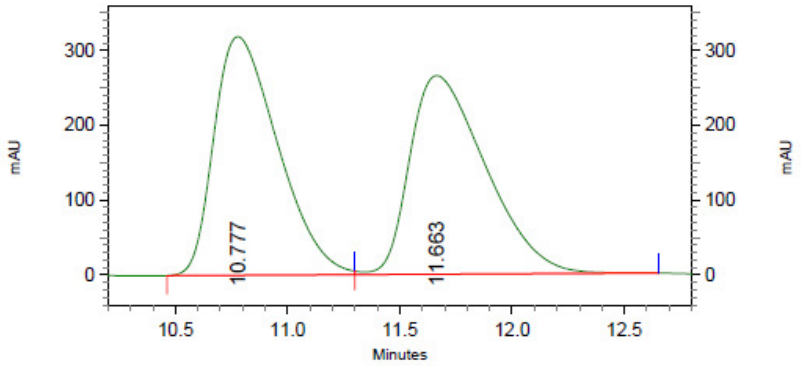
Column: *Chiralpak AS-H*
Method: *n*hexane/*i*PrOH (99.5/0.5), 1.0 ml/min, 250 nm



UV Results

Retention Time	Height	Area	Area Percent
10.68	2508981	90751217	94.93
12.07	167260	4846645	5.07
Totals	2676241	95597862	100.00

Racemic reference:

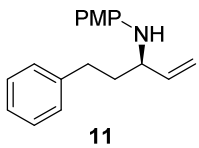


UV Results

Retention Time	Height	Area	Area %
10.78	1274790	25202491	50.29
11.66	1060709	24913499	49.71
Totals	2335499	50115990	100.00

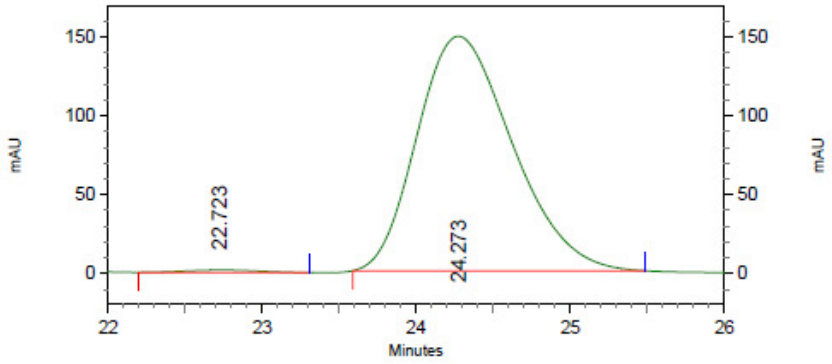
(*R*)-*N*-(4-Methoxyphenyl)-5-phenylpent-1-en-2-amine (11)

Compound:



11

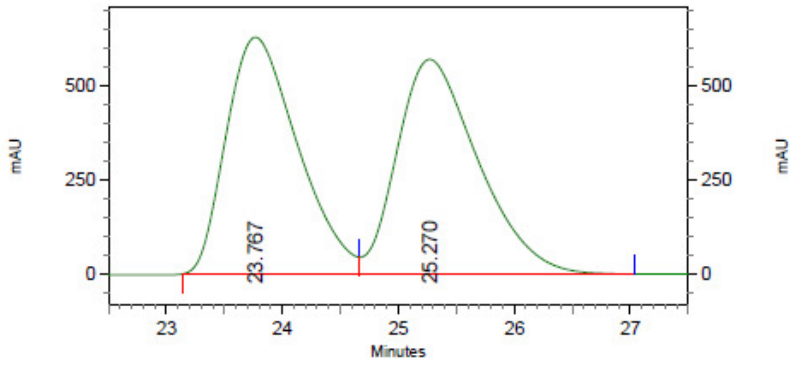
Column: Chiralpak OD-H
Method: *n*hexane/*i*PrOH (98.2/1.8), 0.8 ml/min, 250 nm



UV Results

Retention Time	Height	Area	Area Percent
22.72	6184	206467	0.80
24.27	593878	25547745	99.20
Totals	600062	25754212	100.00

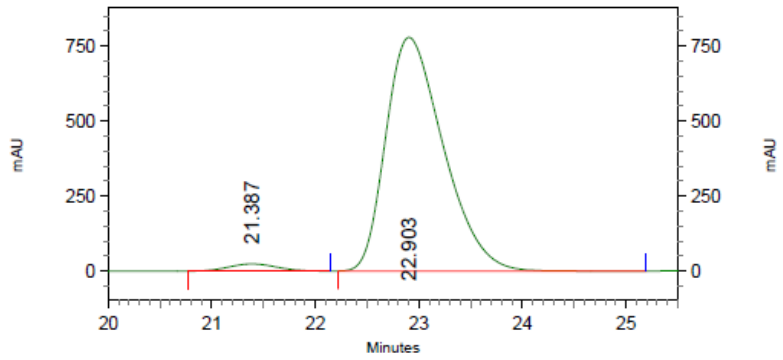
racemic reference:



UV Results

Retention Time	Height	Area	Area %
23.77	2505165	107305928	49.34
25.27	2271522	110191186	50.66
Totals	4776687	217497114	100.00

Reference:

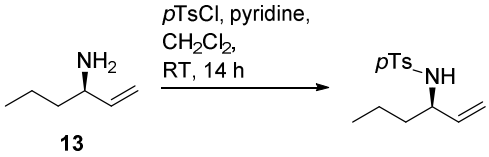


UV Results

Retention Time	Height	Area	Area %
21.39	95660	3085069	2.50
22.90	3113848	120438442	97.50
Totals	3209508	123523511	100.00

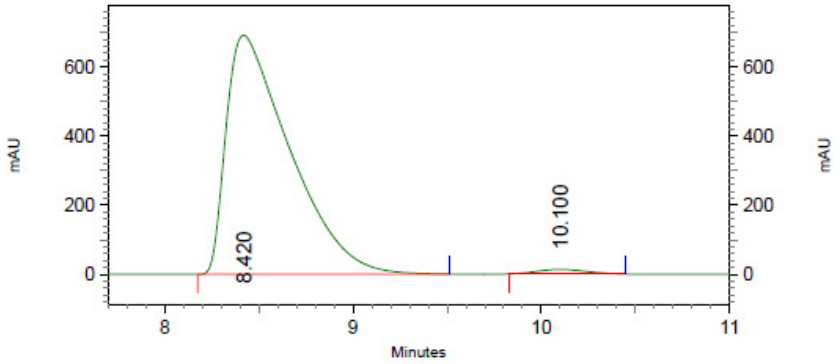
(*R*)-Hex-1-en-3-amine (13) *via* Tosyl Protection

Compound:



13

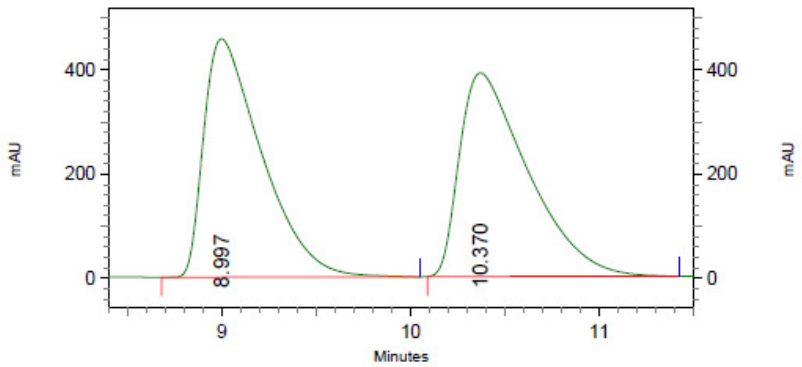
Column: *Chiralpak OD-H*
Method: *n*hexane/*i*PrOH (94/6), 1.0 ml/min, 250 nm



UV Results

Retention Time	Height	Area	Area Percent
8.42	2764665	64944144	98.58
10.10	51550	933230	1.42
Totals	2816215	65877374	100.00

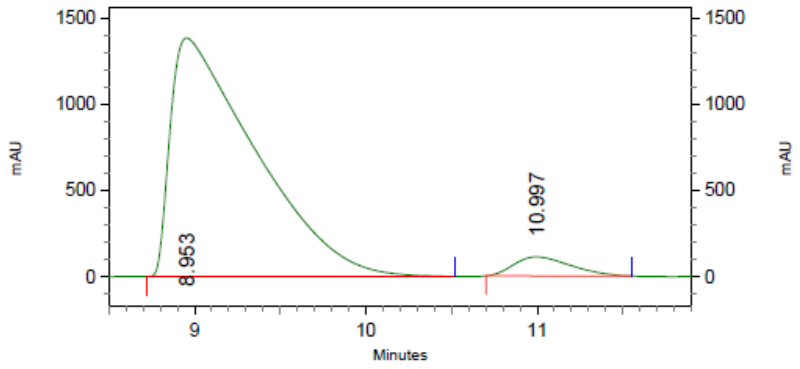
Racemic reference:



UV Results

Retention Time	Height	Area	Area %
9.00	1831669	39100791	49.84
10.37	1563818	39344027	50.16
Totals	3395487	78444818	100.00

Reference:



UV Results

Retention Time	Height	Area	Area %
8.95	5520796	189997385	94.94
11.00	437950	10133548	5.06
Totals	5958746	200130933	100.00

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