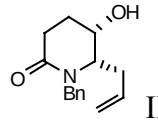
SUPPORTING INFORMATION

Asymmetric synthesis of (+)-isofebrifugine and (–)-sedacryptine from a common chiral non-racemic building block.

Andrew G. H. Wee* and Gao-Jun Fan
Department of Chemistry and Biochemistry
University of Regina, Regina
Saskatchewan, Canada, S4S 0A2

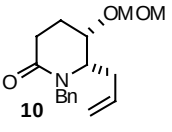
Preparation of compound **10**

To an suspension of $MePh_3P^+ Br^-$ (0.80 g, 2.24 mmol) in THF (8 mL) at 0 °C was added *n*-BuLi (1.5 M in hexane, 1.15 mL, 1.77 mmol) dropwise. After additon was complete, an orange solution was obtained. The reacton mixture was stirred at that 0 °C for 10 min, and then lactol **9** (219 mg, 0.88 mmol) in THF (2 mL) was added via cannula. The mixture was stirred at 0 °C for 30 min and then at rt for 3 h. Saturated aqueous NH_4Cl solution (6 mL) was then added and the the organic layer was separated. The aqueous layer was re-extracted with EtOAc (x4). The combined organic layers were washed with brine, dried (Na_2SO_4), filtered and evaporated under reduced pressure. The residue was purified by flash chromatography (1:1, then 2:3 v/v pet. ether:EtOAc and finally EtOAc) to give recovered lactol **9** (20 mg), and the target alkene alcohol (145 mg, 74%).



IR ν_{max}: 3162–3587, 1613 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz), δ: 7.10–7.40 (m, 5H), 5.70–5.82 (m, 1H), 5.30 (d, 1H, J = 15.1 Hz), 5.07 (dd, 1H, J = 17.3, 1.4 Hz), 5.02 (d, 1H, J = 10.4 Hz), 3.80–3.92 (m, 2H), 3.31 (dd, 1H, J =10.9, 5.9), 2.25–2.65 (m, 4H), 1.75–1.95 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz), δ:169.8, 137.1, 135.5, 128.6, 127.8, 127.3, 117.8, 67.0, 59.1, 48.3, 33.6, 28.5, 25.4; EI-HRMS calcd for $C_{15}H_{20}NO_2$ (MH^+) 246.1494, found 246.1495

A solution of the above alkene alcohol (130 mg, 0.53 mmol), MOM-Cl (80 μL, 1.06 mmol), iPr_2NEt (371 μL, 2.12 mmol) and Bu_4NI (1.9 mg) in 1,2-dichloroethane was heated at reflux overnight. Upon cooling, the reaction was quenched by addition of 10 % aqueous Na_2CO_3. The reaction solution was washed with brine, dried (Na_2SO_4) and evaporated under reduced pressure. The residue was purified by flash chromatography (2:1 and then 3:2 v/v pet. ether:EtOAc) to give MOM ether **10** (139 mg, 91%) as colorless oil.

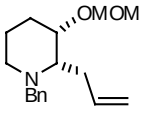


10

$[\alpha]_D^{22} = -72.5^o$ (*c* 1.0, $CHCl_3$); IR ν_{max}: 1643, 1449, 1032 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz), δ: 7.15–7.30 (m, 5H), 5.75–5.85 (m, 1H), 5.40 (d, 1H, J = 15.1 Hz), 5.05–5.15 (m, 2H), 4.56 (d, 1H, J = 6.9 Hz), 4.52 (d, 1H, J = 6.9 Hz), 3.90 (d, 1H, J = 15.1 Hz), 3.77 (q, 1H, J = 4.8 Hz), 3.38–3.46 (m, 1H), 3.27 (s, 3H), 2.45–2.70 (m, 3H), 2.26–2.38 (m, 1H), 1.88–2.08 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz), δ: 169.4, 137.2, 135.5, 128.6, 127.8, 127.3, 117.7, 95.6, 72.9, 57.9, 55.6, 48.7, 33.9, 28.8, 23.3; EI-HRMS calcd for $C_{14}H_{18}NO_3$ ($M–CH_2CH=CH_2$) 248.1287, found 248.1295.

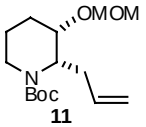
Preparation of compound **11**

To a solution of **10** (289 mg, 0.45 mmol) in THF (5 mL) was added $LiAlH_4$ (1 M in THF, 0.54 mL). The mixture was heated at reflux for 1 h, cooled at 0 °C, and the reaction was quenched by addition of 10 drops of water and 7 drops of 5 M aqueous NaOH solution. The mixture was stirred for another 20 min, at which time the suspension was filtered through a short pad of Celite®. The solvent was removed under reduced pressure and the residue was purified by flash chromatography (CH_2Cl_2) to give *N*-benzyl piperidine derivative (114 mg, 93%) as a light yellow oil.



$[\alpha]_D^{22} = -14.2^o$ (*c* 1.23, $CHCl_3$); IR ν_{max}: 1143, 1096, 1037 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz), δ: 7.20–7.32 (m, 5H), 5.82–5.96 (m, 1H), 5.06 (d, 1H, J = 17.1 Hz), 4.99 (d, 1H J = 10.0 Hz), 4.64 (s, 3H), 3.75–3.85 (m, 1H), 3.74 (s, 2H), 3.36 (s, 3H), 2.83–2.94 (m, 1H), 2.42–2.62 (m, 2H), 2.28–2.40 (m, 2H), 1.40–1.70 (m, 4H); ^{13}C NMR ($CDCl_3$, 75 MHz), δ: 139.5, 138.2, 128.6, 128.1, 126.7, 115.4, 95.2, 74.5, 62.3, 58.2, 55.4, 46.6, 29.0, 27.1, 21.8; EI-HRMS calcd for $C_{14}H_{20}NO_2$ ($M-CH_2CH{=}CH_2$) 234.1494, found 234.1500.

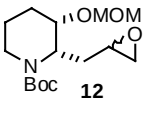
To an ice-cold solution of the above amine (45 mg, 0.16 mmol) in 1,2-dichloroethane (3 mL) was added 1-chloroethyl chloroformate (26 μL, 0.24 mmol). The reaction mixture was heated at reflux for 5 h, at which time the solvent was removed under reduced pressure. The residue was dissolved in methanol (3 mL) and heated at reflux for 30 min. The solvent was evaporated under reduced pressure. The crude secondary amine was dissolved in THF (2 mL) and treated with Boc_2O (52 mg) and $NaHCO_3$ (40 mg) in water (2 mL). The suspension was vigorously stirred for 3 h, at which time it was extracted with ethyl acetate (x3). The combined organic layer was dried (Na_2SO_4), filtered and evaporated under reduced pressure. The residue was purified by flash chromatography (5:1 v/v pet. ether:EtOAc) to give *N*-Boc piperidine derivative **11** (41 mg, 92%) as a light yellow oil.



11

$[\alpha]_D^{22} = +34.8^o$ (*c* 1.15, $CHCl_3$); IR ν_{max}: 1690, 1414, 1361, 1149, 1038 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz), δ: 5.66–5.83 (m, 1H), 4.93–5.11 (m, 2H), 4.69 (d, 1H, J = 6.7 Hz), 4.66 (d, 1H, J = 6.7 Hz), 3.61–3.70 (m, 1H), 3.38 (s, 3H), 2.61–2.75 (m, 1H), 2.32–2.41 (m, 2H), 1.78–1.88 (m, 1H), 1.43–1.71 (m, 5H), 1.52(s, 3H), 1.43(s, 6 H).^{13}C NMR ($CDCl_3$, 75 MHz), δ: 155.0, 135.3, 116.5, 95.1, 85.1, 79.4, 74.2, 55.4, 28.6, 28.3, 27.4, 25.6, 24.2; EI-HRMS calcd for $C_{15}H_{28}NO_4$ (MH^+) 286.2018, found 286.2020.

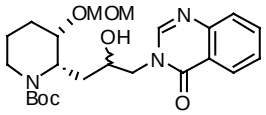
Preparation of compound **12**



12

To a solution of **11** (32 mg, 0.11 mmol) in CH_2Cl_2 (1 mL) was added 30% hydrogen peroxide (0.2 mL), $MeReO_3$ (MTO) (0.2 mg) and 3-cyanopyridine (4.6 mg). The suspension was vigorously stirred for 2.5 days, at which time ice water was added and extracted with CH_2Cl_2 (x3). The combined organic layers were washed with saturated aqueous $CuSO_4$ solution (x3), dried (Na_2SO_4), filtered and then evaporated under reduced pressure. The residue was purified by flash chromatography (5:1 v/v pet. ether:EtOAc) to give recovered **11** (8 mg), and the desired epoxide **12** (20 mg, 80% conversion) as an inseparable mixture of diastereomers. IR ν_{max}: 1689, 1413, 1149, 1037 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz), δ (two diastereomers): 4.50–4.80 (m, 3H), 3.80–4.10 (m, 1H), 3.55–3.75 (m, 1H), 3.35 (s, 3H), 2.85–3.00 (m, 1H), 2.50–2.80 (m., 2H), 2.35–2.45 (m, 1H), 1.55–1.90 (m, 4H), 1.30–1.55 (m, 2H), 1.44 (s, 9H); ^{13}C NMR ($CDCl_3$, 75 MHz), δ (two diastereomers) :154.8, 95.2, 79.9, 74.0, 55.5, 50.5, 47.2, 46.4, 28.3, 27.7, 27.5, 25.7, 24.1; CI(NH_3)-HRMS calcd for $C_{15}H_{28}NO_5$ (MH^+) 302.1967, found 302.1978.

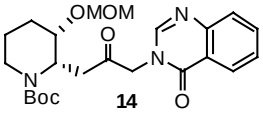
Preparation of compound **13**.



13

To a solution of epoxide **12** (37 mg, 0.123 mmol) in methanol (1.5 mL) was added 4-hydroxyquinazoline (22 mg, 0.148 mmol) and potassium hydroxide (1.3 mg, 0.024 mmol). The reaction mixture was heated at reflux for 48 h. Upon cooling, the solvent was removed under reduced pressure. The crude oil was dissolved in CH_2Cl_2 (10 mL), washed with 1 M aqueous NaOH (x3) and brine. The organic layer was dried (Na_2SO_4), filtered and evaporated under reduced pressure. The residue was purified by flash chromatography (2:1 v/v pet. ether:EtOAc and then EtOAc) to give recovered epoxide **12** (5 mg), and secondary alcohol **13** (32 mg, 68% conversion) as an inseparable diastereomeric mixture. $[\alpha]_D^{22} = +38.6^o$ (*c* 1.10, $CHCl_3$); IR ν_{max}: 3150–3550, 1681, 1612, 1416 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz), δ (two diastereomers): 8.20–8.35 (m, 2H), 7.68–7.78 (m, 2H), 7.45–7.53 (m, 1H), 4.66 (s, 2H), 4.55–4.70 (m, 1H), 4.33–4.55 (m, 2H), 3.80–3.90 (m, 1H), 3.65–3.80 (m, 1H), 3.54–3.65 (m, 1H), 3.36 (s, 3H), 2.62–2.80 (m, 1H), 1.77–1.95 (m, 2H), 1.60–1.77 (m, 2H), 1.44 (s, 3H), 1.36 (s, 6H), 1.20–1.60 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz), δ (two diastereomers): 161.2, 156.6, 148.1, 148.0, 134.3, 134.2, 127.4, 127.2, 127.1, 126.9, 126.7, 126.6, 95.4, 95.0, 80.9, 80.7, 77.2, 73.4, 65.4, 55.7, 55.6, 52.0, 51.8, 49.5, 38.7, 30.0, 28.7, 28.4, 28.2, 28.1, 25.7, 24.0; EI-HRMS calcd for $C_{23}H_{33}N_3O_6$ 447.2369, found 447.2361.

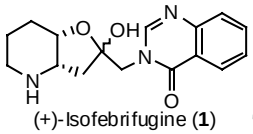
Preparation of compound **14**.



14

To a solution of **13** (16 mg, 0.036 mmol) in CH_2Cl_2 (2 mL) was added Dess-Martin periodinane (0.3 M in CH_2Cl_2, 0.36 mL, 0.10 mmol). The mixture was stirred at rt for 2.5 h, then was quenched by addition of aqueous $Na_2S_2O_3$ and extracted with EtOAc (x3). The combined organic layers were washed with saturated aqueous $NaHCO_3$ (x2), brine, dried (Na_2SO_4), filtered and then evaporated under reduced pressure. The residue was purified by flash chromatography (1:2 v/v pet. ether:EtOAc and then EtOAc) to afford ketone **14** (14.6 mg, 92%): $[\alpha]_D^{22} = +50.0^o$ (*c* 0.60, $CHCl_3$); IR ν_{max}: 1729, 1680, 1612, 1473, 1412, 1364 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz), δ: 8.28 (dd, 1H, J = 8.4, 0.8), 8.02 (br. s, 1H), 7.68–7.80 (m, 2H), 7.49 (ddd, 1H, J = 8.1, 6.5, 1.9 Hz), 5.10–5.30 (m, 1 H), 4.85–5.05 (m, 2H), 4.68 (br. s, 2H), 3.78–3.95 (br. s, 1H), 3.74 (quint, 1H, J = 10.9, 5.6 Hz), 3.40 (s, 3H), 2.96–3.12 (m, 1H), 2.74–2.92 (m, 1H), 2.50–2.72 (m, 1H), 1.84–1.96 (m, 1H), 1.60–1.80 (m, 1H), 1.32–1.56 (m, 2H), 1.44 (s, 9H); ^{13}C NMR ($CDCl_3$, 75 MHz), δ: 201.1, 160.9, 155.4, 148.1, 147.0, 134.3, 127.4, 127.2, 126.8, 121.9, 95.6, 80.6, 77.2, 74.2, 55.8, 53.4, 51.3. 38.5, 36.6, 28.4, 25.5, 23.8; EI-HRMS calcd for $C_{23}H_{31}N_3O_6$ 445.2213, found 445.2209.

(+)-Isofebrifugine (**1**)

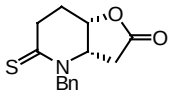


(+)-Isofebrifugine (**1**)

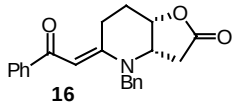
The solution of MOM ether **14** (11 mg, 0.024 mmol) in 6 M aqueous HCl (2 mL) was heated at reflux for 2.5 h. After the reaction mixture was cooled to rt, the mixture was treated with solid Na_2CO_3 and then extracted with chloroform (x3). The combined organic layers were dried (Na_2SO_4), filtered and then evaporated under reduced pressure. The residue was purified by flash

chromatography (2:1 v/v CH_2Cl_2:MeOH) to afford isofebrifugine (**1**) (6.6 mg, 89%): $[\alpha]_D^{22}$ +120.8° (*c* 0.30, $CHCl_3$); IRν_{max}: 3100–3575, 1672, 1609, 1473 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz), δ: 8.30 (dd, 1H, J = 7.8, 1.3 Hz), 8.29 (s, 1H), 7.75 (ddd, 1H, J = 8.1, 8.1, 1.4 Hz), 7.70 (dd, 1H, J = 8.1, 1.4 Hz), 7.49 (dtd, 1H, J = 8.1, 6.8, 1.5 Hz), 4.44 (d, 1H, J = 13.9 Hz), 4.15 (d, 1H, J = 13.9 Hz), 3.89 (q, 1H, J = 2.7 Hz), 3.30 (dd, 1H, J = 3.3, 3.3 Hz), 2.99 (dd, 1H, J = 11.0, 2.8 Hz), 2.54 (ddd, 1H, J = 13.2, 2.0, 13.2 Hz), 2.05–2.15 (m, 2H), 1.88 (d, 1H, J = 13.1 Hz), 1.75–1.86 (m, 1H), 1.47–1.62 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz), δ: 161.5, 148.2, 148.1, 134.3, 127.5, 127.0, 126.9, 121.9, 105.4, 77.6, 55.7, 49.9, 44.5, 43.3, 26.7, 20.0; CI(NH_3)-HRMS calcd for $C_{16}H_{20}N_3O_3$ (MH^+) 302.1504, found 302.1495.

Preparation of compound **16**.



The bicyclic lactam lactone **6** (361 mg, 1.47 mmol) was dissolved in dry THF (20 mL) under Ar. Lawesson's reagent (328 mg, 0.81 mmol, 0.55 mol eq) was added to the solution and the mixture was refluxed. After 3 h, the recation mixture was cooled and then concentrated. The crude product was purified by chromatography, initially using 5:1 v/v CH_2Cl_2-hexane as eluent to remove the non-polar sulfur impurities and then 2:1 v/v pet. ether-EtOAc to obtain the desired thiolactam **17** (376 mg, 98 %) as a viscous pale yellow oil.
$[\alpha]_D^{22}$ = +85.0° (*c* 0.50, $CHCl_3$); IR ν_{max}: 3075, 3050, 1778, 1343, 1161 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz), δ: 7.25–7.38 (m, 5H), 6.10 (d, 1H, J = 14.8 Hz), 4.58 (d, 1H, J = 14.8 Hz), 4.90–4.98 (m, 1H), 4.33 (ddd, 1H, J = 8.8, 7.5, 4.3 Hz), 3.29 (ddd, 1H, J = 17.6, 3.7 Hz), 2.92 (ddd, 1H, J = 16.9, 2.4, 4.1 Hz), 2.83 (dd, 1H, J = 18.6, 9.1 Hz), 2.57 (dd, 1H, J = 18.6, 4.1 Hz), 2.20 (ddd, 1H, J = 14.5, 7.9, 3.9 Hz), 1.91 (dddd, 1H, J = 14.5, 12.5, 3.6 Hz). ^{13}C NMR ($CDCl_3$, 75 MHz), δ: 202.0, 173.1,134.5, 129.1, 128.3, 127.9, 76.0, 56.2, 55.3, 36.6, 35.3, 24.6; EI-HRMS calcd for $C_{14}H_{15}NO_2S$ 261.0823, found 261.0816.

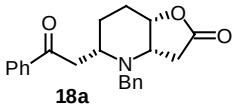


16

The above thiolactam (280 mg, 1.07 mmol) was dissolved in dry MeCN (0.5 mL) under Ar. A solution of 98 % phenacyl bromide (261 mg, 1.29 mmol, 1.2 mol eq) in dry MeCN (2 mL) was added via cannula to the thiolactam solution. The reaction mixture was stirred in the dark at rt for 24 h. The reaction mixture was diluted with dry CH_2Cl_2 (5 mL). A solution of Ph3P (508 mg, 1.94 mmol) in dry CH_2Cl_2 (4 mL) was added via cannula and the mixture was stirred at rt for 15 min. Then 1-methylpiperidine (394 uL, 3.21 mmol) was added and the mixture was stirred in the dark at rt for 24 h. The reaction mixture was concentrated, toluene (10 mL) was added to the residue and the mixture was evaporated. The crude product was purified by chromatography (1:1 v/v pet. ether-EtOAc and then 2:1 v/v EtOAc-pet. ether) to furnish the vinylogous amide **16** (249.8 mg, 67 %) as a light brown solid. mp: 56–56.5 °C; $[\alpha]_D^{22}$ = +104.6° (*c* 2.70, $CHCl_3$); IR ν_{max}: 3050, 3012,1778, 1613, 1600, 1587, 1562, 1525cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz), δ: 7.59 (br d, 2H, J = 7.1 Hz), 7.18–7.44 (m, 8H), 5.88 (br. s, 1H), 4.99 (ddd, 1H, J = 7.4, 3.7, 3.7 Hz), 4.63 (d, 1H, J = 16.3 Hz), 4.39 (d, 1H, J = 16.3 Hz), 4.28 (ddd, 1H, J = 4.3, 7.9, 7.9 Hz), 4.07 (ddd, 1H, J = 18.1, 3.1, 3.1 Hz), 2.81 (dd, 1H, J = 18.3, 8.5 Hz), 2.77–2.90 (m, 1H), 2.57 (dd, 1H, J = 18.3, 4.2 Hz), 2.18 (dddd, 1H, J = 13.4, 4.2, 4.2, 4.2 Hz), 1.82–1.95 (m, 1H). ^{13}C NMR ($CDCl_3$, 75 MHz), δ: 189.1, 174.3,162.2, 142.2, 135.3, 131.0, 129.4, 128.4, 128.2, 127.5, 126.9, 95.3, 76.9, 57.2, 54.3, 36.1, 23.7, 21.7. EI-HRMS calcd for $C_{22}H_{21}NO_3$ 347.1521, found 347.1526.

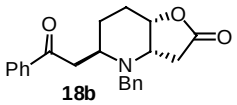
Preparation of compound **18a**,**b**.

A suspension of platinum(IV) oxide (40 mg) in ethyl acetate (20 mL) was activated by mixing with hydrogen (45 psi) in a Parr flask for 30 min. A solution of the vinylogous amide **16** (244 mg, 0.70 mmol) in ethyl acetate (30 mL) was then added to the activated catalyst and the mixture was shaken under hydrogen (45 psi) for 4 h. The mixture was filtered through Celite®, the residue washed several times with ethyl acetate. The combined filtrates were evaporated under reduced pressure and the residue was purified by chromatography (pet. ether:EtOAc/2:1) to give 215 mg (88%) of phenyl ketone **18a** as white solid and the minor epimer **18b** (24 mg, 10%).



18a

Compound **18a**: mp: $[\alpha]_D^{22} = -31.4^o$ (*c* 0.875, CH_2Cl_2); IR ν_{max}: 1778, 1678, 1219, 1155 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz), δ: 7.63 (br d, 2H, J = 7.3 Hz), 7.49 (br t, 1H, J = 7.3 Hz), 7.14–7.40 (m, 7H), 4.55 (ddd, 1H, J = 3.4, 3.0, 3.0 Hz), 3.78 (d, 1H, J = 17.0 Hz), 3.70 (d, 1H, J = 17.0 Hz), 3.35 (t, 1H, J = 4.1 Hz), 3.22–3.32 (m, 1H), 3.09 (dd, 1H, J = 3.9, 17.2 Hz), 2.71 (dd, 1H, J = 17.2, 8.2 Hz), 2.68 (dd, 1H, J = 16.8, 4.7 Hz), 2.52 (d, 1H, J = 16.7 Hz), 2.12–2.24 (m, 1H), 1.60–1.84 (m, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz), δ: 198.4, 176.2, 140.5, 136.7, 132.8, 128.3 128.2, 127.5, 126.63, 126.6, 78.0, 60.7, 57.3, 56.8, 43.7, 38.8, 26.5, 24.8. EI-HRMS calcd for $C_{22}H_{23}NO_3$ 349.1678, found 349.1669;calcd for $C_{15}H_{16}NO_3$ (M–$PhCH_2$) 258.1130, found 258.1127.

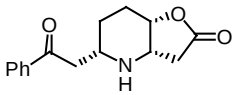


18b

Compound **18b**: IR ν_{max}: 1772, 1672 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz), δ: 7.67 (br d, 2H, J = 7.5 Hz), 7.44 (t, 1H, J = 7.3 Hz), 7.14–7.34 (m, 7H), 4.45–4.54 (m, 1H), 3.76 (d, 1H, J = 14.7 Hz), 3.43 (d, 1H, J = 14.4 Hz), 3.23–3.50 (m, 3H), 2.85 (dd, 1H, J = 14.0, 9.4 Hz), 2.63 (dd, 1H, J = 17.0, 2.0 Hz), 2.54 (dd, 1H, J = 17.0, 4.9 Hz), 1.75–1.98 (m, 3H), 1.25–1.38 (m, 1H). ^{13}C NMR ($CDCl_3$, 75 MHz), δ: 199.4, 175.9, 138.0, 136.4, 133.3, 128.7, 128.6, 128.1, 128.0, 127.5, 77.6, 55.5, 54.3, 50.5, 36.2, 34.4, 24.7, 21.3. EI-HRMS calcd for $C_{22}H_{23}NO_3$ 349.1678, found 349.1680;calcd for $C_{15}H_{16}NO_3$ (M–$PhCH_2$) 258.1130, found 258.1127.

Preparation of compound **19**.

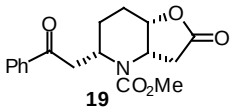
Palladium(II) hydroxide/C (20 wt %, 114 mg) was washed with dry ethanol (10 mL) and then suspended in fresh, dry ethanol (10 mL). Redistilled cyclohexene (3 mL) was added and the mixture was refluxed under Ar for 30 min. and then cooled. A solution of the N-benzyl piperidine **18a** (228 mg) in dry ethanol (20 mL) was added and the mixture was refluxed under Ar. After 2 h, the reaction mixture was cooled to rt and then filtered through Celite®, and the residue washed several times with 95% ethanol. The combined filtrates were concentrated to give crude piperidine derivative (169 mg). An analytical sample was purified by chromatography (EtOAc and then 10:1 v/v EtOAc:MeOH).



$[\alpha]_D^{22} = +20.0^o$, (*c* 1.25, CH_2Cl_2); IR ν_{max}: 1772, 1678 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz), δ: 7.89–7.96 (m, 2H), 7.57 (dt, 1H, J = 7.3, 1.3 Hz), 4.40 (ddd, 1H, J = 3.2, 3.2, 3.2 Hz), 7.40–7.50 (m, 2H), 3.67 (dd, 1H, J = 4.2, 3.4 Hz),3.14–3.25 (m, 1H), 3.08 (dd, 1H, J = 17.6, 3.8 Hz), 2.99 (dd, 1H, J = 17.6, 8.4 Hz), 2.67 (dd, 1H, J = 16.5, 4.4 Hz),2.20–2.36 (m, 2 H),1.67–1.82 (m, 1H), 1.48–1.58 (m, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz), δ: 199.2, 176.9, 136.7, 133.4, 128.6, 127.9, 76.9, 53.4,

49.8, 44.9, 40.4, 26.1, 25.9. HRMS calcd for $C_{15}H_{17}NO_3$ 259.1208, found 259.1200.

The crude amine above (169 mg) was dissolved in CH_2Cl_2 (20 mL) and distilled water (10 mL) was added. Potassium carbonate (1.04 g, 7.53 mmol, 11.5 mol eq) was added to the biphasic mixture. The mixture was cooled to 0°C and methyl chloroformate (466 uL, 6.0 mmol, 9.2 mol eq) was added. The mixture was vigorously stirred for 22 h. Then the aqueous phase was separated and back extracted with CH_2Cl_2. The combined CH_2Cl_2 layers were washed with water, dried, filtered and concentrated. The residual oil was purified by flash chromatography (2:1 v/v pet. ether-EtOAc and then 1:1v/v pet. ether-EtOAc) to yield the carbamate **19** (131.2 mg, 63%).

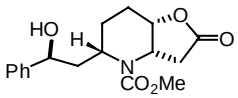


19

$[\alpha]^{29}_D$ = +47 ° (*c* 1.22, CH_2Cl_2), IR ν_{max}: 3060, 1780, 1697, 1681, 1595, 1579 cm^{-1}. ^1H NMR ($CDCl_3$, 300 MHz), δ: 7.89 (d, 2H, J = 7.5 Hz), 7.51 (t, 1 H, J = 7.4 Hz), 7.40 (t, 2H, J = 7.8 Hz), 5.01 (q, 1H, J = 8.9 Hz), 4.72 (ddd, 1H, J = 10.7, 5.6, 5.6 Hz), 3.61 (s, 3H), 4.55–4.61 (m, 1H), 3.28 (br d, 1 H, 15.5 Hz), 3.06 (dd, 1H, J = 15.5, 10.0 Hz), 2.92 (dd, 1H, J = 18.3, 9.8 Hz), 2.50 (dd, 1H, J = 18.3, 8.4 Hz), 1.68–1.93 (m, 3H), 1.55–1.62 (m, 1H). ^{13}C NMR ($CDCl_3$, 75 MHz), δ: 197.2, 174.2, 156.0, 136.2, 133.3, 128.6, 128.0, 76.0, 53.0, 48.8, 48.1, 43.9, 34.4, 23.3, 22.4. EI-HRMS: calcd for $C_{17}H_{19}NO_5$ 317.1263, found 317.1271.

Preparation oc compound **20**.

A mixture of (*R*)-2-methyl-CBS-oxazaborolidine (1.0 M in toluene, 247 μL, 0.247 mmol, 0.6 mol eq) and $BH_3.SMe_2$ complex (1.67 M in ether, 148 μL, 247 mmol, 0.6 mol eq) in dry THF (4 mL) was cooled to –40 °C, under Ar. A solution of the keto lactone **19** (131 mg, 0.413 mmol) in dry THF (3 mL) and was added via cannula and the reaction mixture was stirred at –40 °C for 50 min. Saturated NH_4Cl (1 mL) was added at –40 °C and the reaction mixture was allowed to warm slowly to rt. Brine (5 mL) and EtOAc (10 mL) were added and the mixture was stirred. The aqueous phase was separated and re-extracted with EtOAc (2 x 10 mL). The combined organic phases were washed with brine, dried, filtered and concentrated. The crude oil was purified by chromatography (1:1 v/v pet. ether-EtOAc) to afford the alcohol (113.6 mg, 86 %).

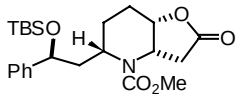


15 (R = Me)

$[\alpha]_D^{29.5}$ = –2.79 ° (*c* 2.68, CH_2Cl_2). IR ν_{max}: 3589–3307, 3048, 1778, 1736, 1689 cm^{-1}. ^1H NMR ($CDCl_3$, 300 MHz), δ: 7.05–7.07 (m, 5H), 5.04 (q, 1H, J = 9.0 Hz), 4.74–4.83 (m, 2H), 4.34–4.43 (m, 1H), 2.87 (dd, 1H, J = 18.3, 9.7 Hz), 2.49 (dd, 1H, J = 18.3, 9.0 Hz), 2.10 (ddd, 1H, J = 13.9, 9.8, 4.0 Hz), 1.85–1.97 (m, 4H), 1.67 –1.75 (m, 1H), 1.58–1.65 (br s, 1H). EI-HRMS: calcd for $C_{17}H_{21}NO_5$ 319.1420, found 319.1416.

The above secondary alcohol (113 mg, 0.356 mmol) was dissolved in dry CH_2Cl_2 (3 mL) under Ar. 2,6-Lutidine (174 μL, 1.49 mmol, 4.2 mol eq) was added. The mixture was cooled to –10 °C. Then TBSOTf (163 μL, 0.712 mmol, 2 mol eq) was added dropwise to the mixture. After addition is complete, the reaction mixture was stirred at –10 °C for 15 min and then at 0 °C for 30 min. Then saturated $NaHCO_3$ (2 mL) was added followed by CH_2Cl_2 (10 mL). The mixture was stirred and the aqueous was separated and back extracted with CH_2Cl_2 (2 x 5 mL). The combined CH_2Cl_2 layers were washed with 0.5 N aqueous H_2SO_4 (2 x 5 mL), water and saturated $NaHCO_3$. The organic extract was

dried, filtered and concentrated to give an oil. Purification by chromatography (2:1 v/v pet. ether-EtOAcgave the TBS ether **20** (153 mg, 100 %).

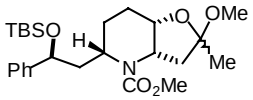


20

$[\alpha]_D^{24}$ = –20.6 ^{o} (*c* 1.34, CH_2Cl_2). IR ν_{max}: 3062, 3029, 1784, 1700 cm^{-1}. ^1H NMR ($CDCl_3$, 300 MHz), δ: 0.06, 0.00 and –0.25 (s, 6H), 0.88 (s, 9H), 1.62–1.85 (m, 5H), 2.04 (ddd, 1H, J = 13.1, 8.6, 2.7 Hz), 2.34 (dd, 1H, J = 18.2, 9.0 Hz), 2.83 (dd, 1H, J = 18.2, 9.8 Hz), 3.65 (s, 3H), 4.17–4.27 (m, 1H), 4.64–4.72 (m, 2H), 5.00 (q, 1H, J = 9.1 Hz), 7.20–7.30 (m, 5H). ^{13}C NMR ($CDCl_3$, 75 MHz), δ: 174.2, 156.1, 144.1, 128.1, 127.3, 125.7, 76.0, 72.3, 52.8, 48.6, 47.9, 46.0, 33.9, 25.6, 23.2, 21.9, 18.0, –3.7, –4.7, –5.3. CI(NH_3)-HRMS: calcd for $C_{23}H_{39}N_2O_5Si$ (M+NH_4) 451.2628, found 451.2633.

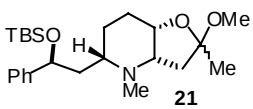
Preparation of compound **21**.

The bicyclic lactone **20** (154.2 mg, 0.356 mmol) was dissolved in dry toluene (9 mL) under Ar, and dry pyridine (0.5M in toluene, 3.56 mL, 1.78 mmol, 5 mol eq) was added. Cl_2TiMe_2 (0.2 M in toluene, 1.07 mmol, 5.35 mL, 3 mol eq) was added and the mixture was heated in an oil bath at 90 ^{o}C. After 1.5 h, the reaction mixture was cooled and a mxture of 2:1 v/v pet. ether-EtOAc containing 1 % Et_3N (10 mL) was added. The reaction mixture was filtered through a short pad of silica gel-60. The residue was washed several times with 2:1 v/v pet. ether-EtOAc containing 1 % Et_3N, and the combined filtrates were evaporated. The residual oil was dried under high vacuum for 10 min and then redissolved, under Ar, in dry MeOH. PPTS (10 mg) was added and the mixture was stirred for 4 h at rt. Et_3N (1 mL) was added, the reaction mixture was concentrated and the residue purified by chromatography (4:1 v/v pet. ether-EtOAc) to furnish the cyclic ketal (92.1 mg, 57 %) as a mixture of anomers [6:1 based on integration of the ketal OMe siglet at δ 3.18 (major) and 3.24 (minor)].



IR ν_{max}: 3061, 3027, 1701, 1603, 1545 cm^{-1}. ^1H NMR (CD_3OD, 300 MHz), δ (minor anomer in brackets): 7.20–7.40 (m, 5H), 4.91 (q, 1H, J = 9.3 Hz) 4.78–4.85 (m, 1H), 4.20–4.29 (m, 1H), 4.03–4.15 (m, 1H), 3.66 (s, 3H), 3.18 (3.24) (s, 3H), 2.16–2.28 (m, 1H), 1.43–1.80 (m, 5H) 1.35 (1.40) (s, 3H), 0.91, (s, 9H), 0.06 and –0.22 (s, 6H).

A solution of the above cyclic ketal (92 mg, 0.198 mmol) in dry THF (5 mL) was transferred via cannula, under Ar, to a suspension of $LiAlH_4$ (75 mg, 1.99 mmol, 10 mol eq) in dry THF (4 mL). The mixture was refluxed for 16 h. The reaction mixture was cooled to rt and then at 0 ^{o}C. Aqueous 10 % NaOH was added dropwise to destroy unreacted $LiAlH_4$ and until all aluminum salts redissolved. Celite was added to the mixture and then CH_2Cl_2 (20 mL). The mixture was stirred for 30 min and then suction filtered. The residue was washed with CH_2Cl_2 (2 x 10 mL), and the combined filtrates were dried, filtered and concentrated. The crude product was purified by chromatography (3:1 v/v pet. ether-EtOAc) to yield the amine **21** (61.5 mg, 74 %).



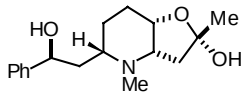
21

IR ν_{max}: 3060, 3028 cm^{-1}. ^1H NMR ($CDCl_3$, 300 MHz), δ: 7.20–7.35 (m, 5H), 4.68 (dd, 1H, J = 9.4, 2.6 Hz), 4.00–4.08 (m, 1H), 3.21 (s, 3H), 2.58–2.66 (m, 1H), 2.05–2.21 (m, 3H), 2.09 (s, 3H), 1.82–1.95 (m, 1H), 1.55–1.70 (m, 3H), 1.46 (s, 3H), 0.86 (s, 9H), 0.05, 0.00 and –0.27 (s, 6H).

^{13}C NMR ($CDCl_3$, 75 MHz), δ: 145.9, 128.0, 126.9, 125.7, 107.1, 76.1, 72.4, 65.6, 58.6, 48.6, 46.3, 45.6, 40.1, 25.7, 24.6, 22.6, 18.0, 1.2, –4.1, –4.5. EI-HRMS: calcd for $C_{24}H_{41}NO_3Si$ 419.2856, found 418.2864.

(–)-Sedacryptine (**2**)

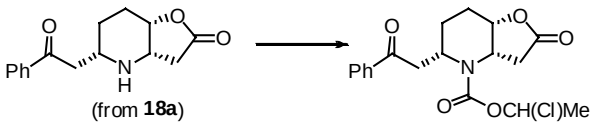
A solution of the cyclic ketal amine **21** (58.6 mg, 0.139 mmol) in dry THF (2 mL) was treated with Bu_4NF (1 M in THF, 167 μL, 0.167 mmol, 1.2 mol eq) at 0 °C, under Ar. The mixture was then stirred at rt for 2.5 h, concentrated and the residue was subjected to chromatography (15:1 v/v EtOAc-MeOH) to give the secondary alcohol (52.6 mg), which was immediately mixed with 0.1 M aqueous HCl (2 mL) and then reluxed for 30 min. The mixture was cooled to rt and then at 0 °C and was treated with 28 % NH_4OH to pH 9. The mixture was extracted with CH_2Cl_2 (2 x 10 mL) and the combined extracts were dried, filtered and concentrated. The residue was filtered through a short pad of silica gel-60 and using 10:1 v/v CH_2Cl_2-MeOH as solvent. The filtrate was concentrated and the residual oil was taken dissolved in MeOH and left to stand, in the dark, overnight (36 h). Then MeOH was evaporated and the residue was purified by chromatography (10:1v/v EtOAc-MeOH) to furnish (–)-sedacryptine (23.5 mg, 57.7 %).



(–)-**2**

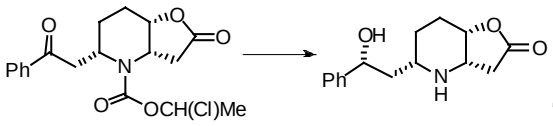
mp (cyclohexane): 123–125 °C; lit.[19d] mp (cyclohexane): 122–123 °C. $[\alpha]_D^{25}$ = –13.5 ° (*c* 0.74, $CHCl_3$). IR ν_{max}: 3530–3201, 3048 cm^{-1}. ^{1}H NMR ($CDCl_3$, 300 MHz), δ: 7.20–7.35 (m, 5H), 6.60–6.80 (br s, 1H), 4.81 (dd, 1H, J = 10.1, 2.8 Hz), 4.04 (q, 1H, J = 2.9 Hz), 2.67 (t, 1H, 3.5 Hz),, 2.25 (s, 3H), 2.23–2.35 (m, 2H), 2.20 (d, 1H, J = 13.2 Hz), 2.16 (ddd, 1H, J = 13.6, 10.2, 2.8 Hz), 2.05 (ddd, 1H, J = 13.6, 6.8, 1.8 Hz), 1.88 (dd, 1H, J = 13.2, 3.9 Hz), 1.77–1.85 (m, 2H), 1.48–1.62 (m, 2H), 1.45 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz), δ: 145.2, 128.6, 127.6, 125.5, 105.0, 76.9, 71.1, 65.5, 59.7, 44.3, 43.5, 39.7, 26.5, 25.5, 24.8. EI-HRMS: calcd for $C_{17}H_{23}NO_2$ (M–H_2O) 273.1729, found 273.1728.

Preparation of cyclic carbamate **22**.

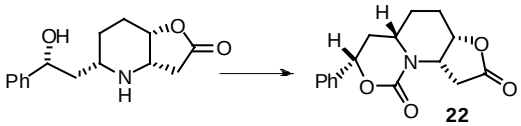


To a solution of secondary amine (43 mg, 0.17 mmol) in CH_2Cl_2 (4 mL) was added K_2CO_3 (140 mg, 1.02 mmol) in water (2 mL) and 1-chloroethyl chloroformate (44 μL, 0.41 mol). The suspension was vigorously stirred at rt for 2 h. Organic layer was separated and aqueous layer was extracted with CH_2Cl_2 (x3). Combined organic layers were washed with brine, dried (Na_2SO_4), filtered and evaporated under reduced pressure. The residue was purified by flash chromatography (1:1 v/v pet. ether:EtOAc) to give the keto carbamate (58 mg, 96%) as a white solid and as a mixture of diastereomers. mp: 55–56 °C; $[\alpha]_D^{24}$ = +27.4° (*c* 1.55, CH_2Cl_2); IR ν_{max}: 1778, 1713, 1678, 1407, 1372, 1296, 1096 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz), δ (two diastereomers): 7.92–8.00 (m, 2H), 7.54–7.62 (m, 1H), 7.42–7.52 (m, 2H), 6.57 (sextet, 1H, J = 5.8 Hz), 4.98–5.12 (m, 1 H), 4.76–4.86 (m, 1H), 4.60–4.76 (m, 1H), 3.43 (dd, 1H, J = 15.9, 3.2 Hz), 3.17 (dt, 1H, J = 15.9, 9.5 Hz), 3.02 (dd, 1H, J = 18.3, 9.7 Hz), 2.58 (dt, 1H, J = 18.3, 8.1 Hz), 1.60–2.06 (m, 7H). ^{13}C NMR ($CDCl_3$, 75 MHz), δ: (major epimer) 196.9, 173.8, 153.0, 136.2, 133.6, 128.7, 128.1, 83.2, 75.9, 49.1,

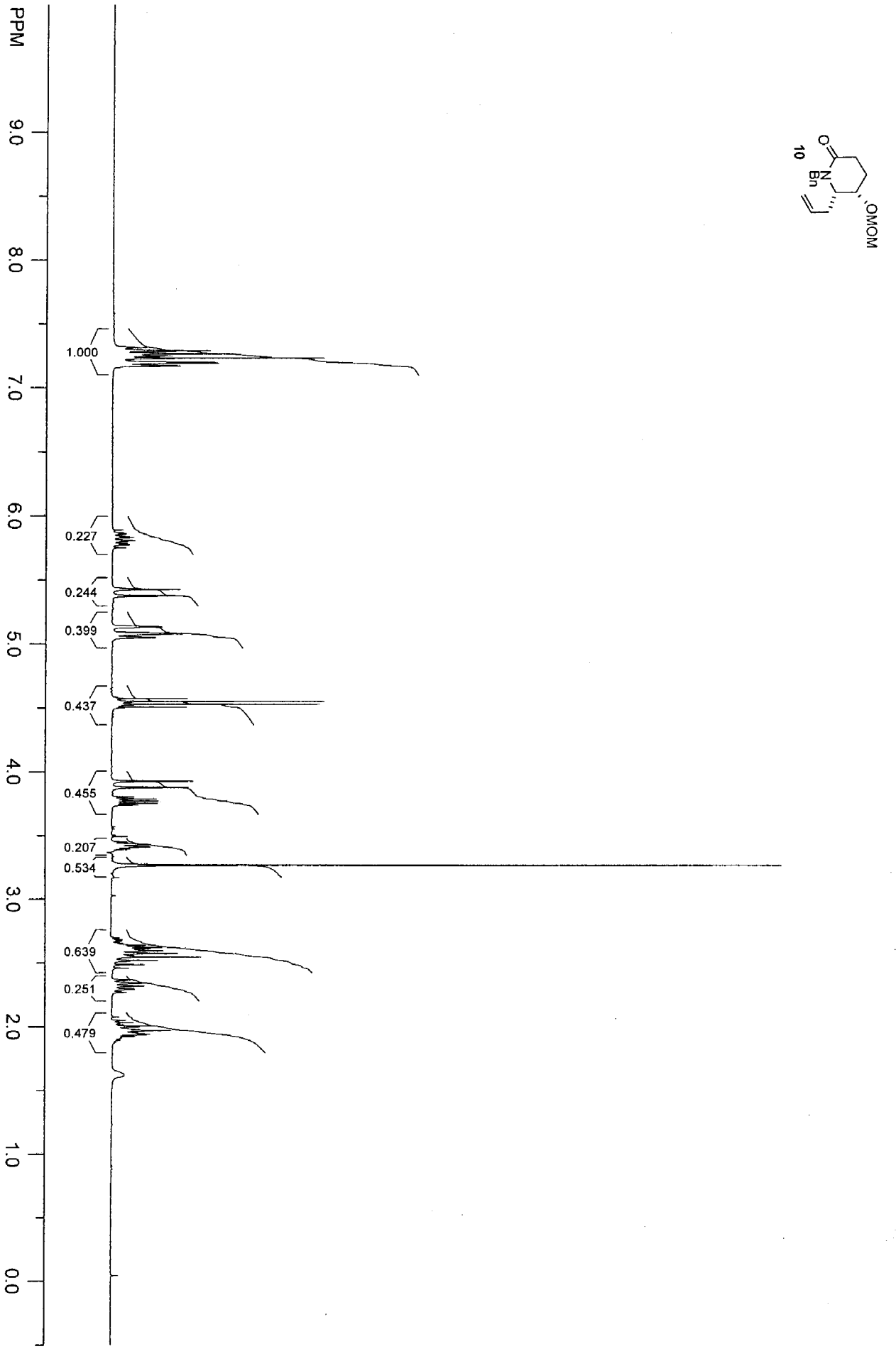
48.3, 43.7, 34.6, 25.2, 23.1, 22.3; δ (minor epimer): 196.8, 173.8, 152.9, 136.2, 133.6, 128.7, 128.1, 83.2, 75.8, 49.1, 48.5, 44.2, 34.5, 25.1, 23.4, 22.5; EI-HRMS calcd for $C_{18}H_{20}ClNO_5$ 365.1030, found 365.1031.



To a solution of phenyl ketone (14.5 mg, 0.04 mmol) in THF (2 mL) at –35 °C was added (*R*)-2-methyl-CBS-oxazaborolidine (1.0 M in toluene, 75 μL, 0.075 mmol) and $BH_3.SMe_2$ complex in ether (5.0 M, 24 μL, 0.024 mmol). It was stirred at –35 °C for 30 min and then reaction mixture was quenched by addition of water and diluted with EtOAc. Organic layer was separated and aqueous layer was extracted with EtOAc (x3). The combine organic layers were washed with brine, dried (Na_2SO_4), filtered and evaporated under reduced pressure. The residue was submitted to flash (1:1 v/v pet. ether:EtOAc) to give 14 mg of secondary alcohol. The secondary alcohol was dissolved in methanol (2 mL) and heated at reflux for 40 min. Upon cooling to rt, the reaction mixture was stirred with 150 mg of Amberlite resin IR-45 ($^-$OH) for 30 min. The resin was filtered off, washed twice with methanol and the combined filtrates were evaporated under reduced pressure. The residue was purified by flash chromatography (2:1 and then 1:1 v/v CH_2Cl_2:acetone) to give the amino alcohol (8.5 mg, 82%): $[\alpha]_D^{23} = -76.7^o$ (*c* 0.88, CH_2Cl_2); IR ν_{max}: 3318 (br.), 1772, 1155 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz), δ: 7.20–7.40 (m, 5H), 4.90 (dd, 1H, J = 10.2, 2.9 Hz), 4.35 (ddd, 1H, J = 4.0, 4.0, 3.0 Hz), 3.62 (dd, 1H, J = 5.4, 3.0 Hz), 3.06 (br. s, 2H), 2.86 (dddd, 1H, J = 13.9, 9.7, 2.5, 2.5 Hz), 2.79 (dd, 1H, J= 13.7, 5.4 Hz), 2.35 (d, 2H, J = 17.1 Hz), 1.70–1.90 (m, 2H), 1.46–1.68 (m, 2H), 1.26–1.43 (m, 1H). ^{13}C NMR ($CDCl_3$, 75 MHz), δ: 176.2, 144.5, 128.4, 127.4, 125.6, 76.3, 75.0, 55.2, 53.7, 45.0, 39.0, 26.5, 26.0. EI-HRMS calcd for $C_{15}H_{19}NO_3$ 261.1365, found 261.1362.



To a solution of amino alcohol **9** (8 mg, 0.031 mmol) in CH_2Cl_2 (3 mL) at –78 °C was added Et_3N (6 μL) and triphogene (12 mg, 0.04 mmol) in CH_2Cl_2 (1 mL) via cannula. The reaction mixture was stirred at –78 °C for 1 h and then was quenched by addition of saturated aqueous $NaHCO_3$ solution. The cold bath was removed and the reaction temperature was allowed towarm slowly to rt. The organic layer was separated and the aqueous phase was extracted with CH_2Cl_2 (x3). The combined organic layers were washed with brine, dried (Na_2SO_4), filtered and evaporated under reduced pressure. The residue was was purified by flash chromatography (2:1 then 1:1 v/v pet. ether: EtOAc and finally EtOAc) to give 5.5 mg (63%) of the cyclic carbamate. $[\alpha]_D^{22} = -11.1^o$ (*c* 0.225, $CHCl_3$); IR ν_{max}: 1778, 1689, 1407 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz), δ: 7.30–7.42 (m, 5H), 5.37 (dd, 1H, J = 12.0, 3.8 Hz), 4.79–4.84 (m, 1H), 4.27 ("t", 1H, J = 5.8 Hz), 3.51–3.62 (m, 1H), 3.27 (d, 1H, J = 18.9 Hz), 3.07 (dd, 1H, J = 18.9, 6.4 Hz), 2.29–2.38 (m, 1H), 2.24 (ddd, 1H, J = 13.8, 3.8, 2.5 Hz), 2.04 (ddd, 1H, J = 13.7, 11.7, 11.2 Hz), 1.70–1.82 (m, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz), δ: 175.6, 154.3, 138.8, 128.71, 128.67, 125.4, 79.2, 76.5, 53.5, 53.2, 39.8, 38.2, 24.6, 24.2. EI-HRMS calcd for $C_{16}H_{17}NO_4$ 287.1158, found 287.1159.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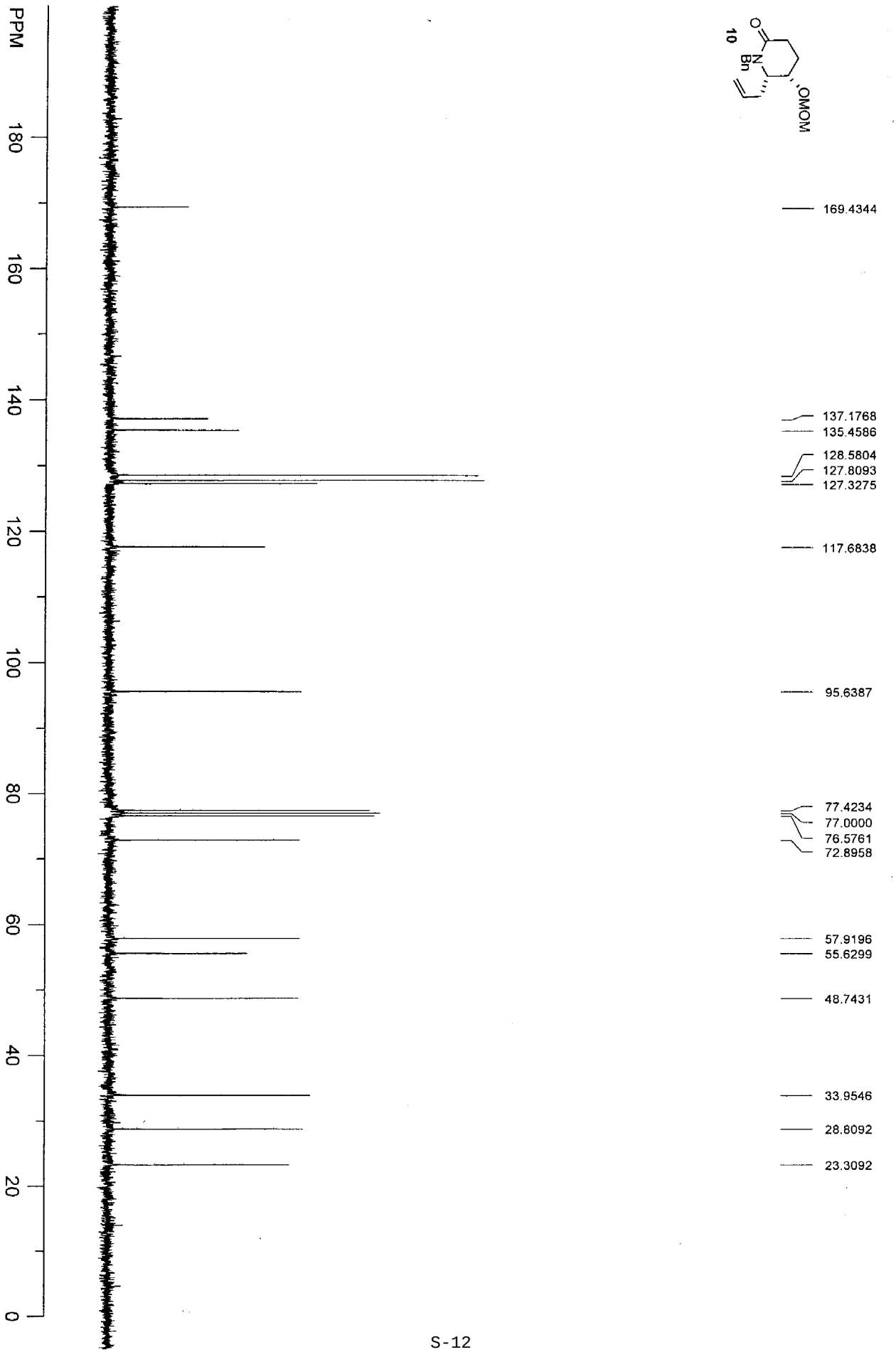
10
OMOM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Bn
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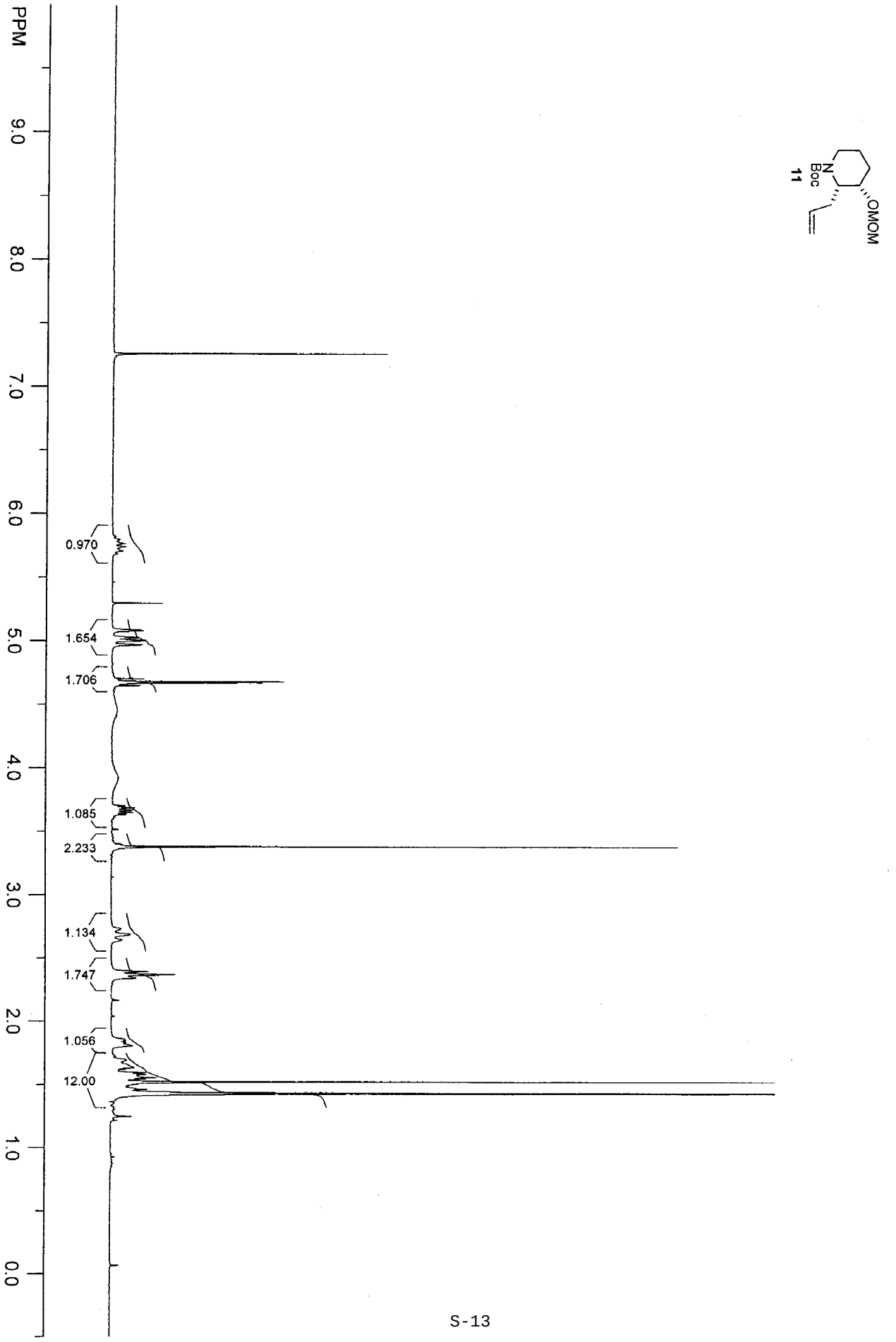
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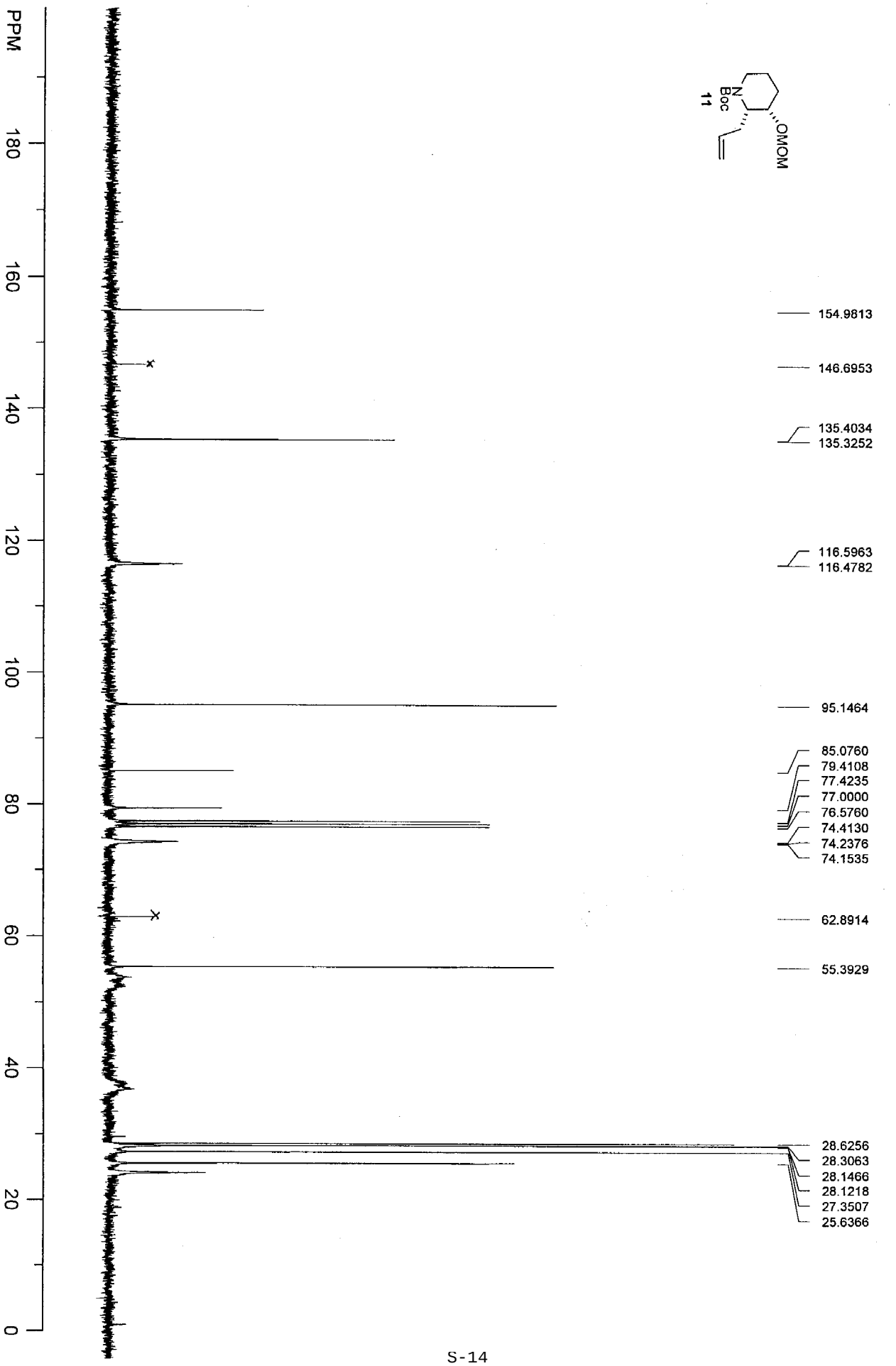
Boc
11
OMOM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40
20
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Boc
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76.5760
74.4130
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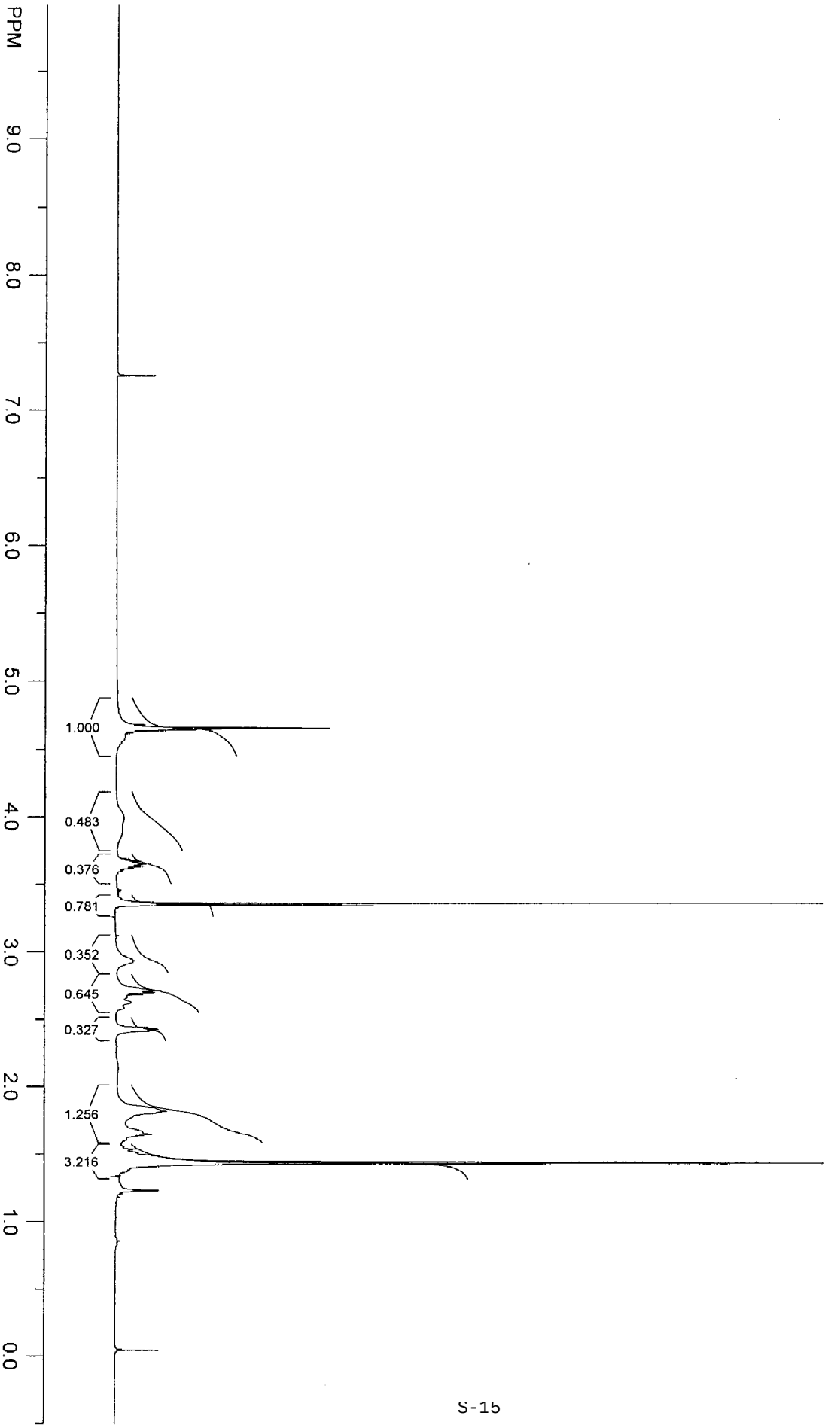
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Boc
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O
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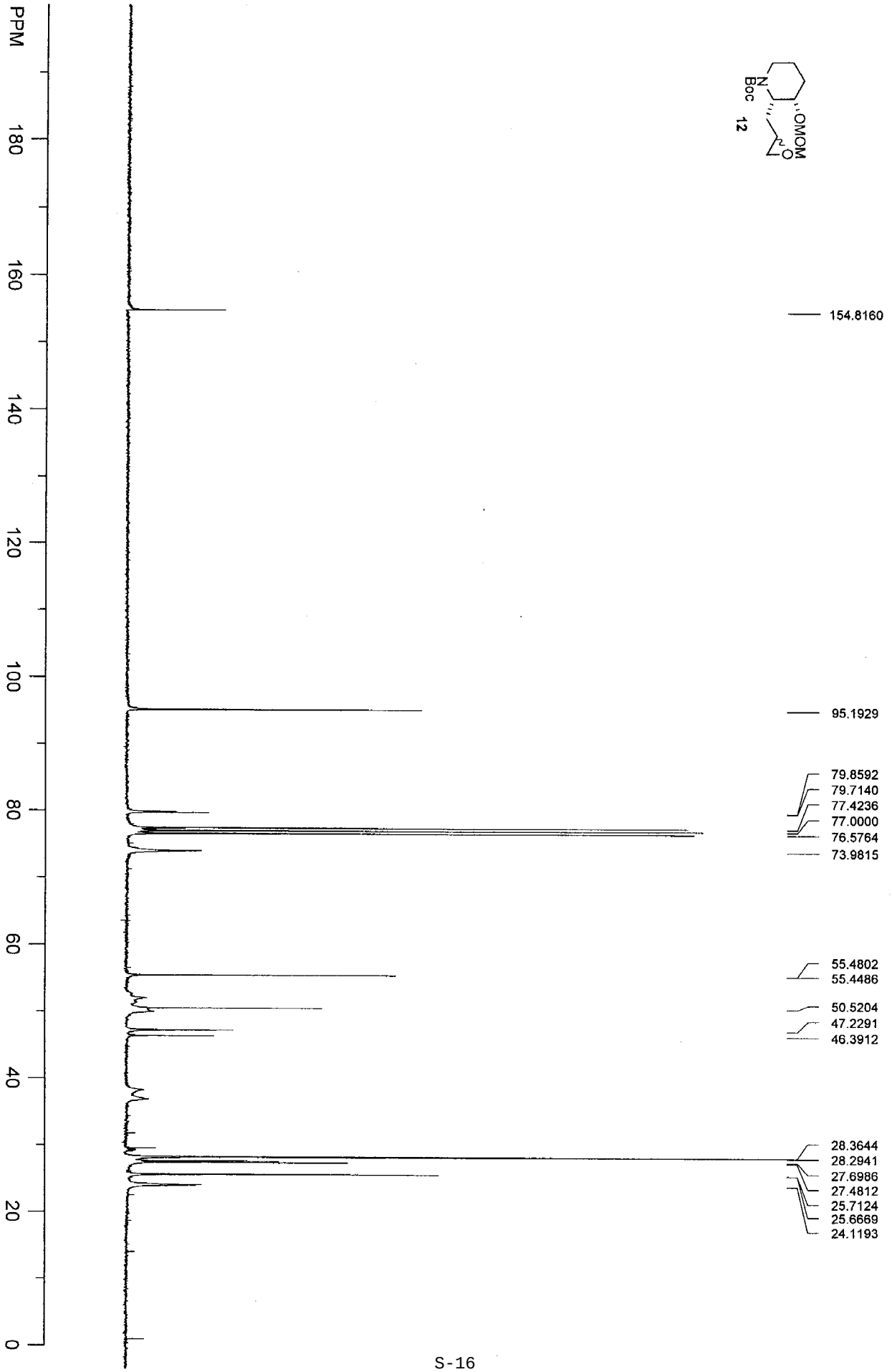
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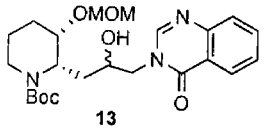
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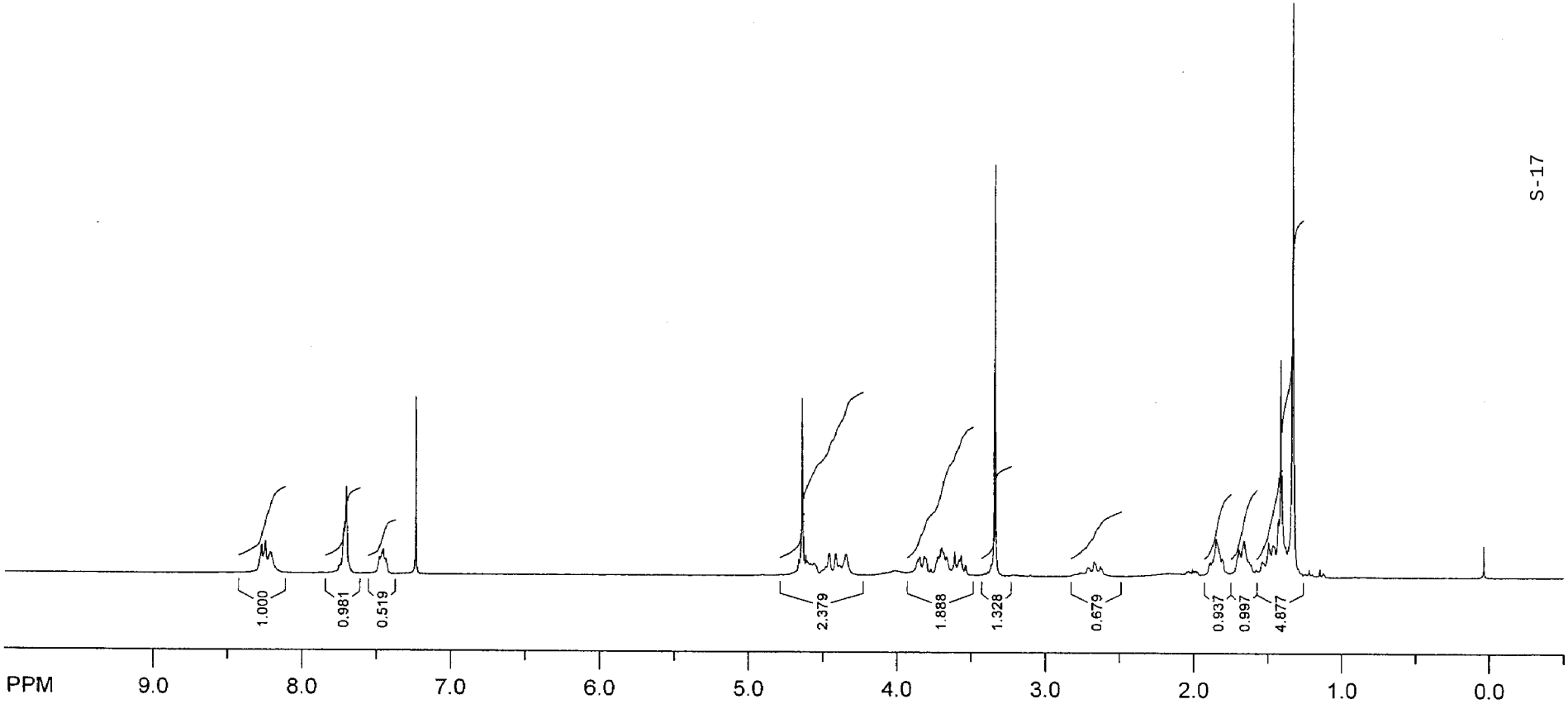
Boc
O

13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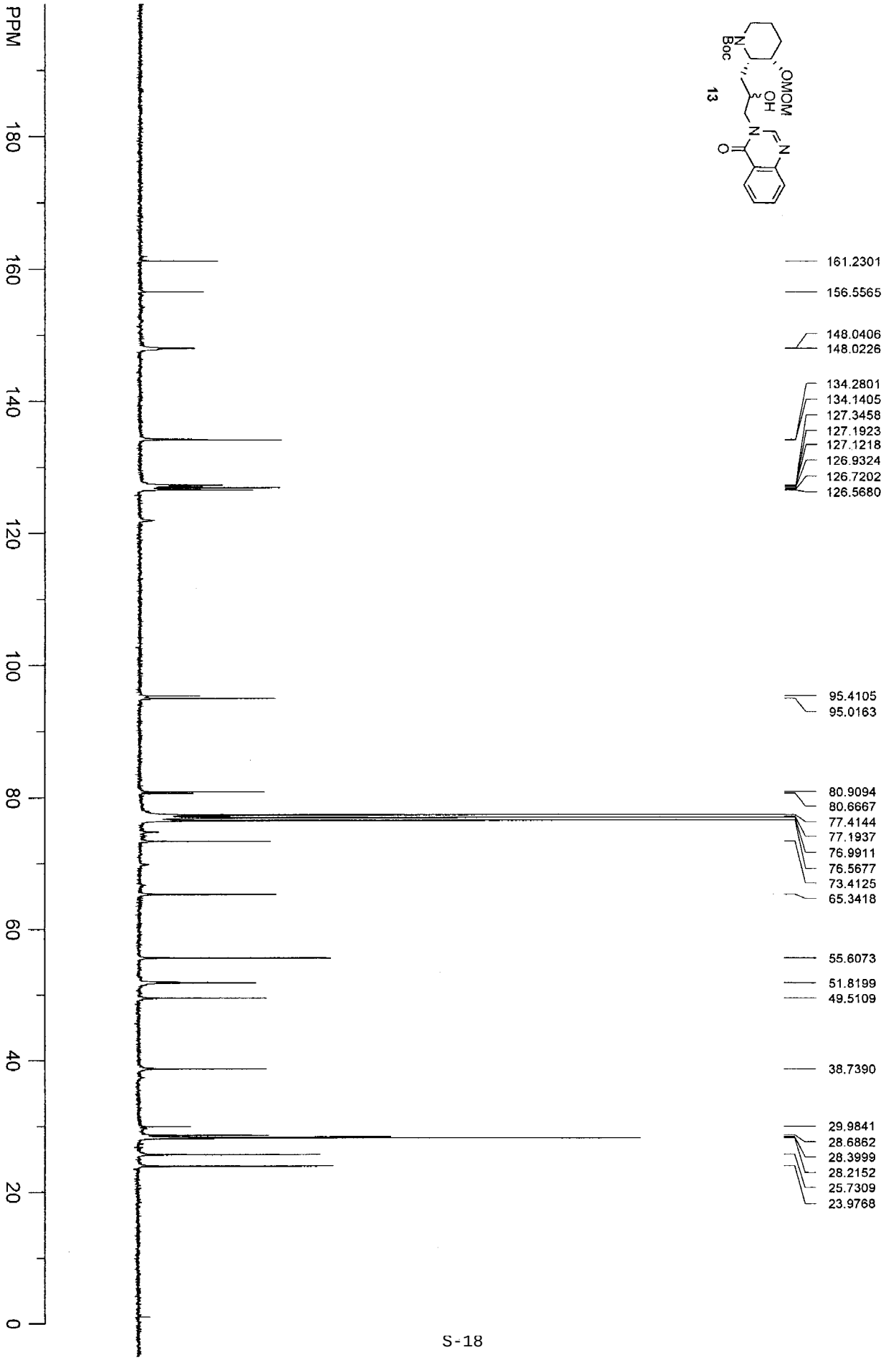
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Boc
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OMOM
OH
N
N
O
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80.9094
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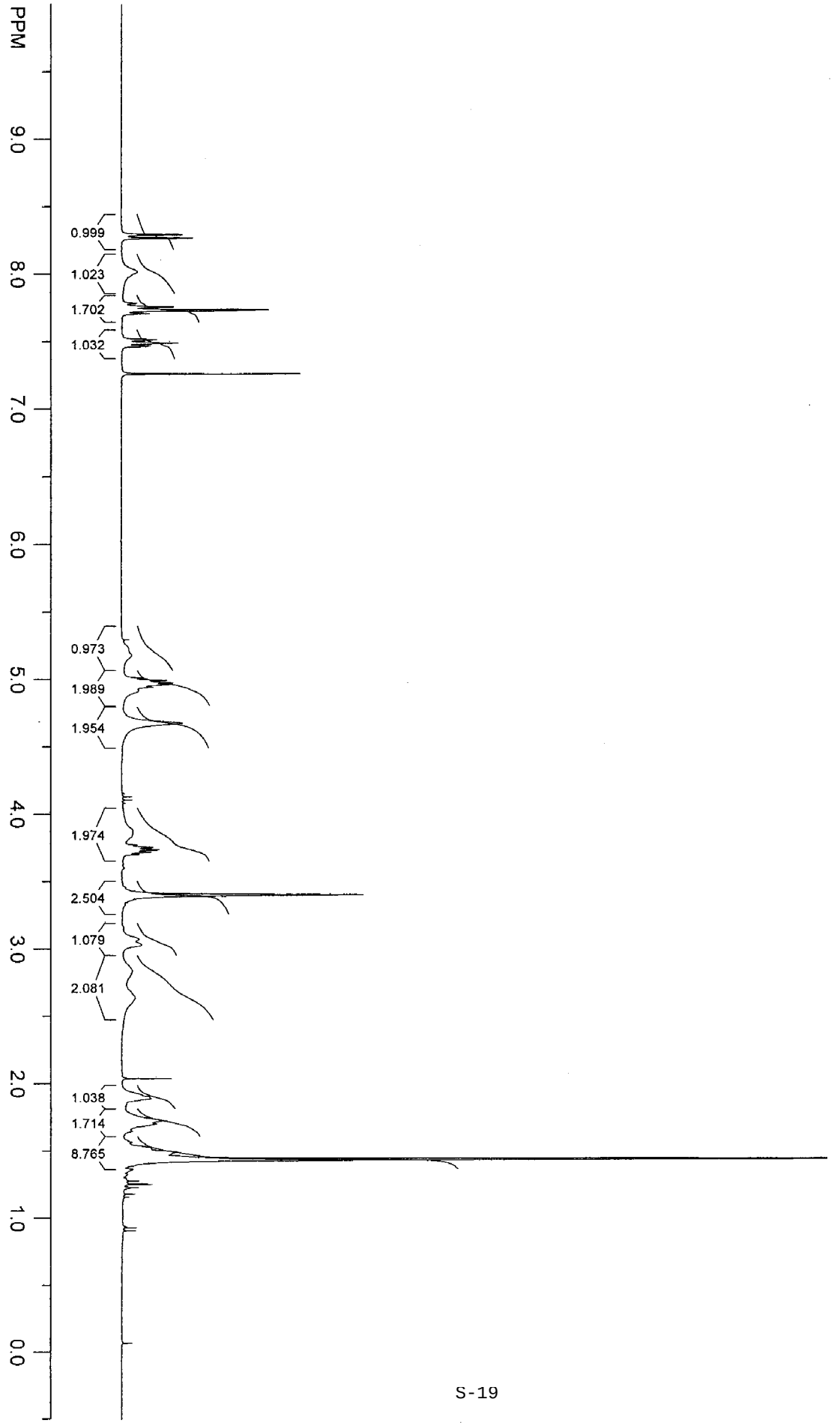
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1.0
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1.702
1.032
0.973
1.989
1.954
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2.081
1.038
1.714
8.765
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Boc
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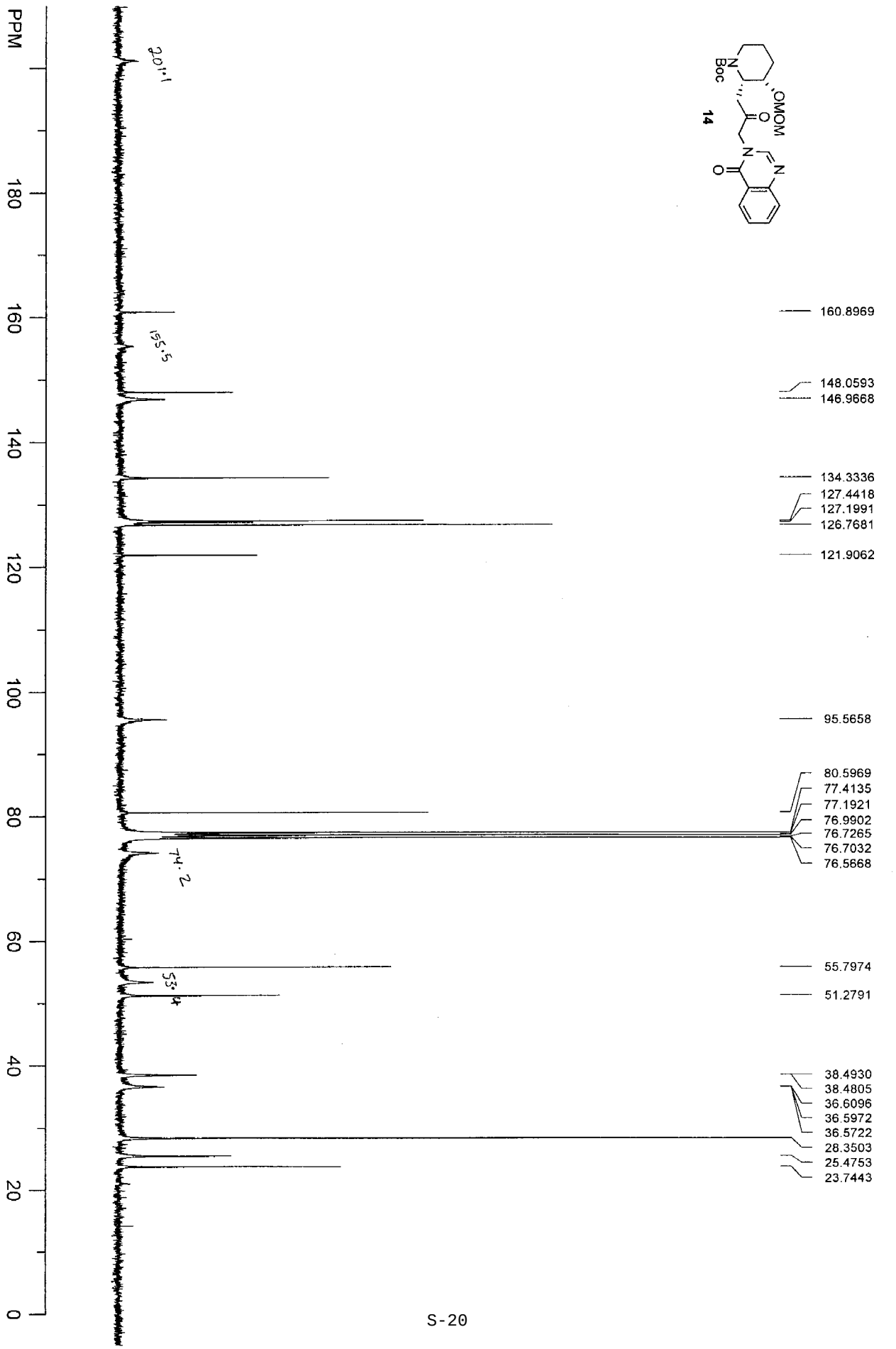
O
N
N
O
14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100
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140
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180
201.1
155.5
74.2
53.4
14
Boc
OMOM
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146.9668
134.3336
127.4418
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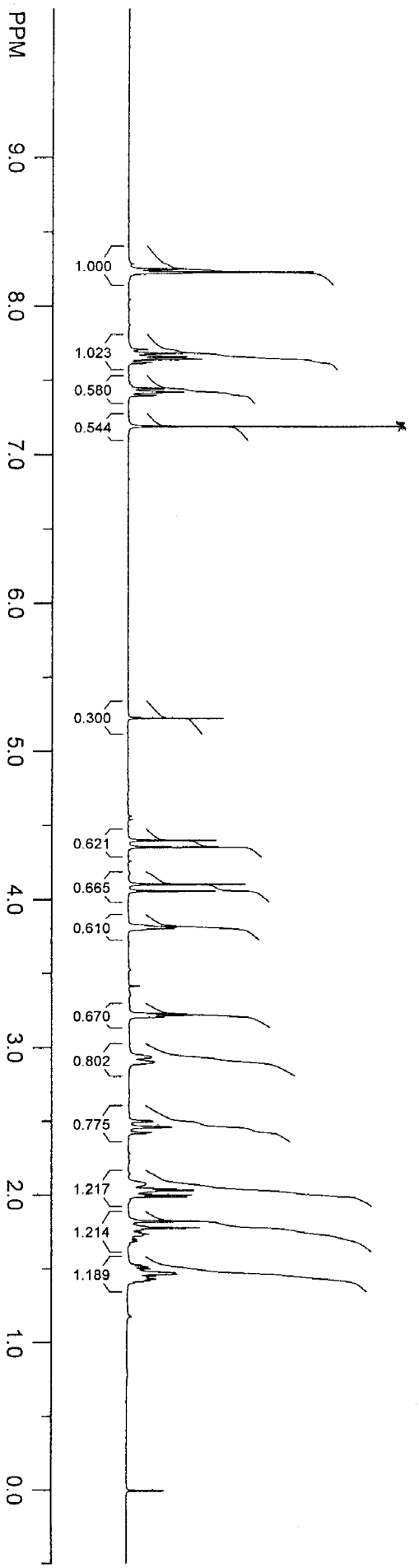
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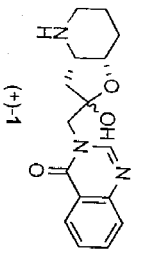
1.214
1.189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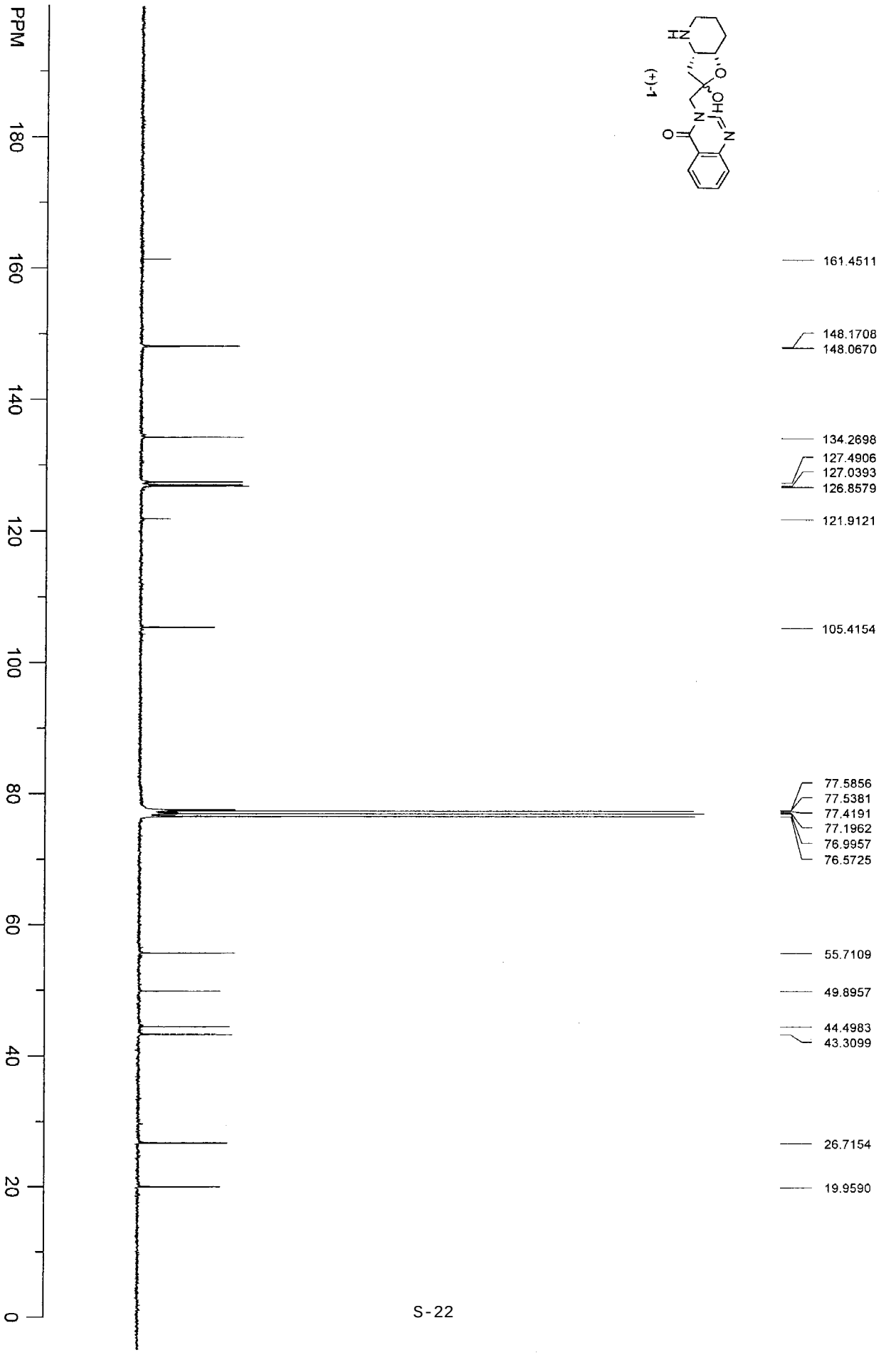
H
N
O
OH
N
N
O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40
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(+)-1
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148.0670
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127.4906
127.0393
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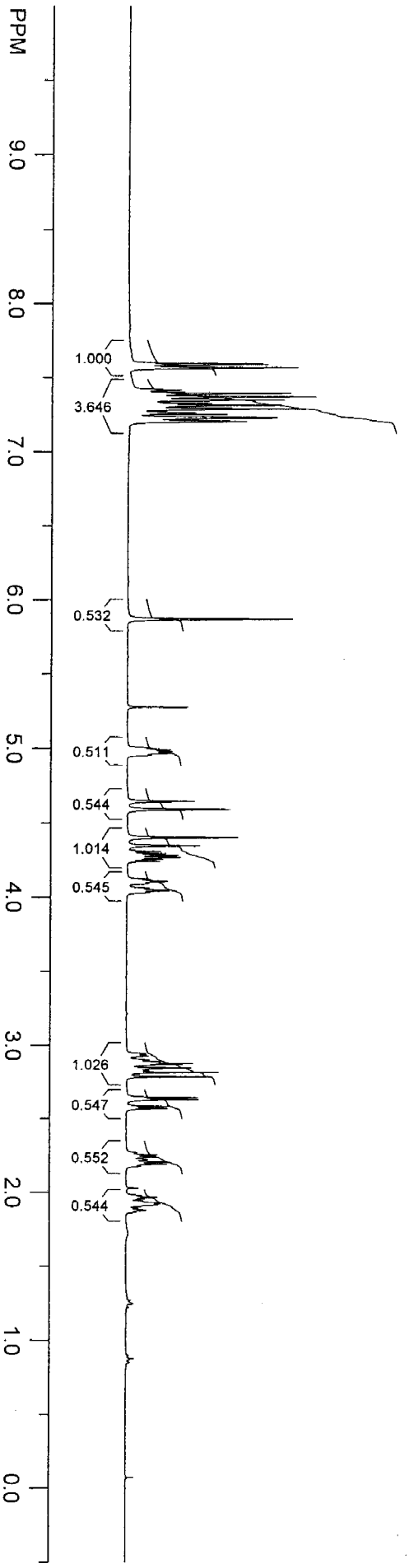
26.7154
19.9590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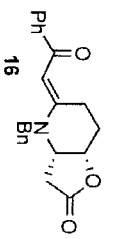
0.545
1.026
0.547
0.552
0.544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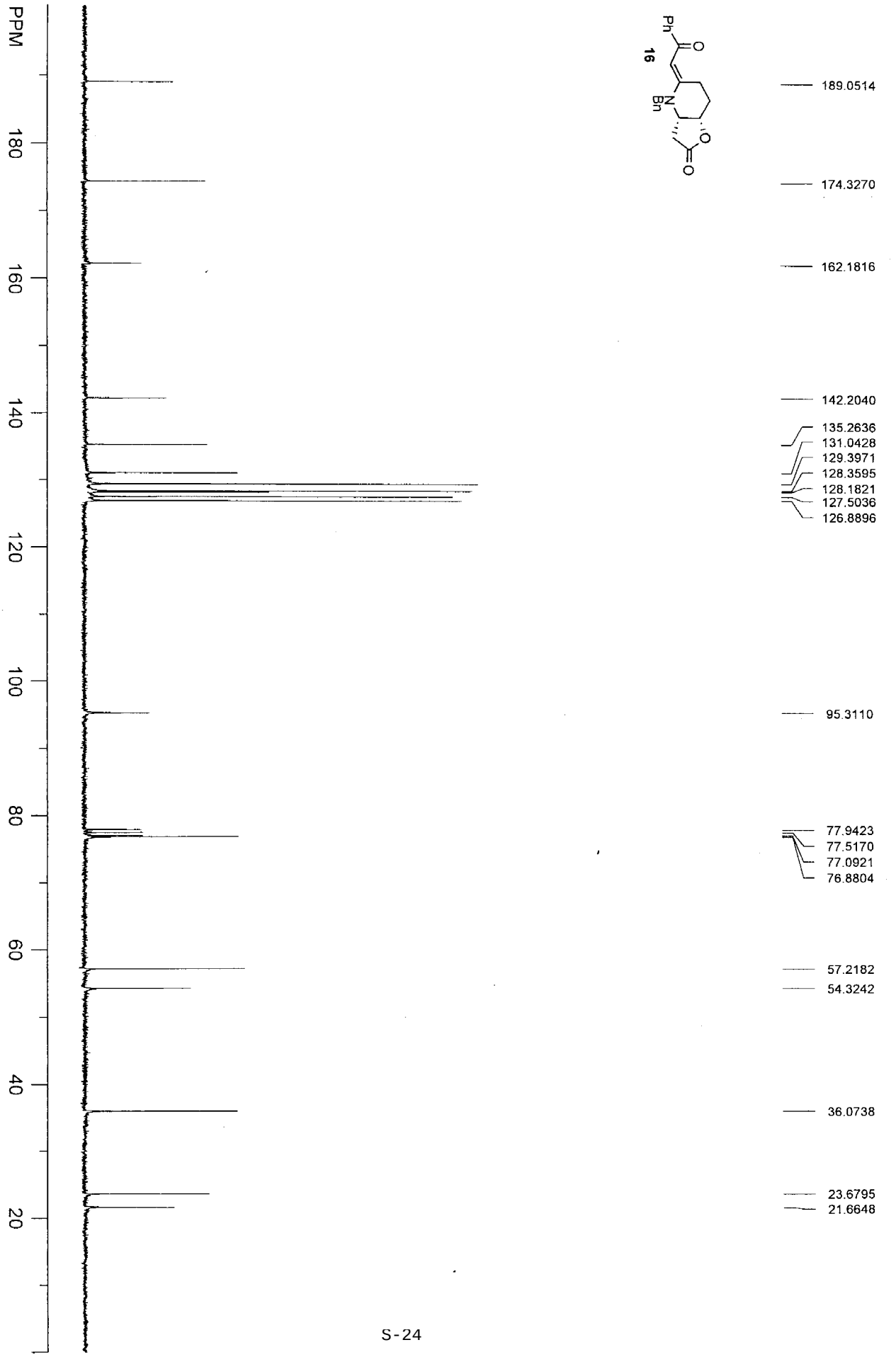
Bn
O
O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40
20
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16
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Bn
O
O
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131.0428
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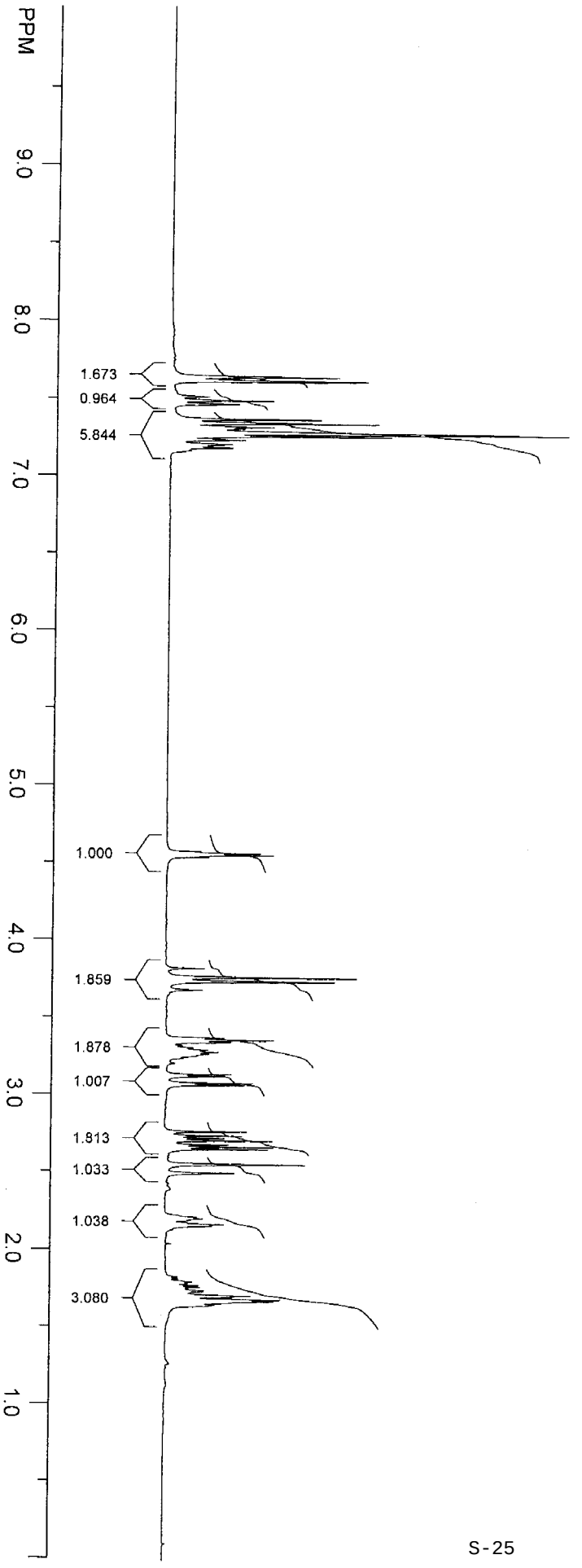
23.6795
21.6648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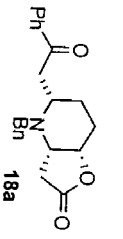
3.080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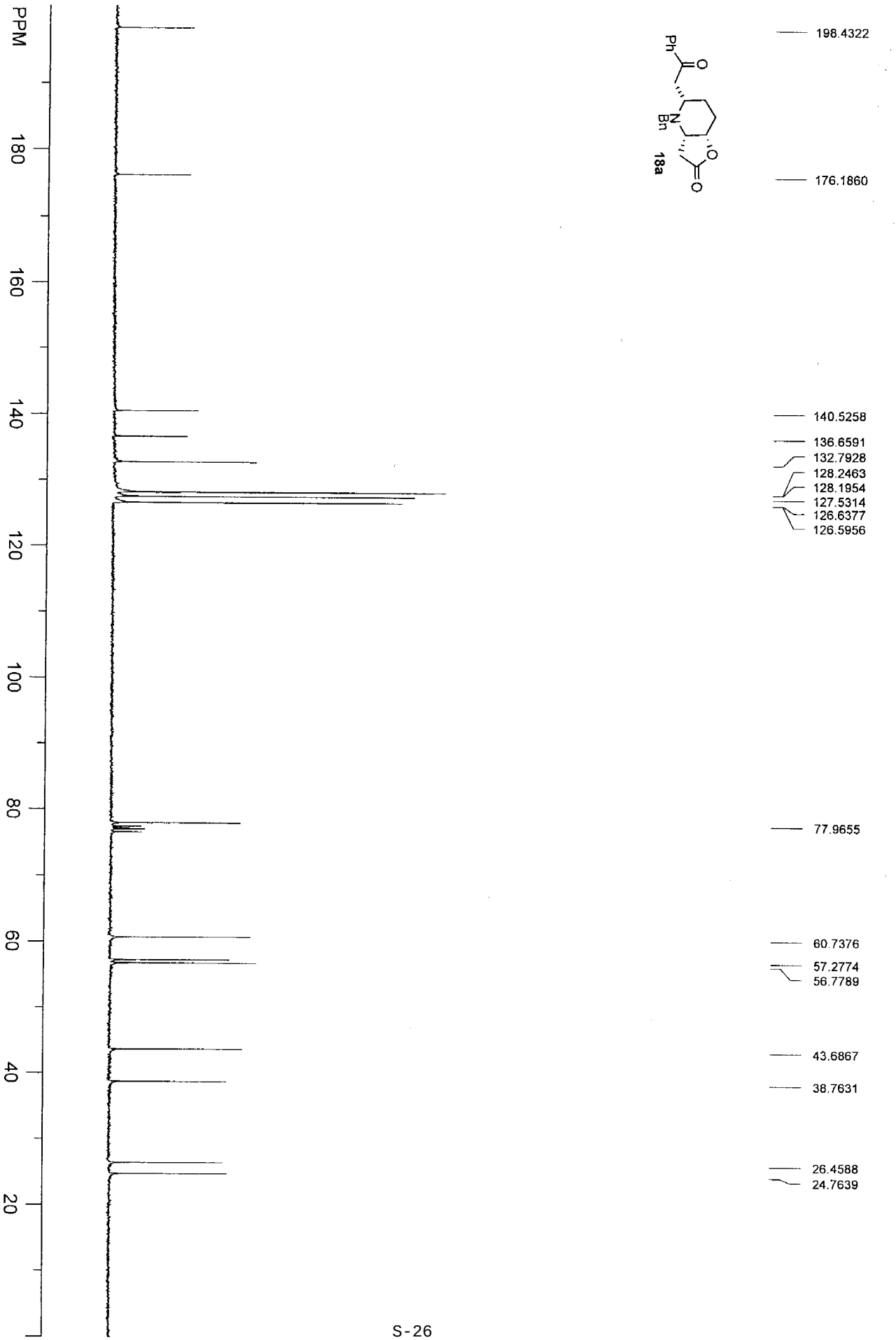
Bn
O
O
O

18a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120
100
80
60
40
20
Ph
O
N
Bn
O
O
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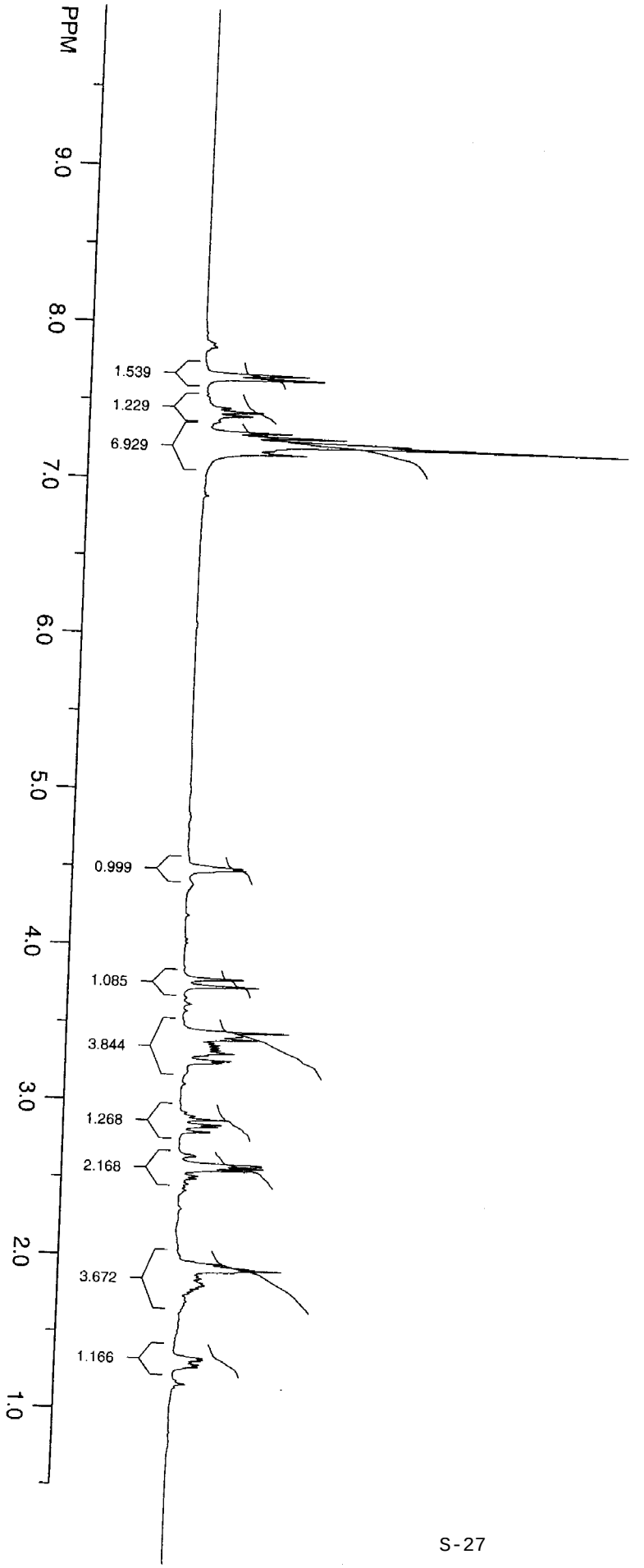
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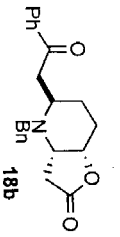
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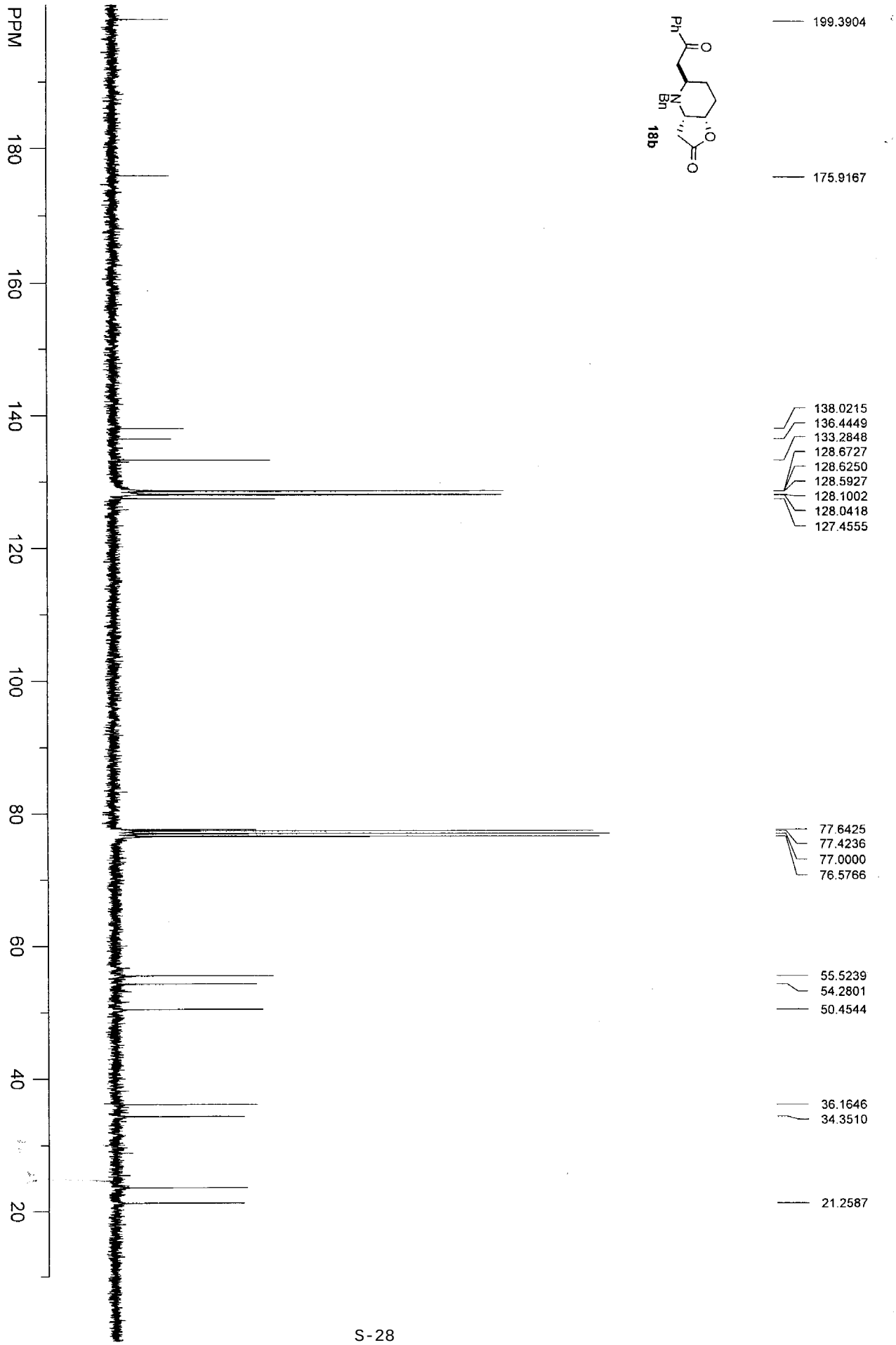
N
Bn
O
O

18b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40
20
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O
N
Bn
O
O
18b
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175.9167
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136.4449
133.2848
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128.6250
128.5927
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128.0418
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54.2801
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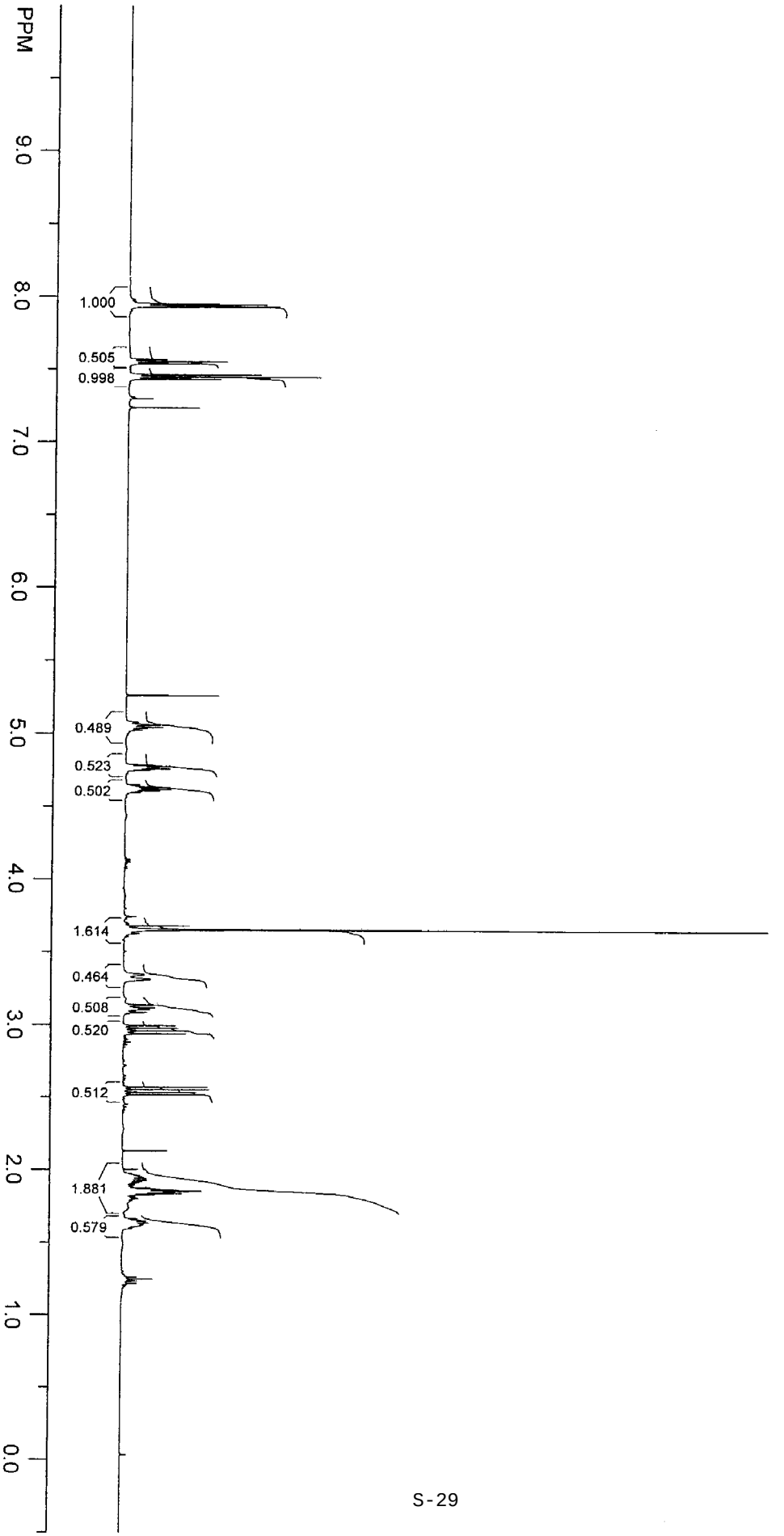
21.2587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0.512
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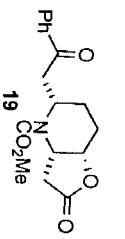
0.579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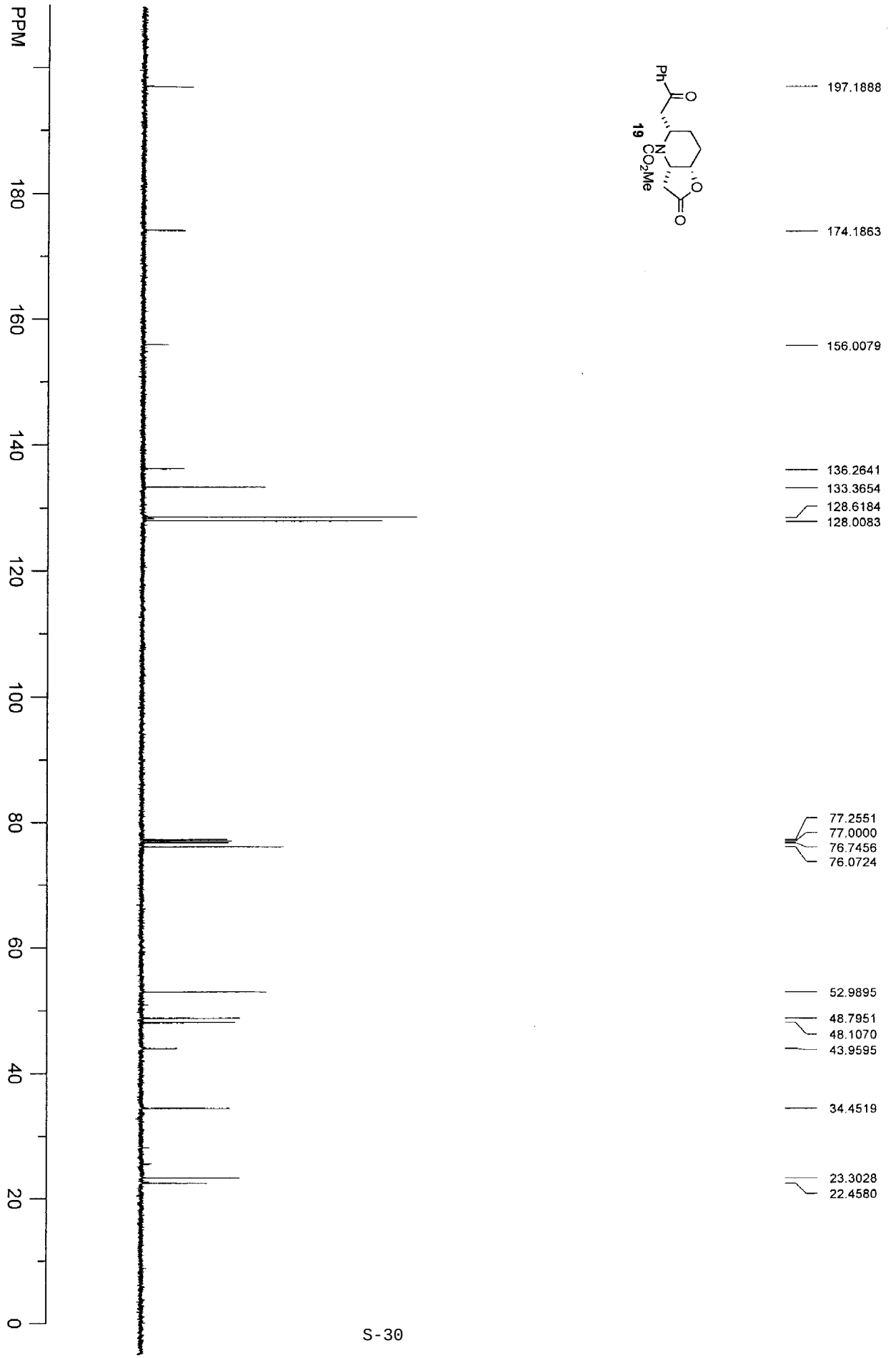
O
N
CO2Me
O
O

19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100
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160
180
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O
19
N
CO2Me
O
O
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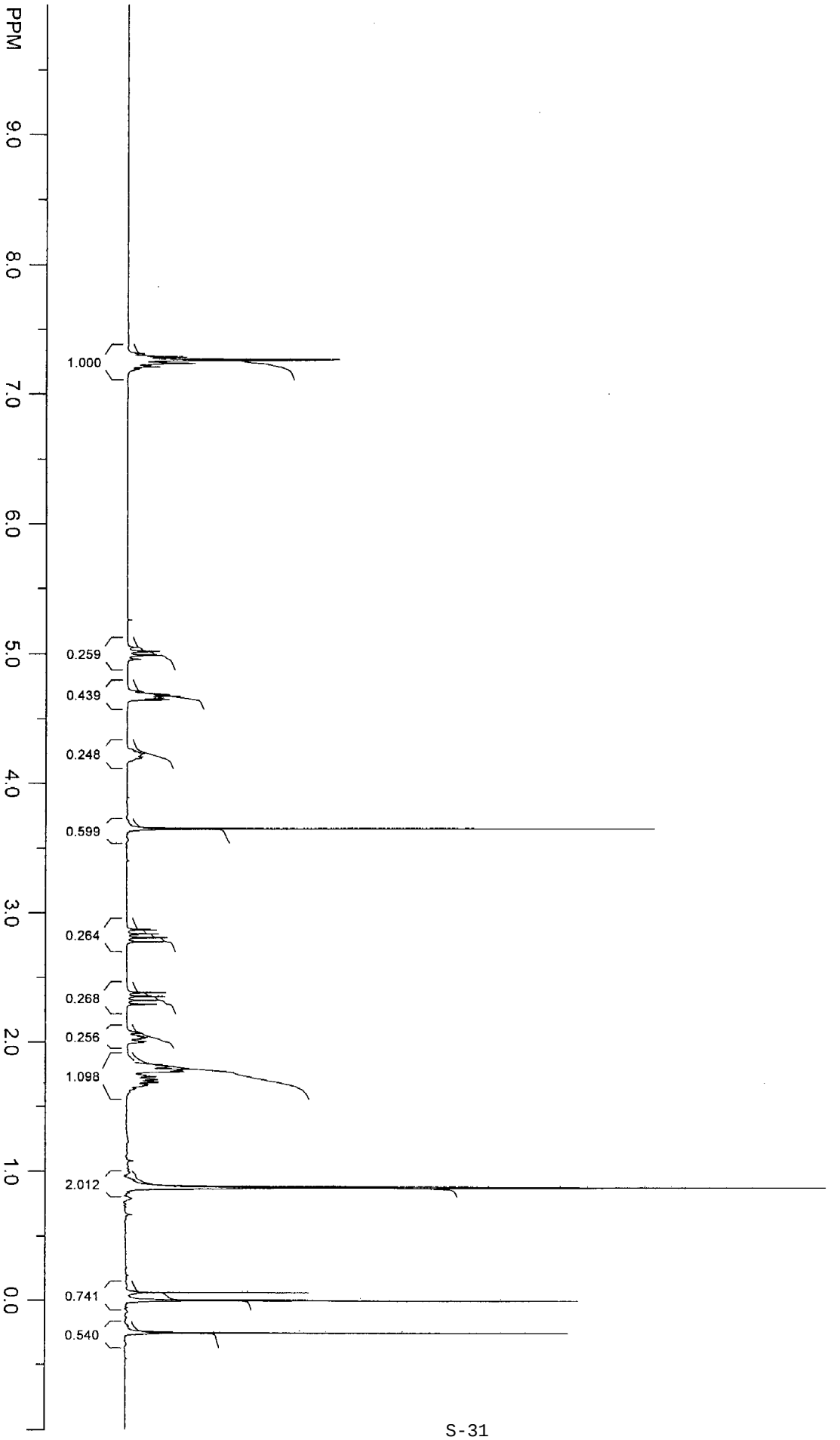
23.3028
22.4580


PPM
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0.599
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TBSO
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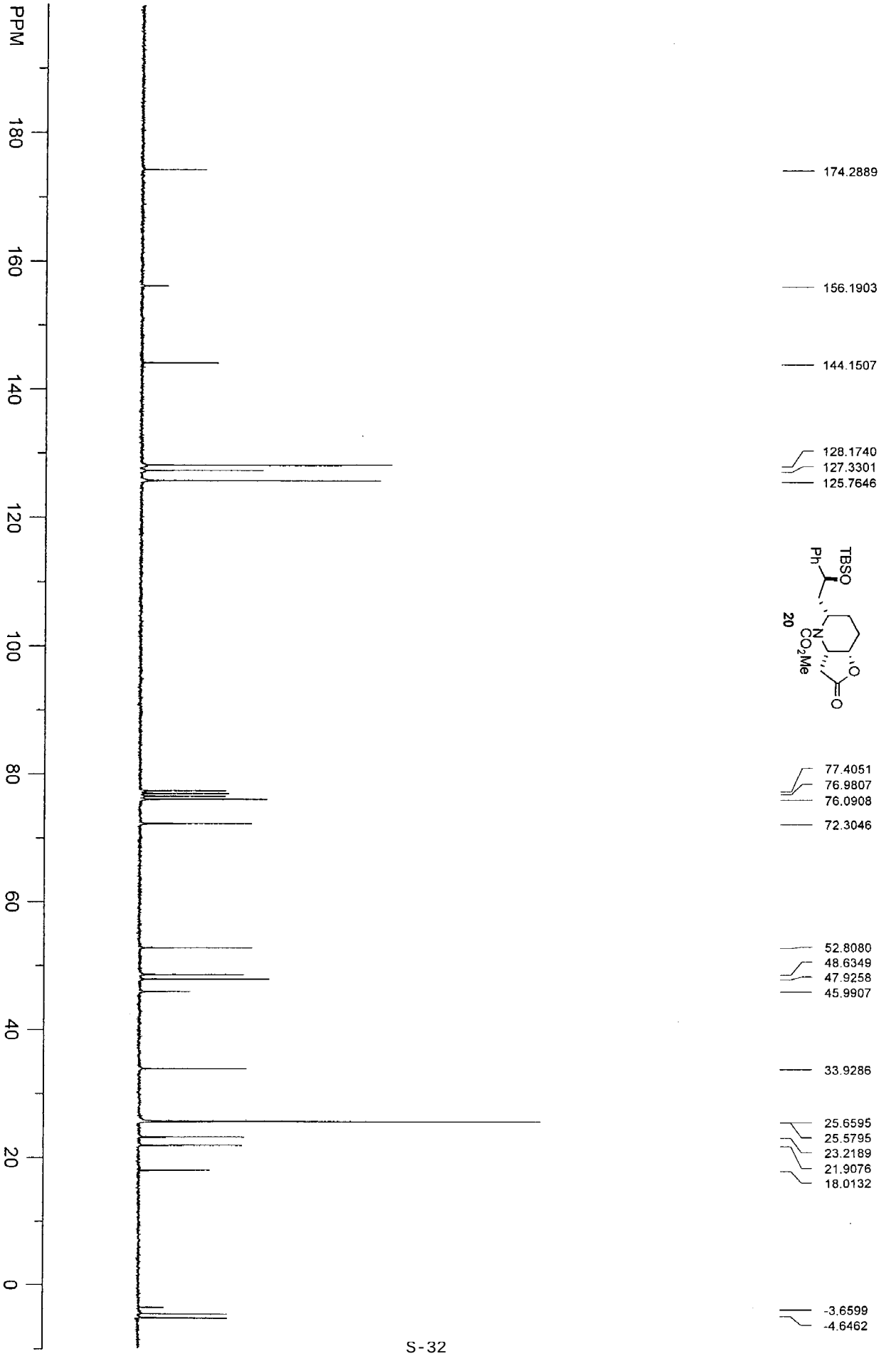
CO2Me
O
O
20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O
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21.9076
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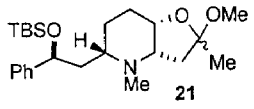
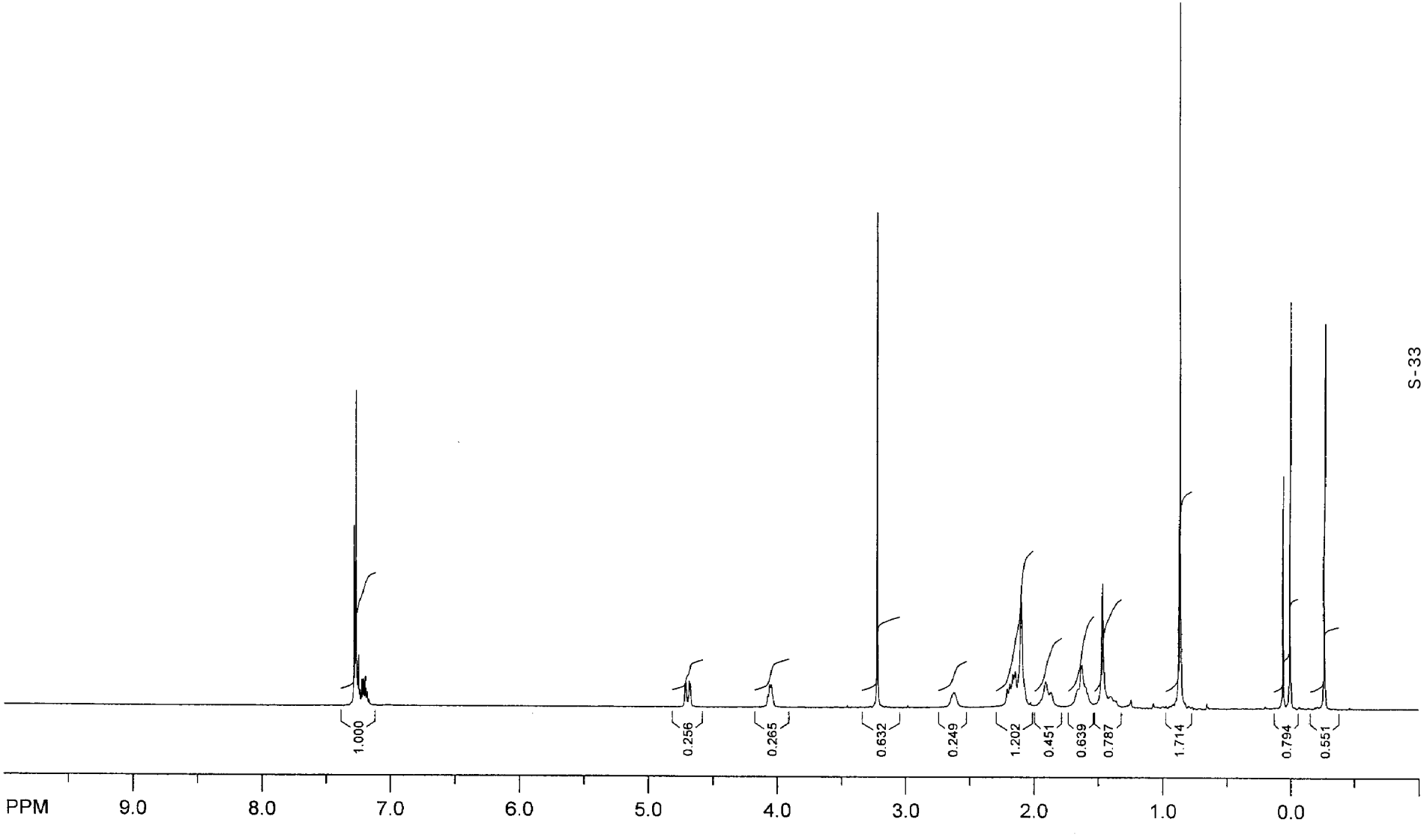
-4.6462


TBSO
H
Ph
N
Me
O
OMe
Me

21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1.0
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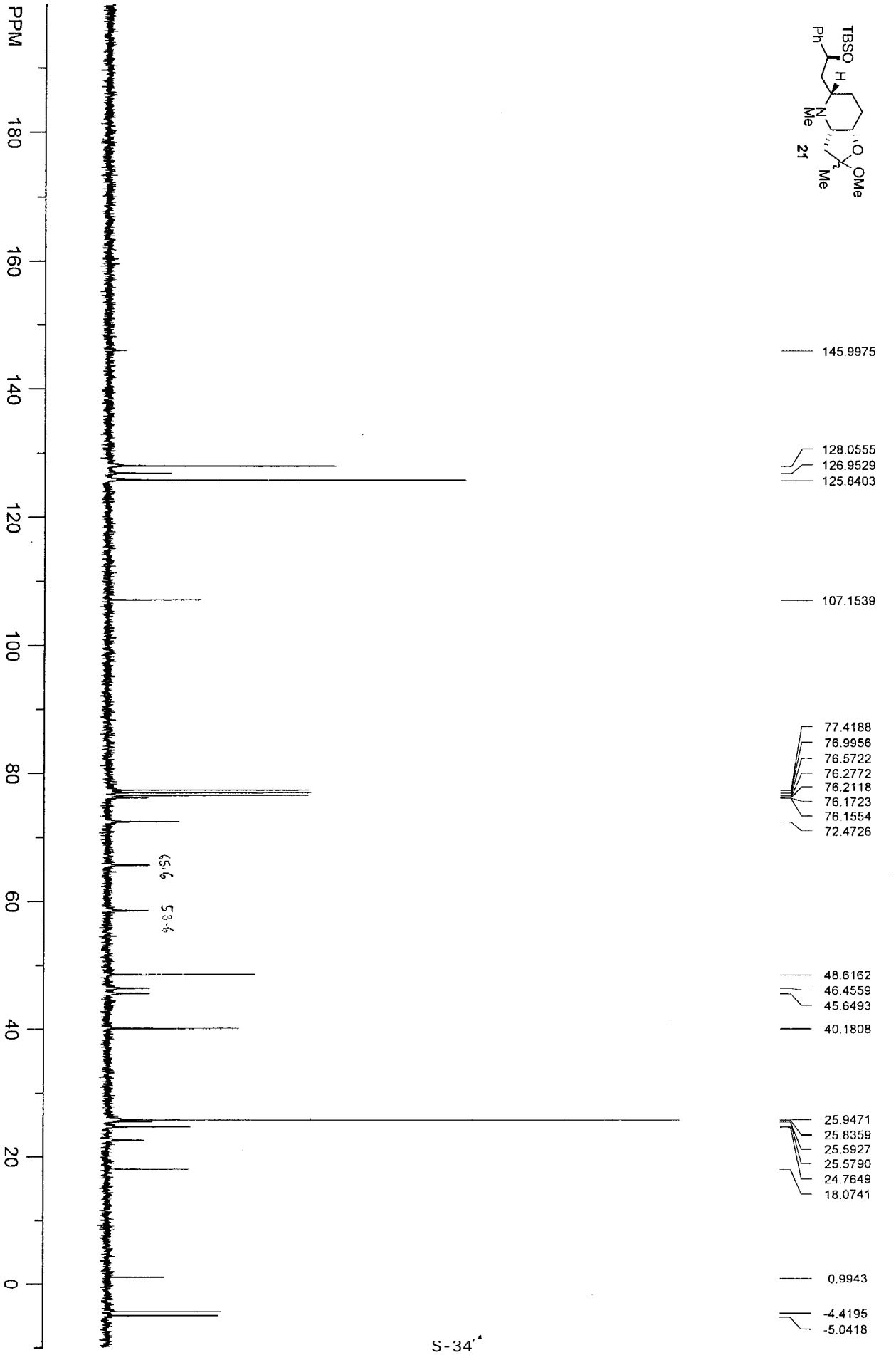
PPM
180
160
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120
100
80
60
40
20
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TBSO
Ph
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N
Me
O
OMe
Me
21
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125.8403
107.1539
77.4188
76.9956
76.5722
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76.2118
76.1723
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72.4726
65.6
58.4
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46.4559
45.6493
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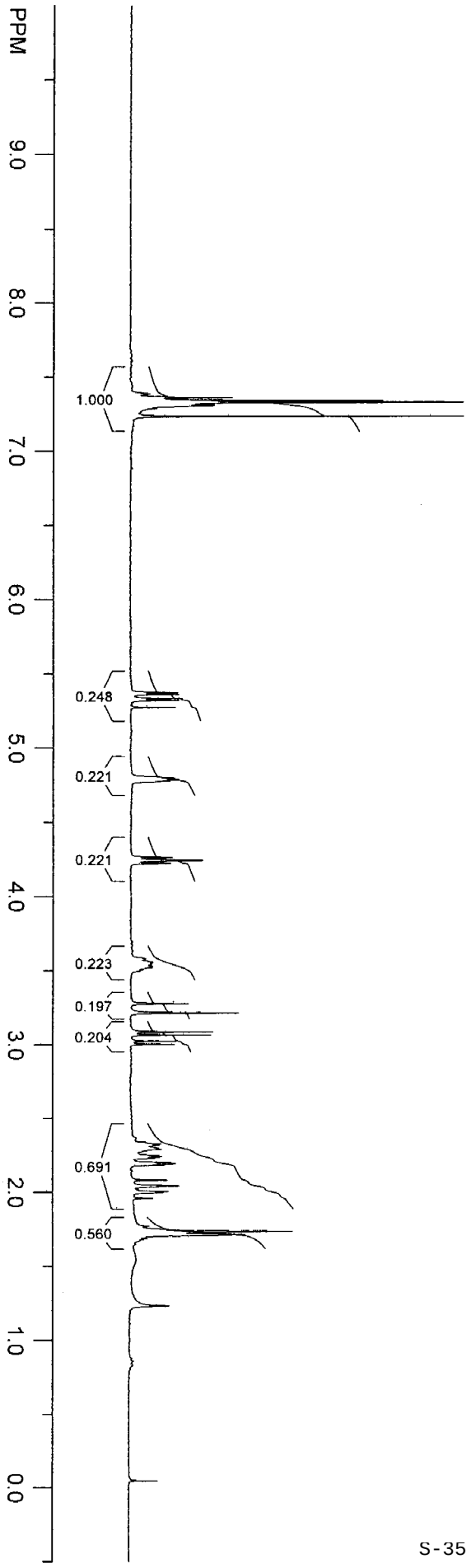
PPM
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6.0
5.0
4.0
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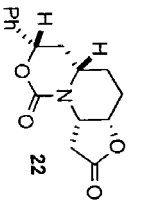
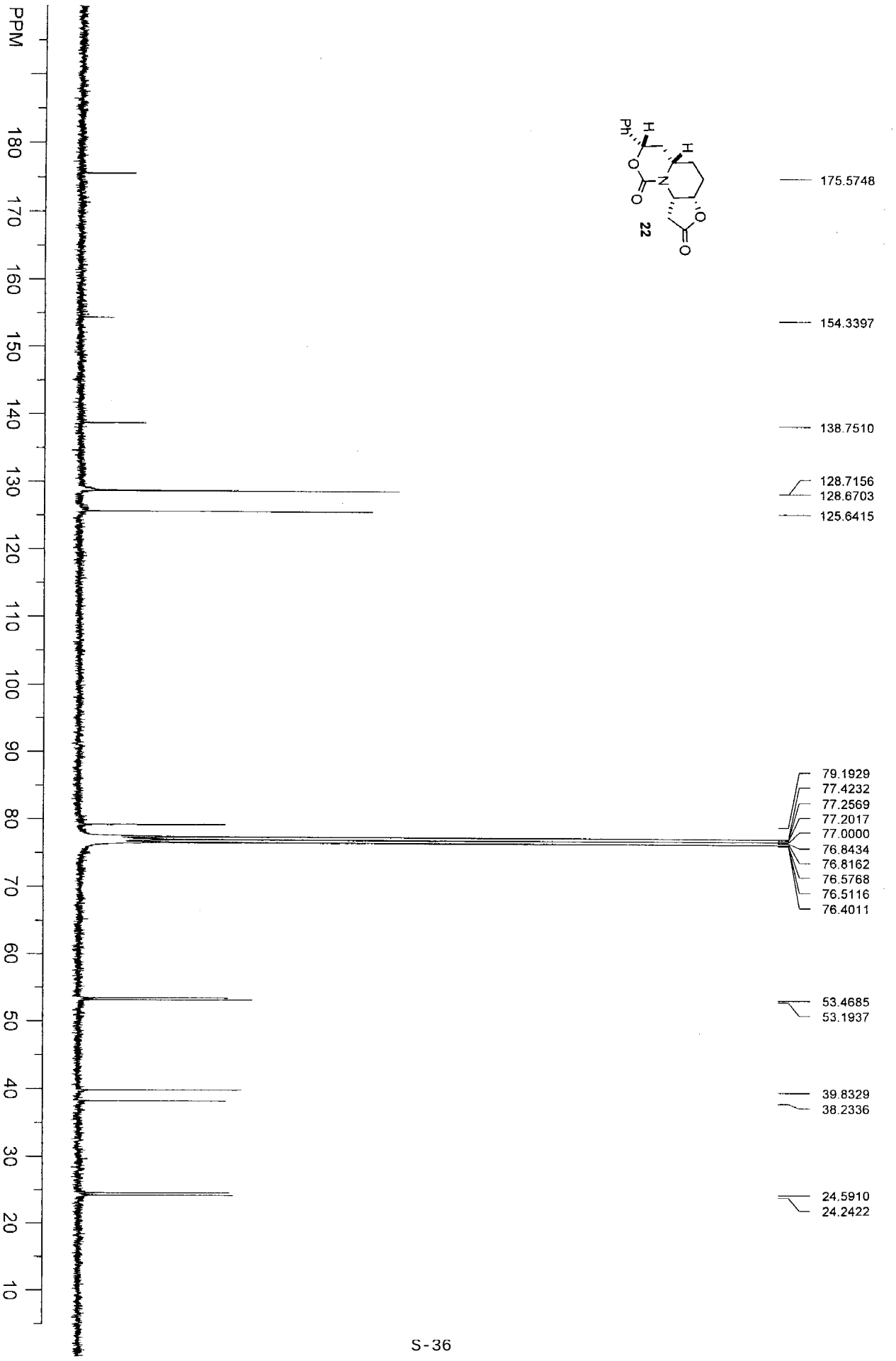
0.221
0.223
0.197
0.204
0.691
0.560


H
Ph
O
O
N
H
O
O
O

22


PPM
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
Ph
H
H
O
N
O
O
O
22
175.5748
154.3397
138.7510
128.7156
128.6703
125.6415
79.1929
77.4232
77.2569
77.2017
77.0000
76.8434
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76.5116
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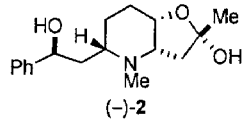
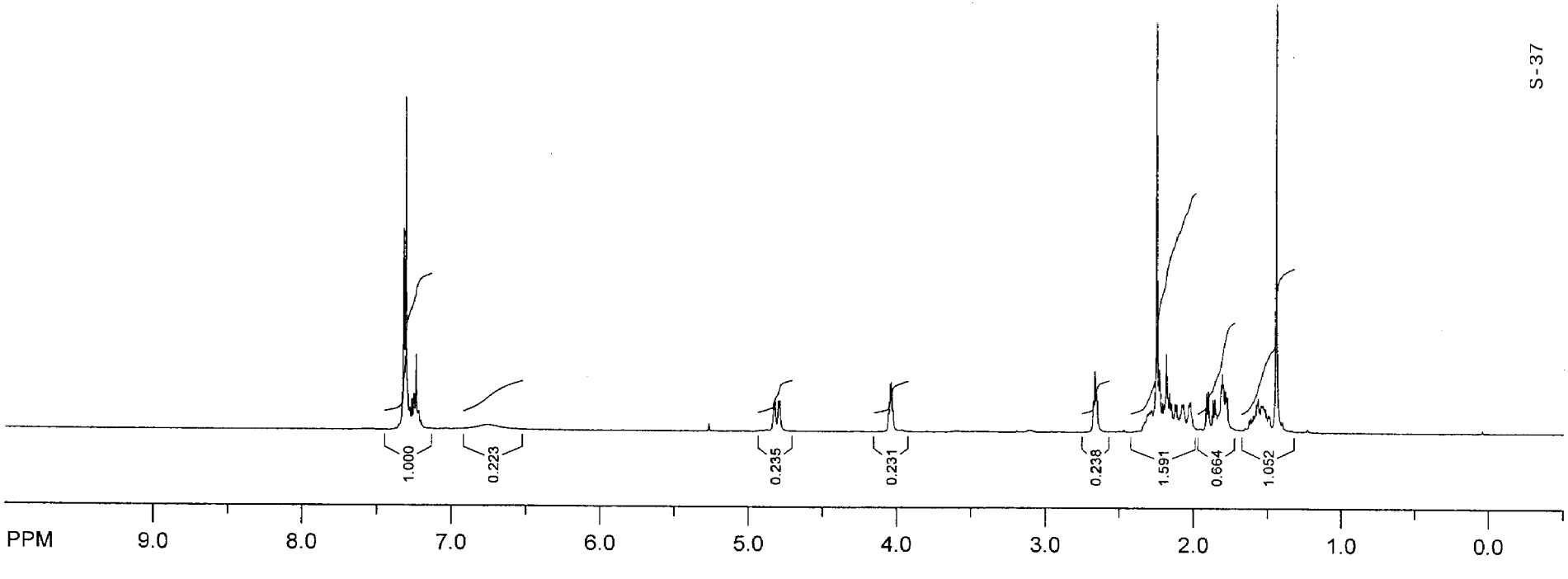
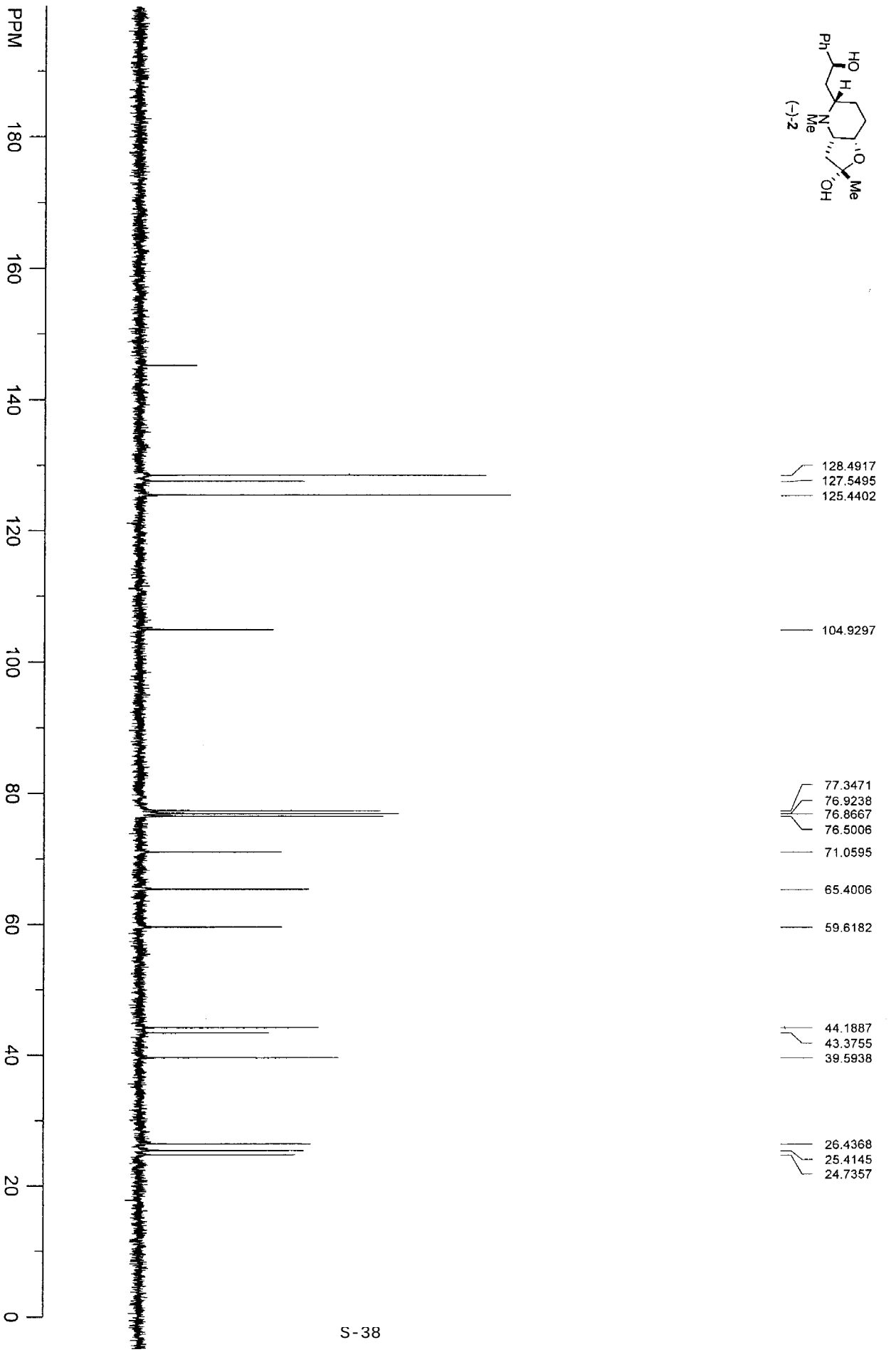
53.4685
53.1937
39.8329
38.2336
24.5910
24.2422


HO
H
Ph
N
Me
O
Me
OH

(–)-**2**


1.000
0.223
0.235
0.231
0.238
1.591
0.664
1.052
PPM
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0


PPM
180
160
140
120
100
80
60
40
20
0
Ph
HO
H
N
Me
(-)-2
O
Me
OH
128.4917
127.5495
125.4402
104.9297
77.3471
76.9238
76.8667
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65.4006
59.6182
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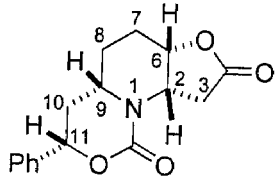
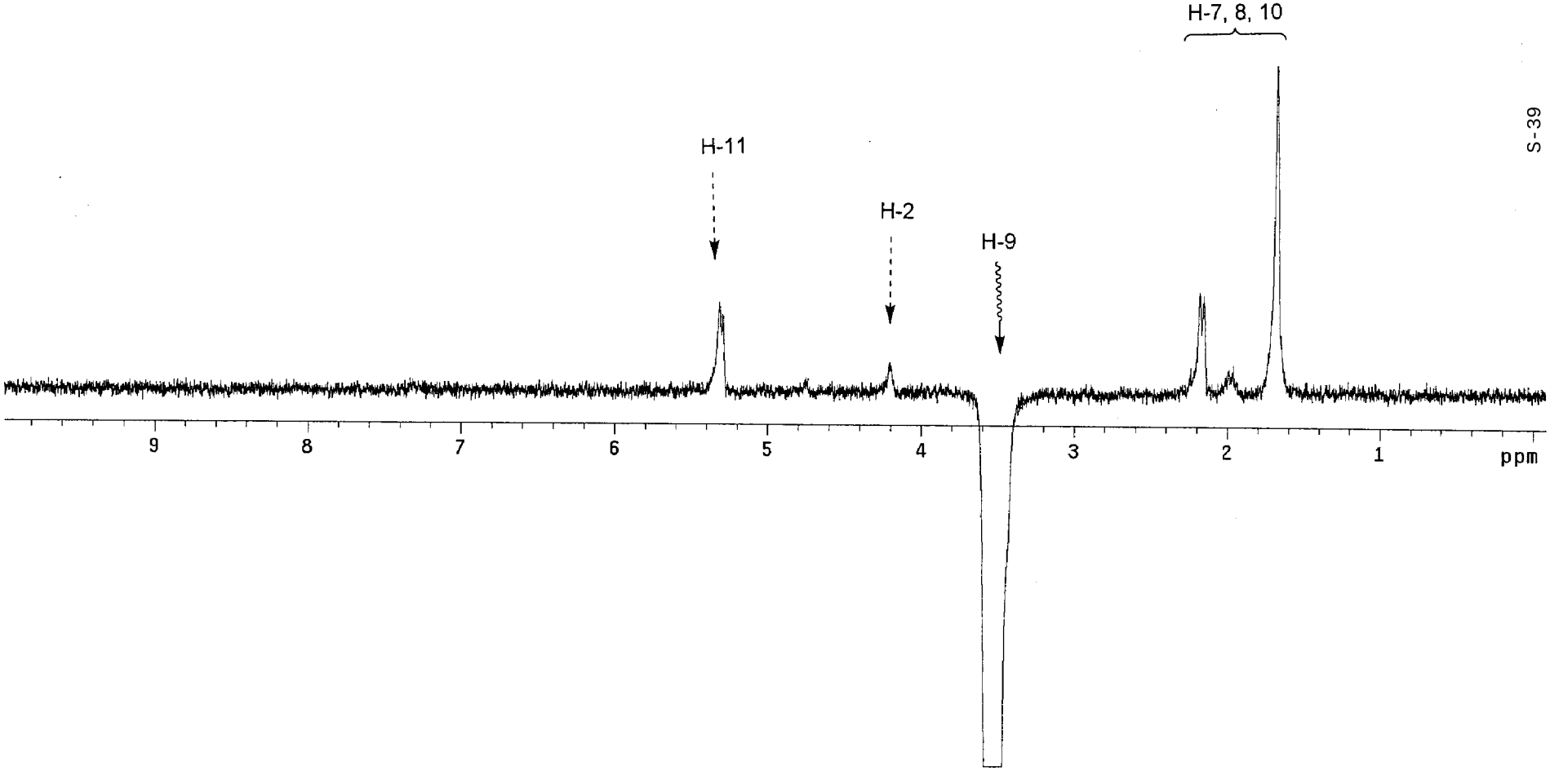
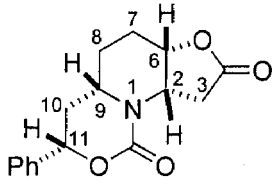
25.4145
24.7357

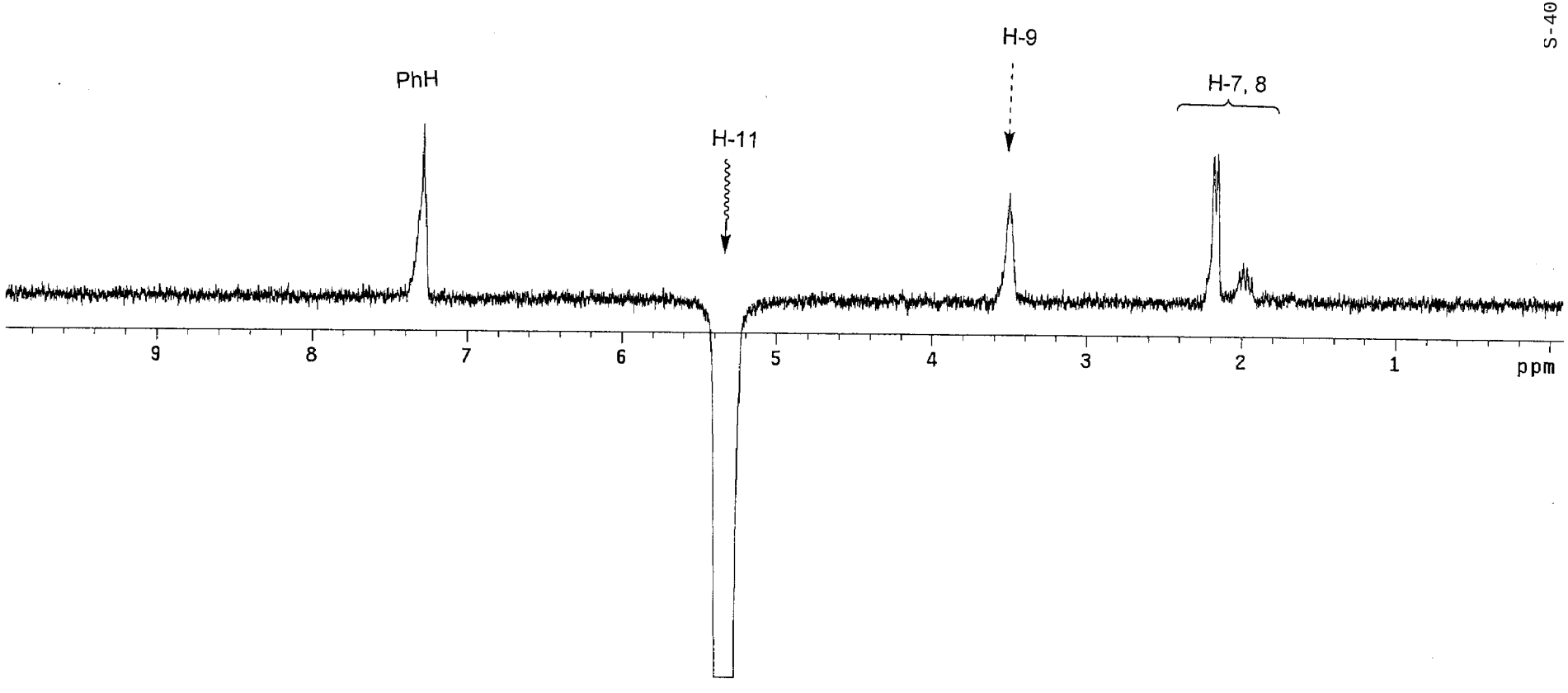


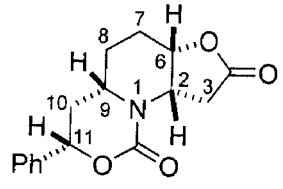
22 (numbering is arbitary)
1D NOESY Experiment





22 (numbering is arbitary)
1D NOESY Experiment





22 (numbering is arbitary)
1D NOESY Experiment