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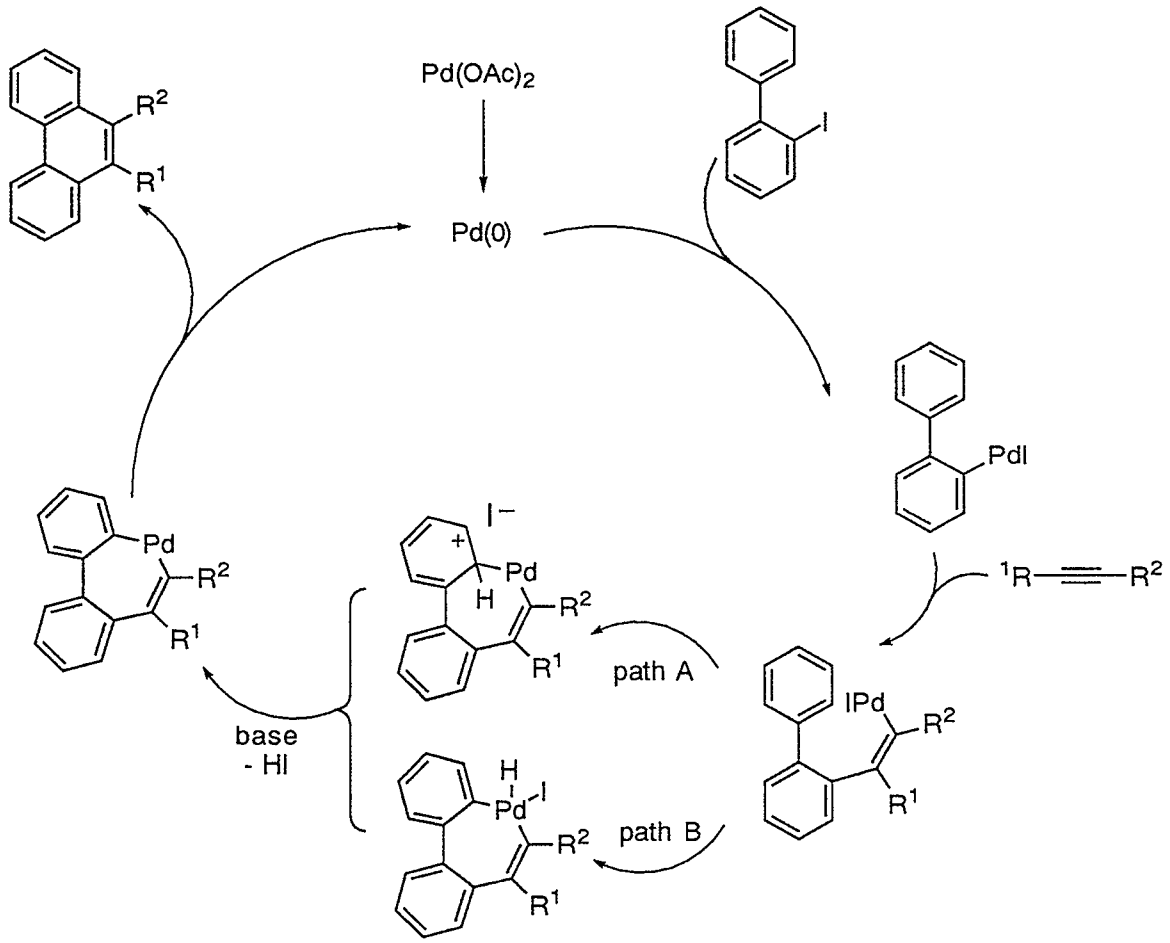
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Supporting Information

Synthesis of Polycyclic Aromatic Hydrocarbons by Pd-Catalyzed Annulation of Alkynes

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General Procedure for the Palladium-catalyzed Annulation of Alkynes (Procedures A and B). To a 2 dram vial equipped with a stir bar is added $Pd(OAc)_2$ (0.125 mmol), NaOAc (0.50 mmol), either LiCl (0.25 mmol, **Procedure A**) or *n*-Bu_4NCl (TBAC, 0.75 mmol, **Procedure B**), the organic iodide or triflate (0.25 mmol), the appropriate alkyne (0.25 or 0.50 mmol) and 5 mL of DMF. The vial is then flushed with N_2, capped with a screw cap containing a teflon liner and placed in an oil bath at 100 °C for the specified length of time (see table). The vial is then removed from the oil bath, diluted with Et_2O and/or EtOAc, washed with sat'd NH_4Cl and water, dried over $MgSO_4$ and concentrated. This residue was then either purified by column chromatography or washed and/or recrystallized with MeOH and/or Et_2O.

9,10-Diphenylphenanthrene (entry 1, procedure A). The spectral properties are identical to those previously reported.[1]

2-Methoxy-9,10-diphenylphenanthrene (entry 2, procedure B). Obtained as a pale yellow solid in 86 % yield from the reaction of 2-iodo-4'-methoxybiphenyl and diphenylacetylene. After the normal workup, drying and concentrating, the resultant solid was washed with MeOH and Et_2O to give essentially pure product: mp 241-242 °C

(toluene); ^{1}H NMR ($CDCl_3$) δ 3.71 (s, 3 H), 6.93 (d, 1 H, J = 2.7 Hz), 7.12-7.27 (m, 9 H), 7.27 (d, 1 H, J = 2.7 Hz), 7.31 (d, 1 H, J = 2.7 Hz), 7.41 (t, 1 H, J = 7.2 Hz), 7.51 (d, 1 H, J = 7.5 Hz), 7.63 (t, 1 H, J = 7.2 Hz), 8.70 (d, 1 H, J = 11.4 Hz), 8.71 (d, 1 H, J = 9.3 Hz); ^{13}C NMR ($CDCl_3$) δ 55.2, 109.1, 116.4, 122.1, 124.2, 124.6, 125.7, 126.5, 126.6, 127.6, 127.7, 128.0, 130.3, 131.1, 131.1, 133.5, 136.9, 137.9, 139.8, 139.8, 158.5 (two sp^2 C missing due to overlap); HRMS for $C_{27}H_{20}O$: calcd 368.1960, found 368.1965; Anal. Calcd for $C_{27}H_{20}O$: C, 89.96; H, 5.60. Found: C, 90.22; H, 5.67.

2-Trifluoromethyl-9,10-diphenylphenanthrene (entry 3, procedure A). Obtained as a white solid in 69 % yield from the reaction of 2-iodo-4'-(trifluoromethyl)biphenyl and diphenylacetylene after purification by column chromatography using hexanes as eluent: mp 176-178 °C (hexanes); ^{1}H NMR ($CDCl_3$) δ 7.08-7.34 (m, 10 H), 7.50-7.59 (m, 2 H), 7.69 (dt, 1 H, J = 6.6, 1.5 Hz), 7.82-7.94 (m, 2 H), 8.79 (d, 1 H, J = 8.4 Hz), 8.87 (d, 1 H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$) δ 122.2, 122.6, 122.9, 123.5, 125.1, 126.2, 126.8, 127.0, 127.0, 127.7, 127.9, 128.1, 128.6, 129.3, 130.9, 130.9, 131.5, 132.1, 132.6, 137.2, 138.5, 138.7, 139.0; IR ($CHCl_3$) 3061, 3019, 1575, 1480 cm^{-1}; HRMS for $C_{27}H_{17}F_3$: calcd 398.1282, found 398.1290.

9-Phenyl-10-(triethylsilyl)phenanthrene (entry 4, procedure A). Obtained as a white solid in 49 % yield from the reaction of 2-iodobiphenyl and 1-phenyl-2-(triethylsilyl)acetylene, followed by purification by column chromatography using hexanes as eluent: mp 134-136 °C (MeOH); ^{1}H NMR ($CDCl_3$) δ 0.57 (q, 6 H, J = 7.8 Hz), 0.79 (t, 9 H, J = 7.5 Hz), 7.26-7.52 (m, 6 H), 7.60 (d quintet, 4 H, J = 5.7, 1.5 Hz), 8.38 (dd, 1 H, J = 7.8, 1.5 Hz), 8.71 (d, 1 H, J = 8.4 Hz), 8.76 (dd, 1 H, J = 8.1, 1.2 Hz); ^{13}C NMR ($CDCl_3$) δ 5.6, 8.3, 122.1, 123.0, 125.7, 125.8, 126.2, 126.8, 127.5, 127.7, 127.9, 128.9, 129.7, 130.8, 131.6, 132.1, 132.3, 135.3, 142.5, 148.3; IR ($CHCl_3$)

3064, 3017, 2955, 2873, 1545, 1485 cm^{-1}; HRMS for $C_{26}H_{28}Si$: calcd 368.1960, found 368.1965.

2-Methoxy-9-phenyl-10-(triethylsilyl)phenanthrene and 2-methoxy-10-phenyl-9-(triethylsilyl)phenanthrene (entry 5, procrdure A). A white solid obtained in 50 % yield as an inseparable 1:1 mixture of regioisomers from the reaction of 2-iodo-4'-methoxybiphenyl and 1-phenyl-2-(triethylsilyl)acetylene and purified by column chromatography using hexanes as eluent and then recrystallization from MeOH. Spectral data for product mixture: ^{1}H NMR ($CDCl_3$) δ 0.58 (q, 6 H, J = 7.8 Hz), 0.59 (q, 6 H, J = 7.8 Hz), 0.79 (t, 9 H, J = 7.8 Hz), 0.80 (t, 9 H, J = 7.8 Hz), 3.65 (s, 3 H), 3.98 (s, 3 H), 6.68 (d, 1 H, J = 3.0 Hz), 7.20-7.39 (m, 8 H), 7.41-7.67 (m, 9 H), 7.80 (d, 1 H, J = 3.0 Hz), 8.34 (dd, 1 H, J = 9.0, 1.2 Hz), 8.61 (d, 1 H, J = 9.0 Hz), 8.62 (d, 1 H, J = 9.0 Hz), 8.64 (m, 2 H); ^{13}C NMR ($CDCl_3$) δ 5.4, 5.5, 8.2, 8.3, 55.1, 55.5, 109.1, 110.4, 115.8, 116.6, 121.7, 122.5, 123.8, 124.0, 124.5, 124.7, 125.2, 125.3, 125.9, 126.9, 127.5, 127.5, 127.7, 127.8, 128.0, 129.0, 129.8, 131.0, 131.2, 131.4, 131.5, 131.6, 132.9, 133.6, 134.4, 136.8, 142.6, 147.7, 149.0, 157.4, 157.8 (one sp^2 C missing due to overlap); HRMS for $C_{27}H_{30}OSi$: calcd 398.2066, found 398.2070.

4-Methoxy-9-phenyl-10-(triethylsilyl)phenanthrene and 4-methoxy-10-phenyl-9-(triethylsilyl)phenanthrene (entry 6, procedure A). A white solid obtained in 61 % yield as a 3 : 1 mixture of isomers from the reaction of 2-iodo-2'-methoxybiphenyl and 1-phenyl-2-(triethylsilyl)acetylene and purified by column chromatography using 30 : 1 hexanes/EtOAc. The nearly pure major isomer was obtained by column chromatography and therefore was fully characterized. For the minor isomer, only ^{1}H NMR spectral data were obtainable. **4-Methoxy-9-phenyl-10-(triethylsilyl)phenanthrene:** ^{1}H NMR ($CDCl_3$) δ 0.55 (q, 6 H, J = 7.8 Hz), 0.77 (t, 9 H, J = 7.5 Hz), 4.13, (s, 3 H), 7.15 (d, 1 H, J = 7.8 Hz), 7.33-7.38 (m, 5 H), 7.42-

7.46 (m, 3 H), 7.49 (q, 1 H, J = 8.1 Hz), 7.99 (dd, 1 H, J = 8.1, 0.9 Hz), 9.68 (dd, 1 H, J = 8.4, 0.9 Hz); ^{13}C NMR ($CDCl_3$) δ 5.8, 8.3, 55.9, 108.0, 121.9, 125.2, 125.5, 125.8, 126.3, 127.3, 127.5, 127.6, 128.0, 130.6, 131.9, 132.4, 132.6, 138.0, 142.6, 149.0, 158.7; IR ($CDCl_3$) 3074, 3018, 2956, 2873, 1215, 1084 cm^{-1}; HRMS for $C_{27}H_{30}OSi$: calcd 398.2066, found 398.2070. **4-Methoxy-9-(triethylsilyl)-10-phenylphenanthrene:** ^{1}H NMR ($CDCl_3$) δ 0.55 (q, 6 H, J = 7.8 Hz), 0.77 (t, 9 H, J = 7.5 Hz), 4.12, (s, 3 H), 6.98 (dd, 1 H, J = 7.2, 1.2 Hz), 7.15 (d, 1 H, J = 7.2 Hz), 7.33-7.38 (m, 5 H), 7.42-7.46 (m, 1 H), 7.54-7.60 (m, 2 H), 8.35 (dd, 1 H, J = 7.2, 1.2 Hz), 9.72 (dd, 1 H, J = 7.8, 1.5 Hz).

3,4-Di(4'-methoxyphenyl)benz[a]anthracene and 5,6-di(4'-methoxyphenyl)chrysene (entry 7, procedure A). A light brown solid obtained as an inseparable, 5 : 1 mixture of isomers in 75 % yield from the reaction of 1-iodo-2-(2'-naphthyl)benzene and di(4-methoxyphenyl)acetylene. The assignment of isomers is based on a singlet at 9.27 ppm for the minor isomer. **3,4-Di(4-methoxyphenyl)benz[a]anthracene** (major isomer): ^{1}H NMR ($CDCl_3$) δ 3.79 (s, 3 H), 3.82 (s, 3 H), 6.72 (d, 2 H, J = 8.4 Hz), 6.80 (d, 2 H, J = 9.3 Hz), 6.95-7.14 (m, 5 H), 7.51 (t, 1 H, J = 7.2 Hz), 7.54 (dd, 2 H, J = 7.8, 0.6 Hz), 7.60-7.70 (m, 2 H), 7.85 (d, 1 H, J = 8.1 Hz), 8.03 (d, 1 H, J = 9.0 Hz), 8.83 (t, 2 H, J = 9.0 Hz). **5,6-Di(4-methoxyphenyl)chrysene** (minor isomer): ^{1}H NMR ($CDCl_3$) δ 3.81 (s, 3 H), 3.83 (s, 3 H), 6.74 (d, 2 H, J = 8.4 Hz), 6.82 (d, 2 H, J = 9.3 Hz), 6.95-7.04 (m, 4 H), 7.51 (t, 1 H, J = 7.2 Hz), 7.54 (dd, 2 H, J = 7.8, 0.6 Hz), 7.60-7.70 (m, 2 H), 7.83 (d, 1 H, J = 8.7 Hz), 8.01 (s, 1 H), 8.12 (d, 1 H, J = 8.1 Hz), 8.92 (d, 1 H, J = 8.4 Hz), 9.27 (s, 1 H). Additional spectral data for the product mixture: ^{13}C NMR ($CDCl_3$) δ 55.2, 113.1, 113.7, 121.1, 123.2, 124.7, 125.5, 126.4, 126.5, 127.8, 128.1, 128.2, 128.3, 128.7, 129.5, 130.0, 131.4, 132.0, 132.2, 132.4, 133.7, 136.1, 136.8, 139.3, 157.9 (after

5000 scans these were the only peaks detected); HRMS for $C_{32}H_{24}O_2$: calcd 440.1776, found 440.1777.

9,10-Diphenyl-1,2,3,4-tetrahydrophenanthrene (entries 8 and 9, procedure B). Obtained as a white solid from the reaction of 1-iodo-2-phenylcyclohexene or 2-phenyl-1-cyclohexenyl triflate and diphenylacetylene after purification by column chromatography (hexanes): mp 190-192 °C (hexanes); ^{1}H NMR ($CDCl_3$) δ 1.80 (m, 2 H), 1.97 (m, 2 H), 2.57 (t, 2 H, J = 6.3 Hz), 3.16 (t, 2 H, J = 6.3 Hz), 7.00-7.65 (m, 13 H), 8.12 (d, 1 H, J = 8.4 Hz); ^{13}C NMR ($CDCl_3$) δ 23.0, 23.3, 26.6, 30.1, 122.7, 127.5, 125.0, 125.7, 126.2, 126.5, 127.4, 127.5, 130.2, 131.1, 131.2, 131.5, 131.8, 133.2, 136.7, 139.7, 139.8, 140.8; IR (CH_2Cl_2) 3054, 1491 cm^{-1}; HRMS for $C_{26}H_{22}$: calcd 334.1721, found 334.1719.

9-*tert*-Butyl-10-phenyl-1,2,3,4-tetrahydrophenanthrene (entries 10 and 11, procedure B). Obtained as a white solid from the reaction of 1-iodo-2-phenylcyclohexene or 2-phenyl-1-cyclohexenyl triflate and 3,3-dimethyl-1-phenyl-1-butyne after purification by column chromatography (hexanes): mp 150-152 °C (hexanes); ^{1}H NMR ($CDCl_3$) δ 1.37 (s, 9 H), 1.61 (m, 2 H), 1.84 (m, 2 H), 2.17 (t, 2 H, J = 5.7 Hz), 3.14 (t, 2 H, J = 6.0 Hz), 7.18-7.48 (m, 7 H), 8.06 (d, 1 H, J = 8.1 Hz), 8.53 (d, 1 H, J = 8.4 Hz); ^{13}C NMR ($CDCl_3$) δ 22.8, 23.6, 27.1, 30.6, 35.3, 38.3, 122.8, 123.3, 124.6, 126.4, 127.3, 128.9, 130.4, 130.9, 131.0, 133.4, 134.1, 139.7, 140.6, 143.9; IR (CH_2Cl_2) 3074, 1429 cm^{-1}; HRMS for $C_{24}H_{26}$: calcd 314.2034, found 314.2042.

10-Phenyl-9-triethylsilyl-1,2,3,4-tetrahydrophenanthrene (entries 12 and 13, procedure B). Obtained as a white solid from the reaction of 1-iodo-2-phenylcyclohexene or 2-phenyl-1-cyclohexenyl triflate and 1-phenyl-2-(triethylsilyl)acetylene after purification by column chromatography (hexanes): mp 124-125 °C (hexanes); ^{1}H NMR ($CDCl_3$) δ 0.52 (q, 6 H, J = 7.5 Hz), 0.80 (t, 9 H, J = 7.5 Hz), 1.68 (m, 2 H), 1.89 (m, 2 H), 2.27

(t, 2 H, J = 6.0 Hz), 3.20 (t, 2 H, J = 6.0 Hz), 7.19-7.22 (m, 2 H), 7.37 (m, 3 H), 7.48 (m, 2 H), 8.07 (dd, 1 H, J = 7.5, 3.0 Hz), 8.30 (dd, 1 H, J = 7.5, 3.0 Hz); ^{13}C NMR ($CDCl_3$) δ 5.6, 8.3, 22.8, 23.3, 26.9, 30.1, 123.3, 123.9, 124.9, 126.9, 127.6, 128.7, 130.6, 130.7, 131.9, 133.1, 133.4, 135.9, 143.7, 150.1; IR (CH_2Cl_2) 3055, 1456 cm^{-1}; HRMS for $C_{26}H_{32}Si$: calcd 372.2273, found 372.2278.

5,6-Diphenyl-7H-benzo[c]fluoren-7-one (entry 14, procedure A). Obtained as an orange solid in 54 % from the reaction of 2-iodo-3-phenylindenone and diphenylacetylene after purification by column chromatography (10:1 hexanes/ethyl acetate): mp 238-240 °C (CH_2Cl_2/MeOH); ^{1}H NMR ($CDCl_3$) δ 7.11 (m, 4 H), 7.19-7.33 (m, 7 H), 7.46-7.67 (m, 5 H), 8.15 (d, 1 H, J = 7.5 Hz), 8.65 (d, 1 H, J = 7.8 Hz); ^{13}C NMR ($CDCl_3$) δ 123.4, 123.9, 124.8, 126.7, 127.0, 127.2, 127.4, 127.7, 128.5, 128.6, 128.7, 128.8, 128.9, 129.9, 131.1, 134.3, 134.7, 136.2, 136.8, 137.2, 138.1, 141.3, 143.0, 144.0, 193.9; IR ($CDCl_3$) 3073, 3022, 1710 (C=O), 1602 cm^{-1}; HRMS for $C_{29}H_{18}O$: calcd 382.1358, found 382.1360.

1-*tert*-Butyl-2,4-diphenylnaphthalene and 2-*tert*-butyl-1,4-diphenylnaphthalene (entry 15, procedure A). Obtained as a 3:1 mixture of regioisomers in 62 % yield from the reaction of 2-iodo-1,1-diphenylethylene and 3,3-dimethyl-1-phenyl-1-butyne after purification by column chromatography (hexanes). The mixture was partially separated through preparative TLC and both pure isomers were isolated. **1-*tert*-Butyl-2,4-diphenylnaphthalene:** mp 139-140 °C (hexanes); ^{1}H NMR ($CDCl_3$) δ 1.26 (s, 9 H), 7.15-7.38 (m, 5 H), 7.45-7.60 (m, 8 H), 7.71 (s, 1 H), 7.89 (d, 1 H, J = 8.1 Hz); ^{13}C NMR ($CDCl_3$) δ 33.2, 37.2, 125.1, 125.4, 127.0, 127.1, 127.2, 127.3, 127.6, 128.3, 129.8, 130.3, 131.7, 134.9, 137.0, 139.3, 141.4, 141.8, 144.2 (one sp^2 C missing due to overlap); IR ($CDCl_3$) 3074, 2962, 1492 cm^{-1}; HRMS for $C_{26}H_{24}$: calcd, 336.1878, found 336.1887. **2-*tert*-Butyl-1,4-diphenylnaphthalene:**

mp 138-140 °C (hexanes); ^{1}H NMR ($CDCl_3$) δ 1.50 (s, 9 H), 7.15 (s, 1 H), 7.33-7.54 (m, 12 H), 7.96 (m, 1 H), 8.63 (d, 1 H, J = 8.7 Hz); ^{13}C NMR ($CDCl_3$) δ 35.0, 38.2, 123.9, 124.6, 126.4, 127.1, 127.2, 127.5, 128.2, 128.4, 129.6, 130.2, 132.1, 132.3, 133.4, 137.5, 139.1, 140.6, 142.4, 147.1; IR (CH_2Cl_2) 3069, 2926, 1596 cm^{-1}; HRMS for $C_{26}H_{24}$: calcd 336.1878, found 336.1880.

1-*tert*-Butyl-3-methyl-2,4-diphenylnaphthalene (entry 17, procedure A). Obtained as a white solid in 46 % from the reaction of 2-iodo-1,1-diphenylethylene and diphenylacetylene after purification by column chromatography (hexanes): mp 171-173 °C (hexanes); ^{1}H NMR ($CDCl_3$) δ 1.41 (s, 9 H), 2.34 (s, 3 H), 6.81-7.08 (m, 10 H), 7.52 (m, 2 H), 8.14 (m, 1 H), 8.63 (m, 1 H); ^{13}C NMR ($CDCl_3$) δ 17.4, 35.2, 38.4, 123.5, 124.8, 125.2, 125.5, 125.6, 126.2, 127.1, 129.0, 129.9, 130.0, 131.8, 132.2, 133.2, 139.1, 140.8, 141.1, 142.2, 143.9; IR ($CDCl_3$) 3064, 2927, 1599 cm^{-1}; HRMS for $C_{27}H_{26}$: calcd 350.2034, found 350.2032.

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(1) Wu, G.; Rheingold, A.; Geib, S. J.; Heck, R. F. *Organometallics* **1987**, *6*, 1941-1946.

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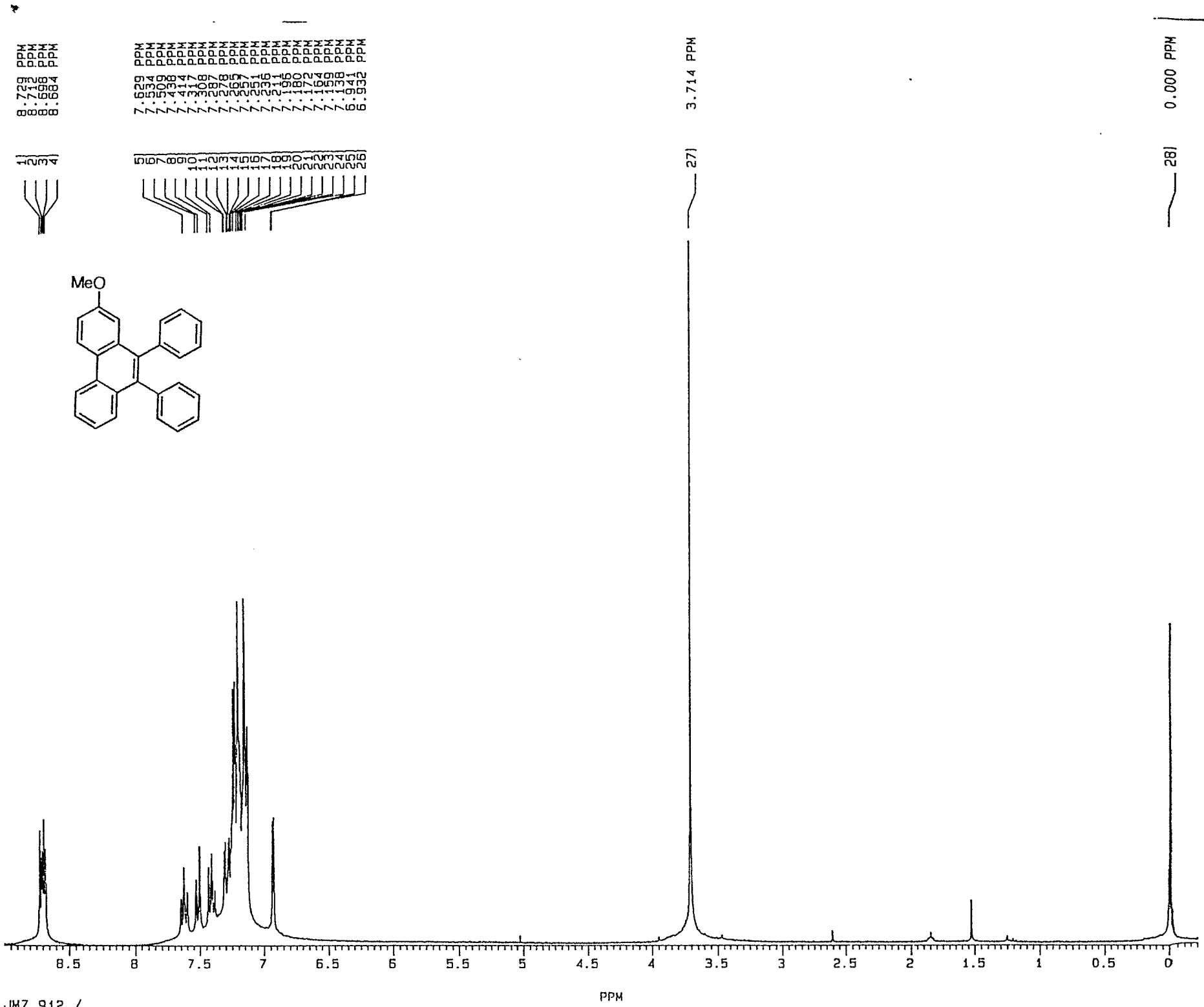

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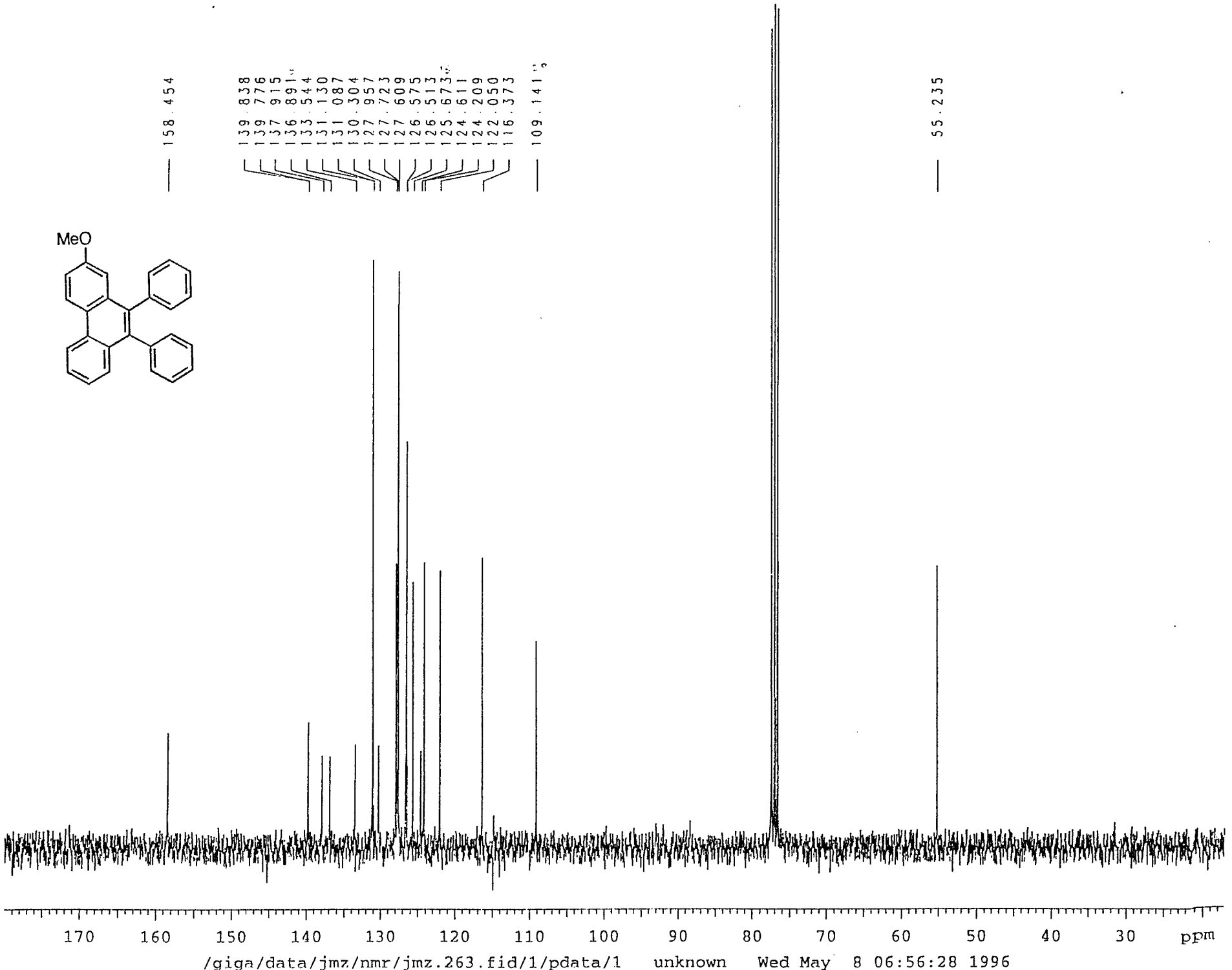

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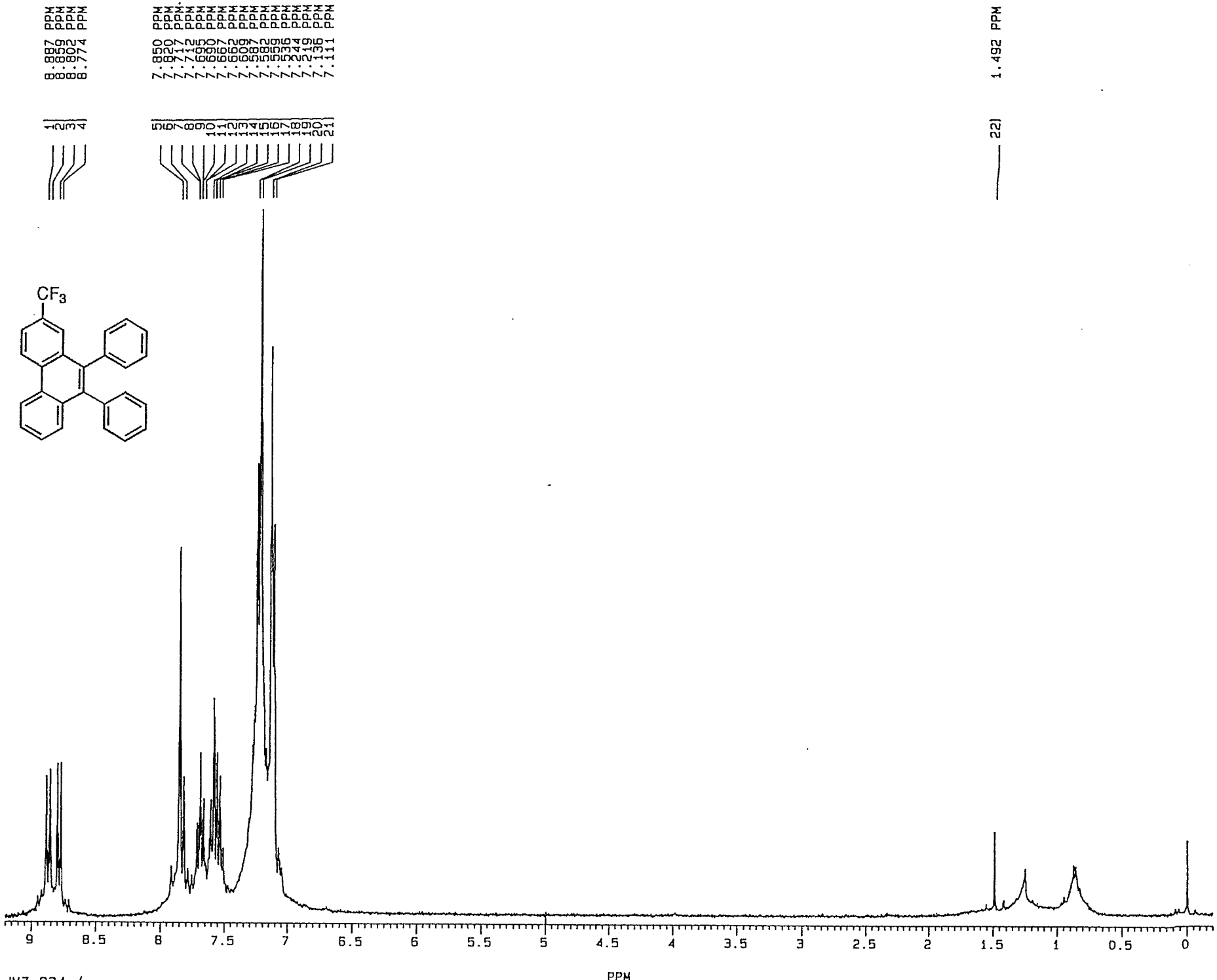

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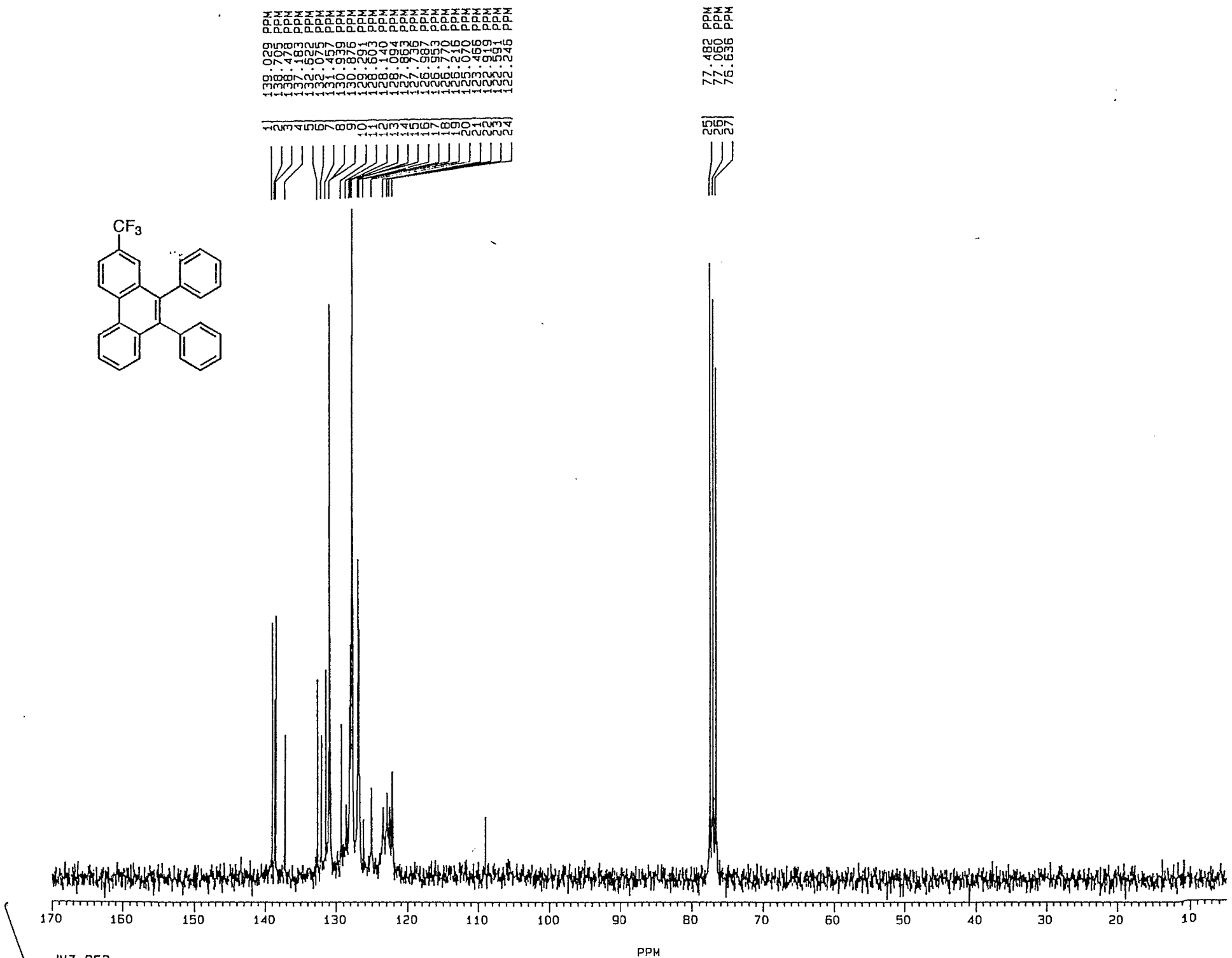

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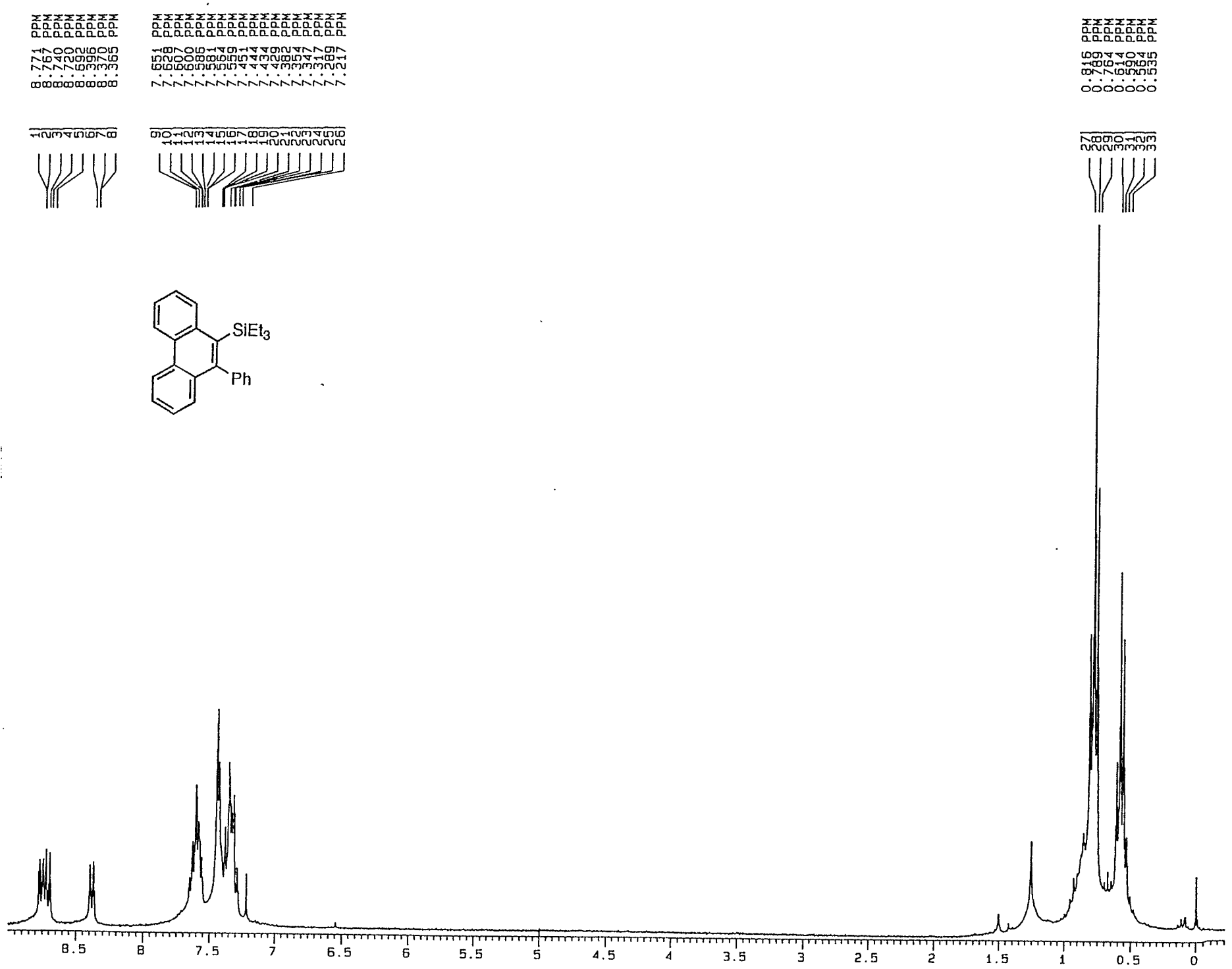

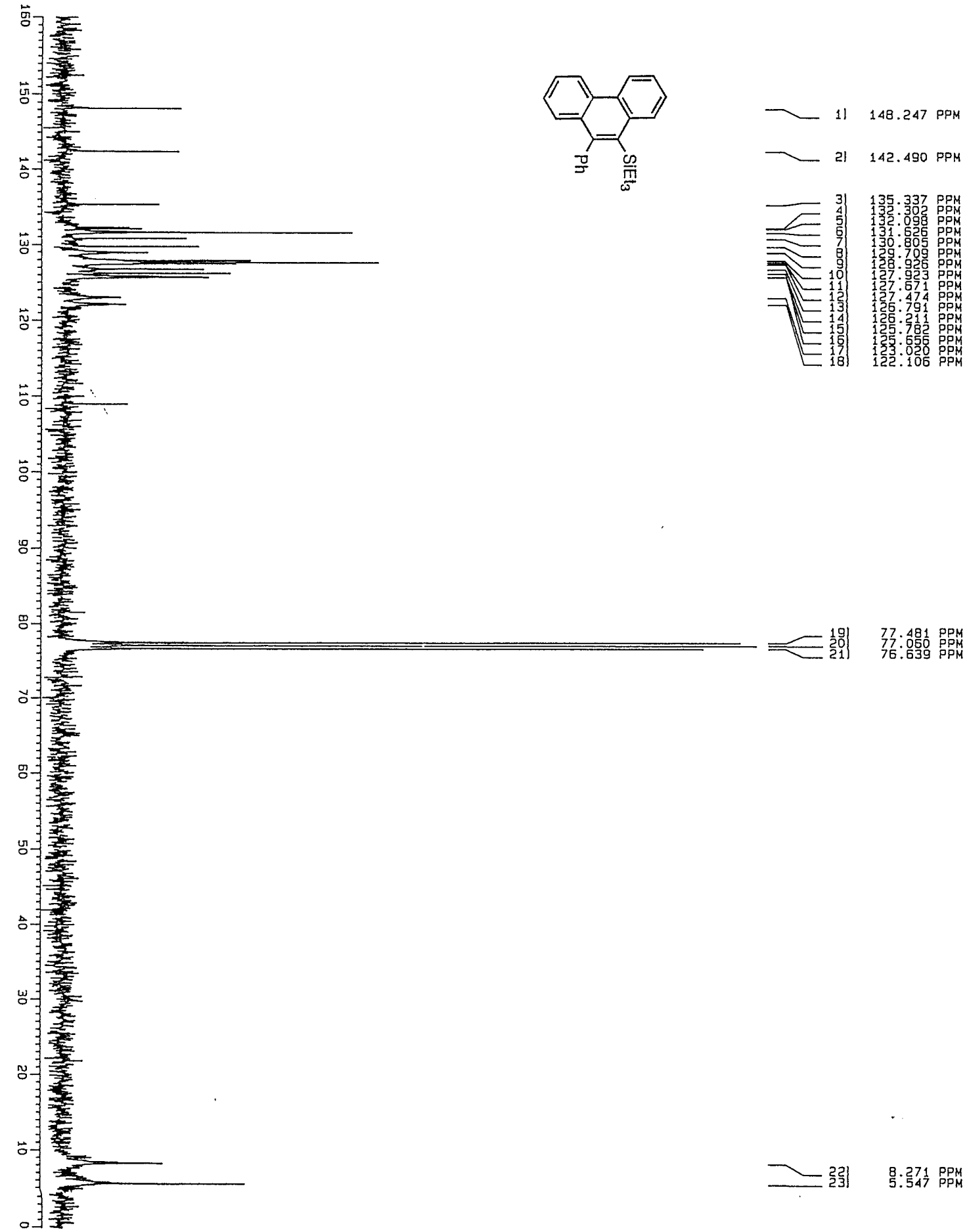
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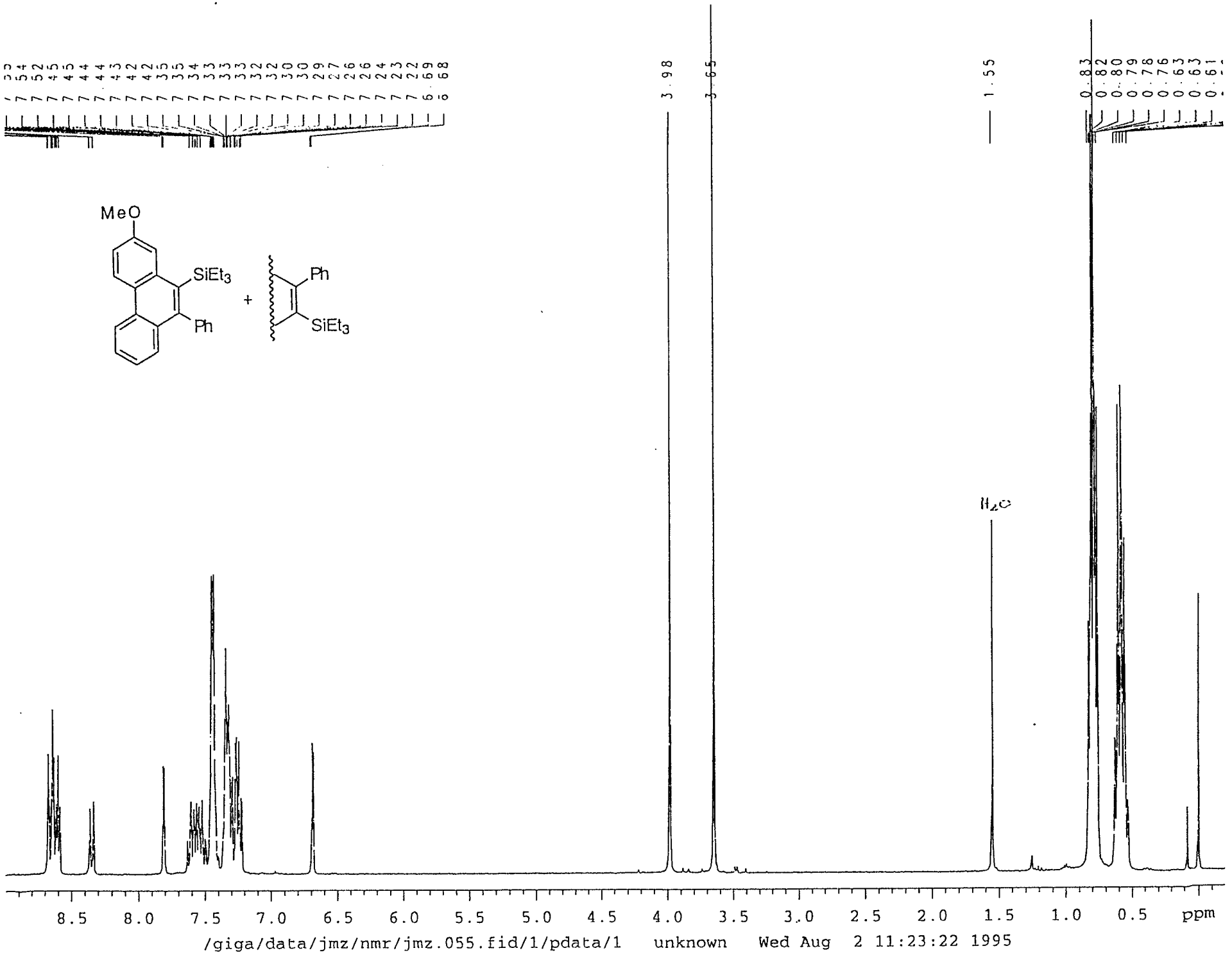

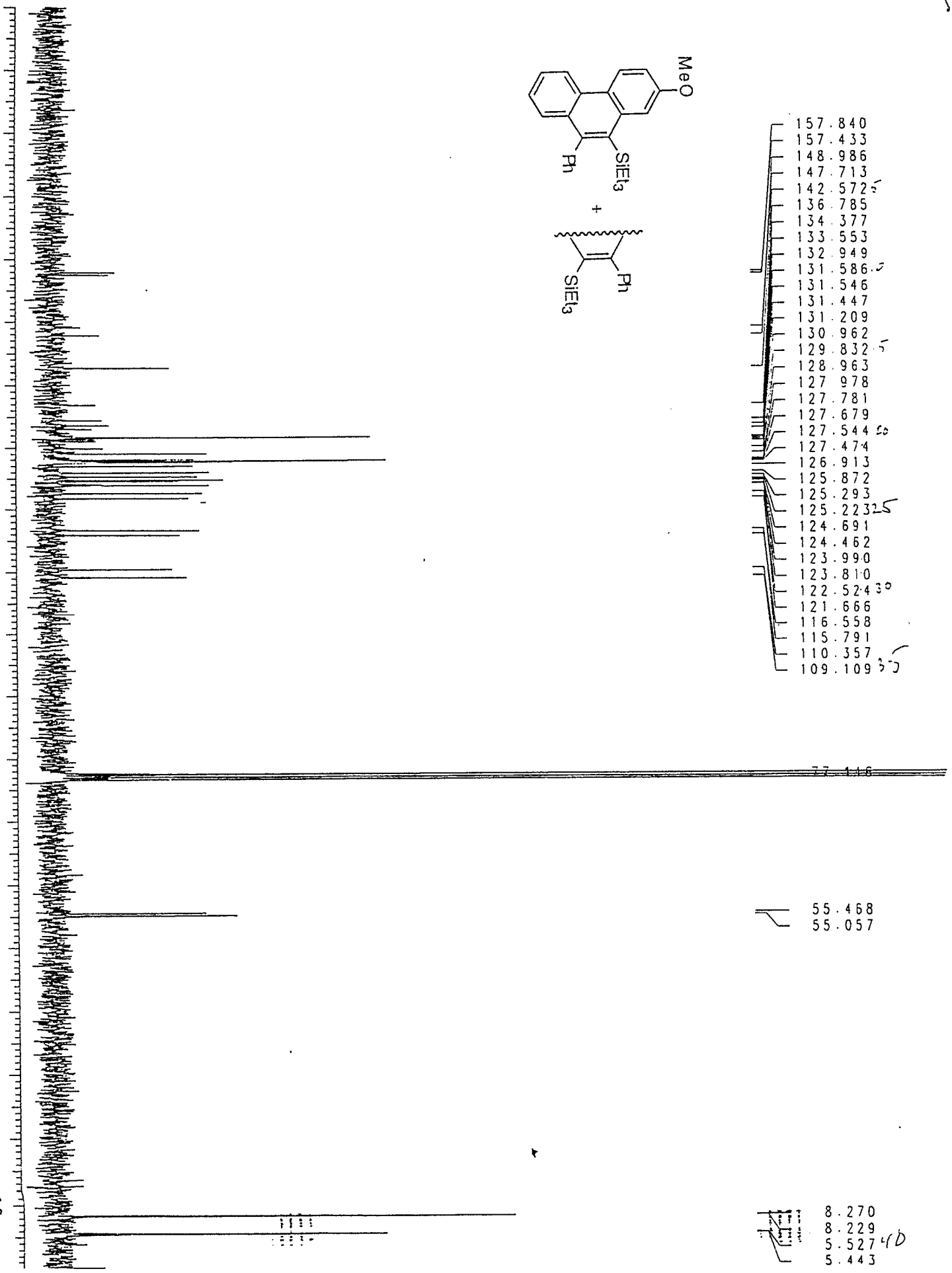
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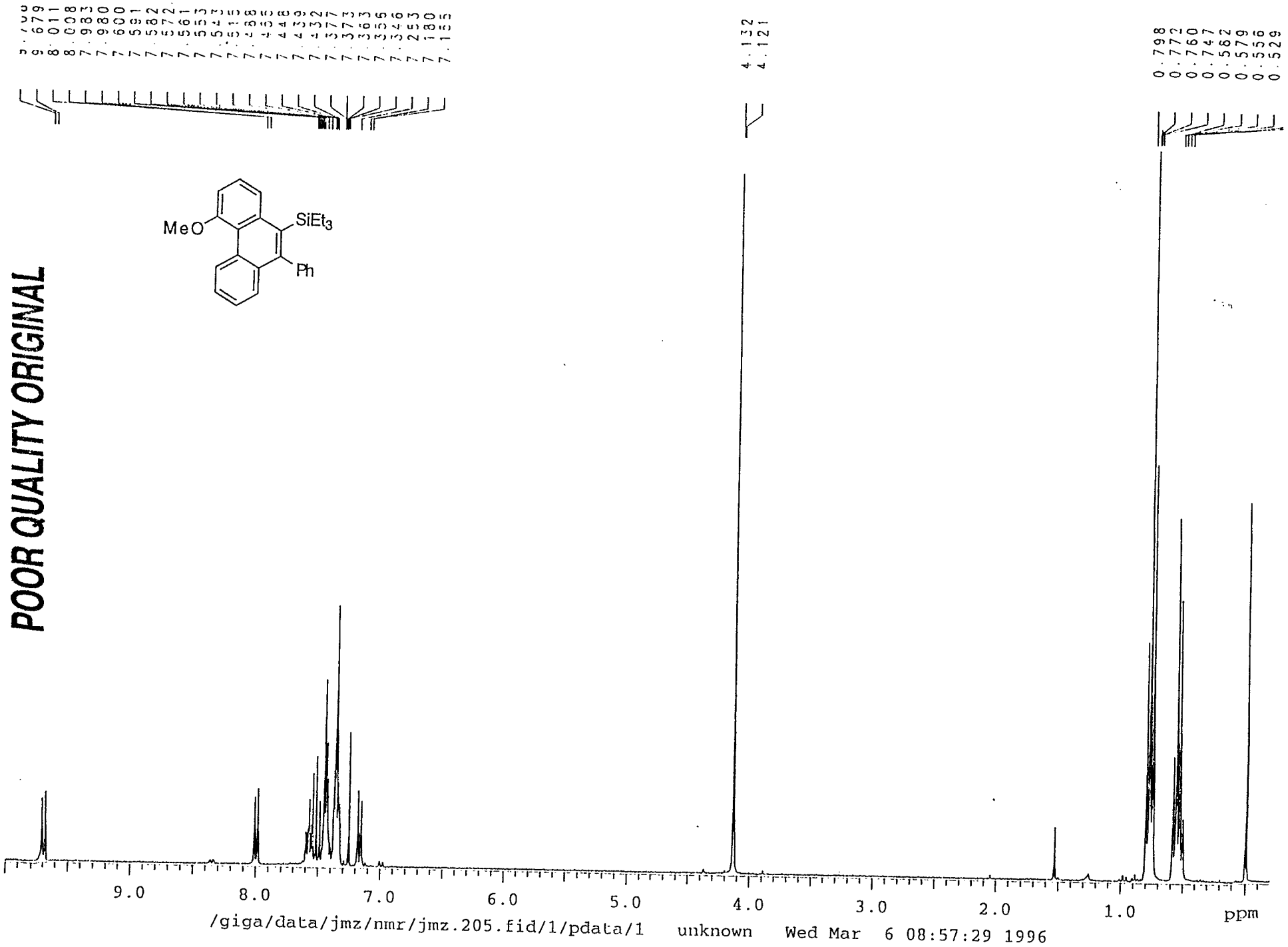

POOR QUALITY ORIGINAL

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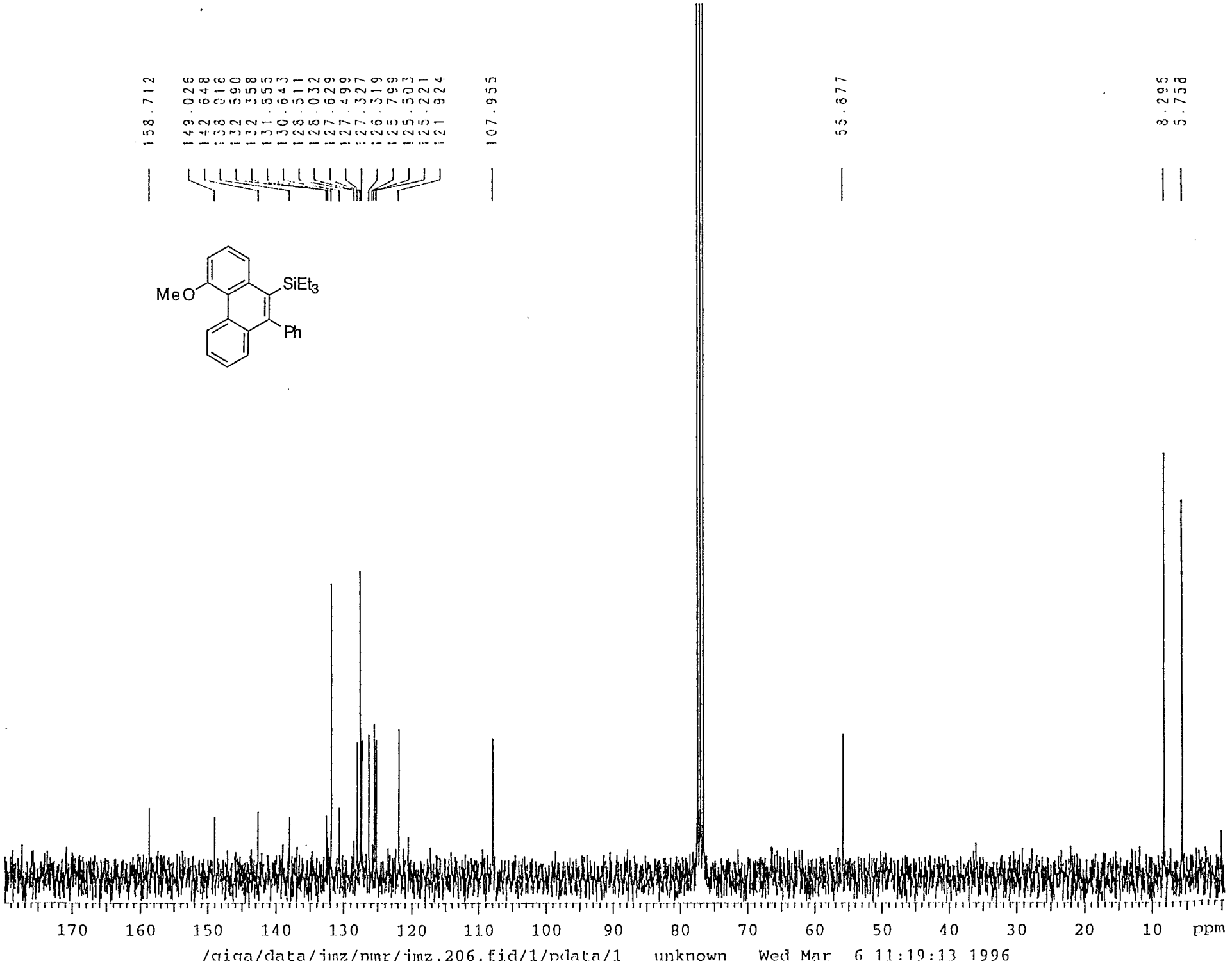

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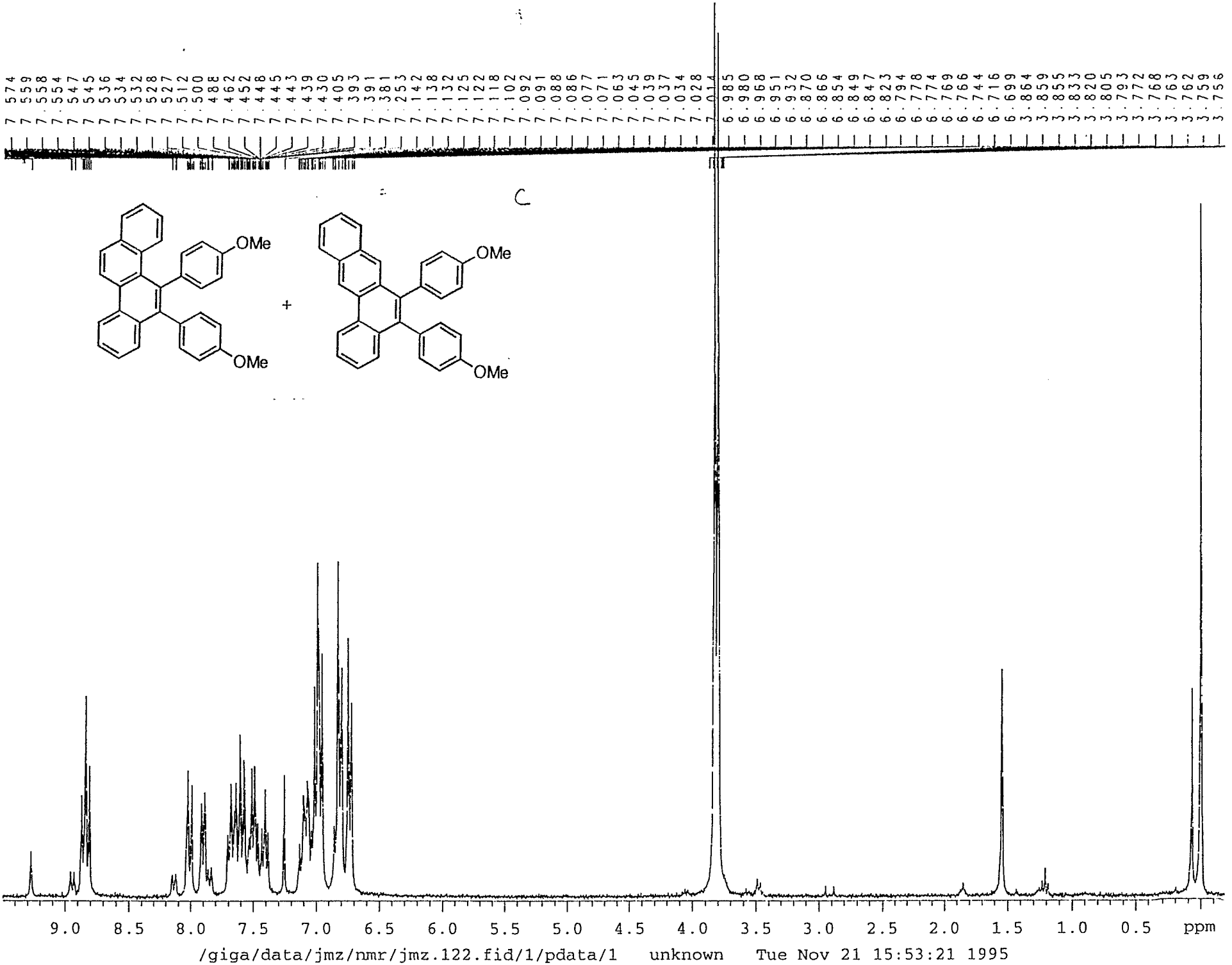

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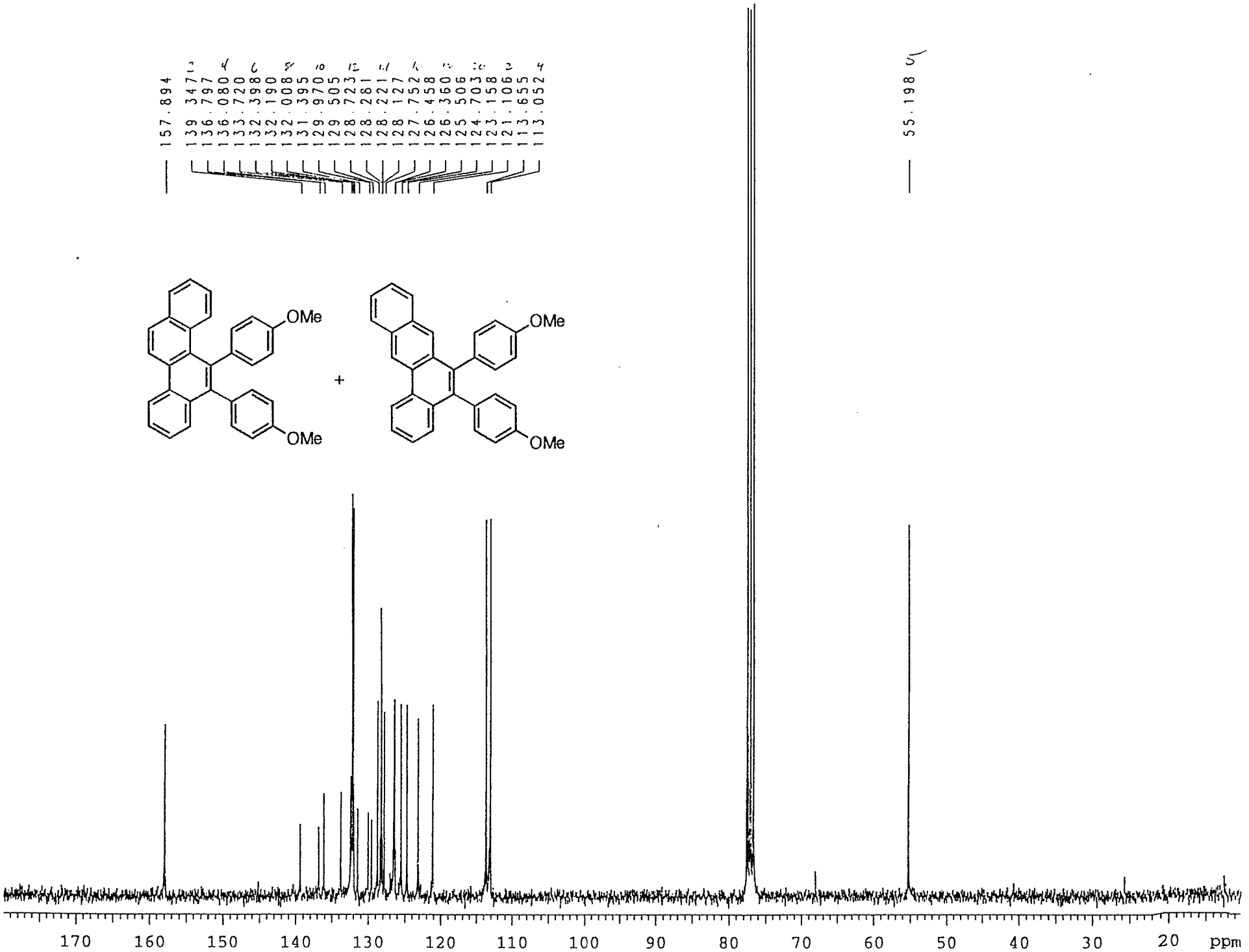

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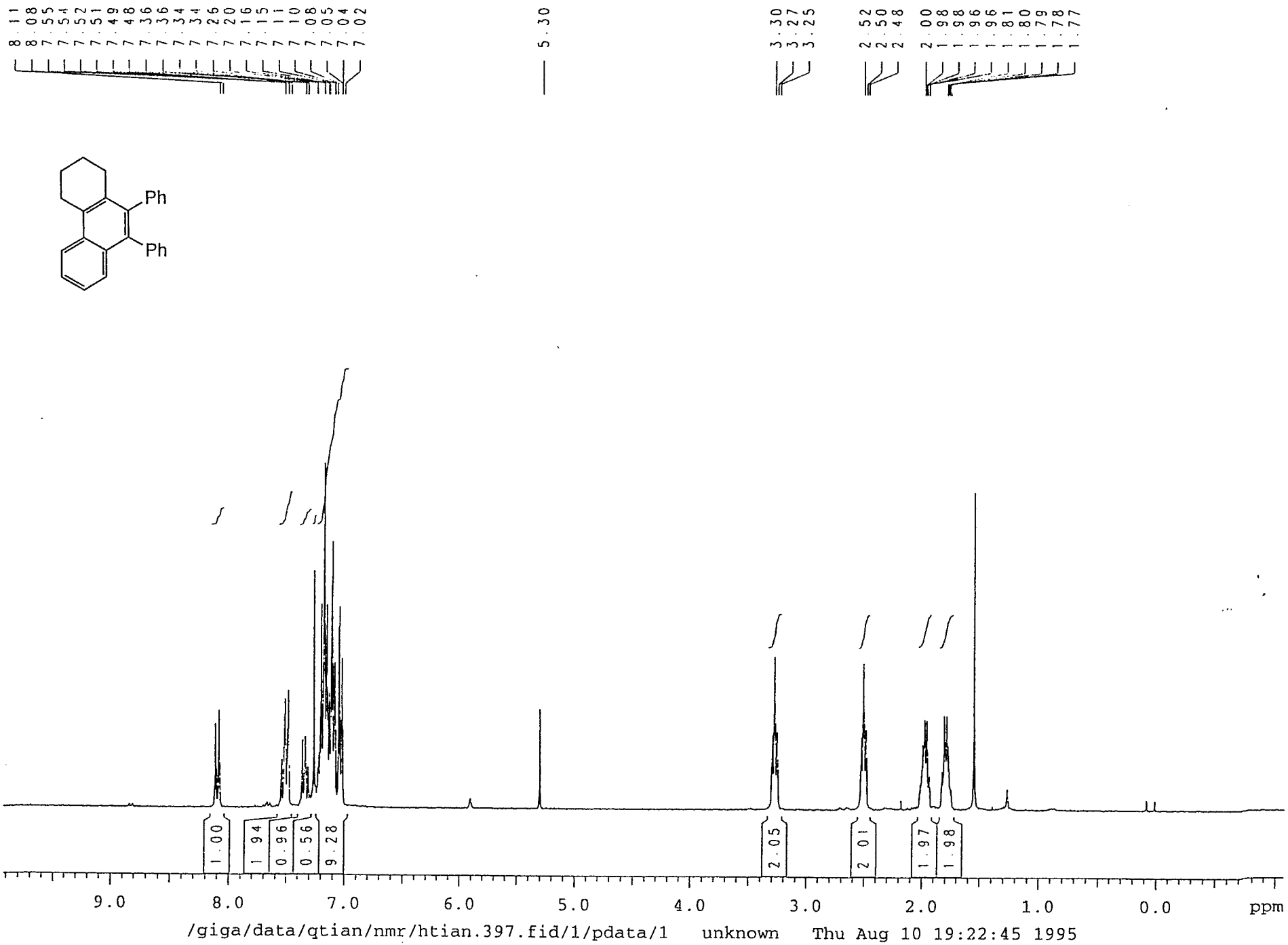

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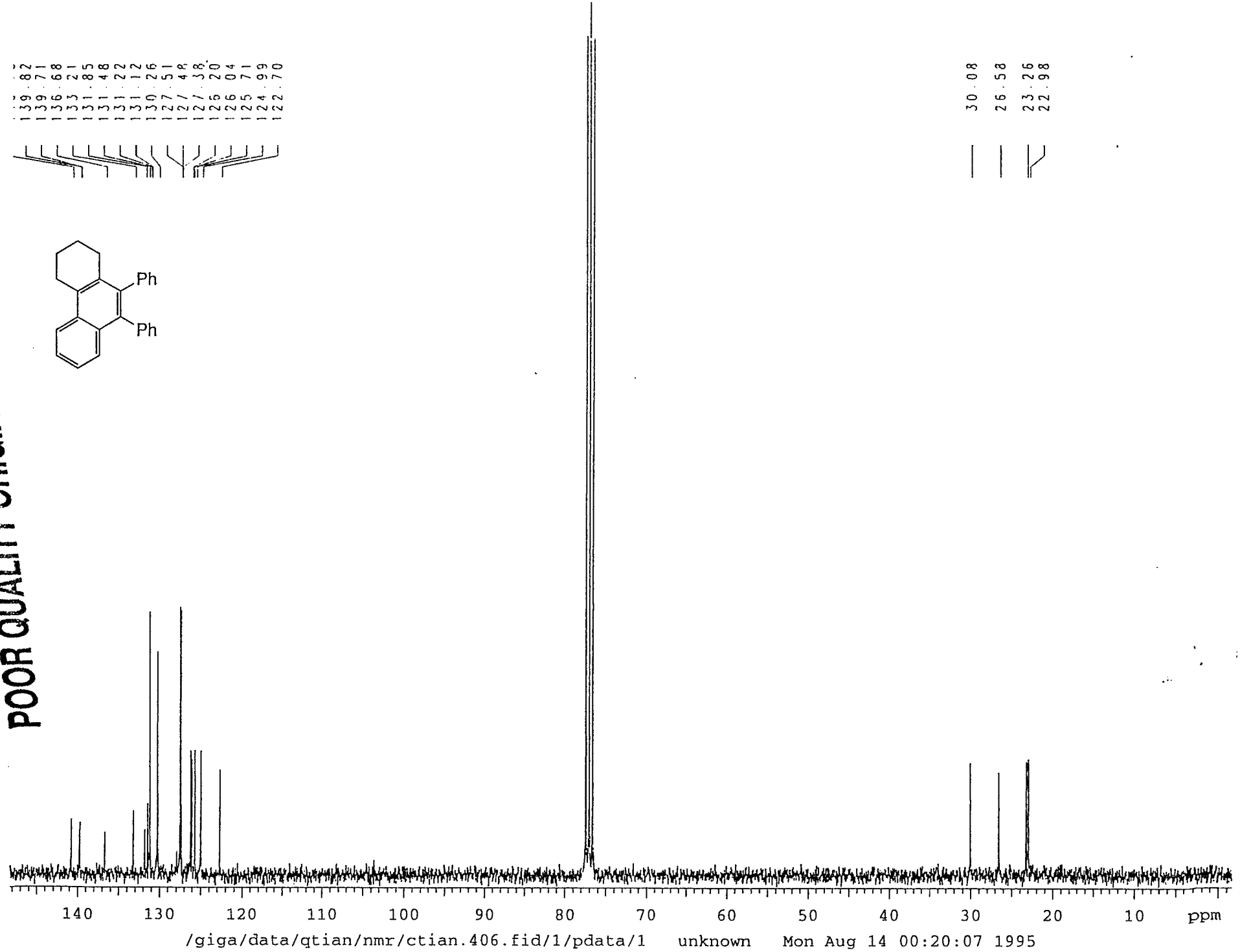

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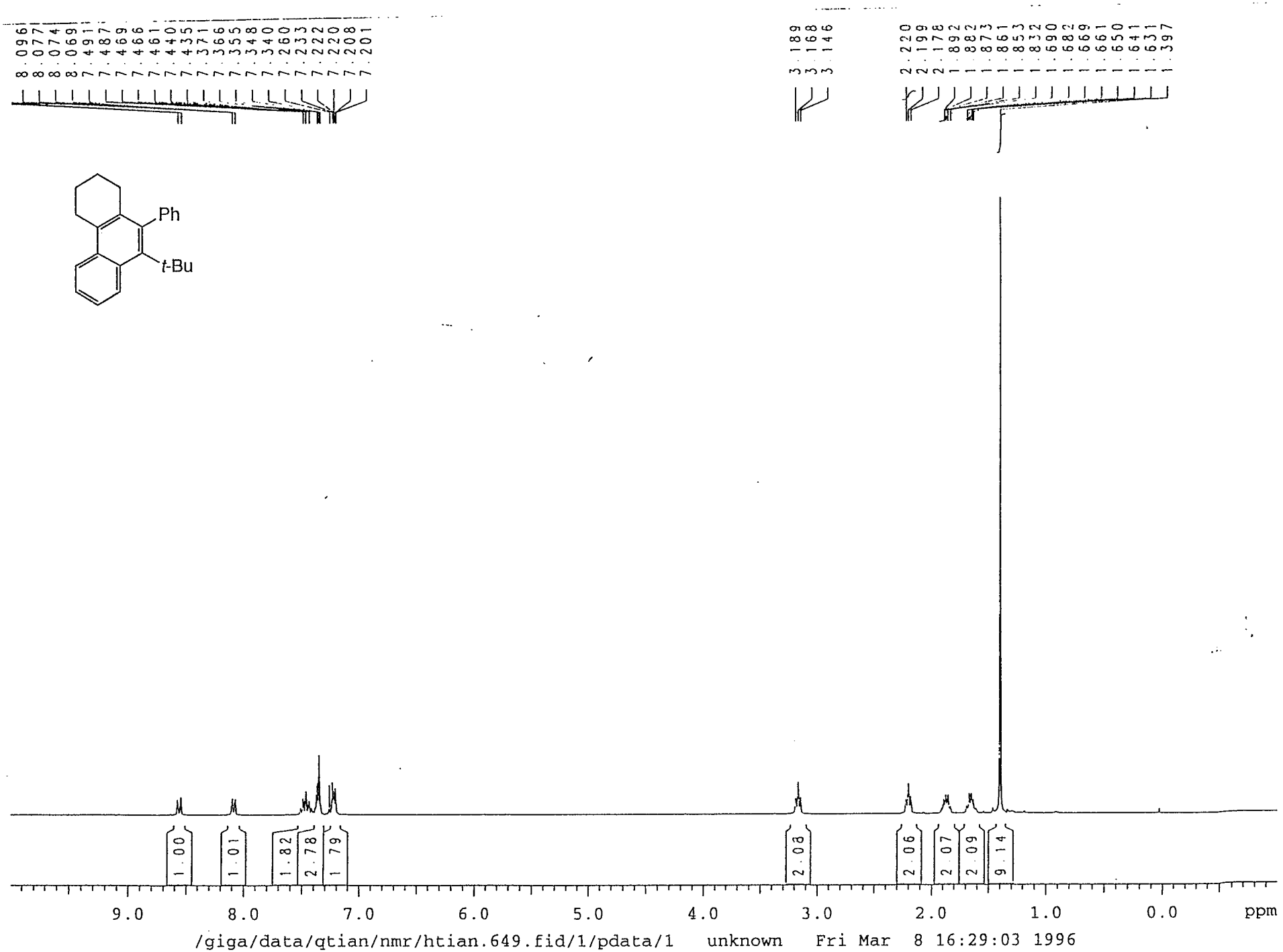

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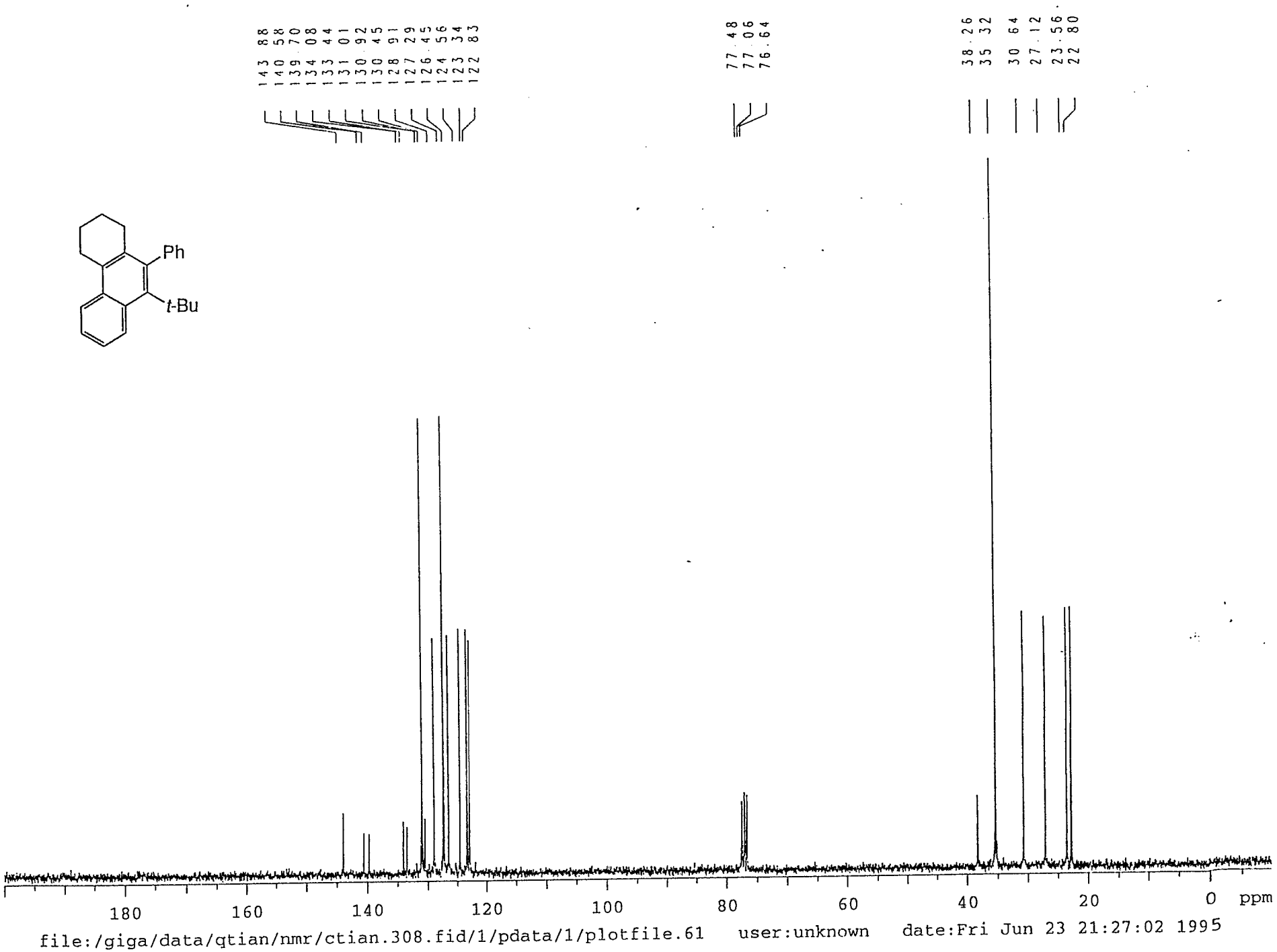

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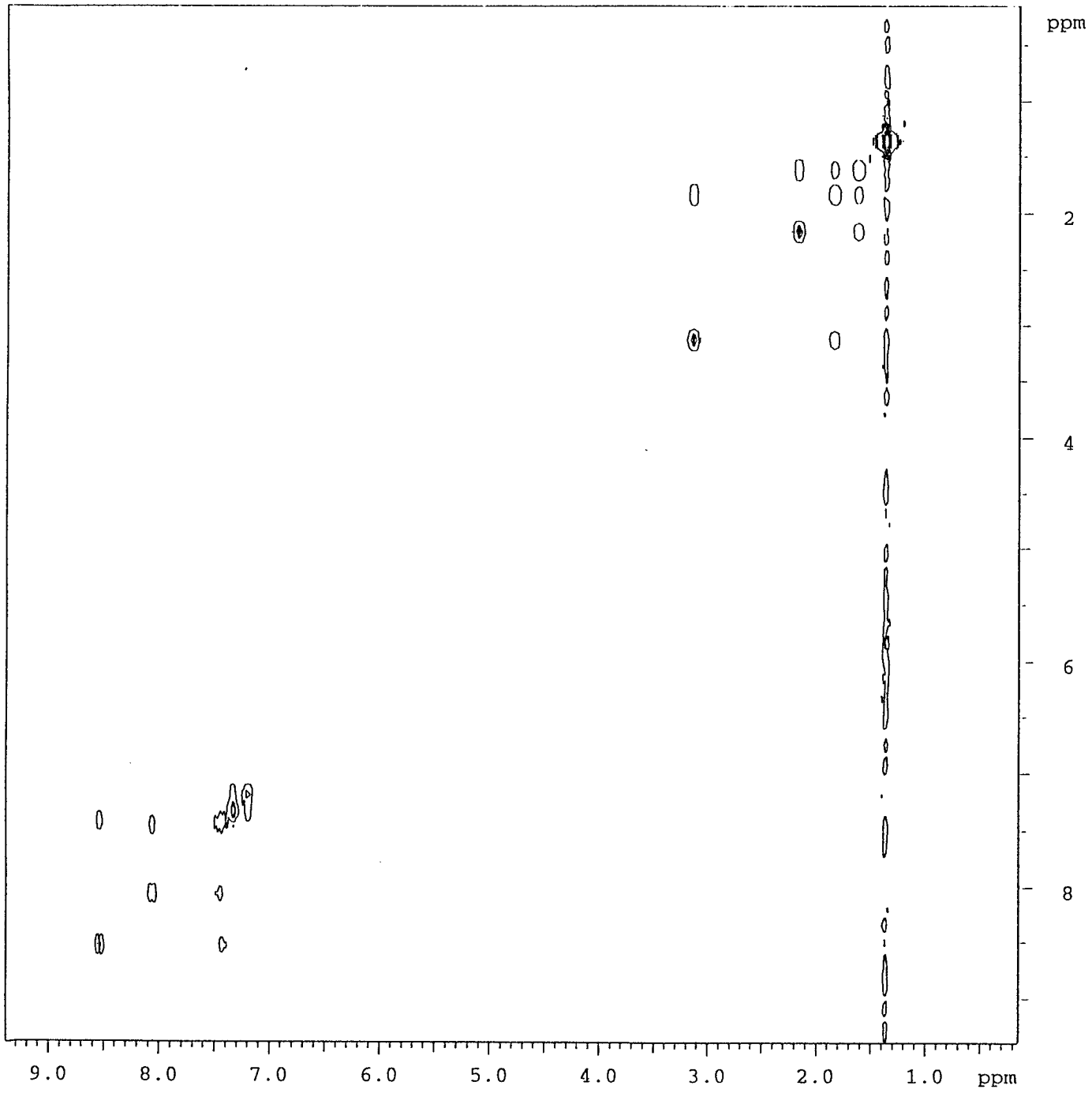

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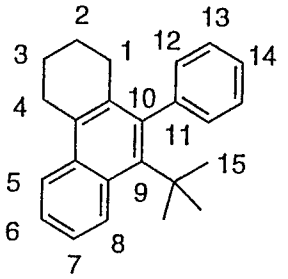

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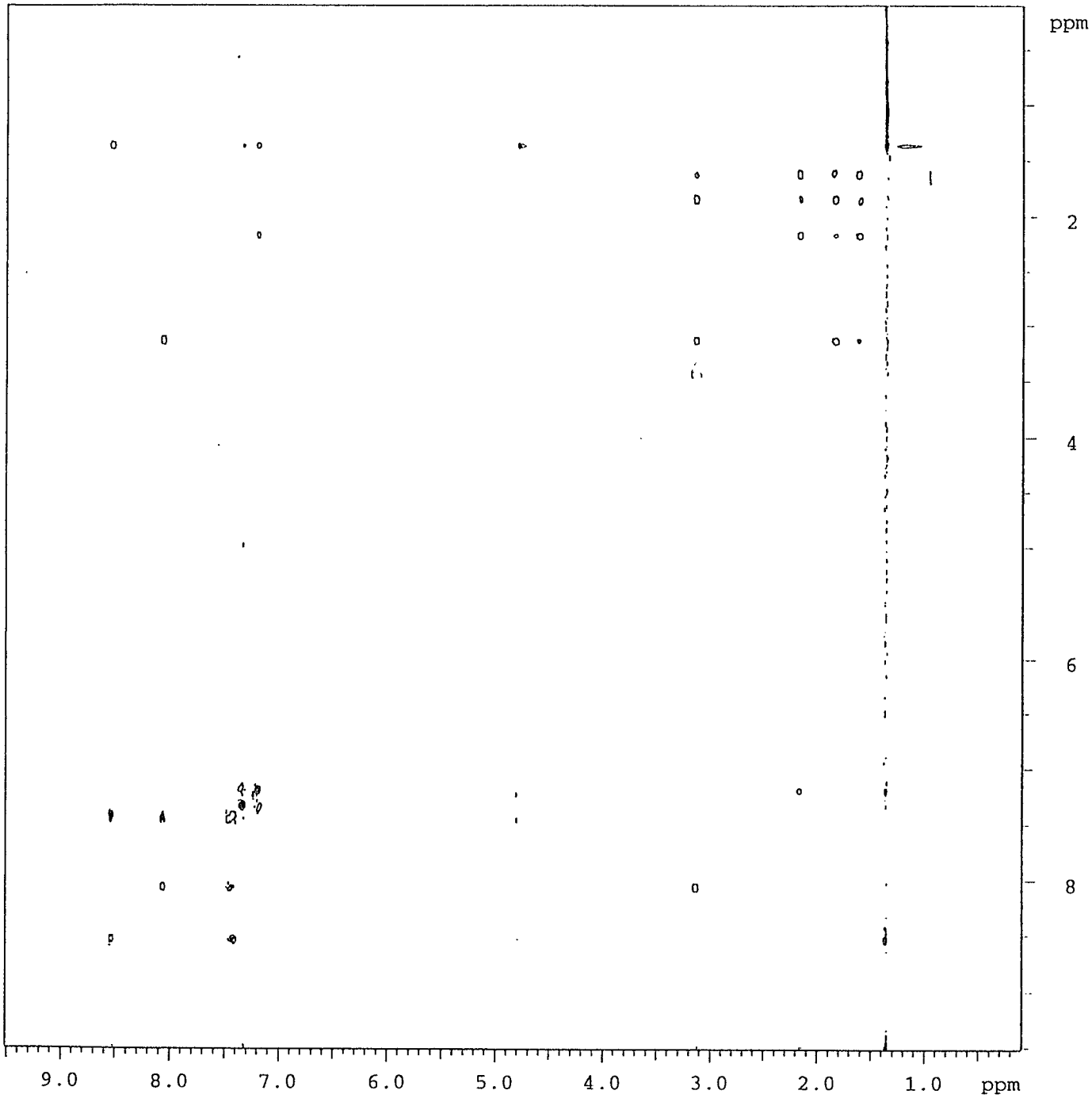

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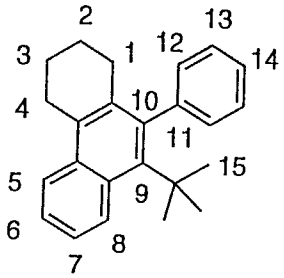

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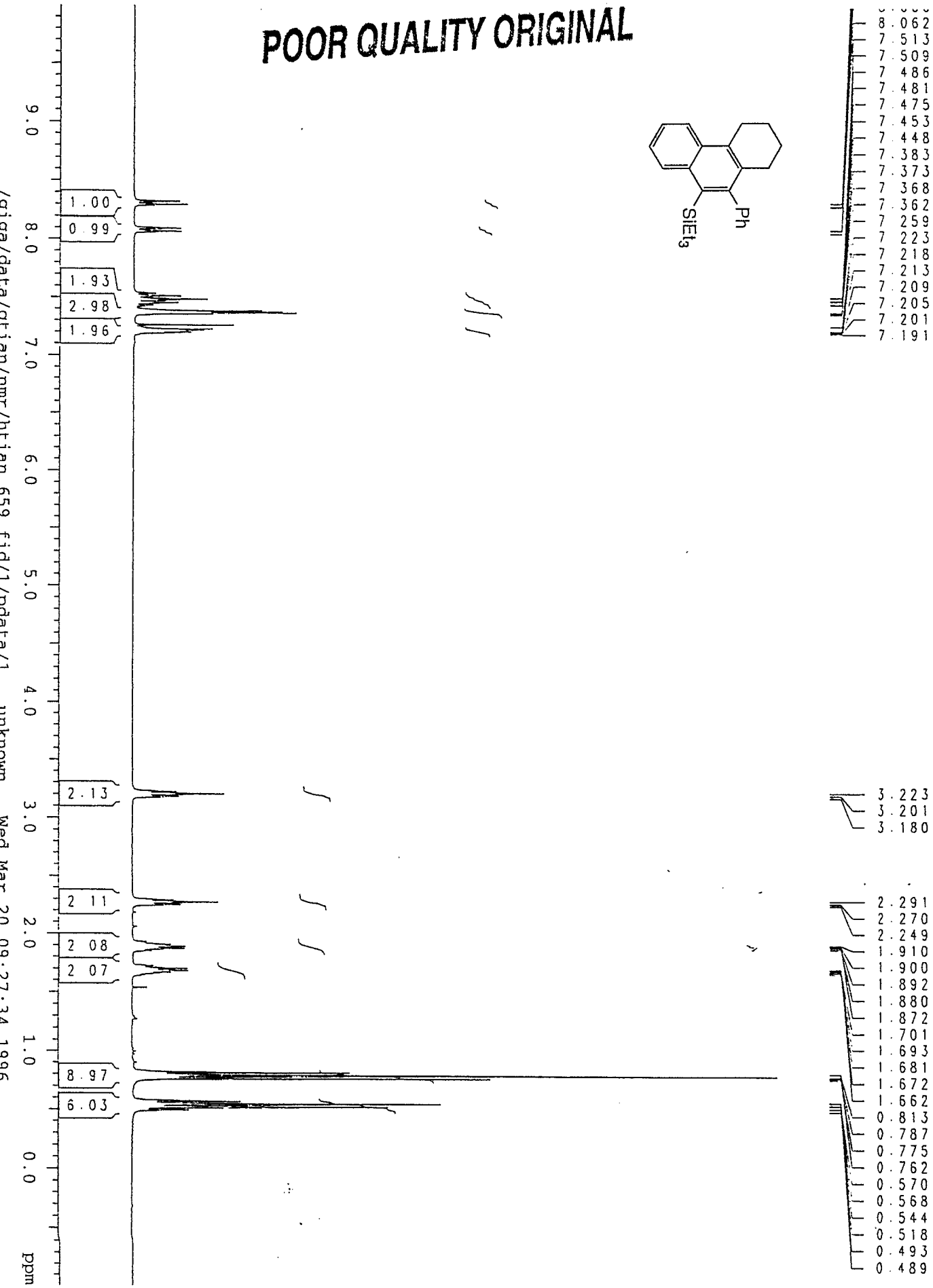
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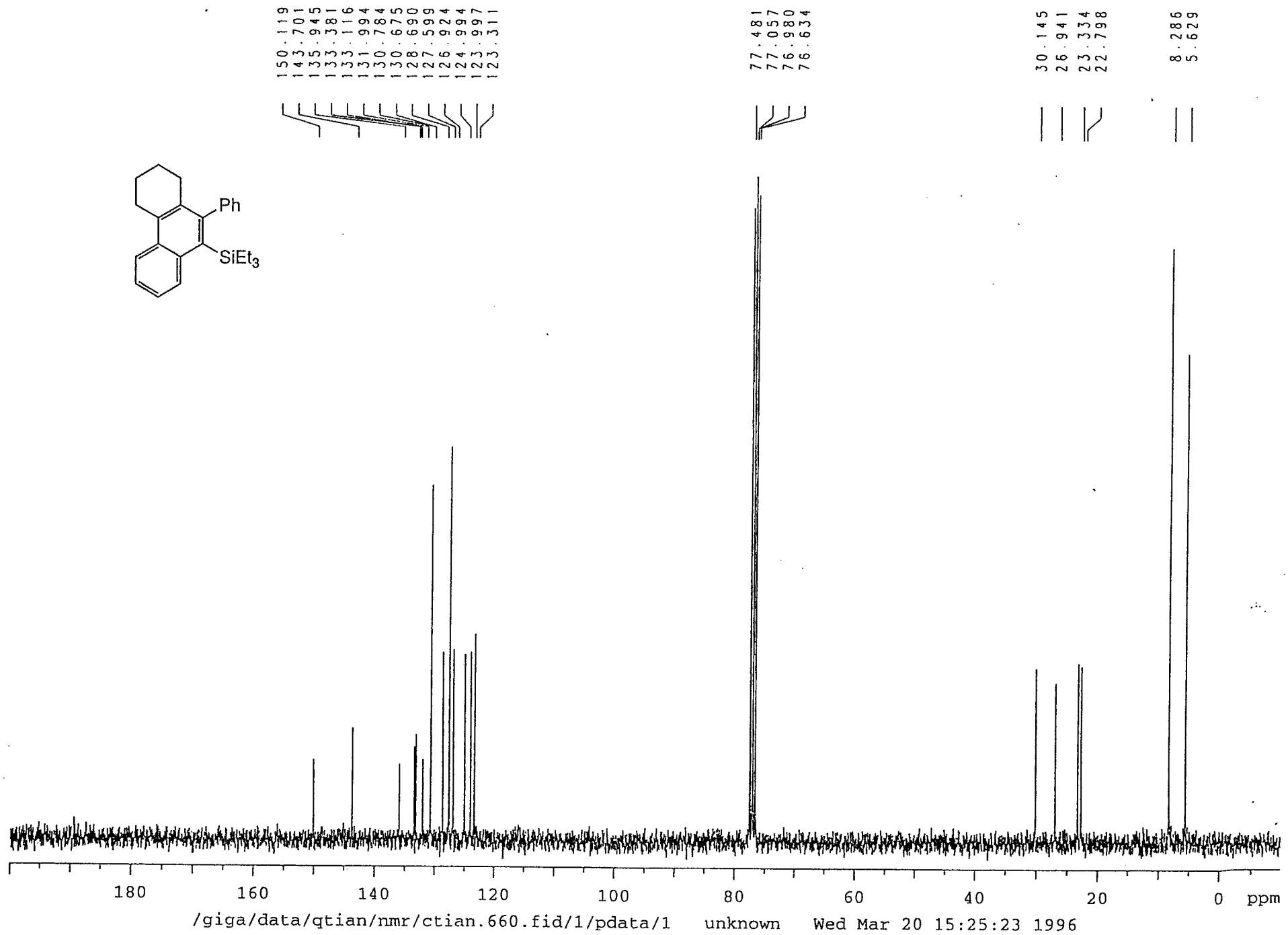

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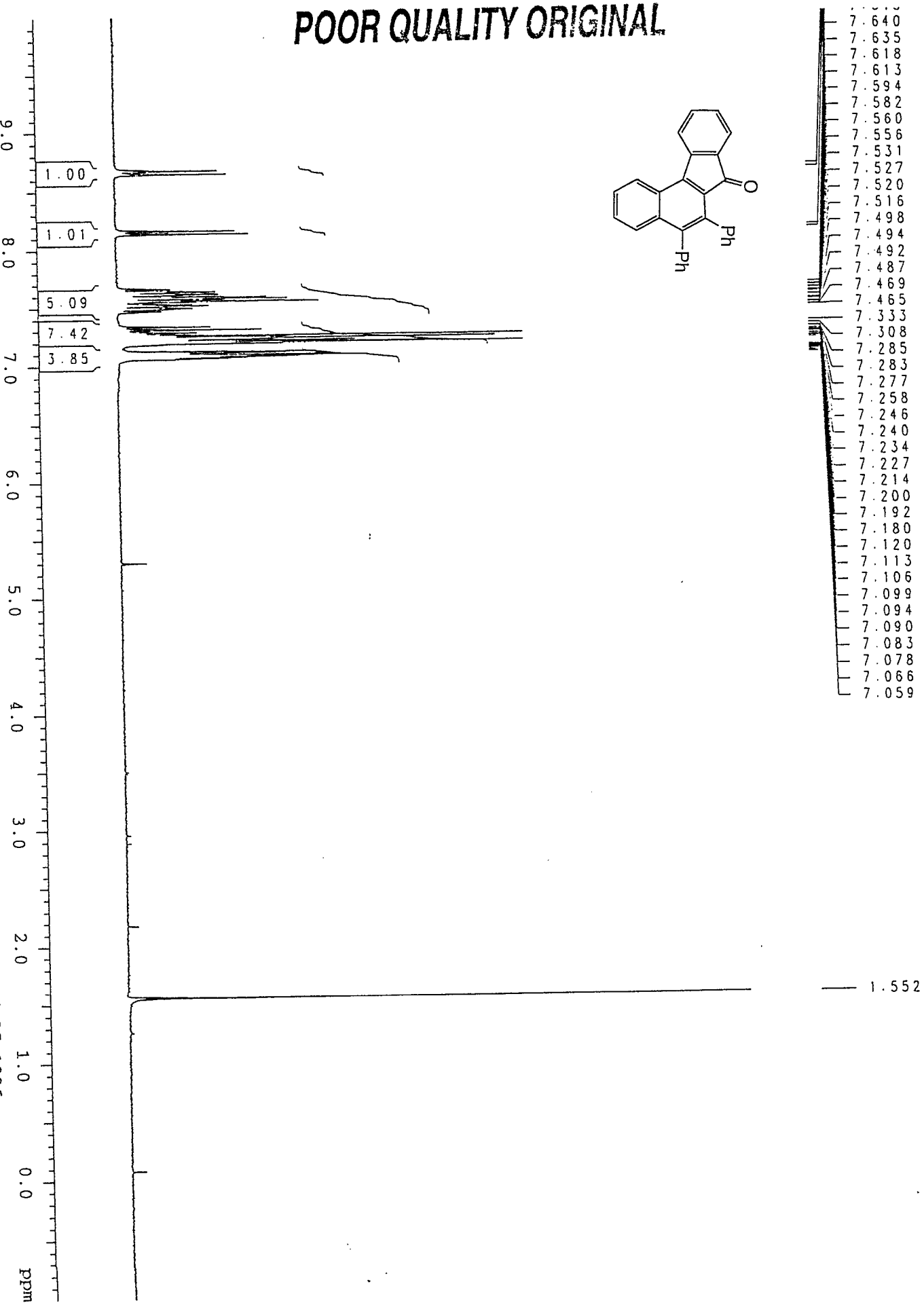

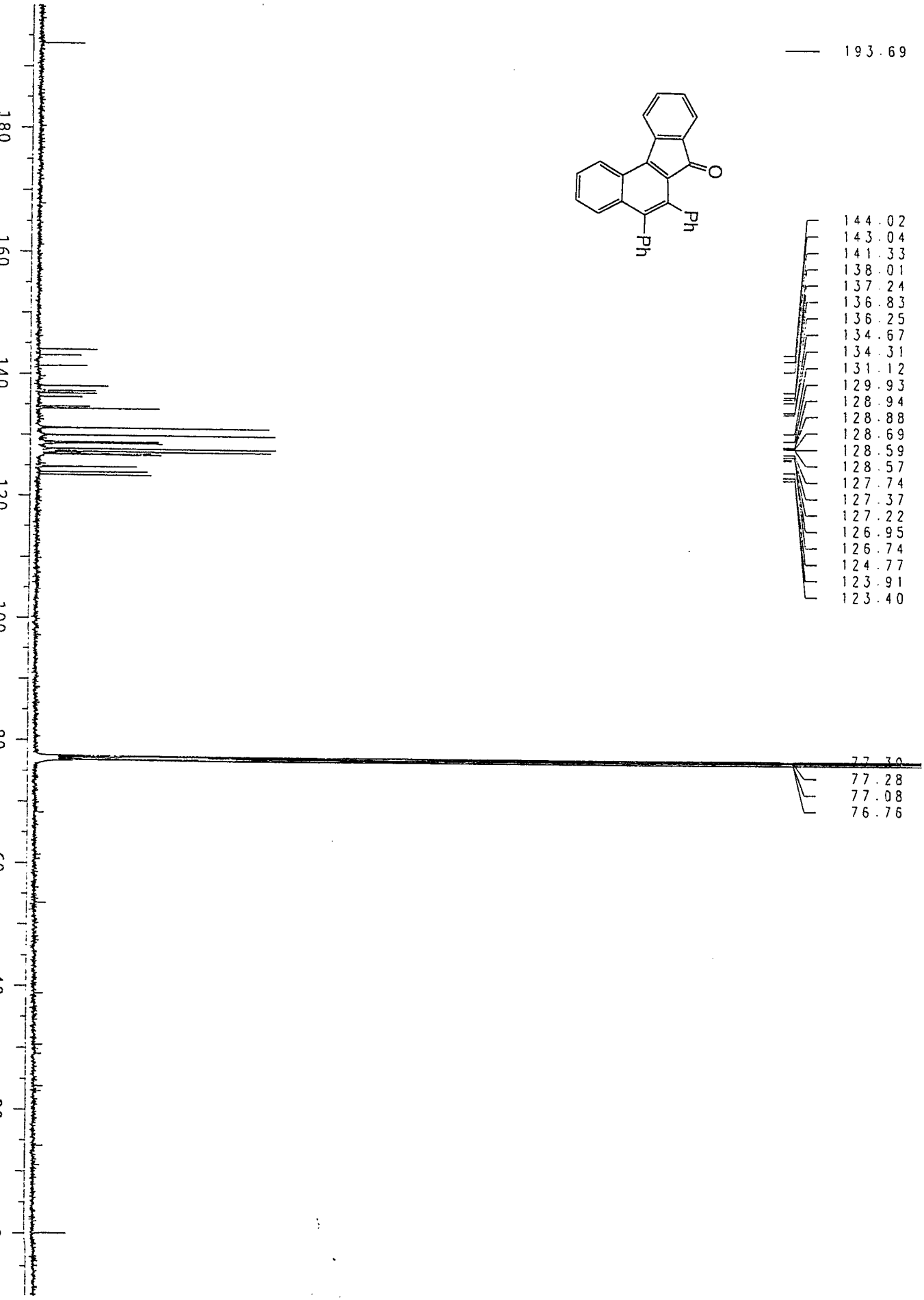
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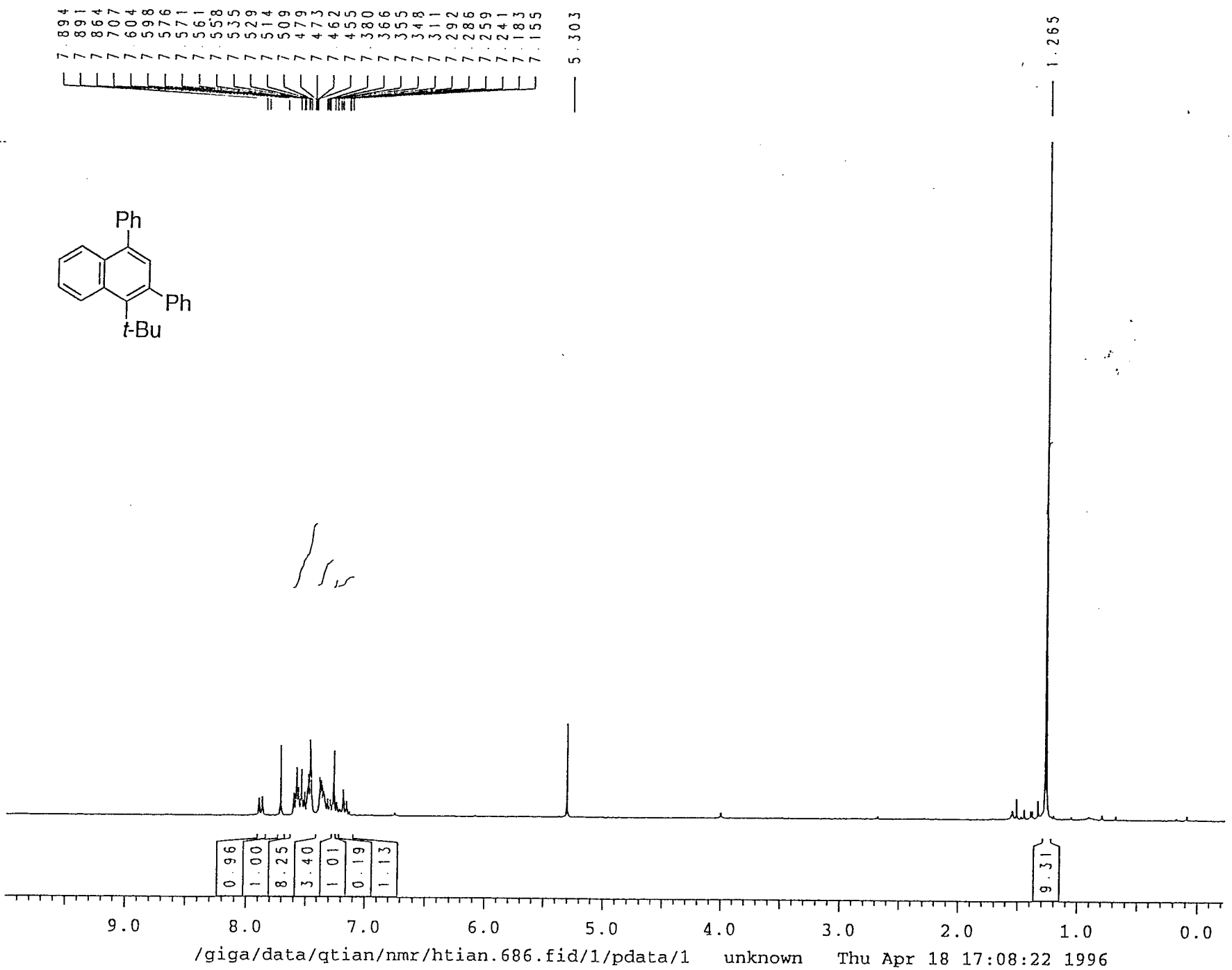

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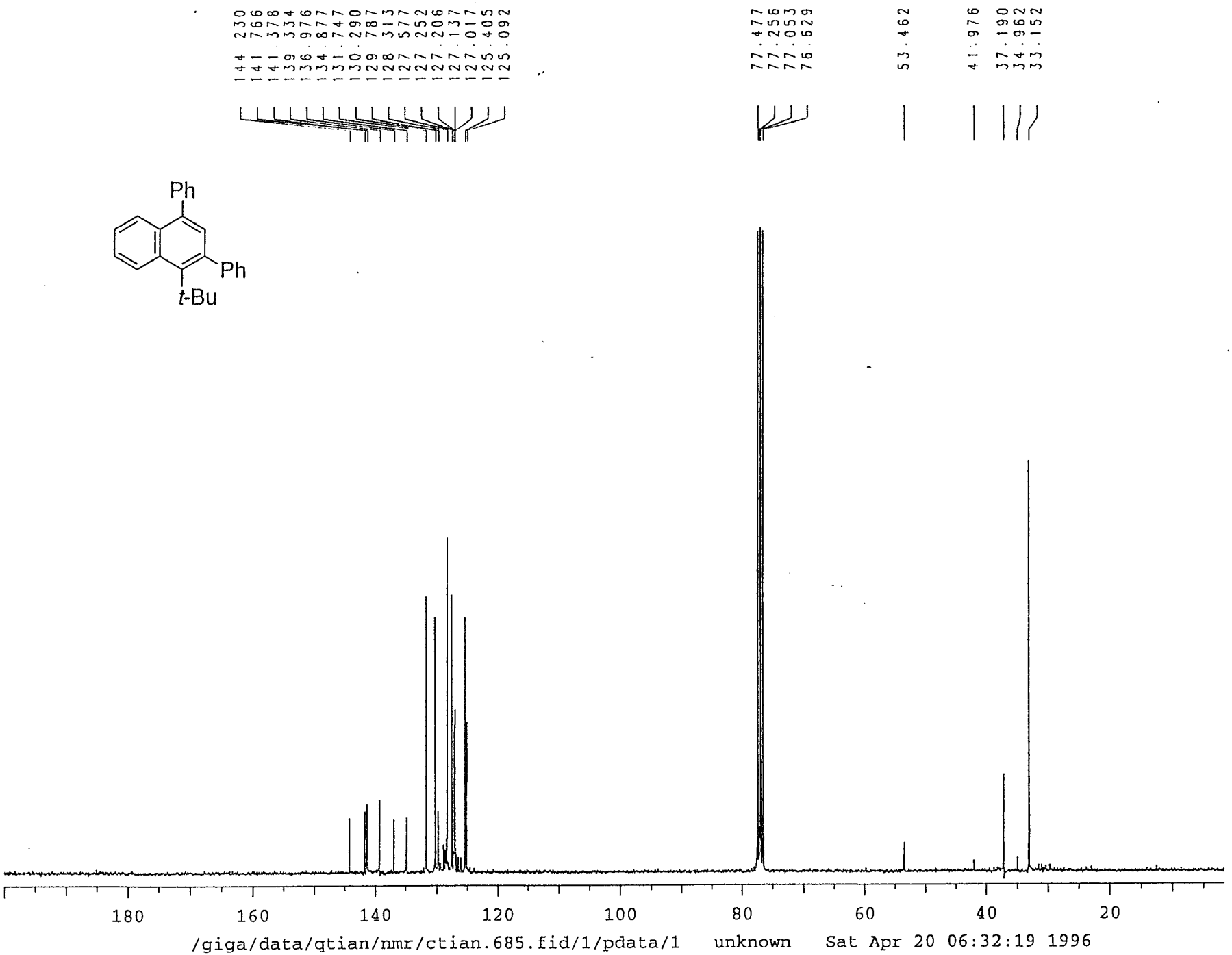

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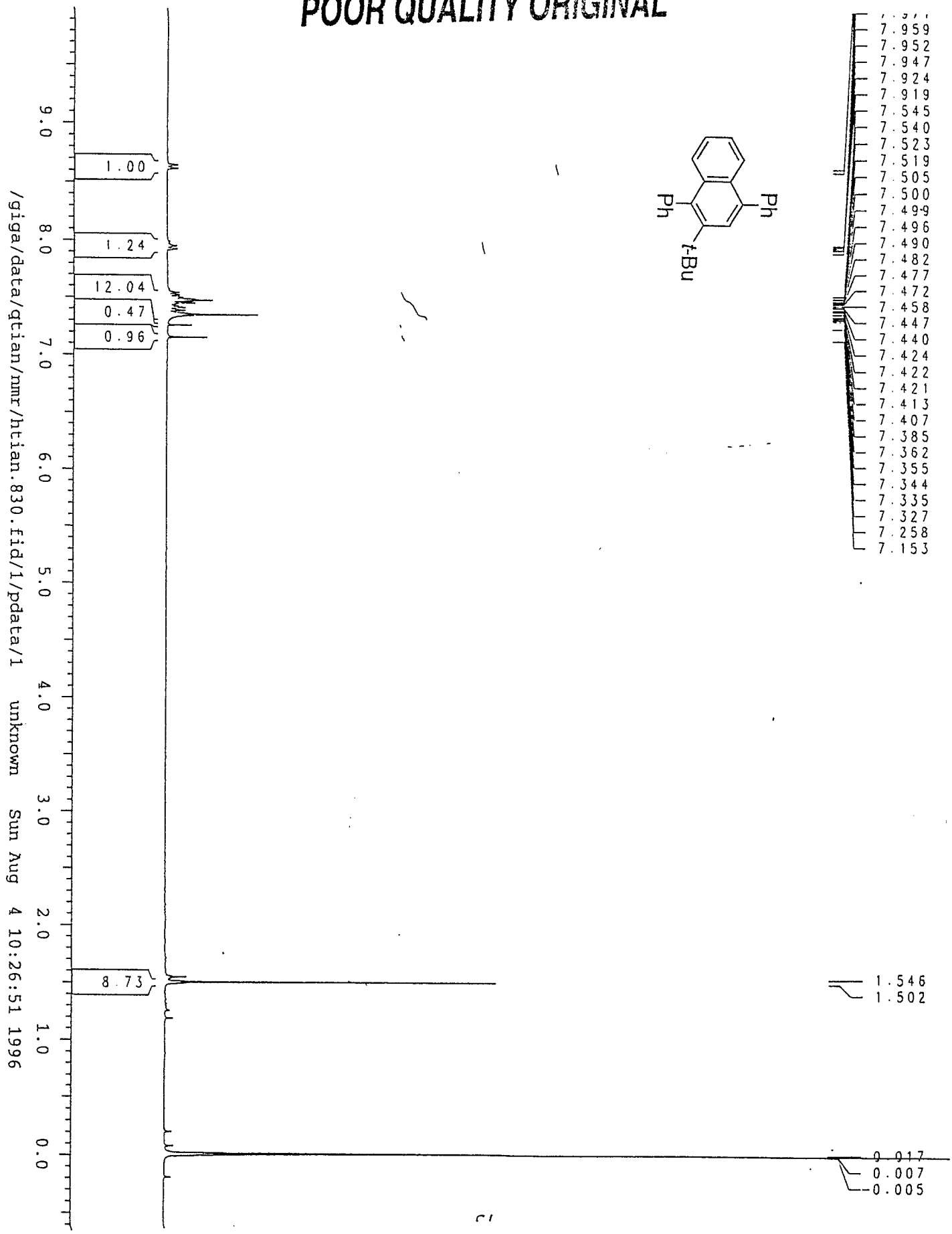

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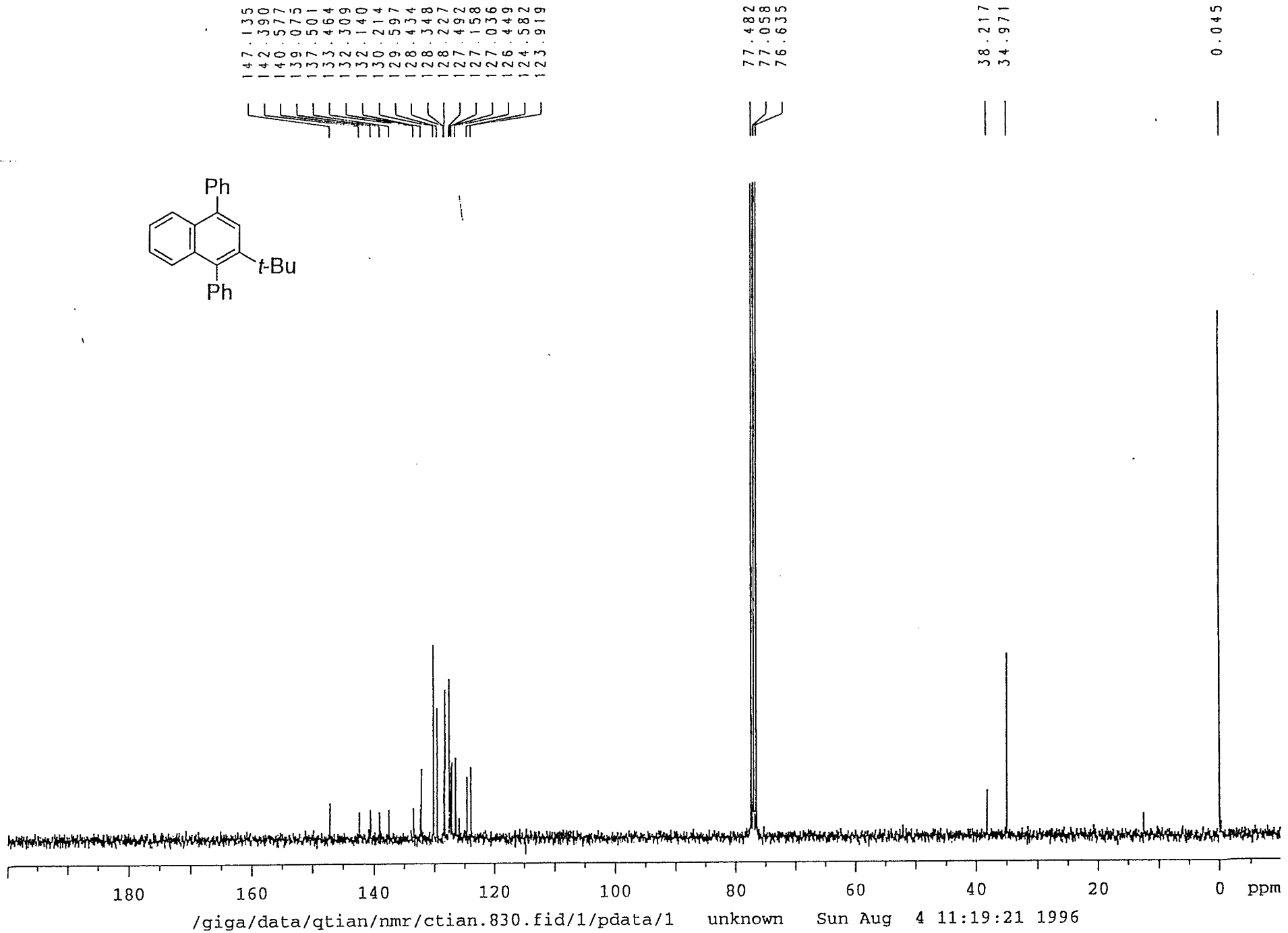

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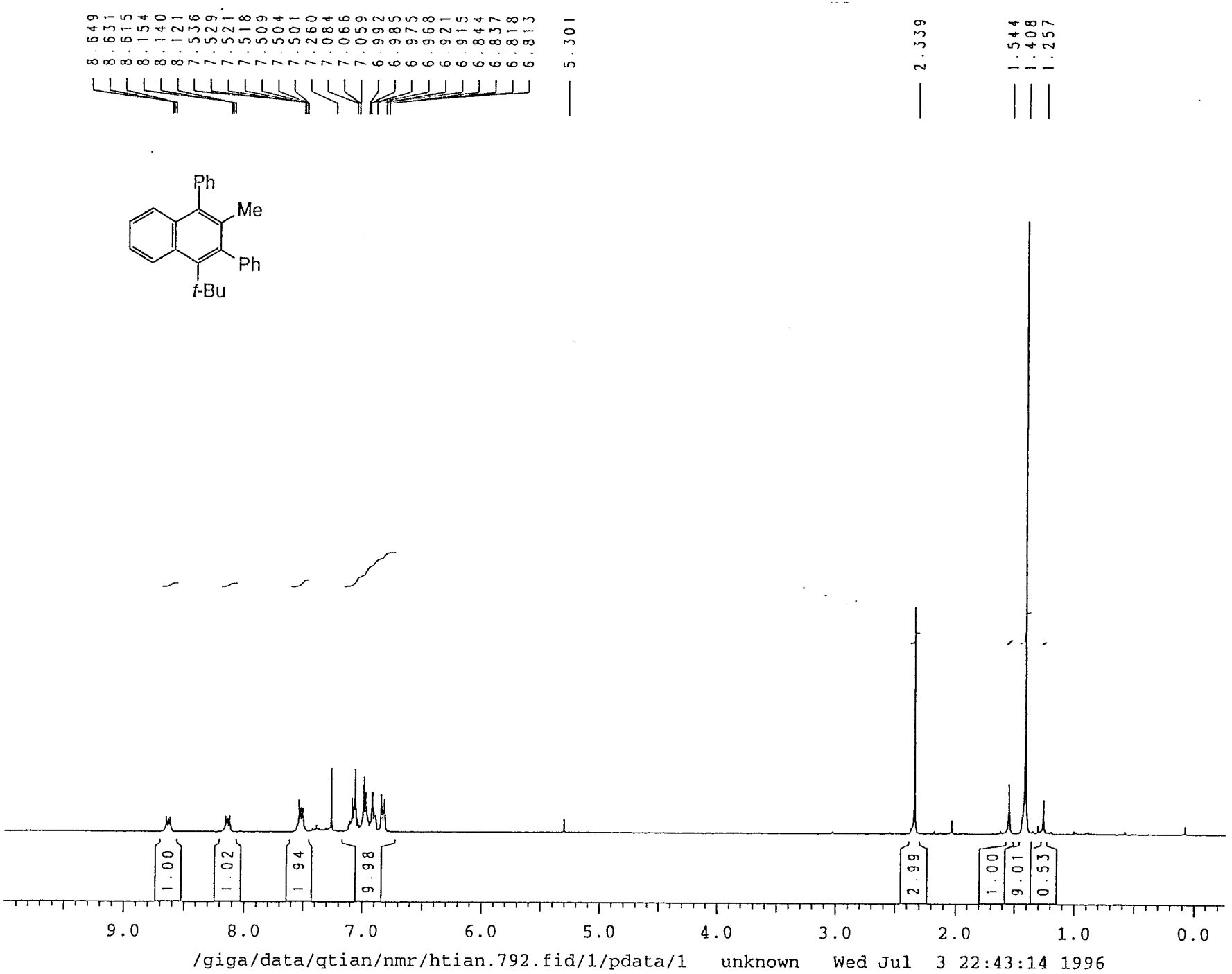
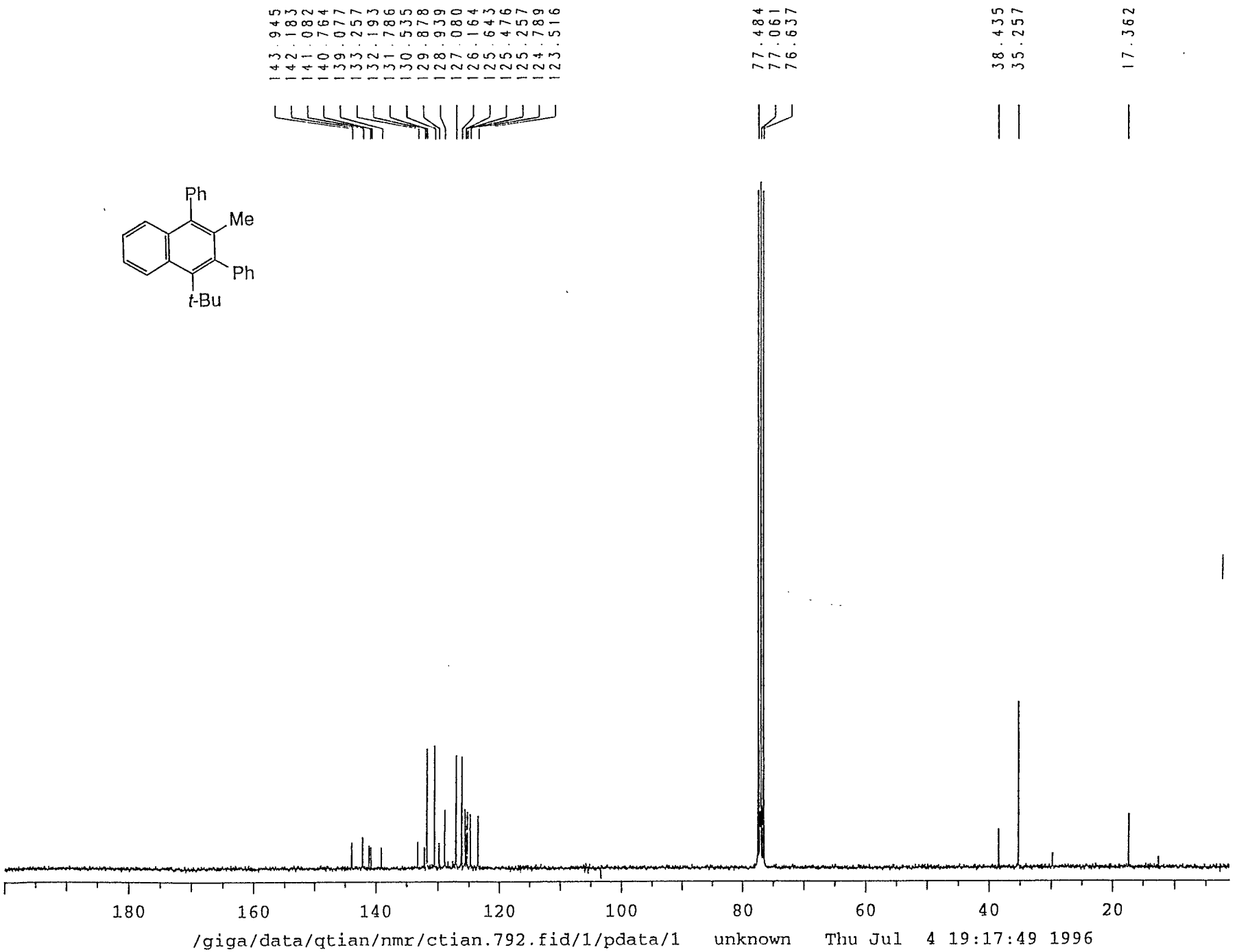
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