## EXPERIMENTAL

All reagents were obtained from the Aldrich Chemical Co. (Milwaukee, WI) or Acros Organics (Pittsburgh, PA) unless stated otherwise. $Et_2O$ and THF were purified by passage through activated alumina columns under nitrogen. Moisture sensitive reactions were carried out in flame dried glassware under $N_2$. TLC was carried out on *MERCK Silica Gel 60* thin layer plates. Silica gel chromatography was performed on *Fisher Brand* silica gel (170-400 mesh). Elemental analysis was performed by Desert Analytics, Tucson AZ.

1,2-Bis(trimethylsilylethynyl)-benzene[1] and 1,2 diethynyl-benzene[2] were prepared according to the literature; spectral data matched accordingly.

**1,2-Bis(bromoethynyl) benzene.** To a solution of 1,2-bis(trimethylsilylethynyl) benzene ( 5.73 g, 21.2 mmol) in 100ml acetone was added N-bromosuccinamide (9.42 g, 52.9 mmol) and $AgNO_3$ ( 0.3 g, 1.77mmol). The suspension was stirred at 25ºC for 10 h, after which the solvent was removed via rotary evaporation. The substrate was passed through a pad of silica gel with hexanes. The solvent was evaporated to yield 6.01 g (100 %) of dibromide as an unstable red oil that was subjected to cycloaromatization immediately.

**2,3-Dibromonaphthalene.** A solution of 1,2-Bis(bromoethynyl)benzene (0.5 g, 1.76 mmol) was deoxygenated under $N_2$ in 45 ml benzene for 1 h. 1,4-Cyclohexadiene (4.5 ml, 10% v/v) was added and the mixture was sealed under $N_2$ in a steel bomb, which was heated to 180ºC for 2h (350 psi was the highest pressure reached). The benzene/cyclohexadiene mixture was recovered in vacuo, after which the residue was taken up in $CH_2Cl_2$ and passed through a pad of silica gel with 9:1 hexane/$CH_2Cl_2$ as eluent, to remove any polymeric / oligomeric materials. The solvent was evaporated and the resulting solid recrystallized from heptane to yield 0.35 g (70%) of a light yellow solid, mp 137-139ºC (lit 138-140ºC[4]). $^1$H NMR (200MHz, $CDCl_3$) $\delta$ 7.92 (s, 2H), 7.51 (m, 2H), 7.30 (m, 2H); $^{13}$C NMR (50MHz, $CDCl_3$) $\delta$ 133.0, 132.2, 127.2, 126.8, 121.9. MS m/z (rel intensity) 286 (100, $M^+$), 207(60, M-$Br^+$). Anal. Calcd for $C_{10}H_6Br_2$: C, 42.00; H, 2.11, found: C, 42.17; H, 2.01.

**2,3-Bis(trimethylsilylethynyl)naphthalene.** To a solution of trimethylsilyl acetylene (0.41 g, 4.2 mmol) in 5 ml THF cooled to -78 ºC under $N_2$ was added BuLi (4.3 mmol,1.74 ml, 2.46 M in hexanes). The mixture was allowed to warm to 25°C and 5 mmol (5 ml, 1M solution in $Et_2O$) $ZnCl_2$ was added quickly. After 15 min [1,1' bis(diphenylphosphino)-ferrocene]palladium(II)chloride (0.1 g, 2 mol %) and 2,3-dibromonaphthalene (0.2 g, 0.7 mmol) were added and the reaction heated to reflux. After 12 h, the reaction was cooled and extracted into hexanes, washed with water, dried, and passed through a short pad of silica gel with hexanes. The solvent was removed in vacuo to afford 0.18 g (81%) of a yellow solid, mp 92-93ºC. FTIR ($CCl_4$) 3055, 2959, 2151 $cm^{-1}$. $^1H$ NMR (200MHz, $CDCl_3$) δ 7.79 (s, 2 H), 7.42 (m, 2 H), 7.22 (m, 2 H), 0.13 (s, 18 H); $^{13}C$ NMR (50MHz, $CDCl_3$) δ 132.50, 132.19, 127.48, 127.24, 122.07, 103.42, 97.71, 0.09; MS m/z (rel intensity) 320.3 (100, $M^+$), 305.3 (60, M-Me), 217.2 (20). Anal. Calcd for $C_{20}H_{24}Si_2$: C, 74.93; H, 7.54, found: C, 75.25; H, 7.34.

**2,3-Bis(bromoethynyl)naphthalene.** To a solution of 2,3-bis(trimethylsilylethynyl) naphthalene (0.36 g, 1.1 mmol) in 15 ml acetone was added N-bromosuccinimide (0.46 g, 2.52 mmol) and $AgNO_3$ (0.20 g, 1.2 mmol). The suspension was stirred at 25ºC for 10 h, after which the solvent was removed in vacuo. The substrate was taken up in hexanes, and passed through a pad of silica gel with hexanes. The solvent was evaporated to yield 0.36 g of an unstable red oil, which was used directly in the next step.

**2,3-Dibromoanthracene.** A solution of 2,3-bis(bromoethynyl) naphthalene (0.33g, 1 mmol) was deoxygenated by sparging under $N_2$ in 40 ml benzene for 1 h. 1,4-Cyclohexadiene (4 ml, 10% v/v) was added and the mixture was sealed under $N_2$ in a steel bomb, which was heated to 180ºC for 2h (300 psi was the highest pressure reached). The benzene/cyclohexadiene mixture was recovered in vacuo, after which the residue was taken up in $CH_2Cl_2$ and passed through a pad of silica gel with 9:1 hexane/$CH_2Cl_2$ as eluent. The solvent was evaporated to yield 0.27g of a light sandy brown solid, which was used directly in the next step.

**2,3-Bis(trimethylsilylethynyl) anthracene.** To a solution of trimethylsilyl acetylene (0.35 g, 3.6 mmol) in 5 ml THF cooled to -78 $^{o}$C under $N_2$ was added butyllithium (3.8 mmol, 1.54 ml, 2.46 M in hexanes), after which the reaction mixture was allowed to warm to 25 $^{o}$C. $ZnCl_2$ (5 mmol, 1M solution in $Et_2O$) was added, followed after 15 min by [1,1' bis(diphenylphosphino)-ferrocene]palladium(II)chloride (0.1 g, 2 mol %) and the crude cycloaromatization product (2,3-dibromoanthracene) (0.2 g, 0.60 mmol), and the reaction was heated to reflux. After 12 h, the reaction was cooled and extracted into hexanes, washed with water, dried, and passed through a short pad of silica gel with hexanes. The crude oil was purified by silica gel chromatography with hexane eluent to afford 0.19g (86%) of canary yellow plates, mp 210-211$^{o}$C. FTIR ($CCl_4$) 2959, 2157 $cm^{-1}$. $^{1}$H NMR (200MHz, $CDCl_3$) $\delta$ 8.02(s, 2 H), 7.92(s, 2 H), 7.69(m, 2 H), 7.20(m, 2 H), 0.11(s, 18 H); $^{13}$C NMR (50MHz, $CDCl_3$) $\delta$ 133.16, 132.43, 129.93, 128.26, 126.21, 126.14, 121.00, 103.60, 98.07, 0.11; MS m/z (rel intensity) 370.2 (100, $M^{+}$), 355.1 (25, M-Me). Anal. Calcd for $C_{24}H_{26}Si_2$: C, 77.77; H, 7.07, found: C, 77.59; H, 7.24.

**Napthacene.** To a solution of 2,3-Bis(trimethylsilylethynyl) anthracene (0.15 g, 0.45 mmol) in 2ml THF was added 5ml methanol, followed by NaH (0.02 g, 0.8 mmol). The suspension was allowed to stir for 2 h, after which it was poured into 500 ml $H_2O$, extracted with 3 X 200 ml $CH_2Cl_2$, dried, and passed through a thin silica pad. Evaporation of solvent led to crude diethynyl anthracene. A solution of 60mg of this substance (0.27 mmol) in 40 ml benzene was deoxygenated under $N_2$ for 1 h. 1,4-Cyclohexadiene (4 ml, 10% v/v) was added and the mixture was sealed under $N_2$ in a steel bomb, which was heated to 160$^{o}$C overnight (240 psi was the highest pressure reached). The benzene/cyclohexadiene mixture was recovered in vacuo, after which the residue was taken up in 1L of $CH_2Cl_2$ and adsorbed onto silica gel by evaporation. The product was purified by silica gel chromatography with hexane eluent. The solvent was evaporated and the solid recrystallized from methylene chloride to yield 40mg (64%) of insoluble

orange plates, mp >300°C (lit 341°C[4]). $^1$H NMR (200MHz, $CDCl_3$) δ 7.40 (dd, 4H), 8.00 (dd, 4H), 8.67 (s, 4H). MS m/z (rel intensity) 228.1 (100%, $M^+$), 114.0 (20%). Anal. Calcd for $C_{18}H_{12}$: C, 94.70; H, 5.29, found: C, 94.75; H, 5.24.

Literature Cited

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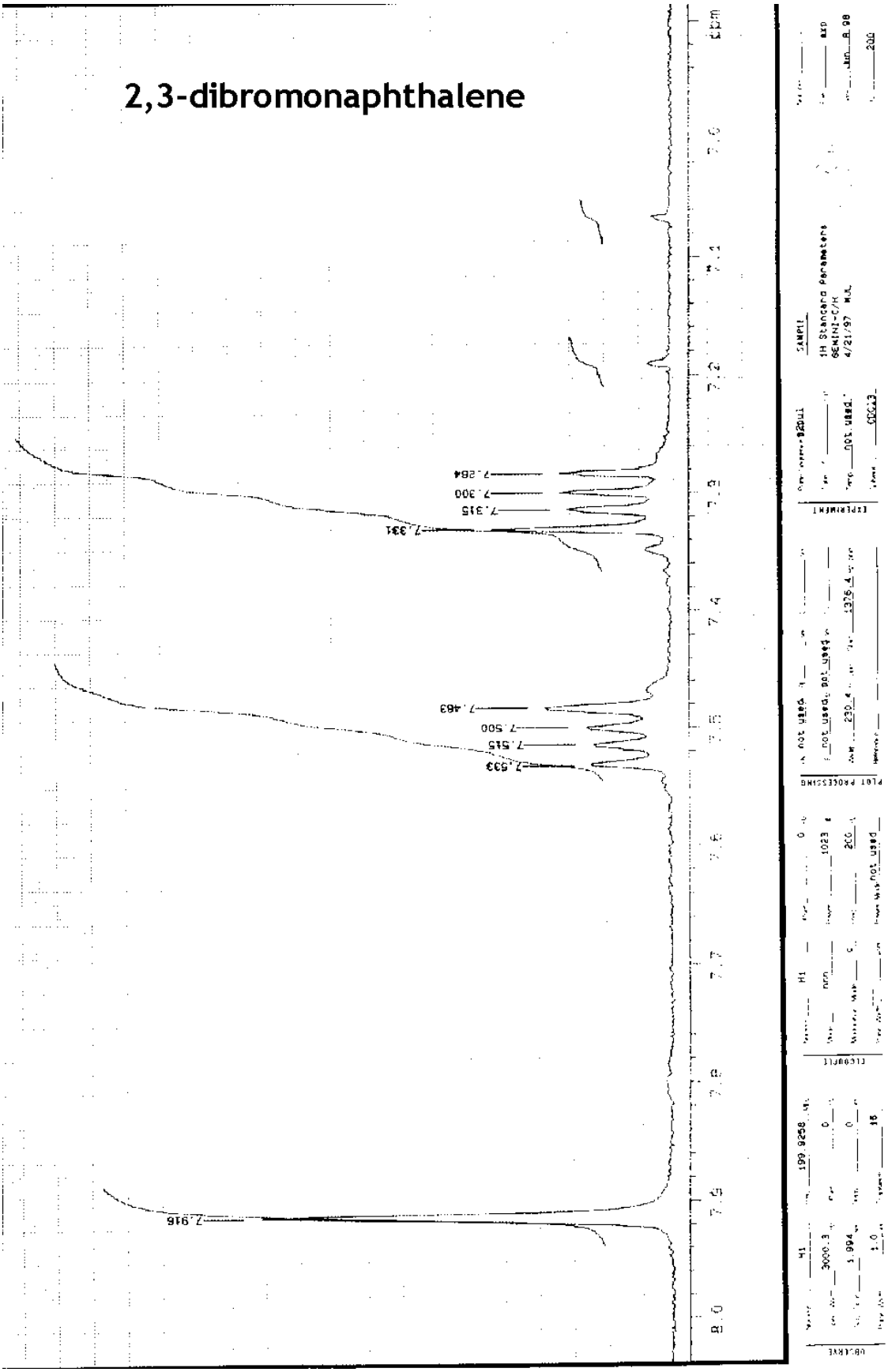
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7.7
7.6
7.5
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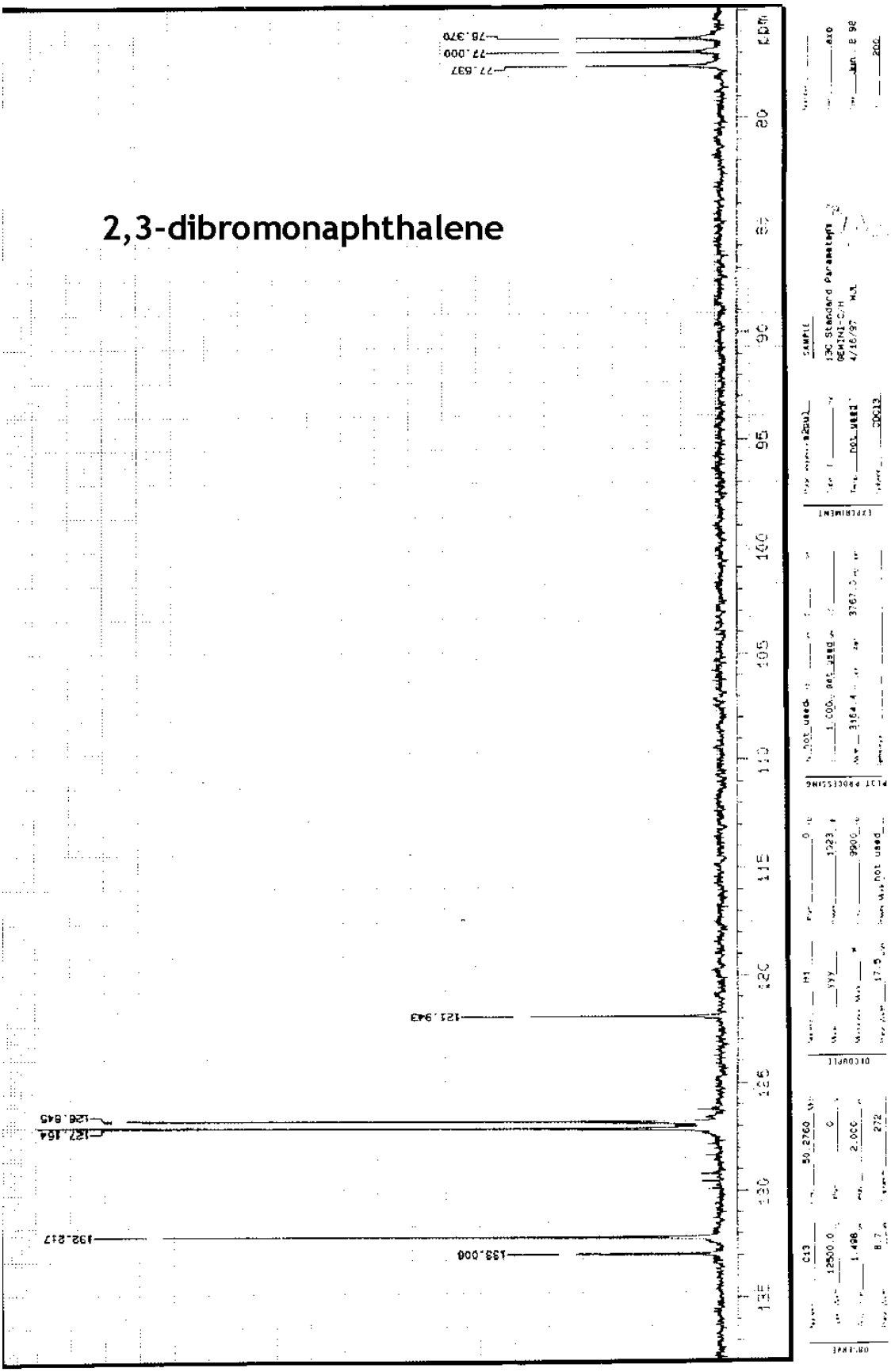
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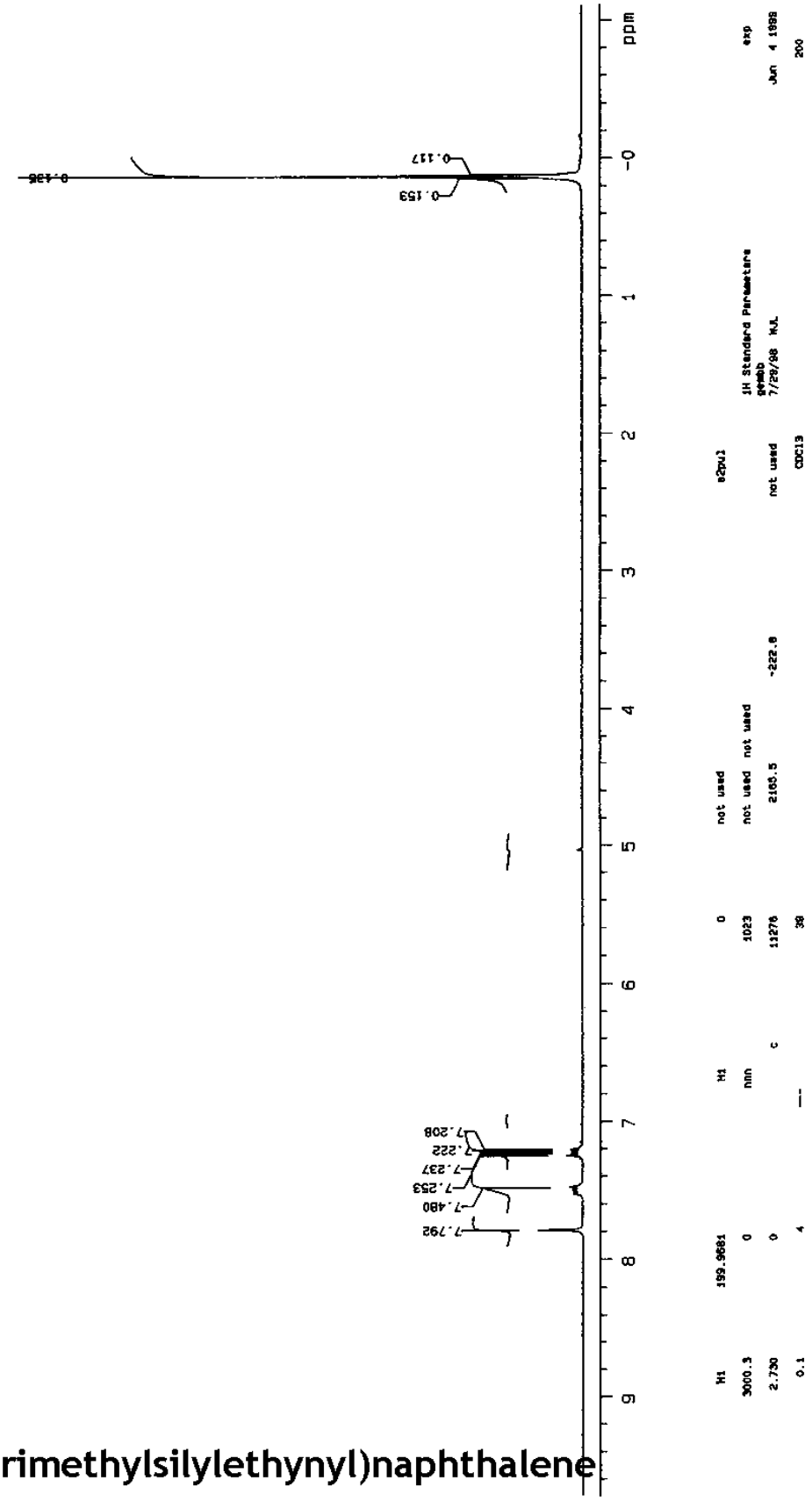


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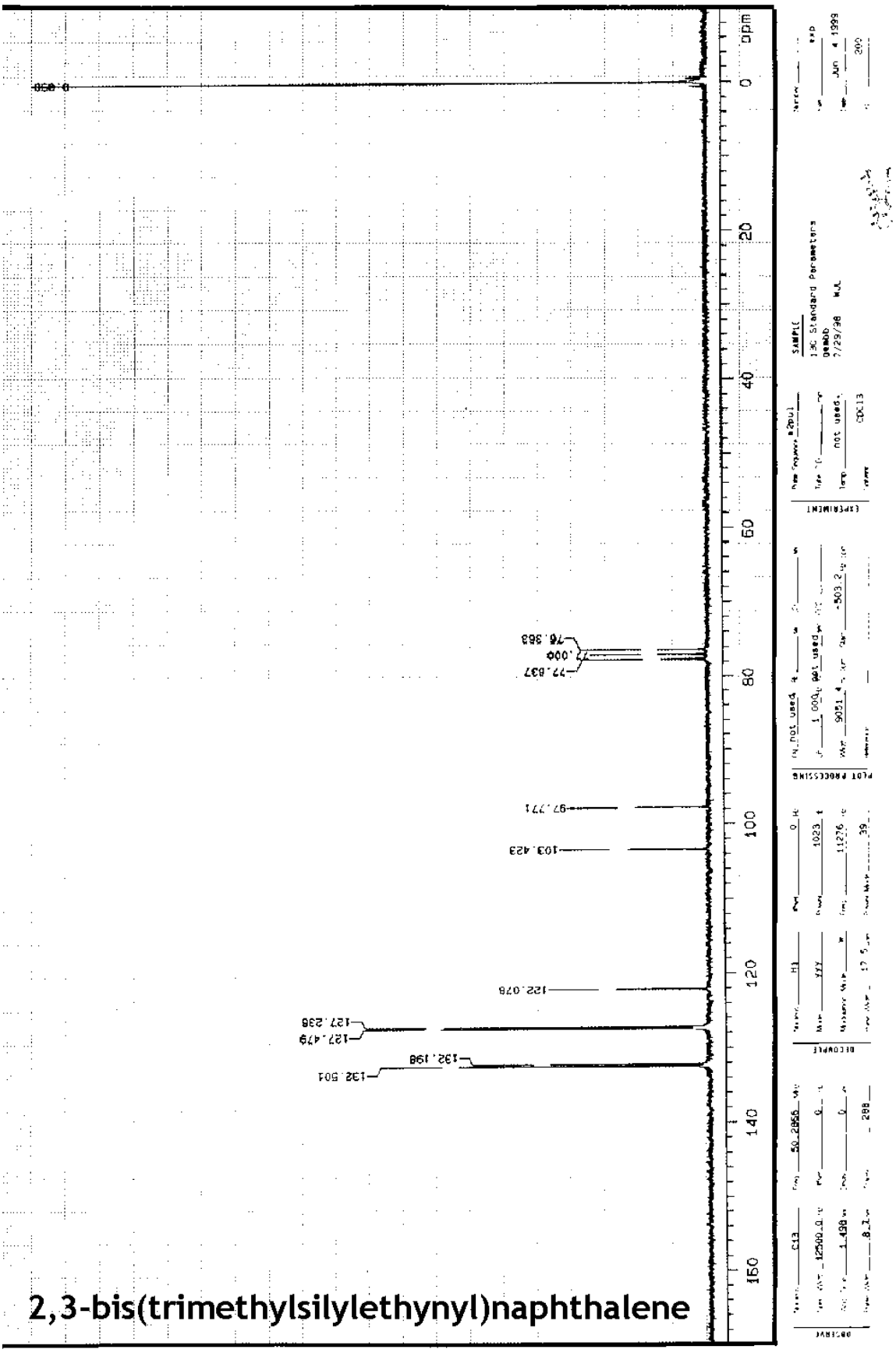
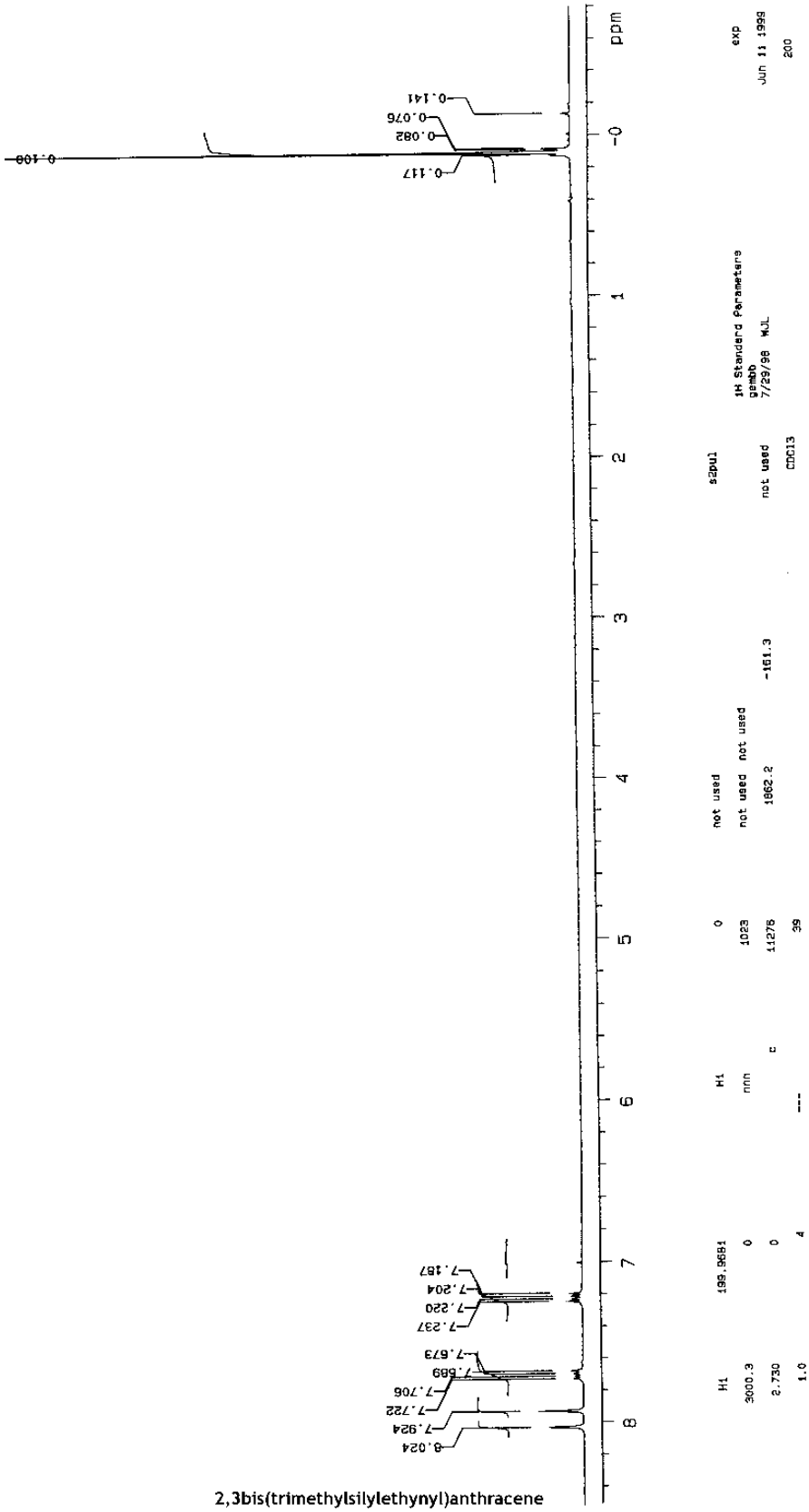
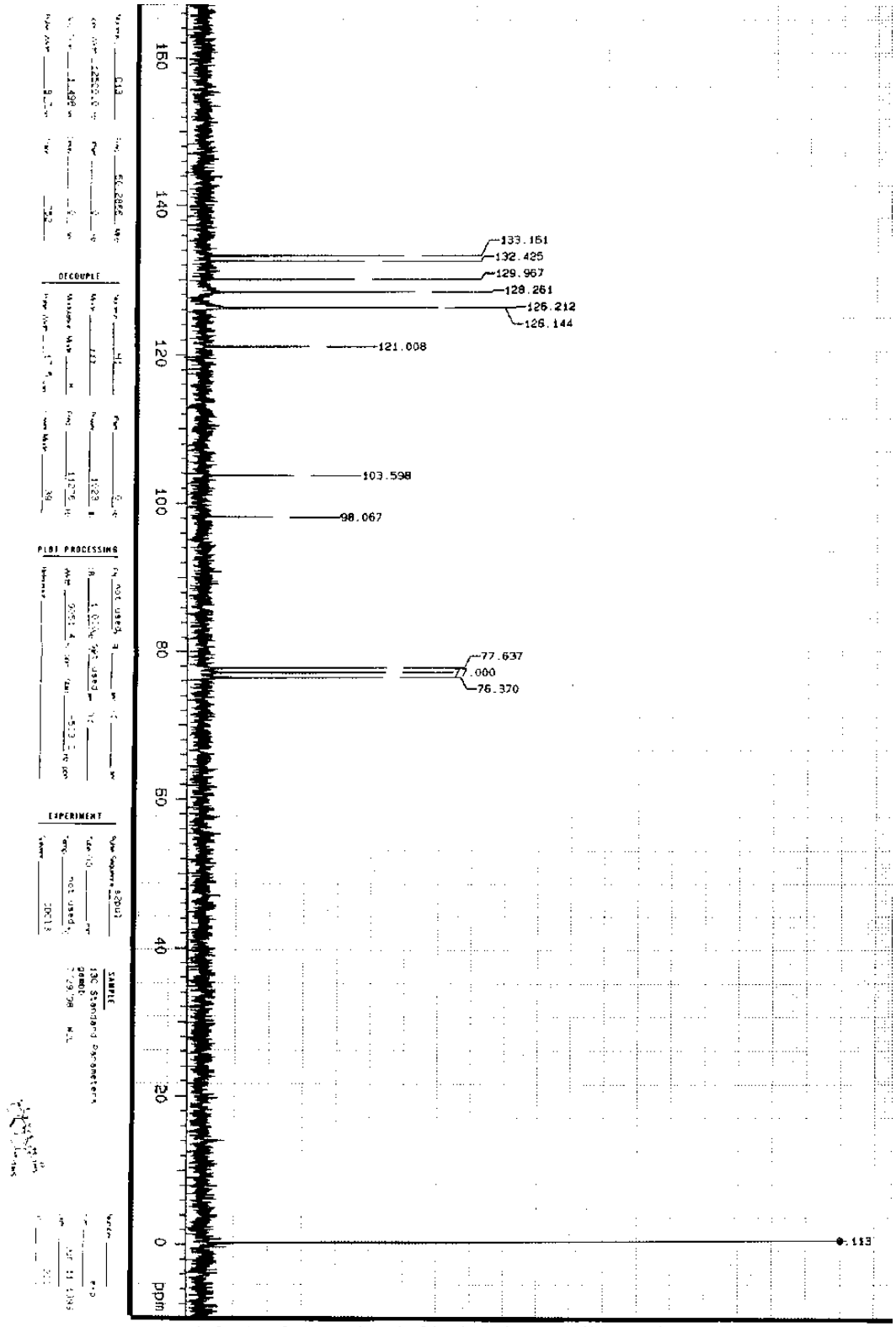



2,3-bis(trimethylsilylethynyl)naphthalene




2,3bis(trimethylsilylethynyl)anthracene




2,3-bis(trimethylsilylethynyl)anthracene