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(*E*)-Selective Friedel-Crafts Acylation of Alkynes to β-Chlorovinyl Ketones: Defying Isomerizations in Batch Reactions by Flow Chemistry Approaches

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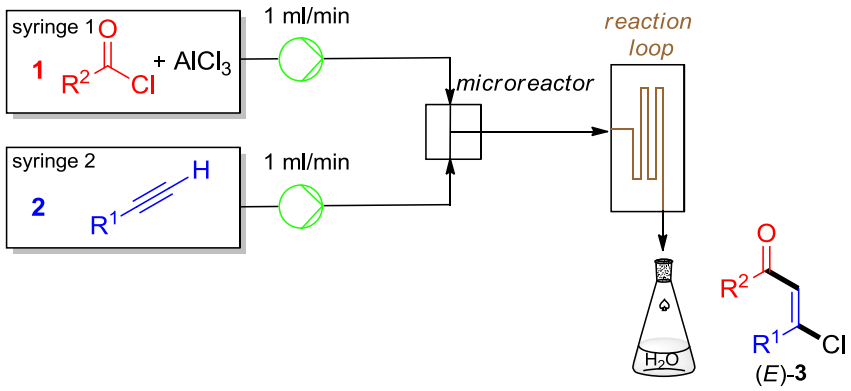
General Methods

All reactions were carried out using oven-dried glasses and leur-lock syringes. The progress of all reactions and the *E*/*Z* ratio were monitored by TLC and ^{1}H-NMR. Unless otherwise specified, all chemicals were purchased from Alfa Aesar, TCI, or Acros, and all solvents were purchased from Fischer Scientific. The syringe pumps and microreactors were purchased from YMC (Keychem mixer; simple Y, static, helix), Techno Applications (Comet; X-01-T, X-01-SS), and MiChS (MiChS β-type mixer). The PTFE tube (1/16" O.D.; 1.0 mm I.D.) and the fitting (1/4-28) were used. ^{1}H and ^{13}C-NMR spectra were measured using Jeol 600 MHz FT-NMR. $CDCl_3$ was used as the NMR solvent and set as the reference point. (^{1}H-NMR 7.26 ppm, ^{13}C-NMR 77.16 ppm) The coupling constant was measured in Hz and the signal patterns were as follows: (s = singlet, d = doublet, t = triplet, dd = doublet of doublet, dt = doublet of triplet, dq = doublet of quartet, td = triplet of doublet, tt = triplet of triplet, m = multiplet, brs = broad singlet). Infrared spectra were obtained using a Thermo Nicolet IR 300 Spectrometer. Silica gel 60 (0.040-0.063 mm, Merck, Inc.) was used for column chromatography.

General Procedure A: Flow Reactions

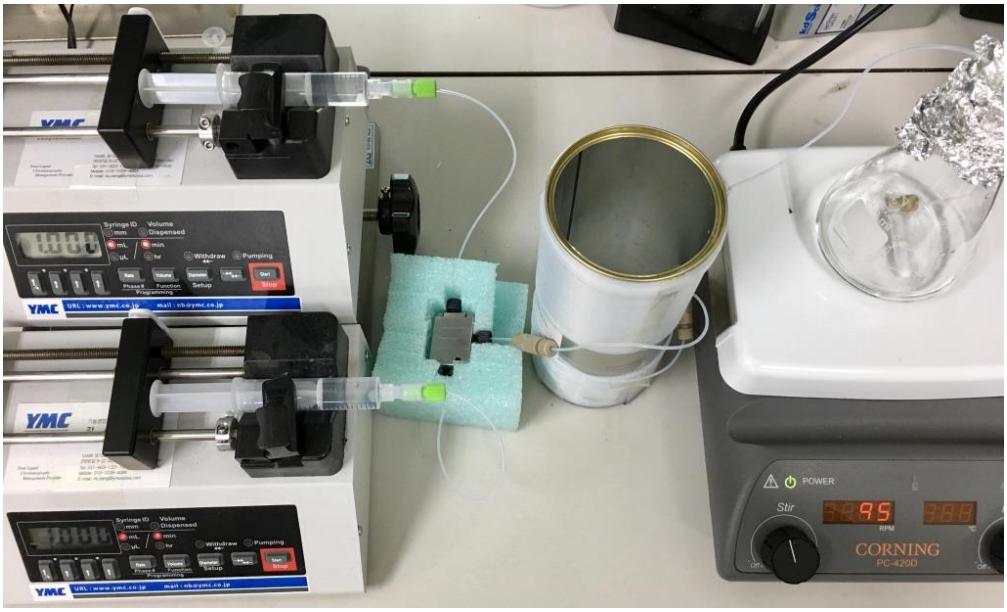


Alkyne solution (**1**, 0.5 mmol in 5 mL DCM) was charged in 5 mL syringe 1. Acyl chlorides (**2**, 0.5 mmol) and $AlCl_3$ (73 mg, 0.55 mmol) were dissolved in 5 mL of DCM in an oven-dried 20 mL glass vial and sonicated for 1 min. This clear solution was then transferred into the 5 mL syringe 2. After fitting syringes to each syringe pump system, the solutions were injected into the flow system at 1

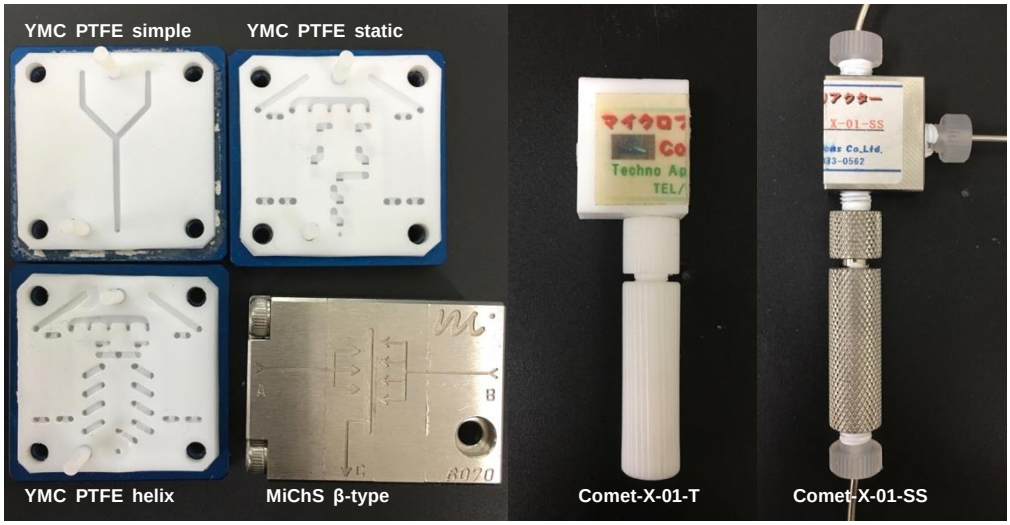
mL/min rate. Two solutions were mixed and the reaction proceeded as passing through the 9 mL reaction loop. A water vessel containing 10 mL of water was placed at the end of the reaction loop. The reaction solution was quenched as it dropped into this water vessel. After the continuous reaction stream was finished, additional 10 mL of DCM was injected into the flow system at the same rate and collected in the same water vessel. The organic layer was extracted with DCM (20 mL x 3) and washed with brine. The solution was dried over Na_2SO_4 and concentrated under the reduced pressure. The *E*/*Z* ratio of the crude mixture was determined by ^{1}H-NMR and the desired (*E*)-isomer was isolated by column chromatography (2-10% ethyl acetate in hexanes).

Reaction in 1 mmol Scale: 1-Heptyne (131 uL, 1 mmol in 10 mL DCM) was charged in 10 mL syringe 1. Benzoyl chloride (118 uL, 1 mmol) and $AlCl_3$ (146 mg, 1.1 mmol) were dissolved in 10 mL of DCM in an oven-dried 20 mL glass vial and sonicated for 1 min. This clear solution was then transferred into the 10 mL syringe 2. Employing these two solution, the compound, (*E*)-**3a** was prepared by flow reaction method described in the general procedure A in 70% yield (165 mg). The *E*/*Z* ratio of the crude mixture was 5/1 by ^{1}H-NMR.

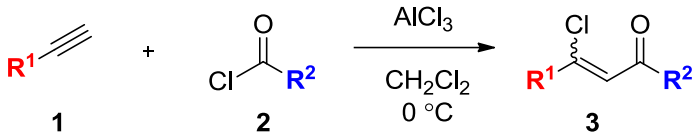
A Representative Flow Reaction System



Types of Microreactors

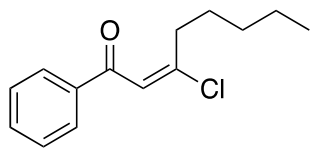


General Procedure B: Batch Reactions

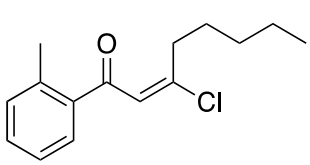


β-Chlorovinyl ketones were synthesized by the previously reported method.[1] $AlCl_3$ (73 mg, 0.55mmol), and 5 mL of DCM were charged into an oven-dried 20 mL glass vial under Ar and the reaction flask was placed in an ice bath (0 °C). Alkynes (**1**, 0.5 mmol) and acyl chlorides (**2**, 0.5 mmol) were then slowly added at the same time. The reaction was continued to stir at the same temperature until the reaction was complete by TLC (30-50 min). The reaction mixture was quenched with water, extracted with DCM (20 mL x 3), and washed with brine. The solution was dried over Na_2SO_4 and the solvent was removed under reduced pressure. The *E*/*Z* ratio of the crude products was determined by ^{1}H-NMR and the crude mixture was purified by column chromatography (2-10% ethyl acetate in hexanes).

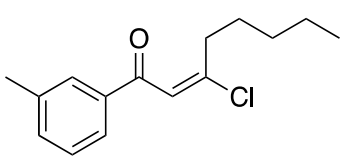
Characterization of (*E*)-β-Chlorovinyl Ketones



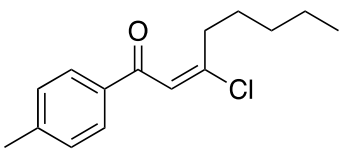
(*E*)-3-Chloro-1-phenyloct-2-en-1-one (3a): The product **3a** was prepared by the general procedure A using benzoyl chloride and 1-heptyne. ^{1}H NMR and ^{13}C NMR spectra for this compound are consistent with the previously reported literature data.[1] 83 mg (70%); pale yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.91-7.93 (m, 2H), 7.55-7.58 (m, 1H), 7.46-7.49 (m, 2H), 7.12 (s, 1H), 2.97 (t, *J* = 7.6 Hz, 2H), 1.68-1.70 (m, 2H), 1.34-1.37 (m, 4H), 0.90 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 188.7, 158.0, 138.3, 133.2, 128.8, 128.4, 123.4, 36.6, 31.2, 27.6, 22.6, 14.1.



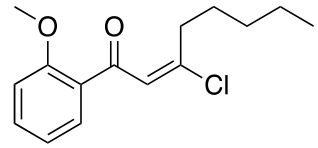
(*E*)-3-Chloro-1-(o-tolyl)oct-2-en-1-one (3b): The product **3b** was prepared by the general procedure A using *ortho*-toluoyl chloride and 1-heptyne. ^{1}H NMR and ^{13}C NMR spectra for this compound are consistent with the previously reported literature data.[2] 88 mg (70%); pale yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.54 (d, *J* = 7.4 Hz, 1H), 7.37 (t, *J* = 7.5 Hz, 1H), 7.24-7.27 (m, 2H), 6.83 (s, 1H), 2.93-2.95 (m, 2H), 2.49 (s, 3H), 1.67-1.69 (m, 2H), 1.34-1.35 (m, 4H), 0.90 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 192.7, 157.1, 139.3, 137.9, 131.9, 131.4, 128.6, 126.8, 125.9, 36.2, 31.2, 27.6, 22.6, 20.8, 14.1.



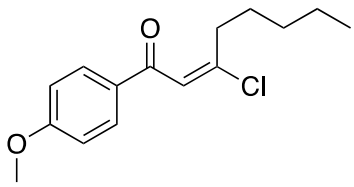
(*E*)-3-Chloro-1-(*m*-tolyl)oct-2-en-1-one (3c): The product **3c** was prepared by the general procedure A using *meta*-toluoyl chloride and 1-heptyne. ^{1}H NMR and ^{13}C NMR spectra for this compound are consistent with the previously reported literature data.[3] 82 mg (66%); pale yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.73 (s, 1H), 7.71 (d, *J* = 7.2 Hz, 1H) 7.34-7.39 (m, 2H), 7.10 (s, 1H), 2.96 (t, *J* = 7.6 Hz, 2H), 2.42 (s, 3H), 1.66-1.71 (m, 2H), 1.33-1.38 (m, 4H), 0.90 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 188.9, 157.6, 138.6, 138.2, 133.9, 128.9, 128.6, 125.6, 123.5, 36.5, 31.1, 27.6, 22.5, 21.4, 14.0.



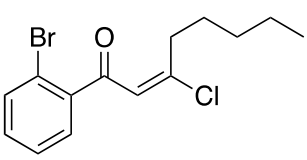
(*E*)-3-Chloro-1-(*p*-tolyl)oct-2-en-1-one (3d): The product **3d** was prepared by the general procedure A using *para*-toluoyl chloride and 1-heptyne. ^{1}H NMR and ^{13}C NMR spectra for this compound are consistent with the previously reported literature data.[1] 100 mg (80%); pale yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.83 (d, *J* = 8.2 Hz, 2H), 7.27 (d, *J* = 7.1 Hz, 2H), 7.10 (s, 1H), 2.96 (t, *J* = 7.6 Hz, 2H), 2.42 (s, 3H), 1.67-1.69 (m, 2H), 1.34-1.37 (m, 4H), 0.89 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 188.4, 157.3, 144.1, 135.8, 129.5, 128.6, 123.5, 36.5, 31.2, 27.6, 22.6, 21.8, 14.1.



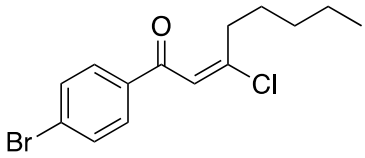
(*E*)-3-Chloro-1-(2-methoxyphenyl)oct-2-en-1-one (3e) : The product **3e** was prepared by the general procedure A using 2-methoxy benzoyl chloride and 1-heptyne. 88 mg (66%); pale yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.61 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.44-7.47 (m, 1H), 7.03 (s, 1H), 7.00 (td, *J* = 7.5, 0.9 Hz, 1H), 6.95 (d, *J* = 8.3 Hz, 1H), 3.90 (s, 3H), 2.97 (t, *J* = 7.6 Hz, 2H), 1.65-1.70 (m, 2H), 1.33-1.38 (m, 4H), 0.90 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 190.0, 158.3, 155.8, 133.6, 130.7, 129.6, 127.8, 120.9, 111.7, 55.8, 36.5, 31.2, 27.6, 22.6, 14.1; IR(neat): 2957, 2931, 2860, 1661, 1600, 1485, 1464, 1436, 1284, 1245, 1214, 1162, 1112, 1022, 839, 753 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{15}H_{20}ClO_2$ $[M+H]^+$ 267.1146 Found 267.1151.



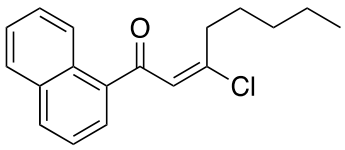
(*E*)-3-Chloro-1-(4-methoxyphenyl)oct-2-en-1-one (3f) : The product **3f** was prepared by the general procedure A using 4-methoxy benzoyl chloride and 1-heptyne. ^{1}H NMR and ^{13}C NMR spectra for this compound are consistent with the previously reported literature data.[1] 111 mg (83%); pale yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.89-7.91 (m, 2H), 7.07 (s, 1H), 6.91-6.94 (m, 2H), 3.85 (s, 3H), 2.94 (t, *J* = 7.6 Hz, 2H), 1.64-1.69 (m, 2H), 1.31-1.37 (m, 4H), 0.88 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 187.3, 163.6, 156.6, 131.2, 130.7, 123.4, 113.9, 55.6, 36.4, 31.2, 27.6, 22.5, 14.0.



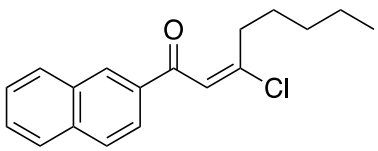
(*E*)-1-(2-Bromophenyl)-3-chlorooct-2-en-1-one (3g): The product **3g** was prepared by the general procedure A using 2-bromo benzoyl chloride and 1-heptyne. 124 mg (79%); yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.60 (dd, *J* = 7.9, 1.0 Hz, 1H), 7.43 (dd, *J* = 7.6, 1.7 Hz, 1H), 7.37 (td, *J* = 7.5, 1.1 Hz, 1H), 7.29 (td, *J* = 7.7, 1.7 Hz, 1H), 6.79 (s, 1H), 3.00 (t, *J* = 7.6 Hz, 2H), 1.67-1.72 (m, 2H), 1.33-1.38 (m, 4H), 0.91 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 190.7, 159.0, 141.8, 133.8, 132.0, 129.4, 127.6, 126.2, 119.5, 36.5, 31.2, 27.6, 22.5, 14.1; IR(neat): 2956, 2930, 2860, 1676, 1593, 1465, 1429, 1355, 1065, 765, 739 cm^{-1}; HRMS(ESI): m/z calcd. For $C_{14}H_{17}BrClO$ $[M+H]^+$ 315.0145 Found 315.0152.



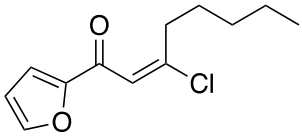
(*E*)-1-(4-Bromophenyl)-3-chlorooct-2-en-1-one (3h): The product **3h** was prepared by the general procedure A using 4-bromo benzoyl chloride and 1-heptyne. ^{1}H NMR and ^{13}C NMR spectra for this compound are consistent with the previously reported literature data.[1] 112 mg (71%); yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.77 (dd, *J* = 6.7, 1.9 Hz, 2H), 7.60 (dd, *J* = 6.7, 1.9 Hz, 2H), 7.05 (s, 1H), 2.96 (t, *J* = 7.6 Hz, 2H), 1.66-1.69 (m, 2H), 1.33-1.36 (m, 4H), 0.89 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 187.6, 158.9, 137.1, 132.1, 129.9, 128.4, 122.8, 36.7, 31.2, 27.6, 22.5, 14.1.



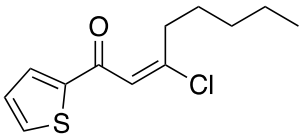
(*E*)-3-Chloro-1-(naphthalen-1-yl)oct-2-en-1-one (3i): The product **3i** was prepared by the general procedure A using 1-naphthoyl chloride and 1-heptyne. 107 mg (75%); yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.49 (d, *J* = 8.1 Hz, 1H), 7.99 (d, *J* = 8.1 Hz, 1H), 7.89 (d, *J* = 7.9 Hz, 1H), 7.81 (dd, *J* = 7.1, 1.0 Hz, 1H), 7.50-7.61 (m, 3H), 6.99 (s, 1H), 3.02 (t, *J* = 7.5 Hz, 2H), 1.73 (m, 2H), 1.37 (m, 4H), 0.91 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 192.2, 157.7, 137.2, 134.0, 132.7, 130.3, 128.7, 128.0, 127.9, 127.2, 126.7, 125.6, 124.6, 36.5, 31.2, 27.7, 22.6, 14.1; IR(neat): 2956, 2930, 2860, 2360, 1665, 1592, 1508, 1097, 804, 777 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{18}H_{20}ClO$ $[M+H]^+$ 287.1197 Found 287.1201.



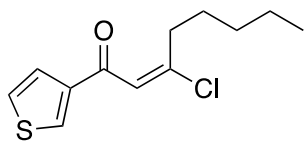
(*E*)-3-Chloro-1-(naphthalen-2-yl)oct-2-en-1-one (3j): The product **3j** was prepared by the general procedure A using 2-naphthoyl chloride and 1-heptyne. ^{1}H NMR and ^{13}C NMR spectra for this compound are consistent with the previously reported literature data.[3] 86 mg (60%); yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.43 (d, *J* = 1.1 Hz, 1H), 8.01 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.97 (d, *J* = 7.8 Hz, 1H), 7.88-7.92 (m, 2H), 7.60-7.62 (m, 1H), 7.55-7.58 (m, 1H), 7.28 (s, 1H), 3.02 (t, *J* = 7.6 Hz, 2H), 1.70-1.75 (m, 2H), 1.34-1.40 (m, 4H), 0.90 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 188.6, 157.8, 135.7, 135.6, 132.6, 130.1, 129.7, 128.8, 128.7, 128.0, 127.0, 124.2, 123.5, 36.6, 31.2, 27.7, 22.6, 14.1.



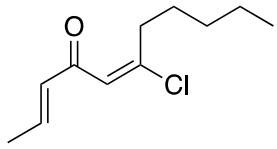
(*E*)-3-Chloro-1-(furan-2-yl)oct-2-en-1-one (3k): The product **3k** was prepared by the general procedure A using 2-furoyl chloride and 1-heptyne. 84 mg (74%); pale yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.59 (brs, 1H), 7.20 (d, *J* = 3.6 Hz, 1H), 7.03 (s, 1H), 6.54 (dd, *J* = 3.4, 1.5 Hz, 1H), 3.06 (t, *J* = 7.6 Hz, 2H), 1.64-1.69 (m, 2H), 1.31-1.38 (m, 4H), 0.89 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 176.3, 159.2, 153.6, 146.6, 122.0, 117.4, 112.6, 36.6, 31.1, 27.6, 22.5, 14.0; IR(neat): 2957, 2932, 1660, 1598, 1568, 1467, 1396, 1255, 1010, 759 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{12}H_{16}ClO_2$ $[M+H]^+$ 227.0833 Found 227.0837.



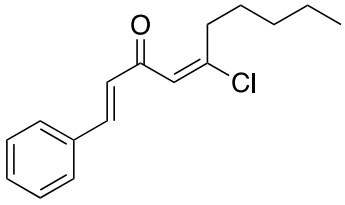
(*E*)-3-Chloro-1-(thiophen-2-yl)oct-2-en-1-one (3l): The product **3l** was prepared by the general procedure A using 2-thenoyl chloride and 1-heptyne. 102 mg (84%); pale yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.70 (dd, *J* = 3.8, 1.1 Hz, 1H), 7.65 (dd, *J* = 4.9, 1.1 Hz, 1H), 7.14 (dd, *J* = 4.9, 3.9 Hz, 1H), 7.02 (s, 1H), 3.03 (t, *J* = 7.6 Hz, 2H), 1.65-1.70 (m, 2H), 1.33-1.37 (m, 4H), 0.89 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 180.7, 158.6, 146.0, 134.3, 131.8, 128.4, 122.6, 36.6, 31.2, 27.7, 22.6, 14.1; IR(neat): 2956, 2931, 1649, 1593, 1239, 721, 678 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{12}H_{16}ClOS$ $[M+H]^+$ 243.0604 Found 243.0609.



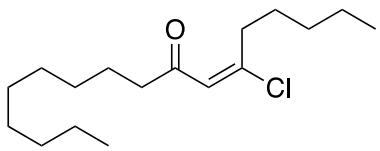
(*E*)-3-Chloro-1-(thiophen-3-yl)oct-2-en-1-one (3m): The product **3m** was prepared by the general procedure A using 3-thenoyl chloride and 1-heptyne. 81mg (67%); pale yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.03 (dd, *J* = 2.8, 1.1 Hz, 1H), 7.56 (dd, *J* = 5.1, 1.1 Hz, 1H), 7.32 (dd, *J* = 5.1, 2.8 Hz, 1H), 7.00 (s, 1H), 3.00 (t, *J* = 7.6 Hz, 2H), 1.65-1.70 (m, 2H), 1.34-1.37 (m, 4H), 0.89 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 182.2, 158.1, 143.5, 132.2, 127.3, 126.7, 123.7, 36.5, 31.2, 27.6, 22.5, 14.1; IR(neat): 2956, 2931, 1649, 1593, 1239, 859, 721, 678 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{12}H_{16}ClOS$ $[M+H]^+$ 243.0604 Found 243.0611.



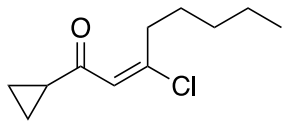
(2*E*,5*E*)-6-Chloroundeca-2,5-dien-4-one (3n): The product **3n** was prepared by the general procedure A using (*E*)-2-butenoyl chloride and 1-heptyne. 61 mg (61%); pale yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 6.85-6.91 (dq, *J* = 15.1, 6.7 Hz, 1H), 6.58 (s, 1H), 6.14 (dq, *J* = 15.6, 1.7 Hz, 1H), 2.92 (t, *J* = 7.6 Hz, 2H), 1.91 (dd, *J* = 6.9, 1.7 Hz, 3H), 1.61-1.64 (m, 2H), 1.31-1.34 (m, 4H), 0.89 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 187.5, 157.0, 143.7, 133.1, 124.8, 36.3, 31.1, 27.5, 22.5, 18.4, 14.0; IR(neat): 2957, 2931, 1681, 1661, 1629, 1594, 1441, 1357, 1078, 967 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{11}H_{18}ClO$ $[M+H]^+$ 201.1040 Found 201.1042.



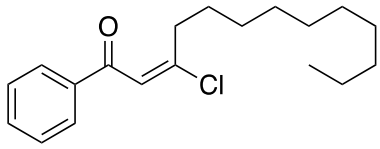
(1*E*,4*E*)-5-Chloro-1-phenyldeca-1,4-dien-3-one (3o): The product **3o** was prepared by the general procedure A using (*E*)-cinnamoyl chloride and 1-heptyne. 104 mg (79%); yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.60 (d, *J* = 16.0 Hz, 1H), 7.55-7.57 (m, 2H), 7.39-7.41 (m, 3H), 6.76 (d, *J* = 16.0 Hz, 1H), 6.71 (s, 1H), 3.01 (t, *J* = 7.6 Hz, 2H), 1.66-1.68 (m, 2H), 1.34-1.37 (m, 4H), 0.91 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 187.3, 157.8, 143.6, 134.6, 130.8, 129.1, 128.5, 127.5, 125.5, 36.4, 31.2, 27.6, 22.5, 14.1; IR(neat): 2956, 2930, 1673, 1651, 1612, 1449, 1358, 1183, 1089, 977, 757, 696 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{16}H_{20}ClO$ $[M+H]^+$ 263.1197 Found 263.1204.



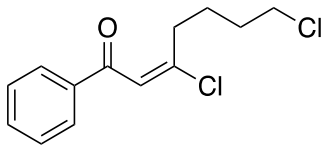
(*E*)-6-Chloroheptadec-6-en-8-one (3p): The product **3p** was prepared by the general procedure A using decanoyl chloride and 1-heptyne. 103 mg (72%); pale yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 6.41 (s, 1H), 2.92 (t, *J* = 7.6 Hz, 2H), 2.42 (t, *J* = 7.4 Hz, 2H), 1.56-1.61 (m, 4H), 1.25-1.33 (m, 16H), 0.86-0.90 (m, 6H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 198.5, 156.7, 125.7, 44.8, 36.1, 32.0, 31.1, 29.6(2C), 29.4, 29.3, 27.5, 24.1, 22.8, 22.5, 14.2, 14.1; IR(neat): 2956, 2927, 2855, 1696, 1601, 1466, 1356 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{17}H_{32}ClO$ $[M+H]^+$ 287.2136 Found 287.2143.



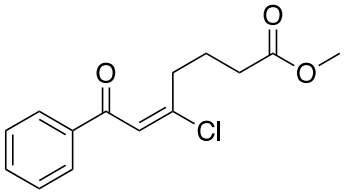
(*E*)-3-Chloro-1-cyclopropyloct-2-en-1-one (3q): The product **3q** was prepared by the general procedure A using cyclopropanecarbonyl chloride and 1-heptyne. 63 mg (63%); pale yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 6.58 (s, 1H), 2.91 (t, *J* = 7.6 Hz, 2H), 1.91 (m, 1H), 1.58-1.62 (m, 2H), 1.29-1.34 (m, 4H), 1.06-1.09 (m, 2H), 0.92 (m, 2H), 0.88 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 198.1, 156.1, 126.2, 36.2, 31.1, 27.5, 22.8, 22.5, 14.1, 11.7; IR(neat): 2958, 2932, 2861, 1680, 1600, 1385, 1085, 1023, 890, 644 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{11}H_{18}ClO$ $[M+H]^+$ 201.1040 Found 201.1045.



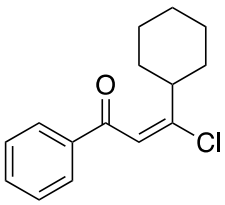
(*E*)-3-Chloro-1-phenyltridec-2-en-1-one (3r): The product **3r** was prepared by the general procedure A using benzoyl chloride and 1-dodecyne. ^{1}H NMR and ^{13}C NMR spectra for this compound are consistent with the previously reported literature data.[1] 115 mg (75%); pale yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.92 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.55-7.58 (m, 1H), 7.46-7.49 (m, 2H), 7.12 (s, 1H), 2.97 (t, *J* = 7.6 Hz, 2H), 1.67-1.69 (m, 2H), 1.37 (t, *J* = 7.7 Hz, 2H), 1.25-1.32 (m, 12H), 0.88 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 188.7, 157.9, 138.3, 133.1, 128.8, 128.4, 123.4, 36.6, 32.0, 29.7, 29.6, 29.4(9), 29.4(5), 29.1, 28.0, 22.8, 14.3.



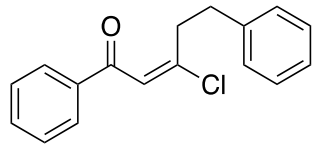
(*E*)-3,7-Dichloro-1-phenylhept-2-en-1-one (3s): The product **3s** was prepared by the general procedure A using benzoyl chloride and 6-chloro-1-hexyne. ^{1}H NMR and ^{13}C NMR spectra for this compound are consistent with the previously reported literature data.[1] 79 mg (62%); pale yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.92 (dd, *J* = 8.3, 1.4 Hz, 2H), 7.56-7.58 (m, 1H), 7.46-7.49 (m, 2H), 7.16 (s, 1H), 3.58 (t, *J* = 6.2 Hz, 2H), 3.02 (t, *J* = 6.9 Hz, 2H), 1.85-1.87 (m, 4H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 188.6, 156.7, 138.1, 133.3, 128.8, 128.4, 123.9, 44.7, 35.6, 31.6, 25.1.



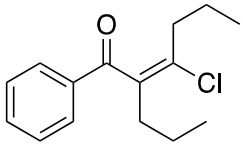
(*E*)-Methyl-5-chloro-7-oxo-7-phenylhept-5-enoate (3t): The product **3t** was prepared by the general procedure A using benzoyl chloride and methyl 5-hexynoate. ^{1}H NMR and ^{13}C NMR spectra for this compound are consistent with the previously reported literature data.[1] 85 mg (64%); pale yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.90 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.55-7.58 (m, 1H), 7.45-7.48 (m, 2H), 7.16 (s, 1H), 3.65 (s, 3H), 3.02 (t, *J* = 7.4 Hz, 2H), 2.41 (t, *J* = 7.6 Hz, 2H), 2.01-2.06 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 188.5, 173.6, 156.0, 138.1, 133.3, 128.8, 128.4, 124.3, 51.7, 35.6, 32.9, 23.0.



(*E*)-3-Chloro-3-cyclohexyl-1-phenylprop-2-en-1-one (3u): The product **3u** was prepared by the general procedure A using benzoyl chloride and cyclohexylacetylene. ^{1}H NMR and ^{13}C NMR spectra for this compound are consistent with the previously reported literature data.[1] 87 mg (70%); pale yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.93 (dd, *J* = 8.4, 1.2 Hz, 2H), 7.55-7.58 (m, 1H), 7.46-7.49 (m, 2H), 7.03 (s, 1H), 3.74 (tt, *J* = 11.5, 3.3 Hz, 1H), 1.77-1.81 (m, 2H), 1.69-1.75 (m, 3H), 1.54-1.61 (m, 2H), 1.35-1.41 (m, 2H), 1.17-1.24 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 188.8, 162.7, 138.3, 133.2, 128.8, 128.5, 122.6, 42.7, 30.8, 25.7(8), 25.7(3).

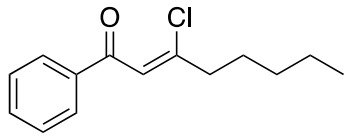


(*E*)-3-Chloro-1,5-diphenylpent-2-en-1-one (3v): The product **3v** was prepared by the general procedure A using benzoyl chloride and 4-phenyl-1-butyne. 101 mg (75%); pale yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.87 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.56-7.59 (m, 1H), 7.46-7.48 (m, 2H), 7.27-7.32 (m, 4H), 7.17-7.20 (m, 1H), 7.13 (s, 1H), 3.3 0-3.33 (m, 2H), 3.02-3.04 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 188.7, 155.8, 140.3, 138.1, 133.1, 128.7(0), 128.6(7), 128.5, 128.4, 126.3, 124.1, 38.5, 34.0; IR(neat): 3062, 3027, 1665, 1600, 1448, 1228, 824, 777, 697 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{17}H_{16}ClO$ $[M+H]^+$ 271.0884 Found 271.0889.

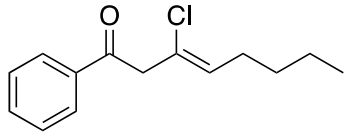


(*E*)-3-Chloro-1-phenyl-2-propylhex-2-en-1-one (3w): The product **3w** was prepared by the general procedure A using benzoyl chloride and 4-octyne. ^{1}H NMR and ^{13}C NMR spectra for this compound are consistent with the previously reported literature data.[4] 99 mg (79%); pale yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.90 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.58-7.61 (m, 1H), 7.47-7.50 (m, 2H), 2.48 (t, *J* = 7.9 Hz, 2H), 2.18 (t, *J* = 7.4 Hz, 2H), 1.53-1.59 (m, 2H), 1.42-1.48 (m, 2H), 0.92 (t, *J* = 7.4 Hz, 3H), 0.78 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 197.6, 137.4, 137.0, 136.5, 133.8, 129.6, 128.9, 39.2, 34.7, 21.1(2C), 14.0, 13.3.

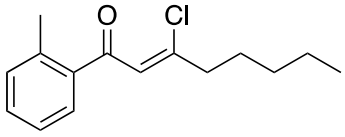
Characterization of (*Z*)-β-Chlorovinyl Ketones



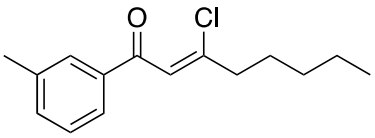
(*Z*)-3-Chloro-1-phenyloct-2-en-1-one (3a): The product **3a** was prepared by the general procedure B using benzoyl chloride and 1-heptyne. ^{1}H NMR and ^{13}C NMR spectra for this compound are consistent with the previously reported literature data.[1] 46 mg (39%); yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.94 (dd, *J* = 8.3, 1.3 Hz, 2H), 7.55-7.58 (m, 1H), 7.46-7.49 (m, 2H), 6.83 (t, *J* = 0.9 Hz, 1H), 2.53-2.55 (td, *J* = 7.5, 0.9 Hz, 2H), 1.69-1.72 (m, 2H), 1.35-1.39 (m, 4H), 0.93 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 189.9, 147.8, 137.8, 133.2, 128.7(3), 128.7(0), 121.3, 41.3, 30.9, 27.1, 22.5, 14.1.



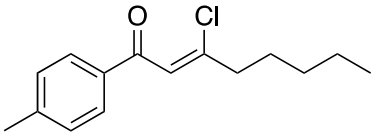
(*Z*)-3-Chloro-1-phenyloct-3-en-1-one (4a): The product **4a** was reported by previous study.[1] Only proton peaks were listed; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.96 (m, 2H), 7.55-7.58 (m, 1H), 7.46-7.49 (m, 2H), 5.66 (t, *J* = 0.9 Hz, 1H), 3.97 (d, *J* = 0.9 Hz, 2H), 2.22-2.26 (m, 2H), 1.35-1.39 (m, 4H), 0.93 (t, *J* = 7.1 Hz, 3H).



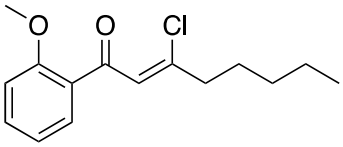
(*Z*)-3-Chloro-1-(o-tolyl)oct-2-en-1-one (3b): The product **3b** was prepared by the general procedure B using *ortho*-toluoyl chloride and 1-heptyne. 56 mg (45%); brown liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.54-7.56 (m, 1H), 7.35-7.38 (m, 1H), 7.24-7.26 (m, 2H), 6.63 (t, *J* = 0.8 Hz, 1H), 2.52 (s, 3H), 2.49 (td, *J* = 7.5, 0.8 Hz, 2H), 1.65-1.70 (m, 2H), 1.34-1.37 (m, 4H), 0.92 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 193.5, 147.7, 138.6, 138.3, 131.8, 131.4, 129.2, 125.8, 124.1, 41.3, 30.9, 27.1, 22.5, 20.9, 14.1; IR(neat): 2956, 2929, 2860, 1675, 1601, 1571, 1457, 1379, 1299, 1256, 1220, 1015, 776, 734 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{15}H_{20}ClO$ $[M+H]^+$ 251.1197 Found 251.1199.



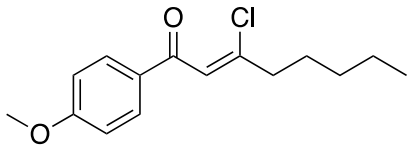
(*Z*)-3-Chloro-1-(*m*-tolyl)oct-2-en-1-one (3c): The product **3c** was prepared by the general procedure B using *meta*-toluoyl chloride and 1-heptyne. 46 mg (37%); brown liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.74 (s, 1H), 7.72 (d, *J* = 7.6 Hz, 1H), 7.33-7.38 (m, 2H), 6.81 (t, *J* = 0.9 Hz, 1H), 2.53 (td, *J* = 7.5, 0.7 Hz, 2H), 2.41 (s, 3H), 1.67-1.72 (m, 2H), 1.35-1.38 (m, 4H), 0.93 (t, *J* = 7.1 Hz, 3H)^{13}C NMR (150 MHz, $CDCl_3$): δ 190.1, 147.5, 138.5, 137.8, 134.0, 129.1, 128.6, 126.0, 121.5, 41.2, 30.9, 27.1, 22.5, 21.5, 14.1; IR(neat): 2955, 2929, 2860, 1669, 1607, 1250, 1169, 792, 682 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{15}H_{20}ClO$ $[M+H]^+$ 251.1197 Found 251.1197.



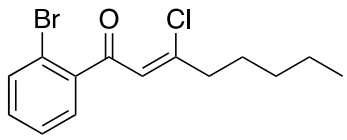
(*Z*)-3-Chloro-1-(*p*-tolyl)oct-2-en-1-one (3d): The product **3d** was prepared by the general procedure B using *para*-toluoyl chloride and 1-heptyne. ^{1}H NMR and ^{13}C NMR spectra for this compound are consistent with the previously reported literature data.[1] 48 mg (38%); brown liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.83 (d, *J* = 8.3 Hz, 2H), 7.25 (d, *J* = 7.7 Hz, 2H), 6.79 (t, *J* = 0.9 Hz, 1H), 2.52 (t, *J* = 7.5, 0.7 Hz, 2H), 2.40 (s, 3H), 1.66-1.71 (m, 2H), 1.35-1.37 (m, 4H), 0.92 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 189.6, 147.0, 144.1, 135.2, 129.4, 128.8, 121.5, 41.1, 30.9, 27.1, 22.4, 21.8, 14.0.



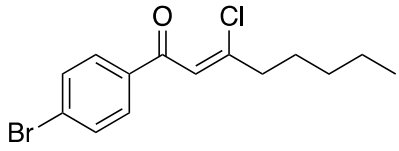
(*Z*)-3-Chloro-1-(2-methoxyphenyl)oct-2-en-1-one (3e): The product **3e** was prepared by the general procedure B using 2-methoxybenzoyl chloride and 1-heptyne. 24 mg (18%); brown liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.66 (dd, *J* = 7.6, 1.7 Hz, 1H), 7.44-7.47 (m, 1H), 7.01 (td, *J* = 7.6, 0.9 Hz, 1H), 6.94 (dd, *J* = 8.3, 0.7 Hz, 1H), 6.86 (t, *J* = 0.8 Hz, 1H), 3.87 (s, 3H), 2.48 (td, *J* = 7.2 Hz, 0.7 Hz, 2H), 1.64-1.69 (m, 2H), 1.33-1.36 (m, 4H), 0.92 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 190.2, 158.3, 146.4, 133.5, 130.9, 129.5, 125.0, 121.0, 111.7, 55.8, 41.5, 30.8, 27.1, 22.5, 14.1; IR(neat): 2956, 2931, 1663, 1602, 1485, 1464, 1436, 1283, 1244, 1211, 1023, 756 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{15}H_{20}ClO_2$ $[M+H]^+$ 267.1146 Found 267.1147.



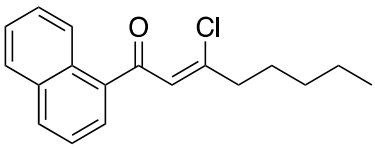
(*Z*)-3-Chloro-1-(4-methoxyphenyl)oct-2-en-1-one (3f): The product **3f** was prepared by the general procedure B using 4-methoxybenzoyl chloride and 1-heptyne. 73 mg (55%); yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.92 (d, *J* = 8.9 Hz, 2H), 6.94 (d, *J* = 9.0 Hz, 2H), 6.75 (t, *J* = 0.9 Hz, 1H), 3.87 (s, 3H), 2.51 (td, *J* = 7.5, 0.7 Hz, 2H), 1.66-1.71 (m, 2H), 1.35-1.37 (m, 4H), 0.92 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 188.9, 163.8, 146.3, 131.2, 130.6, 121.6, 113.9, 55.6, 41.1, 30.9, 27.1, 22.5, 14.1; IR(neat): 2956.0, 2931.2, 1664.1, 1601.1, 1575.3, 1259.2, 1233.1, 1168.2, 1026.6, 827.2 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{15}H_{20}ClO_2$ $[M+H]^+$ 267.1146 Found 267.1147.



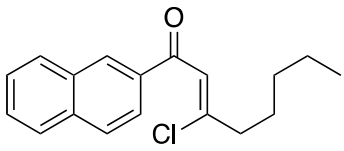
(*Z*)-1-(2-Bromophenyl)-3-chlorooct-2-en-1-one (3g): The product **3g** was prepared by the general procedure B using 2-bromobenzoyl chloride and 1-heptyne. 38 mg (24%); yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.60 (dd, *J* = 7.9, 1.0 Hz, 1H), 7.43 (dd, *J* = 7.6, 1.7 Hz, 1H), 7.38 (td, *J* = 7.6, 1.1 Hz, 1H), 7.30 (td, *J* = 7.7, 1.7 Hz, 1H), 6.65 (t, *J* = 0.9 Hz, 1H), 2.50 (td, *J* = 7.5, 0.8 Hz, 2H), 1.66-1.68 (m, 2H), 1.33-1.36 (m, 4H), 0.91 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 191.6, 150.3, 141.9, 133.6, 131.8, 129.6, 127.7, 123.8, 119.5, 41.7, 30.9, 27.1, 22.5, 14.1; IR(neat): 2955, 2929, 2859, 1682, 1653, 1600.4, 1429, 1217, 1025, 767, 736 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{14}H_{17}BrClO$ $[M+H]^+$ 315.0145 Found 315.0150.



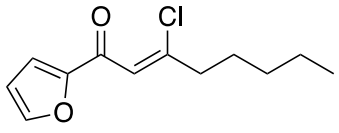
(*Z*)-1-(4-Bromophenyl)-3-chlorooct-2-en-1-one (3h): The product **3h** was prepared by the general procedure B using 4-bromobenzoyl chloride and 1-heptyne. 63 mg (40%); yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.79 (dd, *J* = 6.7, 1.9 Hz, 2H), 7.61 (dd, *J* = 6.7, 1.9 Hz, 2H), 6.76 (s, 1H), 2.53 (t, *J* = 7.2 Hz, 2H), 1.70 (d, *J* = 7.4 Hz, 2H), 1.35-1.37 (m, 4H), 0.93 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 188.9, 148.7, 136.6, 132.1, 130.3, 128.5, 120.9, 41.4, 31.0, 27.2, 22.5, 14.1; IR(neat): 2955, 2929, 2360., 1669, 1602, 1396, 1222, 1070, 1008, 814 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{14}H_{17}BrClO$ $[M+H]^+$ 315.0145 Found 315.0152.



(*Z*)-3-Chloro-1-(naphthalen-1-yl)oct-2-en-1-one (3i): The product **3i** was prepared by the general procedure B using 1-naphthoyl chloride and 1-heptyne. 63 mg (44%); yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$; δ 8.61 (d, *J* = 8.5 Hz, 1H), 7.99 (d, *J* = 8.3 Hz, 1H), 7.89 (d, *J* = 7.7 Hz, 1H), 7.83 (dd, *J* = 7.1, 1.1 Hz, 1H), 7.59-7.61 (m, 1H), 7.50-7.56 (m, 2H), 6.78 (s, 1H), 2.53 (t, *J* = 7.4 Hz, 2H), 1.68-1.72 (m, 2H), 1.34-1.38 (m, 4H), 0.93 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 193.1, 148.2, 136.4, 134.0, 132.8, 130.4, 128.7, 128.6, 128.0, 126.6, 125.8, 124.6, 124.5, 41.3, 31.0, 27.1, 22.5, 14.1; IR(neat): 2955, 2929, 2341, 1668, 1653, 1596, 1507, 804, 777 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{18}H_{20}ClO$ $[M+H]^+$ 287.1197 Found 287.1201.

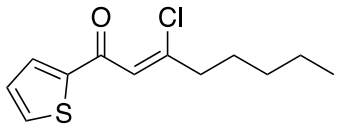


(*Z*)-3-Chloro-1-(naphthalen-2-yl)oct-2-en-1-one (3j): The product **3j** was prepared by the general procedure B using 2-naphthoyl chloride and 1-heptyne. 40 mg (28%); yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.43 (d, *J* = 0.9 Hz, 1H), 8.03 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.97 (d, *J* = 7.7 Hz, 1H), 7.92 (d, *J* = 8.7 Hz, 1H), 7.89 (d, *J* = 8.3 Hz, 1H), 7.60-7.63 (m, 1H), 7.55-7.58 (m, 1H), 6.95 (t, *J* = 0.9 Hz, 1H), 2.58-2.61 (m, 2H), 1.74-1.76 (m, 2H), 1.40-1.43 (m, 4H), 0.96 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 190.0, 147.7, 135.8, 135.2, 132.7, 130.6, 129.7, 128.7(4), 128.7(1), 128.0, 127.0, 124.4, 121.6, 41.3, 31.0, 27.2, 22.6, 14.2; IR(neat): 2955, 2917, 2849, 2360, 1669, 1653, 1558, 1219, 1185, 772, 668 cm^{-1}; HRMS(ESI): m/z calcd. For $C_{18}H_{20}ClO$ $[M+H]^+$ 287.1197 Found 287.1204.

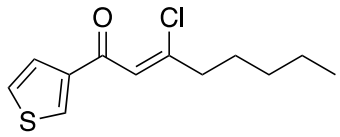


(*Z*)-3-Chloro-1-(furan-2-yl)oct-2-en-1-one (3k) : The product **3k** was prepared by the general procedure B using 2-furanoyl chloride and 1-heptyne. 18 mg (16%); yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.59 (m, 1H), 7.22 (d, *J* = 3.6 Hz, 1H), 6.92 (s, 1H), 6.55 (dd, *J* = 3.6, 1.8 Hz, 1H), 2.52 (t, *J* = 7.5 Hz, 2H), 1.67-1.71 (m, 2H), 1.33-1.37 (m, 4H), 0.92 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 176.5, 153.7, 150.6, 146.4, 119.2, 117.4, 112.7, 41.9, 30.9, 27.2, 22.5, 14.1; IR(neat): 2956, 2929, 1664,

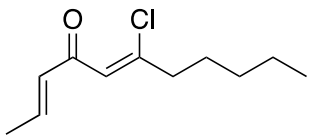
1609, 1568, 1466, 1394, 1254, 1011, 761 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{12}H_{16}ClO_2$ $[M+H]^+$ 227.0833 Found 227.0835.



(*Z*)-3-Chloro-1-(thiophen-2-yl)oct-2-en-1-one (3l) : The product **3l** was prepared by the general procedure B using 2-thenoyl chloride and 1-heptyne. 25 mg (21%); brown liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.70 (dd, *J* = 3.8, 1.0 Hz, 1H), 7.65 (dd, *J* = 4.8, 1.0 Hz, 1H), 7.13 (dd, *J* = 4.9, 3.8 Hz, 1H), 6.83 (s, 1H), 2.52 (t, *J* = 7.6 Hz, 2H), 1.67-1.72 (m, 2H), 1.34-1.37 (m, 4H), 0.92 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 181.2, 149.1, 145.6, 134.2, 132.1, 128.3, 120.3, 41.5, 30.9, 27.2, 22.5, 14.1; IR(neat): 2955, 2930, 1648, 1603, 1516, 1414, 1353, 1237, 721 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{12}H_{16}ClOS$ $[M+H]^+$ 243.0604 Found 243.0608.

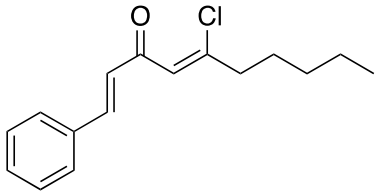


(*Z*)-3-Chloro-1-(thiophen-3-yl)oct-2-en-1-one (3m): The product **3m** was prepared by the general procedure B using 3-thenoyl chloride and 1-heptyne. 24 mg (20%); brown liquid; ^{1}H NMR (600 MHz, $CDCl_3$); δ 8.03 (dd , *J* = 2.4, 1.2 Hz, 1H), 7.57 (dd, *J* = 5.0, 1.2 Hz, 1H), 7.33 (dd, *J* = 5.0, 2.9 Hz, 1H), 6.79 (s, 1H), 2.52 (t, *J* = 7.6 Hz, 2H), 1.67-1.70 (m, 2H), 1.33-1.37 (m, 4H), 0.92 (t, *J* = 7.1 Hz, 3H) ^{13}C NMR (150 MHz, $CDCl_3$) δ 183.1, 148.4, 143.2, 132.5, 127.3, 126.6, 121.4, 41.5, 30.9, 27.2, 22.5, 14.1; IR(neat): 2955, 2929, 2360, 2341, 1662, 1654, 1604, 1231, 772, 700, 668 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{12}H_{16}ClOS$ $[M+H]^+$ 243.0604 Found 243.0607.

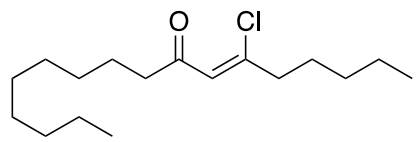


(2*E*,5*Z*)-6-Chloroundeca-2,5-dien-4-one (3n): The product **3n** was prepared by the general procedure B using (*E*)-butenoyl chloride and 1-heptyne. 31 mg (31%); yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 6.85-6.91 (dq, *J* = 15.3, 6.5 Hz, 1H), 6.35 (t, *J* = 0.9 Hz, 1H), 6.28 (dq, *J* = 15.6, 1.6 Hz, 1H), 2.43 (t, *J* = 7.2, 0.8 Hz, 2H), 1.92 (dd, *J* = 6.9, 1.5 Hz, 3H), 1.60-1.65 (m, 2H), 1.28-1.35 (m, 4H), 0.90 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 188.9, 146.8, 144.2, 132.5, 122.9, 41.2, 30.9, 27.1, 22.5, 18.5,

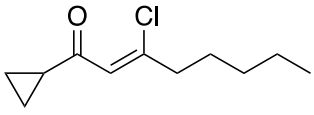
14.1; IR(neat): 2956, 2932, 1683, 1664, 1631, 1605, 1441, 1212, 1143, 969 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{11}H_{18}ClO$ $[M+H]^+$ 201.1040 Found 201.1043.



(1*E*,4*Z*)-5-Chloro-1-phenyldeca-1,4-dien-3-one (3o): The product **3o** was prepared by the general procedure B using (*E*)-cinnamoyl chloride and 1-heptyne. 22 mg (17%); brown liquid; ^1H NMR (600 MHz, $CDCl_3$): δ 7.61 (d, *J* = 16.0 Hz, 1H), 7.57-7.58 (m, 2H), 7.40-7.42 (m, 3H), 6.95 (d, *J* = 15.9 Hz, 1H), 6.48 (t, *J* = 0.8 Hz, 1H), 2.49-2.51 (td, *J* = 7.1, 0.7 Hz, 2H), 1.66-1.71 (m, 2H), 1.33-1.39 (m, 4H), 0.93 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 188.7, 147.5, 143.8, 134.8, 130.7, 129.1, 128.6, 127.0, 123.7, 41.4, 30.9, 27.1, 22.5, 14.1; IR(neat): 2955, 2930, 1653, 1635, 1615, 1506, 1331, 1197, 1134, 978, 771, 698 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{16}H_{20}ClO$ $[M+H]^+$ 263.1197 Found 263.1201.

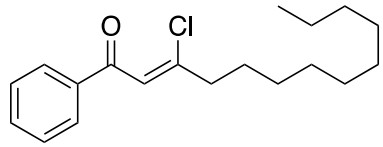


(*Z*)-6-Chloroheptadec-6-en-8-one (3p): The product **3p** was prepared by the general procedure B using decanoyl chloride and 1-heptyne. 42 mg (29%); brown liquid; ^1H NMR (600 MHz, $CDCl_3$): δ 6.24 (s, 1H), 2.57 (t, *J* = 7.6 Hz, 2H), 2.41 (t, *J* = 7.6 Hz, 2H), 1.59-1.63 (m, 4H), 1.25-1.31 (m, 16H), 0.86-0.91 (m, 6H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 199.2, 146.8, 124.2, 44.2, 41.5, 32.0, 30.9, 29.6(2C), 29.4(1), 29.3(5), 27.1, 24.1, 22.8, 22.5, 14.2, 14.0; IR(neat): 2955, 2926, 2855, 1700, 1615, 1457 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{17}H_{32}ClO$ $[M+H]^+$ 287.2136 Found 287.2140.

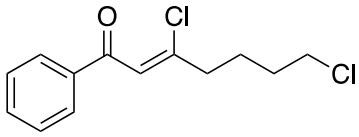


(*Z*)-3-Chloro-1-cyclopropyloct-2-en-1-one (3q): The product **3q** was prepared by the general procedure B using cyclopropanecarbonyl chloride and 1-heptyne. 18 mg (18%); yellow liquid; ^1H NMR (600 MHz, $CDCl_3$): δ 6.36 (t, *J* = 0.8 Hz, 1H), 2.44 (td, *J* = 7.4, 0.7 Hz, 2H), 2.16-2.18 (m, 1H), 1.64 (m, 2H), 1.30-1.35 (m, 4H), 1.11-1.13 (m, 2H), 0.93-0.95 (m, 2H), 0.91 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 199.2, 146.5, 124.4, 41.4, 30.9, 27.1, 22.5, 22.3, 14.1, 12.0; IR(neat): 2957, 2931, 1683, 1653, 1609,

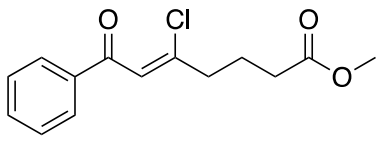
1383, 1133, 1113, 772, 668 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{11}H_{18}ClO$ $[M+H]^+$ 201.1040 Found 201.1042.



(*Z*)-3-Chloro-1-phenyltridec-2-en-1-one (3r): The product **3r** was prepared by the general procedure B using benzoyl chloride and 1-dodecyne. 67 mg (44%); yellow liquid; ^1H NMR (600 MHz $CDCl_3$): δ 7.93 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.57 (m, 1H), 7.46-7.48 (m, 2H), 6.82 (s, 1H), 2.54 (t, *J* = 7.2 Hz, 2H), 1.67-1.72 (m, 2H), 1.27-1.38 (m, 14H), 0.88 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 189.9, 147.9, 137.8, 133.3, 128.8, 128.7, 121.3, 41.3, 32.0, 29.7, 29.6, 29.4(2C), 28.8, 27.5, 22.8, 14.3; IR(neat): 2925, 2854, 1669, 1603, 1448, 1225, 773, 701 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{19}H_{28}ClO$ $[M+H]^+$ 307.1823 Found 307.1834.

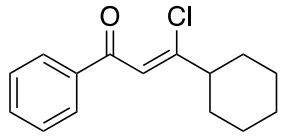


(*Z*)-3,7-Dichloro-1-phenylhept-2-en-1-one (3s): The product **3s** was prepared by the general procedure B using benzoyl chloride and 6-chloro-1-hexyne. 64 mg (50%); yellow liquid; ^1H NMR (600 MHz, $CDCl_3$): δ 7.93 (dd, *J* = 8.3, 1.4 Hz, 2H), 7.56-7.59 (m, 1H), 7.46-7.49 (m, 2H), 6.85 (s, 1H), 3.59 (t, *J* = 6.2 Hz, 2H), 2.58 (t, *J* = 6.5 Hz, 2H), 1.87 (m, 4H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 189.8, 146.6, 137.6, 133.4, 128.8, 128.7, 121.8, 44.6, 40.4, 31.5, 24.7; IR(neat): 2955, 1669, 1604, 1448, 1225, 772, 703, 688, 649 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{13}H_{15}Cl_2O$ $[M+H]^+$ 257.0494 Found 257.0497.

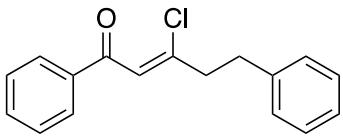


(*Z*)-Methyl-5-chloro-7-oxo-7-phenylhept-5-enoate (3t): The product **3t** (**3t**:**4t** = **2**:1 mixture) was prepared by the general procedure B using benzoyl chloride and methyl 5-hexynoate. 32 mg (24%-mixture); yellow liquid; ^1H NMR (600 MHz, $CDCl_3$): δ 7.92 (dd, *J* = 8.3, 1.3 Hz, 2H), 7.55-7.58 (m, 1H), 7.45-7.48 (m, 2H), 6.84 (t, *J* = 0.9 Hz, 1H), 3.69 (s, 3H), 2.60 (td, *J* = 7.4, 0.8 Hz, 2H), 2.41 (t, *J* = 7.2 Hz, 2H), 2.01-2.06 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 189.7, 173.4, 137.6, 133.4, 128.8,

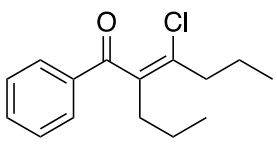
128.7, 122.2, 51.9, 40.3, 32.7, 22.6; IR(neat): 2951, 1669, 1604, 1448, 1225, 772, 703, 688 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{14}H_{16}ClO_3$ $[M+H]^+$ 267.0782 Found 267.0785.



(*Z*)-3-Chloro-3-cyclohexyl-1-phenylprop-2-en-1-one (3u): The product **3u** was prepared by the general procedure B using benzoyl chloride and cyclohexylacetylene. 84 mg (68%); pale brown liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.93 (dd, *J* = 8.3, 1.3 Hz, 2H), 7.55-7.58 (m, 1H), 7.45-7.48 (m, 2H), 6.77 (d, *J* = 0.7 Hz, 1H), 2.38 (tt, *J* = 11.5, 3.2 Hz, 1H), 2.00 (m, 2H), 1.84-1.87 (m, 2H), 1.74 (m, 1H), 1.42-1.49 (m, 2H), 1.34 (m, 2H), 1.23 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 190.6, 152.2, 137.8, 133.3, 128.8, 128.7, 119.7, 48.9, 31.6, 26.1, 25.9; IR(neat): 2930, 2854, 1668, 1600, 1448, 1221, 1016, 772, 702, 688 cm^{-1}; HRMS(ESI): m/z calcd. For $C_{15}H_{18}ClO$ $[M+H]^+$ 249.1040 Found 249.1042.



(*Z*)-3-Chloro-1,5-diphenylpent-2-en-1-one (3v): The product **3v** was prepared by the general procedure B using benzoyl chloride and 4-phenyl-1-butyne. 30 mg (22%); yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.66 (dd, *J* = 8.3, 1.1 Hz, 2H), 7.53-7.56 (m, 1H), 7.39-7.42 (m, 2H), 7.33-7.36 (m, 2H), 7.28-7.29 (m, 1H), 7.24-7.26 (m, 2H), 6.60 (s, 1H), 3.03 (t, *J* = 7.3 Hz, 2H), 2.85 (t, *J* = 7.3 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 190.1, 145.1, 139.9, 137.4, 133.3, 128.8(2C), 128.7, 128.6, 126.5, 123.0, 42.9, 33.5; IR(neat) 3027, 1669, 1603, 1448, 1226, 772, 700 cm^{-1}; HRMS(ESI): m/z calcd. for $C_{17}H_{16}ClO$ $[M+H]^+$ 271.0884 Found 271.0887.



(*Z*)-3-Chloro-1-phenyl-2-propylhex-2-en-1-one (3w): The product **3w** ((*Z*)-**3w**:**4w**:(*E*)-**3w** = 1:1:11 mixture) was prepared by the general procedure B using benzoyl chloride and 4-octyne. ^{1}H NMR and ^{13}C NMR spectra for this compound are consistent with the previously reported literature data.[4] 101mg (81%-mixture); yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.93 (dd, *J* = 7.3, 1.1 Hz, 2H), 7.53 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.2 Hz, 2H), 2.51 (t, *J* = 7.3 Hz, 2H), 2.36 (t, *J* = 7.5 Hz, 2H), 1.68-1.73 (m, 2H), 1.40-1.46 (m, 2H),

1.02 (t, J = 7.4 Hz, 3H), 0.93 (d, J = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 197.2, 136.9, 135.8, 133.5, 132.4, 128.6, 128.5, 36.6, 33.7, 21.8, 21.3, 14.5, 14.2.

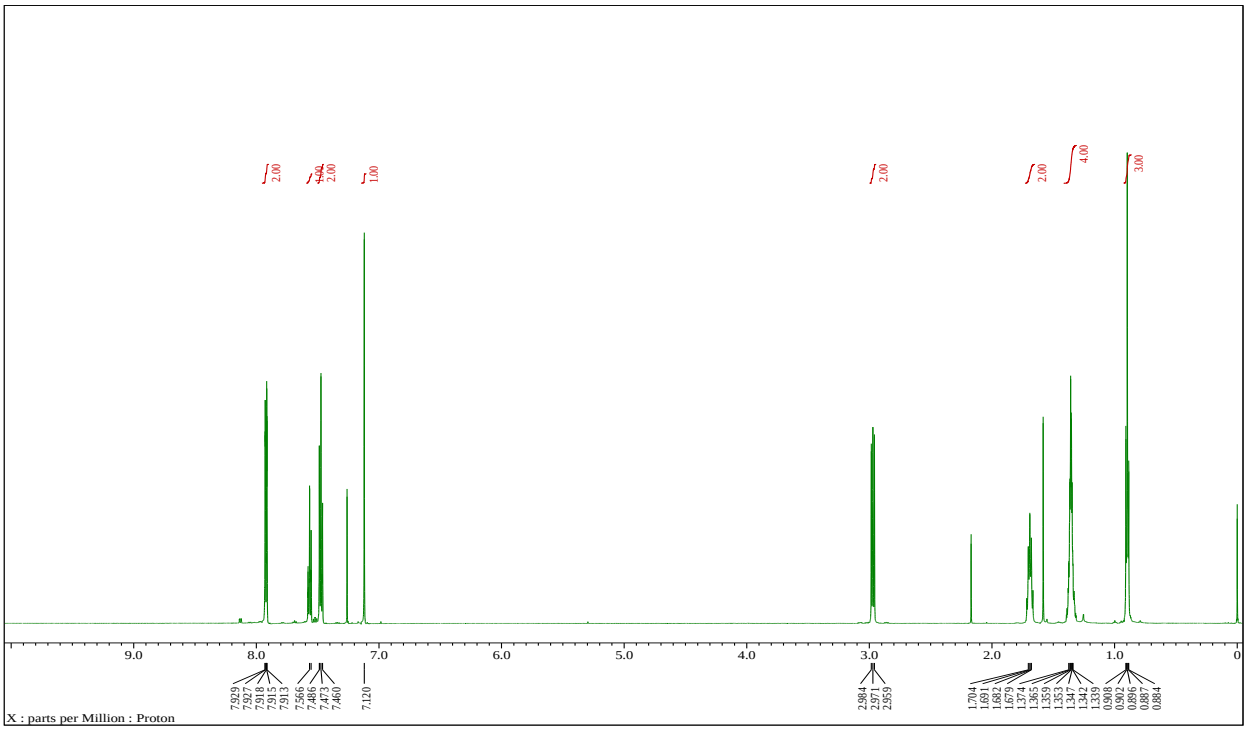
References

(1) Kim, H. Y.; Li, J. Y.; Oh, K. *J. Org. Chem.* **2012**, *77*, 11132–11145.

(2) Kim, H. Y.; Oh, K. *Org. Lett.* **2015**, *17*, 450-453.

(3) Kim, H. Y.; Rooney, E. O.; Meury, R. P.; Oh, K. *Angew. Chem., Int. Ed.* **2013**, *52*, 8026–8030.

(4) Gandeepan, P.; Parthasarathy, K.; Su, T. H.; Cheng, C. H. *Adv. Synth. Catal.* **2012**, *354*, 457–468.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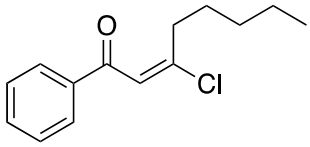
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Cl

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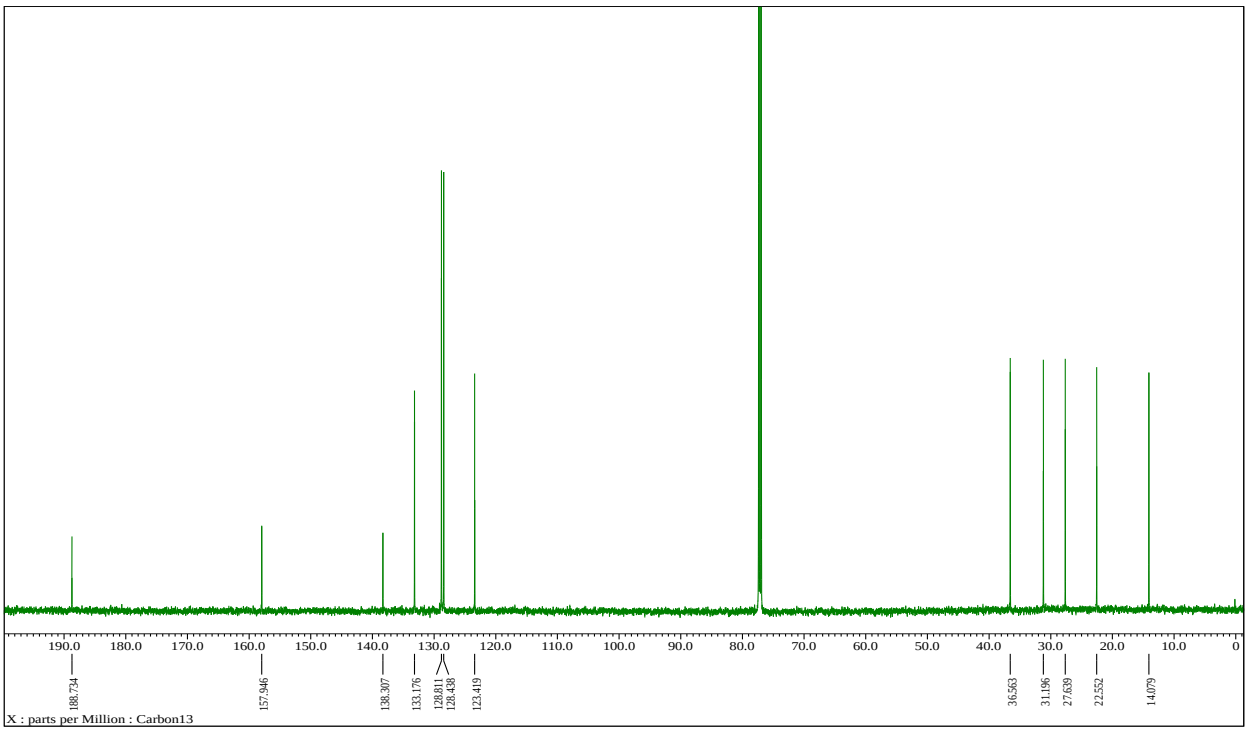
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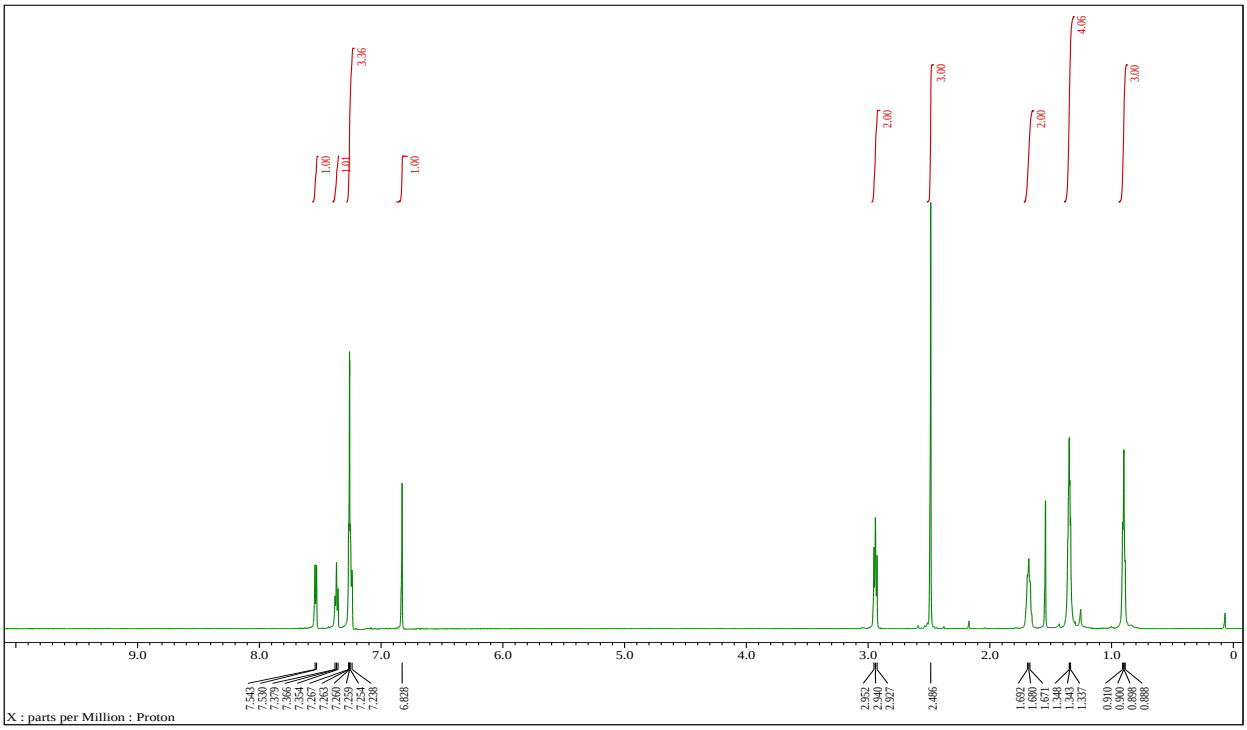
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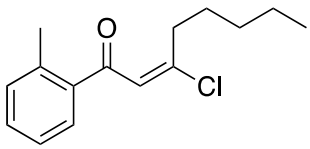
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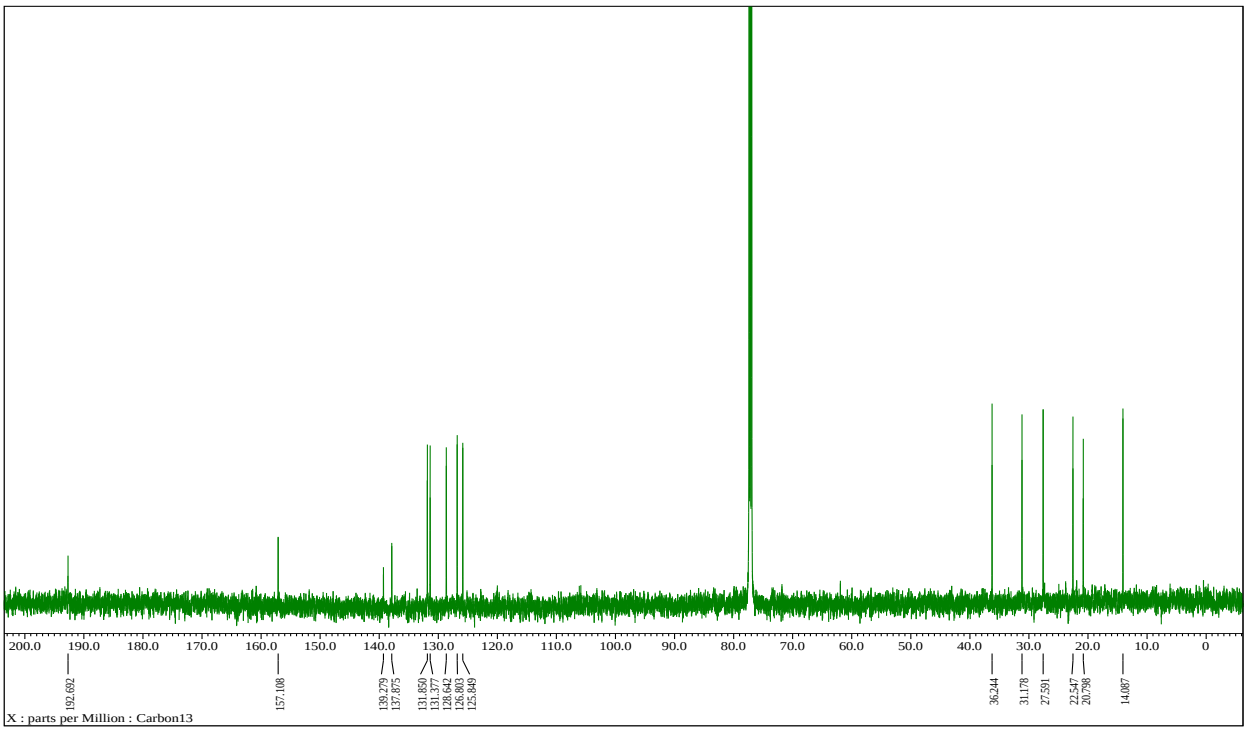
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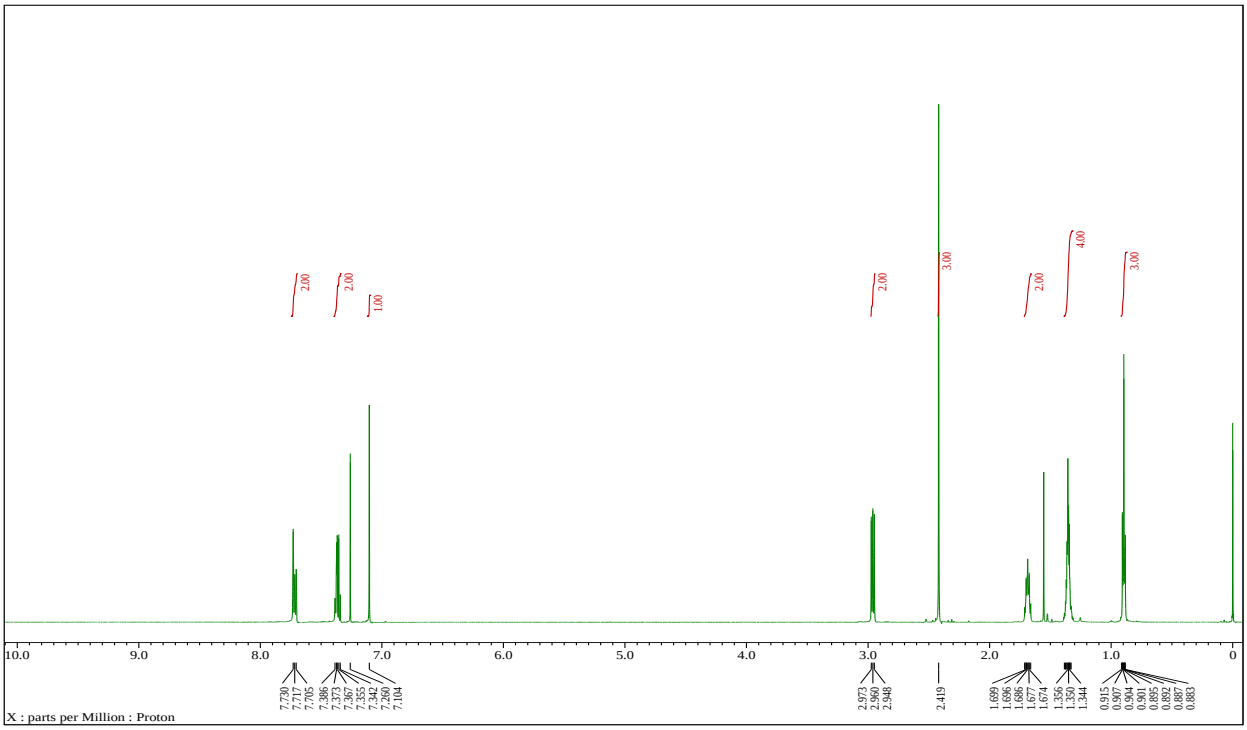
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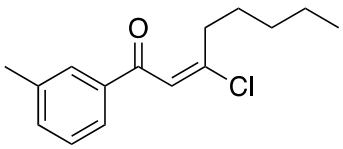
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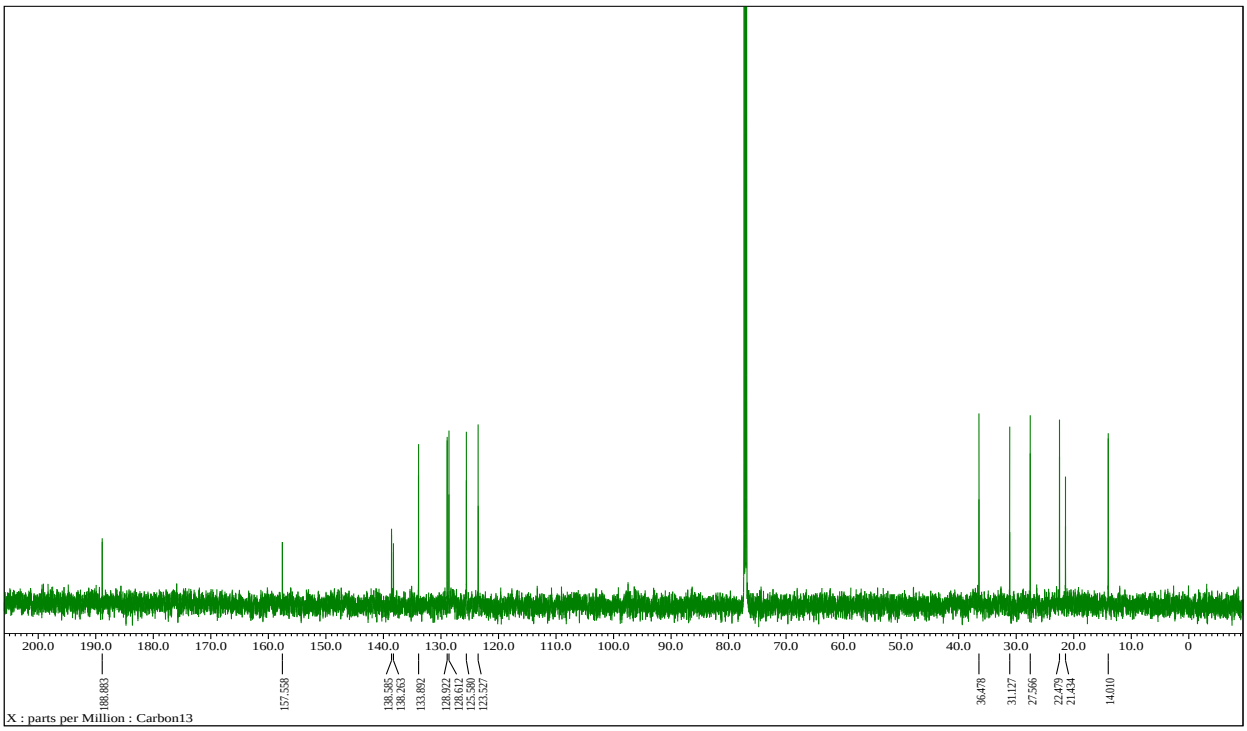
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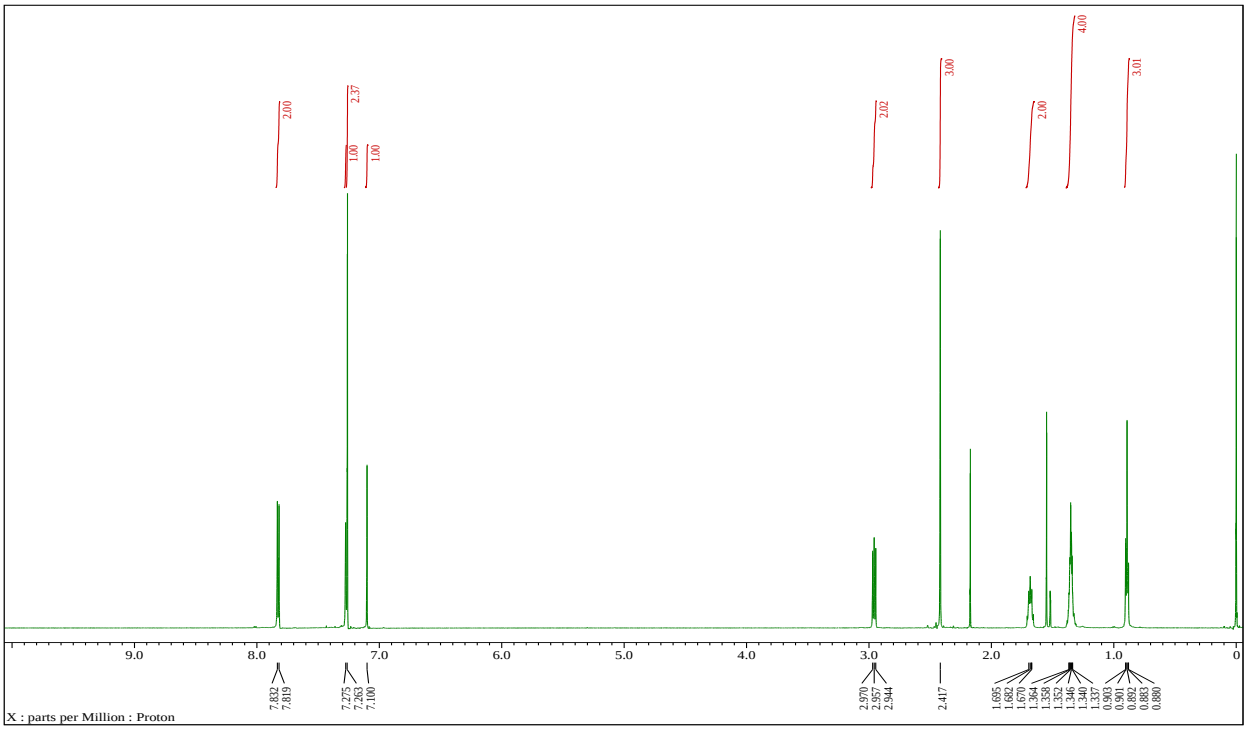
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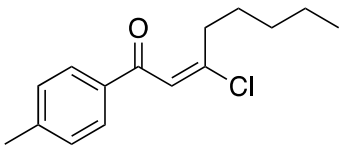
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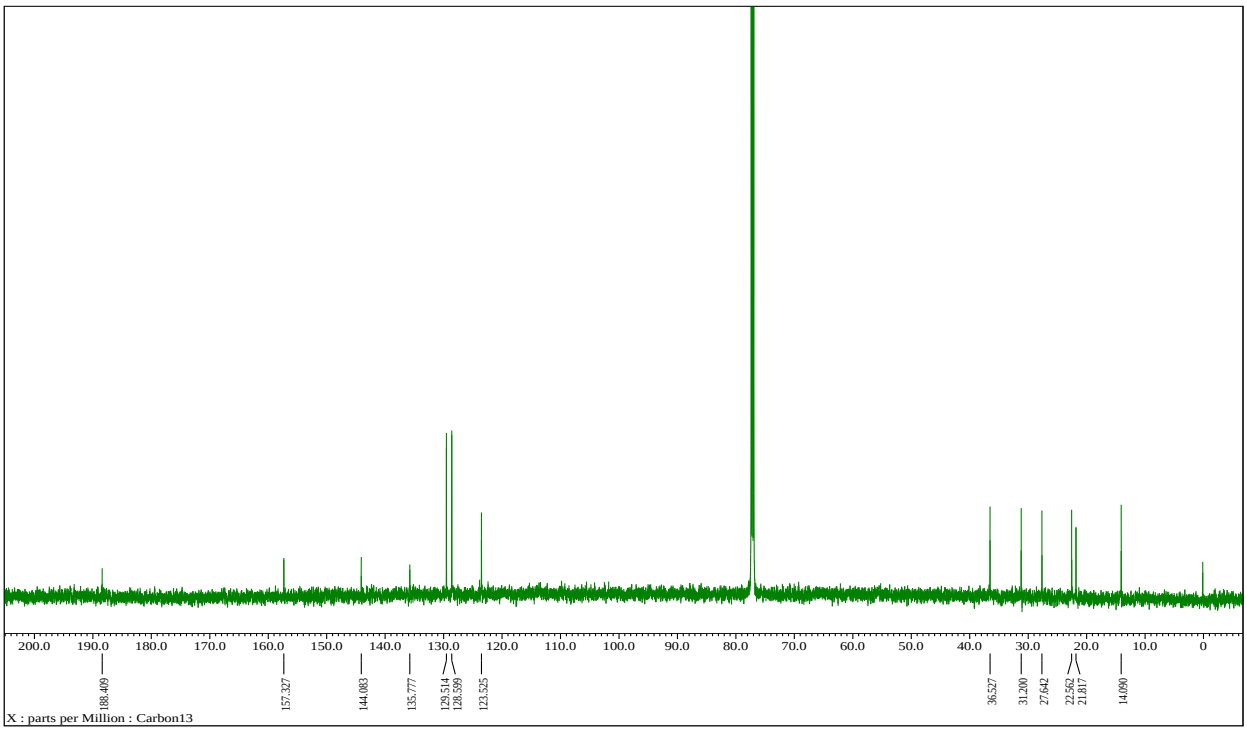
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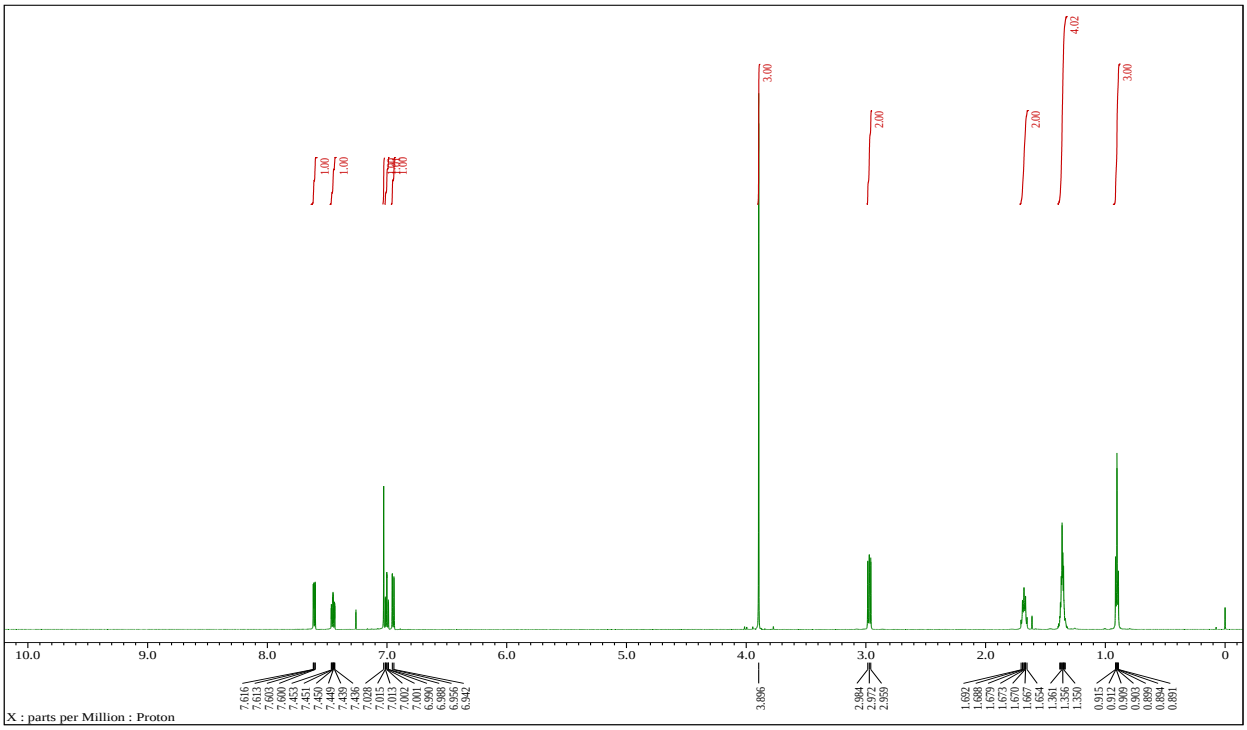
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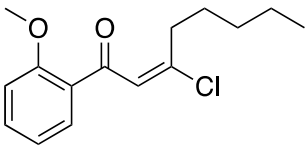
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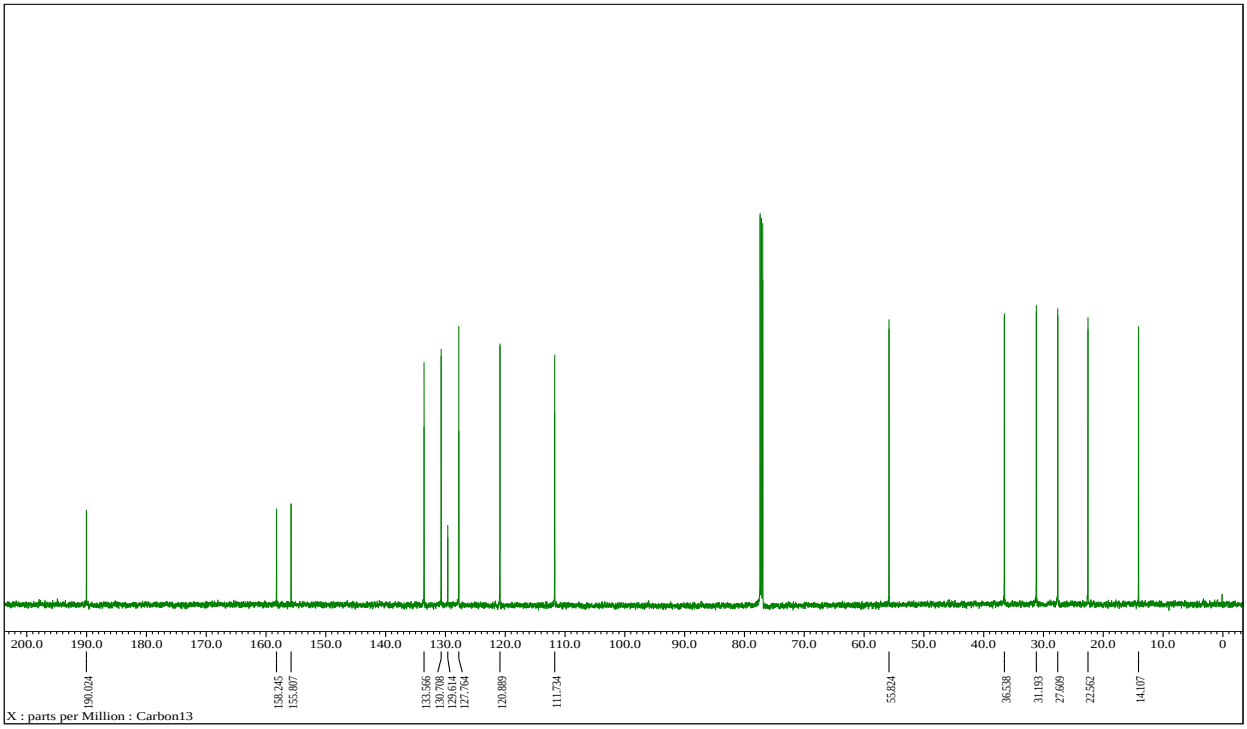
Cl

(*E*)-3e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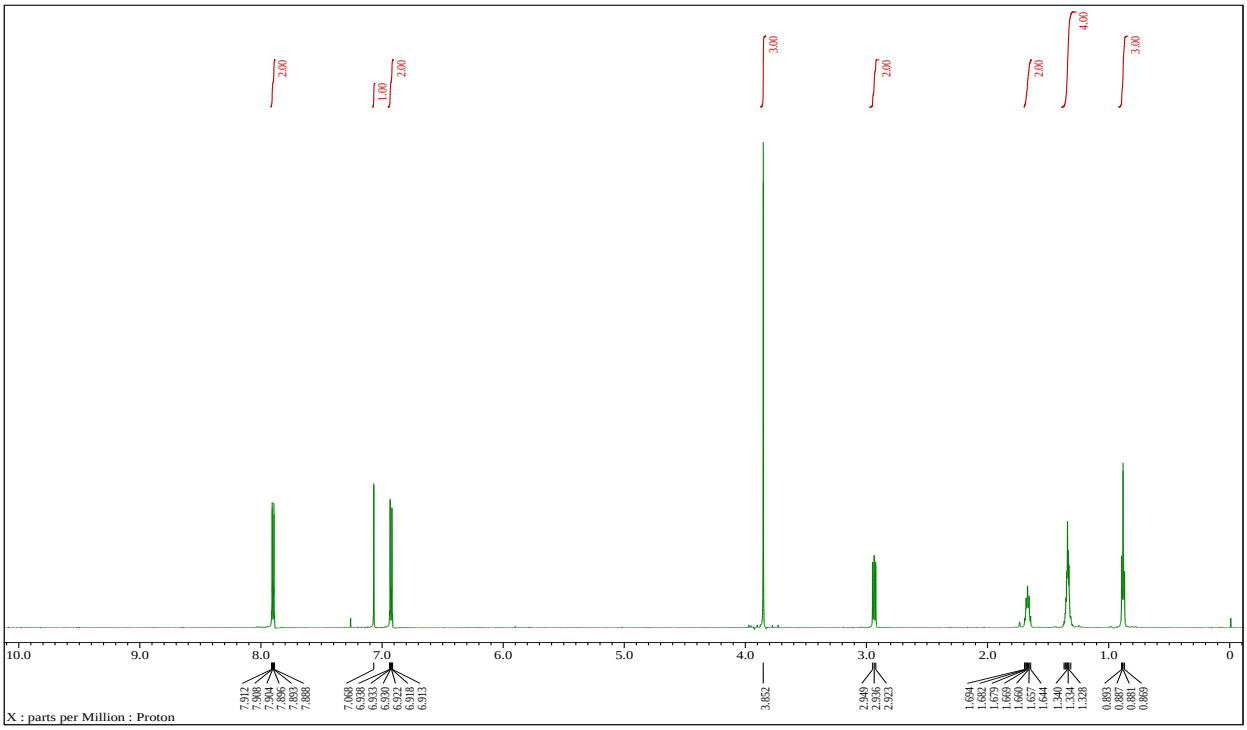
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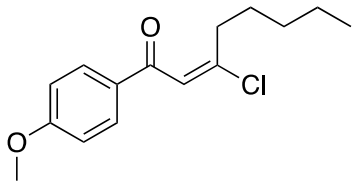
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Cl
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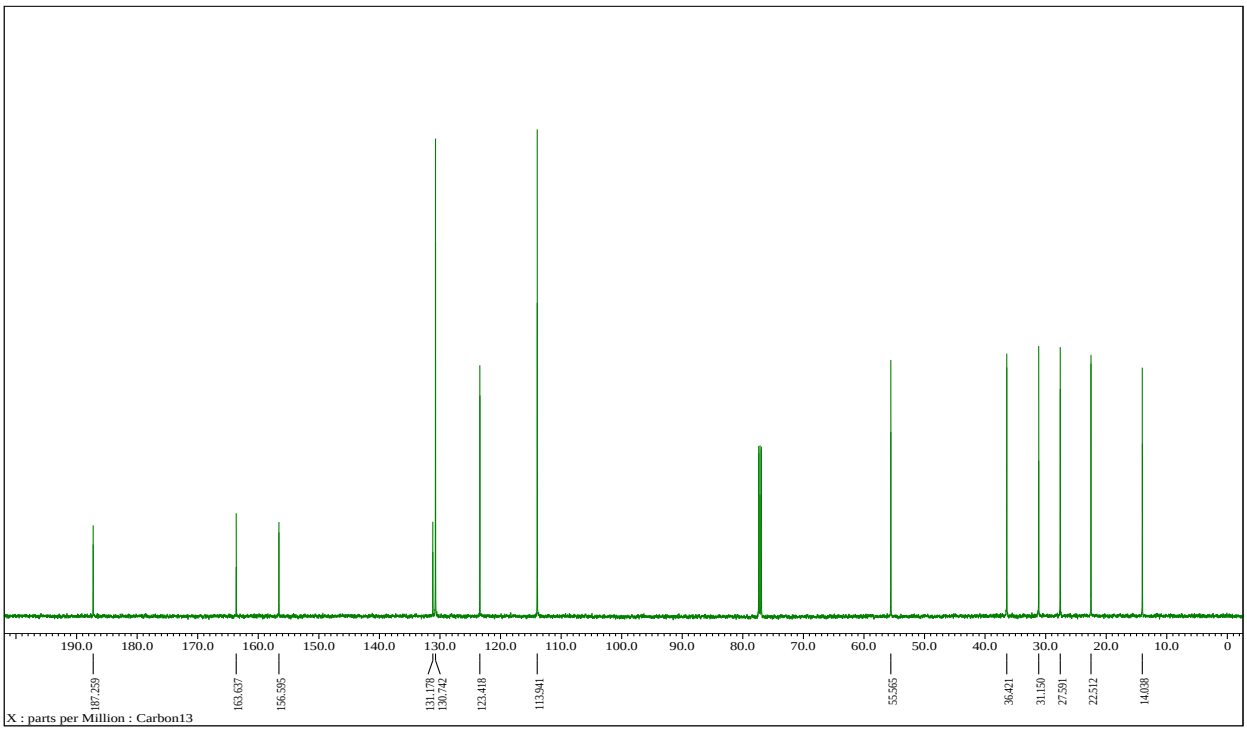
(*E*)-3f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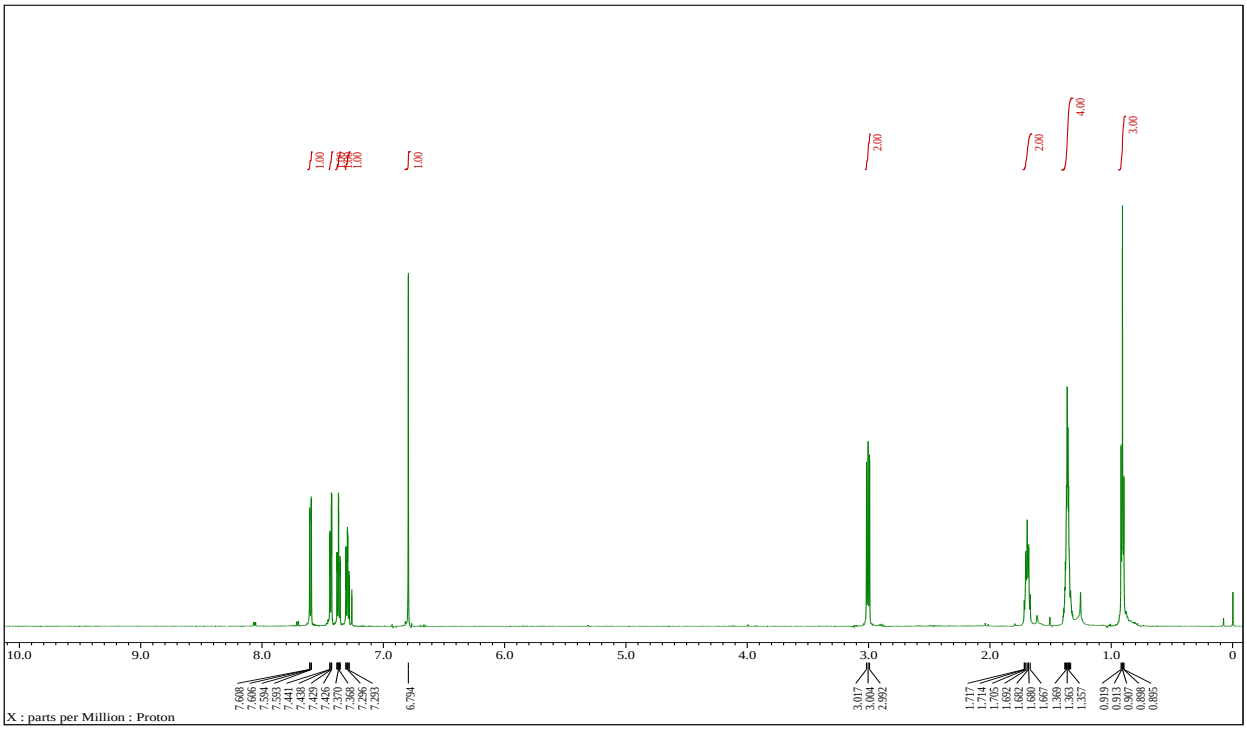
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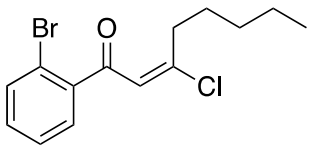
Br
O
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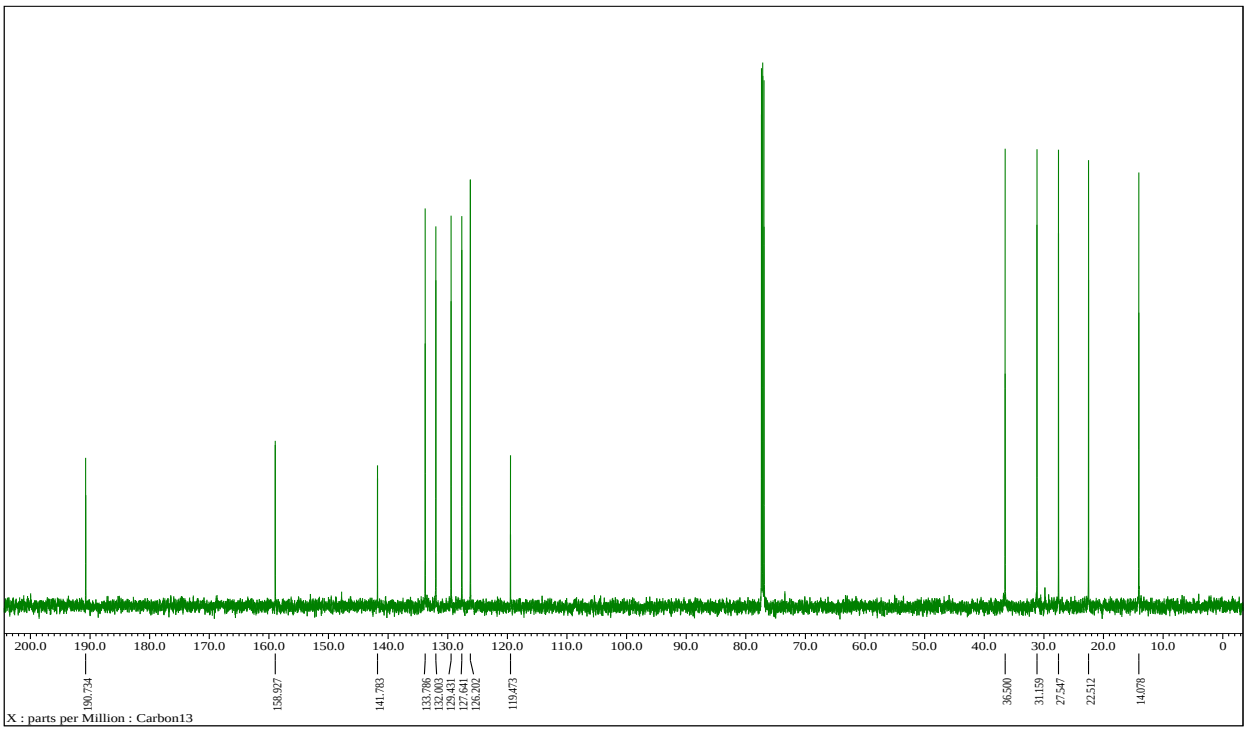
(*E*)-3g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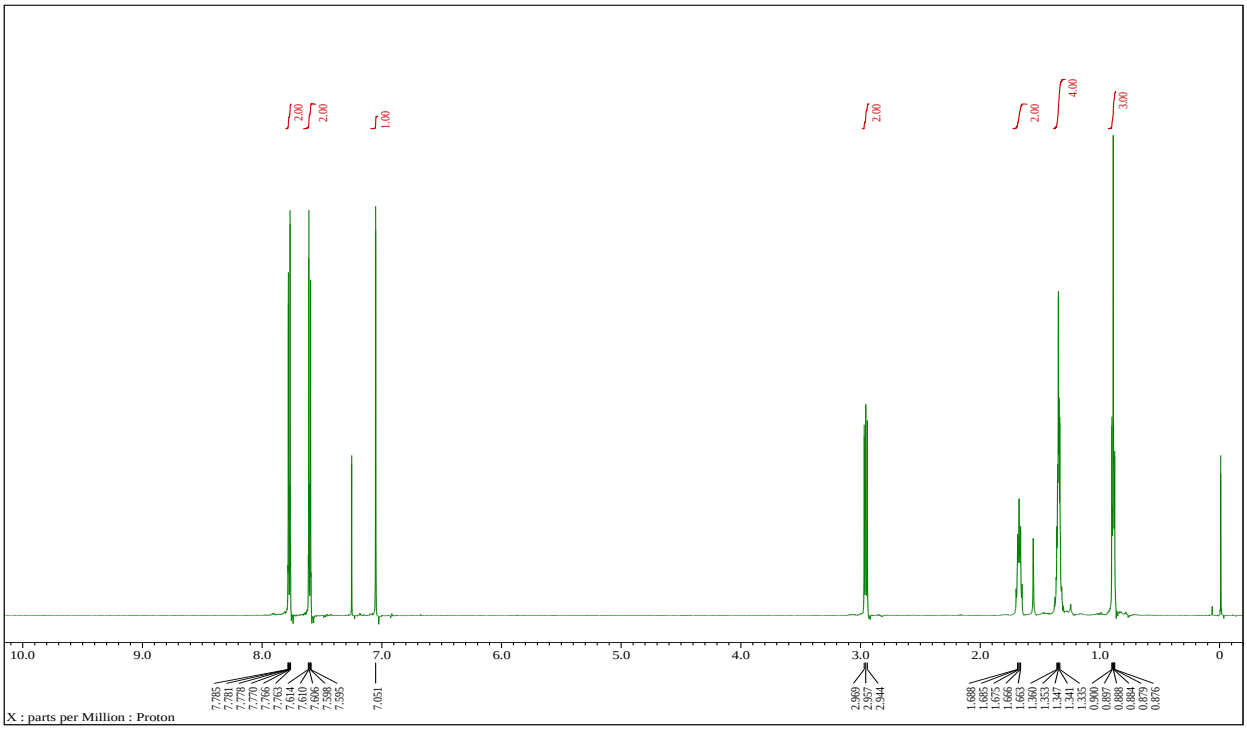
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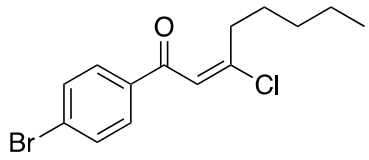
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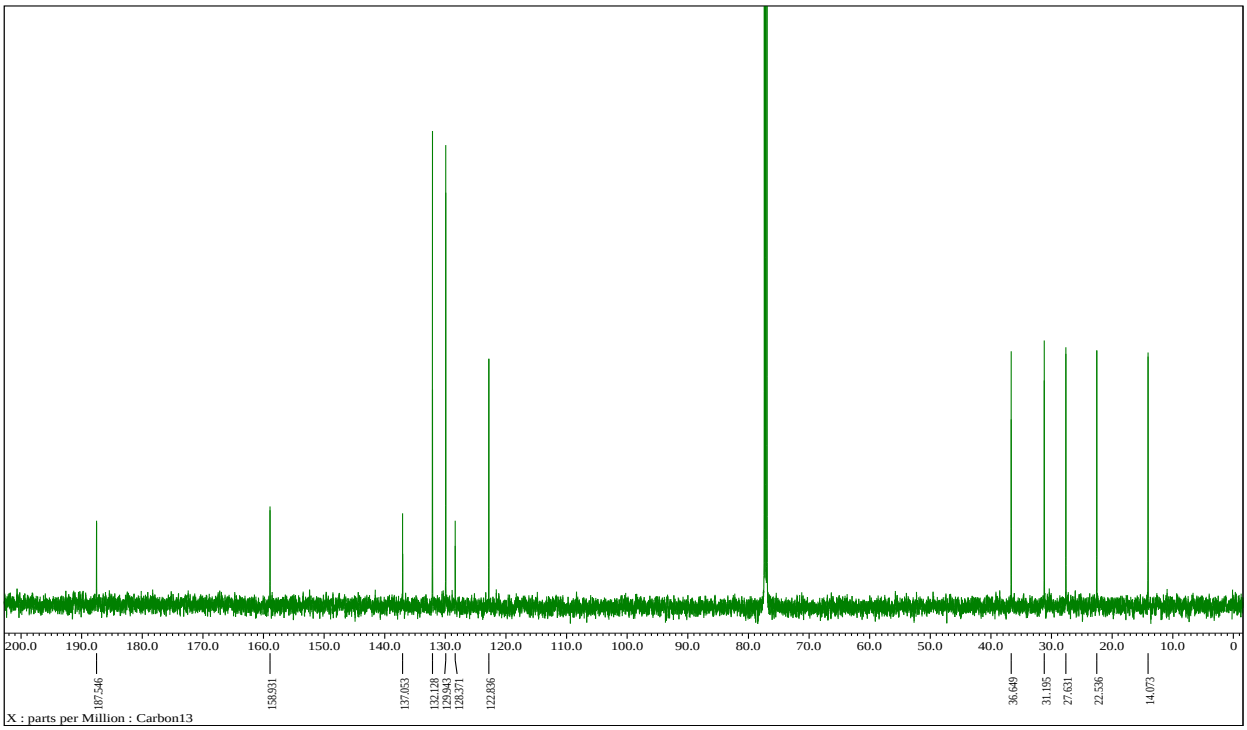
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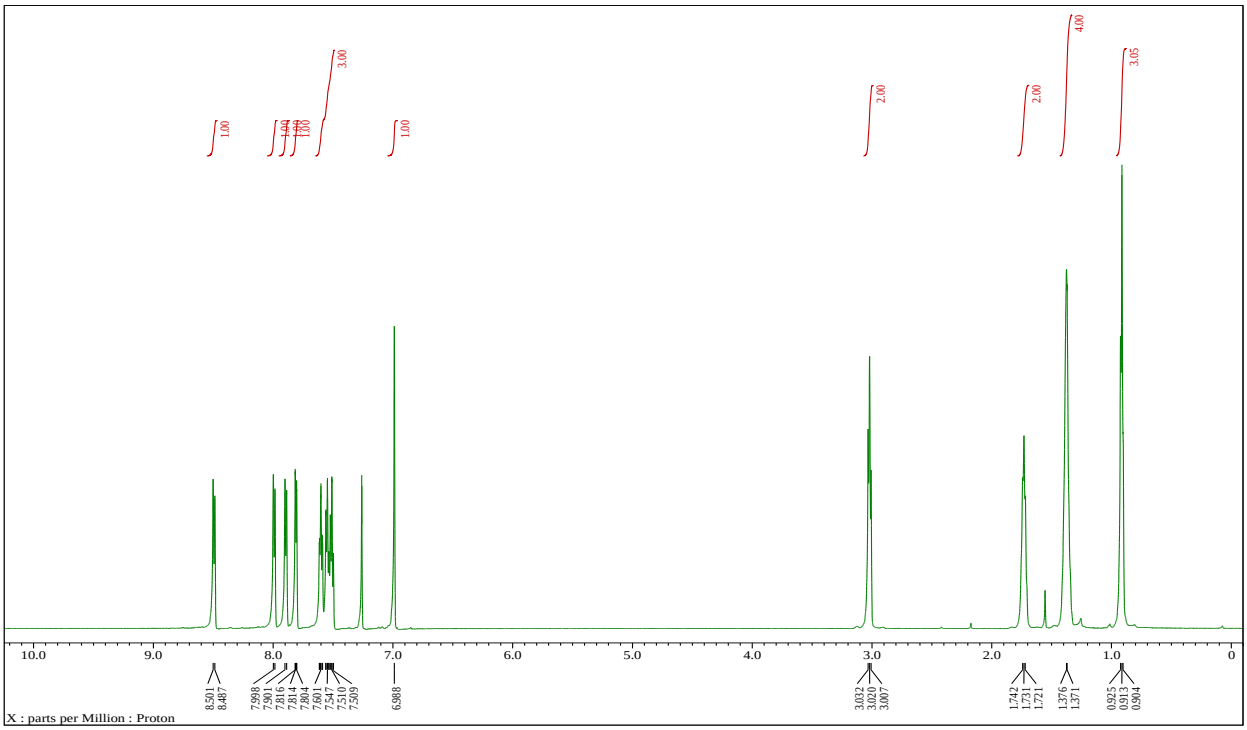
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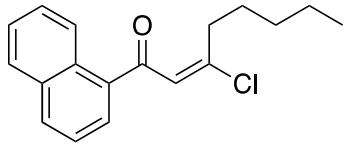
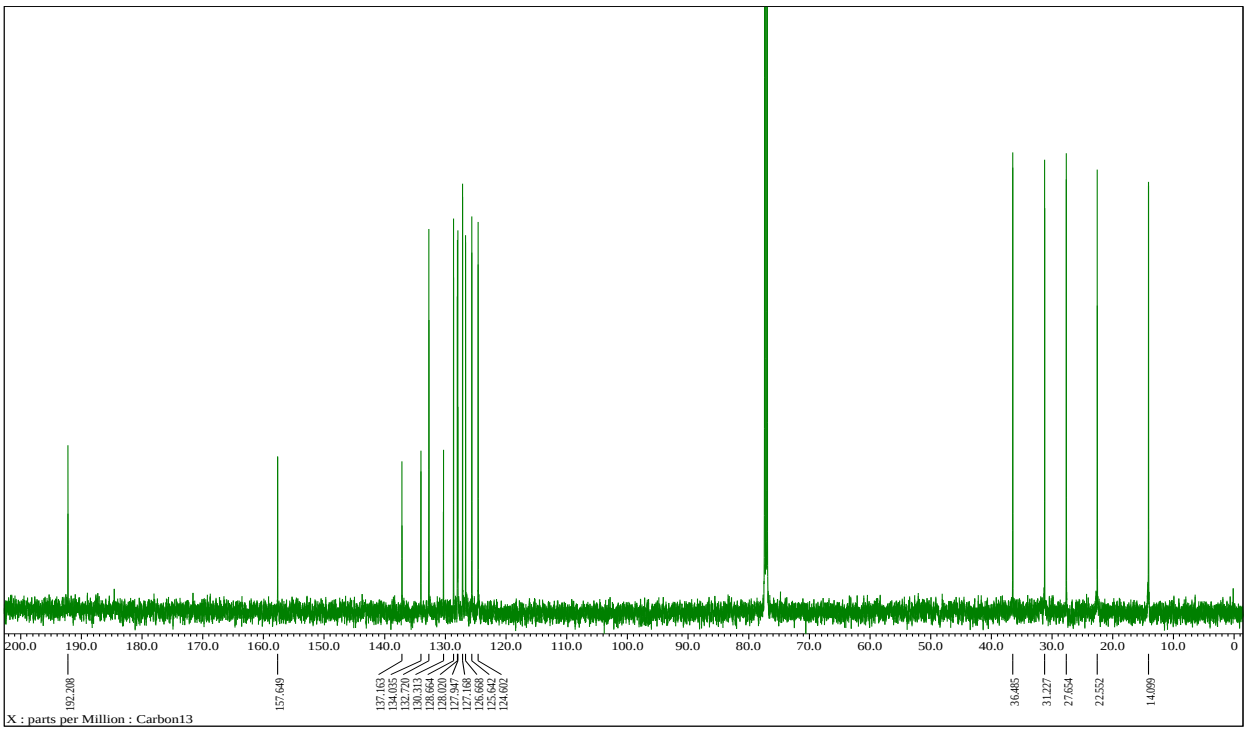
2.00
4.00
3.05
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.501
8.487
7.908
7.901
7.816
7.814
7.804
7.801
7.547
7.510
7.509
6.988
3.022
3.020
3.007
1.742
1.731
1.721
1.376
1.371
0.925
0.913
0.904
X : parts per Million : Proton


O
Cl

(*E*)-3i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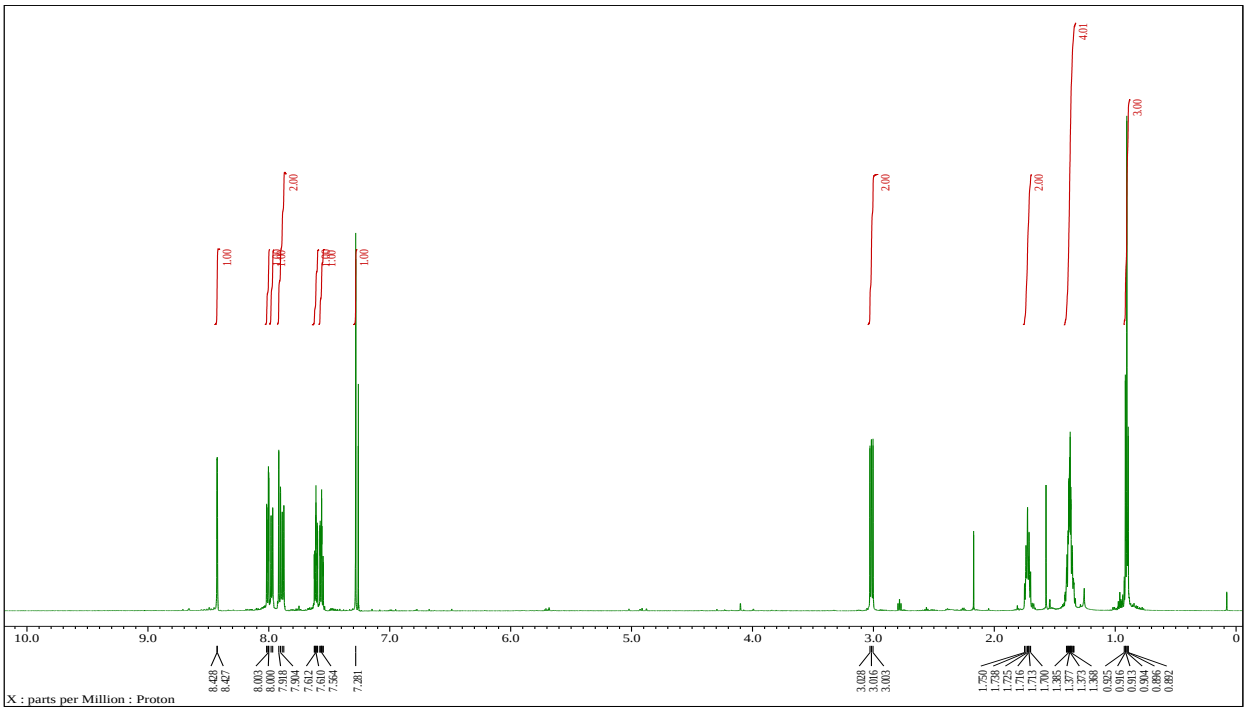
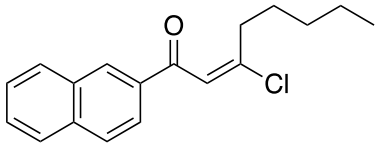
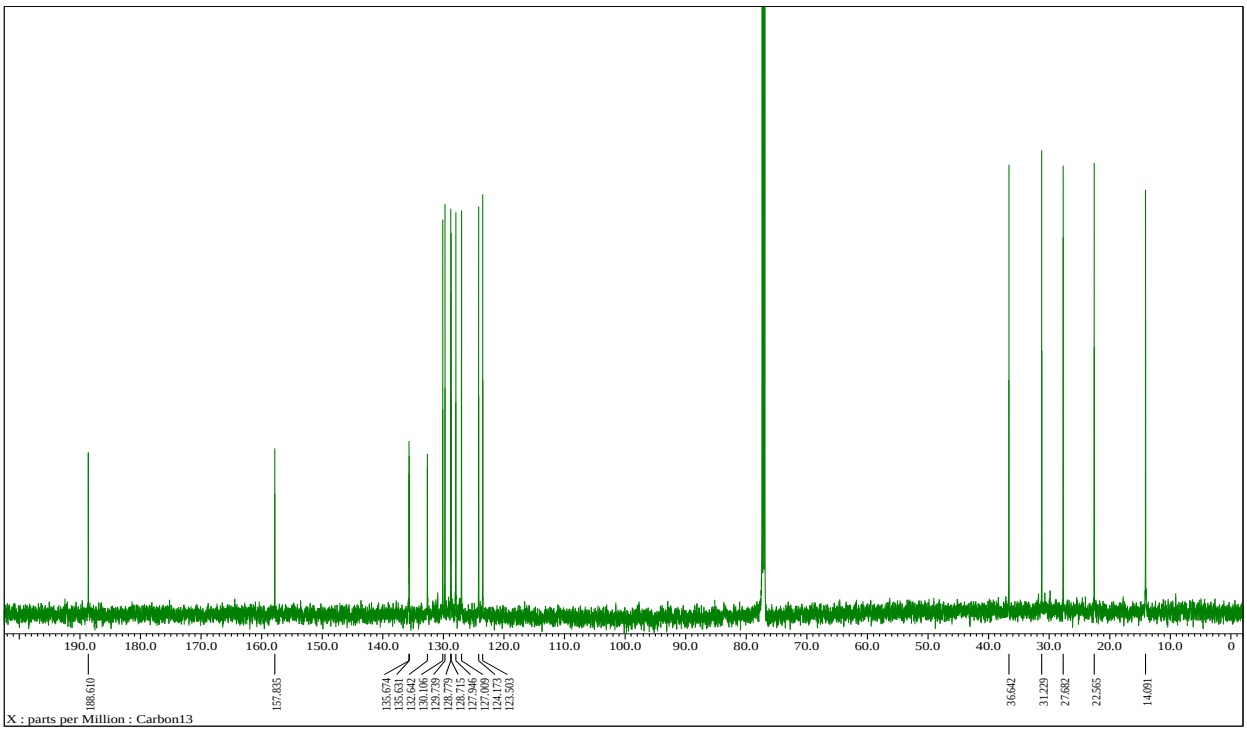
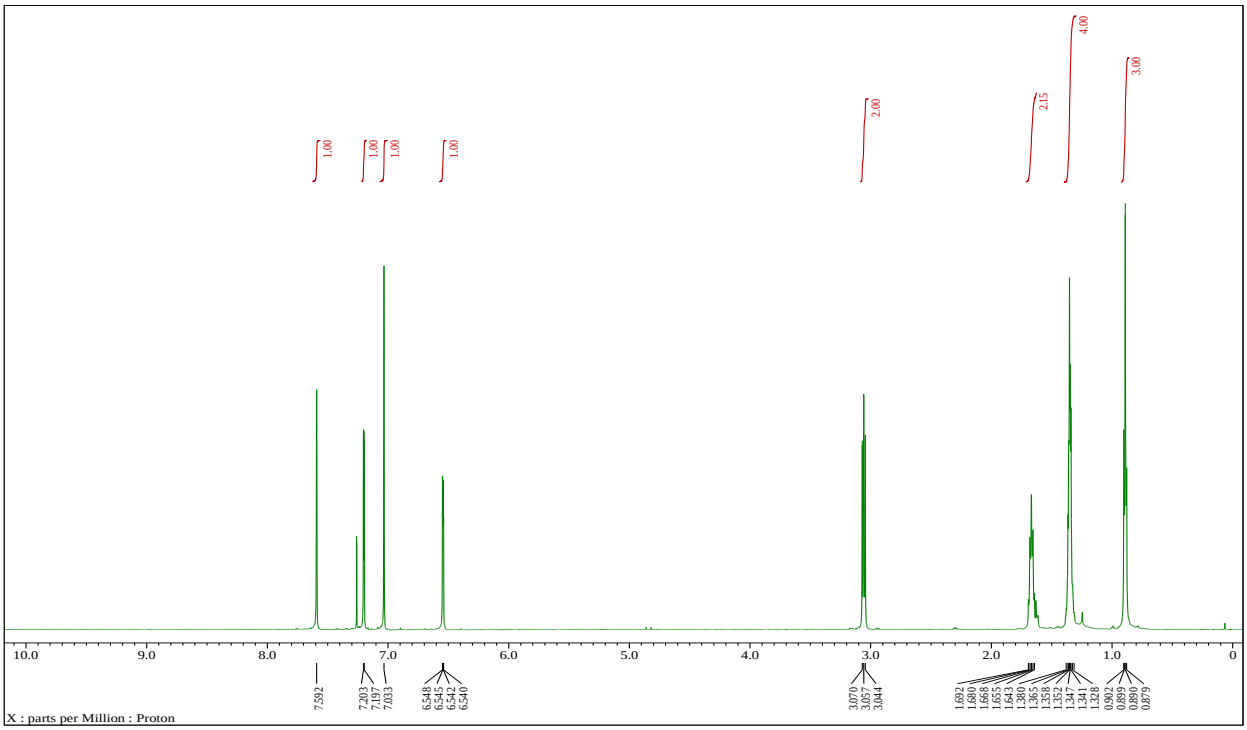
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
192.208
157.649
137.163
134.035
132.720
130.313
128.664
128.020
127.194
127.148
126.668
125.642
124.602
36.485
31.227
27.654
22.552
14.099
X : parts per Million : Carbon13

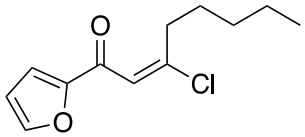




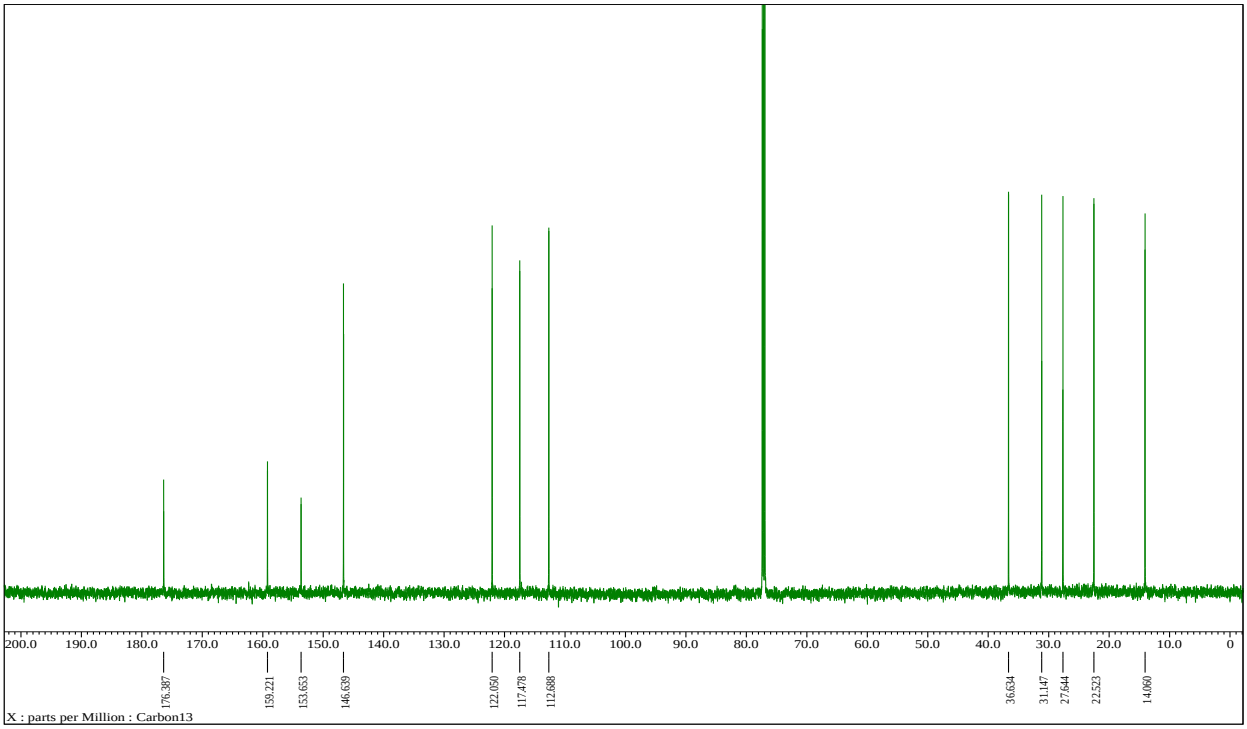
(*E*)-3j

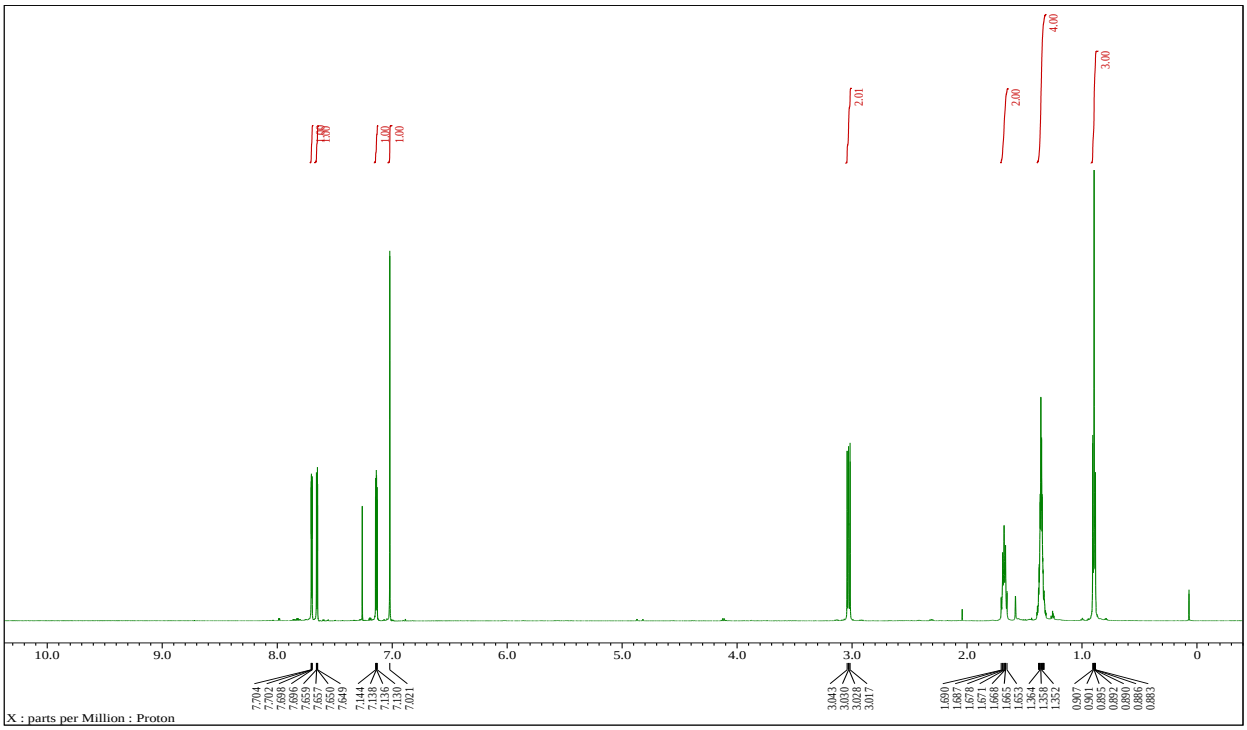


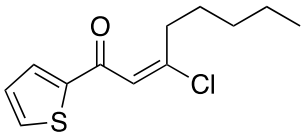

1.00
1.00
1.00
1.00
2.00
2.15
4.00
3.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.592
7.203
7.197
7.033
6.548
6.545
6.542
6.540
3.070
3.057
3.044
1.692
1.690
1.688
1.655
1.643
1.380
1.365
1.358
1.352
1.347
1.341
1.328
0.902
0.899
0.890
0.879
X : parts per Million : Proton


O
Cl
O

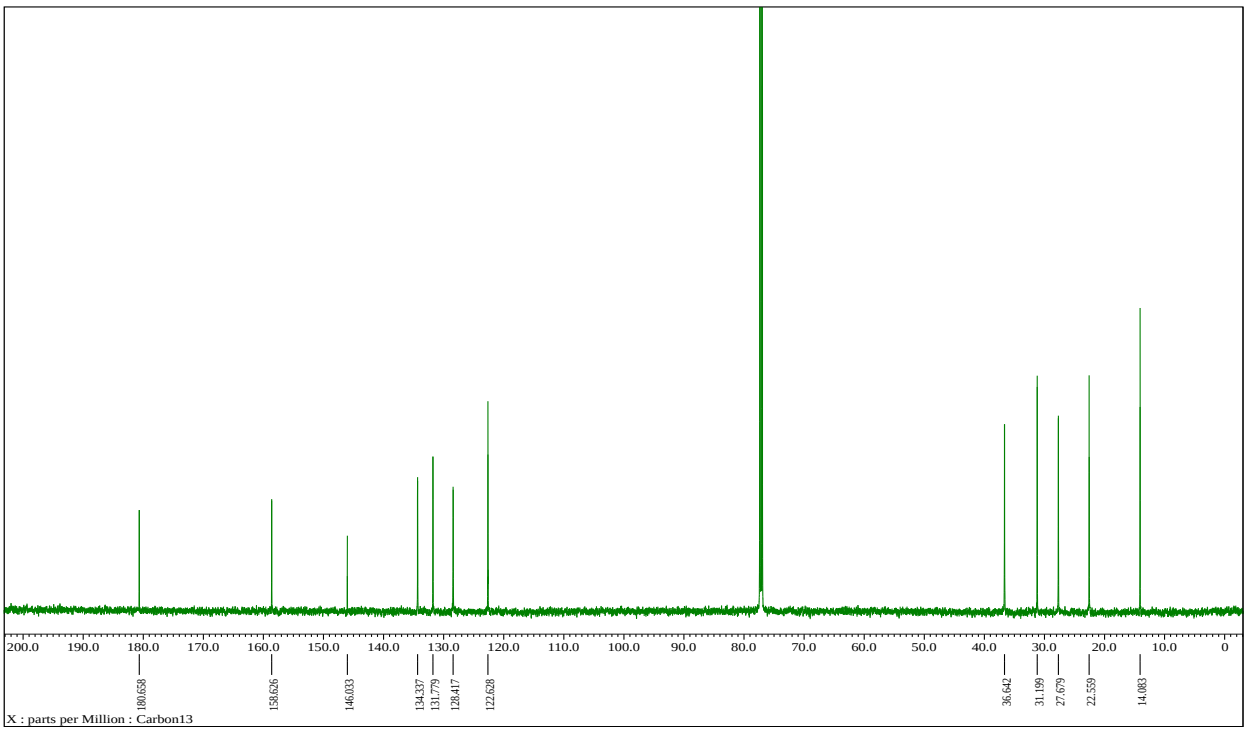
(*E*)-3k


200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
176.387
159.221
153.653
146.639
122.050
117.478
112.688
36.634
31.147
27.644
22.523
14.060
X : parts per Million : Carbon13


4.00
3.00
2.01
2.00
1.00
1.00
1.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.704
7.702
7.698
7.696
7.659
7.657
7.650
7.649
7.144
7.138
7.136
7.130
7.021
3.043
3.039
3.028
3.017
1.690
1.687
1.678
1.671
1.668
1.665
1.653
1.364
1.358
1.352
0.907
0.901
0.895
0.892
0.890
0.886
0.883
X : parts per Million : Proton


O
Cl
S

(*E*)-3l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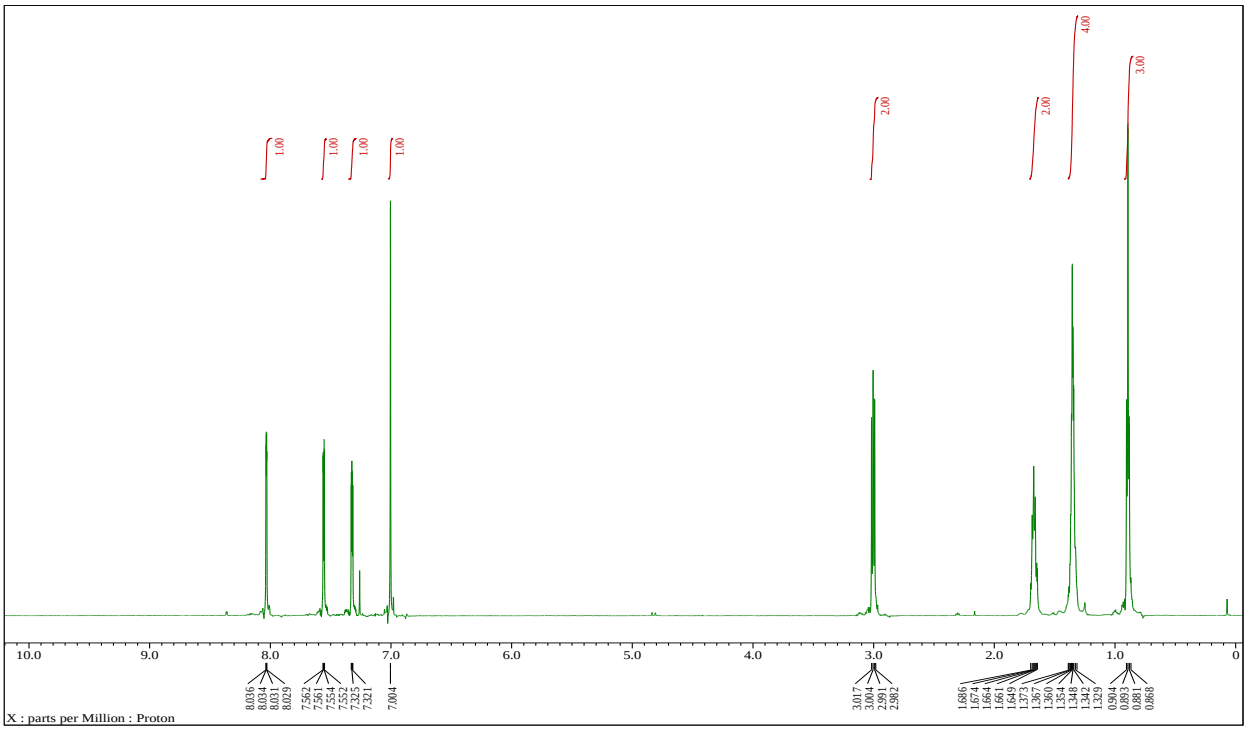
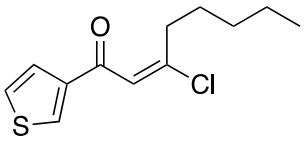
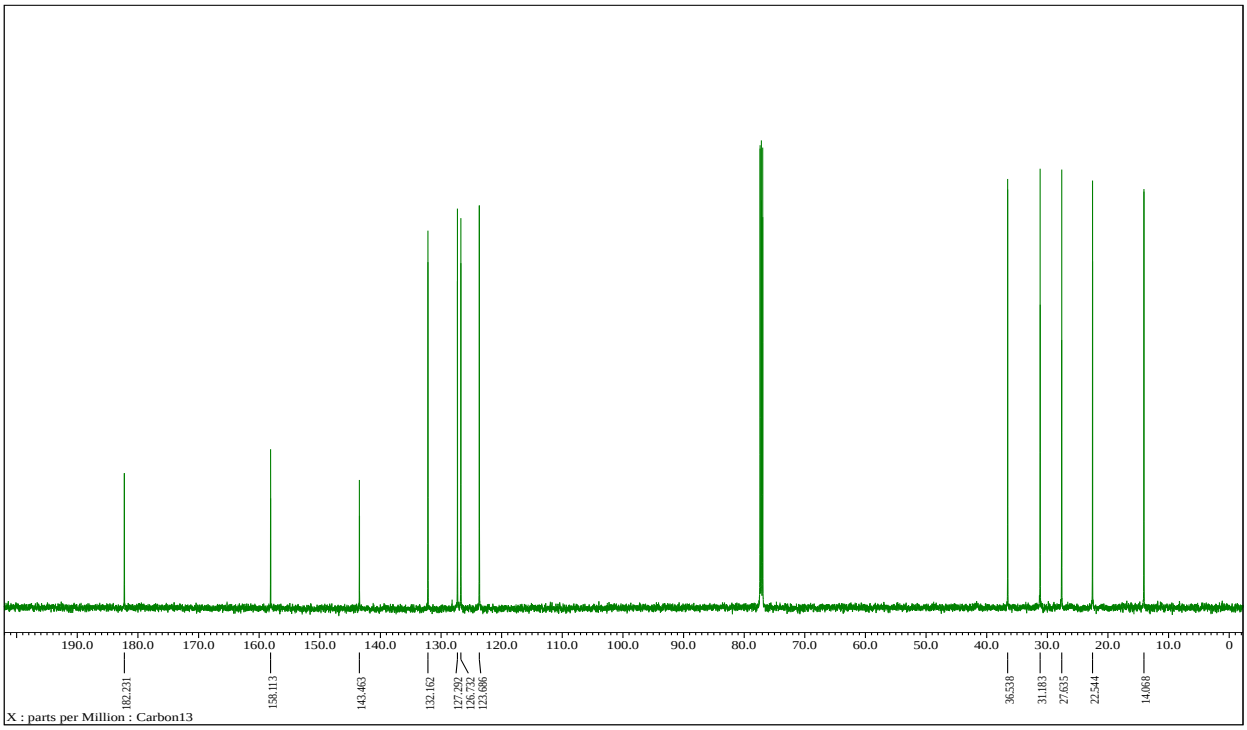
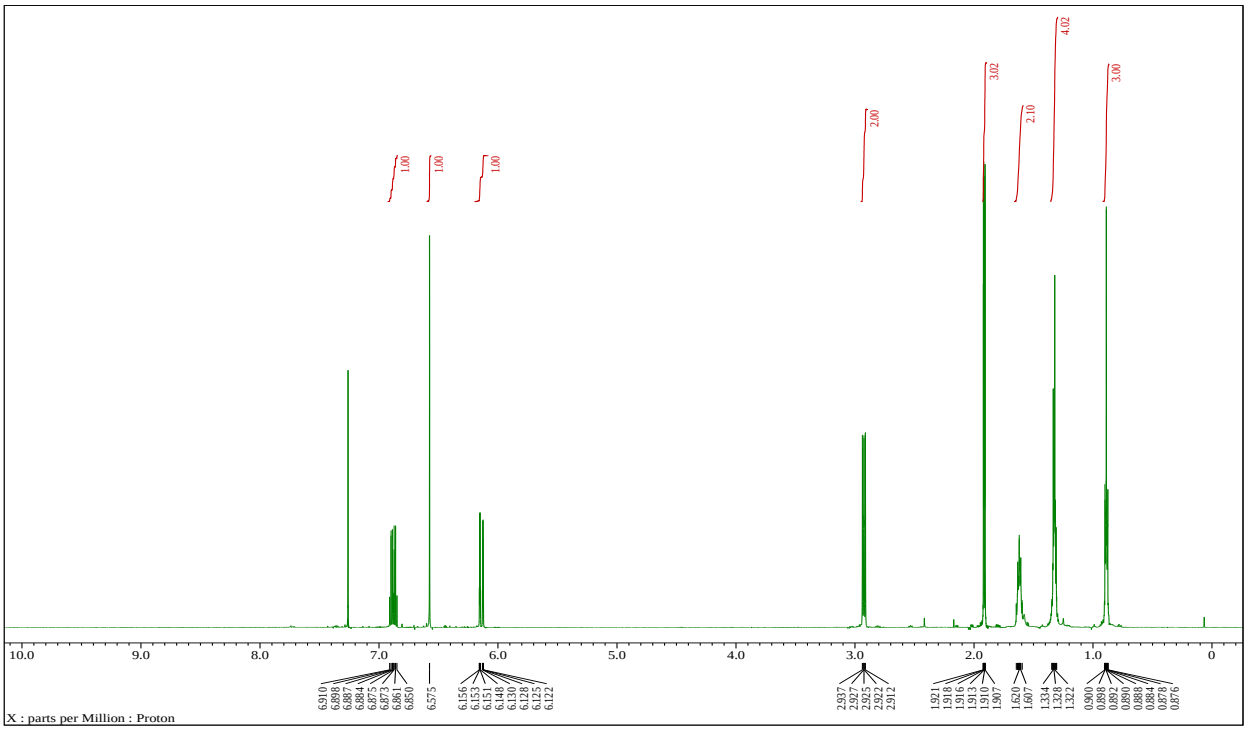
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
180.658
158.626
146.033
134.337
131.779
128.417
122.628
36.642
31.199
27.679
22.559
14.083
X : parts per Million : Carbon13

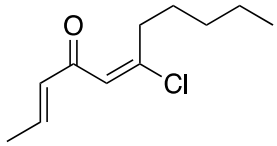

1.00
1.00
1.00
1.00
2.00
2.00
4.00
3.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.036
8.034
8.031
8.029
7.562
7.561
7.554
7.552
7.325
7.321
7.004
3.017
3.004
2.991
2.982
1.686
1.674
1.664
1.651
1.649
1.373
1.367
1.360
1.354
1.348
1.342
1.329
0.904
0.893
0.881
0.868
X : parts per Million : Proton


O
Cl
S

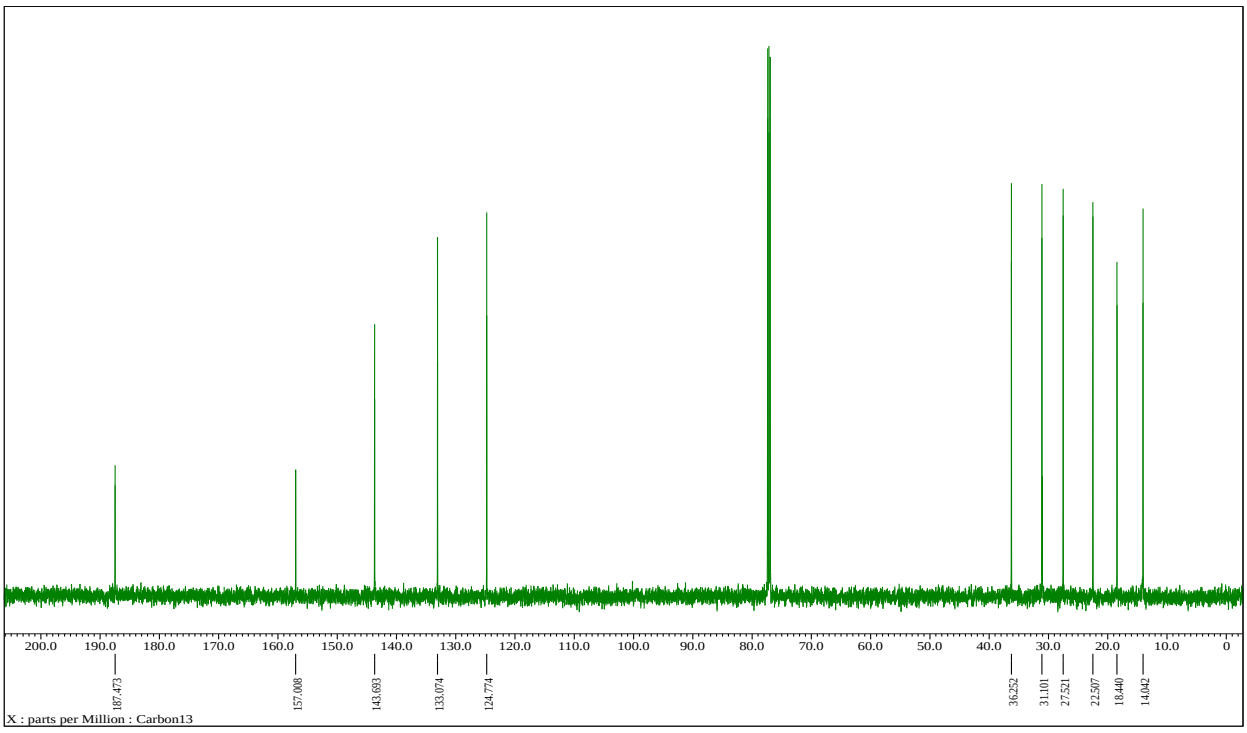
(*E*)-3m

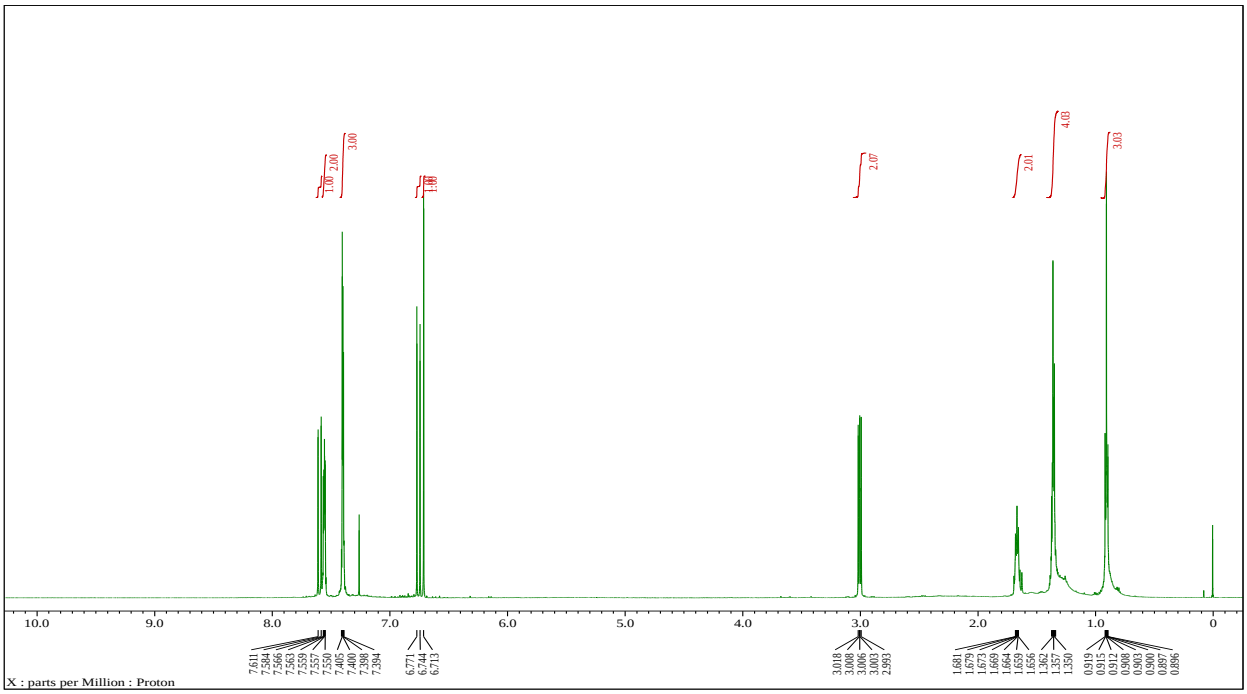

190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
182.231
158.113
143.463
132.162
127.292
126.732
123.086
36.538
31.183
27.635
22.544
14.068
X : parts per Million : Carbon13

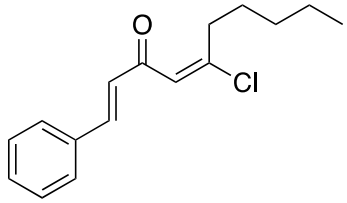

1.00
1.00
1.00
2.00
3.02
2.10
4.02
3.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
6.910
6.898
6.887
6.884
6.875
6.873
6.861
6.850
6.575
6.156
6.153
6.151
6.148
6.130
6.128
6.125
6.122
2.937
2.927
2.925
2.922
2.912
1.921
1.918
1.916
1.913
1.910
1.907
1.620
1.607
1.334
1.328
1.322
0.900
0.898
0.892
0.889
0.888
0.884
0.878
0.876
X : parts per Million : Proton


O
Cl

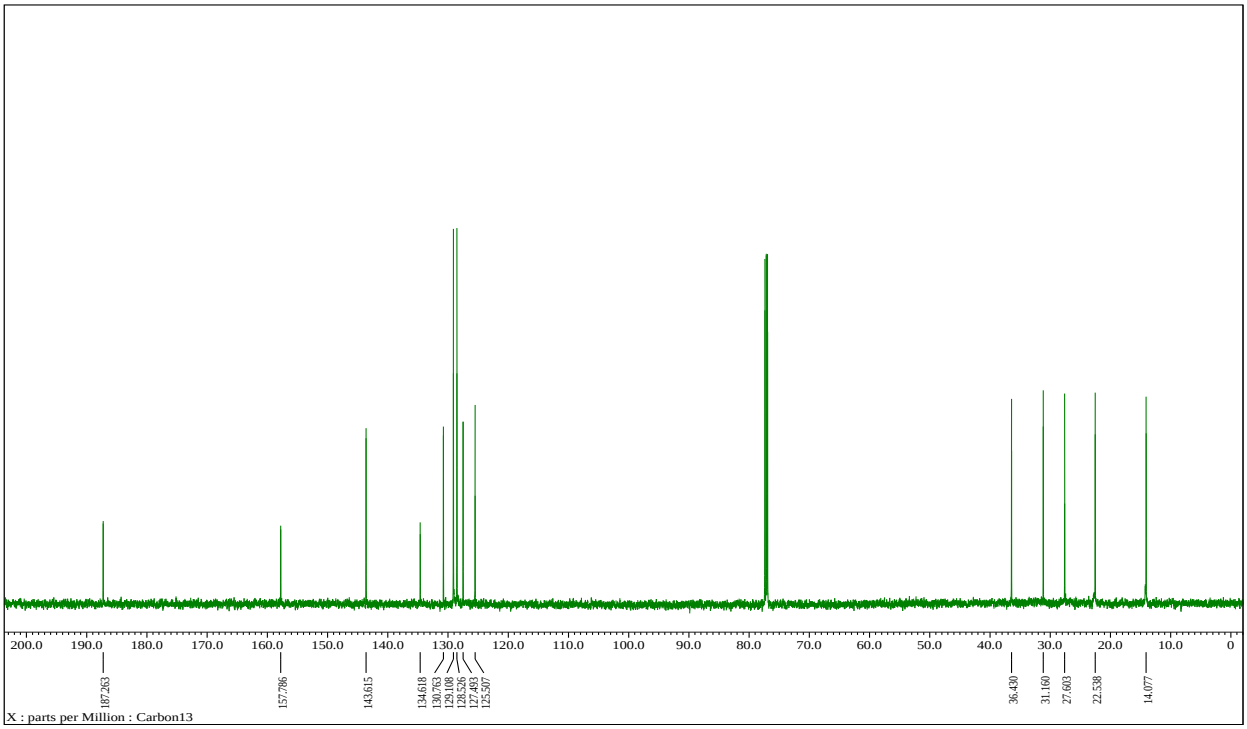
(*E*)-3n

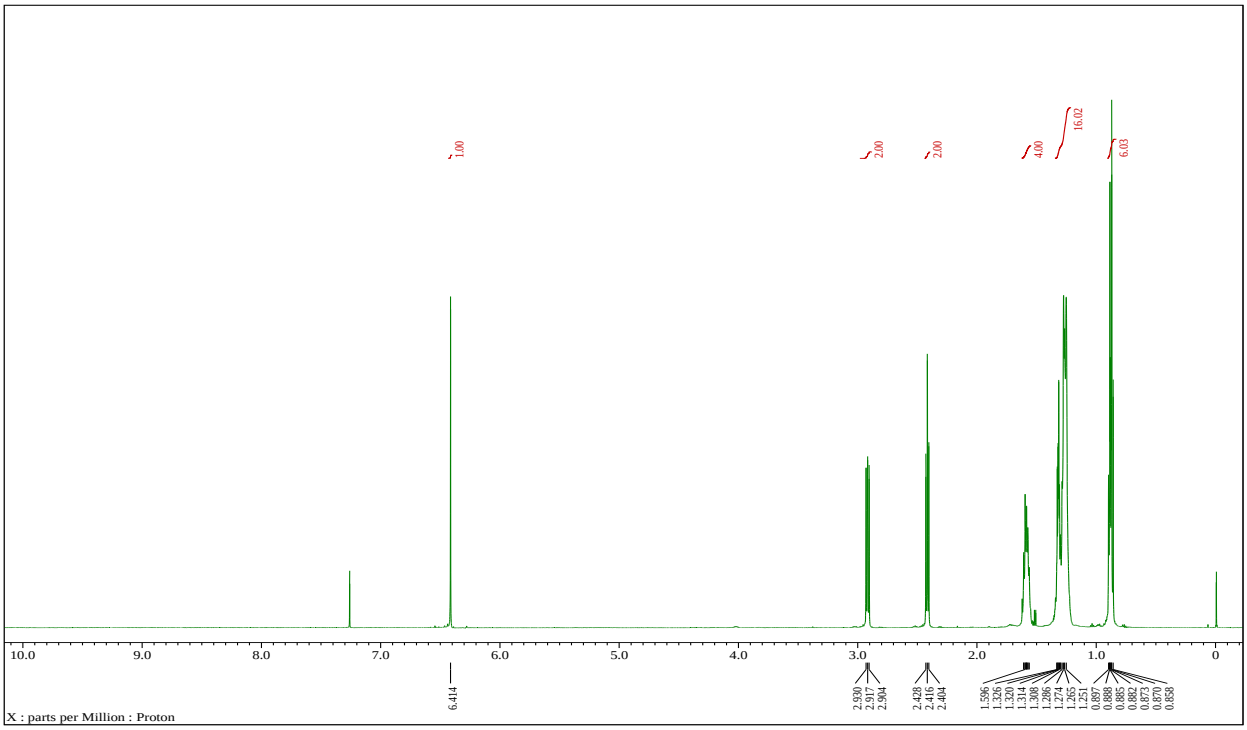

200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
187.473
157.008
143.693
133.074
124.774
36.252
31.101
27.521
22.507
18.440
14.042
X : parts per Million : Carbon13

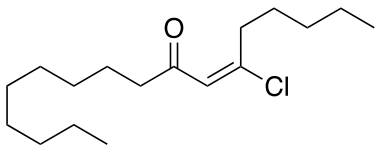




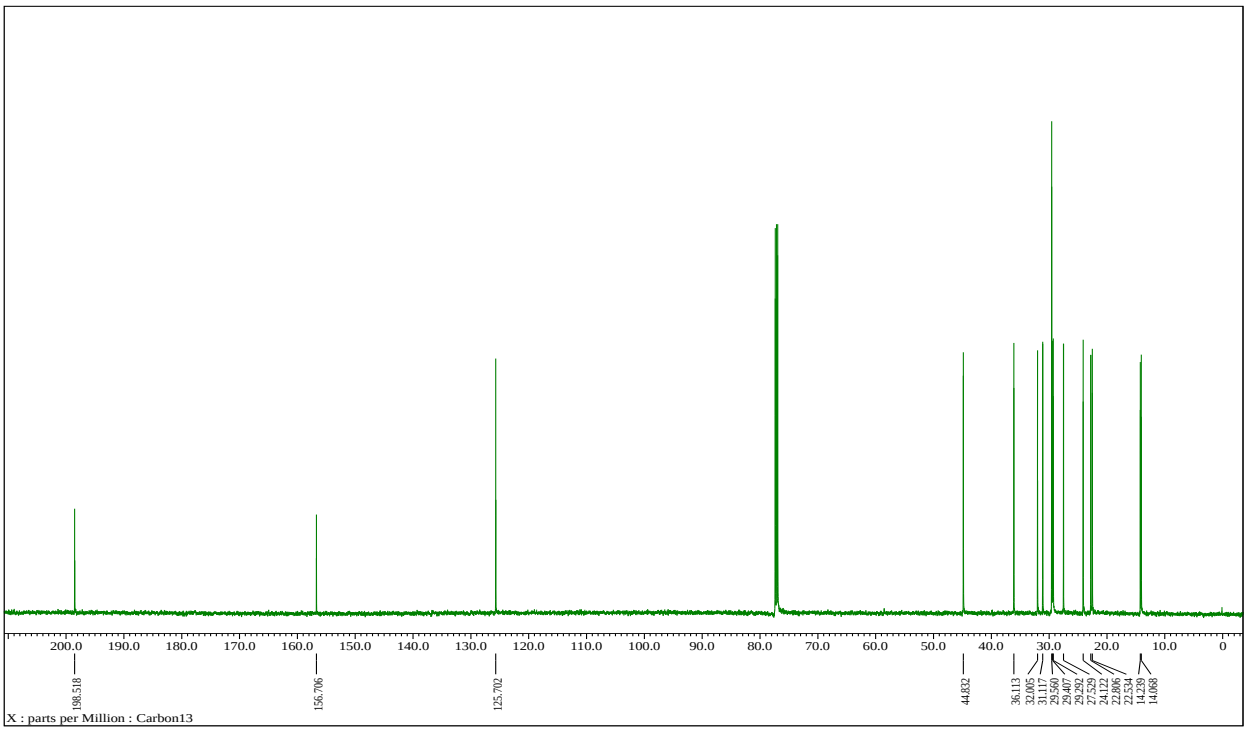
(*E*)-3o

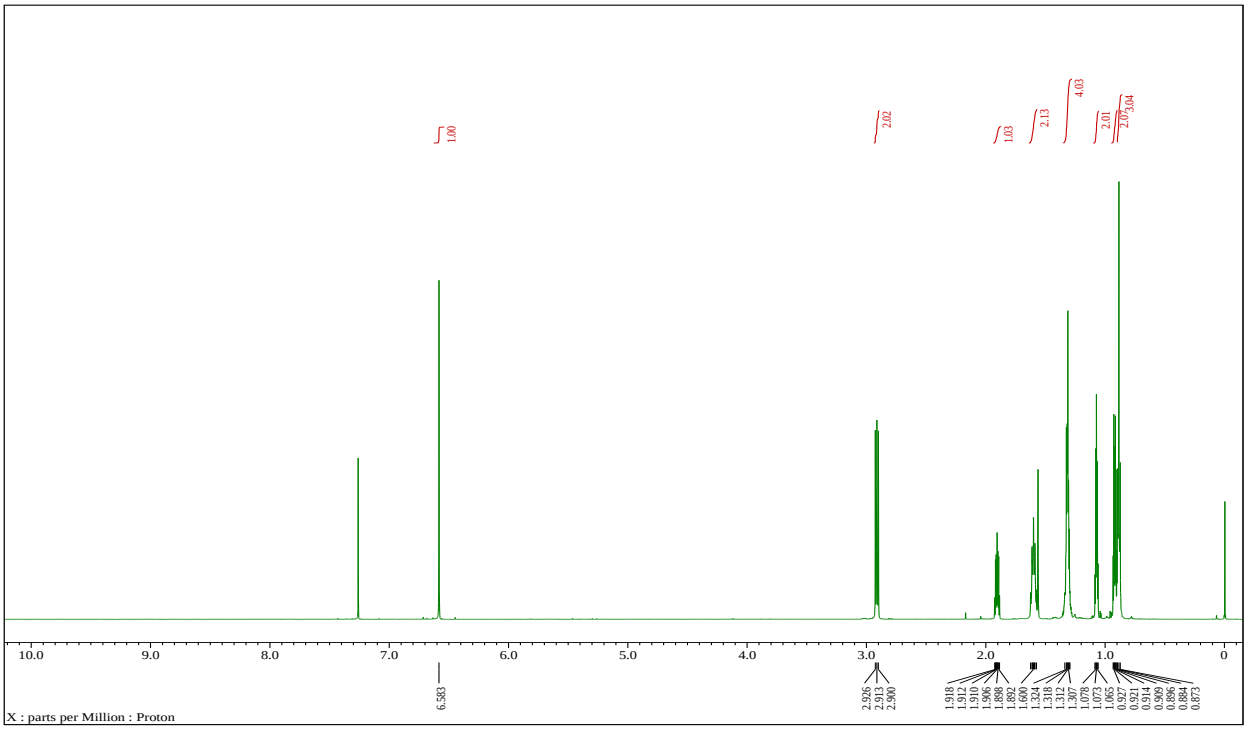



1.00
2.00
2.00
4.00
16.02
6.03
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
6.414
2.930
2.917
2.904
2.428
2.416
2.404
1.596
1.326
1.320
1.314
1.308
1.286
1.274
1.265
1.173
0.897
0.888
0.885
0.882
0.873
0.870
0.858
X : parts per Million : Proton


O
Cl

(*E*)-3p


200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
198.518
156.706
125.702
44.832
36.113
32.005
31.150
29.560
29.407
29.292
27.529
24.122
22.806
22.534
14.238
14.068
X : parts per Million : Carbon13


1.00
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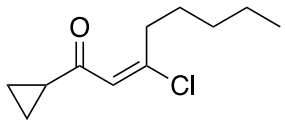
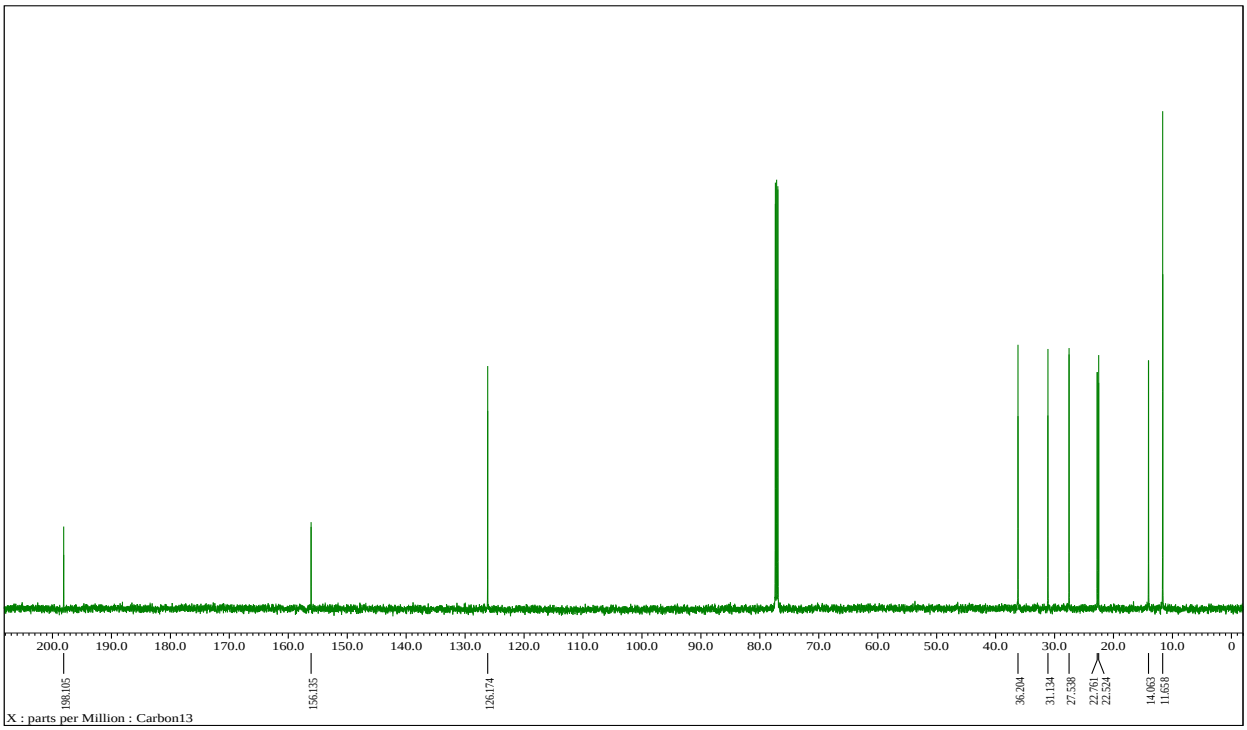
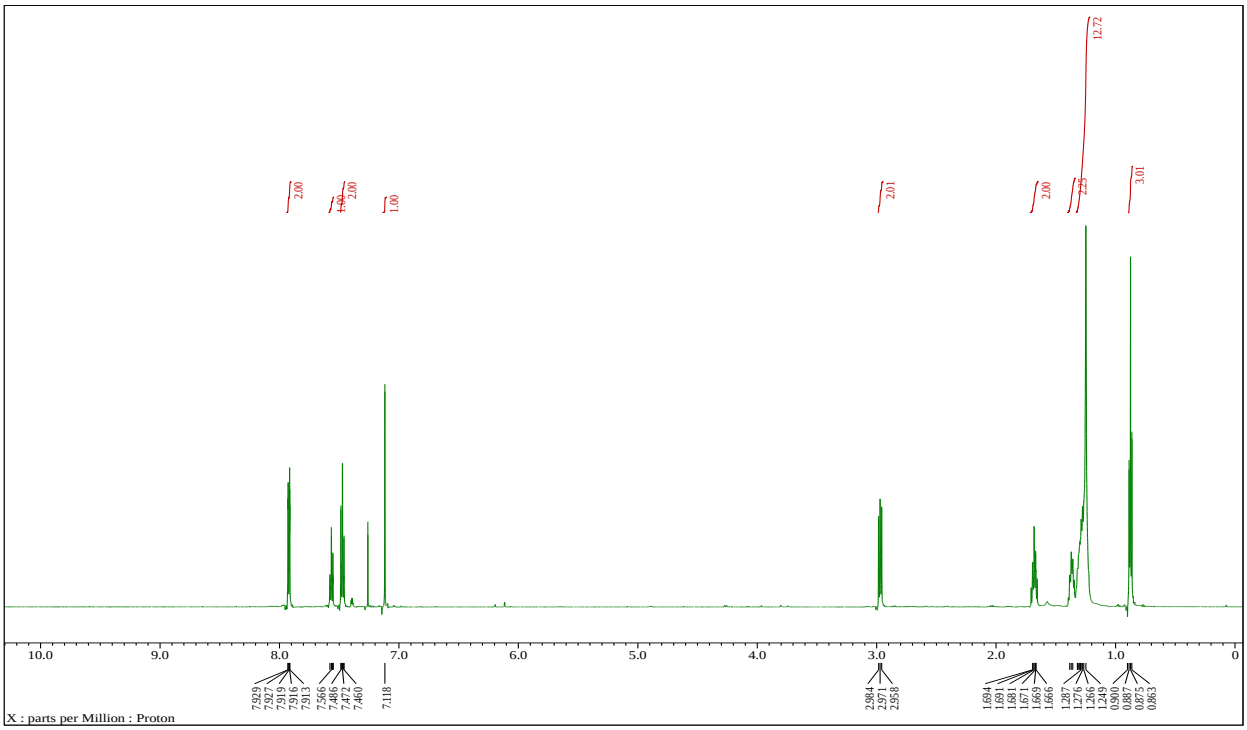
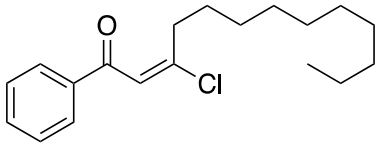
1.03
2.13
4.03
2.01
2.05
3.04
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
6.583
2.926
2.913
2.900
1.918
1.912
1.910
1.906
1.898
1.892
1.600
1.324
1.318
1.312
1.307
1.078
1.073
1.065
0.927
0.921
0.914
0.909
0.896
0.884
0.873
X : parts per Million : Proton


O
Cl

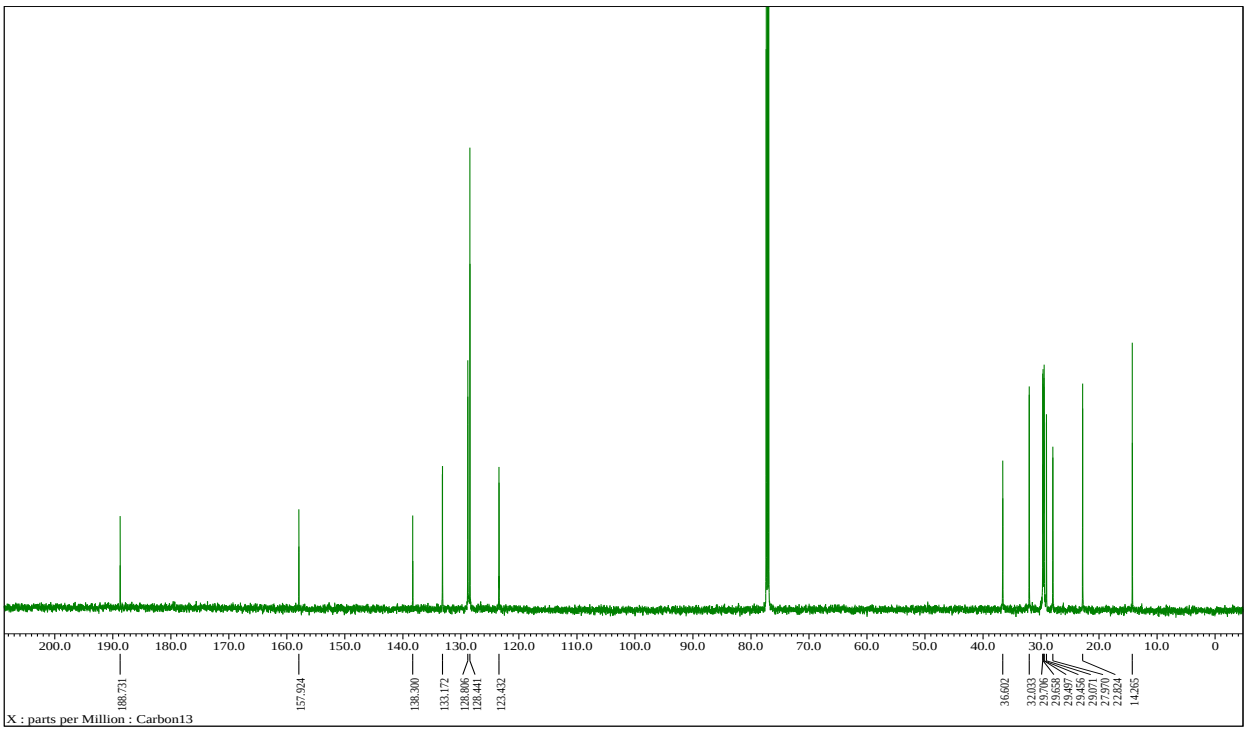
(*E*)-3q

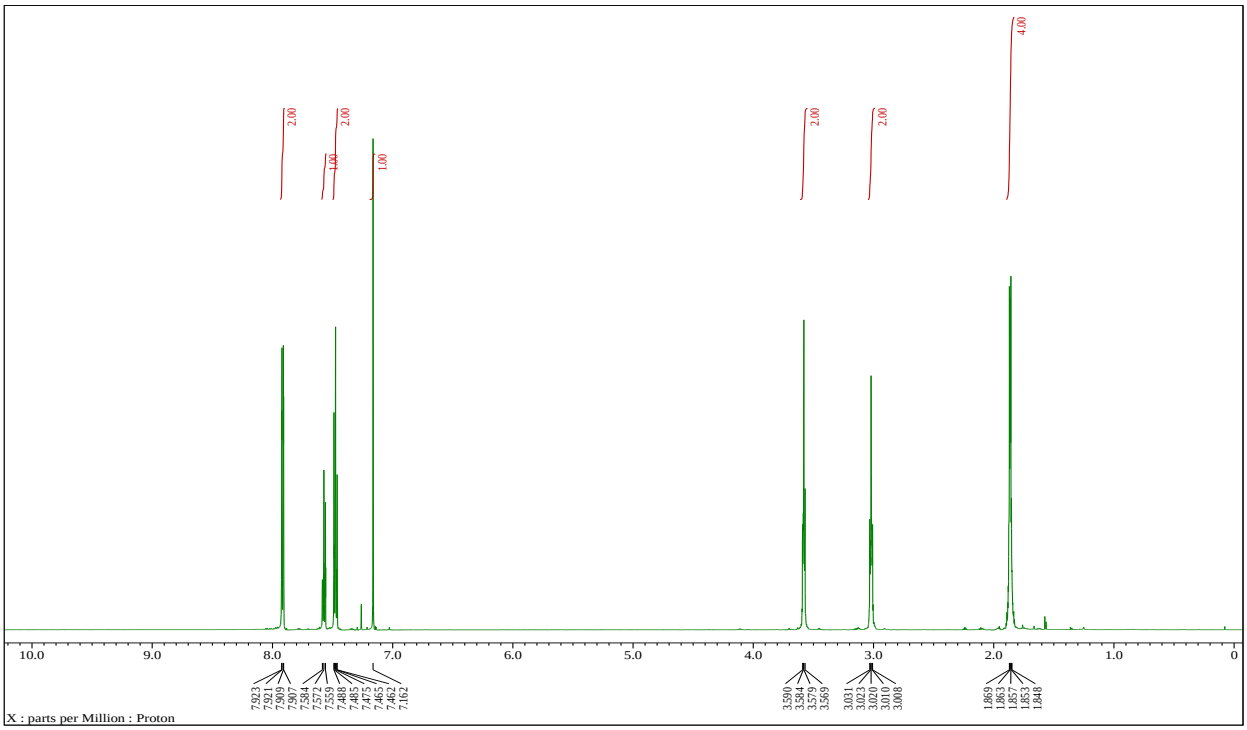

200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
198.105
156.135
126.174
36.204
31.134
27.538
22.761
22.524
14.063
11.658
X : parts per Million : Carbon13

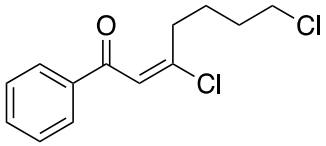

12.72
2.00
2.00
2.00
1.00
2.01
2.00
2.25
3.01
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.929
7.927
7.919
7.916
7.913
7.566
7.486
7.472
7.460
7.118
2.984
2.971
2.958
1.694
1.691
1.681
1.671
1.669
1.666
1.287
1.276
1.266
1.249
0.900
0.887
0.875
0.863
X : parts per Million : Proton


O
Cl

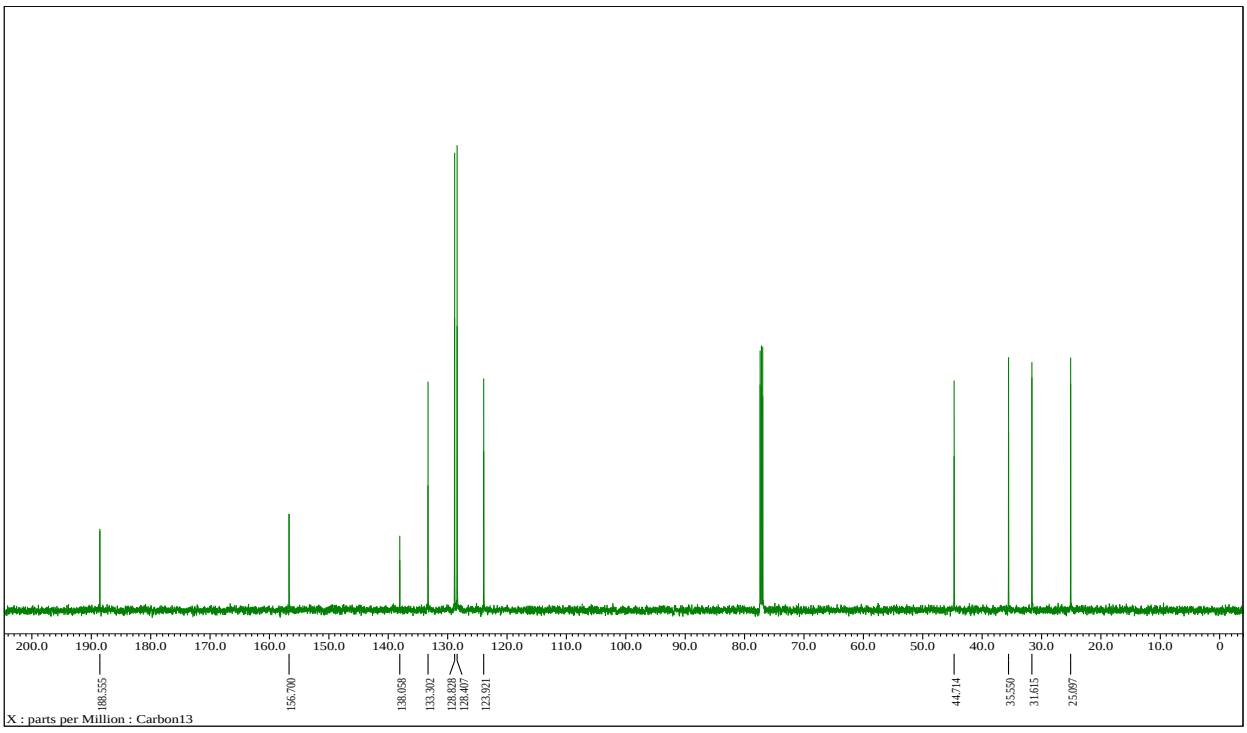
(*E*)-3r

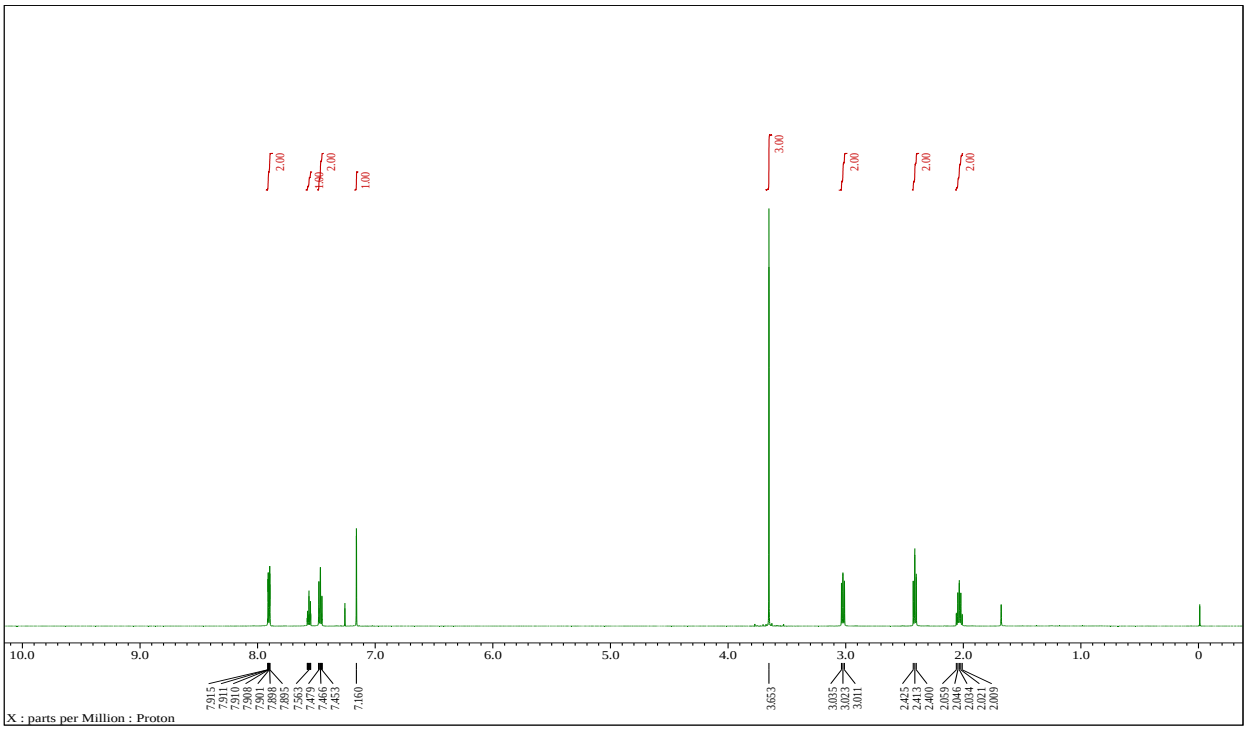

200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
188.731
157.924
138.300
133.172
128.806
128.441
123.432
36.602
32.033
29.706
29.657
29.495
29.456
29.071
27.970
22.824
14.265
X : parts per Million : Carbon13


2.00
2.00
1.00
1.00
2.00
2.00
4.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.923
7.921
7.909
7.907
7.584
7.572
7.559
7.548
7.485
7.475
7.465
7.462
7.162
3.590
3.584
3.579
3.569
3.031
3.023
3.010
3.010
3.008
1.869
1.863
1.857
1.853
1.848
X : parts per Million : Proton


O
Cl
Cl

(*E*)-3s


200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
188.555
156.700
138.058
133.302
128.828
128.407
123.921
44.714
35.550
31.615
25.097
X : parts per Million : Carbon13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1.00
3.00
2.00
2.00
2.00
10.0
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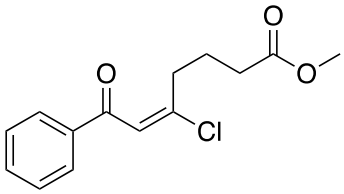
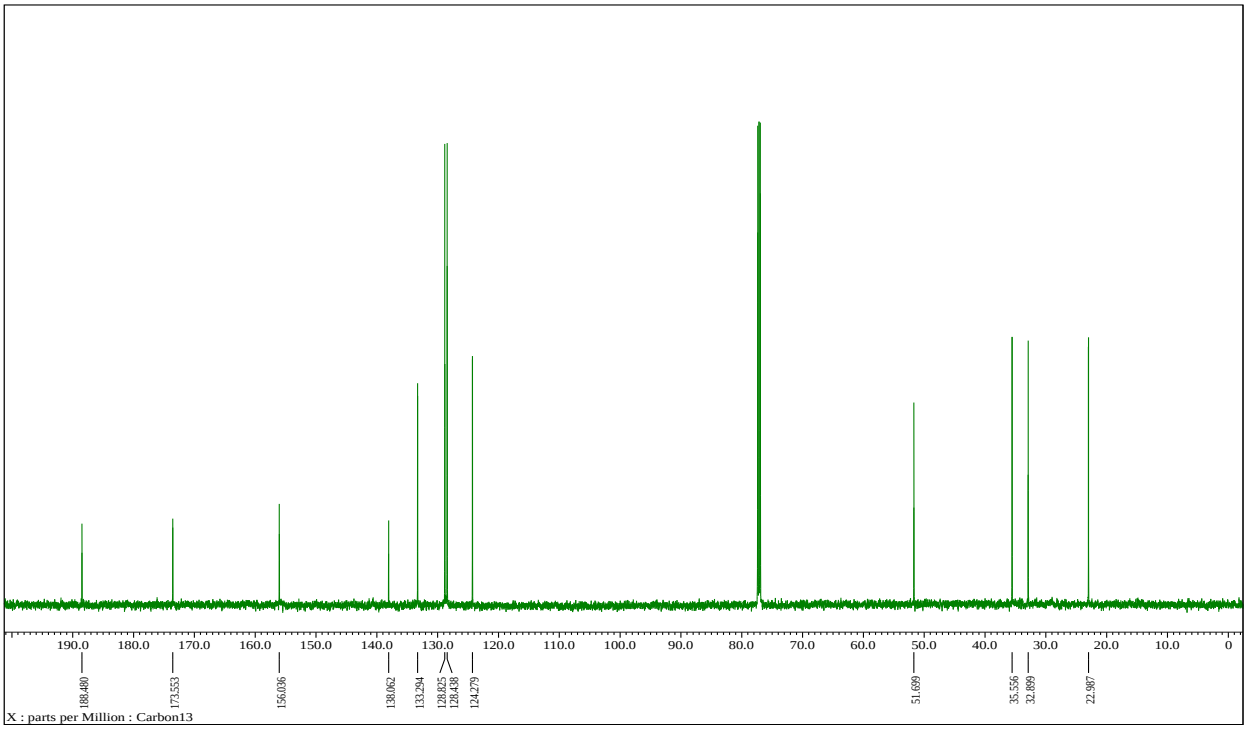
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.915
7.911
7.910
7.908
7.901
7.898
7.895
7.563
7.479
7.466
7.453
7.160
3.653
3.035
3.023
3.011
2.425
2.413
2.400
2.035
2.034
2.021
2.009
X : parts per Million : Proton


O
O
O
Cl

(*E*)-3t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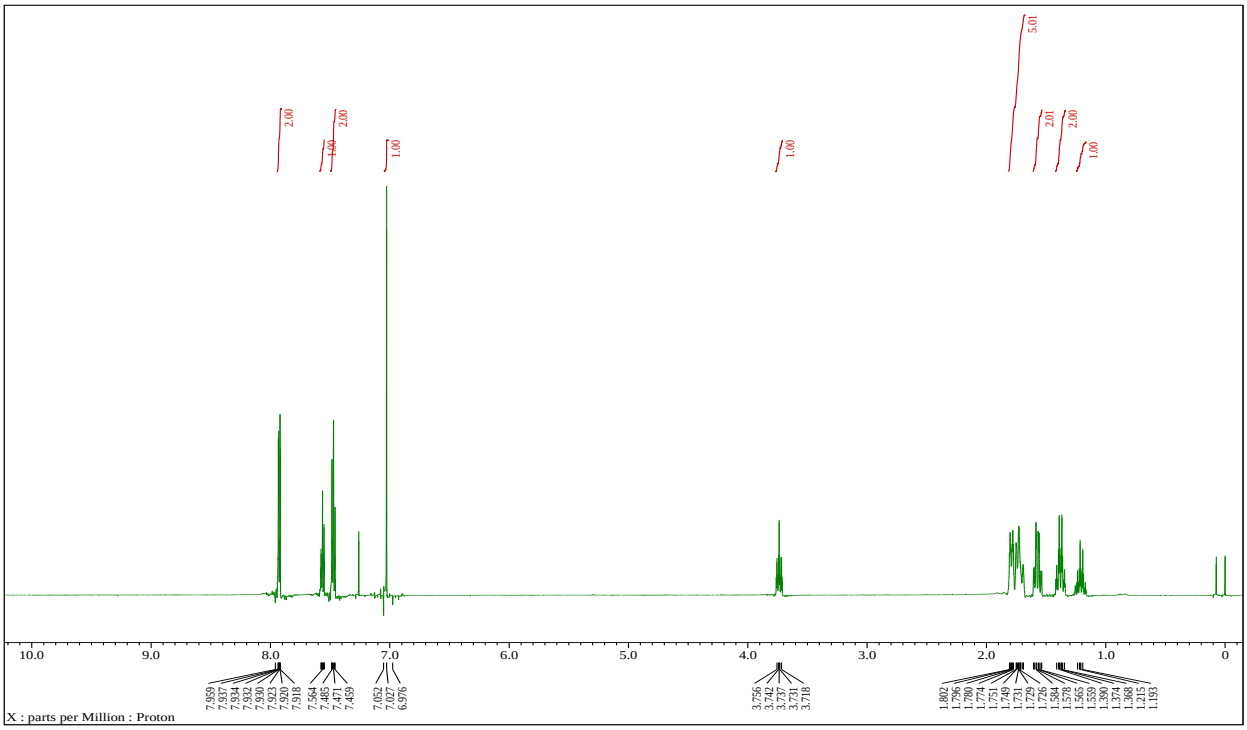
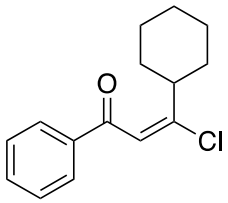
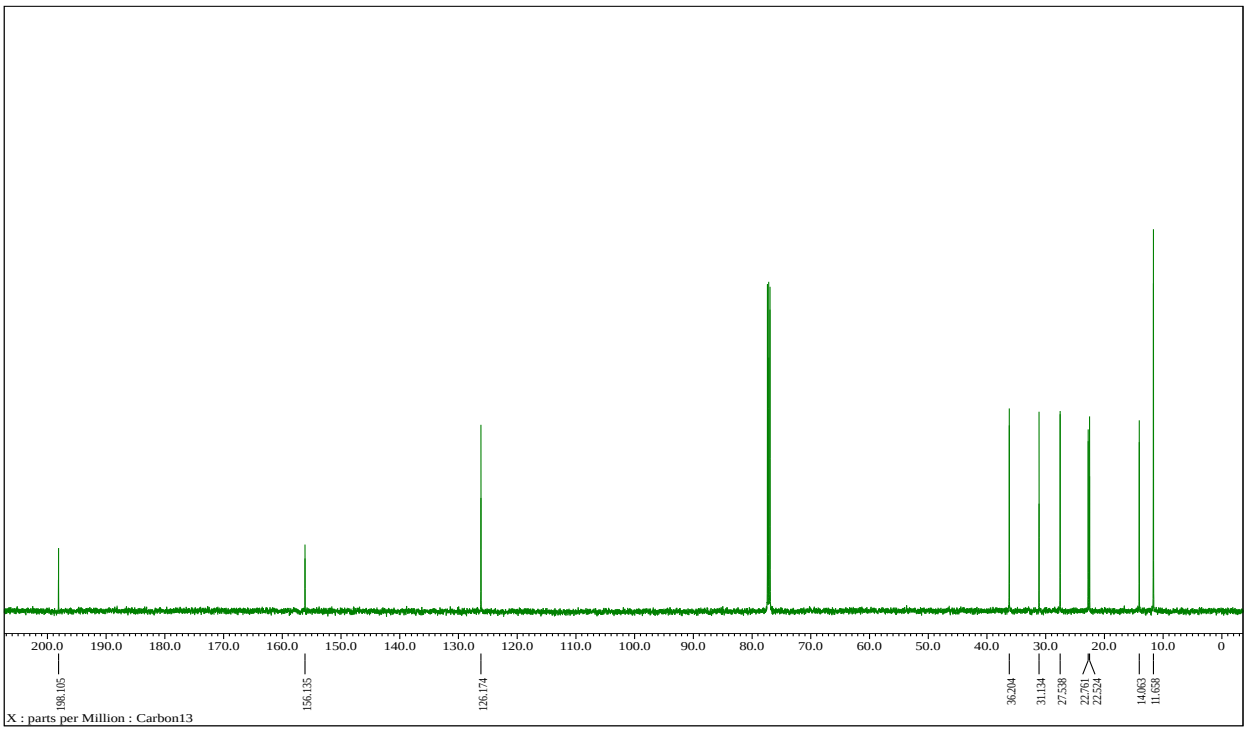
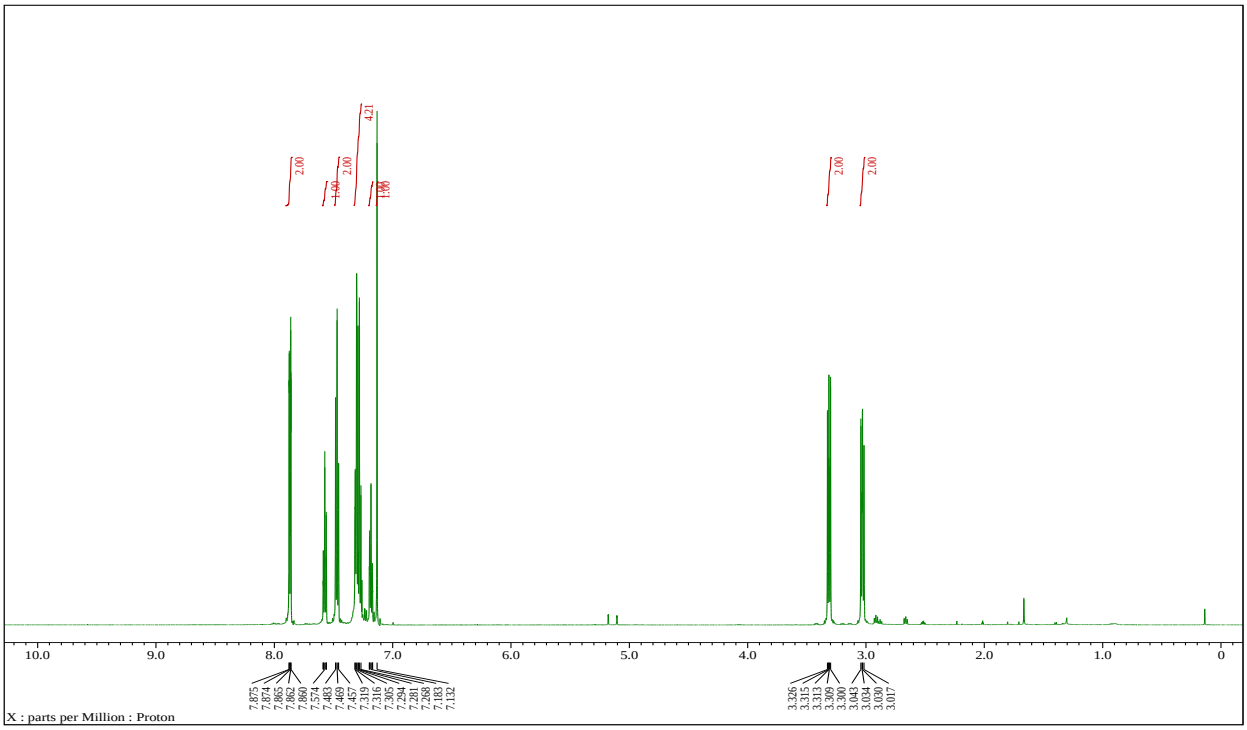
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
188.480
173.553
156.036
138.062
133.294
128.825
128.438
124.279
51.699
35.556
32.899
22.987
X : parts per Million : Carbon13





(*E*)-3u

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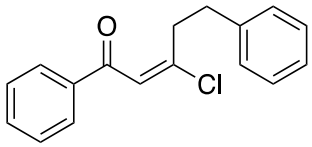
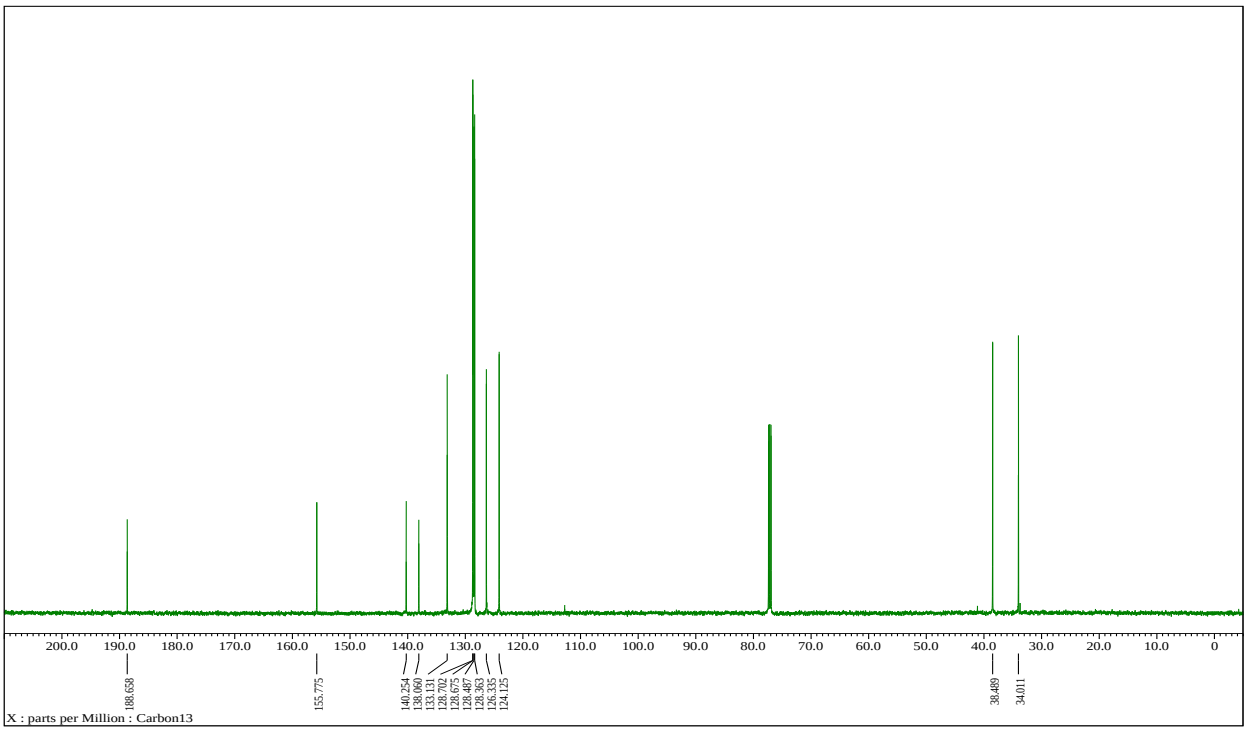
2.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.875
7.874
7.865
7.862
7.860
7.574
7.469
7.457
7.319
7.316
7.305
7.294
7.281
7.188
7.132
3.326
3.315
3.313
3.309
3.300
3.043
3.034
3.030
3.017
X : parts per Million : Proton


O
Cl

(*E*)-3v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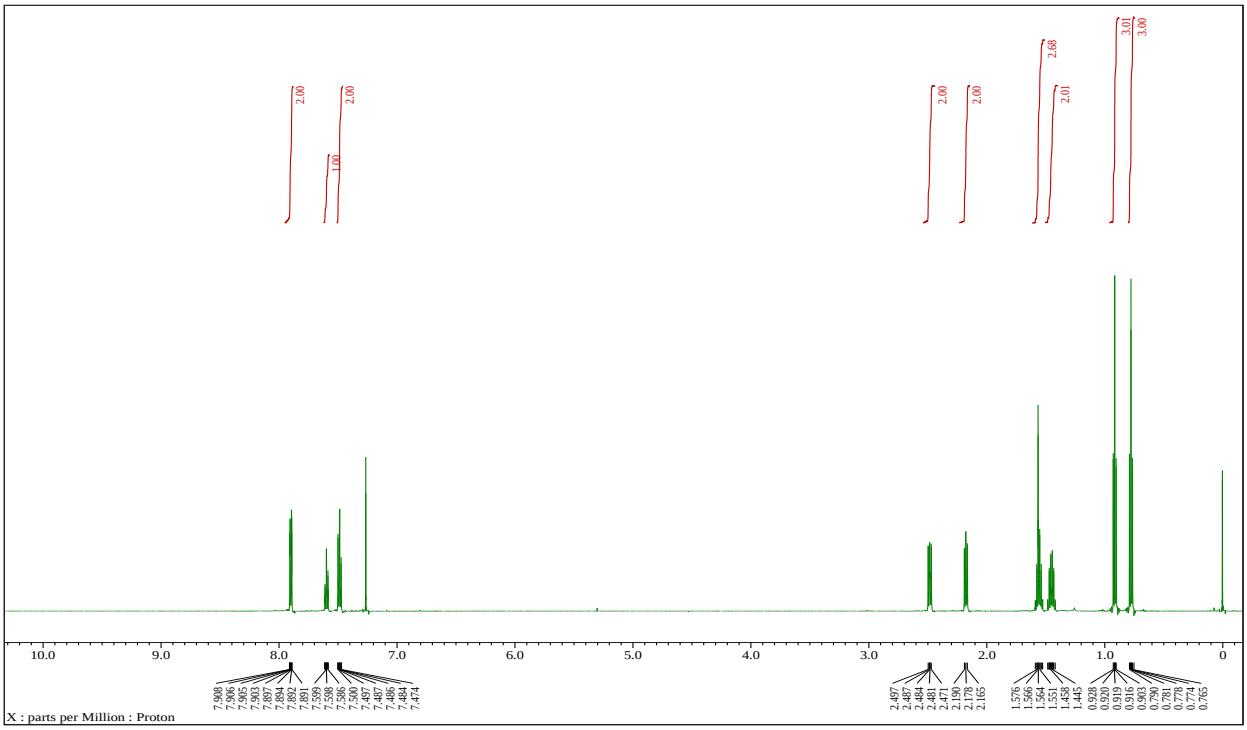
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
188.658
155.775
140.254
138.060
133.131
128.702
128.675
128.485
128.363
126.335
124.125
38.489
34.011
X : parts per Million : Carbon13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3.01
3.00
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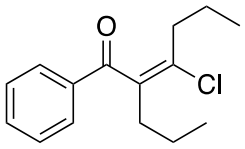
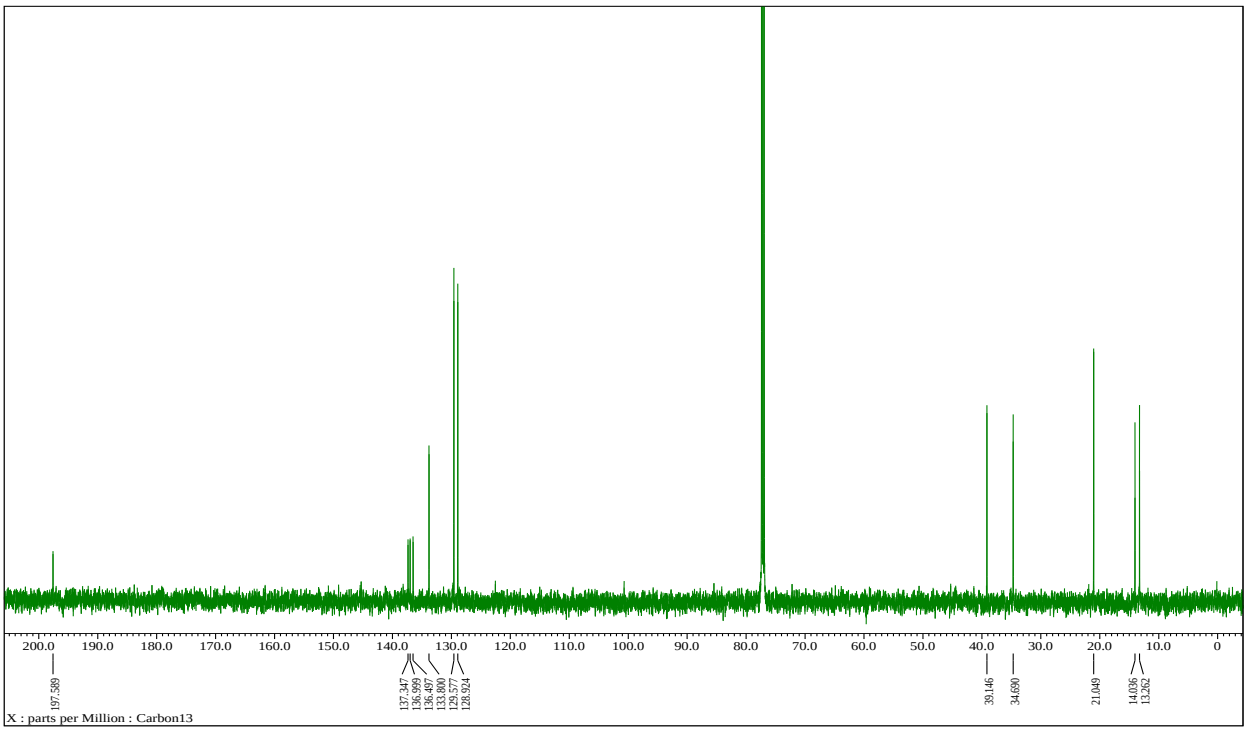
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : Proton


O
Cl

(*E*)-3w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140.0
130.0
120.0
110.0
100.0
90.0
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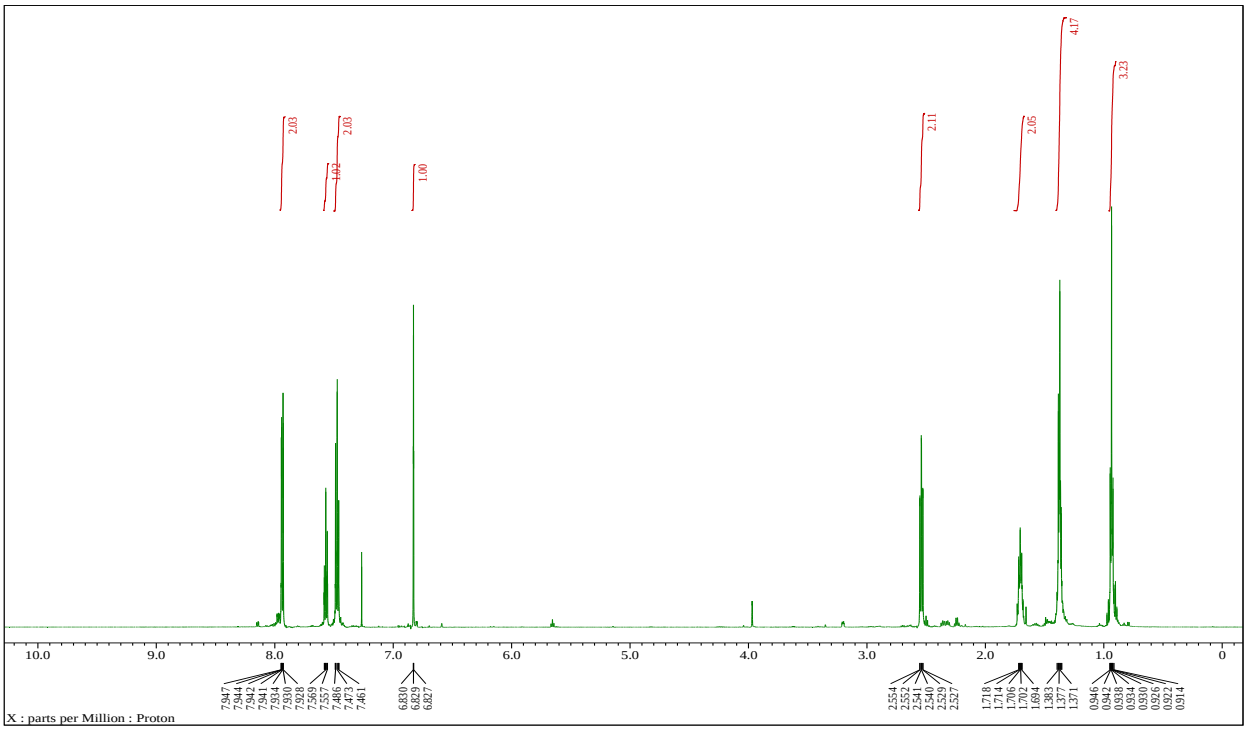
60.0
50.0
40.0
30.0
20.0
10.0
0
197.589
137.347
136.999
136.497
133.800
129.577
128.924
39.146
34.690
21.049
14.036
13.262
X : parts per Million : Carbon13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2.03
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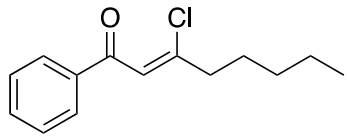
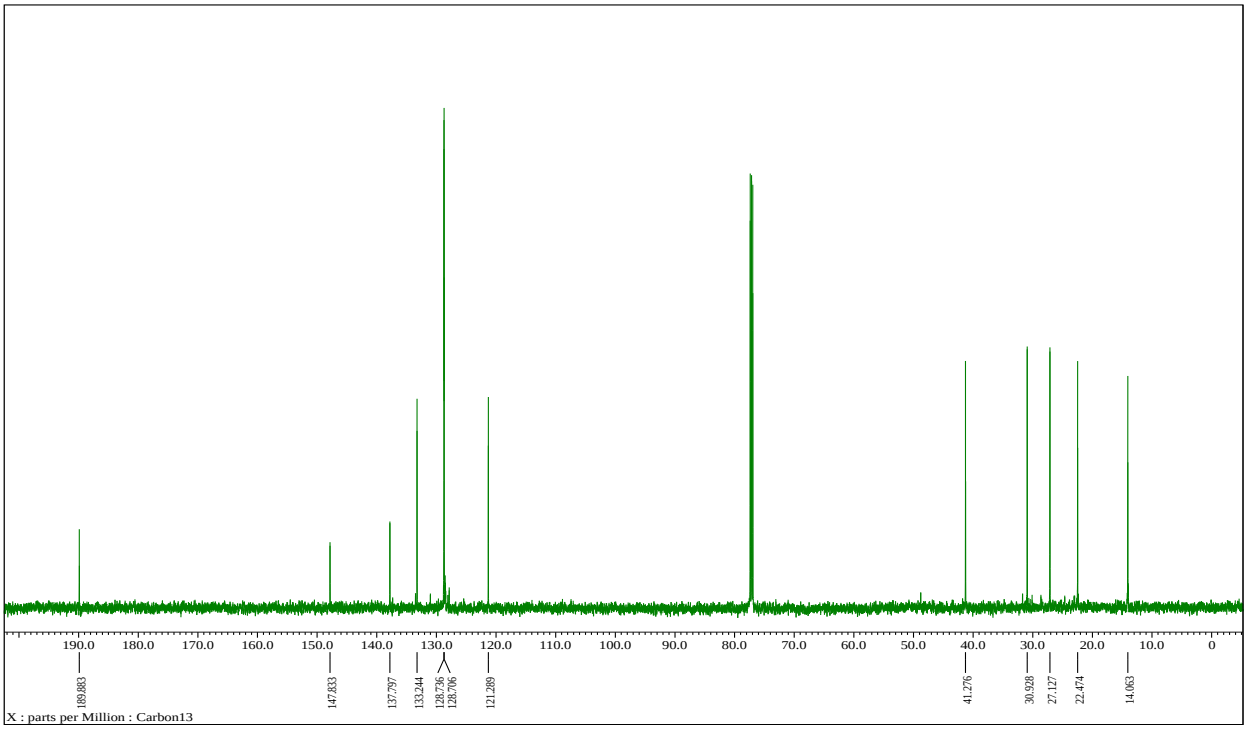
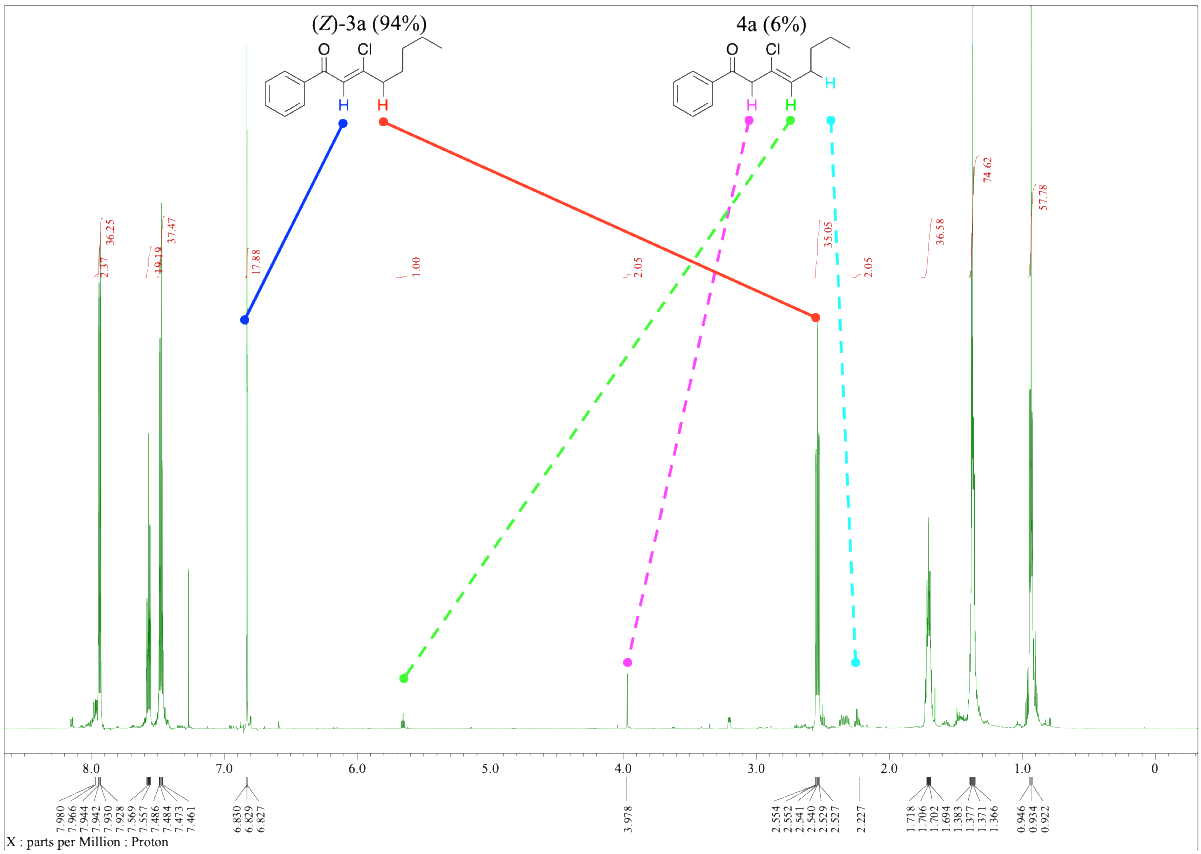
2.11
2.05
4.17
3.23
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.947
7.944
7.942
7.941
7.934
7.930
7.569
7.557
7.486
7.473
7.461
6.830
6.829
6.827
2.554
2.552
2.541
2.540
2.529
2.527
1.718
1.714
1.706
1.702
1.694
1.383
1.377
1.371
0.946
0.942
0.938
0.934
0.930
0.926
0.917
0.914
X : parts per Million : Proton

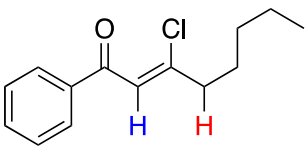

O
Cl

(*Z*)-3a

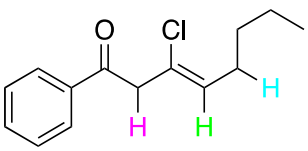

190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
189.883
147.833
137.797
133.244
128.736
128.706
121.289
41.276
30.928
27.127
22.474
14.063
X : parts per Million : Carbon13

(*Z*)-3a/4a mixture identification

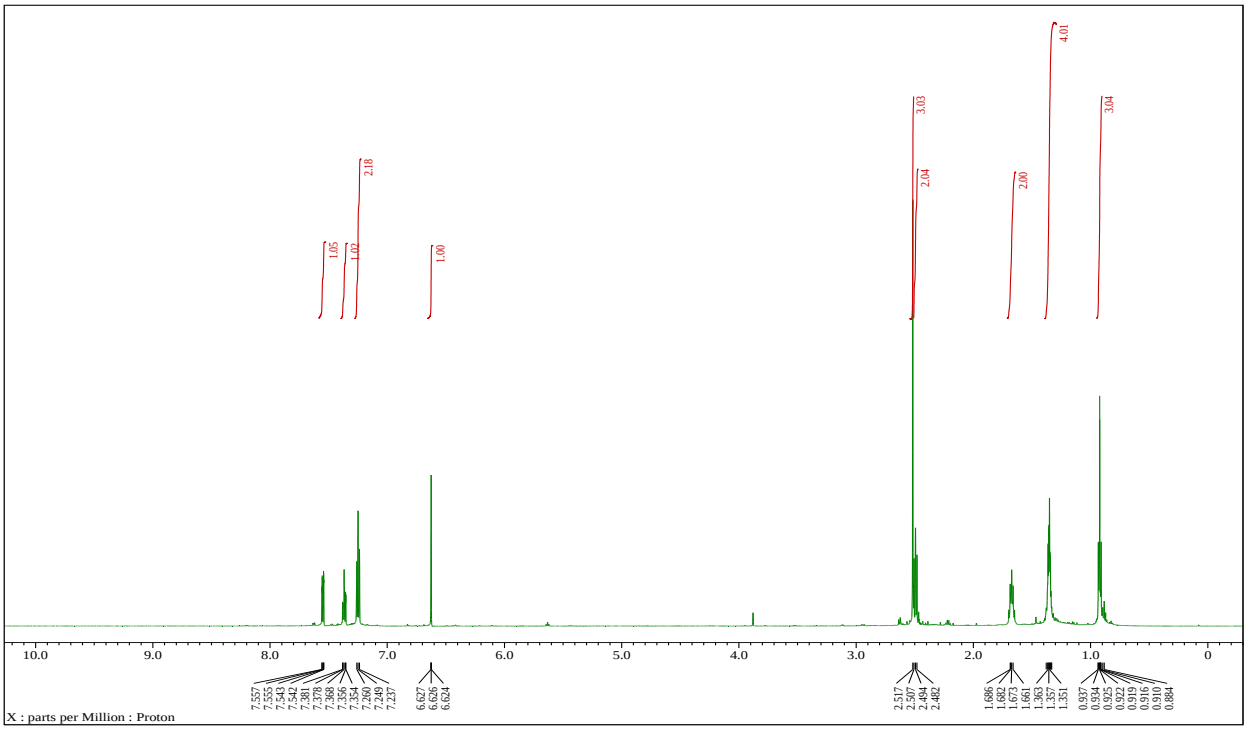


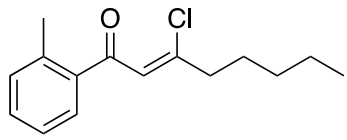


(*Z*)-3a: ^{1}H-NMR (600 MHz, $CDCl_3$): δ 7.94 (dd, *J* = 8.3, 1.3 Hz, 2H), 7.55-7.58 (m, 1H), 7.46-7.49 (m, 2H), **6.83** (t, *J* = 0.9 Hz, 1H), **2.53-2.55** (td, *J* = 7.5, 0.9 Hz, 2H), 1.69-1.72 (m, 2H), 1.35-1.39 (m, 4H), 0.93 (t, *J* = 7.1 Hz, 3H).

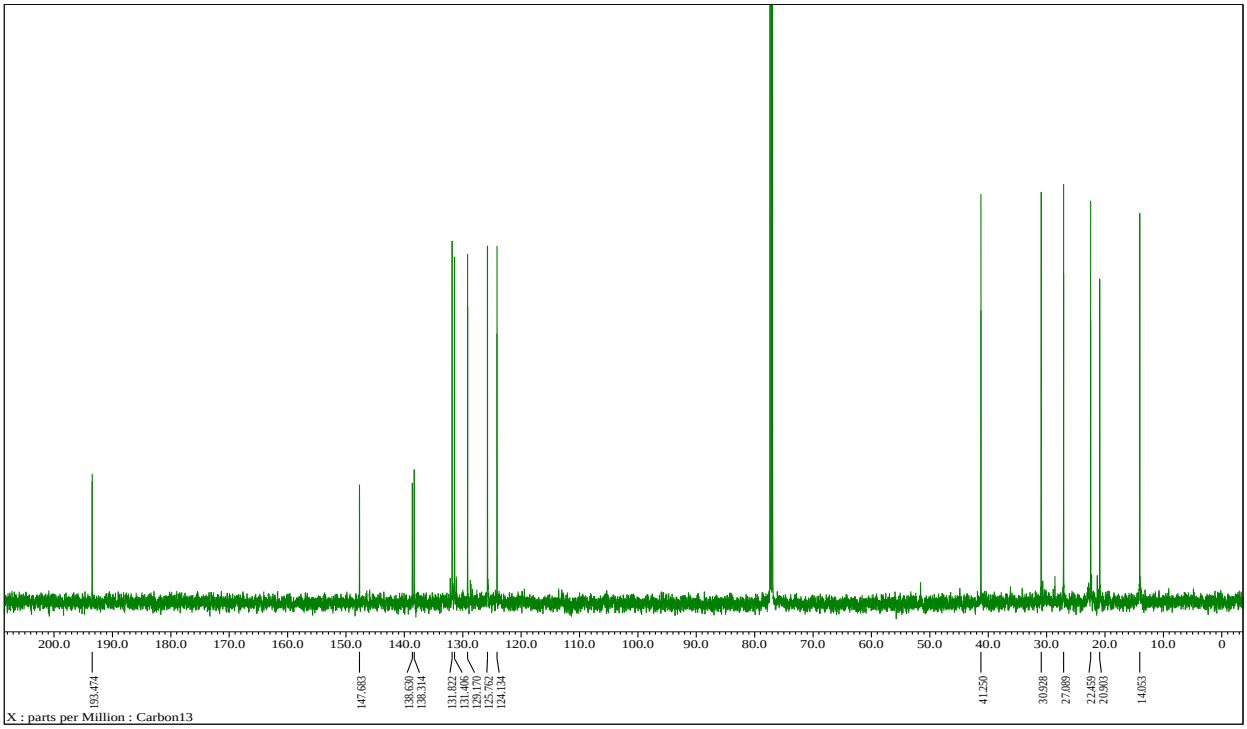


(4a): ^{1}H-NMR (600 MHz, $CDCl_3$): δ 7.96 (m, 2H), 7.55-7.58 (m, 1H), 7.46-7.49 (m, 2H), **5.66** (t, *J* = 0.9 Hz, 1H), **3.97** (d, *J* = 0.9 Hz, 2H), **2.22-2.26** (m, 2H), 1.35-1.39 (m, 4H), 0.93 (t, *J* = 7.1 Hz, 3H).


4.01
3.03
3.04
2.18
2.04
2.00
1.05
1.02
1.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.557
7.555
7.543
7.542
7.381
7.378
7.368
7.356
7.354
7.260
7.249
7.237
6.627
6.626
6.624
2.517
2.507
2.494
2.482
1.686
1.682
1.672
1.661
1.363
1.357
1.351
0.937
0.934
0.925
0.922
0.919
0.916
0.910
0.884
X : parts per Million : Proton


O
Cl

(*Z*)-3b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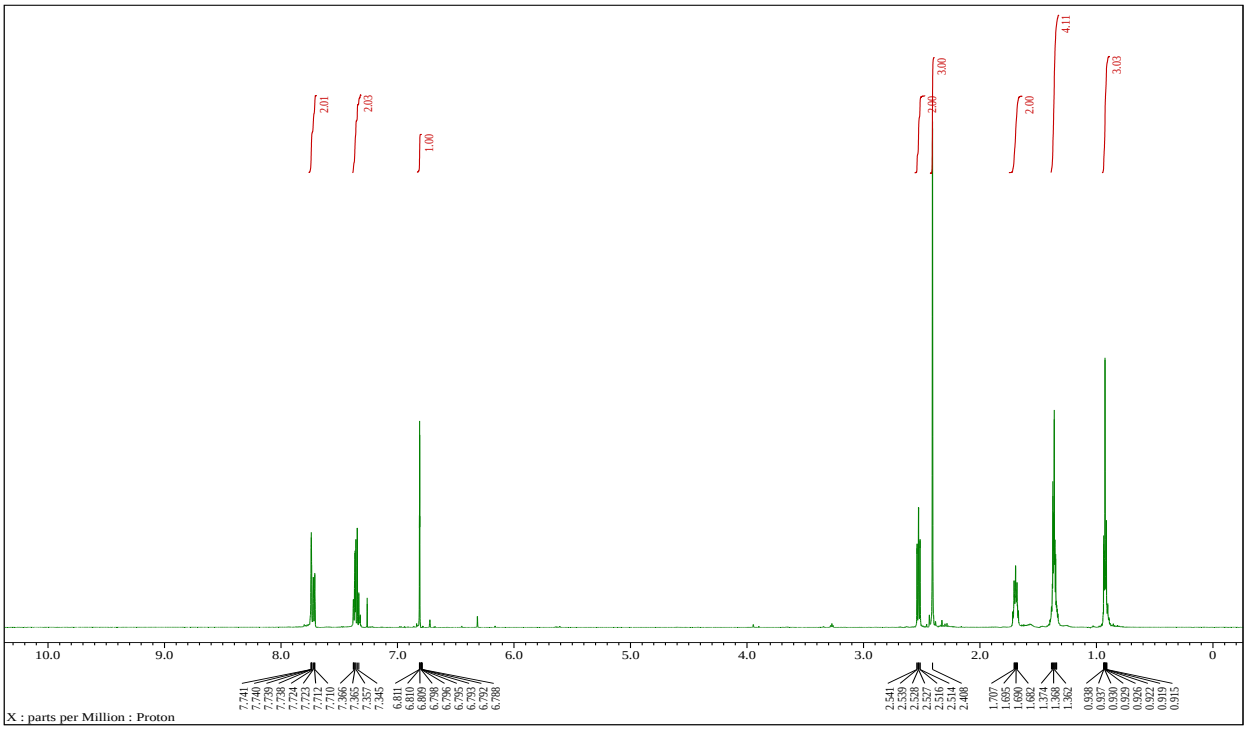
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
193.474
147.683
138.630
138.314
131.822
131.406
129.170
125.762
124.134
41.250
30.928
27.089
22.459
20.903
14.053
X : parts per Million : Carbon13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3.00
2.00
4.11
3.03
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.741
7.740
7.739
7.738
7.734
7.723
7.712
7.710
7.366
7.365
7.357
7.345
6.811
6.810
6.809
6.798
6.796
6.795
6.793
6.792
6.788
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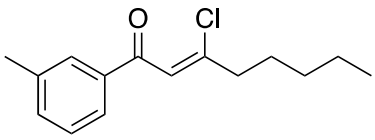
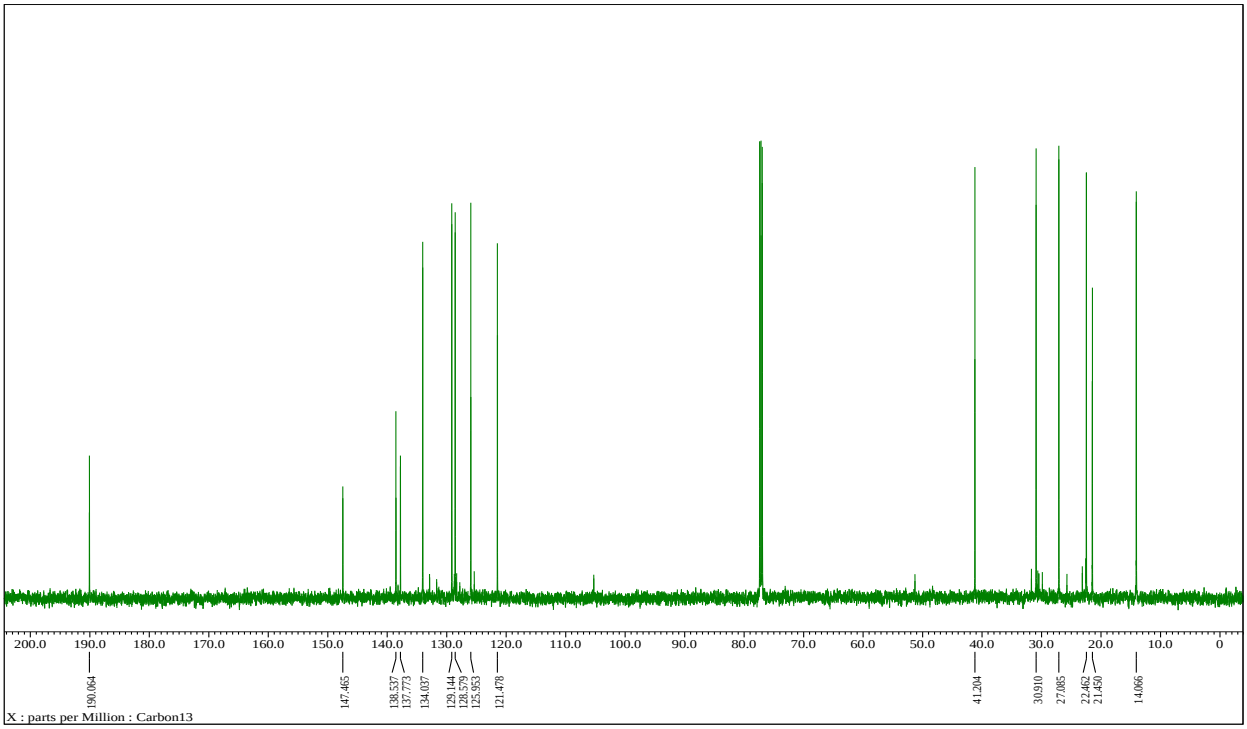
2.530
2.527
2.516
2.514
2.408
1.707
1.695
1.690
1.682
1.374
1.368
1.362
0.938
0.937
0.930
0.928
0.926
0.922
0.919
0.915
X : parts per Million : Proton


O
Cl

(Z)-3c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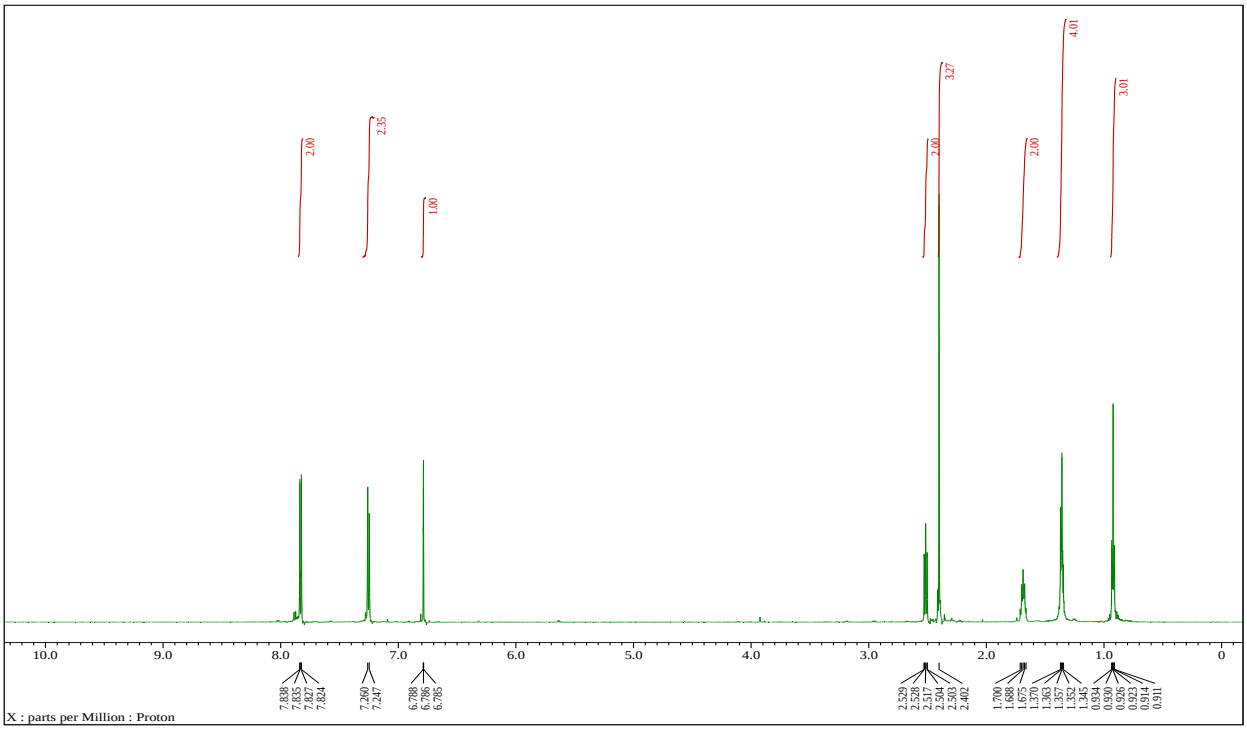
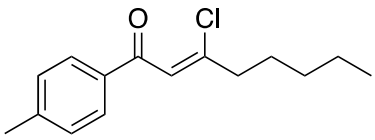
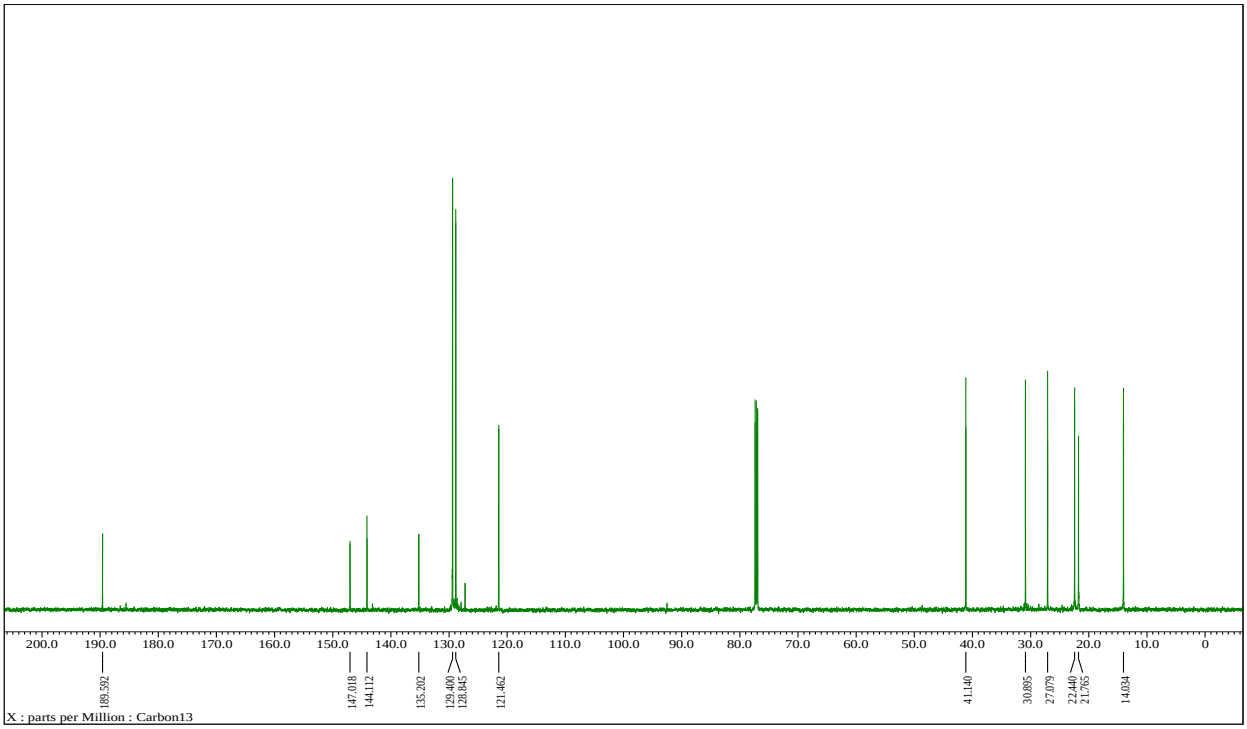
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
190.064
147.465
138.537
137.773
134.037
129.144
128.579
125.953
121.478
41.204
30.910
27.085
22.462
21.450
14.066
X : parts per Million : Carbon13


2.00
2.35
1.00
2.00
3.27
2.00
4.01
3.01
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.838
7.835
7.827
7.824
7.260
7.247
6.788
6.786
6.785
2.530
2.529
2.519
2.517
2.504
2.503
2.402
1.700
1.688
1.675
1.370
1.363
1.357
1.352
1.345
0.934
0.930
0.926
0.923
0.914
0.911
X : parts per Million : Proton


O
Cl

(*Z*)-3d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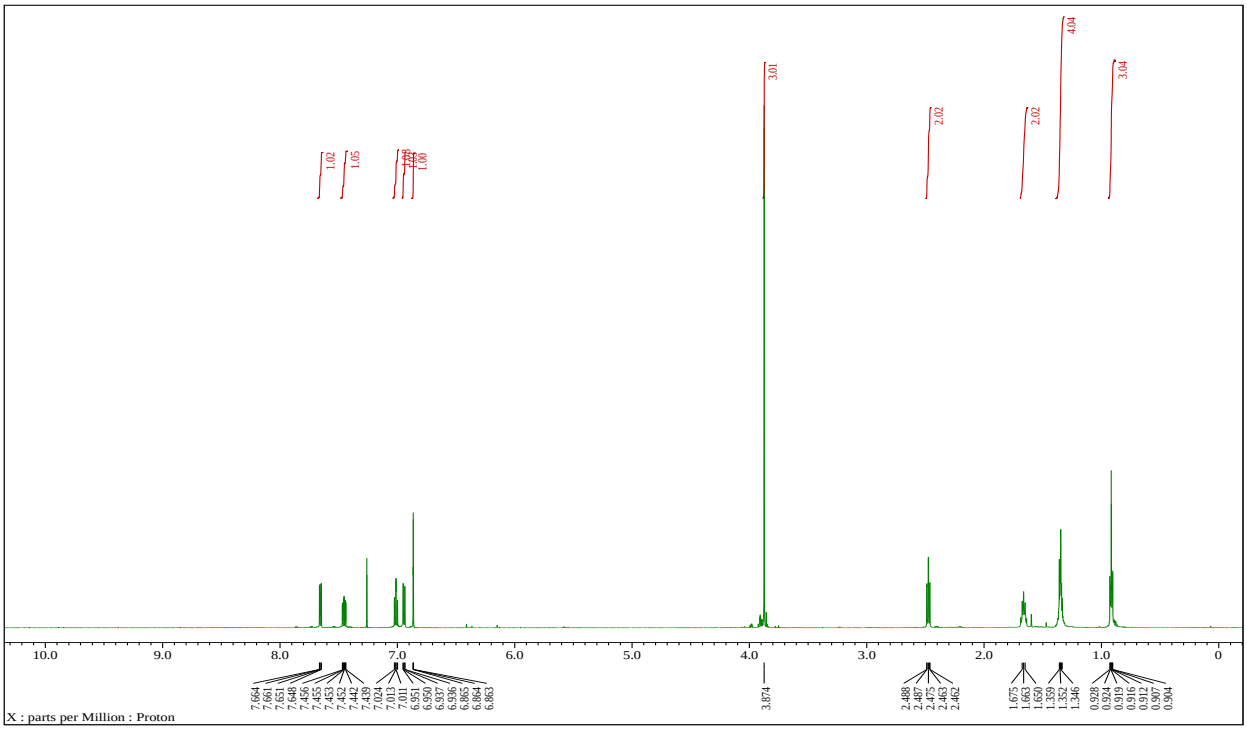
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
189.592
147.018
144.112
135.202
129.400
128.845
121.462
41.140
30.895
27.079
22.440
21.765
14.034
X : parts per Million : Carbon13


4.04
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3.04
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2.02
1.02
1.05
1.00
10.0
9.0
8.0
7.0
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2.487
2.475
2.463
2.462
1.675
1.663
1.650
1.359
1.352
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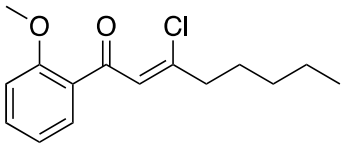
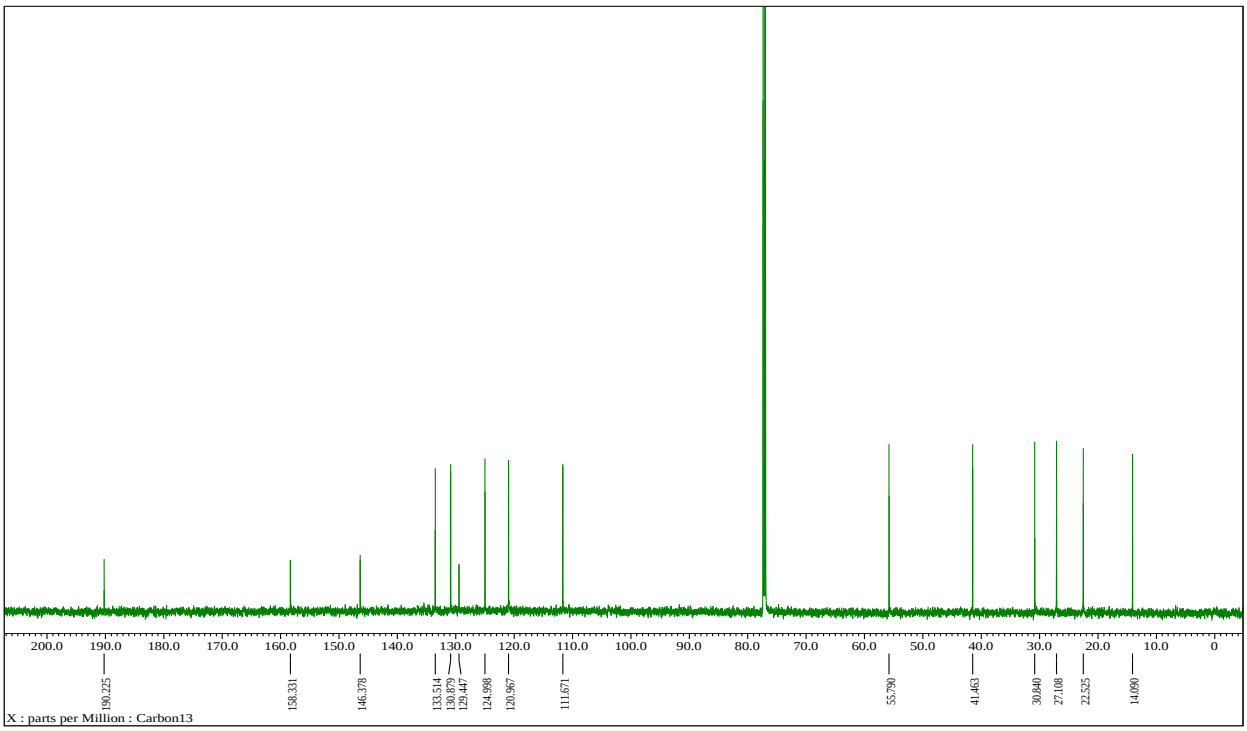
0.924
0.919
0.916
0.912
0.907
0.904
X : parts per Million : Proton


O
O
Cl

(*Z*)-3e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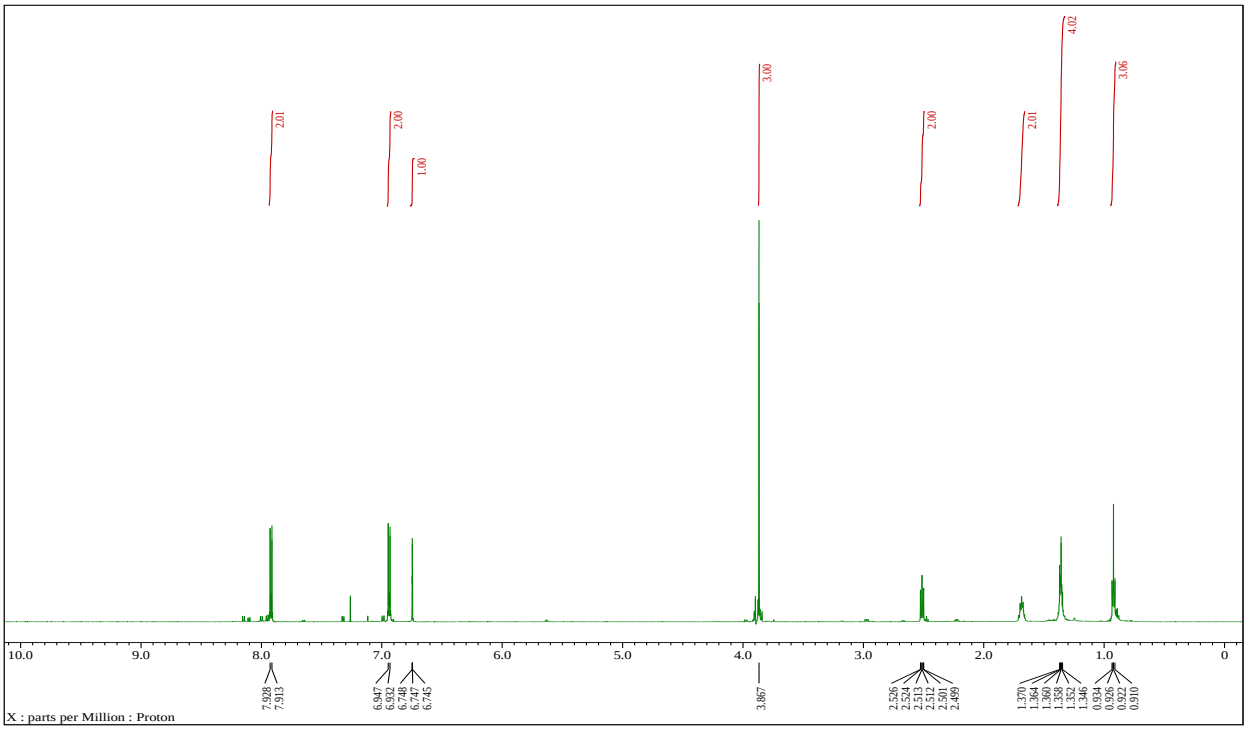
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
190.225
158.331
146.378
133.514
130.879
129.447
124.998
120.967
111.671
55.790
41.463
30.840
27.108
22.525
14.090
X : parts per Million : Carbon13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2.00
2.01
4.02
3.06
10.0
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8.0
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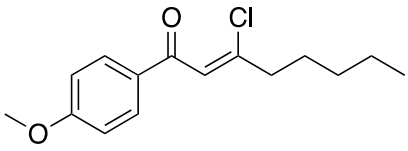
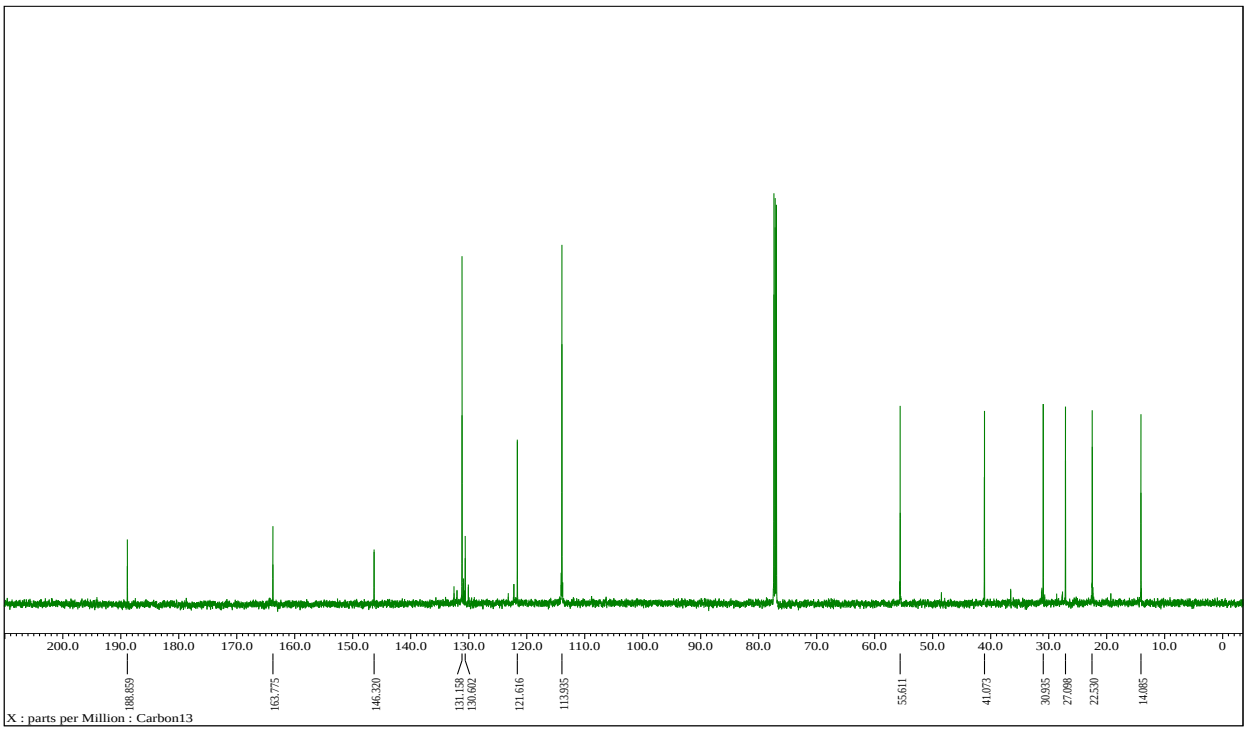
6.0
5.0
4.0
3.0
2.0
1.0
0
7.928
7.913
6.947
6.932
6.748
6.747
6.745
3.867
2.526
2.524
2.513
2.512
2.501
2.499
1.370
1.364
1.360
1.358
1.332
1.146
0.934
0.926
0.922
0.910
X : parts per Million : Proton


O
Cl
O

(*Z*)-3f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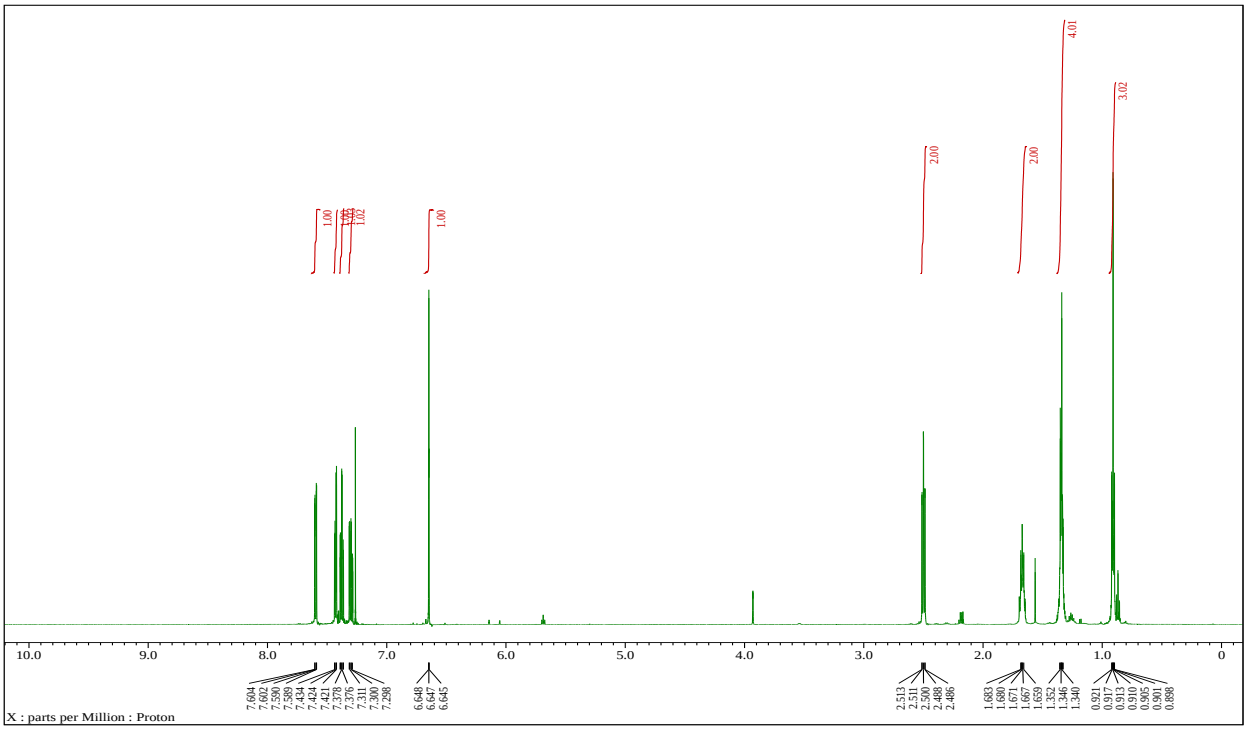
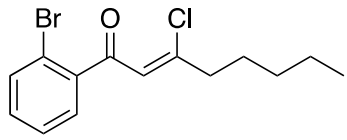
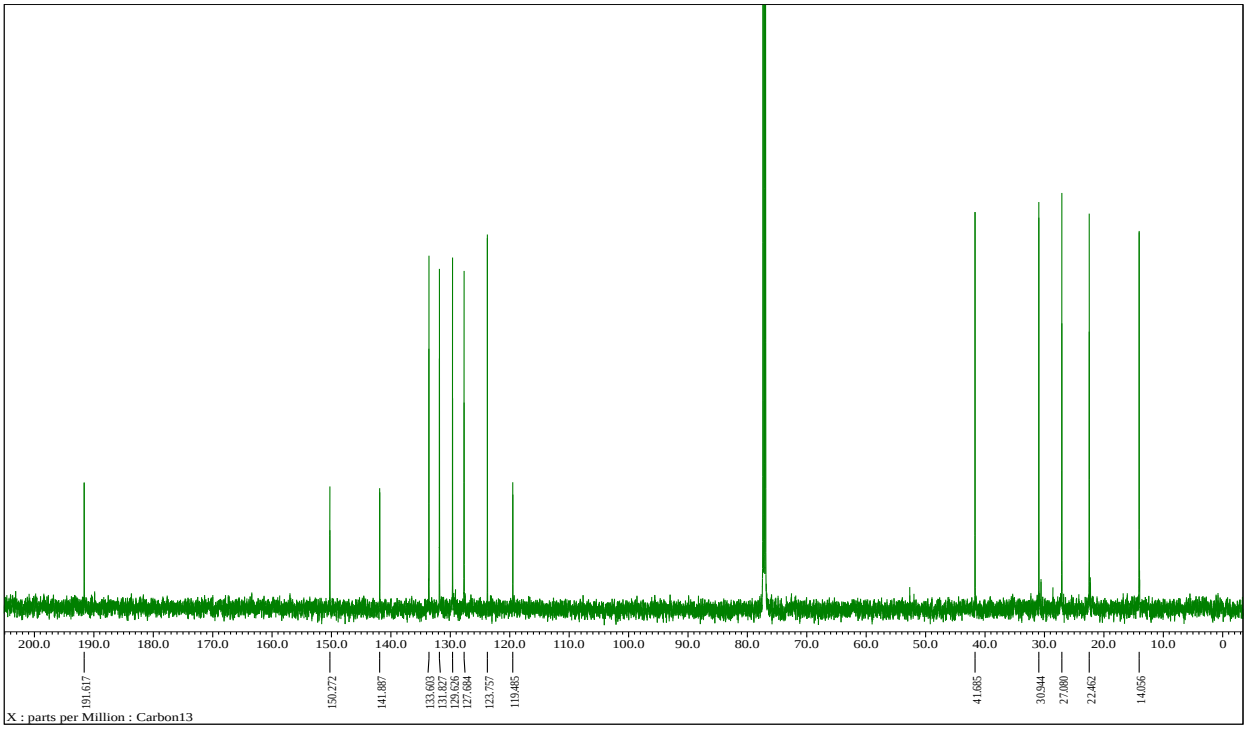
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
188.859
163.775
146.320
131.158
130.602
121.616
113.935
55.611
41.073
30.935
27.098
22.530
14.085
X : parts per Million : Carbon13


4.01
3.02
2.00
2.00
1.00
1.00
1.00
1.00
1.02
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.694
7.602
7.590
7.589
7.434
7.421
7.378
7.376
7.311
7.300
7.298
6.648
6.647
6.645
2.513
2.511
2.500
2.488
2.486
1.683
1.680
1.671
1.667
1.639
1.352
1.346
1.340
0.921
0.917
0.913
0.910
0.905
0.901
0.898
X : parts per Million : Proton


Br
O
Cl

(*Z*)-3g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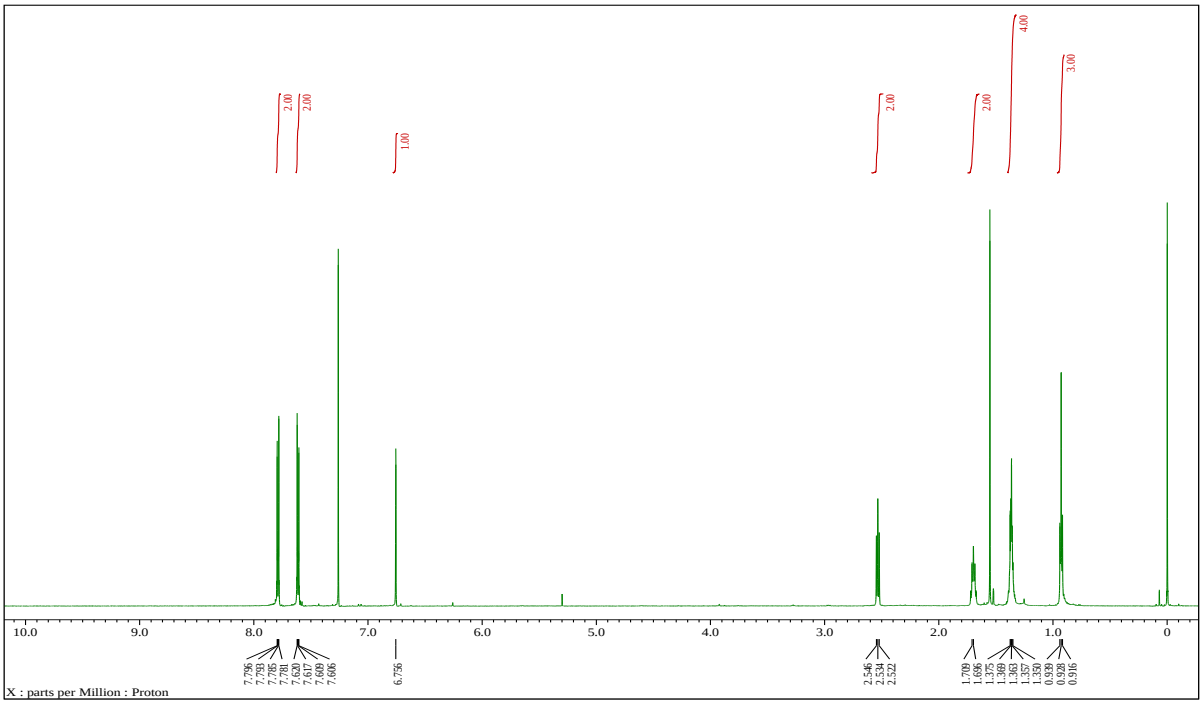
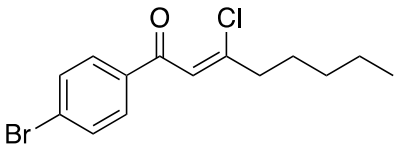
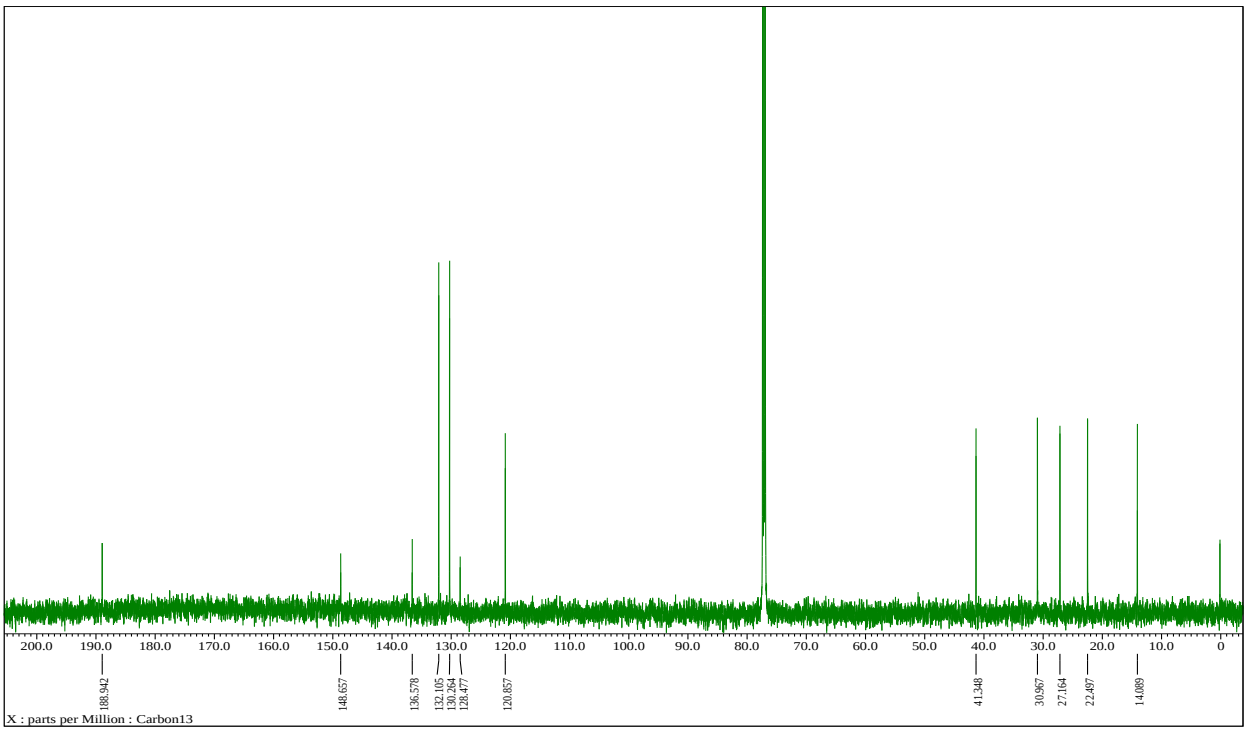
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
191.617
150.272
141.887
133.603
131.827
129.626
127.684
123.757
119.485
41.685
30.944
27.080
22.462
14.056
X : parts per Million : Carbon13


2.00
2.00
1.00
2.00
2.00
4.00
3.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.796
7.793
7.785
7.782
7.620
7.617
7.609
7.606
6.756
2.546
2.534
2.522
1.709
1.696
1.375
1.369
1.363
1.357
1.330
0.939
0.928
0.916
X : parts per Million : Proton


O
Cl
Br

(*Z*)-3h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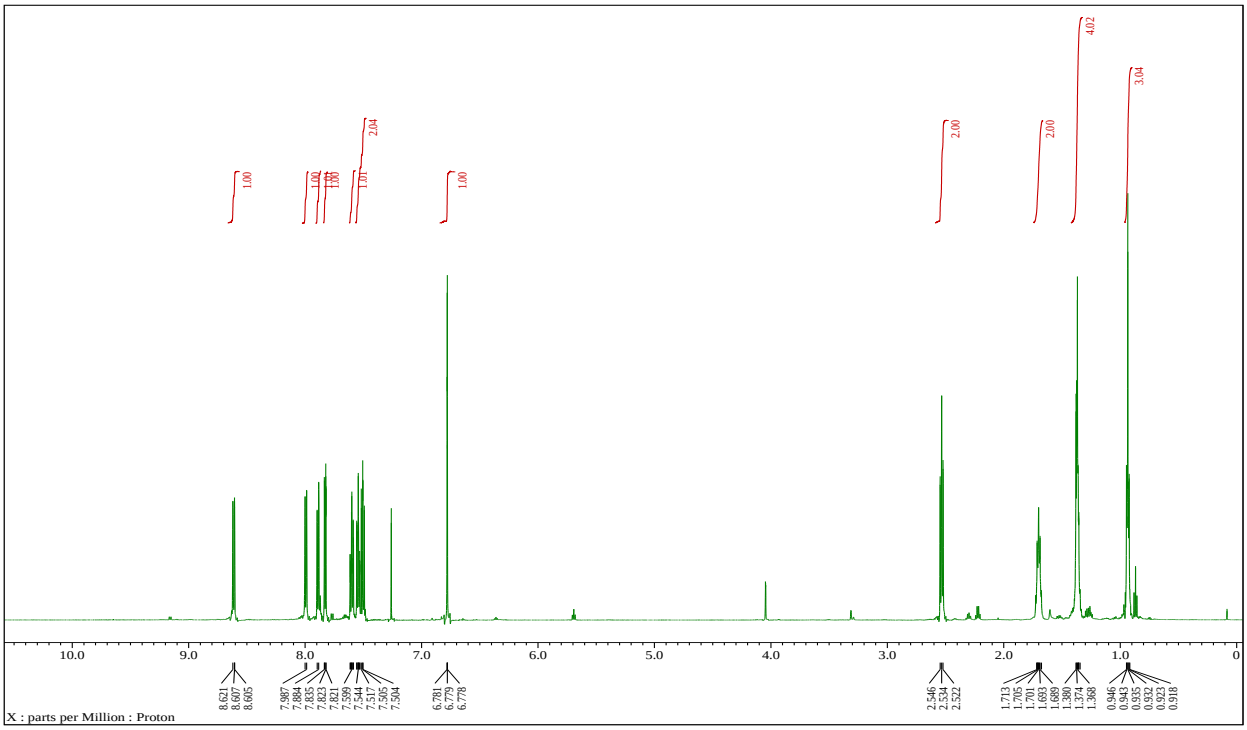
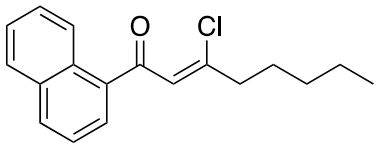
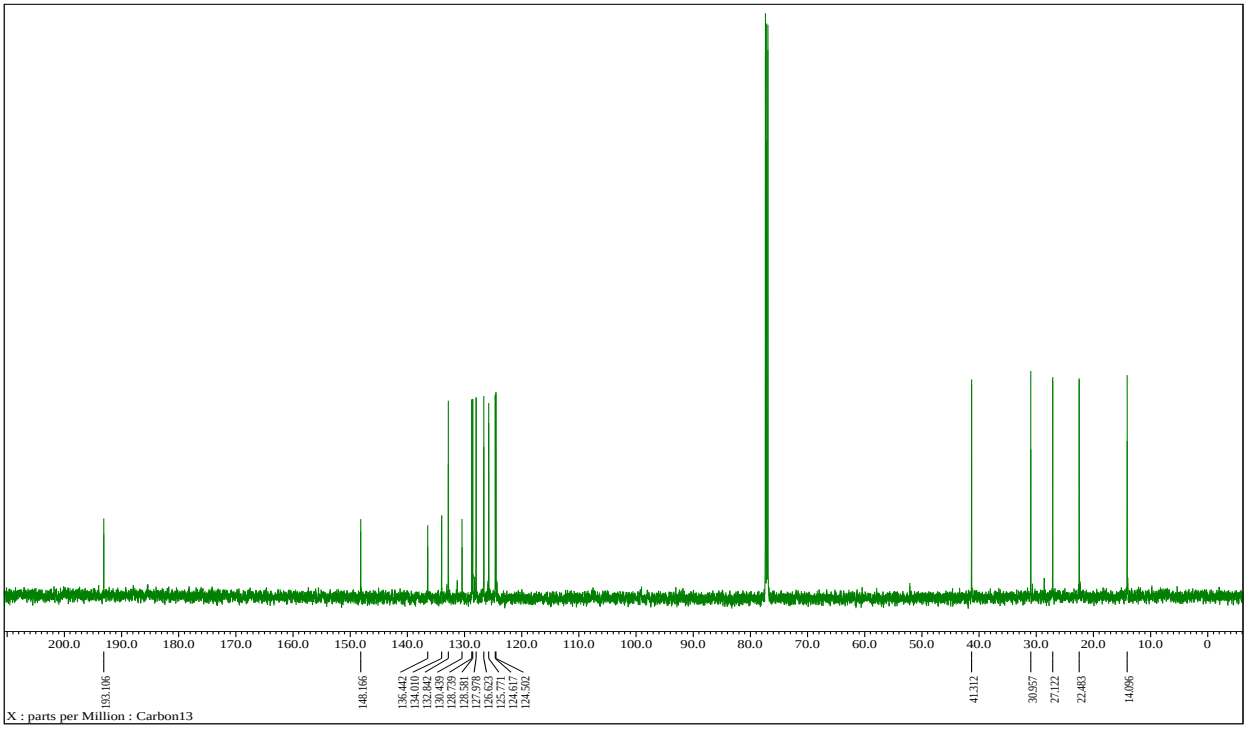
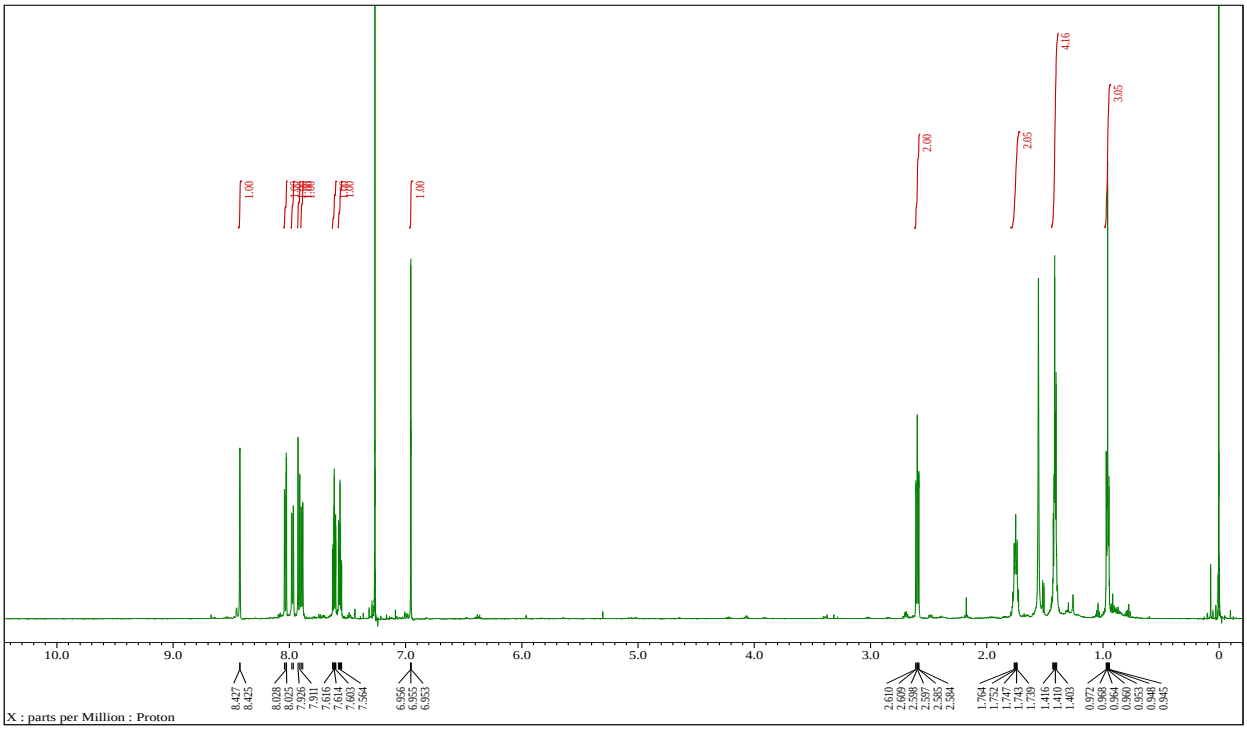
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
188.942
148.657
136.578
132.105
130.264
128.477
120.857
41.348
30.967
27.164
22.497
14.089
X : parts per Million : Carbon13

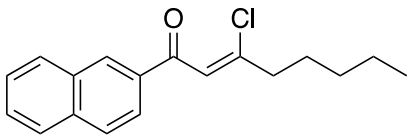




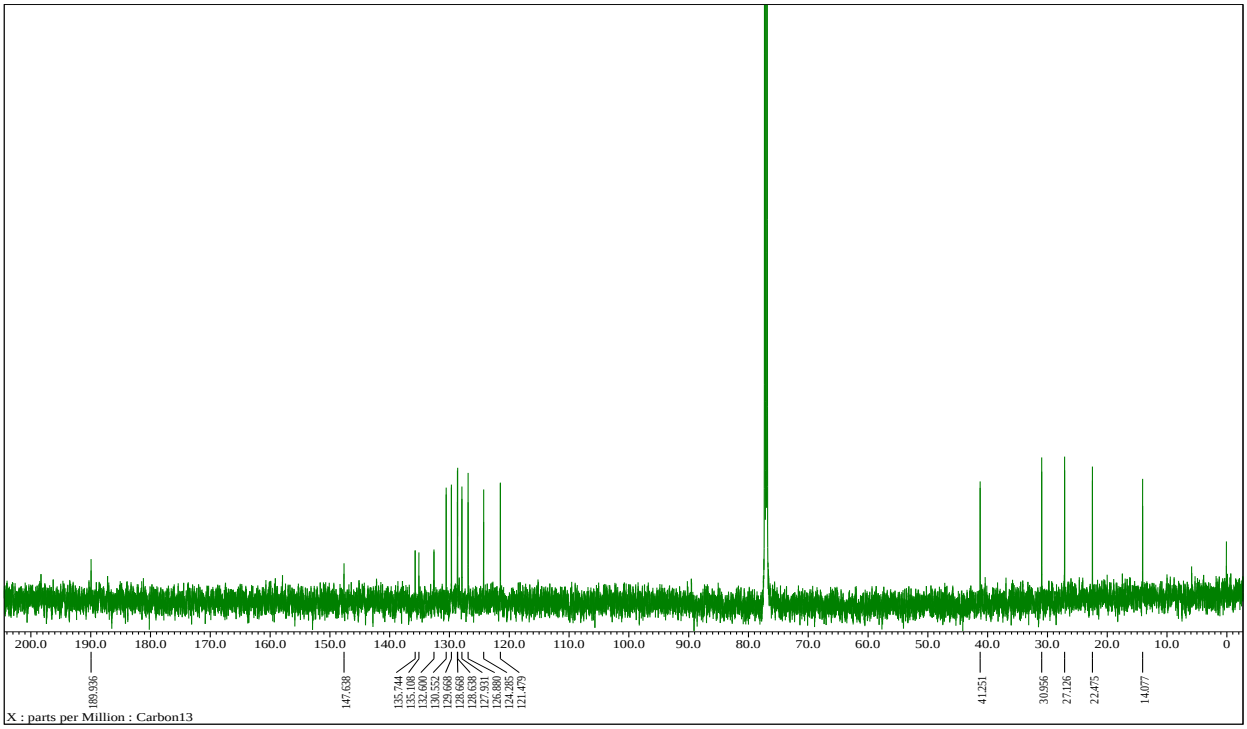
(Z)-3i

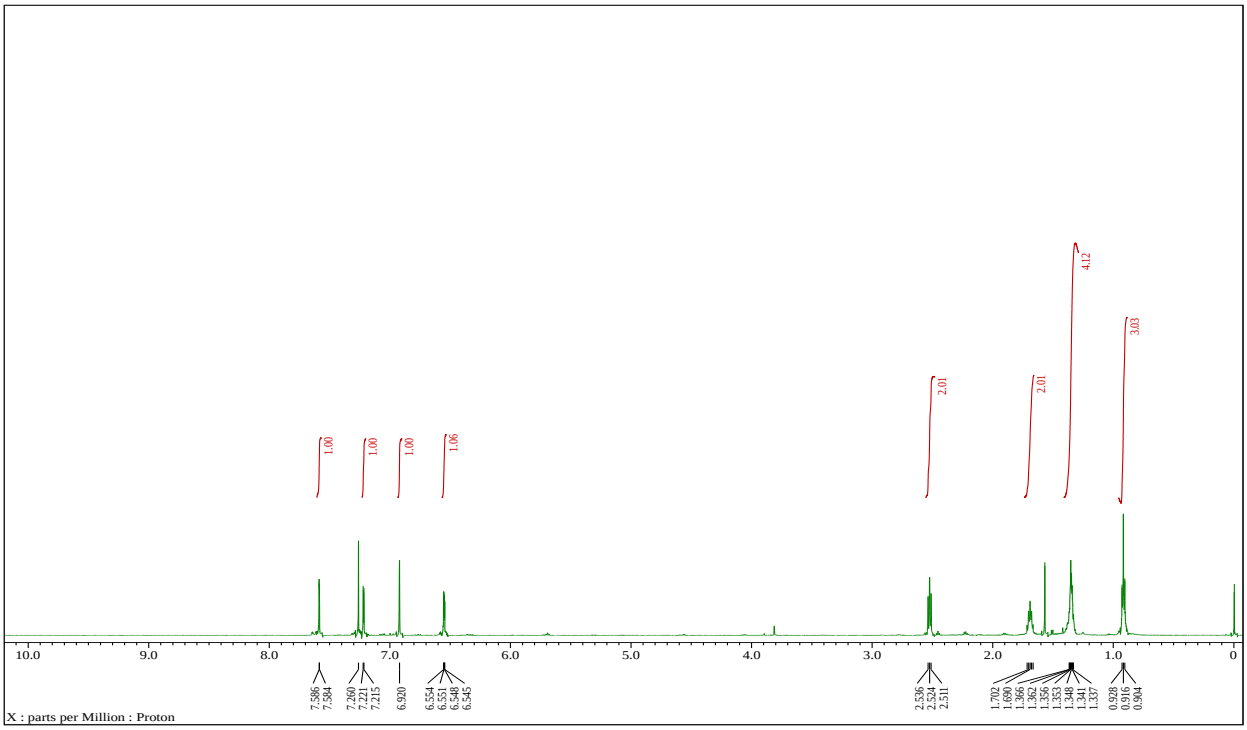


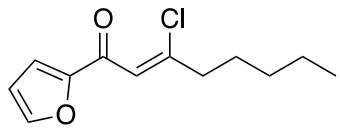

4.16
3.05
2.00
2.05
1.00
1.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.427
8.425
8.028
8.025
7.926
7.911
7.616
7.614
7.603
7.564
6.956
6.955
6.553
2.610
2.609
2.598
2.597
2.585
2.584
1.764
1.752
1.747
1.743
1.739
1.416
1.410
1.403
0.972
0.968
0.964
0.960
0.953
0.948
0.945
X : parts per Million : Proton


O
Cl

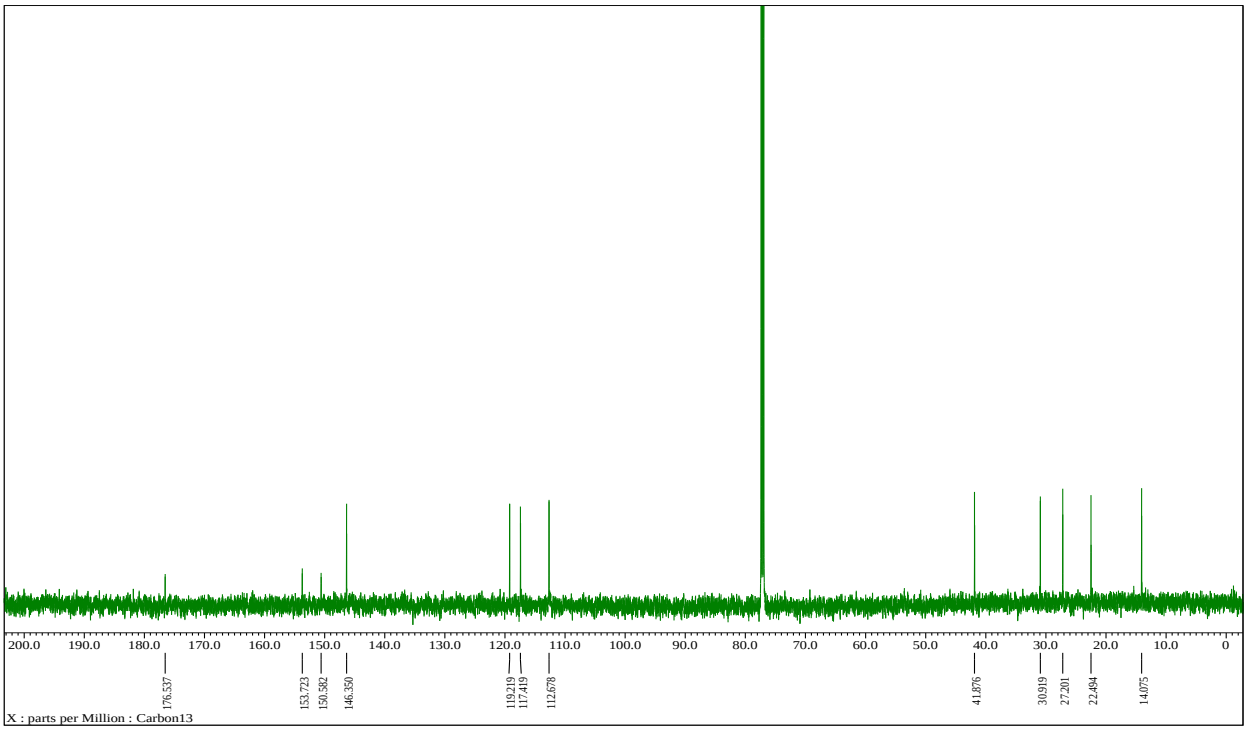
(Z)-3j

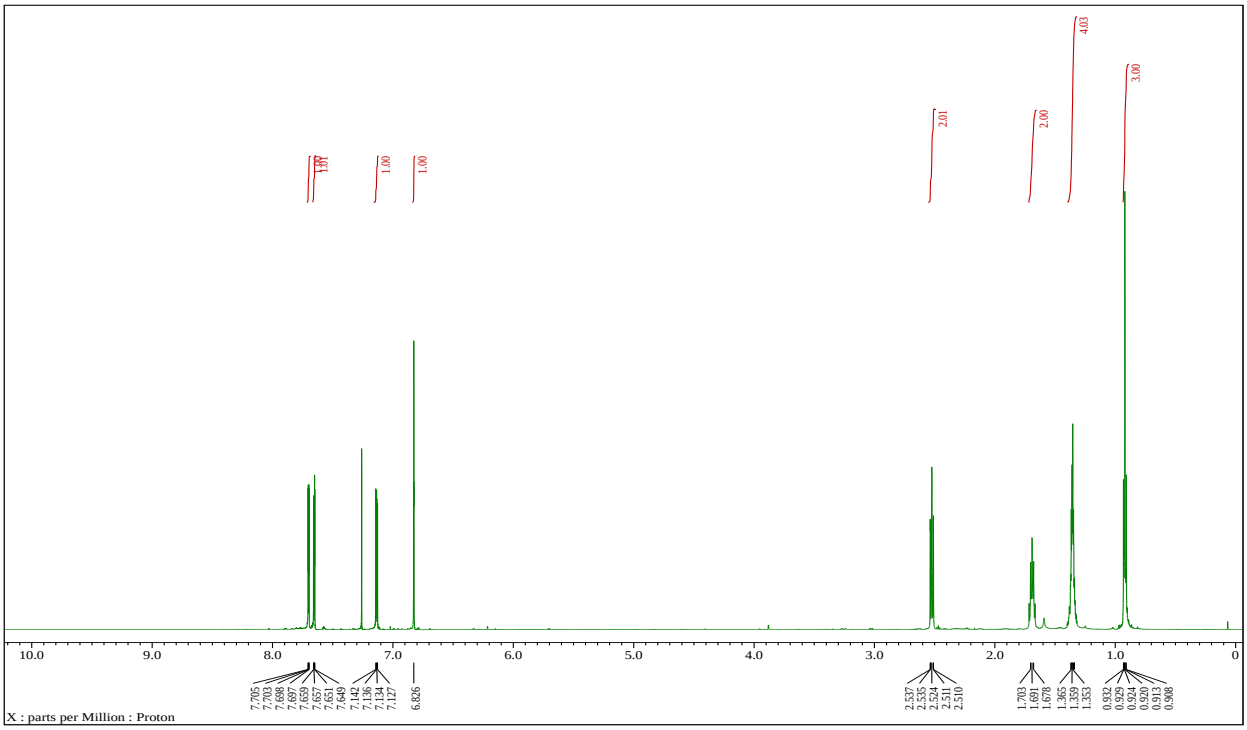

200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
189.936
147.638
135.744
135.189
132.600
130.552
129.668
128.668
128.638
127.951
126.880
124.385
121.479
41.251
30.956
27.126
22.475
14.077
X : parts per Million : Carbon13

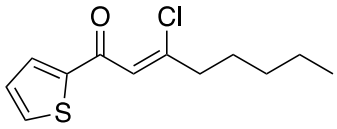

1.00
1.00
1.00
1.06
2.01
2.01
4.12
3.03
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.586
7.584
7.260
7.221
7.215
6.920
6.554
6.551
6.549
6.545
2.536
2.524
2.511
1.702
1.690
1.366
1.362
1.356
1.353
1.348
1.341
1.337
0.928
0.916
0.904
X : parts per Million : Proton


O
Cl
O

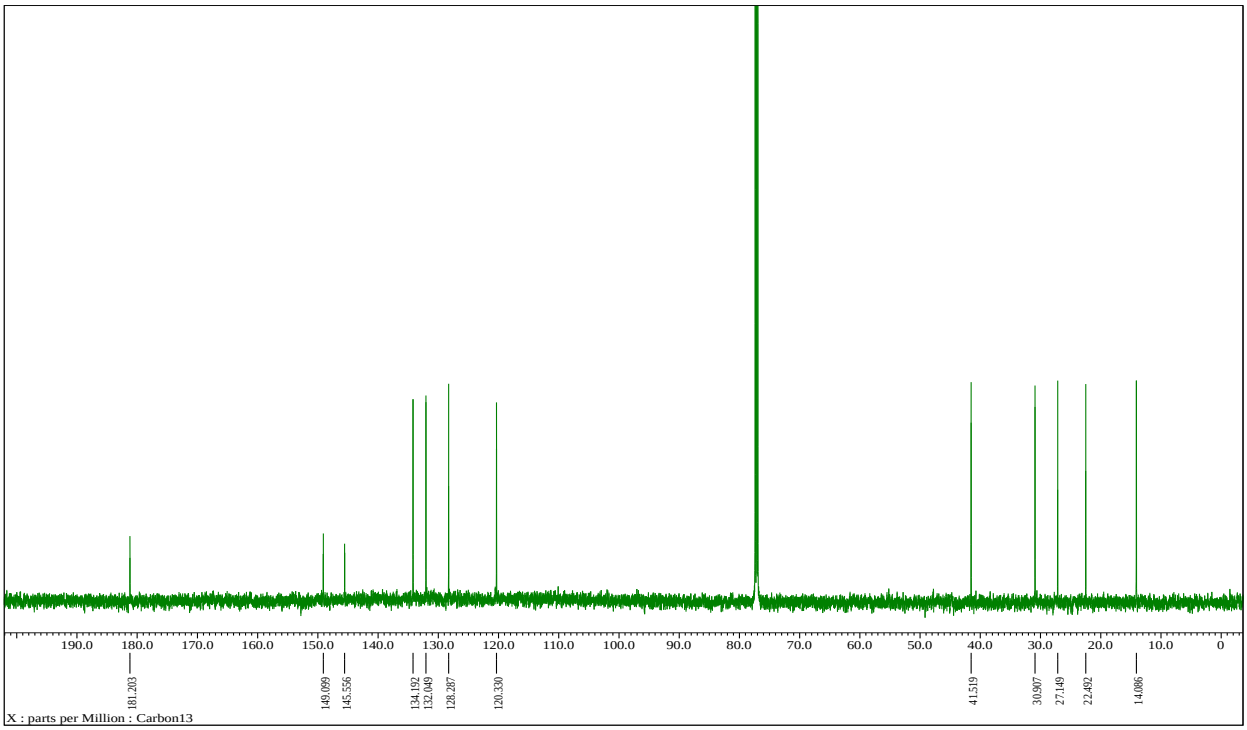
(*Z*)-3k

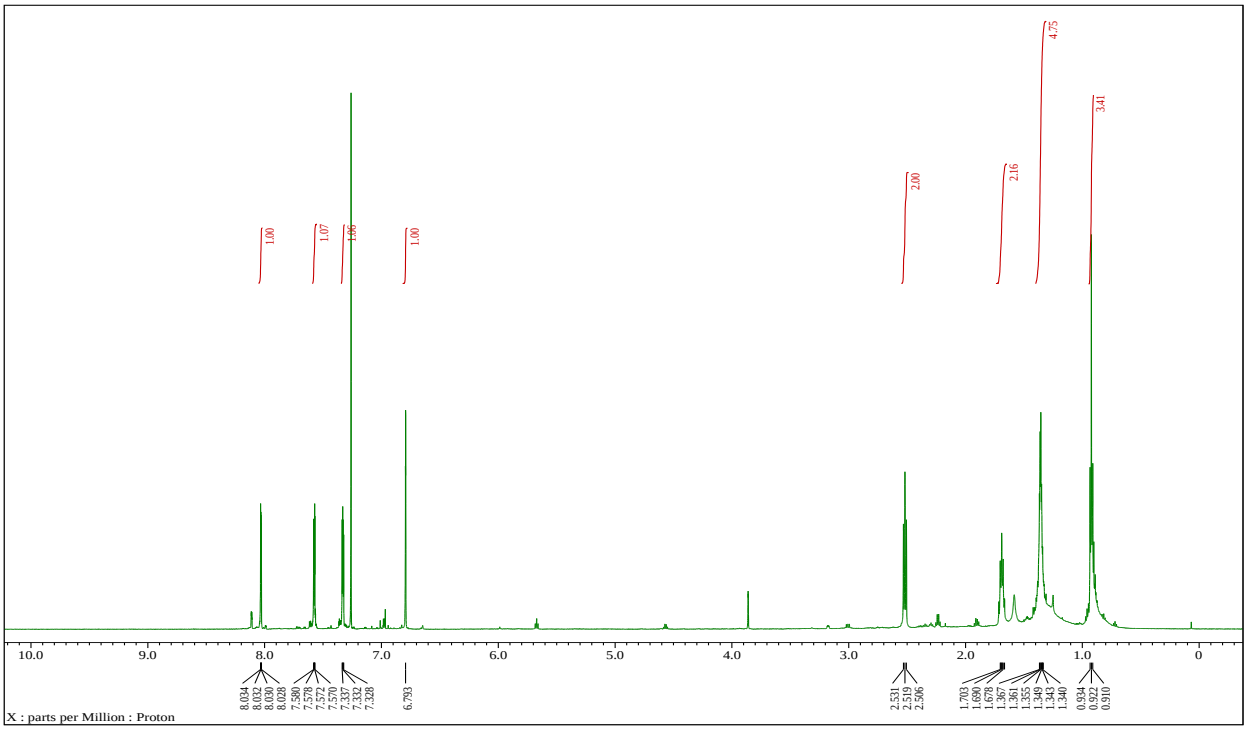

200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
176.537
153.723
150.582
146.350
119.219
117.419
112.678
41.876
30.919
27.201
22.494
14.075
X : parts per Million : Carbon13

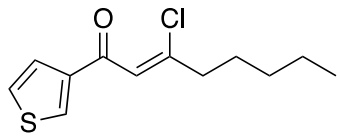

4.03
3.00
2.01
2.00
1.00
1.00
7.705
7.703
7.698
7.697
7.659
7.657
7.651
7.649
7.142
7.136
7.134
7.127
6.826
2.537
2.535
2.524
2.511
2.510
1.703
1.691
1.678
1.365
1.359
1.353
0.932
0.929
0.924
0.920
0.913
0.908
X : parts per Million : Proton


O
Cl
S

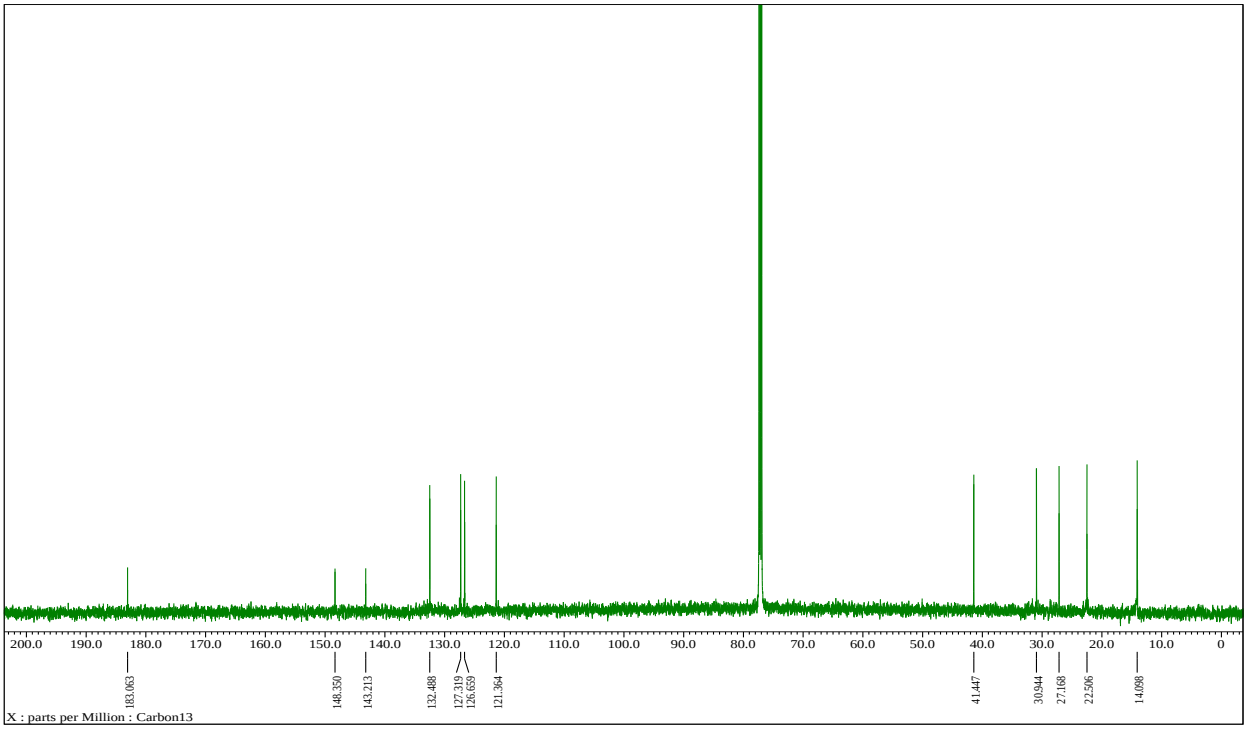
(*Z*)-3l

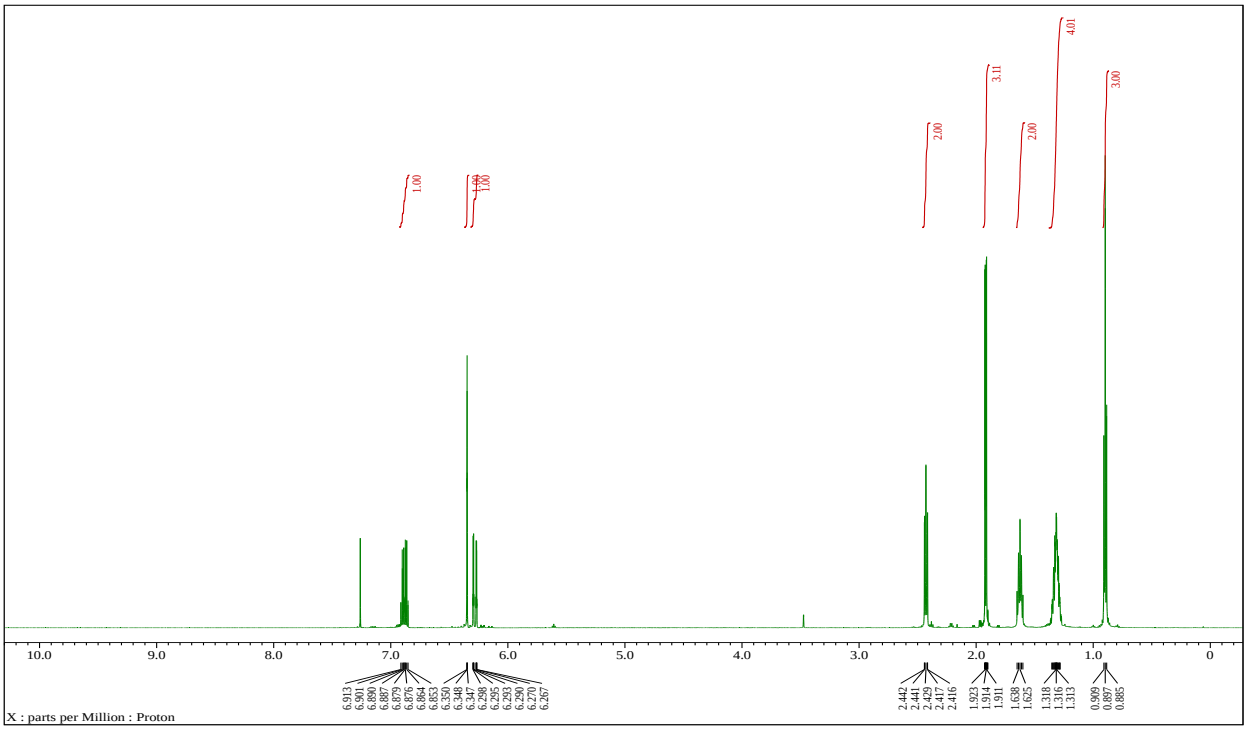

181.203
149.099
145.556
134.192
132.049
128.287
120.330
41.519
30.907
27.149
22.492
14.086
X : parts per Million : Carbon13

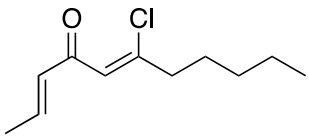

4.75
3.41
2.16
2.00
1.00
1.07
1.66
1.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.034
8.032
8.030
8.028
7.580
7.578
7.572
7.570
7.337
7.332
7.328
6.793
2.531
2.519
2.506
1.703
1.690
1.678
1.367
1.361
1.355
1.349
1.343
1.340
0.934
0.922
0.910
X : parts per Million : Proton


O
Cl
S

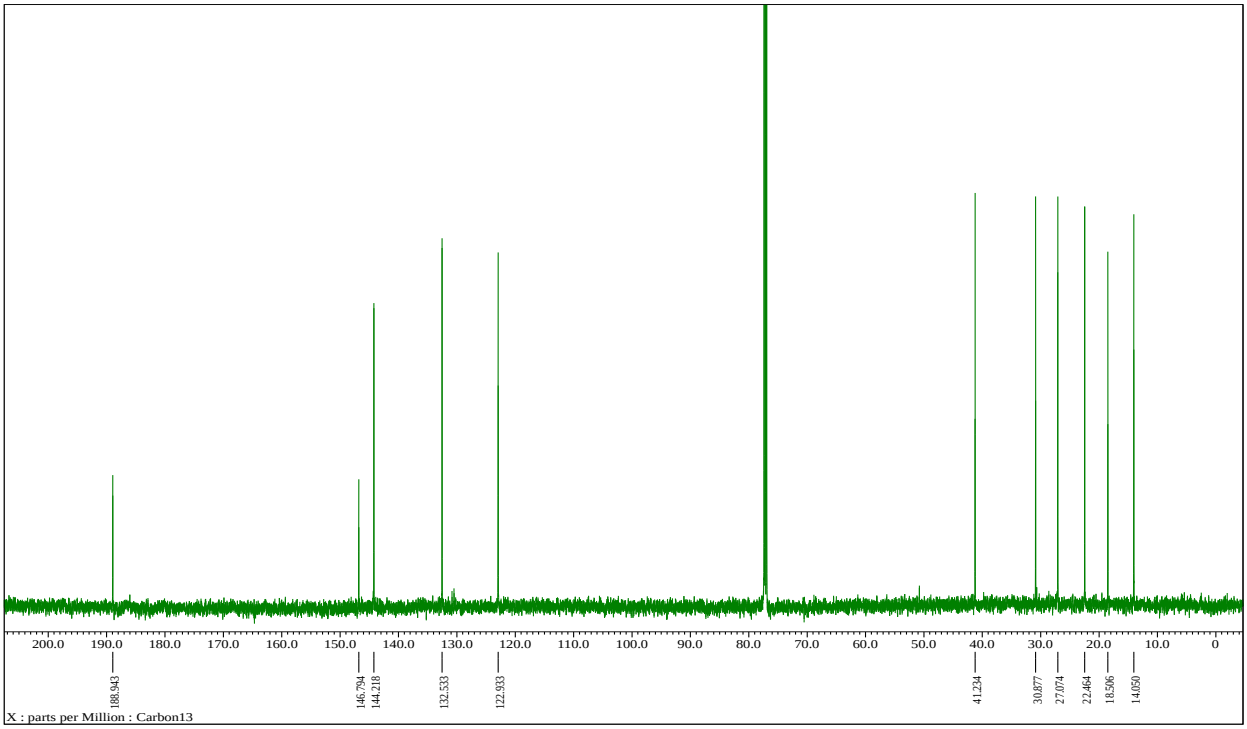
(Z)-3m

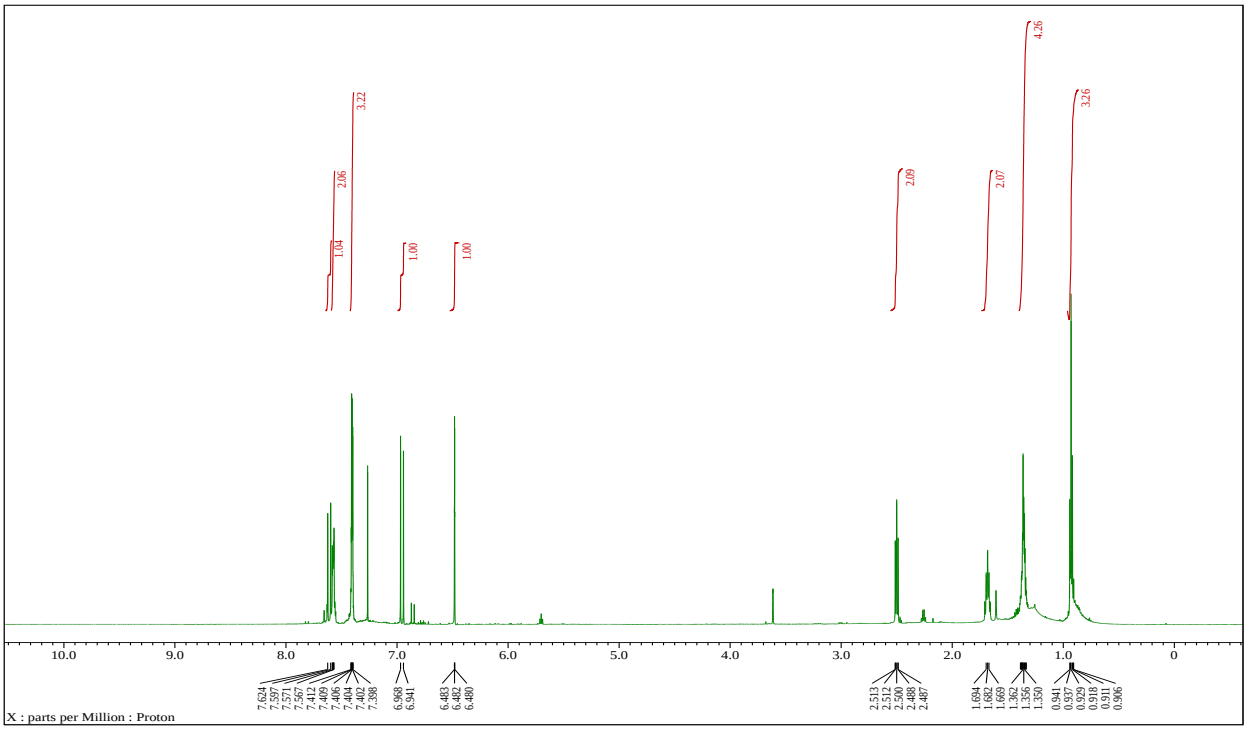

200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
183.063
148.350
143.213
132.488
127.319
126.659
121.364
41.447
30.944
27.168
22.506
14.098
X : parts per Million : Carbon13

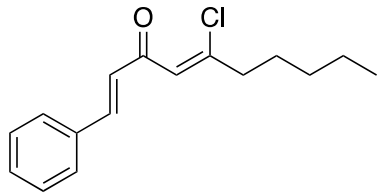

1.00
1.00
1.00
2.00
3.11
2.00
4.01
3.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
6.913
6.901
6.890
6.887
6.879
6.876
6.864
6.853
6.350
6.348
6.347
6.298
6.295
6.293
6.290
6.270
6.267
2.442
2.441
2.429
2.417
2.416
1.923
1.914
1.911
1.638
1.625
1.318
1.316
1.313
0.909
0.897
0.885
X : parts per Million : Proton


O
Cl

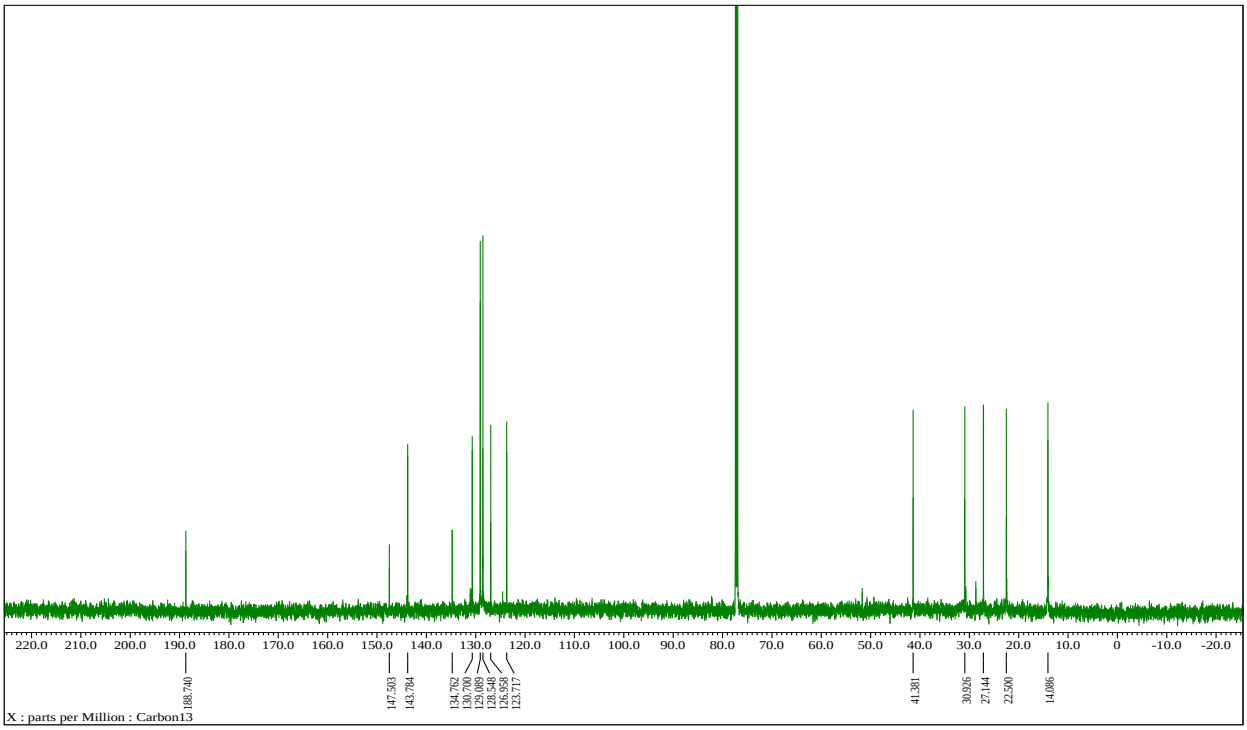
(*Z*)-3n

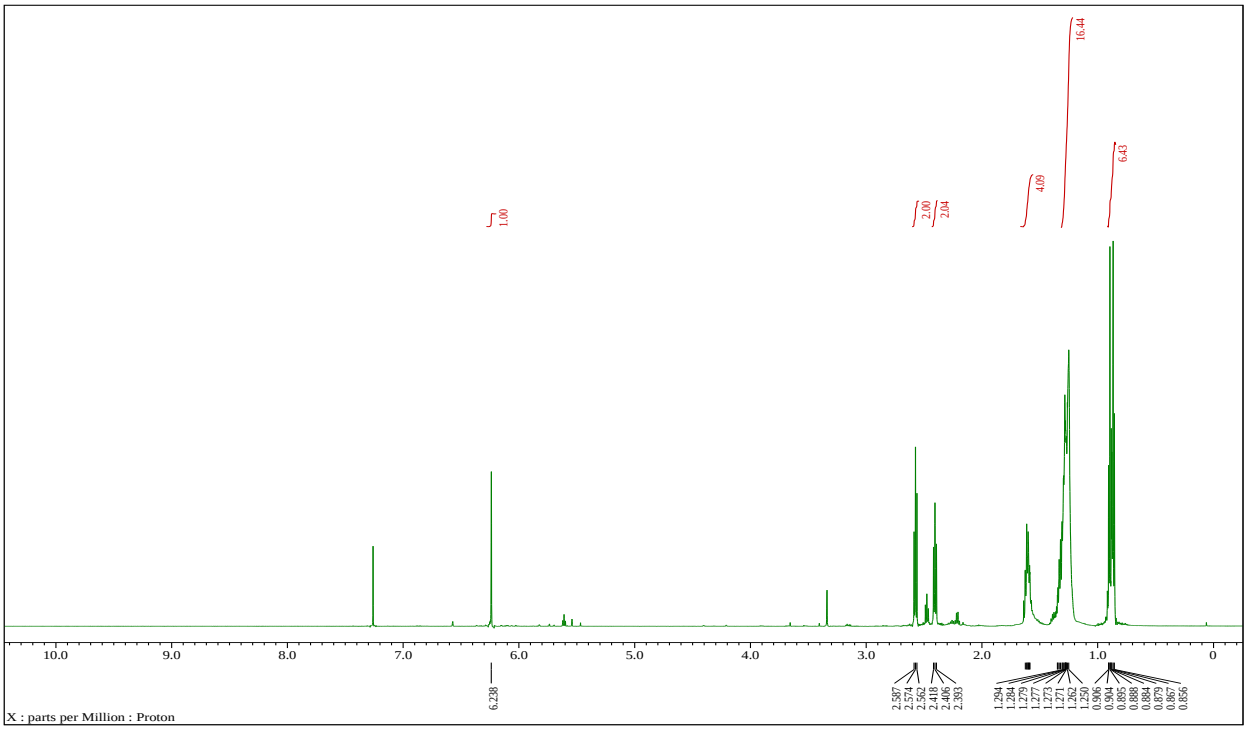

200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
188.943
146.794
144.218
132.533
122.933
41.234
30.877
27.074
22.464
18.506
14.050
X : parts per Million : Carbon13

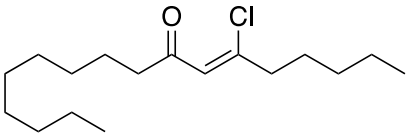

4.26
3.22
3.26
2.06
2.09
2.07
1.04
1.00
1.00
1.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.624
7.597
7.571
7.567
7.412
7.409
7.406
7.404
7.402
7.398
6.968
6.941
6.483
6.482
6.480
2.513
2.512
2.500
2.488
2.487
1.694
1.682
1.669
1.362
1.356
1.350
0.941
0.937
0.929
0.918
0.911
0.906
X : parts per Million : Proton


O
Cl

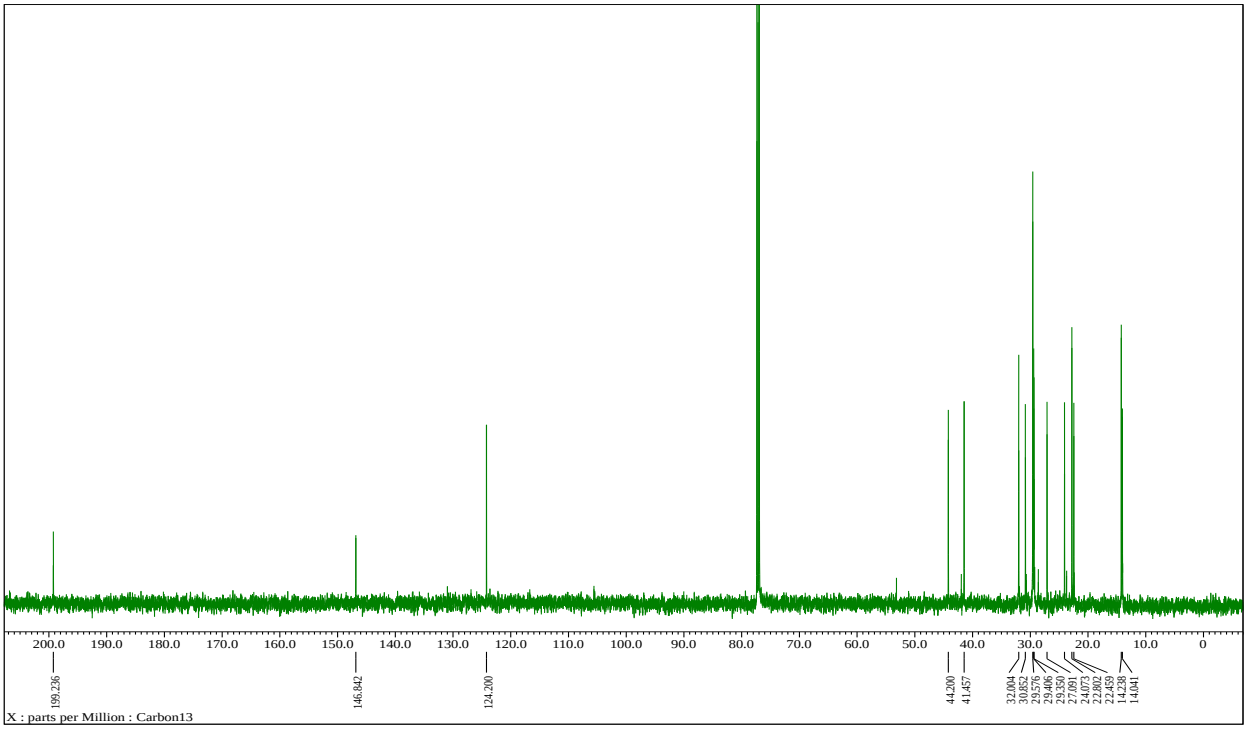
(*Z*)-3o


220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
-20.0
188.740
147.503
143.784
134.762
130.700
129.089
128.548
126.558
123.717
41.381
30.926
27.144
22.500
14.086
X : parts per Million : Carbon13


16.44
6.43
4.09
2.00
2.04
1.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
6.238
2.587
2.574
2.562
2.418
2.406
2.393
1.294
1.284
1.279
1.277
1.273
1.271
1.262
1.250
0.906
0.904
0.895
0.888
0.884
0.879
0.867
0.856
X : parts per Million : Proton


O
Cl

(*Z*)-3p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160.0
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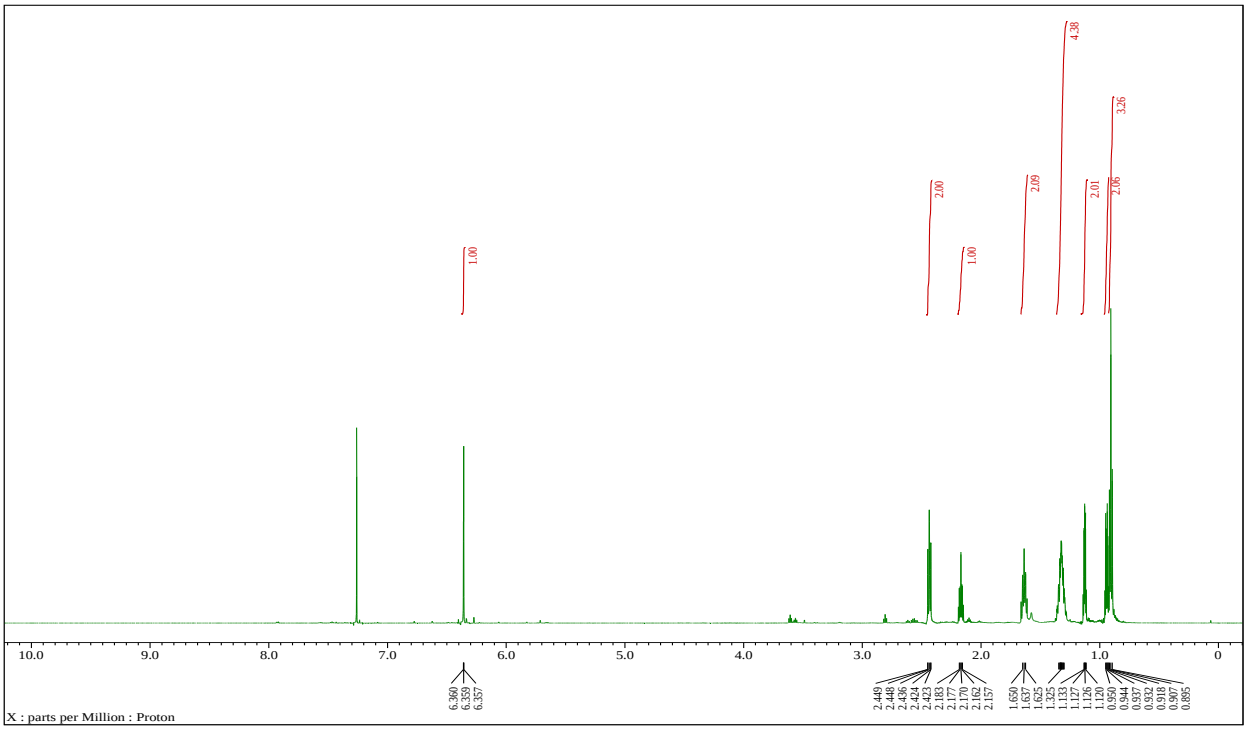
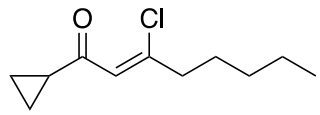
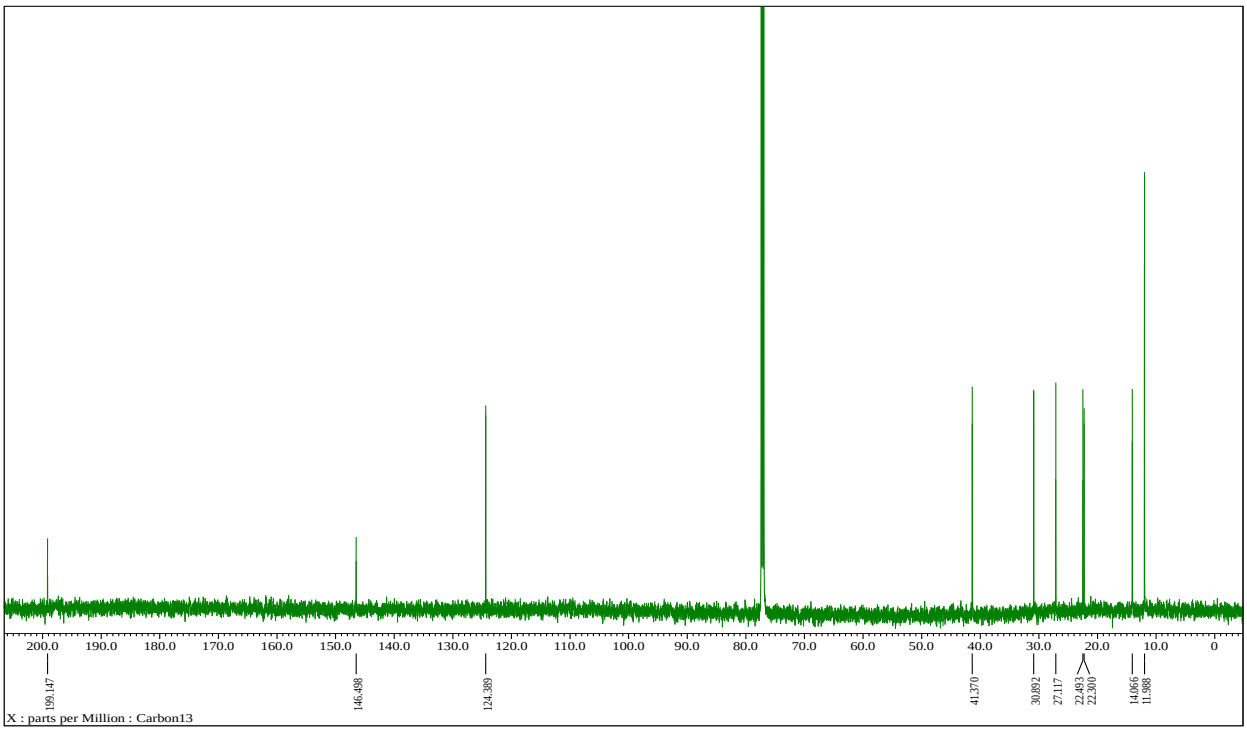
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
199.236
146.842
124.200
44.200
41.457
32.004
30.852
29.576
29.406
29.350
27.091
24.972
22.580
22.459
14.238
14.041
X : parts per Million : Carbon13


4.38
3.26
2.00
2.09
2.01
2.06
1.00
1.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
6.360
6.359
6.357
2.449
2.448
2.436
2.424
2.423
2.183
2.177
2.170
2.162
2.157
1.650
1.637
1.625
1.325
1.133
1.127
1.126
1.120
0.950
0.944
0.537
0.932
0.918
0.907
0.895
X : parts per Million : Proton


O
Cl

(*Z*)-3q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190.0
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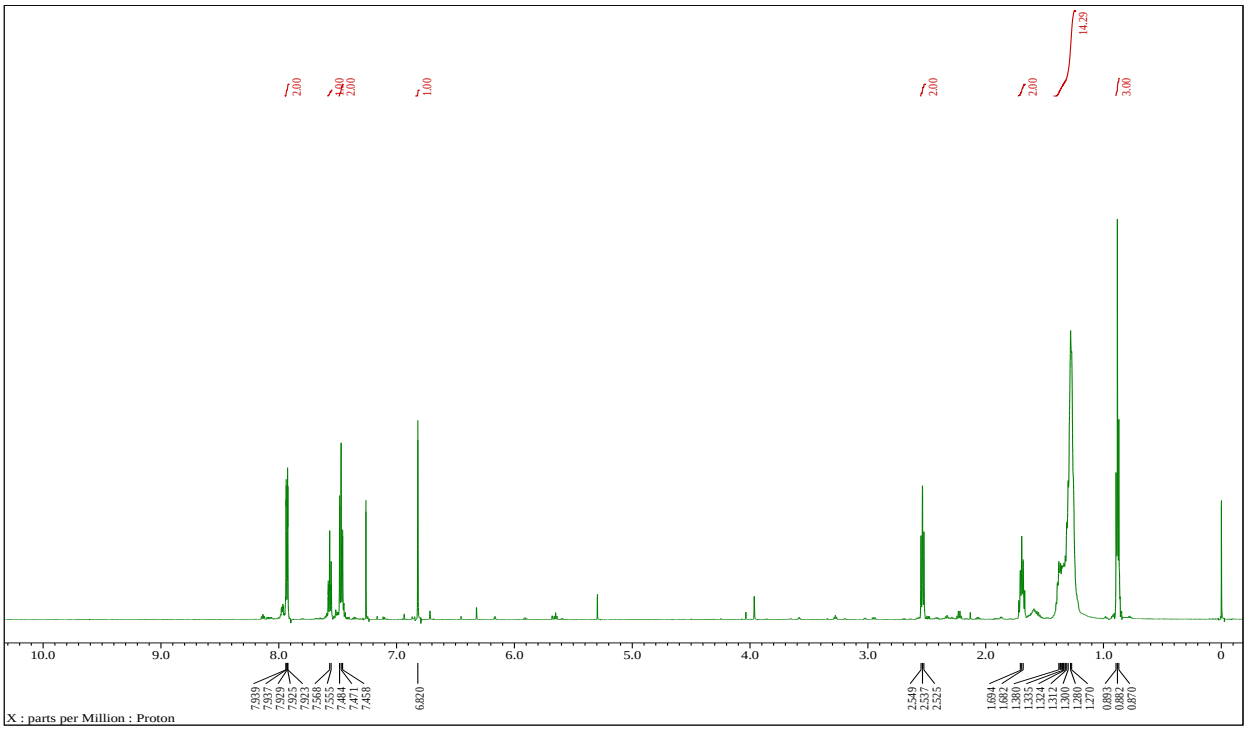
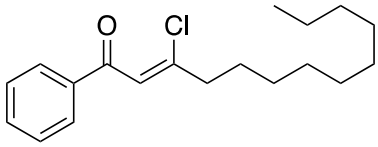
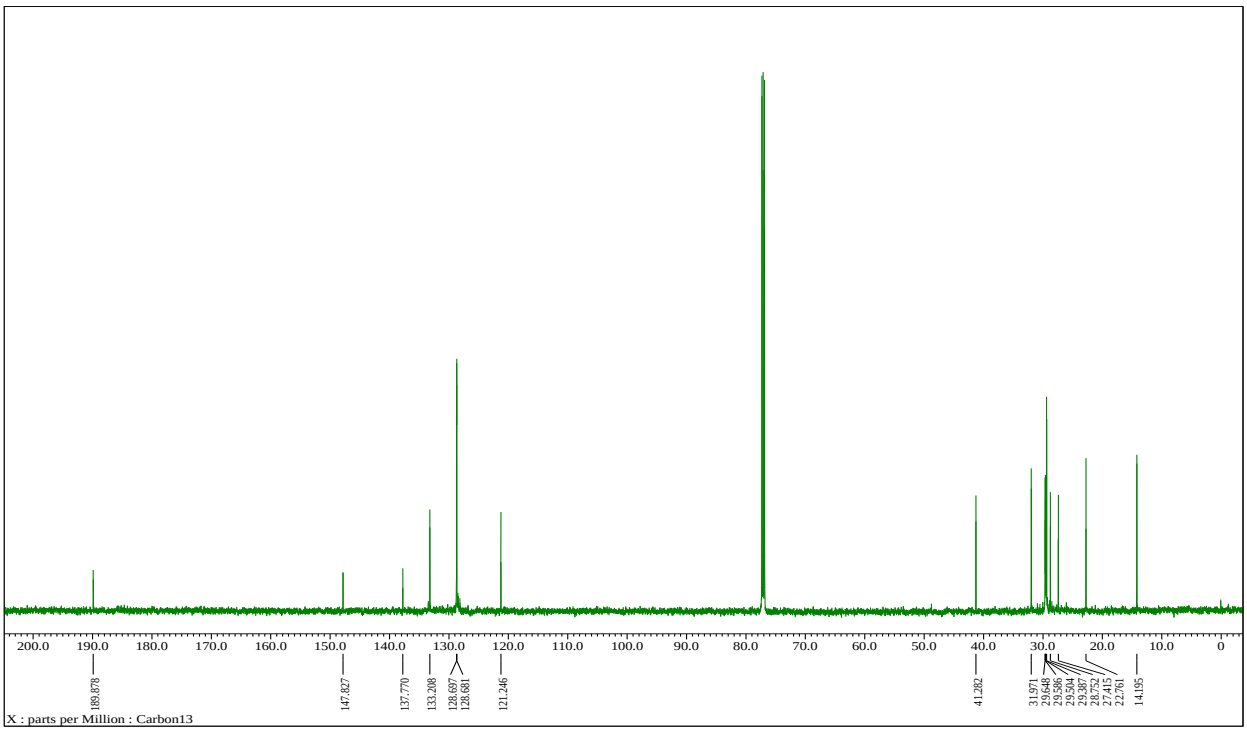
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
199.147
146.498
124.389
41.370
30.892
27.117
22.493
22.300
14.066
11.988
X : parts per Million : Carbon13


2.00
2.00
2.00
1.00
2.00
2.00
14.29
3.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.939
7.937
7.929
7.925
7.923
7.568
7.555
7.544
7.471
7.458
6.820
2.549
2.537
2.525
1.694
1.682
1.380
1.335
1.324
1.312
1.300
1.280
1.270
0.893
0.882
0.870
X : parts per Million : Proton


O
Cl

(*Z*)-3r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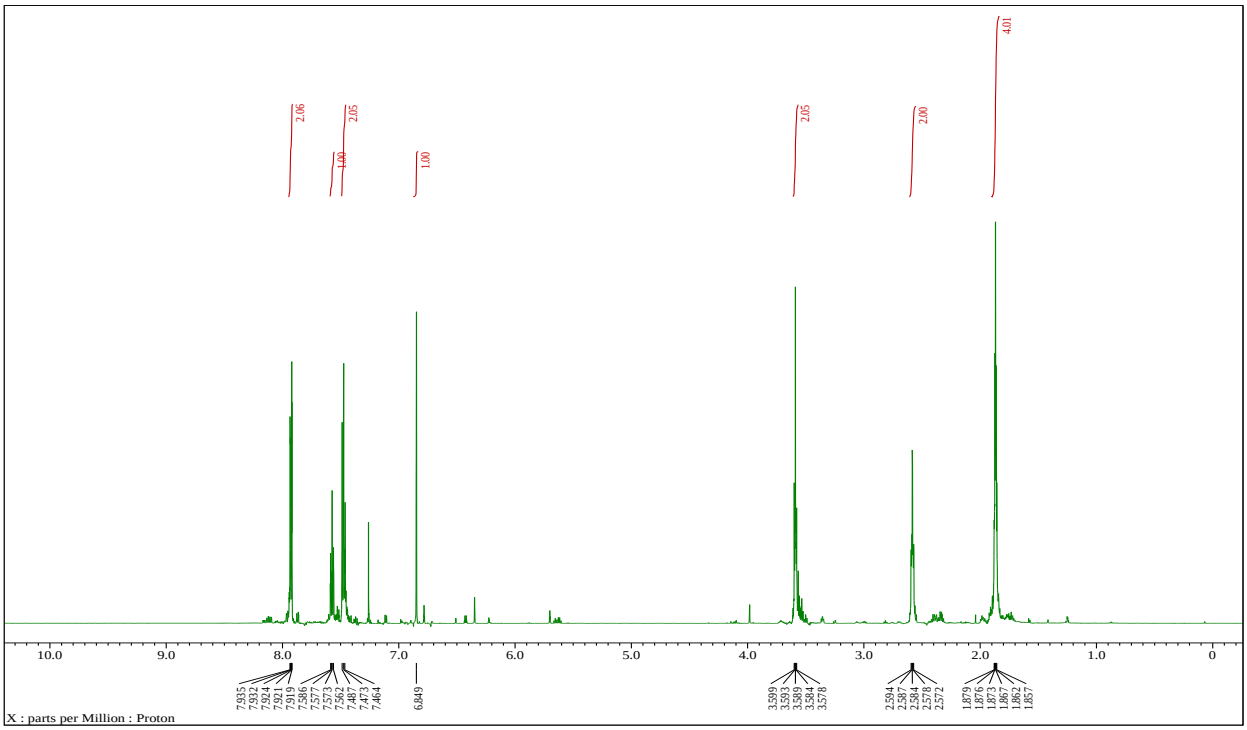
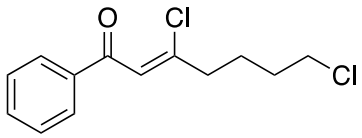
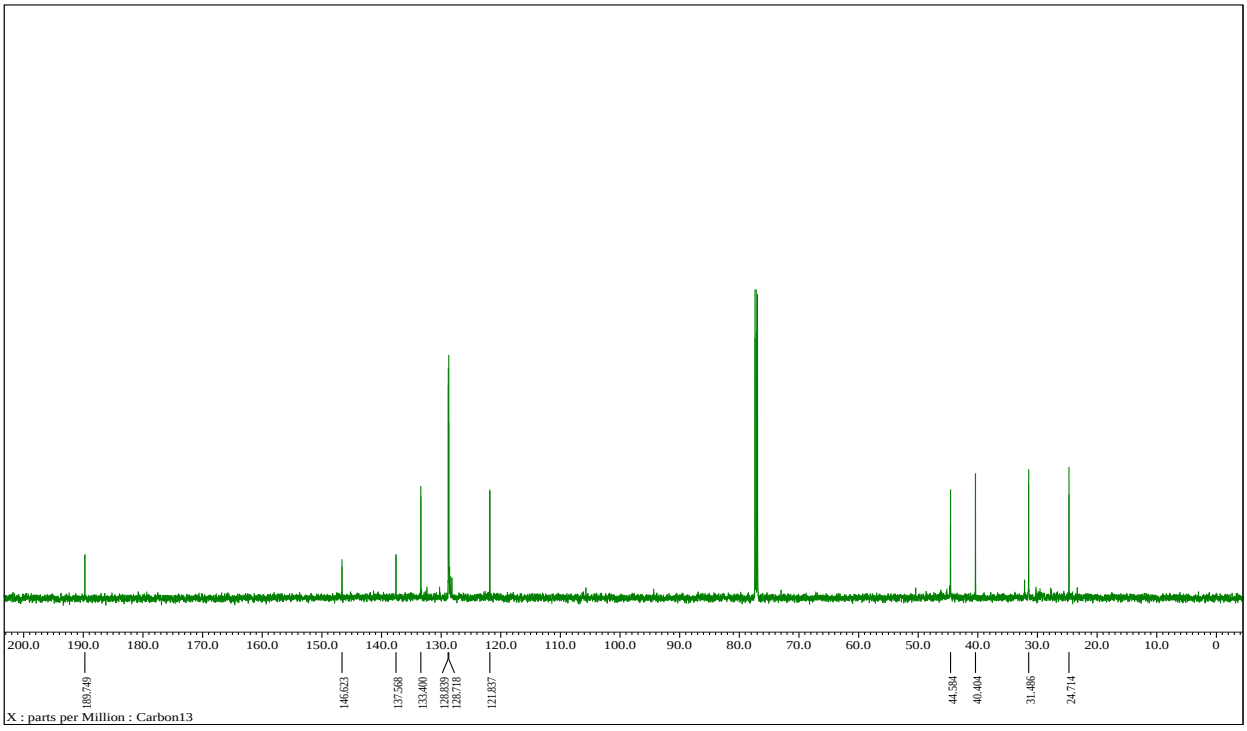
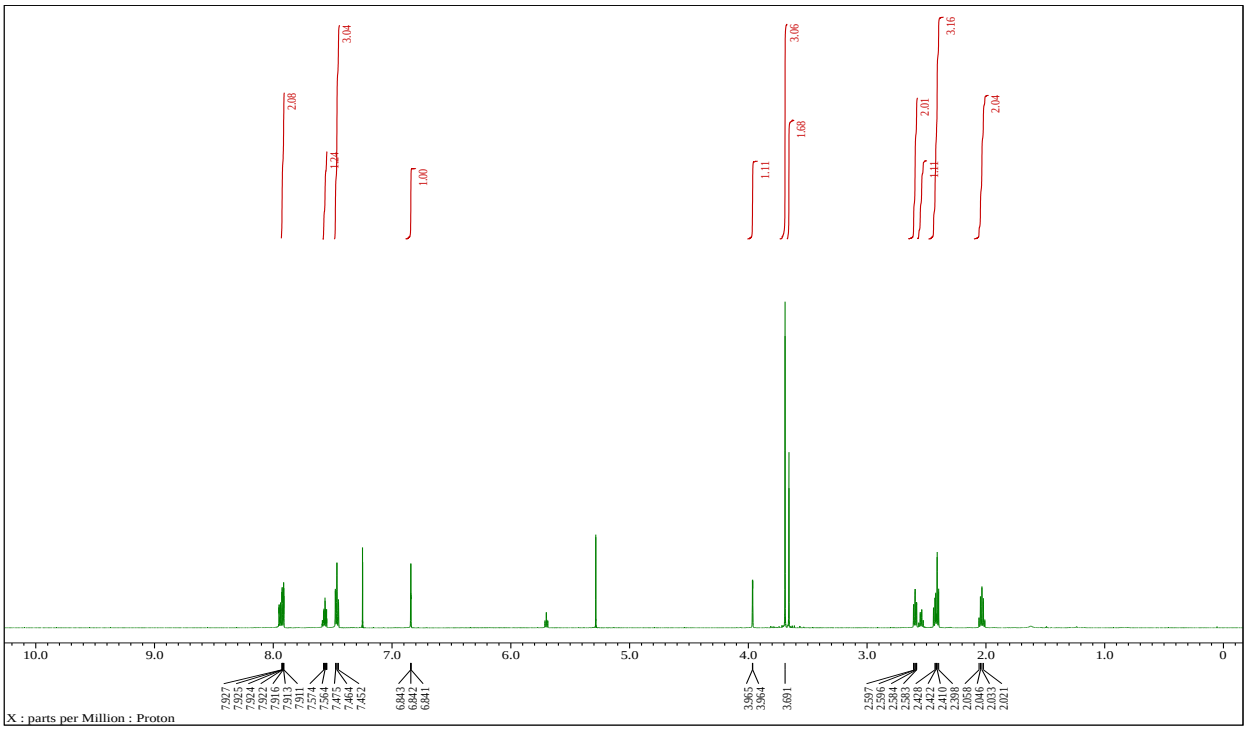
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
189.878
147.827
137.770
133.208
128.697
128.681
121.246
41.282
31.971
29.648
29.586
29.384
29.307
28.752
27.415
22.761
14.195
X : parts per Million : Carbon13

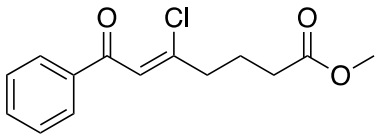

2.06
2.05
1.00
1.00
2.05
2.00
4.01
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.935
7.932
7.924
7.921
7.919
7.586
7.577
7.573
7.562
7.487
7.473
7.464
6.849
3.599
3.593
3.589
3.584
3.578
2.594
2.587
2.584
2.578
2.572
1.879
1.876
1.873
1.867
1.862
1.857
X : parts per Million : Proton


O
Cl
Cl

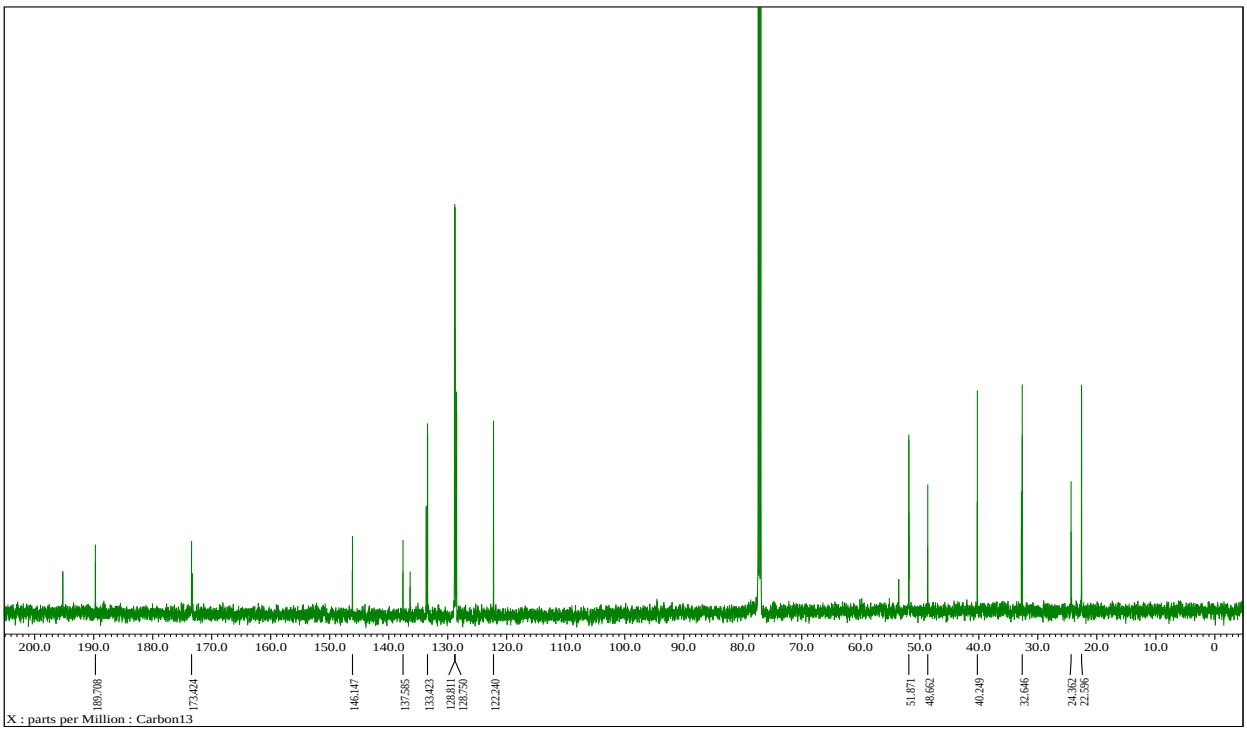
(Z)-3s

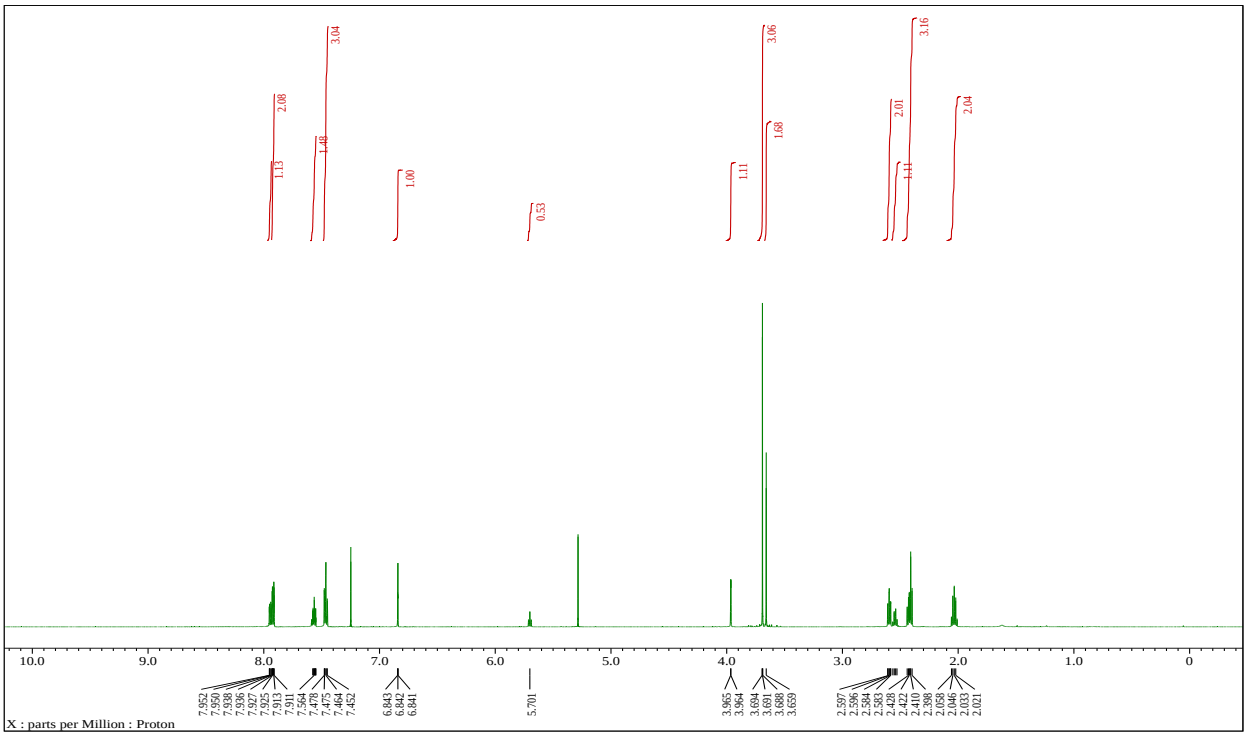

200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
189.749
146.623
137.568
133.400
128.839
128.718
121.837
44.584
40.404
31.486
24.714
X : parts per Million : Carbon13

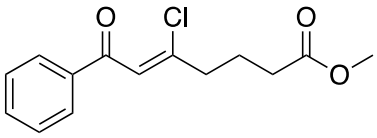

2.08
3.04
1.24
1.00
1.11
3.06
1.68
2.01
1.11
3.16
2.04
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.927
7.925
7.924
7.922
7.916
7.913
7.911
7.574
7.564
7.475
7.464
7.452
6.843
6.842
6.841
3.965
3.964
3.691
2.597
2.596
2.584
2.583
2.428
2.412
2.410
2.398
2.058
2.046
2.033
2.021
X : parts per Million : Proton


O
Cl
O
O

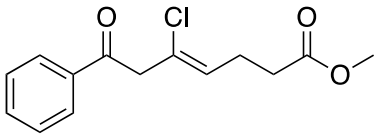
(Z)-3t


200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
189.708
173.424
146.147
137.585
133.423
128.811
128.750
122.240
51.871
48.662
40.249
32.646
24.362
22.396
X : parts per Million : Carbon13

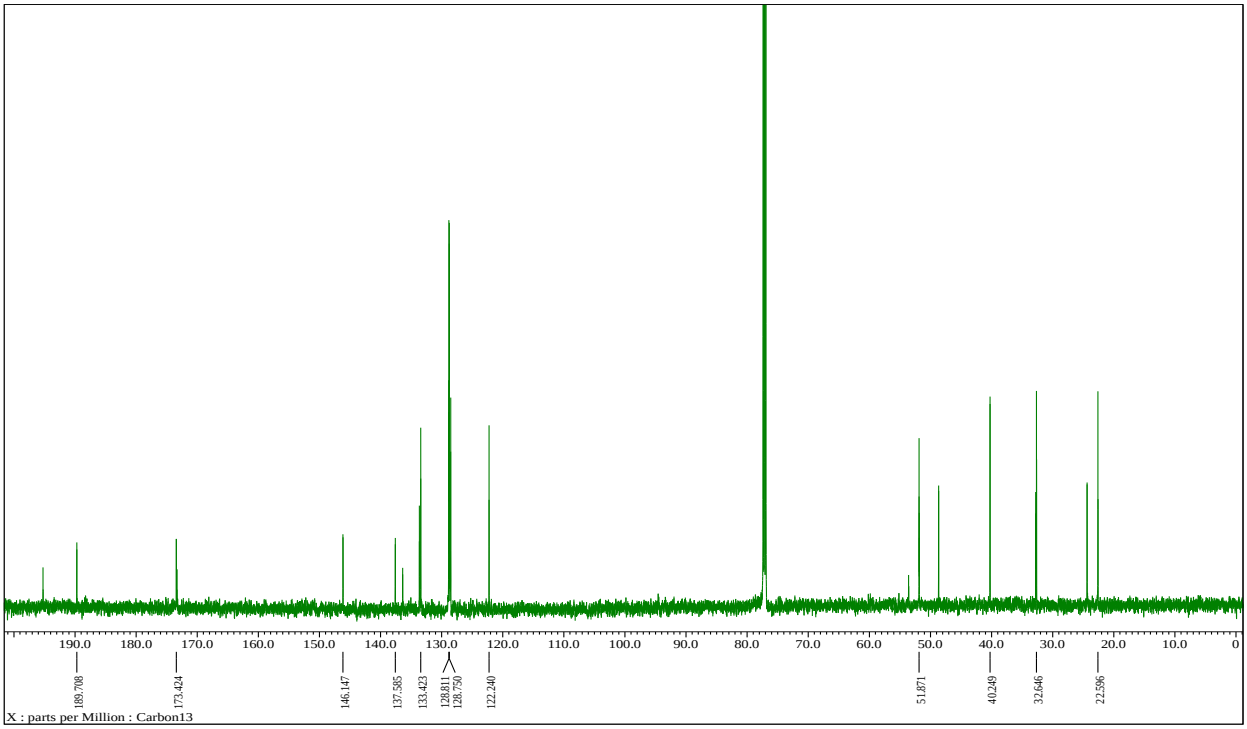

3.04
2.08
1.13
1.48
1.00
0.53
3.06
1.11
1.68
3.16
2.01
1.11
2.04
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.953
7.950
7.938
7.936
7.927
7.925
7.913
7.911
7.564
7.478
7.475
7.464
7.452
6.843
6.842
6.841
5.701
3.965
3.964
3.694
3.691
3.688
3.639
2.597
2.596
2.584
2.583
2.428
2.422
2.410
2.398
2.058
2.046
2.033
2.021
X : parts per Million : Proton

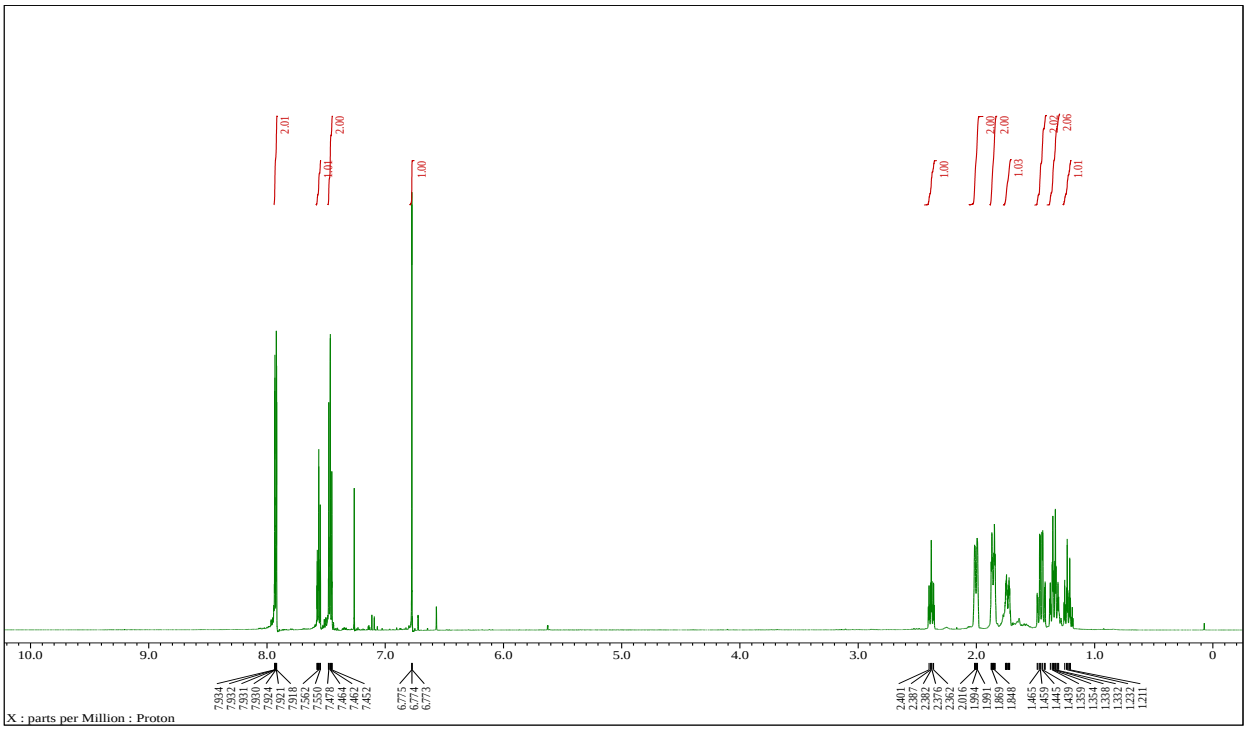

O
Cl
O
O

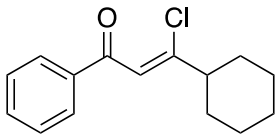
(*Z*)-3t (66%)


O
Cl
O
O

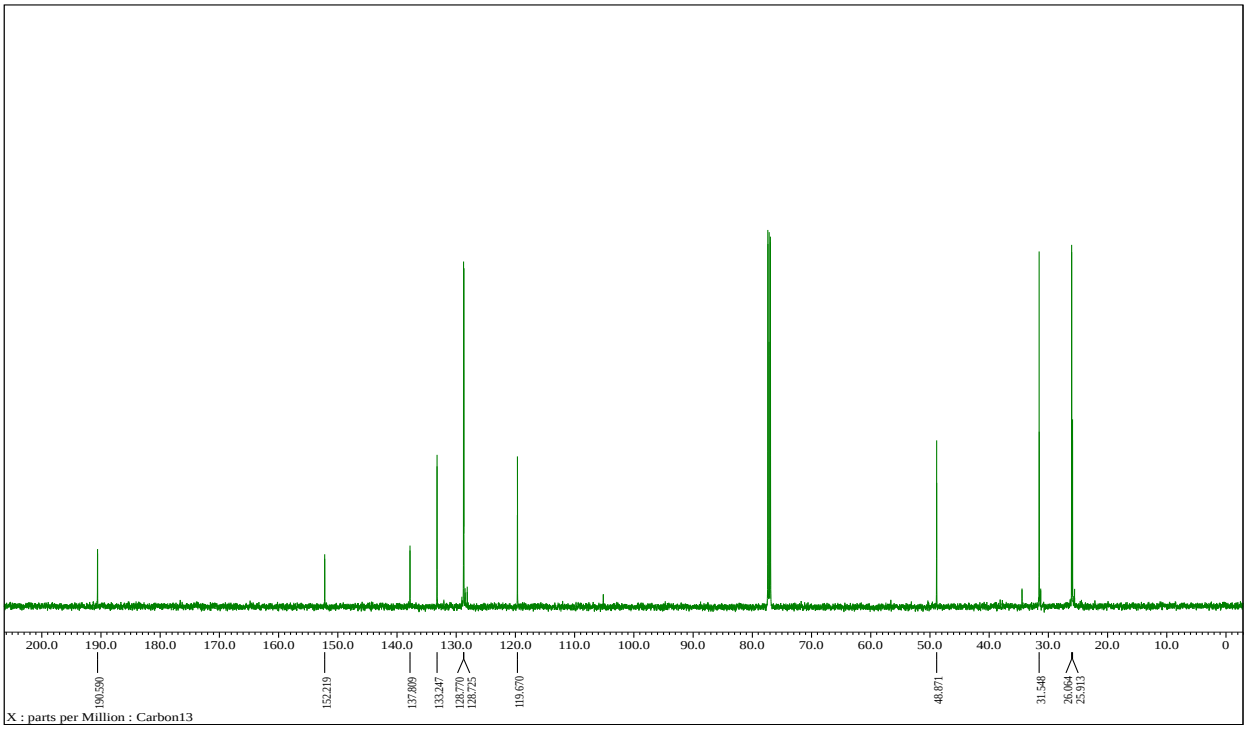
4t (34%)


190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
189.708
173.424
146.147
137.585
133.423
128.811
128.750
122.240
51.871
40.249
32.646
22.596
X : parts per Million : Carbon13


2.01
2.00
1.01
1.00
1.00
2.02
2.00
2.00
2.02
2.06
1.00
1.03
1.01
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.934
7.932
7.931
7.930
7.924
7.921
7.918
7.562
7.550
7.478
7.464
7.462
7.452
6.775
6.774
6.773
2.401
2.387
2.382
2.376
2.362
2.016
1.994
1.991
1.969
1.848
1.465
1.459
1.445
1.439
1.359
1.354
1.338
1.332
1.232
1.211
X : parts per Million : Proton


O
Cl

(*Z*)-3u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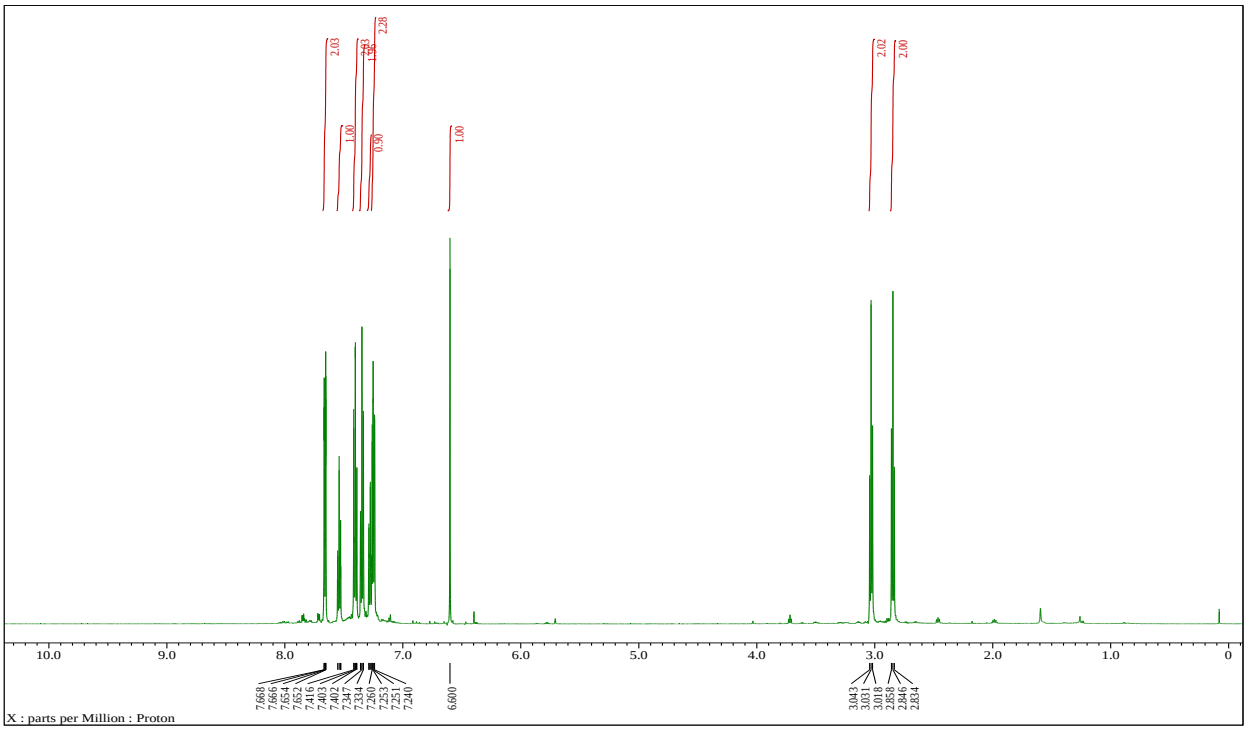
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
190.590
152.219
137.809
133.247
128.770
128.725
119.670
48.871
31.548
26.064
25.913
X : parts per Million : Carbon13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0.90
2.28
1.00
2.02
2.00
10.0
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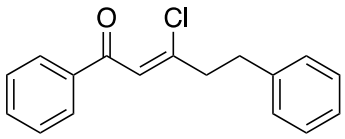
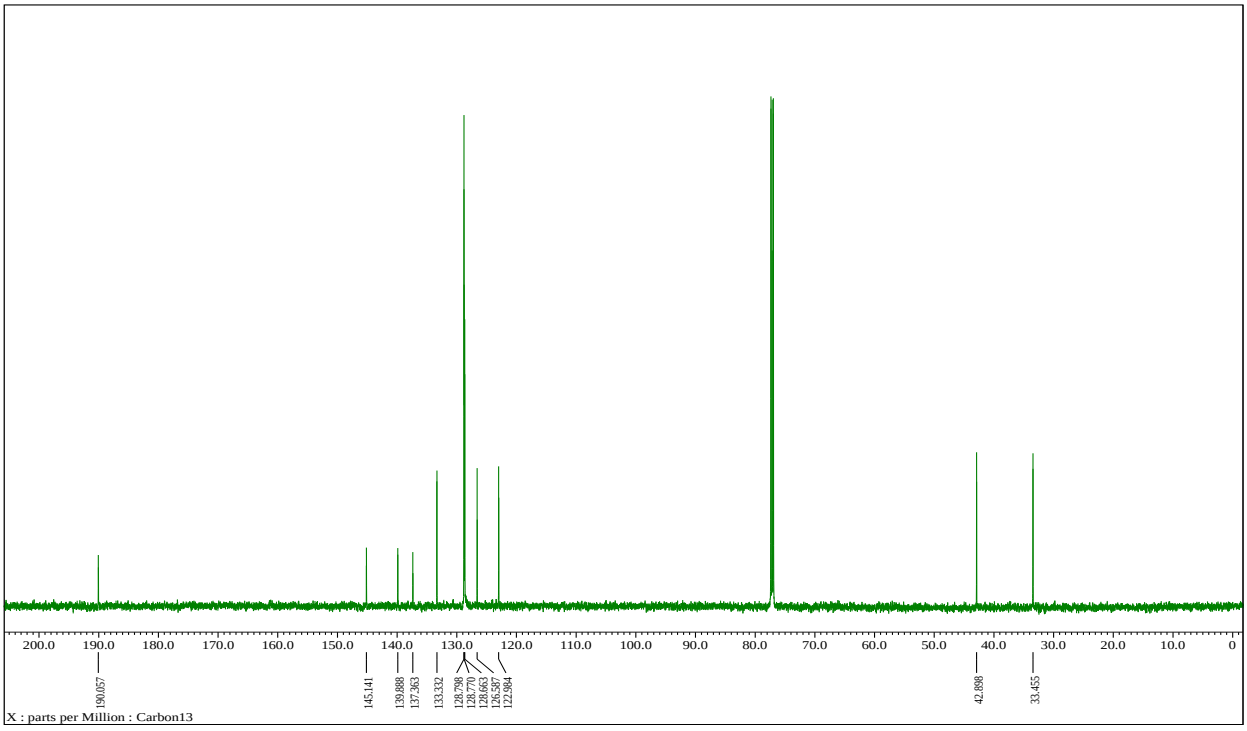
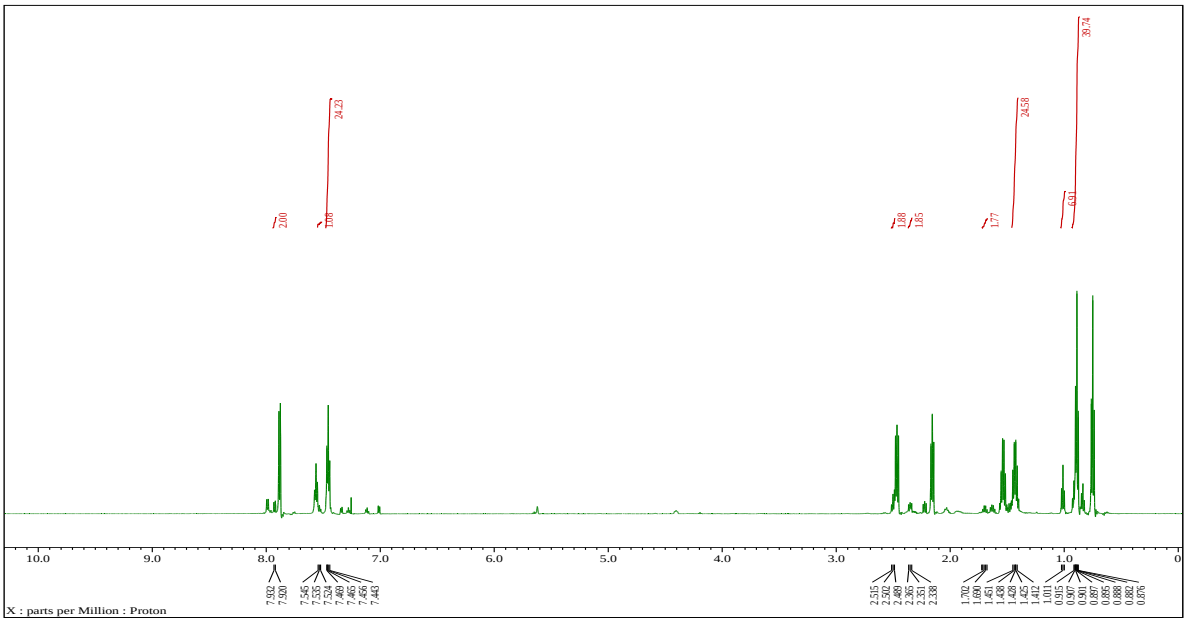
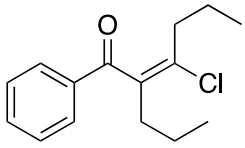
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.668
7.666
7.654
7.652
7.416
7.403
7.402
7.347
7.334
7.260
7.253
7.251
7.240
6.600
3.043
3.031
3.018
2.858
2.846
2.834
X : parts per Million : Proton


O
Cl

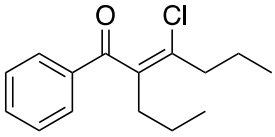
(*Z*)-3v


200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
190.057
145.141
139.888
137.363
133.332
128.798
128.770
128.663
116.357
122.984
42.898
33.455
X : parts per Million : Carbon13

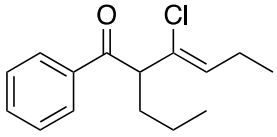




(*E*)-3w (69%)



(Z)-3w (6%)



4w (6%)