Solvo-surfactant properties of dialkyl glycerol ethers: Application as eco-friendly extractants of plant material through a novel Hydrotropic-Cloud-Point-Extraction (HCPE) process

SUPPORTING INFORMATION

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Pages 2 - 7: Synthesis of alkyl glycerol ethers.

Pages 8 -37: ^{1}H NMR, ^{13}C NMR, IR and GC-MS spectra and chromatograms of the compounds.

Page 38: cloud points of [4.0.1] and [4.1.0] mixtures.

Page 40: phases diagrams of dialkyl glycerol ethers [*i*-4.0.1] and [*i*-4.1.0].

Page 41: ^{1}H NMR spectra of HCPE extract of piperine

Synthesis of alkyl glycerol ethers:

General information:

Methyl-, ethyl-, and butyl glycidyl ethers were purchased from TCI (>85%, > 98% and >98% respectively). Epichlorohydrin (>99%), ethylene glycol butyl ether (>99%) and Pd/C (10 wt%) were obtained from Sigma Aldrich. Sodium xylensulfonate (technical, mixture of isomers, >90%) was delivered by Fluka and piperine (>98%) by Alfa Aesar. Water was purified by a Millipore® apparatus Simplicity 185 and collected at the resistivity of 18.2 MΩ.cm.

Dialkyl ethers were purified by spinning band distillation using a Model 800 Micro Distillation System from B/R Instrument Corporation.

Infrared spectra were recorded on a Nicolet 380 ATR FT-IR (Thermo Electron Corporation).

NMR spectra were recorded on a Bruker 300 apparatus and calibrated relative to TMS in CDCl3.

Synthesis of [x.0.0] products:

Synthesis of 3-butoxypropane-1,2-diol [4.0.0]:

Butyl glycidyl ether (25,12 g, 200 mmol) was added to water (500 mL). The two-phase mixture was heated at 100°C for 48 hours under reflux and became homogeneous. After evaporation of water, the crude product was distilled under 4.6 10-2 mbar and the product was collected at 72-82°C (23.09 g, 156 mmol, 78%).

1H NMR (CDCl3, 300 MHz): 3.87 (m, 1H), 3.67 (ABX system, JAB = 39.7 Hz, JAB = 5.4 Hz, JBX = 3.7 Hz, 2H), 3.55-3.45 (m, 4H), 3.00 (bs, 1H, OH), 2.68 (bs, 1H, OH), 1.61-1.52 (m, 2H), 1.43-1.31 (m, 2H), 0.92 (t, J = 7.3 Hz, 3H).

13C NMR (CDCl3, 75 MHz): 72.5 (t), 71.6 (t), 70.7 (d), 64.3 (t), 31.7 (t), 19.4 (t), 14.0 (q).

ATR-FTIR: νmax/cm-1 3372, 2957, 2933, 2867, 1110, 1042.

GG-MS m/z (%): 117 (03) [M-CH2OH]+, 87 (10) [n-BuOCH2]+, 61 (66) [CH2OHCHOH]+, 57 (100) [n-Bu]+.

Synthesis of *3-pentoxypropane-1,2-diol [5.0.0]:*

Same procedure as for [4.0.0] was followed using pentyl glycidyl ether (30.0 g, 200 mmol) and water (600 mL). The product was distilled under 4.6 10^{-2} mbar and collected at 99-106°C (23.98 g, 148 mmol, 74%).

^{1}H NMR ($CDCl_3$, 300 MHz): 3.87 (m, 1H), 3.67 (ABX system, J_{AB} = 40.9 Hz, J_{AB} = 5.6 Hz, J_{BX} = 3.8 Hz, 2H), 3.55-3.44 (m, 4H), 2.95 (bs, 2H, OH), 1.59 (quint*a*, *J* = 6.9 Hz, 2H), 1.34-1.29 (m, 4H), 1.43-1.31 (m, 2H), 0.90 (t, *J* = 6.9 Hz, 3H).

^{13}C NMR ($CDCl_3$, 75 MHz): 72.5 (t), 71.9 (t), 70.7 (d), 64.3 (t), 29.4 (t), 28.3 (t), 22.6 (t), 14.1 (q).

ATR-FTIR: 3366, 2930, 2860, 1458, 1378, 1110, 1042 cm^{-1}.

GC-MS m/z (%): 131 (02) $[M - CH_2OH]^+$, 101 (06) $[n\text{-}PentOCH_2]^+$, 71 (64) $[n\text{-}Pentyl]^+$, 61 (100) $[CH_2OHCHOH]^+$, 57 (100) $[n\text{-}Bu]^+$.

Synthesis of [x.0.1] products:

Synthesis of *1-methoxy-3-propoxypropan-2-ol [3.0.1]:*
Sodium (0.069 g, 30 mmol) was dissolved in methanol (60 mL) under argon flow, then butyl glycidyl ether (34.8 g, 0.30 mol) was added and the solution was heated at 75°C for 16 hours. After evaporation of the most of the methanol, the product was poured into a solution composed of 200 mL of 1M HCl and 200 mL of brine, then extracted three times by diethyl ether. The combined organic fractions were washed with a saturated $NaHCO_3$ solution, dried over magnesium sulfate and filtered. After evaporation of the solvent, the crude product was purified by spinning band distillation under 20 torr and the product was collected at 90-92°C (34.2 g, 0.23 mol, 77%).

^{1}H NMR ($CDCl_3$, 300 MHz): 3.96 (m, 1H), 3.52-3.39 (m, 6H), 3.39 (s, 3H), 2.57 (bs, 1H, OH), 1.60 (hex*a*, *J* = 7.2 Hz, 2H), 0.92 (t, *J* = 7.2 Hz, 3H).
^{13}C NMR ($CDCl_3$, 75 MHz): 74.0 (t), 73.3 (t), 71.8 (t), 69.5 (d), 59.3 (q), 22.9 (t), 10.6 (q).
ATR-FTIR: 3444, 2876, 1455, 1196, 1104, 968 cm^{-1}.
GC-MS m/z (%): 103 (13) [M - $CH_2OMe]^+$, 89 (12) [M – *n*-PrO]$^+$, 75 (75) [$MeOCH_2CHOH]^+$, 73 (68) [n-$PrOCH_2]^+$, 61 (100) [$CH_2OHCHOH]^+$.

Synthesis of 1-butoxy-3-methoxypropan-2-ol [4.0.1]: Sodium (4,6 g, 200 mmol) was dissolved in methanol (1 L) under argon flow, then butyl glycidyl ether (130 g, 1 mol) was added and the solution was heated at 75°C for 16 hours. After evaporation of the most of methanol, the product was poured into a solution composed of 200 mL of 1M HCl and 200 mL of brine, then extracted three times by diethyl ether. The combined organic fractions were washed with a saturated NaHCO3 solution, dried over magnesium sulfate and filtered. After evaporation of the solvents, the crude product was distilled at 1.3 mbar and the product was collected at 58- 62°C (133 g, 0.82 mol, 82%).

1H NMR (CDCl3, 300 MHz): 3.96 (m, 1H), 3.52-3.40 (m, 6H), 3.39 (s, 3H), 2.47 (d, J = 4.0 Hz, 1H, OH), 1.61-1.52 (m, 2H), 1.43-1.31 (m, 2H), 0.92 (t, J = 7.3 Hz, 3H).
13C NMR (CDCl3, 75 MHz): 74.0 (t), 71.9 (t), 71.6 (t), 69.5 (d), 59.4 (q), 31.8 (t), 19.4 (t), 14.0 (q).
ATR-FTIR: νmax/cm-1 3434, 2930, 2871, 1458, 1105, 967.
GC-MS m/z (%): 117 (04) [M-CH2OMe]+, 87 (16) [n-BuOCH2]+, 75 (30) [MeOCH2CHOH]+, 57 (100) [n-Butyl]+.

Synthesis of *1-isobutoxy-3-methoxypropan-2-ol [i-4.0.1]:*
Same procedure as for [3.0.1] was used with the following quantities: sodium (1,6 g, 69.6 mmol), methanol (100 mL) and isobutyl glycidyl ether (25 g, 192 mmol) . The crude oil was distilled under 1.7 torr and the product was collected at 68-70°C (18.3 g, 113 mol, 59%).

^{1}H NMR ($CDCl_3$, 300 MHz): 3.96 (quint*a*, *J* = 5.0 Hz, 1H), 3.51-3.41 (m, 4H), 3.39 (s, 3H), 3.27-3.18 (m, 2H), 2.61 (bs, 1H, OH), 1.87 (nona*a*, *J* = 6.7 Hz, 1H), 0.90 (d, *J* = 6.7 Hz, 6H).
^{13}C NMR ($CDCl_3$, 75 MHz): 78.5 (t), 74.0 (t), 72.0 (t), 69.5 (d), 59.3 (q), 28.4 (d), 19.4 (2 x q).
ATR-FTIR: 3450, 2955, 2872, 1462, 1196, 1104, 970.
GC-MS m/z (%): 117 (03) [M – $CH_2OMe]^+$, 87 (08) [*i*-$BuOCH_2]^+$, 75 (24) [$MeOCH_2CH_2OH]^+$, 57 (46) [isobutyl]$^+$.

Synthesis of [x.1.0] products:

Synthesis of 3-butoxy-2-methoxypropanol [4.1.0]:

First step: synthesis of 1-(benzyloxy)-3-butoxypropan-2-ol [4.0.Bn]: Sodium (0.92 g, 40 mmol) was dissolved in benzyl alcohol (64.8 g, 600 mmol) under argon, then butyl glycidyl ether (26 g, 200 mmol) was added and the solution was heated at 120°C for 16 hours. After cooling, a solution of brine (100 mL) diluted with hydrochloric acid solution (1M, 100 mL) was added, and the resulting mixture was extracted with diethyl ether. The organic layer was washed with brine, dried over magnesium sulfate and filtered. After evaporation of the solvents, the crude product was distilled at 3.9 10-2 mbar to recover first benzyl alcohol, then the product [4.0.Bn] between 125-150°C (30.09 g, 63%).

1H NMR (CDCl3, 300 MHz):7.39-7.27 (m, 5H), 4.56 (s, 2H), 3.97 (m, 1H), 3.58-3.42 (m, 6H), 2.53 (bs, 1H, OH), 1.60-1.50 (m, 2H), 1.42-1.29 (m, 2H), 0.91 (t, J = 7.2 Hz, 3H).

13C NMR (CDCl3, 75 MHz): 138.1 (s), 128.6 (2 x d), 127.9 (2 x d + d), 73.6 (t), 71.9 (t), 71.5 (t + t), 69.7 (d), 31.8 (t), 19.4 (t), 14.0 (q).

ATR-FTIR: νmax/cm-1 3450, 2956, 2930, 2864, 1454, 1092, 734, 697.

GC-MS m/z (%): 107 (05) [PhCH2O]+, 91 (100) [PhCH2]+, 77 (10), 65 (18), 57 (46) [isobutyl]+.

Second and third steps: In a 250 mL flask equipped with a big magnetic stirrer were introduced [4.0.Bn] (29.89 g, 125.6 mmol), powdered potassium hydroxide (14.1 g, 251.2 mmol), tetrabutyl ammonium bromide (2.02 g, 6.28 mmol) then dimethyl sulfate dropwise (11.9 mL, 125.6 mmol). Caution: exotherm. After stirring for 20 hours, a second portion of potassium hydroxide and dimethyl sulfate was added. A solid precipitated, which was dissolved by water addition (30 mL). After stirring for 4 hours more, the product was extracted by diethyl ether. The organic layer was washed with brine, dried over magnesium sulfate and filtered. After evaporation of the solvents, the crude product [4.1.Bn] (28.8 g) was recovered and used for hydrogenation without further purifications. To a part of this crude product (15.34 g, maximum of 60.87 mmol) in ethanol (50 mL) was added 10 wt% Pd/C (645 mg, 0.609 mmol, 1 mol%). The flask was surrounded by a hydrogen balloon and the atmosphere was purged by vacuum and replaced by hydrogen. After stirring for 20h at room temperature, the solution was filtered over celite and the solvents were evaporated. The liquid was distilled by spinning band microdistillation apparatus under vacuum (1 torr) and the fraction between 75-79°C was collected to get [4.1.0] (8.23 g, 50.8 mmol, 76% over two steps).

1H NMR (CDCl3, 300 MHz): 3.71 (ABX system, JAB = 51.8 Hz, JAx = 5.2 Hz, JAB = 4.3 Hz, 2H), 3.59-3.40 (m, 5H), 3.47 (s, 3H), 2.22 (bs, 1H, OH), 1.56 (quinta, J = 6.7 Hz, 2H), 1.42-1.30 (m, 2H), 0.92 (t, J = 7.3 Hz, 3H).

13C NMR (CDCl3, 75 MHz): 80.2 (d), 71.7 (t), 70.7 (t), 62.7 (t), 57.9 (q), 31.8 (t), 19.4 (t), 14.0 (q).

ATR-FTIR: νmax/cm-1 3444, 2957, 2932, 2869, 1464, 1114, 1088 824 cm-1.

GC-MS m/z (%): 131 (01) [M-CH2OH]+, 75 (58) [M – n-BuOCH2]+, 58 (100), 57 (56) [n-Butyl]+.

Synthesis of *3-isobutoxy-2-methoxypropanol* [*i*-4.1.0]:

First step to 1-(benzyloxy)-3-isobutoxypropan-2-ol [i4-0.Bn]:

Sodium (2.87 g, 0.125 mol) was dissolved in benzyl alcohol (150 mL, 1.45 mol) under argon, then isobutyl glycidyl ether (32.5 g, 0.25 mol) was added and the solution was heated at 120°C for 14 hours. After cooling, a solution of brine (100 mL) diluted with hydrochloric acid solution (1M, 100 mL) was added, and the resulting mixture was extracted with diethyl ether. The organic layer was washed with brine, dried over magnesium sulfate and filtered. After evaporation of the solvents, the crude product was distilled at 3.1 10^{-2} mbar to recover first benzyl alcohol, then the product [i4.0.Bn] between 120-135°C (36.4 g, 61%).

^{1}H NMR ($CDCl_3$, 300 MHz): 7.38-7.26 (m, 5H), 4.56 (s, 2H), 3.99 (m, 1H), 3.59-3.42 (m, 4H), 3.22 (AB system, *J* = 26.2 Hz, 2H), 2.54 (bs, 1H, OH), 1.86 (sept, *J* = 6.8 Hz, 1H), 0.89 (d, *J* = 6.8 Hz, 6H).
^{13}C NMR ($CDCl_3$, 75 MHz): 138.1 (s), 128.5 (2 x d), 127.8 (2 x d + d), 78.5 (t), 73.6 (t), 72.0 (t), 71.5 (t), 69.6 (d), 28.5 (d), 19.4 (t x 2).
ATR-FTIR: 3450, 2955, 2869, 1454, 1093, 1008, 734, 697.
GC-MS m/z (%): 107 (05) $[PhCH_2O]^+$, 91 (100) $[PhCH_2]^+$, 77 (10), 65 (18), 57 (46) $[isobutyl]^+$.

Second and third steps:
In a 250 mL flask equipped with a big magnetic stirrer were introduced [*i*4.0.Bn] (35.0 g, 147.06 mmol), powdered potassium hydroxide (10.66 g, 161.8 mmol), then dimethyl sulfate dropwise (15.3 mL, 161.8 mmol). After stirring for 10 hours, a second portion of potassium hydroxide (21.32 g, 323.6 mmol), water (20 mL), and dimethyl sulfate (15.3 g, 161.8 mmol) was added. After stirring for 48 hours, the mixture was diluted in water and extracted by diethyl ether. The organic layer was washed with water, brine, dried over magnesium sulfate and filtered. After evaporation of the solvents, the crude product [*i*4.1.Bn] (39.19 g) was recovered and used for hydrogenation without further purifications. To a part of this crude product (15.0 g, maximum of 59.5 mmol) in ethanol (50 mL) was added 10 wt% Pd/C (315 mg, 0.297 mmol, 0.5 mol%). The flask was surrounded by a hydrogen ballon and the atmosphere was purged by vacuum and displaced by hydrogen. After stirring for 48h at room temperature, the solution was filtered over celite and the solvents were evaporated. The liquid was distilled by spinning band microdistillation apparatus under vacuum (23 torr) and the fraction between 56-78°C was collected to get [*i*4.1.0] (5.56 g, 34.3 mmol, 61% over two steps).

^{1}H NMR ($CDCl_3$, 300 MHz): 3.80-3.62 (m, 2H), 3.59-3.39 (m, 3H), 3.45 (s, 3H), 3.27-3.17 (m, 2H), 2.25 (bs, 1H, OH), 1.87 (non*a*, *J* = 6.7 Hz, 1H), 0.92 (d, *J* = 6.7 Hz, 6H).
^{13}C NMR ($CDCl_3$, 75 MHz): 80.0 (d), 78.8 (t), 70.9 (t), 62.9 (t), 58.0 (q), 28.5 (d), 19.43 (q), 19.40 (q).
ATR-FTIR (cm^{-1}): 3419, 2955, 2872, 1468, 1366, 1086, 820.
GC-MS m/z (%): 87 (04) $[i\text{-}BuOCH_2]^+$, 75 (43) $[MeOCH_2CH_2OH]^+$, 58 (100), 57 (93) $[isobutyl]^+$.

Synthesis of [1.y.0] products:

Synthesis of 2-butoxy-3-methoxypropanol [1.4.0]:

First step: synthesis of 1-(benzyloxy)-3-methoxypropan-2-ol [1.0.Bn]: Sodium (460 mg, 20 mmol) was reacted with benzyl alcohol (20 g, 185 mmol) at room temperature under argon. Once the dissolution was completed, methyl glycidyl ether (3.52 g, 40 mmol) was added and the solution was heated at 80°C for 24 hours. After cooling, the solution was poured into HCl (1M, 50 mL) with brine (50 mL) and extracted with ethyl acetate (100 mL). The organic layer was washed with brine, dried over magnesium sulfate and filtered. After evaporation of the solvent the resulting oil was distilled to remove the excess of benzyl alcohol. The product was collected at 90°C under 3.9 10-2 bar (5.44 g, 27.8 mmol, 69%).

1H NMR (CDCl3, 300 MHz):7.38-7.29 (m, 5H), 4.56 (s, 2H), 3.99 (m, 1H), 3.58-3.42 (m, 4H), 3.38 (s, 3H), 2.56 (bs, 1H, OH).

13C NMR (CDCl3, 75 MHz): 138.1 (s), 128.6 (2 x d), 127.90 (d), 127.87 (2 x d), 73.9 (t), 73.6 (t), 71.4 (t), 69.6 (d), 59.3 (q).

ATR-FTIR: νmax/cm-1 3422, 2876, 1453, 1197, 1089, 968, 737, 697.

GC-MS m/z (%): 107 (08) [PhCH2O]+, 91 (100) [PhCH2]+, 75 (18) [CH3OCH2CH2OH]+, 65 (32).

Second and third steps: synthesis of 1-(benzyloxy)-2-butoxy-3-methoxypropane [1.4.0]: To a solution of [1.0.Bn] (5.30 g, 27.04 mmol) in DMF (50 mL) was added at 0°C sodium hydride (60% in mineral oil, 1.45 g,

37.85 mmol) by portions. After stirring at room temperature for 1 hour, bromobutane (4.1 mL, 37.84 mmol) was added. After 3 hours stirring, another equal portion of sodium hydride was added followed 30 minutes after by another equal portion of bromobutane. After stirring for 14 hours, the excess of sodium hydride was quenched at 0°C with water. The mixture was poured into water (250 mL) and extracted by ethyl acetate (3 x 100 mL). The combined organic fractions were washed with brine (3 x 50 mL), dried over magnesium sulfate and filtered. After evaporation of the solvents, the oil (6.50 g, maximum 25.79 mmol of [1.4.Bn]) was dissolved in ethanol (25 mL) and 10 wt% Pd/C (273 mg, 0.26 mmol, 1 mol%) was added. The flask was surrounded by a hydrogen balloon and the atmosphere was purged by vacuum and replaced by hydrogen. After stirring for 20h at room temperature, the solution was filtered over celite and the solvents were evaporated. The liquid was distilled under vacuum at 1 torr and the fraction between 65-68°C was collected to get [4.1.0] (2.89 g, 17.84 mmol, 66%).

1H NMR (CDCl3, 300 MHz): 3.75-3.46 (m, 7H), 3.37 (s, 3H), 2.16 (bs, 1H, OH), 1.63-1.53 (m, 2H), 1.44-1.32 (m, 2H), 0.92 (t, J = 6.7 Hz, 3H).

13C NMR (CDCl3, 75 MHz): 78.4 (d), 72.8 (t), 70.2 (t), 62.9 (t), 59.5 (q), 32.2 (t), 19.4 (t), 14.0 (q).

ATR-FTIR: νmax/cm-1 3419, 2931, 2873, 1457, 1194, 1090, 1047, 981 cm-1.

GC-MS m/z (%): 131 (35) [M-CH2OH]+, 117 (06) [M-CH2OMe]+, 100 (07) [M-CH2OMe-MeOH]+, 75 (51) [MeOCH2CH2OH]+, 61 (54) [HOCH2CH2OH]+, 57 (100) [n-Bu]+.

Synthesis of [x.1.1] products:

Synthesis of 1-ethoxy-2,3-dimethoxypropane [2.1.1]:

A solution of ethylglycidyl ether (9.5 g, 93.1 mmol) and sulfuric acid (0.1 g) in methanol (100 mL) was refluxed for 16 hours. After cooling to room temperature, potassium hydroxide (1 g) was added and the solvent was evaporated. To the residue was added more potassium hydroxide (6.26 g, 111.8 mmol, 1.2 eq.), tetrabutyl ammonium bromide (1.5 g, 4.66 mmol, 0.05 eq.) and dimethyl sulfate (9.7 mL, 102.4 mmol, 1.1 eq.). *caution: exotherme.* After stirring for one hour at room temperature, a second portion of potassium hydroxide was added to destroyed unreacted dimethyl sulfate. The mixture was partitioned between diethyl ether and brine, the organic layer was dried over magnesium sulfate, filtered and solvent were evaporated. The crude liquid was distilled using spinning band microdistillation under 40 torr and the fraction at 80-81°C was collected to give the product as a colorless fluid liquid (9.91 g, 67.0 mmol, 72%).

^{1}H NMR ($CDCl_3$, 300 MHz): 3.56-3.46 (m, 7H), 3.47 (s, 3H), 3.38 (s, 3H), 1.21 (t, *J* = 6.8 Hz, 3H).
^{13}C NMR ($CDCl_3$, 75 MHz): 79.3 (d), 72.5 (t), 70.2 (t), 66.9 (t), 59.3 (q), 58.0 (q), 15.2 (q)
ATR-FTIR (cm^{-1}): 2976, 2873, 1458, 1110.
GC-MS m/z (%): 103 (03) $[M - CH_2OMe]^+$, 89 (08) $[M – EtOCH_2]^+$, 75 (17), 72 (12), 59 (100) $[EtOCH_2]^+$, 58 (86).

Synthesis of 1,2-dimethoxy-3-propoxypropane [3.1.1]:

Solketal (20.0 g, 0.15 mol), *n*-propyl bromide (56,0 mL, 0,60 mol) and tetrabutyl ammonium bromide (9.8 g, 0.03 mol) were added to 400 mL of a 33% aqueous solution of potassium hydroxide (132.0 g KOH, 2.35 mol). After stirring for 24 hours at reflux, the reaction mixture was cooled and extracted with methylene chloride (4 x 50 mL). The organic phase was washed with water (2 x 20 mL) and dried over sodium sulfate. After filtration and evaporation of the solvents, the resulting oil filtered over a short pad of silica gel. Propyl isopropylidene ether was recovered as a colorless liquid (24,4 g, 91%). The isopropylidene protecting group was then removed adding methanol (90 mL) and HCl (1 M, 60 mL). After stirring 4 hours at room temperature, the solution was neutralized by a few drops of concentrated NaOH and the medium was evaporated until the formation of few NaCl crystals. Few drops of water were then added to dissolve

again crystals and the reaction mixture was extracted with ethyl acetate (4 x 50 mL). The combined organic phases were dried over sodium sulfate, filtered and solvents were evaporated to give a colorless liquid (19.8 g) also used without further purifications. Potassium hydroxide (20.8 g, 0.37 mol) and tetrabutyl ammonium hydrogen sulfate (5.0 g, 14 mmol) were successively introduced into the product dissolved in THF (100 mL) in small portions while stirring. After one hour stirring at room temperature, the mixture was cooled using an ice bath and dimethyl sulfate (30.9 g, 0.25 mol) was added dropwise. After stirring for 24 hours, pentane was added to the crude mixture. Salts (Na_2SO_4) were removed by filtration and washed several time with pentane. The liquid fraction was then concentrated and finally distilled under reduced pressure to get a colorless fluid liquid (13.1 g, 89.9 mmol, 54%).

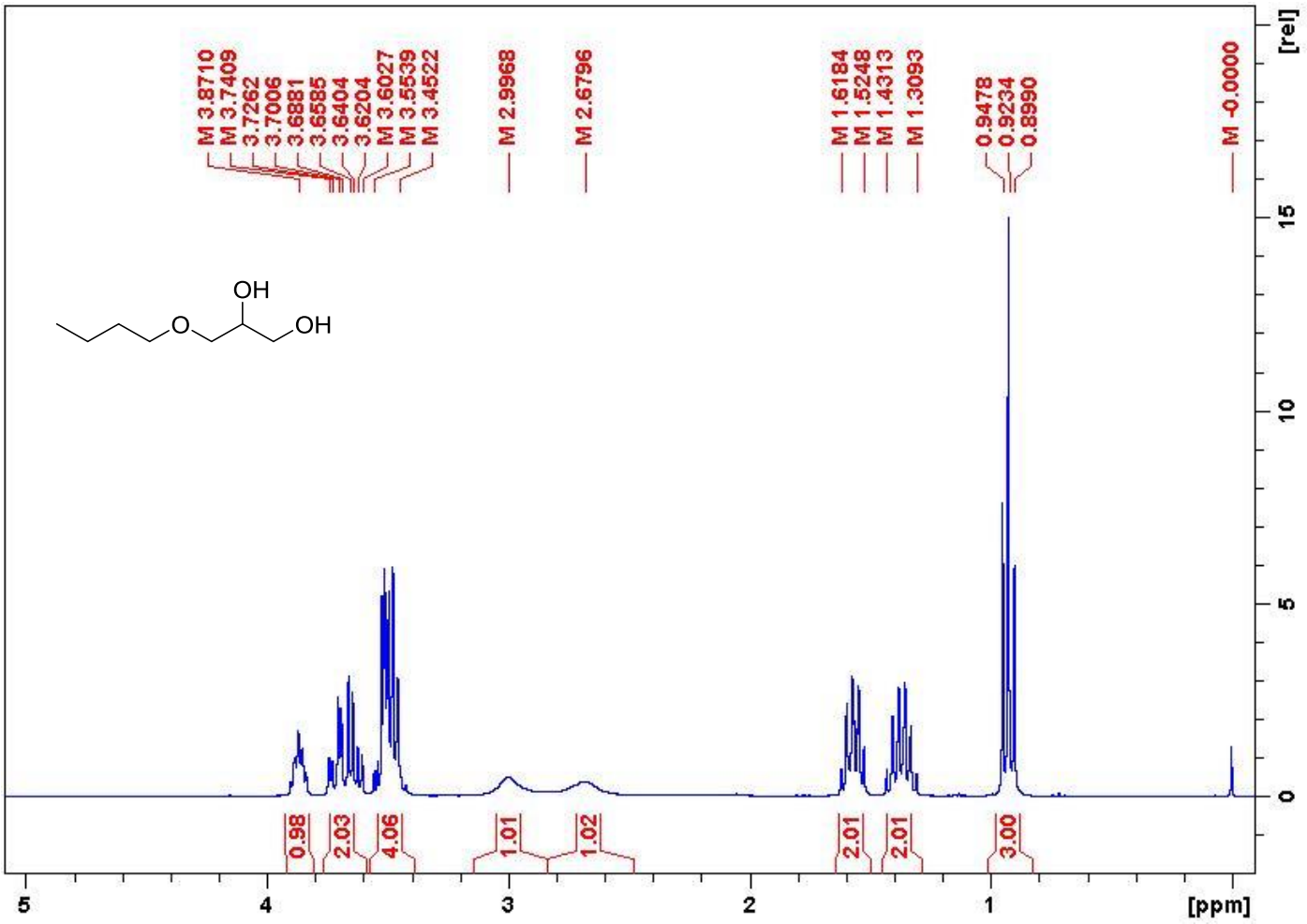
^{1}H NMR ($CDCl_3$, 300 MHz): 0.91 (t, 3H, CH_3), 1.58 (m, 2H, CH_2), 3.37 (s, 3H, CH_3), 3.47 (m, 10H, CH_3, 3 x CH_2, CH).
^{13}C NMR ($CDCl_3$, 75 MHz): 10.44 (CH_3), 22.74 (CH_2), 57.83 (CH_2), 59.14 (CH2), 70.08 (CH_2), 72.41 (CH_2), 73.17 (CH_2), 79.21 (CH).
ATR-FTIR (cm^{-1}): 2876, 1461, 1195, 1109, 955.
GC-MS m/z (%): 130 (03) $[M\text{-}MeOH]^+$, 89 (25) $[M - n\text{-}PrOCH_2]^+$, 75 (51), 72 (26), 59 (55) $[n\text{-}PrO]^+$, 58 (100).
HRMS-ESI: m/z $[MNa]^+$ calcd for $C_8H_{18}NaO_3$: 185.1144 found: 185.1141.

Synthesis of 1-butoxy-(2,3)-dimethoxypropane [4.1.1]:
1-butoxypropane-2,3-diol [4.0.0] (19.2 g, 0.13 mol), potassium hydroxide (18.2 g, 0.32 mol) and tetrabutyl ammonium hydrogen sulfate (4.6 g, 13 mmol) were successively introduced in small portions while stirring in a 500 mL flask containing 100 mL of THF. After one hour stirring at room temperature, the mixture was cooled using an ice bath and dimethyl sulfate (28.7 g, 0.22 mol) was added dropwise. caution: exotherme. After stirring for 24 hours at room temperature, pentane was added to the crude mixture. Salts (Na_2SO_4) were removed by filtration and washed several time with pentane. The liquid fraction was then concentrated and finally distilled under reduced pressure to get a colorless fluid liquid (7.7 g, 43.8 mmol, 34%).
^{1}H NMR ($CDCl_3$, 300 MHz): 0.91 (t, 3H, CH_3), 1.36 (m, 2H, CH_2), 1.56 (m, 2H), 3.37 (s, 3H, CH_3), 3.46 (m, 10H, CH_3, 3 x CH_2, CH).
^{13}C NMR ($CDCl_3$, 75 MHz): 13.54 (CH_3), 18.98 (CH_2), 31.43 (CH_2), 57.52 (CH_2), 58.80 (CH_2), 69.92 (CH_2), 71.00 (CH_2), 72.17 (CH_2), 78.99 (CH).
ATR-FTIR (cm^{-1}): 2931, 2872, 1459, 1195, 1110, 960, 840, 734.
GC-MS m/z (%): 144 (03) $[M\text{-}MeOH]^+$, 89 (28) $[M - n\text{-}BuOCH_2]^+$, 75 (48), 59 (55), 58 (100), 57 (57).
HRMS-ESI: m/z $[MNa]^+$ calcd for $C_9H_{20}NaO_3$: 199.1300 found: 199.1302.

Synthesis of [2.2.2]:

Synthesis of 1,2,3-triethoxypropane [2.2.2]:
Glycerol (14.0 g, 0.15 mol), potassium hydroxide (28.3 g, 0.50 mol) and tetrabutyl ammonium hydrogen sulfate (5.2 g, 15 mmol) were successively introduced in small portions while stirring in a 500 mL flask containing 100 mL of THF. After one hour stirring at room temperature, the mixture was cooled using an ice bath water then diethyl sulfate (49 mL, 0.37 mol) was added dropwise. *caution: exotherme.* After stirring for 24 hours, ethanol (28.0 mL, 0.50 mol) and potassium hydroxide (13.3 g, 0.24 mol) were added to the mixture to destroy unreacted diethyl sulfate. After stirring again for 24 hours, pentane was added to the crude mixture. Salts (Na_2SO_4) were removed by filtration and washed several time with pentane. The liquid fraction was then concentrated and finally distilled under reduced pressure to get a colorless fluid liquid (4.7 g, 26.7 mmol, 18%).

^{1}H NMR ($CDCl_3$, 300 MHz): 1.19 (m, 9H, 3 x CH_3), 3.6 (m, 11H, 5 x CH_2, CH).
^{13}C NMR: 15.02 (2C, CH_3), 15.67 (CH_3), 65.72 (CH_2), 66.85 (2C, 2 x CH_2), 70.63 (2C, 2 x CH_2), 77.76 (CH).
ATR-FTIR (cm^{-1}): 2975, 2866, 1379, 1109.
GC-MS m/z (%): 130 (02) $[M\text{-}EtOH]^{+\circ}$, 117 (06) $[M - CH_2OEt]^{+}$, 89 (12), 85 (10), 73 (20), 61 (57), 59 (100) $[EtOCH_2]^{+}$.
HRMS-ESI: m/z $[MNa]^{+}$ calcd for $C_9H_{20}NaO_3$: 199.1300 found: 199.1299.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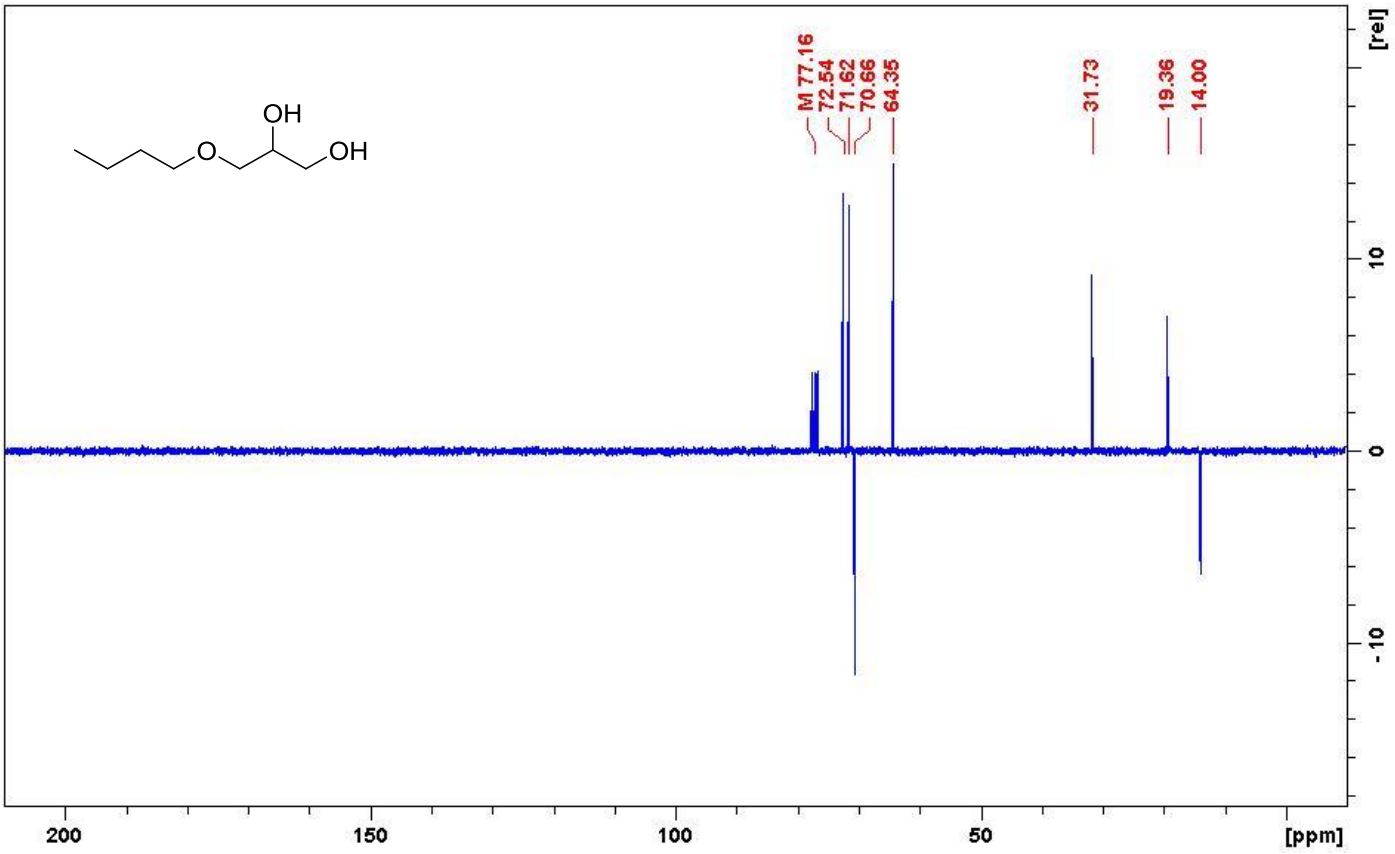
3.7262
3.7006
3.6881
3.6585
3.6404
3.6204
M 3.6027
M 3.5539
M 3.4522
M 2.9968
M 2.6796
M 1.6184
M 1.5248
M 1.4313
M 1.3093
0.9478
0.9234
0.8990
M -0.0000
OH
O
OH
0.98
2.03
4.06
1.01
1.02
2.01
2.01
3.00
[rel]
[ppm]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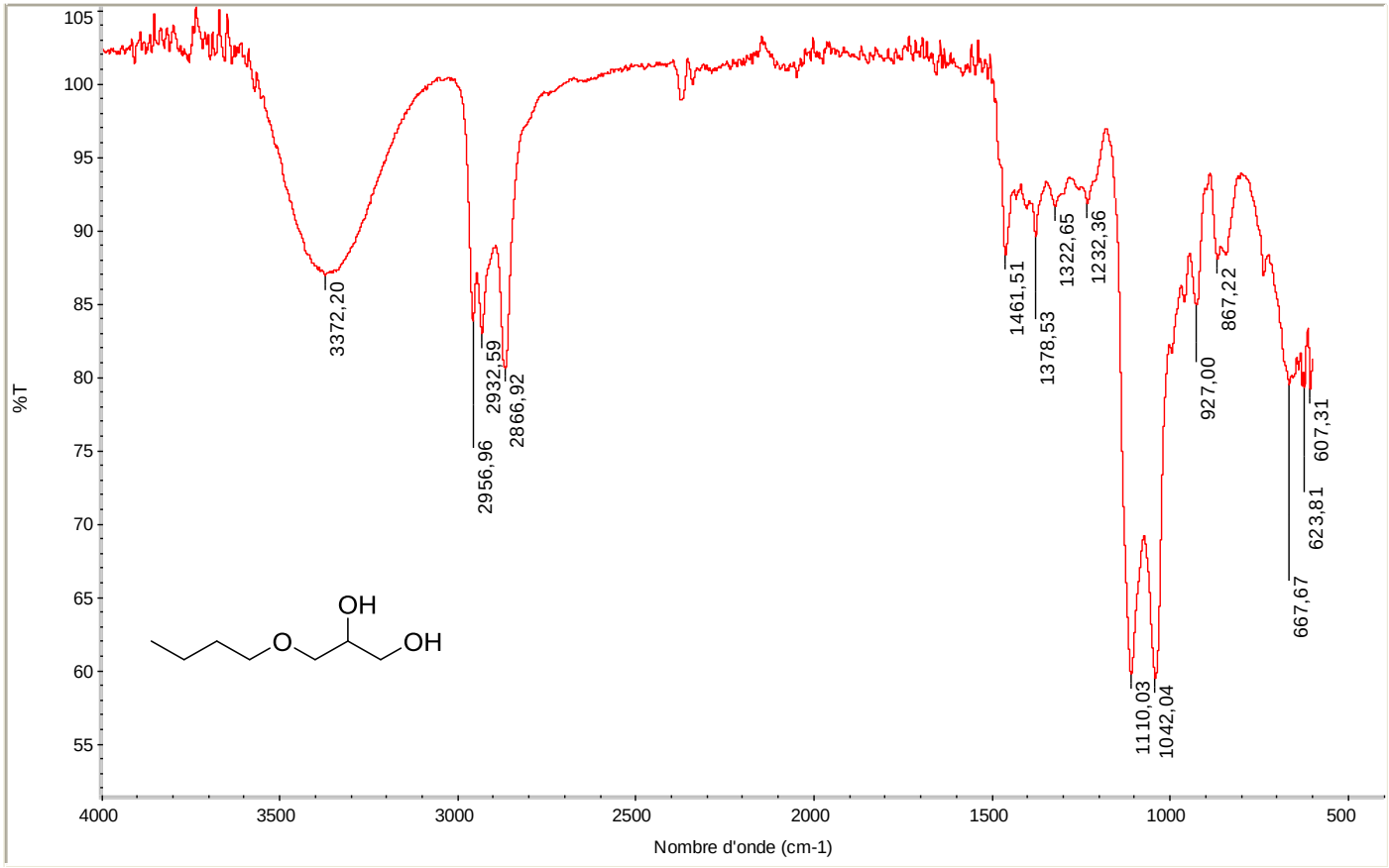
71.62
70.66
64.35
31.73
19.36
14.00
OH
O
OH
[rel]
[ppm]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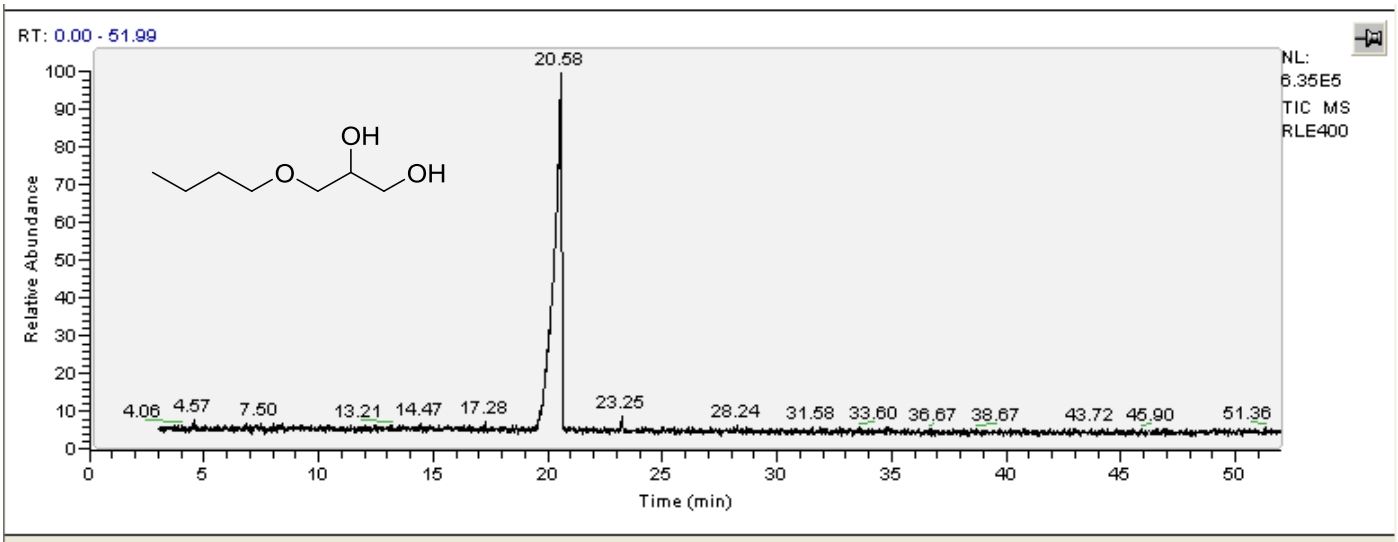
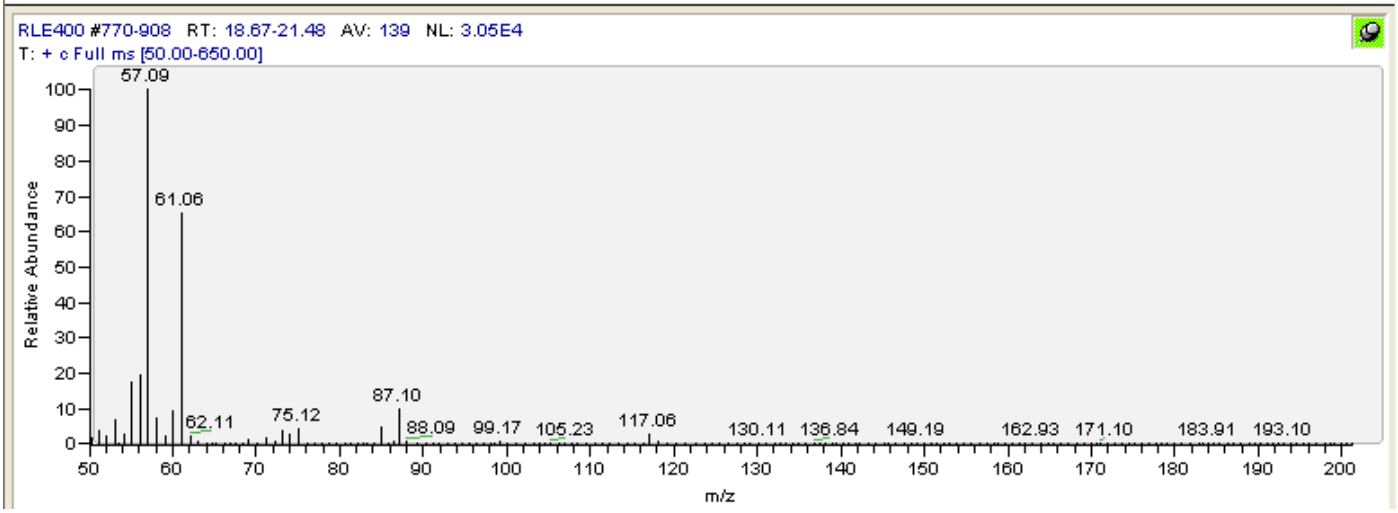
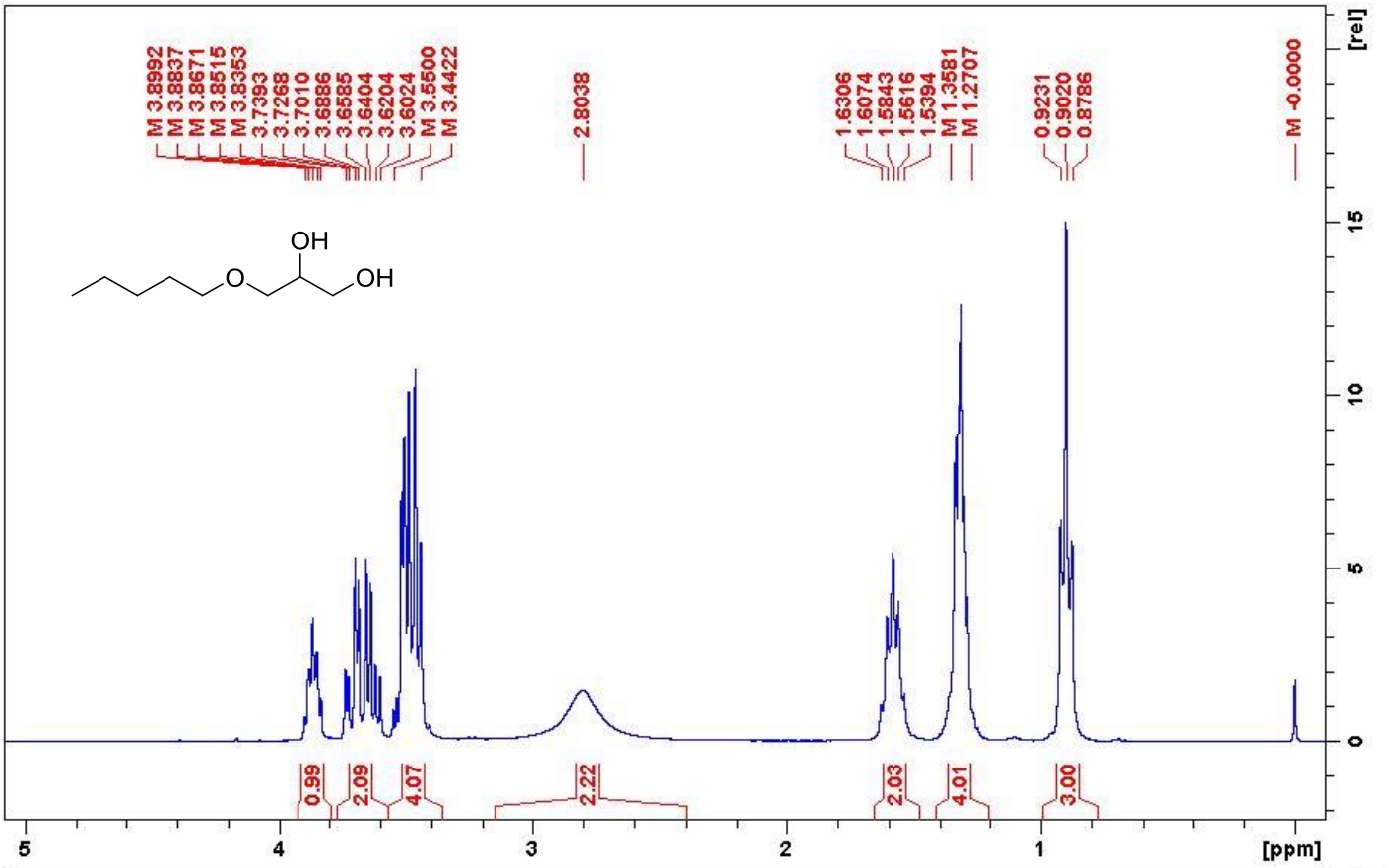
3372,20
2956,96
2932,59
2866,92
1461,51
1378,53
1322,65
1232,36
1110,03
1042,04
927,00
867,22
667,67
623,81
607,31
OH
O
OH


RT: 0.00 - 51.99
Relative Abundance
Time (min)
NL:
6.35E5
TIC MS
RLE400
20.58
4.06
4.57
7.50
13.21
14.47
17.28
23.25
28.24
31.58
33.60
36.67
38.67
43.72
45.90
51.36
OH
O
OH


RLE400 #770-908 RT: 18.67-21.48 AV: 139 NL: 3.05E4
T: + c Full ms [50.00-650.00]
Relative Abundance
m/z
57.09
61.06
87.10
62.11
75.12
88.09
99.17
105.23
117.06
130.11
136.84
149.19
162.93
171.10
183.91
193.10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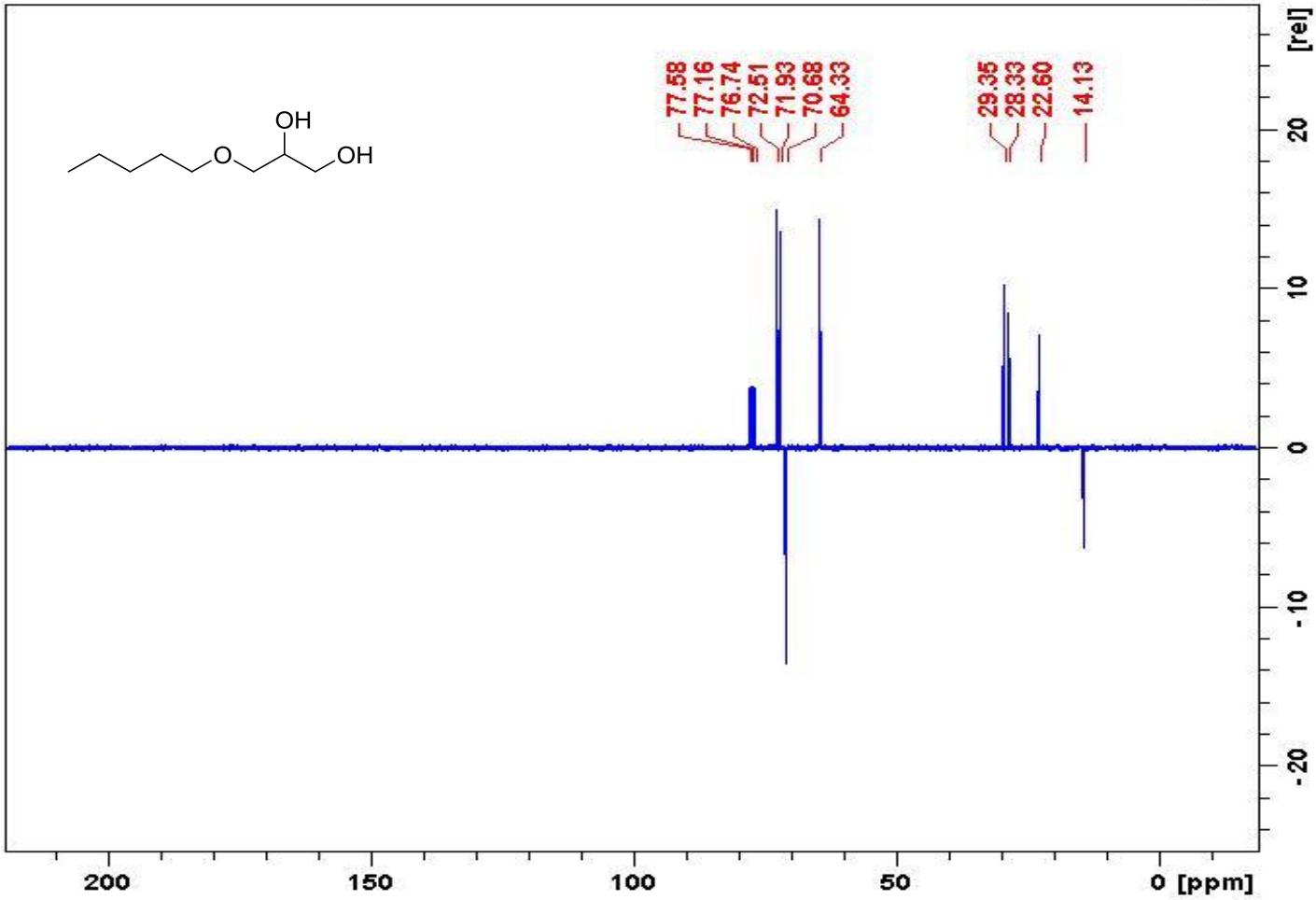
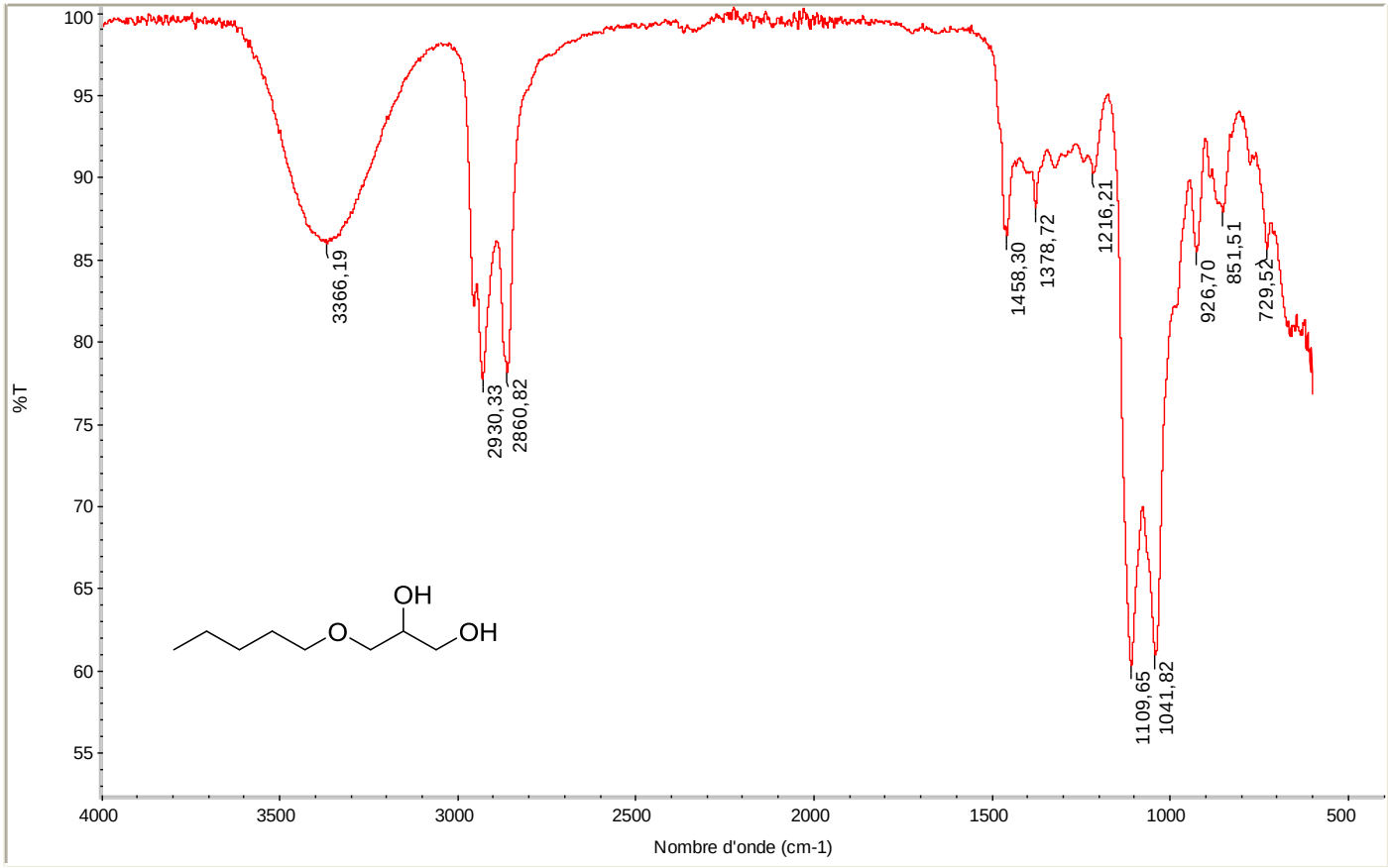
M 3.8837
M 3.8671
M 3.8515
M 3.8353
3.7393
3.7268
3.7010
3.6886
3.6585
3.6404
3.6204
3.6024
M 3.5500
M 3.4422
2.8038
1.6306
1.6074
1.5843
1.5616
1.5394
M 1.3581
M 1.2707
0.9231
0.9020
0.8786
M -0.0000
OH
O
OH
0.99
2.09
4.07
2.22
2.03
4.01
3.00
[rel]
[ppm]


77.58
77.16
76.74
72.51
71.93
70.68
64.33
29.35
28.33
22.60
14.13
OH
O
OH
[rel]
0 [ppm]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Nombre d'onde (cm-1)
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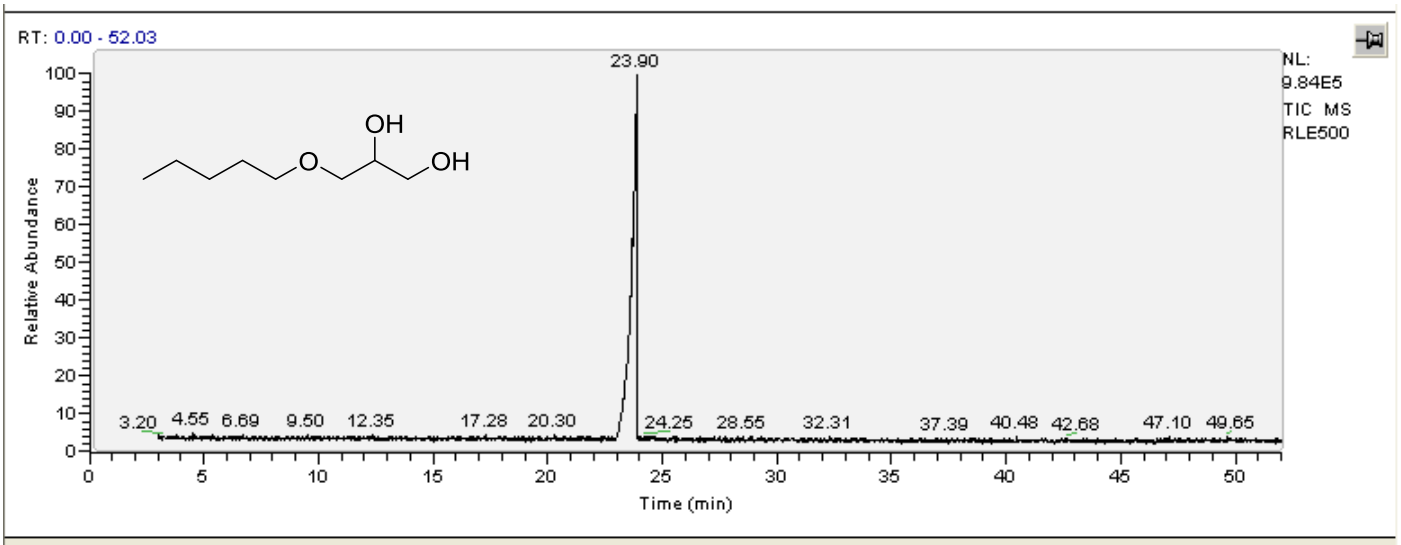
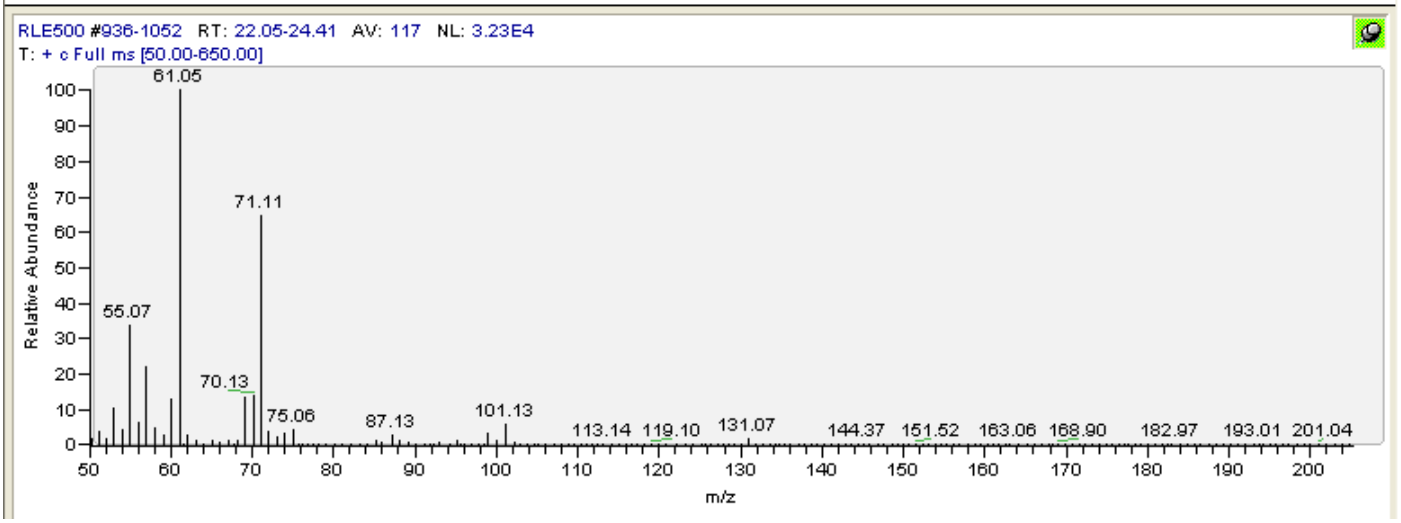
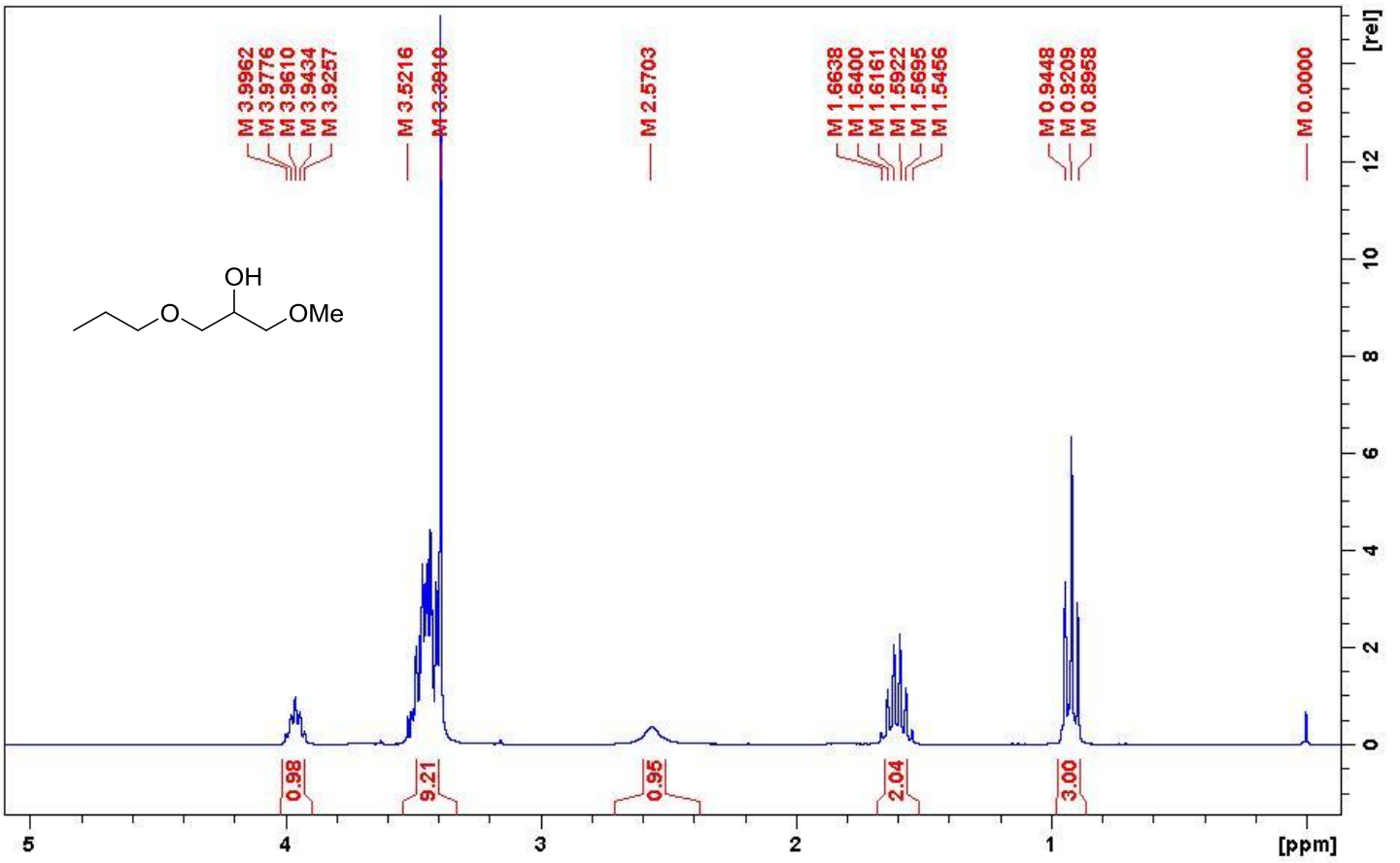
2930,33
2860,82
1458,30
1378,72
1216,21
1109,65
1041,82
926,70
851,51
729,52
OH
O
OH


RT: 0.00 - 52.03
NL:
9.84E5
TIC MS
RLE500
Relative Abundance
Time (min)
23.90
3.20
4.55
6.69
9.50
12.35
17.28
20.30
24.25
28.55
32.31
37.39
40.48
42.68
47.10
49.65
OH
O
OH


RLE500 #936-1052 RT: 22.05-24.41 AV: 117 NL: 3.23E4
T: + c Full ms [50.00-650.00]
Relative Abundance
m/z
61.05
71.11
55.07
70.13
75.06
87.13
101.13
113.14
119.10
131.07
144.37
151.52
163.06
168.90
182.97
193.01
201.04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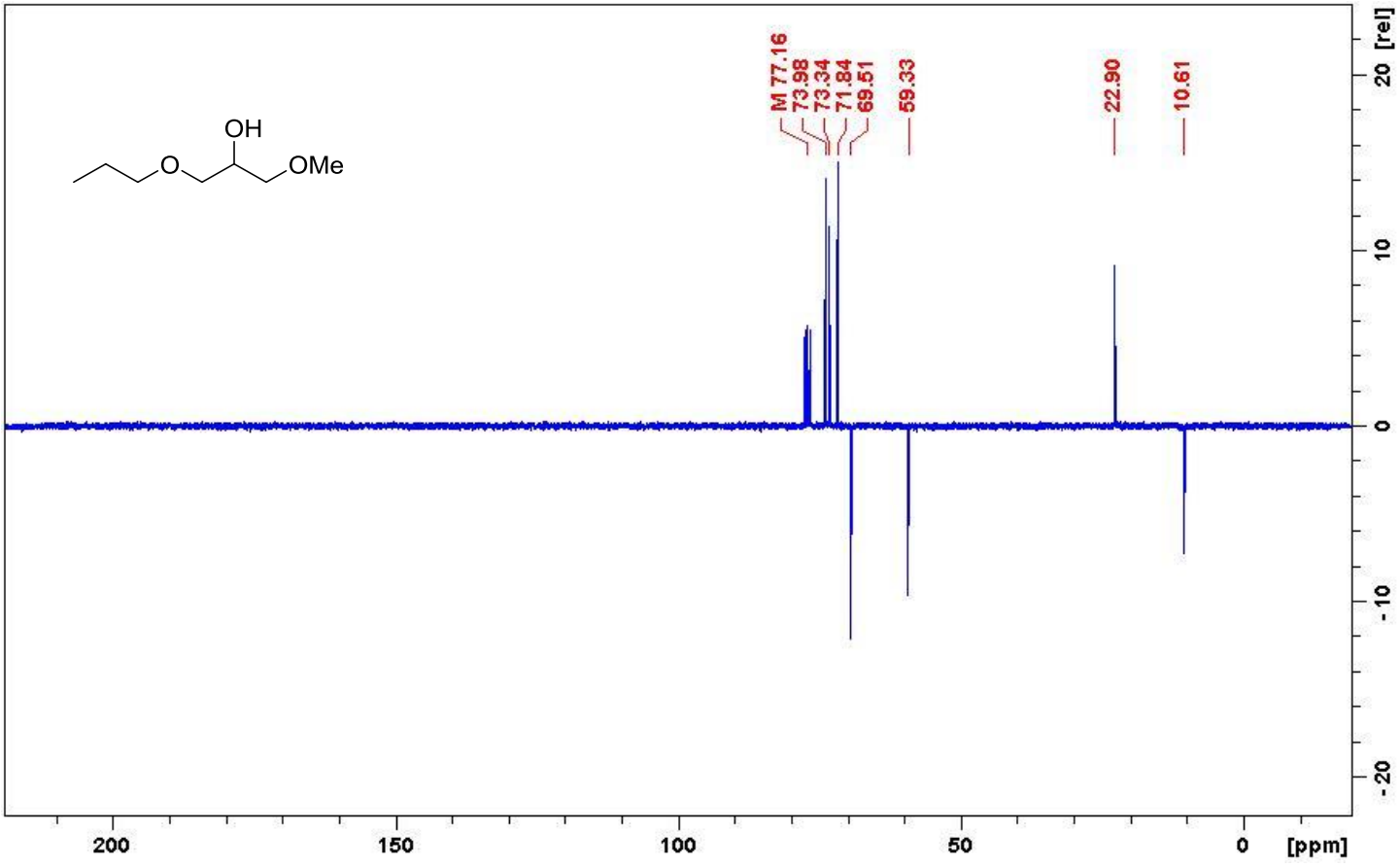
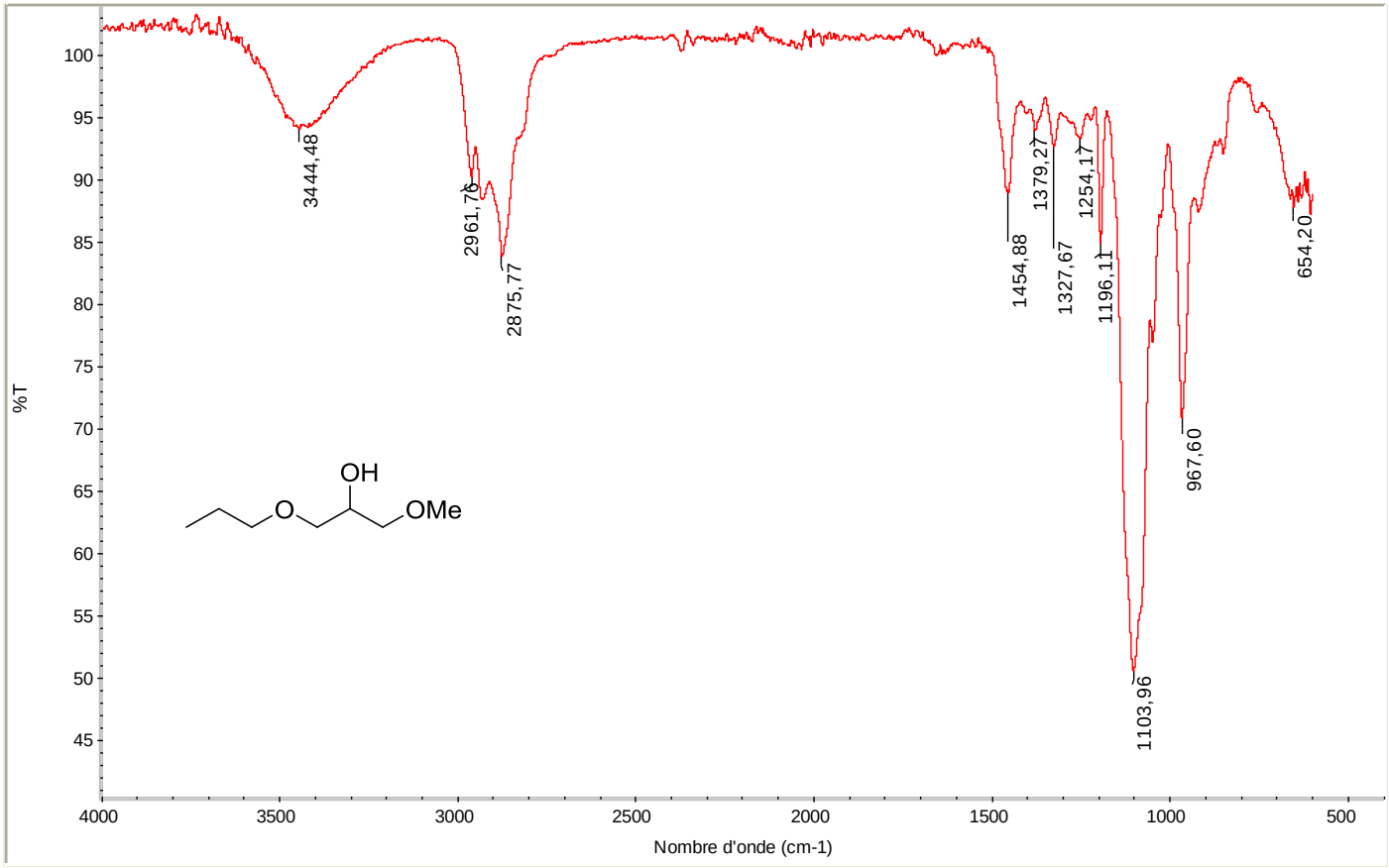
M 3.9776
M 3.9610
M 3.9434
M 3.9257
M 3.5216
M 3.3910
M 2.5703
M 1.6638
M 1.6400
M 1.6161
M 1.5922
M 1.5695
M 1.5456
M 0.9448
M 0.9209
M 0.8958
M 0.0000
OH
O
OMe
0.98
9.21
0.95
2.04
3.00
[rel]
[ppm]


M 77.16
73.98
73.34
71.84
69.51
59.33
22.90
10.61
OH
O
OMe
[rel]
[ppm]


%T
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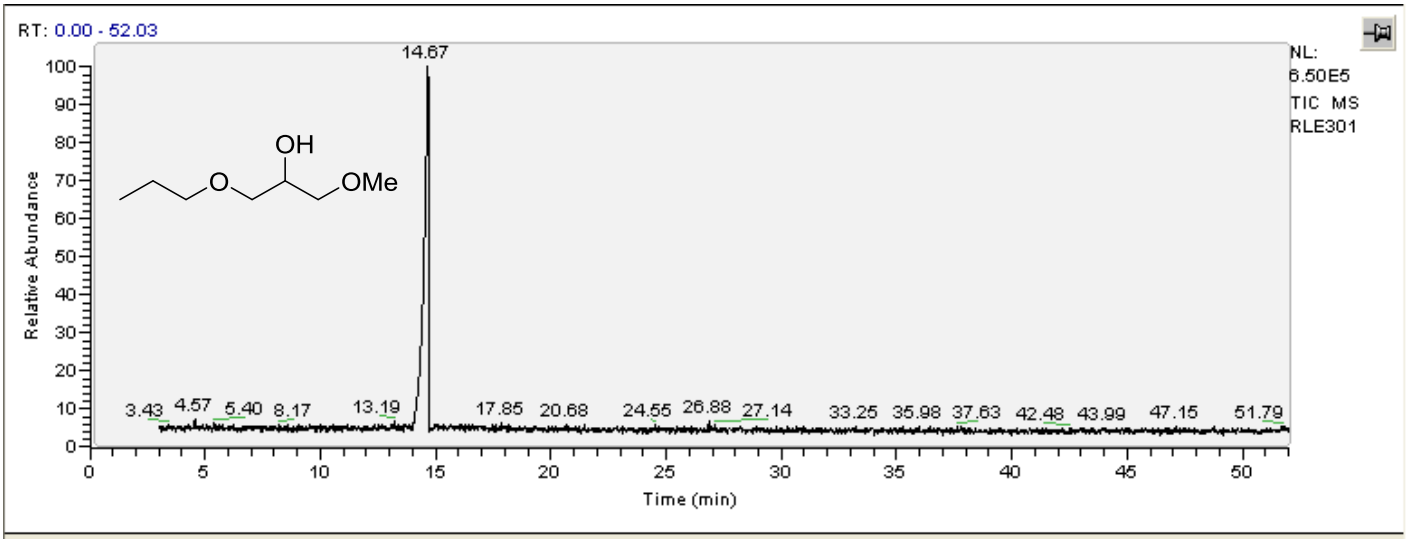
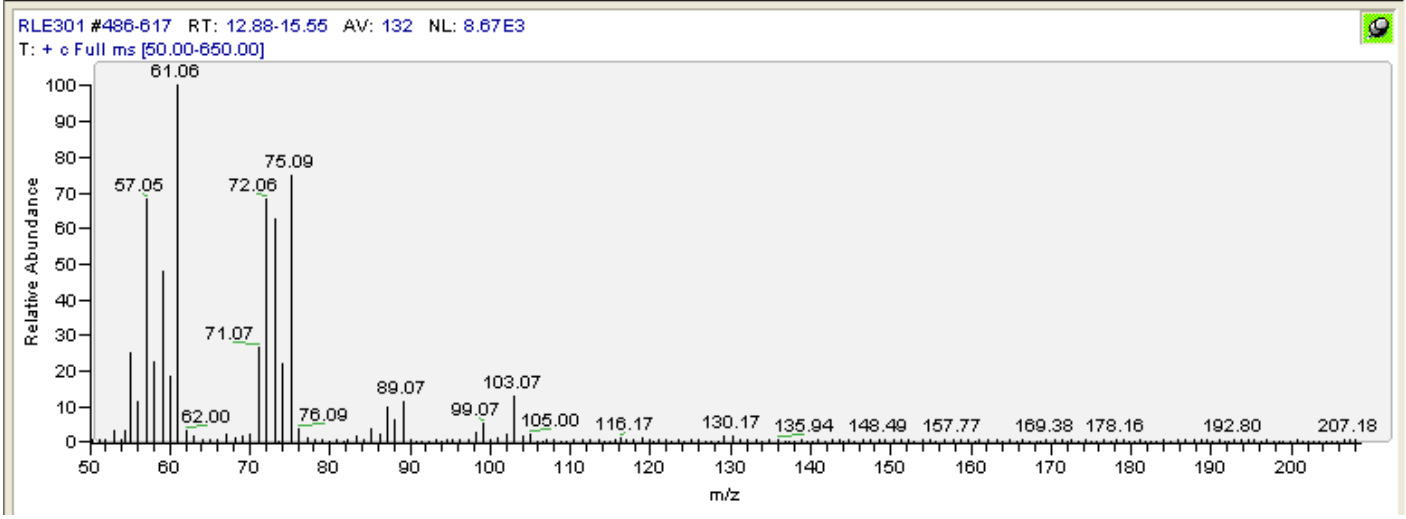
3444,48
2961,76
2875,77
1454,88
1379,27
1327,67
1254,17
1196,11
1103,96
967,60
654,20
OH
O
OMe


RT: 0.00 - 52.03
NL:
6.50E5
TIC MS
RLE301
Relative Abundance
Time (min)
14.67
OH
O
OMe


RLE301 #486-617 RT: 12.88-15.55 AV: 132 NL: 8.67E3
T: + c Full ms [50.00-650.00]
Relative Abundance
m/z
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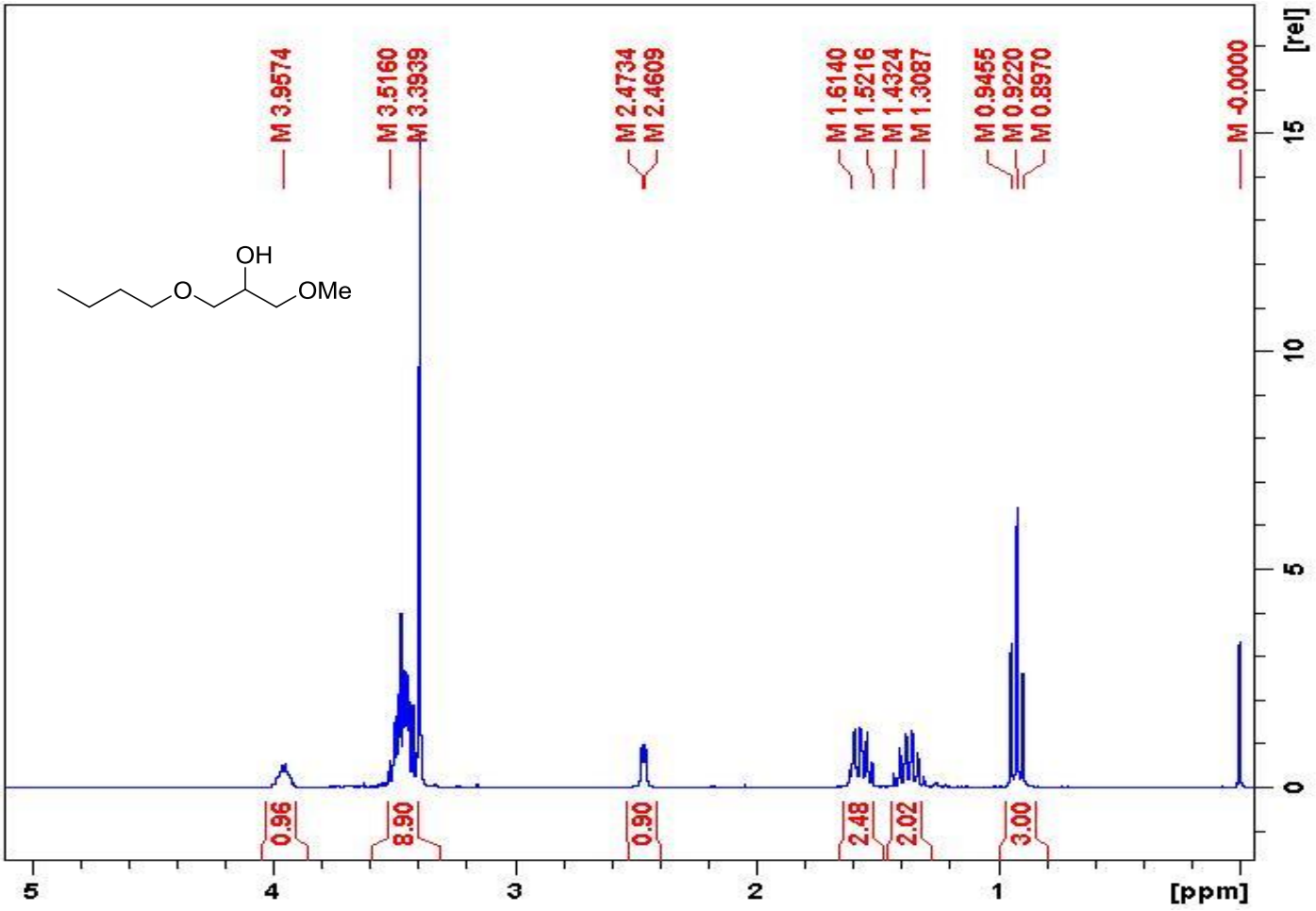
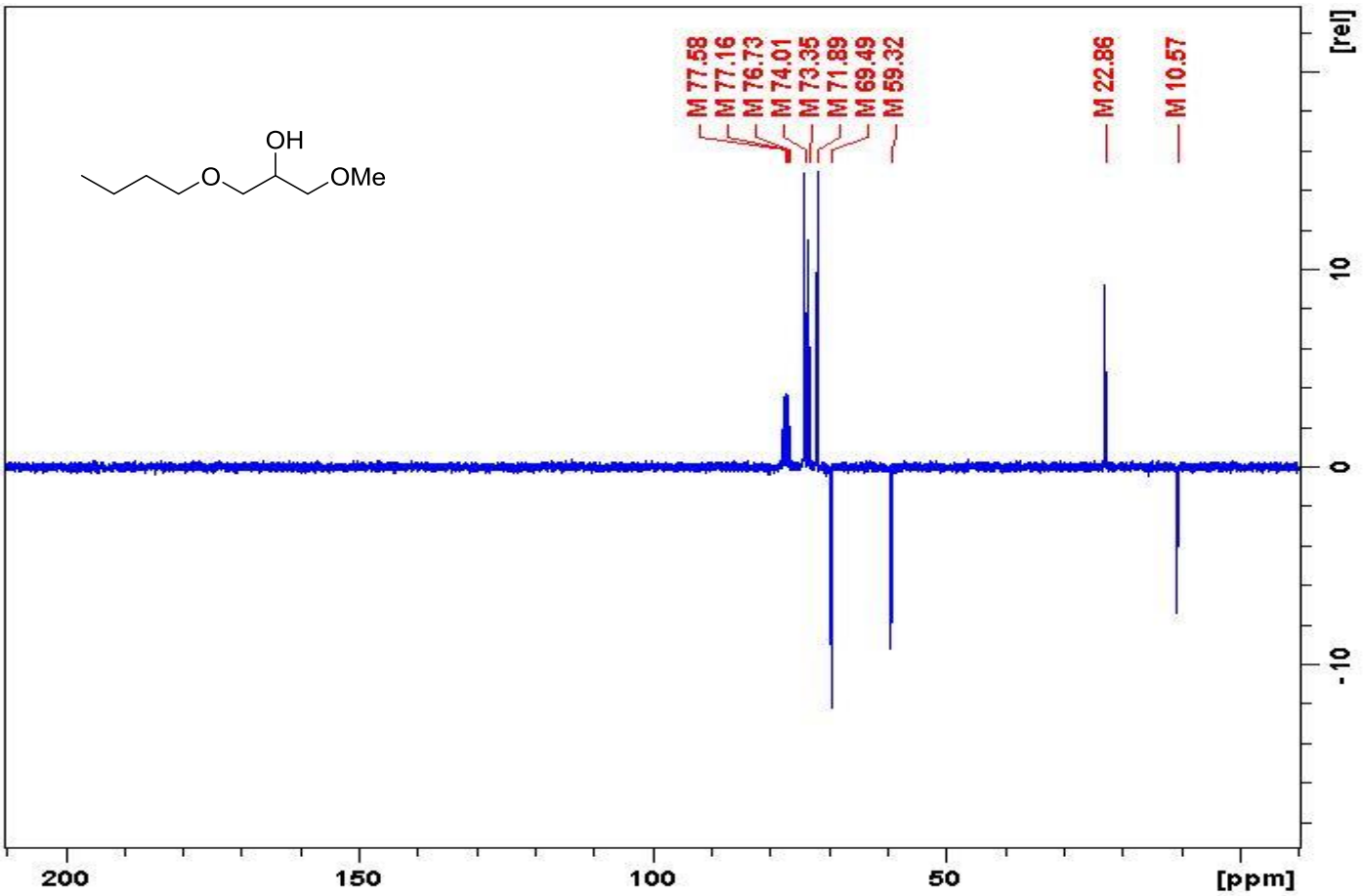
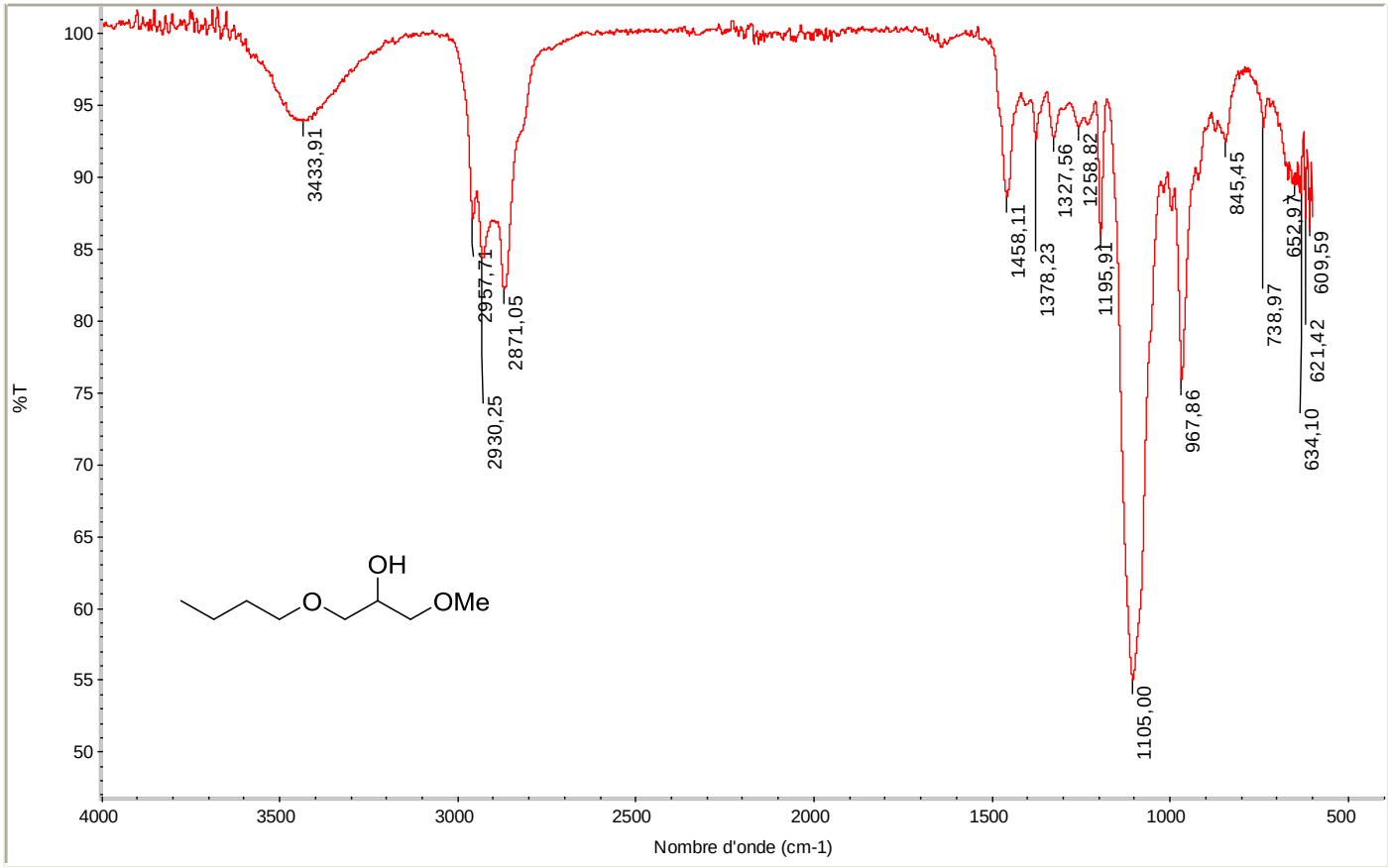
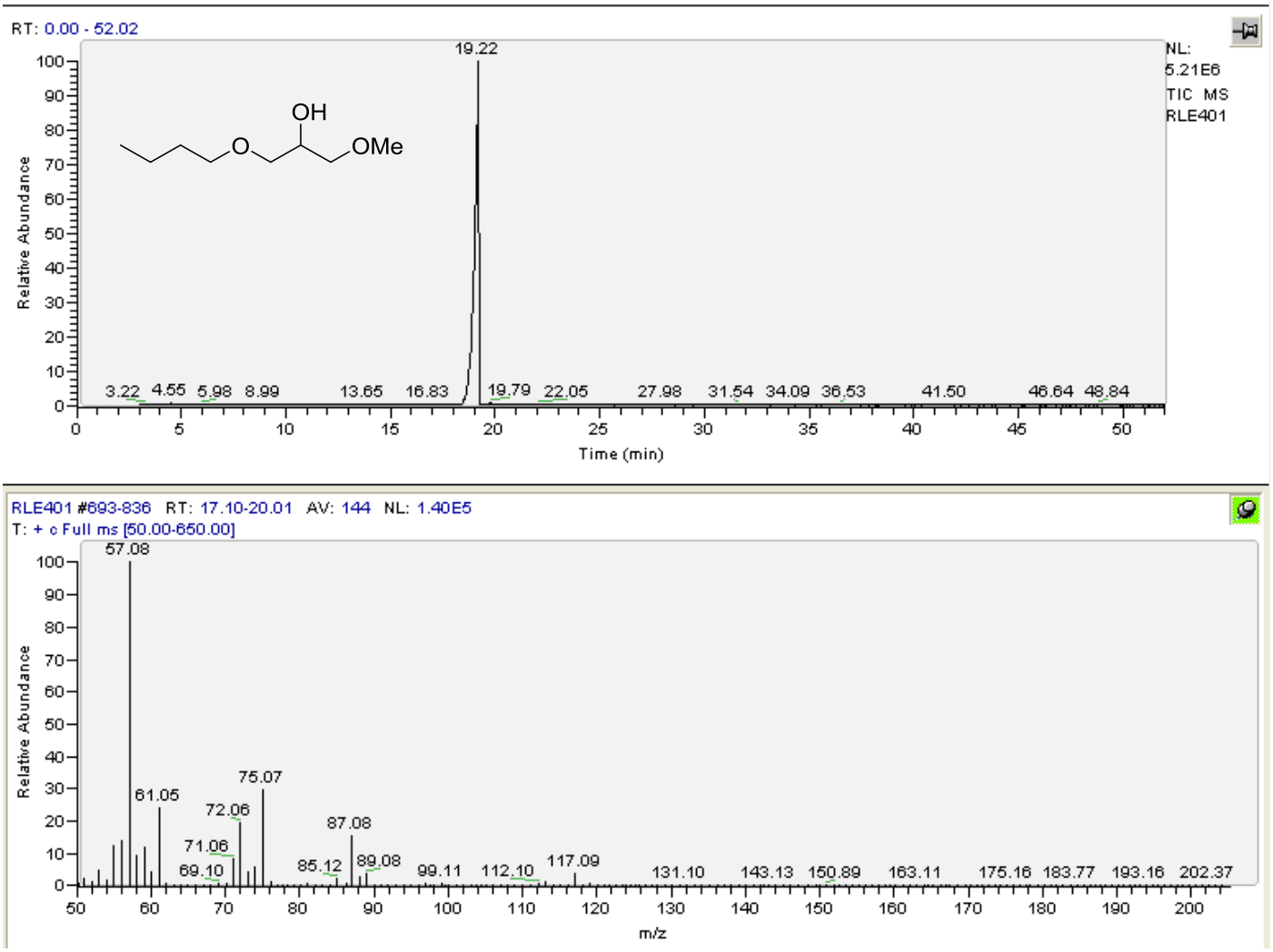
57.05
72.06
75.09
71.07
89.07
103.07
99.07
62.00
76.09
105.00

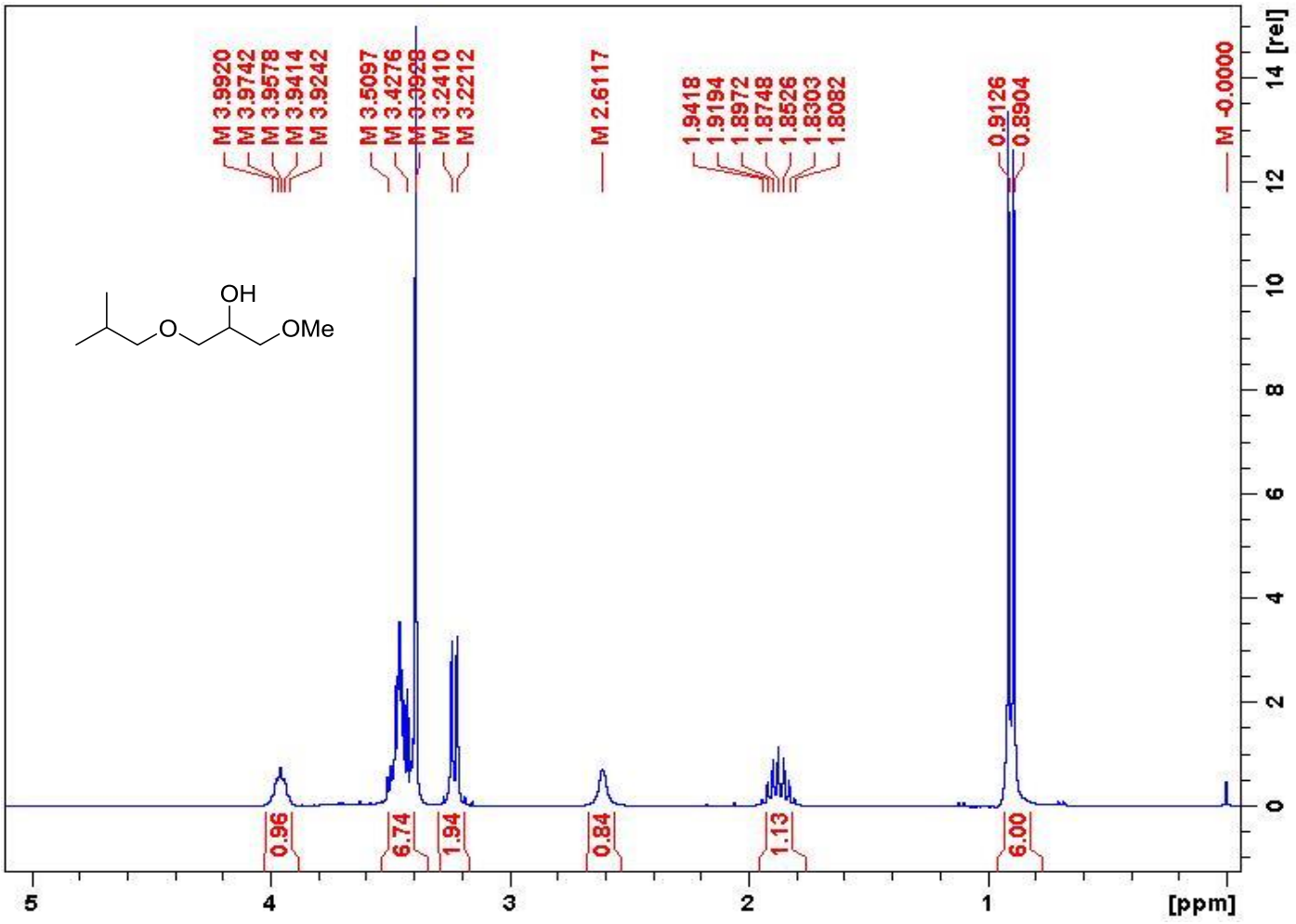

M 3.9574
M 3.5160
M 3.3939
M 2.4734
M 2.4609
M 1.6140
M 1.5216
M 1.4324
M 1.3087
M 0.9455
M 0.9220
M 0.8970
M -0.0000
OH
O
OMe
0.96
8.90
0.90
2.48
2.02
3.00
[rel]
[ppm]


M 77.58
M 77.16
M 76.73
M 74.01
M 73.35
M 71.89
M 69.49
M 59.32
M 22.86
M 10.57
OH
O
OMe
[rel]
[ppm]


%T
Nombre d'onde (cm-1)
3433,91
2957,71
2930,25
2871,05
1458,11
1378,23
1327,56
1258,82
1195,91
1105,00
967,86
845,45
738,97
652,97
634,10
621,42
609,59
OH
O
OMe


RT: 0.00 - 52.02
NL:
5.21E6
TIC MS
RLE401
Relative Abundance
Time (min)
19.22
3.22 4.55 5.98 8.99 13.65 16.83 19.79 22.05 27.98 31.54 34.09 36.53 41.50 46.64 48.84
OH
O
OMe

RLE401 #693-836 RT: 17.10-20.01 AV: 144 NL: 1.40E5
T: + c Full ms [50.00-650.00]
Relative Abundance
m/z
57.08
61.05
75.07
72.06
87.08
71.06
69.10
85.12
89.08
99.11
112.10
117.09
131.10
143.13
150.89
163.11
175.16
183.77
193.16
202.37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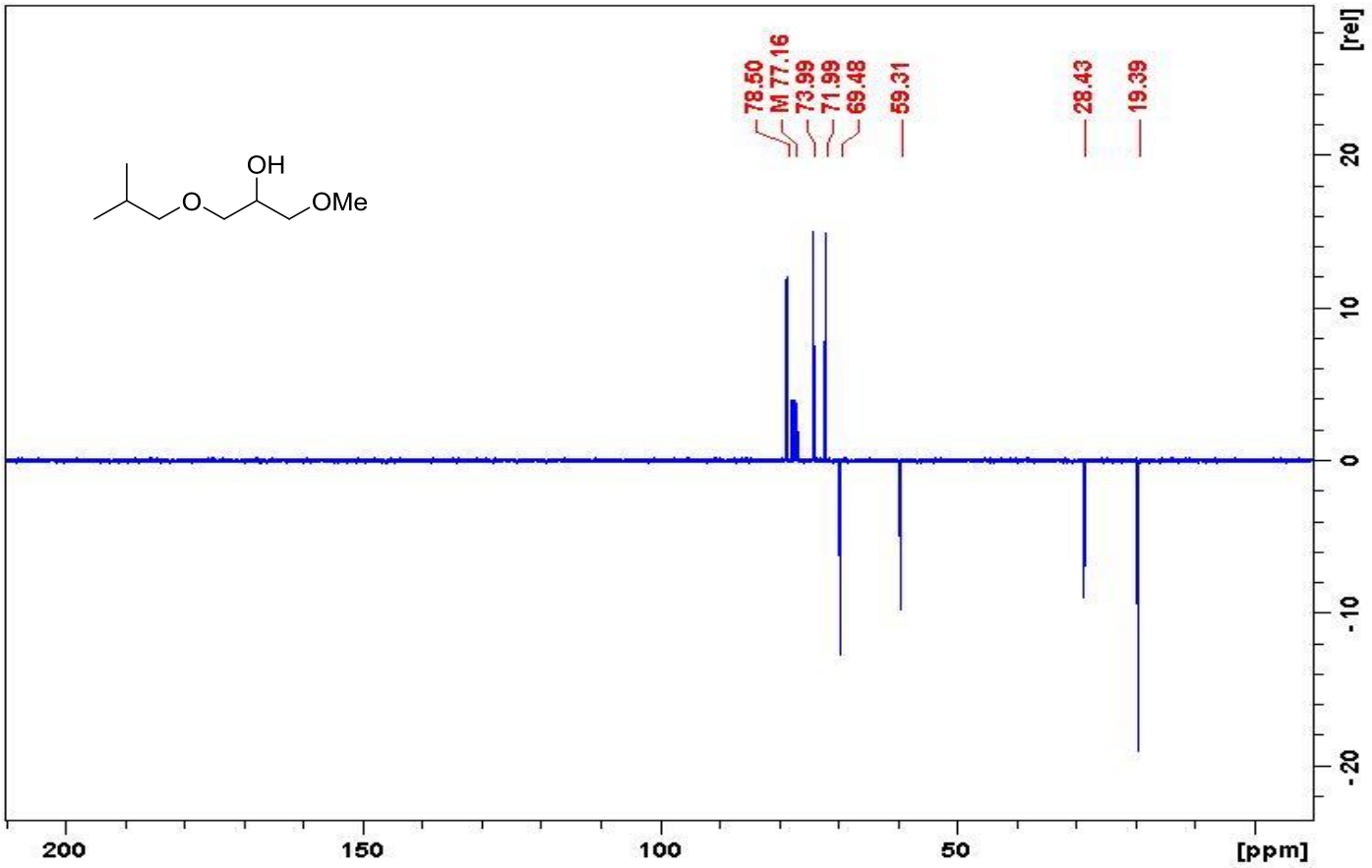
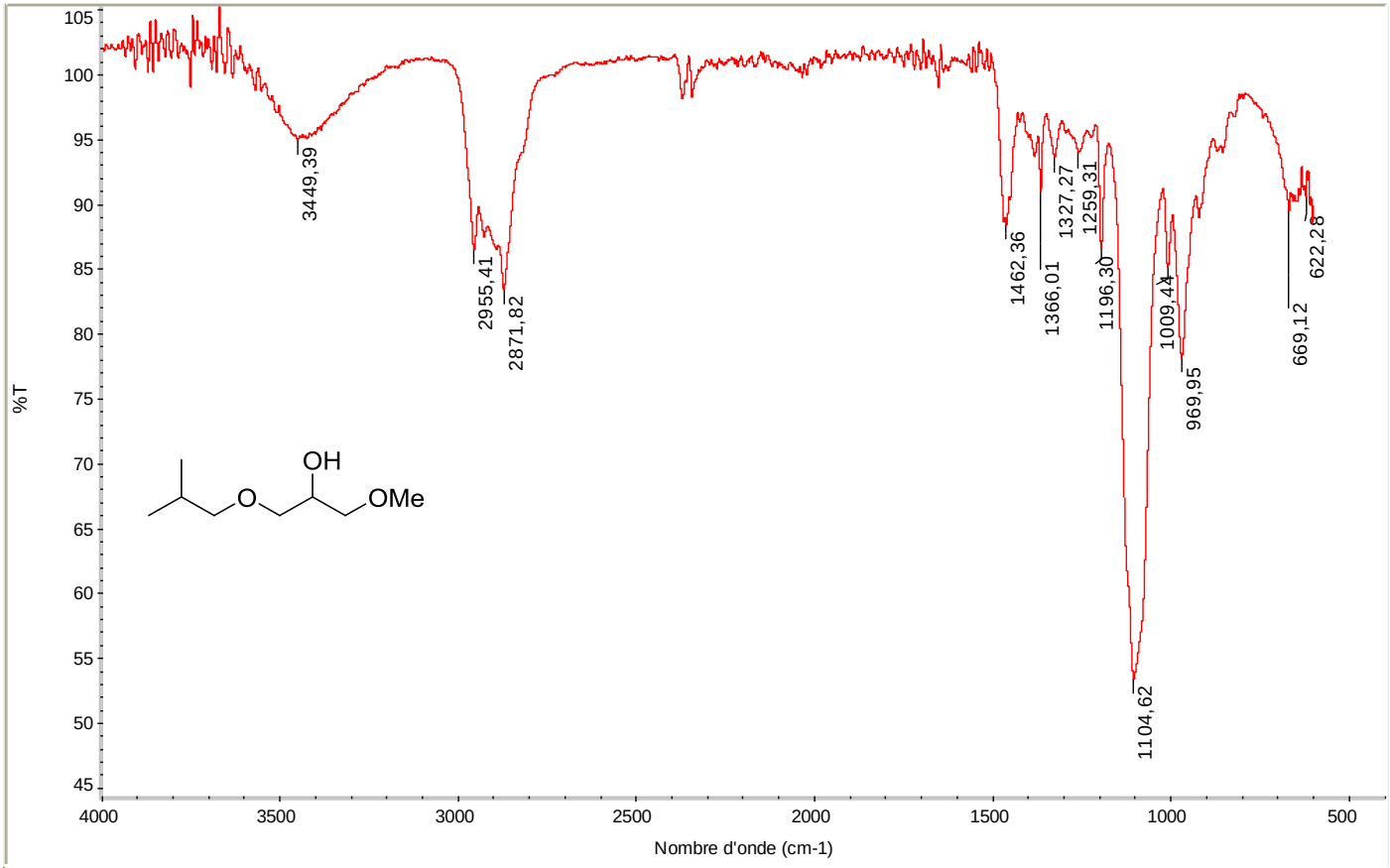
M 3.9742
M 3.9578
M 3.9414
M 3.9242
M 3.5097
M 3.4276
M 3.3928
M 3.2410
M 3.2212
M 2.6117
1.9418
1.9194
1.8972
1.8748
1.8526
1.8303
1.8082
0.9126
0.8904
M -0.0000
OH
O
OMe
0.96
6.74
1.94
0.84
1.13
6.00
[rel]
[ppm]


78.50
M 77.16
73.99
71.99
69.48
59.31
28.43
19.39
OH
O
OMe
[rel]
[ppm]


%T
Nombre d'onde (cm-1)
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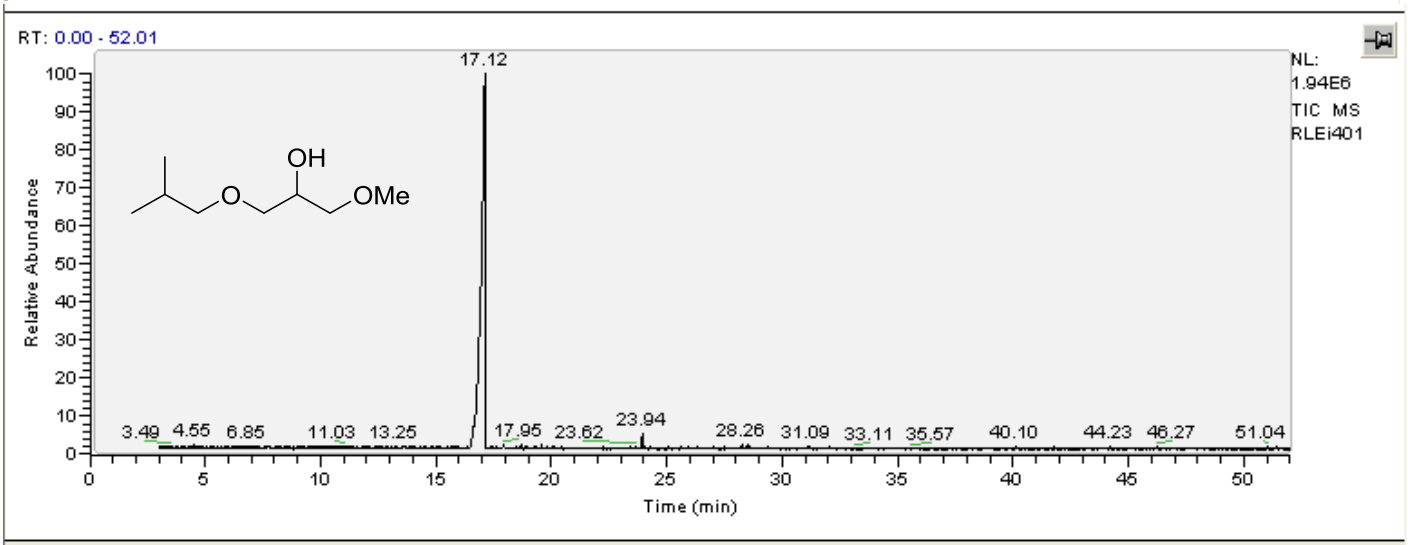
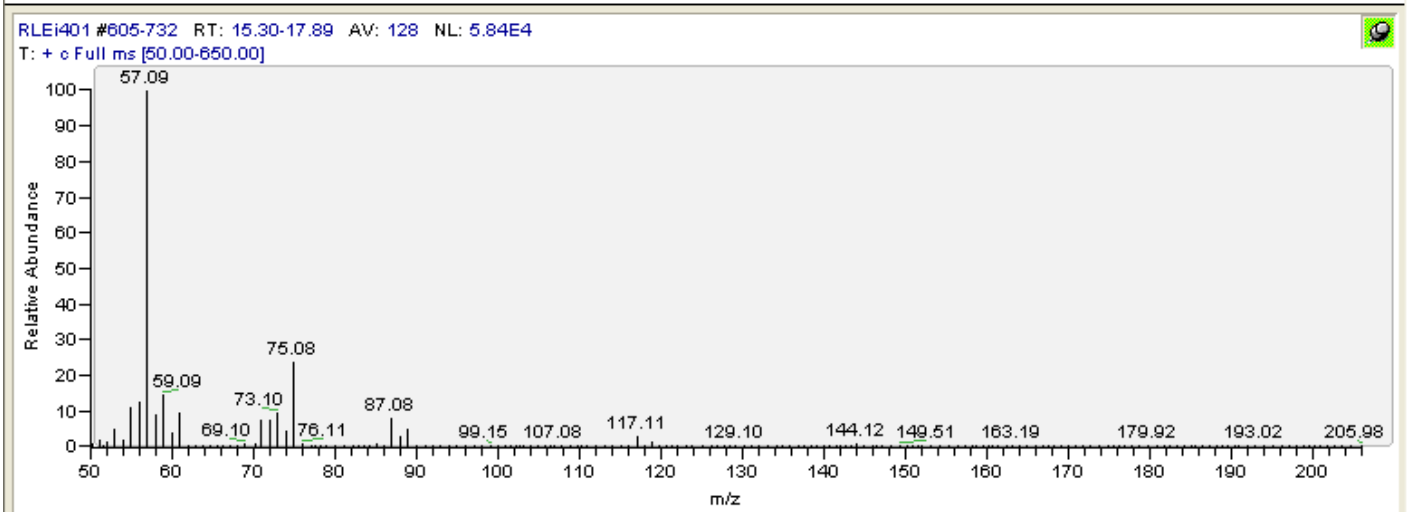
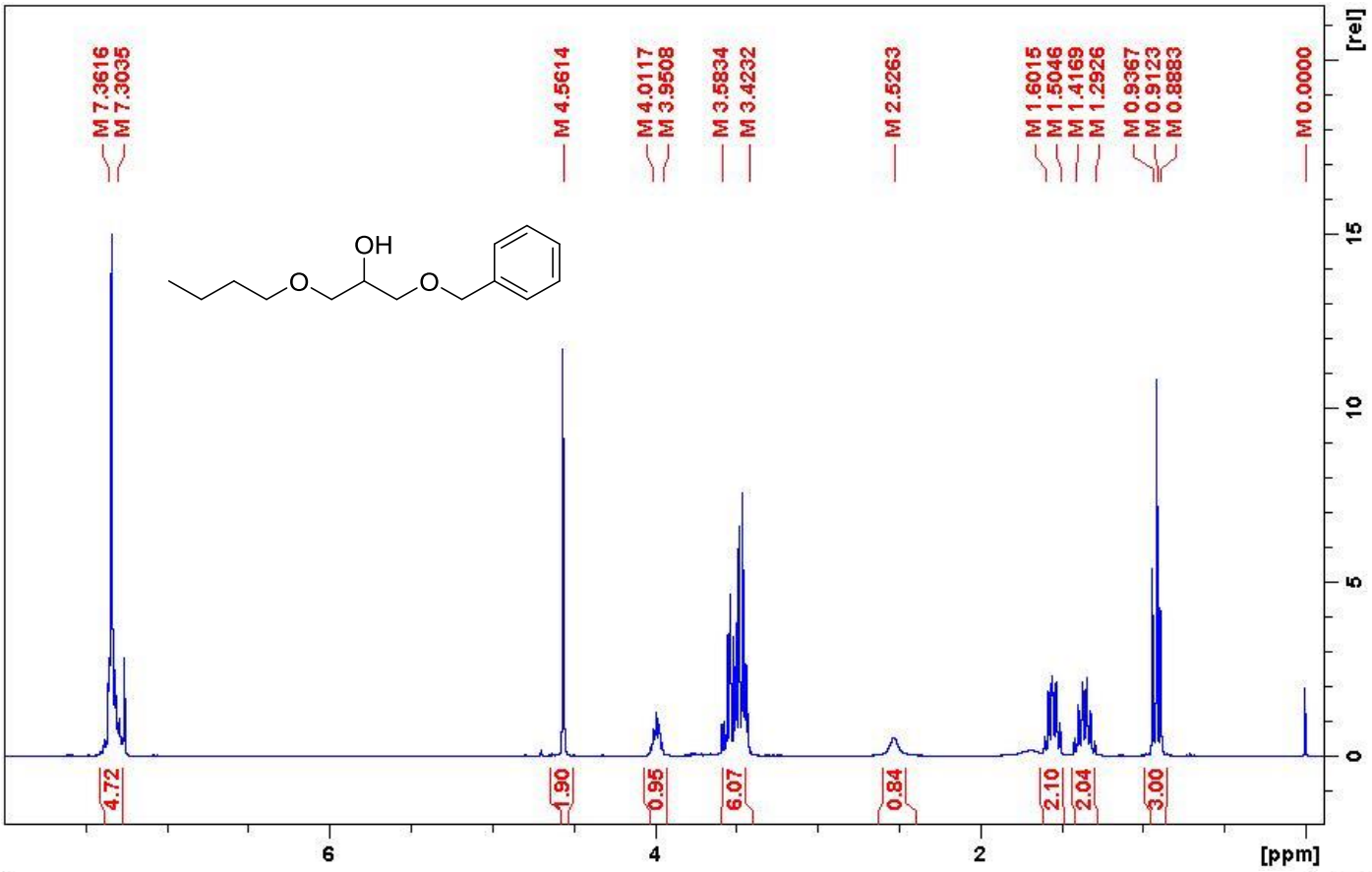
2955,41
2871,82
1462,36
1366,01
1327,27
1259,31
1196,30
1104,62
1009,44
969,95
669,12
622,28
OH
O
OMe


RT: 0.00 - 52.01
NL:
1.94E6
TIC MS
RLEi401
Relative Abundance
Time (min)
17.12
OH
O
OMe


RLEi401 #605-732 RT: 15.30-17.89 AV: 128 NL: 5.84E4
T: + c Full ms [50.00-650.00]
Relative Abundance
m/z
57.09
75.08
59.09
73.10
87.08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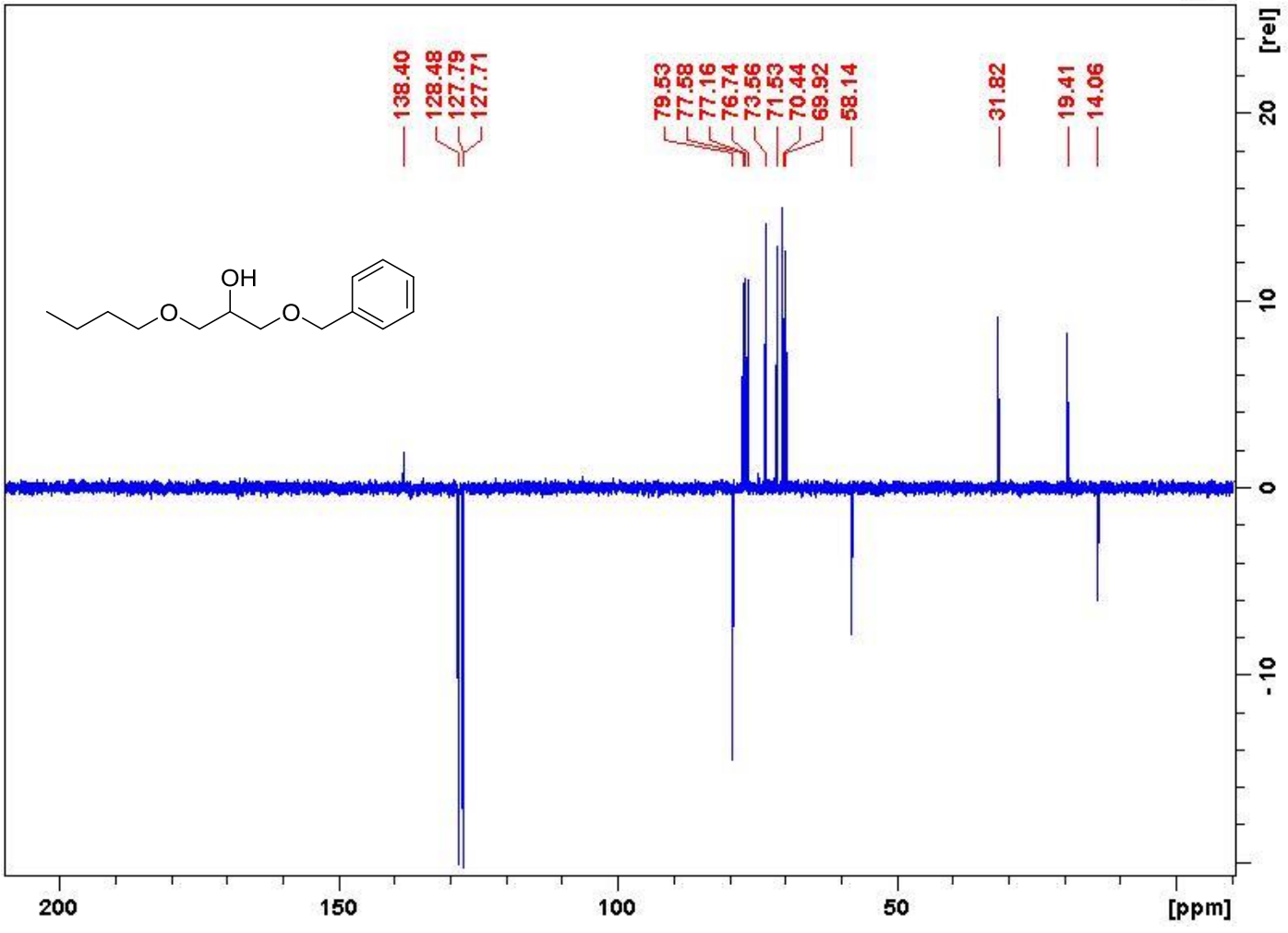
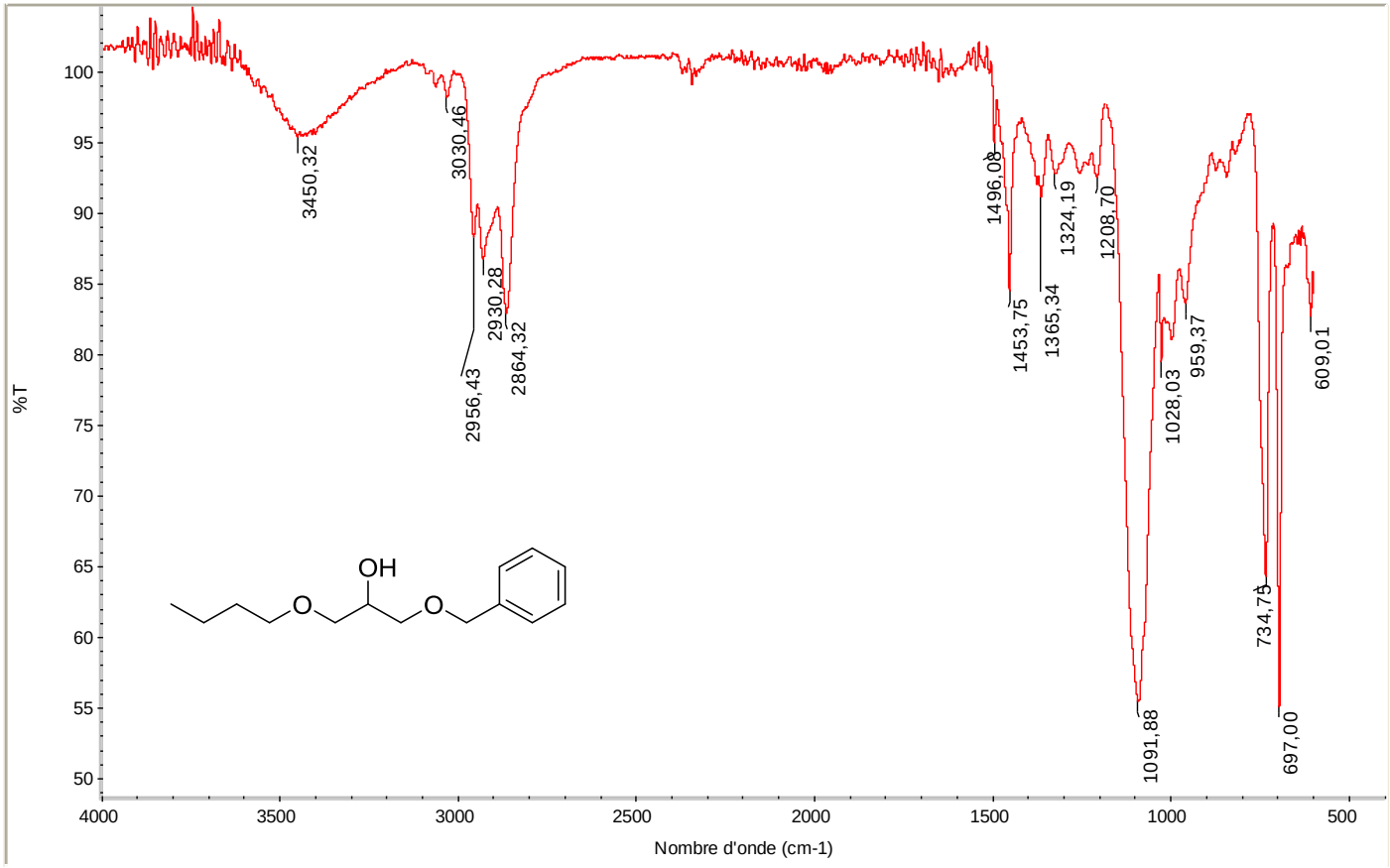
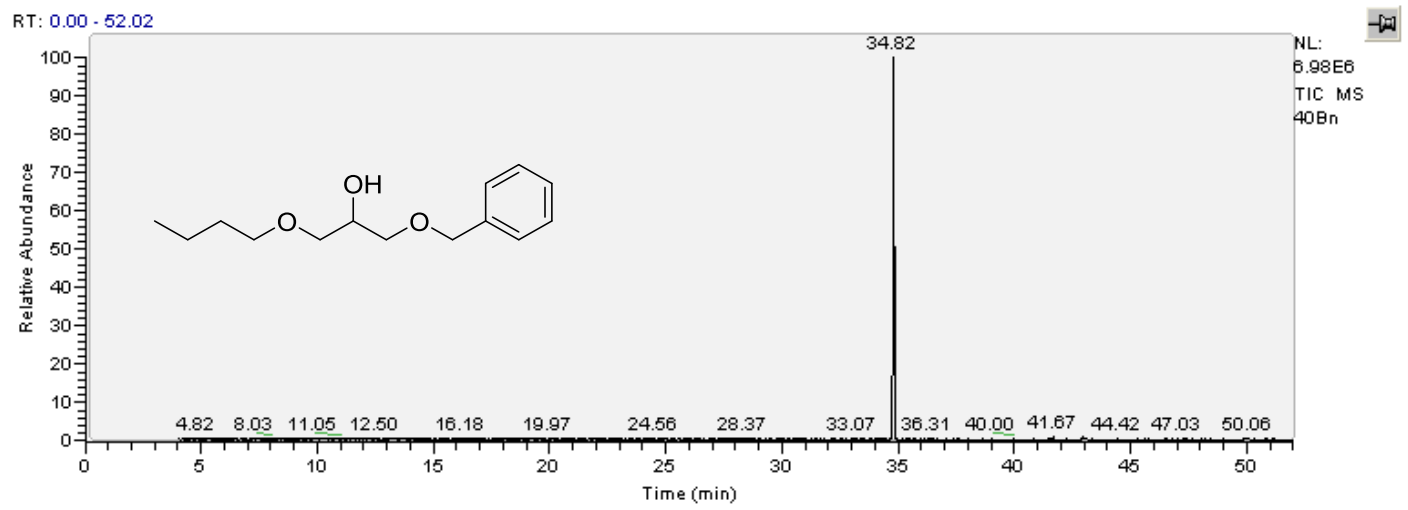
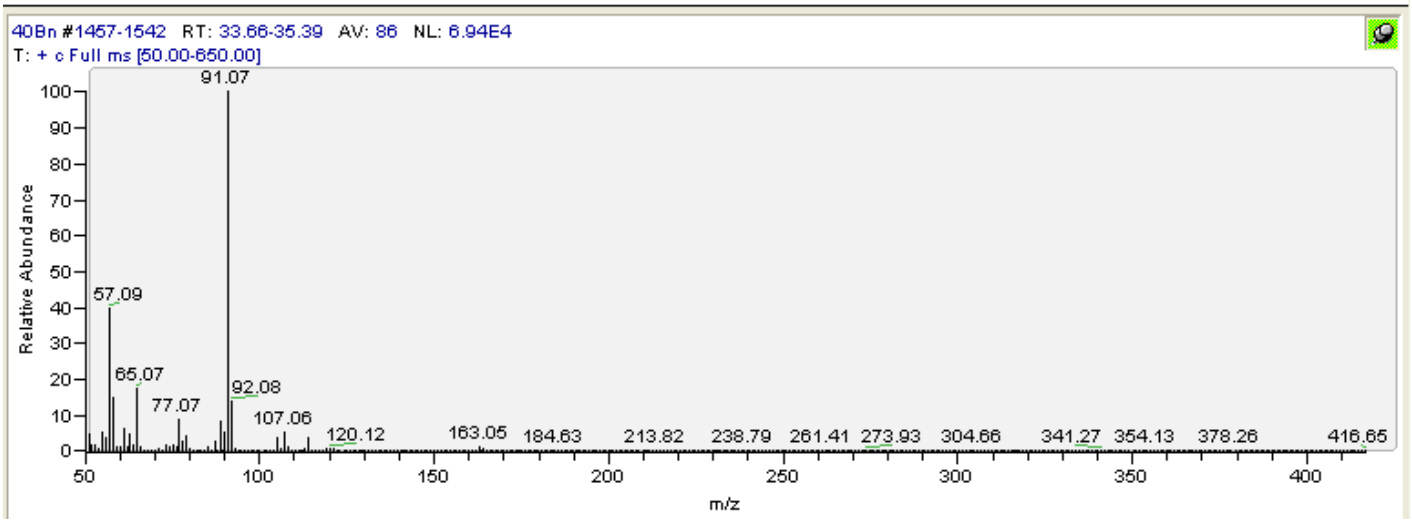
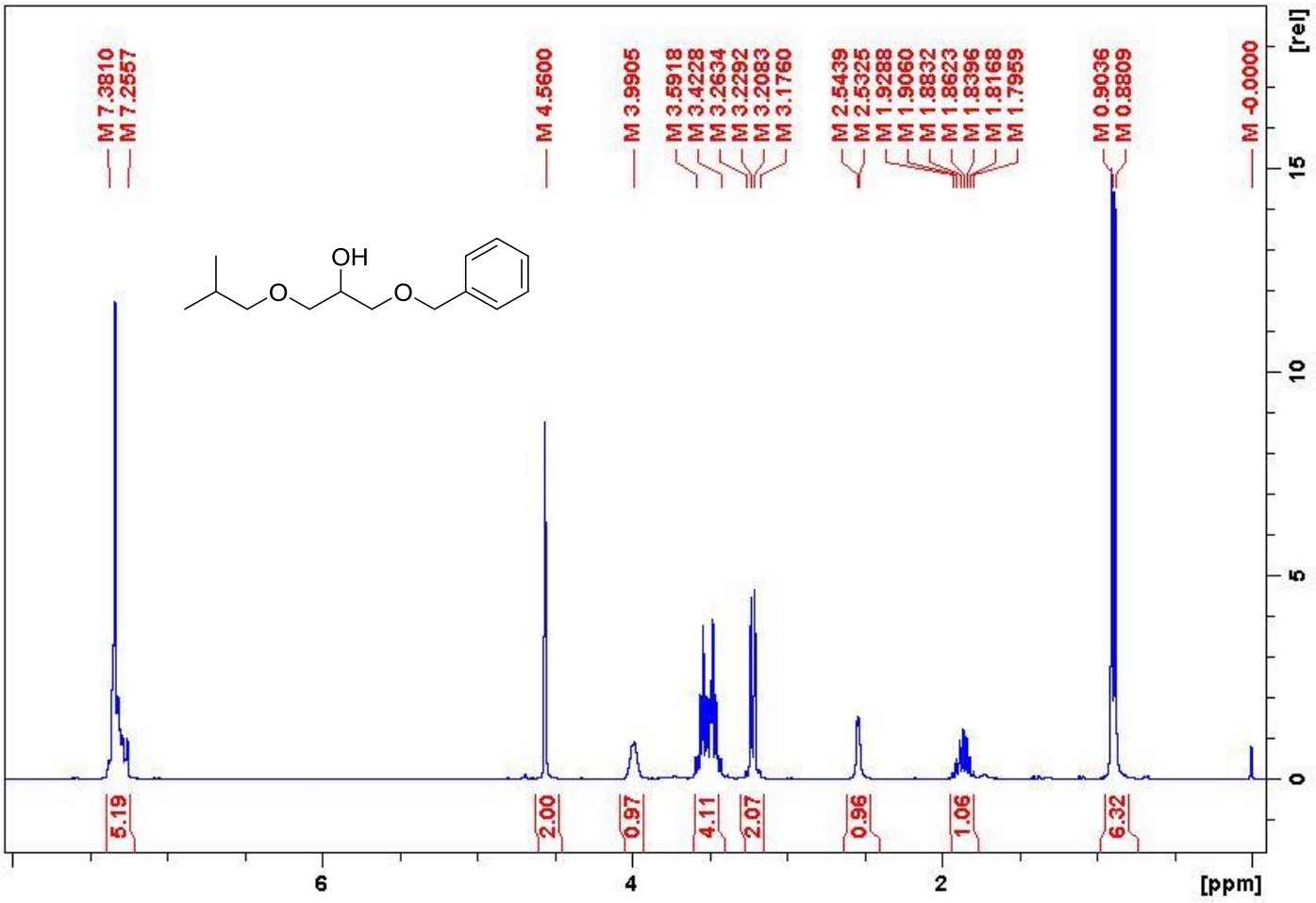
M 7.3035
M 4.5614
M 4.0117
M 3.9508
M 3.5834
M 3.4232
M 2.5263
M 1.6015
M 1.5046
M 1.4169
M 1.2926
M 0.9367
M 0.9123
M 0.8883
M 0.0000
OH
O
O
4.72
1.90
0.95
6.07
0.84
2.10
2.04
3.00
[rel]
[ppm]

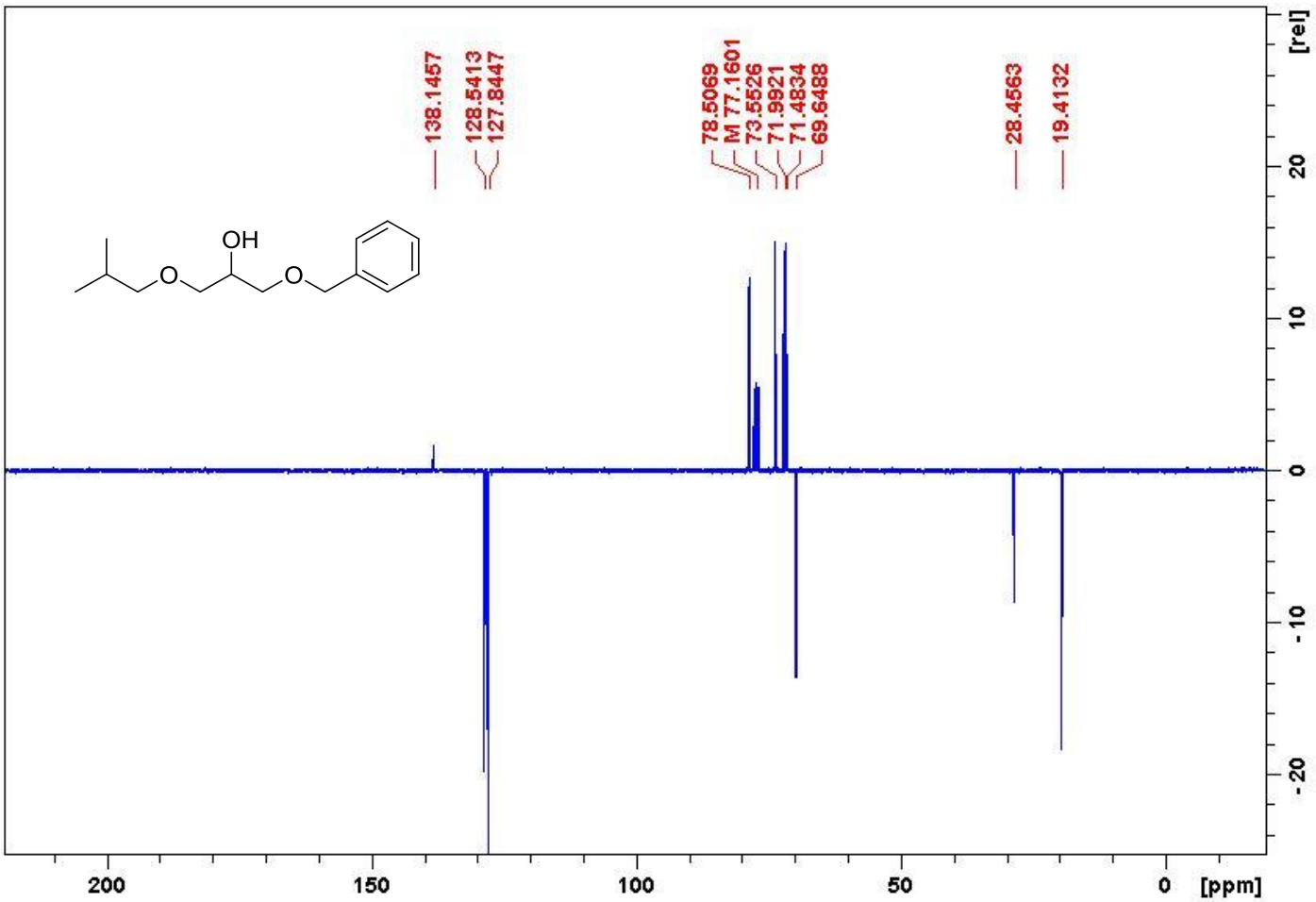

138.40
128.48
127.79
127.71
79.53
77.58
77.16
76.74
73.56
71.53
70.44
69.92
58.14
31.82
19.41
14.06
OH
O
O
[rel]
[ppm]

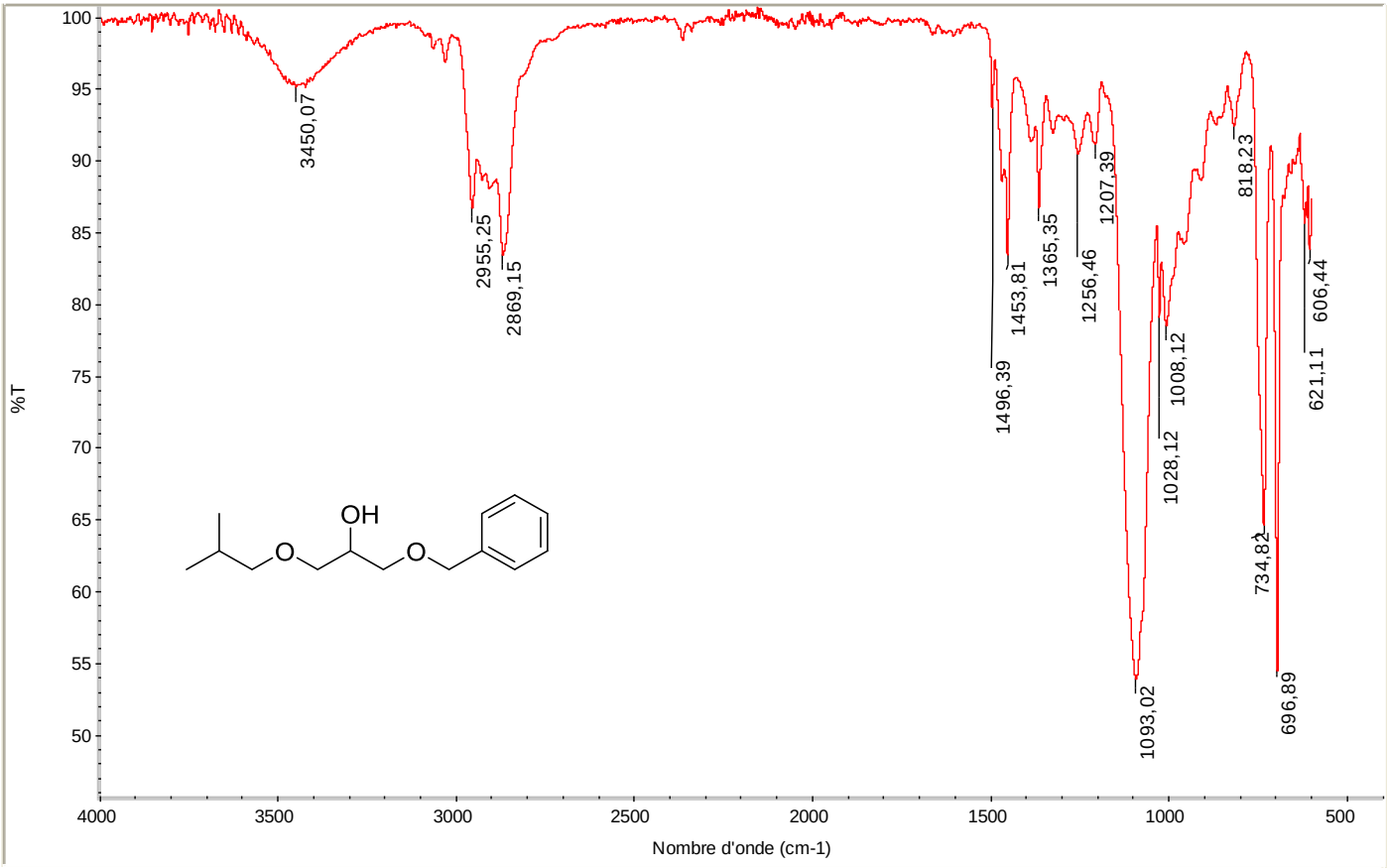

%T
Nombre d'onde (cm-1)
3450,32
3030,46
2956,43
2930,28
2864,32
1496,08
1453,75
1365,34
1324,19
1208,70
1091,88
1028,03
959,37
734,75
697,00
609,01
OH
O
O

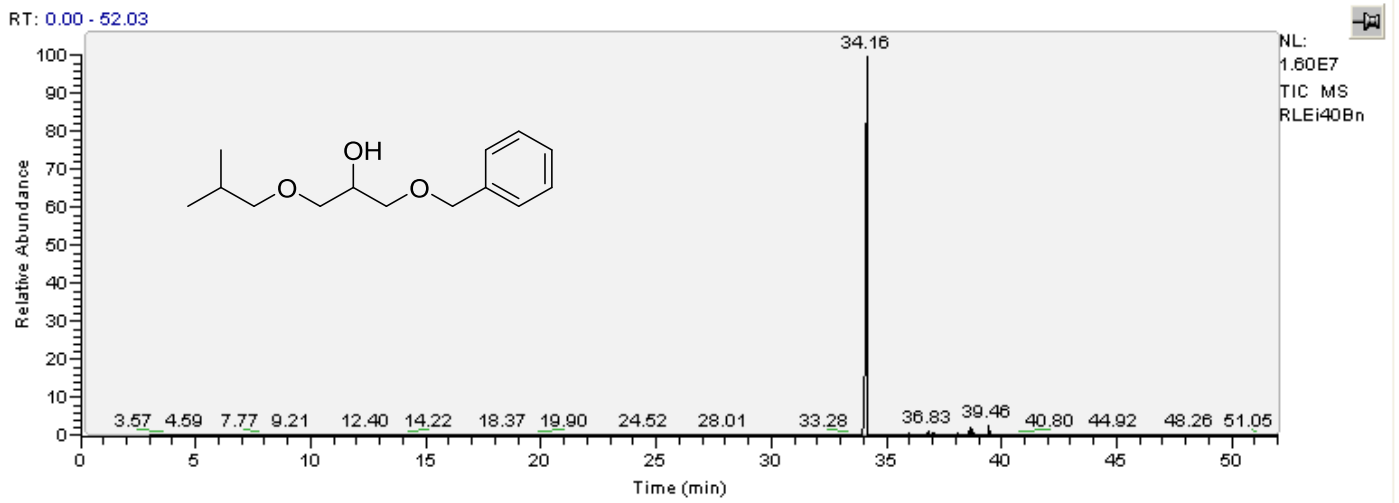

RT: 0.00 - 52.02
Relative Abundance
Time (min)
34.82
NL:
6.98E6
TIC MS
40Bn
4.82 8.03 11.05 12.50 16.18 19.97 24.56 28.37 33.07 36.31 40.00 41.67 44.42 47.03 50.06
OH
O
O

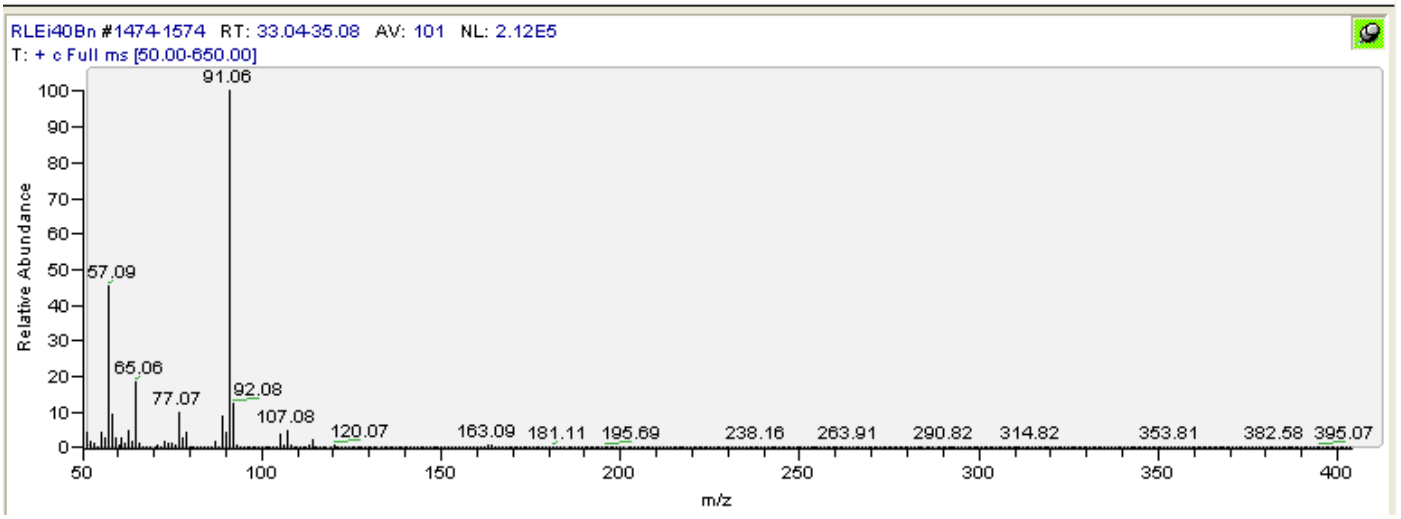

40Bn #1457-1542 RT: 33.66-35.39 AV: 86 NL: 6.94E4
T: + c Full ms [50.00-650.00]
Relative Abundance
m/z
91.07
57.09
65.07
77.07
92.08
107.06
120.12
163.05
184.63
213.82
238.79
261.41
273.93
304.66
341.27
354.13
378.26
416.65

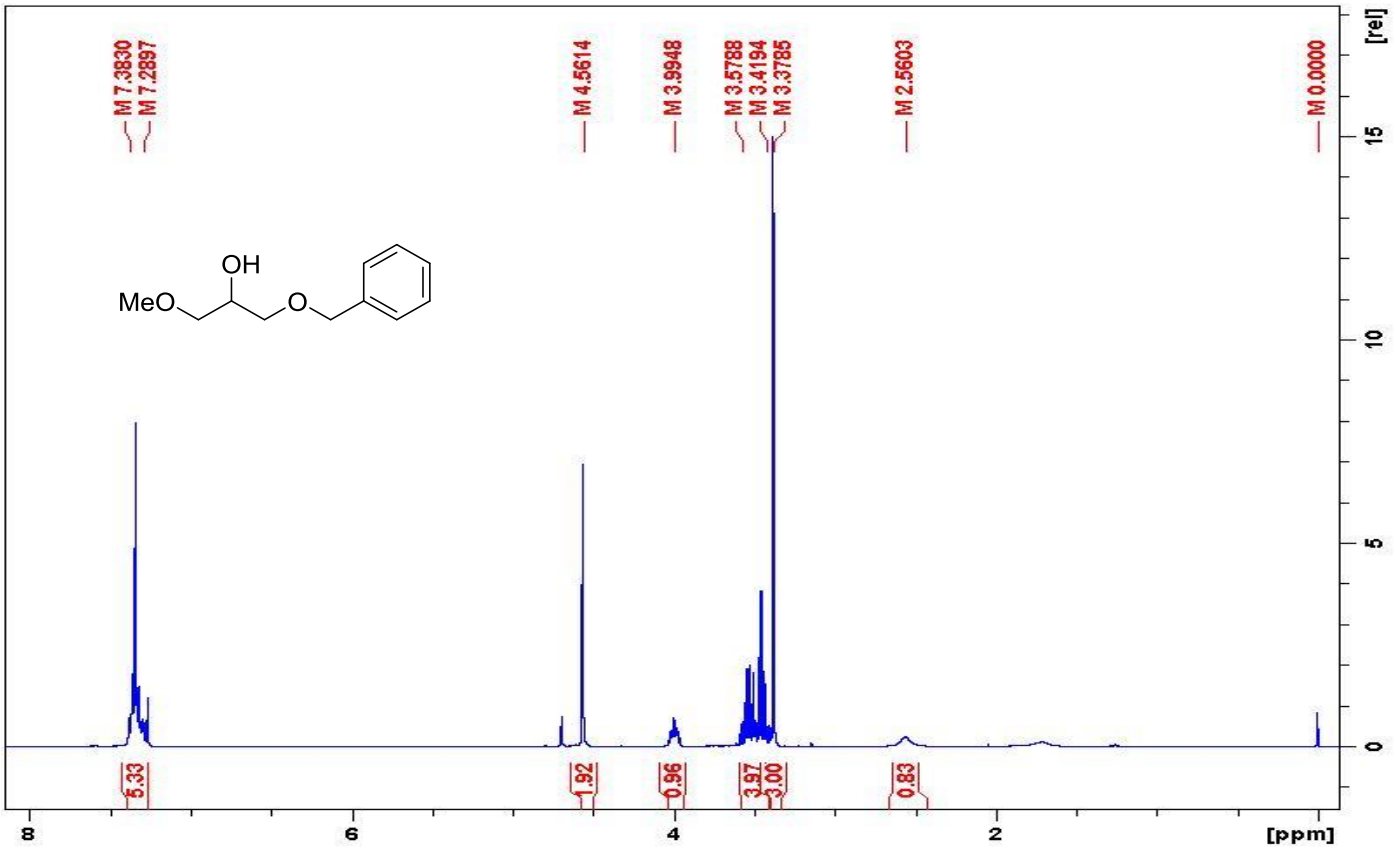

M 7.3810
M 7.2557
M 4.5600
M 3.9905
M 3.5918
M 3.4228
M 3.2634
M 3.2292
M 3.2083
M 3.1760
M 2.5439
M 2.5325
M 1.9288
M 1.9060
M 1.8832
M 1.8623
M 1.8396
M 1.8168
M 1.7959
M 0.9036
M 0.8809
M -0.0000
OH
O
O
5.19
2.00
0.97
4.11
2.07
0.96
1.06
6.32
[rel]
[ppm]

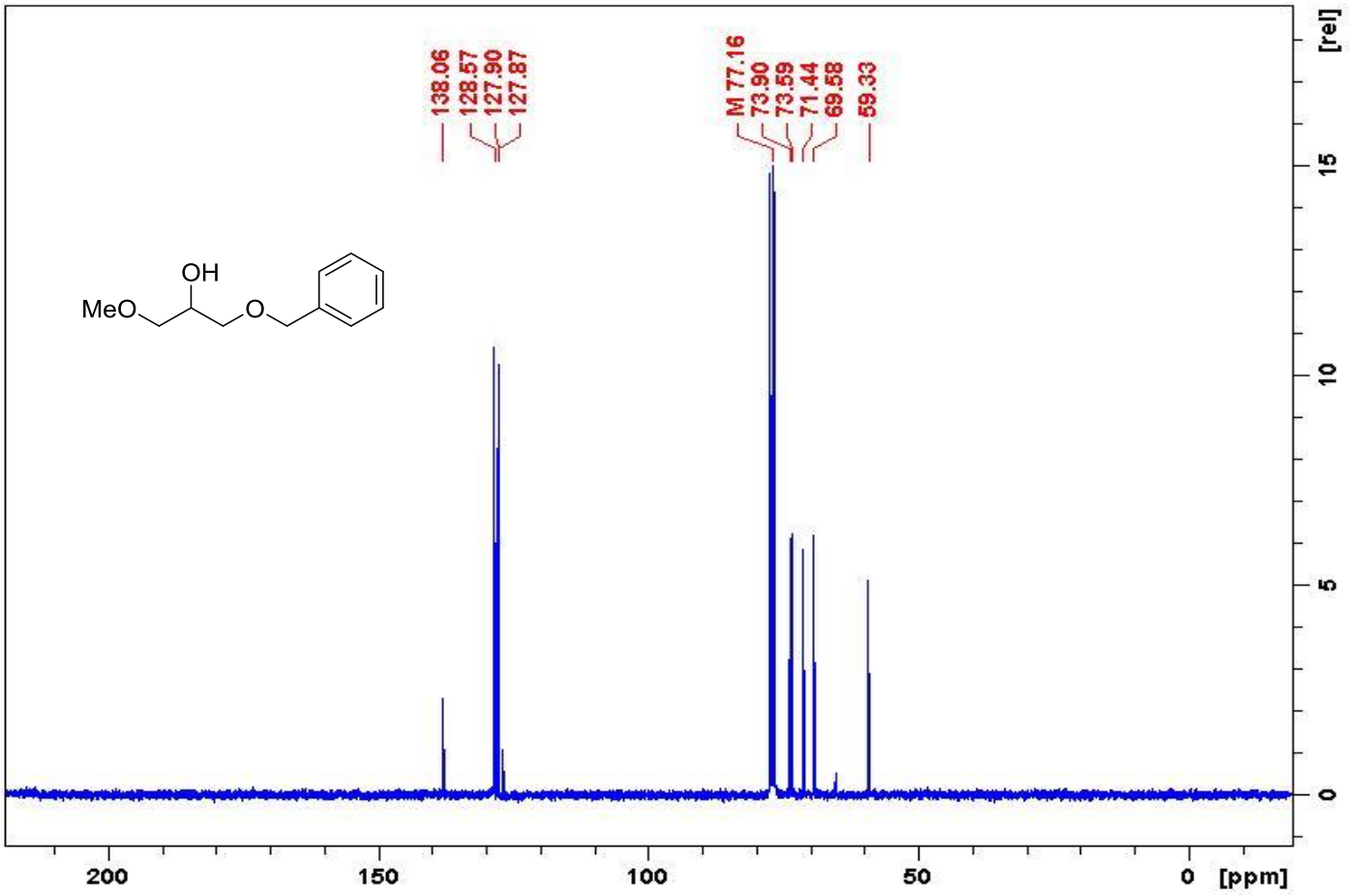

138.1457
128.5413
127.8447
78.5069
M 77.1601
73.5526
71.9921
71.4834
69.6488
28.4563
19.4132
OH
O
O
[rel]
[ppm]

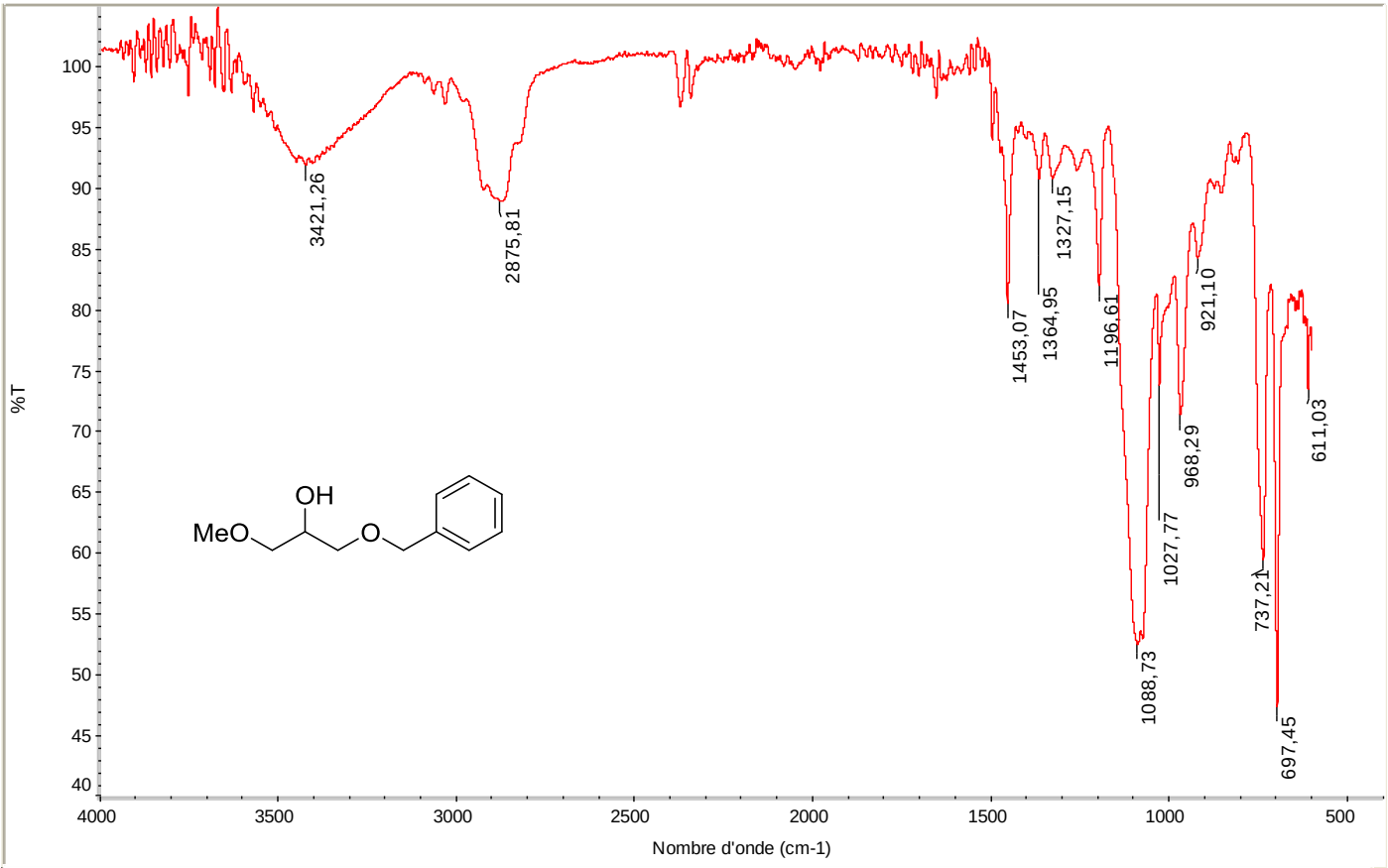

%T
Nombre d'onde (cm-1)
3450,07
2955,25
2869,15
1496,39
1453,81
1365,35
1256,46
1207,39
1093,02
1028,12
1008,12
818,23
734,82
696,89
621,11
606,44
OH
O
O

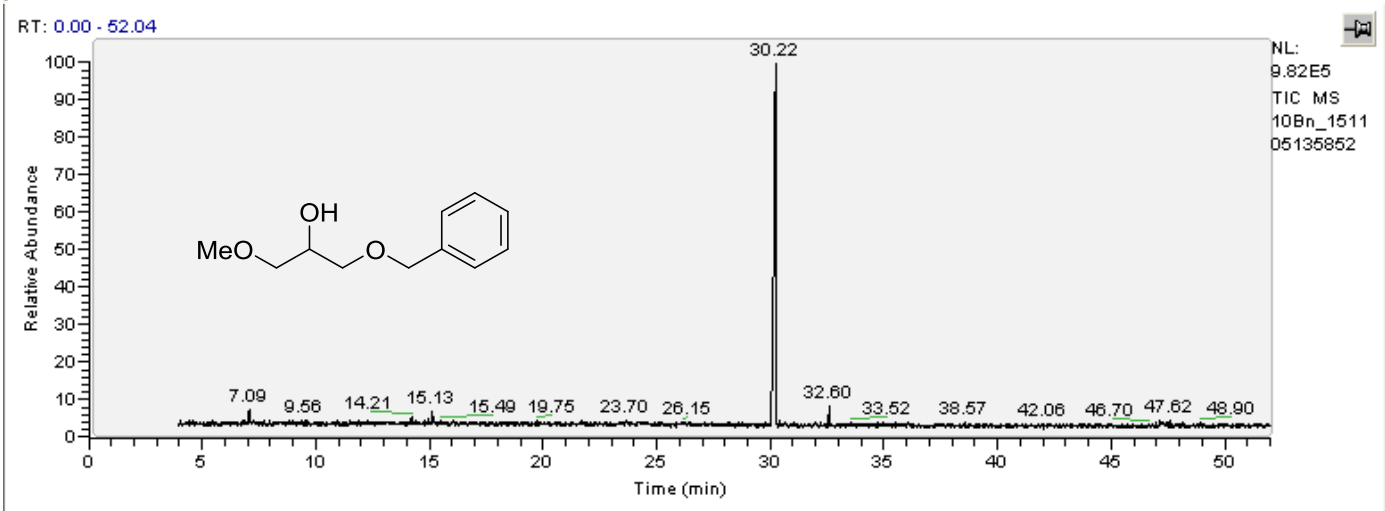

RT: 0.00 - 52.03
NL:
1.60E7
TIC MS
RLEi40Bn
Relative Abundance
Time (min)
34.16
3.57 4.59 7.77 9.21 12.40 14.22 18.37 19.90 24.52 28.01 33.28 36.83 39.46 40.80 44.92 48.26 51.05
OH
O
O

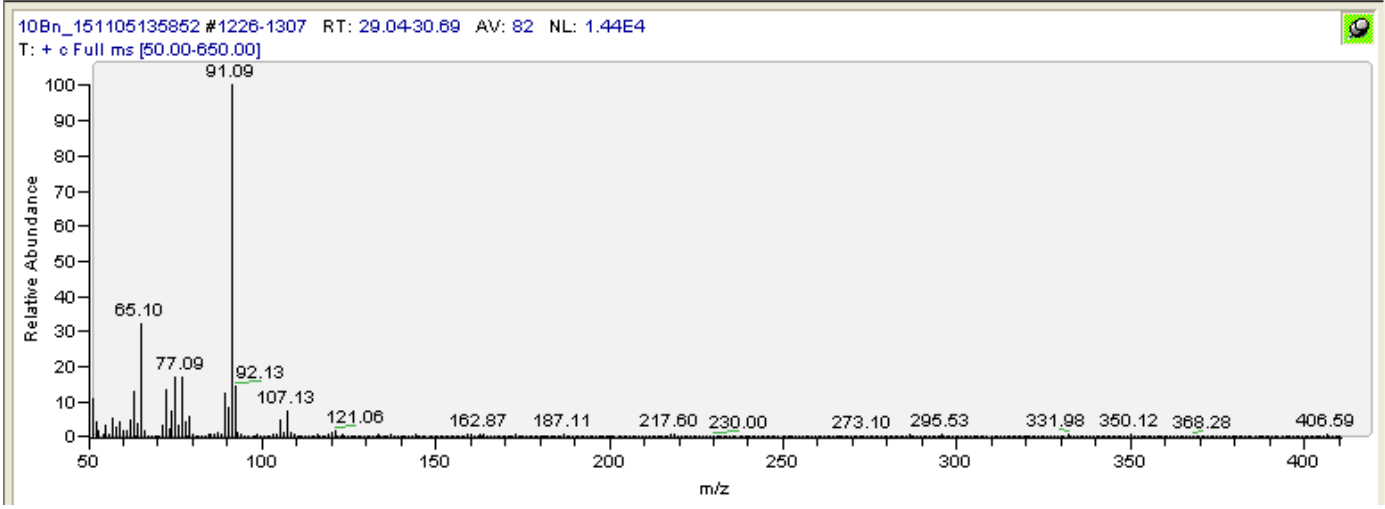

RLEi40Bn #1474-1574 RT: 33.04-35.08 AV: 101 NL: 2.12E5
T: + c Full ms [50.00-650.00]
Relative Abundance
m/z
91.06
57.09
65.06
77.07
92.08
107.08
120.07
163.09
181.11
195.69
238.16
263.91
290.82
314.82
353.81
382.58
395.07

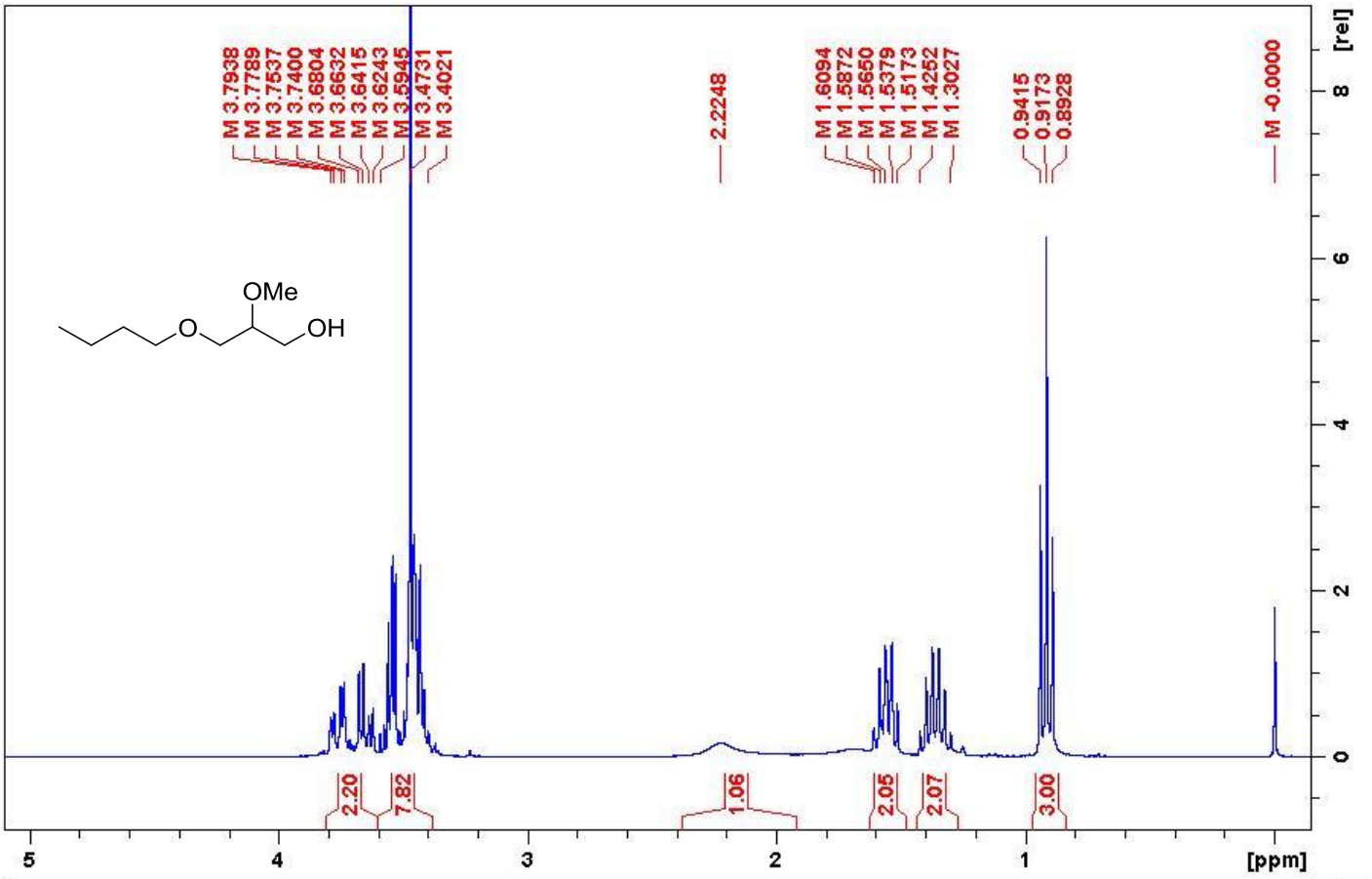

M 7.3830
M 7.2897
M 4.5614
M 3.9948
M 3.5788
M 3.4194
M 3.3785
M 2.5603
M 0.0000
[rel]
OH
MeO
O
5.33
1.92
0.96
3.97
3.00
0.83
8
6
4
2
[ppm]

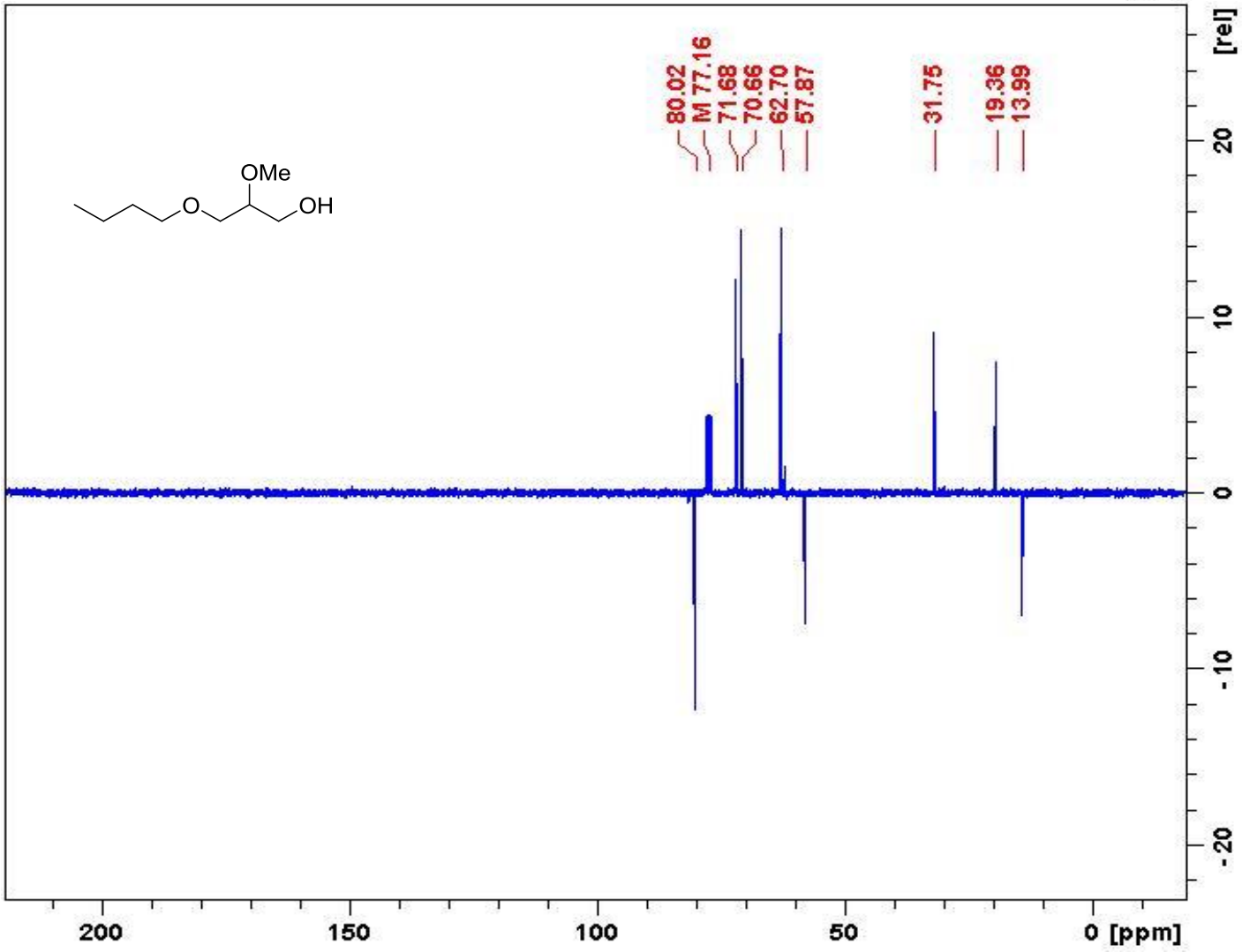

138.06
128.57
127.90
127.87
M 77.16
73.90
73.59
71.44
69.58
59.33
[rel]
OH
MeO
O
200
150
100
50
0
[ppm]

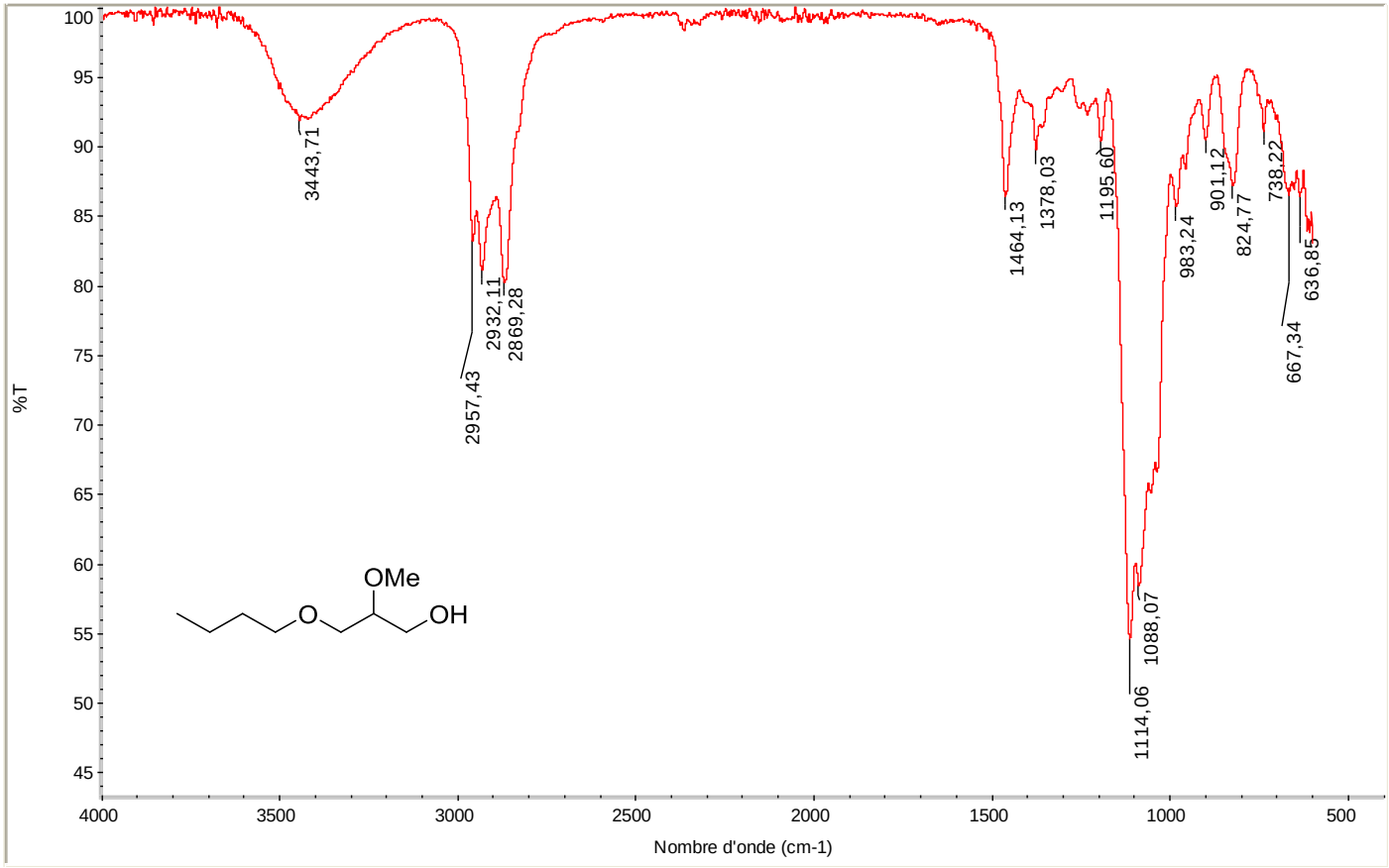

%T
Nombre d'onde (cm-1)
3421,26
2875,81
1453,07
1364,95
1327,15
1196,61
1088,73
1027,77
968,29
921,10
737,21
697,45
611,03
OH
MeO
O

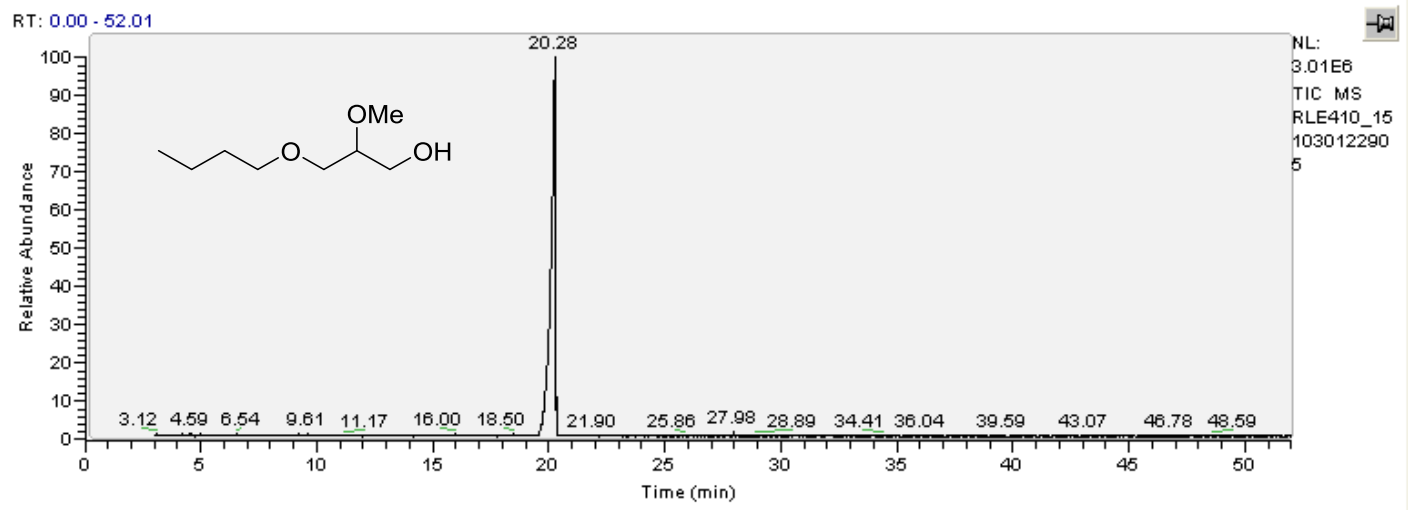

RT: 0.00 - 52.04
NL:
9.82E5
TIC MS
10Bn_1511
05135852
Relative Abundance
Time (min)
30.22
7.09
9.56
14.21
15.13
15.49
19.75
23.70
26.15
32.60
33.52
38.57
42.06
46.70
47.62
48.90
OH
MeO
O

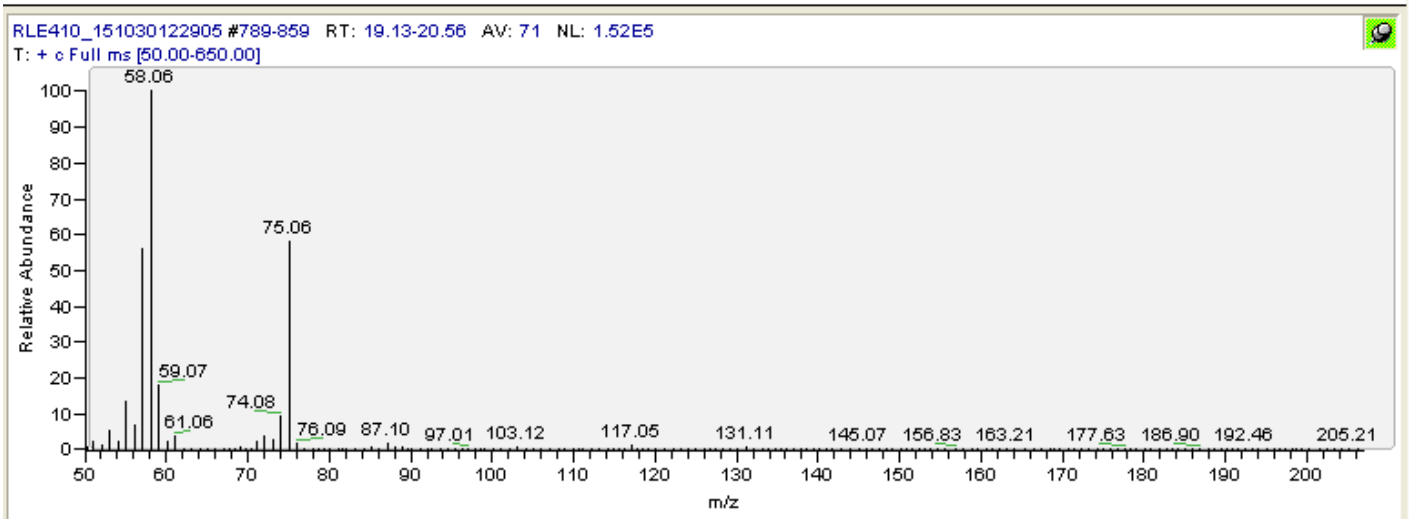

10Bn_151105135852 #1226-1307 RT: 29.04-30.69 AV: 82 NL: 1.44E4
T: + c Full ms [50.00-650.00]
Relative Abundance
m/z
91.09
65.10
77.09
92.13
107.13
121.06
162.87
187.11
217.60
230.00
273.10
295.53
331.98
350.12
368.28
406.59

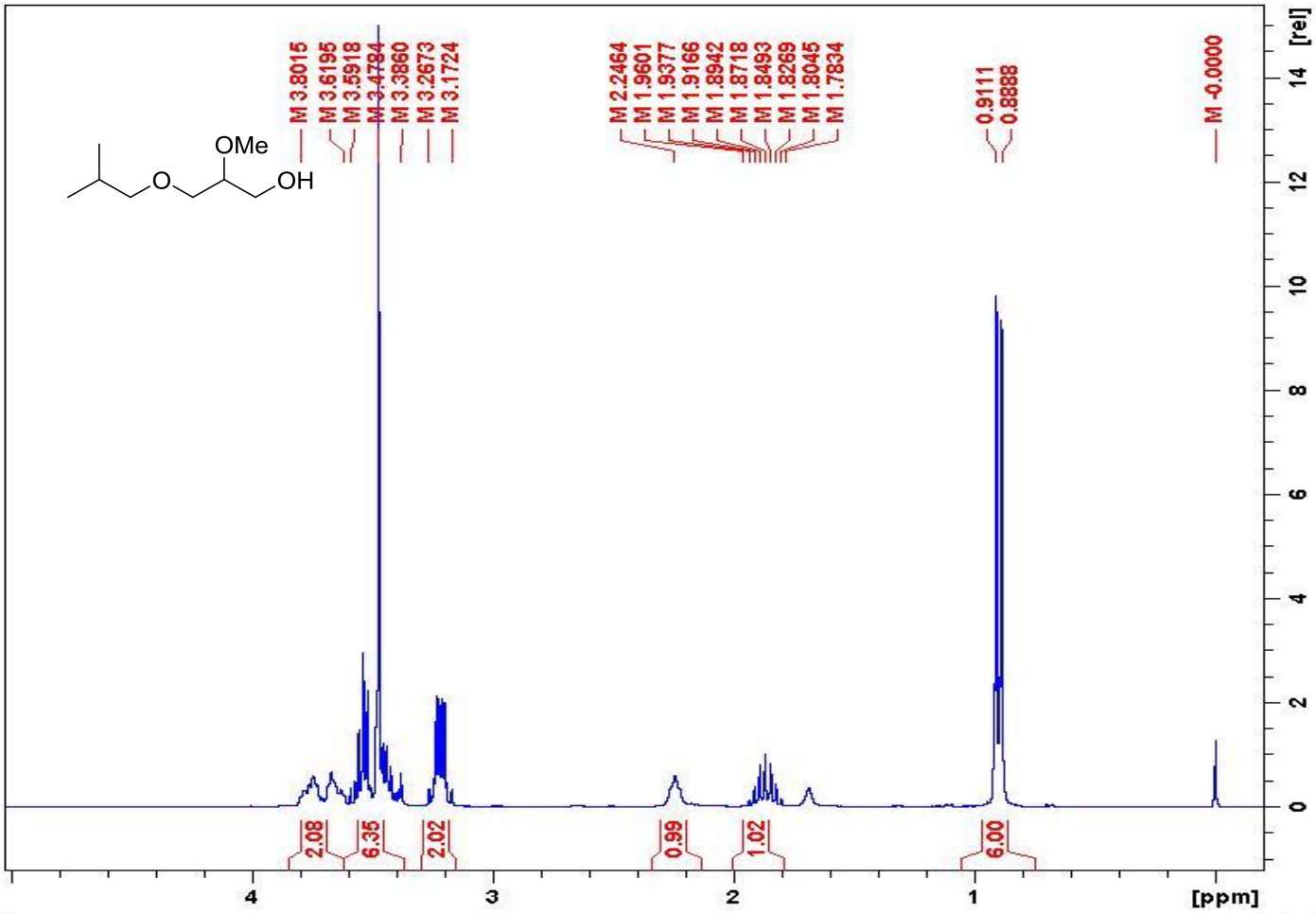

M 3.7938
M 3.7789
M 3.7537
M 3.7400
M 3.6804
M 3.6632
M 3.6415
M 3.6243
M 3.5945
M 3.4731
M 3.4021
2.2248
M 1.6094
M 1.5872
M 1.5650
M 1.5379
M 1.5173
M 1.4252
M 1.3027
0.9415
0.9173
0.8928
M -0.0000
OMe
O
OH
2.20
7.82
1.06
2.05
2.07
3.00
[rel]
[ppm]

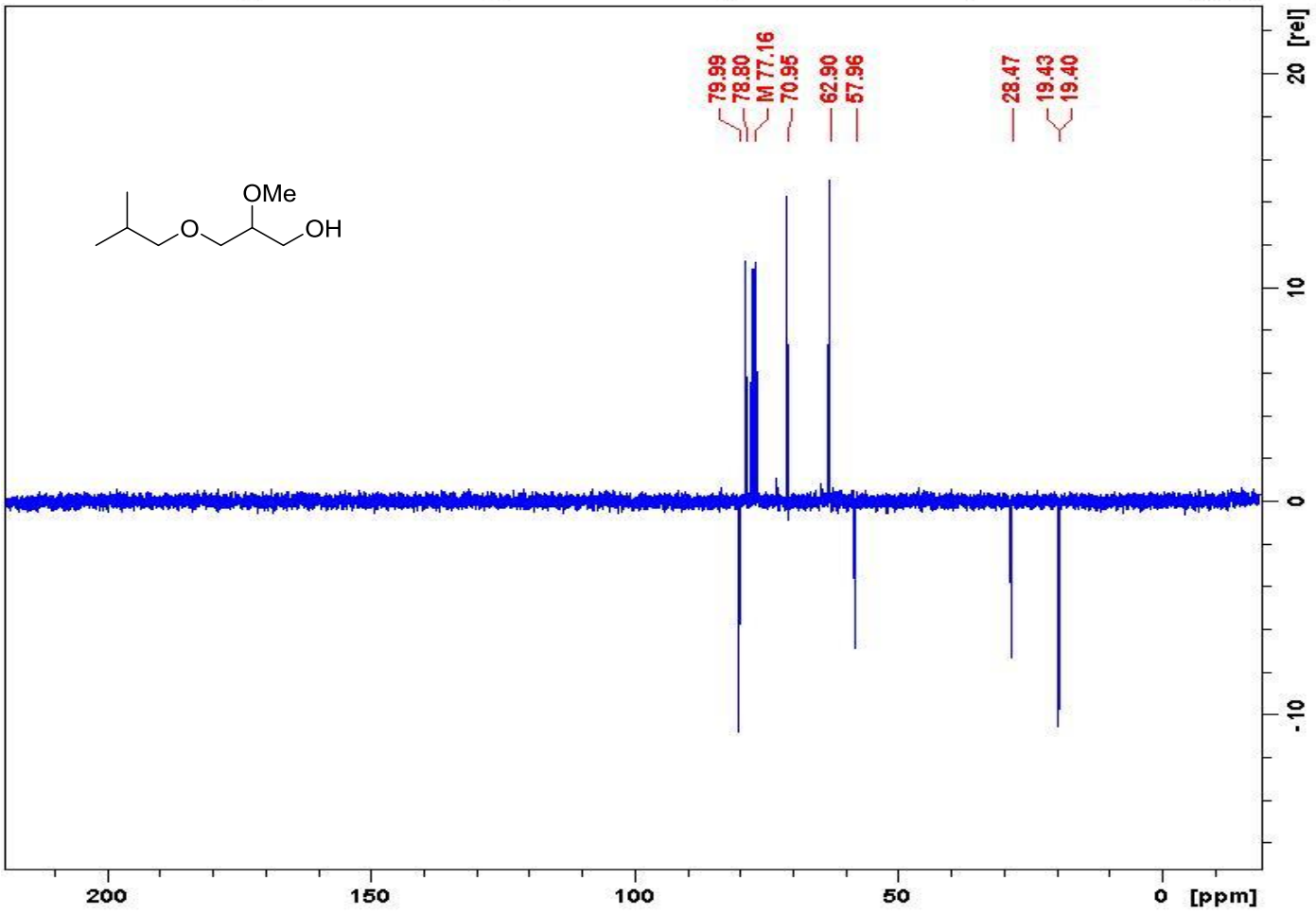

80.02
M 77.16
71.68
70.66
62.70
57.87
31.75
19.36
13.99
OMe
O
OH
[rel]
[ppm]

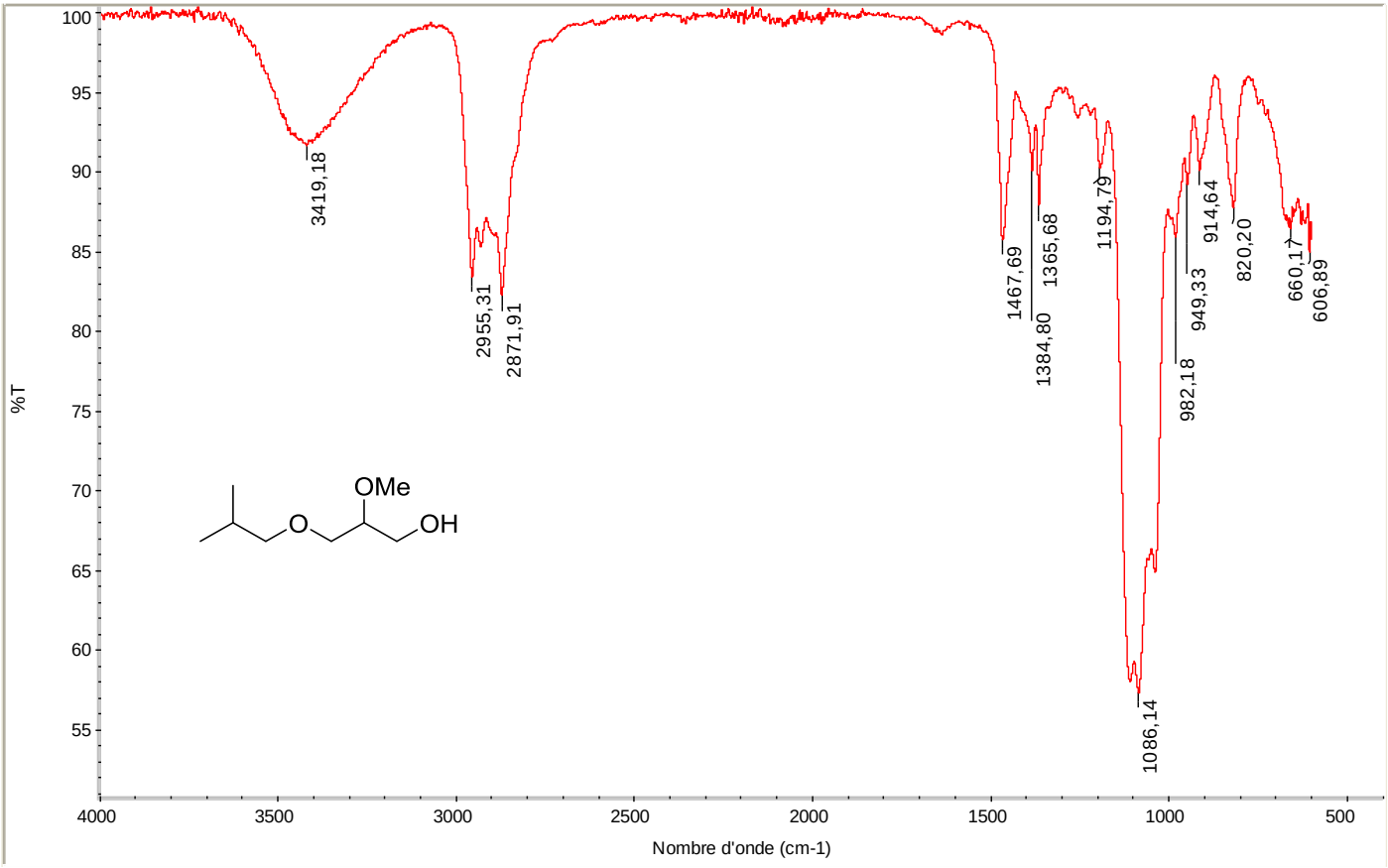

%T
Nombre d'onde (cm-1)
3443,71
2957,43
2932,11
2869,28
1464,13
1378,03
1195,60
1114,06
1088,07
983,24
901,12
824,77
738,22
667,34
636,85
OMe
O
OH

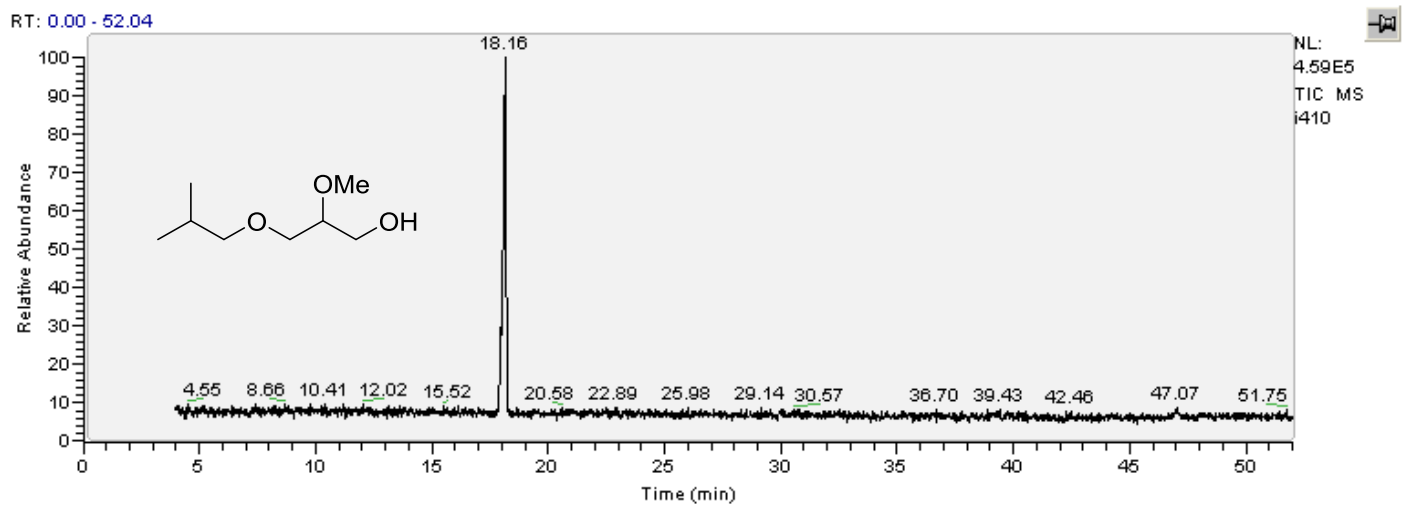

RT: 0.00 - 52.01
Relative Abundance
Time (min)
20.28
OMe
O
OH
NL:
3.01E6
TIC MS
RLE410_15
103012290
5

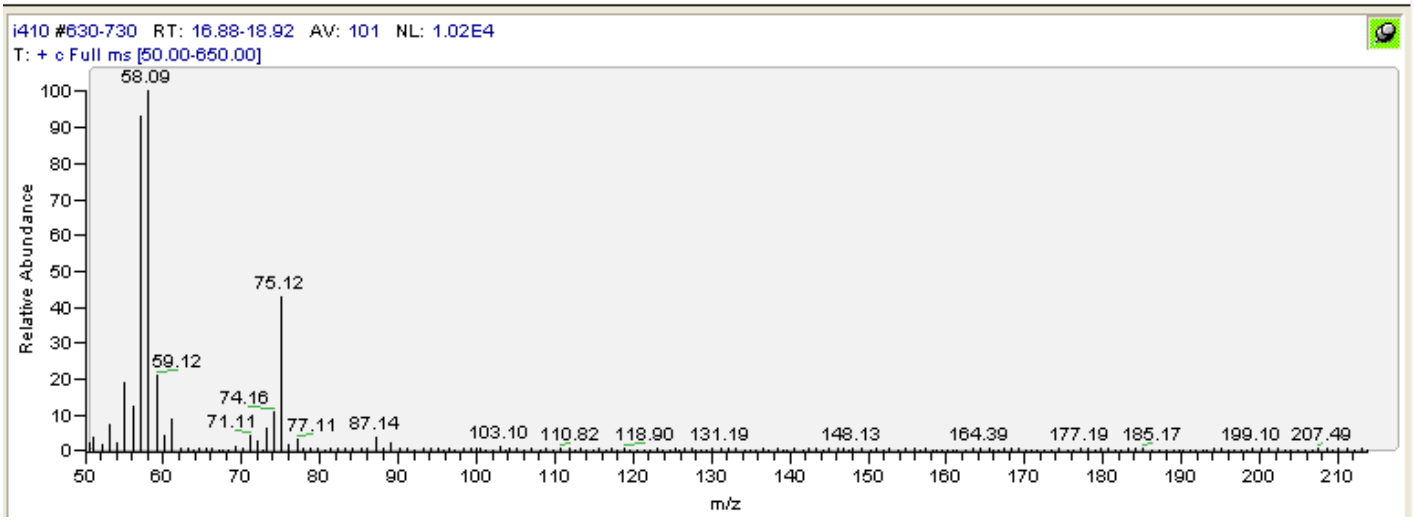

RLE410_151030122905 #789-859 RT: 19.13-20.56 AV: 71 NL: 1.52E5
T: + c Full ms [50.00-650.00]
Relative Abundance
m/z
58.06
75.06
59.07
74.08
61.06

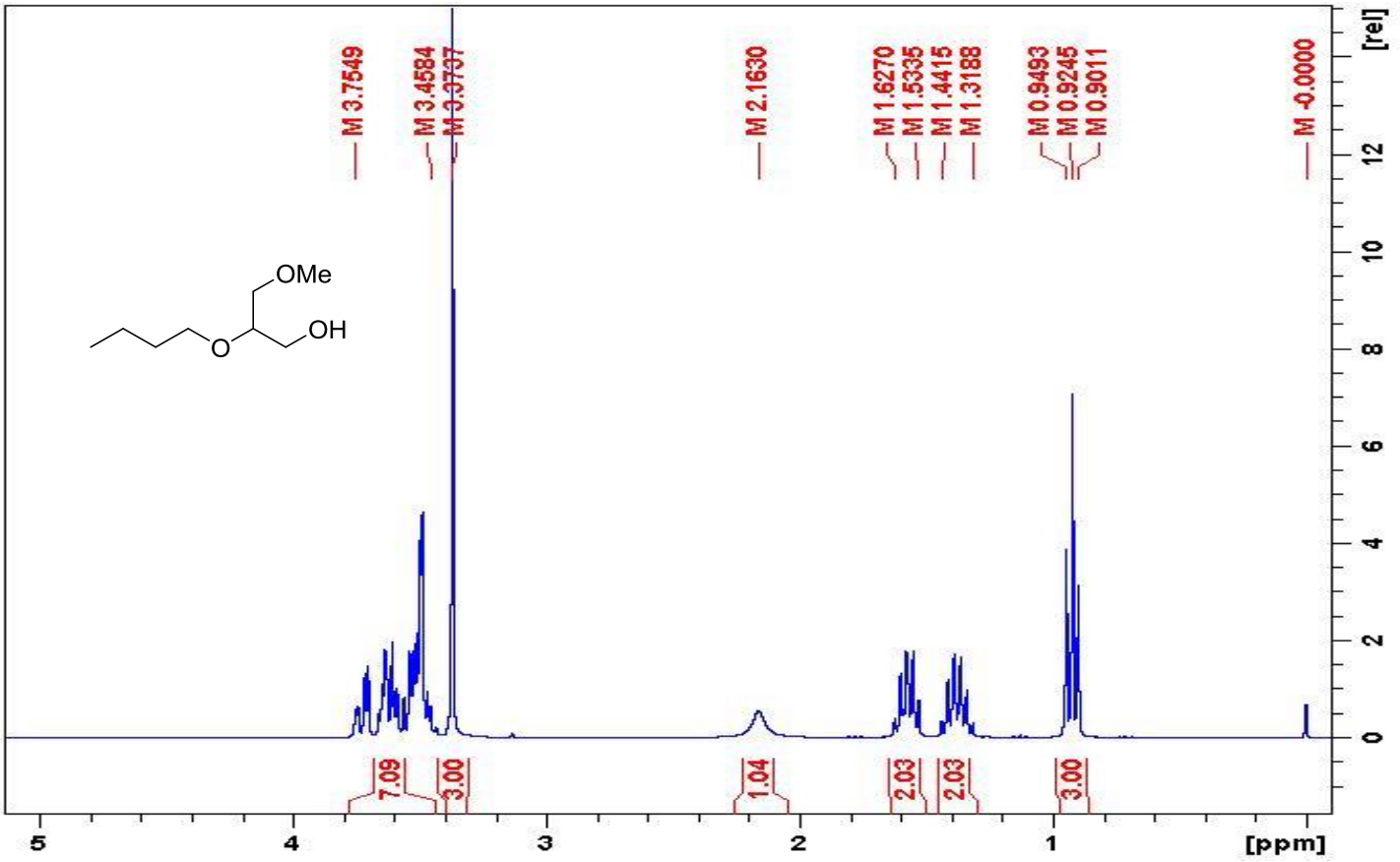

M 3.8015
M 3.6195
M 3.5918
M 3.4784
M 3.3860
M 3.2673
M 3.1724
M 2.2464
M 1.9601
M 1.9377
M 1.9166
M 1.8942
M 1.8718
M 1.8493
M 1.8269
M 1.8045
M 1.7834
0.9111
0.8888
M -0.0000
OMe
O
OH
2.08
6.35
2.02
0.99
1.02
6.00
4
3
2
1
[ppm]
[rel]

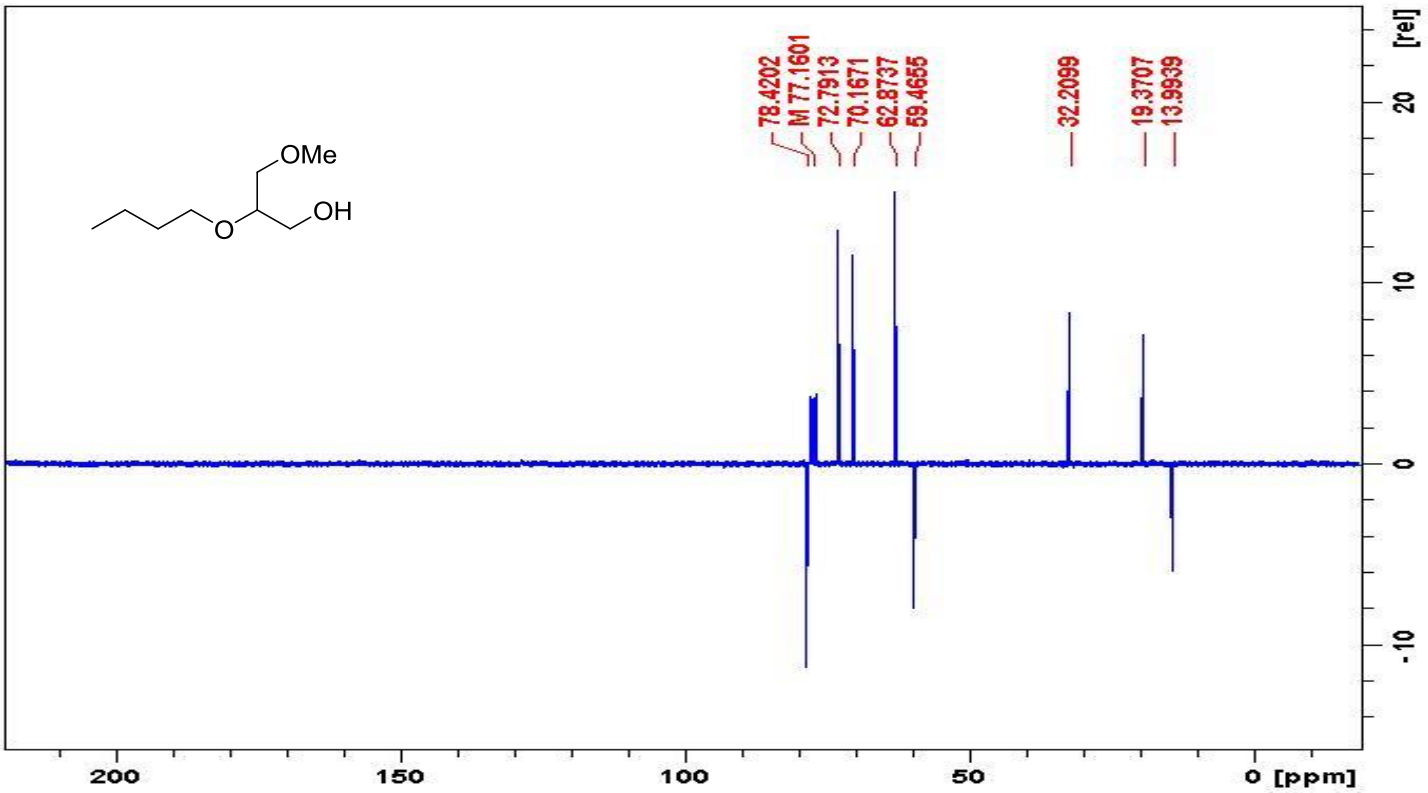

79.99
78.80
M 77.16
70.95
62.90
57.96
28.47
19.43
19.40
OMe
O
OH
200
150
100
50
0
[ppm]
[rel]

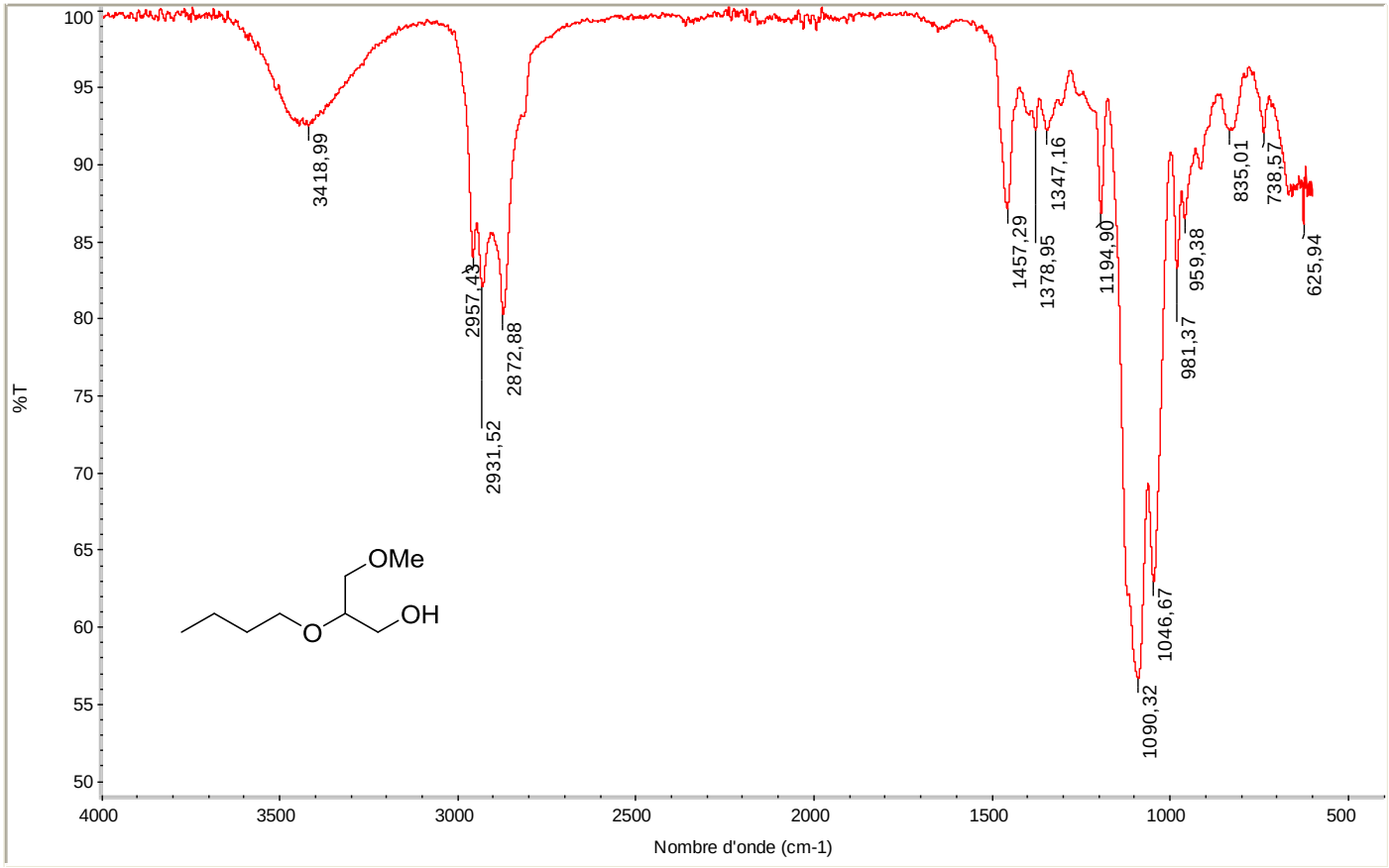

%T
Nombre d'onde (cm-1)
3419,18
2955,31
2871,91
1467,69
1384,80
1365,68
1194,79
1086,14
982,18
949,33
914,64
820,20
660,17
606,89
OMe
O
OH

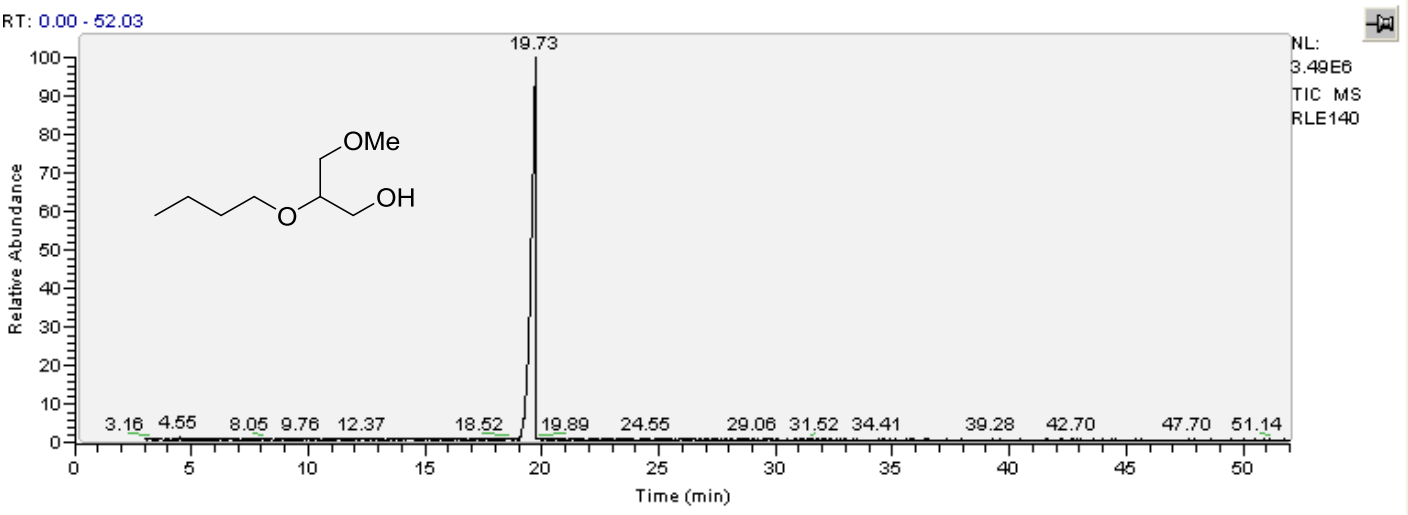

RT: 0.00 - 52.04
Relative Abundance
Time (min)
NL:
4.59E5
TIC MS
i410
18.16
4.55
8.66
10.41
12.02
15.52
20.58
22.89
25.98
29.14
30.57
36.70
39.43
42.46
47.07
51.75
OMe
O
OH

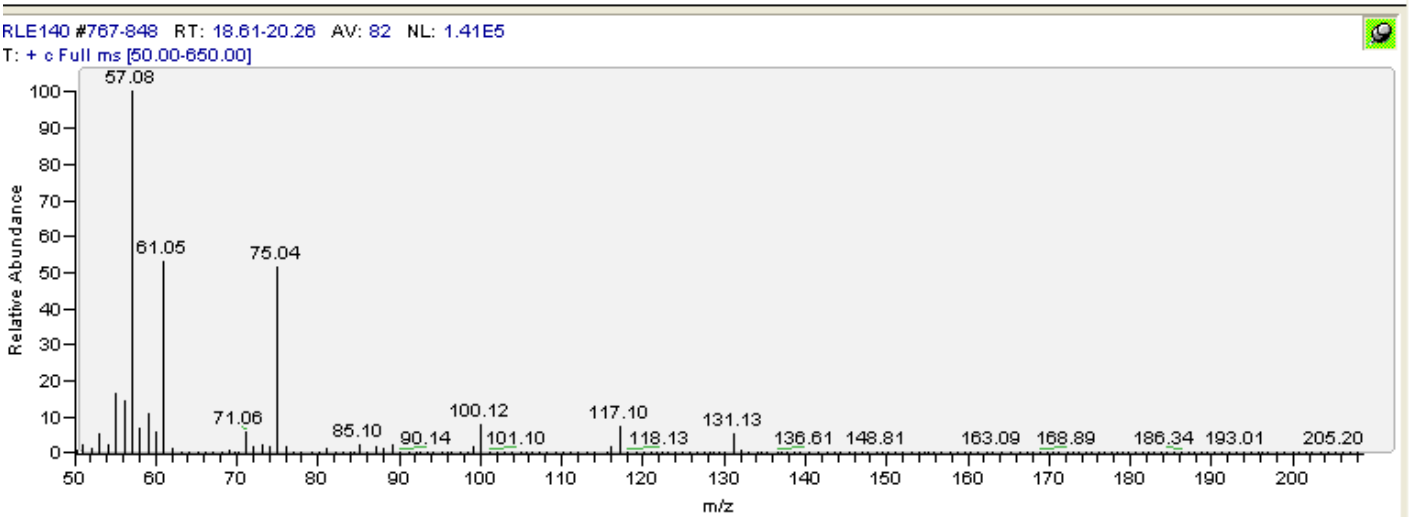

i410 #630-730 RT: 16.88-18.92 AV: 101 NL: 1.02E4
T: + c Full ms [50.00-650.00]
Relative Abundance
m/z
58.09
75.12
59.12
74.16
71.11
77.11
87.14
103.10
110.82
118.90
131.19
148.13
164.39
177.19
185.17
199.10
207.49

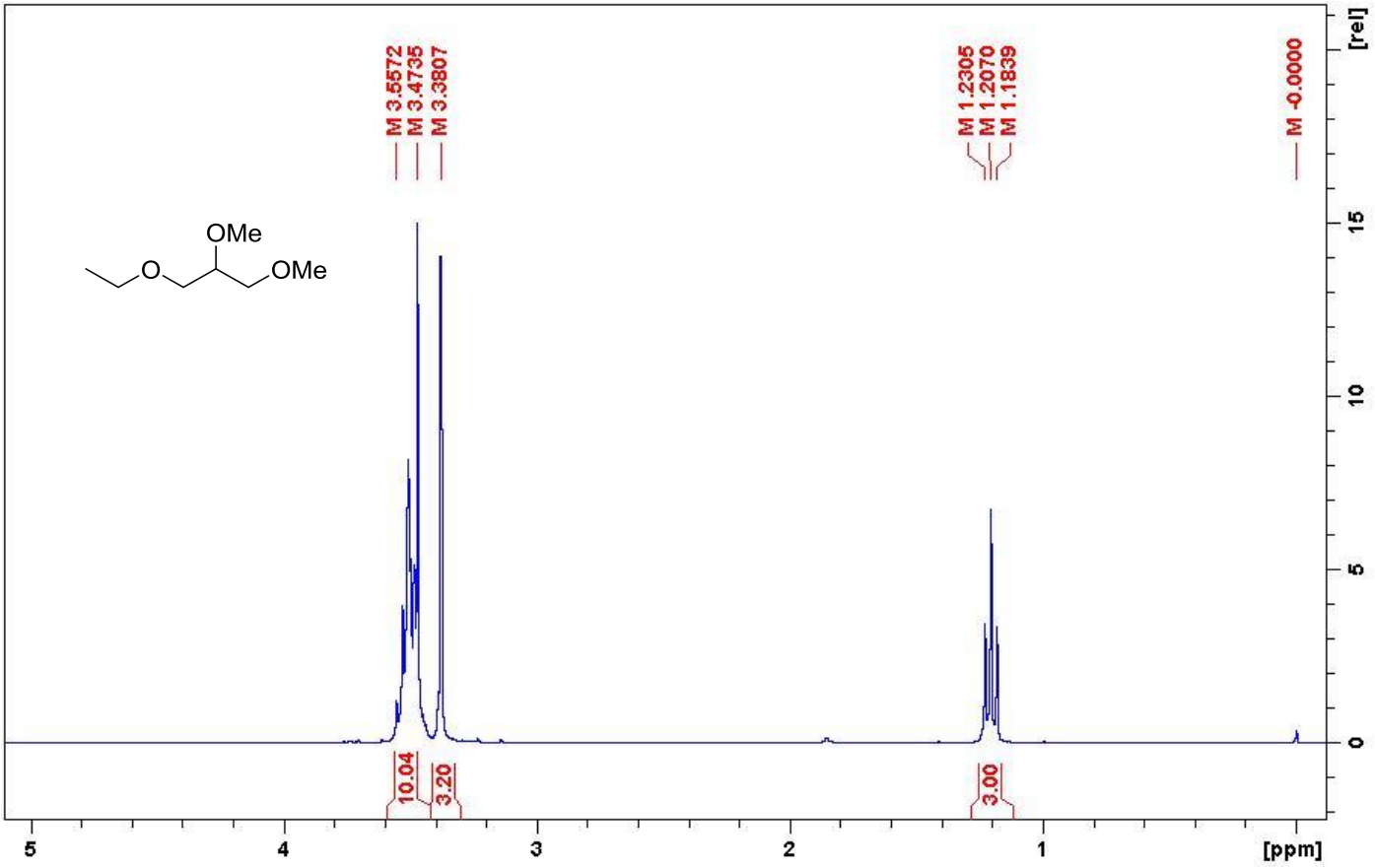

M 3.7549
M 3.4584
M 3.3737
M 2.1630
M 1.6270
M 1.5335
M 1.4415
M 1.3188
M 0.9493
M 0.9245
M 0.9011
M -0.0000
OMe
OH
O
7.09
3.00
1.04
2.03
2.03
3.00
[rel]
[ppm]

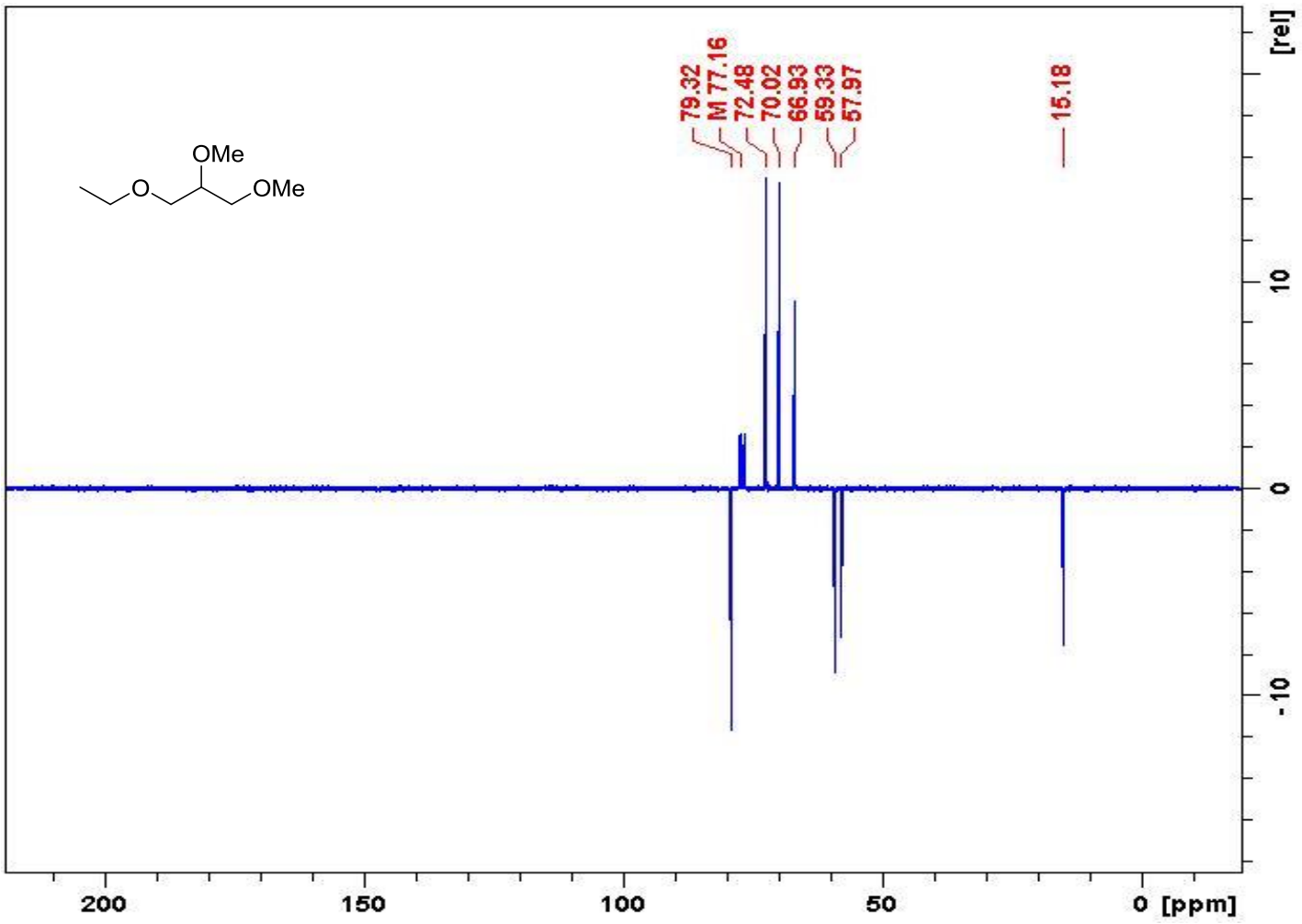

78.4202
M 77.1601
72.7913
70.1671
62.8737
59.4655
32.2099
19.3707
13.9939
OMe
OH
O
[rel]
[ppm]

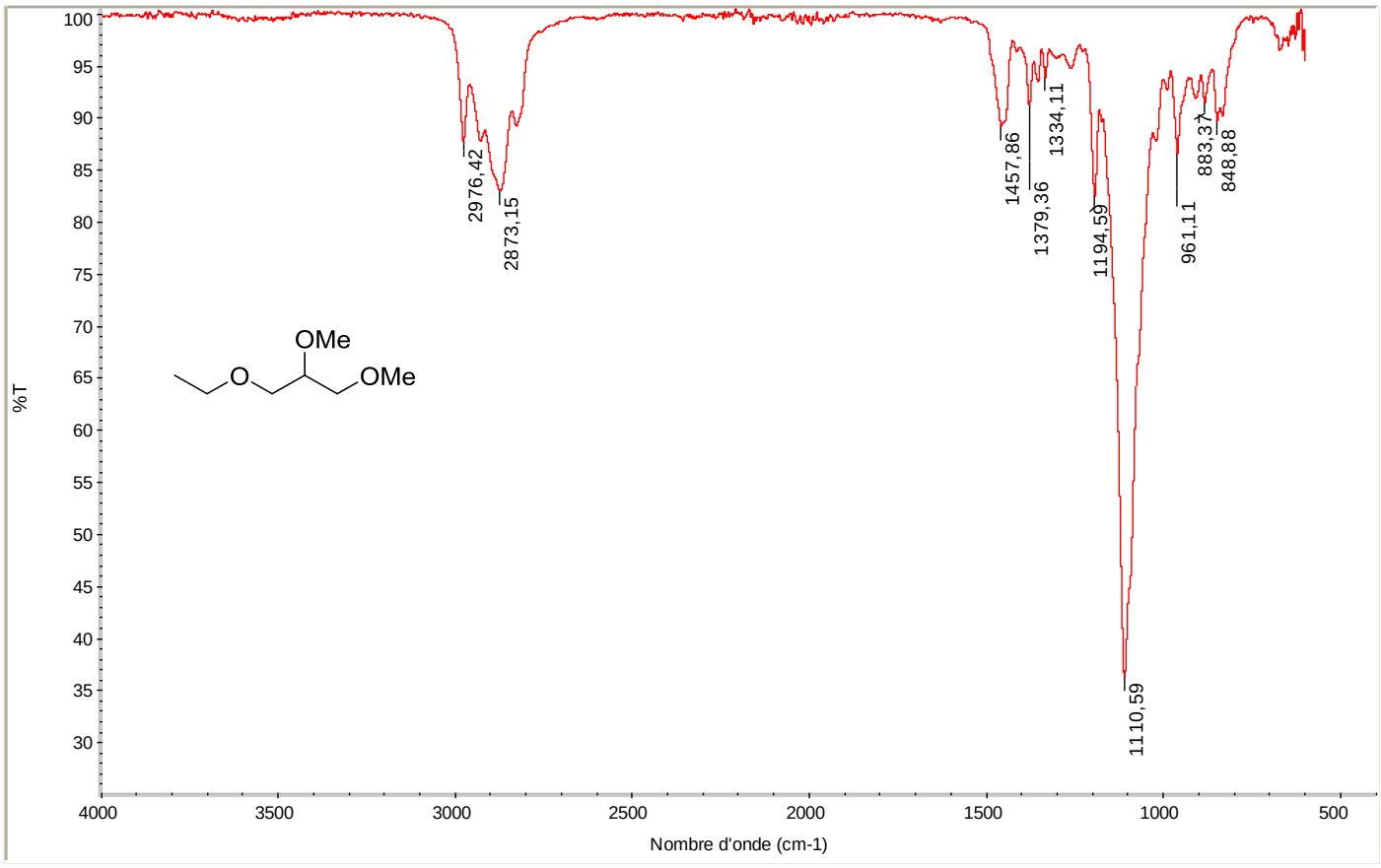

%T
Nombre d'onde (cm-1)
3418,99
2957,43
2931,52
2872,88
1457,29
1378,95
1347,16
1194,90
1090,32
1046,67
981,37
959,38
835,01
738,57
625,94
OMe
OH
O

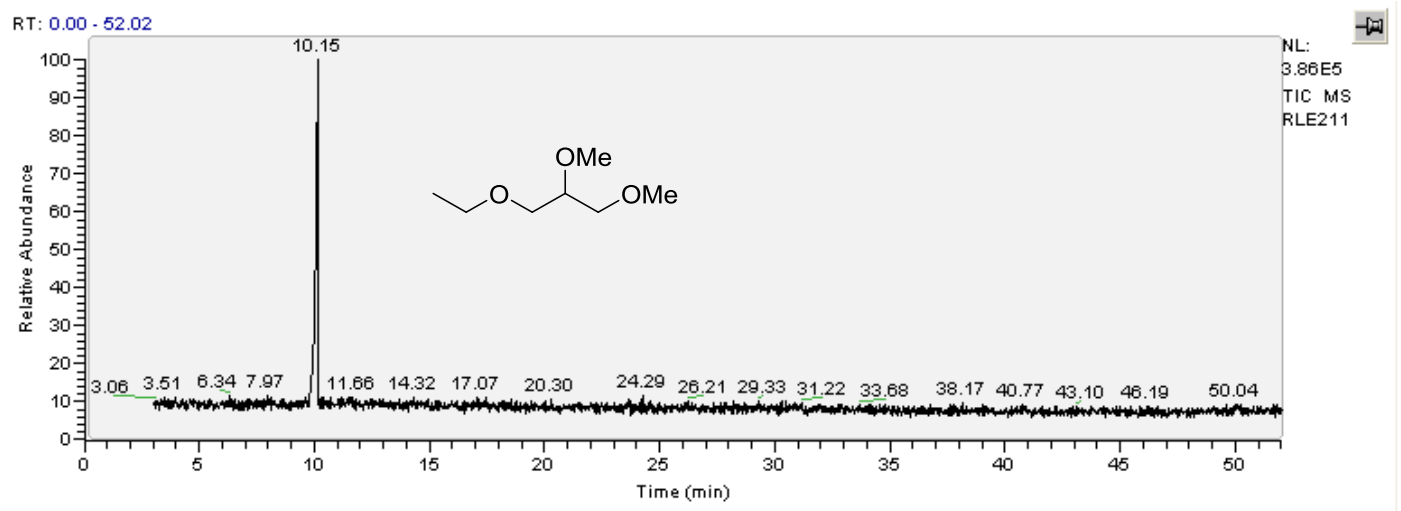

RT: 0.00 - 52.03
NL:
3.49E6
TIC MS
RLE140
Relative Abundance
Time (min)
19.73
3.16
4.55
8.05
9.76
12.37
18.52
19.89
24.55
29.06
31.52
34.41
39.28
42.70
47.70
51.14
OMe
OH
O

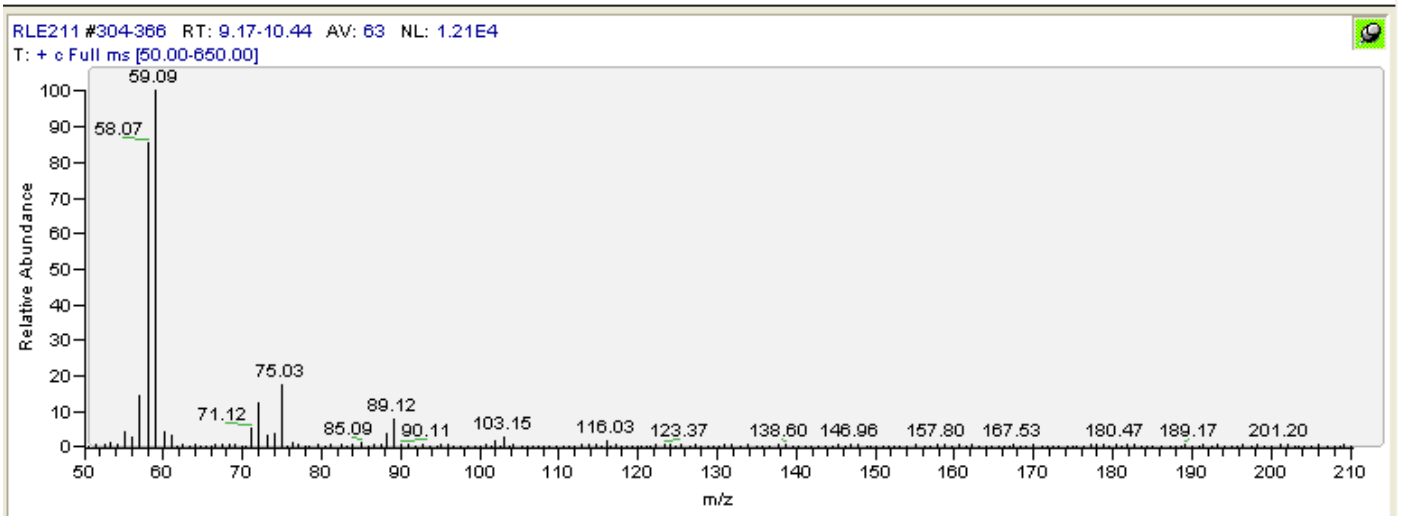

RLE140 #767-848 RT: 18.61-20.26 AV: 82 NL: 1.41E5
T: + c Full ms [50.00-650.00]
Relative Abundance
m/z
57.08
61.05
75.04
71.06
85.10
90.14
100.12
101.10
117.10
118.13
131.13
136.61
148.81
163.09
168.89
186.34
193.01
205.20

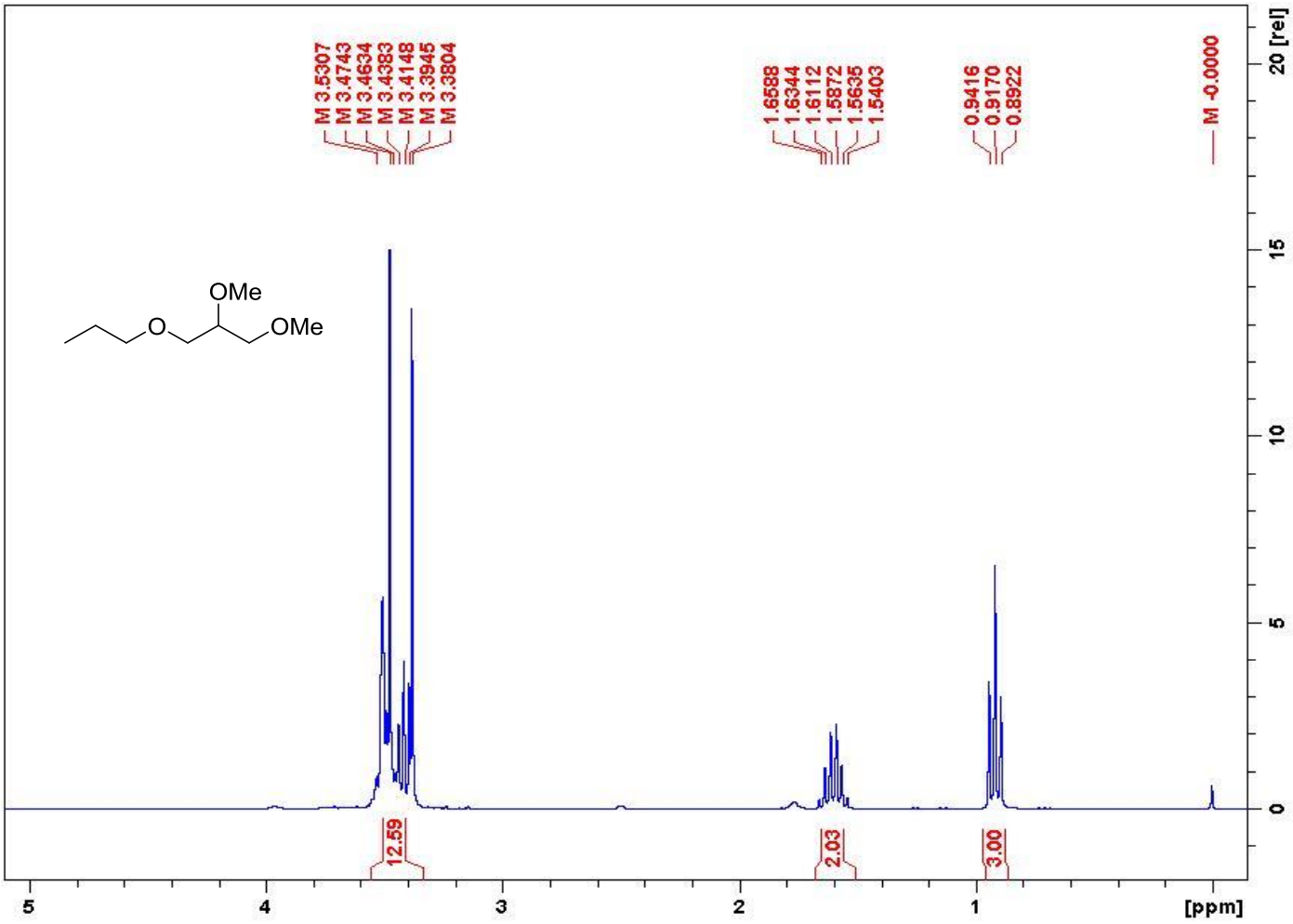

M 3.5572
M 3.4735
M 3.3807
M 1.2305
M 1.2070
M 1.1839
M -0.0000
OMe
O
OMe
10.04
3.20
3.00
[rel]
[ppm]

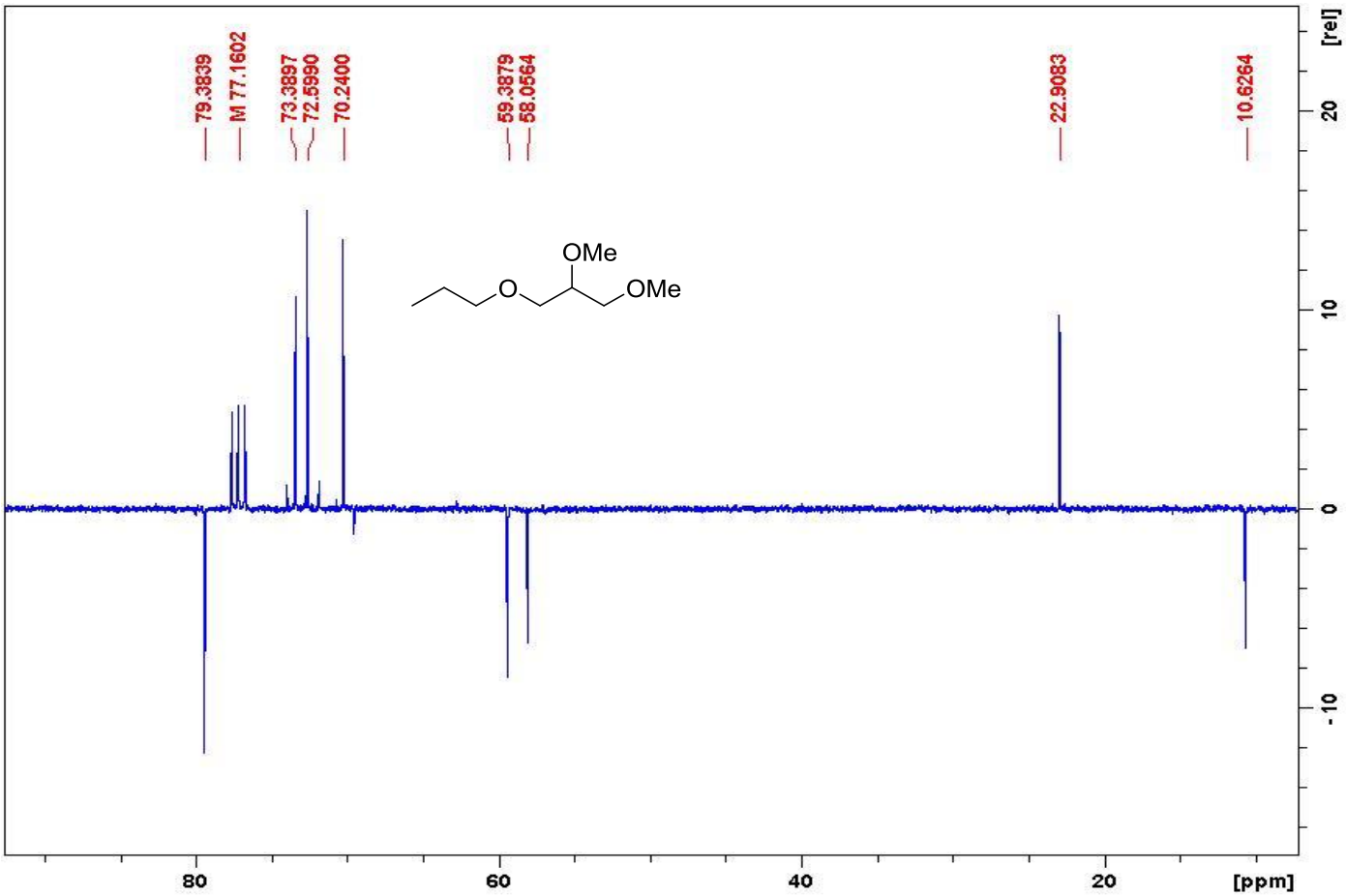

79.32
M 77.16
72.48
70.02
66.93
59.33
57.97
15.18
OMe
O
OMe
[rel]
[ppm]

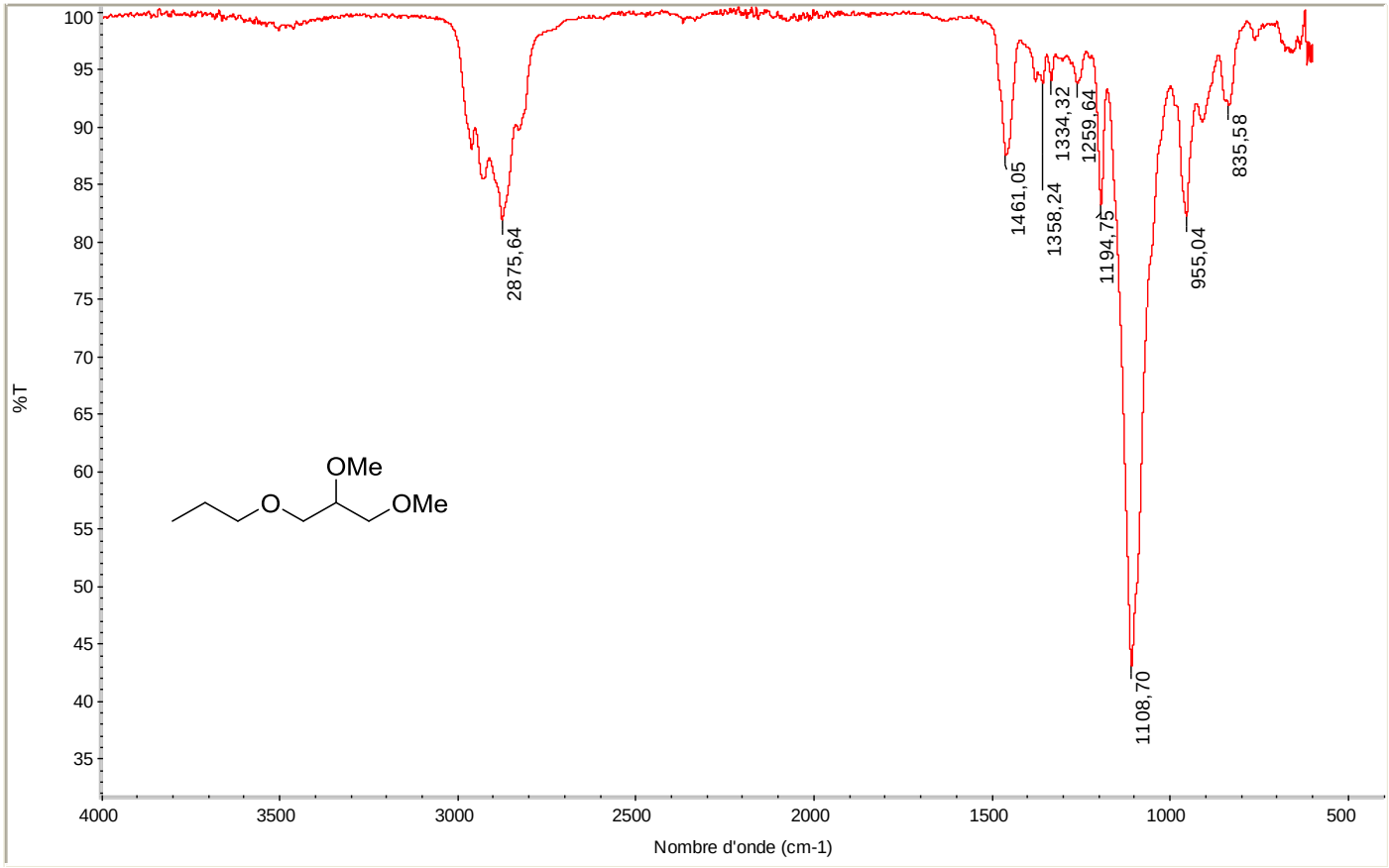

%T
Nombre d'onde (cm-1)
2976,42
2873,15
1457,86
1379,36
1334,11
1194,59
1110,59
961,11
883,37
848,88
OMe
O
OMe

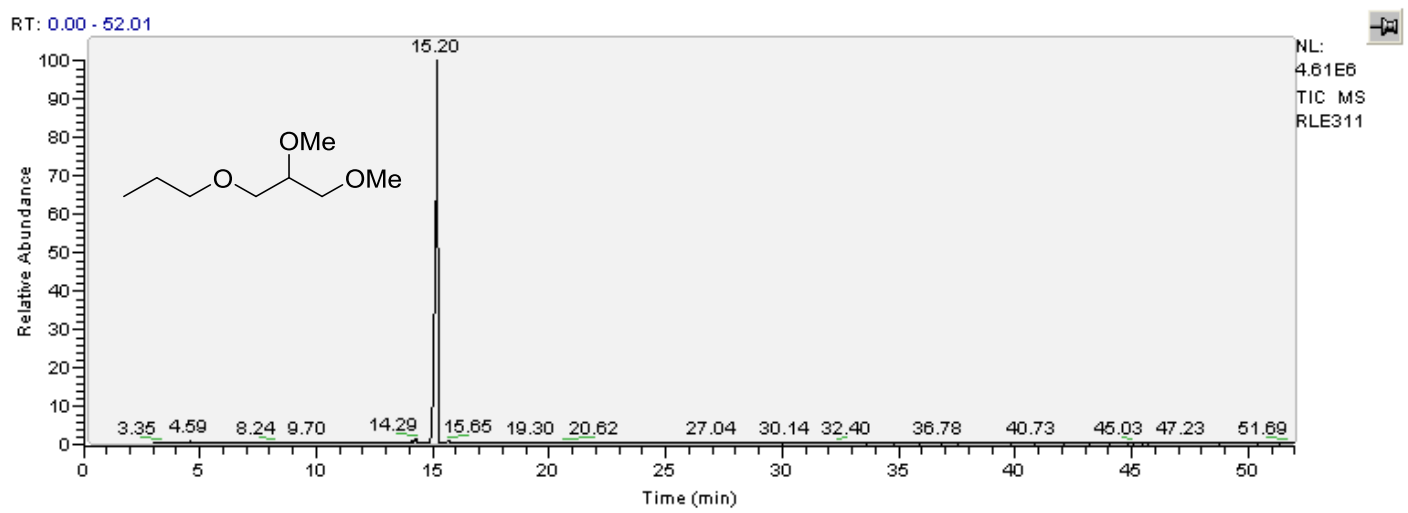

RT: 0.00 - 52.02
NL:
3.86E5
TIC MS
RLE211
Relative Abundance
Time (min)
10.15
3.06
3.51
6.34
7.97
11.66
14.32
17.07
20.30
24.29
26.21
29.33
31.22
33.68
38.17
40.77
43.10
46.19
50.04
OMe
O
OMe

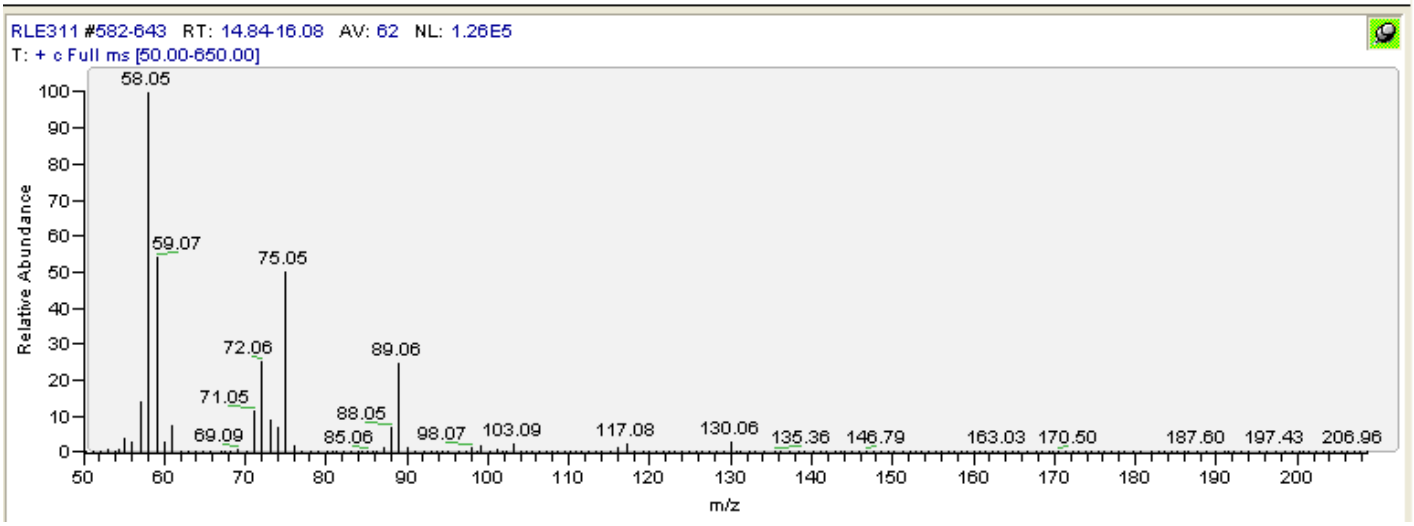

RLE211 #304-366 RT: 9.17-10.44 AV: 63 NL: 1.21E4
T: + c Full ms [50.00-650.00]
Relative Abundance
m/z
59.09
58.07
75.03
71.12
89.12
85.09
90.11
103.15
116.03
123.37
138.60
146.96
157.80
167.53
180.47
189.17
201.20

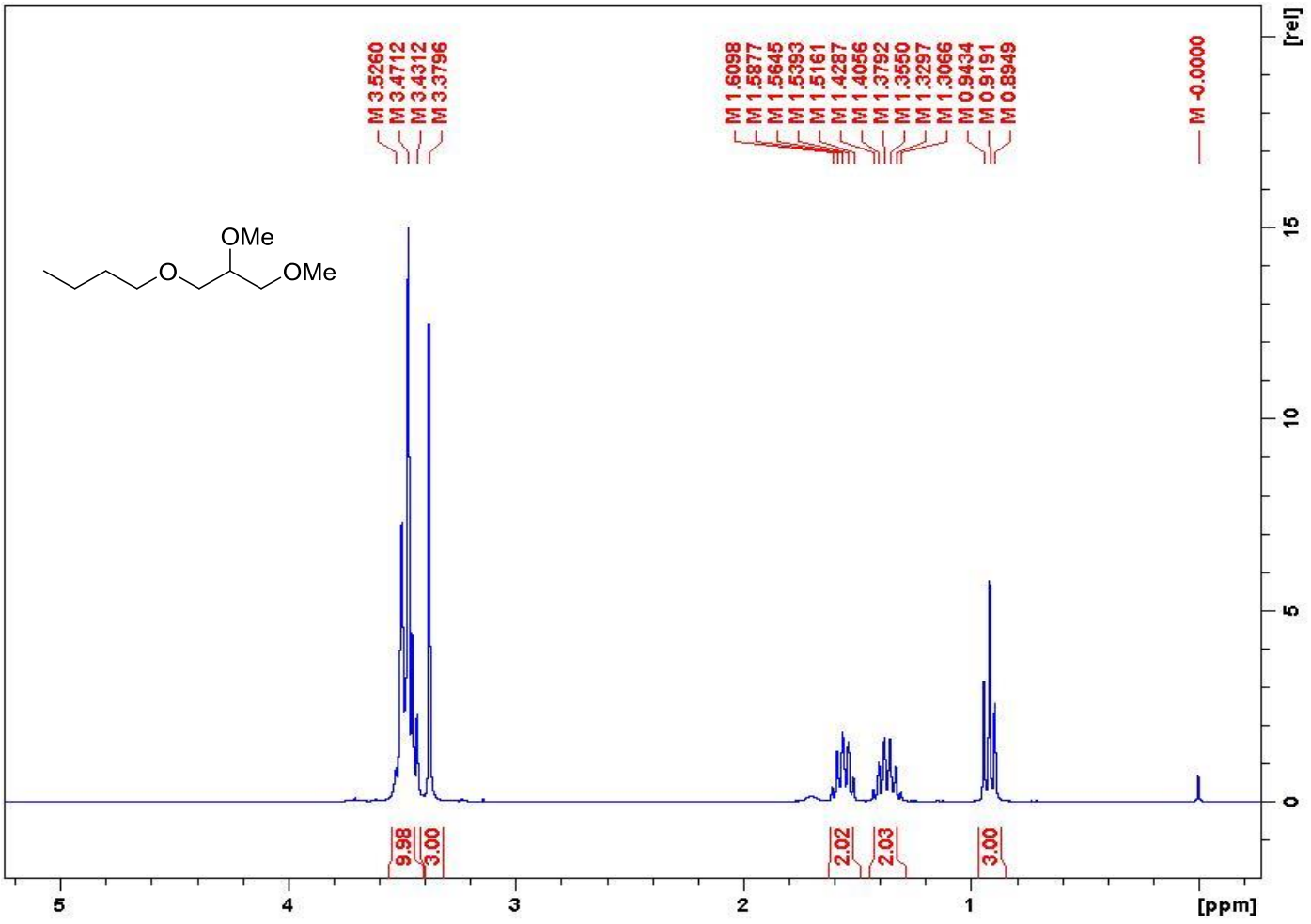

M 3.5307
M 3.4743
M 3.4634
M 3.4383
M 3.4148
M 3.3945
M 3.3804
1.6588
1.6344
1.6112
1.5872
1.5635
1.5403
0.9416
0.9170
0.8922
M -0.0000
OMe
O
OMe
12.59
2.03
3.00
20 [rel]
15
10
5
0
5
4
3
2
1
[ppm]

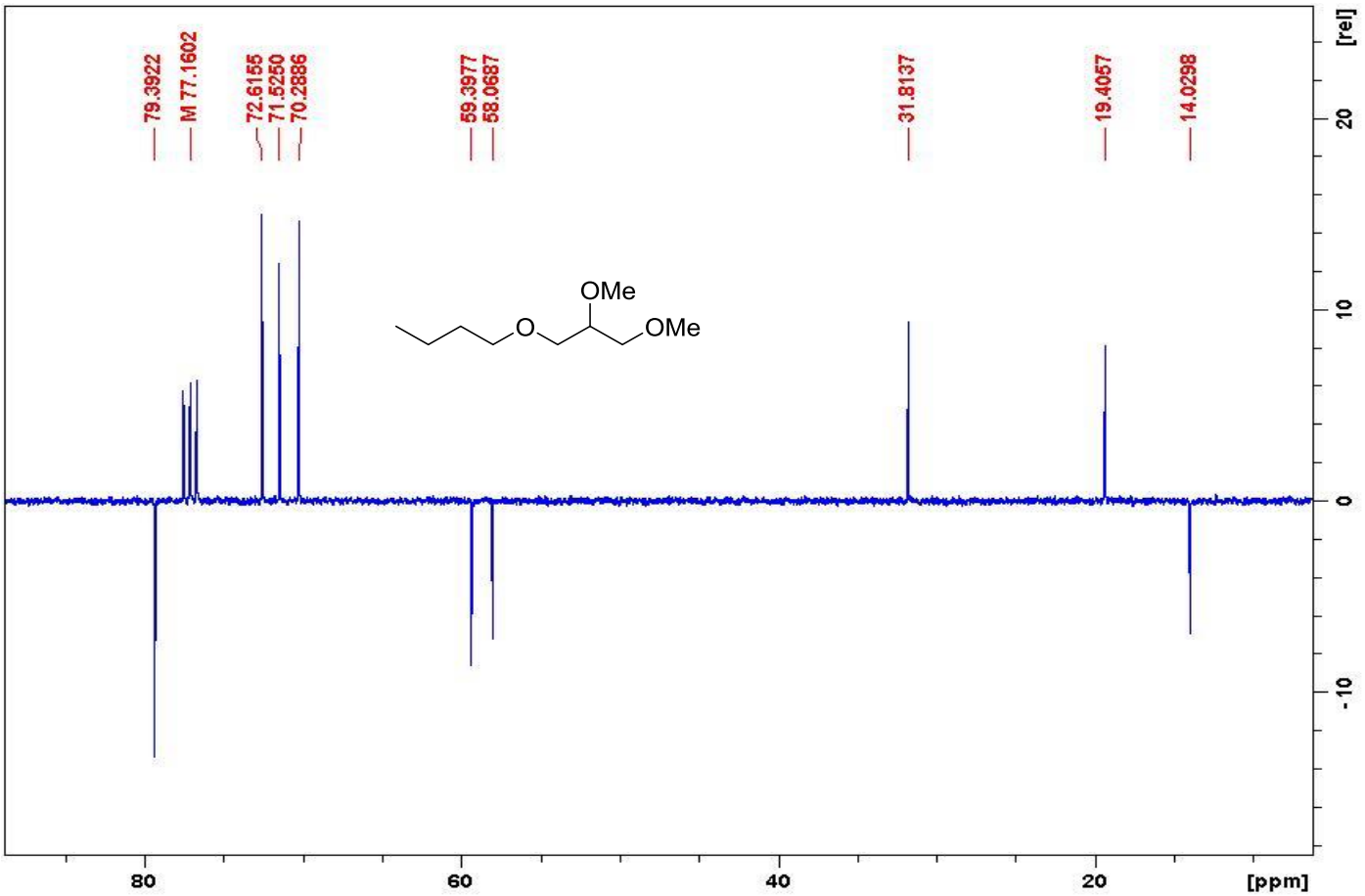

79.3839
M 77.1602
73.3897
72.5990
70.2400
59.3879
58.0564
22.9083
10.6264
OMe
O
OMe
[rel]
20
10
0
-10
80
60
40
20
[ppm]

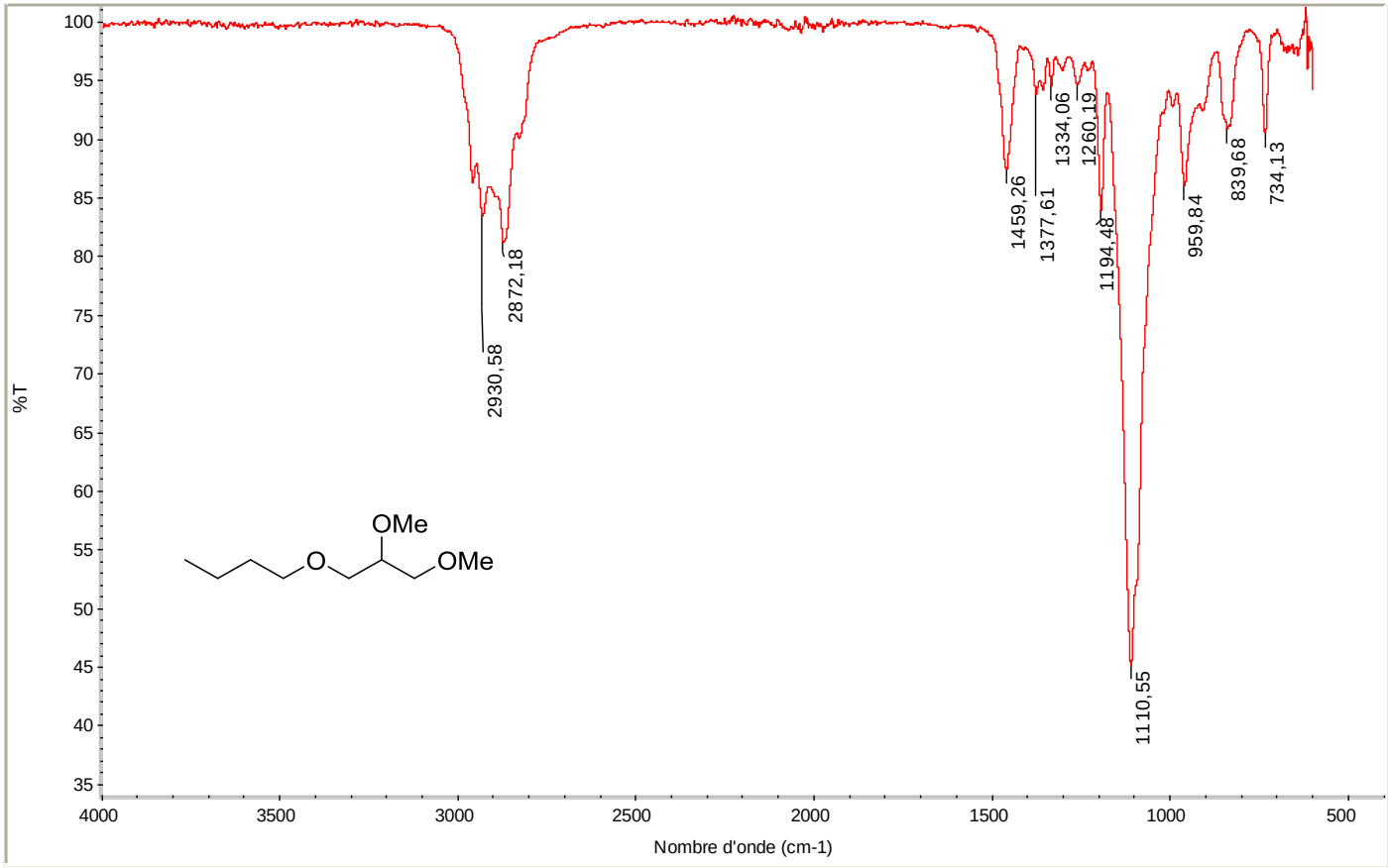

%T
Nombre d'onde (cm-1)
2875,64
1461,05
1358,24
1334,32
1259,64
1194,75
1108,70
955,04
835,58
OMe
O
OMe

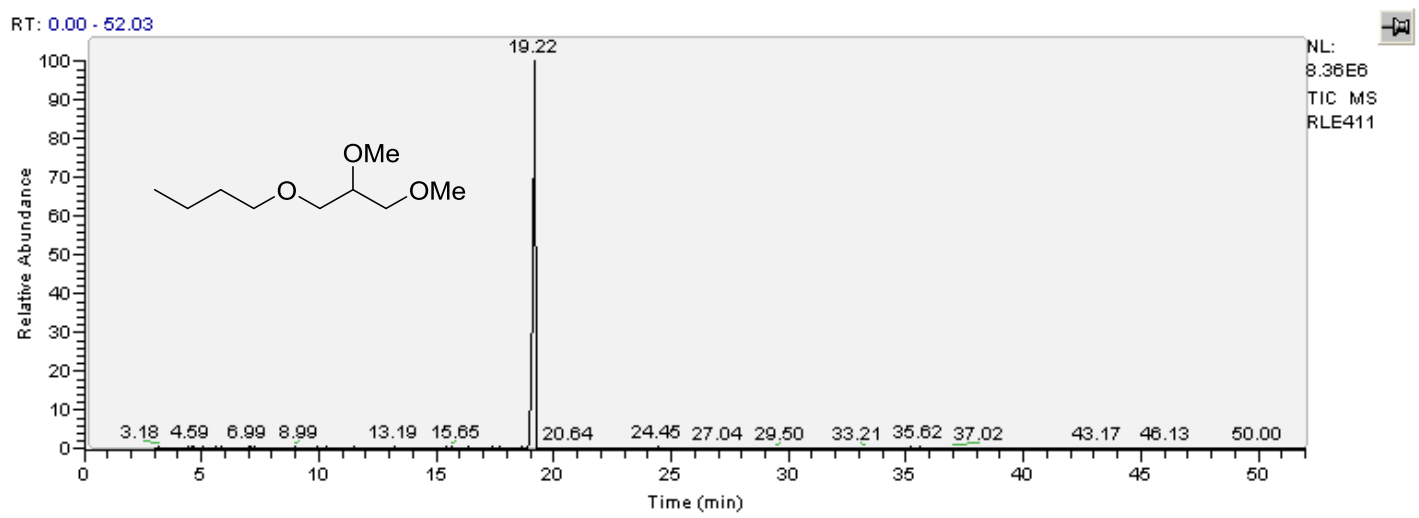

RT: 0.00 - 52.01
NL:
4.61E6
TIC MS
RLE311
Relative Abundance
Time (min)
15.20
3.35 4.59 8.24 9.70 14.29 15.65 19.30 20.62 27.04 30.14 32.40 36.78 40.73 45.03 47.23 51.69
OMe
O
OMe

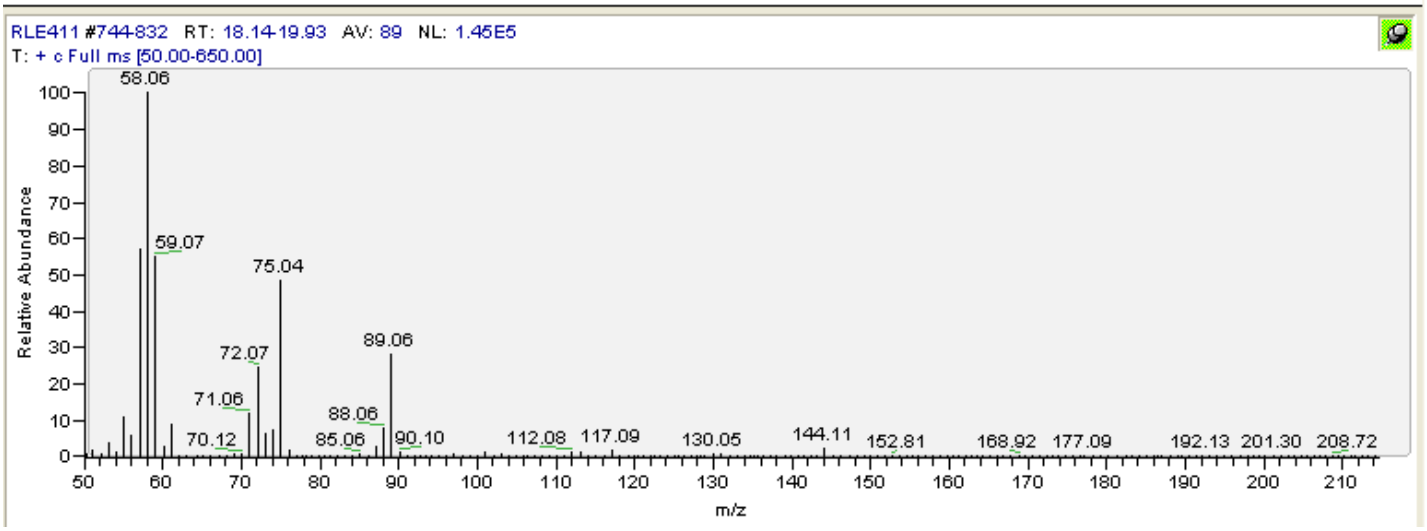

RLE311 #582-643 RT: 14.84-16.08 AV: 62 NL: 1.26E5
T: + c Full ms [50.00-650.00]
Relative Abundance
m/z
58.05
59.07
75.05
72.06
89.06
71.05
88.05
69.09
85.06
98.07
103.09
117.08
130.06
135.36
146.79
163.03
170.50
187.60
197.43
206.96

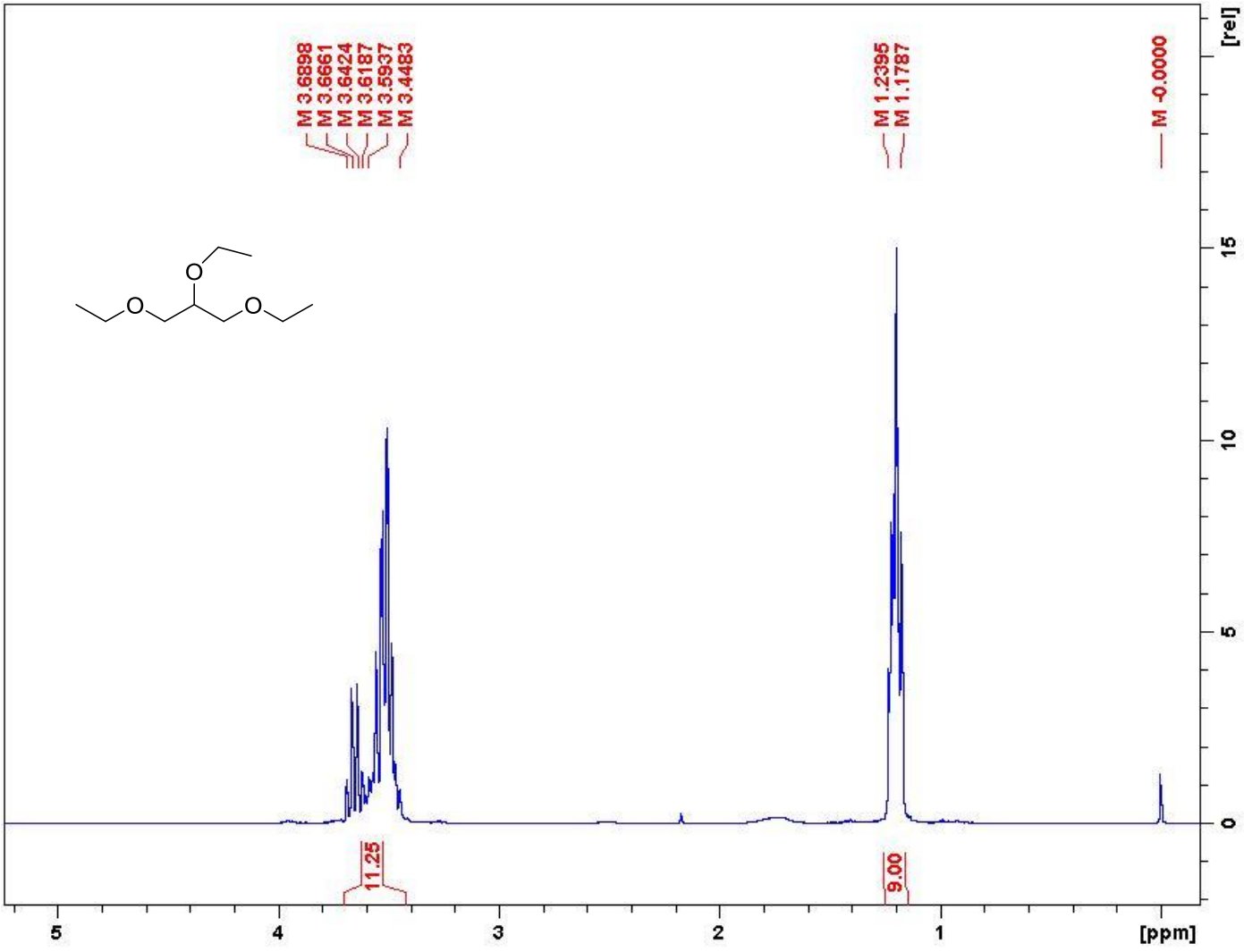

M 3.5260
M 3.4712
M 3.4312
M 3.3796
M 1.6098
M 1.5877
M 1.5645
M 1.5393
M 1.5161
M 1.4287
M 1.4056
M 1.3792
M 1.3550
M 1.3297
M 1.3066
M 0.9434
M 0.9191
M 0.8949
M -0.0000
OMe
O
OMe
9.98
3.00
2.02
2.03
3.00
[rel]
[ppm]

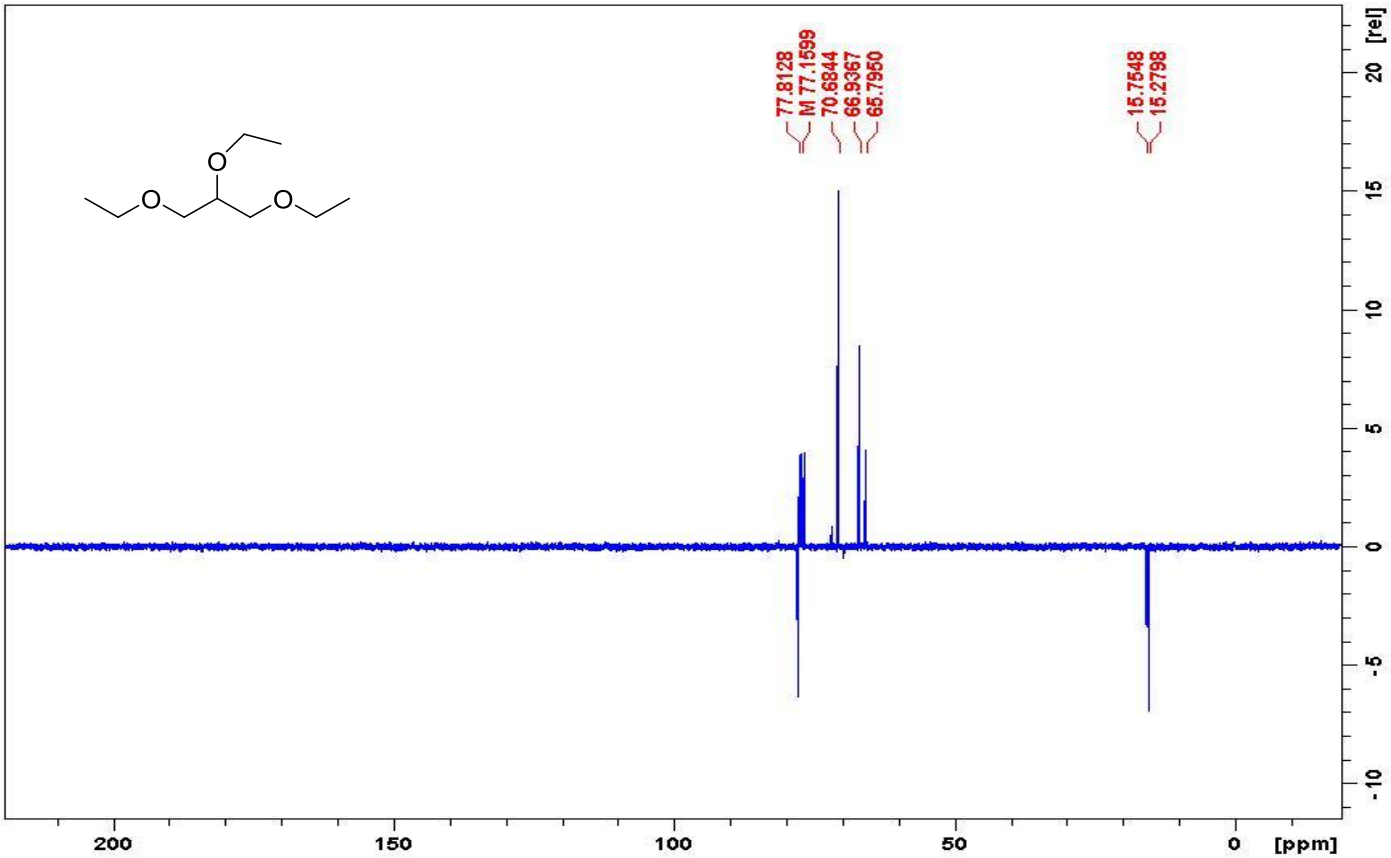

79.3922
M 77.1602
72.6155
71.5250
70.2886
59.3977
58.0687
31.8137
19.4057
14.0298
OMe
O
OMe
[rel]
[ppm]

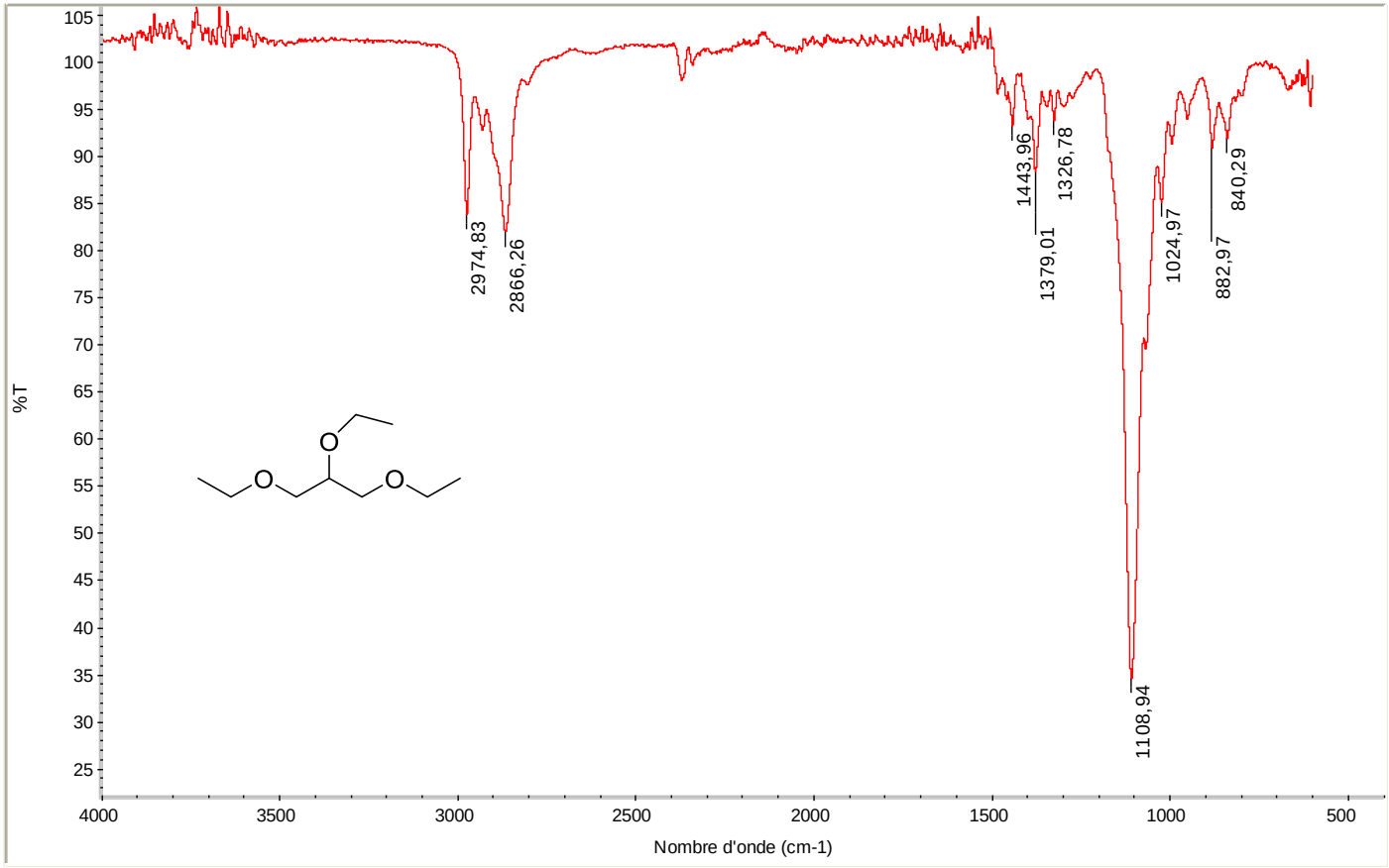

%T
Nombre d'onde (cm-1)
2930,58
2872,18
1459,26
1377,61
1334,06
1260,19
1194,48
1110,55
959,84
839,68
734,13
OMe
O
OMe

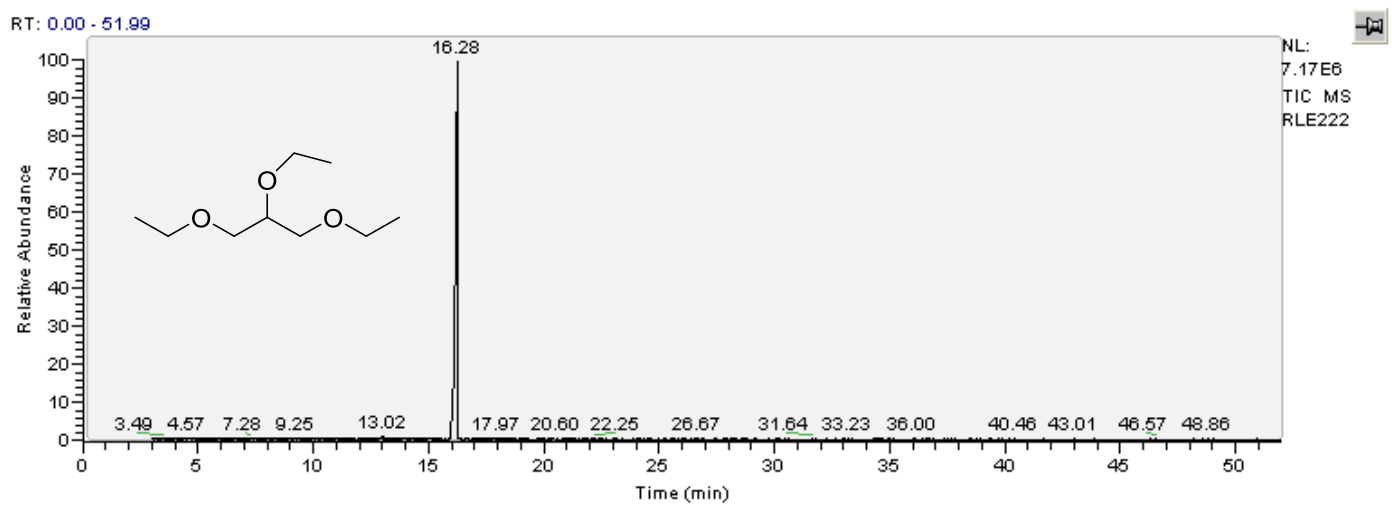

RT: 0.00 - 52.03
NL:
8.36E6
TIC MS
RLE411
Relative Abundance
Time (min)
19.22
OMe
O
OMe

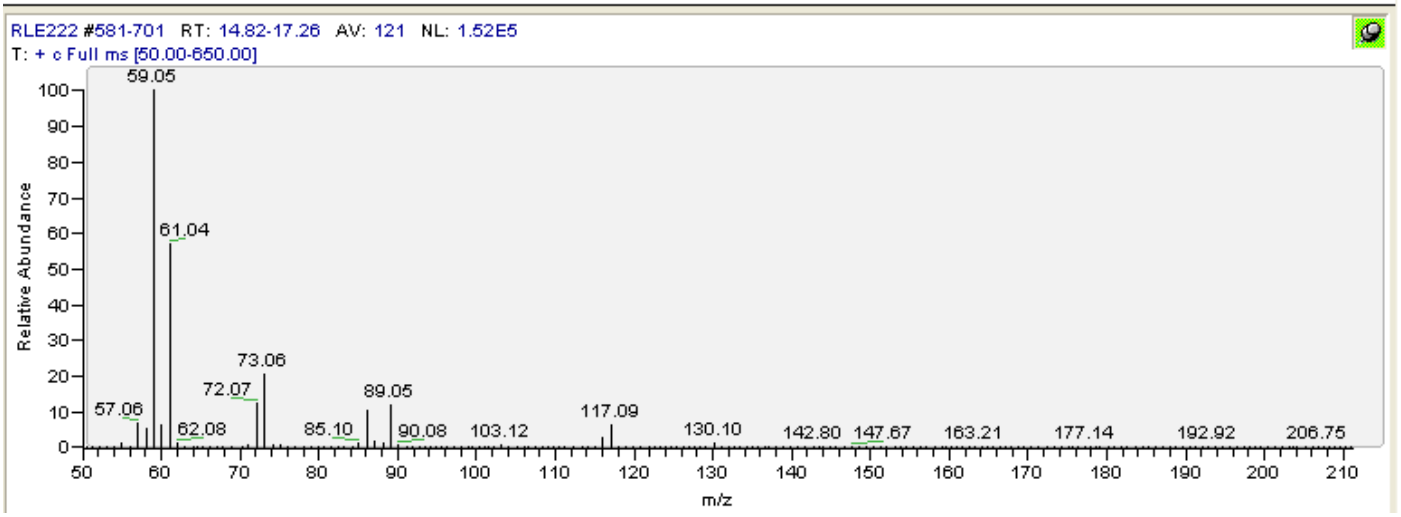

RLE411 #744-832 RT: 18.14-19.93 AV: 89 NL: 1.45E5
T: + c Full ms [50.00-650.00]
Relative Abundance
m/z
58.06
59.07
75.04
72.07
89.06
71.06
88.06
70.12
85.06
90.10


M 3.6898
M 3.6661
M 3.6424
M 3.6187
M 3.5937
M 3.4483
M 1.2395
M 1.1787
M -0.0000
11.25
9.00
[rel]
[ppm]


77.8128
M 77.1599
70.6844
66.9367
65.7950
15.7548
15.2798
[rel]
[ppm]


%T
Nombre d'onde (cm-1)
2974,83
2866,26
1443,96
1379,01
1326,78
1108,94
1024,97
882,97
840,29


RT: 0.00 - 51.99
NL:
7.17E6
TIC MS
RLE222
Relative Abundance
Time (min)
16.28


RLE222 #581-701 RT: 14.82-17.26 AV: 121 NL: 1.52E5
T: + c Full ms [50.00-650.00]
Relative Abundance
m/z
59.05
61.04
73.06
72.07
89.05
57.06
117.09

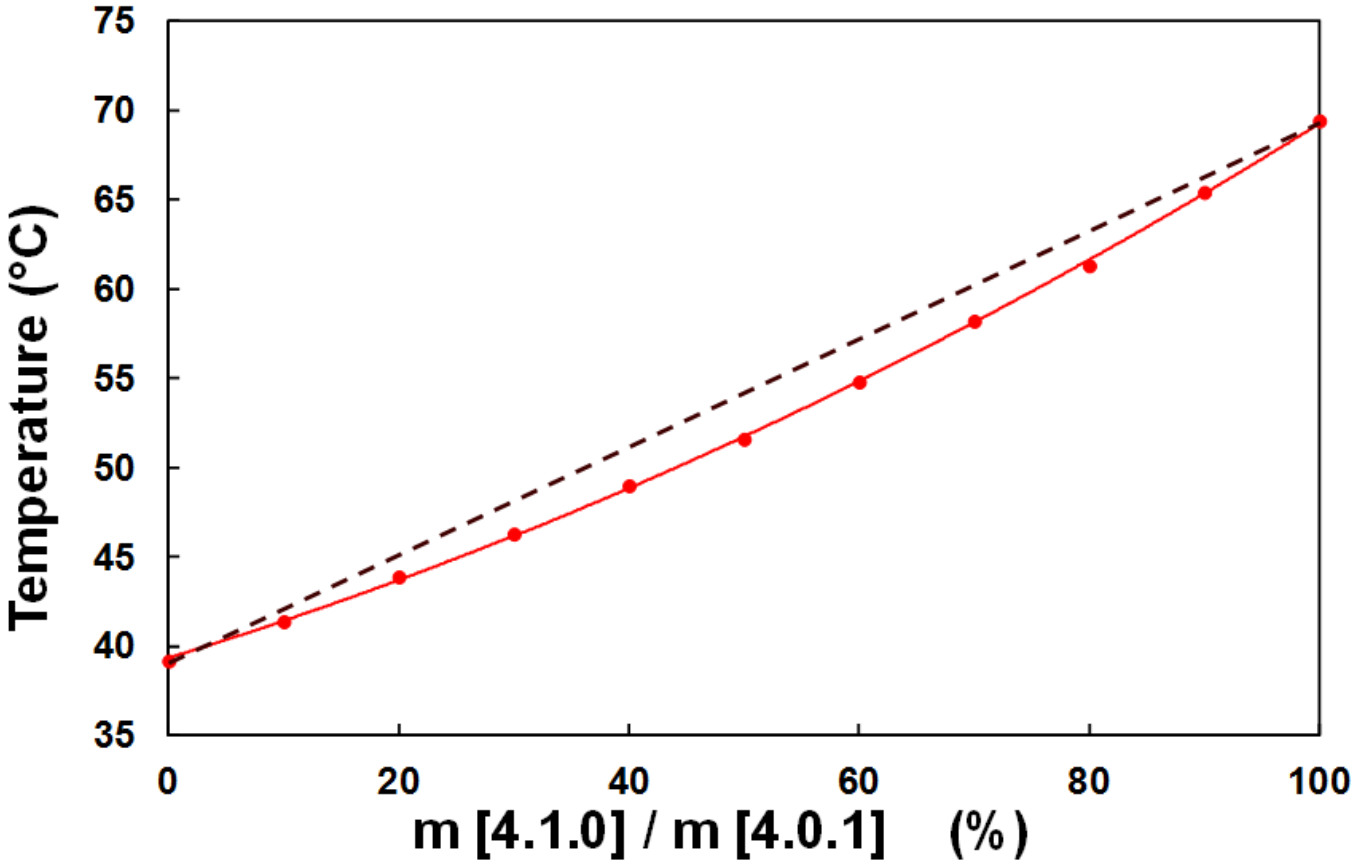


Figure 1. Cloud points of mixtures of [4.1.0] and [4.0.1] isomers at different ratio at 2M (= 24.5 wt%) in water.

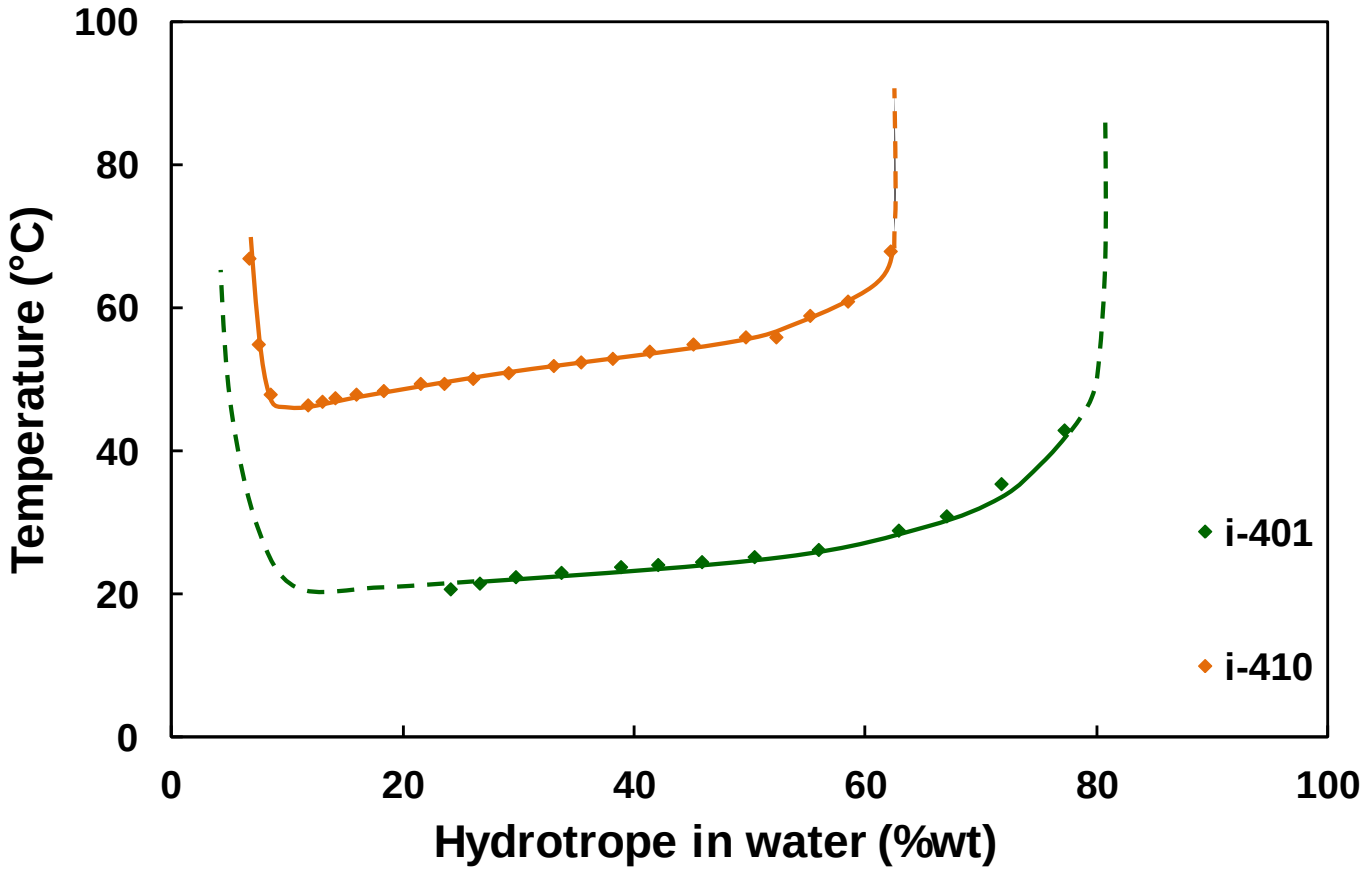


Figure 5. Solubility limits of dialkylglycerol [*i*-4.0.1] and [*i*-4.1.0] in function of the weight fraction of the hydrotrope.

1H NMR spectra in d_6-DMSO of the hydrotrope [4.0.1] (blue), piperine (reference sample, red), and the HCPE extract of piperine from black pepper, after distillation of the hydrotropic solution (green). Piperine is recovered almost pure (still a small residue of hydrotrope and presence of other natural product).