Supporting Information for:

Carbon Networks Based on Benzocyclynes. 6. Synthesis of Graphyne Substructures via Directed Alkyne Metathesis

Charles A. Johnson II,[†] Yunyi Lu,[‡] and Michael M. Haley*[,†]

[†]*Department of Chemistry and Materials Science Institute, University of Oregon, Eugene, OR 97403-1253*

[‡]*Departments of Chemistry and Materials Science & Engineering, University of Illinois at Urbana-Champaign, Urbana, IL 61801*

haley@uoregon.edu

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Experimental

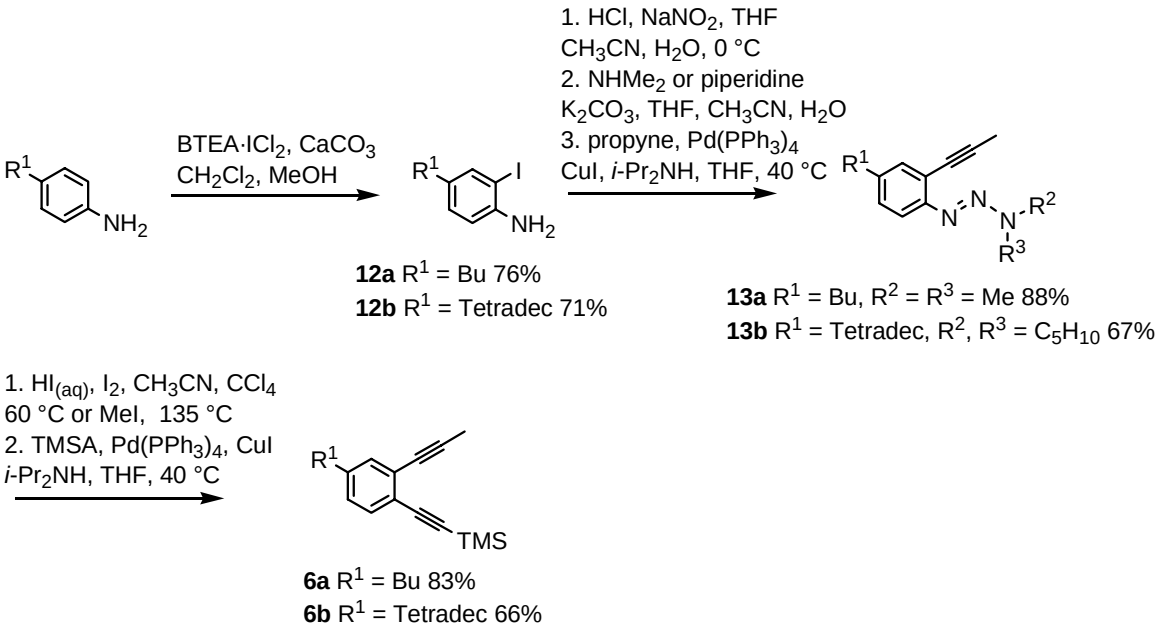
a. General Data. ^{1}H and ^{13}C NMR spectra were recorded using a Varian Inova 300 (^{1}H 299.95 MHz, ^{13}C 75.43 MHz) or Inova 500 (^{1}H 500.10 MHz, ^{13}C 125.75 MHz) spectrometer. Chemical shifts (δ) are expressed in ppm downfield from tetramethylsilane using the residual non-deuterated solvent as internal standard ($CDCl_3$: ^{1}H 7.26 ppm, ^{13}C 77.0 ppm). IR spectra were recorded using a Nicolet Magan-FTIR 550 spectrometer. UV-Vis spectra were recorded using a Hewlett-Packard 8453 spectrophotometer. Mass spectra were recorded using an Agilent 1100 Series LC/MSD or ION-TOF TOF-SIMS Model IV. Melting points were determined on a Meltemp II apparatus or using a TA Instruments 2920 Modulated DSC. THF was freshly distilled from potassium. 1,2,4,5-Tetraiodobenzene,[1] triazene **15**,[2] 1,2-didecyl-4,5-diiodobenzene,[3] and 1,4-dibromo-2,5-diiodobenzene[4] were prepared according to literature. All other chemicals were of reagent grade and used as obtained from manufacturers. Pd-, Mo-, and W-catalyzed reactions were carried out in an inert atmosphere (dry N_2 or Ar). Column chromatography was performed on Whatman reagent grade silica gel (230-400 mesh). Rotary chromatography was performed on a Harrison Research Chromatotron model 7924T with EM-Science $60PF_{254}$ silica gel. Precoated silica gel plates (Sorbent Technology, UV_{254}, 200 μm, 5 × 20 cm) were used for analytical thin-layer chromatography.

General Deprotection/Cross-Coupling Procedure. A suspension consisting of silyl-protected ethynylarene (1 equiv) and K_2CO_3 (3-5 equiv) in Et_2O (0.05 M) and MeOH (0.03 M) was stirred at rt for 1 h. The suspension was diluted with Et_2O and washed thrice with H_2O. The organic layer was dried over $MgSO_4$ and concentrated in vacuo. Without further purification, a syringe pump was used to deliver a deoxygenated solution of the free acetylene in THF (0.05 M) over 12 h to a stirred, deoxygenated suspension of haloarene, $Pd(PPh_3)_4$ (5 mol% per transformation), and CuI (10 mol% per transformation) in THF and *i*-Pr_2NH (1:1 ratio, 0.1 M) at 35-85 °C. The reaction was stirred an additional 12 h at rt under N_2, concentrated, and purified by silica gel chromatography.

General Propyne Coupling Procedure. A suspension consisting of iodoarene (1 equiv), $PdCl_2(PPh_3)_2$ (5 mol% per transformation), and CuI (10 mol% per transformation) in THF (0.1 M) and *i*-Pr_2NH (0.1 M) was degassed by bubbling argon. The reaction

mixture was placed under N_2 and heated between 40-50 °C. Propyne was bubbled through the stirred reaction mixture at a rate of ~75 mL/min for 5-10 min and stirring was continued for an additional 30 min. The reaction mixture was concentrated and purified by silica gel chromatography.

b. Synthesis of 6a and 6b.



4-Butyl-2-iodoaniline (12a). A suspension consisting of 4-butylaniline (2 g, 13 mmol), BTEA·ICl_2 (5.2 g, 13 mmol), and $CaCO_3$ (1.6 g, 16 mmol) in CH_2Cl_2 (50 mL) and MeOH (25 mL) was stirred for 1 h. The mixture was filtered, concentrated by rotary evaporation, and dissolved in Et_2O. 5% $NaHSO_3$ (100 mL) was added and the mixture was extracted four times with Et_2O. The combined organic fractions were dried with $MgSO_4$, concentrated, and chromatographed on silica with 2:1 CH_2Cl_2:hexanes to afford iodoaniline **12a** (2.81 g, 76%) as a red-brown oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.46 (d, J = 1.8 Hz, 1H), 6.95 (dd, J = 8.4, 1.8 Hz, 1H), 6.67 (d, J = 8.4 Hz, 1H), 3.95 (br s, 2H), 2.46 (t, J = 7.6 Hz, 2H), 1.60-1.47 (m, 2H), 1.39-1.26 (m, 2H), 0.91 (t, J = 7.3 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 144.34, 138.29, 134.60, 129.30, 114.57, 84.31, 34.05,

33.68, 22.12, 13.88. IR (neat): ν 3460, 2962, 2903, 2866 cm^{-1}. MS (CI pos) *m/z* (%): 276 (MH^+, 10), 275 (M^+, 100); $C_{10}H_{14}IN$ (275.13).

4-Tetradecyl-2-iodoaniline (12b). A red-brown oil (71%). ^1H NMR (300 MHz, $CDCl_3$): δ 7.46 (d, *J* = 1.8 Hz, 1H), 6.95 (dd, *J* = 8.4, 1.8 Hz, 1H), 6.67 (d, *J* = 8.4 Hz, 1H), 3.93 (br s, 2H), 2.45 (t, *J* = 7.5 Hz, 2H), 1.62-1.43 (m, 2H), 1.26 (br s, 22H), 0.89 (t, *J* = 6.9 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 144.38, 138.39, 134.83, 129.36, 114.62, 84.36, 34.45, 31.90, 31.63, 29.64 (5C), 29.56, 29.45, 29.33, 29.16, 22.67, 14.11. IR (neat): ν 3458, 2959, 2910, 2867 cm^{-1}. MS (CI pos) *m/z* (%): 416 (MH^+, 100), 415 (M^+, 41); $C_{20}H_{34}IN$ (415.17).

Triazene 13a. $NaNO_2$ (2.9 g, 42 mmol) in H_2O (15 mL) was added dropwise to a stirred, cooled solution (0 °C) of iodoaniline **12a** (10.58 g, 38 mmol) in conc. HCl (12 mL), H_2O (20 mL), CH_3CN (20 mL), and THF (50 mL). The reaction was stirred for an additional 20 min at 0 °C. Maintaining temperature below 0 °C, the reaction mixture was added slowly to a stirred solution of Me_2NH (40% aq, 6.5 g, 58 mmol) and K_2CO_3 (8 g, 58 mmol) in THF (20 mL) and H_2O (20 mL) at 0 °C. The mixture was warmed to rt, extracted twice with Et_2O, and the combined organics were washed with H_2O thrice. The organic layer was dried over $MgSO_4$, concentrated by rotary evaporation, and filtered through a 2.5 cm silica plug with 1:1 CH_2Cl_2:hexanes to afford a red oil. Without further purification, the intermediate red oil was reacted according to General Propyne Coupling Procedure. The mixture was concentrated and filtered through a 2.5 cm silica plug with 2:1 hexanes:CH_2Cl_2 to afford **13a** (8.6 g, 88%) as a red-brown oil. ^1H NMR (300 MHz, $CDCl_3$): δ 7.29-7.24 (m, 2H), 7.02 (dd, *J* = 8.3, 2.1 Hz, 1H), 3.37 (br s, 6H), 2.54 (t, *J* = 7.2 Hz, 2H), 2.10 (s, 3H), 1.62-1.52 (m, 2H), 1.39-1.26 (m, 2H), 0.91 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 149.68, 139.48, 132.61, 128.45, 128.32, 116.89, 89.23, 77.80, 34.81, 33.38, 31.51, 22.56, 22.12, 13.85, 4.61. IR (neat): ν 2966, 2933, 2870 cm^{-1}. MS (CI pos) *m/z* (%): 245 (M^++2, 19), 244 (MH^+, 100), 243 (M^+, 40); $C_{15}H_{21}N_3$ (243.35).

Triazene 13b. An orange oil (67%). ^1H NMR (300 MHz, $CDCl_3$): δ 7.30 (d, *J* = 8.4 Hz, 1H), 7.22 (d, *J* = 1.9 Hz, 1H), 7.02 (dd, *J* = 8.4, 1.9 Hz, 1H), 3.80 (br s, 4H), 2.53 (t, *J* = 7.8 Hz, 2H), 2.10 (s, 3H), 1.70 (br s, 6H), 1.61-1.52 (m, 2H), 1.25 (br s, 22H), 0.88 (t, *J* = 6.9 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 149.65, 139.87, 132.71, 128.50, 118.59, 116.77, 89.34, 77.87, 35.22, 31.89, 31.27, 29.64 (5C), 29.57, 29.49, 29.33, 29.16, 25.24

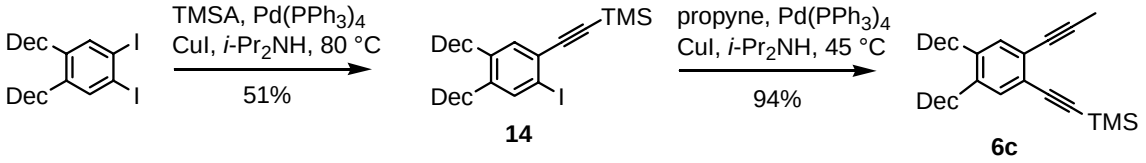
(2C), 24.42, 22.66, 14.09, 4.70. IR (neat): ν 2962, 2940, 2865 cm^{-1}. MS (CI pos) *m/z* (%): 425 (M^++2, 39), 424 (MH^+, 100); $C_{28}H_{45}N_3$ (423.68).

Diyne 6a. Arene **13a** (2.0 g, 8.2 mmol) in CCl_4 (100 mL) was added dropwise over 30 min to a stirred solution of I_2 (10.4 g, 41 mmol) and HI (48% aq, 7.2 mL, 41 mmol) in CH_3CN (100 mL) at 60 °C. The reaction mixture was stirred for an additional 10 min, diluted with CH_2Cl_2, and then washed successively with saturated $NaHCO_3$, H_2O, and 5% $NaHSO_3$. The organic layer was dried with $MgSO_4$ and concentrated in vacuo. The crude product was extracted with hexanes to afford a yellow oil. A suspension of the intermediate yellow oil (2.1 g, 7 mmol), $Pd(PPh_3)_4$ (410 mg, 0.35 mmol), and CuI (133 mg, 0.7 mmol) in THF (100 mL) and *i*-Pr_2NH (100 mL) was degassed by bubbling argon. TMSA (1.4 g, 14 mmol) was added and the mixture was stirred under N_2 for 12 h at 40 °C. The solvent was removed by rotary evaporation and the crude residue was filtered through a 2.5 cm silica plug with 2:1 hexanes:CH_2Cl_2. Silica gel chromatography (hexanes) of the concentrated filtrate afforded **6a** (1.8 g, 83%) as a highly viscous, pale yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.34 (d, *J* = 8.3 Hz, 1H), 7.20 (d, *J* = 1.5 Hz, 1H), 7.00 (dd, *J* = 8.3, 1.5 Hz, 1H), 2.54 (t, *J* = 7.5 Hz, 2H), 2.11 (s, 3H), 1.61-1.50 (m, 2H), 1.36-1.24 (m, 2H), 0.91 (t, *J* = 7.2 Hz, 3H), 0.28 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 143.16, 131.68, 131.46, 127.47, 126.79, 122.66, 104.05, 96.89, 89.57, 78.52, 35.21, 33.06, 22.11, 13.81, 4.32, 0.06. IR (neat): ν 2958, 2931, 2859, 2158 cm^{-1}. MS (CI pos) *m/z* (%): 340 (M^++THF, 41), 268 (M^+, 22); $C_{18}H_{24}Si$ (268.47).

Diyne 6b. Triazene **13b** (500 mg, 1.2 mmol) and MeI (6 mL) were combined in a sealed tube equipped with a stir bar. The reaction was stirred for 12 h at 135 °C and upon cooling, the suspension was filtered. The filtrate was concentrated and chromatographed on silica gel (hexanes) to afford a colorless oil. Without further purification, the intermediate was reacted with TMSA as per **6a** to afford **6b** as a pale yellow oil (328 mg, 66%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.34 (d, *J* = 8.1 Hz, 1H), 7.20 (d, *J* = 1.5 Hz, 1H), 7.00 (dd, *J* = 8.1, 1.5 Hz, 1H), 2.54 (t, *J* = 7.5 Hz, 2H), 2.11 (s, 3H), 1.61-1.47 (m, 2H), 1.26 (br s, 22H), 0.89 (t, *J* = 6.9 Hz, 3H), 0.27 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 143.32, 131.74, 131.54, 127.53, 126.83, 122.70, 104.07, 97.01, 89.67, 78.55, 35.61, 31.93, 30.99, 29.66 (5C), 29.55, 29.44, 29.36, 29.11, 22.69, 14.13, 4.43, 0.02. IR (neat): ν

2924, 2853, 2159 cm^{-1}. MS (CI pos) *m/z* (%): 481 (MH^++THF, 13), 480 (M^++THF, 37), 479 (M^+-1+THF, 100); $C_{28}H_{44}Si$ (408.73).

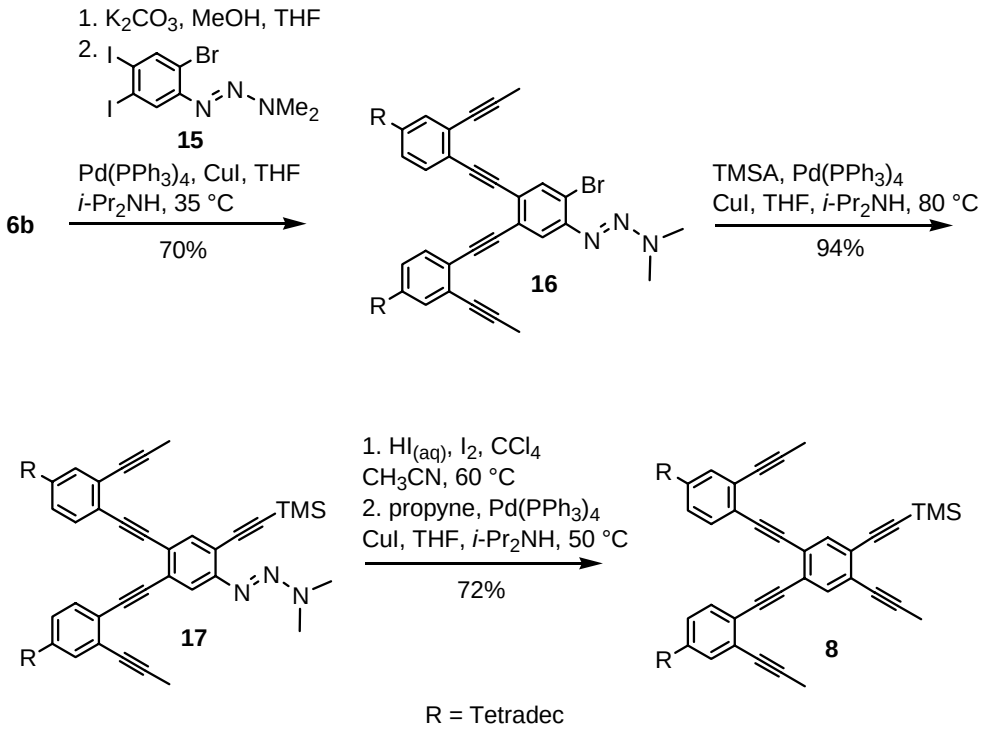
c. Synthesis of 6c.



Iodoarene 14. A suspension of 1,2-didecyl-4,5-diiodobenzene (2.05 g, 3.4 mmol), $Pd(PPh_3)_4$ (194 mg, 0.2 mmol), and CuI (64 mg, 0.4 mmol) in *i*-Pr_2NH (25 mL) was degassed by bubbling argon. TMSA (333 mg, 3.4 mmol) was added and the mixture was stirred in a sealed tube at 80 °C for 3 h. The solvent was removed by rotary evaporation and the crude residue was filtered through a 2.5 cm silica plug with 5:1 hexanes:CH_2Cl_2. Silica gel chromatography (hexanes) of the concentrated filtrate afforded **14** (1 g, 51%) as a pale yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.58 (s, 1H), 7.25 (s, 1H), 2.59-2.41 (m, 4H), 1.60-1.53 (m, 4H), 1.27 (s, 28H), 0.89 (t, *J* = 6.6 Hz, 6H), 0.28 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 143.33, 140.64, 138.97, 133.30, 126.68, 106.90, 97.66, 97.17, 32.22, 32.09, 30.91, 29.60, 29.56, 29.47, 29.41, 29.33, 22.69, 14.13, -0.12. IR (neat): ν 2960, 2943, 2866, 2158 cm^{-1}. MS (CI pos) *m/z* (%): 580 (M^+, 39), 579 (M^+-1, 100); $C_{31}H_{53}ISi$ (580.30).

Diyne 6c. Arene **14** (1.1 g, 1.9 mmol) was reacted according to General Propyne Coupling Procedure. The crude residue was successively filtered through two 2.5 cm silica plugs with Et_2O and then hexanes to afford **6c** (870 mg, 94%) as a yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.21 (s, 1H), 7.15 (s, 1H), 2.54 (t, *J* = 7.5 Hz, 4H), 1.60-1.49 (m, 4H), 1.26 (s, 28H), 0.88 (t, *J* = 6.3 Hz, 6H), 0.26 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 141.28, 140.21, 132.44, 132.17, 124.18, 122.59, 104.35, 96.51, 88.79, 78.52, 32.40, 32.33, 31.90, 30.88, 30.82, 29.66, 29.60, 29.56, 29.50, 29.33, 22.69, 14.11, 4.44, 0.51. IR (neat): ν 2956, 2925, 2853, 2155 cm^{-1}. MS (CI pos) *m/z* (%): 565 (MH^++THF, 39), 564 (M^++THF, 100), 563 (M^+-1+THF, 100); $C_{34}H_{56}Si$ (492.42).

e. Synthesis of 8.



Tetrayne 16. Diethynylarene **6b** (500 mg, 1.2 mmol) was reacted with triazene **15** (266 mg, 0.55 mmol) according to General Deprotection/Cross-Coupling Procedure at 35 °C. The residue was filtered through a 2.5 cm silica plug with 2:1 hexanes:CH_2Cl_2 and the filtrate concentrated in vacuo. Purification via column chromatography (2:1 hexanes:CH_2Cl_2) afforded **16** (343 mg, 70%) as a bright yellow, amorphous solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.81 (s, 1H), 7.67 (s, 1H), 7.48 (dd, *J* = 7.8, 5 Hz, 2H), 7.26 (br s, 2H), 7.05 (d, *J* = 7.8 Hz, 2H), 3.41 (br d, 6H), 2.57 (t, *J* = 7.5 Hz, 4H), 2.03 (s, 3H), 2.01 (s, 3H), 1.65-1.58 (m, 4H), 1.27 (br s, 44H), 0.89 (t, *J* = 7.3 Hz, 6H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 147.39, 143.22, 143.18, 136.37, 131.96, 131.91, 131.71, 131.63, 127.57 (2C), 126.46, 126.36, 125.40, 123.21, 122.93, 122.87, 121.35, 119.13, 93.20, 92.62, 90.78, 90.31, 90.04, 89.99, 78.73, 78.68, 42.50, 36.33, 35.66, 31.91, 31.01, 29.64, 29.55, 29.46, 29.35, 29.20, 22.67, 14.09, 4.51. IR (neat): ν 2943, 2833, 2156 cm^{-1}. MS (CI pos) *m/z* (%): 968 (MH^++THF, 15), 900 (M^++5, 19), 898 (M^++3, 100), 897 (M^++2, 63), 896 (MH^+, 87); $C_{58}H_{78}BrN_3$ (895.44).

Pentayne 17. A degassed suspension consisting of arene **16** (2.05 g, 2.3 mmol), TMSA (562 mg, 5.7 mmol), $Pd(PPh_3)_4$ (132 mg, 0.11 mmol), and CuI (50 mg, 0.23 mmol) in THF (50 mL) and *i*-Pr_2NH (50 mL) was stirred at 80 °C for 12 h in a sealed tube. After cooling of the reaction vessel, the mixture was concentrated, filtered through a 2.5 cm silica plug (2:1 hexanes:CH_2Cl_2), and purified by silica gel chromatography (2:1 hexanes:CH_2Cl_2) to afford **17** (1.97 g, 94%) as a yellow, amorphous solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.71 (s, 1H), 7.69 (s, 1H), 7.48 (dd, *J* = 8.1 , 5.8 Hz, 2H), 7.26 (br s, 2H), 7.05 (d, *J* = 8.1 Hz, 2H), 3.42 (br d, 6H), 2.57 (t, *J* = 7.5 Hz, 4H), 2.01 (s, 6H), 1.65-1.57 (m, 4H), 1.27 (br s, 44H), 0.89 (t, *J* = 6.6 Hz, 6H), 0.27 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 151.07, 143.20, 142.95, 136.87, 131.94 (2C), 131.70, 131.58, 127.51 (2C), 126.48, 126.25 (2C), 123.03, 122.90, 121.94, 120.29, 117.88, 102.32, 100.01, 93.44, 92.53, 91.33, 90.70, 89.99, 89.90, 78.70 (2C), 43.01, 35.99, 35.63, 31.88, 30.93, 29.64, 29.62, 29.52, 29.43, 29.32, 29.16, 22.65, 14.07, 4.47, 4.44, 0.016. IR (neat): ν 2955, 2924, 2850, 2150 cm^{-1}. MS (CI pos) *m/z* (%): 916 (M^++2, 43), 915 (MH^+, 82), 914 (M^+, 100); $C_{63}H_{87}N_3Si$ (914.47).

Hexayne 8. Pentayne **17** (1.05 g, 1.1 mmol) in CCl_4 (100 mL) was added dropwise over 30 min to a stirred solution of I_2 (1.5 g, 5.8 mmol) and HI (55% aq, 0.9 mL, 5.8 mmol) in CH_3CN (100 mL) at 60 °C. The reaction mixture was stirred for an additional 10 min, diluted with CH_2Cl_2, and then washed successively with saturated $NaHCO_3$, H_2O, and 5% $NaHSO_3$. The organic layer was dried with $MgSO_4$, concentrated in vacuo, and extracted with hexanes to afford a light brown, highly viscous oil. Without further purification, the intermediate oil was reacted according to General Propyne Coupling Procedure. Purification by silica gel chromatography (5:1 hexanes:CH_2Cl_2) afforded **8** (698 mg, 72%) as a light brown solid. Mp: 61-63 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.67 (s, 1H), 7.59 (s, 1H), 7.48 (dd, *J* = 7.8, 2.7 Hz, 2H), 7.26 (br s, 2H), 7.07 (d, *J* = 7.8 Hz, 2H), 2.58 (t, *J* = 8.1 Hz, 4H), 2.15 (s, 3H), 2.01 (s, 6H), 1.65-1.56 (m, 4H), 1.28 (br s, 44H), 0.91 (t, *J* = 7.2 Hz, 6H), 0.31 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 143.49, 143.43, 135.48, 135.05, 132.18, 132.12, 131.79, 131.76, 127.57 (2C), 126.44 (2C), 126.22, 125.59, 124.81, 124.78, 122.61, 122.56, 102.91, 99.90, 94.47, 94.15, 92.12, 90.18, 90.14 (2C), 90.11, 78.56, 78.54, 77.78, 35.67, 31.92, 31.58, 31.02, 29.64, 29.55, 29.46, 29.35, 29.20, 22.68, 14.10, 4.55, 4.49, 0.14. IR (neat): ν 2924, 2853, 2156 cm^{-1}.

MS (CI pos) *m/z* (%): 953 (M^++THF, 43), 883 (M^++2, 33), 882 (MH^+, 51), 881 (M^+, 64); $C_{64}H_{64}Si$ (881.44).

e. Synthesis of 3a-c, 4, and 7-11.

Octayne 7a. Diethynylarene **6a** (1.4 g, 5.2 mmol) was reacted with 1,2,4,5-tetraiodobenzene (500 mg, 0.86 mmol) according to General Deprotection/Cross-Coupling Procedure at 50 °C. The residue was filtered through a 2.5 cm silica plug with 1:1 hexanes:CH_2Cl_2 and the filtrate concentrated in vacuo. Purification via Chromatotron (5:1 hexanes:CH_2Cl_2) afforded **7a** (536 mg, 73%) as a light yellow solid. Mp: 140-142 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.77 (s, 2H), 7.49 (d, *J* = 7.8 Hz, 4H), 7.27 (d, *J* = 1.5 Hz, 4H), 7.06 (dd, *J* = 7.8, 1.5 Hz, 4H), 2.59 (t, *J* = 7.5 Hz, 8H), 2.01 (s, 12H), 1.62-1.54 (m, 8H), 1.39-1.31 (m, 8H), 0.93 (t, *J* = 7.2 Hz, 12H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 143.49, 135.63, 132.19, 131.84, 127.66, 126.43, 125.23, 122.62, 94.35, 90.38, 90.21, 78.58, 35.36, 33.17, 22.24, 13.92, 4.55. UV-Vis (CH_2Cl_2): λ_{max} (ε) 233 (89,900), 266 (64,900), 336 (79,000), 367 (41,000) nm. IR (neat): ν 2955, 2929, 2858, 2207 cm^{-1}. MS (CI pos) *m/z* (%): 858 (M^++3, 25), 857 (M^++2, 66), 856 (MH^+, 100), 855 (M^+, 43); $C_{66}H_{62}$ (855.20).

Octayne 7b. A yellow solid (76%). Mp: 77-79 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.76 (s, 2H), 7.48 (d, *J* = 7.8 Hz, 4H), 7.26 (br s, 4H), 7.05 (d, *J* = 8.1 Hz, 4H), 2.57 (t, *J* = 7.5 Hz, 8H), 2.00 (s, 12H), 1.65-1.51 (m, 8H), 1.26 (br s, 88H), 0.88 (t, *J* = 6.3 Hz, 12H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 143.53, 135.64, 132.18, 131.82, 127.65, 126.44, 125.24, 122.63, 94.35, 90.39, 90.21, 78.60, 35.70, 31.92, 31.06, 29.65 (5C), 29.56, 29.47, 29.36, 29.22, 22.69, 14.13, 4.55. IR (neat): ν 2923, 2850, 2199 cm^{-1}. MS (CI pos) *m/z* (%): 1419 (M^++3, 17), 1418 (M^++2, 83), 1417 (MH^+, 98), 1416 (M^+, 100); $C_{106}H_{142}$ (1416.26).

Octayne 7c. An amorphous, yellow solid (77%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.76 (s, 2H), 7.37 (s, 4H), 7.21 (s, 4H), 2.63-2.45 (m, 16H), 1.98 (s, 12H), 1.63-1.51 (m, 16H), 1.26 (br s, 112H), 0.98-0.81 (m, 24H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 141.40, 140.22, 135.24, 133.05, 132.35, 125.38, 123.77, 122.66, 94.62, 90.11, 89.33, 32.52, 32.35, 31.94, 31.92, 30.95, 30.89, 29.76, 29.71, 29.67, 29.64, 29.60, 29.55, 29.39, 29.36, 22.70, 14.11,

4.66, 4.54. IR (neat): ν 2961, 2937, 2840, 2200 cm^{-1}. MS (CI pos) *m/z* (%): 1754 (M^++3, 100), 1753 (M^++2, 95), 1752 (MH^+, 71); $C_{130}H_{190}$ (1751.49).

Bis[12]cyclyne 3c. A suspension of **7a** (70 mg, 0.08 mmol) and (*t*-BuO)$_3$W≡C-*t*-Bu (50 mol%) in toluene was heated for 24 h at 80 °C under an Ar atmosphere. Concentration in vacuo, purification via Chromatotron (2:1 hexanes:CH_2Cl_2), and precipitation with cold Et_2O afforded **3c** (28 mg, 46%) as a bright yellow solid. Mp: 225 °C (dec). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.30 (s, 2H), 7.22 (d, *J* = 8 Hz, 4H), 7.20 (br s 4H), 7.00 (d, *J* = 8 Hz, 4H), 2.59 (t, *J* = 7.5 Hz, 8H), 1.62-1.54 (m, 8H), 1.39-1.31 (m, 8H), 0.93 (t, *J* = 7.5 Hz, 12H). ^{13}C NMR: insufficient solubility to obtain spectrum. UV-Vis (CH_2Cl_2): λ_{max} (ε) 286 (49,100), 295 (61,600), 305 (119,300), 318 (64,700), 329 (70,300), 343 (97,800), 380 (10,600), 399 (10,800) nm. Fluorescent emission ([**3c**] ≤ 5 × 10^{-5} M in toluene; Φ_F = 0.19; 399 nm excitation): λ_{max} 488, 525, 546 nm. IR (neat): ν 2955, 2934, 2861, 2217 cm^{-1}. MS (CI pos) *m/z* (%): 820 (MH^++THF, 52), 819 (M^++THF, 82), 748 (MH^+, 33); $C_{58}H_{50}$ (747.02).

Bis[12]cyclyne 3d. A solution of **7b** (50 mg, 0.035 mmol), EtC≡Mo[N(*t*-Bu)(3,5-$C_6H_3Me_2$)]$_3$ (7 mg, 0.01 mmol), and silanol-POSS (58 mg, 0.06 mmol) in 1,2,4-trichlorobenzene (1.1 mL) was heated for 30 min at 75 °C and 1 torr. Removal of solvent by distillation and trituration of the crude residue with cold Et_2O afforded **3d** (40 mg, 87%) as a bright yellow solid. Mp: 110-112 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.30 (s, 2H), 7.22 (d, *J* = 7.9 Hz, 4H), 7.18 (d, *J* = 1.3 Hz, 4H), 7.00 (dd, *J* = 7.9, 1.3 Hz, 4H), 2.53 (t, *J* = 7.3 Hz, 8H), 1.65-1.50 (m, 8H), 1.26 (br s, 88H), 0.88 (t, *J* = 6.9 Hz, 12H). ^{13}C NMR: insufficient solubility to obtain spectrum. UV-Vis (CH_2Cl_2): λ_{max} (ε) 288 (58,000), 296 (70,600), 306 (115,500), 319 (70,200), 331 (72,200), 344 (97,600), 381 (16,100), 399 (13,100) nm. Fluorescent emission ([**3d**] ≤ 5 × 10^{-5} M in toluene; 399 nm excitation): λ_{max} 488, 525, 546 nm. IR (neat): ν 2925, 2857, 2212 cm^{-1}. MS (CI pos) *m/z* (%): 1380 (MH^++THF, 43), 1379 (M^++THF, 79), 1309 (M^++2, 85), 1308 (MH^+, 100), 1307 (M^+, 83); $C_{98}H_{130}$ (1307.02).

Bis[12]cyclyne 3e. A solution of **7c** (60 mg, 0.03 mmol), EtC≡Mo[N(*t*-Bu)(3,5-$C_6H_3Me_2$)]$_3$ (13.6 mg, 0.02 mmol), and silanol-POSS (112 mg, 0.12 mmol) in 1,2,4-trichlorobenzene (20 mL) was heated for 14 h at 75 °C under 1 torr. The solvent was removed by distillation and the crude material was purified by silica gel chromatography

(3:1 hexanes:CH_2Cl_2) to afford **3e** (44 mg, 91%) as a bright yellow solid. Mp: 111-112 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.28 (s, 2H), 7.11 (s, 4H), 7.07 (s, 4H), 2.51 (t, *J* = 7.5 Hz, 16H), 1.65-1.47 (m, 16H), 1.28 (br s, 112H), 0.89 (t, *J* = 7.2 Hz, 24H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 141.97, 141.37, 134.49, 132.55, 132.32, 126.45, 124.20, 123.43, 95.19, 92.19, 91.07, 32.44, 32.32, 31.90, 30.67, 30.56, 29.70, 29.63, 29.59, 29.52, 29.53, 27.25, 26.99, 22.69, 14.11. UV-Vis (CH_2Cl_2): λ_{max} (ε) 292 (40,000), 300 (54,000), 311 (118,000), 322 (60,000), 333 (62,700), 347 (99,500), 381 (7,700), 401 (6,700) nm. Fluorescent emission ([**3e**] ≤ 5×10^{-5} M in toluene; Φ_F = 0.21; 399 nm excitation): λ_{max} 495, 533, 553 nm. IR (neat): ν 2953, 2923, 2851, 2212 cm^{-1}. MS (TOF-SIMS, pos, Bi_3^+) *m/z*: 1645.6 (M^++2), 1644.6 (MH^+), 1643.6 (M^+); $C_{122}H_{178}$ (1643.39).

Polyyne 9. Hexayne **8** (200 mg, 0.23 mmol) was reacted with 1,2-didecyl-4,5-diiodobenzene (55 mg, 0.09 mmol) according to General Deprotection/Cross-Coupling Procedure at 85 °C. The residue was filtered through a 2.5 cm silica plug with 3:1 hexanes:CH_2Cl_2 and the filtrate concentrated in vacuo. Purification via column chromatography (4:1 hexanes:CH_2Cl_2) afforded **9** (145 mg, 81%) as an amorphous orange-brown solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.79 (s, 2H), 7.62 (s, 2H), 7.48-7.39 (m, 6H), 7.26 (s, 2H), 7.22 (s, 2H), 7.06-7.00 (m, 4H), 2.71-2.43 (m, 12H), 2.01 (s, 6H), 1.98 (s, 6H), 1.87 (s, 6H), 1.67-1.53 (m, 12H), 1.26 (br s, 116H), 0.89 (t, *J* = 7.2 Hz, 18H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 143.41, 143.33, 141.58, 135.65, 135.37, 132.98, 132.26 (2C), 131.71 (2C), 127.61, 127.57, 126.49, 126.39, 125.78, 125.27, 125.22, 124.86, 122.79, 122.75, 122.65, 94.21, 94.18, 93.66, 93.51, 92.41, 90.84, 90.52, 90.30, 90.23, 90.17, 78.57, 77.98, 35.71, 35.70, 32.52, 32.49, 31.92, 31.01, 30.88, 29.66, 29.59, 29.48, 29.36, 29.27, 29.24, 22.69, 14.11, 4.71, 4.51, 4.40. UV-Vis (CH_2Cl_2): λ_{max} (ε) 248 (98,900), 266 (98,100), 314 (87,000), 366 (56,000) nm. IR (neat): ν 3056, 2934, 2852, 2195 cm^{-1}. MS (CI pos) *m/z* (%): 1975 (M^++3, 33), 1974 (M^++2, 37), 1972 (M^+, 63), 962 (100); $C_{148}H_{194}$ (1971.50).

Tris[12]cyclyne 4. a) Using W-catalyst: A suspension of polyyne **9** (140 mg, 0.07 mmol) and (*t*-BuO)$_3$W≡C-*t*-Bu (100 mol%) in toluene was heated for 4 h at 80 °C under an Ar atmosphere. Concentration in vacuo and purification via Chromatotron (5:1 hexanes: CH_2Cl_2) afforded **4** (25 mg, 19%) as a bright orange solid. b) Using Mo-catalyst: Reaction of 9 (40 mg, 0.02 mmol) with EtC≡Mo[N(*t*-Bu)(3,5-$C_6H_3Me_2$)]$_3$ (6

mg, 0.008 mmol) and silanol-POSS (49 mg, 0.05 mmol) in 1,2,4-trichlorobenzene (5 mL) for 3 h at 75 °C and 1 torr afforded 10 mg of **4** (31%). Mp: 183-185 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.21-7.15 (m, 12H), 7.07 (s, 2H), 6.98 (dd, *J* = 7.0, 1.9 Hz, 4H), 2.53 (br s, 12H), 1.62-1.54 (m, 12H), 1.26 (br s, 116H), 0.95-0.92 (m, 18H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 144.90, 144.10, 144.05, 142.19, 134.71, 134.66, 132.49, 131.95 (2C), 128.86, 126.93, 126.75 (2C), 126.73, 126.65, 126.43, 125.89, 123.62, 123.59 (2C), 123.56, 95.36, 95.21, 95.05, 93.91, 92.74, 92.64, 91.50, 91.44, 91.13, 35.66, 31.93, 30.85, 30.42, 29.69, 29.66, 29.61, 29.56, 29.47, 29.36, 29.24, 22.69, 14.12. UV-Vis (CH_2Cl_2): λ_{max} (ε) 267 (54,000), 305 (71,300), 327 (128,000), 318 (64,700), 357 (135,700), 411 (25,200), 430 (10,500), 461 (6,800), 511 (2,700) nm. Fluorescent emission ([**4**] ≤ 5×10^{-5} M in toluene or CH_2Cl_2; Φ_F = 0.27; 411 nm excitation): λ_{max} 515, 557, 579 nm. IR (neat): ν 2924, 2852, 2212 cm^{-1}. MS (tof-SIMS, pos, Bi_3^+) *m/z*: 1811.1 (M^++2), 1810.6 (MH^+), 1809.1 (M^+); $C_{136}H_{176}$ (1809.38).

Tetrayne 10. Diyne **6b** (110 mg, 0.27 mmol) was reacted with 1,4-dibromo-2,5-diiodobenzene (62 mg, 0.13 mmol) according to General Deprotection/Cross-Coupling Procedure at 30 °C. The residue was filtered through a 2.5 cm silica plug with 2:1 hexanes:CH_2Cl_2 and the filtrate concentrated in vacuo. Purification via column chromatography (3:1 hexanes:CH_2Cl_2) afforded **10** (103 mg, 90%) as a pale yellow solid. Mp: 109-110 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.78 (s, 2H), 7.45 (d, *J* = 7.8 Hz, 2H), 7.27 (br s, 2H), 7.09 (br d, *J* = 8.1 Hz, 2H), 2.57 (t, *J* = 7.5 Hz, 4H), 2.15 (s, 6H), 1.63-1.57 (m, 4H), 1.25 (br s, 44H), 0.88 (t, *J* = 6.5 Hz, 6H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 144.19, 136.33, 132.05, 127.79, 126.61, 126.53, 123.15, 121.77, 121.36, 95.99, 90.32, 89.38, 78.51, 35.71, 31.93, 31.00, 29.66, 29.54, 29.44, 29.36, 29.17, 22.70, 14.14, 4.89. IR (neat): ν 2935, 2874, 2165 cm^{-1}. MS (CI pos) *m/z* (%): 976 (M^++2+THF, 43), 906 (M^++4, 13), 905 (M^++3, 27), 903 (MH^+, 17), 743 (100); $C_{56}H_{72}Br_2$ (902.4).

Polyyne 11. Hexayne **8** (134 mg, 0.15 mmol) was reacted with tetrayne **10** (60 mg, 0.06 mmol) according to General Deprotection/Cross-Coupling Procedure at 85 °C. The residue was filtered through a 2.5 cm silica plug with 3:2 hexanes:CH_2Cl_2 and the filtrate concentrated in vacuo. Purification via Chromatotron (3:1 hexanes:CH_2Cl_2) afforded **11** (102 mg, 65%) as a bright yellow, waxy solid. Mp: >175 °C (dec). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.81 (s, 2H), 7.98 (s, 2H), 7.65 (s, 2H), 7.54-7.46 (m, 6H), 7.26 (br s, 6H),

7.10-7.04 (m, 6H), 2.61 (t, J = 7.7 Hz, 8H), 2.52 (t, J = 7.9 Hz, 4H), 2.09 (s, 6H), 2.05 (s, 6H), 2.03 (s, 6H), 1.87 (s, 6H), 2.63-2.47 (m, 12H), 1.28 (br s, 132H), 0.90 (t, J = 6.9 Hz, 18H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 143.82, 143.60, 143.48, 135.81, 135.62, 135.61, 135.44, 132.35, 132.21, 132.19, 131.95, 131.83, 131.50, 127.88, 127.64, 127.53, 126.60, 126.49, 126.39, 125.91, 125.79, 125.61, 125.18, 124.92, 124.72, 122.69, 122.61, 122.32, 94.99, 94.73, 94.22, 93.66, 92.72, 92.71, 90.43, 90.35, 90.23, 90.18, 90.09 (2C), 78.67, 78.58, 78.49, 77.85, 35.72, 35.70, 31.93, 31.06, 31.05, 30.94, 29.68, 29.57, 29.47, 29.36, 29.29, 29.27, 22.69, 14.11, 4.64, 4.56, 4.37. UV-Vis (CH_2Cl_2): λ_{max} (ε) 251 (117,000), 312 (72,300), 365 (59,800), 424 (36,900) nm. IR (neat): ν 2963, 2901, 2873, 2187 cm^{-1}. MS (CI pos) *m/z* (%): 2361 (MH^+, 72), 2360 (M^+, 100); $C_{178}H_{222}$ (2359.74).

References

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(2) Marsden, J. A.; Haley, M. M. *J. Org. Chem.* **2005**, *70*, 10213-10226.

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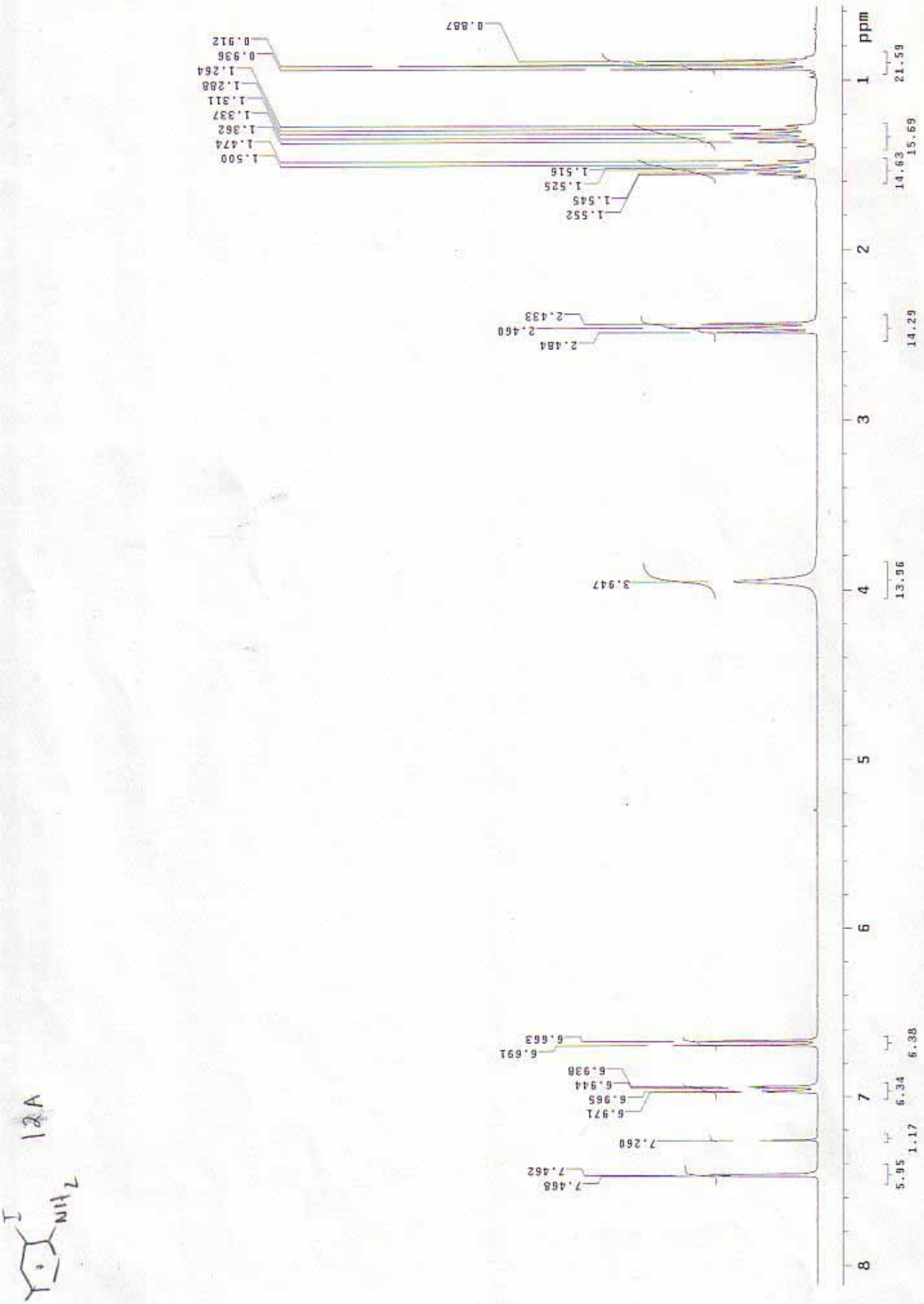
(4) Hart, H.; Harada, K.; Du, C. J. F. *J. Org. Chem* **1985**, *50*, 3104-3110.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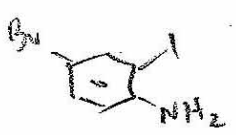

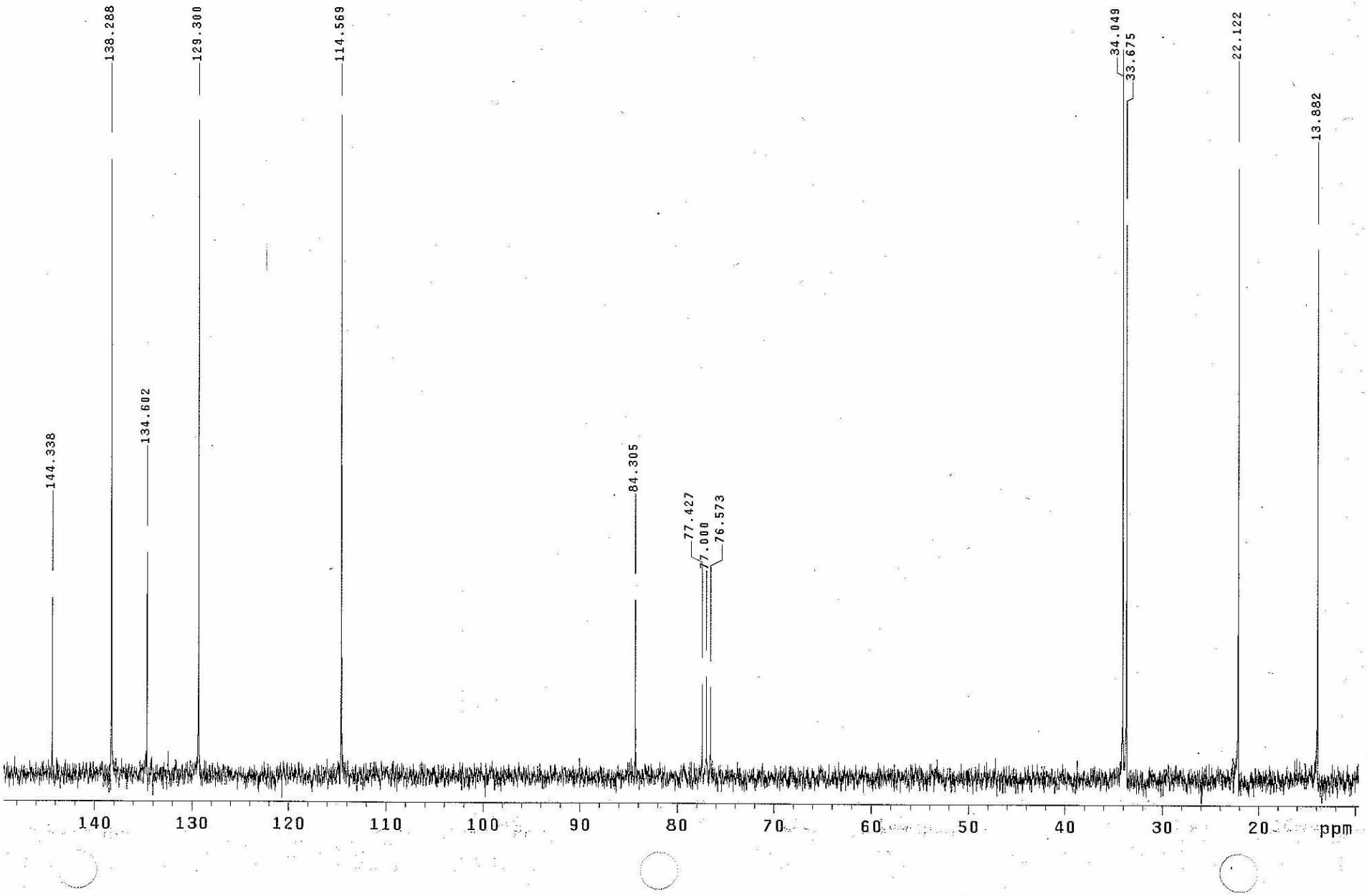
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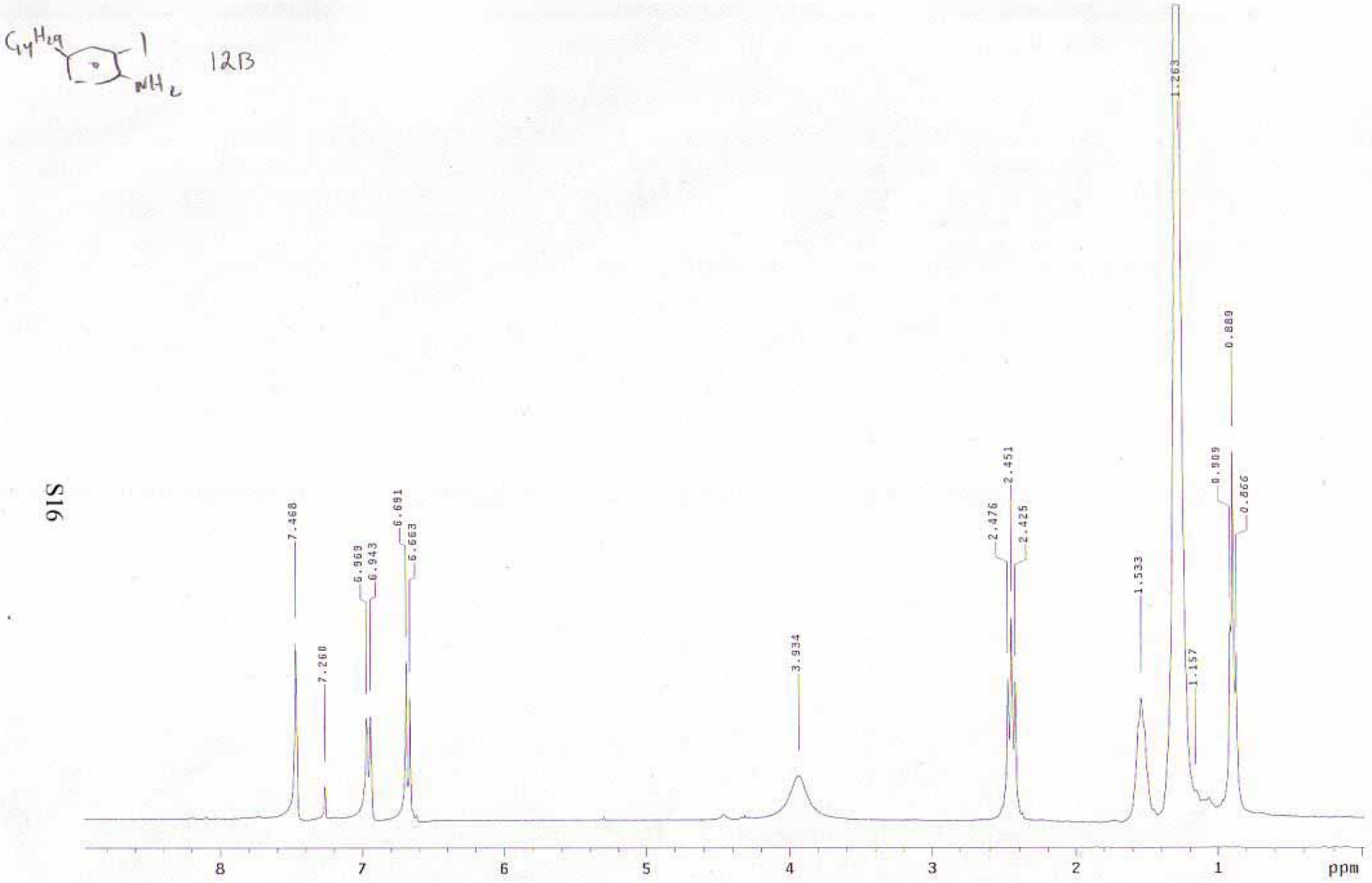
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12B


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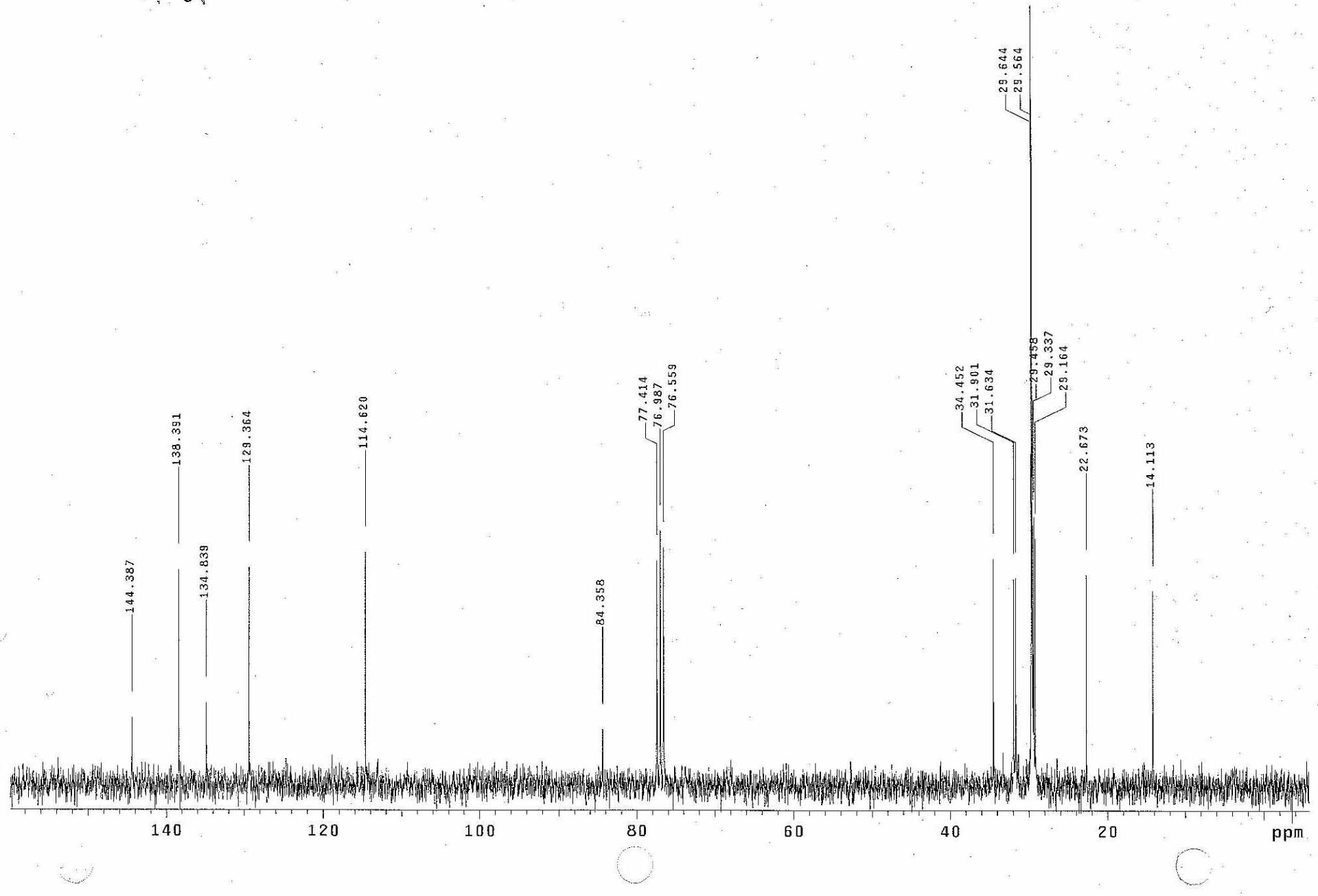
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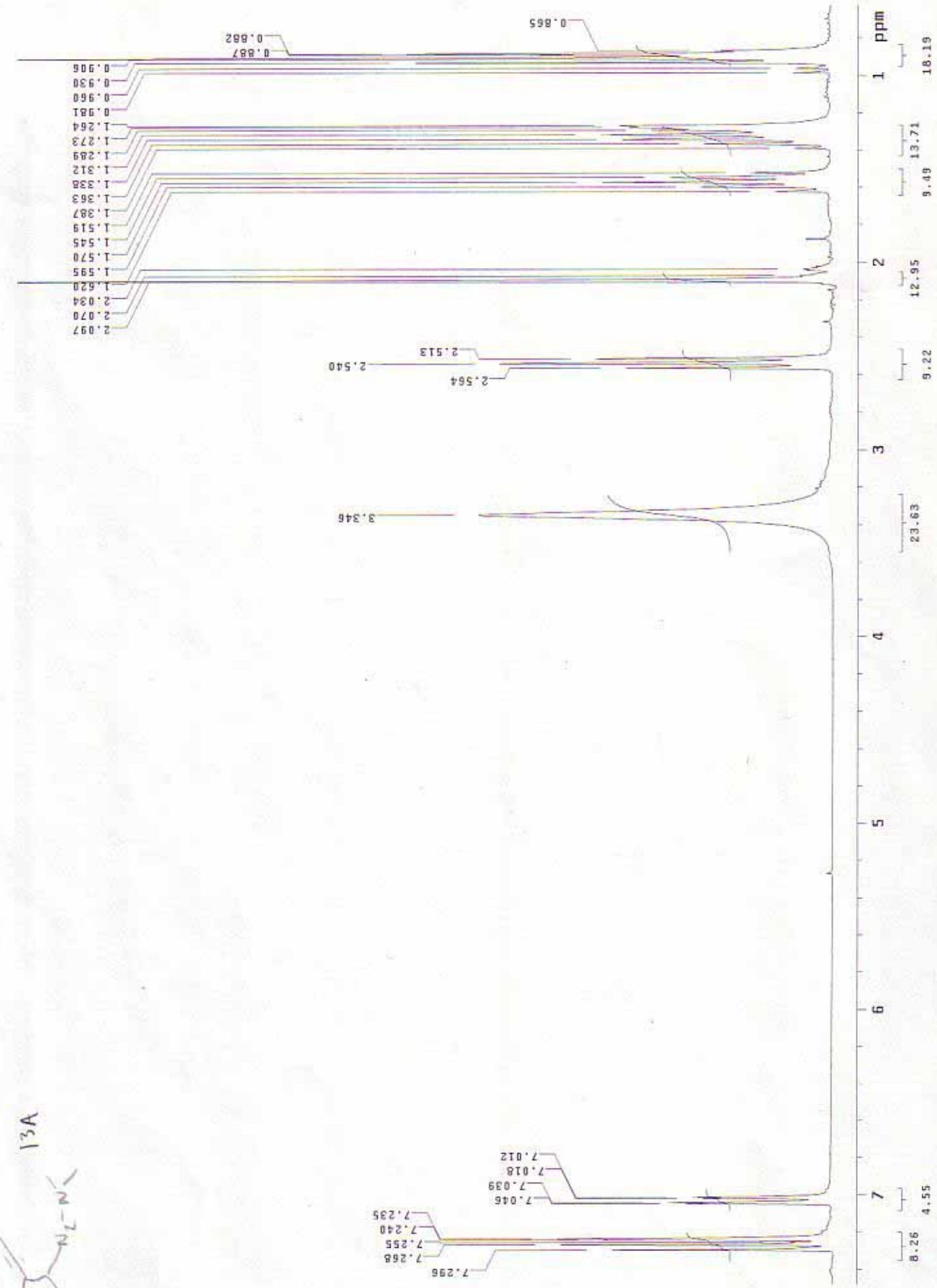
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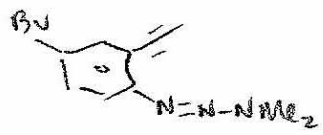
13A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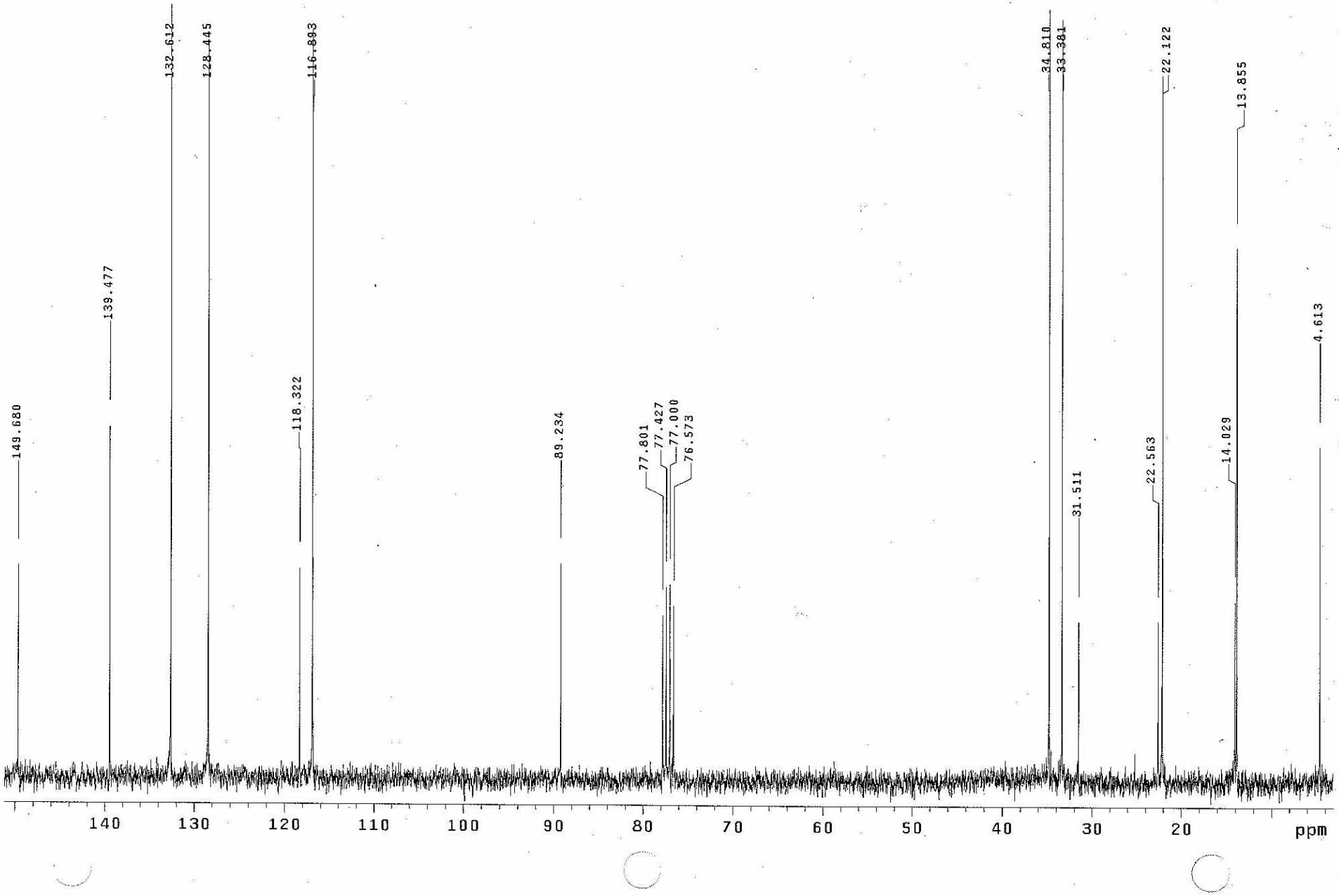
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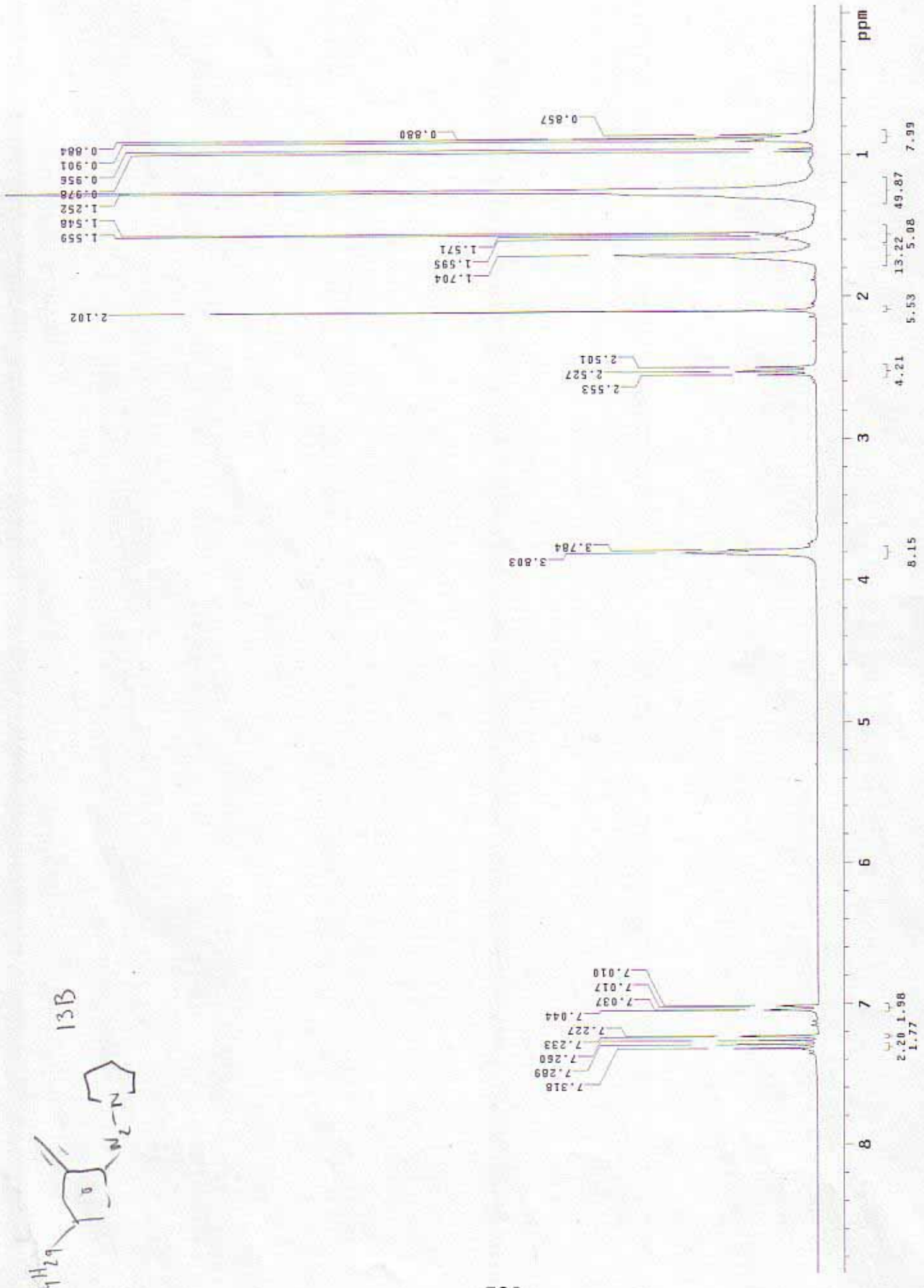
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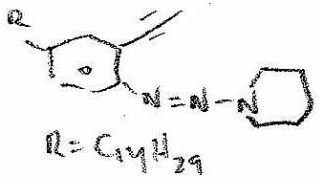
13B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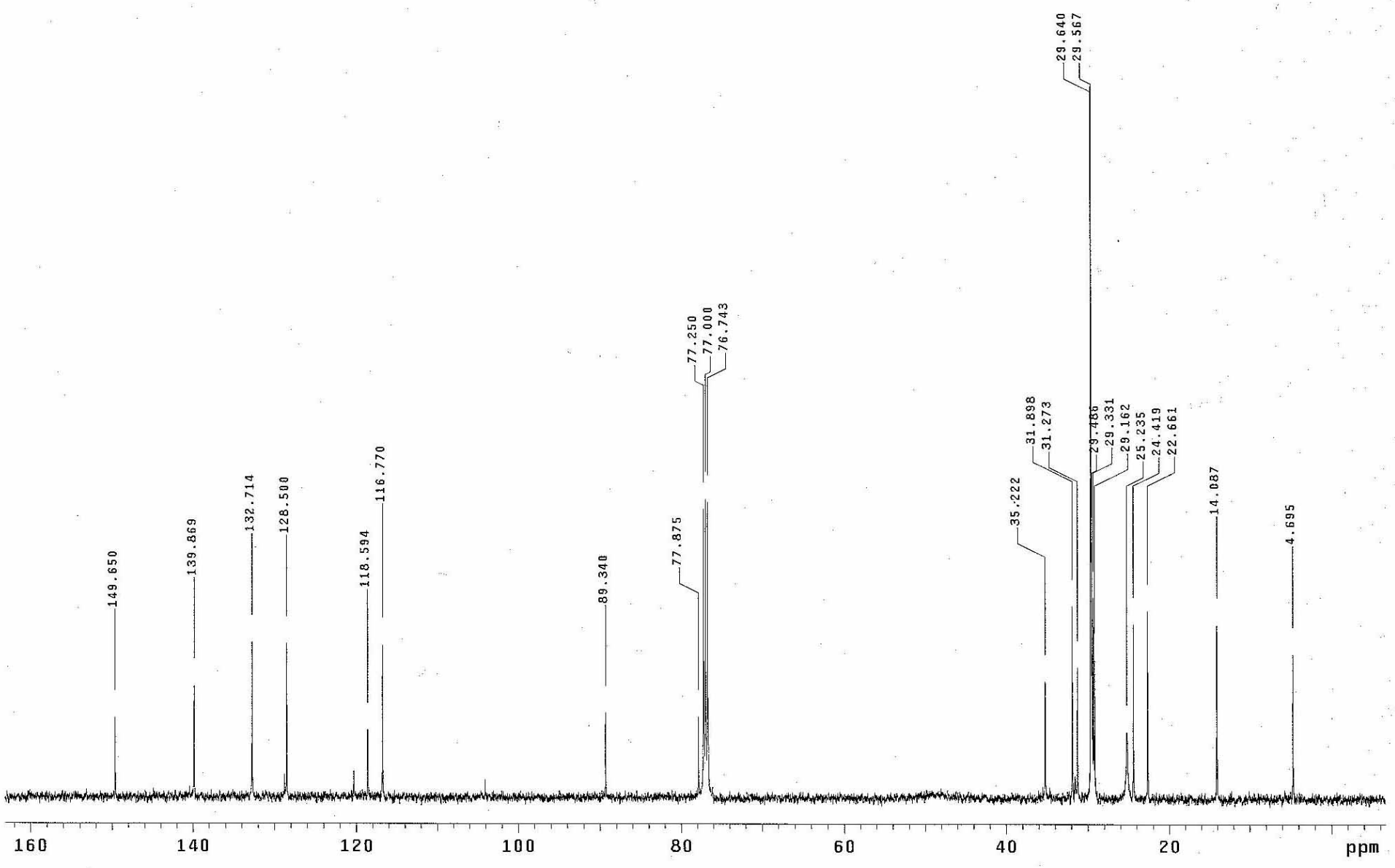
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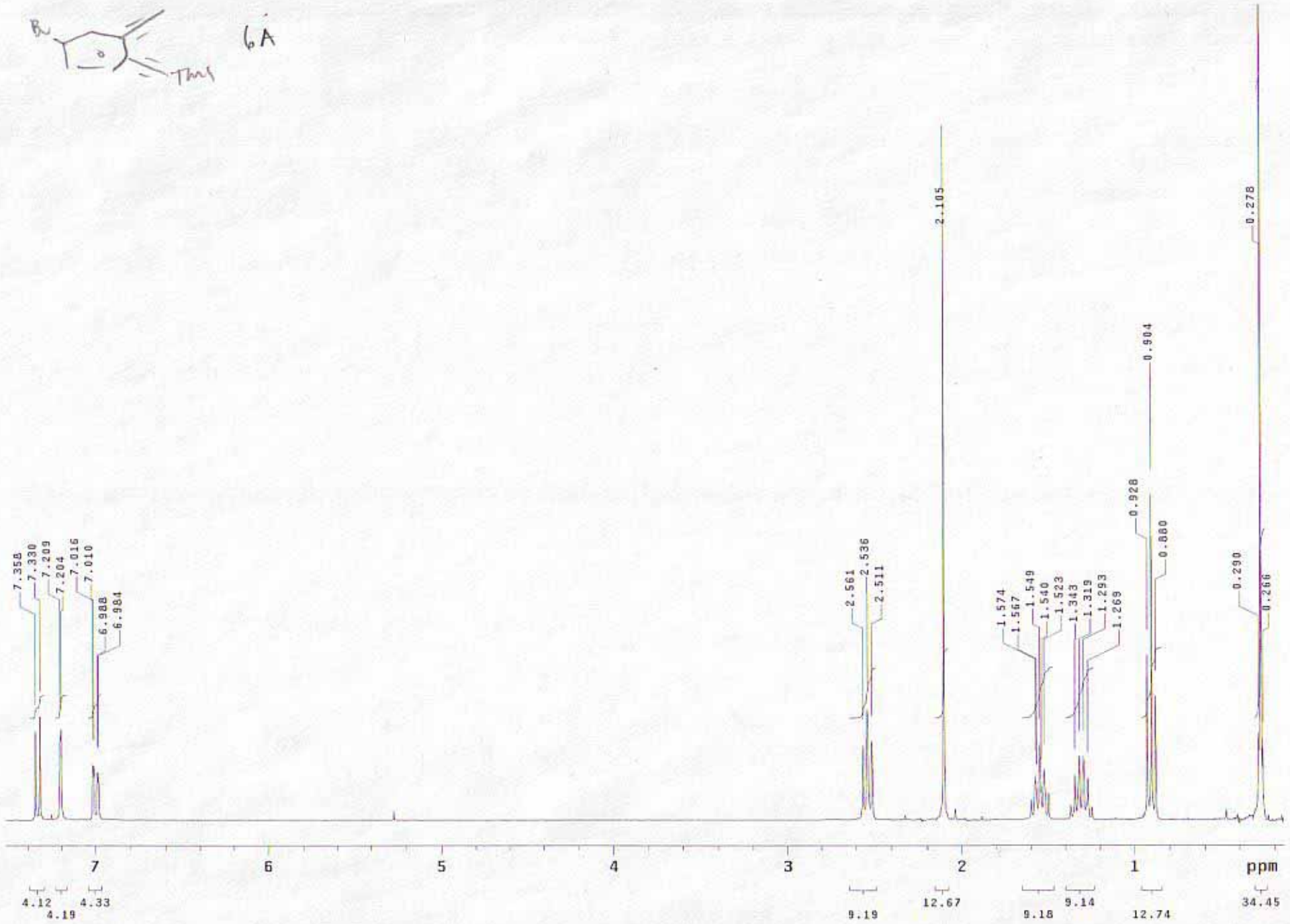
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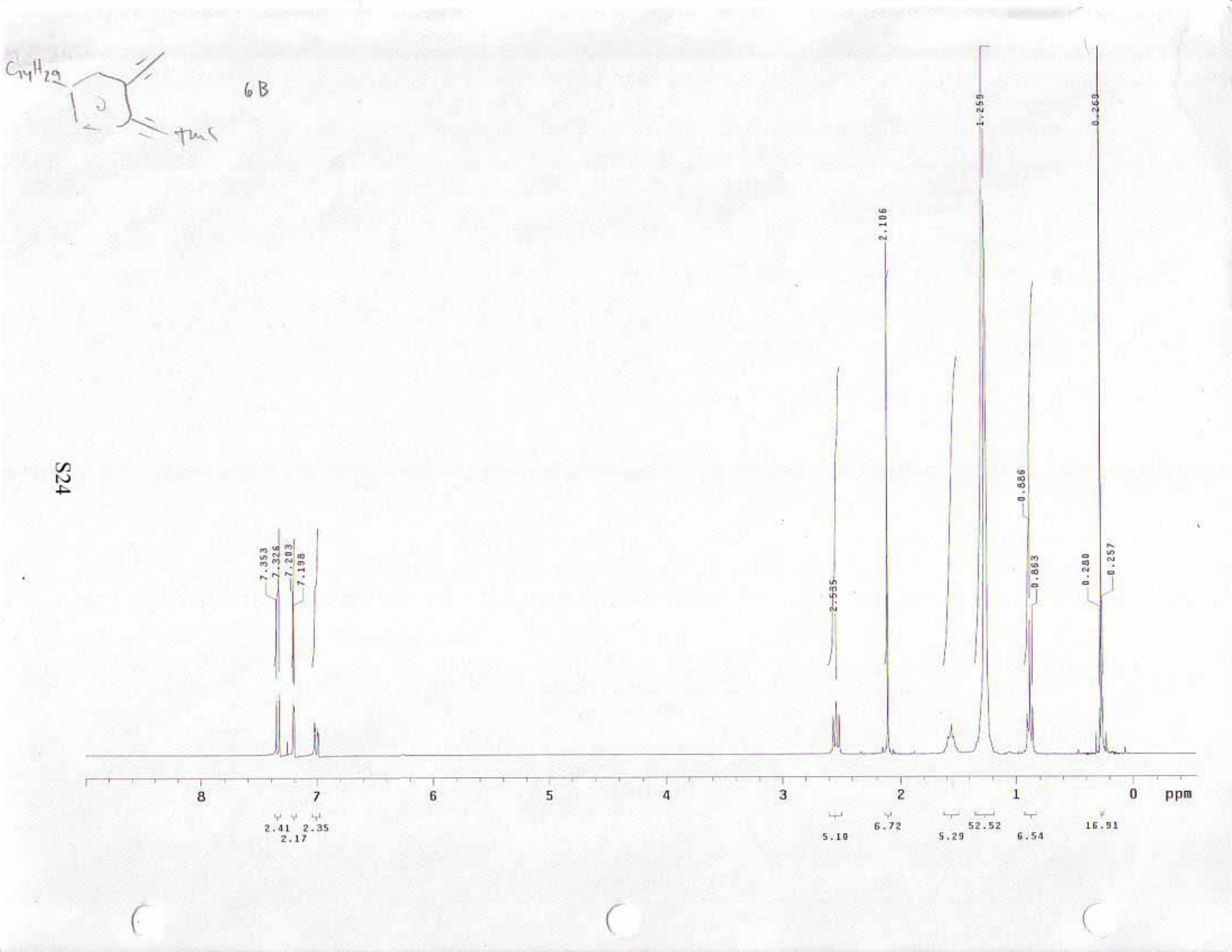
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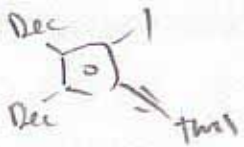
2.17
2.35
5.10
6.72
5.29
52.52
6.54
16.91

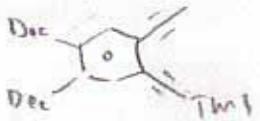
0.024
4.431
14.126
22.687
29.110
29.364
29.444
29.551
29.658
30.993
31.928
35.614
76.586
77.000
77.427
78.549
89.674
97.005
104.070
122.700
126.826
127.534
131.540
131.741
143.319
0 ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140


Dec
Dec
O
TMS

14

7.579
7.250
1.314
1.274
0.914
0.892
0.869
0.282
8
7
6
5
4
3
2
1
0
ppm
1.84
1.97
7.71
8.78
53.65
10.79
15.27

-0.123
14.126
22.687
29.337
29.471
29.564
29.604
30.913
31.901
32.088
32.222
76.573
77.000
77.427
97.165
97.660
106.901
126.679
133.303
138.965
140.635
143.332
0 ppm
20
40
60
80
100
120
140
160
180
200
TMS
D-De


Dec
Dec
Thf

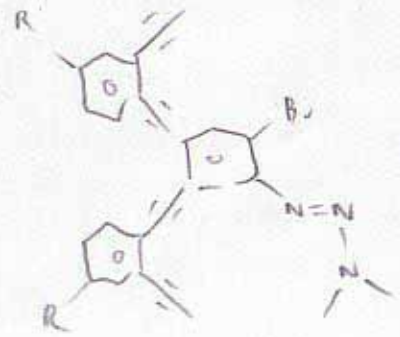
6C

7.260
7.213
7.151
2.539
2.514
2.487
2.091
1.545
1.519
1.301
1.260
0.901
0.880
0.857
0.260
0.248
8
7
6
5
4
3
2
1
0
ppm
1.51
1.59
7.22
5.59
8.67
50.34
11.50
13.59

0.051
4.444
14.113
22.687
29.337
29.498
29.564
29.604
29.658
30.820
30.886
31.901
32.329
32.396
76.573
77.000
77.414
78.522
88.792
96.511
104.350
122.593
124.182
132.435
132.168
140.207
141.276
0
20
40
60
80
100
120
140
160
180
200
ppm

R= Dec

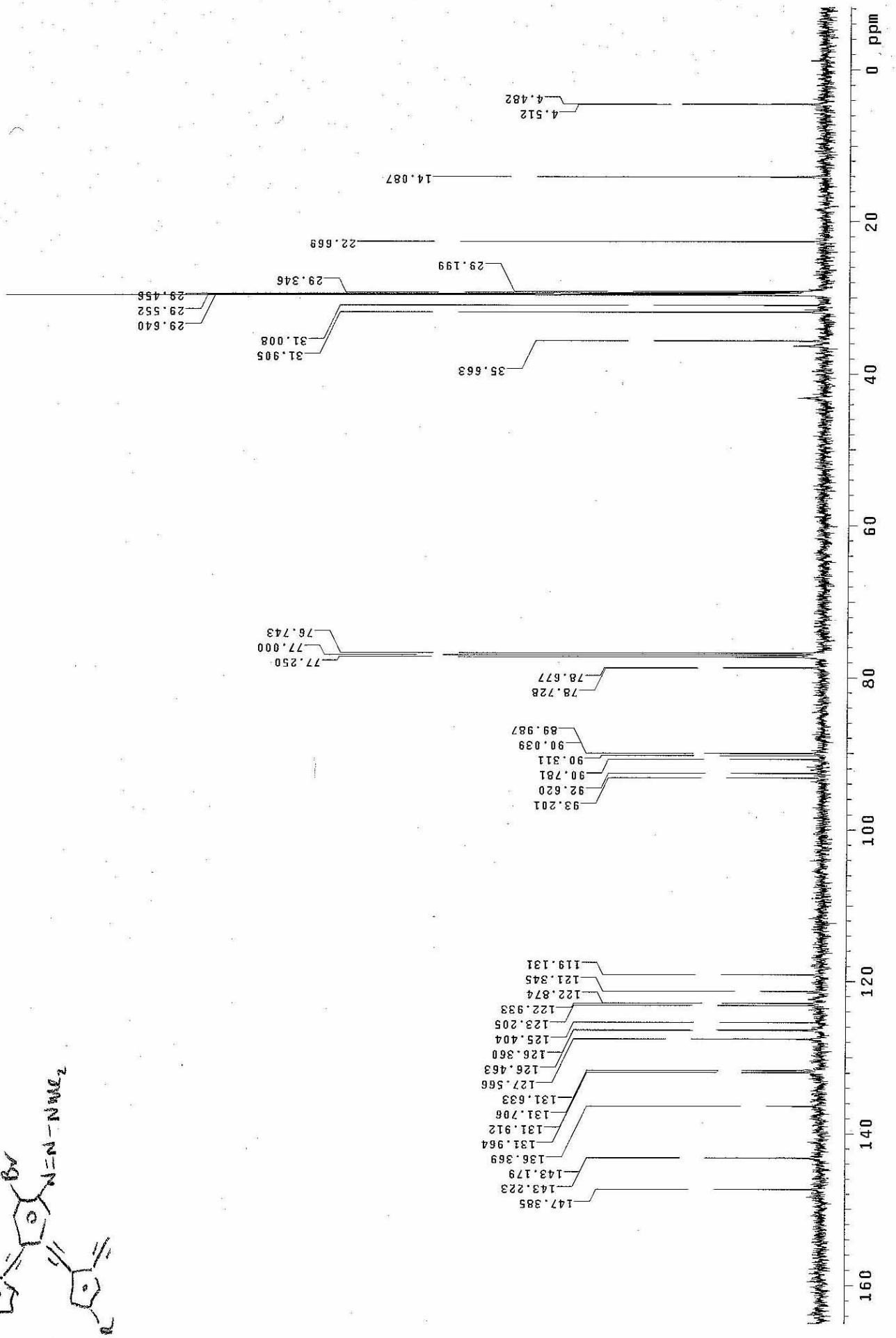
1c

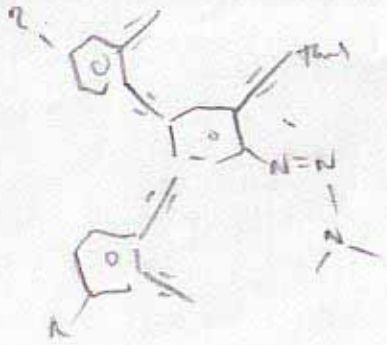


16

R = $C_{14}H_{29}$

S30

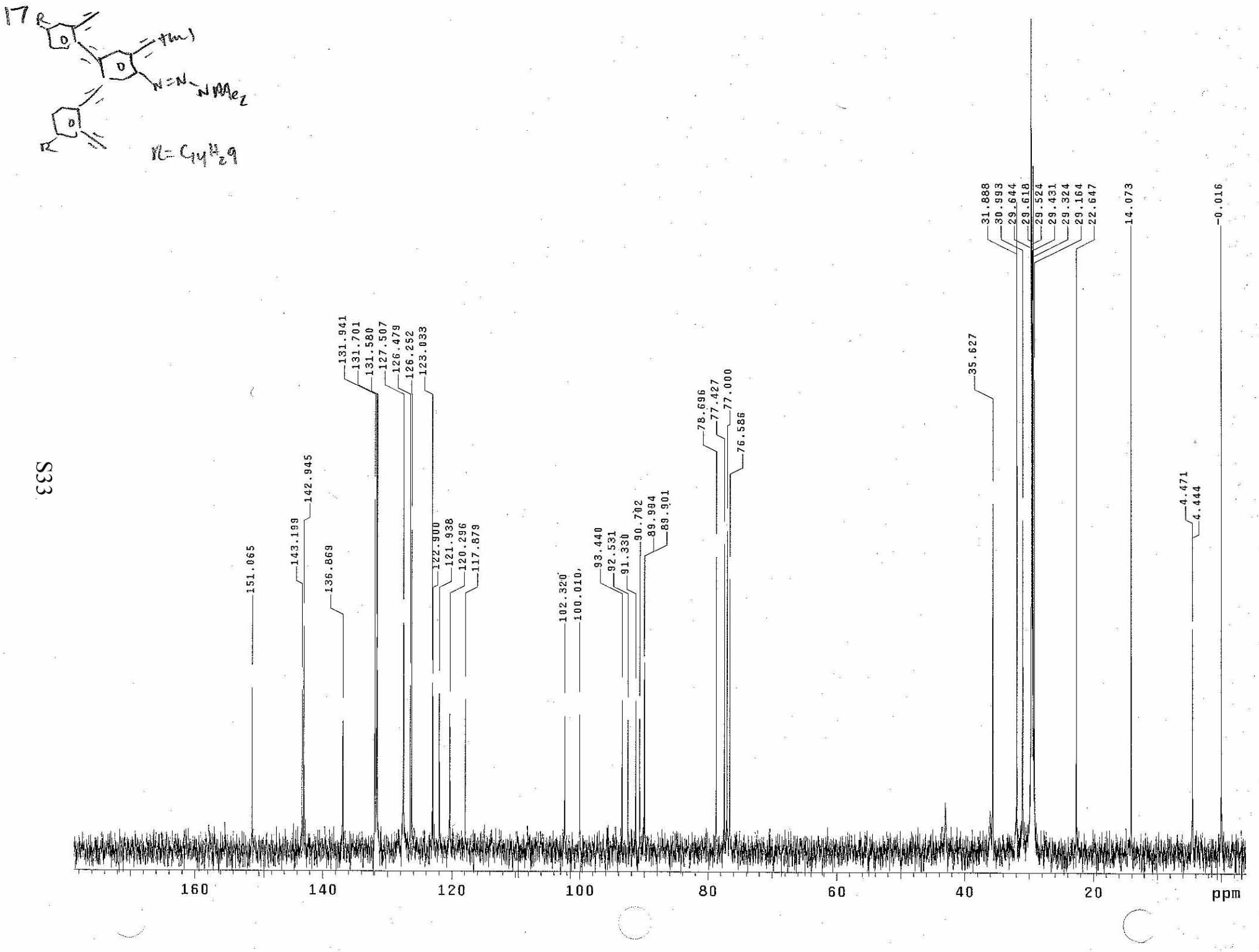

4.482
4.512
14.087
22.669
29.199
29.346
29.456
29.552
29.640
31.008
31.905
35.663
76.743
77.000
77.250
78.677
78.728
89.987
90.039
90.311
90.781
92.620
93.201
119.131
121.345
122.874
122.933
123.205
125.404
126.360
126.463
127.566
131.633
131.706
131.912
131.964
136.369
143.179
143.223
147.385
0 ppm
20
40
60
80
100
120
140
160

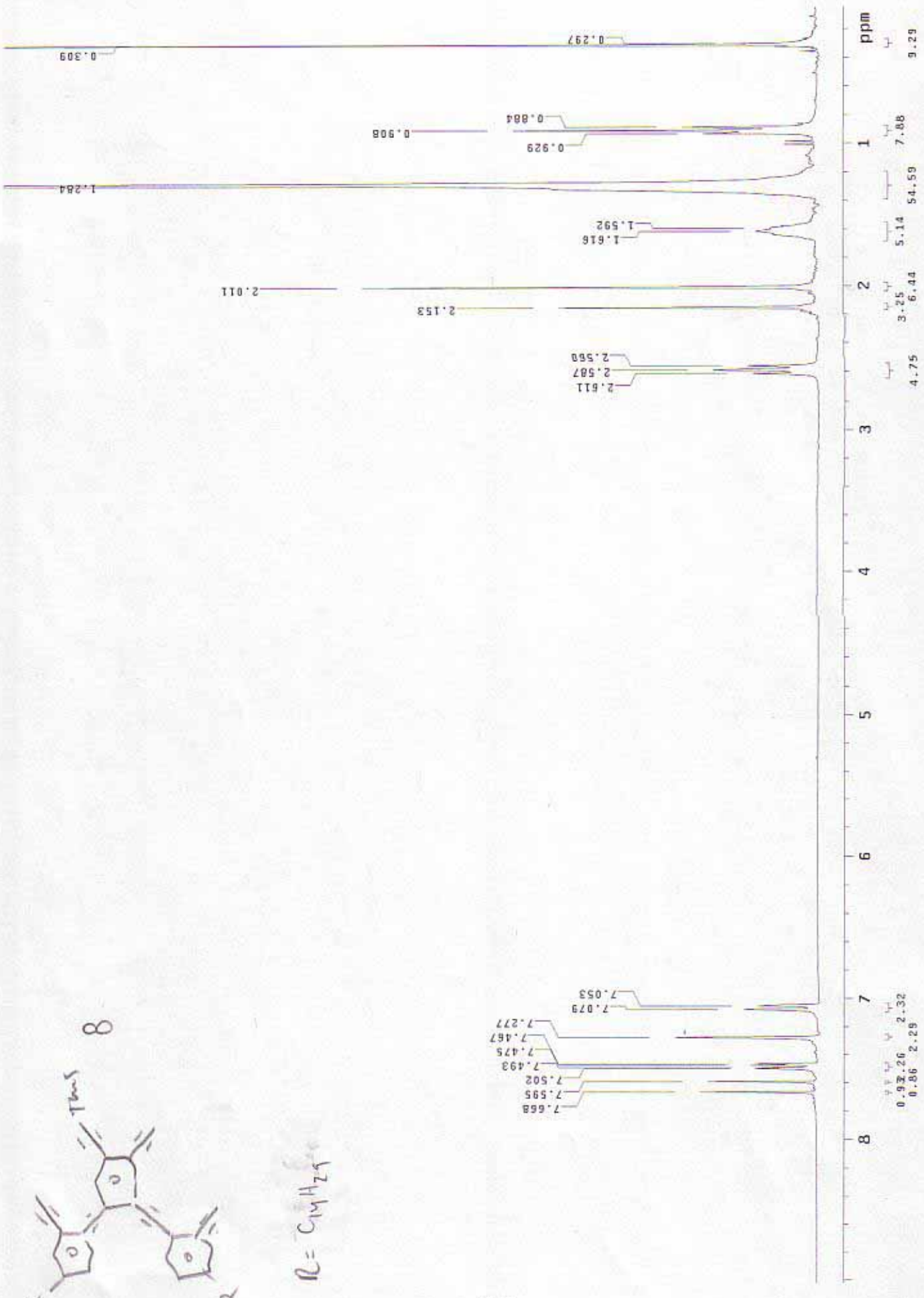


17

R = $C_{14}H_{29}$

7.707
7.693
7.504
7.485
7.478
7.458
7.267
7.260
7.066
7.039
3.562
3.286
2.598
2.573
2.547
2.011
1.625
1.604
1.579
1.577
1.310
1.269
1.223
0.912
0.891
0.868
0.281
0.273
0.270
0.260
0.257
9
8
7
6
5
4
3
2
1
0 ppm
1.94
2.31
2.44
2.22
7.82
4.78
6.18
4.97
50.68
6.64
10.02

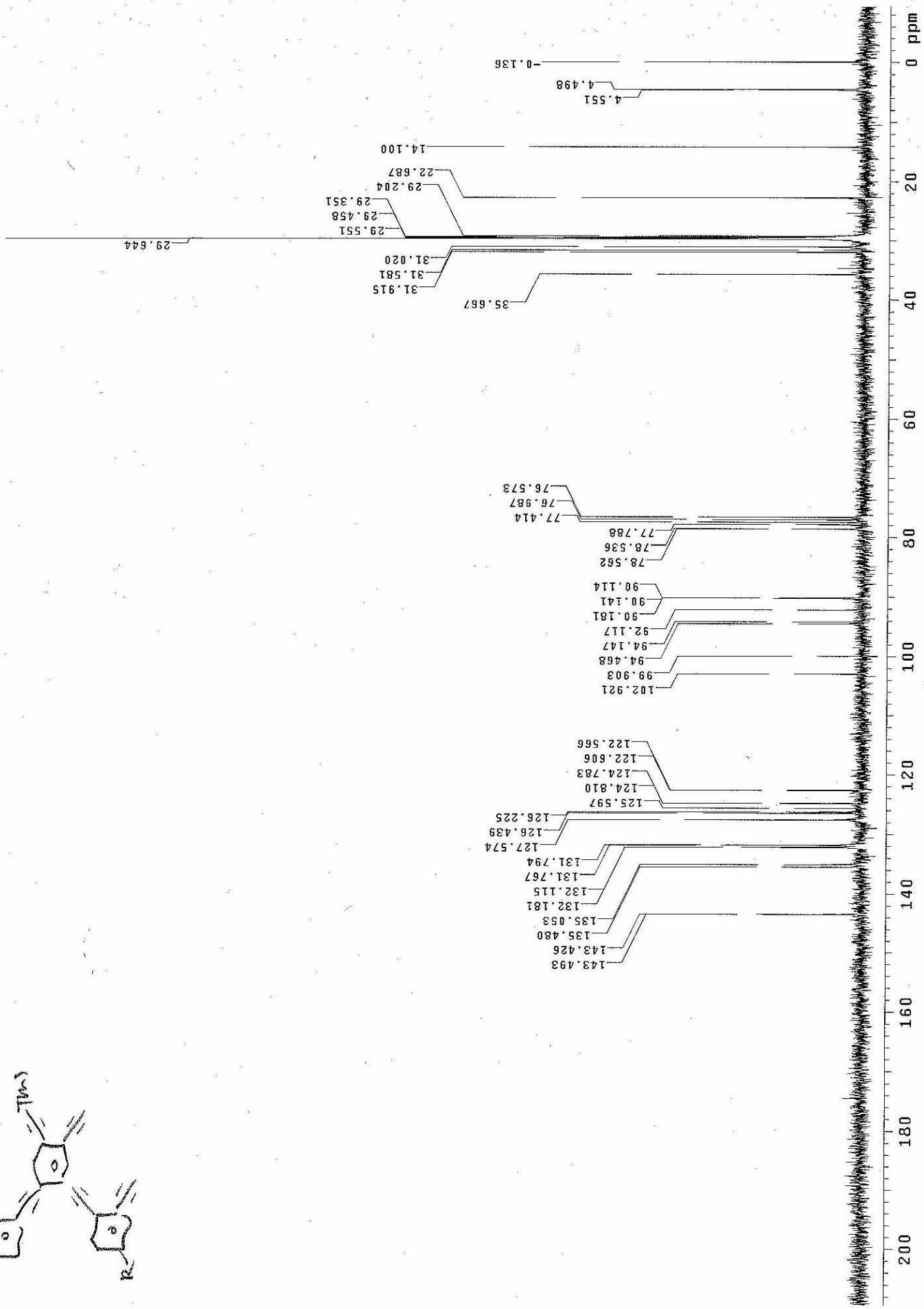

17
R
O
O
O
Tbt
N=N-NMe2
R
R= C14H29
151.065
143.199
142.945
136.869
131.941
131.701
131.580
127.507
126.479
126.252
123.033
122.900
121.938
120.296
117.879
102.320
100.010
93.440
92.531
91.330
90.702
89.994
89.901
78.696
77.427
77.000
76.586
35.627
31.888
30.993
29.644
29.618
29.524
29.431
29.324
29.164
22.647
14.073
4.471
4.444
-0.016
160
140
120
100
80
60
40
20
ppm

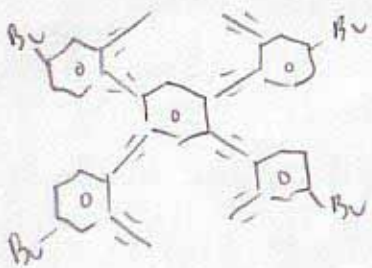

0.297
0.309
0.884
0.908
0.929
1.284
1.592
1.616
2.011
2.153
2.560
2.587
2.611
7.053
7.079
7.277
7.467
7.475
7.493
7.502
7.595
7.668
ppm
9.29
7.88
54.59
5.14
6.44
3.25
4.75
2.32
2.29
3.26
0.93
0.86

TMS
R
R

8

R = $C_{14}H_{29}$

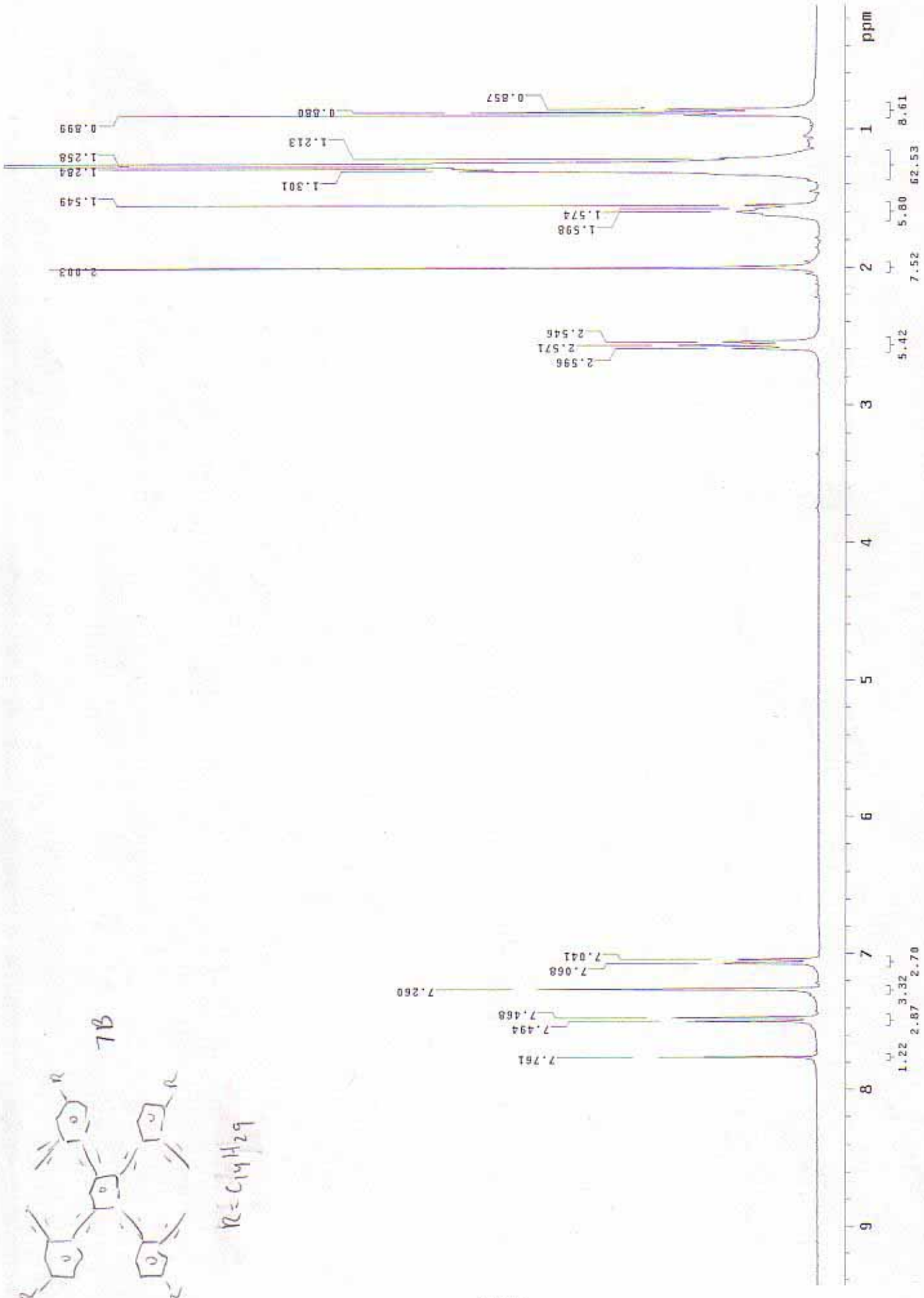

-0.136
4.498
4.551
14.100
22.687
29.204
29.351
29.458
29.551
29.644
31.020
31.581
31.915
35.667
76.573
76.987
77.414
77.788
78.536
78.562
90.114
90.141
90.181
92.117
94.147
94.468
99.903
102.921
122.566
122.606
124.783
124.810
125.597
126.225
126.439
127.574
131.767
131.794
132.115
132.181
135.053
135.480
143.426
143.493
0 ppm
20
40
60
80
100
120
140
160
180
200



7A

7.769
7.502
7.476
7.274
7.270
7.260
7.080
7.075
7.054
7.049
2.612
2.587
2.561
2.009
1.622
1.595
1.587
1.570
1.545
1.385
1.361
1.336
1.311
0.954
0.929
0.905
7
6
5
4
3
2
1
ppm
2.84
6.43
6.69
6.44
12.91
18.45
13.29
13.99
18.95

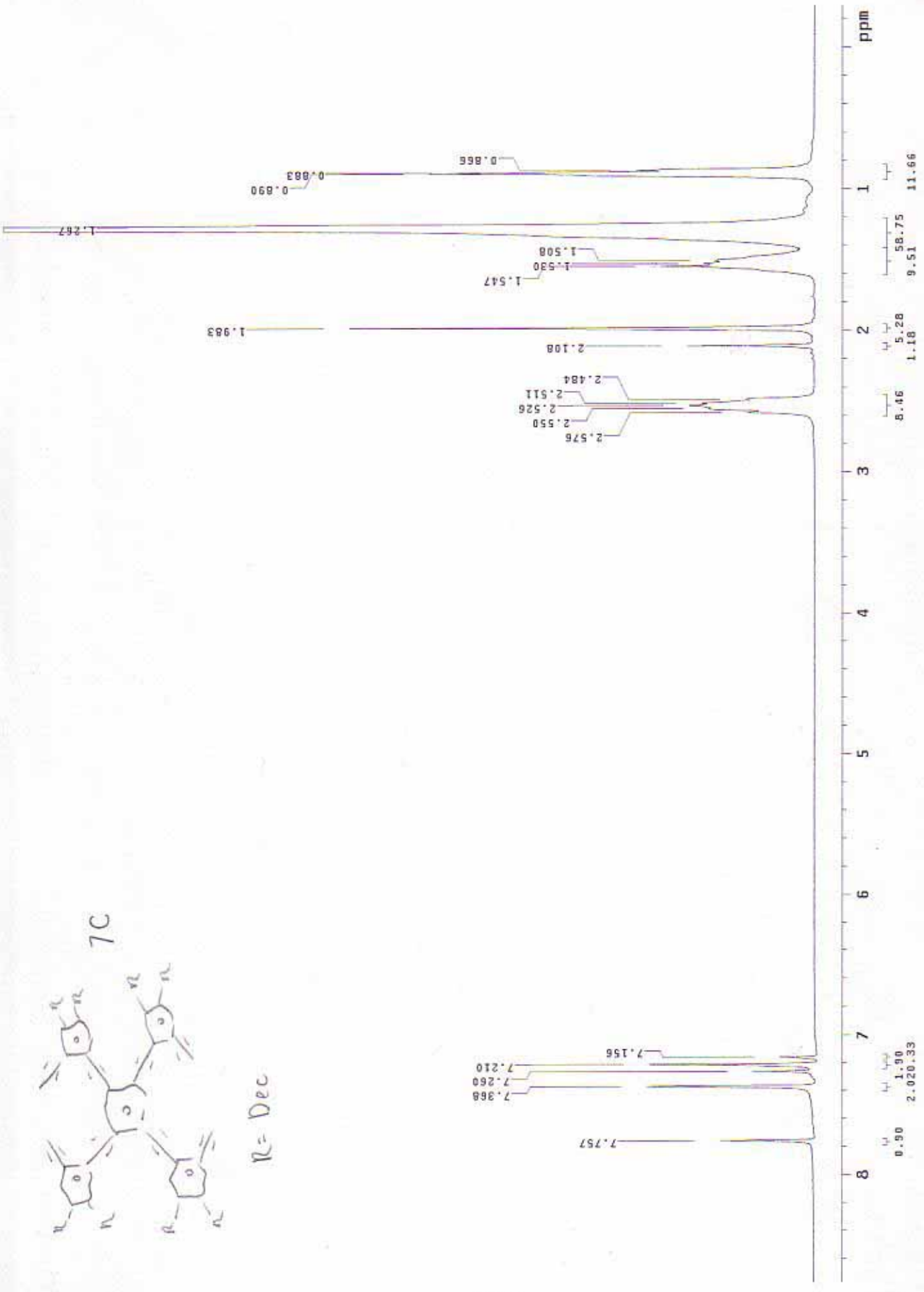
4.547
13.922
22.243
33.167
35.358
76.586
77.013
77.427
78.576
90.209
90.382
94.349
122.622
125.227
126.429
127.657
131.837
132.185
135.630
143.483
R= tBu
ppm


7B
R = C14H29
7.761
7.494
7.468
7.260
7.068
7.041
2.596
2.571
2.546
2.003
1.598
1.574
1.549
1.301
1.284
1.258
1.213
0.899
0.880
0.857
ppm
1.22
2.87
3.32
2.70
5.42
7.52
5.80
62.53
8.61

4.551
14.126
22.687
29.217
29.364
29.471
29.564
29.658
31.060
31.915
35.707
76.573
77.000
77.427
78.603
90.208
90.395
94.348
122.633
125.237
126.439
127.654
131.821
132.181
135.640
143.533
ppm
0
20
40
60
80
100
120
140
160
180
200

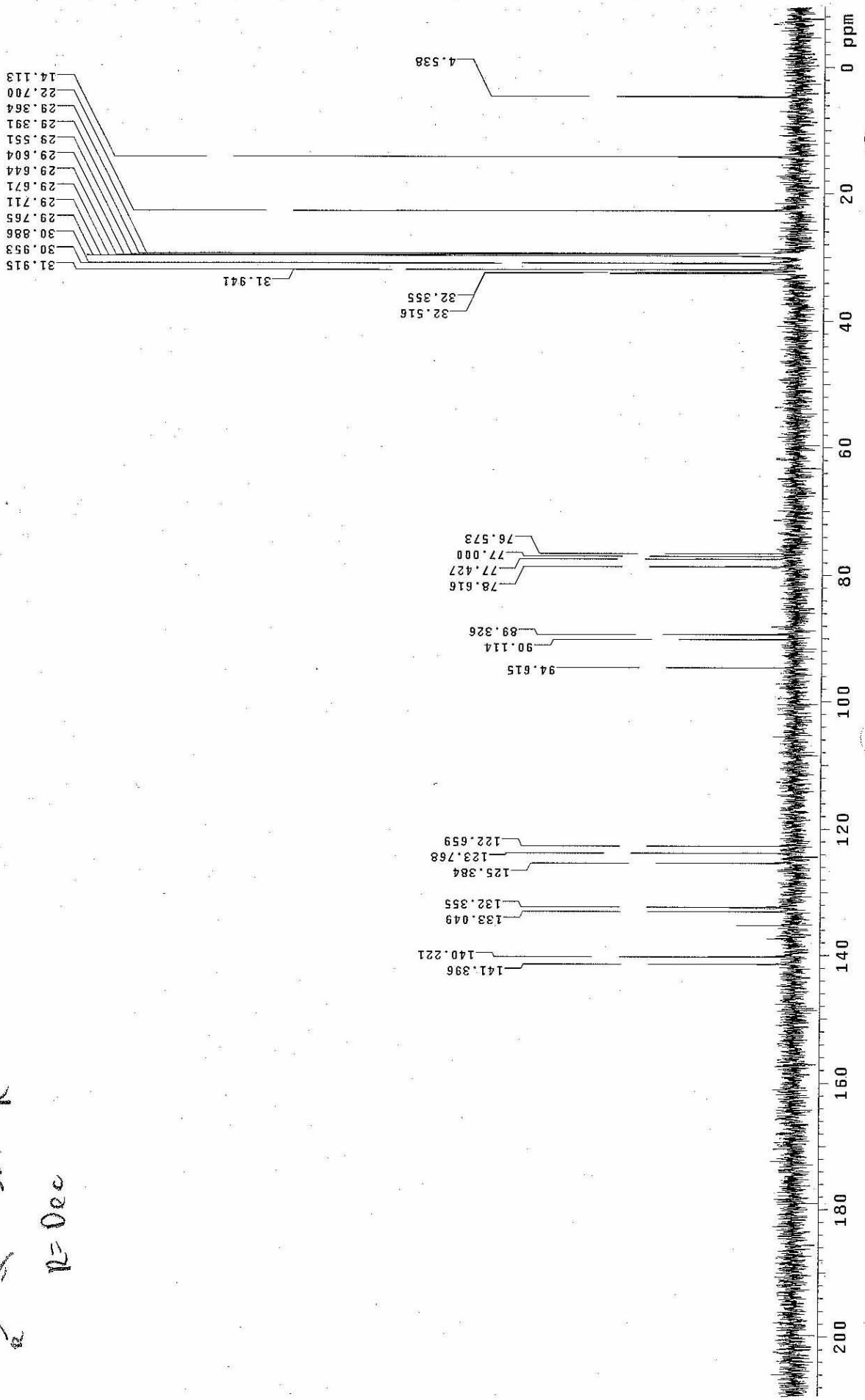
R = $C_{12}H_{25}$

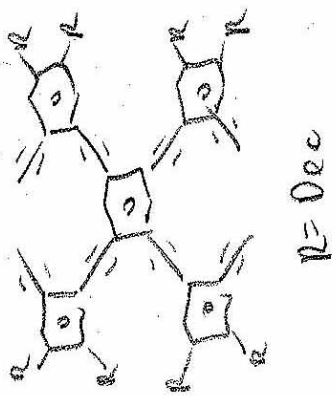
7B


ppm
0.866
0.883
0.890
1.267
1.508
1.530
1.547
1.983
2.108
2.484
2.511
2.526
2.550
2.576
7.156
7.210
7.260
7.368
7.757
0.90
1.90
2.020.33
8.46
5.28
1.18
58.75
9.51
11.66

7C

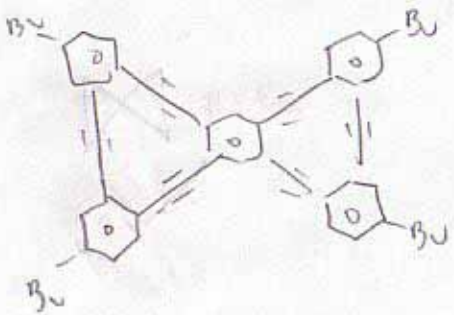
R= Dec


4.538
14.113
22.700
29.364
29.391
29.551
29.604
29.644
29.671
29.711
29.765
30.886
30.953
31.915
31.941
32.355
32.516
76.573
77.000
77.427
78.616
89.326
90.114
94.615
122.659
123.768
125.384
132.355
133.049
140.221
141.396
0 ppm
20
40
60
80
100
120
140
160
180
200

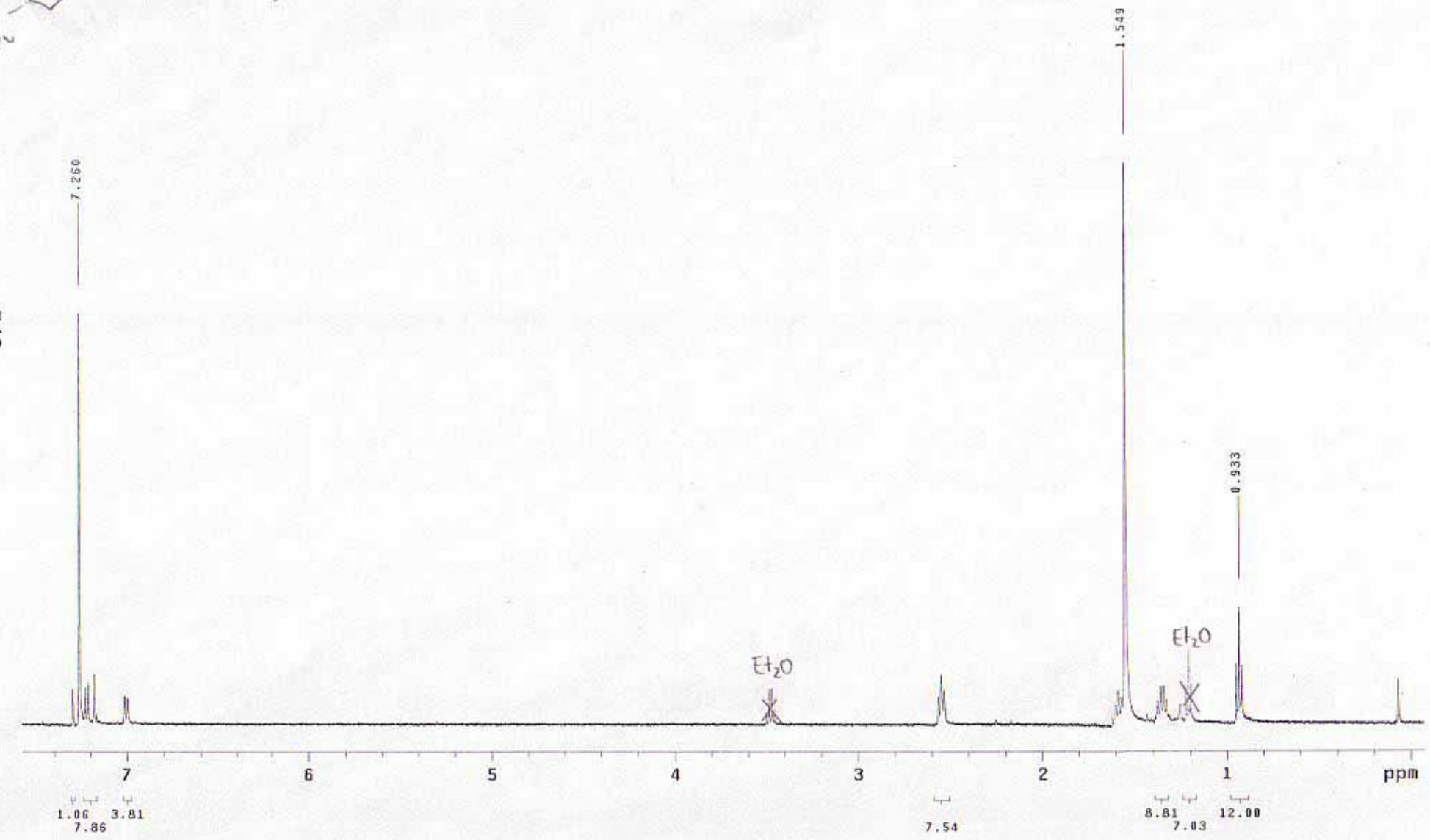


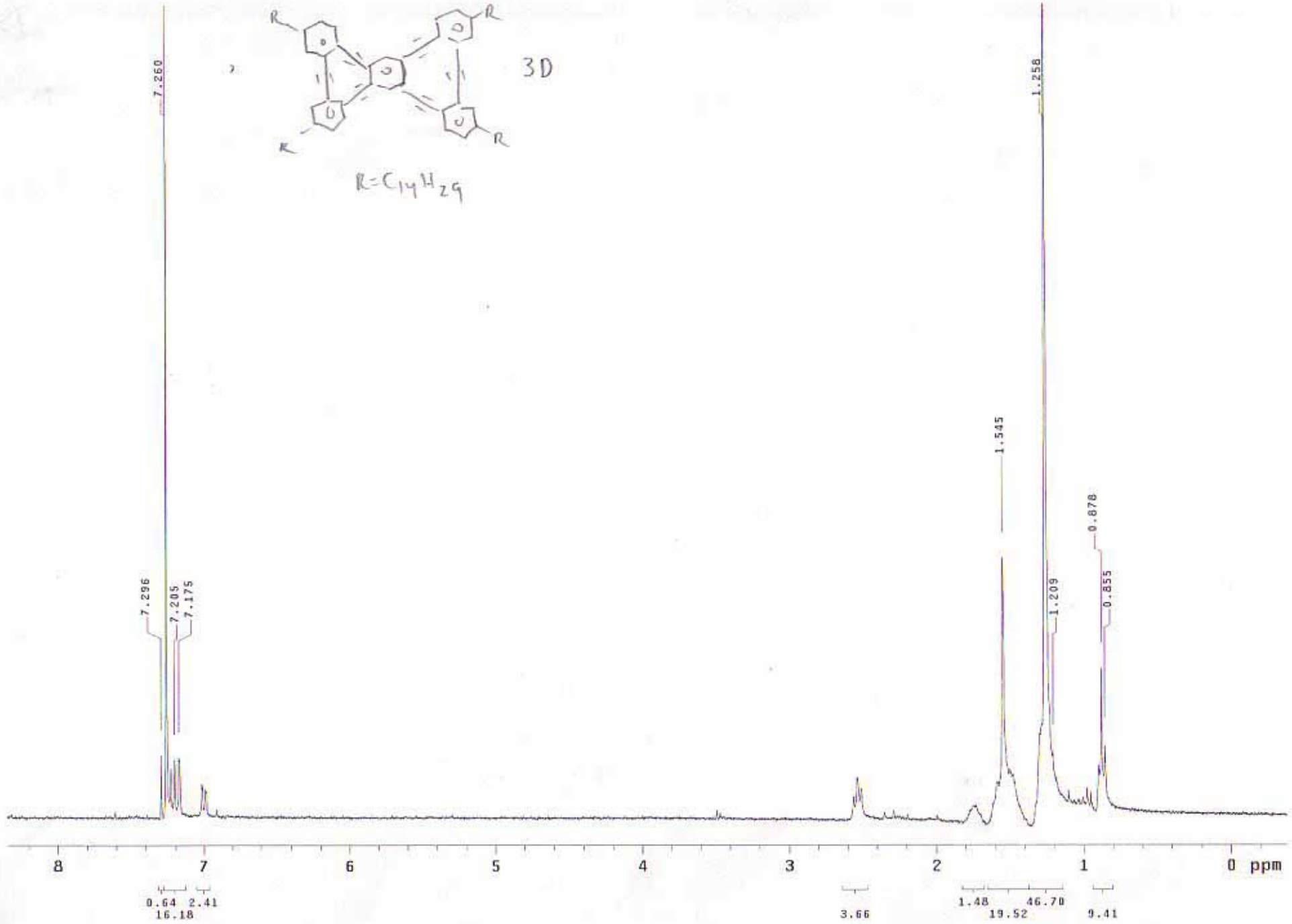
R= Oec

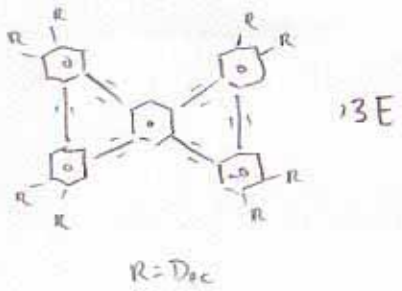
1C



3C

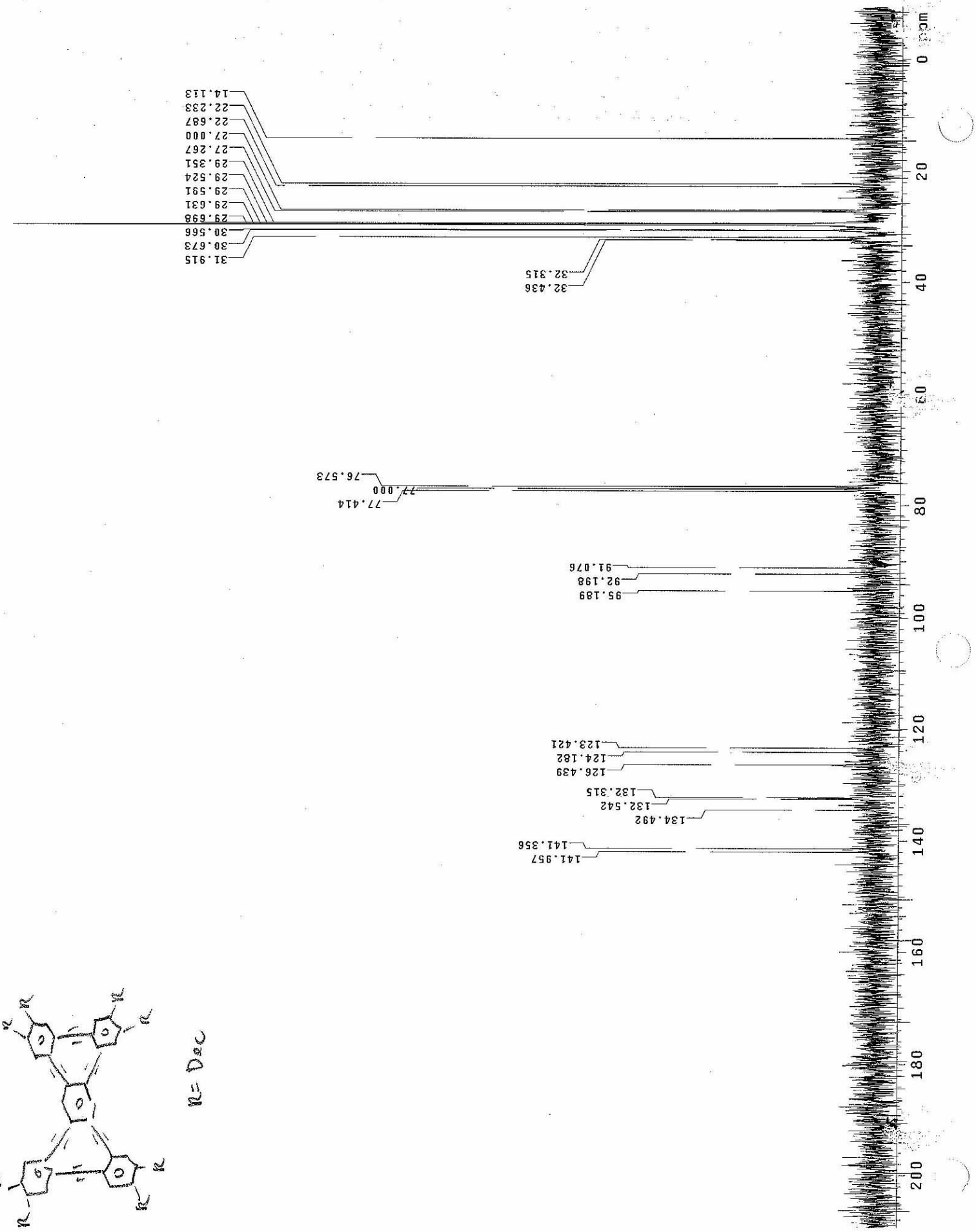

7.260
1.549
0.933
Et2O
Et2O
7
6
5
4
3
2
1
ppm
1.06
7.86
3.81
7.54
8.81
7.03
12.00


3D
R=C14H29
7.260
7.296
7.205
7.175
1.545
1.258
1.209
0.878
0.855
8
7
6
5
4
3
2
1
0 ppm
0.64
2.41
16.18
3.66
1.48
46.70
19.52
9.41


R
R
R
R
R
R
R
R
R
R
R=Doc

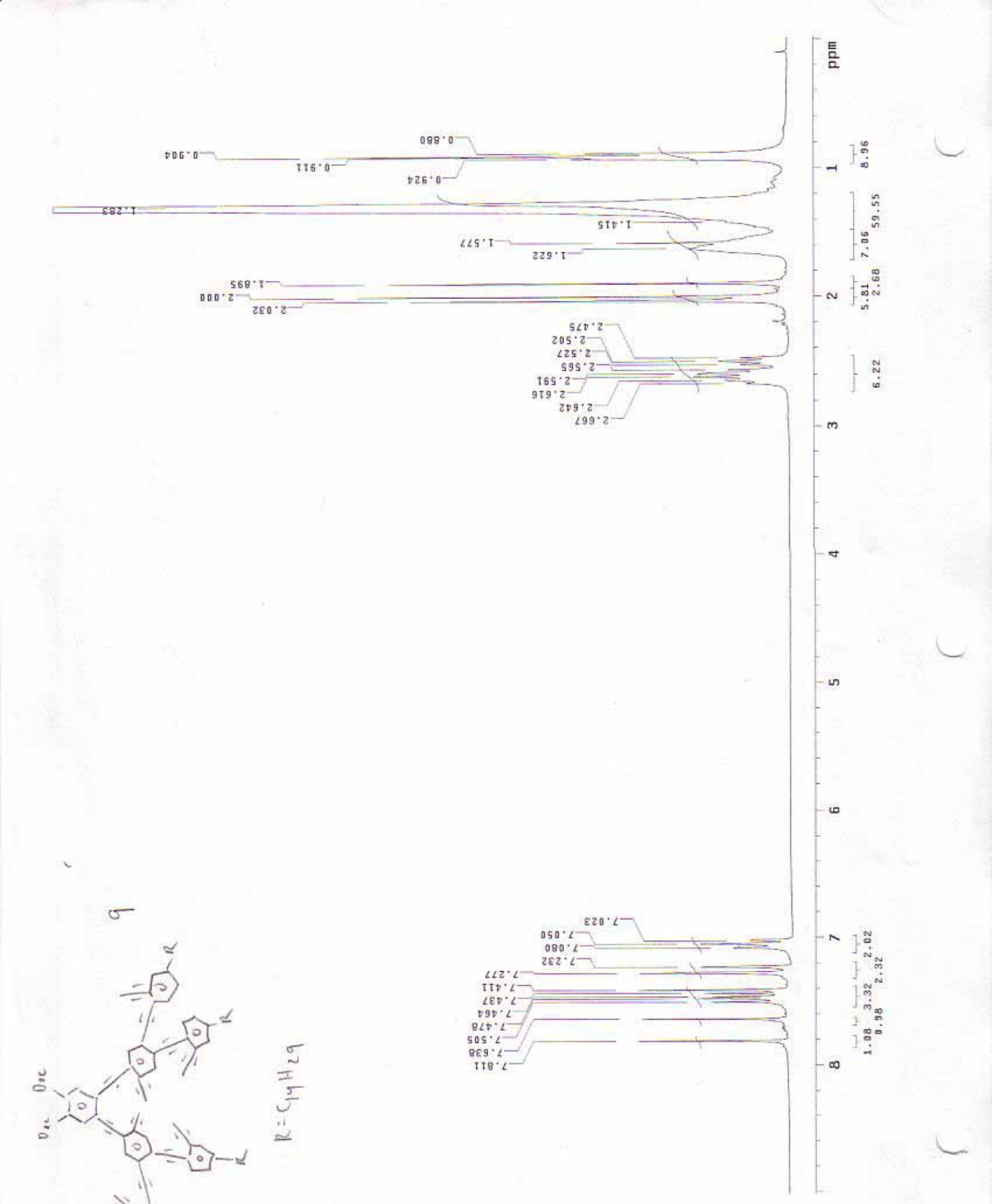
3E

7.276
7.260
7.108
7.069
2.535
2.512
2.485
1.548
1.282
0.908
0.890
0.866
9
8
7
6
5
4
3
2
1
0
ppm
1.12
1.52
0.48
3.85
8.59
10.39
62.49
12.55

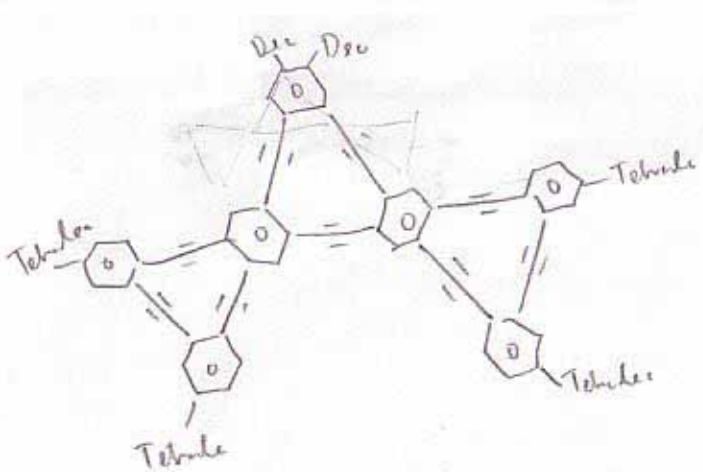

14.113
22.233
22.687
27.000
27.267
29.351
29.524
29.591
29.631
29.698
30.566
30.673
31.915
32.315
32.436
76.573
77.000
77.414
91.076
92.198
95.189
123.421
124.182
126.439
132.315
132.542
134.492
141.356
141.957
200
180
160
140
120
100
80
60
40
20
0
ppm

R= Dec

3E


0.880
0.904
0.911
0.924
1.283
1.415
1.577
1.622
1.895
2.000
2.032
2.475
2.502
2.527
2.565
2.591
2.616
2.642
2.667
7.023
7.050
7.060
7.232
7.277
7.411
7.437
7.464
7.478
7.505
7.636
7.811
ppm
8.96
59.55
7.06
2.68
5.81
6.22
2.02
2.32
3.32
1.08
0.98
R=C14H29
9

4.711
4.511
4.404
14.113
22.687
29.244
29.364
29.484
29.591
29.650
30.886
31.007
31.915
32.489
35.707
76.573
77.000
77.427
77.988
78.576
90.168
90.234
90.515
90.835
92.411
93.667
94.187
122.753
124.863
125.224
125.784
126.385
127.614
131.714
132.261
132.983
135.373
135.654
141.583
143.332
143.413
ppm
20
40
60
80
100
120
140
160
180


Dec
Dec
Tebutle
Tebutle
Tebutle
Tebutle

4

7.260
7.205
7.184
7.166
7.152
7.066
2.526
1.556
1.264
0.894
0.880
0.857
9 8 7 6 5 4 3 2 1 0 ppm
4.67
1.92
0.92
6.07
13.97
62.27
10.19

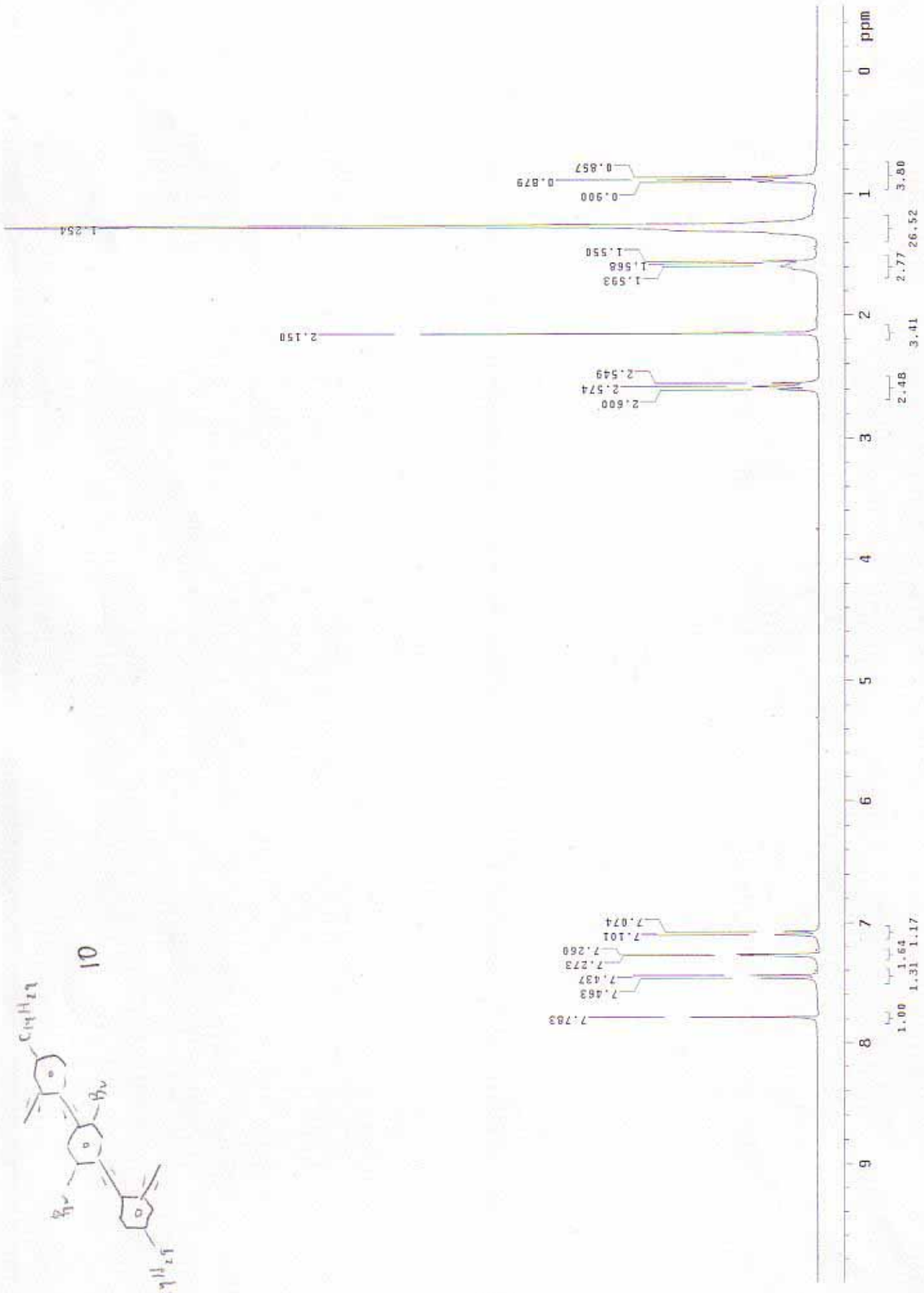
5 2

1

6

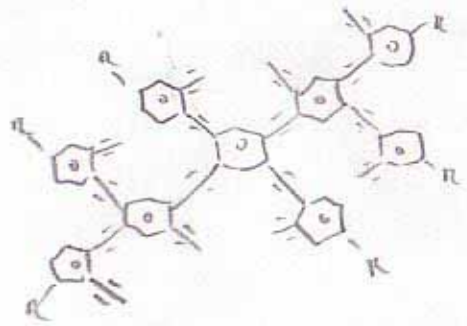
9

14.117
22.685
29.230
29.362
29.465
29.561
29.656
30.840
31.921
35.643
76.738
76.995
77.253
91.123
91.439
91.498
92.638
92.734
95.050
95.205
95.352
123.592
125.886
126.431
126.644
126.725
126.923
128.857
131.946
142.183
144.044
0
20
40
60
80
100
120
140
160
ppm


7.783
7.463
7.437
7.273
7.260
7.101
7.074
2.600
2.574
2.549
2.150
1.593
1.568
1.550
1.254
0.900
0.879
0.857
ppm
0
1
2
3
4
5
6
7
8
9
1.00
1.64
1.31
1.17
2.48
3.41
2.77
26.52
3.80

10

4.898
14.140
22.700
29.177
29.364
29.444
29.538
29.658
31.007
31.928
35.707
76.586
77.000
77.427
78.509
89.380
90.315
95.990
121.765
123.154
126.532
126.612
127.788
132.048
136.335
144.187
0 ppm
20
40
60
80
100
120
140
160



11

$R = C_{14}H_{29}$

7.798
7.280
2.088
2.053
2.029
1.865
1.568
1.323
1.280
0.900
0.877
9
8
7
6
5
4
3
2
1
ppm
1.52 2.77 2.53
0.80 3.32
5.34
7.38
2.41
6.26
59.20
8.47

4.372
4.563
4.637
14.109
22.691
29.287
29.361
29.471
29.684
30.949
31.045
31.927
35.715
76.743
77.000
77.250
77.846
78.486
78.568
78.677
90.090
90.186
90.355
90.436
92.715
93.664
94.216
94.730
94.995
122.323
122.610
122.690
124.720
124.919
125.176
125.610
125.766
125.911
126.492
126.595
127.529
127.640
127.882
131.831
131.949
132.214
135.611
135.810
143.480
143.598
143.818
ppm
0
20
40
60
80
100
120
140
160

R=C14H29