Diene-transmissive hetero-Diels–Alder reaction of 2-vinyl *α,β*-unsaturated aldimines: stereoselective synthesis of hexahydroquinazolin-2-ones

Satoru Kobayashi, Kenji Kudo, Ai Ito, Satoru Hirama, Takashi Otani and Takao Saito*

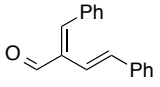
Supplementary information

General information

All melting points were determined on a Yanaco melting point apparatus and are uncorrected. Infrared spectra were recorded on a Hitachi 270-30 or a Horiba FT-710 model spectrophotometer. ^{1}H and ^{13}C NMR spectral data were obtained with a Bruker Avance-600, a JEOL JNM-EX 500, or a JEOL JNM-EX 300 instrument and chemical shifts are reported in ppm down field from tetramethylsilane (TMS) using an internal standard of TMS or $CDCl_3$. HRMS analysis were performed on a Bruker Daltonics microTOF or a Hitachi double-focusing M-80B spectrometer. Elemental analyses were performed with a YANACO CHN-CODER MT-6 model analyzer.

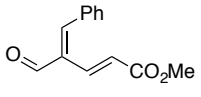
Experimental Procedure

2-Benzylidene-4-phenylbut-3-enal (1a)



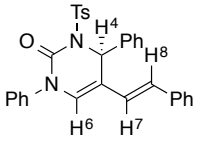
A mixture of α-bromocinnamaldehyde (10.0 g, 47.4 mmol), tri(*o*-tolyl)phosphine (1.44 g, 4.73 mmol), styrene (6.17 g, 59.2 mmol), palladium(II) acetate (531 mg, 2.37 mmol), and triethylamine (20.0 g, 198 mmol) was heated at 80 °C for 8 h. The mixture was condensed under reduced pressure, and the residue was purified by column chromatography on silica gel with to give aldehyde **1a** (7.32 g, 66%) as yellow solid; mp 66–68 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.98 (ddd, 1H, J = 1.0, 2.0, 10.5 Hz), 7.25–7.29 (m, 4H), 7.40–7.49 (m, 5H), 7.54–7.57 (m, 2H), 7.67 (d, 1H, J = 16.6 Hz), 9.76 (d, 1H, J = 2.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 119.20 (CH), 126.77 (3CH), 128.20 (CH), 128.66 (CH), 128.76 (2CH), 129.82 (CH), 130.29 (2CH), 135.03 (C), 135.90 (CH), 136.19 (C), 137.30 (C), 149.85 (CH), 194.09 (C); LRMS-FAB m/z (ion, % relative intensity): 235 ($[M+H]^+$, 100), 205 (62), 154 (40), 91 (54); HRMS-EI m/z $[M]^+$ calcd for $C_{17}H_{14}O$: 234.1045, found: 234.1054.

4-Formyl-5-phenylpenta-2,4-dienoic acid methyl ester (1b)



To a solution of α-bromocinnamaldehyde (1.0 g, 4.7 mmol) in toluene (50 mL) was added (2*E*)-3-(tributylstannyl)-2-propenoic acid methyl ester[S1] (2.11 g, 5.91 mmol) and $Pd(PPh_3)_4$ (277 mg, 0.24 mmol, 5 mol %), and the mixture was heated at 110 °C for 45 h. The mixture was condensed under reduced pressure, and the residue was purified by silica gel column chromatography to yield aldehyde **1b** (941 mg, 92%) as yellow oil; IR (NaCl): 1678 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.79 (s, 3H, Me), 7.02 (d, 1H, J = 16.1 Hz), 7.47–7.60 (m, 7H), 9.73 (d, 1H, J = 2.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 51.79 (CH_3), 125.12 (CH), 129.00 (2CH), 130.62 (2CH), 130.89 (CH), 133.86 (C), 133.93 (C), 134.37 (CH), 154.69 (CH), 167.56 (C), 192.00 (CH); HRMS-ESI calcd for $C_{13}H_{12}O_3Na$ $[M+Na]^+$: 239.0678, found: 239.0679.

1,4-Diphenyl-5-styryl-3-(toluene-4-sulfonyl)-3,4-dihydro-1*H*-pyrimidin-2-one (3a)

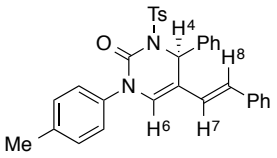


To a 1,2-dichloroethane (10 mL) solution of aldehyde **1** (100 mg, 0.43 mmol), aniline (48.4 mg, 0.52 mmol), and triethylamine (191 mg, 1.89 mmol) cooled by an ice bath, was added a 1.0 M dichloromethane solution of titanium tetrachloride (0.43 mL, 0.43 mmol). After the ice bath was removed, the mixture was stirred for 30 min, and then tosyl isocyanate (0.10 mL, 0.65 mmol) was added. The mixture was heated at 80 °C for 5 h, and the reaction was quenched by aqueous sodium hydrogen carbonate. The mixture was extracted with dichloromethane, dried over anhydrous magnesium sulfate, and then evaporated. The residue was purified by column chromatography on silica gel with AcOEt/hexane (1/3, v/v) as an eluent to yield **3a** (211 mg, 97%) as colorless crystals; mp 204–206 °C; IR (KBr): 1652, 1590, 1484, 1344, 1162 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.29 (s, 3H, Me), 6.52 (s, 1H, H-6), 6.53 (d, 1H, J = 13.4 Hz, H-4), 6.61 (d, 2H, J = 13.4 Hz, H-7+H-8), 7.01 (d, 2H, J = 8.1 Hz, Ar), 7.18–7.43 (m, 15H, Ar), 7.53–7.55 (m, 2H, Ar); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.51 (CH_3), 58.94 (CH), 118.69 (C), 122.97 (CH), 126.13 (2CH), 126.40 (2CH), 127.58 (2CH), 127.60 (CH),

[S1] H. Oda, T. Kobayashi, M. Kosugi and T. Migita, *Tetrahedron*, 1995, **51**, 695–702.

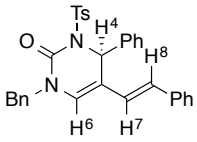
127.63 (CH), 127.88 (CH), 128.09 (CH), 128.66 (2CH), 128.81 (2CH), 128.85 (CH), 128.89 (2CH), 129.13 (2CH), 129.31 (CH), 129.32 (CH), 136.12 (C), 136.77 (C), 139.24 (C), 139.43 (C), 144.15 (C), 148.73 (C); LRMS-FAB *m/z* (ion, % relative intensity): 507 ($[M+H]^+$, 83), 351 ($[M–Ts]^+$, 14), 310 ($[M–TsNCO]^+$, 49), 231 (42), 185 (54); HRMS-FAB *m/z* $[M+H]^+$ calcd for $C_{31}H_{27}N_2O_3S$: 507.1742, found: 507.1749.

4-Phenyl-5-styryl-3-(toluene-4-sulfonyl)-1-*p*-tolyl-3,4-dihydro-1*H*-pyrimidin-2-one (3b)



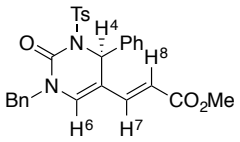
Colorless crystals; mp 135–137 °C; IR (KBr): 1650, 1344, 1162 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 2.29 (s, 3H, Me (Tol)), 2.34 (s, 3H, Me (Ts)), 6.49 (s, 1H, H-4), 6.54 (s, 1H, H-6), 6.58 (d, 1H, *J* = 16.2 Hz, H-7), 6.63 (d, 1H, *J* = 16.2 Hz, H-8), 7.00 (d, 2H, *J* = 8.2 Hz, Ar), 7.18–7.36 (m, 14H, Ar), 7.51–7.54 (m, 2H, Ar); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.00 (CH_3), 21.44 (CH_3), 58.90 (CH), 118.48 (C), 123.06 (CH), 126.12 (2CH), 126.17 (2CH), 127.49 (CH), 127.53 (CH), 127.56 (2CH), 128.30 (CH), 128.62 (2CH), 128.76 (3CH), 128.92 (2CH), 129.07 (2CH), 129.86 (2CH), 136.22 (C), 136.69 (C), 136.83 (C), 137.88 (C), 139.47 (C), 144.01 (C), 148.79 (C); LRMS-FAB *m/z* (ion, % relative intensity): 521 ($[M+H]^+$, 100), 366 ($[M–Ts]^+$, 12), 324 ($[M–TsNCO]^+$, 39), 289 (13), 246 (36), 185 (13), 154 (42); HRMS-FAB *m/z* $[M+H]^+$ calcd for $C_{32}H_{29}N_2O_3S$: 521.1898, found: 521.1898.

1-Benzyl-4-phenyl-5-styryl-3-(toluene-4-sulfonyl)-3,4-dihydro-1*H*-pyrimidin-2-one (3c)



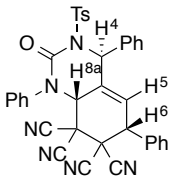
Colorless crystals; mp 181–183 °C; IR (KBr): 1638, 1594, 1340, 1160 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.35 (s, 3H, Me), 4.65 (d, 1H, *J* = 14.9 Hz, CH_2 (Bn)), 4.76 (d, 1H, *J* = 14.9 Hz, CH_2 (Bn)), 6.25 (s, 1H, H-4), 6.48 (d, 1H, *J* = 16.3 Hz, H-7), 6.50 (s, 1H, H-6), 6.57 (d, 1H, *J* = 16.3 Hz, H-8), 7.10–7.19 (m, 5H, Ar), 7.24–7.39 (m, 10H, Ar), 7.37–7.39 (m, 2H, Ar), 7.47 (d, 2H, *J* = 8.5 Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.57 (CH_3), 50.15 (CH_2), 58.33 (CH), 118.48 (C), 123.20 (CH), 126.08 (2CH), 126.71 (CH), 127.19 (CH), 127.48 (2CH), 127.49 (CH), 127.97 (CH), 128.03 (CH), 128.60 (2CH), 128.65 (CH), 128.72 (2CH), 128.82 (2CH), 128.93 (CH), 128.95 (2CH), 135.89 (2CH), 135.90 (C), 136.36 (C), 136.75 (C), 139.07 (C), 144.18 (C), 149.56 (C); LRMS-FAB *m/z* (ion, % relative intensity): 521 ($[M+H]^+$, 93), 366 ($[M–Ts]^+$, 12), 324 ($[M–TsNCO]^+$, 21), 246 (23), 185 (60), 154 (78); HRMS-FAB *m/z* $[M+H]^+$ calcd for $C_{32}H_{29}N_2O_3S$: 521.1898, found: 521.1902.

3-[1-Benzyl-2-oxo-4-phenyl-3-(toluene-4-sulfonyl)-1,2,3,4-tetrahydropyrimidin-5-yl]acrylic acid methyl ester (3d)



Colorless crystals; mp 178–180 °C; IR (KBr): 1678, 1612, 1344, 1160 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 2.34 (s, 3H, Me (Ts)), 3.67 (s, 3H, Me (CO_2Me)), 4.67 (d, 1H, *J* = 15.0 Hz, CH_2 (Bn)), 4.78 (d, 1H, *J* = 15.0 Hz, CH_2 (Bn)), 5.79 (d, 1H, *J* = 15.9 Hz, H-8), 6.30 (s, 1H, H-4), 6.50 (s, 1H, H-6), 7.07–7.18 (m, 5H, H-7+Ar), 7.25–7.31 (m, 8H, Ar), 7.39 (d, 2H, *J* = 8.2 Hz, Ar); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.54 (CH_3), 50.41 (CH_2), 51.53 (CH_3), 58.39 (CH), 115.36 (CH), 115.92 (C), 127.51 (2CH), 128.08 (2CH), 128.34 (CH), 128.81 (2CH), 128.92 (2CH), 128.95 (CH), 128.98 (2CH), 129.04 (2CH) , 133.01 (CH), 135.38 (C), 139.97 (C), 138.50 (C), 139.35 (CH), 144.38 (C), 149.13 (C), 167.05 (C); LRMS-EI 502 ($[M]^+$, 1.4), 471 ($[M–OMe]^+$, 3), 425 (3), 347 ($[M–Ts]^+$, 51), 246 (14), 91 (100); HRMS-EI calcd for $C_{28}H_{26}N_2O_5S$ $[M]^+$: 502.1562, found: 502.1568.

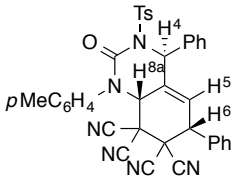
2-Oxo-1,4,6-triphenyl-3-(toluene-4-sulfonyl)-1,2,3,4,6,8a-hexahydroquinazoline-7,7,8,8-tetracarbonitrile (4a)



To a solution of **3a** (100 mg, 0.18 mmol) in dichloromethane (5 mL) was added tetracyanoethylene 38.4 mg (0.30 mmol). The mixture was stirred for 4 h at room temperature, and then condensed under reduced pressure. The residue was purified by column chromatography on silica gel with EtOAc/Hex (1/3, v/v) as an eluent to yield **4a** (101.6 mg, 90%) as colorless crystals; mp 166–168 °C; IR (KBr): 1682, 1594, 1488, 1366, 1168 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.46 (s, 3H, Me), 4.65 (dd, 1H, *J* = 2.4, 4.0 Hz, H-6), 4.86 (dd, 1H, *J* = 2.4, 2.4 Hz, H-8a), 6.57 (s, 1H, H-4), 6.68 (dd, 1H, *J* = 2.4, 4.0 Hz, H-5), 6.80 (d, 2H, *J* =

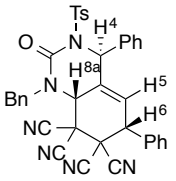
7.6 Hz, Ar), 7.16–7.56 (m, 15H, Ar), 7.98 (d, 2H, J = 8.6 Hz, Ar); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.77 (CH_3), 40.82 (C), 43.72 (C), 47.53 (CH), 61.23 (CH), 61.80 (CH), 107.17 (C), 108.93 (C), 109.05 (C), 111.22 (C), 124.42 (CH), 125.02 (3CH), 128.46 (CH), 129.04 (2CH), 129.13 (3CH), 129.18 (CH), 129.64 (2CH), 129.92 (3CH), 130.10 (CH), 130.19 (C), 130.96 (CH), 131.47 (2CH), 135.00 (C), 135.40 (C), 136.48 (2C), 145.57 (C), 149.63 (C); LRMS-FAB m/z (ion, % relative intensity): 635 ([M+H]$^+$, 69), 507 ([M–TCNE]$^+$, 16), 310 (27), 246 (18), 232 (17), 185 (30); Anal. Calcd. for $C_{37}H_{26}N_6O_3S$: C, 70.02; H, 4.13; N, 13.24; Found: C, 69.62; H, 3.97, N, 13.12.

2-Oxo-4,6-diphenyl-3-(toluene-4-sulfonyl)-1-*p*-tolyl-1,2,3,4,6,8a-hexahydroquinazoline-7,7,8,8-tetracarbonitrile (4b)



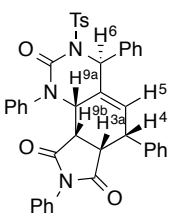
Colorless crystals; mp 152–154 °C; IR (KBr): 1686, 1594, 1490, 1386, 1166 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.29 (s, 3H, Me (*p*-Tol)), 2.46 (s, 3H, Me (Ts)), 4.64 (dd, 1H, J = 2.4, 3.9 Hz, H-6), 4.82 (dd, 1H, J = 2.4, 2.4 Hz, H-8a), 6.59 (s, 1H, H-4), 6.67 (dd, 1H, J = 2.4, 3.9 Hz, H-5), 6.69 (d, 2H, J = 6.6 Hz, Ar), 7.09 (d, 2H, J = 8.3 Hz, Ar), 7.14 (dd, 2H, J = 1.0, 7.1 Hz, Ar), 7.32 (d, 2H, J = 8.3 Hz, Ar), 7.39–7.57 (m, 8H, Ar), 7.97 (d, 2H, J = 8.3 Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.19 (CH_3), 21.72 (CH_3), 40.82 (C), 43.73 (C), 47.47 (CH), 61.14 (CH), 61.78 (2CH), 107.17 (C), 108.95 (C), 109.11 (C), 111.19 (C), 124.28 (CH), 124.99 (2CH), 128.08 (CH), 128.99 (2CH), 129.10 (2CH), 129.56 (2CH), 129.83 (2CH), 130.15 (C), 130.23 (C), 130.45 (2CH), 130.90 (2CH), 131.43 (2CH), 134.04 (C), 134.99 (C), 135.36 (C), 140.40 (C), 145.47 (C), 149.71 (C); LRMS-FAB m/z (ion, % relative intensity): 649 ([M+H]$^+$, 59), 520 (19), 324 (21), 289 (10), 246 (26), 185 (66); HRMS-FAB m/z [M+H]$^+$ calcd for $C_{38}H_{29}N_6O_3S$: 649.2022, found: 649.2025.

1-Benzyl-2-oxo-4,6-diphenyl-3-(toluene-4-sulfonyl)-1,2,3,4,6,8a-hexahydroquinazoline-7,7,8,8-tetracarbonitrile (4c)



Colorless crystals; mp 123–125 °C; IR (KBr): 1688, 1364, 1170 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.49 (s, 3H, Me), 4.34 (d, 1H, J = 15.9 Hz, CH_2 (Bn)), 4.36 (dd, 1H, J = 2.8, 2.8 Hz, H-6), 4.58 (dd, 1H, J = 2.8, 2.8 Hz, H-8a), 5.32 (d, 1H, J = 15.9 Hz, CH_2 (Bn)), 6.42 (s, 1H, H-4), 6.43 (d, 2H, J = 7.3 Hz, Ar), 6.57 (dd, 1H, J = 2.8, 2.8 Hz, H-5), 6.96 (dd, 2H, J = 7.3, 7.3 Hz, Ar), 7.11–7.14 (m, 3H, Ar), 7.26–7.54 (m, 10H, Ar), 8.09 (d, 2H, J = 8.2 Hz, Ar); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.79 (CH_3), 41.95 (C), 45.44 (C), 47.30 (CH), 48.89 (CH_2), 56.26 (CH), 61.58 (CH), 107.54 (C), 108.93 (C), 110.77 (C), 111.54 (C), 125.10 (2CH), 126.55 (CH), 128.28 (CH), 128.73 (2CH), 128.76 (CH), 128.89 (2CH), 129.37 (2CH), 129.41 (2CH), 129.66 (2CH), 129.92 (2CH), 130.45 (2CH), 130.81 (CH), 131.80 (C), 131.92 (C), 132.58 (C), 134.75 (C), 135.41 (C), 145.76 (C), 151.34 (C); LRMS-FAB m/z (ion, % relative intensity): 649 ([M+H]$^+$, 9), 635 (54), 507 ([M–TCNE]$^+$, 16), 310 (31), 232 (24), 185 (35), 154 (100); Anal. Calcd. for $C_{38}H_{28}N_6O_3S$: C, 70.35; H, 4.35; N, 12.95. Found: C, 70.38; H, 4.16; N, 13.08.

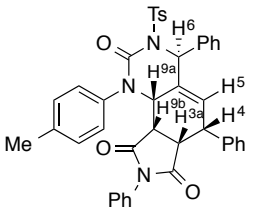
2,4,6,9-Tetraphenyl-7-(toluene-4-sulfonyl)-3a,6,7,9,9a,9b-hexahydro-4*H*-2,7,9-triazacyclopenta[*a*]naphthalene-1,3,8-trione (5a)



A solution of **4a** (100 mg, 0.20 mmol), *N*-phenylmaleimide (52 mg, 0.30 mmol) in toluene (5 mL) was heated at 110 °C for 9 h. The mixture was condensed under reduced pressure, and the residue was purified by silica gel chromatography with AcOEt/Hex (1/2, v/v) as an eluent to yield **5a** (119.6 mg, 88%) as a colorless solid; mp 293–294 °C; IR (KBr): 1688, 1372, 1260, 1164, 1086, 1014 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.32 (s, 3H, Me (Ts)), 3.36 (m, 2H, H-9b+H-4), 3.62 (dd, 1H, J = 4.2, 4.2 Hz, H-3a), 4.42 (dd, 1H, J = 3.2, 2.7 Hz, H-9a), 6.45 (s, 1H, H-6), 6.63 (dd, 1H, J = 3.2, 3.2 Hz, H-5), 7.03 (d, 2H, J = 8.3 Hz, Ar), 7.12 (d, 2H, J = 7.6 Hz, Ar), 7.21–7.46 (m, 18H, Ar), 7.75 (d, 2H, J = 8.2 Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.54 (CH_3), 40.88 (CH), 41.56 (CH), 44.59 (CH), 58.67 (CH), 61.34 (CH), 124.42 (CH), 125.75 (2CH), 126.42 (2CH), 127.54 (CH), 127.94 (CH), 128.17 (CH), 128.43 (CH), 128.51 (2CH), 128.62 (2CH), 128.68 (2CH), 128.85 (2CH), 128.86 (2CH), 128.89 (2CH), 129.25 (2CH), 129.31 (2CH), 131.36 (C), 136.01 (C), 137.08 (C), 137.62 (C), 137.92 (C), 139.34 (C), 141.14 (C), 151.46 (C), 173.11 (C), 173.72 (C); LRMS-FAB m/z

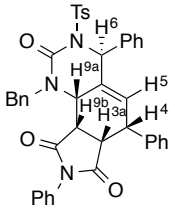
(ion, % relative intensity): 680 ([M+H]$^+$, 100), 352 (19), 310 (43), 243 (46), 91 (47); HRMS-FAB *m*/*z* [M+H]$^+$ calcd for $C_{41}H_{34}N_3O_5S$: 680.2219, found: 680.2217.

2,4,6-Triphenyl-7-(toluene-4-sulfonyl)-9-*p*-tolyl-3a,6,7,9,9a,9b-hexahydro-4*H*-2,7,9-triazacyclopenta[*a*]naphthalene-1,3,8-trione (5b)



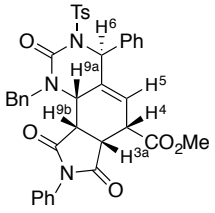
Colorless crystals; mp 281–282 °C; IR (KBr): 1696, 1416, 1374, 1162, 1084, 1014 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.30 (s, 3H, Me (*p*-Tol)), 2.33 (s, 3H, Me (Ts)), 3.38 (dd, 1H, *J* = 6.6, 8.5 Hz, H-9b), 3.43 (dd, 1H, *J* = 6.6, 8.5 Hz, H-3a), 3.62 (br ddd, 1H, *J* = 3.2, 3.2, 6.3 Hz, H-4), 4.40 (m, 1H, H-9a), 6.44 (s, 1H, H-6), 6.64 (dd, 1H, *J* = 3.2, 3.2 Hz, H-5), 7.04 (d, 2H, *J* = 8.5 Hz, Ar), 7.12 (dd, 4H, *J* = 4.9, 7.3 Hz, Ar), 7.19–7.47 (m, 15H, Ar), 7.76 (d, 2H, *J* = 8.3 Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.07 (CH_3), 21.57 (CH_3), 40.89 (CH), 41.56 (CH), 44.68 (CH), 58.75 (CH), 61.39 (CH), 124.34 (CH), 125.80 (2CH), 126.44 (3CH), 127.60 (CH), 127.93 (CH), 128.35 (2CH), 128.45 (CH), 128.55 (2CH), 128.70 (2CH), 128.85 (3CH), 128.91 (2CH), 129.34 (2CH), 129.96 (2CH), 131.36 (C), 136.01 (C), 136.70 (C), 137.22 (C), 137.63 (C), 137.96 (C), 138.23 (C), 144.11 (C), 151.61 (C), 173.14 (C), 173.77 (C); LRMS-FAB *m*/*z* (ion, % relative intensity): 694 ([M+H]$^+$, 100), 456 (10), 366 (13), 324 (28), 289 (14), 246 (35), 243 (29); HRMS-FAB *m*/*z* [M+H]$^+$ calcd for $C_{42}H_{36}N_3O_5S$: 694.2376, found: 694.2376; Anal. Calcd. for $C_{43}H_{39}N_3O_5S$: C, 72.76; H, 5.54; N, 5.92. Found C, 72.35; H, 4.96; N, 6.01.

9-Benzyl-2,4,6-triphenyl-7-(toluene-4-sulfonyl)-3a,6,7,9,9a,9b-hexahydro-4*H*-2,7,9-triazacyclopenta[*a*]naphthalene-1,3,8-trione (5c)



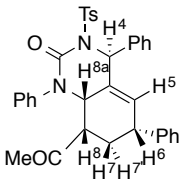
Colorless crystals; mp 259–261 °C; IR (KBr): 1672, 1596, 1486, 1428, 1364, 1332, 1162, 1086 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.33 (s, 3H, Me (Ts)), 3.43 (dd, 1H, *J* = 7.3, 9.0 Hz, H-9b), 3.59 (m, 2H, H-3a+H-4), 3.86 (ddd, 1H, *J* = 2.4, 2.4, 6.4 Hz, H-9a), 4.04 (d, 1H, *J* = 15.9 Hz, CH_2 (Bn)), 5.46 (d, 1H, *J* = 15.9 Hz, CH_2 (Bn)), 6.34 (s, 1H, H-6), 6.58–6.60 (m, 3H, Ar+H-5), 6.98–7.06 (m, 4H, Ar), 7.12–7.18 (m, 2H, Ar), 7.24–7.45 (m, 14H, Ar), 7.84 (d, 2H, *J* = 8.3 Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.65 (CH_3), 39.64 (CH), 40.80 (CH), 47.57 (CH), 51.83 (CH), 53.41 (CH_2), 60.67 (CH), 125.33 (CH), 125.81 (2CH), 126.47 (2CH), 127.59 (CH), 127.70 (CH), 127.74 (2CH), 127.83 (CH), 128.13 (CH), 128.42 (CH), 128.54 (2CH), 128.70 (2CH), 128.75 (2CH), 128.90 (2CH), 128.97 (2CH), 129.01 (2CH), 129.28 (2CH), 131.41 (C), 135.16 (C), 135.73 (C), 136.47 (C), 137.30 (C), 137.54 (C), 137.76 (C), 172.50 (C), 173.82 (C); LRMS-FAB *m*/*z* (ion, % relative intensity): 694 ([M+H]$^+$, 50), 307 (9), 289 (10), 246 (22), 185 (40), 154 (100); HRMS-FAB *m*/*z* [M+H]$^+$ calcd for $C_{42}H_{36}N_3O_5S$: 694.2376, found: 694.2381.

9-Benzyl-1,3,8-trioxo-2,6-diphenyl-7-(toluene-4-sulfonyl)-2,3,3a,4,6,7,8,9,9a,9b-decahydro-1*H*-2,7,9-triazacyclopenta[a]naphthalene-4-carboxylic acid methyl ester (5d)



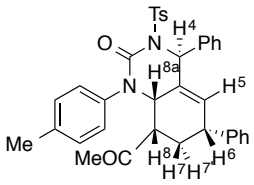
Colorless crystals; mp 273–276 °C; IR (KBr): 1702, 1428, 1340, 1168, 1088 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.34 (s, 3H, Me (Ts)), 3.05 (ddd, 1H, *J* = 2.7, 3.0, 6.1 Hz, H-4), 3.59 (dd, 1H, *J* = 7.5, 9.0 Hz, H-9b), 3.66 (ddd, 1H, *J* = 2.7, 2.9, 7.5 Hz, H-9a), 3.82 (dd, 1H, *J* = 6.1, 9.0 Hz, H-3a), 3.86 (s, 3H, Me (CO_2Me)), 4.05 (d, 1H, *J* = 15.8 Hz, CH_2 (Bn)), 5.43 (d, 1H, *J* = 15.8 Hz, CH_2 (Bn)), 6.27 (s, 1H, H-6), 6.54 (d, 2H, *J* = 7.6 Hz, Ar), 6.71 (dd, 1H, *J* = 2.9, 3.0 Hz, H-5), 7.01 (m, 3H, Ar), 7.04 (d, 3H, *J* = 8.0 Hz, Ar), 7.13 (m, 1H, Ar), 7.23 (dd, 2H, *J* = 7.6, 7.6 Hz, Ar), 7.30 (m, 1H, Ar), 7.39 (d, 3H, *J* = 7.3 Hz, Ar), 7.45 (dd, 2H, *J* = 7.3, 7.3 Hz, Ar), 7.82 (d, 2H, *J* = 8.0 Hz, Ar); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.78 (CH_3), 39.14 (CH), 39.67 (CH), 41.16 (CH), 47.78 (CH_2), 51.91 (CH_3), 52.89 (CH), 60.69 (CH), 121.28 (CH), 125.90 (2CH), 126.65 (2CH), 127.93 (2CH), 127.95 (CH), 128.01 (CH), 128.84 (CH), 128.91 (2CH), 129.08 (2CH), 129.13 (2CH), 129.19 (2CH), 129.37 (2CH), 131.32 (C), 134.98 (C), 135.88 (C), 136.18 (C), 137.31 (C), 144.59 (C), 152.79 (C), 169.95 (C), 172.41 (C), 175.04 (C); LRMS-EI 675 ([M]$^+$, 5), 611 (34), 520 ([M–Ts]$^+$, 61), 347 (31), 91 (100); HRMS-EI calcd for $C_{38}H_{33}N_3O_7S$ [M]$^+$: 675.2039, found: 675.2032.

8-Acetyl-1,4,6-triphenyl-3-(toluene-4-sulfonyl)-3,4,6,7,8,8a-hexahydro-1*H*-quinazolin-2-one (6a)



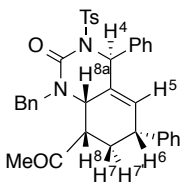
To a solution of **3a** (100 mg, 0.20 mmol) and methyl vinyl ketone (21 mg, 0.30 mmol) in dichloromethane (5 mL) was added 1.0 M dichloromethane solution of trimethylsilyl trifluoromethanesulfonate (40 μL, 40 μmol) at –20 °C. The resulting mixture was warmed to 0 °C and stirred for 36 h. The mixture was quenched by sodium hydrogen carbonate, extracted with dichloromethane, and dried over magnesium sulfate. The solvent was evaporated, and the residue was purified by column chromatography on silica gel with AcOEt/Hex (1/2, v/v) to yield **6a** (61.1 mg, 53%) as colorless crystals; mp 254–256 °C; IR (KBr): 1668, 1592, 1414, 1346, 1242, 1150 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 1.35 (s, 3H, Me (COMe)), 2.31 (m, 2H, H-7+H-7'), 2.39 (dd, 1H, *J* = 4.3, 8.5 Hz, H-8), 2.42 (s, 3H, Me (Ts)), 3.76 (ddd, 1H, *J* = 3.2, 3.2, 9.5 Hz, H-6), 4.11 (ddd, 1H, *J* = 3.2, 3.4, 8.5 Hz, H-8a), 6.27 (dd, 1H, *J* = 3.2, 3.4 Hz, H-5), 6.50 (s, 1H, H-4), 6.76 (d, 2H, *J* = 7.9 Hz, Ar), 7.18 (d, 2H, *J* = 7.2 Hz, Ar), 7.22 (d, 3H, *J* = 7.3, 7.8 Hz, Ar), 7.25 (d, 3H, *J* = 4.2, 8.1 Hz, Ar), 7.32–7.38 (m, 7H, Ar), 7.95 (d, 2H, *J* = 8.2 Hz, Ar); ^{13}C NMR (151 MHz, $CDCl_3$) δ 21.65 (CH_3), 27.47 (CH_3), 31.83 (CH_2), 38.20 (CH), 45.47 (CH), 58.19 (CH), 63.03 (CH), 124.51 (CH), 125.83 (2CH), 126.68 (CH), 127.51 (CH), 127.76 (CH), 127.94 (2CH), 128.47 (2CH), 128.84 (2CH), 128.86 (2CH), 129.03 (4CH), 129.14 (2CH), 133.60 (C), 137.15 (C), 138.57 (C), 139.10 (C), 142.42 (C), 143.80 (C), 151.30 (C), 205.32 (C); LRMS-FAB *m*/*z* (ion, % relative intensity): 577 ($[M+H]^+$, 23), 307 (17), 289 (14), 246 (52), 219 (17), 185 (86), 154 (100); HRMS-EI *m*/*z* $[M]^+$ calcd for $C_{35}H_{32}N_2O_4S$: 576.2083, found: 576.2081.

8-Acetyl-4,6-diphenyl-3-(toluene-4-sulfonyl)-1-*p*-tolyl-3,4,6,7,8,8a-hexahydro-1*H*-quinazolin-2-one (6b)



Colorless crystals; mp 228–230 °C; IR (KBr): 1672, 1412, 1346, 1244, 1156 cm^{-1}; ^1H NMR (600 MHz, C_6D_6) δ 0.96 (s, 3H, Me (COMe)), 1.44 (ddd, 1H, *J* = 5.2, 7.6, 15.0 Hz, H-7), 1.65 (dd, 1H, *J* = 3.1, 15.0 Hz, H-7'), 1.86 (s, 3H, Me (*p*-Tol)), 1.94 (s, 3H, Me (Ts)), 1.96 (ddd, 1H, *J* = 3.1, 5.2, 7.4 Hz, H-8), 3.13 (ddd, 1H, *J* = 3.4, 3.8, 7.6 Hz, H-6), 3.93 (ddd, 1H, *J* = 2.2, 3.4, 7.4 Hz, H-8a), 5.79 (dd, 1H, *J* = 2.2, 3.8 Hz, H-5), 6.72 (d, 2H, *J* = 8.4 Hz, Ar), 6.76 (d, 2H, *J* = 8.4 Hz, Ar), 6.79 (s, 1H, H-4), 6.88 (d, 2H, *J* = 8.1 Hz, Ar), 7.00 (d, 2H, *J* = 7.4 Hz, Ar), 7.04 (dd, 1H, *J* = 7.3, 7.3 Hz, Ar), 7.10–7.15 (m, 5H, Ar), 7.59 (d, 2H, *J* = 7.4 Hz, Ar), 8.37 (d, 2H, *J* = 8.1 Hz, Ar); ^{13}C NMR (151 MHz, $CDCl_3$) δ 21.03 (CH_3), 21.66 (CH_3), 27.53 (CH_3), 31.83 (CH_2), 38.21 (CH), 45.49 (CH), 58.13 (CH_3), 63.08 (CH), 124.45 (CH), 125.85 (2CH), 126.65 (CH), 127.62 (2CH), 127.75 (CH), 128.46 (2CH), 128.82 (2CH), 128.88 (2CH), 128.90 (2CH), 129.07 (2CH), 129.77 (2CH), 133.65 (C), 136.39 (C), 137.17 (C), 137.42 (C), 138.60 (C), 142.52 (C), 143.75 (C), 151.40 (C), 205.40 (C); LRMS-FAB *m*/*z* (ion, % relative intensity): 591 ($[M+H]^+$, 45), 307 (14), 289 (11), 246 (26), 219 (11), 185 (20), 154 (100); HRMS-EI *m*/*z* $[M]^+$ calcd for $C_{36}H_{34}N_2O_4S$: 590.2239, found: 590.2240.

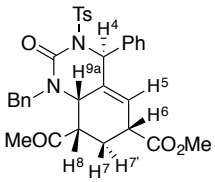
8-Acetyl-1-benzyl-4,6-diphenyl-3-(toluene-4-sulfonyl)-3,4,6,7,8,8a-hexahydro-1*H*-quinazolin-2-one (6c)



Colorless crystals; mp 204–206 °C; IR (KBr): 1662, 1342, 1162 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.27 (s, 3H, Me (COMe)), 2.28–2.29 (m, 1H, H-7), 2.29–2.35 (m, 1H, H-7'), 2.47 (s, 3H, Me (Ts)), 2.72 (dd, 1H, *J* = 3.8, 6.9 Hz, H-8), 3.50 (d, 1H, *J* = 15.5 Hz, CH_2 (Bn)), 3.50–3.53 (m, 1H, H-6), 3.69 (ddd, 1H, *J* = 3.2, 3.4, 6.9 Hz, H-8a), 4.94 (d, 1H, *J* = 15.6 Hz, CH_2 (Bn)), 6.16 (dd, 1H, *J* = 3.1, 3.2 Hz, H-5), 6.36 (s, 1H, H-4), 6.49 (d, 2H, *J* = 7.5 Hz, Ar), 6.97 (dd, 2H, *J* = 7.5, 7.7 Hz, Ar), 7.02–7.13 (m, 3H, Ar), 7.22 (dd, 2H, *J* = 7.2, 7.8 Hz, Ar), 7.30 (dd, 2H, *J* = 7.4, 7.5 Hz, Ar), 7.34–7.41 (m, 6H, Ar), 8.11 (d, 2H, *J* = 8.3 Hz, Ar); ^{13}C NMR (76 MHz, $CDCl_3$) δ 21.87 (CH_3), 27.38 (CH_3), 31.94 (CH_2), 38.00 (CH), 44.01 (CH), 47.48 (CH_2), 52.86 (CH), 62.71 (CH), 125.32 (CH), 125.89 (2CH), 126.78 (CH), 127.40 (CH), 127.71 (3CH), 128.44 (2CH), 128.55 (2CH), 128.84 (2CH), 129.09 (2CH), 129.18 (2CH), 129.25 (2CH), 133.67 (C), 135.50 (C), 136.88 (C), 138.41 (C), 142.54 (C), 144.21 (C), 153.01 (C), 205.16 (C); LRMS-EI *m*/*z* (ion, % relative intensity): 590 (M^+, 15), 520 (M^+–MVK, 34), 435 (M^+–Ts, 20), 393 (M^+–TsNCO, 4), 331 (27), 91 (100); HRMS-EI *m*/*z* $[M]^+$

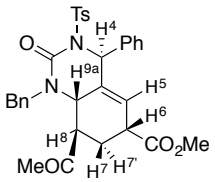
calcd for $C_{36}H_{34}N_2O_4S$: 590.2239, found: 590.2249.

8-Acetyl-1-benzyl-2-oxo-4-phenyl-3-(toluene-4-sulfonyl)-1,2,3,4,6,7,8,8a-octahydroquinazoline-6-carboxylicacid methyl ester (*endo*-6d)



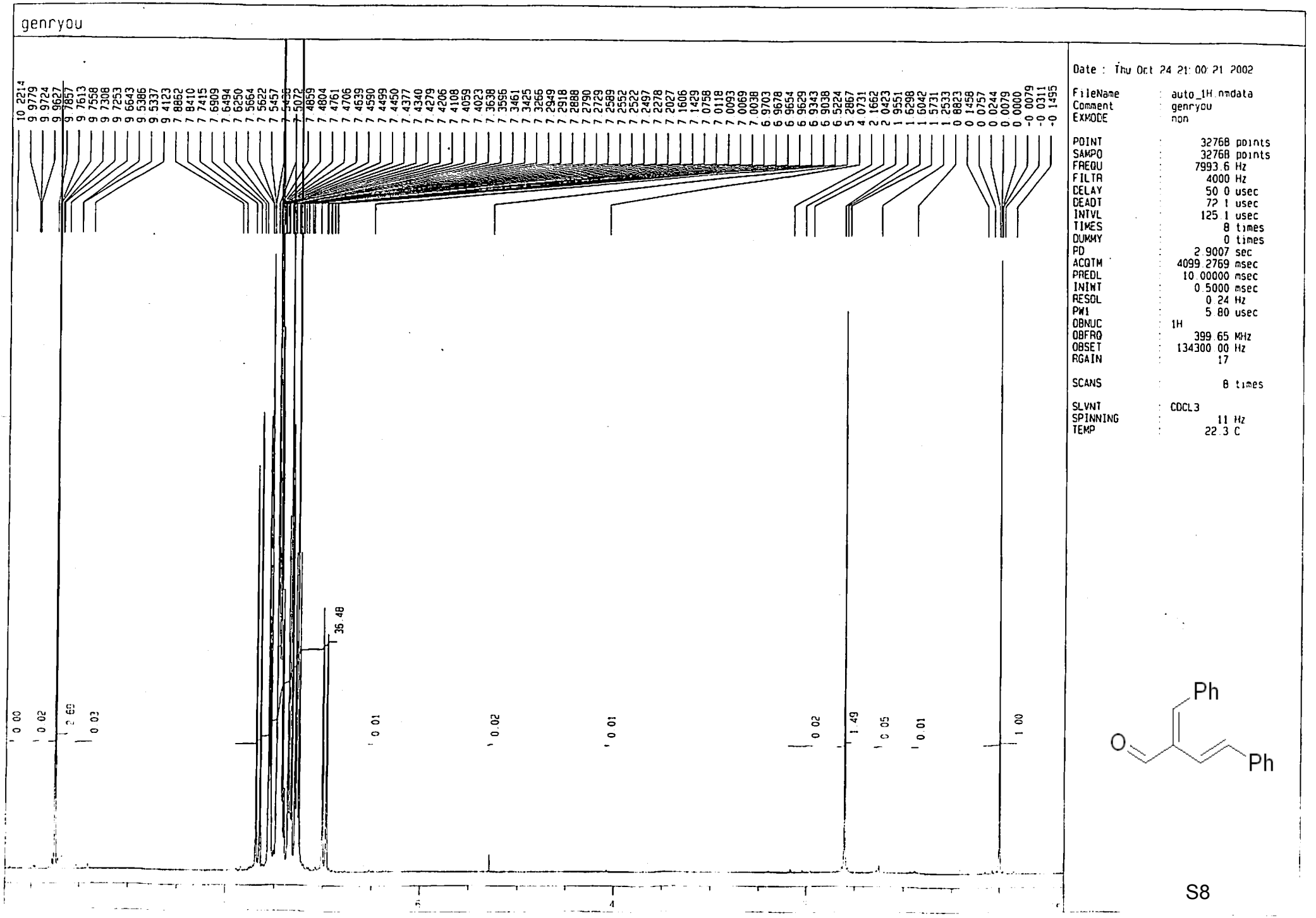
Colorless crystals; ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.81 (ddd, 1H, *J* = 5.1, 7.5, 15.5 Hz, H-7), 1.97 (s, 3H, Me (COMe)), 2.44 (s, 3H, Me (Ts)), 2.75 (ddd, 1H, *J* = 1.6, 2.9, 15.5 Hz, H-7'), 2.99 (dd, 1H, *J* = 3.4, 8.2 Hz, H-8), 3.22 (ddd, 1H, *J* = 3.4, 3.6, 7.3 Hz, H-9a), 3.47 (m, 1H, H-6), 3.56 (d, 1H, *J* = 15.5 Hz, CH_2 (Bn)), 3.76 (s, 3H, Me (CO_2Me)), 4.95 (d, 1H, *J* = 15.5 Hz, CH_2 (Bn)), 6.23–6.25 (m, 1H, H-5), 6.24 (s, 1H, H-4), 6.54 (d, 2H, *J* = 7.7 Hz, Ar), 6.99 (dd, 2H, *J* = 7.4, 7.5 Hz, Ar), 7.09 (dd, 1H, *J* = 7.0, 7.4 Hz, Ar), 7.29–7.36 (m, 7H, Ar), 7.99 (d, 2H, *J* = 8.4 Hz, Ar); ^{13}C NMR (151 MHz, $CDCl_3$) δ 21.86 (CH_3), 24.46 (CH_2), 29.20 (CH_3), 37.99 (CH), 44.35 (CH), 47.85 (CH_2), 52.55 (CH_3), 52.79 (CH), 62.41 (CH), 121.91 (CH), 125.90 (2CH), 127.64 (CH), 127.84 (2CH), 127.86 (CH), 128.72 (2CH), 129.16 (2CH), 129.20 (2CH), 129.33 (2CH), 132.19 (C), 135.23 (C), 136.50 (C), 138.17 (C), 144.25 (C), 152.58 (C), 172.17 (C), 205.24 (C); HRMS-ESI calcd for $C_{32}H_{32}N_2O_6SNa$ $[M+Na]^+$: 595.1877, found: 595.1873.

8-Acetyl-1-benzyl-2-oxo-4-phenyl-3-(toluene-4-sulfonyl)-1,2,3,4,6,7,8,8a-octahydroquinazoline-6-carboxylic acid methyl ester (*exo*-6d)



Colorless crystals; ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.87 (ddd, 1H, *J* = 4.2, 12.2, 15.8 Hz, H-7), 1.99 (s, 3H, Me (COMe)), 2.34–2.40 (m, 1H, H-7'), 2.47 (s, 3H, Me (Ts)), 3.12 (dd, 1H, *J* = 4.2, 8.3 Hz, H-8), 3.31–3.37 (m, 1H, H-9a), 3.55–3.59 (m, 1H, H-6), 3.66 (d, 1H, *J* = 15.5 Hz, CH_2 (Bn)), 3.77 (s, 3H, Me (CO_2Me)), 5.01 (d, 1H, *J* = 15.5 Hz, CH_2 (Bn)), 6.12 (dd, 2H, *J* = 2.7, 2.9 Hz, H-5), 6.22 (s, 1H, H-4), 6.51 (d, 2H, *J* = 7.7 Hz, Ar), 6.99 (dd, 1H, *J* = 7.5, 7.5 Hz, Ar), 7.11 (dd, 1H, *J* = 6.9, 7.2 Hz, Ar), 7.24–7.27 (m, 2H, Ar), 7.31–7.38 (m, 5H, Ar), 8.03 (d, 2H, *J* = 8.0 Hz, Ar); ^{13}C NMR (151 MHz, $CDCl_3$) δ 21.87 (CH_3), 26.34 (CH_2), 28.60 (CH_3), 39.42 (CH), 46.03 (CH), 47.85 (CH_2), 52.61 (CH_3), 52.69 (CH), 62.25 (CH), 122.67 (CH), 125.82 (2CH), 127.73 (CH), 127.84 (2CH), 127.88 (CH), 128.75 (2CH), 129.20 (2CH), 129.25 (2CH), 129.41 (2CH), 132.42 (C), 135.00 (C), 136.68 (C), 137.73 (C), 144.45 (C), 152.56 (C), 173.10 (C), 205.15 (C); HRMS-ESI calcd for $C_{32}H_{32}N_2O_6SNa$ $[M+Na]^+$: 595.1869, found: 595.1873.

^{1}H NMR (**1a**)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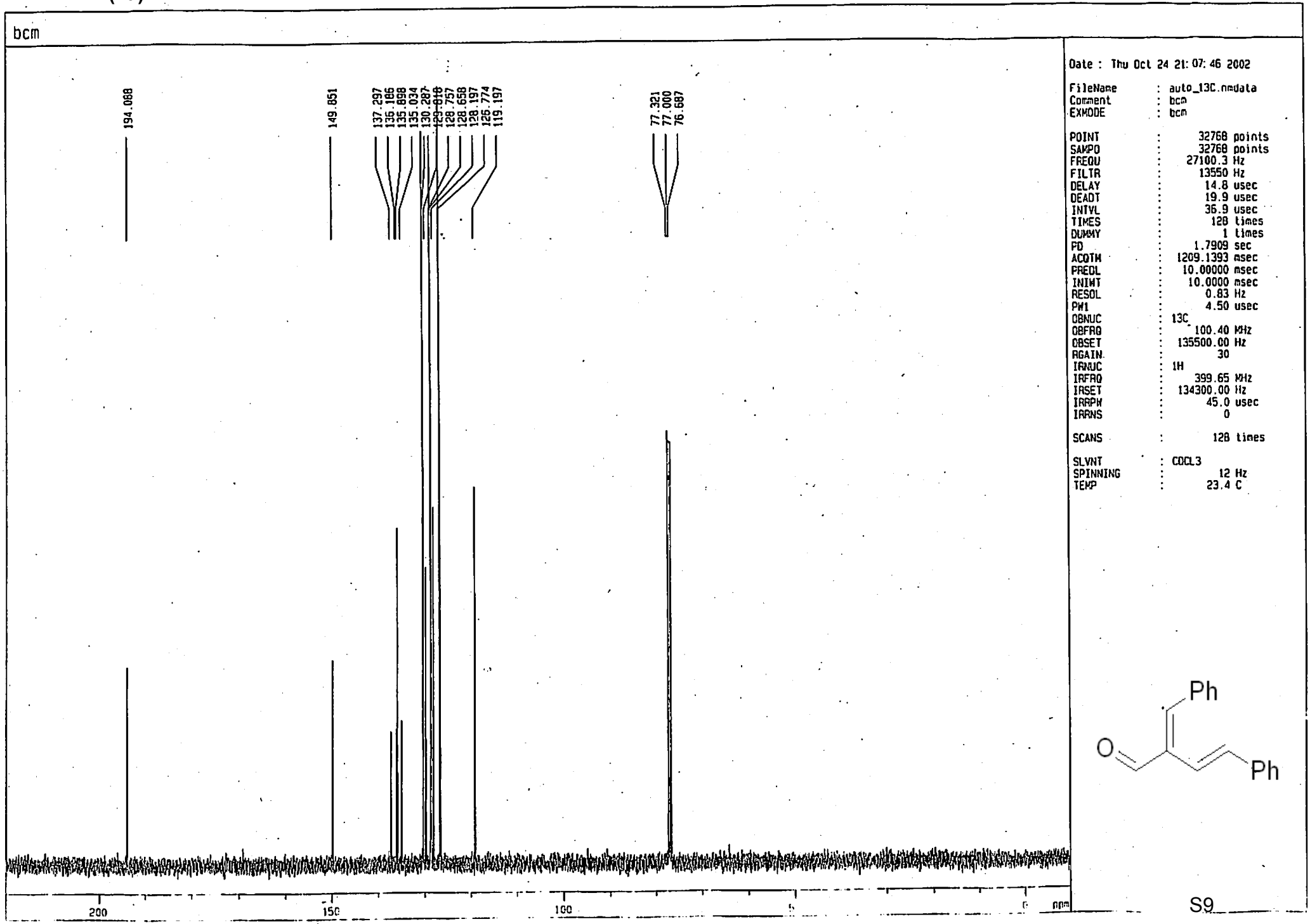
Ph

^{13}C NMR (1a)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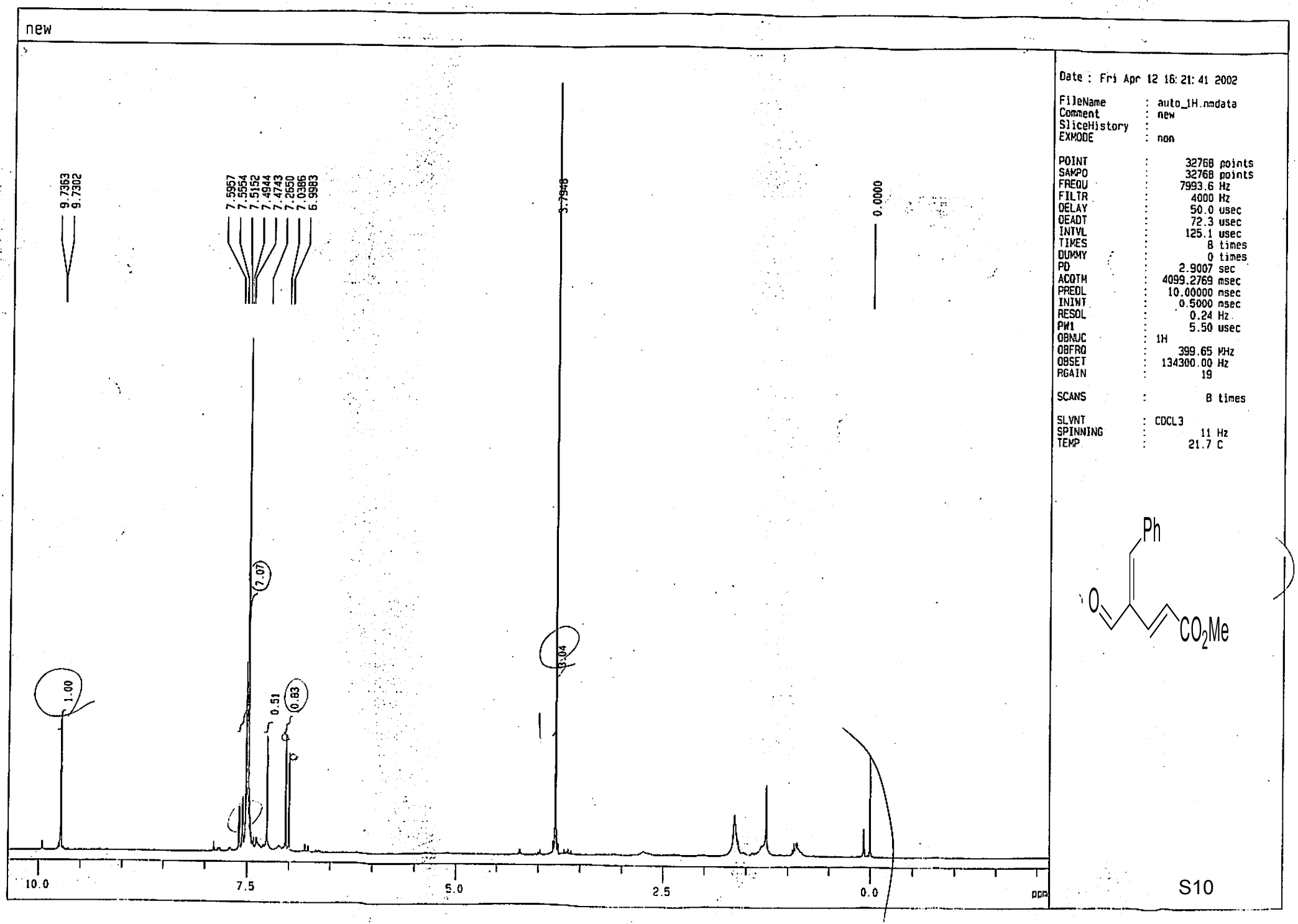
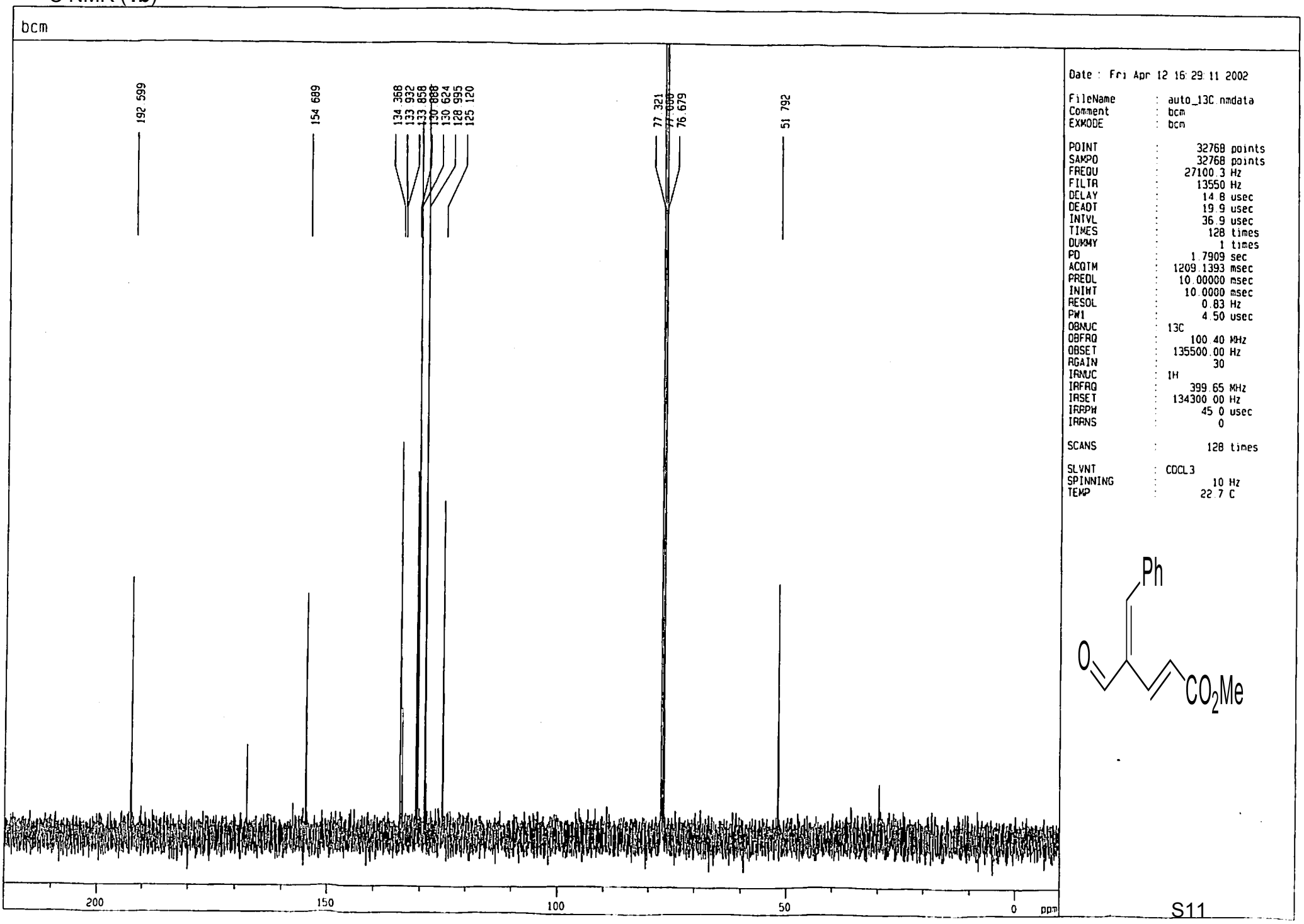
O
Ph

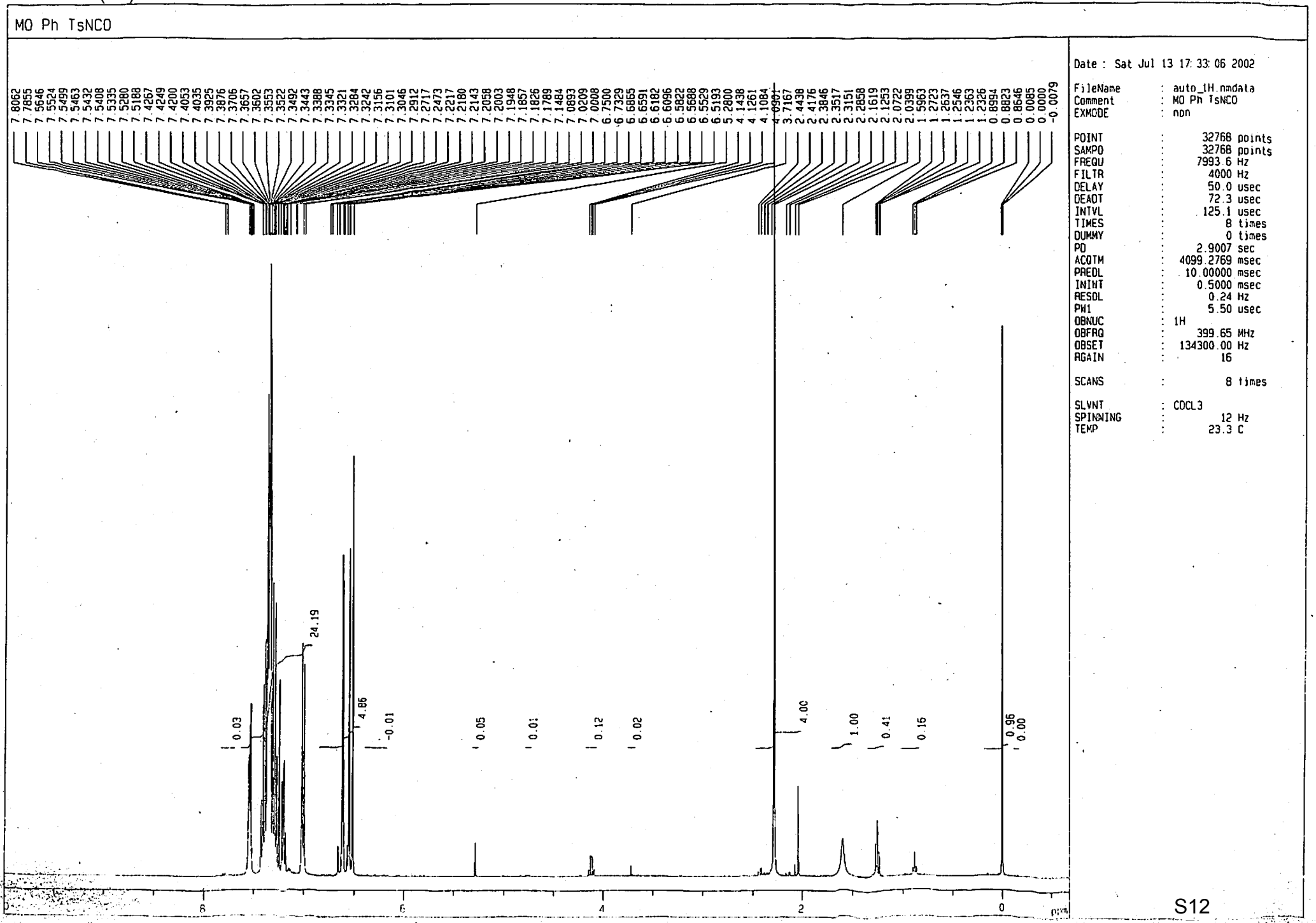
^{1}H NMR (**1b**)


new
9.7363
9.7302
7.5957
7.5554
7.5152
7.4944
7.4743
7.2650
7.0386
6.9983
3.7948
0.0000
1.00
2.07
0.51
0.83
3.04
10.0
7.5
5.0
2.5
0.0
ppm
Date : Fri Apr 12 16:21:41 2002
FileName : auto_1H.nmdata
Comment : new
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7993.6 Hz
FILTR : 4000 Hz
DELAY : 50.0 usec
DEADT : 72.3 usec
INTVL : 125.1 usec
TIMES : 8 times
DUMMY : 0 times
PD : 2.9007 sec
ACQTM : 4099.2769 msec
PREDL : 10.00000 msec
INIWT : 0.5000 msec
RESOL : 0.24 Hz
PW1 : 5.50 usec
OBNUC : 1H
OBFRQ : 399.65 MHz
OBSET : 134300.00 Hz
RGAIN : 19
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 11 Hz
TEMP : 21.7 C
Ph
O
CO2Me

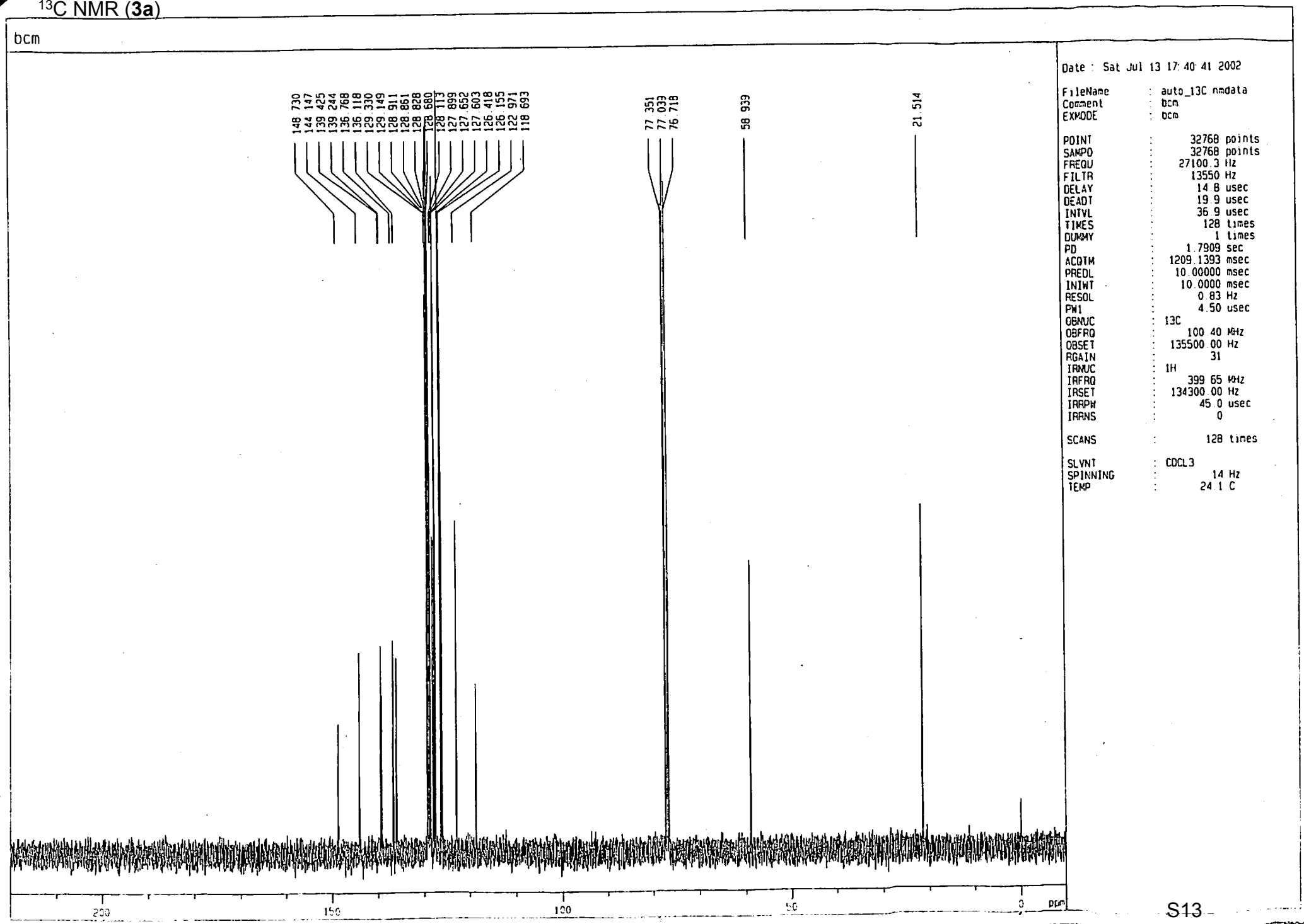
^{13}C NMR (**1b**)


bcm
192.599
154.689
134.368
133.932
133.858
130.888
130.624
128.995
125.120
77.321
76.679
51.792
200
150
100
50
0
ppm
Date : Fri Apr 12 16:29:11 2002
FileName : auto_13C.nmdata
Comment : bcm
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 27100.3 Hz
FILTR : 13550 Hz
DELAY : 14.8 usec
DEADT : 19.9 usec
INTVL : 36.9 usec
TIMES : 128 times
DUMMY : 1 times
PD : 1.7909 sec
ACQTM : 1209.1393 msec
PREDL : 10.00000 msec
INIHT : 10.0000 msec
RESOL : 0.83 Hz
PW1 : 4.50 usec
OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz
RGAIN : 30
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134300.00 Hz
IRPPH : 45.0 usec
IRANS : 0
SCANS : 128 times
SLVNT : CDCL3
SPINNING : 10 Hz
TEMP : 22.7 C
Ph
O
CO2Me

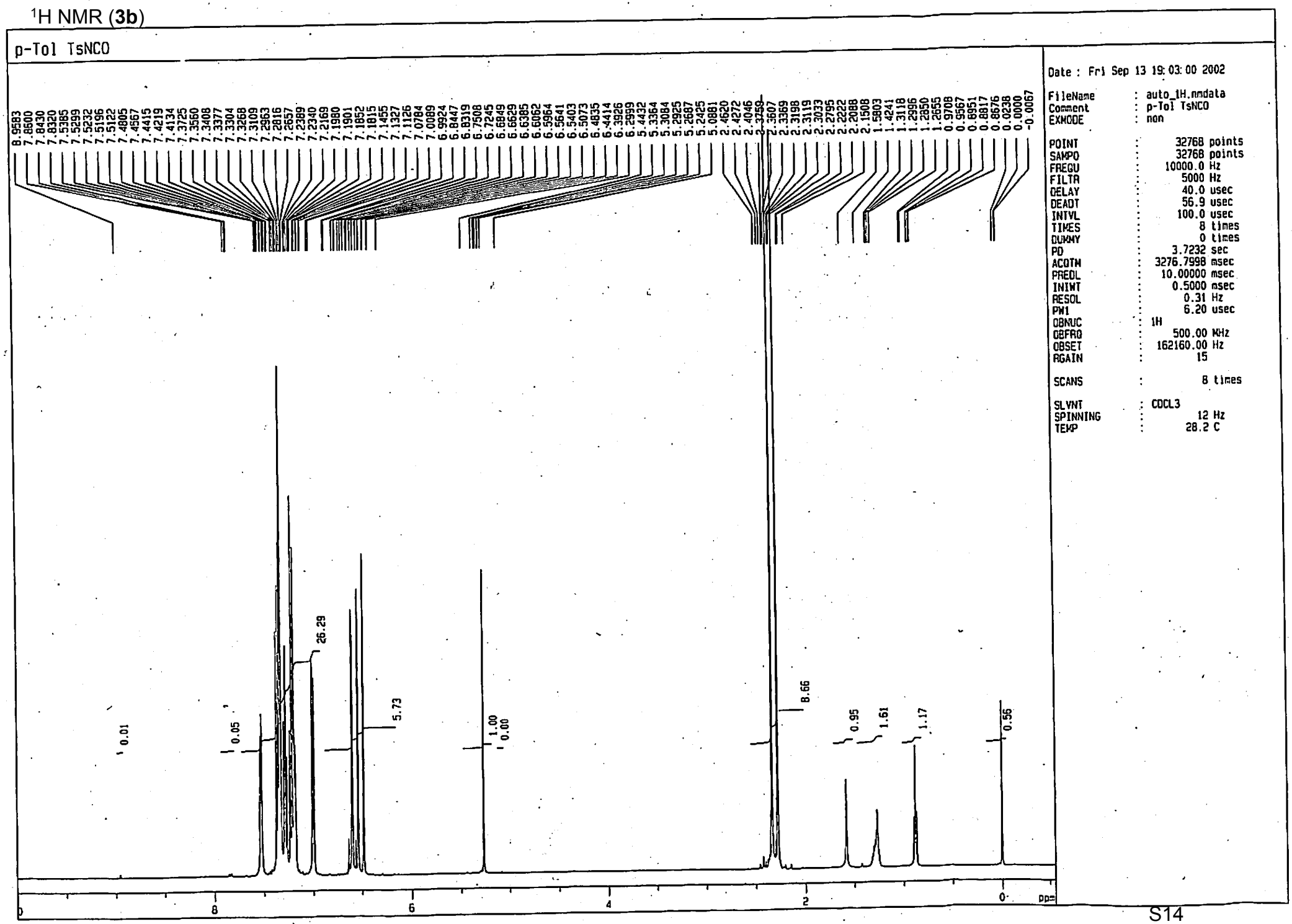
^{1}H NMR (**3a**)


MO Ph TsNCO
7.8062 7.7855 7.5646 7.5524 7.5499 7.5463 7.5432 7.5408 7.5335 7.5280 7.5188 7.4267 7.4249 7.4200 7.4053 7.4035 7.3925 7.3876 7.3706 7.3657 7.3602 7.3553 7.3522 7.3492 7.3443 7.3388 7.3345 7.3321 7.3284 7.3242 7.3156 7.3101 7.3046 7.2912 7.2717 7.2473 7.2217 7.2180 7.2143 7.2058 7.2003 7.1948 7.1857 7.1826 7.1789 7.1484 7.0893 7.0209 7.0008 6.7500 6.7329 6.6865 6.6591 6.6182 6.6096 6.5822 6.5688 6.5529 6.5193 5.2800 4.1438 4.1261 4.1084 4.0901 3.7167 2.4438 2.4176 2.3846 2.3517 2.3151 2.2858 2.1619 2.1253 2.0722 2.0399 1.5963 1.2723 1.2637 1.2546 1.2363 1.2326 0.8994 0.8823 0.8646 0.0085 0.0000 -0.0079
0.03
24.19
4.86
-0.01
0.05
0.01
0.12
0.02
4.00
1.00
0.41
0.15
0.96
0.00
Date : Sat Jul 13 17:33:06 2002
FileName : auto_1H.nmdata
Comment : MO Ph TsNCO
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7993.6 Hz
FILTR : 4000 Hz
DELAY : 50.0 usec
DEADT : 72.3 usec
INTVL : 125.1 usec
TIMES : 8 times
DUMMY : 0 times
PD : 2.9007 sec
ACQTM : 4099.2769 msec
PREDL : 10.00000 msec
ININT : 0.5000 msec
RESOL : 0.24 Hz
PW1 : 5.50 usec
OBNUC : 1H
OBFRQ : 399.65 MHz
OBSET : 134300.00 Hz
RGAIN : 16
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 12 Hz
TEMP : 23.3 C
8
6
4
2
0
ppm

^{13}C NMR (**3a**)

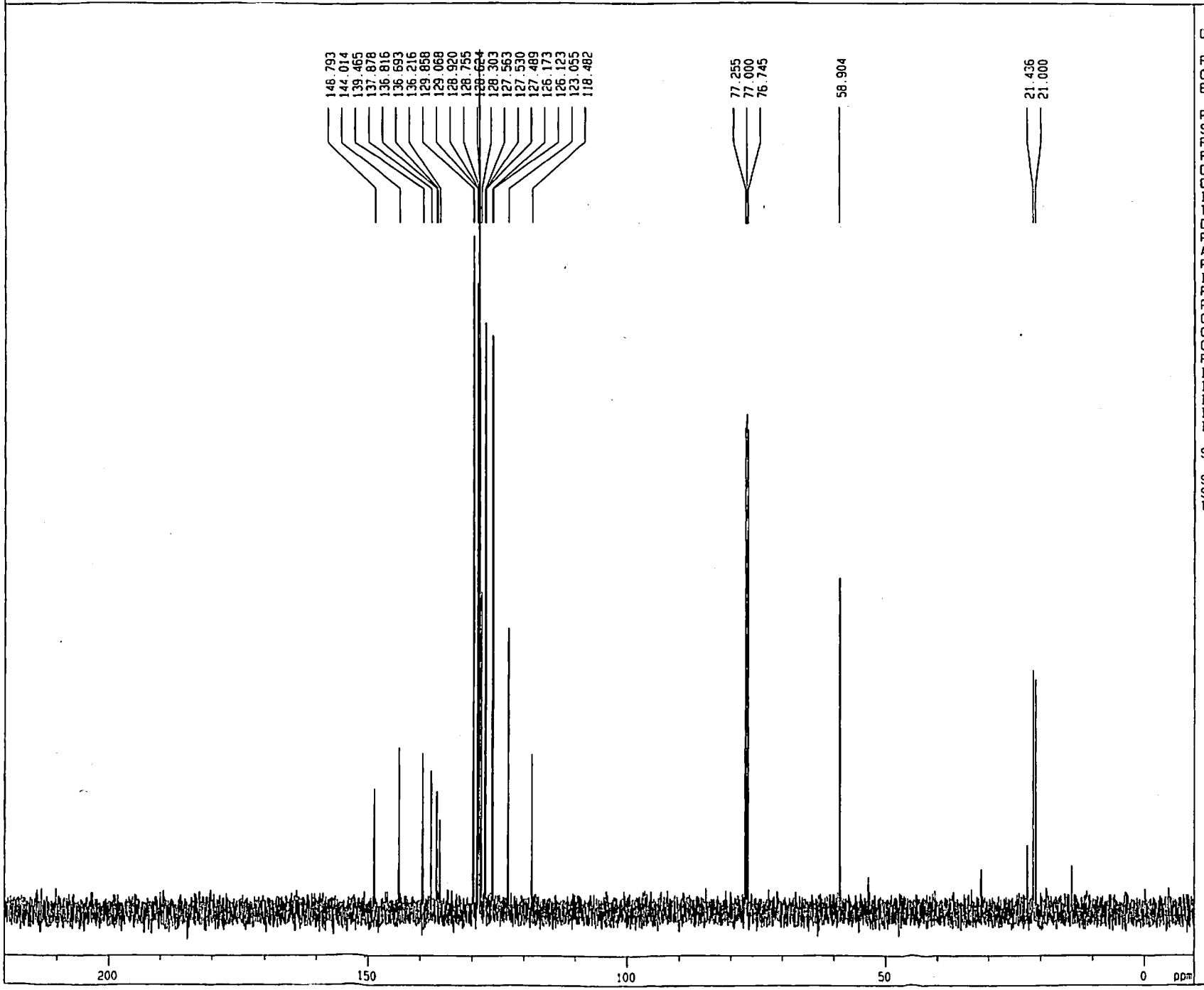

bcm
148.730
144.147
139.425
139.244
136.768
136.118
129.330
129.149
128.911
128.861
128.828
128.680
128.113
127.899
127.652
127.603
126.418
126.155
122.971
118.693
77.351
77.039
76.718
58.939
21.514
200
150
100
50
0
ppm
Date : Sat Jul 13 17:40:41 2002
FileName : auto_13C nmdata
Comment : bcm
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 27100.3 Hz
FILTR : 13550 Hz
DELAY : 14.8 usec
DEADT : 19.9 usec
INTVL : 36.9 usec
TIMES : 128 times
DUMMY : 1 times
PD : 1.7909 sec
ACQTM : 1209.1393 msec
PREDL : 10.00000 msec
INIWT : 10.0000 msec
RESOL : 0.83 Hz
PW1 : 4.50 usec
OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz
RGAIN : 31
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134300.00 Hz
IRPPH : 45.0 usec
IRRNS : 0
SCANS : 128 times
SLVNT : CDCL3
SPINNING : 14 Hz
TEMP : 24.1 C

^{1}H NMR (**3b**)


p-Tol TsNCO
Date : Fri Sep 13 19:03:00 2002
FileName : auto_1H.nmdata
Comment : p-Tol TsNCO
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 10000.0 Hz
FILTR : 5000 Hz
DELAY : 40.0 usec
DEADT : 56.9 usec
INTVL : 100.0 usec
TIMES : 8 times
DUMMY : 0 times
PD : 3.7232 sec
ACQTM : 3276.7998 msec
PREDL : 10.00000 msec
ININT : 0.5000 msec
RESOL : 0.31 Hz
PW1 : 6.20 usec
OBNUC : 1H
OBFRQ : 500.00 MHz
OBSET : 162160.00 Hz
RGAIN : 15
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 12 Hz
TEMP : 28.2 C
0.01
0.05
26.29
5.73
1.00
0.00
8.66
0.95
1.61
1.17
0.56
ppm

^{1}H NMR (**3b**)

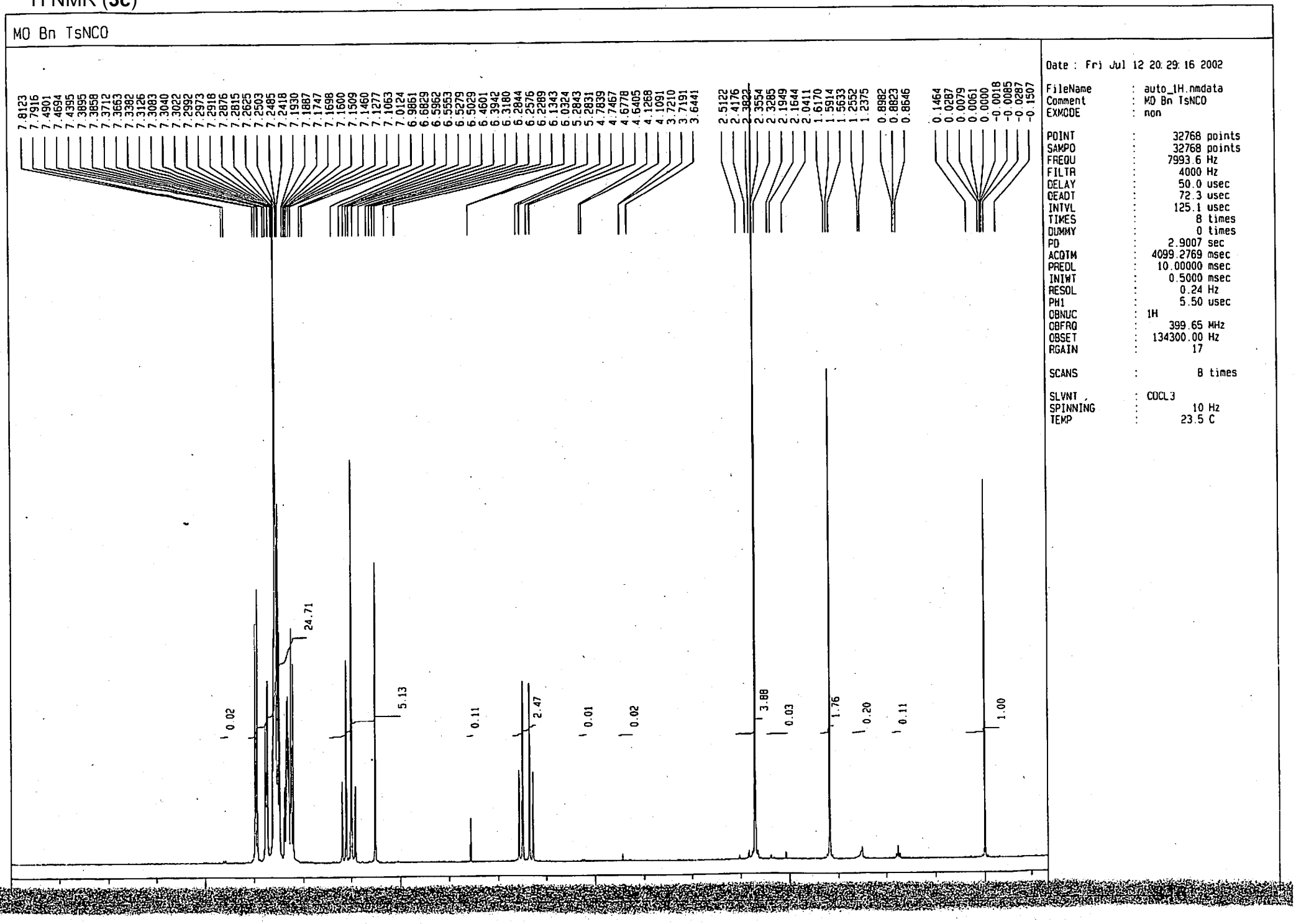
bcm



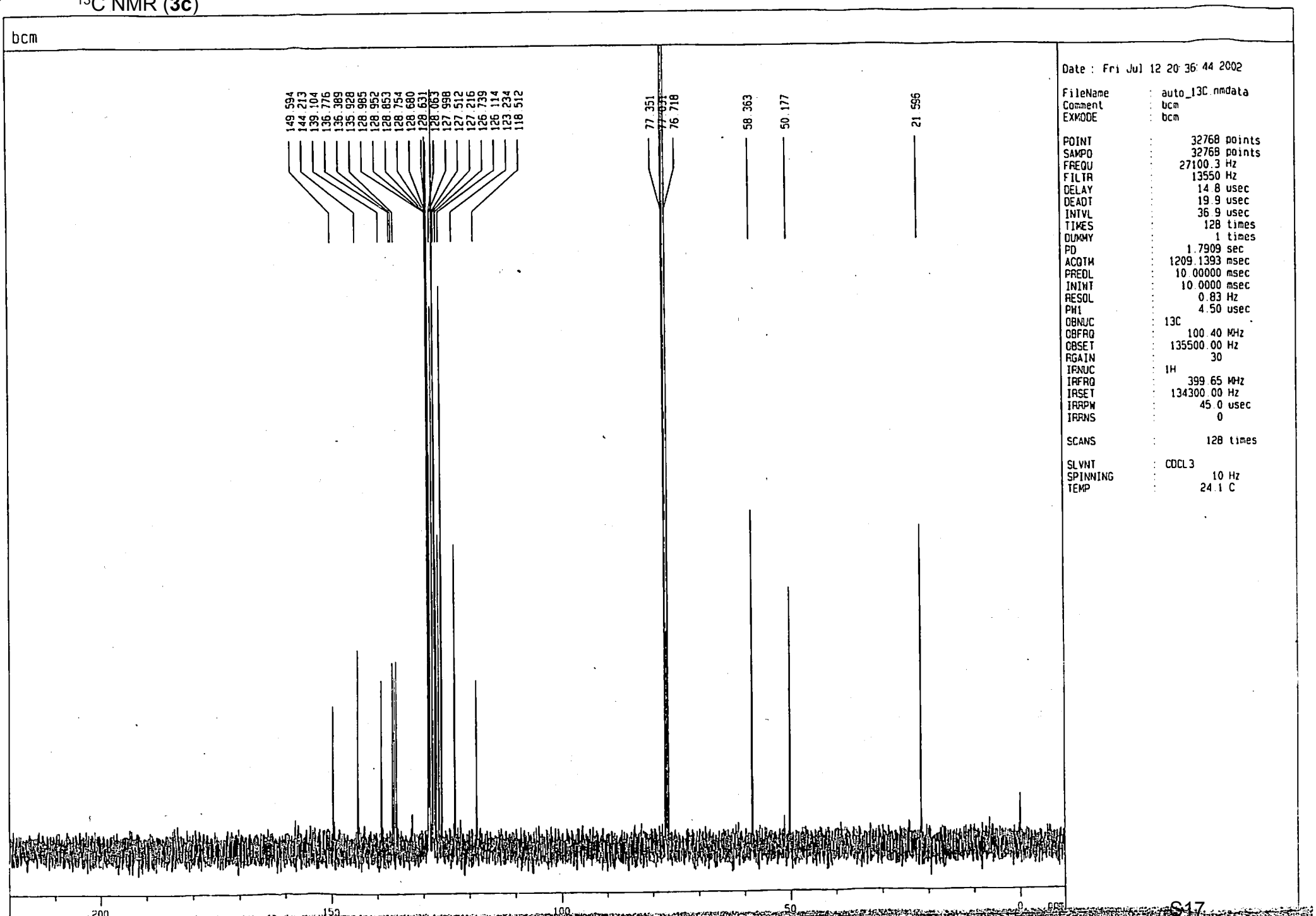
Date : Fri Sep 13 19:10:26 2002

FileName	:	auto_13C.nmdata
Comment	:	bcm
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	33898.3 Hz
FILTR	:	16950 Hz
DELAY	:	11.8 usec
DEADT	:	10.0 usec
INTVL	:	29.5 usec
TIMES	:	128 times
DUMMY	:	1 times
PD	:	2.0333 sec
ACQTM	:	966.6560 msec
PREDL	:	10.00000 msec
INIWT	:	10.0000 msec
RESOL	:	1.03 Hz
PWI	:	5.50 usec
OBNUC	:	13C
OBFRQ	:	125.65 MHz
OBSET	:	127958.00 Hz
RGAIN	:	30
IRNUC	:	1H
IRFRQ	:	500.00 MHz
IRSET	:	162160.00 Hz
IRPPW	:	48.0 usec
IRRNS	:	0
SCANS	:	128 times
SLVNT	:	CDCL3
SPINNING	:	11 Hz
TEMP	:	29.6 C

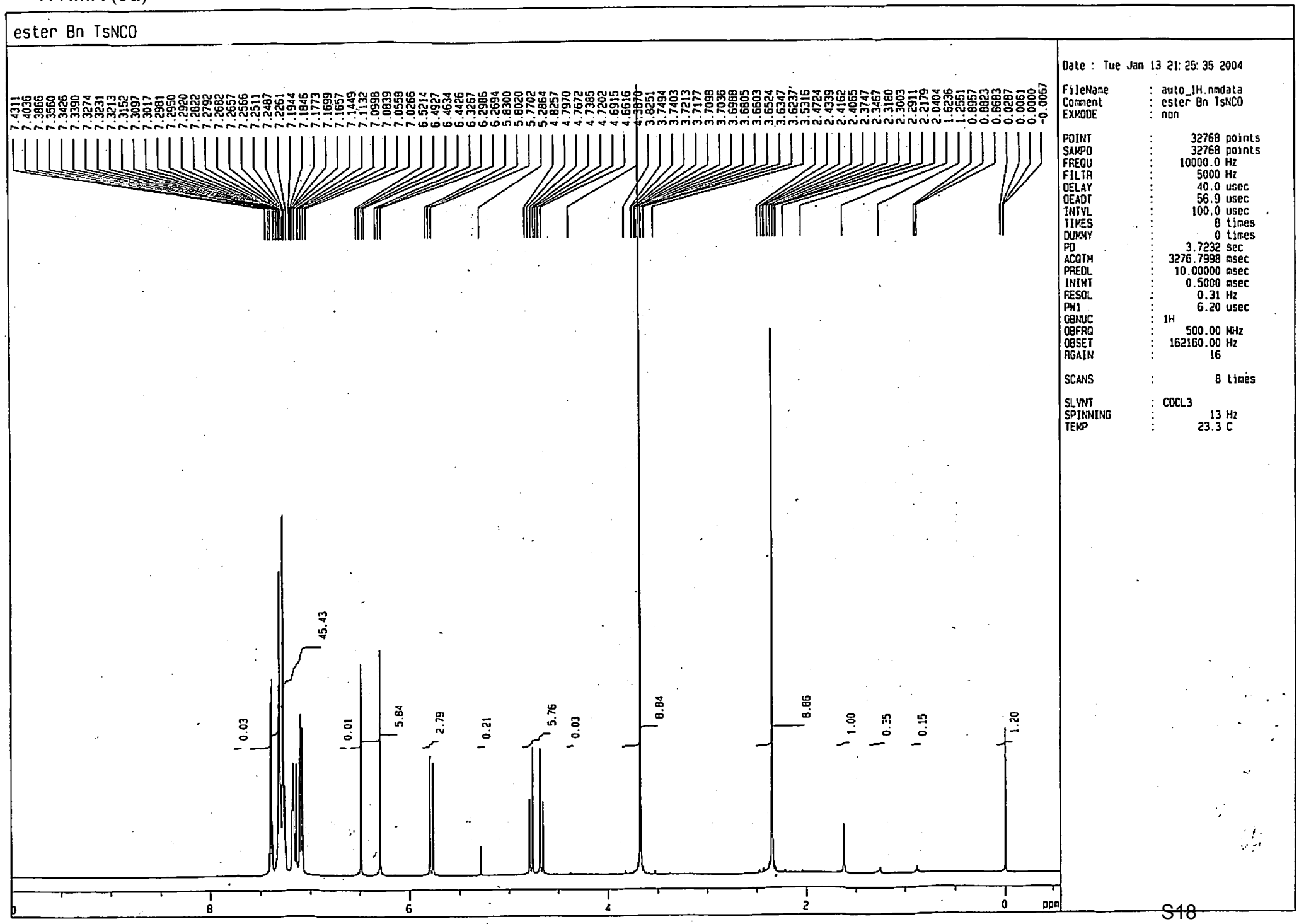
^{1}H NMR (**3c**)


MO Bn TsNCO
7.8123
7.7916
7.4901
7.4694
7.4395
7.3895
7.3858
7.3712
7.3663
7.3382
7.3126
7.3083
7.3040
7.3022
7.2992
7.2973
7.2918
7.2876
7.2815
7.2625
7.2503
7.2485
7.2418
7.1930
7.1887
7.1747
7.1698
7.1600
7.1509
7.1460
7.1277
7.1063
7.0124
6.9861
6.6829
6.5962
6.5553
6.5279
6.5029
6.4601
6.3942
6.3180
6.2844
6.2576
6.2289
6.1343
6.0324
5.2843
5.2831
4.7839
4.7467
4.6778
4.6405
4.1268
4.1091
3.7210
3.7191
3.6441
2.5122
2.4176
2.3822
2.3554
2.3285
2.1949
2.1644
2.0411
1.6170
1.5914
1.5633
1.2552
1.2375
0.8982
0.8823
0.8646
0.1464
0.0287
0.0079
0.0061
0.0000
-0.0018
-0.0085
-0.0287
-0.1507
0.02
24.71
5.13
0.11
2.47
0.01
0.02
3.88
0.03
1.76
0.20
0.11
1.00
Date : Fri Jul 12 20:29:16 2002
FileName : auto_1H.nmdata
Comment : MO Bn TsNCO
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7993.6 Hz
FILTR : 4000 Hz
DELAY : 50.0 usec
QEADT : 72.3 usec
INTVL : 125.1 usec
TIMES : 8 times
DUMMY : 0 times
PD : 2.9007 sec
ACQTM : 4099.2769 msec
PREDL : 10.00000 msec
INIWT : 0.5000 msec
RESOL : 0.24 Hz
PH1 : 5.50 usec
OBNUC : 1H
OBFRQ : 399.65 MHz
OBSET : 134300.00 Hz
RGAIN : 17
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 10 Hz
TEMP : 23.5 C

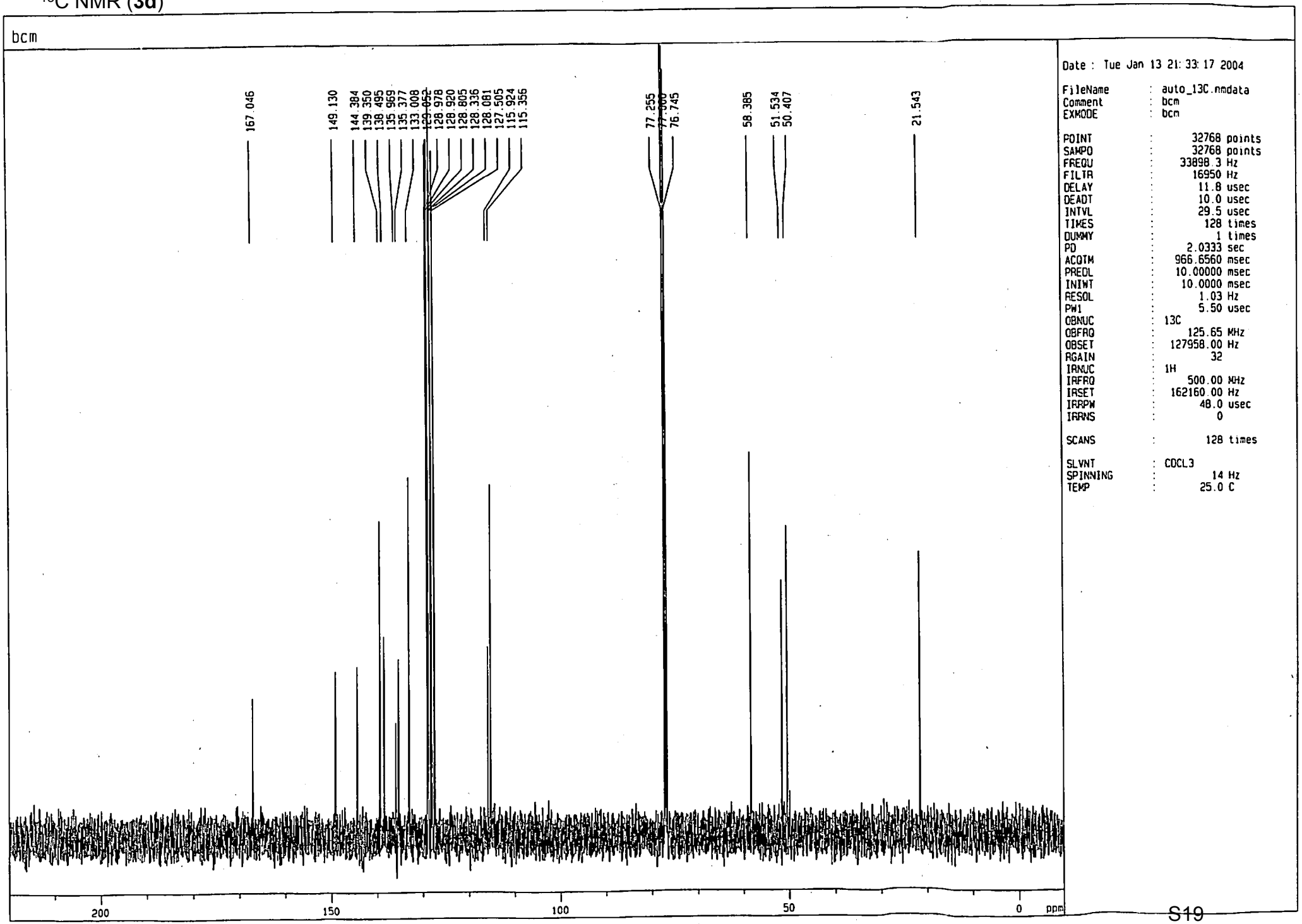
^{13}C NMR (**3c**)


bcm
149.594
144.213
139.104
136.776
136.389
135.928
128.985
128.952
128.853
128.754
128.680
128.631
128.063
127.998
127.512
127.216
126.739
126.114
123.234
118.512
77.351
77.031
76.718
58.363
50.177
21.596
Date : Fri Jul 12 20:36:44 2002
FileName : auto_13C.nmdata
Comment : bcm
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 27100.3 Hz
FILTR : 13550 Hz
DELAY : 14.8 usec
DEADT : 19.9 usec
INTVL : 36.9 usec
TIMES : 128 times
DUMMY : 1 times
PD : 1.7909 sec
ACQTM : 1209.1393 msec
PREDL : 10.00000 msec
ININT : 10.0000 msec
RESOL : 0.83 Hz
PW1 : 4.50 usec
OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz
RGAIN : 30
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134300.00 Hz
IRPW : 45.0 usec
IRNS : 0
SCANS : 128 times
SLVNT : CDCL3
SPINNING : 10 Hz
TEMP : 24.1 C
200
150
100
50
0
PPM

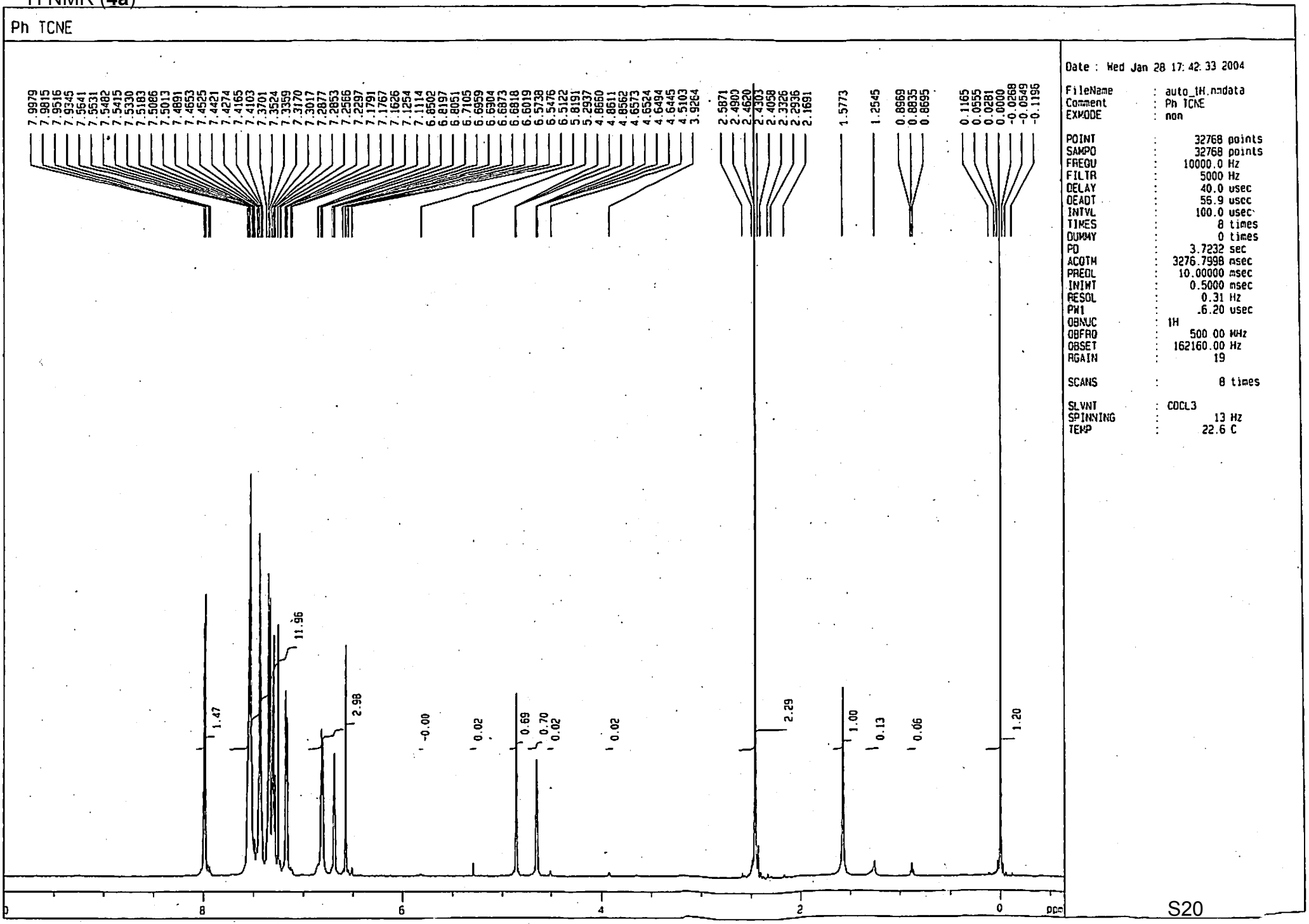
^{1}H NMR (**3d**)


ester Bn TsNCO
7.4311 7.4036 7.3866 7.3560 7.3426 7.3390 7.3274 7.3231 7.3213 7.3152 7.3097 7.3017 7.2981 7.2950 7.2920 7.2822 7.2792 7.2682 7.2657 7.2566 7.2511 7.2487 7.2261 7.1944 7.1846 7.1773 7.1699 7.1657 7.1449 7.1132 7.0998 7.0839 7.0558 7.0266 6.5214 6.4927 6.4634 6.4426 6.3267 6.2986 6.2694 5.8300 5.8020 5.7702 5.2864 4.8257 4.7970 4.7672 4.7385 4.7202 4.6915 4.6616 4.3870 3.8251 3.7494 3.7403 3.7213 3.7177 3.7098 3.7036 3.6988 3.6805 3.6603 3.6524 3.6347 3.6237 3.5316 2.4724 2.4339 2.4162 2.4065 2.3747 2.3467 2.3180 2.3003 2.2911 2.2179 2.0404 1.6236 1.2551 0.8957 0.8823 0.8683 0.0287 0.0061 0.0000 -0.0067
0.03
45.43
0.01
5.84
2.79
0.21
5.76
0.03
8.84
8.86
1.00
0.35
0.15
1.20
8
6
4
2
0
ppm
Date : Tue Jan 13 21:25:35 2004
FileName : auto_1H.nmdata
Comment : ester Bn TsNCO
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 10000.0 Hz
FILTR : 5000 Hz
DELAY : 40.0 usec
DEADT : 56.9 usec
INTVL : 100.0 usec
TIMES : 8 times
DUMMY : 0 times
PD : 3.7232 sec
ACQTM : 3276.7998 msec
PREDL : 10.00000 msec
ININT : 0.5000 msec
RESOL : 0.31 Hz
PW1 : 6.20 usec
OBNUC : 1H
OBFRQ : 500.00 MHz
OBSET : 162160.00 Hz
RGAIN : 16
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 23.3 C

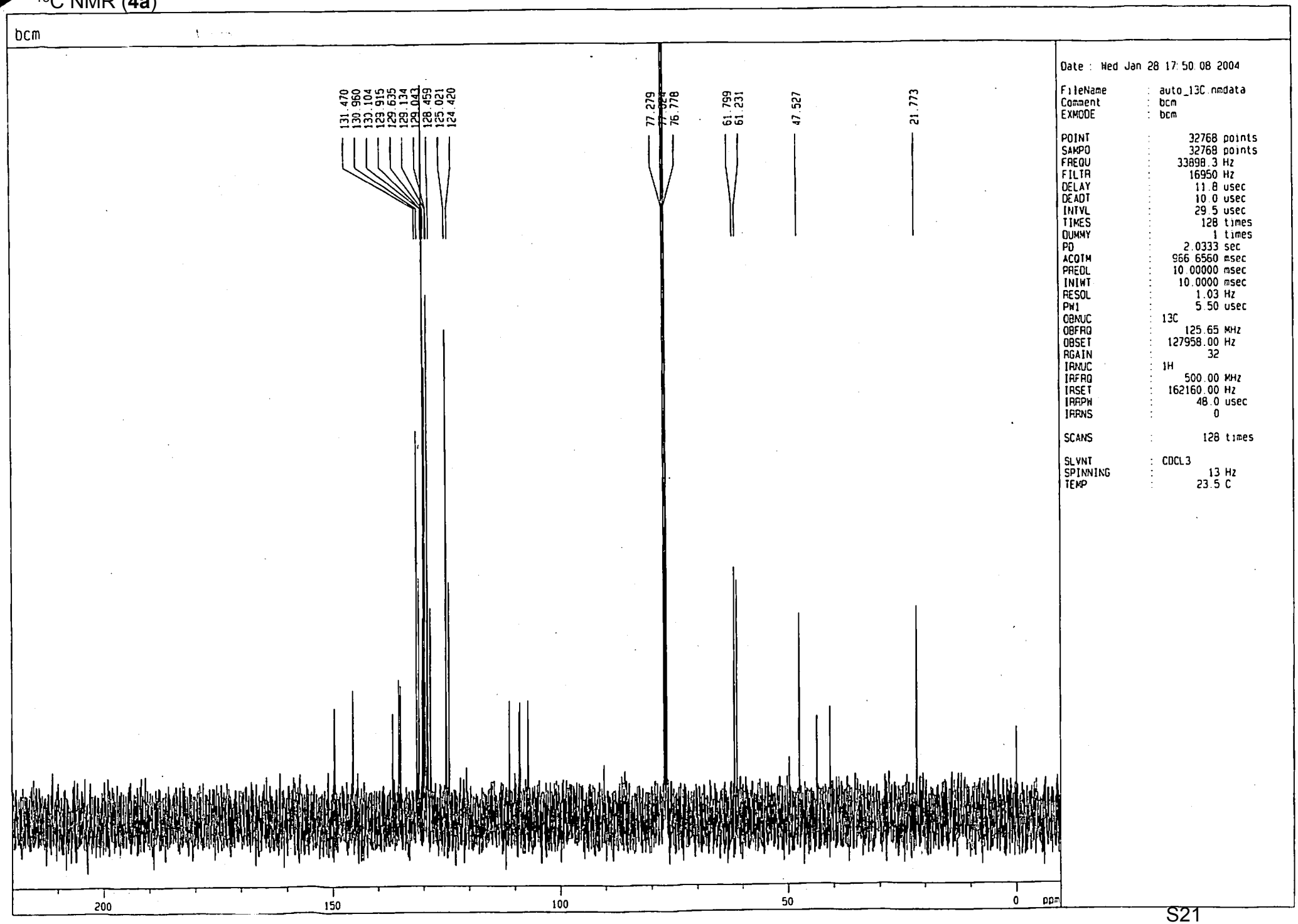
^{13}C NMR (**3d**)


bcm
167.046
149.130
144.384
139.350
138.495
135.969
135.377
133.008
129.052
128.978
128.920
128.805
128.336
128.081
127.505
115.924
115.356
77.255
77.000
76.745
58.385
51.534
50.407
21.543
200
150
100
50
0
ppm
Date : Tue Jan 13 21:33:17 2004
FileName : auto_13C.nmdata
Comment : bcm
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 33898.3 Hz
FILTR : 16950 Hz
DELAY : 11.8 usec
DEADT : 10.0 usec
INTVL : 29.5 usec
TIMES : 128 times
DUMMY : 1 times
PD : 2.0333 sec
ACQTM : 966.6560 msec
PREDL : 10.00000 msec
INIWT : 10.0000 msec
RESOL : 1.03 Hz
PW1 : 5.50 usec
OBNUC : 13C
OBFRQ : 125.65 MHz
OBSET : 127958.00 Hz
RGAIN : 32
IRNUC : 1H
IRFRQ : 500.00 MHz
IRSET : 162160.00 Hz
IRPW : 48.0 usec
IRNS : 0
SCANS : 128 times
SLVNT : CDCL3
SPINNING : 14 Hz
TEMP : 25.0 C

^{1}H NMR (**4a**)

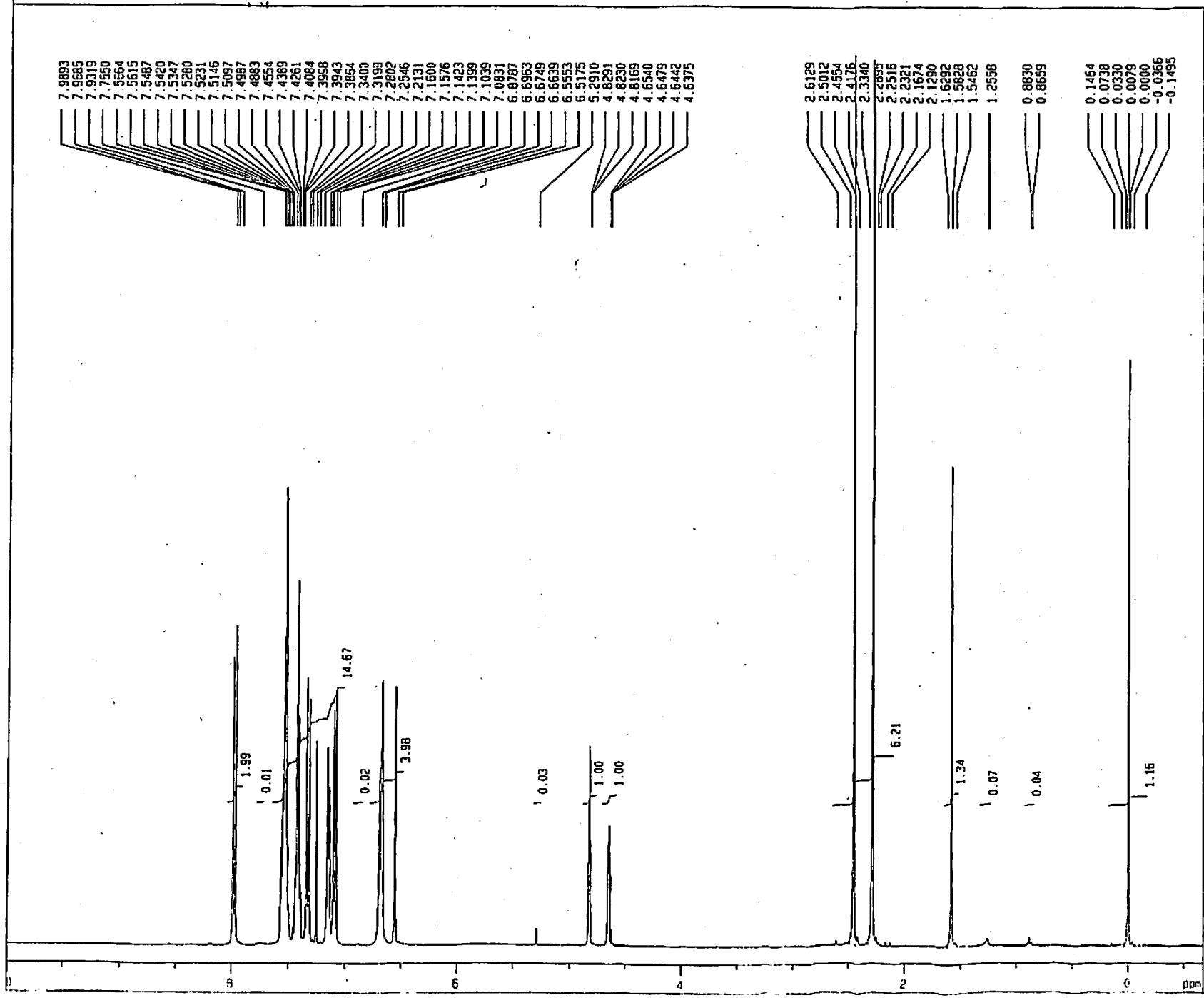

Ph TCNE
7.9979 7.9815 7.9516 7.9345 7.5641 7.5531 7.5482 7.5415 7.5330 7.5183 7.5086 7.5013 7.4891 7.4653 7.4525 7.4421 7.4274 7.4165 7.4103 7.3701 7.3524 7.3359 7.3170 7.3017 7.2877 7.2853 7.2566 7.2297 7.1791 7.1767 7.1626 7.1254 7.1114 6.8502 6.8197 6.8051 6.7105 6.6959 6.6304 6.6873 6.6818 6.6019 6.5738 6.5476 6.5122 5.8191 5.2937 4.8660 4.8611 4.8562 4.6573 4.6524 4.6494 4.6445 4.5103 3.9264
2.5871 2.4900 2.4620 2.4303 2.4058 2.3326 2.2936 2.1691
1.5773 1.2545 0.8969 0.8835 0.8695
0.1165 0.0555 0.0281 0.0000 -0.0268 -0.0549 -0.1196
11.96
1.47 2.98 -0.00 0.02 0.69 0.70 0.02 0.02 2.29 1.00 0.13 0.06 1.20
8 6 4 2 0 ppm
Date : Wed Jan 28 17:42:33 2004
FileName : auto_1H.nmdata
Comment : Ph TCNE
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 10000.0 Hz
FILTR : 5000 Hz
DELAY : 40.0 usec
DEADT : 56.9 usec
INTVL : 100.0 usec
TIMES : 8 times
DUMMY : 0 times
PD : 3.7232 sec
ACQTM : 3276.7998 msec
PREDL : 10.00000 msec
INIWT : 0.5000 msec
RESOL : 0.31 Hz
PW1 : 6.20 usec
OBNUC : 1H
OBFRQ : 500.00 MHz
OBSET : 162160.00 Hz
RGAIN : 19
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 22.6 C

^{13}C NMR (**4a**)


bcm
131.470
130.960
130.104
129.915
129.635
129.134
129.041
128.459
125.021
124.420
77.279
77.024
76.778
61.799
61.231
47.527
21.773
200
150
100
50
0
ppm
Date : Wed Jan 28 17:50:08 2004
FileName : auto_13C.nmdata
Comment : bcm
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 33898.3 Hz
FILTR : 16950 Hz
DELAY : 11.8 usec
DEADT : 10.0 usec
INTVL : 29.5 usec
TIMES : 128 times
DUMMY : 1 times
PD : 2.0333 sec
ACQTM : 966.6560 msec
PREDL : 10.00000 msec
INIWT : 10.0000 msec
RESOL : 1.03 Hz
PW1 : 5.50 usec
OBNUC : 13C
OBFRQ : 125.65 MHz
OBSET : 127958.00 Hz
RGAIN : 32
IRNUC : 1H
IRFRQ : 500.00 MHz
IRSET : 162160.00 Hz
IRPW : 48.0 usec
IRNS : 0
SCANS : 128 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 23.5 C

^{1}H NMR (**4b**)

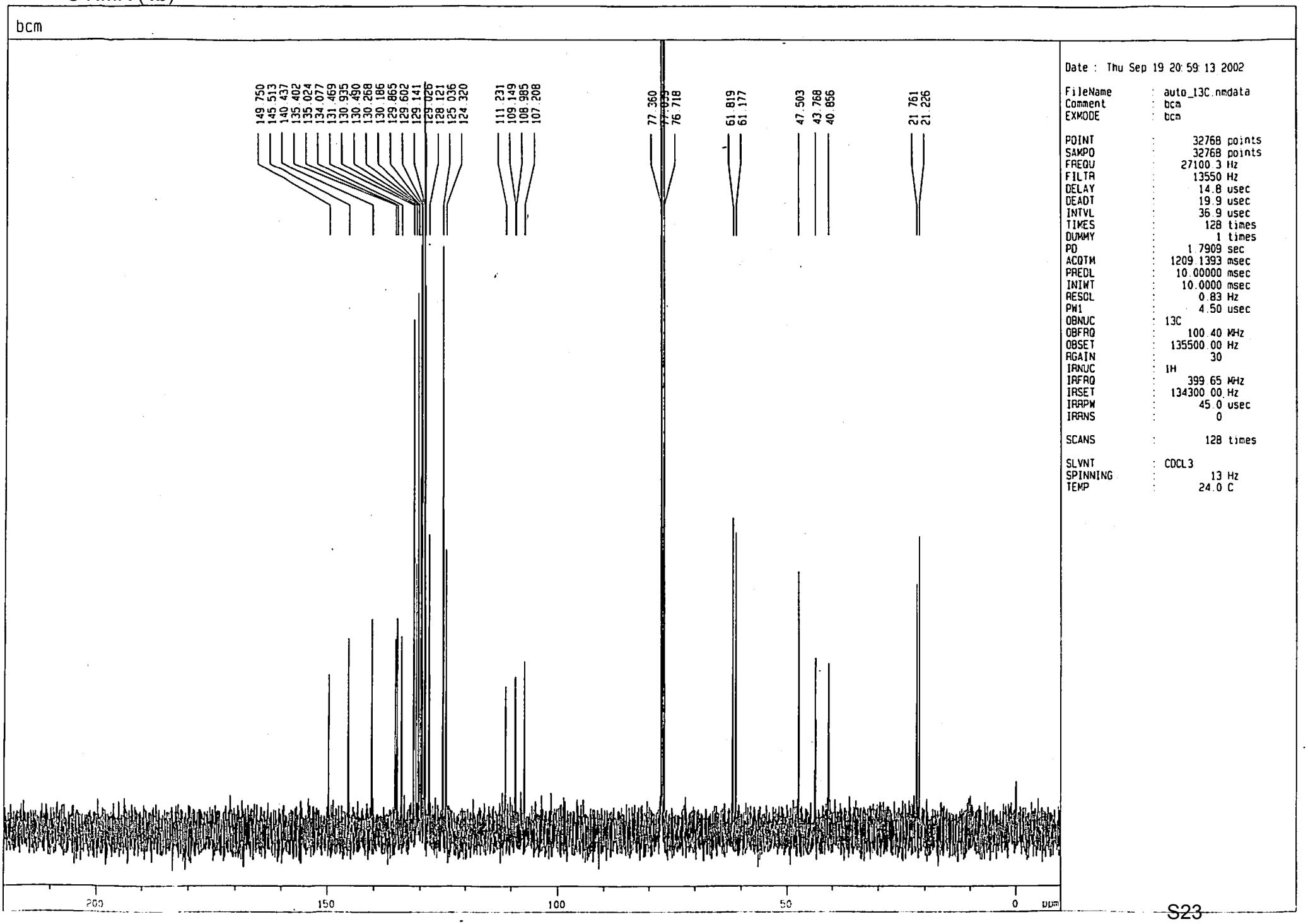
p-Tol TCNE



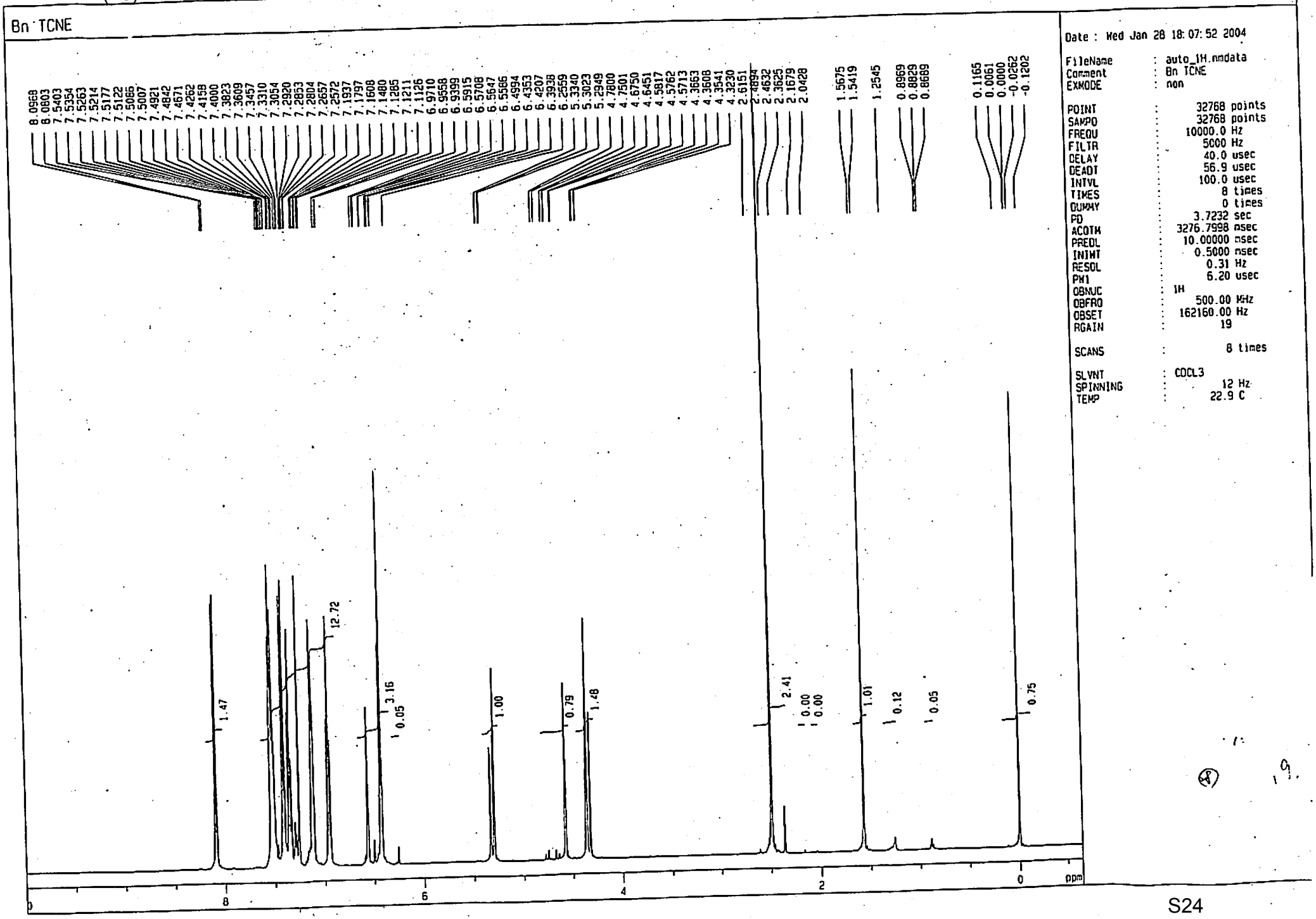
Date : Thu Sep 19 20:51:46 2002

FileName	:	auto_1H.nmdata
Comment	:	p-Tol TCNE
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	7993.6 Hz
FILTR	:	4000 Hz
DELAY	:	50.0 usec
DEADT	:	72.1 usec
INTVL	:	125.1 usec
TIMES	:	8 times
DUMMY	:	0 times
PD	:	2.9007 sec
ACQTM	:	4099.2769 msec
PREDL	:	10.00000 msec
ININT	:	0.5000 msec
RESOL	:	0.24 Hz
PW1	:	5.80 usec
OBNUC	:	1H
OBFRQ	:	399.65 MHz
OBSET	:	134300.00 Hz
RGAIN	:	17
SCANS	:	8 times
SLVNT	:	CDCL3
SPINNING	:	14 Hz
TEMP	:	23.3 C

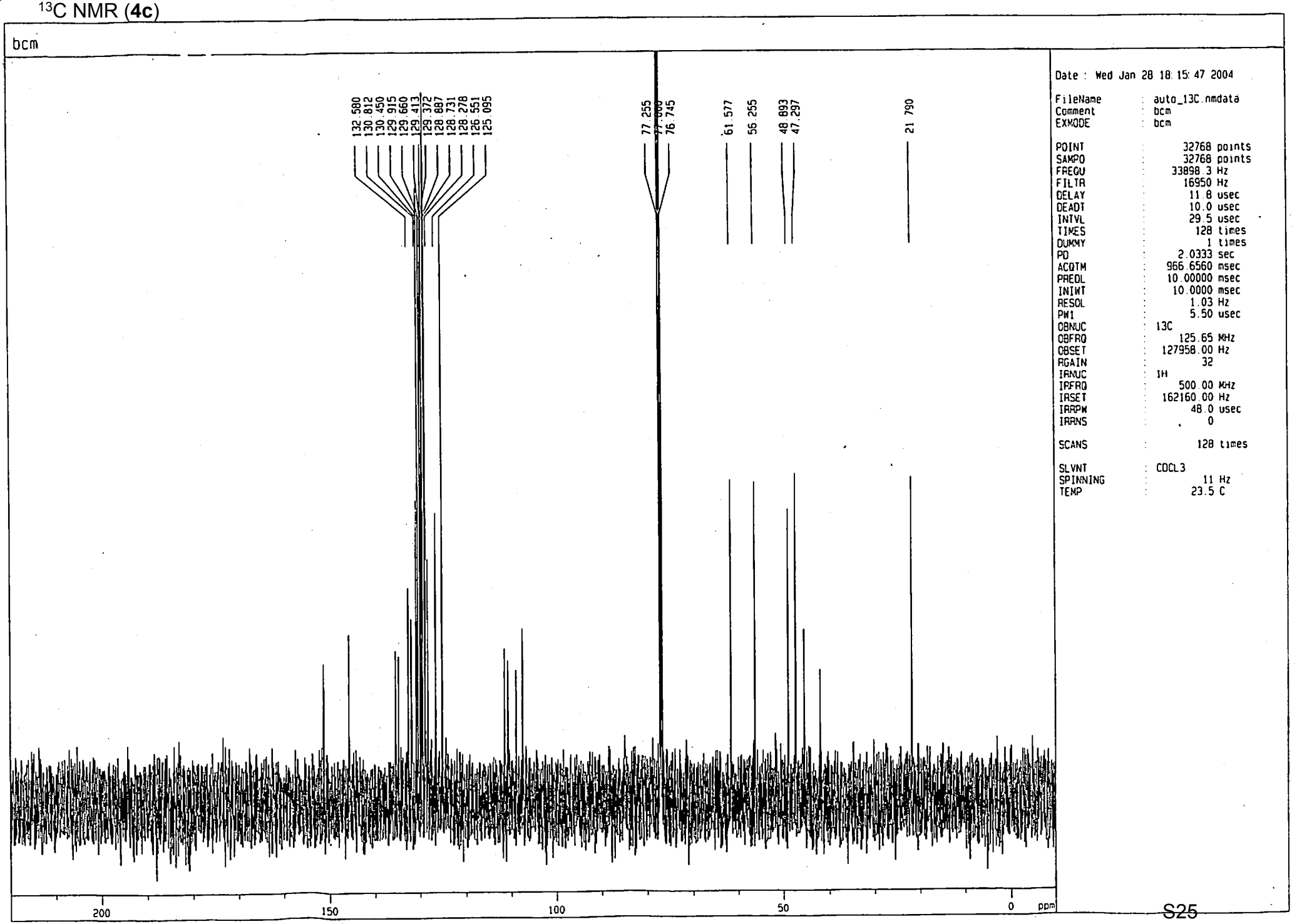
^{13}C NMR (**4b**)


bcm
149.750
145.513
140.437
135.402
135.024
134.077
131.469
130.935
130.490
130.268
130.186
129.865
129.602
129.141
129.026
128.121
125.036
124.320
111.231
109.149
108.985
107.208
77.360
77.039
76.718
61.819
61.177
47.503
43.768
40.856
21.761
21.226
200
150
100
50
0
ppm
Date : Thu Sep 19 20:59:13 2002
FileName : auto_13C.nmdata
Comment : bcm
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 27100.3 Hz
FILTR : 13550 Hz
DELAY : 14.8 usec
DEADT : 19.9 usec
INTVL : 36.9 usec
TIMES : 128 times
DUMMY : 1 times
PD : 1.7909 sec
ACQTM : 1209.1393 msec
PREDL : 10.00000 msec
INIWT : 10.0000 msec
RESOL : 0.83 Hz
PW1 : 4.50 usec
OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz
RGAIN : 30
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134300.00 Hz
IRAPW : 45.0 usec
IRANS : 0
SCANS : 128 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 24.0 C

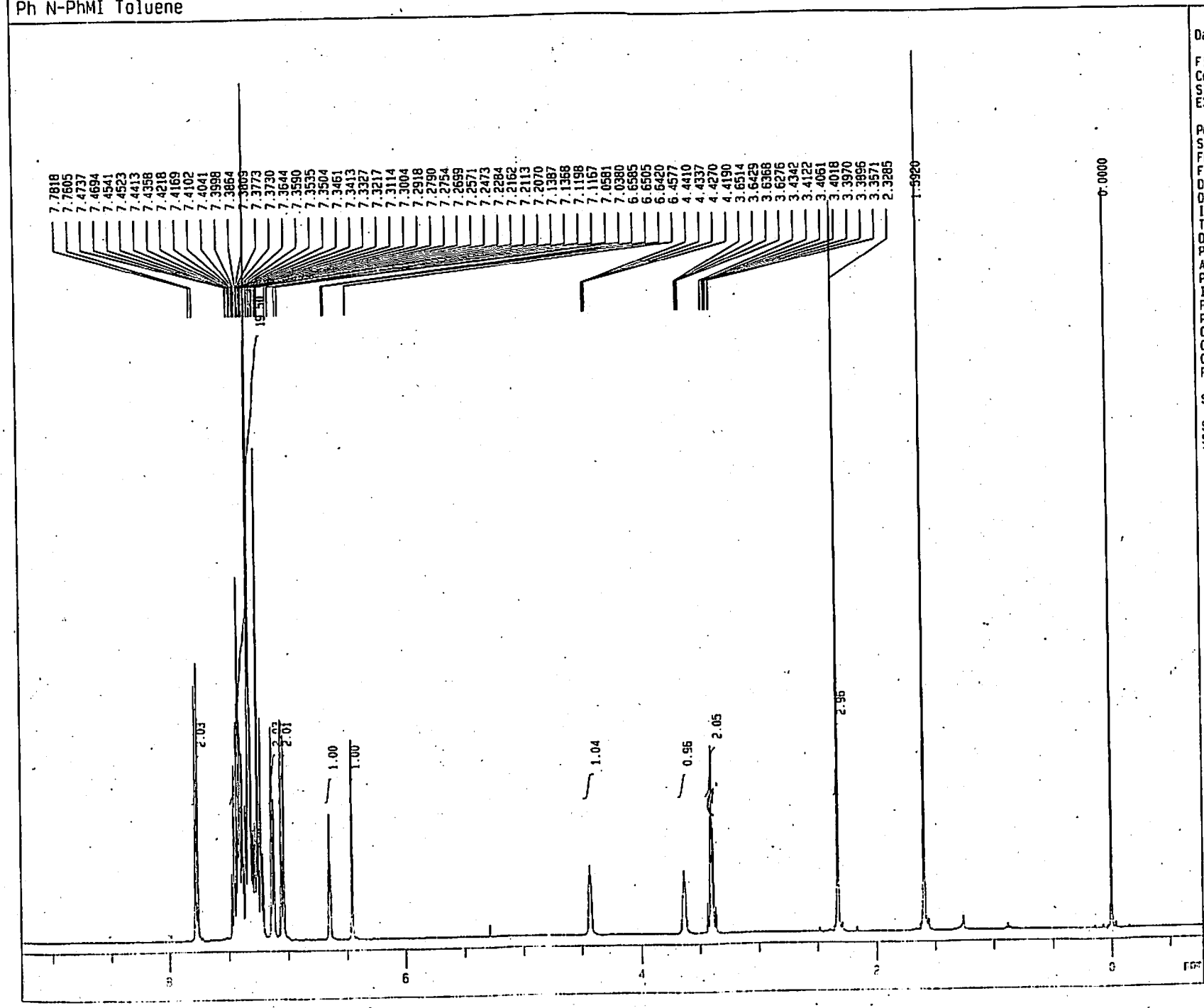
^{1}H NMR (**4c**)


Bn TCNE
Date : Wed Jan 28 18:07:52 2004
FileName : auto_1H.nmdata
Comment : Bn TCNE
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 10000.0 Hz
FILTR : 5000 Hz
DELAY : 40.0 usec
DEADT : 56.9 usec
INTVL : 100.0 usec
TIMES : 8 times
DUMMY : 0 times
PD : 3.7232 sec
ACQTM : 3276.7998 msec
PREDL : 10.00000 msec
INIMT : 0.5000 msec
RESOL : 0.31 Hz
PW1 : 6.20 usec
OBNUC : 1H
OBFRQ : 500.00 MHz
OBSET : 162160.00 Hz
RGAIN : 19
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 12 Hz
TEMP : 22.9 C
ppm

^{13}C NMR (**4c**)


bcm
132.580
130.812
130.450
129.915
129.660
129.413
129.372
128.887
128.731
128.278
126.551
125.095
77.255
76.745
61.577
56.255
48.893
47.297
21.790
200
150
100
50
0
ppm
Date : Wed Jan 28 18:15:47 2004
FileName : auto_13C.nmdata
Comment : bcm
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 33898.3 Hz
FILTR : 16950 Hz
DELAY : 11.8 usec
DEADT : 10.0 usec
INTVL : 29.5 usec
TIMES : 128 times
DUMMY : 1 times
PD : 2.0333 sec
ACQTM : 966.6560 msec
PREDL : 10.00000 msec
ININT : 10.0000 msec
RESOL : 1.03 Hz
PW1 : 5.50 usec
OBNUC : 13C
OBFRQ : 125.65 MHz
OBSET : 127958.00 Hz
RGAIN : 32
IRNUC : 1H
IRFRQ : 500.00 MHz
IRSET : 162160.00 Hz
IRRPW : 48.0 usec
IRRNS : 0
SCANS : 128 times
SLVNT : CDCL3
SPINNING : 11 Hz
TEMP : 23.5 C

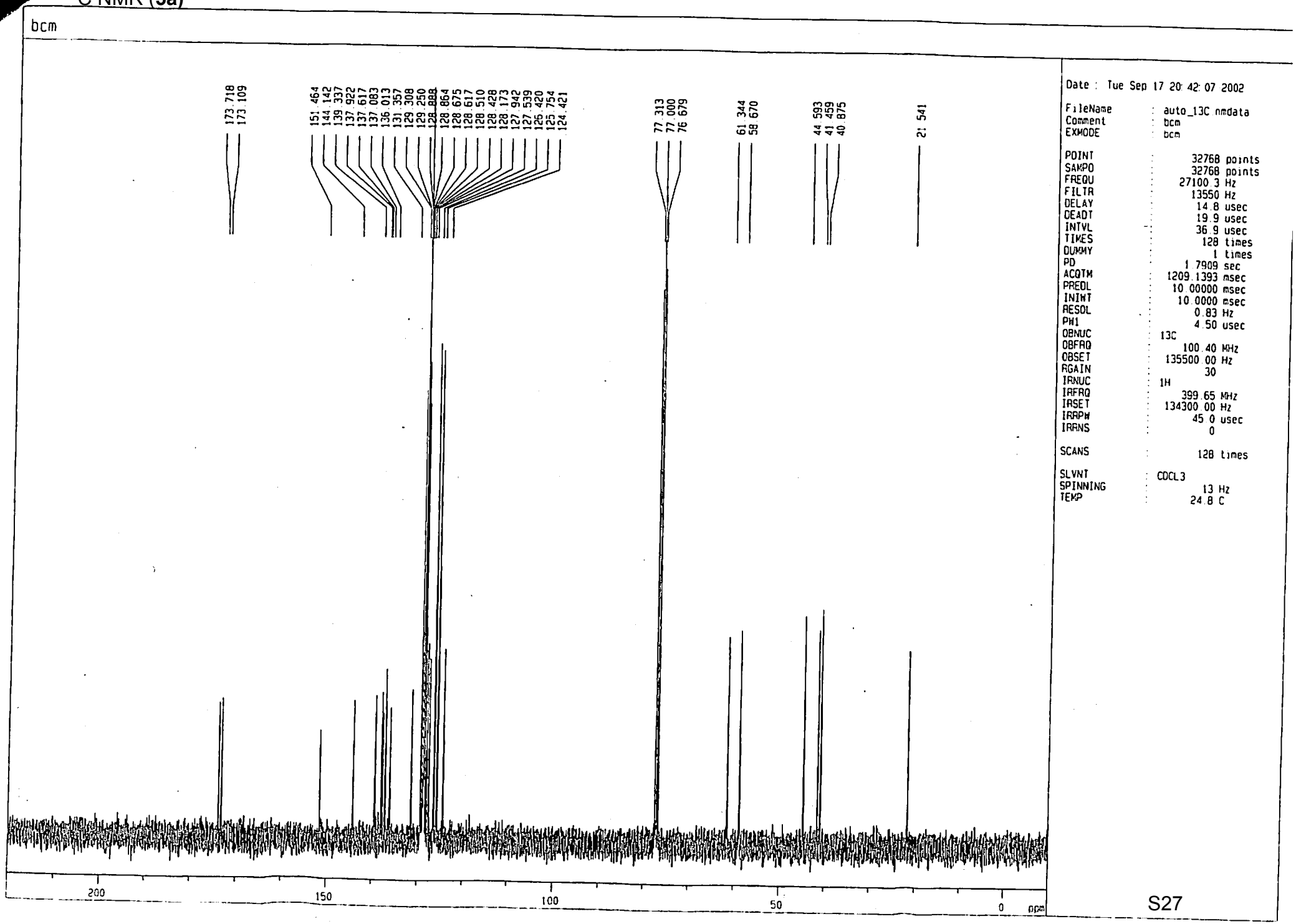
^{1}H NMR (**5a**)



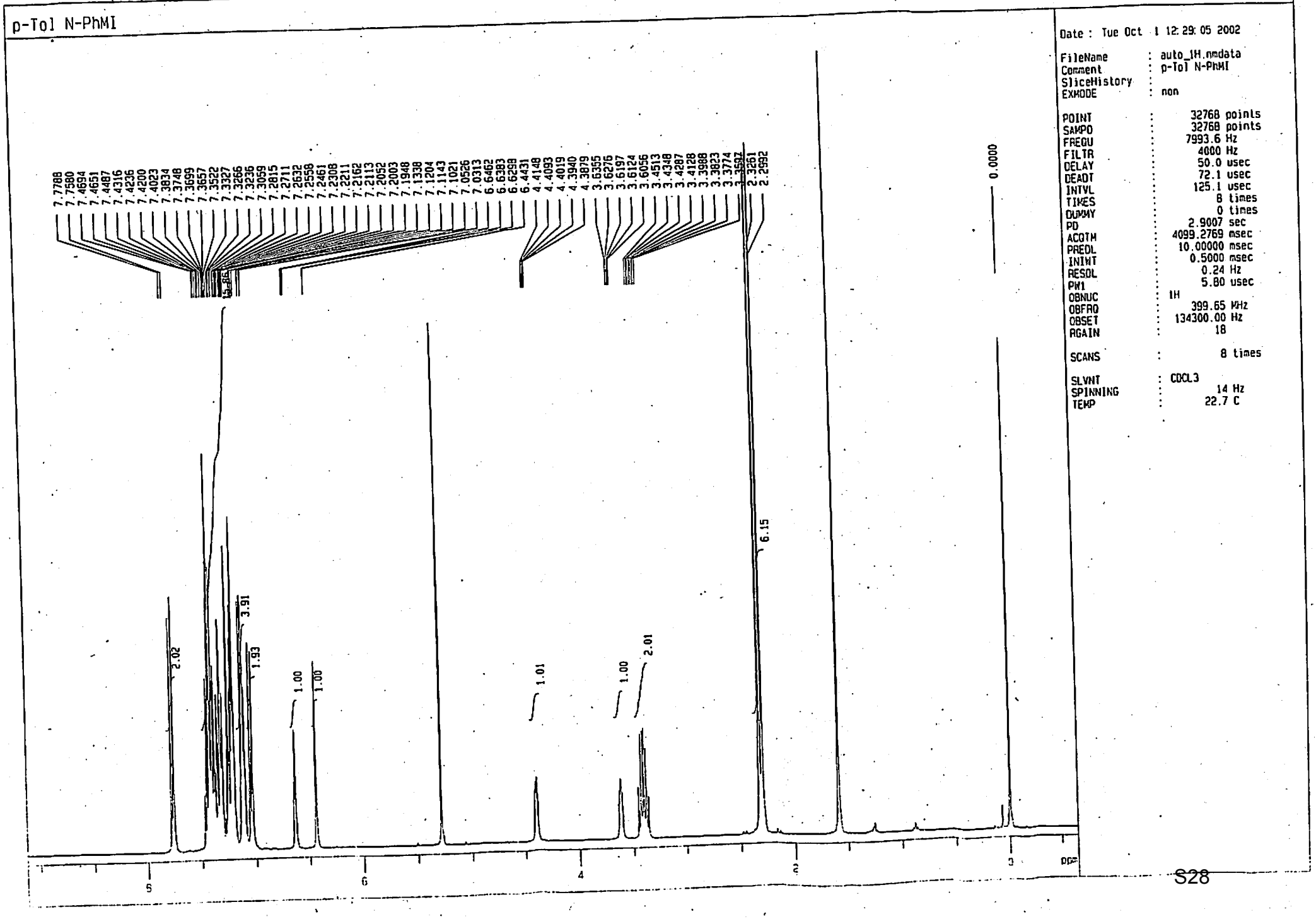
Date : Tue Oct 1 12:20:57 2002

FileName	LoadingFID.nmdata
Comment	Ph N-PhMI Toluene
SliceHistory	
EXMODE	non
POINT	32768 points
SAMPO	32768 points
FREQU	7993.6 Hz
FILTR	4000 Hz
DELAY	50.0 usec
DEADT	72.1 usec
INTVL	125.1 usec
TIMES	8 times
DUMMY	1 times
PD	2.9007 sec
ACQTM	4099.2769 msec
PREDL	0.01000 msec
INIWT	1000.0000 msec
RESOL	0.24 Hz
PW1	5.80 usec
OBNUC	1H
OBFRQ	399.65 MHz
OBSET	134300.00 Hz
RGAIN	18
SCANS	8 times
SLVNT	CDCL3
SPINNING	13 Hz
TEMP	22.9 C

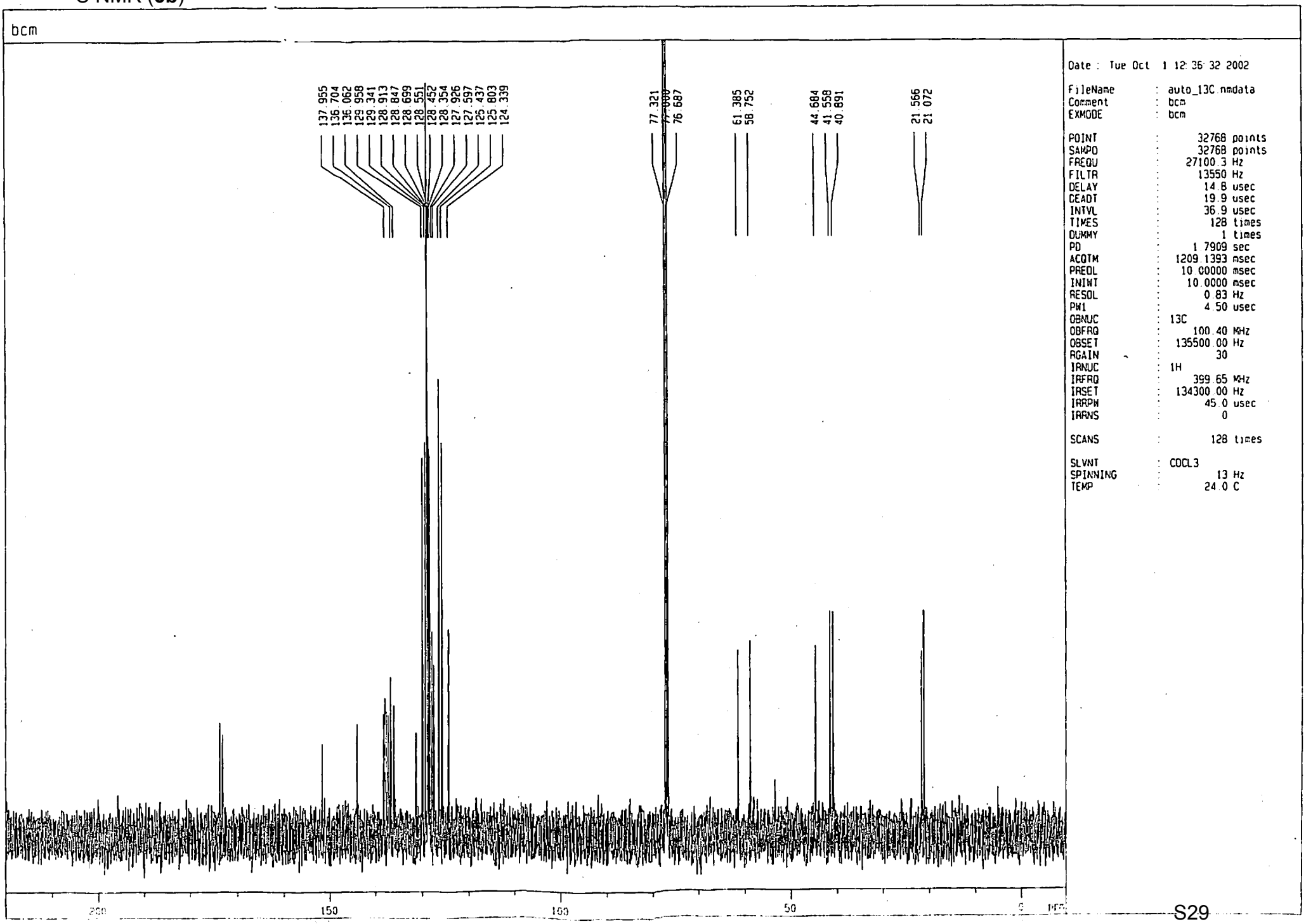
^{13}C NMR (**5a**)


bcm
173.718
173.109
151.464
144.142
139.337
137.922
137.617
137.083
136.013
131.357
129.308
129.250
128.888
128.864
128.675
128.617
128.510
128.428
128.173
127.942
127.539
126.420
125.754
124.421
77.313
77.000
76.679
61.344
58.670
44.593
41.459
40.875
21.541
200
150
100
50
0
ppm
Date : Tue Sep 17 20:42:07 2002
FileName : auto_13C.nmdata
Comment : bcm
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 27100.3 Hz
FILTR : 13550 Hz
DELAY : 14.8 usec
DEADT : 19.9 usec
INTVL : 36.9 usec
TIMES : 128 times
DUMMY : 1 times
PD : 1.7909 sec
ACQTM : 1209.1393 msec
PREDL : 10.00000 msec
INIWT : 10.0000 msec
RESOL : 0.83 Hz
PW1 : 4.50 usec
OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz
RGAIN : 30
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134300.00 Hz
IRPW : 45.0 usec
IRNS : 0
SCANS : 128 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 24.8 C

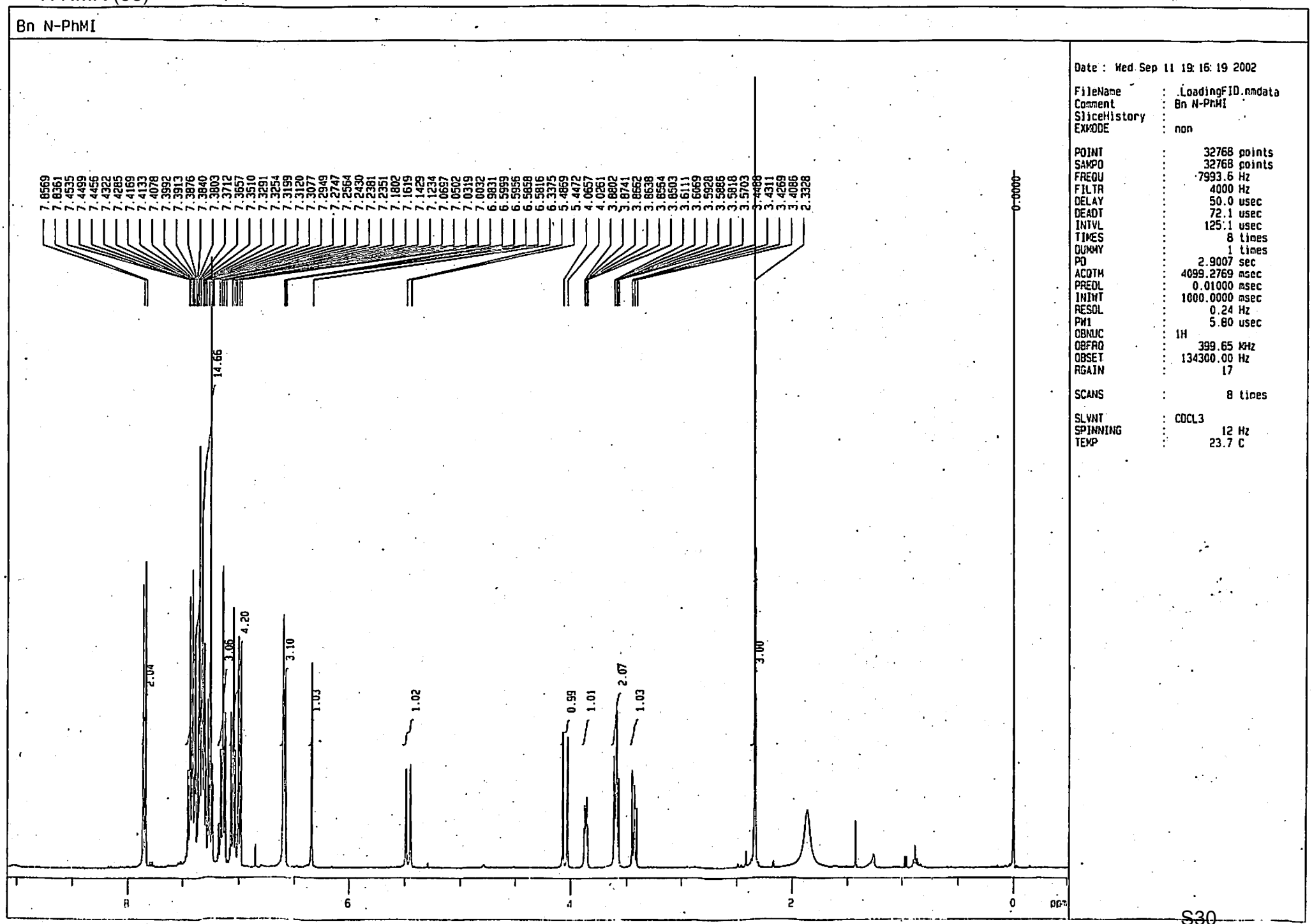
^{1}H NMR (**5b**)


p-Tol N-PhMI
Date : Tue Oct 1 12:29:05 2002
FileName : auto_1H.nmdata
Comment : p-Tol N-PhMI
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7993.6 Hz
FILTR : 4000 Hz
DELAY : 50.0 usec
DEADT : 72.1 usec
INTVL : 125.1 usec
TIMES : 8 times
DUMMY : 0 times
PD : 2.9007 sec
ACQTM : 4099.2769 msec
PREDL : 10.00000 msec
INIWT : 0.5000 msec
RESOL : 0.24 Hz
PW1 : 5.80 usec
OBNUC : 1H
OBFRQ : 399.65 MHz
OBSET : 134300.00 Hz
AGAIN : 18
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 14 Hz
TEMP : 22.7 C
7.7788
7.7580
7.4694
7.4651
7.4487
7.4316
7.4236
7.4200
7.4023
7.3834
7.3748
7.3699
7.3657
7.3522
7.3327
7.3266
7.3236
7.3059
7.2815
7.2711
7.2632
7.2558
7.2461
7.2308
7.2211
7.2162
7.2113
7.2052
7.2003
7.1948
7.1338
7.1204
7.1143
7.1021
7.0526
7.0313
6.6462
6.6383
6.6298
6.4431
4.4148
4.4093
4.4019
4.3940
4.3879
3.6355
3.6276
3.6197
3.6124
3.6056
3.4513
3.4348
3.4287
3.4128
3.3988
3.3823
3.3774
3.3592
3.3261
2.2992
0.0000
2.02
3.91
1.93
1.00
1.00
1.01
1.00
2.01
6.15
ppm

^{13}C NMR (**5b**)

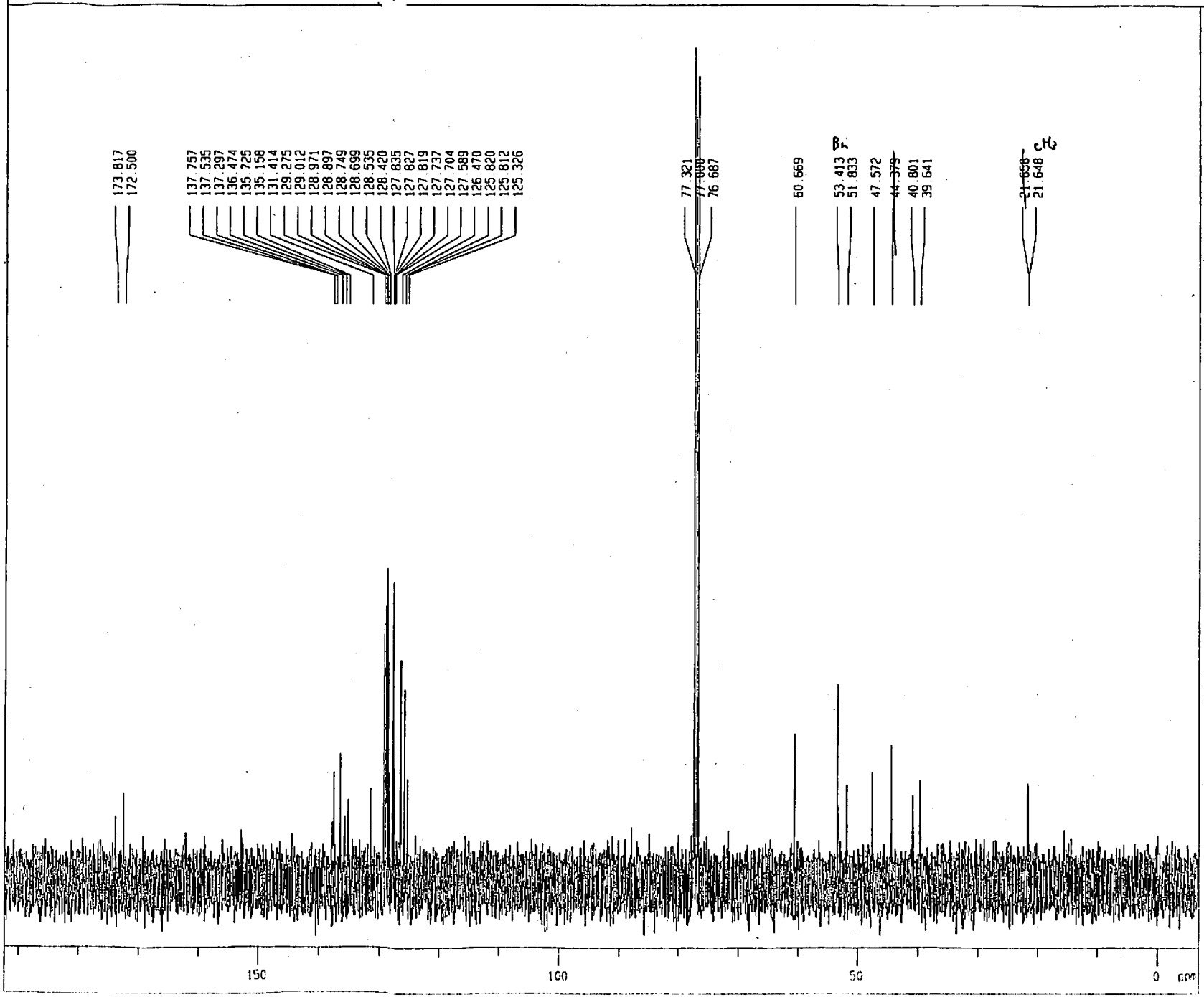

bcm
137.955
136.704
136.062
129.958
129.341
128.913
128.847
128.699
128.551
128.452
128.354
127.926
127.597
125.437
125.803
124.339
77.321
77.000
76.687
61.385
58.752
44.684
41.558
40.891
21.566
21.072
Date : Tue Oct 1 12:36:32 2002
FileName : auto_13C.nmdata
Comment : bcm
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 27100.3 Hz
FILTR : 13550 Hz
DELAY : 14.8 usec
DEADT : 19.9 usec
INTVL : 36.9 usec
TIMES : 128 times
DUMMY : 1 times
PD : 1.7909 sec
ACQTM : 1209.1393 msec
PREDL : 10.00000 msec
ININT : 10.0000 msec
RESOL : 0.83 Hz
PW1 : 4.50 usec
OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz
RGAIN : 30
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134300.00 Hz
IRPW : 45.0 usec
IRNS : 0
SCANS : 128 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 24.0 C
200
150
100
50
0
PPM

^{1}H NMR (**5c**)


Bn N-PhMI
Date : Wed Sep 11 19:16:19 2002
FileName : LoadingFID.nmdata
Comment : Bn N-PhMI
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7993.6 Hz
FILTR : 4000 Hz
DELAY : 50.0 usec
DEADT : 72.1 usec
INTVL : 125.1 usec
TIMES : 8 times
DUMMY : 1 times
PD : 2.9007 sec
ACQTM : 4099.2769 msec
PREDL : 0.01000 msec
ININT : 1000.0000 msec
RESOL : 0.24 Hz
PW1 : 5.80 usec
OBNUC : 1H
OBFRQ : 399.65 MHz
OBSET : 134300.00 Hz
RGAIN : 17
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 12 Hz
TEMP : 23.7 C
14.66
2.04
3.06
4.20
3.10
1.03
1.02
0.99
1.01
2.07
1.03
3.00
0.0000
8
6
4
2
0
ppm

^{13}C NMR (**5c**)

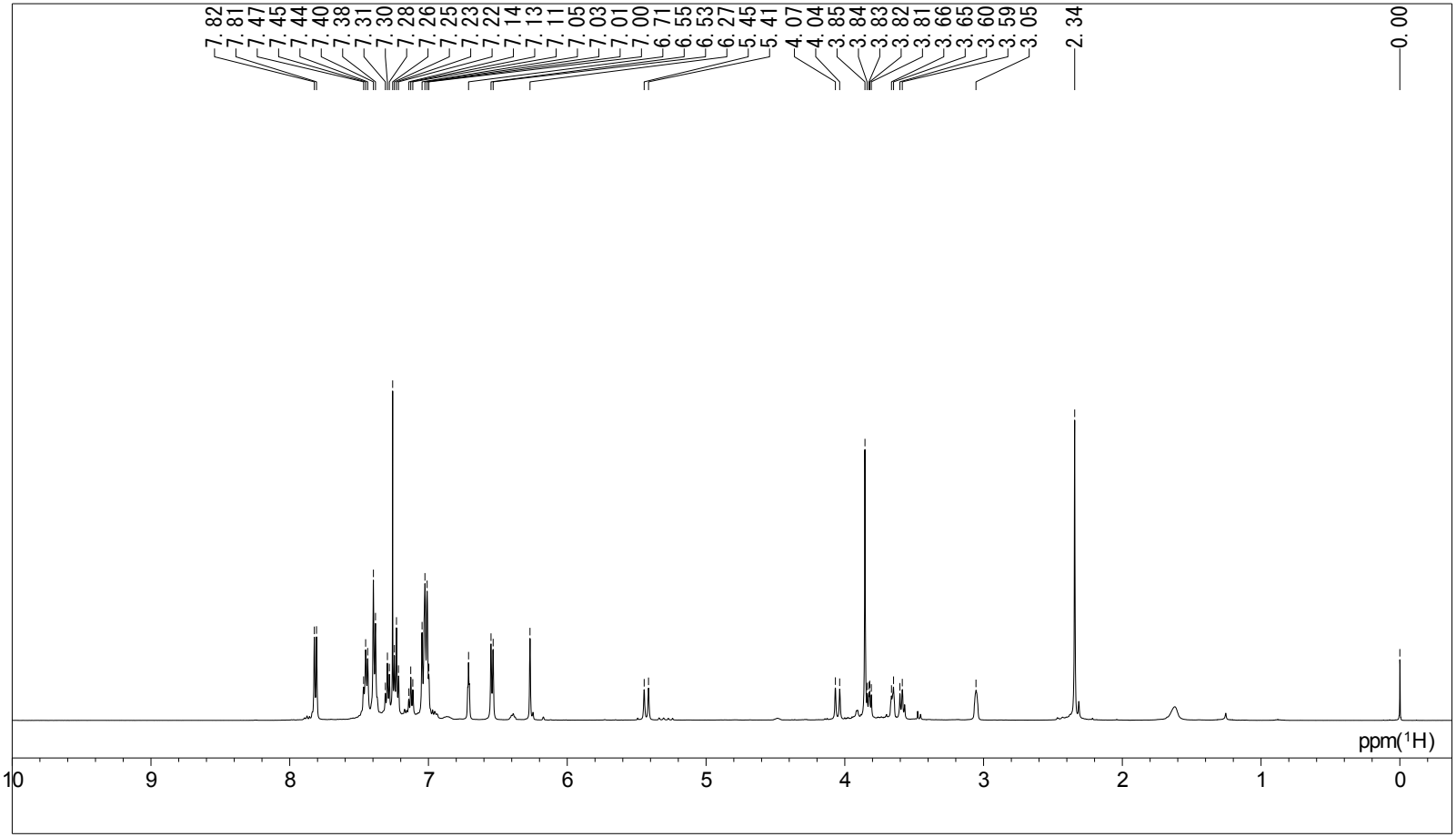
bcm



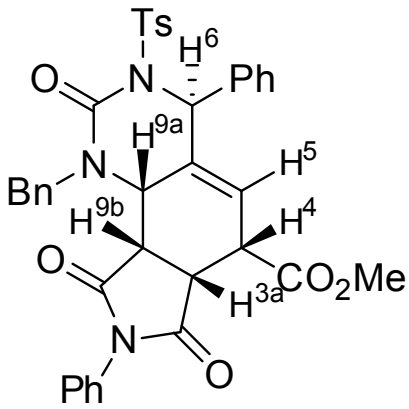
Date : Wed Aug 28 10:42:11 2002

FileName	:	auto_13C.nmdata
Comment	:	bcm
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	27100.3 Hz
FILTR	:	13550 Hz
DELAY	:	14.8 usec
DEADT	:	19.9 usec
INTVL	:	36.9 usec
TIMES	:	128 times
DUMMY	:	1 times
PD	:	1.7909 sec
ACQTM	:	1209.1393 msec
PREDL	:	10.00000 msec
INIWT	:	10.0000 msec
RESOL	:	0.83 Hz
PW1	:	4.50 usec
OBNUC	:	13C
OBFRQ	:	100.40 MHz
OBSET	:	135500.00 Hz
RGAIN	:	31
IRNUC	:	1H
IRFRQ	:	399.65 MHz
IRSET	:	134300.00 Hz
IRPPW	:	45.0 usec
IRRNS	:	0
SCANS	:	128 times
SLVNT	:	CDCL3
SPINNING	:	10 Hz
TEMP	:	24.8 C

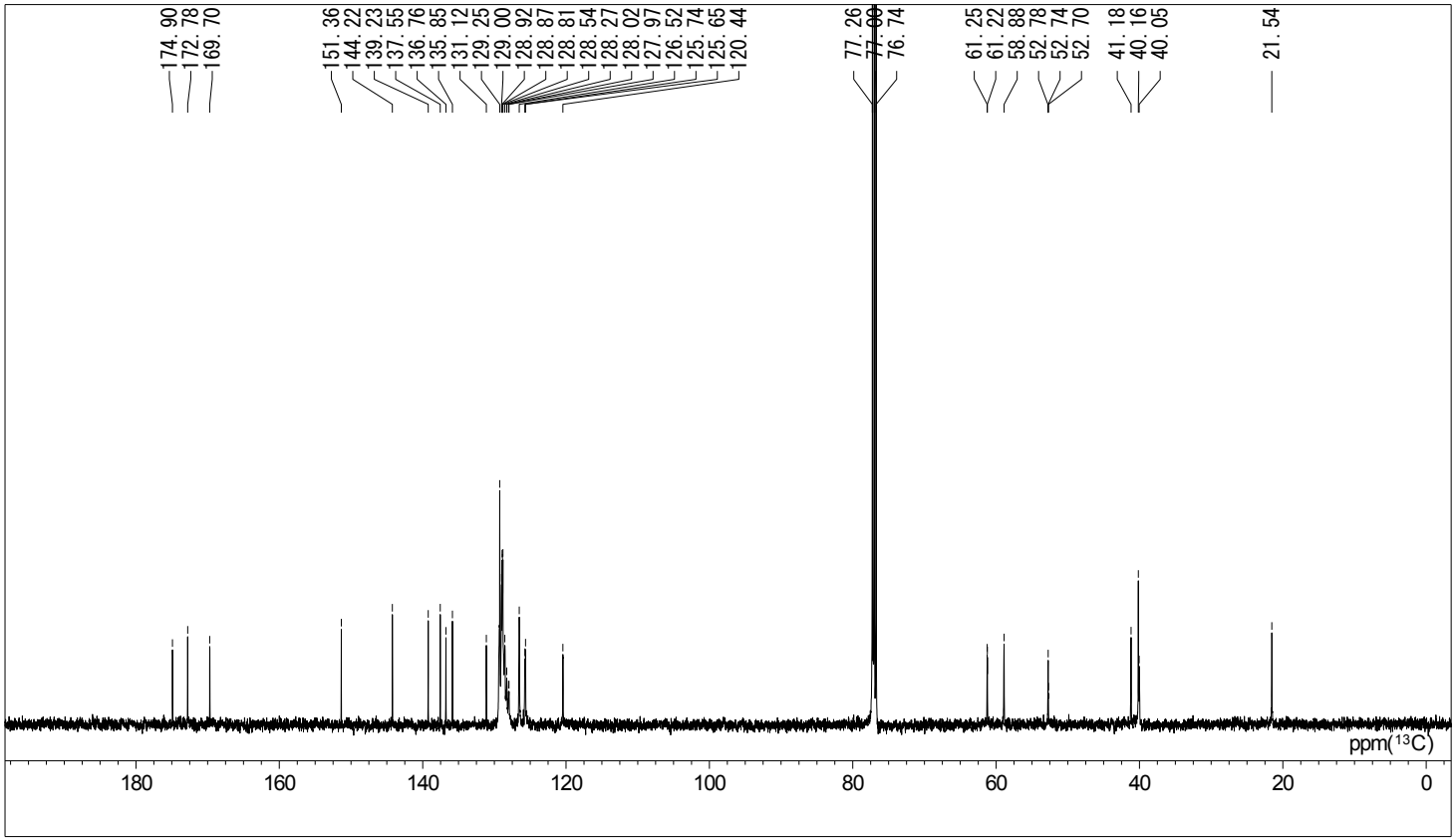
^{1}H NMR (**5d**)



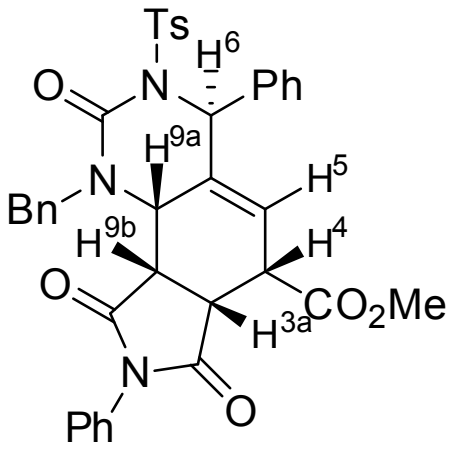
OBFRQ 500.16 MHz
SLVNT CHLOROFORM-D
OBFIN -88.4525 Hz



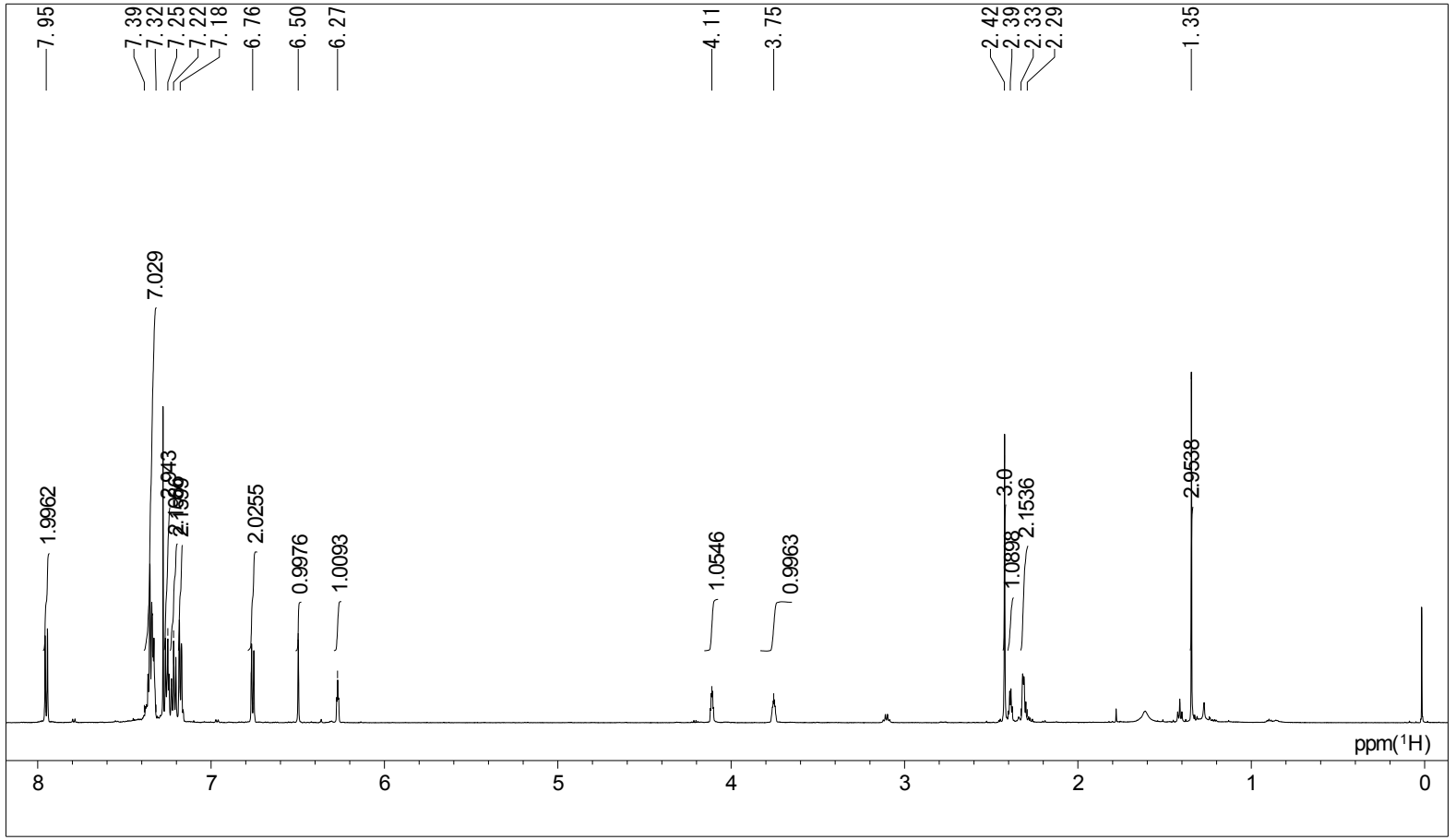
^{13}C NMR (**5d**)



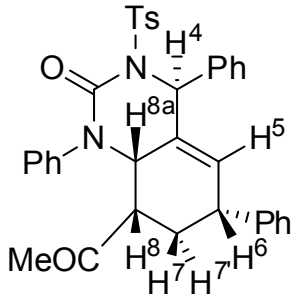
OBFRQ 125.77 MHz
SLVNT CHLOROFORM-D
OBFIN 301.0403 Hz



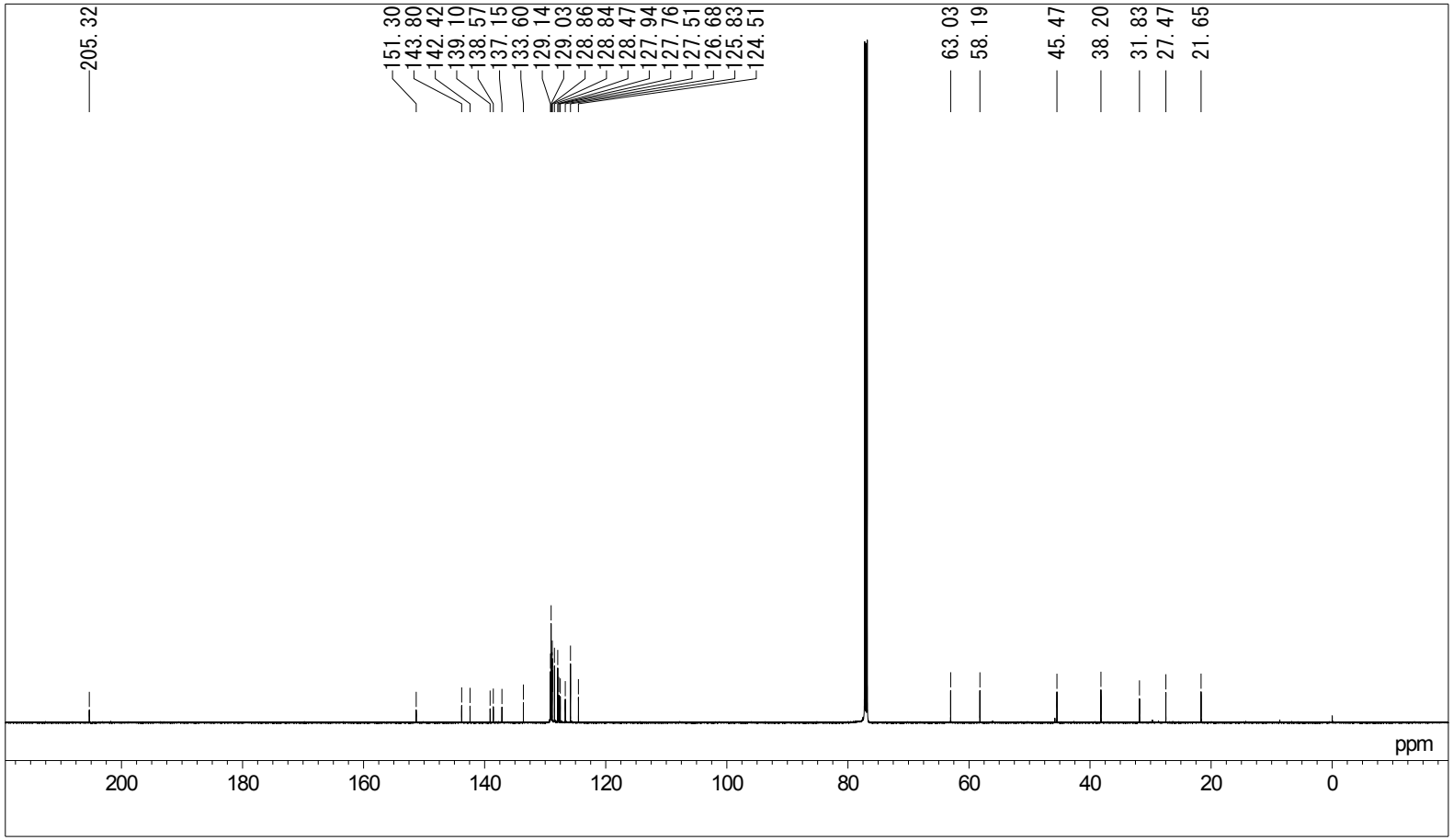
^{1}H NMR (**6a**)



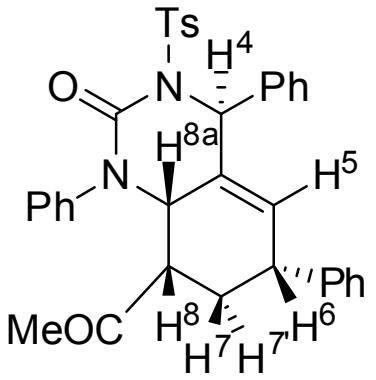
OBFRQ	600.13 MHz
SLVNT	$CDCL_3$
OBFIN	3701.166 Hz



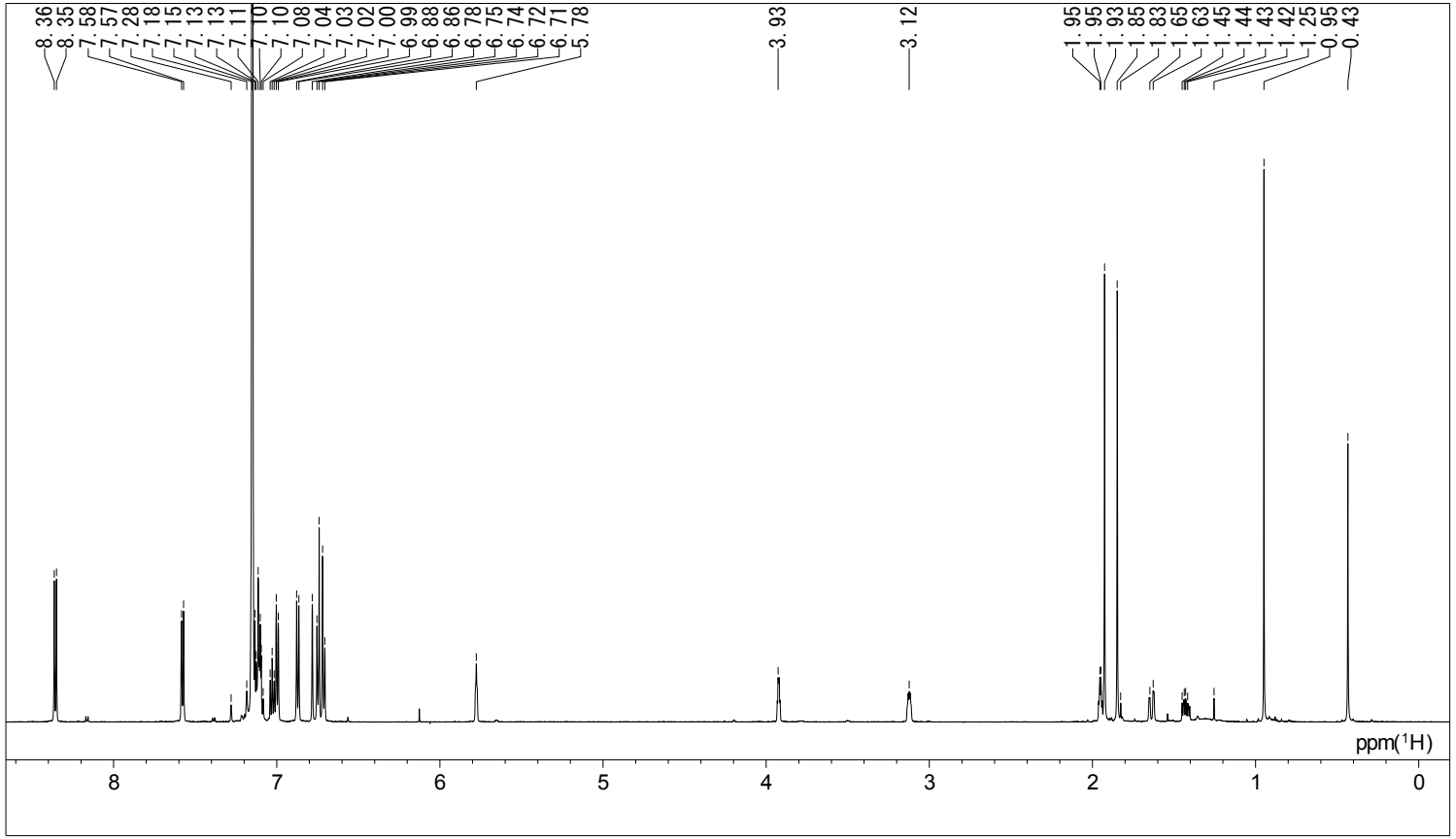
^{13}C NMR (**6a**)



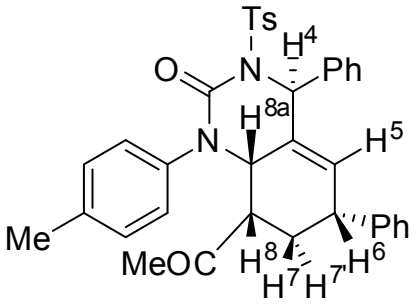
OBFRQ	150.9 MHz
SLVNT	$CDCL_3$
OBFIN	15097.29 Hz



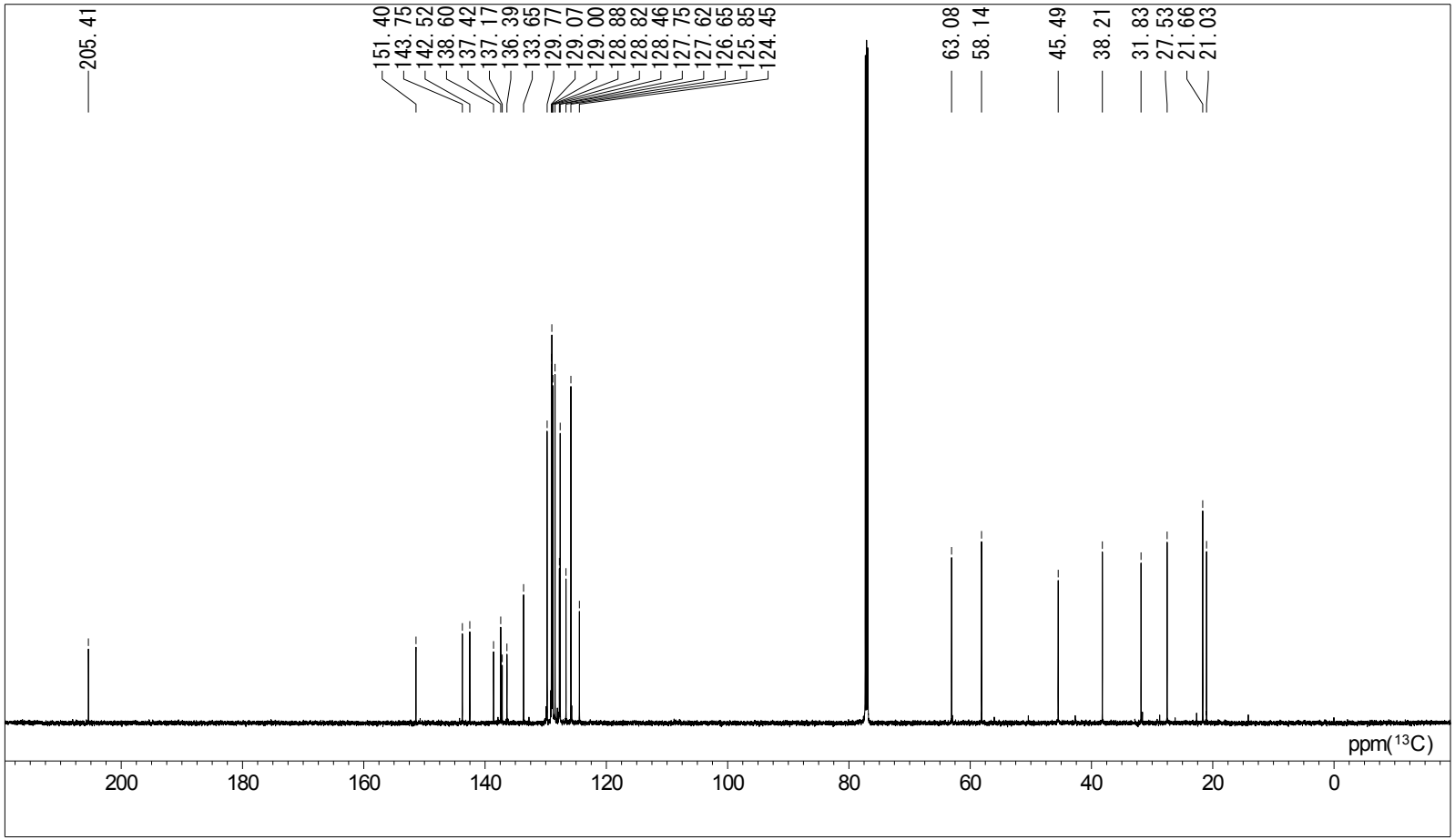
^{1}H NMR (**6b**)



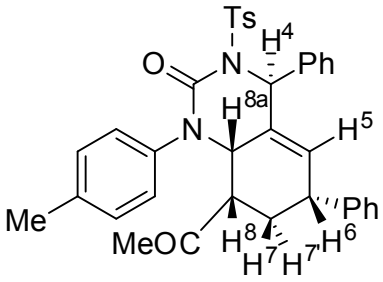
OBFRQ 600.13 MHz
SLVNT $CDCL_3$
OBFIN 3701.166 Hz



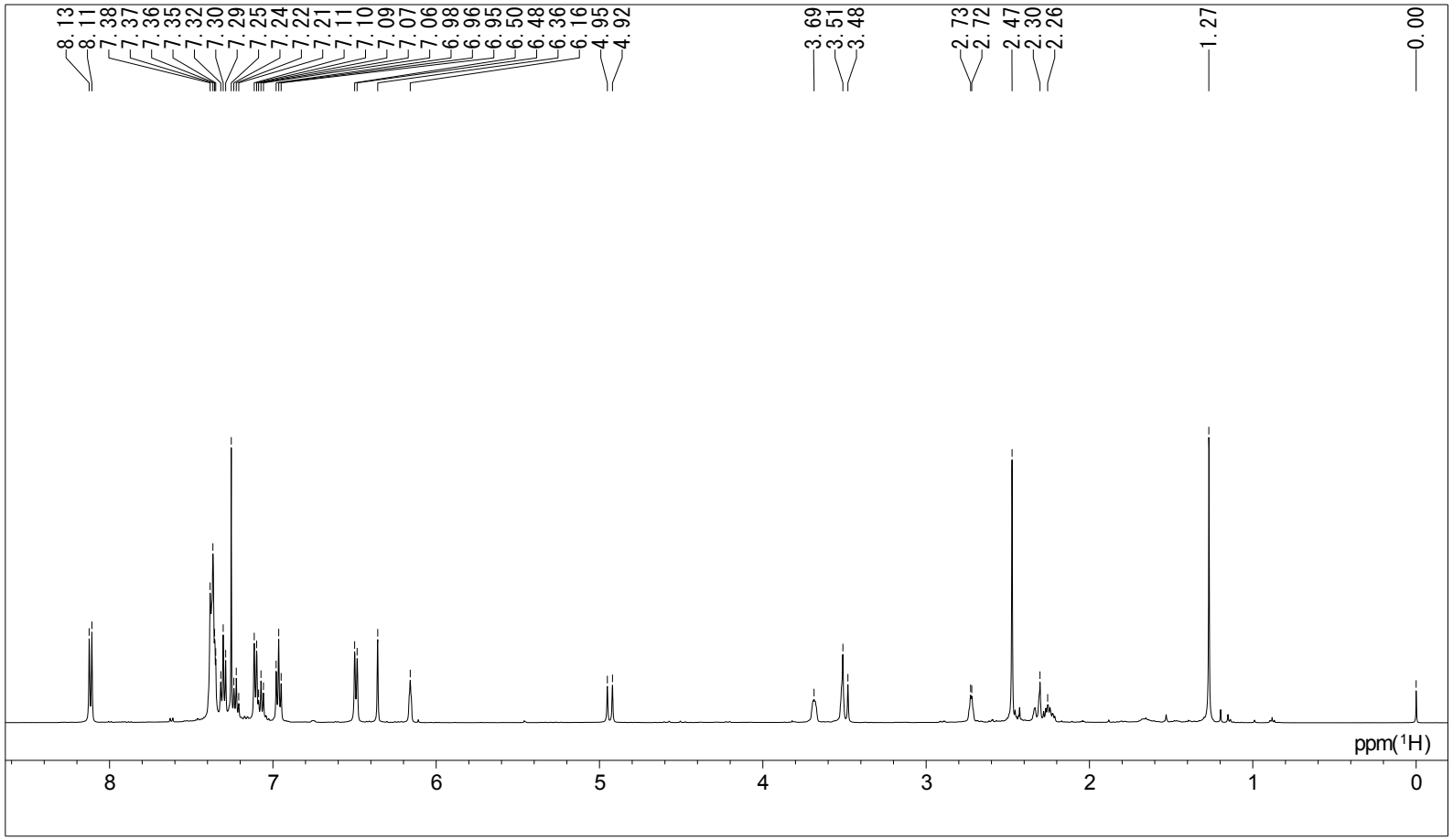
^{13}C NMR (**6b**)



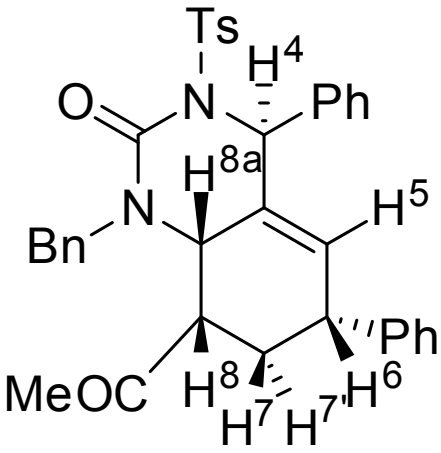
OBFRQ 150.9 MHz
SLVNT $CDCl_3$
OBFIN 15097.29 Hz



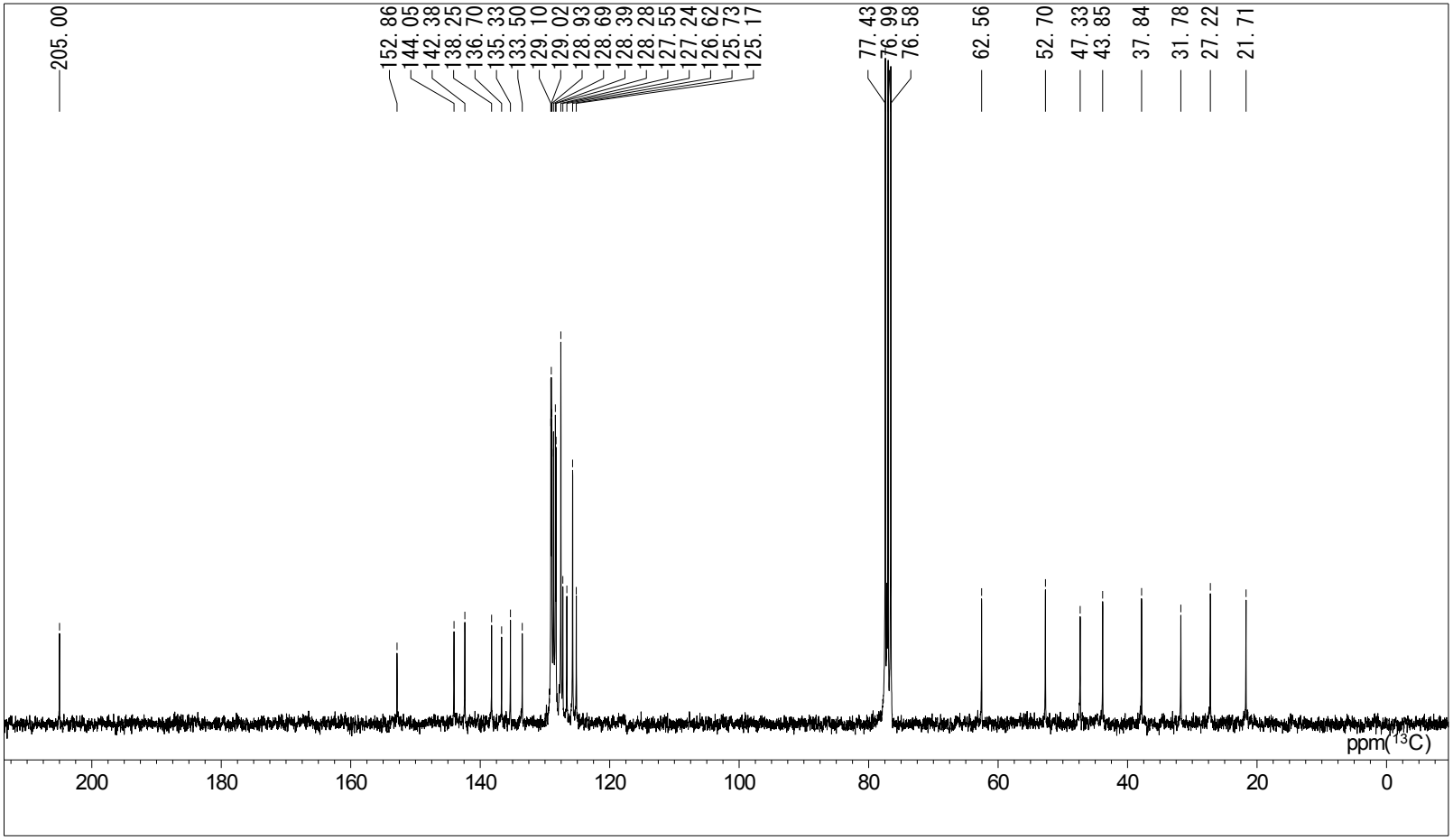
^{1}H NMR (**6c**)



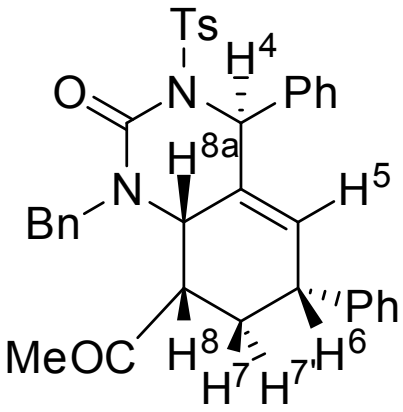
OBFRQ 500.16 MHz
SLVNT CHLOROFORM-D
OBFIN -88.4525 Hz



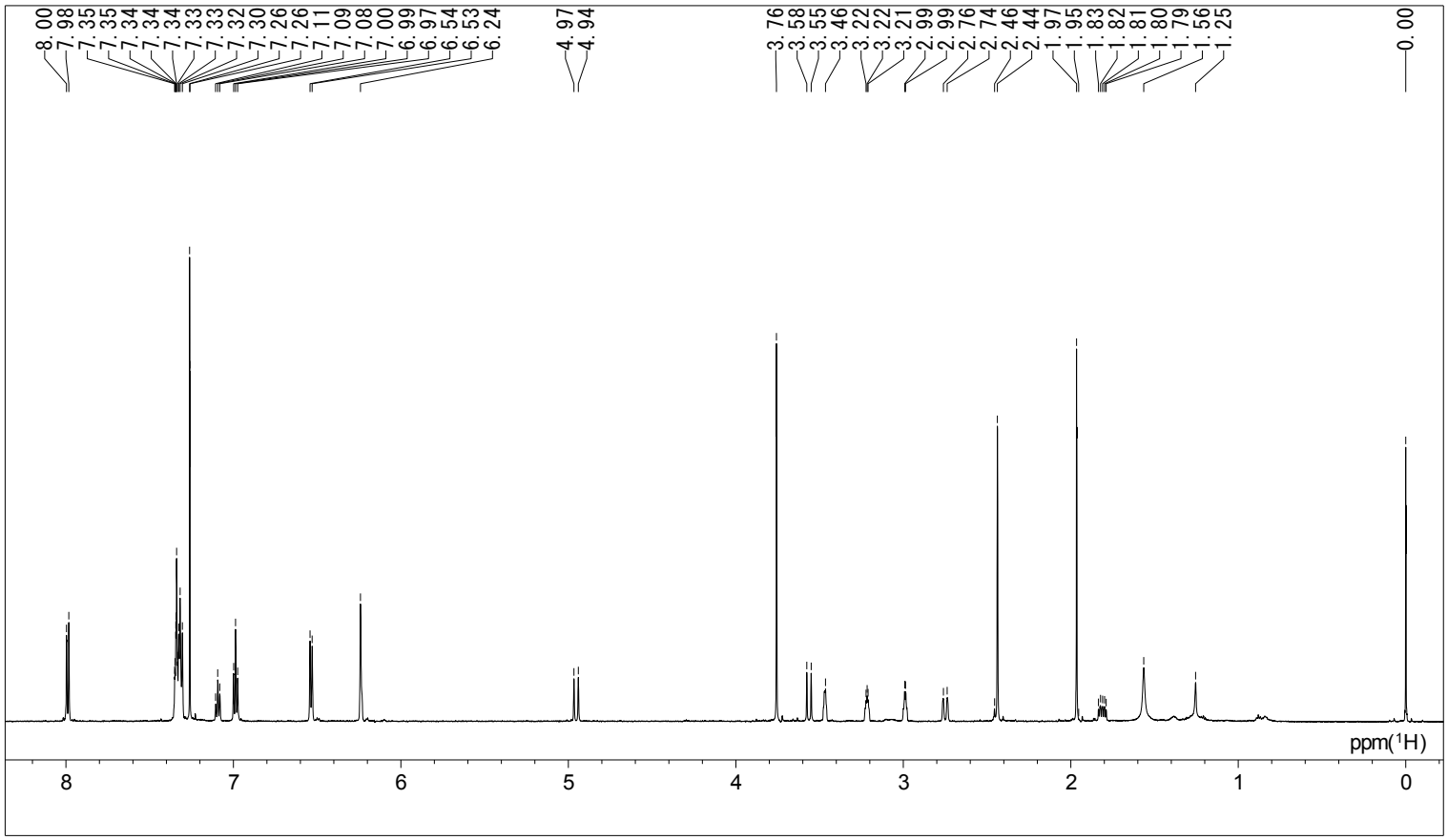
^{13}C NMR (**6c**)



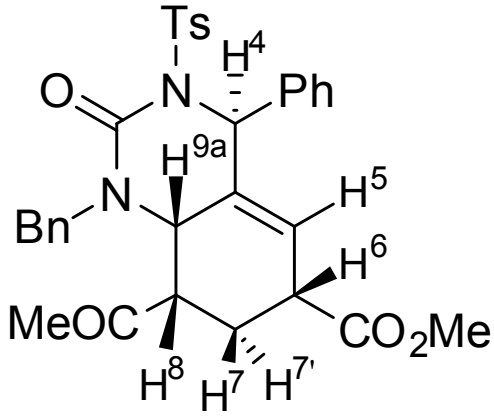
OBFRQ 75.57 MHz
SLVNT CHLOROFORM-D
OBFIN 234.5651 Hz



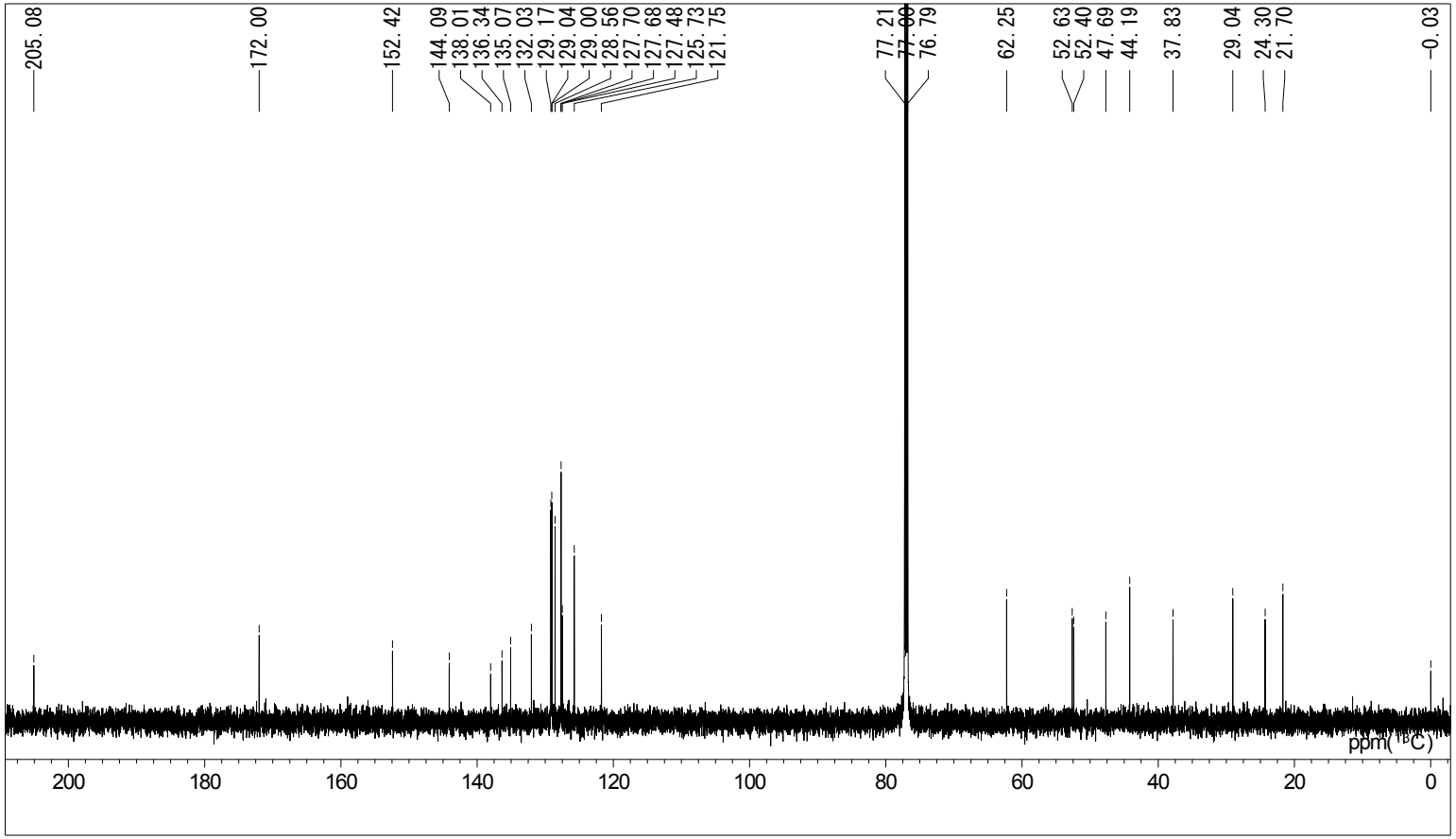
^{1}H NMR (*endo*-**6d**)



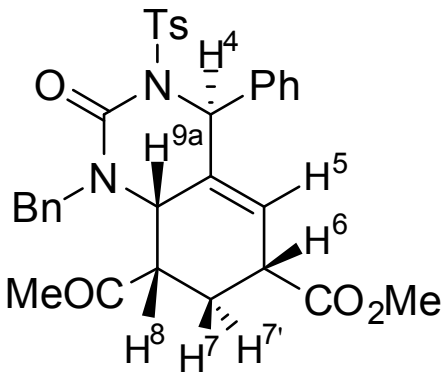
OBFRQ 600.13 MHz
SLVNT $CDCL_3$
OBFIN 3701.166 Hz



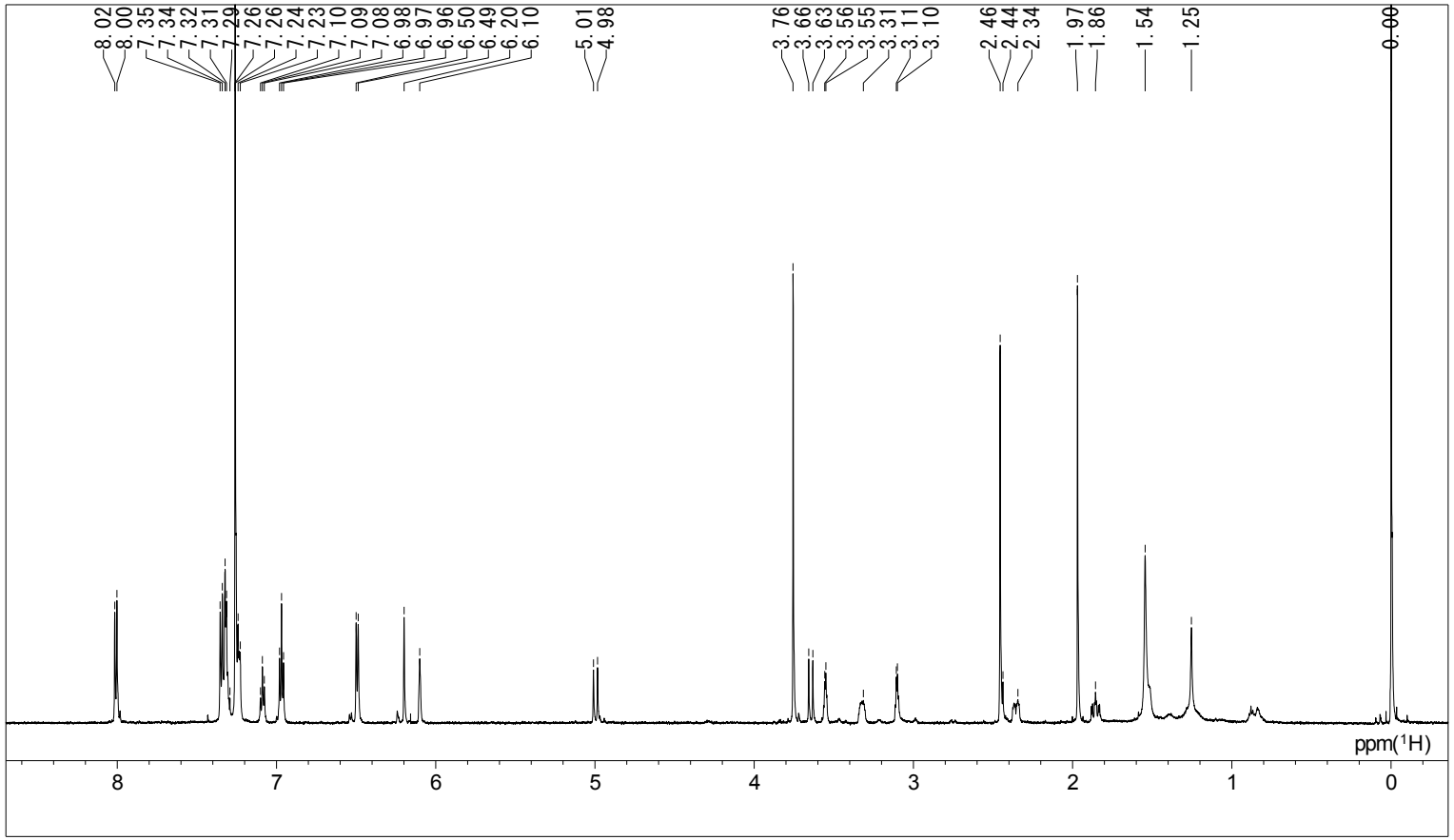
^{13}C NMR (*endo*-**6d**)



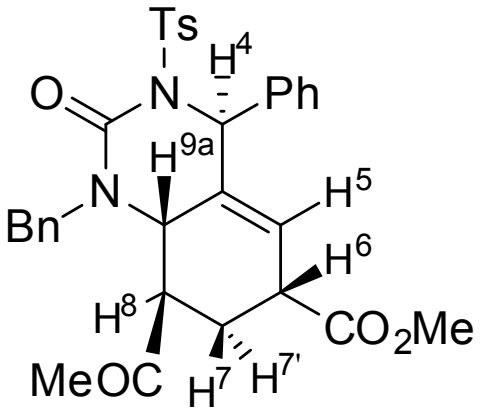
OBFRQ 150.9 MHz
SLVNT $CDCL_3$
OBFIN 15097.29 Hz



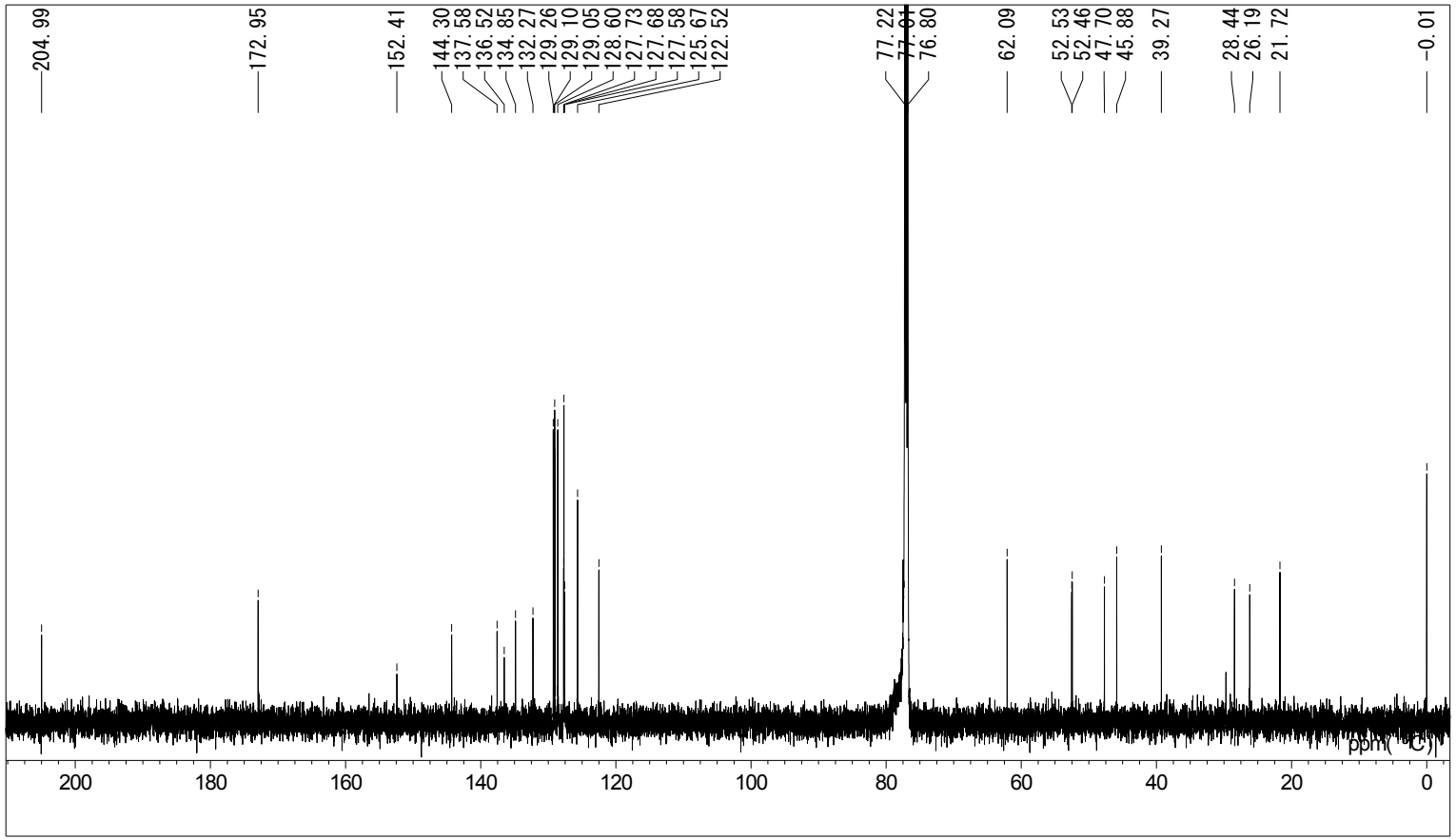
^{1}H NMR (*exo*-**6d**)



OBFRQ 600.13 MHz
SLVNT $CDCL_3$
OBFIN 3701.166 Hz



^{13}C NMR (*exo*-**6d**)



OBFRQ 150.9 MHz
SLVNT $CDCl_3$
OBFIN 15097.29 Hz