Stereoselective Synthesis of the Rocaglamide Skeleton via a Silyl Vinylketene Formation / [4+1] Annulation Sequence

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Supporting Information

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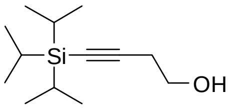
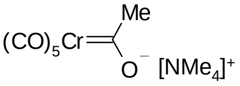
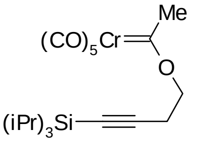
General Procedures. All ^{1}H and ^{13}C NMR were recorded at 300 MHz and 75.5 MHz, respectively, on a GE/Bruker QE 300 FT-NMR spectrometer. Chemical shifts are reported in ppm relative to $(CH_3)_4Si$ (0 ppm, ^{1}H) or $CHCl_3$ (77.0 ppm, ^{13}C). IR spectra were obtained on a Nicolet 5DXB FT-IR spectrometer. High resolution mass spectral analyses were performed at the Mass Spectroscopy Center at Indiana University, Bloomington, IN. Tetrahydrofuran (THF) and diethyl ether (Et_2O) were distilled from the sodium benzophenone ketyl radical prior to use. Benzene and dichloromethane (CH_2Cl_2) were distilled from calcium hydride and stored over 4Å molecular sieves prior to use. Chloroform ($CHCl_3$) and deuterochloroform ($CDCl_3$) were passed through a column of grade I basic alumina prior to use. Hexanes, ethyl acetate (EtOAc), and methanol (MeOH) were used as obtained from commercial sources.



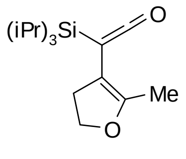
Alkynol 8. To a solution of 3-butyn-1-ol (2.27 mL, 30.0 mmol) and 3,4-dihydro-2H-pyran (6.31 g, 75.0 mmol) in CH_2Cl_2 (100 mL) was added a catalytic amount of *p*-toluenesulfonic acid (TsOH) (30 mg, 0.15 mmol). The resultant reaction mixture was allowed to stir for 1.5 h, affording a pale purple solution. The solution was then diluted with Et_2O and washed with a 1:1:2 mixture of sat. aq. NaCl / sat. aq. $NaHCO_3$ / H_2O. The organic layer was dried over $MgSO_4$, filtered, and solvents were removed under reduced pressure. The residue was chromatographed on a short plug of silica gel (9:1 hexane:EtOAc eluent) to provide the purified tetrahydropyran (THP) ether as a yellow oil. A solution of the THP ether in THF (40 mL) was cooled to -78 °C under argon atmosphere, and *n*-BuLi (22 mL of a 1.5M solution, 33 mmol) was added dropwise via syringe. The resultant pale orange solution was allowed to stir for 1 h at -78 °C, and triisopropylsilyl chloride (7.1 mL, 33 mmol) was added dropwise via syringe. After slowly warming to room temperature overnight, the solution was diluted with Et_2O and washed successively with sat. aq. $NaHCO_3$ and sat. aq. NaCl. The organic layer was dried over $MgSO_4$, filtered, and concentrated under reduced pressure. Without further purification, this material was dissolved in MeOH (50 mL) and treated with TsOH (2.3 g, 16 mmol) for 1.5 h. The MeOH was removed under reduced pressure, and the residue was partitioned between Et_2O and sat. aq. $NaHCO_3$. The organic layer was dried over $MgSO_4$, filtered, and concentrated under reduced pressure. Distillation of this crude product under reduced pressure afforded alkynol **8** (5.20 g, 76% yield over the three steps) as a clear, colorless oil. IR: 3345, 2174 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 3.68 (t, 2H), 2.51 (t, 2H), 1.79 (s, 1H), 1.05 (m, 21H). ^{13}C NMR ($CDCl_3$): δ 104.9, 83.0, 61.2, 24.3, 18.6, 11.2. HRMS (ESI): m/z calcd for $C_{13}H_{26}OSi$ $[M]^+$: 226.1753; found: 226.1754.



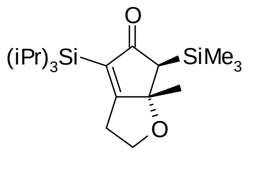
Tetramethylammonium chromium acylate complex 9.[1] A suspension of $Cr(CO)_6$ (3.00 g, 13.6 mmol) in THF (30 mL) was cooled to -78 °C under argon atmosphere, and MeLi (15 mL of a 1.0 M solution, 15 mmol) was added dropwise via syringe. The reaction mixture was allowed to warm to room temperature over 3 h, and the solvent was removed under reduced pressure. The oily residue was dissolved in H_2O (30 mL), and an aqueous solution of tetramethylammonium bromide (2.10 g, 13.6 mmol) was added dropwise via pipette. After stirring for 15 min, the mixture was diluted with CH_2Cl_2, and the solids were collected by filtration. The solids were suspended in $CHCl_3$ and filtered over a plug of Celite. The filtrate was discarded, and the yellow-green solid was flushed through the Celite pad with acetone. Removal of the acetone under reduced pressure provided complex **9** (2.23 g, 53% yield) as a yellow solid. IR: 2031, 1887 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 3.45 (s, 12H), 2.28 (s, 3H).



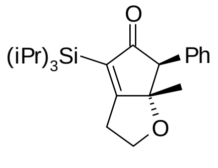
Alkoxy carbene complex 10. A suspension of chromium acylate complex **9** (1.81 g, 5.80 mmol) in CH_2Cl_2 (30 mL) was cooled to -78 °C under argon atmosphere, and acetyl bromide (430 µL, 5.8 mmol) was added dropwise via syringe. After allowing to warm to -40 °C over 1 h, a solution of alkynol **8** (1.31 g, 5.80 mmol) in CH_2Cl_2 (5 mL) was added via cannula. The resultant solution was warmed to room temperature and allowed to stir overnight. The solution was then washed successively with sat. aq. $NaHCO_3$ and sat. aq. NaCl. The organic layer was dried over $MgSO_4$, filtered, and concentrated to an orange oil. Purification via flash chromatography (SiO_2, 30:1 hexane:EtOAc eluent) provided **10** (1.39 g, 54% yield) as an orange oil. ^{1}H NMR ($CDCl_3$): δ 4.95 (br s, 2H), 2.95 (m, 5H), 1.04 (s, 21H). ^{13}C NMR ($CDCl_3$): δ 359.2, 223.2, 216.3, 102.6, 94.7, 83.9, 21.2, 18.5, 11.2. Due to the thermal instability of this complex, HRMS data could not be obtained.



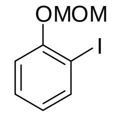
Silyl vinylketene 11. A solution of carbene complex **10** (200 mg, 0.45 mmol) in benzene (30 mL) was degassed by using three cycles of freeze-pump-thaw degassing. The solution was then heated to reflux for 18 h under argon atmosphere, affording a yellow-brown solution which was diluted with hexane and filtered through a plug of Celite, washing with additional hexane. Solvents were then removed under reduced pressure to provide silyl vinylketene **11** (125 mg) as a yellow oil. Further purification of **11** was precluded by instability on silica gel; however, ^{1}H NMR analysis of this material indicated essentially complete conversion and a high level of purity. IR: 2082 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 4.23 (t, *J* = 9.6 Hz, 2H), 2.60 (m, 2H), 1.78 (s, 3H), 1.09 (m, 21H). HRMS (ESI): m/z calcd for $C_{16}H_{28}O_2Si$ $[M]^+$: 280.1859; found 280.1845.



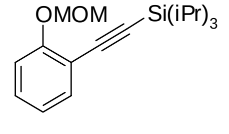
Cyclopentenone 12. To a solution of silyl vinylketene **11** (125 mg) in Et_2O (2 mL) was added (trimethylsilyl)diazomethane (2.25 mL, 4.5 mmol) via syringe. After allowing to stir overnight at room temperature under argon atmosphere, solvents were removed under reduced pressure. The residue was purified via flash chromatography (SiO_2, ramp eluent from 19:1→9:1→4:1 hexane:EtOAc), followed by recrystallization (hexane) to provide **12** (119 mg, 73% yield from carbene complex **10**) as an orange solid. IR: 1691, 1617 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 4.26 (m, 1H), 4.10 (m, 1H), 2.95 (m, 1H), 2.78 (m, 1H), 2.19 (s, 1H), 1.38 (septet, *J* = 7.5 Hz, 3H), 1.29 (s, 3H), 1.04 (d, *J* = 7.4 Hz, 18H), 0.16 (s, 9H). ^{13}C NMR ($CDCl_3$): δ 212.1, 192.6, 131.3, 90.6, 67.1, 55.8, 27.2, 23.5, 18.7, 18.6, 11.4, -1.0. HRMS (ESI): m/z calcd for $C_{19}H_{36}O_2Si_2$ $[M]^+$: 352.2239; found: 352.2189.



Cyclopentenone 13. A solution of silyl vinylketene **11** (125 mg) in Et_2O (5 mL) was added via cannula to an excess of freshly prepared phenyldiazomethane[2] in Et_2O (5 mL). After allowing to stir overnight at room temperature under argon atmosphere, solvents were removed under reduced pressure. The residue was dissolved in THF, and a small amount of silica gel was added to react with excess phenyldiazomethane. The silica gel was removed by filtration through a plug of Celite, washing with Et_2O. The solution was then washed successively with sat. aq. $NaHCO_3$ and sat. aq. NaCl. The organic layer was dried over $MgSO_4$, filtered, and concentrated to a yellow oil. Purification via flash chromatography (SiO_2, 9:1 hexane:Et_2O eluent), followed by recrystallization (hexane) afforded **13** (126 mg, 76% yield from carbene complex **10**) as a pale yellow solid. IR: 1705 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 7.27 (m, 5H), 4.28 (m, 1H), 4.18 (m, 1H), 3.94 (s, 1H), 2.98 (m, 2H), 1.44 (septet, *J* = 7.4 Hz, 3H), 1.07 (m, 18H), 0.93 (s, 3H). ^{13}C NMR ($CDCl_3$): δ 208.7, 189.6, 135.2, 131.3, 130.0, 128.2, 127.1, 90.5, 67.7, 66.9, 27.8, 22.1, 18.7, 18.6, 11.3. HRMS (ESI): m/z calcd for $C_{23}H_{34}O_2Si$ $[M]^+$: 370.2328; found: 370.2328.

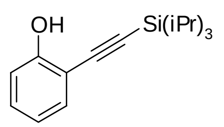


Methoxymethyl 2-iodophenyl ether 15.[3] A solution of 2-iodophenol (5.00 g, 22.7 mmol) and N,N-diisopropylethylamine (5.15 mL, 29.5 mmol) in CH_2Cl_2 (25 mL) was cooled to 0 °C under argon atmosphere, and chloromethyl methyl ether 2.24 mL, 29.5 mmol) was added via syringe. The resultant mixture was allowed to warm to room temperature while stirring. After 2 h, the mixture was washed successively with H_2O, 10% aq. $CuSO_4$, and sat. aq. NaCl. The organic layer was dried over $MgSO_4$, filtered, and concentrated to an orange oil. Purification of this material via flash chromatography (SiO_2, 19:1 hexane:EtOAc eluent) provided **15** (4.99 g, 83% yield) as a clear, colorless oil. ^{1}H NMR ($CDCl_3$): δ 7.78 (dd, *J* = 1.8, 7.7 Hz, 1H), 7.27 (dt, *J* = 1.5, 7.7 Hz, 1H), 7.06 (dd, *J* = 1.5, 8.1 Hz, 1H), 6.74 (dt, *J* = 1.5, 7.7 Hz, 1H), 5.23 (s, 2H), 3.50 (s, 3H). ^{13}C NMR ($CDCl_3$): δ 155.9, 139.3, 129.3, 123.5, 114.8, 94.8, 87.1, 56.2. HRMS (ESI): m/z calcd for $C_8H_9IO_2$ $[M]^+$: 263.9647; found 263.9642.

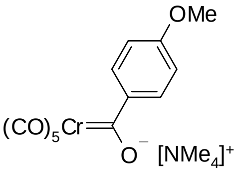


Alkyne 16. A solution of aryl iodide **15** (1.48 g, 5.62 mmol) and triisopropylsilyl acetylene (1.13 g, 6.18 mmol) in diethylamine (15 mL) was degassed using three freeze-pump-thaw cycles, and CuI (321 mg, 1.69 mmol) and $PdCl_2(PPh_3)_2$ (394 mg, 0.56 mmol) were added. The resultant slurry was warmed to 50 °C under argon atmosphere for 4 h. After cooling to room temperature, the mixture was diluted with hexane and washed successively with sat. aq. NH_4Cl and sat. aq. NaCl. The organic layer was dried over $MgSO_4$, filtered, and concentrated to an oil. Purification via flash chromatography (SiO_2, 19:1 hexane:EtOAc eluent) provided **16** (1.36 g, 76% yield) as a clear yellow oil. IR: 2158 cm^{-1}. ^{1}H NMR

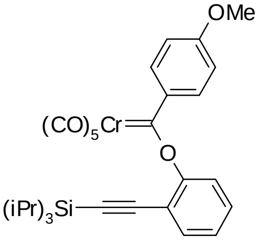
($CDCl_3$): δ 7.44 (dd, *J* = 1.8, 7.7 Hz, 1H), 7.24 (dt, *J* = 1.8, 7.9 Hz, 1H), 7.05 (d, *J* = 7.4 Hz, 1H), 6.93 (t, *J* = 7.4 Hz, 1H), 5.23 (s, 2H), 3.50 (s, 3H), 1.14 (s, 21H); ^{13}C NMR ($CDCl_3$): δ 158.2, 133.8, 129.5, 121.7, 115.3, 114.4, 103.1, 94.9, 94.8, 56.1, 18.6, 11.4. HRMS (ESI): m/z calcd for $C_{19}H_{30}O_2Si$ $[M]^+$: 318.2015; found 318.2008.



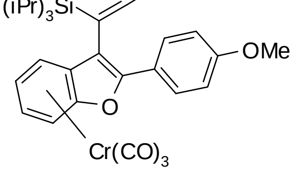
Phenol 6. A solution of compound **16** (5.50 g, 17.3 mmol) in 10% aq. HCl / acetone (170 mL) was allowed to stir at room temperature for 3 h. The reaction mixture was then diluted with Et_2O and washed successively with sat. aq. $NaHCO_3$ and sat. aq. NaCl. The organic layer was dried over $MgSO_4$, filtered, and concentrated to a yellow oil. Purification via flash chromatography (SiO_2, ramp eluent from hexane to 50:1 hexane:EtOAc) provided **6** (3.6 g, 76% yield) as a clear, colorless oil. IR: 3508, 2148 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 7.35 (dd, *J* = 1.5, 6.6 Hz, 1H), 7.24 (dt, *J* = 1.5, 6.6 Hz, 1H), 6.95 (d, *J* = 8.1 Hz, 1H), 5.88 (s, 1H), 1.13 (s, 21H). ^{13}C NMR ($CDCl_3$): δ 157.2, 131.5, 130.5, 120.1, 114.4, 109.8, 100.9, 98.6, 18.6, 11.2. HRMS (ESI): m/z calcd for $C_{17}H_{26}OSi$ $[M]^+$: 274.1753; found 274.1744.

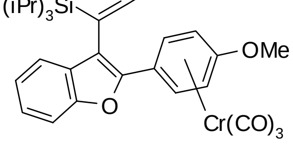


Tetramethylammonium chromium acylate complex 7. A solution of *p*-bromoanisole (2.81 g, 15.0 mmol) in THF (20 mL) was cooled to -78 °C under argon atmosphere, and *n*-BuLi (9.4 mL of a 1.6 M solution, 15.0 mmol) was added dropwise via syringe. After allowing to stir at -78 °C for 2 h, the resultant aryllithium solution was transferred via cannula to a suspension of $Cr(CO)_6$ (3.30 g, 15.0 mmol) in THF (20 mL) at -78 °C under argon atmosphere. The reaction mixture was then warmed to room temperature and allowed to stir for 2 h. The solvents were removed under reduced pressure, and the residue was suspended in H_2O. An aqueous solution of tetramethylammonium bromide (2.31 g, 15.0 mmol) was then added via addition funnel, and the reaction mixture was allowed to stir for an additional 30 min. The solids were collected by filtration, suspended in CH_2Cl_2, and collected by filtration again. Final trituration with acetone provided **7** (4.75 g, 79% yield) as a bright orange solid. IR: 2031, 1949, 1901, 1853 cm^{-1}. ^{1}H NMR (d_6-acetone): δ 7.50 (d, *J* = 8.8 Hz, 2H), 6.82 (d, *J* = 8.8 Hz, 2H), 3.77 (s, 3H), 3.41 (s, 12H). ^{13}C NMR (d_6-acetone): δ 228.8, 224.6, 160.5, 148.7, 127.7, 112.9, 55.99, 55.94, 55.89, 55.4.

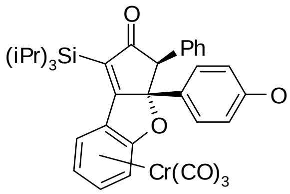


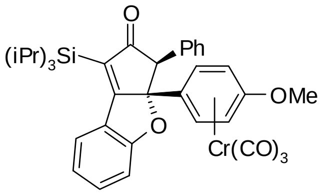
Aryloxy carbene complex 5. A solution of tetramethylammonium chromium acylate complex **7** (803 mg, 2.00 mmol) in CH_2Cl_2 (33 mL) was cooled to -40 °C under argon atmosphere, and freshly distilled acetyl bromide (163 µL, 2.20 mmol) was added via syringe. The resultant red solution was allowed to stir for 1 h at -40 °C. In a separate flask, a solution of phenol **6** (604 mg, 2.20 mmol) in Et_2O (15 mL) was cooled to -78 °C under argon atmosphere and treated with sodium hydride (88 mg, 2.2 mmol, 60% dispersion in oil). This mixture was warmed to 0 °C, allowed to stir for 30 min, and then cooled to -40 °C. The sodium phenolate generated in this manner was then transferred via cannula to the acyloxy carbene complex solution, and the resultant solution was allowed to stir for 2 h at -40 °C. After warming to room temperature, the volume of solvent was reduced to approximately 10 mL, and then diluted with an additional 40 mL of hexane. This mixture was filtered through Celite, washing with hexane. Solvents were removed under reduced pressure, and the residue was purified via flash chromatography (SiO_2, 19:1 hexane:EtOAc) to provide **5** (878 mg, 75% yield) as a red crystalline solid. IR: 2056, 1935 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 7.99 (d, *J* = 8.8 Hz, 2H), 7.61 (d, *J* = 8.1 Hz, 1H), 7.47 (t, *J* = 7.4 Hz, 1H), 7.32 (t, *J* = 7.4 Hz, 1H), 7.23 (d, *J* = 5.9 Hz, 1H), 6.91 (d, *J* = 8.8 Hz, 2H), 3.89 (s, 3H), 0.94 (s, 21H). ^{13}C NMR ($CDCl_3$): δ 342.6, 224.4, 216.0, 163.9, 159.8, 146.6, 134.8, 131.1, 129.6, 127.0, 122.7, 118.3, 113.0, 101.2, 97.7, 55.6, 18.5, 11.2. Due to the thermal instability of this complex, HRMS data could not be obtained.



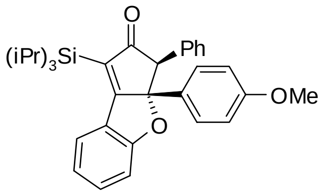


Silyl vinylketenes 17a and 17b. A solution of aryloxy carbene complex **5** (857 mg, 1.47 mmol) in benzene (100 mL) was degassed with three cycles of freeze-pump-thaw degassing. The solution was then heated to 65 °C under argon atmosphere for 2 h, after which time TLC analysis showed complete consumption of starting material. The solvent was removed under reduced pressure to afford an approximately 2:1 mixture of **17a**/**17b** (820 mg) as a yellow-orange solid which was typically utilized without further purification. For characterization purposes, the major isomer **17a** could be crystallized in high purity from Et_2O solution at 0 °C. The mother liquor could then be subjected to a second crystallization at -78 °C, which removed remaining traces of **17a** and left minor isomer **17b** of high purity in solution. Spectral data for **17a**: IR: 2093, 1961, 1882 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 7.83 (d, *J* = 8.8 Hz, 2H), 6.95 (d, *J* = 8.8 Hz, 2H), 6.11 (m, 2H), 5.39 (t, *J* = 6.6 Hz, 1H), 5.02 (t, *J* = 5.9 Hz, 1H), 3.85 (s, 3H), 1.05 (m, 21H); ^{13}C ($CDCl_3$): δ 232.9, 175.1, 160.8, 157.4, 134.5, 128.8, 121.8, 114.1, 104.3, 103.4, 91.0, 87.0, 85.9, 77.2, 55.4, 18.5, 12.8, 5.1. HRMS (ESI): m/z calcd for $C_{29}H_{32}CrO_6Si$ $[M]^+$: 556.1342; found 556.1357. Spectral data for **17b**: IR: 2091, 1967, 1891 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 7.52 (d, *J* = 7.4 Hz, 1H), 7.44 (d, *J* = 8.1 Hz, 1H), 7.28 (m, 2H), 6.36 (d, *J* = 7.4 Hz, 2H), 5.21 (d, *J* = 7.4 Hz, 2H), 3.76 (s, 3H), 1.07 (s, 21H). ^{13}C NMR ($CDCl_3$): δ 232.1, 175.4, 153.8, 148.1, 142.4, 130.9, 125.5, 123.3, 120.2, 111.4, 104.9, 92.3, 91.4, 77.2, 55.8, 18.5, 12.9, 12.7, 5.7. HRMS (ESI): m/z calcd for $C_{29}H_{32}CrO_6Si$ $[M]^+$: 556.1342; found 556.1363.





Cyclopentenones 18a and 18b. A solution of silyl vinylketenes **17a**/**17b** (820 mg) in Et_2O (15 mL) was added via cannula to an excess of freshly prepared phenyldiazomethane^2 in Et_2O (15 mL). After allowing to stir overnight at room temperature under argon atmosphere, solvents were removed under reduced pressure. The residue was dissolved in THF, and a small amount of silica gel was added to react with excess phenyldiazomethane. The silica gel was removed by filtration through a plug of Celite, washing with Et_2O. The solution was then washed successively with sat. aq. $NaHCO_3$ and sat. aq. NaCl. The organic layer was dried over $MgSO_4$, filtered, and concentrated to afford an approximately 2:1 mixture of **18a**/**18b** (855 mg) as a red solid which was typically utilized without further purification. For characterization purposes, the isomers could be separated by flash chromatography (SiO_2, 9:1 hexane:EtOAc), followed by final purification via crystallization (hexane). Spectral data for **18a**: IR: 1968, 1895, 1704 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 7.08 (m, 3H), 6.87 (d, *J* = 9.6 Hz, 2H), 6.72 (m, 2H), 6.59 (d, *J* = 8.8 Hz, 2H), 6.39 (d, *J* = 5.9 Hz, 1H), 5.60 (t, *J* = 6.3 Hz, 1H), 5.30 (d, *J* = 6.6 Hz, 1H), 4.78 (t, *J* = 6.3 Hz, 1H), 4.68 (s, 1H), 1.68 (septet, *J* = 7.4 Hz, 3H), 1.28 (m, 18H). ^{13}C NMR ($CDCl_3$): δ 231.8, 205.5, 177.2, 159.7, 147.3, 137.3, 133.6, 130.3, 129.3, 127.8, 127.4, 127.3, 113.3, 104.3, 94.2, 93.8, 90.6, 83.2, 72.7, 67.5, 55.2, 19.5, 19.4, 12.6. HRMS (ESI): m/z calcd for $C_{36}H_{38}CrO_6Si$ $[M]^+$: 646.1843; found 646.1837. Spectral data for **18b**: IR: 1967, 1889, 1702 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 7.78 (d, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.4 Hz, 1H), 7.21 (m, 3H), 7.07 (m, 2H), 6.96 (m, 2H), 5.43 (dd, *J* = 2.2, 7.4 Hz, 1H), 5.27 (dd, *J* = 1.8, 6.9 Hz, 1H), 4.79 (dd, *J* = 2.2, 7.4 Hz, 1H), 4.55 (s, 1H), 4.52 (dd, *J* = 2.6, 7.4 Hz, 1H), 1.71 (septet, *J* = 7.4 Hz, 3H), 1.16 (m, 18H). ^{13}C NMR ($CDCl_3$): δ 231.7, 204.9, 179.3, 163.7, 144.2, 134.7, 134.5, 133.8, 130.6, 128.1, 127.7, 126.8, 122.6, 122.0, 113.4, 101.8, 98.3, 94.3, 92.3, 74.4, 73.6, 69.8, 55.5, 19.1, 19.0, 12.1. HRMS (ESI): m/z calcd for $C_{36}H_{38}CrO_6Si$ $[M]^+$: 646.1843; found 646.1859.



Cyclopentenone 3. The approximately 2:1 mixture of cyclopentenones **18a**/**18b** (855 mg) was dissolved in MeOH (20 mL) containing several drops of THF. The solution was cooled to 0° C and ammonium cerium(IV) nitrate (2.42 g, 4.41 mmol) was added, resulting in essentially immediate loss of the red color. After stirring at 0° C for 30 min, the solution was diluted with Et_2O and washed successively with H_2O and sat. aq. NaCl. The organic layer was dried over $MgSO_4$, filtered, and solvents were removed under reduced pressure. Purification of the residue via flash chromatography (SiO_2, 19:1 hexane:EtOAc) and recrystallization from hexane provided **3** (566 mg, 81% yield from carbene complex **5**) as a yellow solid. IR: 1703 cm^{-1}. ^{1}H NMR ($CDCl_3$) □ 7.70 (d, *J* = 7.4 Hz, 1H), 7.36 (t, *J* = 7.4 Hz, 1H), 7.10 (m, 3H), 7.01 (m, 4H), 6.85 (m, 2H), 6.59 (d, *J* = 8.8 Hz, 2H), 4.54 (s, 1H), 3.65 (s, 3H), 1.73 (septet, *J* = 7.4 Hz, 3H), 1.16 (d, 18H, *J* = 7.4 Hz). ^{13}C NMR ($CDCl_3$) □ 206.5, 181.6, 164.6, 159.2, 134.5, 134.1, 132.7, 130.3, 127.7, 127.3, 127.0, 126.3, 122.6, 121.9, 113.2, 112.6, 101.8, 68.6, 55.1, 19.0, 18.96, 12.1. HRMS (ESI): m/z calcd for $C_{33}H_{38}O_3Si$ $[M]^+$: 510.2590; found 510.2593.

References

1. (a) Fischer, E.O.; Maasböl, A. *Chem. Ber*. **1967**, *100*, 2445. (b) Murray, C.K.; Yang, D.C.; Wulff, W.D. *J. Am. Chem. Soc*. **1990**, *112*, 5660.
2. Creary, X. In *Organic Syntheses*; Wiley & Sons: New York, 1990; Collect. Vol. VII, pp 438-443.
3. Tsang, K.Y.; Brimble, M.A. *Tetrahedron* **2007**, *63*, 6015.

Crystallographic data for 3. Yellow plate, 0.22 x 0.19 x 0.17 mm^3, $C_{33}H_{38}O_3Si$, M=510.72, Triclinic P-1, a = 10.7432(12) Å, b = 14.6368(16) Å, c = 18.2598(19), V = 2803.3(5) $Å^3$, ρ_{calc} = 1.210 Mg/m^3, μ = 0.116 mm^{-1}. The data collection was carried out using Mo Kα radiation (graphite monochromator) with a frame time of 5 seconds and a detector distance of 5.0 cm. A complete sphere in reciprocal space was surveyed. Four major sections of frames were collected with 0.30º steps in ω at different ϕ settings and a detector position of −43° in 2θ. An additional set of 50 frames was collected in order to model decay. Data to a resolution of 0.77 Å were considered in the reduction. Final cell constants were calculated from the xyz centroids of 7116 strong reflections from the actual data collection after integration. The intensity data were corrected for absorption.

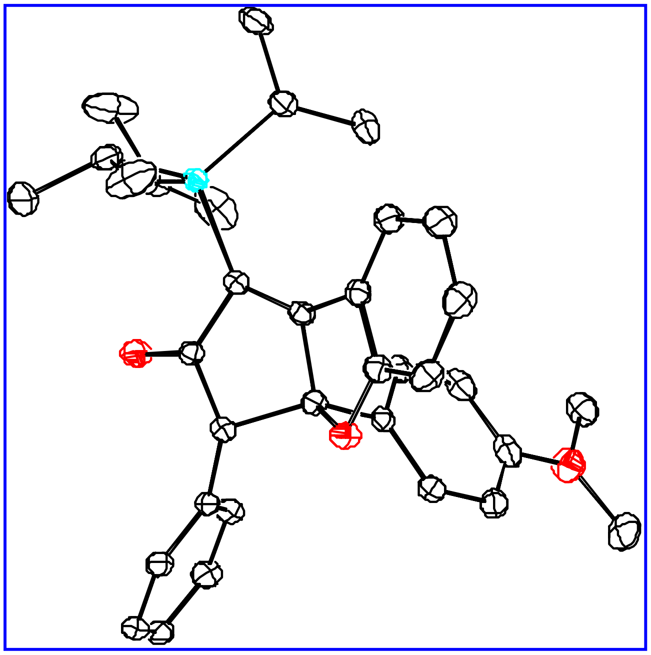
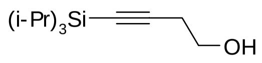


Figure 1. Crystal Structure of cyclopentenone **3** verifying the cis arrangement of the adjacent phenyl and aryl substituents.

10 peaks found in Untitled1

peak	ppm	freq	amp
1	3.716	1117.07	65199.50
2	3.693	1110.45	157399.52
3	3.674	1104.57	80039.69
4	2.529	760.42	75507.26
5	2.507	753.80	127015.69
6	2.488	747.92	81434.73
7	1.847	555.26	148593.89
8	1.040	312.59	681067.12
9	1.032	310.39	1518301.62
10	1.010	303.77	210490.94



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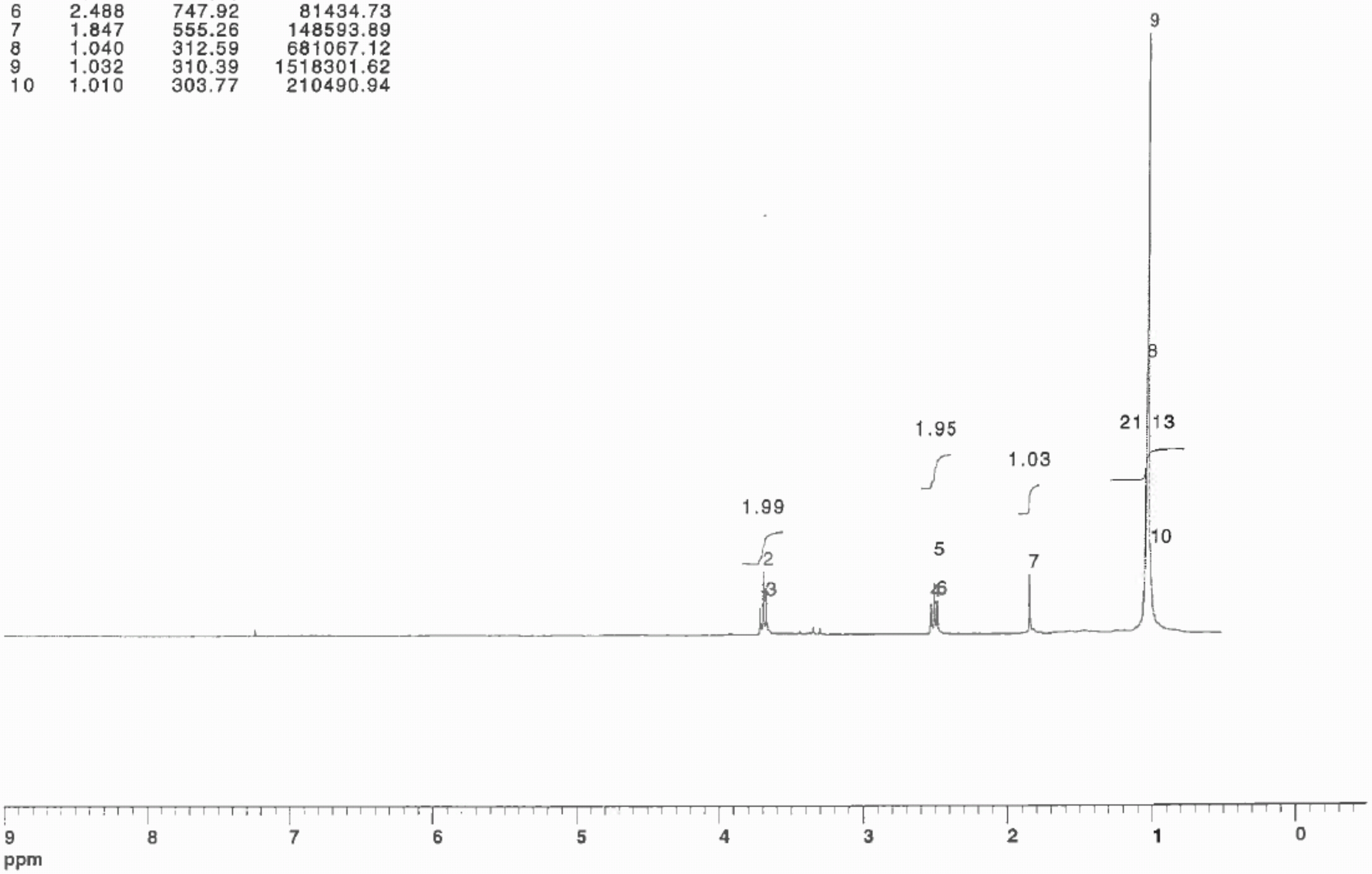
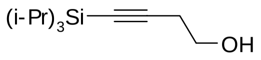


9 peaks found in MG421C

peak	ppm	freq	amp
1	104.935	7932.91	6155182.00
2	82.974	6272.76	5838683.00
3	77.420	5852.83	22862046.00
4	77.000	5821.10	21771480.00
5	76.564	5788.14	21852840.00
6	61.160	4623.59	26374234.00
7	24.328	1839.16	20508244.00
8	18.580	1404.59	111913488.00
9	11.184	845.51	52045392.00



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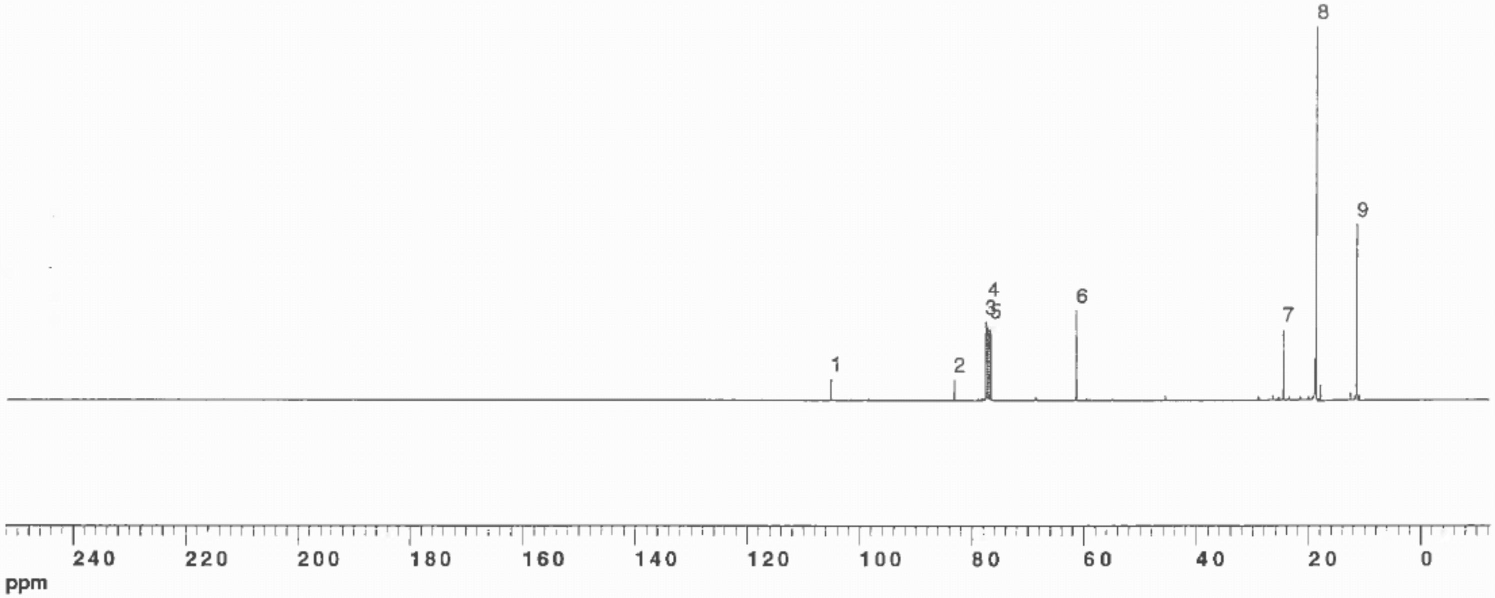

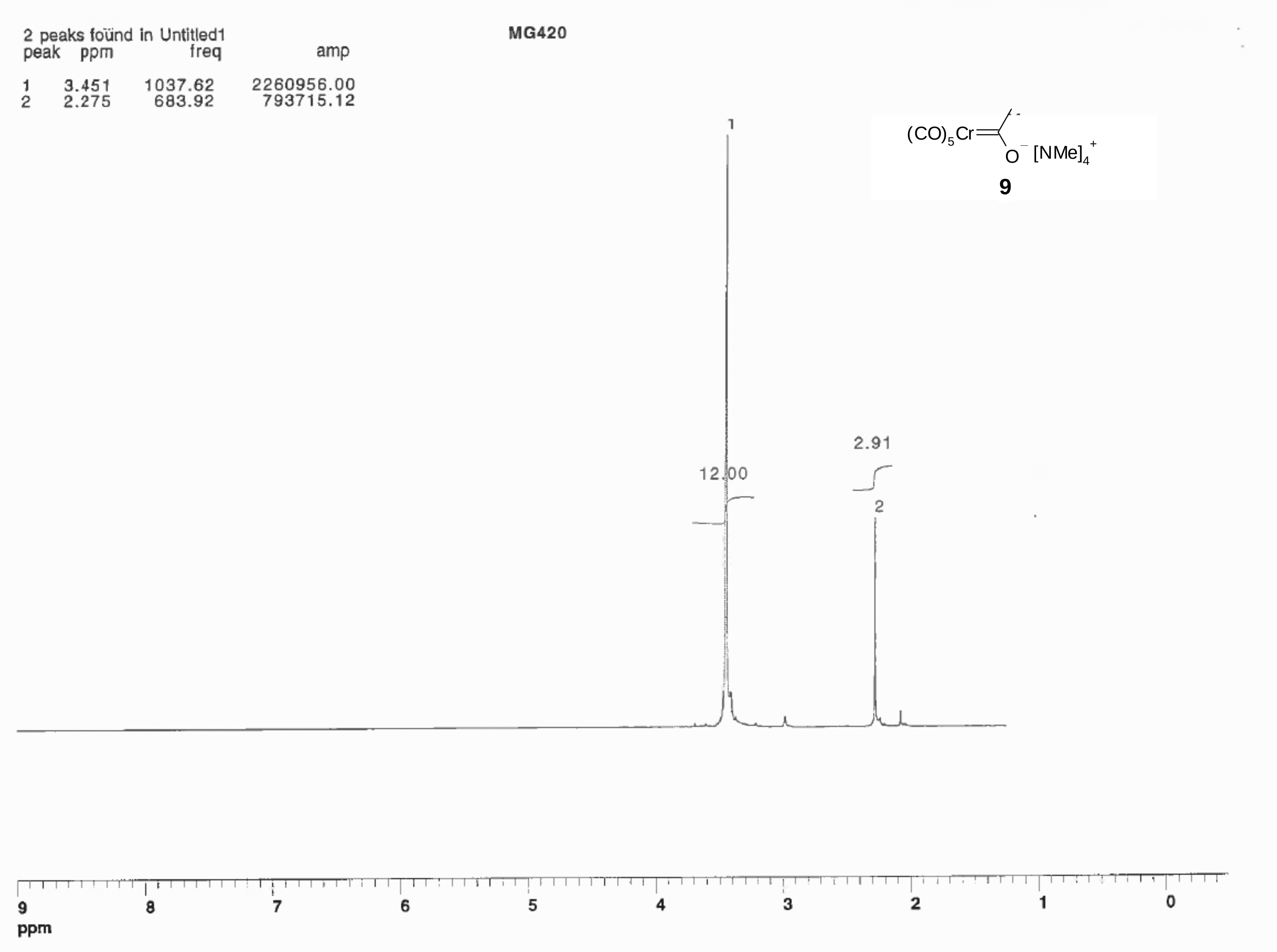

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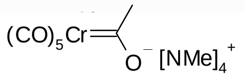
2 peaks foünd in Untitled1
peak   ppm        freq          amp

1     3.451    1037.62    2260956.00
2     2.275     683.92     793715.12
```

9

MG420



9

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14 peaks found in MG420C
peak        ppm        freq             amp

1       228.832    17299.40      6558215.50
2       224.634    16982.02     22140542.00
3       206.000    15573.33     72308048.00
4        55.767     4215.91     37726556.00
5        55.719     4212.24     41211924.00
6        55.670     4208.58     34240268.00
7        52.441     3964.44     17441942.00
8        30.319     2292.08      8771107.00
9        30.061     2272.55     24994032.00
10       29.802     2253.02     47158356.00
11       29.544     2233.48     50000576.00
12       29.302     2215.17     37968308.00
13       29.043     2195.64     21591528.00
14       28.785     2176.11      7890332.50
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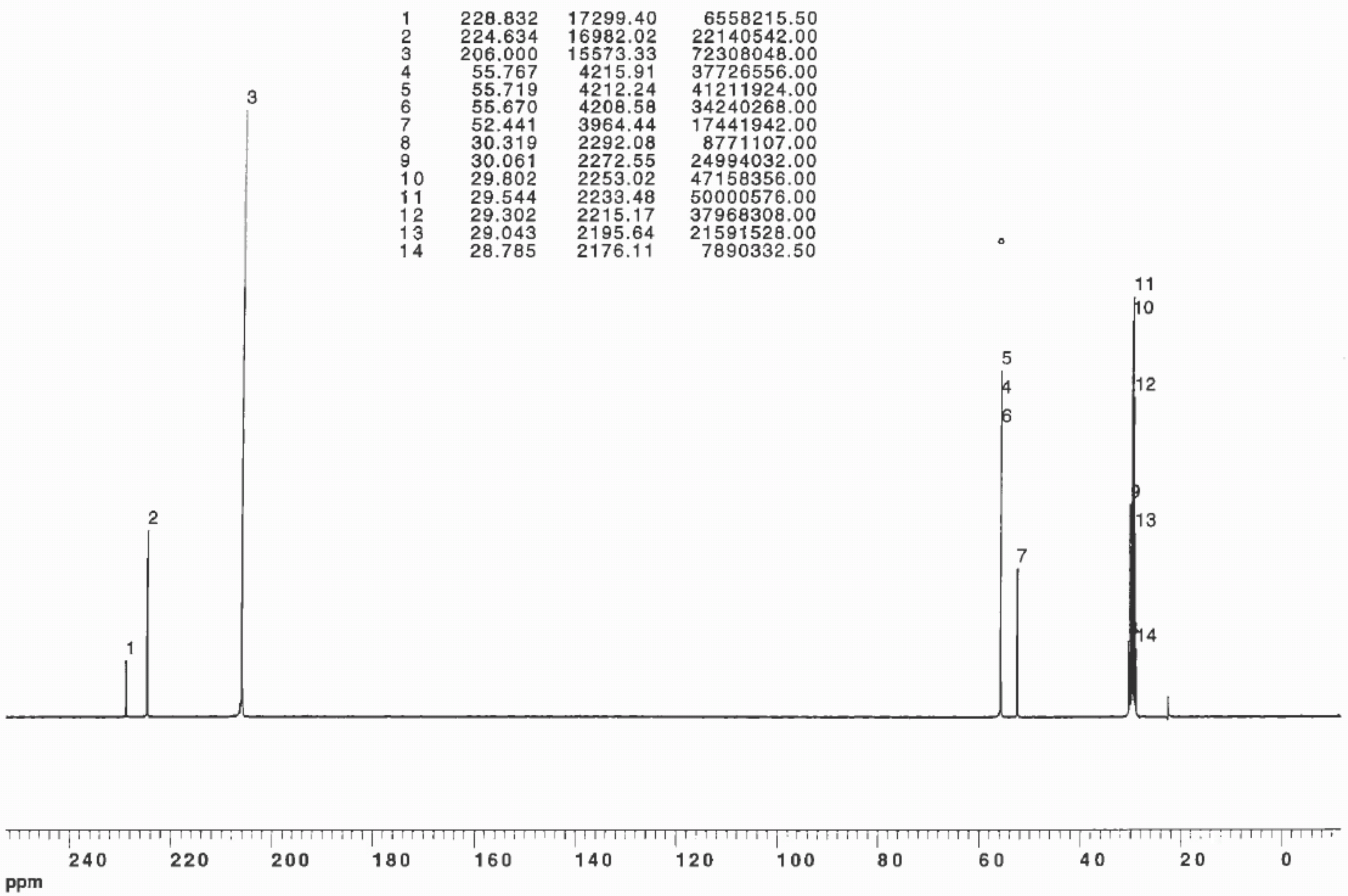

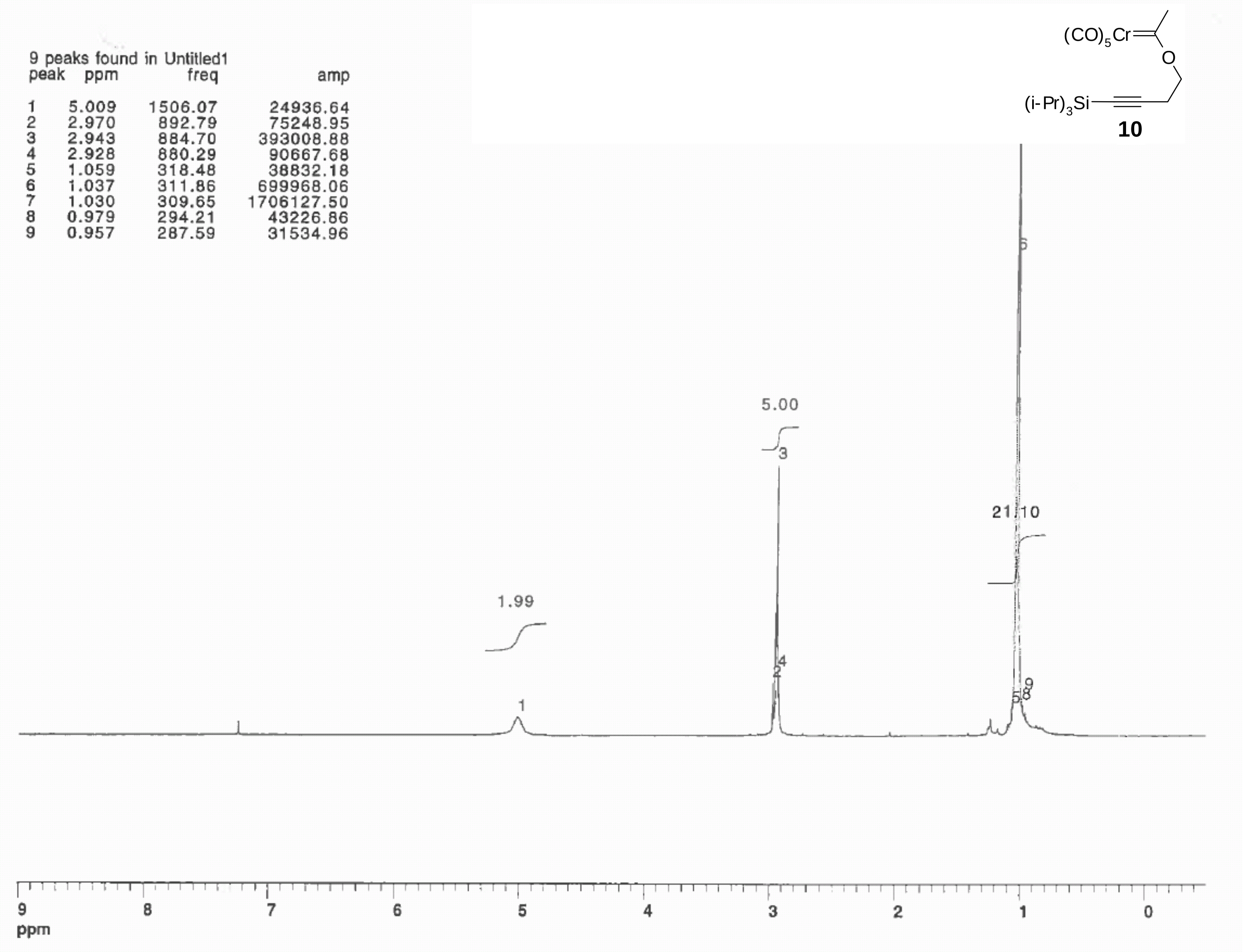

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9 peaks found in Untitled1
peak   ppm      freq          amp

1     5.009   1506.07     24936.64
2     2.970    892.79     75248.95
3     2.943    884.70    393008.88
4     2.928    880.29     90667.68
5     1.059    318.48     38832.18
6     1.037    311.86    699968.06
7     1.030    309.65   1706127.50
8     0.979    294.21     43226.86
9     0.957    287.59     31534.96
```
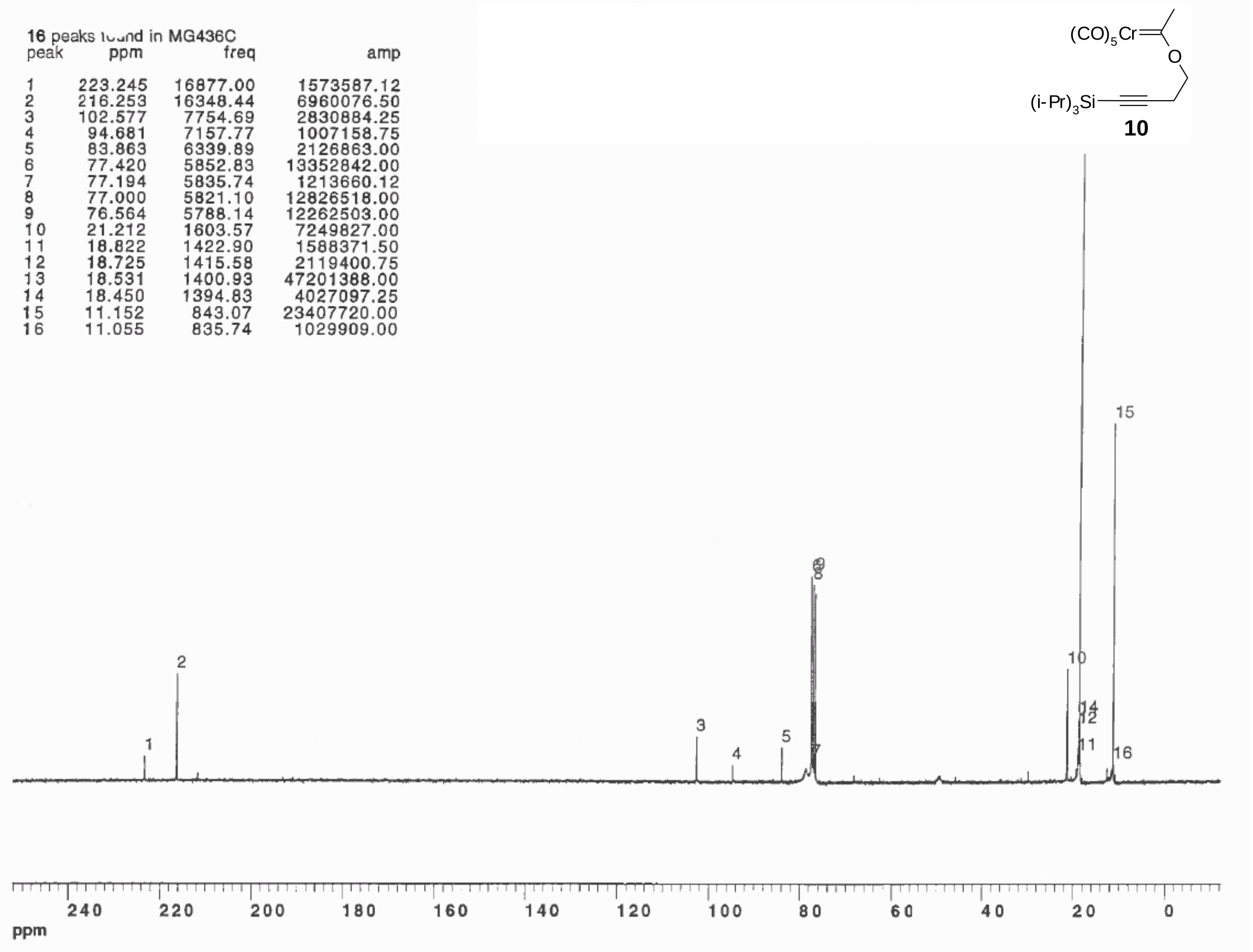

10

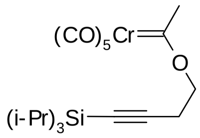


10

16 peaks found in MG436C

peak	ppm	freq	amp
1	223.245	16877.00	1573587.12
2	216.253	16348.44	6960076.50
3	102.577	7754.69	2830884.25
4	94.681	7157.77	1007158.75
5	83.863	6339.89	2126863.00
6	77.420	5852.83	13352842.00
7	77.194	5835.74	1213660.12
8	77.000	5821.10	12826518.00
9	76.564	5788.14	12262503.00
10	21.212	1603.57	7249827.00
11	18.822	1422.90	1588371.50
12	18.725	1415.58	2119400.75
13	18.531	1400.93	47201388.00
14	18.450	1394.83	4027097.25
15	11.152	843.07	23407720.00
16	11.055	835.74	1029909.00

INDEX	FREQUENCY	PPM	HEIGHT
1	18060.571	359.173	35.1



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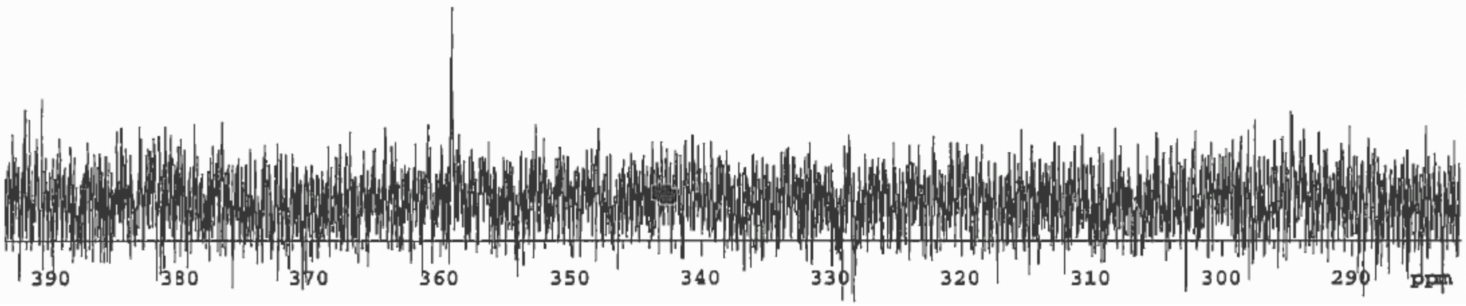

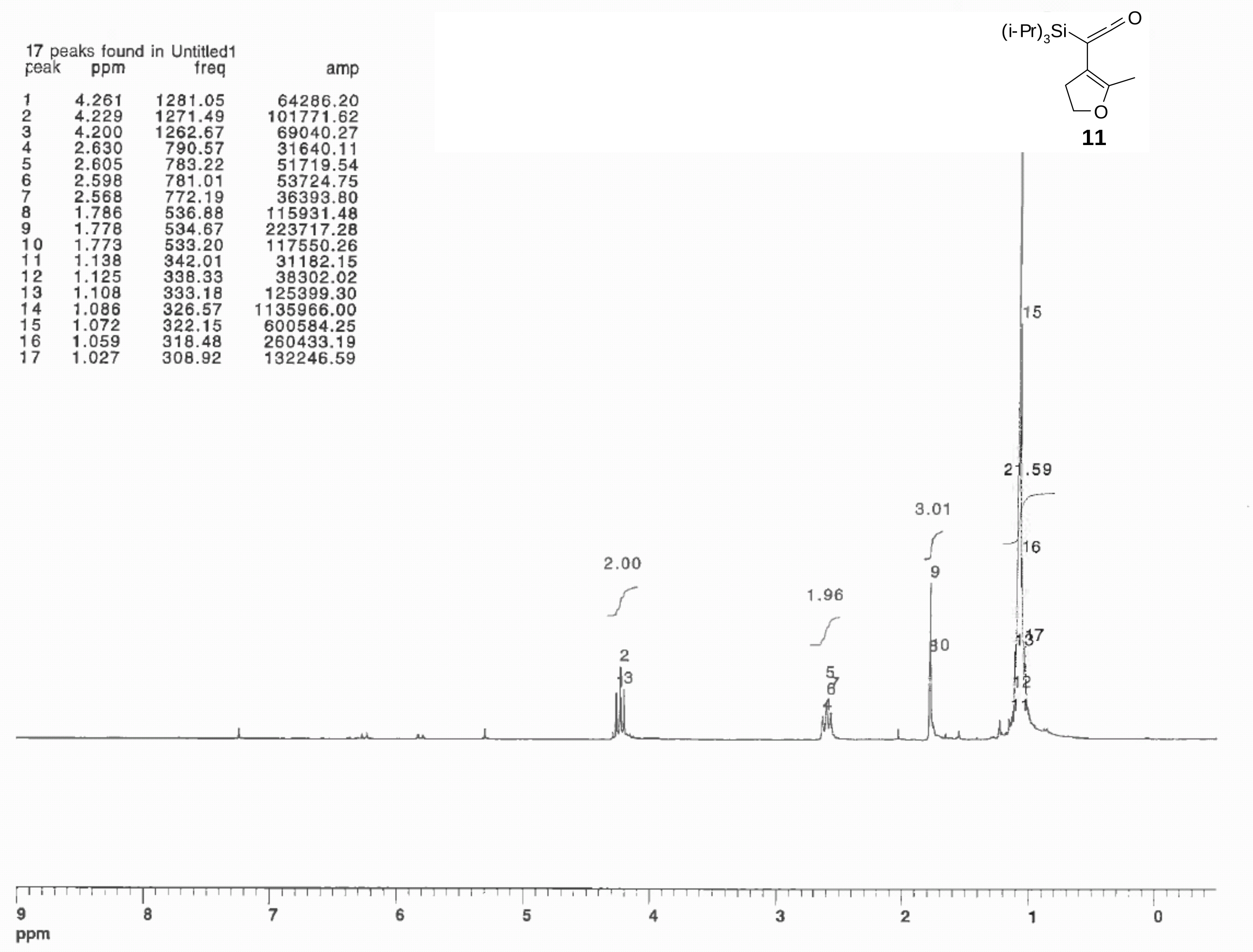

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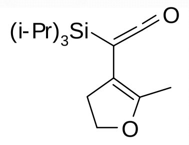
17 peaks found in Untitled1
peak    ppm      freq          amp

 1     4.261   1281.05     64286.20
 2     4.229   1271.49    101771.62
 3     4.200   1262.67     69040.27
 4     2.630    790.57     31640.11
 5     2.605    783.22     51719.54
 6     2.598    781.01     53724.75
 7     2.568    772.19     36393.80
 8     1.786    536.88    115931.48
 9     1.778    534.67    223717.28
10     1.773    533.20    117550.26
11     1.138    342.01     31182.15
12     1.125    338.33     38302.02
13     1.108    333.18    125399.30
14     1.086    326.57   1135966.00
15     1.072    322.15    600584.25
16     1.059    318.48    260433.19
17     1.027    308.92    132246.59
```

20 peaks found in MG437C

peak	ppm	freq	amp
1	150.211	11355.76	3195432.75
2	138.408	10463.43	3898807.75
3	106.420	8045.22	3325125.75
4	94.633	7154.10	3928797.75
5	77.420	5852.83	20456484.00
6	77.000	5821.10	19849504.00
7	76.580	5789.36	17846352.00
8	68.329	5165.58	3962223.25
9	68.151	5152.15	22113736.00
10	36.002	2721.73	19080628.00
11	30.868	2333.55	3308522.50
12	18.806	1421.68	6020459.50
13	18.725	1415.58	6620743.50
14	18.547	1402.15	4589313.50
15	18.450	1394.83	104405448.00
16	17.740	1341.11	20069800.00
17	12.395	937.06	47985696.00
18	12.024	908.99	9104796.00
19	11.975	905.32	11722613.00
20	11.055	835.74	4330837.50



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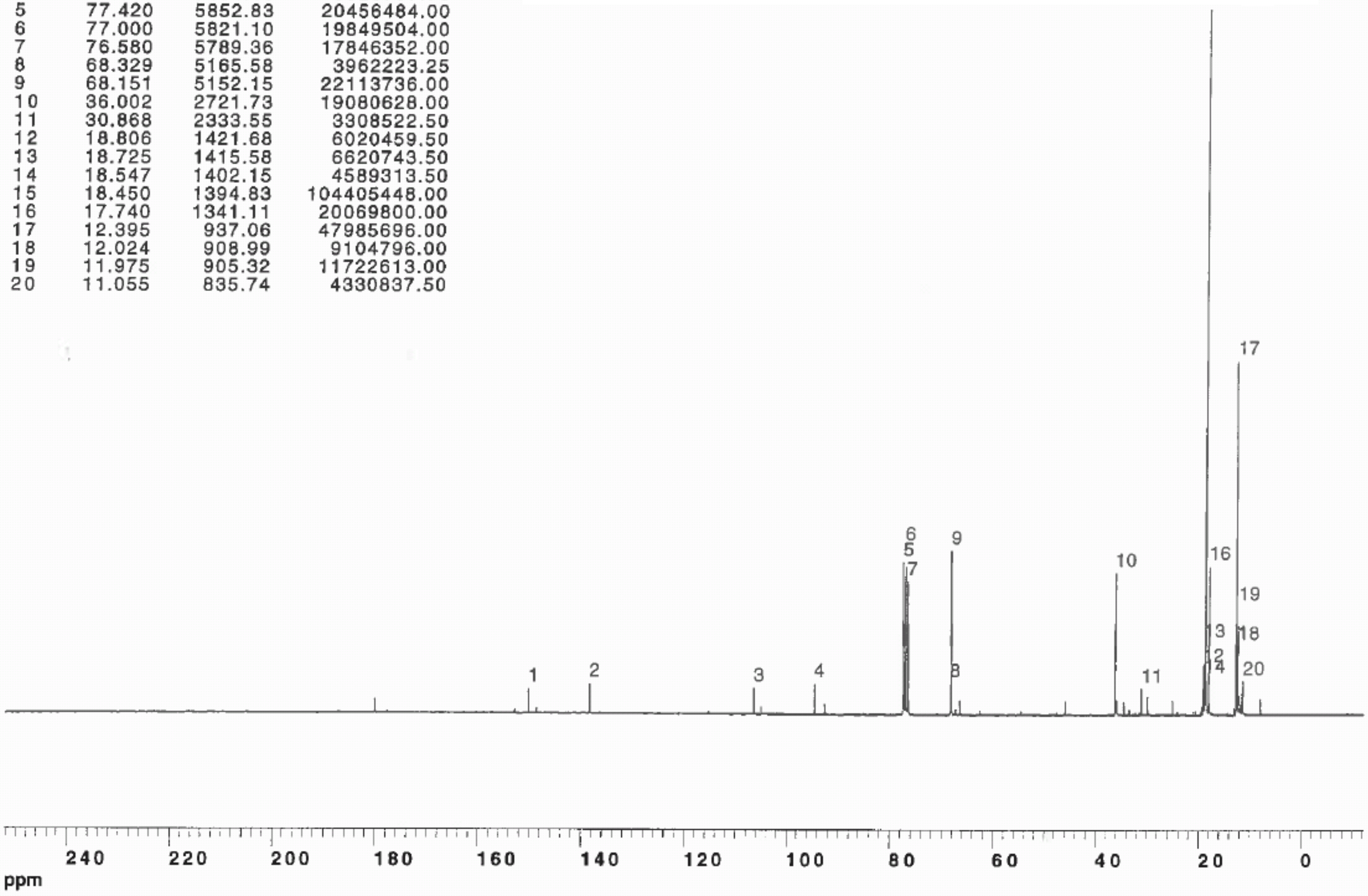

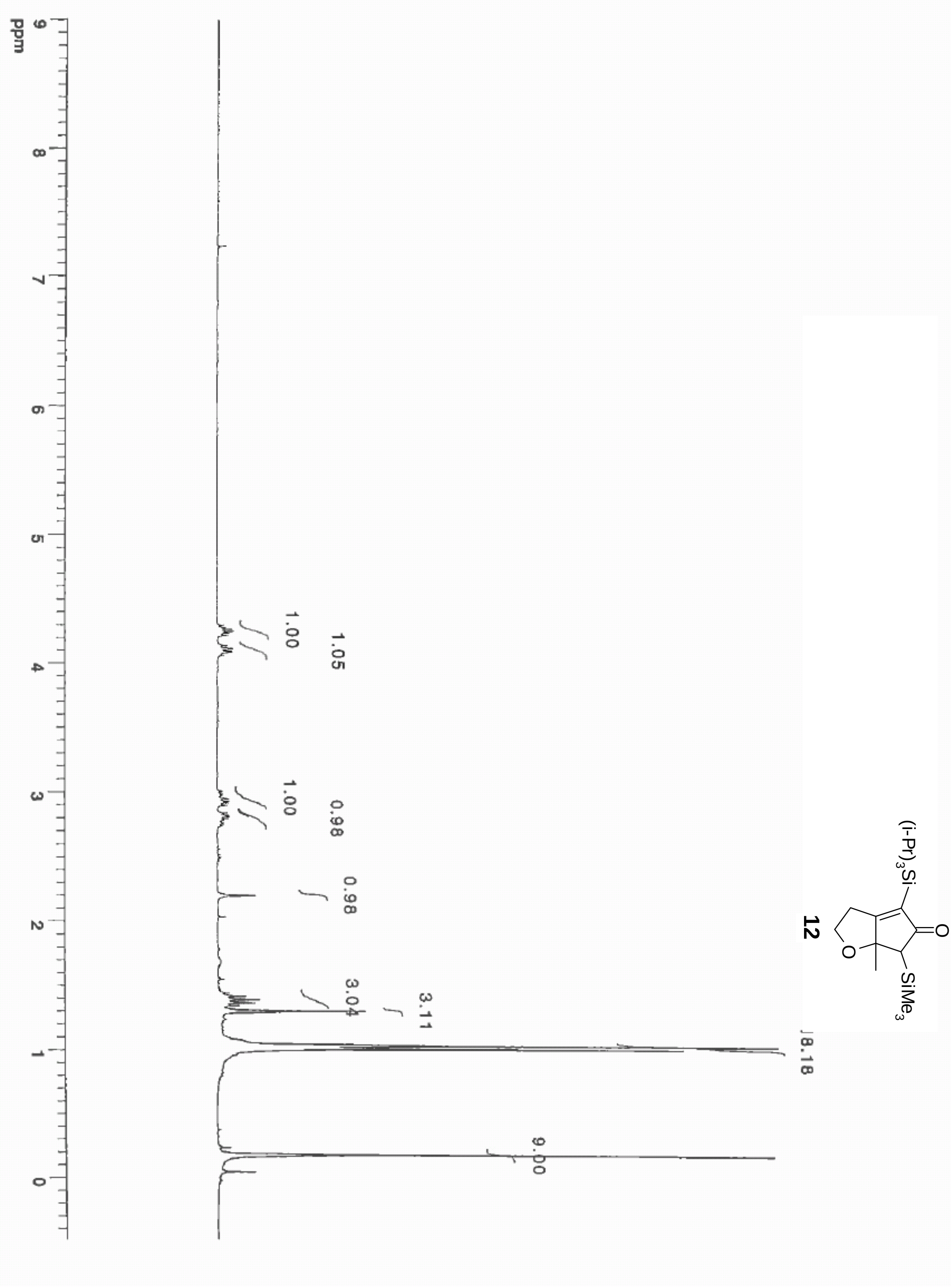
(i-Pr)$_3$Si
O
SiMe$_3$
O
ppm
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8
7
6
5
4
3
2
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0
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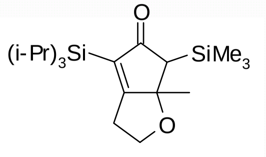
1.05
1.00
0.98
0.98
3.04
3.11
18.18
9.00

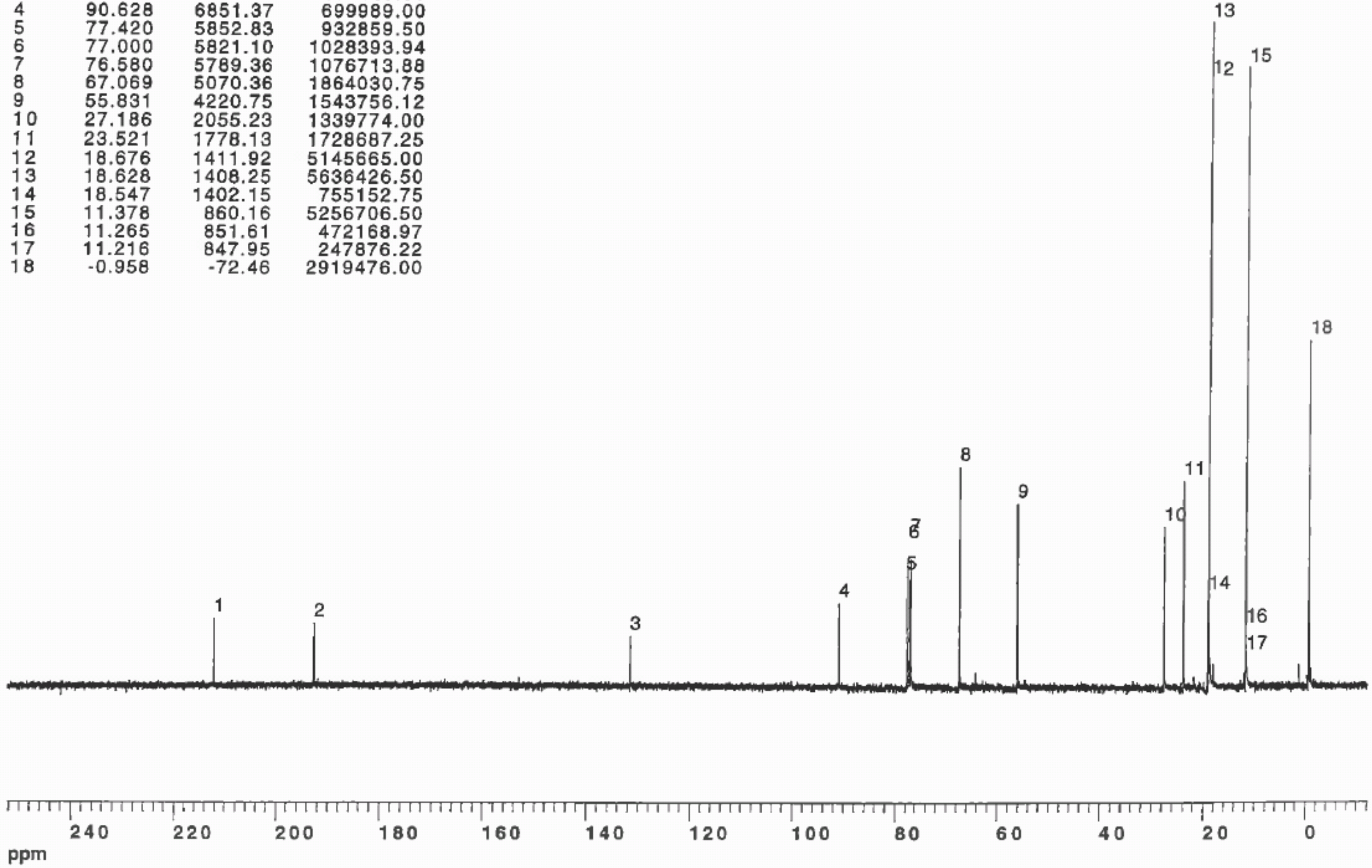
12



12

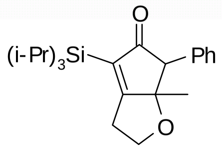
18 peaks found in MG19C

peak	ppm	freq	amp
1	212.055	16031.06	562276.31
2	192.614	14561.33	522177.97
3	131.254	9922.66	417428.66
4	90.628	6851.37	699989.00
5	77.420	5852.83	932859.50
6	77.000	5821.10	1028393.94
7	76.580	5789.36	1076713.88
8	67.069	5070.36	1864030.75
9	55.831	4220.75	1543756.12
10	27.186	2055.23	1339774.00
11	23.521	1778.13	1728687.25
12	18.676	1411.92	5145665.00
13	18.628	1408.25	5636426.50
14	18.547	1402.15	755152.75
15	11.378	860.16	5256706.50
16	11.265	851.61	472168.97
17	11.216	847.95	247876.22
18	-0.958	-72.46	2919476.00

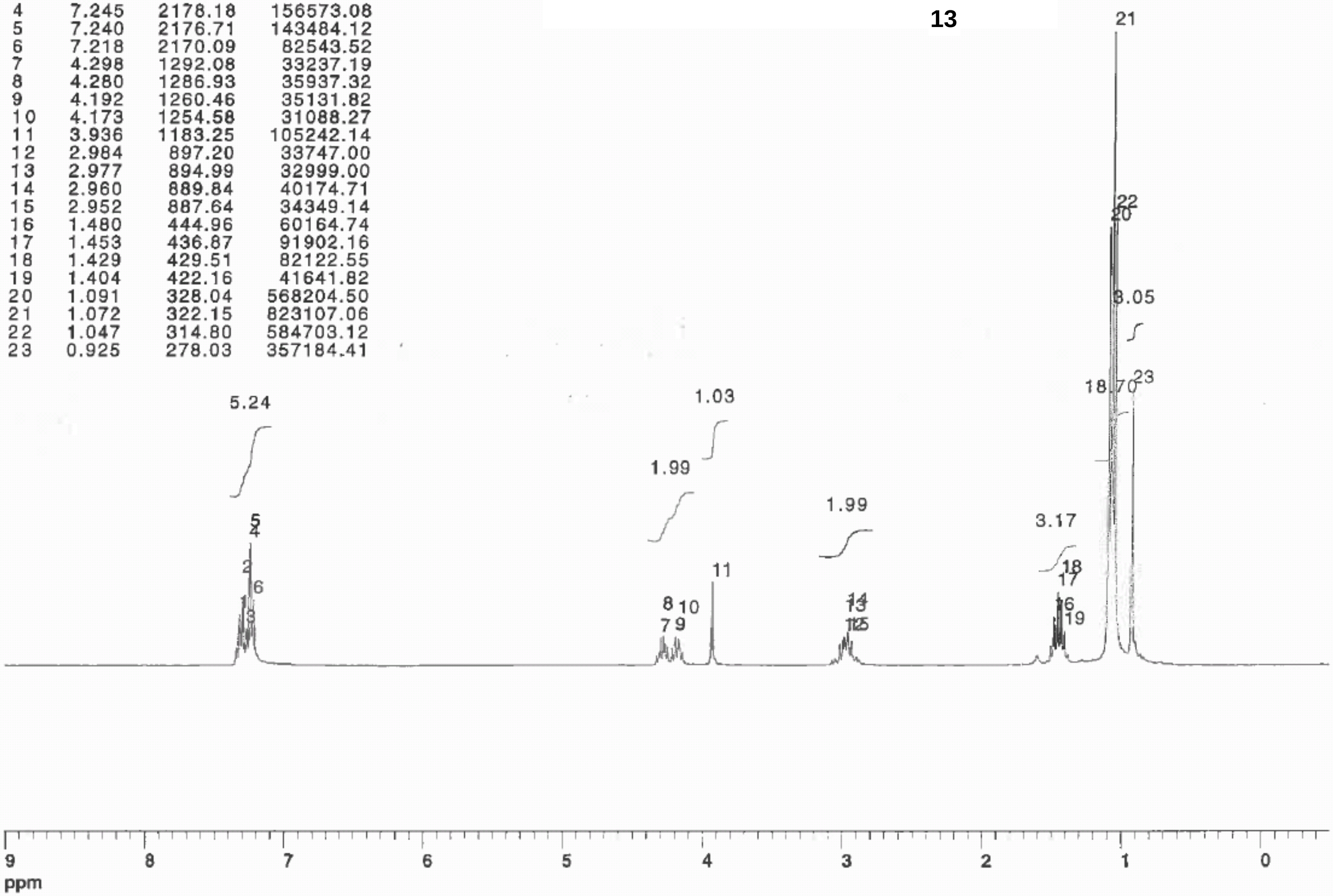


23 peaks found in Untitled1

peak	ppm	freq	amp
1	7.318	2200.24	63347.99
2	7.296	2193.62	83087.71
3	7.267	2184.80	44568.29
4	7.245	2178.18	156573.08
5	7.240	2176.71	143484.12
6	7.218	2170.09	82543.52
7	4.298	1292.08	33237.19
8	4.280	1286.93	35937.32
9	4.192	1260.46	35131.82
10	4.173	1254.58	31088.27
11	3.936	1183.25	105242.14
12	2.984	897.20	33747.00
13	2.977	894.99	32999.00
14	2.960	889.84	40174.71
15	2.952	887.64	34349.14
16	1.480	444.96	60164.74
17	1.453	436.87	91902.16
18	1.429	429.51	82122.55
19	1.404	422.16	41641.82
20	1.091	328.04	568204.50
21	1.072	322.15	823107.06
22	1.047	314.80	584703.12
23	0.925	278.03	357184.41



13



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18 peaks found in MG438C
peak       ppm       freq               amp

1      208.712   15778.37        2303146.00
2      189.594   14333.06        3512833.75
3      135.227   10222.95        3712638.00
4      131.271    9923.88        2606290.75
5      130.043    9831.11       44193440.00
6      128.235    9694.39       44609572.00
7      127.105    9608.94       21967932.00
8       90.499    6841.60        3627008.00
9       77.420    5852.83       21748168.00
10      77.000    5821.10       23653476.00
11      76.580    5789.36       23877096.00
12      67.715    5119.19       15390203.00
13      66.940    5060.60       21996092.00
14      27.848    2105.28       17754688.00
15      22.067    1668.26       16222281.00
16      18.709    1414.36       48633464.00
17      18.644    1409.47       59604936.00
18      11.313     855.28       50703984.00
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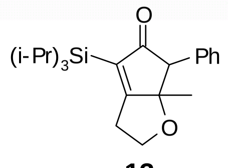

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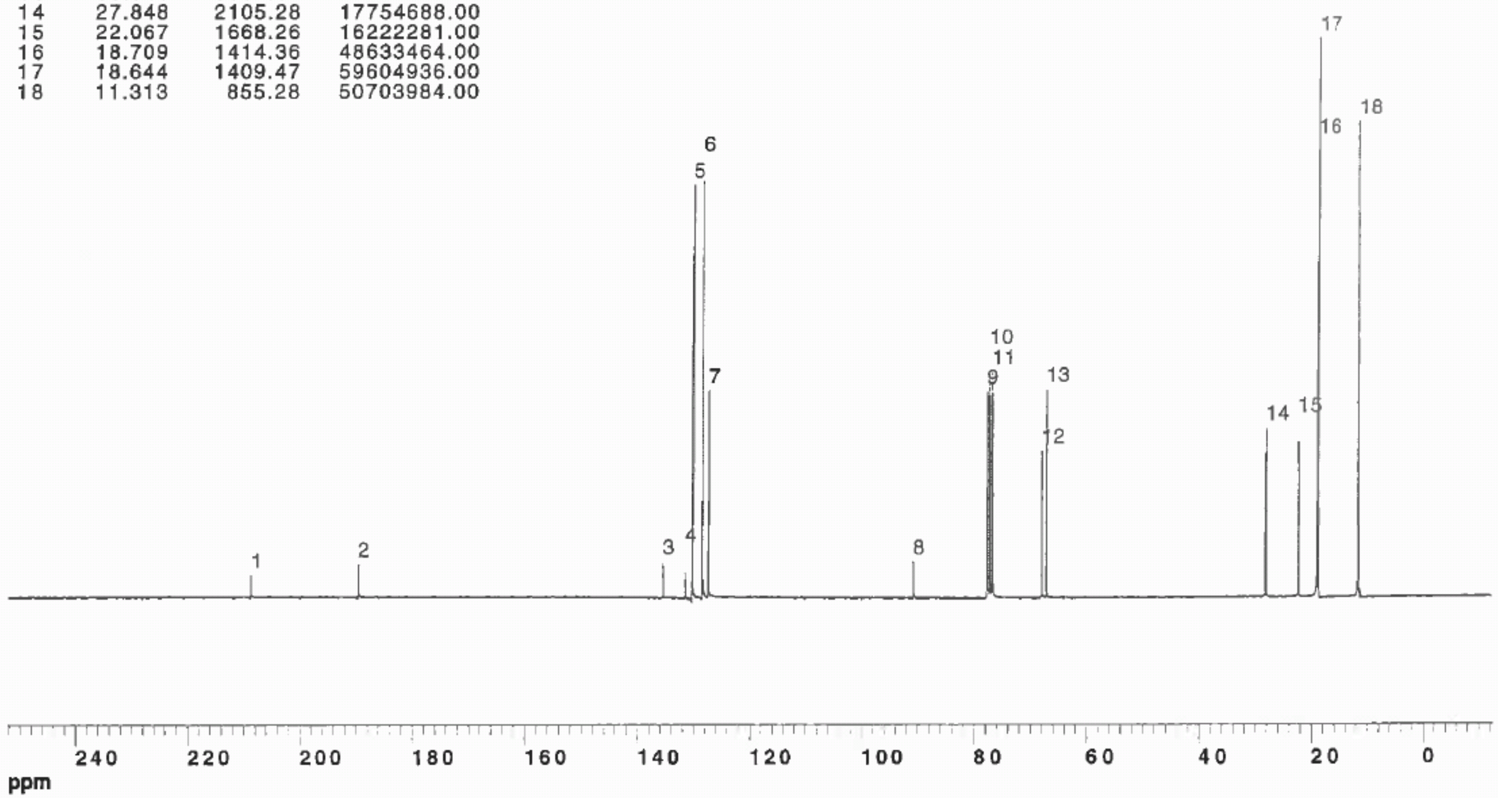
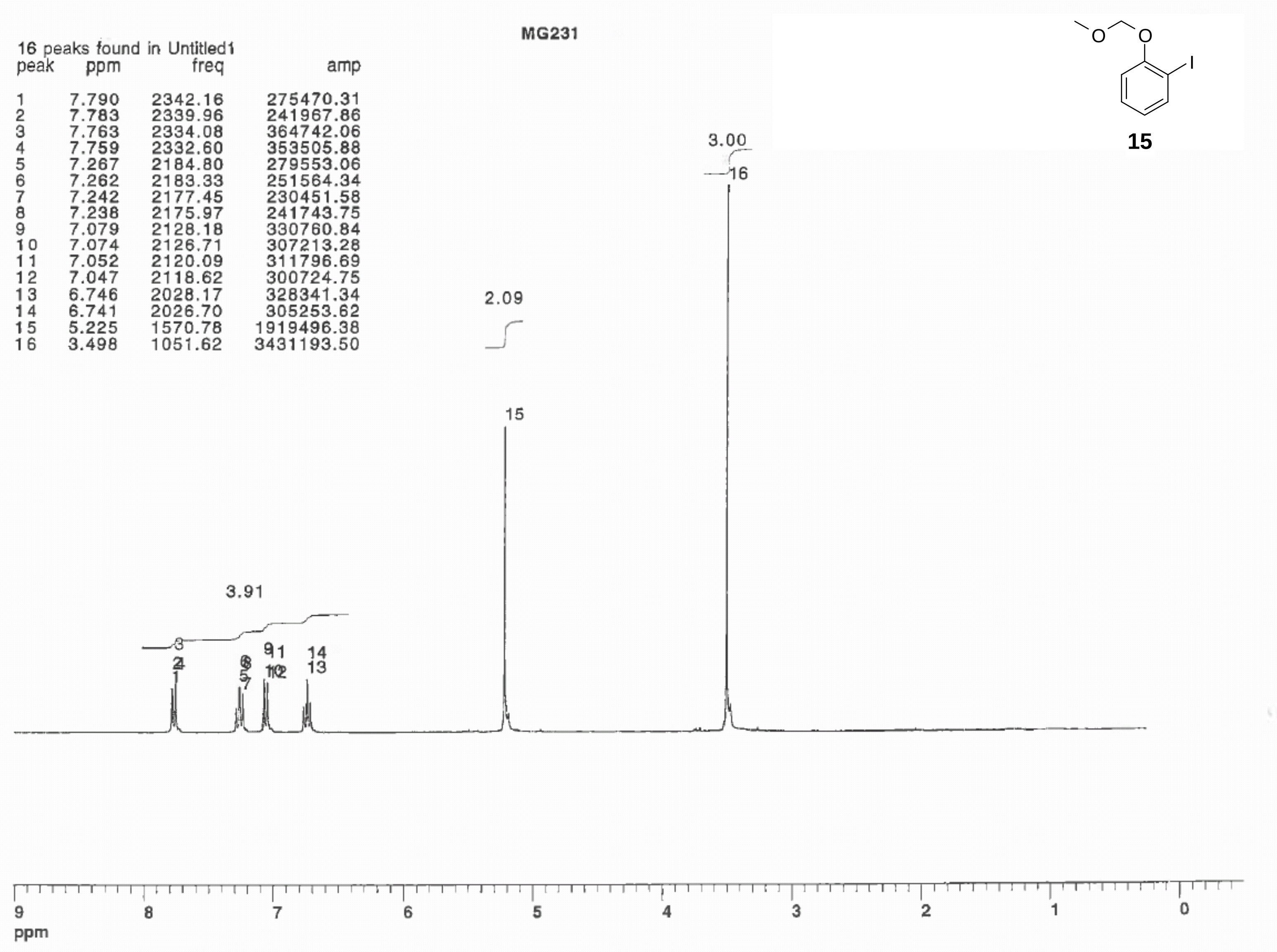




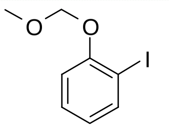
15

16 peaks found in Untitled1

peak	ppm	freq	amp
1	7.790	2342.16	275470.31
2	7.783	2339.96	241967.86
3	7.763	2334.08	364742.06
4	7.759	2332.60	353505.88
5	7.267	2184.80	279553.06
6	7.262	2183.33	251564.34
7	7.242	2177.45	230451.58
8	7.238	2175.97	241743.75
9	7.079	2128.18	330760.84
10	7.074	2126.71	307213.28
11	7.052	2120.09	311796.69
12	7.047	2118.62	300724.75
13	6.746	2028.17	328341.34
14	6.741	2026.70	305253.62
15	5.225	1570.78	1919496.38
16	3.498	1051.62	3431193.50

11 peak und in Untitled2

peak	ppm	freq	amp
1	155.863	11783.01	3182995.00
2	139.296	10530.57	15553644.00
3	129.268	9772.51	23057546.00
4	123.504	9336.72	15490252.00
5	114.800	8678.76	19238782.00
6	94.778	7165.09	18840158.00
7	87.108	6585.26	2549638.50
8	77.420	5852.84	1358299.38
9	77.000	5821.10	1388647.75
10	76.580	5789.36	1241455.25
11	56.219	4250.05	16366769.00



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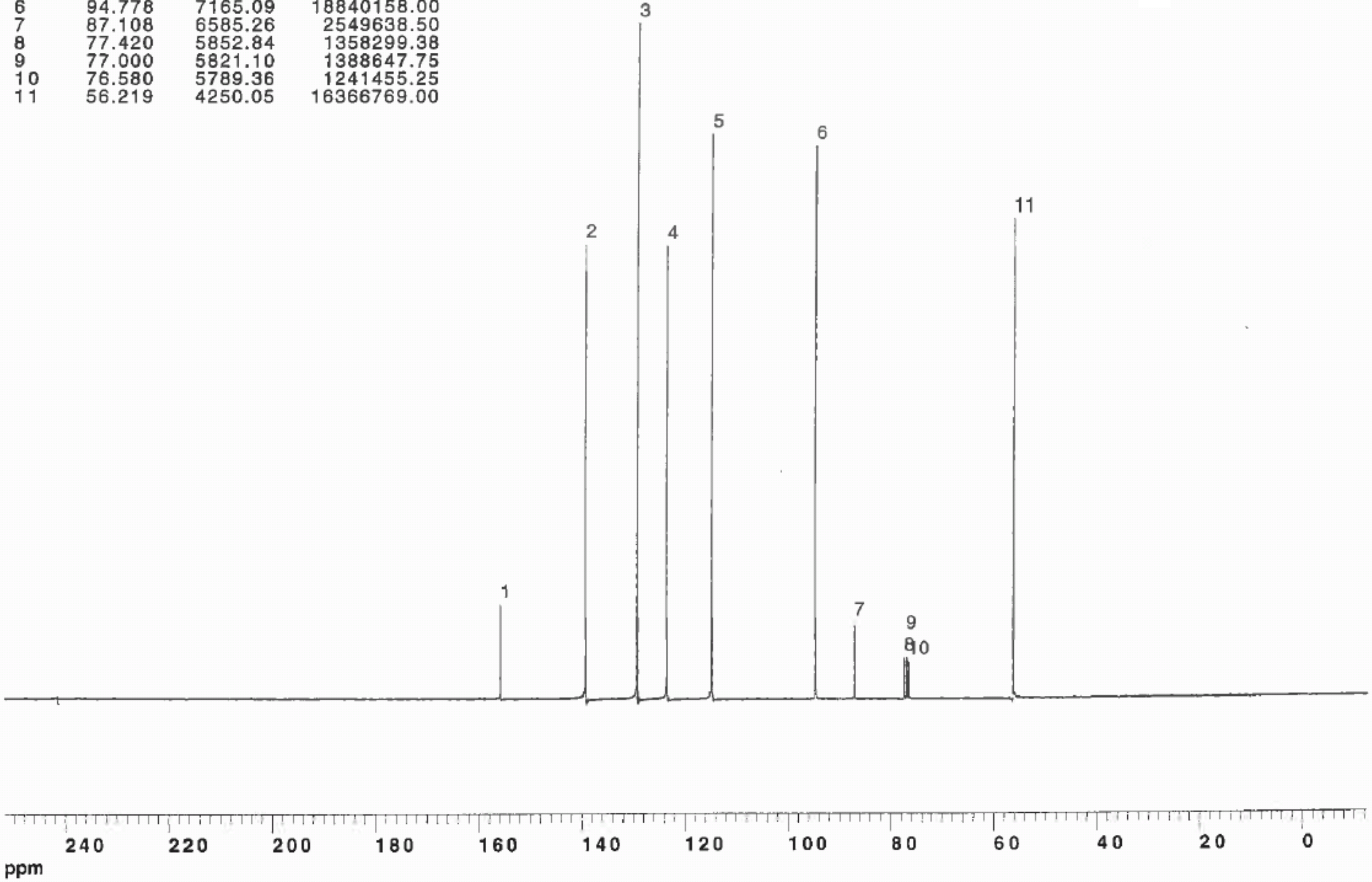

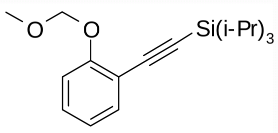

16

```
18 peaks found in Untitled1
peak    ppm       freq          amp

1      7.458    2242.16      48895.83
2      7.453    2240.69      54667.18
3      7.433    2234.80      52329.54
4      7.426    2232.60      61966.61
5      7.240    2176.71      46614.91
6      7.233    2174.50      48342.17
7      7.213    2168.62      41322.81
8      7.208    2167.15      47159.29
9      7.059    2122.29      73921.86
10     7.035    2114.94      56485.95
11     6.959    2092.14      40237.05
12     6.934    2084.79      75790.42
13     5.227    1571.52     441899.69
14     3.495    1050.89     685417.25
15     1.135     341.27    3503486.75
16     1.111     333.92     155523.91
17     1.101     330.98     177106.98
18     1.084     325.83      61953.17
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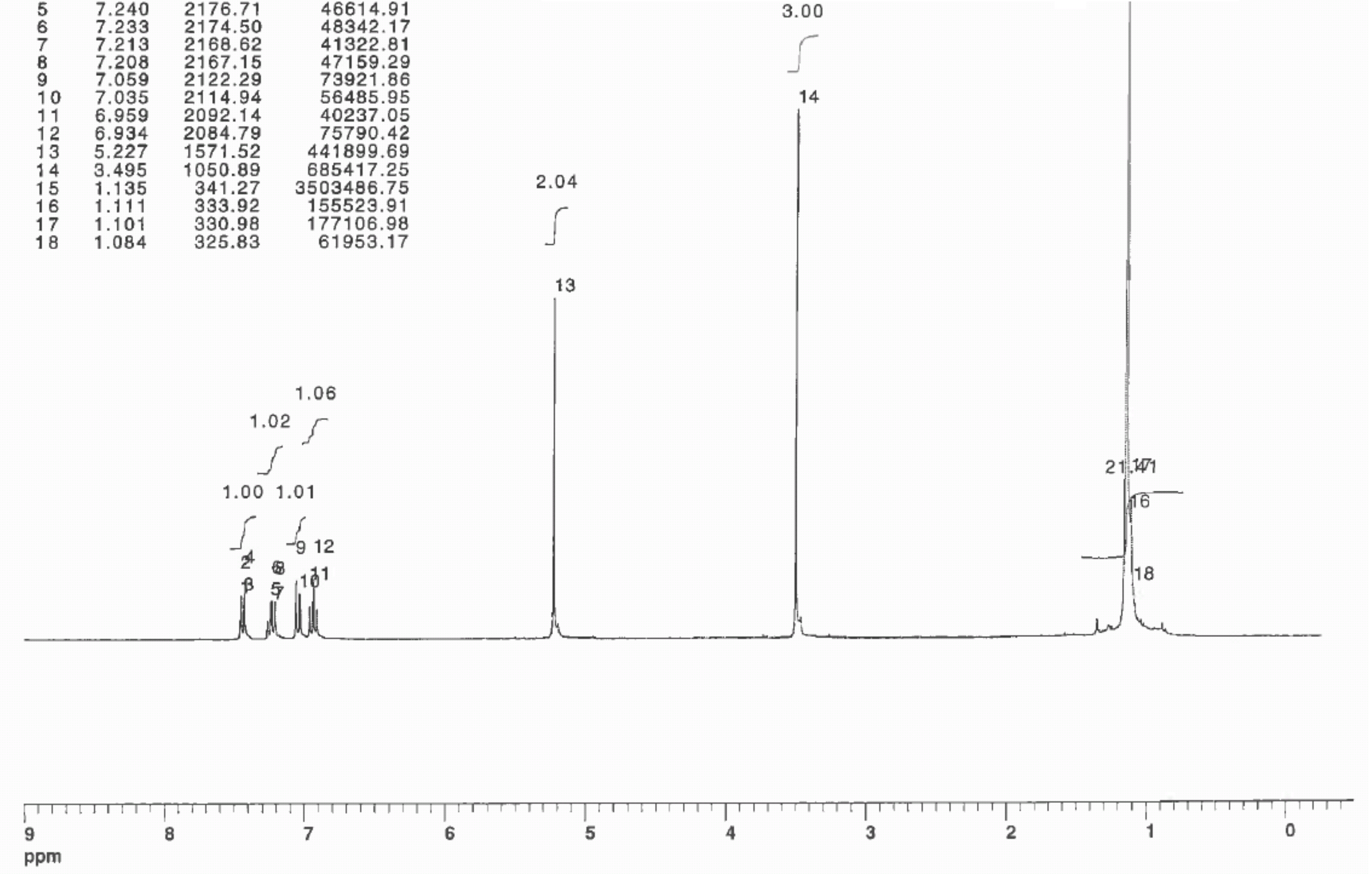
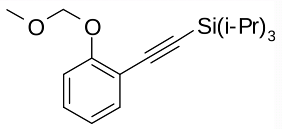
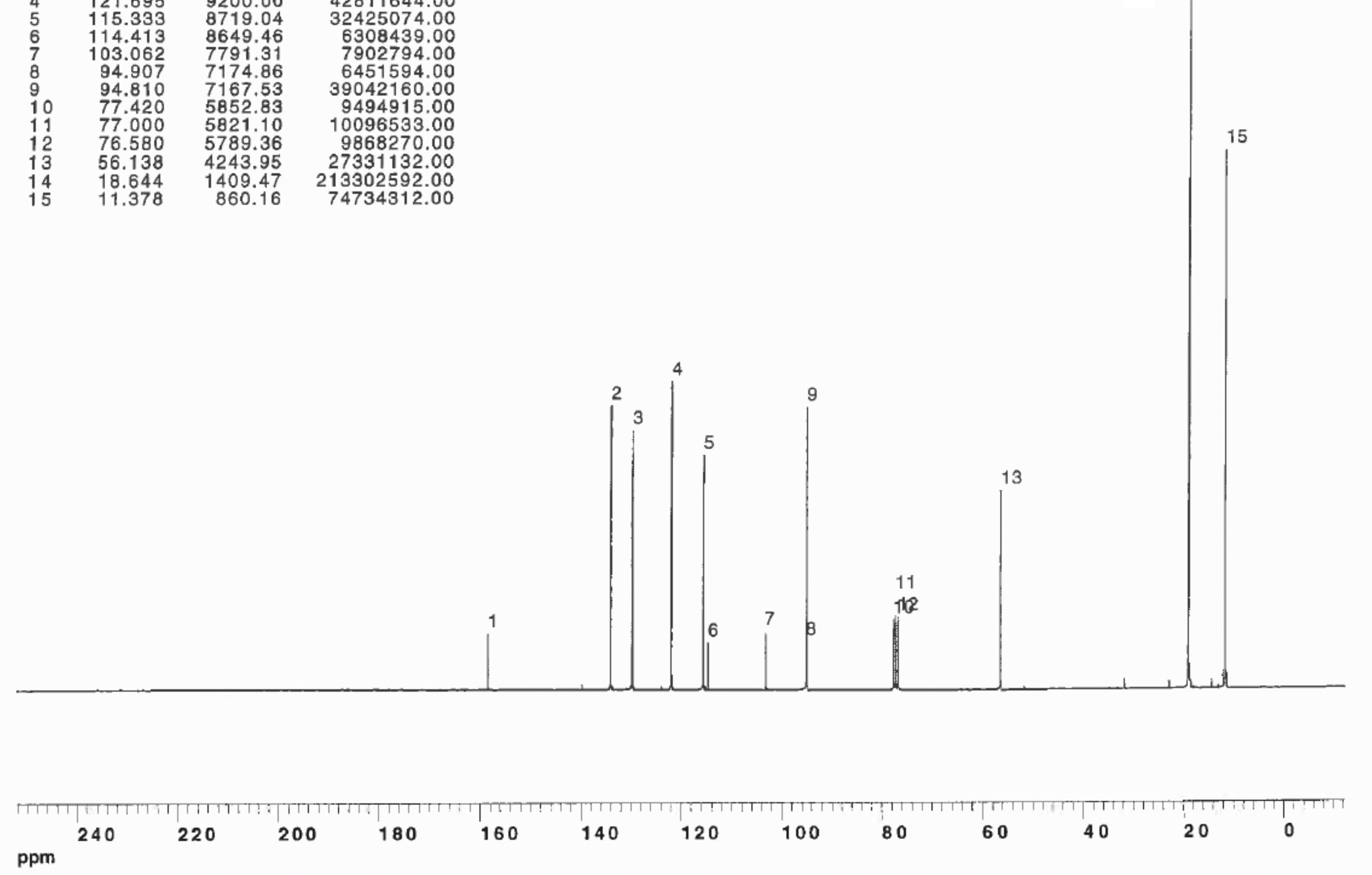


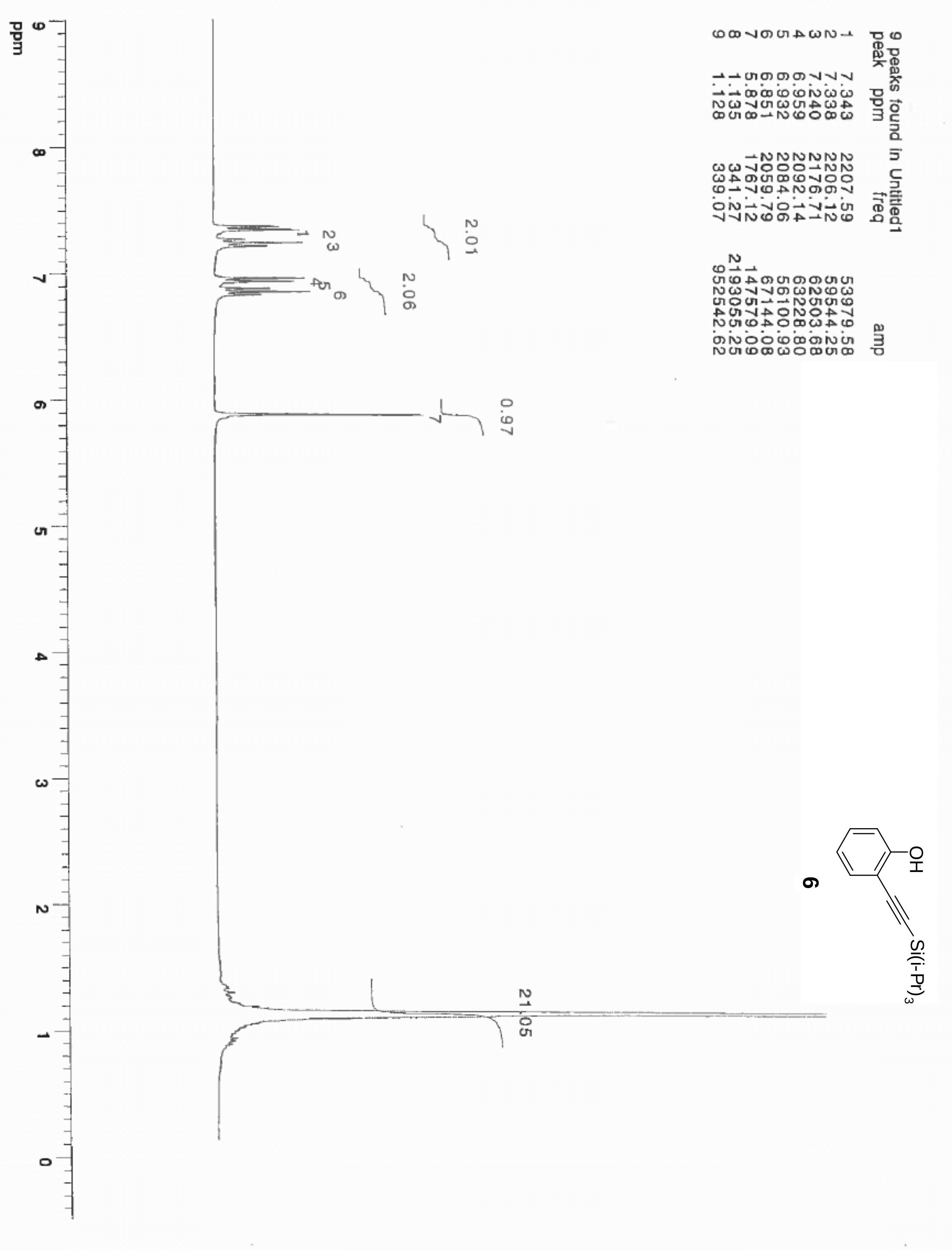


16

15 peaks found in Untitled2

peak	ppm	freq	amp
1	158.204	11960.01	7654480.00
2	133.790	10114.31	39364252.00
3	129.494	9789.60	35904936.00
4	121.695	9200.00	42811644.00
5	115.333	8719.04	32425074.00
6	114.413	8649.46	6308439.00
7	103.062	7791.31	7902794.00
8	94.907	7174.86	6451594.00
9	94.810	7167.53	39042160.00
10	77.420	5852.83	9494915.00
11	77.000	5821.10	10096533.00
12	76.580	5789.36	9868270.00
13	56.138	4243.95	27331132.00
14	18.644	1409.47	213302592.00
15	11.378	860.16	74734312.00





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9 peaks found in Untitled1
peak   ppm      freq          amp
 1    7.343   2207.59     53979.58
 2    7.338   2206.12     59544.25
 3    7.240   2176.71     62503.68
 4    6.959   2092.14     63228.80
 5    6.932   2084.06     56100.93
 6    6.851   2059.79     67144.08
 7    5.878   1767.12    147579.09
 8    1.135    341.27   2193055.25
 9    1.128    339.07    952542.62
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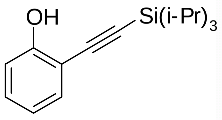
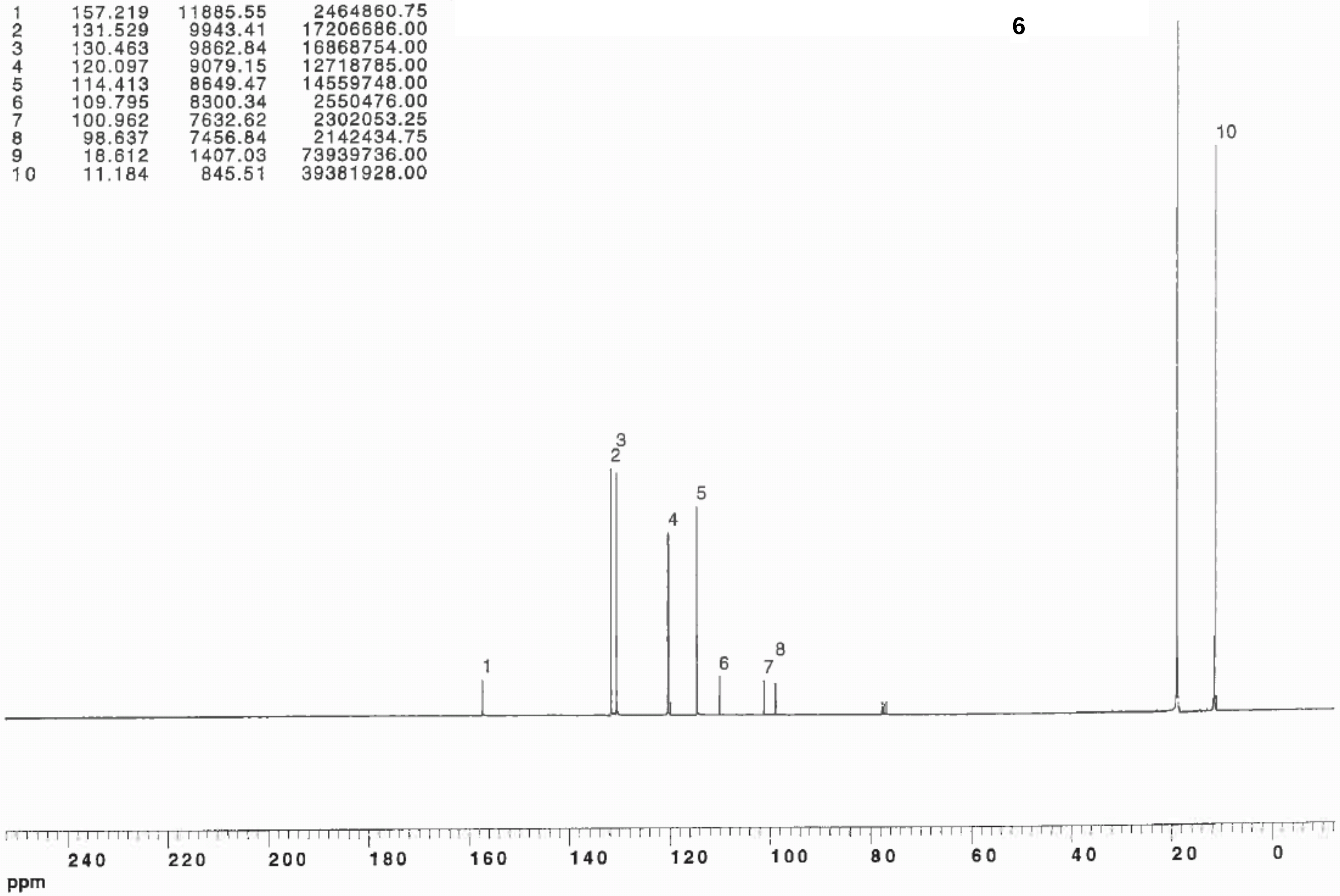
6

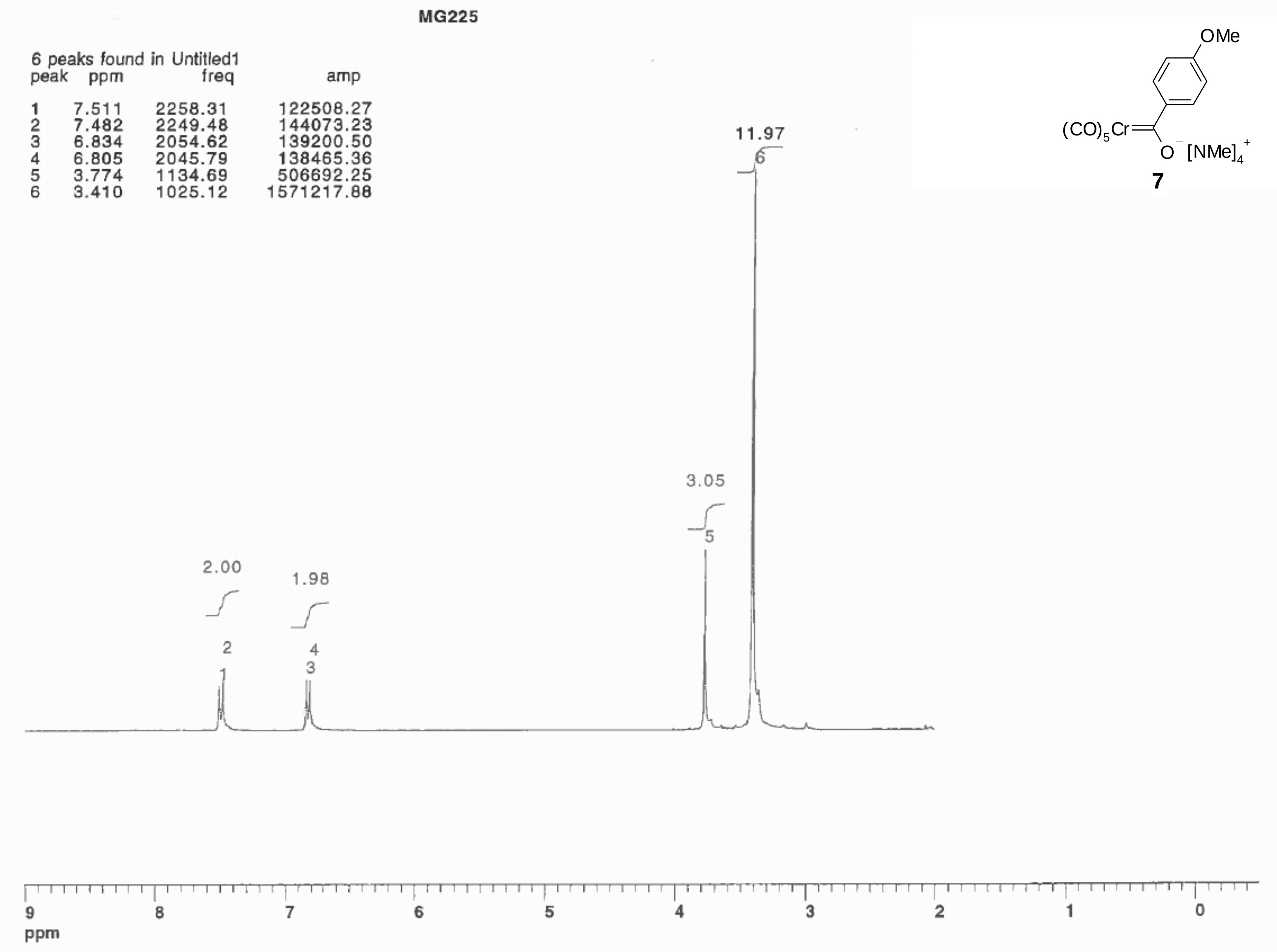


6

10 peaks found in MG242C

peak	ppm	freq	amp
1	157.219	11885.55	2464860.75
2	131.529	9943.41	17206686.00
3	130.463	9862.84	16868754.00
4	120.097	9079.15	12718785.00
5	114.413	8649.47	14559748.00
6	109.795	8300.34	2550476.00
7	100.962	7632.62	2302053.25
8	98.637	7456.84	2142434.75
9	18.612	1407.03	73939736.00
10	11.184	845.51	39381928.00





7

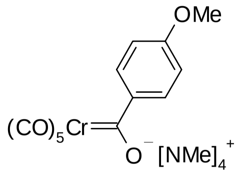
6 peaks found in Untitled1

peak	ppm	freq	amp
1	7.511	2258.31	122508.27
2	7.482	2249.48	144073.23
3	6.834	2054.62	139200.50
4	6.805	2045.79	138465.36
5	3.774	1134.69	506692.25
6	3.410	1025.12	1571217.88

MG225

18 peaks found in Untitled2

peak	ppm	freq	amp
1	228.846	17300.44	990607.88
2	224.551	16975.74	3417164.75
3	206.272	15593.90	5985397.00
4	160.495	12133.21	926142.44
5	148.869	11254.30	397225.78
6	127.652	9650.30	6716734.00
7	112.909	8535.80	7992579.50
8	55.991	4232.82	3307146.50
9	55.942	4229.16	4130527.50
10	55.894	4225.49	4172782.25
11	55.442	4191.31	4054774.75
12	30.559	2310.21	607660.81
13	30.317	2291.90	1738494.62
14	30.058	2272.37	3710433.00
15	29.800	2252.84	4639522.00
16	29.542	2233.31	4027897.50
17	29.283	2213.78	1972101.50
18	29.025	2194.24	629920.12



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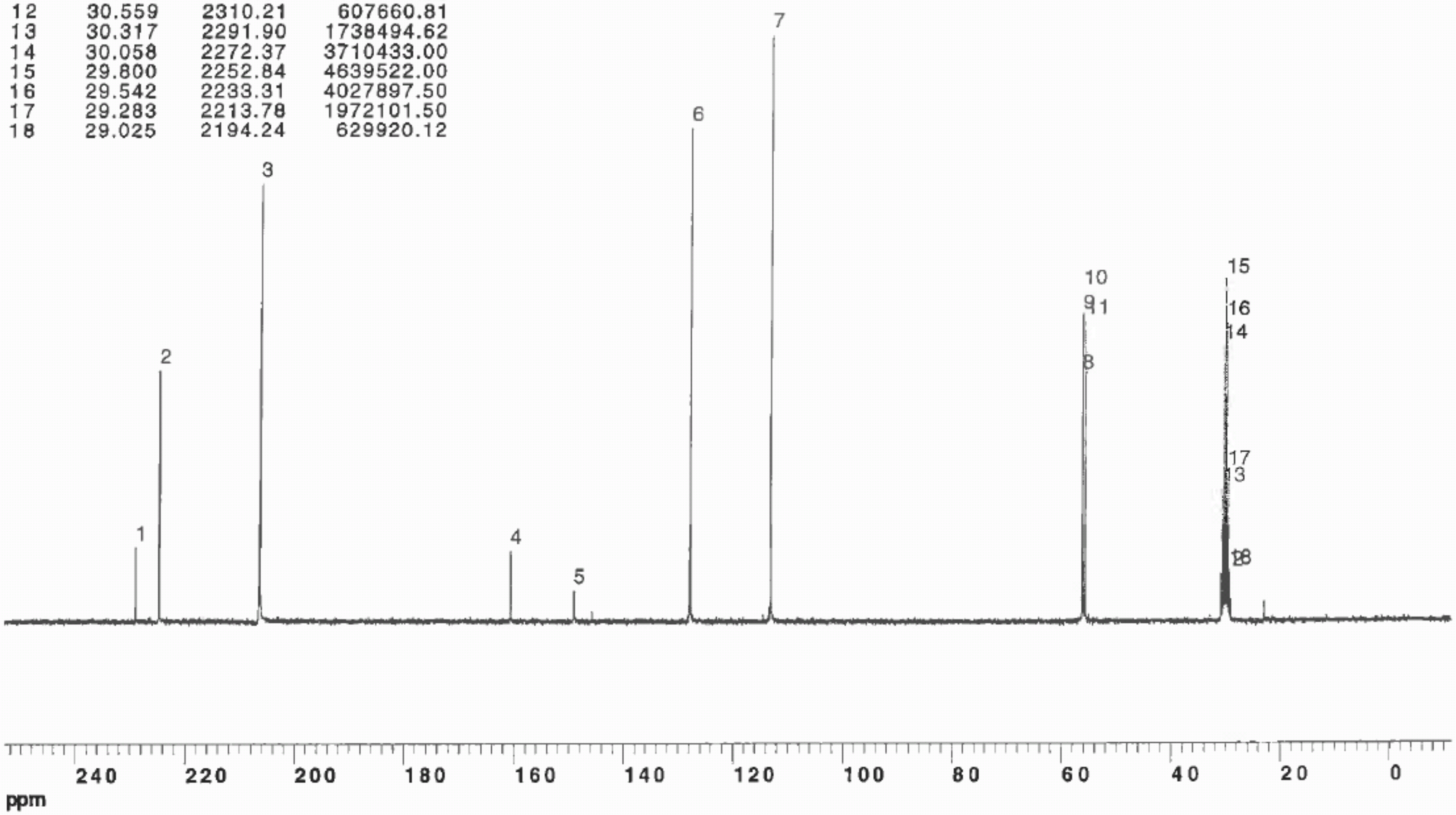
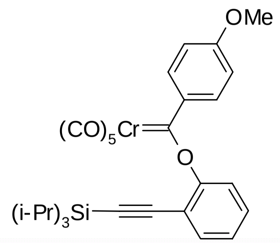


7 peaks found in Untitled1

peak	ppm	freq	amp
1	8.001	2405.40	56889.11
2	7.971	2396.58	66459.20
3	7.240	2176.71	150181.52
4	6.920	2080.38	64845.24
5	6.890	2071.55	69777.49
6	3.892	1170.01	234538.61
7	0.939	282.44	1075606.12



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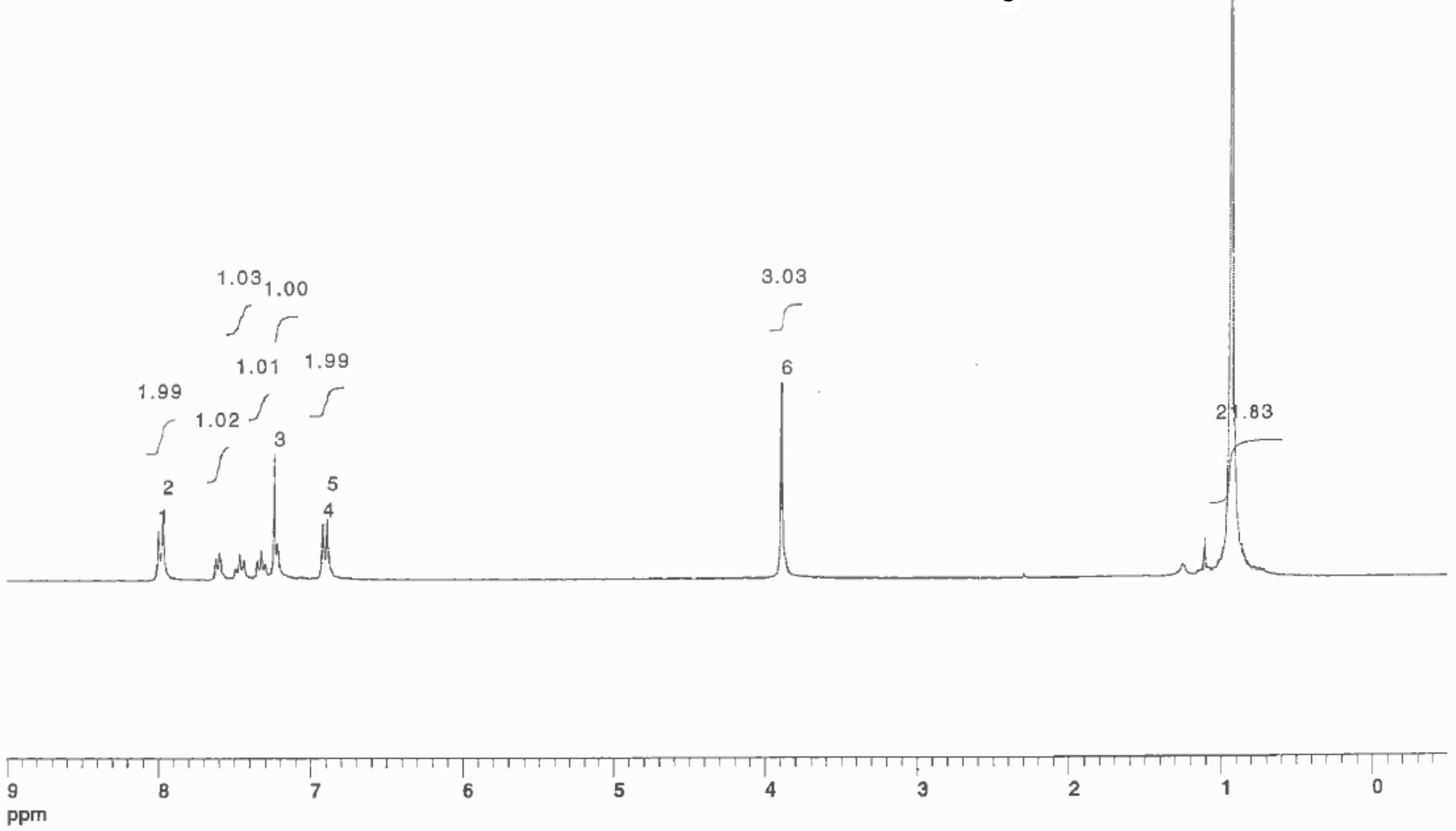
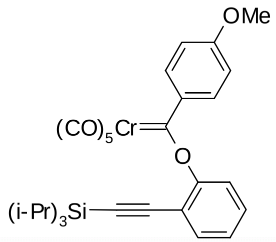


26 peaks found in MG244C

peak	ppm	freq	amp
1	224.407	16964.89	170240.78
2	215.979	16327.69	584564.38
3	163.888	12389.70	148541.33
4	159.786	12079.64	100844.03
5	146.562	11079.88	124242.59
6	134.758	10187.55	768472.69
7	131.061	9908.01	1329391.62
8	129.640	9800.59	777224.31
9	126.959	9597.95	759658.94
10	122.680	9274.46	739579.38
11	118.288	8942.43	107910.91
12	113.008	8543.26	1666422.00
13	101.172	7648.49	110981.83
14	97.701	7386.04	77973.15
15	78.227	5913.87	114694.85
16	77.420	5852.83	886729.12
17	77.210	5836.96	83366.59
18	77.129	5830.86	104271.48
19	77.000	5821.10	954913.62
20	76.580	5789.36	995138.69
21	55.621	4204.88	591751.62
22	18.693	1413.14	107778.61
23	18.628	1408.25	204793.56
24	18.483	1397.27	3558011.00
25	11.281	852.83	87717.19
26	11.168	844.29	1519176.25



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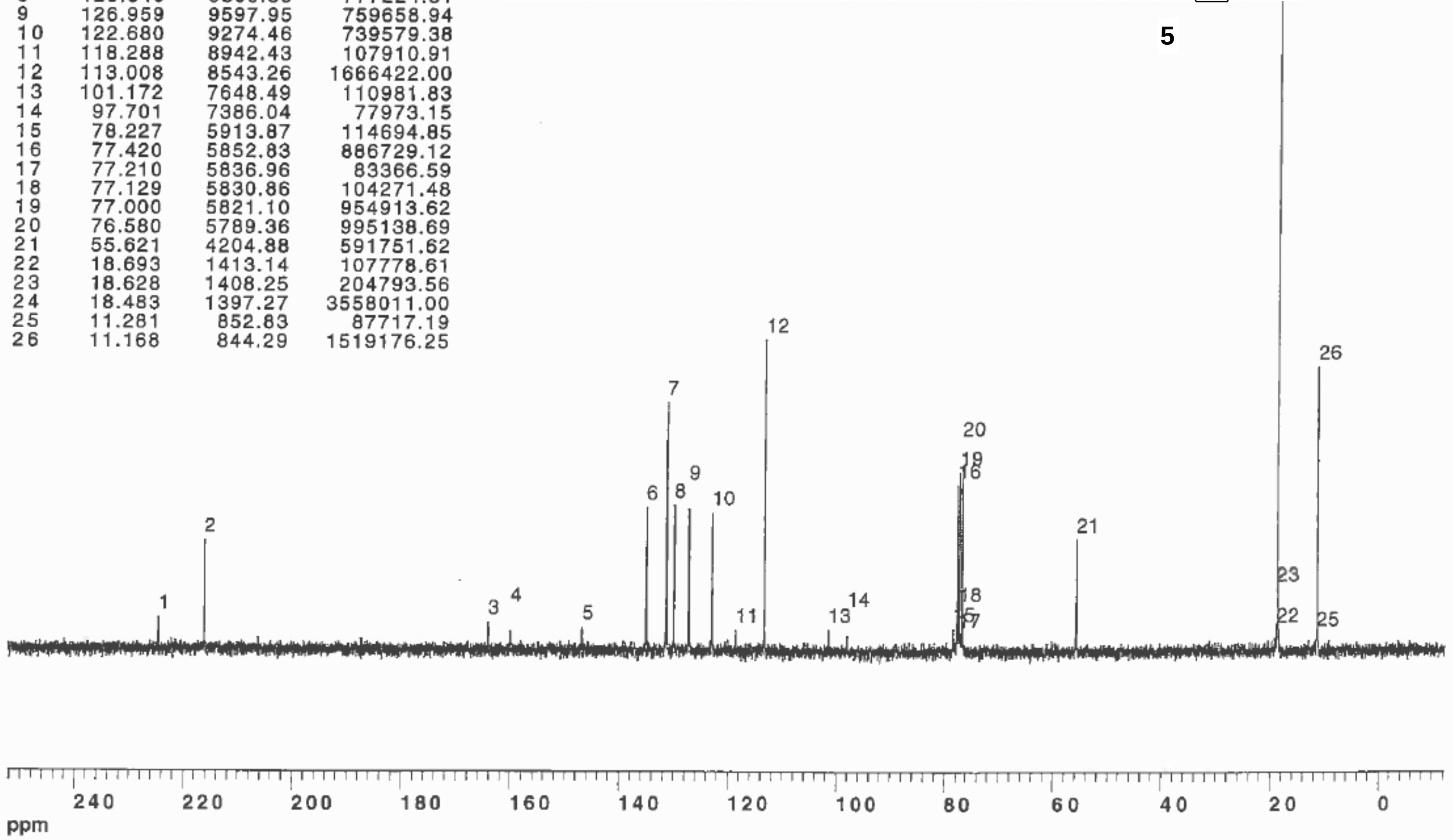

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solvent      CDCl3  dn               H1
file           exp  dpwr             30
   ACQUISITION      dof               0
sfrq        50.301  dm              yyy
tn             C13  dmm               w
at           1.498  dmf            9900
np           16880       PROCESSING
sw          5634.0  lb             1.00
fb            3200  wtfile
bs              16  proc             ft
tpwr            55  fn         not used
pw             8.7
d1           6.000  werr
tof        12099.3  wexp
nt           16384  wbs
ct            9212  wnt
alock            n
gain            20
     FLAGS
il               n
in               n
dp               y
     DISPLAY
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wp          5634.0
vs              34
sc               0
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nm  no  ph
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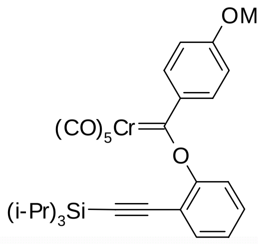

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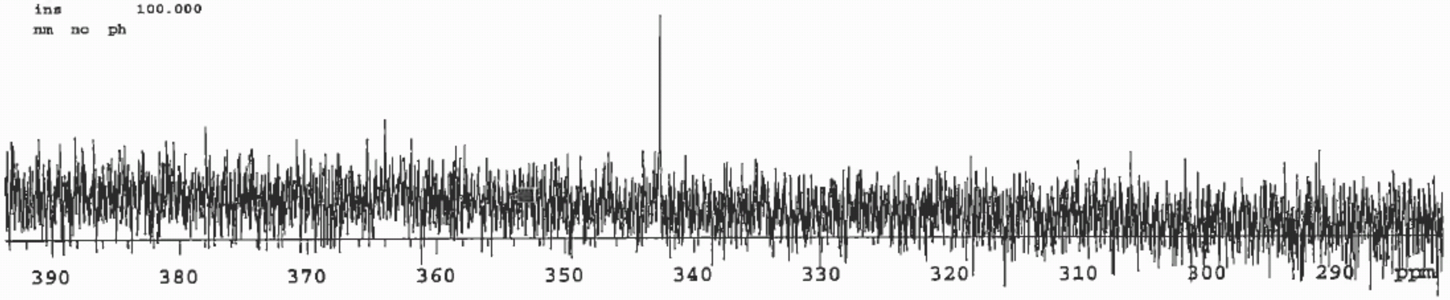
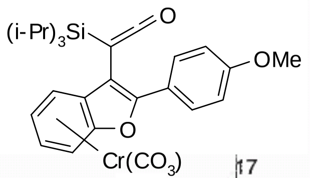
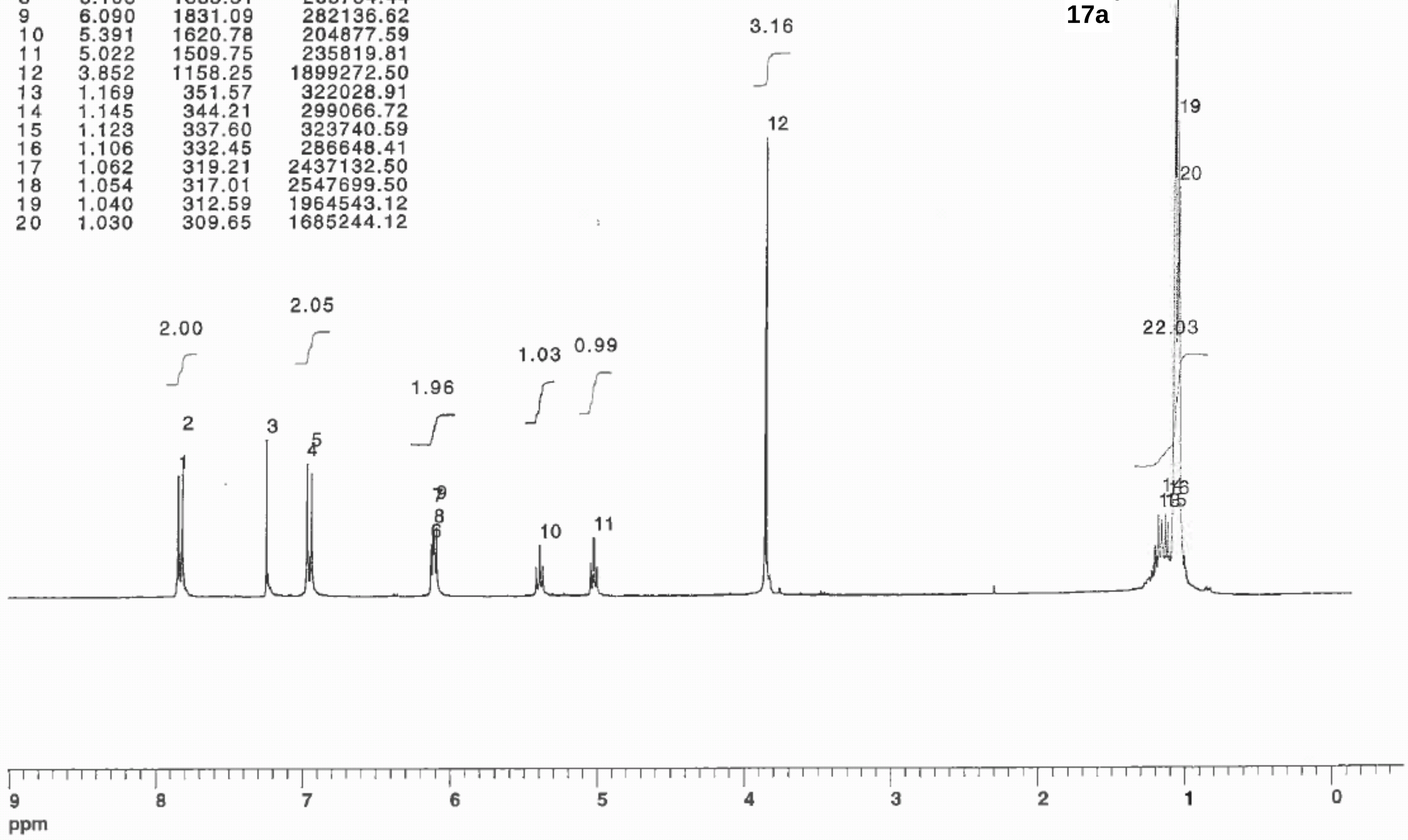


20 peaks found in Untitled1

peak	ppm	freq	amp
1	7.842	2357.61	498670.22
2	7.812	2348.78	575766.25
3	7.240	2176.71	648117.94
4	6.966	2094.35	547762.62
5	6.937	2085.53	502903.25
6	6.125	1841.39	208326.58
7	6.112	1837.71	269987.91
8	6.105	1835.51	268754.44
9	6.090	1831.09	282136.62
10	5.391	1620.78	204877.59
11	5.022	1509.75	235819.81
12	3.852	1158.25	1899272.50
13	1.169	351.57	322028.91
14	1.145	344.21	299066.72
15	1.123	337.60	323740.59
16	1.106	332.45	286648.41
17	1.062	319.21	2437132.50
18	1.054	317.01	2547699.50
19	1.040	312.59	1964543.12
20	1.030	309.65	1685244.12

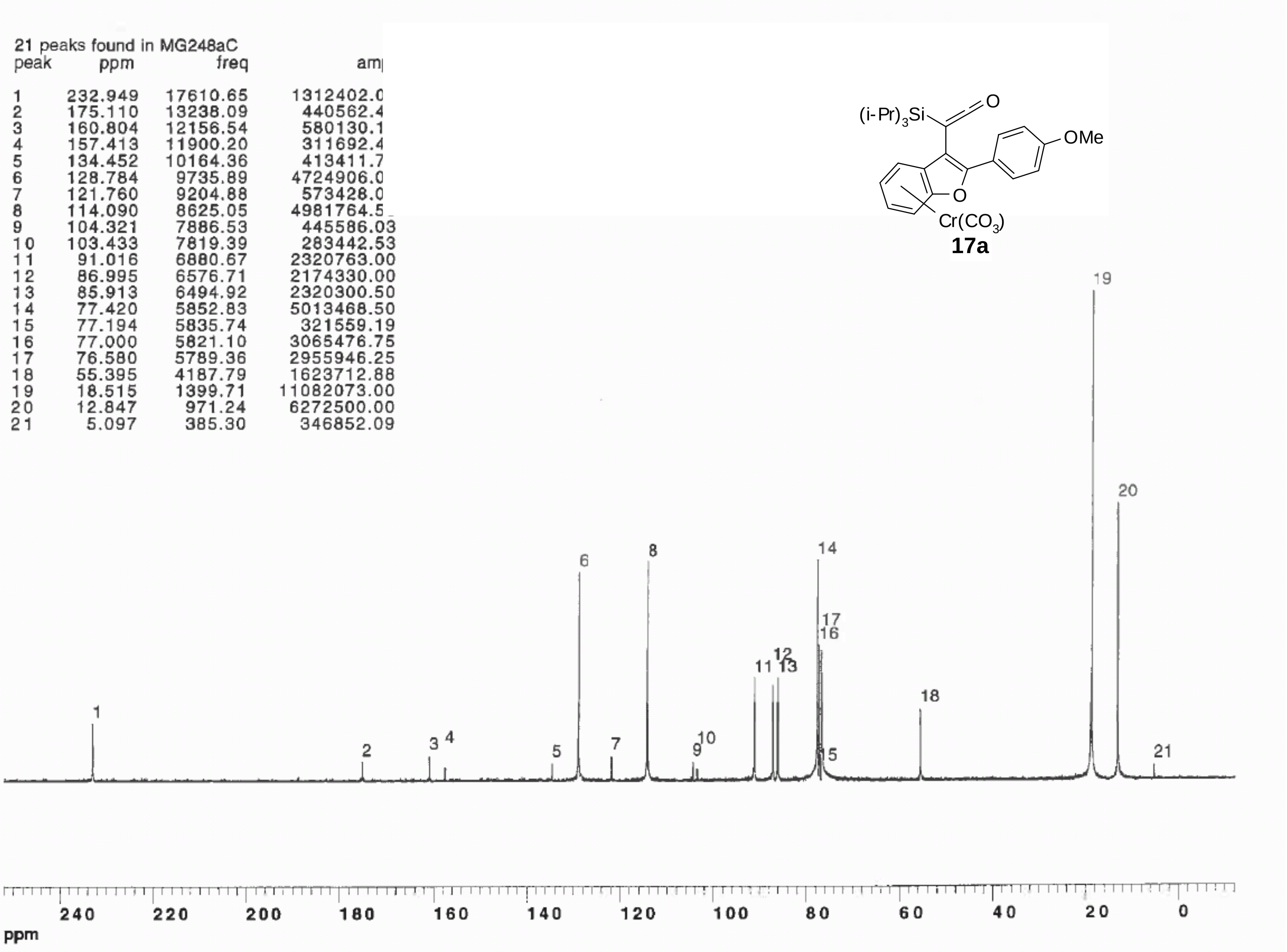


17a



21 peaks found in MG248aC

peak	ppm	freq	am
1	232.949	17610.65	1312402.0
2	175.110	13238.09	440562.4
3	160.804	12156.54	580130.1
4	157.413	11900.20	311692.4
5	134.452	10164.36	413411.7
6	128.784	9735.89	4724906.0
7	121.760	9204.88	573428.0
8	114.090	8625.05	4981764.5
9	104.321	7886.53	445586.03
10	103.433	7819.39	283442.53
11	91.016	6880.67	2320763.00
12	86.995	6576.71	2174330.00
13	85.913	6494.92	2320300.50
14	77.420	5852.83	5013468.50
15	77.194	5835.74	321559.19
16	77.000	5821.10	3065476.75
17	76.580	5789.36	2955946.25
18	55.395	4187.79	1623712.88
19	18.515	1399.71	11082073.00
20	12.847	971.24	6272500.00
21	5.097	385.30	346852.09



17a

```
10 peaks found in Untitled1
peak    ppm      freq          amp

1      7.240   2176.71     941606.44
2      6.372   1915.66     306439.47
3      6.347   1908.31     306019.38
4      5.222   1570.05     291116.53
5      5.198   1562.69     310794.47
6      3.757   1129.57    1074130.12
7      1.189    357.45     252643.36
8      1.069    321.42    2929287.50
9      1.047    314.80    2224273.75
10     1.010    303.77     306899.06
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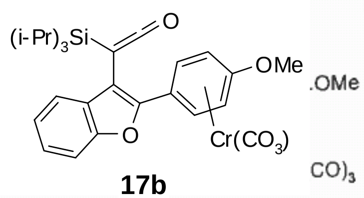
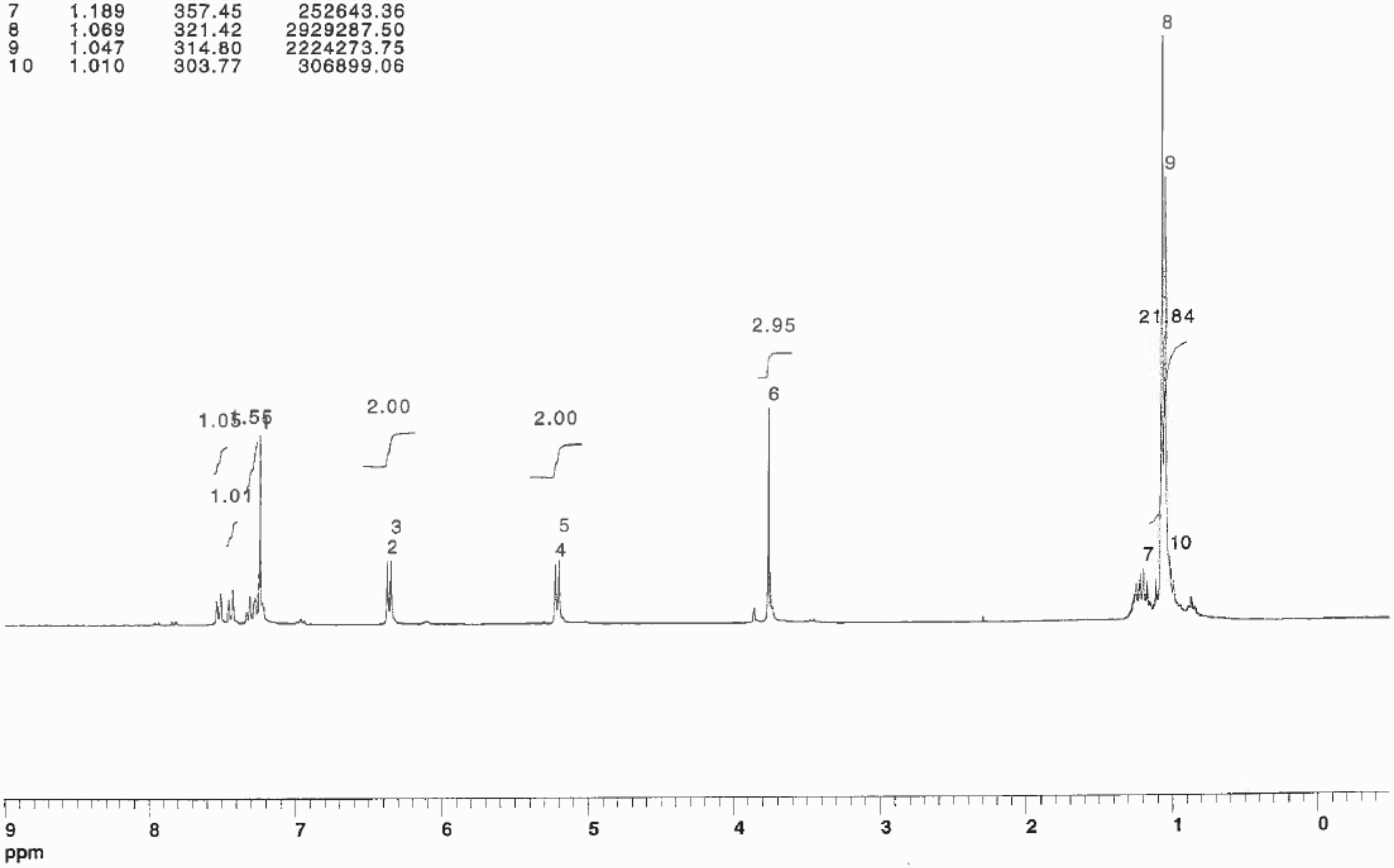


17b



```
22 peaks found in MG248bC
peak      ppm        freq          amp

 1     232.061   17543.51    2822338.75
 2     175.368   13257.62     630648.88
 3     153.780   11625.54     727547.50
 4     148.096   11195.85     531578.19
 5     142.428   10767.38    1129790.88
 6     130.851    9892.14     969544.75
 7     125.490    9486.87    3543109.25
 8     123.262    9318.41    3396541.75
 9     120.242    9090.14    3520109.25
10     111.393    8421.19    3653521.25
11     104.935    7932.91     636534.69
12      92.308    6978.32    7269300.00
13      91.403    6909.96    1107040.75
14      77.420    5852.83   16915038.00
15      77.194    5835.74     725228.88
16      77.000    5821.10   17666302.00
17      76.580    5789.36   19559684.00
18      55.783    4217.09    3032207.50
19      18.547    1402.15   15394476.00
20      12.896     974.90    7402977.50
21      12.734     962.70     672510.38
22       5.743     434.13     606736.69
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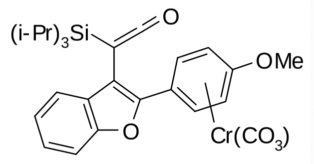

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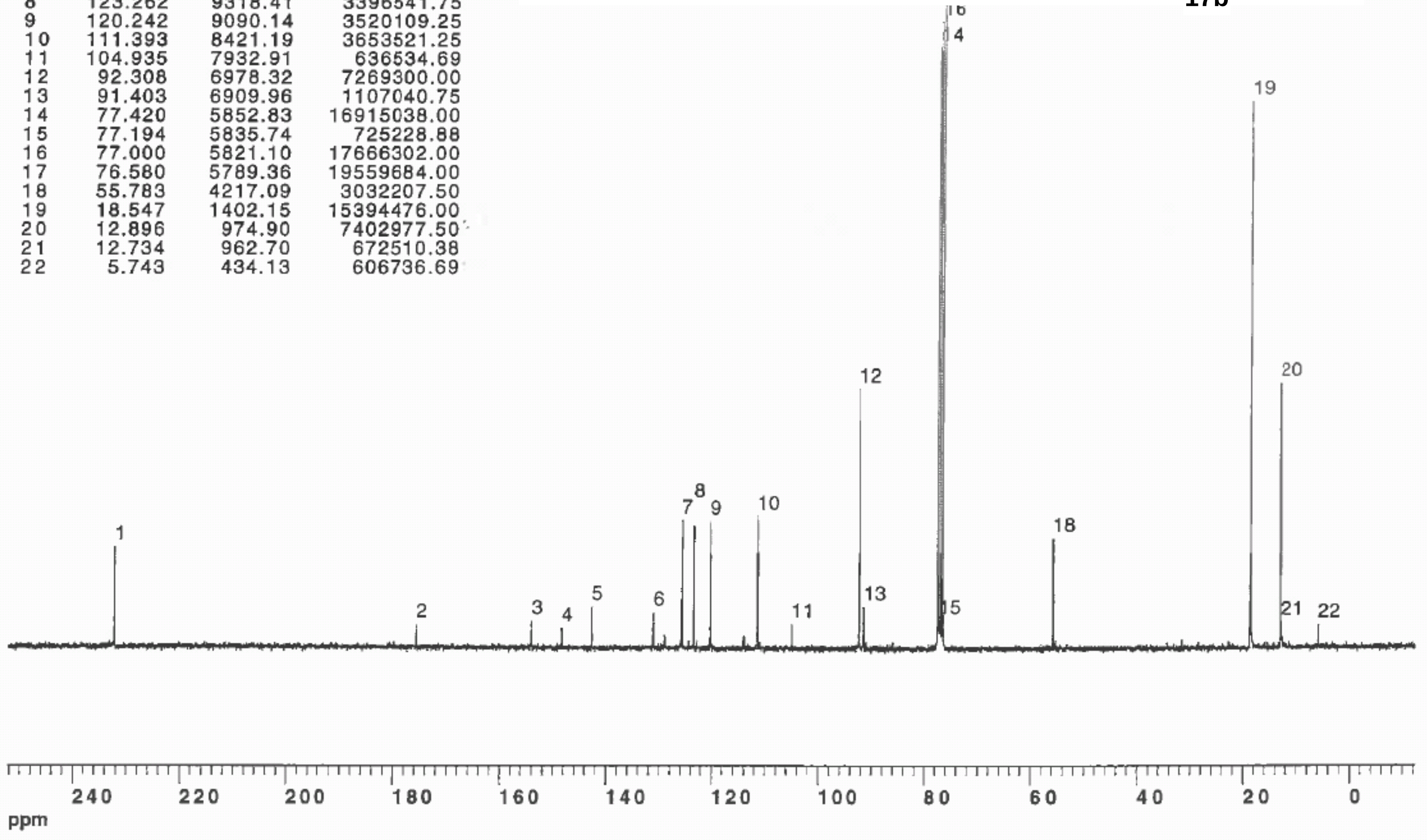

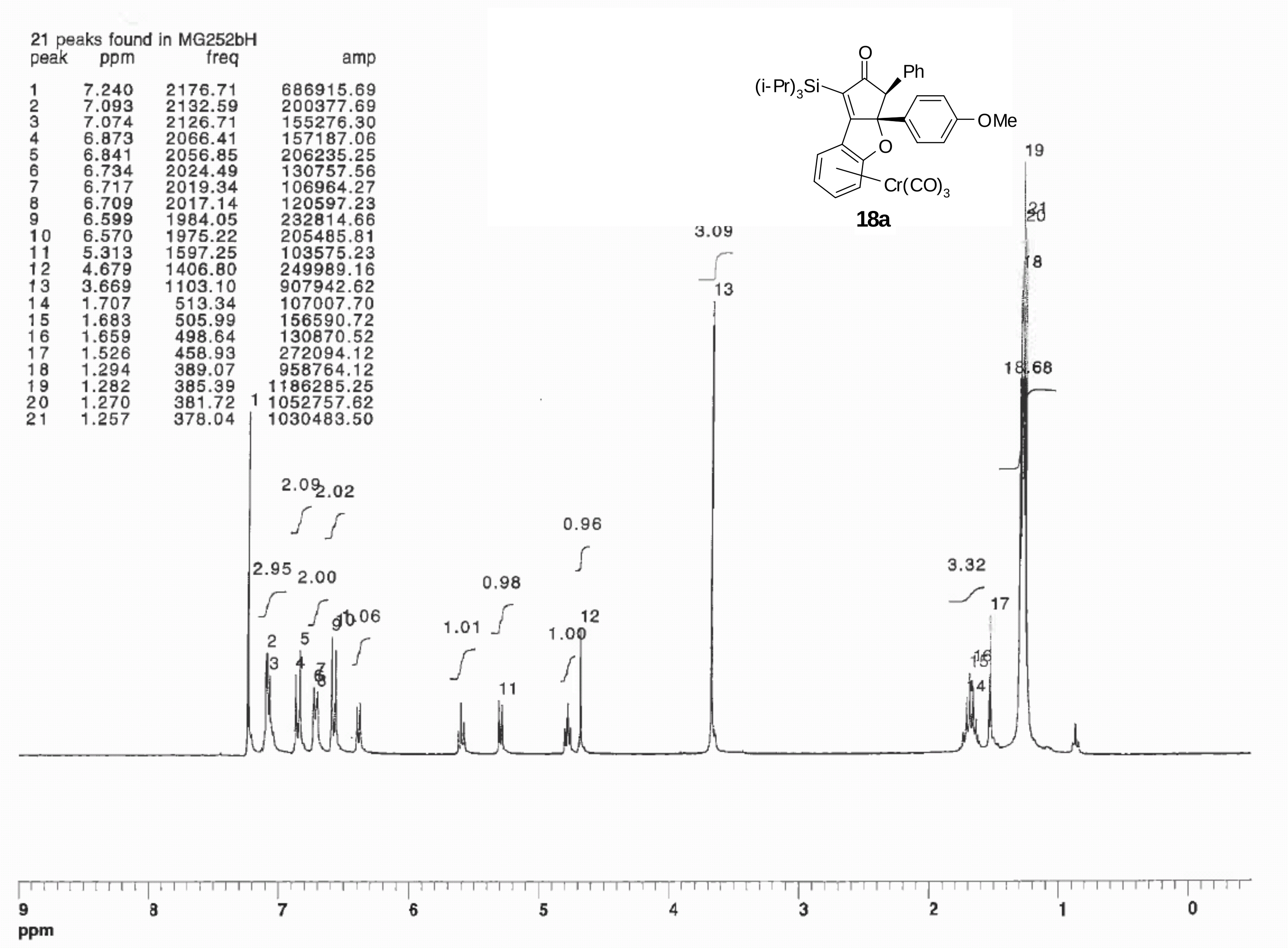

```
21 peaks found in MG252bH
peak    ppm      freq        amp

 1     7.240   2176.71    686915.69
 2     7.093   2132.59    200377.69
 3     7.074   2126.71    155276.30
 4     6.873   2066.41    157187.06
 5     6.841   2056.85    206235.25
 6     6.734   2024.49    130757.56
 7     6.717   2019.34    106964.27
 8     6.709   2017.14    120597.23
 9     6.599   1984.05    232814.66
10     6.570   1975.22    205485.81
11     5.313   1597.25    103575.23
12     4.679   1406.80    249989.16
13     3.669   1103.10    907942.62
14     1.707    513.34    107007.70
15     1.683    505.99    156590.72
16     1.659    498.64    130870.52
17     1.526    458.93    272094.12
18     1.294    389.07    958764.12
19     1.282    385.39   1186285.25
20     1.270    381.72   1052757.62
21     1.257    378.04   1030483.50
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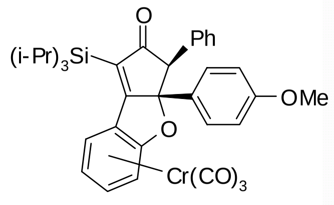
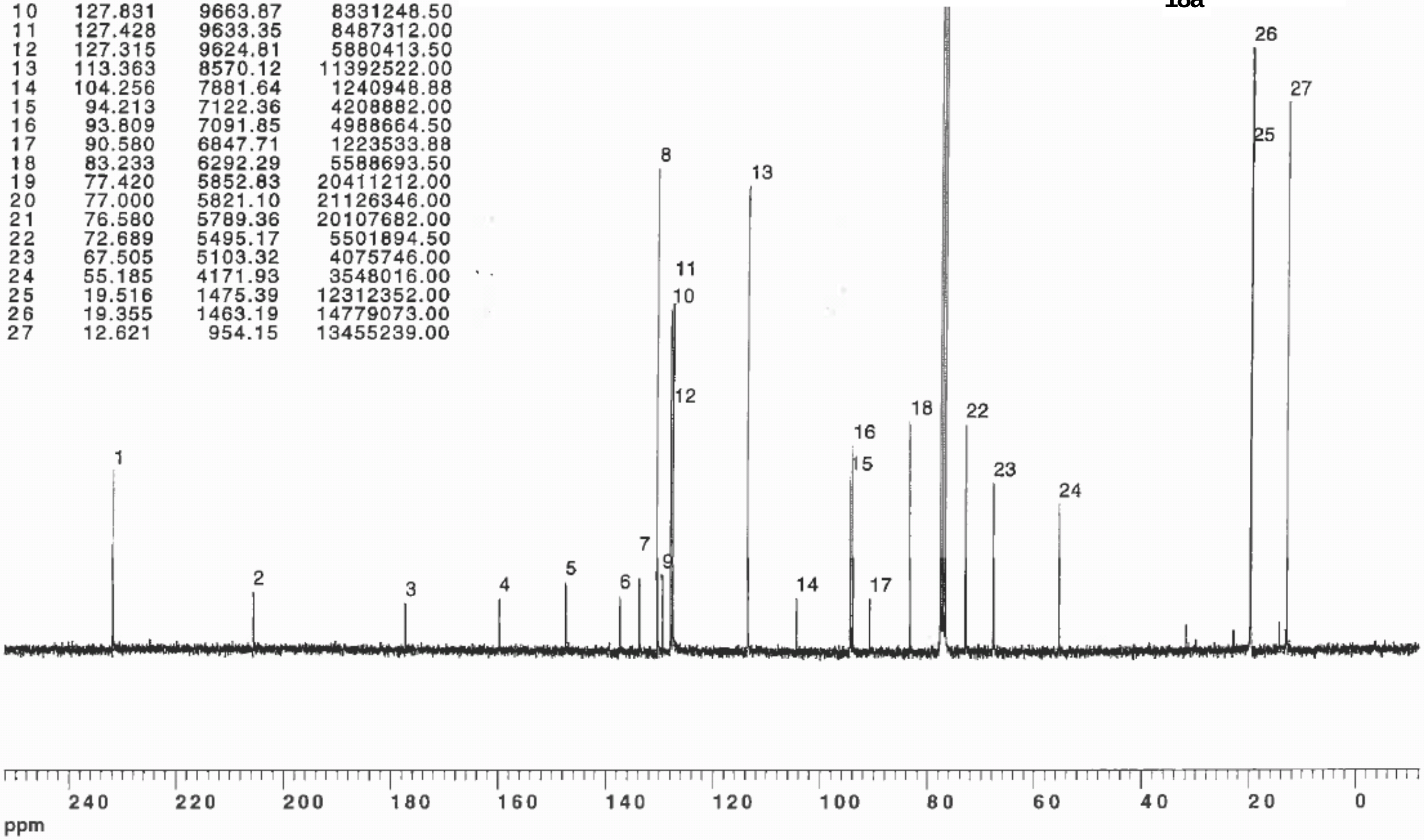
18a

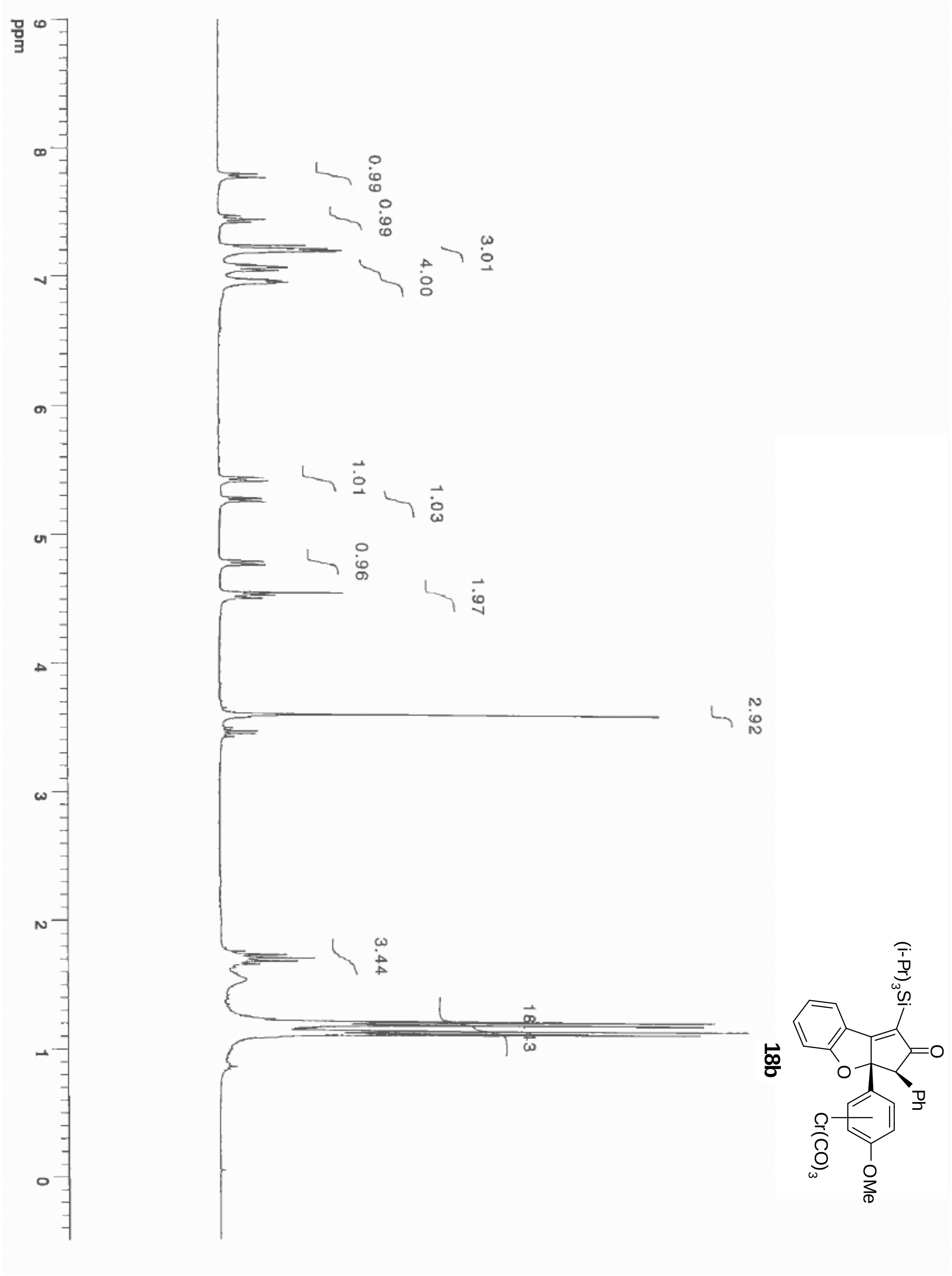


18a

27 peaks found in MG252bC2

peak	ppm	freq	amp
1	231.770	17521.53	4382750.50
2	205.548	15539.11	1402242.12
3	177.225	13398.00	1140419.00
4	159.657	12069.87	1244903.75
5	147.337	11138.48	1652029.12
6	137.293	10379.20	1314857.38
7	133.644	10103.32	1717103.88
8	130.253	9846.97	11830979.00
9	129.349	9778.61	1813604.12
10	127.831	9663.87	8331248.50
11	127.428	9633.35	8487312.00
12	127.315	9624.81	5880413.50
13	113.363	8570.12	11392522.00
14	104.256	7881.64	1240948.88
15	94.213	7122.36	4208882.00
16	93.809	7091.85	4988664.50
17	90.580	6847.71	1223533.88
18	83.233	6292.29	5588693.50
19	77.420	5852.83	20411212.00
20	77.000	5821.10	21126346.00
21	76.580	5789.36	20107682.00
22	72.689	5495.17	5501894.50
23	67.505	5103.32	4075746.00
24	55.185	4171.93	3548016.00
25	19.516	1475.39	12312352.00
26	19.355	1463.19	14779073.00
27	12.621	954.15	13455239.00

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6
5
4
3
2
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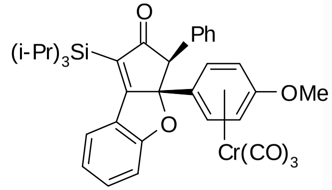
0.99
0.99
3.01
4.00
1.01
1.03
0.96
1.97
2.92
3.44
18.43
(i-Pr)$_3$Si
O
Ph
O
Cr(CO)$_3$
OMe

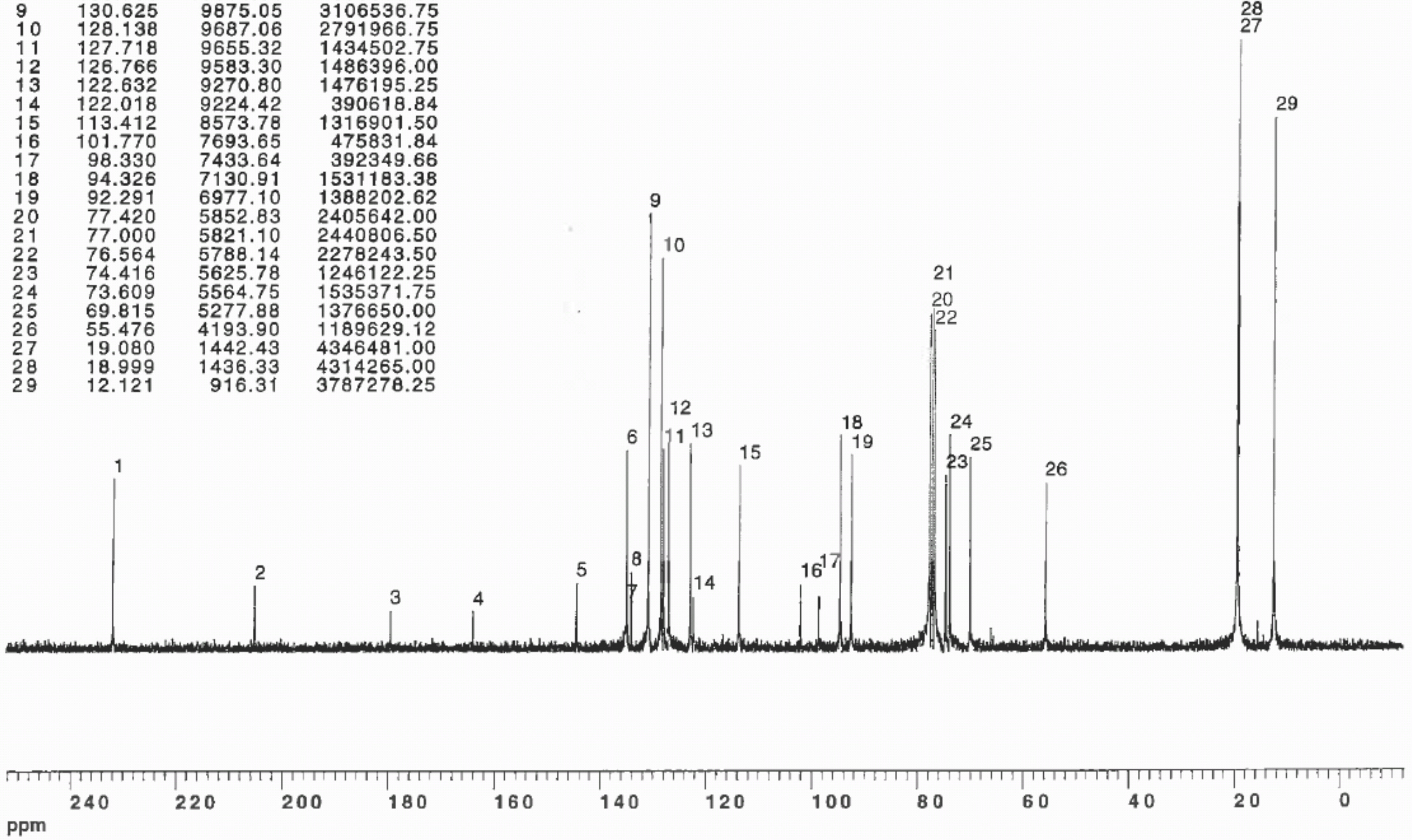
18b

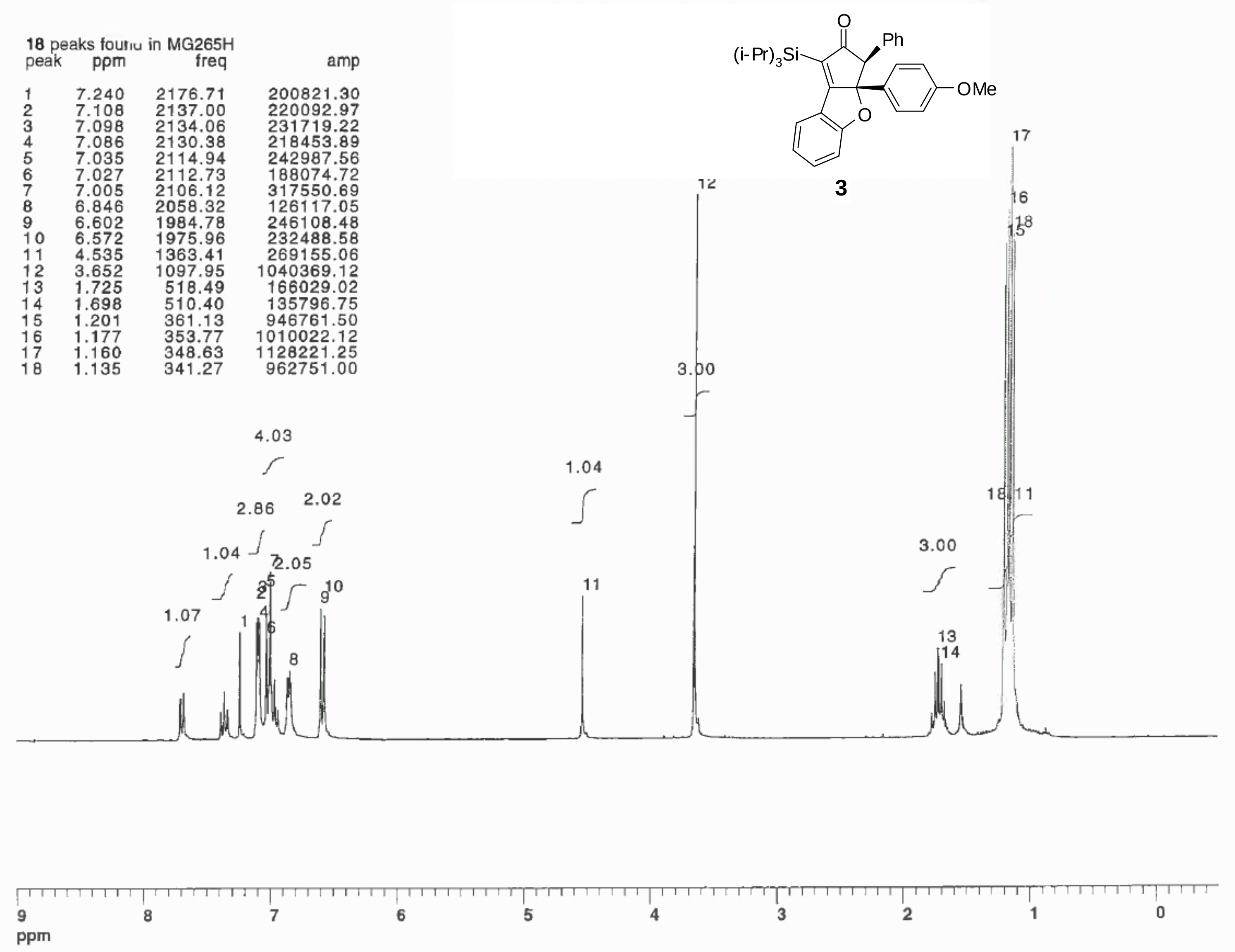
29 peaks found in MG252aC3

peak	ppm	freq	amp
1	231.674	17514.21	1226138.00
2	204.934	15492.73	465840.84
3	179.292	13554.25	292076.16
4	163.743	12378.71	282823.12
5	144.204	10901.66	485144.16
6	134.726	10185.11	1428712.38
7	134.516	10169.24	326924.97
8	133.790	10114.31	558810.69
9	130.625	9875.05	3106536.75
10	128.138	9687.06	2791966.75
11	127.718	9655.32	1434502.75
12	126.766	9583.30	1486396.00
13	122.632	9270.80	1476195.25
14	122.018	9224.42	390618.84
15	113.412	8573.78	1316901.50
16	101.770	7693.65	475831.84
17	98.330	7433.64	392349.66
18	94.326	7130.91	1531183.38
19	92.291	6977.10	1388202.62
20	77.420	5852.83	2405642.00
21	77.000	5821.10	2440806.50
22	76.564	5788.14	2278243.50
23	74.416	5625.78	1246122.25
24	73.609	5564.75	1535371.75
25	69.815	5277.88	1376650.00
26	55.476	4193.90	1189629.12
27	19.080	1442.43	4346481.00
28	18.999	1436.33	4314265.00
29	12.121	916.31	3787278.25



18b





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18 peaks foulu in MG265H
peak    ppm       freq          amp

 1     7.240   2176.71    200821.30
 2     7.108   2137.00    220092.97
 3     7.098   2134.06    231719.22
 4     7.086   2130.38    218453.89
 5     7.035   2114.94    242987.56
 6     7.027   2112.73    188074.72
 7     7.005   2106.12    317550.69
 8     6.846   2058.32    126117.05
 9     6.602   1984.78    246108.48
10     6.572   1975.96    232488.58
11     4.535   1363.41    269155.06
12     3.652   1097.95   1040369.12
13     1.725    518.49    166029.02
14     1.698    510.40    135796.75
15     1.201    361.13    946761.50
16     1.177    353.77   1010022.12
17     1.160    348.63   1128221.25
18     1.135    341.27    962751.00
```
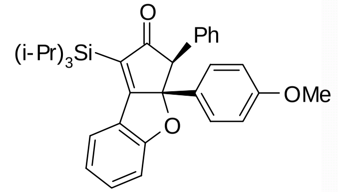

3

25 peaks found in MG265C

peak	ppm	freq	amp
1	206.500	15611.13	1271540.12
2	181.553	13725.15	707754.69
3	164.566	12440.97	1046955.06
4	159.237	12038.14	1331934.00
5	134.484	10166.80	1400981.38
6	134.064	10135.06	4435268.00
7	132.659	10028.86	847227.81
8	130.269	9848.19	8966481.00
9	127.734	9656.54	10000478.00
10	127.282	9622.36	8977263.00
11	127.008	9601.61	5582979.00
12	126.281	9546.68	4406071.00
13	122.600	9268.36	1053187.75
14	121.889	9214.65	5489712.00
15	113.170	8555.47	8939003.00
16	112.572	8510.30	5355412.00
17	101.754	7692.43	919856.56
18	77.420	5852.83	10474795.00
19	77.000	5821.10	11144453.00
20	76.580	5789.36	10950462.00
21	68.620	5187.55	5058952.50
22	55.121	4167.04	4099938.00
23	19.032	1438.77	10937267.00
24	18.967	1433.89	14968161.00
25	12.137	917.53	13003060.00



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