

Supporting Information

Catalytic Allylation of Aldehydes Using Unactivated Alkenes

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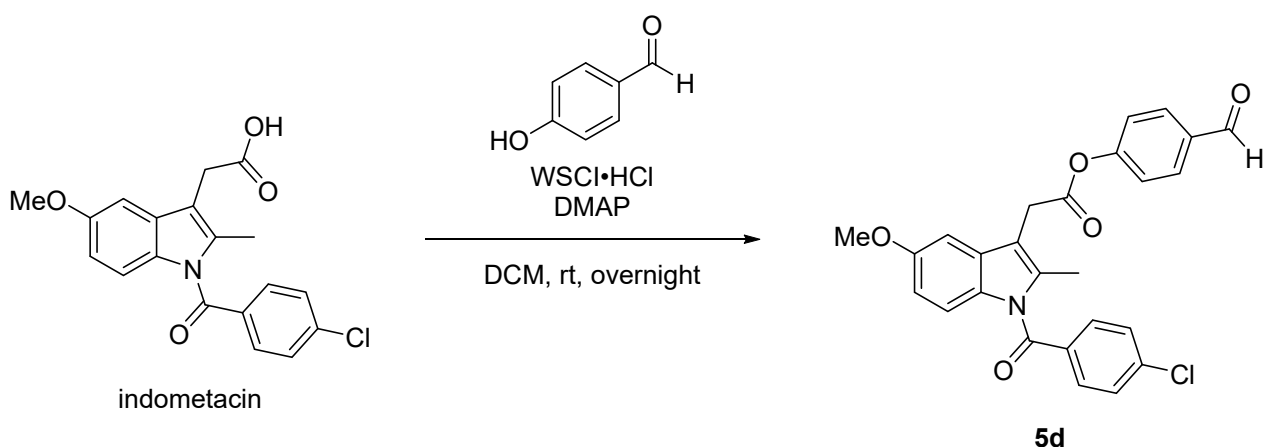
1. General Method

¹H NMR spectra were recorded on JEOL ECX500 (500 MHz for ¹H NMR and 125.65 MHz for ¹³C NMR), and JEOL ECS400 (400 MHz for ¹H NMR and 100 MHz for ¹³C NMR) spectrometer. For ¹H NMR and ¹³C NMR, chemical shifts were reported in the scale relative to CDCl₃ (δ = 7.26 for ¹H NMR and δ = 77.0 for ¹³C NMR) used as an internal reference. Electrospray ionization (ESI)-mass spectra were measured on a JEOL JMS-T100LC AccuTOF spectrometer for HRMS. Infrared (IR) spectra were recorded on a JASCO FT/IR 410 Fourier transform infrared spectrophotometer. The enantiomeric excess (ee) was determined by HPLC analysis (JASCO HPLC systems; pump: PU-986; detector: UV-2075, measured at 210 nm or 254 nm; column: CHIRALPAK IA-3, IC-3). Optical rotations were measured on a JASCO P-1010 polarimeter. Column chromatography was performed with silica gel Merck 60 (230-400 mesh ASTM), Biotage Isolera One and Biotage SNAP Ultra, or Yamazen Smart Flash and Universal Column Premium. All non-commercially available compounds were prepared and characterized as described in Section 2 of this SI. Other reagents were purchased from Aldrich, Tokyo Chemical Industry Co., Ltd. (TCI), Kanto Chemical Co., Inc., Wako Pure Chemical Industries, Ltd., and Strem Chemicals, Inc. and were used as received. GC-MS spectra were recorded on Shimadzu GC-2014. A Valore VBP-L24-C2 with 38W LED lamp (VBL-SE150-BBB (430)) and a Kessil 40W LED lamp (A160WE Tuna Blue) were used as the 430 nm light sources.

2. Preparation of Substrates

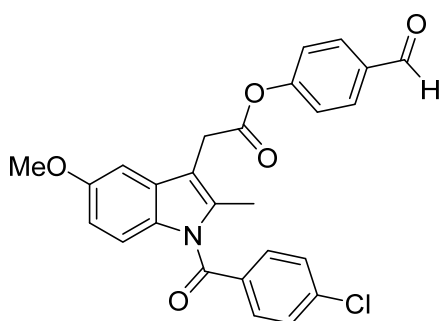
Aldehyde **5b** was prepared according to the reported method.¹ Aldehydes **5d-5g** were synthesized according to the related procedures.² (3*S*,8*S*,9*S*,10*R*,13*R*,14*S*,17*R*)-10,13-dimethyl-17-((*R*)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl 4-formylbenzoate (starting aldehyde for **7af**) was prepared according to the reported method.³ 1-decene-*d*₂ **10-D₂** was prepared according to the reported method.⁴ **HAT1** was synthesized according to a related procedure.⁵ **HAT2** was prepared according to the reported method.⁵ Chiral ligand **8** was prepared according to the reported method.⁶

Preparation of **5d**



To a solution of indometacin (1.46 g, 4.09 mmol), WSCI·HCl (0.942 g, 4.91 mmol), and DMAP (50.0 mg, 0.41 mmol) in CH₂Cl₂ (10 mL), 4-hydroxybenzaldehyde (0.50 g, 4.09 mmol) was added. After overnight of stirring at room temperature, water was added and organic materials were extracted with CH₂Cl₂ three times. The combined organic layers were washed with saturated NaHCO₃ aqueous solution, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. The crude material was purified by silica gel flash column chromatography (MeOH/CH₂Cl₂ = 0% to 5%, v/v) to afford **5d** (529 mg, 1.15 mmol) in 28% yield as a white solid.

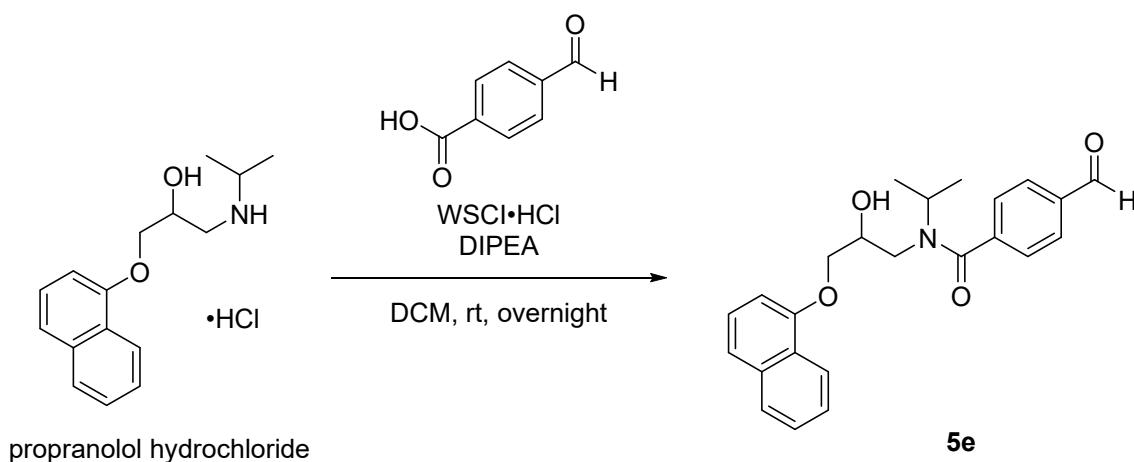
4-formylphenyl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetate (5d**)**



5d

¹H NMR (CDCl₃, 500 MHz): δ 9.98 (1H, s), 7.91 (2H, d, *J* = 8.6 Hz), 7.68 (2H, d, *J* = 8.6 Hz), 7.49 (2H, d, *J* = 8.6 Hz), 7.25 (2H, d, *J* = 8.6 Hz), 7.04 (1H, d, *J* = 2.6 Hz), 6.88 (1H, d, *J* = 9.2 Hz), 6.71 (1H, dd, *J* = 9.2, 2.6 Hz), 3.94 (2H, s), 3.84 (3H, s), 2.47 (3H, s); ¹³C NMR (CDCl₃, 125 MHz): δ 190.8, 168.5, 168.2, 156.1, 155.2, 139.3, 136.3, 134.0, 133.6, 131.1, 131.1, 130.8, 130.3, 129.1, 122.1, 115.0, 111.7, 111.4, 101.1, 55.64, 30.5, 13.3; *m/z* calcd for C₂₆H₂₀NO₅ClNa [M+Na]⁺ 484.0922. Found 484.0931; IR (neat): 2931, 2833, 1698, 1323, 1122, 926 cm⁻¹

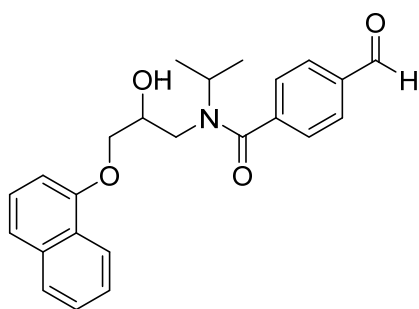
Preparation of **5e**



To a solution of propranolol hydrochloride (3.75 g, 12.7 mmol), terephthalaldehydic acid (2.00 g, 13.3 mmol), and WSCI·HCl (2.92 g, 15.2 mmol) in CH₂Cl₂ (10 mL), diisopropylethylamine (5.53 mL, 31.7

mmol) was added. After overnight of stirring at room temperature, water was added and organic materials were extracted with CH₂Cl₂ three times. The combined organic layers were washed by saturated NaHCO₃ aqueous solution, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. The crude material was purified by silica gel flash column chromatography (MeOH/CH₂Cl₂ = 5% to 10%, v/v) to afford **5e** (203 mg, 0.60 mmol) in 40% yield as a white solid.

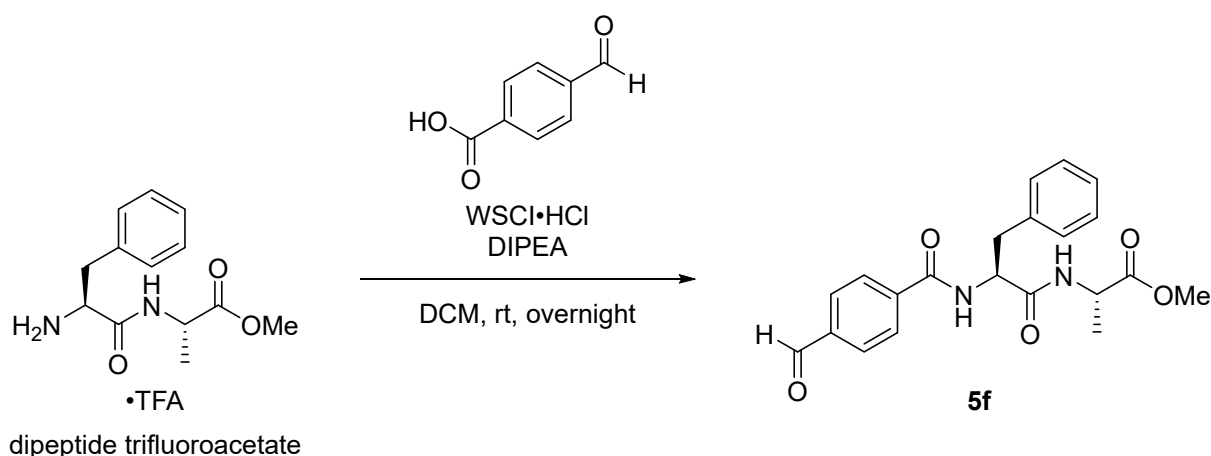
4-formyl-N-(2-hydroxy-3-(naphthalen-1-yloxy)propyl)-N-isopropylbenzamide (**5e**)



5e

¹H NMR (CDCl₃, 500 MHz): δ 10.07 (1H, s), 8.24 (1H, d, *J* = 7.4 Hz), 7.98 (2H, d, *J* = 8.3 Hz), 7.83 (1H, d, *J* = 7.4 Hz), 7.58 (2H, d, *J* = 8.3 Hz), 7.52-7.46 (3H, m), 7.41 (1H, dd, *J* = 7.4, 7.4 Hz), 6.90 (1H, d, *J* = 7.4 Hz), 5.17 (1H, s), 4.39 (1H, m), 4.31 (1H, dd, *J* = 9.2, 4.6 Hz), 4.16 (1H, t, *J* = 8.0 Hz), 4.12-3.88 (2H, m), 3.76 (1H, dd, *J* = 14.3, 1.7 Hz), 1.29 (3H, d, *J* = 6.3 Hz), 1.21 (3H, d, *J* = 6.3 Hz); ¹³C NMR (CDCl₃, 125 MHz): δ 199.3, 173.1, 153.9, 141.8, 136.8, 134.4, 130.0, 127.6, 126.6, 126.4, 125.8, 125.3, 125.2, 121.4, 120.7, 104.8, 71.7, 69.8, 51.2, 46.0, 21.2, 20.7; *m/z* calcd for C₂₄H₂₅NO₄Na [M+Na]⁺ 414.1676. Found 414.1673; IR (neat): 3383, 2978, 2933, 1703, 1606, 1103, 773 cm⁻¹

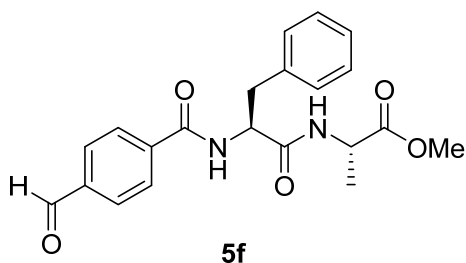
Preparation of **5f**



To a solution of dipeptide trifluoroacetate (728 mg, 2.00 mmol), terephthalaldehydic acid (250 mg, 1.67 mmol), and WSCI·HCl (383 mg, 2.00 mmol) in CH₂Cl₂ (10 mL), diisopropylethylamine (0.725 mL, 4.16 mmol) was added. After overnight of stirring at room temperature, water was added and organic materials were extracted with CH₂Cl₂ three times. The combined organic layers were washed by saturated NaHCO₃

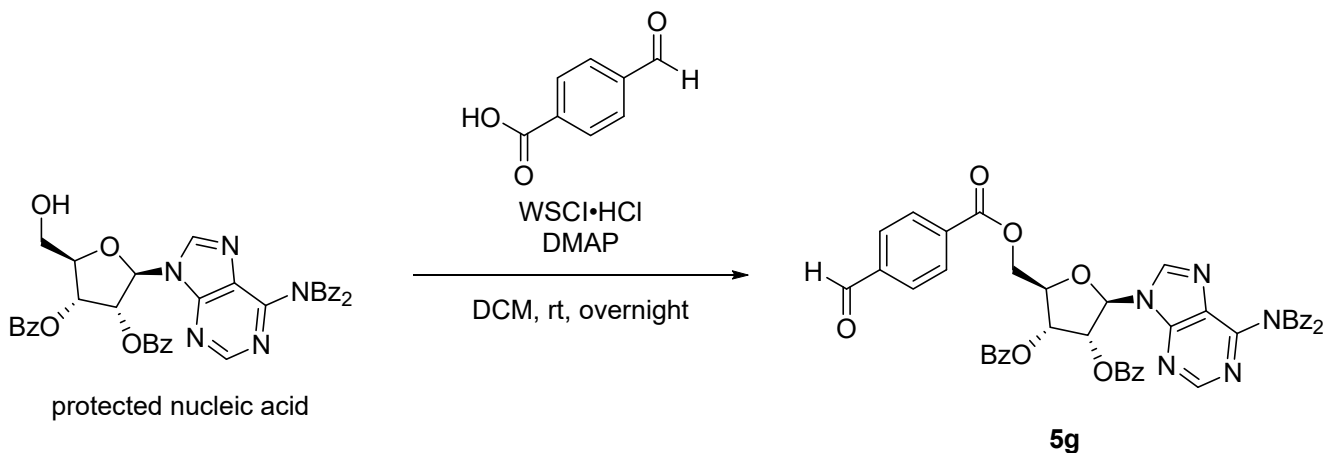
aqueous solution, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. The crude material was purified by silica gel flash column chromatography (MeOH/CH₂Cl₂ = 5% to 10%, v/v) to afford **5f** (214.1 mg, 0.56 mmol) in 34% yield as a white solid.

methyl (4-formylbenzoyl)-L-phenylalanyl-L-alaninate (5f)



¹H NMR (CDCl₃, 500 MHz): δ 10.07 (1H, s), 7.94 (2H, d, *J* = 8.0 Hz), 7.88 (2H, d, *J* = 8.0 Hz), 7.36-7.26 (5H, m), 6.95 (1H, d, *J* = 7.4 Hz), 6.14 (1H, d, *J* = 6.6 Hz), 4.84 (1H, dq, *J* = 7.7, 6.6 Hz), 4.50 (1H, ddd, *J* = 7.4, 7.4, 5.7 Hz), 3.74 (3H, s), 3.27 (1H, dd, *J* = 13.7, 5.7 Hz), 3.14 (1H, dd, *J* = 13.7, 7.4 Hz), 1.36 (3H, d, *J* = 7.7 Hz); ¹³C NMR (CDCl₃, 100 MHz): δ 191.5, 172.7, 170.2, 165.9, 138.9, 138.4, 136.2, 129.8, 129.4, 128.7, 127.8, 127.1, 54.9, 52.6, 48.4, 38.7, 18.2; *m/z* calcd for C₂₁H₂₂N₂O₅Na [M+Na]⁺ 405.1412. Found 405.1439; IR (neat): 3298, 2359, 1747, 1635, 1529, 1205, 822 cm⁻¹

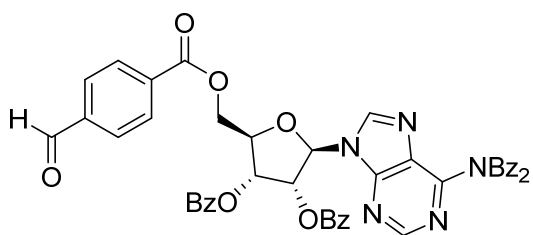
Preparation of 5g



To a solution of terephthalaldehydic acid (330 mg, 2.20 mmol), WSCI·HCl (421 mg, 2.20 mmol) and DMAP (22.4 mg, 0.183 mmol) in CH₂Cl₂ (10 mL), protected nucleic acid (1.20 g, 1.83 mmol) was added. After overnight of stirring at room temperature, water was added and organic materials were extracted with CH₂Cl₂ three times. The combined organic layers were washed by saturated NaHCO₃ aqueous solution, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. The crude material was purified by silica gel flash column chromatography (EtOAc/hexane = 20% to 100%, v/v) three times to afford **5g** (1.33 g, 1.68 mmol) in 92% yield as a colorless oil.

(2*R*,3*R*,4*R*,5*R*)-2-(6-(*N*-benzoylbenzamido)-9*H*-purin-9-yl)-5-(((4-formylbenzoyl)oxy)methyl)tetrahydr

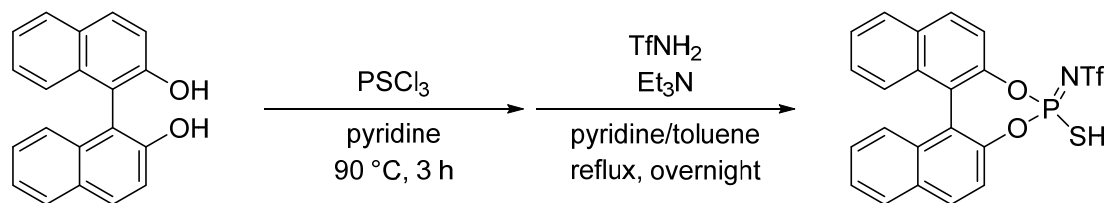
ofuran-3,4-diyl dibenzoate (5g)



5g

^1H NMR (CDCl_3 , 400 MHz): δ 10.08 (1H, s), 8.50 (1H, s), 8.24 (2H, d, $J = 10.9$ Hz), 8.21 (1H, s), 8.14-7.92 (6H, m), 7.90-7.82 (4H, m), 7.64-7.55 (2H, m), 7.54-7.45 (2H, m), 7.44-7.32 (8H, m), 6.50-6.42 (2H, m), 6.35-6.27 (1H, m), 4.93 (1H, dd, $J = 15.7, 4.0$ Hz), 4.85 (1H, dd, $J = 10.3, 5.7$ Hz), 4.74 (1H, dd, $J = 15.7, 6.3$ Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 191.5, 172.1, 165.2, 165.0, 165.0, 152.4, 152.2, 152.0, 143.8, 139.2, 134.1, 133.8, 133.7, 133.0, 130.2, 129.7, 129.7, 129.5, 129.3, 128.7, 128.5, 128.4, 128.1, 127.8, 87.3, 80.4, 73.5, 71.0, 63.8; m/z calcd for $\text{C}_{46}\text{H}_{33}\text{N}_5\text{O}_{10}\text{Na}$ $[\text{M}+\text{Na}]^+$ 838.2120. Found 838.2122; IR (neat): 3062, 1731, 1600, 1267, 1094, 709 cm^{-1}

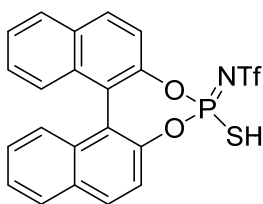
Preparation of HAT1



To a flame-dried round-bottom flask was added 1,1'-bi-2-naphthol (5.73 g, 20 mmol) in pyridine (25 mL). Then, PSCl_3 (2.27 mL, 22 mmol) was added to the mixture dropwise at room temperature. The reaction mixture was heated to 90 °C and stirred for 3 hours. The reaction mixture was cooled to room temperature, and TfNH_2 (4.47 g, 30 mmol), Et_3N (19.4 mL, 140 mmol), and toluene 25 mL were added. The mixture was stirred under reflux for 18 hours. The mixture was cooled to room temperature, and conc. HCl was added until pH became 1 at 0 °C. H_2O (200 mL) was added and organic materials were extracted with EtOAc (3×100 mL). Combined organic layers were washed with H_2O (2×100 mL) and brine, dried over Na_2SO_4 , filtered, and concentrated under reduced pressure. The crude material was purified by silica gel flash column chromatography (EtOAc/hexane = 20% to 100% v/v). Fractions including the product were collected and washed by 4 N HCl aqueous solution twice. This solution was dried over Na_2SO_4 and filtered. After evaporation, the residue was purified by reverse phase flash column chromatography (MeCN/ H_2O = 10% to 100% v/v, ODS column). The obtained material was dissolved in EtOAc and the solution was washed by 4 N HCl aqueous solution twice. This solution was dried over Na_2SO_4 and filtered. Solvent was removed under reduced pressure and the target compound was obtained as a colorless solid (2.15 g, 22%).

1,1,1-trifluoro-*N*-(4-mercapto-4λ5-dinaphtho[2,1-*d*:1',2'-*f*][1,3,2]dioxaphosphin-4-ylidene)methanes

ulfonamide



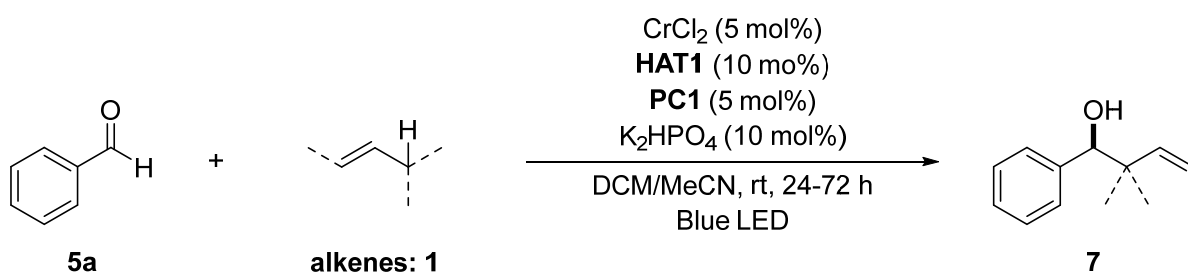
^1H NMR (CDCl_3 , 400 MHz): δ 8.02 (t, $J = 9.2$ Hz, 2H), 7.94 (t, $J = 9.4$ Hz, 2H), 7.62-7.43 (m, 4H), 7.40-7.27 (m, 4H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 147.4 (d, $J = 15.3$ Hz), 145.2 (d, $J = 8.6$ Hz), 132.3 (d, $J = 1.9$ Hz), 132.2 (d, $J = 1.9$ Hz), 132.1 (d, $J = 1.9$ Hz), 131.8 (d, $J = 1.9$ Hz), 131.5, 131.2, 128.5, 127.0 (d, $J = 2.9$ Hz), 126.9, 126.8, 126.1, 126.0, 122.2 (d, $J = 2.9$ Hz), 121.8 (d, $J = 2.9$ Hz), 121.1 (d, $J = 2.9$ Hz), 120.3 (d, $J = 1.9$ Hz), 119.2 (dq, $J = 1.9, 325.2$ Hz). All the spectroscopic data match with the previously reported data.⁵

Preparation of liquid alkenes **1b**, **1c**, **1e**

Commercially available gaseous alkenes **1b**, **1c**, and **1e** were transferred to an argon-filled vessel with septum at -78 °C. The alkenes were liquefied, and stored at -20 °C.

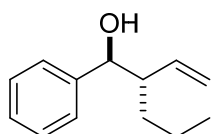
3. General Procedure for Catalytic Allylation of Aldehydes

Procedure for preparation of **7a**, **7d**, **7f-k**, **7n**



In an argon-filled glove box, CrCl₂ (1.5 mg, 0.0125 mmol, 5 mol%), HAT1 (12.4 mg, 0.025 mmol, 10 mol%), PC1 (6.1 mg, 0.0125 mmol, 5 mol%), and K₂HPO₄ (4.4 mg, 0.025 mmol, 10 mol%) were dissolved in degassed CH₂Cl₂ (1.5 mL) and MeCN (1 mL) in a screw-capped vial. Then, alkenes **1** (5.0 mmol, 20 equiv) and benzaldehyde **5a** (25.5 μL , 0.25 mmol, 1 equiv) were added to the reaction mixture. The vial was subjected to 430 nm LED irradiation by a Valore VBP-L24-C2 with 38W LED lamp (VBL-SE150-BBB (430)) for 24–72 hours under temperature control (ca. 27–29 °C). Then, the reaction mixture was diluted by hexane (ca. 2 mL) and passed through a pad of silica gel with CH₂Cl₂ elution. After evaporation, the residue was purified by silica gel flash column chromatography to afford the target homoallylic alcohols **7**. The diastereomeric ratio was determined by ^1H NMR analysis.

1-phenyl-2-vinylpentan-1-ol (**7a**).



7a

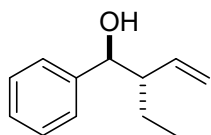
The reaction was conducted using 1-hexene **1a** (628 μ L, 5.0 mmol, 20 equiv) for 24 hours. The residue was purified by silica gel flash column chromatography (CH_2Cl_2 /hexane = 18% to 100%, v/v) to afford **7a** as a yellow oil (43.1 mg, 91%, dr = >20/1).

^1H NMR (CDCl_3 , 500 MHz): δ 7.40-7.27 (m, 5H), 5.66 (ddd, J = 16.6, 10.3, 9.2 Hz, 1H), 5.29-5.12 (m, 2H), 4.39 (d, J = 8.0 Hz, 1H), 2.39-2.20 (m, 1H), 2.05 (brs, 1H), 1.41-1.07 (m, 4H), 0.79 (t, J = 6.9 Hz, 3H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 142.5, 139.4, 128.2, 127.6, 127.0, 118.7, 76.7, 52.6, 32.5, 20.3, 13.9. All the spectroscopic data match with the previously reported data.⁷

Reaction with 3 equiv 1-hexene (**1a**)

The reaction was conducted using 1-hexene **1a** (94.2 μ L, 0.75 mmol, 3 equiv). After 48 h, the reaction mixture was diluted by hexane (ca. 2 mL) and passed through a pad of silica gel with CH_2Cl_2 elution. After evaporation, yield and diastereomeric ratio were determined by ^1H NMR analysis of the crude mixture using 1,1,2,2-tetrachloroethane as an internal standard (64%, dr = >20/1).

2-ethyl-1-phenylbut-3-en-1-ol (**7d**).

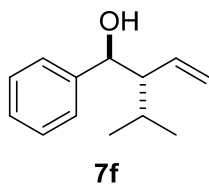


7d

The reaction was conducted using 1-pentene **1d** (548 μ L, 5.0 mmol, 20 equiv) for 24 hours. The residue was purified by silica gel flash column chromatography (CH_2Cl_2 /hexane = 18% to 100%, v/v) to afford **7d** as a light pink oil (35.5 mg, 81%, dr = >20/1).

^1H NMR (CDCl_3 , 500 MHz): δ 7.40-7.19 (m, 5H), 5.66 (ddd, J = 16.6, 10.3, 8.6 Hz, 1H), 5.32-5.12 (m, 2H), 4.41 (d, J = 8.0 Hz, 1H), 2.23-2.16 (m, 1H), 2.14-1.85 (brs, 1H), 1.33-1.08 (m, 2H), 0.80 (t, J = 7.4 Hz, 3H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 142.5, 139.1, 128.2, 127.6, 127.0, 119.0, 76.6, 54.6, 23.4, 11.8. All the spectroscopic data match with the previously reported data.⁸

2-isopropyl-1-phenylbut-3-en-1-ol (**7f**).



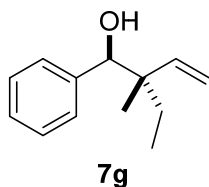
The reaction was conducted using 4-methyl-1-pentene **1f** (638 μ L, 5.0 mmol, 20 equiv) for 24 hours. The residue was purified by silica gel flash column chromatography (CH_2Cl_2 /hexane = 18% to 100%, v/v) to afford **7f** as a light yellow oil (41.1 mg, 86%, dr = >20/1).

^1H NMR (CDCl_3 , 500 MHz): δ 7.38-7.19 (m, 5H), 5.82 (ddd, $J=17.2, 10.3, 9.7$ Hz, 1H), 5.31 (dd, $J=10.3, 1.7$ Hz, 1H), 5.17 (dd, $J=17.2, 1.7$ Hz, 1H), 4.62 (d, $J=8.6$ Hz, 1H), 2.21-2.10 (m, 1H), 1.93 (brs, 1H), 1.52-1.39 (m, 1H), 0.88-0.68 (m, 6H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 142.7, 135.7, 128.3, 127.7, 126.9, 120.2, 74.7, 58.8, 27.5, 21.9, 17.3. All the spectroscopic data match with the previously reported data.⁹

Reaction with 3 equiv 4-methyl-1-pentene (**1f**)

The reaction was conducted using 4-methyl-1-pentene **1f** (95.6 μ L, 0.75 mmol, 3 equiv). After 48 h, the reaction mixture was diluted by hexane (ca. 2 mL) and passed through a pad of silica gel with CH_2Cl_2 elution. After evaporation, yield and diastereomeric ratio were determined by ^1H NMR analysis of the crude mixture using 1,1,2,2-tetrachloroethane as an internal standard (55%, dr = >20/1).

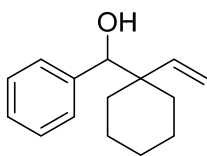
2-ethyl-2-methyl-1-phenylbut-3-en-1-ol (**7g**).



The reaction was conducted using 3-methyl-1-pentene **1g** (628 μ L, 5.0 mmol, 20 equiv) for 24 hours. The residue was purified by silica gel flash column chromatography (CH_2Cl_2 /hexane = 18% to 100%, v/v) to afford **7g** as a light yellow oil (40.4 mg, 85%, dr = 2.3/1).

^1H NMR (CDCl_3 , 500 MHz): major isomer: δ 7.32-7.12 (m 5H), 5.83 (dd, $J=17.8, 10.9$ Hz, 1H), 5.29 (dd, $J=10.9, 1.1$ Hz, 1H), 5.09 (dd, $J=17.8, 1.1$ Hz, 1H), 4.44 (s, 1H), 1.93 (brs, 1H), 1.52-1.42 (m, 1H), 1.32-1.20 (m, 1H), 0.88 (s, 3H), 0.78 (t, $J=7.4$ Hz, 3H); minor isomer: δ 7.32-7.12 (m 5H), 5.74 (dd, $J=17.5, 10.9$ Hz, 1H), 5.21 (dd, $J=10.9, 1.4$ Hz, 1H), 5.01 (dd, $J=17.5, 1.4$ Hz, 1H), 4.45 (s, 1H), 1.93 (brs, 1H), 1.42-1.32 (m, 2H), 1.05 (s, 3H), 0.78 (t, $J=7.4$ Hz, 3H); ^{13}C NMR (CDCl_3 , 125 MHz): major isomer: δ 143.9, 141.2, 128.1, 127.5, 127.4, 116.0, 79.9, 46.2, 29.9, 15.5, 8.3; minor isomer: δ 143.8, 142.4, 127.8, 127.4, 127.3, 115.7, 80.7, 45.9, 28.8, 18.5, 8.4. All the spectroscopic data match with the previously reported data.^{10,11}

phenyl(1-vinylcyclohexyl)methanol (7h).



7h

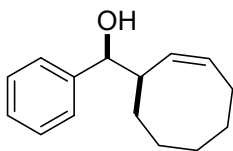
The reaction was conducted using vinylcyclohexane **1h** (680 μ L, 5.0 mmol, 20 equiv) for 36 hours. The residue was purified by silica gel flash column chromatography (CH_2Cl_2 /hexane = 18% to 100%, v/v) to afford **7h** as a light yellow oil (47.6 mg, 88%).

^1H NMR (CDCl_3 , 400 MHz): δ 7.34-7.26 (m, 5H), 5.56 (dd, J = 18.3, 11.0 Hz, 1H), 5.41 (dd, J = 11.0, 1.4 Hz, 1H), 5.12 (dd, J = 18.3, 1.4 Hz, 1H), 4.35 (s, 1H), 2.16-1.84 (m, 2H), 1.61-1.21 (m, 8H), 1.22-1.03 (m, 1H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 141.8, 140.7, 128.0, 127.4, 127.3, 117.6, 81.0, 45.7, 32.8, 31.3, 26.3, 22.1, 22.0. All the spectroscopic data match with the previously reported data.¹²

Reaction with 3 equiv vinylcyclohexane (1h)

The reaction was conducted using vinylcyclohexane **1h** (102 μ L, 0.75 mmol, 3 equiv). After 48 h, the reaction mixture was diluted by hexane (ca. 2 mL) and passed through a pad of silica gel with CH_2Cl_2 elution. After evaporation, yield was determined by ^1H NMR analysis of the crude mixture using 1,1,2,2-tetrachloroethane as an internal standard (71%).

(Z)-cyclooct-2-en-1-yl(phenyl)methanol (7i).

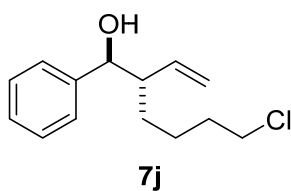


7i

The reaction was conducted using cyclooctene **1i** (648 μ L, 5.0 mmol, 20 equiv) for 36 hours. The residue was purified by silica gel flash column chromatography (CH_2Cl_2 /hexane = 18% to 100%, v/v) to afford **7i** as a light yellow solid (39.4 mg, 73%, dr = 13/1).

^1H NMR (CDCl_3 , 500 MHz): δ 7.40-7.19 (m, 5H), 5.68-5.51 (m, 1H), 5.35-5.22 (m, 1H), 4.59 (d, J = 7.4 Hz, 1H), 3.00-2.87 (m, 1H), 2.28-2.06 (m, 1H), 2.05-1.73 (m, 3H), 1.72-1.09 (m, 7H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 143.4, 130.3, 130.1, 128.3, 127.5, 126.7, 78.1, 43.2, 31.6, 29.3, 26.8, 26.6, 25.5. All the spectroscopic data match with the previously reported data.¹³

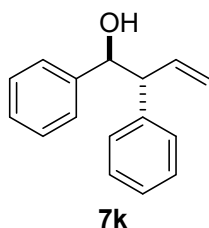
6-chloro-1-phenyl-2-vinylhexan-1-ol (7j).



The reaction was conducted using 7-chlorohept-1-ene **1j** (750 μL , 5.0 mmol, 20 equiv) for 72 hours. A Kessil 40W LED lamp (A160WE Tuna Blue) was used as a light source under temperature control (ca. 35–38 $^{\circ}\text{C}$). The residue was purified by silica gel flash column chromatography ($\text{CH}_2\text{Cl}_2/\text{hexane} = 18\%$ to 100%, v/v) to afford **7j** as a colorless liquid (55.8 mg, 94%, dr = 18/1).

^1H NMR (CDCl_3 , 500 MHz): δ 7.38-7.26 (5H, m), 5.70-5.60 (1H, m), 5.27 (1H, dd, $J = 10.3, 1.7$ Hz), 5.21 (1H, dd, $J = 17.2, 1.7$ Hz), 4.40 (1H, d, $J = 7.4$ Hz), 3.45-3.38 (2H, m), 2.34-2.24 (1H, m), 1.76-1.54 (2H, m), 1.50-1.38 (1H, m), 1.30-1.16 (3H, m); ^{13}C NMR (CDCl_3 , 125 MHz): δ 142.3, 138.8, 128.2, 127.6, 126.8, 119.0, 76.5, 52.5, 44.8, 32.3, 29.5, 24.5; m/z calcd for $\text{C}_{14}\text{H}_{19}\text{OClNa}$ $[\text{M}+\text{Na}]^+$ 261.1017. Found 261.1018; IR (neat): 3420, 2938, 1453, 1092, 1038, 702 cm^{-1}

1,2-diphenylbut-3-en-1-ol (**7k**).

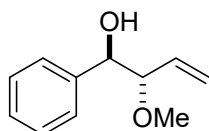


From alkene **1k**: The reaction was conducted using *trans*- β -methylstyrene **1k** (649 μL , 5.0 mmol, 20 equiv) for 36 hours. The residue was purified by silica gel flash column chromatography ($\text{CH}_2\text{Cl}_2/\text{hexane} = 18\%$ to 100%, v/v) to afford **7k** as a light yellow oil (38.1 mg, 68%, dr = >20/1).

From alkene **1l**: The reaction was conducted using allylbenzene **1l** (664 μL , 5.0 mmol, 20 equiv) for 36 hours. The residue was purified by silica gel flash column chromatography ($\text{CH}_2\text{Cl}_2/\text{hexane} = 18\%$ to 100%, v/v) to afford **7k** as a pink oil (41.6 mg, 74%, dr = >20/1).

^1H NMR (CDCl_3 , 400 MHz): δ 7.42-6.97 (m, 10H), 6.26 (ddd, $J = 17.4, 10.5, 9.2$ Hz, 1H), 5.33-5.19 (m, 2H), 4.85 (d, $J = 8.2$ Hz, 1H), 3.61-3.51 (m, 1H), 2.17 (brs, 1H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 141.8, 140.6, 137.8, 128.3, 128.3, 127.9, 127.4, 126.6, 126.6, 118.5, 77.2, 59.2. All the spectroscopic data match with the previously reported data.¹²

2-methoxy-1-phenylbut-3-en-1-ol (**7n**).

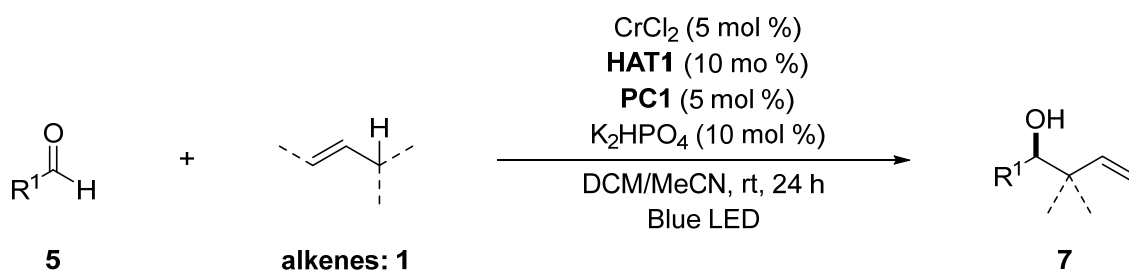


7n

The reaction was conducted using allyl methyl ether **1n** (468 μ L, 5.0 mmol, 20 equiv) for 24 hours. The residue was purified by silica gel flash column chromatography (EtOAc/hexane = 6% to 50%, v/v) to afford **7n** as a light brown oil (36.9 mg, 83%, dr = 1.2/1).

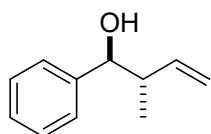
^1H NMR (CDCl_3 , 500 MHz): major isomer: δ 7.39-7.20 (m, 5H), 5.70-5.59 (m, 1H), 5.28 (dd, J = 11.5, 1.1 Hz, 1H), 5.21-5.17 (m, 1H), 4.83 (d, J = 4.6 Hz 1H), 3.81-3.76 (m, 1H), 3.33 (s, 3H), 2.63 (brs, 1H); minor isomer: δ 7.39-7.20 (m, 5H), 5.58-5.47 (m, 1H), 5.21-5.17 (m, 1H), 5.07 (dd, J = 18.3, 1.1 Hz, 1H), 4.50 (d, J = 8.0 Hz, 1H), 3.65-3.59 (m, 1H), 3.38 (s, 3H), 2.63 (brs, 1H); ^{13}C NMR (CDCl_3 , 125 MHz): major isomer: δ 139.6, 133.6, 128.0, 127.5, 126.7, 120.2, 86.6, 75.3, 56.7; minor isomer: δ 140.1, 133.9, 128.1, 127.9, 127.4, 119.7, 87.5, 76.8, 56.8. All the spectroscopic data match with the previously reported data.^{14,15}

Procedure for preparation of **7b**, **7e**, **7o-q**, **7s-w**, **7y**, **7aa**, **7ab**, **7ad**



In an argon-filled glove box, CrCl_2 (1.5 mg, 0.0125 mmol, 5 mol%), **HAT1** (12.4 mg, 0.025 mmol, 10 mol%), **PC1** (6.1 mg, 0.0125 mmol, 5 mol%), and K_2HPO_4 (4.4 mg, 0.025 mmol, 10 mol%) were dissolved in degassed CH_2Cl_2 (1.5 mL) and MeCN (1 mL) in a screw-capped tube. Then, aldehydes **5** (0.25 mmol, 1 equiv) were added to the reaction mixture. The reaction tube was removed from the glove box and the reaction mixture was cooled to -78°C . Liquid alkenes **1** (ca. 1 mL) cooled at -78°C were added to the reaction mixture via syringe. The reaction mixture was warmed to room temperature and subjected to 430 nm LED irradiation by a Kessil 40W LED lamp (A160WE Tuna Blue) for 24 hours under temperature control (ca. $35\text{--}38^\circ\text{C}$). Then, the reaction mixture was diluted by hexane (ca. 2 mL) and passed through a pad of silica gel with CH_2Cl_2 elution. After evaporation, diastereomeric ratio was determined by ^1H NMR analysis. The residue was purified by silica gel flash column chromatography to afford the target homoallylic alcohols **7**.

2-methyl-1-phenylbut-3-en-1-ol (**7b**).



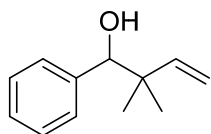
7b

From alkene **1b**: The reaction was conducted using benzaldehyde **5a** (25.5 μ L, 0.25 mmol, 1 equiv) and liquid 1-butene **1b** (ca. 1 mL). The residue was purified by silica gel flash column chromatography (CH_2Cl_2 /hexane = 18% to 100%, v/v) to afford **7b** as a colorless liquid (38.3 mg, 94%, dr = 16/1).

From alkene **1c**: The reaction was conducted using benzaldehyde **5a** (25.5 μ L, 0.25 mmol, 1 equiv) and liquid 2-butene **1c** (ca. 1 mL). The residue was purified by silica gel flash column chromatography (CH_2Cl_2 /hexane = 18% to 100%, v/v) to afford **7b** as a colorless liquid (40.6 mg, 100%, dr = 14/1).

^1H NMR (CDCl_3 , 500 MHz): δ 7.38-7.26 (5H, m), 5.38 (1H, ddd, $J = 17.2, 10.3, 8.0$ Hz), 5.23-5.16 (2H, m), 4.36 (1H, d, $J = 8.0$ Hz), 2.53-2.42 (1H, m), 0.87 (3H, d, $J = 6.9$ Hz); ^{13}C NMR (CDCl_3 , 125 MHz): δ 142.4, 140.6, 128.2, 127.6, 126.8, 116.8, 76.8, 46.2, 16.5. All the spectroscopic data match with the previously reported data.¹⁶

2,2-dimethyl-1-phenylbut-3-en-1-ol (**7e**).

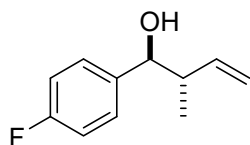


7e

The reaction was conducted using benzaldehyde **5a** (25.5 μ L, 0.25 mmol, 1 equiv) and liquid 3-methyl-1-butene **1e** (ca. 1 mL). The residue was purified by silica gel flash column chromatography (CH_2Cl_2 /hexane = 18% to 100%, v/v) to afford **7e** as a light yellow oil (35.8 mg, 81%).

^1H NMR (CDCl_3 , 500 MHz): δ 7.32-7.19 (m, 5H), 5.92 (dd, $J = 17.8, 10.9$ Hz, 1H), 5.19-5.00 (m, 2H), 4.44 (s, 1H), 1.90 (brs, 1H), 1.02 (s, 3H), 0.96 (s, 3H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 145.1, 140.7, 127.8, 127.5, 127.4, 113.9, 80.6, 42.3, 24.5, 21.0. All the spectroscopic data match with the previously reported data.¹⁰

1-(4-fluorophenyl)-2-methylbut-3-en-1-ol (**7o**).

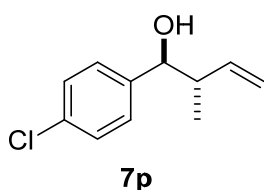


7o

The reaction was conducted using 4-fluorobenzaldehyde (26.8 μ L, 0.25 mmol, 1 equiv) and liquid 2-butene **1c** (ca. 1 mL). The residue was purified by silica gel flash column chromatography (CH_2Cl_2 /hexane = 18% to 100%, v/v) to afford **7o** as a colorless liquid (42.3 mg, 94%, dr = 15/1).

^1H NMR (CDCl_3 , 500 MHz): δ 7.32-7.28 (2H, m), 7.06-7.00 (2H, m), 5.38 (1H, ddd, $J = 16.6, 10.3, 8.0$ Hz), 5.22-5.18 (2H, m), 4.60 (1H, d, $J = 8.0$ Hz), 2.43-2.38 (1H, m), 2.01 (1H, brs), 0.86 (3H, d, $J = 6.9$ Hz); ^{13}C NMR (CDCl_3 , 125 MHz): δ 162.1 (d, $J = 245$ Hz), 140.3, 137.9 (d, $J = 2.8$ Hz), 128.3 (d, $J = 8.5$ Hz), 117.0, 114.9 (d, $J = 20.7$ Hz), 76.6, 46.4, 16.3. All the spectroscopic data match with the previously reported data.¹⁷

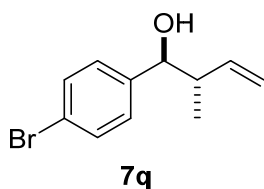
1-(4-chlorophenyl)-2-methylbut-3-en-1-ol (7p).



The reaction was conducted using 4-chlorobenzaldehyde (35.1 mg, 0.25 mmol, 1 equiv) and liquid 2-butene **1c** (ca. 1 mL). The residue was purified by silica gel flash column chromatography (CH_2Cl_2 /hexane = 18% to 100%, v/v) to afford **7p** as a colorless liquid (37.2 mg, 76%, dr = 16/1).

^1H NMR (CDCl_3 , 400 MHz): δ 7.35-7.25 (4H, m), 5.77 (1H, ddd, $J = 18.3, 10.0, 8.2$ Hz), 5.23-5.16 (2H, m), 4.34 (1H, d, $J = 7.8$ Hz), 2.49-2.35 (1H, m), 0.87 (3H, d, $J = 6.9$ Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 140.8, 140.6, 133.2, 128.3, 128.1, 117.2, 76.7, 46.3, 16.3. All the spectroscopic data match with the previously reported data.¹⁸

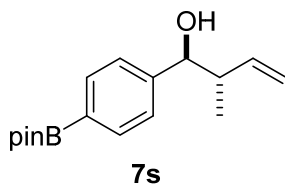
1-(4-bromophenyl)-2-methylbut-3-en-1-ol (7q).



The reaction was conducted using 4-bromobenzaldehyde (46.3 mg, 0.25 mmol, 1 equiv) and liquid 2-butene **1c** (ca. 1 mL). The residue was purified by silica gel flash column chromatography (CH_2Cl_2 /hexane = 18% to 100%, v/v) to afford **7q** as a colorless liquid (57.6 mg, 96%, dr = 20/1).

^1H NMR (CDCl_3 , 500 MHz): δ 7.47 (2H, d, $J = 8.0$ Hz), 7.22 (2H, d, $J = 8.0$ Hz), 5.77 (1H, ddd, $J = 17.8, 9.8, 8.0$ Hz), 5.23-5.16 (2H, m), 4.33 (1H, d, $J = 8.0$ Hz), 2.49-2.36 (1H, m), 1.98 (1H, brs), 0.87 (3H, d, $J = 6.9$ Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 141.3, 140.1, 131.2, 128.5, 121.3, 117.2, 76.7, 46.3, 16.3. All the spectroscopic data match with the previously reported data.¹⁹

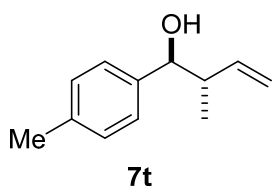
2-methyl-1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)but-3-en-1-ol (7s).



The reaction was conducted using 4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzaldehyde (58.0 mg, 0.25 mmol, 1 equiv) and liquid 2-butene **1c** (ca. 1 mL). The residue was purified by silica gel flash column chromatography (CH₂Cl₂/hexane = 18% to 100%, v/v) to afford **7s** as a colorless liquid (68.3 mg, 95%, dr = 15/1).

¹H NMR (CDCl₃, 500 MHz): δ 7.79 (2H, d, *J* = 8.0 Hz), 7.34 (2H, d, *J* = 8.0 Hz), 5.77 (1H, ddd, *J* = 17.2, 10.3, 8.6 Hz), 5.22-5.14 (2H, m), 4.38 (1H, d, *J* = 7.4 Hz), 2.52-2.41 (1H, m), 1.35 (12H, s), 0.87 (3H, d, *J* = 6.9 Hz); ¹³C NMR (CDCl₃, 100 MHz): δ 145.6, 140.3, 134.6, 126.1, 125.8, 116.7, 83.7, 77.7, 46.0, 24.8, 16.3; *m/z* calcd for C₁₇H₂₅O₃BNa [M+Na]⁺ 311.1789. Found 311.1793; IR (neat): 3466, 2978, 1612, 1362, 1145, 1089, 859, 660 cm⁻¹

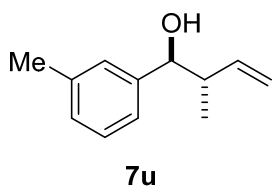
2-methyl-1-(*p*-tolyl)but-3-en-1-ol (**7t**).



The reaction was conducted using *p*-tolualdehyde (29.4 μL, 0.25 mmol, 1 equiv) and liquid 2-butene **1c** (ca. 1 mL). The residue was purified by silica gel flash column chromatography (CH₂Cl₂/hexane = 18% to 100%, v/v) to afford **7t** as a colorless liquid (39.2 mg, 89%, dr = 9.2/1).

¹H NMR (CDCl₃, 400 MHz): δ 7.27-7.12 (4H, m), 5.81 (1H, ddd, *J* = 17.8, 13.2, 8.7 Hz), 5.24-5.15 (2H, m), 4.32 (1H, d, *J* = 8.2 Hz), 2.52-2.42 (1H, m), 2.35 (3H, s), 1.85 (1H, brs), 0.86 (3H, d, *J* = 7.3 Hz); ¹³C NMR (CDCl₃, 100 MHz): δ 140.8, 139.4, 137.2, 128.9, 126.7, 116.6, 77.7, 46.2, 21.1, 16.5. All the spectroscopic data match with the previously reported data.¹⁷

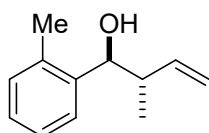
2-methyl-1-(*m*-tolyl)but-3-en-1-ol (**7u**).



The reaction was conducted using *m*-tolualdehyde (29.4 μL, 0.25 mmol, 1 equiv) and liquid 2-butene **1c** (ca. 1 mL). The residue was purified by silica gel flash column chromatography (CH₂Cl₂/hexane = 18% to 100%, v/v) to afford **7u** as a colorless liquid (36.8 mg, 84%, dr = 13/1).

^1H NMR (CDCl_3 , 400 MHz): δ 7.26-7.08 (4H, m), 5.80 (1H, ddd, $J = 17.9, 13.2, 8.7$ Hz), 5.25-5.16 (2H, m), 4.31 (1H, d, $J = 7.8$ Hz), 2.53-2.42 (1H, m), 2.36 (3H, s), 1.90 (1H, brs), 0.87 (3H, d, $J = 6.9$ Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 142.3, 140.7, 137.8, 128.4, 128.0, 127.4, 124.0, 116.7, 77.8, 46.2, 21.4, 16.6; m/z calcd for $\text{C}_{12}\text{H}_{16}\text{ONa}$ $[\text{M}+\text{Na}]^+$ 199.1093. Found 199.1086; IR (neat): 3408, 2975, 1608, 1456, 1031, 912, 786, 706 cm^{-1}

2-methyl-1-(*o*-tolyl)but-3-en-1-ol (7v).

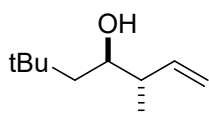


7v

The reaction was conducted using *o*-tolualdehyde (28.9 μL , 0.25 mmol, 1 equiv) and liquid 2-butene **1c** (ca. 1 mL). The residue was purified by silica gel flash column chromatography ($\text{CH}_2\text{Cl}_2/\text{hexane} = 18\%$ to 100%, v/v) to afford **7v** as a colorless liquid (39.9 mg, 91%, dr = 8.3/1).

^1H NMR (CDCl_3 , 400 MHz): δ 7.44-7.38 (1H, m), 7.26-7.12 (3H, m), 5.85 (1H, ddd, $J = 17.4, 10.5, 8.2$ Hz), 5.25-5.16 (2H, m), 4.67 (1H, d, $J = 8.2$ Hz), 2.60-2.49 (1H, m), 2.37 (3H, s), 1.82 (1H, brs), 0.91 (3H, d, $J = 6.9$ Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 140.7, 140.6, 135.3, 130.2, 127.2, 126.3, 126.1, 116.8, 73.4, 45.7, 19.5, 16.5. All the spectroscopic data match with the previously reported data.²⁰

3,6,6-trimethylhept-1-en-4-ol (7w).

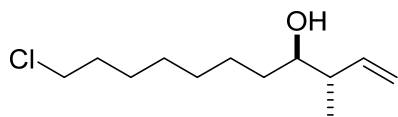


7w

The reaction was conducted using 3,3-dimethylbutyraldehyde (31.0 μL , 0.25 mmol, 1 equiv) and liquid 2-butene **1c** (ca. 1 mL). The residue was purified by silica gel flash column chromatography ($\text{CH}_2\text{Cl}_2/\text{hexane} = 18\%$ to 100%, v/v) to afford **7w** as a colorless liquid (28.4 mg, 73%, dr = 14/1).

^1H NMR (CDCl_3 , 500 MHz): δ 5.74 (1H, ddd, $J = 17.2, 10.3, 8.0$ Hz), 5.13-5.06 (2H, m), 3.57-3.51 (1H, m), 2.20-2.12 (1H, m), 1.41 (1H, dd, $J = 14.6, 1.7$ Hz), 1.26 (1H, dd, $J = 14.6, 8.6$ Hz), 1.03 (3H, d, $J = 6.9$ Hz), 0.96 (9H, s); ^{13}C NMR (CDCl_3 , 100 MHz): δ 140.2, 116.3, 81.1, 72.4, 48.1, 45.3, 30.1, 15.9; m/z calcd for $\text{C}_{10}\text{H}_{20}\text{ONa}$ $[\text{M}+\text{Na}]^+$ 179.1406. Found 179.1396; IR (neat): 3446, 2955, 1465, 1334, 1206, 970 cm^{-1}

11-chloro-3-methylundec-1-en-4-ol (7y).

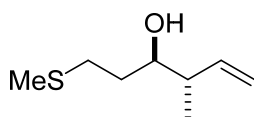


7y

The reaction was conducted using 8-chlorooctanal (35.4 μL , 0.25 mmol, 1 equiv) and liquid 2-butene **1c** (ca. 1 mL). The residue was purified by silica gel flash column chromatography ($\text{CH}_2\text{Cl}_2/\text{hexane} = 18\%$ to 100%, v/v) to afford **7y** as a colorless liquid (41.3 mg, 76%, dr = 13/1).

^1H NMR (CDCl_3 , 500 MHz): δ 5.75 (1H, ddd, $J = 16.6, 10.3, 8.0$ Hz), 5.14-5.06 (2H, m), 3.53 (2H, t, $J = 6.9$ Hz), 3.42-3.36 (1H, m), 2.24-2.16 (1H, m), 1.80-1.72 (2H, m), 1.56-1.28 (9H, m), 1.26 (1H, dd, $J = 14.3, 8.6$ Hz), 1.03 (3H, d, $J = 6.9$ Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 140.3, 116.2, 74.6, 45.1, 44.1, 34.1, 32.5, 29.5, 28.8, 26.8, 25.6, 16.2; m/z calcd for $\text{C}_{12}\text{H}_{23}\text{OCiNa}$ $[\text{M}+\text{Na}]^+$ 241.1330. Found 241.1318; IR (neat): 3397, 2931, 2856, 1458, 1001, 913 cm^{-1}

4-methyl-1-(methylthio)hex-5-en-3-ol (7aa).

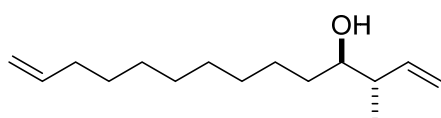


7aa

The reaction was conducted using 3-(methylthio)propanal (25.0 μL , 0.25 mmol, 1 equiv) and liquid 2-butene **1c** (ca. 1 mL). The residue was purified by silica gel flash column chromatography ($\text{CH}_2\text{Cl}_2/\text{hexane} = 18\%$ to 100%, v/v) to afford **7aa** as a colorless liquid (26.7 mg, 67%, dr = 21/1).

^1H NMR (CDCl_3 , 500 MHz): δ 5.75 (1H, ddd, $J = 17.2, 10.9, 8.6$ Hz), 5.15-5.06 (2H, m), 3.58-3.50 (1H, m), 2.72-2.58 (2H, m), 2.26-2.16 (1H, m), 2.12 (3H, s), 1.84-1.62 (2H, m), 1.05 (3H, d, $J = 6.9$ Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 140.0, 116.5, 73.6, 44.3, 33.2, 30.9, 16.1, 15.5; m/z calcd for $\text{C}_8\text{H}_{16}\text{OSNa}$ $[\text{M}+\text{Na}]^+$ 183.0814. Found 183.0804; IR (neat): 3431, 2964, 2916, 1637, 1427, 999, 915 cm^{-1}

3-methyltetradeca-1,13-dien-4-ol (7ab).



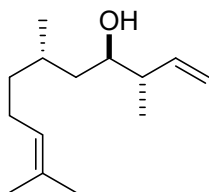
7ab

The reaction was conducted using undec-10-enal (50.0 μL , 0.25 mmol, 1 equiv) and liquid 2-butene **1c** (ca. 1 mL). The residue was purified by silica gel flash column chromatography ($\text{CH}_2\text{Cl}_2/\text{hexane} = 18\%$ to 100%, v/v) to afford **7ab** as a colorless liquid (49.3 mg, 88%, dr = 10/1).

^1H NMR (CDCl_3 , 500 MHz): δ 5.86-5.69 (2H, m), 5.15-5.06 (2H, m), 5.03-4.89 (2H, m), 3.42-3.34 (1H, m),

2.36-2.14 (1H, m), 2.08-1.98 (2H, m), 1.56-1.23 (14H, m), 1.03 (3H, d, $J = 6.9$ Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 140.3, 139.2, 116.2, 114.1, 74.6, 44.1, 34.2, 33.8, 29.7, 29.5, 29.4, 29.1, 28.9, 25.7, 16.2. All the spectroscopic data match with the previously reported data.²¹

(6S)-3,6,10-trimethylundeca-1,9-dien-4-ol (7ad).

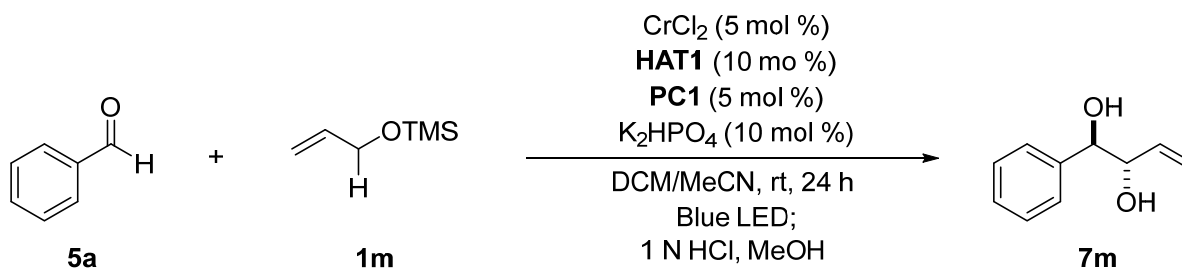


7ad

The reaction was conducted using (–)-citronellal (45.0 μL , 0.25 mmol, 1 equiv) and liquid 2-butene **1c** (ca. 1 mL). After 24 h, the reaction mixture was diluted by hexane (ca. 2 mL) and passed through a pad of silica gel with CH_2Cl_2 elution. After evaporation, yield and diastereomeric ratio were determined by ^1H NMR analysis of the crude mixture using 1,1,2,2-tetrachloroethane as an internal standard (70%, dr = 14/1). NMR data match with the previously reported data.²²

^1H NMR (CDCl_3 , 500 MHz): δ 5.82-5.74 (1H, m), 5.20-5.12 (3H, m), 3.49-3.42 (1H, m), 2.20-0.78 (17H, m)

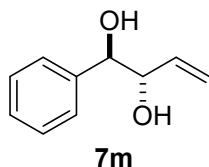
Procedure for preparation of 7m



In an argon-filled glove box, CrCl_2 (1.5 mg, 0.0125 mmol, 5 mol%), **HAT1** (12.4 mg, 0.025 mmol, 10 mol%), **PC1** (6.1 mg, 0.0125 mmol, 5 mol%), and K_2HPO_4 (4.4 mg, 0.025 mmol, 10 mol%) were dissolved in degassed CH_2Cl_2 (1.5 mL) and MeCN (1 mL) in a screw-capped vial. Then, allyloxytrimethylsilane **1m** (824 μL , 5.0 mmol, 20 equiv) and benzaldehyde **5a** (25.5 μL , 0.25 mmol, 1 equiv) were added to the reaction mixture. The vial was subjected to 430 nm LED irradiation by a Valore VBP-L24-C2 with 38W LED lamp (VBL-SE150-BBB (430)) for 24 hours under temperature control (ca. 27–29 $^\circ\text{C}$). Then, the reaction mixture was diluted by hexane (ca. 2 mL) and passed through a pad of silica gel with CH_2Cl_2 elution. After evaporation, the residue was dissolved in MeOH (ca. 2 mL) and 1 N HCl aqueous solution (ca. 3 mL) was added. The mixture was stirred for 30 min at room temperature. Organic materials were extracted with EtOAc three times. Combined organic layers were washed with brine, dried over Na_2SO_4 , filtered, and concentrated by evaporation. The residue was purified by silica gel flash column chromatography

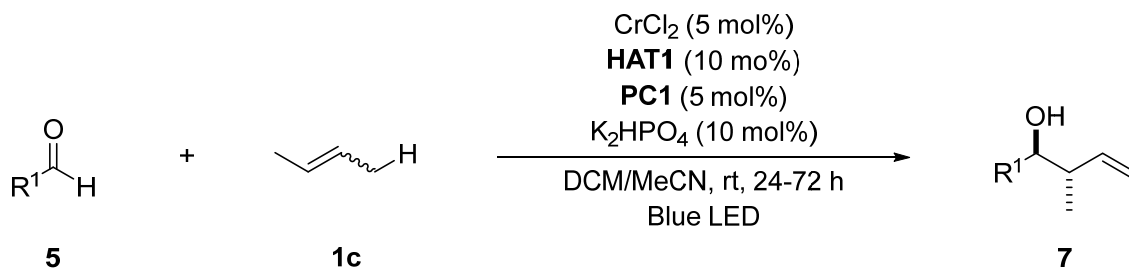
(EtOAc/hexane = 6% to 50%, v/v) to afford **7m** as a yellow oil (33.4 mg, 81%). Diastereomeric ratio was determined by ¹H NMR analysis (1.7/1 dr).

1-phenylbut-3-ene-1,2-diol (**7m**).



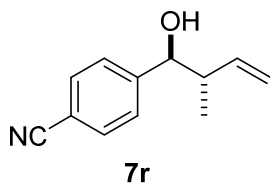
¹H NMR (CDCl₃, 500 MHz): major isomer: δ 7.38-7.26 (m, 5H), 5.86-5.77 (m, 1H), 5.33-5.19 (m, 2H), 4.77 (d, *J* = 4.6 Hz 1H), 4.36-4.29 (m, 1H), 2.27 (m, 2H); minor isomer: δ 7.38-7.26 (m, 5H), 5.74 (ddd, *J* = 17.2, 10.3, 5.2 Hz, 1H), 5.29-5.19 (m, 1H), 5.18-5.12 (m, 1H), 4.51 (d, *J* = 6.9 Hz, 1H), 4.27-4.20 (m, 1H), 2.27 (m, 2H); ¹³C NMR (CDCl₃, 125 MHz): major isomer: δ 139.7, 135.8, 128.4, 128.0, 126.6, 117.9, 76.7, 76.5; minor isomer: δ 140.1, 136.3, 128.4, 128.1, 127.0, 117.1, 77.5, 76.9. All the spectroscopic data match with the previously reported data.^{23,24}

Procedure for preparation of **7r**, **7x**, **7ac**, **7ae-ai**



In an argon-filled glove box, CrCl₂ (1.5 mg, 0.0125 mmol, 5 mol%), **HAT1** (12.4 mg, 0.025 mmol, 10 mol%), **PC1** (6.1 mg, 0.0125 mmol, 5 mol%), and K₂HPO₄ (4.4 mg, 0.025 mmol, 10 mol%) were dissolved in degassed CH₂Cl₂ (1.5 mL) and MeCN (1 mL) in a screw-capped tube. Then, aldehydes **5** (0.25 mmol, 1 equiv) were added to the reaction mixture. The reaction tube was removed from the glove box and the reaction mixture was cooled to -78 °C. Liquid 2-butene **1c** (ca. 1 mL) cooled at -78 °C was added to the reaction mixture via syringe. The reaction mixture was warmed to room temperature and subjected to 430 nm LED irradiation by a Kessil 40W LED lamp (A160WE Tuna Blue) for 24–72 hours under temperature control (ca. 35–38 °C). Then, the reaction mixture was added to water and organic materials were extracted with EtOAc three times. Combined organic layers were washed with brine, dried over Na₂SO₄, filtered, and concentrated by evaporation. Diastereomeric ratio was determined by ¹H NMR analysis. The residue was purified by silica gel flash column chromatography to afford the target homoallylic alcohols **7**.

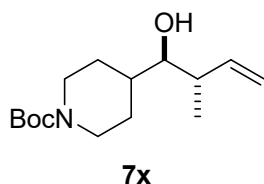
4-(1-hydroxy-2-methylbut-3-en-1-yl)benzotrile (**7r**).



The reaction was conducted using 4-formylbenzonitrile (32.8 mg, 0.25 mmol, 1 equiv) for 24 hours. The residue was purified by silica gel flash column chromatography (EtOAc/hexane = 8% to 40%, v/v) to afford **7r** as a colorless liquid (45.6 mg, 98%, dr = 9.4/1).

¹H NMR (CDCl₃, 500 MHz): δ 7.64 (2H, d, *J* = 8.3 Hz), 7.45 (2H, d, *J* = 8.3 Hz), 5.72 (1H, ddd, *J* = 17.2, 10.3, 8.0 Hz), 5.23-5.16 (2H, m), 4.45 (1H, d, *J* = 7.4 Hz), 2.49-2.39 (1H, m), 0.91 (3H, d, *J* = 6.9 Hz); ¹³C NMR (CDCl₃, 100 MHz): δ 147.8, 139.3, 132.0, 127.5, 118.8, 117.9, 111.4, 76.8, 46.4, 16.3. All the spectroscopic data match with the previously reported data.²⁵

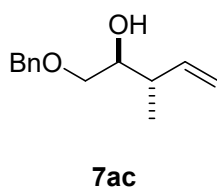
***tert*-butyl 4-(1-hydroxy-2-methylbut-3-en-1-yl)piperidine-1-carboxylate (7x).**



The reaction was conducted using *tert*-butyl 4-formylpiperidine-1-carboxylate (53.3 mg, 0.25 mmol, 1 equiv) for 24 hours. The residue was purified by silica gel flash column chromatography (EtOAc/hexane = 0% to 100%, v/v) to afford **7x** as a colorless liquid (67.3 mg, 100%, dr = 16/1).

¹H NMR (CDCl₃, 500 MHz): δ 5.78 (1H, ddd, *J* = 17.2, 10.3, 8.0 Hz), 5.16-5.08 (2H, m), 4.20-4.10 (2H, m), 3.15 (1H, t, *J* = 5.7 Hz), 2.70-2.58 (2H, m), 2.41-2.32 (1H, m), 1.80-1.72 (1H, m), 1.60-1.50 (2H, m), 1.45 (9H, s), 1.38-1.22 (2H, m), 1.05 (3H, d, *J* = 6.9 Hz); ¹³C NMR (CDCl₃, 100 MHz): δ 154.7, 139.6, 116.4, 79.2, 77.8, 43.7, 40.4, 38.9, 28.4, 26.7, 17.0; *m/z* calcd for C₁₅H₂₇NO₃Na [M+Na]⁺ 292.1883. Found 292.1888; IR (neat): 3457, 2974, 2929, 1673, 1427, 1171 cm⁻¹

1-(benzyloxy)-3-methylpent-4-en-2-ol (7ac).



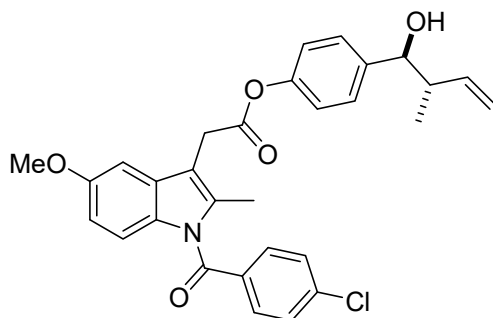
The reaction was conducted using benzyloxyacetaldehyde (34.4 μL, 0.25 mmol, 1 equiv) for 24 hours. The residue was purified by silica gel flash column chromatography (EtOAc/hexane = 8% to 40%, v/v) to afford **7ac** as a colorless liquid (37.4 mg, 73%, dr = 16/1).

¹H NMR (CDCl₃, 400 MHz): δ 7.39-7.26 (5H, m), 5.88-5.76 (1H, m), 5.12-5.04 (2H, m), 4.56 (2H, s),

3.71-3.64 (1H, m), 3.55 (1H, dd, $J = 9.6, 3.2$ Hz), 3.42 (1H, dd, $J = 9.6, 7.9$ Hz), 2.41-2.30 (1H, m), 1.03 (3H, d, $J = 6.9$ Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 140.0, 138.0, 128.4, 127.7, 127.7, 115.5, 73.5, 73.3, 72.5, 40.8, 16.1; m/z calcd for $\text{C}_{13}\text{H}_{18}\text{O}_2\text{Na}$ $[\text{M}+\text{Na}]^+$ 229.1199. Found 229.1208; IR (neat): 3447, 2868, 1638, 1454, 1098, 698 cm^{-1}

4-(1-hydroxy-2-methylbut-3-en-1-yl)phenyl

2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1H-indol-3-yl)acetate (7ae).

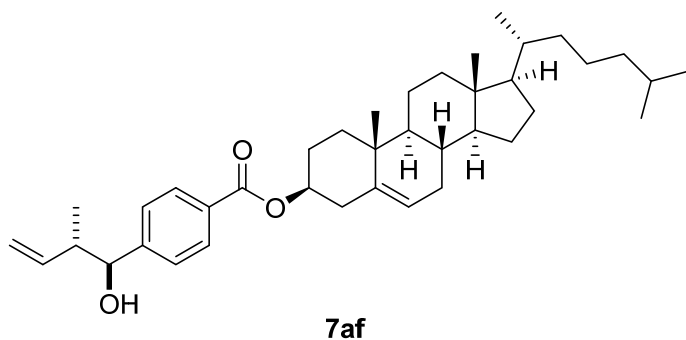


7ae

The reaction was conducted using aldehyde **5c** (115 mg, 0.25 mmol, 1 equiv) for 72 hours. The residue was purified by silica gel flash column chromatography (EtOAc/hexane = 12% to 60%, v/v) to afford **7ae** as a white solid (115 mg, 89%, dr = 14/1).

^1H NMR (CDCl_3 , 500 MHz): δ 7.68 (2H, d, $J = 8.6$ Hz), 7.48 (2H, d, $J = 8.6$ Hz), 7.32 (2H, d, $J = 8.6$ Hz), 7.08-7.02 (3H, m), 6.89 (1H, d, $J = 9.2$ Hz), 6.69 (1H, dd, $J = 9.2, 2.9$ Hz), 5.78 (1H, ddd, $J = 17.2, 10.9, 8.6$ Hz), 5.22-5.16 (2H, m), 4.36 (1H, d, $J = 7.4$ Hz), 3.90 (2H, s), 3.84 (3H, s), 2.48-2.39 (4H, m), 0.86 (3H, d, $J = 6.9$ Hz); ^{13}C NMR (CDCl_3 , 125 MHz): δ 169.2, 168.2, 156.0, 149.9, 140.2, 140.1, 139.2, 136.1, 133.7, 131.1, 130.7, 130.4, 129.0, 127.7, 121.0, 116.8, 114.9, 111.9, 111.7, 101.1, 76.7, 55.6, 46.1, 30.4, 16.3, 13.3; m/z calcd for $\text{C}_{30}\text{H}_{28}\text{O}_5\text{NCINa}$ $[\text{M}+\text{Na}]^+$ 540.1548. Found 540.1541; IR (neat): 3524, 2966, 2930, 1684, 1322, 1130, 926 cm^{-1}

(3*S*,8*S*,9*S*,10*R*,13*R*,14*S*,17*R*)-10,13-dimethyl-17-((*R*)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl 4-(1-hydroxy-2-methylbut-3-en-1-yl)benzoate (7af).

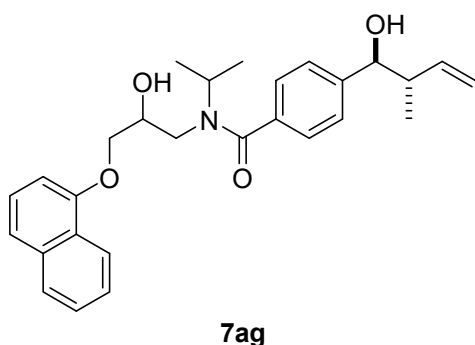


The reaction was conducted using (3*S*,8*S*,9*S*,10*R*,13*R*,14*S*,17*R*)-10,13-dimethyl-17-((*R*)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl 4-formylbenzoate (130 mg, 0.25 mmol, 1 equiv) for 24 hours. The residue was purified by silica gel flash column chromatography (EtOAc/hexane = 8% to 40%, v/v) to afford **7af** as a white solid (86.3 mg, 60%, dr = 16/1).

¹H NMR (CDCl₃, 400 MHz): δ 8.01 (2H, d, *J* = 8.5 Hz), 7.39 (2H, d, *J* = 8.5 Hz), 5.83-5.70 (1H, m), 5.42 (1H, d, *J* = 4.6 Hz), 5.24-5.16 (2H, m), 4.92-4.80 (1H, m), 4.44 (1H, d, *J* = 7.3 Hz), 2.53-2.41 (3H, m), 2.08-0.82 (42H, m), 0.69 (3H, s); ¹³C NMR (CDCl₃, 100 MHz): δ 165.8, 147.4, 139.9, 139.6, 130.1, 129.6, 129.5, 126.7, 125.6, 122.8, 117.3, 74.5, 60.4, 56.7, 56.1, 50.0, 46.3, 42.3, 39.7, 39.5, 38.2, 37.0, 36.6, 36.2, 35.8, 31.9, 31.8, 28.2, 28.0, 27.9, 24.3, 23.8, 22.8, 22.6, 21.0, 19.4, 18.7, 16.3, 11.8; *m/z* calcd for C₃₉H₅₈O₃Na [M+Na]⁺ 597.4278. Found 597.4261; IR (neat): 3502, 2947, 1716, 1457, 1275, 1113, 1017 cm⁻¹

Note: In the ¹³C NMR spectra additional signals were observed. These correspond to the pseudoenantiomer forms of the product. In the ¹H NMR spectra the signals could not be distinguished.

4-(1-hydroxy-2-methylbut-3-en-1-yl)-*N*-(2-hydroxy-3-(naphthalen-1-yloxy)propyl)-*N*-isopropylbenzamide (7ag).



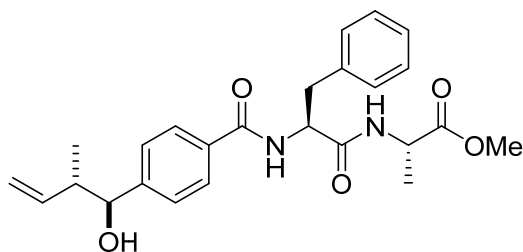
The reaction was conducted using aldehyde **5d** (97.9 mg, 0.25 mmol, 1 equiv) for 72 hours. The residue was purified by silica gel flash column chromatography (MeOH/CH₂Cl₂ = 0% to 20%, v/v) to afford **7ag** as a white solid (63.7 mg, 57%, dr = 10/1).

¹H NMR (CDCl₃, 400 MHz): δ 8.24 (1H, d, *J* = 7.3 Hz), 7.86-7.80 (1H, m), 7.52-7.35 (7H, m), 6.89 (1H, d, *J*

= 9.2 Hz), 6.90 (1H, d, $J = 7.8$ Hz), 5.78 (1H, ddd, $J = 17.4, 10.5, 8.7$ Hz), 5.29-5.20 (2H, m), 4.43-4.26 (3H, m), 4.18-4.02 (2H, m), 4.00-3.86 (1H, m), 3.78-3.69 (1H, m), 2.52-2.42 (1H, m), 1.34-1.16 (6H, m), 0.90 (3H, d, $J = 6.9$ Hz); ^{13}C NMR (CDCl_3 , 100 MHz): δ 171.2, 154.0, 140.1, 134.5, 127.6, 127.3, 126.4, 126.1, 126.1, 125.9, 125.4, 125.3, 121.5, 120.7, 117.6, 117.6, 104.8, 76.7, 72.2, 69.8, 60.4, 51.2, 51.1, 46.5, 21.4, 21.0, 20.8, 16.4, 14.2; m/z calcd for $\text{C}_{28}\text{H}_{33}\text{O}_4\text{NNa}$ $[\text{M}+\text{Na}]^+$ 470.2302. Found 470.2305; IR (neat): 3395, 2967, 2929, 1605, 1457, 1102, 772 cm^{-1}

Note: In the ^{13}C NMR spectra additional signals were observed. These correspond to the pseudoenantiomer forms of the product. In the ^1H NMR spectra the signals could not be distinguished.

methyl (4-(1-hydroxy-2-methylbut-3-en-1-yl)benzoyl)-*L*-phenylalanyl-*L*-alaninate (7ah).



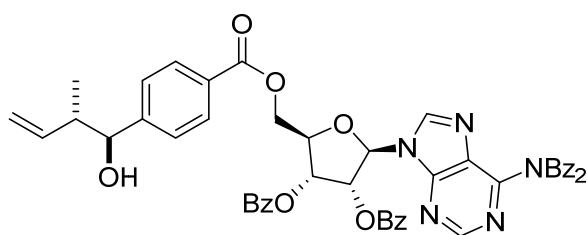
7ah

The reaction was conducted using aldehyde **5e** (95.6 mg, 0.25 mmol, 1 equiv) for 24 hours. The residue was purified by silica gel flash column chromatography ($\text{MeOH}/\text{CH}_2\text{Cl}_2 = 0\%$ to 20% , v/v) to afford **7ah** as a white solid (108 mg, 98%, dr = 11/1).

^1H NMR (CDCl_3 , 500 MHz): δ 7.71 (2H, d, $J = 7.7$ Hz), 7.39 (2H, d, $J = 7.7$ Hz), 7.34-7.22 (5H, m), 6.88-6.76 (1H, m), 6.32-6.24 (1H, m), 5.81-5.71 (1H, m), 5.22-5.16 (2H, m), 4.90-4.80 (1H, m), 4.54-4.44 (1H, m), 4.42 (1H, d, $J = 7.4$ Hz), 3.73 (3H, s), 3.26 (1H, dd, $J = 13.7, 5.7$ Hz), 3.13 (1H, dd, $J = 13.7, 8.0$ Hz), 2.50-2.42 (1H, m), 1.35 (3H, d, $J = 7.4$ Hz), 0.89 (3H, d, $J = 6.9$ Hz); ^{13}C NMR (CDCl_3 , 125 MHz): δ 172.7, 171.3, 171.2, 167.2, 167.1, 146.5, 146.5, 139.9, 139.9, 136.6, 132.5, 132.5, 129.3, 128.3, 127.0, 126.7, 116.6, 116.6, 77.3, 54.7, 52.2, 48.1, 45.8, 45.8, 38.2, 17.6, 17.6, 16.2; m/z calcd for $\text{C}_{25}\text{H}_{30}\text{O}_5\text{N}_2\text{Na}$ $[\text{M}+\text{Na}]^+$ 461.2047. Found 461.2050; IR (neat): 3290, 3066, 2955, 1748, 1634, 1541, 1207, 739 cm^{-1}

Note: In the ^{13}C NMR spectra additional signals were observed. These correspond to the pseudoenantiomer forms of the product. In the ^1H NMR spectra the signals could not be distinguished.

(2*R*,3*R*,4*R*,5*R*)-2-(6-(*N*-benzoylbenzamido)-9*H*-purin-9-yl)-5-(((4-(1-hydroxy-2-methylbut-3-en-1-yl)benzoyl)oxy)methyl)tetrahydrofuran-3,4-diyl dibenzoate (7ai).



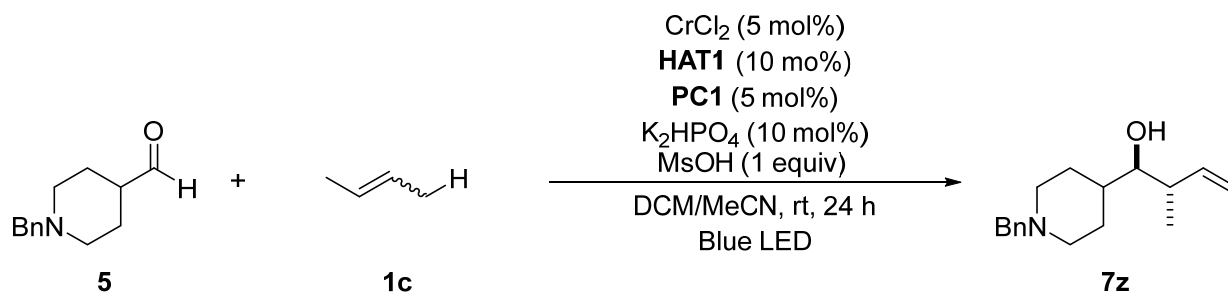
7ai

The reaction was conducted using aldehyde **5f** (204 mg, 0.25 mmol, 1 equiv) for 24 hours. The residue was purified by silica gel flash column chromatography (EtOAc/hexane = 20% to 100%, v/v) to afford **7ai** as a white solid (163 mg, 75%, dr = 14/1).

^1H NMR (CDCl_3 , 500 MHz): δ 8.52 (1H, d, $J = 4.0$ Hz), 8.22 (1H, s), 8.08-7.78 (10H, m), 7.62-7.34 (14H, m), 6.50-6.44 (1H, m), 6.41-6.35 (1H, m), 6.28-6.20 (1H, m), 5.82-5.70 (1H, m), 5.22-5.12 (2H, m), 4.94-4.78 (2H, m), 4.73-4.62 (1H, m), 4.44 (1H, d, $J = 7.4$ Hz), 2.52-2.42 (1H, s), 2.23 (1H, brs), 0.89 (3H, d, $J = 6.9$ Hz); ^{13}C NMR (CDCl_3 , 125 MHz): δ 172.1, 165.9, 165.2, 165.0, 152.5, 151.9, 148.4, 143.6, 139.7, 133.7, 133.6, 132.9, 129.7, 129.7, 129.5, 129.3, 128.6, 128.5, 128.4, 128.3, 128.2, 127.7, 126.9, 116.9, 87.0, 80.7, 77.3, 73.7, 71.1, 63.3, 45.8, 45.8, 16.2; m/z calcd for $\text{C}_{50}\text{H}_{41}\text{O}_{10}\text{N}_5\text{Na}$ $[\text{M}+\text{Na}]^+$ 894.2746. Found 894.2766; IR (neat): 3525, 3063, 2973, 1725, 1600, 1578, 1450, 1267, 1094, 710 cm^{-1}

Note: In the ^{13}C NMR spectra additional signals were observed. These correspond to the pseudoenantiomer forms of the product. In the ^1H NMR spectra the signals could not be distinguished.

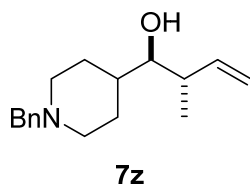
Procedure for preparation of **7z**



In an argon-filled glove box, CrCl_2 (1.5 mg, 0.0125 mmol, 5 mol%), **HAT1** (12.4 mg, 0.025 mmol, 10 mol%), **PC1** (6.1 mg, 0.0125 mmol, 5 mol%), and K_2HPO_4 (4.4 mg, 0.025 mmol, 10 mol%) were dissolved in degassed CH_2Cl_2 (1.5 mL) and MeCN (1 mL) in a screw-capped tube. Then, 1-benzylpiperidine-4-carbaldehyde (48.9 μL , 0.25 mmol, 1 equiv) and MsOH (16.2 μL , 0.25 mmol, 1 equiv) were added to the reaction mixture. The reaction tube was removed from the glove box and the reaction mixture was cooled to -78 $^\circ\text{C}$. Liquid 2-butene **1c** (ca. 1 mL) cooled at -78 $^\circ\text{C}$ was added to the reaction mixture via syringe. The reaction mixture was warmed to room temperature and subjected to 430 nm LED irradiation by a Kessil 40W LED lamp (A160WE Tuna Blue) for 24 hours under temperature control (ca.

35–38 °C). Then, the reaction mixture was added to water and organic materials were extracted with EtOAc three times. Combined organic layers were washed with brine, dried over Na₂SO₄, filtered, and concentrated by evaporation. Diastereomeric ratio was determined by ¹H NMR analysis (12/1 dr). The residue was purified by silica gel flash column chromatography (MeOH/CH₂Cl₂ = 0% to 20%, v/v) to afford **7z** as a colorless liquid (44.5 mg, 69%).

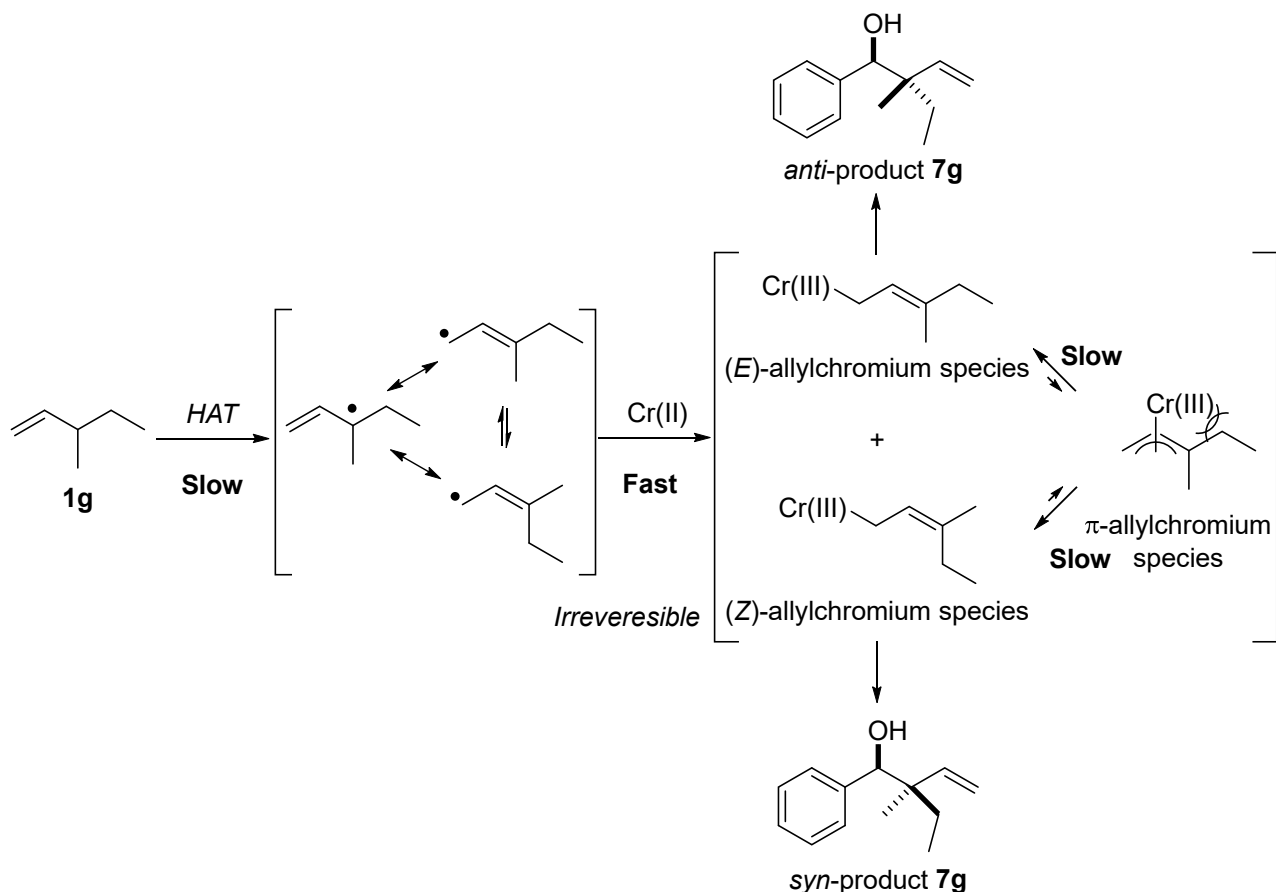
1-(1-benzylpiperidin-4-yl)-2-methylbut-3-en-1-ol (7z).



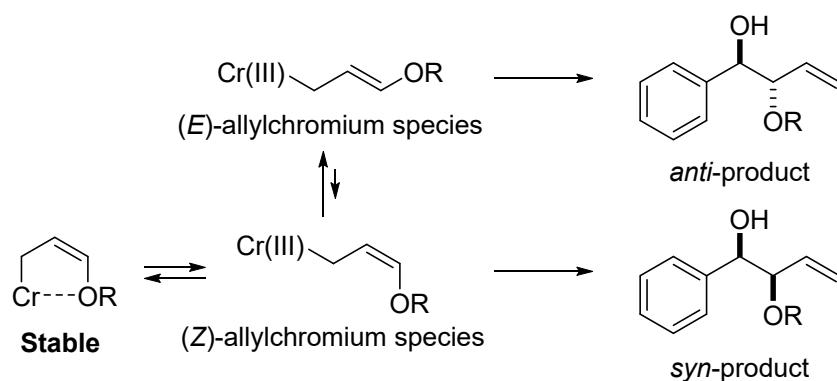
¹H NMR (CDCl₃, 500 MHz): δ 7.34-7.24 (5H, m), 5.78 (1H, ddd, *J* = 17.2, 10.3, 8.0 Hz), 5.14-5.06 (2H, m), 3.56 (2H, m), 3.15 (1H, dd, *J* = 5.2, 5.2 Hz), 2.99 (2H, t, *J* = 10.9 Hz), 2.40-2.32 (1H, m), 2.08-1.68 (4H, m), 1.62-1.38 (5H, m), 1.04 (3H, d, *J* = 6.9 Hz); ¹³C NMR (CDCl₃, 100 MHz): δ 139.5, 129.4, 128.2, 127.2, 116.1, 78.2, 63.0, 53.4, 53.4, 40.2, 38.6, 28.3, 26.9, 17.1; *m/z* calcd for C₁₇H₂₅ONNa [M+Na]⁺ 282.1828. Found 282.1815; IR (neat): 3393, 2916, 2360, 1456, 1095 cm⁻¹

4. Rationalization of Low Diastereoselectivity of 7g, 7m, and 7n

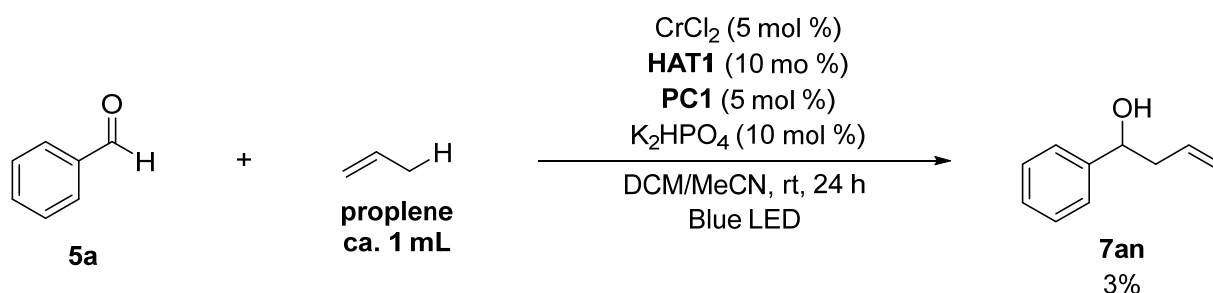
We suppose that low diastereoselectivity of **7g** is due to a low *E/Z* ratio of the intermediate allylchromium species, which is attributable to slow isomerization between (*E*)- and (*Z*)-allylchromium species derived from **1g** (see the scheme below). In this reaction, allyl radical generated by the HAT process from **1g** was oxidatively trapped by the chromium(II) complex, affording a mixture of (*E*)- and (*Z*)-allylchromium species irreversibly.²⁶ The σ-allylchromium(III) species containing a tri-substituted C=C double bond is known to be configurationally stable due to the steric hindrance.²⁷ Thus, steric hindrance prevents the formation of the π-allylchromium complex(III), making isomerization between (*E*)- and (*Z*)-allylchromium species difficult. This will cause a low *E/Z* ratio of the allylchromium species. Because the C-C bond formation between allylchromium species and aldehydes proceeds stereoselectively through a Zimmerman-Traxler transition state, low *E/Z*-selectivity and slow isomerization rate between (*E*)- and (*Z*)-allylchromium species will lead to low diastereoselectivity of **7g** (2.3/1).



Low diastereoselectivity of **7m** and **7n** is likely due to coordination of the oxygen atom to the chromium center in the **(Z)-allylchromium species** derived from **1m** and **1n**. Without the oxygen atom, **(E)-allylchromium species** are thermodynamically more stable than **(Z)-allylchromium species**. However, coordination of the oxygen atom to the chromium center will favorably contribute to increasing the concentration of the **(Z)-allylchromium species**. Furthermore, this intramolecular coordination will also decrease the *E/Z* isomerization rate.²⁸ These two factors should cause low *E/Z* ratio of the allylchromium species. Thus, the allylation reaction could proceed from both **(E)-** and **(Z)-allylchromium species**, resulting in low diastereoselectivity.

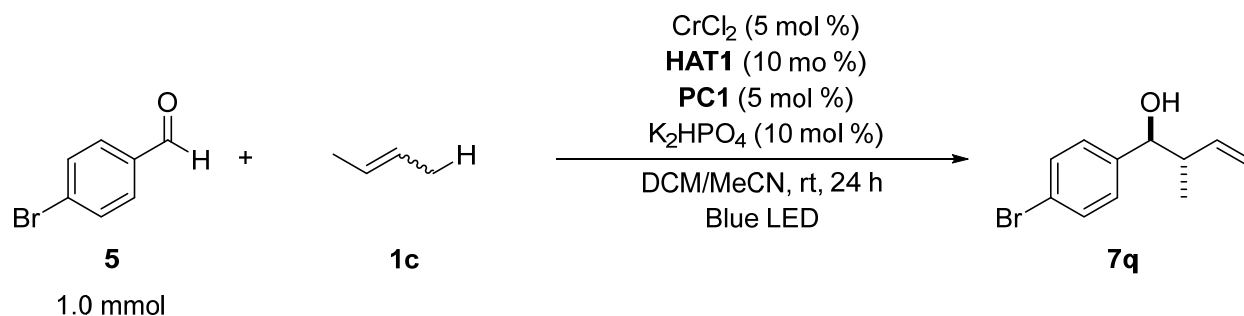


5. Reaction with Propene



In an argon-filled glove box, CrCl_2 (1.5 mg, 0.0125 mmol, 5 mol%), **HAT1** (12.4 mg, 0.025 mmol, 10 mol%), **PC1** (6.1 mg, 0.0125 mmol, 5 mol%), and K_2HPO_4 (4.4 mg, 0.025 mmol, 10 mol%) were dissolved in degassed CH_2Cl_2 (1.5 mL) and MeCN (1 mL) in a screw-capped tube. Then, benzaldehyde **5a** (25.5 μL , 0.25 mmol, 1 equiv) was added to the reaction mixture. The reaction tube was removed from the glove box, and the reaction mixture was cooled to -78°C . Gaseous propylene (GL science Cat.No. 1020-11006 ca. 1 mL) was directly added into the reaction mixture cooled at -78°C via needle. The reaction mixture was warmed to room temperature and subjected to 430 nm LED irradiation by a Kessil 40W LED lamp (A160WE Tuna Blue) for 24 hours under temperature control (ca. $35\text{--}38^\circ\text{C}$). Then, the reaction mixture was diluted with hexane (ca. 2 mL) and passed through a pad of silica gel with CH_2Cl_2 elution. After evaporation, yield of **7an** was determined to be 3% by ^1H NMR analysis using 1,1,2,2-tetrachloroethane as an internal standard. Benzaldehyde **5a** was recovered in 92%.

6. Experimental Procedure for the Large-Scale Reaction

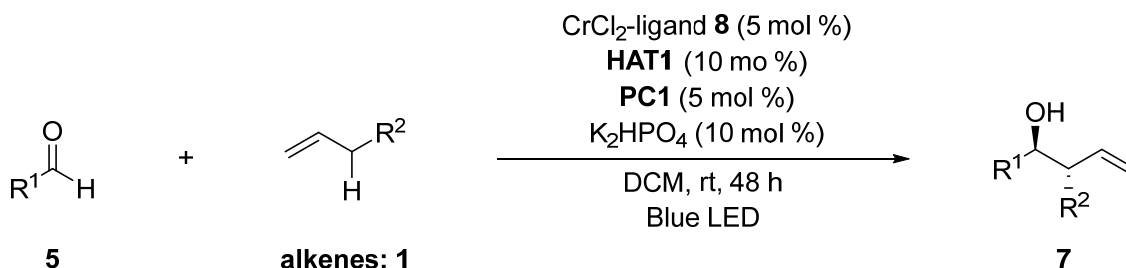


In an argon-filled glove box, CrCl_2 (6.2 mg, 0.050 mmol, 5 mol%), **HAT1** (49.5 mg, 0.10 mmol, 10 mol%), **PC1** (24.5 mg, 0.050 mmol, 5 mol%), and K_2HPO_4 (17.4 mg, 0.010 mmol, 10 mol%) were dissolved in degassed CH_2Cl_2 (6.0 mL) and MeCN (4.0 mL) in a screw-capped tube. Then, 4-bromobenzaldehyde (185 mg, 0.10 mmol, 1 equiv) was added to the reaction mixture. The reaction tube was removed from the glove box and the reaction mixture was cooled to -78°C . Liquid 2-butene **1c** (ca. 4 mL) cooled at -78°C was added to the reaction mixture via syringe. The reaction mixture was warmed to room temperature and subjected to 430 nm LED irradiation by a Kessil 40W LED lamp (A160WE Tuna Blue) for 24 hours under temperature control (ca. $35\text{--}38^\circ\text{C}$). Then, the reaction mixture was diluted by hexane (ca. 8 mL) and passed through a pad of silica gel with CH_2Cl_2 elution. After evaporation, diastereomeric ratio was determined by

^1H NMR analysis (19/1 dr). The residue was purified by silica gel flash column chromatography ($\text{CH}_2\text{Cl}_2/\text{hexane} = 18\%$ to 100% , v/v) to afford **7q** as a colorless liquid (233 mg, 97%).

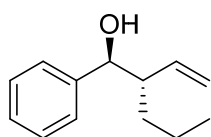
7. General Procedure for Catalytic Asymmetric Allylation of Aldehydes

Procedure for preparation of (*S,S*)-**7a**, (*S,S*)-**7f**, (*S,R*)-**7k**



In an argon-filled glove box, CrCl_2 (1.5 mg, 0.0125 mmol, 5 mol%) and ligand **8** (4.5 mg, 0.0125 mmol, 5 mol%) were dissolved in degassed CH_2Cl_2 (2.5 mL) in a screw-capped vial and the mixture was stirred for 1 hour at room temperature. Then, **HAT1** (12.4 mg, 0.025 mmol, 10 mol%), **PC1** (6.1 mg, 0.0125 mmol, 5 mol%), K_2HPO_4 (4.4 mg, 0.025 mmol, 10 mol%), alkenes **1** (5.0 mmol, 20 equiv) and aldehydes **5** (0.25 mmol, 1 equiv) were added to the reaction mixture. The vial was subjected to 430 nm LED irradiation by a Valore VBP-L24-C2 with 38W LED lamp (VBL-SE150-BBB (430)) for 48 hours under temperature control (ca. 27–29 °C). Then, the reaction mixture was passed through a pad of silica gel with CH_2Cl_2 elution. After evaporation, diastereomeric ratio was determined by ^1H NMR analysis. The residue was purified by silica gel flash column chromatography to afford the target homoallylic alcohols **7**. The enantiomeric excess was determined by chiral HPLC analysis.

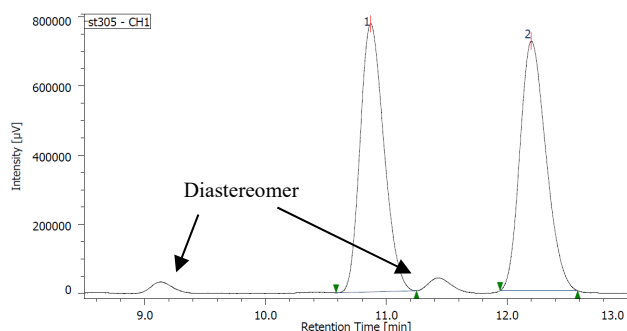
(*1S,2S*)-1-phenyl-2-vinylpentan-1-ol ((*S,S*)-**7a**).



(*S,S*)-**7a**

The reaction was conducted using benzaldehyde **5a** (25.5 μL , 0.25 mmol, 1 equiv) and 1-hexene **1a** (628 μL , 5.0 mmol, 20 equiv). The residue was purified by silica gel flash column chromatography ($\text{CH}_2\text{Cl}_2/\text{hexane} = 18\%$ to 100% , v/v) to afford (*S,S*)-**7a** as a light yellow oil (43.1 mg, 91%, dr = 4.2/1, 88 % ee).

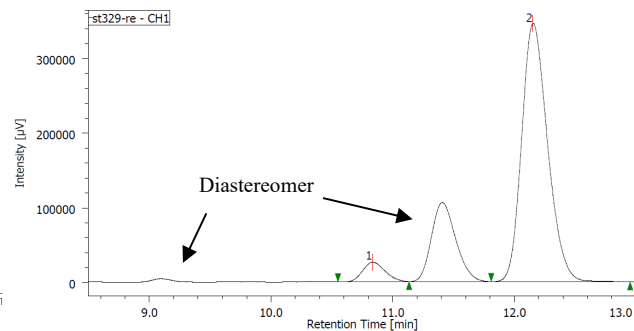
HPLC (chiral column: CHIRALPAK IC-3; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm): $t_{\text{R}} = 10.8$ min (minor) and 12.2 min (major); $[\alpha]_{\text{D}}^{20} = -60$ ($c = 1.66$, CHCl_3 , for 88% ee sample). All the spectroscopic data match with the previously reported data.⁷



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 コントロールメソッド Morita_1.0ml_210nm
 ピークID テーブル
 検量線テーブル
 追加情報

チャンネル情報+ピーク情報
 クロマトグラム名 st305-CH1
 サンプル名 OHI
 チャンネル名 OHI
 データ取り込み間隔 500 [msec]
 波形処理メソッド (マニュアル)
 数値計算式
 判定式

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1	Unknown	1	10.867	10602389	774719	49.542	51.809	N/A	14217	3.508	1.240	
2	Unknown	1	12.200	10788283	720738	50.458	48.195	N/A	15021	N/A	1.228	

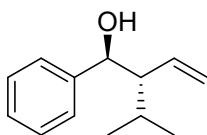


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チャンネル情報+ピーク情報
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 データ取り込み間隔 500 [msec]
 波形処理メソッド (マニュアル)
 数値計算式
 判定式

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1	Unknown	1	10.833	340568	26248	6.217	7.051	N/A	15879	3.610	1.220	
2	Unknown	1	12.150	5137160	346048	93.783	92.949	N/A	15736	N/A	1.240	

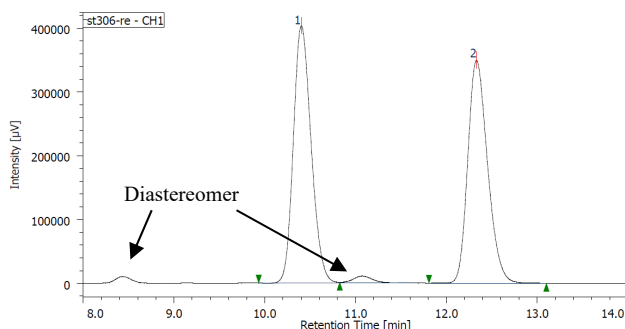
(1*S*,2*S*)-2-isopropyl-1-phenylbut-3-en-1-ol ((*S*, *S*)-7f).



(*S*,*S*)-7f

The reaction was conducted using benzaldehyde **5a** (25.5 μL, 0.25 mmol, 1 equiv) and 4-methyl-1-pentene **1f** (638 μL, 5 mmol, 20 equiv). The residue was purified by silica gel flash column chromatography (CH₂Cl₂/hexane = 18% to 100%, v/v) to afford (**S,S**)-**7f** as a light yellow oil (40.7 mg, 86%, dr = >20/1, 73 % ee).

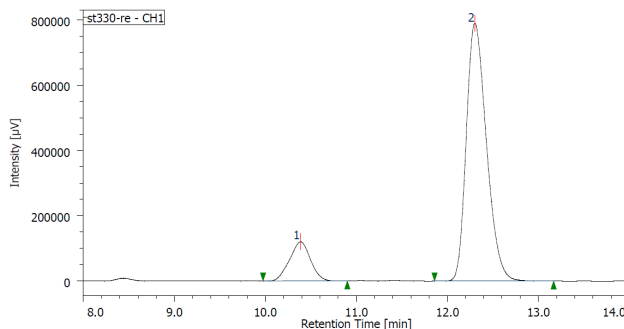
HPLC (chiral column: CHIRALPAK IC-3; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm): *t*_R = 10.4 min (minor) and 12.3 min (major); [α]_D²⁰ = -89 (*c* = 1.30, CHCl₃, for 73% ee sample). All the spectroscopic data match with the previously reported data.⁹



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 ピークID テーブル
 検量線テーブル
 追加情報

チャンネル情報+ピーク情報
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 チャンネル名 CH1
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 数値計算式
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1	Unknown	1	10.400	5481428	403157	50.900	53.584	N/A	13542	5.147	1.197	
2	Unknown	1	12.333	5287667	349233	49.100	46.416	N/A	15542	N/A	1.221	

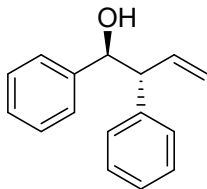


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チャンネル情報+ピーク情報
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 波形処理メソッド (マニュアル)
 数値計算式
 判定式

#	ピーク名	CH	tR [min]	面積 [μVsec]	高さ [μV]	面積%	高さ	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	10.383	1904910	119480	13.568	13.162	N/A	9729	4.858	0.993	
2	Unknown	1	12.300	12135287	788623	86.432	86.838	N/A	14833	N/A	1.302	

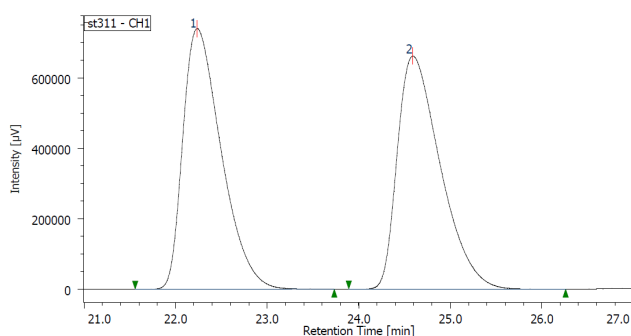
(1*S*,2*R*)-1,2-diphenylbut-3-en-1-ol ((*S*,*R*)-7k).



(*S*,*R*)-7k

The reaction was conducted using benzaldehyde **5a** (25.5 μL, 0.25 mmol, 1 equiv) and allylbenzene **11** (664 μL, 5.0 mmol, 20 equiv). The residue was purified by silica gel flash column chromatography (CH₂Cl₂/hexane = 18% to 100%, v/v) to afford (*S*,*R*)-**7k** as a light brown oil (38.2 mg, 68%, dr = >20/1, 72 % ee).

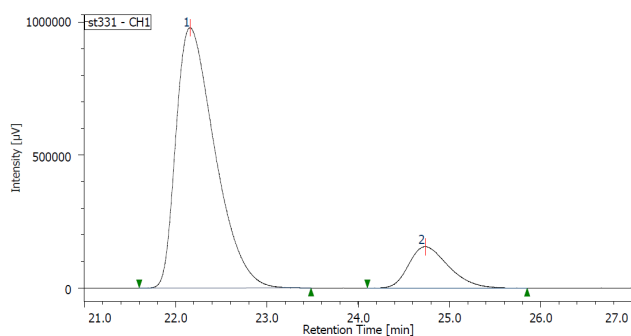
HPLC (chiral column: CHIRALPAK IC-3; solvent: hexane/2-propanol = 200/1; flow rate: 1.0 mL/min; detection: at 210 nm): $t_R = 22.2$ min (major) and 24.7 min (minor); $[\alpha]_D^{20} = -16$ ($c = 0.89$, CHCl₃, for 72% ee sample). All the spectroscopic data match with the previously reported data.¹²



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 追加情報

チャンネル情報+ピーク情報
 クロマトグラム名 st311-CH1
 サンプル名
 チャンネル名 CH1
 データ取り込み間隔 500 [msec]
 波形処理メソッド (マニュアル)
 数値計算式
 判定式

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1	Unknown	1	22.33	21521889	739247	49.93%	52.77%	N/A	13483	2.910	1.564	
2	Unknown	1	24.592	21575169	661432	50.06%	47.22%	N/A	13105	N/A	1.603	

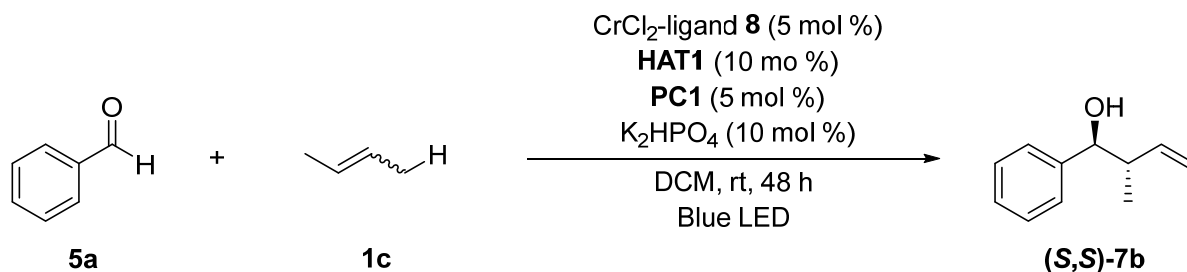


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 追加情報

チャンネル情報+ピーク情報
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 サンプル名
 チャンネル名 CH1
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 波形処理メソッド (マニュアル)
 数値計算式
 判定式

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1	Unknown	1	22.158	28971895	978645	88.04%	86.21%	N/A	12949	3.284	1.619	
2	Unknown	1	24.733	4700328	155331	13.99%	13.69%	N/A	15548	N/A	1.338	

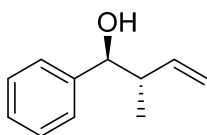
Procedure for preparation of (S,S)-7b



In an argon-filled glove box, CrCl_2 (1.5 mg, 0.0125 mmol, 5 mol%) and ligand **8** (4.5 mg, 0.0125 mmol, 5 mol%) were dissolved in degassed CH_2Cl_2 (2.5 mL) in a screw-capped tube and the mixture was stirred for 1 hour at room temperature. Then, **HAT1** (12.4 mg, 0.025 mmol, 10 mol%), **PC1** (6.1 mg, 0.0125 mmol, 5 mol%), K_2HPO_4 (4.4 mg, 0.025 mmol, 10 mol%), and benzaldehyde **5a** (25.5 μL, 0.25 mmol, 1 equiv) were added to the reaction mixture. The reaction tube was removed from the glove box and the reaction mixture was cooled to -78°C . Liquid 2-butene **1c** (ca. 1 mL) cooled at -78°C was added to the reaction mixture via syringe. The reaction mixture was warmed to room temperature and subjected to 430 nm LED irradiation by a Kessil 40W LED lamp (A160WE Tuna Blue) for 48 hours under temperature control (ca. 35 – 38°C). Then, the reaction mixture was passed through a pad of silica gel with CH_2Cl_2 elution. After evaporation, diastereomeric ratio was determined by ^1H NMR analysis (2.7/1 dr). The residue was purified by silica gel flash column chromatography (CH_2Cl_2 /hexane = 18% to 100%, v/v) to afford **(S,S)-7b** as a light yellow oil

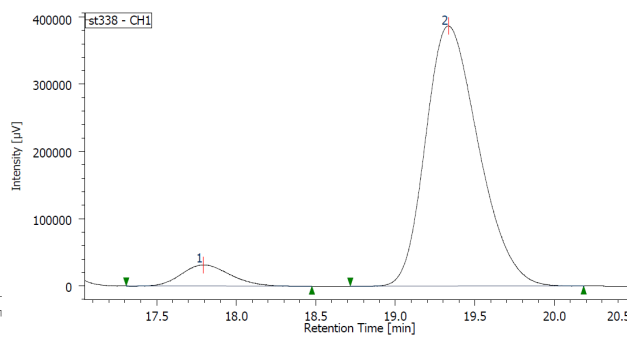
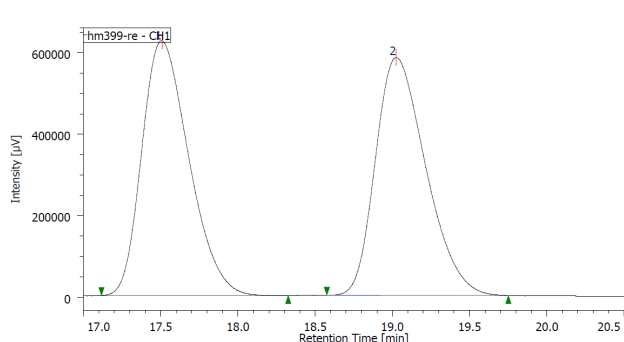
(29.5 mg, 73%). The enantiomeric excess was determined by chiral HPLC analysis (86 % ee).

(1*S*,2*S*)-2-methyl-1-phenylbut-3-en-1-ol ((*S,S*)-7b).



(*S,S*)-7b

HPLC (chiral column: CHIRALPAK IC-3; solvent: hexane/2-propanol = 200/1; flow rate: 1.0 mL/min; detection: at 210 nm): t_R = 17.8 min (minor) and 19.3 min (major); $[\alpha]_D^{20} = -87$ ($c = 1.16$, CHCl_3 , for 86% ee sample). All the spectroscopic data match with the previously reported data.¹⁶



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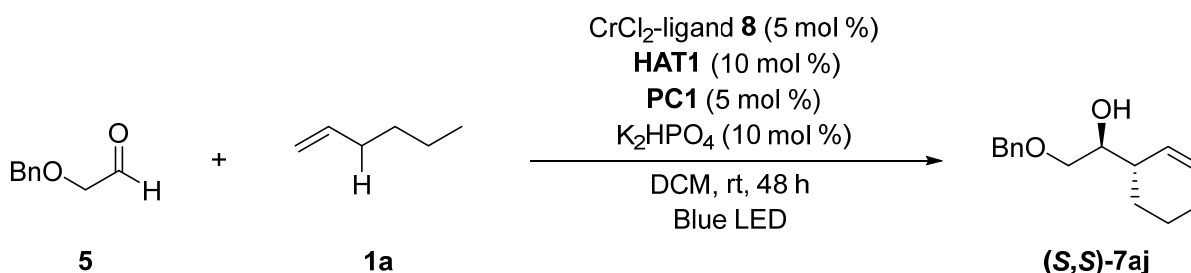
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#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シメトリー係数	警告
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2	Unknown	1	19.333	8946437	386676	93.022	92.472	N/A	16002	N/A	1.262	

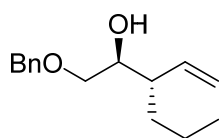
Procedure for preparation of (*S,S*)-7aj



In an argon-filled glove box, CrCl_2 (1.5 mg, 0.0125 mmol, 5 mol%) and ligand **8** (4.5 mg, 0.0125 mmol, 5 mol%) were dissolved in degassed CH_2Cl_2 (2.5 mL) in a screw-capped vial and the mixture was stirred for

1 hour at room temperature. Then, **HAT1** (12.4 mg, 0.025 mmol, 10 mol%), **PC1** (6.1 mg, 0.0125 mmol, 5 mol%), K_2HPO_4 (4.4 mg, 0.025 mmol, 10 mol%), 1-hexene **1a** (628 μ L, 5.0 mmol, 20 equiv) and benzyloxyacetaldehyde (35.1 μ L, 0.25 mmol, 1 equiv) were added to the reaction mixture. The vial was subjected to 430 nm LED irradiation by a Valore VBP-L24-C2 with 38W LED lamp (VBL-SE150-BBB (430)) for 48 hours under temperature control (ca. 27–29 °C). Then, the reaction mixture was added to water and organic materials were extracted with EtOAc three times. Combined organic layers were washed with brine, dried over Na_2SO_4 , filtered, and concentrated by evaporation. Diastereomeric ratio was determined by 1H NMR analysis (4.6/1 dr). The residue was purified by silica gel flash column chromatography (EtOAc/hexane = 5% to 100%, v/v) to afford **(S,S)-7aj** as a light yellow oil (42.6 mg, 73%). The enantiomeric excess was determined by chiral HPLC analysis (59 % ee).

(2S,3S)-1-(benzyloxy)-3-vinylhexan-2-ol ((S,S)-7aj).

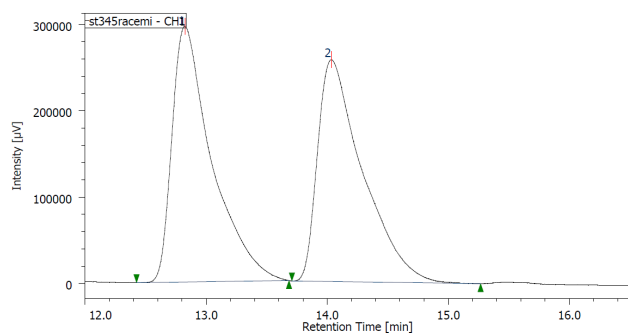


(S,S)-7aj

1H NMR ($CDCl_3$, 500 MHz): major isomer: δ 7.38-7.27 (m, 5H), 5.69 (ddd, $J = 17.2, 10.3, 9.2$ Hz, 1H), 5.12 (dd, $J = 10.3, 1.7$ Hz, 1H), 5.04 (dd, $J = 17.2, 1.7$ Hz, 1H), 4.55 (s, 2H), 3.75 (ddd, $J = 8.0, 5.2, 3.4$ Hz, 1H), 3.52 (dd, $J = 9.7, 3.4$ Hz, 1H), 3.43 (dd, $J = 9.7, 8.0$ Hz, 1H), 2.20-2.09 (m, 1H), 2.01 (brs, 1H), 1.50-1.14 (m, 4H), 0.88 (t, $J = 6.9$ Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): major isomer: δ 138.5, 138.0, 128.4, 127.7, 117.1, 105.3, 73.4, 72.9, 72.4, 46.7, 32.9, 20.3, 14.0; m/z calcd for $C_{15}H_{22}O_2Na$ $[M+Na]^+$ 257.1512. Found 257.1507; IR (neat): 3464, 2956, 2928, 2870, 1637, 1455, 1101, 913, 736, 698 cm^{-1} ; HPLC (chiral column: CHIRALPAK IA-3; solvent: hexane/2-propanol = 100/1; flow rate: 1.0 mL/min; detection: at 210 nm): $t_R = 13.3$ min (major) and 14.6 min (minor); $[\alpha]_D^{20} = +7.9$ ($c = 1.76$, $CHCl_3$, for 59% ee sample).

Resolved signals of the minor isomer

1H NMR ($CDCl_3$, 500 MHz): δ 5.53 (ddd, $J = 17.2, 10.3, 9.7$ Hz, 1H), 3.65 (ddd, $J = 10.9, 8.0, 2.9$ Hz, 1H), 3.57 (dd, $J = 9.4, 2.9$ Hz, 1H), 3.37 (dd, $J = 9.4, 8.0$ Hz, 1H).

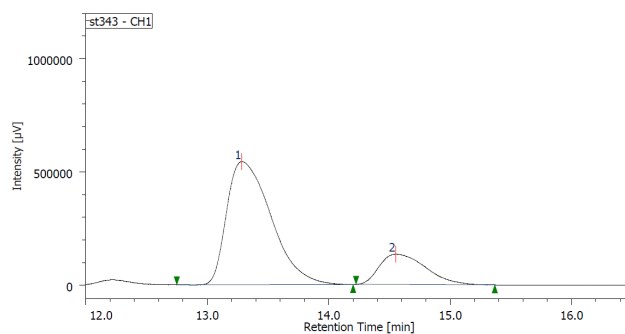


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 追加情報

チャンネル情報+ピーク情報

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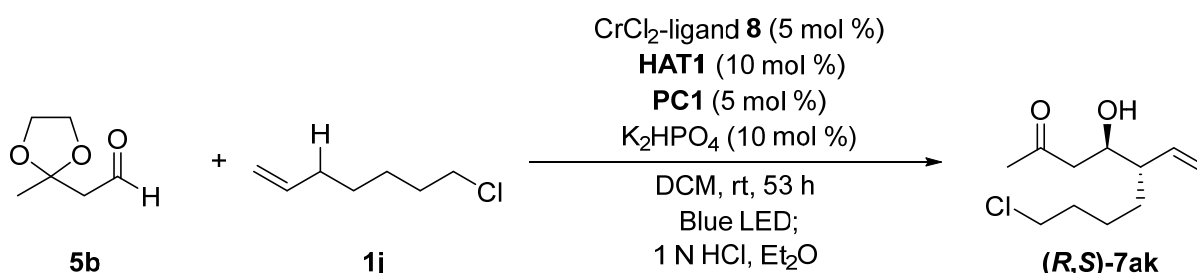
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チャンネル情報+ピーク情報

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 判定式

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1	Unknown	13.263	13794154	542792	78.52%	80.33%	N/A	6091	1.815	1.673	
2	Unknown	14.556	3550593	132745	20.47%	19.76%	N/A	6562	N/A	1.561	

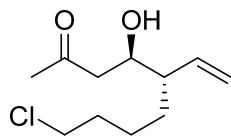
Procedure for preparation of (*R,S*)-7ak



In an argon-filled glove box, CrCl_2 (1.5 mg, 0.0125 mmol, 5 mol%) and ligand **8** (4.5 mg, 0.0125 mmol, 5 mol%) were dissolved in degassed CH_2Cl_2 (2.5 mL) in a screw-capped vial and the mixture was stirred for 1 hour at room temperature. Then, **HAT1** (12.4 mg, 0.025 mmol, 10 mol%), **PC1** (6.1 mg, 0.0125 mmol, 5 mol%), K_2HPO_4 (4.4 mg, 0.025 mmol, 10 mol%), 7-chlorohept-1-ene **1j** (750 μL, 5.0 mmol, 20 equiv) and aldehyde **5b** (32.5 mg, 0.25 mmol, 1 equiv) were added to the reaction mixture. The vial was subjected to 430 nm LED irradiation by a Valore VBP-L24-C2 with 38W LED lamp (VBL-SE150-BBB (430)) for 53 hours under temperature control (ca. 27–29 °C). Then, the reaction mixture was passed through a pad of silica gel with CH_2Cl_2 elution. The crude mixture was concentrated and re-suspended in Et₂O (1.5 mL), and 1N HCl aqueous solution (1.5 mL). After stirring overnight, the aqueous layer of the reaction mixture was separated and extracted with EtOAc three times. The combined organic layers were washed with saturated NaHCO_3 aqueous solution, dried over Na_2SO_4 , filtered, and concentrated. Diastereomeric ratio was determined by ¹H NMR analysis (3.6/1 dr). The residue was purified by silica gel flash column

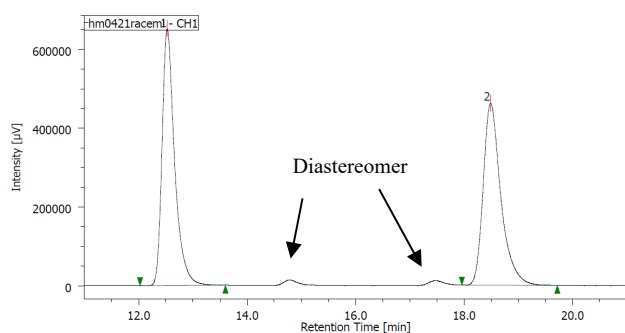
chromatography (EtOAc/hexane = 8% to 65%, v/v) to afford (**R,S**)-**7ak** as a colorless liquid (25.0 mg, 47%). The enantiomeric excess was determined by chiral HPLC analysis after protection of (**R,S**)-**7ak** by *p*-NO₂-benzoyl chloride (82 % ee).

(**4R,5S**)-9-chloro-4-hydroxy-5-vinylnonan-2-one ((**R,S**)-**7ak**).



(R,S)-7ak

¹H NMR (CDCl₃, 400 MHz): major isomer: δ 5.78 (1H, ddd, *J* = 17.4, 10.5, 9.6 Hz), 5.20-5.03 (2H, m), 4.08-4.00 (1H, m), 3.53 (2H, t, *J* = 6.9 Hz), 2.73-2.44 (2H, m), 2.18 (3H, s), 2.04-1.92 (1H, m), 1.84-1.68 (2H, m), 1.56-1.18 (4H, m); ¹³C NMR (CDCl₃, 100 MHz): major isomer: δ 209.9, 137.8, 117.8, 69.5, 49.5, 47.8, 44.9, 32.5, 30.8, 29.9, 24.6; HPLC (chiral column: CHIRALPAK IA-3; solvent: hexane/2-propanol = 10/1; flow rate: 1.0 mL/min; detection: at 254 nm): *t*_R = 12.5 min (major) and 18.5 min (minor); [α]_D²⁰ = +37 (*c* = 0.50, CHCl₃, for 82% ee sample). All the spectroscopic data match with the previously reported data.¹

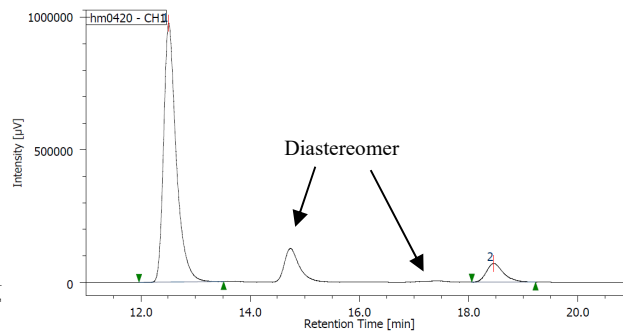


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 検査線テーブル
 追加情報

チャンネル情報+ピーク情報

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1	Unknown	1	12.52	1048152	652539	50.062	88.563	N/A	16009	12.468	1.473	
2	Unknown	1	18.483	10455381	461713	49.938	41.437	N/A	17287	N/A	1.429	



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チャンネル情報+ピーク情報

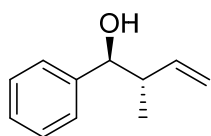
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1	Unknown	1	12.508	15640094	977118	90.988	93.277	N/A	15957	12.584	1.498	
2	Unknown	1	18.458	1549403	70425	9.014	6.723	N/A	17896	N/A	1.357	

Determination of absolute configuration

The absolute configurations of **7** were determined as follows:

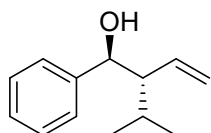
The absolute configuration was determined by optical rotation value of **7b**, **7f** and **7k**. Those of others were assigned by analogy.



(S,S)-7b

This time: $[\alpha]_D^{20} = -89$ ($c = 1.30$, CHCl_3 , for 73% ee sample)

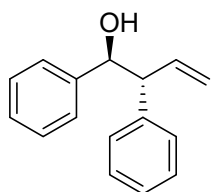
Reported value¹⁶: $[\alpha]_D^{26} = +93.18$ ($c = 1.09$, CHCl_3 , for 96% ee sample of **(R,R)-7b**)



(S,S)-7f

This time: $[\alpha]_D^{20} = -87$ ($c = 1.16$, CHCl_3 , for 86% ee sample)

Reported value²⁹: $[\alpha]_D^{25} = -83.4$ ($c = 0.5$, CHCl_3 , for 95% ee sample of **(S,S)-7f**)



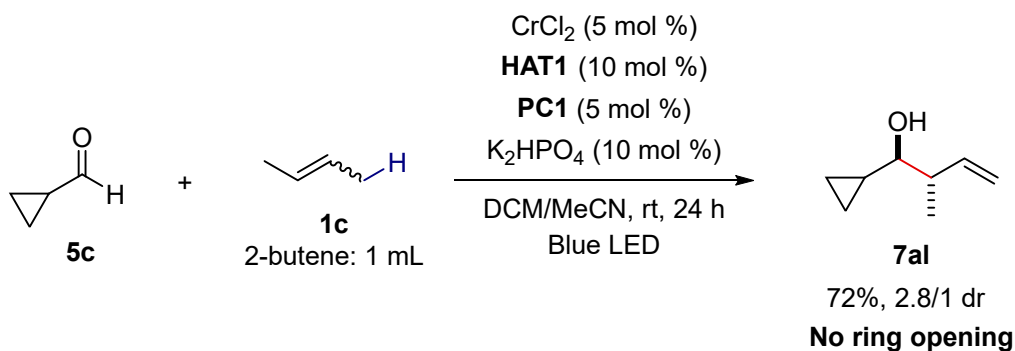
(S,R)-7k

This time: $[\alpha]_D^{20} = -16$ ($c = 0.89$, CHCl_3 , for 72% ee sample)

Reported value³⁰: $[\alpha]_D^{20} = +19.8$ ($c = 0.22$, CHCl_3 , for 90% ee sample of **(R,S)-7k**)

8. Radical Clock Experiment

To rule out the involvement of ketyl radicals, a radical clock experiment was performed using cyclopropanecarboxaldehyde as electrophile.

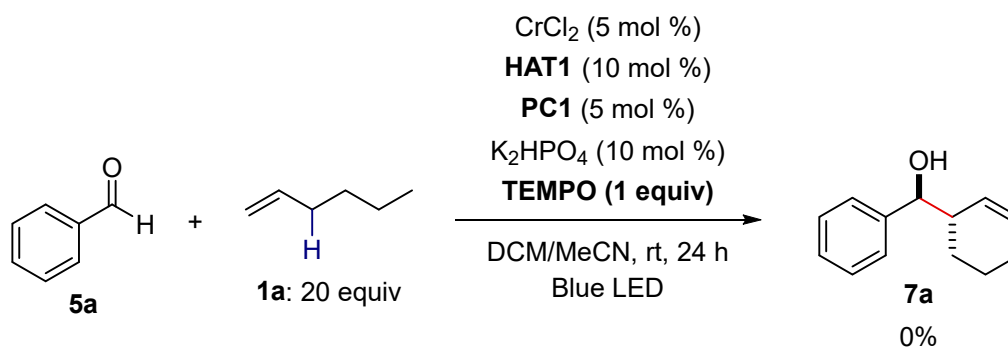


In an argon-filled glove box, CrCl_2 (1.5 mg, 0.0125 mmol, 5 mol%), **HAT1** (12.4 mg, 0.025 mmol, 10 mol%), **PC1** (6.1 mg, 0.0125 mmol, 5 mol%), and K_2HPO_4 (4.4 mg, 0.025 mmol, 10 mol%) were dissolved

in degassed CH₂Cl₂ (1.5 mL) and MeCN (1 mL) in a screw-capped tube. Then, cyclopropanecarboxaldehyde **5c** (18.8 μL, 0.25 mmol, 1 equiv) was added to the reaction mixture. The reaction tube was removed from the glove box and the reaction mixture was cooled to -78 °C. Liquid 2-butene **1c** (ca. 1 mL) cooled at -78 °C was added to the reaction mixture via syringe. The reaction mixture was warmed to room temperature and subjected to 430 nm LED irradiation by a Kessil 40W LED lamp (A160WE Tuna Blue) for 24 hours under temperature control (ca. 35–38 °C). Then, the reaction mixture was diluted by hexane (ca. 2 mL) and passed through a pad of silica gel with CH₂Cl₂ elution. After evaporation, the crude mixture was analyzed by ¹H NMR spectroscopy using 1,1,2,2-tetrachloroethane as internal standard. Desired product **7al** was formed (72%, 2.8/1 dr), and no ring opened product was observed. Therefore, we assume that ketyl radicals are not formed in the reaction.

9. TEMPO Trapping Experiment

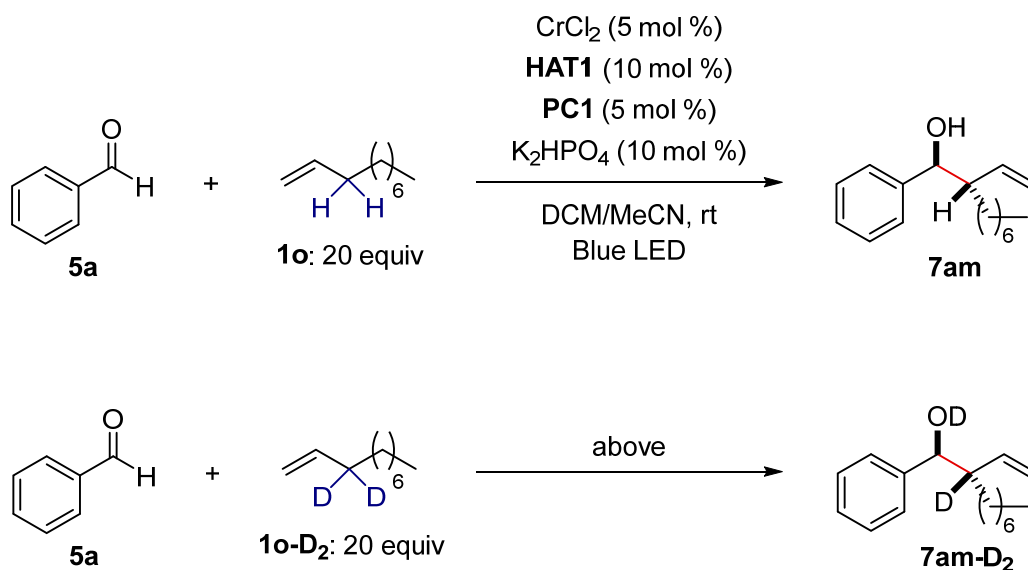
To confirm the generation of radical intermediate in the reaction, a radical trapping experiment by the addition of TEMPO (2,2,6,6-tetramethyl-1-piperidinyloxy, free radical) was conducted.



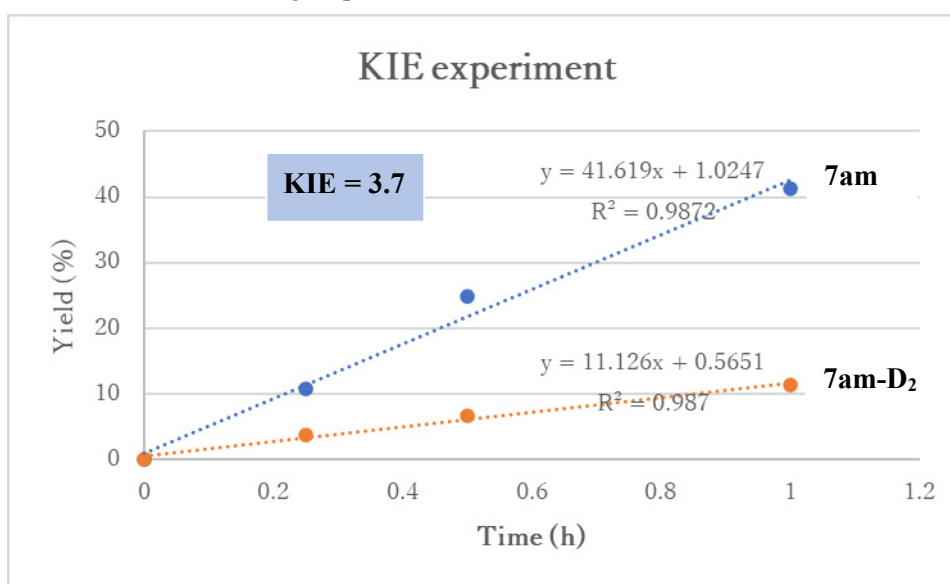
In an argon-filled glove box, CrCl₂ (1.5 mg, 0.0125 mmol, 5 mol%), **HAT1** (12.4 mg, 0.025 mmol, 10 mol%), **PC1** (6.1 mg, 0.0125 mmol, 5 mol%), and K₂HPO₄ (4.4 mg, 0.025 mmol, 10 mol%) were dissolved in degassed CH₂Cl₂ (1.5 mL) and MeCN (1 mL) in a screw-capped vial. Then, 1-hexene **1a** (628 μL, 5.0 mmol, 20 equiv) and benzaldehyde **5a** (25.5 μL, 0.25 mmol, 1 equiv) were added to the reaction mixture. Then, TEMPO (39.1 mg, 0.25 mmol, 1 equiv) were added to the reaction mixture. The vial was subjected to 430 nm LED irradiation by a Valore VBP-L24-C2 with 38W LED lamp (VBL-SE150-BBB (430)) for 24 hours under temperature control (ca. 27–29 °C). Then, the reaction mixture was diluted by hexane (ca. 2 mL) and passed through a pad of silica gel with CH₂Cl₂ elution. After evaporation, the crude mixture was analyzed by NMR spectroscopy using 1,1,2,2-tetrachloroethane as internal standard. No desired product **7a** was formed. This result is in consistent with our working hypothesis.

10. Kinetic Experiment

To gain insight into the rate limiting step, kinetic experiments using deuterated decene were performed.



In an argon-filled glove box, CrCl_2 (0.75 mg, 0.00625 mmol, 5 mol%), **HAT1** (6.2 mg, 0.0125 mmol, 10 mol%), **PC1** (3.1 mg, 0.00625 mmol, 5 mol%), and K_2HPO_4 (2.2 mg, 0.0125 mmol, 10 mol%) were dissolved in degassed CH_2Cl_2 (0.75 mL) and MeCN (0.5 mL) in a screw-capped vial. Then, 1-decene **1o** or 1-decene- d_2 **1o-D₂** (470 μL , 2.5 mmol, 20 equiv) and benzaldehyde **5a** (25.5 μL , 0.25 mmol, 1 equiv) were added to the reaction mixture. *n*-Hexadecane (3.7 μL , 0.0125 mmol, 10 mol%) was added to the mixture as an internal standard. The vial was subjected to 430 nm LED irradiation by a Valore VBP-L24-C2 with 38W LED lamp (VBL-SE150-BBB (430)) under temperature control (ca. 27–29 °C). The reaction progress was monitored by GC analysis by comparison of integrated area of target materials (**7am** or **7am-D₂**) and *n*-hexadecane. A significant kinetic isotope effect (KIE = 3.7) was observed, suggesting that the HAT process of allylic C–H bond was a rate-limiting step.



11. References

- 1) Ho, S.; Sackett, D.; Leighton, J. A. "Methyl Extension" Strategy for Polyketide Natural Product Linker

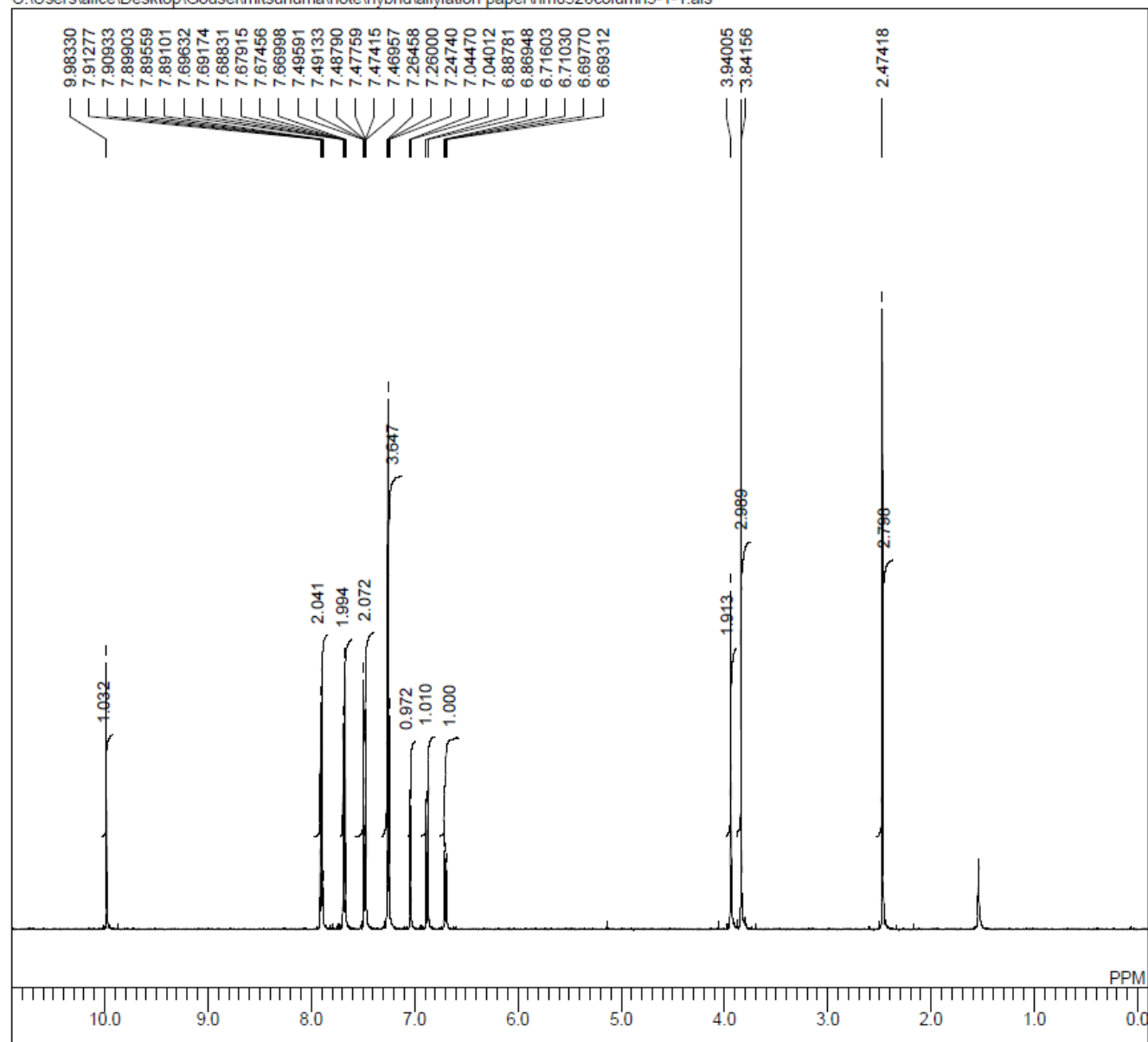
- Site Validation and Its Application to Dictyostatin. *J. Am. Chem. Soc.* **2015**, *137*, 14047-14050.
- 2) Fuse, H.; Mitsunuma, H.; Kanai, M. Catalytic Acceptorless Dehydrogenation of Aliphatic Alcohols. *J. Am. Chem. Soc.* **2020**, *142*, 4493-4499.
 - 3) Li, C.; Wang, S.-M.; Qin, H.-L. A Rh-Catalyzed Air and Moisture Tolerable Aldehyde (Ketone)-Directed Fluorosulfonylvinylation of Aryl C(sp²)-H Bonds. *Org. Lett.* **2018**, *20*, 4699-4703.
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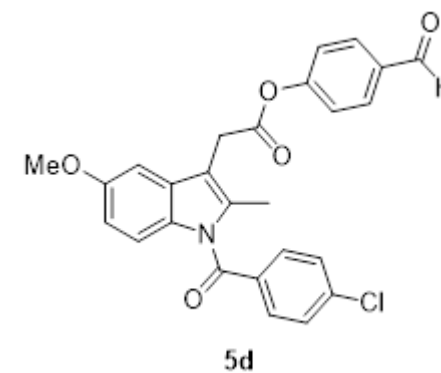
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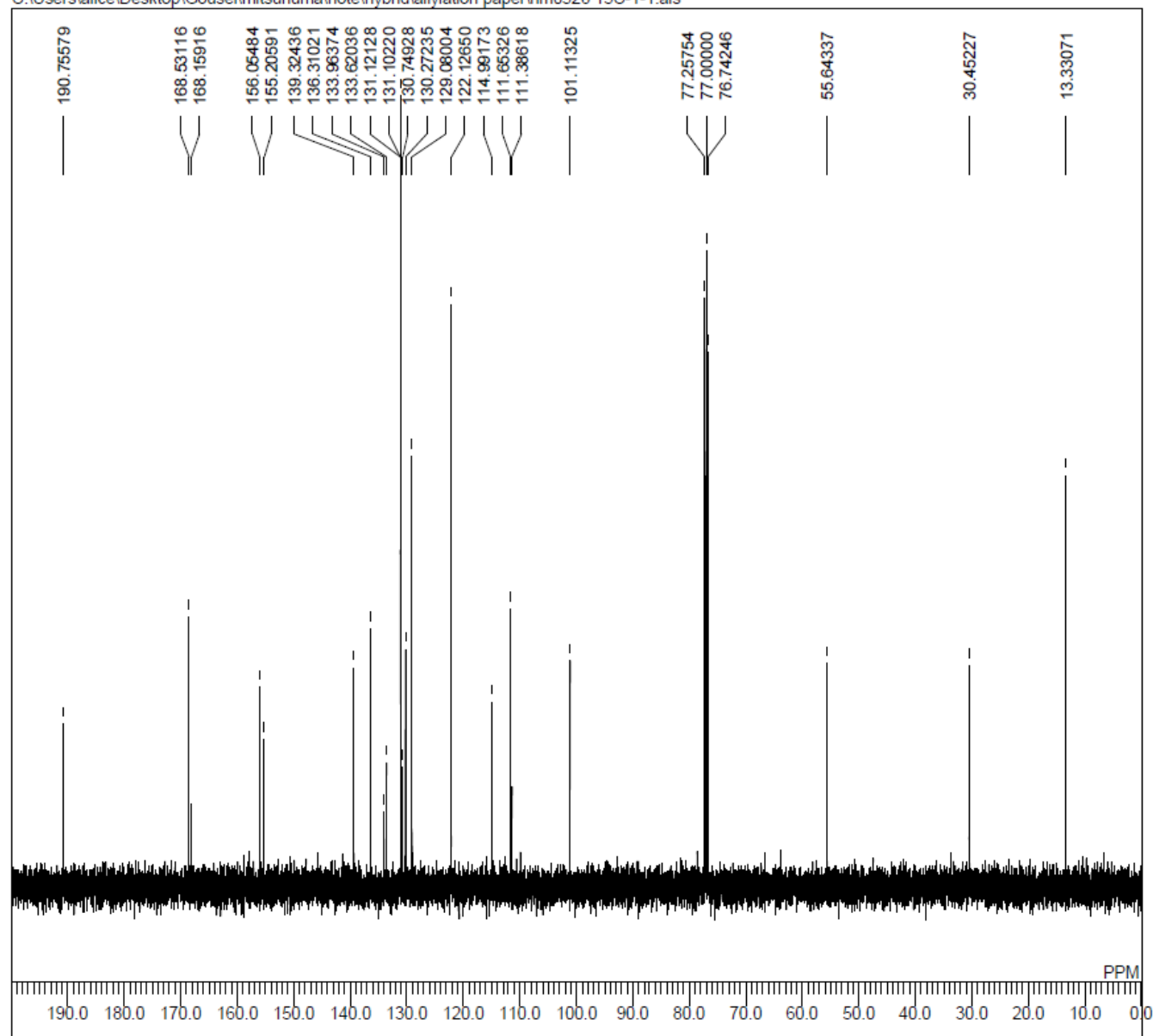
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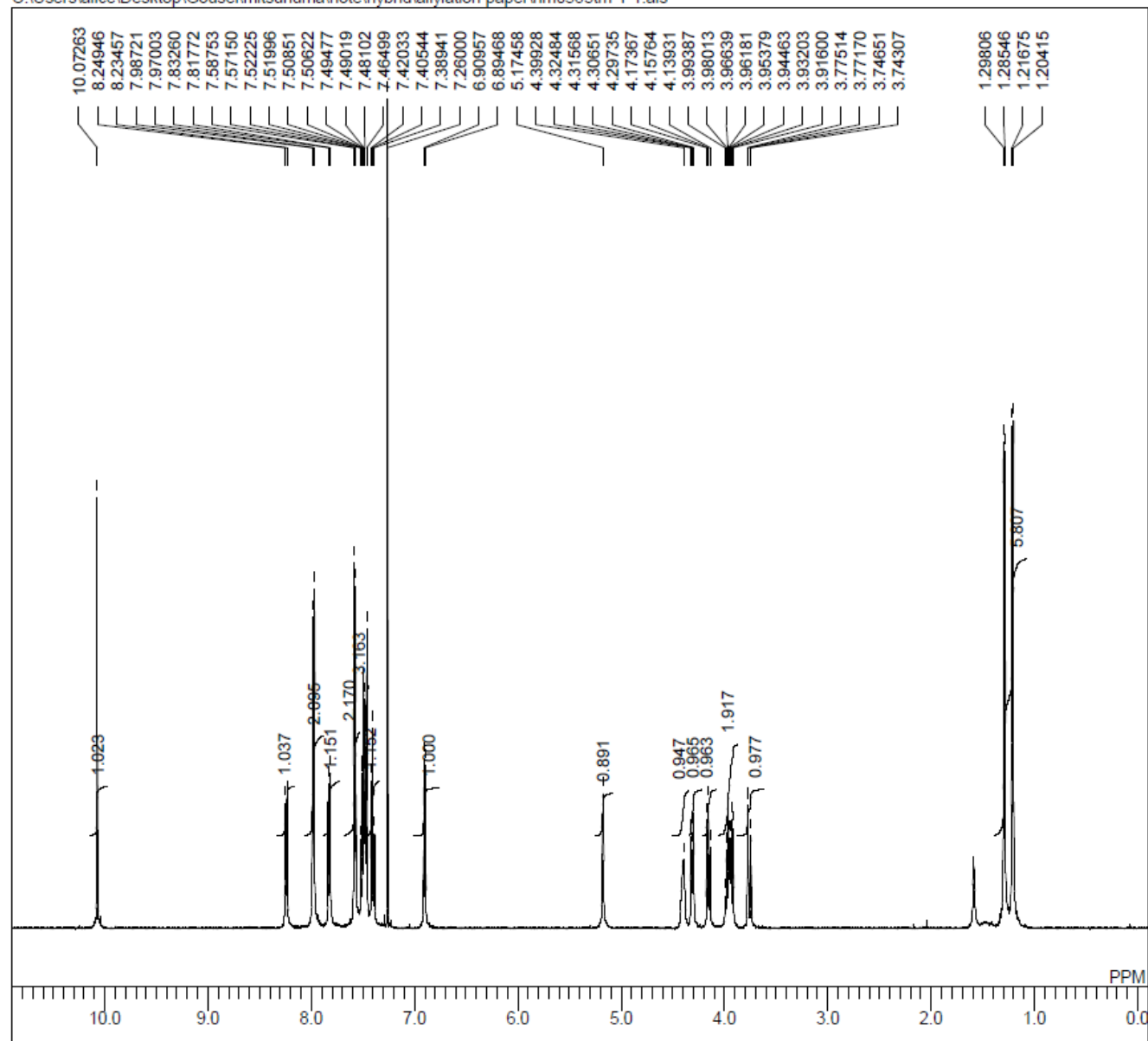


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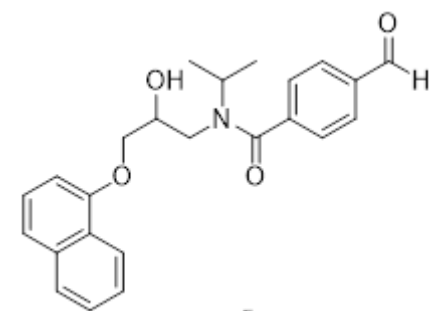


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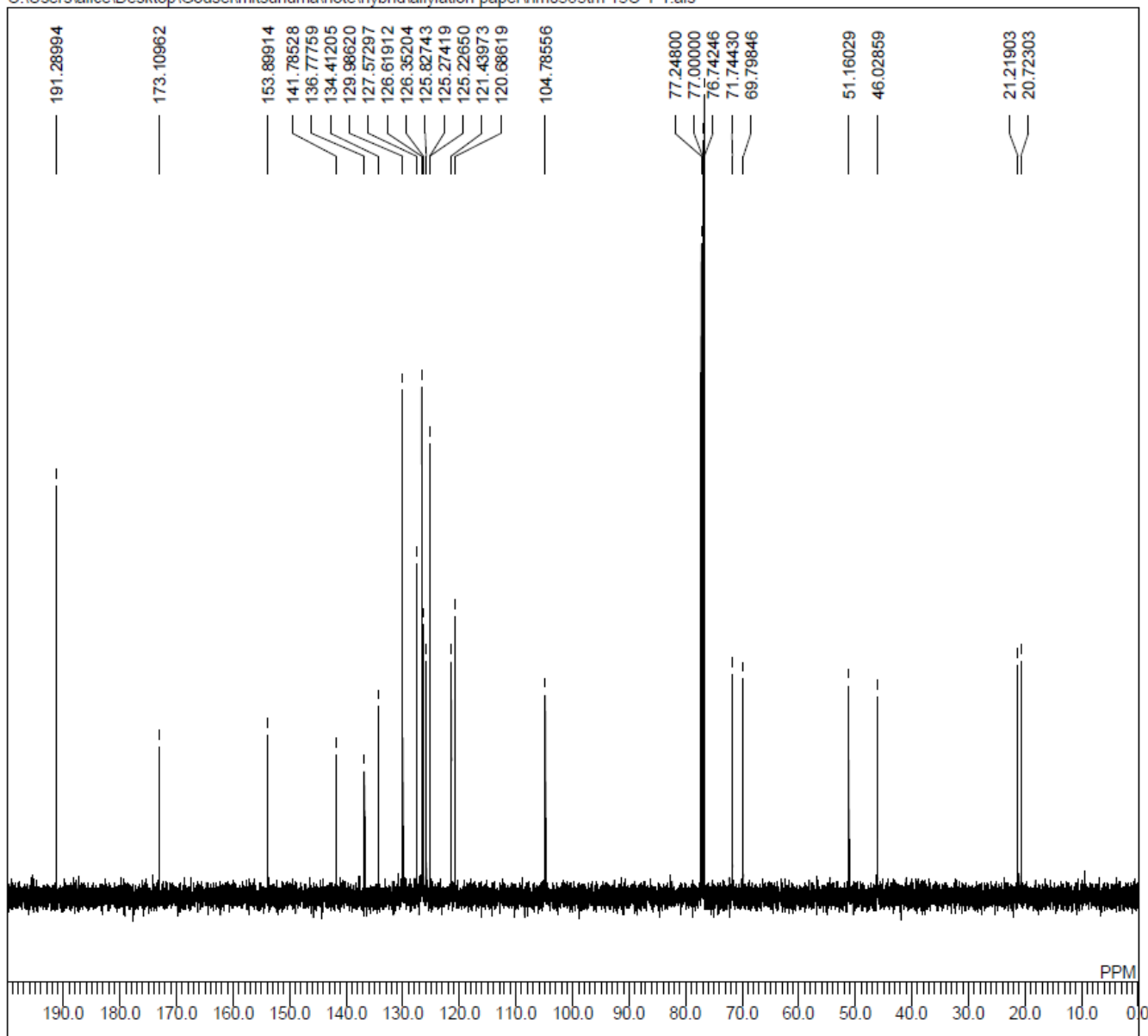


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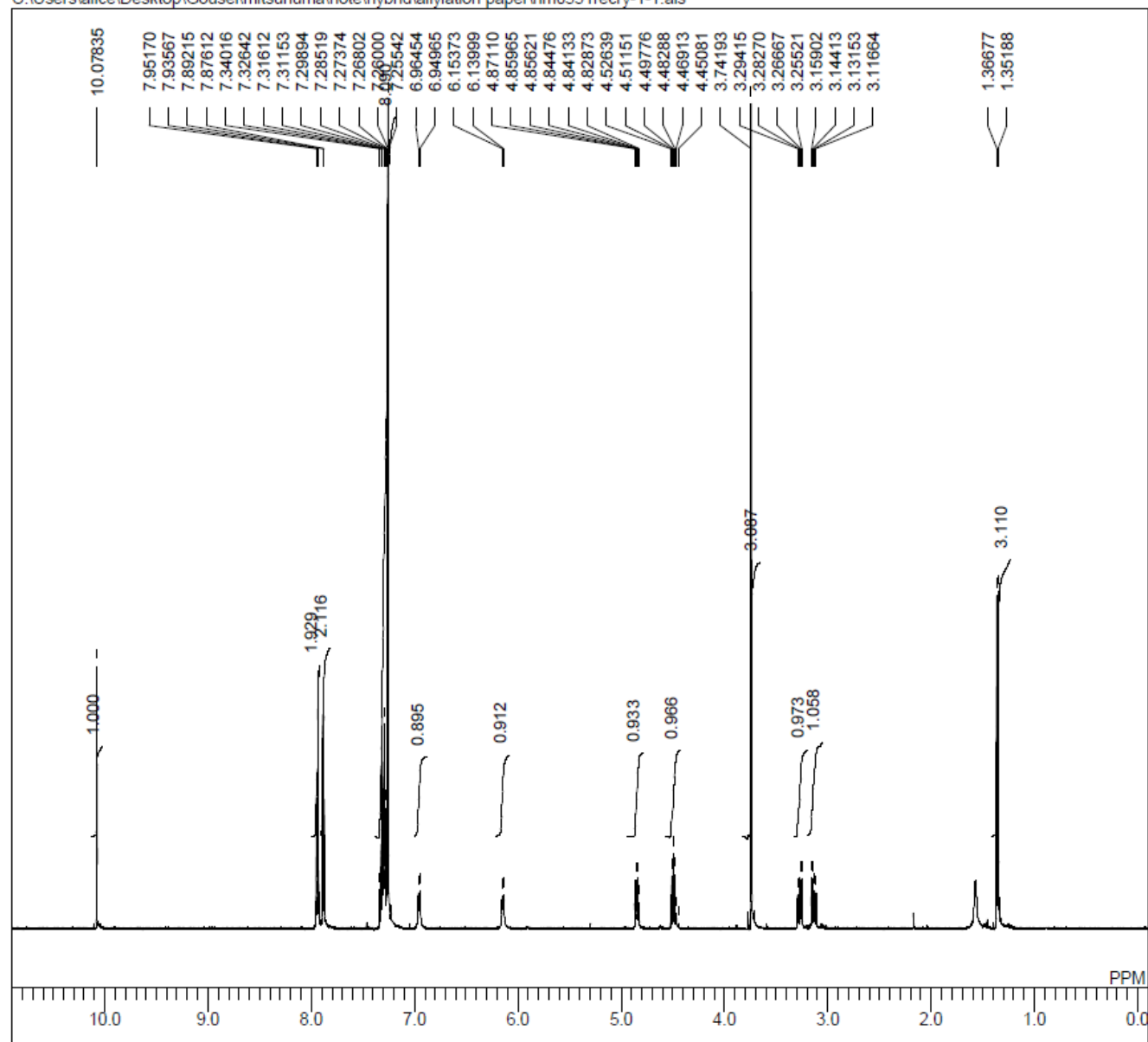
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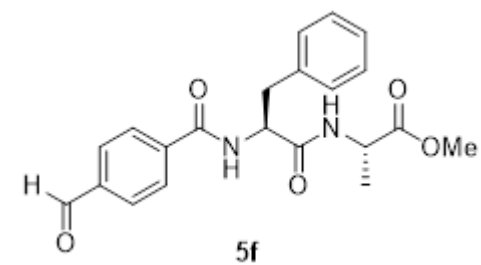


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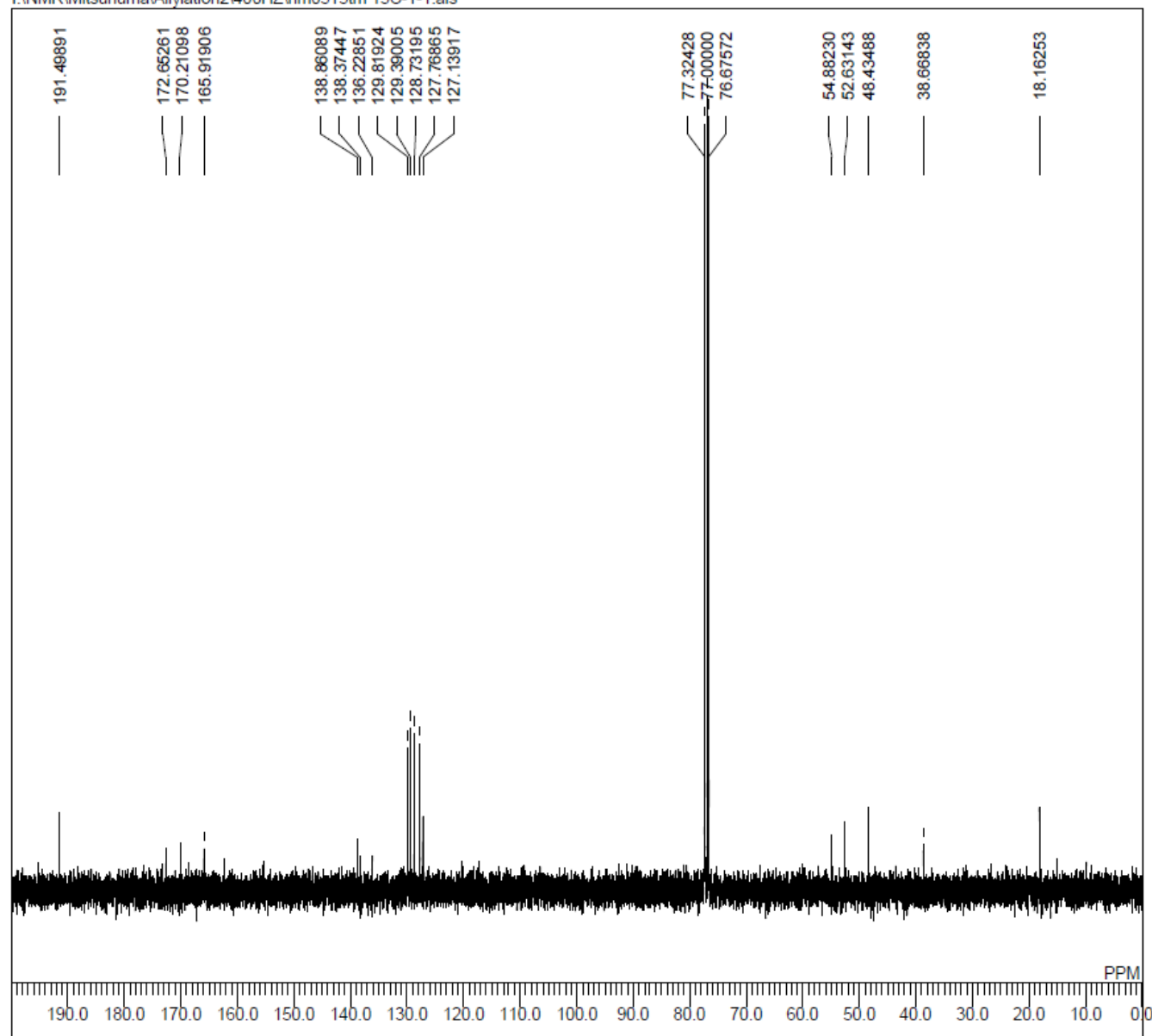
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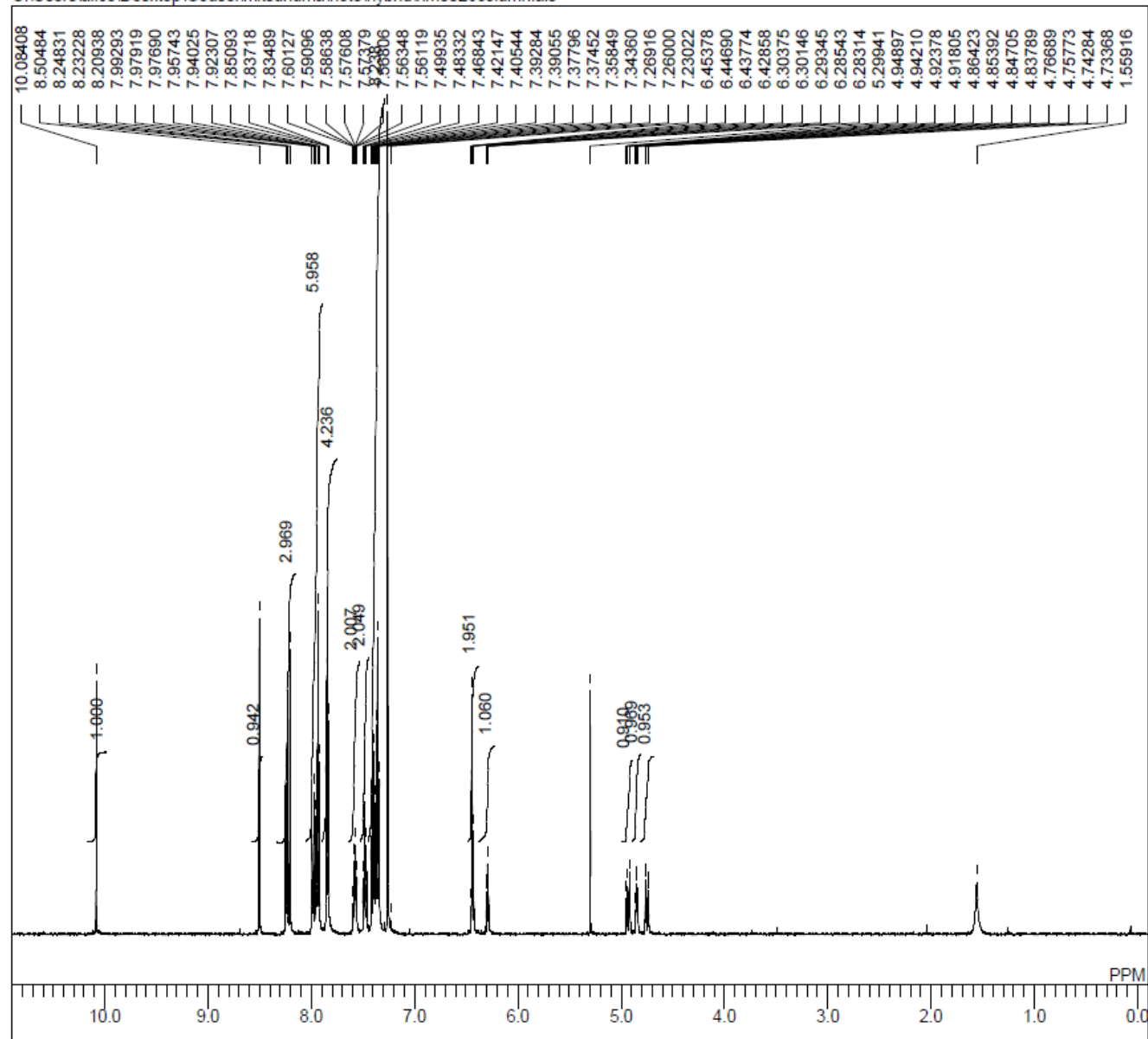


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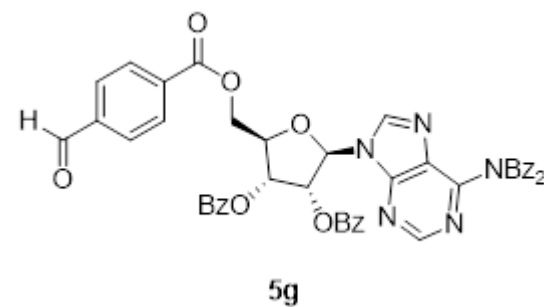


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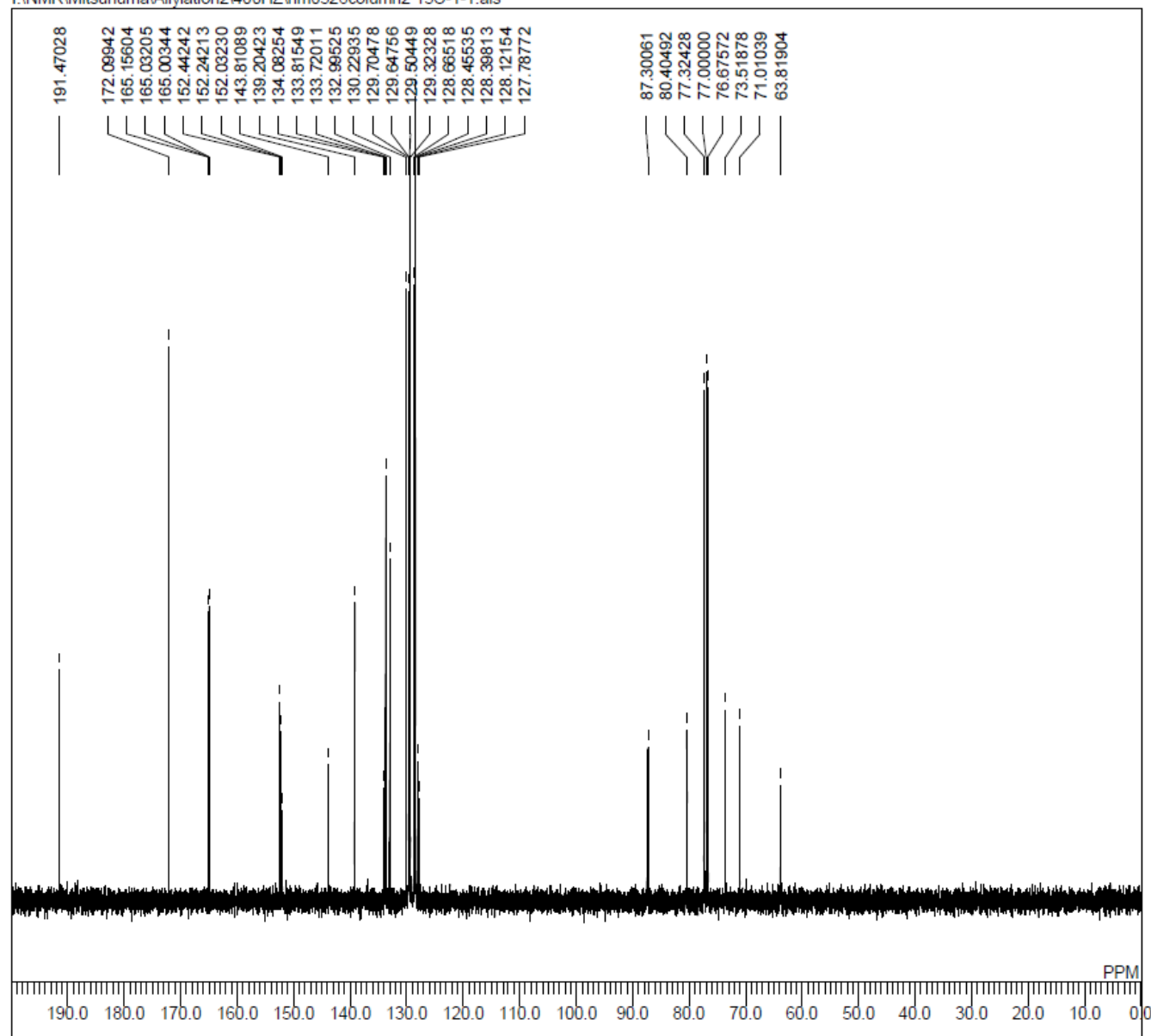
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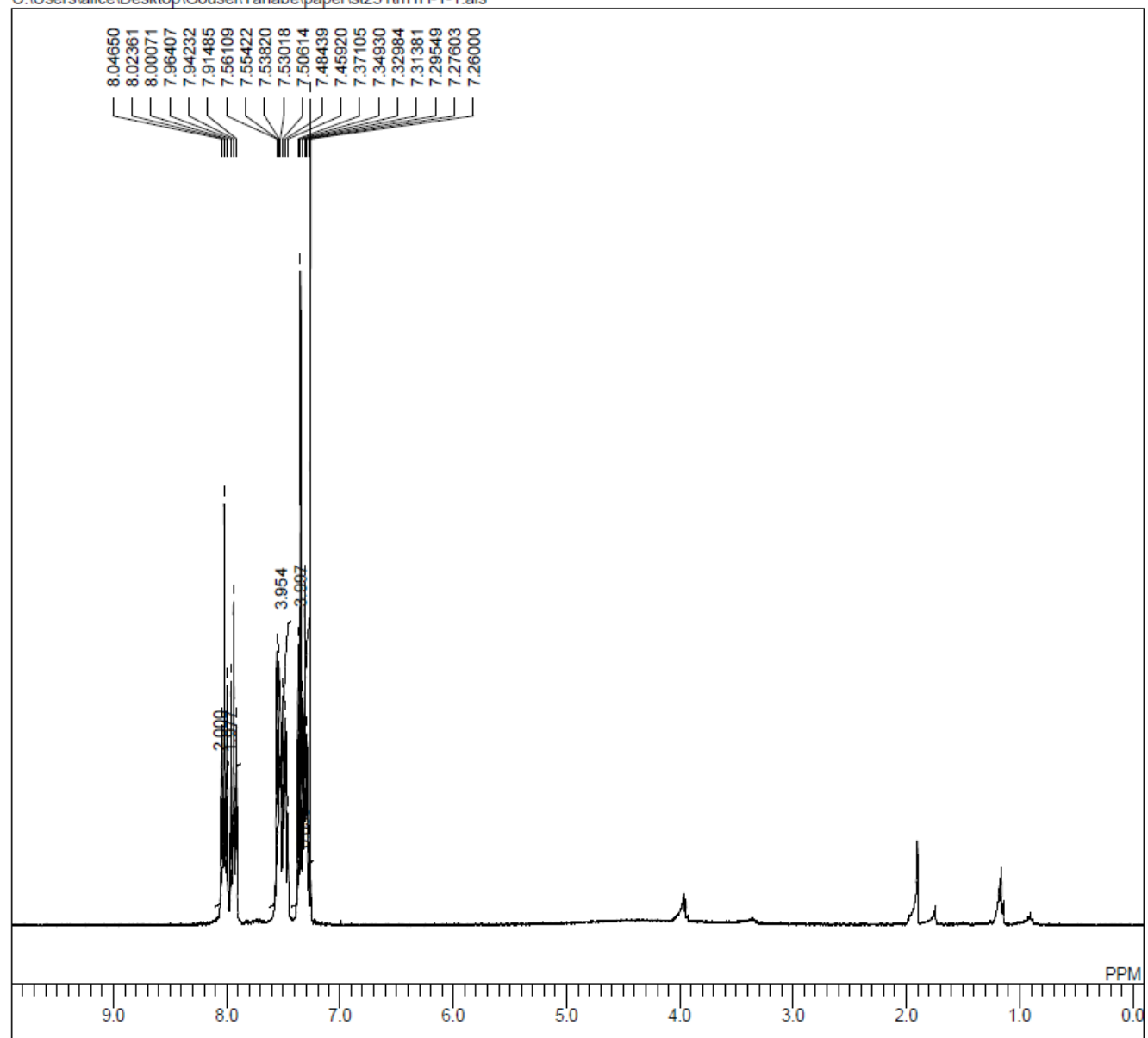


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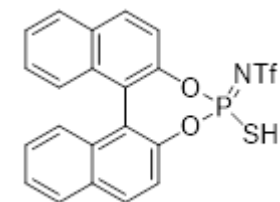


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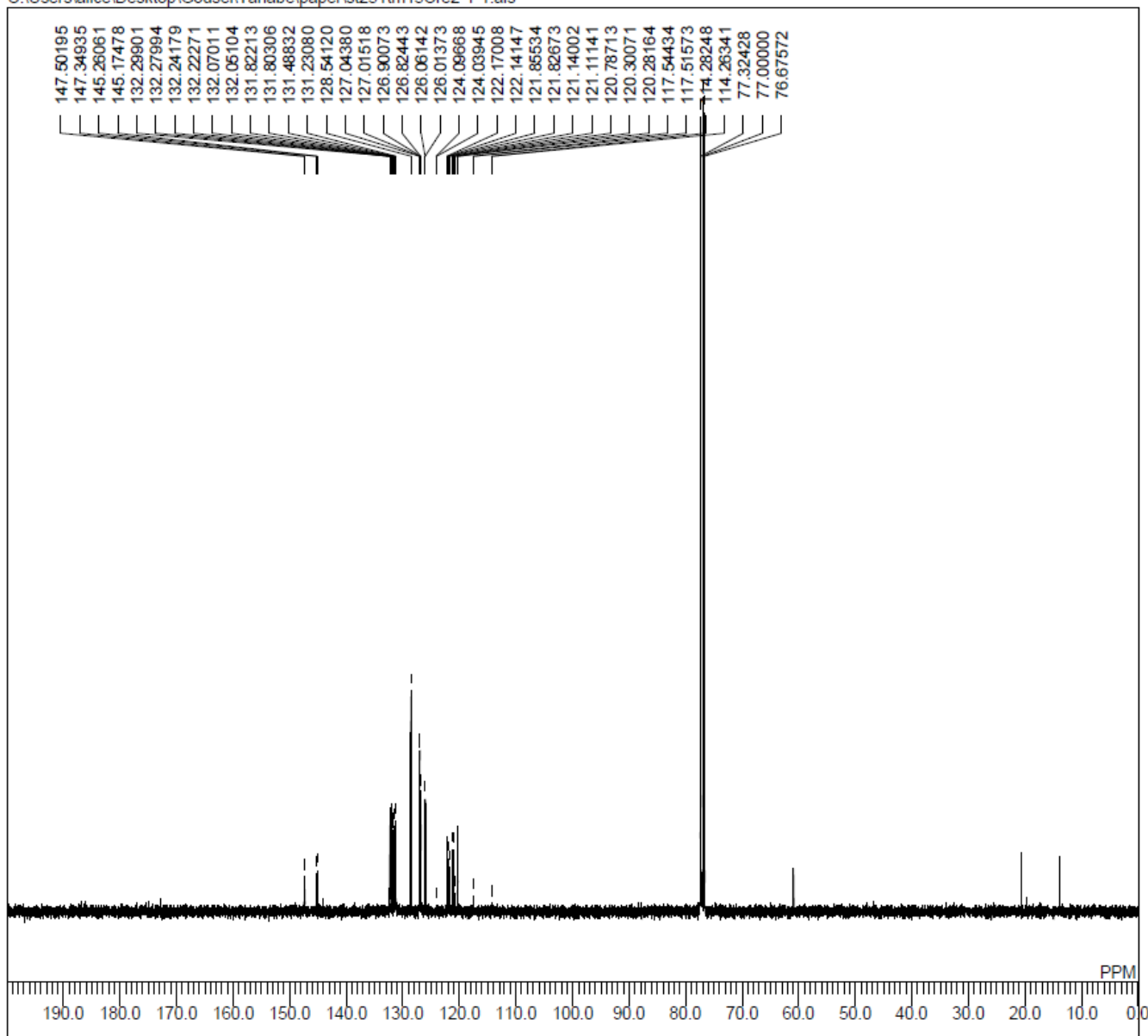
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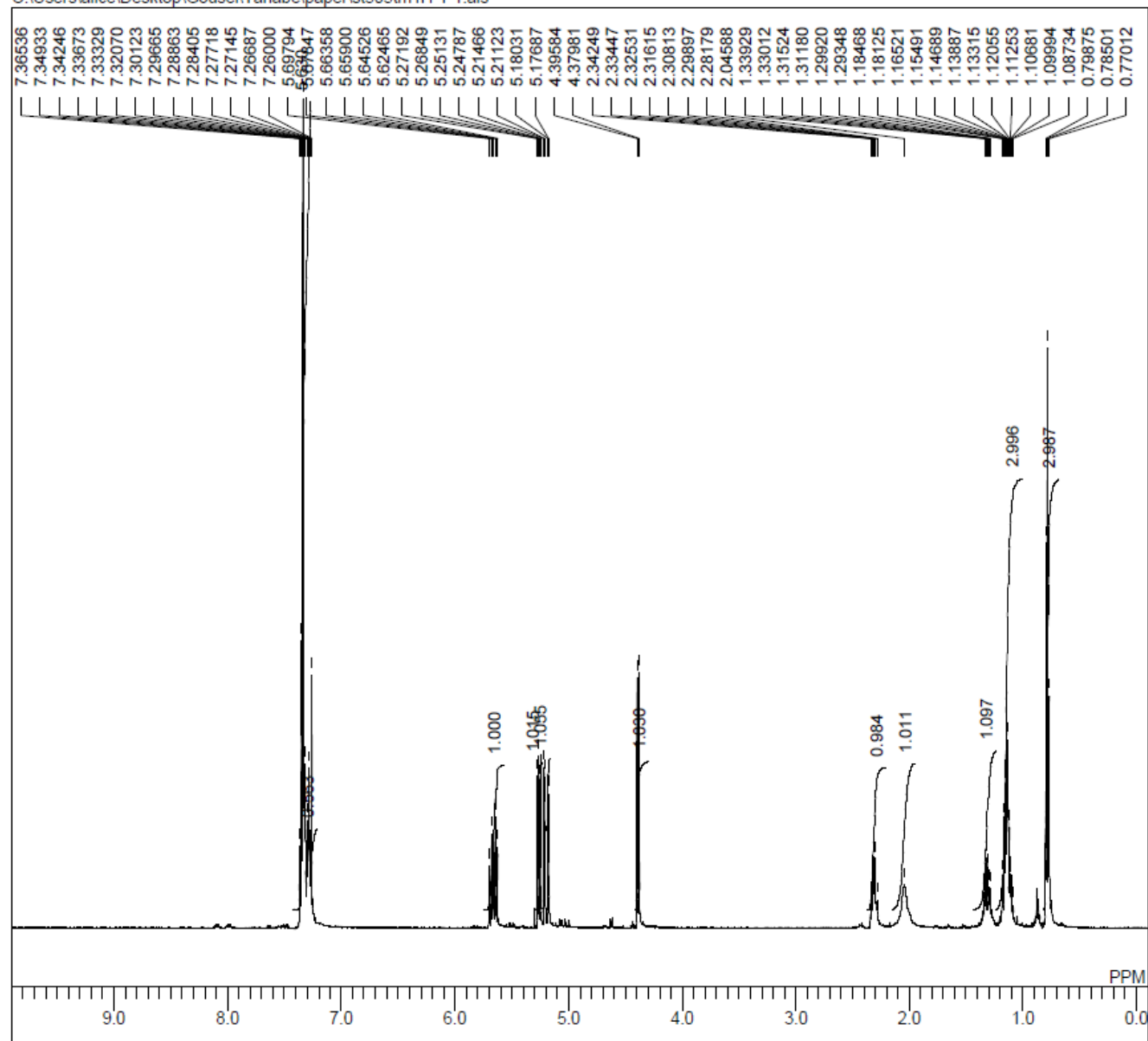


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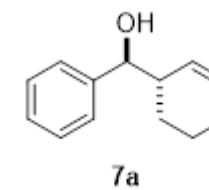


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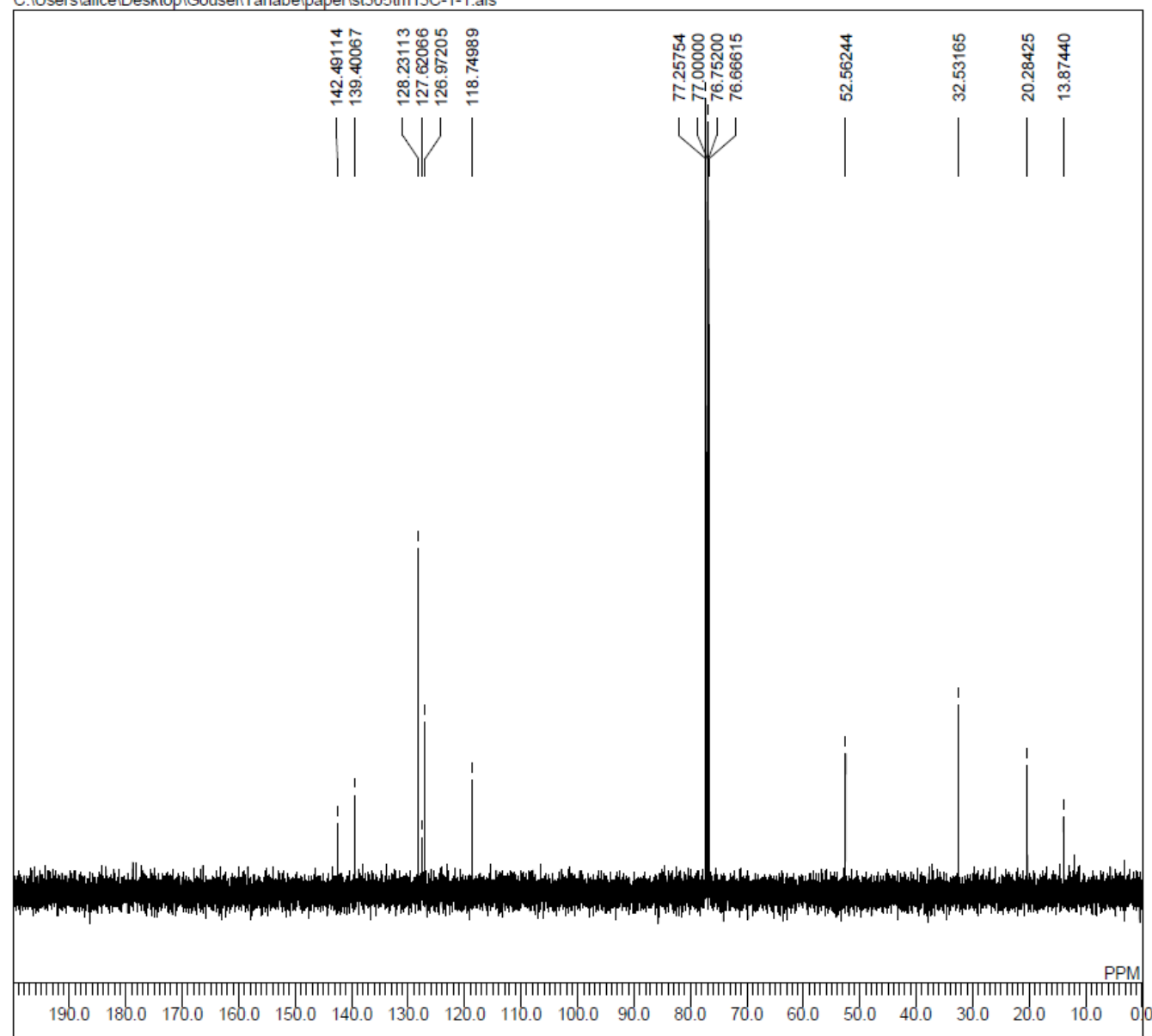
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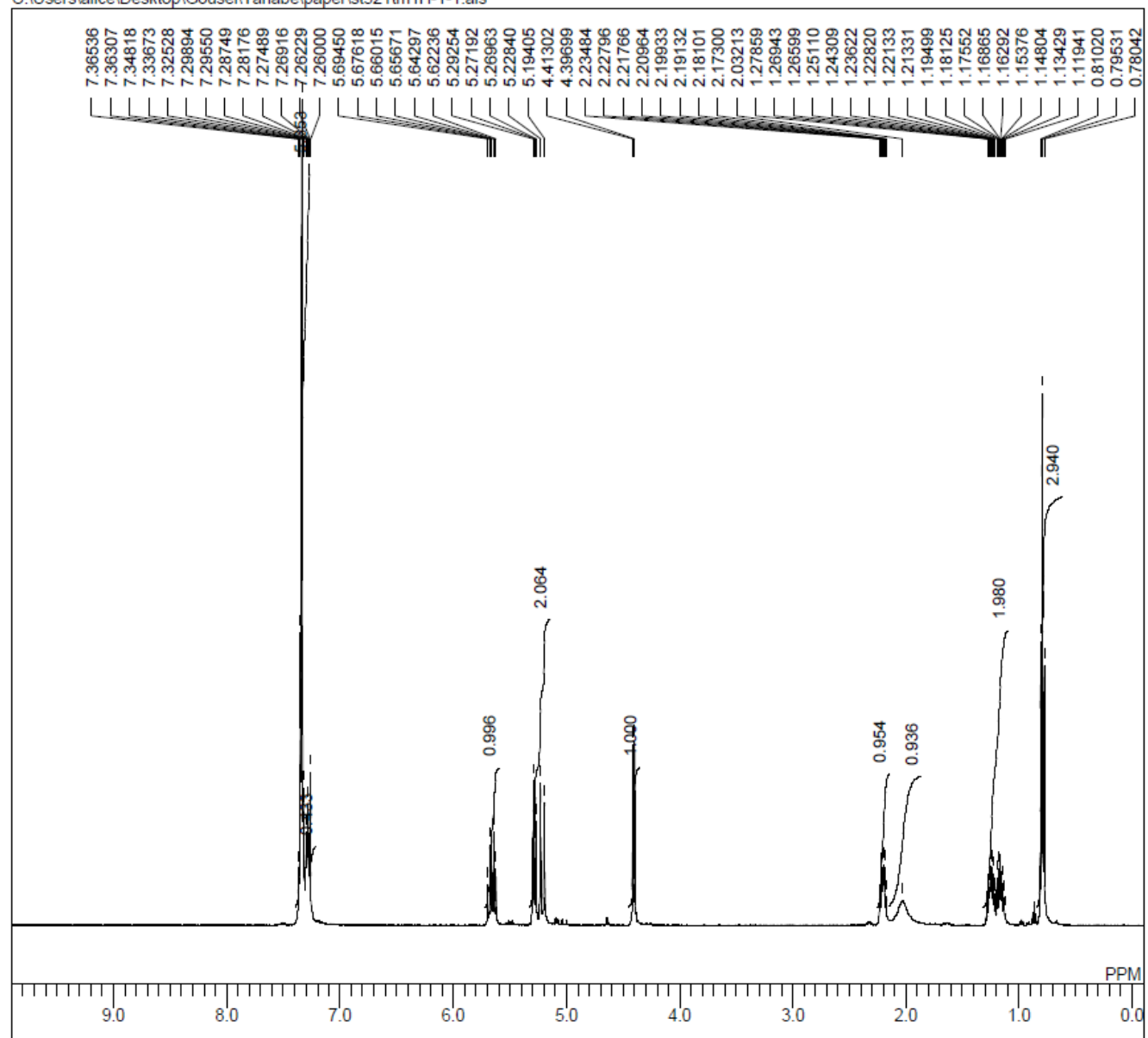


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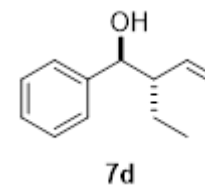


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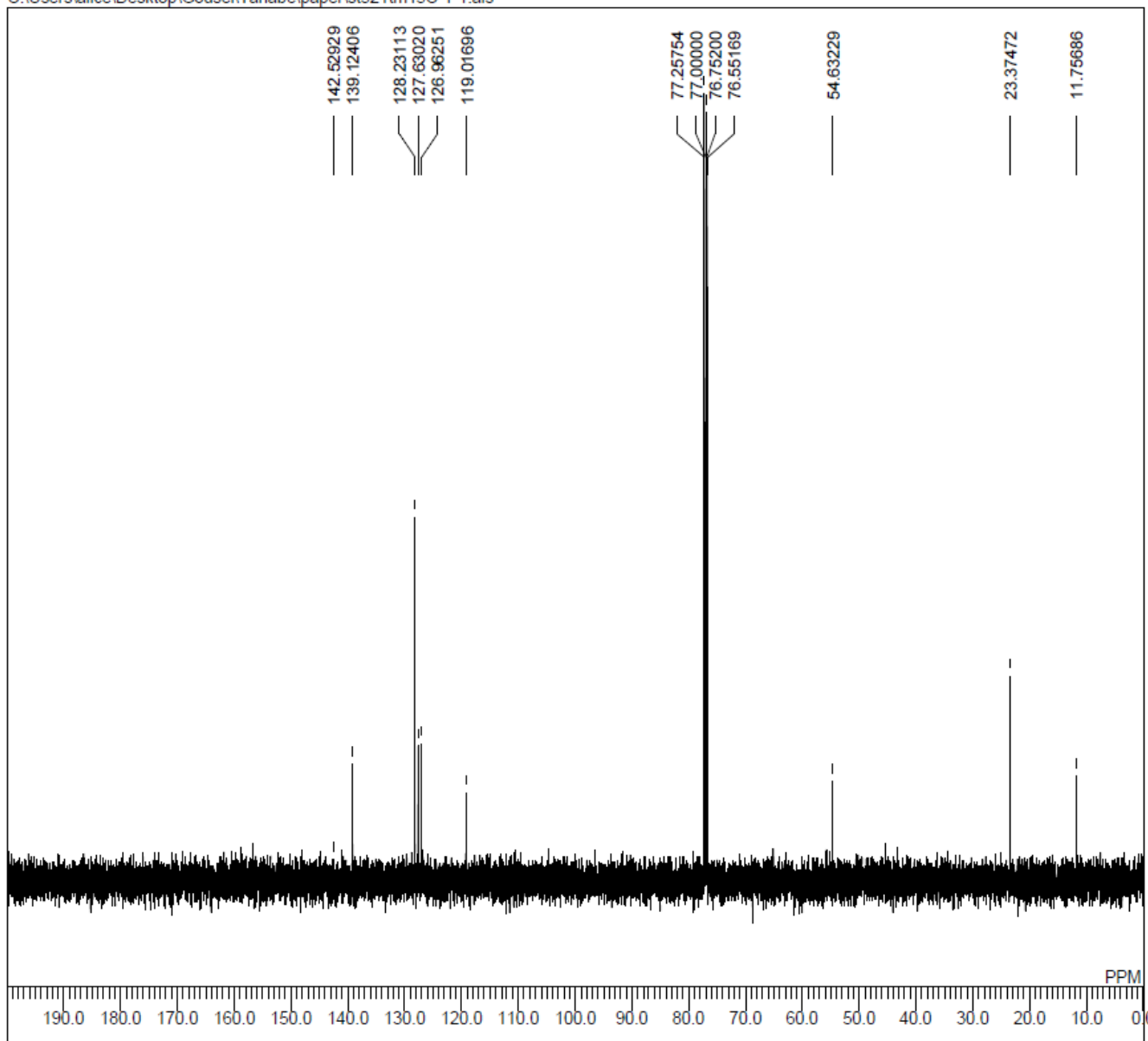
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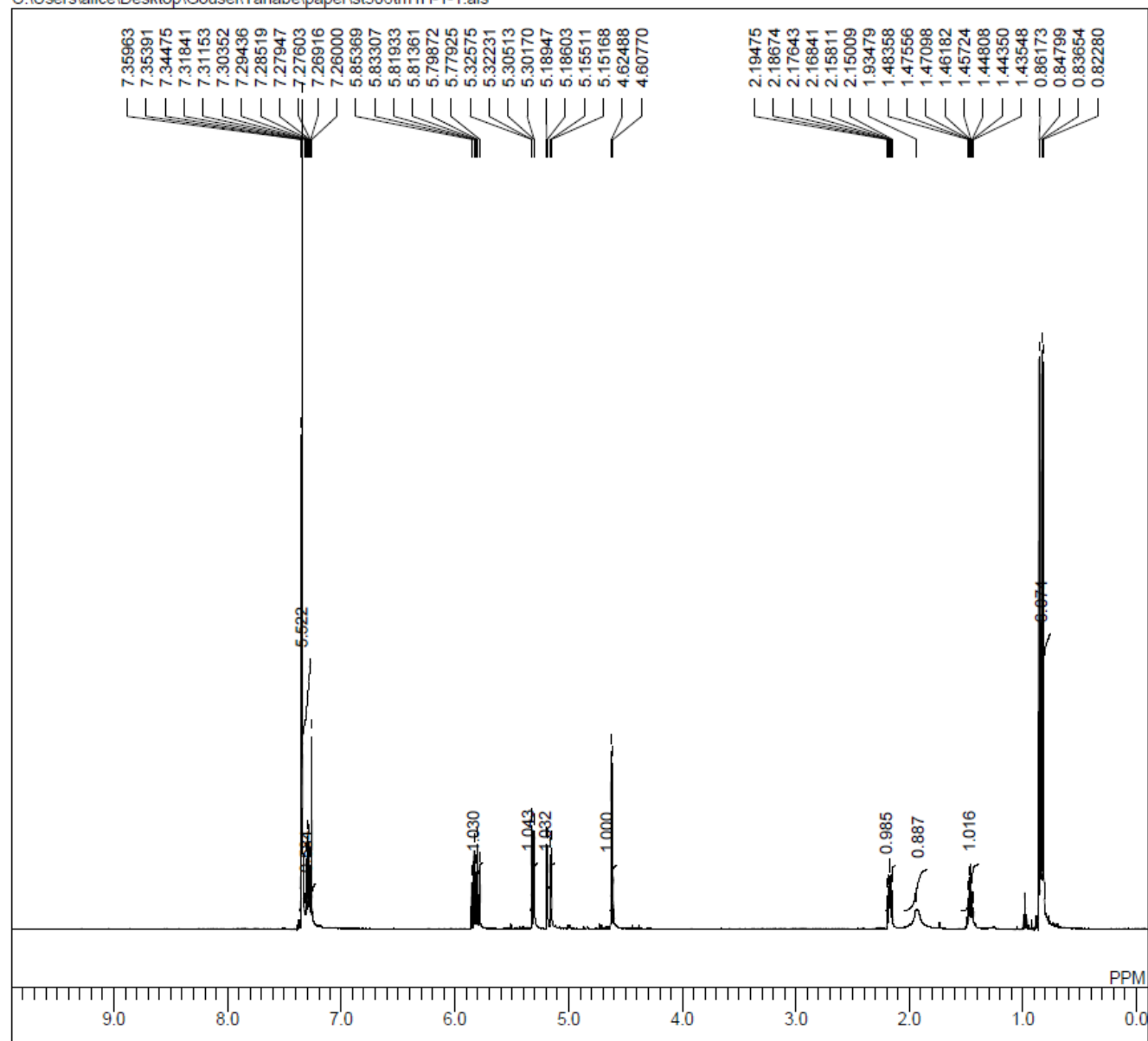


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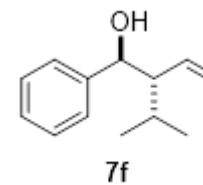


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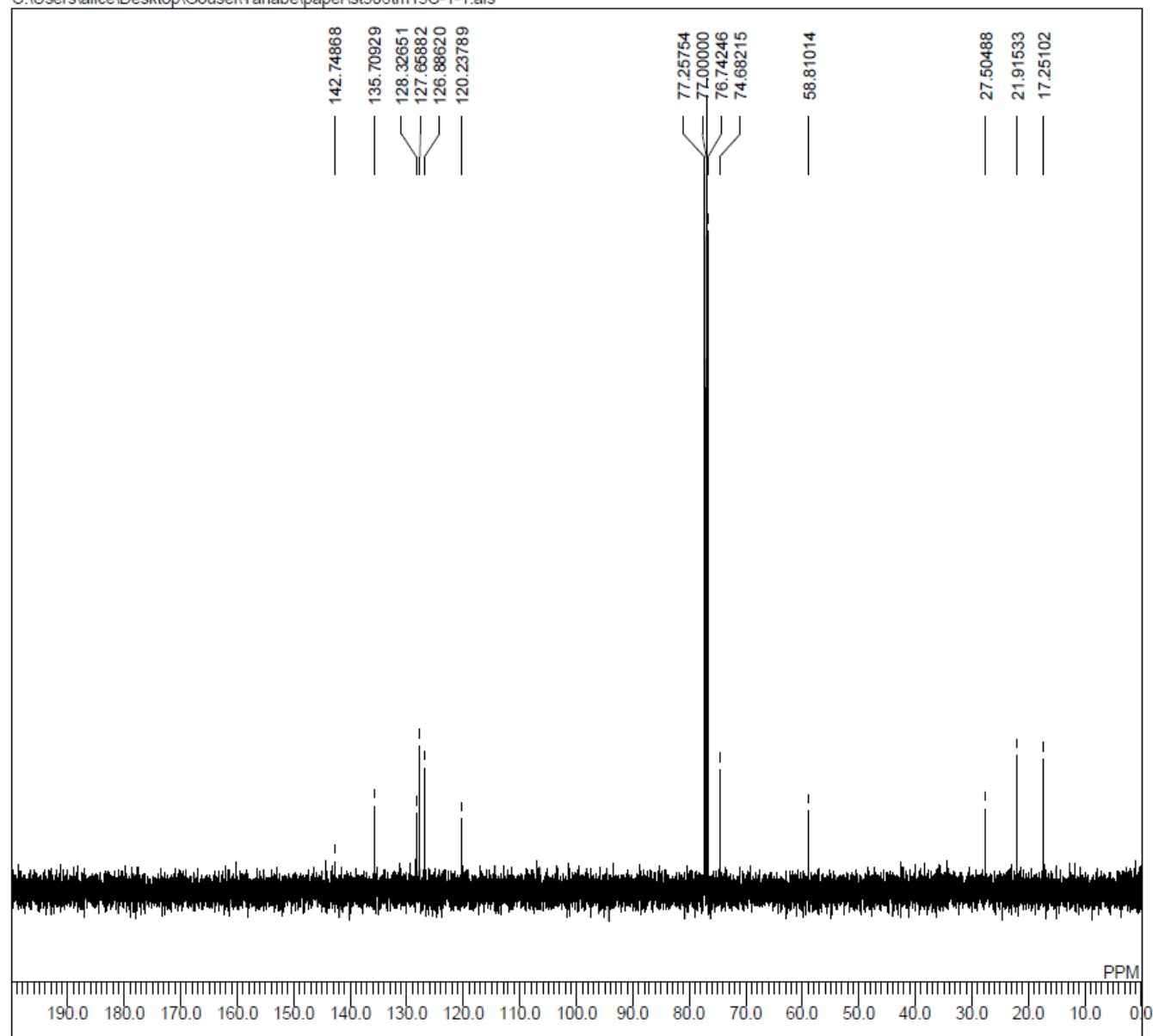
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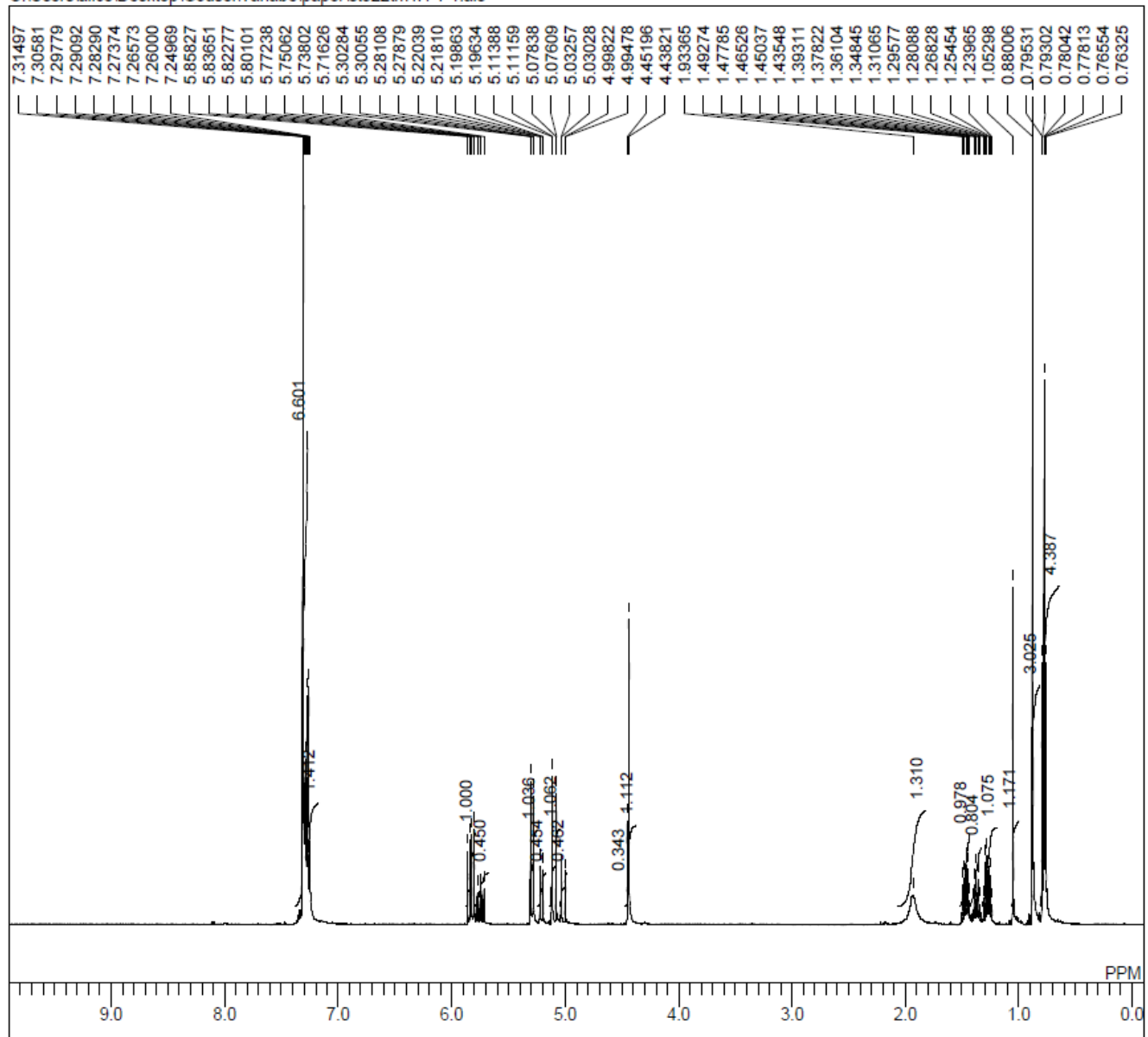


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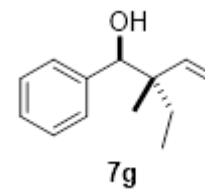


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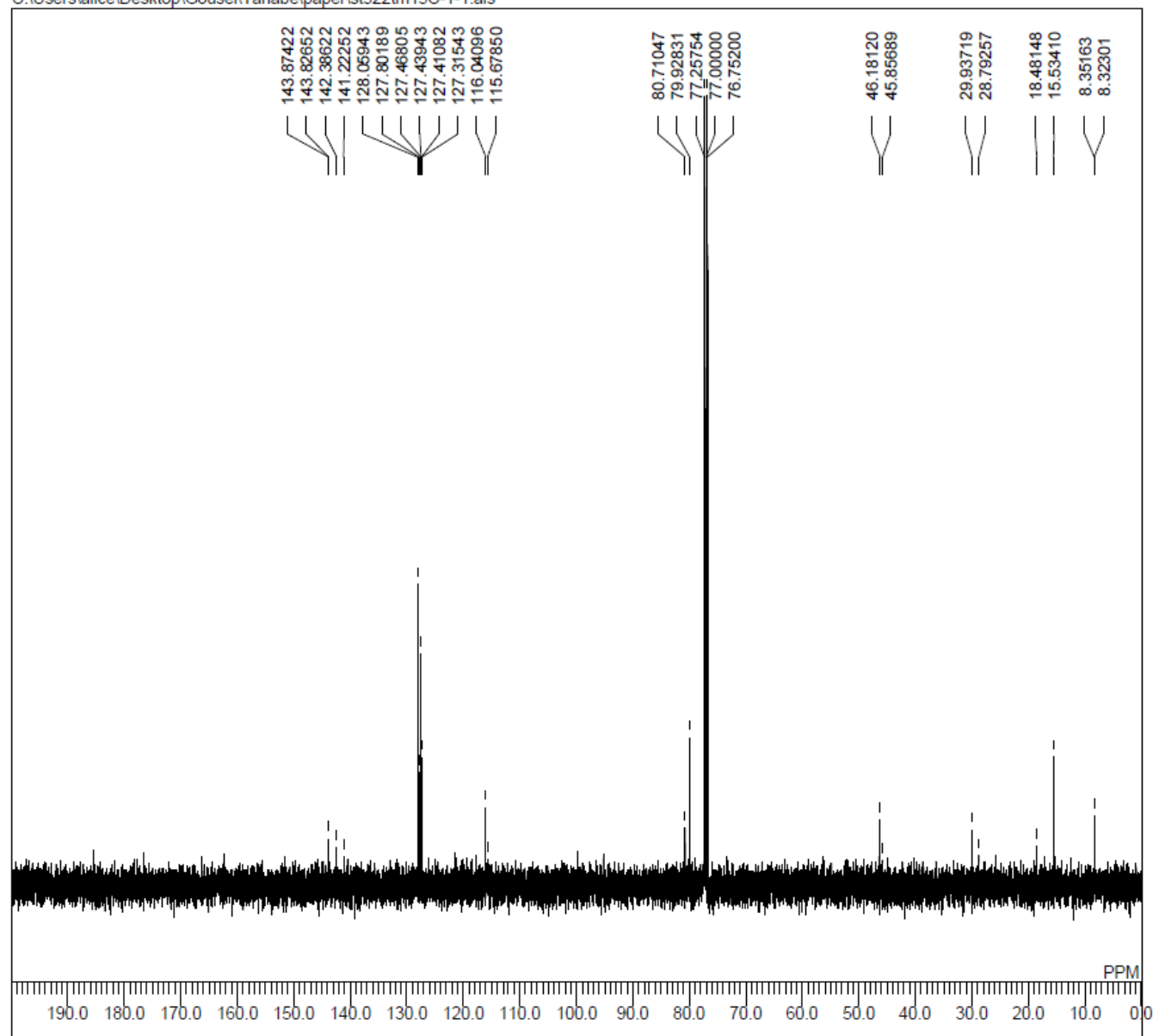
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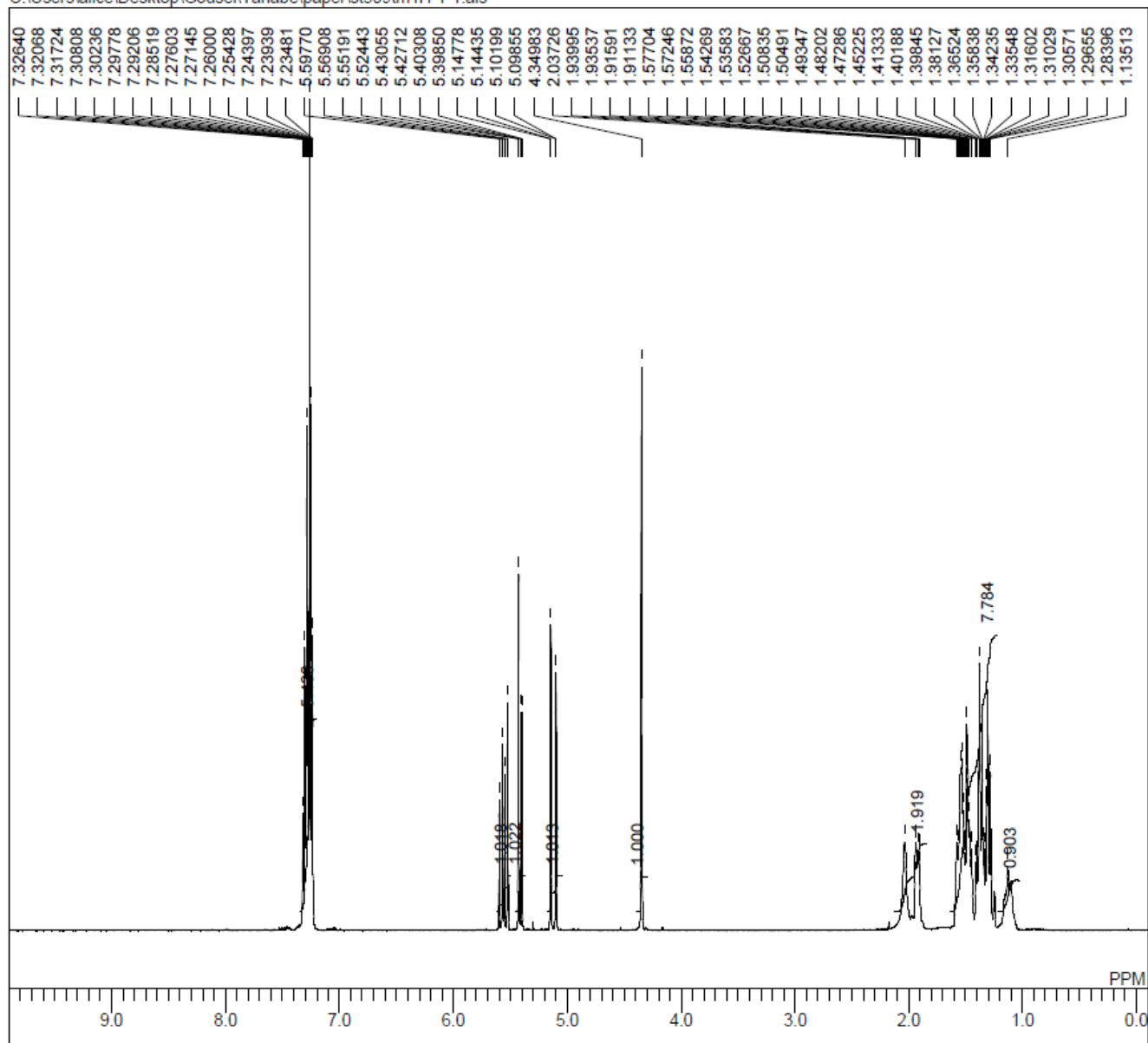


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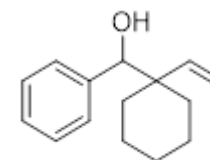


DFILE st322tm13C-1-1.als
COMNT
DATIM 2020-03-19 15:04:17
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 41
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

C:\Users\alice\Desktop\Gousei\Tanabe\paper\st309tm1H-1-1.als

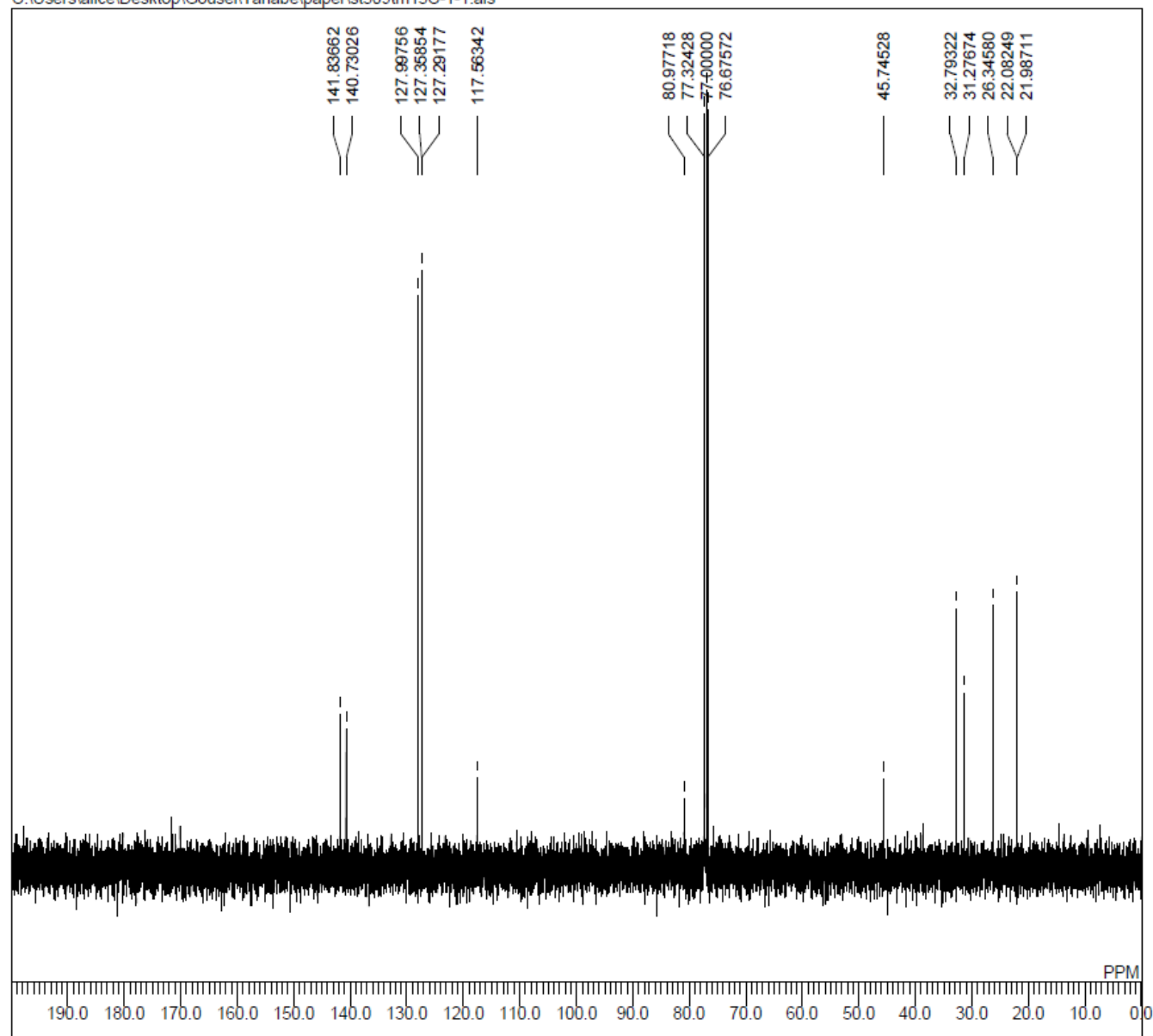


DFILE st309tm1H-1-1.als
COMNT
DATIM 03-03-2020 19:34:02
OBNUC 1H
EXMOD proton.jxp
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 13107
FREQU 5878.90 Hz
SCANS 8
ACQTM 2.2295 sec
PD 6.0000 sec
PW1 5.17 usec
IRNUC 1H
CTEMP 20.3 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 36



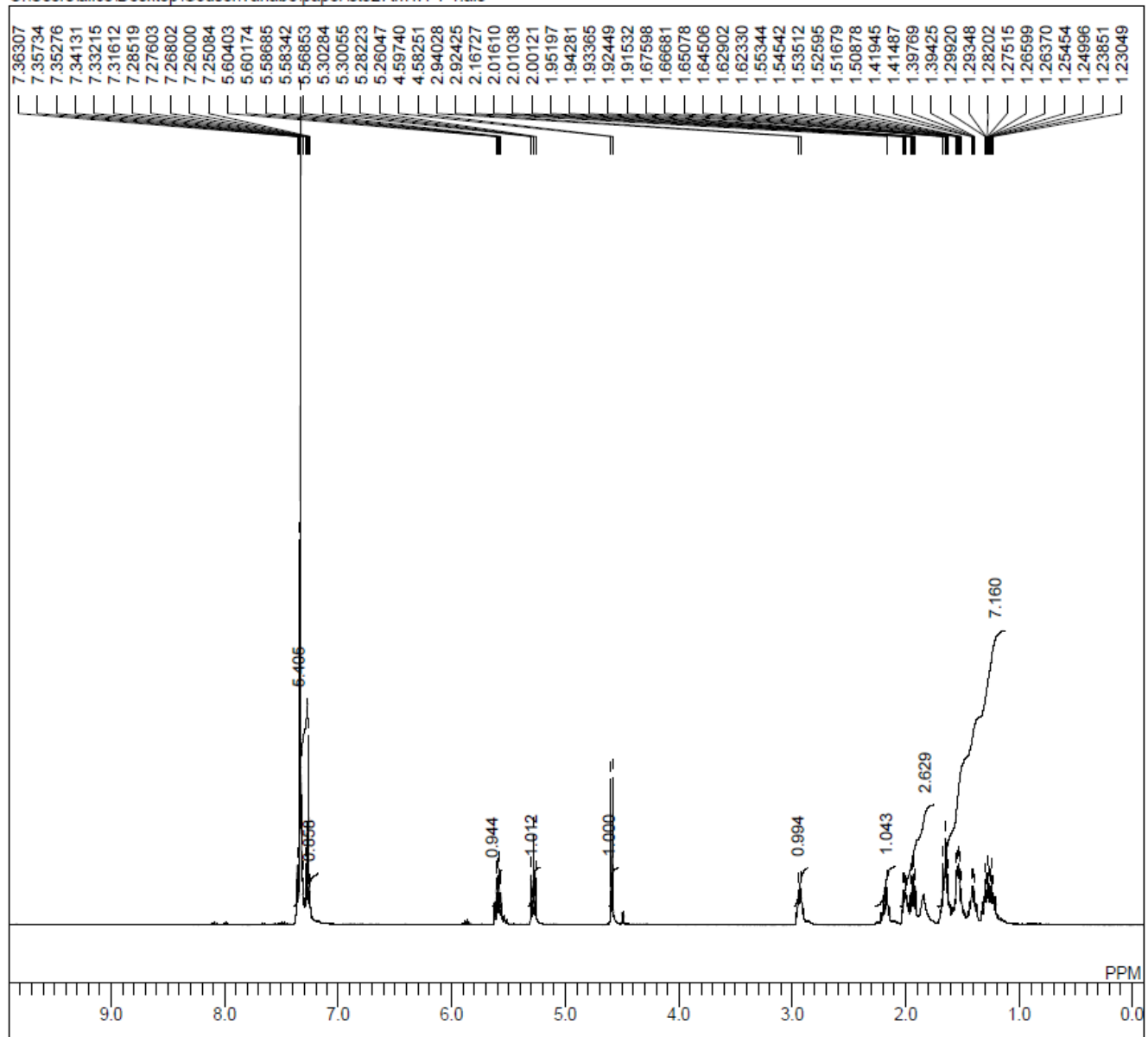
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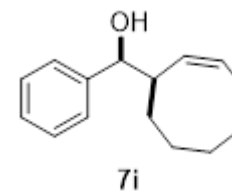


DFILE st309tm13C-1-1.als
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EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 40
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

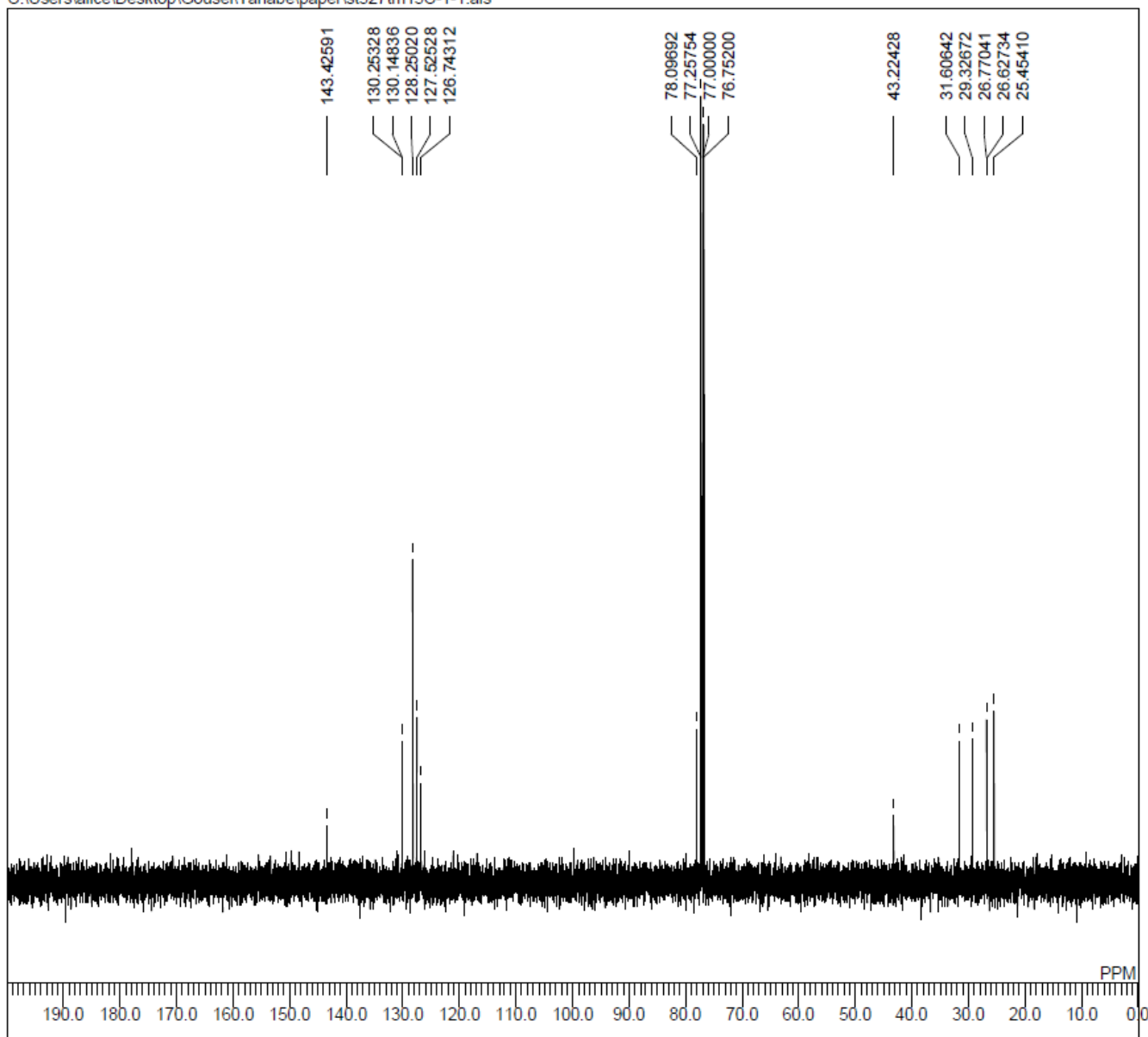
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DFILE st327m1H-1-1.als
COMNT
DATIM 2020-03-24 16:50:14
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 40

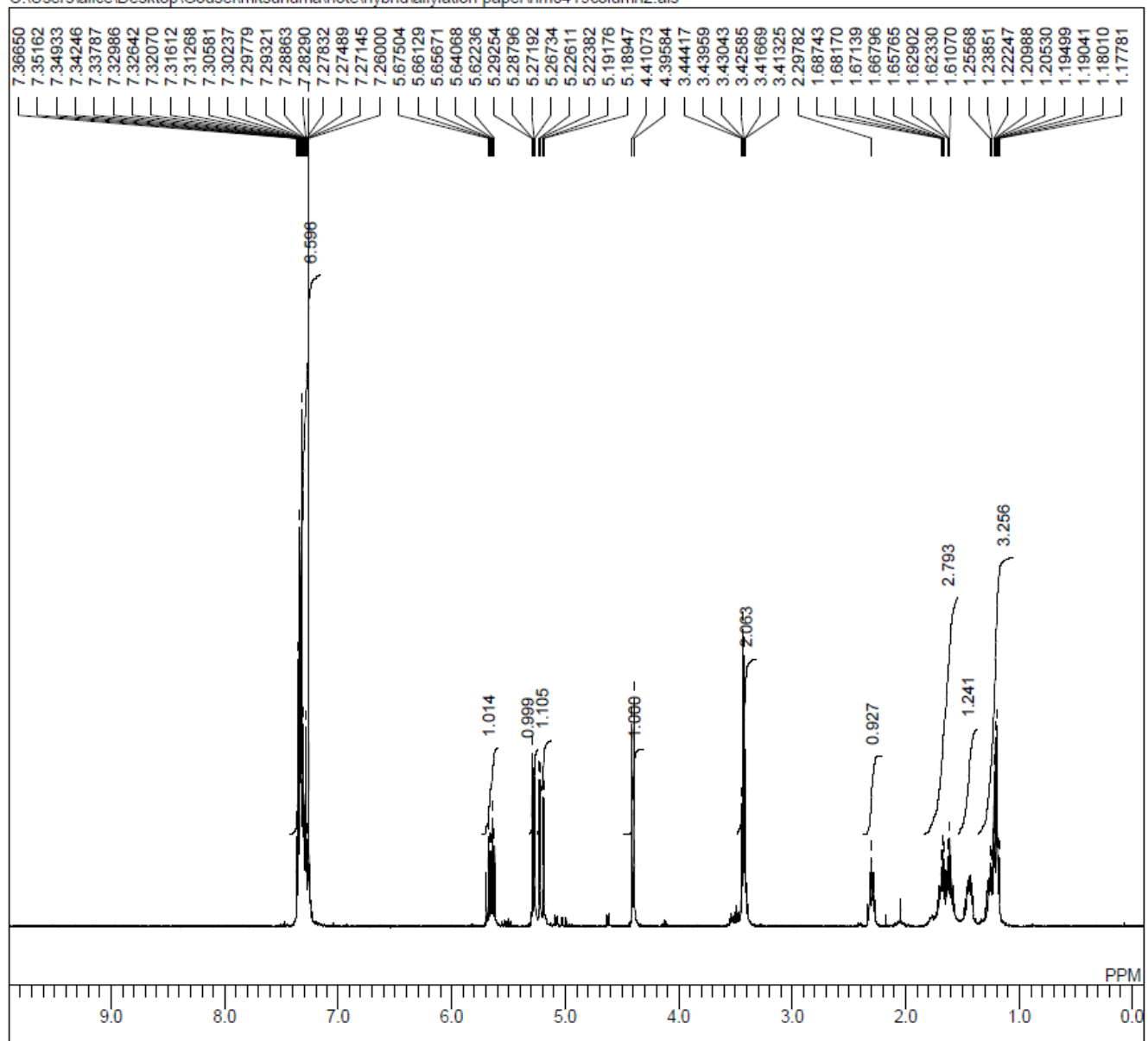


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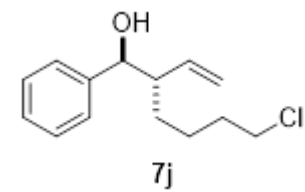


DFILE st327tm13C-1-1.als
COMNT
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OBNUC 13C
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OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 43
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

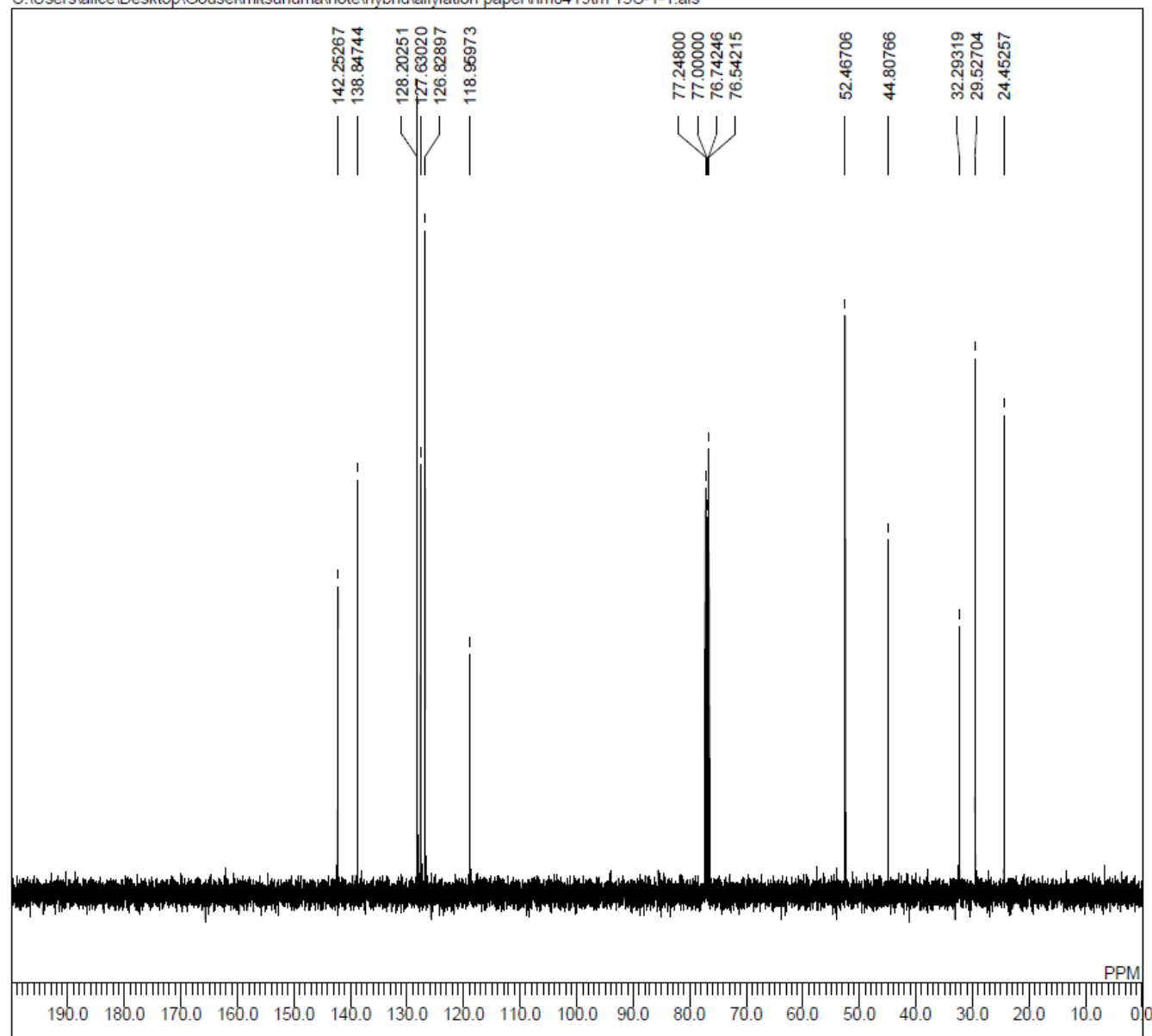
C:\Users\alice\Desktop\Gousei\mitsunuma\note\hybrid\allylation paper\hm0419column2.als



DFILE hm0419column2.als
COMNT
DATIM 2020-03-31 18:02:47
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 42

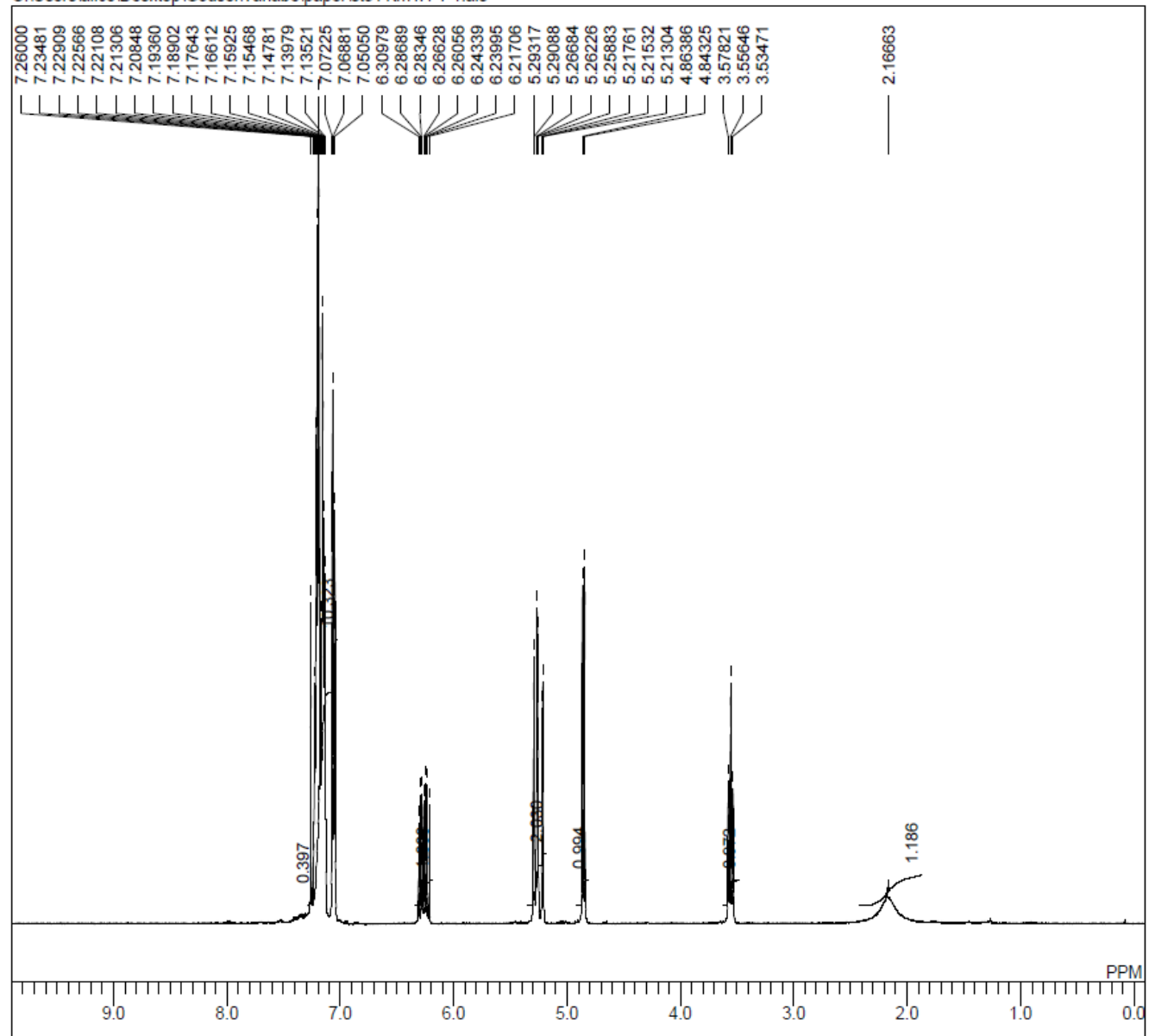


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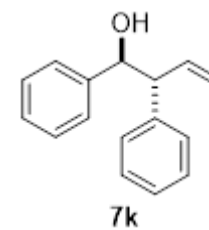


DFILE hm0419tm 13C-1-1.als
COMNT
DATIM 2020-03-31 18:16:10
OBNUC 13C
EXMOD carbon.jpg
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 24
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

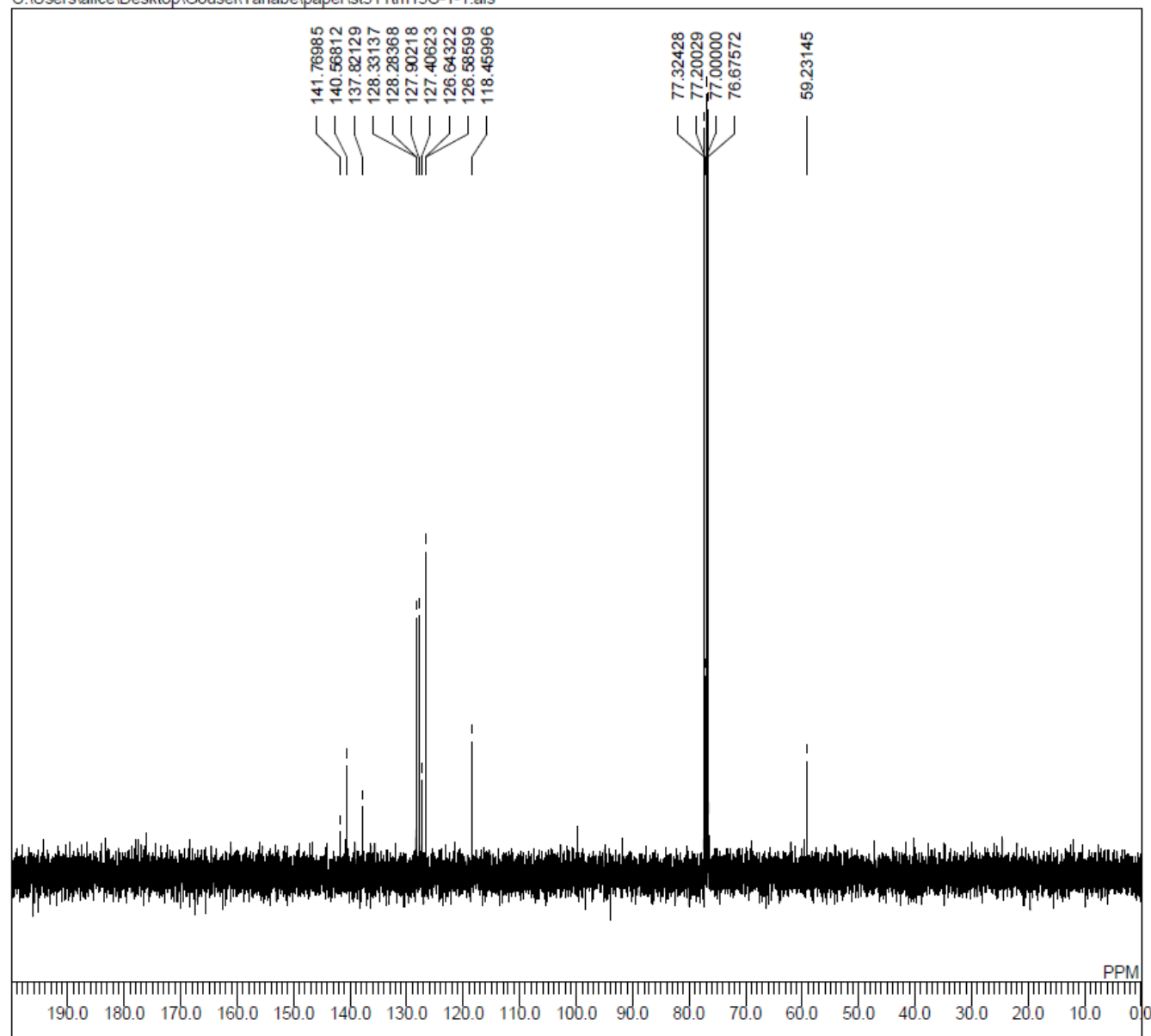
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DFILE st311tm1H-1-1.als
COMNT
DATIM 03-03-2020 19:49:49
OBNUC 1H
EXMOD proton.jxp
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 13107
FREQU 5878.90 Hz
SCANS 8
ACQTM 2.2295 sec
PD 6.0000 sec
PW1 5.17 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 40

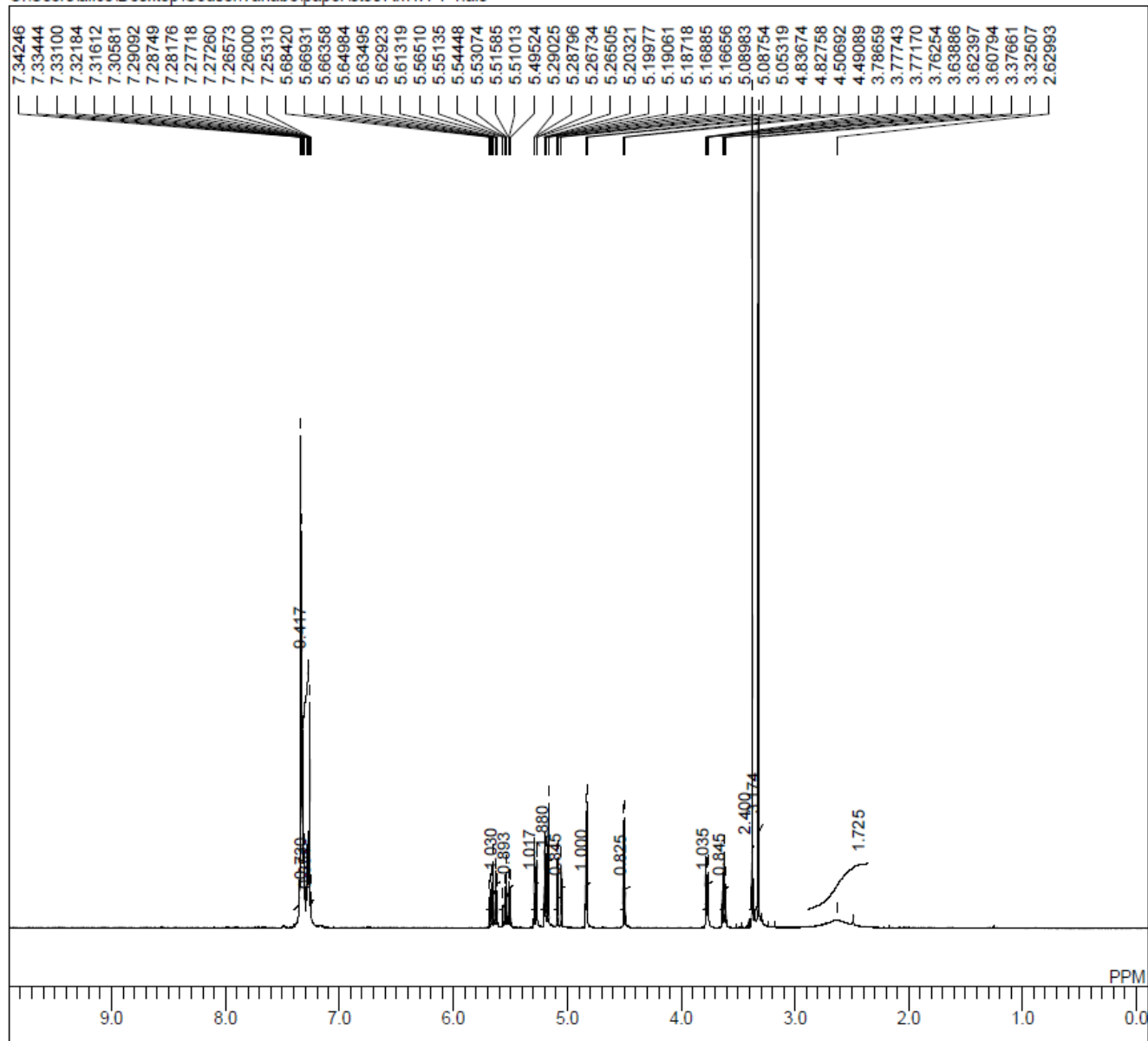


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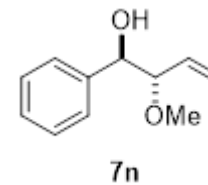


DFILE	st311tm13C-1-1.als
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OBNUC	13C
EXMOD	carbon.jpg
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.54 Hz
SCANS	47
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.12 usec
IRNUC	1H
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

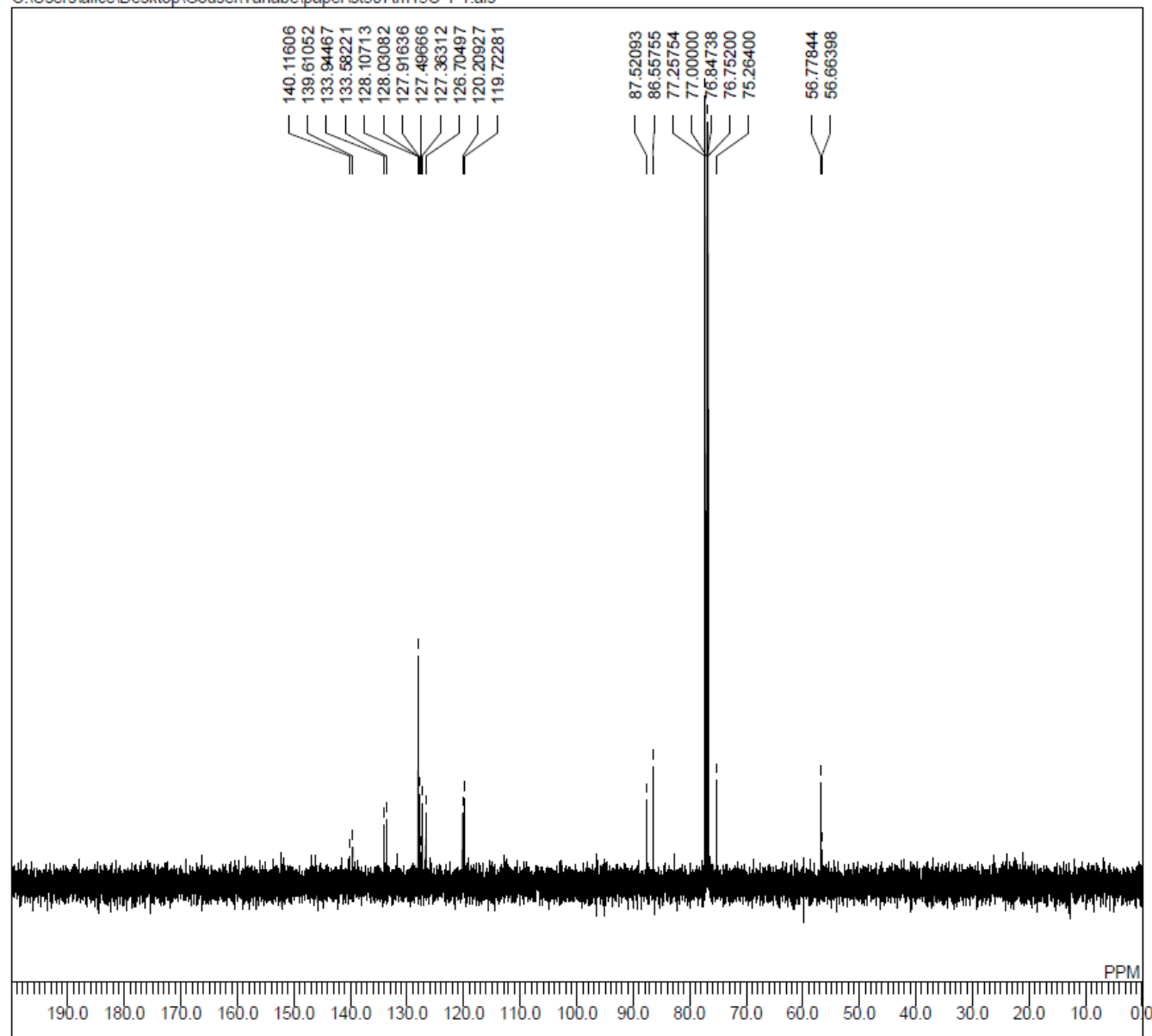
C:\Users\alice\Desktop\Gousei\Tanabe\paper\st307m1H-1-1.als



DFILE st307m1H-1-1.als
COMNT
DATIM 2020-03-03 15:30:03
OBNUC 1H
EXMOD proton.jpg
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 40

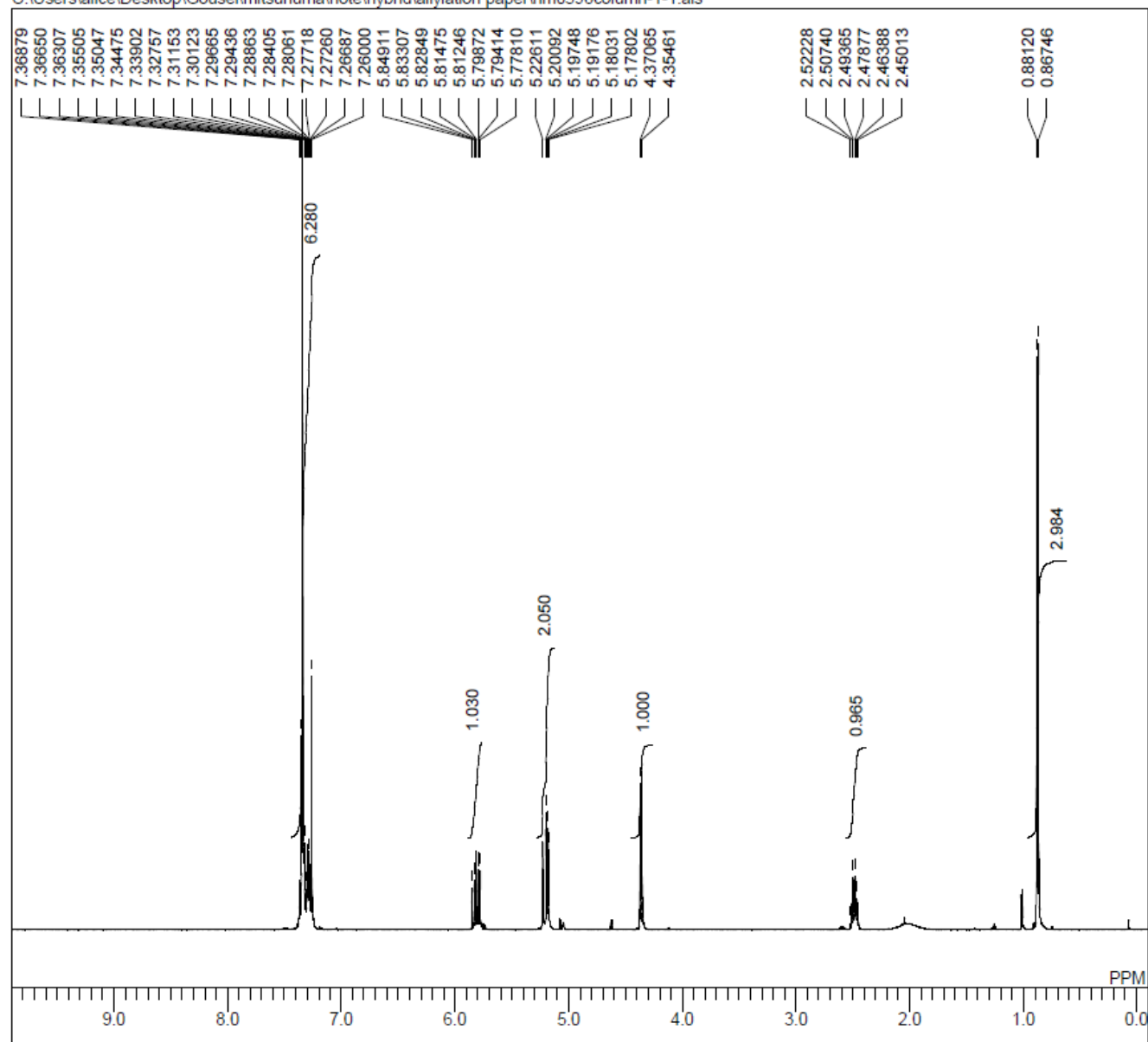


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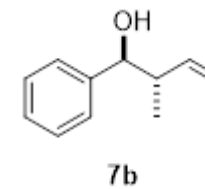


DFILE st307m13C-1-1.als
COMNT
DATIM 2020-03-03 15:32:20
OBNUC 13C
EXMOD carbon.jpg
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 41
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

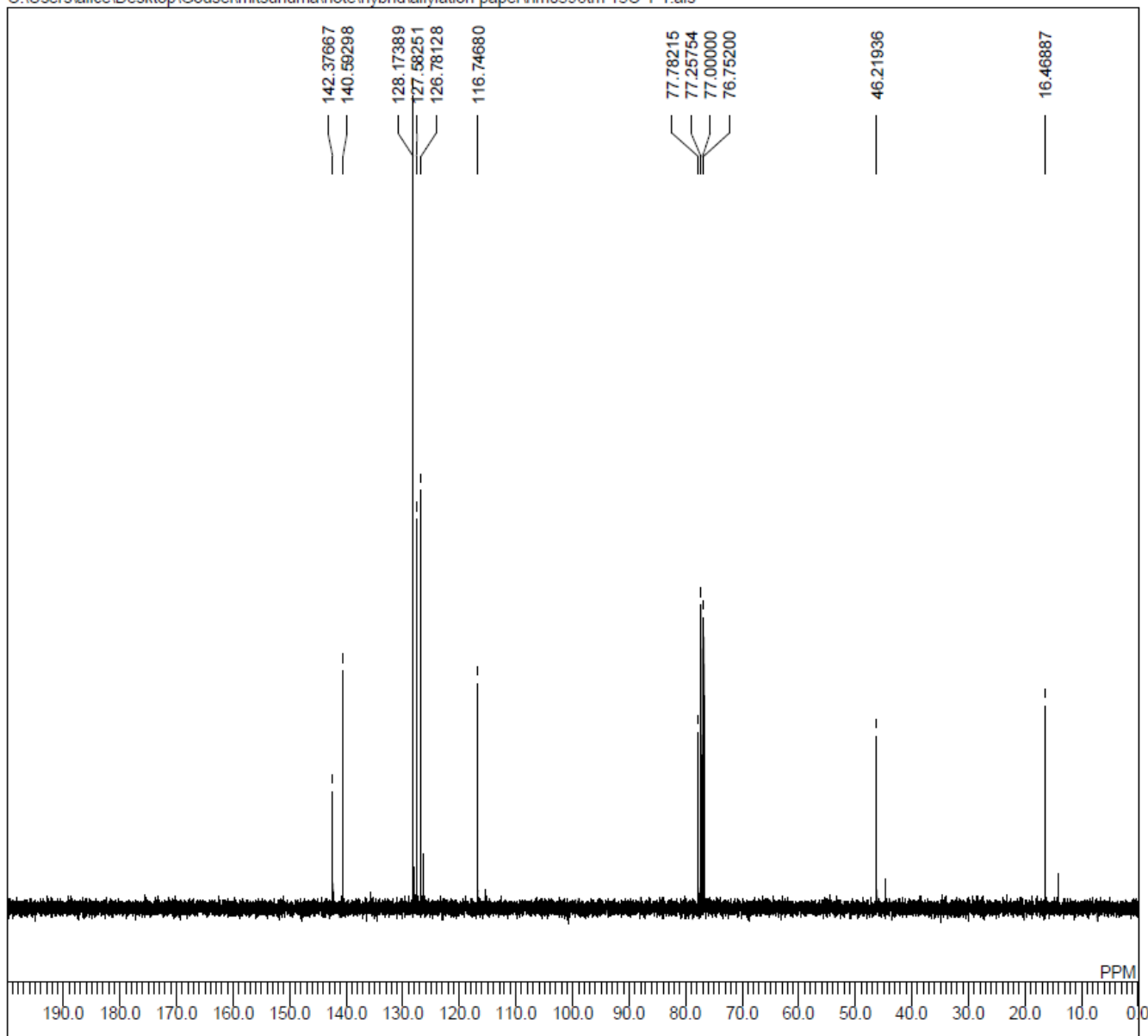
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DFILE hm0398column-1-1.als
COMNT
DATIM 2020-03-11 23:44:50
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 42

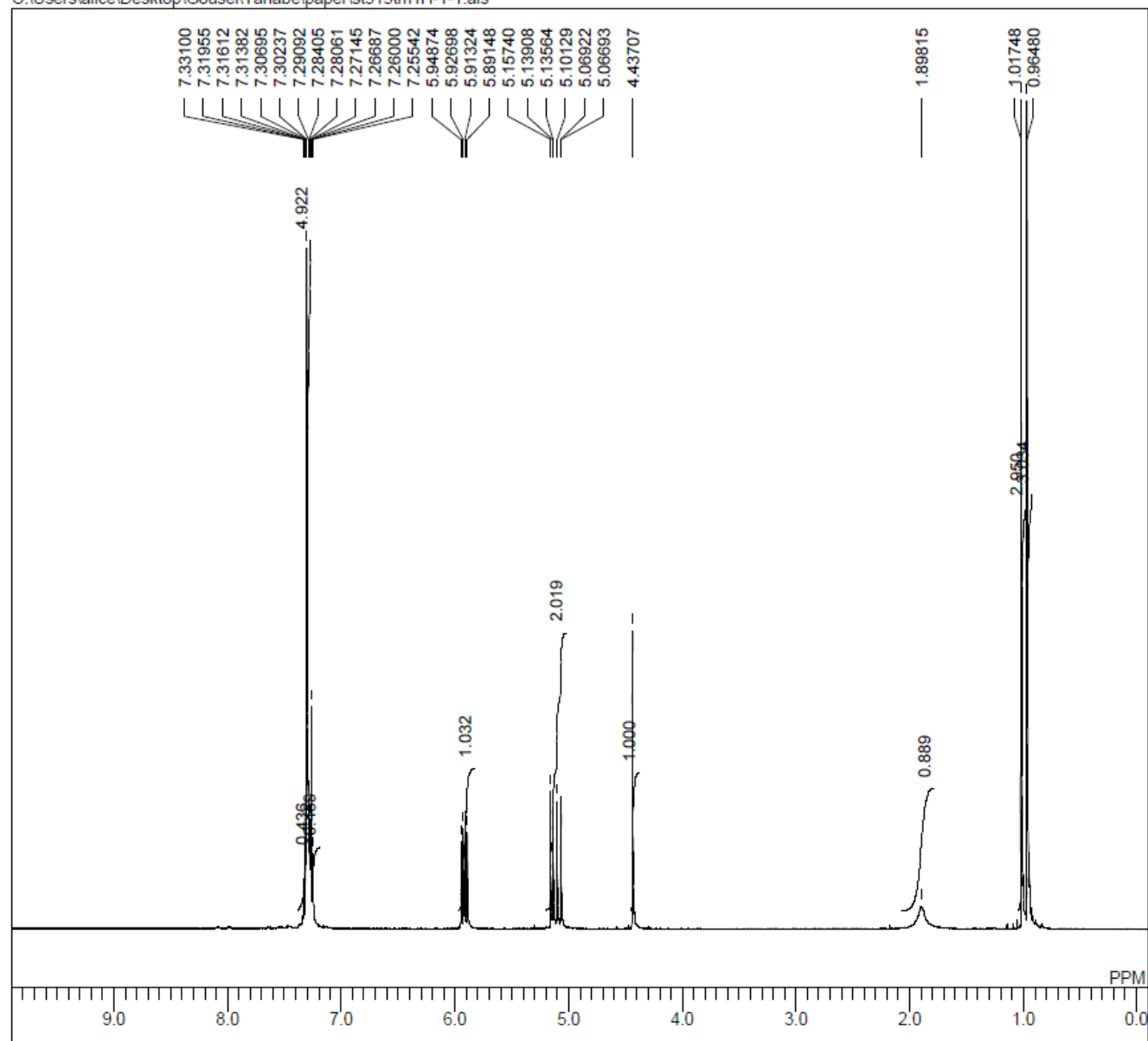


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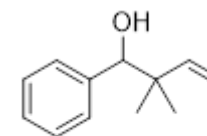


DFILE hm0398tm 13C-1-1.als
COMNT
DATIM 2020-03-12 10:38:58
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 32
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

C:\Users\alice\Desktop\Gousei\Tanabe\paper\st315tm1H-1-1.als

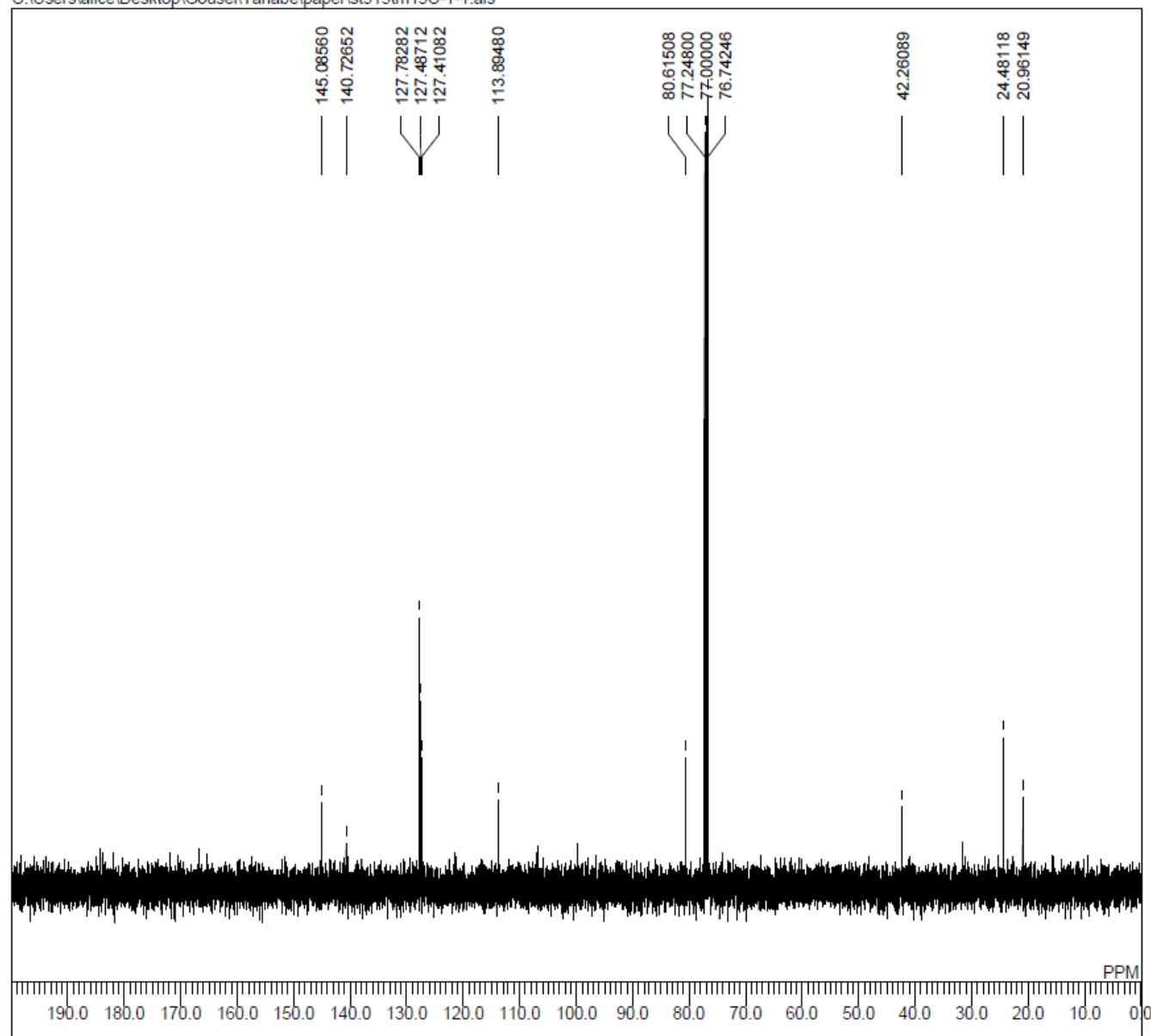


DFILE st315tm1H-1-1.als
COMNT
DATIM 2020-03-06 17:26:11
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 42



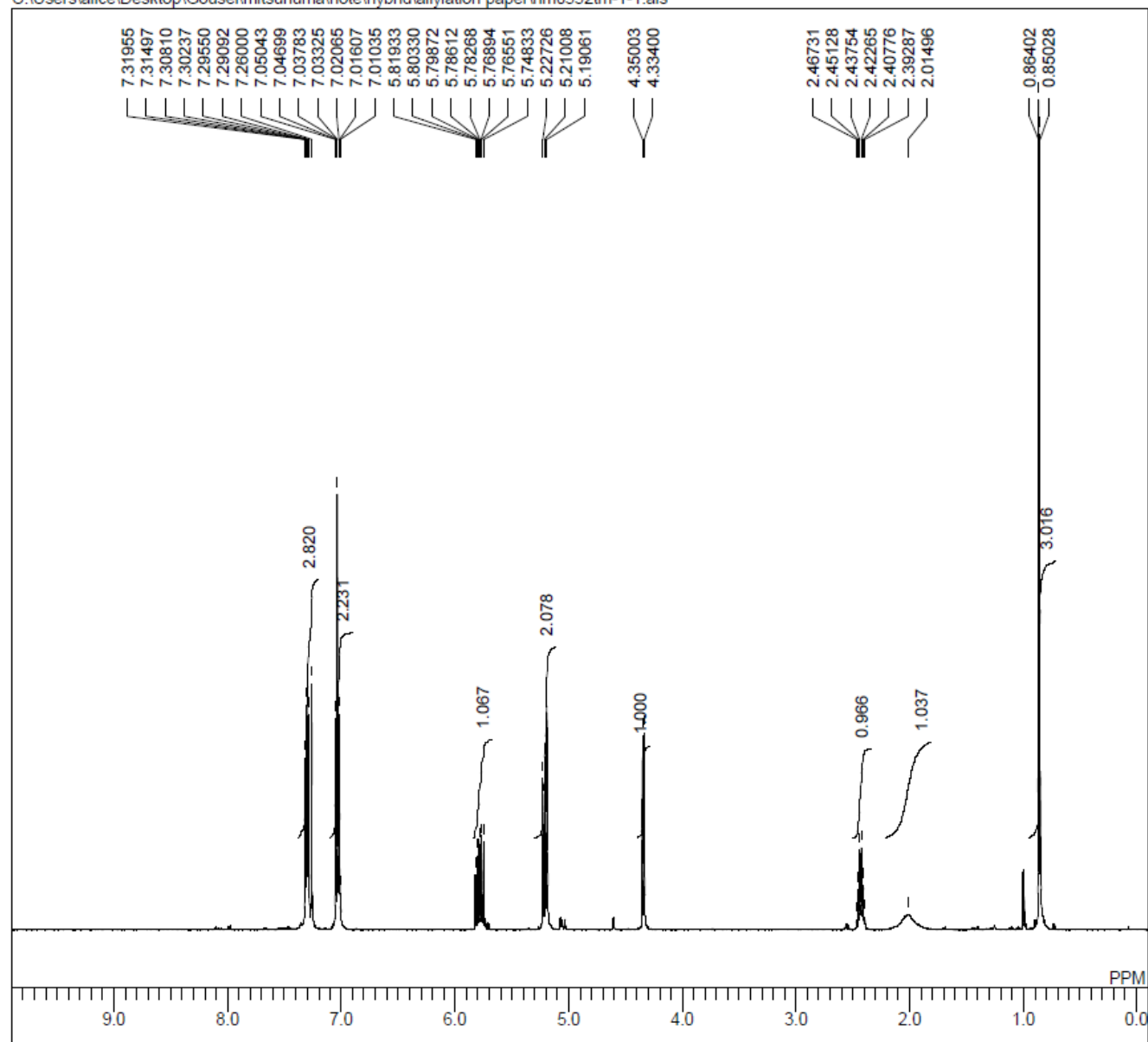
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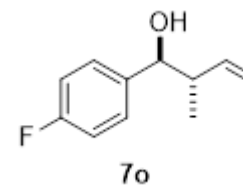


DFILE st315tm13C-1-1.als
COMNT
DATIM 2020-03-06 17:27:50
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 43
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

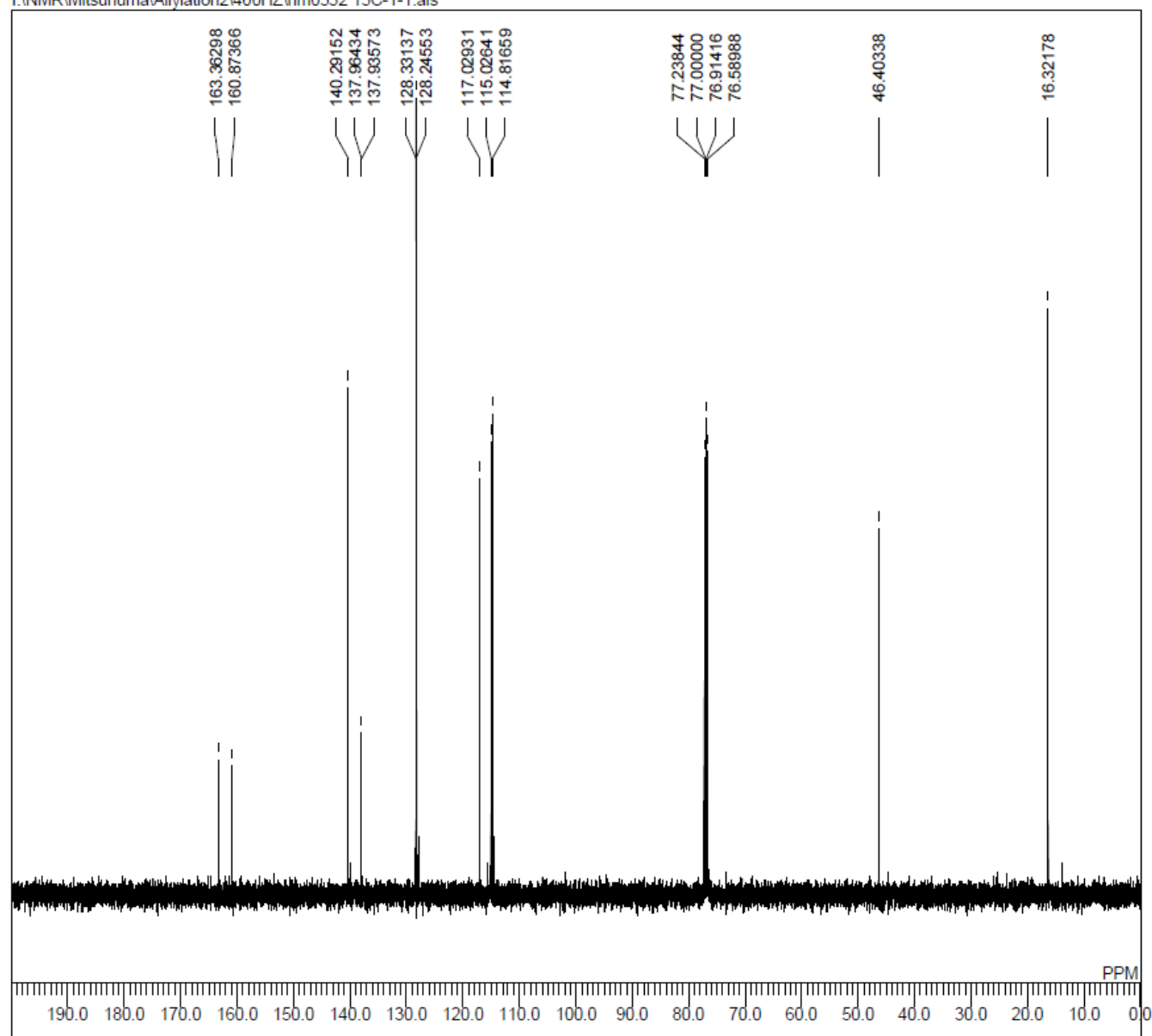
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DFILE hm0332tm-1-1.als
 COMNT
 DATIM 2020-02-10 18:00:30
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 13107
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 6.0000 sec
 PW1 5.55 usec
 IRNUC 1H
 CTEMP 20.7 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 42

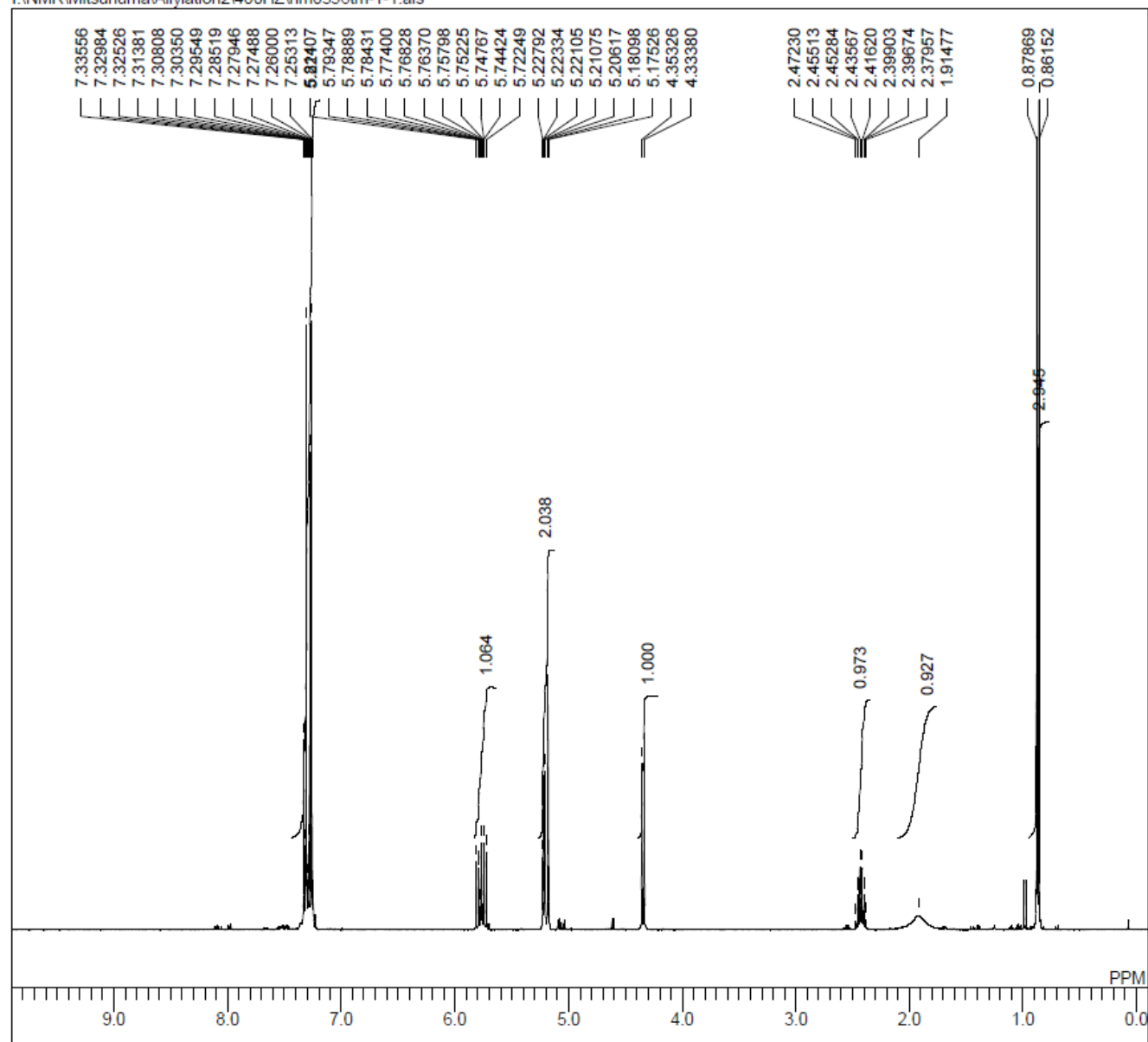


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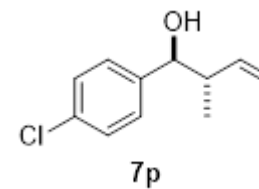


DFILE hm0332 13C-1-1.als
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DATIM 10-02-2020 19:49:20
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 48
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

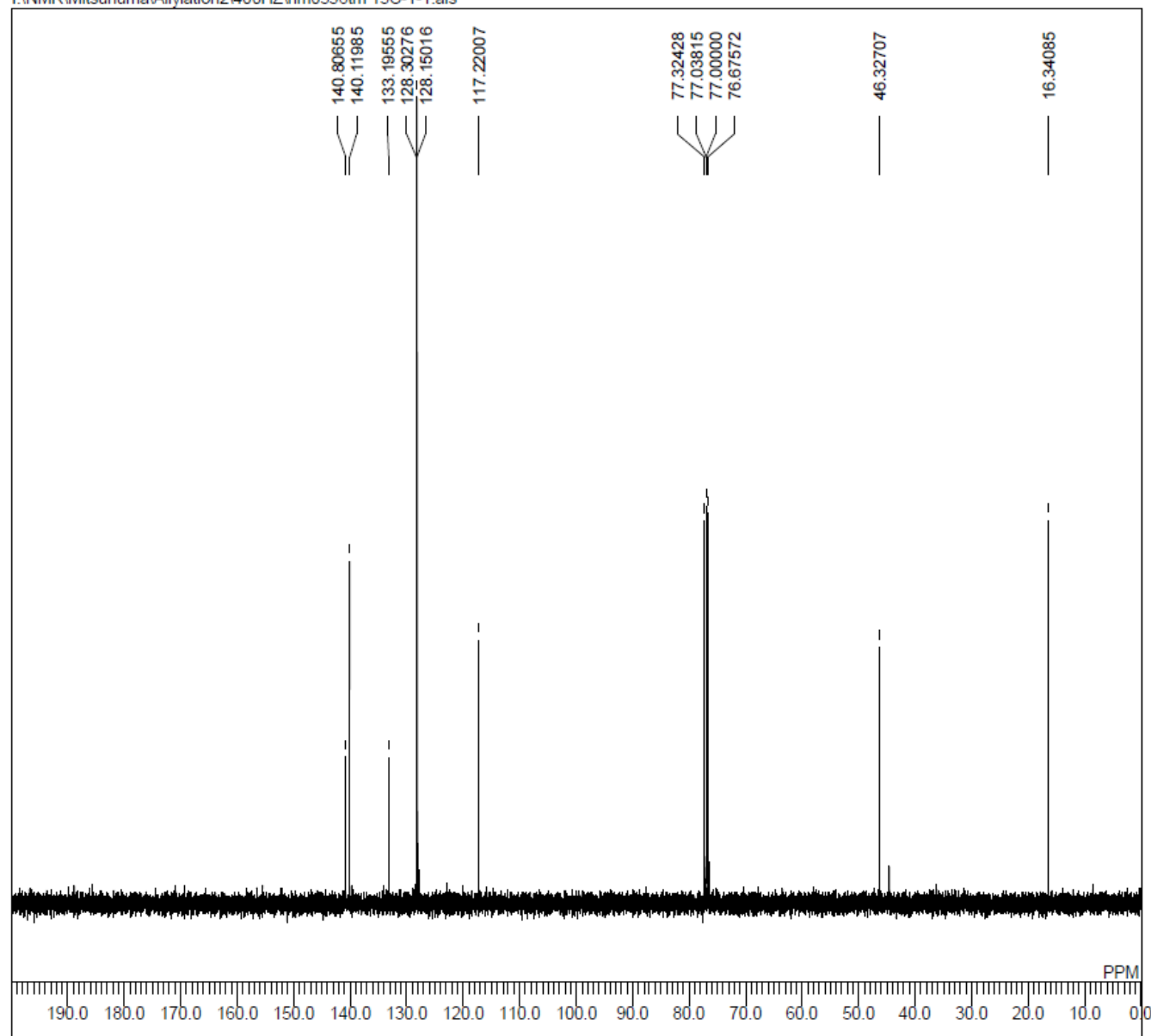
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DFILE hm0356tm-1-1.als
 COMNT
 DATIM 03-03-2020 15:56:36
 OBNUC 1H
 EXMOD proton.jpg
 OBFRQ 391.78 MHz
 OBSET 8.51 KHz
 OBFIN 3.34 Hz
 POINT 13107
 FREQU 5878.90 Hz
 SCANS 8
 ACQTM 2.2295 sec
 PD 6.0000 sec
 PW1 5.17 usec
 IRNUC 1H
 CTEMP 20.2 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 46

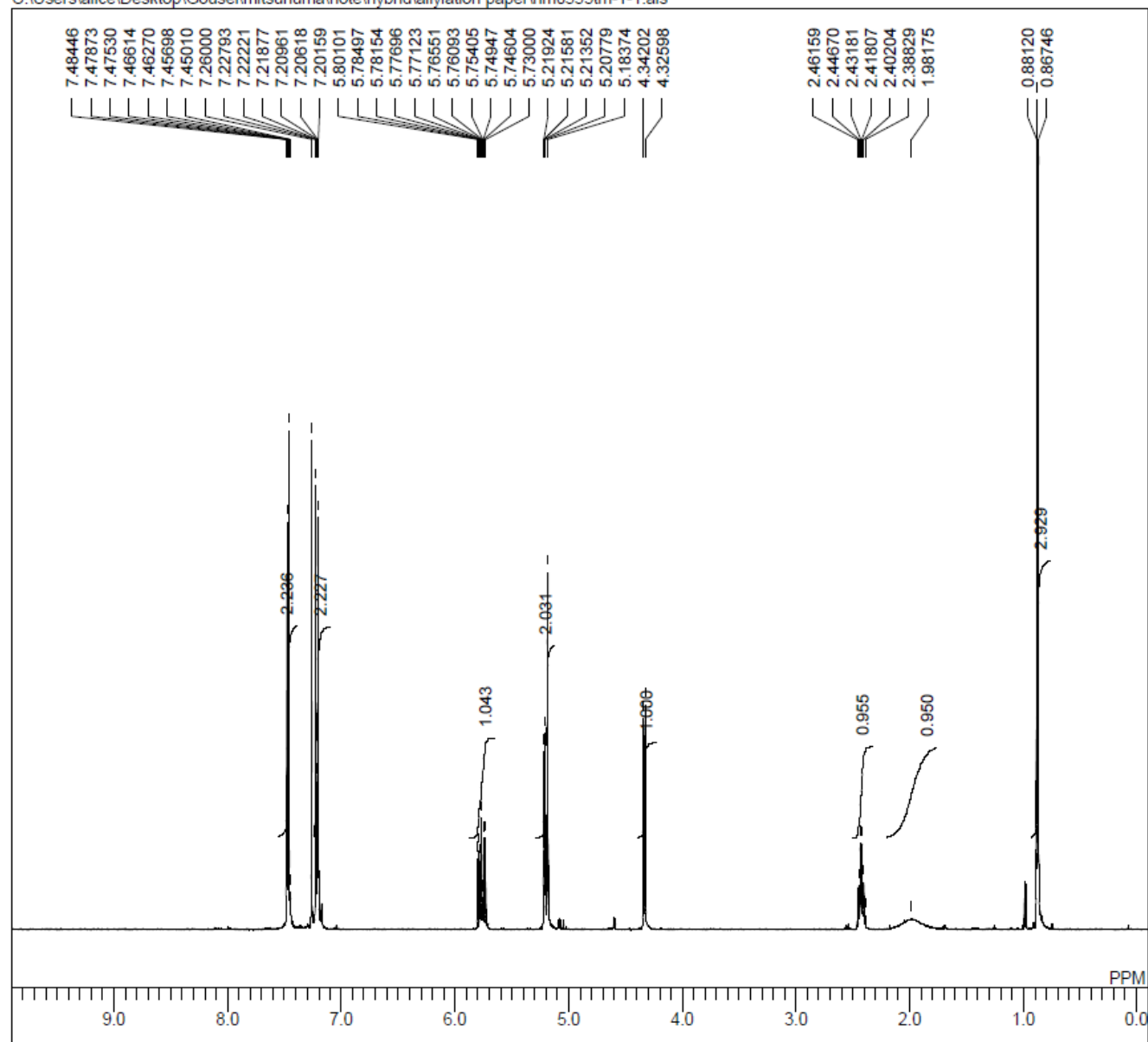


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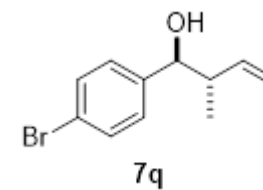


DFILE hm0356tm 13C-1-1.als
COMNT
DATIM 03-03-2020 16:47:33
OBNUC 13C
EXMOD carbon.jpg
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 56
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

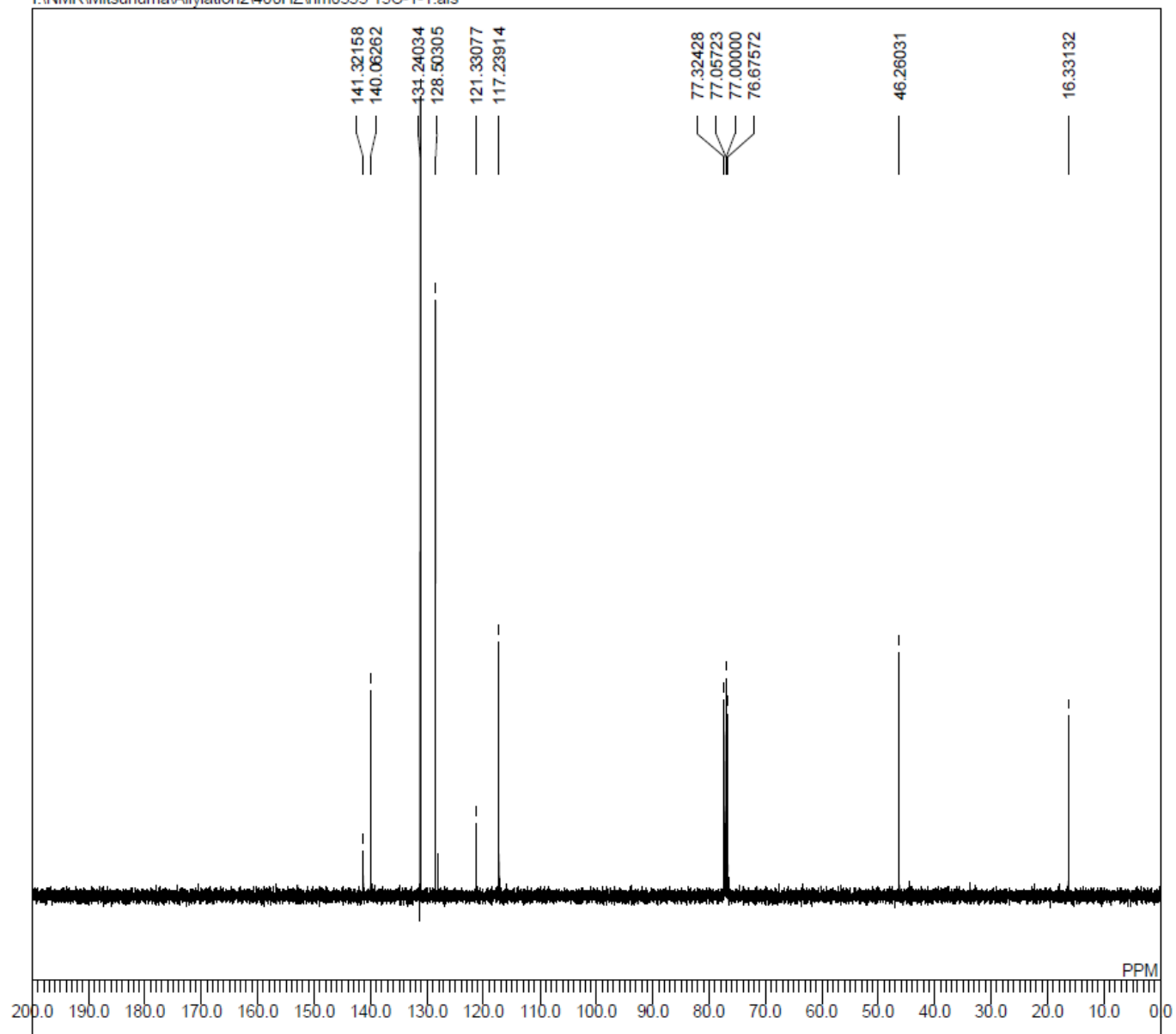
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DFILE hm0333tm-1-1.als
COMNT
DATIM 2020-02-10 18:04:27
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 42

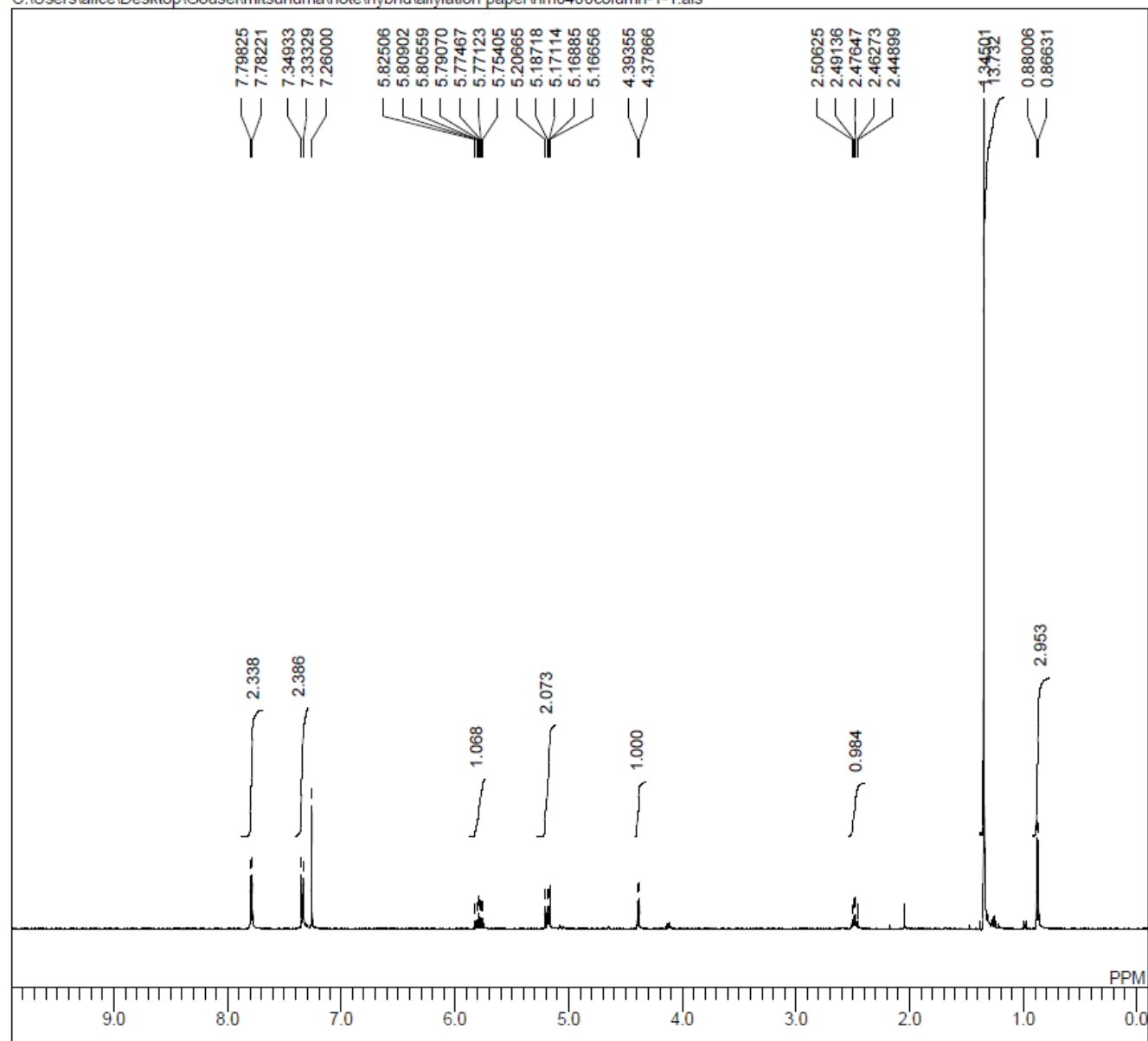


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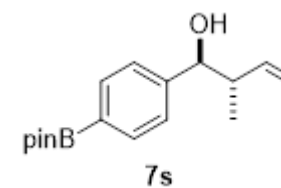


DFILE hm0333 13C-1-1.als
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EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 40
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

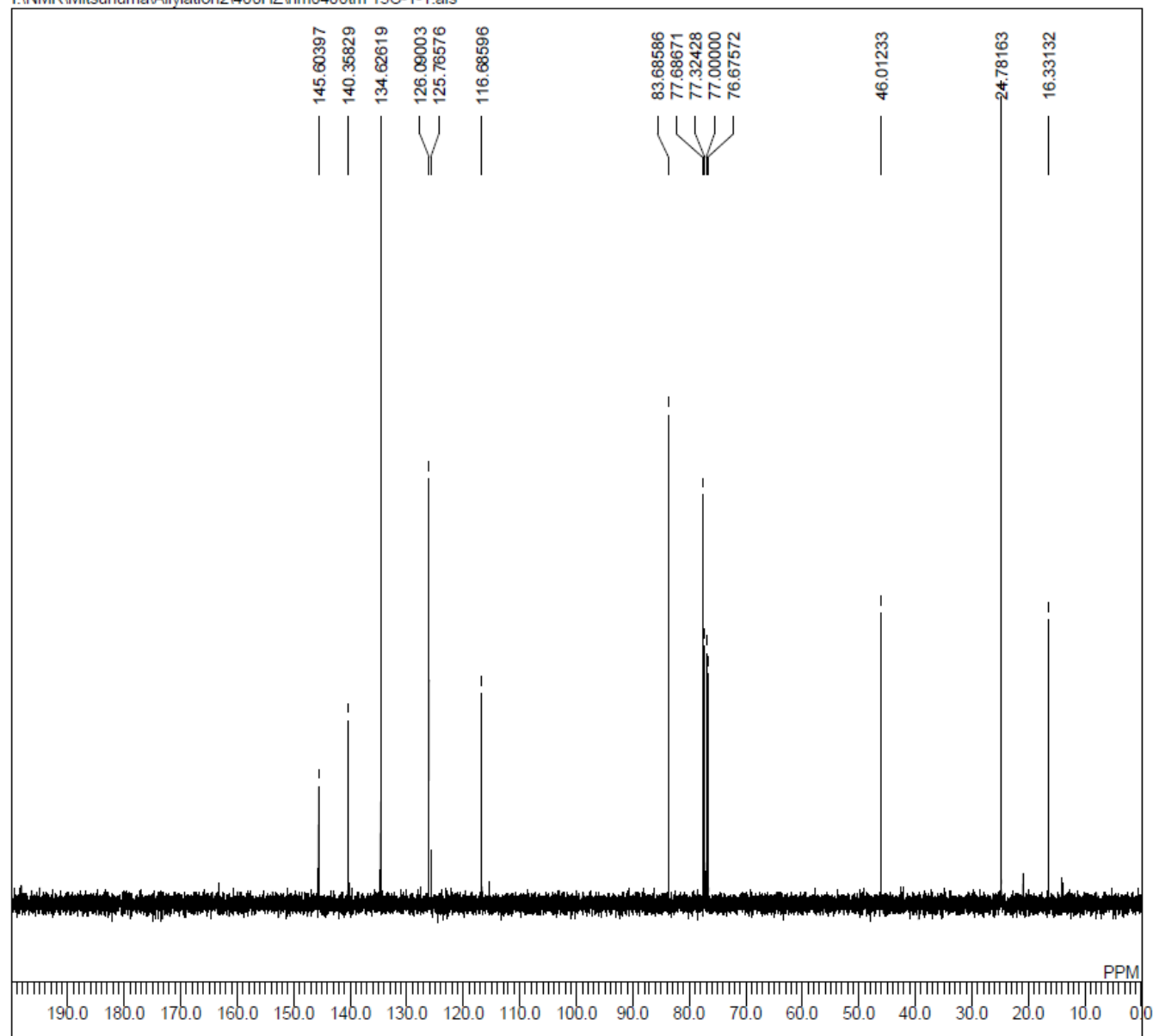
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DFILE hm0400column-1-1.als
COMNT
DATIM 2020-03-12 17:38:26
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 42

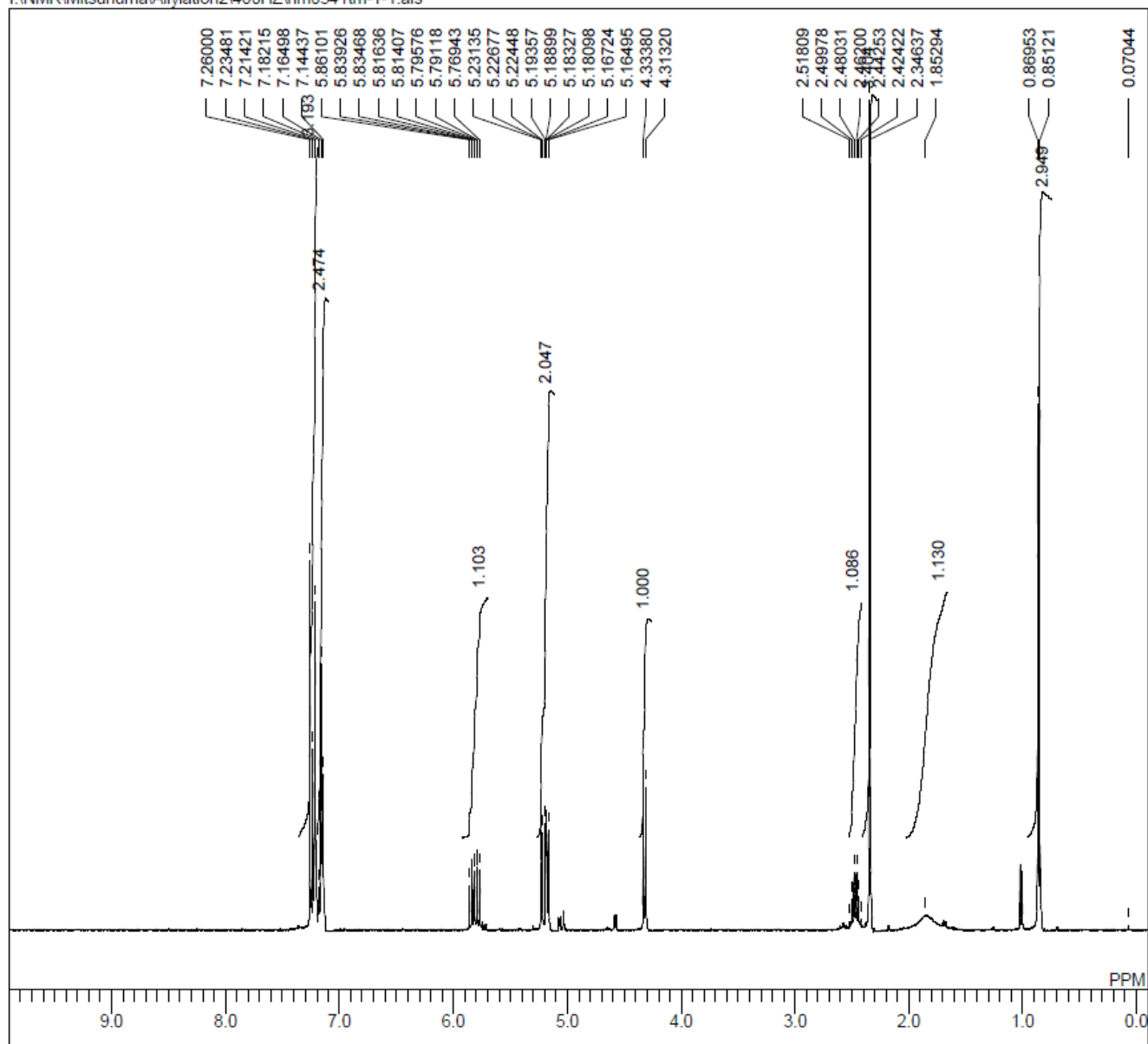


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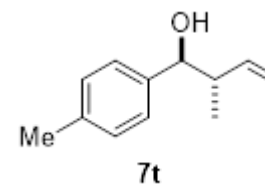


DFILE hm0400tm 13C-1-1.als
COMNT
DATIM 12-03-2020 18:34:47
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 20
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

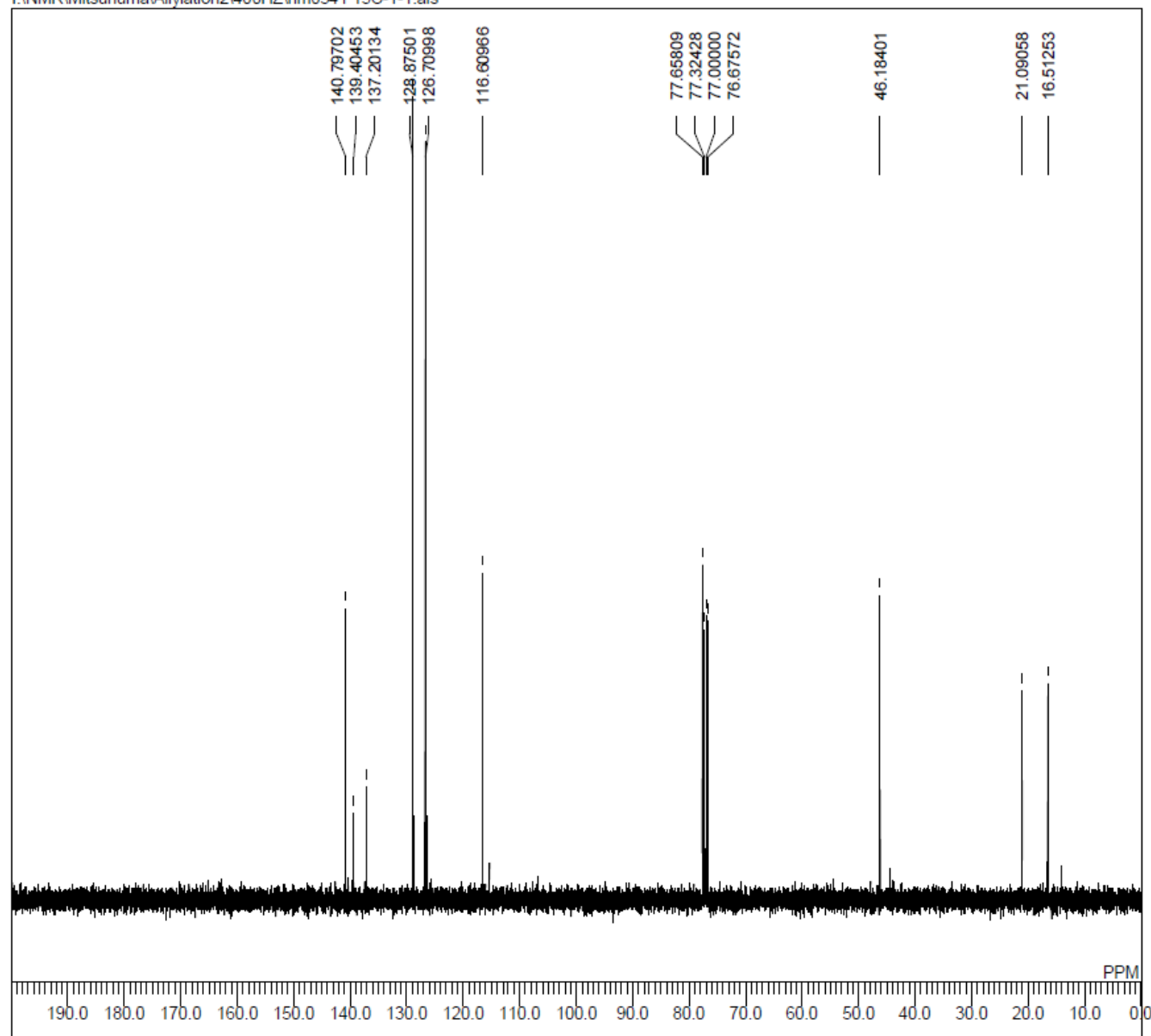
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DFILE hm0341tm-1-1.als
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 DATIM 13-02-2020 17:15:58
 OBNUC 1H
 EXMOD proton.jpg
 OBFRQ 391.78 MHz
 OBSET 8.51 KHz
 OBFIN 3.34 Hz
 POINT 13107
 FREQU 5878.90 Hz
 SCANS 8
 ACQTM 2.2295 sec
 PD 6.0000 sec
 PW1 5.17 usec
 IRNUC 1H
 CTEMP 20.5 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 46

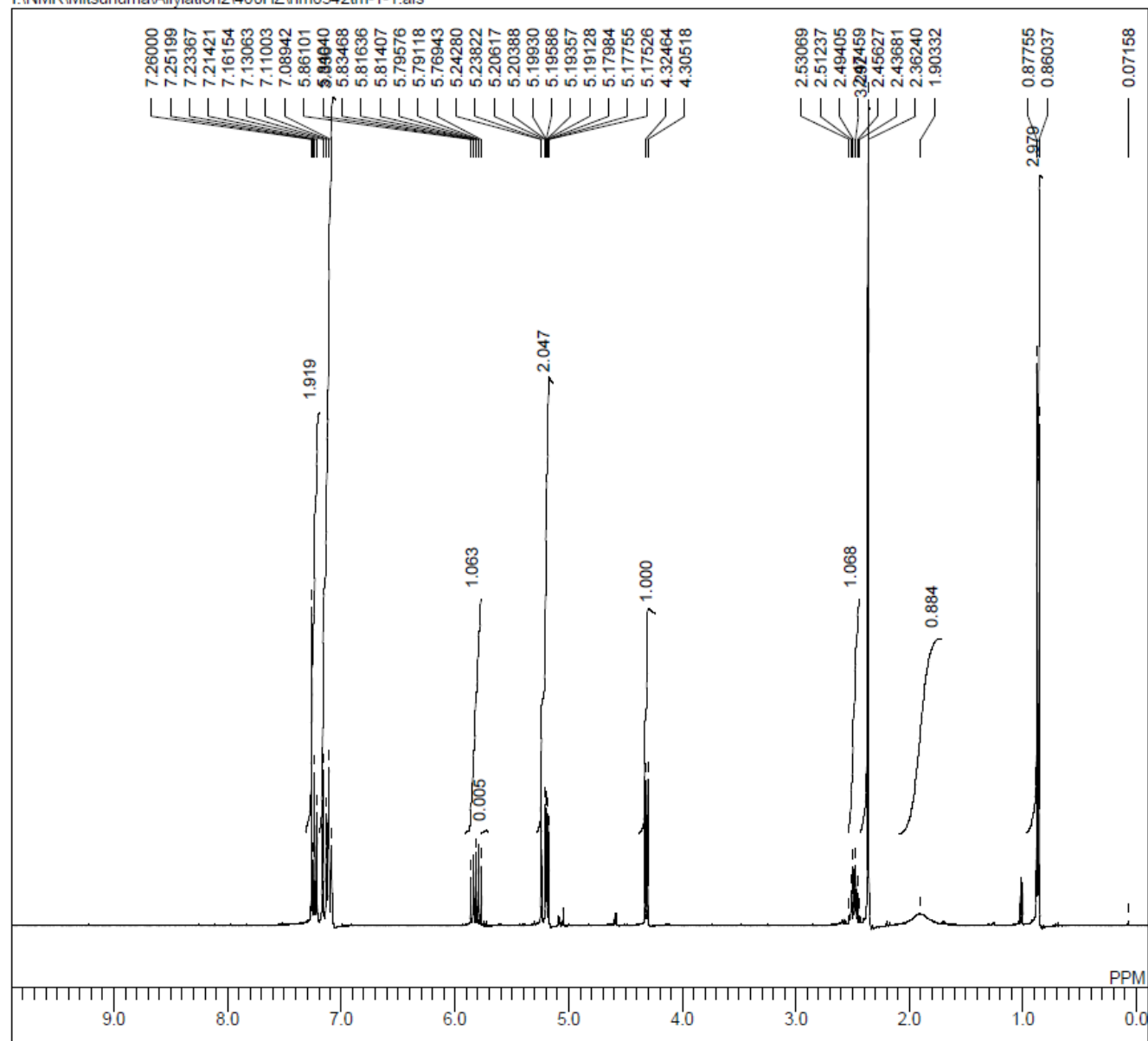


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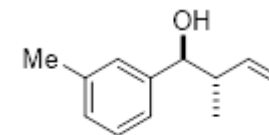


DFILE hm0341 13C-1-1.als
COMNT
DATIM 13-02-2020 19:13:05
OBNUC 13C
EXMOD carbon.jpg
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 24
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

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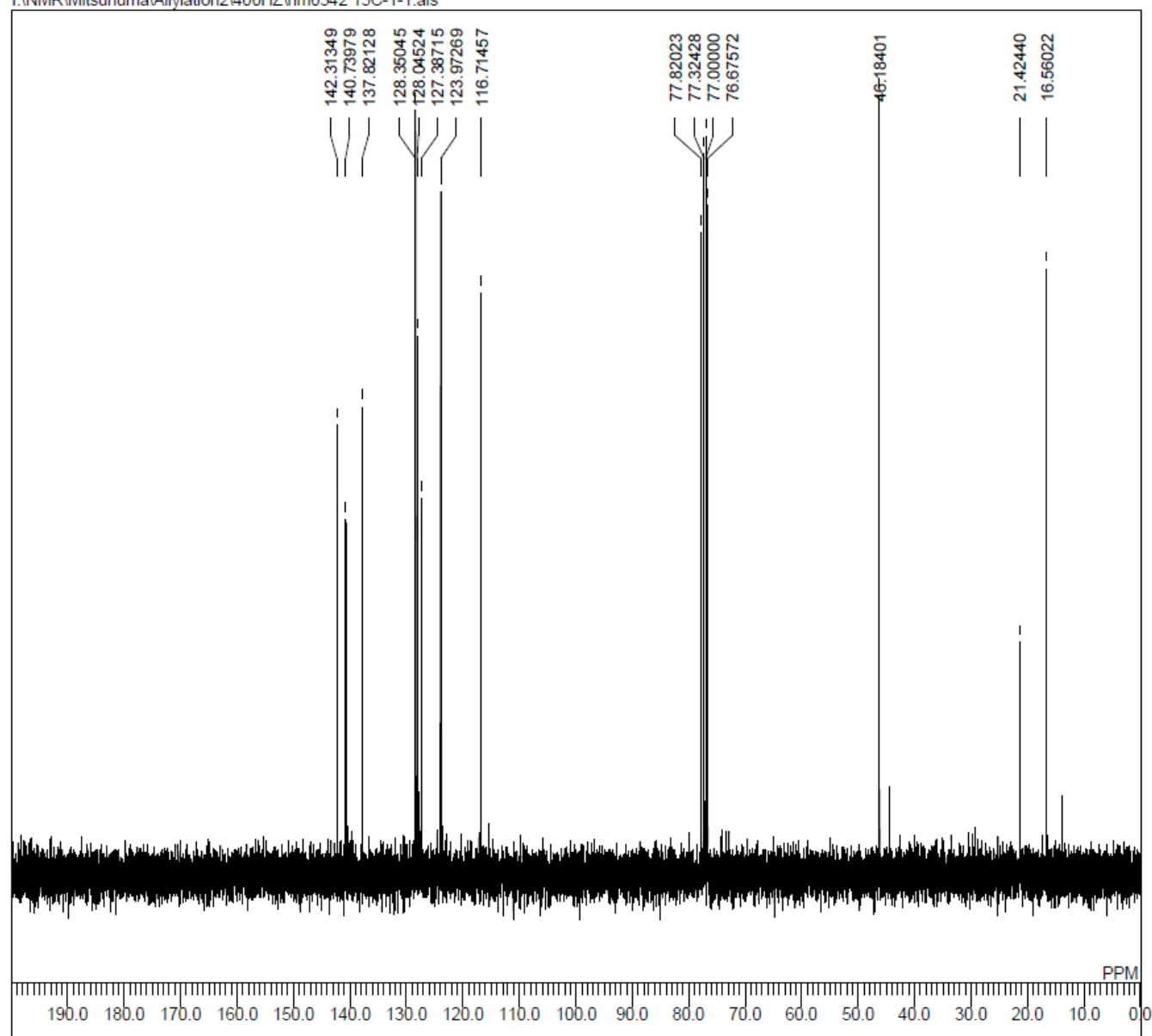


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 OBSET 8.51 KHz
 OBFIN 3.34 Hz
 POINT 13107
 FREQU 5878.90 Hz
 SCANS 8
 ACQTM 2.2295 sec
 PD 6.0000 sec
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 CTEMP 20.6 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 44



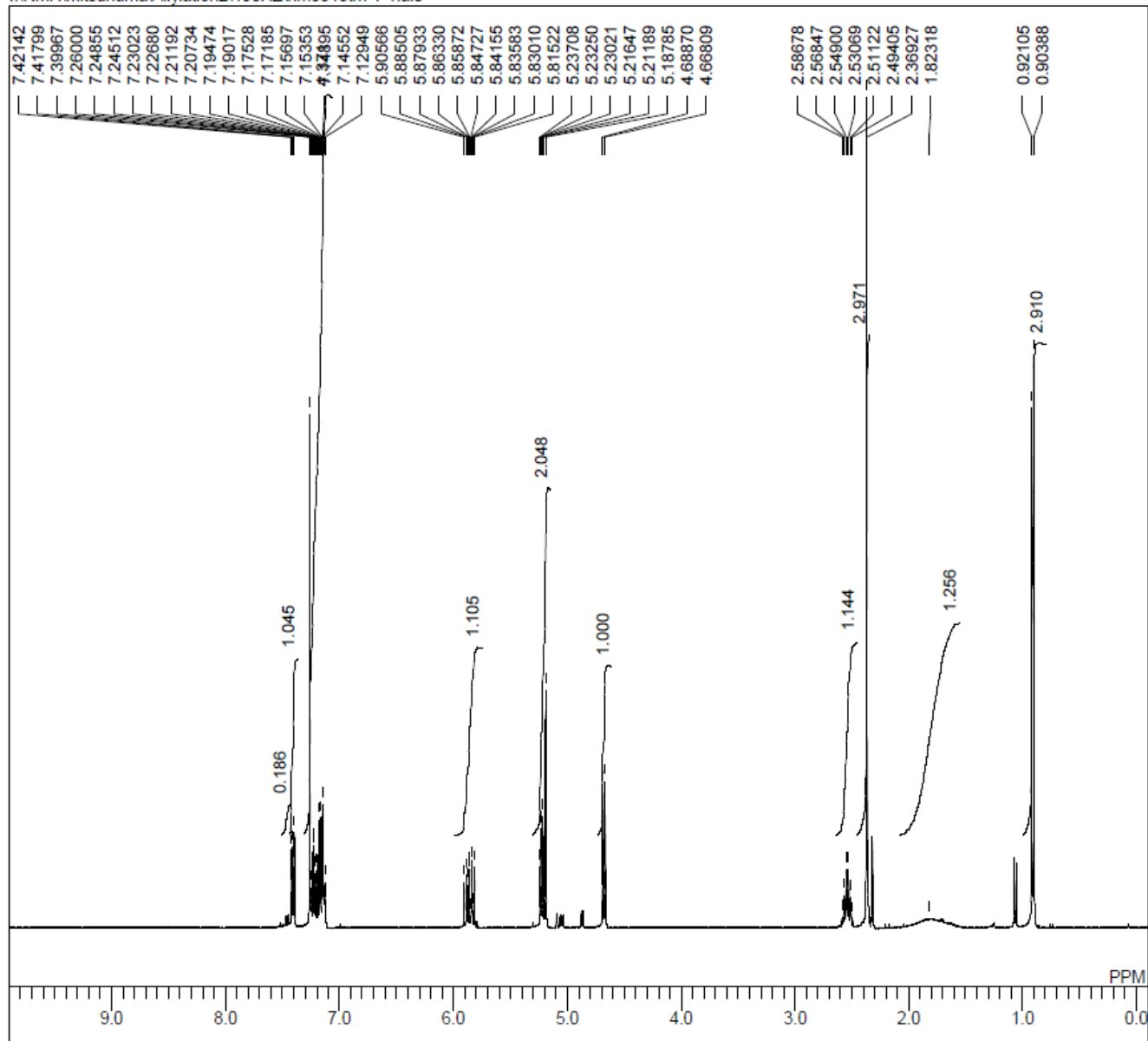
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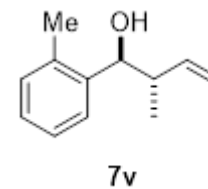


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EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 20
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

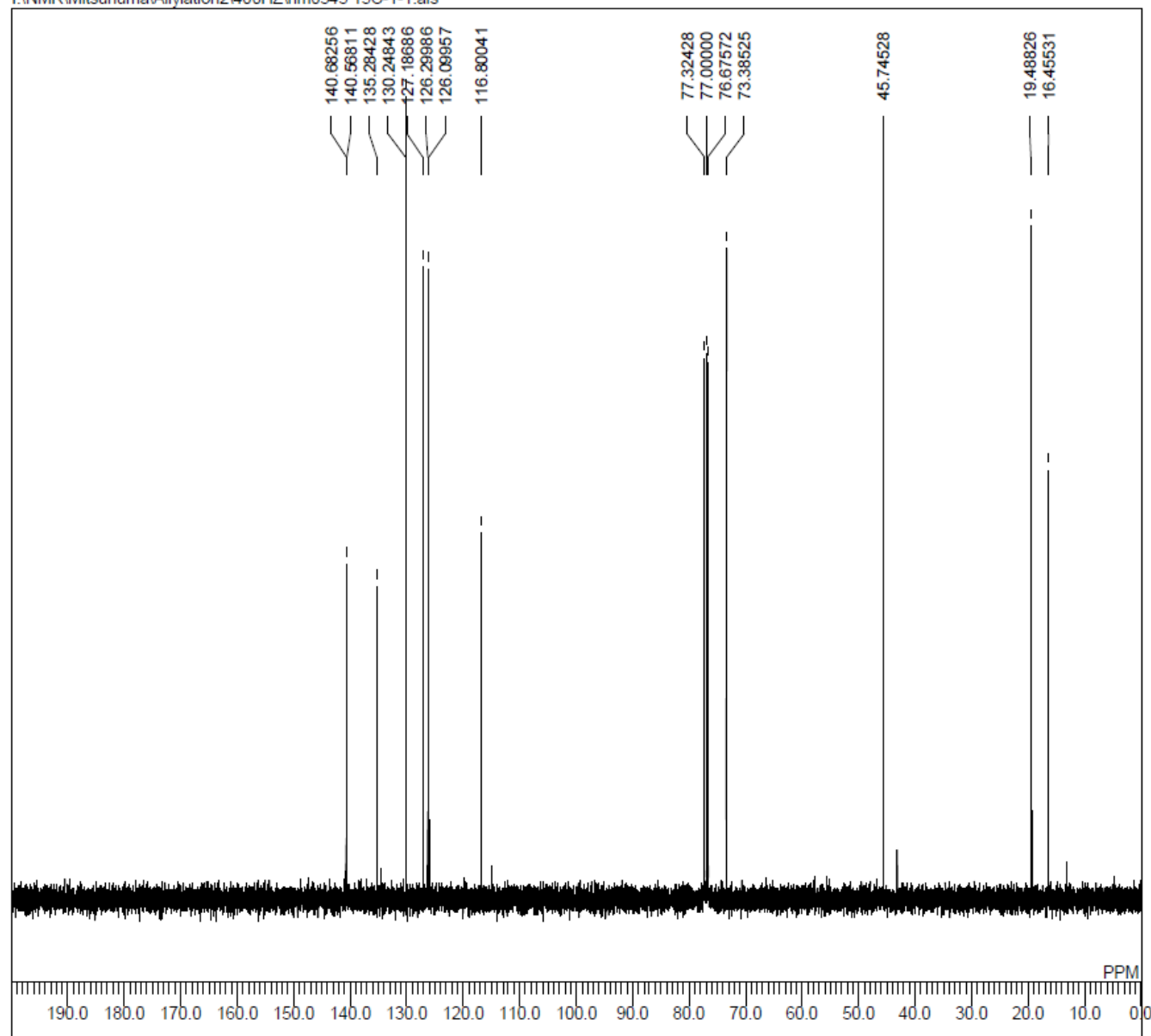
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DFILE hm0343tm-1-1.als
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 OBNUC 1H
 EXMOD proton.jpg
 OBFRQ 391.78 MHz
 OBSET 8.51 KHz
 OBFIN 3.34 Hz
 POINT 13107
 FREQU 5878.90 Hz
 SCANS 8
 ACQTM 2.2295 sec
 PD 6.0000 sec
 PW1 5.17 usec
 IRNUC 1H
 CTEMP 20.5 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 46

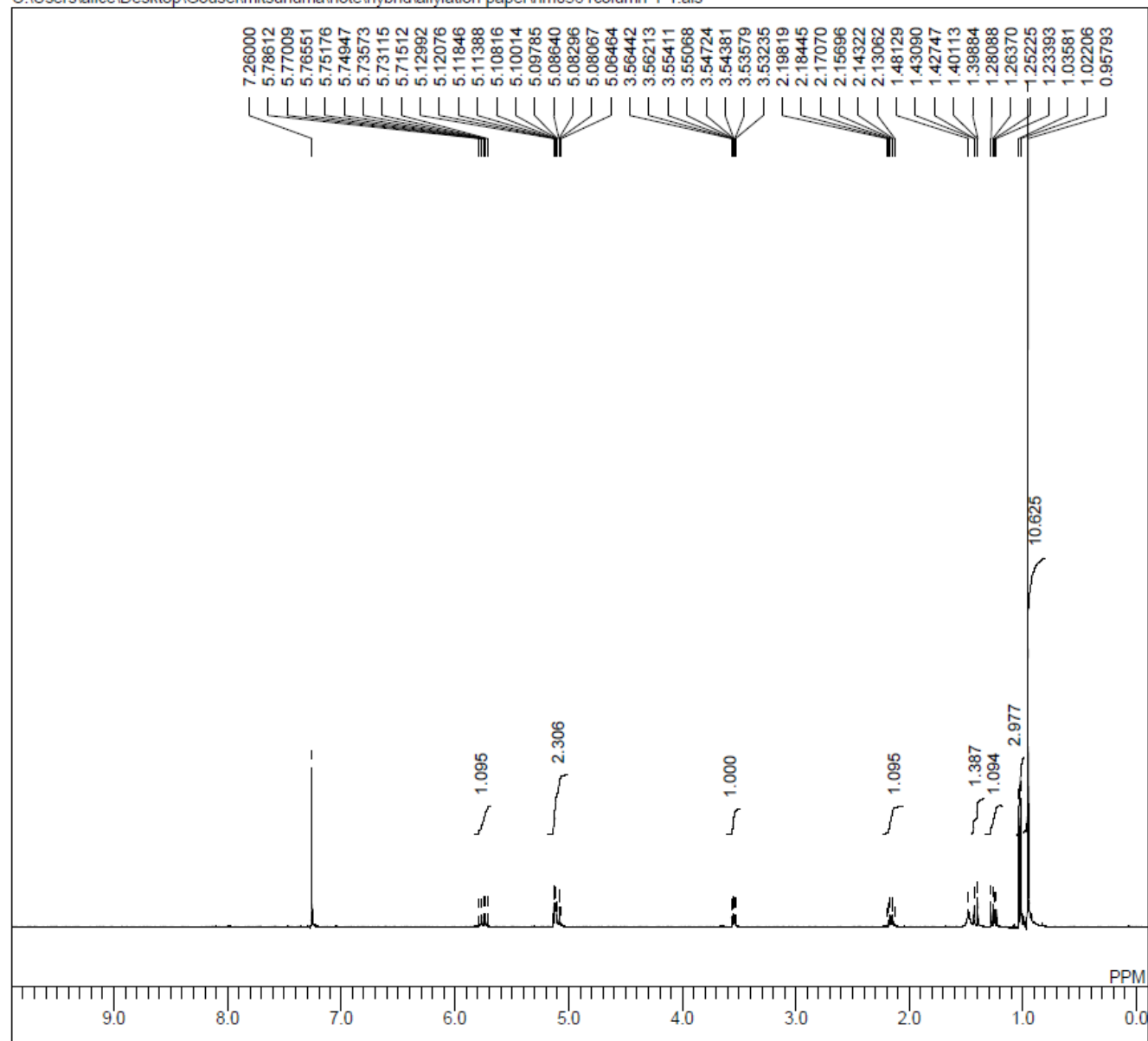


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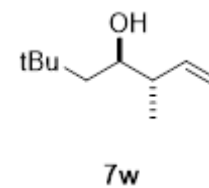


DFILE hm0343 13C-1-1.als
COMNT
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OBFRQ 98.52 MHz
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OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 60
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

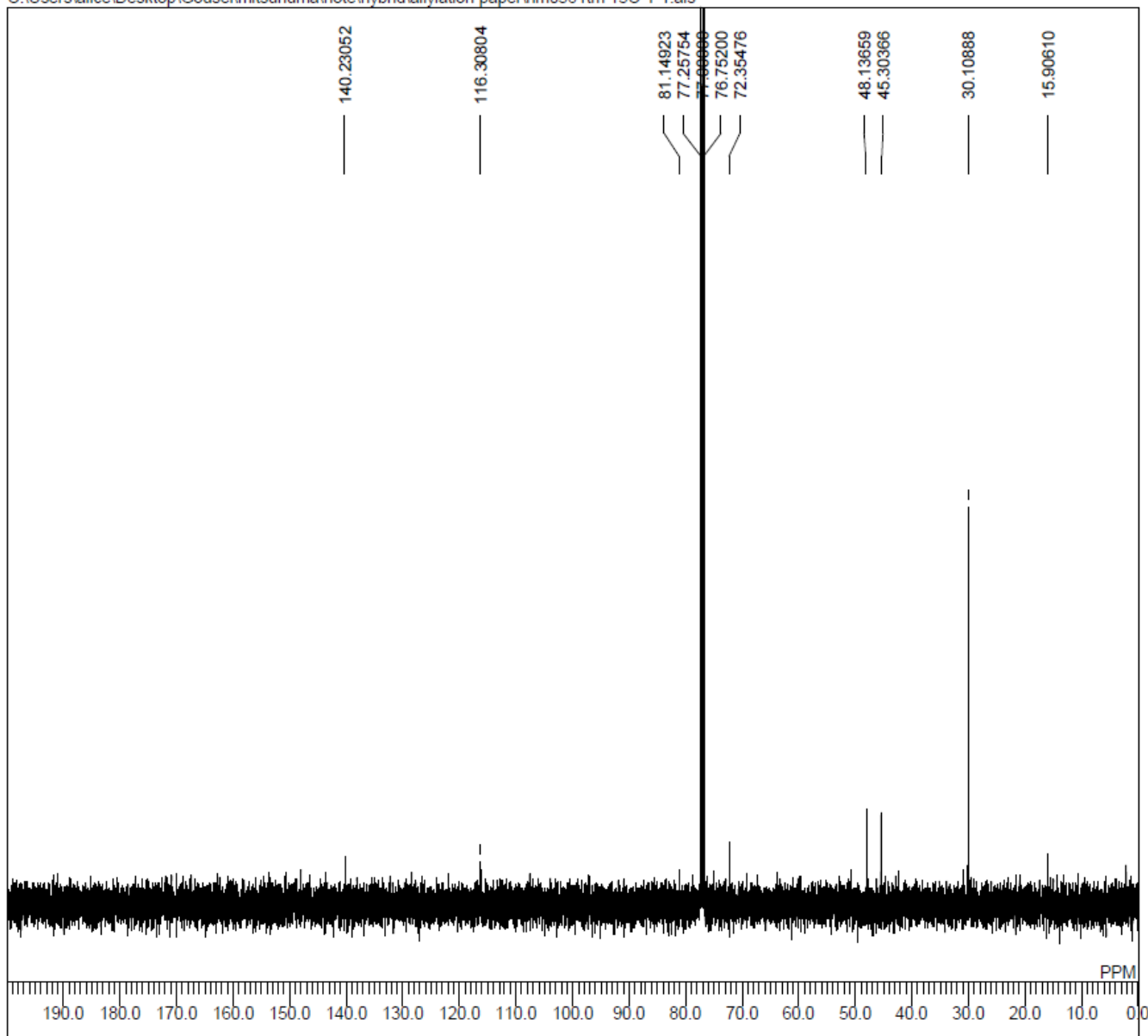
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DFILE hm0361column-1-1.als
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 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 13107
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 6.0000 sec
 PW1 5.55 usec
 IRNUC 1H
 CTEMP 21.1 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 42

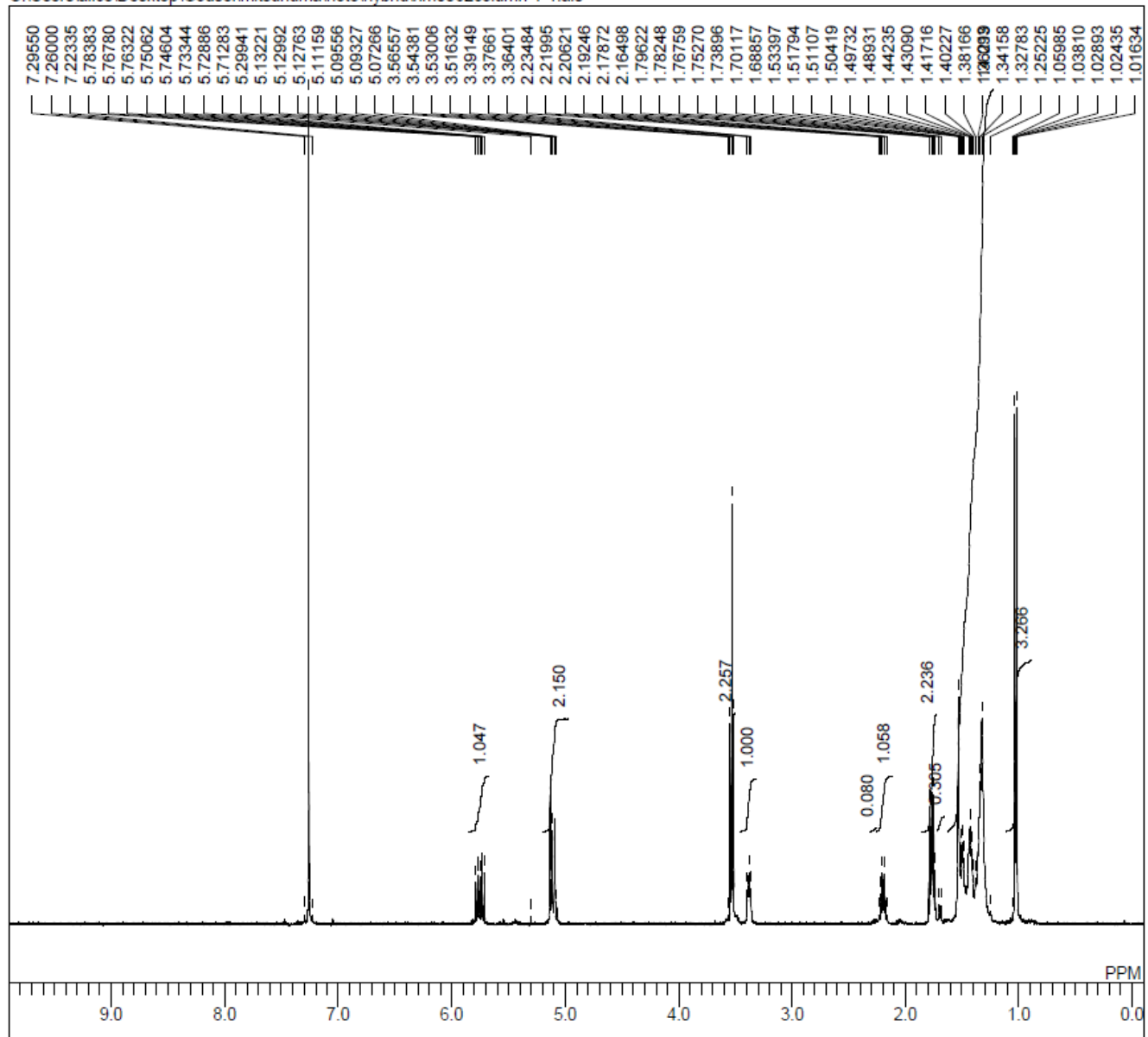


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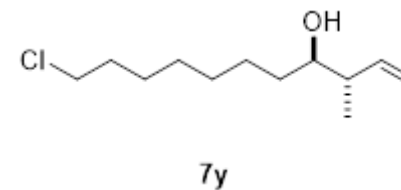


DFILE hm0361tm 13C-1-1.als
COMNT
DATIM 2020-03-03 13:04:06
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 180
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

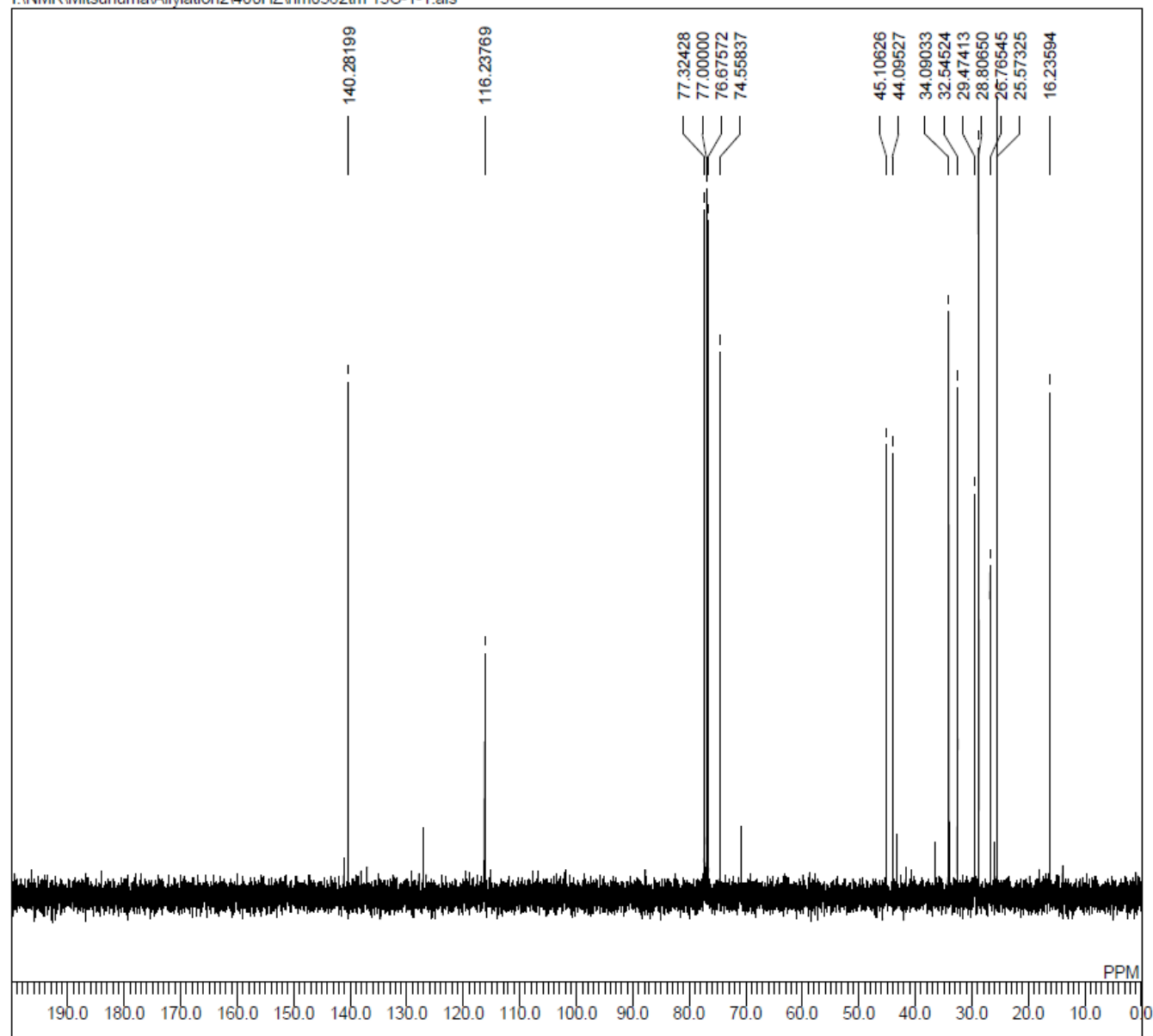
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DFILE hm0362column-1-1.als
COMNT
DATIM 2020-02-20 14:50:02
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 42

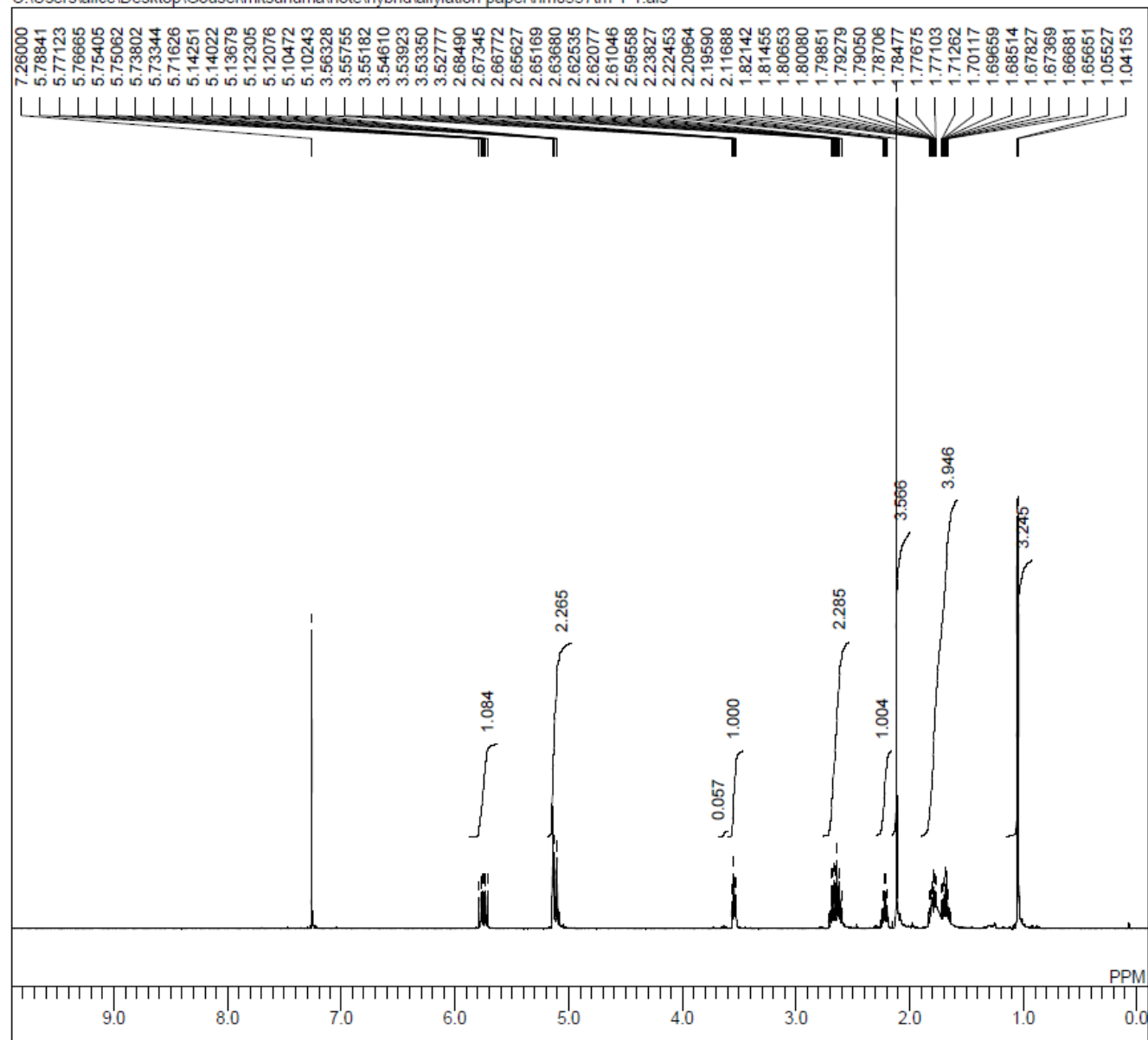


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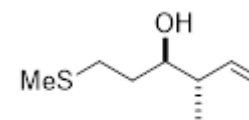


DFILE hm0362tm 13C-1-1.als
COMNT
DATIM 03-03-2020 21:34:38
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 80
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

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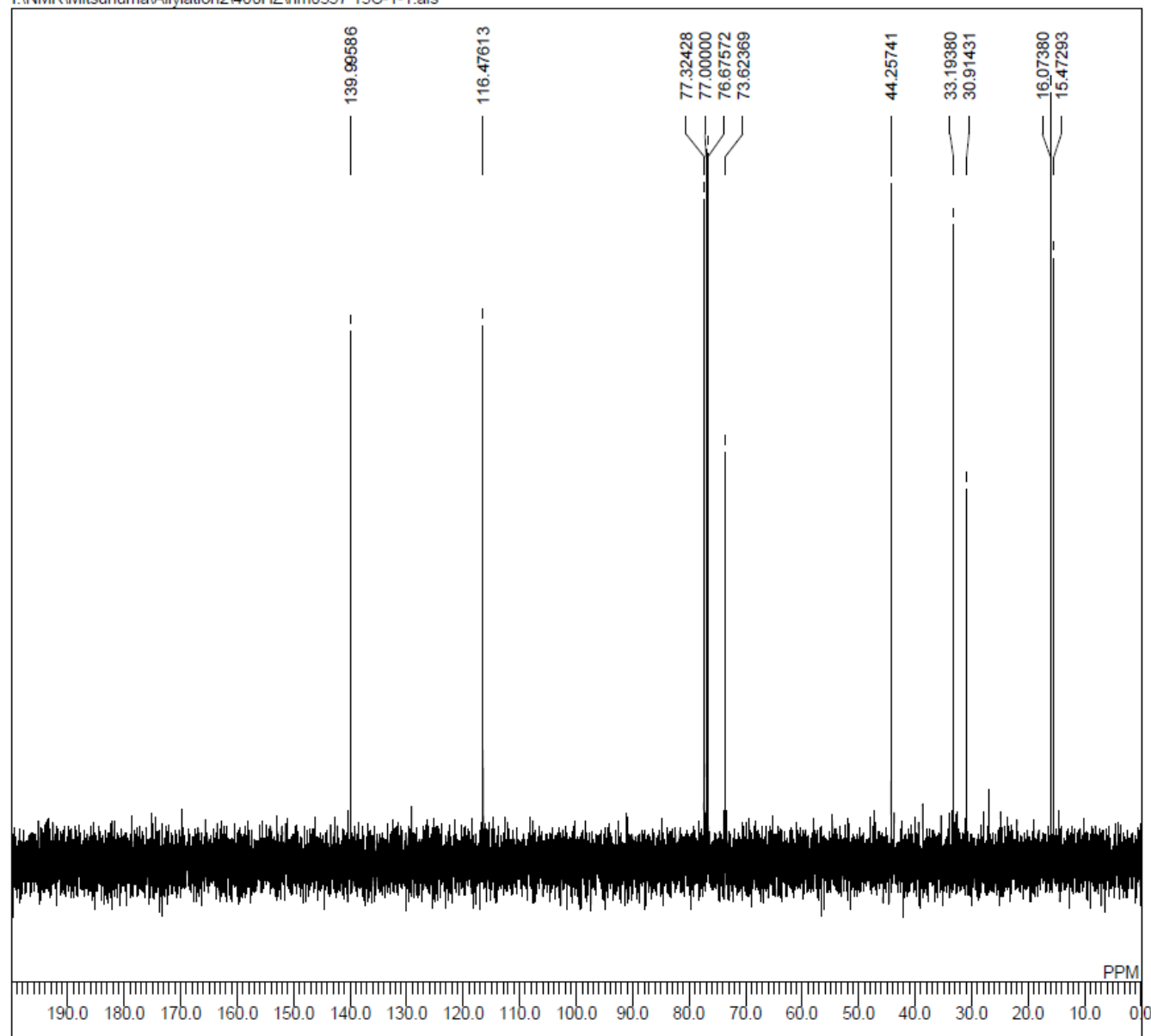


DFILE hm0337m-1-1.als
COMNT
DATIM 2020-02-10 18:12:44
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 42



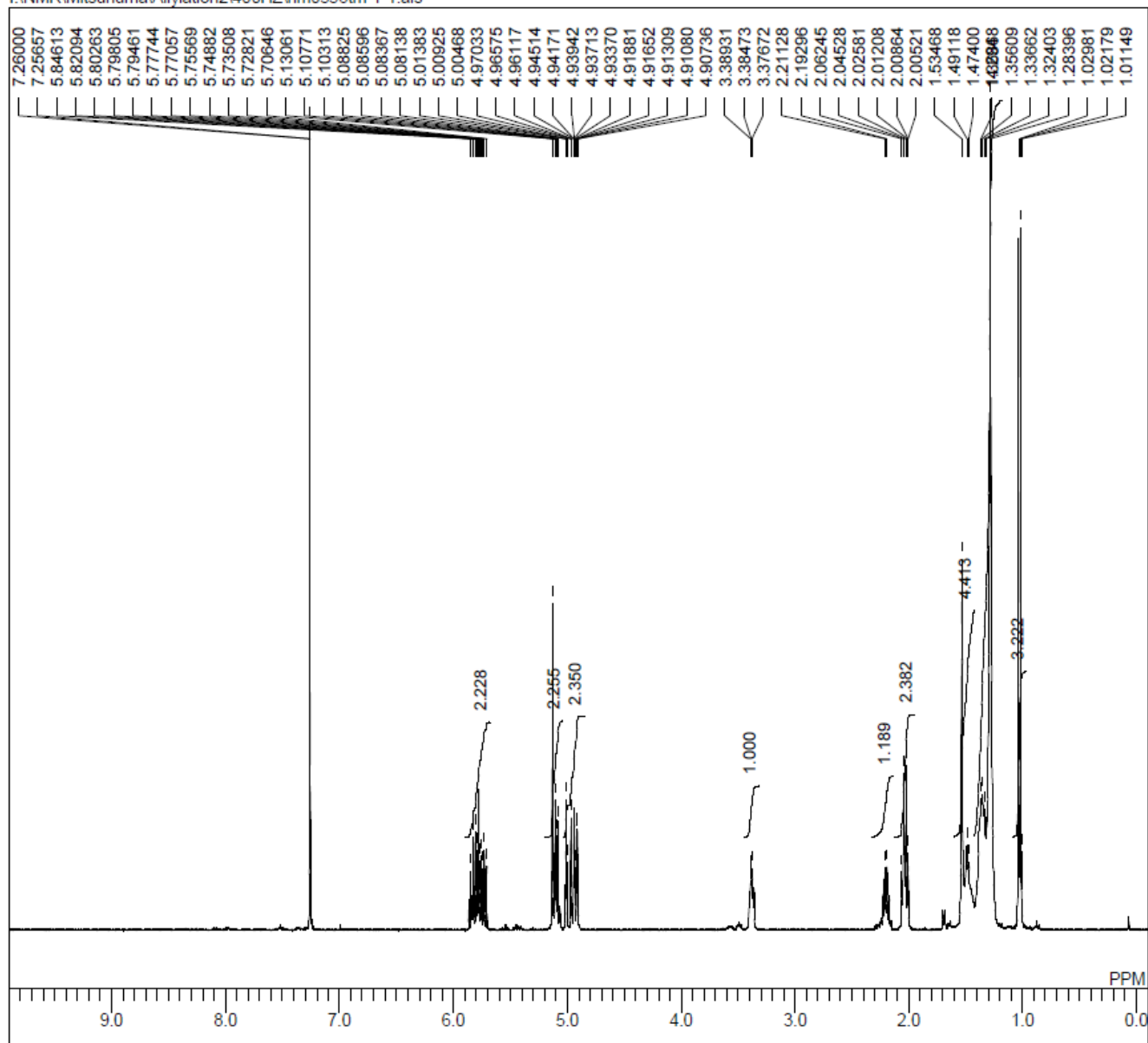
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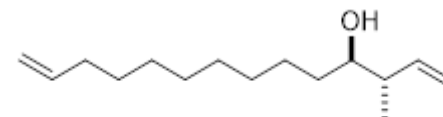


DFILE hm0337 13C-1-1.als
COMNT
DATIM 10-02-2020 20:08:55
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 24
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

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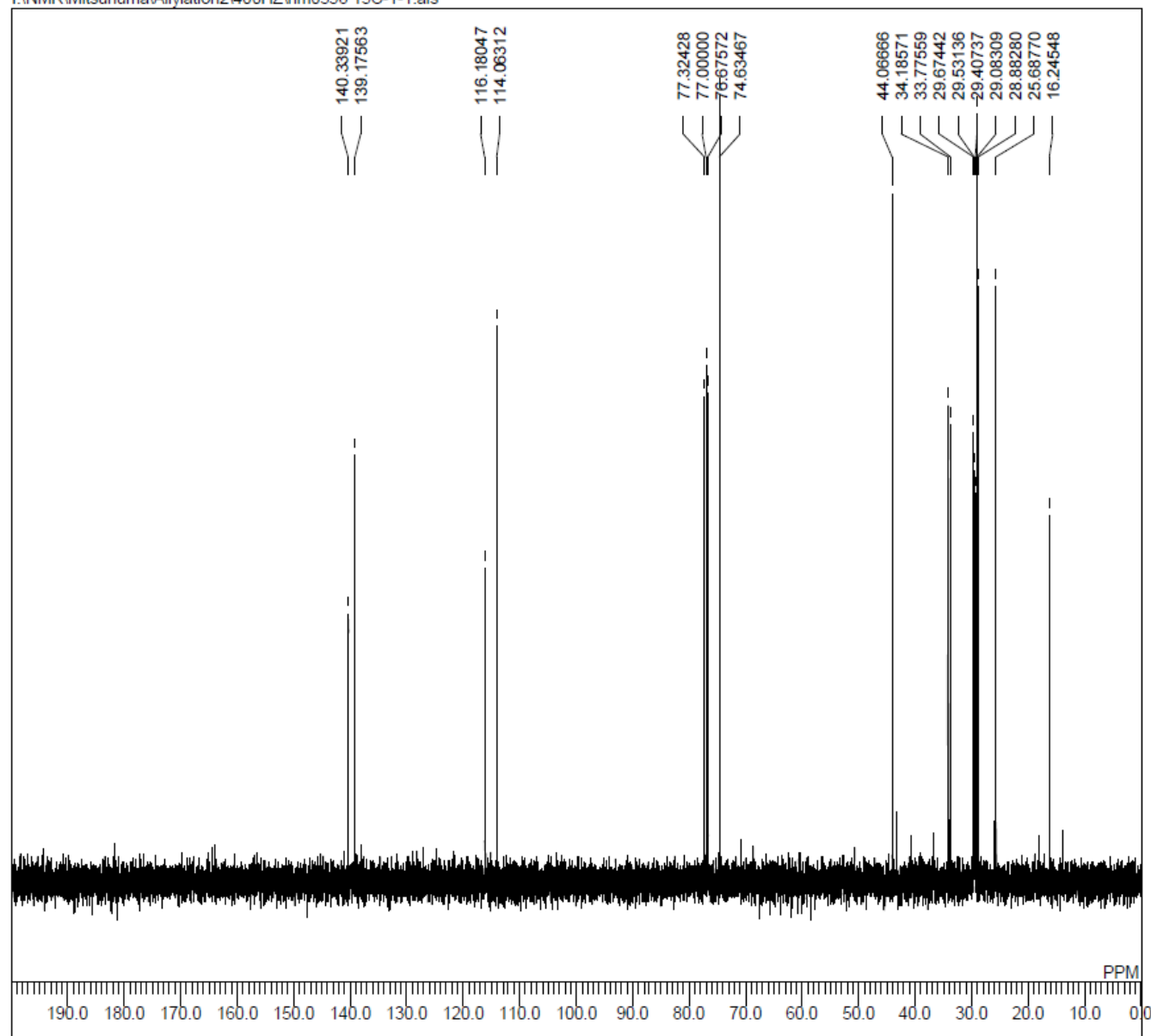


DFILE hm0338tm-1-1.als
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 DATIM 10-02-2020 19:45:48
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 391.78 MHz
 OBSET 8.51 KHz
 OBFIN 3.34 Hz
 POINT 13107
 FREQU 5878.90 Hz
 SCANS 8
 ACQTM 2.2295 sec
 PD 6.0000 sec
 PW1 5.17 usec
 IRNUC 1H
 CTEMP 20.2 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 44



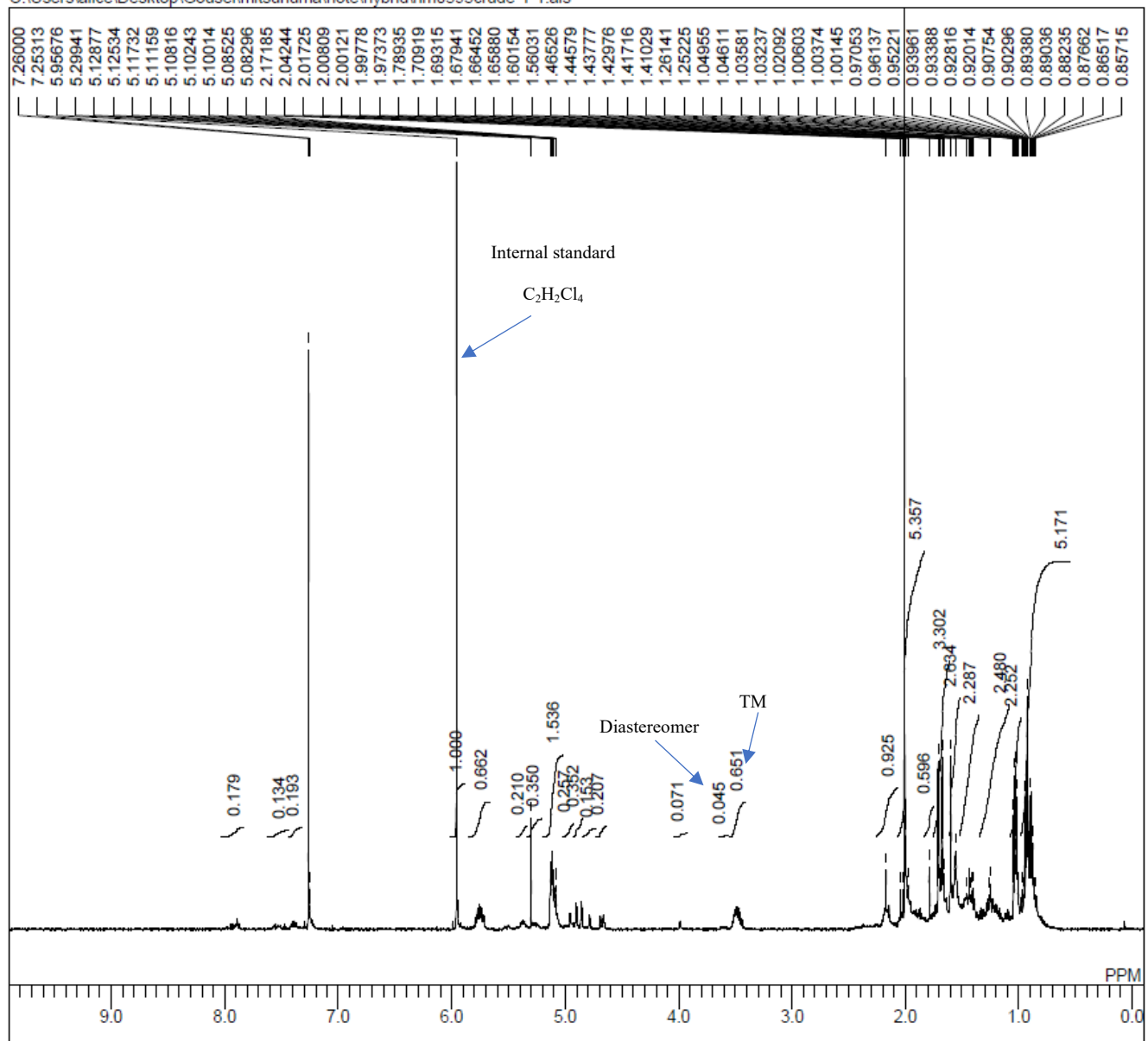
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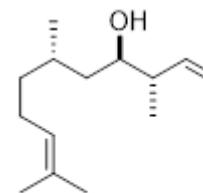


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EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 24
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

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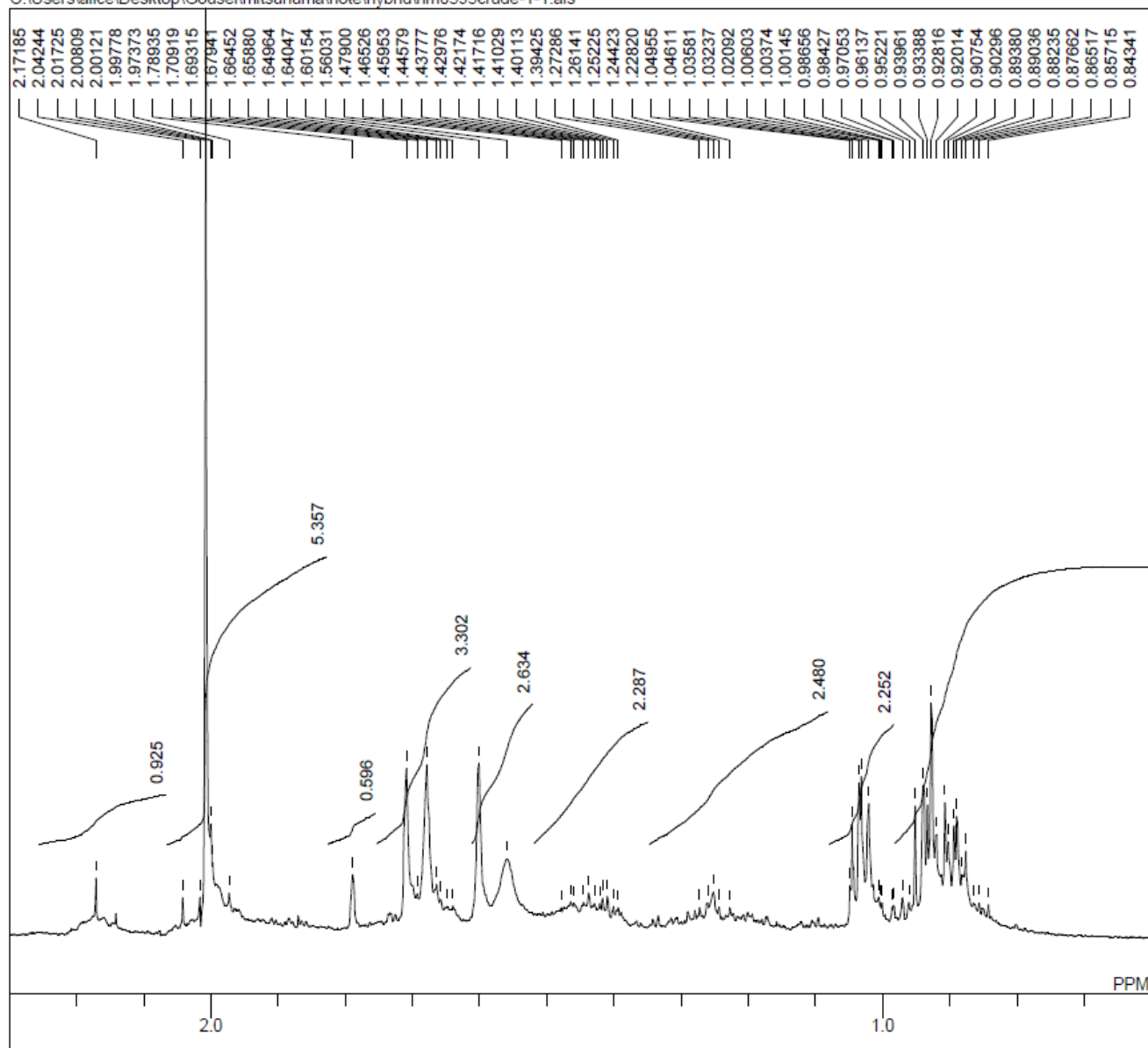


DFILE hm0393crude-1-1.als
 COMNT
 DATIM 2020-03-07 20:09:38
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 13107
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 6.0000 sec
 PW1 5.55 usec
 IRNUC 1H
 CTEMP 21.2 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 42

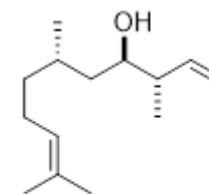


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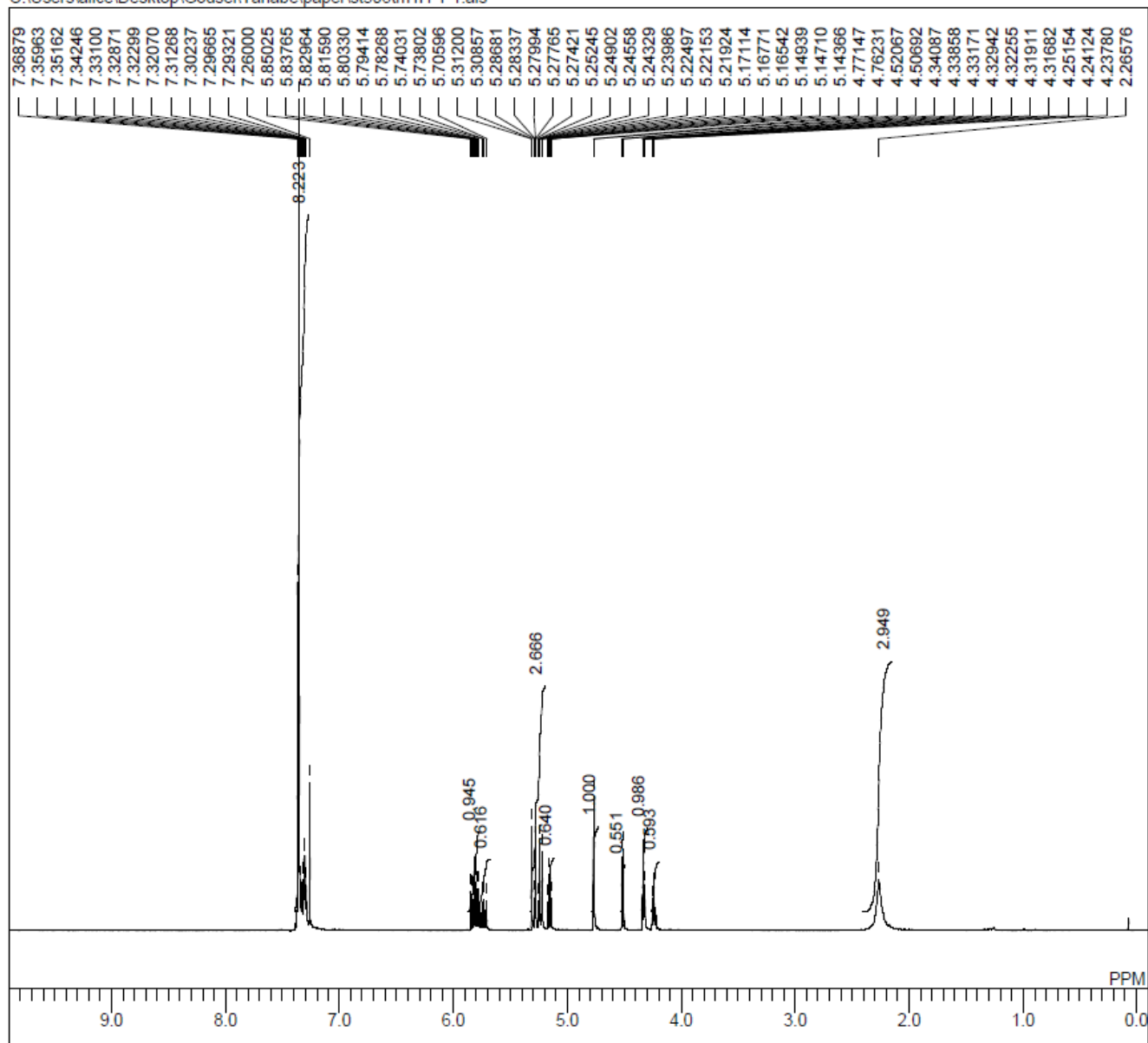


DFILE hm0393crude-1-1.als
COMNT
DATIM 2020-03-07 20:09:38
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EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 42

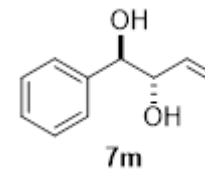


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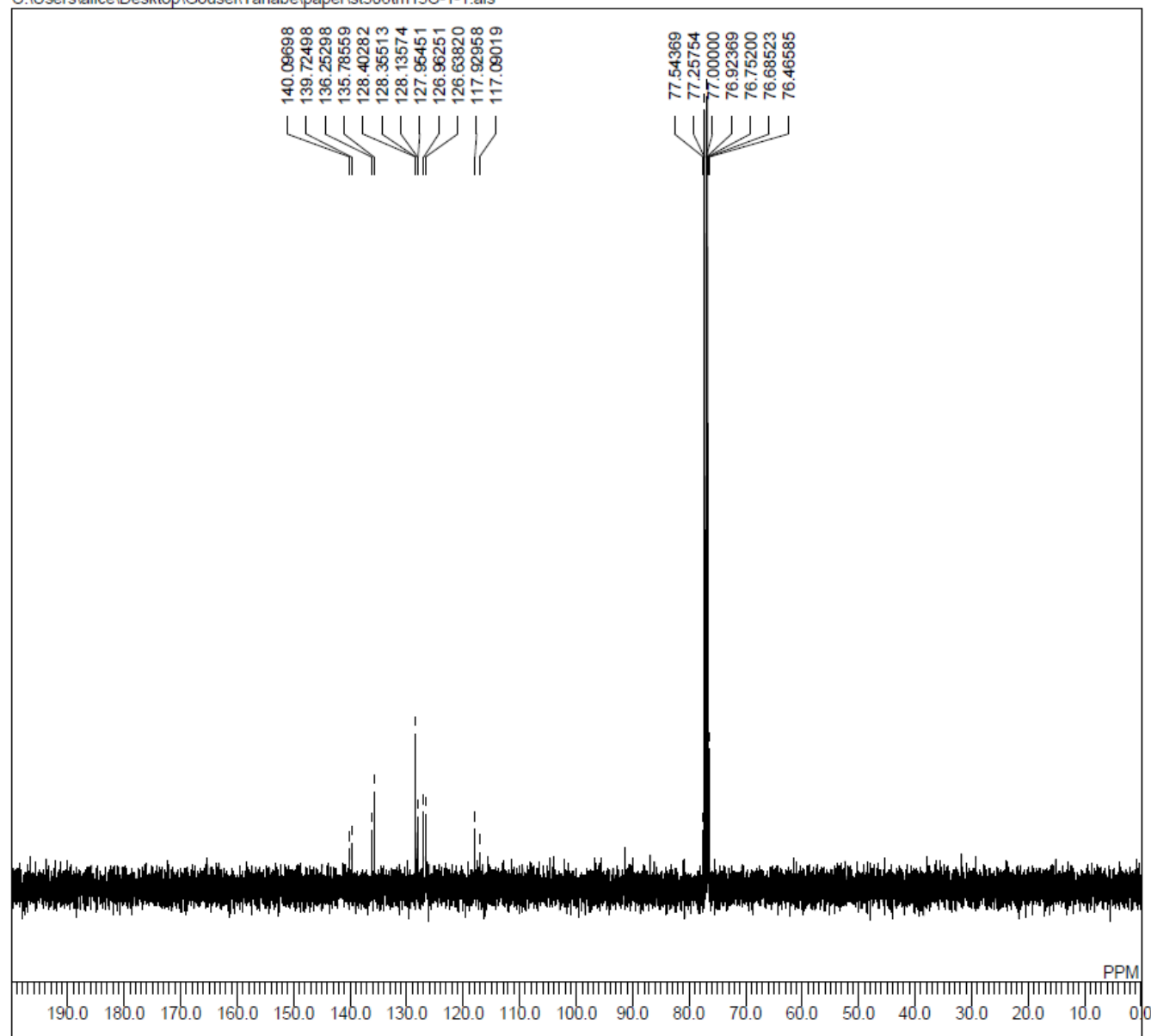
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DFILE st308tm1H-1-1.als
 COMNT
 DATIM 2020-03-03 15:38:13
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 13107
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 6.0000 sec
 PW1 5.55 usec
 IRNUC 1H
 CTEMP 21.4 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 40

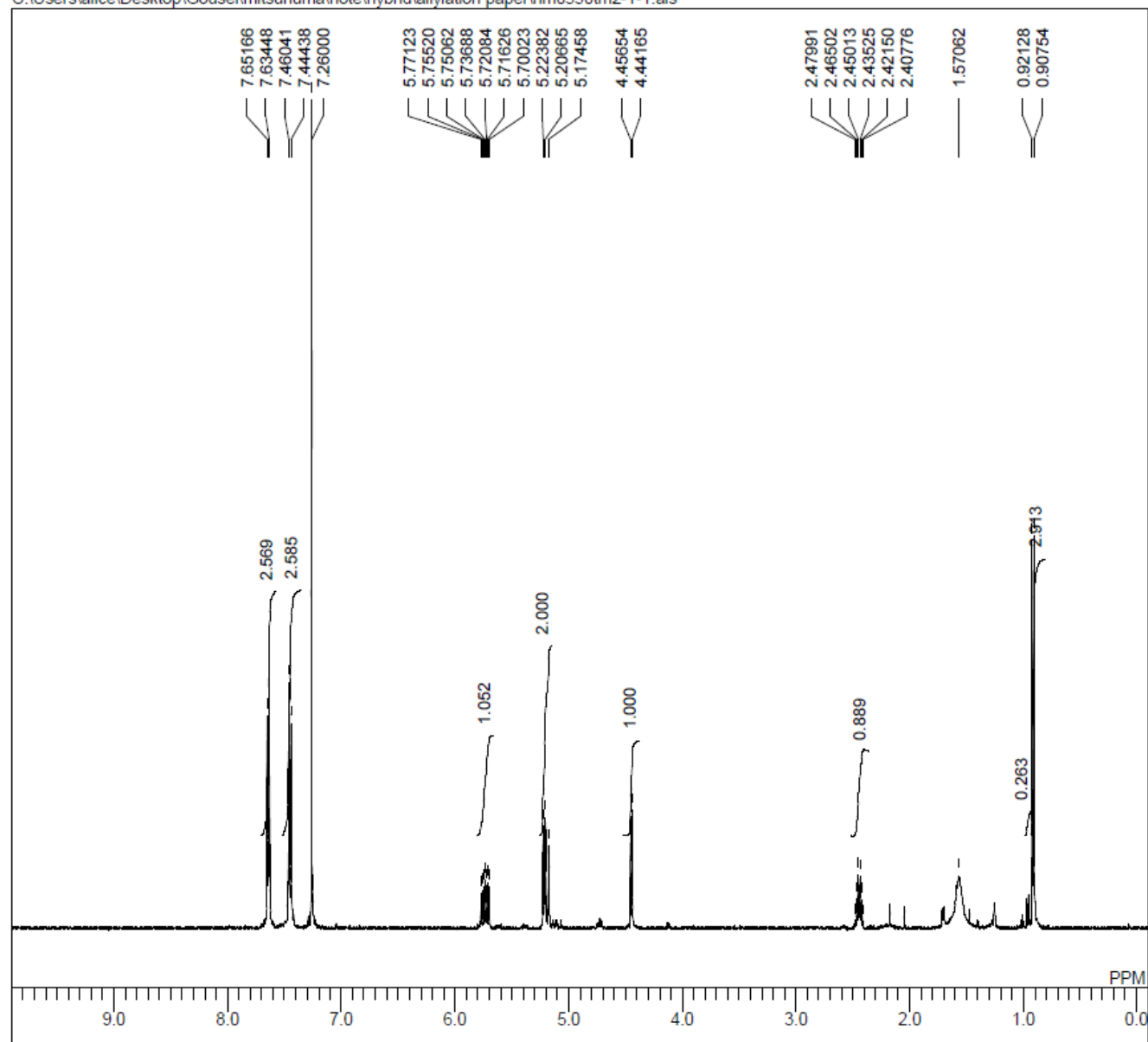


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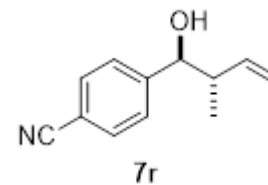


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EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 40
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

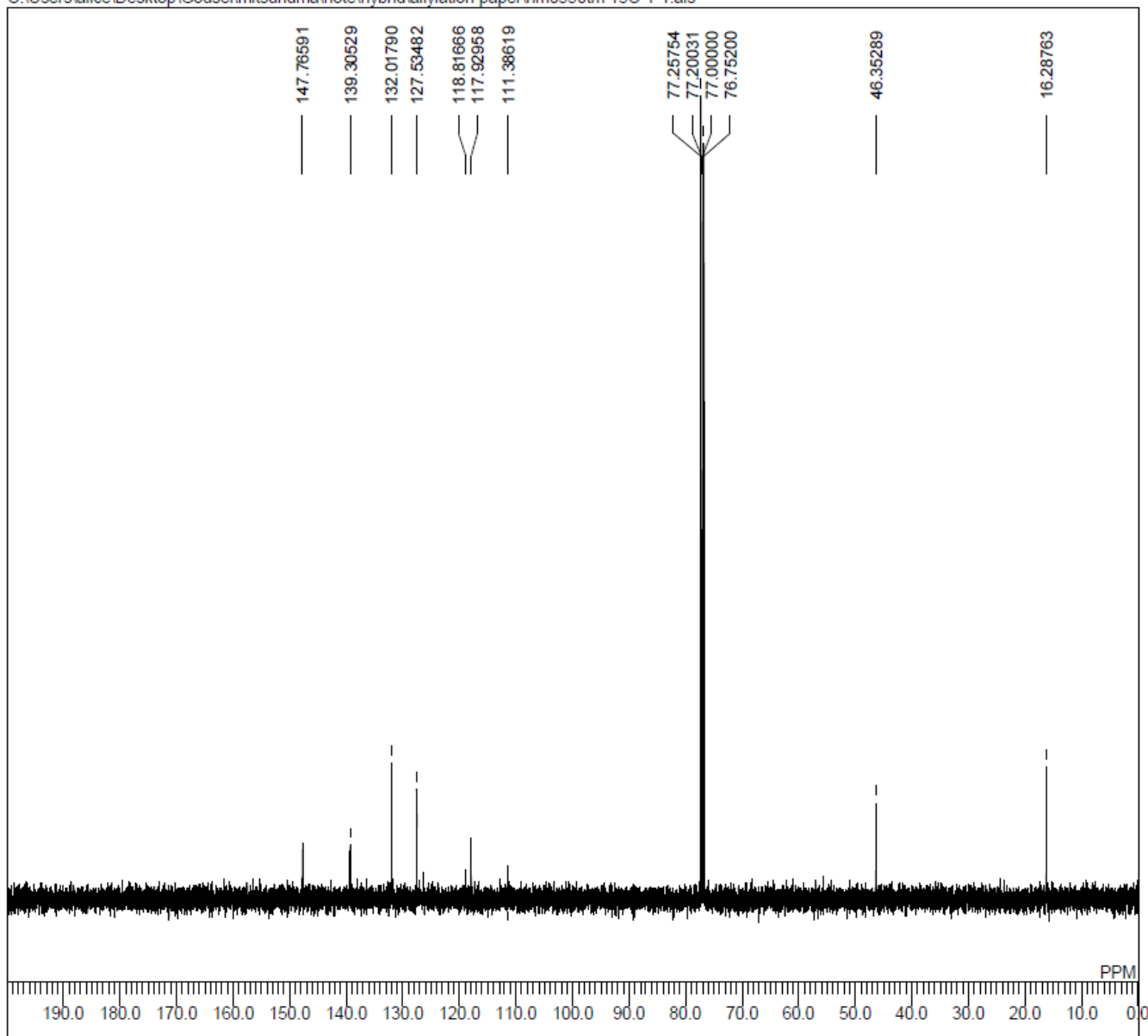
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DFILE hm0350tm2-1-1.als
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EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 44

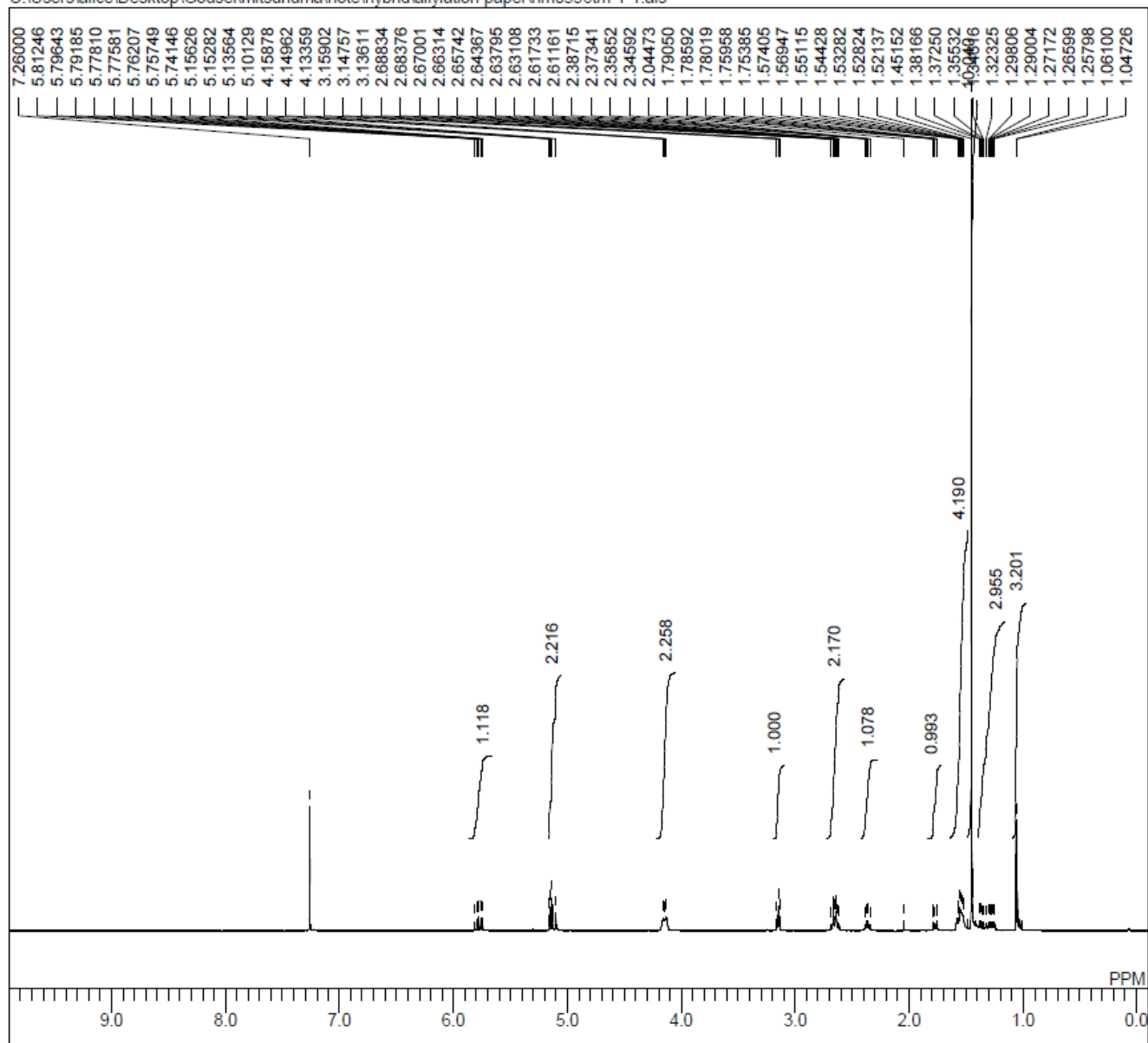


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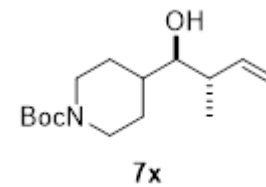


DFILE hm0350tm 13C-1-1.als
COMNT
DATIM 2020-03-27 20:55:33
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 120
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

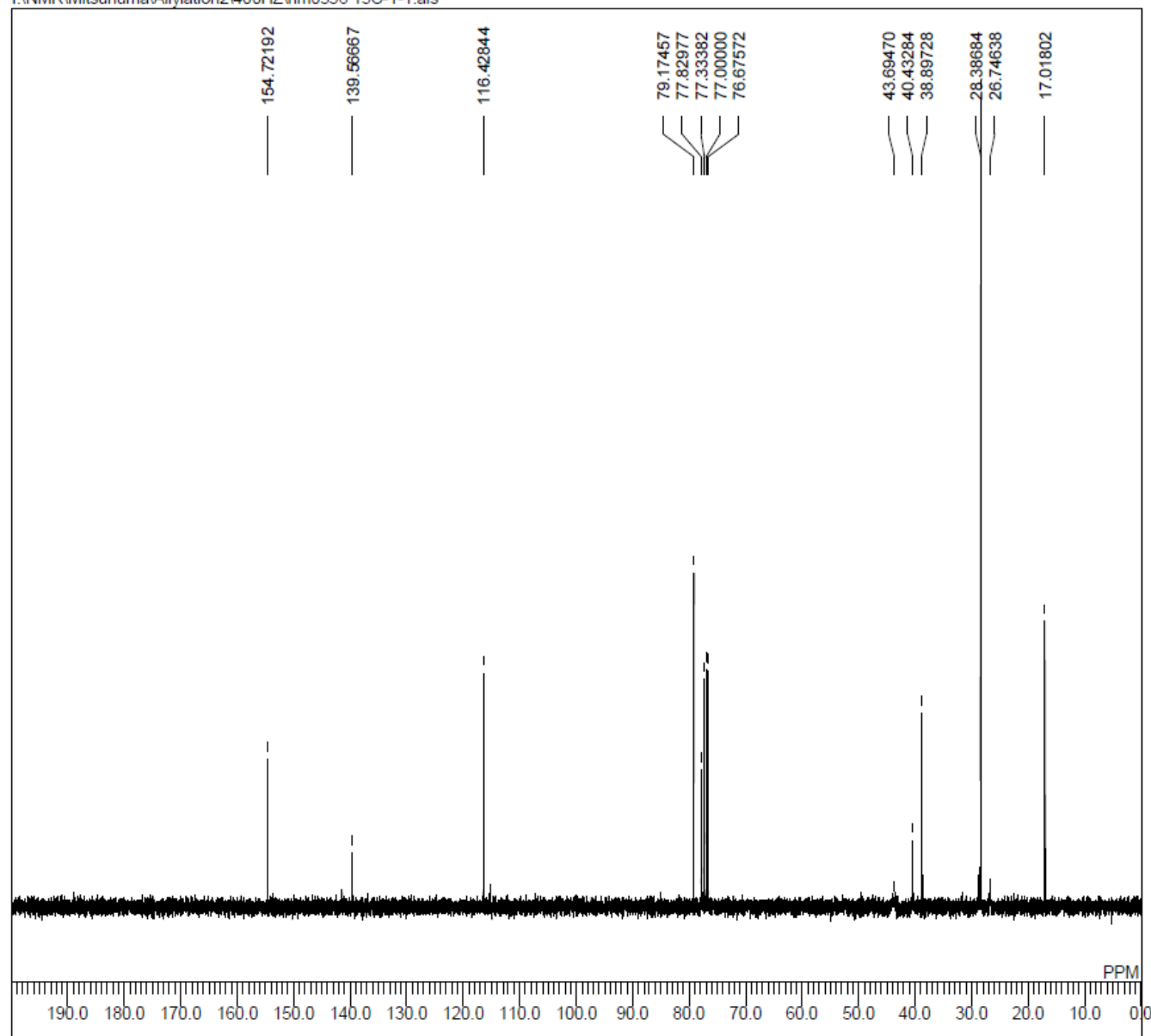
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DFILE hm0336tm-1-1.als
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 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 13107
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 6.0000 sec
 PW1 5.55 usec
 IRNUC 1H
 CTEMP 21.1 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 42

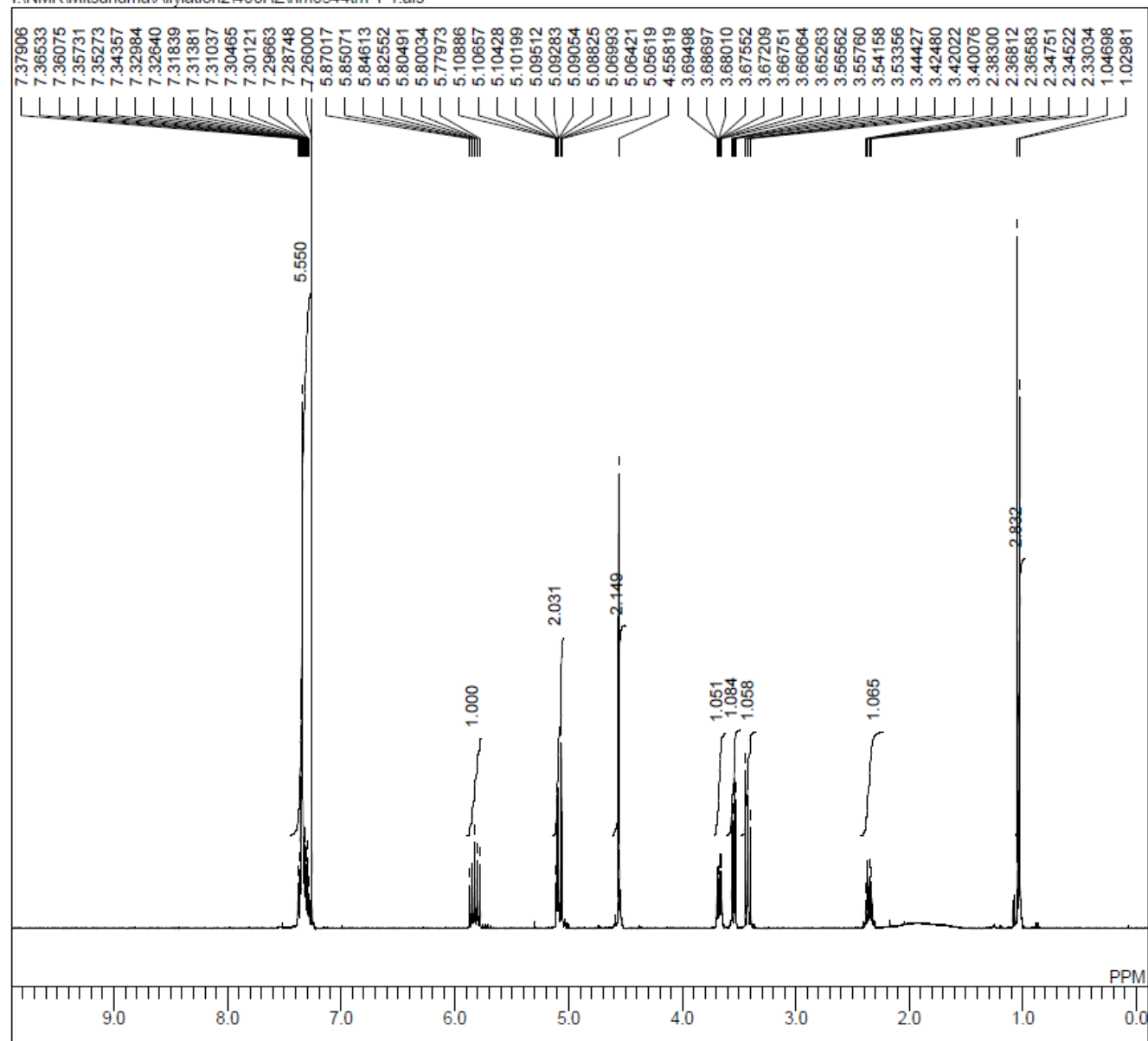


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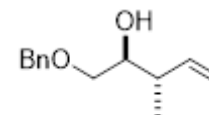


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COMNT
DATIM 10-02-2020 20:02:22
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 40
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

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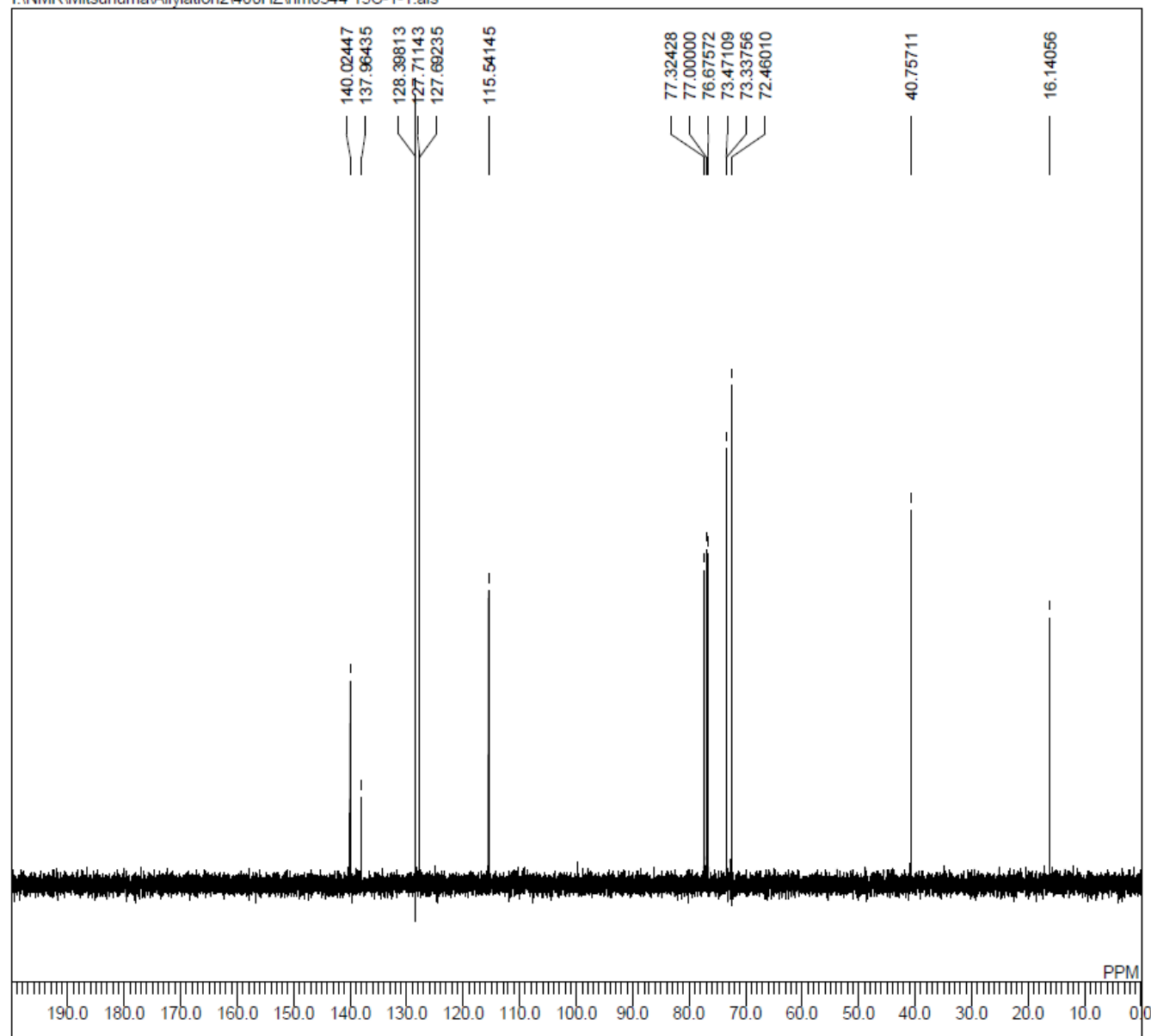


DFILE hm0344tm-1-1.als
 COMNT
 DATIM 13-02-2020 17:27:30
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 391.78 MHz
 OBSET 8.51 KHz
 OBFIN 3.34 Hz
 POINT 13107
 FREQU 5878.90 Hz
 SCANS 8
 ACQTM 2.2295 sec
 PD 6.0000 sec
 PW1 5.17 usec
 IRNUC 1H
 CTEMP 20.3 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 48



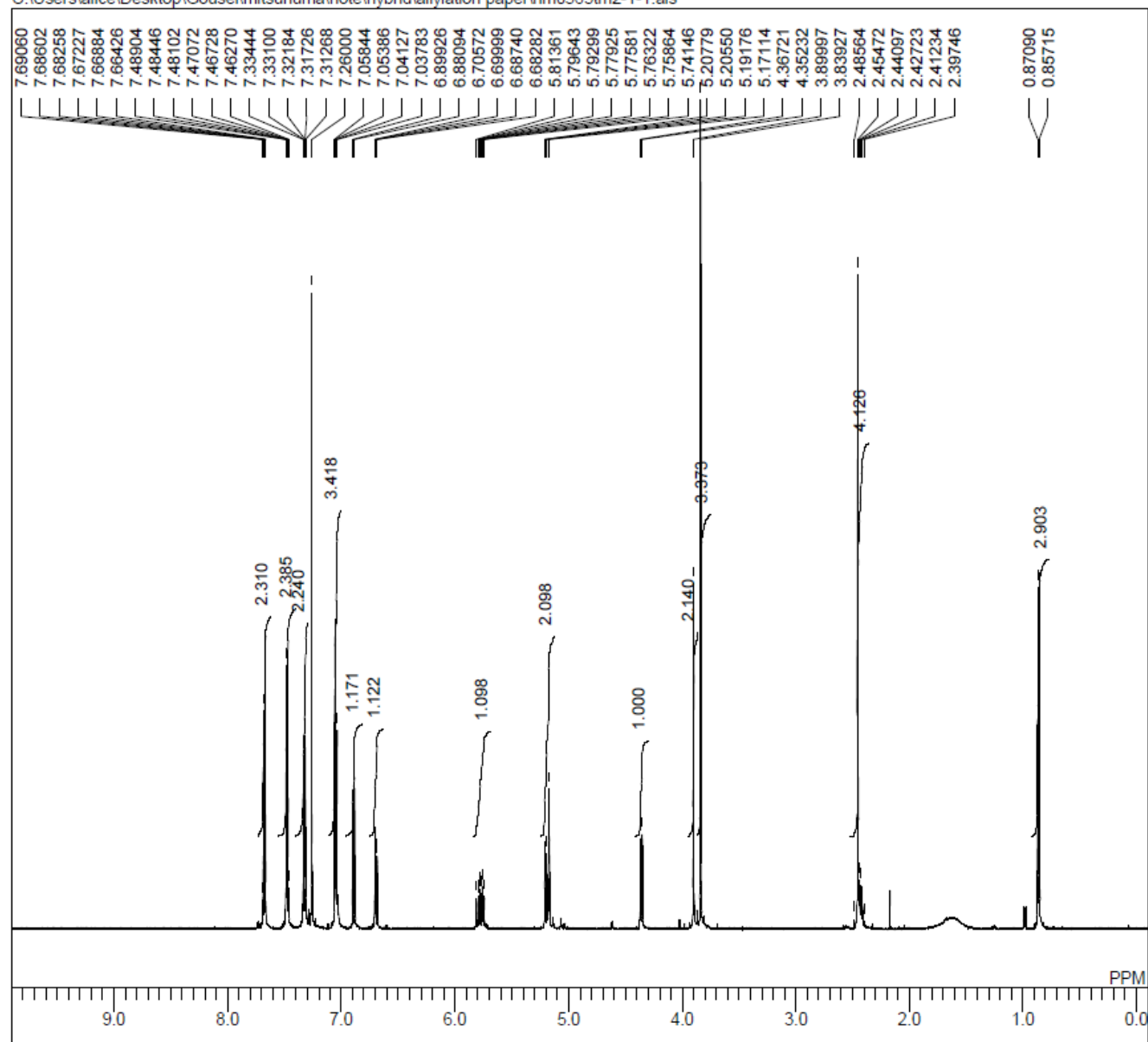
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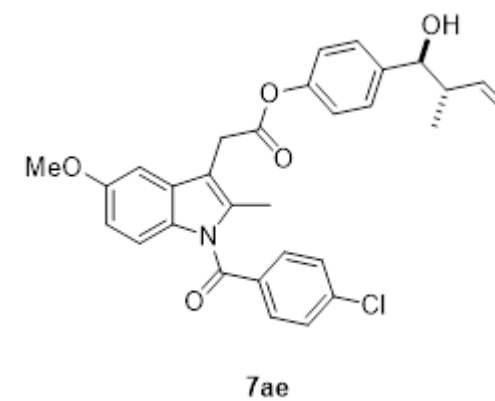


DFILE hm0344 13C-1-1.als
COMNT
DATIM 13-02-2020 19:27:23
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 41
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

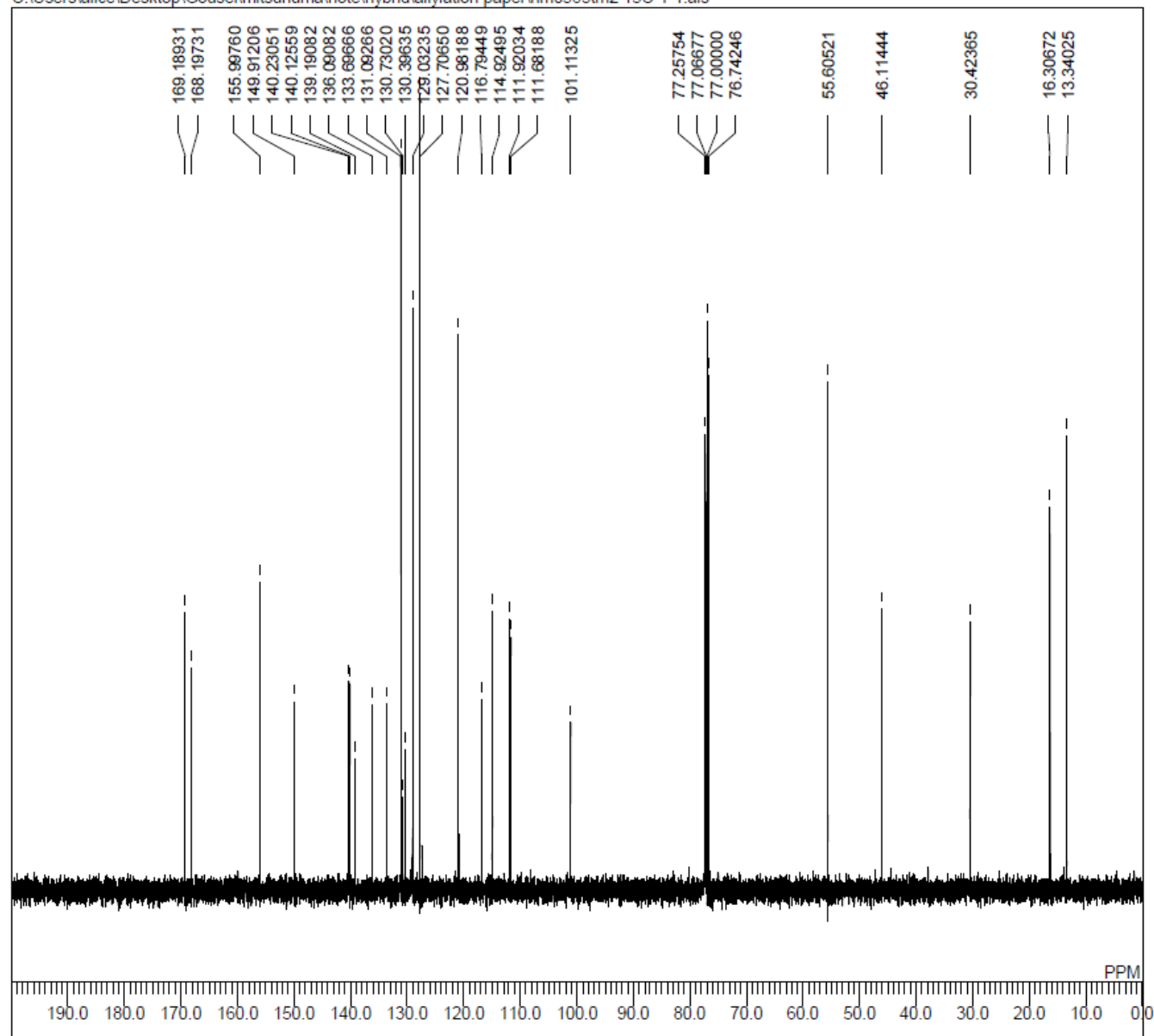
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DFILE hm0363tm2-1-1.als
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 OBNUC 1H
 EXMOD proton.jpg
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 13107
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 6.0000 sec
 PW1 5.55 usec
 IRNUC 1H
 CTEMP 21.4 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 42

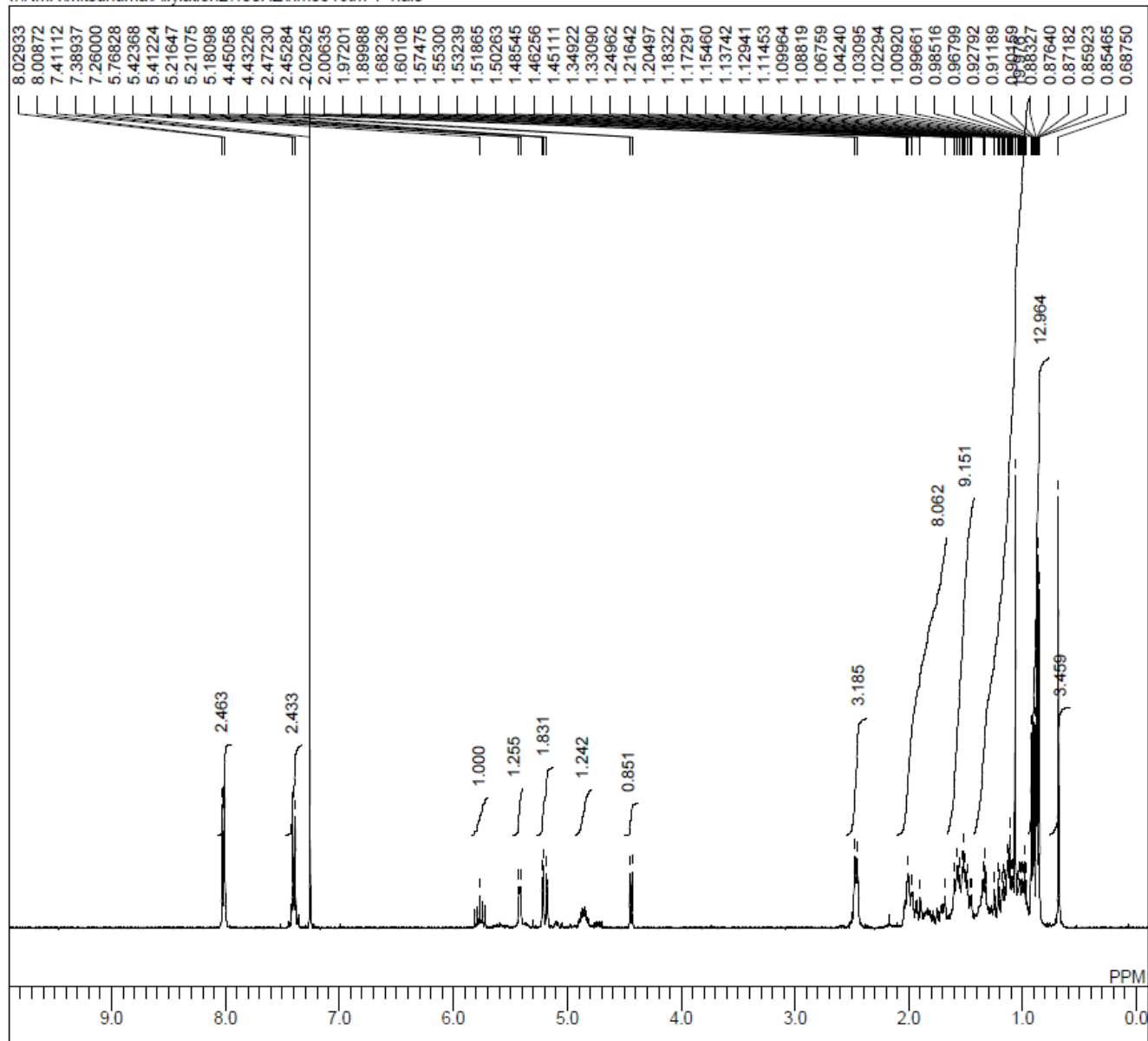


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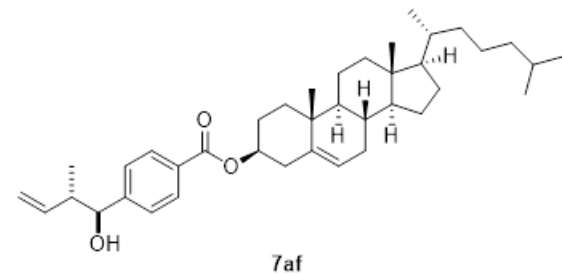


DFILE hm0363tm2 13C-1-1.als
COMNT
DATIM 2020-03-03 19:04:42
OBNUC 13C
EXMOD carbon.jpg
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 40
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

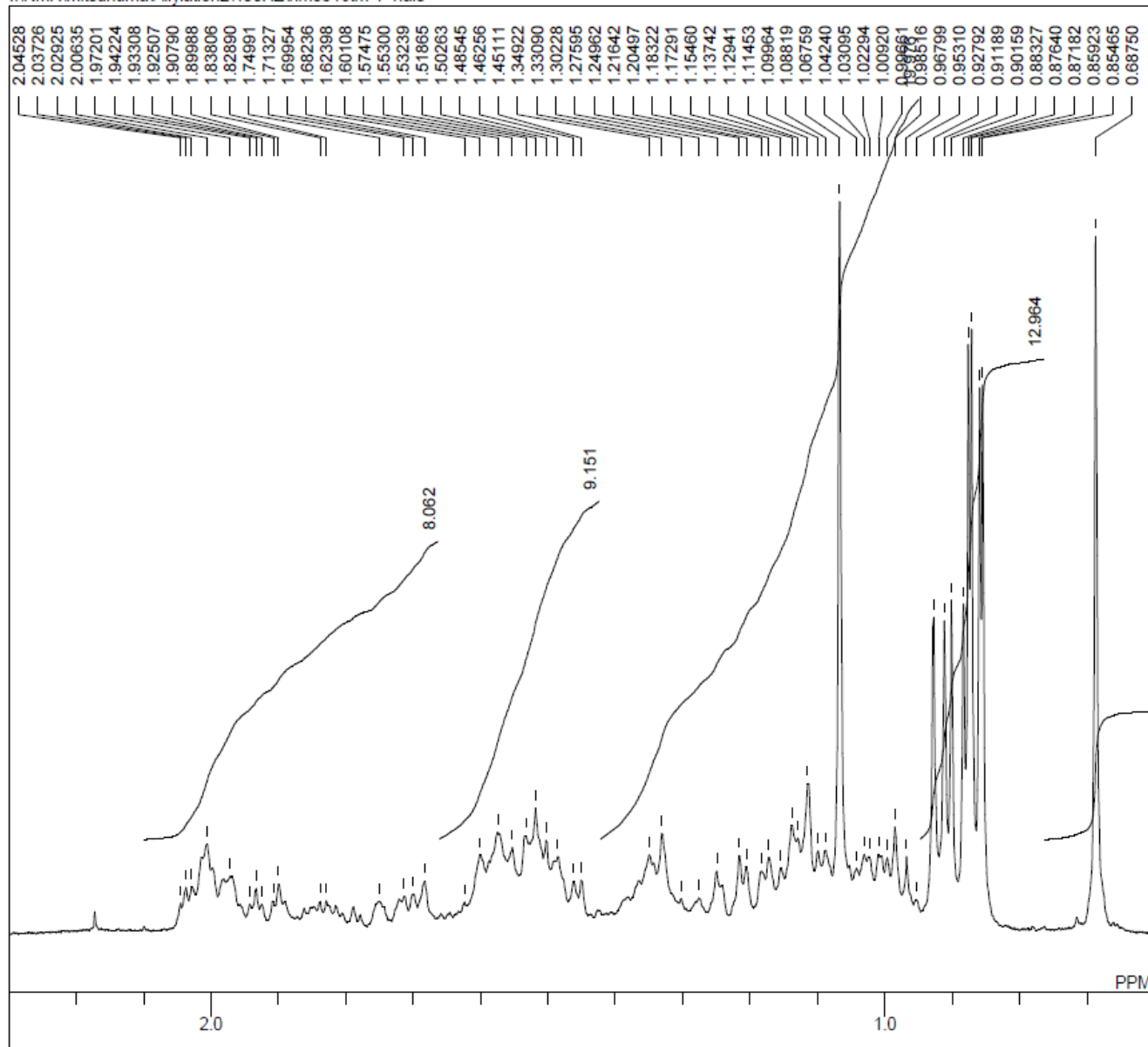
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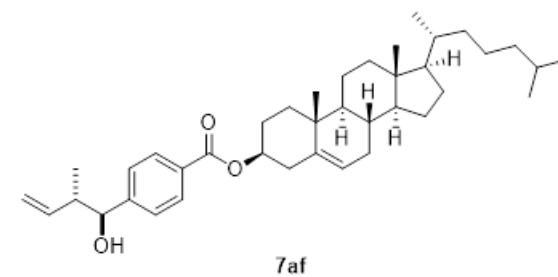
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 COMNT
 DATIM 13-02-2020 17:31:19
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 391.78 MHz
 OBSET 8.51 KHz
 OBFIN 3.34 Hz
 POINT 13107
 FREQU 5878.90 Hz
 SCANS 8
 ACQTM 2.2295 sec
 PD 6.0000 sec
 PW1 5.17 usec
 IRNUC 1H
 CTEMP 20.3 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 44



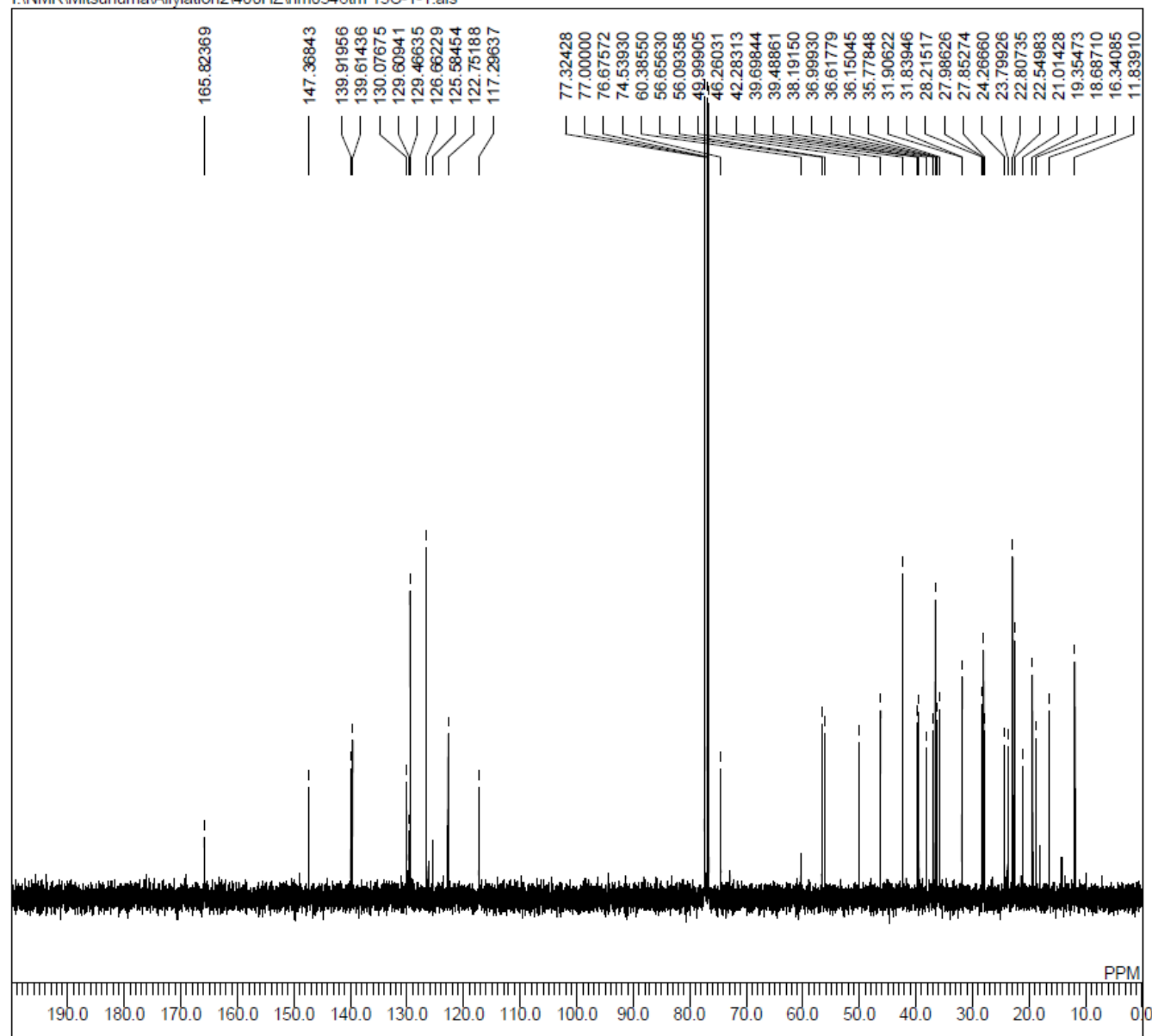
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DFILE hm0346tm-1-1.als
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 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 391.78 MHz
 OBSET 8.51 KHz
 OBFIN 3.34 Hz
 POINT 13107
 FREQU 5878.90 Hz
 SCANS 8
 ACQTM 2.2295 sec
 PD 6.0000 sec
 PW1 5.17 usec
 IRNUC 1H
 CTEMP 20.3 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 44

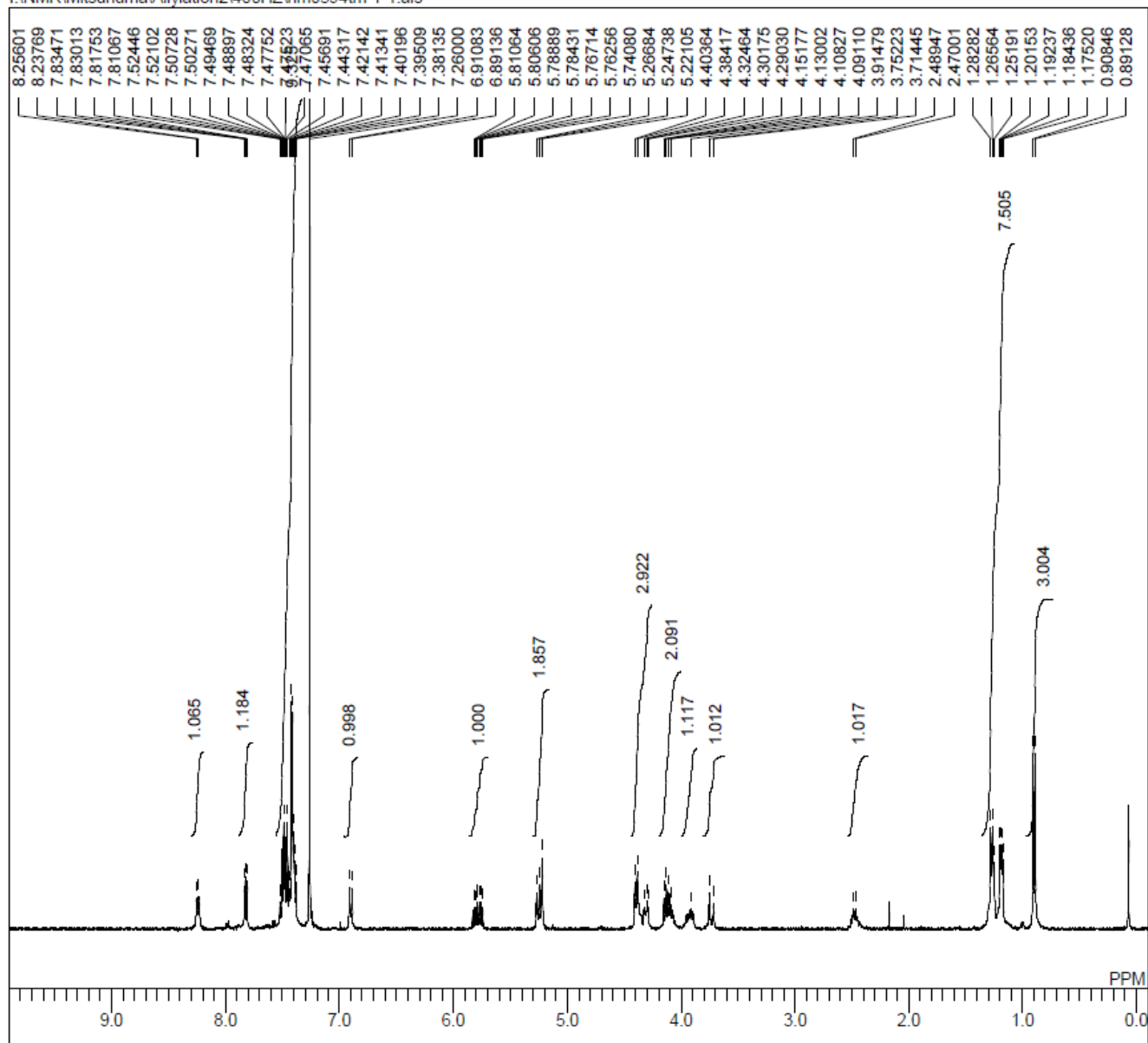


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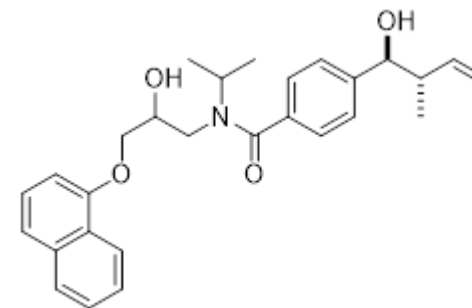


DFILE hm0346tm 13C-1-1.als
COMNT
DATIM 26-03-2020 16:27:42
OBNUC 13C
EXMOD carbon.jpg
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 144
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

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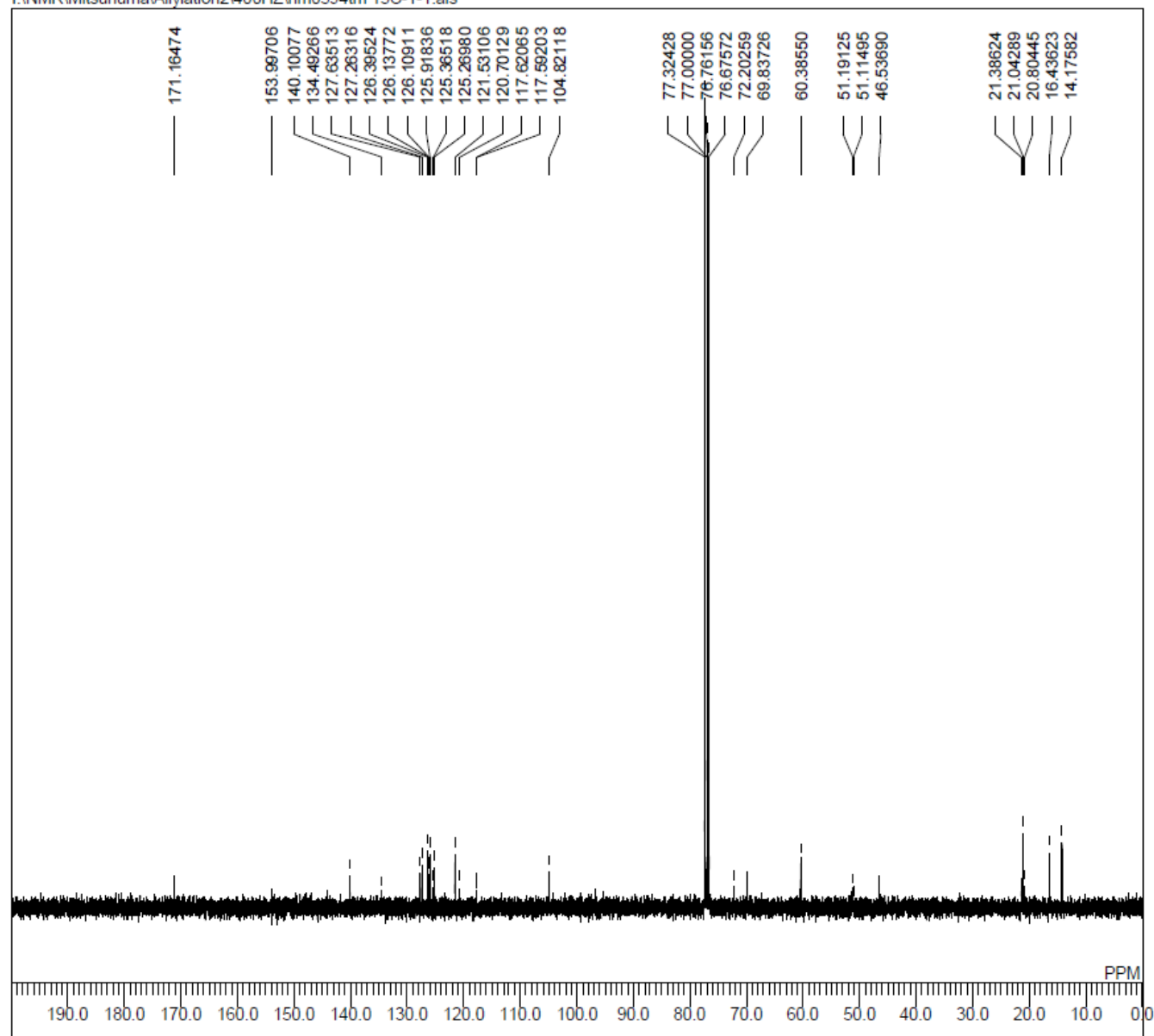


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 OBNUC 1H
 EXMOD proton.jpg
 OBFRQ 391.78 MHz
 OBSET 8.51 KHz
 OBFIN 3.34 Hz
 POINT 13107
 FREQU 5878.90 Hz
 SCANS 8
 ACQTM 2.2295 sec
 PD 6.0000 sec
 PW1 5.17 usec
 IRNUC 1H
 CTEMP 20.4 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 50



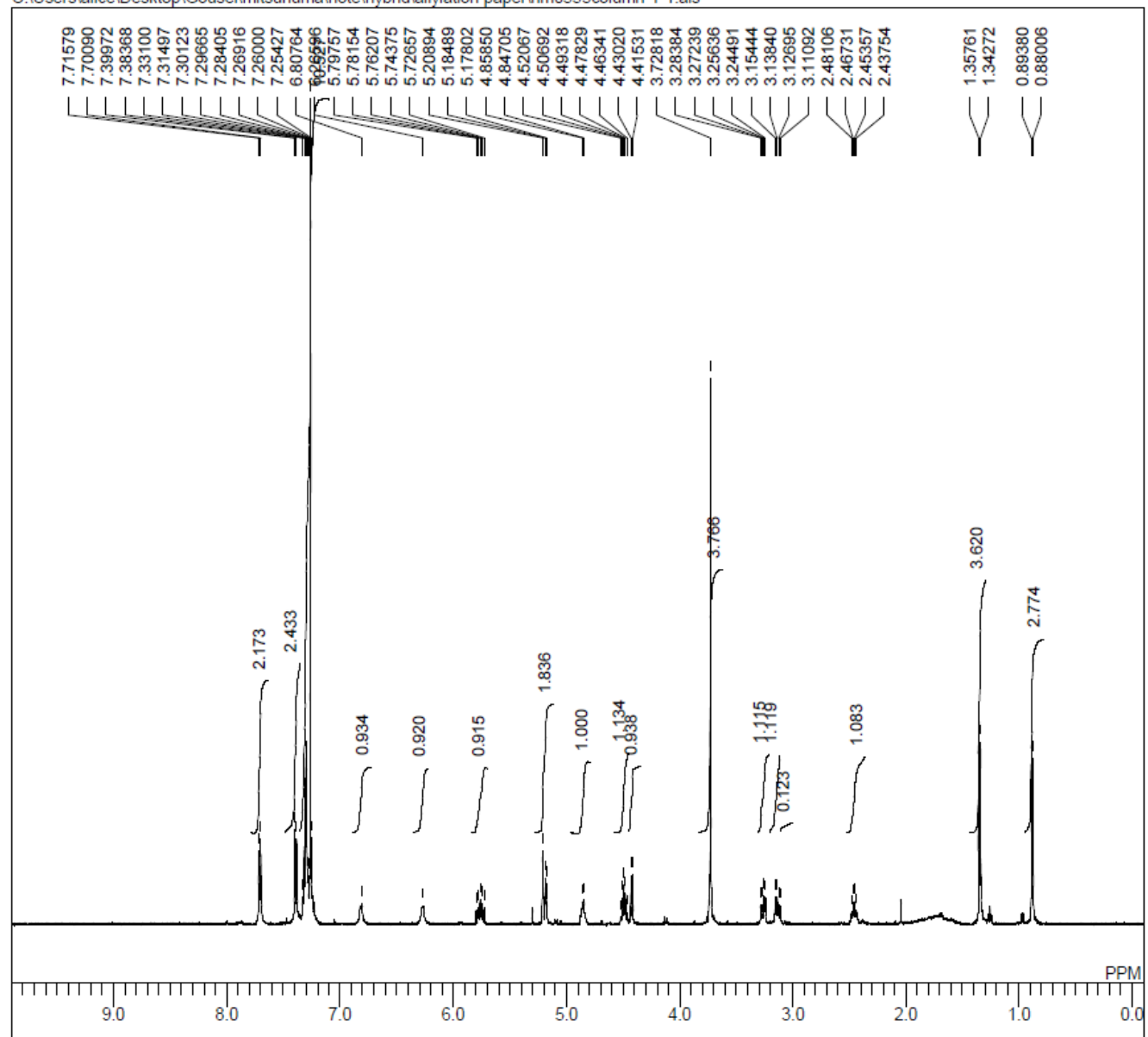
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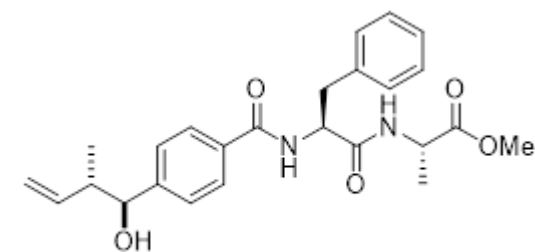


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COMNT
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OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 280
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

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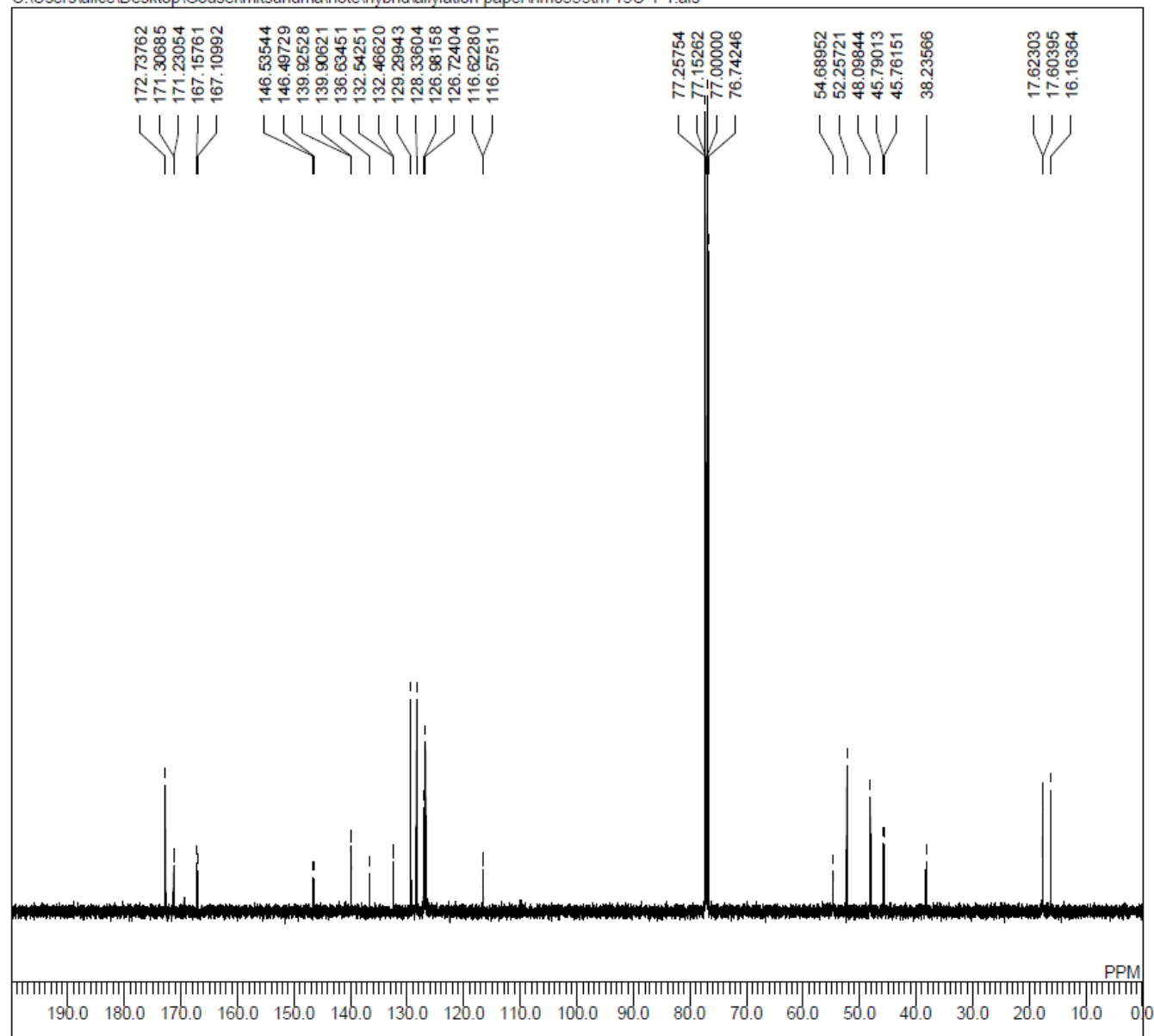


DFILE hm0353column-1-1.als
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 DATIM 2020-02-14 14:39:20
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 13107
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 6.0000 sec
 PW1 5.55 usec
 IRNUC 1H
 CTEMP 21.4 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 44



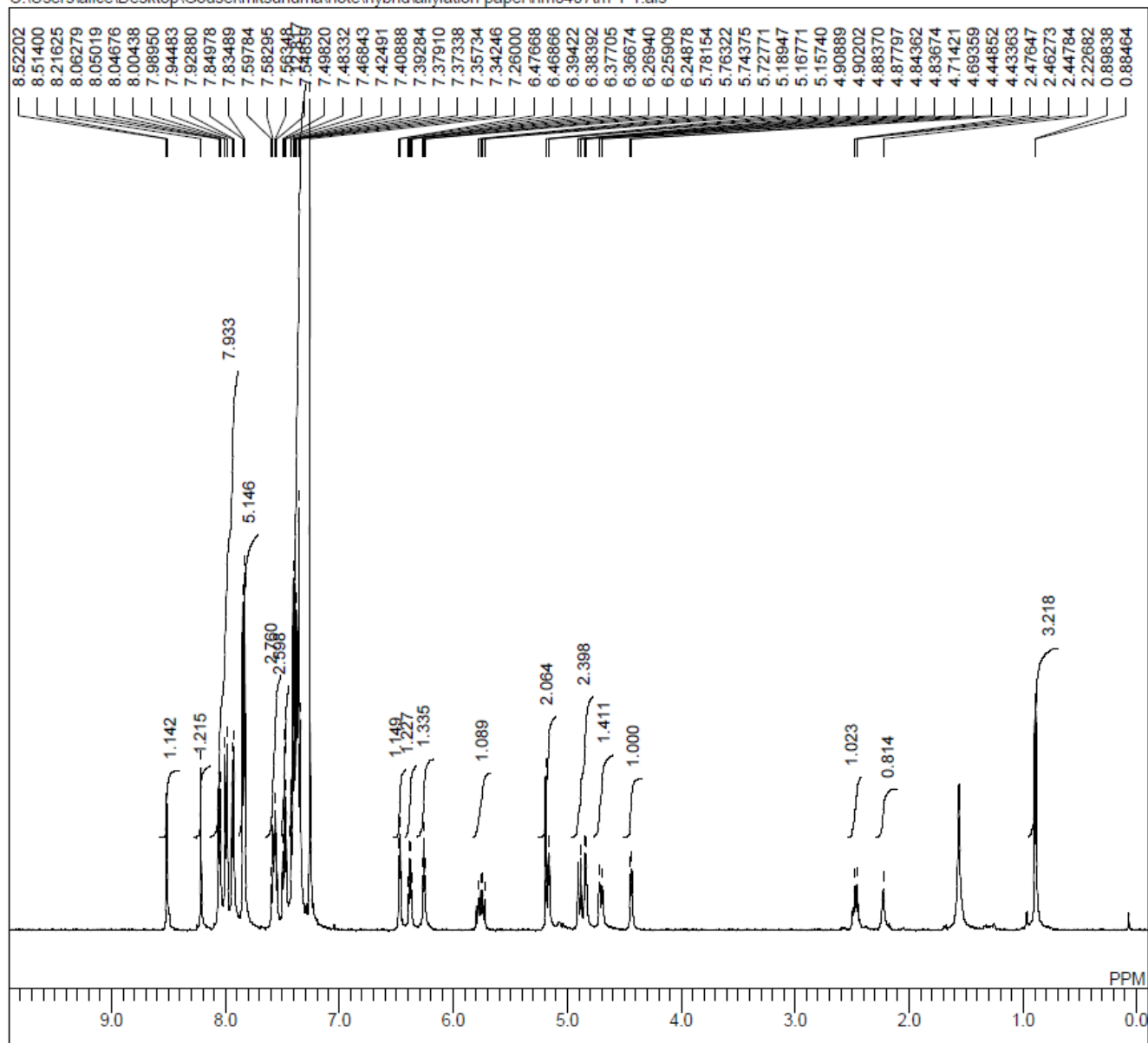
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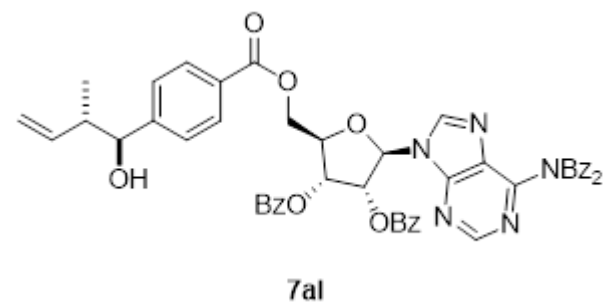


DFILE hm0353tm 13C-1-1.als
COMNT
DATIM 2020-03-03 20:15:59
OBNUC 13C
EXMOD carbon.jpg
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 112
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

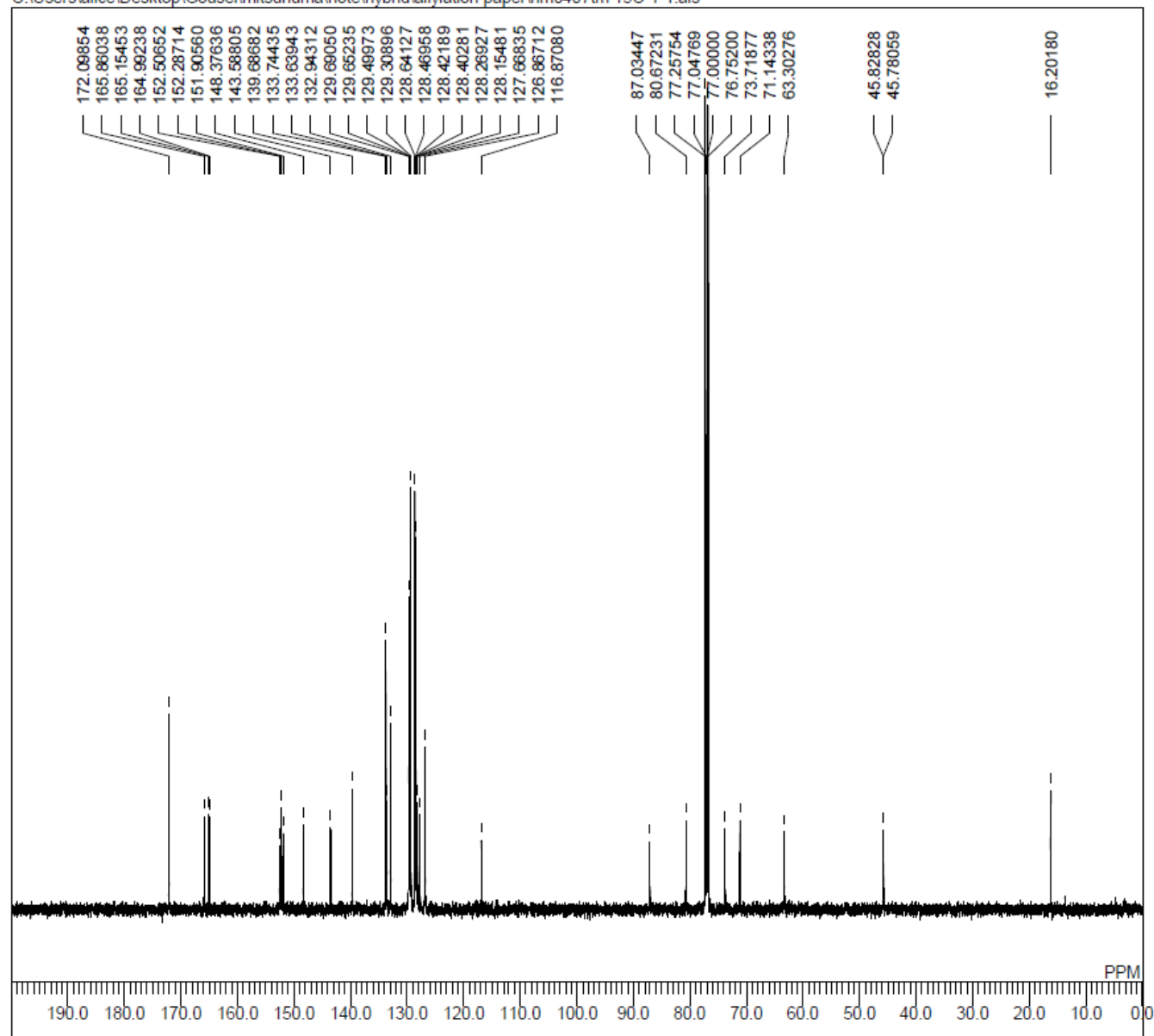
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DFILE hm0407tm-1-1.als
 COMNT
 DATIM 2020-03-23 20:23:27
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 13107
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 6.0000 sec
 PW1 5.55 usec
 IRNUC 1H
 CTEMP 21.3 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 1.20 Hz
 RGAIN 42

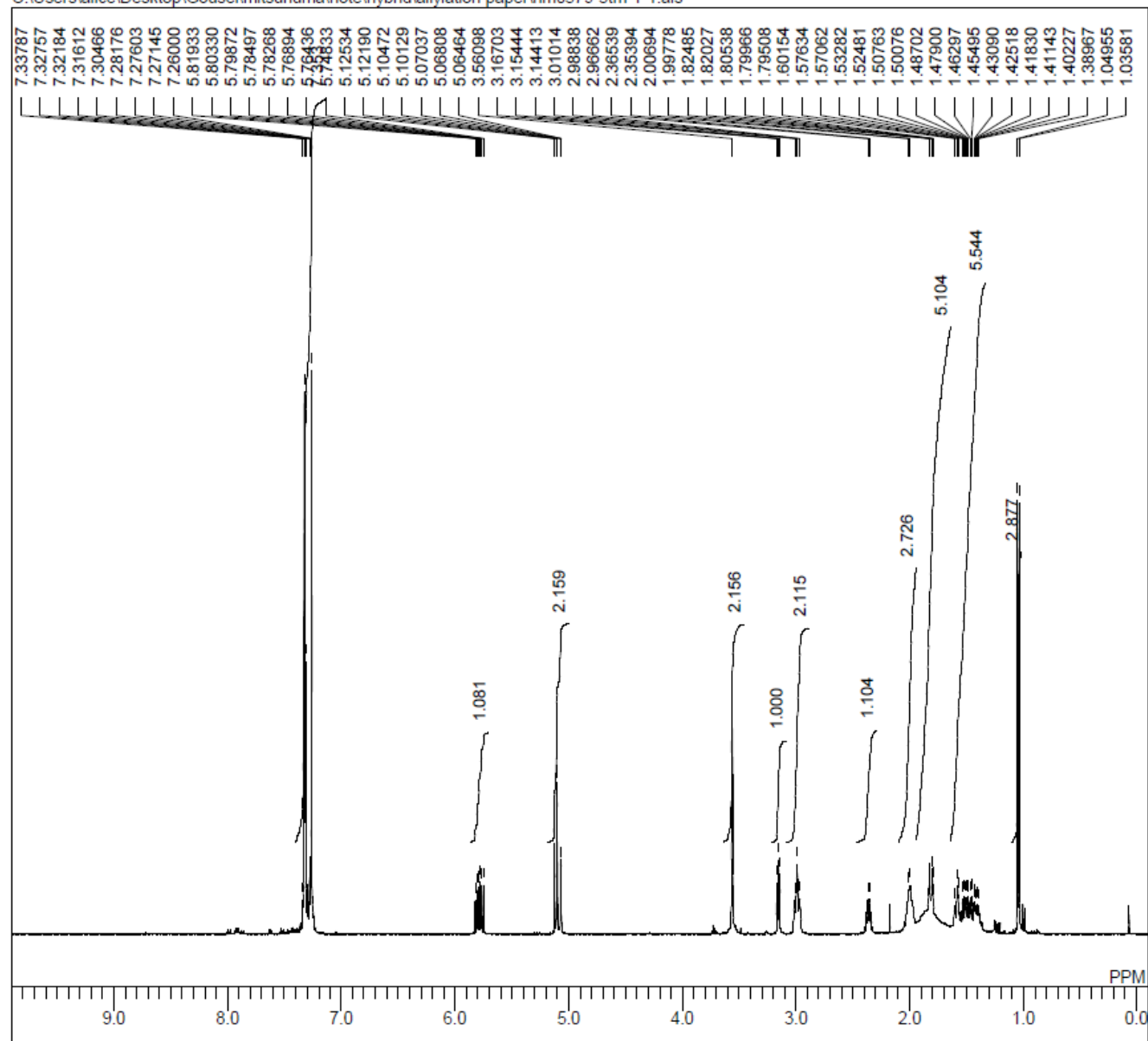


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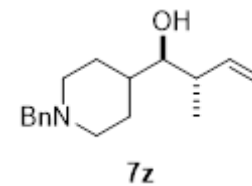


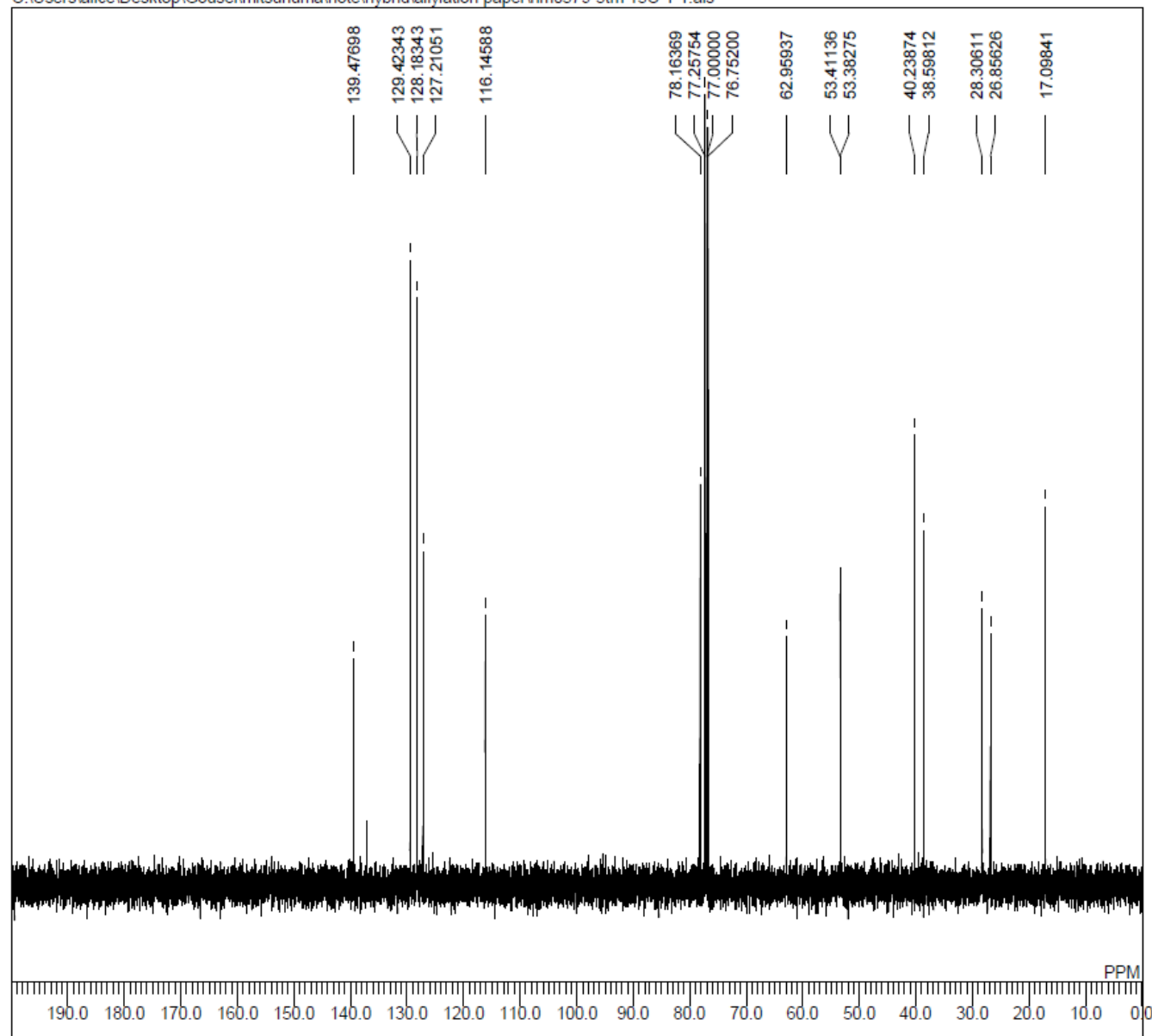
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COMNT
DATIM 2020-03-23 20:35:22
OBNUC 13C
EXMOD carbon.jpg
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 124
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

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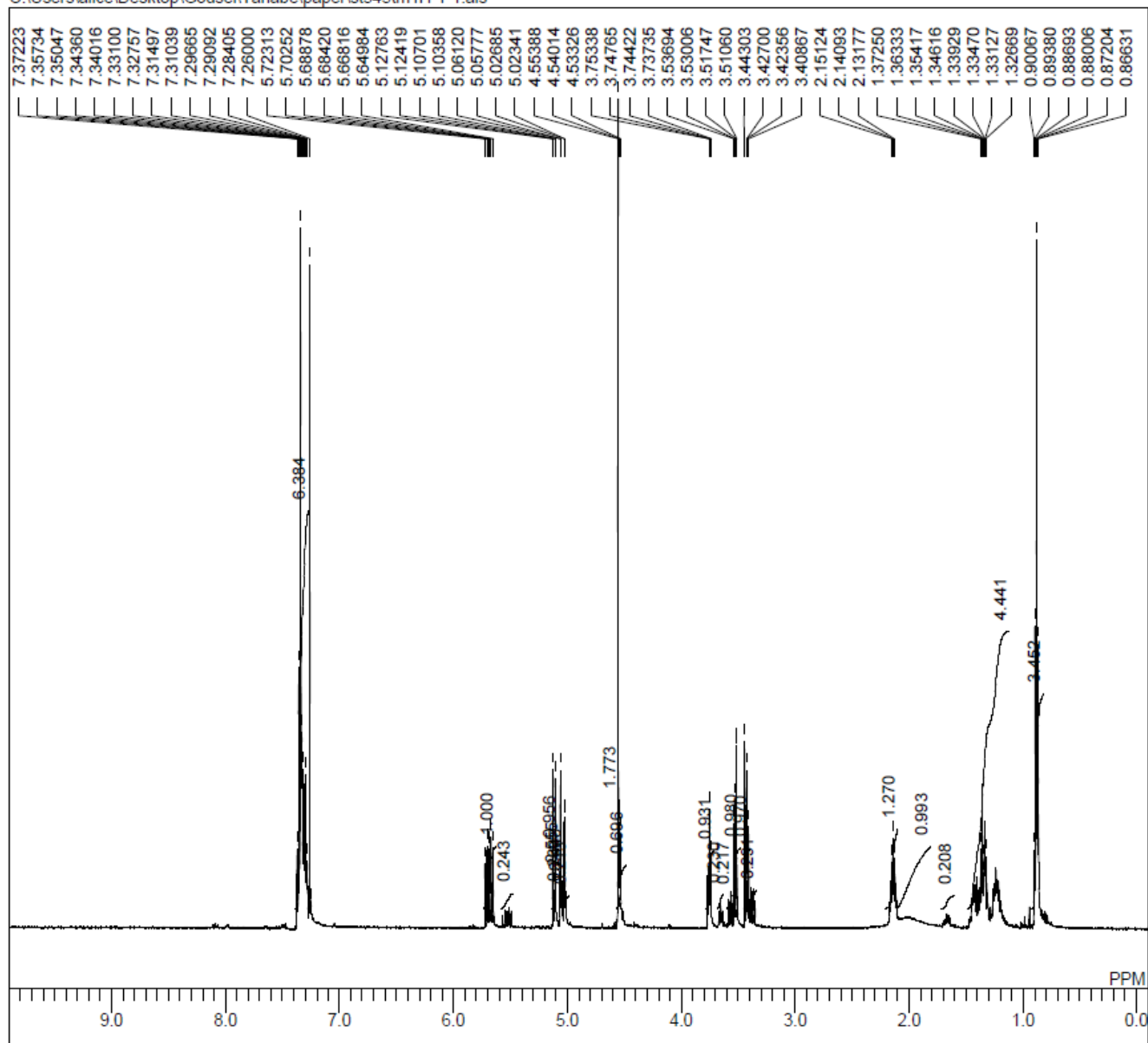
DFILE hm0379-3tm-1-1.als
COMNT
DATIM 2020-03-03 19:27:23
OBNUC 1H
EXMOD proton.jpg
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 6.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 42



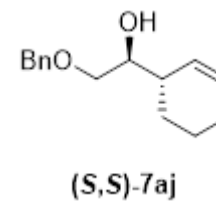


DFILE hm0379-3tm 13C-1-1.als
COMNT
DATIM 2020-03-03 19:40:56
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 36
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

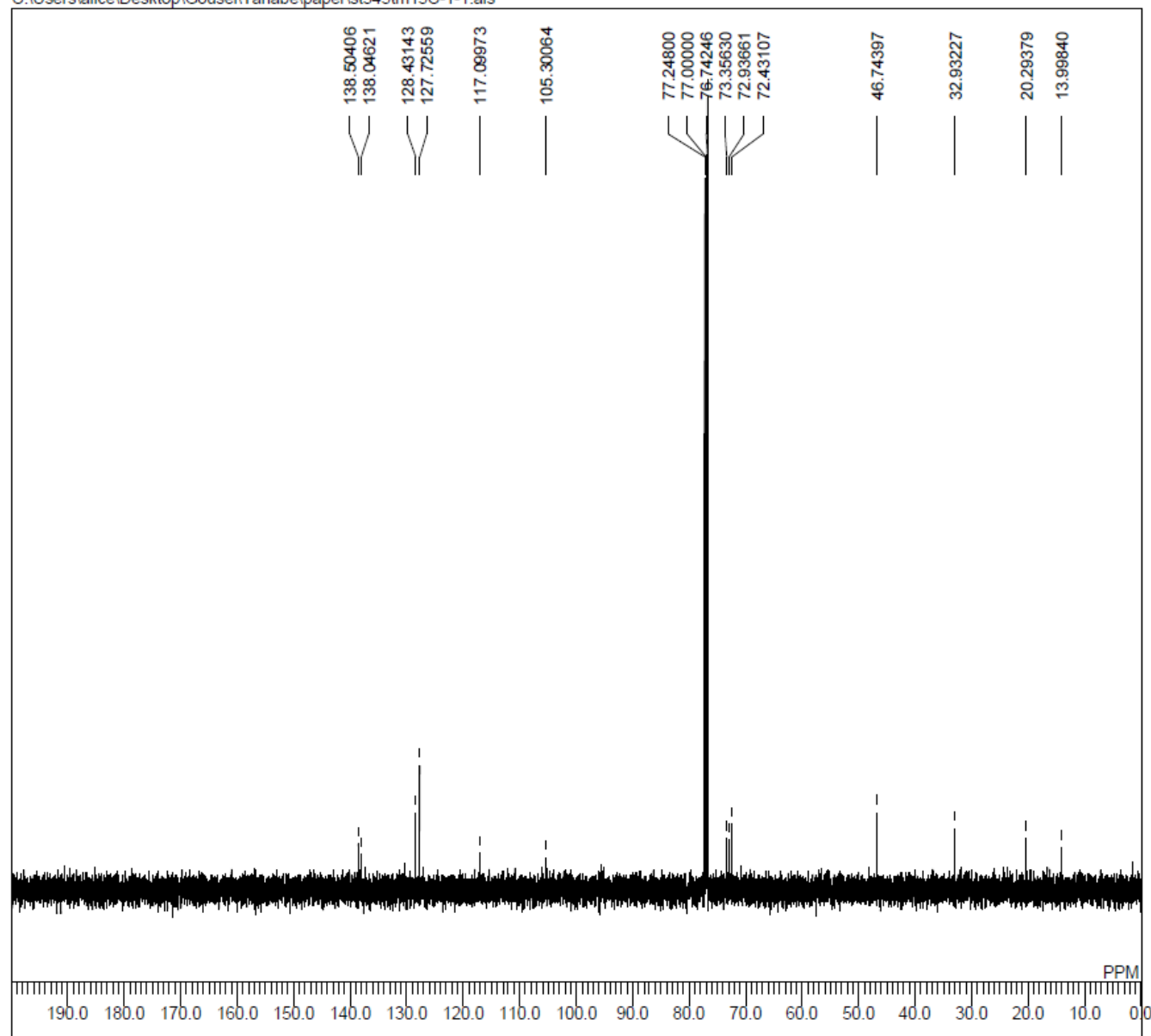
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DFILE st343tm1H-1-1.als
 COMNT
 DATIM 2020-04-07 12:02:48
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 13107
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 6.0000 sec
 PW1 5.55 usec
 IRNUC 1H
 CTEMP 21.5 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 42

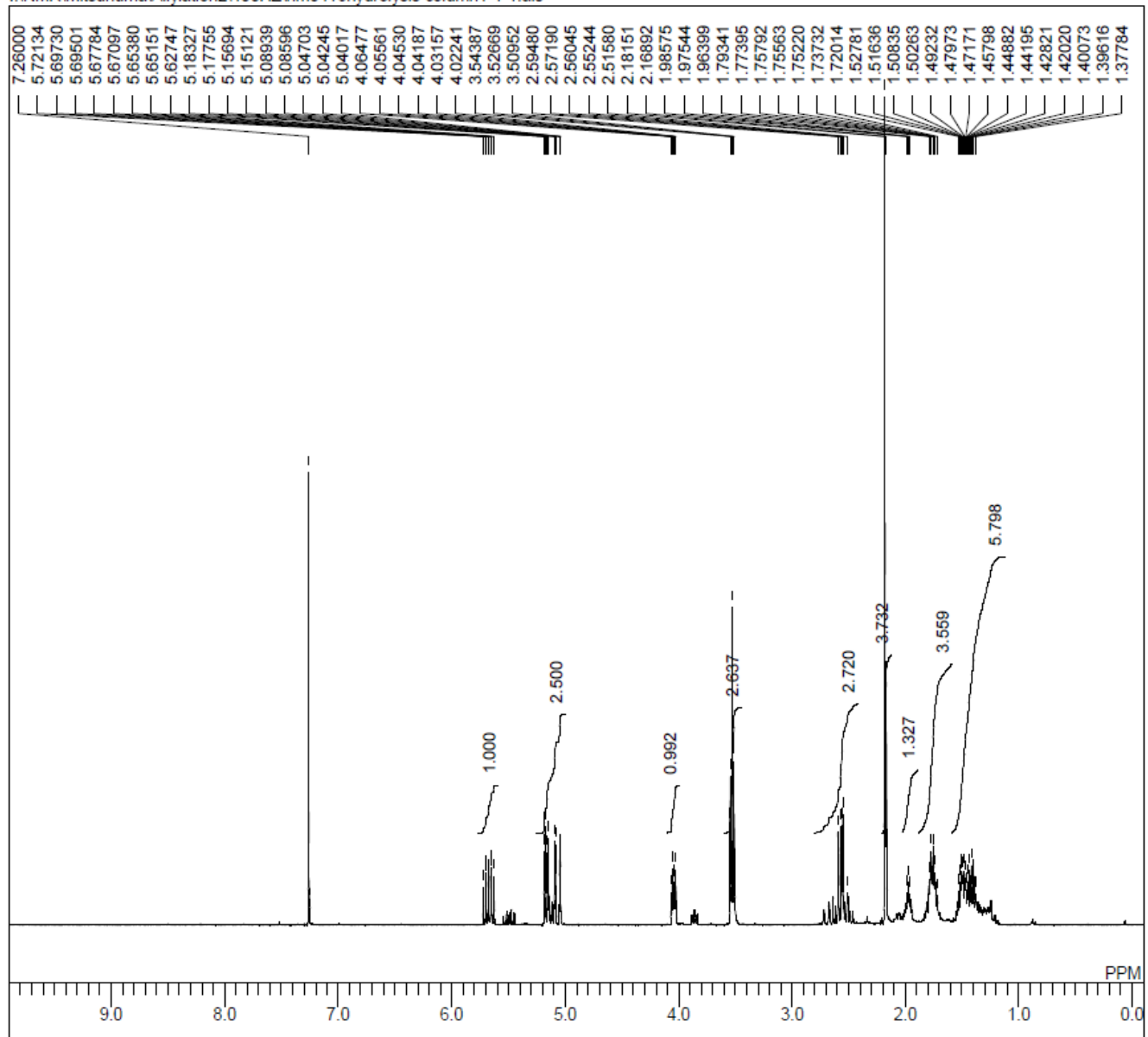


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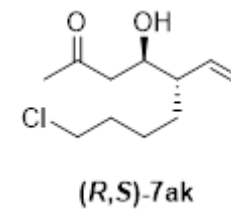


DFILE st343tm13C-1-1.als
COMNT
DATIM 2020-04-07 12:04:27
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 58
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.40 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

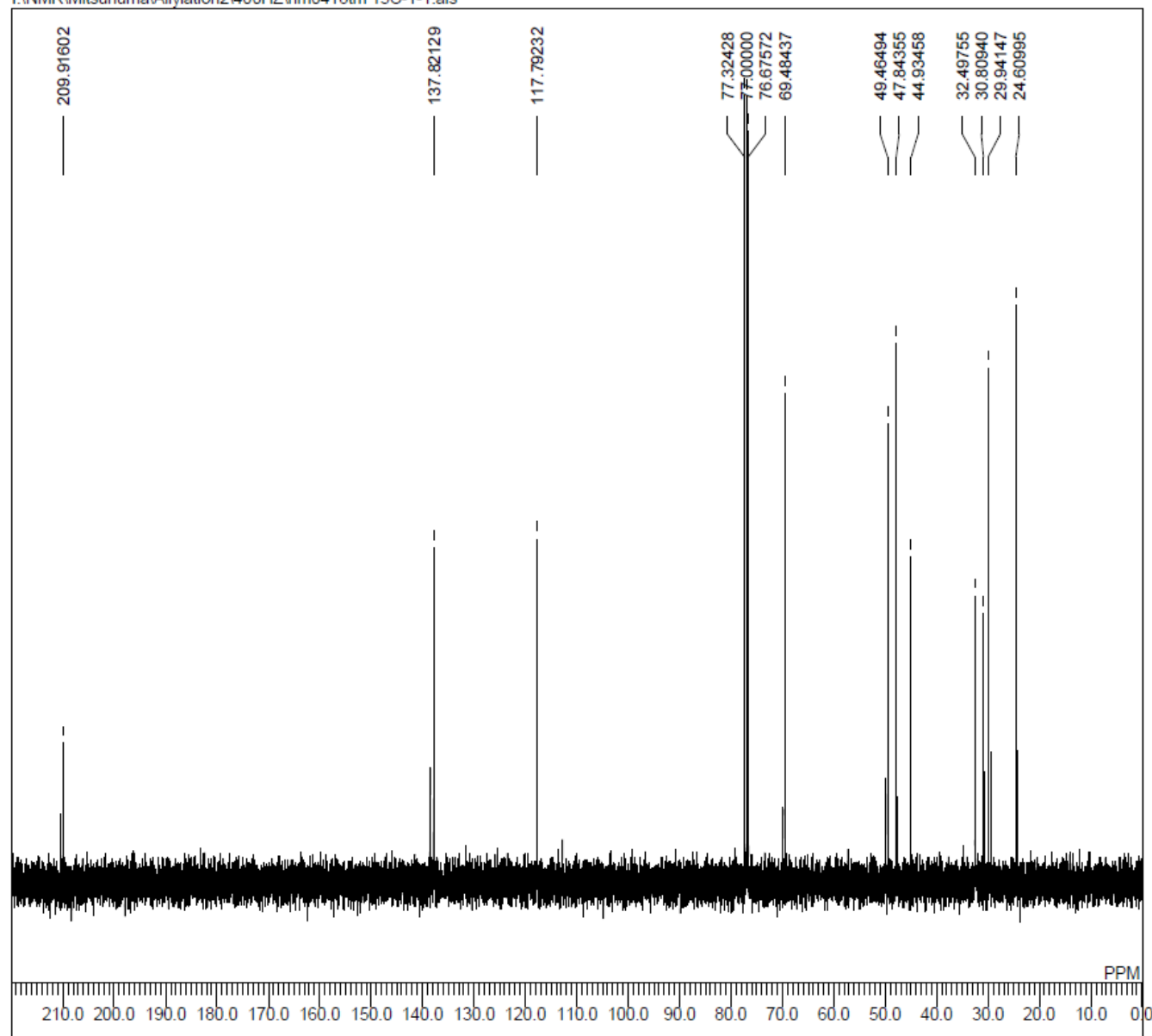
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DFILE hm0418hydrolysis column1-1-1.als
COMNT
DATIM 02-04-2020 15:46:25
OBNUC 1H
EXMOD proton.jxp
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 13107
FREQU 5878.90 Hz
SCANS 8
ACQTM 2.2295 sec
PD 6.0000 sec
PW1 5.17 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 44



I:\NMR\Mitsunuma\Allylation2\400HZ\hm0418tm 13C-1-1.als



DFILE hm0418tm 13C-1-1.als
COMNT
DATIM 02-04-2020 18:22:34
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.54 Hz
SCANS 57
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60