ANGEWANDTE CHEMIE
A Journal of the Gesellschaft Deutscher Chemiker

Supporting Information

for

Angew. Chem. Int. Ed. Z50184

69451 Weinheim, Germany

A New Diversity Oriented Synthesis of α-Amino Acids Derivatives by Silyltelluride-Mediated Radical Coupling Reaction of Imines and Isonitriles

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General. ^{1}H and ^{13}C NMR spectra were measured on Varian Gemini 2000 (300 MHz for ^{1}H) or JEOL JNM-A400 (400 MHz for ^{1}H) instruments, and are reported in parts per million (δ) from tetramethylsilane. IR spectra were recorded on a Shimadzu FTIR-8200, and are reported in cm^{-1}. FAB-MS spectra were recorded on JEOL IMS-300 spectrometer. 3-Nitrobenzyl alcohol was used as a matrix for FAB-MS. Melting points were measured by Yanaco micro melting point apparatus. Gel permeation chromatography was performed on Japan Analytical Industry LC-908 equipped with JAIGEL-1H and 2H using $CHCl_3$ as eluent.

Materials. Unless otherwise noted, materials obtained from commercial suppliers were used without further purification. EtCN was distilled successively from P_2O_5 and K_2CO_3 and stored over molecular sieves. Imines were prepared according to literatures.[1] 2,6-Xylylisonitrile was prepared by $POCl_3$ mediated dehydroxylation of *N*-formyl-2,6-xylylamine.[2]

Phenyl triethoxysilyl telluride (1). To a suspension of tellurium powder (12.8 g, 100 mmol) in THF (100 mL) was slowly added a solution of phenyllithium (1.04 M in cyclohexane/ether, 106 mL, 110 mmol) over 30 min at room temperature, and the resulting mixture was stirred for 1 h. To this solution was added chlorotriethoxysilane (21.9 g, 110 mmol) dropwisely over 15 min at room temperature, and the reaction mixture was stirred for 2 h. Solvent was removed in vacuo followed by distilllation (83-109 ^{o}C/0.55 mmHg) afforded the title compound in 62% yield (22.8 g) as orange oil. While **1** is sensitive to moisture, it can be stored for long period under inert atomosphere. IR (neat) 1476, 1435, 1391, 1165, 1076, 1017, 997, 785, 733, 693, 486, 455; ^{1}H NMR (300 MHz, $CDCl_3$) 1.19 (t, J = 7.1 Hz, 9 H),

3.85 (q, *J* = 7.0 Hz, 6 H), 7.07-7.16 (m, 2 H), 7.20-7.30 (m, 1 H), 7.72-7.82 (m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) 17.61, 59.65, 104.27, 127.37, 129.13, 140.22.

Silyltelluride-mediated coupling reaction.

Phenyl 2-(*N*-benzylamino)-*N*-(2,6-dimethylphenyl)-2-phenyltelluroacetimidate. A solution of phenyl triethoxylsilyl telluride (143.6 mg, 0.39 mmol), *N*-benzylidenebenzylamine (58.9 mg, 0.30 mmol) and 2,6-dimethylphenylisonitrile (78.7 mg, 0.60 mmol) in CD_3CN (0.6 mL) in a NMR tube was heated at 80 ^{o}C for 2 h. The crude mixture was purified by flash column chromatography (silica gel: 14.3 g, elution with 7% ethyl acetate in hexane) to give the title compound in 92% yield (148.3 mg) as light yellow solid. Mp 87.2-87.5 ^{o}C; IR (KBr) 1632, 1454, 1433, 858, 835, 754, 718, 731, 698, 692; ^{1}H NMR (300 MHz, $CDCl_3$) 2.07 (s, 3 H), 2.19 (s, 3 H), 2.72 (br s, 1 H), 3.90 (d, *J* = 13.2 Hz, 1 H), 4.01 (d, *J* = 13.5 Hz, 1 H), 4.59 (s, 1 H), 6.85-6.97 (m, 3 H), 7.02 (t, *J* = 7.7 Hz, 2 H), 7.08-7.16 (m, 2 H), 7.19-7.39 (m, 9 H), 7.52 (d, *J* = 8.1 Hz, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) 18.42, 18.59, 51.93, 69.56, 112.32, 124.46, 126.03, 126.07, 127.00, 127.48, 127.99, 128.15, 128.20, 128.40, 128.44, 128.49, 129.09, 139.10, 139.86, 141.71, 150.11, 167.19; HRMS (FAB) *m/z*: Calcd for $C_{29}H_{29}N_2Te$ $(M + H)^+$, 535.1400, found 535.1370; Anal. Calcd for $C_{29}H_{28}N_2Te$: C, 65.45; H, 5.30; N, 5.26. Found: C, 65.45; H, 5.34; N, 5.16.

Phenyl 2-(*N*-benzylamino)-*N*-(2,6-dimethylphenyl)-2-(*p*-methylphenyl)-telluroacetimidate. A solution of phenyl triethoxylsilyl telluride (143.6 mg, 0.39 mmol), *N*-(*p*-methylphenylmethylidene)benzylamine (62.4 mg, 0.30 mmol) and 2,6-dimethylphenylisonitrile (78.7 mg, 0.60 mmol) in CD_3CN (0.6 mL) in a NMR tube was heated at 80 ^{o}C for 2 h. The crude mixture was purified by flash column chromatography (silica gel: 14.3 g, elution with 7% ethyl acetate in hexane) to give the title compound in 93% yield (150.7 mg) as yellow solid. Mp 67.9-68.2 ^{o}C; IR (KBr) 1624, 1589, 1466, 845, 773, 733, 696; ^{1}H NMR (300 MHz, $CDCl_3$) 2.09 (s, 3 H), 2.20 (s, 3 H), 2.31 (s, 3 H), 2.77 (br s, 1 H), 3.90 (d, *J* = 13.2 Hz, 1 H), 4.00 (d, *J* = 13.5 Hz, 1 H), 4.55 (s, 1 H), 6.86-7.08 (m, 9 H), 7.22-7.38 (m, 6 H), 7.52 (d, *J* = 6.9 Hz, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) 18.48, 18.64, 21.13, 51.84, 69.26, 112.33, 124.42, 126.06, 126.10, 126.95, 127.88, 128.12, 128.35, 128.41, 128.91, 129.03, 135.93, 137.10, 139.86, 141.70, 150.11, 167.31; HRMS (FAB) *m/z*: Calcd for $C_{30}H_{31}N_2Te$ $(M + H)^+$, 549.1557, found 549.1528; Anal. Calcd for $C_{30}H_{30}N_2Te$: C, 65.97; H, 5.54; N, 5.13. Found: C, 65.77; H, 5.56; N, 4.88.

Phenyl 2-(*N*-benzylamino)-*N*-(2,6-dimethylphenyl)-2-(*p*-methoxyphenyl)-telluroacetimidate. A solution of phenyl triethoxylsilyl telluride (143.6 mg, 0.39 mmol), *N*-(*p*-methoxyphenylmethylidene)benzylamine (67.5 mg, 0.30 mmol) and 2,6-dimethylphenylisonitrile (78.8 mg, 0.60 mmol) in CD_3CN (0.6 mL) in a NMR tube was heated at 80 ^oC for 4 h. The crude mixture was purified by flash column chromatography (silica gel: 14.5 g, elution with 15% ethyl acetate in hexane) to give the title compound in 88% yield (149.0 mg) as yellow solid. Mp 74.1-74.3 ^oC; IR (KBr) 1624, 1611, 1588, 1510, 1466, 1252, 1177, 847, 781, 737, 696; ^1H NMR (300 MHz, $CDCl_3$) 2.09 (s, 3 H), 2.20 (s, 3 H), 2.64 (br s, 1 H), 3.77 (s, 3 H) , 3.90 (d, *J* = 13.2 Hz, 1 H), 4.00 (d, *J* = 13.2 Hz, 1 H), 4.53 (s, 1 H), 6.77 (d, *J* = 8.7 Hz, 2 H), 6.86-7.08 (m, 7 H), 7.21-7.39 (m, 6 H), 7.51 (d, *J* = 6.9 Hz, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) 18.53, 18.65, 51.86, 55.15, 68.92, 112.32, 113.59, 124.43, 126.03, 126.15, 126.98, 128.15, 128.40, 128.44, 129.06, 129.17, 131.07, 139.89, 141.73, 150.14, 158.96, 167.48; HRMS (FAB) *m/z*: Calcd for $C_{30}H_{31}N_2OTe$ $(M + H)^+$, 565.1506, found 565.1494; Anal. Calcd for $C_{30}H_{30}N_2OTe$: C, 64.09; H, 5.38; N, 4.98. Found: C, 63.83; H, 5.40; N, 4.96.

Phenyl 2-(*N*-benzylamino)-2-(*p*-dimethylaminophenyl)-*N*-(2,6-dimethyl-phenyl)-telluroacetimidate. A solution of phenyl triethoxylsilyl telluride (143.6 mg, 0.39 mmol), *N*-(*p*-dimethylaminophenylmethylidene)benzylamine (71.6 mg, 0.30 mmol) and 2,6-dimethylphenylisonitrile (78.8 mg, 0.60 mmol) in CD_3CN (0.6 mL) in a sealed NMR tube was heated at 80 ^oC for 4 h. The crude mixture was purified by flash column chromatography (silica gel: 15.0 g, elution with 15% ethyl acetate in hexane) to give the title compound in 91% yield (157.6 mg) as yellow solid. Mp 97.7-98.1 ^oC; IR (KBr) 1655, 1613, 1522, 1433, 760, 731; ^1H NMR (300 MHz, $CDCl_3$) 2.12 (s, 3 H), 2.17 (s, 3 H), 2.57 (br s, 1 H), 2.91 (s, 6 H) , 3.90 (d, *J* = 13.2 Hz, 1 H), 4.00 (d, *J* = 13.2 Hz, 1 H), 4.49 (s, 1 H), 6.60 (d, *J* = 8.7 Hz, 2 H), 6.83-7.09 (m, 7 H), 7.17-7.40 (m, 6 H), 7.52 (d, *J* = 7.5 Hz, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) 18.59, 18.65, 40.48, 51.83, 69.21, 112.23, 112.53, 124.25, 126.01, 126.24, 126.45, 126.86, 128.14, 128.26,128.32, 128.88, 140.06, 141.67, 149.99, 150.08, 167.54; HRMS (FAB) *m/z*: Calcd for $C_{31}H_{34}N_3Te$ $(M + H)^+$, 578.1822, found 578.1813; Anal. Calcd for $C_{31}H_{33}N_3Te$: C, 64.73; H, 5.78; N, 7.31. Found: C, 64.59; H, 5.90; N, 7.27.

Phenyl 2-(*N*-benzylamino)-2-(*p*-carbomethoxyphenyl)-*N*-(2,6-dimethylphenyl)-telluroacetimidate. A solution of phenyl triethoxylsilyl telluride (143.6 mg, 0.39 mmol), *N*-

(*p*-carbomethoxyphenylmethylidene)benzylamine (76.5 mg, 0.30 mmol) and 2,6-dimethylphenylisonitrile (79.0 mg, 0.60 mmol) in CD_3CN (0.6 mL) in a sealed NMR tube was heated at 80 ^oC for 2 h. The crude mixture was purified by flash column chromatography (silica gel: 15.0 g, elution with 15% ethyl acetate in hexane) to give the title compound in 50% yield (89.1 mg) as light yellow solid. Mp 89.7-90.1 ^oC; IR (KBr) 1725, 1622, 1284, 1115, 843, 762; ^1H NMR (300 MHz, $CDCl_3$) 2.05 (s, 3 H), 2.24 (s, 3 H), 2.68 (br s, 1 H), 3.89 (d, *J* = 12.9 Hz, 1 H), 3.90 (s, 3 H), 3.99 (d, *J* = 13.2 Hz, 1 H), 4.65 (s, 1 H), 6.89-7.01 (m, 3 H), 7.06 (t, *J* = 7.7 Hz, 2 H), 7.15 (d, *J* = 6.6 Hz, 2 H), 7.22-7.39 (m, 6 H), 7.52 (d, *J* = 7.8 Hz, 2 H), 7.88-7.96 (m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) 18.41, 18.62, 51.86, 52.05, 69.06, 112.01, 124.69, 125.98, 126.06, 127.15, 127.99, 128.15,128.47, 128.55, 128.59, 128.81, 129.20, 129.32, 129.53, 139.59, 141.80, 144.32, 150.20, 166.57, 166.92; HRMS (FAB) *m/z*: Calcd for $C_{31}H_{31}O_2N_2Te$ $(M + H)^+$, 593.1455, found 593.1454; Anal. Calcd for $C_{31}H_{30}O_2N_2Te$: C, 63.09; H, 5.12; N, 4.75. Found: C, 63.01; H, 5.42; N, 4.47.

Phenyl 2-(*N*-benzylamino)-*N*-(2,6-dimethylphenyl)-tellurohexanoimidate. A solution of phenyl triethoxylsilyl telluride (143.6 mg, 0.39 mmol), *N*-pentylidenebenzylamine (52.3 mg, 0.30 mmol) and 2,6-dimethylphenylisonitrile (79.1 mg, 0.60 mmol) in CD_3CN (0.6 mL) in a sealed NMR tube was stood at room temperature for 24 h. The crude mixture was purified by flash column chromatography (silica gel: 14.0 g, elution with 5% ethyl acetate in hexane) to give the title compound in 63% yield (96.6 mg) as yellow oil. IR (neat) 2955, 1628, 1592, 1472, 1435, 847, 768, 733, 695; ^1H NMR (300 MHz, $CDCl_3$) 0.80 (t, *J* = 7.2 Hz, 3 H), 0.84-0.97 (m, 1 H), 1.08 (quintet, *J* = 7.3 Hz, 2 H), 1.32-1.52 (m, 2 H), 1.64-1.77 (m, 1 H), 2.17 (s, 3 H), 2.27 (s, 3 H), 2.34 (br s, 1 H), 3.27 (dd, *J* = 9.3, 3.6 Hz, 1 H), 3.81 (d, *J* = 13.2 Hz, 1 H), 4.12 (d, *J* = 12.9 Hz, 1 H), 6.89-7.09 (m, 5 H), 7.20-7.37 (m, 6 H), 7.61-7.69 (m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) 14.01, 18.18, 18.64, 22.39, 28.34, 34.81, 52.12, 65.78, 111.86, 124.43, 125.97, 126.12, 126.89, 128.18, 128.32, 128.50, 129.19, 140.36, 141.80, 150.31, 170.98; HRMS (FAB) *m/z*: Calcd for $C_{27}H_{33}N_2Te$ $(M + H)^+$, 515.1706, found 515.1706; Anal. Calcd for $C_{27}H_{32}N_2Te$: C, 63.32; H, 6.30; N, 5.47. Found: C, 63.17; H, 6.33; N, 5.36.

Phenyl 2-(*N*-benzylamino)-2-cyclohexyl-*N*-(2,6-dimethylphenyl)-telluroacetimidate. A solution of phenyl triethoxylsilyl telluride (143.6 mg, 0.39 mmol), *N*-(cyclohexylmethylidene)benzylamine (60.7 mg, 0.30 mmol) and 2,6-dimethylphenylisonitrile

(79.1 mg, 0.60 mmol) in CD_3CN (0.6 mL) in a sealed NMR tube was stood at room temperature for 24 h. The crude mixture was purified by flash column chromatography (silica gel: 14.0 g, elution with 5% ethyl acetate in hexane) to give the title compound in 92% yield (149.8 mg) as white solid. Mp 108.6-109.0 ^oC; IR (KBr) 2921, 1636, 839, 770, 729, 696, 689; ^1H NMR (300 MHz, $CDCl_3$) 0.66 (distorted d, *J* = 12.9 Hz, 1 H), 0.98-1.33 (m, 5 H), 1.48-1.69 (m, 3 H), 1.71-1.82 (m, 1 H), 1.91 (distorted d, *J* = 11.7 Hz, 1 H), 2.18 (br s, 1 H), 2.20 (s, 3 H), 2.29 (s, 3 H), 3.16 (d, *J* = 3.3 Hz, 1 H), 3.81 (d, *J* = 13.2 Hz, 1 H), 4.15 (d, *J* = 13.2 Hz, 1 H), 6.92-7.10 (m, 5 H), 7.21-7.38 (m, 6 H), 7.61-7.68 (m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) 18.74, 18.96, 26.11, 26.29, 26.47, 26.71, 30.77, 40.79, 52.36, 69.62, 112.16, 124.48, 125.98, 126.39, 126.85, 128.27, 128.49, 128.65, 128.68, 129.11, 140.39, 141.94, 150.73, 170.45; HRMS (FAB) *m/z*: Calcd for $C_{29}H_{35}N_2Te$ $(M + H)^+$, 541.1862, found 541.1867; Anal. Calcd for $C_{29}H_{34}N_2Te$: C, 64.72; H, 6.37; N, 5.21. Found: C, 64.54; H, 6.48; N, 5.11.

Phenyl 2-(*N*-benzylamino)-3,3-dimethyl-*N*-(2,6-dimethylphenyl)-tellurobutano-imidate. A solution of phenyl triethoxylsilyl telluride (143.6 mg, 0.39 mmol), *N*-(*tert*-butylmethylidene)benzylamine (52.9 mg, 0.30 mmol) and 2,6-dimethylphenylisonitrile (79.1 mg, 0.60 mmol) in CD_3CN (0.6 mL) in a sealed NMR tube was stood at room temperature for 24 h. The crude mixture was purified by flash column chromatography (silica gel: 14.0 g, elution with 3% ethyl acetate in hexane) to give the title compound in 74% yield (114.2 mg) as yellow solid. Mp 53.9-54.3 ^oC; IR (KBr) 2950, 1609, 1588, 1464, 847, 804, 764, 731, 695; ^1H NMR (300 MHz, $CDCl_3$) 1.04 (s, 9 H), 2.27 (s, 6 H), 2.39 (br s, 1 H), 3.26 (s, 1 H), 3.77 (d, *J* = 12.9 Hz, 1 H), 4.26 (d, *J* = 12.9 Hz, 1 H), 6.90-6.99 (m, 2 H), 6.99-7.05 (m, 1 H), 7.13 (t, *J* = 7.5 Hz, 2 H), 7.23-7.42 (m, 6 H), 7.70-7.77 (m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) 18.94, 19.11, 27.28, 35.99, 53.18, 71.43, 114.17, 124.55, 125.75, 126.09, 126.91, 128.00, 128.27, 128.65, 129.17, 140.80, 141.92, 150.59, 170.06; HRMS (FAB) *m/z*: Calcd for $C_{27}H_{33}N_2Te$ $(M + H)^+$, 515.1706, found 515.1699; Anal. Calcd for $C_{27}H_{32}N_2Te$: C, 63.32; H, 6.30; N, 5.47. Found: C, 63.08; H, 6.33; N, 5.33.

Phenyl 2-(*N*-benzylamino)-*N*-(2,6-dimethylphenyl)-telluro-6-heptenoimidate. A solution of phenyl triethoxylsilyl telluride (143.6 mg, 0.39 mmol), *N*-(5-hexenylidene)-benzylamine (56.3 mg, 0.30 mmol) and 2,6-dimethylphenylisonitrile (79.0 mg, 0.60 mmol) in CD_3CN (0.6 mL) in a sealed NMR tube was stood at room temperature for 24 h. The crude mixture was purified by flash column chromatography (silica gel: 14.0 g, elution with 5%

ethyl acetate in hexane) to give the title compound in 67% yield (105.3 mg) as orange solid. Mp 40.1-40.5 ^{o}C; IR (KBr) 2960, 1638, 837, 735, 690; ^{1}H NMR (300 MHz, $CDCl_3$) 0.92-1.08 (m, 1 H), 1.33-1.85 (m, 5 H), 2.17 (s, 3 H), 2.20-2.40 (envelop involving s at 2.28, 4 H), 3.27 (dd, *J* = 9.3, 3.5 Hz, 1 H), 3.81 (d, *J* = 13.2 Hz, 1 H), 4.12 (d, *J* = 13.2 Hz, 1 H), 4.88-4.97 (m, 2 H), 5.62-5.77 (m, 1 H), 6.91-7.11 (m, 5 H), 7.22-7.37 (m, 6 H), 7.63-7.69 (m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) 18.15, 18.64, 25.45, 33.33, 34.53, 52.01, 65.42, 111.72, 114.32, 124.48, 125.98, 126.09, 126.92, 128.19, 128.34, 128.52, 128.56, 128.59, 129.25, 138.63, 140.22, 141.82, 150.35, 170.79; HRMS (FAB) *m/z*: Calcd for $C_{28}H_{33}N_2Te$ $(M + H)^+$, 527.1706, found 527.1702; Anal. Calcd for $C_{28}H_{32}N_2Te$: C, 64.16; H, 6.15; N, 5.34. Found: C, 63.91; H, 6.17; N, 5.07.

Phenyl 2-(*N*-benzylamino)-*N*-(2,6-dimethylphenyl)-telluro-6-heptynoimidate. A solution of phenyl triethoxylsilyl telluride (143.6 mg, 0.39 mmol), *N*-(5-hexynylidene)-benzylamine (55.4 mg, 0.30 mmol) and 2,6-dimethylphenylisonitrile (79.0 mg, 0.60 mmol) in CD_3CN (0.6 mL) in a sealed NMR tube was stood at room temperature for 24 h. The crude mixture was purified by flash column chromatography (silica gel: 14.0 g, elution with 6% ethyl acetate in hexane) to give the title compound in 68% yield (105.8 mg) as orange solid. Mp 60.3-60.8 ^{o}C; IR (KBr) 3332, 2920, 1640, 835, 774, 737; ^{1}H NMR (300 MHz, $CDCl_3$) 1.06-1.27 (m, 1 H), 1.38-1.53 (m, 1 H), 1.60-1.98 (m, 5 H), 2.17 (s, 3 H), 2.29 (s, 3 H), 2.34 (br s, 1 H), 3.22 (dd, *J* = 9.3, 3.2 Hz, 1 H), 3.78 (d, *J* = 12.9 Hz, 1 H), 4.11 (d, *J* = 12.9 Hz, 1 H), 6.92-7.13 (m, 5 H), 7.22-7.36 (m, 6 H), 7.65-7.73 (m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) 17.95, 18.14, 18.64, 25.16, 33.99, 51.95, 64.98, 68.32, 84.32, 111.57, 124.52, 126.03, 126.92, 128.17, 128.32, 128.56, 128.61, 128.70, 129.37, 140.22, 141.86, 150.47, 170.67; HRMS (FAB) *m/z*: Calcd for $C_{28}H_{31}N_2Te$ $(M + H)^+$, 525.1549, found 525.1564; Anal. Calcd for $C_{28}H_{30}N_2Te$: C, 64.41; H, 5.79; N, 5.37. Found: C, 64.31; H, 5.67 N, 5.33.

Phenyl 2-(*N*-benzylamino)-3-benzyloxy-*N*-(2,6-dimethylphenyl)-tellurobutano-imidate. A solution of phenyl triethoxylsilyl telluride (143.6 mg, 0.39 mmol), *N*-(2-benzyloxypropylidene)benzylamine (75.6 mg, 0.30 mmol) and 2,6-dimethylphenylisonitrile (79.4 mg, 0.61 mmol) in CD_3CN (0.6 mL) in a sealed NMR tube was stood at room temperature for 24 h. The crude mixture was purified by flash column chromatography (silica gel: 15.0 g, elution with 5% ethyl acetate in hexane) to give the title compound in 77% yield (135.3 mg) as a 74:26 mixture of diastereomers. The diastereomers were partially separated by

flash column chromatography. **Major isomer**; light yellow oil. IR (neat) 1620, 1590, 1472, 1455, 1109, 1090, 849, 735, 696; ^{1}H NMR (300 MHz, $CDCl_3$) 1.38 (d, *J* = 6.3 Hz, 3 H), 2.16 (s, 3 H), 2.21 (br s, 1 H), 2.29 (s, 3 H), 3.74 (d, *J* = 11.7 Hz, 1 H), 3.77-3.97 (m, 4 H), 4.17 (d, *J* = 13.5 Hz, 1 H), 6.89-7.05 (m, 5 H), 7.12-7.39 (m, 11 H), 7.58-7.66 (m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) 15.01, 18.58, 18.85, 51.93, 66.43, 70.00, 76.00, 112.57, 124.57, 125.83, 125.92, 126.94, 127.15, 128.05, 128.26, 128.37, 128.56, 129.26, 138.60, 140.10, 141.61, 150.43, 167.58; HRMS (FAB) *m/z*: Calcd for $C_{32}H_{35}N_2OTe$ $(M + H)^+$, 593.1812, found 593.1816; Anal. Calcd for $C_{32}H_{34}N_2OTe$: C, 65.12; H, 5.81; N, 4.75. Found: C, 65.06; H, 5.89; N, 4.64. **Minor isomer**; light yellow oil. IR (neat) 1632, 1592, 1472, 1455, 1435, 1123, 1094, 845, 766, 733, 696; ^{1}H NMR (300 MHz, $CDCl_3$) 1.13 (d, *J* = 6.0 Hz, 3 H), 2.14 (s, 3 H), 2.16 (s, 3 H), 2.61 (br s, 1 H), 3.35 (d, *J* = 4.8 Hz, 1 H), 3.85-3.98 (m, 2 H), 4.15 (d, *J* = 13.2 Hz, 1 H), 4.43 (d, *J* = 11.7 Hz, 1 H), 4.63 (d, *J* = 11.4 Hz, 1 H), 6.75 (d, *J* = 7.2 Hz, 1 H), 6.82 (t, *J* = 7.5 Hz, 1 H), 6.90-7.02 (m, 3 H), 7.12-7.19 (m, 1 H), 7.21-7.42 (m, 10 H), 7.48-7.55 (m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) 16.39, 18.47, 18.99, 52.05, 70.65, 70.85, 75.44, 112.57, 124.27, 125.40, 126.04, 126.95, 127.38, 127.47, 128.05, 128.08, 128.21, 128.34, 128.87, 138.46, 140.07, 141.41, 149.49, 166.63; HRMS (FAB) *m/z*: Calcd for $C_{32}H_{35}N_2OTe$ $(M + H)^+$, 593.1812, found 593.1817.

Phenyl *N*-(2,6-dimethylphenyl)-1,2,3,4-tetrahydro-1-isoquinolinetelluroform-imidate. A solution of phenyl triethoxylsilyl telluride (143.6 mg, 0.39 mmol), 3,4-dihydroisoquinoline (39.8 mg, 0.30 mmol) and 2,6-dimethylphenylisonitrile (78.8 mg, 0.60 mmol) in CD_3CN (0.6 mL) in a sealed NMR tube was stood at room temperature for 24 h. The crude mixture was purified by flash column chromatography (silica gel: 13.6 g, elution with 25% ethyl acetate in hexane) to give the title compound in 61% yield (86.3 mg) as light yellow solid. Mp 85.0-85.3 ^{o}C; IR (KBr) 2830, 1667, 1653, 1472, 747, 735; ^{1}H NMR (300 MHz, $CDCl_3$) 1.93 (br s, 1 H), 2.03 (s, 3 H), 2.16 (s, 3 H), 2.66-2.89 (m, 2 H), 3.01-3.12 (m, 1 H), 3.32-3.43 (m, 1 H), 4.95 (s, 1 H), 6.73 (d, *J* = 7.5 Hz, 1 H), 6.81 (t, *J* = 7.4 Hz, 1 H), 6.91 (d, *J* = 7.2 Hz, 1 H), 6.99-7.13 (m, 3 H), 7.15-7.24 (m, 3 H), 7.32-7.39 (m, 1 H), 7.57-7.64 (m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) 18.48, 18.55, 29.39, 40.18, 65.26, 113.17, 124.24, 125.44, 125.68, 125.92, 126.74, 127.86, 127.97, 128.20, 128.94, 129.26, 134.41, 135.61, 141.35, 149.32, 166.99; HRMS (FAB) *m/z*: Calcd for $C_{24}H_{25}N_2Te$ $(M + H)^+$, 471.1080, found 471.1089; Anal. Calcd for $C_{24}H_{24}N_2Te$: C, 61.59; H, 5.17; N, 5.99. Found: C, 61.44; H, 5.31;

N, 5.82.

Phenyl *N*-(2,6-dimethylphenyl)-2-isopropyl-2-pyrrolidinetelluroformimidate. A solution of phenyl triethoxylsilyl telluride (143.6 mg, 0.39 mmol), 2-isopropyl-1-pyrroline (33.5 mg, 0.30 mmol) and 2,6-dimethylphenylisonitrile (79.0 mg, 0.60 mmol) in CD_3CN (0.6 mL) in a sealed NMR tube was stood at room temperature for 24 h. The crude mixture was purified by flash column chromatography (silica gel: 12.8 g, elution with 5% ethyl acetate in hexane) to give the title compound in 50% yield (67.2 mg) as yellow solid. Mp 85.1-85.3 ^oC; IR (KBr) 2955, 2874, 1663, 1647, 735; ^1H NMR (300 MHz, $CDCl_3$) 1.13 (d, J = 6.9 Hz, 1 H), 1.19 (d, J = 6.9 Hz, 1 H), 1.93-2.18 (envelop involving s at 1.90, 7 H), 2.21 (s, 3 H), 2.33-2.52 (m, 2 H), 3.04-3.18 (m, 2 H), 6.31 (d, J = 7.5 Hz, 1 H), 6.46 (t, J = 7.5 Hz, 1 H), 6.66 (d, J = 7.5 Hz, 1 H), 6.74-6.83 (m, 2 H), 6.90-6.98 (m, 1 H), 7.40-7.48 (m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) 18.03, 18.38, 18.89, 19.09, 25.98, 35.25, 35.34, 47.83, 78.26, 117.84, 123.51, 124.02, 125.34, 126.73, 127.29, 127.52, 127.77, 140.00, 145.46, 172.78; HRMS (FAB) *m/z*: Calcd for $C_{22}H_{29}N_2Te$ $(M + H)^+$, 451.1393, found 451.1404.

Phenyl *N*-(2,6-dimethylphenyl)-2-phenyl-2-pyrrolidinetelluroformimidate. A solution of phenyl triethoxylsilyl telluride (143.6 mg, 0.39 mmol), 2-phenyl-1-pyrroline (43.8 mg, 0.30 mmol) and 2,6-dimethylphenylisonitrile (78.6 mg, 0.60 mmol) in CD_3CN (0.6 mL) in a sealed NMR tube was stood at room temperature for 24 h. The crude mixture was purified by flash column chromatography (silica gel: 13.3 g, elution with 15% ethyl acetate in hexane) to give the title compound in 43% yield (62.6 mg) as yellow solid. Mp 129.2-129.5 ^oC; IR (KBr) 3320, 2969, 1667, 1647, 733, 702, 695; ^1H NMR (300 MHz, $CDCl_3$) 1.42 (s, 3 H), 1.93-2.18 (m, 3 H), 2.19 (s, 3 H), 2.42 (br s, 1 H), 3.20-3.35 (m, 3 H), 6.25 (d, J = 7.2 Hz, 1 H), 6.41 (t, J = 7.5 Hz, 1 H), 6.62 (d, J = 7.5 Hz, 1 H), 6.68-6.76 (m, 2 H), 6.86-6.93 (m, 1 H), 7.27-7.43 (m, 5 H), 7.60-7.67 (m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) 17.79, 18.55, 25.33, 38.27, 47.30, 78.32, 115.61, 123.57, 123.87, 125.54, 126.33, 126.91, 127.11, 127.20, 127.24, 127.77, 128.20, 140.13, 143.72, 144.95, 169.77; HRMS (FAB) *m/z*: Calcd for $C_{25}H_{27}N_2Te$ $(M + H)^+$, 485.1236, found 485.1235; Anal. Calcd for $C_{25}H_{26}N_2Te$: C, 62.42; H, 5.24; N, 5.82. Found: C, 62.02; H, 5.47; N, 5.51.

Phenyl 2-(*N*-butylamino)-*N*-(2,6-dimethylphenyl)-2-phenyltelluroacetimidate. A solution of phenyl triethoxylsilyl telluride (143.6 mg, 0.39 mmol), *N*-benzylidenebutylamine (48.4 mg, 0.30 mmol) and 2,6-dimethylphenylisonitrile (78.9 mg, 0.60 mmol) in CD_3CN (0.6

mL) in a sealed NMR tube was heated at 80 ^{o}C for 2 h. The crude mixture was purified by flash column chromatography (silica gel: 13.5 g, elution with 10% ethyl acetate in hexane) to give the title compound in 94% yield (141.2 mg) as yellow oil. IR (neat) 2957, 2928, 1622, 1590, 1472, 1435, 766, 733, 693; ^{1}H NMR (300 MHz, $CDCl_3$) 0.91 (t, *J* = 7.4 Hz, 3 H), 1.29-1.56 (m, 4 H), 2.03 (s, 3 H), 2.07 (s, 3 H), 2.21 (br s, 1 H), 2.63 (dt, *J* = 11.4, 7.0 Hz, 1 H), 2.89 (dt, *J* = 11.4, 7.3 Hz, 1 H), 4.52 (s, 1 H), 6.83-6.95 (m, 3 H), 7.12-7.36 (m, 8 H), 7.65-7.72 (m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) 13.99, 18.24, 18.32, 20.40, 32.33, 48.36, 70.70, 112.76, 124.30, 125.84, 126.06, 127.39, 127.79, 128.17, 128.21, 128.30, 128.52, 129.06, 139.36, 141.70, 149.80, 167.86; HRMS (FAB) *m/z*: Calcd for $C_{26}H_{31}N_2Te$ $(M + H)^+$, 501.1557, found 501.1560.

Phenyl 2-[*N*-(2-methoxyethyl)amino]-*N*-(2,6-dimethylphenyl)-2-phenyltelluro-acetimidate. A solution of phenyl triethoxylsilyl telluride (143.6 mg, 0.39 mmol), *N*-benzylidene-2-methoxyethylamine (49.2 mg, 0.30 mmol) and 2,6-dimethylphenylisonitrile (79.2 mg, 0.60 mmol) in CD_3CN (0.6 mL) in a sealed NMR tube was heated at 80 ^{o}C for 2 h. The crude mixture was purified by flash column chromatography (silica gel: 13.6 g, elution with 24% ethyl acetate in hexane) to give the title compound in 78% yield (117.0 mg) as light orange oil. IR (neat) 1624, 1590, 1472, 1455, 1435, 1123, 1092, 768, 735, 695; ^{1}H NMR (300 MHz, $CDCl_3$) 2.03 (s, 3 H), 2.08 (s, 3 H), 2.30 (br s, 1 H), 2.78 (dt, *J* = 12.3, 5.3 Hz, 1 H), 2.89 (dt, *J* = 12.9, 5.4 Hz, 1 H), 3.33 (s, 3 H), 3.50 (t, *J* = 5.1 Hz, 2 H), 4.56 (s, 1 H), 6.83-6.95 (m, 3 H), 7.13-7.36 (m, 8 H), 7.65-7.73 (m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) 18.32, 18.48, 47.95, 58.81, 70.85, 72.19, 112.68, 124.39, 125.92, 126.19, 127.53, 127.97, 128.24, 128.32, 128.62, 129.15, 139.07, 141.82, 149.90, 167.31; HRMS (FAB) *m/z*: Calcd for $C_{25}H_{29}N_2OTe$ $(M + H)^+$, 503.1342, found 503.1330.

Phenyl 2-(*N*-allylamino)-*N*-(2,6-dimethylphenyl)-2-phenyltelluroacetimidate. A solution of phenyl triethoxylsilyl telluride (143.6 mg, 0.39 mmol), *N*-benzylideneallylamine (43.2 mg, 0.30 mmol) and 2,6-dimethylphenylisonitrile (78.4 mg, 0.60 mmol) in CD_3CN (0.6 mL) in a sealed NMR tube was heated at 80 ^{o}C for 2 h. The crude mixture was purified by flash column chromatography (silica gel: 13.3 g, elution with 12% ethyl acetate in hexane) to give the title compound in 95% yield (136.0 mg) as yellow oil. IR (neat) 1622, 1590, 1472, 1455, 1435, 766, 735, 698; ^{1}H NMR (300 MHz, $CDCl_3$) 2.04 (s, 3 H), 2.13 (s, 3 H), 2.45 (br s, 1 H), 3.31 (dd, *J* = 13.8, 6.2 Hz, 1 H), 3.45 (dd, *J* = 14.1, 5.9 Hz, 1 H), 4.57 (s, 1 H), 5.05-

5.23 (m, 2 H), 5.79-5.95 (m, 1 H), 6.84-6.96 (m, 3 H), 7.10-7.36 (m, 8 H), 7.63-7.71 (m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) 18.33, 18.47, 50.76, 69.71, 112.50, 116.34, 124.45, 126.00, 126.07, 127.48, 127.96, 128.23, 128.35, 128.41, 128.67, 129.15, 136.43, 139.08, 141.89, 150.03, 167.23; HRMS (FAB) *m/z*: Calcd for $C_{26}H_{31}N_2Te$ $(M + H)^+$, 485.1244, found 485.1248.

Transformations of the coupling products.

2-(Acetyl-benzyl-amino)-N-(2,6-dimethyl-phenyl)-3,3-dimethyl-butyramide (8). To a solution of 2-(*N*-benzylamino)-3,3-dimethyl- *N*-(2,6-dimethylphenyl)-tellurobutanoimidate (**5b**, 81.6 mg, 0.16 mmol) and BSA (60 μL, 0.24 mmol) in H_2O/THF (a 1/9 mixture, 0.4 mL) was added $Hg(OAc)_2$ (51.9 mg, 0.16 mmol) at 0 ^oC, and the resulting mixture was stirred for 15 min. Aqueous saturated sodium thiosulfate and ether were added, and the water layer was extracted with ether. The combined organic extracts were washed with aqueous saturated sodium bicarbonate and saturated brine, and were dried over anhydrous sodium suflate. After solvent was removed, purification of the crude mixture by flush column chromatography (silica gel 8.7g, elution with 20% ethyl acetate in hexane) gave **8** in 94% yield (56.1 mg, 0.15 mol). IR (KBr) 3254, 1686, 1624, 1525, 1470, 1368, 1163, 770, 729, 694; ^{1}H NMR (300 MHz, $CDCl_3$) 1.16 (s, 9 H), 2.13 (br s, 3 H), 2.19 (s, 6 H), 4.99 (br s, 2 H), 5.45 (br s, 1 H), 7.00-7.10 (m, 3 H), 7.17-7.37 (m, 4 H), 7.85 (br s, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) 18.74 (CH_3 x 3), 22.63 (CH_3), 27.70 (CH_3 x 2), 35.40 (C), 49.96 (CH_2), 62.67 (CH_2), 126.00 (CH), 127.12 (CH), 128.18 (CH x 4), 128.67 (CH x 2), 133.62 (C), 134.89 (C x 2), 138.05 (C), 168.24 (C=O), 174.67 (C=O); HRMS (FAB) *m/z*: Calcd for $C_{23}H_{31}N_2O_2$ $(M + H)^+$, 367.2386, found 367.2389.

***N*-(2-(*N*-benzylamino)-3,3-dimethylbutylidene)-2,6-dimethylaniline (9). Tributyltin hydride reduction of 5b**. A solution of **5b** (510.2 mg, 1.00 mmol), tributyltin hydride (0.32 mL, 1.20 mmol), 2,2'-azobisisobutyronitrile (16.0 mg, 0.10 mmol) in benzene (4.0 mL) was heated at 80 ^oC for 2 h. After the solvent was removed under reduced pressure, the crude product was purified by flash chromatography (silica gel 24.6 g; elution with 6 % ethyl acetate in hexane) to give **9** in 70% yield (216.5 mg) as light yellow oil. IR (KBr) 2957, 1653, 1471, 766, 735, 698; ^{1}H NMR (300 MHz, $CDCl_3$) 1.04 (s, 9 H), 2.13 (br s, 1 H), 2.14 (s, 6 H), 3.04 (d, J = 4.5 Hz, 1 H), 3.79 (d, J = 13.2 Hz, 1 H), 4.02 (d, J = 13.2 Hz, 1 H), 6.88-6.98 (m, 1 H), 7.00-7.09 (m, 2 H), 7.19-7.36 (m, 3 H), 7.36-7.44 (m, 2 H), 7.70 (d, J = 4.5 Hz,

1 H); ^{13}C NMR (75 MHz, $CDCl_3$) 18.89, 26.90, 35.14, 53.22, 70.05, 123.51, 126.71, 126.85, 128.09, 128.21, 128.32, 140.65, 151.03, 168.49; HRMS (FAB) *m/z*: Calcd for $C_{21}H_{29}N_2$ $(M + H)^+$, 309.2331, found 309.2337.

Transmetallation of 5b by butyl lithium. To a solution of **5b** (106.7 mg, 0.21 mmol) in THF (0.4 mL) was added a hexane solution of butyl lithium (0.33 mL, 0.48 mmol) at –72 ^{o}C, and the resulting solution was stirred for 10 min at this temperature. Aqueous saturated ammonium chloride was added, and the organic phase was extracted by ethyl acetate. The combined organic extracts were washed with aqueous saturated sodium bicarbonate, saturated brine, and were dried over anhydrous sodium sulfate. After removal of solvent, the crude product was purified by flash chromatography to give **9** in 74% yield (47.6 mg, 0.15 mmol).

2-(*N*-benzylamino)-3,3-dimethylbutyraldehyde (10). A solution of *N*-(2-(*N*-benzylamino)-3,3-dimethylbutylidene)-2,6-dimethylaniline (95.6 mg, 0.31 mmol), 2 N aqueous hydrogen chloride (0.45 mL, 0.90 mmol) in THF and ethanol (1:1 mixture, 3.0 mL) was stirred at room temperature for 1 h. After the solvent was removed under reduced pressure, ethyl acetate and aqueous saturated sodium bicarbonate were added to the crude mixture. The water layer was extracted three times with ethyl acetate, and the combined organic extracts were washed with saturated brine, dried over Na_2SO_4 and concentrated under reduced pressure. The crude product was purified by flash chromatography (silica gel 3.0 g; elution with 6 % ethyl acetate in hexane) to give **10** in 74% yield (46.8 mg) as light yellow oil. IR (KBr) 2959, 2870, 1721, 1474, 1455, 1368, 737, 698; ^{1}H NMR (300 MHz, $CDCl_3$) 1.02 (s, 9 H), 1.95 (br s, 1 H), 2.85 (d, J = 2.4 Hz, 1 H), 3.60 (d, J = 13.2 Hz, 1 H), 3.81 (d, J = 13.2 Hz, 1 H), 7.20-7.40 (m, 5 H), 9.81 (d, J = 2.7 Hz, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) 26.87, 34.99, 52.98, 74.63, 127.04, 128.20, 128.30, 140.03, 205.48; HRMS (FAB) *m/z*: Calcd for $C_{13}H_{19}NO$ $(M + H)^+$, 206.1545, found 205.1546.

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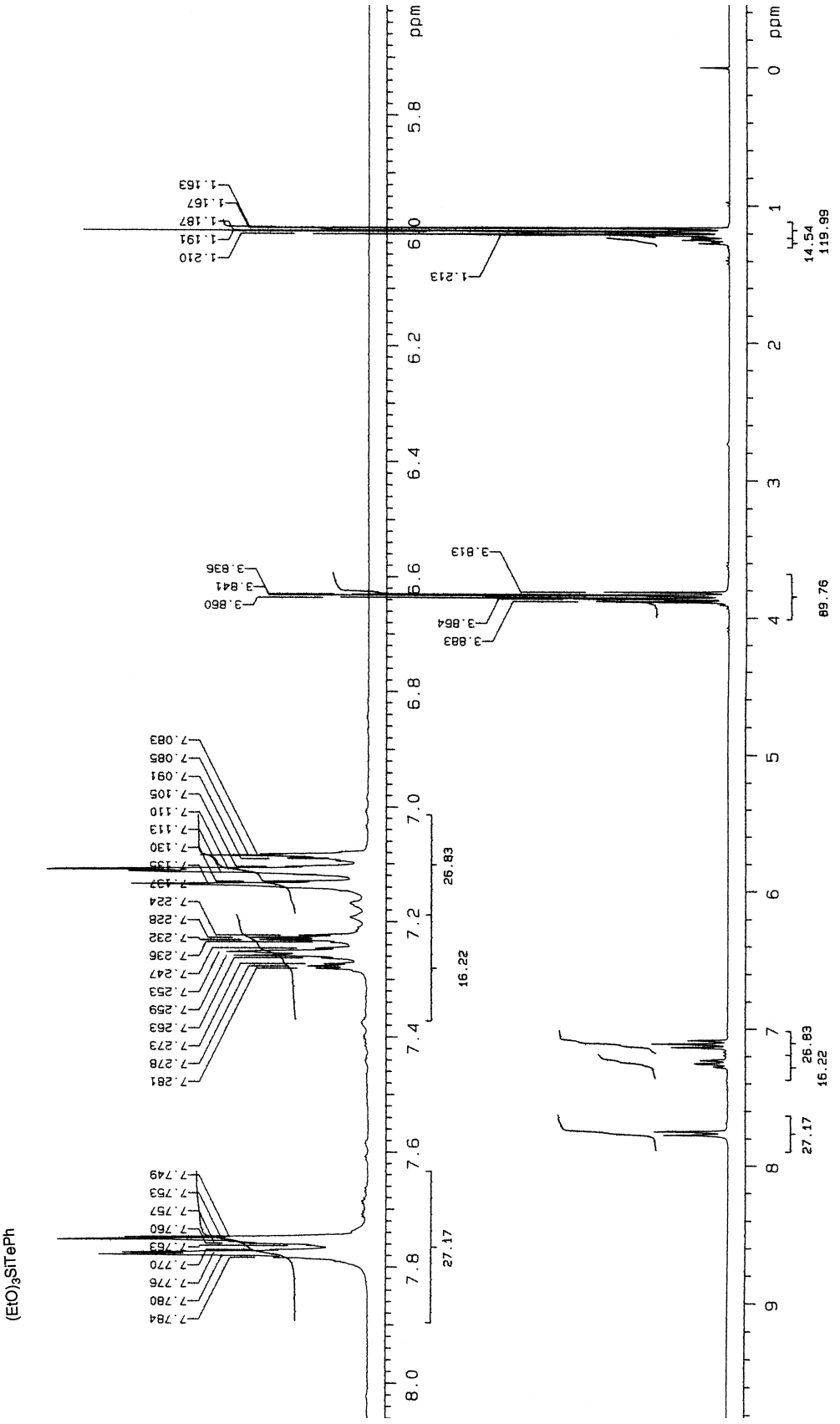
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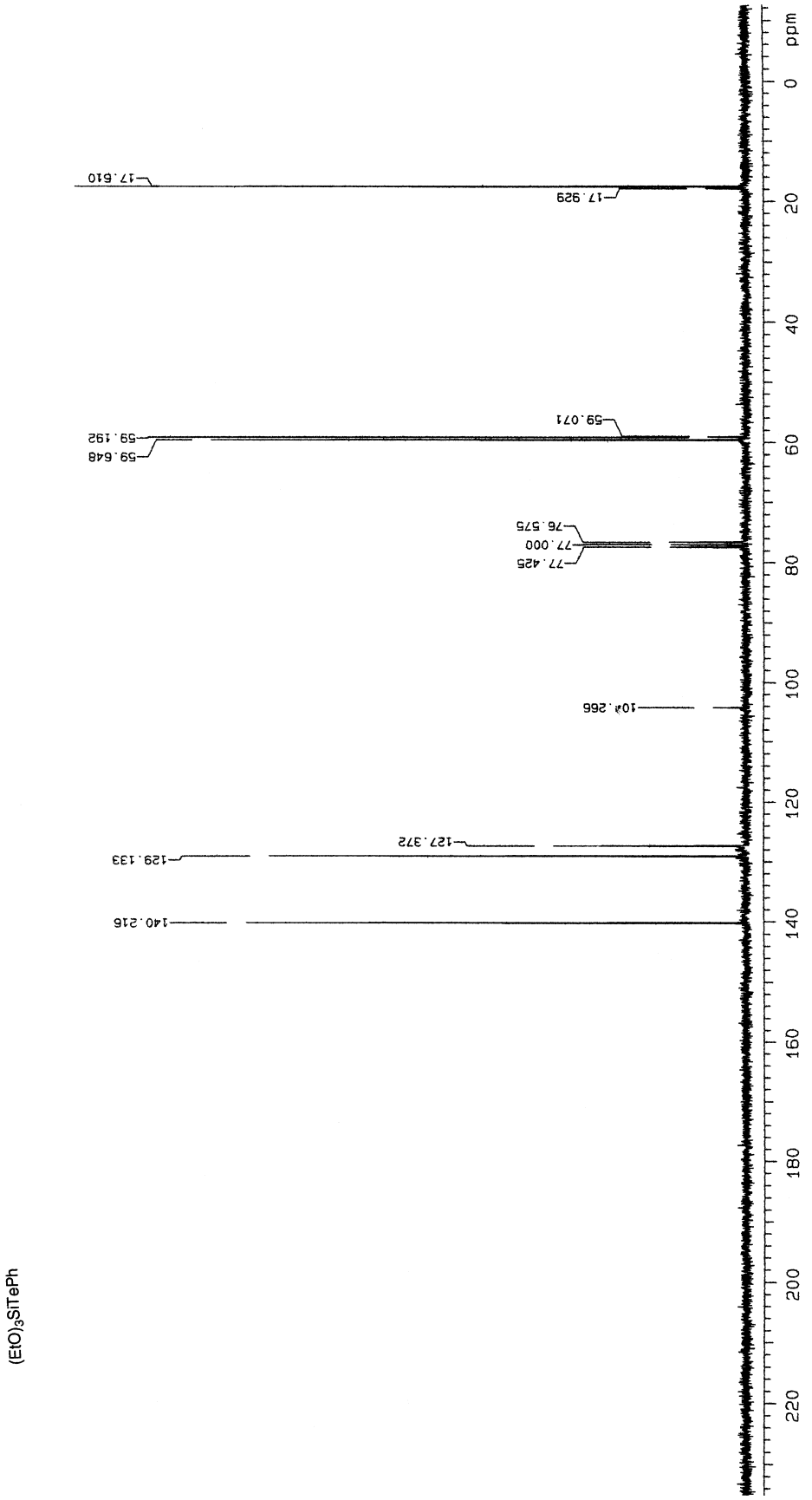
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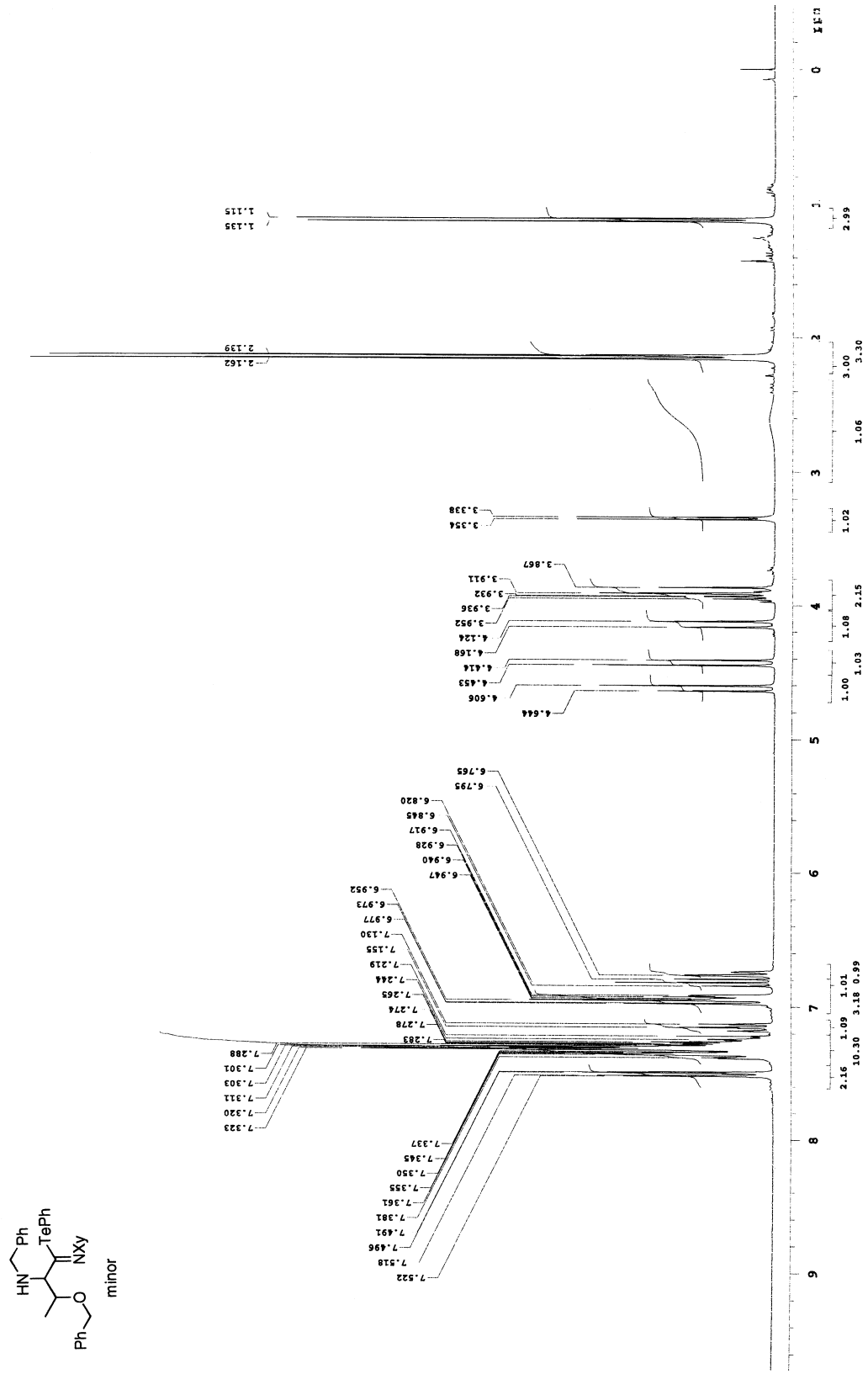
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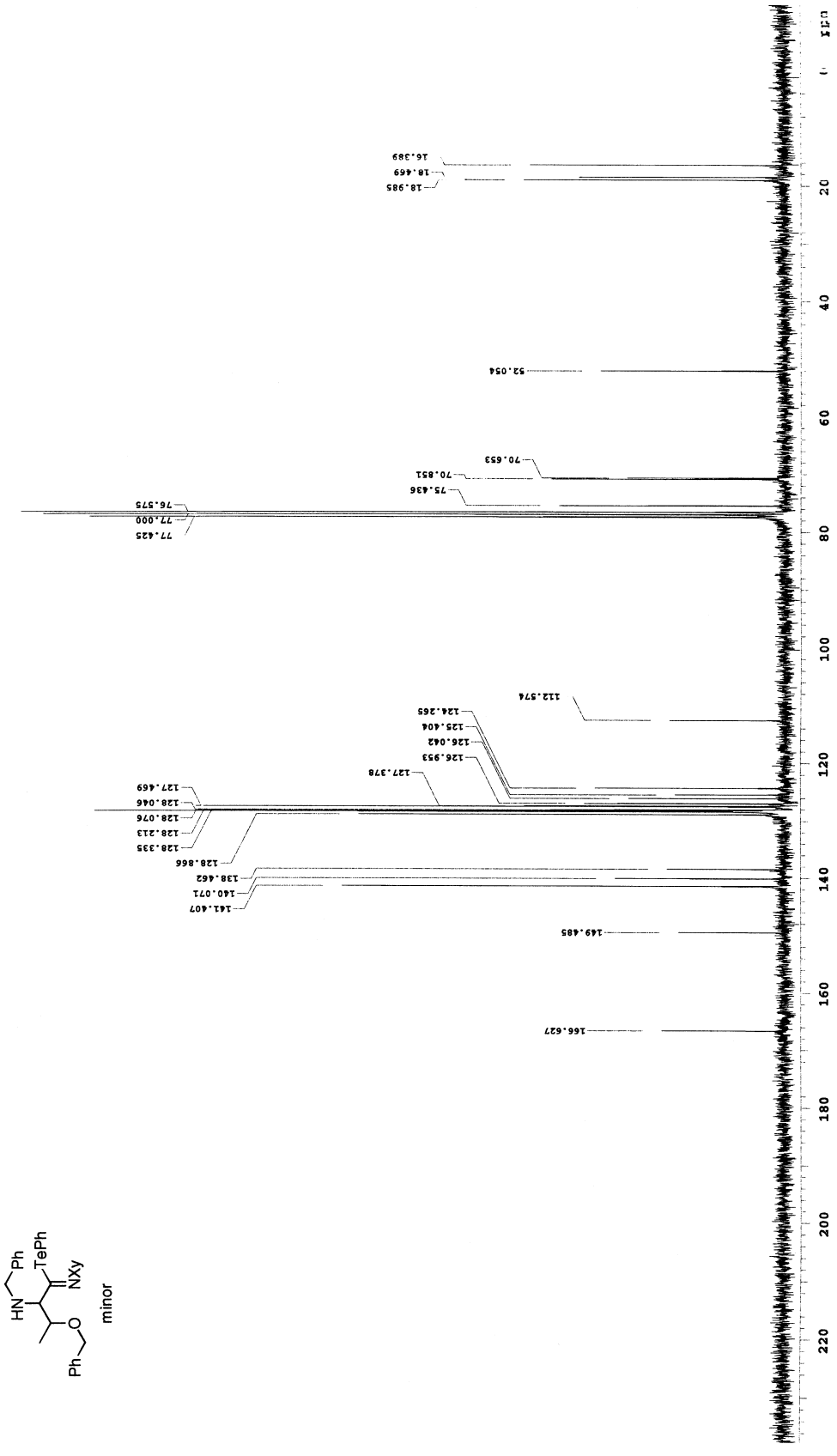
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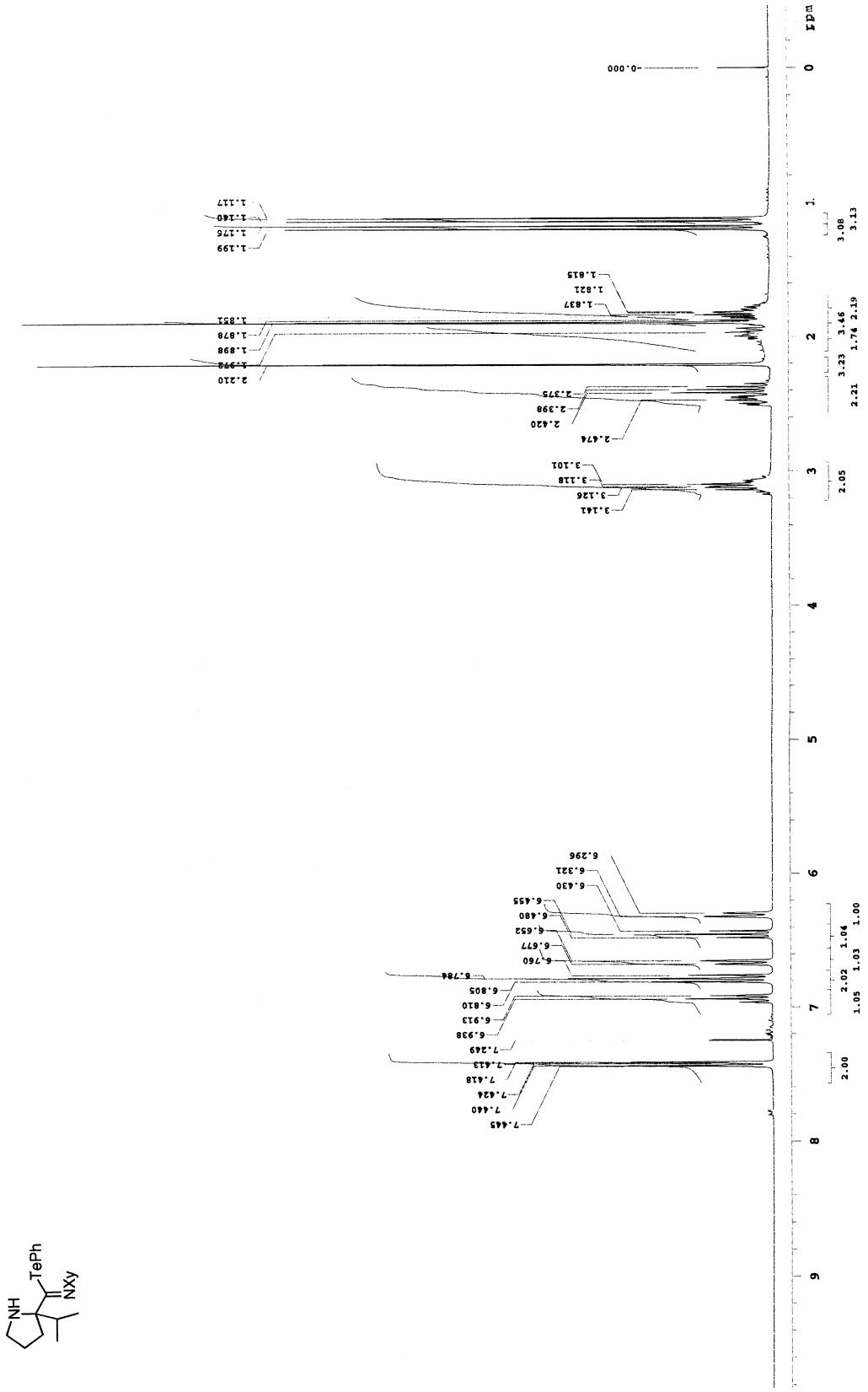
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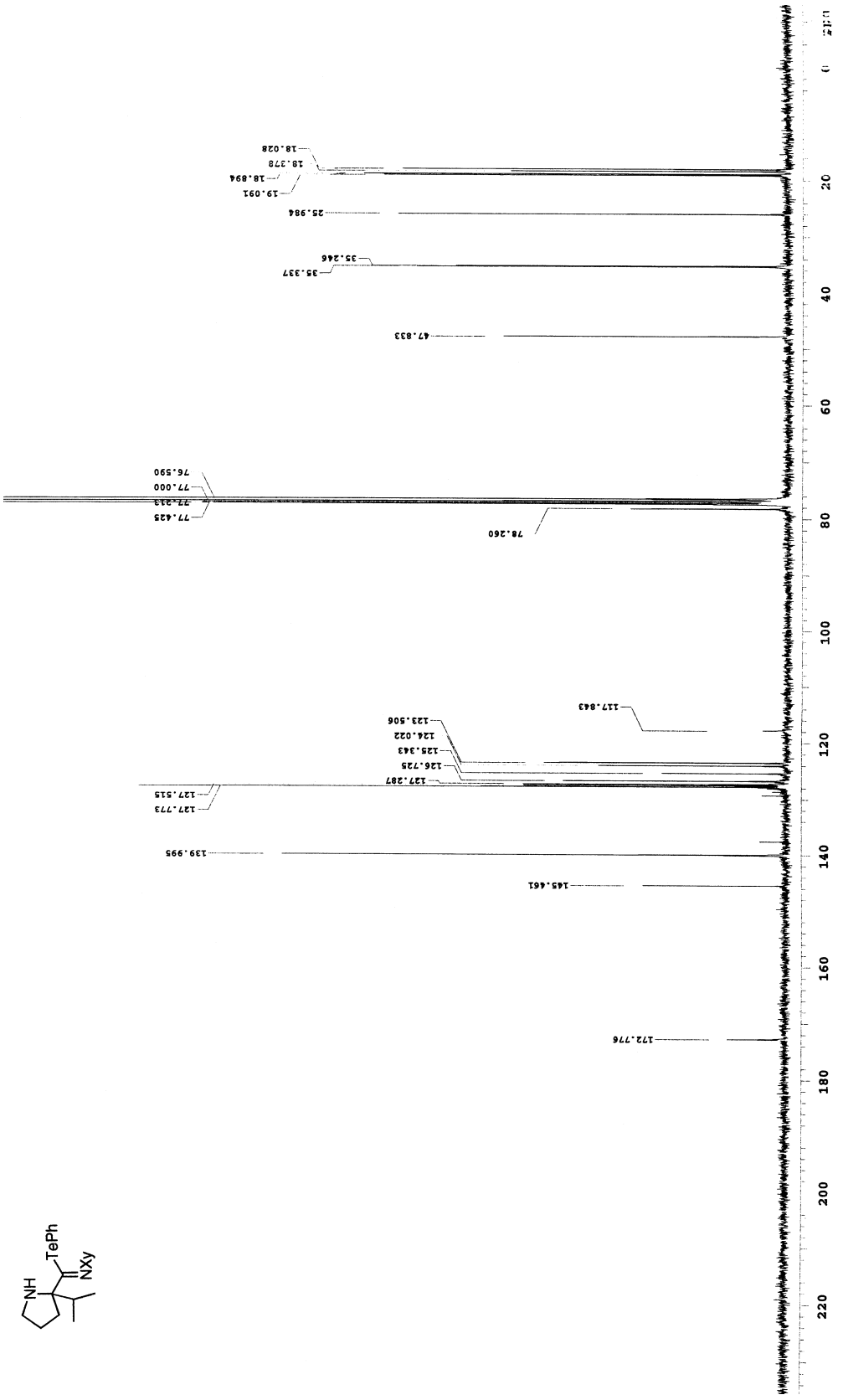
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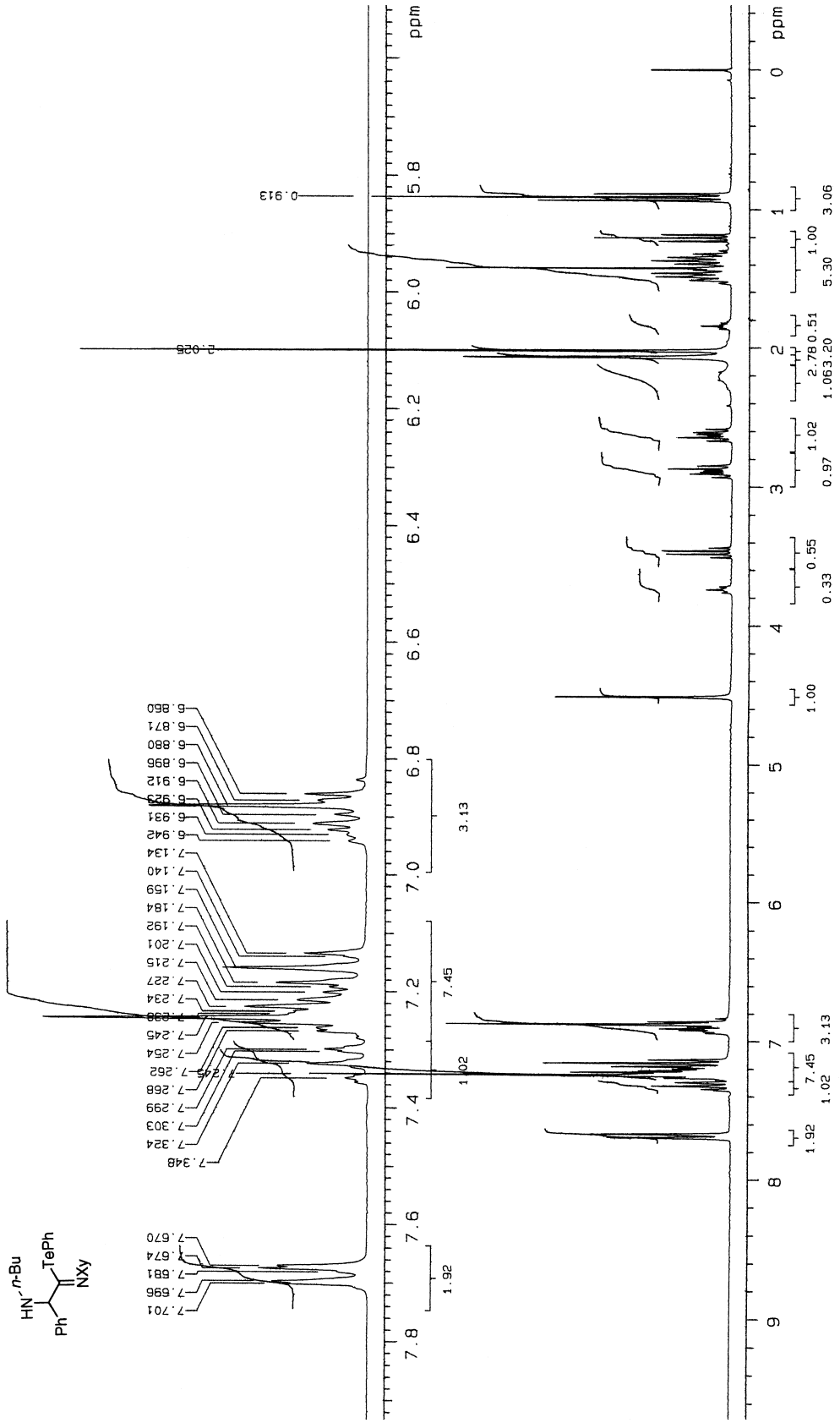
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7.245
7.238
7.234
7.227
7.215
7.201
7.192
7.184
7.159
7.140
7.134
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6.925
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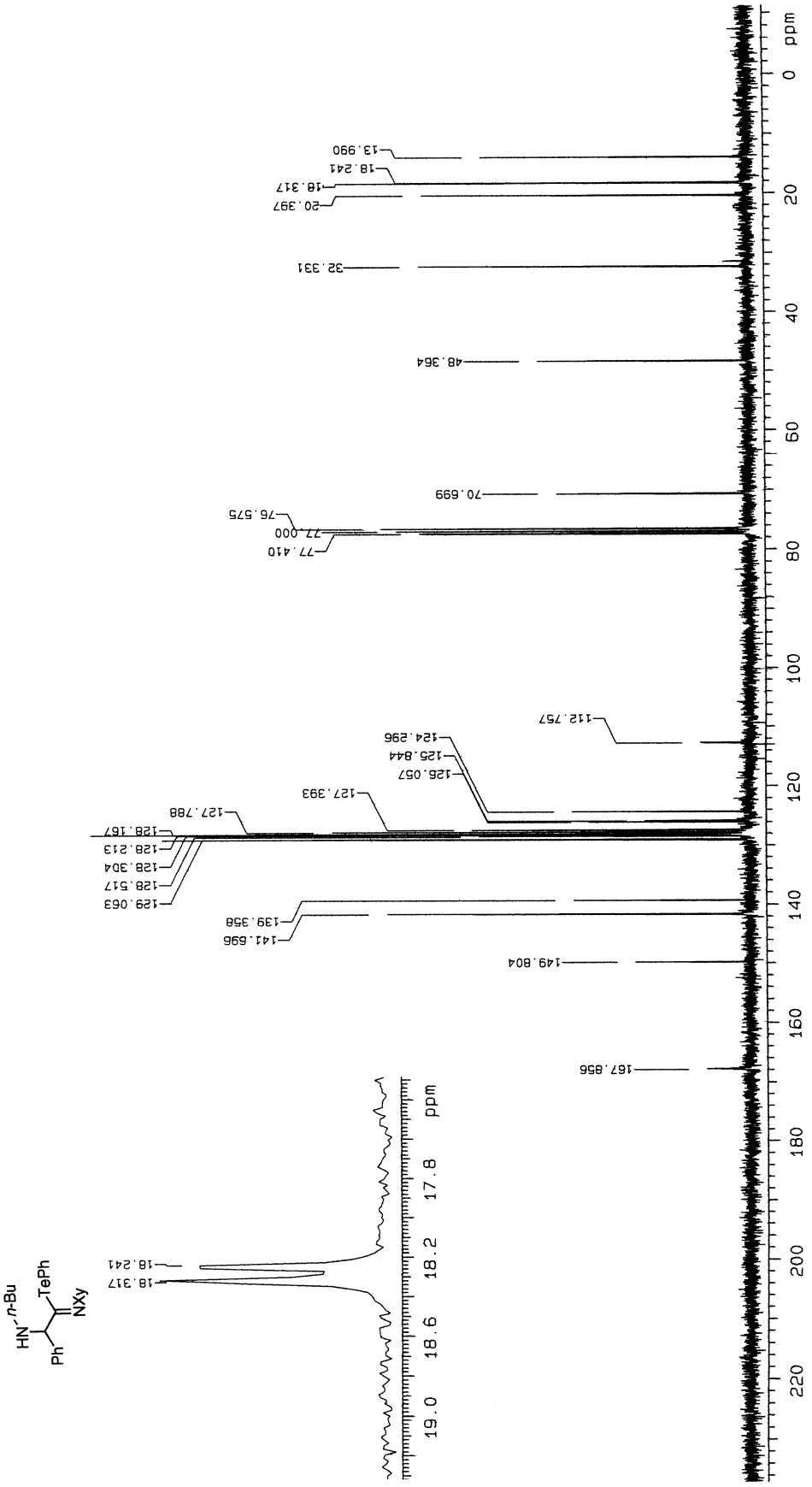
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Ph
TePh
NXy
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139.358
129.063
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128.304
128.213
128.167
127.788
127.393
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125.844
124.296
112.757
77.410
77.000
76.575
70.699
48.364
32.331
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80
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200
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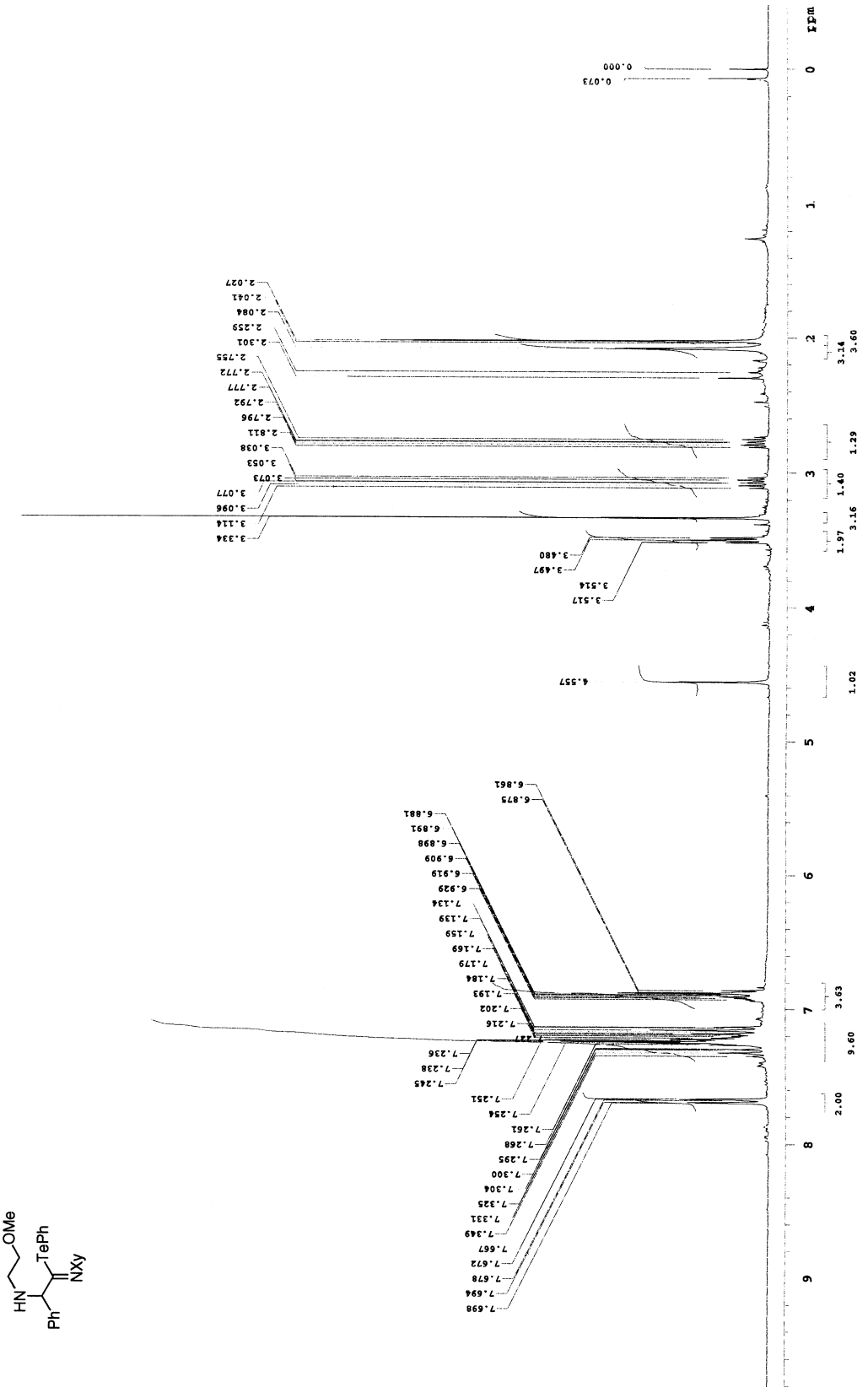
OMe
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HN
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2.772
2.777
2.792
2.796
2.811
3.038
3.053
3.073
3.077
3.096
3.114
3.334
3.480
3.497
3.514
3.517
4.557
6.861
6.875
6.882
6.892
6.898
6.909
6.919
6.929
7.134
7.139
7.159
7.169
7.179
7.184
7.193
7.202
7.216
7.236
7.238
7.245
7.251
7.254
7.261
7.268
7.295
7.300
7.304
7.325
7.331
7.349
7.667
7.672
7.678
7.694
7.698
ppm
0
1
2
3
4
5
6
7
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3.60
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3.16
1.97
1.02
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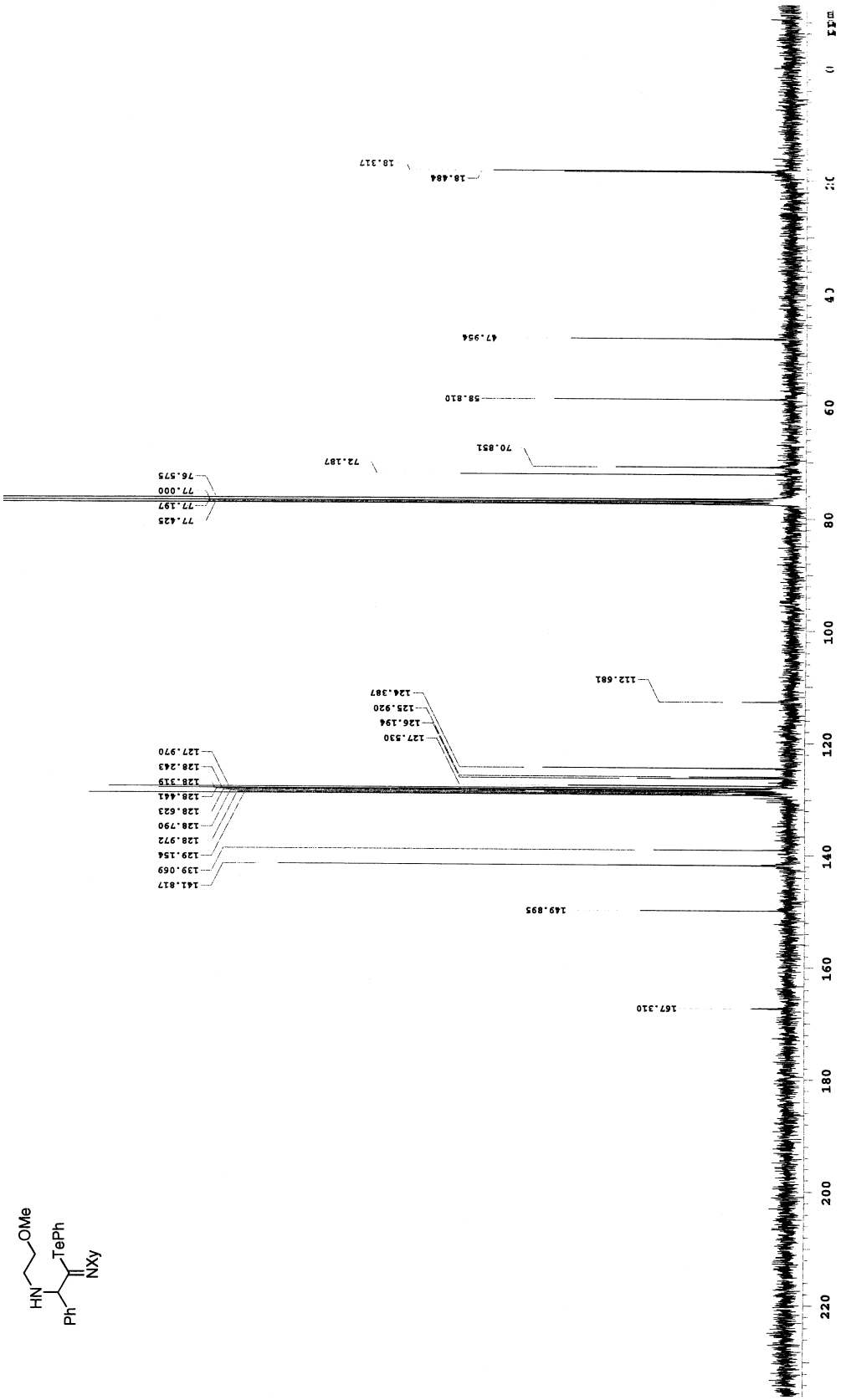
2.00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HN
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77.425
112.681
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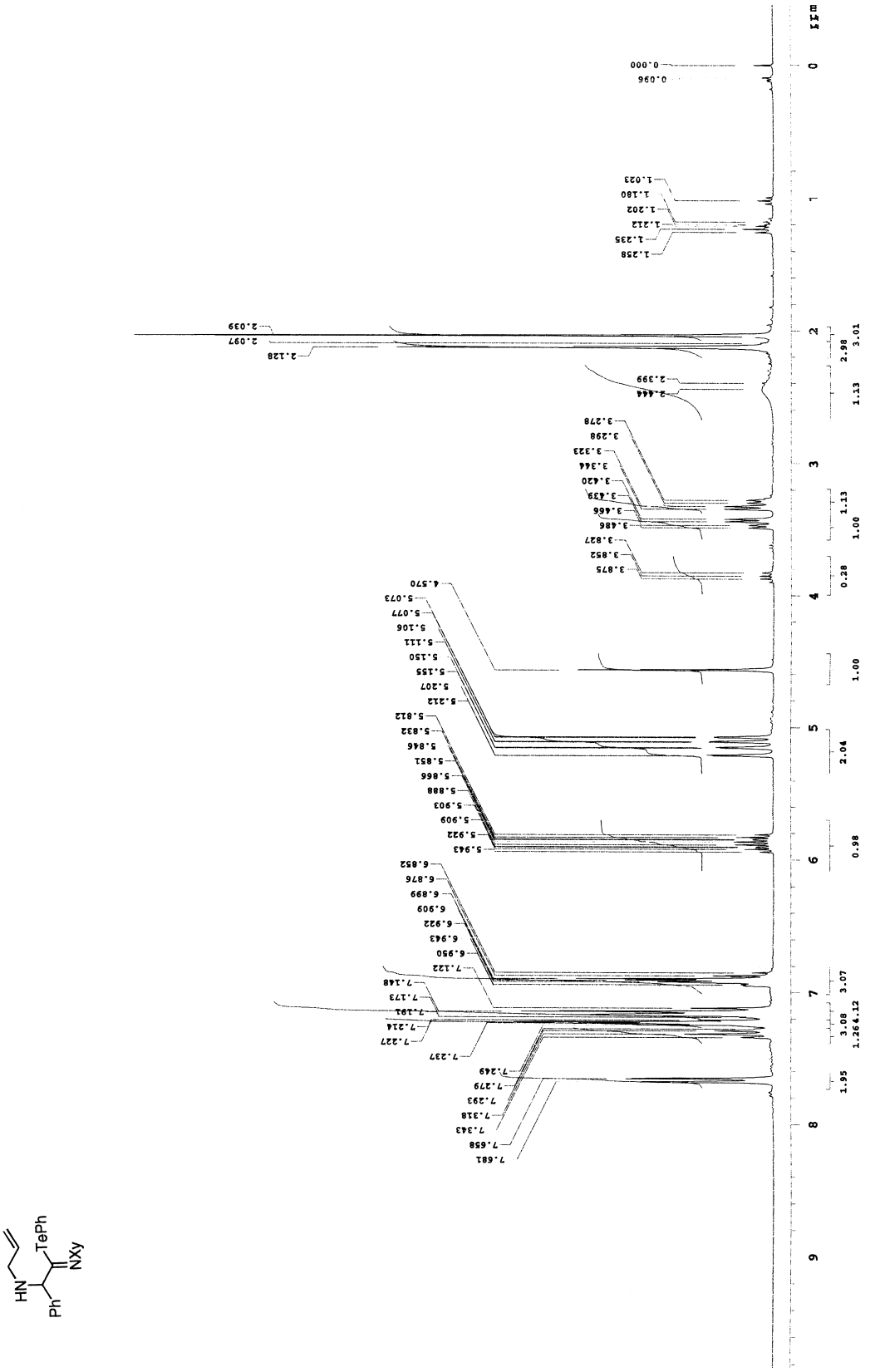
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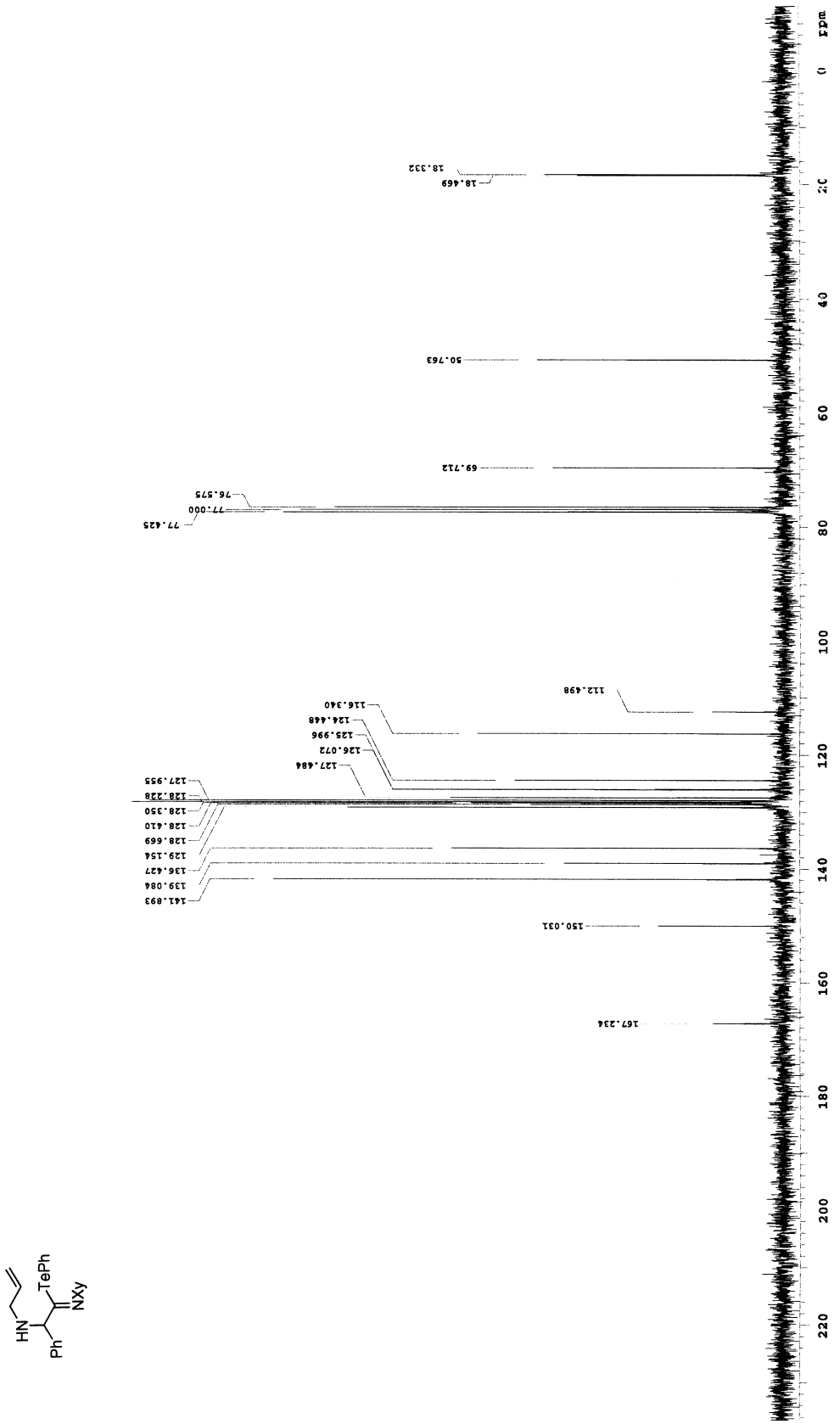
NXy
HN
Ph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128.328
127.955
127.484
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116.340
112.498
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69.712
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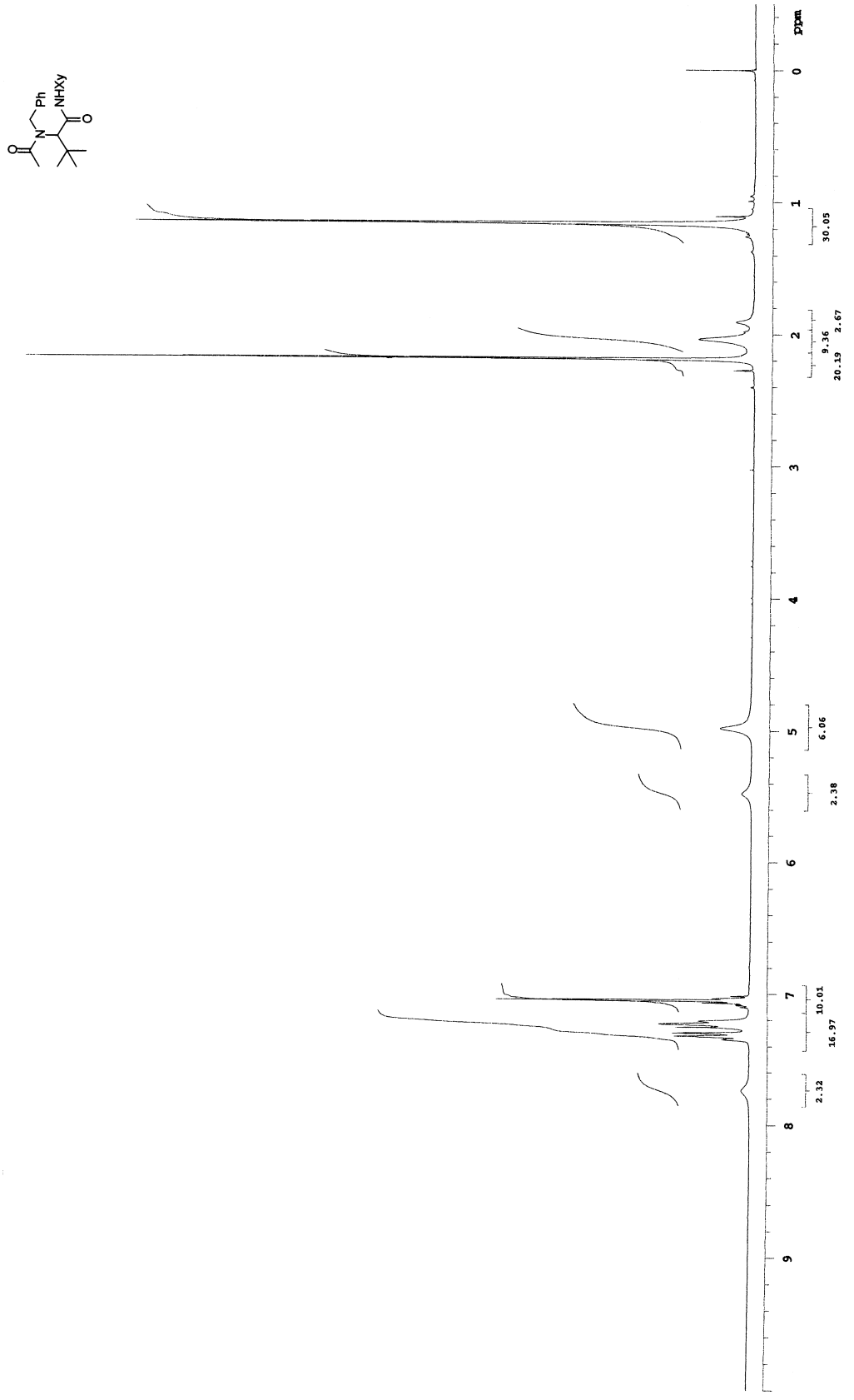
ppm


Ph
NHXy
O
O
N
ppm
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1
2
3
4
5
6
7
8
9
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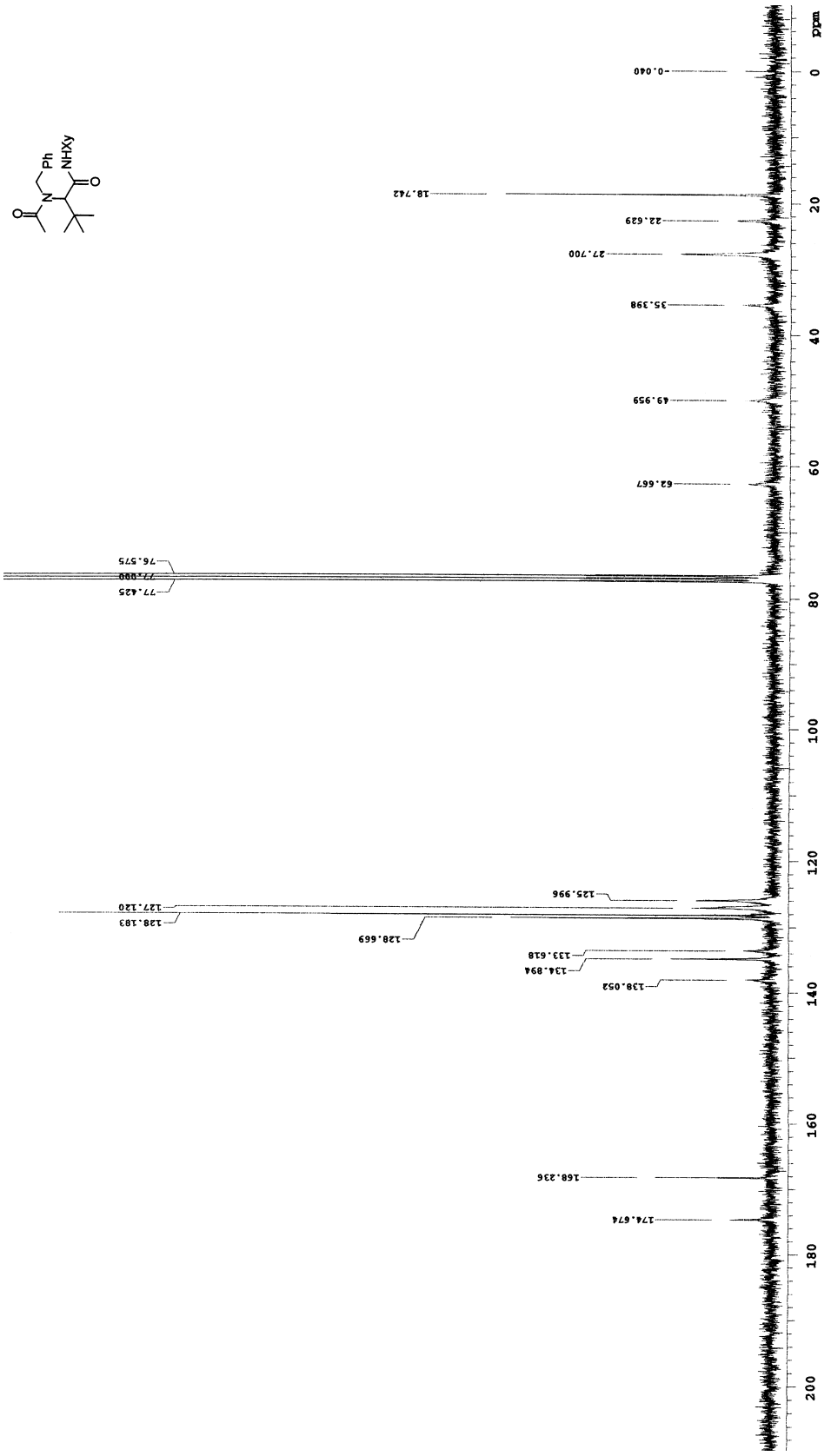
9.36
20.19
2.67
6.06
2.38
10.01
16.97
2.32


Ph
NHXy
O
N
O
ppm
0
20
40
60
80
100
120
140
160
180
200
-0.040
18.742
22.629
27.700
35.398
49.959
62.667
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77.000
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125.996
127.120
128.183
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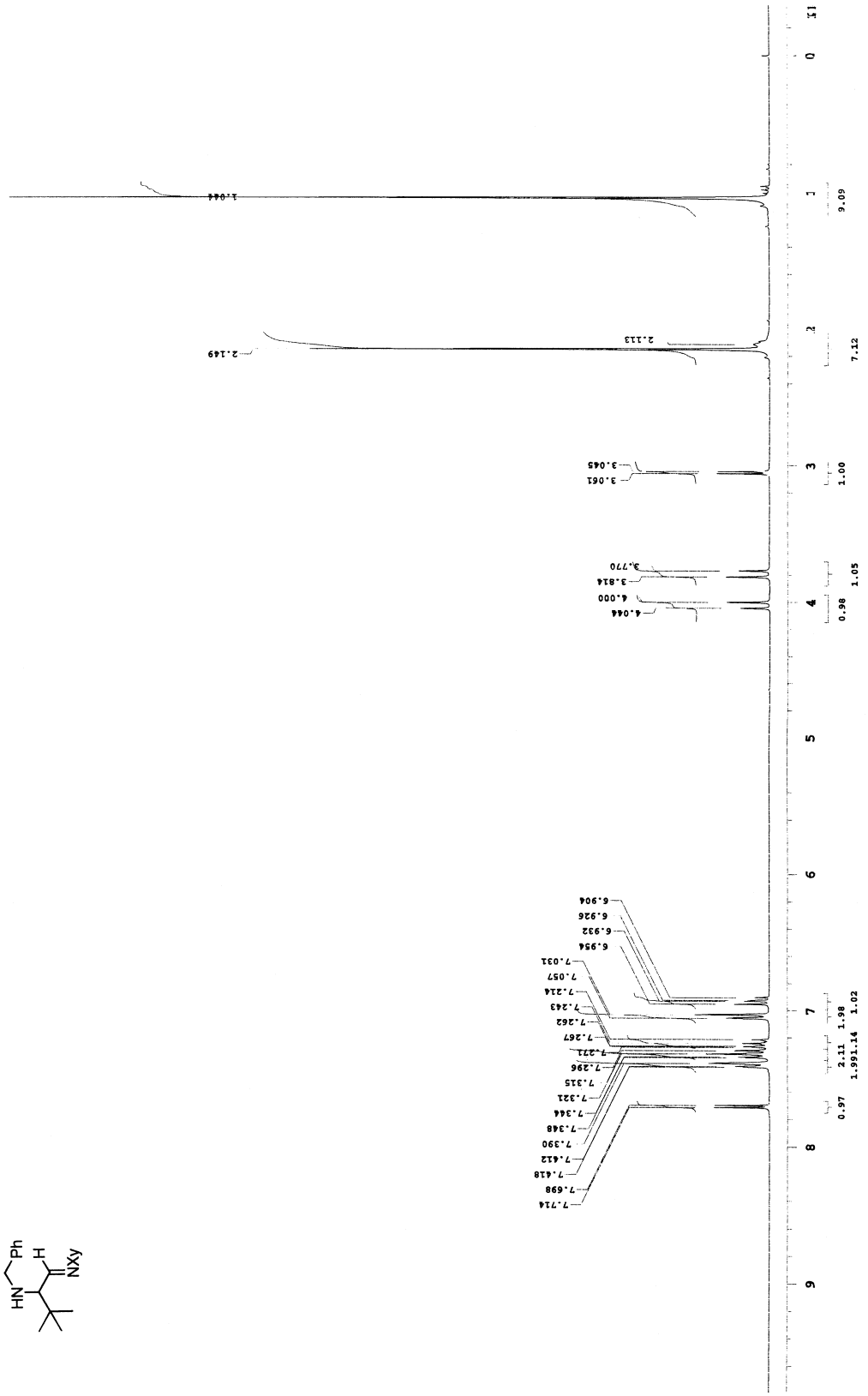
133.618
134.894
138.052
168.336
174.674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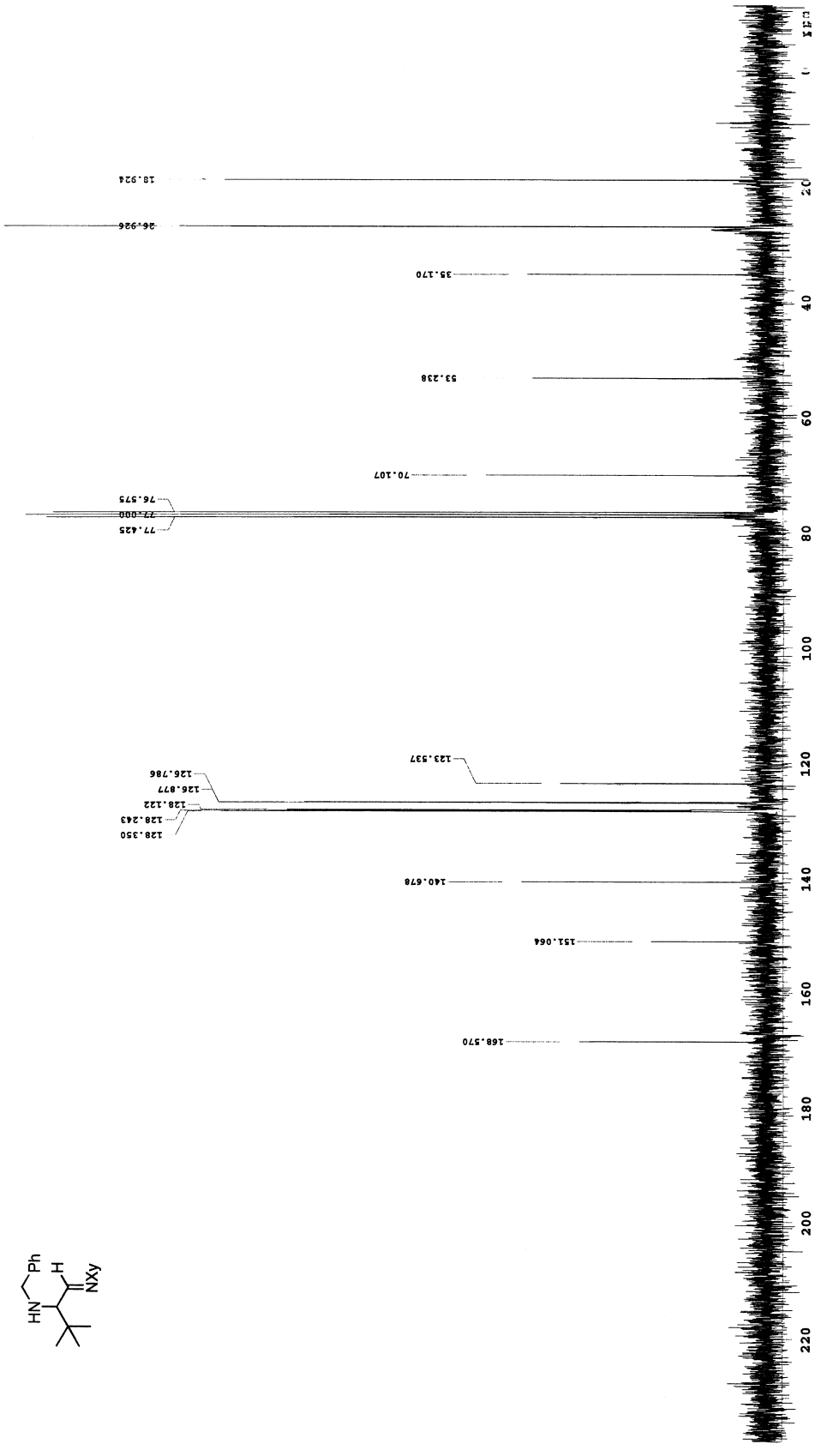
2.113
2.149
3.045
3.061
3.770
3.814
4.000
4.044
6.904
6.926
6.932
6.954
7.031
7.057
7.314
7.343
7.362
7.367
7.371
7.396
7.415
7.321
7.344
7.348
7.390
7.412
7.418
7.698
7.714
9.09
7.12
1.00
1.05
0.98
1.02
1.98
2.11
1.99
1.14
0.97
Ph
H
NXy
HN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140.678
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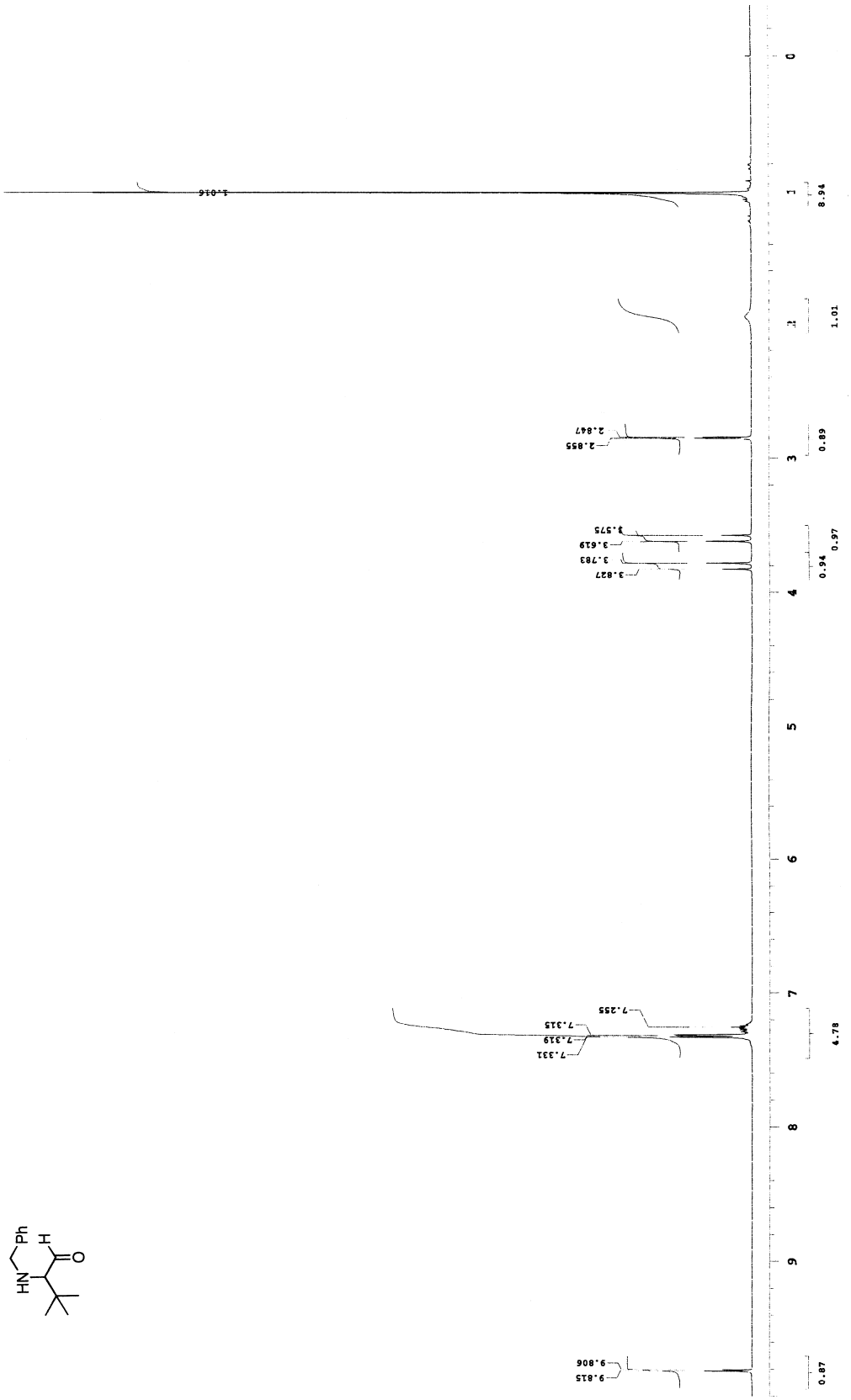
126.786
123.537
77.425
77.000
76.575
70.107
53.238
35.170
26.926
18.924
Ph
H
NXy
HN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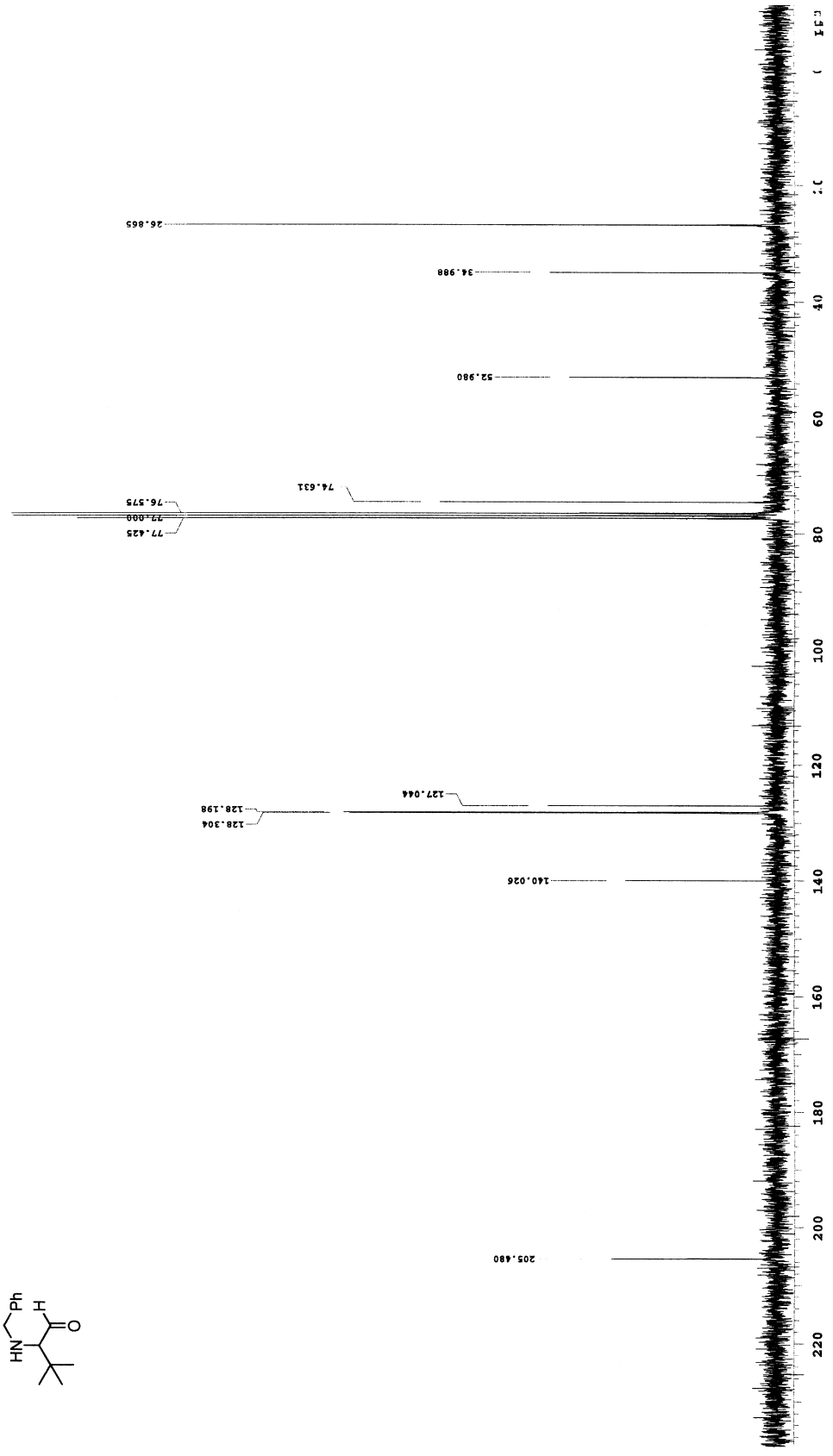
2.847
2.855
3.575
3.619
3.783
3.827
7.255
7.313
7.319
7.331
9.806
9.815
0
1
2
3
4
5
6
7
8
9
8.94
1.01
0.89
0.94
0.97
4.78
0.87
Ph
H
O
HN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127.044
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77.000
76.575
74.631
52.980
34.988
26.865
220
200
180
160
140
120
100
80
60
40
Ph
H
O
HN