Angewandte Chemie
Eine Zeitschrift der Gesellschaft Deutscher Chemiker

Supporting Information

69451 Weinheim, Germany

Asymmetric Formation of Allylic Amines with N-Substituted Quaternary Stereocenters by Pd(II)-Catalyzed Aza-Claisen Rearrangements

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Table of Contents

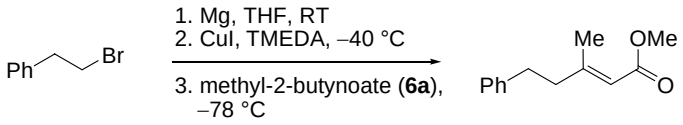
Experimental

Except as otherwise indicated, all reactions were carried out in oven dried glassware under a positive pressure of nitrogen. Anhydrous solvents were prepared by passage of distilled technical grade solvents over a column of activated alumina or molecular sieves under nitrogen atmosphere or purchased as crown-cap solvents. Silver trifluoroacetate (Aldrich, >99.99%) was ground to powder and stored in a glovebox.[1] All other laboratory chemicals were purchased from *Fluka*, *Merck*, *J.T. Baker*, *ABCR* or *Aldrich* and were used without purification. For work-up procedures and flash chromatography, distilled technical grade solvents were used. Unless otherwise indicated, all liquids were added *via* syringe, solids were added neat against a nitrogen flow. Solvents were removed at a heating bath temperature of 40 to 45 °C by rotary evaporation using an appropriate pressure. Non-volatile compounds were dried *in vacuo* at 0.1 mbar. Yields refer to chromatographically purified compounds and are calculated in mol% of the used starting material. Except as otherwise indicated, reactions were magnetically stirred and monitored by high performance liquid chromatography (HPLC) (reversed phase column, H_2O/MeCN gradient) or by thin layer chromatography (TLC) using silica gel plates from *Merck (silica gel 60 F_{254})*. Visualization occurred by fluorescence quenching under UV light and by staining with $KMnO_4$ (0.5% in 1 M aqueous NaOH). Purification by flash chromatography was performed on silica gel 60 (32-62) provided by *Fluka* using a forced flow of eluent at 0.2-0.4 bar pressure. Racemic reference samples of the rearrangement products **5** needed for chiral HPLC were prepared by stirring the corresponding imidate (30 mg) with $PdCl_2(MeCN)_2$ (2 mg) in DCM (1 mL) for 1-2 hours, followed by filtration over a plug of silica with DCM. NMR spectra were recorded on a *Varian Gemini 300* and a *Varian Mercury 300* spectrometer operating at 300 MHz (^{1}H), 75 MHz (^{13}C), 122 MHz (^{31}P) and 282 MHz (^{19}F). Chemical shifts δ are referred in terms of ppm and *J* coupling constants are given in Hz. Abbreviations for multiplicity are

as follows: *s* (singulet), *d* (doublet), *t* (triplet), *q* (quadruplet), *m* (multiplet), *bs/bm* (broad singulet/broad multiplet). IR spectra were recorded on a *Perkin Elmer Spectrum RX I FT-IR* and the signals are given by wave numbers (cm^{-1}). Samples were prepared by the thin film technique. Optical rotation was measured on a *Jasco DIP-100 digital polarimeter* operating at the sodium D line with a 100 mm path length cell. Melting points were measured using a *Büchi 535 melting point apparatus* in open glass capillaries and are uncorrected. Mass spectra were obtained from the ETH Zürich MS Service. HI-RES MALDI spectra were recorded using *anIon Spec 4.7 T Ultima HiRres FT-ICR MS MALDI-FT-ICR MS* employing 3HPA (3-Hydroxy-picolinic acid) and two layer technique in positive or negative mode. High resolution EI mass spectra were performed on a *Micromass AutoSpec Ultima* and were calibrated with perfluorotributylamine (PFTBA) and Ultramark 1621 as internal standard. Low resolution ESI was performed on a TSQ7000 with a home-built ESI-source and flow rates of MeOH:water 19:1 of 2-3 µL/min or on a 4.7 T IonsSpec HiRes FT-ICR MS employing a waters 2spray source. Combustion analysis was performed by the Mikroelementaranalytisches Laboratorium at ETH Zürich.

Preparation of 3,3-disubstituted allylic alcohols and precursors

(*E*)-3-Methyl-5-phenylpent-2-enoic acid methyl ester



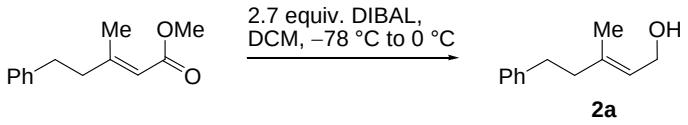
Magnesium turnings were activated by grinding in a mortar inside a glovebox, the metal (0.840 g, 35 mmol, ca. 1.4 equiv.) was suspended in THF (50 mL) and a few drops of a solution of iodine in MTBE were added. Then 2-phenylethylbromide (4.625 g, 25 mmol, 1 equiv.) was added portionwise at RT. After addition of the first portion, the flask was placed in an ultrasonic bath for five minutes or until the reaction had started, visible by the disappearance of the yellow color of iodine. The temperature was kept below 45 °C by cooling in a water bath.

A suspension of CuI (1.840 g, 9.6 mmol, 1.2 equiv.) in THF (25 mL) was cooled to ca. –40 °C. TMEDA (N,N,N',N'-tetramethylethylenediamine) (2.78 g, 3.61 mL, 24.0 mmol, 3 equiv.) was subsequently added followed by the solution of 2-phenylethylmagnesiumbromide (16.0 mL, 8.0 mmol, 1 equiv.). The grey suspension, which may turn to yellow-brown, was stirred at –40 °C for 30 min and then cooled to –78 °C. Methyl-2-butynoate (**6a**, 0.784 g, 784 µL, 8.0 mmol, 1.0 equiv.) was added in one portion and the resulting grey to orange suspension was stirred for 2 h at –78 °C. The reaction was quenched by addition of methanol (technical grade, ca. 10 mL) and the cooling bath was subsequently removed. Saturated aqueous ammonium sulfate (ca. 10 mL) was added and the suspension was warmed to RT. Aqueous ammonia (ca. 20%) was added until complete dissolution of all solids, followed by extraction with MTBE (2 x) and washing of the combined organic phases with additional aqueous ammonia. The solution was dried over $MgSO_4$ and the volatiles were removed *in vacuo*. The (*E*)/(*Z*)-ratio of 98:2 was determined by ^{1}H-NMR (allylic

CH_3-group: (*E*): 2.21 ppm, (*Z*): 1.89 ppm). The isomers were separated by column chromatography (pentane / diethylether 16:1) to give the desired (*E*)-isomer as a colorless oil (1.440 g, 7.06 mmol, 88%). Similar results were obtained with ethyl ester **6b**.

$C_{13}H_{16}O_2$, MW: 204.26 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.29-7.16 (*m*, 5 H, Ar-*H*), 5.69 (*app d*, *J* = 1.2, 1H, C=C*H*), 3.68 (*s*, 3 H, OCH_3), 2.78 (*dd*, *J* = 10.5, *J* = 7.8, 2 H, Ar-CH_2-CH_2), 2.45 (*dd*, *J* = 10.5, *J* = 7.8, 2 H, Ar-CH_2-CH_2), 2.21 (*app d*, *J* = 1.2, 3H, C=C-CH_3). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 166.9, 159.1, 140.8, 128.3, 126.0, 115.5, 50.8, 42.7, 33.9, 19.0. **IR (film):** ν = 3027, 2948, 1721, 1650, 1603, 1495, 1435, 1385, 1359, 1280, 1225, 1147, 1080, 1048. **MS (EI) *m/z*:** 91.0 [100%], 204.1 [2.2%, MH^+]. **HRMS (EI) *m/z*:** Calc. for [MH^+]: 204.1145. Found: 204.1147. **Anal. Calcd. for $C_{13}H_{16}O_2$:** C, 76.44; H, 7.89. Found: C, 76.16, H, 7.91.

(*E*)-3-Methyl-5-phenylpent-2-enol (2a)



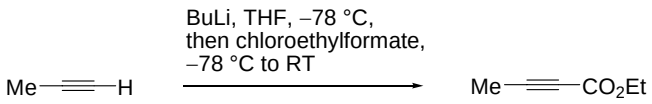
2a

(*E*)-3-Methyl-5-phenylpent-2-enoic acid methyl ester (1.440 g, 7.06 mmol, 1.0 equiv) was dissolved in DCM (20 mL) and cooled to −78 °C. DIBAL (1 M in DCM or hexanes, 20 mL, 20 mmol, 2.8 equiv.) was added in portions within 1 min. After 30 min stirring at −78 °C, the solution was slowly warmed to 0 °C and stirred for 1 h, then cooled again to −78 °C. The reaction was quenched by dropwise addition of 1 N aqueous HCl. After warming to RT, additional 1 N HCl and DCM were added until complete dissolution of the precipitate. The aqueous phase was extracted twice with DCM and the combined organic phases were dried over $MgSO_4$. Subsequent solvent removal under

reduced pressure gave **2a** (1.205 g, 6.84 mmol, 97%) as colorless oil which did not require further purification.

$C_{12}H_{16}O$, MW: 176.25 g/mol. **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 7.30-7.28 (*m*, 2 H, Ar-*H*), 7.20-7.16 (*m*, 2 H, Ar-*H*), 5.41 (*t*, *J* = 6.9, 1 H, C=C*H*), 4.14 (*t*, *J* = , 2 H, CH_2OH), 2.27 & 2.32 (*t*, *J* = 7.5, 2 H each, CH_2CH_2), 1.73 (*s*, 3 H, C=C-CH_3), 1.03 (*t*, *J* = 5.4, 1 H, CH$_2$O*H*). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 141.8, 138.8, 128.25, 128.20, 125.7, 123.8, 59.2, 41.4, 34.4, 16.5. **IR (film):** ν = 3334, 2927, 2858, 1669, 1602, 1495, 1453, 1382, 1001. **MS (EI) *m/z*:** 91.0 [100%], 176.1 [3.3%, M^+]. **HRMS (EI) *m/z*:** Calc. for [M^+]: 176.1196. Found: 176.1198. **Anal. Calcd. for $C_{12}H_{16}O$:** C, 81.77; H, 9.15. Found: C, 81.66, H, 9.23.

Ethyl-2-butynoate (6b)[2]

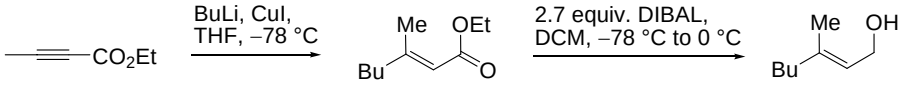


6b

Propyne (6.0 g, 0.150 mol, 1 equiv.) was condensed at –78 °C into a flask containing THF (100 mL). *n*-Butyllithium (1.6 M in hexane, 93 mL, 0.150 mol, 1 equiv.) was added *via* cannula within 10 min and the resulting turbid mixture was stirred at –78 °C for 30 min. Ethylchloroformate (17.4 g, 15.4 mL, 0.160 mol, 1.07 equiv.) was added in one portion. After 10 min, the mixture was allowed to warm to RT, then saturated aqueous ammonium chloride (ca. 10 mL) was added resulting in the formation of a white precipitate. The suspension was washed with water (50 mL), the organic phase was dried over $MgSO_4$ and most of the solvent was removed *in vacuo*. Distillation (60 mbar, 80 °C bath temperature) afforded **6b** as colorless oil (13.76 g, 0.122 mol, 81%).

$C_6H_8O_2$, MW: 112.13 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 4.20 (*q, J* = 7.2, 2 H, $CO_2CH_2CH_3$), 1.97 (s, 3 H, $CCCH_3$), 1.29 (*t, J* = 7.2, 3 H, $CO_2CH_2CH_3$). All other analytical data were in accordance with the purchased material.

(*E*)-3-Methylhept-2-enol (2b)



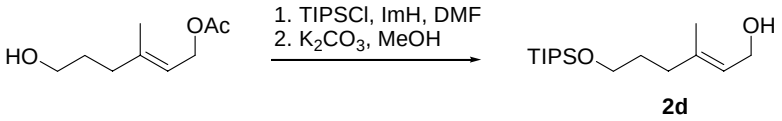
6b **2b**

3-Methylhept-2-enoic acid ethyl ester was prepared by CuI-mediated addition of BuLi to ethylbutynoate, following a literature procedure.[3] To a solution of this ester (0.520 g, 1.88 mmol, 1.0 equiv.) in DCM (10 mL) at −78 °C was added DIBAL (1 M in DCM, 5 mL, 5 mmol, 2.7 equiv.) in portions within 1 min. After 30 min stirring at −78 °C, the solution was slowly warmed to 0 °C and stirred for 1 h, then cooled again to −78 °C. The reaction was quenched by dropwise addition of 1 N aqueous HCl. After warming to RT, additional 1 N aqueous HCl and DCM were added until complete dissolution of the precipitate. The aqueous phase was extracted twice with DCM and the combined organic phases were dried over $MgSO_4$. Subsequent solvent removal under reduced pressure gave **2b** (0.400 g, 1.7 mmol, 90%) as colorless oil which did not require further purification.

$C_8H_{16}O$, MW: 128.21 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 5.36 (*t*, *J* = 0.9, 1 H, C=C*H*), 4.10 (*d*, *J* = 5.4, 2 H, CH_2OH), 1.98 (*t*, *J* = 7.2, 2 H, $CH_2CH_2CH_2CH_3$), 1.89 (*bs*, 1 H, O*H*), 1.63 (*s*, 3 H, C=CCH_3), 1.39-1.27 (*m*, 4 H, $CH_2CH_2CH_2CH_3$), 0.89 (*t*, *J* = 6.9, 3 H, $CH_2CH_2CH_2CH_3$). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 139.7, 124.0, 59.1, 39.1, 29.7, 22.2, 15.9, 13.8. **IR (film):** ν = 3330, 2958, 2873, 1670, 1467, 1380, 1238, 1181, 1075, 999, 789, 730. **MS (EI) *m/z*:** 128.1 [4.5%,

M^+]. **HRMS (EI) *m/z*:** Calc. for [M^+]: 128.1196. Found: 128.1195. **Anal. Calcd. for $C_8H_{16}O$:** C, 74.94; H, 12.58. Found: C, 74.65, H, 12.44.

(*E*)-3-Methyl-6-triisopropylsilanyloxyhex-2-enol (2d)

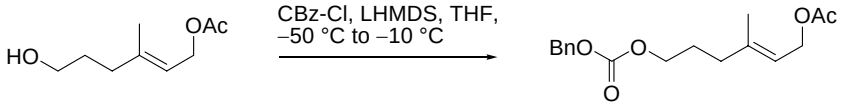


2d

Triisopropylsilylchloride (0.425 g, 473 µL, 2.2 mmol, 1.1 equiv.) was added to a solution of (*E*)-acetic acid 6-hydroxy-3-methylhex-2-enyl ester[4] (0.344 g, 2.0 mmol, 1.0 equiv.) and imidazole (0.544 g, 8.0 mmol, 4 equiv.) in DMF (20 mL). The resulting clear solution was stirred for 2-3 days at RT (TLC-monitoring, pentane / EtOAc 9:1). The reaction was quenched by addition of water and the mixture was extracted twice with MTBE. The combined organic phases were washed three times with water to remove residual DMF, dried over $MgSO_4$ and the solvent was removed under reduced pressure to give silylether **2d** as a slightly yellowish oil which was utilized without purification. [^{1}**H-NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 5.36 (*t*, *J* = 7.2, 1 H, C=C*H*), 4.58 (*d*, *J* = 7.2, 2 H, CH_2OH), 3.66 (*t*, *J* = 6.3, 2 H, $CH_2CH_2CH_2$OTIPS), 2.14 (*t*, *J* = 6.9, 2 H, $CH_2CH_2CH_2$OTIPS), 2.05 (*s*, 3 H, $OCOCH_3$), 1.71-1.61 (*m*, 2 + 3 H, C=C-CH_3 & $CH_2CH_2CH_2$OTIPS), 1.10 (*b*, 1 H, CH_2OH), 1.10-1.00 (*m*, 3 + 18 H, Si(C*H*(CH_3)$_2$)$_3$).] The crude silyl ether was dissolved in a 0.2 M solution of potassium carbonate in methanol (ca. 15 mL) and stirred at RT overnight. Water and MTBE were then added and the aqueous phase was extracted with additional MTBE. The combined organic phases were dried over $MgSO_4$, the solvent was removed under reduced pressure and the residue was purified by column chromatography (pentane / EtOAc 4:1) to give **2d** as colorless oil (0.449 g, 1.57 mmol, 78% over two steps).

$C_{16}H_{34}O_2Si$, MW: 286.53 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 5.41 (*t*, *J* = 6.9, 1 H, C=C*H*), 4.12 (*d*, *J* = 6.9, 2 H, CH_2OH), 3.66 (*t*, *J* = 6.3, 2 H, $CH_2CH_2CH_2$OTIPS), 2.08 (*t*, *J* = 7.2, 2 H, C$H_2CH_2CH_2$OTIPS), 1.67-1.60 (*m*, 2 + 3 H, C=C-CH_3 & $CH_2CH_2CH_2$OTIPS), 1.10 (*b*, 1 H, CH_2O*H*), 1.10-1.00 (*m*, 3 + 18 H, Si(C*H*(CH_3)$_2$)$_3$). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 139.4, 123.2, 62.9, 59.4, 35.8, 31.2, 18.1, 16.3, 12.1. **IR (film):** ν = 3324, 2943, 2867, 1463, 1382, 1247, 1108, 1069, 996. **MS (EI) *m/z*:** 95.0 [100%], 243.1 [0.7%, (M-C_3H_7)$^+$]. **HRMS (EI) *m/z*:** Calc. for [M-C_3H_7]$^+$: 243.1775. Found: 243.1773. **Anal. Calcd. for $C_{16}H_{34}O_2Si$:** C, 67.07; H, 11.96; Found: C, 67.05, H, 11.92.

(*E*)-3-Methyl-6-benzyloxycarbonyloxyhex-2-enol acetic acid ester

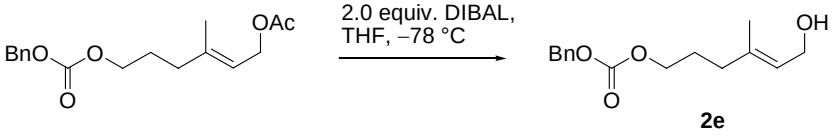


LHMDS (1.0 M in THF, 5.0 mmol, 5.0 mL, 1 equiv.) was added to a solution of (*E*)-acetic acid 6-hydroxy-3-methylhex-2-enyl ester[4] (0.860 g, 5.0 mmol, 1 equiv.) and chloroformic acid benzylester (CBz-Cl, 1.020 g, 6.0 mmol, 1.2 equiv.) in THF (15 mL) at –50 °C. The reaction was allowed to warm to –10 °C, then water was added and the mixture was extracted three times with DCM. The combined organic phases were dried over $MgSO_4$ and the solvent was removed under reduced pressure. The residue was purified by column chromatography (CyH / EtOAc 7:1) to give (*E*)-3-methyl-6-benzyloxycarbonyloxyhex-2-enol acetic acid ester as colorless oil (1.140 g, 3.72 mmol, 75%).

$C_{17}H_{22}O_5$, MW: 306.35 g/mol. **^{1}H-NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.38-7.30 (m, 5 H, arom-H), 5.32 (*t*, *J* = 6.9, 1 H, C=C*H*), 5.14 (*s*, 2 H, OCH_2Ph), 4.56 (*d*, *J* = 7.2, CH_2OAc), 4.12 (*t*, *J* = 6.6, 2 H, OC(O)OC$H_2CH_2CH_2$), 2.10 (*t*, *J* = 7.2, 2 H, OC(O)O$CH_2CH_2CH_2$), 2.03 (*s*, 3 H, OCOCH_3),

1.80 (*dd*, *J* = 7.2, *J* = 6.6, 2 H, OC(O)OCH$_2$C*H*$_2$CH$_2$), 1.68 (*s*, 3 H, C=C-C*H*$_3$). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 170.8, 155.0, 140.5, 135.1, 128.5, 128.4, 128.2, 119.0, 69.3, 67.4, 61.0, 35.2, 26.3, 20.9, 16.2. **IR (film):** ν = 3066, 3034, 2958, 1745, 1671, 1586, 1498, 1455, 1398, 1375, 1265, 1079, 1024, 952, 908, 790, 754, 698. **MS (EI) *m/z*:** 91.0 [100%], 215.0 [1.6%, $(M\text{-}C_7H_7)^+$]. **HRMS (EI) *m/z*:** Calc. for [$(M\text{-}C_7H_7)^+$]: 215.0914. Found: 215.0914. **Anal. Calcd. for $C_{17}H_{22}O_5$:** C, 66.65; H, 7.24; Found: C, 66.73, H, 7.19.

(*E*)-3-Methyl-6-benzyloxycarbonyloxyhex-2-enol (2e)



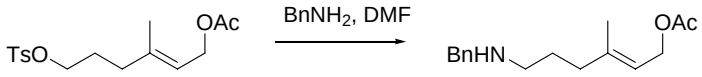
2e

(*E*)-3-Methyl-6-benzyloxycarbonyloxyhex-2-enol acetic acid ester (0.450 g, 1.47 mmol, 1 equiv.) was dissolved in THF (20 mL) and cooled to –78 °C. DIBAL (1.0 M in DCM, 2.94 mmol, 2.94 mL, 2.0 equiv.) was added in two portions within one hour. After two hours reaction time, a saturated solution of potassium sodium tartrate was added. The mixture was warmed to RT and extracted three times with DCM. The combined organic phases were washed with brine and dried over $MgSO_4$. The solvent was removed under reduced pressure and the residue was purified by column chromatography (CyH / EtOAc 2:1 + 3% NEt_3) to yield **2e** as colorless oil (0.314 g, 1.19 mmol, 81%).

$C_{15}H_{20}O_4$, MW: 264.31 g/mol. **^{1}H-NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.39-7.34 (m, 5 H, Ar-H), 5.40 (*t*, *J* = 6.9, 1 H, C=C*H*), 5.14 (*s*, 2 H, OC*H*$_2$Ph), 4.16-4.10 (*m*, 2+ 2 H, C*H*$_2$OAc & OC(O)OC*H*$_2$CH$_2$CH$_2$), 2.09 (*t*, *J* = 7.2, 2 H, OC(O)OCH$_2$CH$_2$C*H*$_2$), 1.84-1.77 (*m*, 2 H, OC(O)OCH$_2$C*H*$_2$CH$_2$), 1.65 (*s*, 3 H, C=C-C*H*$_3$). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 155.1, 137.8, 135.1, 128.5, 128.4, 128.2, 124.2, 69.4, 67.5, 59.0, 35.2, 26.4, 15.9. **IR (film):** ν = 3401,

2959, 1748, 1669, 1653, 1558, 1498, 1456, 1398, 1375, 1265, 1001, 906. **MS (EI) *m/z*:** 91.0 [100%], 173.0 [5%, $(M\text{-}C_7H_7)^+$]. **HRMS (EI) *m/z*:** Calc. for [$(M\text{-}C_7H_7)^+$]: 173.0808. Found: 173.0809. **Anal. Calcd. for $C_{15}H_{20}O_4$:** C, 68.16; H, 7.63; Found: C, 67.90, H, 7.61.

(*E*)-3-Methyl-6-benzylaminohex-2-enol acetic acid ester

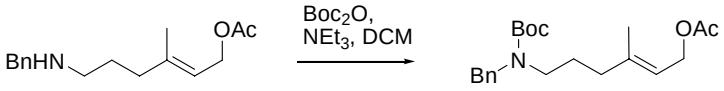


To a solution of (*E*)-3-methyl-6-(toluene-4-sulfonyloxy)hex-2-enol acetic acid ester[4] (1.140 g, 3.50 mmol, 1 equiv.) in DMF (20 mL) at RT was added benzylamine (3.745 g, 3.82 mL, 35 mmol, 10 equiv.). The solution was stirred overnight and then diluted with MTBE and cooled to 0 °C. Saturated sodium hydrogencarbonate-solution was added and the aqueous phase extracted 3 times with MTBE. The combined organic phases were washed with brine and dried over $MgSO_4$. After solvent removal, the residue was purified by column chromatography (CyH / EtOAc 4:1 + 3% NEt_3) to give (*E*)-3-methyl-6-benzylaminohex-2-enol acetic acid ester as colorless oil (0.840 g, 3.21 mmol, 92%).

$C_{16}H_{23}NO_2$, MW: 261.36 g/mol. **^{1}H-NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.32-7.21 (m, 5 H, Ar-*H*), 5.33 (*t*, *J* = 7.2, 1 H, C=C*H*), 4.56 (*d*, *J* = 6.9, 2 H, CH_2OAc), 3.77 (*s*, 2 H, NCH_2Ph), 2.61 (*t*, *J* = 7.2, 2 H, NCH_2CH$_2$CH$_2$), 2.07 (*t, J* = 7.5, 2 H, NCH$_2$CH$_2$CH_2), 2.03 (*s*, 3H, OCOCH_3), 1.69 (*s*, 3 H, C=C-CH_3), 1.61 (*app t*, *J* = 5.4, 2 H, NCH$_2$CH_2CH$_2$), 1.23 (bs, 1H, N*H*). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 170.9, 141.9, 140.4, 128.2, 127.9, 126.7, 118.3, 61.3, 54.1, 49.0, 37.3, 28.0, 21.1, 16.4. **IR (film):** ν = 3026, 2935, 1734, 1700, 1670, 1558, 1495, 1455, 1366, 1234, 1119, 1025, 954, 735. **MS (EI) *m/z*:** 91.0 [100%], 261.1 [1.7%, M^+]. **HRMS (EI) *m/z*:** Calc. for $[M]^+$: 261.1723.

Found: 261.1723. **Anal. Calcd. for $C_{16}H_{23}NO_2$:** C, 73.53; H, 8.87; N, 5.36; Found: C, 73.45, H, 8.90, N, 5.50.

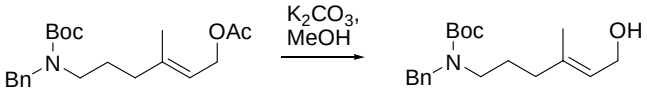
(*E*)-3-Methyl-6-(benzyl-*t*-butyloxycarbonylamino)hex-2-enol acetic acid ester



Triethylamine (0.430 g, 0.598 mL, 4.26 mmol, 1.5 equiv.) was added at 0 °C to a solution of (*E*)-3-methyl-6-benzylaminohex-2-enol acetic acid ester (0.740 g, 2.84 mmol, 1 equiv.) and $(Boc)_2O$ (0.743 g, 3.41 mmol, 1.2 equiv.) in DCM (15 mL). The solution was warmed to RT and stirred for four hours before it was cooled again to 0 °C, followed by the addition of saturated aqueous $NaHCO_3$. The mixture was three times extracted with DCM. The combined organic phases were washed with brine, dried over $MgSO_4$, and the solvent was removed under reduced pressure. The residue was purified by column chromatography (CyH / EtOAc 4:1) to give (*E*)-3-methyl-6-(benzyl-*t*-butyloxycarbonylamino)hex-2-enol acetic acid ester as colorless oil (0.960 g, 2.66 mmol, 94%).

$C_{21}H_{31}NO_4$, MW: 361.47 g/mol. **^{1}H-NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.32-7.19 (m, 5 H, Ar-*H*), 5.28 (*t*, *J* = 6.6, 1 H, C=C*H*), 4.53 (*d*, *J* = 6.9, 2 H, CH_2OAc), 4.40 (*bs*, 2 H, NCH_2Ph), 3.12 (*bs*, 2 H, NCH_2CH$_2$CH$_2$), 2.00 (*s*, 3H, OCOCH_3), 2.00-1.95 (*bm*, 2 H, NCH$_2$CH$_2$CH_2), 1.63 (bs, 3+2 H, C=C-CH_3 & NCH$_2$CH_2CH$_2$), 1.43 (bs, 9 H, C(CH_3)$_3$). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 170.9, 156.0, 141.3, 138.4, 128.3, 127.5, 127.0, 118.5, 79.4, 61.1, 50.4 & 49.8, 46.1, 36.5, 28.3, 25.7, 20.9, 16.2. **IR (film):** ν = 2976, 2934, 1739, 1696, 1558, 1455, 1417, 1366, 1234, 1162, 1126, 1025, 956, 895. **MS (MALDI) *m/z*:** 384.2 [29%, $(MNa)^+$]. **HRMS (MALDI) *m/z*:** Calc. for $[MNa]^+$: 384.2145. Found: 384.2142. **Anal. Calcd. for $C_{21}H_{31}NO_4$:** C, 69.78; H, 8.64; N, 3.87; Found: C, 69.77, H, 8.93, N, 4.04.

(*E*)-3-Methyl-6-(benzyl-*t*-butyloxycarbonylamino)hex-2-enol (2f)

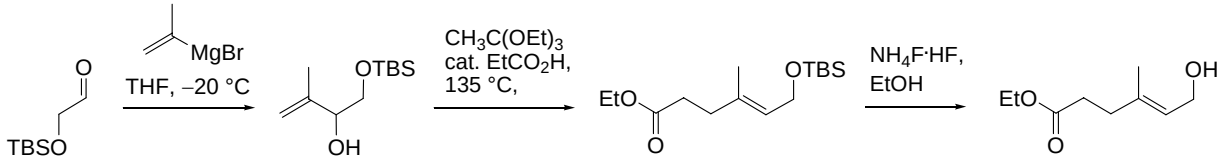


2f

(*E*)-3-Methyl-6-(benzyl-*t*-butyloxycarbonylamino)hex-2-enol acetic acid ester (0.810 g, 2.24 mmol, 1 equiv.) was dissolved in a solution of potassium carbonate (0.031 g, 0.224 mmol, 0.1 equiv.) in methanol (15 mL). After six hours, water and MTBE were added. The phases were separated and the organic layer was washed with brine and dried over $MgSO_4$. After solvent removal, the residue was purified by column chromatography (CyH / EtOAc 2:1 + 3% NEt_3) to give **2f** as colorless oil (0.710 g, 2.23 mmol, quant.).

$C_{19}H_{29}NO_3$, MW: 319.44 g/mol. **^{1}H-NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.32-7.22 (m, 5 H, Ar-*H*), 5.35 (*t*, *J* = 6.6, 1 H, C=C*H*), 4.40 (*bs*, 2 H, NCH_2Ph), 4.09 (*bs*, 2 H, CH_2OH), 3.13 (*bs*, 2 H, NCH_2CH$_2$CH$_2$), 1.94 (*bm*, 2 H, NCH$_2$CH$_2$CH_2), 1.80 (*bs*, 1H, O*H*), 1.61 (*bs*, 3+2 H, C=C-CH_3 & NCH$_2$CH_2CH$_2$), 1.43 (*bs*, 9, C(CH_3)$_3$). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 155.8, 155.4, 138.3, 128.3, 127.5, 127.0, 123.8, 79.6, 59.1, 50.4 & 50.0, 46.2, 36.6, 28.5, 26.1, 25.7, 16.2. **IR (film):** ν = 3432, 2975, 2925, 1692, 1451, 1413, 1367, 1303, 1244, 1159, 1121, 1007, 885, 737. **MS (MALDI) *m/z*:** 343.2 [21%, $(MNa)^+$]. **HRMS (MALDI) *m/z*:** Calc. for $[MNa]^+$: 342.2040. Found: 342.2035. **Anal. Calcd. for $C_{19}H_{29}NO_3$:** C, 71.44; H, 9.15; N, 4.38; Found: C, 71.52, H, 8.92, N, 4.43.

6-Hydroxy-4-methylhex-4-enoic acid ethyl ester (2g)



2g

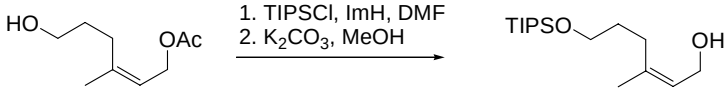
2-(*tert*-Butyldimethylsilyloxy)acetaldehyde was prepared following a literature procedure.[5] Isopropenylmagnesiumbromide (0.5 M in THF, 40 mL, 20 mmol, 1.17 equiv.) was added to a solution of the aldehyde (2.98 mmol, 17.1 mmol, 1 equiv.) in THF (50 mL) at –40 °C. The solution was warmed to RT and saturated aqueous ammonium chloride was added. The mixture was extracted with MTBE. The organic phase was washed with brine, dried over $MgSO_4$ and the solvents were removed under reduced pressure to give 1-(*tert*-butyldimethylsilyloxy)-3-methylbut-3-en-2-ol (1.477 g, 6.84 mmol, 40% yield) as a colorless oil. The alcohol was then refluxed in triethyl orthoacetate (5.54 g, 34 mmol, 5 equiv.) in the presence of propionic acid (0.50 g, 6.8 mmol, 1 equiv.) for 24 h to give the corresponding (*E*)-configured γ,δ-unsaturated ester (0.606 g, 2.1 mmol, 31% yield) after purification by column chromatography (pentane / Et_2O 16:1).

$C_{15}H_{30}O_3Si$, MW: 286.48 g/mol. **^{1}H-NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 5.29 (*t*, *J* = 5.7, 1 H, C=C*H*), 4.15 (*d*, *J* = 6.3, 2 H, CH_2OTBS), 4.12 (*q*, *J* = 7.2, 2 H, OCH_2CH$_3$), 2.42-2.38 & 2.31-2.26 (*m*, 2 H each, CH_2CH_2CO$_2$Et), 1.61 (*s*, 3 H, C=C-CH_3), 1.22 (*t*, *J* = 7.2, 3 H, OCH$_2$CH_3), 0.97 (*s*, 9 H, SiC(CH_3)$_3$), 0.03 (*s*, 6 H, Si(CH$_3$)$_2$. **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 172.9, 134.9, 124.9, 60.2, 60.1, 34.3, 32.8, 26.0, 18.4, 16.3, 14.3, –4.9. **IR (film):** ν = 2956, 2930, 2857, 2362, 1739, 1477, 1446, 1371, 1292, 1255, 1158, 1111, 1068. **MS (EI) *m/z*:** 229.1 [1.7 %, $(M\text{-}C_4H_9)^+$]. **HRMS (EI) *m/z*:** Calc. for $[M\text{-}C_4H_9]^+$: 229.1255. Found: 229.1254. **Anal. Calcd. for $C_{15}H_{30}O_3Si$:** C, 62.89; H, 10.55; Found: C, 62.62, H, 10.45.

The ester was subsequently treated with ammonium fluoride – hydrogen fluoride (0.240 mg, 4.2 mmol, 2 equiv.) in ethanol (15 mL) overnight to give **2g** after purification by column chromatography (pentane / EtOAc 9:1) as colorless oil (0.223 g, 1.26 mmol, 60%). Due to its low stability, only the preceding TBS-protected allylic alcohol was obtained in analytically pure form.

$C_9H_{16}O_3$, MW: 177.22 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 5.42 (*t*, *J* = 5.7, 1 H, C=C*H*), 4.14 (*m*, 2+2 H, CH_2OH & OCH_2CH$_3$), 2.45-2.30 (*m*, 4 H, CH_2CH_2), 1.69 (*s*, 3 H, C=C-CH_3), 1.25 (*t*, *J* = 7.2, 3 H, OCH$_2$CH_3). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 173.1, 137.2, 124.1, 60.2, 58.9, 34.2, 23.5, 16.0, 14.0. **IR (film):** ν = 3422, 2982, 2937, 1732, 1671, 1447, 1373, 1298, 1263, 1159, 1029. **MS (EI) *m/z*:** 81.0 [100 %], 172.1 [0.25 %, M^+]. **HRMS (EI) *m/z*:** Calc. for $[M]^+$: 172.1094. Found: 172.1094.

(*Z*)-3-Methyl-6-triisopropylsilanyloxyhex-2-enol (2h)

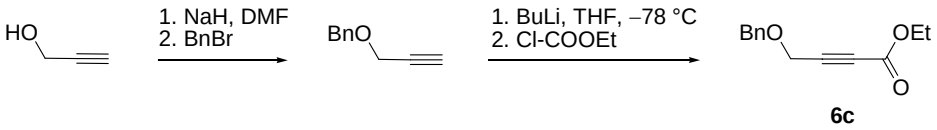


2h

Alcohol **2h** was prepared in analogy to **2d**, but with nerylacetate instead of geranylacetate as starting point.[6] Yield starting from (*Z*)-6-hydroxy-3-methylhex-2-enyl acetate: 92% (two steps, 1 mmol scale).

$C_{16}H_{34}O_2Si$, MW: 286.53 g/mol. **^{1}H-NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 5.51 (*t*, *J* = 6.9, 1 H, C=C*H*), 4.10 (*t*, *J* = 7.2, 2 H, CH_2OH), 3.67 (*t*, *J* = 6.0, 2 H, CH$_2$CH$_2$CH_2OTIPS), 2.21 (*t*, *J* = 7.2, 2 H, CH_2CH$_2$CH$_2$OTIPS), 1.73-1.60 (*m*, 2 + 3 H, C=C-CH_3 & CH$_2$CH_2CH$_2$OTIPS), 1.10-1.00 (*m*, 3 + 18 + 1 H, Si(C*H*(CH_3)$_2$)$_3$ & O*H*). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 139.4, 124.8, 62.1, 58.4, 30.8, 27.6, 23.1, 17.8, 11.8. **IR (film):** ν = 3339, 2944, 2867, 1464, 1381, 1247, 1109, 1069, 1013, 882. **MS (EI) *m/z*:** 95.0 [100%], 243.1 [1.5%, $(M-C_3H_7)^+$]. **HRMS (EI) *m/z*:** Calc. for $[M-C_3H_7]^+$: 243.1775. Found: 243.1775. **Anal. Calcd. for $C_{16}H_{34}O_2Si$:** C, 67.07; H, 11.96; Found: C, 66.82, H, 11.76.

4-Benzyloxybut-2-ynoic acid ethyl ester (6c)



6c

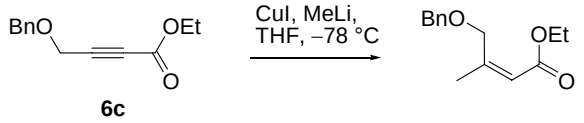
Propargylic alcohol (3.360 g, 3.48 mL, 60 mmol, 1 equiv.) was added in portions to a suspension of NaH (60% in mineral oil, 2.40 g, 60 mmol, 1 equiv.) in DMF (100 mL) at 0 °C. After 30 min at RT, the now homogeneous solution was cooled again to 0 °C and benzyl bromide (10.26 g, 7.20 mL, 60 mmol, 1 equiv.) was added. The solution was stirred for 30 min at RT, then ammonia (in water or methanol, 20 mL of a saturated solution) was added and the mixture was stirred at RT overnight. Water was added and the mixture was extracted three times with MTBE. The combined organic phases were washed three times with water and once with 1 N aqueous HCl and were then dried over $MgSO_4$. The solvent was removed *in vacuo* to give benzyl propargylether as slightly yellowish oil (6.570 g, 45 mmol, 75%) after filtration over silica with pentane / Et_2O 1:1. [**^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.37-7.26 (*m*, 5 H, Ar-*H*), 4.62 (*s*, 2 H, PhCH_2O), 4.18 (*s*, 2 H, BnOCH_2), 2.47 (*s*, 1 H, C*H*).]

Benzyl propargylether (3.30 g, 22.6 mmol, 1.0 equiv.) was dissolved in THF (30 mL) and cooled to −78 °C. *n*BuLi (1.6 M in hexanes, 15.0 mL, 24.0 mmol, 1.06 equiv.) was added and the solution was stirred for 30 min followed by addition of ethylchloroformate (2.72 g, 2.40 mL, 25 mmol, 1.1 equiv.). After five minutes, the reaction mixture was allowed to warm to RT and then quenched by addition of saturated aqueous ammonium chloride. After addition of water the aqueous phase was extracted twice with MTBE, the combined organic phases were dried over $MgSO_4$ and the solvent was removed under reduced pressure. Column chromatography (pentane / diethylether 16:1 → pentane / diethylether 1:1) gave **6c** as colorless to slightly yellow oil (3.560 g, 16.3 mmol, 72%)

which contained an unidentified impurity. Alternatively, **6c** can be purified by distillation (Kugelrohr distillation, 0.1 mbar, 170 °C).

$C_{13}H_{14}O_3$, MW: 218.25 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.36-7.26 (*m*, 5 H, Ar-*H*), 4.62 (*s*, 2 H, $C_6H_5CH_2O$), 4.29 (*s*, 2 H, $OCH_2CCCOOEt$), 4.24 (*q*, *J* = 7.2, 2 H, OCH_2CH_3), 1.32 (*t*, *J* = 7.2, 3 H, OCH_2CH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 152.9, 136.7, 128.6, 128.4, 128.2, 128.0, 128.0, 68.1, 78.3, 72.0, 62.1, 56.8, 14.1. **IR (film):** ν = 2983, 2859, 2235, 1715, 1455, 1366, 1251, 1090, 1056, 749, 698. **MS (EI)**: no unambiguously assignable peak was found. **Anal. Calcd. for $C_{13}H_{14}O_3$:** C, 71.54; H, 6.47; Found: C, 71.52, H, 6.70.

(*Z*)-4-Benzyloxy-3-methylbut-2-enoic acid ethyl ester

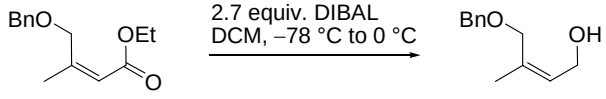


CuI (1.140 g, 6.0 mmol, 1.1 equiv.) was suspended in THF (16 mL) and cooled to ca. –40 °C. To the suspension was added MeLi (1.6 M in diethylether, 3.50 mL, 5.5 mmol, 1 equiv.). The mixture was stirred at –40 °C for 30 min and was then cooled to –78 °C. A solution of 4-benzyloxybut-2-ynoic acid ethyl ester **6c** (1.200 g, 5.5 mmol, 1 equiv.) in THF (4 mL) was added in one portion and the resulting suspension was stirred for 4 h at –78 °C. The reaction was quenched by addition of methanol (technical grade, ca. 2 mL), and the cooling bath was subsequently removed. Saturated aqueous ammonium chloride (ca. 10 mL) was added. The mixture was extracted three times with MTBE, and the combined organic phases were dried over $MgSO_4$. The solvent was removed under reduced pressure and the residue was purified by column chromatography (pentane / Et_2O 16:1) to

give (*Z*)-4-benzyloxy-3-methylbut-2-enoic acid ethyl ester as colorless oil (1.222 g, 5.22 mmol, 95%). The (*E*)-isomer could not be detected by ^{1}H-NMR.

$C_{14}H_{18}O_3$, MW: 234.29 g/mol**. ^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 7.36-7.28 (*m*, 5 H, Ar-*H*), 5.77 (*t, J* = 1.5, 1H, C=C*H*), 4.68 (*s*, 2 H, CH_2Ph), 4.52 (*s*, 2 H, CH_2OBn), 4.14 (q, *J* = 7.2, 2 H, OCH_2CH$_3$), 2.02 (*s*, 3 H, C=C-CH_3), 1.27 (*t, J* = 6.9, 3 H, OCH$_2$CH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 165.7, 156.8, 138.1, 128.2, 127.5, 117.2, 72.7, 69.3, 59.8, 21.8, 14.4. **IR (film):** ν = 3032, 2981, 2860, 1713, 1649, 1497, 1445, 1375, 1336, 1223, 1150, 1096, 1065, 1029, 852. **MS (EI) *m/z*:** 91.0 [100%], 235.1 [0.1%, MH$^+$]. **HRMS (EI) *m/z*:** Calc. for [MH$^+$]: 235.1329. Found: 235.1329. **Anal. Calcd. for $C_{14}H_{18}O_3$:** C, 71.77; H, 7.75. Found: C, 71.81, H, 7.90.

(*Z*)-4-Benzyloxy-3-methylbut-2-enol (2i)

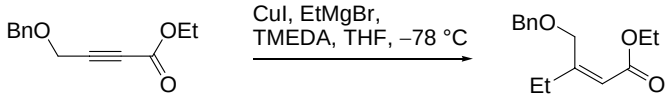


2i

(*Z*)-4-Benzyloxy-3-methylbut-2-enoic acid ethyl ester (1.220 g, 5.2 mmol, 1 equiv.) was dissolved in DCM (10 mL) and cooled to −78 °C. DIBAL (1 M in DCM or hexanes, 14.0 mL, 14.0 mmol, 2.7 equiv.) was added in portions within 1 min. After 30 min stirring at −78 °C, the solution was slowly warmed to 0 °C and stirred for 1 h, then cooled again to −78 °C and quenched by dropwise addition of 1 N HCl. After warming to RT, additional 1 N aqueous HCl and DCM were added until complete dissolution of the precipitate. The aqueous phase was extracted twice with DCM and the combined organic phases were dried over $MgSO_4$. Subsequent solvent removal under reduced pressure gave **2i** (0.800 g, 4.16 mmol, 80%) as colorless oil which did not require further purification.

$C_{12}H_{16}O_2$, MW: 192.26 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.39-7.26 (*m*, 5 H, Ar-*H*), 5.63 (*t, J* = 6.9, 1H, C=C*H*), 4.48 (*s*, 2 H, CH_2Ph), 4.09 (*t, J* = 6.0, CH_2OH), 4.02 (*s*, 2 H, CH_2OBn), 2.36 (*app d, J* = 4.5, 1H, CH_2O*H*), 1.83 (*d*, *J* = 1.2, 3 H, C=C-CH_3). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 137.9, 135.8, 128.4, 128.3, 127.7, 72.2, 68.6, 58.5, 22.1. **IR (film):** ν = 3390, 2064, 3031, 2971, 2917, 2862, 1671, 1606, 1496, 1453, 1366, 1311, 1248, 1205, 1073, 1027, 1000, 738, 698. **MS (EI) *m/z*:** 91.0 [100%], 174.1 [1.5%, $(M-H_2O)^+$]. **HRMS (EI) *m/z*:** Calc. for [$(M-H_2O)^+$]: 174.1039. Found: 174.1040. **Anal. Calcd. for $C_{12}H_{16}O_2$:** C, 74.97; H, 8.39. Found: C, 74.70, H, 8.22.

(*Z*)-4-Benzyloxy-3-ethylbut-2-enoic acid ethyl ester



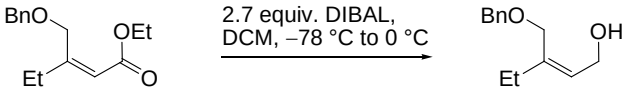
6c

To a suspension of CuI (0.950 g, 5.0 mmol, 1.25 equiv.) in THF (30 mL) at –40 °C ethylmagnesiumbromide (3 M in diethylether, 1.34 mL, 4 mmol, 1 equiv.) and TMEDA (1.74 g, 2.26 mL, 15.0 mmol, 3.75 equiv.) were added successively. The grey suspension which may turn to purple was stirred at –40 °C for 30 min and then cooled to –78 °C. 4-Benzyloxybut-2-ynoic acid ethyl ester **6c** (0.872 g, 4.0 mmol, 1.0 equiv.) was added in one portion and the resulting suspension was stirred for 2 h at –78 °C. The reaction was quenched by dropwise addition of methanol (technical grade, ca. 5 mL) and the cooling bath was subsequently removed. Saturated aqueous ammonium sulfate (ca. 5 mL) was added and the suspension was allowed to warm to RT. Then aqueous ammonia (ca. 20%) was added until complete dissolution of all solids, followed by extraction with MTBE (2 x) and washing of the combined organic phases with additional aqueous

ammonia and brine. The solution was dried over $MgSO_4$ and the volatiles were removed *in vacuo*. Purification by column chromatography (pentane / Et_2O 16:1) afforded (*Z*)-4-benzyloxy-3-ethylbut-2-enoic acid ethyl ester (492 mg, 1.98 mmol, 49%) as a colorless oil. The (*E*)/(*Z*)-ratio of 4:96 was determined by ^{1}H-NMR (characteristic signals: *E*: 5.95 ppm, *Z*: 5.74 ppm, C=C*H*).[7]

$C_{15}H_{20}O_3$, MW: 248.32 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.36-7.26 (*m*, 5 H, Ar-*H*), 5.75 (*t, J* = 1.5, 1 H, C=C*H*), 4.69 (*s*, 2 H, CH_2Ph), 4.52 (*s*, 2 H, CH_2OBn), 4.14 (q, *J* = 7.2, 2 H, OCH_2CH$_3$), 2.38 (*q, J* = 7.5, 2 H, C=C-CH_2CH$_3$), 1.25 (*t, J* = 6.9, 3 H, OCH$_2$CH_3), 1.10 (*t, J* = 7.5, 3 H, C=C-CH$_2$CH_3). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 166.1, 161.5, 128.2, 128.2, 127.5, 127.4, 115.6, 72.6, 68.2, 59.7, 27.5, 14.1, 12.0. **IR (film):** ν = 2975, 2934, 2878, 2363, 1713, 1647, 1497, 1454, 1377, 1309, 1212, 1150, 1074, 1036, 868, 736. **MS (EI) *m/z*:** 91.0 [100%], 157.0 [25%, (M-C_7H_7)$^+$]. **HRMS (EI) *m/z*:** Calc. for [M-C_7H_7)$^+$]: 157.0860. Found: 157.0860. **Anal. Calcd. for $C_{15}H_{20}O_3$:** C, 72.55; H, 8.12. Found: C, 72.47, H, 8.10.

(*Z*)-4-Benzyloxy-3-ethylbut-2-enol (2j)



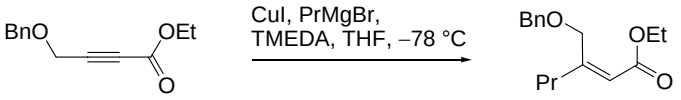
2j

(*Z*)-4-Benzyloxy-3-ethylbut-2-enoic acid ethyl ester (0.434 g, 1.75 mmol, 1.0 equiv) was dissolved in DCM (10 mL) and cooled to –78 °C. DIBAL (1.1 M in cyclohexane, 4.3 mL, 4.7 mmol, 2.7 equiv.) was added in portions within 1 min. After 30 min stirring at –78 °C, the solution was slowly warmed to 0 °C and stirred for an additional hour, then cooled again to –78 °C and quenched by dropwise addition of 1 N aqueous HCl. After warming to RT, additional 1 N HCl and DCM were added until complete dissolution of the precipitate. The aqueous phase was extracted twice with

DCM and the combined organic phases were dried over $MgSO_4$. Subsequent solvent removal under reduced pressure gave **2j** (0.299 g, 1.45 mmol, 83%) as colorless oil which did not require further purification.

$C_{13}H_{18}O_2$, MW: 206.28 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.36-7.26 (*m*, 5 H, Ar-*H*), 5.65 (*t, J* = 6.6, 1H, C=C*H*), 4.48 (*s*, 2 H, CH_2Ph), 4.13 (d, *J* = 6.9, CH_2OH), 4.02 (*s*, 2 H, CH_2OBn), 2.40 (*bs*, 1H, CH_2O*H*), 2.17 (*q, J* = 7.5, 2 H, C=C-C$H_2$$CH_3$), 1.04 (*t*, *J* = 7.5, 3 H, C=C-CH_2CH_3). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 141.1, 137.8, 128.3, 127.7, 127.6, 126.9, 72.3, 67.6, 58.5, 28.3, 12.2. **IR (film):** ν = 3391, 3064, 3031, 2965, 2932, 2874, 1953, 1870, 1811, 1668, 1607, 1586, 1496, 1454, 1366, 1310, 1246, 1205, 1174, 1071, 1012. **MS (EI) *m/z*:** 91.0 [100%], 188.1 [1.5%, $(M-H_2O)^+$]. **HRMS (EI) *m/z*:** Calc. for [$(M-H_2O)^+$]: 188.1196. Found: 188.1196. **Anal. Calcd. for $C_{13}H_{18}O_2$:** C, 75.69; H, 8.79. Found: C, 75.41, H, 8.69.

(*Z*)-3-Benzyloxymethylhex-2-enoic acid ethyl ester



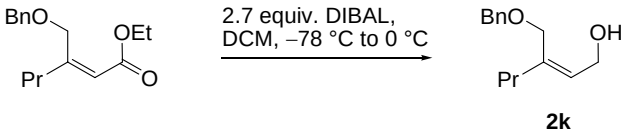
6c

To a suspension of CuI (0.760 g, 4 mmol, 1.33 equiv.) in THF (10 mL) at –40 °C propylmagnesiumbromide (2 M in diethylether, 1.65 mL, 3.3 mmol, 1.1 equiv.) and TMEDA (1.39 g, 1.80 mL, 12.0 mmol, 4 equiv.) were added successively. The grey suspension that may turn to purple was stirred at –40 °C for 30 min and then cooled to –78 °C. 4-Benzyloxy-but-2-ynoic acid ethyl ester (**6c**, 0.654 g, 3.0 mmol, 1.0 equiv.) was added in one portion and the resulting suspension was stirred for 2 h at –78 °C. The reaction was quenched by dropwise addition of methanol (technical grade, ca. 5 mL) and the cooling bath was subsequently removed. Saturated aqueous

ammonium sulfate (ca. 5 mL) was added and the suspension was allowed to warm to RT. Then aqueous ammonia (ca. 20%) was added until complete dissolution of all solids, followed by extraction with MTBE (2 x) and washing of the combined organic phases with additional aqueous ammonia and brine. The solution was dried over $MgSO_4$ and the volatiles were removed *in vacuo*. Purification by column chromatography (pentane / Et_2O 16:1) afforded (*Z*)-3-benzyloxymethylhex-2-enoic acid ethyl ester (592 mg, 2.25 mmol, 75%) as a colorless oil. The (*E*)/(*Z*)-ratio of 0.5:99.5 was determined by ^{1}H-NMR (characteristic signals: *E*: 6.02 ppm, *Z*: 5.75 ppm, C=C*H*).[7]

$C_{16}H_{22}O_3$, MW: 262.34 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.37-7.25 (*m*, 5 H, Ar-*H*), 5.75 (*t, J* = 2.5, 1 H, C=C*H*), 4.68 (s, 2 H, CH_2Ph), 4.52 (s, 2 H, CH_2OBn), 4.14 (q, *J* = 7.2, 2 H, OCH_2CH$_3$), 2.33 (t, *J* = 7.5, 2 H, C=C-CH_2CH$_2$CH$_3$), 1.51 (*dt, J* = 7.5, *J* = 7.2, 2H, C=C-CH$_2$CH_2CH$_3$), 1.25 (*t, J* = 6.9, 3 H, OCH$_2$CH_3), 0.97 (*t, J* = 7.2, 3 H, C=C-CH$_2$CH_3). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 166.0, 159.9, 138.2, 128.2, 127.5, 127.5, 116.7, 72.7, 68.1, 59.9, 36.9, 21.1, 14.4, 14.0. **IR (film):** ν = 2961, 1934, 2873, 1643, 1497, 1454, 1377, 1255, 1209, 1151, 1096, 1037, 881. **MS (EI) *m/z*:** 91.0 [100%], 171.1 [25%, $(M-C_7H_7)^+$]. **HRMS (EI) *m/z*:** Calc. for [$(M-C_7H_7)^+$]: 171.1016. Found: 171.1015. **Anal. Calcd. for $C_{16}H_{22}O_3$:** C, 73.25; H, 8.45. Found: C, 72.98, H, 8.41.

(*Z*)-3-Benzyloxymethylhex-2-enol (2k)



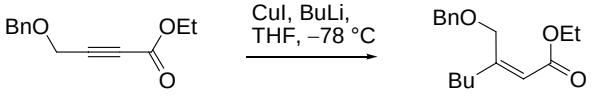
2k

(*Z*)-3-Benzyloxymethylhex-2-enoic acid ethyl ester (0.300 g, 1.14 mmol, 1.0 equiv) was dissolved in DCM (5 mL) and cooled to −78 °C. DIBAL (1.1 M in cyclohexane, 2.8 mL, 3.0 mmol, 2.7

equiv.) was added in portions within 1 min. After 30 min stirring at −78 °C, the solution was slowly warmed to 0 °C and stirred for an additional hour, then cooled again to −78 °C and quenched by dropwise addition of 1 N aqueous HCl. After warming to RT, additional 1 N HCl and DCM were added until complete dissolution of the precipitate. The aqueous phase was extracted twice with DCM and the combined organic phases were dried over $MgSO_4$. Subsequent solvent removal under reduced pressure gave **2k** (0.249 g, 1.13 mmol, 99%) as colorless oil which did not require further purification.

$C_{14}H_{20}O_2$, MW: 220.31 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.38-7.25 (*m*, 5 H, Ar-*H*), 5.64 (*t, J* = 6.6, 1H, C=C*H*), 4.48 (*s*, 2 H, CH_2Ph), 4.11 (t, *J* = 6.3, CH_2OH), 4.01 (*s*, 2 H, CH_2OBn), 2.35 (*bs*, 1H, CH_2OH), 2.10 (*t, J* = 7.8, 2 H, C=C-C$H_2CH_2CH_3$), 1.52-1.29 (dt, *J* = 7.5, *J* = 7.2, 2 H, C=C-$CH_2CH_2CH_3$), 0.91 (*t*, *J* = 7.2, 3 H, C=C-$CH_2CH_2CH_3$). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 139.5, 137.8, 128.3, 128.1, 127.7, 127.6, 72.4, 67.6, 58.6, 37.9, 21.1, 13.9. **IR (film):** ν = 3372, 2950, 2930, 2861, 1449, 1371, 1067, 1008. **MS (EI) *m/z*:** 91.0 [100%], 202.1 [1.3%, $(M-H_2O)^+$]. **HRMS (EI) *m/z*:** Calc. for [$(M-H_2O)^+$]: 202.1353. Found: 202.1351. **Anal. Calcd. for $C_{14}H_{20}O_2$:** C, 76.33; H, 9.15. Found: C, 76.08, H, 9.06.

(*Z*)-3-Benzyloxymethylhept-2-enoic acid ethyl ester

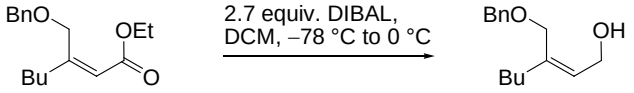


To a suspension of CuI (0.760 g, 4 mmol, 1.33 equiv.) in THF (10 mL) at −40 °C *n*-BuLi (1.6 M in hexane, 2.06 mL, 3.3 mmol, 1.1 equiv.) was added. The grey suspension that may turn to purple was stirred at −40 °C for 30 min and then cooled to −78 °C. 4-Benzyloxy-but-2-ynoic acid ethyl ester

(**6c**, 0.654 g, 3.0 mmol, 1.0 equiv.) was added in one portion and the resulting suspension stirred for 2 h at −78 °C. The reaction was quenched by dropwise addition of methanol (technical grade, ca. 5 mL) and the cooling bath was subsequently removed. Saturated aqueous ammonium sulfate (ca. 5 mL) was added and the suspension was allowed to warm to RT. Then aqueous ammonia (ca. 20%) was added until complete dissolution of all solids, followed by extraction with MTBE (2 x) and washing of the combined organic phases with additional aqueous ammonia and brine. The solution was dried over $MgSO_4$ and the volatiles were removed *in vacuo*. Purification by column chromatography (pentane / Et_2O 16:1, R_f = 0.19) afforded (*Z*)-3-benzyloxymethylhept-2-enoic acid ethyl ester (608 mg, 2.20 mmol, 73%) as a colorless oil. An unidentified impurity could only be removed after the subsequent reduction step. The (*Z*)/(*E*)-ratio of > 100:1 was determined by ^{1}H-NMR (characteristic signals: *E*: 5.99 ppm, Z: 5.76 ppm, C=C*H*).[7]

$C_{17}H_{24}O_3$, MW: 276.37 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.36-7.16 (*m*, 5 H, Ar-*H*), 5.76 (*t*, *J* = 1.5, 1H, C=C*H*), 4.68 (*s*, 2H, OCH_2Ph), 4.52 (*s*, 2H, CH_2OBn), 4.15 (*q*, *J* = 7.2, 2 H, OCH_2H$_3$), 2.35 (*t*, *J* = 8.1, 2 H, C=CH-CH_2CH$_2$CH$_2$CH$_3$), 1.60-1.29 (*m*, 4 H, C=CH-CH$_2$CH_2CH_2CH$_3$), 1.29 (*t*, *J* = 7.2, 3H, OCH$_2$CH_3), 0.92 (*t*, *J* = 7.2, 3H, C=CH-CH$_2$CH$_2$CH$_2$CH_3). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 166.0, 160.3, 138.2, 128.2, 127.5, 127.4, 116.5, 72.6, 68.0, 59.7, 34.4, 29.8, 22.3, 14.1, 13.8. **IR (film):** ν =2958, 2932, 2862, 1713, 1642, 1497, 1454, 1377, 1261, 1206, 1151, 1096, 1038, 735, 697. **MS (EI) *m/z*:** 91.0 [100%], 185.1 [25%, $(M-C_7H_7)^+$]. **HRMS (EI) *m/z*:** Calc. for $[(M-C_7H_7)^+]$: 185.1173. Found: 185.1173.

(*Z*)-3-Benzyloxymethylhept-2-enol (2l)

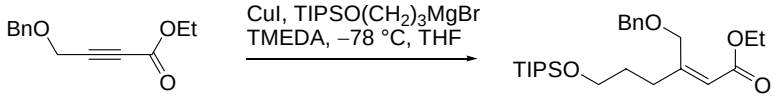


2l

(*Z*)-3-Benzyloxymethylhept-2-enoic acid ethyl ester (0.278 g, 1.0 mmol, 1.0 equiv) was dissolved in DCM (5 mL) and cooled to −78 °C. DIBAL (1.1 M cyclohexane, 2.5 mL, 2.75 mmol, 2.75 equiv.) was added in portions within 1 min. After 30 min stirring at −78 °C, the solution was slowly warmed to 0 °C and stirred for an additional hour then cooled again to −78 °C and quenched by dropwise addition of 1 N aqueous HCl. After warming to RT, additional 1 N HCl and DCM were added until complete dissolution of the precipitate. The aqueous phase was extracted twice with DCM and the combined organic phases were dried over $MgSO_4$. After solvent removal under reduced pressure the crude product was purified by column chromatography (pentane / Et_2O 1:1) furnishing **2l** (0.224 g, 0.95 mmol, 95%) as a colorless oil.

$C_{15}H_{22}O_2$, MW: 234.33 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.38-7.25 (*m*, 5 H, Ar-*H*), 5.65 (*t, J* = 6.9, 1H, C=C*H*), 4.49 (*s*, 2 H, CH_2Ph), 4.11 (t, *J* = 6.3, CH_2OH), 4.01 (*s*, 2 H, CH_2OBn), 2.25 (*bs*, 1H, CH_2OH), 2.12 (*t, J* = 6.9, 2 H, C=C-C$H_2CH_2CH_2CH_3$), 1.44-1.28 (*m*, 4 H, C=C-$CH_2CH_2CH_2CH_3$), 0.91 (*t*, *J* = 7.2, 3 H, C=C-$CH_2CH_2CH_2CH_3$). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 139.9, 137.8, 128.3, 127.9, 127.7, 127.6, 72.4, 67.6, 58.6, 35.5, 30.1, 22.5, 14.0. **IR (film):** ν = 3368, 3031, 2956, 2929, 2860, 2363, 1664, 1496, 1454, 1363, 1245, 1205, 1071, 1010. **MS (EI) *m/z*:** 91.0 [100%], 216.1 [1.2%, $(M-H_2O)^+$]. **HRMS (EI) *m/z*:** Calc. for [$(M-H_2O)^+$]: 216.1509. Found: 216.1507. **Anal. Calcd. for $C_{15}H_{22}O_2$:** C, 76.88; H, 9.46. Found: C, 76.65, H, 9.49.

(*Z*)-3-Benzyloxymethyl-6-triisopropylsilanyloxyhex-2-enoic acid ethyl ester

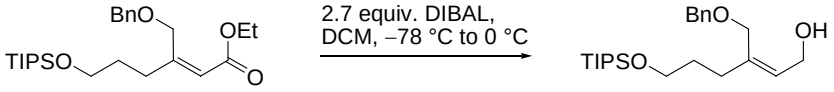


6c

3-(Triisopropylsilyloxy)propylbromide was prepared following a literature procedure from 3-bromopropanol.[8] Magnesium turnings were activated by grinding in a mortar inside a glovebox, the metal (0.210 g, 5 mmol, ca. 1.2 equiv.) was suspended in THF (5 mL), followed by addition of a few drops of a solution of iodine in MTBE. Then 3-(triisopropylsilyloxy)propylbromide (1.220 g, 4.1 mmol, 1 equiv.) was added at RT in one portion. The flask was placed in an ultrasonic bath until the reaction had started, visible by the disappearance of the yellow color of iodine. The temperature was kept below ca. 45 °C by cooling in a water bath. This Grignard solution (1 equiv., 5 mL, 4.1 mmol) was added to a suspension of CuI (0.760 g, 4 mmol, 1.33 equiv.) in THF (10 mL) at –40 °C . The grey suspension that may turn to purple was stirred at –40 °C for 30 min and then cooled to –78 °C. 4-Benzyloxy-but-2-ynoic acid ethyl ester (**6c**, 0.736 g, 3.5 mmol, 1.0 equiv.) was added in one portion and the resulting suspension stirred for 2 h at –78 °C. The reaction was quenched by dropwise addition of methanol (technical grade, ca. 5 mL) and the cooling bath was subsequently removed. After 0 °C was reached, saturated aqueous ammonium sulfate (ca. 5 mL) was added and the suspension was allowed to warm to RT. Then aqueous ammonia (ca. 20%) was added until complete dissolution of all solids, followed by extraction with MTBE (2 x) and washing of the combined organic phases with additional aqueous ammonia and brine. The solution was dried over $MgSO_4$ and the volatiles were removed *in vacuo*. Purification by column chromatography (pentane / Et_2O 16:1) afforded (*Z*)-3-benzyloxymethyl-6-triisopropylsilanyloxyhex-2-enoic acid ethyl ester (1026 mg, 2.36 mmol, 67%) as a colorless oil. The (*Z*)/(*E*)-ratio of > 100:1 was determined by ^{1}H-NMR (characteristic signals: *E*: expected ca. 6.00 ppm, *Z*: 5.77 ppm, C=C*H*).

$C_{25}H_{42}O_4Si$, MW: 434.68 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.35-7.26 (*m*, 5 H, Ar-*H*), 5.79 (*t*, *J* = 1.2, 1H, C=C*H*), 4.68 (*s*, 2H, OCH_2Ph), 4.52 (*s*, 2H, CH_2OBn), 4.15 (*q*, *J* = 7.2, 2 H, OCH_2H$_3$), 3.72 (*t*, *J* = 6.3, 2 H, CH_2CH$_2$CH$_2$OTIPS), 2.46 (*t*, *J* = 6.6, 2 H, CH$_2$CH$_2$CH_2OTIPS), 1.78-1.72 (*m*, 2 H, CH$_2$CH_2CH$_2$OTIPS), 1.29 (*t*, *J* = 7.2, 3H, OCH$_2$CH_3), 1.11-1.02 (*m*, 21 H, Si(C*H*(CH_3)$_2$)$_3$). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 166.0, 160.0, 138.2, 128.2, 127.5, 127.4, 116.6, 72.6, 68.1, 62.6, 59.7, 31.2, 31.0, 17.9, 14.1, 11.8. **IR (film):** ν = 2943, 2892, 2866, 2727, 1746, 1714, 1643, 1497, 1463, 1378, 1257, 1212, 1148, 1107, 1037, 882. **MS (EI) *m/z*:** 91.0 [100%], 391.2 [17.6%, (M-C_3H_7)$^+$]. **HRMS (EI) *m/z*:** Calc. for [(M-C_3H_7)$^+$]. 391.2300. Found: 391.2302. **Anal. Calcd. for $C_{25}H_{42}O_4Si$:** C, 69.08; H, 9.74. Found: C, 68.94, H, 9.63.

(*Z*)-3-Benzyloxymethyl-6-triisopropylsilanyloxyhex-2-enol (2m)

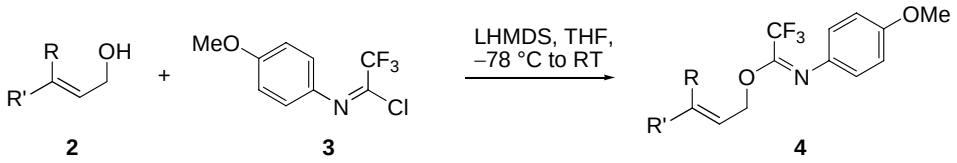


2m

(*Z*)-3-Benzyloxymethyl-6-triisopropylsilanyloxyhex-2-enoic acid ethyl ester (0.635 g, 1.46 mmol, 1.0 equiv) was dissolved in DCM (5 mL) and cooled to −78 °C. DIBAL (1.1 M in cyclohexane, 3.6 mL, 3.9 mmol, 2.7 equiv.) was added in portions within 1 min. After 30 min stirring at −78 °C, the solution was slowly warmed to 0 °C and stirred for an additional hour then cooled again to −78 °C and quenched by dropwise addition of 1 N aqueous HCl. After warming to RT, additional 1 N HCl and DCM were added until complete dissolution of the precipitate. The aqueous phase was extracted twice with DCM and the combined organic phases were dried over $MgSO_4$. Subsequent solvent removal under reduced pressure gave **2m** (0.530 g, 1.35 mmol, 92%) as colorless oil which did not require further purification.

$C_{23}H_{40}O_3Si$, MW: 392.65 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.40-7.26 (*m*, 5 H, Ar-*H*), 5.68 (*t, J* = 6.9, 1 H, C=C*H*), 4.49 (*s*, 2 H, OCH_2Ph), 4.12 (*d, J* = 7.2, 2 H, CH_2OH), 4.03 (s, 2 H, CH_2OBn), 3.68 (*t, J* = 12.9, 2 H, $CH_2CH_2CH_2$OTIPS), 2.22 (*t, J* = 8.4, 2 H, C$H_2CH_2CH_2$OTIPS), 2.10 (*bs*, 1 H, CH_2OH), 1.73-1.63 (*m*, 2 H, $CH_2CH_2CH_2$OTIPS), 1.13-1.02 (*m*, 21 H, Si(C*H*(CH_3)$_2$)$_3$). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 139.7, 137.8, 128.3, 128.1, 127.7, 127.6, 71.5, 67.8, 62.9, 58.7, 32.2, 31.4, 18.1, 12.1. **IR (film):** ν = 3380, 2943, 2866, 2376, 1462, 1382, 1249, 1108, 013, 882. **MS (EI) *m/z*:** 91.0 [100%, $(C_7H_7)^+$], 241.1 [9%]. **Anal. Calcd. for $C_{23}H_{40}O_3Si$:** C, 70.36; H, 10.27. Found: C, 70.46, H, 10.39.

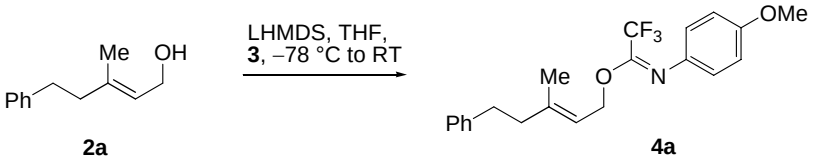
General procedure for the preparation of allylic N-(4-methoxyphenyl) trifluoroacetimidates 4 (GP1)



2 **3** **4**

To a solution of the corresponding allylic alcohol **2** in THF (2 mL/mmol) at −78 °C was added lithium hexamethyldisilazane[9] (LHMDS, 1.0 equiv.) as 1 M solution in THF, followed by 2,2,2-trifluoro-N-(4-methoxyphenyl)acetimidoyl chloride[10] **3** (1.0 equiv.). The solution was allowed to warm to RT and stirred for additional 2 hours before filtration over silica (with MTBE). The solvent was removed under reduced pressure, followed by HV to remove residual $HN(SiMe_3)_2$. The yellow residue was then purified by column chromatography (pentane/3% NEt_3). In the case of allylic alcohols bearing alcoholate sensitive functional groups, modified procedures which are given below were applied.

(*E*)-2,2,2-Trifluoro-N-(4-methoxyphenyl) acetimidic acid 3-methyl-5-phenyl-pent-2-enyl ester (4a)

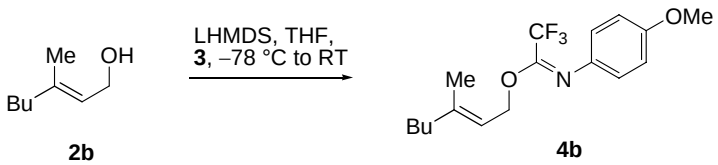


2a **4a**

According to GP1, imidate **4a** was obtained from allylic alcohol **2a** (430 mg, 2.48 mmol) as a colorless solid (917 mg, 2.43 mmol, 98%).

$C_{21}H_{22}F_3NO_2$, MW: 377.40 g/mol. **Mp.:** 28.5-29.5 °C. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.32-7.25 & 7.21- 7.18 (*m*, 5 H, C_6H_5), 6.87-6.83 & 6.82-6.74 (*m*, 4 H, C_6H_4OMe), 5.47 (*t, J* = 6.9, 1 H, C=C*H*), 4.76 (*d, J* = 6.9, 2 H, CH_2O), 3.79 (*s*, 3 H, OCH_3), 2.78 & 2.38 (*t, J* = 8.7, 2 H each, CH_2CH_2), 1.79 (*s*, 3 H, C=C-CH_3). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 156.0, 145.5 (q, $J_{C\text{-}C\text{-}F}$ = 32.7), 142.5, 141.5, 137.5, 128.3, 128.2, 125.9, 120.6, 117.9, 116.0 (q, $J_{C\text{-}F}$ = 285.4), 113.8, 64.7, 55.4, 41.4, 34.2, 16.8. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –65.5. **IR (film):** ν = 3389, 2958, 2912, 2823, 2359, 1698, 1507, 1314, 1243, 1205, 1140. **MS (EI) *m/z*:** 91.0 [100%], 377.1 [0.8%, M^+]. **HRMS (EI) *m/z*:** Calc. for [M^+]: 377.1597. Found: 377.1599. **Anal. Calcd. for $C_{21}H_{22}F_3NO_2$:** C, 66.83; H, 5.88; N, 3.71. Found: C, 66.95, H, 6.04, N, 3.67.

(*E*)-2,2,2-Trifluoro-N-(4-methoxyphenyl)acetimidic acid 3-methylhept-2-enyl ester (4b)

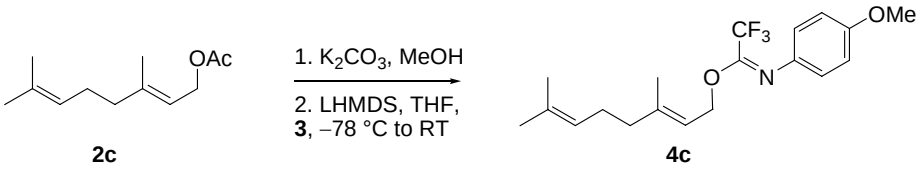


2b **4b**

According to GP1, imidate **4b** was obtained from allylic alcohol **2b** (256 mg, 2 mmol) as a colorless oil (440 mg, 1.33 mmol, 67%, single run, not optimized).

$C_{17}H_{22}F_3NO_2$, MW: 329.36 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 6.86-6.74 (*m*, 4 H, Ar-*H*), 5.45 (*t*, *J* = 6.9, 1 H, C=C*H*), 4.76 (*d*, *J* = 11.7, 2 H, C=CHC*H*$_2$O), 3.79 (s, 3 H, OCH_3), 2.07 (*t, J* = 7.2, C*H*$_2$CH$_2$CH$_2$CH$_3$), 1.72 (*s*, 3H, C=C-C*H*$_3$), 1.46-1.26 (m, 4 H, CH$_2$C*H*$_2$C*H*$_2$CH$_3$), 0.92 (*t, J* = 7.2, CH$_2$CH$_2$CH$_2$C*H*$_3$). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 156.0, 145.5 (q, $J_{C\text{-}C\text{-}F}$ = 32.7), 143.5, 137.5, 120.6, 117.9, 116.0 (q, $J_{C\text{-}F}$ = 285.4), 113.8, 64.8, 55.4, 39.3, 29.8, 22.3, 16.6, 14.0. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –65.0. **IR (film):** ν = 2933, 2362, 1702, 1611, 1508, 1443, 1382, 1347, 1315, 1290, 1243, 1205, 1140, 1106, 1038, 833. **MS (EI) *m/z*:** 329.1 [1.3%, M^+]. **HRMS (EI) *m/z*:** Calc. for [M^+]: 329.1597. Found: 329.1599. **Anal. Calcd. for $C_{17}H_{22}F_3NO_2$:** C, 61.99; H, 6.73; N, 4.25. Found: C, 61.84, H, 6.98, N, 4.15.

(*E*)-2,2,2-Trifluoro-N-(4-methoxyphenyl)acetimidic acid 3,7-dimethyloct-2,6-dienyl ester (4c)



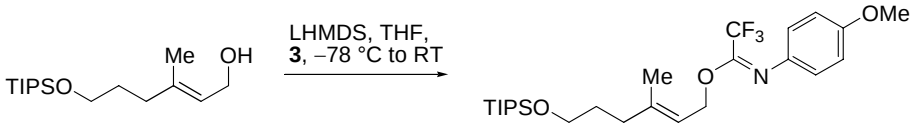
2c **4c**

Diastereomerically pure geraniol was prepared in quantitative yield from geranylacetate by treatment with a saturated K_2CO_3 solution in methanol (ca. 15 mL/mmol) overnight, followed by addition of water and extraction with MTBE. According to GP1, imidate **4c** was obtained from allylic alcohol **2c** (308 mg, 2 mmol) as a colorless oil (560 mg, 1.57 mmol, 78%).

$C_{19}H_{24}F_3NO_2$, MW: 355.39 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 6.85-6.73 (*m*, 4 H, arom-*H*), 5.45 (*t*, *J* = 7.2, 1H, C=C*H*CH$_2$O), 5.10 (*t*, *J* = 1.2, 1 H, Me$_2$C=C*H*), 4.76 (*d*, *J* = 6.6, 2 H,

C=CHCH_2O), 3.79 (*s*, 3 H, OCH_3), 2.16-2.03 (*m*, 4 H, CH_2CH_2), 1.73 (*s*, 3 H, OCH_2C=C-CH_3), 1.69 & 1.61 (s, 3 H each, C=C(CH_3)$_2$). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 156.0, 145.5 (q, J_{C-C-F} = 32.7), 143.0, 137.5, 131.8, 123.6, 120.6, 117,4, 116.0 (q, J_{C-F} = 285.0), 114.2, 113.8, 64.8, 55.4, 39.6, 26.3, 25.7, 17.7, 16.7. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –65.0. **IR (film):** ν = 2931, 1701, 1508, 1458, 1314, 1243, 1204, 1140, 1038, 833. **MS (EI) *m/z*:** 69.0 [85%], 219.0 [100%], 355.1 [0.7%, M^+]. **HRMS (EI) *m/z*:** Calc. for $[M]^+$: 3255.1754. Found: $[M]^+$: 355.1757. **Anal. Calcd. for** $C_{19}H_{24}F_3NO_2$**:** C, 64.21; H, 6.81; N, 3.94. Found: C, 64.07, H, 7.01, N, 3.89.

(*E*)-2,2,2-Trifluoro-N-(4-methoxyphenyl)acetimidic acid 3-methyl-6-triisopropylsilyloxyhex-2-enyl ester (4d)



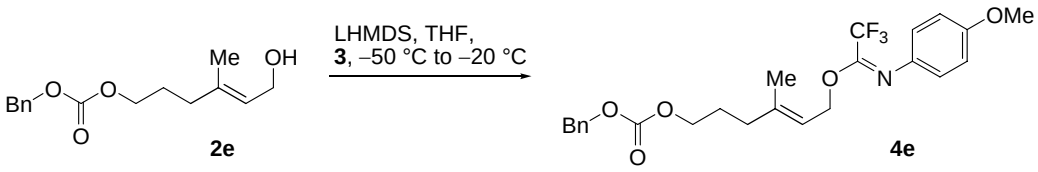
2d **4d**

According to GP1, imidate **4d** was obtained from allylic alcohol **2d** (290 mg, 1 mmol) as a colorless oil (394 mg, 0.80 mmol, 80%).

$C_{25}H_{40}F_3NO_3Si$, MW: 487.67 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 6.85-6.74 (*m*, 4 H, arom-*H*), 5.47 (*t*, *J* = 7.2, 1 H, C=C*H*), 4.76 (*d*, *J* = 6.6, 2 H, C=CHCH_2O), 3.79 (*s*, 3 H, OCH_3), 3.71 (*t*, *J* = 6.3, 2 H, TIPSOCH_2), 2.15 (*t*, *J* = 7.5, 2 H, C$H_2CH_2CH_2$OTIPS), 1.73 (*s*, 3H, C=C-CH_3), 1.73-1.64 (*m*, 2 H, $CH_2CH_2CH_2$OTIPS), 1.13-1.02 (*m*, 3 + 18 H, Si(C*H*(CH_3)$_2$)$_3$). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 156.1, 145.5 (q, J_{C-C-F} = 32.7), 143.1, 137.6, 120.6, 117.4, 116.0 (q, J_{C-F} = 285.4), 113.8, 64.5, 62.6, 55.2, 35.6, 30.8, 17.9, 16.5, 11.8. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –65.5. **IR (film):** ν = 3397, 1945, 2859, 1703, 1507, 1243, 1205, 1153, 1141. **MS (EI) *m/z*:** 95.0 [100%], 219.0 [22%], 444.2 [1.8%, $(M-C_3H_7)^+$], 487.2 [0.21%, M^+]. **HRMS (EI) *m/z*:** Calc. for [M-

$C_3H_7]^+$: 444.2176. Found: $[M\text{-}C_3H_7]^+$: 444.2176. **Anal. Calcd. for $C_{25}H_{40}F_3NO_3Si$:** C, 61.57; H, 8.27; N, 2.87. Found: C, 61.85, H, 8.18, N, 2.99.

(*E*)-2,2,2-Trifluoro-N-(4-methoxyphenyl)acetimidic acid 3-methyl-6-benzyloxycarbonyloxyhex-2-enyl ester (4e)

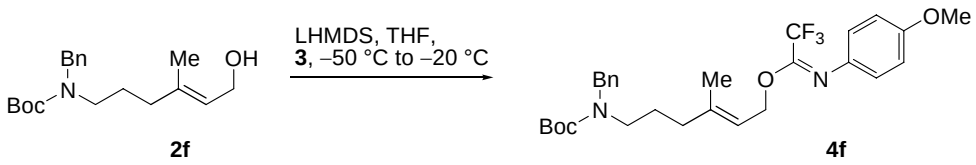


A solution of (*E*)-3-methyl-6-benzyloxycarbonyloxyhex-2-enol (**2e**, 0.163 g, 0.62 mmol, 1 equiv.) in THF (3 mL) and LHMDS (1.0 M in THF, 0.62 mmol, 0.62 mL, 1 equiv.) were added simultaneously to a solution of 2,2,2-trifluoro-N-(4-methoxy-phenyl)acetimidoyl chloride **3**[10] (0.220 g, 0.93 mmol, 1.5 equiv.) in THF (3 mL) at – 50 °C. The solution was allowed to warm to –20 °C and quenched by addition of water after 1.5 hours. The mixture was extracted 3 times with MTBE and the combined organic phases were washed with brine and dried over $MgSO_4$. Solvent removal and purification by column chromatography (CyH / 1% → 2% NEt_3) gave **4e** as slightly yellowish oil (0.260 g, 0.56 mmol, 90%).

$C_{24}H_{26}F_3NO_5$, MW: 465.46 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.42-7.32 (*m*, 5 H, C_6H_5), 6.87-6.76 (*m*, 4 H, C_6H_4OMe), 5.50 (*t*, *J* = 7.2, 1 H, C=C*H*), 5.17 (*s*, 2 H, OCH_2Ph), 4.77 (*d*, *J* = 6.6, 2 H, C=CHCH_2O), 4.17 (*t*, *J* = 6.9, 2 H, $CH_2CH_2CH_2O$), 3.78 (*s*, 3 H, OCH_3), 2.17 (*t*, *J* = 7.2, 2 H, $CH_2CH_2CH_2O$), 1.84 (*tt*, *J* = 7.2, *J* = 6.9, 2 H, $CH_2CH_2CH_2O$), 1.73 (*s*, 3H, C=C-CH_3). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 156.0, 155.0, 145.3 (q, $J_{C\text{-}C\text{-}F}$ = 33.9), 141.5, 137.4, 135.1, 128.4, 128.2, 128.0, 120.6, 118.1, 116.0 (q, $J_{C\text{-}F}$ = 284.4), 114.2, 113.9, 113.4, 69.5, 67.5, 55.4, 35.5, 26.6, 15.5. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –65.0. **IR (film):** ν = 2957, 2837, 1747, 1698,

1610, 1582, 1507, 1456, 1397, 1315, 1265, 1204, 1140, 1106, 1035, 947, 909, 834. **MS (EI) *m/z*:** 91.0 [100%], 219.0 [69%], 465.1 [0.4%, M^+]. **HRMS (EI) *m/z*:** Calc. for [M^+]: 465.1758. Found: 465.1753. **Anal. Calcd. for $C_{24}H_{26}F_3NO_5$:** C, 61.93; H, 5.63; N, 3.01. Found: C, 61.83, H, 5.90, N, 3.24.

(*E*)-2,2,2-Trifluoro-N-(4-methoxyphenyl)acetimidic acid 3-methyl-6-(benzyl-*t*-butyloxycarbonylamino)hex-2-enyl ester (4f)



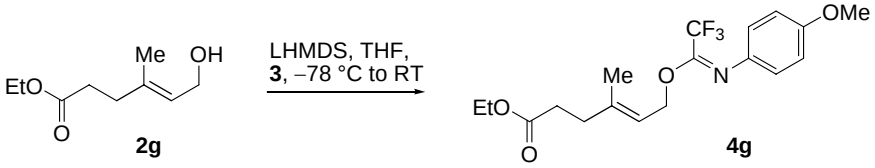
2f **4f**

A solution of (*E*)-3-methyl-6-(benzyl-*t*-butyloxycarbonylamino)hex-2-enol (**2f**, 0.313 g, 0.98 mmol, 1 equiv.) in THF (5 mL) and LHMDS (1.0 M in THF, 0.98 mL, 0.98 mmol, 1 equiv.) were simultaneously added to a solution of 2,2,2-trifluoro-N-(4-methoxyphenyl)acetimidoyl chloride **3** (0.286 g, 1.2 mmol, 1.22 equiv.) in THF (5 mL) at –50 °C. The solution was warmed to –20 °C and quenched by addition of water after 2 hours. It was extracted 3 times with MTBE and the combined organic phases were washed with brine and dried over $MgSO_4$. Solvent removal and purification by column chromatography (CyH + 1% → 2% NEt_3) gave **4f** as slightly yellowish oil (0.470 g, 0.90 mmol, 92%).

$C_{28}H_{35}F_3N_2O_4$, MW: 520.59 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.35-7.22 (*m*, 5 H, C_6H_5), 6.87-6.83 & 6.82-6.74 (*m*, 4 H, C_6H_4OMe), 5.42 (*t, J* = 6.0, 1 H, C=C*H*), 4.75 (*d, J* = 6.3, 2 H, CH_2O), 4.44 (*s*, 2 H, NCH_2Ph), 3.78 (*s*, 3 H, OCH_3), 3.15 (*bm*, 2 H, $NCH_2CH_2CH_2$), 2.02 (*bm*, 2 H, $NCH_2CH_2CH_2$), 1.68 (*s*, 3 H, C=C-CH_3), 1.67 (*bm*, 2 H, $NCH_2CH_2CH_2$), 1.49 (*bs*, 9 H, $C(CH_3)_3$). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 156.4, 156.2, 145.5 (q, $J_{C\text{-}C\text{-}F}$ = 32.7), 142.6, 138.8, 137.8,

128.6, 128.3, 127.8, 127.3, 120.9, 117.9, 116.0 (q, $J_{C\text{-}F}$ = 285.4), 114.4, 114.1, 79.8, 64.9, 55.5, 50.8 & 50.2, 46.6, 36.9, 28.6, 26.0, 16.6. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –64.9. **IR (film):** ν = 2975, 2935, 2836, 1640, 1609, 1582, 1507, 1465, 1416, 1366, 1314, 1290, 1249, 1204, 1157, 1036, 834. **MS (MALDI) *m/z*:** 543.3 [48%, $(MNa)^+$]. **HRMS (MALDI) *m/z*:** Calc. for $[MNa]^+$: 543.2441. Found: 543.2448. **Anal. Calcd. for $C_{28}H_{35}F_3N_2O_4$:** C, 64.60; H, 6.78; N, 5.38. Found: C, 64.60, H, 6.74, N, 5.47.

(*E*)-2,2,2-Trifluoro-N-(4-methoxyphenyl)acetimidic acid 3-methyl-6-oxo-7-oxa-non-2-enyl ester (4g)

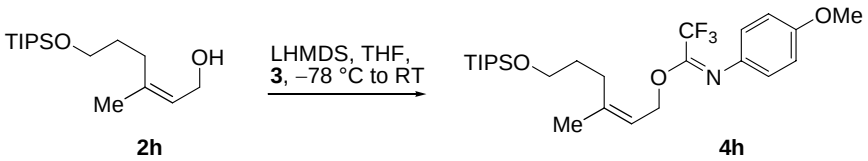


According to GP1, imidate **4g** was obtained from allylic alcohol **2g** (172 mg, 1 mmol) as a colorless oil (160 mg, 0.43 mmol, 43%).

$C_{18}H_{22}F_3NO_4$, MW: 373.37 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 6.85-6.73 (*m*, 4 H, arom-*H*), 5.47 (*t*, *J* = 6.0, 1 H, C=C*H*), 4.75 (*d*, *J* = 11.7, 2 H, C=CHCH_2O), 4.13 (*q*, *J* = 7.2, 2 H, OCH_2CH$_3$), 3.79 (*s*, 3 H, OCH_3), 2.55-2.35 (*m*, 4 H, CH_2CH_2), 1.74 (*s*, 3 H, C=C-CH_3). 1.25 (*t*, *J* = 6.9, 3 H, OCH_2CH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 173.1, 156.4, 145.5 (q, $J_{C\text{-}C\text{-}F}$ = 32.7), 141.4, 137.7, 120.8, 118.4, 116.0 (q, $J_{C\text{-}F}$ = 285.1), 114.1, 64.7, 60.5, 55.5, 34.5, 32.7, 16.7, 14.3. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –65.5. **IR (film):** ν = 2983, 2374, 1736, 1702, 1647, 1610, 1508, 1444, 1374, 1315, 1291, 1243, 1204, 1140, 1106, 1036, 835. **MS (EI) *m/z*:** 81.0 [100%], 109.0 [46%], 155.1 [56%], 219.0 [28%], 328.1 [1.1%, $(M\text{-}C_2H_5O)^+$], 373.1 [0.25%, M^+]. **HRMS**

(EI) *m/z*: Calc. for $[M\text{-}C_2H_5O]^+$: 328.1155. Found: $[M\text{-}C_2H_5O]^+$: 328.1156. **Anal. Calcd. for $C_{18}H_{22}F_3NO_4$:** C, 57.90; H, 5.94; N, 3.75. Found: C, 58.15, H, 5.95, N, 3.74.

(*Z*)-2,2,2-Trifluoro-N-(4-methoxyphenyl)acetimidic acid 3-methyl-6-triisopropylsilyloxyhex-2-enyl ester (4h)

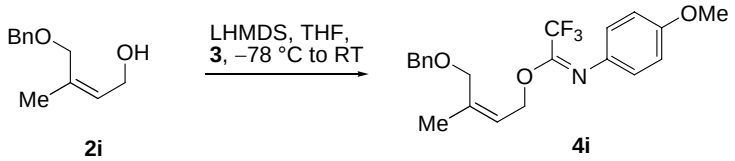


2h **4h**

According to GP1, imidate **4h** was obtained from allylic alcohol **2h** (152 mg, 0.53 mmol) as a colorless oil (257 mg, 0.53 mmol, 99%).

$C_{25}H_{40}F_3NO_3Si$, MW: 487.67 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 6.85-6.73 (*m*, 4 H, Ar-*H*), 5.48 (*t*, *J* = 6.9, 1 H, C=C*H*), 4.75 (*d*, *J* = 6,9, 2 H, C=CHCH_2O), 3.79 (*s*, 3 H, OCH_3), 3.67 (*t*, *J* = 6.3, 2 H, $CH_2CH_2CH_2$OTIPS), 2.22 (*t*, *J* = 7.5, 2 H, $CH_2CH_2CH_2$OTIPS), 1.69 (*s*, 3 H, C=C-CH_3), 1.69-1.60 (*m*, 2 H, $CH_2CH_2CH_2$OTIPS), 1.15-0.99 (*m*, 3 + 18 H, Si(C*H*(CH_3)$_3$). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 155.9, 145.3 (q, $J_{C\text{-}C\text{-}F}$ = 33.9), 143.4, 137.5, 120.6, 118.3, 116.0 (q, $J_{C\text{-}F}$ = 285.4), 113.8, 64.5, 62.6, 55.4, 31.4, 28.5, 23.6, 18.0, 12.0. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –65.1. **IR (film):** ν = 3374, 2944, 2867, 1699, 1507, 1464, 1318, 1243, 1204, 1141, 1106. **MS (EI) *m/z*:** 95.0 [100%], 219.0 [41%], 444.2 [1.5%, $(M\text{-}C_3H_7)^+$]. **HRMS (EI) *m/z*:** Calc. for $[M\text{-}C_3H_7]^+$: 444.2176. Found: 444.2178. **Anal. Calcd. for $C_{25}H_{40}F_3NO_3Si$:** C, 61.57; H, 8.27; N, 2.87. Found: C, 61.82, H, 8.26, N, 2.99.

(*Z*)-2,2,2-Trifluoro-N-(4-methoxyphenyl)acetimidic acid 3-methyl-5-benzyloxypent-2-enyl ester (4i)

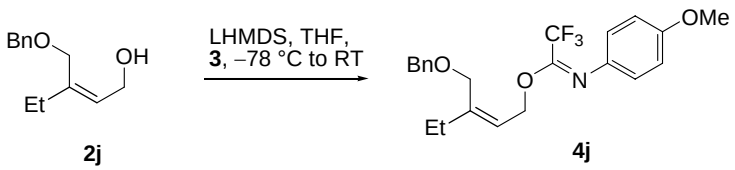


2i **4i**

According to GP1, imidate **4i** was obtained from allylic alcohol **2i** (380 mg, 2 mmol) as a colorless oil (730 mg, 1.86 mmol, 93%).

$C_{21}H_{22}F_3NO_3$, MW: 393.40 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.36-7.25 & 7.21- 7.18 (*m*, 5 H, C_6H_5), 6.87-6.83 & 6.82-6.74 (*m*, 4 H, C_6H_4OMe), 5.69 (*t, J* = 6.3, 1 H, C=C*H*), 4.81 (*d, J* = 6.6, 2 H, CH_2O), 4.49 (*s*, 2 H, OCH_2Ph), 4.11 (*s*, 2 H, CH_2OBn), 3.80 (*s*, 3 H, OCH_3), 1.92 (*s*, 3 H, C=C-CH_3). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 156.1, 145.5 (q, $J_{C\text{-}C\text{-}F}$ = 32.7), 139.4, 138.0, 137.4, 128.3, 127.6, 121.8, 120.6, 116.0 (q, $J_{C\text{-}F}$ = 285.4), 113.9, 72.1, 68.4, 63.9, 55.4, 21.8. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –65.0. **IR (film):** ν = 2950, 2837, 1699, 1610, 1587, 1454, 1380, 1338, 1314, 1290, 1243, 1205, 1141, 1106, 1036, 949, 834, 737. **MS (MALDI) *m/z*:** 416.1 [20%, $(MNa)^+$]. **HRMS (MALDI) *m/z*:** Calc. for $[MNa]^+$: 416.1444. Found: 416.1440. **Anal. Calcd. for $C_{21}H_{22}F_3NO_3$:** C, 64.11; H, 5.64; N, 3.56. Found: C, 64.15, H, 5.66, N, 3.78.

(Z)-2,2,2-Trifluoro-N-(4-methoxyphenyl)acetimidic acid 3-ethyl-5-benzyloxypent-2-enyl ester (4j)

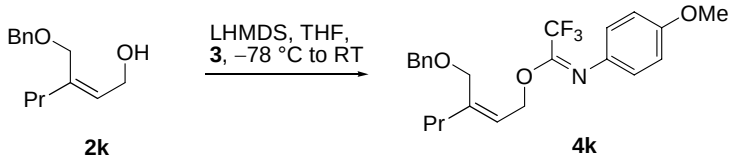


2j **4j**

According to GP1, imidate **4j** was obtained from allylic alcohol **2j** (250 mg, 1.21 mmol) as a colorless oil (298 mg, 0.73 mmol, 60%, single run, not optimised).

$C_{22}H_{24}F_3NO_3$, MW: 407,43 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.36-7.25 (*m*, 5 H, C_6H_5), 6.85-6.81 & 6.80-6.72 (*m*, 4 H, $C_6H_4OCH_3$), 5.64 (*t*, *J* = 6.3, 1 H, C=C*H*), 4.82 (*d*, *J* = 7.2, 2 H, C=CHCH_2O), 4.47 (*s*, 2 H, OCH_2Ph), 4.08 (*s*, 2 H, CH_2OBn), 3.79 (*s*, 3 H, OCH_3), 2.22 (*q*, *J* = 6.0, 2 H, C$H_2$$CH_3$), 1.05 (*t*, *J* = 7.2, 3 H, CH_2CH_3). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 156.1, 145.5 (q, $J_{C\text{-}C\text{-}F}$ = 32.7), 144.3, 138.0, 137.4, 128.3, 127.64, 127.60, 120.6, 120.4, 116.0 (q, $J_{C\text{-}F}$ = 285.4), 113.9, 72.2, 67.5, 64.1, 55.4, 28.1, 12.4. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –65.0. **IR (film):** ν = 3033, 2966, 2877, 1700, 1610, 1582, 1507, 1455, 1321, 1290, 1243, 1204, 1141, 1105, 1036, 947, 834. **MS (EI) *m/z*:** 91.0 [100%], 407.1 [0.3%, M^+]. **HRMS (EI) *m/z*:** Calc. for [M^+]: 407.1705. Found:407.1705. **Anal. Calcd. for $C_{22}H_{24}F_3NO_3$:** C, 64.86; H, 5.94; N, 3.44. Found: C, 64.91, H, 5.97, N, 3.61.

(*Z*)-2,2,2-Trifluoro-N-(4-methoxyphenyl)acetimidic acid 3-propyl-5-benzyloxy-pent-2-enyl ester (4k)



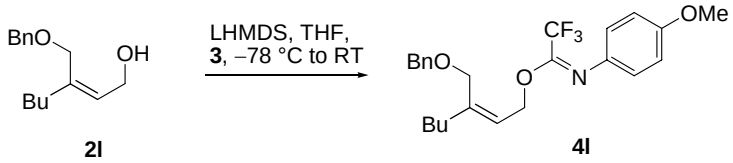
2k **4k**

According to GP1, imidate **4k** was obtained from allylic alcohol **2k** (190 mg, 0.86 mmol) as a colorless oil (267 mg, 0.63 mmol, 73%).

$C_{23}H_{26}F_3NO_3$, MW: 421.45 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.37-7.25 (*m*, 5 H, C_6H_5), 6.85-6.81 & 6.80-6.72 (*m*, 4 H, $C_6H_4OCH_3$), 5.65 (*t*, *J* = 6.6, 1 H, C=C*H*), 4.82 (*d*, *J* = 7.2, 2 H, C=CHCH_2O), 4.47 (*s*, 2H, OCH_2Ph), 4.07 (*s*, 2 H, CH_2OBn), 3.79 (*s*, 3 H, OCH_3), 2.17 (t, *J* =

6.9, 2 H, CH_2CH$_2$CH$_3$), 1.57-1.44 (m, 2 H, CH$_2$CH_2CH$_3$), 0.93 (*t*, *J* = 7.2, 3 H, CH$_2$CH$_2$CH_3). **^{13}C NMR** (75 MHz, CDCl$_3$, 21 °C): δ = 156.0, 145.5 (q, $J_{C\text{-}C\text{-}F}$ = 32.7), 142.6, 137.9, 137.4, 128.0, 127.58, 127.54, 121.6, 120.4, 116.0 (q, $J_{C\text{-}F}$ = 285.4), 113.8, 72.1, 67.3, 64.1, 55.4, 37.3, 20.9, 13.9. **^{19}F NMR** (282 MHz, CDCl$_3$, 21 °C): δ = –65.0. **IR (film):** ν = 2958, 1702, 1610, 1507, 1454, 1314, 1290, 1243, 1204, 1141, 1105, 1036, 948, 834. **MS (EI) *m/z*:** 91.0 [100%], 421.1 [0.12%, M^+]. **HRMS (EI) *m/z*:** Calc. for $[M]^+$: 421.1860. Found: 421.1860. **Anal. Calcd. for $C_{23}H_{26}F_3NO_3$:** C, 65.55; H, 6.22; N, 3.32. Found: C, 65.93, H, 6.40, N, 3.45.

(*Z*)-2,2,2-Trifluoro-N-(4-methoxyphenyl)acetimidic acid 3-butyl-5-benzyloxypent-2-enyl ester (4l)



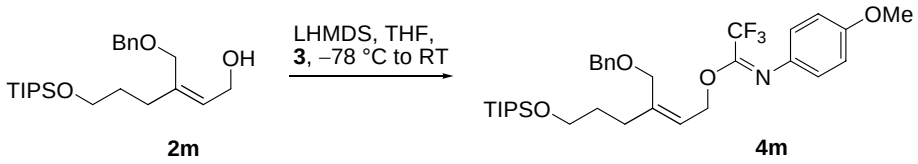
2l **4l**

According to GP1, imidate **4l** was obtained from allylic alcohol **2l** (122 mg, 0.52 mmol) as a colorless oil (177 mg, 0.4 mmol, 78%).

$C_{24}H_{28}F_3NO_3$, MW: 435,48 g/mol. **^{1}H NMR** (300 MHz, CDCl$_3$, 21 °C): δ = 7.34-7.26 (*m*, 5 H, C$_6H_5$), 6.85-6.81 & 6.80-6.72 (*m*, 4 H, C$_6H_4$OCH$_3$), 5.65 (*t*, *J* = 6.6, 1 H, C=C*H*), 4.83 (*d*, *J* = 7.2, 2 H, C=CHCH_2O), 4.47 (*s*, 2 H, OCH_2Ph), 4.07 (*s*, 2 H, CH_2OBn), 3.79 (*s*, 3 H, OCH$_3$), 2.19 (t, *J* = 67.2, 2 H, CH_2CH$_2$CH$_2$CH$_3$), 1.48-1.30 (m, 4 H, CH$_2$CH_2CH_2CH$_3$), 0.92 (*t*, *J* = 7.2, 3 H, CH$_2$CH$_2$CH$_2$CH_3). **^{13}C NMR** (75 MHz, CDCl$_3$, 21 °C): δ = 156.0, 145.5 (q, $J_{C\text{-}C\text{-}F}$ = 32.7), 142.5, 137.9, 137.4, 128.2, 128.0, 127.6, 127.5, 121.4, 120.6, 116.0 (q, $J_{C\text{-}F}$ = 285.4), 113.8, 72.1, 67.3, 64.1, 55.4, 35.0, 30.0, 22.5, 14.0. **^{19}F NMR** (282 MHz, CDCl$_3$, 21 °C): δ = –65.0. **IR (film):** ν = 2932, 2860, 1702, 1611, 1507, 1458, 1315, 1290, 1243, 1204, 1141, 1105. **MS (EI) *m/z*:** 91.0 [100],

435.2 [0.1%, M^+]. **HRMS (EI) *m/z*:** Calc. for $[M]^+$: 435.2016. Found: 435.2006. **Anal. Calcd. for $C_{24}H_{28}F_3NO_3$:** C, 66.19; H, 6.48; N, 3.22. Found: C, 66.15, H, 6.54, N, 3.35.

(*Z*)-2,2,2-Trifluoro-N-(4-methoxyphenyl)acetimidic acid 3-(3-triisopropyloxysilylpropyl)-5-benzyloxypent-2-enyl ester (4m)

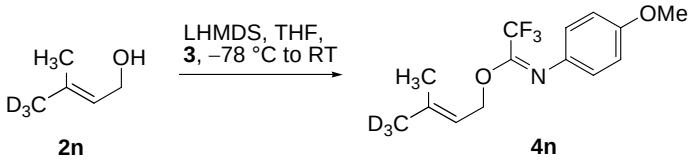


2m **4m**

According to GP1, imidate **4m** was obtained from allylic alcohol **2m** (484 mg mg, 1.23 mmol) as a colorless oil (597 mg, 1 mmol, 81%).

$C_{32}H_{46}F_3NO_4Si$, MW: 593.79 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.39-7.26 (*m*, 5 H, C_6H_5), 6.85-6.81 & 6.80-6.72 (*m*, 4 H, $C_6H_4OCH_3$), 5.68 (*t*, *J* = 6.6, 1 H, C=C*H*), 4.83 (*d*, *J* = 6.9, 2 H, C=CHCH_2O), 4.48 (*s*, 2 H, OCH_2Ph), 4.09 (*s*, 2 H, CH_2OBn), 3.79 (*s*, 3 H, OCH_3), 3.72 (*t*, *J* = 6.2, 2 H, $CH_2CH_2CH_2$OTIPS), 2.30 (*t*, *J* = 7.5, 2 H, $CH_2CH_2CH_2$OTIPS), 1.77-1.68 (m, 2 H, $CH_2CH_2CH_2$OTIPS), 1.09-1.01 (*m*, 3 + 18 H, Si(C*H*(CH_3))$_3$). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 156.1, 145.5 (q, $J_{C\text{-}C\text{-}F}$ = 32.7), 142.6, 137.9, 137.4, 128.3, 127.6, 127.5, 121.6, 120.6, 116.0 (q, $J_{C\text{-}F}$ = 285.4), 113.9, 72.2, 67.5, 64.1, 62.8, 55.4, 31.7, 31.2, 18.1, 12.1. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –65.5. **IR (film):** ν = 2944, 2866, 1699, 1611, 1582, 1507, 1464, 1384, 1365, 1339, 1315, 1290, 1243, 1205, 1141, 1105, 1037, 883, 833. **MS (EI):** 91.0 [100%, $(C_7H_7)^+$]. **Anal. Calcd. for $C_{32}H_{46}F_3NO_4Si$:** C, 64.73; H, 7.81; N, 2.36. Found: C, 64.45, H, 7.66, N, 2.57.

(*E*)-2,2,2-Trifluoro-N-(4-methoxyphenyl)acetimidic acid 3-methyl-4,4,4-trideuterobut-2-enyl ester (4n)

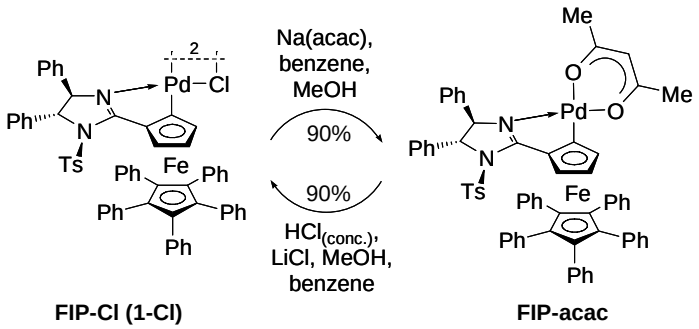


2n **4n**

Geometrically pure trideuterated prenyl alcohol **2n** was prepared following a literature procedure.[11] According to GP1, imidate **4n** was obtained from allylic alcohol **2n** (0.6 mmol) as a colorless oil (210 mg, 0.55 mmol pure material, 91%). To avoid losses of the relatively volatile deuterated allylic alcohol **2n**, it was used after reduction without further purification. The crude alcohol contained ca. 20% 3-methyl-butanol (generated by over-reduction) which gave an imidate that could not be removed by column chromatography. However, the side product does not disturb the catalytic rearrangement and can be easily removed afterwards.

$C_{14}H_{13}D_3F_3NO_2$, MW: 290.30 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 6.85-6.74 (*m*, 4 H, $C_6H_4OCH_3$), 5.45 (*t*, *J* = 6.9, 1 H, C=C*H*), 4.74 (*d*, *J* = 6.9, 2 H, C=CHCH_2O), 3.79 (s, 3 H, OCH_3), 1.73 (s, 3 H, C=C-CH_3). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 156.0, 145.5 (q, $J_{C\text{-}C\text{-}F}$ = 32.7), 139.8, 137.5, 120.6, 117.8, 116.0 (q, $J_{C\text{-}F}$ = 285.4), 114.1, 64.8, 55.4, 18.2, 18.1 (m, *C*D_3). **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –65.1. **IR (film):** ν = 2956, 2837, 1700, 1611, 1582, 1508, 1467, 1433, 1384, 1342, 1314, 1290, 1243, 1204, 1141, 1037, 834. **MS (EI) *m/z*:** 290.1 [1.6%, M^+]. **HRMS (EI) *m/z*:** Calc. for $[M]^+$: 290.1316. Found: 290.1314.

Preparation of diastereomerically pure precatalyst



FIP-Cl (1-Cl) **FIP-acac**

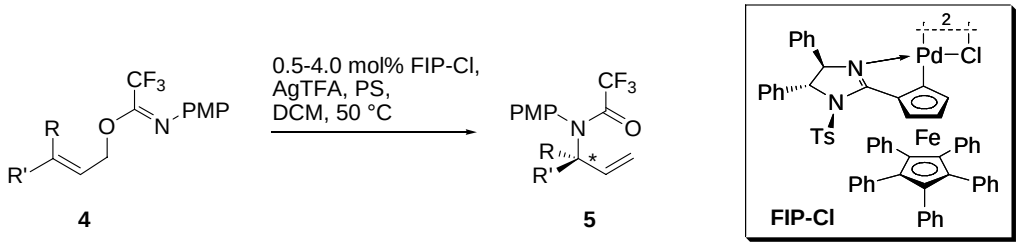
To obtain a diastereomerically pure precatalyst, the corresponding acac-complex (FIP-acac) was prepared from crude FIP-Cl (**1-Cl**) as previously described.[12] FIP-acac was dissolved in a minimum of DCE (ca. 5 mL for 1 g of crude complex) and transferred into a crystallization beaker (ca. 3 mm height of the solution, 5 cm diameter) which was placed into a desiccator containing *n*-pentane (height ca. 2 cm, ca. 25 cm diameter). After generally 1 to 2 days, dark red-purple crystals of diastereomerically pure FIP-acac had formed. The supernatant was decanted and the crystals were dried *in vacuo*. Depending of the purity of the starting material, the yield is typically 90%. In the previous cyclopalladation step, it is not necessary to completely remove all unreacted ligand since the acac-complex forms large crystals whereas the free ligand precipitates in the form of microcrystals.

FIP-acac (850 mg, 0.742 mmol, 1 equiv.) was dissolved in benzene (50 mL). A solution of conc. aqueous HCl (2 mL) and lithium chloride (ca. 5 g) in methanol (30 mL) was added. The mixture was stirred for 10 min, then it was washed 2 times with water and saturated aqueous $NaHCO_3$. The solvent was removed and the residue was purified by column chromatography (pentane / DCM 1:1) to give diastereomerically pure FIP-Cl as a dark red solid (720 mg, 90%).

The palladium-complex FIP-Cl used in catalysis contained ca. 1.5 equiv. (determined by elementary analysis) of dichloromethane or chloroform (if the complex was evaporated with chloroform prior to

use) since these solvents remain in the crystalline material and cannot be removed even after several days under high vacuum.

General procedure for the rearrangement of 3,3-disubstituted allylic trifluoroacetimidates 4 (GP2)



Catalyst activation:

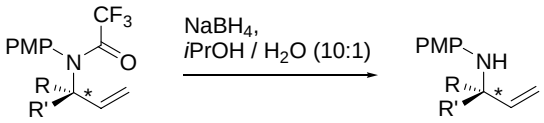
A solution of FIP-Cl (**1-Cl**, 1 equiv.) in dry DCM (0.33 mL/µmol) is added into a dry pear-shaped flask equipped with a magnetic stirring bar and containing $AgOCOCF_3$ (3.75 equiv.). The flask is sealed with a plastic cap and the suspension is stirred at RT, shielded from light, for one day. The resulting wine-red suspension is filtrated under N_2 atmosphere through *celite* / CaH_2 (~1:1) and the filter cake is washed with dry DCM (0.33 mL/µmol). Proton sponge (1 M in DCM, 3 equiv.) is added. $AgOCOCF_3$ may be handled under air but should be stored in a glove box. The following operations should be performed under dry atmosphere.

Catalysis:

The activated catalyst solution (0.5-4 mol%) is transferred into a dry flask equipped with a magnetic stirring bar containing the corresponding imidate substrate **4** (1 equiv). A stream of N_2 is passed through the flask until the solvent is nearly removed. The septum is replaced by a plastic cap and the

mixture stirred at 50 °C for the indicated time. Then, the usually highly viscous or solid residue is dissolved in a small amount of DCM (ca. 100 µL for 20 mg of product) and the resulting solution is suspended in pentane / EtOAc 9:1 (2 mL for 20 mg of product) and filtrated through a short plug of silica (eluent: pentane / EtOAc 9:1, ca. 5-10 mL for 20 mg of product), followed by removal of the solvent *in vacuo*. Further purification is performed by column chromatography (pentane:EtOAc 9:1).

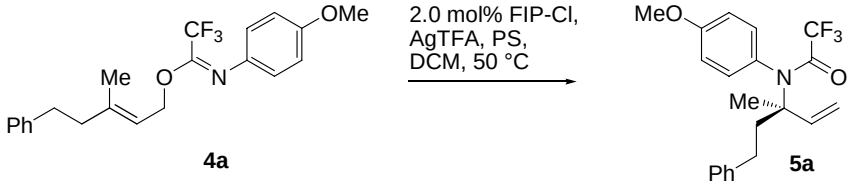
General procedure for the removal of the trifluoroacetyl protecting group (GP3)



5

The allylic amide is dissolved in an isopropanol/water mixture (10:1, ca. 1 mL for small-scale experiments up to 60 µmol, or 5 mL/mmol for larger scale experiments). Sodium borohydride (6 equiv.) is added in one portion at RT (small scale) / in three portions within 30 min at 0 °C (larger scale). The solution is then allowed to warm to RT and stirred overnight. Water is subsequently added and the mixture extracted three times with DCM or MTBE. The combined organic phases are dried over $MgSO_4$ and the solvent is removed *in vacuo*.

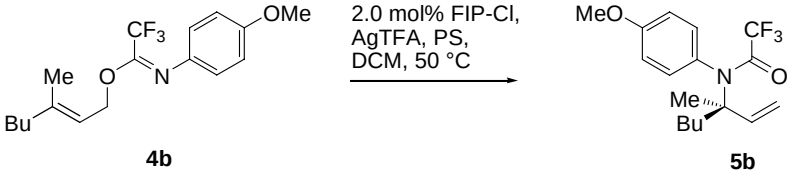
(*R*)-2,2,2-Trifluoro-*N*-(4-methoxyphenyl)-*N*-(1-phenethyl-1-methylallyl)acetamide (5a)



According to GP2, allylic amide **5a** was obtained from imidate **4a** (113/377 mg, 0.3/1 mmol, reaction time 2.5/10 d, precatalyst loading: 2.0/0.5 mol%) as a colorless oil (106/298 mg, 0.28/0.79 mmol, 94/79%, *ee* = 99.6/97%). The *ee* values were determined by chiral column HPLC: Chiralcel OD-H, *n*-hexane/*i*PrOH 99:1, 0.8 mL/min, detection at 210 nm.

$C_{21}H_{22}F_3NO_2$, MW: 377.40 g /mol. **$[\alpha]_D^{22.5°C}$** (c = 2.18, $CHCl_3$) = –22.3 (@ 99.6% *ee*). **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.29-7.17 (*m*, 5 H, C_6H_5), 7.16 & 6.89 (*d*, *J* = 9.0, 2 + 2 H, C_6H_4OMe), 6.20 (*dd*, *J* = 17.4, *J* = 10.8, 1 H, C*H*=CH_2), 5.16 (*dd*, *J* = 17.4, *J* = 10.8, 2 H, CH=CH_2), 3.84 (*s*, 3 H, OCH_3), 2.65-2.55 & 2.23-2.10 (*m*, 3 + 1 H, CH_2CH_2), 1.31 (*s*, 3 H, NCCH_3). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 159.6, 156.3 (q, $J_{C\text{-}C\text{-}F}$ = 33.3), 141.4, 140.8, 131.8, 131.7, 129.3, 128.3, 128.2, 125.8, 116.0 (q, $J_{C\text{-}F}$ = 288.1), 113.8, 113.5, 113.3, 66.5, 55.5, 39.9, 31.1, 23.3. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –67.2. **IR (film):** ν = 3063, 3027, 2990, 2937, 1697, 1606, 1583, 1510, 1456, 1444, 1397, 1299, 1525, 1202, 1183, 1151, 1108, 1072. **MS (EI) *m/z*:** 91.0 [100%], 377.1 [1.1%, M^+]. **HRMS (EI) *m/z*:** Calc. for [M^+]: 377.1597. Found: 377.1597. **Anal. Calcd. for $C_{21}H_{22}F_3NO_2$:** C, 66.83; H, 5.88; N, 3.71. Found: C, 67.09, H, 6.00, N, 3.73.

(*R*)-2,2,2-Trifluoro-*N*-(4-methoxyphenyl)-*N*-(1-butyl-1-methylallyl)acetamide (5b)

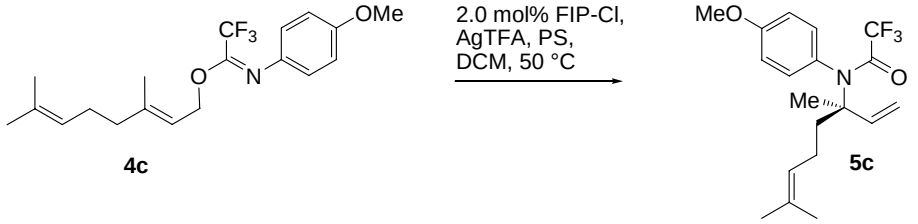


4b **5b**

According to GP2, allylic amide **5b** was obtained from imidate **4b** (25.5 mg, 77 µmol, reaction time 2.5 d, precatalyst loading: 2.0 mol%) as a colorless oil (15.9 mg, 49 µmol, 63%, *ee* = 93 %). The *ee* value was determined after removal of the trifluoroacetyl group according to GP3 [**^{1}H NMR** of the secondary amine (300 MHz, $CDCl_3$, 21 °C): δ = 6.76-6.63 (*m*, 4 H, Ar-*H*), 5.94 (*dd, J* = 16.8, *J* = 11.4, 1 H, C*H*=CH_2), 5.10 (*dd, J* = 16.8, *J* = 11.4, CH=CH_2), 3.74 (*s*, 3 H, OCH_3), 1.75-1.53 & 1.28-1.24 (*m*, 6 H, CH_2CH_2C$H_2$$CH_3$), 0.88 (*t*, *J* = 7.2, 2 H, $CH_2CH_2CH_2CH_3$)] by chiral column HPLC: Chiralcel OD-H, *n*-hexane/EtOH 99.8:0.2, 0.8 mL/min, detection at 250 nm.

$C_{17}H_{22}F_3NO_2$, MW: 329.36 g /mol. **[α]** $_D^{29.0°C}$ (c = 0.350, $CHCl_3$) = – 28.0 (@ 93% *ee*). **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.25-7.06 & 6.87-6.83 (*m*, 4 H, C_6H_4OMe), 6.11 (*dd*, *J* = 17.4, *J* = 10.8, 1 H, C*H*=CH_2), 5.09 (*dd*, *J* = 17.4, *J* = 10.8, 2 H, CH=CH_2), 3.83 (*s*, 3 H, OCH_3), 2.20-2.18 & 1.82-1.77 & 1.32-1.27 (*m*, 1+1+4 H, CH_2CH_2C$H_2$$CH_3$) 1.19 (*s*, 3 H, NCC$H_3$), 0.90 (*t, J* = 6.6, 3 H, $CH_2CH_2CH_2CH_3$). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 159.5, 156.5 (q, $J_{C\text{-}C\text{-}F}$ = 33.9), 141.3, 131.8, 129.5, 116.0 (q, $J_{C\text{-}F}$ = 287.5), 113.3, 66.6, 55.3, 37.5, 26.7, 23.2, 23.0, 14.1. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –67.2. **IR (film):** ν = 2960, 2361, 2342, 1699, 1510, 1466, 1299, 1253, 1202, 1182, 1151, 1035. **MS (EI) *m/z*:** 329.1 [0.8%, M^+]. **HRMS (EI) *m/z*:** Calc. for $[M]^+$: 329.1598. Found: 329.1595. **Anal. Calcd. for $C_{17}H_{22}F_3NO_2$:** C, 61.99; H, 6.73; N, 4.25. Found: C, 62.12, H, 6.87, N, 4.50.

(*R*)-2,2,2-Trifluoro-*N*-(4-methoxyphenyl)-*N*-(1-[4-methylpent-3-enyl]-1-methylallyl)acetamide (5c)



4c **5c**

According to GP2, allylic amide **5c** was obtained from imidate **4c** (21.9 mg, 62 µmol, reaction time 2.5 d, precatalyst loading: 2.0 mol%) as a colorless oil (16.2 mg, 46 mmol, 74%, *ee* = 98%). The *ee* value was determined by chiral column HPLC: Chiralcel OD-H, 99.8:0.2 *n*-hexane/*i*PrOH, 0.8 mL/min, detection at 210 nm.

$C_{19}H_{24}F_3NO_2$, MW: 355.39 g/mol. **[α]** $_D^{28.9°C}$ (c = 0.695, $CHCl_3$) = –24.3 (@ 98% *ee*). **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.08 (*d*, *J* = 8.1, 2 H, Ar-*H*), 6.87-6.83 (*m*, 2 H, Ar-*H*), 6.08 (*dd*, *J* = 17.4, *J* = 10.8, 1 H, C*H*=CH_2), 5.11 (m, 3 H, CH=CH_2 & Me_2C=C*H*), 3.83 (*s*, 3 H, OCH_3), 2.26-2.16 & 2.02-1.95 & 1.86-1.76 (*m*, 4 H, CH_2CH_2), 1.67 & 1.59 (s, 3 H each, C=C(CH_3)$_2$), 1.22 (*s*, 3 H, NCCH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 159.6, 156.6 (q, $J_{C\text{-}C\text{-}F}$ = 34.0), 141.1, 131.8, 129.0, 123.3, 118.0, 116.0 (q, $J_{C\text{-}F}$ = 284.2), 114.0, 113.5, 66.4, 55.2, 37.5, 25.5, 22.9, 17.4. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –67.3. **IR (film):** ν = 2928, 1699, 1608, 1511, 1465, 1379, 1299, 1253, 1206, 1183, 1152, 1108, 1035, 926. **MS (EI) *m/z*:** 219.0 [100%], 355.1 [3.3%, M^+]. **HRMS (EI) *m/z*:** Calc. for $[M]^+$: 355.1754. Found: $[M]^+$: 355.1754. **Anal. Calcd. for $C_{19}H_{24}F_3NO_2$:** C, 64.21; H, 6.81; N, 3.94. Found: C, 63.93, H, 7.01, N, 3.74.

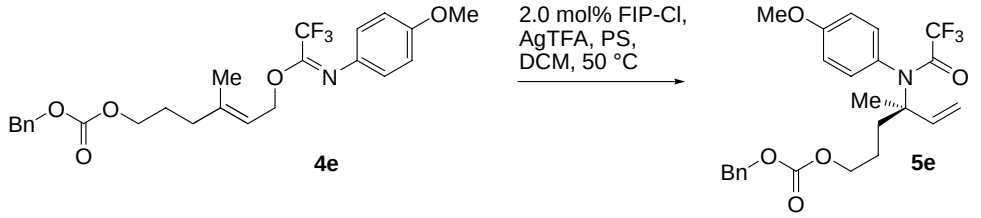
(*R*)-2,2,2-Trifluoro-*N*-(4-methoxyphenyl)-*N*-(1-triisopropylsilyloxypropyl-1-methylallyl)acetamide (5d)

4d **5d**

According to GP2, allylic amide **5d** was obtained from imidate **4d** (30.0 mg, 61 µmol, reaction time 2.5 d, precatalyst loading: 2.0 mol%) as a colorless oil (21.9 mg, 45 µmol, 73%, *ee* = 96%). The *ee* value was determined by chiral column HPLC: Chiralcel OD-H, 99.94:0.06 *n*-hexane/*i*PrOH, 0.8 mL/min, detection at 210 nm.

$C_{25}H_{40}F_3NO_3Si$, MW: 487.67 g /mol. **$[\alpha]_D^{28.9°C}$** (c = 0.740, $CHCl_3$) = –19.7 (@ 96% *ee*). All other analytical data are in accordance with the (*S*)-isomer **5h**.

(*R*)-2,2,2-Trifluoro-*N*-(4-methoxyphenyl)-*N*-(1-(3-benzyloxycarbonyloxy)-1-methylallyl)acetamide (5e)

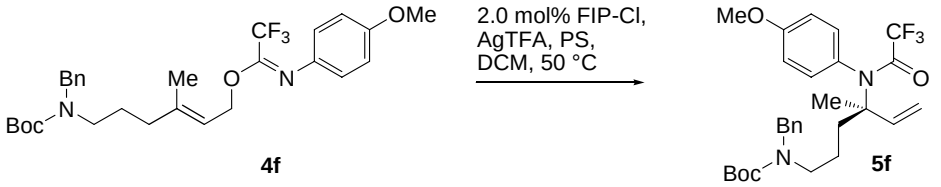


4e **5e**

According to GP2, allylic amide **5e** was obtained from imidate **4e** (31.5 mg, 68 µmol, reaction time 2.5 d, precatalyst loading: 2.0 mol%) as a colorless oil (26.4 mg, 57 µmol, 84%, *ee* = 98%). The *ee* value was determined by chiral column HPLC: Chiralcel OD-H, 97:3 *n*-hexane/*i*PrOH, 0.8 mL/min, detection at 210 nm.

$C_{24}H_{26}F_3NO_5$, MW: 465.46 g/mol. **[α]** $_D^{26.5°C}$ (c = 0.850, $CHCl_3$) = –31.3 (@98% *ee*). **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.40 – 7.32 (*m*, 5 H, C_6H_5), 7.09 (*d*, *J* = 7.3, 2 H, C_6H_4OMe), 6.85 (*d*, *J* = 7.3, 2H, C_6H_4OMe), 6.08 (*dd*, *J* = 17.4, *J* = 10.5, 1H, C*H*=CH_2), 5.15 (s, 2 H, OCH_2Ph), 5.11 (*dd*, *J* = 17.4, *J* = 10.5, 2 H, CH=CH_2), 4.17-4.11 (m, 2 H, $CH_2CH_2CH_2O$), 3.81 (*s*, 3 H, OCH_3), 2.30-2.24 & 1.93-1.86 (*m*, 1 H each, C*HH*CH_2CH_2O), 1.72-1.67 (*m*, 2 H, $CH_2CH_2CH_2O$), 1.20 (*s*, 3H, NCCH_3). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 159.7, 156.3 (q, $J_{C\text{-}C\text{-}F}$ = 34.0), 154,9, 140.6, 135.1, 131.7, 129.2, 128,4, 128.2, 116.0 (q, $J_{C\text{-}F}$ = 288.6), 113.9, 113.4, 69.4, 67.8, 66.1, 55.2, 33.6, 23.8, 22.9. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –67.7. **IR (film):** ν = 2958, 2840, 1747, 1698, 1607, 1583, 1511, 1456, 1398, 1377, 1253, 1201, 1182, 1152, 1109, 1032. 960. **MS (MALDI) *m/z*: 488.2**, [100%, $(MNa)^+$]. **HRMS (MALDI) *m/z*:** Calc. for $[MNa]^+$: 488.1655. Found: $[MNa]^+$: 488.1650. **Anal. Calcd. for $C_{24}H_{26}F_3NO_5$:** C, 61.93; H, 5.63; N, 3.01. Found: C, 61.64, H, 5.71, N, 3.08.

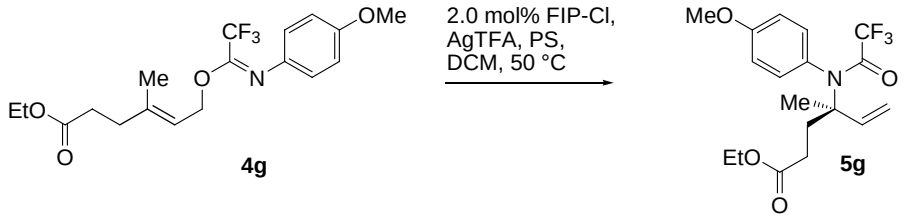
(*R*)-tert-Butyl benzyl(4-methyl-4-(2,2,2-trifluoro-N-(4-methoxyphenyl)acetamido)hex-5-enyl)carbamate (5f)



According to GP2, allylic amide **5f** was obtained from imidate **4f** (30.0 mg, 58 µmol, reaction time 2.5 d, precatalyst loading: 2.0 mol%) as a colorless oil (19.2 mg, 37 mmol, 64%, *ee* = 93%). The *ee* value was determined by chiral column HPLC: Chiralcel OD-H, 99:1 *n*-hexane/*i*PrOH, 0.8 mL/min, detection at 210 nm.

$C_{28}H_{35}F_3N_2O_4$, MW: 520.58 g/mol. **[α]** $_D^{26.6°C}$ (c = 0.500, $CHCl_3$) = –28.3 (@93% *ee*). **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.34-7.22 & 7.02-6.93 & 6.84-6.81 (*m*, 5+4 H, Ar-*H*), 6.04 (*dd*, *J* = 17.7, *J* = 10.8, 1 H, C*H*=CH_2), 5.08 (*dd*, *J* = 17.4, *J* = 10.8, 2 H, CH=CH_2), 4.42 (*bs*, 2 H, NCH_2Ph), 3.82 (*s*, 3 H, OCH_3), 3.13 (*bm*, 2 H, $CH_2CH_2CH_2N$), 2.11 (*bm*, 1 H, $CHHCH_2CH_2N$), 1.68 (*bm*, 1 H, $CHHCH_2CH_2N$), 1.46 (bm, 2 + 9 H, $CH_2CH_2CH_2N$ & $C(CH_3)_3$), 1.13 (*s*, 3 H, $NCCH_3$). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 159.9, 156.3 (q, J_{C-C-F} = 34.0), 156.0, 141.2, 138.8, 132.0, 131.9, 129.5, 128.6, 127.9, 127.3, 116.0 (q, J_{C-F} = 288.6), 113.8, 113.7, 113.5, 79.8, 66.4, 55.5, 50.9, 50.5, 47.1, 35.0, 28.5, 23.7, 23.3. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –67.7. **IR (film):** ν = 2977, 2936, 1694, 1607, 1511, 1465, 1415, 1366, 1299, 1252, 1201, 1182, 1152, 1033, 927, 877, 834, 802, 760. **MS (MALDI) *m/z*:** 542.3 [32%, $(MNa)^+$]. **HRMS (MALDI) *m/z*:** Calc. for $[MNa]^+$: 543.2441. Found: $[MNa]^+$: 543.2448. **Anal. Calcd. for $C_{28}H_{35}F_3N_2O_4$:** a sufficient elementary analysis was not obtained due to solvent inclusion.

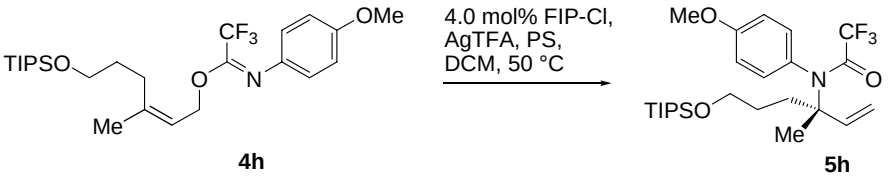
(*R*)-2,2,2-Trifluoro-*N*-(4-methoxyphenyl)-*N*-(1-[3-oxo-4-oxaheptyl]-1-methylallyl)acetamide (5g)



According to GP2, allylic amide **5g** was obtained from imidate **4g** (23.0 mg, 61 µmol, reaction time 2.5 d, precatalyst loading: 2.0 mol%) as a colorless oil (11.5 mg, 31 µmol, 50%, *ee* = 96%). The *ee* value was determined by chiral column HPLC: Chiralcel OD-H, 99.5:0.5 *n*-hexane/*i*PrOH, 0.8 mL/min, detection at 210 nm.

$C_{18}H_{22}F_3NO_4$, MW: 373.37 g /mol. **[α]** $_D^{29.2°C}$ (c = 0.420, $CHCl_3$) = –30.4 (@96% *ee*). **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.10-7.07 (*m*, 2 H, Ar-*H*), 6.87-6.84 (*m*, 2 H, Ar-*H*), 6.08 (*dd*, *J* = 17.7, *J* = 11.1, 1 H, C*H*=CH_2), 5.17 (*m*, 2 H, CH=CH_2), 4.09 (*q*, *J* = 7.2, 2 H, OC$H_2$$CH_3$), 3.83 (*s*, 3 H, OCH_3), 2.55-2.18 (*m*, 4 H, CH_2CH_2), 1.25 (*t*, *J* = 6.6, 3 H, OCH_2CH_3), 1.23 (*s*, 3H, NCCH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 172.8, 159.7, 156.6 (q, $J_{C\text{-}C\text{-}F}$ = 34.0), 140.3, 131.8, 129.0, 116.0 (q, $J_{C\text{-}F}$ = 284.2), 114.2, 113.5, 65.8, 60.5, 55.3, 32.4, 29.7, 22.5, 14.0. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –67.2. **IR (film):** ν = 2987, 1735, 1698, 1608, 1511, 1465, 1376, 1300, 1253, 1202, 1183, 1152, 1107, 1033. **MS (EI) *m/z*:** 81.0 [60%], 109.0 [46%], 155.1 [100%], 219.0 [45%], 328.1 [14%, $(M\text{-}C_2H_5O)^+$], 373.1 [0.5%, M^+]. **HRMS (EI) *m/z*:** Calc. for $[M\text{-}C_2H_5O]^+$: 328.1155. Found $[M\text{-}C_2H_5O]^+$: 328.1156. **Anal. Calcd. for $C_{18}H_{22}F_3NO_4$:** C, 57.90; H, 5.94; N, 3.75. Found: C, 58.08, H, 6.09, N, 3.91.

(*S*)-2,2,2-Trifluoro-*N*-(4-methoxyphenyl)-*N*-(1-triisopropylsilyloxypropyl-1-methylallyl)acetamide (5h)

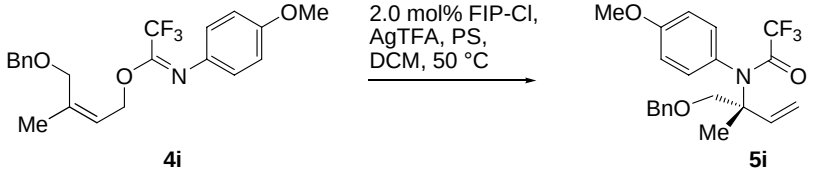


4h **5h**

According to GP2, allylic amide **5h** was obtained from imidate **4h** (34.6 mg, 70 μmol, reaction time 2.5 d, precatalyst loading: 2.0 mol%) as a colorless oil (25.2 mg, 51 μmol, 74%, *ee* = 98%). The *ee* value was determined by chiral column HPLC: Chiralcel OD-H, 99.94:0.06 *n*-hexane/*i*PrOH, 0.8 mL/min, detection at 210 nm.

$C_{25}H_{40}F_3NO_3Si$, MW: 487.67 g/mol. **[α]** $_D^{28.9°C}$ (c = 1.010, $CHCl_3$) = + 24.7 (@ 98% *ee*). **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.09 (*d*, *J* = 7.3, 2 H, Ar-*H*), 6.85 (*d*, *J* = 7.3, 2 H, ar-*H*), 6.08 (*dd*, *J* = 17.4, *J* = 10.8, 1 H, C*H*=CH_2), 5.10 (*dd*, *J* = 17.4, *J* = 10.8, 2 H, CH=CH_2), 3.83 (*s*, 3 H, OCH_3), 3.66 (*m*, 2 H, $CH_2CH_2CH_2O$), 2.10-2.04 (*m*, 1 H, C*H*HCH_2CH_2O), 2.79-2.91 (*m*, 1 H, CH*H*CH_2CH_2O), 1.45-1.61 (m, 2H, $CH_2CH_2CH_2O$), 1.25 (*s*, 3 H, $NCCH_3$), 1.05 (m, 18 + 3 H, $Si(CH(CH_3)_2)_3$). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 159.6, 156.3 (q, $J_{C\text{-}C\text{-}F}$ = 33.3), 141.2, 131.8, 131.7, 129.3, 116.0 (q, $J_{C\text{-}F}$ = 288.1), 114.2, 113.4, 113.3, 66.3, 63.1, 55.2, 33.1, 27.9, 22.9, 17.8, 11.8. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –67.2. **IR (film):** ν = 2959, 2867, 1704, 1511, 1464, 1252, 1203, 1176, 1149. **MS (EI) *m/z*:** 95.0 [100%], 444.2 [19%, $(M\text{-}C_3H_7)^+$]. **HRMS (EI) *m/z*:** Calc. for $[M\text{-}C_3H_7]^+$: 444.2177. Found: 444.2178. **Anal. Calcd. for $C_{25}H_{40}F_3NO_3Si$:** C, 61.57; H, 8.27; N, 2.87. Found: C, 61.83, H, 8.54, N, 2.91.

(*R*)-2,2,2-Trifluoro-*N*-(4-methoxyphenyl)-*N*-(1-benzyloxymethyl-1-methylallyl)acetamide (5i)

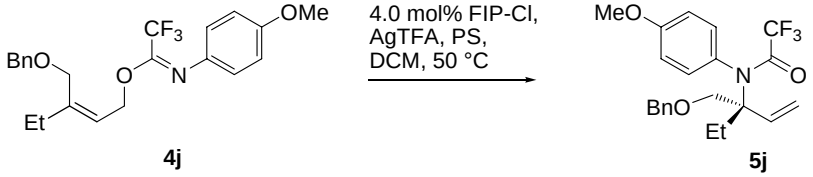


4i **5i**

According to GP2, allylic amide **5i** was obtained from imidate **4i** (25.3 mg, 64 µmol, reaction time 2.5 d, precatalyst loading: 2.0 mol%) as a colorless oil (21 mg, 54 µmol, 84%, *ee* = 99%). The *ee* value was determined by chiral column HPLC: Chiralcel OD-H, *n*-hexane/*i*PrOH 99.8:0:2, 0.8 mL/min, detection at 210 nm.

$C_{21}H_{22}F_3NO_3$, MW: 393.40 g /mol. **[α]** $_D^{28.9°C}$ (c = 0.870, $CHCl_3$) = + 3.6 (@ 99% *ee*). **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.36-7.12 (*m*, 5 + 2 H, C_6H_5 & C_6H_4OMe), 6.84-6.79 (*m*, 2 H, C_6H_4OMe), 6.17 (*dd*, *J* = 17.4, *J* = 10.8, 1 H, C*H*=CH_2), 5.17 (*dd*, *J* = 17.4, *J* = 10.8, 2 H, CH=CH_2), 3.84 (*s*, 3 H, OCH_3), 4.53 (app *dd*, *J* =17.1, *J* = 12.3, 2 H, OCH_2Ph), 4.17 & 3.64 (*d*, *J* = 9.0, 1 H each, CH_2OBn), 3.82 (*s*, 2 H, CH_2OBn), 1.22 (*s*, 3 H, NCCH_3). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 159.8, 156.5 (q, $J_{C\text{-}C\text{-}F}$ = 33.9), 139.6, 138.4, 132.9, 132.3, 129.7, 128.5, 127.8, 127.7, 116.0 (q, $J_{C\text{-}F}$ = 287.5), 114.2, 113.4, 113.2, 73.5, 73.0, 66.4, 55.5, 23.0. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –67.2. **IR (film):** ν = 3090, 3032, 299, 2864, 1697, 1607, 1582, 1510, 1455, 1404, 1271, 1300, 1251, 1202, 1184, 1151, 1105, 1033, 931, 841, 803, 761, 737. **MS (MALDI) *m/z*:** 416.2 [56%, $(MNa)^+$]. **HRMS (MALDI) *m/z*:** Calc. for $[MNa]^+$: 416.1444. Found: 416.1447. **Anal. Calcd. for $C_{21}H_{22}F_3NO_3$:** C, 64.11; H, 5.64; N, 356. Found: C, 64.23, H, 5.59, N, 3.57.

(*R*)-2,2,2-Trifluoro-*N*-(4-methoxyphenyl)-*N*-(1-benzyloxymethyl-1-ethylallyl)acetamide (5j)

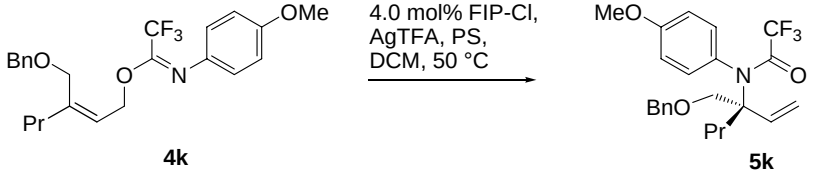


4j **5j**

According to GP2, allylic amide **5j** was obtained from imidate **4j** (26.7 mg, 65 µmol, (*E/Z)* = 4:96, reaction time 2.5 d, precatalyst loading: 4.0 mol%) as a colorless oil (18.3 mg, 44 µmol, 68%, *ee* = 91%). The *ee* value was determined by chiral column HPLC: Chiralcel OD-H, *n*-hexane/*i*PrOH 99.8:0.2, 0.8 mL/min, detection at 210 nm. Note: since the undesired (*E)*-isomer is converted to the minor enantiomer, the highest possible *ee* value would be ca. 92%.

$C_{22}H_{24}F_3NO_3$, MW: 407.43 g/mol. **[α]** $^{28.9°C}_{D}$ (c = 0.740, $CHCl_3$) = –23.5 (@ 91% *ee*). **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.35-7.19 (*m*, 5 + 2 H, C_6H_5 & C_6H_4OMe), 6.78 (*d*, *J* = 6.9, 2 H, C_6H_4OMe), 5.80 (*dd*, *J* = 17.7, *J* = 11.1, 1 H, C*H*=CH_2), 5.17 (*dd*, *J* = 17.4, *J* = 10.8, 2 H, CH=CH_2), 4.50 (*s*, 2 H, OCH_2Ph), 4.00 & 3.88 (*d, J* = 9.6, 1 H each, CH_2OBn), 3.84 (*s*, 3 H, OCH_3), 3.81 (*s*, 2 H, CH_2OBn), 1.82-1.69 (*m*, 2 H, C$H_2$$CH_3$), 0.83 (*t*, *J* = 7.5, 2 H, CH_2CH_3). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 159.4, 156.5 (q, $J_{C\text{-}C\text{-}F}$ = 33.3), 138.0, 137.8, 132.7, 132.5, 129.2, 128.5, 127.6, 127.5, 116.0 (q, $J_{C\text{-}F}$ = 288.5), 114.4, 112.9, 112.8, 73.2, 69.3, 69.0, 55.2, 26.9, 8.3. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –67.1. **IR (film):** ν = 3089, 3032, 2970, 2939, 2884, 1694, 1607, 1584, 1511, 1455, 1404, 1364, 1299, 1251, 1202, 1183, 1151, 1106, 1034, 929, 841. **MS (EI) *m/z*:** 91.0 [100%], 407.1 [0.28%, M^+]. **HRMS (EI) *m/z*:** Calc. for [M^+]: 407.1703. Found: 407.1706. **Anal. Calcd. for $C_{22}H_{24}F_3NO_3$:** C, 64.86; H, 5.94; N, 3.44. Found: C, 64.99, H, 6.01, N, 3.43.

(*R*)-2,2,2-Trifluoro-*N*-(4-methoxyphenyl)-*N*-(1-benzyloxymethyl-1-propylallyl)acetamide (5k)

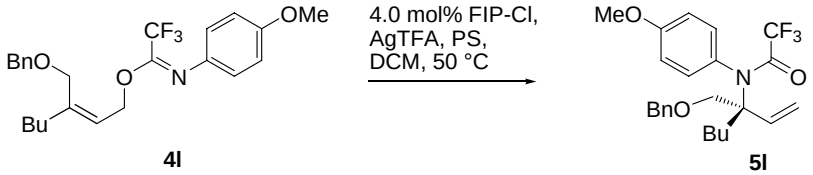


According to GP2, allylic amide **5k** was obtained from imidate **4k** (23.0 mg, 55 µmol, reaction time 3.5 d, precatalyst loading: 4.0 mol%) as a colorless oil (14.5 mg, 35 µmol, 63%, *ee* >99.5%). The *ee* value was determined after removal of the trifluoroacetyl group according to GP3 [**^{1}H NMR** of the secondary amine (300 MHz, $CDCl_3$, 21 °C): δ = 7.39-7.26 (*m*, 5 H, C_6H_5), 6.77-6.72 (*m*, C_6H_4OMe), 5.96 (*dd*, *J* = 17.7, *J* = 6.9, 1H, C*H*=CH_2), 5.20 (*dd*, *J* = 17.4, *J* = 6.9, 2 H, CH=CH_2), 4.47 (*s*, 2 H,

OCH_2Ph), 3.74 (*s*, 3 H, OCH_3), 3.45 (*s*, 2 H, CH_2OBn), 1.79-1.60 & 1.42-1.20 (*m*, 4 H, CH_2C$H_2$$CH_3$), 0.89 (*t*, *J* = 7.2, 2 H, $CH_2CH_2CH_3$)] by chiral column HPLC: Chiralcel OD-H, *n*-hexane/*i*PrOH 99.5:0.5, 0.8 mL/min, detection at 250 nm.

$C_{23}H_{26}F_3NO_3$, MW: 421.45 g /mol. **[α]** $_D^{29.5°C}$ (c = 0.312, $CHCl_3$) = –32.0 (@>99.5% *ee*). **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.38-7.19 (*m*, 5 + 2 H, C_6H_5 & C_6H_4OMe), 6.79 (*d*, *J* = 6.9, 2 H, C_6H_4OMe), 5.76 (*dd*, *J* = 17.7, *J* = 10.8, 1H, C*H*=CH_2), 5.09 (*dd*, *J* = 17.4, *J* = 10.8, 2 H, CH=CH_2), 4.49 (*s*, 2 H, OCH_2Ph), 3.93 (*s*, 2 H, CH_2OBn), 3.82 (*s*, 3 H, OCH_3), 1.78-1.59 & 1.40-1.20 (*m*, 4 H, CH_2C$H_2$$CH_3$), 0.84 (*t*, *J* = 7.2, 2 H, $CH_2CH_2CH_3$). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 159.4, 156.3 (q, $J_{C\text{-}C\text{-}F}$ = 33.3), 138.1, 132.8, 132.4, 129.3, 128.2, 128.0, 127.5, 116.0 (q, $J_{C\text{-}F}$ = 288.1), 114.2, 112.9, 72.3, 69.8, 69.2, 55.4, 36.4, 17.4, 14.5. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –67.1. **IR (film):** ν = 2965, 2871, 1697, 1608, 1510, 1455, 1400, 1300, 1250, 1201, 1183, 1151, 1106. **MS (EI) *m/z*:** 91.0 [100%], 421.1 [0.24%, M^+]. **HRMS (EI) *m/z*:** Calc. for [M^+]: 421.1860. Found: 421.1863. **Anal. Calcd. for $C_{23}H_{26}F_3NO_3$:** C, 65.55; H, 6.22; N, 3.32. Found: C, 65.64, H, 6.17, N, 3.29.

(*R*)-2,2,2-Trifluoro-*N*-(4-methoxyphenyl)-*N*-(1-benzyloxymethyl-1-butylallyl)acetamide (5l)



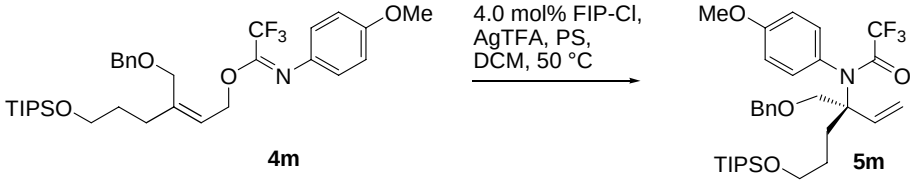
4l **5l**

According to GP2, allylic amide **5l** was obtained from imidate **4l** (13.7 mg, 30 µmol, reaction time 3.5 d, precatalyst loading: 4.0 mol%) as a colorless oil (8.4 mg, 18 µmol, 61%, *ee* = 98%). The *ee* value was determined after removal of the trifluoroacetyl-group according to GP3 [**^{1}H NMR** of the

secondary amine (300 MHz, $CDCl_3$, 21 °C): δ = 7.33-7.29 (*m*, 5 H, C_6H_5), 6.77-6.69 (*m*, 4 H, C_6H_4OMe), 5.95 (*dd*, *J* = 17.4, *J* = 10.8, 1 H, C*H*=CH_2), 5.23 (*dd*, *J* = 17.4, *J* = 10.8, 2 H, CH=CH_2), 4.47 (*s*, 2 H, OCH_2Ph), 3.74 (*s*, 3 H, OCH_3), 3.45 (*s*, 2 H, CH_2OBn), 1.78-1.57 & 1.31-1.19 (*m*, 6 H, CH_2CH_2C$H_2$$CH_3$), 0.88 (*t*, *J* = 6.9, $CH_2CH_2CH_2CH_3$)] by chiral column HPLC: Chiralcel OD-H, *n*-hexane/*i*PrOH 95:5, 0.8 mL/min, detection at 210 nm.

$C_{24}H_{28}F_3NO_3$, MW: 435.48 g/mol. **[α]** $_D^{28.9°C}$ (c = 0.567, $CHCl_3$) = –25.2 (@98% *ee*). **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.37-7.20 (*m*, 5 +2 H, C_6H_5 & C_6H_4OMe), 6.81-6.77 (m, 2 H, C_6H_4OMe), 5.77 (*dd*, *J* = 17.4, *J* = 10.8, 1 H, C*H*=CH_2), 5.10 (*dd*, *J* = 17.1, *J* = 10.8, 2 H, CH=CH_2), 4.49 (*s*, 2 H, OCH_2Ph), 3.81 (*s*, 3 H, OCH_3), 3.39 (*s*, 2 H, CH_2OBn), 1.78-1.57 & 1.23-1.17 (*m*, 6 H, CH_2CH_2C$H_2$$CH_3$), 0.84 (*t*, *J* = 6.6, $CH_2CH_2CH_2CH_3$). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 159.3, 156.3 (q, $J_{C\text{-}C\text{-}F}$ = 33.3), 138.1, 132.7, 132.4, 129.3, 128.2, 127.5, 116.0 (q, $J_{C\text{-}F}$ = 288.1), 114.3, 113.8, 112.8, 73.3, 69.7, 69.1, 55.3, 33.9, 26.2, 23.1, 14.0. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –67.1. **IR (film):** ν = 2968, 2939, 2872, 1698, 1608, 1510, 1456, 1400, 1299, 1251, 1202, 1182, 1151, 1107, 1034, 927, 841, 803, 737. **MS (EI) *m/z*:** 91.0 [100%], 435.2 [0.59%, M^+]. **HRMS (EI) *m/z*:** Calc. for [M^+]: 435.2016. Found: 435.2020. **Anal. Calcd. for $C_{24}H_{28}F_3NO_3$:** C, 66.19; H, 6.48; N, 3.22. Found: C, 66.34, H, 6.62, N, 3.30.

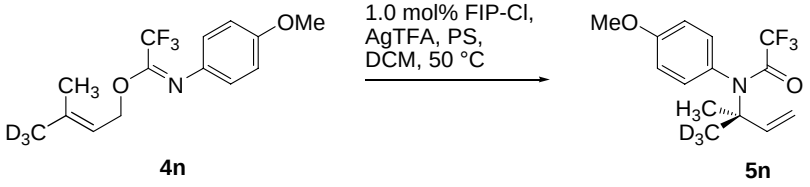
(*R*)-2,2,2-Trifluoro-*N*-(4-methoxyphenyl)-*N*-(1-benzyloxymethyl-1-[3-triisopropylsilyloxypropyl]allyl)acetamide (5m)



According to GP2, allylic amide **5m** was obtained from imidate **4m** (18.0 mg, 30 µmol, reaction time 3.5 d, precatalyst loading: 4.0 mol%) as a colorless oil (9.2 mg, 15 µmol, 51%, (*ee* = 97%). The *ee* value was determined by chiral column HPLC: Chiralcel OD-H, *n*-hexane/EtOH 99.97:0.03 (i.e. 300 ppm EtOH), 0.8 mL/min, detection at 210 nm.

$C_{32}H_{46}F_3NO_4Si$, MW: 593.79 g/mol. **$[\alpha]_D^{29.0°C}$** (c = 0.805, $CHCl_3$) = –15.8 (@97% *ee*). **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.37-7.18 (*m*, 5+2 H, C_6H_5 & C_6H_4OMe), 6.77 (*d, J* = 7.5, 2 H, C_6H_4OMe), 5.80 (*dd*, *J* = 17.7, *J* = 11.1, 1 H, C*H*=CH_2), 5.15 (*dd*, *J* = 17.7, *J* = 11.1, 2 H, CH=CH_2), 4.51 (*app q, J* = 3.9, 2 H, OCH_2Ph), 4.07 & 3.87 (*d, J* = 9.9, 1 H each, CH_2OBn), 3.81 (*s*, 3 H, OCH_3), 3.58 (*t*, *J* = 5.7, 2 H, $CH_2CH_2CH_2O$), 1.95-1.80 & 1.70-1.50 & 1.49-1.44 (*m*, 2 H each, $CH_2CH_2CH_2O$), 1.07-0.95 (m, 18 + 3 H, Si(C*H*(CH_3)$_2$)$_3$). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 159.4, 156.3 (q, $J_{C\text{-}C\text{-}F}$ = 33.3), 138.07, 138.04, 132.6, 129.1, 128.2, 127.5, 116.0 (q, $J_{C\text{-}F}$ = 288.1), 114.5, 113.0, 112.9, 73.3, 69.6, 68.8, 63.2, 55.3, 30.9, 27.6, 18.1, 12.0. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –67.2. **IR (film):** ν = 2944, 2866, 1698, 1608, 1510, 1463, 1390, 1366, 1300, 1251, 1202, 1182, 1152, 1106, 1035, 882. **MS (MALDI) *m/z*:** 616.3 [100%, $(MNa)^+$]. **HRMS (MALDI) *m/z*:** Calc. for $[MNa]^+$: 616.3040. Found: 616.3029. **Anal. Calcd. for $C_{32}H_{46}F_3NO_4Si$:** C, 64.73; H, 7.81; N, 2.36. Found: C, 65.01, H, 7.77, N, 2.28.

(*R*)-2,2,2-Trifluoro-N-(4-methoxyphenyl)-N-(1-trideuteromethyl-1-methyl]-allyl)acetamide (5n)

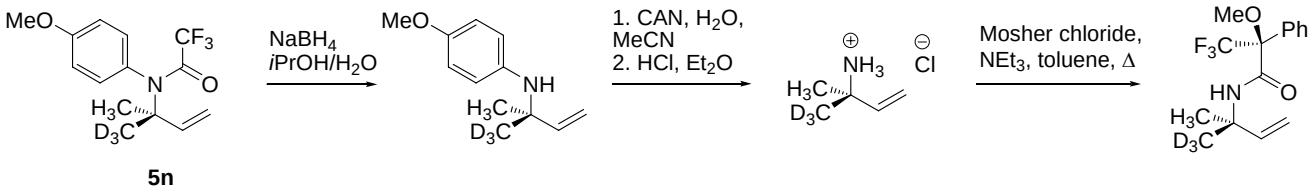


4n **5n**

According to GP2, allylic amide **5n** was obtained from imidate **4n** (200 mg, 0.55 mmol, reaction time 2.5 d, precatalyst loading: 1.0 mol%) as a colorless oil (155 mg, 0.53 mmol, 95%, *ee* = 96%). The *ee* value was determined by ^{1}H-NMR after complete deprotection of the amino group and formation of the corresponding Mosher amide (see below).

$C_{14}H_{13}D_3F_3NO_2$, MW: 290.30 g/mol. **[α]** $[\alpha]_D^{22.3°C}$ (c = 0.435, $CHCl_3$) = –0.8 (@ 96% *ee*). **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.10 (*d*, *J* = 9.0, 2 H, C_6H_4OMe), 6.85 (*d*, *J* = 9.0, 2 H, C_6H_4OMe), 6.13 (*dd*, *J* = 18.0, *J* = 10.8, 1H, C*H*=CH_2), 5.10 (*dd*, *J* = 17.4, *J* = 10.8, 2 H, CH=CH_2), 3.82 (*s*, 3 H, OCH_3), 1.35 (*s*, 3 H, NCCH_3). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 159.7, 156.3 (q, $J_{C\text{-}C\text{-}F}$ = 33.3), 142.6, 131.5, 116.0 (q, $J_{C\text{-}F}$ = 288.1), 113.4, 112.3, 63.2, 55.4, 26.4. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –67.4. **IR (film):** ν = 3089, 2982, 2939, 2841, 2360, 2234, 1698, 1608, 1511, 1465, 1444, 1374, 1300, 1253, 1203, 1184, 1151, 1108, 1035. **MS (EI) *m/z*:** 290.1 [4.5%, M^+]. **HRMS (EI) *m/z*:** Calc. for $[M]^+$: 290.1316. Found: 290.1316. **Anal. Calcd. for $C_{14}H_{13}D_3F_3NO_2$:** C, 57.92; H*, 5.61; N, 4.82. Found: C, 58.01, H*: 5.73, N, 5.01. * Deuterium was for technical reasons measured as hydrogen.

(*2S*)-3,3,3-Trifluoro-2-methoxy-N-((*1R*)-1-methyl-1-trideuteromethylallyl)-2-phenylpropionamide



Allylic amide **5n** (80 mg, 0.27 mmol) was treated with sodium borohydride following GP3 to give the corresponding secondary amine (62 mg, 0.21 mmol, 78%) as colorless oil which was used in the

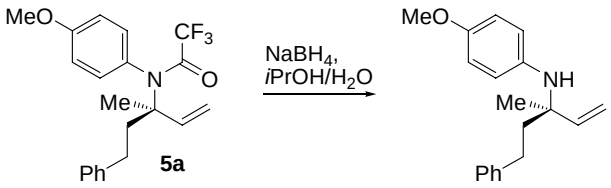
next step without further purification. [**^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 6.72 (s, 4 H, C_6H_4OMe), 6.02 (*dd*, *J* = 18.0, *J* = 10.8, 1H, C*H*=CH_2), 5.10 (*dd*, *J* = 17.4, *J* = 10.8, 2 H, CH=CH_2), 3.74 (*s*, 3 H, OCH_3), 1.32 (*s*, 3 H, NCCH_3).]. A solution of this amine (0.067 mmol, 1 equiv.) in acetonitrile (2 mL) was added dropwise within 10 min to a solution of CAN (92 mg, 0.168 mmol, 2.5 equiv.) in water (2 mL) at 0 °C. After 3 h at RT, diethylether (2 mL) and water (2 mL) were added, the phases were separated and the aqueous phase was washed with ether, whereas the organic phase was discarded. The aqueous phase was then basified with sodium carbonate to pH 10 and extracted four times with diethylether (5 mL each). The combined organic phases were dried over $MgSO_4$, then HCl (1 M in diethylether, 1 mL, 1 mmol) was added and the solvents were removed *in vacuo* to give the corresponding hydrochloride salt of the primary amine as light brown solid which was used in the next step without further purification [**^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 8.5 (*bs*, 3 H, $^+$NH_3Cl), 6.00 (*m*, 1H, C*H*=CH_2), 5.44 & 5.25 (*d*, *J* = 10.8, 2 H, CH=C*HH*), 1.42 (*s*, 3 H, NCCH_3)]. To a solution of the hydrochloride salt in toluene (1 mL) was added triethylamine (0.1 mL) and (*S*)-configured Mosher's acid chloride (24 mg, 0.1 mmol). The solution was heated to 110 °C for 1 h, then water was added and the mixture was extracted twice with MTBE. The combined organic phases were dried over $MgSO_4$ and the solvent was removed *in vacuo*. Purification by column chromatography (CyH / EtOAc 20:1) gave the amide as colorless oil (8 mg, 26 µmol, 39% over two steps from the PMP-protected amine). The diastereomeric ratio of 98:2 was determined by comparison of the integrals of the diastereotopic methyl groups (1.240 and 1.230 ppm, C_6D_6**,** 600 MHz).

$C_{15}H_{15}D_3F_3NO_2$, MW = 304.32 g/mol. **[α]** $_D^{22.4°C}$ (c = 0.140, $CHCl_3$) = –14.4 **^{1}H-NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.71 (*s*, 1 H, NH), 7.15-6.98 (m, 5 H, C_6H_5), 5.85 (*dd*, *J* = 18.0, *J* = 10.8, 1H, C*H*=CH_2), 4.92 & 4.86 (*d*, *J* = 10.8, 2 H, CH=C*HH*), 3.12 (s, 3 H, OCH_3),1.23 (*s*, 3 H, NCCH_3)].

^{13}C NMR (75 MHz, $CDCl_3$, 21 °C): δ = 164.9, 142.9, 132.7, 129.2, 128.4, 127.5, 112.3, 55.0, 54.4, 29.8, 26.7. **^{19}F NMR** (282 MHz, $CDCl_3$, 21 °C): δ = –68.5. **IR (film):** ν = 2490, 1702, 1508, 1268, 1166, 1106, 1008, 939. **MS (EI) *m/z*:** 189.0 [100%, $(PhC(OMe)CF_3)^+$]. **HRMS (EI) *m/z*:** Calc. for $[PhC(OMe)CF_3]^+$: 189.0527. Found: 189.0517.

Formation of enantioenriched quaternary amino acids

(*R*)-*N*-(4-Methoxyphenyl)-*N*-(1-phenethyl-1-methylallyl)amine

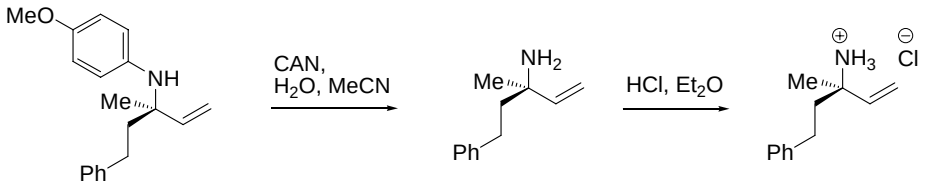


Following GP3 (*R*)-2,2,2-Trifluoro-*N*-(4-methoxyphenyl)-*N*-(1-phenethyl-1-methylallyl)acetamide (**5a**, 1.720 g, 4.55 mmol) was converted to (*R*)-*N*-(4-methoxyphenyl)-*N*-(1-phenethyl-1-methyl-allyl)amine which was obtained as colorless oil (0.997 g, 3.55 mmol, 78%) after purification by column chromatographe (pentane / EtOAc 9:1).

$C_{19}H_{23}NO$, MW: 281.18 g/mol. **$[\alpha]_D^{26.3°C}$** (c = 0.760, $CHCl_3$) = –19.1. **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C): δ** = 7.32-7.15 (*m*, 5 H, C_6H_5), 6.76 (s, 4 H, C_6H_4OMe), 6.03 (*dd*, *J* = 18.0, *J* = 10.8, 1 H, $CH{=}CH_2$), 5.25 (*dd*, *J* = 17.4, *J* = 10.8, 2 H, $CH{=}CH_2$), 3.77 (*s*, 3 H, OCH_3), 3.41 (*bs*, 1 H, N*H*), 2.74-2.62 & 2.09-1.85 (*m*, 2 H each, CH_2CH_2), 1.41 (*s*, 3 H, $NCCH_3$). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C): δ** = 152.5, 145.4, 142.3, 140.0, 128.4, 128.3, 125.7, 118.6, 114.2, 113.6, 57.8, 55.7, 42.9, 30.3, 25.1. **IR (film): ν** = 3400, 3061, 3026, 2948, 2832, 1603, 1511, 1454, 1411, 1371, 1297, 1236, 1179, 1039. **MS (MALDI) m/z**: 281.1 [56%, M^+], 282.1 [80%, $(MH)^+$]. **HRMS (MALDI) m/z:**

Calc. for $[M]^+$: 281.1774. Found: 281.1772. **Anal. Calcd. for $C_{19}H_{23}NO$**: C, 81.10; H, 8.24; N, 4.98. Found: C, 81.05, H, 8.25, N, 5.05.

(*R*)-(1-Phenethyl-1-methylallyl)amine and (*R*)-(1-phenethyl-1-methylallyl)amine hydrochloride

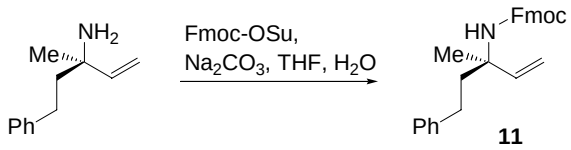


(*R*)-*N*-(4-Methoxyphenyl)-*N*-(1-phenethyl-1-methylallyl)amine (0.730 g, 2.60 mmol, 1 equiv.) in acetonitrile (20 mL) was added dropwise within 15 minutes to a solution of ceric ammonium nitrate (3.559 g, 6.49 mmol, 2.5 equiv.) in water (20 mL) at 0 °C. After 15 minutes, the reaction was allowed to warm to RT and stirred for additional 2 hours before diethylether and water were added. The phases were separated and the aqueous phase was washed with diethylether. The aqueous phase was basified to pH 10 with sodium carbonate and extracted three times with diethylether. The combined organic phases were dried over $MgSO_4$ and the solvent was removed under reduced pressure to give (*R*)-(1-Phenethyl-1-methylallyl)amine as a brown oil that was used in the next step without further purification. Purification is possible by formation of the hydrochloride salt with hydrogen chloride in diethylether (1.0 M), followed by trituration with pentane. All analytical data are given for the hydrochloride salt.

$C_{12}H_{18}NCl$, MW: 211.73 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 8.82 (*bs*, 3 H, $^+NH_3$), 7.26-7.15 (*m*, 5 H, C_6H_5), 5.93 (*dd*, *J* = 17.7, *J* = 11.1, 1 H, C*H*=CH_2), 5.48 & 5.28 (*d*, *J* = 17.4, 1 H each, CH=C*HH*), 2.77-2.67 & 2.19-2.05 (*m*, 2 H each, CH_2CH_2), 1.57 (*s*, 3 H, NCCH_3). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 140.4, 137.7, 128.4, 138.3, 126.0, 116.9, 58.3, 41.9, 30.2, 24.3. **IR**

(film): ν = 2887, 2064, 1615, 1516, 1454, 1428, 1384, 1324, 1234, 1074, 1029, 885, 925. **MS (ESI) *m/z*:** 211.1 [0.5%, M^+]. **Anal. Calcd. for $C_{12}H_{18}NCl$:** C, 68.07; H, 8.57; N, 6.62. Found: C, 67.80, H, 8.46, N, 6.63.

(*R*)-(1-Methyl-1-phenethylallyl) carbamic acid 9*H*-fluoren-9-ylmethyl ester (11)



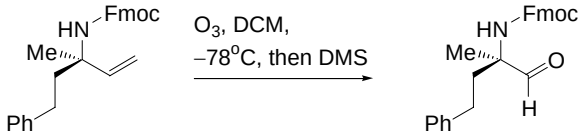
11

Crude (*R*)-(1-phenethyl-1-methylallyl)amine (0.455 g, 2.6 mmol, 1 equiv.) and Fmoc-N-hydroxysuccinimide ester (0.876 g, 2.6 mmol, 1 equiv.) were dissolved in THF (10 mL). At RT, a solution of sodium carbonate (0.276 g, 2.6 mmol, 1 equiv.) in water (1.5 mL) was added. The reaction mixture was stirred at RT overnight, then water was added and the mixture was extracted three times with MTBE. The combined organic phases were washed with 1 N aqueous HCl and dried over $MgSO_4$. The solvent was removed under reduced pressure and the residue purified by column chromatography (CyH / EtOAc 9:1) to give **11** as beige solid (0.730 g, 1.84 mmol, 73% over two steps from the trifluoroacetamide).

$C_{27}H_{27}NO_2$, MW: 397.20 g/mol. **$[\alpha]_D^{24.7°C}$** (c = 1.25, $CHCl_3$) = –8.9. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.79-7.76 & 7.63-7.60 & 7.41-7.15 (*m*, 5+8 H, Ar-*H*), 5.95 (*bm*, 1 H, C*H*=CH_2), 5.16 (*bm*, 2 H, CH=CH_2), 4.08 (*b*s, N*H*), 4.42 (*bs*, 2 H, OCH_2CHAr$_2$), 4.20 (*t, J* = 6.6, 1 H, OCH$_2$C*H*Ar$_2$), 2.57 & 2.07 (*bs*, 2 H each, CH_2CH_2), 1.46 (*s*, 3 H, NCCH_3). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 154.7, 144.2, 143.1, 142.1, 141.5, 128.6, 127.8, 127.2, 126.0, 125.1, 120.1, 113.0, 66.1, 56.7, 47.5, 41.4, 30.4, 24.9. **IR (film):** ν = 3414, 3347, 3064, 3025, 3940, 1728, 1641, 1604, 1503, 1450, 1412, 1372, 1330, 1247, 1085. **MS (MALDI) *m/z*:** 420.2 [100%, $(MNa)^+$]. **HRMS (MALDI) *m/z*:** Calc.

for $[MNa]^+$: 420.1934. Found: 420.1937. **Anal. Calcd. for $C_{27}H_{27}NO_2$:** C, 81.58; H, 6.85; N, 3.52. Found: C, 81.40, H, 6.90, N, 3.55.

(*R*)-2-(9*H*-Fluoren-9-ylmethoxycarbonylamino)-2-methyl-4-phenylbutanal

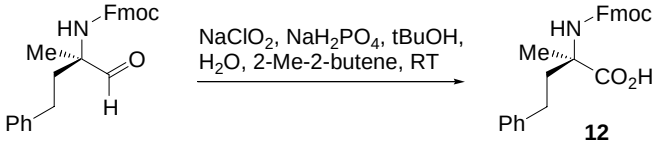


11

(*R*)-N-Fmoc-(1-phenethyl-1-methylallyl)amine (**11**, 0.650 g, 1.64 mmol, 1 equiv.) was dissolved in DCM (50 mL) and cooled to –78 °C. Ozone (gas flow 50 L/h) was passed through the solution until a blue color appeared. The solution was purged with nitrogen until the color had disappeared, then dimethylsulfide (1.1 mL) was added. After additional 30 minutes at –78 °C, the solution was warmed to RT and the solvent was removed under reduced pressure. The crude residue was used in the next step without further purification.

$C_{26}H_{25}NO_3$, MW: 429.19 g/mol. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 9.28 (*s*, 1 H, CHO), 7.78-7.76 & 7.61-7.59 & 7.42-7.10 (*m*, 5+8 H, Ar-*H*), 5.50 (*bs*, 1 H, N*H*), 4.39 (*bs*, 2 H, OCH_2CHAr$_2$), 4.21 (*t, J* = 6.6, 1 H, OCH$_2$C*H*Ar$_2$), 2.40 & 2.00 (*bs*, 2+2 H, CH_2CH_2), 1.44 (*s*, 3 H, NCCH_3).

(*R*)-2-(9*H*-Fluoren-9-ylmethoxycarbonylamino)-2-methyl-4-phenyl-butanoic acid (12)

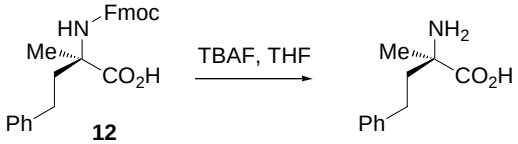


12

(*R*)-3-(9*H*-Fluoren-9-ylmethoxycarbonylamino)-2-methyl-4-phenyl-butanal (0.650 g, 1.63 mmol) was dissolved in *t*-butanol (20 mL) and 2-methyl-2-butene (2.5 mL). A solution of sodium chlorite (1.476 g, 16.3 mmol, 10 equiv.) and sodium dihydrogenphosphate (0.880 g, 5 mmol, 3 equiv.) was added portionwise within 10 minutes and the reaction mixture was stirred at RT for two hours. Subsequently, aqueous HCl (0.1 M, 20 mL) was added and the mixture was extracted three times with MTBE. The combined organic phases were washed with brine and dried over $MgSO_4$. After solvent removal, the residue was extracted with pentane and DCM. The extracts was evaporated and purified by column chromatography (DCM / MeOH 40:1 + 1% acetic acid) to give acid **12** as colorless oil (0.575 g, 1.38 mmol, 85% over two steps).

$C_{26}H_{25}NO_4$, MW: 415.17 g/mol. **$[\alpha]_D^{23.9°C}$** (c = 1.60, $CHCl_3$) = –6.3. **^{1}H NMR** (300 MHz, CD_3OD, 21 °C): δ = 7.79-7.76 & 7.61-7.59 & 7.39-7.10 (*m*, 5+8 H, Ar-*H*), 4.87 (*bs*, 2 H, OCH_2CHAr_2), 4.32 (*bs*, 1 H, N*H*), 4.19 (*t, J* = 6.6, 1 H, OCH_2CHAr_2), 2.55 & 2.17 (*t, J* = 8.1, 2 H each, CH_2CH_2), 1.52 (*s*, 3 H, $NCCH_3$). **^{13}C NMR** (75 MHz, CD_3OD, 21 °C): δ = 177.3, 156.9, 145.1, 142.3, 142.2, 129.1, 128.5, 127.9, 126.5, 120.6, 67.2, 60.1, 39.6, 31.2, 23.2. **IR (AT-IR):** ν = 3013, 2950, 2412, 2083, 1949, 1686, 1603, 1498, 1477, 1466, 1451, 1417, 1338, 1293, 1272, 1249, 1211, 1129, 1086, 1037, 932, 905. **MS (MALDI) *m/z*:** 438.2, [100%, $(MNa)^+$]. **HRMS (MALDI) *m/z*:** Calc. for $[MNa]^+$: 438.1676. Found: 438.1681. A sufficient microanalysis could not be obtained due to solvent inclusion.

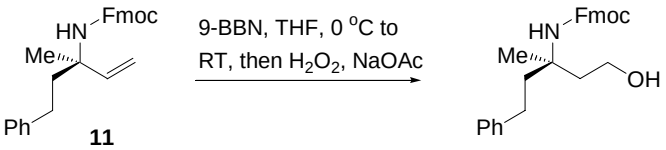
(*R*)-2-Amino-2-methyl-4-phenyl-butanoic acid



(*R*)-2-(9H-Fluoren-9-ylmethoxycarbonylamino)-2-methyl-4-phenylbutanoic acid (**12**, 42 mg, 0.1 mmol, 1 equiv.) was dissolved in DMF (0.5 mL) and a solution of tetra-*n*-butylammoniumfluoride (1.0 M in THF, 0.3 mL, 0.3 mmol, 3 equiv.) was added at RT. The solution was stirred overnight, then water was added and the mixture was washed with MTBE. The aqueous phase was lyophilized and the remaining solid was triturated with THF to give (*R*)-2-amino-2-methyl-4-phenyl-butanoic acid as a white powder (18 mg, 0.093 mmol, 93%). The absolute configuration was determined by comparison of the specific optical rotation ($[\alpha]_D^{21.2°C}$ (c = 0.46, MeOH) = –15.7) with reported data (for the (*R*)-isomer: $[\alpha]_D^{20°C}$ (c = 0.24, MeOH) = –27).[13]

$C_{26}H_{25}NO_4$, MW: 415.17 g/mol. $[\alpha]_D^{21.2°C}$ (c = 0.46, MeOH) = –15.7. **^{1}H NMR** (300 MHz, CD_3OD, 21 °C): δ = 7.26-7.18 (*m*, 5 H, arom-*H*), 2.76 & 2.54 & 2.19-2.08 & 1.95-1.84 (m, 2+1+1 H, CH_2CH_2), 1.49 (*s*, 3 H, $NCCH_3$). All other analytical data were in accordance with literature.[13,14]

(*R*)-(9H-Fluoren-9-yl)methyl 1-hydroxy-3-methyl-5-phenylpentan-3-ylcarbamate

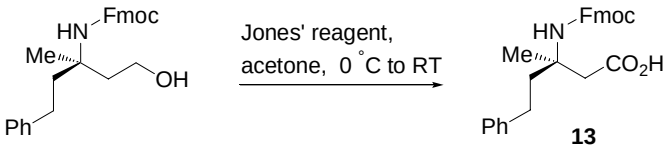


(*R*)-N-Fmoc-(1-phenethyl-1-methylallyl)amine (**11**, 0.240 g, 0.60 mmol, 1 equiv.) was dissolved in THF (8 mL) and cooled to 0 °C. A solution of 9-BBN (0.5 M in THF, 3.60 mL, 1.8 mmol, 3 equiv.) was added dropwise and the reaction was subsequently stirred at RT for 2 hours. Then a solution of sodium acetate (0.298 g, 3.65 mmol, 6.1 equiv.) and aqueous hydrogen peroxide (30%, 1.2 mL, 9.1 mmol, 15.2 equiv.) in water (1.2 mL) was added at 0 °C and stirring was continued for one hour. The mixture was extracted three times with MTBE and the combined organic phases were dried over $MgSO_4$. The solvent was removed under reduced pressure and the residue purified by column

chromatography (CyH / EtOAc 4:1 → 2:1) to give (*R*)-(9H-fluoren-9-yl)methyl 1-hydroxy-3-methyl-5-phenylpentan-3-ylcarbamate as a colorless oil (0.180 g, 0.43 mmol, 73%).

$C_{27}H_{29}NO_3$, MW: 415.21 g/mol. **$[\alpha]_D^{22.4°C}$** (c = 0.94, $CHCl_3$) = –6.2. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.79-7.76 & 7.63-7.60 & 7.41-7.16 (*m*, 5+8 H, Ar-*H*), 5.29 (*bs*, 1 H, N*H*), 4.40 (*bs*, 2 H, OC*H*$_2$CH-Ar$_2$), 4.23 (*t, J* = 6.6, 1 H, OCH$_2$C*H*Ar$_2$), 3.77 (*bs*, 2 H, CH$_2$C*H*$_2$OH), 2.57 & 2.07 & 1.85 (*bs*, 2 H each, C*H*$_2$C*H*$_2$ & C*H*$_2$CH$_2$OH), 1.37 (*s*, 3 H, NCC*H*$_3$). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 154.7, 143.9, 141.9, 141.2, 128.3, 127.5, 126.9, 125.7, 124.8, 119.8, 65.7, 59.7, 47.3, 41.1, 40.8, 30.1, 24.7. **IR (film):** ν = 3347, 3064, 3025, 2943, 1706, 1603, 1513, 1451, 1378, 1256, 1088. **MS (MALDI) *m/z*** 438.2 [100%, $(MNa)^+$]. **HRMS (MALDI) *m/z*:** Calc. for [$(MNa)^+$]: 438.2040. Found: 438.2047. A sufficient microanalysis could not be obtained due to solvent inclusion.

(*R*)-3-(9H-Fluoren-9-ylmethoxycarbonylamino)-3-methyl-5-phenylpentanoic acid (13)



13

Jones-reagent was prepared by dissolving chromium trioxide (0.54 g, 5.4 mmol) in concentrated sulfuric acid (0.46 mL, 8.6 mmol), followed by addition of water (2 mL). To a solution of (*R*)-(3-hydroxy-1-methyl-1-phenethyl-propyl) carbamic acid 9*H*-fluoren-9-ylmethyl ester (0.130 g, 0.313 mmol, 1 equiv.) in acetone (9 mL) at 0 °C was added the *Jones*-reagent (0.350 mL, 0.94 mmol, 3 equiv.). The mixture was stirred at RT for three hours, then isopropanol was added. The mixture was filtrated through a pad of cotton wool, diluted with DCM, washed with brine and dried over $MgSO_4$.

The solvent was removed under reduced pressure and the residue purified by column chromatography (DCM / MeOH 20:1) to give **13** (0.107 g, 0.25 mmol, 80%).

$C_{27}H_{27}NO_4$, MW: 429.19 g/mol. **[α]** $_D^{23.0°C}$ (c = 0.95, $CHCl_3$) = –5.8. **^{1}H NMR** (300 MHz, $CDCl_3$, 21 °C): δ = 7.74-7.71 & 7.58-7.56 & 7.38-7.14 (*m*, 5+8 H, Ar-*H*), 5.18 (*bs*, 1 H, N*H*), 4.38 (*bs*, 2 H, OCH_2CH-Ar_2), 4.19 (*t, J* = 6.6, 1 H, OCH_2CH-Ar_2), 2.94 & 2.57 & 2.21 & 1.96 (*bs*, 1+3+1+1 H, CH_2CH_2 & CH_2COOH), 1.42 (*s*, 3 H, NCCH_3). **^{13}C NMR** (75 MHz, $CDCl_3$, 21 °C): δ = 176.6, 155.0, 144.0, 141.7, 141.5, 128.6, 127.8, 126.7, 126.1, 120.1, 66.4, 54.0, 47.4, 42.9, 41.1, 30.2, 23.8. **IR (film):** ν = 3026, 1711, 1512, 1451, 1338, 1240, 1115, 1085. **MS (MALDI) *m/z*:** 452.2 [100%, $(MNa)^+$]. **HRMS (MALDI) *m/z*:** Calc. for [$(MNa)^+$]: 452.1832. Found:452.1835. A sufficient microanalysis could not be obtained due to solvent inclusion.

References

1 Silver trifluoroacetate may also be stored under nitrogen over phosphorous pentoxide.

2 Ethyl-2-butynoate is commercially available from various suppliers, though at a relatively high price.

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[7] For comparison, a sample with low *E*:*Z*-ratio was prepared by quenching the cuprate complex at RT.

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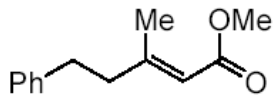
STANDARD 1H OBSERVE

Sample directory: df-317

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-317_ester_1H
INOVA-500 "nmroc"

Pulse 29.5 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
16 repetitions
OBSERVE H1, 300.0836333 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 52 sec



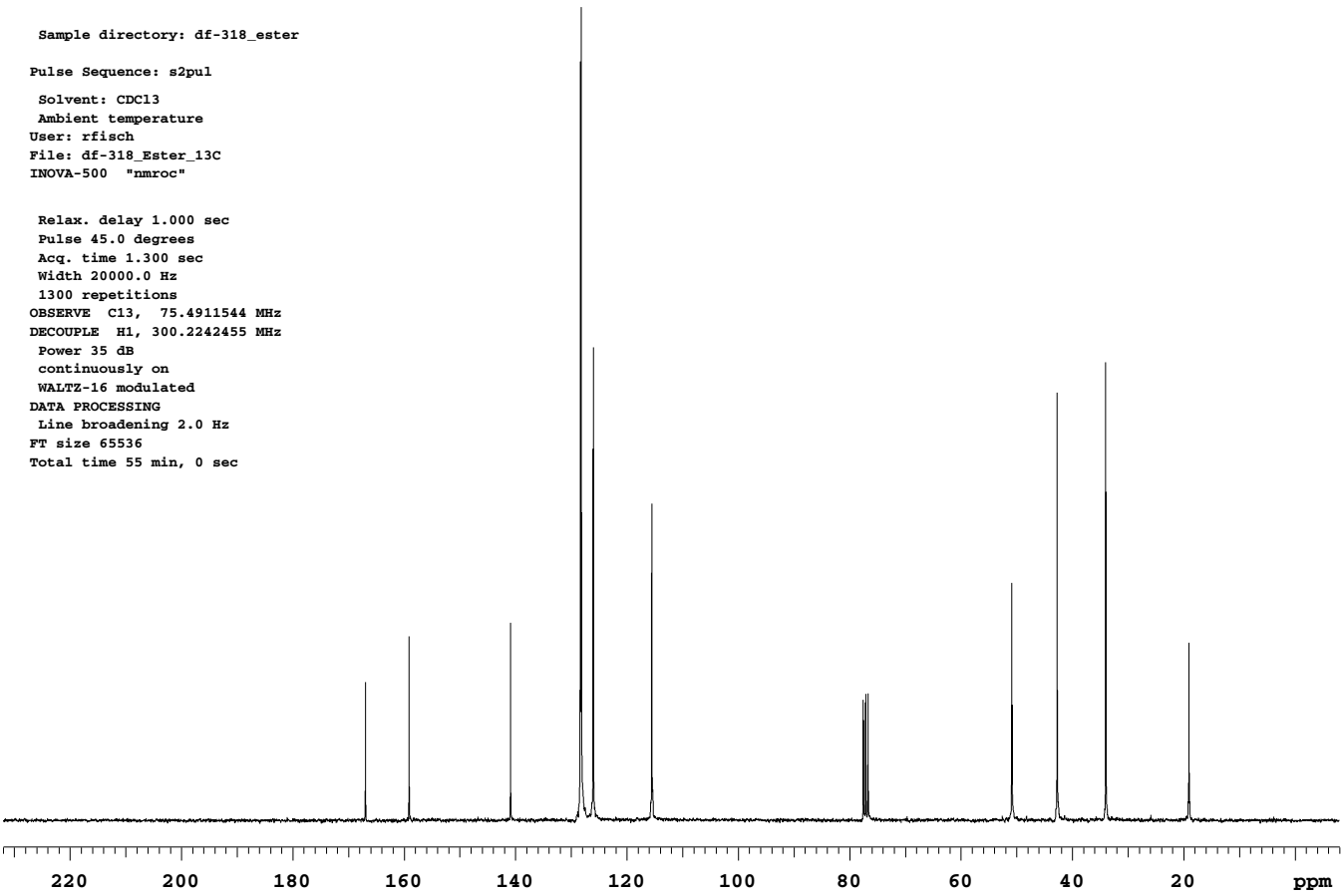
13C OBSERVE

Sample directory: df-318_ester

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-318_Ester_13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1300 repetitions
OBSERVE C13, 75.4911544 MHz
DECOUPLE H1, 300.2242455 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 55 min, 0 sec



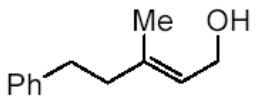
STANDARD 1H OBSERVE

Sample directory: df-317_OH

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-317_OH_1H
INOVA-500 "nmroc"

Pulse 29.5 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
16 repetitions
OBSERVE H1, 300.0836333 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 52 sec



2a

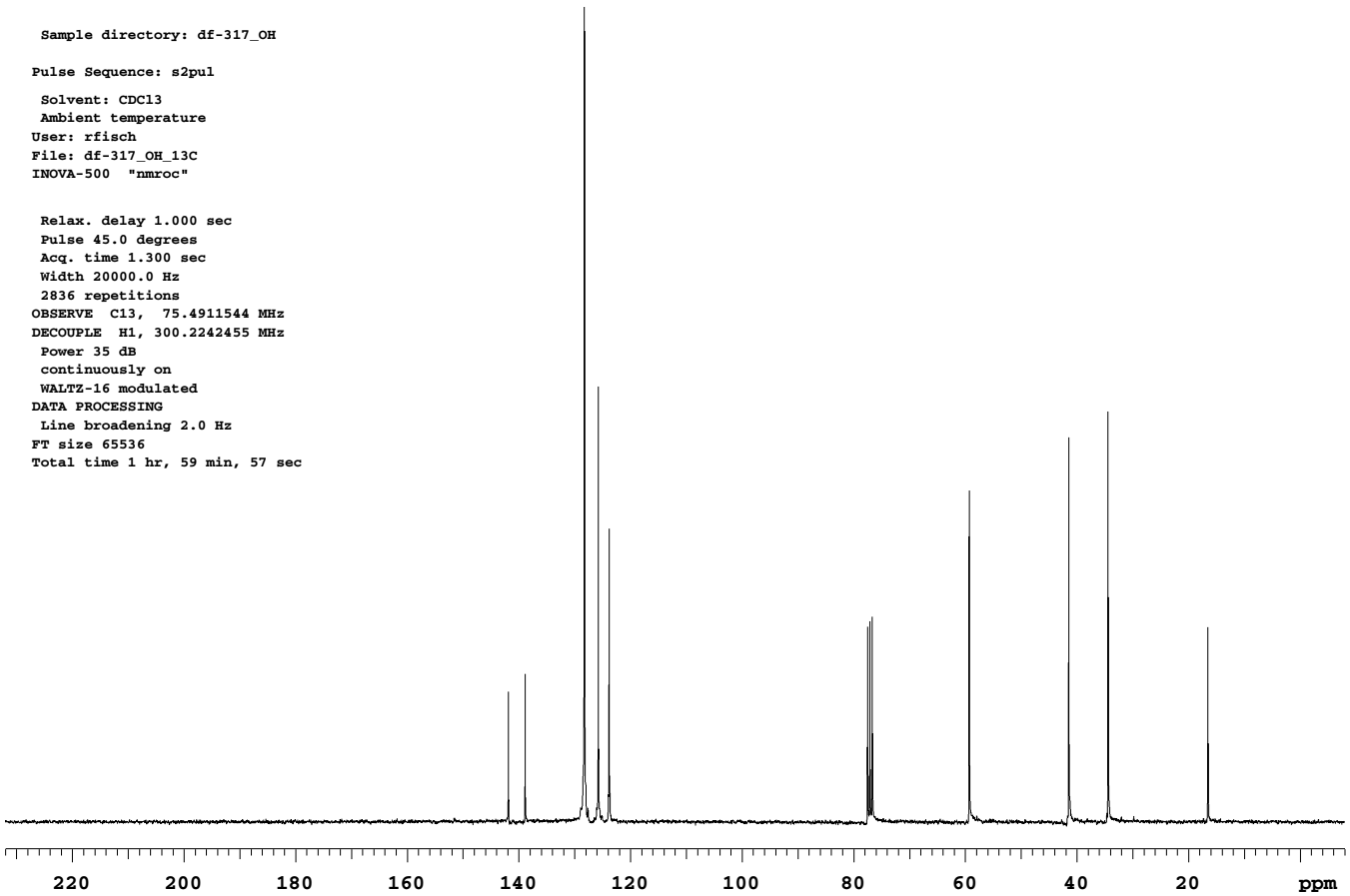
13C OBSERVE

Sample directory: df-317_OH

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-317_OH_13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
2836 repetitions
OBSERVE C13, 75.4911544 MHz
DECOUPLE H1, 300.2242455 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 1 hr, 59 min, 57 sec



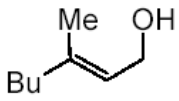
STANDARD 1H OBSERVE

Sample directory: df-338_OH

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-338_OH_1H
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 30.2 degrees
Acq. time 3.138 sec
Width 4500.5 Hz
16 repetitions
OBSERVE H1, 299.7729174 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 8 sec



2b

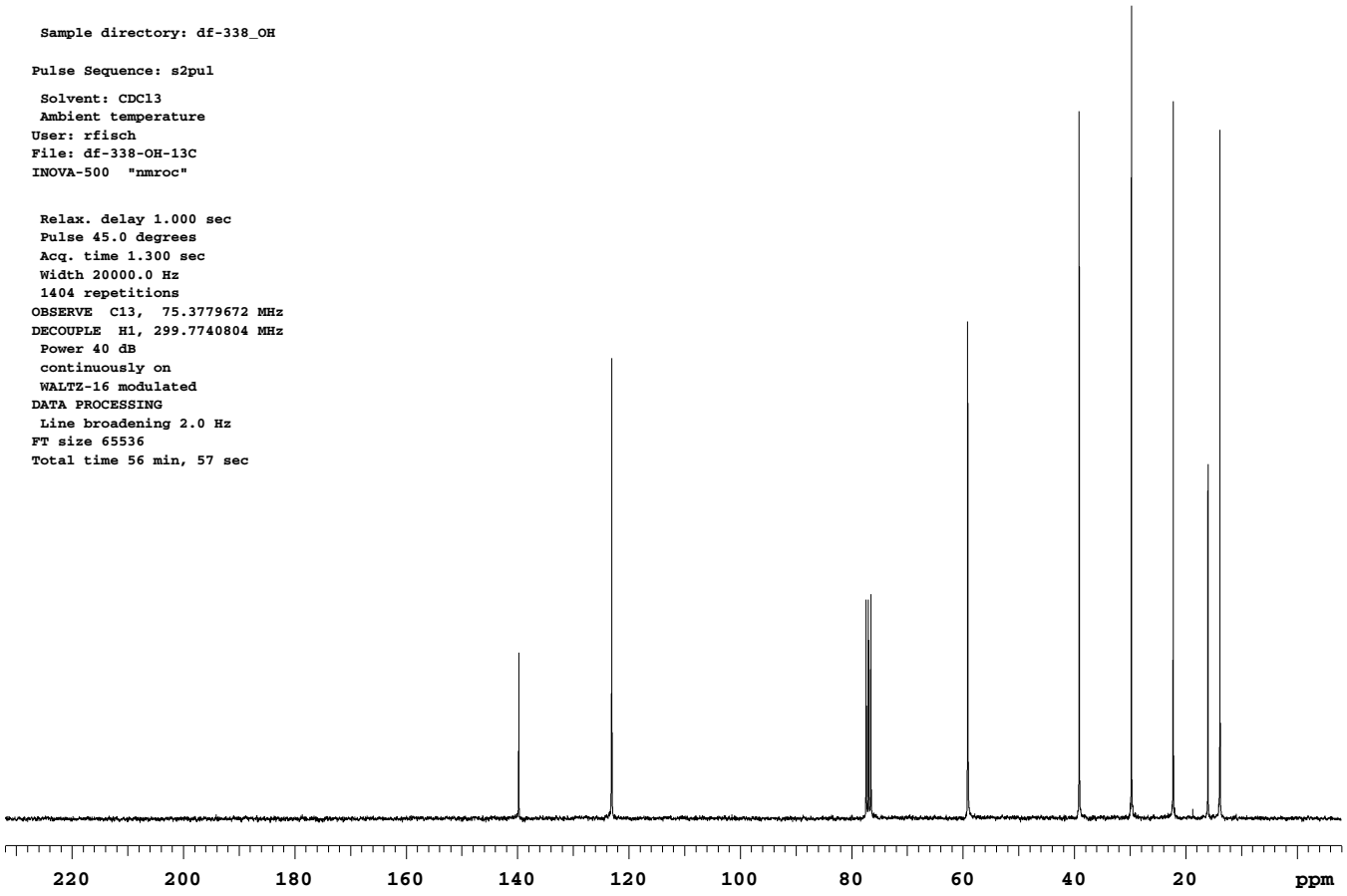
13C OBSERVE

Sample directory: df-338_OH

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-338-OH-13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1404 repetitions
OBSERVE C13, 75.3779672 MHz
DECOUPLE H1, 299.7740804 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 56 min, 57 sec



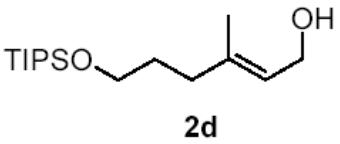
STANDARD 1H OBSERVE

Sample directory: df-300_OH

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-300_OH_1H
INOVA-500 "nmroc"

Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
16 repetitions
OBSERVE H1, 299.9012510 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 52 sec



2d

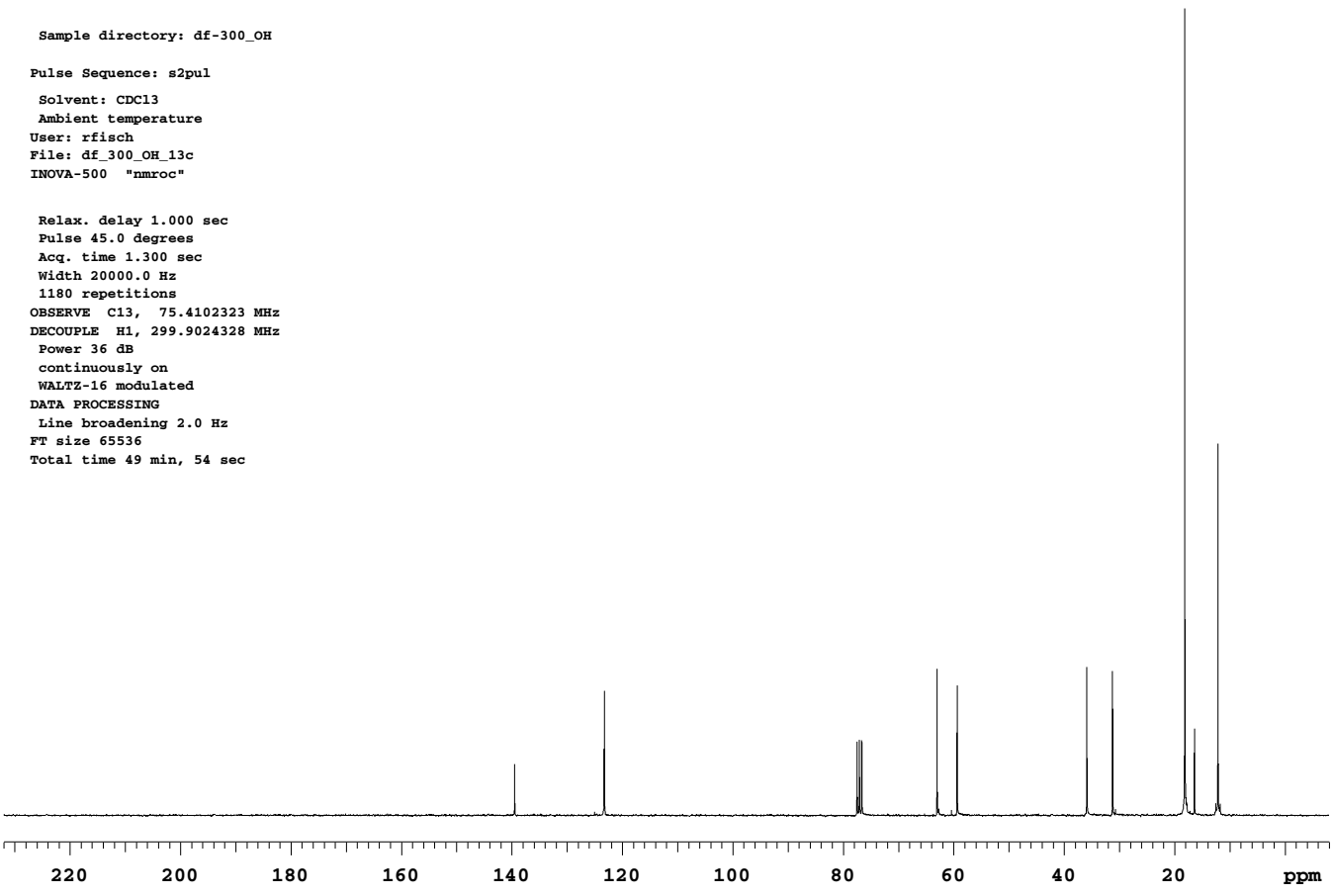
13C OBSERVE

Sample directory: df-300_OH

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df_300_OH_13c
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1180 repetitions
OBSERVE C13, 75.4102323 MHz
DECOUPLE H1, 299.9024328 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 49 min, 54 sec



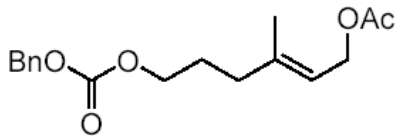
STANDARD 1H OBSERVE

Sample directory: X341PPF-19

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X341PP-F19
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 56.2 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
56 repetitions
OBSERVE H1, 299.7729174 MHz
DATA PROCESSING
FT size 32768
Total time 2 min, 54 sec



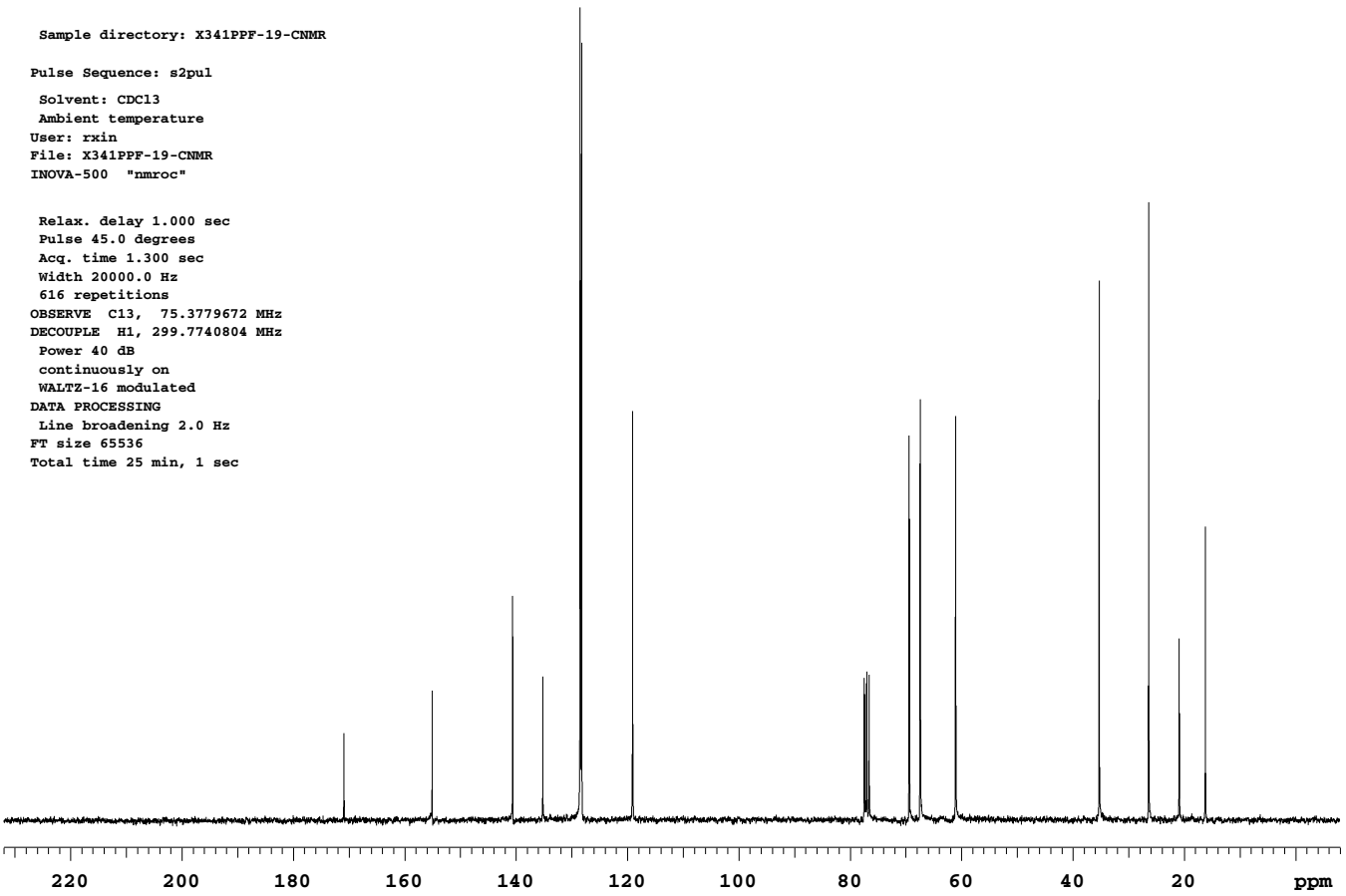
13C OBSERVE

Sample directory: X341PPF-19-CNMR

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X341PPF-19-CNMR
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
616 repetitions
OBSERVE C13, 75.3779672 MHz
DECOUPLE H1, 299.7740804 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 25 min, 1 sec



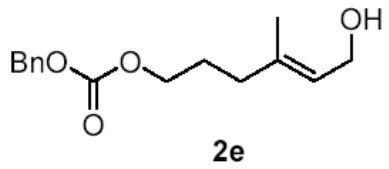
STANDARD 1H OBSERVE

Sample directory: X347PP

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X347PP
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 56.2 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
32 repetitions
OBSERVE H1, 299.7729174 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 39 sec



2e

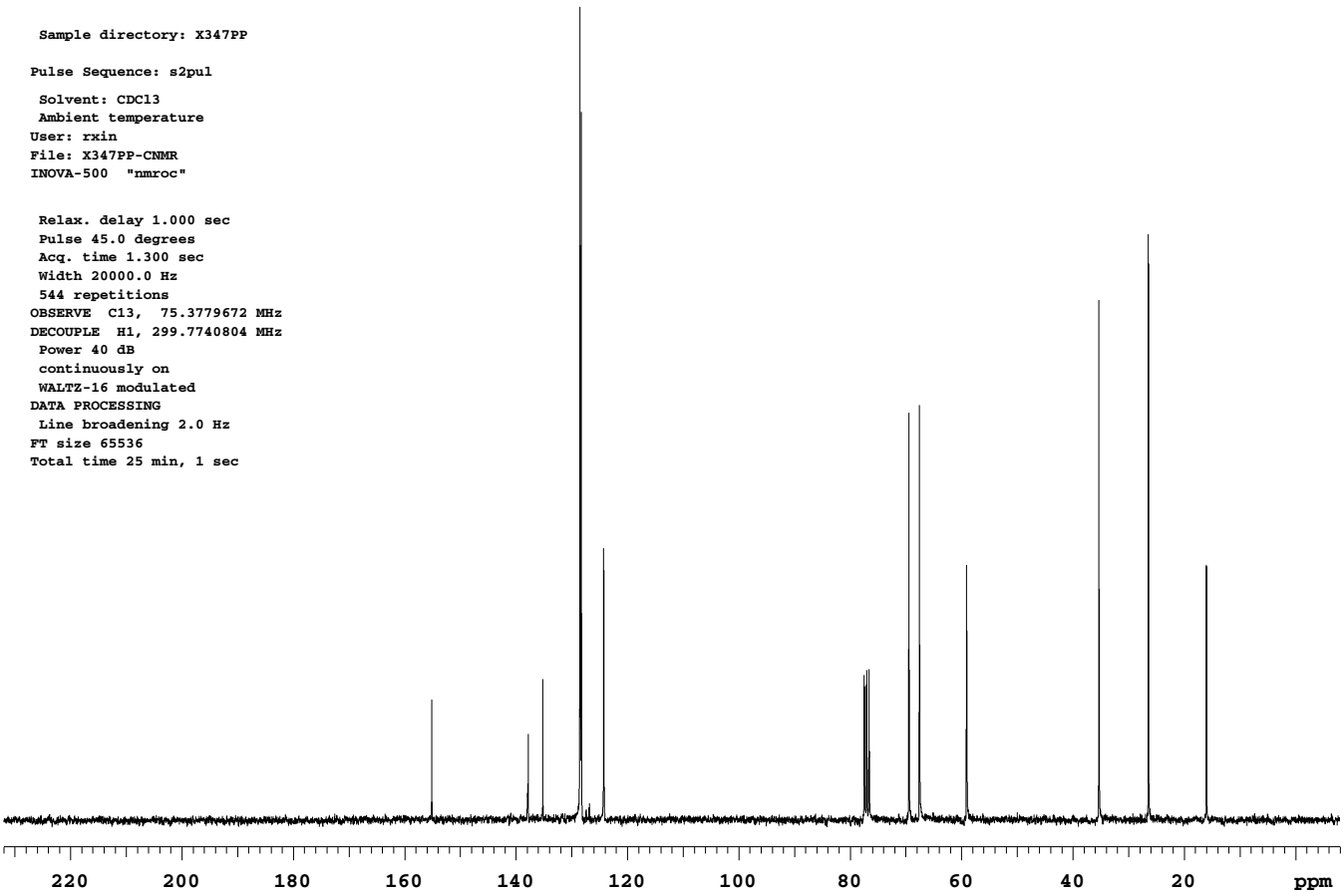
13C OBSERVE

Sample directory: X347PP

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X347PP-CNMR
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
544 repetitions
OBSERVE C13, 75.3779672 MHz
DECOUPLE H1, 299.7740804 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 25 min, 1 sec



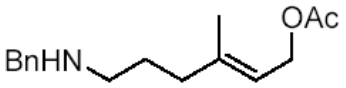
STANDARD 1H OBSERVE

Sample directory: X349PPF-28

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X349PPF-28
INOVA-500 "nmroc"

Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
88 repetitions
OBSERVE H1, 299.9012510 MHz
DATA PROCESSING
FT size 32768
Total time 4 min, 51 sec



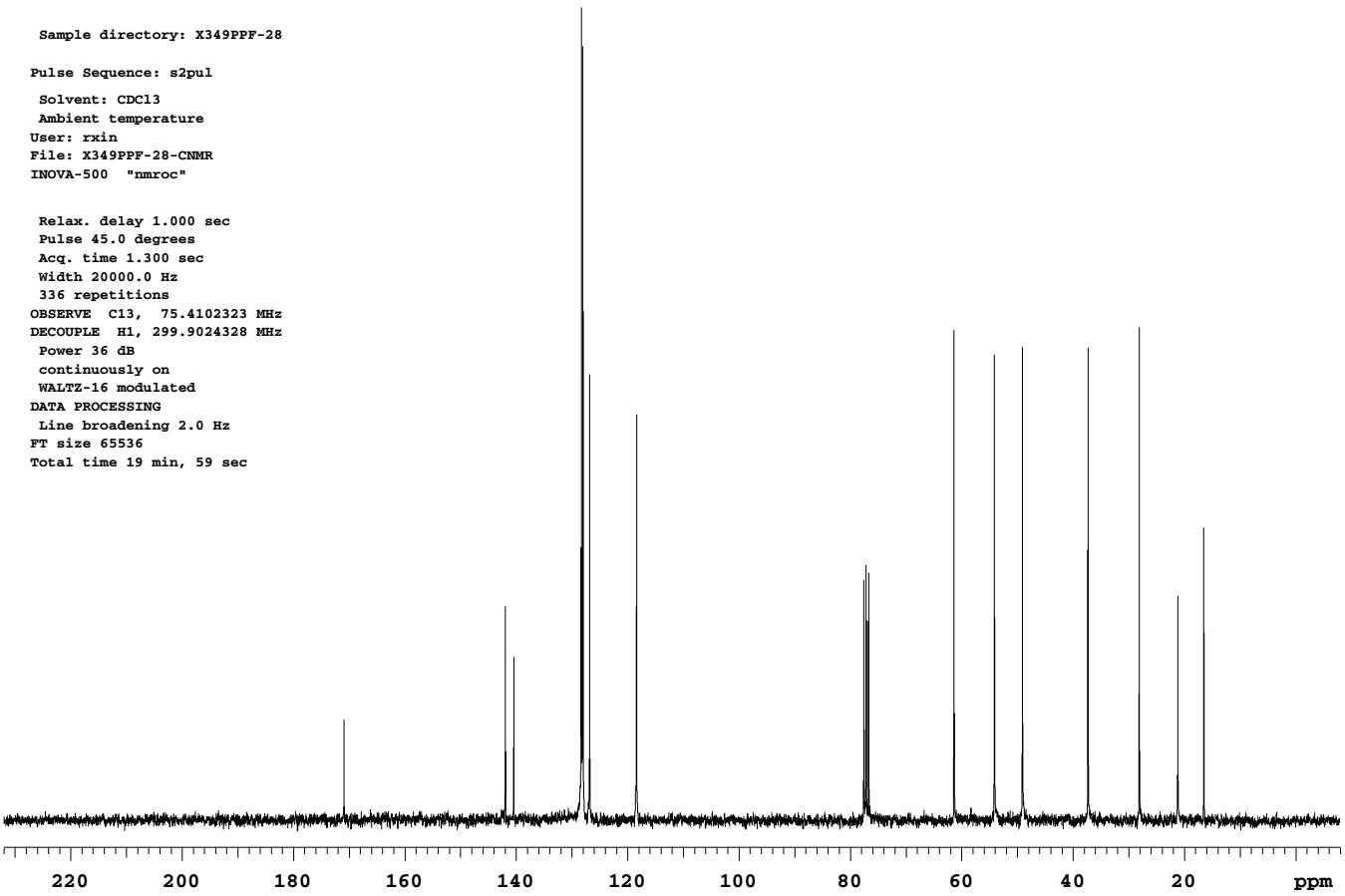
13C OBSERVE

Sample directory: X349PPF-28

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X349PPF-28-CNMR
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
336 repetitions
OBSERVE C13, 75.4102323 MHz
DECOUPLE H1, 299.9024328 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 19 min, 59 sec



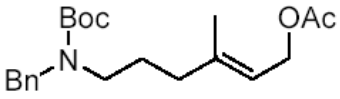
STANDARD 1H OBSERVE

Sample directory: X352PPF-15

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X352PPF-15
INOVA-500 "nmroc"

Pulse 29.5 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
88 repetitions
OBSERVE H1, 300.0836333 MHz
DATA PROCESSING
FT size 32768
Total time 4 min, 51 sec



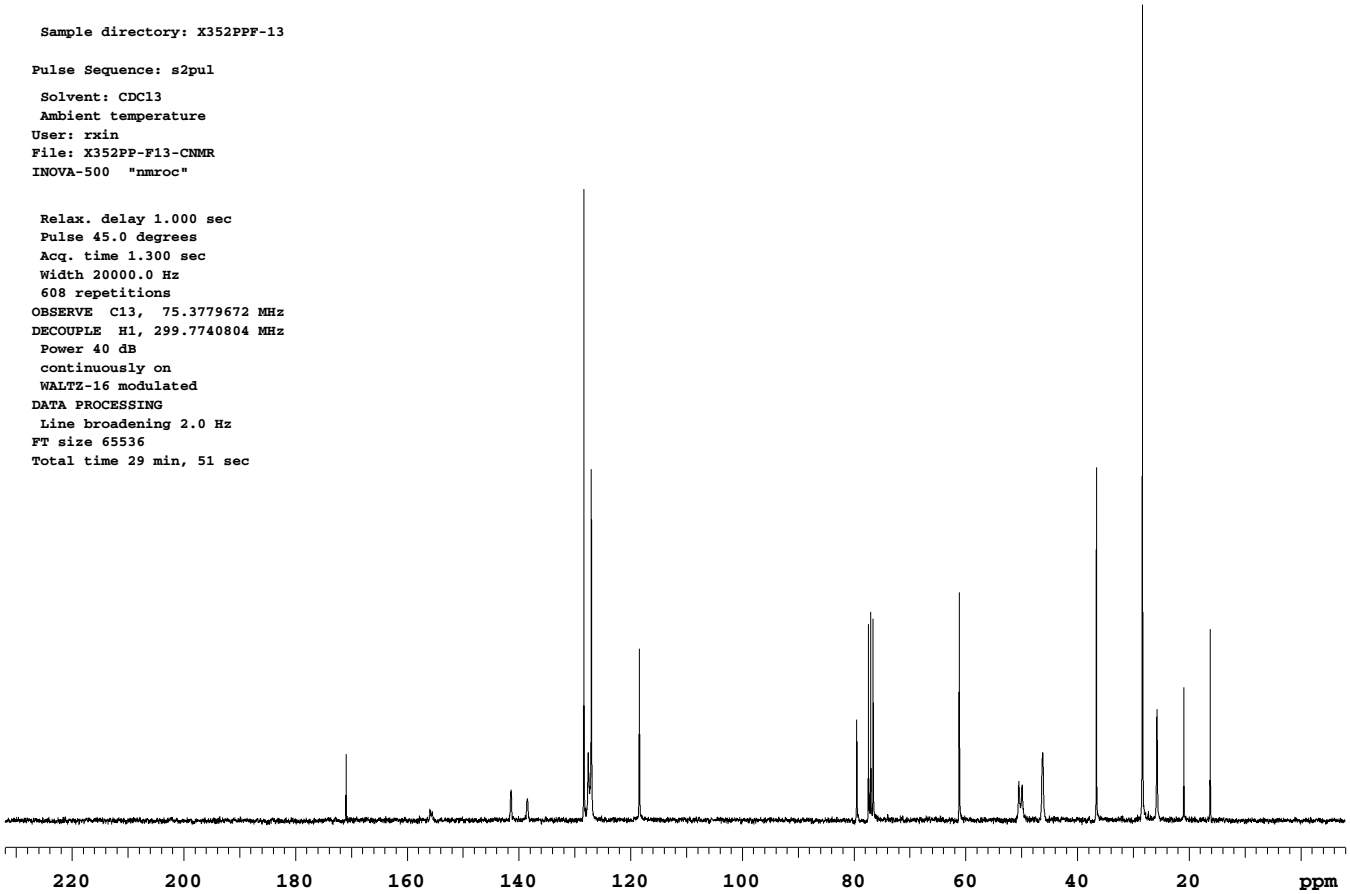
13C OBSERVE

Sample directory: X352PPF-13

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X352PP-F13-CNMR
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
608 repetitions
OBSERVE C13, 75.3779672 MHz
DECOUPLE H1, 299.7740804 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 29 min, 51 sec



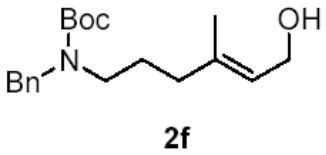
STANDARD 1H OBSERVE

Sample directory: X353PPF-10

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X353PPF-10
INOVA-500 "nmroc"

Pulse 29.5 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
88 repetitions
OBSERVE H1, 300.0836333 MHz
DATA PROCESSING
FT size 32768
Total time 4 min, 51 sec



2f

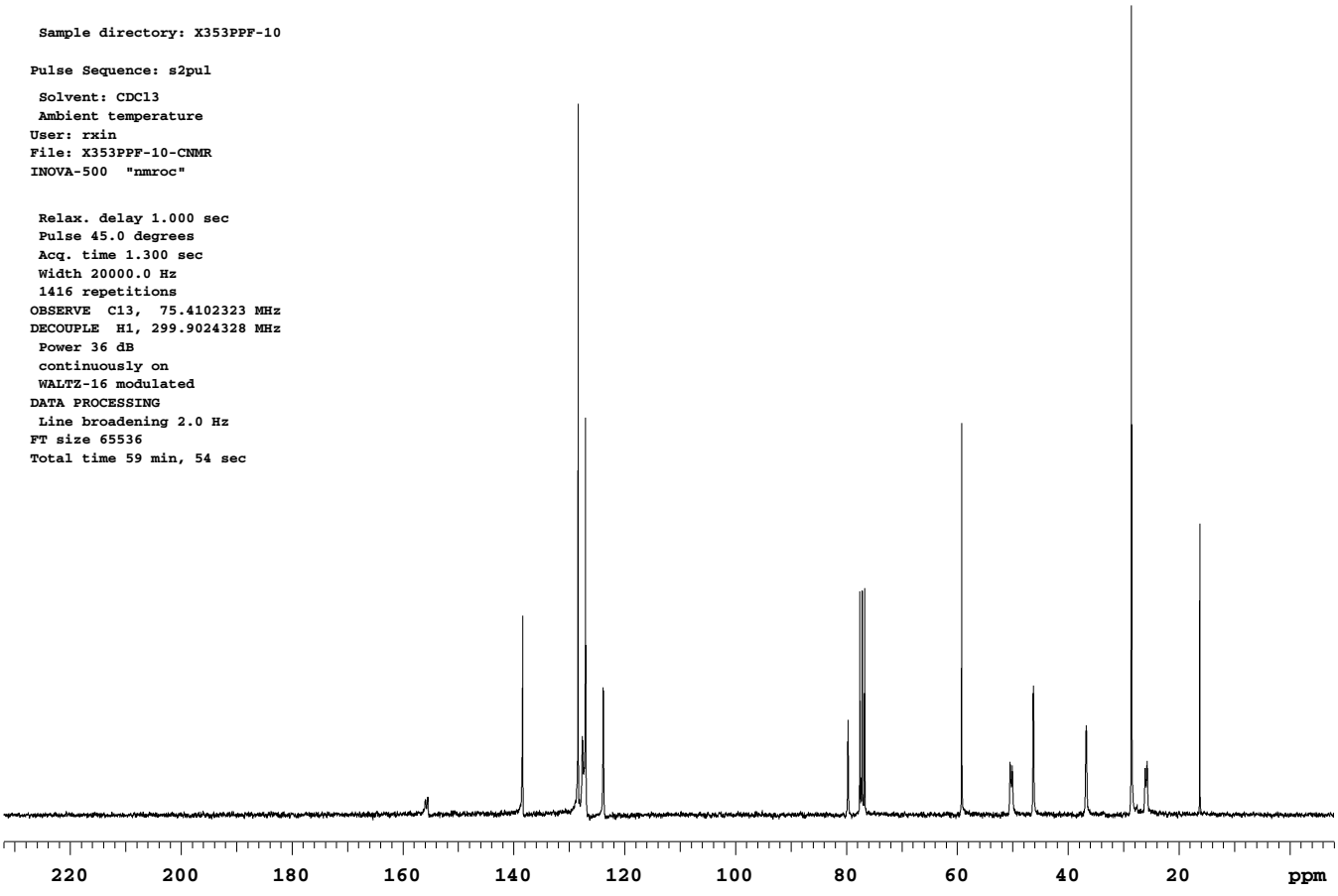
13C OBSERVE

Sample directory: X353PPF-10

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X353PPF-10-CNMR
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1416 repetitions
OBSERVE C13, 75.4102323 MHz
DECOUPLE H1, 299.9024328 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 59 min, 54 sec



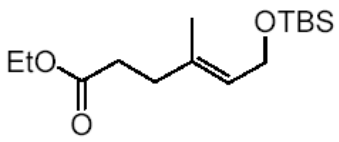
STANDARD 1H OBSERVE

Sample directory: X262PP

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X262PP
INOVA-500 "nmroc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
32 repetitions
OBSERVE H1, 300.2230602 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 45 sec



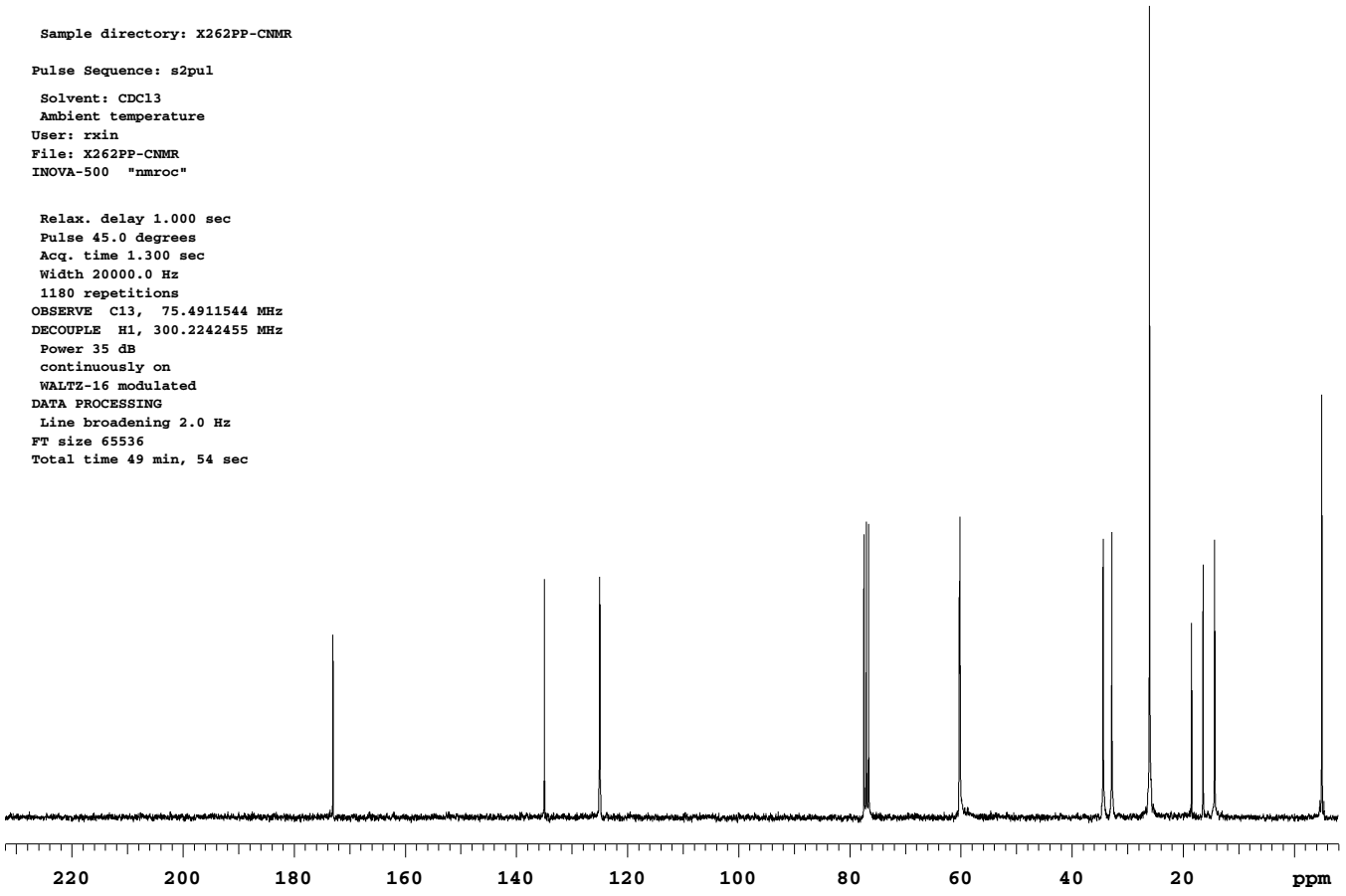
13C OBSERVE

Sample directory: X262PP-CNMR

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X262PP-CNMR
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1180 repetitions
OBSERVE C13, 75.4911544 MHz
DECOUPLE H1, 300.2242455 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 49 min, 54 sec



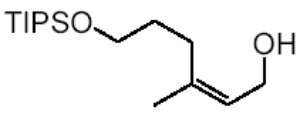
STANDARD 1H OBSERVE

Sample directory: df-314_OH

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-314_OH_1H
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 30.2 degrees
Acq. time 3.138 sec
Width 4500.5 Hz
16 repetitions
OBSERVE H1, 299.7729174 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 8 sec



2h

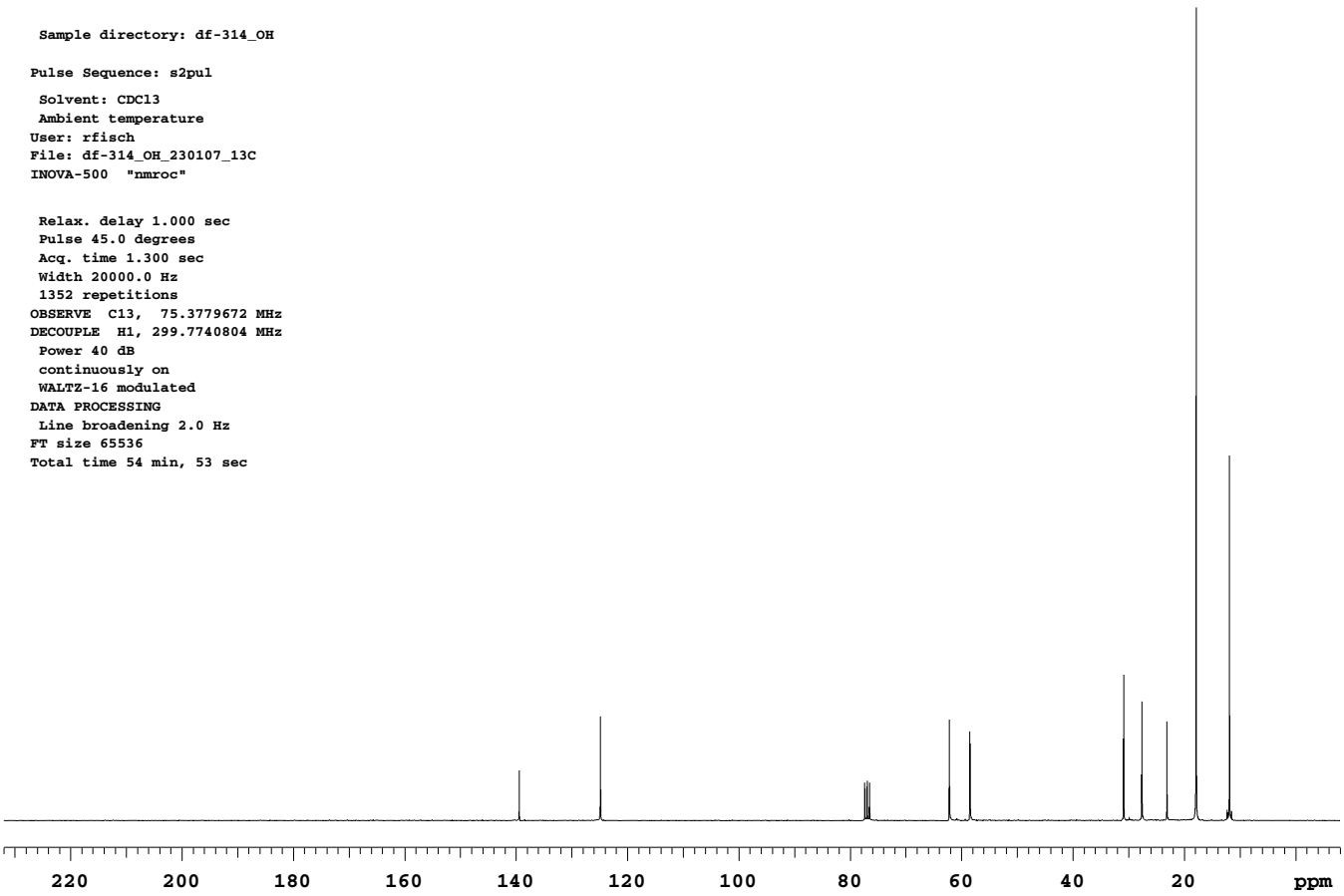
13C OBSERVE

Sample directory: df-314_OH

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-314_OH_230107_13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1352 repetitions
OBSERVE C13, 75.3779672 MHz
DECOUPLE H1, 299.7740804 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 54 min, 53 sec



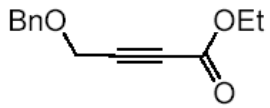
STANDARD 1H OBSERVE

Sample directory: df-334_ester

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-334_ester_1H
INOVA-500 "nmroc"

Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
8 repetitions
OBSERVE H1, 299.9012510 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 27 sec



6c

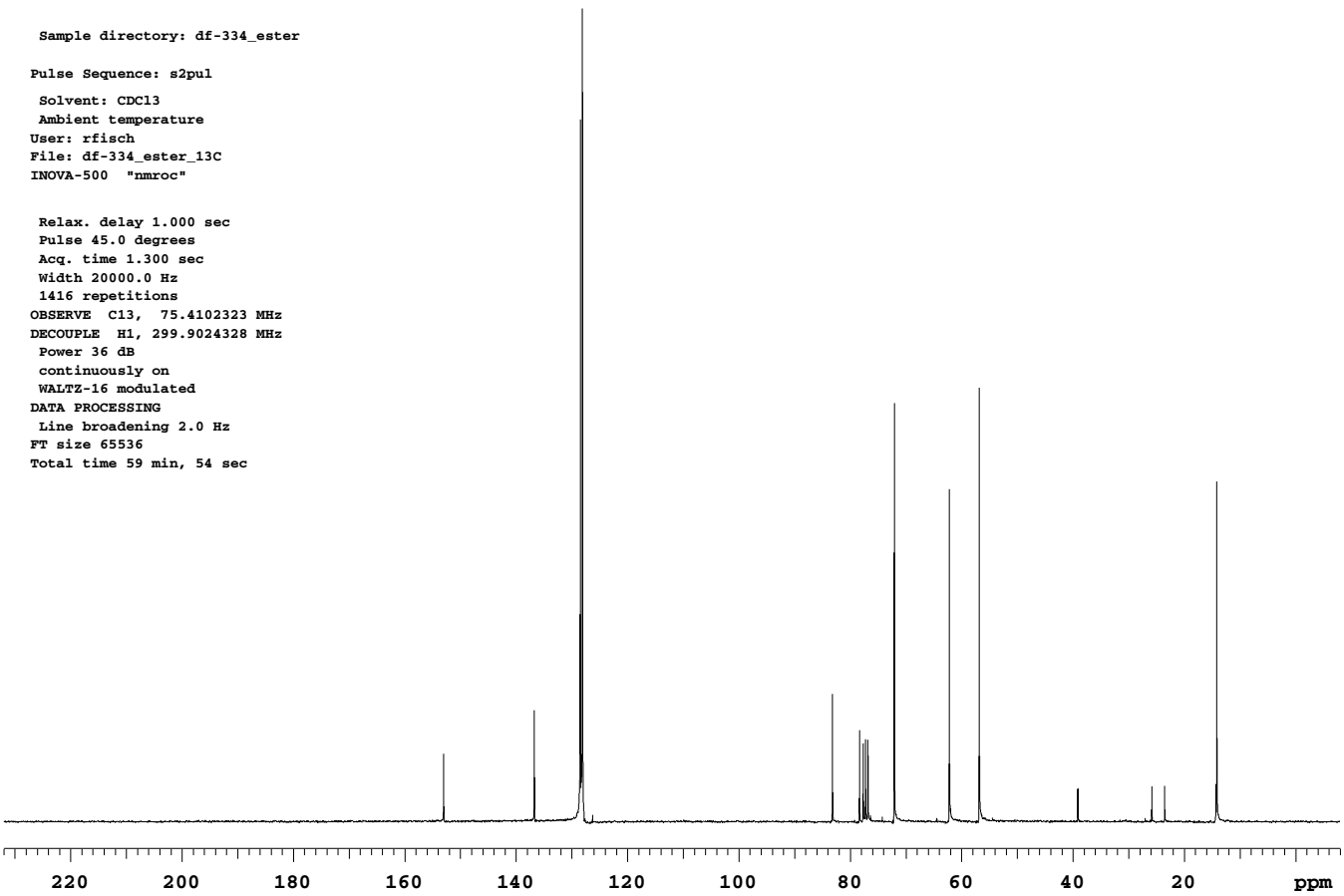
13C OBSERVE

Sample directory: df-334_ester

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-334_ester_13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1416 repetitions
OBSERVE C13, 75.4102323 MHz
DECOUPLE H1, 299.9024328 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 59 min, 54 sec



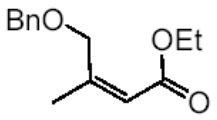
STANDARD 1H OBSERVE

Sample directory: X361PP2-F-13

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X361PP2-F-13
INOVA-500 "nmroc"

Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
52 repetitions
OBSERVE H1, 299.9012510 MHz
DATA PROCESSING
FT size 32768
Total time 2 min, 53 sec



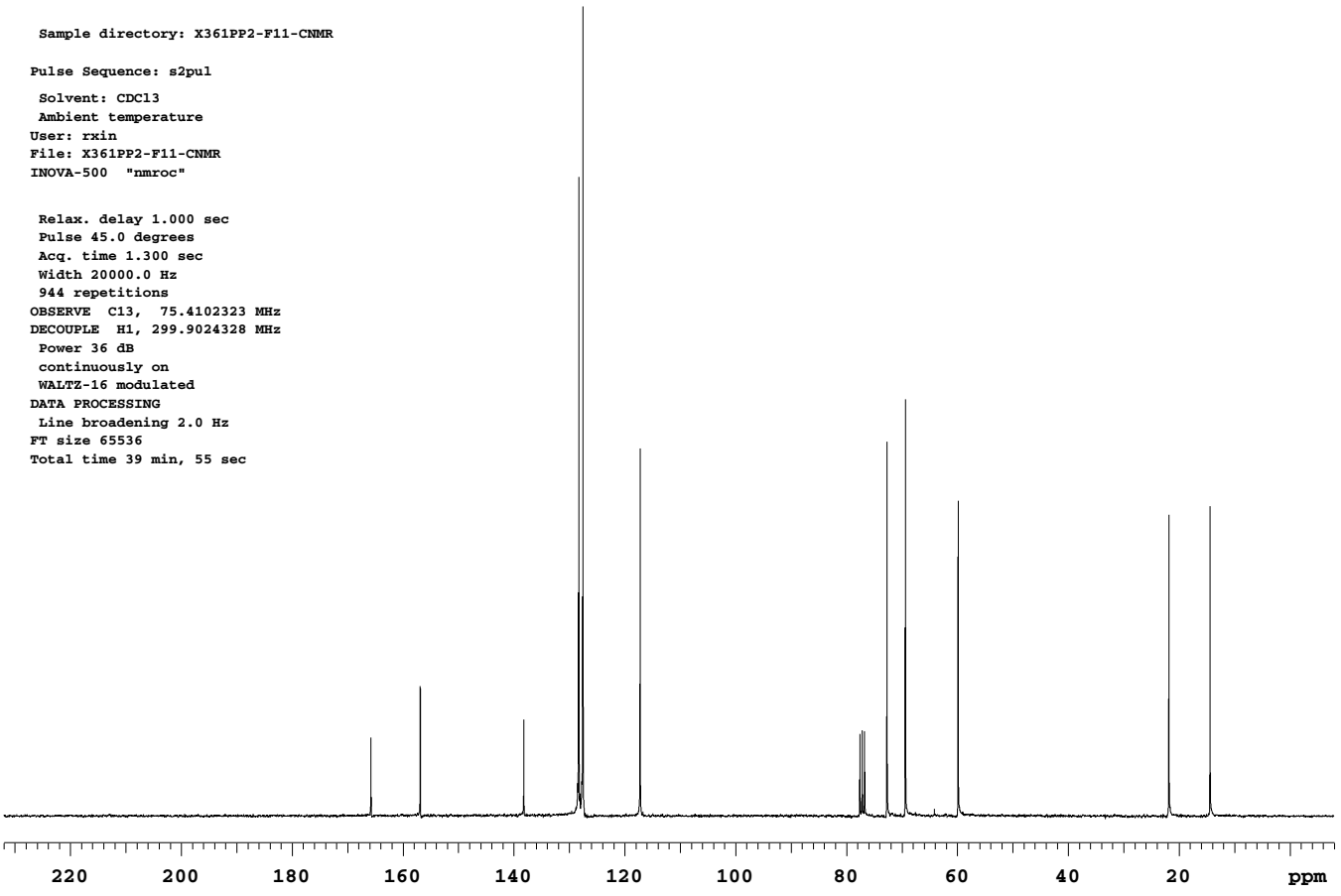
13C OBSERVE

Sample directory: X361PP2-F11-CNMR

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X361PP2-F11-CNMR
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
944 repetitions
OBSERVE C13, 75.4102323 MHz
DECOUPLE H1, 299.9024328 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 39 min, 55 sec



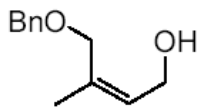
STANDARD 1H OBSERVE

Sample directory: X362PPF-20

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X362PPF-20
INOVA-500 "nmroc"

Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
52 repetitions
OBSERVE H1, 299.9012510 MHz
DATA PROCESSING
FT size 32768
Total time 2 min, 53 sec



2i

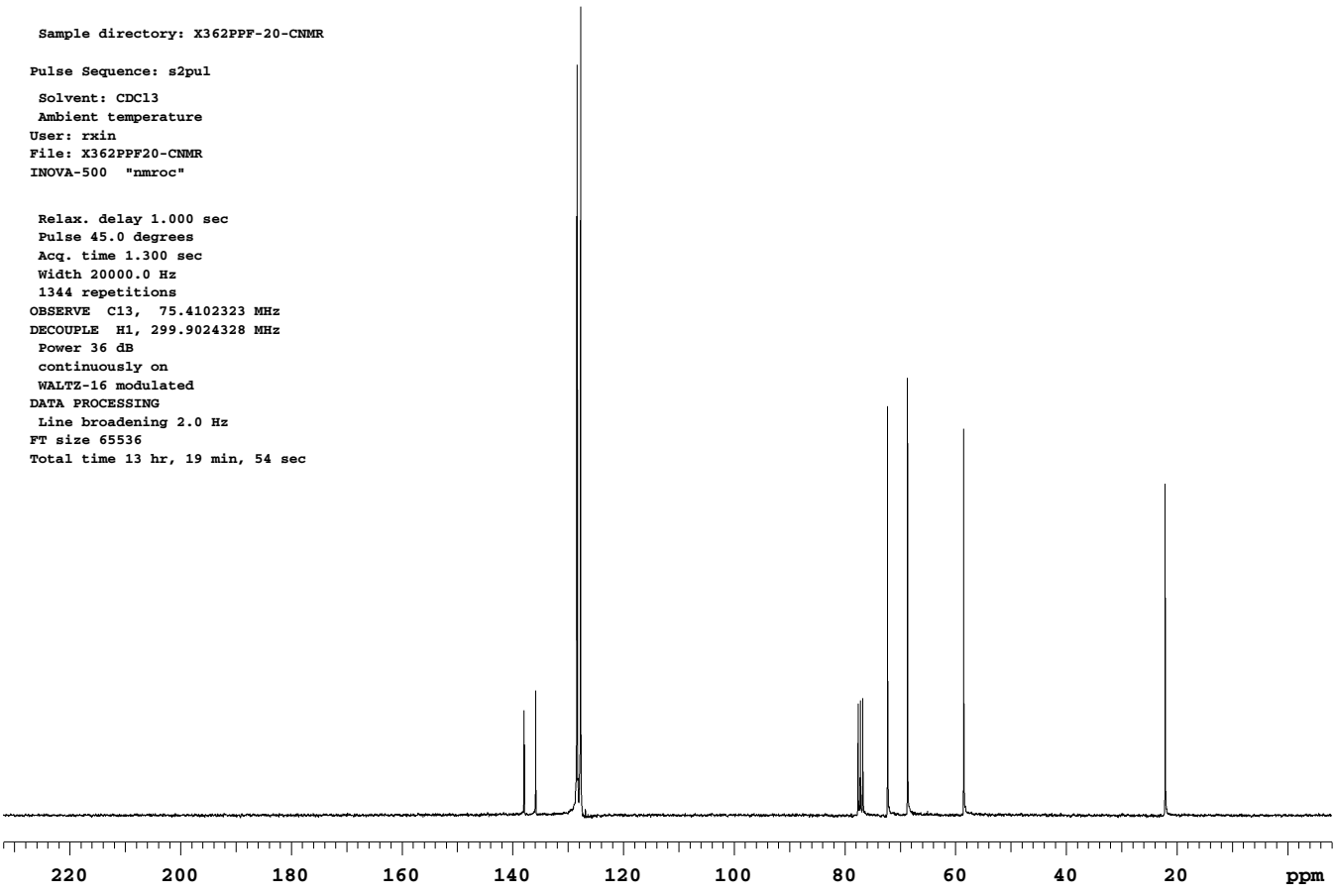
13C OBSERVE

Sample directory: X362PPF-20-CNMR

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X362PPF20-CNMR
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1344 repetitions
OBSERVE C13, 75.4102323 MHz
DECOUPLE H1, 299.9024328 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 13 hr, 19 min, 54 sec



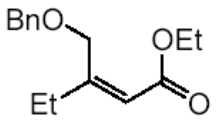
STANDARD 1H OBSERVE

Sample directory: df-340_ester

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-340_ester_1H
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 30.2 degrees
Acq. time 3.138 sec
Width 4500.5 Hz
8 repetitions
OBSERVE H1, 299.7729174 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 35 sec



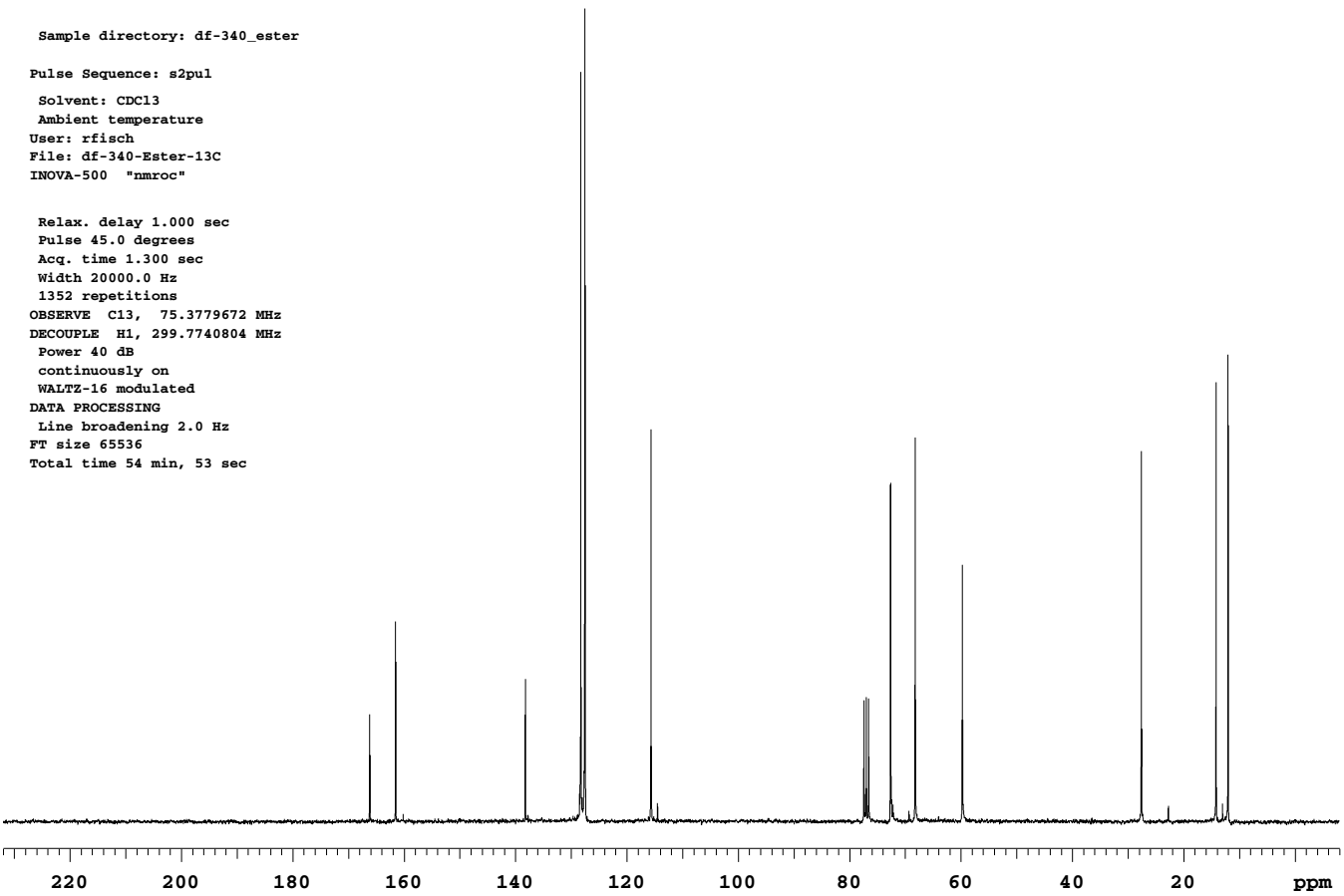
13C OBSERVE

Sample directory: df-340_ester

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-340-Ester-13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1352 repetitions
OBSERVE C13, 75.3779672 MHz
DECOUPLE H1, 299.7740804 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 54 min, 53 sec



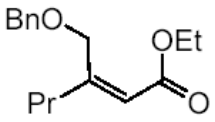
STANDARD 1H OBSERVE

Sample directory: svf('cn-372-3fr')df-339_ester

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-339_ester_1H
INOVA-500 "nmroc"

Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
8 repetitions
OBSERVE H1, 299.9012510 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 27 sec



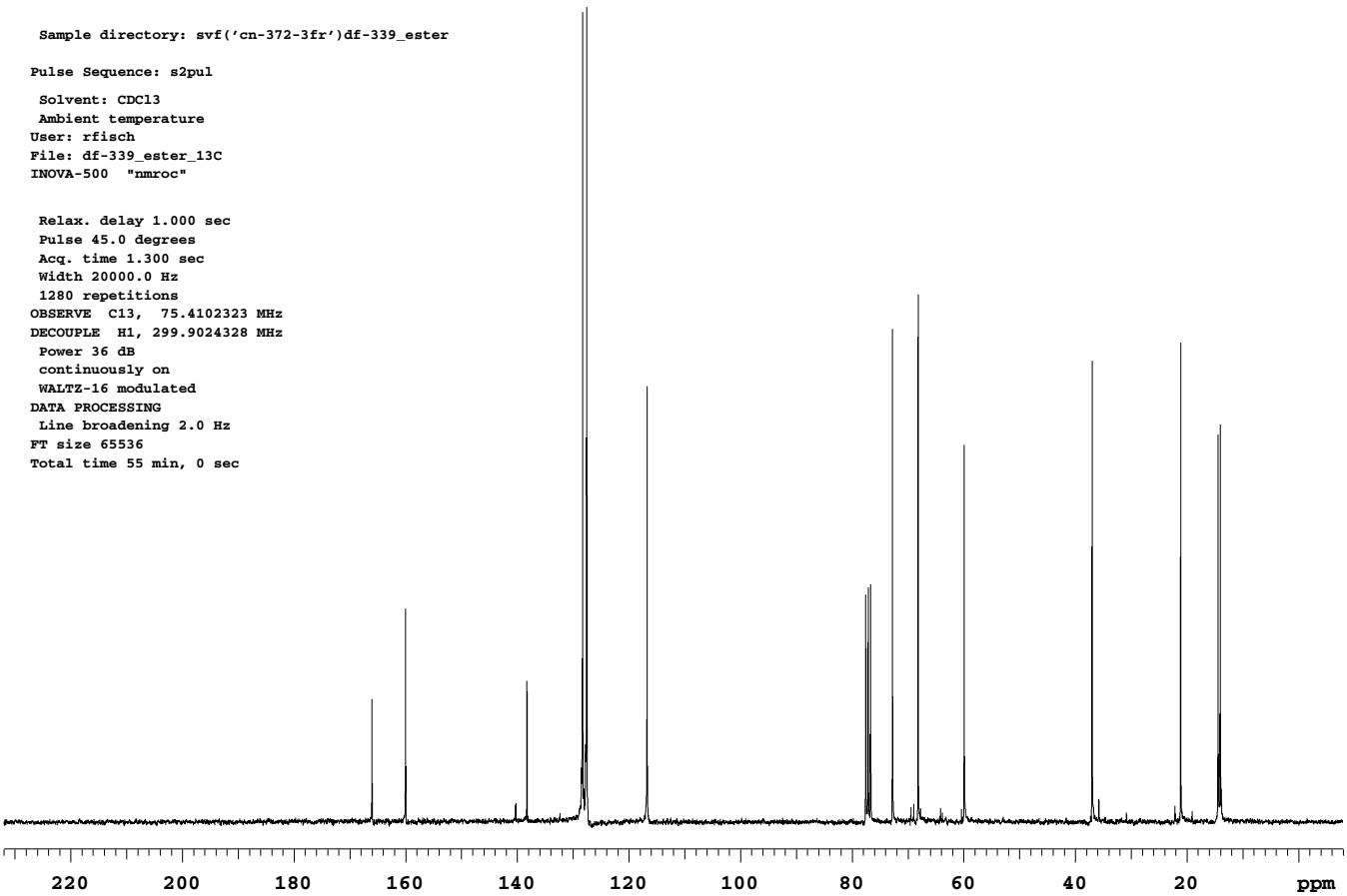
13C OBSERVE

Sample directory: svf('cn-372-3fr')df-339_ester

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-339_ester_13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1280 repetitions
OBSERVE C13, 75.4102323 MHz
DECOUPLE H1, 299.9024328 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 55 min, 0 sec



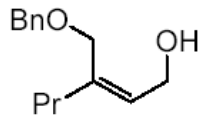
STANDARD 1H OBSERVE

Sample directory: df-339_OH

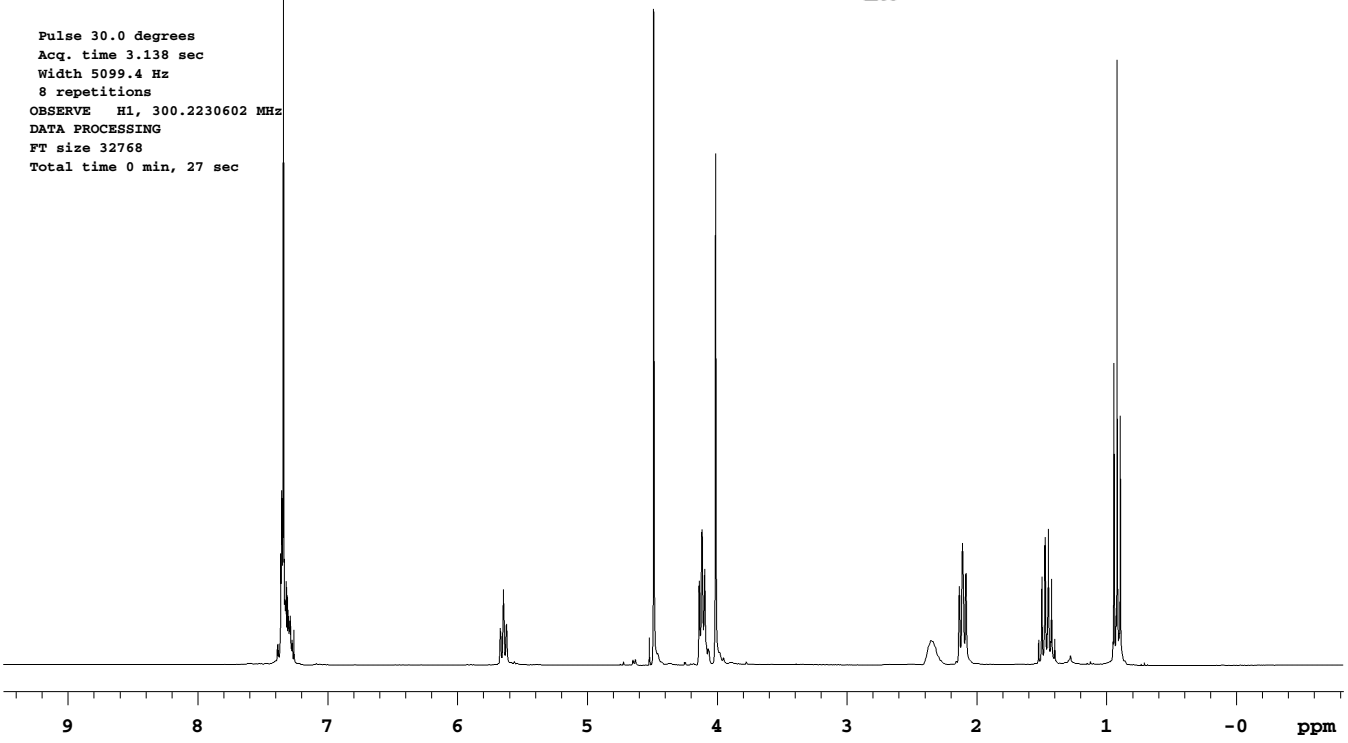
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-339_OH_1H
INOVA-500 "nmroc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
8 repetitions
OBSERVE H1, 300.2230602 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 27 sec



2k



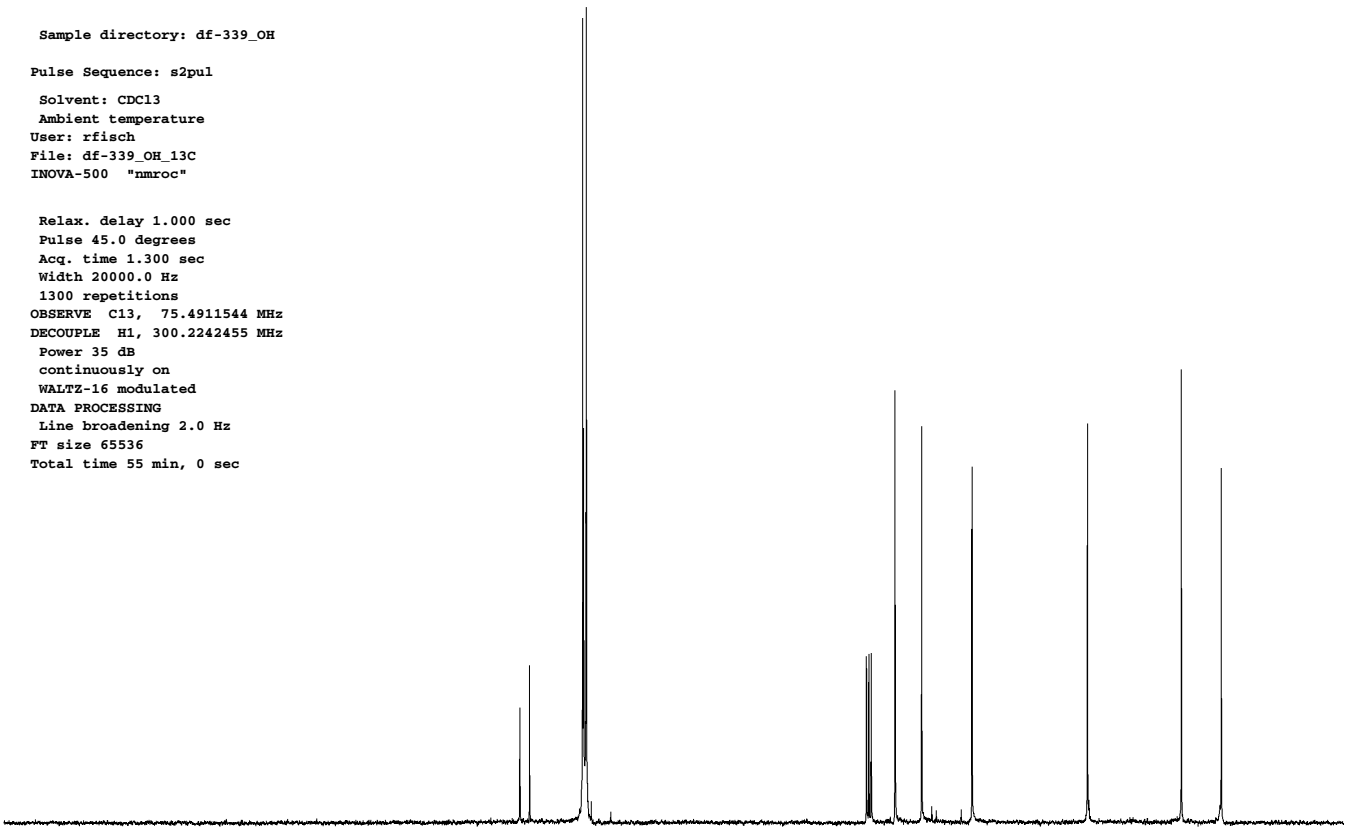
13C OBSERVE

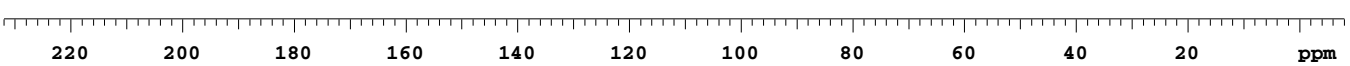
Sample directory: df-339_OH

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-339_OH_13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1300 repetitions
OBSERVE C13, 75.4911544 MHz
DECOUPLE H1, 300.2242455 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 55 min, 0 sec





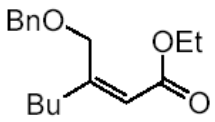
STANDARD 1H OBSERVE

Sample directory: df-337_ester

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-337_ester_1H
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 30.2 degrees
Acq. time 3.138 sec
Width 4500.5 Hz
8 repetitions
OBSERVE H1, 299.7729174 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 35 sec



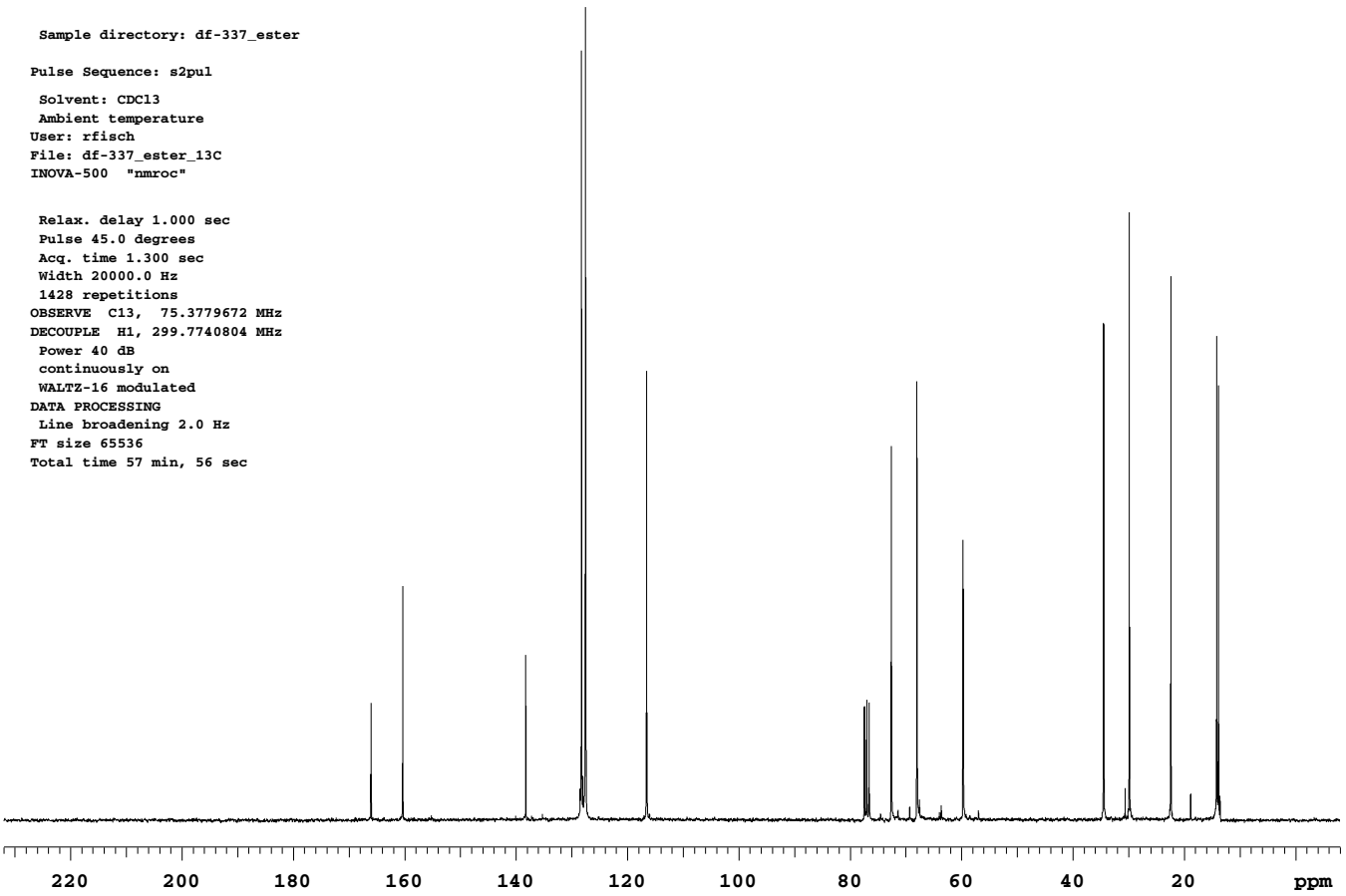
13C OBSERVE

Sample directory: df-337_ester

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-337_ester_13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1428 repetitions
OBSERVE C13, 75.3779672 MHz
DECOUPLE H1, 299.7740804 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 57 min, 56 sec



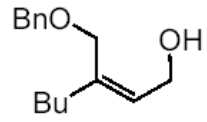
STANDARD 1H OBSERVE

Sample directory: df-337_OH

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-337_OH_1H
INOVA-500 "nmroc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
8 repetitions
OBSERVE H1, 300.2230602 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 27 sec



2l

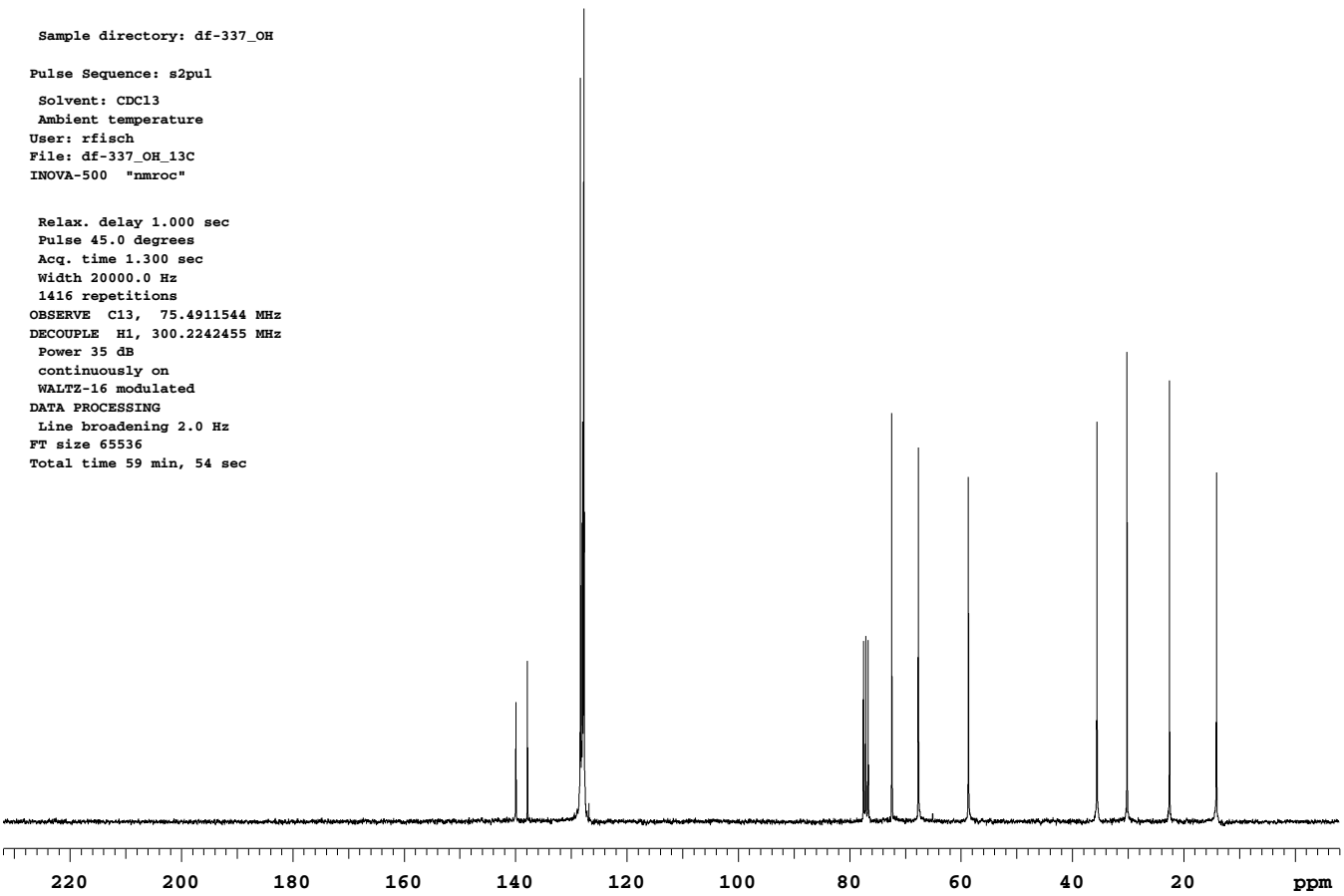
13C OBSERVE

Sample directory: df-337_OH

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-337_OH_13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1416 repetitions
OBSERVE C13, 75.4911544 MHz
DECOUPLE H1, 300.2242455 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 59 min, 54 sec

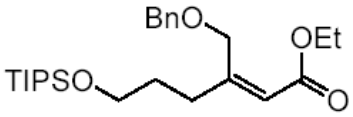


STANDARD 1H OBSERVE

Sample directory: df-341_ester

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-341_ester_1H
INOVA-500 "nmroc"



Relax. delay 1.000 sec
Pulse 30.2 degrees
Acq. time 3.138 sec
Width 4500.5 Hz
8 repetitions
OBSERVE H1, 299.7729174 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 35 sec

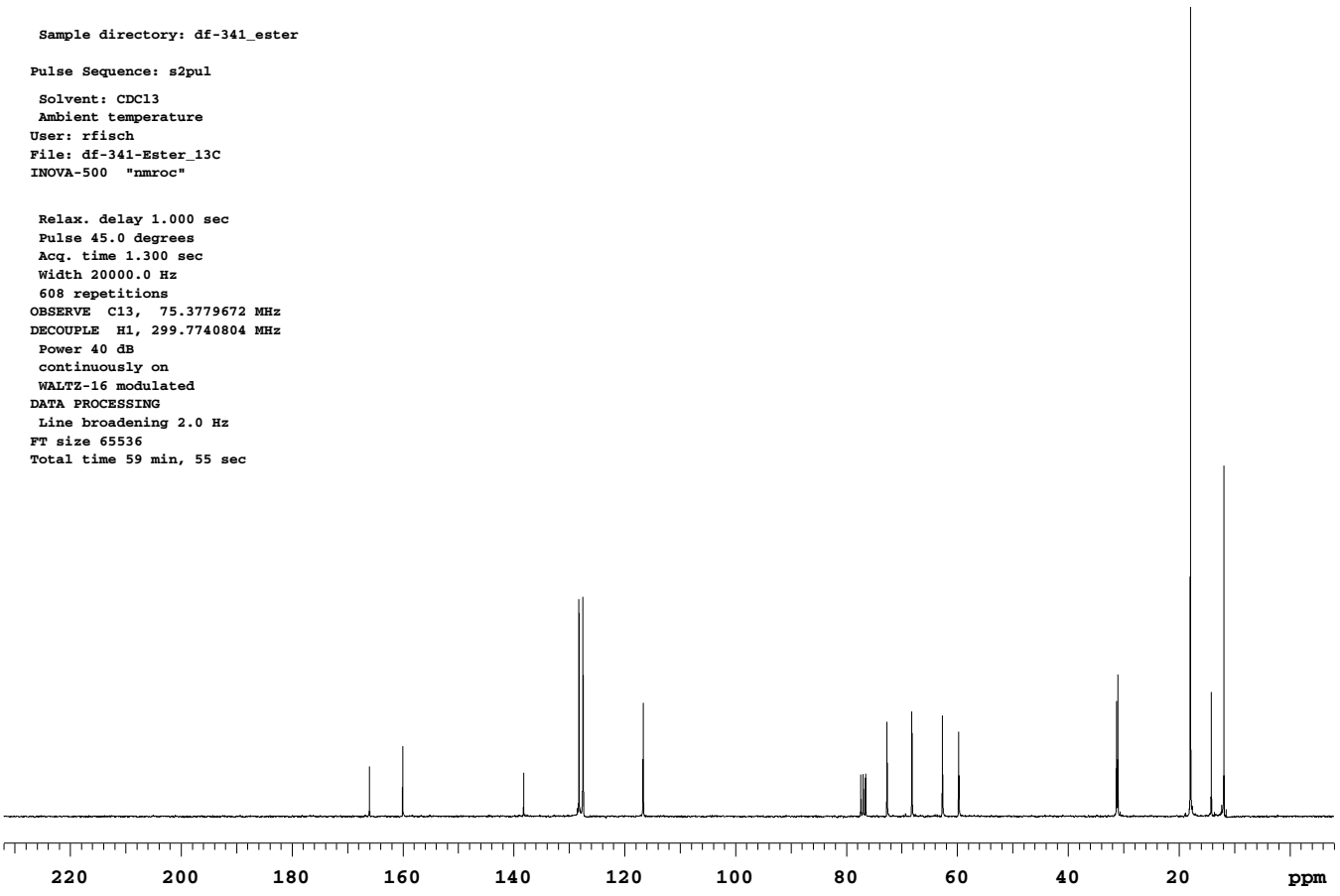
13C OBSERVE

Sample directory: df-341_ester

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-341-Ester_13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
608 repetitions
OBSERVE C13, 75.3779672 MHz
DECOUPLE H1, 299.7740804 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 59 min, 55 sec



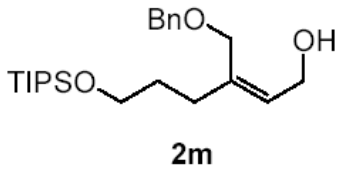
STANDARD 1H OBSERVE

Sample directory: df-341_OH

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-341_OH_1H
INOVA-500 "nmroc"

Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
8 repetitions
OBSERVE H1, 299.9012510 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 27 sec



2m

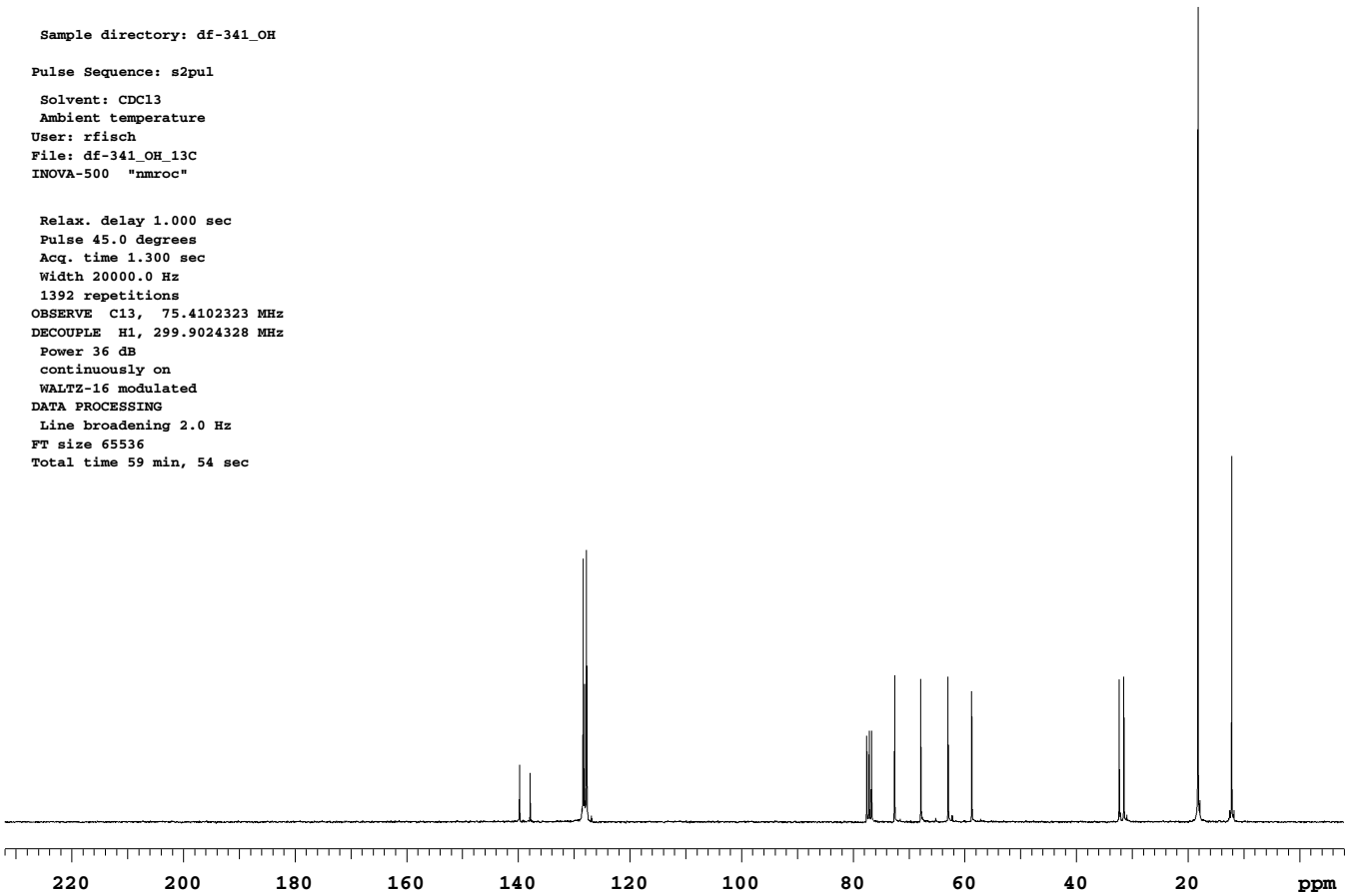
13C OBSERVE

Sample directory: df-341_OH

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-341_OH_13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
1392 repetitions
OBSERVE C13, 75.4102323 MHz
DECOUPLE H1, 299.9024328 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 59 min, 54 sec



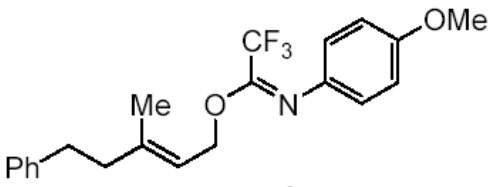
STANDARD 1H OBSERVE

Sample directory: df-336_imidat

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-336_imidat_160407_1H
INOVA-500 "nmroc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
16 repetitions
OBSERVE H1, 300.2230602 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 52 sec



4a

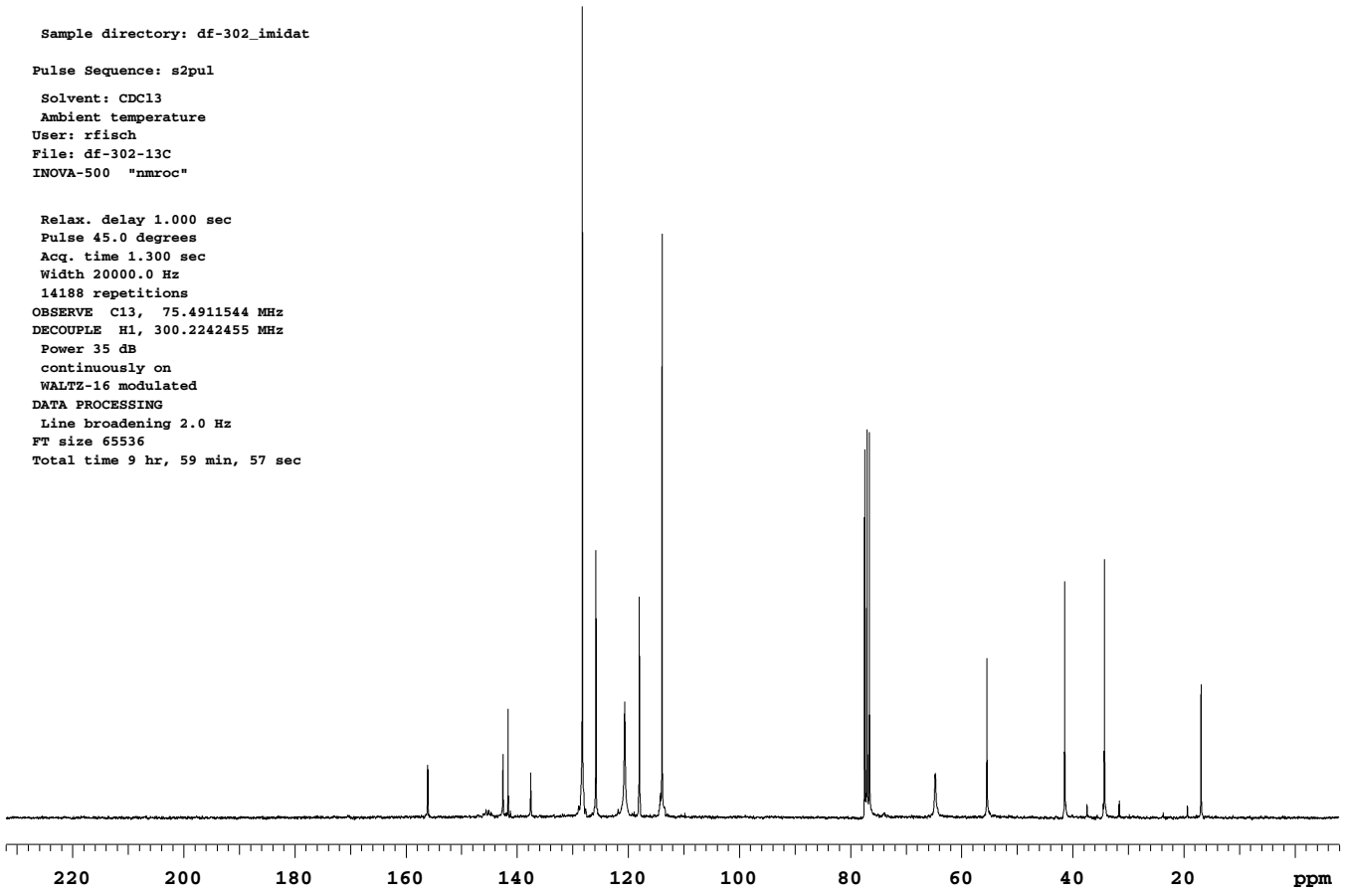
13C OBSERVE

Sample directory: df-302_imidat

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-302-13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
14188 repetitions
OBSERVE C13, 75.4911544 MHz
DECOUPLE H1, 300.2242455 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 9 hr, 59 min, 57 sec



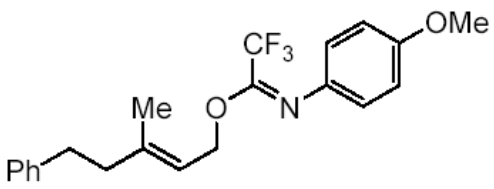
19F OBSERVE
STANDARD PARAMETERS

Sample directory: df-318

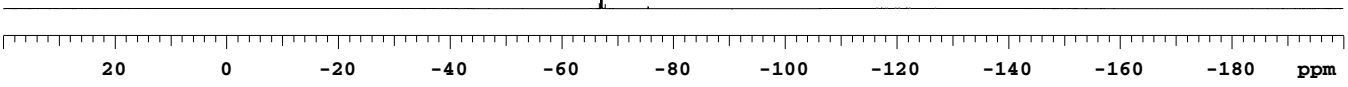
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-318_19F
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
16 repetitions
OBSERVE F19, 282.0683162 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 1 min, 20 sec



4a



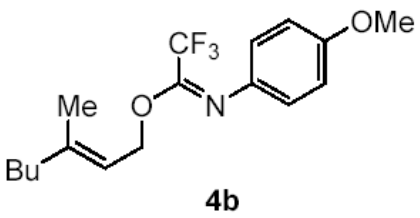
STANDARD 1H OBSERVE

Sample directory: df-252c

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-252c_1H
INOVA-500 "nmroc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
16 repetitions
OBSERVE H1, 300.2230602 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 52 sec



4b

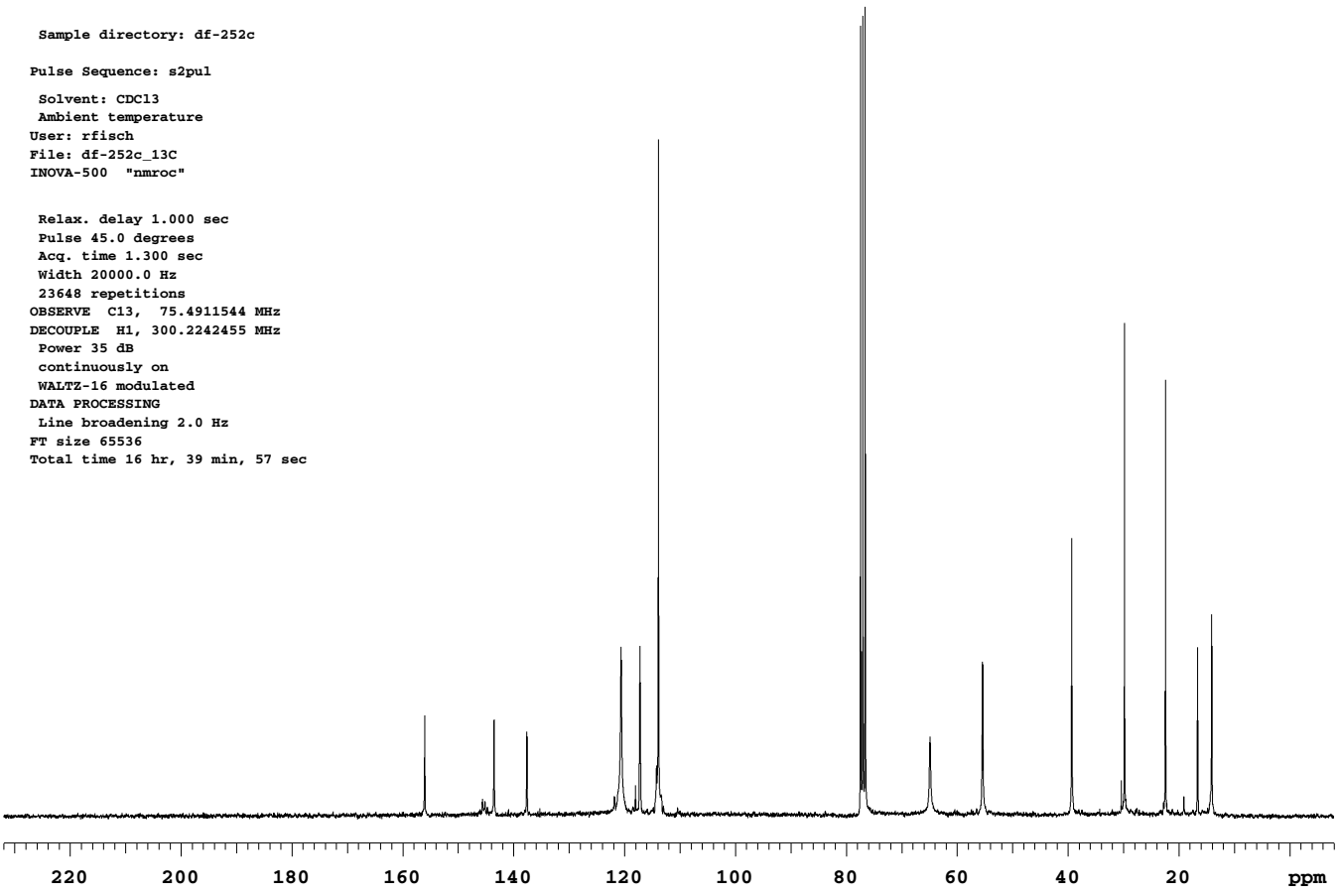
13C OBSERVE

Sample directory: df-252c

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-252c_13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
23648 repetitions
OBSERVE C13, 75.4911544 MHz
DECOUPLE H1, 300.2242455 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 16 hr, 39 min, 57 sec



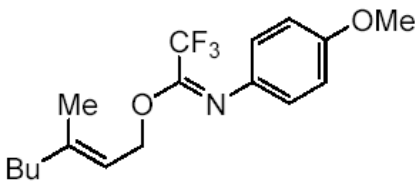
19F OBSERVE
STANDARD PARAMETERS

Sample directory: df-252c

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-252c_19F
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
16 repetitions
OBSERVE F19, 282.4918708 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 1 min, 20 sec



4b

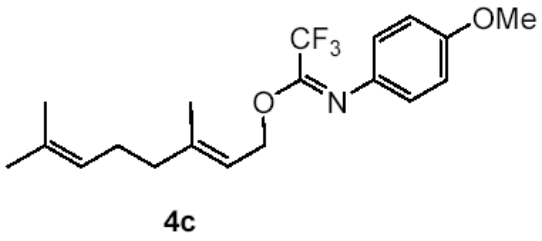
STANDARD 1H OBSERVE

Sample directory: df-252c

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-252c_geraniol_1H
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 56.2 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
16 repetitions
OBSERVE H1, 299.7729174 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec



4c

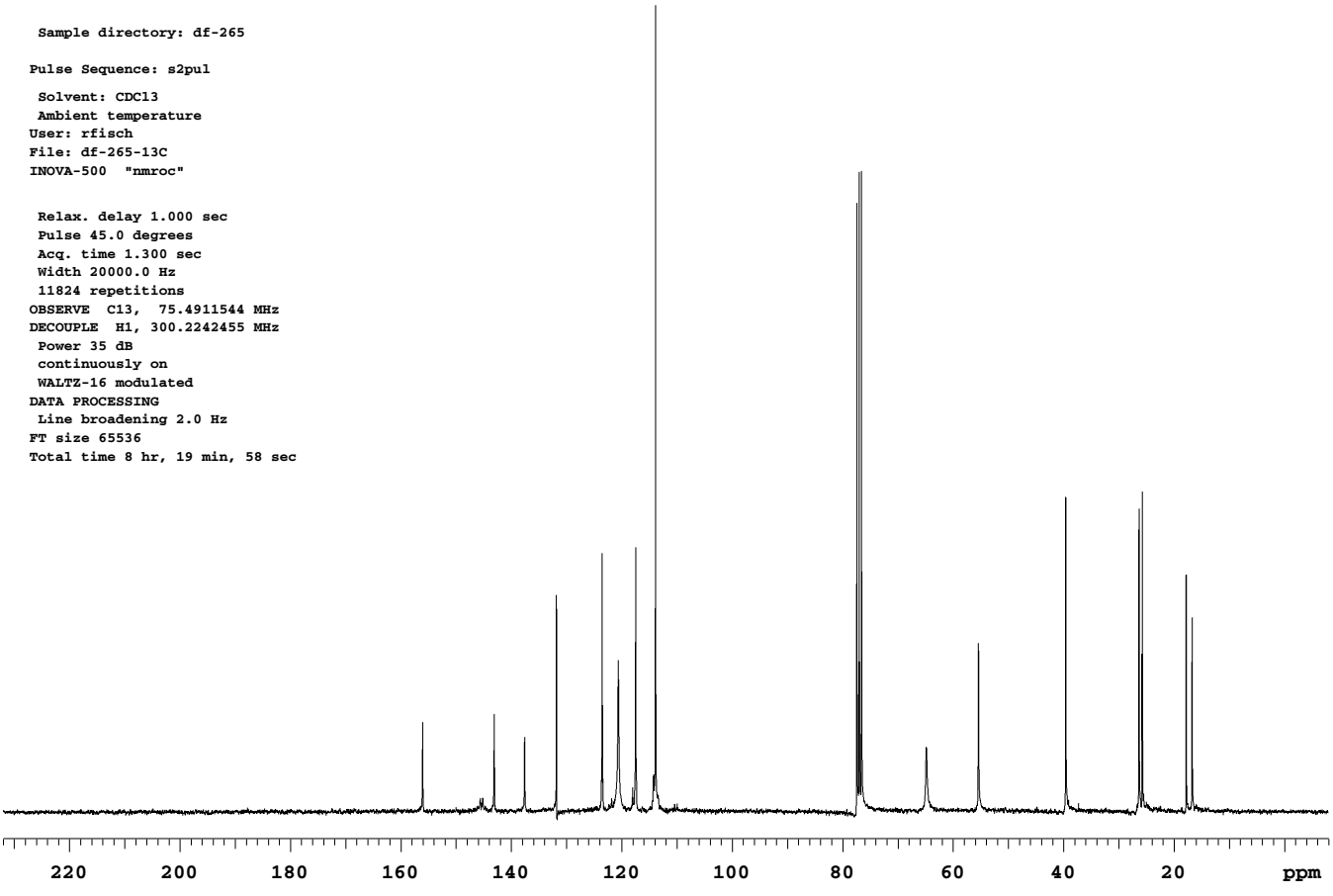
13C OBSERVE

Sample directory: df-265

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-265-13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
11824 repetitions
OBSERVE C13, 75.4911544 MHz
DECOUPLE H1, 300.2242455 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 8 hr, 19 min, 58 sec



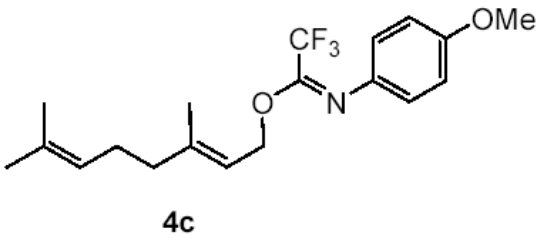
19F OBSERVE
STANDARD PARAMETERS

Sample directory: df-252c

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-252c_geraniol_19F
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
16 repetitions
OBSERVE F19, 282.0683162 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 1 min, 20 sec



4c

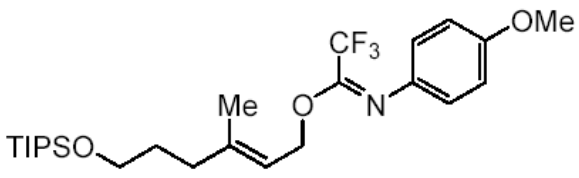
STANDARD 1H OBSERVE

Sample directory: df-300_imidat

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-300_imidat_1H_030407
INOVA-500 "nmroc"

Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
16 repetitions
OBSERVE H1, 299.9012510 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 52 sec



4d

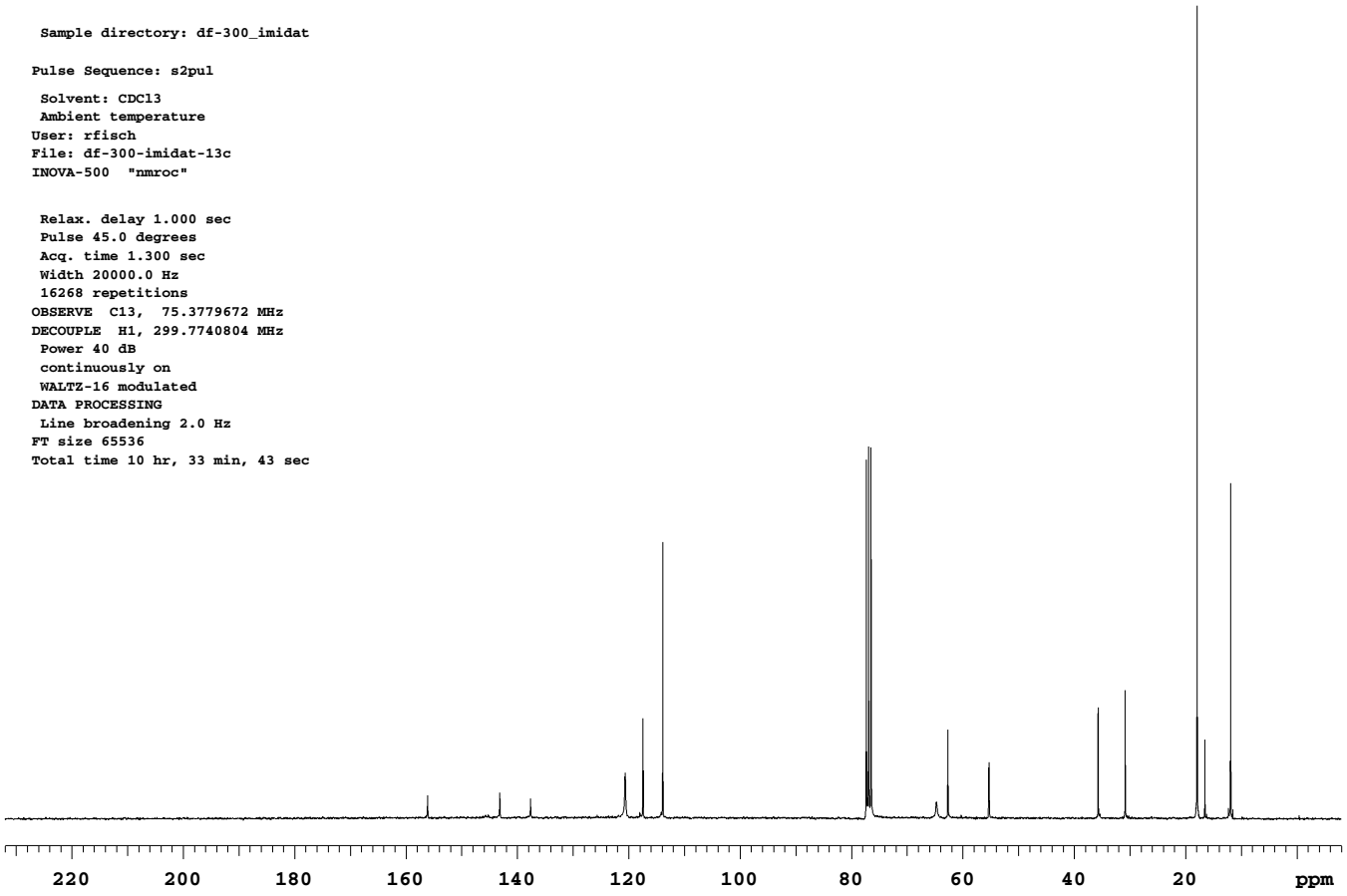
13C OBSERVE

Sample directory: df-300_imidat

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-300-imidat-13c
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
16268 repetitions
OBSERVE C13, 75.3779672 MHz
DECOUPLE H1, 299.7740804 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 10 hr, 33 min, 43 sec



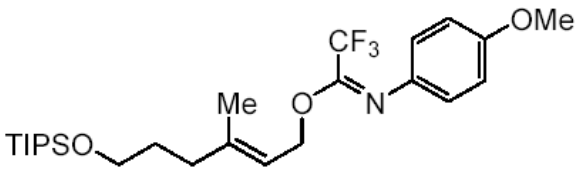
19F OBSERVE
STANDARD PARAMETERS

Sample directory: df-302_imidat

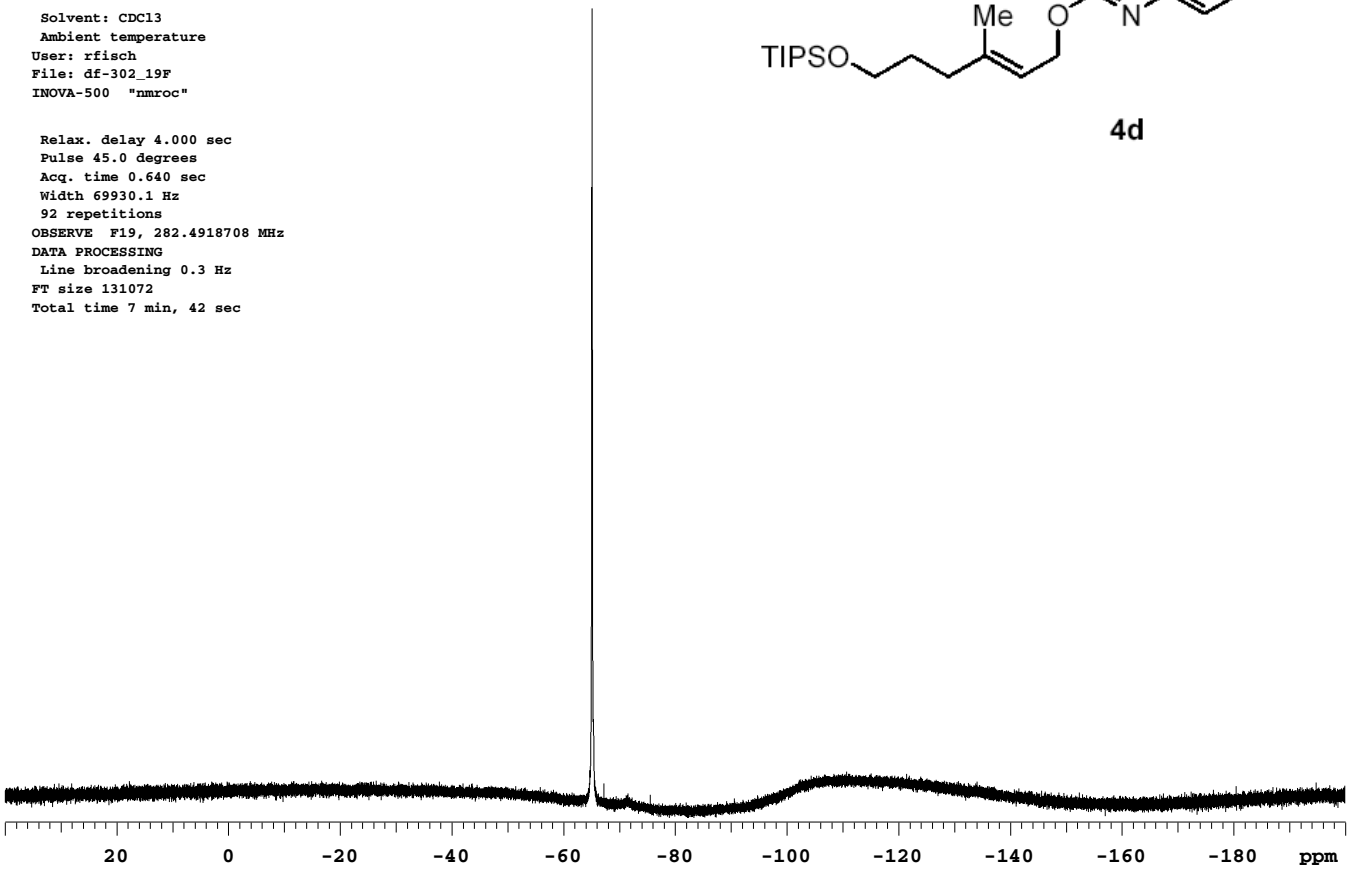
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-302_19F
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
92 repetitions
OBSERVE F19, 282.4918708 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 7 min, 42 sec



4d



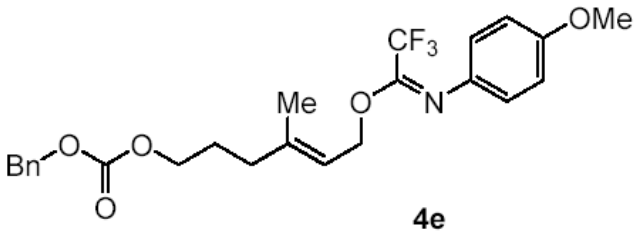
STANDARD 1H OBSERVE

Sample directory: X359PPF-15

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X359PPF-15
INOVA-500 "nmroc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
16 repetitions
OBSERVE H1, 300.2230602 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 52 sec



4e

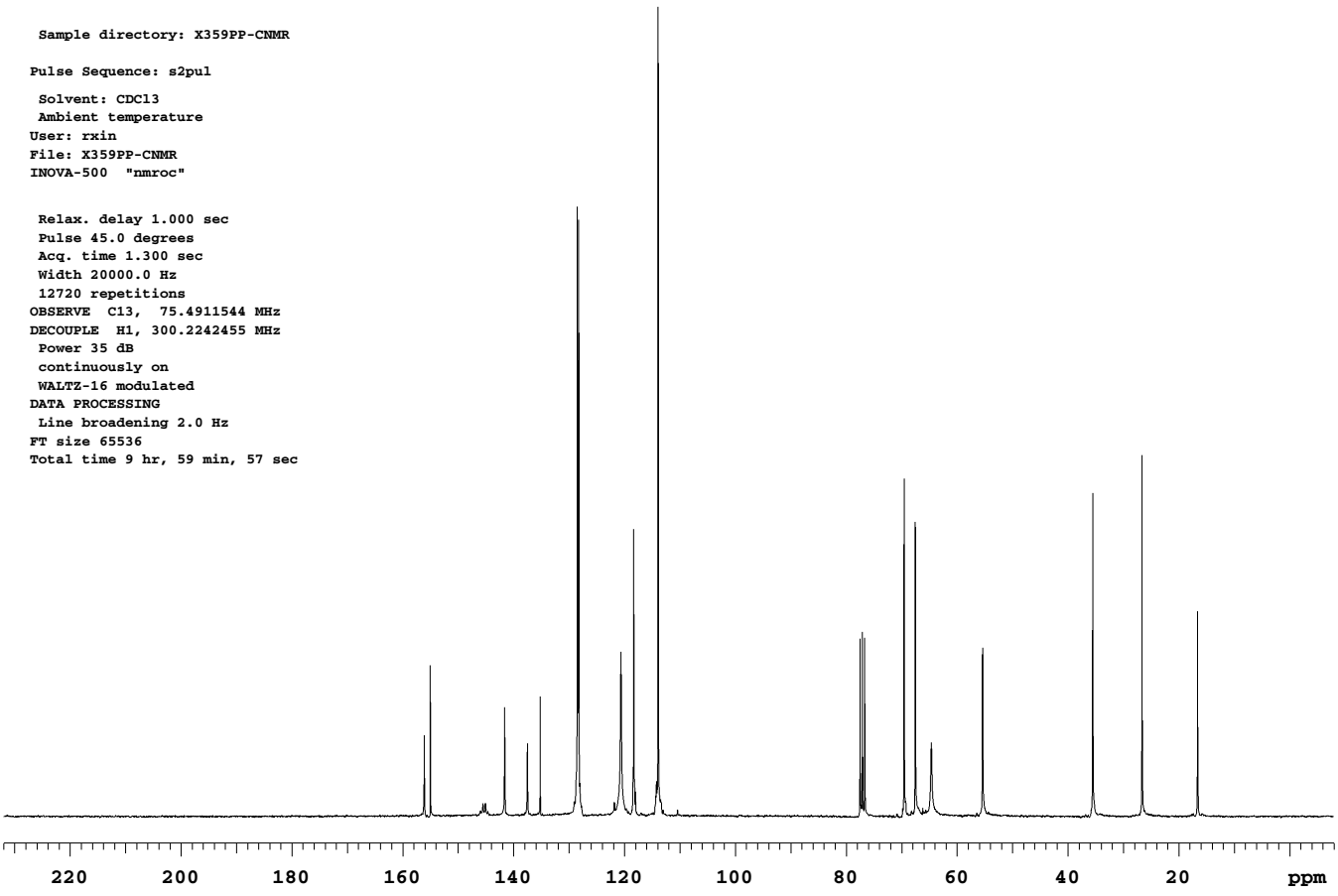
13C OBSERVE

Sample directory: X359PP-CNMR

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X359PP-CNMR
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
12720 repetitions
OBSERVE C13, 75.4911544 MHz
DECOUPLE H1, 300.2242455 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 9 hr, 59 min, 57 sec



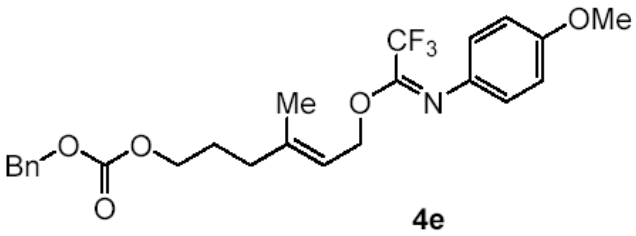
19F OBSERVE
STANDARD PARAMETERS

Sample directory: X359PPF-15

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X359PPF-15-FNMR
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
19 repetitions
OBSERVE F19, 282.4918708 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 1 min, 39 sec



4e

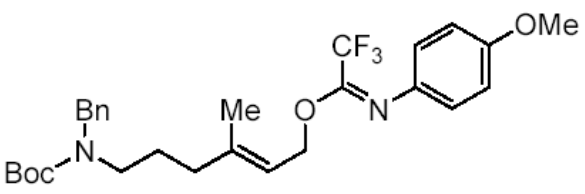
STANDARD 1H OBSERVE

Sample directory: X357PPF-10

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X357PPF-10
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 56.2 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
56 repetitions
OBSERVE H1, 299.7729174 MHz
DATA PROCESSING
FT size 32768
Total time 2 min, 54 sec



4f

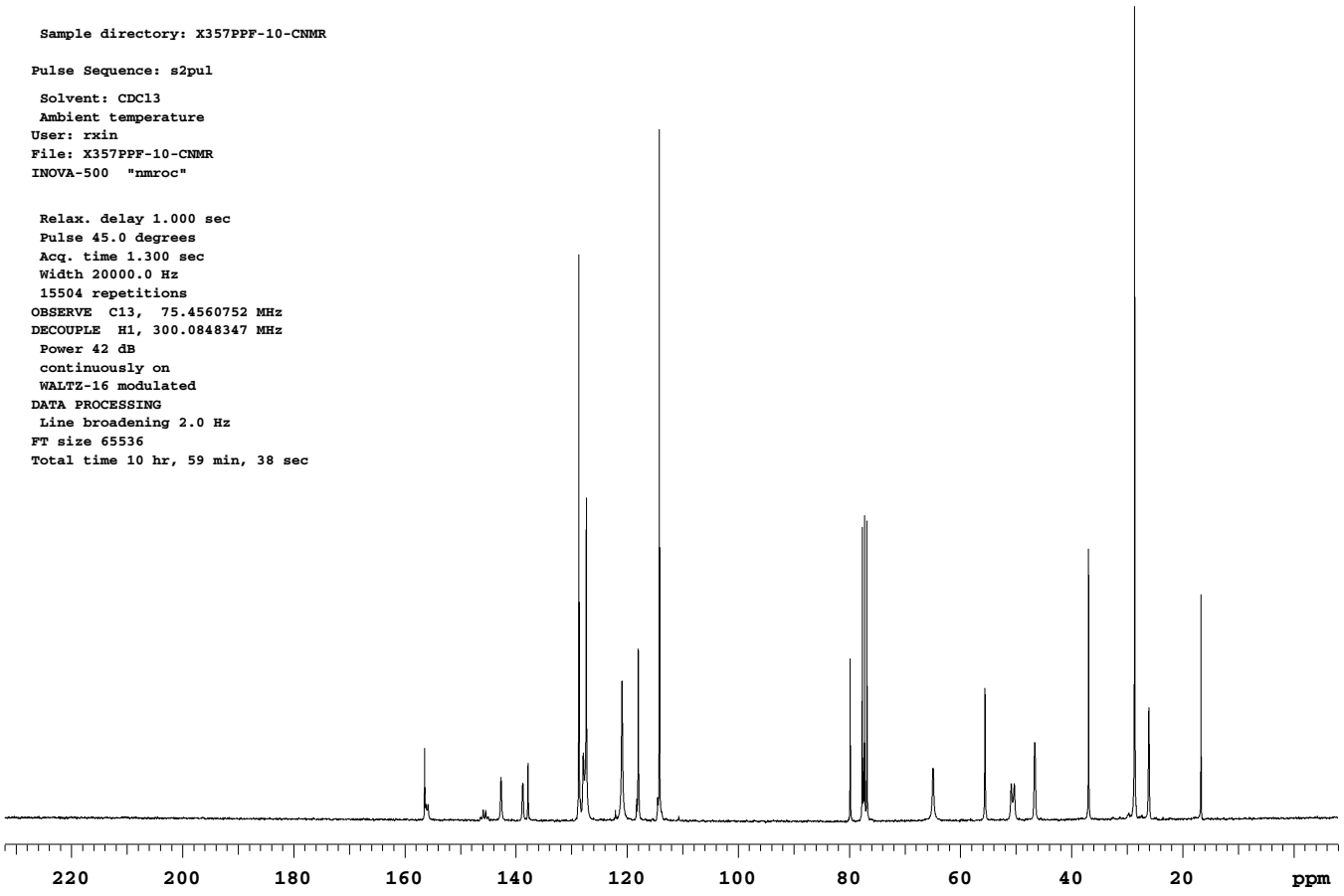
13C OBSERVE

Sample directory: X357PPF-10-CNMR

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X357PPF-10-CNMR
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
15504 repetitions
OBSERVE C13, 75.4560752 MHz
DECOUPLE H1, 300.0848347 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 10 hr, 59 min, 38 sec



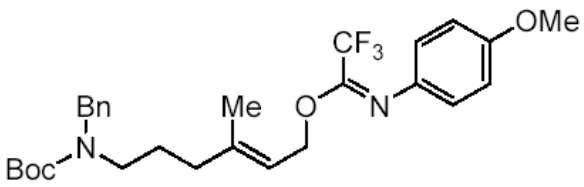
19F OBSERVE
STANDARD PARAMETERS

Sample directory: X357PPF-10

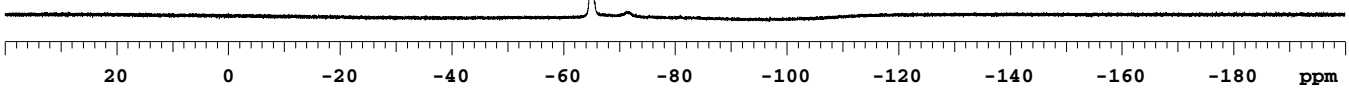
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X357PPF-10-FNMR
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
19 repetitions
OBSERVE F19, 282.0683162 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 1 min, 39 sec



4f



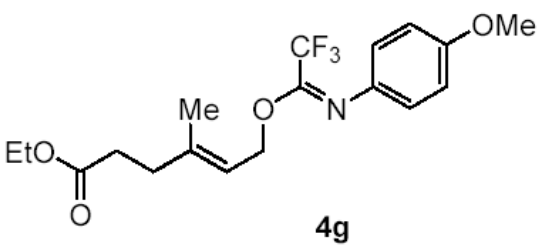
STANDARD 1H OBSERVE

Sample directory: X275PP-F25

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X275PP-F25
INOVA-500 "nmroc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
32 repetitions
OBSERVE H1, 300.2230602 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 45 sec



4g

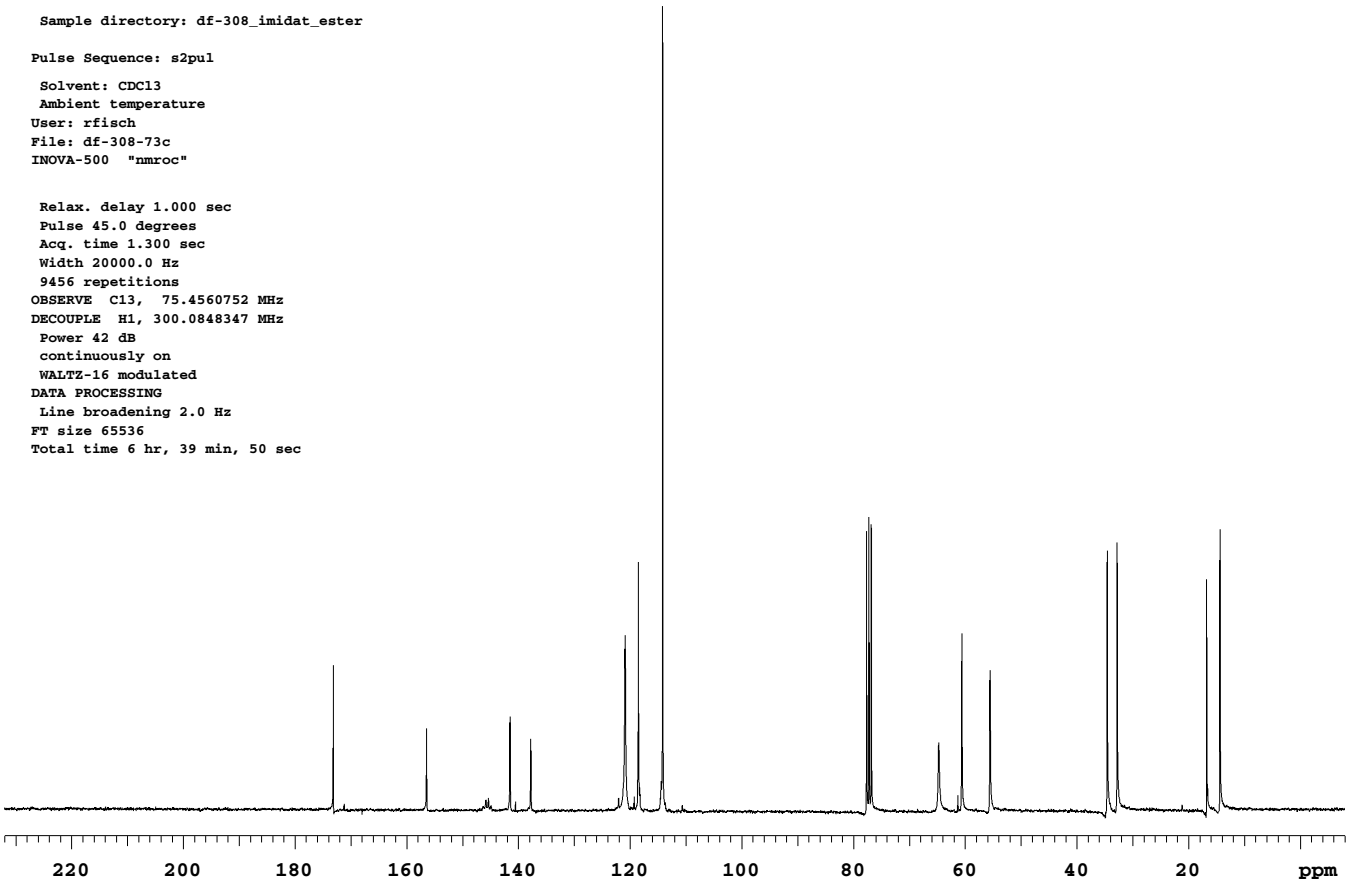
13C OBSERVE

Sample directory: df-308_imidat_ester

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-308-73c
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
9456 repetitions
OBSERVE C13, 75.4560752 MHz
DECOUPLE H1, 300.0848347 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 6 hr, 39 min, 50 sec



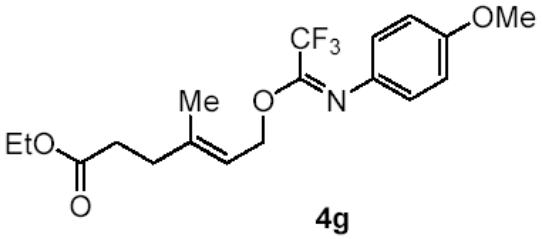
19F OBSERVE
STANDARD PARAMETERS

Sample directory: df-308_imidat_ester

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-308_19F
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
16 repetitions
OBSERVE F19, 282.3608271 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 1 min, 20 sec



4g

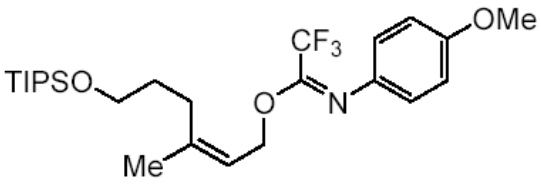
STANDARD 1H OBSERVE

Sample directory: df-314_imidat

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-314_imidat_160407_1H
INOVA-500 "nmroc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
16 repetitions
OBSERVE H1, 300.2230602 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 52 sec



4h

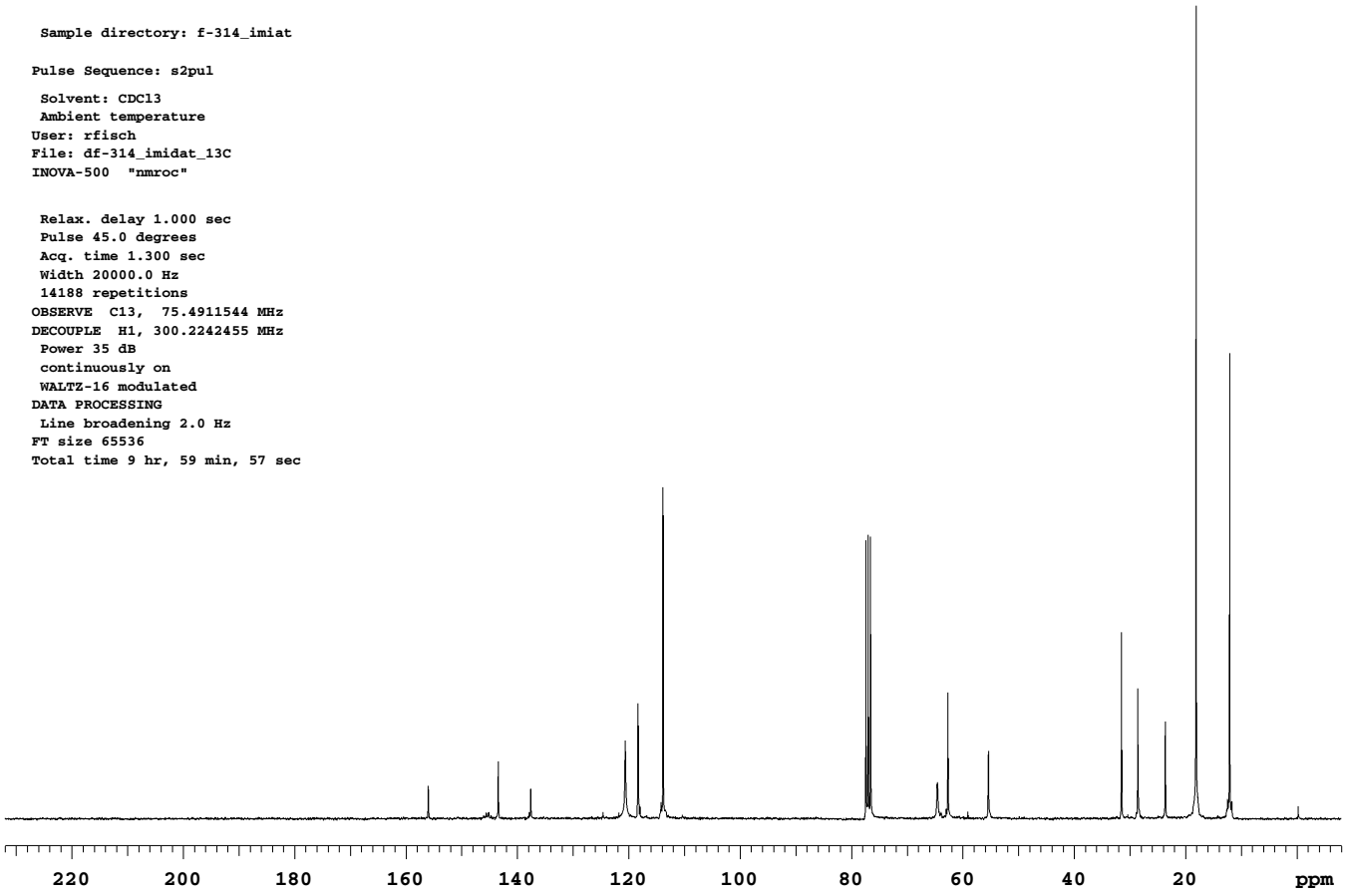
13C OBSERVE

Sample directory: f-314_imiat

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-314_imidat_13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
14188 repetitions
OBSERVE C13, 75.4911544 MHz
DECOUPLE H1, 300.2242455 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 9 hr, 59 min, 57 sec



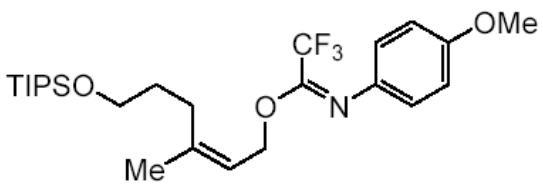
19F OBSERVE
STANDARD PARAMETERS

Sample directory: df-314_imidat

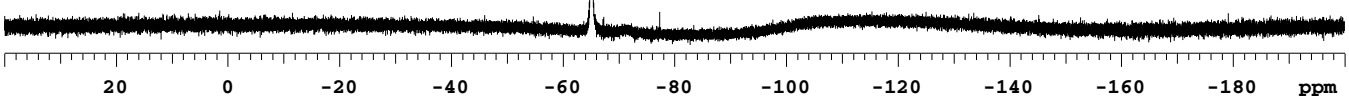
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-314_imidat_160407_19F
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
16 repetitions
OBSERVE F19, 282.4918708 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 1 min, 20 sec



4h



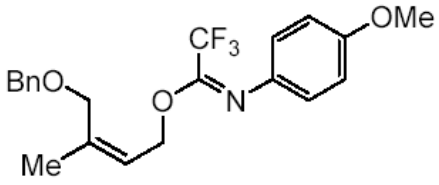
STANDARD 1H OBSERVE

Sample directory: X363PPF24

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X363PPF24
INOVA-500 "nmroc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
88 repetitions
OBSERVE H1, 300.2230602 MHz
DATA PROCESSING
FT size 32768
Total time 4 min, 51 sec



4i

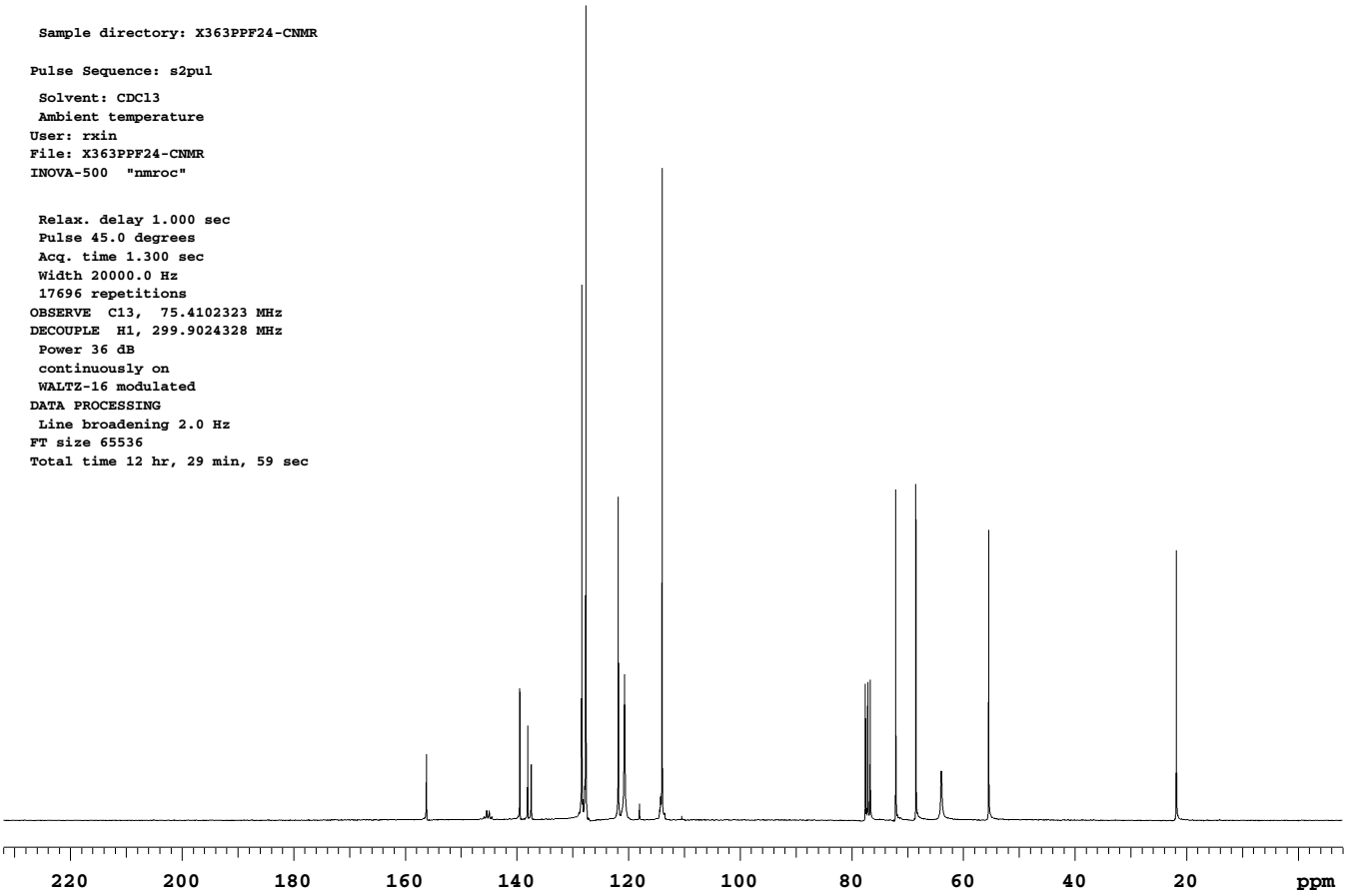
13C OBSERVE

Sample directory: X363PPF24-CNMR

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X363PPF24-CNMR
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
17696 repetitions
OBSERVE C13, 75.4102323 MHz
DECOUPLE H1, 299.9024328 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 12 hr, 29 min, 59 sec



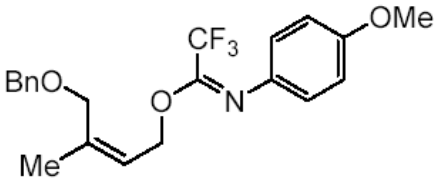
19F OBSERVE
STANDARD PARAMETERS

Sample directory: X363PPF24

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X363PPF24-FNMR
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
19 repetitions
OBSERVE F19, 282.4918708 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 1 min, 39 sec



4i

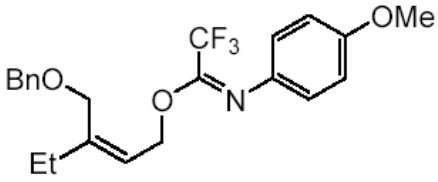
STANDARD 1H OBSERVE

Sample directory: df-340_imidat_column

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-340_imidat_1H
INOVA-500 "nmroc"

Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
16 repetitions
OBSERVE H1, 299.9012510 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 52 sec



4j

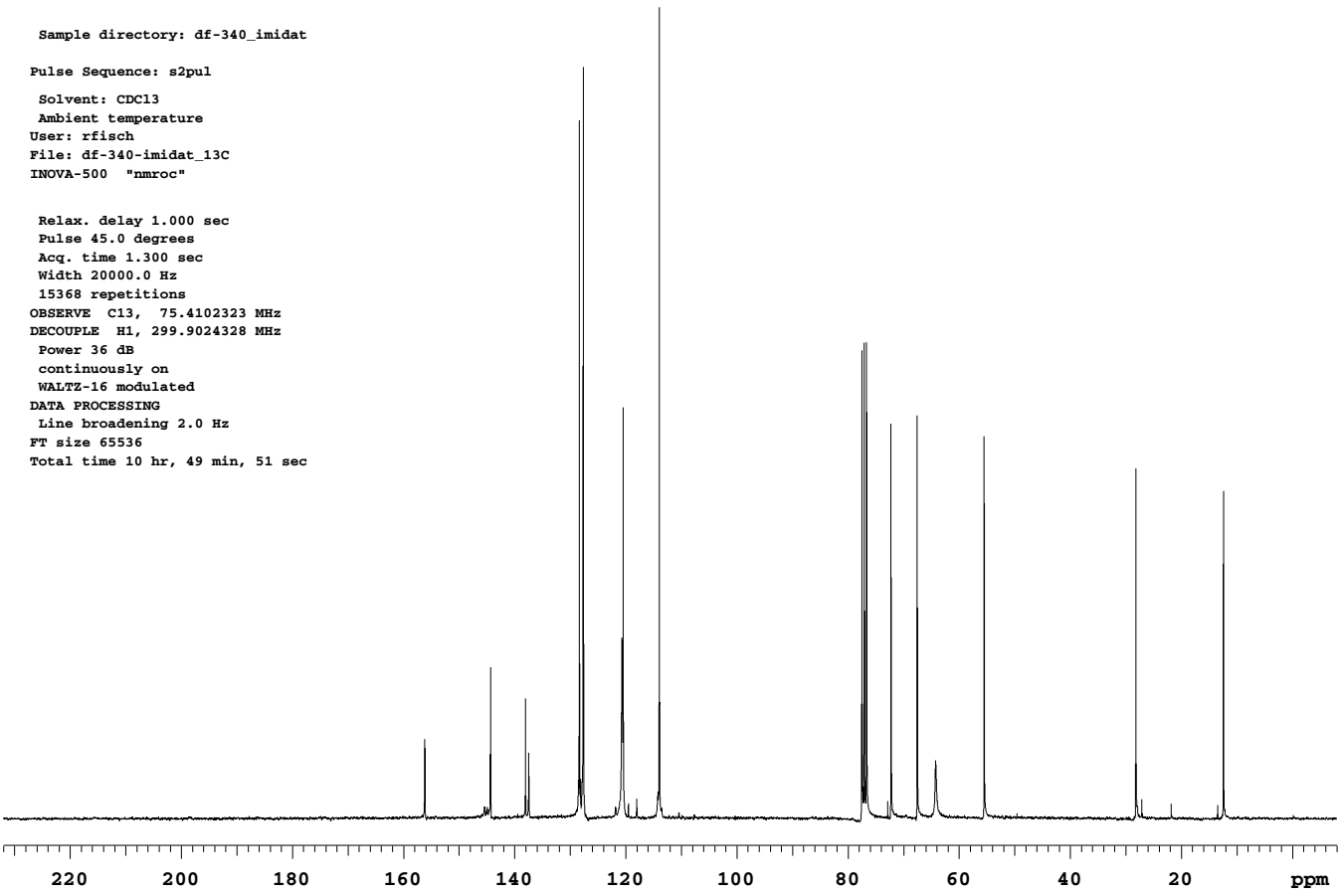
13C OBSERVE

Sample directory: df-340_imidat

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-340-imidat_13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
15368 repetitions
OBSERVE C13, 75.4102323 MHz
DECOUPLE H1, 299.9024328 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 10 hr, 49 min, 51 sec



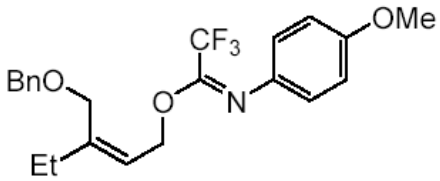
19F OBSERVE
STANDARD PARAMETERS

Sample directory: df-340_imidat

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-340_imidat_19F
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
16 repetitions
OBSERVE F19, 282.1890544 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 1 min, 20 sec



4j

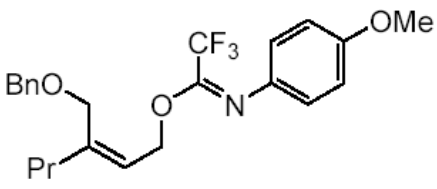
STANDARD 1H OBSERVE

Sample directory: df-339_imidat

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-339_imidat_130407_1H
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 30.2 degrees
Acq. time 3.138 sec
Width 4500.5 Hz
16 repetitions
OBSERVE H1, 299.7729174 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 8 sec



4k

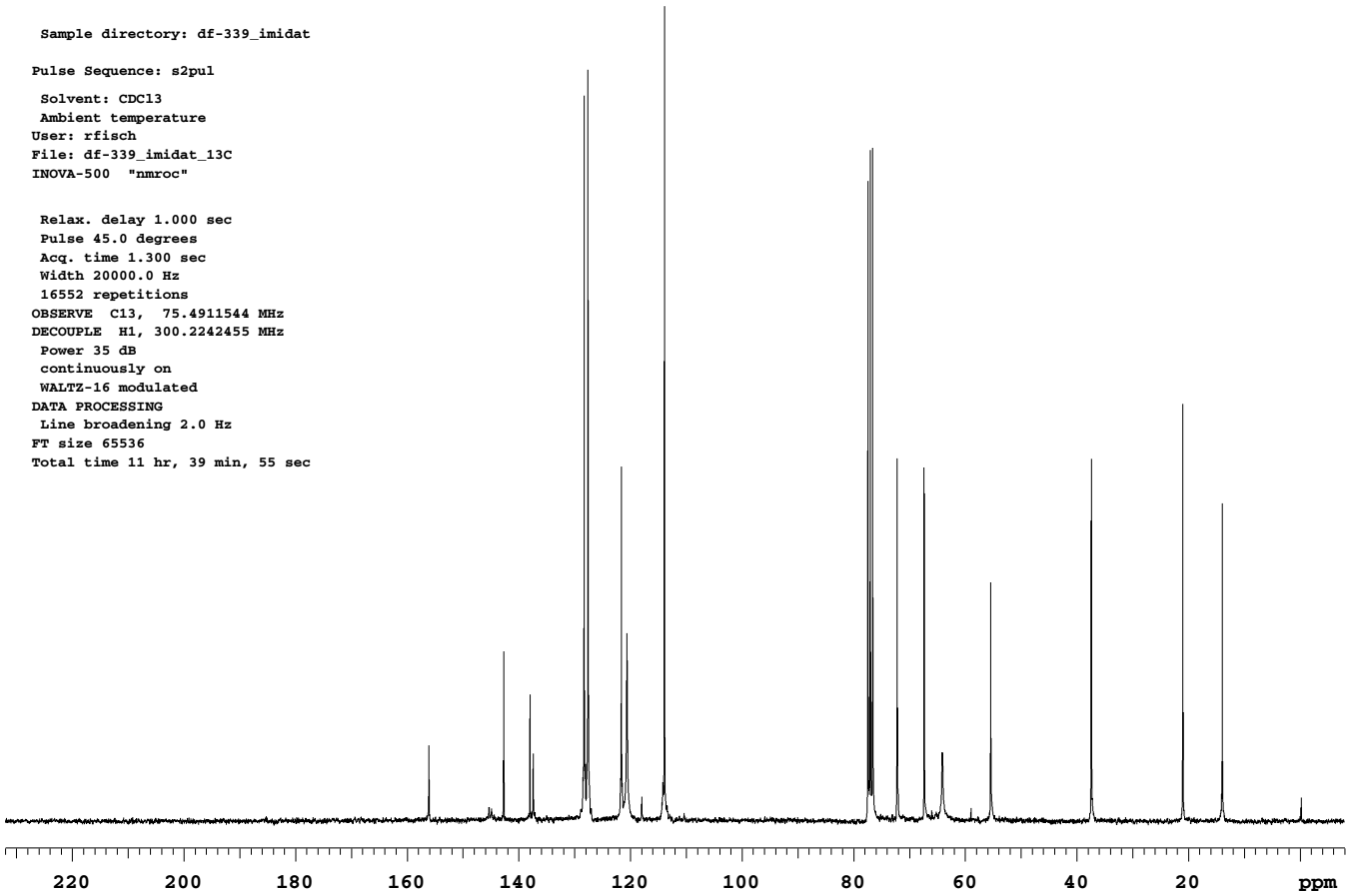
13C OBSERVE

Sample directory: df-339_imidat

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-339_imidat_13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
16552 repetitions
OBSERVE C13, 75.4911544 MHz
DECOUPLE H1, 300.2242455 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 11 hr, 39 min, 55 sec



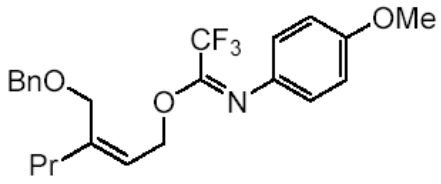
19F OBSERVE
STANDARD PARAMETERS

Sample directory: df-339_imidat

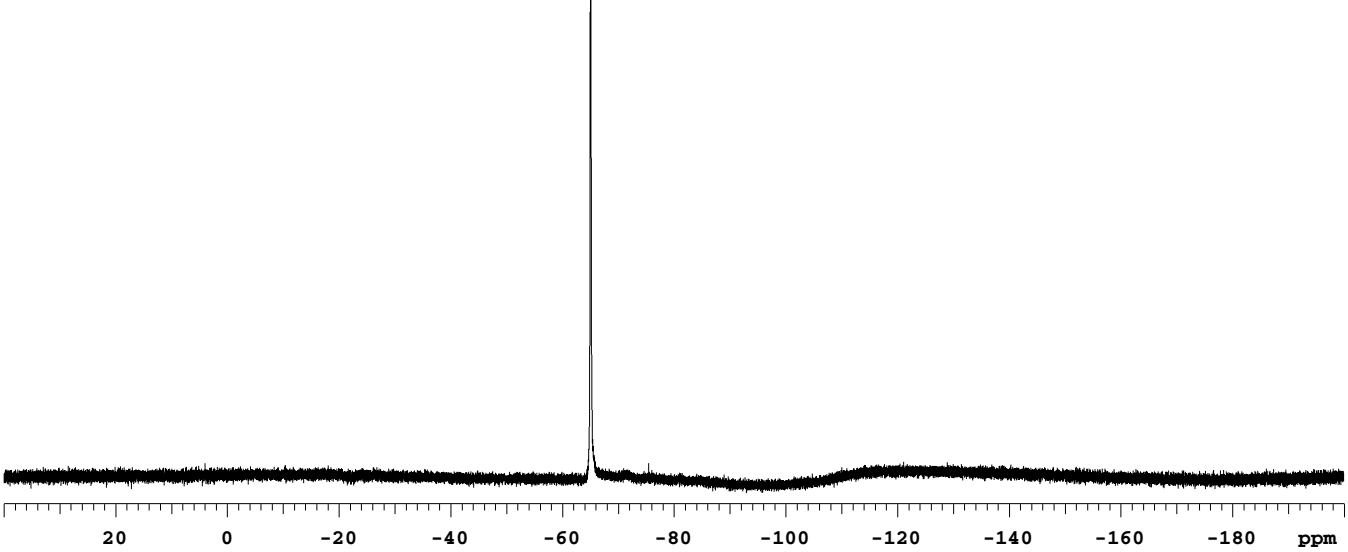
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-339_imidat_130407_19F
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
16 repetitions
OBSERVE F19, 282.0683162 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 1 min, 20 sec



4k



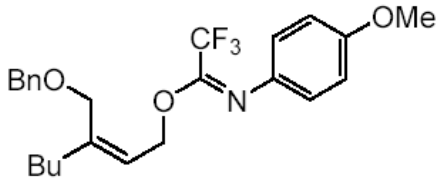
STANDARD 1H OBSERVE

Sample directory: df-337_imidat

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-337_imidat_1H
INOVA-500 "nmroc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
16 repetitions
OBSERVE H1, 300.2230602 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 52 sec



4l

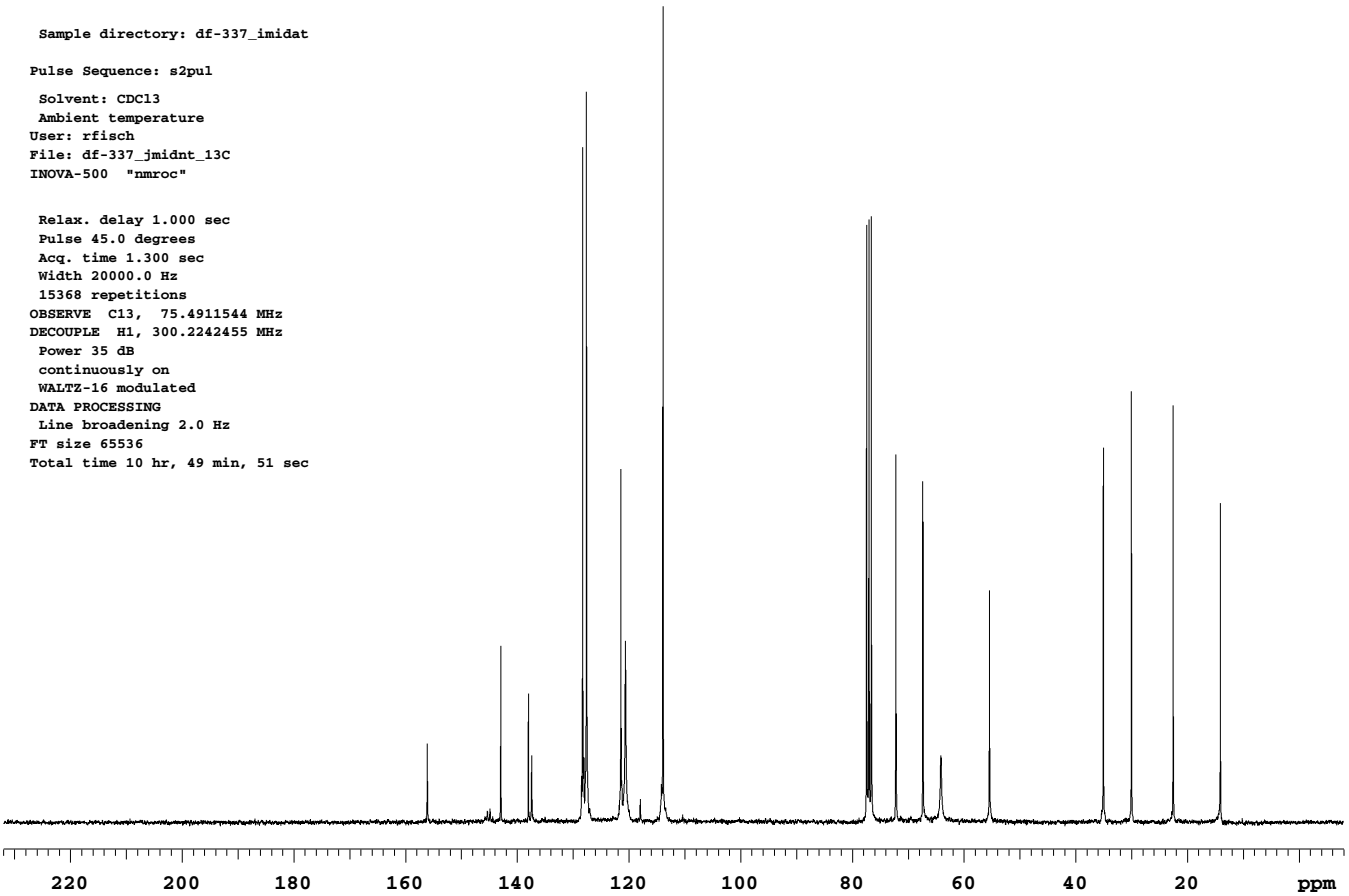
13C OBSERVE

Sample directory: df-337_imidat

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-337_jmidnt_13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
15368 repetitions
OBSERVE C13, 75.4911544 MHz
DECOUPLE H1, 300.2242455 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 10 hr, 49 min, 51 sec



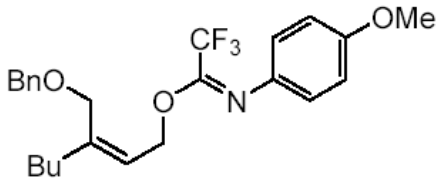
19F OBSERVE
STANDARD PARAMETERS

Sample directory: df-337_imidat

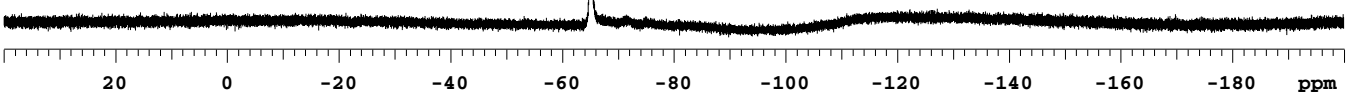
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-337_imidat_130407_19F
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
16 repetitions
OBSERVE F19, 282.0683162 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 1 min, 20 sec



4l



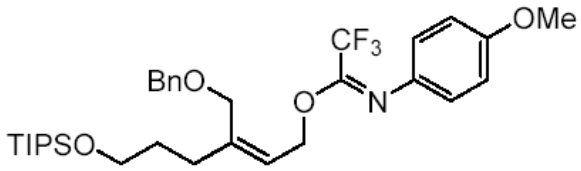
STANDARD 1H OBSERVE

Sample directory: df-341_imidat

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-341_imidat_1H
INOVA-500 "nmroc"

Pulse 29.5 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
8 repetitions
OBSERVE H1, 300.0836333 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 27 sec



4m

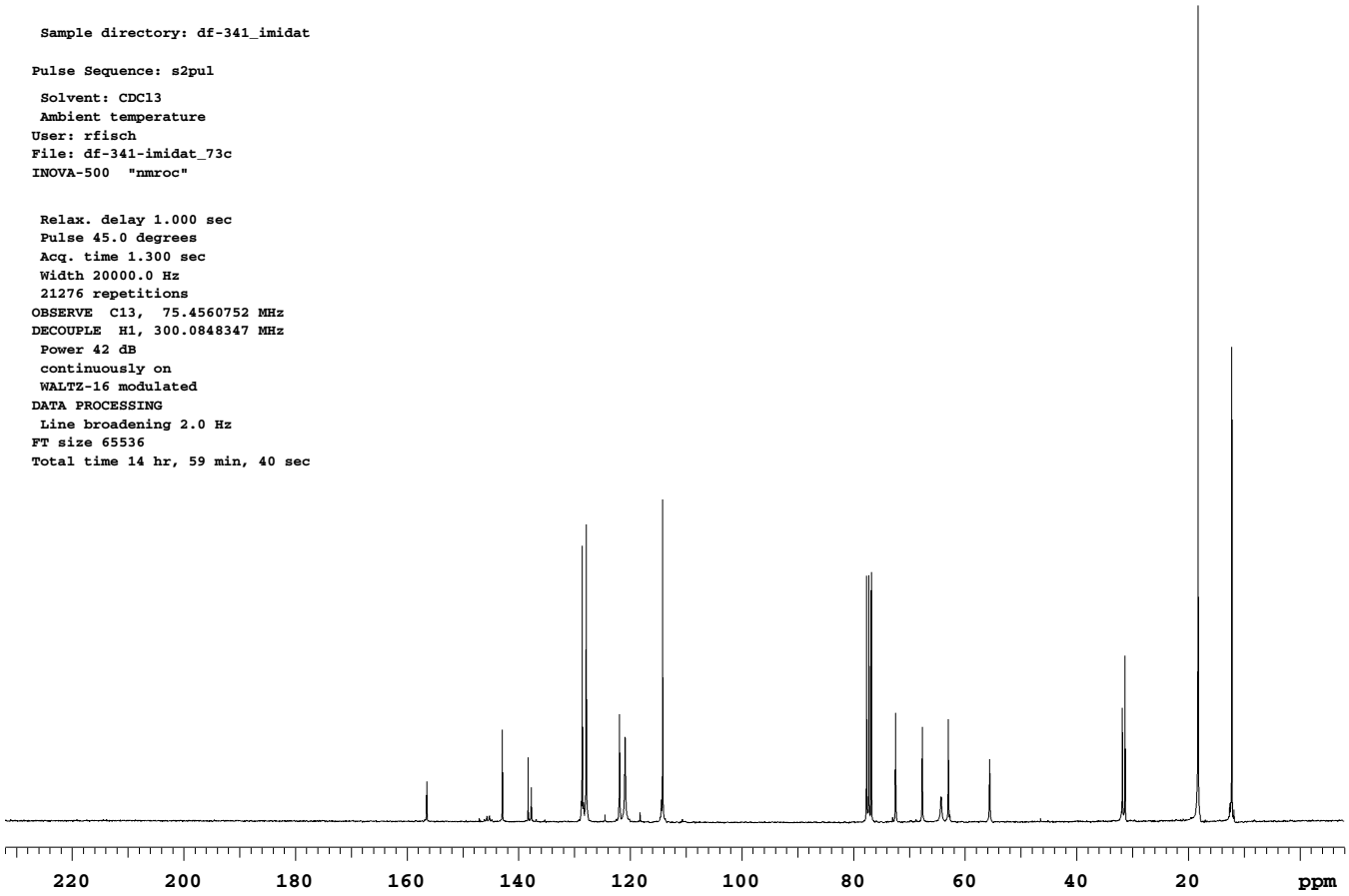
13C OBSERVE

Sample directory: df-341_imidat

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-341-imidat_73c
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
21276 repetitions
OBSERVE C13, 75.4560752 MHz
DECOUPLE H1, 300.0848347 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 14 hr, 59 min, 40 sec



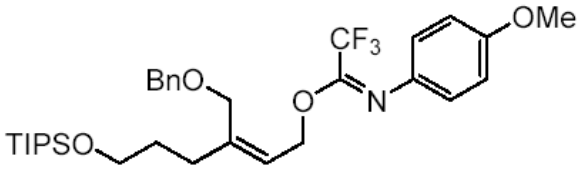
19F OBSERVE
STANDARD PARAMETERS

Sample directory: df-341_imidat

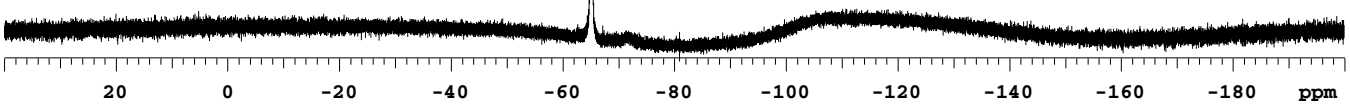
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-341_imidat_160407_19F
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
48 repetitions
OBSERVE F19, 282.4918708 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 4 min, 0 sec



4m



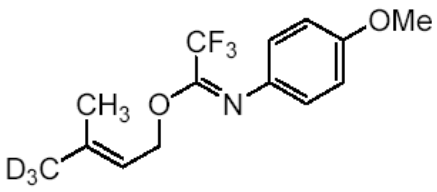
STANDARD 1H OBSERVE

Sample directory: df-343_imidat

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-343_imidat_1H
INOVA-500 "nmroc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
16 repetitions
OBSERVE H1, 300.2230602 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 52 sec



4n

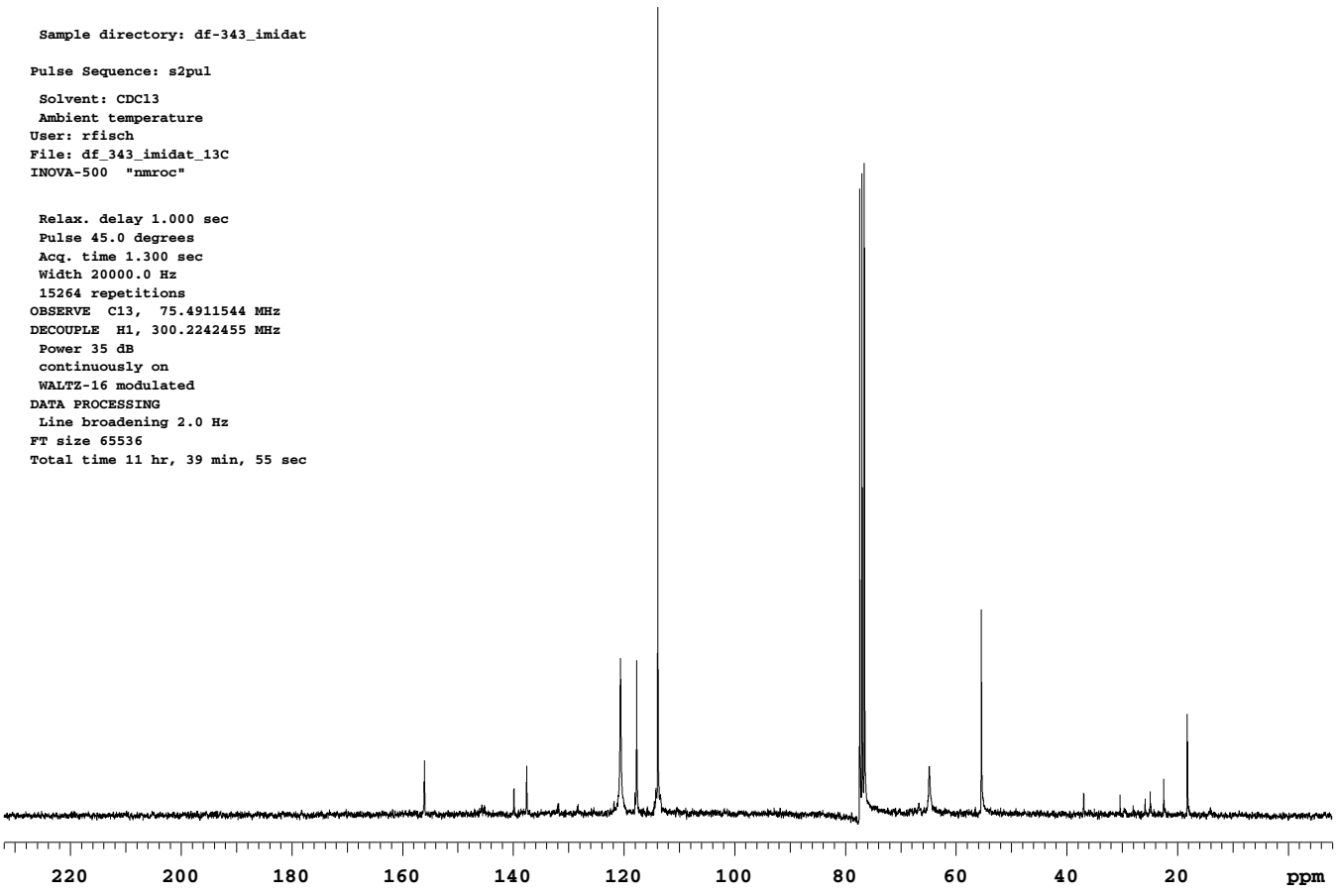
13C OBSERVE

Sample directory: df-343_imidat

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df_343_imidat_13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
15264 repetitions
OBSERVE C13, 75.4911544 MHz
DECOUPLE H1, 300.2242455 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 11 hr, 39 min, 55 sec



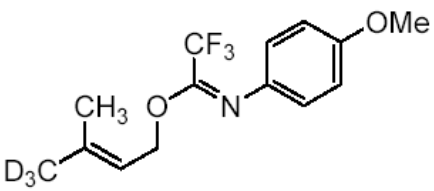
19F OBSERVE
STANDARD PARAMETERS

Sample directory: df-343_imidat

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-343_imidat_19F
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
16 repetitions
OBSERVE F19, 282.4918708 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 1 min, 20 sec



4n

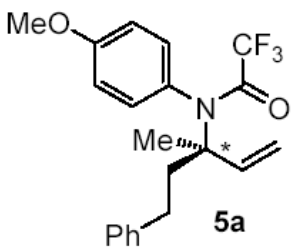
STANDARD 1H OBSERVE

Sample directory: X403-2-PP-2

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X403-2-PP-2_110507
INOVA-500 "nmroc"

Pulse 29.5 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
32 repetitions
OBSERVE H1, 300.0836333 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 45 sec



5a

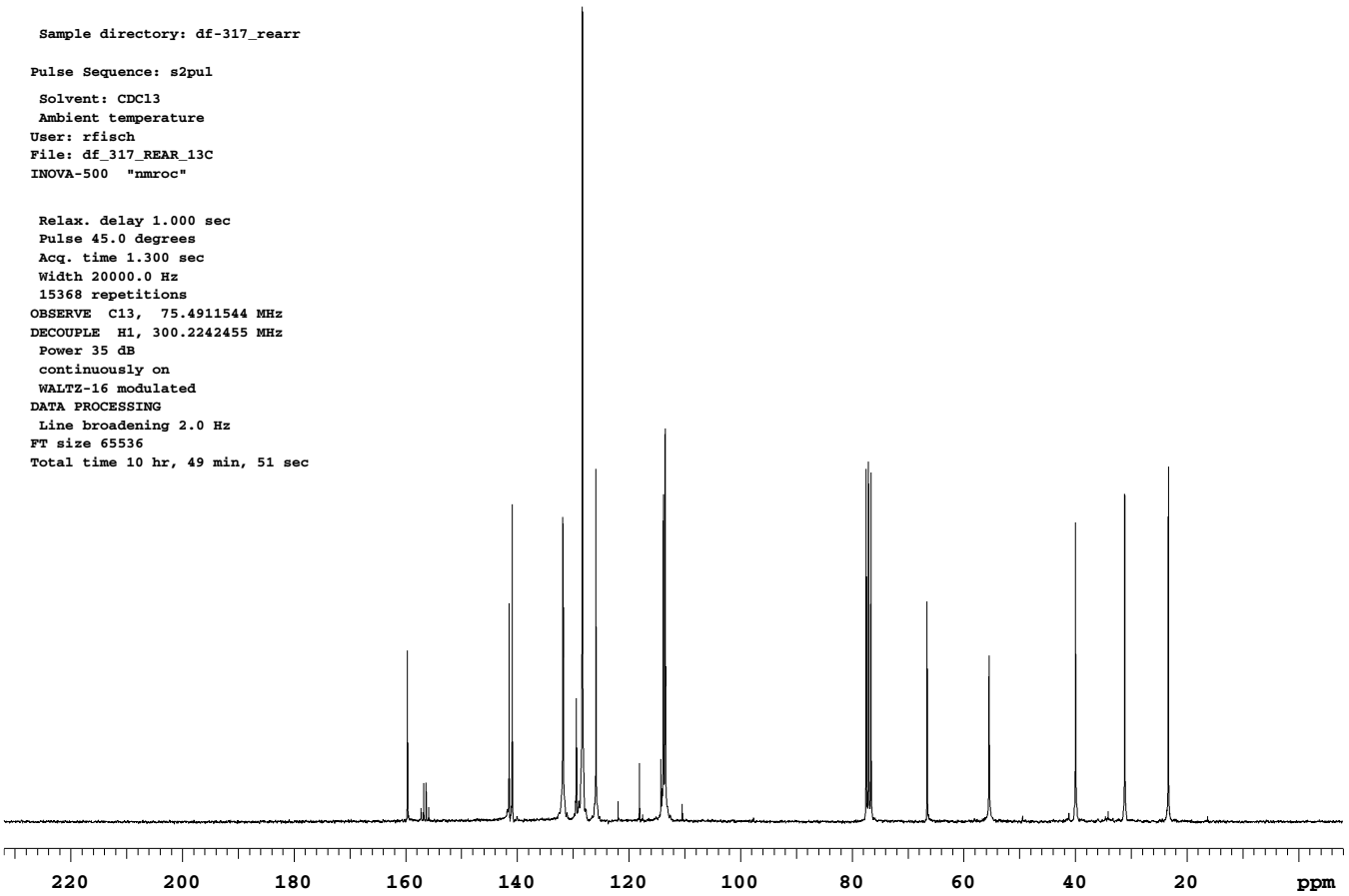
13C OBSERVE

Sample directory: df-317_rearr

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df_317_REAR_13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
15368 repetitions
OBSERVE C13, 75.4911544 MHz
DECOUPLE H1, 300.2242455 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 10 hr, 49 min, 51 sec



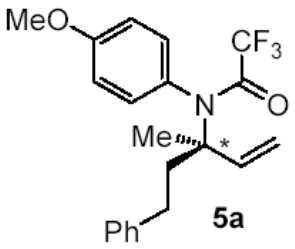
19F OBSERVE
STANDARD PARAMETERS

Sample directory: df-317_rearr

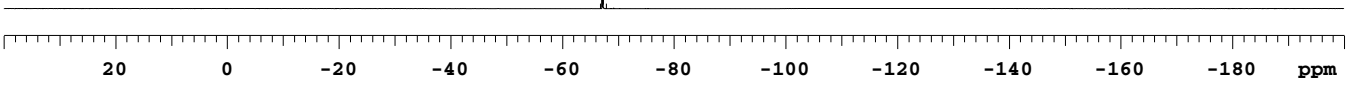
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-317_rearr_19F
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
16 repetitions
OBSERVE F19, 282.4918708 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 1 min, 20 sec



5a



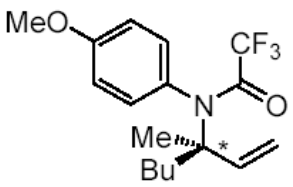
STANDARD 1H OBSERVE

Sample directory: df-338_rear

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-338_Rear_1H
INOVA-500 "nmroc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
16 repetitions
OBSERVE H1, 300.2230602 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 52 sec



5b

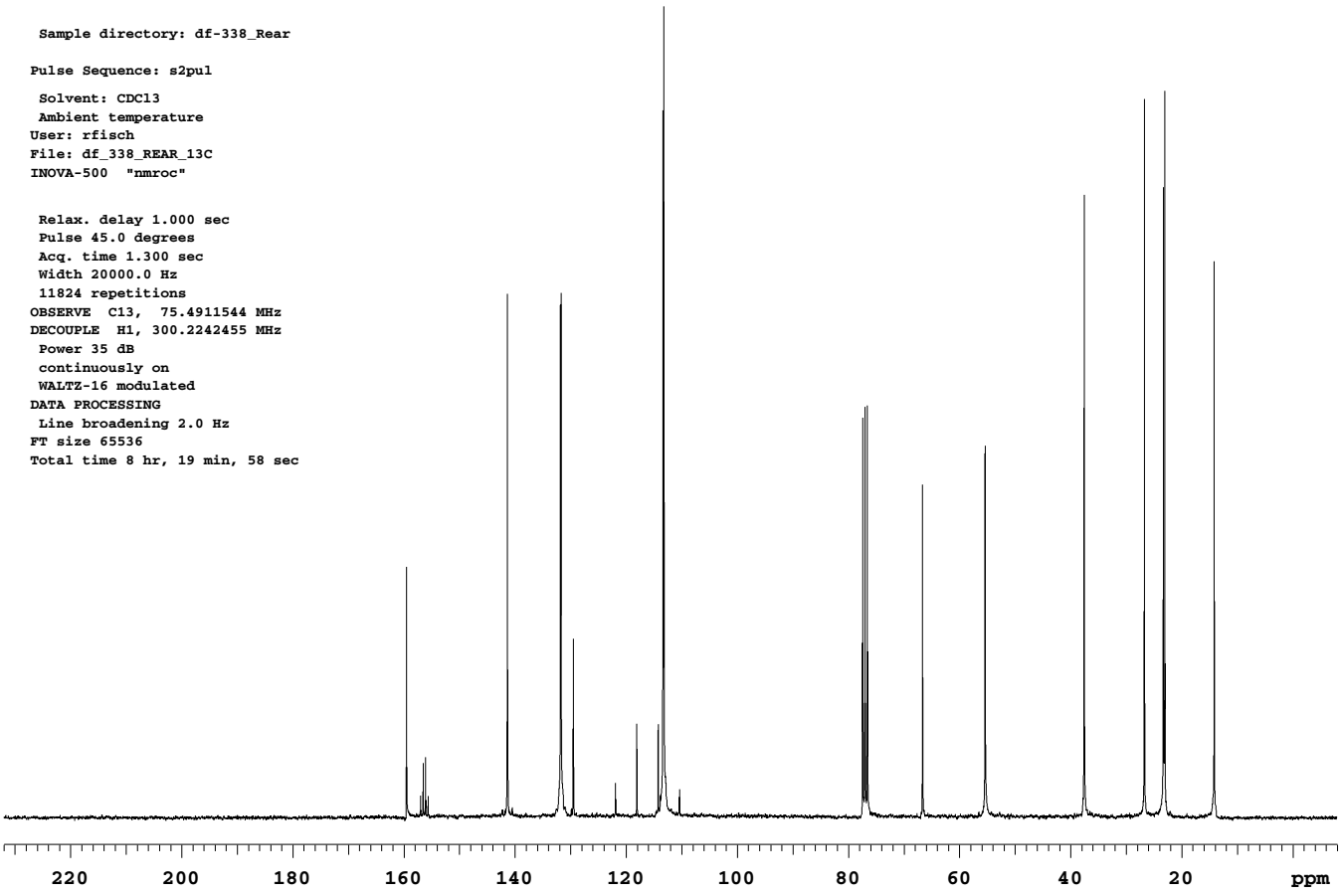
13C OBSERVE

Sample directory: df-338_Rear

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df_338_REAR_13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
11824 repetitions
OBSERVE C13, 75.4911544 MHz
DECOUPLE H1, 300.2242455 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 8 hr, 19 min, 58 sec



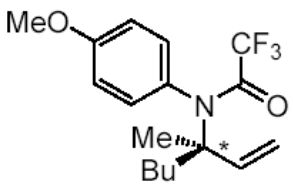
19F OBSERVE
STANDARD PARAMETERS

Sample directory: df-338_rear

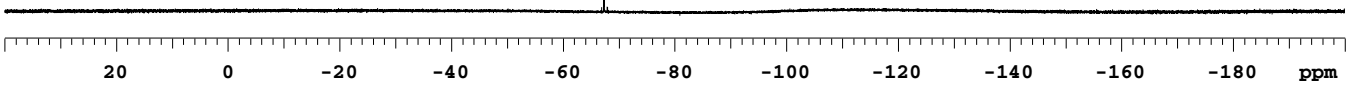
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-338_Rear_19F
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
16 repetitions
OBSERVE F19, 282.4918708 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 1 min, 20 sec



5b



20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 ppm

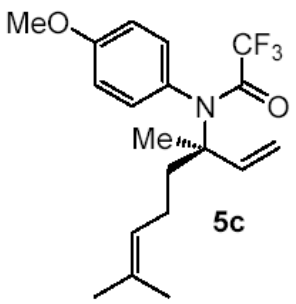
STANDARD 1H OBSERVE

Sample directory: df-252b

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-252b
INOVA-500 "nmroc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
16 repetitions
OBSERVE H1, 300.2230602 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 52 sec



5c

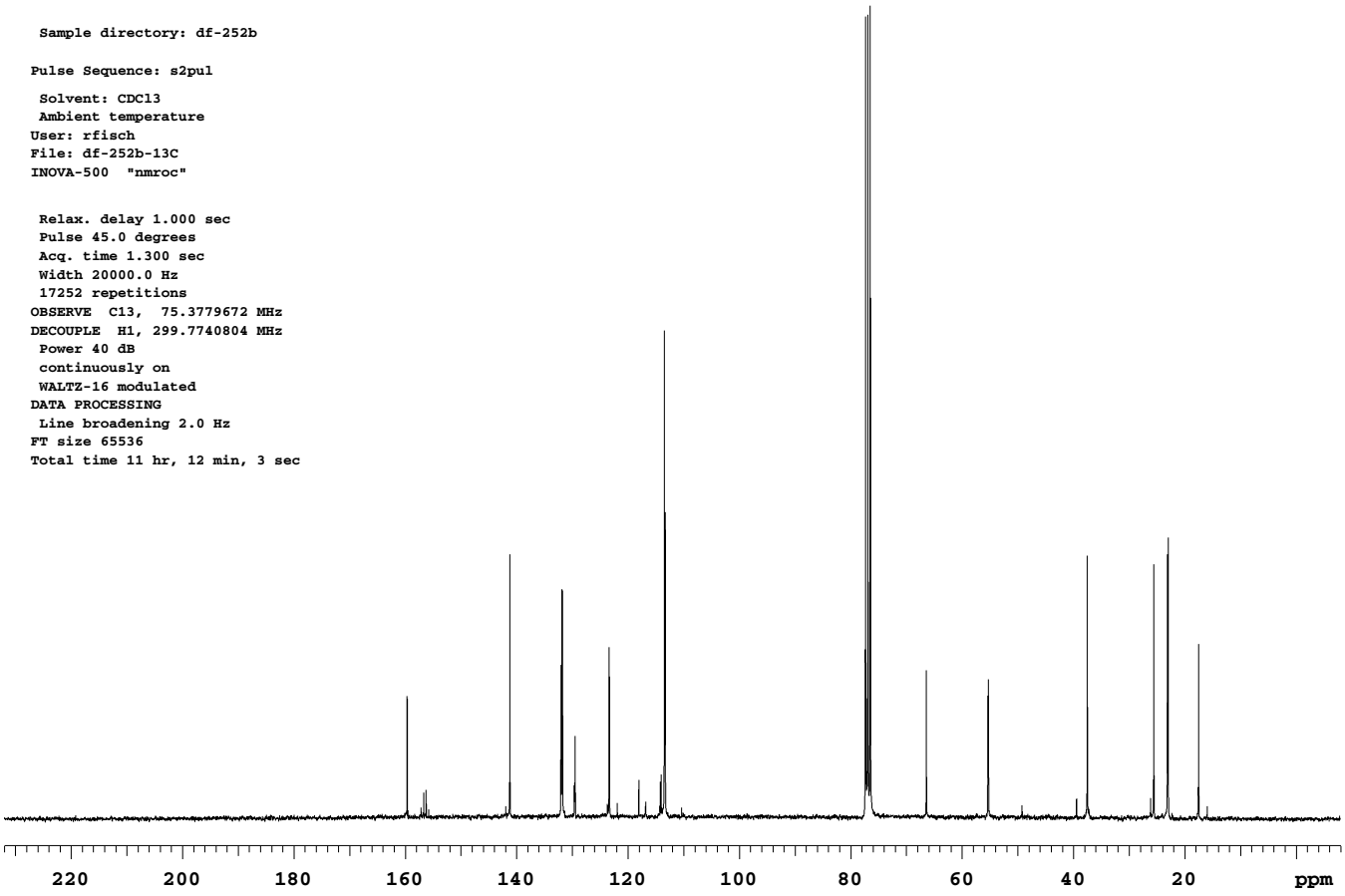
13C OBSERVE

Sample directory: df-252b

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-252b-13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
17252 repetitions
OBSERVE C13, 75.3779672 MHz
DECOUPLE H1, 299.7740804 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 11 hr, 12 min, 3 sec



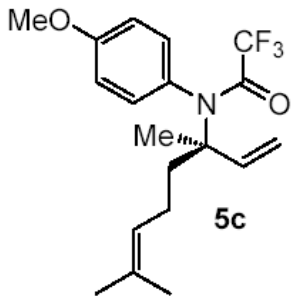
19F OBSERVE
STANDARD PARAMETERS

Sample directory: df-252b

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-252b_19F
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
32 repetitions
OBSERVE F19, 282.4918708 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 2 min, 40 sec



5c

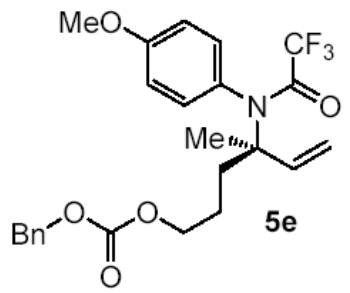
STANDARD 1H OBSERVE

Sample directory: X360PP

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X360PP
INOVA-500 "nmroc"

Pulse 29.5 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
88 repetitions
OBSERVE H1, 300.0836333 MHz
DATA PROCESSING
FT size 32768
Total time 4 min, 51 sec



5e

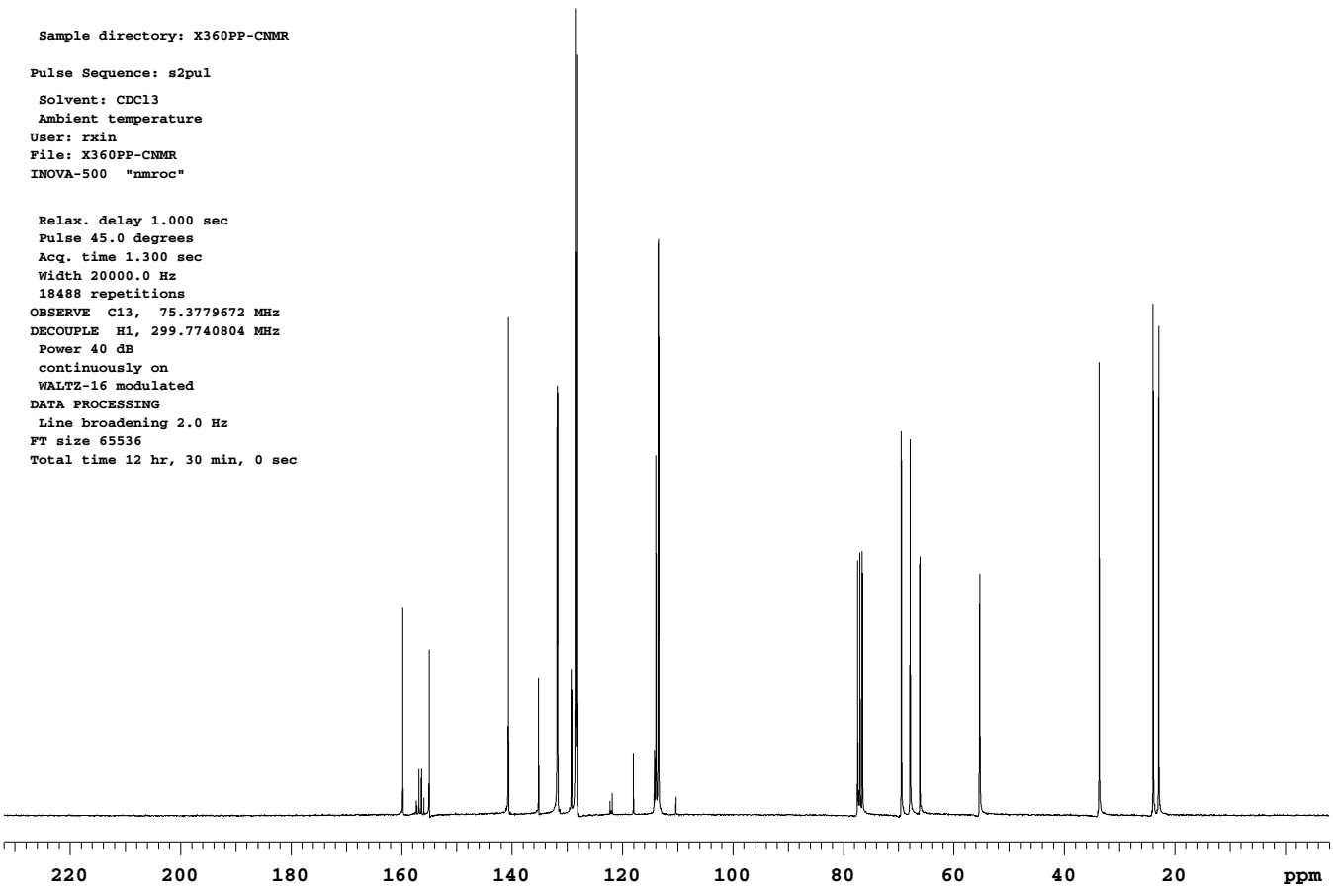
13C OBSERVE

Sample directory: X360PP-CNMR

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X360PP-CNMR
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
18488 repetitions
OBSERVE C13, 75.3779672 MHz
DECOUPLE H1, 299.7740804 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 12 hr, 30 min, 0 sec



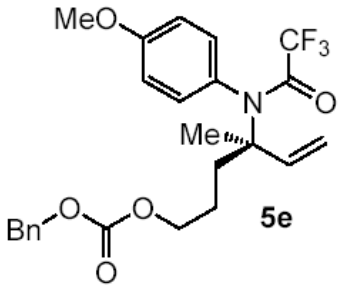
19F OBSERVE
STANDARD PARAMETERS

Sample directory: X360PP

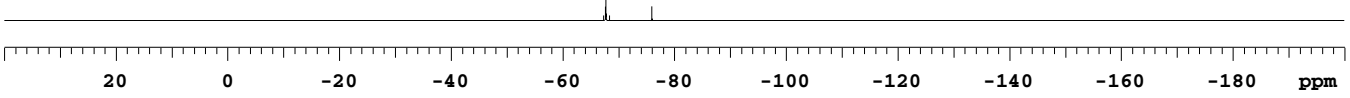
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X360PP-FNMR
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
38 repetitions
OBSERVE F19, 282.3608271 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 3 min, 13 sec



5e



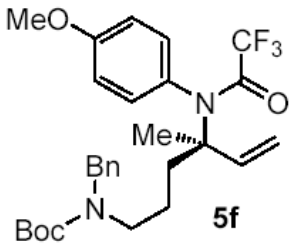
STANDARD 1H OBSERVE

Sample directory: X358PP

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X358PP
INOVA-500 "nmroc"

Pulse 29.5 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
88 repetitions
OBSERVE H1, 300.0836333 MHz
DATA PROCESSING
FT size 32768
Total time 4 min, 51 sec



5f

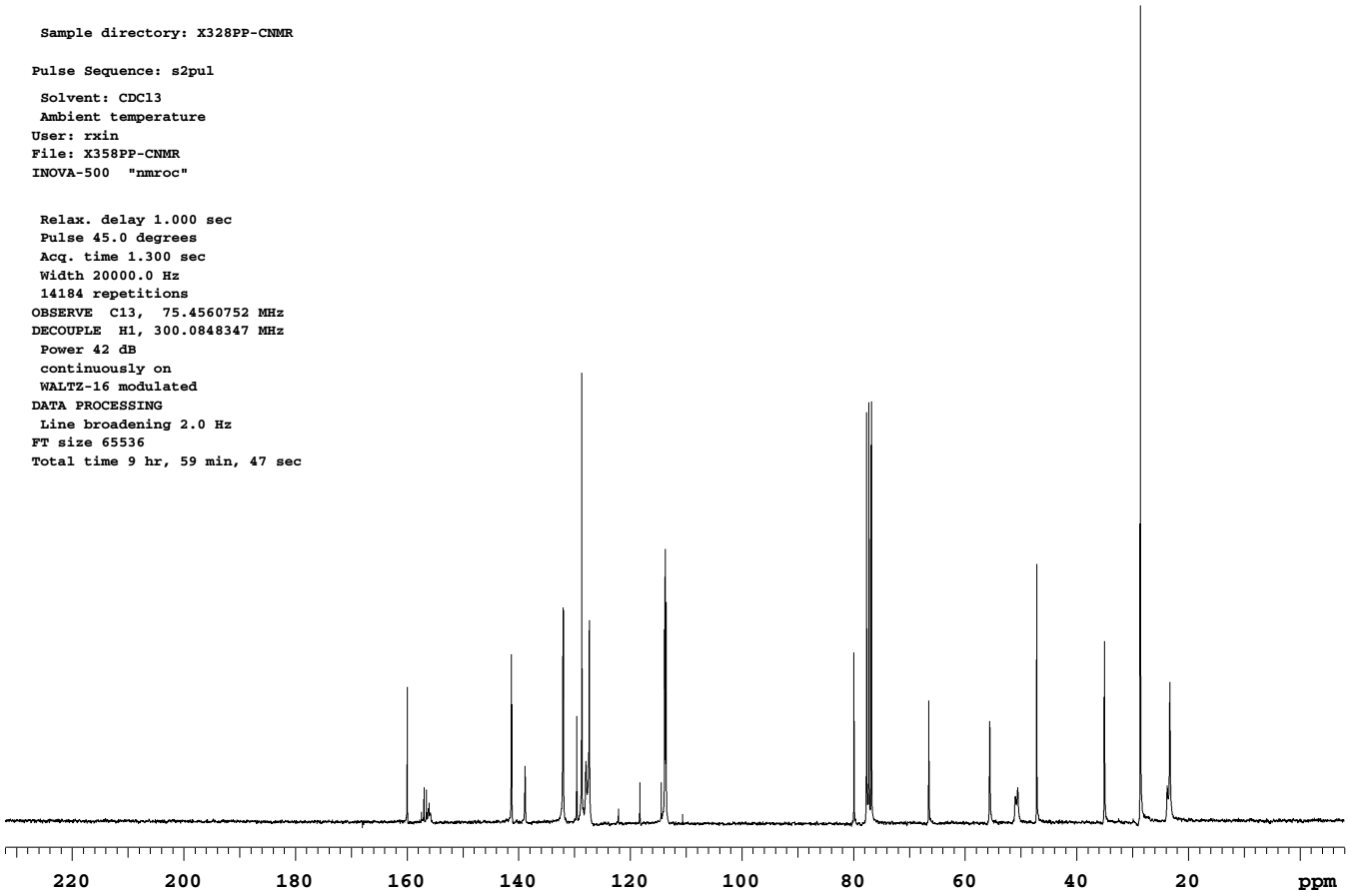
13C OBSERVE

Sample directory: X328PP-CNMR

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X358PP-CNMR
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
14184 repetitions
OBSERVE C13, 75.4560752 MHz
DECOUPLE H1, 300.0848347 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 9 hr, 59 min, 47 sec



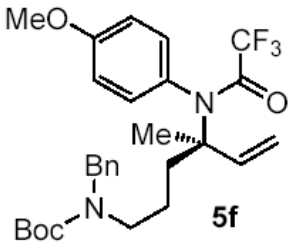
19F OBSERVE
STANDARD PARAMETERS

Sample directory: X358PP

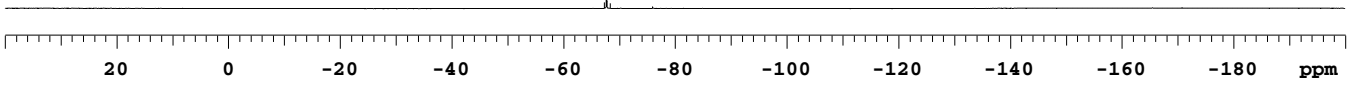
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X358PP-FNMR
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
32 repetitions
OBSERVE F19, 282.3608271 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 2 min, 40 sec



5f



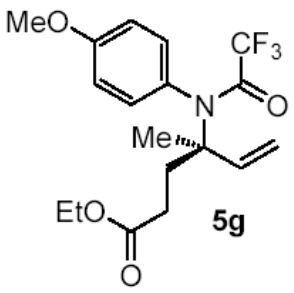
STANDARD 1H OBSERVE

Sample directory: df-308_umlagerung

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-308_rearr_1H
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 56.2 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
16 repetitions
OBSERVE H1, 299.7729174 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 49 sec



5g

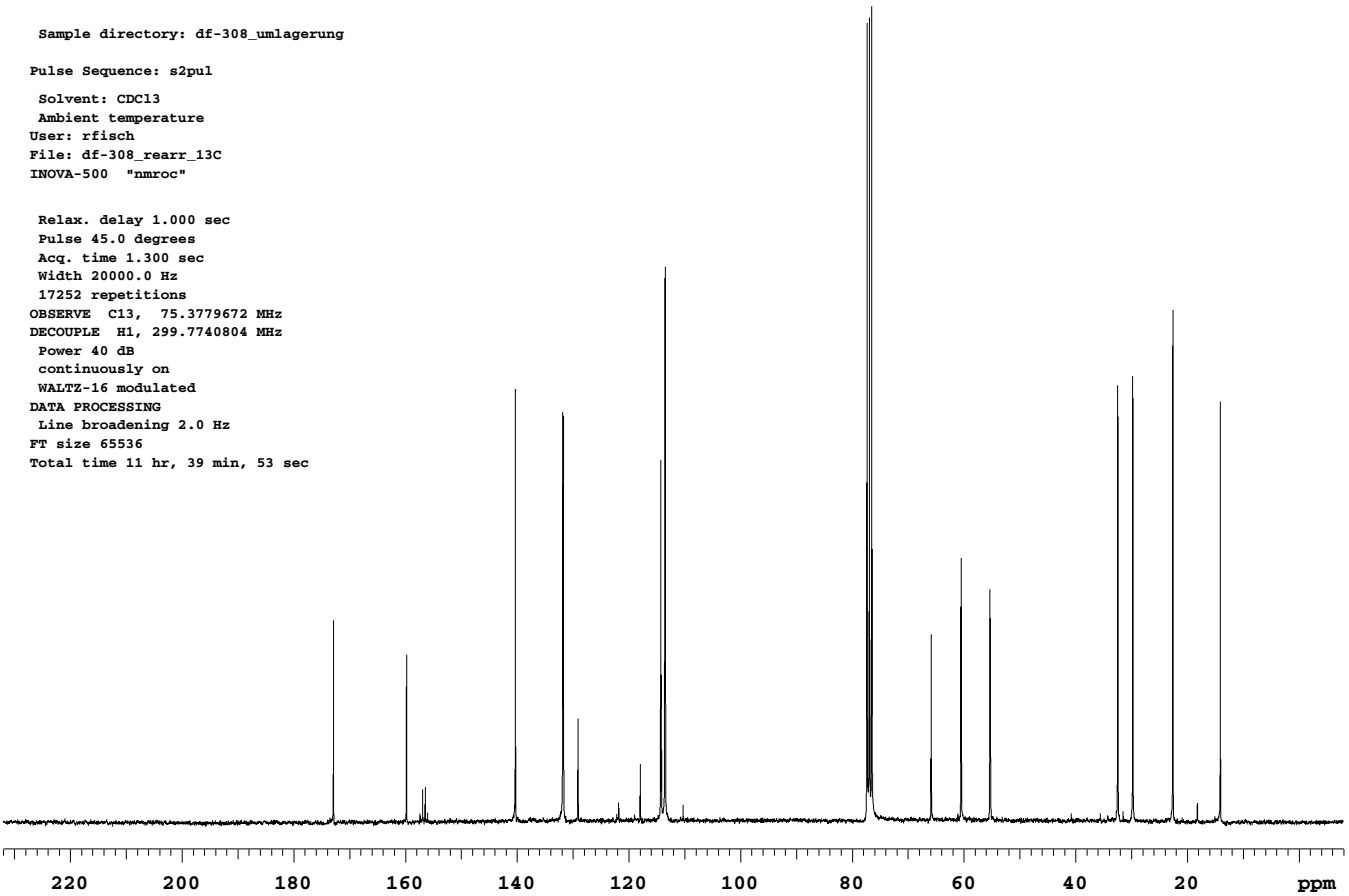
13C OBSERVE

Sample directory: df-308_umlagerung

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-308_rearr_13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
17252 repetitions
OBSERVE C13, 75.3779672 MHz
DECOUPLE H1, 299.7740804 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 11 hr, 39 min, 53 sec



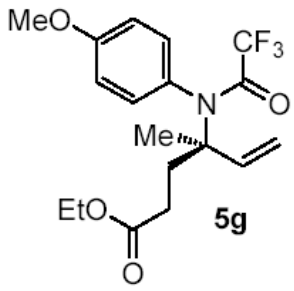
19F OBSERVE
STANDARD PARAMETERS

Sample directory: df-308_umlagerung

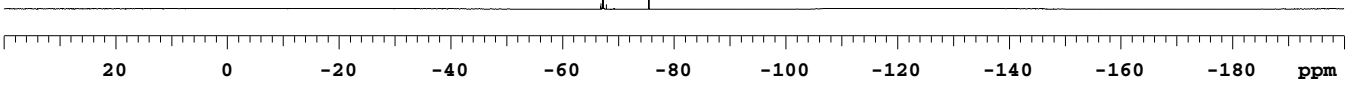
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-308_rearr_19F
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
16 repetitions
OBSERVE F19, 282.0683162 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 1 min, 20 sec



5g



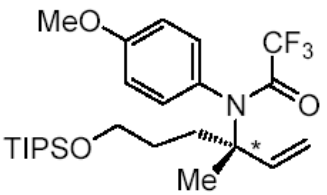
STANDARD 1H OBSERVE

Sample directory: df-314_rear

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-314_rear_1H
INOVA-500 "nmroc"

Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
16 repetitions
OBSERVE H1, 299.9012510 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 52 sec



5h

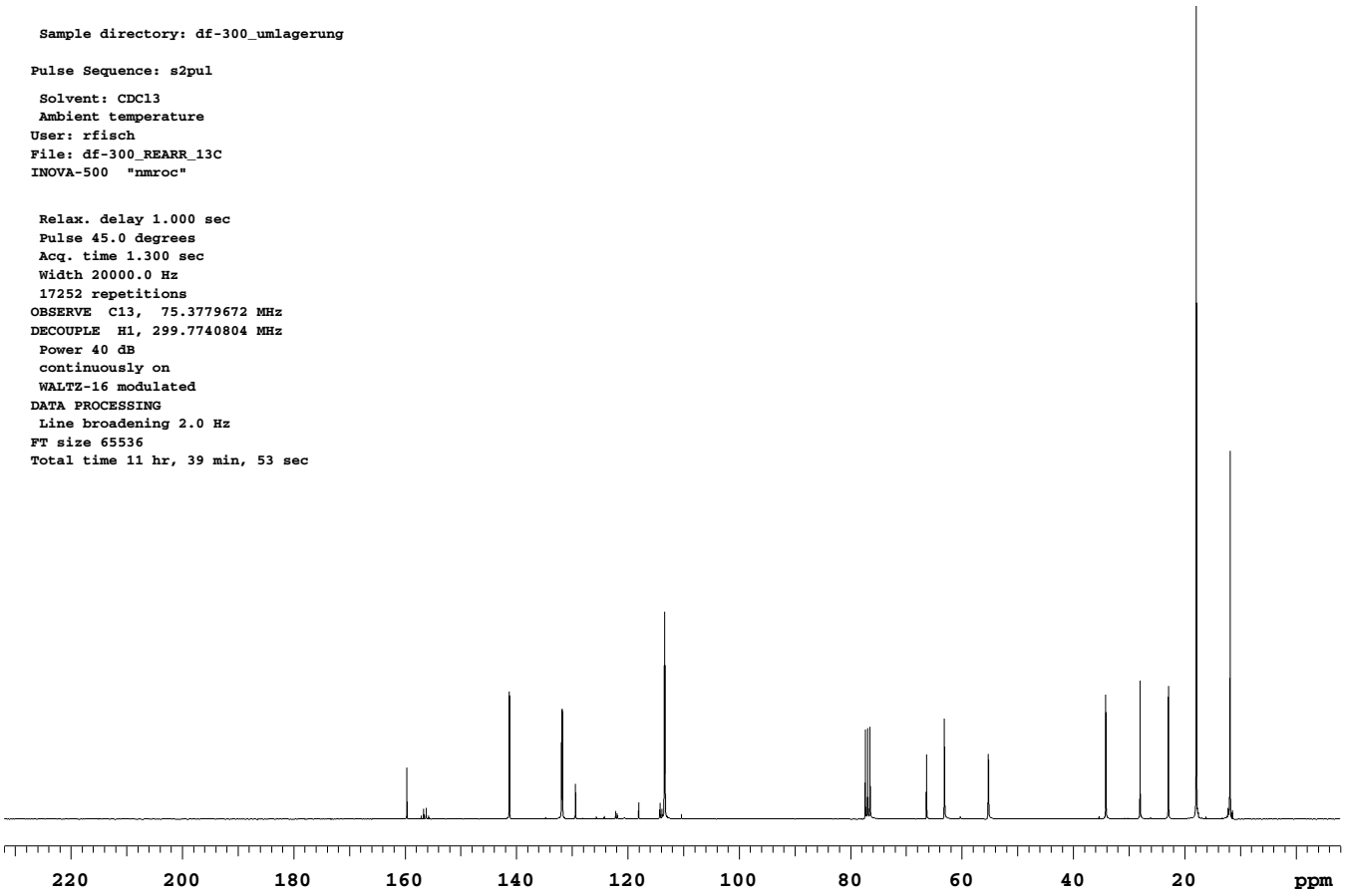
13C OBSERVE

Sample directory: df-300_umlagerung

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-300_REARR_13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
17252 repetitions
OBSERVE C13, 75.3779672 MHz
DECOUPLE H1, 299.7740804 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 11 hr, 39 min, 53 sec



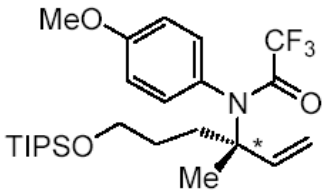
19F OBSERVE
STANDARD PARAMETERS

Sample directory: df-314_rear

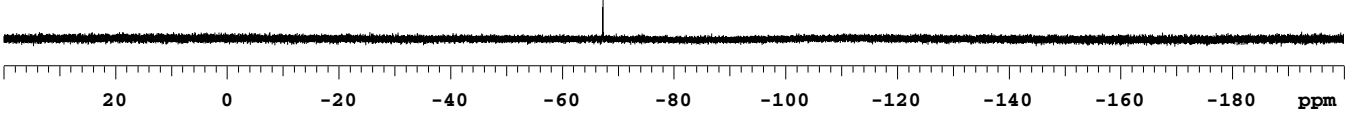
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-314_rear_19F
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
24 repetitions
OBSERVE F19, 282.1890544 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 2 min, 3 sec



5h



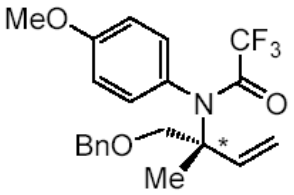
STANDARD 1H OBSERVE

Sample directory: X365PPF14

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X365PPF14
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 56.2 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
96 repetitions
OBSERVE H1, 299.7729174 MHz
DATA PROCESSING
FT size 32768
Total time 4 min, 58 sec



5i

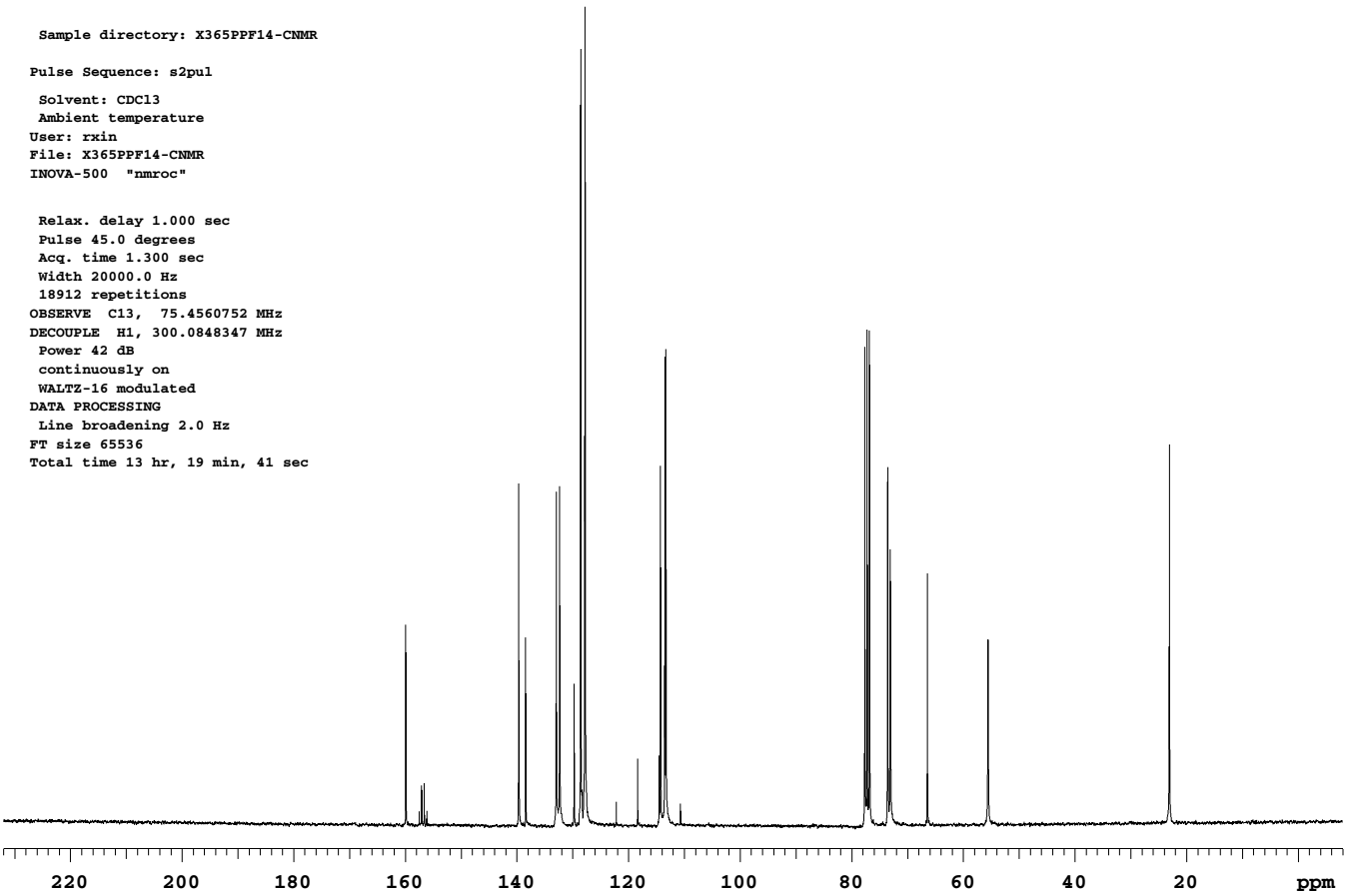
13C OBSERVE

Sample directory: X365PPF14-CNMR

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X365PPF14-CNMR
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
18912 repetitions
OBSERVE C13, 75.4560752 MHz
DECOUPLE H1, 300.0848347 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 13 hr, 19 min, 41 sec



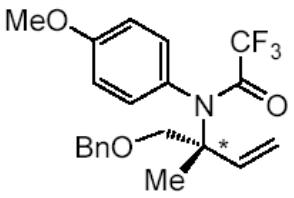
19F OBSERVE
STANDARD PARAMETERS

Sample directory: X365PPF14

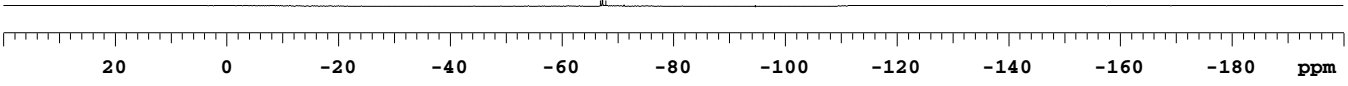
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X365PPF14-FNMR
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
19 repetitions
OBSERVE F19, 282.0683162 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 1 min, 39 sec



5i



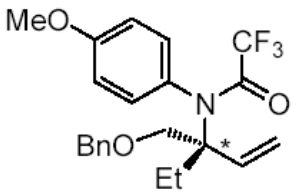
STANDARD 1H OBSERVE

Sample directory: X408PP2

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X408PP2
INOVA-500 "nmroc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
180 repetitions
OBSERVE H1, 300.2230602 MHz
DATA PROCESSING
FT size 32768
Total time 9 min, 55 sec



5j

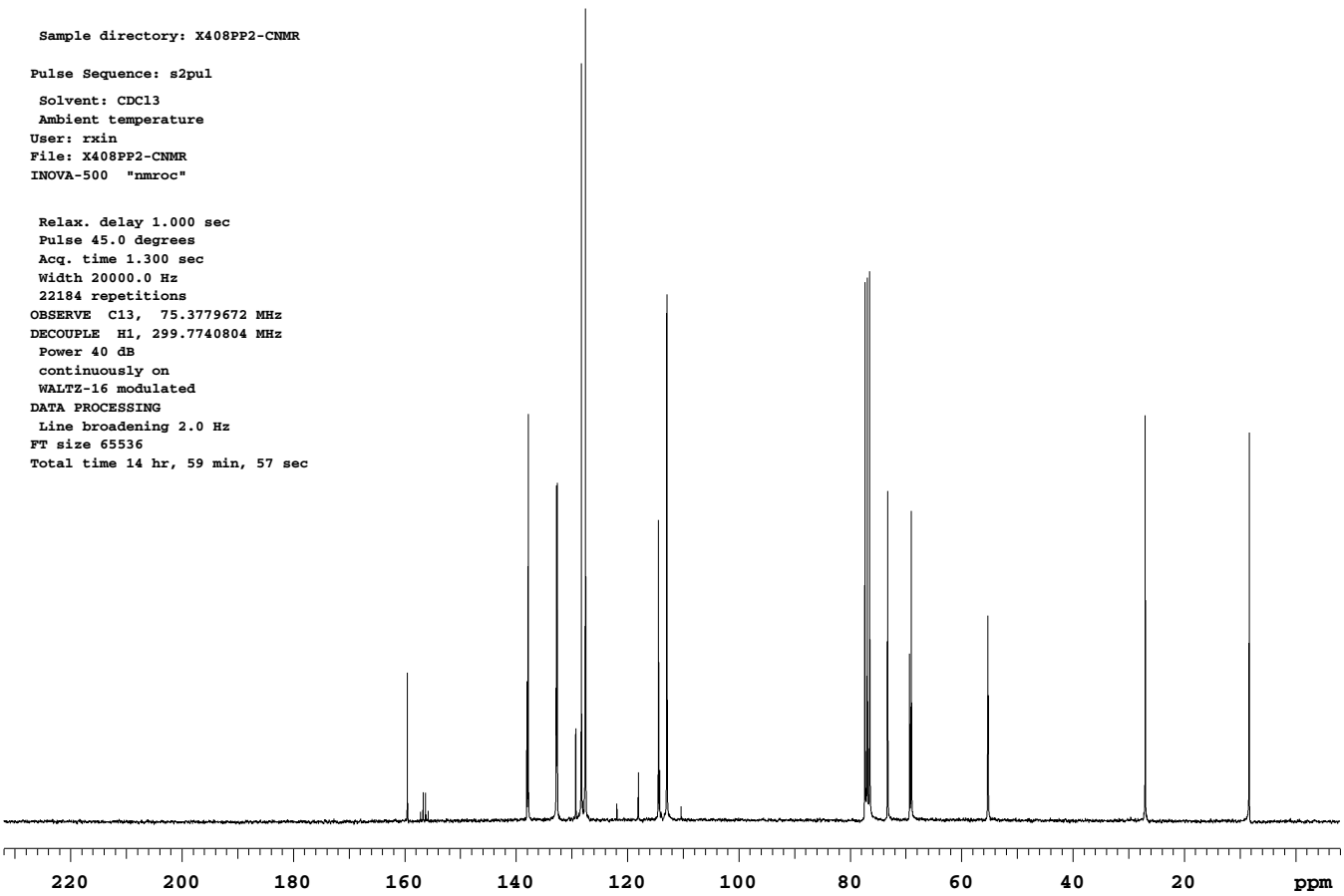
13C OBSERVE

Sample directory: X408PP2-CNMR

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X408PP2-CNMR
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
22184 repetitions
OBSERVE C13, 75.3779672 MHz
DECOUPLE H1, 299.7740804 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 14 hr, 59 min, 57 sec



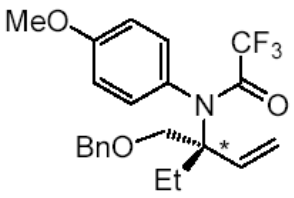
19F OBSERVE
STANDARD PARAMETERS

Sample directory: X408PP

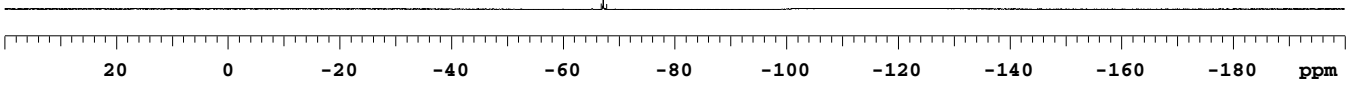
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X408PP1-FNMR
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
19 repetitions
OBSERVE F19, 282.4918708 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 1 min, 39 sec



5j



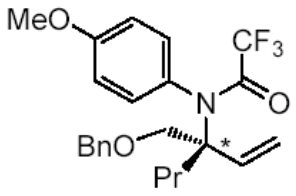
STANDARD 1H OBSERVE

Sample directory: df-339_Rear

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-339_rear_1H
INOVA-500 "nmroc"

Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
16 repetitions
OBSERVE H1, 299.9012510 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 52 sec



5k

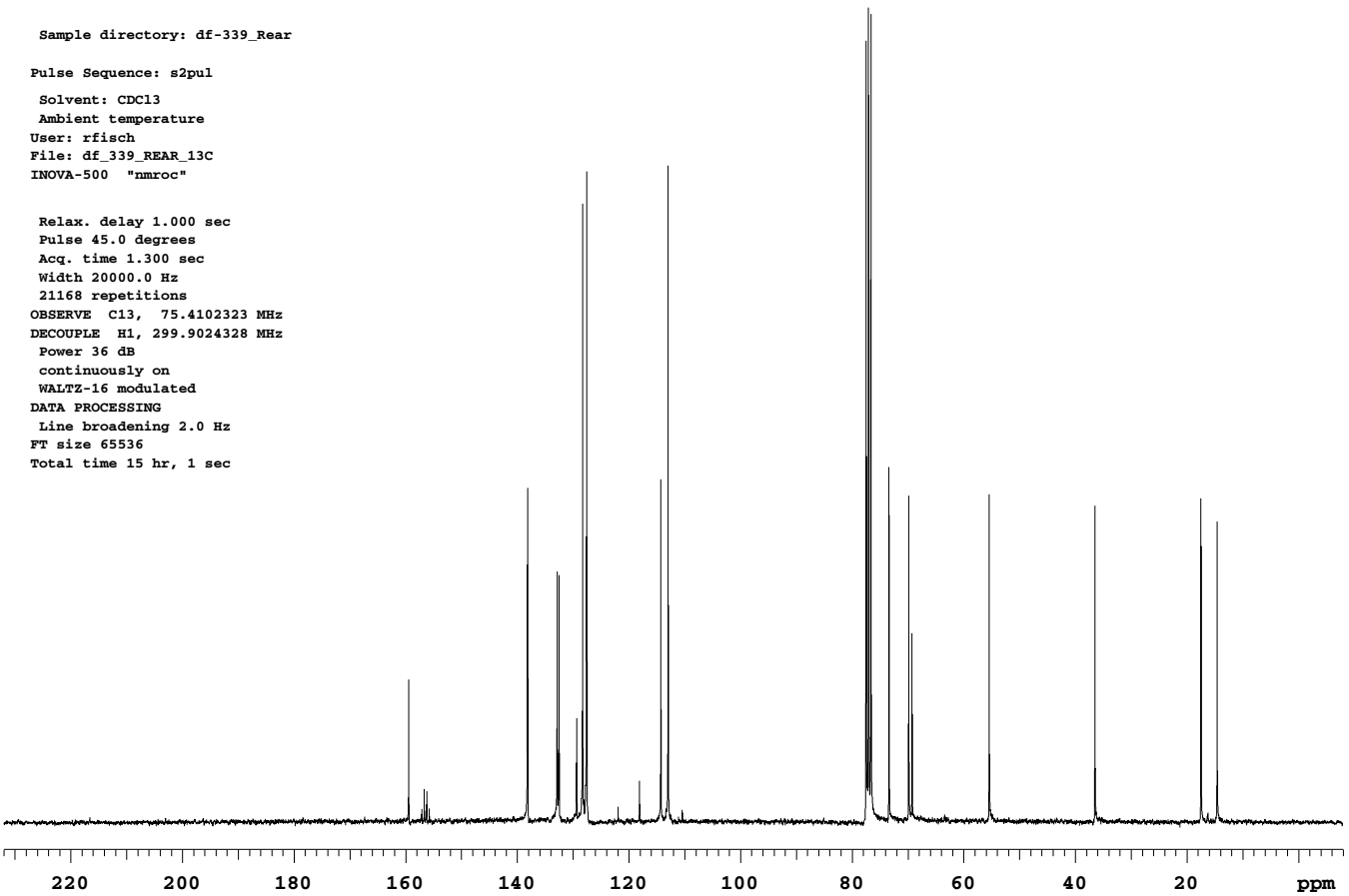
13C OBSERVE

Sample directory: df-339_Rear

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df_339_REAR_13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
21168 repetitions
OBSERVE C13, 75.4102323 MHz
DECOUPLE H1, 299.9024328 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 15 hr, 1 sec



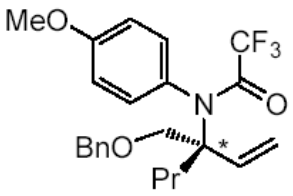
19F OBSERVE
STANDARD PARAMETERS

Sample directory: df-339_Rear

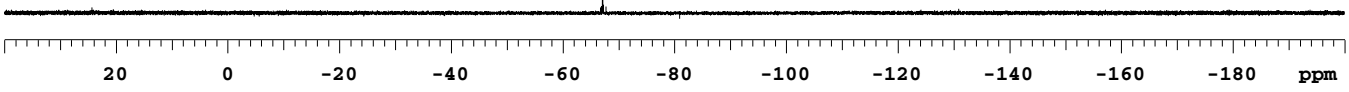
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-339_rear_19F
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
2 repetitions
OBSERVE F19, 282.1890544 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 0 min, 14 sec



5k



20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 ppm

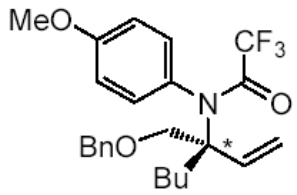
STANDARD 1H OBSERVE

Sample directory: df-337_Rear

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-337_Rear_1H
INOVA-500 "nmroc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
16 repetitions
OBSERVE H1, 300.2230602 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 52 sec



5l

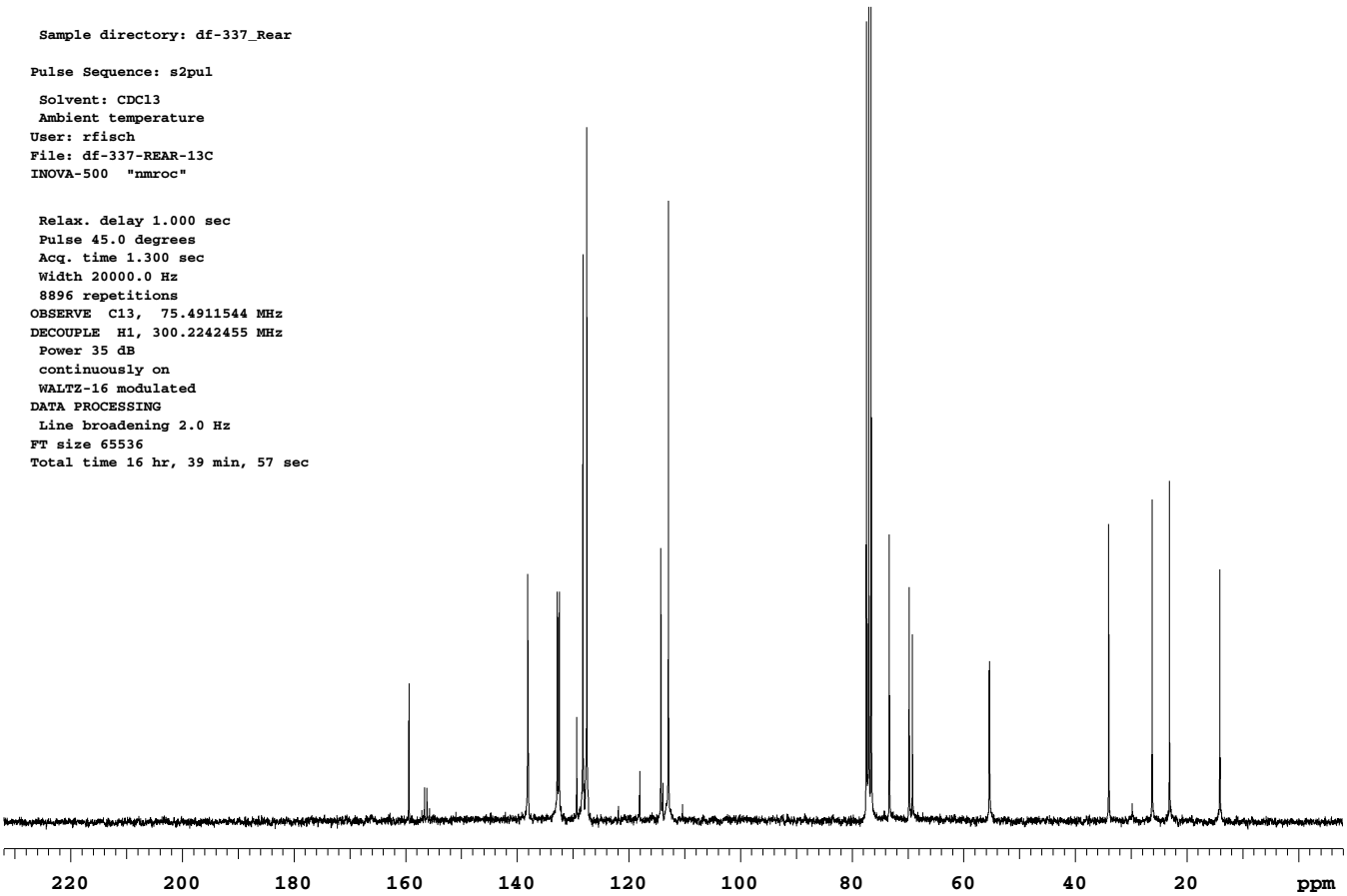
13C OBSERVE

Sample directory: df-337_Rear

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-337-REAR-13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
8896 repetitions
OBSERVE C13, 75.4911544 MHz
DECOUPLE H1, 300.2242455 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 16 hr, 39 min, 57 sec



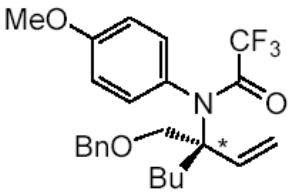
19F OBSERVE
STANDARD PARAMETERS

Sample directory: df-337_Rear

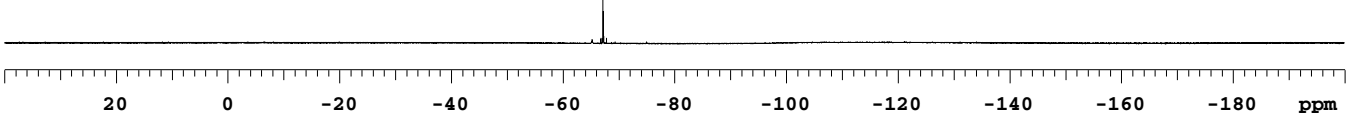
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-337_Rear_19F
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
16 repetitions
OBSERVE F19, 282.4918708 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 1 min, 20 sec



5l



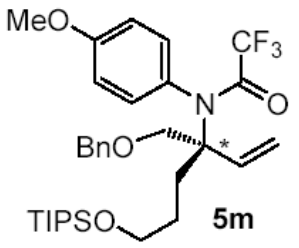
STANDARD 1H OBSERVE

Sample directory: X407PP2

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X407PP2
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 30.2 degrees
Acq. time 3.138 sec
Width 4500.5 Hz
68 repetitions
OBSERVE H1, 299.7729174 MHz
DATA PROCESSING
FT size 32768
Total time 4 min, 53 sec



5m

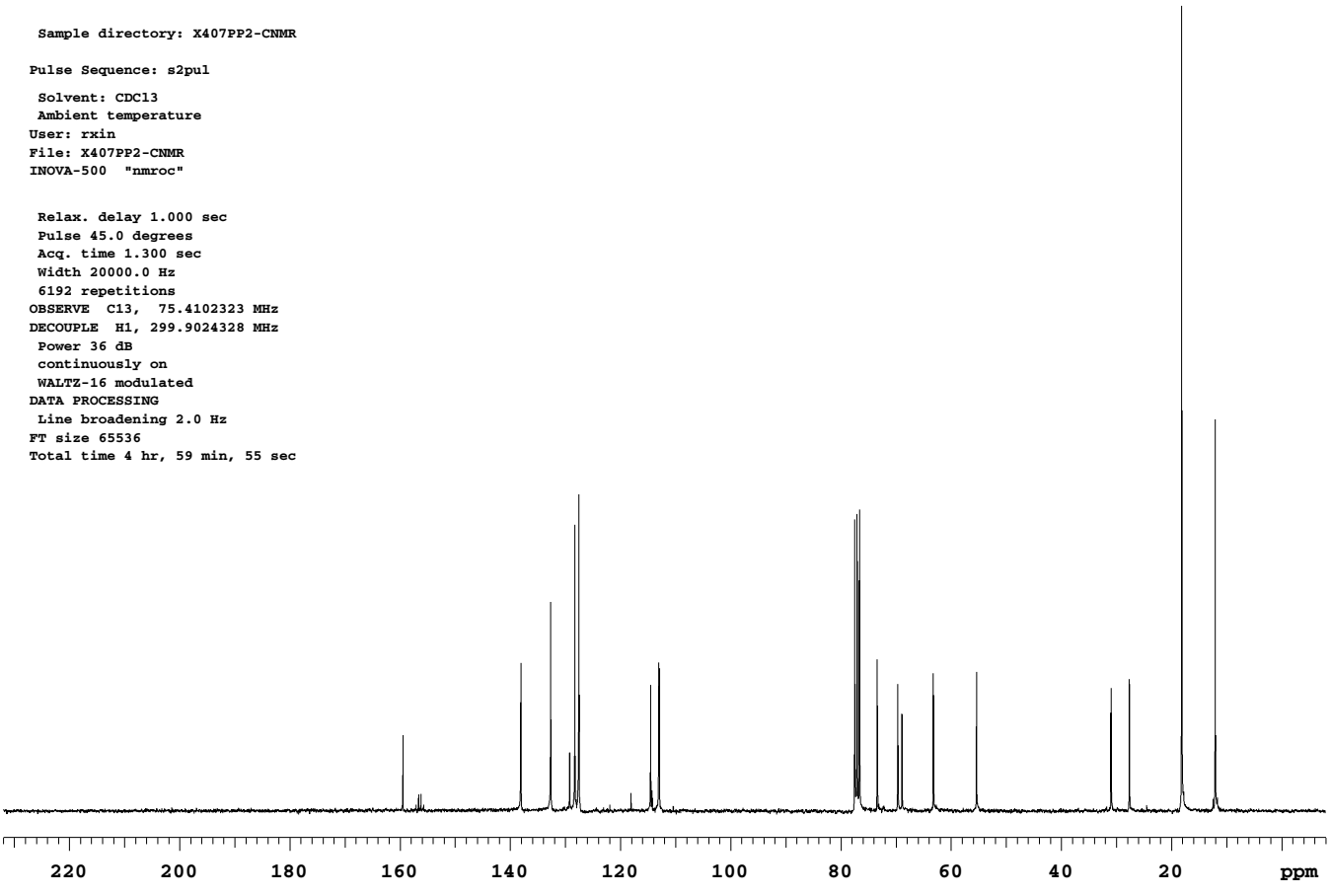
13C OBSERVE

Sample directory: X407PP2-CNMR

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X407PP2-CNMR
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
6192 repetitions
OBSERVE C13, 75.4102323 MHz
DECOUPLE H1, 299.9024328 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 4 hr, 59 min, 55 sec



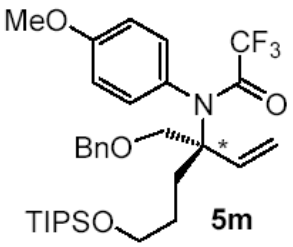
19F OBSERVE
STANDARD PARAMETERS

Sample directory: X407PP

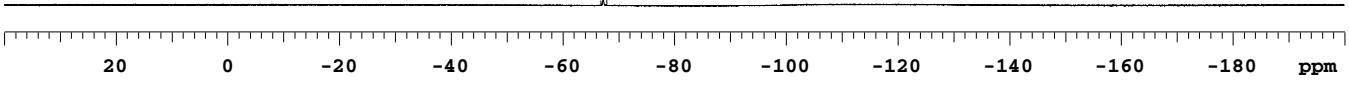
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X407PP1-FNMR
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
19 repetitions
OBSERVE F19, 282.4918708 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 1 min, 39 sec



5m



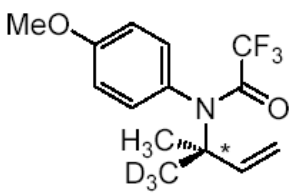
STANDARD 1H OBSERVE

Sample directory: df-343_Rear

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-343_Rear_1H
INOVA-500 "nmroc"

Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
16 repetitions
OBSERVE H1, 299.9012510 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 52 sec



5n

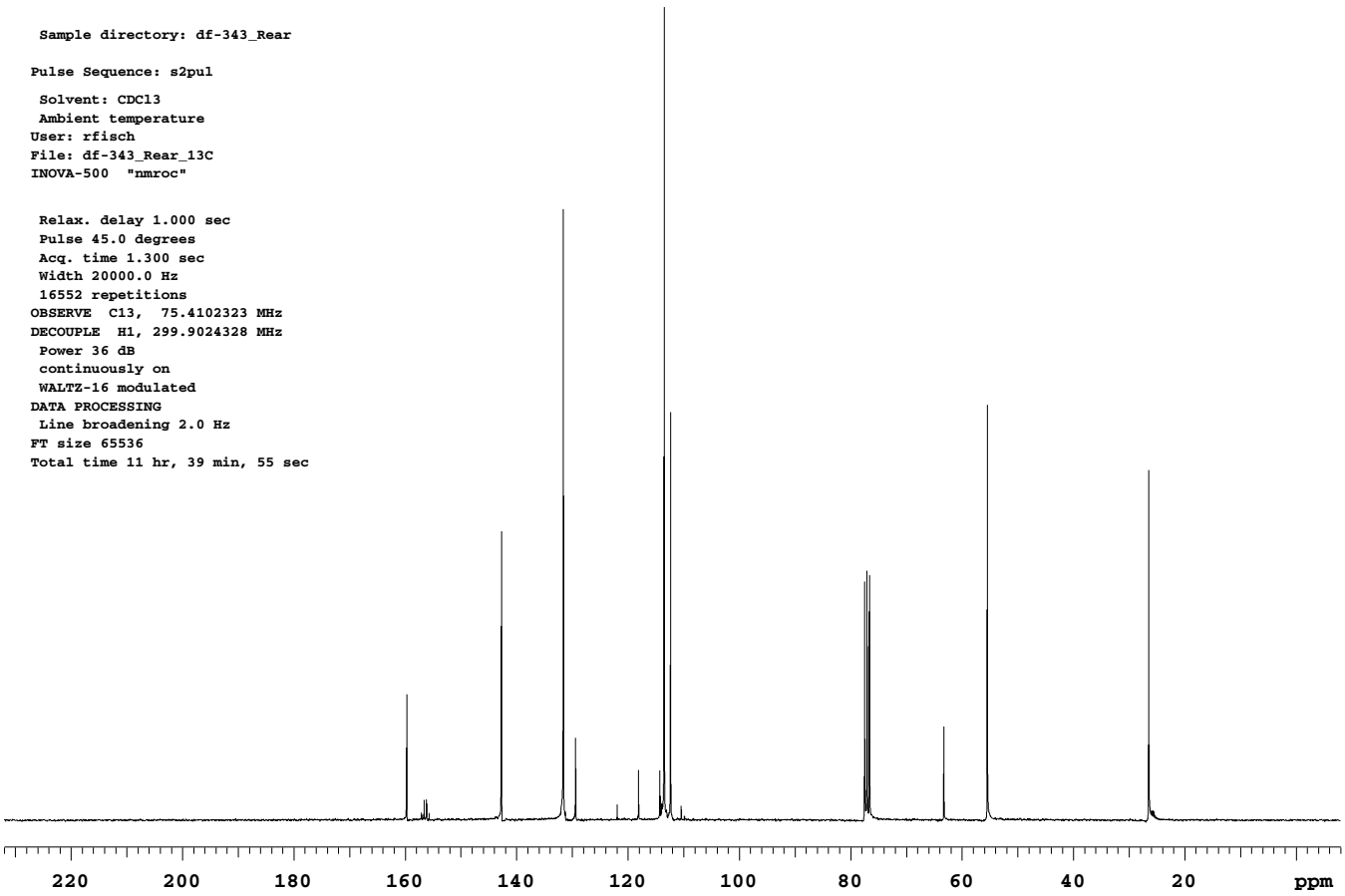
13C OBSERVE

Sample directory: df-343_Rear

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-343_Rear_13C
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
16552 repetitions
OBSERVE C13, 75.4102323 MHz
DECOUPLE H1, 299.9024328 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 11 hr, 39 min, 55 sec



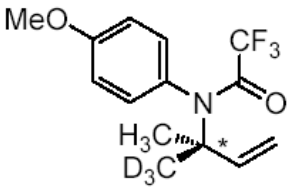
19F OBSERVE
STANDARD PARAMETERS

Sample directory: df-343_Rear

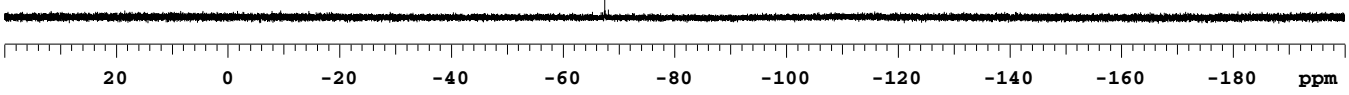
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rfisch
File: df-343_Rear_19F
INOVA-500 "nmroc"

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 0.640 sec
Width 69930.1 Hz
16 repetitions
OBSERVE F19, 282.1890544 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 1 min, 20 sec



5n



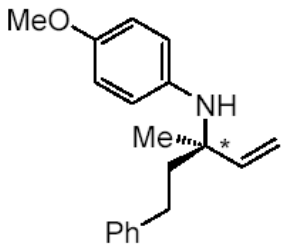
STANDARD 1H OBSERVE

Sample directory: X401PPF11

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X401PPF11
INOVA-500 "nmroc"

Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
88 repetitions
OBSERVE H1, 299.9012510 MHz
DATA PROCESSING
FT size 32768
Total time 4 min, 51 sec



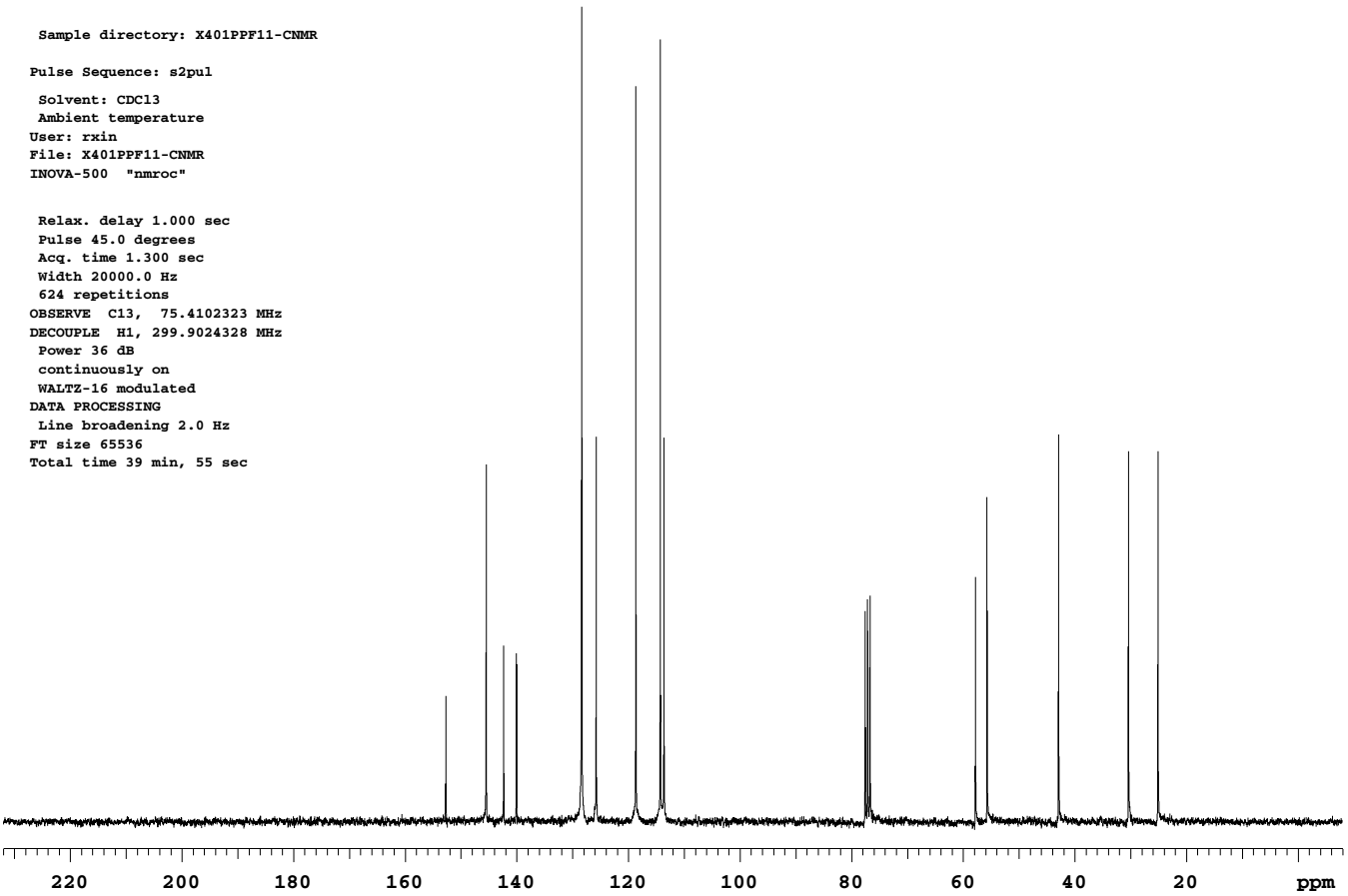
13C OBSERVE

Sample directory: X401PPF11-CNMR

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X401PPF11-CNMR
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
624 repetitions
OBSERVE C13, 75.4102323 MHz
DECOUPLE H1, 299.9024328 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 39 min, 55 sec



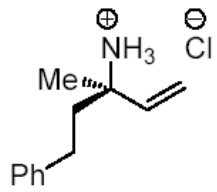
STANDARD 1H OBSERVE

Sample directory: X406PP1

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X406PP1
INOVA-500 "nmroc"

Pulse 29.5 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
52 repetitions
OBSERVE H1, 300.0836333 MHz
DATA PROCESSING
FT size 32768
Total time 2 min, 53 sec



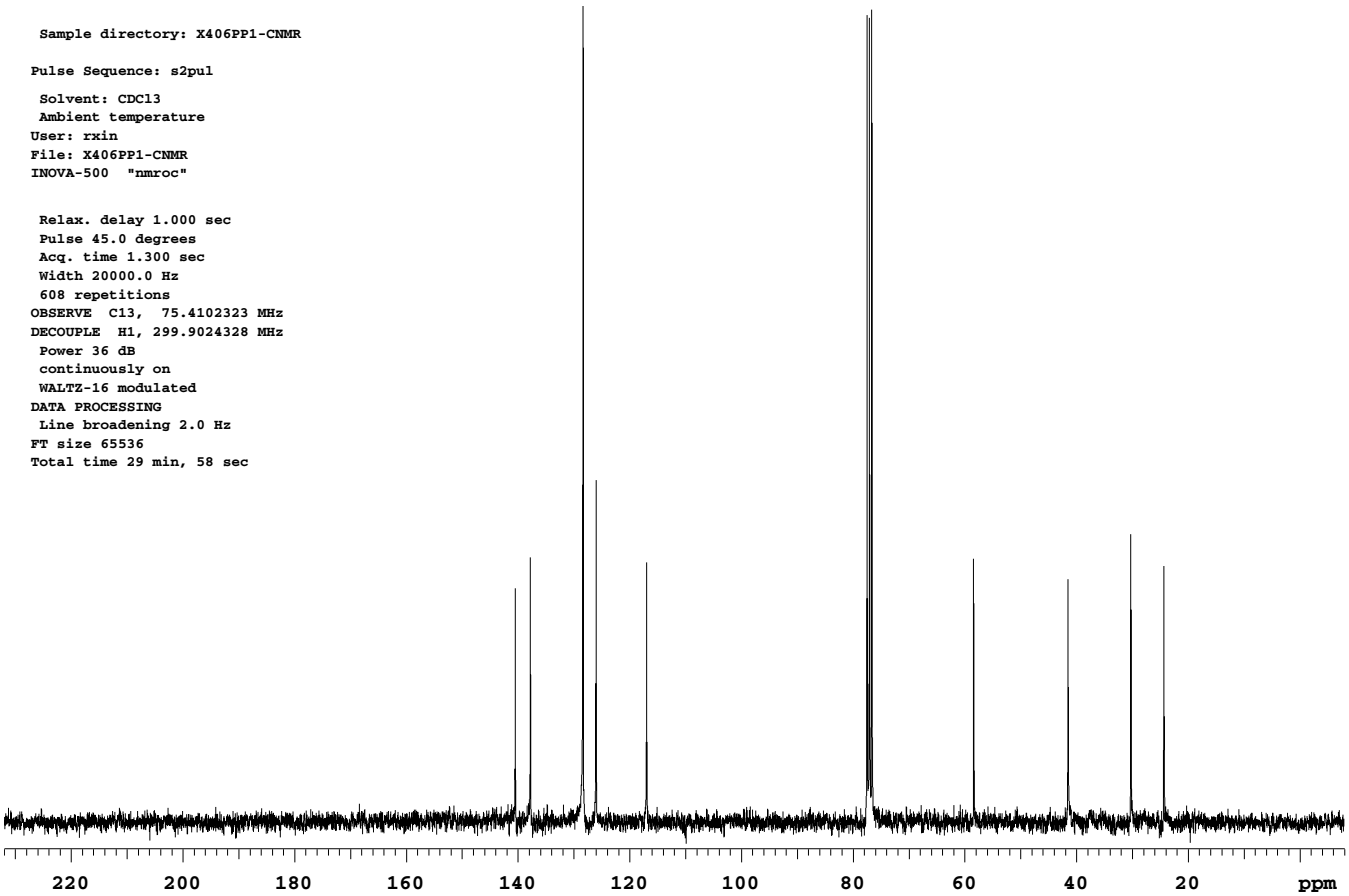
13C OBSERVE

Sample directory: X406PP1-CNMR

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X406PP1-CNMR
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
608 repetitions
OBSERVE C13, 75.4102323 MHz
DECOUPLE H1, 299.9024328 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 29 min, 58 sec



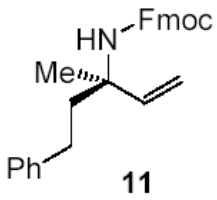
STANDARD 1H OBSERVE

Sample directory: X412PP3

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X412PP3
INOVA-500 "nmroc"

Pulse 29.5 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
32 repetitions
OBSERVE H1, 300.0836333 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 45 sec



11

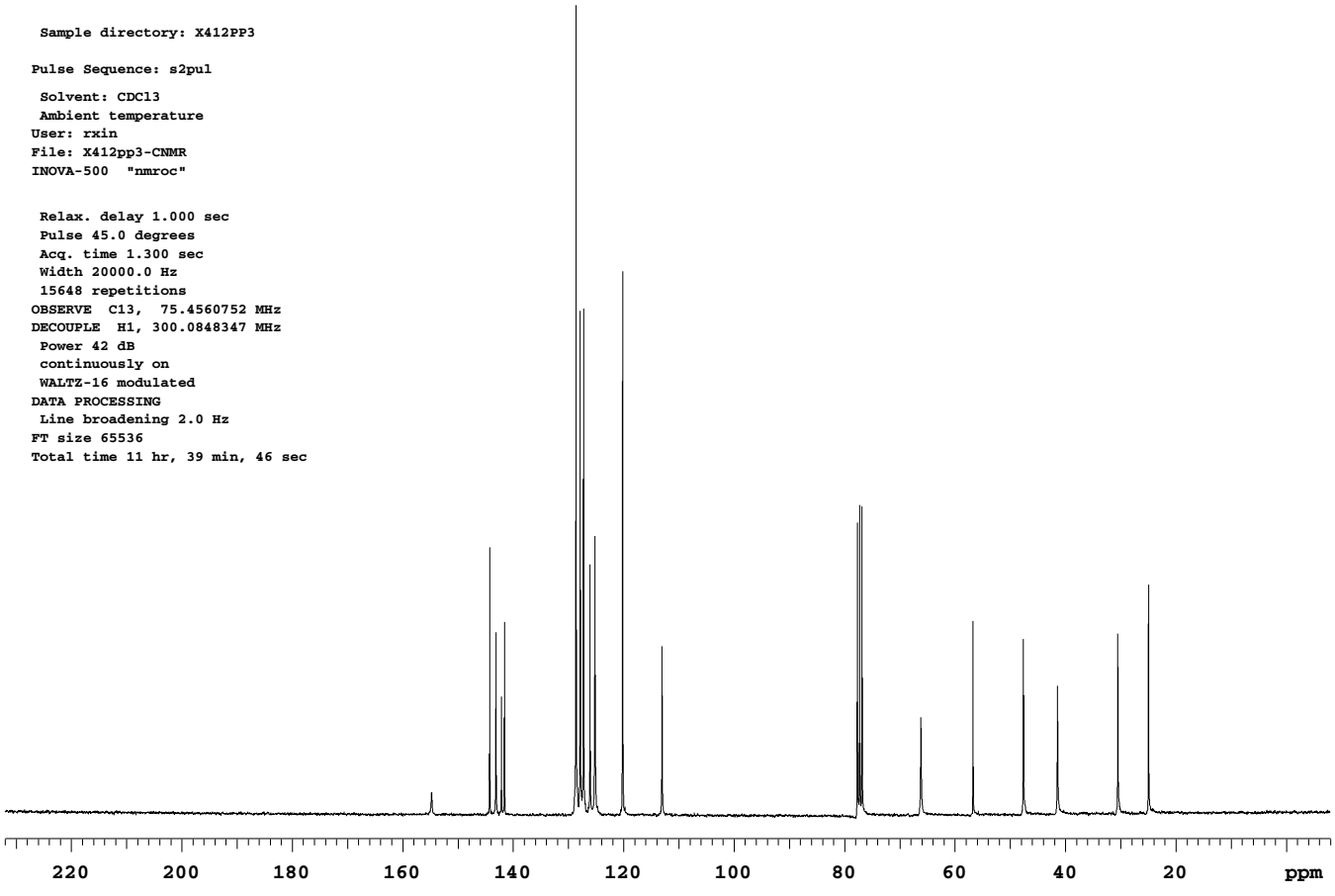
13C OBSERVE

Sample directory: X412PP3

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X412pp3-CNMR
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
15648 repetitions
OBSERVE C13, 75.4560752 MHz
DECOUPLE H1, 300.0848347 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 11 hr, 39 min, 46 sec



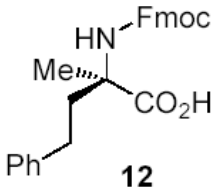
STANDARD 1H OBSERVE

Sample directory: X437PP-4-2

Pulse Sequence: s2pul

Solvent: CD3OD
Ambient temperature
User: rxin
File: X437PP-4-2
INOVA-500 "nmroc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
88 repetitions
OBSERVE H1, 300.2242441 MHz
DATA PROCESSING
FT size 32768
Total time 4 min, 51 sec



12

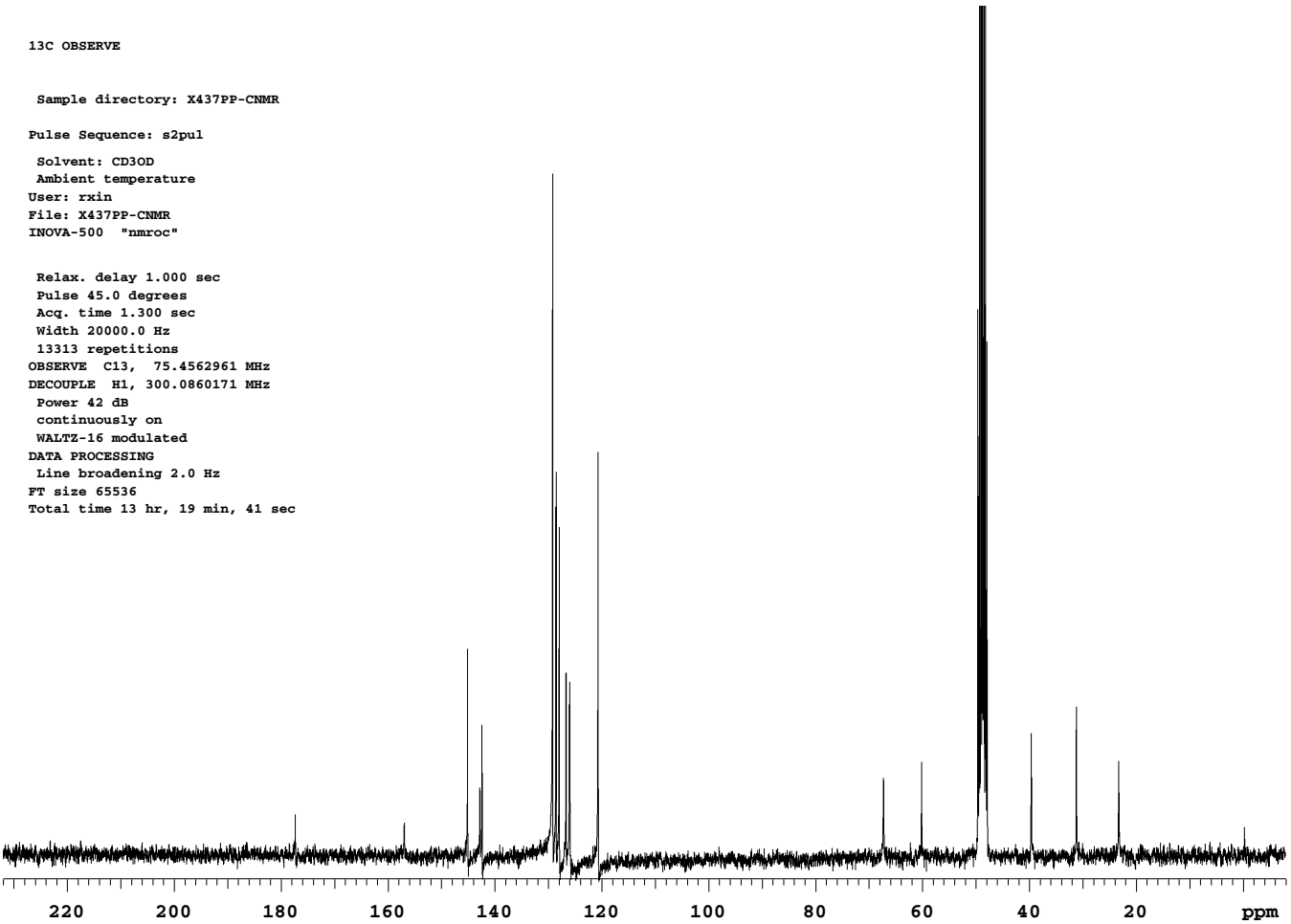
13C OBSERVE

Sample directory: X437PP-CNMR

Pulse Sequence: s2pul

Solvent: CD3OD
Ambient temperature
User: rxin
File: X437PP-CNMR
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
13313 repetitions
OBSERVE C13, 75.4562961 MHz
DECOUPLE H1, 300.0860171 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 13 hr, 19 min, 41 sec



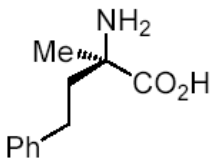
STANDARD 1H OBSERVE

Sample directory: X440PP

Pulse Sequence: s2pul

Solvent: CD3OD
Ambient temperature
User: rxin
File: X440PP
INOVA-500 "nmroc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
52 repetitions
OBSERVE H1, 300.2242441 MHz
DATA PROCESSING
FT size 32768
Total time 2 min, 53 sec



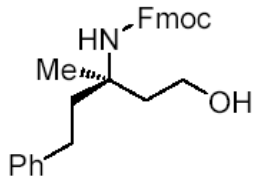
STANDARD 1H OBSERVE

Sample directory: X415PPF23

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X415PPF23-2
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 30.2 degrees
Acq. time 3.138 sec
Width 4500.5 Hz
8 repetitions
OBSERVE H1, 299.7729174 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 35 sec



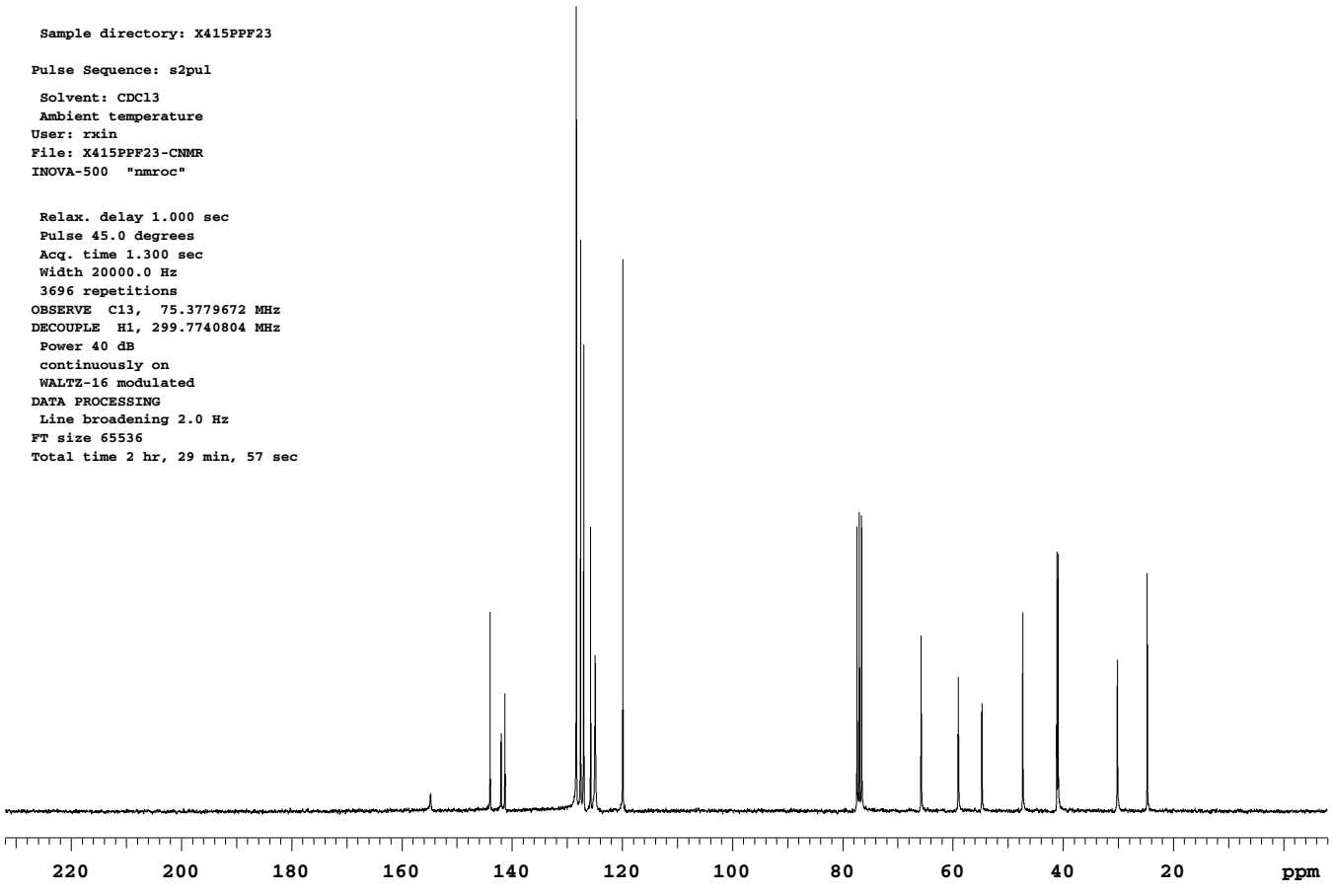
13C OBSERVE

Sample directory: X415PPF23

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X415PPF23-CNMR
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
3696 repetitions
OBSERVE C13, 75.3779672 MHz
DECOUPLE H1, 299.7740804 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 2 hr, 29 min, 57 sec



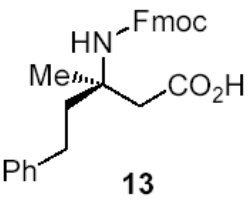
STANDARD 1H OBSERVE

Sample directory: X419PPF30-CNMR

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rxin
File: X419PPF30-3
INOVA-500 "nmroc"

Pulse 29.5 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
32 repetitions
OBSERVE H1, 300.0836333 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 45 sec



13

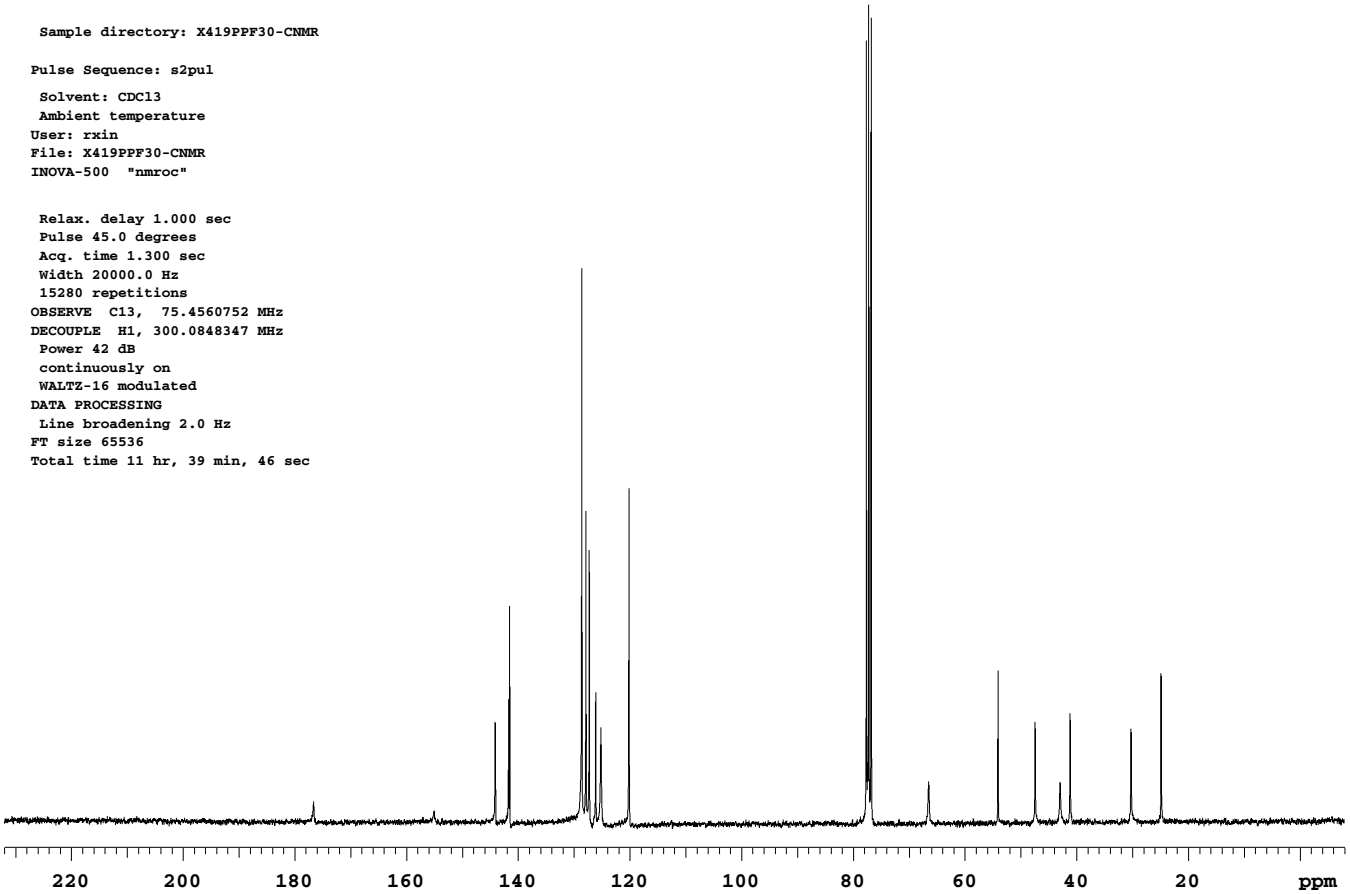
13C OBSERVE

Sample directory: X419PPF30-CNMR

Pulse Sequence: s2pul

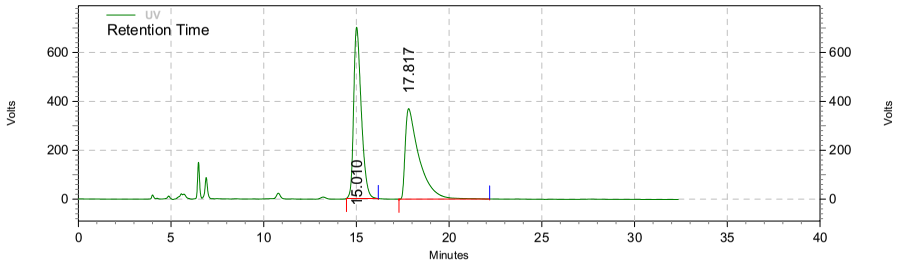
Solvent: CDCl3
Ambient temperature
User: rxin
File: X419PPF30-CNMR
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
15280 repetitions
OBSERVE C13, 75.4560752 MHz
DECOUPLE H1, 300.0848347 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 11 hr, 39 min, 46 sec



Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\New Folder\racemat_PhEt_daniel_99to1_210nm.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_99to1_210nm.met
Acquired: 1/16/2007 10:48:59 AM
Printed: 4/27/2007 10:24:24 AM

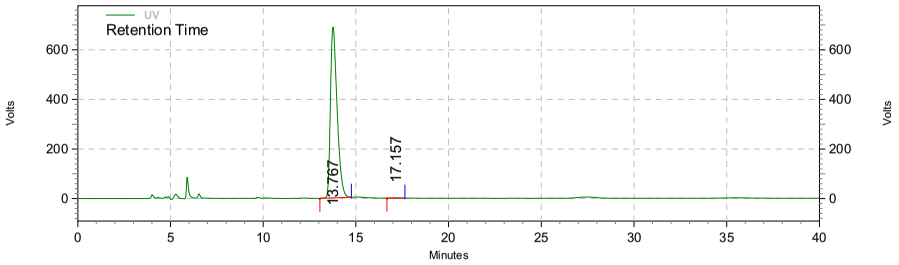


UV Results

Retention Time	Area	Area %	Height	Height %
15.010	79857039	51.02	2802977	65.41
17.817	76662328	48.98	1482187	34.59
Totals	156519367	100.00	4285164	100.00

Area % Report

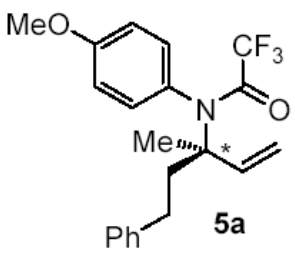
Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\New Folder\df-335A_2daniel_99to1_210nm.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_99to1_210nm.met
Acquired: 2/12/2007 4:21:42 PM
Printed: 4/27/2007 10:12:27 AM



UV Results

Retention Time	Area	Area %	Height	Height %
13.767	68724859	99.82	2750933	99.86
17.157	121274	0.18	3826	0.14
Totals	68846133	100.00	2754759	100.00

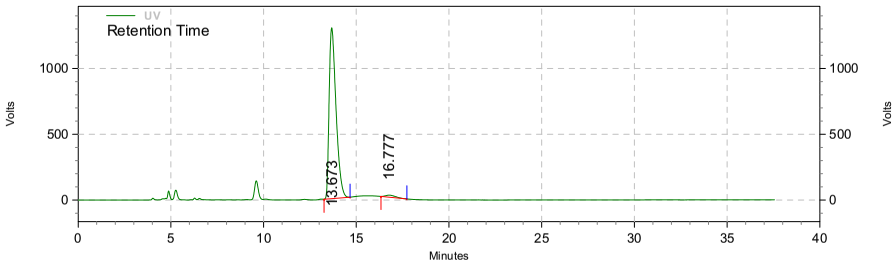
2 mol-% FIP-Cl



5a

Area % Report

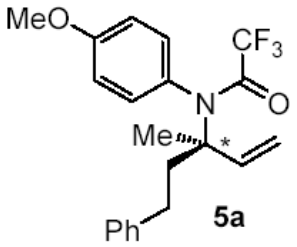
Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\New Folder\df-335G_verduennt_daniel_99to1_210nm.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_99to1_210nm.met
Acquired: 4/1/2007 6:08:39 PM
Printed: 4/27/2007 10:27:31 AM



UV Results

Retention Time	Area	Area %	Height	Height %
13.673	134315818	98.33	5191374	98.80
16.777	2278824	1.67	62799	1.20
Totals	136594642	100.00	5254173	100.00

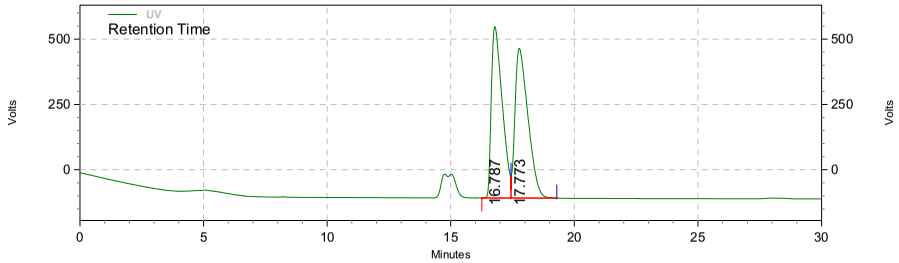
0.5 mol-% FIP-Cl



5a

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\Neu\Racemat_df-338_Bu-Me_daniel_99,8_210nm.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_99,8_210nm.met
Acquired: 5/10/2007 8:17:09 PM
Printed: 5/10/2007 9:16:39 PM

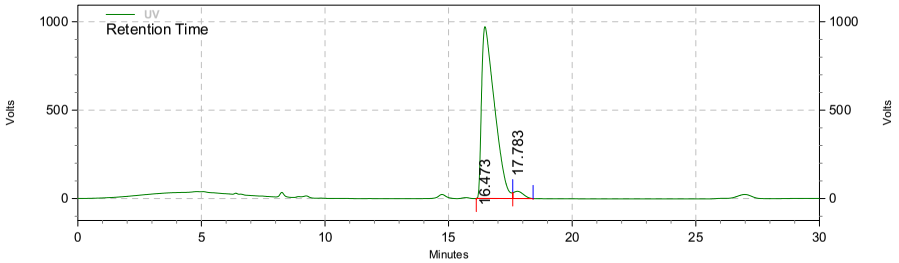


UV Results

Retention Time	Area	Area %	Height	Height %
16.787	80284810	48.98	2623344	53.37
17.773	83622884	51.02	2292224	46.63
Totals	163907694	100.00	4915568	100.00

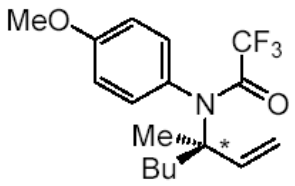
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\Neu\df-351Adaniel_99,8_210nm.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_99,8_210nm.met
Acquired: 5/10/2007 8:58:20 PM
Printed: 5/10/2007 9:33:12 PM



UV Results

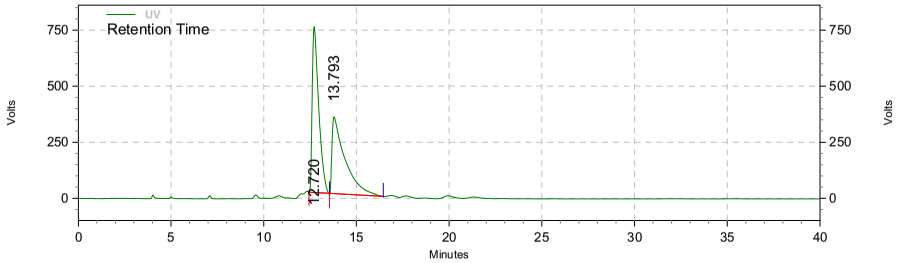
Retention Time	Area	Area %	Height	Height %
16.473	141752245	96.93	3886870	95.96
17.783	4484575	3.07	163530	4.04
Totals	146236820	100.00	4050400	100.00



5b

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\New Folder\racemat_geranioldaniel_99,8_210nm.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_99,8_210nm.met
Acquired: 1/16/2007 1:18:14 PM
Printed: 4/27/2007 10:42:32 AM

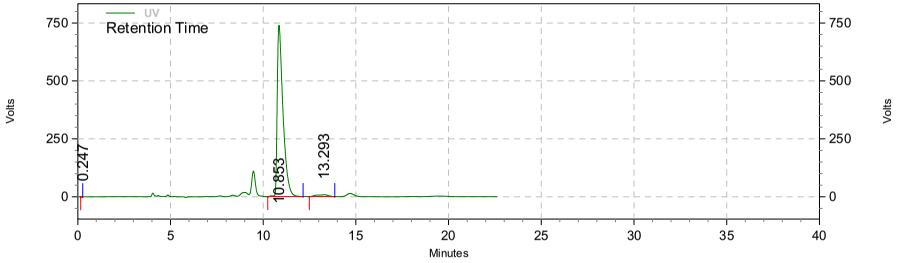


UV Results

Retention Time	Area	Area %	Height	Height %
12.720	71912491	51.83	2958043	68.34
13.793	66835831	48.17	1370268	31.66
Totals	138748322	100.00	4328311	100.00

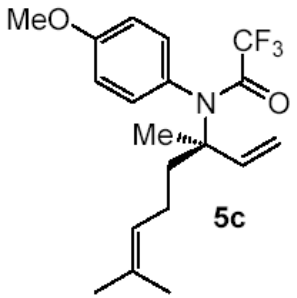
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\New Folder\df-237A_geraniol_2ndrundaniel_99,8_210nm.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_99,8_210nm.met
Acquired: 1/30/2007 3:32:35 PM
Printed: 4/27/2007 10:37:53 AM



UV Results

Retention Time	Area	Area %	Height	Height %
0.247	111	0.00	36	0.00
10.853	63900001	97.86	2957088	98.93
13.293	1398782	2.14	31813	1.06
Totals	65298894	100.00	2988937	100.00

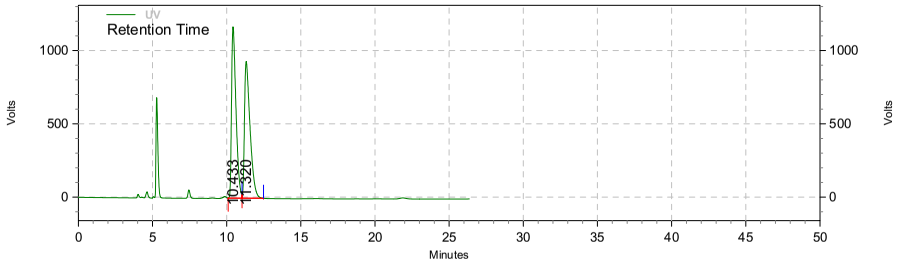


5c

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\New Folder\rac_TIPS_0,6promille_210nm_run1.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\test_0,6promille_iPrOH_210nm.met

Acquired: 11/22/2006 11:37:47 AM
Printed: 4/27/2007 10:57:57 AM



UV Results

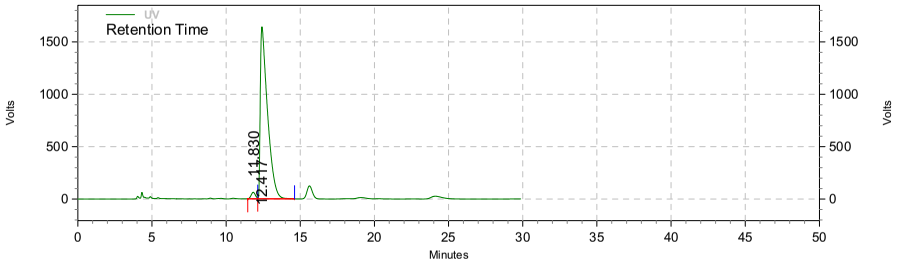
Retention Time	Area	Area %	Height	Height %
10.433	96953239	49.88	4674152	55.62
11.320	97435357	50.12	3730204	44.38
Totals	194388596	100.00	8404356	100.00

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\New Folder\df-303E_OTIPSdiluted.dat

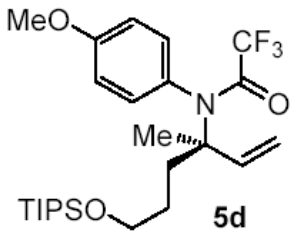
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\test_0,6promille_iPrOH_210nm.met

Acquired: 11/22/2006 3:37:41 PM
Printed: 4/27/2007 11:37:52 AM



UV Results

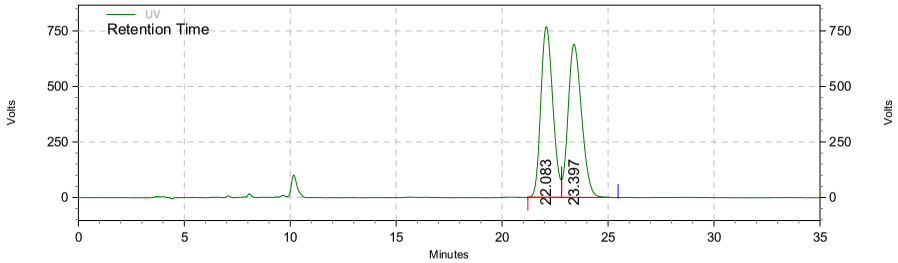
Retention Time	Area	Area %	Height	Height %
11.830	4956860	2.18	249558	3.66
12.417	222601582	97.82	6566214	96.34
Totals	227558442	100.00	6815772	100.00



5d

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\New Folder\racemat_carbonatdaniel-97to3-210nm.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel-97to3-210nm.met
Acquired: 5/9/2007 3:32:12 PM
Printed: 5/9/2007 5:23:49 PM

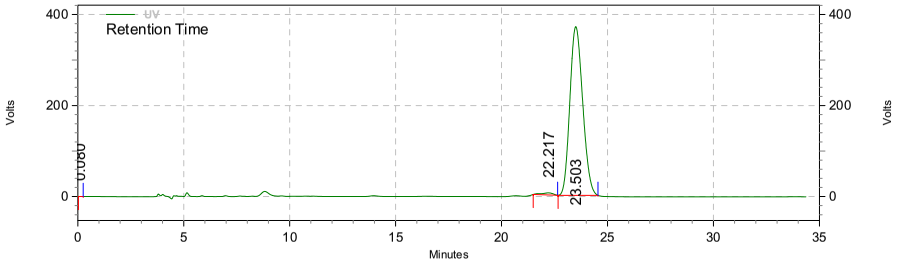


UV Results

Retention Time	Area	Area %	Height	Height %
22.083	120930818	49.66	3075220	52.72
23.397	122598823	50.34	2758178	47.28
Totals	243529641	100.00	5833398	100.00

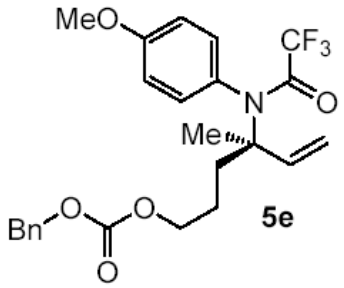
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\New Folder\df-351T2_daniel-97to3-210nm.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel-97to3-210nm.met
Acquired: 5/9/2007 4:57:18 PM
Printed: 5/9/2007 5:31:29 PM



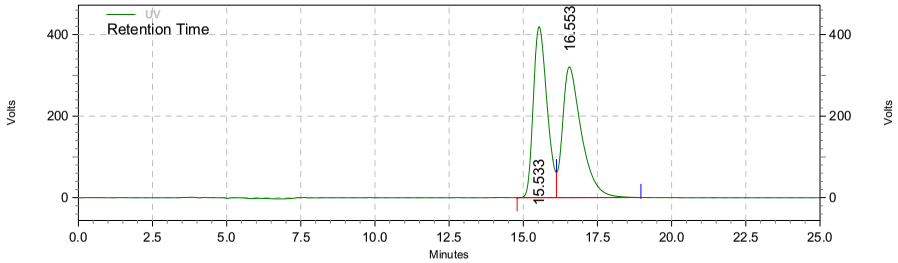
UV Results

Retention Time	Area	Area %	Height	Height %
0.080	729	0.00	95	0.01
22.217	711874	1.13	18400	1.23
23.503	62253714	98.87	1482372	98.77
Totals	62966317	100.00	1500867	100.00



Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\Neu\racemat_BnBoc-Medaniel_99to1_210nm.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_99to1_210nm.met
Acquired: 5/11/2007 2:05:59 PM
Printed: 5/11/2007 3:40:48 PM

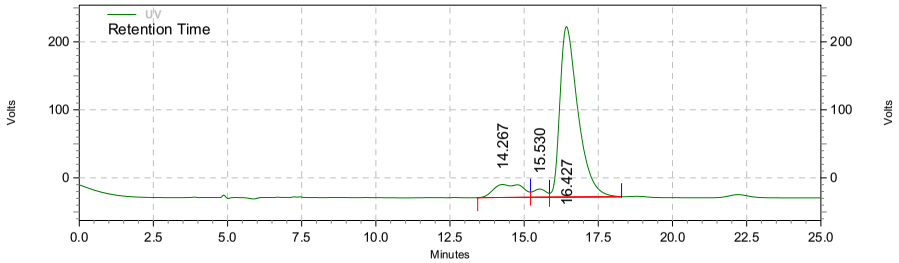


UV Results

Retention Time	Area	Area %	Height	Height %
15.533	53867890	48.73	1676699	56.69
16.553	56666321	51.27	1281033	43.31
Totals	110534211	100.00	2957732	100.00

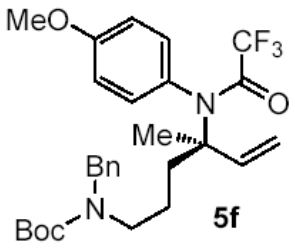
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\Neu\df-351Uc_verd_daniel_99to1_210nm.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_99to1_210nm.met
Acquired: 5/11/2007 3:18:22 PM
Printed: 5/11/2007 3:43:19 PM



UV Results

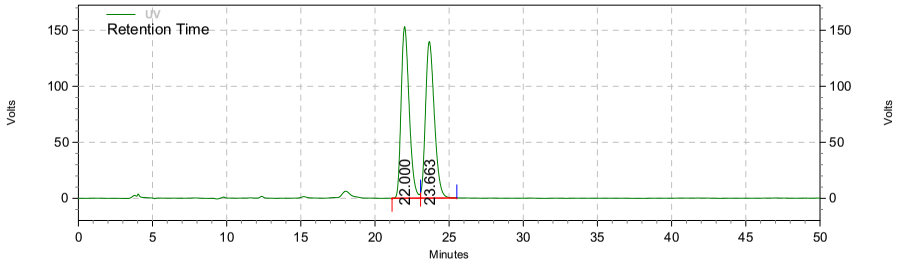
Retention Time	Area	Area %	Height	Height %
14.267	5245379	10.66	76377	6.78
15.530	1461627	2.97	48283	4.29
16.427	42491277	86.37	1002105	88.94
Totals	49198283	100.00	1126765	100.00



5f

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\New Folder\racemat_Ester_090507_daniel_99,5_to_0,5_210nm.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_99,5_to_0,5_210nm.met
Acquired: 5/9/2007 12:19:52 PM
Printed: 5/9/2007 1:12:58 PM

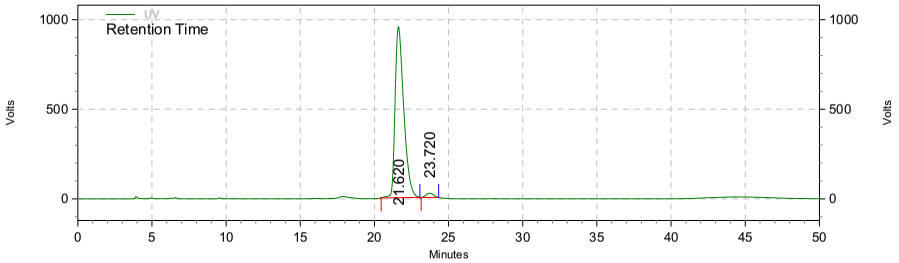


UV Results

Retention Time	Area	Area %	Height	Height %
22.000	23099694	49.96	612343	52.27
23.663	23133742	50.04	559109	47.73
Totals	46233436	100.00	1171452	100.00

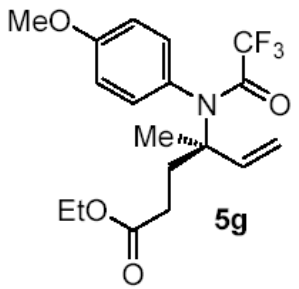
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\New Folder\df-351C_daniel_99,5_to_0,5_210nm.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_99,5_to_0,5_210nm.met
Acquired: 5/9/2007 1:11:10 PM
Printed: 5/9/2007 2:01:56 PM



UV Results

Retention Time	Area	Area %	Height	Height %
21.620	155084950	97.90	3817074	97.58
23.720	3319910	2.10	94839	2.42
Totals	158404860	100.00	3911913	100.00

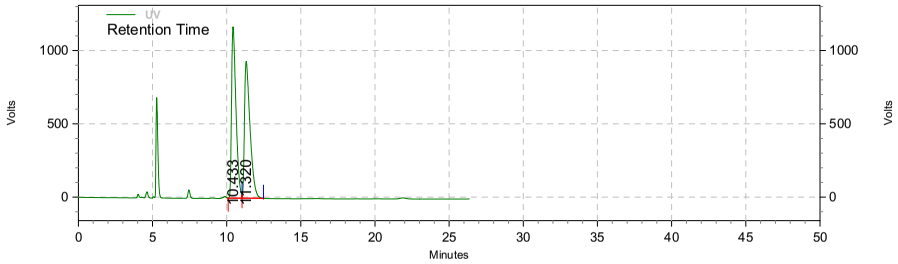


5g

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\New Folder\rac_TIPS_0,6promille_210nm_run1.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\test_0,6promille_iPrOH_210nm.met

Acquired: 11/22/2006 11:37:47 AM
Printed: 4/27/2007 10:57:57 AM



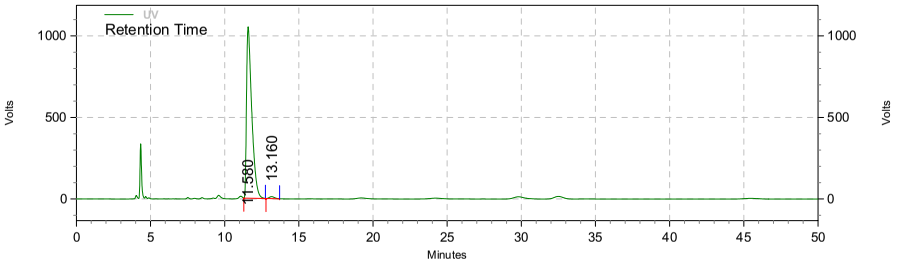
UV Results

Retention Time	Area	Area %	Height	Height %
10.433	96953239	49.88	4674152	55.62
11.320	97435357	50.12	3730204	44.38
Totals	194388596	100.00	8404356	100.00

Area % Report

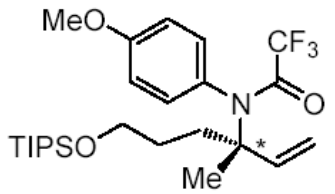
Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\New Folder\df-303Z.test_0,6promille_iPrOH_210nm.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\test_0,6promille_iPrOH_210nm.met

Acquired: 12/13/2006 8:32:59 PM
Printed: 4/27/2007 11:43:48 AM



UV Results

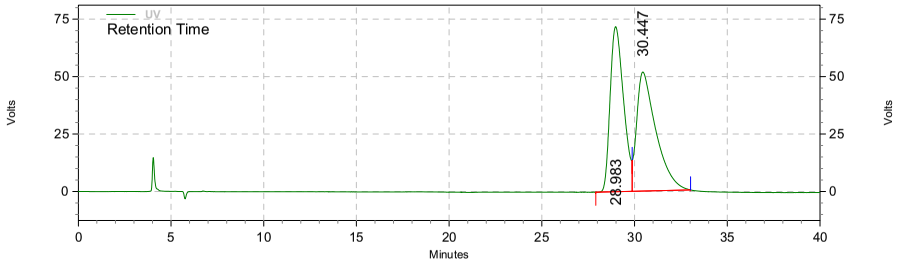
Retention Time	Area	Area %	Height	Height %
11.580	102988326	98.90	4203505	98.87
13.160	1140922	1.10	47954	1.13
Totals	104129248	100.00	4251459	100.00



5h

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\New Folder\racemat_CH2OBn_verduennt_daniel_99,8_210nm.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_99,8_210nm.met
Acquired: 2/7/2007 4:39:13 PM
Printed: 4/27/2007 10:56:35 AM

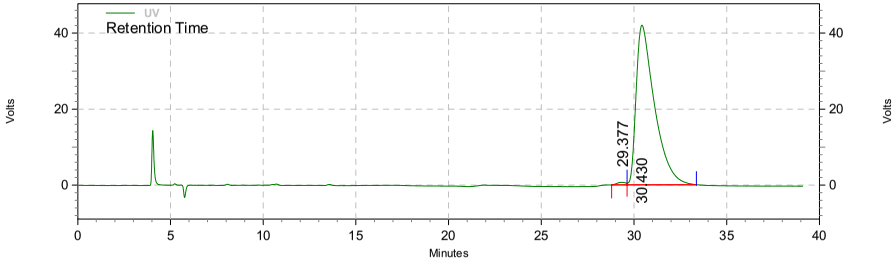


UV Results

Retention Time	Area	Area %	Height	Height %
28.983	14450763	49.13	287298	58.09
30.447	14962957	50.87	207242	41.91
Totals	29413720	100.00	494540	100.00

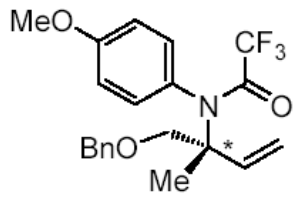
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\New Folder\df-327H_verddaniel_99,8_210nm.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_99,8_210nm.met
Acquired: 2/7/2007 2:13:23 PM
Printed: 4/27/2007 11:48:29 AM



UV Results

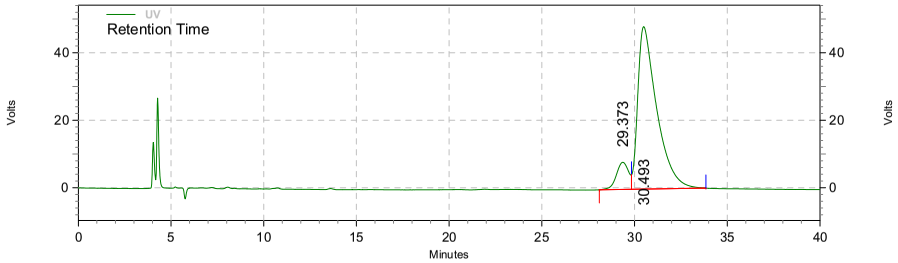
Retention Time	Area	Area %	Height	Height %
29.377	85050	0.73	2678	1.57
30.430	11557089	99.27	167961	98.43
Totals	11642139	100.00	170639	100.00



5i

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\New Folder\df-327H+racematdaniel_99,8_210nm.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_99,8_210nm.met
Acquired: 2/7/2007 2:53:42 PM
Printed: 6/16/2007 7:57:10 PM



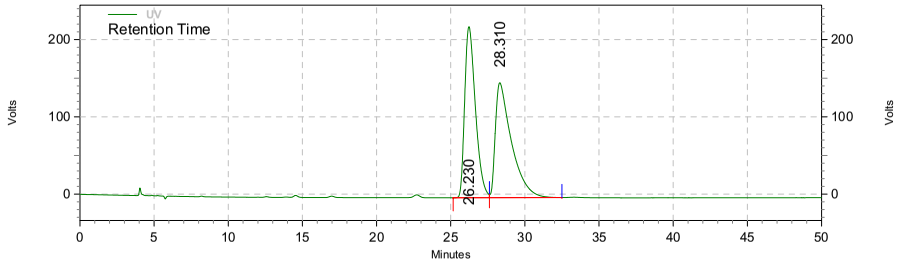
UV Results

Retention Time	Area	Area %	Height	Height %
29.373	1464908	9.68	32118	14.30
30.493	13676224	90.32	192555	85.70
Totals	15141132	100.00	224673	100.00

5i + racemate

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\New Folder\rac_CH2OBn-Et_110407_daniel_99,8_210nm.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_99,8_210nm.met
Acquired: 4/11/2007 10:18:45 AM
Printed: 4/29/2007 7:58:36 PM

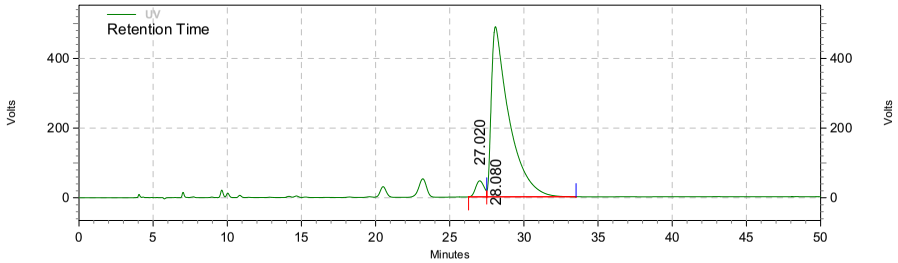


UV Results

Retention Time	Area	Area %	Height	Height %
26.230	44480894	49.79	885611	59.81
28.310	44849254	50.21	595001	40.19
Totals	89330148	100.00	1480612	100.00

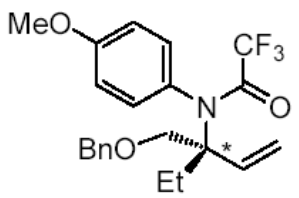
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\New Folder\df-344H_110407_daniel_99,8_210nm.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_99,8_210nm.met
Acquired: 4/11/2007 11:12:10 AM
Printed: 5/5/2007 3:30:01 PM



UV Results

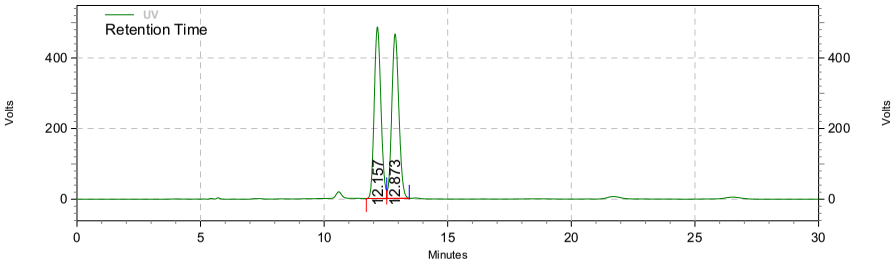
Retention Time	Area	Area %	Height	Height %
27.020	7204169	4.26	184579	8.63
28.080	162014273	95.74	1953372	91.37
Totals	169218442	100.00	2137951	100.00



5j

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\racemat_130607_CH2OBn-Pr_NH_daniel_99,5_to_0,5_250nm_30min.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_99,5_to_0,5_250nm_30min.met
Acquired: 6/13/2007 6:23:26 PM
Printed: 6/13/2007 9:00:43 PM

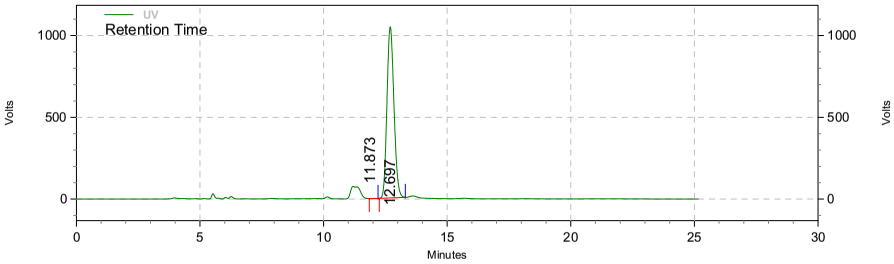


UV Results

Retention Time	Area	Area %	Height	Height %
12.157	36480867	49.70	1941524	51.06
12.873	36916882	50.30	1861154	48.94
Totals	73397749	100.00	3802678	100.00

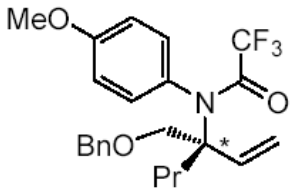
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\df-344F_NH_130607_daniel_99,5_to_0,5_250nm_30min.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_99,5_to_0,5_250nm_30min.met
Acquired: 6/13/2007 6:55:00 PM
Printed: 6/13/2007 9:03:04 PM



UV Results

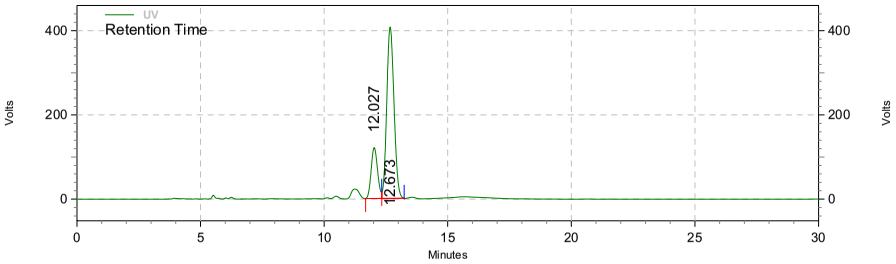
Retention Time	Area	Area %	Height	Height %
11.873	8670	0.01	334	0.01
12.697	82205848	99.99	4183923	99.99
Totals	82214518	100.00	4184257	100.00



5k

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\df-344F_NH+racemat_130607_daniel_99,5_to_0,5_250nm_30min.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_99,5_to_0,5_250nm_30min.met
Acquired: 6/13/2007 8:24:00 PM
Printed: 6/13/2007 8:59:06 PM



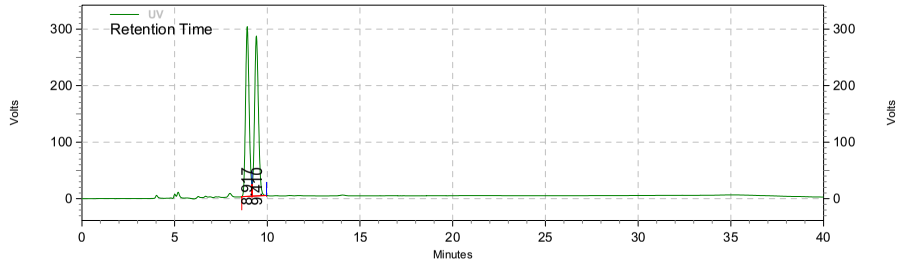
UV Results

Retention Time	Area	Area %	Height	Height %
12.027	8640951	20.63	483119	22.89
12.673	33251997	79.37	1627711	77.11
Totals	41892948	100.00	2110830	100.00

5k + racemate

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\Lukas\racemat_CH2OBn-Bu_daniel_99to1_210nm.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_99to1_210nm.met
Acquired: 4/24/2007 1:21:21 PM
Printed: 4/29/2007 7:52:43 PM

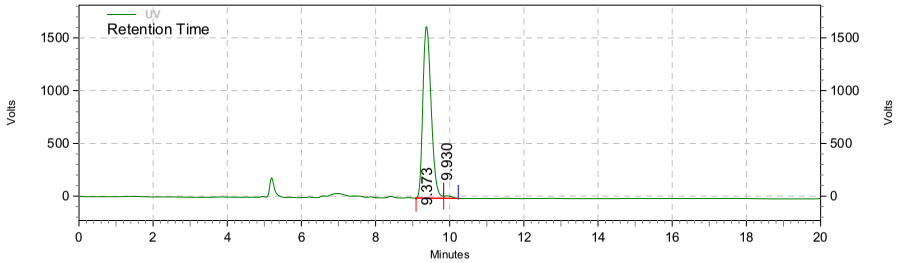


UV Results

Retention Time	Area	Area %	Height	Height %
8.917	16066997	49.43	1200868	51.47
9.410	16438186	50.57	1132416	48.53
Totals	32505183	100.00	2333284	100.00

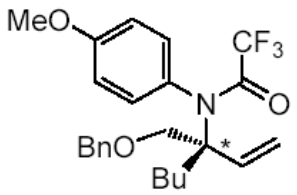
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\Neu\df-351L_verduennt_daniel_99to1_210nm_20min.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_99to1_210nm_20min.met
Acquired: 5/10/2007 11:06:48 PM
Printed: 6/16/2007 7:51:27 PM



UV Results

Retention Time	Area	Area %	Height	Height %
9.373	101009148	98.76	6500018	98.63
9.930	1271128	1.24	90469	1.37
Totals	102280276	100.00	6590487	100.00



5l

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\New Folder\racemat_CH2OBn-PrOTIPS_daniel_300ppm_EtOH_70min_.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_300ppm_EtOH_70min_.met
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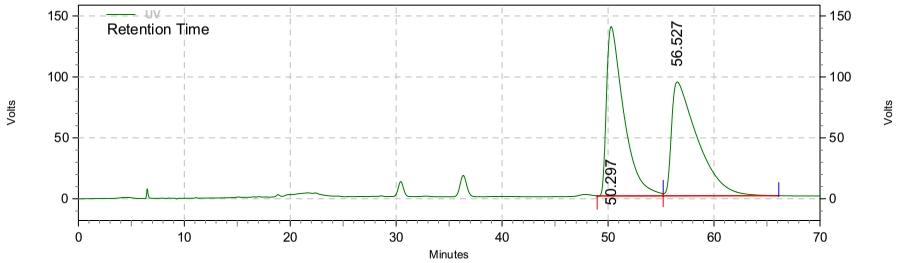

UV Results

Retention Time	Area	Area %	Height	Height %
50.297	63138498	49.77	555434	59.78
56.527	63721077	50.23	373741	40.22
Totals	126859575	100.00	929175	100.00

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\New Folder\df-351M_daniel_300ppm_EtOH_70min_.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_300ppm_EtOH_70min_.met
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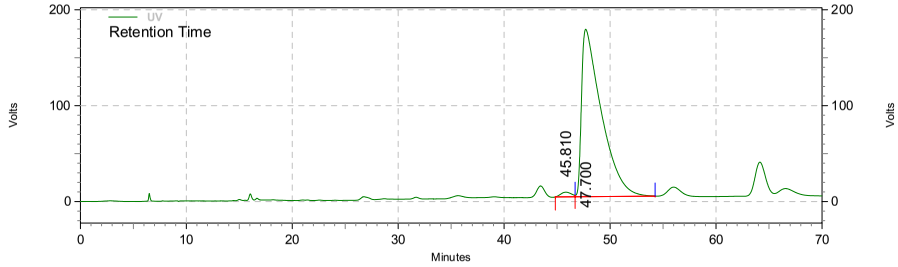
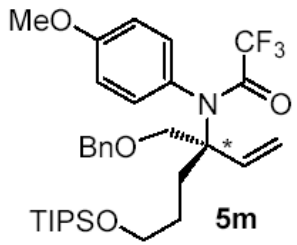


UV Results

Retention Time	Area	Area %	Height	Height %
45.810	1348409	1.46	19588	2.73
47.700	90701033	98.54	698629	97.27
Totals	92049442	100.00	718217	100.00



5m

Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\fcamide\Data\New Folder\df-351M+racemat_daniel_300ppm_EtOH_70min_.met.dat
Method: C:\EZChrom Elite\Enterprise\Projects\fcamide\Method\daniel_300ppm_EtOH_70min_.met
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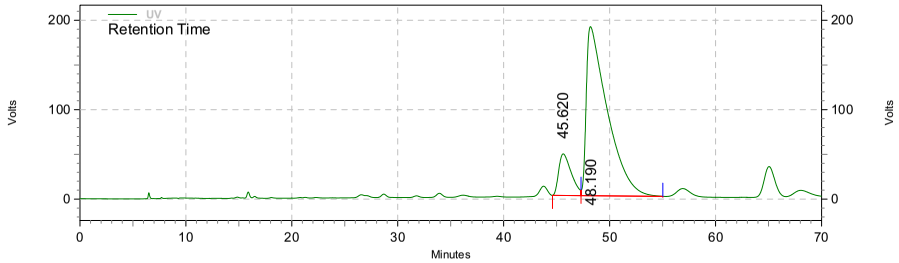

UV Results

Retention Time	Area	Area %	Height	Height %
45.620	15325561	12.81	186067	19.75
48.190	104300178	87.19	756167	80.25
Totals	119625739	100.00	942234	100.00

5m + racemate