Highly Selective Diels-Alder Reactions of Dienophiles with 1,3-cyclohexadiene Mediated by $Yb(OTf)_3 \cdot 2H_2O$ and Ultra High Pressures (Supporting Information).

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Experimental

General

Melting points were determined using a Gallenkamp melting point apparatus and are uncorrected. Infrared spectra were recorded on a Perkin Elmer 2000 FT-IR instrument as thin films on NaCl plates. NMR experiments were performed on a Varian INOVA 400 or Mercury 400 instrument (unless otherwise indicated) and spectra were obtained in $CDCl_3$ (referenced to residual $CHCl_3$ at 7.26 ppm for ^{1}H or solvent at 77.00 ppm for ^{13}C). Coupling values (*J*) are in Hz. Diastereomeric excesses were determined by integrations of well separated peaks of the mixtures of adducts. For inseparable diastereomers, minor (*exo*-product) resonances are indicated in square brackets. Mass spectra were obtained on a Finnigan MAT 8200 spectrometer at 70 eV or as noted. Elemental analyses were performed at Chemisar Laboratories Inc., Guelph, Ontario.

Hyperbaric conditions were achieved using a LECO™ Tempres high pressure chemical reactor. Dichloromethane (DCM), toluene and THF were distilled according to the standard procedures.[1] *trans*-Cinnamaldehyde was distilled prior to use. All other reagents were used as purchased from Aldrich or Lancaster. Reactions were checked for completion by TLC (EM Science, silica gel 60 F_{254}) and/or ^{1}H NMR. Flash chromatography (FC) was done using silica gel purchased from Silicycle Chemical Division Inc. (230-400 Mesh).

Procedures

1-[(1*S,2*R**,3*R**,4*S**)-3-phenylbicyclo[2.2.2]oct-5-en-2-yl]ethanone (*endo*-9) and 1-[(1*S**,2*S**,3*S**,4*S**)-3-phenylbicyclo[2.2.2]oct-5-en-2-yl]ethanone (*exo*-9).** A mixture of benzylidene acetone **8** (1.07 g, 7.32 mmol) and 1,3-cyclohexadiene **7** (2.80 mL, 29.4 mmol) were heated in a sealed tube at 180-220 °C for 4 d. After cooling to RT, excess **7** was removed *in vacuo* and the remaining residue purified by FC (50:50, DCM:hexane) to afford ***endo*-9** (788 mg, 47%) and ***exo*-9** (457 mg, 28%) as a colorless oils. ***endo*-9**: See Table 1 for NMR data and assignments and Figure 1 for numbering and relevant NOESY correlations; IR 1710, 1600, 1498 cm^{-1}; MS *m/z* (relative intensity) 226 (M$^+$, 27), 147 (100), 80 (72); HRMS calcd for $C_{16}H_{18}O$: 226.1358, found: 226.1355. ***exo*-9**: See Table 2 for NMR data and assignments and Figure 2 for numbering and relevant NOESY correlations; IR 1707, 1603, 1495 cm^{-1}; MS *m/z* (relative intensity) 226 (M$^+$, 5), 147 (94), 80 (100); HRMS calcd for $C_{16}H_{18}O$: 226.1358, found: 226.1357.

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(1) Perrin, D. D.; Armarego, W. L. F. In *Purification of Laboratory Chemicals*, 3rd ed.; Pergamon: New York, 1988.

Diethyl 3-(*N*-tosylindolylidene)malonate (10h). Similar to a literature method,[2] a mixture of toluene (15 mL), *N*-tosylindole-3-carboxaldehyde[3] (1.58 g, 5.29 mmol), diethylmalonate (0.85 mL, 5.60 mmol), piperidine (0.11 mL, 1.11 mmol), HOAc (0.15 mL, 2.62 mmol) and 4 Å molecular sieves (1.5 g) were refluxed for 22 h. The reaction mixture was cooled to RT and filtered through Celite™. The filtrate was washed with water, separated and the organics dried over $MgSO_4$, filtered and concentrated *in vacuo*. The crude residue was purified by FC (Et_2O:hexanes, 1:1) then recrystallized (DCM/hexanes) to afford **10h** (1.03 g, 44%): mp 108-109 °C (DCM/hexanes); ^1H NMR (Varian Gemini, 300 MHz) δ 8.00-7.96 (m, 2H), 7.87 (d, J = 0.8, 1H), 7.81-7.76 (m, 2H), 7.66 (dd, J = 6.6, 1.8, 1H), 7.42-7.30 (m, 2H), 7.26-7.23 (m, 2H), 4.41 (q, J = 7.1, 2H), 4.32 (q, J = 7.1, 2H), 2.35 (s, 3H), 1.36 (t, J = 7.1, 3H), 1.34 (t, J = 7.1, 3H); ^{13}C NMR δ 166.64, 164.10, 145.58, 134.62, 134.41, 131.71, 130.07, 129.65, 127.16, 126.98, 125.50, 125.42, 123.93, 119.13, 115.15, 113.59, 61.95, 61.61, 21.59, 14.16, 14.02; IR 1733, 1626, 1250 cm^{-1}; MS *m/z* (relative intensity) 441 (M^+, 77), 212 (29), 140 (86), 91 (100), 77 (84); Anal. Calcd for $C_{23}H_{23}NO_6S$: C, 62.57; H, 5.25; N, 3.18. Found: C, 62.72; H, 5.34; N, 3.18.

3-(N-tosylindolylidene)acetone (10i). A mixture of 3-(indolylidene)acetone[4] (1.78 g, 9.83 mmol), K_2CO_3 (5.47 g, 39.6 mmol), TsCl (3.95 g, 20.7 mmol) and THF (40 mL) were heated at reflux for 60 h then cooled to RT in a fashion similar to a literature protocol.[3] The reaction mixture was diluted with water and EtOAc (~50 mL each) and separated. The aqueous portion was extracted again with EtOAc and then the combined organics were washed with brine, dried over $MgSO_4$, filtered and concentrated *in vacuo*. The crude product was purified by successive FC (EtOAc:Hexane, 1:2) and hexane trituration to afford **10i** (2.95 g, 88%): mp 117-120 °C (DCM/hexanes); ^1H NMR δ 7.99 (d, J = 9.0, 1H), 7.86 (s, 1H), 7.80-7.77 (m, 3H), 7.61 (d, J = 16.4, 1H), 7.37 (ddd, J = 8.2, 7.4, 1.2, 1H), 7.32 (ddd, J = 7.8, 7.4, 1.2, 1H), 7.25-7.33 (m, 2H), 6.80 (d, J = 16.4, 1H), 2.37 (s, 3H), 2.34 (s, 3H); ^{13}C NMR δ 198.12, 145.59, 135.55, 134.57, 134.40, 130.08, 128.75, 127.95, 127.10, 126.94, 125.55, 124.10, 120.64, 118.06, 113.78, 27.51, 21.56; IR 1666, 1627, 1609, 1448, 1375, 1176, 976 cm^{-1}; MS *m/z* (relative intensity) 339 (M^+,71), 184 (100), 155 (22), 91 (55); Anal. Calcd for $C_{19}H_{17}NO_3S$: C, 67.24; H, 5.05; N, 4.13. Found: C, 66.89; H, 4.99; N, 4.11.

Phenyl[(1*S,2*R**,3*R**,4*S**)-3-phenylbicyclo[2.2.2]oct-5-en-2-yl]methanone (*endo*-11a) and 1-[(1*S**,2*S**,3*S**,4*S**)-3-phenylbicyclo[2.2.2]oct-5-en-2-yl]methanone (*exo*-11a).** According to the general cycloaddition procedure. FC (50:50, DCM:hexanes) afforded a white crystalline solid (285 mg, 94%) found to be a mixture of 95:5 *endo:exo*. Further FC (33:67, DCM:hexanes) permitted isolation of the isomers or alternatively, the *exo*-isomer could be removed by recrystallization. White solid ***endo*-11a**: mp 124-125 °C

(2) Perron, Y. G.; Minor, W. F. *J. Org. Chem.* **1959**, *24*, 1165.

(3) Evans, D. D. *Aust. J. Chem.* **1973**, *26*, 2555.

(4) Bergman, J. *Acta Chem. Scand.* **1972**, *26*, 970.

(DCM/hexanes); See Table 3 for NMR data and assignments and Figure 3 for numbering and relevant NOESY correlations; IR 1685, 1448 cm^{-1}; MS *m/z* (relative intensity) 288 (M$^+$, 25), 209 (100), 131 (19), 105 (49), 80 (49); Anal. Calcd for $C_{21}H_{20}O$: C, 87.46; H, 6.99. Found: C, 87.27; H, 7.05. Colorless film ***exo*-11a**: ^{1}H NMR δ 7.93 (app d, J = 8.2, 2H), 7.53 (m, 1H), 7.44 (m, 2H), 7.21 (m, 2H), 7.15 (m, 1H), 6.54 (app t, J = 7.8, 6.7, 1H), 6.48 (app t, J = 7.8, 6.6, 1H), 3.78 (d, J = 6.6, 1H), 3.57 (dd, J = 6.3, 1.6, 1H), 2.88 (m, 1H), 2.82 (m, 1H), 1.90-1.84 (m, 1H), 1.61-1.54 (m, 1H), 1.36-1.28 (m, 1H), 1.12-1.05 (m, 1H); ^{13}C NMR δ 200.31, 147.02, 136.70, 134.49, 133.47, 132.80, 128.57, 128.39, 128.25, 127.42, 125.91, 55.85, 43.70, 36.24, 34.50, 26.50, 19.19; IR 1679, 1447 cm^{-1}; MS (CI: isobutane) *m/z* (relative intensity) 289 (M+H, 100), 209 (42), 105 (8); HRMS calcd for $C_{21}H_{21}O$ (M+H): 289.1592, found: 289.1315.

1-[(1*S,2*S**,3*R**,4*S**)-3-methylbicyclo[2.2.2]oct-5-en-2-yl]ethanone (*endo*-11b) and 1-[(1*S**,2*R**,3*S**,4*S**)-3-methylbicyclo[2.2.2]oct-5-en-2-yl]ethanone (*exo*-11b).** A mixture of 3-penten-2-one **10a** (167 mg, 1.98 mmol) and cyclohexadiene **7** (0.47 mL, 4.9 mmol) were heated in a sealed tube at 180-220 °C for 4 d. After cooling to RT, excess **7** was removed *in vacuo* and the remaining residue purified by FC (10:90, EtOAc:hexane) to afford each isomer contaminated with polymeric material. Each sample was dissolved in hexanes, treated with charcoal, filtered through Celite™ and concentrated to afford ***endo*-11b** (136 mg, 42%) and ***exo*-11b** (79 mg, 24%) as colorless oils. ***endo*-11b**: ^{1}H NMR δ 6.38 (dd, J = 7.9, 7.0, 1H), 6.08 (app t, J = 7.9, 6.7, 1H), 2.79 (m, 1H), 2.28 (m, 1H), 2.11 (s, 3H), 2.06 (d, J = 6.2, 1H), 1.90-1.86 (m, 1H), 1.80-1.73 (m, 1H), 1.58-1.49 (m, 2H), 1.35-1.27 (m, 1H), 1.09 (d, J = 7.0, 3H); ^{13}C NMR δ 210.02, 136.74, 130.52, 60.45, 35.86, 33.65, 32.78, 28.23, 26.05, 19.89, 18.04; IR 1709, 1369, 1354 cm^{-1}; MS *m/z* (relative intensity) 164 (M$^+$, 19), 121 (10), 93 (45), 80 (100); HRMS calcd for $C_{11}H_{16}O$: 164.1201, found: 164.1204. ***exo*-11b**: ^{1}H NMR δ 6.30 (app t, J = 7.6, 7.0, 1H), 6.20 (app t, J = 7.6, 6.6, 1H), 2.77 (m, 1H), 2.30 (m, 1H), 2.21 (app t, J = 7.0, 6.6, 1H), 2.15 (s, 3H), 1.92 (m, 1H), 1.50-1.46 (m, 1H), 1.38-1.31 (m, 1H), 1.23-1.16 (m, 1H), 1.06-0.98 (m, 1H), 0.77 (d, J = 6.6, 3H); ^{13}C NMR δ 209.83, 133.75, 133.05, 60.43, 36.58, 32.83, 29.32, 25.36, 22.79, 19.55; IR 1707, 1374, 1352 cm^{-1}. Alternatively, ***endo*-11b** was prepared according to the general procedure. Following FC (15:85, EtOAc:hexane), the mixture of isomers was distilled (52-65 ºC at ~0.35 mm Hg) to afford ***endo*-11b** (221 mg, 68%, 87% de): Anal. Calcd for $C_{11}H_{16}O$: C, 80.44; H, 9.82. Found: C, 80.08; H, 9.65.

1-[(1*S,2*R**,3*R**,4*S**)-3-isopropylbicyclo[2.2.2]oct-5-en-2-yl]ethanone (*endo*-11c) and 1-[(1*S**,2*S**,3*S**,4*S**)-3-isopropylbicyclo[2.2.2]oct-5-en-2-yl]ethanone (*exo*-11c).** According to the general cycloaddition procedure. FC (33:67, DCM:hexanes) afforded a colorless oil ***endo*-11c** (189 mg, 67%) and impure colorless film ***exo*-11c** (<3 mg, <1%). ***endo*-11c**: ^{1}H NMR δ 6.40 (app t, J = 7.5, 1H), 5.96 (app t, J = 7.5, 6.6, 1H), 2.76 (m, 1H), 2.59 (m, 1H), 2.23 (app d, J = 5.5, 1H), 2.14 (s, 3H), 1.66-1.51 (m, 3H), 1.40-1.29 (m, 2H), 1.06-0.99 (m, 1H), 0.90 (d, J = 6.6, 3H), 0.75 (d, J = 6.6, 3H); ^{13}C NMR δ 209.97, 137.50, 129.81, 59.10, 45.07, 33.94, 31.73, 31.35, 28.56, 26.54, 22.31, 20.95, 18.25; IR 2948, 1710, 1471 cm^{-1}; MS *m/z* (relative intensity) 192 (M$^+$, 22), 149 (26), 113 (38), 80 (100); HRMS calcd for $C_{13}H_{20}O$: 192.1514, found: 192.1516. ***exo*-11c**: ^{1}H NMR δ 6.30 (app t, J = 7.6, 6.6, 1H), 6.21 (app t, J = 7.6, 7.0, 1H), 2.76 (m, 1H), 2.62 (m, 1H), 2.20 (s, 3H), 2.17 (m, 1H), 1.86 (m, 1H), 1.53-1.45 (m, 1H), 0.86 (d, J = 6.6, 3H), 0.66 (d, J = 6.6, 3H); ^{13}C NMR δ 209.90, 133.84, 132.77, 57.85, 45.54, 33.22, 33.13, 32.18, 29.69, 25.32, 20.86, 20.26, 20.18.

(1*S,4*S**,5*R**,6*S**)-5-nitro-6-phenylbicyclo[2.2.2]oct-2-ene (*endo*-11g).**[5] According to the general cycloaddition procedure. FC (50:50, DCM:hexanes) afforded a viscous colorless oil (195 mg, 83%, 92% de) which gave a white solid upon trituration with hexanes: mp 47-48 °C; ^{1}H NMR δ 7.40-7.36 (m, 2H), 7.30-7.23 (m, 3H), 6.63 (dd, J = 7.9, 7.1, 1H), [6.41 (app t, J = 7.5, 1H)], [6.37 (app t, J = 7.5, 1H)], 6.21 (app t, J = 7.1, 1H), 4.91 (dd, J = 5.3, 2.1, 1H), [4.55 (m, 1H)], [3.75 (d, J = 5.6, 1H)], 3.53 (app t, J = 2.6, 2.1, 1H), 3.43 (m, 1H), [3.37 (m, 1H)], [2.81 (m, 1H)], 2.71 (dd, J = 6.2, 2.6, 1H), 1.77-1.70 (m, 1H), 1.66-1.50 (m, 2H), 1.10-1.02 (m, 1H); ^{13}C NMR δ 139.64, 136.72, [135.57], [130.09], 129.00, 128.69, [128.51], [128.46], 127.53, [127.21], 126.88, [92.65], 88.74, 47.79, [46.23], 36.67, [36.24], [36.08], 35.26, [26.01], 23.37, [18.21], 17.28; IR 2959, 1540, 1370 cm^{-1}; MS *m/z* (relative intensity) 182 (44), 154 (54), 91 (34), 80 (100); Anal. Calcd for $C_{14}H_{15}NO_2$: C, 73.34; H, 6.59; N, 6.11. Found: C, 73.52; H, 6.73; N, 6.14.

Diethyl-(1*S,3*S**,4*S**)-3-{1-tosyl-1*H*-indol-3-yl}bicyclo[2.2.2]oct-5-ene-2,2-dicarboxylate (*endo*-11h).** According to the general cycloaddition procedure with the exceptions noted in Table 2. FC (1:4, EtOAc:hexanes) afforded a white solid (98 mg, 79%, 60% de): mp 146-147 °C (DCM/hexanes); ^{1}H NMR δ [7.97 (d, 8.2, 1H)], 7.91 (app d, J = 8.2, 1H), [7.77 (d, 8.6, 1H)], 7.71 (m, 3H), 7.35 (s, 1H), 7.32 (s, 1H), 7.24-7.18 (m, 4H), 6.73 (app t, J = 7.4, 7.0, 1H), 6.51 (app t, J = 7.4, 7.0, 1H), [6.12 (app t, J = 7.0, 1H)], 4.40 (s, 1H), [4.37 (s, 1H)], 4.26-4.11 (m, 2H), [3.45 (m, 1H), 3.37 (m, 1H)] 3.21 (m, 1H), 3.03-2.95 (m, 1H), [2.86-2.81 (m, 1H)], 2.76 (br s, 1H), [2.56 (m, 1H), 2.31 (s, 3H)], 2.29 (s, 3H), 2.09-2.01 (m, 1H), [1.99-1.93 (m, 1H)], 1.71-1.67 (m 1H), 1.46-1.36 (m, 1H), 1.33-1.25 (m, 1H), 1.19 (t, J = 7.0, 3H), [1.16-1.05 (m, 1H), 0.29 (t, J = 7.0, 3H)], 0.29 (t, J = 7.0, 3H); ^{13}C NMR δ 171.75, [171.43, 170.25], 169.39, [144.75], 144.52, [137.96], 135.74, [135.25], 134.98, [133.98], 133.69, 132.29, [132.06], 130.98, [130.68, 129.74], 129.62, 126.89, [126.82], 125.09, [124.66], 124.35, 123.02, 122.93, [122.40, 121.47], 120.68, [119.85, 113.11], 113.02, 67.16, 61.55, [60.67, 60.04], 59.46. 40.57, 36.11, 35.11, 35.98, [35.92, 34.98, 34.80, 31.50], 24.28, [22.58], 21.63, [21.44], 21.37, [20.22, 18.63, 14.06], 13.95, [12.86], 12.28; IR 1728, 1246, 1197, 1178 cm^{-1}; MS *m/z* (relative intensity) 521 (M^+, 21), 441 (90), 369 (31), 212 (25), 155 (20), 91 (100); Anal. Calcd for $C_{29}H_{31}NO_6S$: C, 66.78; H, 5.99; N, 2.69. Found: C, 66.64; H, 6.02; N, 2.67.

1-((1*S,2*R**,3*R**,4*S**)-3-{1-tosyl-1H-indol-3-yl}bicyclo[2.2.2]oct-5-en-2-yl)ethanone (*endo*-11i).** According to the general cycloaddition procedure. FC (1:4, EtOAc:hexanes) afforded a white solid (165 mg, 78%, >95% de): mp 146-147 °C (DCM/hexanes); ^{1}H

(5) Only boiling point and combustion analysis data were reported previously for **11g**: Allen, C. F. H.; Bell, A.; Gates Jr., J. W. *J. Org. Chem.* **1943**, *8*, 373. Allen, C. F. H.; Bell, A. *J. Am. Chem. Soc.* **1939**, *61*, 521.

NMR δ 7.95 (dd, 8.2, 0.8, 1H), 7.70 (app d, J = 8.2, 2H), 7.51 (dd, 7.8, 0.8, 1H), 7.43 (s, 1H), 7.30 (ddd, J = 8.2, 7.4, 1.2, 1H), 7.22 (app t, J = 7.8, 1H), 7.19 (app d, J = 8.2, 2H), 6.45 (app t, J = 7.8, 7.0, 1H), 6.20 (app t, J = 7.4, 7.0, 1H) 3.30 (br d, J = 6.6, 1H), 3.00 (m, 1H), 2.85 (d, J = 6.6, 1H), 2.65 (m, 1H), 2.31 (s, 3H), 2.05 (s, 3H), 1.76-1.68 (m 1H), 1.53-1.46 (m, 1H), 1.43-1.35 (m, 1H), 1.03-0.96 (m, 1H); ^{13}C NMR δ 208.31, 144.89, 135.61, 135.20, 134.91, 131.31, 130.75, 129.78, 126.61, 124.95, 124.85, 123.16, 122.31, 119.65, 113.81, 56.02, 36.11, 34.42, 32.53, 28.32, 25.92, 21.49, 18.62; IR 1707, 1368, 1175 cm^{-1}; MS *m/z* (relative intensity) 419 (M$^+$, 7), 339 (52), 184 (67), 91 (100); Anal. Calcd for $C_{25}H_{25}NO_3S$: C, 71.57; H, 6.01; N, 3.34. Found: C, 71.51; H, 6.15; N, 3.33. Also isolated from the above FC was a colorless gum (~10 mg) found to contain approximately a 5:1 mixture of ***endo*-11i** and ***exo*-11i**: ^{1}H NMR (minor signals) δ 7.84 (d, 8.2), .6.23 (app t, J = 7.0), 3.61 (d, J = 5.5), 2.90 (m), 2.69 (m), 2.54 (m), 2.09 (s), 1.98 (s).

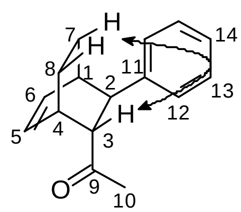


Figure 1. Numbering for ***endo*-9** and relevant NOESY correlation.

Table 1. ^{1}H and ^{13}C NMR Data for ***endo*-9** in $CDCl_3$ at 400 MHz.

Position	δC	δH, m, *J* in Hz	HMBC (H → C)[a]
1	32.33	3.00 m	3, 8
2	56.20	2.92 m	1, 6, 7, 9, 11
3	45.19	3.10 d 6.7	2, 4, 5, 8, 9, 11, 12
4	37.01	2.51 m	2
5	135.76	6.45 app t, 7.5, 7.1	1, 4
6	131.32	6.20 app t, 7.5, 6.7	1, 4
7	25.77	1.63-1.72 m	2, 3, 4, 5, 6[b]
		1.40-1.47 m	1, 2, 6, 8
8	18.17	1.63-1.72 m	2, 3, 4, 5, 6[b]
		0.99-1.05 m	3
9	208.64	-	-
10	28.16	2.01 s[c]	9
11	142.41	-	-
12	127.90		
13	128.34	7.22-7.35 m[d]	11, 12, 13, 14
14	126.18		

[a]Correlations for $^2J_{C-H}$ and $^3J_{C-H}$ couplings of 8 Hz. [b]Chemical shift similarities of H7 and H8 precluded differentiation of correlations. [c]3H. [d]5H.

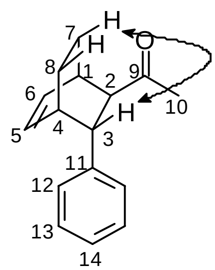


Figure 2. Numbering for ***exo*-9** and relevant NOESY correlation.

Table 2. ^{1}H and ^{13}C NMR Data for ***exo*-9** in $CDCl_3$.

Position	δC	δH (m, *J* in Hz)	HMBC (H → C)[a]
1	32.82	2.99 m	-
2	61.02	2.71 m	1, 3, 5, 7, 9, 11[b]
3	43.67	3.48 d 6.7	2, 4, 5, 8, 9, 11, 12
4	36.56	2.71 m	1, 3, 5, 7, 9, 11[b]
5	134.04	6.40 app t, 7.6, 7.2	1, 4
6	133.50	6.51 app t, 7.6, 6.7	1, 4
7	19.46	1.21-1.45 m	8
		1.51-1.58 m	-
8	26.42	1.27-1.35 m	7
		1.72-1.77 m	-
9	208.62	-	-
10	29.24	2.16 s[c]	9
11	146.78	-	-
12	125.88		
13	127.36	7.15-7.29 m[d]	3, 11, 12, 13, 14
14	128.13		

[a]Correlations for $^{2}J_{C\text{-}H}$ and $^{3}J_{C\text{-}H}$ couplings of 8 Hz. [b]Since chemical shifts of 2 and 4 are identical the correlations cannot be differentiated. [c]3H. [d]5H.

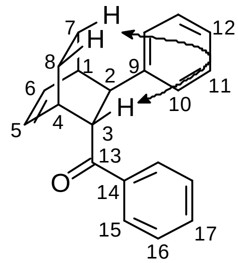


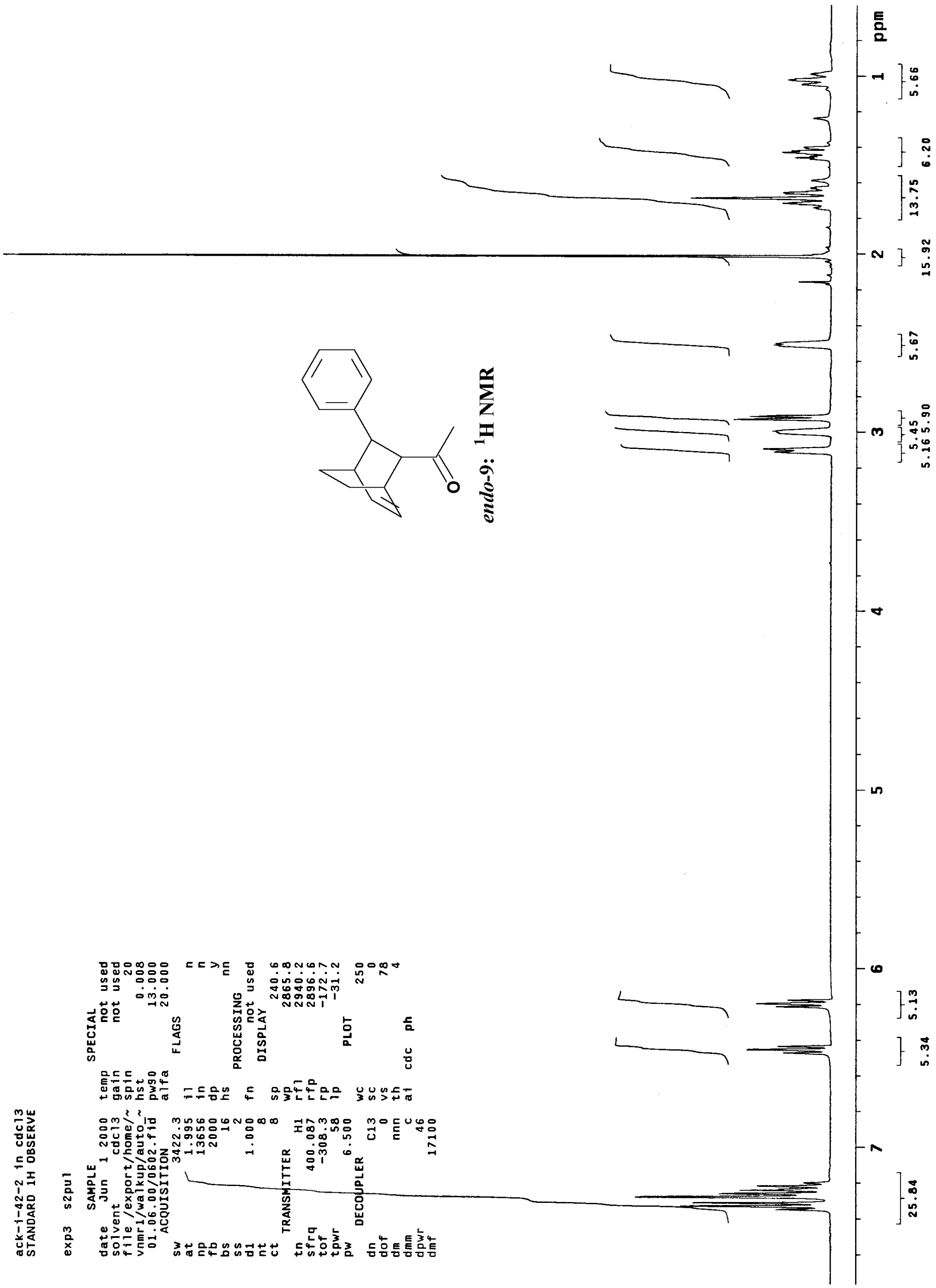
Figure 3. Numbering for ***endo*-11a** and selected NOESY correlations.

Table 3. ^{1}H and ^{13}C NMR Data for ***endo*-11a** in $CDCl_3$.

Position	δC	δH (m, *J* in Hz)	HMBC (H → C)[a]
1	34.50	2.96 m	3, 8
2	50.91	3.79 d 6.4	1, 6, 7, 9, 11
3	44.62	3.47 d 6.4	2, 4, 5, 8, 9, 11, 12
4	36.42	2.66 m	2
5	136.26	6.55 app t, 7.7, 6.8	1, 4
6	130.63	6.10 app t, 8.1, 6.8	1, 4
7	18.47	1.78-1.92 m	4, 5, 6[b]
		1.08-1.14 m	-
8	26.41	1.78-1.92 m	4, 5, 6[b]
		1.43-1.50 m	-
9	142.81	-	-
10	~128[d]	7.26-7.38 m	NA[e]
11	~128[d]	7.26-7.38 m	NA[e]
12	126.14	7.17-7.21 m	NA[e]
13	200.74	-	-
14	136.34	-	-
15	~128[d]	7.85 app d 7.7[c]	NA[e]
16	~128[d]	7.26-7.38 m	NA[e]
17	132.69	7.49	NA[e]

[a]Correlations for $^{2}J_{C\text{-}H}$ and $^{3}J_{C\text{-}H}$ couplings of 8 Hz. [b]Since chemical shifts of 7 and 8 are identical the correlations cannot be differentiated. [c]2H. [d]Exact assignment was precluded by chemical shift similarities. [e]Assignment of correlations was precluded by the complexity of the spectrum.

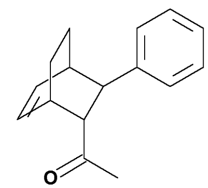
Appendix I: Spectroscopic Data



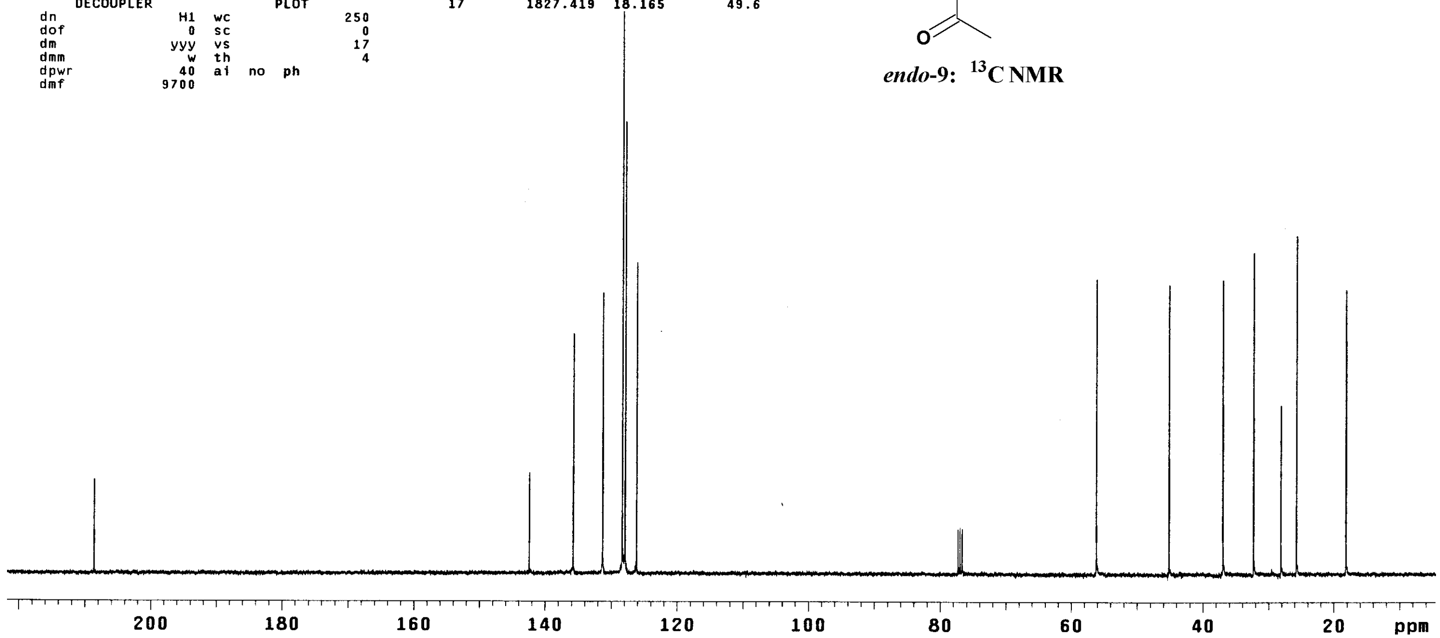
endo-9: ^{1}H NMR

```
ack-1-42-2 in cdcl3
13C OBSERVE

pad=2 run with gradshim before acquisiti
on

exp3  s2pul

      SAMPLE             SPECIAL
date   Jun  1 2000  temp       not used
solvent      cdcl3  gain       not used
file /export/home/~ spin             20
vnmr1/walkup/auto_~ hst           0.008
 01.06.00/0701.fid  pw90         11.700
   ACQUISITION      alfa         20.000
sw         25125.6          FLAGS
at           1.199  il                n
np           60270  in                n
fb           13800  dp                y
bs              64  hs               nn
ss               2        PROCESSING
d1           1.000  lb             1.00
nt             256  fn         not used
ct              64         DISPLAY
   TRANSMITTER      sp            454.1
tn             C13  wp          21853.7
sfrq       100.613  rfl          9287.4
tof         1550.9  rfp          7746.3
tpwr            59  rp            102.5
pw           5.850  lp           -288.4
    DECOUPLER               PLOT
dn              H1  wc              250
dof              0  sc                0
dm             yyy  vs               17
dmm              w  th                4
dpwr            40  ai   no  ph
dmf           9700
```

INDEX	FREQUENCY	PPM	HEIGHT
1	20989.669	208.642	16.3
2	14326.972	142.413	17.5
3	13657.559	135.759	41.8
4	13210.516	131.315	49.0
5	12911.465	128.343	98.4
6	12866.991	127.900	79.0
7	12693.695	126.178	54.2
8	7778.528	77.320	7.7
9	7746.323	77.000	8.0
10	7714.117	76.680	7.9
11	5653.735	56.199	51.4
12	4545.713	45.185	50.4
13	3723.707	37.014	51.3
14	3252.894	32.334	56.1
15	2832.689	28.157	29.4
16	2592.682	25.772	59.1
17	1827.419	18.165	49.6



endo-9: ^{13}C NMR



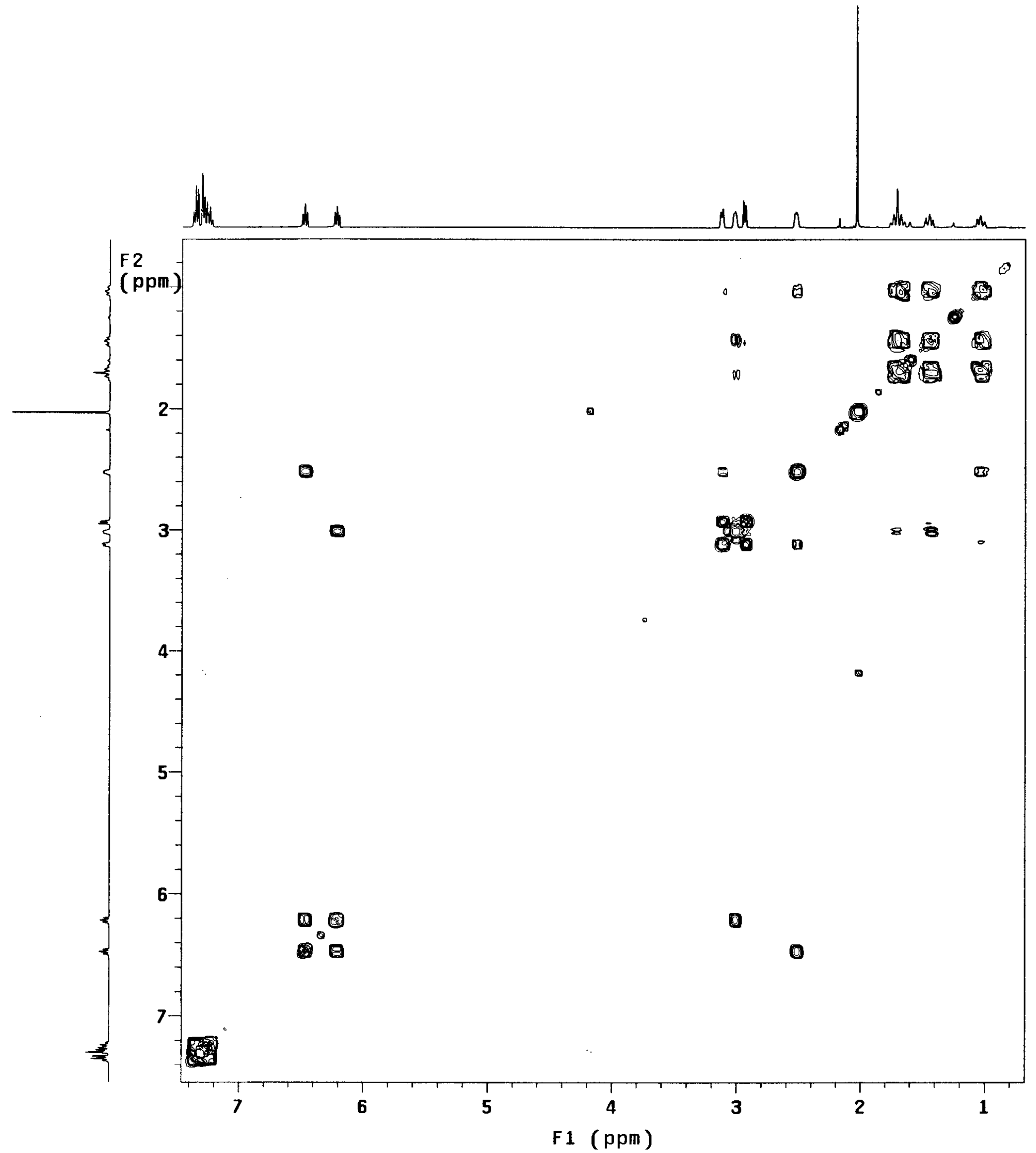
ack-i-42-2 in cdcl3
STANDARD 1H OBSERVE

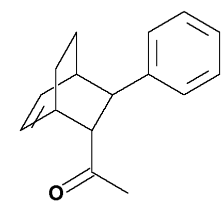
Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory: auto_01Jun2000-19:11:48

Pulse Sequence: gCOSY

Solvent: cdcl3
Ambient temperature
File: 0603
Mercury-400BB "nmrm400"

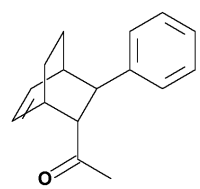
Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 3422.3 Hz
2D Width 3422.3 Hz
Single scan
128 increments
OBSERVE H1, 400.0854801 MHz
DATA PROCESSING
Sq. sine bell 0.075 sec
F1 DATA PROCESSING
Sq. sine bell 0.037 sec
FT size 1024 x 1024
Total time 3 min, 36 sec



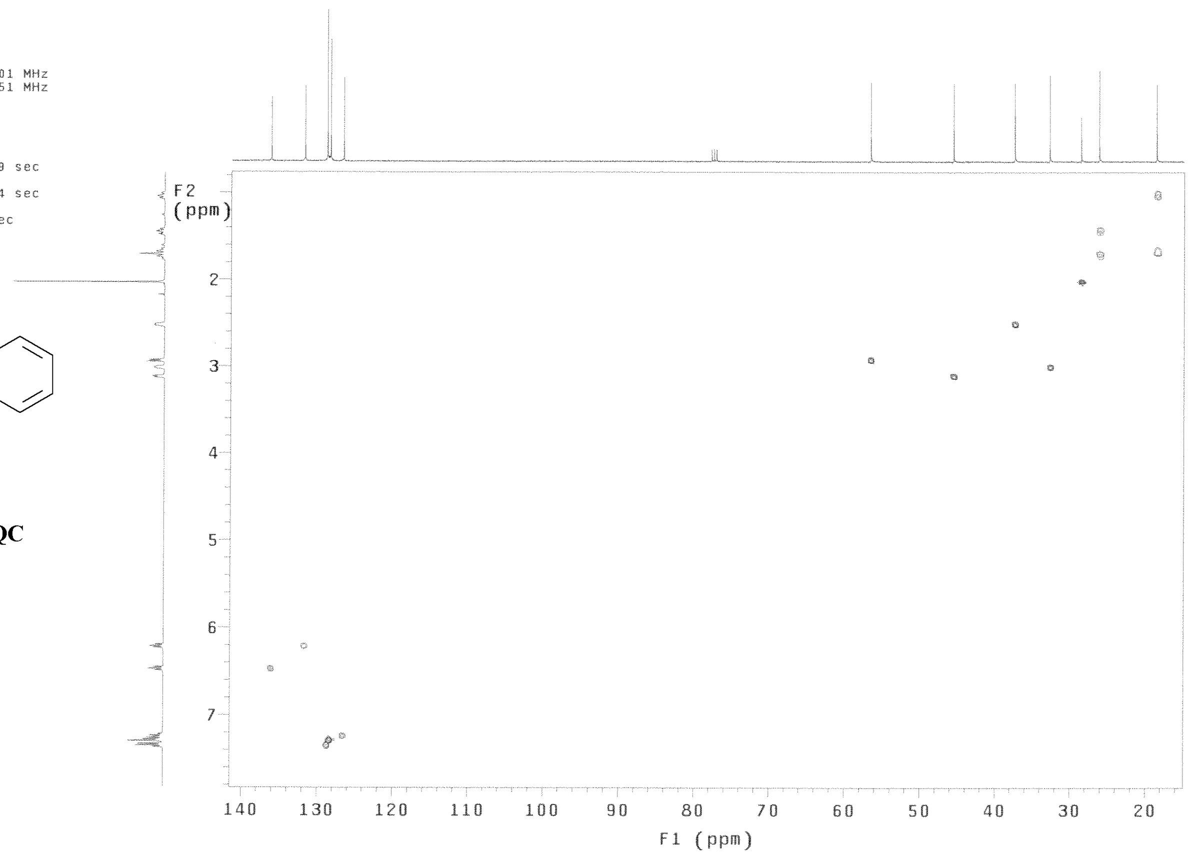


endo-9: **gCOSY**

```
ack-i-42-2 in cdcl3
STANDARD 1H OBSERVE

 Archive directory: /export/home/vnmr1/vnmrsys/data
 Sample directory: auto_01Jun2000-19:11:48

Pulse Sequence: gHSQC

 Solvent: cdcl3
 Ambient temperature
File: 0604
Mercury-400BB  "nmrm400"

 Relax. delay 1.000 sec
 Acq. time 0.150 sec
 Width    3422.3 Hz
 2D Width 17094.0 Hz
 4 repetitions
 2 x 128 increments
OBSERVE   H1, 400.0854801 MHz
DECOUPLE C13, 100.6090951 MHz
 Power 46 dB
 on during acquisition
 off during delay
 GARP-1 modulated
DATA PROCESSING
 Gauss apodization 0.069 sec
F1 DATA PROCESSING
 Gauss apodization 0.014 sec
FT size 1024 x 2048
Total time 23 min, 19 sec
```



***endo*-9: gHSQC**



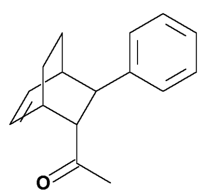
ack-i-40-2 in cdcl3
STANDARD 1H OBSERVE

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory: auto_14Jun2000-19:27:47

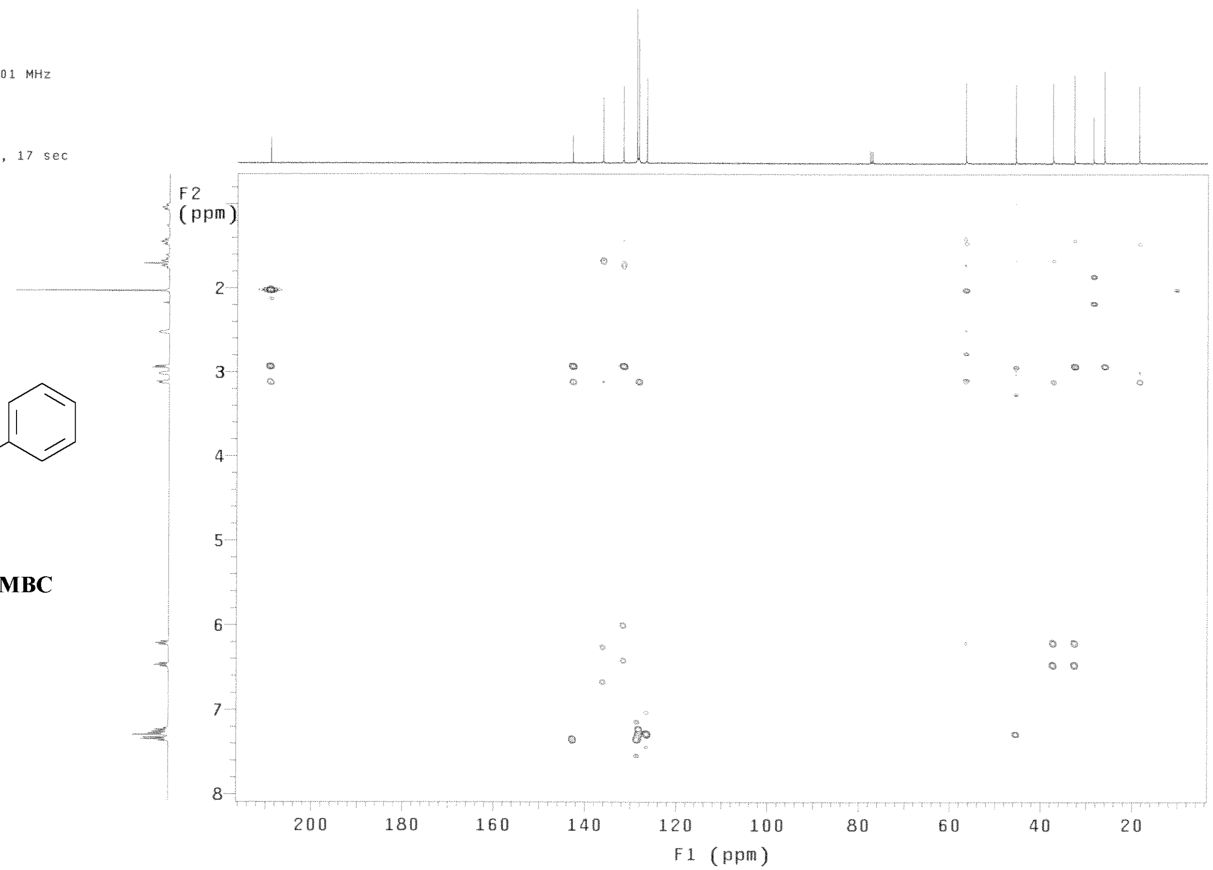
Pulse Sequence: gHMBC

Solvent: cdcl3
Ambient temperature
File: 1007
Mercury-400BB "nmrm400"

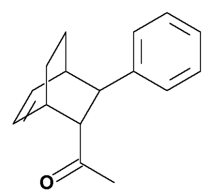
Relax. delay 1.000 sec
Acq. time 0.153 sec
Width 3352.3 Hz
2D Width 24154.6 Hz
8 repetitions
400 increments
OBSERVE H1, 400.0854801 MHz
DATA PROCESSING
Sine bell 0.076 sec
F1 DATA PROCESSING
Sine bell 0.008 sec
FT size 1024 x 2048
Total time 1 hr, 10 min, 17 sec



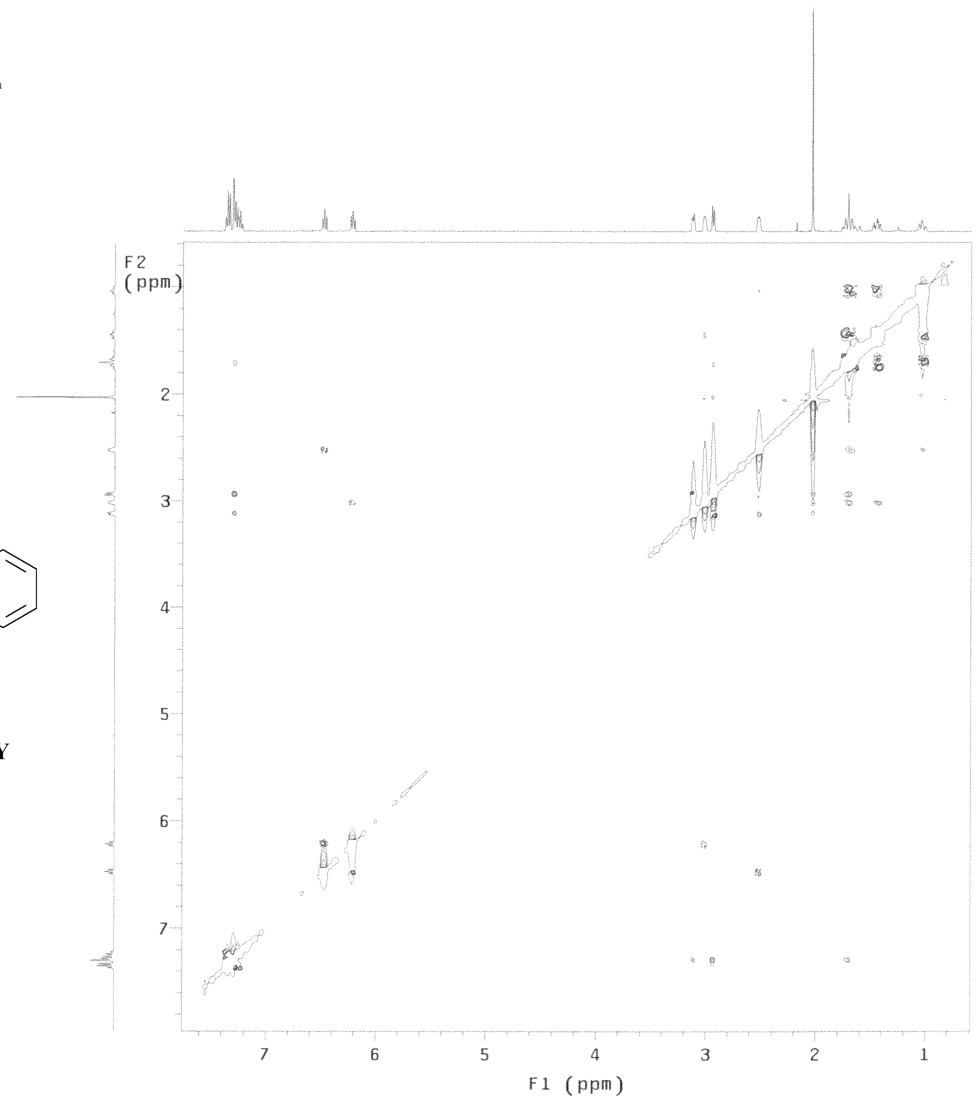
***endo*-9: gHMBC**

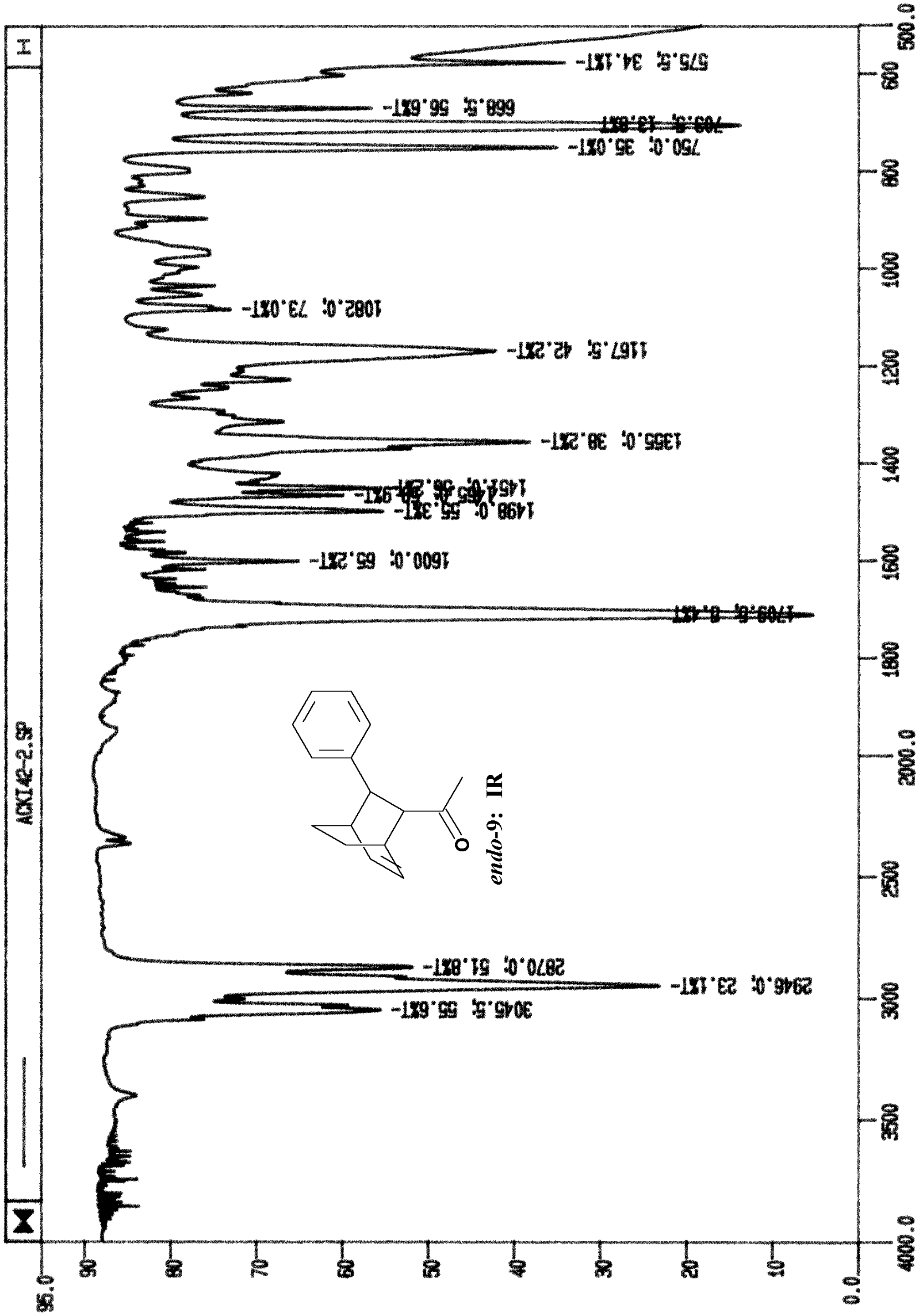


```
ack-i-40-2 in cdcl3
STANDARD 1H OBSERVE

 Archive directory: /export/home/vnmr1/vnmrsys/data
 Sample directory: auto_14Jun2000-19:27:47

Pulse Sequence: NOESY

 Solvent: cdcl3
 Ambient temperature
File: 1006
Mercury-400BB  "nmrm400"

 Relax. delay 1.000 sec
 Mixing 0.200 sec
 Acq. time 0.153 sec
 Width    3352.3 Hz
 2D Width 3352.3 Hz
 16 repetitions
 2 x 200 increments
OBSERVE   H1, 400.0854801 MHz
DATA PROCESSING
 Gauss apodization 0.071 sec
F1 DATA PROCESSING
 Gauss apodization 0.055 sec
FT size 2048 x 2048
Total time 2 hr, 34 min, 10 sec
```

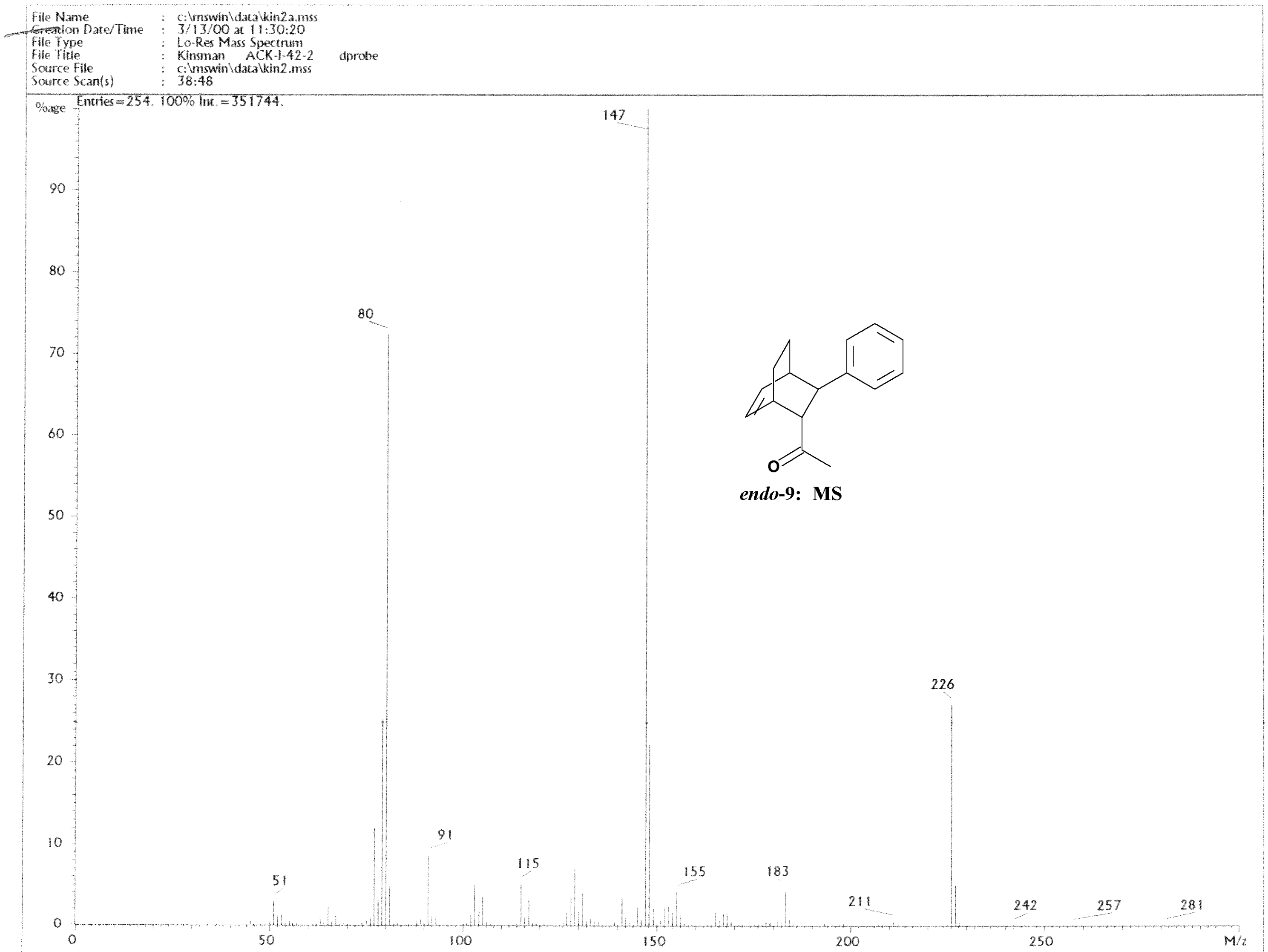


***endo*-9: NOESY**




ACK142-2.SP
1709.6; 6.4%T
2946.0; 23.1%T
2870.0; 51.8%T
3045.5; 55.6%T
1600.0; 65.2%T
1498.0; 55.3%T
1355.0; 38.2%T
1167.5; 42.2%T
1082.0; 73.0%T
750.0; 35.0%T
668.5; 56.6%T
575.5; 34.1%T

endo-9: **IR**


File Name : c:\mswin\data\kin2a.mss
Creation Date/Time : 3/13/00 at 11:30:20
File Type : Lo-Res Mass Spectrum
File Title : Kinsman ACK-I-42-2 dprobe
Source File : c:\mswin\data\kin2.mss
Source Scan(s) : 38:48
Entries=254. 100% Int.=351744.
%age
90
80
70
60
50
40
30
20
10
0
50
100
150
200
250
M/z
147
80
226
91
115
51
155
183
211
242
257
281
O

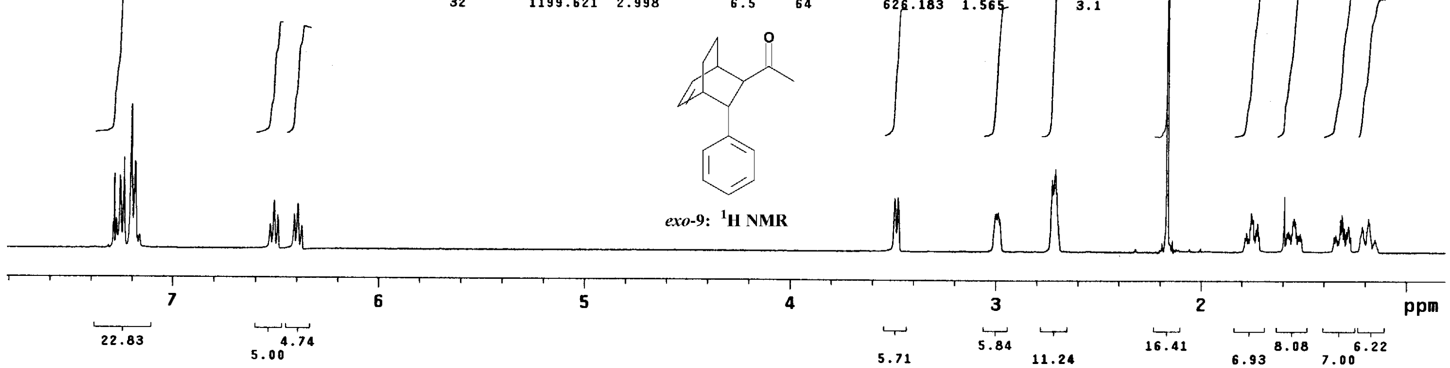
endo-9: **MS**

ack-i-40-4 in cdcl3
STANDARD 1H OBSERVE

exp2 s2pul

SAMPLE		SPECIAL	
date	Jun 7 2000	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~	spin	20
vnmr1/walkup/auto_~		hst	0.008
07.06.00/0211.fid		pw90	13.000
ACQUISITION		alfa	20.000
sw	3672.4	FLAGS	
at	1.995	il	n
np	14654	in	n
fb	2000	dp	y
bs	16	hs	nn
ss	2	PROCESSING	
d1	1.000	fn	not used
nt	8	DISPLAY	
ct	8	sp	323.5
TRANSMITTER		wp	2800.8
tn	H1	rfl	3247.8
sfrq	400.087	rfp	2896.6
tof	-509.5	rp	-170.1
tpwr	58	lp	-33.2
pw	6.500	PLOT	
DECOUPLER		wc	250
dn	C13	sc	0
dof	0	vs	86
dm	nnn	th	1
dmm	c	ai	cdc ph
dpwr	46		
dmf	17100		

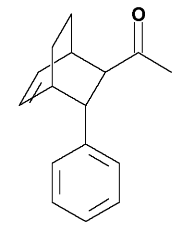
INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	2918.140	7.294	4.5	33	1198.276	2.995	6.6	65	623.045	1.557	3.3
2	2916.346	7.289	12.1	34	1197.379	2.993	6.6	66	620.355	1.551	5.5
3	2915.449	7.287	12.8	35	1195.137	2.987	6.8	67	618.562	1.546	5.8
4	2911.863	7.278	5.2	36	1194.241	2.985	6.2	68	616.320	1.540	5.4
5	2910.966	7.276	4.9	37	1192.896	2.982	5.7	69	613.630	1.534	3.5
6	2909.173	7.271	2.7	38	1191.999	2.979	5.1	70	610.491	1.526	2.9
7	2904.689	7.260	12.5	39	1190.654	2.976	3.6	71	608.250	1.520	3.0
8	2903.344	7.257	11.6	40	1090.672	2.726	12.5	72	606.008	1.515	3.3
9	2902.447	7.255	8.0	41	1087.982	2.719	11.4	73	603.766	1.509	2.5
10	2898.861	7.246	7.4	42	1086.189	2.715	13.1	74	540.549	1.351	2.7
11	2896.619	7.240	15.6	43	1083.947	2.709	14.4	75	537.411	1.343	3.1
12	2882.272	7.204	24.8	44	1082.154	2.705	10.3	76	532.927	1.332	2.1
13	2880.478	7.200	12.2	45	876.810	2.192	1.6	77	528.444	1.321	5.7
14	2875.546	7.187	15.0	46	867.843	2.169	36.6	78	525.305	1.313	6.6
15	2874.650	7.185	14.8	47	865.153	2.162	97.5	79	520.822	1.302	4.2
16	2873.305	7.182	9.8	48	856.186	2.140	2.1	80	516.338	1.291	3.6
17	2870.615	7.175	1.8	49	714.060	1.785	2.6	81	513.200	1.283	4.5
18	2869.718	7.173	2.1	50	710.921	1.777	3.4	82	509.165	1.273	2.7
19	2867.028	7.166	2.2	51	707.783	1.769	2.5	83	485.851	1.214	4.5
20	2866.131	7.164	2.3	52	704.196	1.760	4.4	84	484.057	1.210	4.0
21	2863.889	7.158	1.2	53	701.506	1.753	6.8	85	480.470	1.201	2.0
22	2612.366	6.530	4.3	54	698.368	1.746	5.7	86	473.745	1.184	6.0
23	2604.744	6.510	8.3	55	695.677	1.739	2.9	87	471.952	1.180	5.2
24	2597.571	6.493	5.8	56	692.539	1.731	3.9	88	461.191	1.153	2.3
25	2565.738	6.413	6.0	57	689.401	1.723	5.0	89	459.398	1.148	1.9
26	2559.013	6.396	7.8	58	686.262	1.715	2.8				
27	2551.391	6.377	4.0	59	639.634	1.599	3.4				
28	1397.342	3.493	9.3	60	636.944	1.592	9.6				
29	1390.617	3.476	9.3	61	632.909	1.582	3.3				
30	1201.863	3.004	6.0	62	630.667	1.576	3.5				
31	1200.966	3.002	6.5	63	628.425	1.571	3.7				
32	1199.621	2.998	6.5	64	626.183	1.565	3.1				



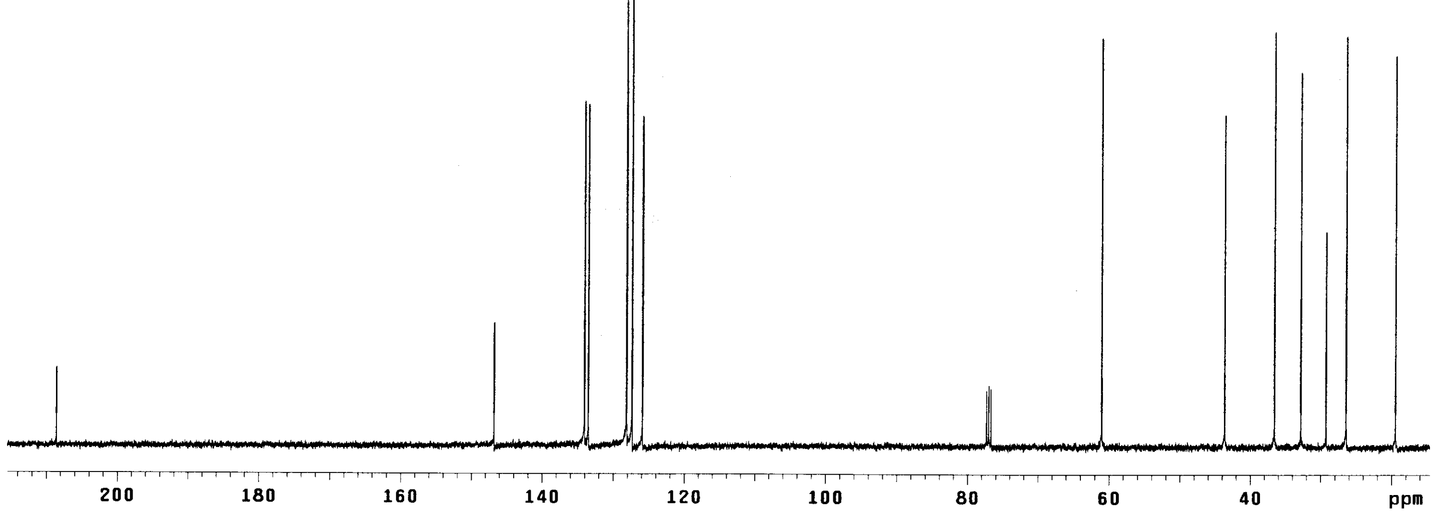
exo-9: ^{1}H NMR

```
ack-i-40-4 in cdcl3
13C OBSERVE

pad=2 run with gradshim before acquisiti
on

exp3  s2pul

      SAMPLE              SPECIAL
date   Jun  8 2000  temp       not used
solvent      cdcl3  gain       not used
file /export/home/~ spin             20
vnmr1/walkup/auto_~ hst           0.008
 08.06.00/0306.fid  pw90         11.700
   ACQUISITION      alfa         20.000
sw         25125.6          FLAGS
at           1.199  il                n
np           60270  in                n
fb           13800  dp                y
bs              64  hs               nn
ss               2       PROCESSING
d1           1.000  lb             1.00
nt             256  fn         not used
ct              64         DISPLAY
   TRANSMITTER      sp           1467.0
tn             C13  wp          20209.7
sfrq       100.613  rfl          9282.8
tof         1550.9  rfp          7746.3
tpwr            59  rp            115.5
pw           5.850  lp           -292.3
    DECOUPLER              PLOT
dn              H1  wc              250
dof              0  sc                0
dm             yyy  vs               28
dmm              w  th                7
dpwr            40  ai  no  ph
dmf           9700
```

INDEX	FREQUENCY	PPM	HEIGHT
1	20987.368	208.619	13.5
2	14766.346	146.780	21.5
3	13484.262	134.036	60.4
4	13429.820	133.495	59.8
5	12889.995	128.129	126.0
6	12812.548	127.359	116.9
7	12664.557	125.888	57.7
8	7777.761	77.313	9.8
9	7746.322	77.000	10.7
10	7714.117	76.680	10.1
11	6138.350	61.016	71.7
12	4393.120	43.668	58.1
13	3677.699	36.557	72.9
14	3301.202	32.815	65.7
15	2941.574	29.240	37.6
16	2657.859	26.420	72.1
17	1957.774	19.461	68.7



exo-9: ^{13}C NMR



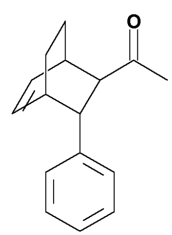
ack-i-40-4 in cdcl3
STANDARD 1H OBSERVE

Automation directory: /export/home/vnmr1/walkup/auto_07.06.00
File : 0213

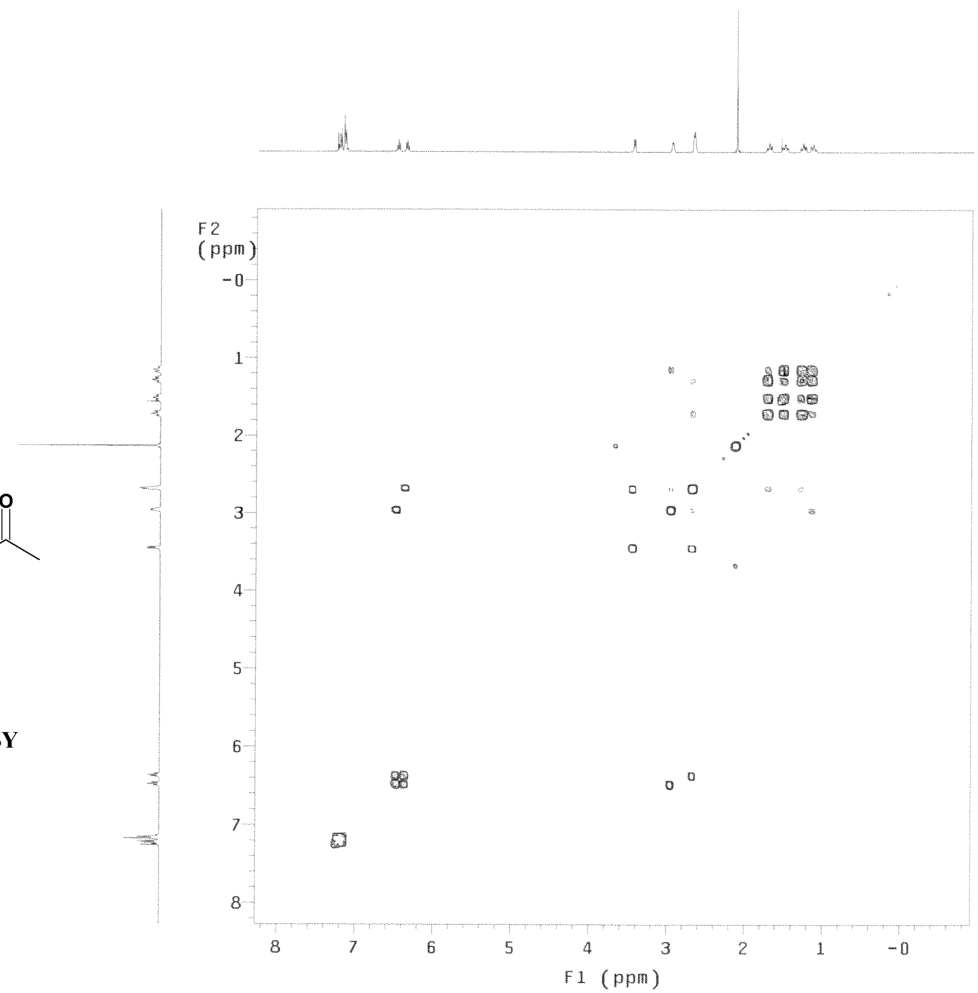
Pulse Sequence: gCOSY

Solvent: cdcl3
Ambient temperature
Sample #2
File: 0212
Mercury-400BB "nmrm400"

Relax. delay 1.000 sec
Acq. time 0.139 sec
Width 3672.4 Hz
2D Width 3672.4 Hz
Single scan
128 increments
OBSERVE H1, 400.0854801 MHz
DATA PROCESSING
Sq. sine bell 0.070 sec
F1 DATA PROCESSING
Sq. sine bell 0.035 sec
FT size 1024 x 1024
Total time 3 min, 34 sec



exo-9: **gCOSY**



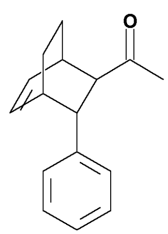
ack-i-40-4 in cdcl3
STANDARD 1H OBSERVE

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory: auto_13Jun2000

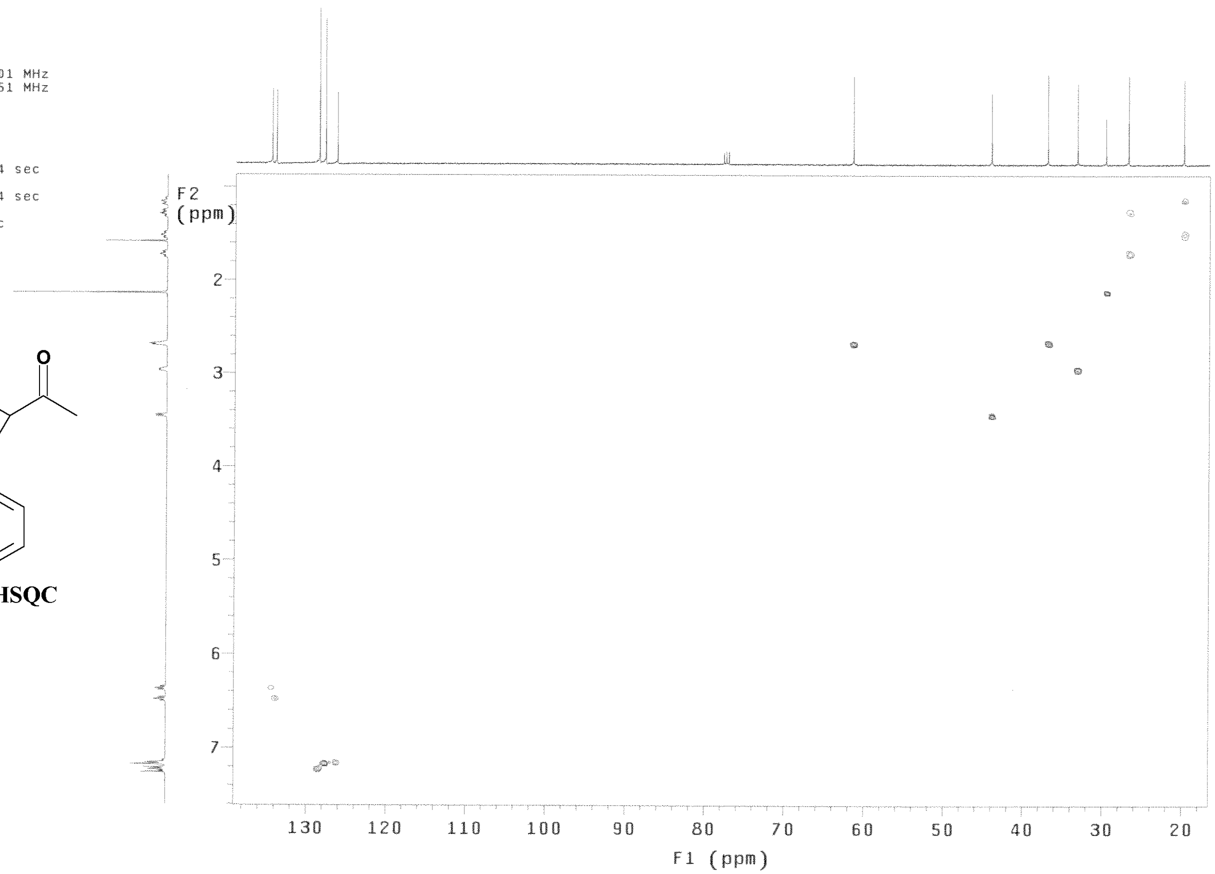
Pulse Sequence: gHSQC

Solvent: cdcl3
Ambient temperature
File: 0603
Mercury-400BB "nmrm400"

Relax. delay 1.000 sec
Acq. time 0.139 sec
Width 3672.4 Hz
2D Width 17094.0 Hz
4 repetitions
2 x 128 increments
OBSERVE H1, 400.0854801 MHz
DECOUPLE C13, 100.6090951 MHz
Power 46 dB
on during acquisition
off during delay
GARP-1 modulated
DATA PROCESSING
Gauss apodization 0.064 sec
F1 DATA PROCESSING
Gauss apodization 0.014 sec
FT size 1024 x 2048
Total time 23 min, 9 sec



exo-9: **gHSQC**



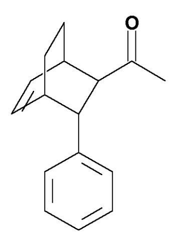
ack-i-40-4 in cdcl3
STANDARD 1H OBSERVE

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory: auto_08Jun2000

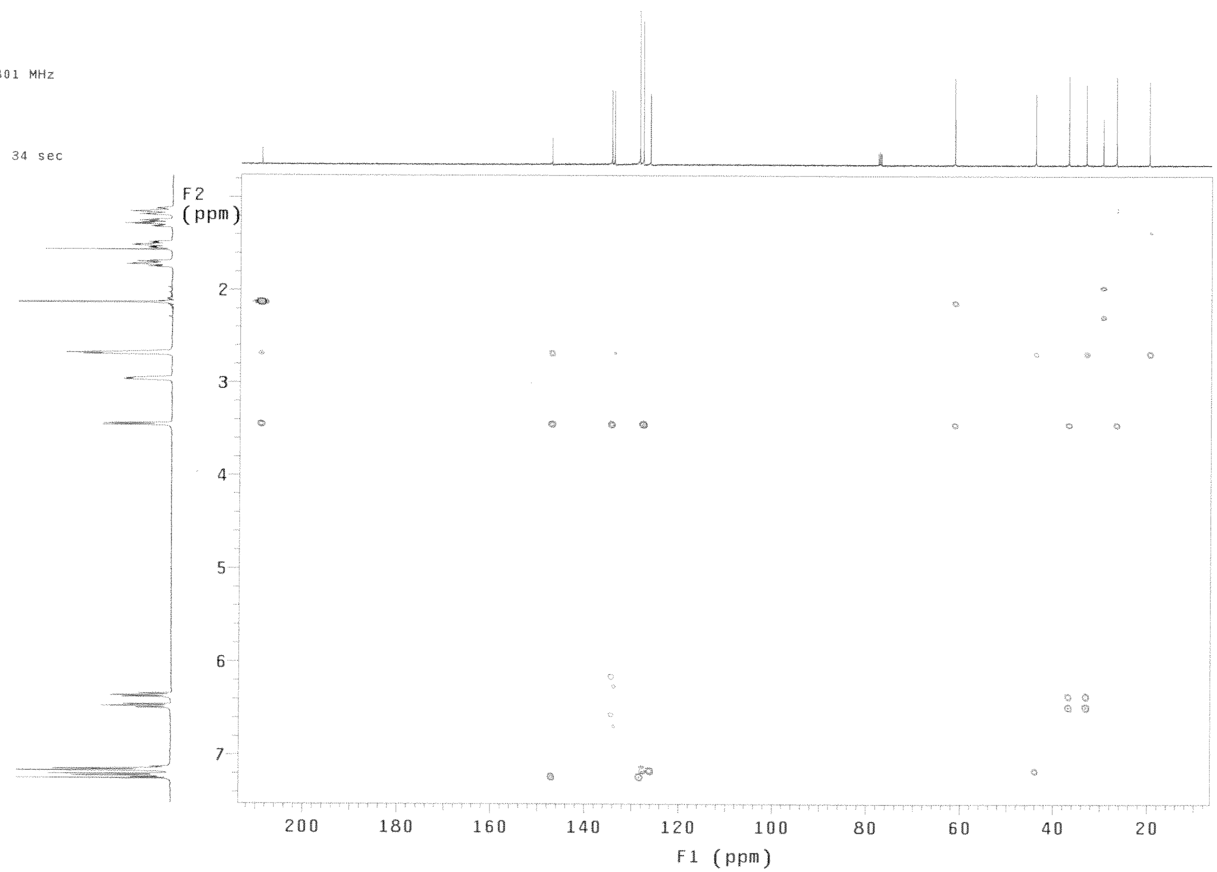
Pulse Sequence: gHMBC

Solvent: cdcl3
Ambient temperature
File: 0801
Mercury-400BB "nmrm400"

Relax. delay 1.000 sec
Acq. time 0.140 sec
Width 3665.7 Hz
2D Width 24154.6 Hz
8 repetitions
400 increments
OBSERVE H1, 400.0854801 MHz
DATA PROCESSING
Sine bell 0.070 sec
F1 DATA PROCESSING
Sine bell 0.008 sec
FT size 1024 x 2048
Total time 1 hr, 9 min, 34 sec



***exo*-9: gHMBC**



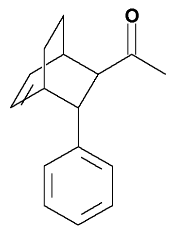
ack-i-40-4 in cdcl3
STANDARD 1H OBSERVE

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory: auto_08Jun2000

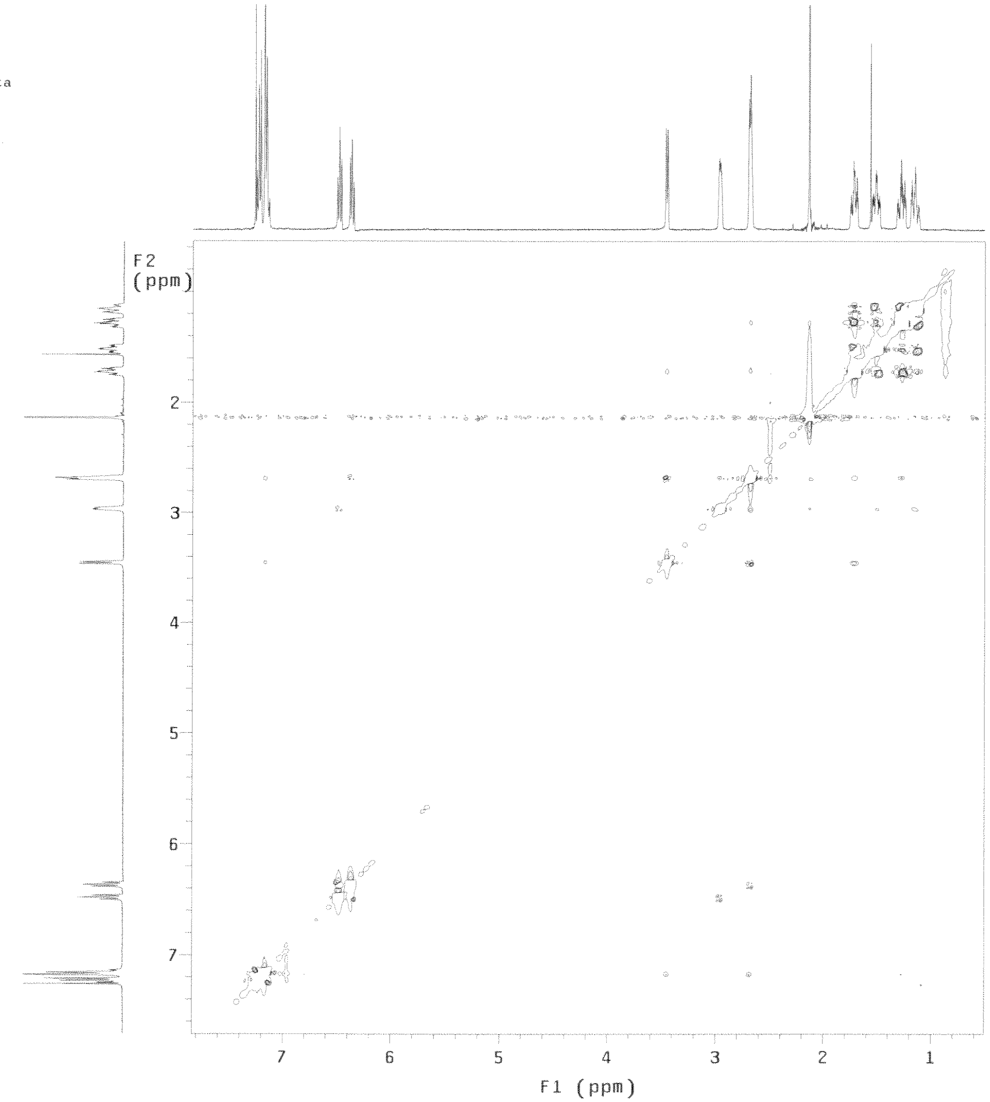
Pulse Sequence: NOESY

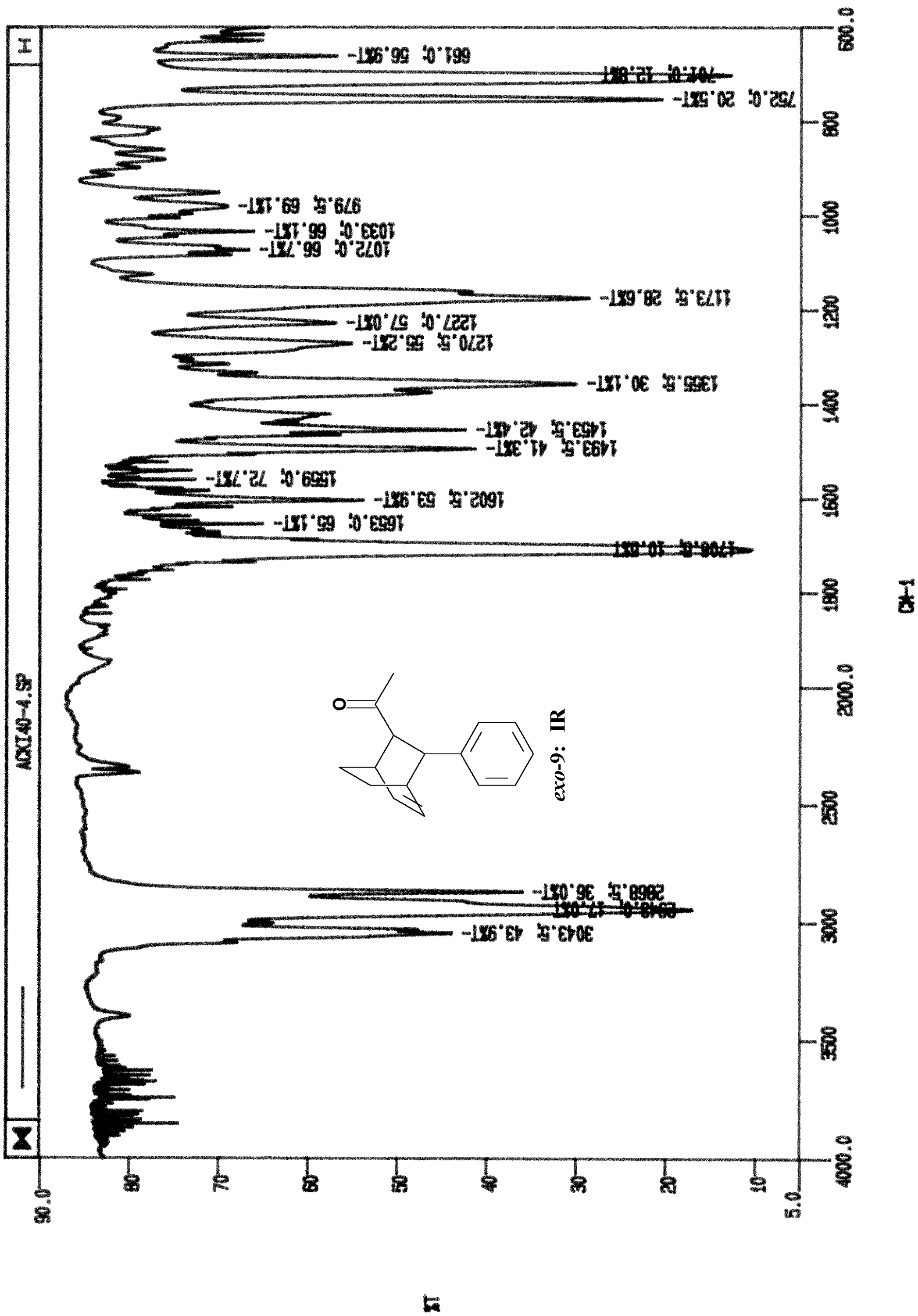
Solvent: cdcl3
Ambient temperature
File: 0803
Mercury-400BB "nmrm400"

Relax. delay 1.000 sec
Mixing 0.200 sec
Acq. time 0.140 sec
Width 3665.7 Hz
2D Width 3665.7 Hz
16 repetitions
2 x 200 increments
OBSERVE H1, 400.0854801 MHz
DATA PROCESSING
Gauss apodization 0.065 sec
F1 DATA PROCESSING
Gauss apodization 0.050 sec
FT size 2048 x 2048
Total time 2 hr, 32 min, 30 sec

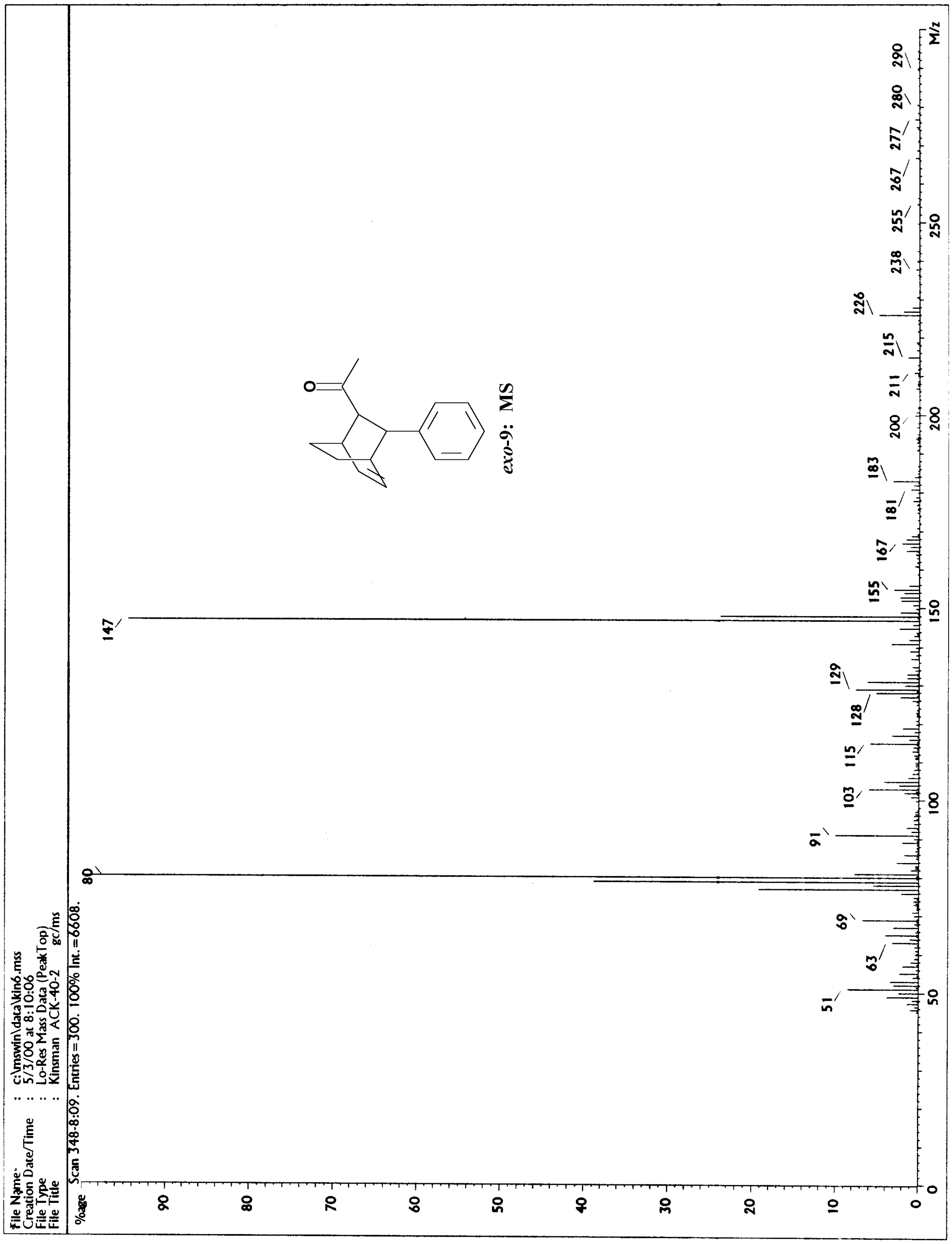


***exo*-9: NOESY**

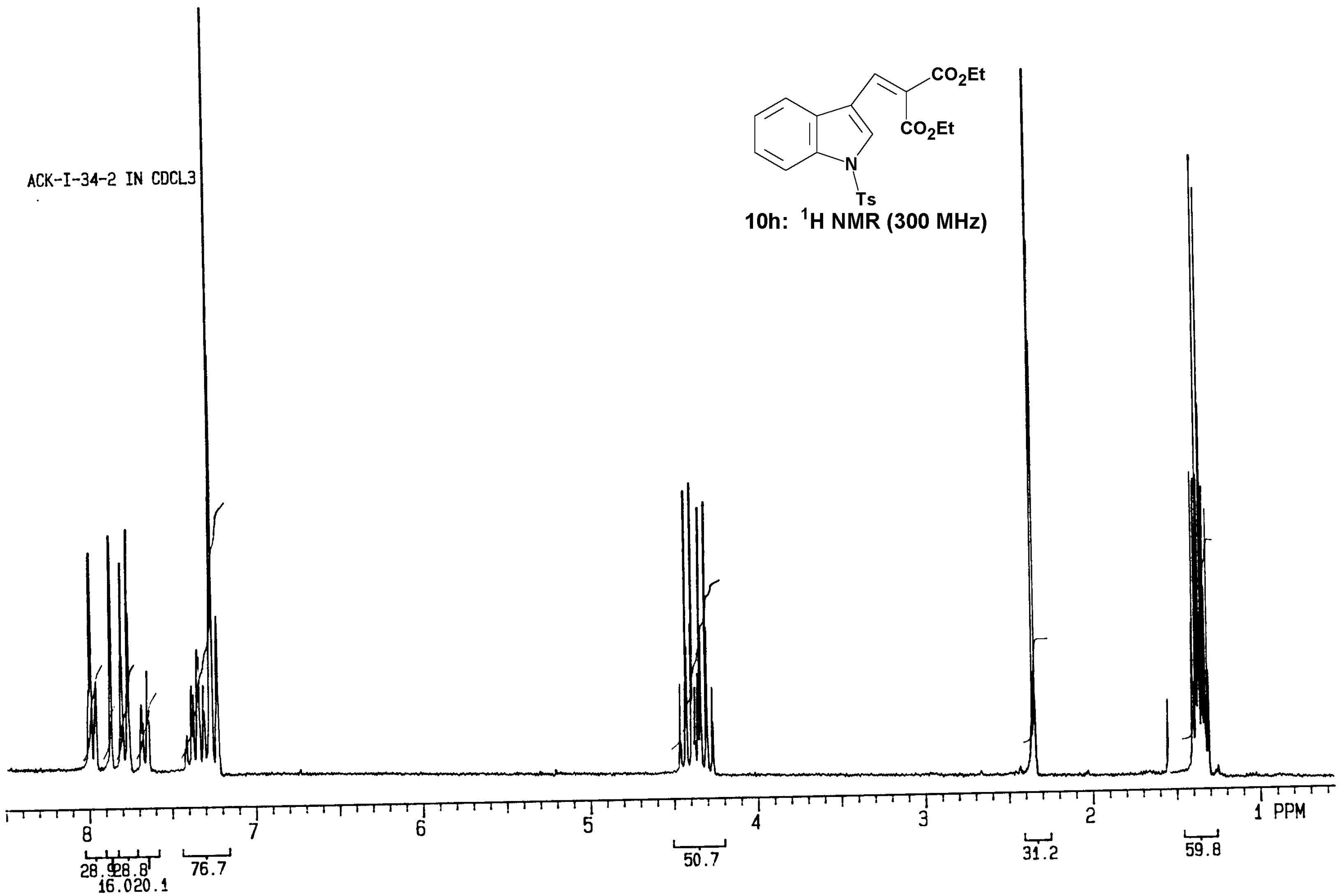




ACKI40-4.SP
exo-9: IR
%T
CM-1


File Name : c:\mswin\data\kin6.mss
Creation Date/Time : 5/3/00 at 8:10:06
File Type : Lo-Res Mass Data (PeakTop)
File Title : Kinsman ACK-40-2 gc/ms
Scan 348-8:09. Entries=300. 100% Int.=6608.
%age
M/z
80
147
51
63
69
91
103
115
128
129
155
167
181
183
200
211
215
226
238
255
267
277
280
290
O

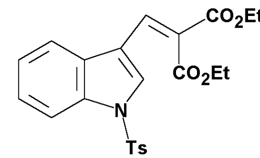
exo-9: MS



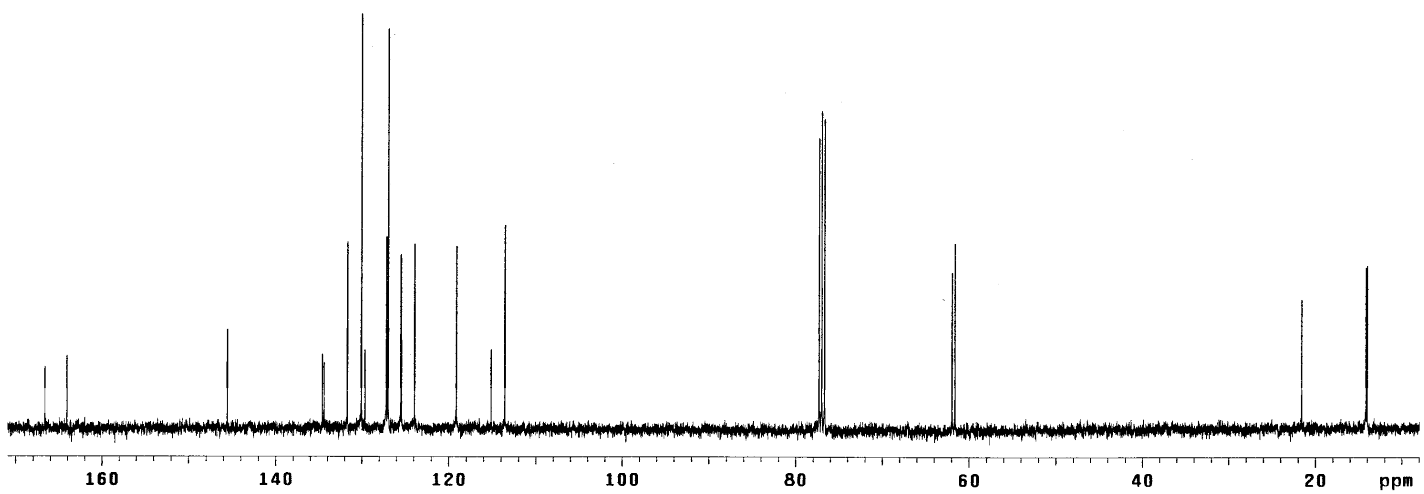
10h: ^{1}H NMR (300 MHz)

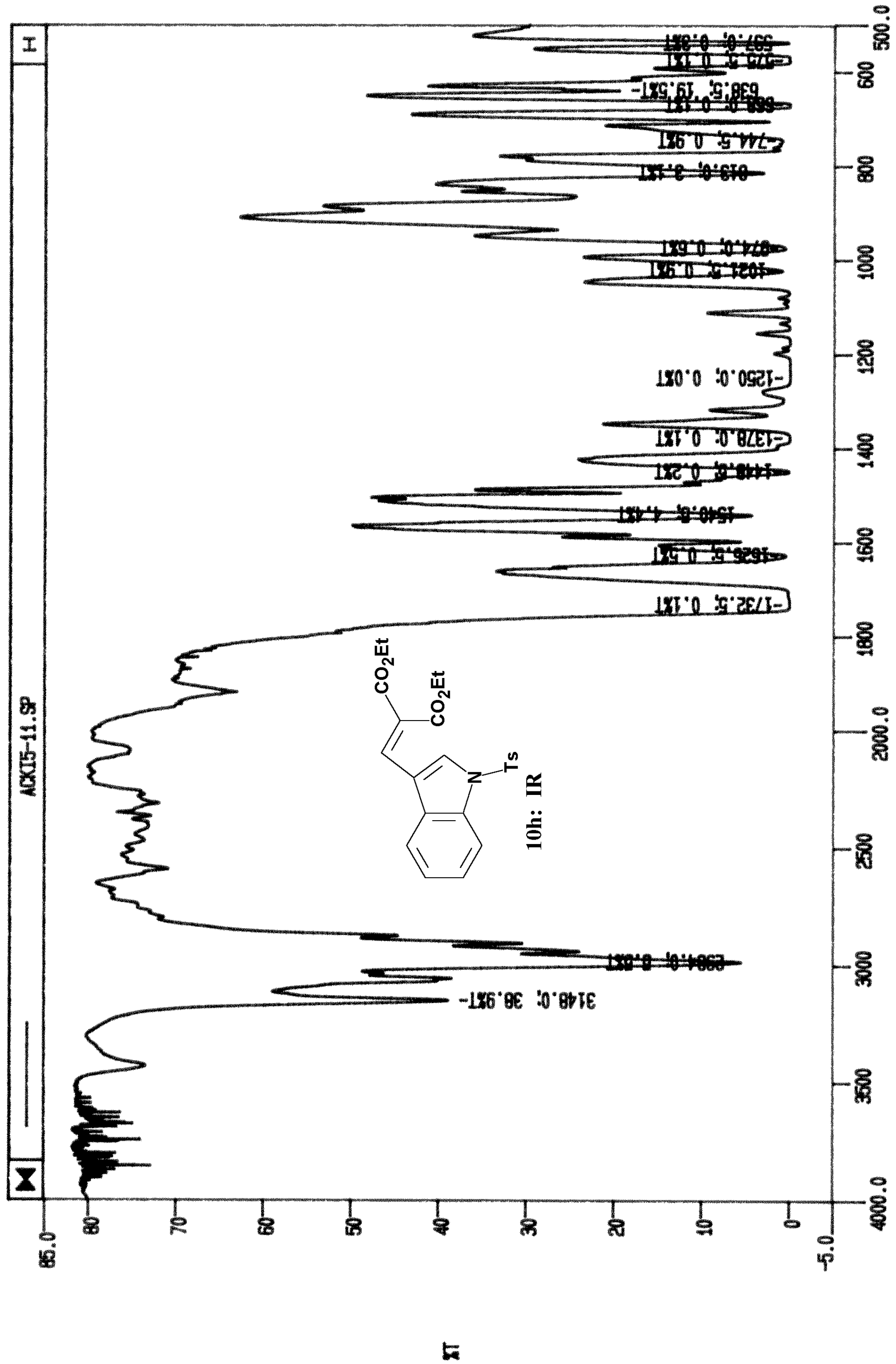
```
ack-1-34-2 in cdcl3
13C OBSERVE

pad=2 run with gradshim before acquisiti
on

exp3  s2pul

     SAMPLE              SPECIAL
date   Jun  6 2000  temp       not used
solvent      cdcl3  gain       not used
file /export/home/~ spin             20
vnmr1/walkup/auto_~ hst           0.008
 06.06.00/0213.fid  pw90         11.700
   ACQUISITION      alfa         20.000
sw         25125.6           FLAGS
at           1.199  il                n
np           60270  in                n
fb           13800  dp                y
bs              64  hs               nn
ss               2         PROCESSING
d1           1.000  lb             1.00
nt             256  fn         not used
ct             256          DISPLAY
  TRANSMITTER       sp            795.3
tn             C13  wp          16401.8
sfrq       100.613  rfl          9270.6
tof         1550.9  rfp          7746.3
tpwr            59  rp            109.7
pw           5.850  lp           -318.1
   DECOUPLER                 PLOT
dn              H1  wc              250
dof              0  sc                0
dm             yyy  vs               55
dmm              w  th                8
dpwr            40  ai   no  ph
dmf           9700
```

INDEX	FREQUENCY	PPM	HEIGHT
1	16764.618	166.644	10.9
2	16508.508	164.098	12.9
3	14645.192	145.576	17.6
4	13542.538	134.616	13.2
5	13521.834	134.410	11.7
6	13250.388	131.712	33.0
7	13085.527	130.073	73.5
8	13042.586	129.646	14.0
9	12792.611	127.161	34.0
10	12774.208	126.978	70.8
11	12625.449	125.500	30.8
12	12617.781	125.423	15.7
13	12467.489	123.929	32.7
14	11984.407	119.127	32.2
15	11584.139	115.149	14.1
16	11426.946	113.586	36.0
17	7778.527	77.320	51.4
18	7746.321	77.000	56.2
19	7714.116	76.680	54.8
20	6231.898	61.946	27.6
21	6198.159	61.611	32.8
22	2171.709	21.587	22.9
23	1424.083	14.156	28.6
24	1410.280	14.018	28.9



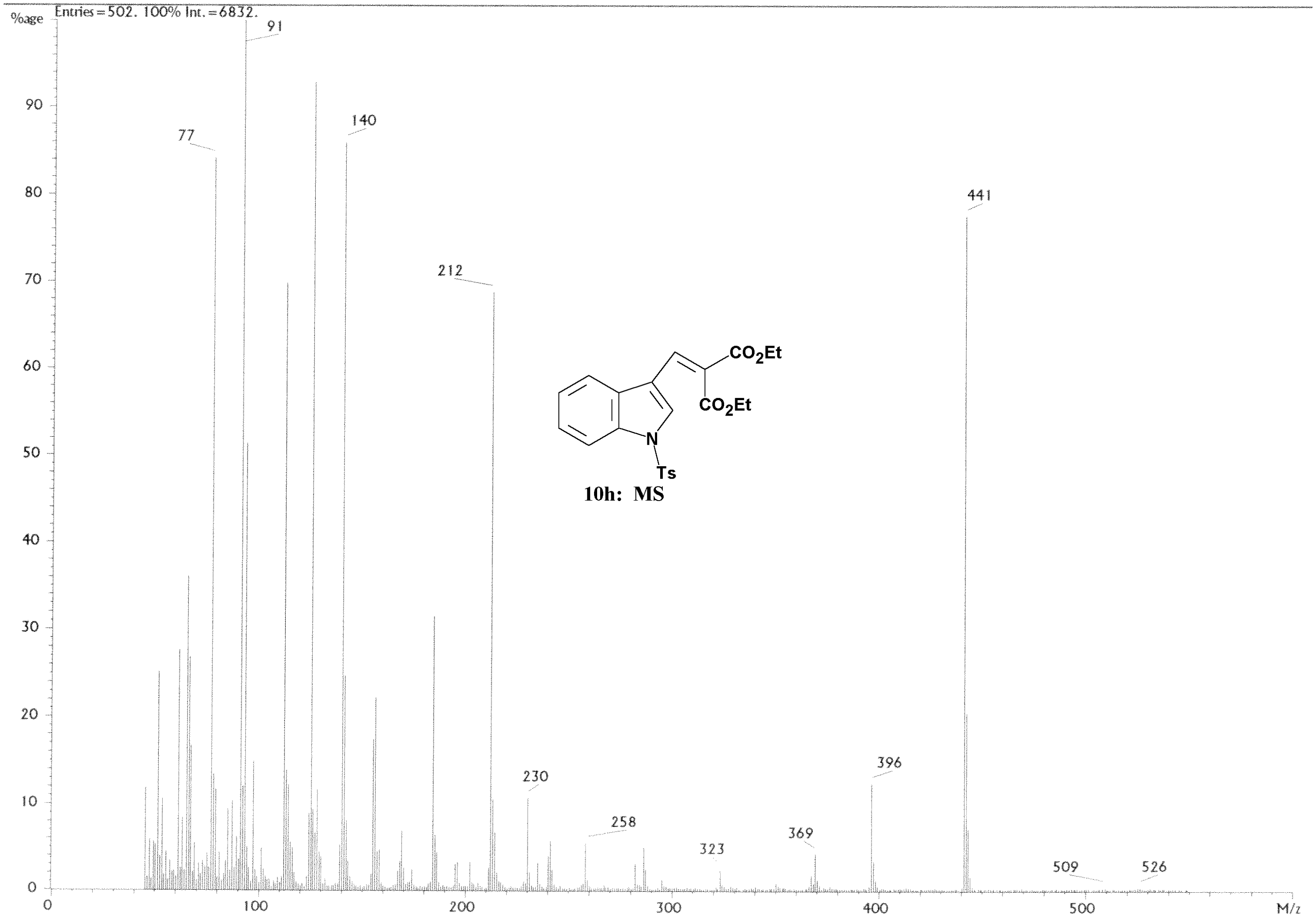
10h: ^{13}C NMR





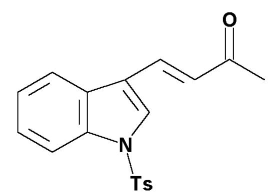
10h: IR

File Name : c:\mswin\data\kin5a.mss
Creation Date/Time : 10/26/99 at 4:47:53
File Type : Lo-Res Mass Spectrum
File Title : Kingsman ACK-I-5-11 dprobe
Source File : c:\mswin\data\kin5.mss
Source Scan(s) : 44:65

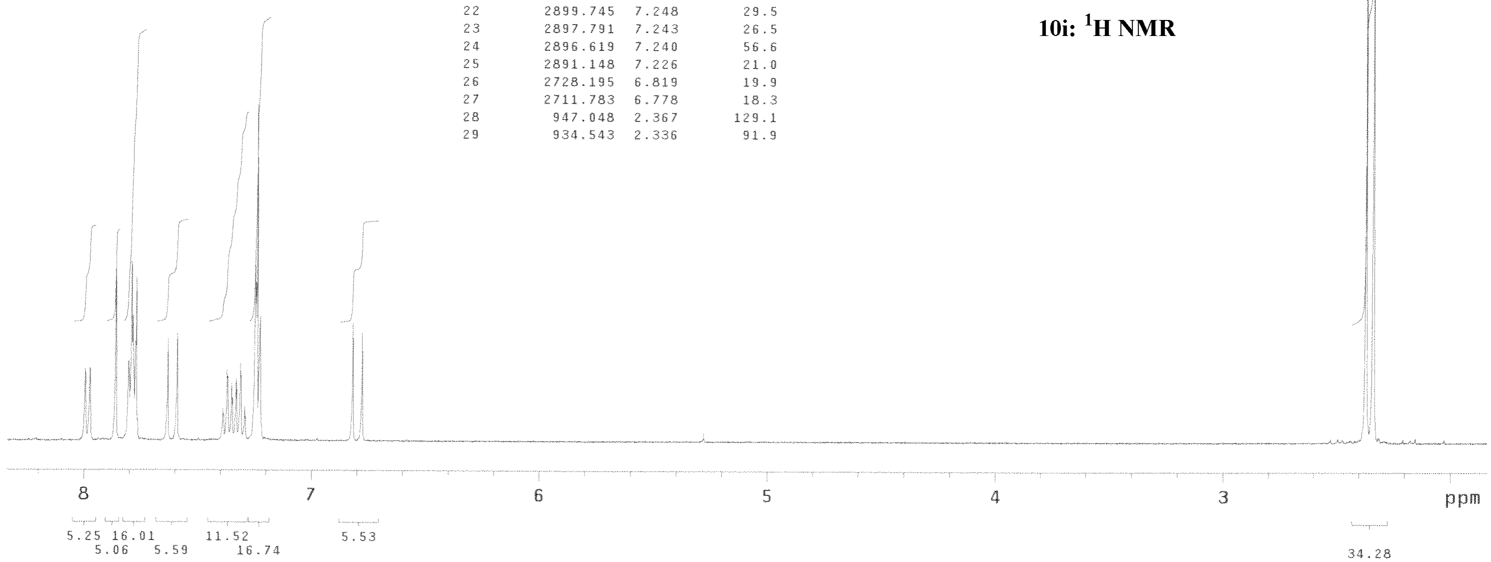


```
ack-ii-1-2 in cdcl3
STANDARD 1H OBSERVE

exp7  s2pul

      SAMPLE                SPECIAL
date    Jun 14 2000  temp       not used
solvent       cdcl3  gain       not used
file /export/home/~  spin             20
vnmr1/walkup/auto_~  hst           0.008
 14.06.00/0120.fid   pw90         13.000
   ACQUISITION       alfa         20.000
sw           6402.0          FLAGS
at            1.995  il                n
np            25546  in                n
fb             3600  dp                y
bs               16  hs               nn
ss                2       PROCESSING
d1            1.000  fn         not used
nt                8          DISPLAY
ct                8  sp            734.5
   TRANSMITTER       wp           2601.0
tn               H1  rfl          3704.9
sfrq        400.088  rfp          2896.6
tof           417.6  rp           -144.7
tpwr             58  lp            -94.7
pw            6.500            PLOT
    DECOUPLER        wc              250
dn              C13  sc                0
dof               0  vs               49
dm              nnn  th                2
dmm               c  ai  cdc  ph
dpwr             46
dmf           17100
```

INDEX	FREQUENCY	PPM	HEIGHT
1	3199.078	7.996	12.2
2	3190.090	7.974	12.2
3	3144.760	7.860	31.0
4	3122.095	7.804	13.5
5	3117.406	7.792	30.2
6	3115.062	7.786	21.0
7	3110.372	7.774	16.7
8	3108.809	7.770	27.5
9	3053.319	7.632	17.0
10	3036.907	7.591	18.1
11	2956.798	7.390	5.5
12	2955.626	7.387	4.7
13	2949.373	7.372	11.9
14	2948.201	7.369	10.8
15	2941.167	7.351	9.7
16	2939.995	7.348	7.2
17	2933.352	7.332	10.4
18	2932.179	7.329	8.6
19	2925.927	7.313	13.1
20	2924.755	7.310	10.9
21	2918.111	7.294	5.8
22	2899.745	7.248	29.5
23	2897.791	7.243	26.5
24	2896.619	7.240	56.6
25	2891.148	7.226	21.0
26	2728.195	6.819	19.9
27	2711.783	6.778	18.3
28	947.048	2.367	129.1
29	934.543	2.336	91.9

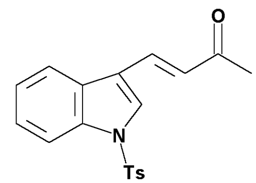


10i: ^{1}H NMR

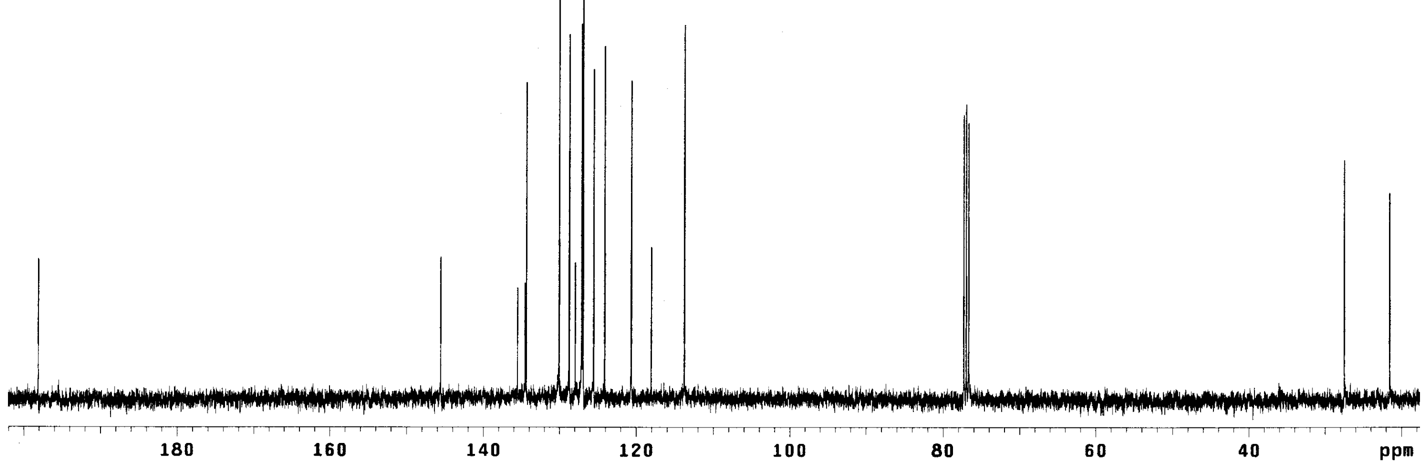


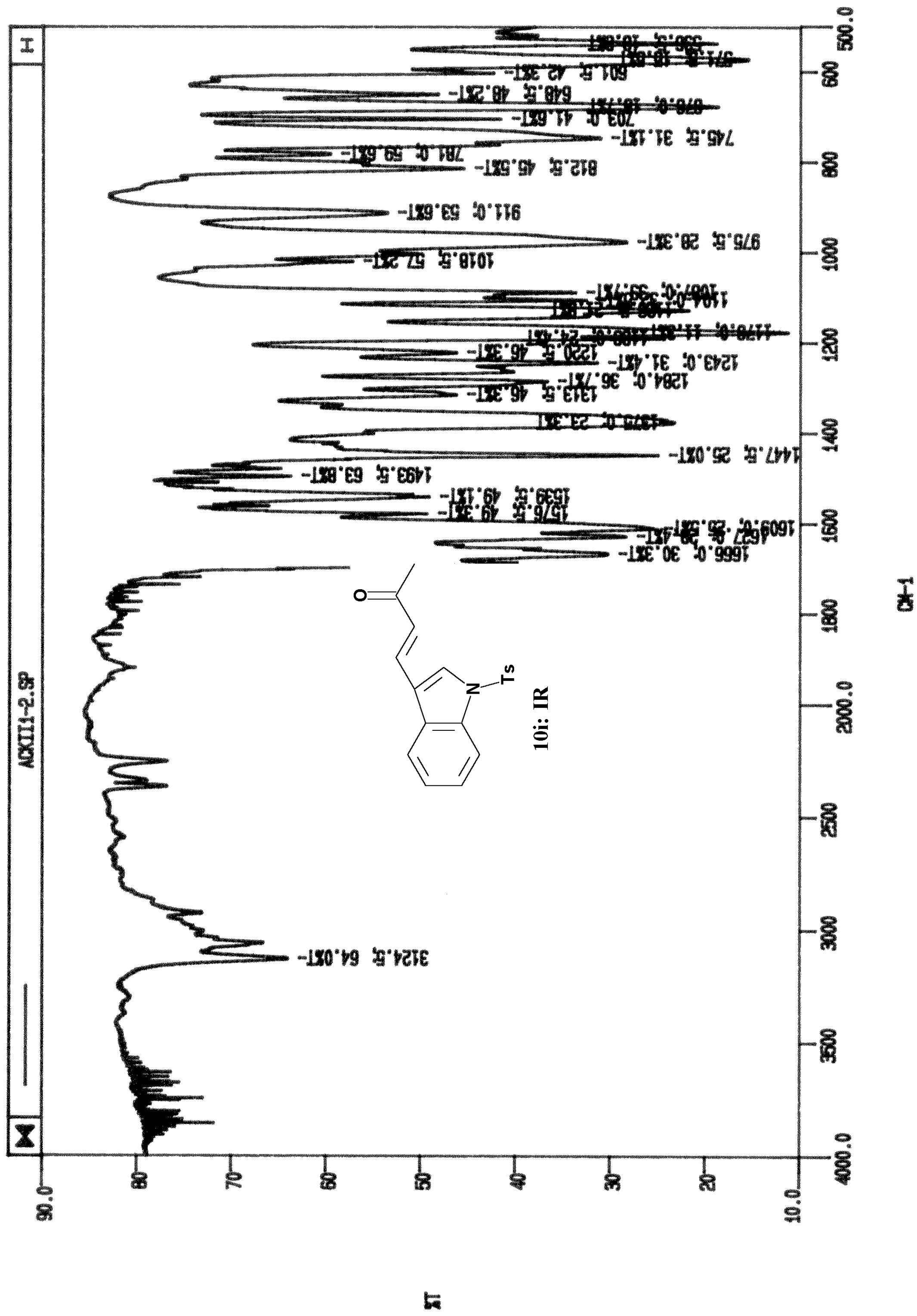
```
ack-ii-2-1 in cdcl3
13C OBSERVE

pad=2 run with gradshim before acquisiti
on

exp7  s2pul

     SAMPLE                SPECIAL
date   Jun 14 2000  temp      not used
solvent      cdcl3  gain      not used
file /export/home/~ spin            20
vnmr1/walkup/auto_~ hst          0.008
 14.06.00/0804.fid  pw90        11.700
   ACQUISITION      alfa        20.000
sw         25125.6          FLAGS
at           1.199  il               n
np           60270  in               n
fb           13800  dp               y
bs              64  hs              nn
ss               2       PROCESSING
d1           1.000  lb            1.00
nt             256  fn        not used
ct             128         DISPLAY
   TRANSMITTER      sp          1767.6
tn             C13  wp         18559.5
sfrq       100.613  rfl         9272.9
tof         1550.9  rfp         7746.3
tpwr            59  rp            91.2
pw           5.850  lp          -263.5
    DECOUPLER               PLOT
dn              H1  wc             250
dof              0  sc               0
dm             yyy  vs              57
dmm              w  th               6
dpwr            40  ai       ph
dmf           9700
```

INDEX	FREQUENCY	PPM	HEIGHT
1	19931.488	198.123	24.8
2	14646.725	145.591	25.1
3	13636.854	135.553	19.7
4	13537.937	134.570	20.6
5	13520.301	134.395	56.0
6	13086.294	130.080	123.8
7	12952.105	128.747	64.6
8	12872.358	127.954	24.2
9	12786.476	127.100	66.4
10	12770.374	126.940	110.8
11	12630.050	125.545	58.3
12	12484.359	124.097	62.4
13	12137.000	120.644	56.3
14	11877.056	118.060	26.9
15	11446.116	113.777	66.2
16	7778.527	77.320	50.2
17	7746.322	77.000	52.2
18	7714.116	76.680	48.8
19	2767.511	27.510	42.3
20	2169.409	21.564	36.4

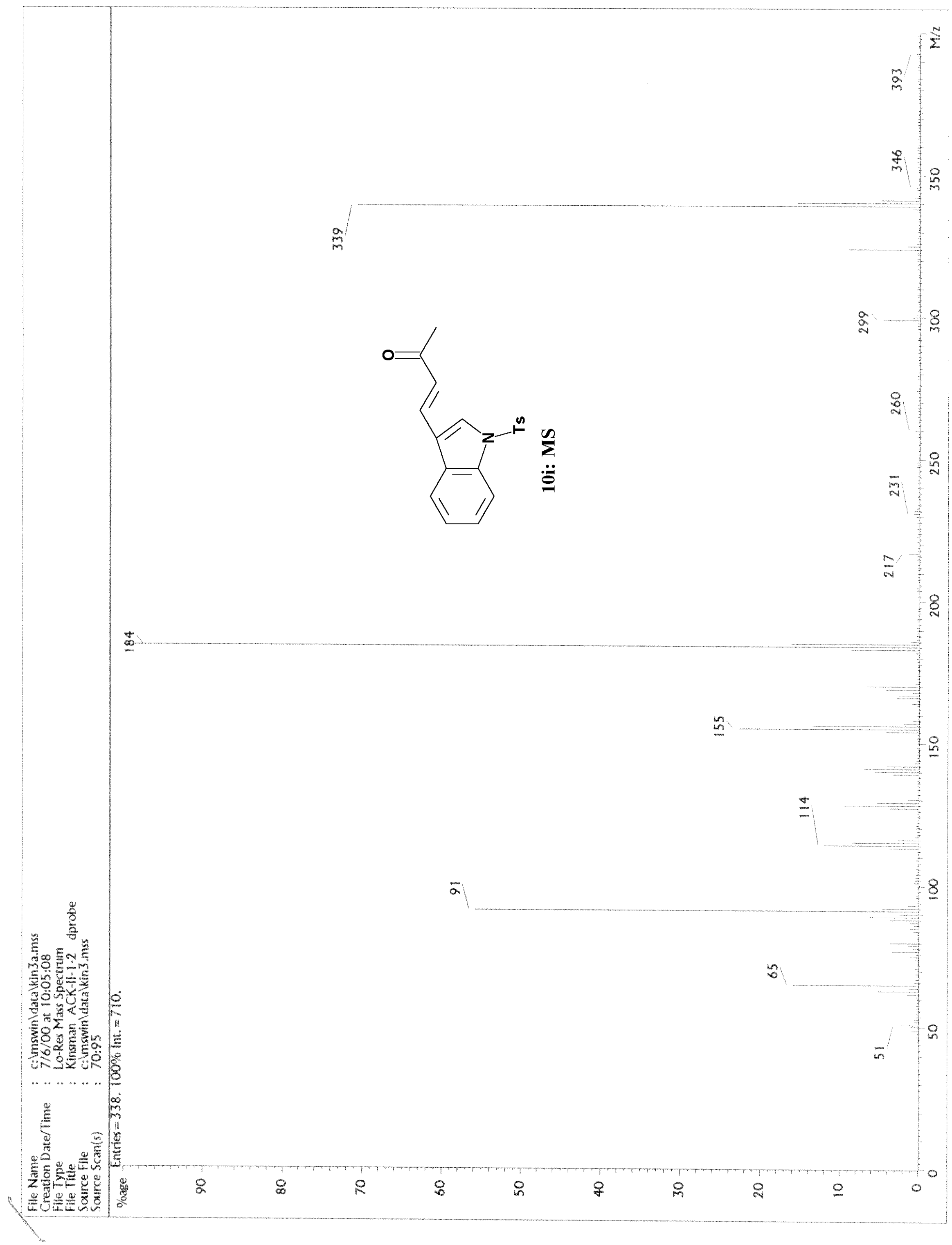


10i: ^{13}C NMR



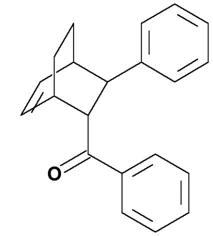


10i: IR

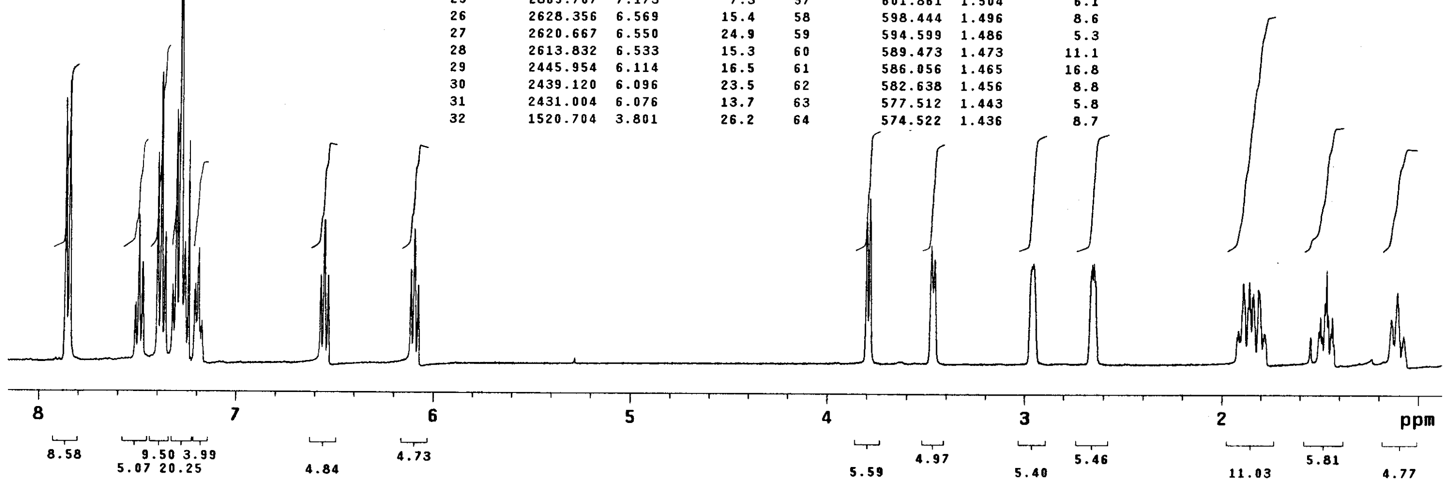

File Name : c:\mswin\data\kin3a.mss
Creation Date/Time : 7/6/00 at 10:05:08
File Type : Lo-Res Mass Spectrum
File Title : Kinsman ACK-II-1-2 dprobe
Source File : c:\mswin\data\kin3.mss
Source Scan(s) : 70:95
Entries = 338. 100% Int. = 710.
%age
M/z
51
65
91
114
155
184
217
231
260
299
339
346
393
O
N
Ts
10i: MS

```
ack-i-60-4 in cdcl3
STANDARD 1H OBSERVE

exp3  s2pul

     SAMPLE                    SPECIAL
date    Jun  6 2000   temp        not used
solvent        cdcl3  gain        not used
file /export/home/~   spin              20
vnmr1/walkup/auto_~   hst            0.008
 06.06.00/0115.fid    pw90          13.000
   ACQUISITION        alfa          20.000
sw            3499.0          FLAGS
at             1.995  il                 n
np             13962  in                 n
fb              2000  dp                 y
bs                16  hs                nn
ss                 2       PROCESSING
d1             1.000  fn          not used
nt                 8         DISPLAY
ct                 8  sp             352.8
   TRANSMITTER        wp            2909.9
tn                H1  rfl           2850.5
sfrq         400.087  rfp           2896.6
tof           -179.9  rp            -168.4
tpwr              58  lp             -34.0
pw             6.500           PLOT
   DECOUPLER          wc               250
dn               C13  sc                 0
dof                0  vs               154
dm               nnn  th                 3
dmm                c  ai        ph
dpwr              46
dmf            17100
```

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3146.513	7.865	46.0	33	1514.296	3.785	29.0	65	454.487	1.136	8.3
2	3138.824	7.845	49.0	34	1389.990	3.474	20.9	66	442.527	1.106	12.9
3	3137.116	7.841	31.0	35	1383.582	3.458	18.5	67	430.139	1.075	5.4
4	3005.547	7.512	9.9	36	1185.375	2.963	17.3				
5	3004.266	7.509	10.4	37	1181.958	2.954	17.8				
6	3002.984	7.506	5.6	38	1064.914	2.662	16.6				
7	2997.431	7.492	25.8	39	1062.778	2.656	17.6				
8	2990.596	7.475	17.4	40	1059.360	2.648	17.8				
9	2989.742	7.473	17.4	41	768.885	1.922	5.1				
10	2959.413	7.397	36.5	42	766.322	1.915	6.2				
11	2951.724	7.378	50.6	43	762.477	1.906	4.9				
12	2944.035	7.359	22.7	44	759.060	1.897	9.8				
13	2929.511	7.322	13.6	45	756.497	1.891	14.5				
14	2921.395	7.302	44.0	46	746.672	1.866	10.3				
15	2914.560	7.285	94.1	47	744.109	1.860	14.7				
16	2911.570	7.277	70.6	48	741.546	1.853	10.1				
17	2904.735	7.260	21.0	49	737.701	1.844	12.1				
18	2896.619	7.240	38.6	50	735.565	1.839	12.7				
19	2885.085	7.211	12.7	51	725.740	1.814	13.4				
20	2883.377	7.207	13.8	52	723.605	1.809	12.9				
21	2880.814	7.200	8.4	53	719.333	1.798	7.1				
22	2876.542	7.190	20.0	54	715.916	1.789	5.0				
23	2873.979	7.183	5.8	55	713.353	1.783	5.7				
24	2871.843	7.178	6.4	56	618.521	1.546	5.2				
25	2869.707	7.173	7.3	57	601.861	1.504	6.1				
26	2628.356	6.569	15.4	58	598.444	1.496	8.6				
27	2620.667	6.550	24.9	59	594.599	1.486	5.3				
28	2613.832	6.533	15.3	60	589.473	1.473	11.1				
29	2445.954	6.114	16.5	61	586.056	1.465	16.8				
30	2439.120	6.096	23.5	62	582.638	1.456	8.8				
31	2431.004	6.076	13.7	63	577.512	1.443	5.8				
32	1520.704	3.801	26.2	64	574.522	1.436	8.7				

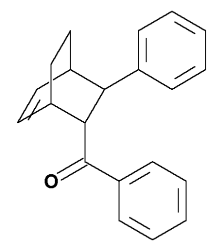


endo-11a: ^{1}H NMR

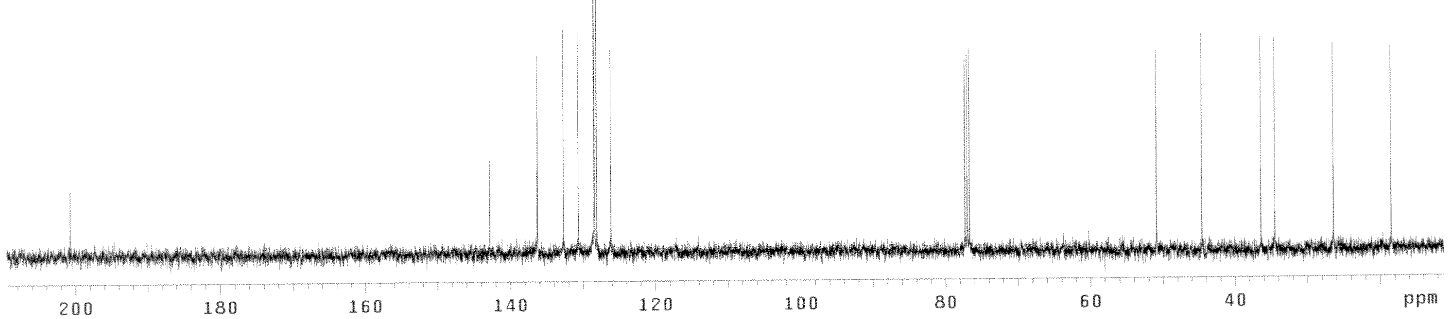


```
ack-i-60-4 in cdcl3
13C OBSERVE

pad=2 run with gradshim before acquisiti
on

exp4  s2pul

     SAMPLE              SPECIAL
date   Jun 13 2000  temp      not used
solvent       cdcl3  gain      not used
file /export/home/~ spin            20
vnmr1/walkup/auto_~ hst          0.008
 13.06.00/0701.fid  pw90        11.700
   ACQUISITION      alfa        20.000
sw          25125.6        FLAGS
at            1.199  il               n
np            60270  in               n
fb            13800  dp               y
bs               64  hs              nn
ss                2      PROCESSING
d1            1.000  lb            1.00
nt              256  fn        not used
ct               64       DISPLAY
   TRANSMITTER      sp          1119.7
tn              C13  wp         19951.3
sfrq        100.613  rfl         9271.3
tof          1550.9  rfp         7746.3
tpwr             59  rp          102.1
pw            5.850  lp         -304.3
    DECOUPLER              PLOT
dn               H1  wc             250
dof               0  sc               0
dm              yyy  vs              64
dmm               w  th               5
dpwr             40  ai  no  ph
dmf            9700
```

INDEX	FREQUENCY	PPM	HEIGHT
1	20194.499	200.737	11.2
2	14366.844	142.809	16.4
3	13715.834	136.338	14.5
4	13708.166	136.262	34.4
5	13348.538	132.687	38.8
6	13141.503	130.629	38.5
7	12923.733	128.465	73.9
8	12918.365	128.411	127.0
9	12886.927	128.099	63.0
10	12689.860	126.140	35.3
11	7778.527	77.320	33.2
12	7746.322	77.000	34.0
13	7714.883	76.687	35.1
14	5121.576	50.910	34.5
15	4488.969	44.621	37.4
16	3663.896	36.420	36.6
17	3470.663	34.499	36.6
18	2657.092	26.412	35.6
19	1858.090	18.470	35.0



endo-**11a: ^{13}C NMR**

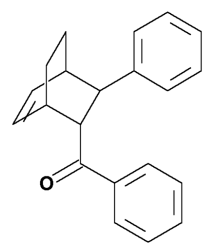


ack-i-60-4 in cdcl3
STANDARD 1H OBSERVE

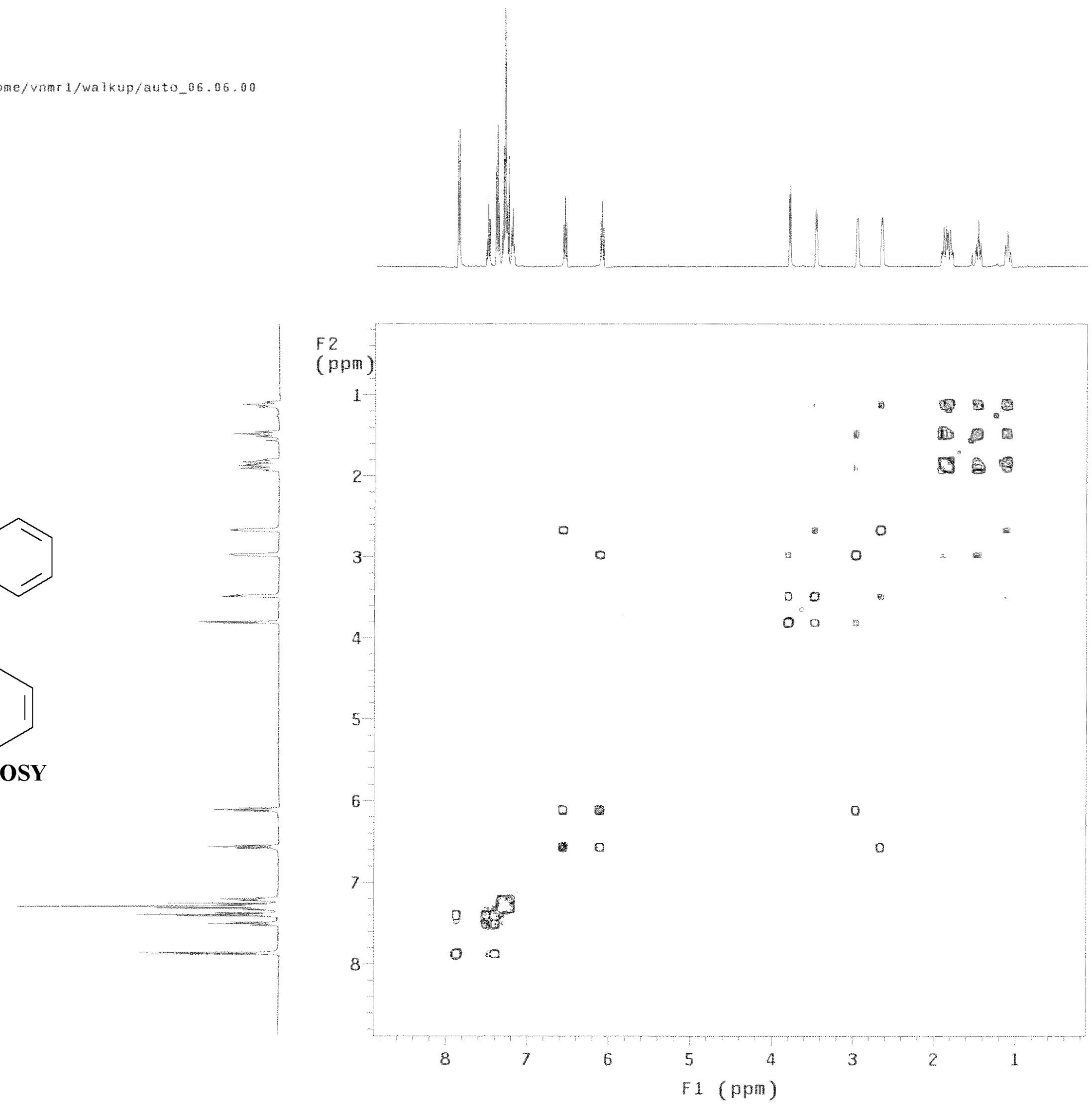
Automation directory: /export/home/vnmr1/walkup/auto_06.06.00
File : 0117

Pulse Sequence: gCOSY
Solvent: cdcl3
Ambient temperature
Sample #1
File: 0116
Mercury-400BB "nmrm400"

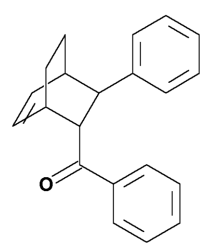
Relax. delay 1.000 sec
Acq. time 0.146 sec
Width 3499.0 Hz
2D Width 3499.0 Hz
Single scan
128 increments
OBSERVE H1, 400.0854801 MHz
DATA PROCESSING
Sq. sine bell 0.073 sec
F1 DATA PROCESSING
Sq. sine bell 0.037 sec
FT size 1024 x 1024
Total time 3 min, 35 sec



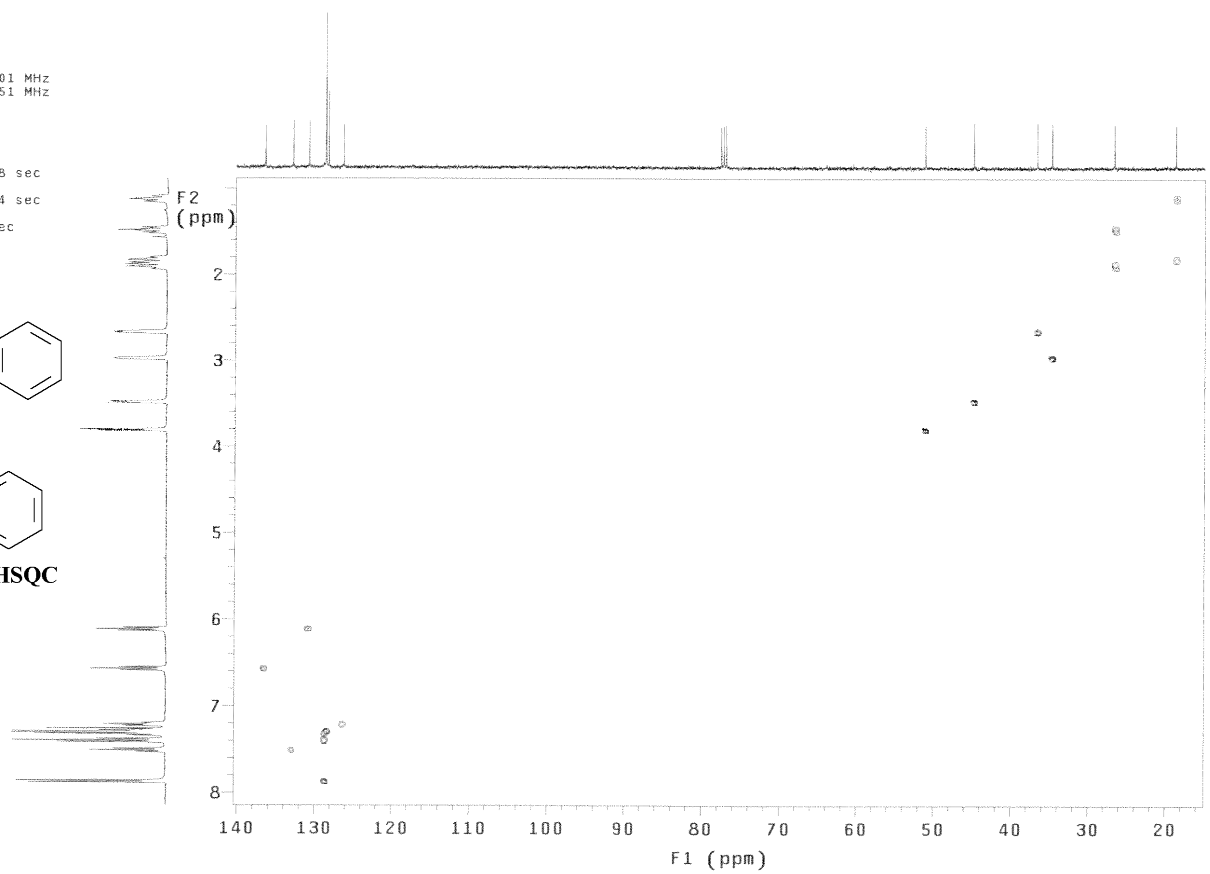
endo-**11a: gCOSY**



```
ack-i-60-4 in cdcl3
STANDARD 1H OBSERVE

 Archive directory: /export/home/vnmr1/vnmrsys/data
 Sample directory: auto_06Jun2000

Pulse Sequence: gHSQC

 Solvent: cdcl3
 Ambient temperature
File: 0117
Mercury-400BB  "nmrm400"

 Relax. delay 1.000 sec
 Acq. time 0.146 sec
 Width    3499.0 Hz
 2D Width 17094.0 Hz
 4 repetitions
 2 x 128 increments
OBSERVE   H1, 400.0854801 MHz
DECOUPLE C13, 100.6090951 MHz
 Power 46 dB
 on during acquisition
 off during delay
 GARP-1 modulated
DATA PROCESSING
 Gauss apodization 0.068 sec
F1 DATA PROCESSING
 Gauss apodization 0.014 sec
FT size 1024 x 2048
Total time 23 min, 16 sec
```



***endo*-11a: gHSQC**



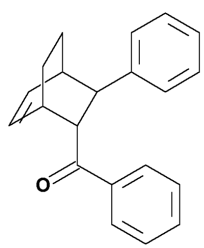
ack-i-60-4 in cdcl3
STANDARD 1H OBSERVE

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory: auto_08Jun2000

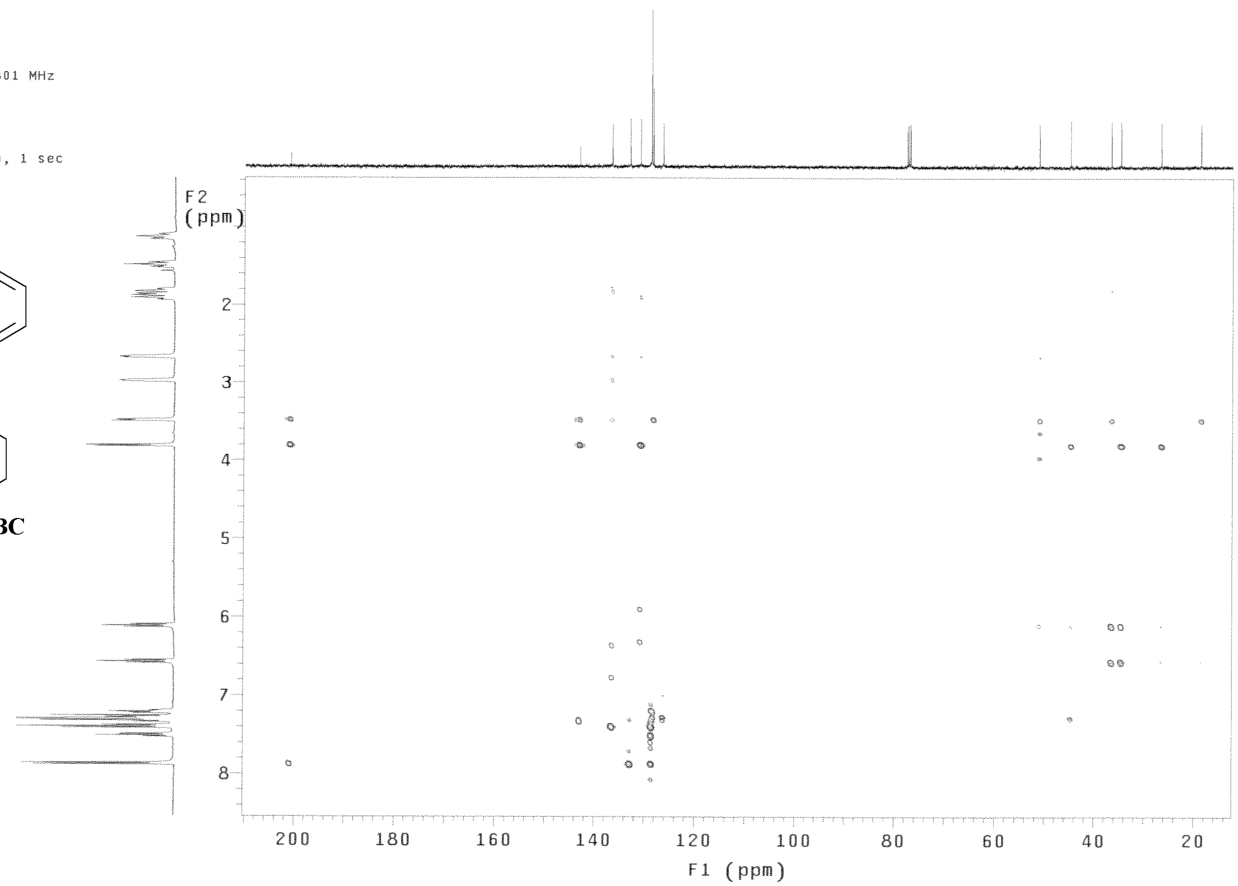
Pulse Sequence: gHMBC

Solvent: cdcl3
Ambient temperature
File: 0703
Mercury-400BB "nmrm400"

Relax. delay 1.000 sec
Acq. time 0.143 sec
Width 3585.5 Hz
2D Width 24154.6 Hz
16 repetitions
512 increments
OBSERVE H1, 400.0854801 MHz
DATA PROCESSING
Sine bell 0.071 sec
F1 DATA PROCESSING
Sine bell 0.011 sec
FT size 1024 x 2048
Total time 2 hr, 55 min, 1 sec



***endo*-11a: HMBC**



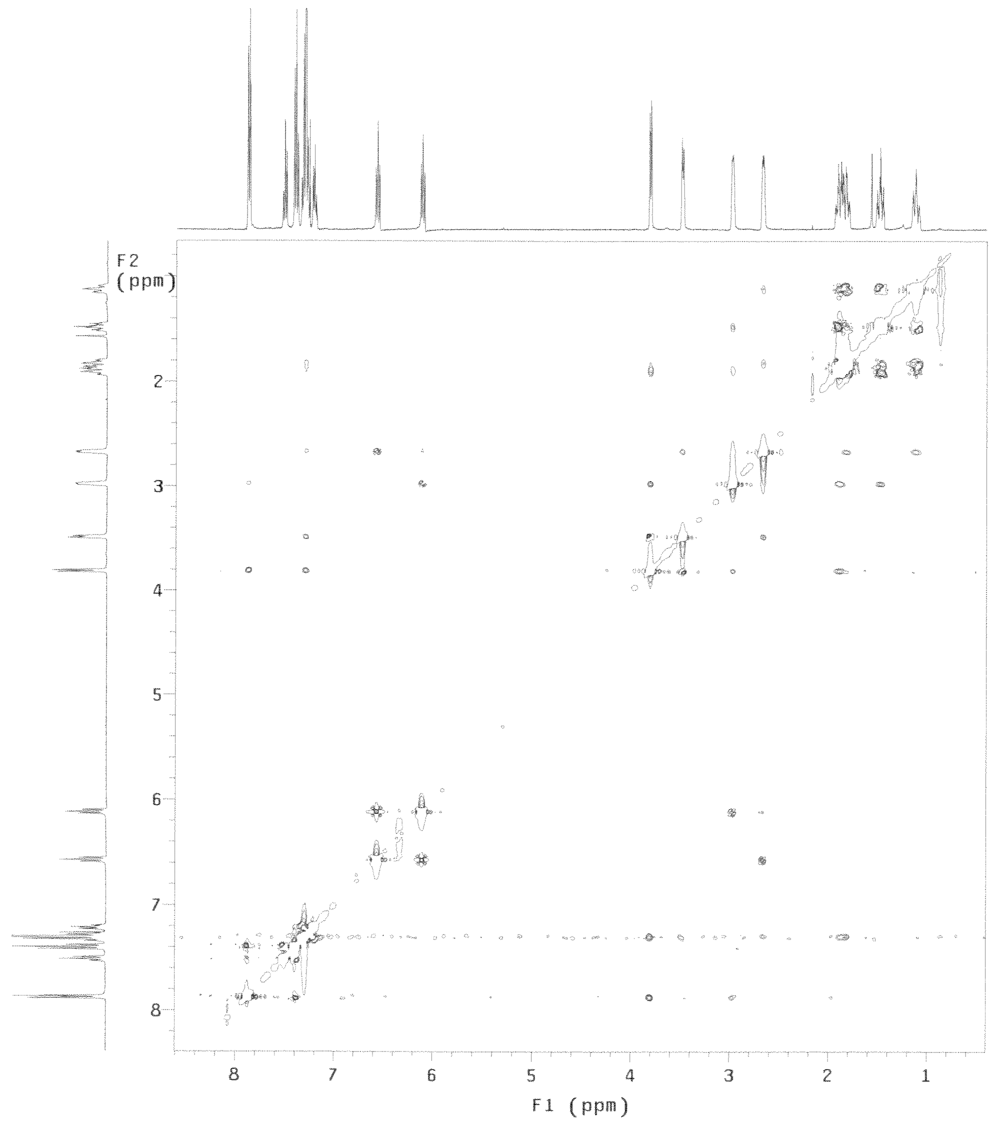
ack-i-60-4 in cdcl3
STANDARD 1H OBSERVE

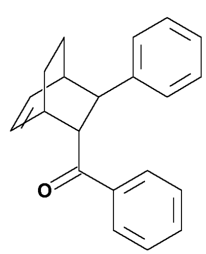
Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory: auto_07Jun2000

Pulse Sequence: NOESY

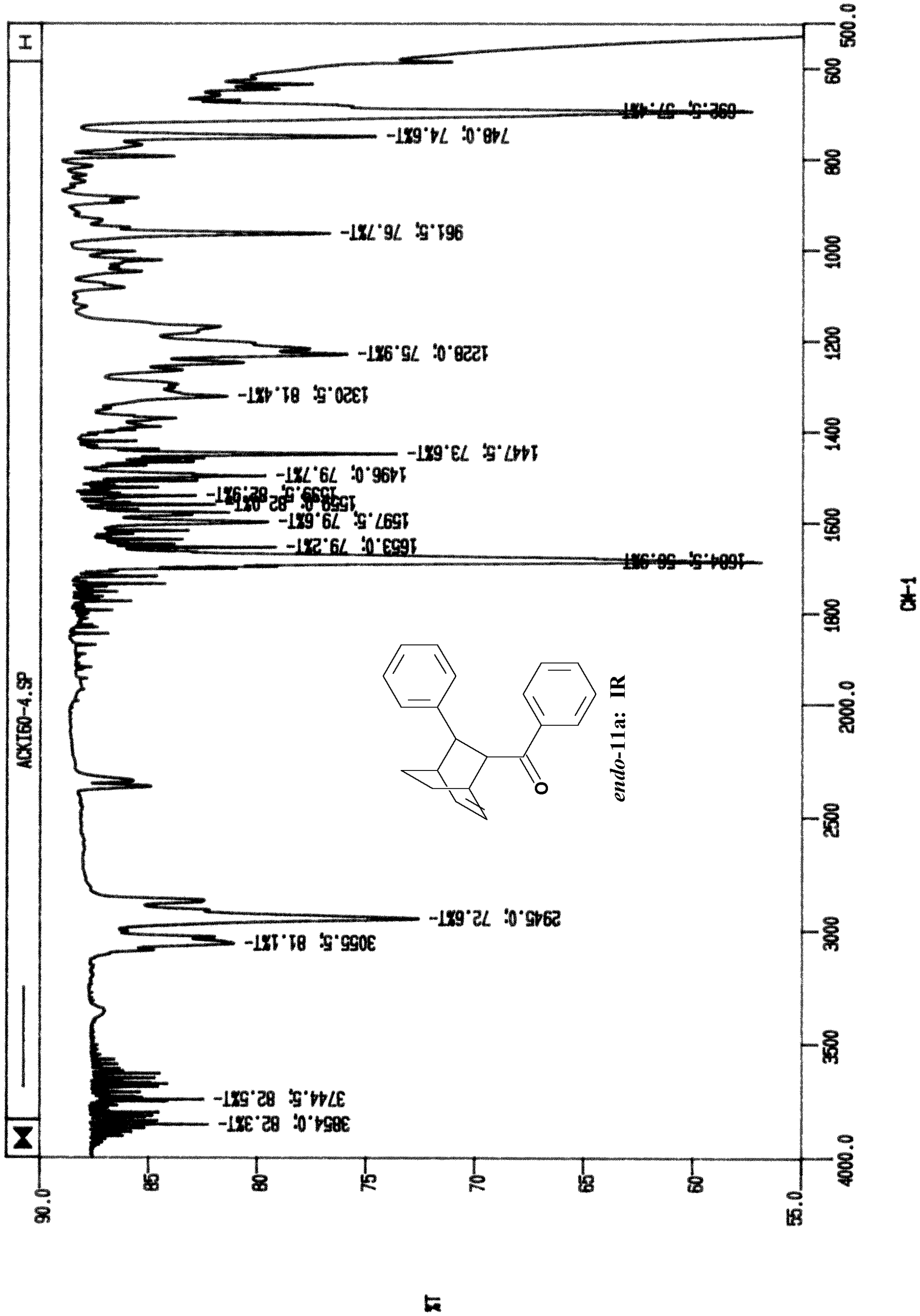
Solvent: cdcl3
Ambient temperature
File: 0115
Mercury-400BB "nmrm400"

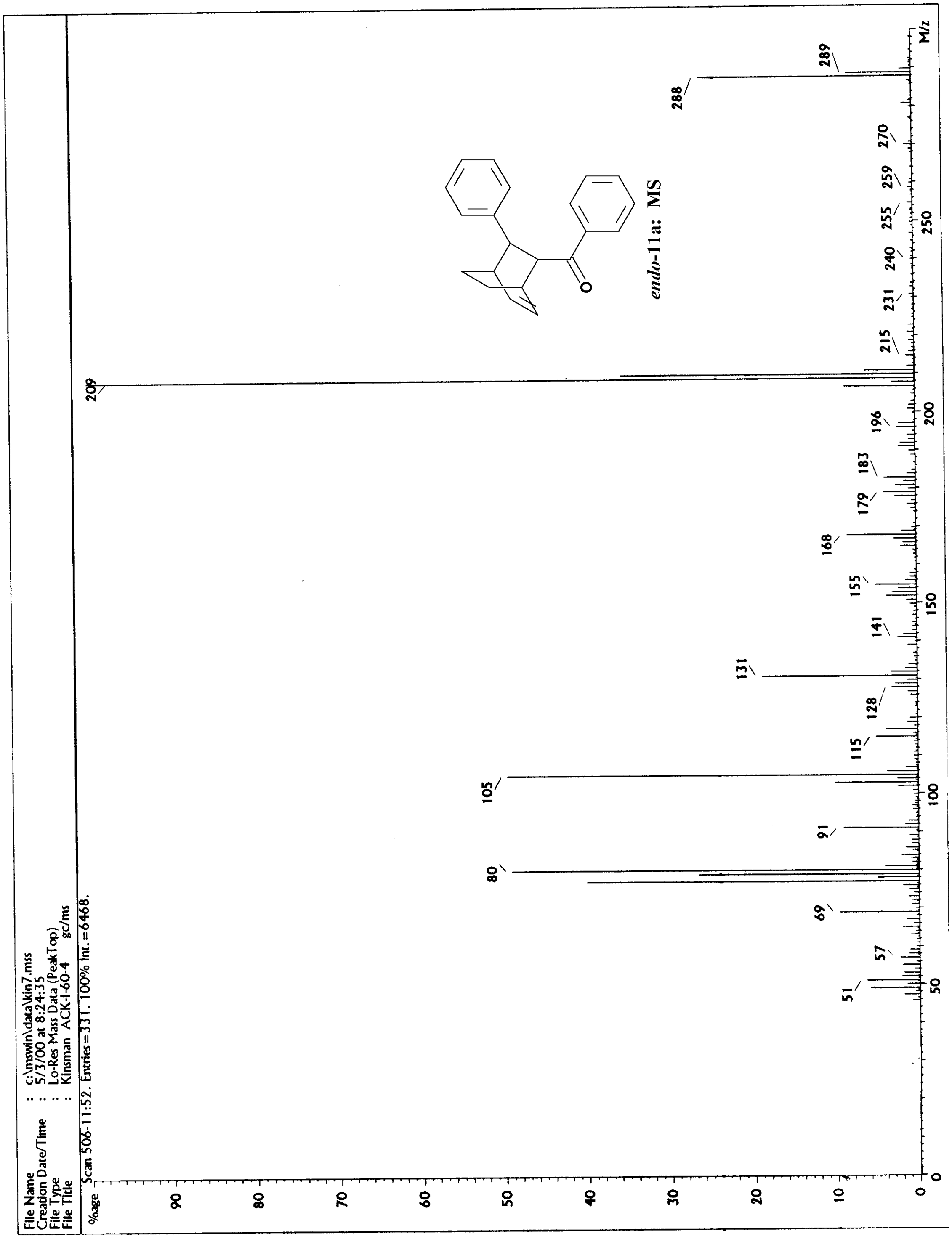
Relax. delay 1.000 sec
Mixing 0.200 sec
Acq. time 0.142 sec
Width 3597.1 Hz
2D Width 3597.1 Hz
16 repetitions
2 x 200 increments
OBSERVE H1, 400.0854801 MHz
DATA PROCESSING
Gauss apodization 0.066 sec
F1 DATA PROCESSING
Gauss apodization 0.051 sec
FT size 2048 x 2048
Total time 2 hr, 32 min, 50 sec





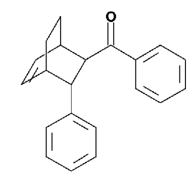
endo-**11a: NOESY**


ACKI60-4.SP
3854.0; 82.3%T
3744.5; 82.5%T
3055.5; 81.1%T
2945.0; 72.6%T
1684.5; 56.9%T
1653.0; 79.2%T
1597.5; 79.6%T
1496.0; 79.7%T
1447.5; 73.6%T
1320.5; 81.4%T
1228.0; 75.9%T
961.5; 76.7%T
748.0; 74.6%T
692.5; 57.4%T
CM-1
%T
O
endo-11a: IR

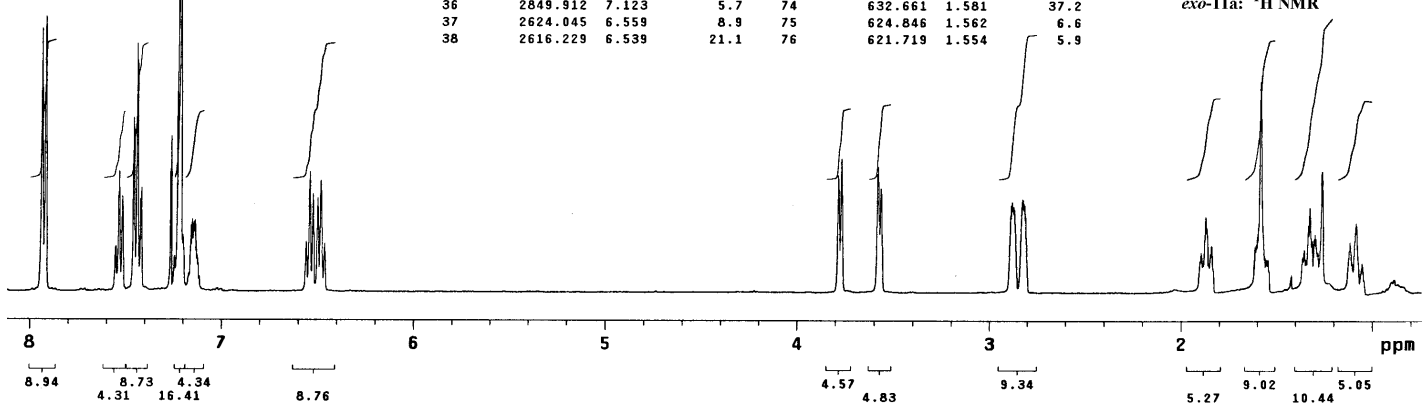

File Name : c:\mswin\data\kin7.mss
Creation Date/Time : 5/3/00 at 8:24:35
File Type : Lo-Res Mass Data (PeakTop)
File Title : Kinsman ACK-1-60-4 gc/ms
Scan 506-11:52. Entries=331. 100% Int.=6468.
%age
M/z
51
57
69
80
91
105
115
128
131
141
155
168
179
183
196
209
215
231
240
255
259
270
288
289
endo-11a: MS

```
ack166_5
STANDARD 1H OBSERVE

expl1  s2pul

      SAMPLE                 SPECIAL
date   Sep  1 2000   temp       not used
solvent       cdcl3  gain       not used
file /export/home/~  spin             20
vnmr1/vnmrsys/data~  hst           0.008
/ackinsma/ack166_5~  pw90         14.200
      /PROTON.fid    alfa         20.000
   ACQUISITION               FLAGS
sw           6402.0  il                n
at            1.995  in                n
np            25546  dp                y
fb             3600  hs               nn
bs               16        PROCESSING
ss                2  fn         not used
d1            1.000         DISPLAY
nt               32  sp            297.8
ct               32  wp           2949.2
   TRANSMITTER       rfl          3702.2
tn               H1  rfp          2904.6
sfrq        400.088  rp           -141.7
tof           417.6  lp           -101.0
tpwr             57            PLOT
pw            7.100  wc              250
    DECOUPLER        sc                0
dn              C13  vs              131
dof               0  th                3
dm              nnn  ai   cdc   ph
dmm               c
dpwr             44
dmf           15900
```

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3175.427	7.937	46.4	39	2609.586	6.523	17.3	77	619.766	1.549	5.8
2	3167.221	7.916	48.5	40	2600.208	6.499	16.6	78	617.421	1.543	5.8
3	3165.658	7.912	26.4	41	2593.565	6.483	19.6	79	544.737	1.362	6.3
4	3022.244	7.554	7.7	42	2585.749	6.463	8.5	80	541.611	1.354	7.6
5	3020.680	7.550	8.0	43	1513.856	3.784	20.6	81	536.922	1.342	5.9
6	3019.508	7.547	4.9	44	1507.213	3.767	23.6	82	532.623	1.331	11.9
7	3015.600	7.537	16.9	45	1432.966	3.582	20.9	83	529.497	1.323	15.0
8	3013.646	7.533	21.2	46	1431.403	3.578	22.2	84	525.198	1.313	9.2
9	3011.302	7.527	6.1	47	1429.449	3.573	13.6	85	520.509	1.301	9.6
10	3007.394	7.517	16.8	48	1426.714	3.566	18.3	86	518.946	1.297	10.3
11	3006.222	7.514	15.9	49	1425.151	3.562	18.3	87	517.383	1.293	9.5
12	3004.659	7.510	6.1	50	1155.126	2.887	13.6	88	513.084	1.282	7.1
13	2983.166	7.456	30.7	51	1153.954	2.884	15.5	89	504.097	1.260	21.4
14	2976.132	7.439	43.8	52	1152.781	2.881	15.2	90	449.388	1.123	6.2
15	2974.960	7.436	40.2	53	1151.609	2.878	16.0	91	447.434	1.118	7.5
16	2969.098	7.421	16.3	54	1150.437	2.875	15.1	92	445.871	1.114	8.9
17	2967.535	7.417	18.3	55	1149.655	2.874	14.8	93	444.308	1.111	7.2
18	2906.965	7.266	21.0	56	1148.483	2.871	15.1	94	442.354	1.106	5.3
19	2906.184	7.264	21.5	57	1147.310	2.868	14.7	95	440.400	1.101	4.3
20	2904.621	7.260	27.3	58	1131.679	2.829	15.2	96	433.757	1.084	12.3
21	2898.759	7.245	6.2	59	1130.116	2.825	16.5	97	424.379	1.061	3.5
22	2897.977	7.243	5.9	60	1128.553	2.821	16.3	98	422.425	1.056	4.4
23	2890.162	7.224	61.9	61	1126.990	2.817	15.7	99	420.862	1.052	5.2
24	2884.691	7.210	133.5	62	1125.818	2.814	15.4				
25	2881.174	7.201	10.0	63	1124.255	2.810	13.4				
26	2879.220	7.197	7.6	64	760.444	1.901	5.6				
27	2863.980	7.158	11.7	65	757.709	1.894	7.1				
28	2861.245	7.152	12.7	66	754.192	1.885	5.3				
29	2860.463	7.150	11.3	67	748.330	1.870	13.4				
30	2858.900	7.146	11.9	68	742.078	1.855	5.3				
31	2857.728	7.143	12.0	69	738.561	1.846	6.9				
32	2856.946	7.141	12.4	70	736.216	1.840	8.3				
33	2855.383	7.137	12.6	71	644.384	1.611	8.1				
34	2853.429	7.132	8.9	72	642.040	1.605	8.5				
35	2851.475	7.127	6.4	73	639.695	1.599	9.9				
36	2849.912	7.123	5.7	74	632.661	1.581	37.2				
37	2624.045	6.559	8.9	75	624.846	1.562	6.6				
38	2616.229	6.539	21.1	76	621.719	1.554	5.9				

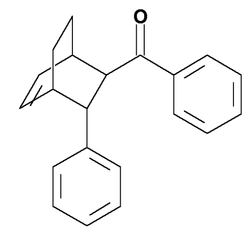


exo-**11a**: [1]H NMR

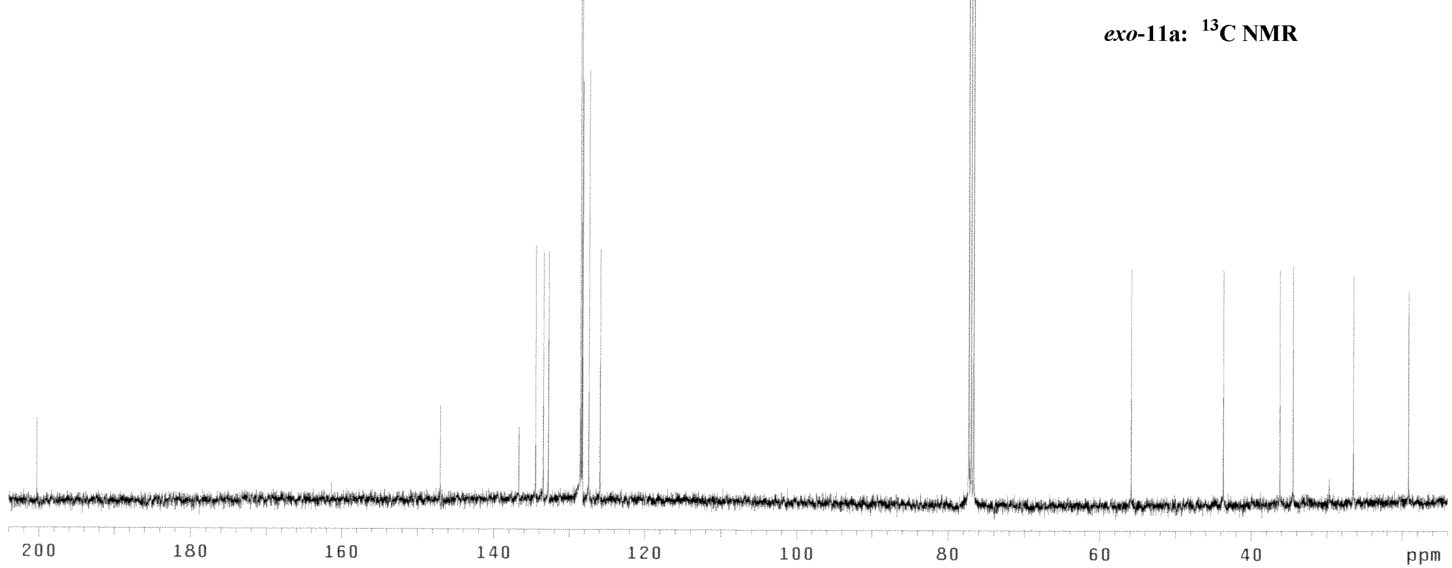


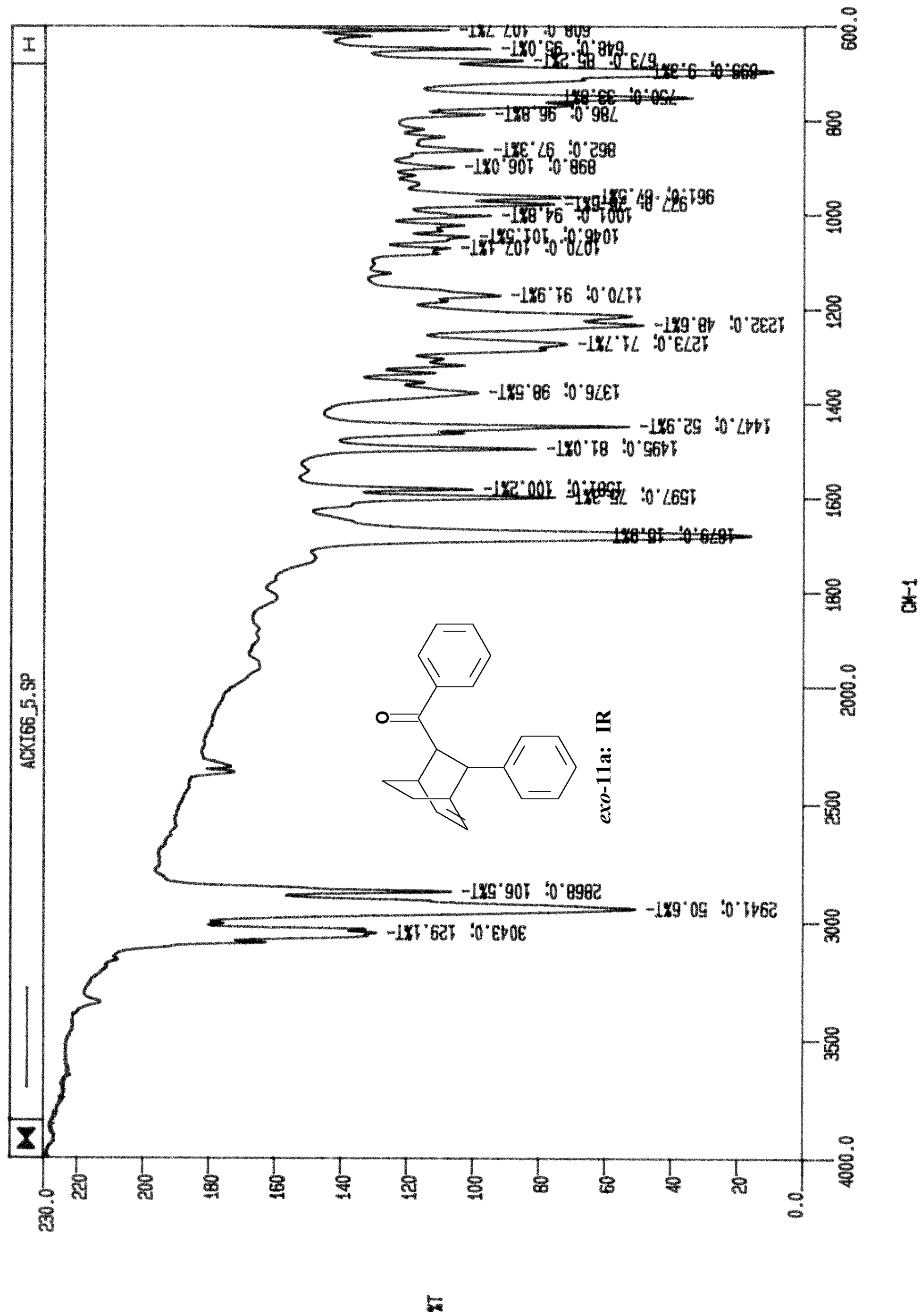
```
acki66_5
STANDARD 1H OBSERVE

exp11  s2pul

      SAMPLE              SPECIAL
date   Sep  1 2000  temp       not used
solvent      cdcl3  gain       not used
file /export/home/~ spin             20
vnmr1/vnmrsys/data~ hst           0.008
/ackinsma/acki66_5~ pw90         14.700
     /CARBON.fid    alfa         20.000
   ACQUISITION             FLAGS
sw         25125.6  il                n
at           1.199  in                n
np           60270  dp                y
fb           13800  hs               nn
bs              64        PROCESSING
ss               2  lb             1.00
d1           1.000  fn         not used
nt            1000         DISPLAY
ct            1000  sp           1407.2
   TRANSMITTER      wp          19119.3
tn             C13  rfl          9269.0
sfrq       100.613  rfp          7746.3
tof         1550.9  rp            118.9
tpwr            56  lp           -299.8
pw           7.350           PLOT
    DECOUPLER       wc              250
dn              H1  sc                0
dof              0  vs              129
dm             yyy  th                7
dmm              w  ai  no  ph
dpwr            39
dmf           8500
```

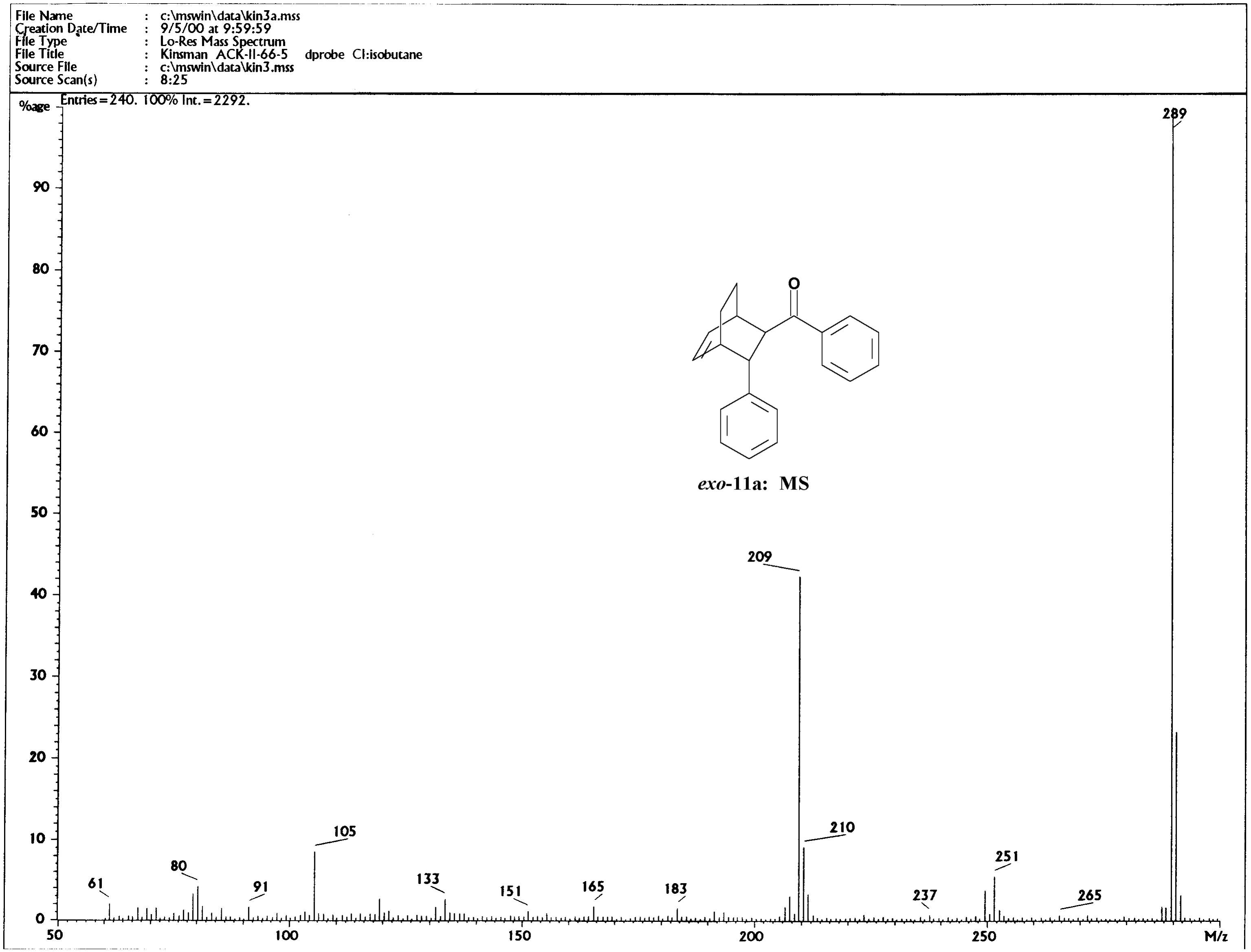
INDEX	FREQUENCY	PPM	HEIGHT
1	20151.559	200.311	14.2
2	14790.116	147.017	16.5
3	13751.873	136.696	12.8
4	13530.269	134.494	44.5
5	13427.518	133.472	43.1
6	13360.040	132.801	43.3
7	12934.468	128.571	87.4
8	12916.065	128.388	87.7
9	12902.263	128.251	72.9
10	12818.682	127.420	75.0
11	12666.856	125.911	43.8
12	7777.760	77.313	135.4
13	7746.321	77.000	144.8
14	7714.116	76.680	144.8
15	5618.460	55.849	40.4
16	4396.186	43.699	40.3
17	3645.492	36.237	40.4
18	3470.663	34.499	41.3
19	2665.526	26.496	39.4
20	1930.935	19.194	36.8



***exo*-11a: ^{13}C NMR**




ACKI66_5.SP
exo-11a: IR
%T
CM-1
3043.0; 129.1%T
2941.0; 50.6%T
2868.0; 106.5%T
1679.0; 18.8%T
1597.0; 75.3%T
1581.0; 100.2%T
1495.0; 81.0%T
1447.0; 52.9%T
1376.0; 98.5%T
1273.0; 71.7%T
1232.0; 48.6%T
1170.0; 91.9%T
1070.0; 107.1%T
1046.0; 101.5%T
1001.0; 94.8%T
961.6; 87.5%T
898.0; 106.0%T
862.0; 97.3%T
786.0; 96.8%T
750.0; 38.8%T
695.0; 9.3%T
673.0; 85.2%T
648.0; 95.0%T
608.0; 107.7%T

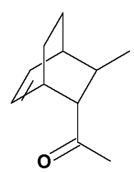


File Name : c:\mswin\data\kin3a.mss
Creation Date/Time : 9/5/00 at 9:59:59
File Type : Lo-Res Mass Spectrum
File Title : Kinsman ACK-II-66-5 dprobe CI:isobutane
Source File : c:\mswin\data\kin3.mss
Source Scan(s) : 8:25
Entries=240. 100% Int.=2292.
%age
0
10
20
30
40
50
60
70
80
90
50
100
150
200
250
M/z
61
80
91
105
133
151
165
183
209
210
237
251
265
289
O

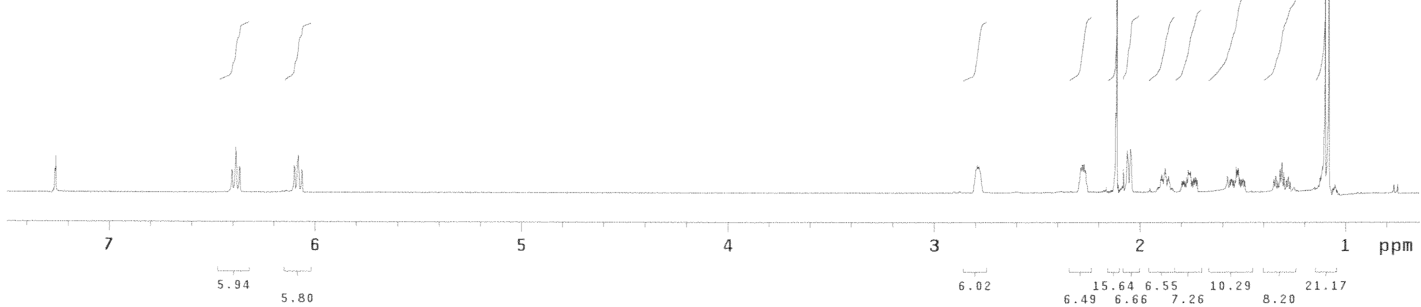
exo-11a: MS

```
ack-ii-30-8 in cdcl3
STANDARD 1H OBSERVE

exp11  s2pul
      SAMPLE              SPECIAL
date   Sep  1 2000  temp       not used
solvent      cdcl3  gain       not used
file /export/home/~ spin             20
vnmr1/walkup/auto_~ hst           0.008
 01.09.00/0704.fid  pw90         14.200
   ACQUISITION      alfa         20.000
sw          3298.2          FLAGS
at           1.995  il                n
np           13160  in                n
fb            1800  dp                y
bs              16  hs               nn
ss               2        PROCESSING
d1           1.000  fn         not used
nt              32          DISPLAY
ct              16  sp            256.4
   TRANSMITTER      wp           2744.1
tn              H1  rfl          2953.5
sfrq       400.087  rfp          2904.6
tof         -385.0  rp           -169.3
tpwr            57  lp            -37.5
pw           7.100           PLOT
    DECOUPLER       wc              250
dn             C13  sc                0
dof              0  vs               68
dm             nnn  th                1
dmm              c  ai   cdc  ph
dpwr            44
dmf          15900
```

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	2906.231	7.264	4.4	33	758.063	1.895	3.4	65	601.027	1.502	2.8
2	2904.621	7.260	6.6	34	755.647	1.889	2.7	66	599.417	1.498	2.7
3	2563.169	6.407	4.2	35	752.828	1.882	3.7	67	597.001	1.492	2.5
4	2561.961	6.404	4.1	36	751.218	1.878	4.7	68	539.824	1.349	2.6
5	2556.323	6.389	5.3	37	749.204	1.873	3.0	69	536.200	1.340	3.2
6	2555.115	6.386	8.1	38	746.386	1.866	2.6	70	532.173	1.330	2.5
7	2548.270	6.369	4.8	39	744.373	1.861	3.2	71	527.744	1.319	4.5
8	2547.062	6.366	4.4	40	742.359	1.856	2.1	72	524.120	1.310	5.7
9	2441.164	6.102	4.9	41	719.408	1.798	2.2	73	520.093	1.300	3.9
10	2433.513	6.082	6.7	42	716.992	1.792	2.3	74	515.664	1.289	2.7
11	2426.668	6.065	4.2	43	714.979	1.787	2.4	75	512.040	1.280	3.1
12	1116.829	2.791	5.1	44	712.563	1.781	2.3	76	508.014	1.270	2.2
13	1115.219	2.787	4.8	45	709.342	1.773	3.0	77	501.169	1.253	1.2
14	1113.608	2.783	5.0	46	706.523	1.766	4.3	78	459.695	1.149	1.3
15	914.696	2.286	4.8	47	704.912	1.762	3.8	79	452.850	1.132	1.6
16	911.475	2.278	5.2	48	702.496	1.756	4.0	80	449.629	1.124	2.9
17	910.267	2.275	4.1	49	699.678	1.749	2.4	81	448.018	1.120	3.2
18	909.059	2.272	5.3	50	696.457	1.741	2.9	82	445.199	1.113	4.9
19	905.838	2.264	4.3	51	694.041	1.735	3.0	83	439.562	1.099	55.0
20	865.572	2.163	1.2	52	692.027	1.730	3.0	84	432.717	1.082	53.0
21	847.050	2.117	14.4	53	689.611	1.724	2.5	85	421.845	1.054	1.2
22	845.037	2.112	113.8	54	630.421	1.576	3.2	86	420.235	1.050	1.7
23	842.621	2.106	1.8	55	625.992	1.565	2.6	87	305.881	0.765	1.7
24	834.567	2.086	1.5	56	623.173	1.558	2.8	88	299.035	0.747	1.8
25	832.152	2.080	4.4	57	621.563	1.554	2.7				
26	824.904	2.062	7.8	58	619.147	1.548	2.5				
27	823.293	2.058	6.3	59	615.925	1.539	2.5				
28	818.864	2.047	8.1	60	613.509	1.533	4.9				
29	817.253	2.043	7.0	61	611.094	1.527	4.3				
30	764.908	1.912	1.3	62	609.080	1.522	4.6				
31	762.492	1.906	1.3	63	606.664	1.516	3.0				
32	759.674	1.899	2.7	64	603.846	1.509	2.6				



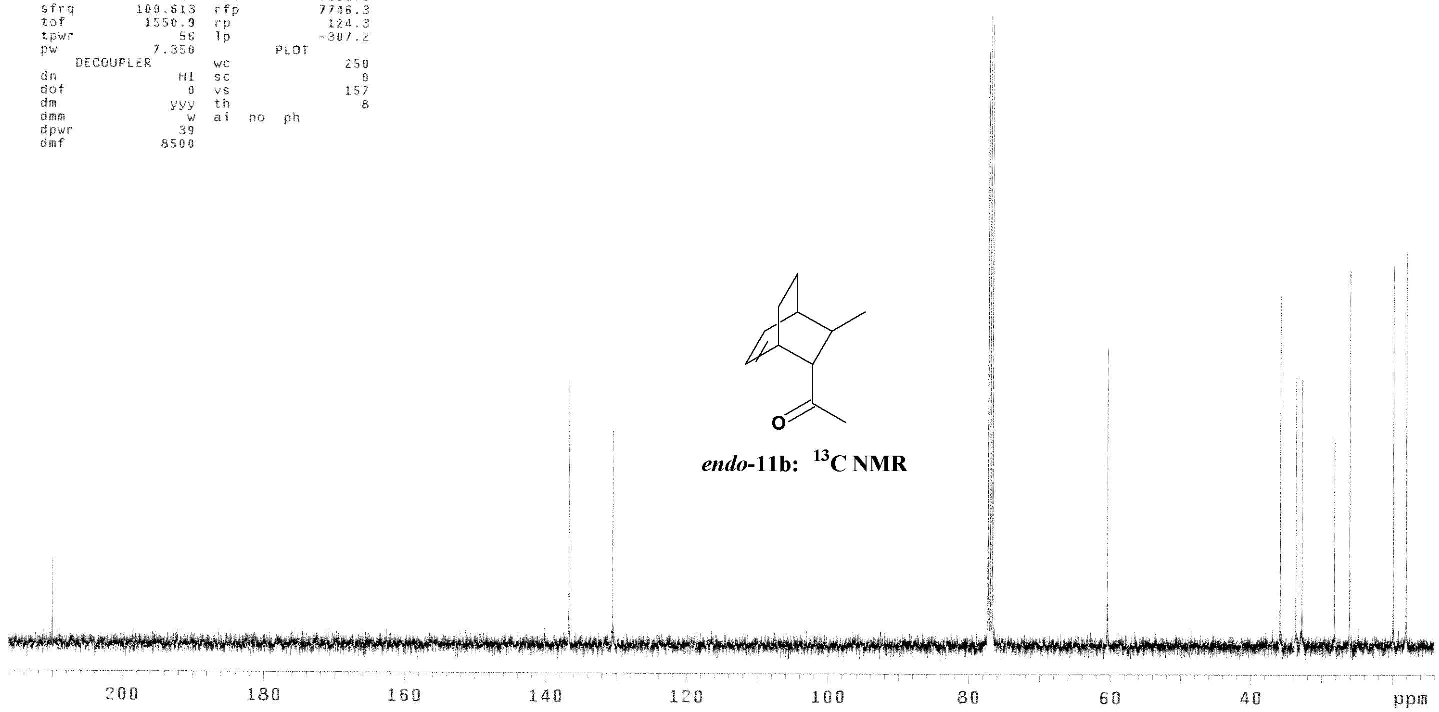
***endo*-11b: ¹H NMR**

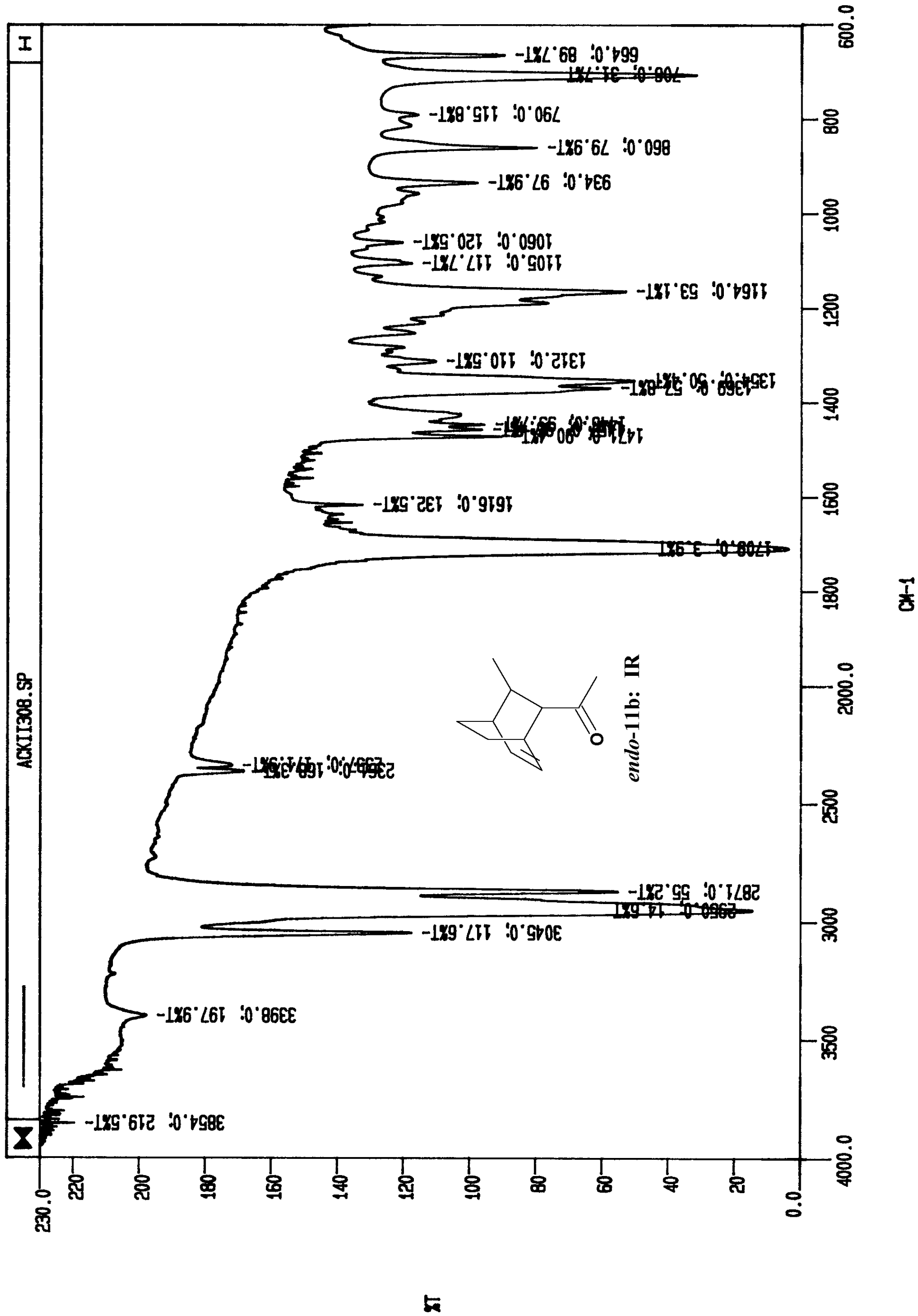


```
ack-ii-30-8 in cdcl3
STANDARD 1H OBSERVE


exp11  s2pul

      SAMPLE                SPECIAL
date   Sep  1 2000  temp       not used
solvent       cdcl3  gain       not used
file /export/home/~ spin             20
vnmr1/vnmrsys/data~ hst           0.008
/ackinsma/acki30_8~ pw90         14.700
      /CARBON.fid   alfa         20.000
   ACQUISITION              FLAGS
sw          25125.6  il               n
at            1.199  in               n
np            60270  dp               y
fb            13800  hs              nn
bs               64        PROCESSING
ss                2  lb            1.00
d1            1.000  fn        not used
nt             1000         DISPLAY
ct             1000  sp          1408.0
   TRANSMITTER       wp         20345.4
tn              C13  rfl         9268.3
sfrq        100.613  rfp         7746.3
tof          1550.9  rp           124.3
tpwr             56  lp          -307.2
pw            7.350           PLOT
    DECOUPLER        wc             250
dn               H1  sc               0
dof               0  vs             157
dm              yyy  th               8
dmm               w  ai  no  ph
dpwr             39
dmf            8500
```

INDEX	FREQUENCY	PPM	HEIGHT
1	21128.458	210.021	14.8
2	13756.474	136.742	46.5
3	13130.001	130.515	37.8
4	7777.760	77.313	104.6
5	7746.321	77.000	110.9
6	7714.116	76.680	109.4
7	6080.839	60.445	52.5
8	3607.153	35.856	61.6
9	3384.782	33.645	47.3
10	3298.134	32.784	46.9
11	2840.356	28.234	36.6
12	2621.819	26.061	66.0
13	2000.713	19.887	66.8
14	1814.382	18.035	69.5

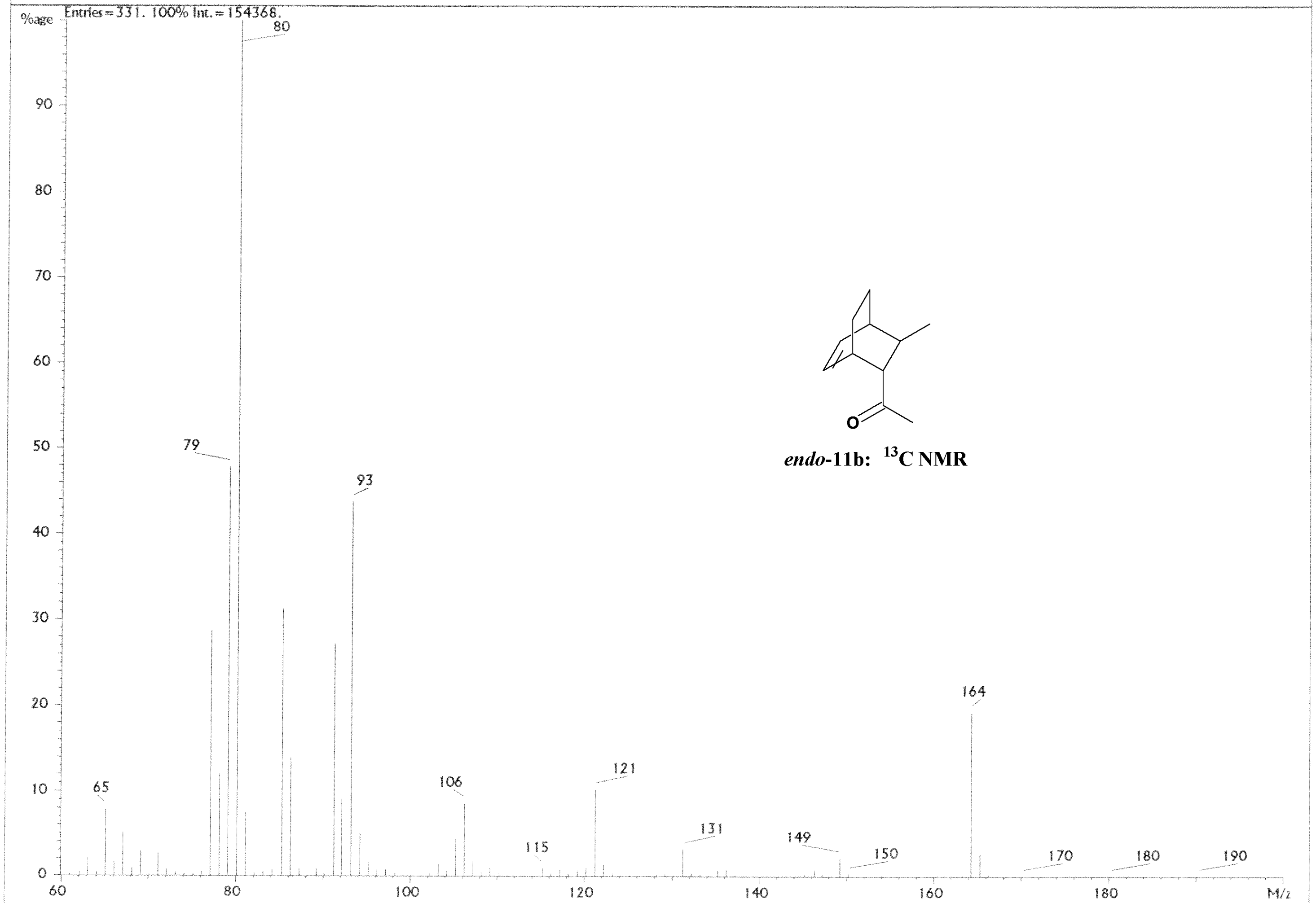
endo-**11b: ^{13}C NMR**





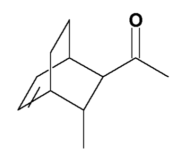
endo-11b: IR

File Name : c:\mswin\data\kin21a.mss
Creation Date/Time : 9/8/00 at 12:35:24
File Type : Lo-Res Mass Spectrum
File Title : Kinsman ACK-II-30-8 glass probe
Source File : c:\mswin\data\kin21.mss
Source Scan(s) : 20:60

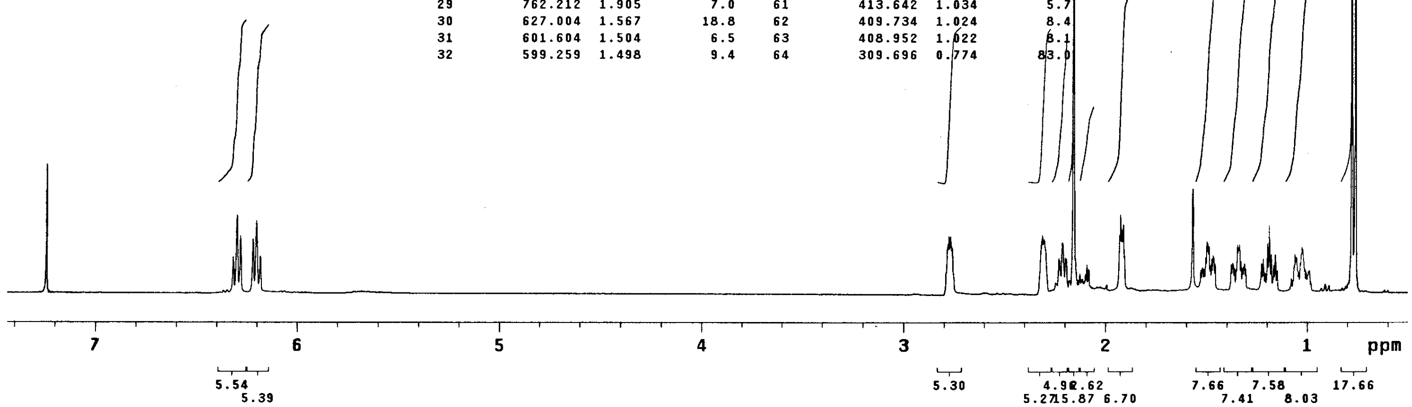


```
ack-ii-30-4 in cdcl3
STANDARD 1H OBSERVE

expl1  s2pul

        SAMPLE                    SPECIAL
date    Aug 22 2000     temp        not used
solvent        cdcl3    gain        not used
file /export/home/~    spin              20
vnmr1/vnmrsys/data~    hst            0.008
/ackinsma/ackii304~    pw90          11.500
          /PROTON.fid  alfa          20.000
     ACQUISITION                 FLAGS
sw          6402.0     il                 n
at           1.995     in                 n
np           25546     dp                 y
fb            3600     hs                nn
bs              16            PROCESSING
ss               2     fn          not used
d1           1.000             DISPLAY
nt              32     sp             200.3
ct              32     wp            2774.5
     TRANSMITTER       rfl           3702.2
tn              H1     rfp           2896.6
sfrq       400.088     rp            -154.7
tof          417.6     lp             -77.5
tpwr            59              PLOT
pw           5.750     wc               250
     DECOUPLER         sc                 0
dn             C13     vs                84
dof              0     th                 5
dm             nnn     ai        ph
dmm              c
dpwr            44
dmf          17100
```

INDEX	FREQUENCY	PPM	HEIGHT
1	2898.964	7.246	6.8
2	2897.400	7.242	18.5
3	2896.619	7.240	23.1
4	2528.119	6.319	6.7
5	2520.304	6.299	14.2
6	2513.270	6.282	10.4
7	2488.260	6.219	9.9
8	2481.617	6.203	13.1
9	2474.192	6.184	6.8
10	1112.736	2.781	9.0
11	1110.391	2.775	10.3
12	1108.047	2.770	9.1
13	1106.874	2.767	10.4
14	1104.921	2.762	8.2
15	1103.748	2.759	8.4
16	924.383	2.310	10.5
17	920.866	2.302	9.8
18	918.912	2.297	8.4
19	891.558	2.228	6.4
20	884.915	2.212	9.3
21	877.881	2.194	6.5
22	861.859	2.154	165.0
23	859.124	2.147	6.5
24	836.459	2.091	5.3
25	771.981	1.930	10.5
26	770.027	1.925	14.2
27	767.292	1.918	11.5
28	764.557	1.911	12.5
29	762.212	1.905	7.0
30	627.004	1.567	18.8
31	601.604	1.504	6.5
32	599.259	1.498	9.4
33	597.305	1.493	9.1
34	595.351	1.488	8.6
35	592.616	1.481	5.2
36	589.490	1.473	6.3
37	587.145	1.468	7.0
38	585.582	1.464	6.7
39	583.237	1.458	5.5
40	550.803	1.377	5.3
41	548.459	1.371	5.6
42	546.896	1.367	5.5
43	538.298	1.345	8.9
44	536.345	1.341	8.4
45	534.391	1.336	8.9
46	532.046	1.330	5.9
47	526.184	1.315	4.9
48	524.621	1.311	5.5
49	490.233	1.225	5.5
50	487.107	1.218	6.3
51	478.510	1.196	9.0
52	474.993	1.187	12.2
53	471.085	1.177	7.1
54	466.396	1.166	5.1
55	462.879	1.157	7.1
56	425.756	1.064	5.1
57	423.802	1.059	7.1
58	422.239	1.055	6.5
59	421.457	1.053	6.6
60	420.676	1.051	6.4
61	413.642	1.034	5.7
62	409.734	1.024	8.4
63	408.952	1.022	6.1
64	309.696	0.774	83.0
65	303.053	0.757	84.1

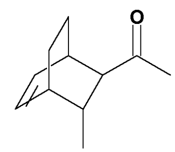


exo-11b: ^{1}H NMR

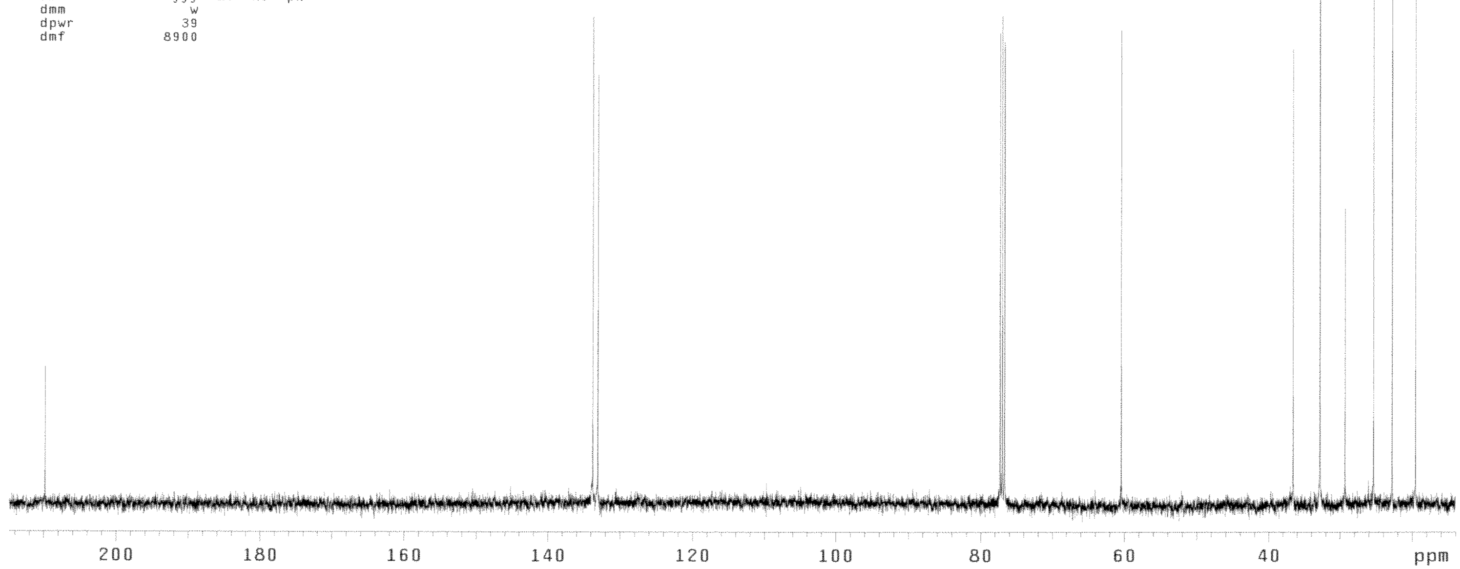


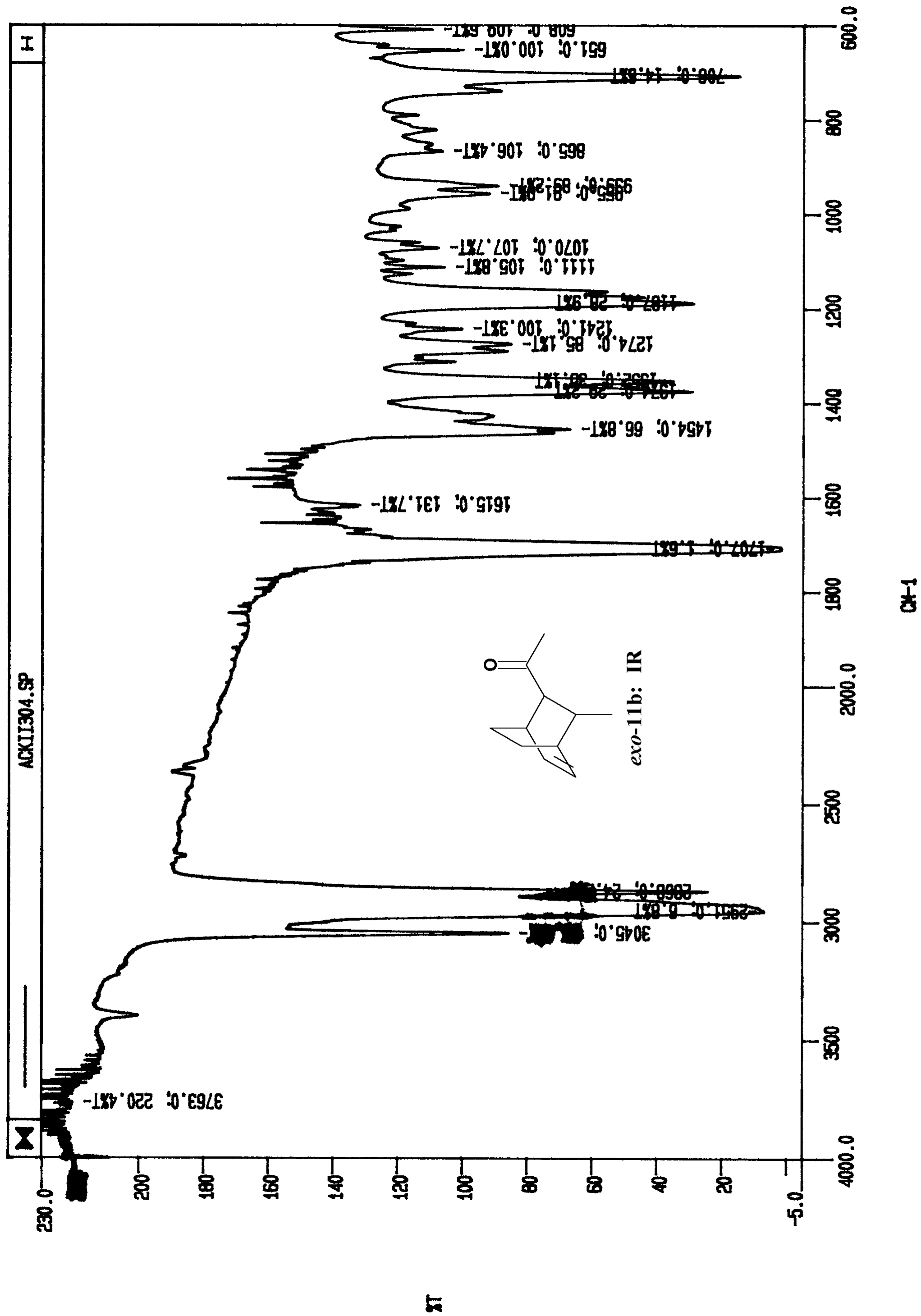
```
ack-ii-30-4 in cdcl3
13C OBSERVE

pad=2 run with gradshim before acquisiti
on

exp11  s2pul

      SAMPLE                SPECIAL
date   Aug 22 2000  temp      not used
solvent      cdcl3  gain      not used
file /export/home/~ spin            20
vnmr1/vnmrsys/data~ hst          0.008
/ackinsma/ackii304~ pw90        10.600
/auto_22Aug2000/CA~ alfa        20.000
         RBON.fid            FLAGS
   ACQUISITION      il               n
sw         25125.6  in               n
at           1.199  dp               y
np           60270  hs              nn
fb           13800        PROCESSING
bs              64  lb            1.00
ss               2  fn        not used
d1           1.000          DISPLAY
nt            1000  sp          1407.2
ct             384  wp         20199.7
   TRANSMITTER      rfl         9269.0
tn             C13  rfp         7746.3
sfrq       100.613  rp           103.5
tof         1550.9  lp          -297.7
tpwr            59             PLOT
pw           5.300  wc             250
    DECOUPLER       sc               0
dn              H1  vs              84
dof              0  th               9
dm             yyy  ai  no  ph
dmm              w
dpwr            39
dmf           8900
```

INDEX	FREQUENCY	PPM	HEIGHT
1	21109.288	209.831	23.6
2	13455.890	133.754	84.3
3	13385.344	133.053	74.2
4	7778.527	77.320	81.4
5	7746.321	77.000	84.5
6	7714.116	76.680	80.0
7	6079.305	60.430	82.0
8	3679.998	36.580	78.8
9	3302.734	32.830	105.8
10	2949.241	29.316	51.1
11	2551.274	25.360	105.7
12	2292.863	22.792	102.5
13	1966.974	19.552	104.6



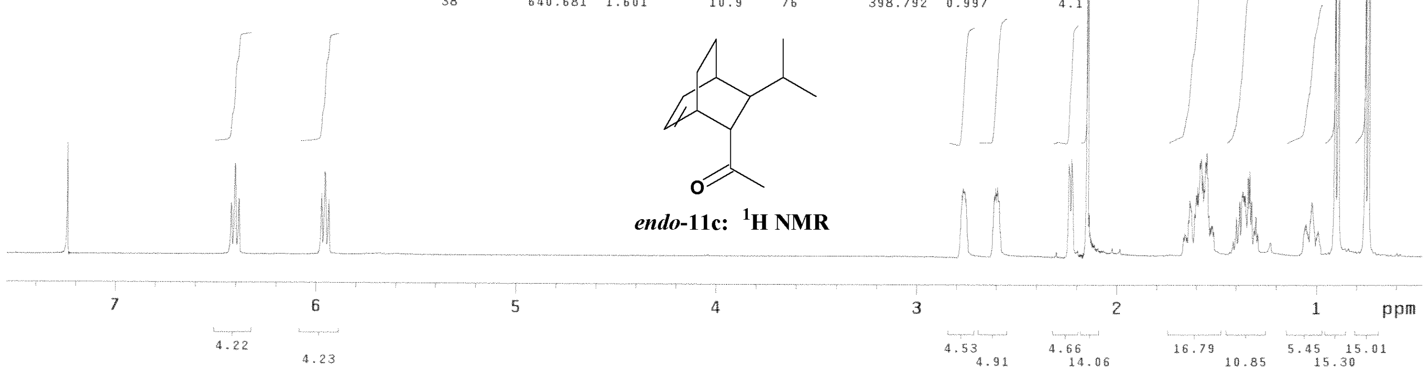
exo-**11b**: ^{13}C NMR




ACKII304.SP
exo-11b: IR
CM-1
%T

```
ack-ii-8-4 in cdcl3
STANDARD 1H OBSERVE

expl1  s2pul

      SAMPLE                SPECIAL
date   Aug 22 2000   temp       not used
solvent       cdcl3  gain       not used
file /export/home/~  spin             20
vnmr1/vnmrsys/data~  hst           0.008
/ackinsma/ackii84/~  pw90         11.500
        PROTON.fid   alfa         20.000
   ACQUISITION               FLAGS
sw           6402.0  il                n
at            1.995  in                n
np            25546  dp                y
fb             3600  hs               nn
bs               16         PROCESSING
ss                2  fn         not used
d1            1.000          DISPLAY
nt               32  sp            187.4
ct               32  wp           2831.5
   TRANSMITTER       rfl          3702.2
tn               H1  rfp          2896.6
sfrq        400.088  rp           -156.8
tof           417.6  lp            -68.3
tpwr             59          PLOT
pw            5.750  wc              250
    DECOUPLER        sc                0
dn              C13  vs               71
dof               0  th                3
dm              nnn  ai   cdc  ph
dmm               c
dpwr             44
dmf           17100
```

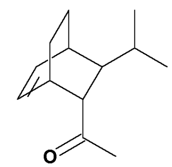
INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	2897.400	7.242	16.6	39	637.555	1.594	12.4	77	397.620	0.994	4.8
2	2896.619	7.240	19.6	40	632.866	1.582	16.6	78	396.057	0.990	4.2
3	2569.541	6.422	9.1	41	630.912	1.577	17.2	79	362.450	0.906	89.0
4	2562.116	6.404	16.0	42	627.395	1.568	14.4	80	355.807	0.889	81.0
5	2554.692	6.385	9.7	43	624.660	1.561	11.9	81	301.880	0.755	88.1
6	2389.394	5.972	10.8	44	621.533	1.554	16.7	82	295.237	0.738	81.9
7	2382.751	5.956	14.6	45	619.189	1.548	18.4				
8	2374.936	5.936	9.8	46	617.626	1.544	13.9				
9	1108.828	2.771	10.8	47	614.890	1.537	7.3				
10	1107.656	2.769	11.3	48	611.764	1.529	5.2				
11	1106.874	2.767	12.0	49	609.419	1.523	5.6				
12	1104.530	2.761	11.4	50	607.465	1.518	5.4				
13	1103.358	2.758	11.4	51	605.121	1.512	4.5				
14	1102.576	2.756	11.1	52	559.400	1.398	7.0				
15	1101.404	2.753	10.2	53	552.757	1.382	9.9				
16	1043.960	2.609	10.8	54	548.068	1.370	11.0				
17	1041.615	2.603	12.0	55	546.505	1.366	11.5				
18	1040.443	2.601	11.0	56	544.551	1.361	10.8				
19	1038.098	2.595	12.2	57	541.815	1.354	10.8				
20	1035.754	2.589	9.9	58	535.954	1.340	14.1				
21	1034.972	2.587	9.8	59	532.828	1.332	15.2				
22	1032.237	2.580	4.4	60	528.920	1.322	9.8				
23	895.466	2.238	16.3	61	524.231	1.310	5.2				
24	893.903	2.234	14.4	62	520.714	1.302	7.0				
25	889.995	2.225	17.3	63	516.806	1.292	4.2				
26	857.952	2.144	169.0	64	425.365	1.063	4.3				
27	855.607	2.139	7.6	65	423.411	1.058	5.1				
28	853.653	2.134	4.3	66	421.848	1.054	5.7				
29	852.090	2.130	3.4	67	421.066	1.052	5.8				
30	665.691	1.664	3.6	68	419.112	1.048	4.5				
31	662.955	1.657	4.0	69	417.940	1.045	4.1				
32	659.438	1.648	3.4	70	415.986	1.040	3.6				
33	655.921	1.639	6.4	71	412.860	1.032	6.7				
34	653.186	1.633	9.9	72	410.906	1.027	8.1				
35	651.623	1.629	9.0	73	409.343	1.023	9.8				
36	650.060	1.625	8.2	74	407.780	1.019	8.9				
37	643.417	1.608	7.6	75	400.746	1.002	3.4				
38	640.681	1.601	10.9	76	398.792	0.997	4.1				



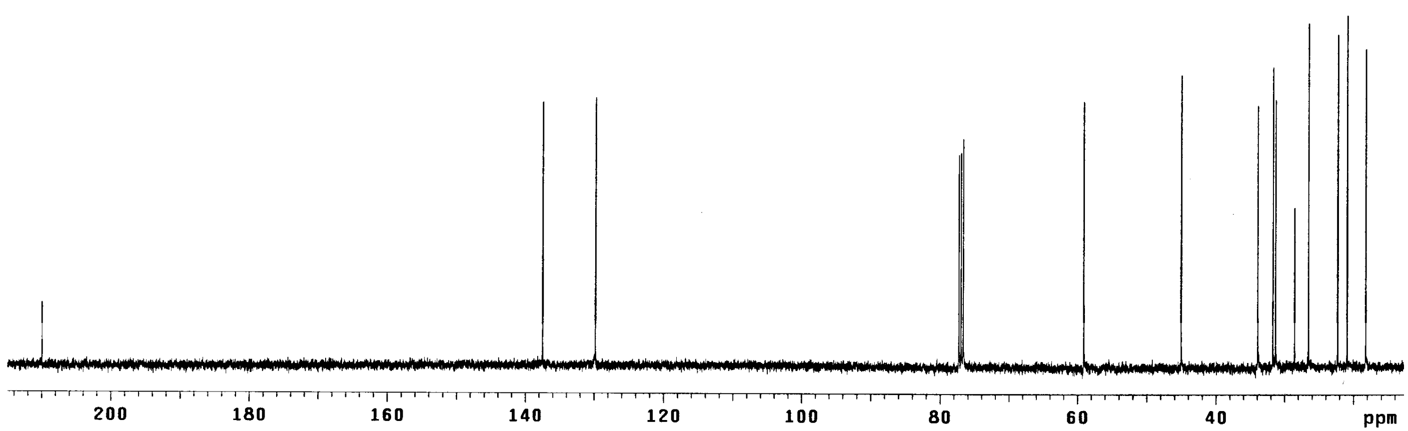
endo-11c: ^{1}H NMR

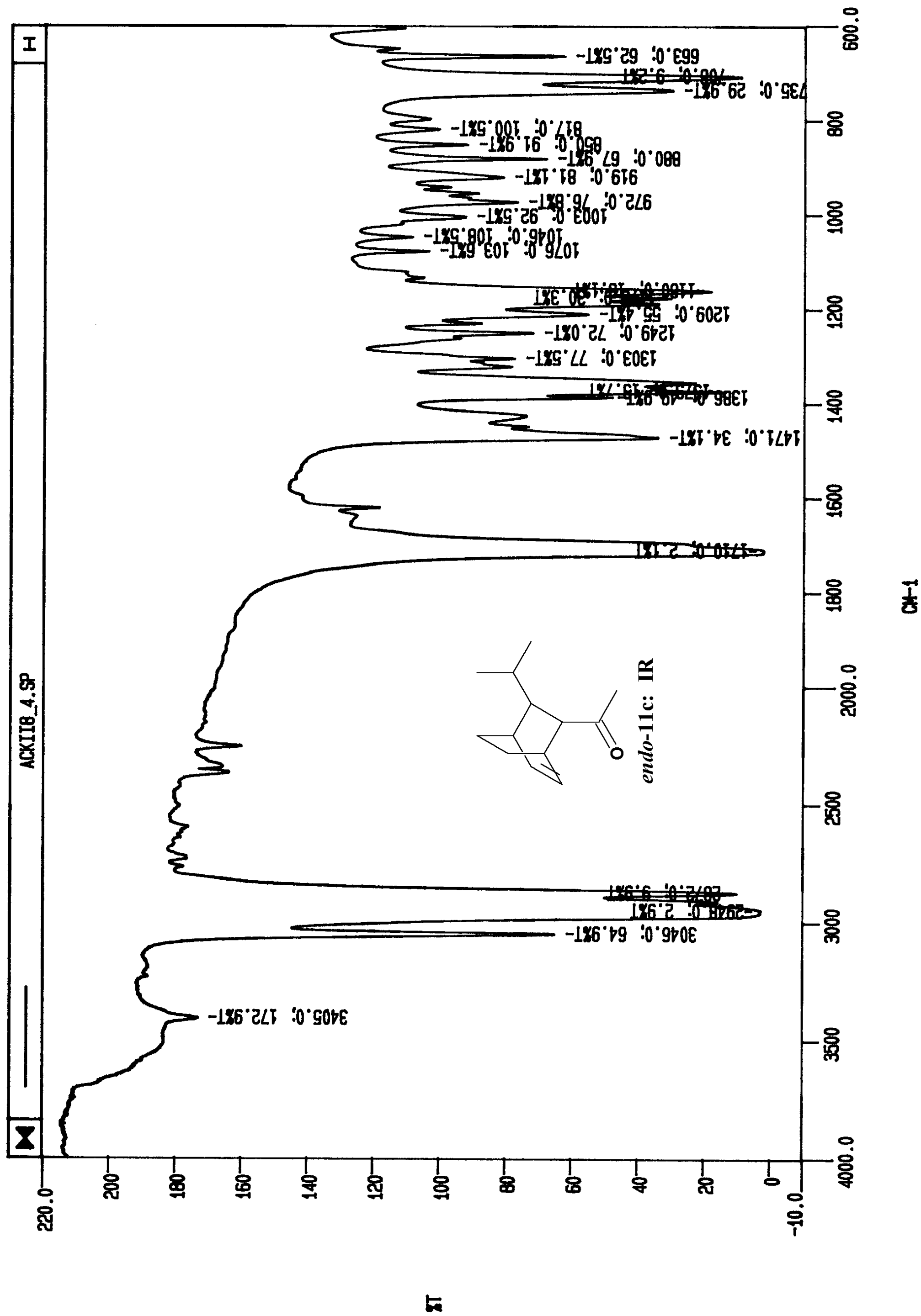
```
ack-ii-8-4 in cdcl3
13C OBSERVE

pad=2 run with gradshim before acquisiti
on

expl1  s2pul

      SAMPLE                 SPECIAL
date   Aug 24 2000  temp       not used
solvent       cdcl3  gain       not used
file /export/home/~  spin             20
vnmr1/vnmrsys/data~  hst           0.008
/ackinsma/ackii84/~  pw90         14.700
auto_24Aug2000/CAR~  alfa         20.000
          BON.fid            FLAGS
   ACQUISITION        il                 n
sw          25125.6   in                 n
at            1.199   dp                 y
np            60270   hs                nn
fb            13800         PROCESSING
bs               64   lb              1.00
ss                2   fn          not used
d1            1.000          DISPLAY
nt              512   sp            1280.7
ct              192   wp           20345.4
   TRANSMITTER        rfl           9270.6
tn              C13   rfp           7746.3
sfrq        100.613   rp             110.5
tof          1550.9   lp            -292.7
tpwr             56            PLOT
pw            7.350   wc               250
    DECOUPLER         sc                 0
dn               H1   vs                39
dof               0   th                 7
dm              yyy   ai   no  ph
dmm               w
dpwr             39
dmf            8500
```

INDEX	FREQUENCY	PPM	HEIGHT
1	21123.090	209.968	11.1
2	13832.387	137.497	47.2
3	13058.689	129.806	47.9
4	7777.760	77.313	37.9
5	7746.321	77.000	38.2
6	7714.116	76.680	40.7
7	5945.116	59.096	47.4
8	4534.210	45.071	52.2
9	3414.687	33.943	46.7
10	3192.316	31.732	53.7
11	3153.976	31.351	47.8
12	2873.328	28.561	28.5
13	2670.127	26.542	61.6
14	2244.555	22.311	59.5
15	2107.298	20.947	63.0
16	1835.852	18.249	56.9

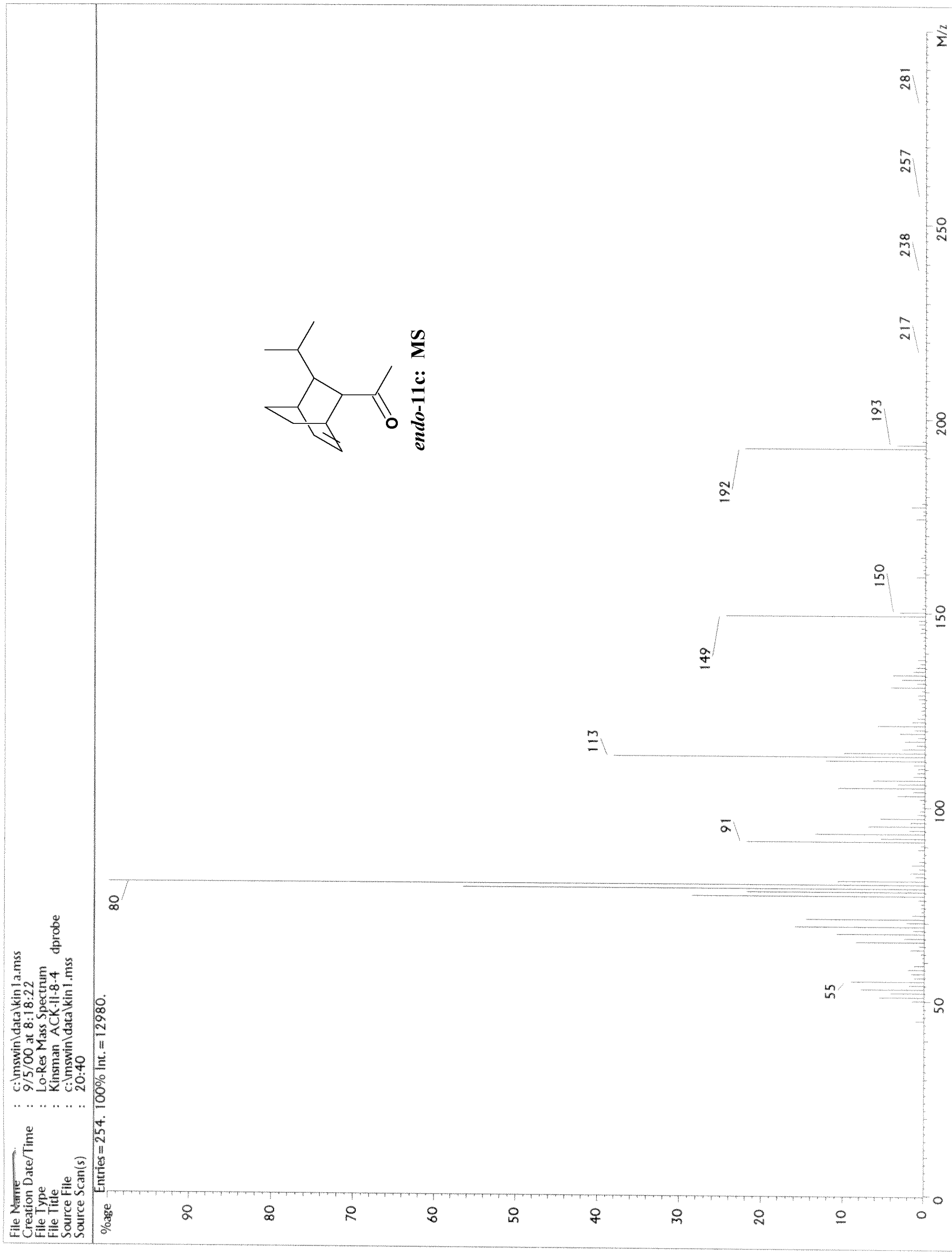


endo-11c: ^{13}C NMR



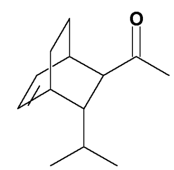

ACKII8_4.SP
%T
CM-1
3405.0; 172.9%T-
3046.0; 64.9%T-
2948.0; 2.9%T
2872.0; 9.9%T
1710.0; 2.1%T
1471.0; 34.1%T-
1386.0; 40.9%T
1303.0; 77.5%T-
1249.0; 72.0%T-
1209.0; 55.4%T-
1076.0; 103.6%T-
1046.0; 108.5%T-
1003.0; 92.5%T-
972.0; 76.8%T-
919.0; 81.1%T-
880.0; 67.9%T-
850.0; 91.9%T-
817.0; 100.5%T-
735.0; 29.9%T-
708.0; 9.2%T
663.0; 62.5%T-
O

endo-11c: IR

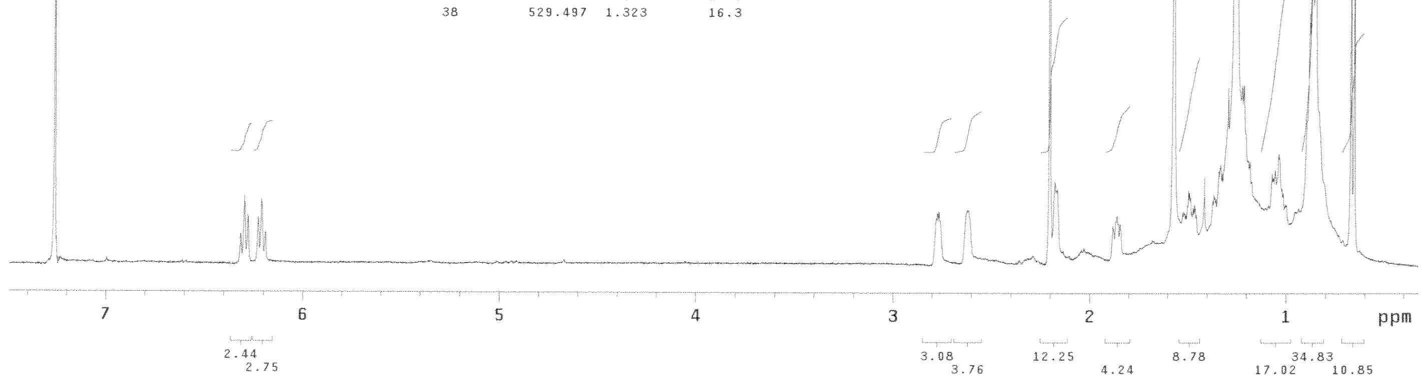

File Name : c:\mswin\data\kin1a.mss
Creation Date/Time : 9/5/00 at 8:18:22
File Type : Lo-Res Mass Spectrum
File Title : Kinsman ACK-II-8-4 dprobe
Source File : c:\mswin\data\kin1.mss
Source Scan(s) : 20:40
Entries=254. 100% Int.=12980.
%age
M/z
80
55
91
113
149
150
192
193
217
238
257
281
O
endo-11c: MS

```
ack-ii-8-7 in cdcl3
STANDARD 1H OBSERVE

exp11  s2pul

     SAMPLE                 SPECIAL
date   Aug 24 2000   temp        not used
solvent      cdcl3   gain        not used
file /export/home/~  spin              20
vnmr1/vnmrsys/data~  hst            0.008
/ackinsma/upto_aug~  pw90          14.200
24_2000/ackii87/au~  alfa          20.000
to_24Aug2000/PROTO~          FLAGS
             N.fid   il                 n
   ACQUISITION       in                 n
sw          6402.0   dp                 y
at           1.995   hs                nn
np           25546         PROCESSING
fb            3600   fn          not used
bs              16           DISPLAY
ss               2   sp             130.1
d1           1.000   wp            2866.7
nt              32   rfl           3702.6
ct              32   rfp           2904.6
   TRANSMITTER       rp            -161.7
tn              H1   lp             -74.8
sfrq       400.088           PLOT
tof          417.6   wc               250
tpwr            57   sc                 0
pw           7.100   vs               394
    DECOUPLER        th                 4
dn             C13   ai     ph
dof              0
dm             nnn
dmm              c
dpwr            44
dmf          15900
```

INDEX	FREQUENCY	PPM	HEIGHT
1	2904.621	7.260	110.9
2	2902.667	7.255	5.6
3	2526.742	6.316	5.3
4	2518.927	6.296	12.1
5	2512.284	6.279	8.6
6	2491.573	6.228	8.3
7	2484.539	6.210	11.5
8	2477.114	6.191	5.6
9	1108.624	2.771	9.2
10	1105.888	2.764	9.6
11	1048.445	2.621	9.7
12	880.412	2.201	131.8
13	870.642	2.176	14.8
14	869.470	2.173	14.5
15	865.562	2.163	13.5
16	752.629	1.881	7.0
17	743.641	1.859	8.9
18	738.170	1.845	7.4
19	670.957	1.677	4.5
20	627.581	1.569	103.6
21	609.996	1.525	9.2
22	607.652	1.519	9.6
23	603.353	1.508	9.1
24	597.882	1.494	13.4
25	595.928	1.490	12.9
26	593.975	1.485	12.0
27	591.630	1.479	9.5
28	588.504	1.471	10.2
29	586.159	1.465	10.7
30	584.205	1.460	10.1
31	581.861	1.454	9.0
32	565.057	1.412	15.7
33	548.645	1.371	11.4
34	546.300	1.365	12.6
35	542.002	1.355	11.8
36	536.140	1.340	16.9
37	532.232	1.330	17.9
38	529.497	1.323	16.3
39	516.601	1.291	31.6
40	500.970	1.252	162.2
41	488.466	1.221	31.9
42	484.949	1.212	32.0
43	473.225	1.183	18.4
44	434.539	1.086	10.7
45	427.896	1.070	16.2
46	425.160	1.063	15.5
47	422.034	1.055	16.9
48	414.219	1.035	19.9
49	398.978	0.997	10.6
50	401.714	1.004	10.8
51	375.141	0.938	10.1
52	350.913	0.877	44.8
53	345.833	0.864	98.9
54	339.190	0.848	82.3
55	283.700	0.709	4.5
56	267.288	0.668	64.1
57	260.644	0.651	57.3

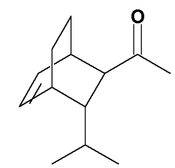


exo-**11c**: ^{1}H NMR

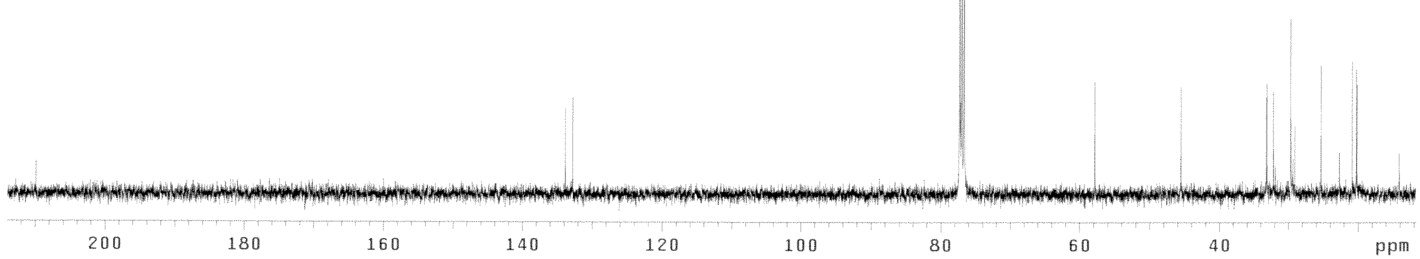


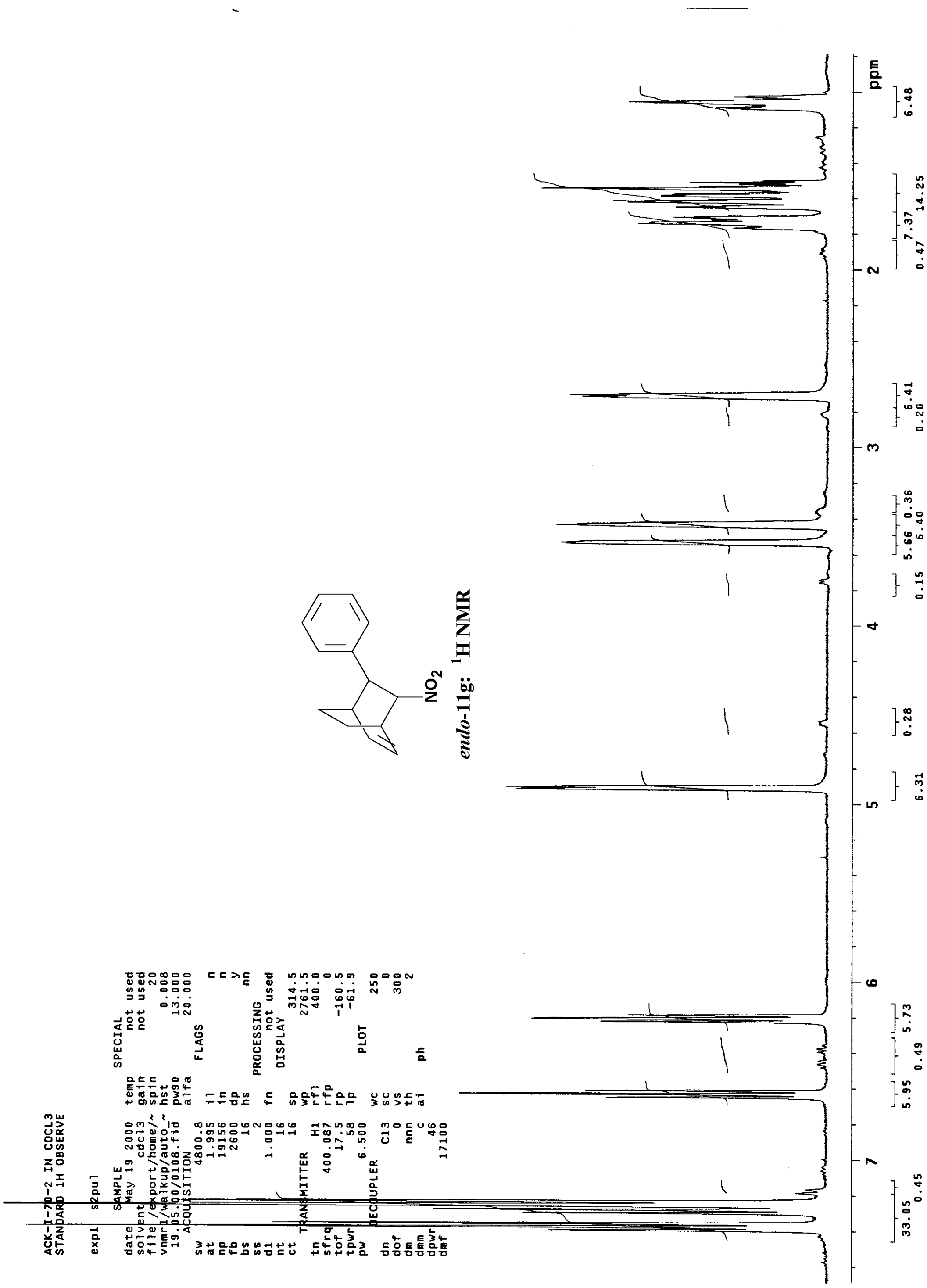
```
ack-ii-8-7 in cdcl3
13C OBSERVE

pad=2 run with gradshim before acquisiti
on

exp11  s2pul

      SAMPLE              SPECIAL
date   Aug 23 2000  temp       not used
solvent      cdcl3  gain       not used
file /export/home/~ spin             20
vnmr1/vnmrsys/data~ hst           0.008
/ackinsma/ackii87/~ pw90         14.700
        CARBON.fid  alfa         20.000
   ACQUISITION             FLAGS
sw         25125.6  il                n
at           1.199  in                n
np           60270  dp                y
fb           13800  hs               nn
bs              64       PROCESSING
ss               2  lb             1.00
d1           1.000  fn         not used
nt            5000        DISPLAY
ct            5000  sp           1180.2
   TRANSMITTER      wp          20345.4
tn             C13  rfl          9267.5
sfrq       100.613  rfp          7746.3
tof         1550.9  rp             99.8
tpwr            56  lp           -271.4
pw           7.350          PLOT
    DECOUPLER       wc              250
dn              H1  sc                0
dof              0  vs              332
dm             yyy  th                5
dmm              w  ai  no  ph
dpwr            39
dmf           8500
```

INDEX	FREQUENCY	PPM	HEIGHT
1	21116.189	209.899	5.9
2	13464.324	133.838	15.3
3	13356.973	132.771	17.2
4	7778.527	77.320	287.2
5	7767.025	77.206	16.4
6	7746.321	77.000	293.0
7	7714.883	76.687	280.0
8	5819.361	57.846	20.0
9	4581.751	45.544	19.2
10	3341.841	33.219	17.3
11	3332.639	33.127	19.8
12	3237.557	32.182	18.2
13	3210.719	31.915	5.0
14	2986.814	29.690	31.2
15	2953.842	29.362	6.5
16	2926.237	29.087	12.4
17	2547.439	25.322	22.9
18	2282.128	22.685	7.4
19	2098.097	20.856	23.6
20	2038.286	20.261	22.2
21	2029.852	20.177	19.6
22	1421.015	14.125	7.4



exo-11c: ^{13}C NMR

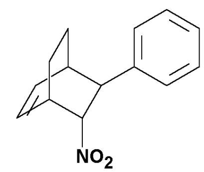




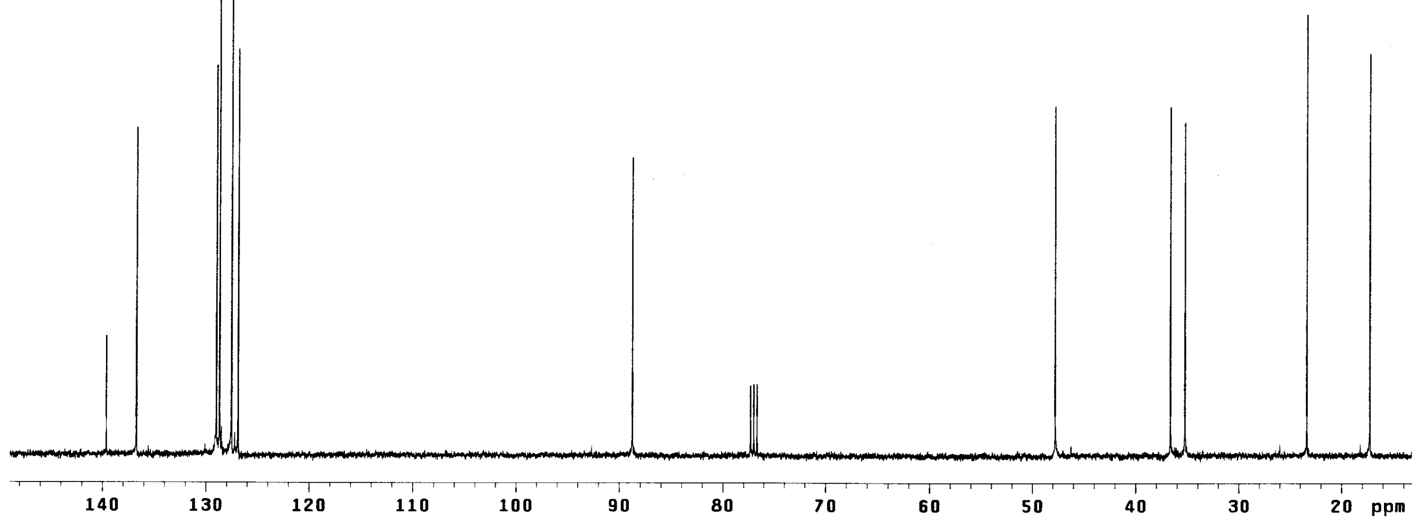
endo-11g: ^{1}H NMR

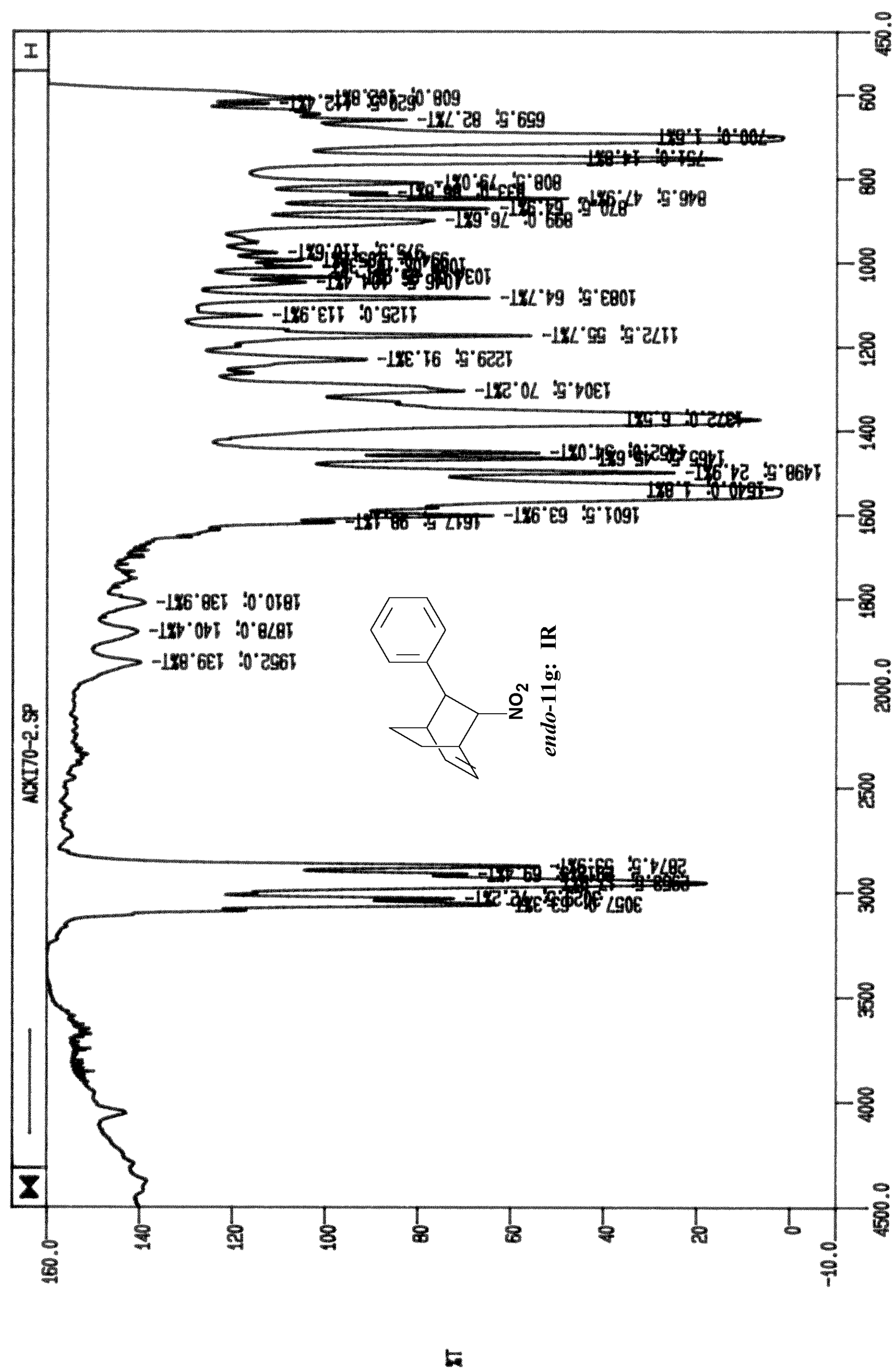
```
ack-i-70-2 in cdcl3
13C OBSERVE

pad=2 run with gradshim before acquisiti
on

exp4  s2pul

      SAMPLE                SPECIAL
date   May 23 2000  temp       not used
solvent      cdcl3  gain       not used
file /export/home/~ spin             20
vnmr1/walkup/auto_~ hst           0.008
 23.05.00/0303.fid  pw90         11.700
   ACQUISITION      alfa         20.000
sw         21141.6          FLAGS
at           1.199  il                n
np           50714  in                n
fb           11600  dp                y
bs              64  hs               nn
ss               2        PROCESSING
d1           1.000  lb             1.00
nt             256  fn         not used
ct              64         DISPLAY
   TRANSMITTER      sp           1330.3
tn             C13  wp          13650.7
sfrq       100.611  rfl          8799.4
tof           41.9  rfp          7746.3
tpwr            59  rp             91.2
pw           5.850  lp           -250.8
    DECOUPLER               PLOT
dn              H1  wc              250
dof              0  sc                0
dm             yyy  vs               31
dmm              w  th                5
dpwr            40  ai   no  ph
dmf           9700
```

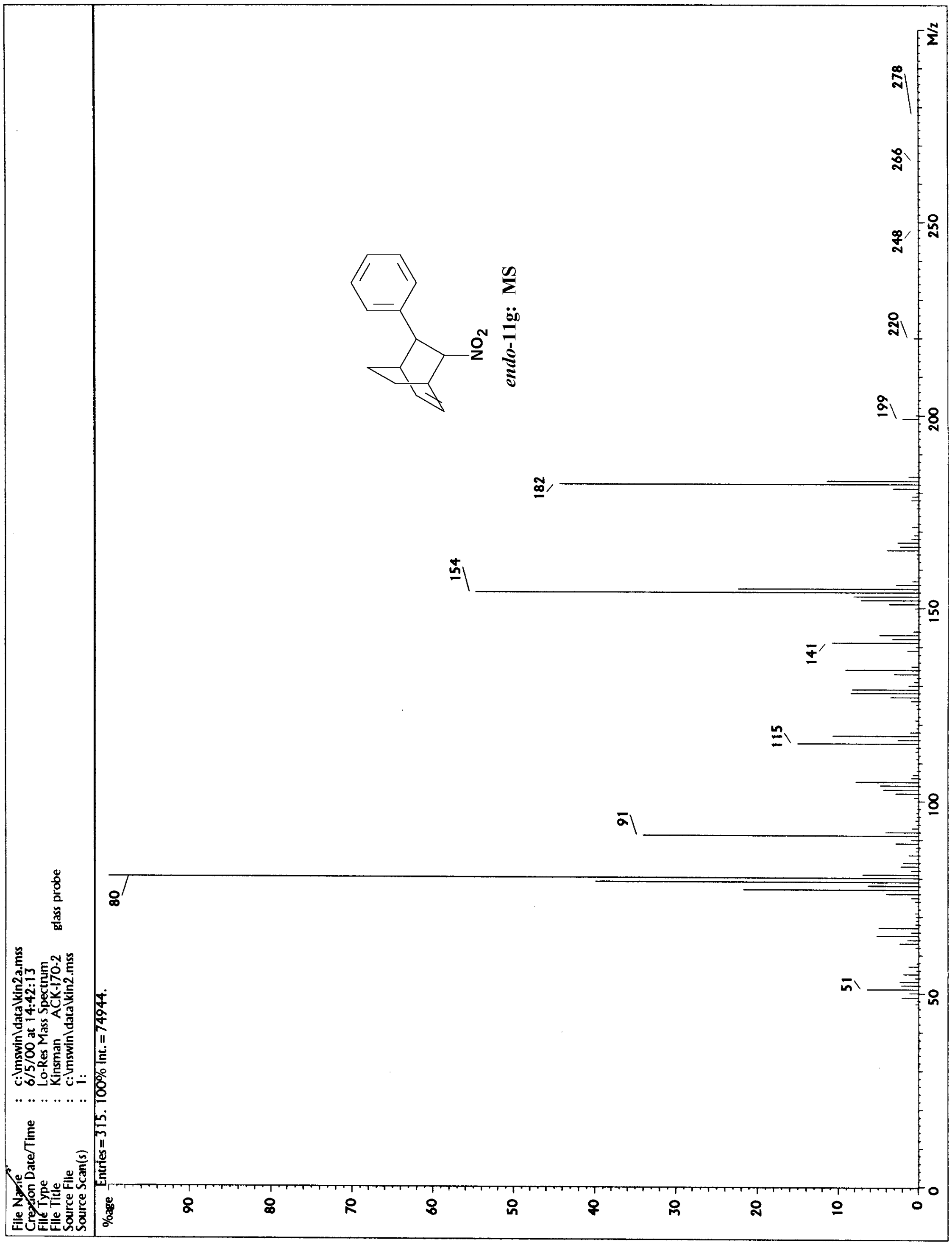
INDEX	FREQUENCY	PPM	HEIGHT
1	14047.459	139.635	21.2
2	13754.533	136.723	58.3
3	12977.053	128.995	69.5
4	12946.728	128.693	126.0
5	12928.017	128.507	5.1
6	12829.299	127.526	119.8
7	12764.778	126.884	72.4
8	8927.060	88.737	53.0
9	7777.938	77.314	12.4
10	7746.322	77.000	12.6
11	7714.062	76.679	12.6
12	4807.383	47.786	62.3
13	3688.587	36.665	62.2
14	3547.285	35.261	59.4
15	2351.063	23.370	78.7
16	1738.757	17.284	71.7



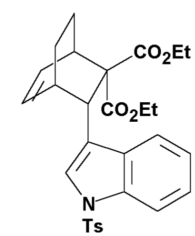
endo-**11g**: ^{13}C NMR



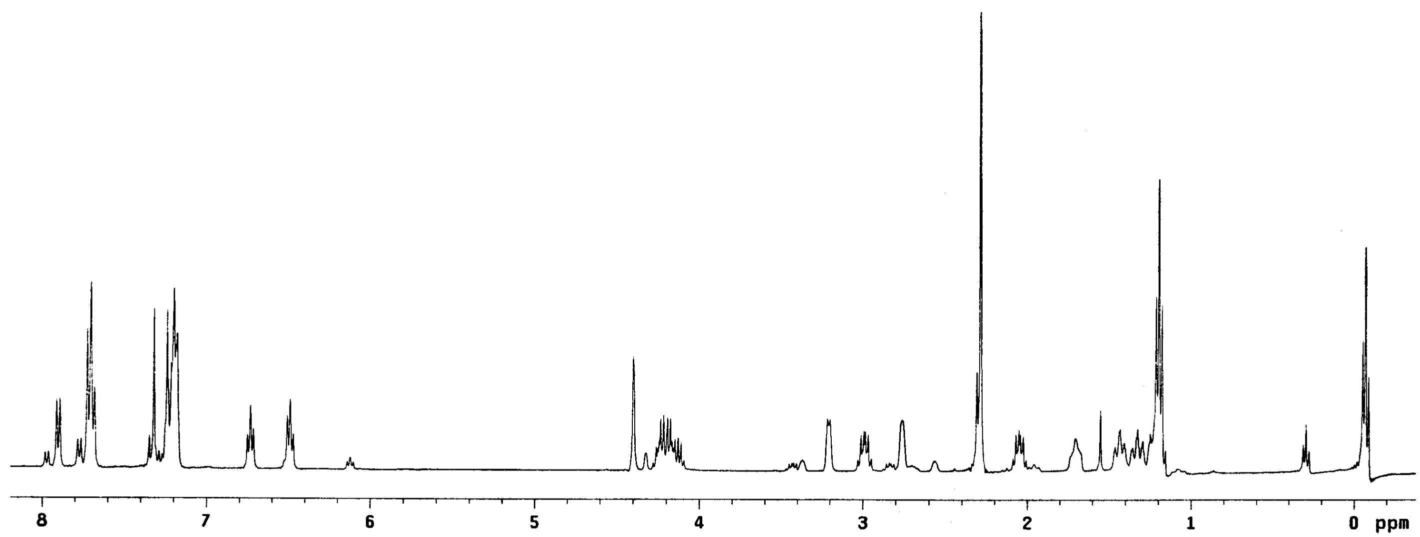

endo-11g: IR


File Name : c:\mswin\data\kin2a.mss
Creation Date/Time : 6/5/00 at 14:42:13
File Type : Lo-Res Mass Spectrum
File Title : Kinsman ACK-170-2 glass probe
Source File : c:\mswin\data\kin2.mss
Source Scan(s) : 1:
Entries=315. 100% Int.=74944.
%age
M/z
NO2
endo-11g: MS
80
91
115
141
154
182
199
220
248
266
278
51

```
ack-ii-15-3 in cdcl3
STANDARD 1H OBSERVE

expl1  s2pul

      SAMPLE              SPECIAL
date    Jul 19 2000   temp      not used
solvent       cdcl3   gain      not used
file /export/home/~   spin            20
vnmr1/vnmrsys/data~   hst          0.008
/ackinsma/ackii153~   pw90        12.700
        /PROTON.fid   alfa        20.000
   ACQUISITION             FLAGS
sw           6402.0   il               n
at            1.995   in               n
np            25546   dp               y
fb             3600   hs              nn
bs               16         PROCESSING
ss                2   fn        not used
d1            1.000          DISPLAY
nt               32   sp          -151.4
ct               32   wp          3431.0
   TRANSMITTER        rfl         3704.5
tn               H1   rfp         2896.6
sfrq        400.088   rp          -143.9
tof           417.6   lp           -95.1
tpwr             58            PLOT
pw            6.350   wc             250
    DECOUPLER         sc               0
dn              C13   vs             132
dof               0   th               3
dm              nnn   ai     ph
dmm               c
dpwr             45
dmf           18500
```

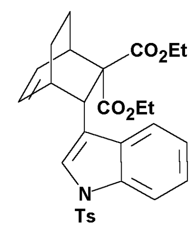


endo-11h (major isomer): ^{1}H NMR

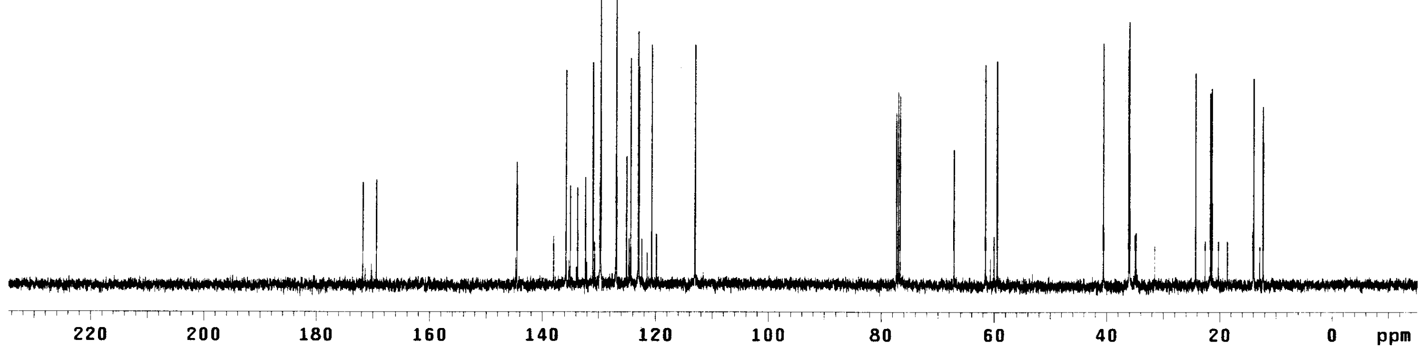


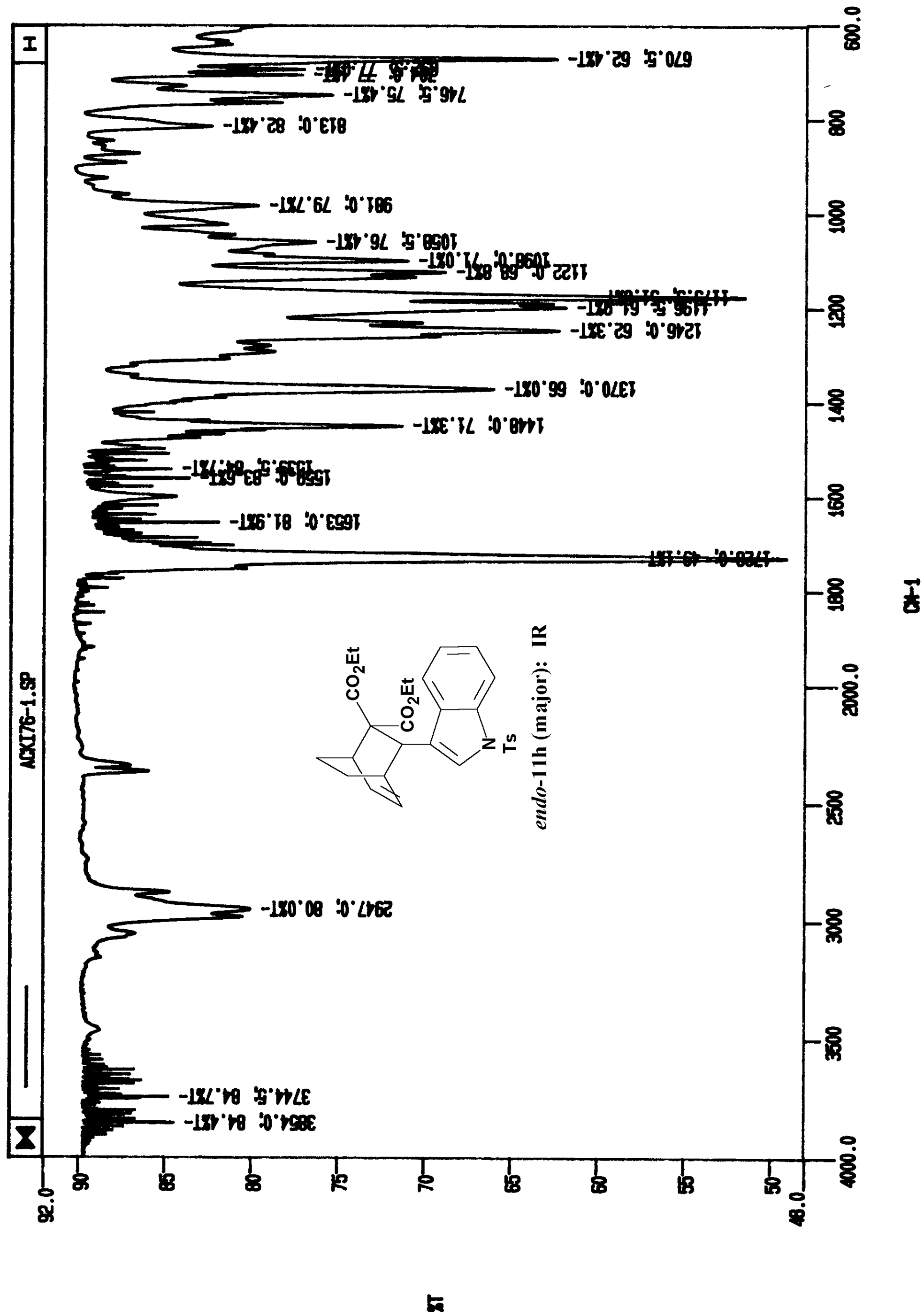
```
ack-ii-15-1 in cdcl3
13C OBSERVE

pad=2 run with gradshim before acquisiti
on

expl  s2pul

    SAMPLE                  SPECIAL
date   Jul 12 2000   temp       not used
solvent       cdcl3  gain       not used
file /export/home/~  spin             20
vnmr1/vnmrsys/data~  hst           0.008
/ackinsma/ackii151~  pw90         11.700
     c/CARBON.fid    alfa         20.000
   ACQUISITION               FLAGS
sw          25125.6  il                n
at            1.199  in                n
np            60270  dp                y
fb            13800  hs               nn
bs               64        PROCESSING
ss                2  lb             1.00
d1            1.000  fn         not used
nt              256         DISPLAY
ct              128  sp          -1527.3
   TRANSMITTER       wp          25125.6
tn              C13  rfl          9273.6
sfrq        100.613  rfp          7746.3
tof          1550.9  rp            110.9
tpwr             59  lp           -300.8
pw            5.850             PLOT
    DECOUPLER        wc              250
dn               H1  sc                0
dof               0  vs               36
dm              yyy  th                3
dmm               w  ai   no  ph
dpwr             39
dmf            8500
```

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	17278.372	171.750	18.0	33	6756.387	67.160	23.8
2	17127.313	170.249	3.5	34	6192.025	61.550	38.8
3	17040.665	169.388	18.4	35	6103.076	60.666	4.5
4	14561.611	144.745	4.8	36	6040.199	60.041	8.4
5	14539.374	144.524	21.6	37	5981.922	59.462	39.4
6	13879.162	137.962	8.5	38	4081.800	40.574	42.6
7	13656.024	135.744	37.8	39	3633.224	36.115	41.5
8	13606.182	135.248	4.2	40	3619.422	35.978	46.4
9	13579.344	134.981	17.4	41	3614.054	35.924	13.1
10	13448.989	133.686	17.1	42	3518.971	34.979	8.7
11	13308.665	132.291	18.9	43	3500.568	34.796	9.1
12	13284.894	132.055	3.9	44	3169.312	31.504	6.8
13	13176.776	130.980	39.2	45	2442.389	24.278	37.3
14	13146.871	130.683	7.5	46	2271.393	22.578	7.7
15	13051.788	129.737	18.1	47	2176.310	21.633	33.8
16	13040.286	129.623	78.0	48	2156.374	21.435	8.0
17	12765.773	126.894	84.0	49	2150.239	21.374	34.6
18	12758.105	126.818	18.9	50	2034.453	20.223	7.6
19	12584.042	125.088	22.6	51	1874.192	18.630	7.5
20	12541.102	124.661	8.0	52	1414.881	14.064	9.3
21	12509.663	124.349	39.9	53	1403.379	13.950	36.3
22	12376.240	123.022	44.7	54	1293.727	12.860	6.6
23	12367.039	122.931	38.4	55	1235.451	12.281	31.4
24	12313.363	122.397	8.0				
25	12219.814	121.467	5.6				
26	12140.067	120.675	42.3				
27	12057.253	119.852	8.8				
28	11379.405	113.114	9.8				
29	11370.203	113.022	42.3				
30	7778.527	77.320	30.4				
31	7746.322	77.000	34.0				
32	7714.116	76.680	33.3				



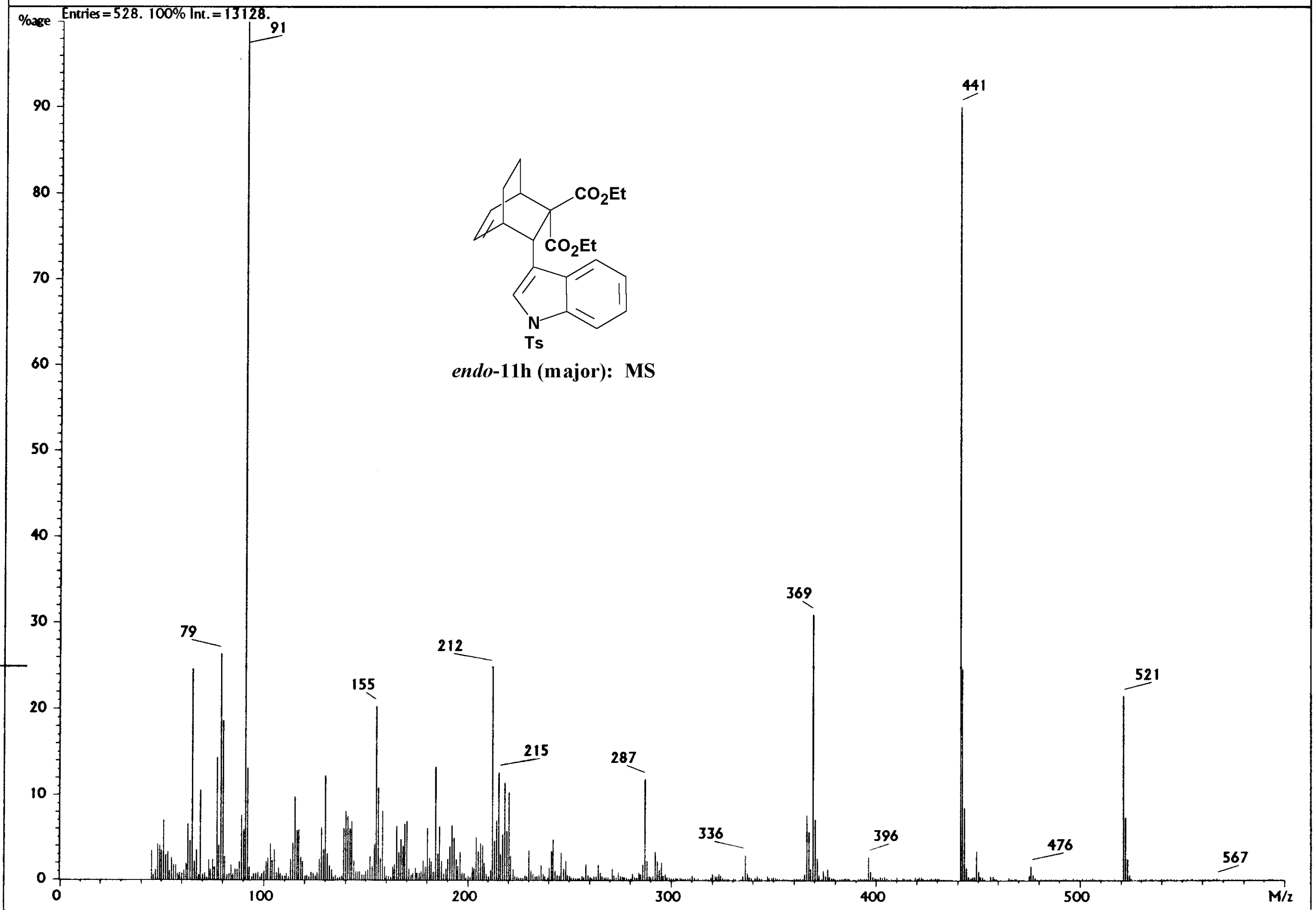
endo-11h (major): ^{13}C NMR





endo-11h (major): IR

File Name : c:\mswin\data\kin7a.mss
Creation Date/Time : 3/13/00 at 15:08:56
File Type : Lo-Res Mass Spectrum
File Title : Kinsman ACK-I-17-6 dprobe
Source File : c:\mswin\data\kin7.mss
Source Scan(s) : 70:80

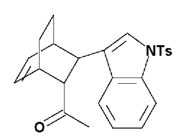


endo-**11h (major): MS**

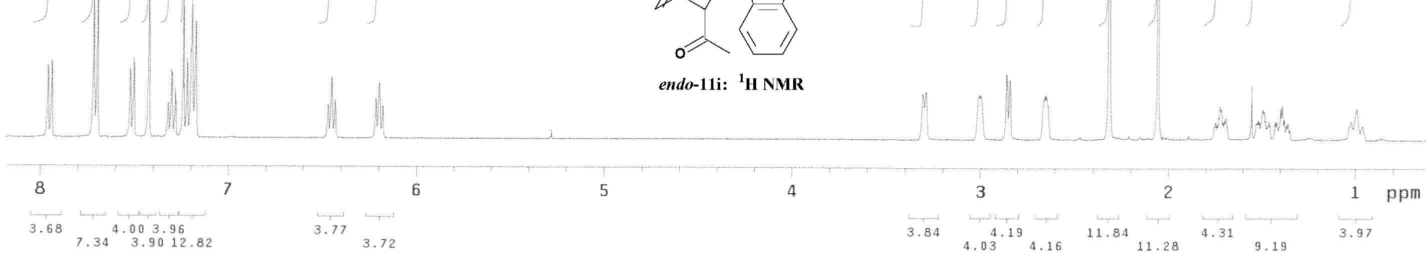
ack-ii-6-5 in cdcl3
STANDARD 1H OBSERVE

```
exp1  s2pul

      SAMPLE                 SPECIAL
date    Jun 26 2000   temp       not used
solvent       cdcl3   gain       not used
file /export/home/~   spin             20
vnmr1/walkup/auto_~   hst           0.008
 26.06.00/0801.fid    pw90         13.000
   ACQUISITION        alfa         20.000
sw           6402.0          FLAGS
at            1.995   il                n
np            25546   in                n
fb             3600   dp                y
bs               16   hs               nn
ss                2        PROCESSING
d1            1.000   fn         not used
nt                8         DISPLAY
ct                8   sp            247.2
   TRANSMITTER        wp           3026.5
tn               H1   rfl          3701.4
sfrq        400.088   rfp          2896.6
tof           417.6   rp           -149.0
tpwr             58   lp            -85.5
pw            6.500          PLOT
   DECOUPLER          wc              250
dn              C13   sc                0
dof               0   vs               80
dm              nnn   th                1
dmm               c   ai   cdc  ph
dpwr             46
dmf           17100
```

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3185.010	7.961	12.7	33	1199.097	2.997	7.7	65	551.976	1.380	3.6
2	3176.804	7.940	13.5	34	1143.998	2.859	11.5	66	547.286	1.368	2.7
3	3176.022	7.938	13.1	35	1137.355	2.843	10.6	67	544.160	1.360	3.0
4	3087.707	7.718	27.1	36	1064.280	2.660	7.4	68	539.862	1.349	1.7
5	3079.501	7.697	26.0	37	1061.545	2.653	7.9	69	410.125	1.025	3.4
6	3010.334	7.524	12.2	38	1058.419	2.645	7.8	70	397.620	0.994	5.5
7	3002.519	7.505	14.0	39	924.383	2.310	89.4	71	385.506	0.964	2.5
8	3001.737	7.503	13.1	40	820.828	2.052	126.7				
9	2970.475	7.425	27.2	41	702.033	1.755	2.4				
10	2929.835	7.323	6.1	42	699.297	1.748	3.1				
11	2922.410	7.304	12.1	43	695.780	1.739	2.7				
12	2921.238	7.302	11.3	44	692.654	1.731	4.0				
13	2914.204	7.284	8.7	45	689.919	1.724	6.2				
14	2913.031	7.281	6.1	46	686.402	1.716	5.5				
15	2896.619	7.240	35.1	47	683.276	1.708	3.2				
16	2895.447	7.237	9.3	48	680.540	1.701	3.4				
17	2889.194	7.221	13.7	49	677.805	1.694	3.9				
18	2881.379	7.202	15.5	50	673.897	1.684	2.4				
19	2879.034	7.196	24.4	51	621.143	1.553	9.7				
20	2870.828	7.176	20.5	52	612.155	1.530	2.9				
21	2587.908	6.468	6.0	53	609.810	1.524	3.2				
22	2580.092	6.449	10.9	54	607.465	1.518	3.5				
23	2573.058	6.431	6.8	55	605.121	1.512	3.1				
24	2487.088	6.216	7.0	56	597.305	1.493	5.4				
25	2479.663	6.198	9.8	57	594.961	1.487	5.0				
26	2472.629	6.180	5.8	58	592.225	1.480	3.5				
27	2111.945	5.279	1.7	59	589.881	1.474	2.3				
28	1322.582	3.306	8.0	60	585.191	1.463	3.3				
29	1323.363	3.308	8.0	61	571.514	1.428	3.2				
30	1316.720	3.291	8.4	62	568.388	1.421	3.4				
31	1203.396	3.008	7.7	63	559.791	1.399	5.5				
32	1202.223	3.005	8.0	64	556.274	1.390	6.1				

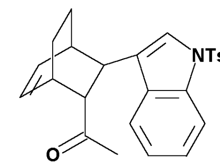


endo-11i: ^{1}H NMR

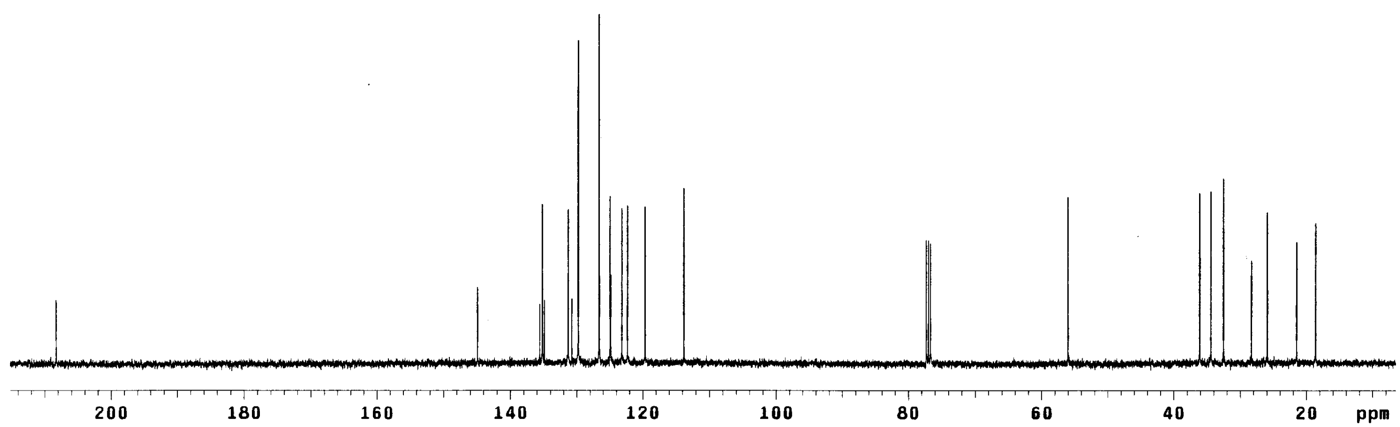


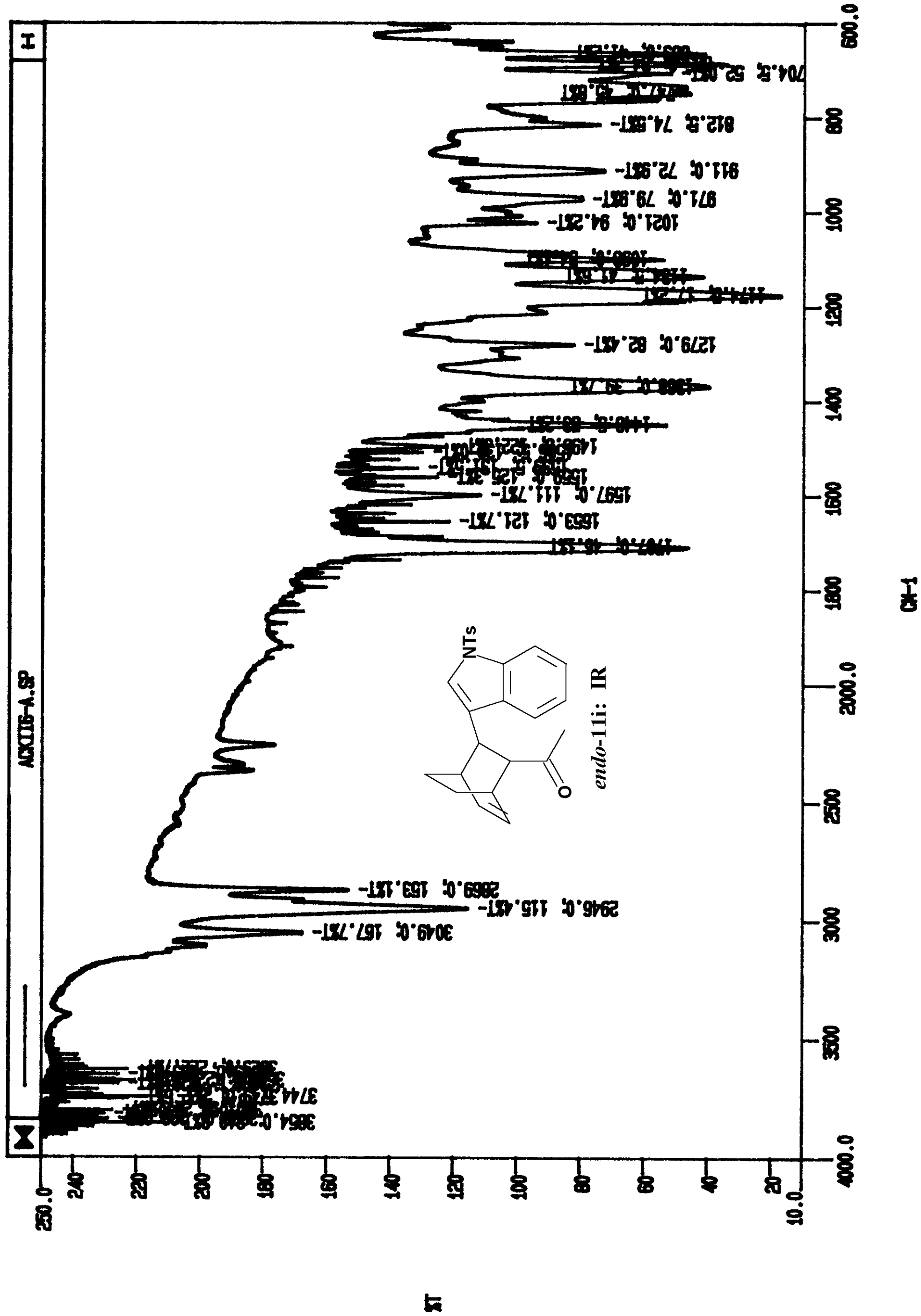
```
ack-ii-6-5 in cdcl3
13C OBSERVE

pad=2 run with gradshim before acquisiti
on

exp1  s2pul

      SAMPLE                  SPECIAL
date    Jun 26 2000   temp        not used
solvent       cdcl3   gain        not used
file /export/home/~   spin              20
vnmr1/walkup/auto_~   hst            0.008
 26.06.00/0501.fid    pw90          11.700
   ACQUISITION        alfa          20.000
sw          25125.6          FLAGS
at            1.199   il                 n
np            60270   in                 n
fb            13800   dp                 y
bs               64   hs                nn
ss                2        PROCESSING
d1            1.000   lb              1.00
nt              256   fn          not used
ct              128         DISPLAY
   TRANSMITTER        sp             651.2
tn              C13   wp           20998.0
sfrq        100.613   rfl           9273.6
tof          1550.9   rfp           7746.3
tpwr             59   rp             113.9
pw            5.850   lp            -319.1
    DECOUPLER                 PLOT
dn               H1   wc               250
dof               0   sc                 0
dm              yyy   vs                42
dmm               w   th                 7
dpwr             40   ai  no  ph
dmf            9700
```

INDEX	FREQUENCY	PPM	HEIGHT
1	20956.696	208.314	11.4
2	14576.180	144.890	13.8
3	13642.222	135.606	10.7
4	13601.581	135.202	28.7
5	13572.443	134.913	11.4
6	13209.748	131.308	27.8
7	13153.772	130.751	11.6
8	13056.389	129.783	58.3
9	12737.402	126.612	63.0
10	12570.240	124.951	30.1
11	12560.272	124.852	16.0
12	12390.043	123.160	27.9
13	12304.928	122.313	28.4
14	12037.316	119.653	28.3
15	11449.183	113.807	31.6
16	7778.527	77.320	22.2
17	7746.322	77.000	22.2
18	7714.883	76.687	21.6
19	5636.097	56.024	30.0
20	3632.457	36.107	30.7
21	3462.995	34.423	31.0
22	3272.830	32.533	33.3
23	2848.791	28.318	18.5
24	2607.250	25.917	27.2
25	2161.741	21.488	21.9
26	1873.426	18.622	25.3

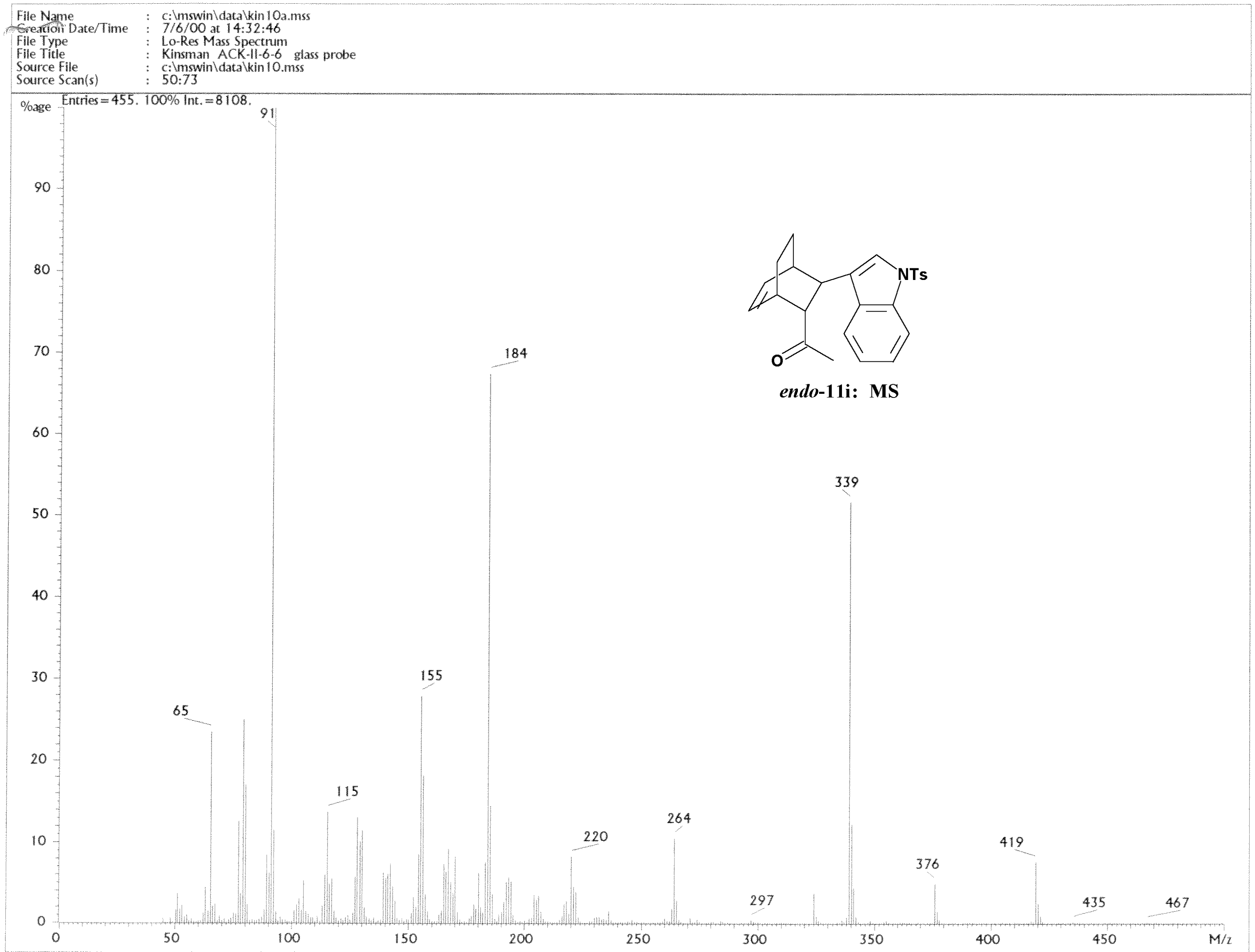


endo-11i: ^{13}C NMR



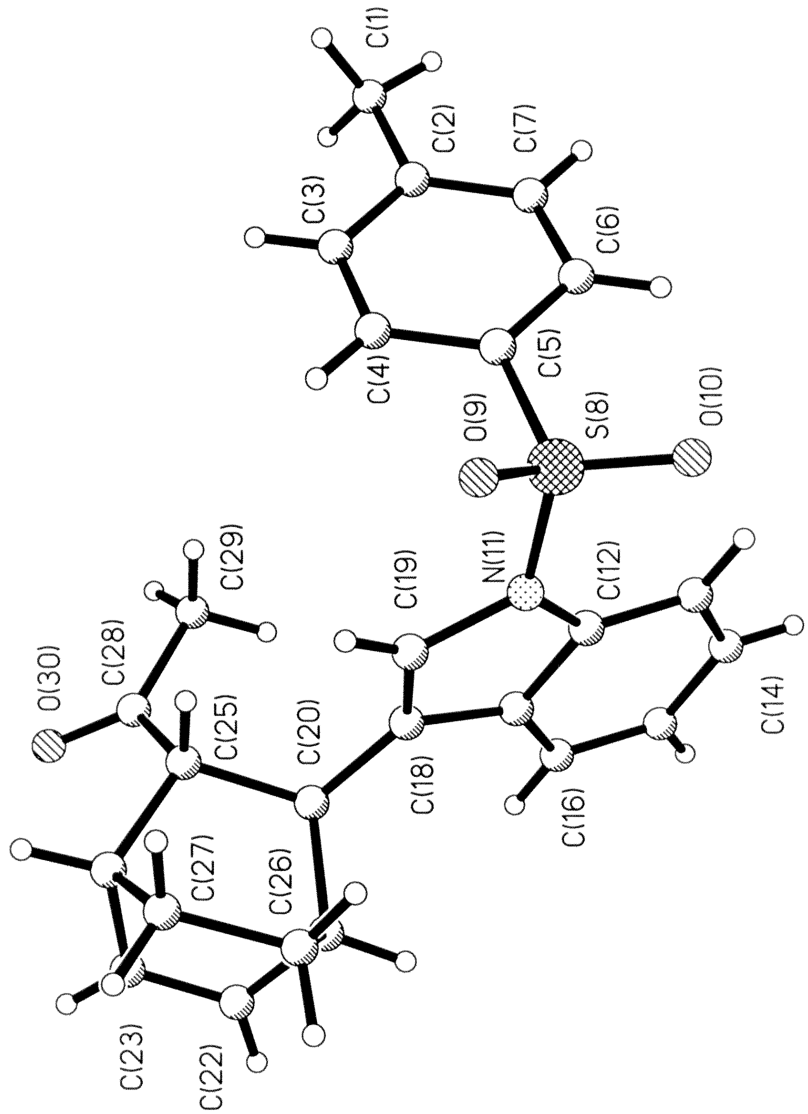


endo-11i: IR
NTs
O


File Name : c:\mswin\data\kin10a.mss
Creation Date/Time : 7/6/00 at 14:32:46
File Type : Lo-Res Mass Spectrum
File Title : Kinsman ACK-II-6-6 glass probe
Source File : c:\mswin\data\kin10.mss
Source Scan(s) : 50:73
Entries=455. 100% Int.=8108.
%age
91
184
155
65
115
220
264
297
339
376
419
435
467
NTs
O
M/z

endo-**11i: MS**

Appendix II: X-Ray Crystallographic Structures and Data for *endo*-11i.



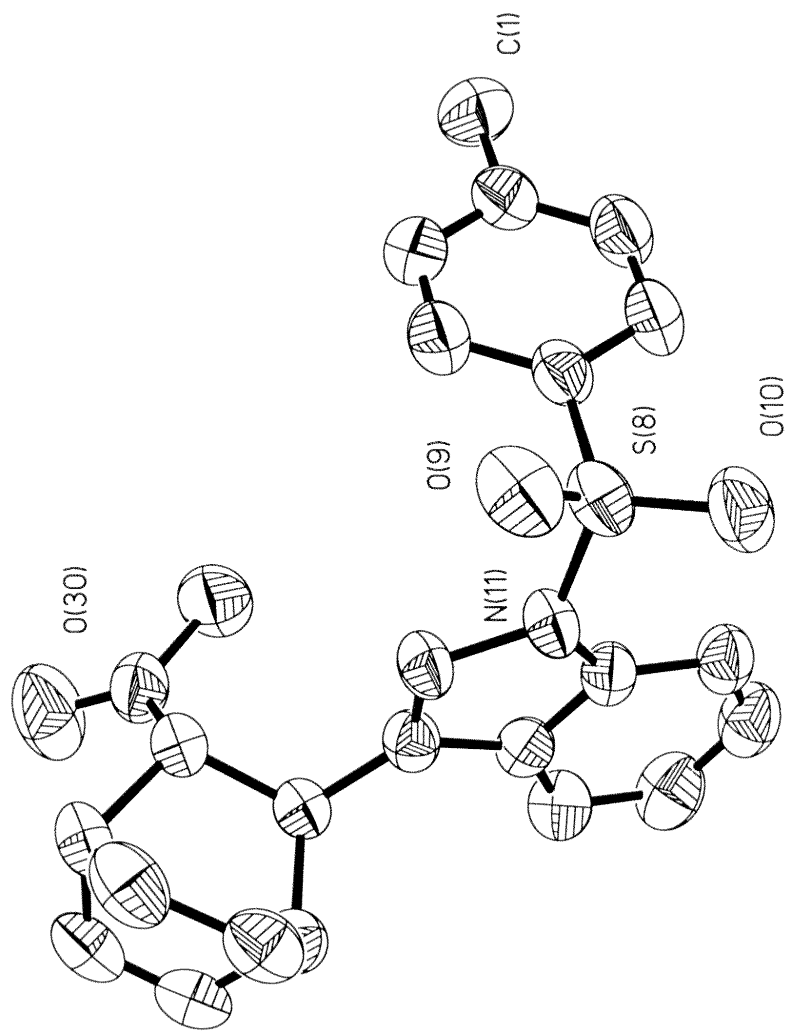

C(1)
O(9)
S(8)
O(10)
N(11)
O(30)

Table 1. Crystal data and structure refinement for 00094.

Identification code	*00094*	
Empirical formula	*C25 H25 N O3 S*	
Formula weight	*419.52*	
Temperature	*298(2) K*	
Wavelength	*0.71073 Å*	
Crystal system	*Monoclinic*	
Space group	*P2(1)*	
Unit cell dimensions	*a = 9.9766(4) Å*	α = *90°.*
	b = 20.0939(5) Å	β = *106.255(2)°.*
	c = 11.1572(5) Å	γ = *90°.*
Volume	*2147.26(14)* $Å^3$	
Z	*4*	
Density (calculated)	*1.298* Mg/m^3	
Absorption coefficient	*0.177* mm^{-1}	
F(000)	*888*	
Crystal size	*0.23 x 0.17 x 0.06* mm^3	
Theta range for data collection	*2.63 to 27.48°.*	
Index ranges	*-12<=h<=12, -22<=k<=26, -14<=l<=14*	
Reflections collected	*16634*	
Independent reflections	*9201 [R(int) = 0.0620]*	
Completeness to theta = 27.48°	*98.7 %*	
Absorption correction	*Integration*	
Max. and min. transmission	*0.9889 and 0.9603*	
Refinement method	*Full-matrix least-squares on* F^2	
Data / restraints / parameters	*9201 / 1 / 542*	
Goodness-of-fit on F^2	*1.014*	
Final R indices [I>2sigma(I)]	*R1 = 0.0540, wR2 = 0.1134*	
R indices (all data)	*R1 = 0.0972, wR2 = 0.1299*	
Absolute structure parameter	*0.00(7)*	
Largest diff. peak and hole	*0.223 and -0.257 e.*$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 00094. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	12288(4)	3625(2)	9379(4)	81(1)
C(2)	11089(4)	3226(2)	8607(3)	63(1)
C(3)	10777(4)	3211(2)	7306(4)	69(1)
C(4)	9688(4)	2850(2)	6574(3)	65(1)
C(5)	8870(3)	2480(2)	7153(3)	51(1)
C(6)	9148(4)	2485(2)	8441(3)	62(1)
C(7)	10231(4)	2861(2)	9137(3)	66(1)
S(8)	7518(1)	1986(1)	6239(1)	57(1)
O(9)	6838(3)	2336(1)	5120(2)	73(1)
O(10)	6725(2)	1722(1)	7002(2)	71(1)
N(11)	8307(3)	1350(1)	5786(2)	49(1)
C(12)	8936(3)	806(2)	6518(3)	47(1)
C(13)	8801(3)	575(2)	7650(3)	57(1)
C(14)	9511(4)	13(2)	8120(3)	68(1)
C(15)	10320(4)	-329(2)	7505(4)	71(1)
C(16)	10471(4)	-94(2)	6386(3)	60(1)
C(17)	9772(3)	485(2)	5882(3)	46(1)
C(18)	9711(3)	864(2)	4762(3)	46(1)
C(19)	8856(3)	1380(2)	4742(3)	49(1)
C(20)	10465(3)	671(1)	3814(3)	47(1)
C(21)	9624(4)	174(2)	2823(3)	56(1)
C(22)	10538(4)	-34(2)	2037(3)	60(1)
C(23)	10998(4)	452(2)	1483(3)	65(1)
C(24)	10473(4)	1126(2)	1701(3)	60(1)
C(25)	10895(3)	1272(2)	3115(3)	53(1)
C(26)	8348(4)	532(2)	1967(3)	65(1)
C(27)	8851(4)	1090(2)	1271(3)	70(1)
C(28)	12425(4)	1422(2)	3641(4)	67(1)
C(29)	12937(4)	1598(2)	4989(4)	87(1)
O(30)	13216(3)	1416(2)	2993(3)	108(1)

C(31)	17707(4)	-3550(2)	1665(4)	81(1)
C(32)	16488(3)	-3164(2)	878(3)	60(1)
C(33)	16160(4)	-3188(2)	-415(4)	65(1)
C(34)	15019(4)	-2853(2)	-1142(3)	60(1)
C(35)	14173(3)	-2480(2)	-592(3)	51(1)
C(36)	14492(4)	-2453(2)	696(3)	58(1)
C(37)	15636(4)	-2799(2)	1419(3)	65(1)
S(38)	12750(1)	-2047(1)	-1524(1)	54(1)
O(39)	12114(3)	-2428(1)	-2618(2)	71(1)
O(40)	11941(2)	-1796(1)	-759(2)	67(1)
N(41)	13430(3)	-1392(1)	-2044(2)	50(1)
C(42)	14006(3)	-824(2)	-1352(3)	48(1)
C(43)	13813(4)	-561(2)	-259(3)	61(1)
C(44)	14485(4)	24(2)	174(4)	70(1)
C(45)	15293(4)	344(2)	-454(4)	69(1)
C(46)	15499(3)	91(2)	-1537(3)	59(1)
C(47)	14864(3)	-515(1)	-1996(3)	48(1)
C(48)	14873(3)	-922(2)	-3049(3)	49(1)
C(49)	14011(3)	-1448(2)	-3050(3)	51(1)
C(50)	15701(3)	-761(2)	-3944(3)	52(1)
C(51)	14973(4)	-217(2)	-4917(4)	67(1)
C(52)	15974(4)	-37(2)	-5646(4)	75(1)
C(53)	16306(4)	-550(2)	-6252(4)	71(1)
C(54)	15650(4)	-1187(2)	-6092(3)	66(1)
C(55)	16028(4)	-1360(2)	-4689(3)	55(1)
C(56)	13687(4)	-529(2)	-5788(4)	80(1)
C(57)	14090(4)	-1084(2)	-6542(4)	80(1)
C(58)	17513(4)	-1600(2)	-4159(4)	70(1)
C(59)	17976(5)	-1794(2)	-2818(4)	83(1)
O(60)	18280(3)	-1665(2)	-4806(3)	121(1)

Table 3. Bond lengths [Å] and angles [°] for 00094.

C(1)-C(2)	1.497(5)
C(2)-C(7)	1.379(5)
C(2)-C(3)	1.398(5)
C(3)-C(4)	1.370(5)
C(4)-C(5)	1.389(5)
C(5)-C(6)	1.386(4)
C(5)-S(8)	1.753(3)
C(6)-C(7)	1.368(5)
S(8)-O(10)	1.418(2)
S(8)-O(9)	1.427(3)
S(8)-N(11)	1.653(3)
N(11)-C(12)	1.404(4)
N(11)-C(19)	1.420(4)
C(12)-C(13)	1.388(4)
C(12)-C(17)	1.396(4)
C(13)-C(14)	1.358(5)
C(14)-C(15)	1.380(5)
C(15)-C(16)	1.381(5)
C(16)-C(17)	1.391(5)
C(17)-C(18)	1.451(4)
C(18)-C(19)	1.338(4)
C(18)-C(20)	1.510(4)
C(20)-C(21)	1.551(4)
C(20)-C(25)	1.561(4)
C(21)-C(22)	1.492(5)
C(21)-C(26)	1.538(5)
C(22)-C(23)	1.306(5)
C(23)-C(24)	1.497(5)
C(24)-C(25)	1.544(4)
C(24)-C(27)	1.555(5)
C(25)-C(28)	1.504(5)
C(26)-C(27)	1.527(5)
C(28)-O(30)	1.210(4)
C(28)-C(29)	1.490(5)

C(31)-C(32)	*1.501(5)*
C(32)-C(37)	*1.382(5)*
C(32)-C(33)	*1.388(5)*
C(33)-C(34)	*1.375(5)*
C(34)-C(35)	*1.393(4)*
C(35)-C(36)	*1.383(4)*
C(35)-S(38)	*1.740(3)*
C(36)-C(37)	*1.386(5)*
S(38)-O(40)	*1.422(2)*
S(38)-O(39)	*1.431(2)*
S(38)-N(41)	*1.659(2)*
N(41)-C(49)	*1.404(4)*
N(41)-C(42)	*1.406(4)*
C(42)-C(43)	*1.392(4)*
C(42)-C(47)	*1.407(4)*
C(43)-C(44)	*1.371(5)*
C(44)-C(45)	*1.367(5)*
C(45)-C(46)	*1.378(5)*
C(46)-C(47)	*1.401(4)*
C(47)-C(48)	*1.434(4)*
C(48)-C(49)	*1.363(4)*
C(48)-C(50)	*1.499(4)*
C(50)-C(55)	*1.549(4)*
C(50)-C(51)	*1.568(5)*
C(51)-C(52)	*1.498(5)*
C(51)-C(56)	*1.512(5)*
C(52)-C(53)	*1.326(5)*
C(53)-C(54)	*1.470(5)*
C(54)-C(57)	*1.510(5)*
C(54)-C(55)	*1.545(5)*
C(55)-C(58)	*1.511(5)*
C(56)-C(57)	*1.518(6)*
C(58)-O(60)	*1.197(4)*
C(58)-C(59)	*1.489(5)*
C(7)-C(2)-C(3)	*116.7(3)*

C(7)-C(2)-C(1)	*122.0(3)*
C(3)-C(2)-C(1)	*121.3(3)*
C(4)-C(3)-C(2)	*122.7(3)*
C(3)-C(4)-C(5)	*118.5(3)*
C(6)-C(5)-C(4)	*120.3(3)*
C(6)-C(5)-S(8)	*120.4(3)*
C(4)-C(5)-S(8)	*119.3(3)*
C(7)-C(6)-C(5)	*119.3(3)*
C(6)-C(7)-C(2)	*122.5(3)*
O(10)-S(8)-O(9)	*120.09(16)*
O(10)-S(8)-N(11)	*106.71(14)*
O(9)-S(8)-N(11)	*105.26(14)*
O(10)-S(8)-C(5)	*109.28(16)*
O(9)-S(8)-C(5)	*109.31(17)*
N(11)-S(8)-C(5)	*105.09(14)*
C(12)-N(11)-C(19)	*107.3(2)*
C(12)-N(11)-S(8)	*126.7(2)*
C(19)-N(11)-S(8)	*123.0(2)*
C(13)-C(12)-C(17)	*122.1(3)*
C(13)-C(12)-N(11)	*130.5(3)*
C(17)-C(12)-N(11)	*107.4(3)*
C(14)-C(13)-C(12)	*117.1(3)*
C(13)-C(14)-C(15)	*122.5(3)*
C(14)-C(15)-C(16)	*120.4(4)*
C(15)-C(16)-C(17)	*118.8(3)*
C(16)-C(17)-C(12)	*119.0(3)*
C(16)-C(17)-C(18)	*133.1(3)*
C(12)-C(17)-C(18)	*107.9(3)*
C(19)-C(18)-C(17)	*107.1(3)*
C(19)-C(18)-C(20)	*128.9(3)*
C(17)-C(18)-C(20)	*124.0(3)*
C(18)-C(19)-N(11)	*110.2(3)*
C(18)-C(20)-C(21)	*112.9(3)*
C(18)-C(20)-C(25)	*114.3(2)*
C(21)-C(20)-C(25)	*108.2(3)*
C(22)-C(21)-C(26)	*107.6(3)*

C(22)-C(21)-C(20)	*107.6(3)*
C(26)-C(21)-C(20)	*109.3(3)*
C(23)-C(22)-C(21)	*115.0(3)*
C(22)-C(23)-C(24)	*114.4(3)*
C(23)-C(24)-C(25)	*109.4(3)*
C(23)-C(24)-C(27)	*106.9(3)*
C(25)-C(24)-C(27)	*106.7(3)*
C(28)-C(25)-C(24)	*113.2(3)*
C(28)-C(25)-C(20)	*110.5(3)*
C(24)-C(25)-C(20)	*109.2(3)*
C(27)-C(26)-C(21)	*109.0(3)*
C(26)-C(27)-C(24)	*109.8(3)*
O(30)-C(28)-C(29)	*120.5(3)*
O(30)-C(28)-C(25)	*121.8(3)*
C(29)-C(28)-C(25)	*117.7(3)*
C(37)-C(32)-C(33)	*118.5(3)*
C(37)-C(32)-C(31)	*121.0(4)*
C(33)-C(32)-C(31)	*120.4(3)*
C(34)-C(33)-C(32)	*120.7(3)*
C(33)-C(34)-C(35)	*120.5(3)*
C(36)-C(35)-C(34)	*119.2(3)*
C(36)-C(35)-S(38)	*120.9(2)*
C(34)-C(35)-S(38)	*120.0(3)*
C(35)-C(36)-C(37)	*119.8(3)*
C(32)-C(37)-C(36)	*121.3(3)*
O(40)-S(38)-O(39)	*120.80(15)*
O(40)-S(38)-N(41)	*106.53(14)*
O(39)-S(38)-N(41)	*104.70(14)*
O(40)-S(38)-C(35)	*108.94(15)*
O(39)-S(38)-C(35)	*109.55(16)*
N(41)-S(38)-C(35)	*105.13(14)*
C(49)-N(41)-C(42)	*107.9(2)*
C(49)-N(41)-S(38)	*121.1(2)*
C(42)-N(41)-S(38)	*126.6(2)*
C(43)-C(42)-N(41)	*130.9(3)*
C(43)-C(42)-C(47)	*122.1(3)*

N(41)-C(42)-C(47)	*107.0(3)*
C(44)-C(43)-C(42)	*117.4(4)*
C(45)-C(44)-C(43)	*121.5(4)*
C(44)-C(45)-C(46)	*122.0(4)*
C(45)-C(46)-C(47)	*118.5(3)*
C(46)-C(47)-C(42)	*118.4(3)*
C(46)-C(47)-C(48)	*133.6(3)*
C(42)-C(47)-C(48)	*108.0(3)*
C(49)-C(48)-C(47)	*107.2(3)*
C(49)-C(48)-C(50)	*129.2(3)*
C(47)-C(48)-C(50)	*123.6(3)*
C(48)-C(49)-N(41)	*109.8(3)*
C(48)-C(50)-C(55)	*115.3(3)*
C(48)-C(50)-C(51)	*112.2(3)*
C(55)-C(50)-C(51)	*107.3(3)*
C(52)-C(51)-C(56)	*109.0(3)*
C(52)-C(51)-C(50)	*107.2(3)*
C(56)-C(51)-C(50)	*107.5(3)*
C(53)-C(52)-C(51)	*112.8(3)*
C(52)-C(53)-C(54)	*115.7(3)*
C(53)-C(54)-C(57)	*107.0(3)*
C(53)-C(54)-C(55)	*109.1(3)*
C(57)-C(54)-C(55)	*107.7(3)*
C(58)-C(55)-C(54)	*113.8(3)*
C(58)-C(55)-C(50)	*111.7(3)*
C(54)-C(55)-C(50)	*109.9(3)*
C(51)-C(56)-C(57)	*110.6(3)*
C(54)-C(57)-C(56)	*109.0(3)*
O(60)-C(58)-C(59)	*120.4(4)*
O(60)-C(58)-C(55)	*121.4(4)*
C(59)-C(58)-C(55)	*118.1(3)*

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x 10^3$) for 00094. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	86(3)	69(2)	86(3)	-9(2)	23(2)	-17(2)
C(2)	67(2)	61(2)	65(2)	-5(2)	28(2)	6(2)
C(3)	80(3)	61(2)	75(3)	-3(2)	38(2)	-10(2)
C(4)	79(2)	69(2)	54(2)	3(2)	30(2)	1(2)
C(5)	54(2)	55(2)	51(2)	0(1)	24(2)	7(1)
C(6)	64(2)	82(2)	49(2)	-1(2)	28(2)	-1(2)
C(7)	74(2)	81(2)	50(2)	-5(2)	26(2)	-3(2)
S(8)	53(1)	69(1)	52(1)	-1(1)	22(1)	9(1)
O(9)	70(2)	87(2)	61(2)	6(1)	15(1)	26(1)
O(10)	58(1)	96(2)	69(2)	-10(1)	36(1)	-9(1)
N(11)	54(2)	59(2)	38(1)	4(1)	19(1)	5(1)
C(12)	45(2)	55(2)	41(2)	4(1)	12(1)	-7(1)
C(13)	56(2)	72(2)	41(2)	3(2)	14(2)	-15(2)
C(14)	71(2)	81(3)	48(2)	14(2)	11(2)	-16(2)
C(15)	71(2)	69(2)	65(2)	26(2)	8(2)	4(2)
C(16)	56(2)	65(2)	59(2)	8(2)	14(2)	5(2)
C(17)	45(2)	49(2)	41(2)	3(1)	7(1)	-4(1)
C(18)	44(2)	54(2)	39(2)	2(1)	9(1)	-3(1)
C(19)	57(2)	57(2)	36(2)	5(1)	17(1)	4(2)
C(20)	49(2)	50(2)	42(2)	0(1)	14(1)	1(1)
C(21)	55(2)	57(2)	56(2)	-3(2)	18(2)	-1(2)
C(22)	63(2)	61(2)	52(2)	-15(2)	9(2)	7(2)
C(23)	59(2)	89(3)	48(2)	-16(2)	16(2)	6(2)
C(24)	69(2)	73(2)	43(2)	5(2)	24(2)	3(2)
C(25)	62(2)	56(2)	46(2)	-1(1)	22(2)	-4(2)
C(26)	52(2)	81(2)	56(2)	-10(2)	9(2)	4(2)
C(27)	67(2)	91(3)	48(2)	4(2)	9(2)	23(2)
C(28)	69(2)	76(2)	60(2)	1(2)	26(2)	-18(2)
C(29)	77(3)	108(3)	71(3)	-22(2)	16(2)	-34(2)
O(30)	77(2)	173(3)	83(2)	-14(2)	39(2)	-36(2)

C(31)	66(2)	80(3)	93(3)	15(2)	17(2)	10(2)
C(32)	59(2)	55(2)	68(2)	8(2)	18(2)	-1(2)
C(33)	67(2)	61(2)	76(3)	4(2)	37(2)	8(2)
C(34)	77(2)	56(2)	54(2)	4(2)	33(2)	3(2)
C(35)	53(2)	52(2)	54(2)	3(1)	23(2)	-5(1)
C(36)	61(2)	64(2)	55(2)	1(2)	24(2)	6(2)
C(37)	72(2)	72(2)	53(2)	1(2)	21(2)	5(2)
S(38)	52(1)	60(1)	54(1)	6(1)	21(1)	-7(1)
O(39)	74(2)	70(2)	63(2)	-5(1)	12(1)	-26(1)
O(40)	58(1)	86(2)	66(2)	15(1)	33(1)	6(1)
N(41)	59(2)	50(1)	44(2)	0(1)	21(1)	-5(1)
C(42)	49(2)	54(2)	40(2)	0(1)	10(1)	3(1)
C(43)	64(2)	74(2)	45(2)	-3(2)	14(2)	6(2)
C(44)	65(2)	77(3)	61(2)	-19(2)	5(2)	13(2)
C(45)	56(2)	64(2)	80(3)	-20(2)	9(2)	7(2)
C(46)	44(2)	60(2)	73(2)	-3(2)	15(2)	-5(1)
C(47)	42(2)	52(2)	51(2)	5(1)	16(1)	6(1)
C(48)	51(2)	54(2)	45(2)	5(1)	19(1)	4(1)
C(49)	58(2)	57(2)	42(2)	-2(1)	22(2)	-1(2)
C(50)	51(2)	55(2)	50(2)	1(1)	16(2)	-4(1)
C(51)	81(3)	63(2)	61(2)	6(2)	29(2)	9(2)
C(52)	88(3)	81(3)	61(2)	6(2)	28(2)	-34(2)
C(53)	71(2)	82(3)	70(2)	7(2)	36(2)	-7(2)
C(54)	77(3)	71(2)	52(2)	-8(2)	22(2)	-7(2)
C(55)	62(2)	61(2)	45(2)	-3(1)	20(2)	-4(2)
C(56)	58(2)	126(4)	58(2)	22(2)	19(2)	9(2)
C(57)	74(3)	101(3)	55(2)	10(2)	3(2)	-18(2)
C(58)	69(2)	79(2)	69(3)	-4(2)	28(2)	15(2)
C(59)	85(3)	88(3)	71(3)	14(2)	15(2)	24(2)
O(60)	90(2)	199(4)	88(2)	12(2)	50(2)	51(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 00094.

	x	y	z	U(eq)
H(1A)	12341	3575	10247	121
H(1B)	12152	4086	9150	121
H(1C)	13141	3471	9236	121
H(3A)	11331	3456	6921	83
H(4A)	9501	2852	5709	78
H(6A)	8604	2236	8829	75
H(7A)	10395	2871	9999	80
H(13A)	8247	795	8070	68
H(14A)	9450	-147	8884	81
H(15A)	10767	-720	7846	85
H(16A)	11029	-319	5977	72
H(19A)	8651	1709	4133	59
H(20A)	11327	446	4270	56
H(21A)	9334	-212	3225	67
H(22A)	10764	-477	1948	73
H(23A)	11603	384	993	78
H(24A)	10822	1470	1243	72
H(25A)	10370	1663	3253	64
H(26A)	7786	218	1374	77
H(26B)	7776	714	2461	77
H(27A)	8464	1511	1439	84
H(27B)	8534	1008	379	84
H(29A)	13924	1682	5205	130
H(29B)	12756	1236	5483	130
H(29C)	12463	1990	5147	130
H(31A)	18165	-3777	1134	121
H(31B)	17383	-3868	2162	121
H(31C)	18352	-3250	2203	121
H(33A)	16718	-3434	-794	78

H(34A)	*14811*	*-2875*	*-2007*	*71*
H(36A)	*13940*	*-2204*	*1076*	*70*
H(37A)	*15834*	*-2786*	*2284*	*78*
H(43A)	*13249*	*-772*	*162*	*74*
H(44A)	*14390*	*206*	*911*	*84*
H(45A)	*15717*	*744*	*-140*	*83*
H(46A)	*16047*	*318*	*-1954*	*71*
H(49A)	*13834*	*-1793*	*-3630*	*61*
H(50A)	*16596*	*-577*	*-3455*	*62*
H(51A)	*14728*	*174*	*-4498*	*80*
H(52A)	*16324*	*390*	*-5672*	*90*
H(53A)	*16915*	*-511*	*-6745*	*85*
H(54A)	*15945*	*-1542*	*-6566*	*79*
H(55A)	*15414*	*-1726*	*-4596*	*66*
H(56A)	*13160*	*-193*	*-6348*	*96*
H(56B)	*13096*	*-707*	*-5305*	*96*
H(57A)	*13612*	*-1492*	*-6441*	*96*
H(57B)	*13818*	*-968*	*-7421*	*96*
H(59A)	*18934*	*-1935*	*-2605*	*124*
H(59B)	*17406*	*-2154*	*-2674*	*124*
H(59C)	*17888*	*-1420*	*-2311*	*124*