Supporting Information

Regiospecific Synthesis of Tetra-Substituted Furans

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EXPERIMENTAL

IR spectra were run on a Nicolet Impact 410 infrared spectrophotometer or Nicolet 380 FT-IR spectrophotometer. NMR spectra were recorded on a Bruker Spectrospin DPX-400 MHz spectrometer. Chemical shifts are reported in ppm downfield from TMS. TLC analyses of the reaction mixtures were performed with Silica gel (60 F_{254}) on aluminum sheets with mixtures of hexanes (a commercial mixture of isomeric hexanes) and ethyl acetate as the mobile phase. Flash chromatography was carried out with Silica gel (230-400 mesh) as the stationary phase and mixtures of ethyl acetate and hexanes (a commercial mixture of isomeric hexanes) as the mobile phase. The eluent composition is given in each case. Mass spectra were obtained on a JEOL AccuTOF T100GC spectrometer. The instruments were operated in the DART/ESI+ mode at 10-15 eV.

Synthesis of Propargylic Alcohols 2

By bromomagnesium acetylide: To TEB (**1**) (5.00 g, 22.0 mmol) in anhydrous THF (150 mL) under nitrogen a THF solution of ethylmagnesium bromide (7.3 mL, 3.0 M, 22

mmol) was added dropwise over 10 min at rt. The mixture was refluxed for 2 h and cooled to 0 °C before aldehyde (22.0 mmol) in anhydrous THF (25 mL) was added dropwise. Stirring was continues at 20 °C until all the starting material was consumed (monitored by TLC).

By lithium acetylide: TEB (**1**) (5.00 g, 22.0 mmol) was dissolved in anhydrous THF (100 mL) under nitrogen and cooled to -78 °C before a 1.6 M hexane solution of *n*-butyllithium (15 mL, 22.0 mmol) was added dropwise. The mixture was stirred at -78 °C for 30 min and then slowly heated to 0 °C before aldehyde (22.0 mmol) in THF was added dropwise. The mixture was stirred at rt until all the starting material was consumed (monitored by TLC).

Work-up for both procedures: The reaction mixture was cooled to 0 °C and quenched with saturated aq NH_4Cl (100 mL). The phases were separated and the aq phase was extracted (Et_2O; 3 x 50 mL). The combined organic extracts were dried ($MgSO_4$) and concentrated. From the residue **2** was obtained pure by flash chromatography (hexanes and ethyl acetate, 80:20).

Compounds **2a**-**2c**, **2e** and **2f** are described in the literature[9,11] whereas 1,1,2,2-tetraethoxydodec-3-yn-5-ol (**2d**) and 4,4,5,5-tetraethoxy-1-(*p*-methylphenyl)pent-2-yn-1-ol (**2g**), isolated as yellowish liquids by flash chromatography, are new compounds.

2d: 6.38 g (82%). IR (film): 3387 (m), 2970 (s), 2955 (s), 2925 (s), 2855 (s), 2241 (w), 1739 (w), 1638 (w), 1456 (m), 1444 (m), 1371 (m), 1351 (m), 1332 (m), 1295 (w), 1230 (m), 1117 (s), 1070 (s), 1019 (s), 876 (w), 802 (w), 723 (w) cm^{-1}; ^1H NMR (400 MHz, $CDCI_3$): δ 4.41 (t, *J* = 6.5 Hz, 1H), 4.38 (s, 1H), 3.81-3.68 (m, 8H), 2.41 (bs, 1H), 1.75-1.19 (m, 24H), 0.87 (t, *J* = 6.5 Hz, 3H); ^{13}C NMR (100 MHz, $CDCI_3$): δ 104.6, 99.4, 88.9, 79.9, 65.5, 62.9, 60.3, 38.1, 32.4, 29.9, 26.4, 25.7, 23.3, 16.0, 15.9, 14.7; HRMS Calcd for $C_{20}H_{37}0_4^+$ $[M - OH]^+$ 341.26918, found 341.27273.

2g: 5.70 g (75%). IR (film): 3411 (m), 2975 (s), 2929 (s), 2889 (s), 1739 (w), 1702 (w), 1650 (w), 1605 (w), 1513 (w), 1479 (m), 1443 (m), 1388 (m), 1372 (m), 1328 (m), 1243 (m), 1176 (m), 1111 (s), 1067 (s), 1017 (m), 873 (w), 818 (w), 758 (w), 560 (m), 538 (m) cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.46 (d, *J* = 7.5 Hz, 2H), 7.16 (d, *J* = 7.8 Hz, 3H), 5.50 (d, *J* = 5.7 Hz 1H), 4.41 (s, 1H), 3.80-3.67 (m, 8H), 2.82 (d, *J* = 6.0 Hz, 1H), 2.37 (s, 3H), 1.25-1.18 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.7, 138.1, 129.8, 127.5, 104.7, 99.5, 87.5, 81.8, 65.4, 64.9, 60.5, 21.8, 16.0, 15.9; HRMS Calcd for $C_{20}H_{29}0_4^+$ $[M - OH]^+$ 333.20649, found 333.20658.

Synthesis of 3 by Deketalization of 2

Propargylic alcohol **2** (15 – 5.5 mmol) was dissolved in 100 mL of a 7:3 mixture of THF and H_2O. *p*-Toluenesulfonic acid monohydrate (0.57 g, 3.0 mmol) was added and the mixture was refluxed for 2 h until all the starting material was consumed (followed by TLC). Most of the THF was evaporated, and the residue was mixed with a saturated aq solution of NaCl (50 mL) and CH_2Cl_2 (50 mL). After thorough stirring the phases were separated, the aq phase was extracted (CH_2Cl_2; 3 x 20 mL), and the combined organic phases were washed with a saturated aq solution of $NaHCO_3$ (50 mL) and dried ($MgSO_4$). Filtration and evaporation gave the corresponding ketone essentially pure (^1H NMR).

Compounds **3a**-**3c**, **3e** and **3f** are described in the literature[9,11] whereas 1,1-diethoxy-5-hydroxydodec-3-yn-2-one (**3d**) and 1,1-diethoxy-5-hydroxy-5-(4-methylphenyl)pent-3-yn-2-one (**3g**), isolated as yellow liquids by flash chromatography, are new compounds.

3d: 4.05 g (95%). IR (film): 3450 (m), 2954 (m), 2924 (s), 2855 (s), 2210 (s), 1687 (s), 1456 (m), 1376 (m), 1318 (m), 1231 (m), 1217 (m), 1163 (m), 1108 (s), 1059 (s), 908 (w), 890 (w), 8375 (w), 723 (w) cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 4.74 (s, 1H), 4.55 (t, *J* = 6.7

Hz, 1H), 3.78-3.58 (m, 4H), 3.32 (bs, 1H), 1.84-1.20 (m, 18H), 0.89 (t**,** *J* = 6.7 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 183.3, 102.2, 97.3, 82.5, 63.8, 63.0, 37.5, 32.4, 30.3, 29.8, 25.6, 23.3, 23.3, 15.7, 14.7; HRMS Calcd for $C_{16}H_{29}O_4^+$ $[M + H]^+$ 285.20658, found 285.20565.

3g: 3.81 g (92%). IR (film): 3370 (m), 2978 (s), 2930 (s), 2874 (s), 2207 (s), 1686 (s), 1605 (w), 1512 (w), 1479 (m), 1444 (m), 1392 (m), 1372 (w), 1319 (m), 1244 (s), 1169 (m), 1106 (s), 1055 (s), 907 (m), 838 (m), 818 (m) 758 (m), 735 (m), 702 (w), 649 (m), 634 (m) cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 8.0 Hz, 2H), 5.58 (d, *J* = 5.8 Hz, 1H), 4.75 (s, 1H), 3.74-3.58 (m, 4H), 3.53 (bs, 1H), 2.35 (s, 3H), 1.25 (t, *J* = 7.0 Hz, 6H); ^{13}C NMR (100 MHz, $CDCI_3$) □ 183.3, 139.3, 136.5, 130.2, 127.4, 102.1, 95.8, 83.6, 64.8, 63.8, 21.8, 15.7; HRMS Calcd for $C_{16}H_{I9}O_3^+$ $[M - OH]^+$ 259.13342, found 259.13463.

Furan Synthesis; Reaction of Ethyl Acetoacetate with Conjugated γ-Hydroxyalkynones (3); Ethyl 4-(3,3-Diethoxy-2-oxopropyl)-2-methylfuran-3-carboxylate (4a)

Ethyl acetoacetate (0.13 g, 1.0 mmol) was added to sodium ethoxide (0.034 g, 0.5 mmol) in ethanol (10 mL). The reaction mixture was stirred at rt for 30 minutes. Then 1,1-diethoxy-5-hydroxypent-3-yn-2-one (**3a**) (0.18 g, 1.0 mmol) in ethanol (5 mL) was added dropwise and the mixture was stirred at 80 °C for additional 3 h before the mixture was allowed to cool down to rt. Water (30 mL) and CH_2Cl_2 (30 mL) were added and the phases separated. The aq phase was extracted (CH_2Cl_2; 3 x 30 mL) and the combined organic phases were dried ($MgSO_4$), filtered and concentrated. Flash chromatography (hexane/ethyl acetate; 90:10) gave **4a** (0.24 g, 80%; colorless liquid). IR (film): 2978 (s), 2932 (s), 2900 (s), 1738 (s), 1709 (s), 1611 (w), 1567 (m), 1429 (m), 1388 (m), 1366 (w), 1297 (s), 1273 (s), 1218 (m), 1205 (m), 1153 (m), 1093 (s), 1057 (s), 939 (w), 836 (w), 780 (m), 609 (w) cm^{-1}; ^{1}H NMR

($CDCl_3$, 400 MHz): δ 7.20 (s, 1H), 4.76 (s, 1H), 4.23 (q, *J* = 7.2 Hz, 2H), 3.92 (s, 2H), 3.76-3.60 (m, 4H), 2.55 (s, 3H), 1.32-1.25 (m, 9H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 203.2, 164.7, 160.6, 140.0, 118.5, 113.6, 102.9, 63.8, 60.5, 34.0, 15.8, 15.0, 14.8; HRMS Calcd for $C_{15}H_{23}O_6^+$ $[M + H]^+$ 299.14946, found 299.14861.

Ethyl 4-(3,3-Diethoxy-2-oxopropyl)-2,5-dimethylfuran-3-carboxylate (4b)

1,1-Diethoxy-5-hydroxyhex-3-yn-2-one (**3b**) (1.0 g, 5.0 mmol) was reacted with ethyl acetoacetate (0.65 g, 5.0 mmol) for 3 h at 80 °C. Flash chromatography (hexanes/ethyl acetate; 80:20) provided **4b** (1.1 g, 74%; colorless liquid). IR (film): 2977 (s), 2927 (s), 2877 (s), 1738 (s), 1707 (s), 1643 (w), 1588 (s), 1432 (m), 1381 (m), 1335 (m), 1315 (w), 1288 (s), 1256 (w), 1228 (s), 1170 (m), 1115 (m), 1079 (s), 1058 (s), 958 (m), 947 (m), 843 (w), 833 (m), 781 (m), 652 (m), 637 (m), 628 (m), 602 (w) cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ 4.75 (s, 1H), 4.22 (q, *J* = 7.1 Hz, 2H), 3.82 (s, 2H), 3.75-3.58 (m, 4H), 2.51 (s, 3H), 2,14 (s, 3H), 1.31-1.25 (m, 9H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 202.8, 164.4, 157.6, 147.9, 113.3, 111.9, 102.3, 63.3, 59.8, 33.5, 15.2, 14.3, 11.2. HRMS Calcd for $C_{16}H_{25}O_6^+$ $[M + H]^+$ 313.16511, found 313.16363.

Ethyl 4-(3,3-Diethoxy-2-oxopropyl)-5-isopropyl-2-methylfuran-3-carboxylate (4c)

1,1-Diethoxy-5-hydroxy-6-methylhept-3-yn-2-one (**3c**) (0.23 g, 1.0 mmol) was reacted with ethyl acetoacetate (0.13 g, 1.0 mmol) for 3 h at 80 °C. Flash chromatography (hexanes/ethyl acetate; 90:10) provided **4c** (0.30 g, 90%; colorless liquid). IR (film): 2976 (s), 2933 (s), 2875 (s), 1739 (s), 1712 (s), 1640 (w), 1587 (m), 1434 (m), 1384 (m), 1289 (s), 1234 (m), 1173 (m), 1102 (s), 1075 (s), 1058 (s), 976 (w), 943 (m), 847 (w), 829 (m), 781 (w), 651 (m), 632 (m), 619 (m), 599 (w) cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ 4.75 (s, 1H), 4.20 (q, *J* =

7.2 Hz, 2H), 3.87 (s, 2H), 3.76-3.60 (m, 4H), 2.84 (septet, J = 7.0 Hz, 1H), 2.51 (s, 3H), 1.27-1.22 (m, 9H); 1.19 (d, J = 7.0 Hz, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 203.0, 164.8, 157.6, 156.3, 113.4, 110.1, 102.6, 63.5, 59.9, 33.2, 26.0, 21.5, 15.4, 14.5; HRMS Calcd for $C_{18}H_{28}O_6^+$ $[M]^+$ 341.19641, found 341.19644.

Ethyl 4-(3,3-Diethoxy-2-oxopropyl)-2-methyl-5-hexylfuran-3-carboxylate (4d)

1,1-Diethoxy-5-hydroxyundec-3-yn-2-one (**3d**) (1.0 g, 3.7 mmol) was reacted with ethyl acetoacetate (0.48 g, 3.7 mmol) for 3 h at 80 °C. Flash chromatography (hexanes/ethyl acetate; 80:20) provided **4d** (1.19 g, 84%; colorless liquid). IR (film): 2976 (m), 2955 (m), 2927 (s), 2872 (m), 2858 (m), 1739 (s), 1709 (s), 1649 (w), 1606 (w), 1587 (s), 1457 (m), 1433 (m), 1394 (m), 1377 (m), 1334 (m), 1314 (w), 1285 (s), 1247 (m), 1231 (m), 1170 (m), 1123 (s), 1083 (s), 1058 (s), 947 (m), 906 (m), 858 (m), 810 (w), 781 (w), 726 (w), 636 (m) cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.75 (s, 1H), 4.20 (q, J = 7.2 Hz, 2H), 3.83 (s, 2H), 3.76-3.60 (m, 4H), 2.51 (s, 3H), 2.45 (t, J = 7.6 Hz, 2H), 1.57-1.25 (m, 17H), 0.87 (t, J = 5.6 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 203.4, 165.2, 158.3, 152.6, 113.9, 112.3, 103.0, 64.0, 60.4, 33.9, 32.2, 29.4, 29.1, 26.3, 23.2, 15.8, 15.0, 14.9, 14.7; HRMS Calcd for $C_{21}H_{35}O_6^+$ $[M + H]^+$ 383.24336, found 383.24180.

Ethyl 4-(3,3-Diethoxy-2-oxopropyl)-2-methyl-5-heptylfuran-3-carboxylate (4e)

1,1-Diethoxy-5-hydroxydodec-3-yn-2-one (**3e**) (1.0 g, 3.5 mmol) was reacted with ethyl acetoacetate (0.46 g, 3.5 mmol) for 3 h at 80 ^{o}C. Flash chromatography (hexanes/ethyl acetate; 80:20) provided **4e** (0.97 g, 70%; colorless liquid). IR (film): 2970 (m), 2954 (m), 2926 (s), 2872 (m), 2856 (m), 1739 (s), 1710 (s), 1649 (w), 1587 (s), 1456 (m), 1433 (m), 1367 (m), 1336 (w), 1314 (m), 1282 (s), 1227 (m), 1218 (m), 1160 (m), 1123 (s), 1083 (s),

1058 (s), 972 (m), 949 (w), 904 (w), 849 (m), 781 (w), 742 (w), 636 (m) cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ 4.75 (s, 1H), 4.20 (q, *J* = 7.0 Hz, 2H), 3.83 (s, 2H), 3.76-3.60 (m, 4H), 2.51 (s, 3H), 2.45 (t, *J* = 7.5 Hz, 2H), 1.57-1.25 (m, 19H), 0.87 (t, *J* = 5.3 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 203.4, 165.2, 158.3, 152.6, 113.9, 112.3, 103.0, 64.0, 60.4, 33.9, 32.4, 29.7, 29.6, 29.1, 26.3, 23.3, 15.8, 15.0, 14.8, 14.7; HRMS Calcd for $C_{22}H_{37}O_6^+$ $[M + H]^+$ 397.25901, found 397.25747.

Ethyl 4-(3,3-Diethoxy-2-oxopropyl)-2-methyl-5-phenylfuran-3-carboxylate (4f)

1,1-Diethoxy-5-hydroxy-5-phenylpent-3-yn-2-one(**3f**) (1 g, 3.8 mmol) was reacted with ethyl acetoacetate (0.50 g, 3.8 mmol) for 5 h at 80 ^oC. Flash chromatography (hexanes/ethyl acetate; 80:20) provided **4f** (1.21 g, 85%; colorless liquid). IR (film): 2978 (s), 2931 (m), 2898 (m), 1736 (s), 1708 (s), 1620 (w), 1603(m), 1583 (w), 1570 (w), 1494 (w), 1479 (m), 1445 (m), 1430 (w), 1400 (m), 1366 (w), 1318 (s), 1302 (m), 1247 (s), 1176 (m), 1152 (m), 1127 (m), 1092 (s), 1058 (s), 1013 (m), 980 (s), 949 (m), 912 (w), 841 (w), 765 (s), 734 (w), 696 (s), 670 (s), 617 (m), 572 (w) cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ 7.49-7.48 (m, 2H), 7.39-7.36 (m, 2H), 7.32-7.28 (m, 1H), 4.81 (s, 1H), 4.25 (q, *J* = 6.9 Hz, 2H), 4.16 (s, 2H), 3.79-3.63 (m, 4H), 2.62 (s, 3H), 1.34-1.26 (m, 9H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 203.5, 164.9, 159.4, 150.4, 130.8, 129.2, 128.5, 127.1, 115.5, 114.3, 103.3, 64.2, 60.6, 34.9, 15.8, 15.2, 14.9; HRMS Calcd for $C_{19}H_{21}O_6^+$ $[M - OEt]^+$ 329.13890, found 329.14008.

Ethyl 4-(3,3-Diethoxy-2-oxopropyl)-2-methyl-5-(*p*-methyl)phenylfuran-3-carboxylate (4g)

1,1-Diethoxy-5-hydroxy-5-(*p*-methyl) phenylpent-3-yn-2-one (**3g**) (1 g, 3.6 mmol) was reacted with ethyl acetoacetate (0.47 g, 3.6 mmol) for 3 h at 80 ^oC. Flash chromatography (hexanes/ethyl acetate; 80:20) provided **4g** (1.23 g, 88%; colorless liquid). IR (film): 2977 (s),

2929 (m), 2898 (m), 1737 (s), 1707 (s), 1610 (w), 1583(m), 1509 (s), 1431 (m), 1394 (m), 1366 (w), 1318 (m), 1301 (w), 1247 (s), 1176 (m), 1153 (m), 1118 (m), 1091 (s), 1058 (s), 1018 (m), 979 (s), 948 (m), 935 (w), 905 (w), 819 (s), 779 (w), 723 (w), 673 (w), 644 (m), 625 (m), 594 (w) cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ 7.36 (d, *J* = 8.1 Hz, 2H), 7.18 (d, *J* = 8.0 Hz 2H), 4.80 (s, 1H), 4.25 (q, *J* = 7.3 Hz, 2H), 4.13 (s, 2H), 3.79-3.62 (m, 4H), 2.61 (s, 3H), 2.36 (s, 3H), 1.33-1.26 (m, 9H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 203.6, 165.0, 159.2, 150.6, 138.5, 129.9, 128.1, 127.1, 115.4, 113.7, 103.3, 64.1, 60.6, 35.0, 21.9, 15.9, 15.2, 14.9. HRMS Calcd for $C_{20}H_{23}0_5^+$ $[M - OEt]^+$ 343.15455, found 343.15452.

Reaction with 2-Methylacetoacetate; Formation of 4,4-Diethoxy-2-diethoxymethyl-2-hydroxytetrahydrofuran (5) and 1,1-Diethoxy-3-(2-(diethoxymethyl)-2-(3-hydroxyprop-1-ynyl)-1,3-dioxolan-4-ylid-ene)prop-an-2-one (6)

1,1-Diethoxy-5-hydroxy-6-methylhept-3-yn-2-one (**3a**) (0.50 g, 2.7 mmol) reacted with ethyl 2-methylacetoacetate (0.39 g, 2.7 mmol) for 3 h at 80 °C. Flash chromatography (hexanes/ethyl acetate; 80:20) provided **5** (0.45 g, 60%; yellowish) and **6** (0.15 g, 15%; yellow) as liquids. Data for **5**: IR (film): 3469 (m), 2975 (s), 2931 (w), 2883 (m), 1727 (m), 1608 (m), 1481 (m), 1444 (w), 1390 (m), 1368 (w), 1322 (m), 1285 (m), 1238 (m), 1158 (m), 1106 (m), 1052 (s), 976 (m), 908 (m), 870 (m), 820 (m), 790 (m), 765 (w). cm^{-1} ^1H NMR (400 MHz, $CDCl_3$): δ 4.39 (s, 1H), 4.07 (d, *J* = 9.1 Hz, 1H), 3.81-3.47 (m, 9H), 2.39 (d, *J* = 13.2 Hz, 1H), 2.16 (d, *J* = 13.4 Hz, 1H), 1.27-1.19 (m, 15H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 108.8, 106.3, 104.1, 72.7, 65.5, 65.2, 59.0, 58.6, 42.5, 16.0; HRMS Calcd for $C_{11}H_{19}0_4^+$ $[M – OEt – H_2O]^+$ 215.12833, found 215.12734. Data for **6**: IR (film): 3450 (s), 2977 (s), 2932 (s), 2882 (m), 2252 (m), 1736 (s), 1689 (s), 1605 (s), 1480 (m), 1444 (w), 1372 (s), 1322 (m), 1270 (m), 1237 (s), 1158 (m), 1097 (s), 1069 (s), 1039 (s), 969 (m), 900 (s), 864 (s), 831 (w),

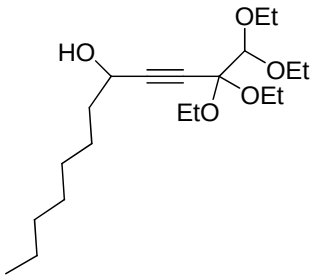
759 (w), 763 (m), 633 (w), 606 (m) cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.26 (m, 1H, X part of ABX system), 5.18 (dd, 1H, *J* = 16.1 and 1.66 Hz, A part of ABX system), 5.06 (dd, 1H, *J* = 16.1 and 1.74 Hz, B part of ABX system), 4.65 (s, 1H), 4.51 (s, 1H), 4.34 (s, 2H), 3.85-3.55 (m, 8H), 1.98 (bs, 1H), 1 28-1.23 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.9, 171.0, 106.4, 103.7, 102.7, 94.5, 86.8, 79.2, 72.3, 66.3, 63.5, 61.1, 51.5, 15.8, 14.8. HRMS Calcd for $C_{18}H_{29}0_8^+$ $[M + H]^+$ 373.18624, found 373.18581.

Reaction with Diethyl Malonate

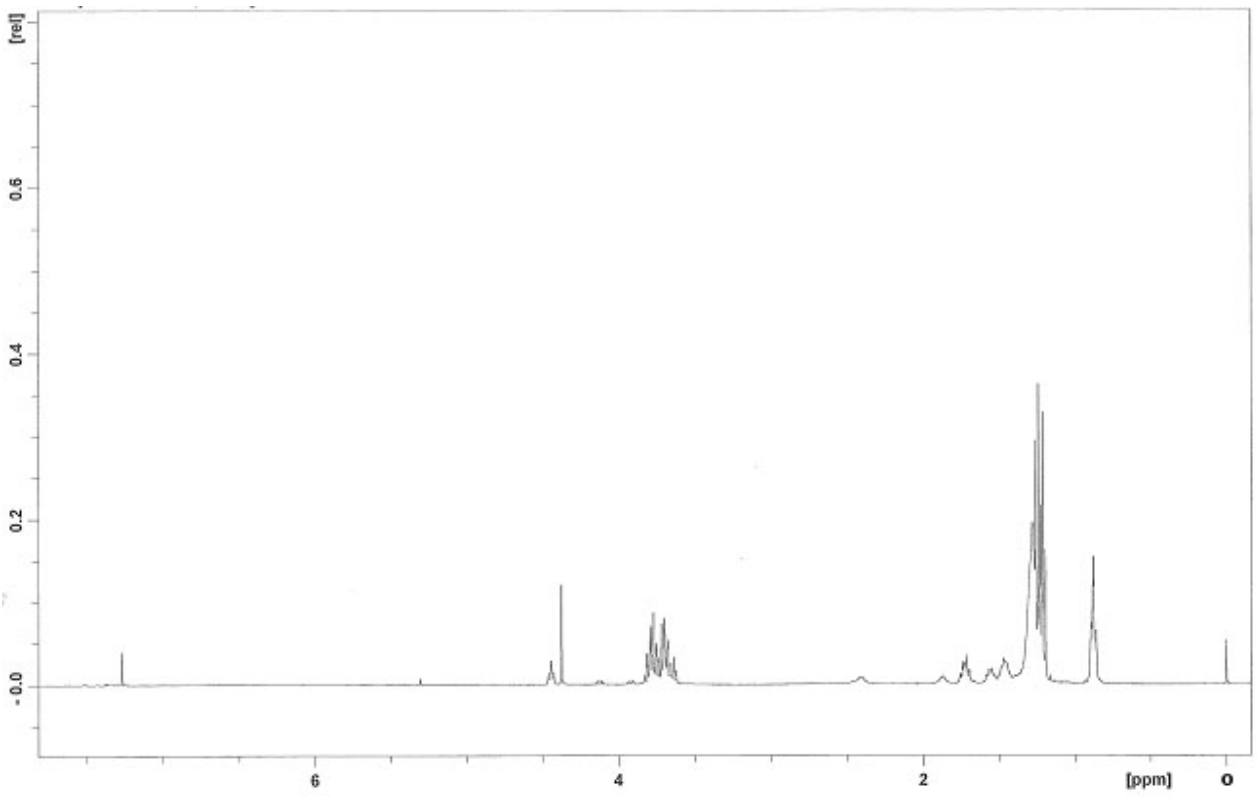
Diethoxy-5-hydroxy-6-methylhept-3-yn-2-one (**3a**) (0.50 g, 2.7 mmol) reacted with diethyl malonate (0.43 g, 2.7 mmol) for 3 h at 80 ^{o}C. Flash chromatography (hexanes/ethyl acetate; 80:20) provided **6** (0.50 g, 50%; yellowish liquid).

NMR spectra

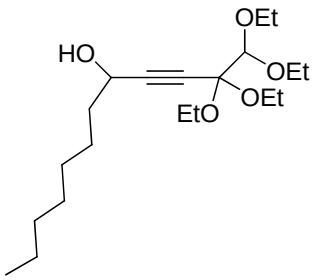
^{1}H NMR spectrum



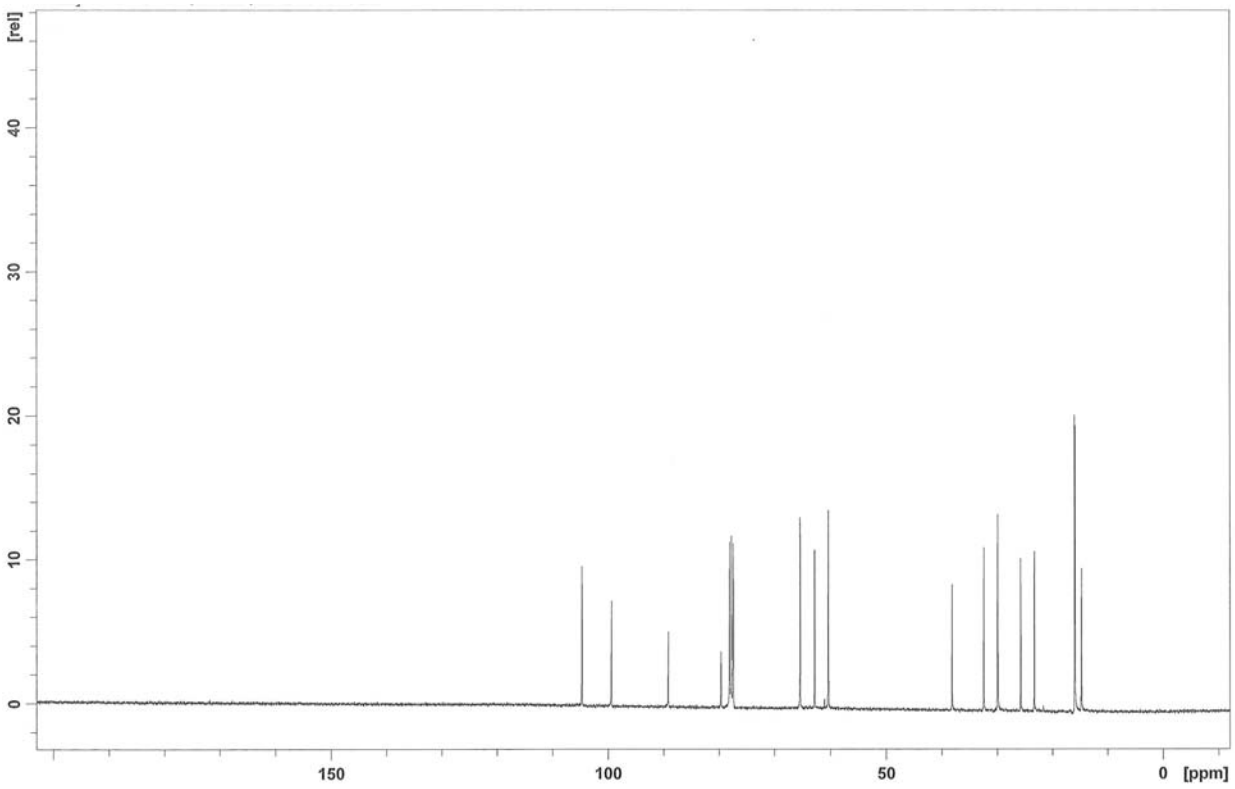
2d



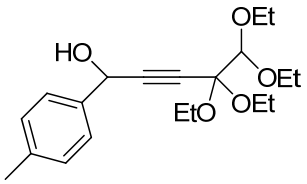
^{13}C NMR spectrum



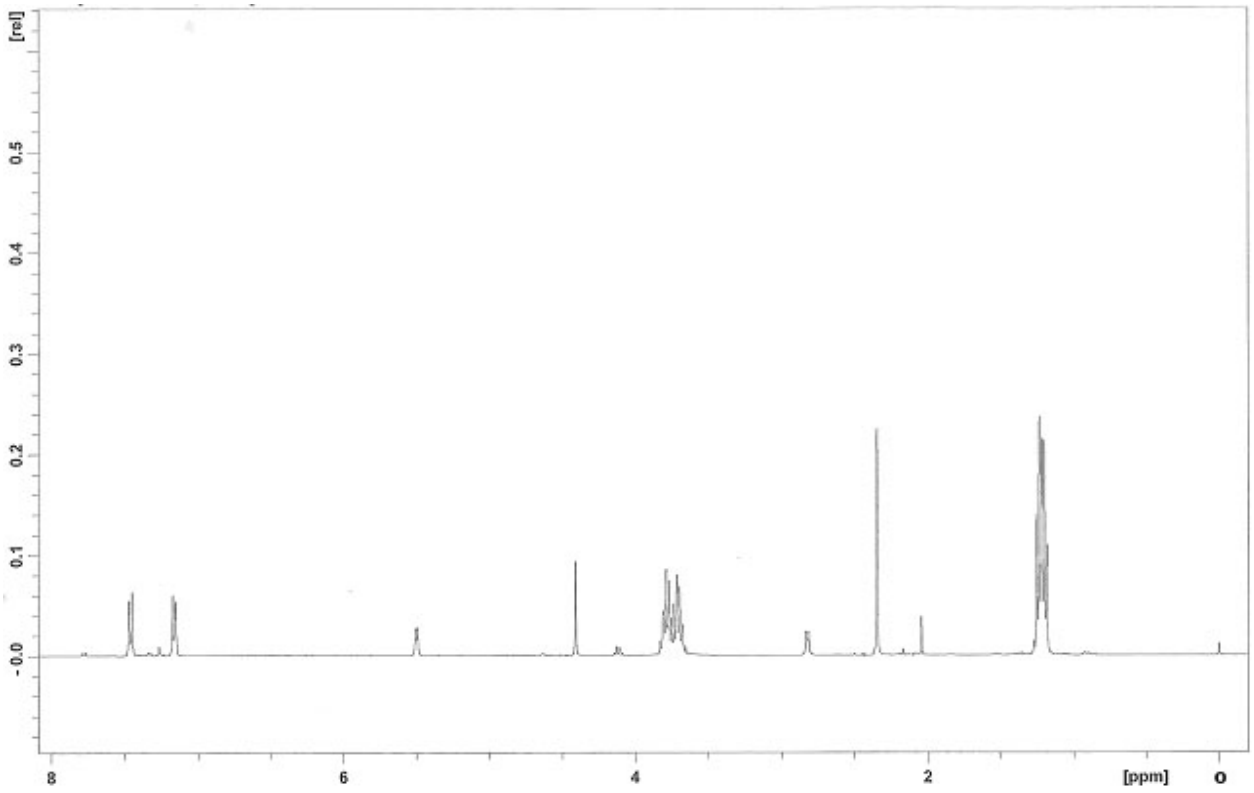
2d



^{1}H NMR spectrum

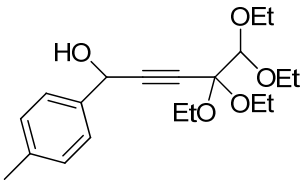


2g

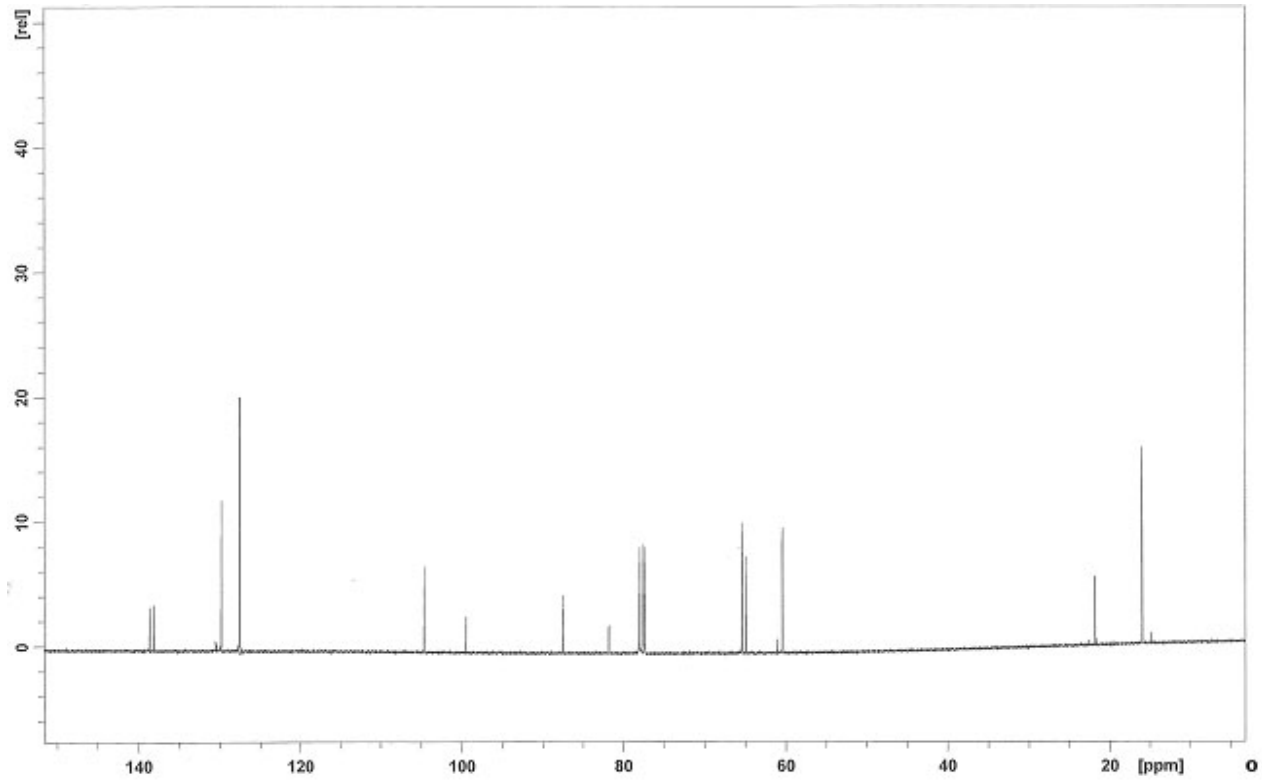


Extra peaks from EtOAc (q 4.16, s 2.04 ppm)

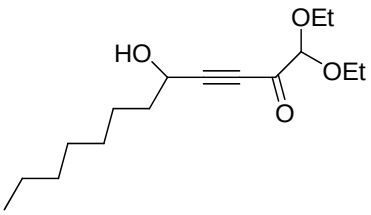
^{13}C NMR spectrum



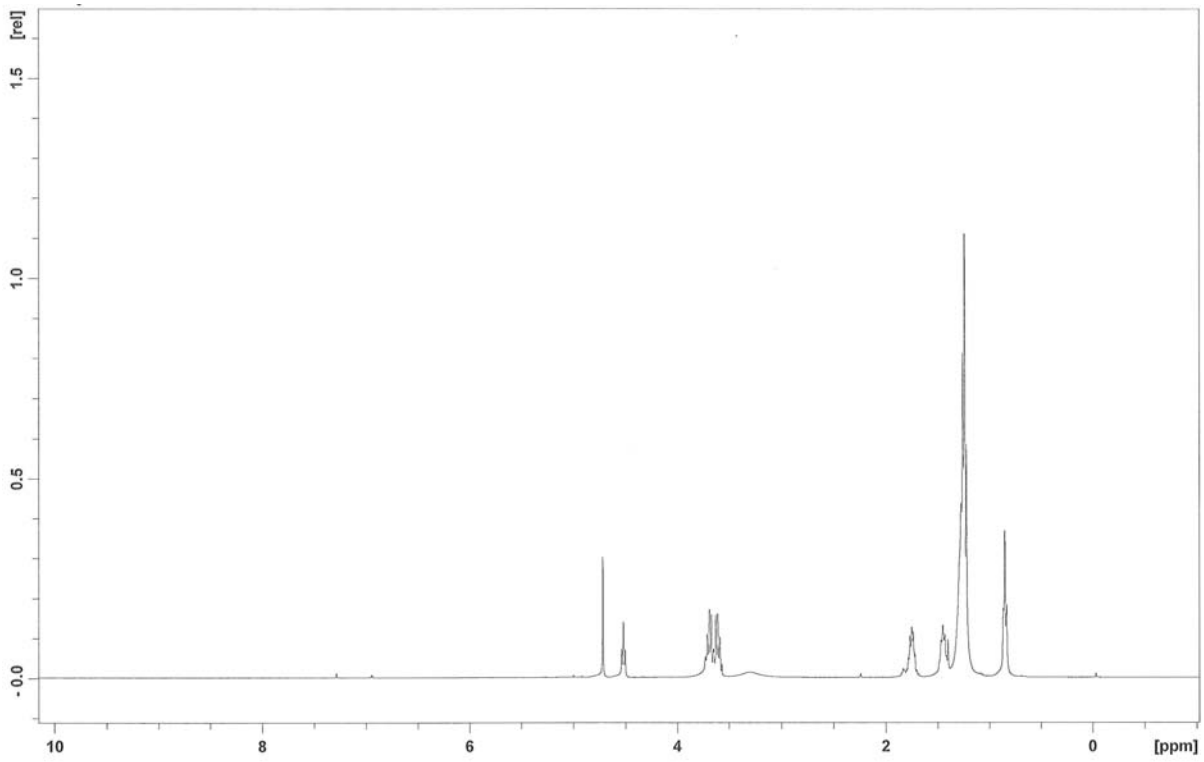
2g



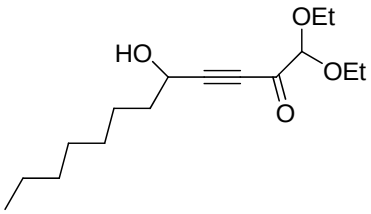
^{1}H NMR spectrum



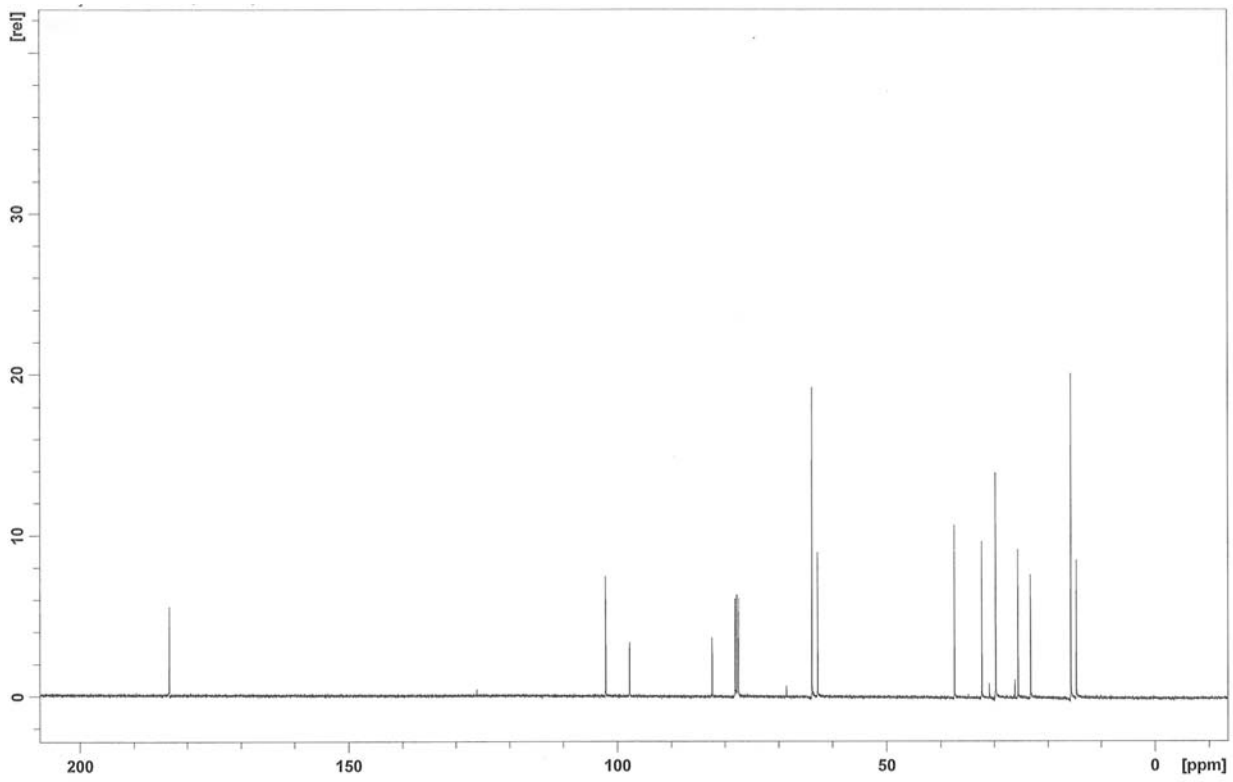
3d



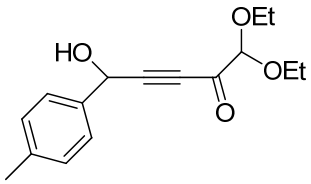
^{13}C NMR spectrum



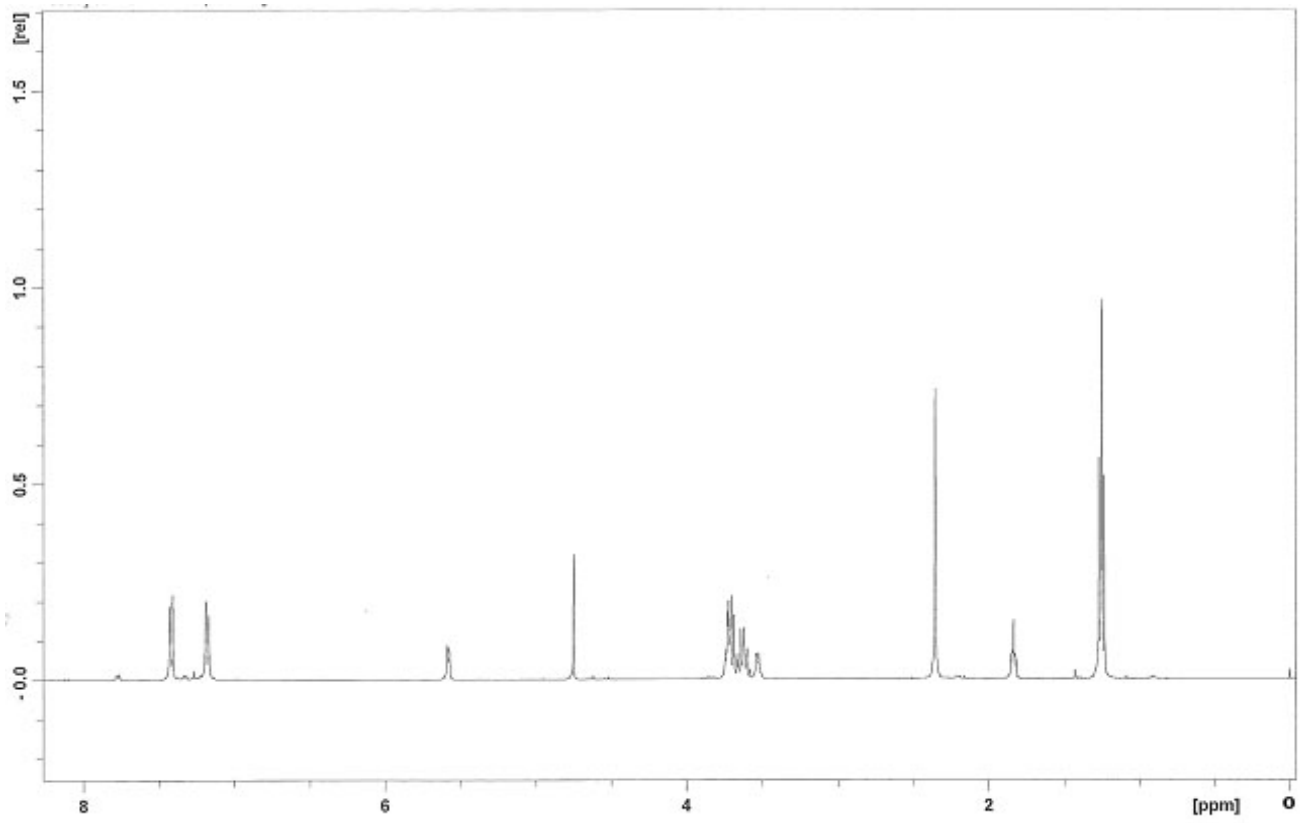
3d



^{1}H NMR spectrum

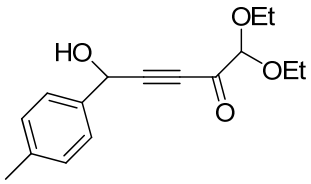


3g

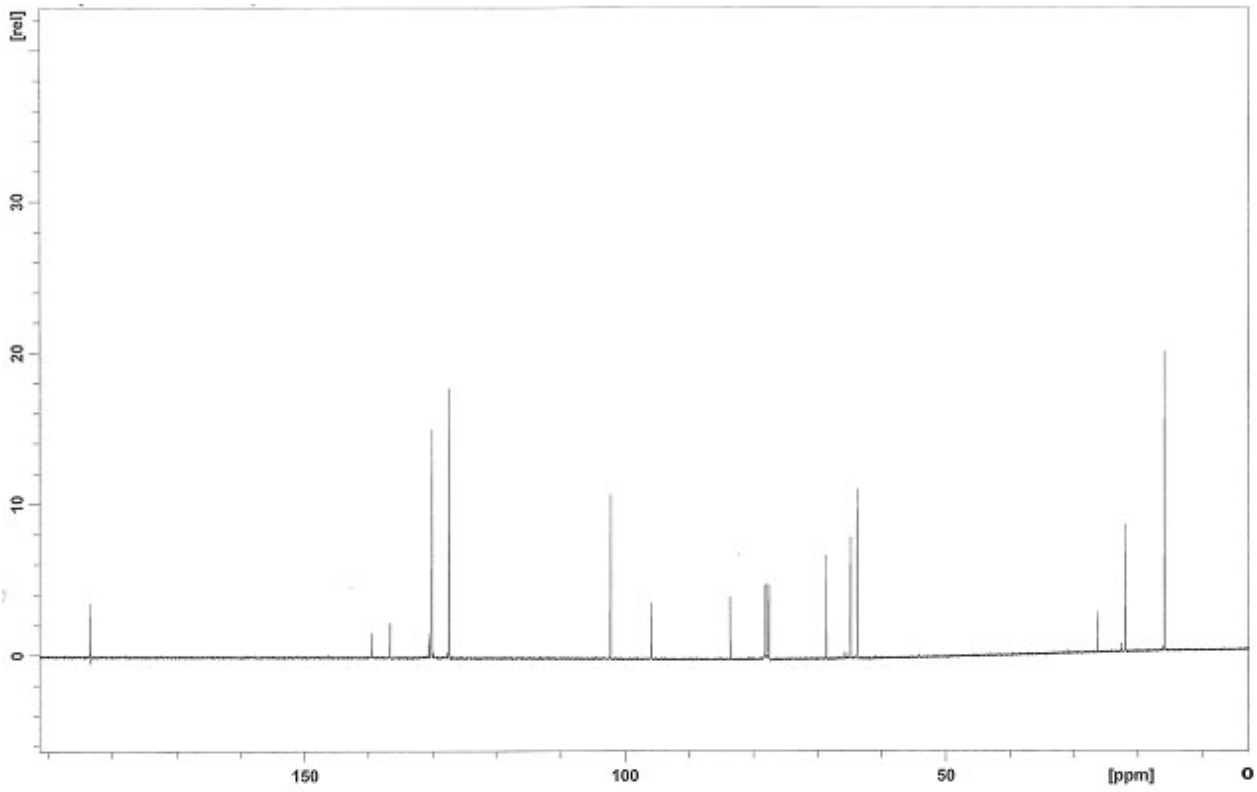


Extra peaks (m 3.78 ppm, m 1.86 ppm) from THF

^{13}C NMR spectrum

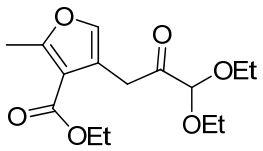


3g

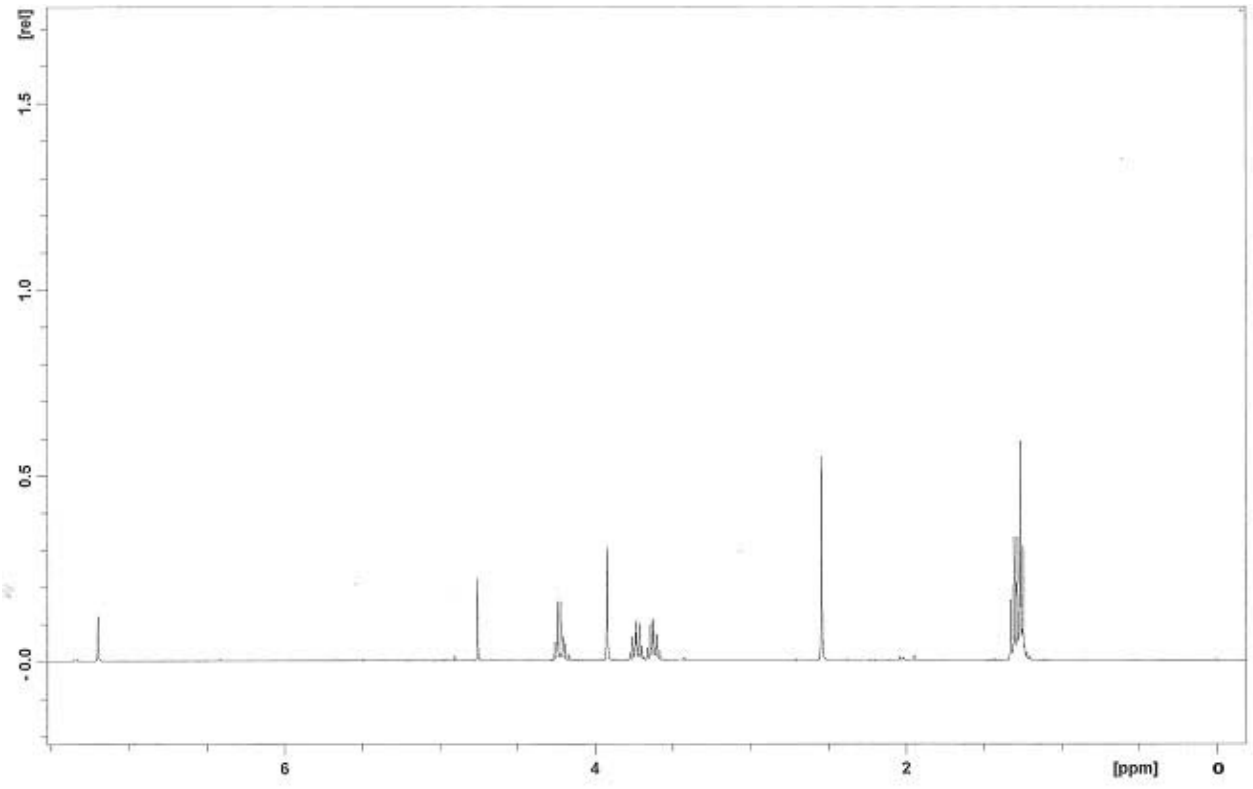


Extra peaks (26.5 ppm , 68.4 ppm) from THF

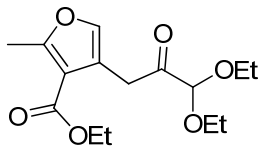
^{1}H NMR spectrum



4a



^{13}C NMR spectrum



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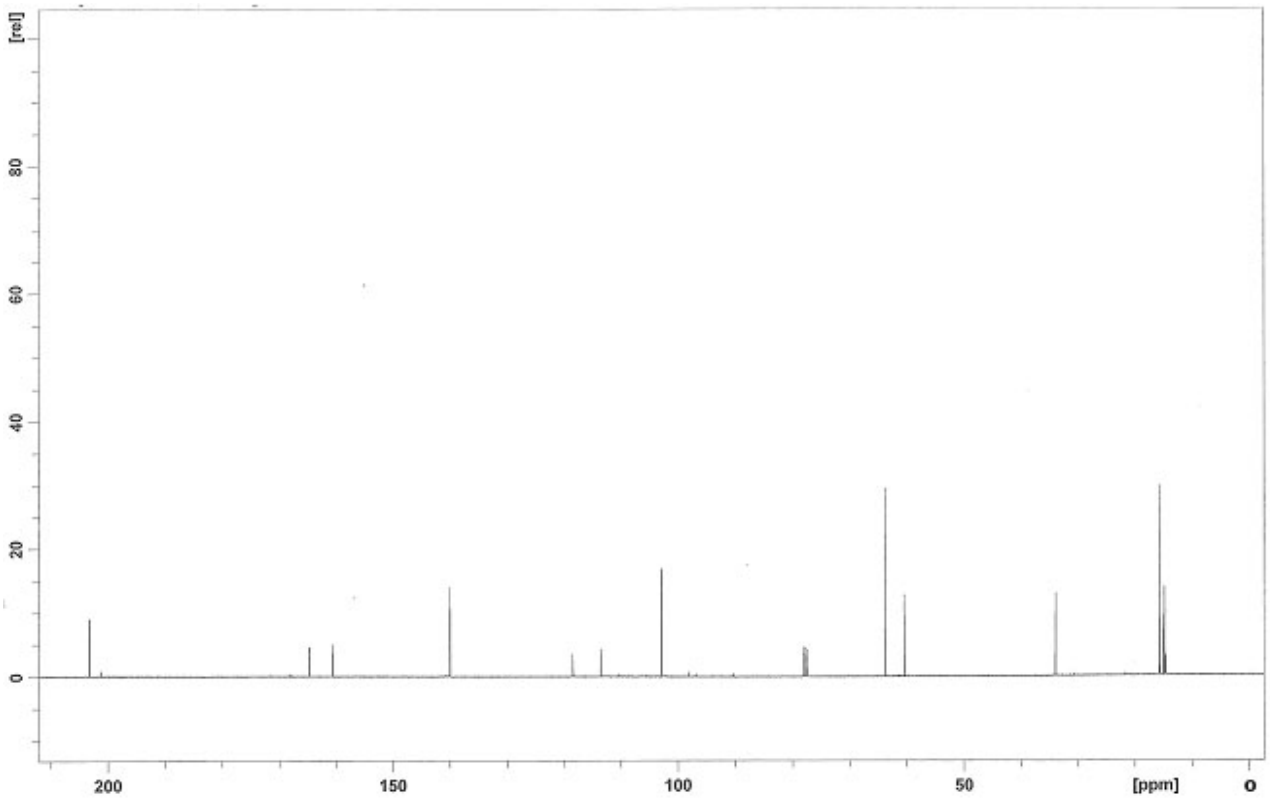
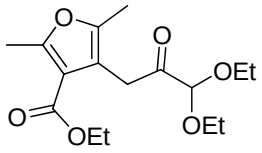


^{1}H NMR spectrum



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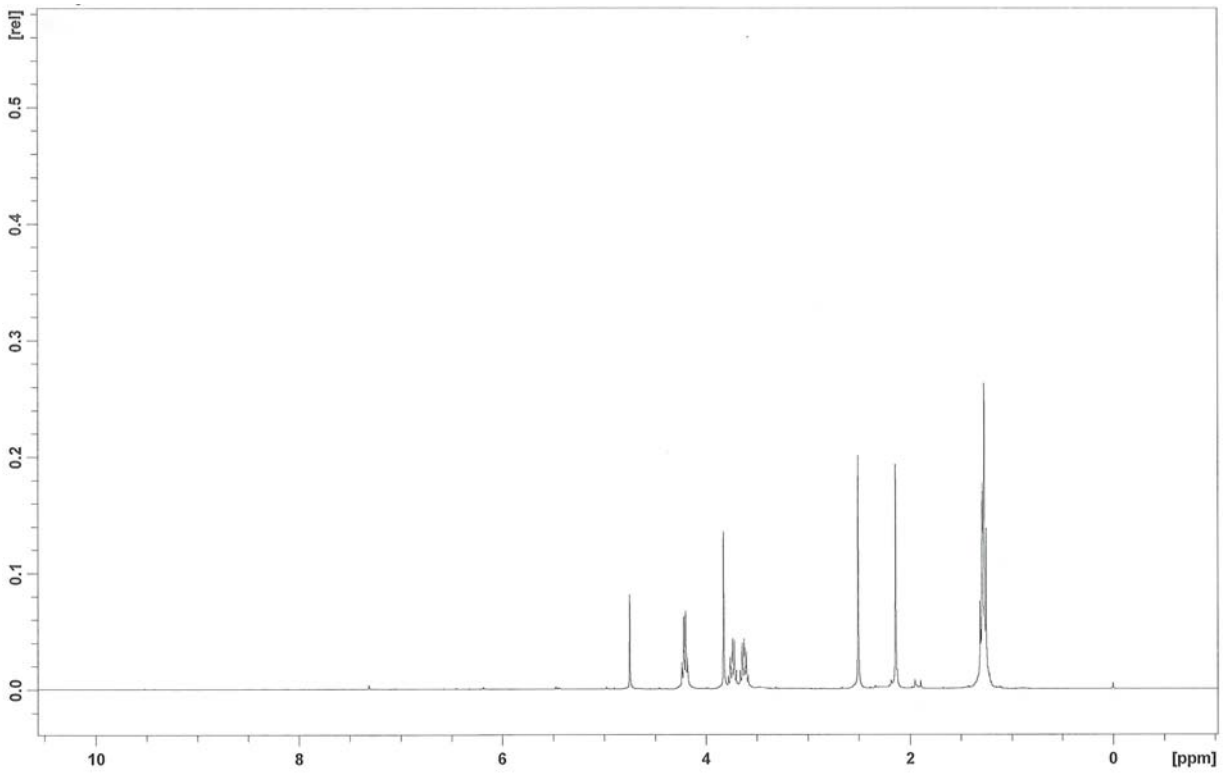
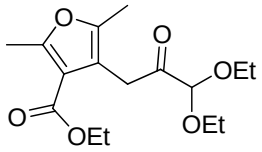


^{13}C NMR spectrum



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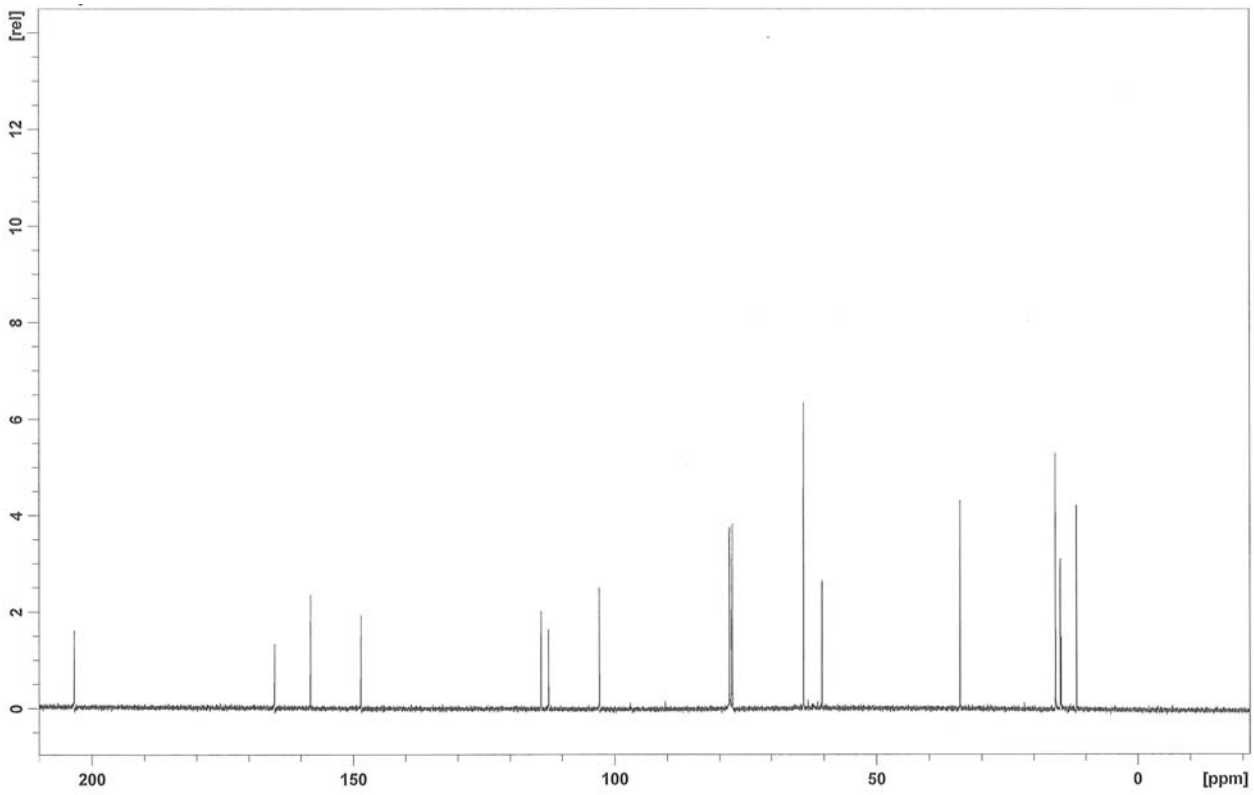
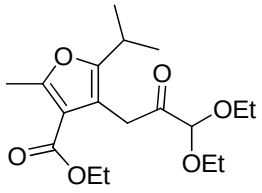


^{1}H NMR spectrum



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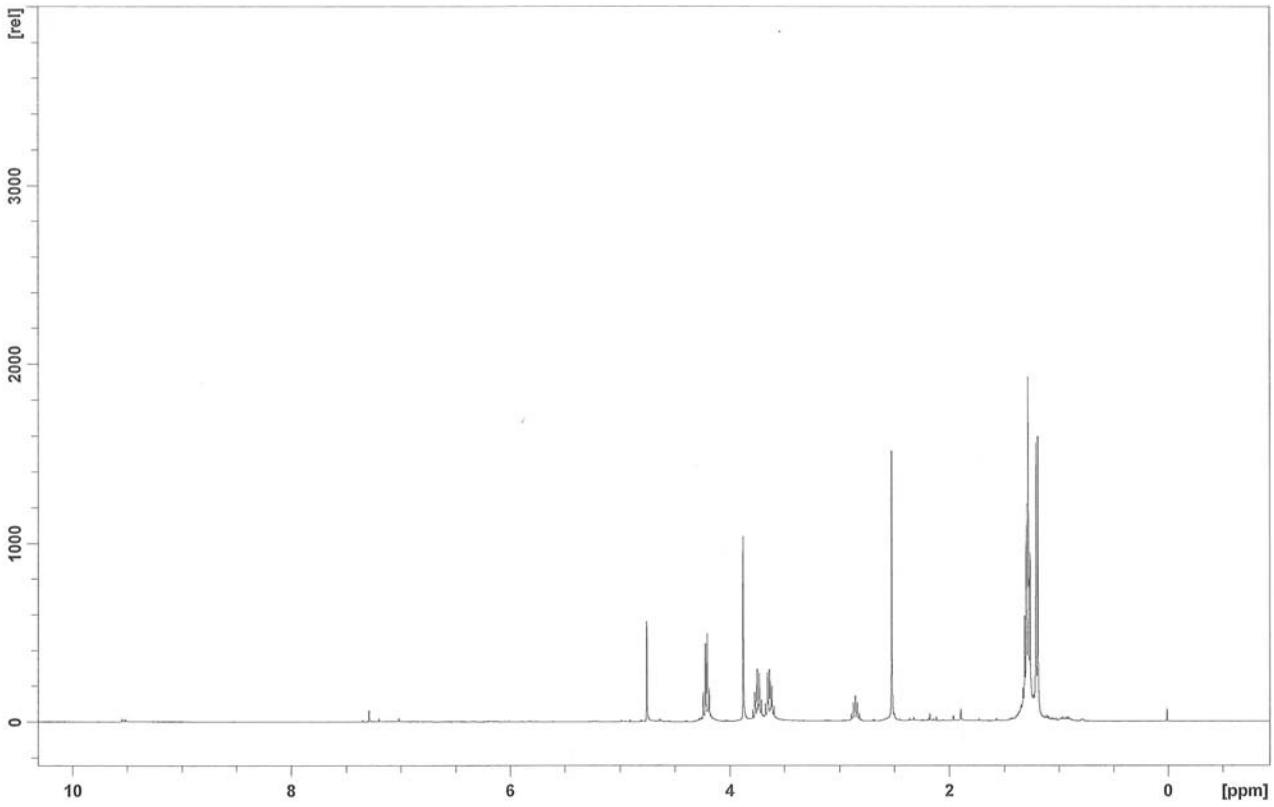
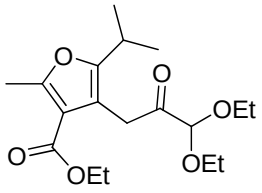


^{13}C NMR spectrum



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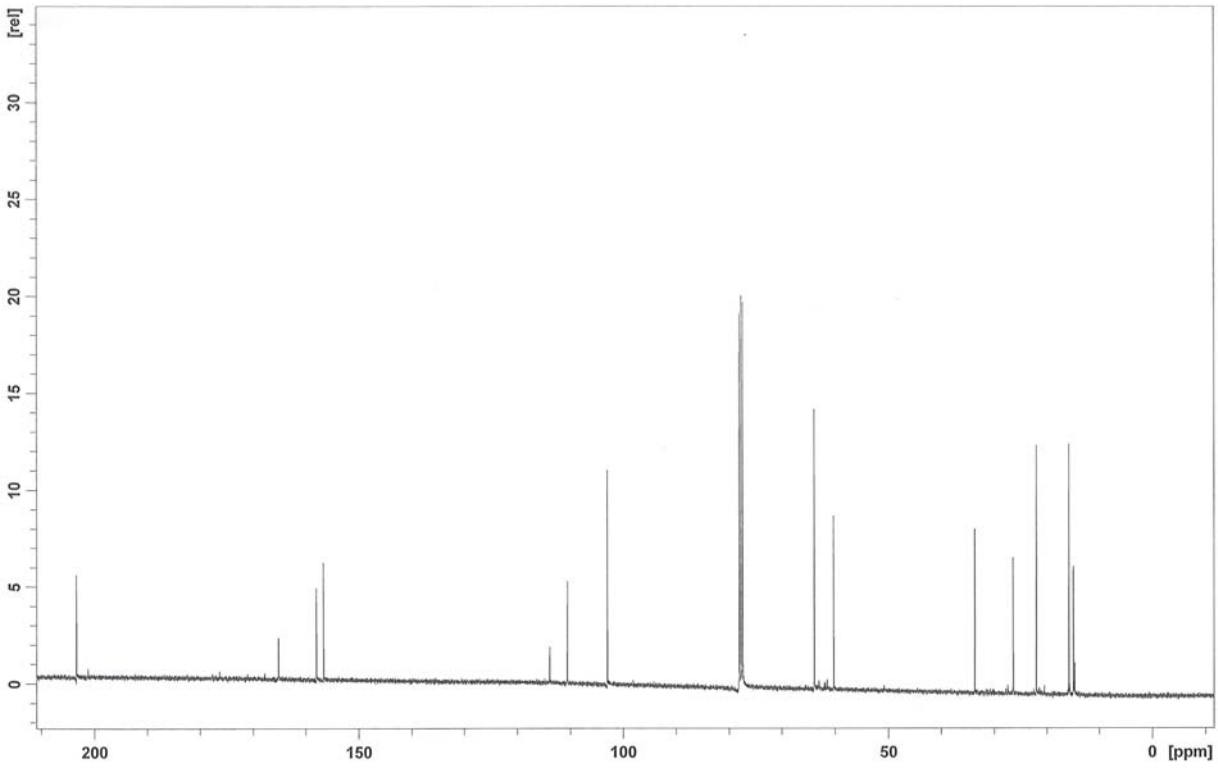
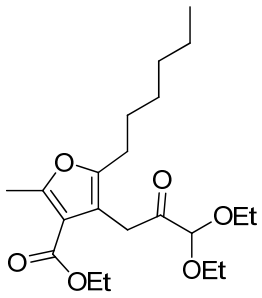


^{1}H NMR spectrum



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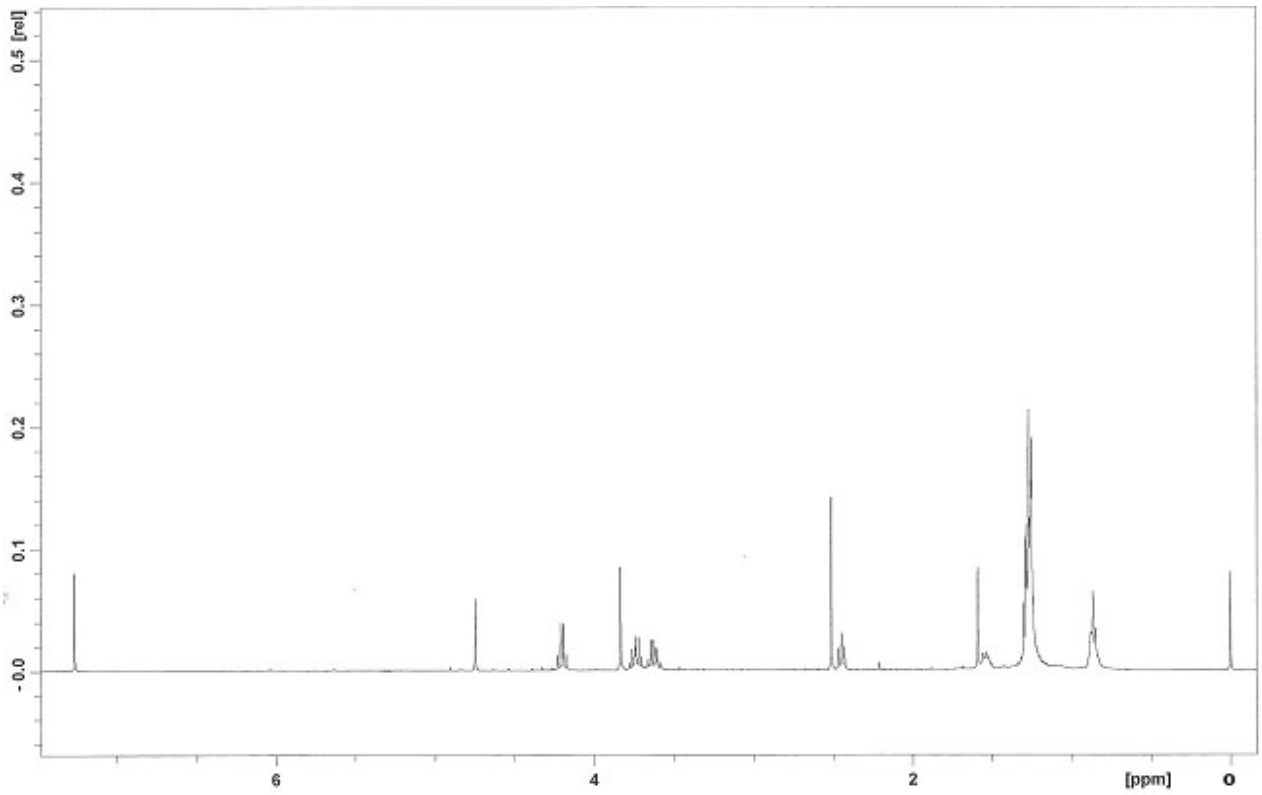
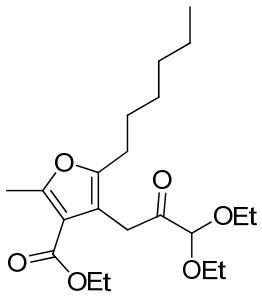
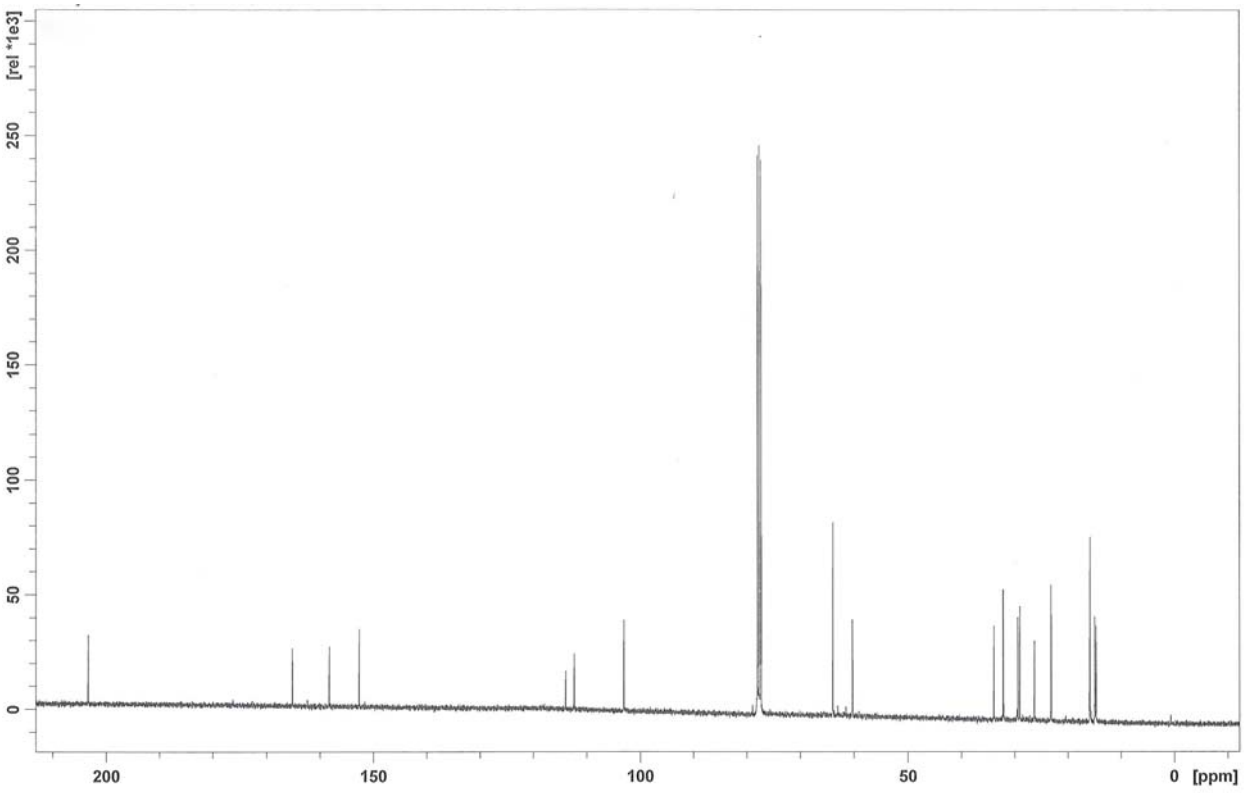


Extra peak (1.61 ppm) due to water in $CDCl_3$

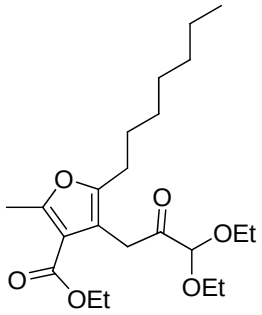
^{13}C NMR spectrum



4d



^{1}H NMR spectrum



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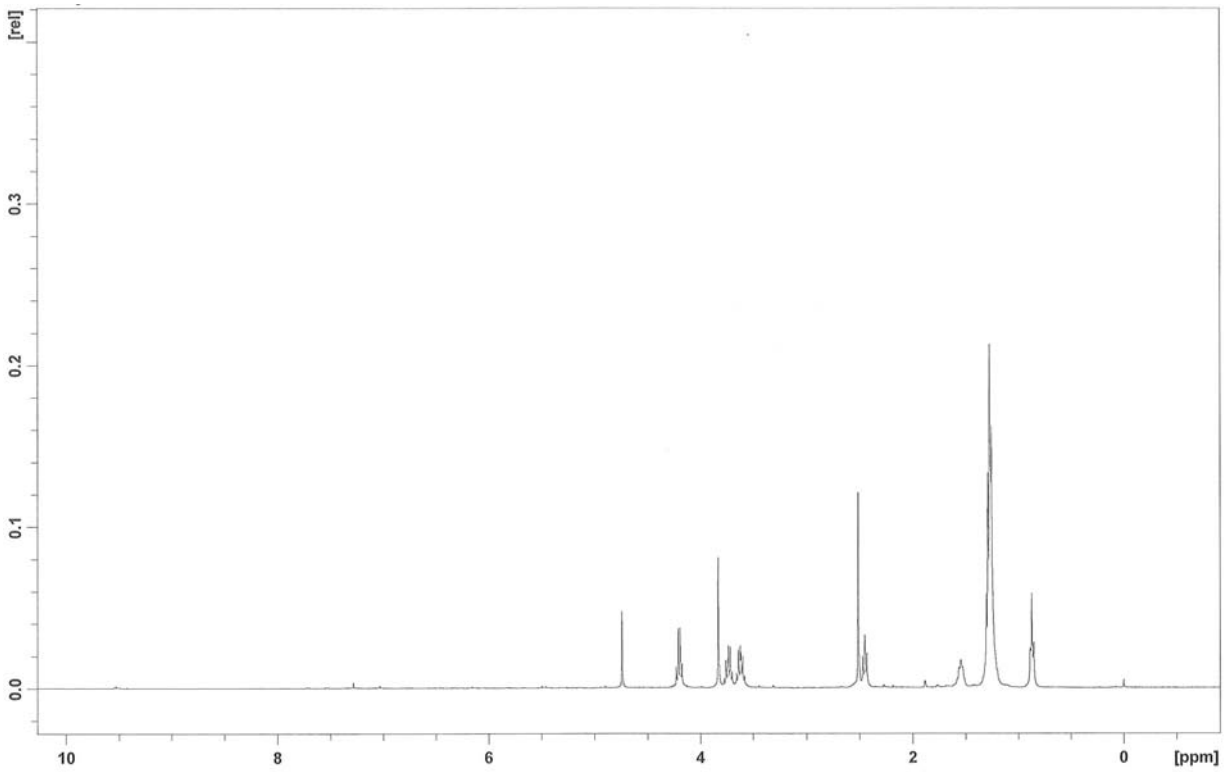
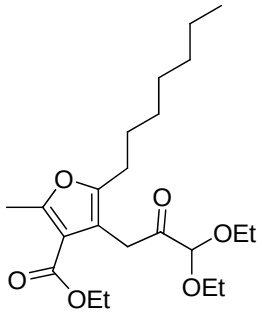


^{13}C NMR spectrum



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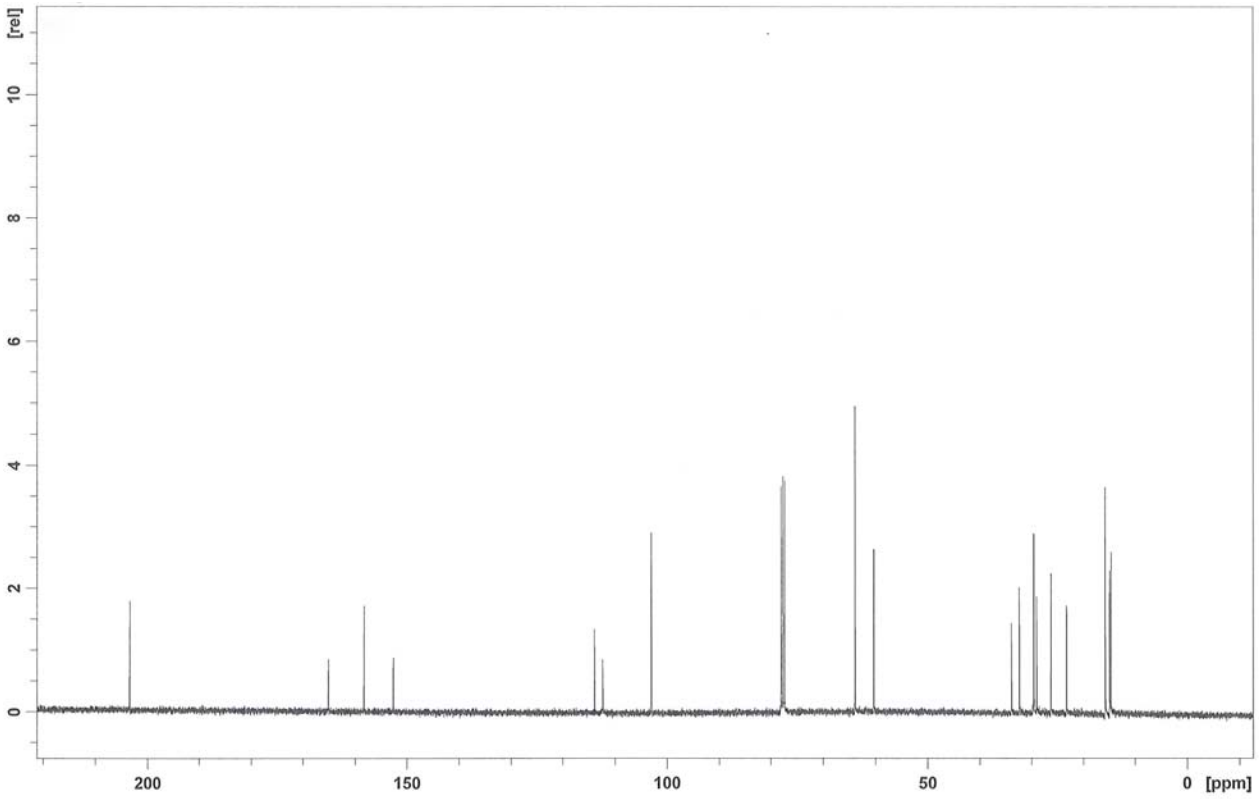
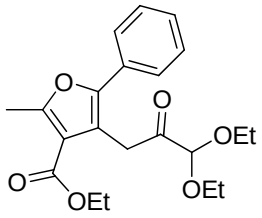


^{1}H NMR spectrum



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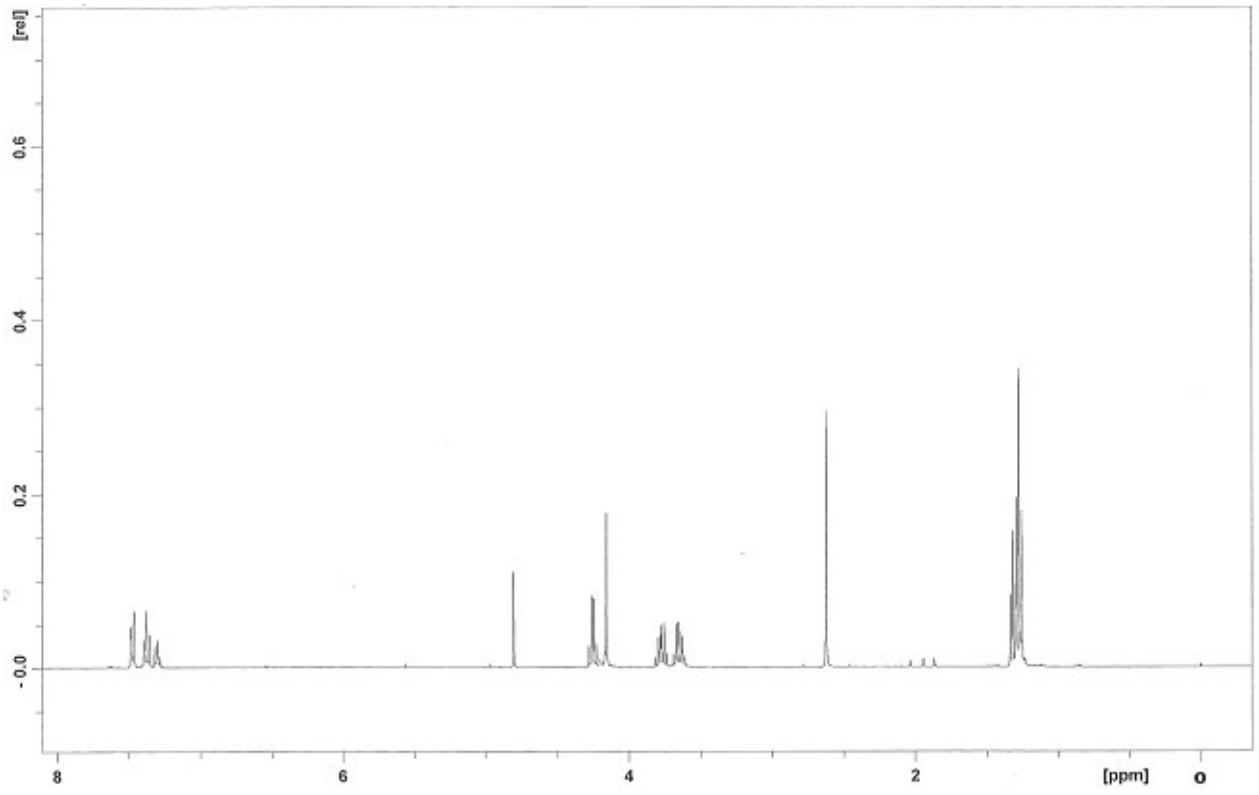
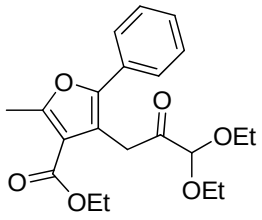


^{13}C NMR spectrum



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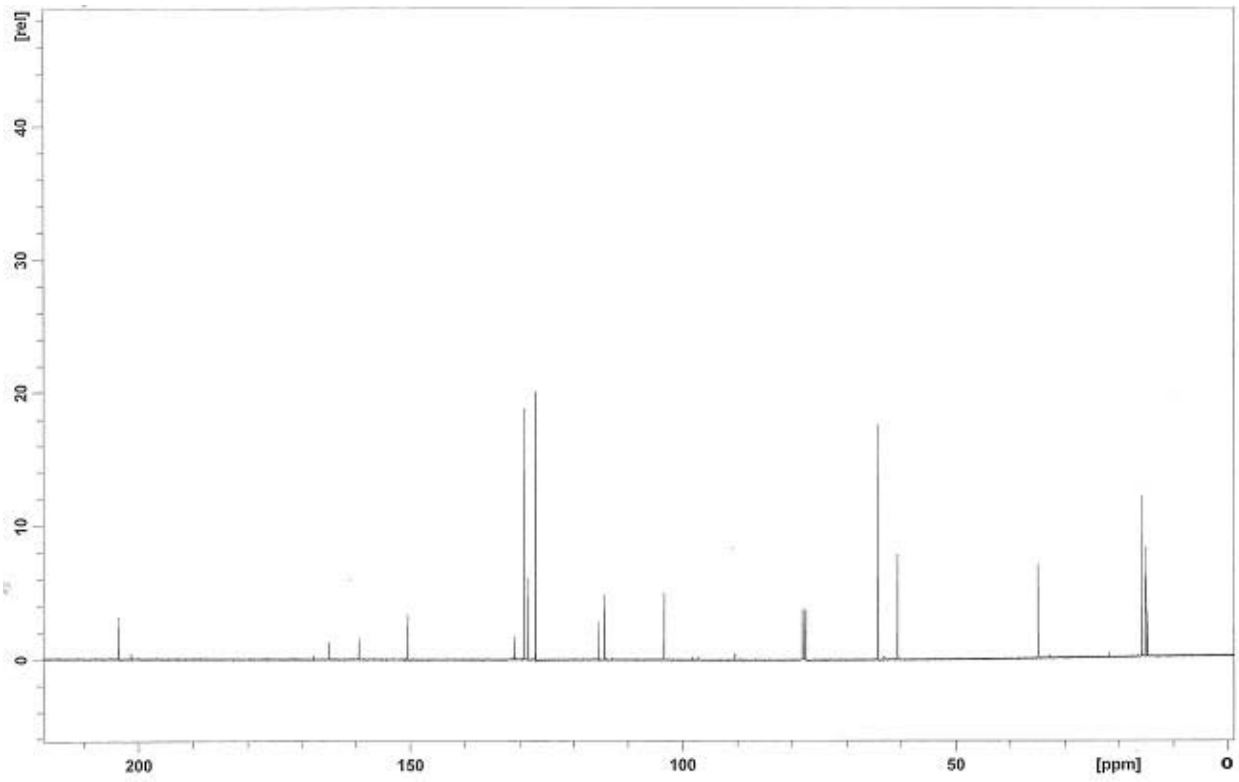
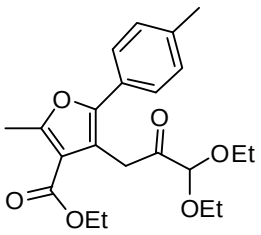
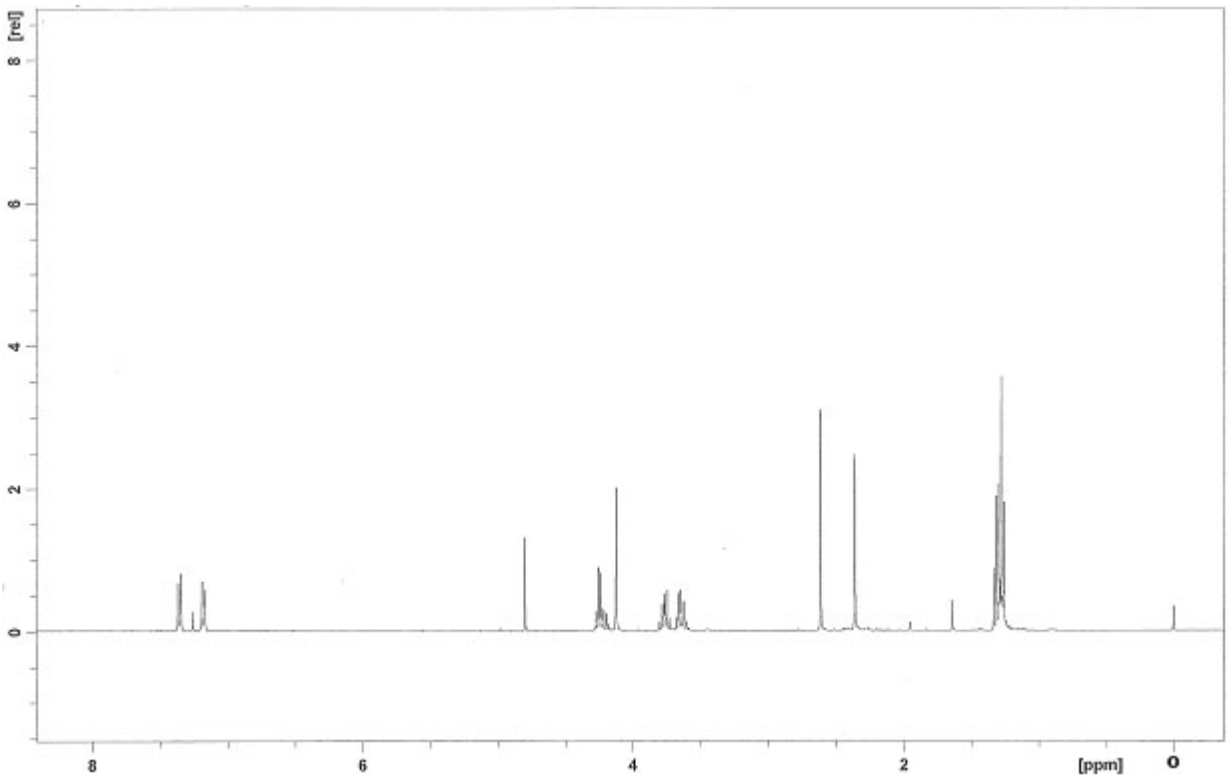


^{1}H NMR spectrum

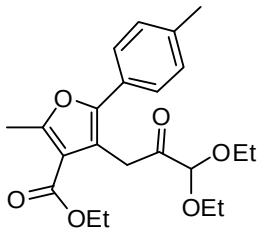


4g

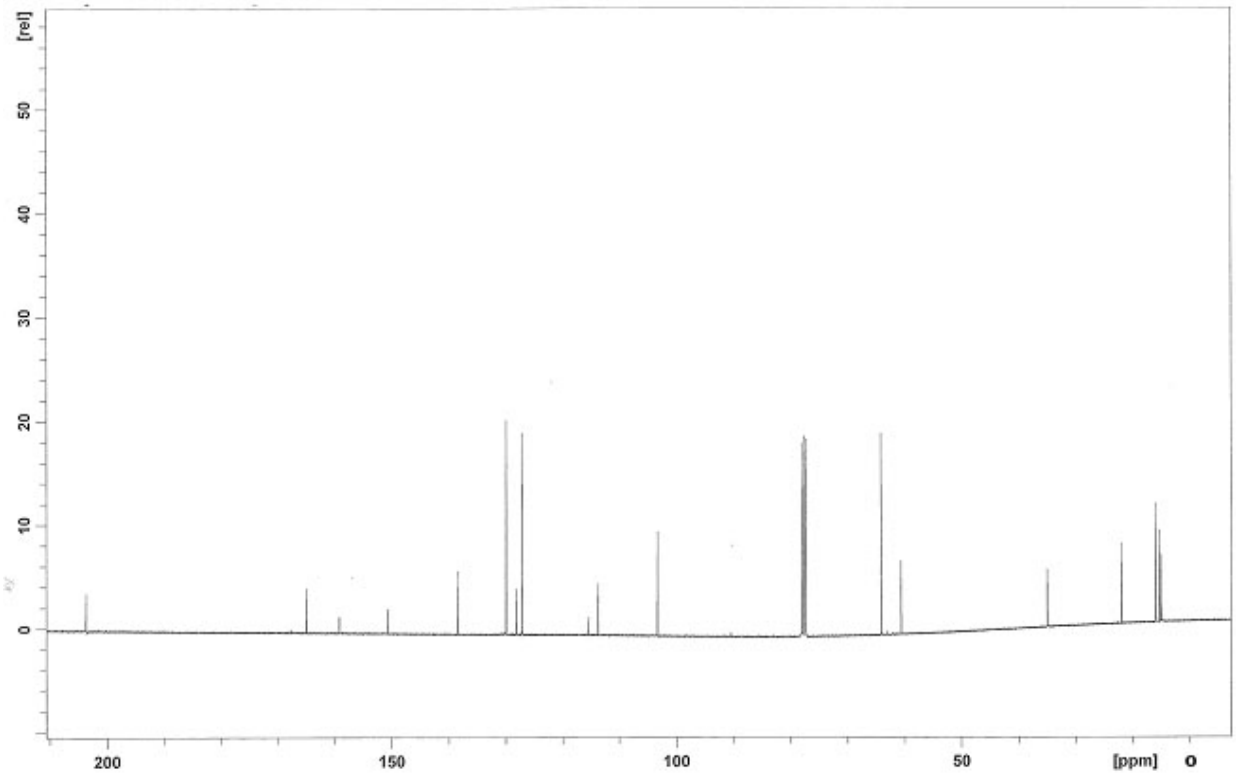


Extra peak (1.63 ppm) water in $CDCl_3$

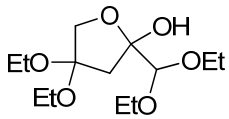
^{13}C NMR spectrum



4g



^{1}H NMR spectrum



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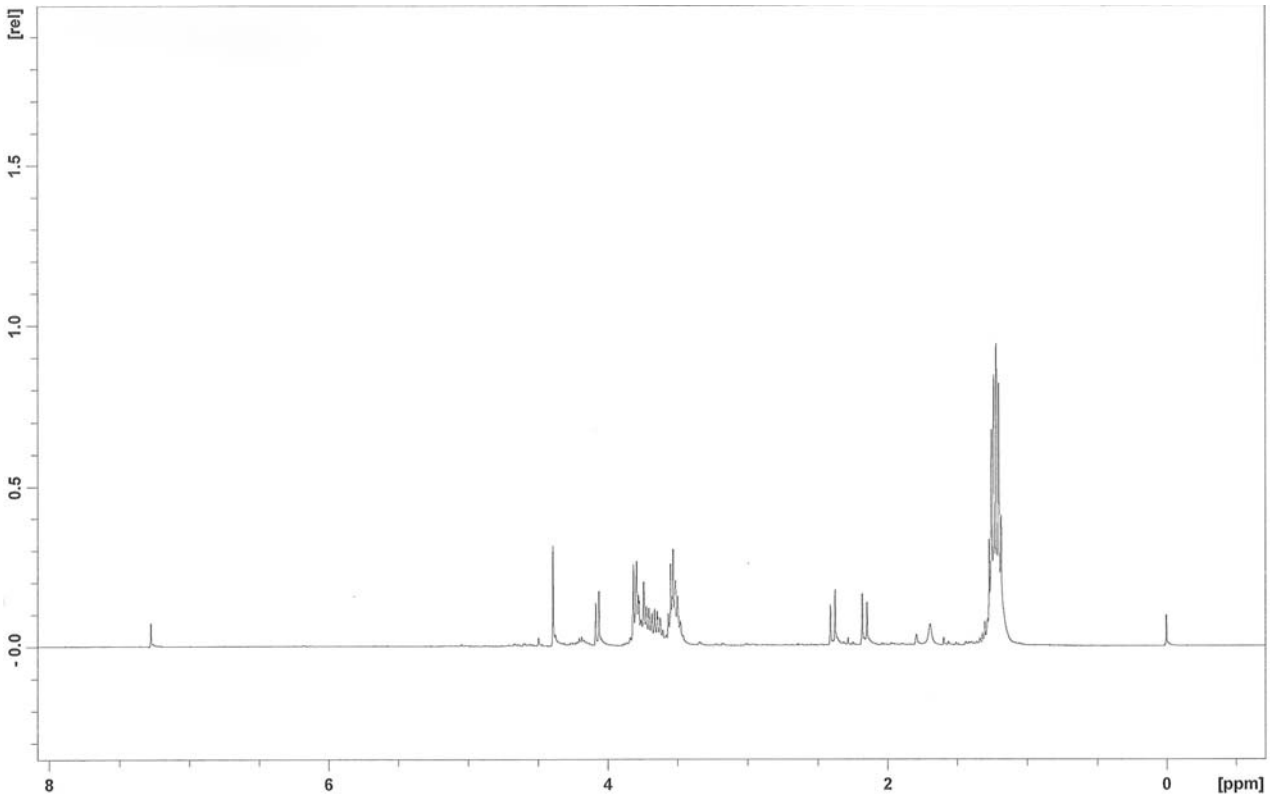
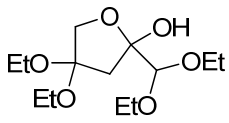


Extra peak (1.65 ppm) water in $CDCl_3$

^{13}C NMR spectrum



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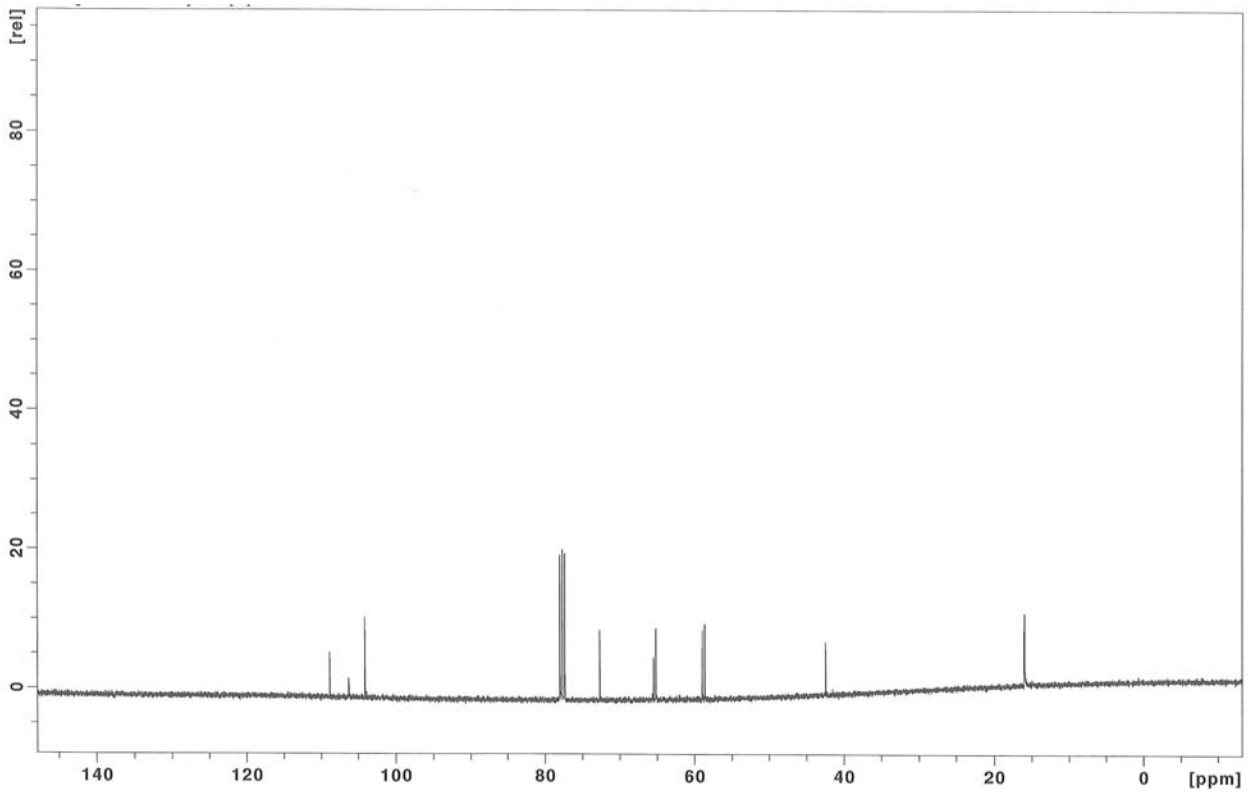
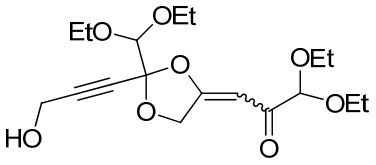
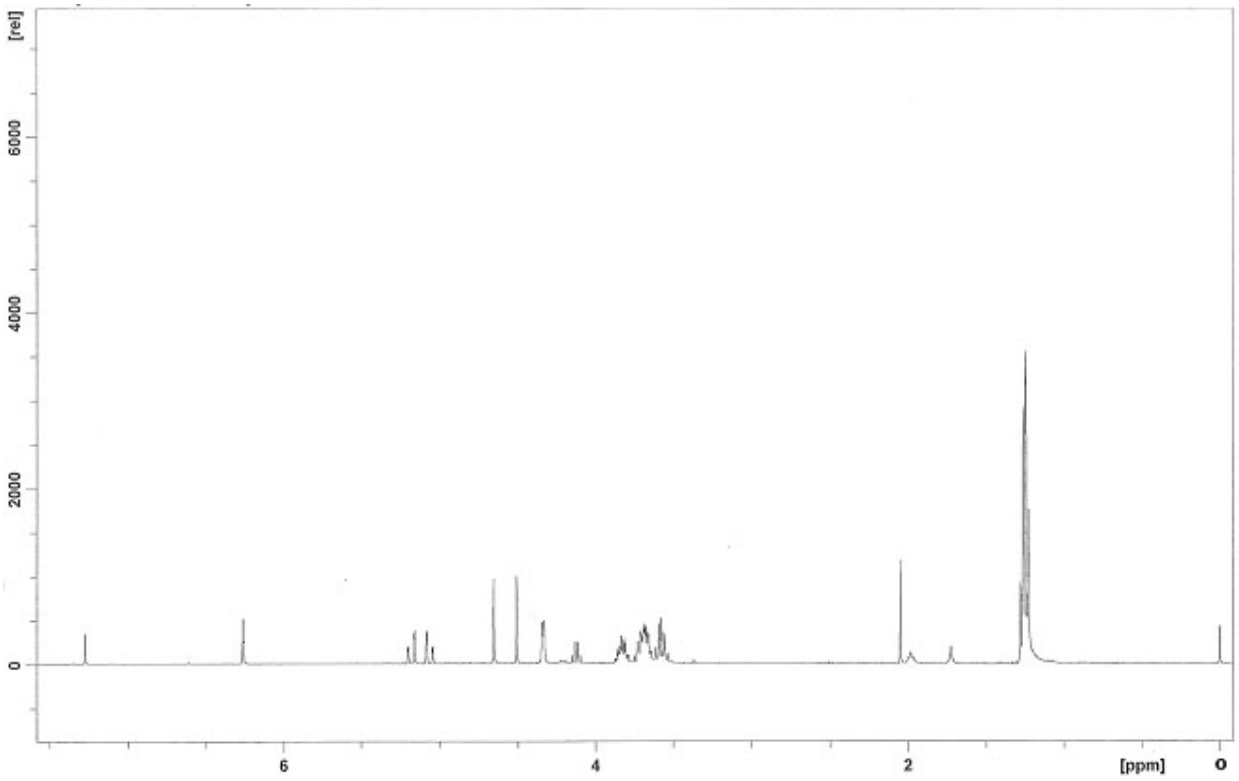


^{1}H NMR spectrum

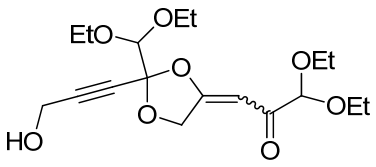


6



Extra peaks from ethyl acetate (q 4.23, s 2.05) and from water (1.73) in $CDCl_3$

^{13}C NMR spectrum



6