**"Supporting Information"**

# Mild and Efficient Removal of Hydroxyethyl Unit from 2-Hydroxyethyl Ether Derivatives Leading to Alcohols

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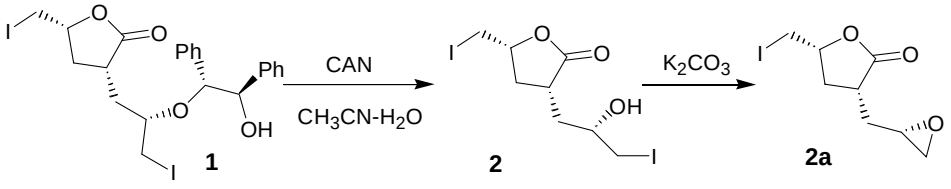
The $^1$H NMR spectra were measured at 300 MHz or 270 MHz with tetramethylsilane as the internal standard at 20–25 °C. IR spectra were recorded by a diffuse reflectance measurement of samples dispersed in KBr powder. E. Merck silica gel 60 for column chromatography and E. Merck pre-coated TLC plates, silica gel $F_{254}$, for preparative thin-layer chromatography were used respectively.

**Typical procedure of cerium ammonium nitrate (CAN) mediated C-C cleavage**

CAN was added to a stirred solution of the substrate (0.1 mmol) in acetonitrile (0.5 mL) and water (0.5 mL) at r.t. under an air. The resulting mixture became immediately red-brown and the color discharged after the required amount of time (ca. 30 min) to give a slightly yellow solution. $K_2CO_3$ was added to a solution and the mixture was stirred for 10 min. After filtrating $K_2CO_3$, the filtrate was concentrated in vacuo. The crude product was purified by $SiO_2$ column chromatography.

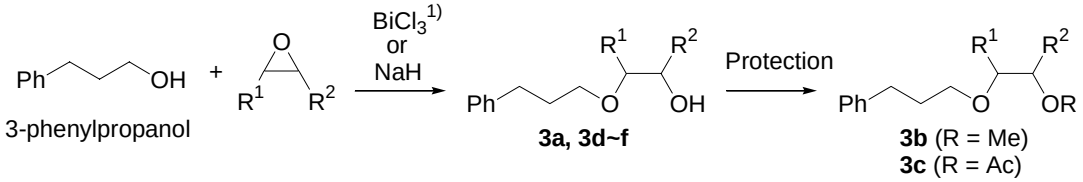
Reaction of **1** with CAN (Table 1).

Compound **1** was reacted with CAN as shown in typical procedure. Compound **2** was rather unstable and tended to give the epoxide **2a**. Then **2** was treated with $K_2CO_3$ and epoxide **2a** was isolated as shown in ref. 6 in the text.[1]



Syntheses of the substrates in Scheme 2 and Table 2.

Compounds **3a**–**3d** were synthesized as shown below. The yield of each compound was not optimized.




***Trans*-1,2-diphenyl-2-(3-phenylpropoxy)ethanol (3a)**

A mixture of *cis*-stilbene oxide (866 mg, 4.41 mmol) and 3-phenylpropanol (1.2 mL, 8.81 mmol) was cooled to 0 °C, and $BiCl_3$ (348 mg, 1.10 mmol) was added under $N_2$. The reaction mixture was stirred at r.t. for 6 hr. $K_2CO_3$ was added to the reaction mixture and stirred for 10 min. After filtrating $K_2CO_3$ , the filtrate was concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 5/1) gave **3a** (325 mg, 22 %) as a colorless crystal.

mp. 74.4–74.5 $^oC$; IR (KBr) 3549, 1092, 750 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 270 MHz): δ 1.94 (2H, quintet, *J* = 6.2 Hz), 2.64–2.72 (2H, m), 3.33–3.45 (2H, m), 3.45 (1H, br s), 4.18 (1H, d, *J* = 9.3 Hz), 4.66 (1H, d, *J* = 9.3 Hz), 6.97–7.30 (15H, m); $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 31.4, 32.5, 68.4, 78.5, 87.5, 125.7, 127.1, 127.5, 127.5, 127.7, 127.8, 127.9, 128.2, 128.2, 137.8, 139.1, 141.6; *Anal.* Calcd for $C_{23}H_{24}O_2$: C, 83.10; H, 7.28; Found: C, 83.14; H, 7.31.

***Trans*-1,2-diphenyl-1-methoxy-2-(3-phenylpropoxy)ethane (3b)**

A mixture of NaH (60% in oil, 7 mg, 0.181 mmol) in THF (1.5 mL) was cooled to 0 °C, and **3a** (50 mg, 0.15 mmol) was added under $N_2$. The reaction mixture was stirred at r.t. for 1 hr and MeI (43 mg, 0.30 mmol) was added dropwise. The reaction mixture was stirred at r.t. for 3 hr and poured into saturated aqueous $NH_4Cl$. The organic layer was separated, and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 5/1) gave **3b** (52 mg, 100%) as a colorless oil.

IR (KBr) 1111, 748 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 300 MHz): δ 1.79 (2H, quintet, *J* = 6.2 Hz), 2.50–2.56 (2H, m), 3.20–3.30 (2H, m), 3.21 (3H, s), 4.25 (1H, d, *J* = 6.3 Hz), 4.32 (1H, d, *J* = 6.3 Hz), 6.99–7.17 (15H, m); $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 31.4, 32.2, 68.4, 57.5, 68.6, 85.9, 87.5, 125.6, 127.4, 127.5, 127.7, 127.7, 127.8, 128.2, 128.4, 138.6, 139.1, 142.1; LRMS (FAB) *m/z* 369 ($MNa^+$); HRMS (FAB) calcd for $C_{24}H_{26}O_2Na$, 369.1830; found, 369.1841.

***Trans*-1,2-diphenyl-1-acetoxy-2-(3-phenylpropoxy)ethane (3c)**

A solution of **3a** (68 mg, 0.20 mmol) in pyridine (0.21 mL) was cooled to 0 °C, and $Ac_2O$ (0.11 mL) was added to the reaction mixture under $N_2$. The reaction mixture was stirred at r.t. for 24 hr and concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 4/1) gave **3c** (61 mg, 79%) as a colorless oil.

IR (KBr) 1744, 743 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 270 MHz): δ 1.78–1.86 (2H, m), 2.03 (3H, s), 2.61–2.57 (2H, m), 3.24–3.42 (2H, m), 4.48 (1H, d, *J* = 7.0 Hz), 5.96 (1H, d, *J* = 7.0 Hz), 7.01–7.27 (15H, m); $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 21.3, 31.4, 32.2, 68.3, 78.4, 84.3, 125.6, 127.4, 127.6, 127.7, 127.8, 128.1, 128.3, 128.5, 137.1, 137.8, 141.9, 169.8; *Anal.* Calcd for $C_{25}H_{26}O_3$: C, 80.18; H, 7.00; Found: C, 80.34; H, 6.99.

**1-Phenyl-2-(3-phenylpropoxy)ethanol (3d)**

A solution of NaH (60% in oil, 350 mg, 8.74 mmol) in THF (40 mL) was cooled to 0 °C, and 3-phenylpropanol (1.2 mL, 8.74 mmol) was added under $N_2$. The reaction mixture was stirred at r.t. for 1 hr and styrene oxide (525 mg, 4.37 mmol) was added dropwise to the resulting mixture. The reaction mixture was stirred for 3 days at 60°C and then poured into saturated aqueous $NH_4Cl$. The organic layer was separated, and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 5/1) gave **3d** (200 mg, 18%) as a colorless oil.

IR (KBr) 3445, 1115, 750 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 270 MHz): δ 1.93 (2H, quintet, *J* = 6.2 Hz), 2.69 (2H, t, *J* = 8.1 Hz), 3.38–3.59 (4H, m), 4.87 (1H, dd, *J* = 10.2, 3.9 Hz), 7.15–7.36 (10H, m); $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 31.1, 32.3, 70.4, 72.6, 76.3, 125.7, 125.9, 127.6, 128.2, 128.3, 140.2, 141.5; LRMS (FAB) *m/z* 279 ($MNa^+$); HRMS (FAB) calcd for $C_{17}H_{20}O_2Na$, 27901361; found, 279.1355.

**2-Phenyl-2-(3-phenylpropoxy)ethanol (3e)**

A mixture of styrene oxide (525 mg, 4.37 mmol) and 3-phenylpropanol (1.2 mL, 8.81 mmol) was cooled to 0 °C, and $BiCl_3$ (348 mg, 1.10 mmol) was added to the mixture. The reaction mixture was stirred at r.t. for 6 hr. $K_2CO_3$ was added to the resulting mixture and stirred for 10 min. After filtrating $K_2CO_3$, the filtrate was concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 5/1) gave **3e** (418 mg, 37 %) as a colorless oil.

IR (KBr) 3361, 1105, 750 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 300 MHz): δ 1.88 (2H, quintet, *J* = 6.2 Hz), 2.61–2.75 (2H, m), 3.31–3.50 (2H, m), 3.60–3.70 (2H, m), 4.37 (1H, dd, *J* = 8.4, 4.3 Hz), 7.13–7.37 (10H, m) ; $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 31.4, 32.4, 67.2, 68.3, 82.9, 125.6, 126.6, 127.8, 128.1, 128.2, 128.3, 138.7, 141.6; LRMS (FAB) *m/z* 257 ($MH^+$); HRMS (FAB) calcd for $C_{17}H_{21}O_2$,

257.1542; found, 257.1544.

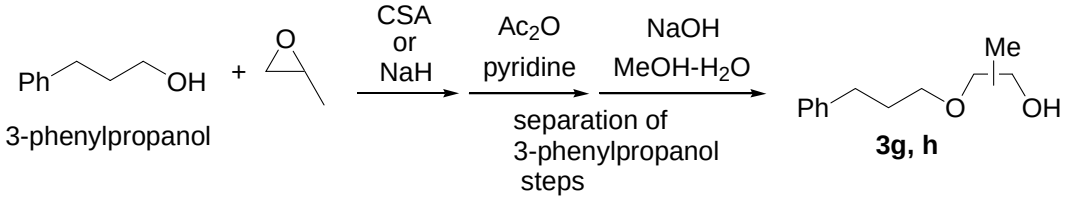
**1,2-Dimethyl-2-(3-phenylpropoxy)ethanol (3f)**

A mixture of 2,3-epoxy-butane (cis/trans mixture, 159 mg, 2.20 mmol) and 3-phenylpropanol (0.6 mL, 4.40 mmol) was cooled to 0 °C, and $BiCl_3$ (174 mg, 0.55 mmol) was added under $N_2$. The reaction mixture was stirred at r.t. for 6 hr. $K_2CO_3$ was added to the reaction mixture and stirred for 10 min. After filtrating $K_2CO_3$ , the filtrate was concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 5/1) gave diastereo-mixture (1/1) of **3f** (229 mg, 50 %) as a colorless oil. Diastereo-mixture (1/1) of **3f** was used for the reaction.

Diastereomers could be separated by $SiO_2$ column chromatography (benzene/AcOEt = 10/1).

IR (KBr) 3416, 1099, 746 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 300 MHz): δ 1.07 (3H, d,, $J$ = 6.5 Hz), 1.13 (3H, d, $J$ = 6.5 Hz), 1.90 (2H, quintet,, $J$ = 6.2 Hz), 2.68 (2H, dd, $J$ = 8.1 Hz), 3.11 (1H, quintet, $J$ = 6.2 Hz), 3.28–3.34 (1H, m), 3.51–3.65 (2H, m), 7.16–7.26 (5H, m) ; $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 15.4, 18.6, 31.6, 32.5, 68.6, 71.2, 80.5, 125.7, 128.2, 128.4, 141.6.

IR (KBr) 3390, 1043, 748 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 300 MHz): δ 1.09 (3H, d,, $J$ = 6.5 Hz), 1.14 (3H, d, $J$ = 6.5 Hz), 1.90 (2H, quintet, $J$ = 6.2 Hz), 3.27–3.50 (3H, m), 3.53–3.85 (1H, m), 7.16–7.29 (5H, m) ; $^{13}C$ NMR ($CDCl_3$,67.8 MHz): δ 13.5, 17.7, 31.4, 32.2, 67.7, 68.9, 78.6, 125.4, 127.9, 128.0, 141.5; ***Anal.*** Calcd for $C_{13}H_{20}O_2$: C, 74.96; H, 9.68; Found: C, 75.02; H, 9.69.




**1-(3-Phenylpropoxy)-2-propanol (3g)**

NaH (60% in oil, 294 mg, 7.34 mmol) in DMF (7.3 mL) was cooled to 0°C, and 3-phenylpropanol (1.0 g, 7.34 mmol) was added under $N_2$. The reaction mixture was stirred at r.t. for 1 hr and 1-epoxypropane (5.1 mL, 73.4 mmol) was added dropwise. The reaction mixture was stirred at r.t. for 24 hr and poured into saturated aqueous $NH_4Cl$. The organic layer was separated, and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated in vacuo. Because resulting product **3g** and 3-phenylpropanol were not separatable, we selected step-wise procedure, acetylation and separation, followed by deprotection of acetylated alcohol.

Crude product in pyridine (8.0 mL) was cooled to 0°C, and $Ac_2O$ (4.0 mL) was added to the reaction mixture under $N_2$. The reaction mixture was stirred at r.t. for 24 hr and concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 30/1) gave Ac

derivative of **3g** (**Ac-3g**). **Ac-3g** in MeOH-$H_2O$ (2.5-0.25 mL) was cooled to 0°C, and NaOH (118 mg, 2.95 mmol) was added to the reaction mixture under $N_2$. The reaction mixture was stirred at r.t. for 24 hr and poured into water. The organic layer was separated, and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 5/1) gave **3g** (453 mg, 32%) as a colorless oil.

IR (KBr) 3422, 1115, 745 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 270 MHz): δ 1.14 (2H, d *J* = 6.5 Hz), 1.86–1.97 (2H, m), 2.15 (1H, brs), 2.69 (1H, t, *J* = 7.6 Hz), 3.21 (1H, dt, *J* = 8.4, 1.0 Hz), 3.38–3.55 (3H, m), 3.90–3.99 (1H, m), 7.17–7.31 (5H, m); $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 18.7, 31.2, 32.4, 66.4, 70.4, 76.3, 125.7, 128.2, 128.3, 141.7;

**Mixture of 3g and 2-(3-Phenylpropoxy)-1-propanol (3h)**

1-Epoxypropane (1 mL, 14.4 mmol) and 3-phenylpropanol (1 mL, 7.33 mmol) was cooled to 0°C, and CSA (3.0 g, 12.9 mmol) was added under $N_2$. The reaction mixture was stirred at r.t. for 6 hr and $K_2CO_3$ was added to the reaction mixture and stirred for 10 min. After filterating $K_2CO_3$, the filtrate was concentrated in vacuo. Because resulting product **3g** and 3-phenylpropanol were not separatable, we selected step-wise procedure, acetylation and separation, followed by deprotection of acetylated alcohol.

Crude product in pyridine (5.0 mL) was cooled to 0°C, and $Ac_2O$ (2.5 mL) was added to the reaction mixture under $N_2$. The reaction mixture was stirred at r.t. for 24 hr and concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 30/1) gave Ac derivative of **3h** (**Ac-3h**). **Ac-3h** in MeOH-$H_2O$ (2.0-0.2 mL) was cooled to 0°C, and NaOH (97 mg, 2.43 mmol) was added to the reaction mixture under $N_2$. The reaction mixture was stirred at r.t. for 24 hr and poured into water. The organic layer was separated, and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 5/1) gave ca. 1/1 (**3g/2-(3-Phenylpropoxy)-1-propanol**) regio-mixture **3h** (367 mg, 26%) as a colorless oil.

$^1H$ NMR ($CDCl_3$, 270 MHz): δ 1.86–1.14 (3H, m), 1.85–1.95 (2H, m), 2.08 (1H, brs), 2.68 (1H, t, *J* = 7.6 Hz), 3.19 (1H, dt, *J* = 8.4, 1.0 Hz), 3.22–3.61 (4H, m), 3.88–3.99 (1H, m), 7.17–7.31 (5H, m).

Compounds **4** by CAN mediated C-C cleavage of **3** (Table 2)

Every reaction was carried out according to the typical procedure.

**(entry 1)**

**4** (24 mg, 100%) was obtained as a colorless oil from **3a** (60 mg, 0.18 mmol), CAN (198 mg, 0.36

mmol), and $CH_3CN$-water (v/v = 1/1, 1.8 mL). ($SiO_2$ column chromatography: *n*-hexane/AcOEt = 3/1)

**(entry 2)**

**4** (32 mg, 100%) was obtained as a colorless oil from **3d** (60 mg, 0.24 mmol), CAN (256 mg, 0.46 mmol), and $CH_3CN$-water (v/v = 1/1, 2.4 mL). ($SiO_2$ column chromatography: *n*-hexane/AcOEt = 3/1)

**(entry 3)**

**4** (26 mg, 92%) was obtained as a colorless oil from **3e** (52 mg, 0.20 mmol), CAN (220 mg, 0.40 mmol), and $CH_3CN$-water (v/v = 1/1, 2.0 mL). ($SiO_2$ column chromatography: *n*-hexane/AcOEt = 3/1)

**(entry 4)**

**4** (32 mg, 80%) was obtained as a colorless oil from **3f** (60 mg, 0.28 mmol), CAN (316 mg, 0.56 mmol), and $CH_3CN$-water (v/v = 1/1, 2.8 mL). ($SiO_2$ column chromatography: *n*-hexane/AcOEt = 3/1)

**(entry 5)**

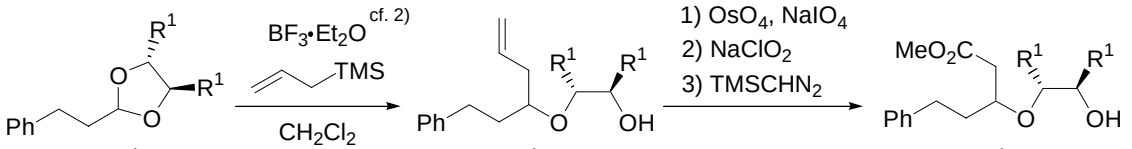
**4** (28 mg, 80%) was obtained as a colorless oil from **3g** (50 mg, 0.26 mmol), CAN (564 mg, 1.03 mmol), and $CH_3CN$-water (v/v = 1/1, 2.6 mL). ($SiO_2$ column chromatography: *n*-hexane/AcOEt = 3/1)

**(entry 6)**

**4** (22.4 mg, 66%) was obtained as a colorless oil from **3h** (49 mg, 0.25 mmol), CAN (825 mg, 1.50 mmol), and $CH_3CN$-water (v/v = 1/1, 2.5 mL). ($SiO_2$ column chromatography: *n*-hexane/AcOEt = 3/1)

Syntheses of substrates **5a**–**5d** in Table 3.

Compounds **5a**–**5d** were synthesized as shown below. The yield of each compound was not optimized.




**12a** ($R^1$ = Ph) **12b** ($R^1$ = Me) **5c** ($R^1$ = Ph) **5d** ($R^1$ = Me) **5a** ($R^1$ = Ph) **5b** ($R^1$ = Me)

**2-(1-Phenethyl-3-butenyloxy)-1,2-diphenylethanol (5c)**

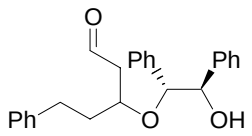
A solution of **12a** (5.1 g, 15.4 mmol) in $CH_3CN$ (31 mL) was cooled to 0 °C, and allyltrimethylsilane (7.4 mL, 46.3 mmol) and $BF_3 \cdot Et_2O$ (9.9 mL, 77.2 mmol) was added to the mixture under $N_2$. The

reaction mixture was stirred at 0 °C for 2 hr and poured into saturated aqueous $NaHCO_3$. The organic layer was separated, and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 20/1) gave **5c** (almost one isomer containing small amount of other stereo-isomer, 460 mg, 8%) as a colorless oil and recovered **12a** (3.8 g, 75%).

IR (KBr) 3560, 1454, 1063 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 300 MHz): δ 1.86–1.94 (2H, m), 2.21–2.26 (2H, m), 2.63–2.71 (2H, m), 3.45 (1H, m), 3.53 (1H, br s), 4.32 (1H, d, *J* = 7.8 Hz), 4.68 (1H, d, *J* = 7.8 Hz), 4.97–5.02 (2H, m), 5.63–5.77 (1H, m), 6.92–7.30 (15H, m); $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 30.8, 34.1, 18.8, 76.1, 78.4, 85.2, 116.9, 125.7, 126.9, 127.6, 127.7, 127.8, 128.1, 128.2, 134.3, 138.1, 141.7; *Anal.* Calcd for $C_{26}H_{28}O_2$: C, 83.83; H, 7.58; Found: C, 83.55; H, 7.75.

**2-(5-phenyl-3-pentanaloxy)-1,2-diphenylethanol (13a)**

A solution of **5c** (813 mg, 2.18 mmol) in dioxane-water (v/v = 3/1, 9 mL) was cooled to 0 °C, and cat. $OsO_4$, $NaIO_4$ (1.9 g, 8.73 mmol) and 2,6-lutidine (0.5 mL, 4.36 mmol) were added to the reaction mixture under an air. The reaction mixture was stirred at r.t. for 3 hr. After filtration, the residue was concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 10/1) gave **13a** (734 mg, 53%) as a colorless oil.



**13a**

IR (KBr) 3443, 1728, 742 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 300 MHz): δ 1.92–2.14 (2H, m), 2.48–2.75 (4H, m), 3.63 (1H, br s), 3.92–4.00 (1H, m), 4.40 (1H, d, *J* = 8.1 Hz), 4.76 (1H, d, *J* = 8.1 Hz), 6.95–7.37 (15H, m), 9.53 (1H, s); $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 30.7, 34.6, 48.1, 71.8, 78.0, 85.3, 125.9, 127.0, 127.5, 127.7, 127.9, 128.0, 128.1, 128.1, 128.4, 137.5, 139.0, 141.0, 201.0; LRMS (FAB) *m/z* 397 ($MNa^+$); HRMS (FAB) calcd for $C_{25}H_{26}O_3Na$, 397.1780; found, 397.1802.

**2-(4-phenyl-1-methoxycarbonyl-2-butanoxy)-1,2-diphenylethanol (5a)**

$NaH_2PO_4$ (558 mg, 4.65 mmol), 2-metyl-2-butene (0.82 mL, 7.75 mmol) and $NaClO_2$ (263 mg, 2.33 mmol) were added to a stirred solution (r.t.) of **13a** (580 mg, 1.55 mmol) in tBuOH-water (v/v = 5/1, 16 mL) under an air. The reaction mixture was stirred at r.t. for 6 hr, and poured into 10% aqueous HCl. The organic layer was separated, and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated in vacuo to give the residue. $TMSCHN_2$ (2M in hexane, 1 mL, 2.02 mmol) was added to a stirred solution of the residue in benzene-MeOH (v/v = 4/1, 15 mL) at 0 °C. The reaction mixture was stirred at r.t. for 1hr, and concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 5/1) gave **5a** (564 mg, 90% in 2 steps) as a colorless oil.

IR (KBr) 3562, 1732, 912 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 270 MHz): δ 1.80–1.91 (2H, m), 2.28–2.66 (4H,

m), 3.41 (3H, s), 3.78 (1H, quintet, $J$ = 5.9 Hz), 4.26 (1H, d, $J$ = 8.7 Hz), 4.58 (1H, d, $J$ = 8.7 Hz), 6.86–7.24 (15H, m); $^{13}$C NMR ($CDCl_3$, 67.8 MHz): δ 30.7, 34.6, 39.8, 51.3, 73.6, 78.2, 85.1, 125.7, 126.6, 127.3, 127.5, 127.6, 127.7, 127.7, 128.0, 128.2, 137.6, 139.0, 141.2, 171.0; LRMS (FAB) *m/z* 405 ($MH^+$); HRMS (FAB) calcd for $C_{26}H_{29}O_4$, 405.2066; found, 405.2070.

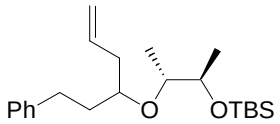
**3-(1-Phenethyl-3-butenyloxy)-2-butanol (5d)**

A solution of **12b** (1.19 g, 5.77 mmol) in $CH_3CN$ (12 mL) was cooled to 0 °C, and allyltrimethylsilane (2.8 mL, 17.31 mmol) and $BF_3 \cdot Et_2O$ (3.7 mL, 28.85 mmol) was added to the mixture under $N_2$. The reaction mixture was stirred at 0 °C for 3 hr and poured into saturated aqueous $NaHCO_3$. The organic layer was separated, and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 10/1) gave **5d** (753 mg, 53%) as a colorless oil.

ca.1/1 diastereo-mixture; IR (KBr) 3450, 1454, 1063 $cm^{-1}$; $^1$H NMR ($CDCl_3$, 300 MHz): δ 1.07–1.16 (6H, m), 1.72–1.86 (2H, m), 2.29–2.34 (2H, m), 2.36–2.71 (3H, m), 3.24–3.56 (3H, m), 5.05–5.15 (2H, m), 5.75–5.90 (1H, m), 7.17–7.30 (5H, m); $^{13}$C NMR ($CDCl_3$, 67.8 MHz): δ 16.5, 16.6, 18.6, 18.6, 31.5, 31.8, 35.6, 36.3, 38.4, 39.2, 71.1, 71.3, 76.1, 76.5, 78.4, 78.5, 116.9, 117.8, 125.6, 125.7, 128.1, 128.2, 128.2, 128.2, 134.3, 134.7, 141.7, 142.0; ***Anal.*** Calcd for $C_{16}H_{24}O_2$: C, 77.38; H, 9.74; Found: C, 77.62; H, 9.67.

Compound **5c** was synthesized from **5d** via silyl ether **14b**.

**3-(1-Phenethyl-3-butenyloxy)-2-*tert*butyldimethysilyloxybutane (14b)**



**14b**

*t*Butyldimethylsilyl chloride (1.19 g, 7.89 mmol) and imidazol (894 mg, 13.13 mmol) were added to a stirred solution of **5d** (652 mg, 2.63 mmol) in $CH_2Cl_2$ (2.6 mL) at 0 °C under $N_2$. The reaction mixture was stirred at r.t. for 24 hr and poured into saturated aqueous $NaHCO_3$. The organic layer was separated, and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 50/1) gave **14b** (902 mg, 95%) as a colorless oil.

IR (KBr) 1103, 912 $cm^{-1}$; $^1$H NMR ($CDCl_3$, 300 MHz): δ -0.01–0.00 (6H, m), 0.84–0.85 (9H, m), 1.02–1.06 (6H, m), 1.72–1.76 (2H, m), 2.24–2.26 (2H, m), 2.51–2.70 (2H, m), 3.36–3.38 (2H, m), 3.77–3.79 (1H, m), 5.01–5.04 (2H, m), 5.72–5.83 (1H, m), 7.13–7.26 (5H, m); $^{13}$C NMR ($CDCl_3$, 270 MHz): δ -4.7, -4.6, -4.5, 13.7, 14.0, 17.1, 17.4, 25.9, 31.9, 36.0, 36.3, 39.0, 39.3, 69.1, 69.9, 76.1, 76.5, 76.8, 76.8, 116.8, 116.9, 125.6, 125.6, 128.2, 128.2, 134.8, 135.0, 142.1, 142.4; LRMS (FAB) *m/z* 363 ($MH^+$); HRMS (FAB) calcd for $C_{22}H_{39}O_2Si$, 363.2720; found, 363.2715.

**3-(4-phenyl-1-methoxycarbonyl-2-butanoxy)-2-butanol (5b)**

A solution of **14b** (842 mg, 2.32 mmol) in dioxane-water (v/v = 3/1, 6.1 mL) was cooled to 0 °C, and $OsO_4$ (60 mg, 0.05 mmol), $NaIO_4$ (1.99 g, 9.29 mmol) and 2,6-lutidine (0.54 mL, 4.64 mmol) were added to the reaction mixture under an air. The reaction mixture was stirred at r.t. for 3 hr. After filtrating, the filtrate was concentrated in vacuo to give the residue.

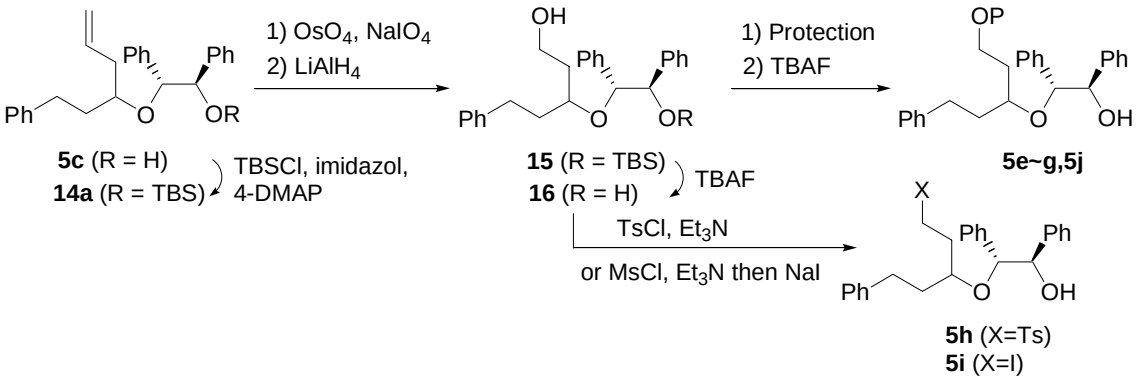
$NaH_2PO_4$ (843 mg, 7.03 mmol), 2-metyl-2-butene (1.2 mL, 11.71 mmol) and $NaClO_2$ (318 mg, 3.52 mmol) were added to the solution of above residue in tBuOH-water (v/v = 5/1, 23 mL) under an air. The reaction mixture was stirred at r.t. for 12 hr, and poured into saturated 10% aqueous HCl. The organic layer was separated, and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated in vacuo.

The obtained residue was dissolved in benzene-MeOH (v/v = 4/1, 14 mL), and the solution was cooled to 0 °C. $TMSCHN_2$ (2M in hexane, 1.52 mL, 1.52 mmol) was added to the reaction mixture. The reaction mixture was stirred at r.t. for 1hr, and concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 4/1) gave **5b** (501 mg, 77% in 3 steps) as a colorless oil.

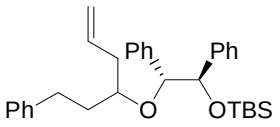
IR (KBr) 3459, 1730, 912 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 300 MHz) δ: 1.05–1.11 (6H, m), 1.75–1.97 (2H, m), 2.49–2.74 (4H, m), 3.24 (1H, dt, *J* = 21.0, 6.8 Hz), 3.53 (1H, dt, *J* = 20.3, 6.8 Hz), 3.67 (3H, s), 3.90 (1H, dd, *J* = 12.1, 6.5 Hz), 7.17–7.27 (5H, m); $^{13}C$ NMR ($CDCl_3$, 270 MHz): δ: 15.9, 17.39, 18.6, 31.3, 31.5, 35.8, 36.1, 38.8, 40.3, 51.6, 51.9, 70.9, 71.5, 73.3, 74.3, 78.3, 79.7, 125.8, 125.9, 128.1, 128.3, 141.2, 141.4, 171.7, 172.5; *Anal.* Calcd for $C_{16}H_{24}O_4$: C, 68.54; H, 8.63; Found: C, 68.47; H, 8.46.

Syntheses of substrates **5e**–**5j** in Table 3.

Compounds **5e**–**5j** were synthesized as shown below. The yield of each compound was not optimized. The compounds having alkaline labile functions, **5h** and **5i**, were synthesized via compound **16**, because TBAF treatment of the tosylate or the iodide from compound **15** gave poor results.



**1-*tert*-butyldimethysilyloxy-2-(1-phenethyl-3-butenyloxy)-1,2-diphenylethane (14a)**

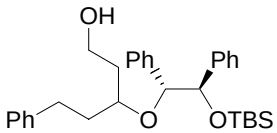


**14a**

By the same procedure as **14b, 14a** (511 mg, 88%) was obtained as a colorless oil by purification of $SiO_2$ column chromatography (*n*-hexane/AcOEt = 20/1) from **5c** (460 mg, 1.2 mmol) in $CH_2Cl_2$ (2.6 mL) and *tert*-butyl-dimethylsilyl chloride (559 mg, 3.7 mmol) and imidazol (421 mg, 6.2 mmol).

IR (KBr) 1454, 1067 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 300 MHz): δ -0.14 (3H, s), 0.01 (3H, s), 0.86 (9H, s), 1.79–1.82 (2H, m), 2.20–2.24 (2H, m), 2.60–2.66 (2H, m), 3.30–3.40 (1H, m), 4.43 (1H, d, *J* = 6.3 Hz), 4.75 (1H, d, *J* = 6.3 Hz), 4.95–5.00 (2H, m), 5.60–5.91 (1H, m), 6.95–7.30 (15H, m); $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ -4.8, -2.8, 18.4, 25.7 (3C), 30.6, 34.0, 39.0, 75.1, 78.8, 84.0, 116.4, 125.4, 126.8, 127.1, 127.1, 127.2, 127.3, 127.3, 128.1, 128.2, 128.2, 128.2, 135.2, 139.2, 141.4, 142.6; LRMS (FAB) *m/z* 509 ($MNa^+$); HRMS (FAB) calcd for $C_{32}H_{42}O_2SiNa$, 509.2852; found, 509.2860.

**1-*tert*-Butyldimethysilyloxy-2-(5-phenyl-1-hydroxy-3-pentanoxy)-1,2-diphenylethane (15)**



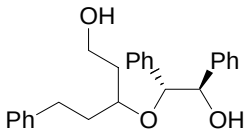
**15**

A solution of **14a** (1.8 g, 3.7 mmol) in dioxane-water (v/v = 3/1, 7.5 mL) was cooled to 0 °C, and $OsO_4$ (95 mg, 0.37 mmol), $NaIO_4$ (3.2 g, 15.0 mmol) and 2,6-lutidine (0.87 mL, 7.4 mmol) were added to the reaction mixture under an air. The reaction mixture was stirred at r.t. for 3 hr. After filtration, the filtrate was concentrated in vacuo.

$LiAlH_4$ (147 mg, 3.90 mmol) in THF (22 mL) was cooled to 0 °C, and a solution of the obtained residue in THF (10 mL) was added to the reaction mixture under $N_2$. The mixture was stirred at 0 °C for 1 hr and poured into water (0.15 mL), 15% aqueous NaOH (0.15 mL), and water (0.45 mL). After filtration by celite, the filtrate was concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 4/1) gave **15** (1.30 g, 61%) as a colorless oil.

IR (KBr) 3481, 1028, 742 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 300 MHz): δ -0.10 (3H, s), 0.09 (3H, s), 1.10 (9H, s), 1.89–1.98 (4H, m), 2.59 (2H, t, *J* = 8.1 Hz), 3.79–3.82 (1H, m), 3.99–4.05 (2H, m), 4.76 (1H, d, *J* = 4.5 Hz), 5.00 (1H, d, *J* = 4.5 Hz), 7.26–7.52 (15H, m); $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ -5.2, 5.0, 14.3, 18.2, 21.1, 25.8 (3C), 29.9, 33.4, 35.7, 60.3, 61.4, 75.4, 78.5, 83.0, 125.6, 127.0, 127.1, 127.4, 127.5, 127.6, 127.7, 127.7, 128.0, 128.1, 128.2, 138.4, 141.6, 142.0; LRMS (FAB) *m/z* 513 ($M+Na^+$); HRMS (FAB) calcd for $C_{31}H_{42}O_3Si$, 513.2801; found, 513.2804.

**2-(5-Phenyl-1-hydroxy-3-pentanoxy)-1,2-diphenylethanol (16)**



**16**

A solution of **15** (167 mg, 0.34 mmol) in THF (0.34 mL) was cooled to 0 °C, and tertbutylammonium fluoride (TBAF) (1M in toluene, 051 mL, 0.51 mmol) was added to the reaction mixture under $N_2$. The mixture was stirred at r.t. for 24 hr and poured into saturated aqueous $NH_4Cl$.

The organic layer was separated, and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 1/1) gave **16** (86 mg, 100%) as a colorless oil.

IR (KBr) 3442, 1198, 773 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 300 MHz): δ 1.68–1.96 (4H, m), 2.53–2.67 (2H, m), 3.02 (2H, br s), 3.48–3.51 (1H, m), 3.61 (2H, t, *J* = 6.0 Hz), 4.33 (1H, d, *J* = 7.8 Hz), 4.72 (1H, d, *J* = 7.8 Hz), 6.98–7.30 (15H, m); $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 30.7, 34.3, 36.4, 59.9, 74.1, 84.3, 125.7, 126.8, 127.6, 127.8, 128.0, 128.0, 128.0, 128.2, 137.6, 139.1, 141.5; *Anal.* Calcd for $C_{25}H_{28}O_3$: C, 79.75; H, 7.50; Found: C, 75.92; H, 7.22; LRMS (FAB) *m/z* 377 ($MH^+$); HRMS (FAB) calcd for $C_{25}H_{29}O_3$, 377.2116; found, 377.2111.

**2-[1-(2-Methoxyethyl)-3-phenylpropoxy]-1,2-diphenylethanol (5e)**

A solution of NaH (60% in oil, 14 mg, 0.35 mmol) in THF (0.24 mL) was cooled to 0 °C, and **15** (116 mg, 0.24 mmol) was added to the reaction mixture under $N_2$. The mixture was stirred at 0 $^oC$ for 30 min and MeI (0.02 mL, 0.35 mmol) was added dropwise. The resulting mixture was stirred at r.t. for 24 hr and poured into water. The organic layer was separated, and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated in vacuo.

TBAF (1M in toluene, 0.35 mL, 0.35 mmol) was added to the solution of the obtained residue in THF (0.24 mL) at 0 °C under $N_2$. The reaction mixture was stirred at r.t. for 24 hr and poured into saturated aqueous $NH_4Cl$. The organic layer was separated, and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 4/1) gave **5e** (91 mg, 97%) as a colorless oil.

IR (KBr) 3557, 912 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 270 MHz): δ1.72–1.95 (4H, m), 2.59–2.70 (2H, m), 3.16 (3H, s), 3.26–3.31 (1H, m), 3.43–3.49 (2H, m), 4.31 (1H, d, *J* = 8.1 Hz), 4.70 (1H, d, *J* = 8.1 Hz), 6.96–7.31 (15H, m).; $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 30.8, 34.4, 34.6, 58.4, 69.2, 73.4, 78.2, 84.6, 125.7, 127.0, 127.6, 127.8, 128.0, 128.2, 128.3, 137.9, 139.2, 141.8; *Anal.* Calcd for $C_{26}H_{30}O_3$: C, 79.97; H, 7.74; Found: C, 79.82; H, 7.80.

**2-[1-(2-Benzyloxyethyl)-3-phenylpropoxy]-1,2-diphenylethanol (5f)**

A solution of NaH (60% in oil, 22 mg, 0.56 mmol) in THF-DMF (v/v = 1/1, 0.4 mL) was cooled to 0 °C, and **15** (182 mg, 0.37 mmol) was added to the reaction mixture under $N_2$. The reaction mixture was stirred at 0 $^oC$ for 30 min and BnBr (0.07 mL, 0.56 mmol) was added dropwise. The reaction mixture was stirred at r.t. for 3 hr and poured into saturated aqueous $NH_4Cl$. The organic layer was separated, and the aqueous layer was extracted with AcOEt. The combined organic layer was washed

with brine, dried over $Na_2SO_4$, and concentrated in vacuo.

TBAF (1M in toluene, 0.58 mL, 0.58 mmol) was added to the solution of the obtained residue in THF (0.6 mL) at $0^{O}$C under $N_2$. The reaction mixture was stirred at r.t. for 24 h and poured into saturated aqueous $NH_4Cl$. The organic layer was separated, and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 4/1) gave **5f** (125 mg, 72%) as a colorless oil.

IR (KBr) 35605, 912 $cm^{-1}$; $^1$H NMR ($CDCl_3$, 300 MHz): δ 2.04–1.87 (4H, m), 2.84–2.63 (2H, m), 3.66–3.43 (3H, m), 4.38–4.37(3H, m), 4.79 (1H, d, *J* = 8.2 Hz), 7.44–7.03 (20H, m); $^{13}$C NMR ($CDCl_3$, 67.8 MHz): δ 31.2, 34.8, 35.0, 67.3, 73.1, 73.7, 78.5, 85.0, 126.1, 127.3, 127.6, 127.8, 127.8, 128.0, 128.1, 128.1, 128.2, 128.3, 128.4, 128.5, 128.6, 138.3, 138.5, 140.0, 142.1; ***Anal.*** Calcd for $C_{32}H_{34}O_3$: C, 82.37; H, 7.34; Found: C, 82.15; H, 7.42.

**2-[1-(2-Acetoxyethyl)-3-phenylpropoxy]-1,2-diphenylethanol (5g)**

A solution of **15** (163 mg, 0.33 mmol) in pyridine (0.33 mL) was cooled to 0 °C, and $Ac_2O$ (0.17 mL) was added to the solution under $N_2$. The mixture was stirred at r.t. for 24 hr and concentrated in vacuo.

TBAF (1M in toluene, 0.5 mL, 0.50 mmol) was added to the solution of the obtained residue in THF (0.4 mL) at 0 °C under $N_2$. The reaction mixture was stirred at r.t. for 24 hr and poured into saturated aqueous $NH_4Cl$. The organic layer was separated, and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 8/1) gave **5g** (132 mg, 96%) as a colorless oil.

IR (KBr) 3560, 1730, 912 $cm^{-1}$; $^1$H NMR ($CDCl_3$, 300 MHz) δ: 1.89 (3H, s), 1.96–2.05 (4H, m), 2.66–2.78 (2H, m), 3.49–3.56 (1H, m), 4.11–4.17 (2H, m), 4.36 (1H, d, *J* = 8.3 Hz), 4.79 (1H, d, *J* = 8.3 Hz), 6.95–7.38 (15H, m); $^{13}$C NMR ($CDCl_3$, 67.8 MHz): δ 20.7, 30.7, 32.9, 34.3, 60.9, 72.0, 77.9, 84.5, 125.8, 126.9, 127.4, 127.6, 127.8, 127.9, 128.1, 128.2, 137.5, 139.0, 141.4, 170.6; ***Anal.*** Calcd for $C_{27}H_{30}O_4$: C, 77.48; H, 7.22; Found: C, 77.34; H, 7.21.

**2-[1-(2-Tosyloxyethyl)-3-phenylpropoxy]-1,2-diphenylethanol (5h)**

A solution of **16** (210 mg, 0.83 mmol) in $CH_2Cl_2$ (0.83 mL) was cooled to 0 °C, and $Et_3N$ (0.12 mL, 1.22 mmol) and TsCl (117 mg, 0.91 mmol) were added to the solution. The reaction mixture was stirred at r.t. for 24 hr and poured into water. The organic layer was separated, and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 4/1) gave **5h** (304 mg, 69%) as a colorless oil.

IR (KBr) 3650, 1248 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 270 MHz): δ 1.78–1.98 (4H, m), 2.43 (3H, s), 2.53–2.62 (2H, m), 3.21 (1H, br s), 3.40–3.44 (1H, m), 4.09–4.12 (2H, m), 4.26 (1H, d, *J* = 8.2 Hz), 4.66 (1H, d, *J* = 8.2 Hz), 6.87–7.33 (17H, m), 7.73 (2H, d, *J* = 8.4 Hz).; $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 21.7, 30.6, 33.5, 34.3, 72.3, 76.5, 78.2, 84.7, 125.9, 127.0, 127.5, 127.7, 127.9, 128.0, 128.1, 128.1, 128.1, 128.4, 129.6, 129.7, 132.9, 137.3, 138.9, 141.2, 144.6; LRMS (FAB) *m/z* 553 ($MNa^+$); HRMS (FAB) calcd for $C_{32}H_{34}O_5SNa$, 553.2025; found, 553.1990.

**2-[1-(2-Iodoethyl)-3-phenylpropoxy]-1,2-diphenylethanol (5i)**

A solution of **16** (128 mg, 0.51 mmol) in $CH_2Cl_2$ (0.5 mL) was cooled to 0 °C, and $Et_3N$ (0.09 mL, 0.62 mmol) and MsCl (0.04 mL, 0.62 mmol) were added to the solution. The reaction mixture was stirred at 0 °C for 3 hr and poured into water. The organic layer was separated, and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated in vacuo.

NaI (227 mg, 1.53 mmol) was added to the solution of the obtained residue in acetone (0.5 mL) under $N_2$. The reaction mixture was refluxed for 3 hr. After filtration by celite, the filtrate was concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 8/1) gave **5i** (44 mg, 18%) as a colorless oil.

IR (KBr) 3560, 912, 743 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 270 MHz) δ: 1.95–2.10 (4H, m), 2.59–2.67 (2H, m), 2.97–3.03 (1H, m), 3.19–3.28 (1H, m), 3.38–3.42 (1H, m), 4.28 (1H, d, *J* = 8.2 Hz), 4.73 (1H, d, *J* = 8.2 Hz), 6.94–7.37 (17H, m); $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 2.1, 30.7, 34.0, 38.8, 75.8, 76.4, 84.6, 125.8, 126.9, 127.4, 127.6, 127.9, 128.0, 128.1, 128.3, 137.4, 138.9, 141.2; LRMS (FAB) *m/z* 487 ($MH^+$); HRMS (FAB) calcd for $C_{25}H_{28}O_2I$, 487.1145; found, 487.1124.

**2-{1-[2-(4-Methoxybenzyloxy)ethyl]-3-phenylpropoxy}-1,2-diphenylethanol (5j)**

A solution of NaH (60% in oil, 19 mg, 0.47 mmol) in THF-DMF (v/v = 1/1, 0.3 mL) was cooled to 0 °C, and **15** (153 mg, 0.31 mmol) was added to the solution. The reaction mixture was stirred at 0 $^oC$ for 30 min and PMBCl (0.06 mL, 0.47 mmol) was added dropwise. The reaction mixture was stirred at r.t. for 3 hr and poured into saturated aqueous $NH_4Cl$. The organic layer was separated, and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated in vacuo.

TBAF (1M in toluene, 0.55 mL, 0.55 mmol) was added to the solution of the obtained residue in THF (0.4 mL) at 0 ° C under $N_2$. The reaction mixture was stirred at r.t. for 24 hr and poured into saturated aqueous $NH_4Cl$. The organic layer was separated, and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated in vacuo. Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 4/1) gave **5j** (92 mg, 60%) as a colorless oil.

IR (KBr) 3553, 912, 743 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 300 MHz): δ 1.97–1.90 (5H, m) 2.75–2.68 (2H, m), 3.60–3.44 (3H, m), 3.86 (3H, s), 4.30 (2H, s), 4.37 (1H, d, *J* = 8.1 Hz), 4.78 (2H, d, *J* = 8.3 Hz), 6.90–7.38 (14H, m); $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 30.7, 34.4, 34.6, 55.1, 66.6, 72.4, 73.3, 78.1, 84.6, 113.4, 113.5, 125.6, 126.9, 127.6, 127.7, 127.8, 127.9, 128.1, 128.2, 129.0, 130.2, 137.9, 139.1, 141.7, 158.8; *Anal.* Calcd for $C_{33}H_{36}O_4$: C, 79.81; H, 7.31; Found: C, 79.48; H, 7.27.

Compounds **6** by CAN mediated C-C cleavage of **5** (Table 3)

Every reaction was carried out according to the typical procedure.

**4-Phenyl-1-methoxycarbonyl-2-butanol (6a)**

**6a** (30 mg, 97%) was obtained as a colorless oil from **5a** (60 mg, 0.14 mmol), CAN (162 mg, 0.30 mmol), and $CH_3CN$-water (v/v = 1/1, 1.4 mL). ($SiO_2$ column chromatography: *n*-hexane/AcOEt = 4/1)

**6a** (43 mg, 90%) was obtained as a colorless oil from **5b** (64 mg, 0.23 mmol), CAN (251 mg, 0.46 mmol), and $CH_3CN$-water (v/v = 1/1, 2.2 mL). ($SiO_2$ column chromatography: *n*-hexane/AcOEt = 4/1)

IR (KBr) 3526, 1728, 912 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 270 MHz): δ 1.65–1.80 (2H, m), 2.33–2.48 (2H, m), 2.57–2.76 (2H, m), 2.94 (1H, br s), 3.63 (3H, s), 3.95 (1H, quintet, *J* = 4.3 Hz), 7.11–7.24 (5H, m); $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 31.8, 38.1, 41.1, 51.8, 67.2, 125.8, 128.3, 128.3, 141.5, 173.2; *Anal.* Calcd for $C_{12}H_{16}O_3$: C, 69.21; H, 7.74; Found: C, 69.01; H, 7.73.

**1-Phenyl-5-hexen-3-ol (6b)**

**6b** (28 mg, 100%) was obtained as a colorless oil from **5c** (60 mg, 0.16 mmol), CAN (176 mg, 0.32 mmol), and $CH_3CN$-water (v/v = 1/1, 1.6 mL). ($SiO_2$ column chromatography: *n*-hexane/AcOEt = 10/1).

**6b** (35 mg, 98%) was obtained as a colorless oil from **5d** (50 mg, 0.20 mmol), CAN (220 mg, 0.40 mmol), and $CH_3CN$-water (v/v = 1/1, 2.0 mL). ($SiO_2$ column chromatography: *n*-hexane/AcOEt = 4/1)

IR (KBr) 3361, 912, 743 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 270 MHz): δ 1.67 (1H, br s), 1.67–1.76 (2H, m), 2.10–2.23 (2H, m), 2.56–2.73 (2H, m), 3.60 (1H, quintet, *J* = 4.3 Hz), 5.04–5.10 (2H, m), 5.67–5.79 (1H, m), 7.11–7.24 (5H, m); $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 32.1, 38.5, 42.1, 69.9, 118.3, 125.7, 128.3, 128.3, 134.5, 141.9.0; *Anal.* Calcd for $C_{12}H_{16}O$: C, 81.77; H, 9.15; Found: C, 81.81; H, 9.19.

**1-Methoxy-5-phenyl-3-pentanol (6c)**

**6c** (32 mg, 86%) was obtained as a colorless oil from **5e** (75 mg, 0.19 mmol), CAN (209 mg, 0.38 mmol), and $CH_3CN$-water (v/v = 1/1, 2 mL). ($SiO_2$ column chromatography: *n*-hexane/AcOEt = 2/1)

IR (KBr) 3472, 1454, 1113 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 270 MHz): δ 1.71–1.86 (4H, m), 2.66–2.85 (2H,

m), 3.10 (1H, br s), 3.37 (3H, s), 3.52–3.69 (2H, m), 3.81–3.86 (1H, m), 7.17–7.33 (5H, m); $^{13}$C NMR ($CDCl_3$, 67.8 MHz): δ 31.9, 36.2, 39.1, 58.8, 70.8, 71.7, 125.5, 128.1, 128.2, 142.0; *Anal.* Calcd for $C_{12}H_{18}O$: C, 74.19; H, 9.34; Found: C, 73.90; H, 9.22.

**1-Benzyloxy-5-phenyl-3-pentanol (6d)**

**6d** (26 mg, 78%) was obtained as a colorless oil from **5f** (60 mg, 0.12 mmol), CAN (140 mg, 0.26 mmol), and $CH_3CN$-water (v/v = 1/1, 1.2 mL). ($SiO_2$ column chromatography: *n*-hexane/AcOEt = 5/1)

IR (KBr) 3420, 1192, 912 cm$^{-1}$; $^1$H NMR ($CDCl_3$, 270 MHz): δ 1.75–1.79 (4H, m), 2.61–2.85 (2H, m), 3.01 (1H, br s), 3.61–3.75 (2H, m), 3.77–3.89 (1H, m), 4.52 (2H, s), 7.18–7.32 (10H, m); $^{13}$C NMR ($CDCl_3$, 67.8 MHz): δ 32.0, 36.5, 39.2, 69.3, 70.9, 73.3, 125.6, 127.6, 127.7, 128.2, 128.3, 128.4, 142.1; *Anal.* Calcd for $C_{18}H_{22}O_2$: C, 79.96; H, 8.20; Found: C, 79.90; H, 8.32.

**1- Acetoxy-5-phenyl-3-pentanol (6e)**

**6e** (26 mg, 80%) was obtained as a colorless oil from **5g** (62 mg, 0.15 mmol), CAN (162 mg, 0.3 mmol), and $CH_3CN$-water (v/v = 1/1, 1.4 mL). ($SiO_2$ column chromatography: *n*-hexane/AcOEt = 3/1)

IR (KBr) 3420, 1732, 912 cm$^{-1}$; $^1$H NMR ($CDCl_3$, 270 MHz): δ 1.67–1.83 (4H, m), 1.98 (3H s), 2.55–2.79 (2H, m), 3.57–3.66 (1H, m), 4.02–4.10 (1H, m), 4.25–4.34 (1H, m), 7.11–7.24 (5H, m); $^{13}$C NMR ($CDCl_3$, 67.8 MHz): δ 21.1, 32.1, 36.5, 39.1, 61.7, 68.0, 125.8, 128.3, 128.3, 141.7, 171.3; *Anal.* Calcd for $C_{13}H_{18}O_3$: C, 70.24; H, 8.16; Found: C, 70.29; H, 8.20.

**1-Tosyloxy-5-phenyl-3-pentanol (6f)**

**6f** (42 mg, 90%) was obtained as a colorless oil from **5h** (76 mg, 0.14 mmol), CAN (156 mg, 0.28 mmol), and $CH_3CN$-water (v/v = 1/1, 1.4 mL). ($SiO_2$ column chromatography: *n*-hexane/AcOEt = 3/1)

IR (KBr) 3539, 1356, 1175 cm$^{-1}$; $^1$H NMR ($CDCl_3$, 270 MHz): δ 1.68–1.93 (5H, m), 2.45 (3H s), 2.61–2.79 (2H, m), 3.73–3.79 (1H, m), 4.08–4.16 (1H, m), 4.22–4.31 (1H, m), 7.15–7.32 (7H, m) , 7.77 (2H, d, $J$ = 8.4 Hz); $^{13}$C NMR ($CDCl_3$, 67.8 MHz): δ 32.0, 36.4, 39.1, 67.3, 37.7, 125.8, 127.8, 128.2, 128.4, 129.8, 132.7, 141.5, 144.7; *Anal.* Calcd for $C_{18}H_{22}O_4S$: C, 64.64; H, 6.63; S, 9.59 Found: C, 64.44; H, 6.74; S, 9.10; LRMS (FAB) *m/z* 335 ($MH^+$); HRMS (FAB) calcd for $C_{18}H_{23}O_4S$, 335.1317; found, 335.1332.

**1-Iodo-5-phenyl-3-pentanol (6g)**

**6g** (46 mg, 80%) was obtained as a colorless oil from **5i** (96 mg, 0.2 mmol), CAN (216 mg, 0.40 mmol), and $CH_3CN$-water (v/v = 1/1, 2.0 mL). ($SiO_2$ column chromatography: *n*-hexane/AcOEt =

5/1)

IR (KBr) 3366, 1495, 912 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 270 MHz): δ 1.69–1.99 (5H, m), 2.55–2.78 (2H, m), 3.19–3.25 (2H, m), 3.66–3.73 (1H, m), 7.11–7.25 (5H, m); $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 3.0, 32.0, 38.9, 40.7, 71.3, 125.9, 128.2, 128.4, 141.4; *Anal.* Calcd for $C_{11}H_{15}IO$: C, 45.54; H, 5.21; I, 43.74; Found: C, 45.83; H, 5.16; I, 43.00; LRMS (FAB) *m/z* 291 ($MH^+$); HRMS (FAB) calcd for $C_{11}H_{16}OI$, 291.0246; found, 291.0251.

**5-Phenyl-pentane-1,3-diol (6h)**

**6h** (24 mg, 73%) was obtained as a colorless oil from **5j** (90 mg, 0.18 mmol), CAN (396 mg, 0.72 mmol), and $CH_3CN$-water (v/v = 1/1, 1.2 mL). ($SiO_2$ column chromatography: *n*-hexane/AcOEt = 1/1 to AcOEt only)

IR (KBr) 3308, 912 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 270 MHz): δ 1.63–1.7 (4H, m), 2.26 (2H, br s), 2.56–2.77 (2H, m), 3.72–3.88 (3H, m), 7.09–7.25 (5H, m); $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 31.9, 38.3, 39.4, 61.9, 71.7, 125.8, 128.3, 128.3; LRMS (FAB) *m/z* 181 ($MH^+$); HRMS (FAB) calcd for $C_{11}H_{17}O_2$, 181.1228; found, 181.1227.

Reactions in Scheme 4

**(7*R*,3a*R*,7a*R*)-7-Bromo-2,3,3a,6,7,7a-hexahydrobenzofuran-2-ol (8)**

CAN (462 mg, 0.84 mmol) was added to a stirred solution of **7**[3)] (104 mg, 0.24 mmol) in acetonitrile (0.6 mL) and water (0.6 mL) at r.t. under an air. The reaction mixture was stirred at 60 °C for 30 min. and $K_2CO_3$ was added to the resulting solution. After filtration $K_2CO_3$, the filtrate was concentrated in vacuo. The crude product was purified by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 5/1) gave **8** (30 mg, 58%) as a colorless oil.

2/1 mixture of 2-position chiral carbon ($^1H$ NMR)

IR (KBr) 3379, 1649 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 270 MHz): δ 1.81–1.86 (1H, m), 2.13–2.41 (1H, m), 2.53–2.65 (1H, m), 2.91–3.11 (2H, m), 4.04–4.07 (0.6H, m), 4.33 (0.3H, t, *J* = 5.7 Hz), 4.44–4.53 (1H, m), 5.58–5.73 (3H, m); $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 30.1, 32.5, 36.4, 37.6, 39.5, 80.6, 81.0, 98.1, 100.0, 124.1, 124.1, 127.8, 128.9; LRMS (FAB) *m/z* 241 ($MNa^+$); HRMS (FAB) calcd for $C_8H_{11}O_2{}^{79}BrNa$, 240.9840; found, 240.9865, calcd for $C_8H_{11}O_2{}^{81}BrNa$, 242.9819; found, 242.9857.

**(3a*R*,7a*R*,7*R* )-7-Bromo-3a,6,7,7a-tetrahydro-3H-benzofuran-2-one (9)**

$NaH_2PO_4$ (35 mg, 0.29 mmol), 2-metyl-2-butene (0.05 mL, 0.48 mmol) and $NaClO_2$ (13 mg, 014 mmol) were added to a stirred solution of **8** (21 mg, 0.10 mmol) in tBuOH-water (v/v = 5/1, 1.0 mL) at r.t. under an air. The reaction mixture was stirred at r.t. for 6 hr, and poured into saturated 10% aqueous HCl. The organic layer was separated, and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated in vacuo.

Purification of the residue by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 4/1) gave **9** (18 mg, 86%) as a colorless oil.

IR (KBr) 1788, 1217 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 270 MHz): δ 2.40–2.51 (2H, m), 2.77–2.89 (2H, m), 3.28 (1H, br s), 4.44–4.46 (1H, m), 4.78–4.80 (1H, m), 5.56–5.60 (1H, m), 5.72–5.76 (1H, m); $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 28.8, 33.3, 35.1, 43.3, 79.2, 124.2, 125.2, 175.0; LRMS (FAB) *m/z* 217 ($MH^+$); HRMS (FAB) calcd for $C_8H_{10}O_2{}^{79}Br$, 216.9865; found, 216.9870, calcd for $C_8H_{10}O_2{}^{81}Br$, 218.9844; found, 218.9870.

**(2*S*,5*R*,3a*R*,6a*S*)-2,5-bisiodomethyl-hexahydrofuro[2,3-b]furan (11a)**

CAN (428 mg, 0.78 mmol) was added to a stirred solution of **10a**[4)] (116 mg, 0.20 mmol) in acetonitrile (1.0 mL) and water (1.0 mL) at r.t. under an air. The reaction mixture was stirred at 60 °C for 30 min. $K_2CO_3$ was added to the reaction mixture. After filtrating $K_2CO_3$, the filtrate was concentrated in vacuo. The crude product was purified by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 4/1) gave **11a** (46 mg, 61%) as a colorless oil.

IR (KBr) 1018, 742 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 270 MHz): δ 1.68–1.77 (2H, m), 2.36–2.41 (2H, m), 2.88–3.07 (1H, m), 3.37–3.43 (4H, m), 4.18–4.22 (2H, m), 5.75 (1H, d, *J* = 5.4 Hz); $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 9.9, 37.9, 43.6, 80.2, 110.6; LRMS (FAB) *m/z* 395 ($MH^+$); HRMS (FAB) calcd for $C_8H_{13}O_2I_2$, 394.9005; found, 394.8982.

**(1*S*,3*R*,4*R*,6*S*,8*R*,10*R*)-10-iodomethyl-6-cyanomethyl-3,4-diphenyl-2,5,11-trioxabicyclo[6.3.0]undecane (10b)**

NaCN (106 mg, 2.16 mmol) was added to a stirred solution of **10a** (850 mg, 1.44 mmol) in DMSO (2.9 mL) at r.t. under an air. The reaction mixture was stirred at 70 °C for 2 hr and poured into water. The organic layer was separated, and the aqueous layer was extracted with AcOEt. The combined organic layer was washed with brine, dried over $Na_2SO_4$, and concentrated in vacuo. The crude product was purified by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 4/1) gave **10b** (284 mg, 40%) as a colorless oil.

IR (KBr) 1452, 742 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 270 MHz): δ 1.52-1.61 (1H, m), 2.31-2.45 (6H, m), 3.11 (1H, t, *J* = 9.2 Hz), 3.35 (1H, q, *J* = 4.8 Hz), 4.19-4.65 (4H, m), 5.92 (1H, d, *J* = 3.8 Hz), 6.86-6.88 (4H, m), 7.10-7.13 (6H, m); $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 11.0, 22.6, 32.4, 37.1, 44.5, 73.5, 78.8, 80.7, 86.6, 107.2, 127.0, 127.2, 127.3, 127.5, 127.6, 127.7, 127.9, 137.9, 138.1; LRMS (FAB) *m/z* 490 ($MH^+$); HRMS (FAB) calcd for $C_{23}H_{25}O_3NI$, 490.0879; found, 490.0861.

**(2*R*,5*S*,3a*R*,6a*S*)-2- iodomethyl-5-cyanomethyl-hexahydrofuro[2,3-b]furan (11b)**

CAN (420 mg, 0.78 mmol) was added to a stirred solution of **10b** (108 mg, 0.22 mmol) in acetonitrile (1.1 mL) and water (1.1 mL) at r.t. under an air. The reaction mixture was stirred at 60 °C

for 30 min. $K_2CO_3$ was added to the reaction mixture. After filtrating $K_2CO_3$, the filtrate was concentrated in vacuo. The crude product was purified by $SiO_2$ column chromatography (*n*-hexane/AcOEt = 2/1) gave **11b** (36 mg, 56%) as a colorless oil.

IR (KBr) 1456, 912 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 270 MHz): δ 1.73–1.78 (2H, m), 2.41–2.44 (2H, m), 2.80–2.96 (3H, m), 3.37–3.43 (2H, m), 4.17–4.36 (2H, m), 5.68 (1H, d, *J* = 5.4 Hz); $^{13}C$ NMR ($CDCl_3$, 67.8 MHz): δ 9.7, 24.8, 37.0, 37.8, 43.5, 75.4, 80.4, 110.3, 117.0; LRMS (FAB) *m/z* 294 ($MH^+$); HRMS (FAB) calcd for $C_9H_{13}O_2NI$, 293.9991; found, 293.9993.

**References**


1) Mohammadpoor-Baltork, I.; Tangestaninejad, S.; Aliyan, H.; Mirkhani, V. *Synthetic Commun*. **2000**, *30*, 2365.
2) Johnson, W. S.; Crackett, P. H.; Elliott, J. D.; Jagodzinski, J. J.; Lindel, S. D.; Natarajan, S. *Tetrahedron Lett*. **1984**, *25*, 3951.
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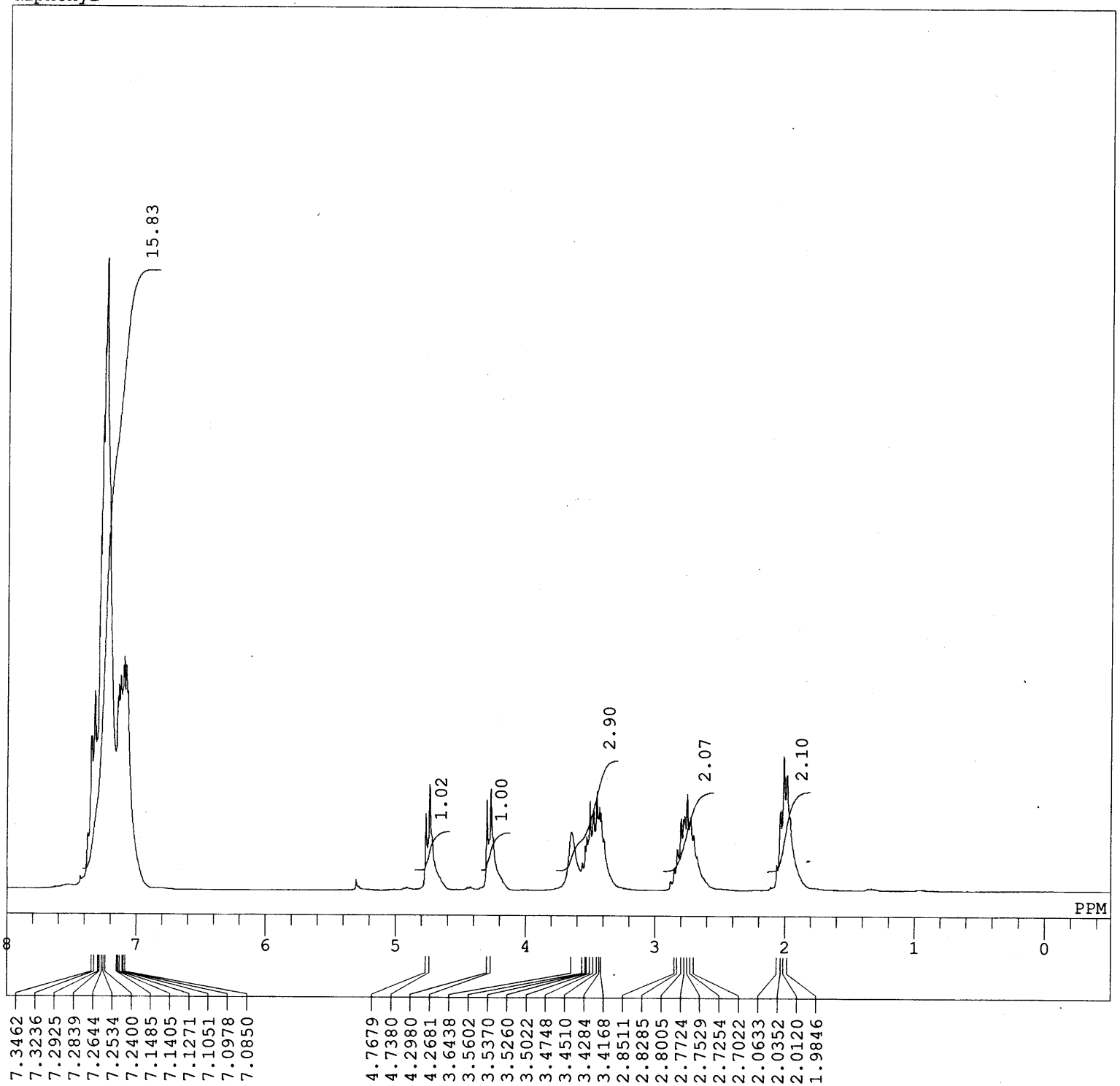
4) Fujioka, H.; Ohba, Y.; Hirose, H.; Murai, K.; Kita, Y. *Angew. Chem. Int. Ed.* **2005**, *44*, 734.

diphenyl

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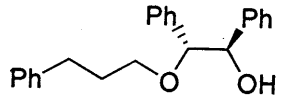
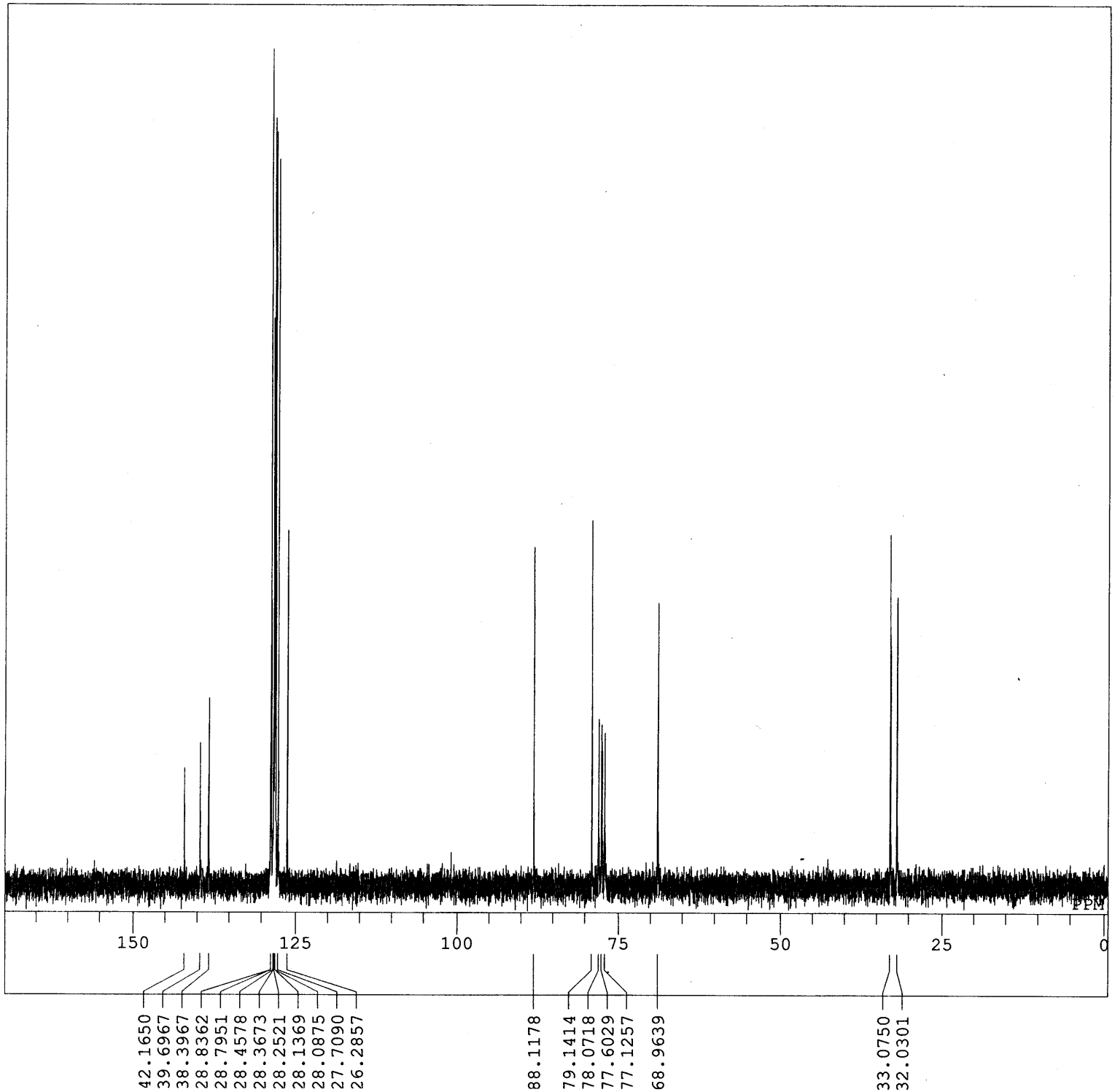



**3a**

3a

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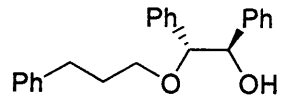


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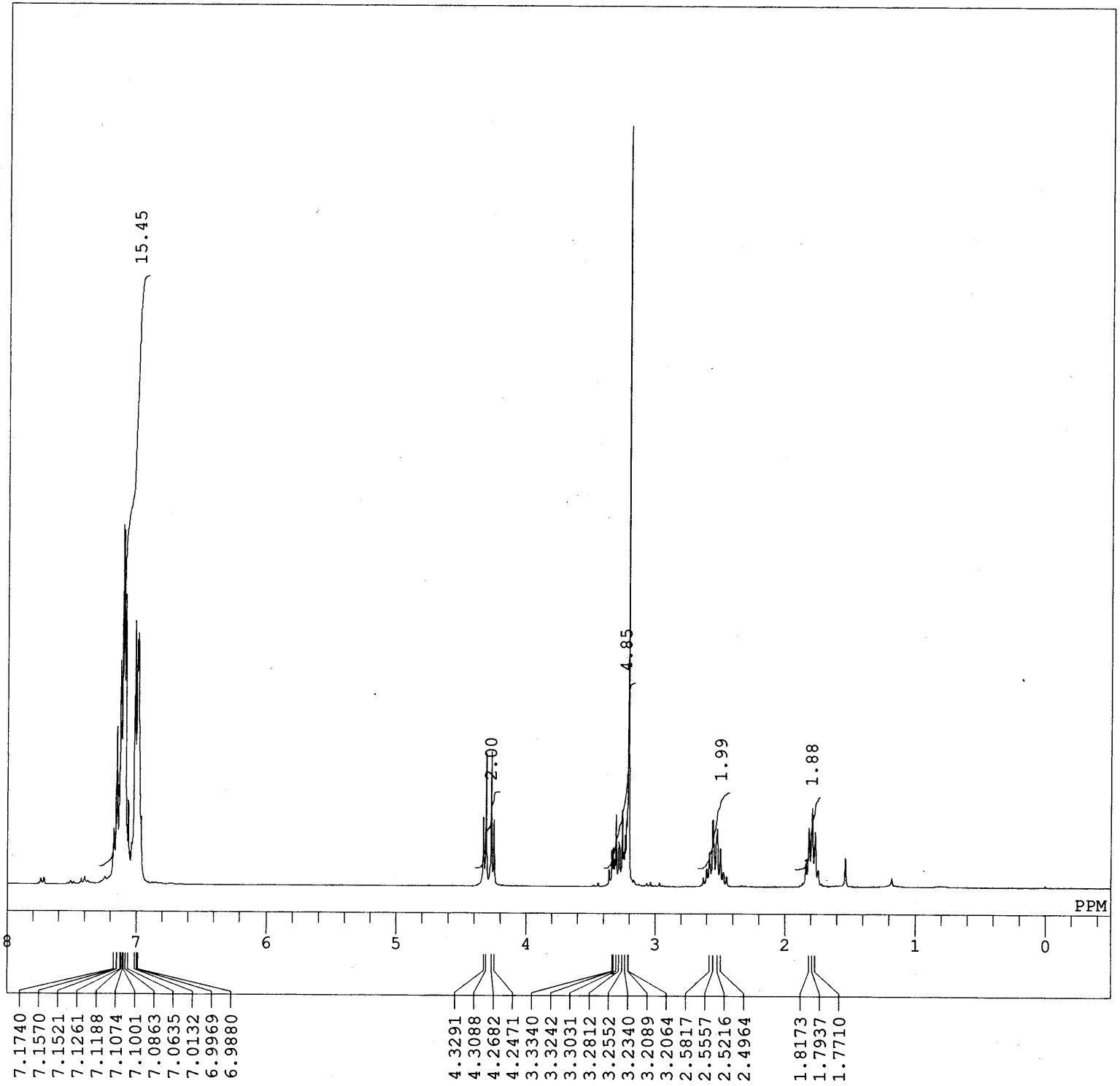
3b

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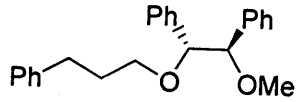
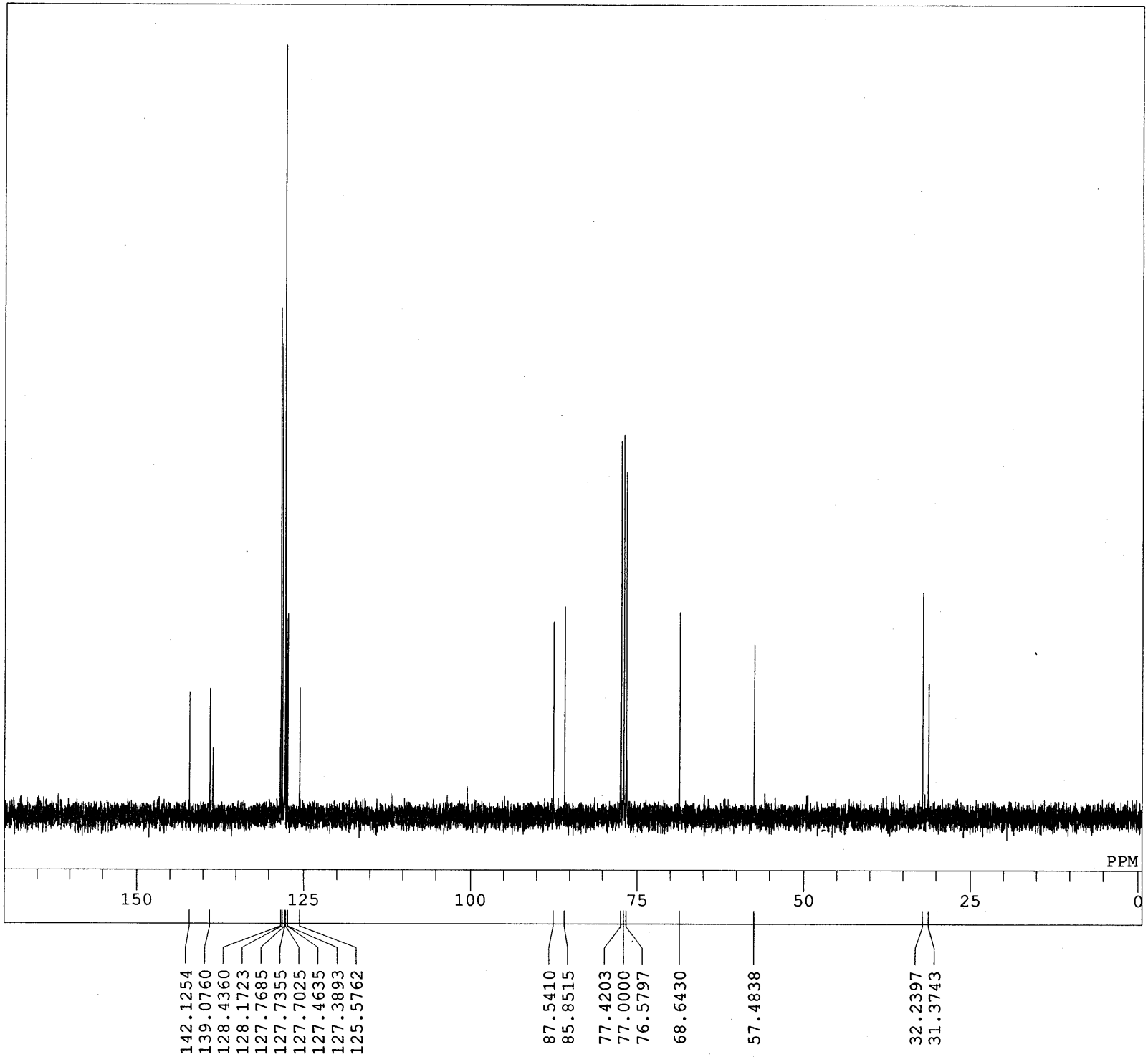



**3b**

3b

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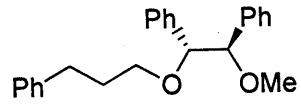


**3b**

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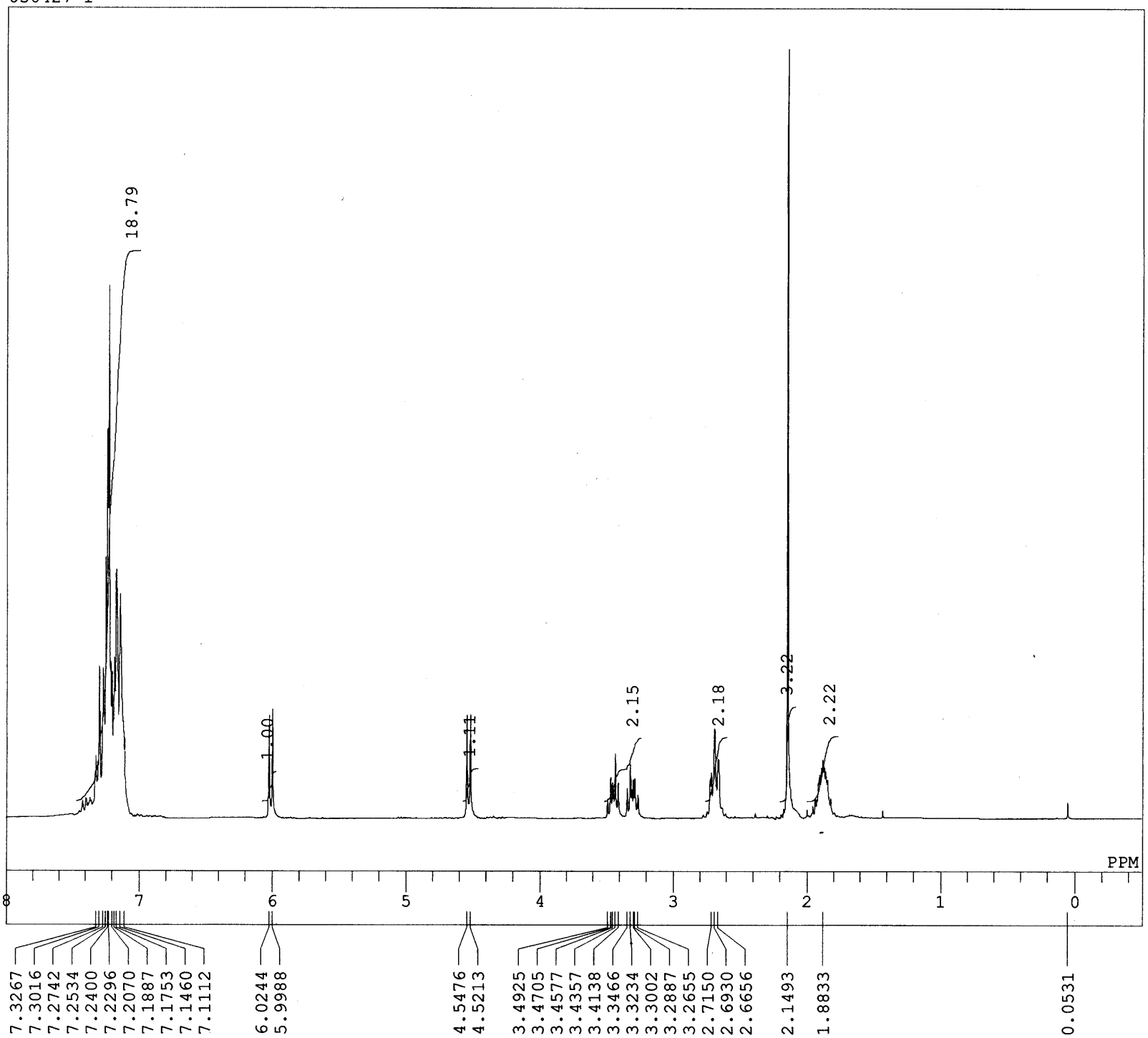


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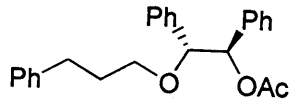
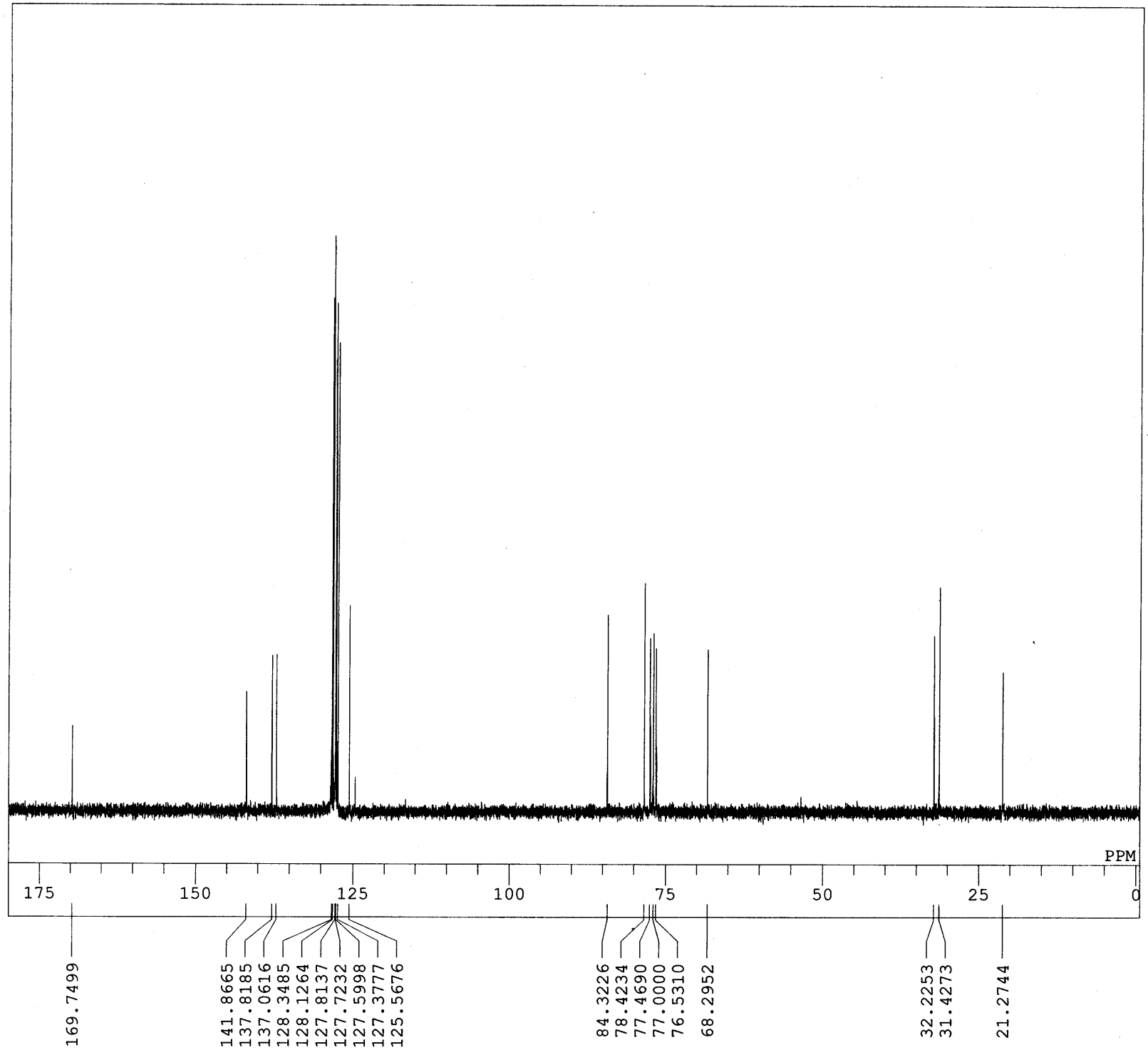
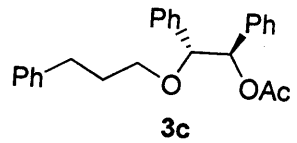



**3c**

3c

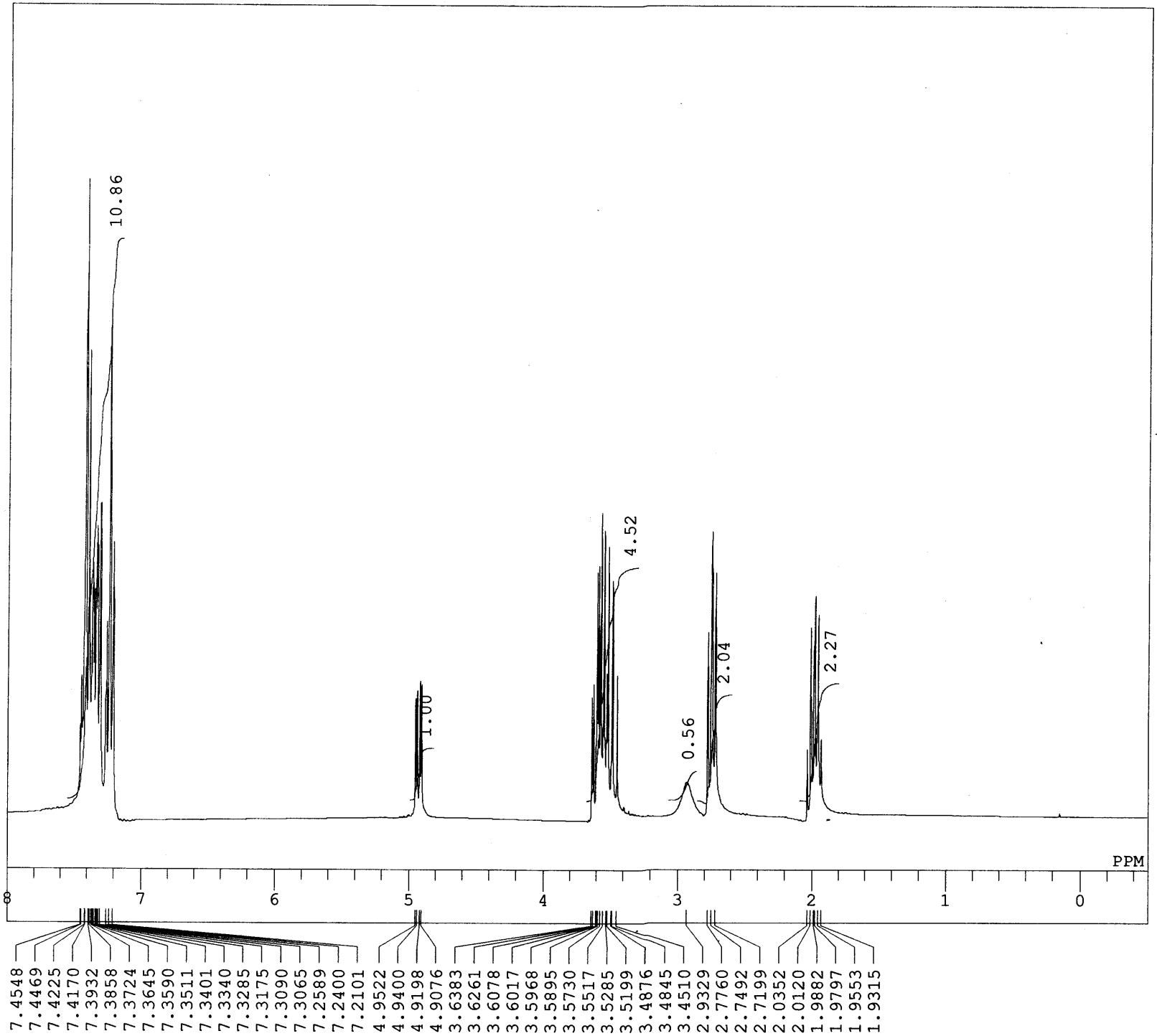



| | |
|---|---|
| DFILE | E:\030427-2.als |
| COMNT | 3c |
| DATIM | Sat Jan 22 14:08:00 2005 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 225 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.00 usec |
| IRNUC | 1H |
| CTEMP | 19.8 c |
| SLVNT | CDCL3 |
| EXREF | 77.00 ppm |
| BF | 0.12 Hz |
| RGAIN | 26 |

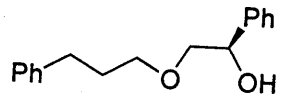



**3c**

3d

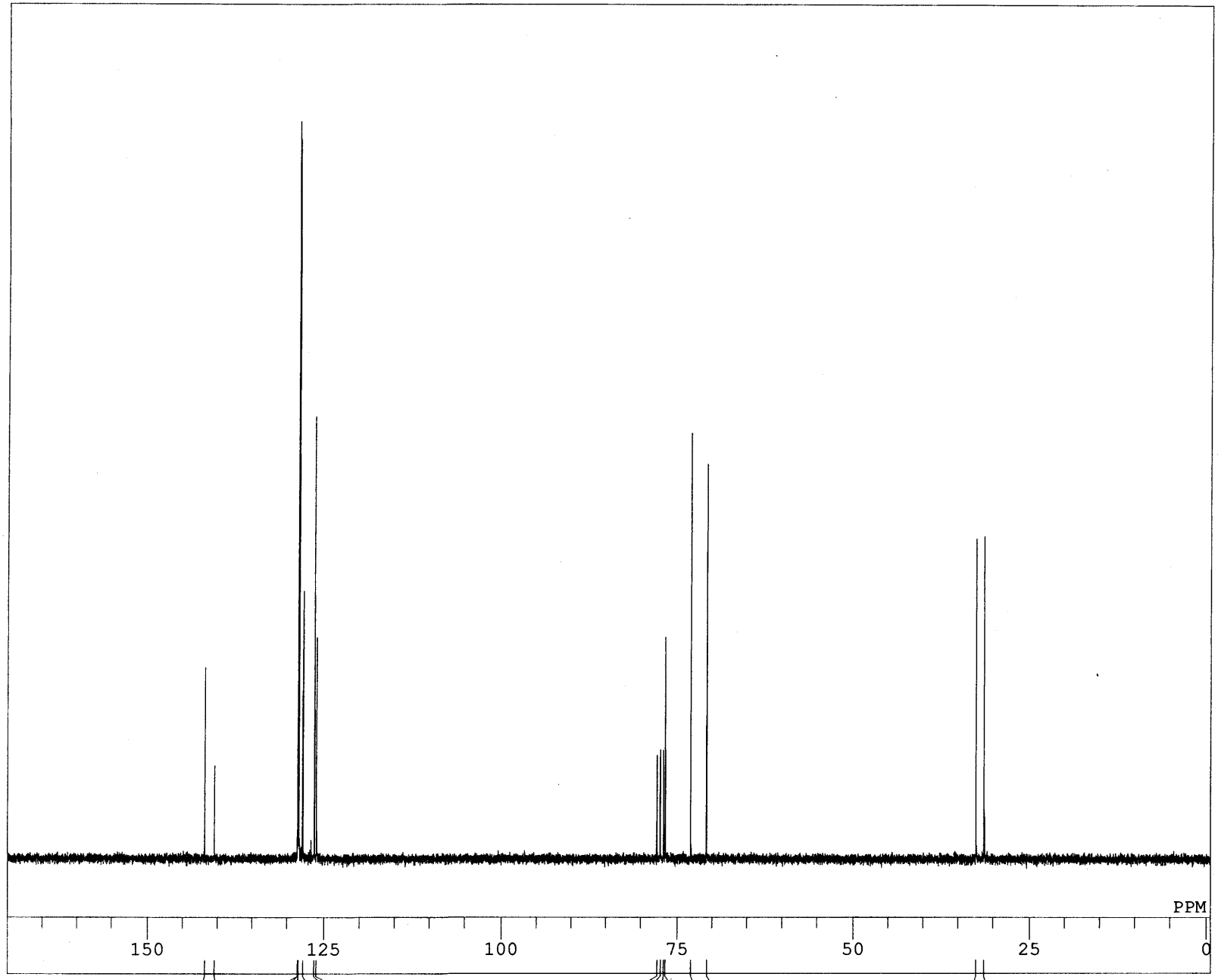



| | |
|---|---|
| DFILE | E:\CAN\1-phenyl\H-NMR.als |
| COMNT | 3d |
| DATIM | Sat Dec 11 10:21:09 2004 |
| OBNUC | 1H |
| EXMOD | NON |
| OBFRQ | 270.05 MHz |
| OBSET | 112.00 KHz |
| OBFIN | 5800.00 Hz |
| POINT | 32768 |
| FREQU | 5402.40 Hz |
| SCANS | 16 |
| ACQTM | 6.0655 sec |
| PD | 0.9350 sec |
| PW1 | 5.90 usec |
| IRNUC | 1H |
| CTEMP | 20.4 c |
| SLVNT | CDCL3 |
| EXREF | 7.24 ppm |
| BF | 0.12 Hz |
| RGAIN | 12 |

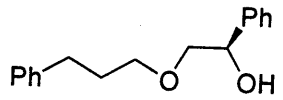



**3d**

3d

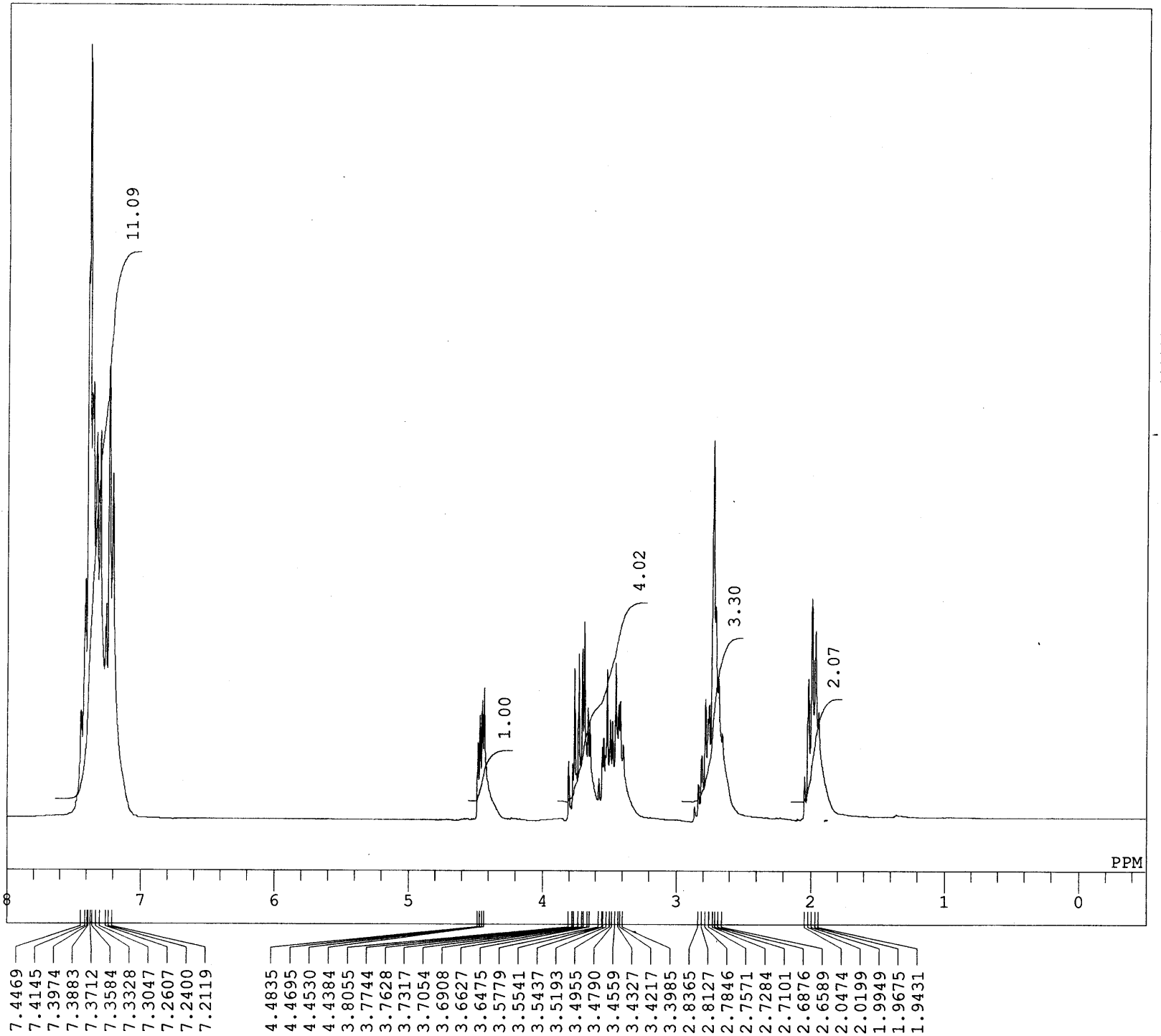



| | |
|---|---|
| DFILE | E:\CAN\1-phenyl\C-NMR.als |
| COMNT | 3d |
| DATIM | Sat Dec 11 10:30:40 2004 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 129 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.10 usec |
| IRNUC | 1H |
| CTEMP | 21.0 c |
| SLVNT | CDCL3 |
| EXREF | 0.00 ppm |
| BF | 0.12 Hz |
| RGAIN | 26 |

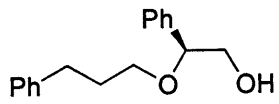



**3d**

3e




| | |
|---|---|
| DFILE | E:\CAN\2-phenyl\H-NMR.als |
| COMNT | 3e |
| DATIM | Sat Dec 11 10:06:53 2004 |
| OBNUC | 1H |
| EXMOD | NON |
| OBFRQ | 270.05 MHz |
| OBSET | 112.00 KHz |
| OBFIN | 5800.00 Hz |
| POINT | 32768 |
| FREQU | 5402.40 Hz |
| SCANS | 16 |
| ACQTM | 6.0655 sec |
| PD | 0.9350 sec |
| PW1 | 5.90 usec |
| IRNUC | 1H |
| CTEMP | 20.7 c |
| SLVNT | CDCL3 |
| EXREF | 7.24 ppm |
| BF | 0.12 Hz |
| RGAIN | 10 |

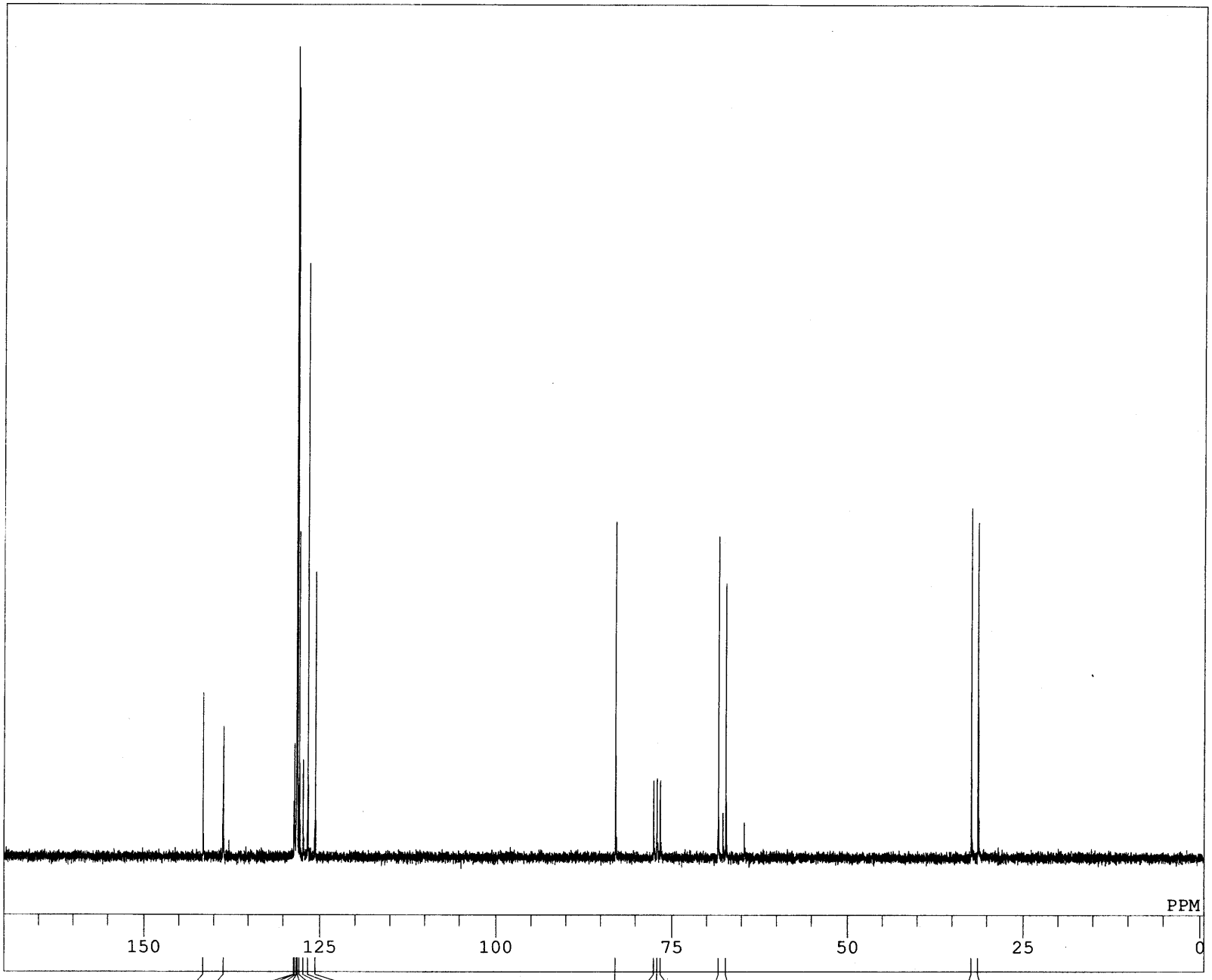



**3e**

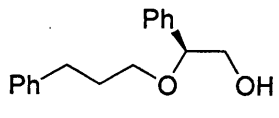
7.4469
7.4145
7.3974
7.3883
7.3712
7.3584
7.3328
7.3047
7.2607
7.2400
7.2119

4.4835
4.4695
4.4530
4.4384
3.8055
3.7744
3.7628
3.7317
3.7054
3.6908
3.6627
3.6475
3.5779
3.5541
3.5437
3.5193
3.4955
3.4790
3.4559
3.4327
3.4217
3.3985
2.8365
2.8127
2.7846
2.7571
2.7284
2.7101
2.6876
2.6589
2.0474
2.0199
1.9949
1.9675
1.9431

3e

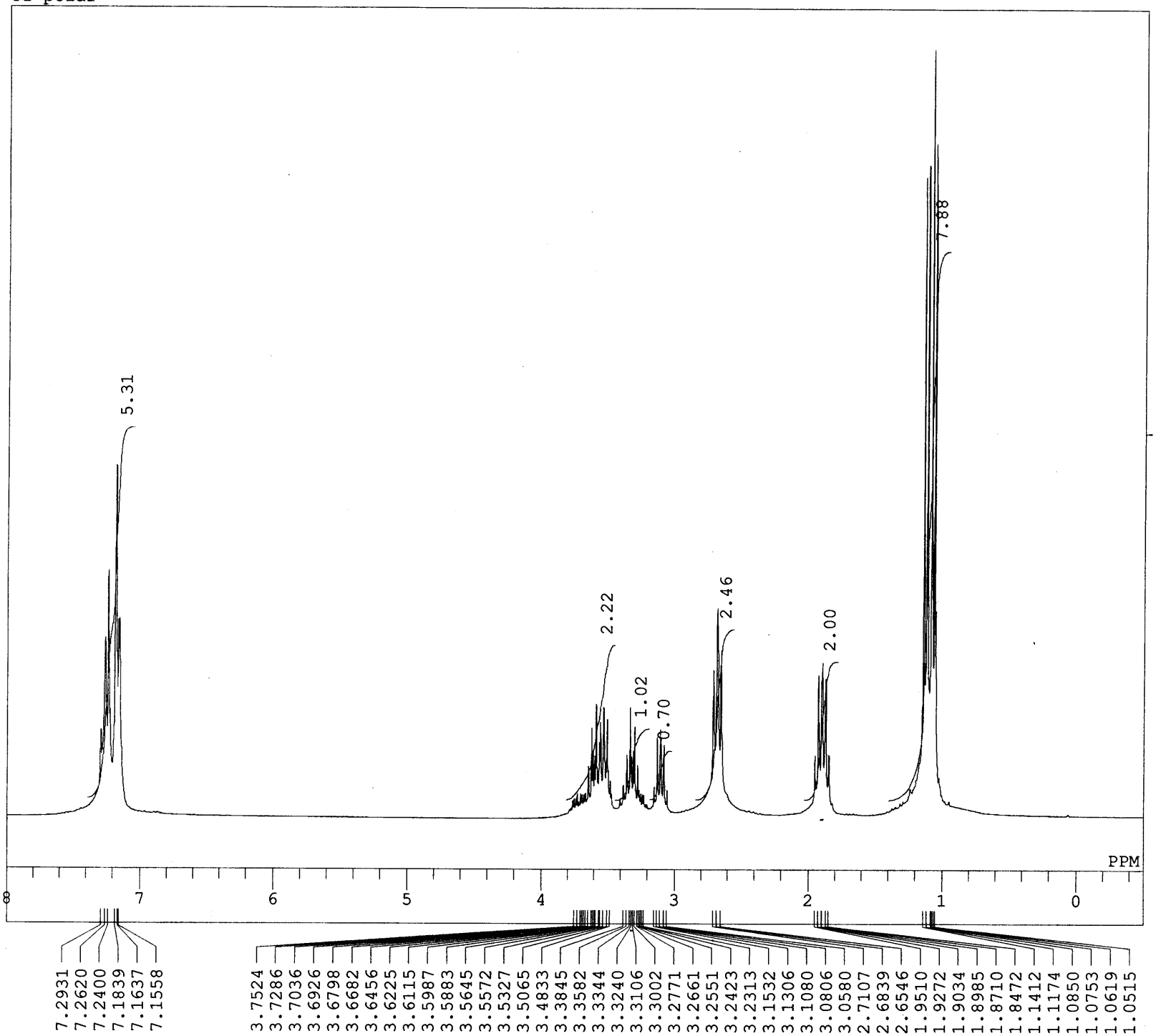



DFILE E:\CAN\2-phenyl\C-NMR.als
COMNT 3e
DATIM Sat Dec 11 10:14:30 2004
OBNUC 13C
EXMOD BCM
OBFRQ 67.80 MHz
OBSET 135.00 KHz
OBFIN 5200.00 Hz
POINT 32768
FREQU 18315.00 Hz
SCANS 129
ACQTM 1.7891 sec
PD 1.2110 sec
PW1 4.10 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 25

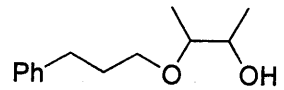



**3e**

3f-polar



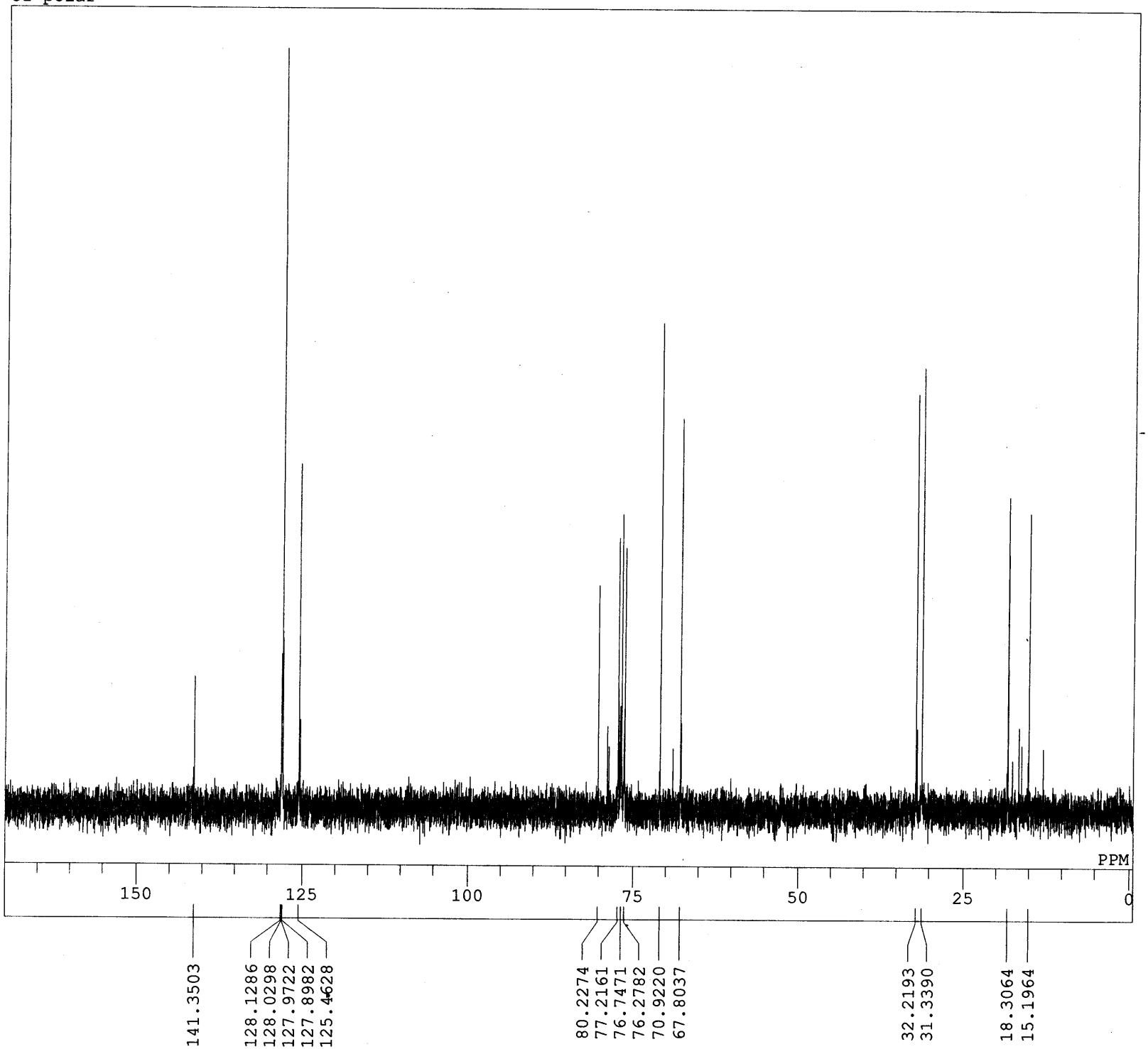

| | |
|---|---|
| DFILE | E:\CAN\1,2-dimethyl up\H-NMR |
| COMNT | 3f-polar |
| DATIM | Sat Dec 11 10:35:08 2004 |
| OBNUC | 1H |
| EXMOD | NON |
| OBFRQ | 270.05 MHz |
| OBSET | 112.00 KHz |
| OBFIN | 5800.00 Hz |
| POINT | 32768 |
| FREQU | 5402.40 Hz |
| SCANS | 16 |
| ACQTM | 6.0655 sec |
| PD | 0.9350 sec |
| PW1 | 5.90 usec |
| IRNUC | 1H |
| CTEMP | 20.3 c |
| SLVNT | CDCL3 |
| EXREF | 7.24 ppm |
| BF | 0.12 Hz |
| RGAIN | 14 |



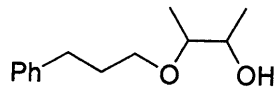

(cis / trans = 1 / 1)

**3f**

3f polar



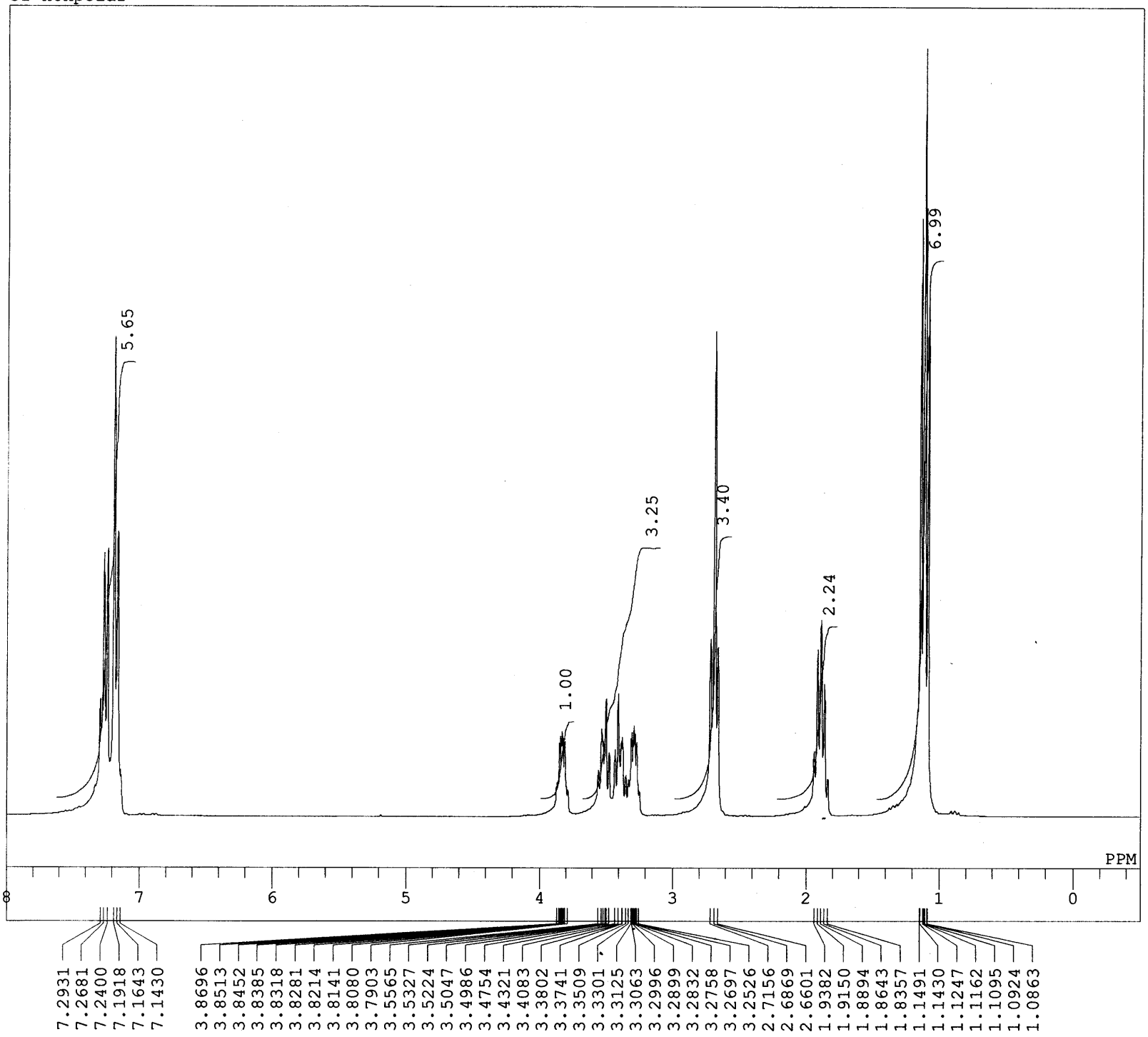

| | |
|---|---|
| DFILE | E:\CAN\1,2-dimethyl up\C-NMR |
| COMNT | 3f polar |
| DATIM | Sat Dec 11 10:43:31 2004 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 134 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.10 usec |
| IRNUC | 1H |
| CTEMP | 20.9 c |
| SLVNT | CDCL3 |
| EXREF | 0.00 ppm |
| BF | 0.12 Hz |
| RGAIN | 26 |



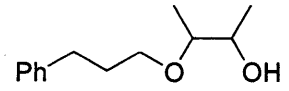

(cis / trans = 1 / 1)

**3f**

3f nonpolar



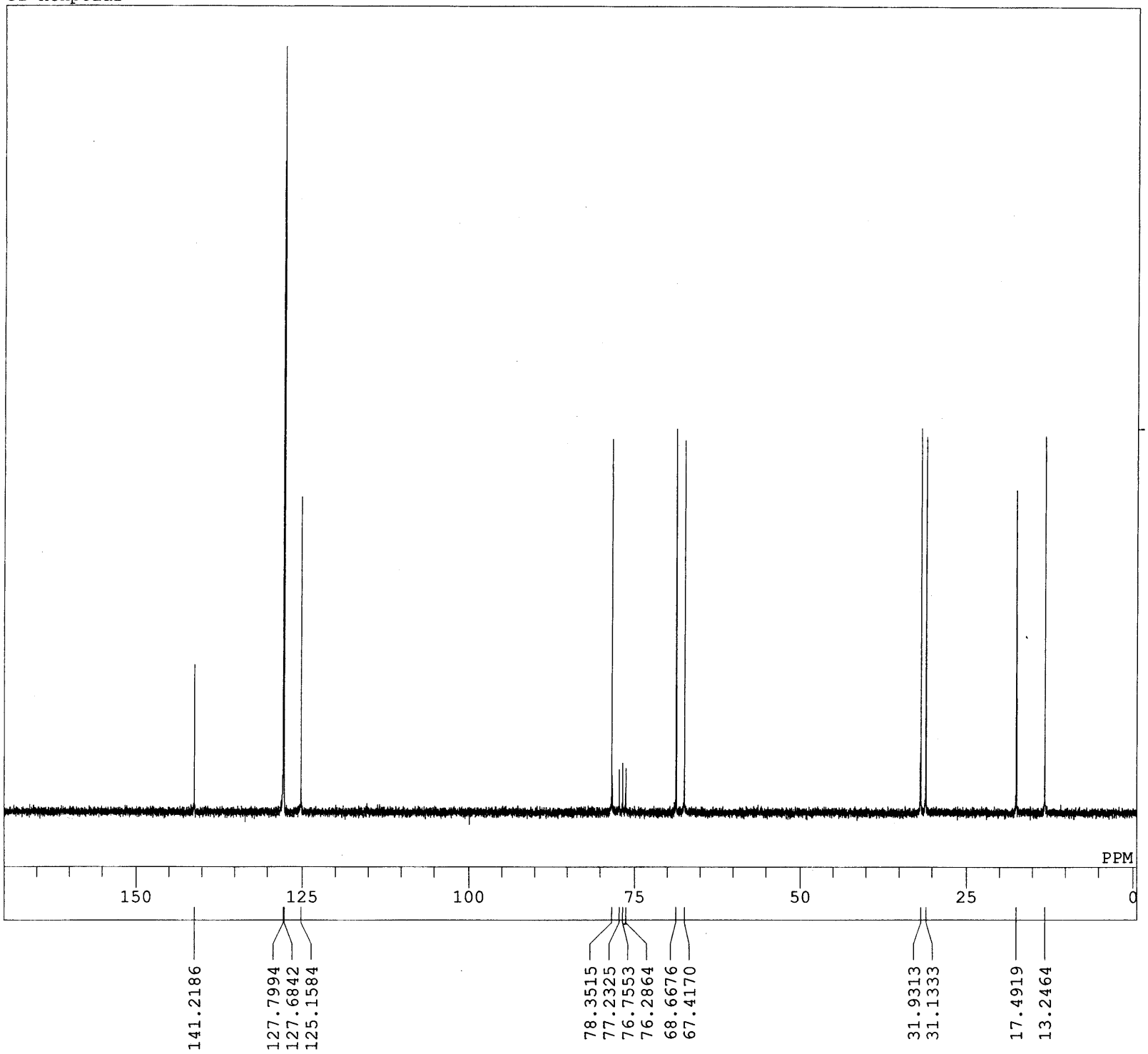

DFILE E:\CAN\1,2-dimethyl down\H-N
COMNT 3f nonpolar
DATIM Sat Dec 11 10:49:57 2004
OBNUC 1H
EXMOD NON
OBFRQ 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.00 Hz
POINT 32768
FREQU 5402.40 Hz
SCANS 16
ACQTM 6.0655 sec
PD 0.9350 sec
PW1 5.90 usec
IRNUC 1H
CTEMP 20.0 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.12 Hz
RGAIN 7



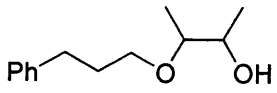

(cis / trans = 1 / 1)

**3f**

3f nonpolar




| | |
|---|---|
| DFILE | E:\CAN\1,2-dimethyl down\C-N |
| COMNT | 3f nonpolar |
| DATIM | Sat Dec 11 10:53:31 2004 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 33 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.10 usec |
| IRNUC | 1H |
| CTEMP | 20.5 c |
| SLVNT | CDCL3 |
| EXREF | 0.00 ppm |
| BF | 0.12 Hz |
| RGAIN | 24 |

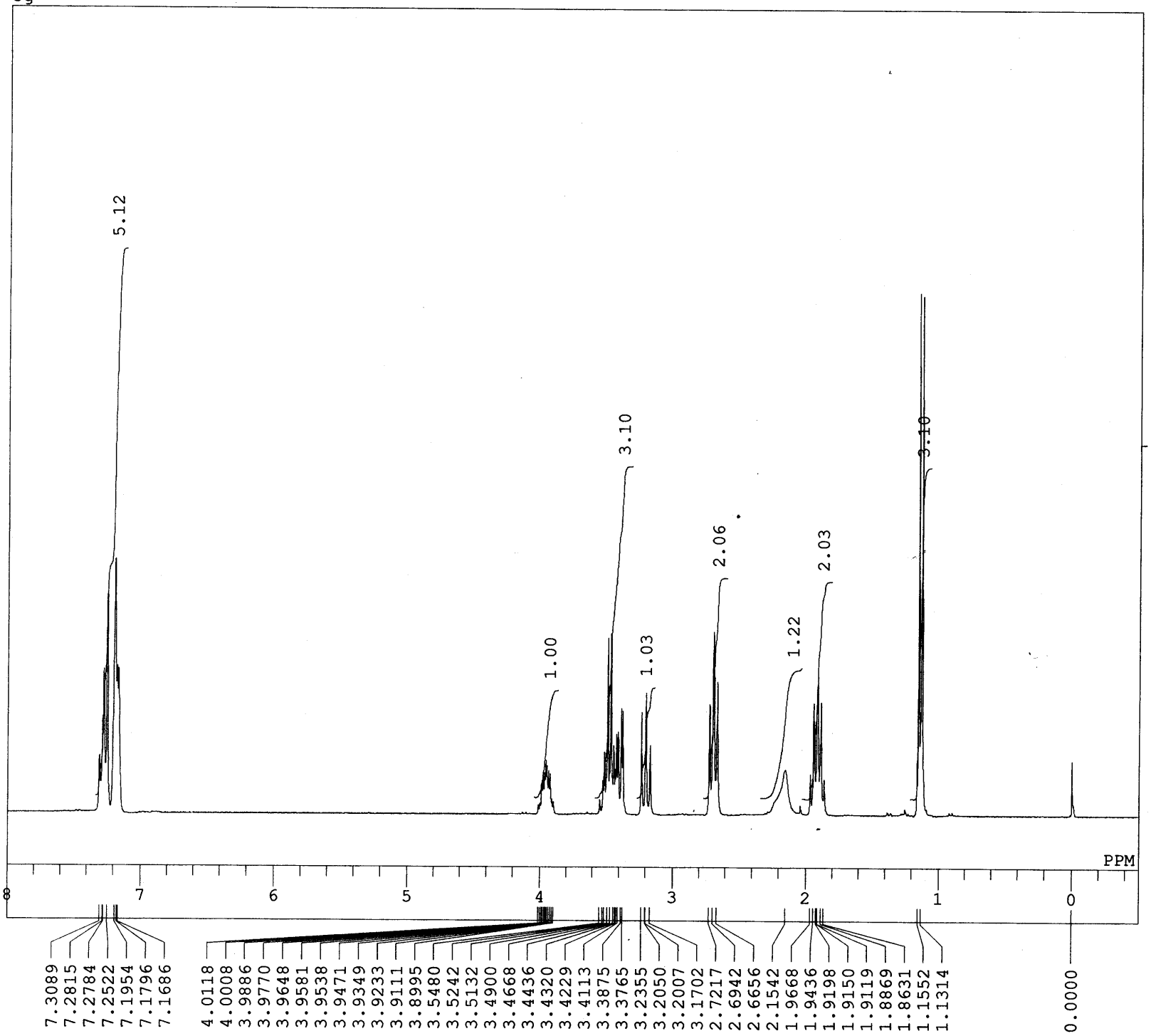



(cis / trans = 1 / 1)

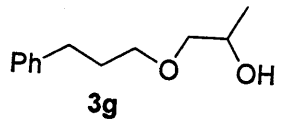
**3f**

E:\030638-3.als

3g



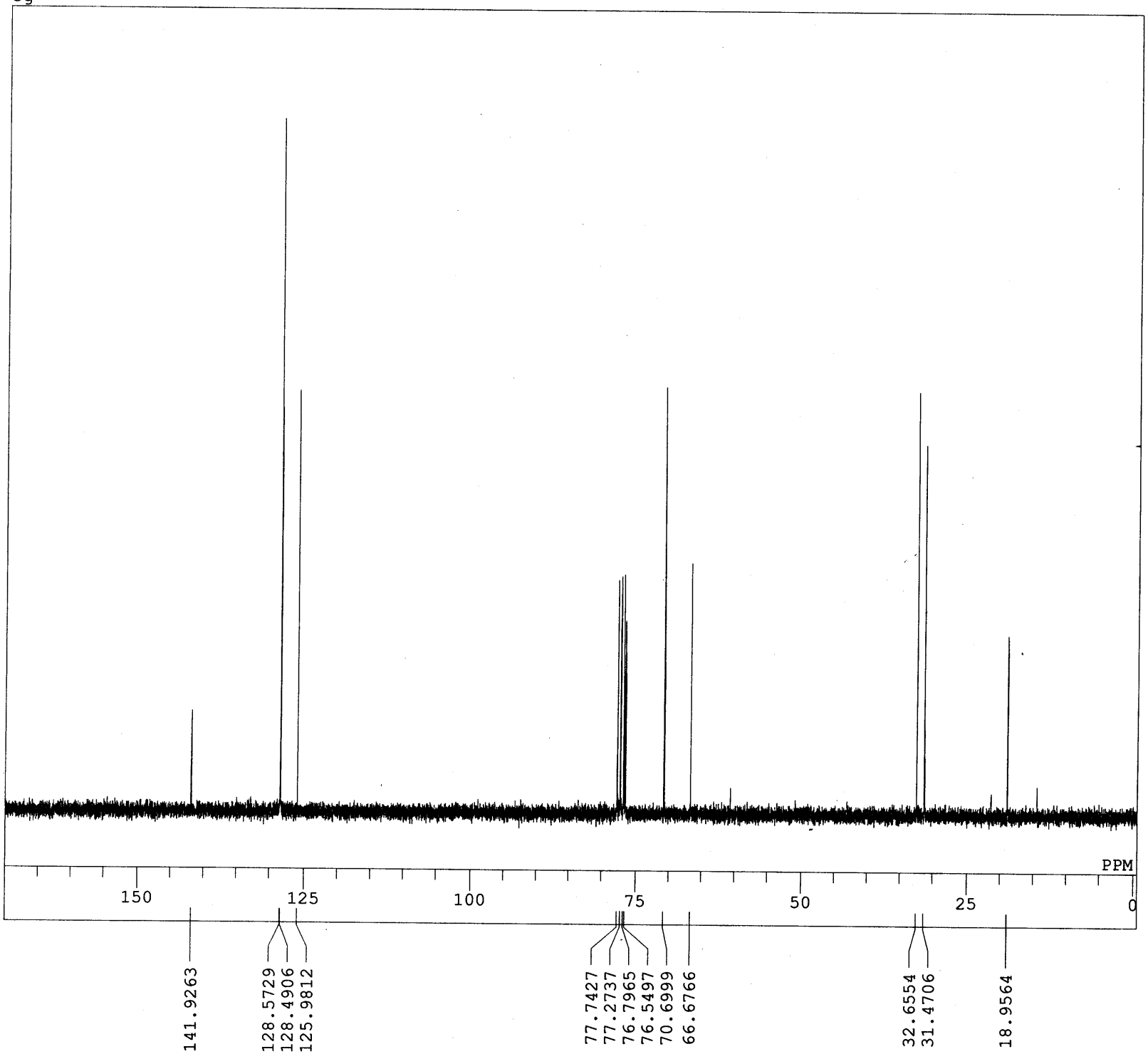

DFILE E:\030638-3.als
COMNT 3g
DATIM Wed Jun 08 14:35:30 2005
OBNUC 1H
EXMOD NON
OBFRQ 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.00 Hz
POINT 32768
FREQU 5402.40 Hz
SCANS 16
ACQTM 6.0655 sec
PD 0.9350 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 16



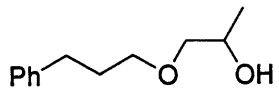

**3g**

E:\030638-2.als

3g



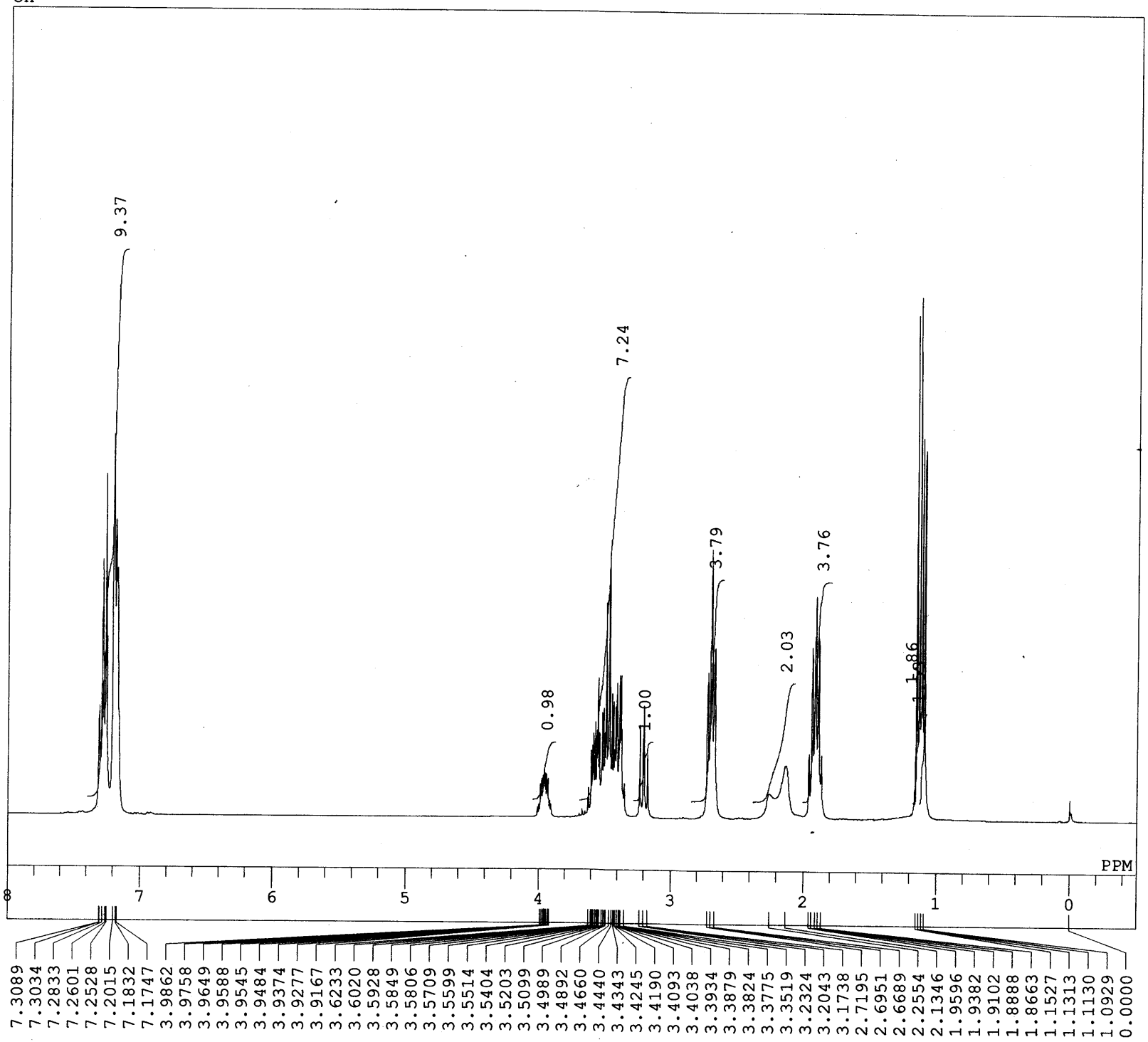

| | |
|---|---|
| DFILE | E:\030638-2.als |
| COMNT | 3g |
| DATIM | Wed Jun 08 14:29:58 2005 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 425 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.00 usec |
| IRNUC | 1H |
| CTEMP | 23.0 c |
| SLVNT | CDCL3 |
| EXREF | 0.00 ppm |
| BF | 0.12 Hz |
| RGAIN | 26 |



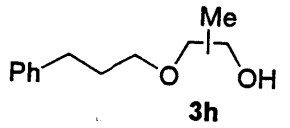

**3g**

E:\030644-1.als

3h



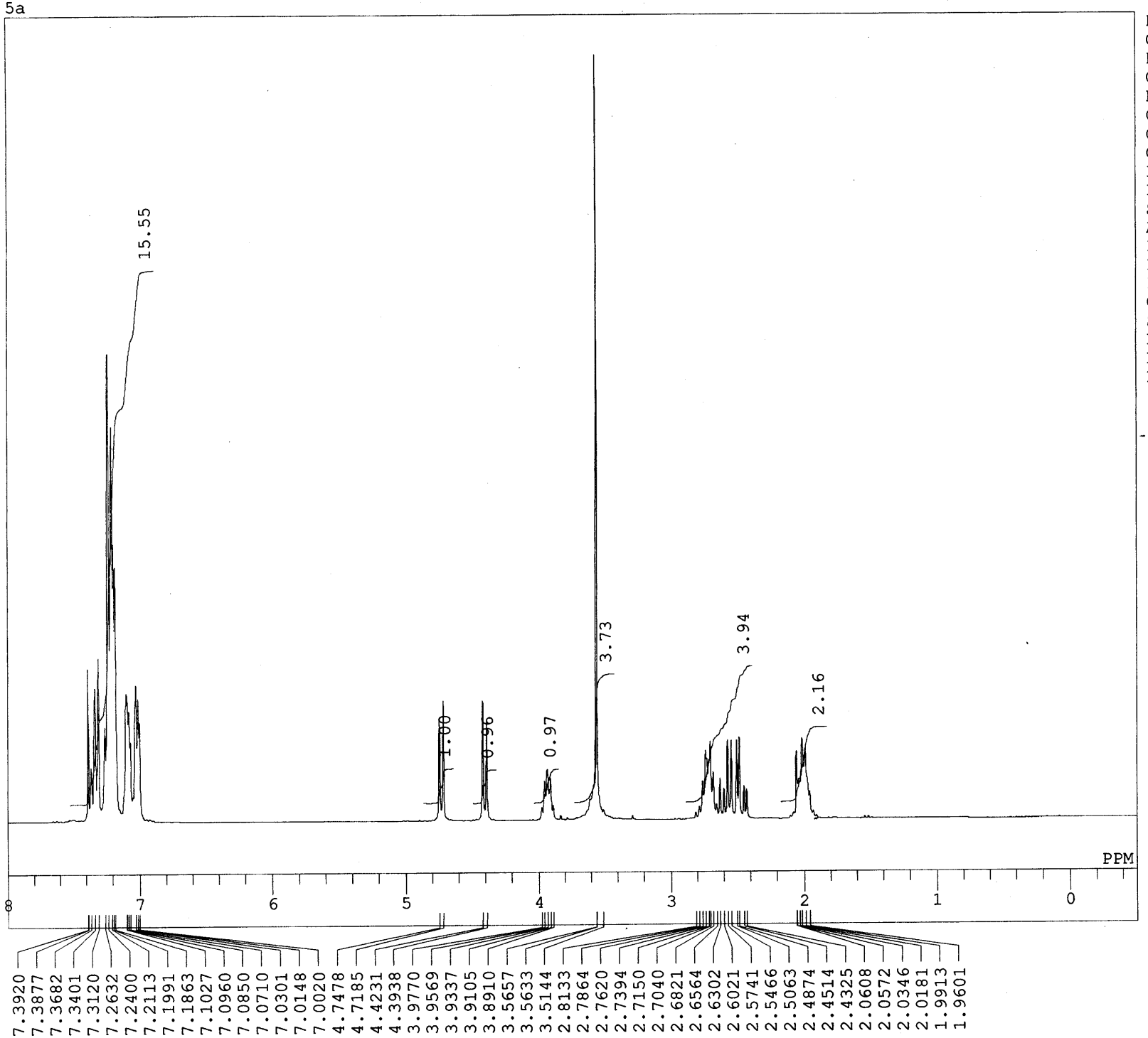

DFILE E:\030644-1.als
COMNT 3h
DATIM Thu Jun 09 19:02:54 2005
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.00 Hz
POINT 32768
FREQU 6006.01 Hz
SCANS 16
ACQTM 5.4559 sec
PD 1.5440 sec
PW1 5.60 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 14



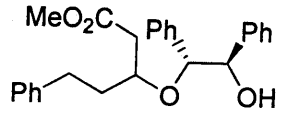

**3h**

(ca. 1/1 mixture)

5a

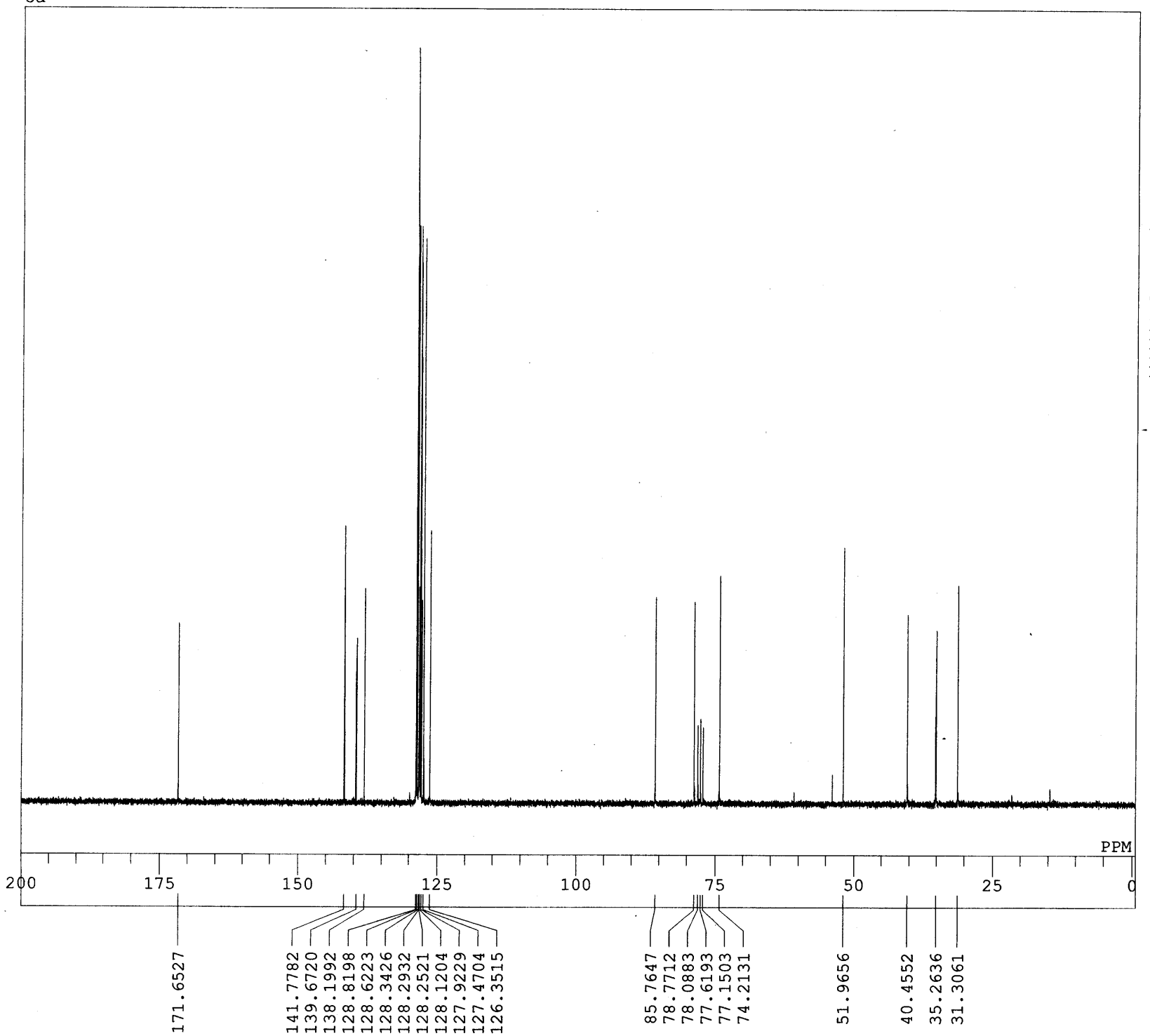



| | |
|---|---|
| DFILE | E:\CAN\Ph-CO2Me\H-NMR.als |
| COMNT | 5a |
| DATIM | Wed Dec 15 15:07:57 2004 |
| OBNUC | 1H |
| EXMOD | NON |
| OBFRQ | 270.05 MHz |
| OBSET | 112.00 KHz |
| OBFIN | 5800.00 Hz |
| POINT | 32768 |
| FREQU | 5402.40 Hz |
| SCANS | 32 |
| ACQTM | 6.0655 sec |
| PD | 0.9350 sec |
| PW1 | 5.90 usec |
| IRNUC | 1H |
| CTEMP | 19.3 c |
| SLVNT | CDCL3 |
| EXREF | 7.24 ppm |
| BF | 0.12 Hz |
| RGAIN | 9 |

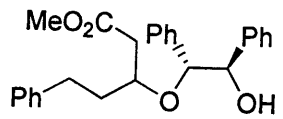



**5a**

5a

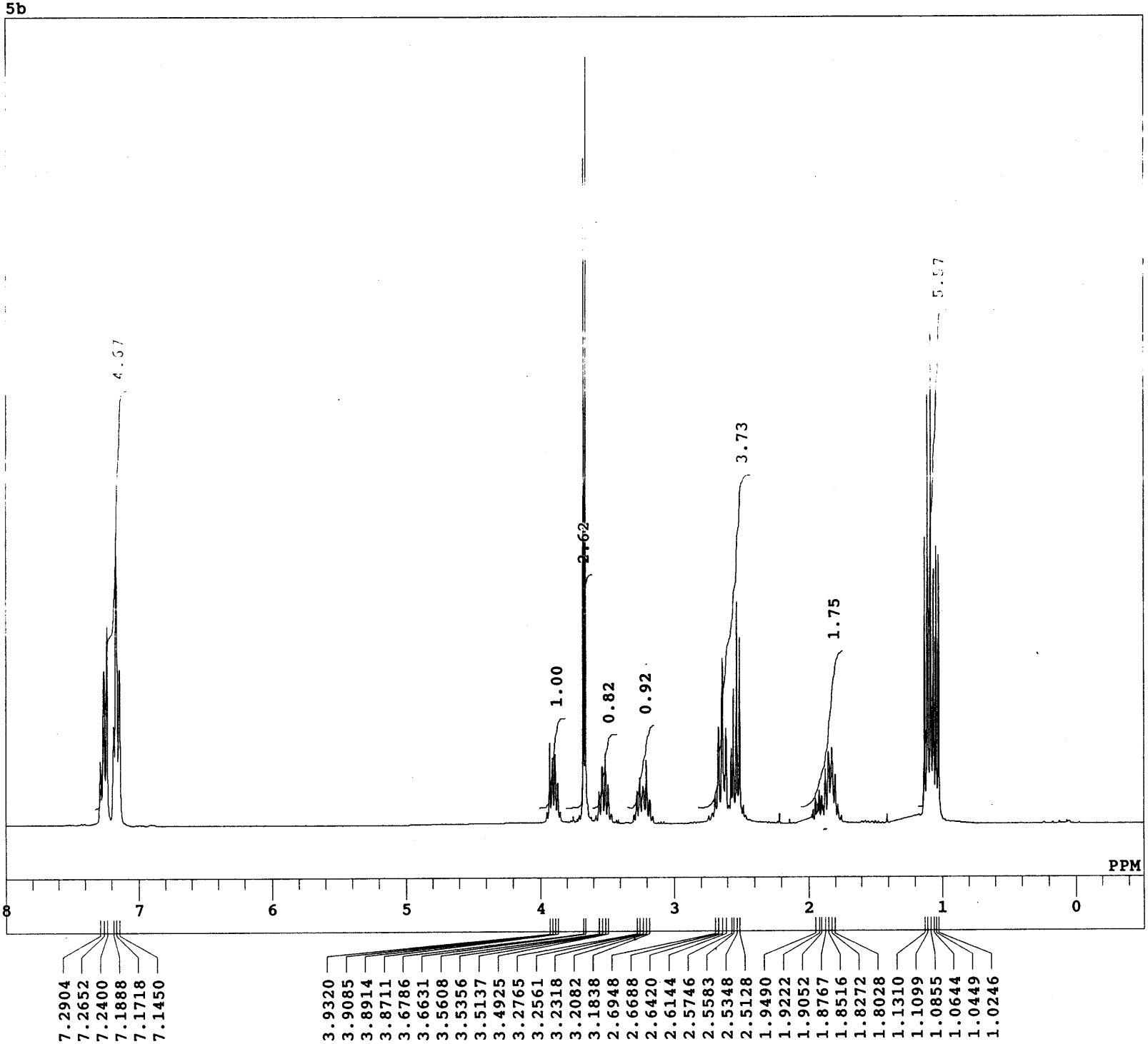



| | |
|---|---|
| DFILE | E:\CAN\Ph-CO2Me\C-NMR.als |
| COMNT | 5a |
| DATIM | Wed Dec 15 15:14:49 2004 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 128 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.10 usec |
| IRNUC | 1H |
| CTEMP | 20.2 c |
| SLVNT | CDCL3 |
| EXREF | 0.00 ppm |
| BF | 0.12 Hz |
| RGAIN | 25 |

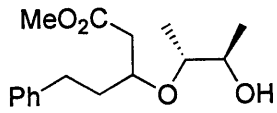



**5a**

5b

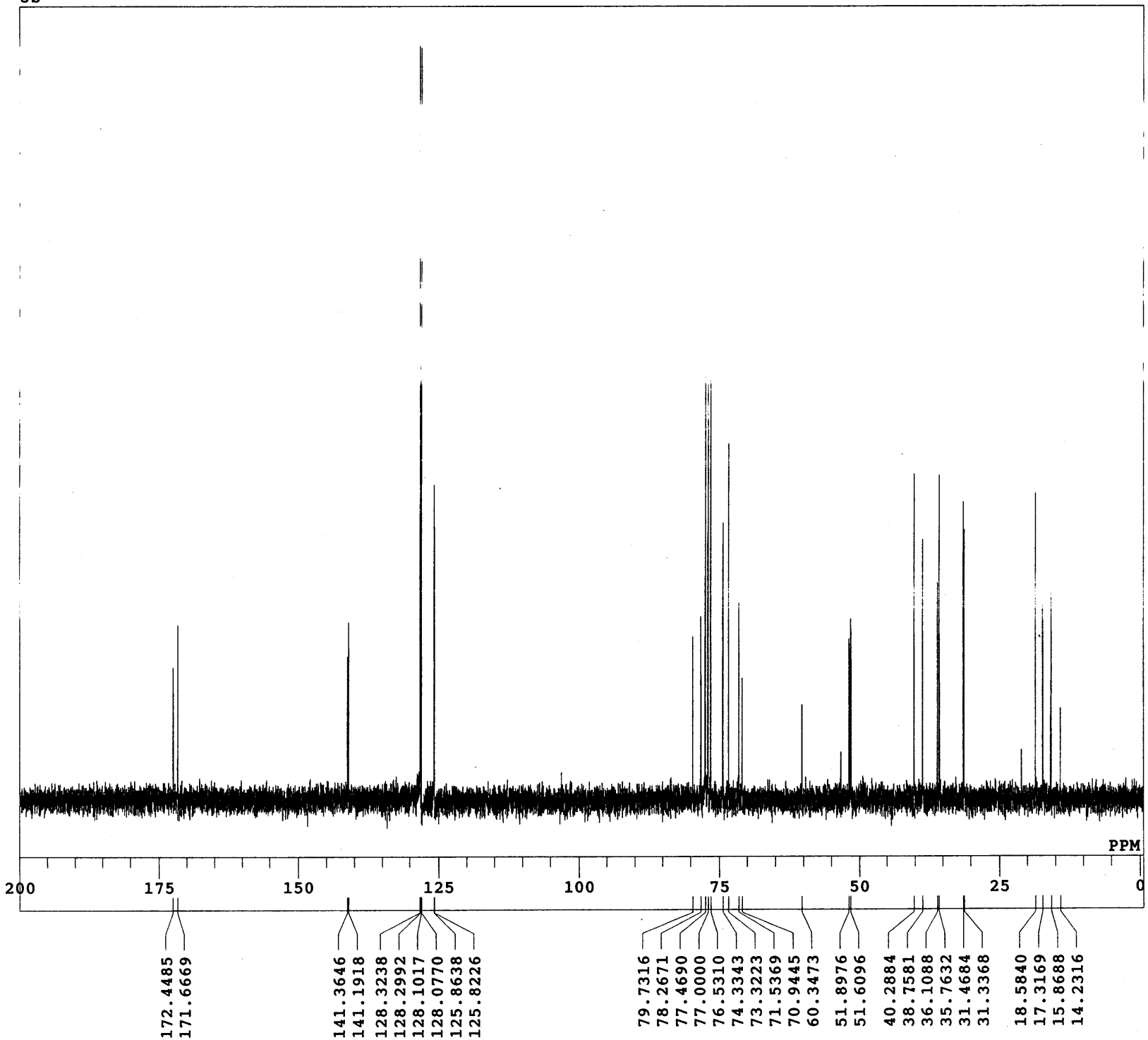



| | |
|---|---|
| DFILE | E:\030404-1.als |
| COMNT | 5b |
| DATIM | Tue Dec 28 10:19:47 2004 |
| OBNUC | 1H |
| EXMOD | NON |
| OBFRQ | 300.40 MHz |
| OBSET | 131.10 KHz |
| OBFIN | 31.00 Hz |
| POINT | 32768 |
| [illegible] | [illegible] |
| [illegible]COTM | 4.0960 sec |
| [illegible]D | 2.0000 sec |
| PW1 | 4.30 usec |
| IRNUC | 1H |
| CTEMP | 22.9 c |
| SLVNT | CDCL3 |
| EXREF | 7.24 ppm |
| RF | 0.12 Hz |
| RGAIN | 13 |

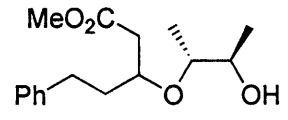



**5b**

5b

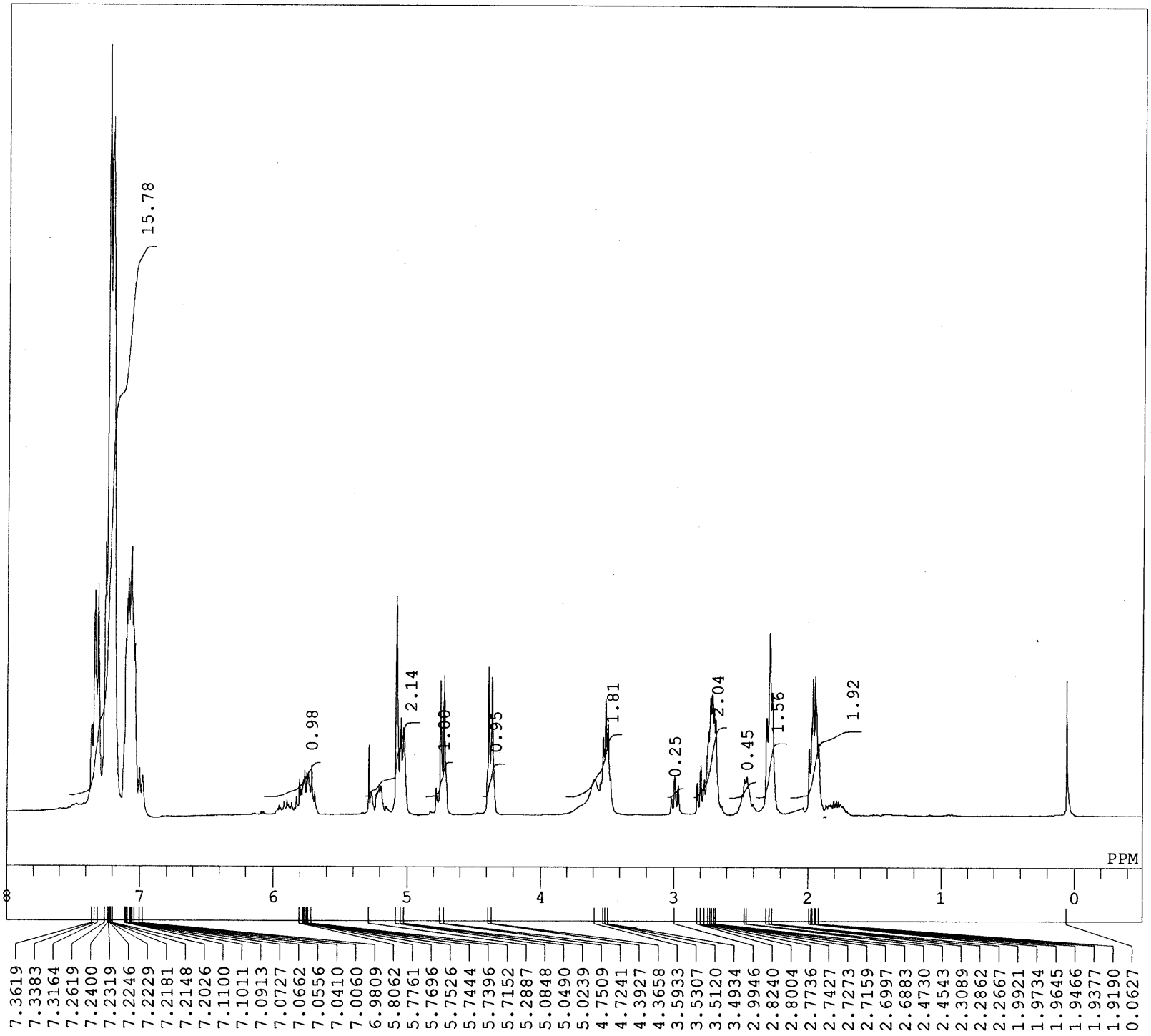



DFILE E:\030404-2.als
COMNT 5b
DATIM Tue Dec 28 11:00:29 2004
OBNUC 13C
EXMOD BCM
OBFRQ 67.80 MHz
OBSET 135.00 KHz
OBFIN 5200.00 Hz
IRNUC 1H
CTEMP 18.8 c
SLVNT CDCL3
RGAIN 26

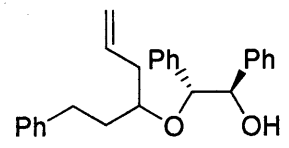



**5b**

5c

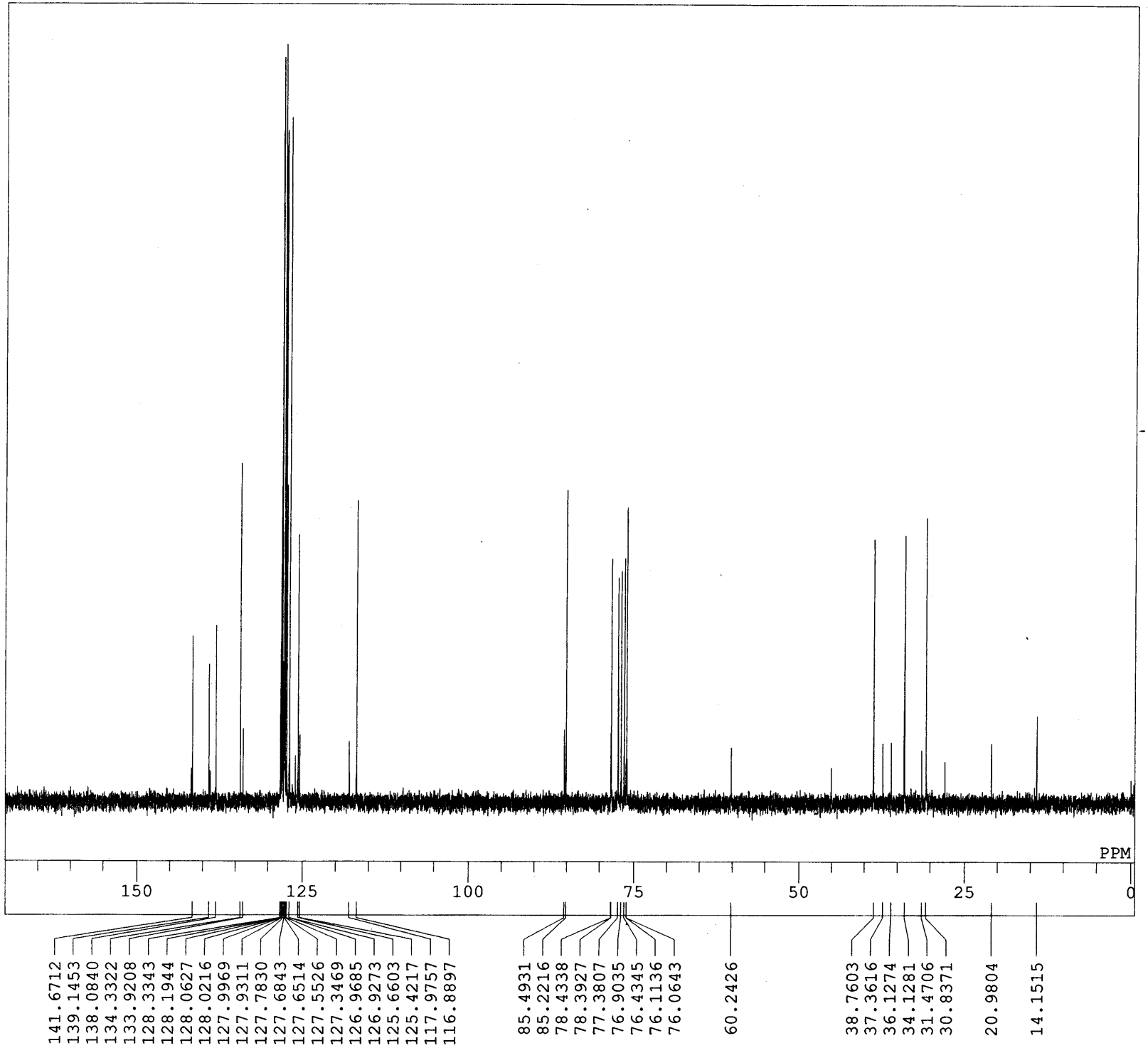



| | |
|---|---|
| DFILE | E:\030375-1.als |
| COMNT | 5c |
| DATIM | Thu Dec 09 13:09:12 2004 |
| OBNUC | 1H |
| EXMOD | NON |
| OBFRQ | 300.40 MHz |
| OBSET | 131.10 KHz |
| OBFIN | 31.00 Hz |
| POINT | 32768 |
| FREQU | 8000.00 Hz |
| SCANS | 16 |
| ACQTM | 4.0960 sec |
| PD | 2.0000 sec |
| PW1 | 4.30 usec |
| IRNUC | 1H |
| CTEMP | 22.3 c |
| SLVNT | CDCL3 |
| EXREF | 7.24 ppm |
| BF | 0.12 Hz |
| RGAIN | 11 |

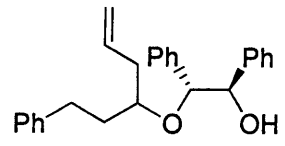



**5c**

5c

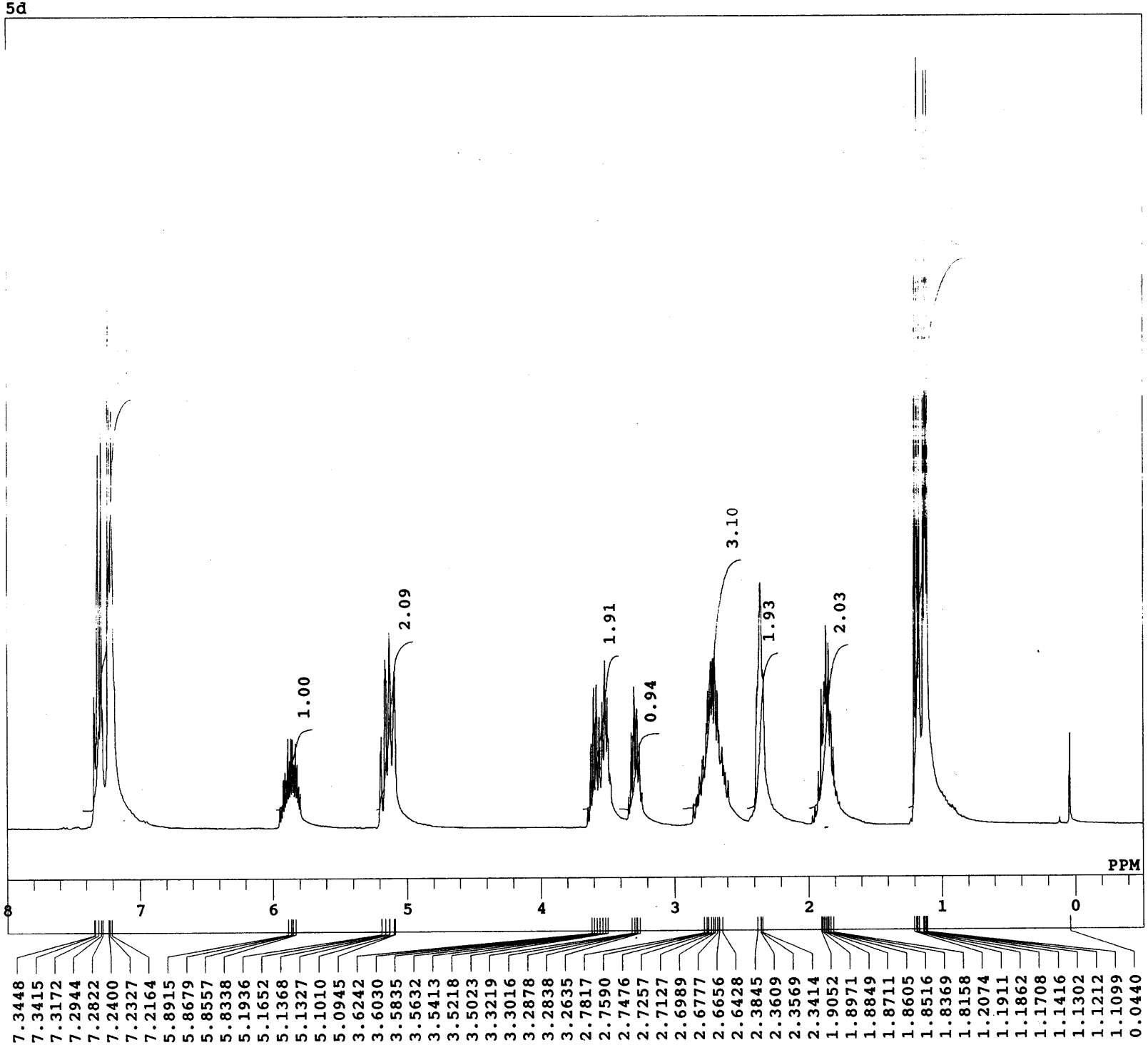



| | |
|---|---|
| DFILE | E:\030375-2.als |
| COMNT | 5c |
| DATIM | Thu Dec 09 13:33:32 2004 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 209 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.10 usec |
| IRNUC | 1H |
| CTEMP | 21.1 c |
| SLVNT | CDCL3 |
| EXREF | 0.00 ppm |
| BF | 0.12 Hz |
| RGAIN | 25 |

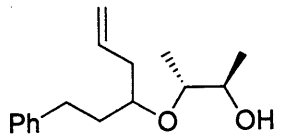



**5c**

5d

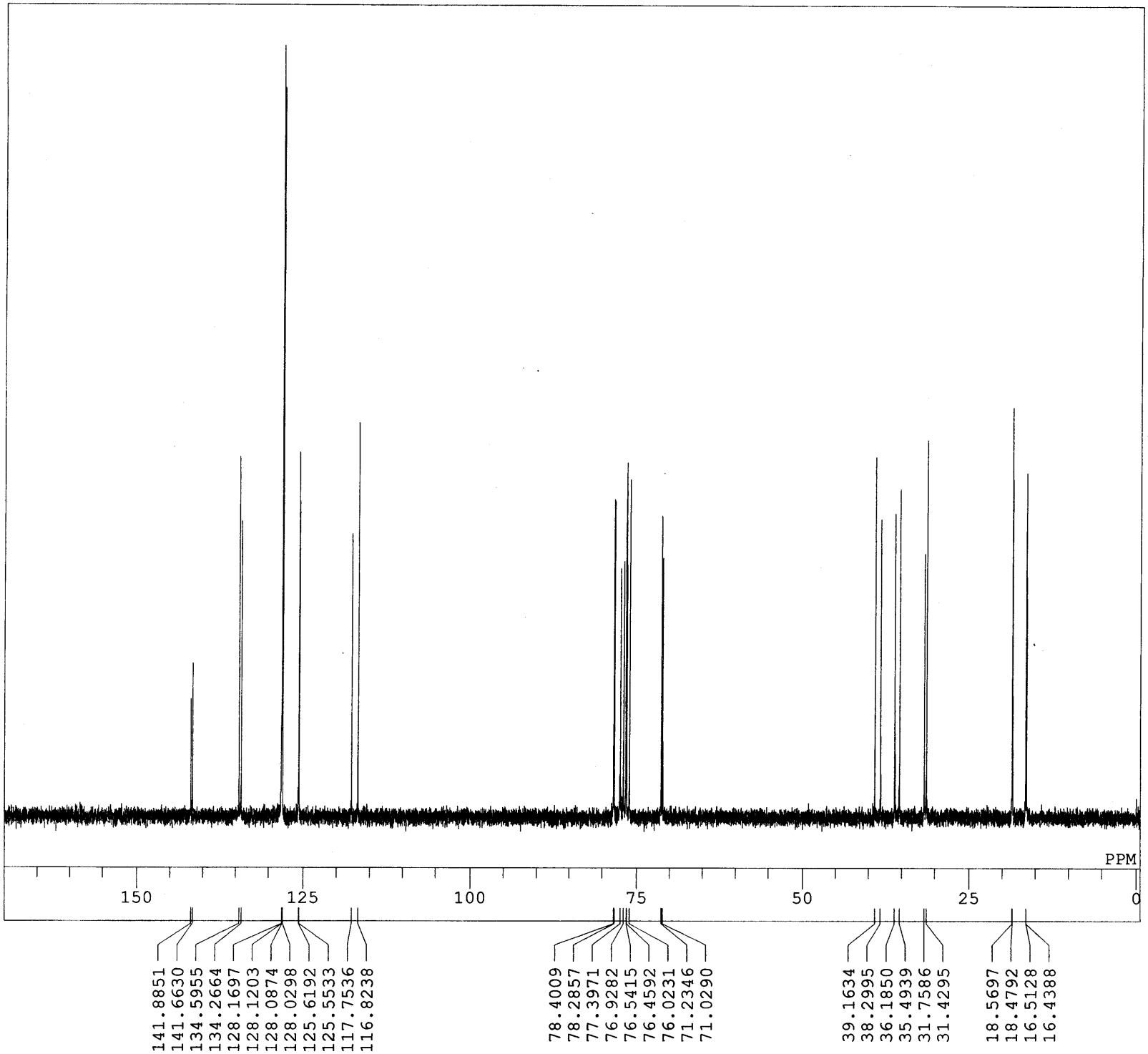



DFILE E:\030397-1.als
COMNT 5d
DATIM Fri Dec 24 18:37:07 2004
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 131.10 KHz
[illegible]
[illegible]
[illegible]
[illegible]
D1 2.0000 sec
IW1 4.30 usec
IRNUC 1H
CTEMP 22.5 c
SLVNT CDCL3
EXREF 7.24 ppm
[illegible]
RGAIN 12

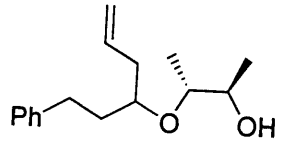



**5d**

030397-1

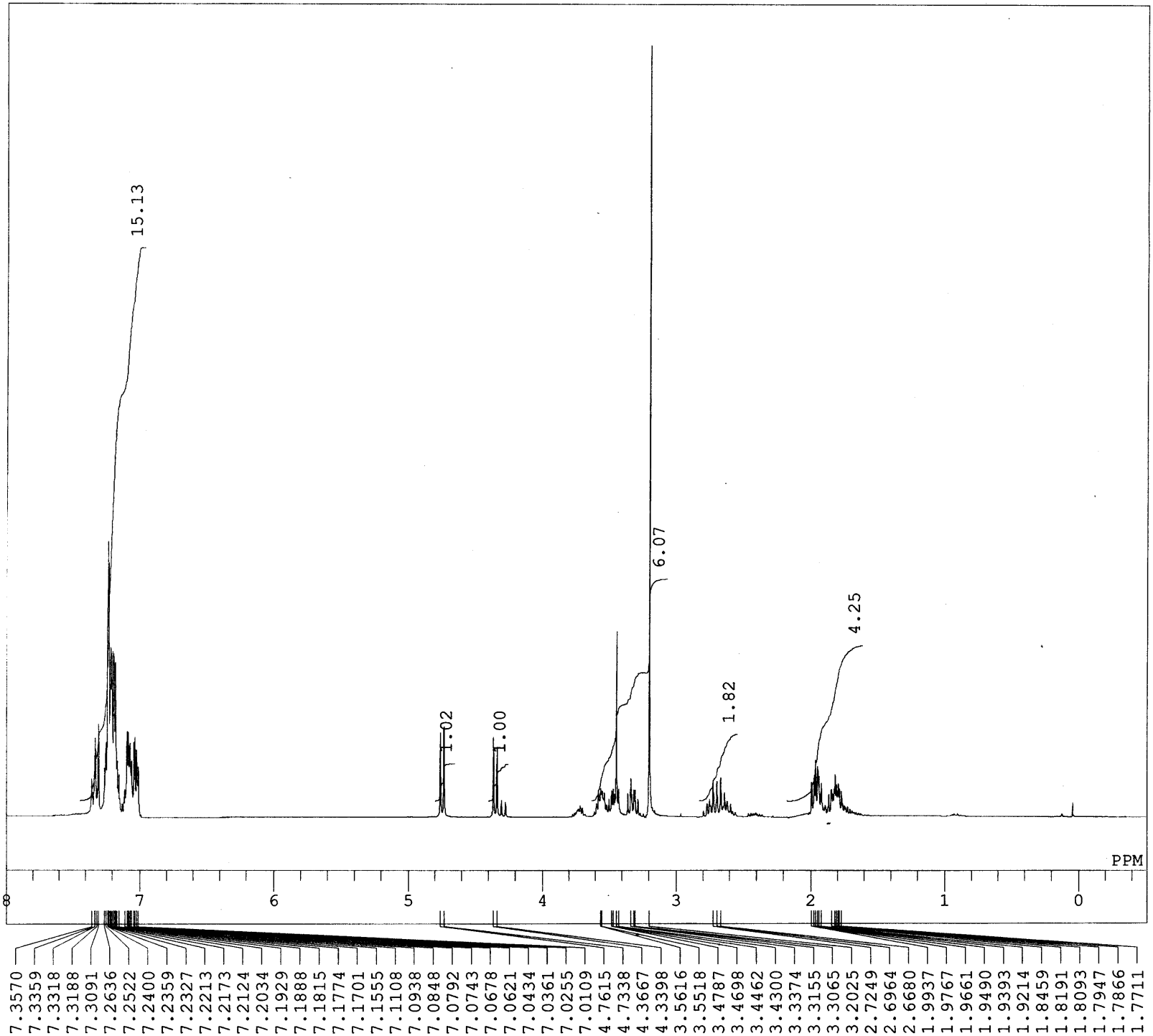



| | |
|---|---|
| DFILE | E:\030397-2.als |
| COMNT | 030397-1 |
| DATIM | Fri Dec 24 19:09:44 2004 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 425 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.10 usec |
| IRNUC | 1H |
| CTEMP | 19.8 c |
| SLVNT | CDCL3 |
| EXREF | 0.00 ppm |
| BF | 0.12 Hz |
| RGAIN | 26 |

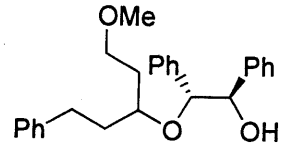



**5d**

5e

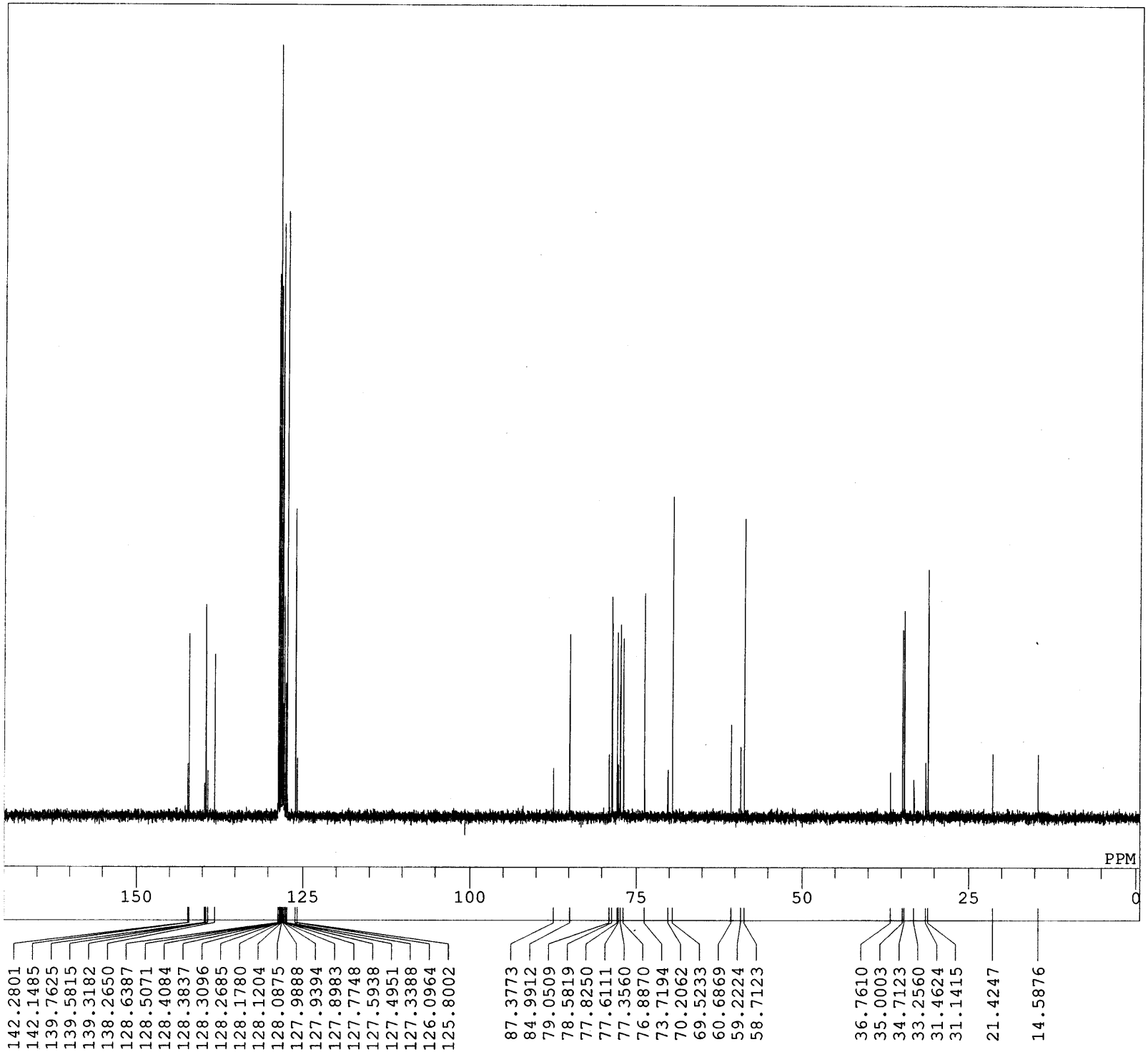



| | |
|---|---|
| DFILE | E:\030408-1.als |
| COMNT | 5e |
| DATIM | Sat Jan 08 09:39:55 2005 |
| OBNUC | 1H |
| EXMOD | NON |
| OBFRQ | 300.40 MHz |
| OBSET | 131.10 KHz |
| OBFIN | 31.00 Hz |
| POINT | 32768 |
| FREQU | 8000.00 Hz |
| SCANS | 16 |
| ACQTM | 4.0960 sec |
| PD | 2.0000 sec |
| PW1 | 4.30 usec |
| IRNUC | 1H |
| CTEMP | 22.1 c |
| SLVNT | CDCL3 |
| EXREF | 7.24 ppm |
| BF | 0.12 Hz |
| RGAIN | 11 |

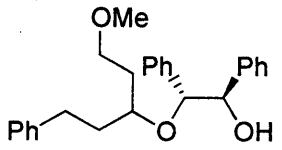



**5e**

5e

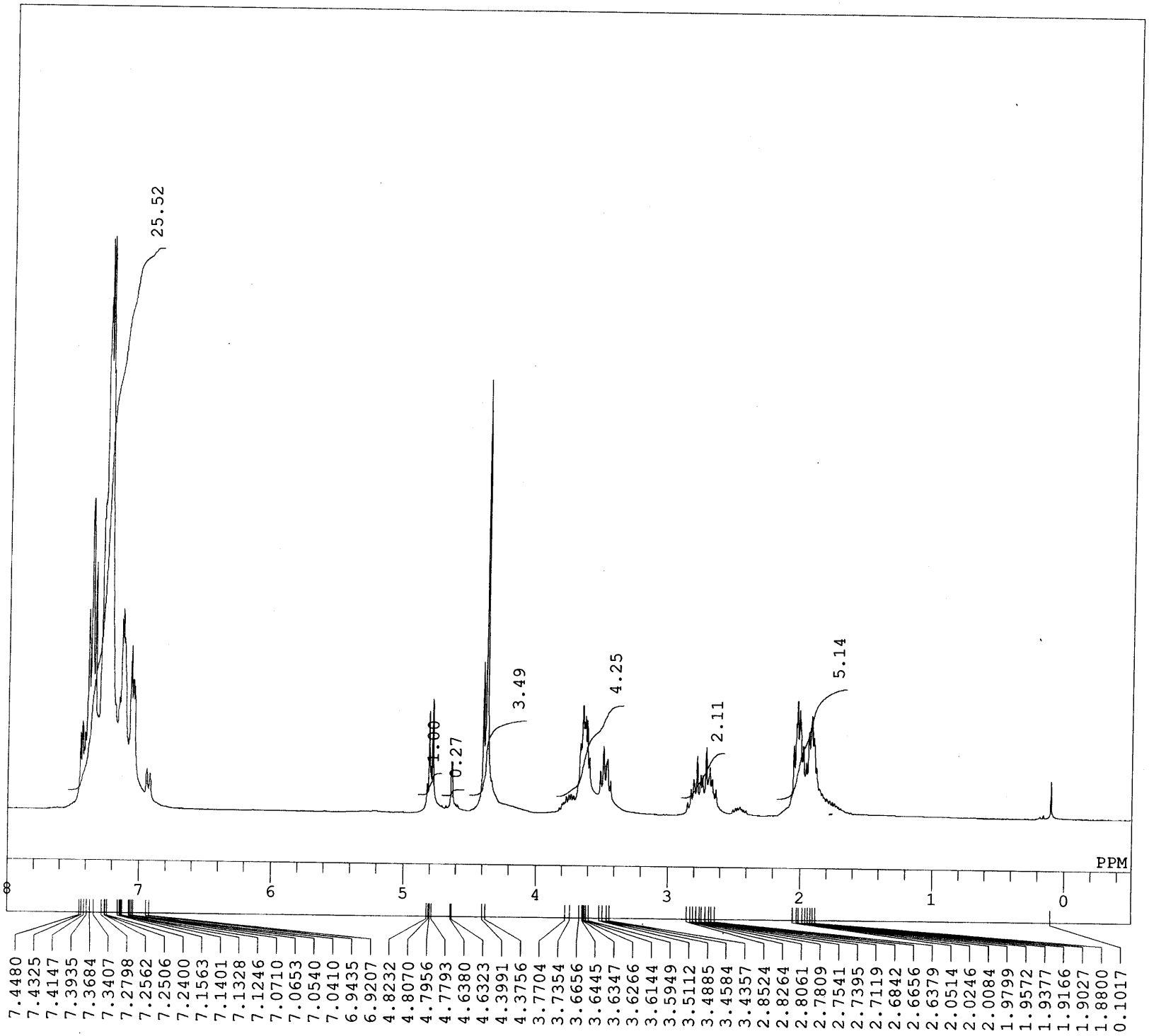



| | |
|---|---|
| DFILE | E:\030408-2.als |
| COMNT | 5e |
| DATIM | Sat Jan 08 10:09:11 2005 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 225 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.00 usec |
| IRNUC | 1H |
| CTEMP | 20.1 c |
| SLVNT | CDCL3 |
| EXREF | 0.00 ppm |
| BF | 0.12 Hz |
| RGAIN | 26 |

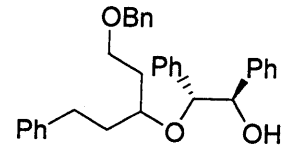



**5e**

5f

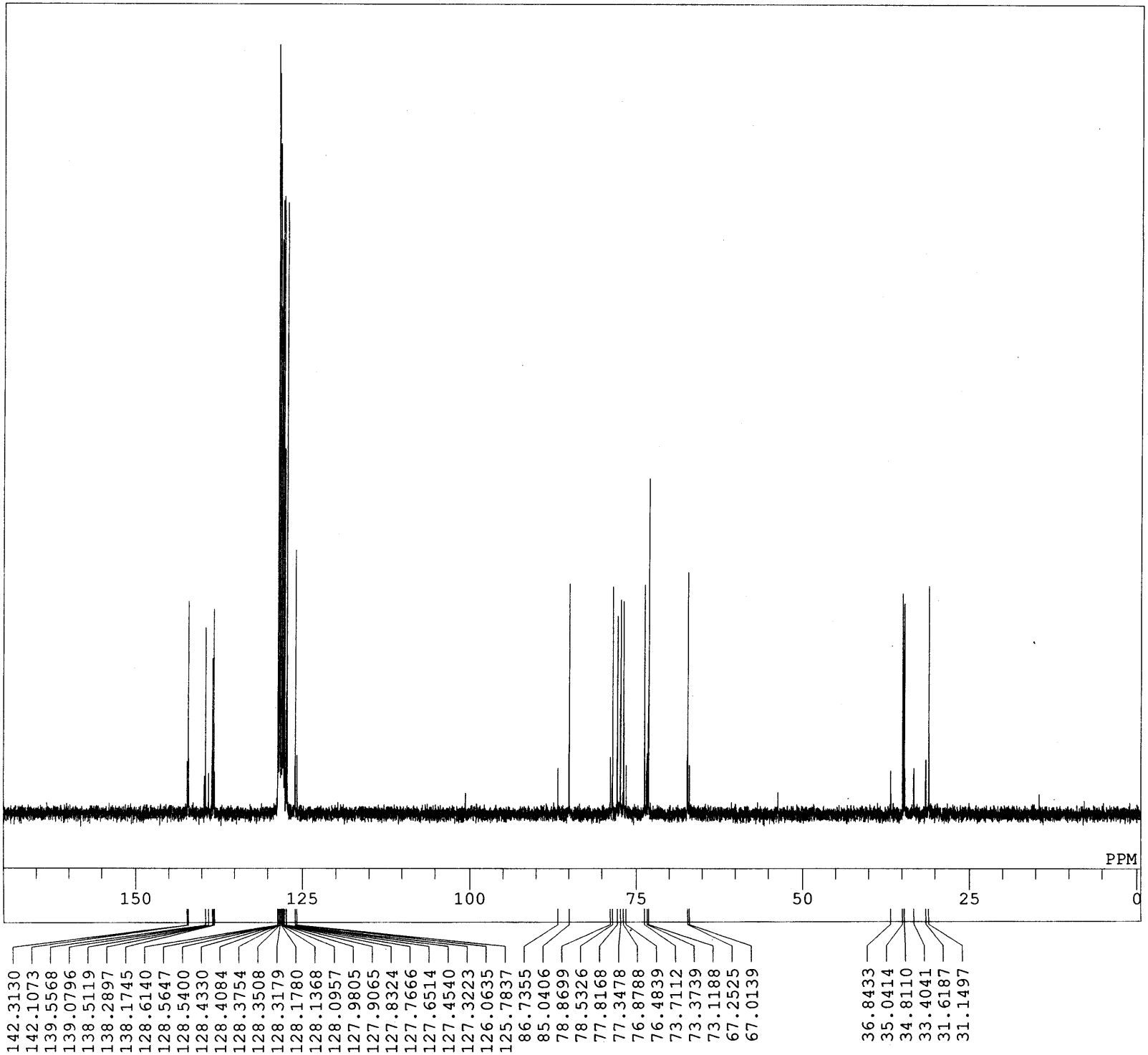



| | |
|---|---|
| DFILE | E:\030391-1.als |
| COMNT | 5f |
| DATIM | Tue Dec 21 19:05:16 2004 |
| OBNUC | 1H |
| EXMOD | NON |
| OBFRQ | 300.40 MHz |
| OBSET | 131.10 KHz |
| OBFIN | 31.00 Hz |
| POINT | 32768 |
| FREQU | 8000.00 Hz |
| SCANS | 16 |
| ACQTM | 4.0960 sec |
| PD | 2.0000 sec |
| PW1 | 4.30 usec |
| IRNUC | 1H |
| CTEMP | 22.5 c |
| SLVNT | CDCL3 |
| EXREF | 7.24 ppm |
| BF | 0.12 Hz |
| RGAIN | 10 |

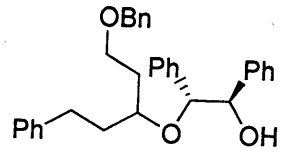



**5f**

5f

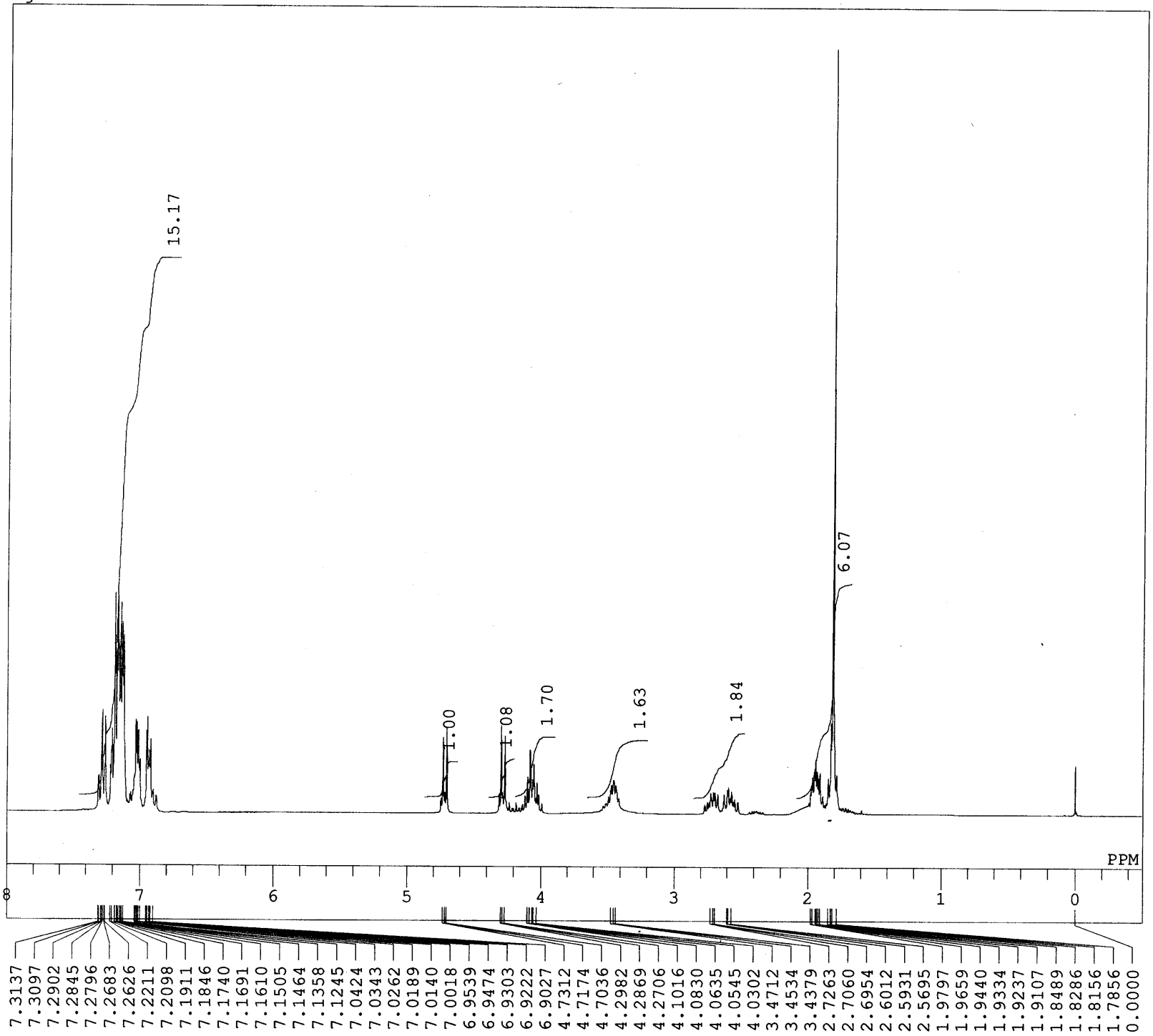



DFILE E:\030391-2.als
COMNT 5f
DATIM Tue Dec 21 19:39:27 2004
OBNUC 13C
EXMOD BCM
OBFRQ 67.80 MHz
OBSET 135.00 KHz
OBFIN 5200.00 Hz
POINT 32768
FREQU 18315.00 Hz
SCANS 345
ACQTM 1.7891 sec
PD 1.2110 sec
PW1 4.10 usec
IRNUC 1H
CTEMP 19.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 25

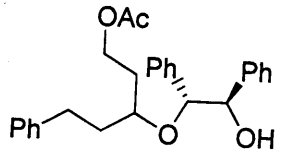



**5f**

5g

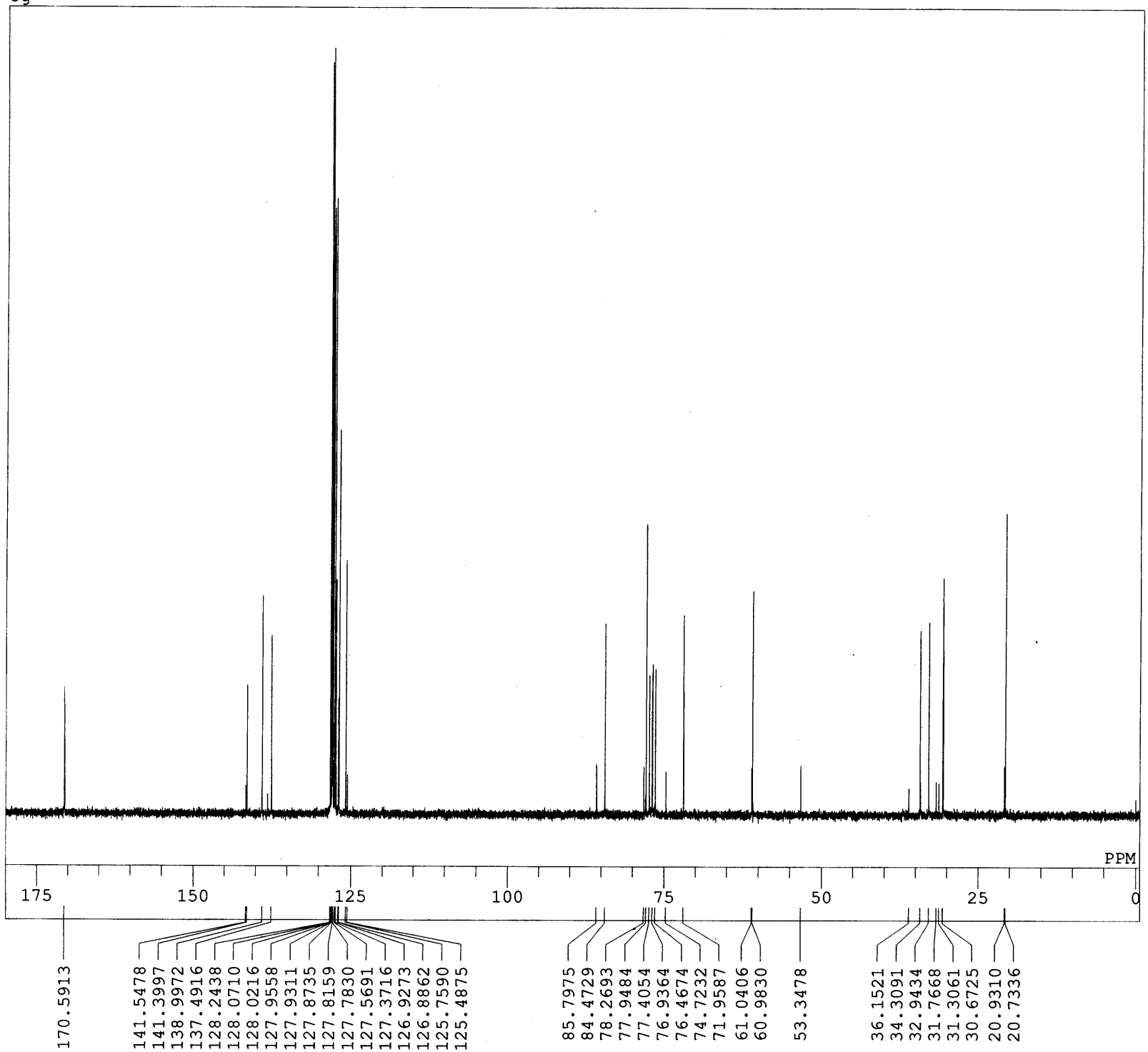



| | |
|---|---|
| DFILE | E:\030386-1.als |
| COMNT | 5g |
| DATIM | Mon Dec 20 09:52:07 2004 |
| OBNUC | 1H |
| EXMOD | NON |
| OBFRQ | 300.40 MHz |
| OBSET | 131.10 KHz |
| OBFIN | 31.00 Hz |
| POINT | 32768 |
| FREQU | 8000.00 Hz |
| SCANS | 16 |
| ACQTM | 4.0960 sec |
| PD | 2.0000 sec |
| PW1 | 4.30 usec |
| IRNUC | 1H |
| CTEMP | 22.7 c |
| SLVNT | CDCL3 |
| EXREF | 0.00 ppm |
| BF | 0.12 Hz |
| RGAIN | 11 |

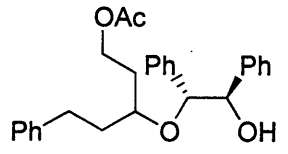



**5g**

5g

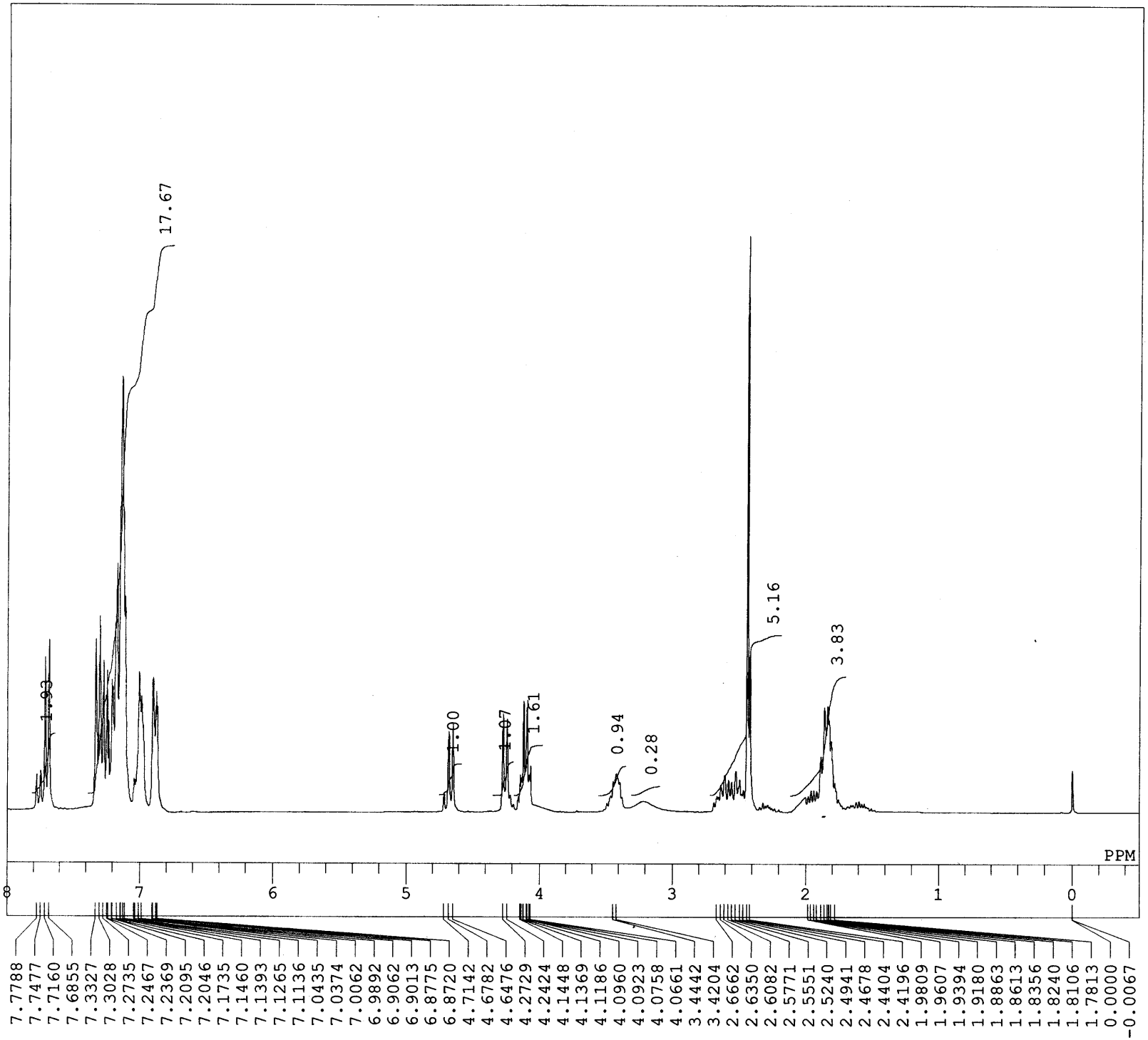



| | |
|---|---|
| DFILE | E:\030386-2.als |
| COMNT | 5g |
| DATIM | Mon Dec 20 10:20:16 2004 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 361 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.10 usec |
| IRNUC | 1H |
| CTEMP | 20.6 c |
| SLVNT | CDCL3 |
| EXREF | 0.00 ppm |
| BF | 0.12 Hz |
| RGAIN | 26 |

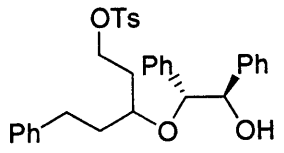



**5g**

5h

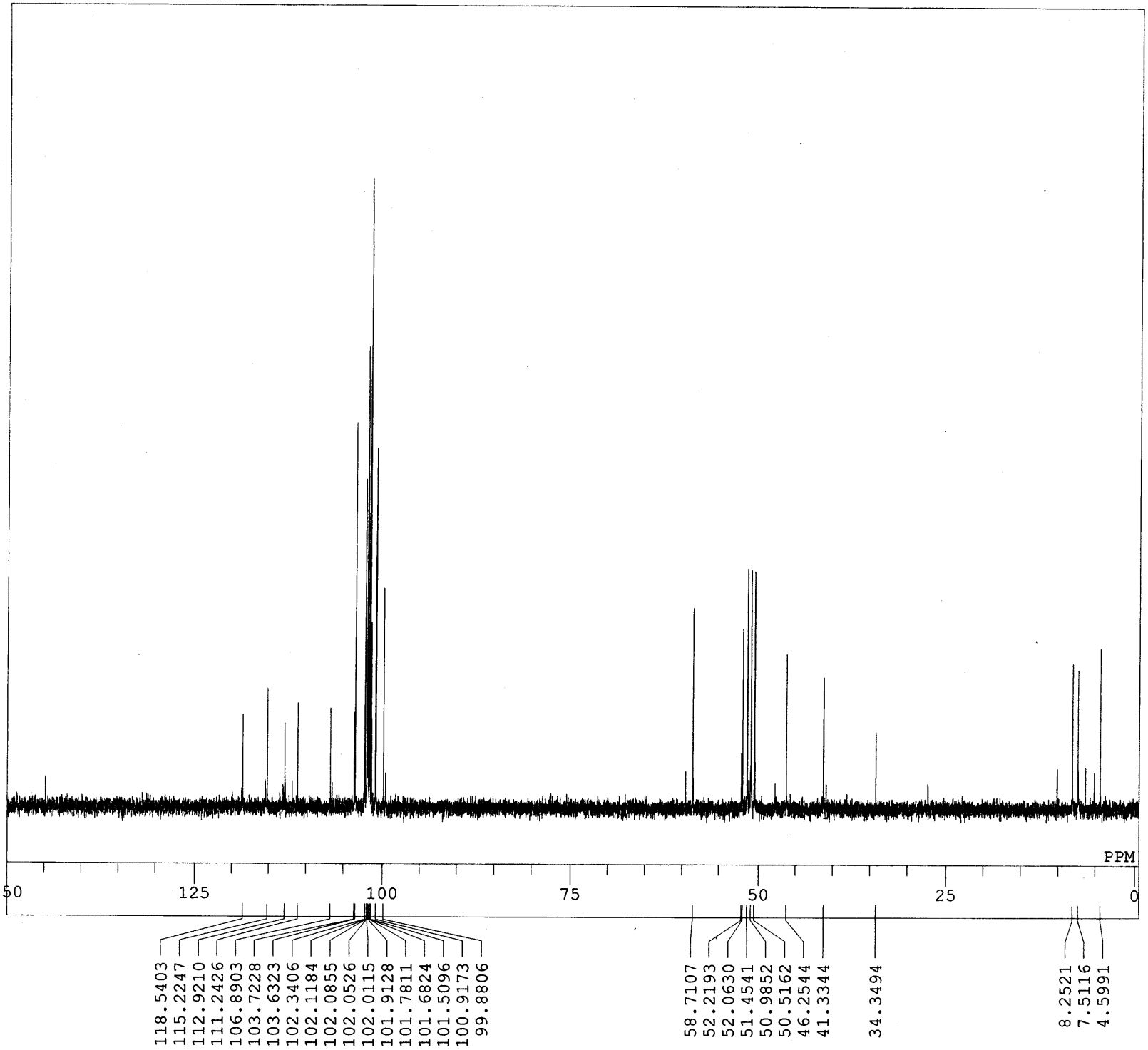



| | |
|---|---|
| DFILE | E:\030421-1.als |
| COMNT | 5h |
| DATIM | Tue Jan 18 17:40:55 2005 |
| OBNUC | 1H |
| EXMOD | NON |
| OBFRQ | 270.05 MHz |
| OBSET | 112.00 KHz |
| OBFIN | 5800.00 Hz |
| POINT | 32768 |
| FREQU | 5402.40 Hz |
| SCANS | 16 |
| ACQTM | 6.0655 sec |
| PD | 0.9350 sec |
| PW1 | 5.50 usec |
| IRNUC | 1H |
| CTEMP | 18.4 c |
| SLVNT | CDCL3 |
| EXREF | 0.00 ppm |
| BF | 0.12 Hz |
| RGAIN | 14 |

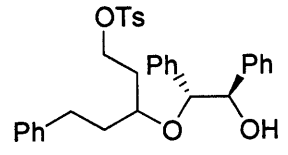



**5h**

5f

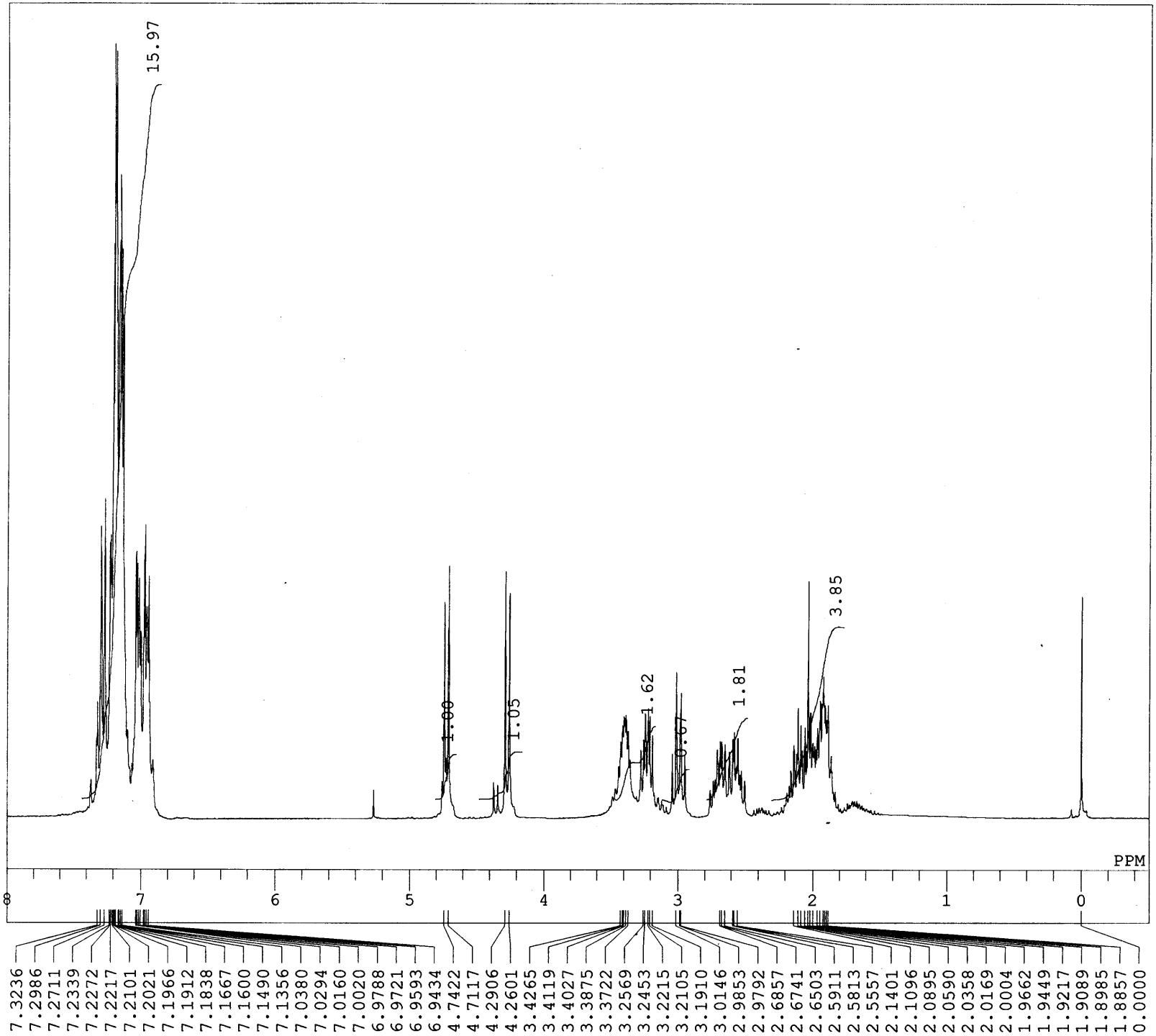



| | |
|---|---|
| DFILE | E:\030421-2.als |
| COMNT | 5f |
| DATIM | Tue Jan 18 18:10:19 2005 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 442 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.00 usec |
| IRNUC | 1H |
| CTEMP | 18.9 c |
| SLVNT | CDCL3 |
| EXREF | 0.00 ppm |
| BF | 0.12 Hz |
| RGAIN | 26 |

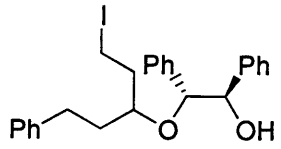



**5h**

5i

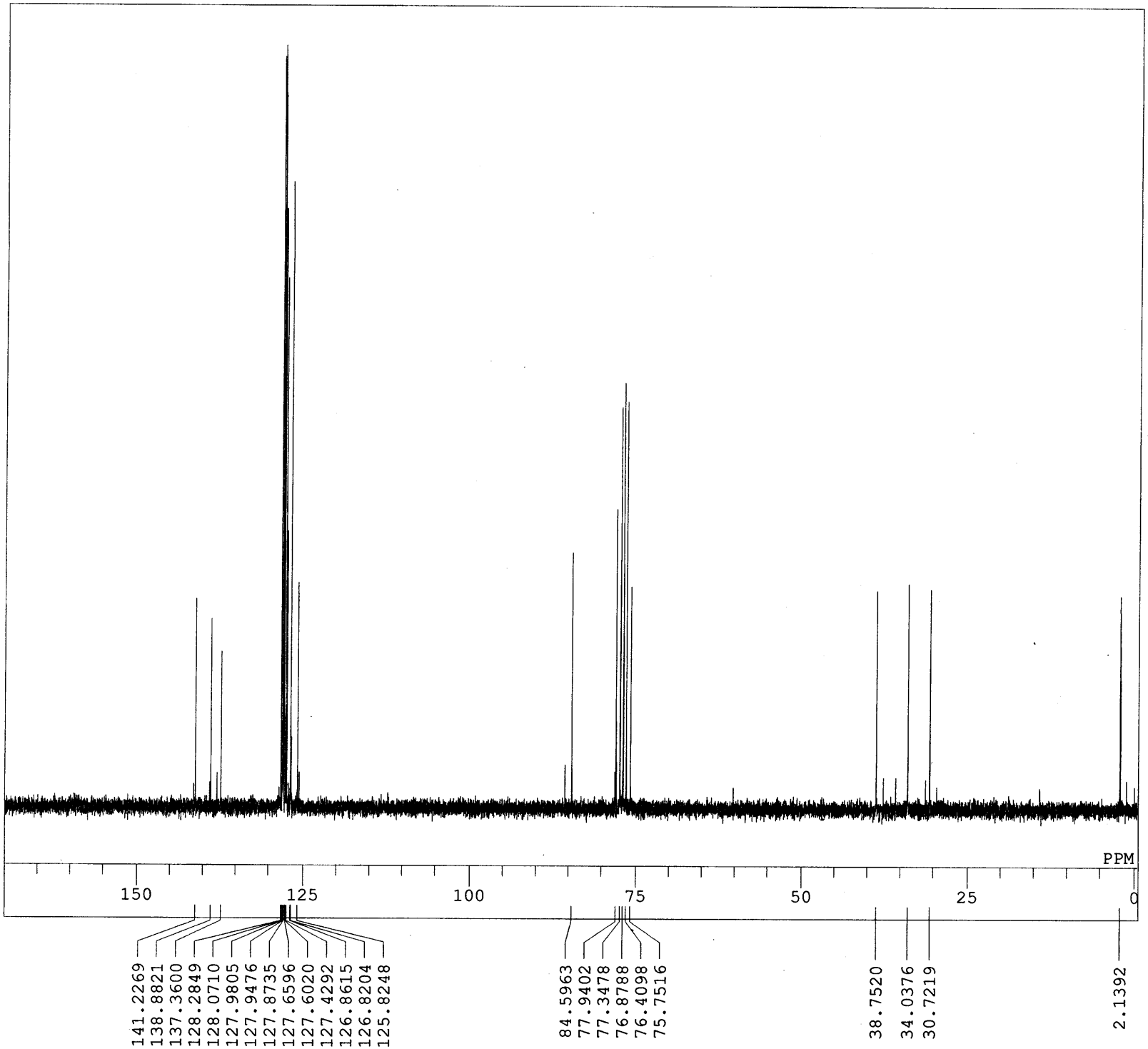



DFILE E:\030402-1.als
COMNT 5i
DATIM Mon Dec 27 07:32:18 2004
OBNUC 1H
EXMOD NON
OBFRQ 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.00 Hz
POINT 32768
FREQU 5402.40 Hz
SCANS 16
ACQTM 6.0655 sec
PD 0.9350 sec
PW1 5.90 usec
IRNUC 1H
CTEMP 19.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 15

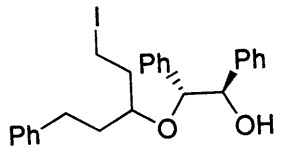



**5i**

5i

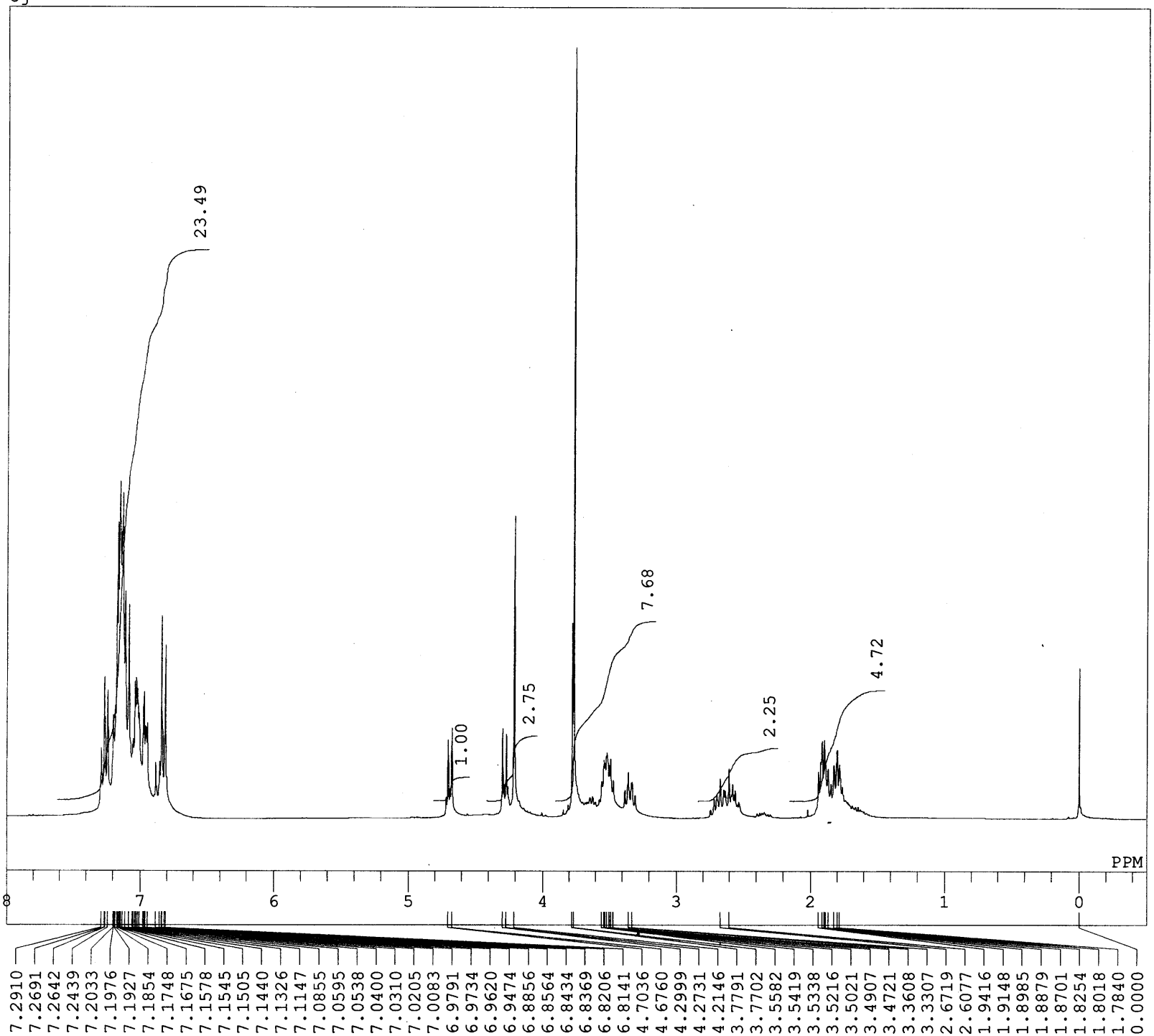



| | |
|---|---|
| DFILE | E:\030402-3.als |
| COMNT | 5i |
| DATIM | Mon Dec 27 08:12:29 2004 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 449 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.10 usec |
| IRNUC | 1H |
| CTEMP | 19.0 c |
| SLVNT | CDCL3 |
| EXREF | 0.00 ppm |
| BF | 0.12 Hz |
| RGAIN | 26 |

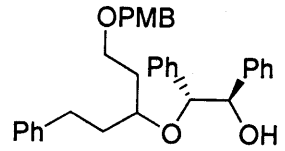



**5i**

5j

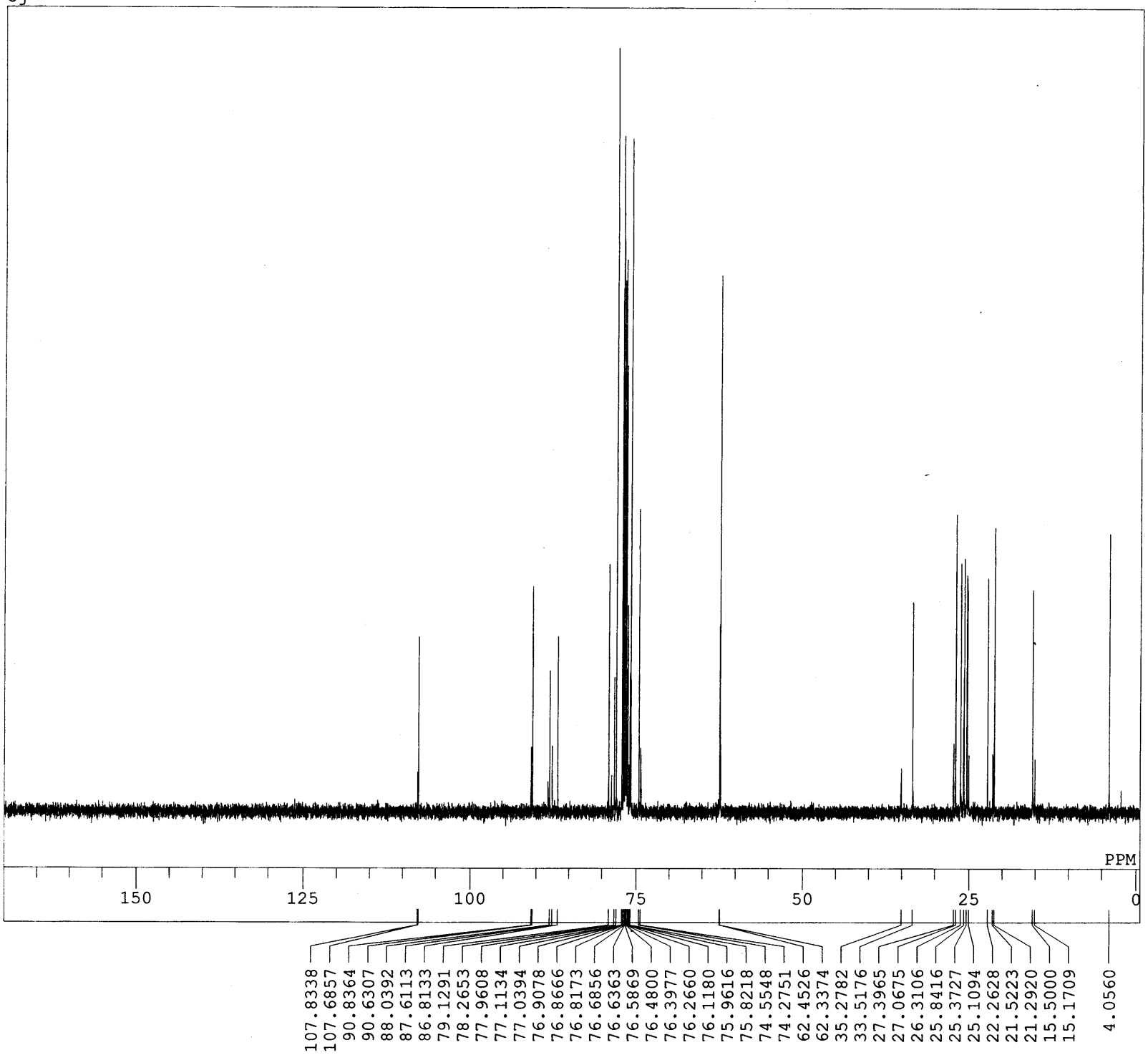



DFILE E:\030390-1.als
COMNT 5j
DATIM Fri Dec 24 11:10:28 2004
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 131.10 KHz
OBFIN 31.00 Hz
POINT 32768
FREQU 8000.00 Hz
SCANS 16
ACQTM 4.0960 sec
PD 2.0000 sec
PW1 4.30 usec
IRNUC 1H
CTEMP 22.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 11

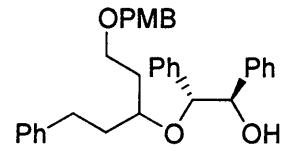



**5j**

5j

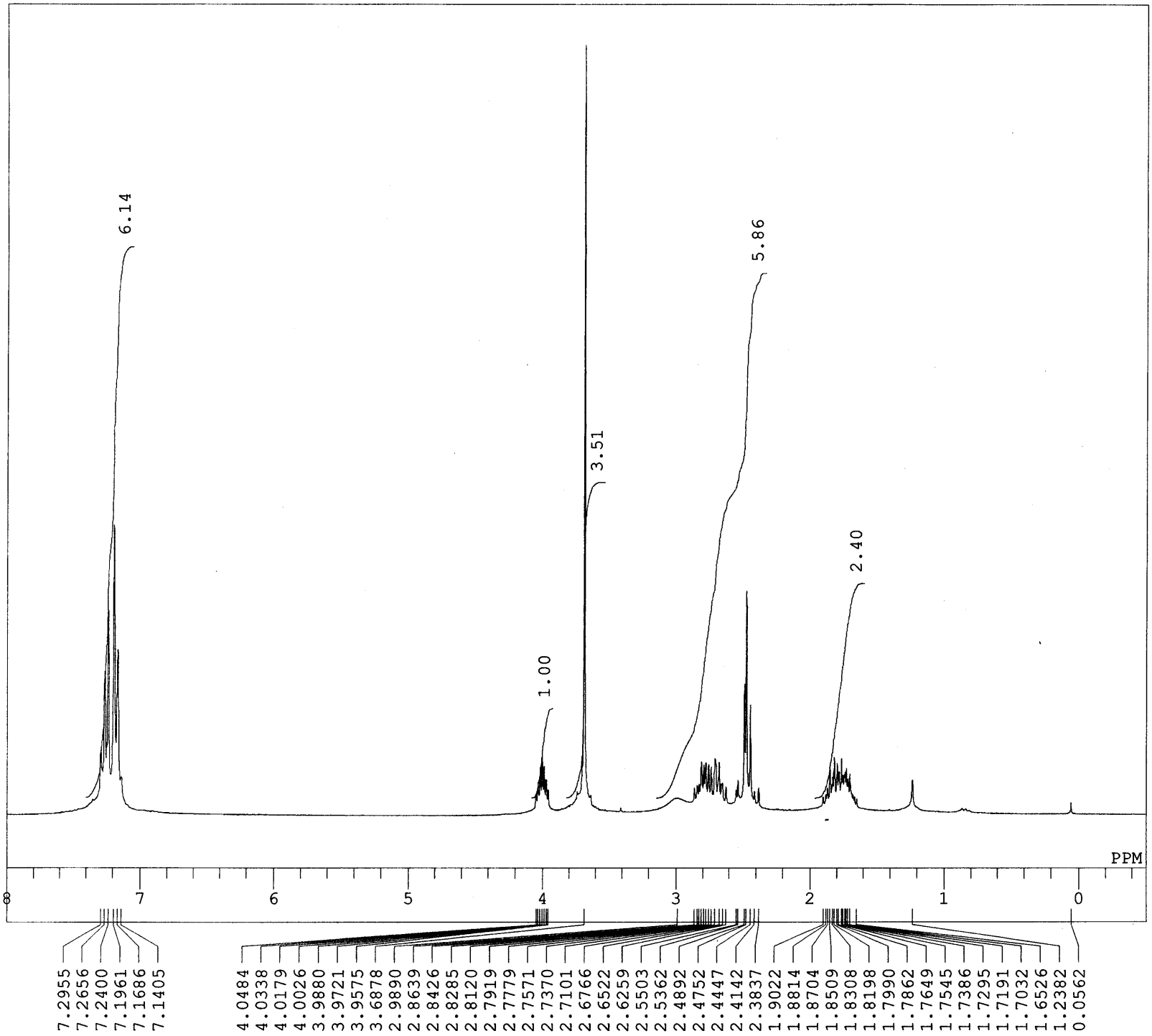



DFILE E:\030390-2.als
COMNT 5j
DATIM Fri Dec 24 11:48:19 2004
OBNUC 13C
EXMOD BCM
OBFRQ 67.80 MHz
OBSET 135.00 KHz
OBFIN 5200.00 Hz
POINT 32768
FREQU 18315.00 Hz
SCANS 321
ACQTM 1.7891 sec
PD 1.2110 sec
PW1 4.10 usec
IRNUC 1H
CTEMP 19.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 26

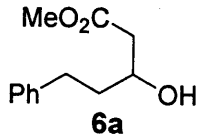



**5j**

6a

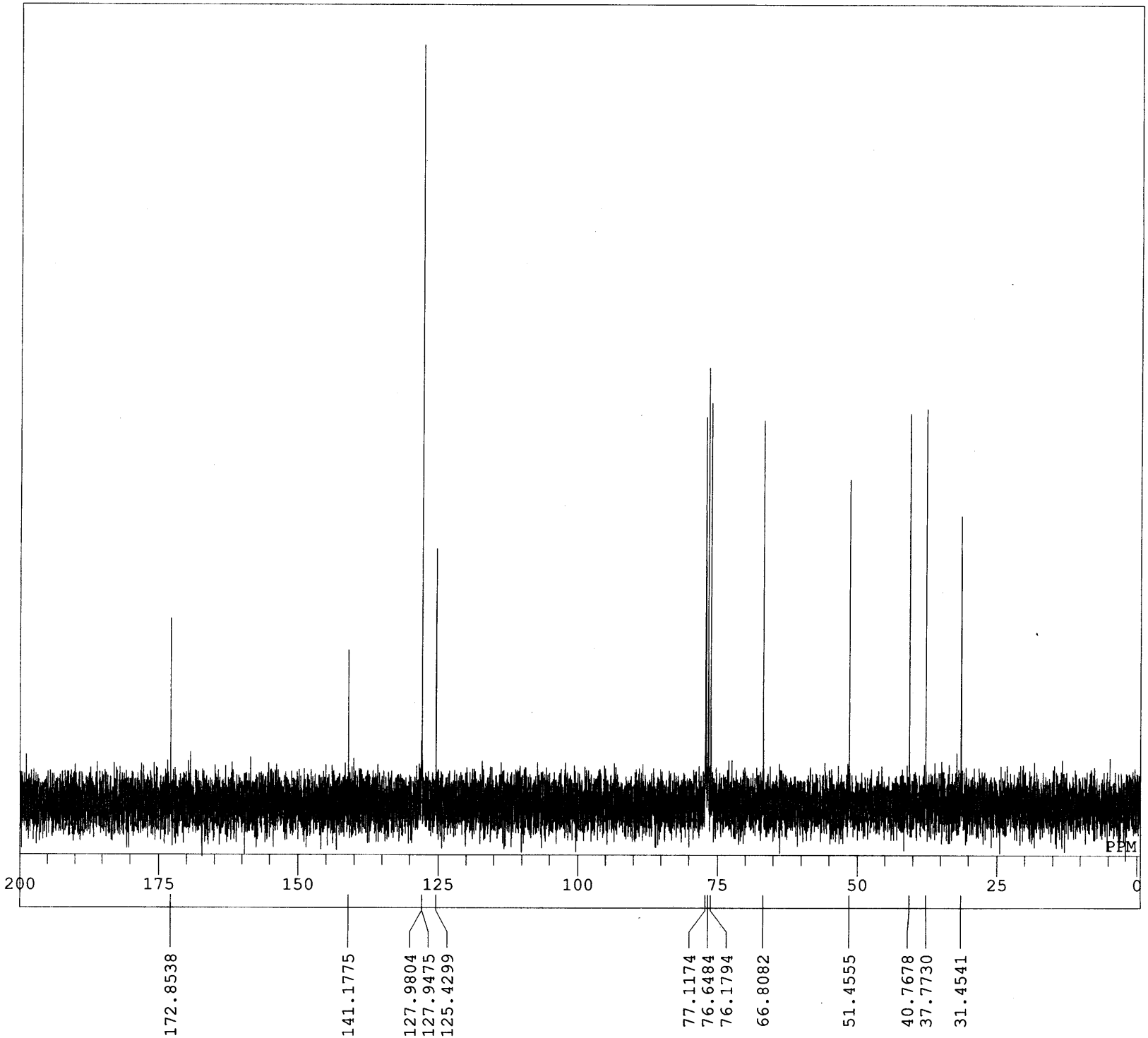



DFILE E:\CAN\CO2Me\H-NMR.als
COMNT 6a
DATIM Fri Dec 17 17:01:01 2004
OBNUC 1H
EXMOD NON
OBFRQ 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.00 Hz
POINT 32768
FREQU 5402.40 Hz
SCANS 16
ACQTM 6.0655 sec
PD 0.9350 sec
PW1 5.90 usec
IRNUC 1H
CTEMP 18.9 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.12 Hz
RGAIN 16

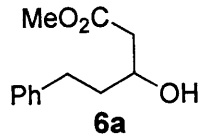



**6a**

6a

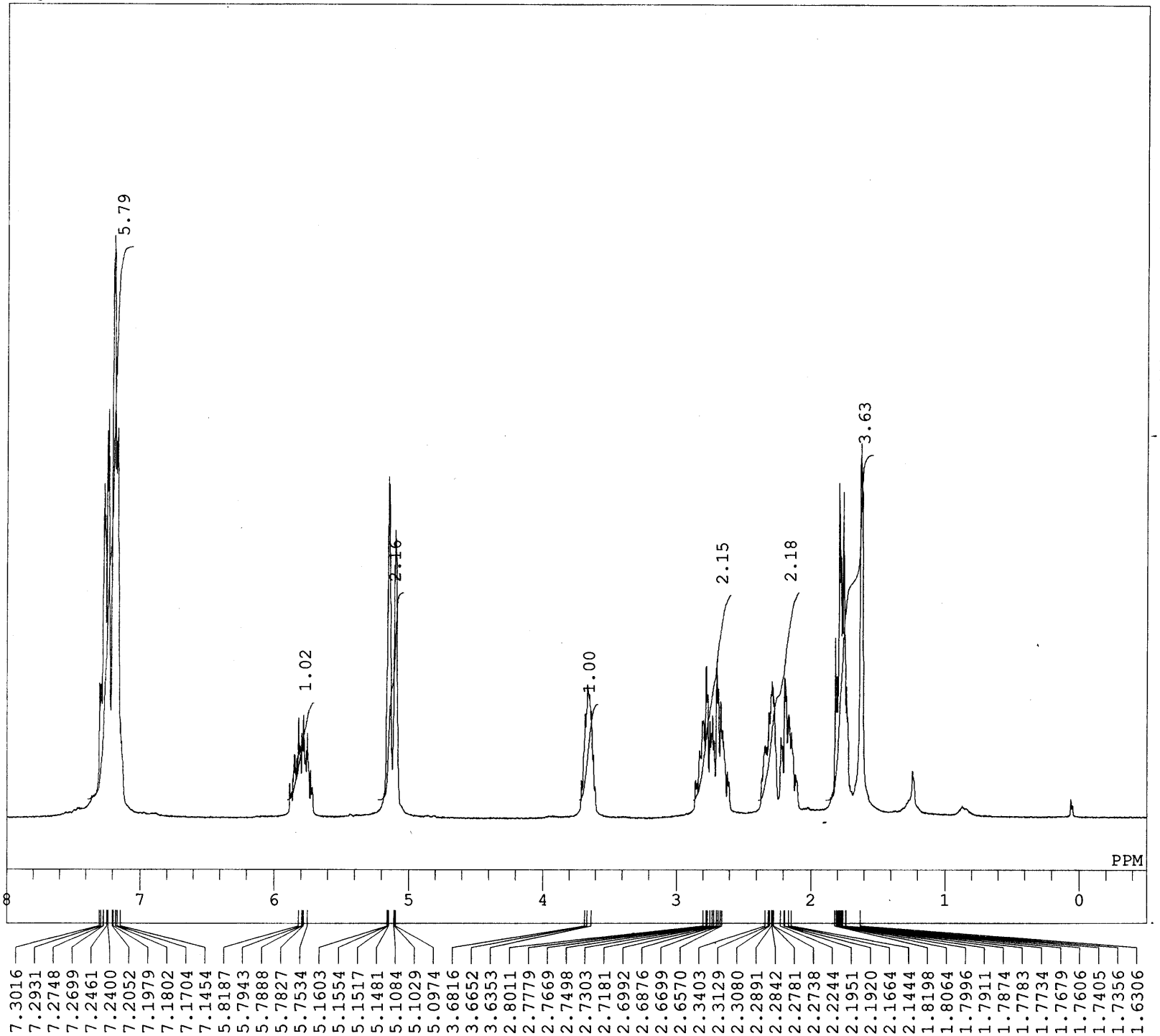



DFILE E:\CAN\CO2Me\C-NMR.als
COMNT 6a
DATIM Fri Dec 17 17:09:38 2004
OBNUC 13C
EXMOD BCM
OBFRQ 67.80 MHz
OBSET 135.00 KHz
OBFIN 5200.00 Hz
POINT 32768
FREQU 18315.00 Hz
SCANS 128
ACQTM 1.7891 sec
PD 1.2110 sec
PW1 4.10 usec
IRNUC 1H
CTEMP 19.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 26

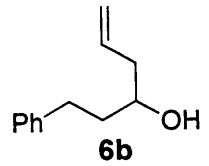



**6a**

6a 6b

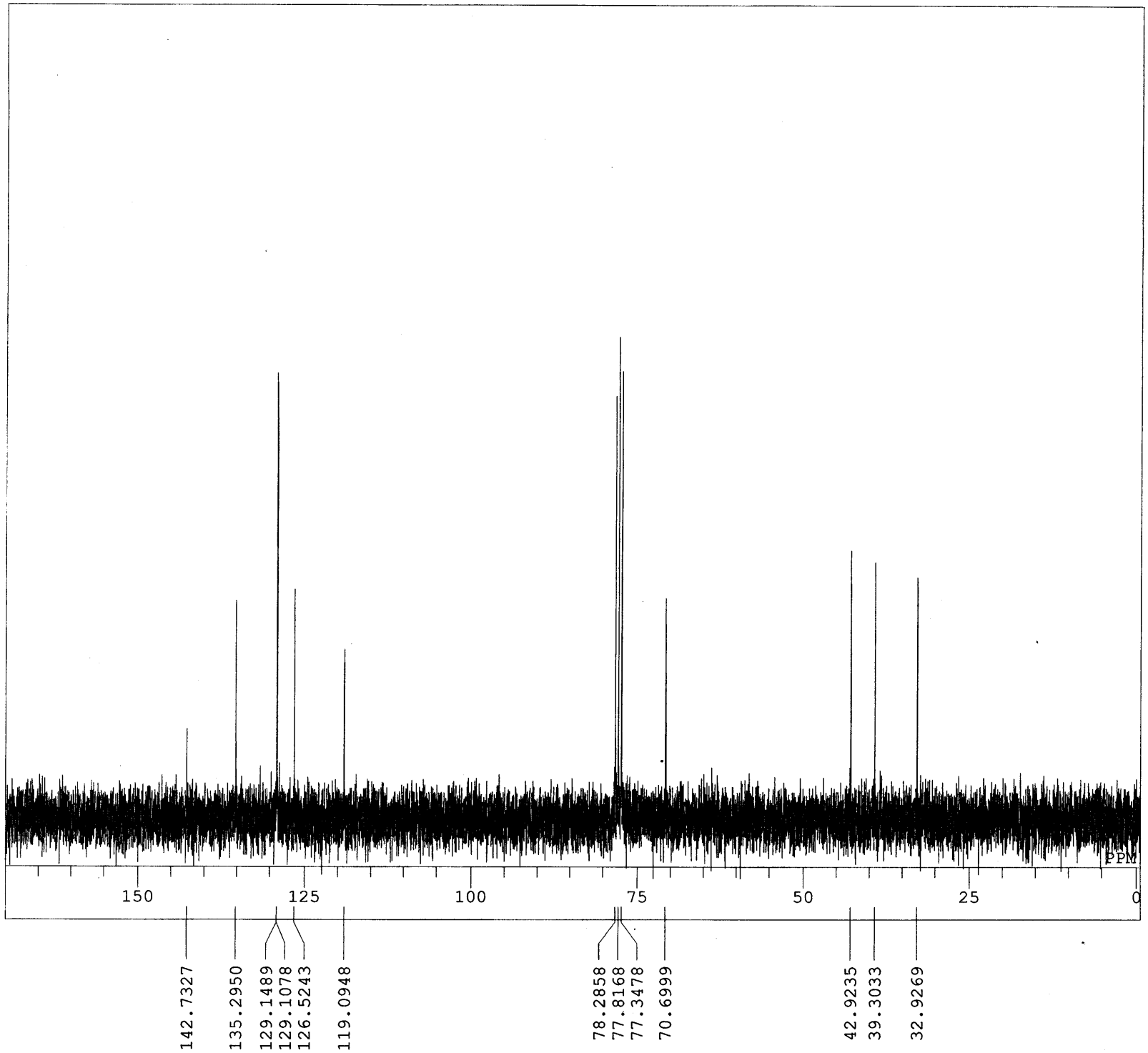



| | |
|---|---|
| DFILE | E:\CAN\Allyl\H-NMR.als |
| COMNT | 6a |
| DATIM | Mon Dec 20 21:25:13 2004 |
| OBNUC | 1H |
| EXMOD | NON |
| OBFRQ | 270.05 MHz |
| OBSET | 112.00 KHz |
| OBFIN | 5800.00 Hz |
| POINT | 32768 |
| FREQU | 5402.40 Hz |
| SCANS | 16 |
| ACQTM | 6.0655 sec |
| PD | 0.9350 sec |
| PW1 | 5.90 usec |
| IRNUC | 1H |
| CTEMP | 19.7 c |
| SLVNT | CDCL3 |
| EXREF | 7.24 ppm |
| BF | 0.12 Hz |
| RGAIN | 16 |

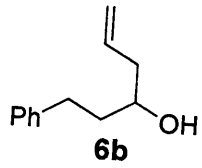



**6b**

6b

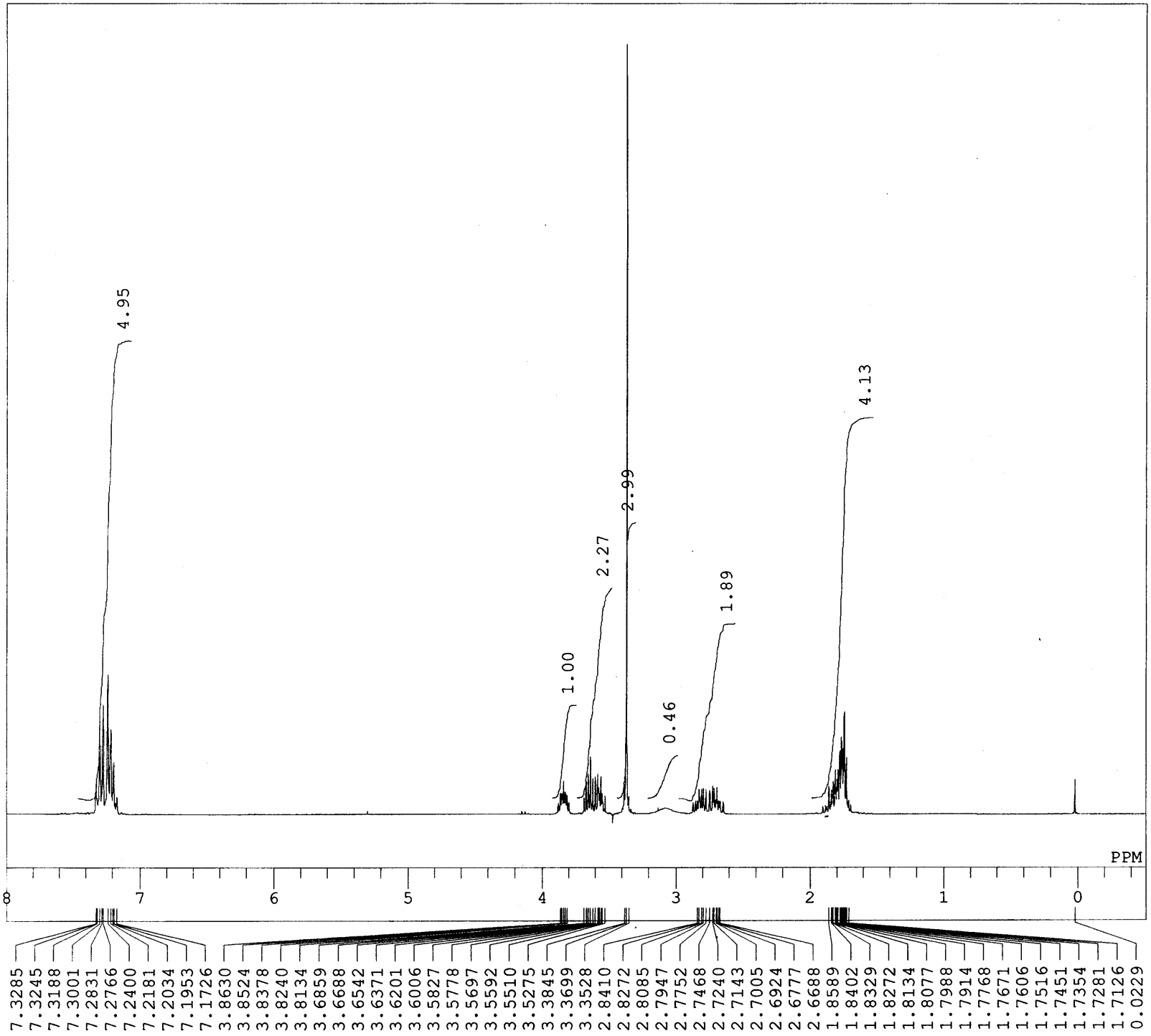



| | |
|---|---|
| DFILE | E:\CAN\Allyl\C-NMR.als |
| COMNT | 6b |
| DATIM | Mon Dec 20 21:22:41 2004 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 128 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.10 usec |
| IRNUC | 1H |
| CTEMP | 20.3 c |
| SLVNT | CDCL3 |
| EXREF | 0.00 ppm |
| BF | 0.12 Hz |
| RGAIN | 26 |

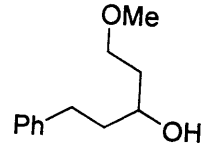



**6b**

6c

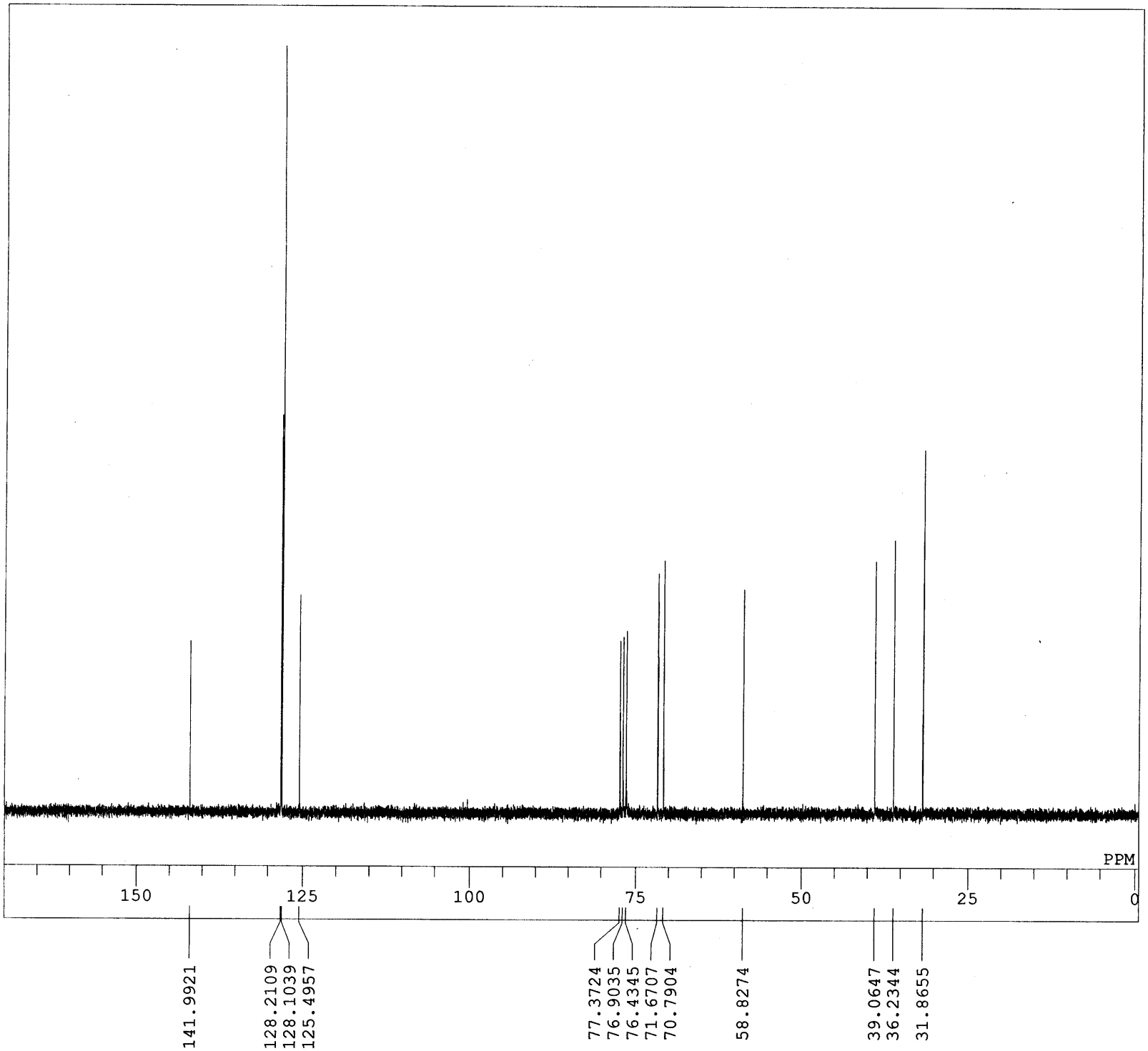



| | |
|---|---|
| DFILE | E:\030412-1.als |
| COMNT | 6c |
| DATIM | Tue Jan 11 08:51:23 2005 |
| OBNUC | 1H |
| EXMOD | NON |
| OBFRQ | 300.40 MHz |
| OBSET | 131.10 KHz |
| OBFIN | 31.00 Hz |
| POINT | 32768 |
| FREQU | 8000.00 Hz |
| SCANS | 16 |
| ACQTM | 4.0960 sec |
| PD | 1.0000 sec |
| PW1 | 4.30 usec |
| IRNUC | 1H |
| CTEMP | 23.3 c |
| SLVNT | CDCL3 |
| EXREF | 7.24 ppm |
| BF | 0.12 Hz |
| RGAIN | 13 |

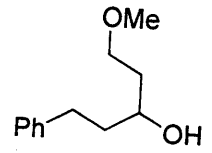



**6c**

6c

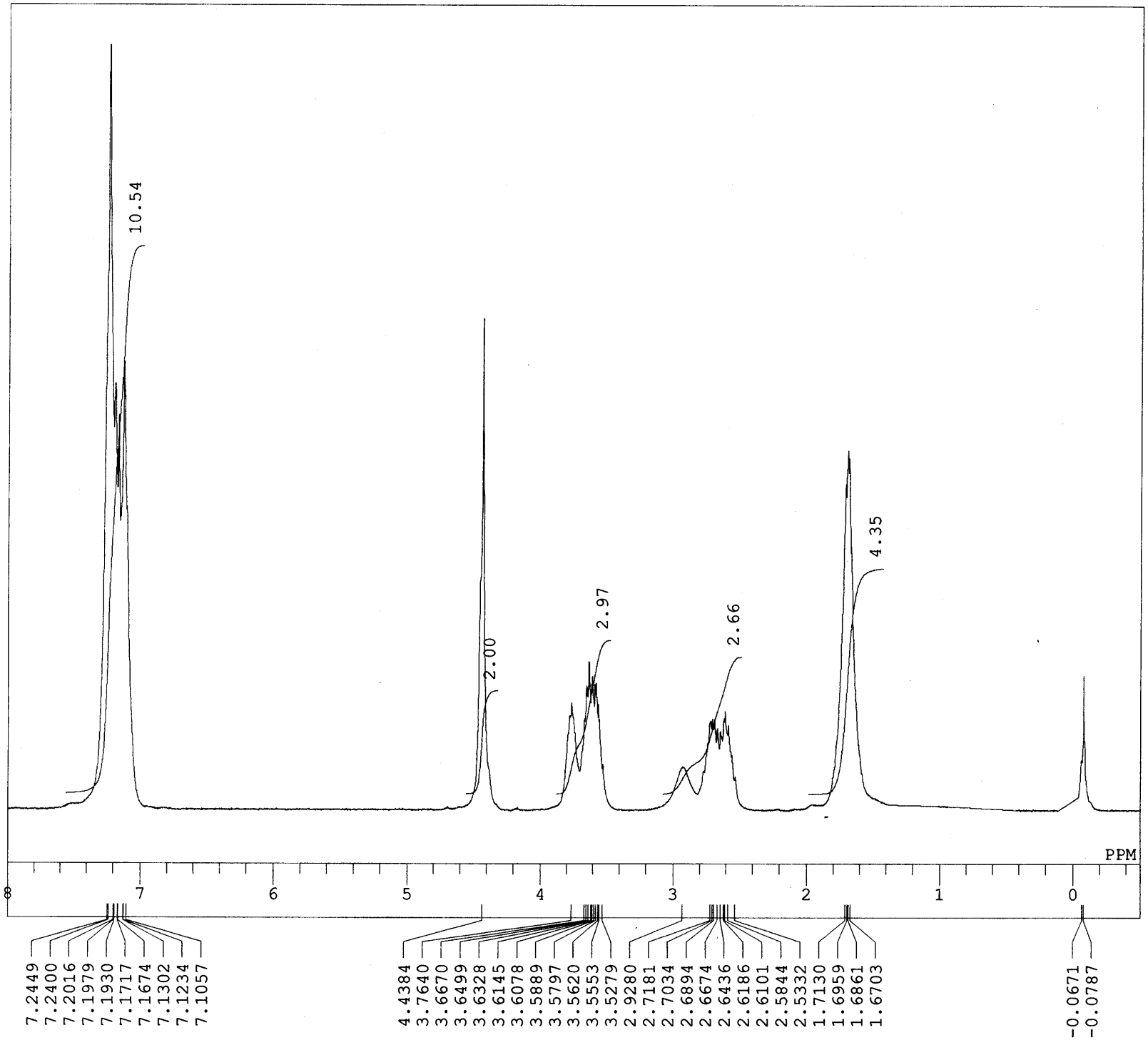



| | |
|---|---|
| DFILE | E:\030412-2.als |
| COMNT | 6c |
| DATIM | Tue Jan 11 09:15:45 2005 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 233 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.00 usec |
| IRNUC | 1H |
| CTEMP | 17.4 c |
| SLVNT | CDCL3 |
| EXREF | 0.00 ppm |
| BF | 0.12 Hz |
| RGAIN | 26 |

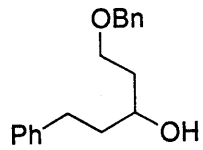



**6c**

6d

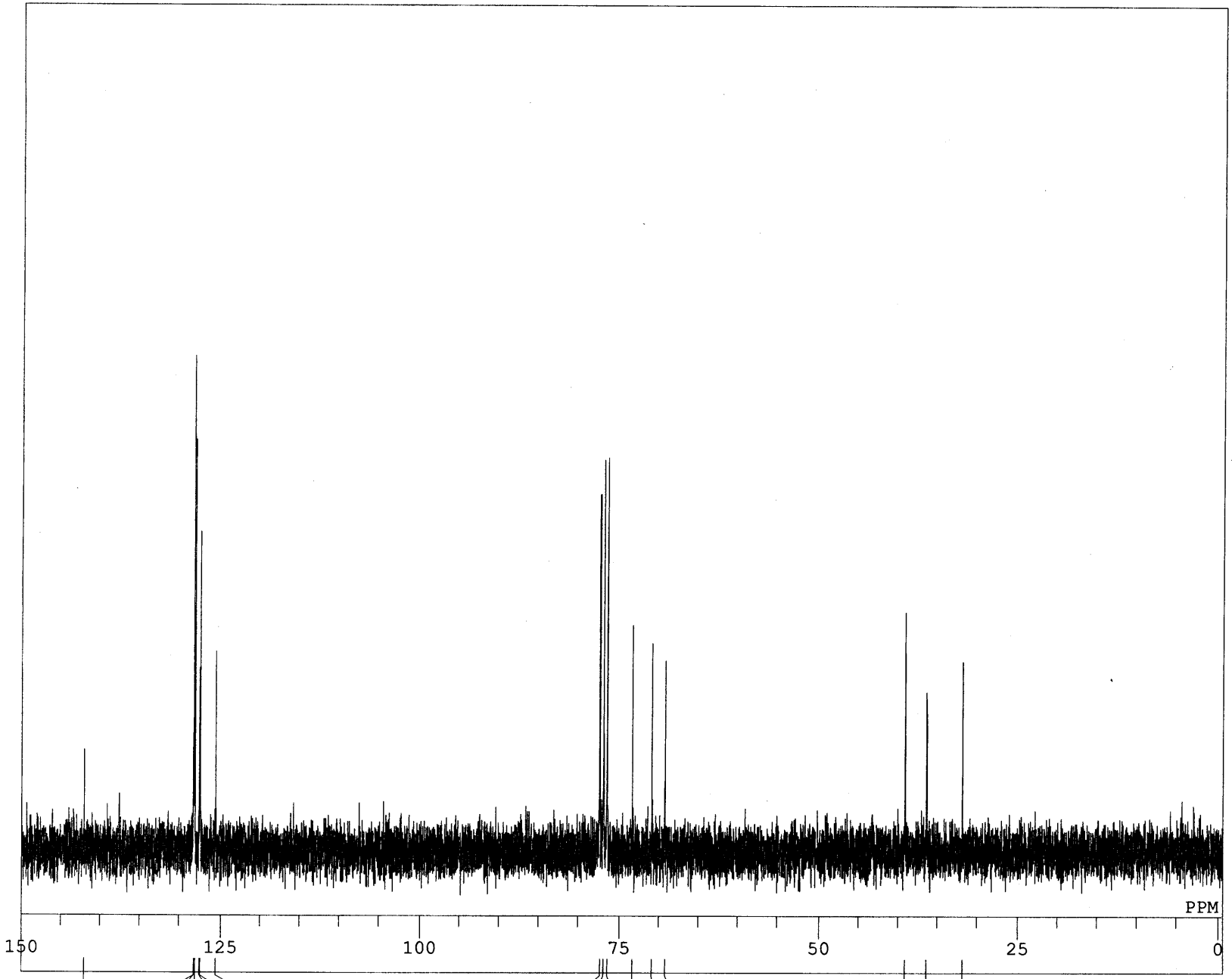



| | |
|---|---|
| DFILE | E:\CAN\OBn\H-NMR.als |
| COMNT | 6d |
| DATIM | Fri Dec 24 17:09:30 2004 |
| OBNUC | 1H |
| EXMOD | NON |
| OBFRQ | 270.05 MHz |
| OBSET | 112.00 KHz |
| OBFIN | 5800.00 Hz |
| POINT | 32768 |
| FREQU | 5402.40 Hz |
| SCANS | 16 |
| ACQTM | 6.0655 sec |
| PD | 0.9350 sec |
| PW1 | 5.90 usec |
| IRNUC | 1H |
| CTEMP | 18.7 c |
| SLVNT | CDCL3 |
| EXREF | 7.24 ppm |
| BF | 0.12 Hz |
| RGAIN | 16 |

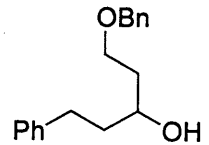



**6d**

6d

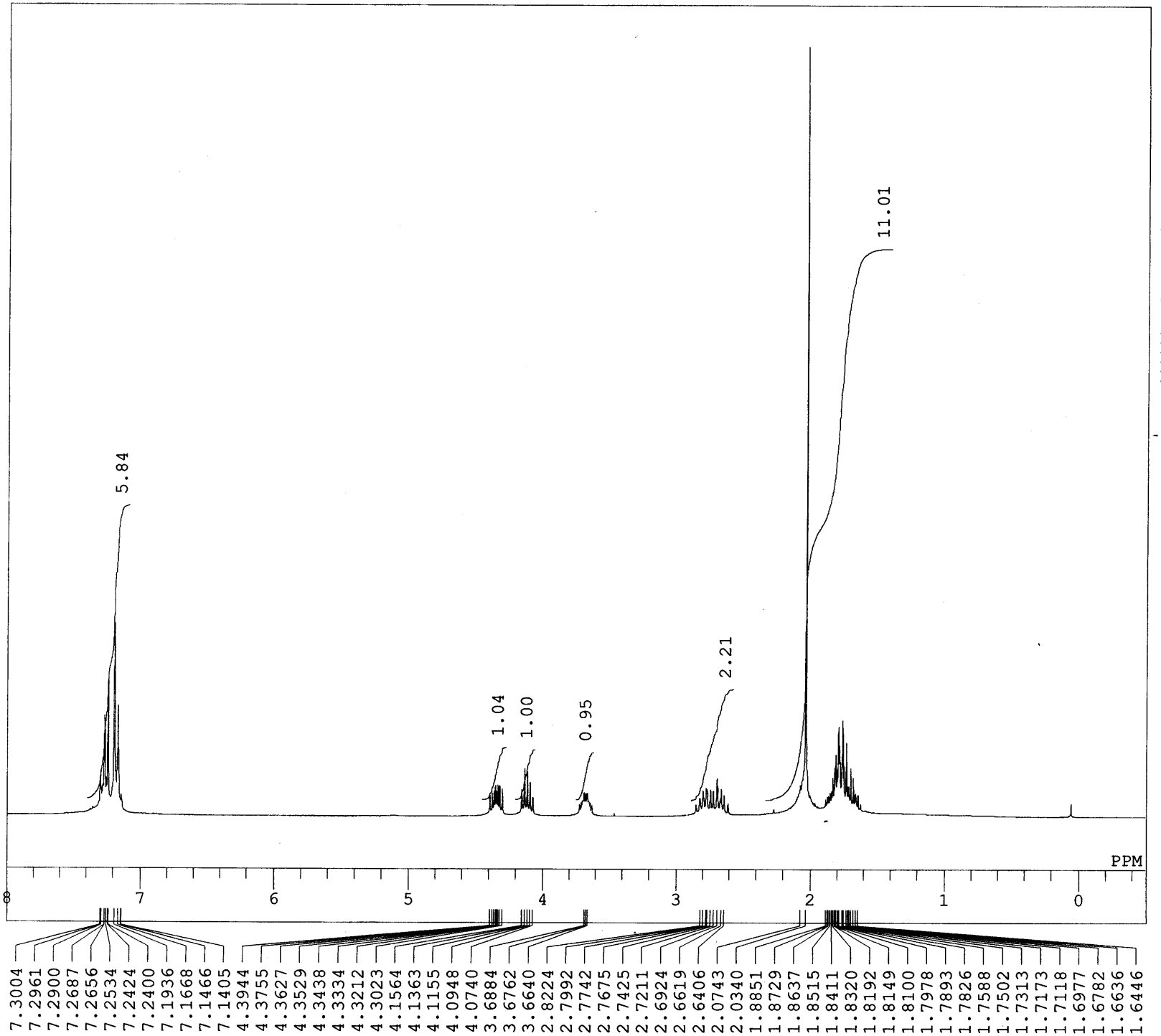



| | |
|---|---|
| DFILE | E:\CAN\OBn\C-NMR.als |
| COMNT | 6d |
| DATIM | Fri Dec 24 17:16:21 2004 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 128 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.10 usec |
| IRNUC | 1H |
| CTEMP | 19.6 c |
| SLVNT | CDCL3 |
| EXREF | 77.00 ppm |
| BF | 0.12 Hz |
| RGAIN | 26 |

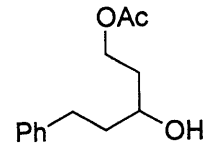



**6d**

6e

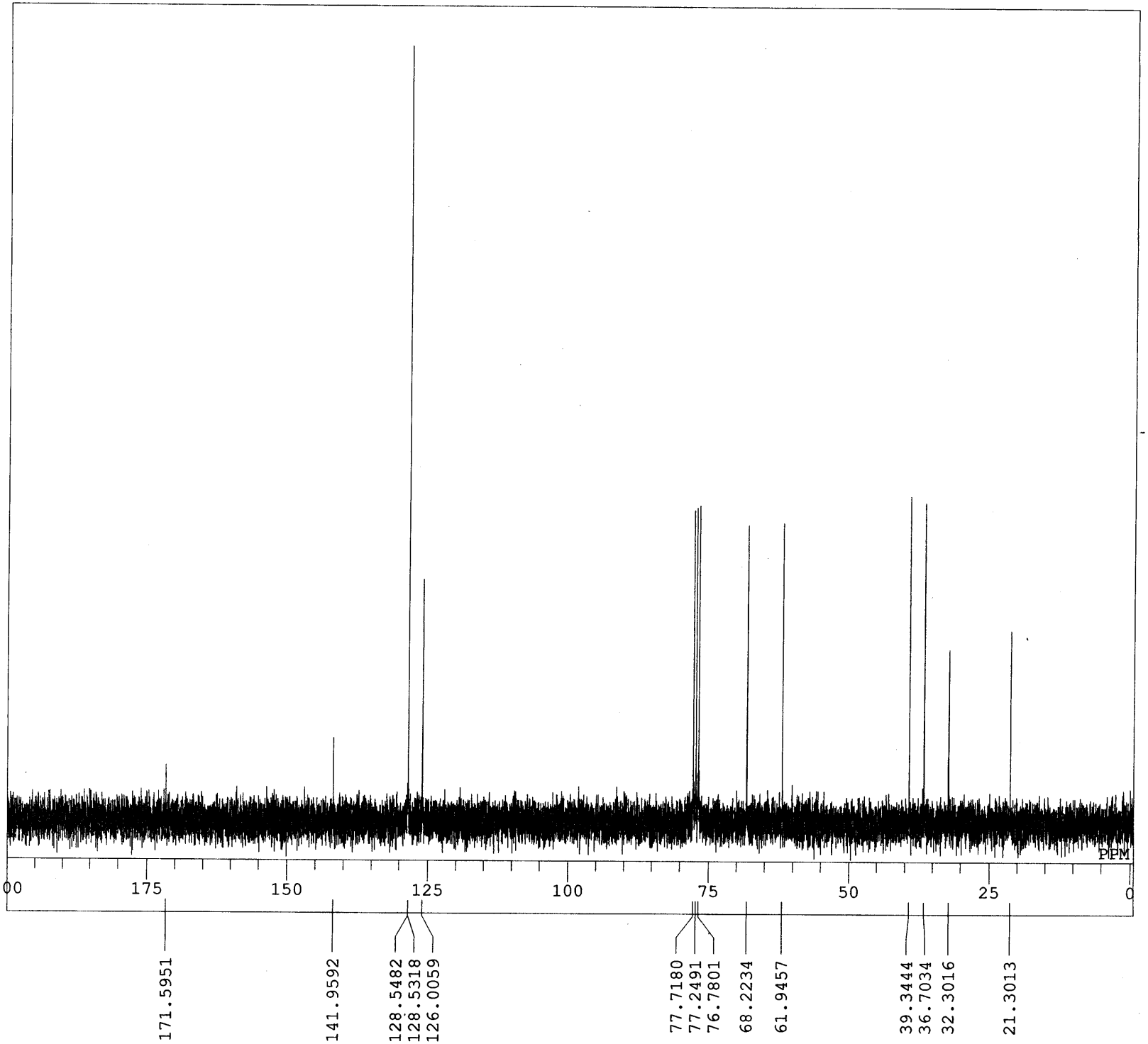



| | |
|---|---|
| DFILE | E:\CAN\OAc\H-NMR.als |
| COMNT | 6e |
| DATIM | Mon Dec 20 21:07:31 2004 |
| OBNUC | 1H |
| EXMOD | NON |
| OBFRQ | 270.05 MHz |
| OBSET | 112.00 KHz |
| OBFIN | 5800.00 Hz |
| POINT | 32768 |
| FREQU | 5402.40 Hz |
| SCANS | 16 |
| ACQTM | 6.0655 sec |
| PD | 0.9350 sec |
| PW1 | 5.90 usec |
| IRNUC | 1H |
| CTEMP | 19.5 c |
| SLVNT | CDCL3 |
| EXREF | 7.24 ppm |
| BF | 0.12 Hz |
| RGAIN | 16 |

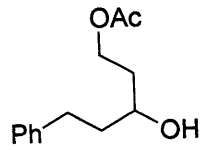



**6e**

6e

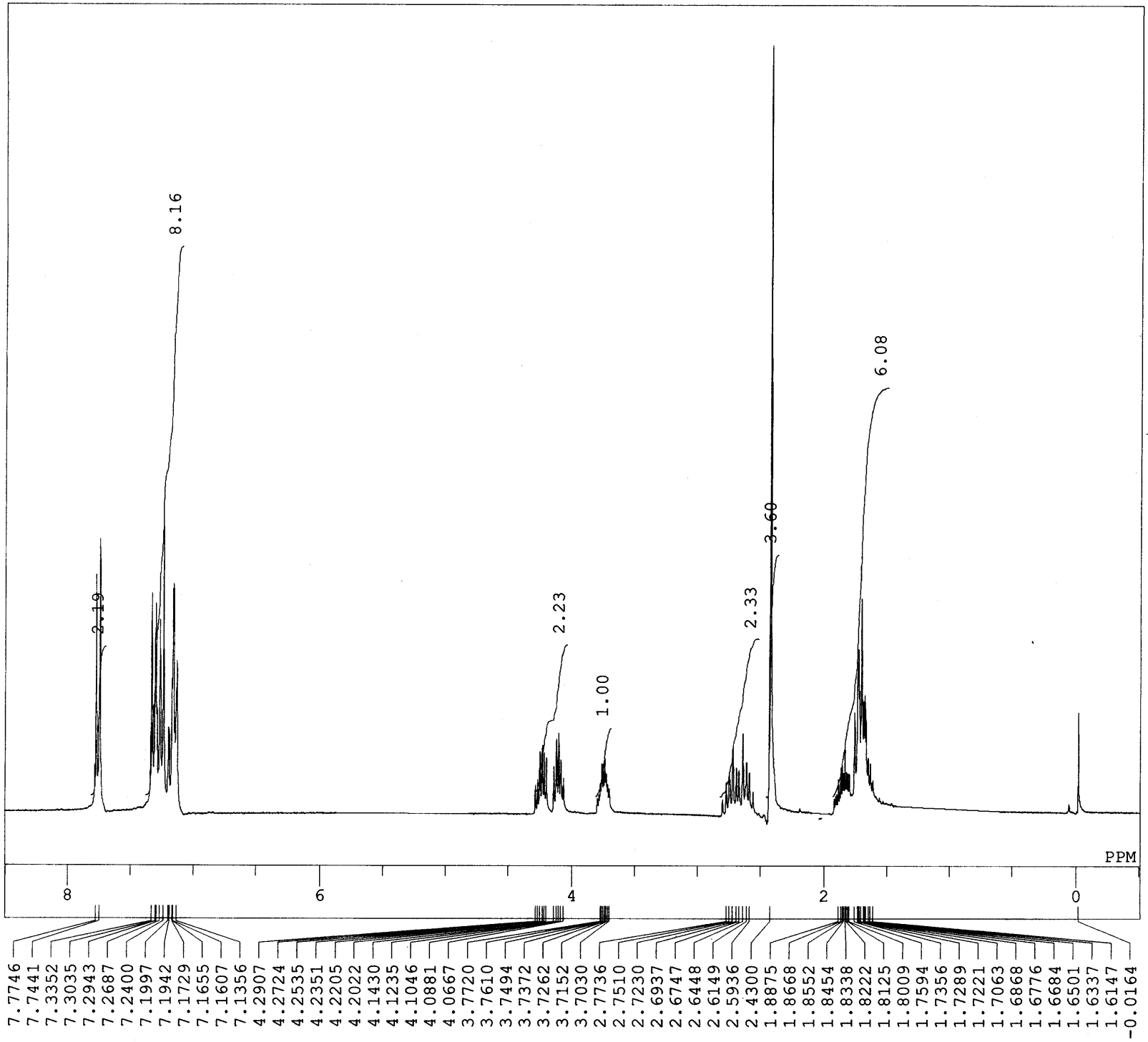



| | |
|---|---|
| DFILE | E:\CAN\OAc\C-NMR.als |
| COMNT | 6e |
| DATIM | Mon Dec 20 21:14:24 2004 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 128 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.10 usec |
| IRNUC | 1H |
| CTEMP | 20.7 c |
| SLVNT | CDCL3 |
| EXREF | 0.00 ppm |
| BF | 0.12 Hz |
| RGAIN | 26 |

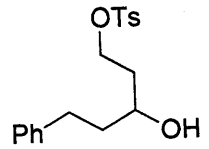



**6e**

6f

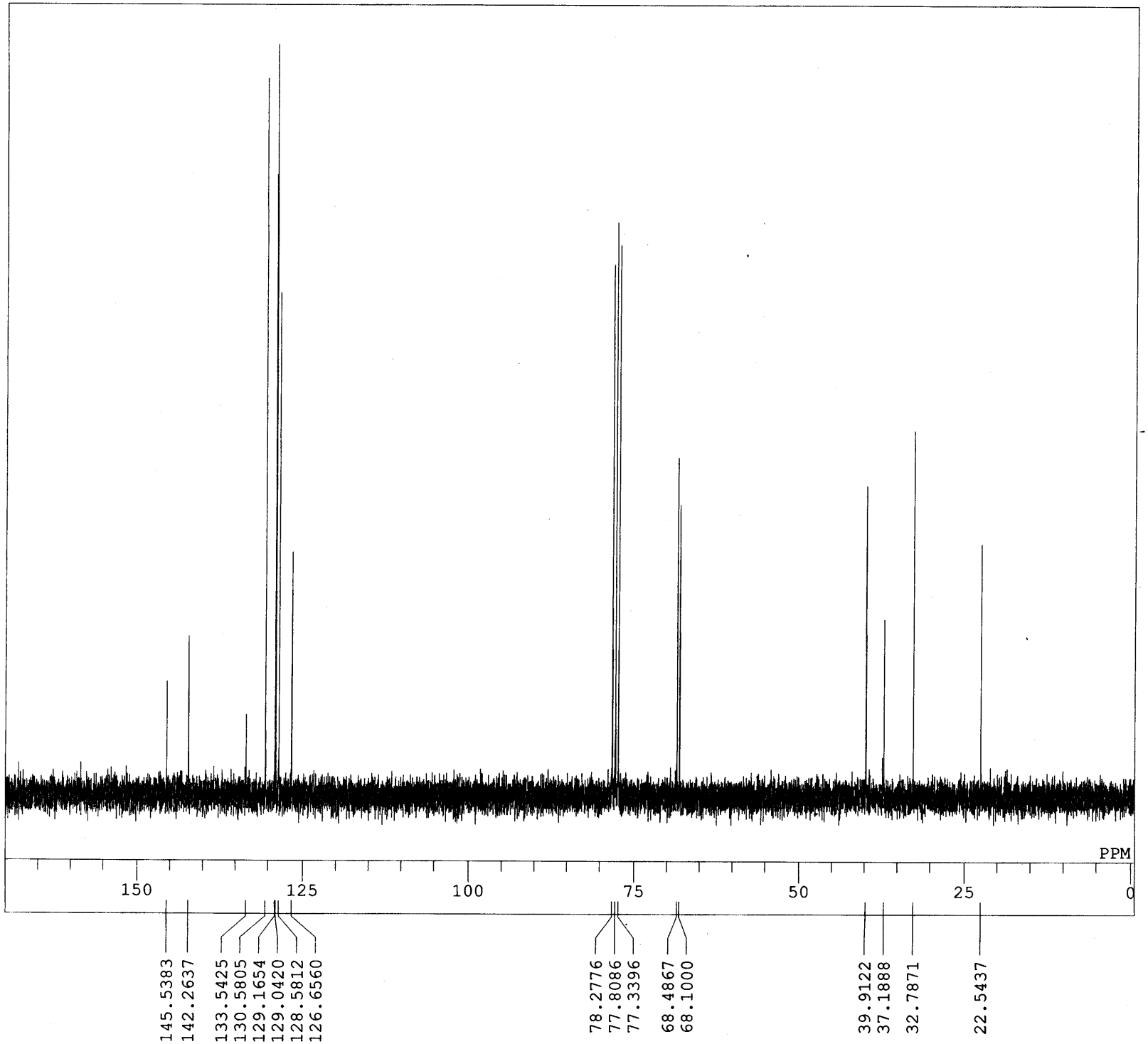



| | |
|---|---|
| DFILE | E:\030426-1.als |
| COMNT | 6f |
| DATIM | Fri Jan 21 18:21:28 2005 |
| OBNUC | 1H |
| EXMOD | NON |
| OBFRQ | 270.05 MHz |
| OBSET | 112.00 KHz |
| OBFIN | 5800.00 Hz |
| POINT | 32768 |
| FREQU | 5402.40 Hz |
| SCANS | 16 |
| ACQTM | 6.0655 sec |
| PD | 0.9350 sec |
| PW1 | 5.50 usec |
| IRNUC | 1H |
| CTEMP | 18.3 c |
| SLVNT | CDCL3 |
| EXREF | 7.24 ppm |
| BF | 0.00 Hz |
| RGAIN | 16 |

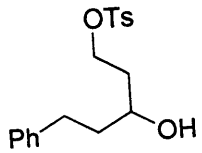



**6f**

6f

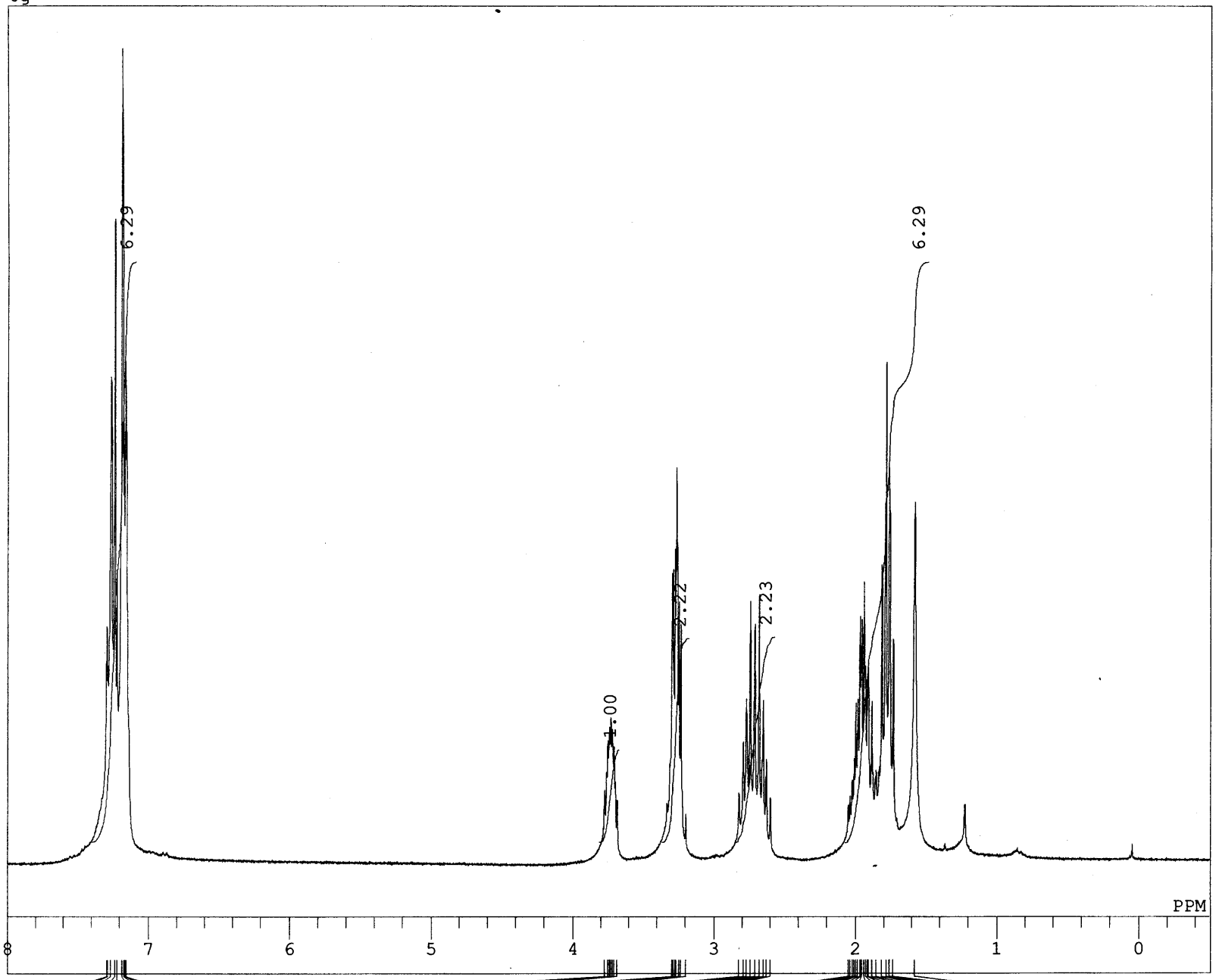



| | |
|---|---|
| DFILE | E:\030426-2.als |
| COMNT | 6f |
| DATIM | Fri Jan 21 18:55:52 2005 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 537 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.00 usec |
| IRNUC | 1H |
| CTEMP | 18.9 c |
| SLVNT | CDCL3 |
| EXREF | 0.00 ppm |
| BF | 0.00 Hz |
| RGAIN | 26 |

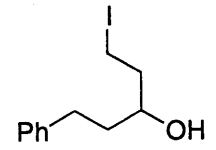



**6f**

6g




| | |
|---|---|
| DFILE | E:\CAN\I\H-NMR.als |
| COMNT | 6g |
| DATIM | Tue Dec 28 17:02:01 2004 |
| OBNUC | 1H |
| EXMOD | NON |
| OBFRQ | 270.05 MHz |
| OBSET | 112.00 KHz |
| OBFIN | 5800.00 Hz |
| POINT | 32768 |
| FREQU | 5402.40 Hz |
| SCANS | 16 |
| ACQTM | 6.0655 sec |
| PD | 0.9350 sec |
| PW1 | 5.50 usec |
| IRNUC | 1H |
| CTEMP | 18.5 c |
| SLVNT | CDCL3 |
| EXREF | 7.24 ppm |
| BF | 0.00 Hz |
| RGAIN | 16 |

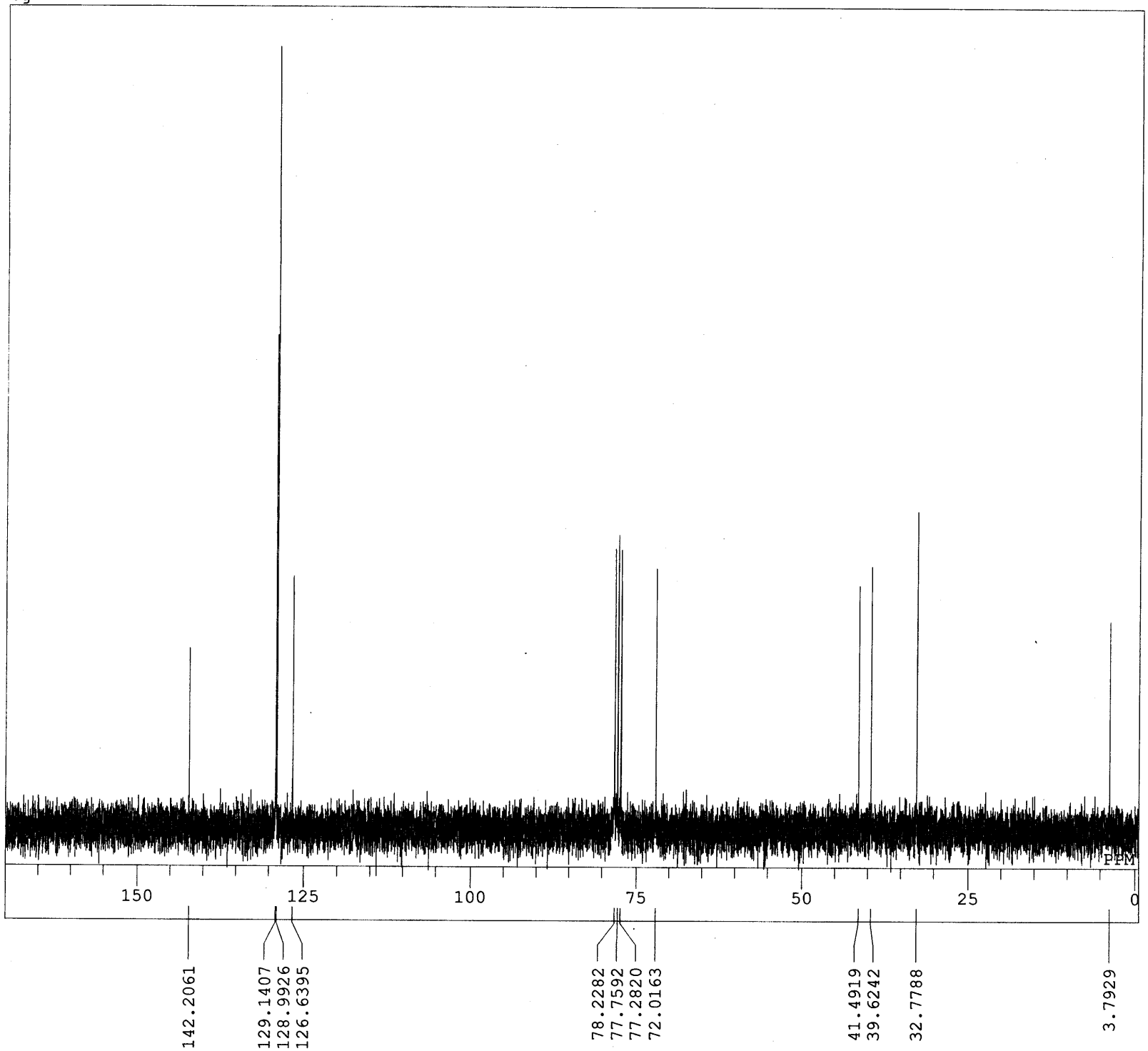



**6g**

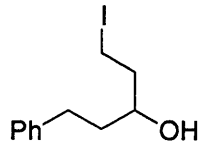
7.2937 7.2870 7.2656 7.2400 7.2223 7.1857 7.1741 7.1649 7.1576

3.7750 3.7530 3.7390 3.7299 3.7213 3.7085 3.6853 3.2978 3.2911 3.2777 3.2691 3.2496 3.2362 3.2002 2.8230 2.7944 2.7718 2.7437 2.7126 2.6808 2.6509 2.6290 2.6003 2.0523 2.0395 2.0248 2.0108 1.9992 1.9852 1.9711 1.9632 1.9571 1.9418 1.9315 1.9168 1.9107 1.8863 1.8564 1.8149 1.7856 1.7624 1.7582 1.7337 1.5818

6g

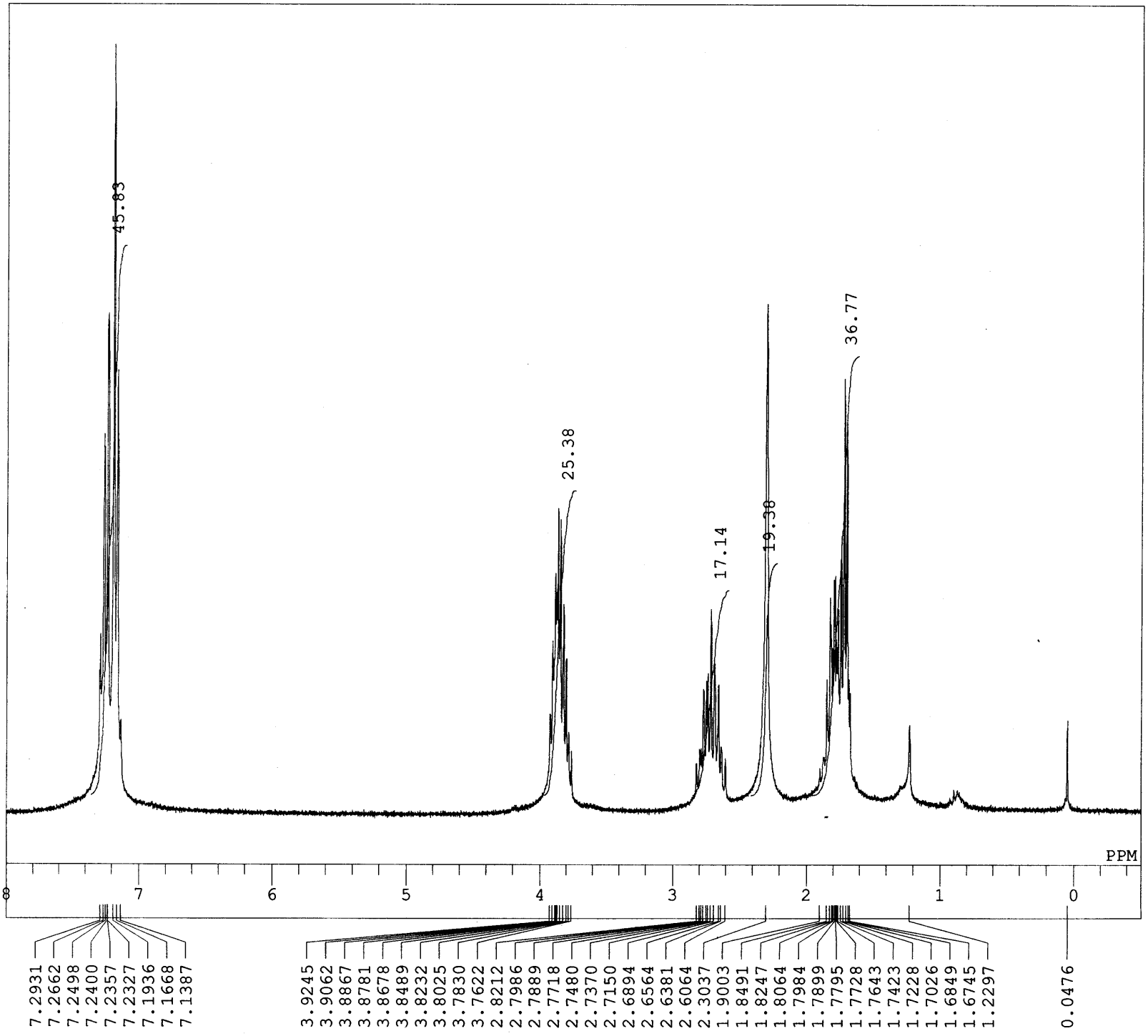



| | |
|---|---|
| DFILE | E:\CAN\I\C-NMR.als |
| COMNT | 6g |
| DATIM | Tue Dec 28 17:08:50 2004 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 128 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.00 usec |
| IRNUC | 1H |
| CTEMP | 19.3 c |
| SLVNT | CDCL3 |
| EXREF | 0.00 ppm |
| BF | 0.00 Hz |
| RGAIN | 26 |

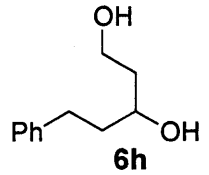



**6g**

6h

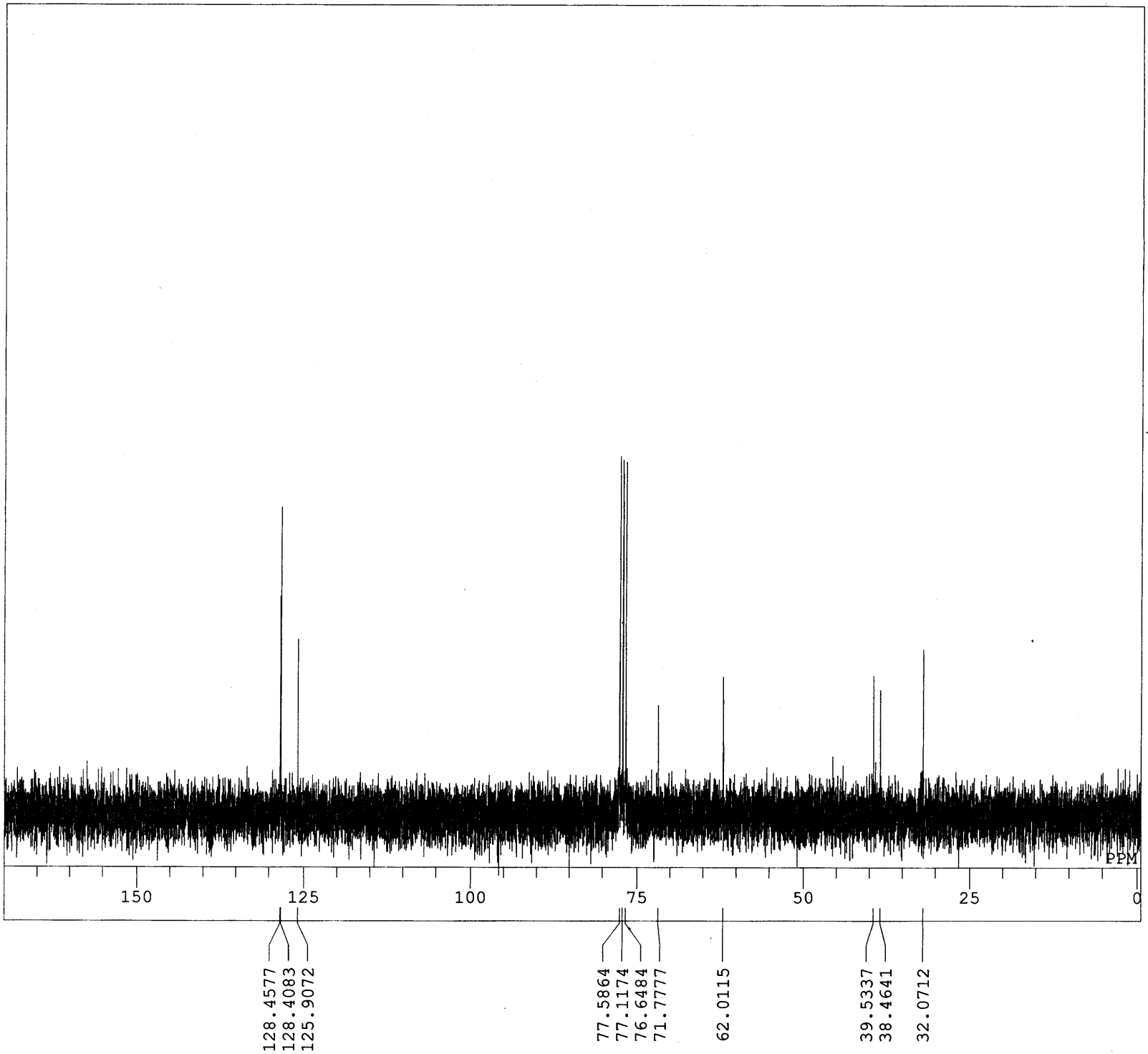



| | |
|---|---|
| DFILE | E:\CAN\OPMB\H-NMR.als |
| COMNT | 6h |
| DATIM | Tue Dec 28 17:14:35 2004 |
| OBNUC | 1H |
| EXMOD | NON |
| OBFRQ | 270.05 MHz |
| OBSET | 112.00 KHz |
| OBFIN | 5800.00 Hz |
| POINT | 32768 |
| FREQU | 5402.40 Hz |
| SCANS | 16 |
| ACQTM | 6.0655 sec |
| PD | 0.9350 sec |
| PW1 | 5.50 usec |
| IRNUC | 1H |
| CTEMP | 18.6 c |
| SLVNT | CDCL3 |
| EXREF | 7.24 ppm |
| BF | 0.00 Hz |
| RGAIN | 20 |

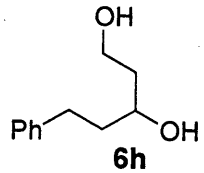



**6h**

6h

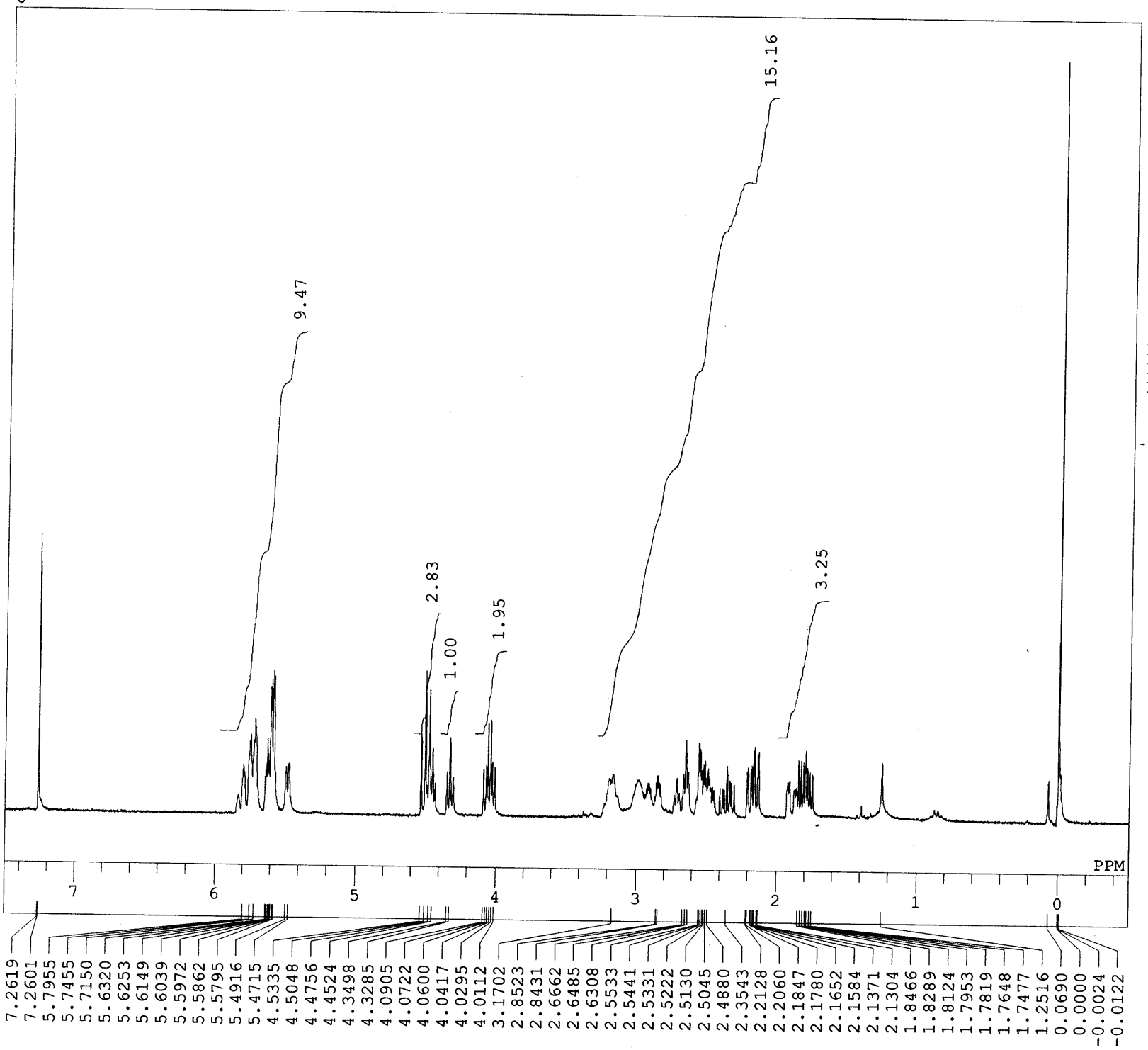



| | |
|---|---|
| DFILE | E:\CAN\OPMB\C-NMR.als |
| COMNT | 6h |
| DATIM | Tue Dec 28 17:21:25 2004 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 128 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.00 usec |
| IRNUC | 1H |
| CTEMP | 18.9 c |
| SLVNT | CDCL3 |
| EXREF | 0.00 ppm |
| BF | 0.00 Hz |
| RGAIN | 26 |

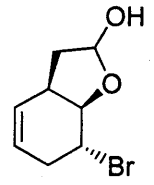



**6h**

8



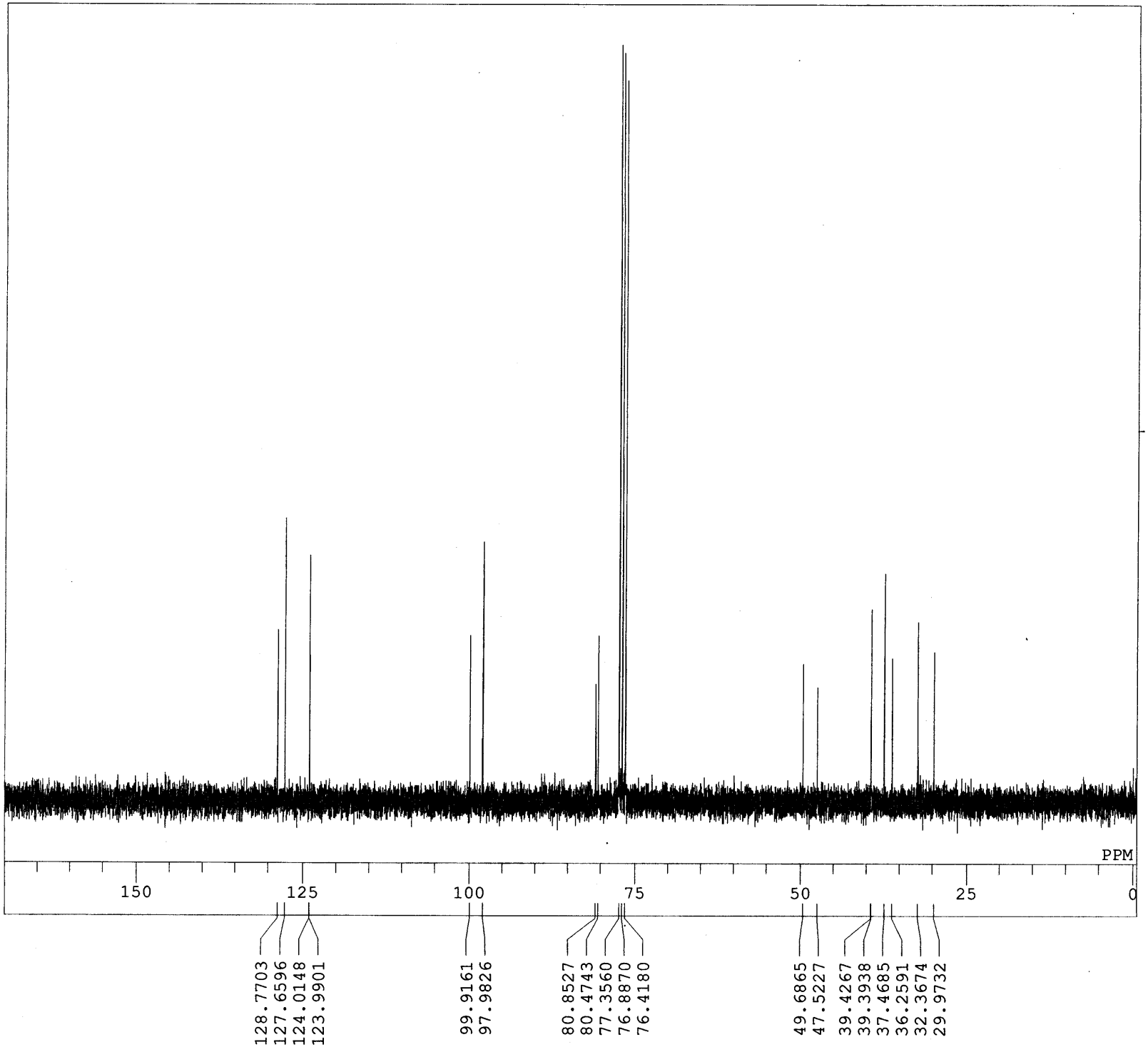

| | |
|---|---|
| DFILE | E:\030455-1.als |
| COMNT | 8 |
| DATIM | Tue Feb 08 09:01:21 2005 |
| OBNUC | 1H |
| EXMOD | NON |
| OBFRQ | 270.05 MHz |
| OBSET | 112.00 KHz |
| OBFIN | 5800.00 Hz |
| POINT | 32768 |
| FREQU | 5402.40 Hz |
| SCANS | 16 |
| ACQTM | 6.0655 sec |
| PD | 0.9350 sec |
| PW1 | 5.50 usec |
| IRNUC | 1H |
| CTEMP | 18.3 c |
| SLVNT | CDCL3 |
| EXREF | 0.00 ppm |
| BF | 0.00 Hz |
| RGAIN | 20 |



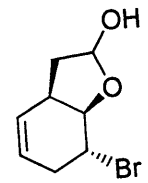

**8**

7.2619
7.2601
5.7955
5.7455
5.7150
5.6320
5.6253
5.6149
5.6039
5.5972
5.5862
5.5795
5.4916
5.4715
4.5335
4.5048
4.4756
4.4524
4.3498
4.3285
4.0905
4.0722
4.0600
4.0417
4.0295
4.0112
3.1702
2.8523
2.8431
2.6662
2.6485
2.6308
2.5533
2.5441
2.5331
2.5222
2.5130
2.5045
2.4880
2.3543
2.2128
2.2060
2.1847
2.1780
2.1652
2.1584
2.1371
2.1304
1.8466
1.8289
1.8124
1.7953
1.7819
1.7648
1.7477
1.2516
0.0690
0.0000
-0.0024
-0.0122

8

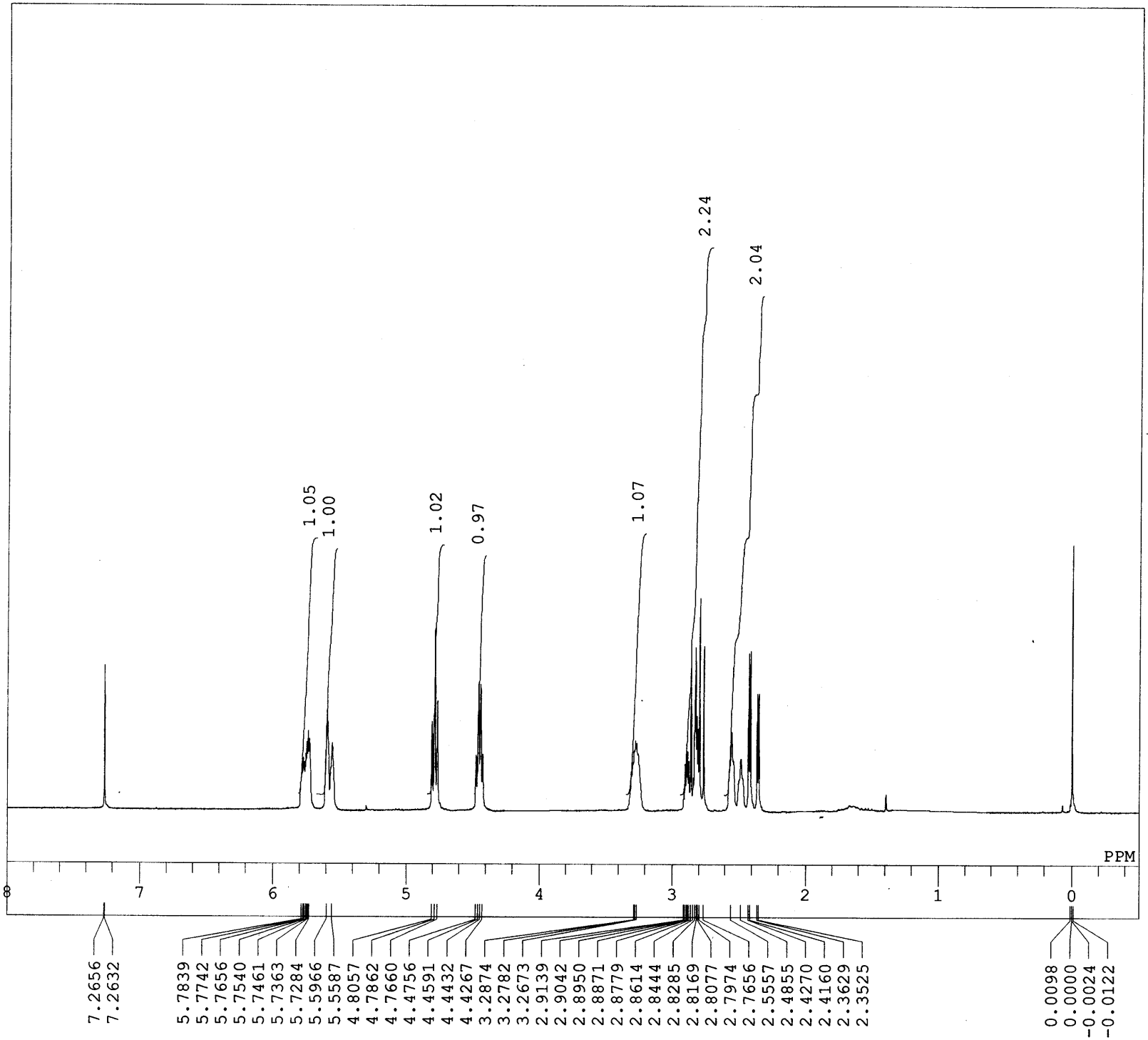



| | |
|---|---|
| DFILE | E:\030455-2.als |
| COMNT | 8 |
| DATIM | Tue Feb 08 09:51:53 2005 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 935 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.00 usec |
| IRNUC | 1H |
| CTEMP | 18.2 c |
| SLVNT | CDCL3 |
| EXREF | 0.00 ppm |
| BF | 0.00 Hz |
| RGAIN | 26 |

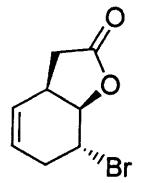



**8**

9

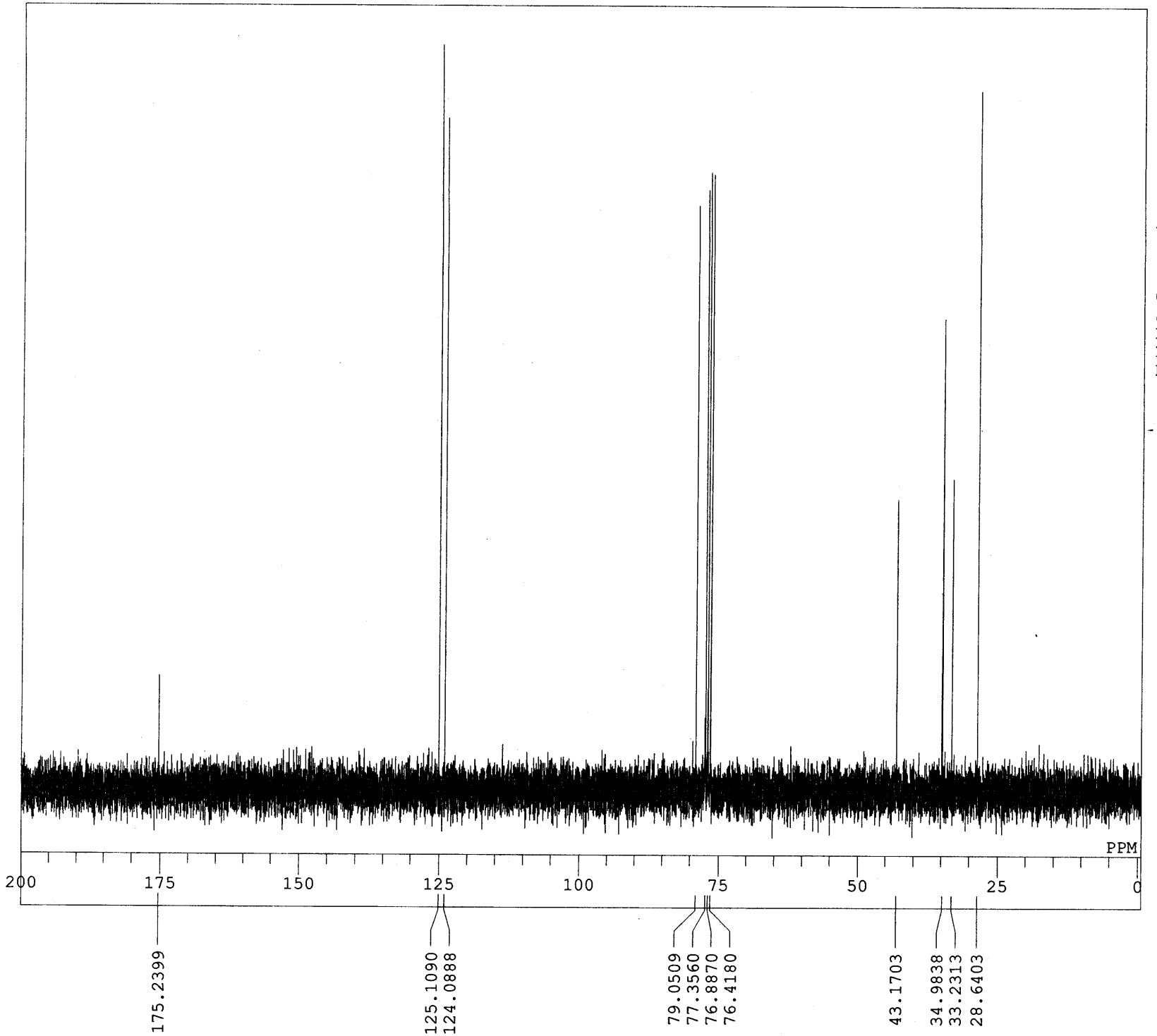



DFILE E:\030453-1.als
COMNT 9
DATIM Mon Feb 07 17:11:49 2005
OBNUC 1H
EXMOD NON
OBFRQ 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.00 Hz
POINT 32768
FREQU 5402.40 Hz
SCANS 16
ACQTM 6.0655 sec
PD 0.9350 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 17.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 19

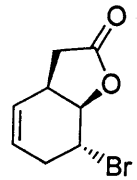



**9**

9

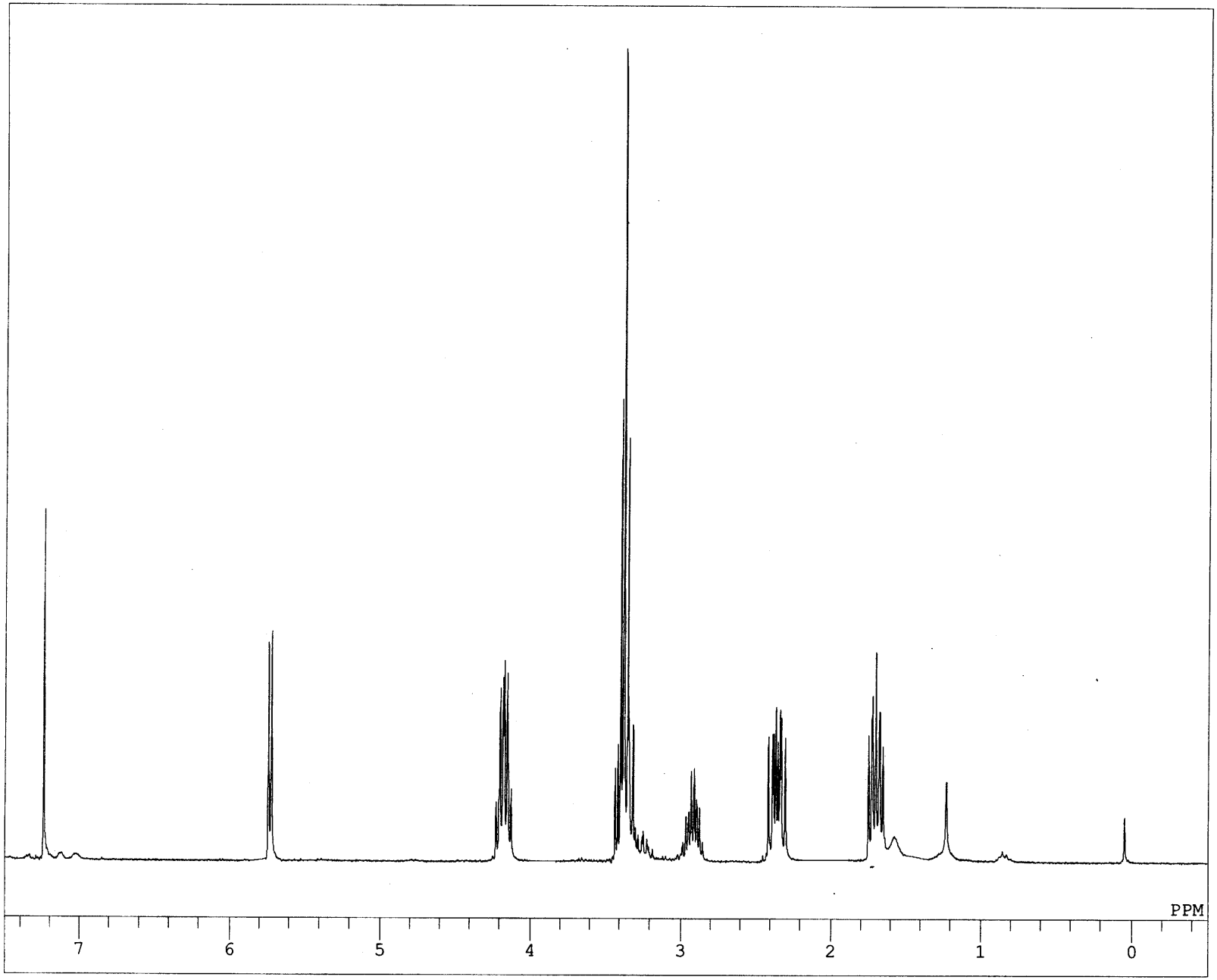



| | |
|---|---|
| DFILE | E:\030453-2.als |
| COMNT | 9 |
| DATIM | Mon Feb 07 17:29:22 2005 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 249 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.00 usec |
| IRNUC | 1H |
| CTEMP | 18.5 c |
| SLVNT | CDCL3 |
| EXREF | 0.00 ppm |
| BF | 0.00 Hz |
| RGAIN | 26 |

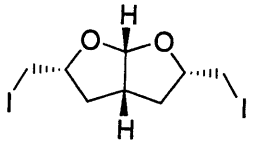



**9**

11a

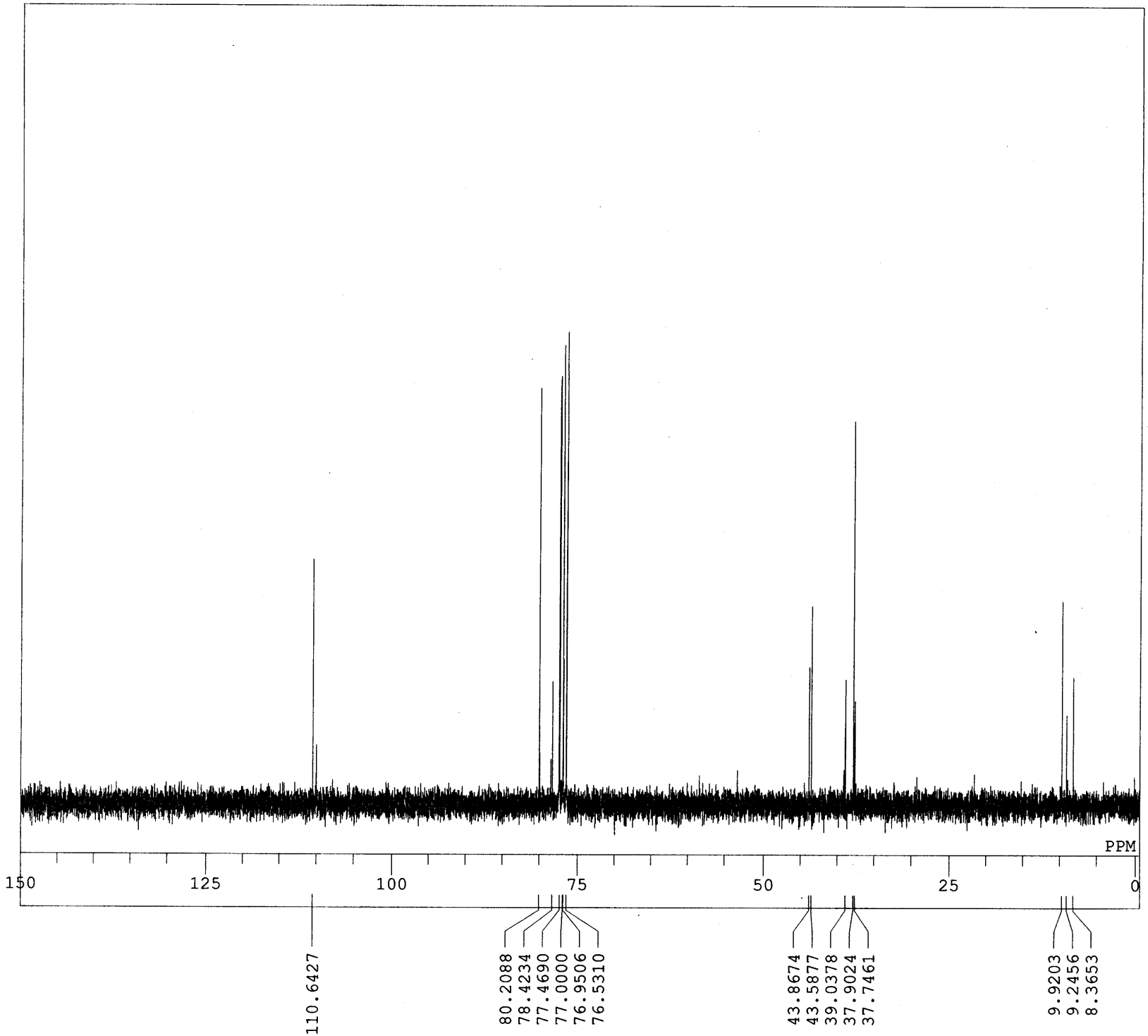



| | |
|---|---|
| DFILE | E:\domino\011163.als |
| COMNT | 11a |
| DATIM | Fri Jan 09 22:46:10 2004 |
| OBNUC | 1H |
| EXMOD | NON |
| OBFRQ | 270.05 MHz |
| OBSET | 112.00 KHz |
| OBFIN | 5800.00 Hz |
| POINT | 32768 |
| FREQU | 5402.40 Hz |
| SCANS | 16 |
| ACQTM | 6.0655 sec |
| PD | 0.9350 sec |
| PW1 | 5.60 usec |
| IRNUC | 1H |
| CTEMP | 18.5 c |
| SLVNT | CDCL3 |
| EXREF | 7.24 ppm |
| BF | 0.12 Hz |
| RGAIN | 21 |

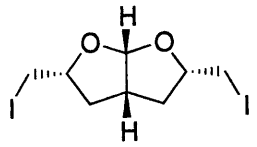



**11a**

11a

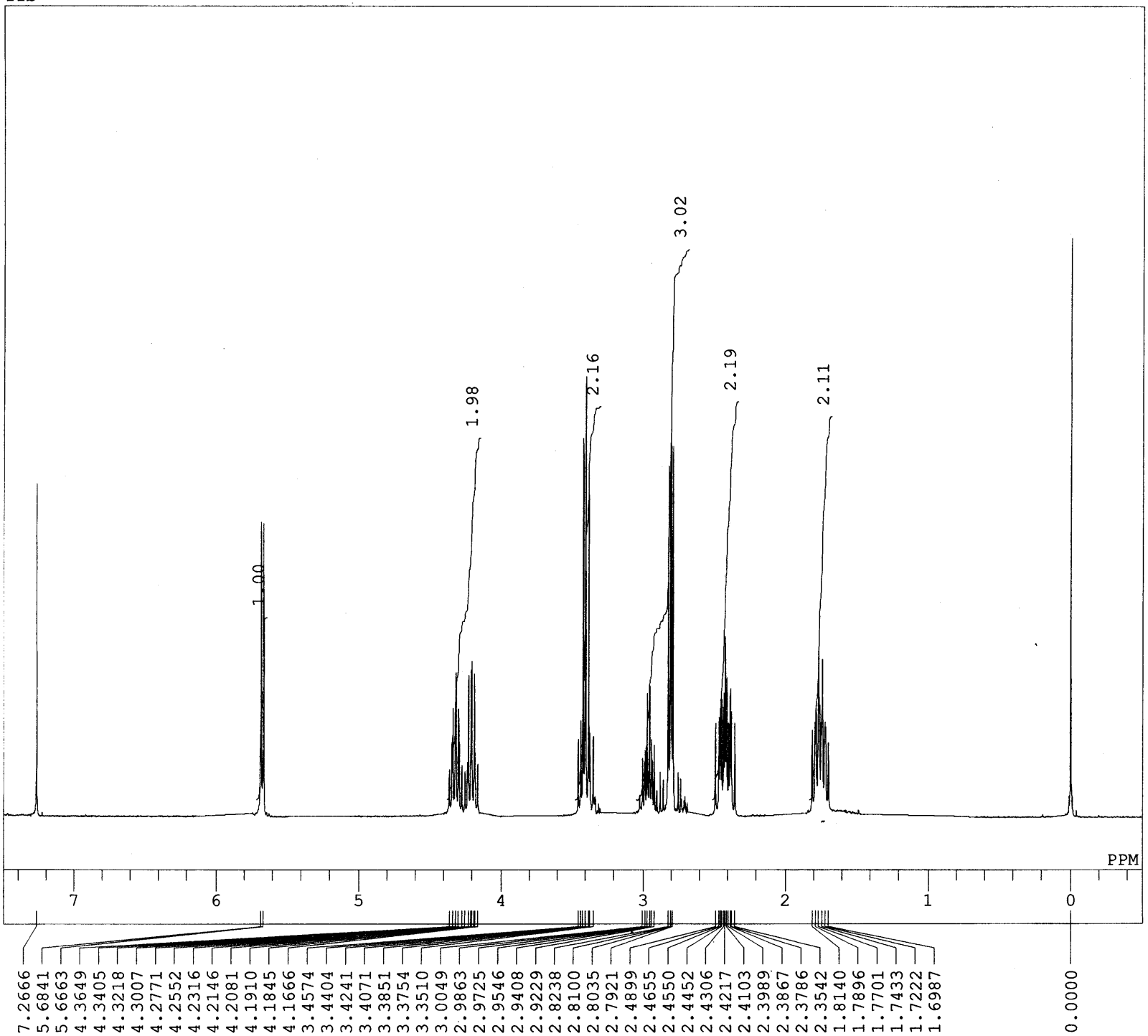



| | |
|---|---|
| DFILE | E:\030437-2.als |
| COMNT | 11a |
| DATIM | Fri Jan 28 16:17:09 2005 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 473 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.00 usec |
| IRNUC | 1H |
| CTEMP | 19.7 c |
| SLVNT | CDCL3 |
| EXREF | 77.00 ppm |
| BF | 0.00 Hz |
| RGAIN | 26 |

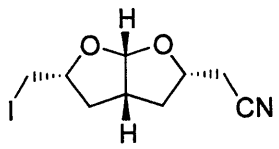



**11a**

11b

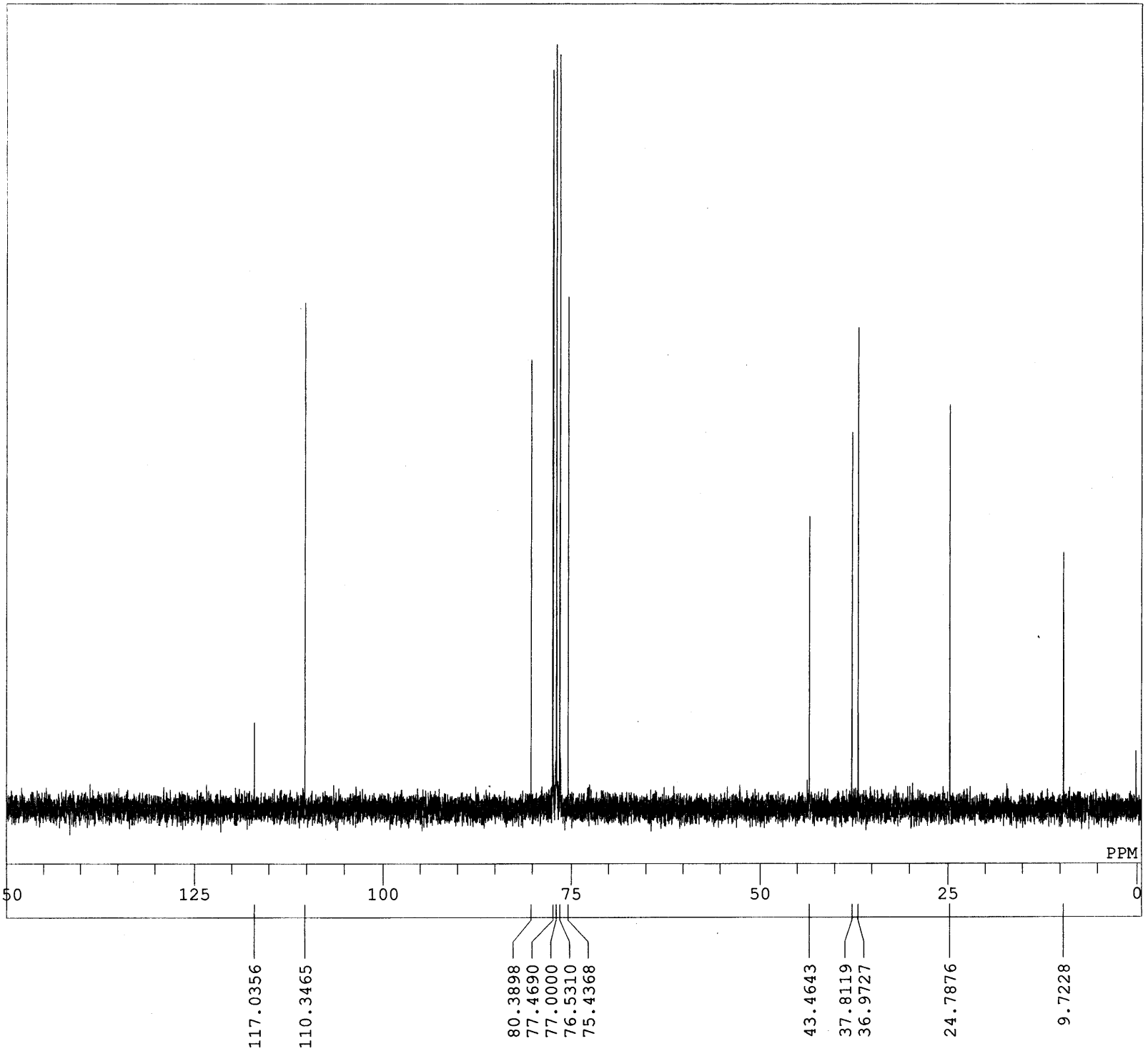



DFILE E:\030481-1.als
COMNT 11b
DATIM Mon Feb 21 12:39:49 2005
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.00 Hz
POINT 32768
FREQU 8000.00 Hz
SCANS 16
ACQTM 4.0960 sec
PD 2.0000 sec
PW1 4.30 usec
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 19

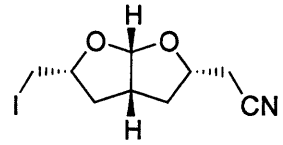



**11b**

11b




| | |
|---|---|
| DFILE | E:\030481-2.als |
| COMNT | 11b |
| DATIM | Mon Feb 21 13:33:03 2005 |
| OBNUC | 13C |
| EXMOD | BCM |
| OBFRQ | 67.80 MHz |
| OBSET | 135.00 KHz |
| OBFIN | 5200.00 Hz |
| POINT | 32768 |
| FREQU | 18315.00 Hz |
| SCANS | 802 |
| ACQTM | 1.7891 sec |
| PD | 1.2110 sec |
| PW1 | 4.00 usec |
| IRNUC | 1H |
| CTEMP | 18.2 c |
| SLVNT | CDCL3 |
| EXREF | 77.00 ppm |
| BF | 0.12 Hz |
| RGAIN | 26 |




**11b**