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Fe(III)-catalyzed α-terpinyl derivatives synthesis from β-pinene via reactions with hydrogen peroxide in alcoholic solutions

M. J. da Silva[a], D. M. Carari[b] and Adalberto Manoel da Silva[a]

Suplementar material

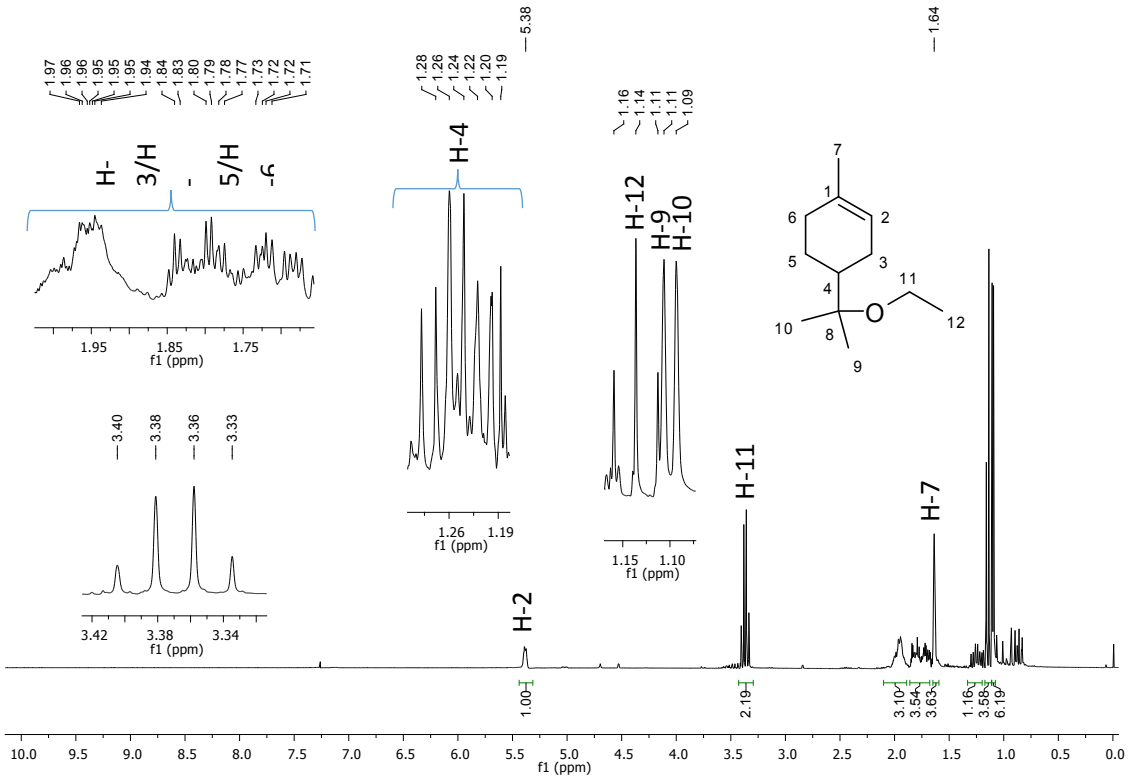


FIGURE 1: NMR ^{1}H of spectrum 4-(2-ethoxypropan-2-yl)-1-methylcyclohex-1-ene ($CDCl_3$, 300 MHz).

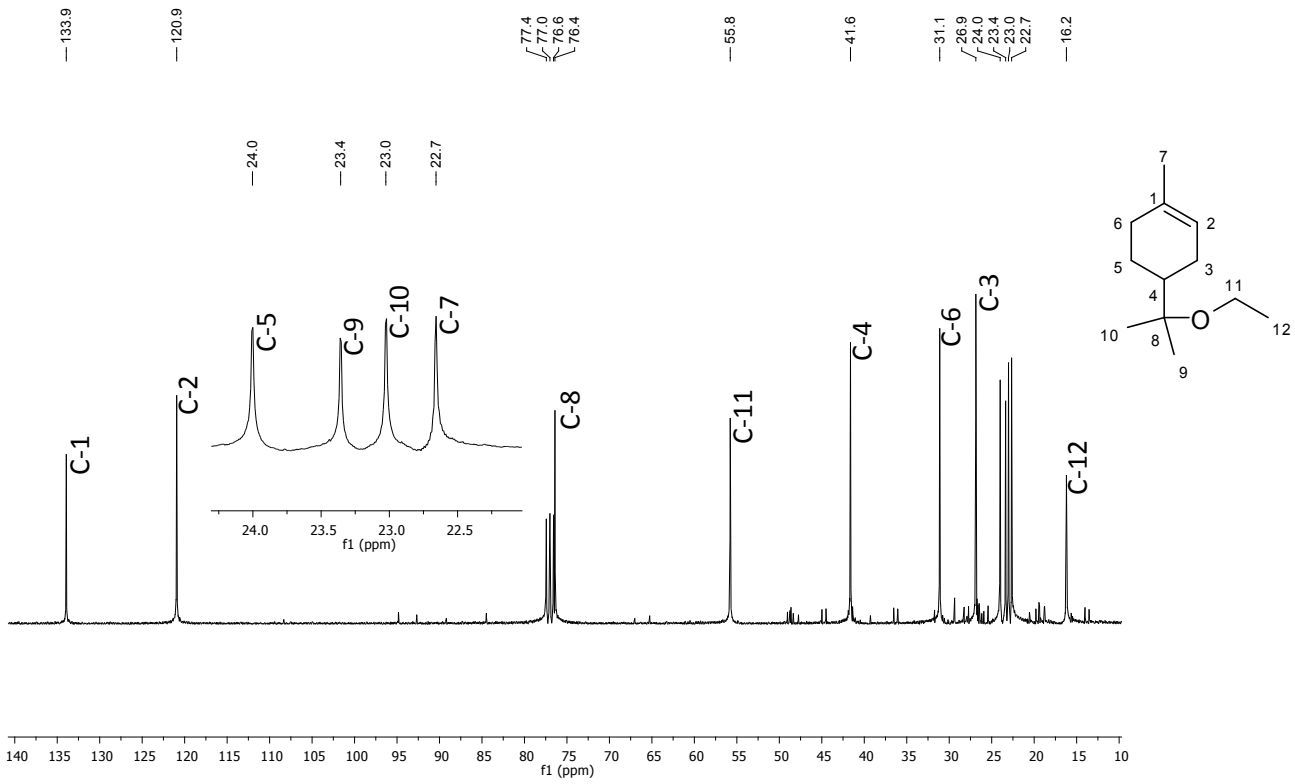


FIGURE 2: NMR ^{13}C spectrum of 4-(2-ethoxypropan-2-yl)-1-methylcyclohex-1-ene ($CDCl_3$, 75 MHz).

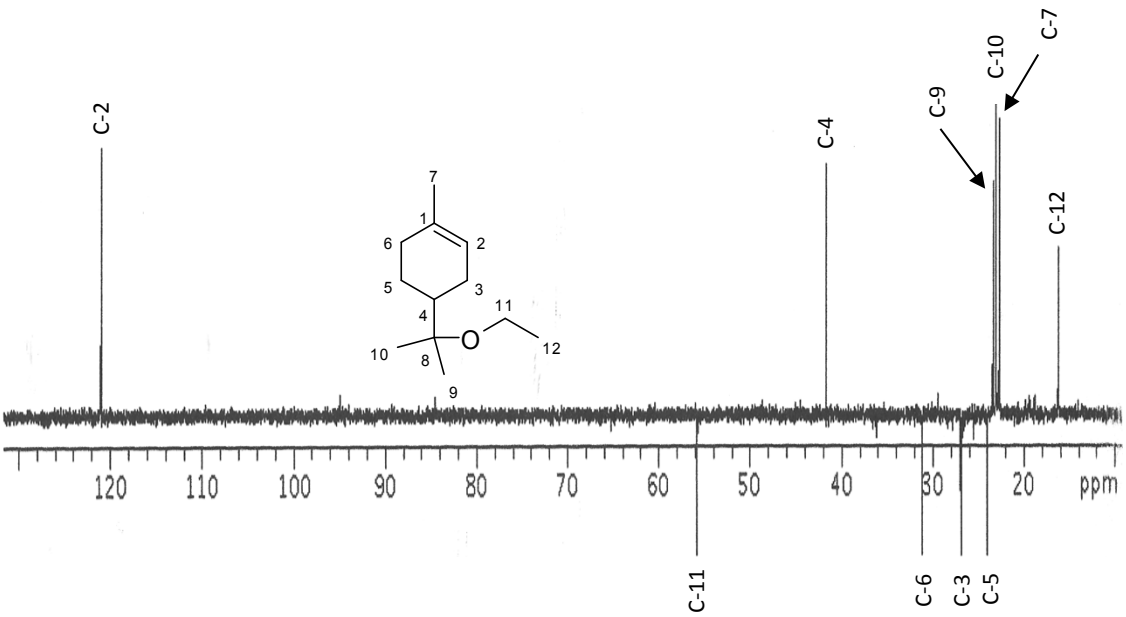


FIGURE 3: DEPT 135 of 4-(2-ethoxypropan-2-yl)-1-methylcyclohex-1-ene.

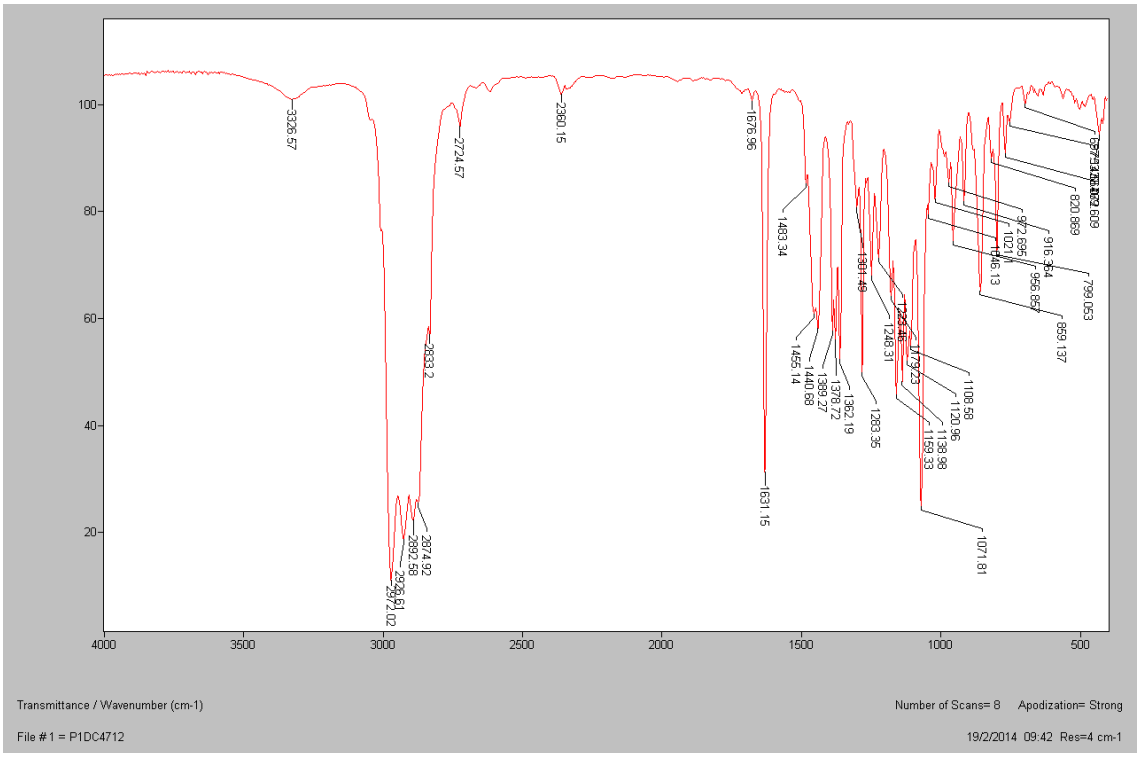


FIGURE 4: Infrared spectrum of 4-(2-ethoxypropan-2-yl)-1-methylcyclohex-1-ene (KBr).

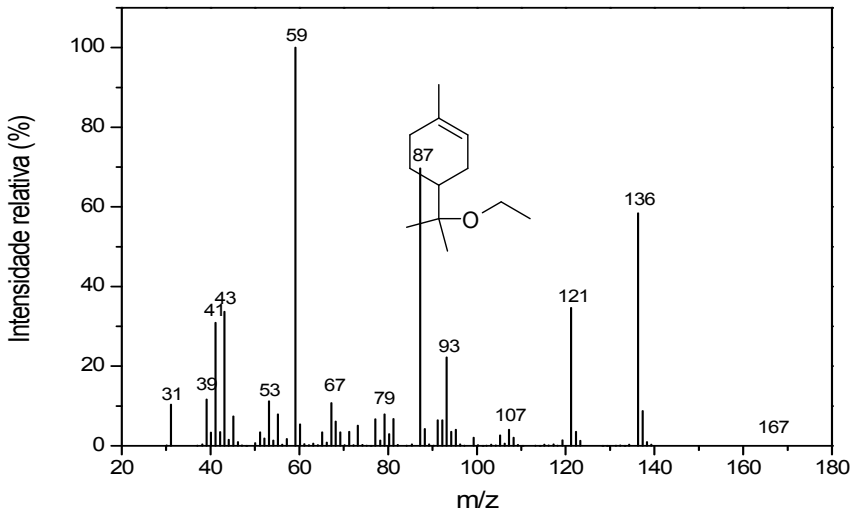
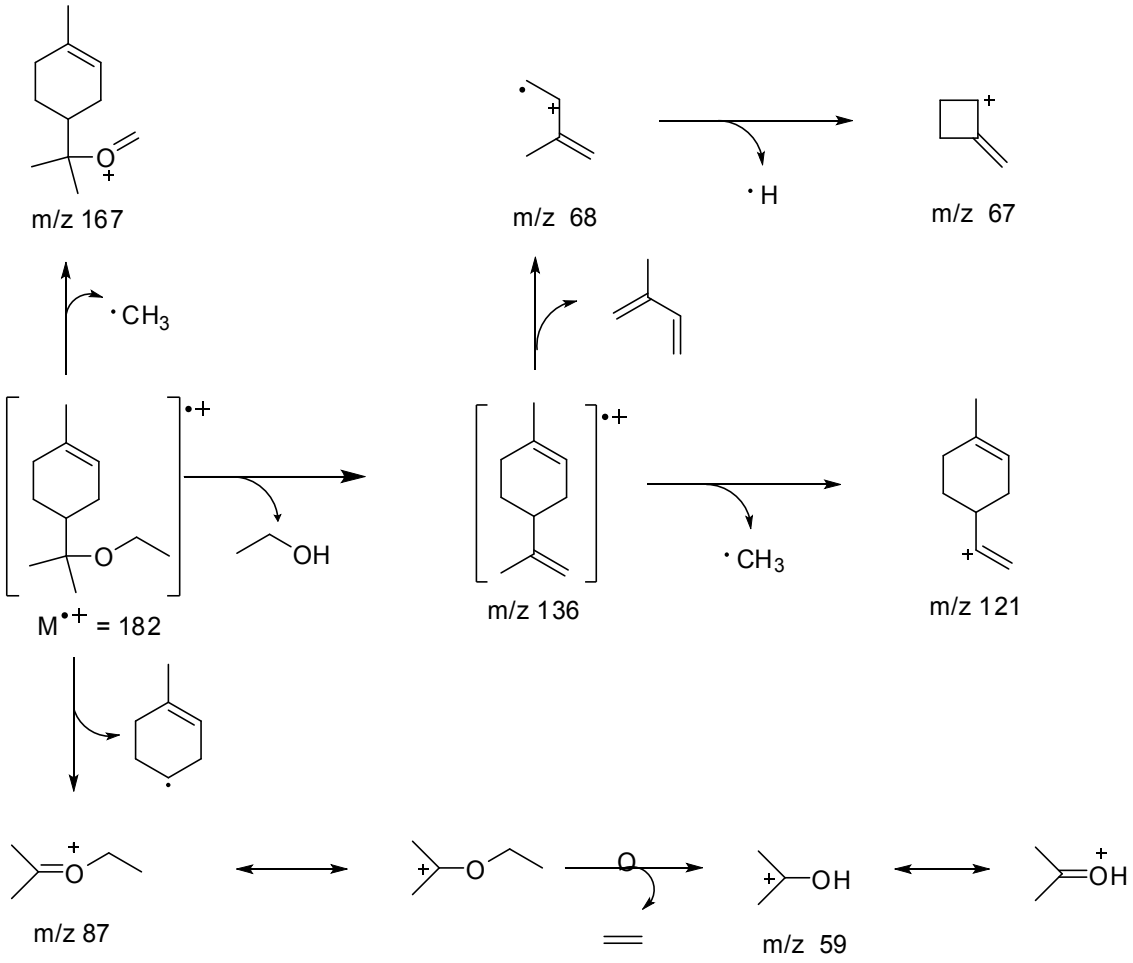


FIGURE 5: Mass spectrum of 4-(2-ethoxypropan-2-yl)-1-methylcyclohex-1-ene (EI - 70 eV).


m/z 167
m/z 68
·H
m/z 67
·CH3
OH
M•+ = 182
m/z 136
·CH3
m/z 121
m/z 87
OH
m/z 59

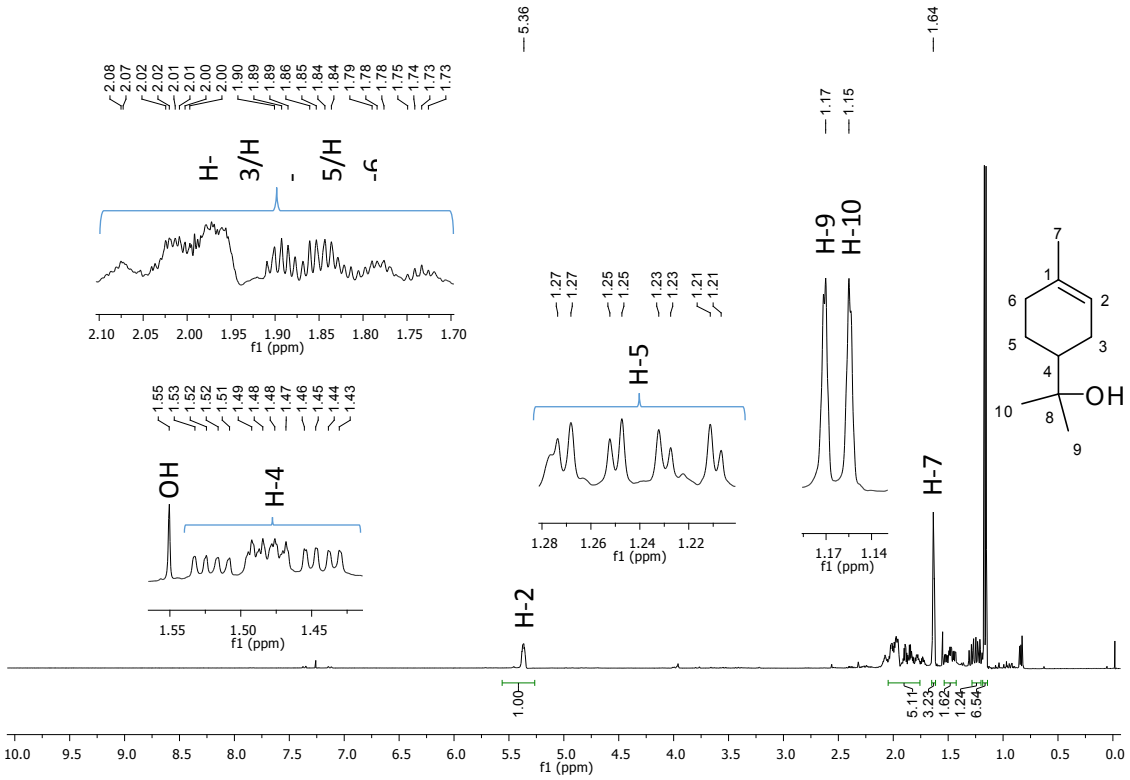


FIGURE 6: NMR ^{1}H spectrum of α-terpineol (2-(4-methylcyclohex-3-enyl)propan-2-ol) ($CDCl_3$, 300 MHz).

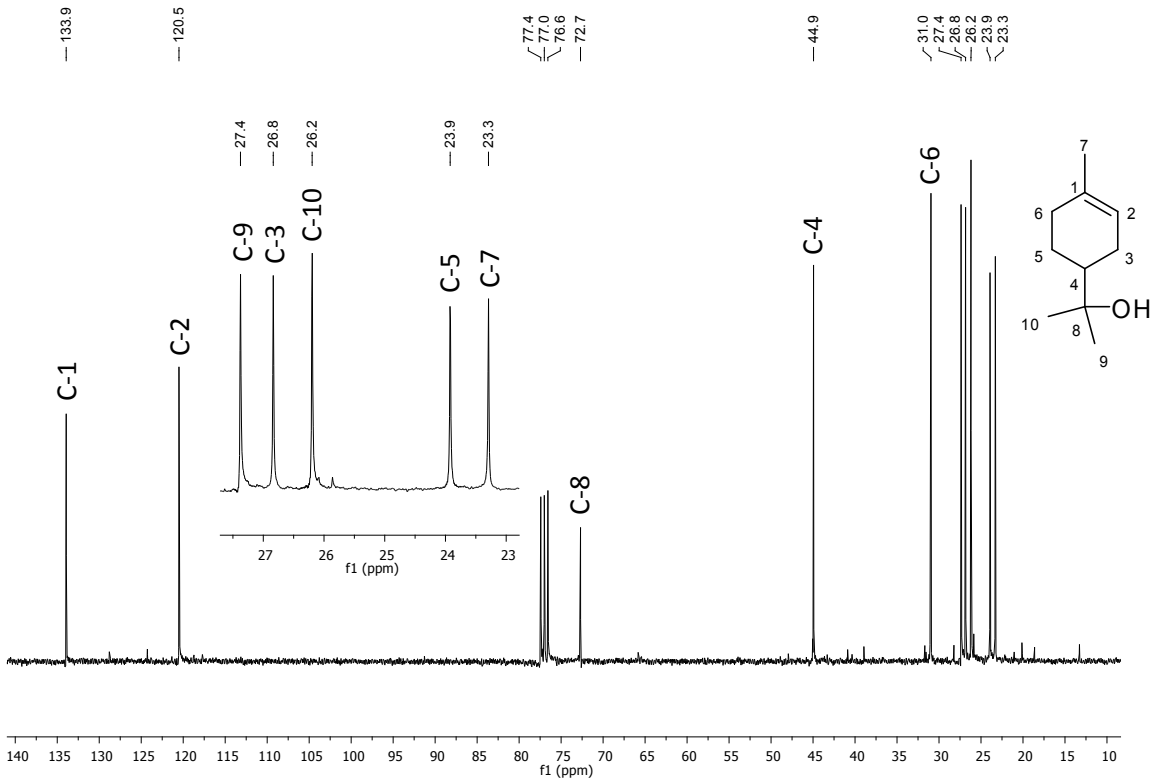


FIGURE 7: NMR ^{13}C spectrum of α -terpineol (2-(4-methylcyclohex-3-enyl)propan-2-ol) ($CDCl_3$, 75 MHz).

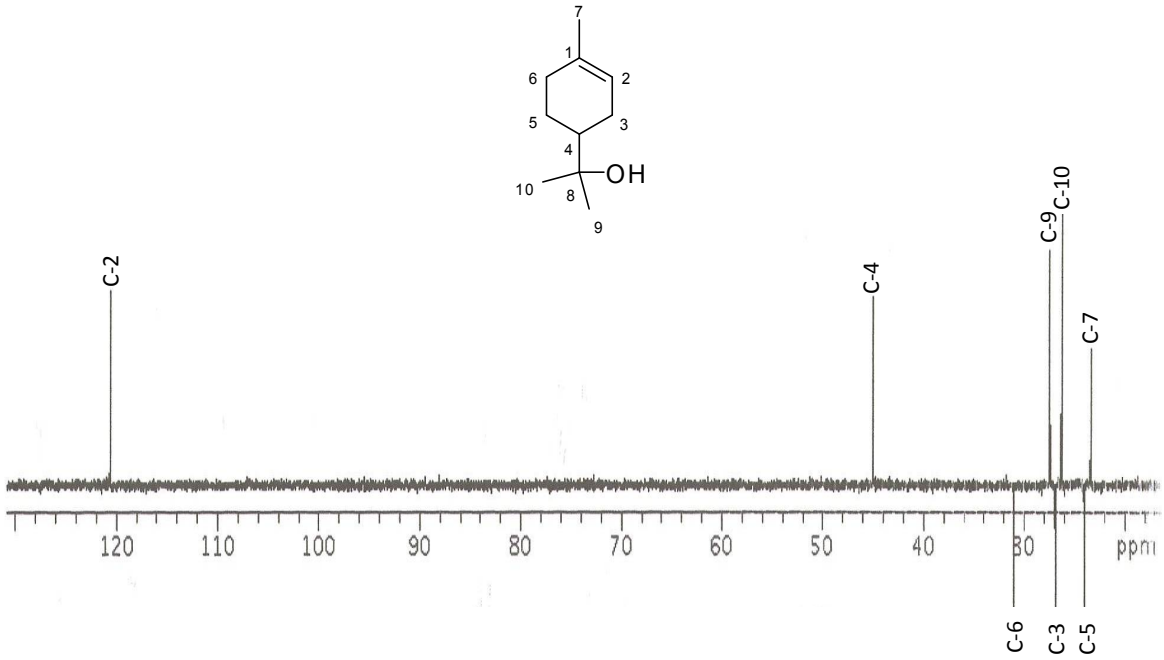


FIGURE 8: DEPT 135 of alpha-terpineol (2-(4-methylcyclohex-3-enyl)propan-2-ol).

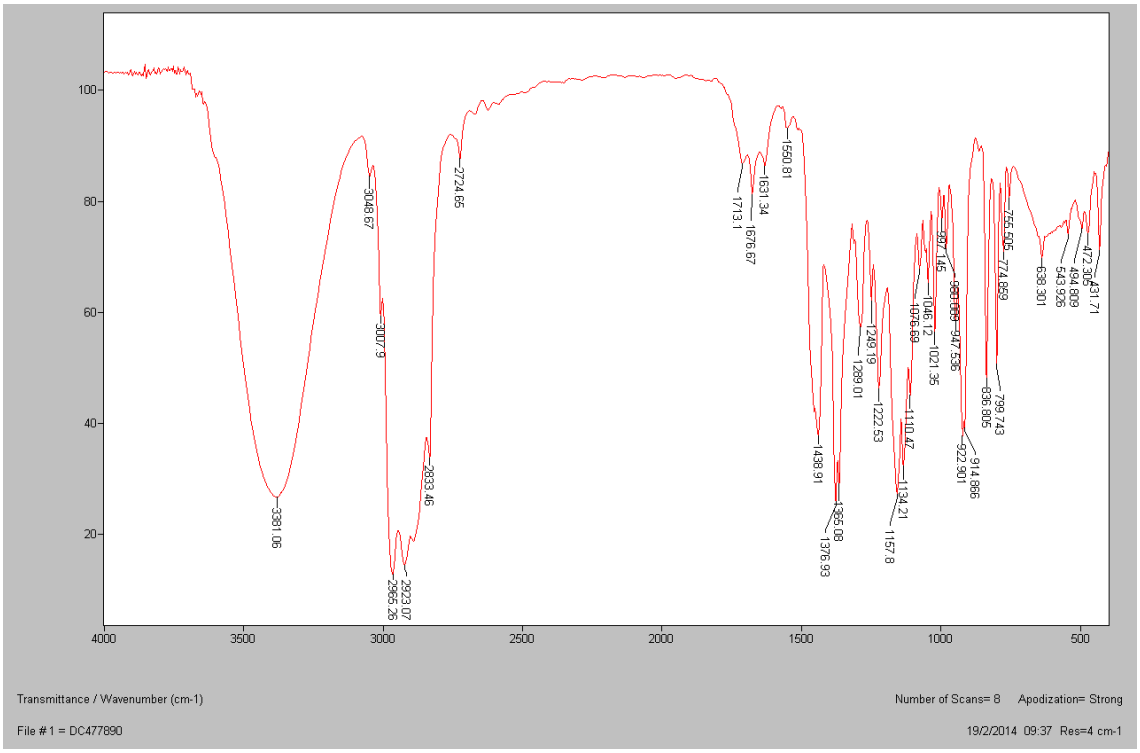


FIGURE 9: Infrared spectrum of alpha-terpineol (2-(4-methylcyclohex-3-enyl)propan-2-ol) (KBr).

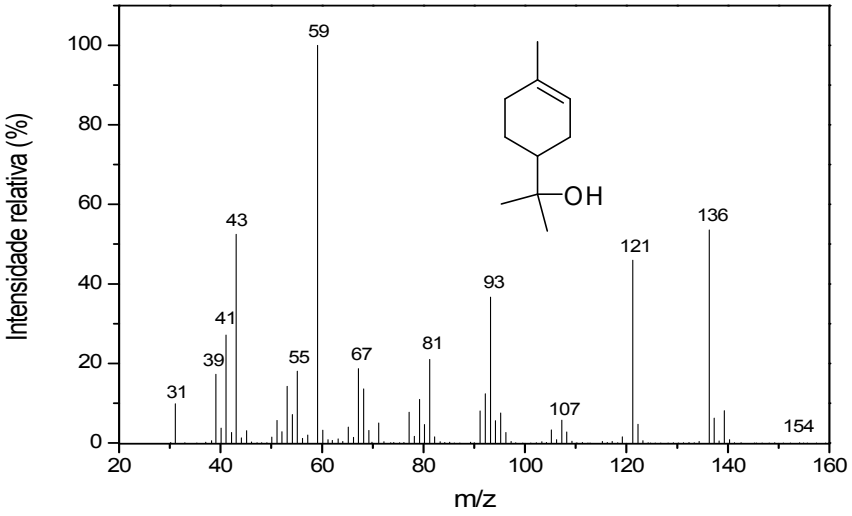
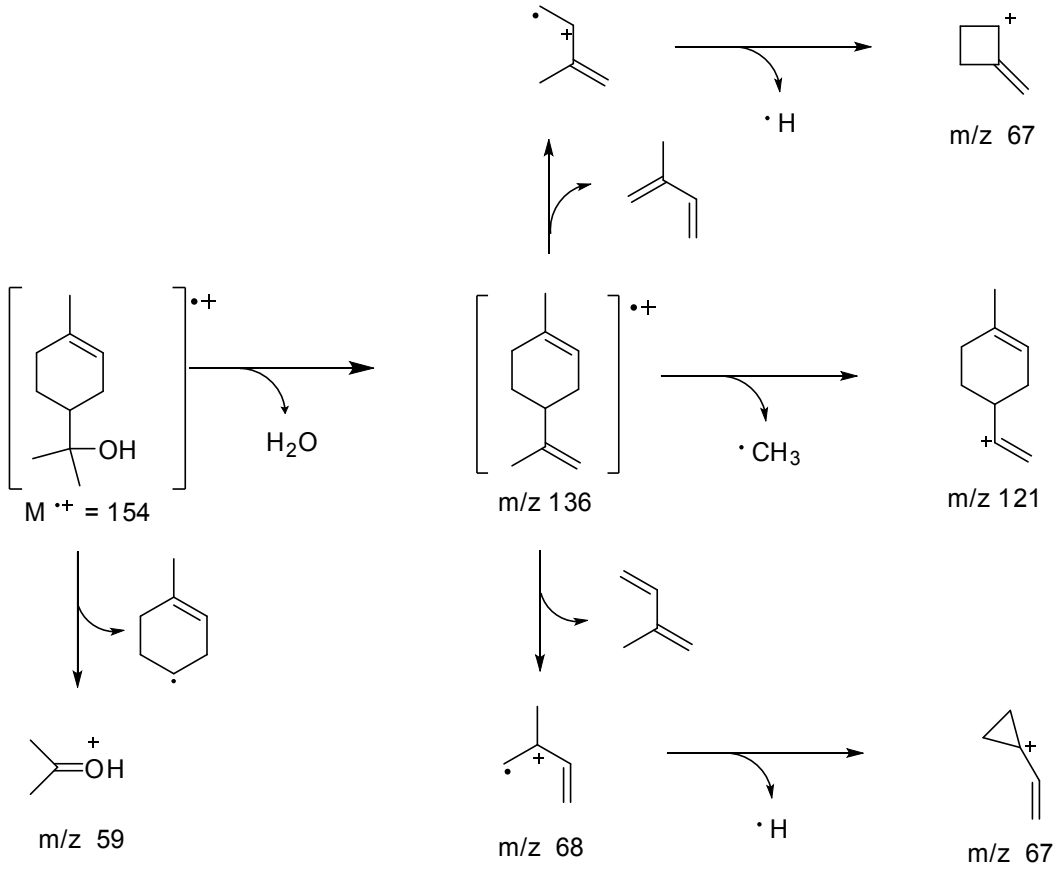
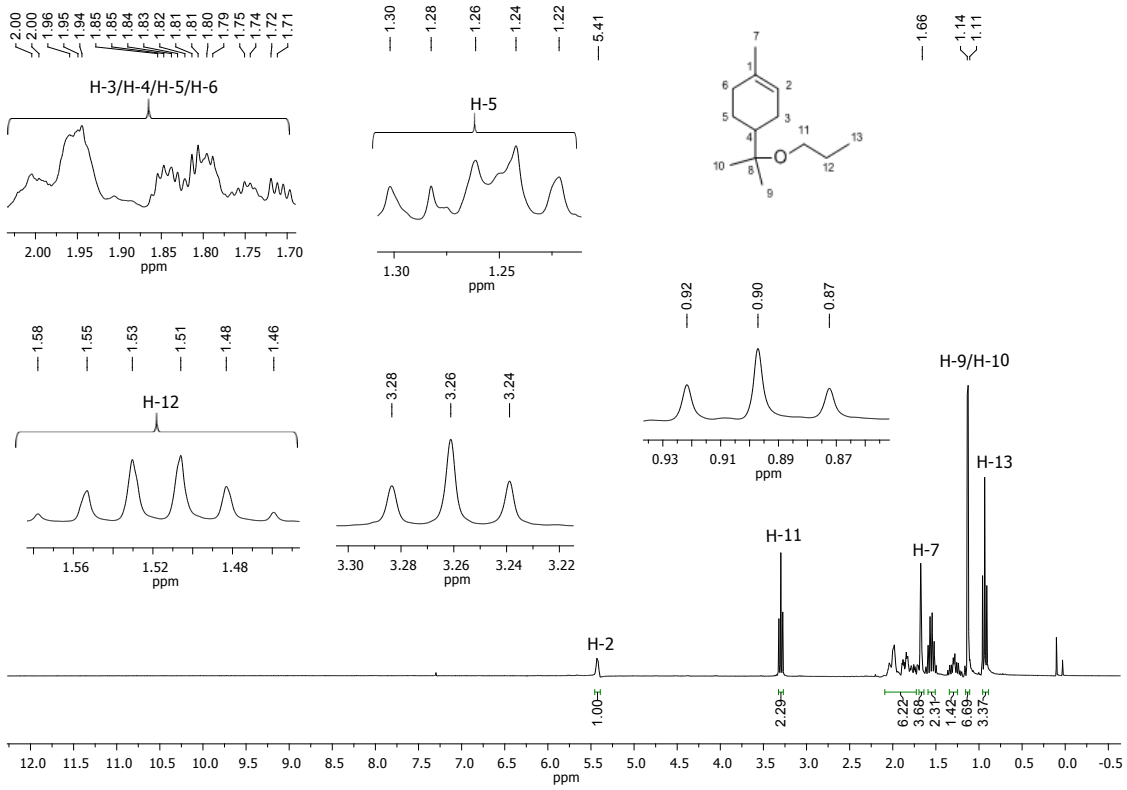


FIGURE 10: Mass spectrum of α-terpineol (2-(4-methylcyclohex-3-enyl)propan-2-ol) (EI - 70 eV).

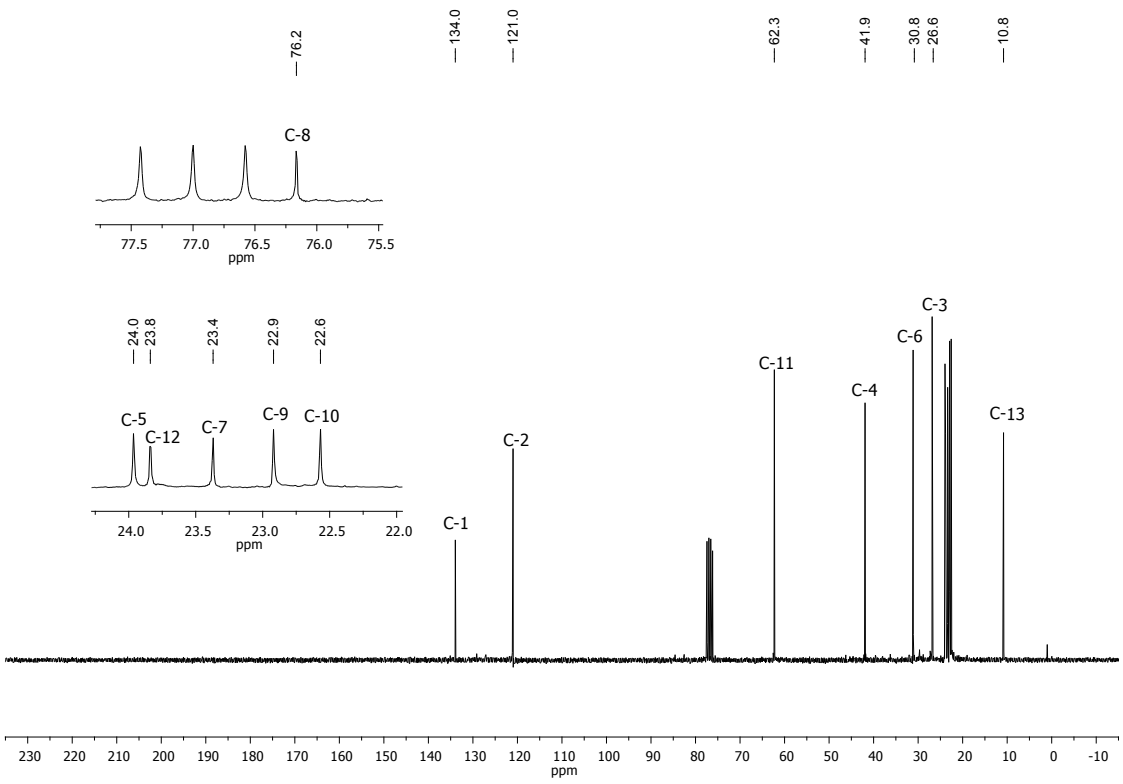


1. ^{1}H NMR of spectrum 1-methyl-4-(2-propoxypropan-2-yl)cyclohex-1-ene ($CDCl_3$, 300 MHz).



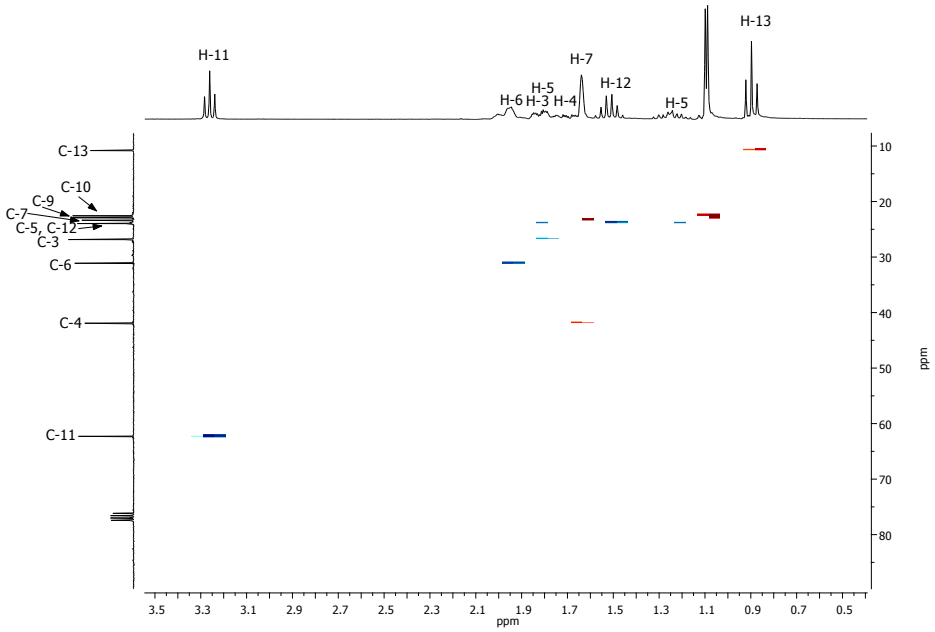
^{1}H NMR (300 MHz, $CDCl_3$): δ 0.90 (t, 3H, J = 7.4 Hz, H-13); 1,11 (s, 3H, H-9 or H-10); 1.14 (s, 3H, H9 or H10); 1,22 – 1,30 (m, 1H, H-5); 1.46 – 1,58 (m, 2H, H-12); 1.66 (s, 3H, H-7); 1.71 – 2.00 (m, 6H, H-3/H-4/H-5/H-6); 3,26 (t, 2H, J = 6,7 Hz, H-11); 5,41 (br s, 1H, H-2).

2. NMR ^{13}C of spectrum 1-methyl-4-(2-propoxypropan-2-yl)cyclohex-1-ene ($CDCl_3$, 75 MHz).

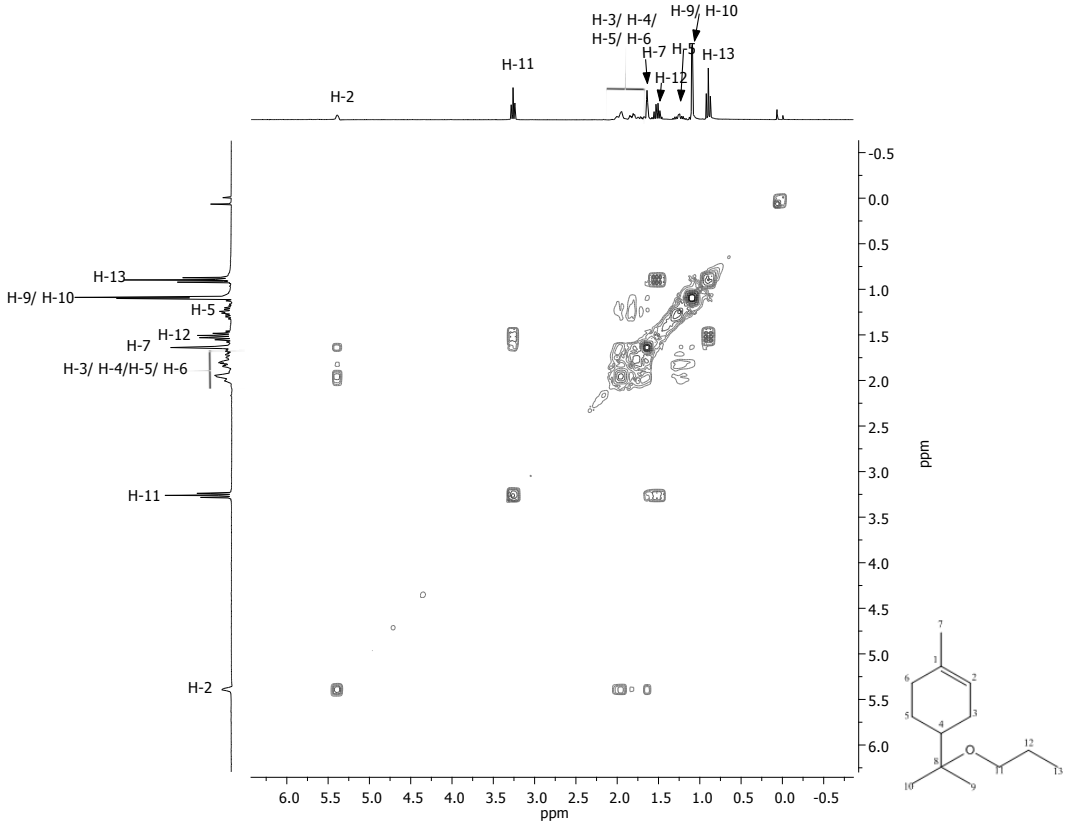


^{13}C NMR (75 MHz, $CDCl_3$): δ 10.8 (C-13); 22.6 (C-10); 22.9 (C-9); 23.4 (C-7); 23.8 (C12); 24.0 (C-5); 26.8 (C-3); 31.1 (C-6), 41.9 (C-4); 62.3 (C-11); 76.2 (C-8); 121.0 (C-2); 134.0 (C-1).

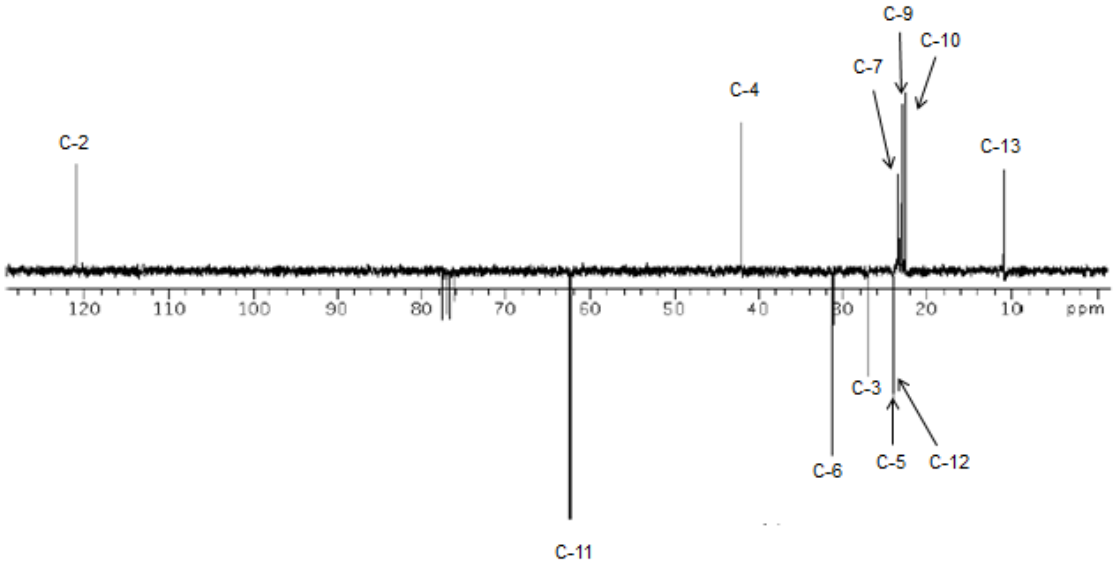
3. NMR ^{1}H, ^{13}C of spectrum – HSQC of 1-methyl-4-(2-propoxypropan-2-yl)cyclohex-1-ene



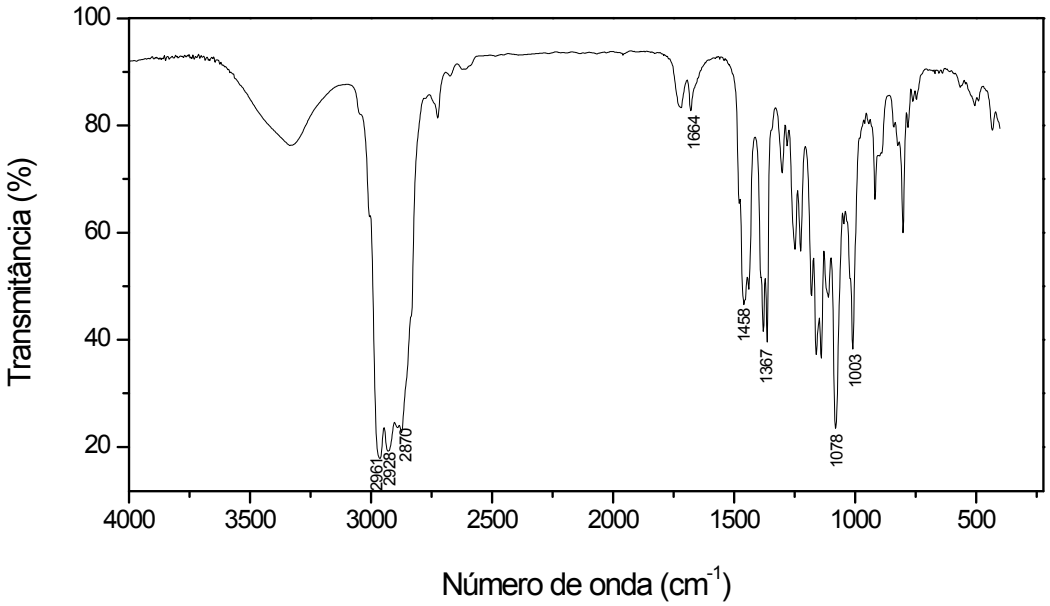
4. **NMR ^{1}H, ^{1}H of spectrum – COSY of 1-methyl-4-(2-propoxypropan-2-yl)cyclohex-1-ene**



5. **DEPT 135 of 1-methyl-4-(2-propoxypropan-2-yl)cyclohex-1-ene**

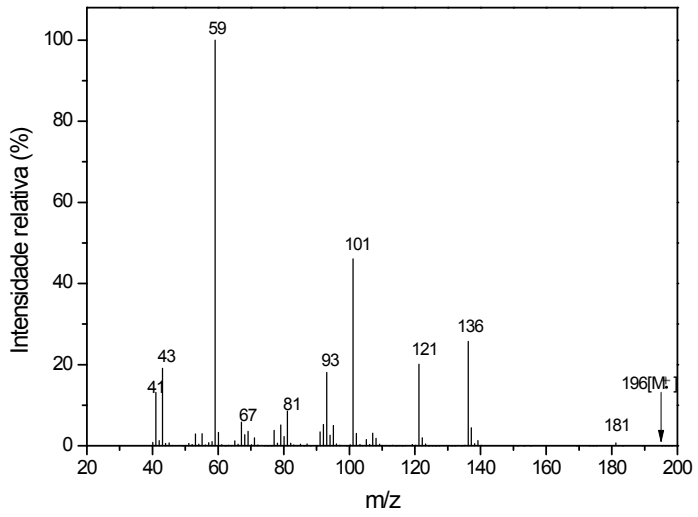


6. **Infrared spectrum of 1-methyl-4-(2-propoxypropan-2-yl)cyclohex-1-ene (KBr)**

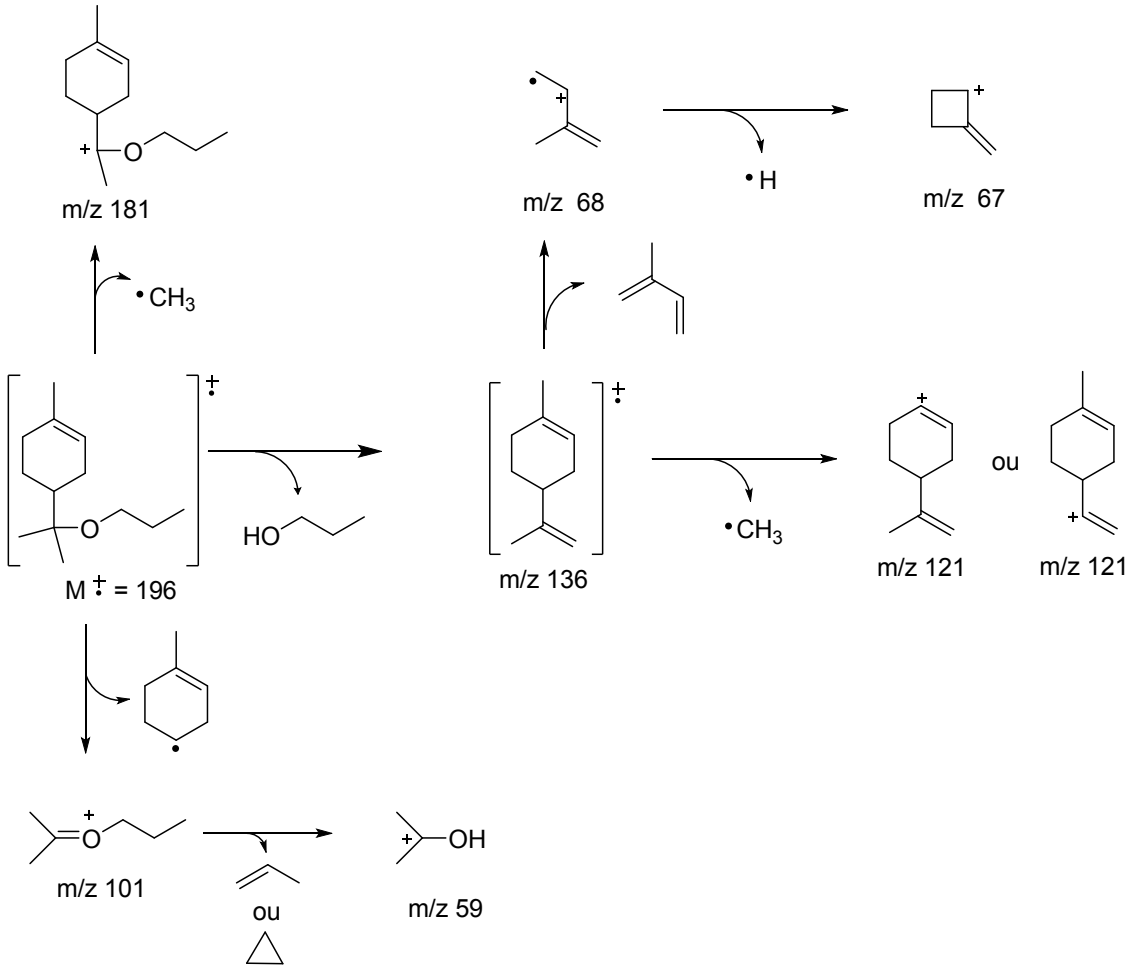


IR (KBr) ν_{max}/cm^{-1}: 2961, 2918, 2870 (ν_s e ν_{as} -CH_2 e -CH_3); 1664 (ν C=C endocyclic); 1458, 1367 (δ –CH_2 and –CH_3); 1078 (ν_{as} C-O); 1003 (ν_s C-O).

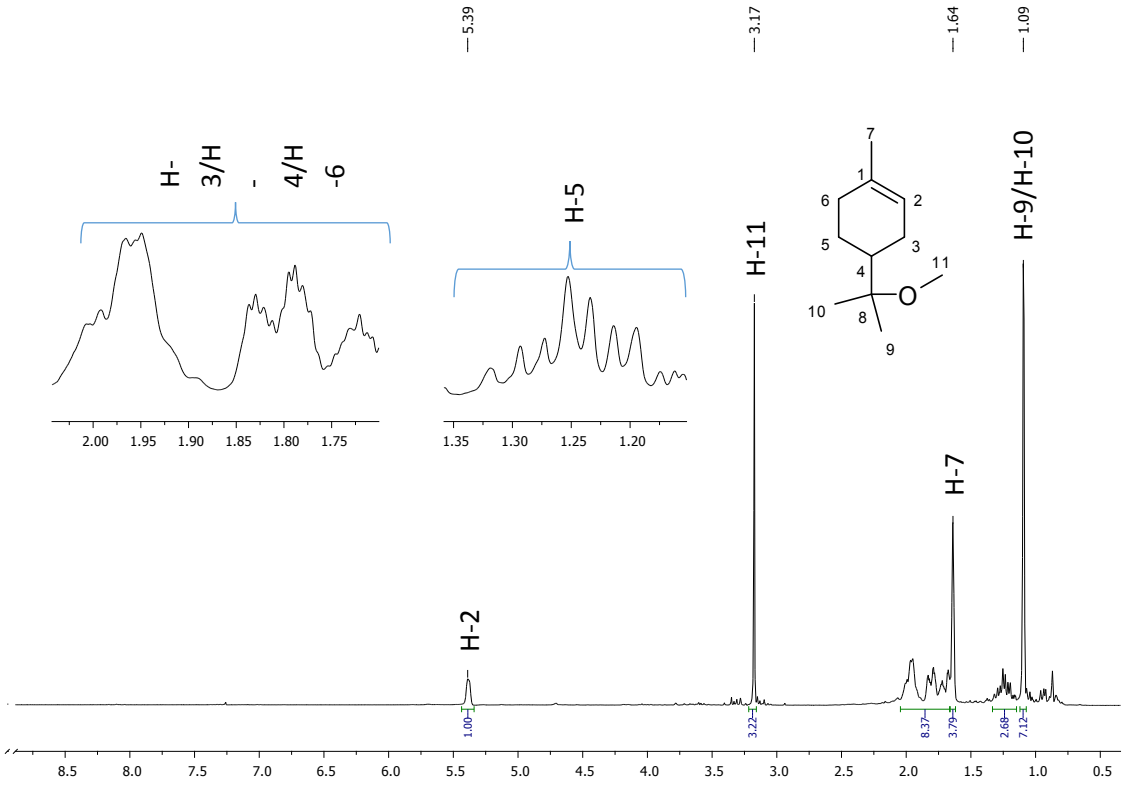
7. **Mass spectrum of 1-methyl-4-(2-propoxypropan-2-yl)cyclohex-1-ene (EI – 70 eV)**



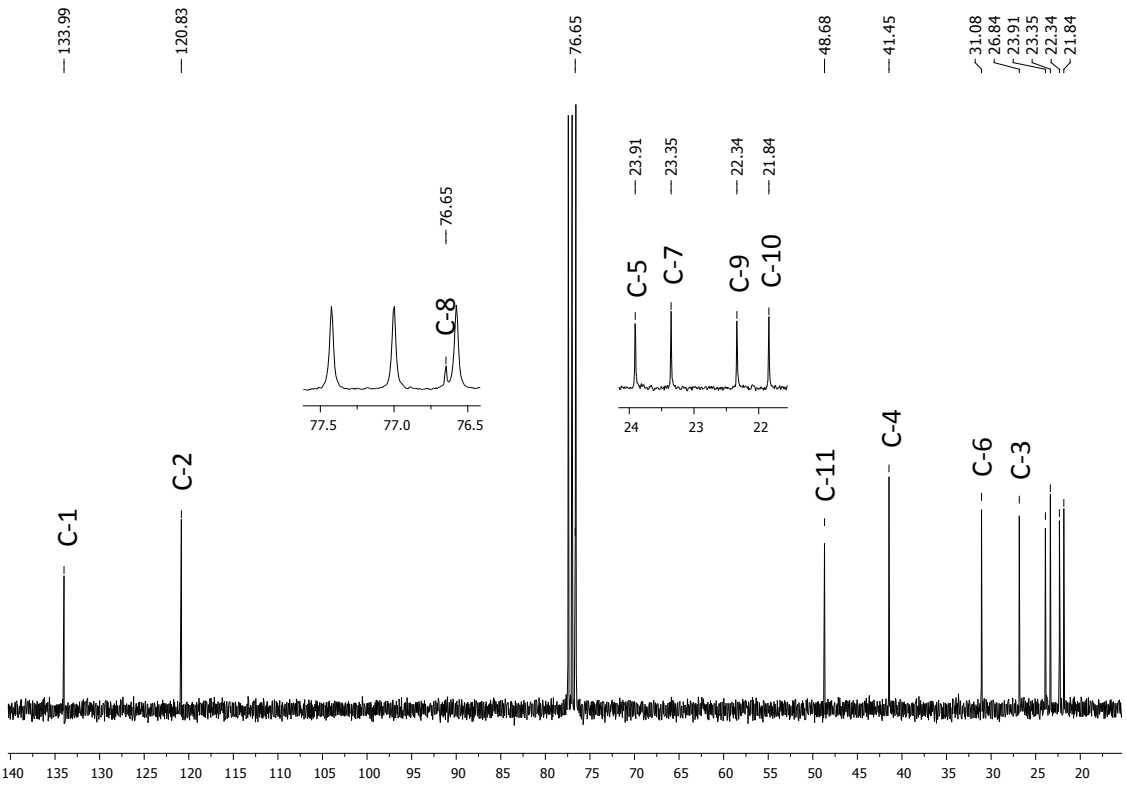
MS (m/z/int.rel.): 196/0 (M^{+}); 181/1; 136/26; 121/20; 101/46; 93/18; 81/9; 67/6; 59/100; 43/19; 41/13.



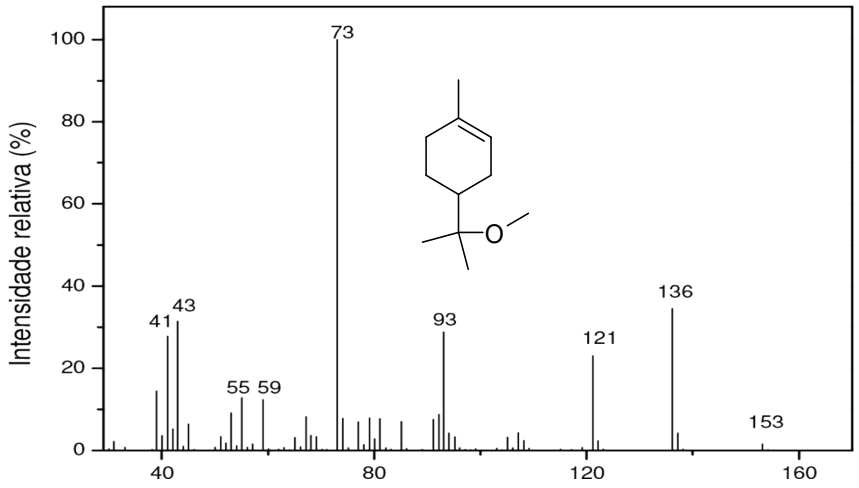
4-(2-methoxypropan-2-il)-1-methylcyclohex-1-ene



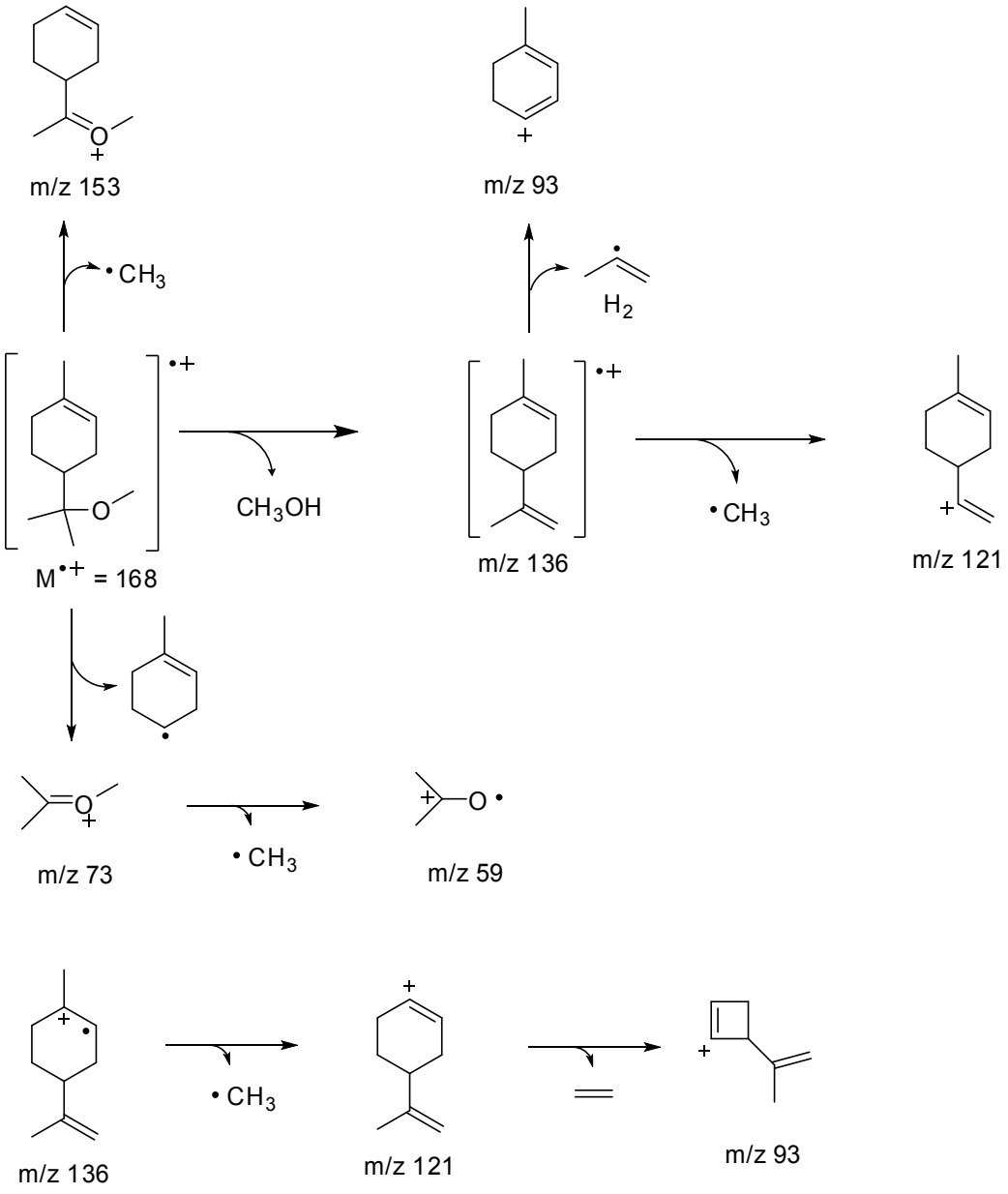
^{1}H NMR spectrum of 4-(2-methoxypropan-2-il)-1-metilciclohex-1-ene



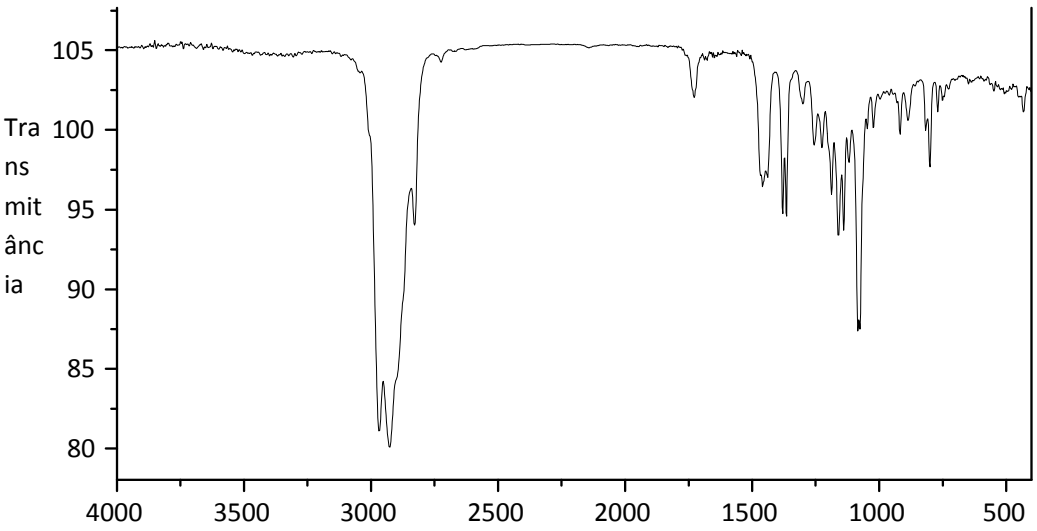
^{13}C NMR of 4-(2-methoxypropan-2-il)-1-methylcycloex-1-ene



Mass spectrum


m/z 153
m/z 93
• CH3
H2
CH3OH
• CH3
M•+ = 168
m/z 136
m/z 121
m/z 73
• CH3
m/z 59
• CH3
m/z 136
m/z 121
m/z 93

.



FT-IR spectrum (ATR) of 4-(2-methoxypropan-2-il)-1-methylcyclohex-1-ene

^{1}H NMR ^{1}H (300 MHz, $CDCl_3$): □ 5.39 (sl, 1H, H-2).); 3.17 (s, 2H, H-11); 2.05 – 1.65 (m, 6H, H-3/H-4/H-6); 1.64 (s, 3H, H-11); 1.35 – 1.10 (m, 1H, H-5); 1.09 (s, 6H, H-9/H-10).

^{13}C NMR (75 MHz, $CDCl_3$) □: 134.0 (C1); 120.8 (C2); 76.6 (C8); 48.7 (C11); 41.5 (C4); 31.1 (C6); 26.8 (C3); 23.9 (C5); 23.4 (C7); 22.3 (C9 or C10); 21.8 (C10 or C9).

IV (ATR) □ (cm^{-1}): 2967; 2926; 2828; 1438; 1378; 1362; 1074.

MS/EI (*m/z*/int.rel.): 153/2; 136/34; 121/23; 93/29; 73/100; 59/12; 55/13; 43/31; 41/28.