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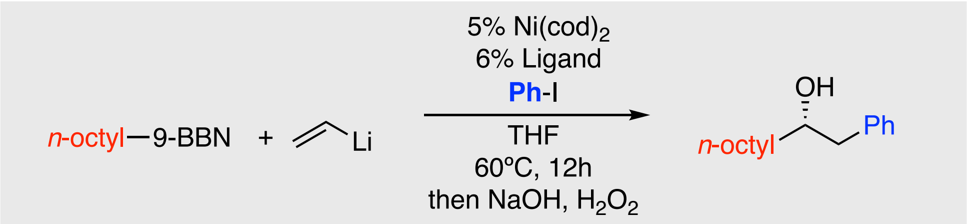
General Information

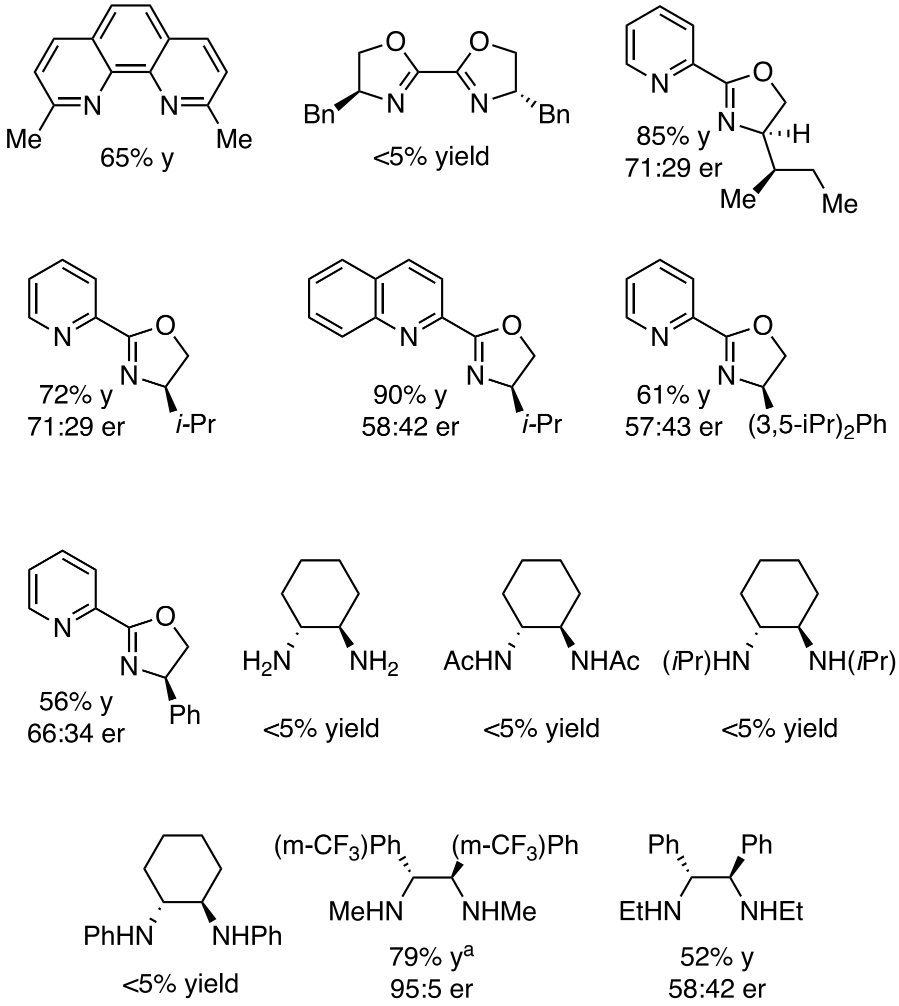
^{1}H NMR spectra were recorded on either a Varian Gemini-500 (500 MHz), Varian Gemini-600 (600 MHz), or Varian Inova-500 (500 MHz) spectrometer. Chemical shifts are reported in ppm with the solvent resonance as the internal standard ($CDCl_3$: 7.24 ppm). Data are reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, m = multiplet), and coupling constants (Hz). ^{13}C NMR spectra were recorded on either a Varian Gemini-500 (125 MHz), Varian Gemini-600 (150 MHz) or a Varian Inova-500 (125 MHz) spectrometer with complete proton decoupling. Chemical shifts are reported in ppm with the solvent resonance as the internal standard ($CDCl_3$: 77.16 ppm). Chemical shifts are reported in ppm using phosphoric acid as the external standard (H_3PO_4: 0.0 ppm). ^{11}B NMR spectra were recorded on a Varian Gemini-500 (128 MHz) spectrometer. ^{19}F NMR spectra were recorded on a Varian Gemini-500 (470 MHz) spectrometer. Infrared (IR) spectra were recorded on a Bruker alpha-P Spectrometer. Frequencies are reported in wavenumbers (cm^{-1}) as follows: strong (s), broad (br), medium (m), and weak (w). Optical rotations were measured on a Rudolph Analytical Research Autopol IV Polarimeter. High-resolution mass spectrometry (DART+) was performed at the Mass Spectrometry Facility, Boston College, Chestnut Hill, MA. Liquid chromatography was performed using forced flow (flash chromatography) on silica gel (SiO_2, 230 x 450 Mesh) purchased from Silicycle. Thin layer chromatography (TLC) was performed on 25 μm silica gel glass backed plates from Silicycle. Visualization was performed using ultraviolet light (254 nm), ceric ammonium molybdate (CAM) in ethanol or phosphomolybdic acid, and cerium(IV) sulfate in ethanol with sulfuric acid (Seebach).

Analytical chiral supercritical fluid chromatography (SFC) was performed on a TharSFC Method Station II equipped with Waters 2998 Photodiode Array Detector with isopropanol as the modifier.

All reactions were conducted in oven- or flame-dried glassware under an inert atmosphere of nitrogen or argon. Tetrahydrofuran (THF), diethyl ether (Et_2O), dichloromethane (DCM) and toluene were purified using Pure Solv MD-4 solvent purification system, from Innovative Technology, Inc., by passing the solvent through two activated alumina columns after purging with argon. Nickel(II) acetylacetonate was purchased from Acros Organics, (1*R*,2*R*)-*N*,*N*'-Dimethyl-1,2-diphenylethane-1,2-diamine (***(R,R)*-L6**) was purchased from Astatech Inc., and 9-Borabicyclo[3.3.1]nonane 0.5M solution in THF was purchased from Alfa Aesar (of note, cross coupling reactions resulted in slightly diminished yields when a BBN solution from Sigma Aldrich was employed, or when borane reagents were prepared from BBN dimer). All other reagents were purchased from Sigma Aldrich, Alfa Aesar, Oakwood Chemicals, Combi-Blocks, or Acros Organics and used without further purification.

Expanded Survey of Ligands





Conditions: reactions were run according to **procedure A** with 9-BBN in THF (0.5 M, 0.22 mmol, 1.1 equiv.), 1-octene (0.22 mmol, 1.1 equiv.), halide-free vinyllithium in THF (0.20 mmol, 1.0 equiv.), a solution of [Ni] (0.010 mmol, 0.050 equiv.) and ligand (0.012 mmol, 0.060 equiv.) in THF (0.4 mL), and iodobenzene (0.20 mmol, 1.00 equiv.). a: $Ni(acac)_2$ used instead of $Ni(cod)_2$.

Survey of Nickel Sources

An evaluation of Ni salts showed a slight increase in yield when using $Ni(acac)_2$, presumably because the latter is more efficient at pre-complexing to the diamine ligand (a solution of $Ni(cod)_2$ and **L6** in THF-d_8 was monitored by ^{1}H NMR, and no change in the starting materials was observed over several hours) Using a slight excess of boron 'ate' to help reduce the Ni(II) pre-catalyst (making the electrophile the limiting reagent) resulted in a further increase in yield. Results from the studies for the reaction of n-octyl-BBN, vinyl-Li, and iodobenzene are summarized in the table below (**SI-table 2**).

SI-table 2

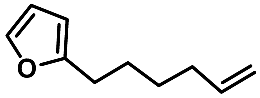
Ni source	**Ligand**	**Yield**	**e.r.**
Ni(COD)2	**L5**	60%	93:07
Ni(COD)2	**L6**	62%	95:05
$Ni(acac)_2$	**L6**	71%	95:05
Ni(Br)2 (glyme)	**L6**	58%	95:05
Ni(I)2	**L6**	<5%	N/A
NiCO3	**L6**	<5%	N/A
$Ni(acac)_2$*	**L6**	80%	95:05

*1.1 equiv. of vinyl-Li and 1.2 of octyl-BBN

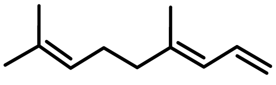
Conditions: reactions were run according to **procedure A** with 9-BBN in THF (0.5 M, 0.22 mmol, 1.1 equiv.), 1-octene (0.22 mmol, 1.1 equiv.), halide-free vinyllithium in THF (0.20 mmol, 1.0 equiv.), a solution of [Ni] (0.010 mmol, 0.050 equiv.) and ligand (0.012 mmol, 0.060 equiv.) in THF (0.4 mL), and iodobenzene (0.20 mmol, 1.00 equiv.).

Experimental Procedures

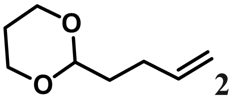
I. Procedure for Preparation of Alkenyl Substrates



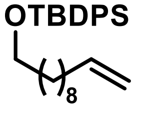
2-(hex-5-enyl)furan (S-24). The title compound was prepared according to the procedure reported in the literature.[1] All spectral data was in accordance with previously published results.



(*E*)-4,8-dimethylnona-1,3,7-triene (S-21). The title compound was synthesized in two steps starting with oxidation of geraniol as reported by Stahl *et al.*[2] followed by Wittig olefination. The spectral data was in accordance with the literature.[3]



2-(but-3-en-1-yl)-1,3-dioxane (S-13). Prepared from 4-pentenal according to the procedure reported by Karimi *et al*.[4] The spectral data was in accordance with the literature.[5]



***tert*-butyldiphenyl(undec-10-en-1-yloxy)silane (S-14).** To a flame-dried round bottom flask equipped with a stir bar was added imidazole (1.02g, 15 mmol). The flask was purged with nitrogen for 5 minutes and dichloromethane (20 mL), undec-10-en-ol (10 mmol), and Et$_3$N (2.09 mL, 15 mmol) were then added. *tert*-Butyl(chloro)diphenylsilane (2.57 mL, 10 mmol) was added to the reaction mixture drop-wise. The reaction was allowed to stir at room temperature for 16 hours, after which water (20 mL) was added to the reaction. The water layer was extracted 3 times using dichloromethane. The organic layers were combined, dried over sodium sulfate, filtered, and condensed under vacuum. The crude product was purified by silica gel column chromatography (Hexanes, stain in KMnO$_4$) to afford a clear yellow oil (3.92 g, 96% yield).

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[1] S. Hobson, R. Marquez, *Org. Biomol. Chem.* **2006**, *4*, 3808.

[2] J. Hoover, S. Stahl, *J. Am. Chem. Soc.* **2011**, *133*, 16901.

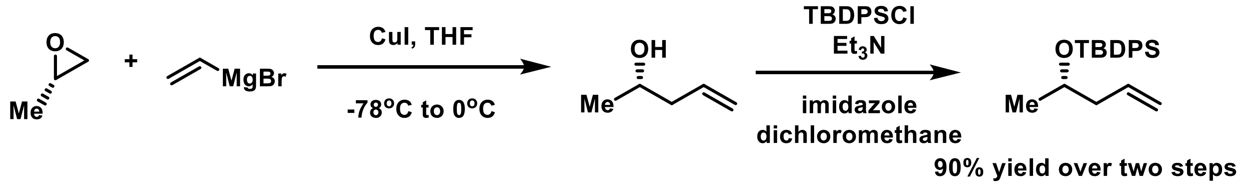
[3] H. Davies, Ø Loe, D. Stafford*, Org. Lett.* **2005**, *7*, 5561.

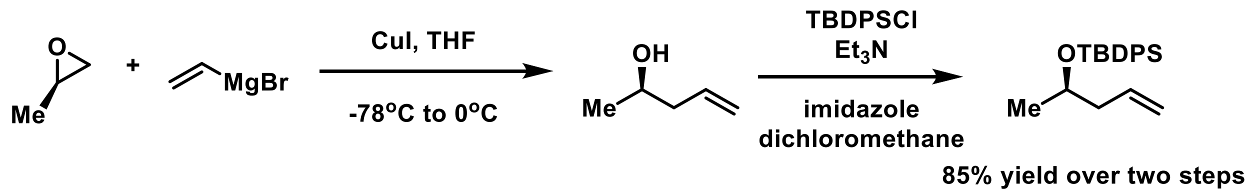
[4] H. Firouzabadi, N, Iranpoor, B. Karimi, *Synlett*. **1999**, 321.

[5] B. Lin, Y, Zhao, Y. Lai, T. Loh, *Angew. Chem. Int. Ed.* **2012**, *51*, 8041.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.70-7.66 (m, 4H), 7.44-7.35 (m, 6H), 5.82 (ddt, *J* = 17.0, 10.2, 6.7 Hz, 1H), 5.00 (dd, *J* = 17.0, 1.4 Hz, 1H), 4.94 (dd, *J* = 10.2, 1.4 Hz, 1H), 3.66 (t, *J* = 6.5 Hz, 1H), 2.08-2.01 (m, 2H), 1.60-1.52 (m, 2H), 1.41-1.23 (m, 14H), 1.05 (s, 9H). **^{13}(150** MHz, $CDCl_3$) δ 139.35, 135.74, 134.36, 129.62, 127.72, 114.28, 64.17, 33.99, 32.75, 29.72, 29.60, 29.52, 29.29, 29.12, 27.05, 25.94, 19.38. **IR** (neat) ν_{max} 2926 (m), 2865 (m), 1427 (m), 1107 (s), 909 (m), 823 (m), 738 (m), 700 (s), 688 (m), 613 (m), 504 (s), 488 (m) cm^{-1}. **HRMS** (DART) for $C_{27}H_{41}OSi$ $[M+H]^+$: Calc'd: 409.2927, found: 409.2926.

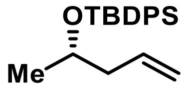
Preparation of (*R*)-tert-butyl(pent-4-en-2-yloxy)diphenylsilane and (*S*)-tert-butyl(pent-4-en-2-yloxy)diphenylsilane.



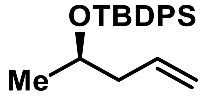


In an argon filled glovebox, a flame-dried round bottom flask equipped with a stir bar was charged with CuI (381mg, 2.0 mmol). The flask was sealed with a rubber septum and taken outside. THF (30 mL) was added under nitrogen. The reaction was cooled down to -78°C with a dry ice/ acetone bath and vinyl magnesium bromide (1M in THF, 20 mL, 20 mmol) was then added dropwise. The reaction was allowed to stir at -78°C for another 15 min, and propylene oxide (0.70 mL, 10 mmol) was added in one portion. The reaction was allowed to stir at -78°C for one hour and at room temperature for another hour. Upon completion the reaction flask was cooled to 0°C and a saturated aqueous ammonium chloride solution was added to the mixture. The organic layer was collected and the aqueous layer was extracted twice with diethyl ether. The organic layers were combined, dried over sodium sulfate, filtered, and concentrated in vacuo. The residue was taken to next step without further purification.

To a flame-dried round bottom flask equipped with a stir bar was added imidazole (1.02g, 15 mmol). The flask was then purged with nitrogen for 10 min. A solution of Crude product and trimethylamine (2.09 mL, 15 mmol) in dichloromethane (15 mL) was added, followed by drop-wise addition of *tert*-butyl(chloro)diphenylsilane (2.60 mL, 10 mmol). The reaction was allowed to stir at room temperature for 16 hours, after which water (20 mL) was added. The organic layer was extracted 3 times using dichloromethane. The organic layers were combined, dried over sodium sulfate, filtered, and condensed under vacuum to afford the crude product.



(*R*)-*tert*-butyl(pent-4-en-2-yloxy)diphenylsilane (S-26). The crude material was purified through silica gel column chromatography (hexane, stain in $KMnO_4$) to afford the product as a yellow oil (2.93g, 90% yield over 2 steps). Spectral data were in accordance with previous literature reports.[6]



(*S*)-*tert*-butyl(pent-4-en-2-yloxy)diphenylsilane (S-27). The crude material was purified through silica gel column chromatography (hexane, stain in $KMnO_4$) to afford the product as a yellow oil (2.76g, 85% yield over 2 steps). Spectral data were in accordance with previous literature report.[7]

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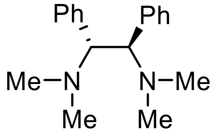
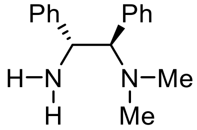
[6] B. Thirupathi, R. Gundapaneni, D. Mohapatra, *Synlett*. **2011***,* 2667.

[7] S. Bujaranipalli, S. Das, *Tetrahedron Lett.* **2015**, *56*, 3747.

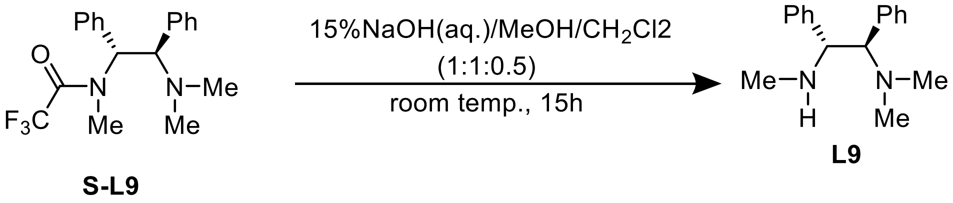
II. Procedures for Preparation of diamine Ligands



(1*R*,2*R*)-N1,N1,N2,N2-tetramethyl-1,2-diphenylethane-1,2-diamine (L7). The title compound was prepared according to the procedure reported in the literature.[8] All spectral data was in accordance with the literature.[8]



(1*R*,2*R*)-N,N-dimethyl-1,2-diphenylethane-1,2-diamine (L8). The title compound was prepared according to the procedure reported in the literature.[9] All spectral data was in accordance with the literature.[10]



(1*R*,2*R*)-N1,N1,N2-trimethyl-1,2-diphenylethane-1,2-diamine (L9). The title compound was prepared from hydrolysis of **S-L9**.[9] To a scintillation vial equipped with a magnetic stir bar was added **S-L16** (30mg, 0.0856 mmol, 1.0 equiv.) and CH_2Cl_2 (0.5 mL). 15% aq. NaOH (1 mL) and MeOH (1 mL) were added to the vial at room temperature. The reaction mixture was stirred vigorously for 15 hours at room temperature. At the end of 15 hours, H_2O (10 mL) was added. The aqueous phase was extracted with ethyl acetate (10 mLx3) and the combined organic phase was washed with brine (20 mL), dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The crude mixture was purified by silica gel chromatography (10% MeOH and 2% Et_3N in DCM, stained with $KMnO_4$) to afford **L9**[11] as a white solid (11 mg, 50% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32-6.98 (m, 10H), 4.00 (d, *J* = 11.0 Hz, 1H), 3.85 (d, *J* = 11.0 Hz, 1H), 2.36 (s, 3H), 2.22 (s, 6H). **HRMS** (ESI) for $C_{17}H_{23}N_2$ $[M+H]^+$: Calc'd: 255.1861, found: 255.1867.

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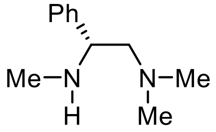
[8] S. Benson, P. Cai, M. Colon, M. Haiza, M. Tokles, J. Snyder, *J. Org. Chem*. **1988**, *53*, 5335.

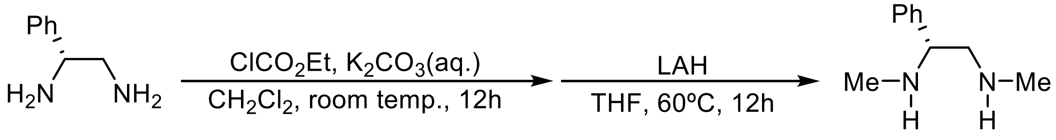
[9] M. Shang, X. Wang, S. Koo, J. Youn, J. Chan, W. Yao, B. Hastings, M. Wasa, *J. Am. Chem. Soc.* **2017**, *139*, 95.

[10] H. Yue, H. Huang, G. Bian, H. Zong, F. Li, L. Song., *Tetrahedron: Asymmetry* **2014**, 25, 170.

[11] T. Honjo, S. Sano, M. Shiro, Y. Nagao, *Angew. Chem. Int. Ed.* **2005**, 5838.



(R)-N1,N2,N2-trimethyl-1-phenylethane-1,2-diamine (L10). The title compound was prepared according to the procedure reported in the literature.[12]All spectral data was in accordance with the literature.[12]



S-L11 **L11**

Diamine (L11). Diamine **S-L11** was prepared by LAH reduction of commercially available D(-)-phenylglycinamide following literature procedures.[13] The yield for the following reactions was not optimized. **S-L11** (430 mg, 3.16 mmol) was dissolved in THF (25 mL) in a round bottom flask flushed with argon and cooled to 0 °C. Potassium carbonate (1.09 g, 7.89 mmol, 476.38 uL) in water (3 mL) was added to the flask followed ethylchlorofomate (2.06 g, 18.94 mmol, 1.80 mL). The resulting two-phase mixture was stirred vigorously at ambient temperature for 12 h. Na_2SO_4 was added directly to the reaction mixture which was filtered through a fritted funnel, and solids were further washed with EtOAc. The filtrate was concentrated in vacuo and the crude material was purified by silica gel chromatography (1% Et_3N in DCM, stained in CAM) to afford ethyl-N-[2-(ethoxycarbonylamino)-1-phenyl-ethyl]carbamate **S2-L11** (124.8 mg, 0.45 mmol, 14.10% yield) as a white solid. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.37 – 7.28 (m, 5H), 5.58 (s, 1H), 4.91 (s, 1H), 4.81 (s, 1H), 4.16 – 4.07 (m, 4H), 3.51 (s, 3H), 1.24 (t, *J* = 6.5 Hz, 6H).

Step 2: In an argon filled glovebox, Lithium Aluminum Hydride was placed in a two-neck round bottom flask equipped with a stir-bar and waterless reflux condenser. X mL of THF where added to the flask which was cooled to 0 ºC. Once cooled, ethyl-N-[2-(ethoxycarbonylamino)-1-phenyl-ethyl]carbamate (124.8 mg, 0.45 mmol, 1.0 equiv.) was added as a solution in 5 mL of THF drop-wise, the mixture was allowed to warm to room temperature over an hour and then heated to reflux for 12 hours. The flask was then cooled to 0 ºC and carefully quenched with x mL of H_2O followed by addition of x mL of a 3M NaOH solution. The mixture was stirred at room temperature for 1 hour before addition of Na_2SO_4. The suspension was then filtered through celite, the solids washed with EtOAc and the filtrate concentrated in vacuo. The crude material was purified by silica gel chromatography (1-10% MeOH in CH_2Cl_2 with 1% Et_3N, stain in ninhydrin) to afford the product as a pale yellow oil (21 mg, 29% yield). All spectral data was in accordance with the literature. [14]

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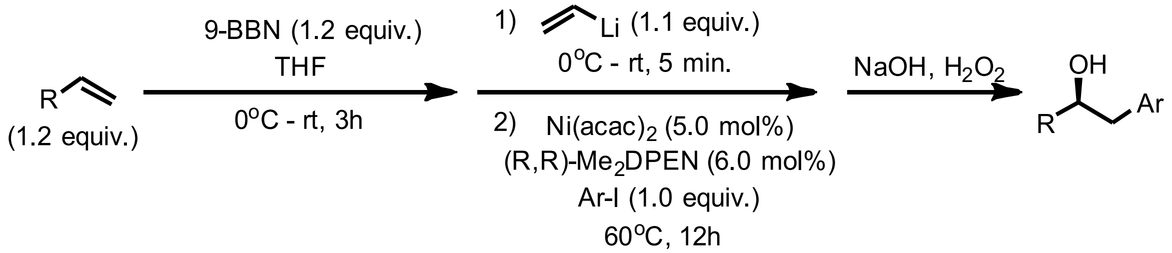
[12] S. de Sousa, P. O' Brien, C. Pilgram, *Tetrahedron* **2002**, *58*, 4643.

[13] Y. Belokon, L.Pritula, V. Tararov, V. Bakhmutov, Y. Struchkov, *J. Chem. Soc., Dalton Trans.* **1990**, 179.

[14] G. Buono, C. Triantaphylides, G. Peiffer, F. Petit, *ChemInform.* **1983**, 14.

III. General Procedure for Conjunctive Cross-Coupling

Procedure A



In a glovebox, under argon, an oven-dried 2-dram vial equipped with a magnetic stir bar was charged with a solution of 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.). The vial was cooled to 0℃, and the olefin (0.24 mmol, 1.20 equiv.) was added. The reaction mixture was allowed to warm to room temperature and stir for 3 hours before being cooled back to 0℃. Vinyllithium (for synthesis of halide free vinyllithium see ref[15]) in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.) was added to the reaction mixture which was then warmed to room temperature and stirred for 5 minutes. Meanwhile a solution of Ni(acac)$_2$ (0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (0.012 mmol, 0.060 equiv.) in THF (0.4 mL) , was allowed to complex for 5 minutes under inert atmosphere. The catalyst solution was added to the boron 'ate' mixture at room temperature followed by addition of aryl iodide (0.20 mmol, 1.00 equiv.) The reaction vial was sealed with a septum cap, taken out of the glovebox and heated at 60 for 12 hours, after which point the reaction mixture was cooled to 0℃ and 30% H_2O_2 (0.5 mL) were added along with 3 M NaOH (0.5 mL). The reaction mixture was allowed to warm up to room temperature and stirred for 3 hours. Aq. saturated $Na_2S_2O_3$ (1 mL) solution was then added to quench the reaction. The aqueous phase was extracted with Et_2O (2 x 2 mL), followed by EtOAc (2 x 2 mL). The combined organic layers were dried over $MgSO_4$, filtered, concentrated under reduced pressure, and subsequently purified via silica gel column chromatography to provide the desired products.

Note: In all cases, a stock solution of 9-BBN derivatives could be prepared and stored in a freezer for as long as one month, before addition of vinyllithium, without any diminishing yield or stereoselectivity.

Note: The reaction can be carried out using aryl-bromides instead of aryl-iodides by following the same method described in **procedure A** with a slight modification. The reaction requires

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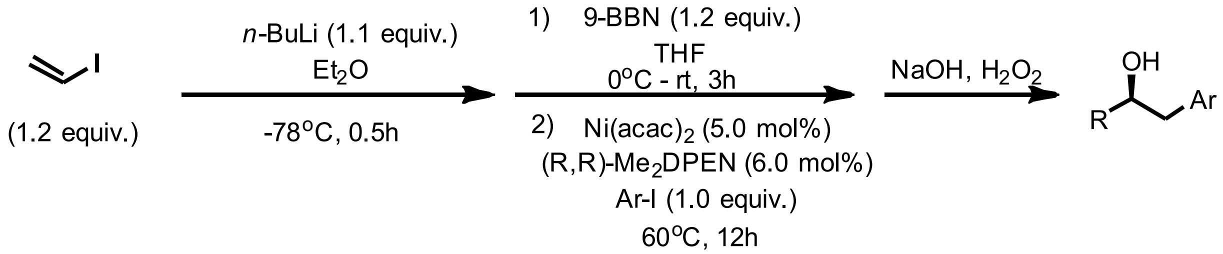
[15] E. Edelstein, S. Namirembe, J. Morken, *J. Am. Chem. Soc.* **2017**, *139*, 5027.

anhydrous NaI (33.0 mg, 0.22 mmol, 1.1 equiv.) which can be added to the solution of 9-BBN and alkene in THF, prior to addition of vinyllithium. These reactions resulted in slightly lower yields (PhBr: 63% yield and PhI 75% yield) and no erosion in selectivity.

Procedure B



In an argon filled glovebox, an oven-dried 2-dram vial equipped with a magnetic stir bar was charged with a solution of 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), cooled to 0℃, and the olefin (0.24 mmol, 1.20 equiv.) was added. The reaction mixture was allowed to warm to room temperature and stir for an additional 3 hours. Meanwhile, inside the glovebox, a separate oven-dried 2 dram vial was charged with vinyliodide (37.0 mg, 0.24 mmol, 1.2 equiv.) and dissolved in 0.4 mL of Et_2O. The vial was brought outside and cooled to -78ºC. A solution of n-butyllithium (90 µL, 0.22 mmol, 1.10 equiv.) was added drop-wise to the reaction vial and the mixture was stirred at -78ºC for 30 minutes. At this point the alkyl-BBN solution generated previously was added to the vial at -78ºC in a drop-wise fashion. The reaction vessel was warmed to room temperature and the solvent was carefully removed under reduced pressure through a Schlenk line. The vial was brought back inside the glovebox and the contents were dissolved in 0.4 mL of THF. Meanwhile, a solution of $Ni(acac)_2$ (0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (0.012 mmol, 0.060 equiv.) in THF (0.4 mL) , was allowed to complex for 5 minutes under inert atmosphere. The catalyst solution was added to the boron 'ate' mixture at room temperature followed by addition of aryl iodide (0.20 mmol, 1.00 equiv.) The reaction vial was sealed with a septum cap and taken out of glovebox and heated at 60 for 12 hours, after which point the reaction mixture was cooled to 0℃, and 30% H_2O_2 (0.5 mL) were added along with 3 M NaOH (0.5 mL). The reaction mixture was allowed to warm up to room temperature and stirred for 3 hours. Aq. saturated $Na_2S_2O_3$ (1 mL) solution was then added to quench the reaction mixture. The aqueous phase was extracted with Et_2O (2 x 2 mL), followed by EtOAc (2 x 2 mL). The combined organic layer was dried over $MgSO_4$, filtered, concentrated under reduced pressure, and subsequently purified via silica gel column chromatography to provide the desired products.

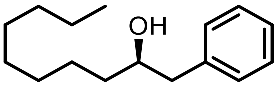
Procedure for large scale synthesis of *(S)*-1-phenyldecan-2-ol (1).

The opposite enantiomer of ligand was used for this reaction, resulting in equal and opposite enantioselectivity in the product.

A 250 mL two-neck round bottom flask equipped with stirbar and reflux condenser was backfilled with N_2, charged with 9-BBN solution in THF (19.2 mL, 0.5 M, 9.6 mmol, 1.20 equiv.) and cooled to 0 ºC. 1-Octene (1.51 mL, 9.60 mmol, 1.20 equiv.) was added and the reaction mixture was warmed to room temperature and stirred for 3 hours. Meanwhile, in an argon filled glove box, Ni(acac)$_2$ (102.76 mg, 0.40 mmol, 0.05 equiv.) and ***(S, S)*-L6** (115.36 mg, 0.48 mmol, 0.06 equiv.) were placed in a 20 mL vial, dissolved in 16 mL of THF and allowed to stir for 2 hours at room temperature.

Outside the glove box, the octyl-BBN solution was cooled to 0 ºC and a solution of vinyllithium in THF (4.89 mL, 1.8 M, 8.80 mmol, 1.1 equiv.) was added to the flask drop-wise. The solution was warmed to room temperature and stirred for 20 minutes, after which the catalyst solution was added (the solution should goes from light blue to, yellow/red), followed by addition of iodobenzene (0.90 mL, 8.0 mmol, 1.00 equiv.). The reaction mixture was heated at 60 ºC for 12 hours, then it was cooled to 0º C and 16 mL of aqueous 3M NaOH solution was added, followed by 16 mL of 30% H_2O_2. The biphasic mixture was allowed to warm to room temperature over 4 hoursn with vigorous stirring. The mixture was cooled to 0 ºC once more and 16 mL of aq. saturated $Na_2S_2O_3$ solution was added. The organic layer was extracted twice with 100 mL of Et_2O and twice with 100 mL of EtOAc, dried over Na_2SO_4, filtered and concentrated in vacuo. The crude mixture was purified by column chromatography (50% DCM in pentane, stain in CAM) to afford the product as a colorless oil (1.31 g, 70% yield).

IV. Characterization of Conjunctive Cross Coupling Products and Analysis of Stereochemistry



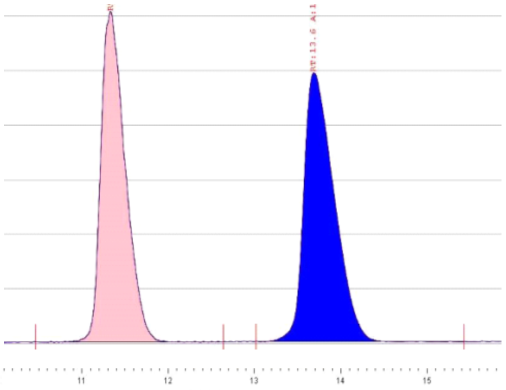
***(R)*-1-phenyldecan-2-ol (1).** The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), 1-octene (26.9 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), iodobenzene (40.8 mg, 0.20 mmol, 1.00 equiv.). The crude mixture was purified by column chromatography (50% DCM in pentane, stain in CAM) to afford the product as a colorless oil (37 mg, 79% yield). **^{1}H NMR** (600 MHz, CDCl$_3$) δ 7.33-7.30 (m, 2H), 7.25-7.12 (m, 3H), 3.84-3.8 (m, 1H), 2.84 (dd, *J* = 13.6, 4.2 Hz, 1H), 2.65 (dd, *J* = 13.6, 8.4 Hz, 1H), 1.53-1.25 (m, 15 H), 0.89 (t, *J* = 6.7 Hz, 3H). **^{13}C NMR** (126 MHz, CDCl$_3$) δ 138.81, 129.56, 128.69, 126.56, 72.84, 44.22, 37.02, 32.03, 29.81, 29.73, 29.42, 25.91, 22.82, 14.25. **IR** (neat) ν_{max} 3373.2 (br, s), 3027.7 (w, s), 2923.2 (s), 2853.8 (s), 1495.4 (s), 1454.1 (s), 1377.2 (s), 1126.3 (s), 1031.3 (m), 744.0 (s), 699.7 (s). **HRMS** (DART) for $C_{16}H_{30}NO$ $(M+NH_4)^+$: Calc'd: 252.2337, found: 252.2327. **$[\alpha]_D^{20}$** = -1.02 (*c* =1.4, CHCl$_3$, *l* = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

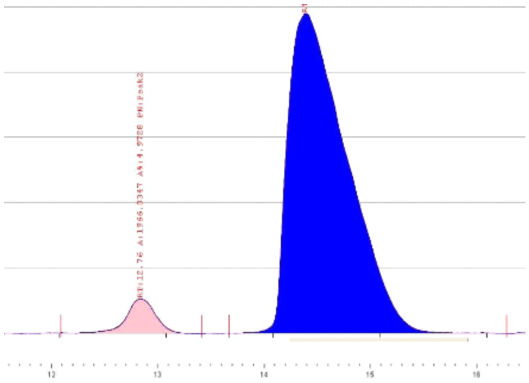
Chiral SFC (Chiracel OD-H, 2% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-1-phenyldecan-2-ol and (R)-1-phenyldecan-2-ol (from gram scale reaction).

Racemic Material



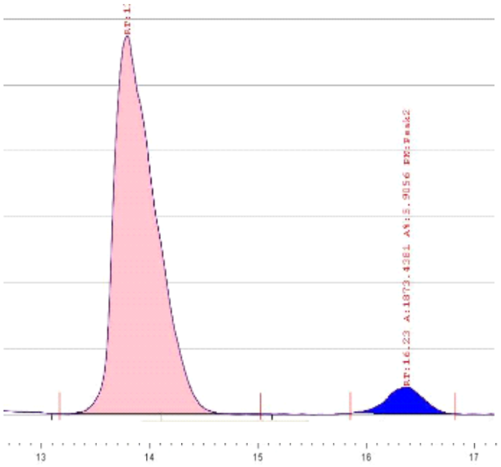
Peak No	% Area	Area	RT (min)
1	49.7027	11395.6892	11.34
2	50.2973	11531.9944	13.6
Total:	100	22927.6836	

Enantioenriched Material

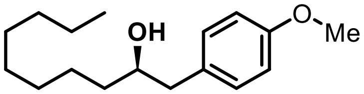


Peak No	% Area	Area	RT (min)
1	4.9728	1966.0347	12.76
2	95.0272	37570.0141	14.3
Total:	100	39536.0488	

Enantioenriched Material from Gram Scale Reaction



Peak No	% Area	Area	RT (min)
1	94.0944	29849.536	13.69
2	5.9056	1873.4381	16.23
Total:	100	31722.9741	



(*R*)-1-(4-methoxyphenyl)decan-2-ol (2). The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), 1-octene (26.9 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), 4-iodoanisole (46.8 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (80% DCM in hexanes, stained with CAM) to afford a white solid (22.7 mg, 43% yield).

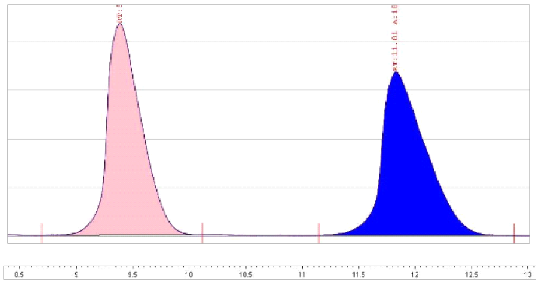
^{1}H NMR (500 MHz, CDCl$_3$) δ 7.13 (d, *J* = 8.7 Hz, 2H), 6.86 (d, *J* = 8.7 Hz, 2H), 3.82-3.73 (m, 4H), 2.78 (dd, *J* = 13.7, 4.2 Hz, 1H), 2.58 (dd, *J* = 13.7, 8.4 Hz, 1H), 1.55-1.22 (m, 14H), 0.88 (t, *J* = 7.0 Hz, 3H). **^{13}C NMR** (150 MHz, CDCl$_3$) δ 158.43, 130.73, 130.51, 114.15, 72.92, 55.41, 43.25, 36.93, 32.02, 29.82, 29.73, 29.42, 25.92, 22.81, 14.25. **IR** (neat) ν_{max} 3382.00 (br, w), 2923.76 (s), 2853.47 (s), 1612.24 (m), 1511.44 (s), 1464.39 (m), 1441.35 (m), 1299.98 (m), 1245.73 (s), 1177.39 (m), 1038.10 (s), 817.67 (m), 570.95 (w), 521.58 (w) cm^{-1}. **HRMS** (DART) for $C_{17}H_{27}O$ $[M+H-H_2O]^+$: Calc'd: 247.2062, found: 247.2072. **$[\alpha]_D^{20}$** = -4.8678 (*c* = 10.6, CHCl$_3$, *l* = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

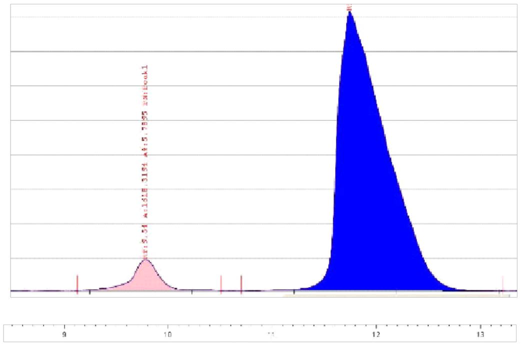
Chiral SFC (Chiracel OJ-H, 5% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-1-(4-methoxyphenyl)decan-2-ol.

Racemic Material

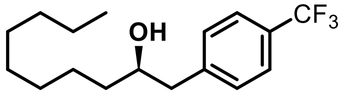


Peak No	% Area	Area	RT (min)
1	50.2402	18343.5134	9.36
2	49.7598	18168.1204	11.81
Total:	100	36511.6338	

Enantioenriched Material



Peak No	% Area	Area	RT (min)
1	5.7895	1618.3194	9.54
2	94.2105	26334.4714	11.54
Total:	100	27952.7908	



(*R*)-1-(4-(trifluoromethyl)phenyl)decan-2-ol (3). The reaction was performed according to the general **procedure B** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), 1-octene (26.9 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), 4-iodobenzotrifluoride (54.4 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (80% DCM in hexanes, stained with CAM) to afford a white solid (42.4 mg, 70% yield).

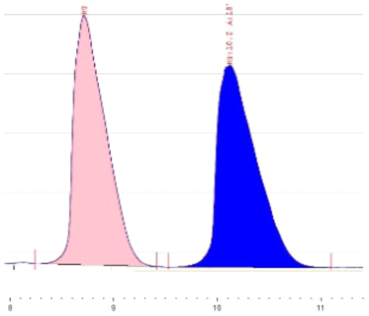
^{1}H NMR (500 MHz, CDCl$_3$) δ 7.56 (d, *J* = 8.0 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 3.89-3.80 (m, 1H), 2.87 (dd, *J* = 13.6, 4.3 Hz, 1H), 2.73 (dd, *J* = 13.6, 8.3 Hz, 1H), 1.57-1.22 (m, 14H), 0.88 (t, *J* = 6.3 Hz, 3H).**^{13}C NMR** (150 MHz, CDCl$_3$) δ 129.88, 125.54, 125.50, 125.46, 125.42, 72.64, 43.90, 37.19, 32.01, 29.74, 29.71, 29.40, 25.84, 22.81, 14.24. **IR** (neat) ν_{max} 3382.00 (br, w), 2923.76 (s), 2853.47 (s), 1612.24 (m), 1511.44 (s), 1464.39 (m), 1441.35 (m), 1299.98 (m), 1245.73 (s), 1177.39 (m), 1038.10 (s), 817.67 (m), 570.95 (w), 521.58 (w) cm^{-1}. **HRMS** (DART) for $C_{17}H_{24}F_3$ $[M+H-H_2O]^+$: Calc'd: 285.183, found: 285.1837. **$[\alpha]_D^{20}$** = -6.195 (*c* = 1.9, CHCl$_3$, *l* = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure B** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

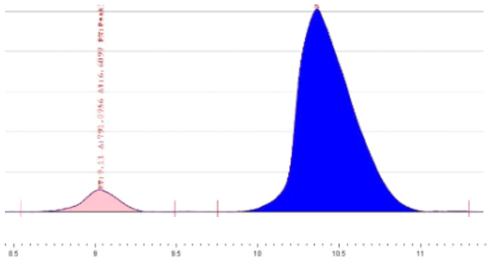
Chiral SFC (Chiracel OJ-H, 0% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-1-(4-(trifluoromethyl)phenyl)decan-2-ol.

Racemic Material

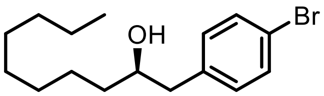


Peak No	% Area	Area	RT (min)
1	50.089	18835.8429	8.72
2	49.911	18768.9061	10.2
Total:	100	37604.749	

Enantioenriched Material



Peak No	% Area	Area	RT (min)
1	6.6099	791.0956	9.11
2	93.3901	11177.2677	10.4
Total:	100	11968.3633	



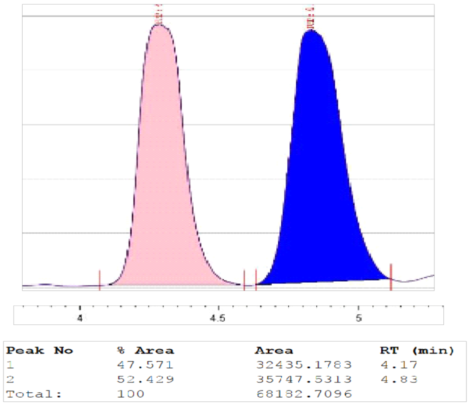
***(R)*-1-(4-bromophenyl)decan-2-ol (4).** The reaction was performed according to the general **procedure A,** with slight deviation. The reaction was run at room temperature for 12 hours. heating the reaction resulted in over-coupling. Reagents: 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), 1-octene (26.9 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), 1,4-bromoiodobenzene (56.6 mg, 0.20 mmol, 1.00 equiv.). The crude mixture was purified by silica gel chromatography (50% DCM in hexanes, stained with CAM) to afford a white solid (50.1 mg, 80% yield).

^{1}H NMR (500 MHz, CDCl$_3$) δ 7.43 (d, J = 7.7 Hz 2H), 7.10 (d, J = 7.7 Hz, 2H), 3.81-3.76 (m, 1H), 2.77 (dd, J = 13.7, 4.3 Hz, 1H), 2.61 (dd, J = 13.7, 8.2 Hz, 1H), 1.52-1.22 (m, 14H), 0.88 (t, J = 6.7 Hz, 3H). **^{13}C NMR** (150 MHz, CDCl$_3$) δ 137.86, 131.68, 131.30, 120.42, 72.66, 43.51, 37.05, 32.01, 29.76, 29.71, 29.40, 25.86, 22.81, 14.25. **IR** (neat) ν_{max} 3354.91 (br, w), 3291.22 (br, w), 2918.89 (s), 2850.85 (s), 1486.49 (m), 1465.52 (m), 1085.32 (w), 1070.61 (m), 1012.27 (m), 799.33 (m) cm^{-1}. **HRMS** (DART) for $C_{16}H_{26}BrO$ [M+H]$^+$: Calc'd: 330.1433, found: 330.142. **$[\alpha]_D^{20}$** = – 6.073 (c = 1.63, CHCl$_3$, l = 50 mm).

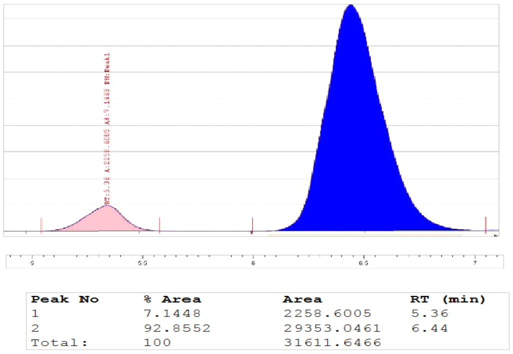
Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

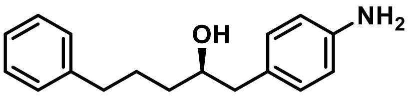
Chiral SFC (Chiracel OJ-H, 5% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-1-(4-bromophenyl)decan-2-ol.


Racemic Material

Peak No	% Area	Area	RT (min)
1	47.571	32435.1783	4.17
2	52.429	35747.5313	4.83
Total:	100	68182.7096	


Enantioenriched Material

Peak No	% Area	Area	RT (min)
1	7.1448	2258.6005	5.36
2	92.8552	29353.0461	6.44
Total:	100	31611.6466	



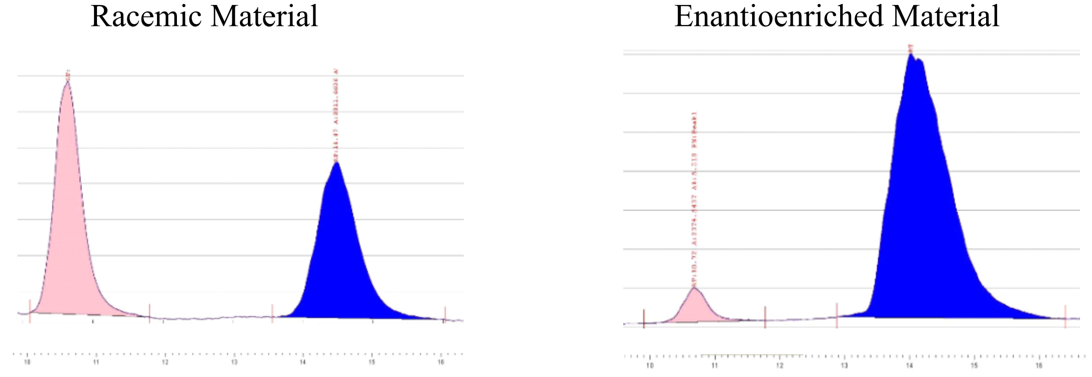
(*R*)-1-(4-aminophenyl)-5-phenylpentan-2-ol (5). The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), allylbenzene (28.4 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), 4-iodoaniline (43.8 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (60% EtAOc in hexanes, stained with CAM) to afford a yellow solid (27.6 mg, 54% yield).

^{1}H NMR (500 MHz, CDCl$_3$) δ 7.31-7.15 (m, 5H), 6.98 (d, J = 8.2 Hz, 2H), 6.64 (d, J = 8.2 Hz, 2H), 3.91-3.29 (m, 3H), 2.72 (dd, J = 13.7, 4.1 Hz, 1H), 2.68-2.60 (m, 2H), 2.51 (dd, J = 13.7, 8.5 Hz, 1H), 1.90-1.76 (m, 1H), 1.75-1.64 (m, 1H), 1.61-1.48 (m, 2H). **^{13}C NMR** (150 MHz, CDCl$_3$) δ 145.04, 142.60, 130.38, 128.56, 128.42, 127.43, 125.84, 115.53, 72.74, 43.31, 36.39, 36.05, 27.79. **IR** (neat) ν_{max} 3343.63 (br, m), 3034.98 (w), 2930.39 (m), 2856.48 (w), 1621.91 (m), 1515.60 (s), 1501.35 (m), 1452.64 (w), 1272.58 (w), 1178.86 (w), 1087.70 (w), 820.26 (m), 749.76 (m), 699.81 (m), 562.06 (w), 509.25 (w) cm^{-1}. **HRMS** (DART) for $C_{17}H_{22}NO$ [M+H]$^+$: Calc'd: 256.1701, found: 256.1702. **$[\alpha]_D^{20}$** = -1.74 (c = 1.40, CHCl$_3$, l = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

Chiral SFC (Chiracel OJ-H, 20% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-1-(4-aminophenyl)-5-phenylpentan-2-ol.

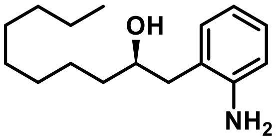


Racemic Material

Enantioenriched Material

Peak No	% Area	Area	RT (min)
1	50.5473	9006.692	10.64
2	49.4527	8811.6606	14.47
Total:	100	17818.3526	

Peak No	% Area	Area	RT (min)
1	5.318	2374.5437	10.72
2	94.682	42276.4197	14.08
Total:	100	44650.9634	



(*R*)-1-(2-aminophenyl)decan-2-ol (6). The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), 1-octene (32.7 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), 1-octene (26.9 mg, 0.24 mmol, 1.20 equiv.), a solution of $Ni(acac)_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), 2-iodoaniline (43.8 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (35% EtOAc in hexanes, stained with CAM) to afford a dark oil (30.9 mg, 62% yield).

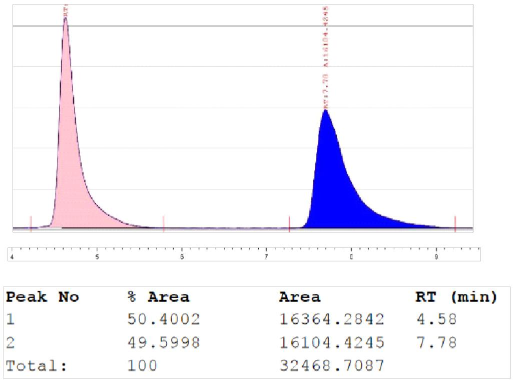
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.10-7.01 (m, 2H), 6.81-6.72 (m, 2H), 3.94-3.84 (m, 1H), 3.80-3.44 (br s, 1H). 2.74 (dd, *J* = 14.3, 3.7 Hz, 1H), 2.66 (dd, *J* = 14.3, 8.1 Hz, 1H), 1.58-1.19 (m, 14H), 0.88 (t, *J* = 6.20 Hz, 1H) . **^{13}C NMR** (150 MHz, $CDCl_3$) δ 144.73, 131.40, 127.75, 124.92, 119.67, 116.91, 73.22, 39.65, 37.49, 32.01, 29.78, 29.73, 29.41, 25.97, 22.81, 14.24. **IR** (neat) 3347.00 (br, w), 2922.38 (s), 2852.76 (s), 1621.62 (m), 1583.64 (w), 1495.75 (m), 1465.75 (w), 1311.84 (w), 1264.58 (w), 1076.47 (w), 747.99 (s), 722.18 (w), 649.21 (w), 616.58 (w), 533.16 (w), 491.23 (w) cm^{-1}. **HRMS** (DART) for $C_{16}H_{28}NO$ $[M+H]^+$: Calc'd: 250.2171, found: 250.2181. **$[\alpha]_D^{20}$** = +8.08 (*c* = 4.12, $CHCl_3$, *l* = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

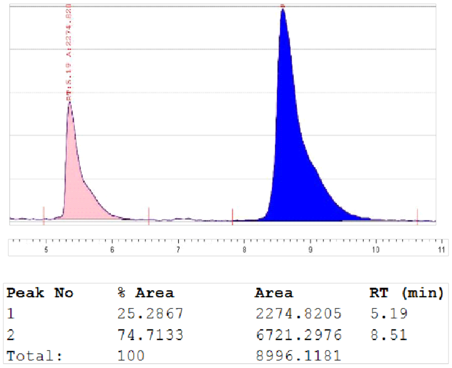
Chiral SFC (Chiracel OJ-H, 10% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-1-(2-aminophenyl)decan-2-ol.

Racemic Material

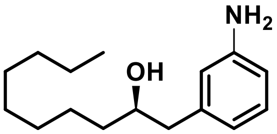


Peak No	% Area	Area	RT (min)
1	50.4002	16364.2842	4.58
2	49.5998	16104.4245	7.78
Total:	100	32468.7087	

Enantioenriched Material



Peak No	% Area	Area	RT (min)
1	25.2867	2274.8205	5.19
2	74.7133	6721.2976	8.51
Total:	100	8996.1181	



(R)-1-(3-aminophenyl)decan-2-ol (7). The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), (32.7 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), 1-octene (26.9 mg, 0.24 mmol, 1.20 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), 3-iodoaniline (43.8 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (30% EtOAc in hexanes, stained with CAM) to afford a yellow solid (39.9 mg, 80% yield).

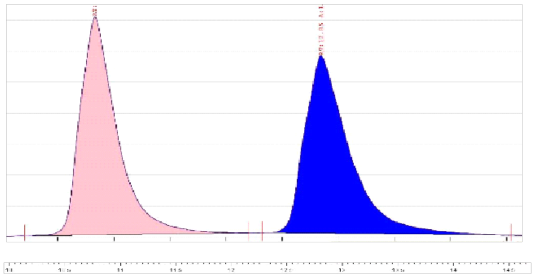
^{1}H NMR (500 MHz, CDCl$_3$) δ 7.10 (t, *J* = 7.6 Hz, 1H), 6.64-6.51 (m, 3H), 3.89-3.36 (m, 3H), 2.75 (dd, *J* = 13.5, 4.0 Hz, 1H), 2.53 (dd, *J* = 13.5, 8.6 Hz, 1H), 1.58-1.18 (m, 14H), 0.89 (t, *J* = 6.8 Hz, 3H). **^{13}C NMR** (150 MHz, CDCl$_3$) δ 146.74, 140.00, 129.66, 119.76, 116.23, 113.44, 72.70, 44.25, 37.00, 32.02, 29.82, 29.73, 29.41, 25.92, 22.81, 14.24. **IR** (neat) ν_{max} 3382.23 (br, w), 3316.11 (m), 2953.10 (m), 2922.65 (s), 2853.03 (s), 1601.73 (m), 1463.40 (m), 789.09 (m), 697.50 (m) cm^{-1}. **HRMS** (DART) for $C_{16}H_{28}NO$ $[M+H]^+$: Calc'd: 250.2171, found: 250.2169. **$[\alpha]_D^{20}$** = -11.157 (*c* = 0.72, CHCl$_3$, *l* = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

Chiral SFC (Chiracel OJ-H, 8% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-1-(3-aminophenyl)decan-2-ol.

Racemic Material



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Peak No      % Area        Area          RT (min)
1            50.4733       16097.1974    10.83
2            49.5267       15795.286     12.85
Total:       100           31892.4834
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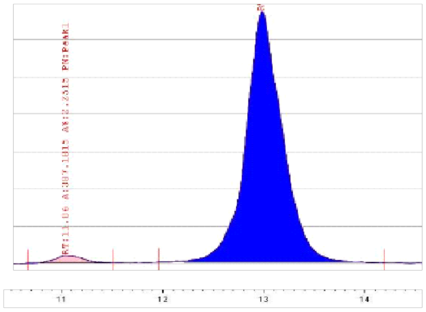
Enantioenriched Material



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Peak No      % Area        Area          RT (min)
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2            97.7485       16809.2242    12.98
Total:       100           17196.4057
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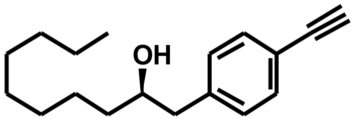


(*R*)-1-(4-ethynylphenyl)decan-2-ol (8). The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), 1-octene (26.9 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), (4-iodophenylethynyl)trimethylsilane (60.04 mg, 0.20 mmol, 1.00 equiv.). After oxidation and work-up methanol (1.5 mL) and K$_2$CO$_3$ (110.56 mg, 0.80 mmol, 4.00 equiv.) were added to the crude mixture. The reaction mixture was allowed to stir at room temperature for 3 hours. Methanol was then evaporated off under reduced pressure. The resulting residue was diluted with CH$_2$Cl$_2$ (4 mL), washed with aq. Saturated NaHCO$_3$ (2mL x 3), dried with Na$_2$SO$_4$, filtered, and concentrated under reduced pressure. The crude mixture was purified by silica gel chromatography (50% DCM in hexanes, stained with CAM) to afford a white solid (28.5 mg, 55% yield).

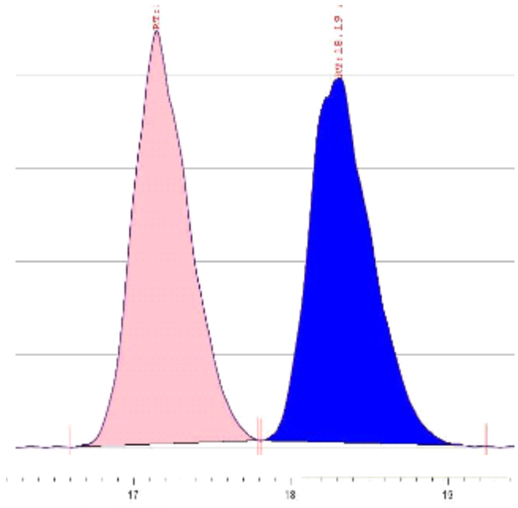
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.44 (d, *J* = 7.9 Hz, 2H), 7.18 (d, *J* = 7.9 Hz, 2H), 3.89-3.75 (m, 1H), 3.05 (s, 1H), 2.82 (dd, *J* = 13.6, 4.3 Hz, 1H), 2.66 (dd, *J* = 13.6, 8.2 Hz, 1H), 1.59-1.18 (m, 14H), 0.88 (t, *J* = 6.7 Hz, 3H).**^{13}C NMR** (126 MHz, CDCl$_3$) δ 139.88, 132.40, 129.58, 120.29, 83.72, 77.08, 72.69, 44.06, 37.06, 32.01, 29.75, 29.70, 29.40, 25.85, 22.81, 14.25. **IR** (neat) ν_{max} 3356.92 (w), 3278.76 (m), 2955.76 (w), 2920.35 (s), 2852.12 (s), 1506.45 (w), 1467.54 (m), 1431.34 (w), 1410.04 (w), 1128.54 (w), 1106.54 (w), 1084.63(m), 1058.94 (w), 1022.94 (m), 907.26 (w), 837.69 (m), 818.04 (s), 722.54 (m), 642.49 (s), 614.39 (s), 578.83 (s), 527.84 (m), 517.54 (m) cm^{-1}. **HRMS** (DART) for C$_{17}$H$_{27}$O [M+H-H$_2$O]$^+$: Calc'd: 247.2062, found: 247.2072. **[α]$_D^{20}$** = -8.4280(*c* = 0.775, CHCl$_3$, *l* = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

Chiral SFC (Chiracel ODR-H, 2% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-1-(4-ethynylphenyl)decan-2-ol.

Racemic Material



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Peak No    % Area     Area          RT (min)
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2          49.8913    11039.1609    18.19
Total:     100        22126.4384
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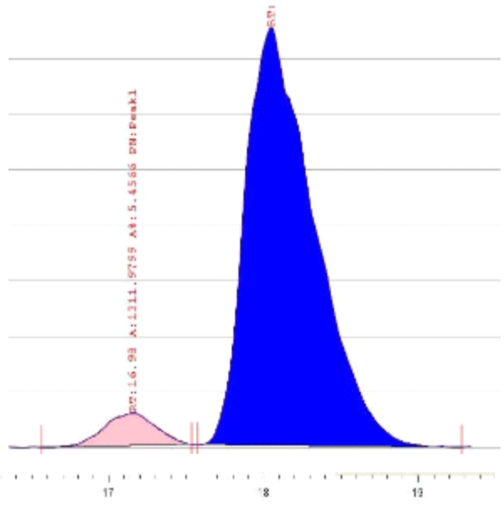
Enantioenriched Material



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Peak No    % Area     Area          RT (min)
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2          94.5434    22731.7993    17.85
Total:     100        24043.7792
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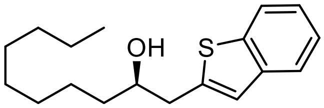


(*R*)-1-(benzo[*b*]thiophen-2-yl)decan-2-ol (9).

The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), (32.7 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), 1-octene (26.9 mg, 0.24 mmol, 1.20 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), 2-iodobenzothiophene (52.02 mg, 0.20 mmol, 1.00 equiv.). The crude mixture was purified by silica gel chromatography (5% EtOAc in hexanes, stained with CAM) to afford a yellow oil (29.3 mg, 50% yield). ^{1}**H NMR** (500 MHz, CDCl$_3$)

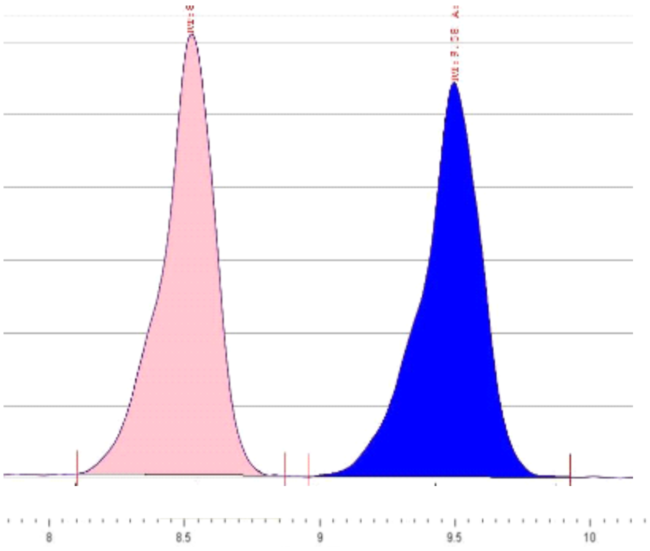
7.78 (d, J = 7.9 Hz, 1H), 7.70 (d, J = 7.8 Hz, 1H), 7.35 – 7.26 (m, 2H), 7.10 (s, 1H), 3.92 (tq, J = 8.5, 4.4 Hz, 1H), 3.10 (dd, J = 14.7, 4.0 Hz, 1H), 2.97 (dd, J = 14.7, 8.1 Hz, 1H), 1.75 (d, J = 4.1 Hz, 1H), 1.60 – 1.47 (m, 3H), 1.44 – 1.19 (m, 12H), 0.89 (t, J = 6.8 Hz, 3H). **^{13}C NMR** (126 MHz, CDCl$_3$) δ 142.02, 140.17, 139.91, 124.36, 123.88, 123.03, 122.74, 122.27, 72.27, 39.07, 36.85, 32.01, 29.74, 29.70, 29.40, 25.86, 22.81, 14.25. **IR** (neat) ν_{max} 3371.6 (br), 3058.9 (s, w), 2923.4 (s), 2853.2 (s), 1457.8 (s), 1453.8 (s) , 1435.8 (s, w), 1307.1 (s, w), 1253.8 (s, w), 1154.3 (s, w), 1125.3 (s), 1102.9 (s), 1066.2 (s), 854.0 (s), 743.9 (s), 725.9 (s) . **HRMS** (DART) for $C_{18}H_{25}S$ $[M+H-H_2O]^+$: Calc'd: 273.1677, found: 273.1668. **$[\alpha]_D^{20}$** = -7.97 (c = 1.00, CHCl$_3$, l = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

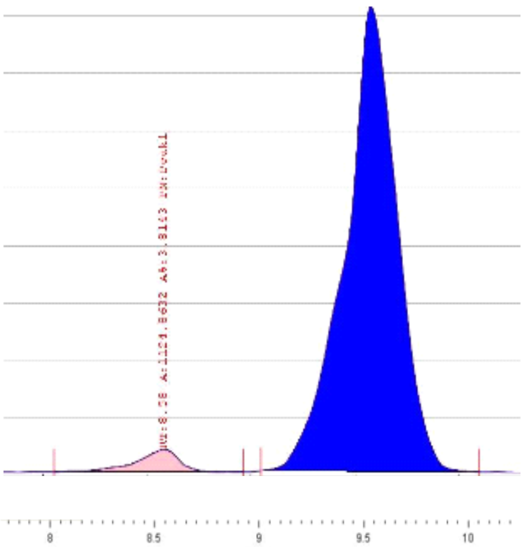
Chiral SFC (Chiracel OJ-H, 10% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-1-(benzo[b]thiophen-2-yl)decan-2-ol.

Racemic Material

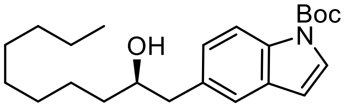


Peak No	% Area	Area	RT (min)
1	49.717	9348.6838	8.48
2	50.283	9455.0956	9.58
Total:	100	18803.7794	

Enantioenriched Material



Peak No	% Area	Area	RT (min)
1	3.8143	1124.8632	8.58
2	96.1857	28365.5159	9.57
Total:	100	29490.3791	



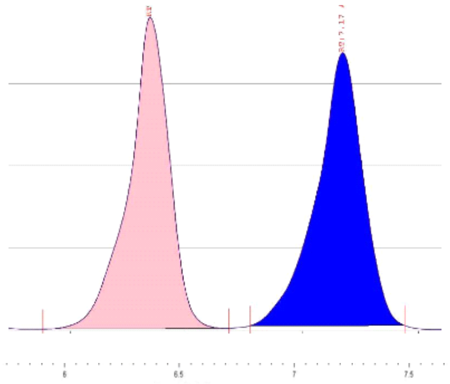
***tert*-butyl (*R*)-5-(2-hydroxydecyl)-1*H*-indole-1-carboxylate (10).** The reaction was performed according to the general **procedure A,** with slight deviation. The reaction was run at room temperature for 12 hours. Reaction was prepared with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), 1-octene (26.9 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL tert-butyl 5-iodoindole-1-carboxylate (68.63 mg, 0.20 mmol, 1.00 equiv.). The crude mixture was purified by silica gel chromatography (5% EtOAc in hexanes, stained with CAM) to afford a colorless oil (41 mg, 55% yield). **^{1}H NMR** (500 MHz, CDCl$_3$) δ 8.07 (d, *J* = 8.4 Hz, 1H), 7.58 (d, *J* = 3.7 Hz, 1H), 7.40 (d, *J* = 1.6 Hz, 1H), 7.17 (dd, *J* = 8.5, 1.7 Hz, 1H), 6.53 (dd, *J* = 3.7, 0.9 Hz, 1H), 3.88 – 3.79 (m, 1H), 2.93 (dd, *J* = 13.6, 4.2 Hz, 1H), 2.72 (dd, *J* = 13.6, 8.4 Hz, 1H), 1.67 (s, 9H), 1.57 – 1.46 (m, 4H), 1.42 – 1.19 (m, 11H), 0.88 (t, *J* = 6.8 Hz, 3H). **^{13}C NMR** (150 MHz, CDCl$_3$) δ 149.89, 134.17, 132.95, 131.07, 126.33, 125.83, 121.62, 115.29, 107.22, 83.76, 73.09, 44.07, 36.93, 32.02, 29.83, 29.74, 29.42, 28.35, 25.95, 22.81, 14.25. **IR** (neat) ν_{max} 3429.9 (br), 2924.5 (s), 2854.1 (s), 1733.1(s), 1580.6 (s, w), 1537.9 (s, w), 1469.1 (s), 1443.1 (s), 1292.9 (s), 1163.5 (s), 1130.7 (s), 1039.8 (s), 1023.2 (s), 886.2 (s), 855.4 (s), 804.7 (s, w), 804.7 (s, w), 765.4 (s), 723.9 (s). **HRMS** (DART) for $C_{23}H_{36}NO_3$ [M+H]$^+$: Calc'd: 374.2913, found: 374.2695. **$[\alpha]_D^{20}$** = -1.43 (*c* = 1.00, CHCl$_3$, *l* = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

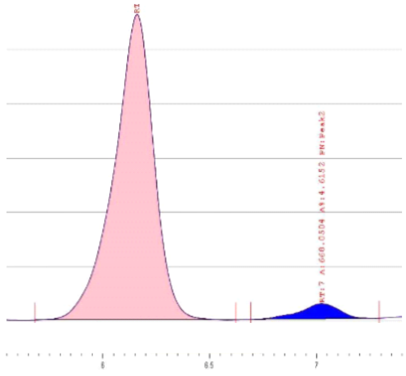
Chiral SFC (Chiracel OJ-H, 6% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of tert-butyl (R)-5-(2-hydroxydecyl)-1H-indole-1-carboxylate.

Racemic Material

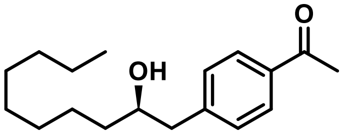


Peak No	% Area	Area	RT (min)
1	49.8935	23002.6909	6.34
2	50.1065	23100.8532	7.17
Total:	100	46103.5441	

Enantioenriched Material



Peak No	% Area	Area	RT (min)
1	95.3848	13806.9061	6.18
2	4.6152	668.0504	7
Total:	100	14474.9565	



(*R*)-1-(4-(2-hydroxydecyl)phenyl)ethan-1-one (11). The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), (32.7 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), 1-octene (26.9 mg, 0.24 mmol, 1.20 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), 4'-iodoacetophenone (55.3 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (20% EtOAc in hexanes, stained with CAM) to afford a yellow solid (43.1 mg, 78% yield).

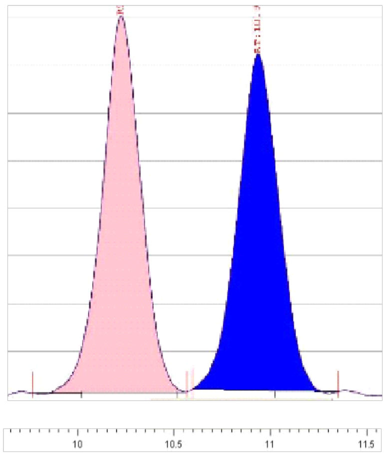
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 3.89-3.82 (m, 1H), 2.88 (dd, *J* = 13.6, 4.3 Hz, 1H), 2.73 (dd, *J* = 13.6, 8.3 Hz, 1H), 2.59 (s, 3H), 1.58-1.20 (m, 14H), 0.88 (t, *J* = 6.9 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 197.95, 144.77, 135.68, 129.79, 128.73, 72.64, 44.12, 37.21, 32.00, 29.74, 29.69, 29.39, 26.72, 25.84, 22.81, 14.25. **IR** (neat) ν_{max} 3425.67 (br, w), 2922.79 (s), 2853.10 (m), 1678.84 (s), 1605.64 (m), 1569.98 (w), 1464.42 (w), 1413.10 (w), 1357.42 (w), 1304.74 (w), 1266.38 (s), 1182.21 (w), 1117.60 (w), 1075.11 (w), 956.57 (w), 837.28 (w), 813.44 (w), 599.71 (m), 585.76 (w) cm^{-1}. **HRMS** (DART) for $C_{18}H_{29}O_2$ $[M+H]^+$: Calc'd: 277.2168, found: 277.2156. **$[\alpha]_D^{20}$** = -8.148 (*c* = 3.46, $CHCl_3$, *l* = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

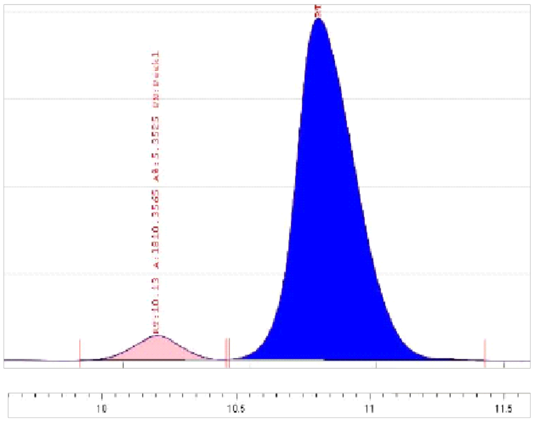
Chiral SFC (Chiracel AD-H, 10% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-1-(4-(2-hydroxydecyl)phenyl)ethan-1-one.

Racemic Material

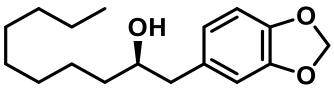


Peak No	% Area	Area	RT (min)
1	51.5576	2225.3493	10.2
2	48.4424	2090.8911	10.91
Total:	100	4316.2404	

Enantioenriched Material



Peak No	% Area	Area	RT (min)
1	5.3525	1810.3565	10.13
2	94.6475	32012.4898	10.77
Total:	100	33822.8463	



(*R*)-1-(benzo[d][1,3]dioxol-5-yl)decan-2-ol (12). The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), 1-octene (26.9 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), 5-iodo-1,3-benzodioxole (49.6 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (50% DCM in hexanes, stained with CAM) to afford a white solid (36.1 mg, 65% yield).

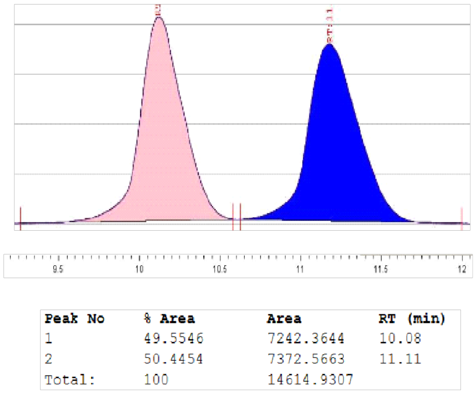
^{1}H NMR (500 MHz, CDCl$_3$) δ 6.76 (d, *J* = 7.8 Hz, 1H), 6.71 (d, *J* = 1.7 Hz, 1H), 6.66 (dd, *J* = 7.8, 1.7 Hz, 1H), 5.94 (s, 2H), 3.84-3.71 (m, 1H), 2.75 (dd, *J* = 13.7, 4.1 Hz, 1H), 2.55 (dd, *J* = 13.7, 8.5 Hz, 1H), 1.55-1.15 (m, 14H), 0.88 (t, *J* = 6.7 Hz, 3H). **^{13}C NMR** (150 MHz, CDCl$_3$) δ 147.92, 146.32, 132.51, 122.41, 109.85, 108.44, 101.01, 72.87, 43.88, 36.93, 32.02, 29.81, 29.73, 29.41, 25.91, 22.81, 14.24. **IR** (neat) ν_{max} 3406.47 (br, w), 2924.26 (s), 2854.22 (m), 1503.03 (m), 1489.12 (s), 1466.03 (w), 1332.03 (m), 1245.98 (s), 1189.19 (w), 1040.89 (s), 940.18 (m), 928.68 (m), 807.76 (m) cm^{-1}. **HRMS** (DART) for $C_{17}H_{25}O_2$ $[M+H-H_2O]^+$: Calc'd: 261.1855, found: 261.1849. **$[\alpha]_D^{20}$** = – 9.291 (*c* = 1.58, CHCl$_3$, *l* = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

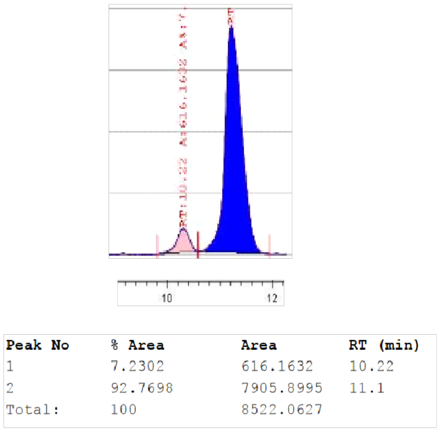
Chiral SFC (Chiracel OD-H, 5% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-1-(benzo[d][1,3]dioxol-5-yl)decan-2-ol.

Racemic Material

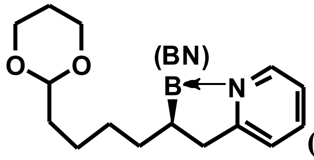


Peak No	% Area	Area	RT (min)
1	49.5546	7242.3644	10.08
2	50.4454	7372.5663	11.11
Total:	100	14614.9307	

Enantioenriched Material



Peak No	% Area	Area	RT (min)
1	7.2302	616.1632	10.22
2	92.7698	7905.8995	11.1
Total:	100	8522.0627	



(*R*)-2-(2-(9-borabicyclo[3.3.1]nonan-9-yl)-6-(1,3-dioxan-2-yl)hexyl)pyridine (13). The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), 2-(but-3-en-1-yl)-1,3-dioxane (**S-14**) (34.1 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), 2-iodopyridine (41.0 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (15% EtOAc in pentane, stained with CAM) to afford a colorless oil (54.9 mg, 73% yield).

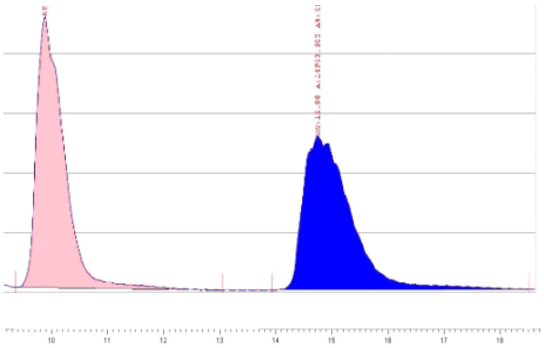
^{1}H NMR (600 MHz, CDCl$_3$) δ 8.77 (d, *J* = 5.7 Hz, 1H), 7.74 (td, *J* = 7.7, 1.4 Hz, 1H), 7.36 (d, *J* = 7.8 Hz, 1H), 7.26 (m, 1H), 4.47 (t, *J* = 5.2 Hz, 1H), 4.08 (ddd, *J* = 11.2, 5.1, 1.7 Hz, 2H), 3.74 (tdd, *J* = 12.1, 2.6, 1.3 Hz, 2H), 3.14 (dd, *J* = 16.8, 6.2 Hz, 1H), 2.77 (d, *J* = 16.8 Hz, 1H), 2.32 – 2.14 (m, 1H), 2.10 – 1.13 (m, 21H), 1.07 – 0.92 (m, 1H), 0.63 – 0.41 (m, 2H). **^{13}C NMR** (151 MHz, CDCl$_3$) δ 164.37, 144.94, 138.42, 123.45, 121.47, 102.76, 67.03, 36.97, 35.71, 34.89, 33.43, 30.83, 30.21 (br) 30.02, 29.34, 28.96, 26.04, 25.10, 24.66, 24.34, 23.93 (br), 22.15 (br **^{11}B NMR** (160 MHz, CDCl$_3$) δ 2.35. **IR** (neat) ν_{max} 2917.5 (s), 2837.1 (s), 1616.6 (s), 1480.3 (s), 1451.1 (s), 1240.4 (s), 1143.1 (s), 1111.5 (m), 993.2 (s), 867.3 (s), 765 (s),723 (s). **HRMS** (DART) for $C_{23}H_{37}NO_2B$ [M+H]$^+$: Calc'd: 370.2917, found: 370.2917.

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

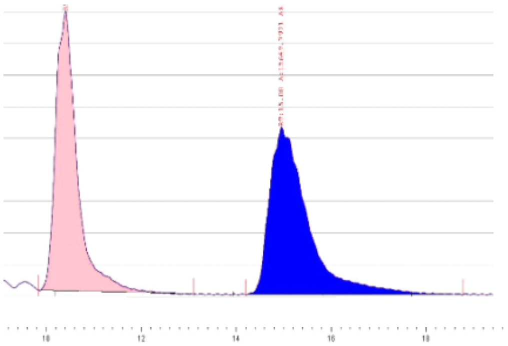
Chiral SFC (Chiracel OJ-H, 10% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-2-(2-(9-borabicyclo[3.3.1]nonan-9-yl)-6-(1,3-dioxan-2-yl)hexyl)pyridine.

Racemic Material

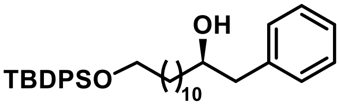


Peak No	% Area	Area	RT (min)
1	50.3138	15132.3538	10.06
2	49.6862	14943.605	14.88
Total:	100	30075.9588	

Enantioenriched Material



Peak No	% Area	Area	RT (min)
1	48.529	14755.2636	10.25
2	51.471	15649.7971	15.08
Total:	100	30405.0607	



(*R*)-13-((*tert*-butyldiphenylsilyl)oxy)-1-phenyltridecan-2-ol (14). The reaction was performed according to the general **procedure B** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), (32.7 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), tert-butyldiphenyl(undec-10-en-1-yloxy)silane (**S-11**) (98.1 mg, 0.24 mmol, 1.20 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), iodobenzene (40.8 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (40% DCM in hexanes, stained with CAM) to afford a colorless oil (78.5 mg, 74% yield).

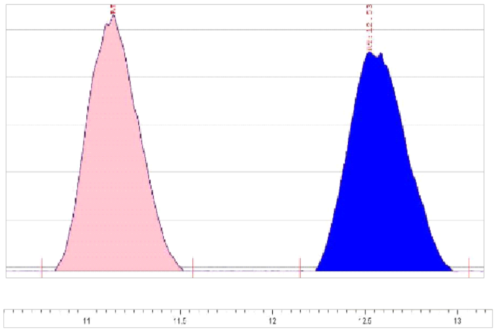
^{1}**H NMR** (600 MHz, CDCl$_3$) δ 7.69-7.65 (m, 4H), 7.44-7.35 (m, 6H), 7.34-7.29 (m, 2H), 7.26-7.20 (m, 3H), 3.86 (m, 1H), 3.65 (t, *J* = 6.5 Hz, 2H), 2.84 (dd, *J* = 13.6, 4.2 Hz, 1H), 2.65 (dd, *J* = 13.6, 8.5 Hz, 1H), 1.59-1.27 (m, 20H), 1.05 (s, 9H). ^{13}**C NMR** (150 MHz, CD$_3$CN) δ 138.94, 134.54, 133.19, 128.85, 128.59, 127.24, 126.87, 125.02, 71.16, 62.94, 42.93, 35.98, 31.40, 29.04, 28.53, 28.49, 28.43, 28.40, 28.13, 25.43, 24.63, 24.59, 17.94. **IR** (neat) ν_{max} 3387.23 (br, w), 2926.31 (s), 2854.13 (m), 1463.04 (w), 1427.68 (w), 1389.36 (w), 1360.85 (w), 1109.82 (s), 1030.21 (w), 1007.61 (w), 823.15 (w), 739.64 (m), 700.25 (s), 613.40 (m), 504.73 (m), 490.04 (w) cm^{-1}. **HRMS** (DART) for $C_{35}H_{51}O_2Si$ $[M+H]^+$: Calc'd: 531.3658, found: 531.3682. **$[\alpha]_D^{20}$** = -0.546 (*c* = 1.83, $CHCl_3$, *l* = 50 mm).

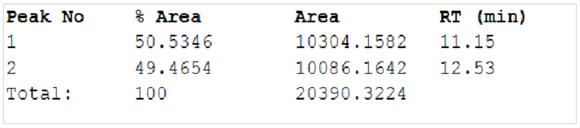
Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure B** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

Chiral SFC (Chiracel ODR-H, 10% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-13-((tert-butyldiphenylsilyl)oxy)-1-phenyltridecan-2-ol.

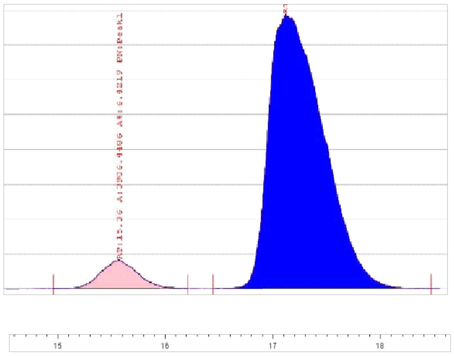
Racemic Material

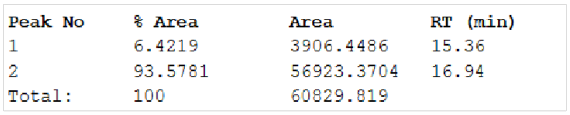




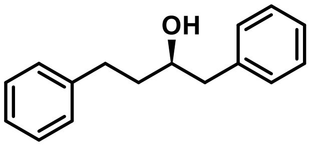
Peak No	% Area	Area	RT (min)
1	50.5346	10304.1582	11.15
2	49.4654	10086.1642	12.53
Total:	100	20390.3224	

Enantioenriched Material





Peak No	% Area	Area	RT (min)
1	6.4219	3906.4486	15.36
2	93.5781	56923.3704	16.94
Total:	100	60829.819	



(*R*)-1,4-diphenylbutan-2-ol (15). The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), (32.7 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), styrene (25.0 mg, 0.24 mmol, 1.20 equiv.), a solution of $Ni(acac)_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), iodobenzene (40.8 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (60% DCM in hexanes, stained with CAM) to afford a colorless oil (22.6 mg, 50% yield).

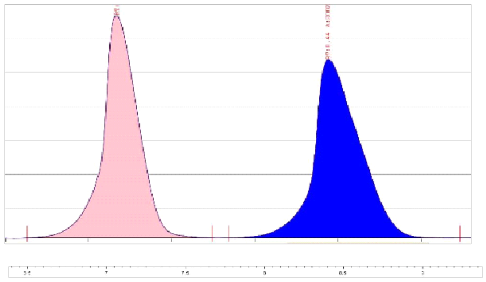
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38-7.14 (m, 10H), 3.92-3.80 (m, 1H), 2.90-2.82 (m, 2H), 2.76-2.66 (m, 2H), 1.94-1.77 (m, 2H), 1.53 (s, 1H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 142.18, 138.49, 129.58, 128.75, 128.60, 128.56, 126.69, 125.98, 72.10, 44.31, 38.59, 32.27. **IR** (neat) ν_{max} 3405.14 (br,s), 3083.98 (w), 3061.19 (w), 3025.99 (m), 2919.73 (w), 2857.89 (w), 1602.16 (w), 1494.75 (m), 1453.54 (m), 1081.23 (m), 1048.45 (m), 1030.25 (m), 746.25 (s), 698.64 (s), 494.02 (w) cm^{-1}. **HRMS** (DART) for $C_{16}H_{19}O$ $[M+NH_4]^+$: Calc'd: 244.1701, found: 244.1697. **$[\alpha]_D^{20}$** = +12.00 (c = 0.83, $CHCl_3$, l = 50 mm).

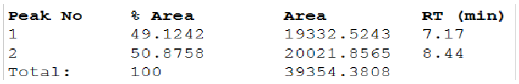
Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

Chiral SFC (Chiracel OJ-H, 10% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-1,4-diphenylbutan-2-ol.

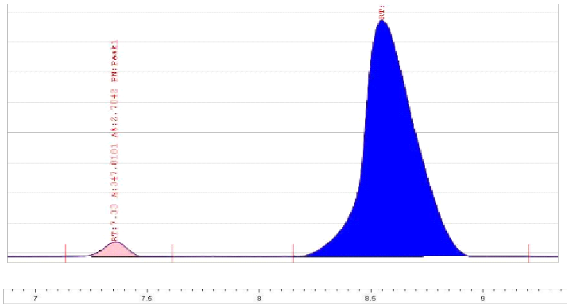
Racemic Material

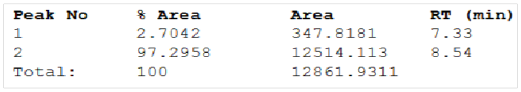




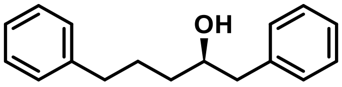
Peak No	% Area	Area	RT (min)
1	49.1242	19332.5243	7.17
2	50.8758	20021.8565	8.44
Total:	100	39354.3808	

Enantioenriched Material





Peak No	% Area	Area	RT (min)
1	2.7042	347.8181	7.33
2	97.2958	12514.113	8.54
Total:	100	12861.9311	



(*R*)-1,5-diphenylpentan-2-ol (16). The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), (32.7 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), allylbenzene (28.4 mg, 0.24 mmol, 1.20 equiv.), a solution of $Ni(acac)_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), iodobenzene (40.8 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (65% DCM in hexanes, stained with CAM) to afford a colorless oil (24.0 mg, 50% yield).

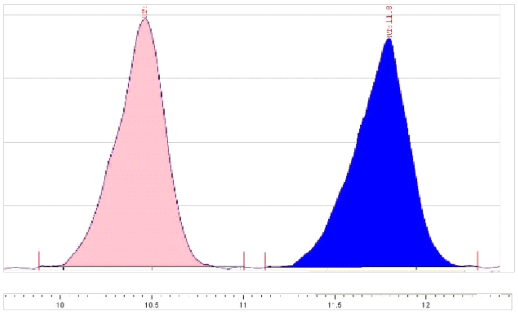
^{1}H NMR (500 MHz, CDCl$_3$) δ 7.37-7.14 (m, 10H), 3.90-3.80 (m, 1H), 3.83 (dd, *J* = 13.6, 4.1 Hz, 1H), 2.69-2.60 (m, 3H), 1.91-1.80 (m, 1H), 1.78-1.65 (m, 1H), 1.64-1.51(m, 2H), 1.48 (s, 1H). **^{13}C NMR** (150 MHz, CDCl$_3$) δ 142.52, 138.65, 129.56, 128.74, 128.57, 128.46, 126.64, 125.89, 72.67, 44.24, 36.55, 36.01, 27.75. **IR** (neat) ν_{max} 3405.14 (br,s), 3083.98 (w), 3061.19 (w), 3025.99 (m), 2919.73 (w), 2857.89 (w), 1602.16 (w), 1494.75 (m), 1453.54 (m), 1081.23 (m), 1048.45 (m), 1030.25 (m), 746.25 (s), 698.64 (s), 494.02 (w) cm^{-1}. **HRMS** (DART) for $C_{17}H_{24}NO$ $[M+NH_4]^+$: Calc'd: 258.1858, found: 258.1864. **$[\alpha]_D^{20}$** = -4.35 (*c* = 0.86, CHCl$_3$, *l* = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

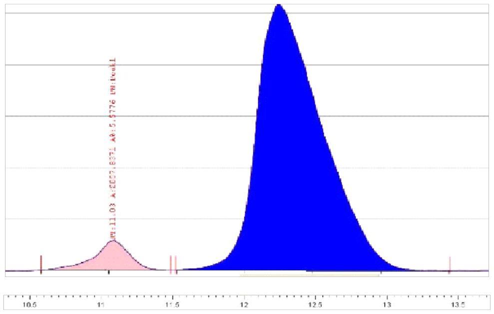
Chiral SFC (Chiracel OJ-H, 9% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-1,5-diphenylpentan-2-ol.

Racemic Material

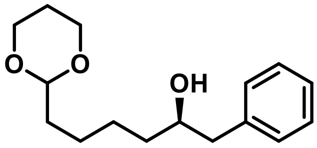


Peak No	% Area	Area	RT (min)
1	49.8175	3788.1127	10.46
2	50.1825	3815.8682	11.85
Total:	100	7603.9809	

Enantioenriched Material



Peak No	% Area	Area	RT (min)
1	5.5776	2057.8371	11.03
2	94.4224	34836.7459	12.25
Total:	100	36894.583	



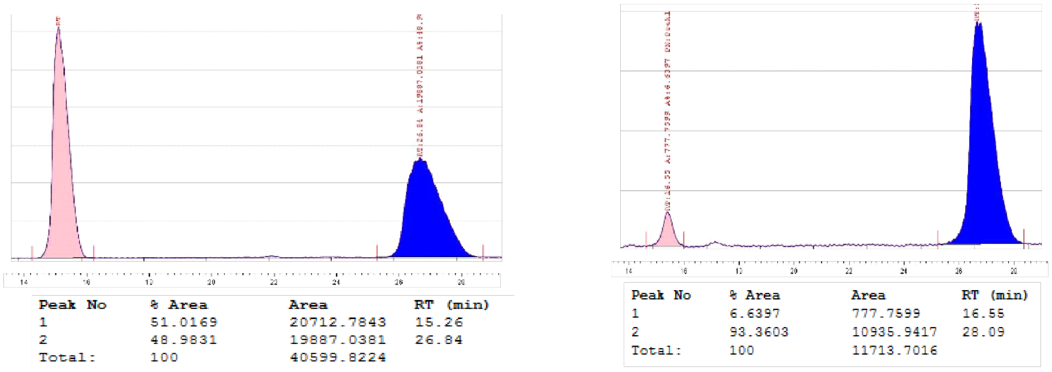
(*R*)-6-(1,3-dioxan-2-yl)-1-phenylhexan-2-ol (17). The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), 2-(but-3-en-1-yl)-1,3-dioxane (**S-14**) (34.1 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), iodobenzene (40.8 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (30% EtOAc in hexanes, stained with CAM) to afford a colorless oil (33.4 mg, 63% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41-7.12 (m, 5H), 4.51 (t, *J* = 5.1 Hz, 1H), 4.09 (dd, *J* = 12.2, 4.9 Hz, 2H), 3.83-3.72 (m, 1H), 3.75 (td, *J* = 12.2, 2.4 Hz, 2H), 2.81 (dd, *J* = 13.5, 4.2 Hz, 1H), 2.64 (dd, *J* = 13.5, 8.3 Hz, 1H), 2.14-2.01 (m, 1H), 1.73-1.24 (m, 9H).**^{13}(150** MHz, $CDCl_3$) δ 138.71, 129.54, 128.65, 126.54, 102.40, 72.63, 67.01, 44.19, 36.81, 35.27, 25.97, 25.74, 24.07. **IR** (neat) ν_{max} 3451.27 (br, w), 2929.58 (s), 2855.69 (s), 1454.85 (w), 1403.76 (w), 1377.78 (w), 1240.40 (w), 1144.35 (s), 1093.42 (m), 1029.47 (m), 997.56 (m), 747.24 (w), 700.91 (m) cm^{-1}. **HRMS** (DART) for $C_{16}H_{25}O_3$ $[M+H]^+$: Calc'd: 265.1804, found: 265.1796. $[\alpha]_D^{20}$ = -5.473 (*c* = 1.90, $CHCl_3$, *l* = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

Chiral SFC (Chiracel OD-H, 2% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-6-(1,3-dioxan-2-yl)-1-phenylhexan-2-ol.

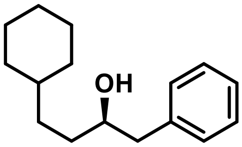


Racemic Material

Peak No	% Area	Area	RT (min)
1	51.0169	20712.7843	15.26
2	48.9831	19887.0381	26.84
Total:	100	40599.8224	

Enantioenriched Material

Peak No	% Area	Area	RT (min)
1	6.6397	777.7599	16.55
2	93.3603	10935.9417	28.09
Total:	100	11713.7016	



(*R*)-4-cyclohexyl-1-phenylbutan-2-ol (18). The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20

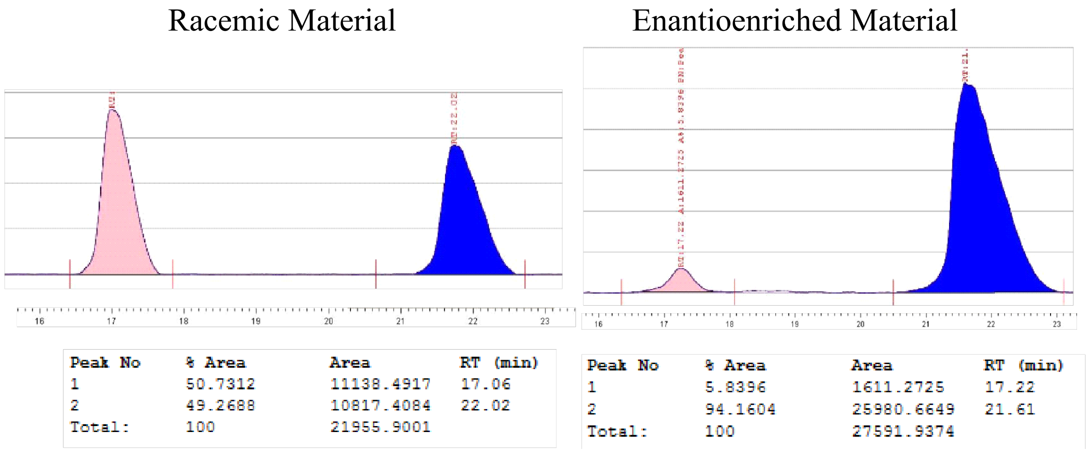
equiv.), vinylcyclohexane (26.4 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), iodobenzene (40.8 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (50% DCM in hexanes, stained with CAM) to afford a white solid (27.0 mg, 58% yield).

^{1}H NMR (500 MHz, CDCl$_3$) δ 7.32-7.20 (m, 5H), 3.82-3.75 (m, 1H), 2.84 (dd, *J* = 13.6, 4.2 Hz, 1H), 2.65 (dd, *J* = 13.6, 8.4 Hz, 1H), 1.75-0.80 (m, 15H). **^{13}C NMR** (150 MHz, CDCl$_3$) δ 138.81, 129.56, 129.55, 128.68, 128.67, 126.56, 73.18, 44.17, 44.15, 37.92, 34.33, 33.64, 33.57, 33.46, 26.84, 26.55, 26.53. **IR** (neat) ν_{max} 3364.05 (br, w), 2920.43 (s), 2849.78 (m), 1495.12 (w), 1450.29 (m), 1079.83 (w), 1030.64 (m), 741.30 (w), 699.12 (m) cm^{-1}. **HRMS** (DART) for $C_{16}H_{28}NO$ $[M+NH_4]^+$: Calc'd: 250.2171, found: 250.2173. **$[\alpha]_D^{20}$** = -8.394 (*c* = 1.42, CHCl$_3$, *l* = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

Chiral SFC (Chiracel OD-H, 2% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-4-cyclohexyl-1-phenylbutan-2-ol.

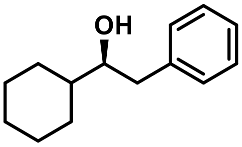


Racemic Material

Peak No	% Area	Area	RT (min)
1	50.7312	11138.4917	17.06
2	49.2688	10817.4084	22.02
Total:	100	21955.9001	

Enantioenriched Material

Peak No	% Area	Area	RT (min)
1	5.8396	1611.2725	17.22
2	94.1604	25980.6649	21.61
Total:	100	27591.9374	

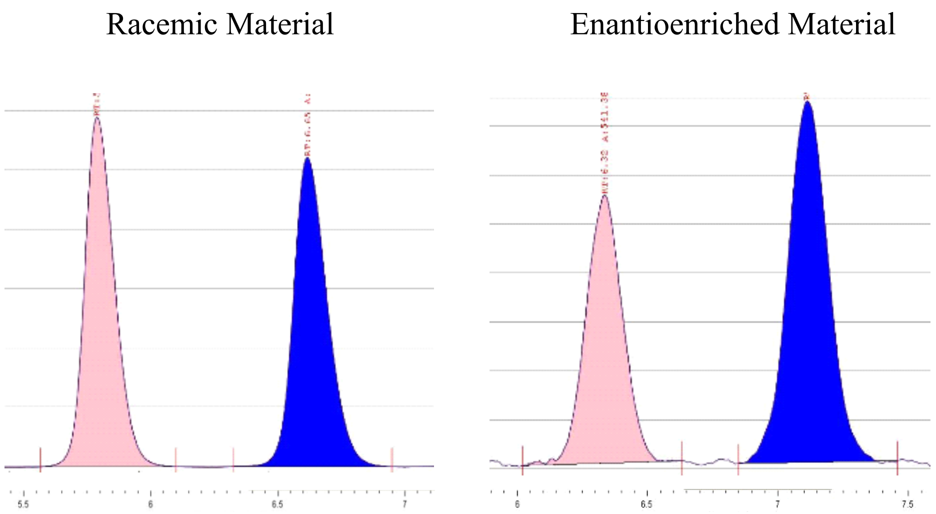


(*R*)-1-cyclohexyl-2-phenylethan-1-ol (19). The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), cyclohexene (19.7 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), iodobenzene (40.8 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (50% DCM in hexanes, stained with CAM) to afford a white solid (20.4 mg, 50% yield). Spectral data matches previously published results.[16]The optical rotation and SFC traces obtained support the assignment of absolute configuration $[\alpha]_D^{20}$ = -3.839 (c = 1.00, CHCl$_3$, l = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

Chiral SFC (Chiracel OD-H, 5% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-1-cyclohexyl-2-phenylethan-1-ol.



Racemic Material

Enantioenriched Material

```
Peak No     % Area      Area        RT (min)
1           50.048      4701.3726   5.84
2           49.952      4692.3455   6.65
Total:      100         9393.7181
```

```
Peak No     % Area      Area        RT (min)
1           40.2339     541.3831    6.32
2           59.7661     804.2063    7.1
Total:      100         1345.5894
```

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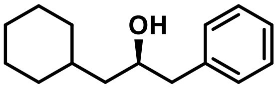
[16] Zhang, L.; Lovinger, G. J.; Edelstein, E. K.; Szymaniak, A. A.; Chierchia, M. P.; Morken, J. P. *Science* **2015**, *351* (6268), 70.



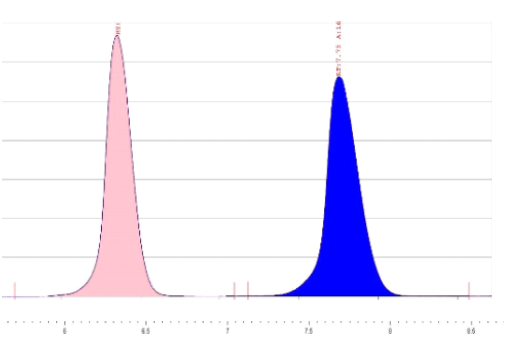
(*R*)-1-cyclohexyl-3-phenylpropan-2-ol (20). The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), methylenecyclohexane (23.1 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), iodobenzene (40.8 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (50% DCM in hexanes, stained with CAM) to afford a white solid (27.0 mg, 58% yield). **^{1}H NMR** (500 MHz, CDCl$_3$) δ 7.38-7.25 (m, 2H), 7.28-7.15 (m, 3H), 4.02-3.90 (m, 1H), 2.81 (dd, *J* = 13.6, 4.1 Hz, 1H), 2.63 (dd, *J* = 13.6, 8.4 Hz, 1H), 1.85-1.74 (m, 1H), 1.75-1.60 (m, 4H), 1.57-1.08 (m, 8H) 1.03-0.79 (2H). **^{13}C NMR** (126 MHz, CDCl$_3$) δ 138.61, 129.40, 128.52, 126.41, 70.02, 44.69, 44.62, 34.17, 32.87, 26.59, 26.37, 26.19. : **IR** (neat) ν_{max} 3399.8 (br, s), 3027.2 (w, m), 2920.4 (s), 2850 (s), 1601.1 (w), 1495.1 (s), 1134.4 (s), 1077 (s), 745 (s), 699 (s), 523 (w, s). **HRMS** (DART) for $C_{15}H_{21}$ [M+H-H$_2$O]$^+$: Calc'd:201.1643, found 201.1650: 201.165. **$[\alpha]_D^{20}$** = .9333 (*c* = 1.00, CHCl$_3$, *l* = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

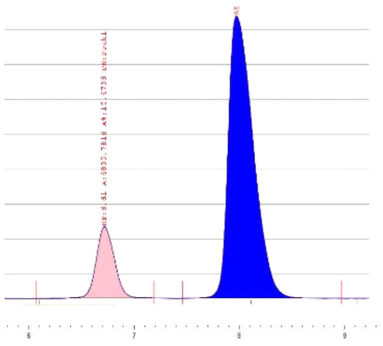
Chiral SFC (Chiracel OD-H, 5% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-4-cyclohexyl-1-phenylbutan-2-ol.

Racemic Material

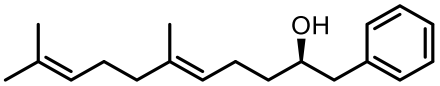


Peak No	% Area	Area	RT (min)
1	49.072	15475.7004	6.36
2	50.928	16061.0519	7.75
Total:	100	31536.7523	

Enantioenriched Material



Peak No	% Area	Area	RT (min)
1	15.4736	4835.7816	6.61
2	84.5264	26416.0117	7.86
Total:	100	31251.7933	



***(R,E)*-6,10-dimethyl-1-phenylundeca-5,9-dien-2-ol (21).** The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), (E)-4,8-dimethylnona-1,3,7-triene (**S-18**) (36 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), iodobenzene (40.8 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (50% DCM in hexanes, stained with CAM) to afford a colorless oil (21.2 mg, 39% yield).

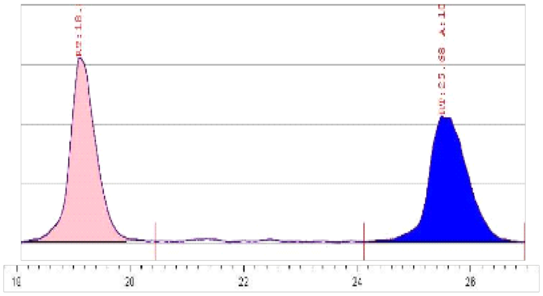
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.35-7.20 (m, 5H), 5.18-5.10 (m, 1H), 5.12-5.05 (m, 1H), 3.89-3.79 (m, 1H), 2.83 (dd, J = 13.6, 4.3 Hz, 1H), 2.67 (dd, J = 13.6, 8.3 Hz, 1H), 2.25-1.97 (m, 6H), 1.68 (s, 3H), 1.64-1.53 (m, 8H). **^{13}C NMR** (150 MHz, CDCl$_3$) δ 138.78, 135.91, 131.57, 129.58, 128.67, 126.56, 124.40, 124.04, 72.56, 44.21, 39.87, 36.87, 26.81, 25.84, 24.48, 17.84, 16.18. **IR** (neat) ν_{max} 3398.31 (br, w), 3027.30 (w), 2963.82 (m), 2923.30 (s), 2854.82 (m), 1495.43 (w), 1452.74 (m), 1376.82 (w), 1081.10 (w), 740.88 (w), 699.60 (w) cm^{-1}. **HRMS** (DART) for $C_{19}H_{29}O$ $[M+H]^+$: Calc'd: 273.2218, found: 273.2222. **$[\alpha]_D^{20}$** = – 2.695 (c = 1.63, CHCl$_3$, l = 50 mm).

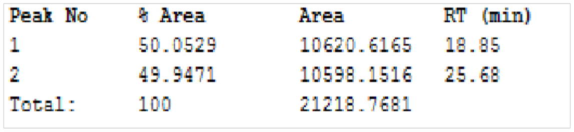
Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

Chiral SFC (Chiracel OD-H, 2% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R,E)-6,10-dimethyl-1-phenylundeca-5,9-dien-2-ol.

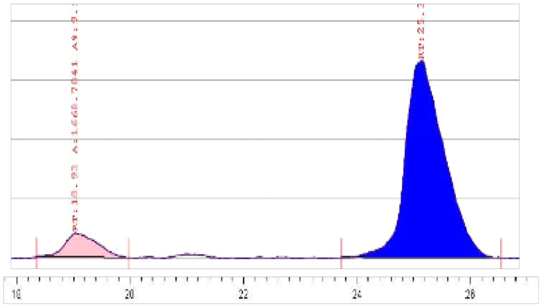
Racemic Material

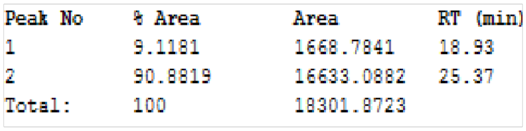




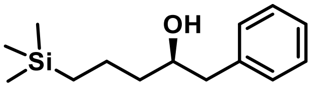
Peak No	% Area	Area	RT (min)
1	50.0529	10620.6165	18.85
2	49.9471	10598.1516	25.68
Total:	100	21218.7681	

Enantioenriched Material





Peak No	% Area	Area	RT (min)
1	9.1181	1668.7841	18.93
2	90.8819	16633.0882	25.37
Total:	100	18301.8723	

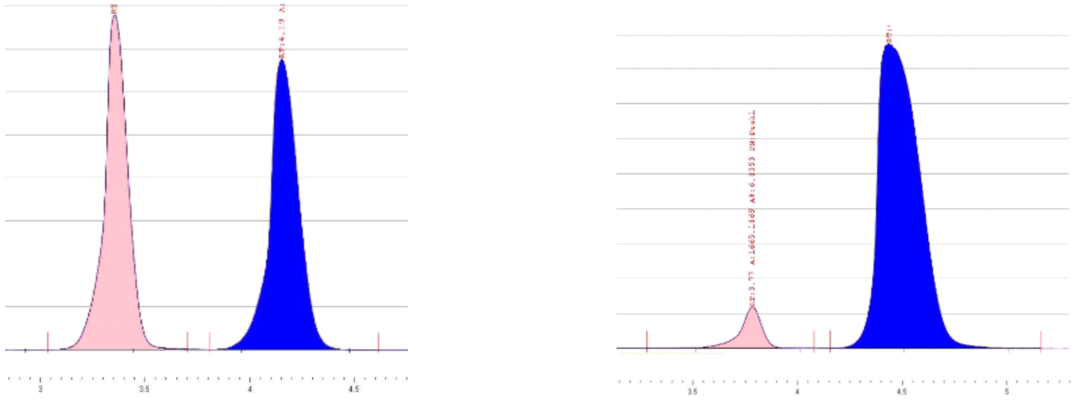


(*R*)-1-phenyl-5-(trimethylsilyl)pentan-2-ol (22). The reaction was performed according to the general **procedure B** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), allyltrimethylsilane (27.4 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), iodobenzene (40.8 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (60% DCM in hexanes, stained with CAM) to afford a colorless oil (26.1 mg, 55% yield).
^{1}H NMR (600 MHz, CDCl$_3$) δ 7.7.34-7.30 (m, 2H), 7.25-7.21 (m, 3H), 3.88-3.80 (m, 1H), 2.82 (dd, *J* = 13.6, 4.3 Hz, 1H), 2.66 (dd, *J* = 13.4, 8.4 Hz, 1H), 1.63-1.43 (m, 4H), 1.43-1.34 (m, 1H), 0.66-0.42 (m, 2H), -0.01 (s, 9H).**^{13}C NMR** (126 MHz, CDCl$_3$) δ 138.62, 129.40, 128.52, 126.41, 72.40, 44.15, 40.74, 20.17, 16.71, -1.66. **IR** (neat) ν_{max} 3404.2 (br, s) 3028.4 (w, s), 2950.1 (s), 2950 (s), 2858.1 , 1495.0 (w, m), 1409.3 (w, s), 1247.2 (s), 1173.1 (s), 861.9 (m), 740.5 (s), 698.3 (s) cm^{-1}. **HRMS** (DART) for $C_{14}H_{28}OSiN$ $[M+NH_4]^+$: Calc'd: 254.1953, found: 254.1940 **$[\alpha]_D^{20}$** = -1.741 (*c* = 1.00, CHCl$_3$, *l* = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure B** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

Chiral SFC (Chiracel OJ-H, 2% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-1-phenyl-5-(trimethylsilyl)pentan-2-ol.



Racemic Material

```
Peak No     % Area       Area         RT (min)
1           48.6805      11888.1196   3.41
2           51.3195      12532.593    4.19
Total:      100          24420.7126
```

Enantioenriched Material

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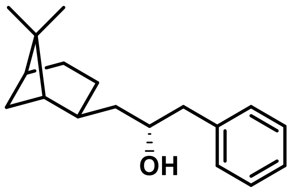
Peak No     % Area       Area         RT (min)
1           6.4353       1665.1469    3.77
2           93.5647      24209.8898   4.43
Total:      100          25875.0367
```



(*R*)-1-((1*S*,2*S*,5*S*)-6,6-dimethylbicyclo[3.1.1]heptan-2-yl)-3-phenylpropan-2-ol (23). The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), (1*S*)-(−)-β-pinene (32.7 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), iodobenzene (40.8 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (70% DCM in hexanes, stained with CAM) to afford an inseparable mixture of diastereomers as a colorless oil (30.5 mg, 59% yield).

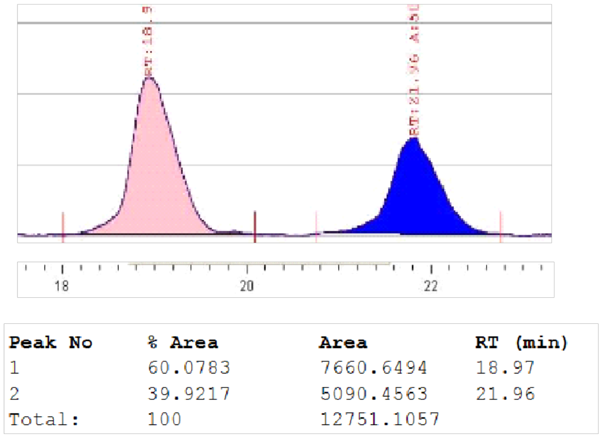
^{1}**H NMR** (500 MHz, CDCl$_3$) Major diastereomer: δ 7.35-7.19 (m, 5H), 3.91-3.83 (m, 1H), 2.83 (dd, *J* = 13.5, 4.5 Hz, 1H), 2.63 (dd, *J* = 13.5, 8.4 Hz, 1H), 2.37-2.22 (m, 2H), 2.07-1.80 (m, 5H), 1.71-1.39 (m, 4H), 1.18 (s, 3H), 0.96 (s, 3H). Minor diastereomer: δ 7.35-7.19 (m, 5H), 3.91-3.83 (m, 1H), 2.82 (dd, *J* = 13.6, 4.8 Hz, 1H), 2.63 (dd, *J* = 13.6, 8.1 Hz, 1H), 2.37-2.22 (m, 2H), 2.07-1.80 (m, 5H), 1.71-1.39 (m, 4H), 1.20 (s, 3H), 0.99 (s, 3H). ^{13}**C NMR** (150 MHz, CDCl$_3$) Major diastereomer: δ 138.70, 129.56, 128.67, 126.55, 71.24, 45.83, 44.84, 44.33, 41.61, 38.81, 37.83, 33.64, 28.34, 26.65, 23.35, 23.12. Minor diastereomer: δ 138.70, 129.56, 128.67, 126.55, 70.93, 47.48, 45.33, 44.91, 38.86, 37.37, 33.89, 31.73, 26.57, 23.42, 22.79, 22.19. **IR** (neat) ν_{max} 3357.47 (br, s), 2903.85 (s), 1945.09 (w), 1467.93 (m), 1452.88 (m), 1382.91 (w), 1365.42 (w), 1080.38 (m), 1032.04 (m), 744.04 (s), 698.40 (s), 601.25 (w), 534.46 (w), 493.51 (w) cm^{-1}. **HRMS** (DART) for $C_{18}H_{27}O$ [M+H-H$_2$O]$^+$: Calc'd: 241.1956, found: 241.196. **$[\alpha]_D^{20}$** = -14.7488 (*c* = 1.92, CHCl$_3$, *l* = 50 mm).

Analysis of Stereochemistry:

The mixture of diastereomers was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Hydroboration has previously been shown to occur from the face opposite to *gem*-dimethyl group of **β-pinene**.[17]Absolute stereochemistry was assigned by analogy (see product **34, 19**).

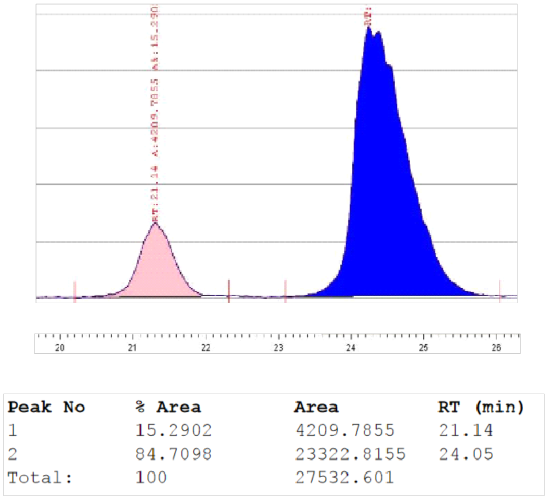
Chiral SFC (Chiracel OD-H, 2% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-1-((1S,2S,5S)-6,6-dimethylbicyclo[3.1.1]heptan-2-yl)-3-phenylpropan-2-ol.

Diastereomic Mixture

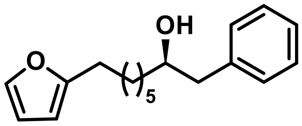


Peak No	% Area	Area	RT (min)
1	60.0783	7660.6494	18.97
2	39.9217	5090.4563	21.96
Total:	100	12751.1057	

Diastereoenriched Material



Peak No	% Area	Area	RT (min)
1	15.2902	4209.7855	21.14
2	84.7098	23322.8155	24.05
Total:	100	27532.601	



(*R*)-8-(furan-2-yl)-1-phenyloctan-2-ol (24). The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), (32.7 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), 2-(hex-5-enyl)furan (**S-21**) (36.1 mg, 0.24 mmol, 1.20 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg,

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[17] G. Zweifel, H. Brown, *J. Am. Chem. Soc.* **1964**, *86*, 393.

0.012 mmol, 0.060 equiv.) in THF (0.4 mL), iodobenzene (40.8 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (10% EtOAc in hexanes, stained with CAM) to afford a yellow oil (36.8 mg, 73% yield).

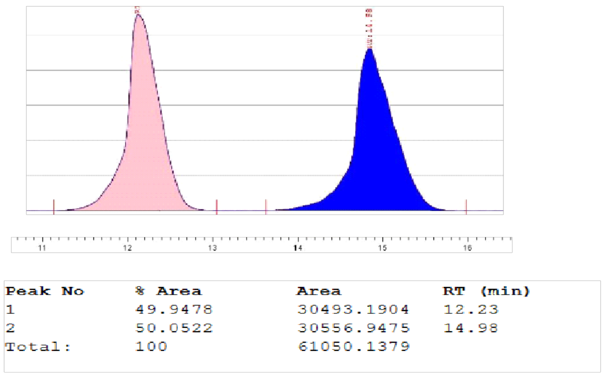
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41-7.06 (m, 6H), 6.28 (dd, *J* = 3.2, 1.9 Hz, 1H), 5.98 (d, *J* = 3.2 Hz, 1H), 3.90-3.77 (m, 1H), 2.83 (dd, *J* = 13.5, 4.2 Hz, 1H), 2.69-2.58 (m, 3H), 1.74-1.25 (m, 10H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 156.63, 140.76, 138.75, 129.55, 128.69, 126.58, 110.16, 104.69, 72.78, 44.22, 36.92, 29.47, 29.24, 28.10, 28.06, 25.77. **IR** (neat) ν_{max} 3388.75 (br, w), 3026.89 (w), 2927.20 (s), 2855.02 (m), 1597.52 (w), 1506.89 (m), 1495.34 (m), 1453.85 (m), 1146.13 (m), 1076.43 (m), 1029.92 (m), 1006.94 (s), 922.05 (w), 884.47 (w), 852.54 (w), 795.29 (m), 726.11 (s), 699.16 (s), 599.28 (m), 543.22 (w), 505.67 (w) cm^{-1}. **HRMS** (DART) for $C_{18}H_{25}O_2$ $[M+H]^+$: Calc'd:273.1855, found: 273.1845. **$[\alpha]_D^{20}$** = -5.92 (*c* = 3.14, $CHCl_3$, *l* = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

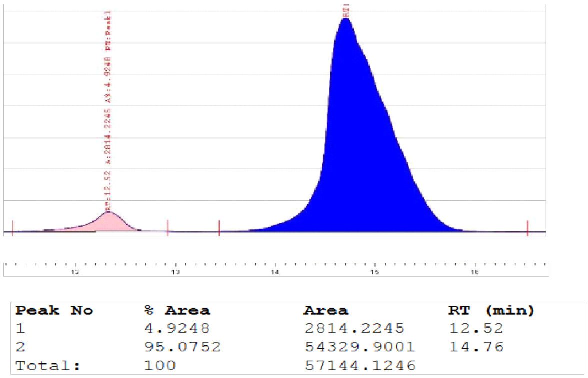
Chiral SFC (Chiracel OJ-H, 5% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-8-(furan-2-yl)-1-phenyloctan-2-ol.

Racemic Material



```
Peak No     % Area      Area          RT (min)
1           49.9478     30493.1904    12.23
2           50.0522     30556.9475    14.98
Total:      100         61050.1379
```

Enantioenriched Material



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Peak No     % Area      Area          RT (min)
1           4.9248      2814.2245     12.52
2           95.0752     54329.9001    14.76
Total:      100         57144.1246
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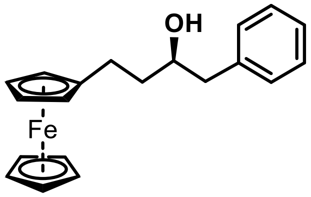


(*R*)-1-phenyl-4-ferrocenylbutan-2-ol (25). The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20

equiv.), vinylferrocene (50.9mg mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), iodobenzene (40.8 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (10% EtOAc in pentane, stained with CAM) to afford a red oil (36.1 mg, 53% yield).

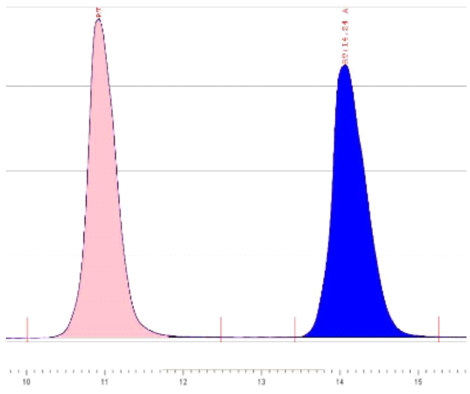
^{1}H NMR (600 MHz, CDCl$_3$) δ 7.36-7.30 (m, 2H), 7.25-7.20 (m, 3H), 4.12-4.03 (m, 9H), 3.88-3.81 (m, 1H), 2.85 (dd, *J* = 13.6, 4.3 Hz, 1H), 2.69 (dd, *J* = 13.5, 8.4 Hz, 1H), 2.56 (ddd, *J* = 14.2, 9.8, 5.8 Hz, 1H) 2.42 (ddd, *J* = 14.3, 9.9, 6.4 Hz, 1H), 1.81-1.71 (m, 2H), 1.52 (s, 1H) **^{13}C NMR** (151 MHz, CDCl$_3$) δ 138.52, 129.55, 128.73, 126.66, 88.88, 72.48, 68.66, 68.29, 68.08, 67.32, 44.25, 38.21, 25.92. **IR** (neat) ν_{max} 3559.7 (w, s), 3400.5 (br, s), 3086.6 (m), 3025.9 (s), 2919.9 (s), 2853.5 (s), 1639.07 (br, s), 1601.7 (w, s), 1494.4 (s), 1470.9 (s), 1410.4 (s), 1154.7 (s), 1080.7 (m), 1080.71 (s), 1041.9 (s), 929.8 (s), 816.6 (s), 743.9 (s), 700.0 (s), 599.4 (w, s), 482.7 (s) cm^{-1}. **HRMS** (DART) for $C_{20}H_{23}OFe$ [M+H]$^+$: Calc'd: 335.1098, found: 335.1113. **$[\alpha]_D^{20}$** = -15.472 (*c* = 1.00, CHCl$_3$, *l* = 50 mm). **$[\alpha]_D^{20}$** = -1.741 (*c* = 1.00, CHCl$_3$, *l* = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

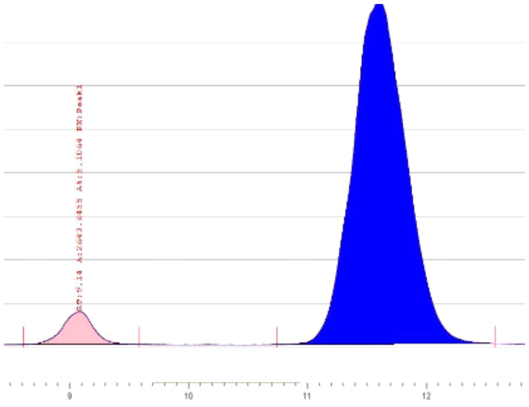
Chiral SFC (Chiracel OJ-H, 13% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-1-phenyl-4-ferrocenylbutan-2-ol.

Racemic Material

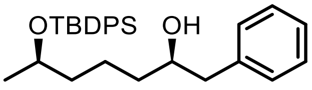


Peak No	% Area	Area	RT (min)
1	49.4303	46023.6816	11.21
2	50.5697	47084.5491	14.24
Total:	100	93108.2307	

Enantioenriched Material



Peak No	% Area	Area	RT (min)
1	5.1064	2643.6455	9.14
2	94.8936	49127.9267	11.64
Total:	100	51771.5722	



(2*R*,6*R*)-6-((*tert*-butyldiphenylsilyl)oxy)-1-phenylheptan-2-ol (26). The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), (R)-*tert*-butyl(pent-4-en-2-yloxy)diphenylsilane (**23**) (77.9 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), iodobenzene (40.8 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (50% DCM in hexanes, stained with CAM) to afford a colorless oil (66.0 mg, 74% yield).

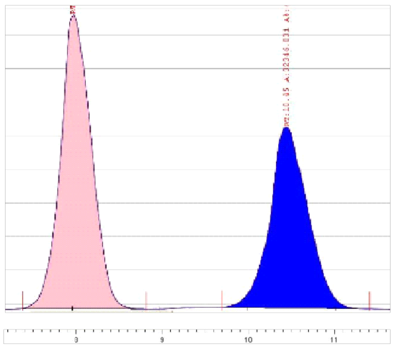
^{1}**H NMR** (500 MHz, CDCl$_3$) δ 7.73-7.67 (m, 4H), 7.45-7.16 (m, 11H), 3.91-3.83 (m, 1H), 3.78-3.69 (m, 1H), 2.77 (dd, J = 13.6, 4.2 Hz, 1H), 2.60 (dd, J = 13.6, 6.8 Hz, 1H), 1.58-1.29 (m, 8 H), 1.15-1.10 (m, 12H).^{13}**C NMR** (150 MHz, CDCl$_3$) δ 136.04, 136.01, 129.60, 129.54, 128.67, 127.61, 127.54, 126.56, 72.67, 69.57, 44.12, 39.48, 36.90, 27.20, 23.37, 21.55, 19.42. **IR** (neat) ν_{max} 3431.69 (br, w), 2930.37 (s), 2856.98 (m), 1472.08 (w), 1454.48 (w), 1427.39 (m), 1476.44 (w), 1134.39 (m), 1109.43 (s), 1075.12 (m), 1029.38 (m), 997.42 (w), 822.01 (w), 739.73 (s), 700.63 (s), 611.44 (m), 597.58 (s) cm^{-1}. **HRMS** (DART) for $C_{29}H_{39}O_2Si$ [M+H]$^+$: Calc'd: 447.2719, found: 447.2723. **[α]$_D^{20}$** = +10.626 (c = 3.67, CHCl$_3$, l = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

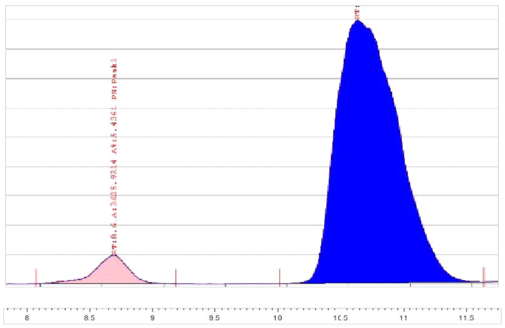
Chiral SFC (Chiracel OJ-H, 5% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (2R,6R)-6-((tert-butyldiphenylsilyl)oxy)-1-phenylheptan-2-ol.

Diastereomic Mixture

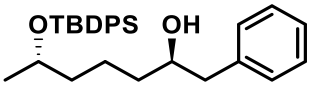


Peak No	% Area	Area	RT (min)
1	57.0781	43014.1003	8.01
2	42.9219	32346.031	10.45
Total:	100	75360.1313	

Diastereoenriched Material



Peak No	% Area	Area	RT (min)
1	5.4341	3625.9214	8.6
2	94.5659	63099.2017	10.57
Total:	100	66725.1231	



(2*R*,6*S*)-6-((*tert*-butyldiphenylsilyl)oxy)-1-phenylheptan-2-ol (27). The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), (S)-*tert*-butyl(pent-4-en-2-yloxy)diphenylsilane (**24**) (77.9 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), iodobenzene (40.8 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (50% DCM in hexanes, stained with CAM) to afford a colorless oil (66.0 mg, 74% yield).

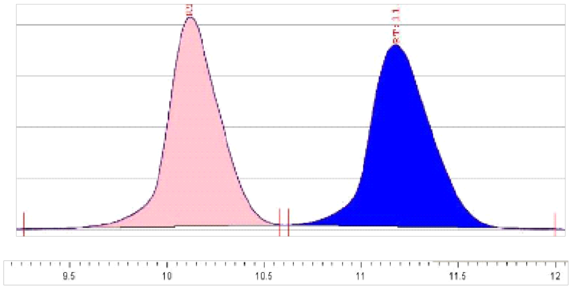
1H NMR (500 MHz, CDCl$_3$) δ 7.75-7.63 (m, 4H), 7.46-7.17 (m, 11H), 3.91-3.84 (m, 1H), 3.79-3.69 (m, 1H), 2.77 (dd, *J* = 13.6, 4.2 Hz, 1H), 2.60 (dd, *J* = 13.6, 8.4 Hz, 1H), 1.57-1.29 (m, 8H), 1.11-1.04 (m, 12H). **^{13}C NMR** (150 MHz, CDCl$_3$) δ 136.05, 136.04, 129.60, 129.56, 129.54, 128.67, 127.62, 127.54, 126.56, 72.63, 69.67, 44.07, 39.49, 36.93, 27.20, 23.40, 21.56, 19.41. **IR** (neat) ν_{max} 3421.77 (br, w), 2930.61 (m), 2856.83 (m), 1495.23 (w), 1472.11 (w) 1427.36 (m), 1376.40 (w), 1361.13 (w), 1134.17 (w), 1109.29 (s), 1078.69 (m), 1049.03 (m), 1028.98 (m), 821.99 (w), 739.64 (m), 700.49 (s), 611.48 (m), 507.32 (m) cm^{-1}. **HRMS** (DART) for $C_{29}H_{39}O_2Si$ $[M+H]^+$: Calc'd: 447.2719, found: 447.2723. **$[\alpha]_D^{20}$** = -16.5218 (*c* = 3.06, CHCl$_3$, *l* = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

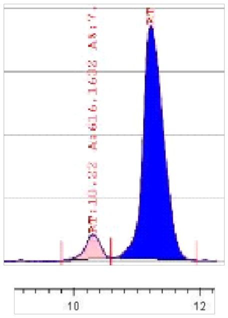
Chiral SFC (Chiracel OJ-H, 5% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (2R,6S)-6-((tert-butyldiphenylsilyl)oxy)-1-phenylheptan-2-ol.

Diastereomic Mixture

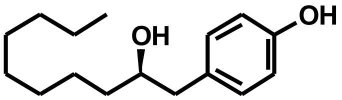


Peak No	% Area	Area	RT (min)
1	49.5546	7242.3644	10.08
2	50.4454	7372.5663	11.11
Total:	100	14614.9307	

Diastereoenriched Material



Peak No	% Area	Area	RT (min)
1	7.2302	616.1632	10.22
2	92.7698	7905.8995	11.1
Total:	100	8522.0627	



(*R*)-4-(2-hydroxydecyl)phenol (32). The reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), 1-octene (26.9 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L14** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), 4-iodophenylboronic acid pinacol ester (66.0 mg, 0.20 mmol, 1.00 equiv.) The crude mixture was purified by silica gel chromatography (20% EtOAc in hexanes, stained with CAM) to afford a white solid (37.8 mg, 75% yield).

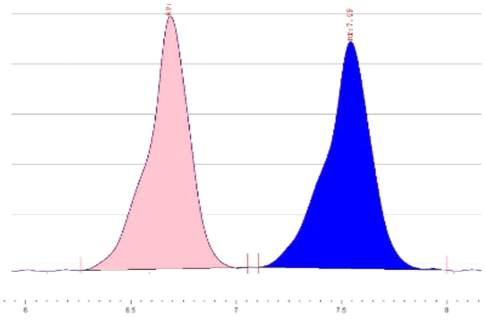
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.05 (d, *J* = 8.4 Hz, 2H), 6.75 (d, *J* = 8.4 Hz, 2H), 6.09-5.41 (br s, 1H), 4.06-3.55 (m, 1H), 2.77 (dd, *J* = 13.8, 4.2 Hz, 1H), 2.56 (dd, *J* = 13.8, 8.5 Hz, 1H), 1.64-1.06 (m, 14H), 0.88 (t, *J* = 6.5 Hz, 3H).**^{13}C NMR** (126 MHz, CDCl$_3$) δ 154.57, 130.65, 130.36, 115.63, 73.21, 43.11, 36.81, 32.00, 29.78, 29.70, 29.40, 25.90, 22.80, 14.24. **IR** (neat) ν_{max} 3339.98 (br, w), 3212.56 (br, w), 3019.54 (w), 2956.78 (m), 2923.21 (s), 2854.02 (m), 1614.50 (w), 1598.03 (w), 1516.78 (s), 1455.78 (m), 1377.79 (w), 1253.90 (s), 1175.83 (w), 1102.56 (w), 1082.13 (w), 906.34 (s), 812.56 (s), 731.23 (s), 649.33 (w) cm^{-1}. **HRMS** (DART) for $C_{16}H_{30}O_2N$ [M+NH4]$^+$: Calc'd: 268.2277, found: 268.228. **[α]$_D^{20}$** = -20.6985 (*c* = 0.93, CHCl$_3$, *l* = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared according to the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **32, 16**).

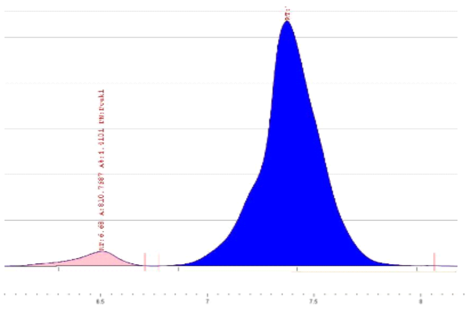
Chiral SFC (Chiracel OJ-H, 8% IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-4-(2-hydroxydecyl)phenol.

Racemic Material



Peak No	% Area	Area	RT (min)
1	50.1544	3558.2552	6.6
2	49.8456	3536.3432	7.49
Total:	100	7094.5984	

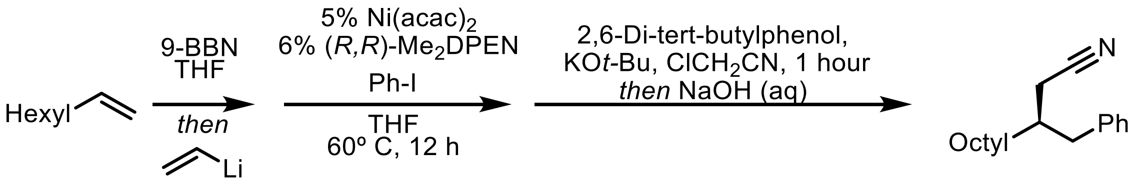
Enantioenriched Material



Peak No	% Area	Area	RT (min)
1	4.4101	810.7687	6.68
2	95.5899	17573.7663	7.46
Total:	100	18384.535	

V. Procedures and Characterization for Transformations of secondary 9-BBN Borates

Cyanomethylation



***(R)*-3-benzylundecanenitrile (28).**

The title compound was prepared from enantiomerically enriched 9-BBN products obtained through the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), 1-octene (26.9 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), and iodobenzene (40.8 mg, 0.20 mmol, 1.00 equiv.), by replacing the oxidation step with a modification of the procedure reported by Brown.[18] After stirring at 60º C overnight the reaction mixture was cooled to 0ºC and a solution of 2,6-di-tert-butylphenol (51.6 mg, 0.25 mmol, 1.25 equiv.) and KO*t*Bu (28.05 mg, 0.25 mmol, 1.25 equiv.) in 400 µL of THF was added to the vial followed by the addition of a solution of ClCH$_2$CN (18.2 mg, 0.24 mmol, 1.20 equiv.) in 200 µL of THF. The reaction mixture was warmed to room temperature and stirred for 1 hour, after which time 2 mL of a 3M NaOH aqueous solution was added. The biphasic mixture was stirrged for 30 minutes and the organic layer was extracted with Et$_2$O 3 times, dried over MgSO$_4$ and concentrated in vacuo. The crude mixture was purified by silica gel chromatography (15% DCM in pentane, stain in PMA) to afford a yellow oil (27.7 mg, 57% yield). **^{1}H NMR** (600 MHz, CDCl$_3$) δ 7.56 – 6.88 (m, 5H), 2.83 (dd, *J* = 13.8, 5.7 Hz, 1H), 2.57 (dd, *J* = 13.8, 9.1 Hz, 1H), 2.28 (dd, *J* = 16.9, 5.3 Hz, 1H), 2.20 (dd, *J* = 16.8, 5.6 Hz, 1H), 2.01 – 1.92 (m, 1H), 1.53 – 1.21 (m, 14H), 0.89 (t, *J* = 7.0 Hz, 3H). **^{13}C NMR** (151 MHz, CDCl$_3$) δ 139.23, 129.18, 128.76, 126.65, 118.73, 39.99, 37.45, 33.57, 31.99, 29.70, 29.62, 29.39, 26.93, 22.81, 21.16, 14.25. **IR** (neat) ν_{max} 3027 (w, s), 3034.8 (s), 2024.3 (s), 2854.4 (s), 2245.2 (s), 1603.1 (w, m), 1496.6 (s), 1377.3 (w, m), 1079.6 (w), 1030.5 (w), 741.1 (s), 701.8 (s) cm^{-1}. **HRMS** (DART) for $C_{18}H_{31}N_2$ [M+NH$_4$]$^+$: Calc'd: 275.2487, found: 275.2475. **$[\alpha]_D^{20}$** = -21.315 (*c* =1.0, CHCl$_3$, *l* = 50 mm).

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[18] H. Brown, H. Nambu,.M. Rogić, *J. Am. Chem. Soc.* **1969**, *91*, 6854.

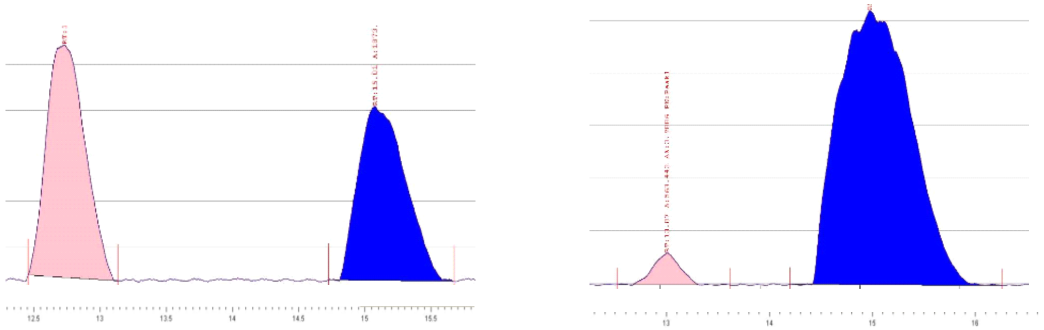
Analysis of Stereochemistry:

Racemic compound was prepared through to same reaction sequence with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

Chiral SFC (Chiracel AD-H, 1 % IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of tert-butyl (R)-3-benzylundecanenitrile.

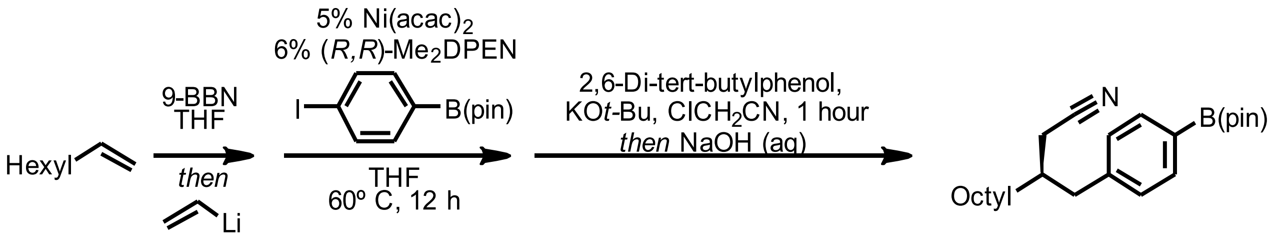
Racemic material

Enantioenriched Material



Peak No	% Area	Area	RT (min)
1	52.1976	2045.4617	12.71
2	47.8024	1873.2305	15.01
Total:	100	3918.6922	

Peak No	% Area	Area	RT (min)
1	3.9886	563.443	13.07
2	96.0114	13563.0475	15.1
Total:	100	14126.4905	



***(R)-3-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)undecanenitrile* (33).**

The title compound was prepared from enantiomerically enriched 9-BBN products obtained through the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), 1-octene (26.9 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)***-

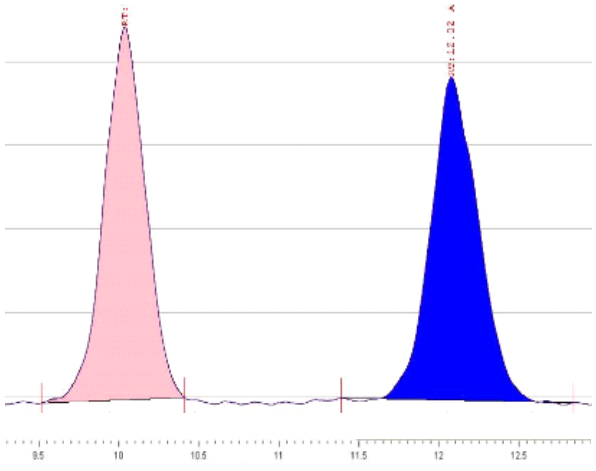
L14 (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), and 4-iodophenylboronic acid pinacol ester (66.0 mg, 0.20 mmol, 1.00 equiv.), by replacing the oxidation step with the procedure as in the synthesis of compound **28**. The crude mixture was purified by silica gel chromatography (70% DCM in hexane, stain in PMA) to afford a yellow oil (35.8 mg, 47% yield). **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.76 (d, *J* = 7.4 Hz, 2H), 7.18 (d, *J* = 7.4 Hz, 2H), 2.83 (dd, *J* = 13.7, 5.7 Hz, 1H), 2.59 (dd, *J* = 13.7, 8.9 Hz, 1H), 2.26 (dd, *J* = 16.8, 5.3 Hz, 1H), 2.18 (dd, *J* = 16.8, 5.5 Hz, 1H), 2.01 – 1.94 (m, 1H), 1.53 – 1.19 (m, 26H), 0.89 (t, *J* = 6.9 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 142.54, 135.24, 128.61, 118.64, 83.90, 40.13, 37.34, 33.52, 31.96, 29.67, 29.58, 29.36, 26.89, 24.99, 22.78, 21.14, 14.23. **11B NMR** (160 MHz, CDCl3) 30.6. **IR** (neat) ν_{max} 2977.06 (w), 2924.63 (m), 2854.60 (w), 2245.59 (w), 1611.72 (m), 1518.02 (w), 1465.45 (w), 1398.25 (m), 1358.18 (s), 1321.23 (m), 1271.48 (m), 1214.01 (w), 1143.48 (s), 1089.36 (s), 1021.79 (m), 962.54 (m), 859.14 (m), 829.74 (w), 736.36 (w), 659.62 (s) cm^{-1}. **HRMS** (DART) for $C_{24}H_{42}N_2O_2B$ $[M+NH_4]^+$: Calc'd: 401.3339, found: 401.3335. $[\alpha]_D^{20}$ = -13.998 (*c* =1.54, $CHCl_3$, *l* = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared through to same reaction sequence with 2,2'-bipy (6 mol%) as the ligand. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

Chiral SFC (Chiracel AD-H, 3 % IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of (R)-3-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)undecanenitrile.

Racemic material



```
Peak No      % Area        Area         RT (min)
1            49.9809       3793.2285    10.09
2            50.0191       3796.1337    12.02
Total:       100           7589.3622
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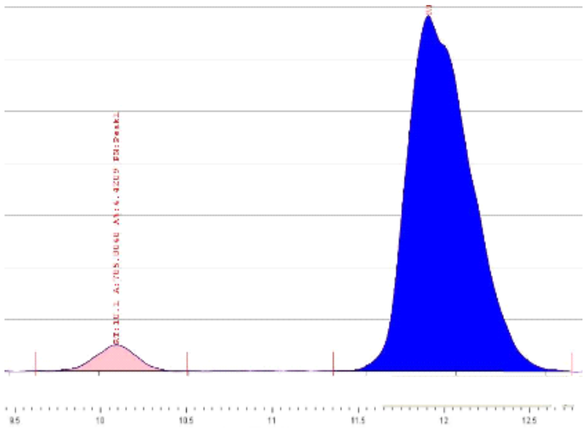
Enantioenriched Material



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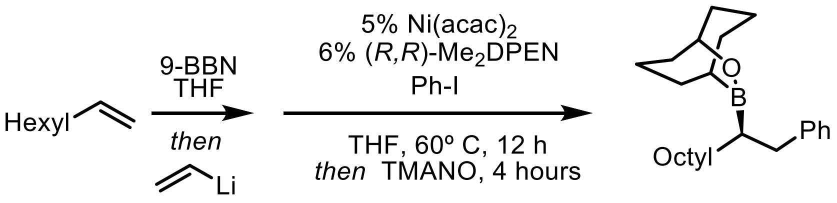
Peak No     % Area      Area          RT (min)
1           4.4209      785.8048      10.1
2           95.5791     16989.1147    11.85
Total:      100         17774.9195
```

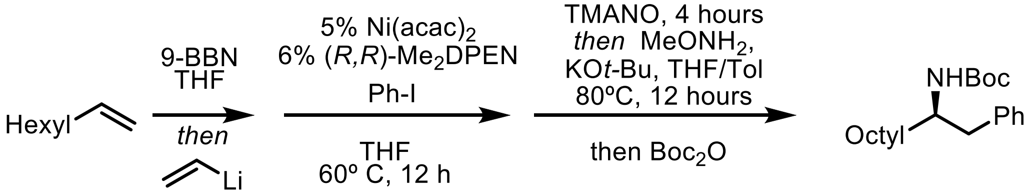
Mono Oxidation



(*R*)-10-(1-phenyldecan-2-yl)-9-oxa-10-borabicyclo[3.3.2]decane (29)

The title compound was prepared from enantiomerically enriched 9-BBN products obtained through the general **procedure A** with modification, using 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), 1-octene (26.9 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), and iodobenzene (40.8 mg, 0.20 mmol, 1.00 equiv.). After stirring overnight at 60ºC, the mixture was brought back into the glovebox where anhydrous trimethylamine oxide (18.78 mg, 0.25 mmol) was added to the vial. The resulting suspension was sealed and stirred vigorously at room temperature for 4 hours. Afterwards, the mixture was filtered through celite and concentrated in vacuo. The material was purified by neutral alumina column chromatography (pentane, stain in CAM) to afford a colorless oil (58.9 mg, 83% yield). **^{1}H NMR** (600 MHz, CDCl$_3$) δ 7.27-7.11 (m, 5H), 4.60-4.49 (m, 1H), 2.69 (dd, *J* = 13.8, 7.8 Hz, 1H), 2.58 (dd, *J* = 13.8, 7.3 Hz, 1H), 1.80-1.16 (m, 27H), 0.88 (s, 3H). **^{13}C NMR** (150 MHz, CDCl$_3$) δ 143.35, 128.99, 128.08, 125.41, 73.39, 37.06, 35.59 (br), 32.11, 32.04, 31.38, 30.78, 30.24, 29.70, 29.65, 29.45, 26.57, 26.36, 24.33 (br), 22.84, 22.34, 22.09, 14.26**.** **^{11}B NMR** (160 MHz, CDCl$_3$) δ 53.30. **IR** (neat) ν_{max} 3061.6 (w, m) 3025.0 (w, s), 2919.9 (s), 2851.8 (s), 1602.4 (w, s), 1494.6 (s), 1413.6 (m), 1364.9 (s), 1337.9 (s), 1285.6 (s), 1160.3 (s) 1020.44 (m), 870.9 (w, s), 697.9 (s), 643.4 (s) cm^{-1}. **HRMS** (DART) for $C_{24}H_{40}BO$ [M+H]$^+$, Calc'd: 355.3172, found:355.3186 **$[\alpha]_D^{20}$** =10.632 (*c* =1.0, CHCl$_3$, *l* = 50 mm).

Amination



***tert*-butyl (*R*)-(1-phenyldecan-2-yl)carbamate (30)**

The title compound was prepared from enantiomerically enriched 9-BBN products obtained through the general **procedure A** with modification, using with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), 1-octene (26.9 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), and iodobenzene (40.8 mg, 0.20 mmol, 1.00 equiv.). After stirring overnight at 60ºC, the mixture was brought back into the glovebox where anhydrous trimethylamine oxide (18.78 mg, 0.25 mmol) was added to the vial. The resulting suspension was sealed and stirred vigorously at room temperature for 4 hours. The solvent was then removed in vacuo using a schlenk line. The crude oil was brought back into glove box and KO*t*Bu (123.43 mg, 1.10 mmol, 5.50 equiv.) was added along with .8 mL of Toluene. The reaction mixture was sealed with a septum cap and a solution of O-methylhydroxylamine (2.53 M, .474 mL, 1.20 mmol, 6.00 equiv.) in THF was added to the vial at room temperature.

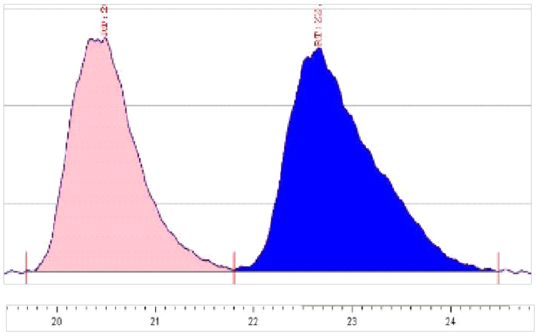
The reaction was stirred at 80°C overnight. Afterwards the mixture was cooled down to room temperature and a solution of di-tert-butyl-dicarbonate in THF (1M solution, 6.5 equiv. 1.3 mL) was added. After having stirred at rt for 4 hours, 1 mL of H$_2$O was added and the water layer was extracted 4 times with EtOAc, dried over MgSO4 and concentrated in vacuo. The product was Isolated by silica gel chromatography (2% EtOAc in pentane, ninhydrin stain) to afford a colorless oil (39.4 mg, 59% i. y.). **^{1}H NMR** (500 MHz, CDCl$_3$) δ 7.30-7.14 (m, 5H), 4.28 (br s, 1H), 3.80 (br s, 1H), 2.75 (br s, 2H), 1.50-1.19 (m, 23H), 0.87 (t, *J* = 6.9 Hz, 3H). **^{13}C NMR** (150 MHz, CD$_3$CN) δ 155.60, 138.51, 129.65, 128.37, 126.31, 79.04, 77.41, 77.16, 76.91, 51.69, 41.51, 34.35, 31.98, 29.65, 29.59, 29.36, 28.53, 26.12, 22.78, 14.22. **IR** (neat) ν_{max} 3368.23 (br, w), 2956.23 (m), 2949.89 (s), 2854.58 (m), 1810.82 (w), 1699.86 (s), 1520.70 (m), 1496.61 (m), 1454.96 (m), 1390.24 (m), 1365.57 (m), 1248.73 (m), 1170.91 (s), 1118.82 (m), 1069.15 (m), 740.32 (w), 699.79 (m) cm^{-1}. **HRMS** (DART) for $C_{21}H_{36}NO_2$ [M+H]$^+$: Calc'd: 334.2746, found: 334.276. **$[\alpha]_D^{20}$** = +6.50 (*c* = 0.84, CHCl$_3$, *l* = 50 mm).

Analysis of Stereochemistry:

Racemic compound was prepared by employing the same reaction sequence with 2,2'-bipy (6 mol%) as the ligands. Absolute stereochemistry was assigned by analogy (see product **34, 19**).

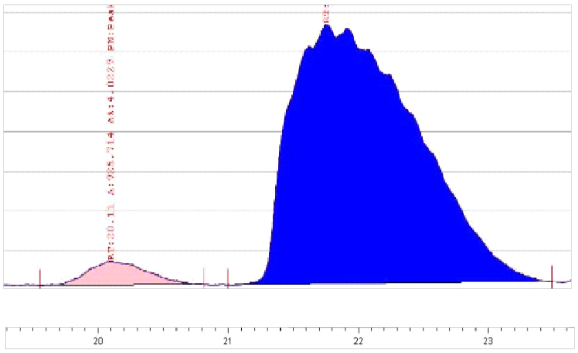
Chiral SFC (Chiracel OD-H, 1 % IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of tert-butyl (R)-(1-phenyldecan-2-yl)carbamate.

Racemic Material



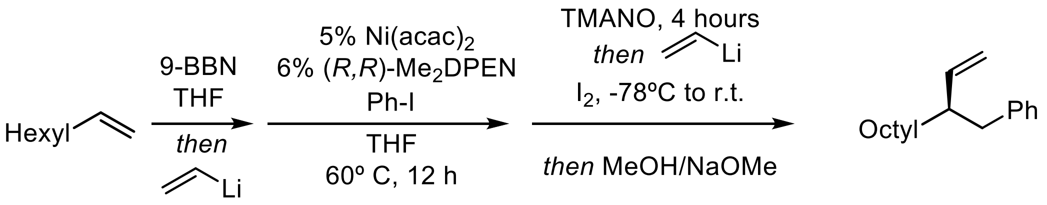
Peak No	% Area	Area	RT (min)
1	45.7469	5763.3965	20.6
2	54.2531	6835.0571	22.78
Total:	100	12598.4536	

Enantioenriched Material



Peak No	% Area	Area	RT (min)
1	4.0229	925.714	20.11
2	95.9771	22085.2978	21.76
Total:	100	23011.0118	

Olefination



(*R*)-(2-vinyldecyl)benzene (*31*)

The title compound was prepared from enantiomerically enriched 9-BBN products obtained through the general **procedure A** with modification, using 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), 1-octene (26.9 mg, 0.24 mmol, 1.20 equiv.), halide-free vinyllithium in THF (0.15 mL, 1.47 M, 0.22 mmol, 1.10 equiv.), a solution of Ni(acac)$_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), and iodobenzene (40.8 mg, 0.20 mmol, 1.00 equiv.). After stirring overnight at 60ºC, the mixture was brought back into the glovebox where anhydrous trimethylamine oxide (18.78 mg, 0.25 mmol) was added to the vial. The resulting suspension was sealed and stirred vigorously at room temperature for 4 hours.

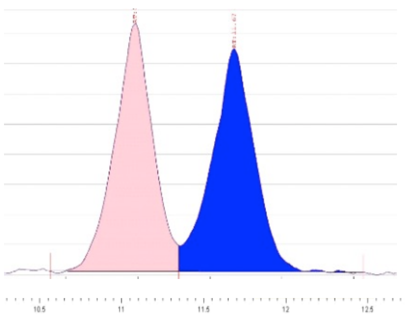
The mixture was cooled to -78° C and vinyllithium (1.65 M, 490 uL, 4.00 equiv.), was added to the vial which was allowed to warm up to room temperature and stir for 10 minutes. The mixture was cooled again to -78º C and Iodine (304.57 mg, 1.20 mmol, 6.00 equiv.) was added in 0.5 mL of THF. This mixture was Stirred at -78° C for 1 hour and then allowed to warm to r.t. for 10 min. The reaction was cooled once more to -78º C. Sodium methoxide (129.66 mg, 2.40 mmol, 133.80 uL) was added in 1.5 mL of MeOH. After warming to R.T. the reaction mixture was stirred for 12 hours. Finally, 1 mL of saturated sodium thiosulfate solution was added to the reaction mixture at 0°C. The water layer was extracted 4 times with Pentane, dried over $MgSO_4$ and concentrated under reduced pressure. The crude product was purified by silica gel chromatography (pentane, stain in CAM) to afford a colorless oil (40.2 mg, 82% yield). **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.29-7.07 (m, 5H), 5.64-5.55 (m, 1H), 3.92 (dd, J = 10.3, 1.8 Hz, 1H), 4.89 (ddd, J = 17.1, 2.0, 0.9 Hz, 1H), 2.65 (dd, J = 13.5, 6.6 Hz, 1H), 2.59 (dd, J = 13.5, 7.6 Hz, 1H), 2.28 (m, 1H), 1.44 – 1.12 (m, 14H), 0.88 (s, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 142.69, 140.87, 129.39, 128.12, 125.79, 114.49, 45.76, 41.96, 34.29, 32.01, 29.81, 29.70, 29.43, 27.27, 22.79, 14.23. **IR** (neat) ν_{max} 3065.1 (w, m), 3027 (s), 2955.4 (s), 2924.3 (s), 2854.1 (s), 1639.0 (w, s), 1495.2 (s), 1495.3 (w), 1378.1 (w, s), 1030.3 (w, s), 993.1 (s), 745.2 (w), 698.3 (s). **HRMS** (DART) for $C_{18}H_{29}$ $[M+H]^+$: Calc'd: 245.2269, found: 245.2261. $[\alpha]_D^{20}$ = 4.840 (c =1.0, $CHCl_3$, l = 50 mm).

Analysis of Stereochemistry:

In order to obtain separation conditions on the SFC, the title compound was submitted to a hydroboration oxidation sequence and the resulting primary alcohol was compared against the racemic product obtained through the same reaction with 2,2'-bipyridine.

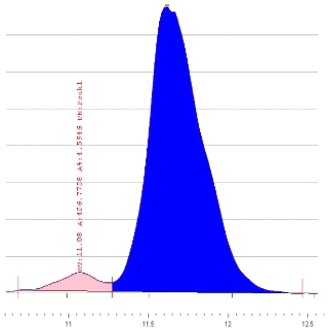
Chiral SFC (Chiracel OJ-H, 2 % IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of tert-butyl (R)-3-benzylundecan-1-ol.

Racemic Material



Peak No	% Area	Area	RT (min)
1	50.0039	3057.1549	10.98
2	49.9961	3056.672	11.67
Total:	100	6113.8269	

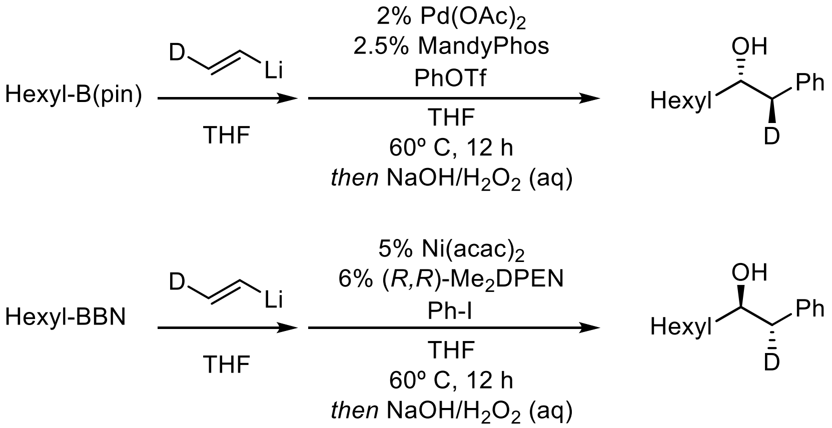
Enantioenriched Material



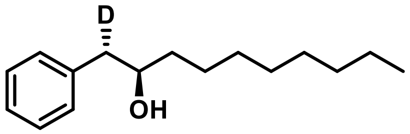
Peak No	% Area	Area	RT (min)
1	4.5946	426.7758	11.08
2	95.4054	8861.79	11.61
Total:	100	9288.5658	

VI. Deuterium-labeling Experiment

Procedure for the Preparation of *Trans*-deuterium-labeled Vinyl Lithium

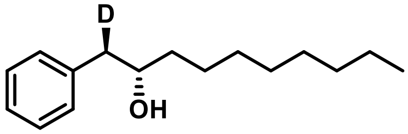


The *trans*-deuterium labeled vinyl lithium was prepared as described in previous reports.[16]



(1*S*,2*R*)-1-phenyldecan-1-*d*-2-ol (34). The cross-couping reaction was performed according to the general **procedure A** with 9-BBN in THF (0.48 mL, 0.5 M, 0.24 mmol, 1.20 equiv.), 1-hexene (20.2 mg, 0.24 mmol, 1.20 equiv.), deuterium labeled vinyllithium in THF (0.14 mL, 1.57 M, 0.22 mmol, 1.10 equiv.), a solution of $Ni(acac)_2$ (2.57 mg, 0.010 mmol, 0.050 equiv.) and ***(R,R)*-L6** (2.88 mg, 0.012 mmol, 0.060 equiv.) in THF (0.4 mL), iodobenzene (40.8 mg, 0.20 mmol, 1.00 equiv.). The crude material was purified by column chromatography (40% DCM in pentane, stain in CAM) to afford a white solid (26 mg, 63% yield). **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.68 – 6.84 (m, 5H), 4.00 – 3.69 (m, 1H), 2.63 (d, J = 8.1 Hz, 1H), 1.61 – 1.18 (m, 11H), 0.89 (t, J = 6.8 Hz, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 138.60, 129.39, 128.52, 126.40, 72.64, 43.70, 36.84, 31.82, 29.30, 25.70, 22.59, 14.05. **IR** (neat) ν_{max} cm^{-1} 3363.48 (br s), 3026.64 (s), 2954.49 (s), 2925.27 (s), 2855.42 (s), 1495.09 (s), 1495.09 (s), 1377.75 (s), 1282.19 (m), 1076.80 (m), 737.64 (m), 699.12 (s). **HRMS** (DART) for $C_{14}H_{20}D$ $[M+H-H_2O]^+$: Calc'd: 190.1706, found: 190.1702. **$[\alpha]_D^{20}$** -3.66 (c =3.92, $CHCl_3$, l = 50 mm).

In order to determine the relative stereochemistry of the product, a parallel reaction with known stereochemical outcome was carried out according to our previously published procedure .[16]



(1*R*,2*S*)-1-phenyldecan-1-*d*-2-ol (S-34). To an oven-dried 2-dram vial equipped with a magnetic stir bar in an Ar-filled glovebox was added alkyl/aryl boronic ester (63.6 mg, 0.30 mmol, 1.0 equiv.) and diethyl ether (0.3 mL), sealed with a septum cap, and removed from the glovebox. The reaction vial was cooled to 0°C, and a deuterium labeled vinyllithium solution (.20 mL, 1.57 M, 0.30 mmol, 1.0 equiv.) was added at 0°C. The reaction vial was allowed to warm to room temperature and stirred for 30 minutes. Then, the solvent was carefully removed under reduced pressure, and the reaction vial was brought back into the glovebox. To a separate oven-dried 2-dram vial equipped with a magnetic stir bar in the glovebox was added $Pd(OAc)_2$ (1.4 mg, 0.006 mmol, 0.02 equiv.), (S_P,S'_P)-1,1′-Bis[bis(4-methoxy-3,5-dimethylphenyl)phosphino]-2,2′-bis[(*R*)-α-(dimethylamino)benzyl]ferrocene (**MandyPhos**, 7.6 mg 0.0072 mmol, 0.024 equiv.), and THF (0.6 mL). The $Pd(OAc)_2$ /(S_P, S_P)-**MandyPhos** solution was allowed to stir for 20 minutes at room temperature. Then the $Pd(OAc)_2$ /(S_P, S_P)- **MandyPhos** solution was transferred into the reaction vial, followed by THF (0.6 mL), and aryl/vinyl triflate (0.33 mmol, 1.10 equiv.) was added. The reaction vial was sealed with a polypropylene cap, taped, and brought out of the glovebox where it was allowed to stir at 60°C for 14 hours. The resulting mixture was cooled to room temperature, filtered through a silica gel plug with diethyl ether, concentrated under reduced pressure. The reaction mixture was diluted with THF (3 mL), cooled to 0°C, 3M NaOH (2 mL) was added, followed by 30% H_2O_2 (1.0 mL), dropwise. The reaction mixture was allowed to warm to room temperature, and was allowed to stir for 4 hours. The reaction mixture was cooled to 0°C and saturated aq. $Na_2S_2O_3$ (3 mL) was added dropwise. The reaction mixture was allowed to warm to room temperature and the aqueous layer was extracted with ethyl acetate (3 x 20 mL). The combined organic layers were dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The crude mixture was purified by silica gel chromatography (40% DCM in pentane, stain in CAM) to afford a white solid (24.0 mg, 58% yield). All spectroscopic data matched those obtained from the Ni catalyzed reaction. $[\alpha]_D^{20}$ = 10.56 (*c* =0.76, $CHCl_3$, *l* = 50 mm). Ni catalyzed $[\alpha]_D^{20}$ -3.66 (*c* =3.92, $CHCl_3$, *l* = 50 mm).

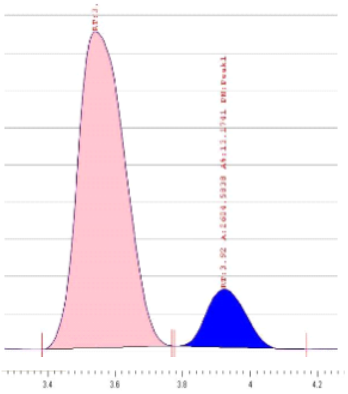
In our previous report,[16] a similar deuterium labelled vinyllithium was used in palladium/Mandyphos catalyzed conjunctive cross-coupling, and it was found to generate products with anti stereochemistry between the hydroxyl group and the deuterium atom, consistent with an anti-migration of the organo lithium reagent with respect to the metal-bound alkene. Thus, by comparing the products of the two reactions (where the diastereotopic benzylic protons of the product are clearly distinguishable by ^{1}H NMR) it was determined that the Ni catalyzed conjunctive cross-coupling yielded the same diastereomer as the products obtained from Palladium catalysis.

Analysis of Stereochemistry:

Racemic compound was prepared through the general **procedure A** with 2,2'-bipy (6 mol%) as the ligand, and non-labeled vinyllithium.

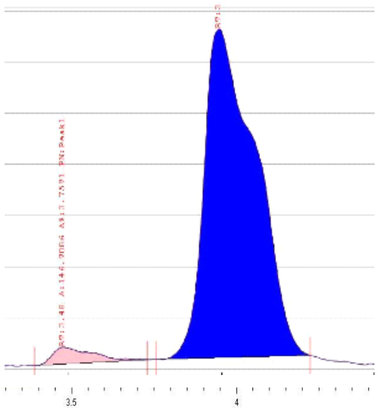
Chiral SFC (Chiracel OD-H, 3 % IPA, 3 mL/min, 100 bar, 35 °C, 210-289 nm) – analysis of tert-butyl (1R,2S)-1-phenyldecan-1-d-2-ol.

S-33



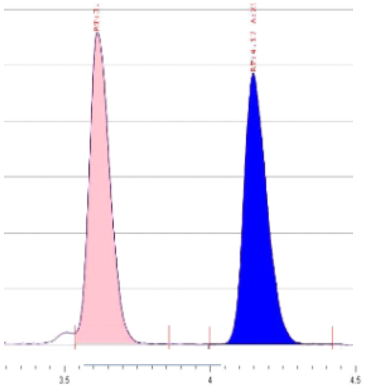
Peak No	% Area	Area	RT (min)
1	86.8259	17165.9122	3.52
2	13.1741	2604.5838	3.92
Total:	100	19770.496	

33



Peak No	% Area	Area	RT (min)
1	3.7591	146.9086	3.48
2	96.2409	3761.1672	3.99
Total:	100	3908.0758	

Racemic material

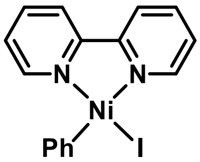


Peak No	% Area	Area	RT (min)
1	50.7244	2991.7195	3.58
2	49.2756	2906.2689	4.17
Total:	100	5897.9884	

Assignment of Absolute Stereochemistry

Comparison of the SFC traces and optical rotation of **32** and **S-32** allowed for the assignment of the absolute stereochemistry for the Ni catalyzed conjunctive cross-coupling products. This assignment was also supported by comparison of the SFC trace and optical rotation of the substrate **16** with those previously reported.[16]

VII Stoichiometric experiments.

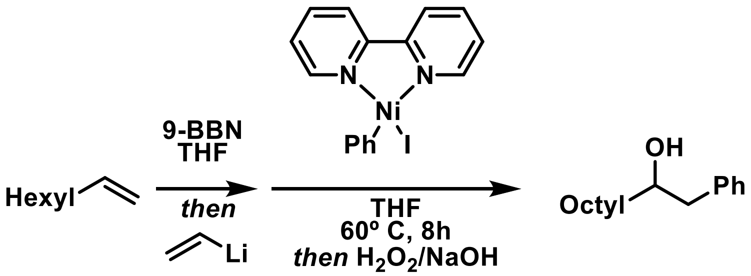


Phenyl(dipyridyl) Nickel Iodide (35)

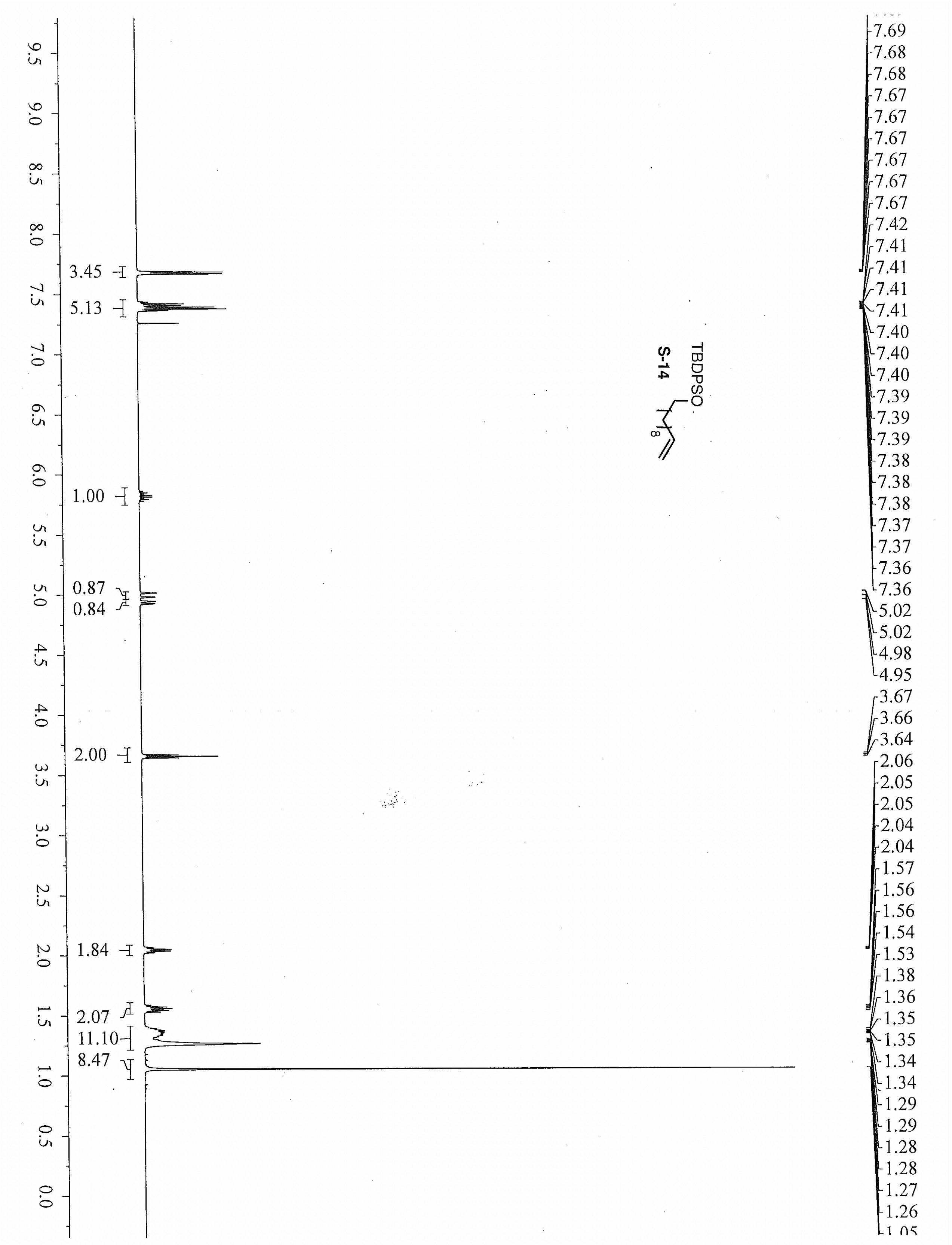
In an argon filled glovebox, to an oven dried scintillation vial equipped with a magneti stirbar was added Bis(cyclooctadiene)nickel(0) (82.5 mg, 0.3 mmol, 1.0 equiv.) 2,2'-bipyridine (46.9 mg, 0.3 mmol, 1.0 equiv.) and 2 mL of THF. The vial was sealed with a septum-cap and brought outside. The dark purple solution was allowed to stir for 3 hours at room temperature, after which the solvent was carefully removed through a Schlenk line under reduced pressure. The solid was then mostly redissolved in 8 mL of pentane (the BipyNiCOD complex is slightly soluble in pentane), and iodobenzene (104.1 mg, 0.51 mmol, 1.7 equiv.) was added the vial. The solution turned cloudy and a dark red precipitate began to form. After stirring for 2 hours the suspension was brought back into the glovebox and allowed to settle. The colorless pentane solution was removed via syringe leaving behind a dark red solid which was triturated 4 times with 2 mL portions of Pentane. The vial was sealed and placed under vacuum overnight to remove any residual solvent, leaving behind the product as a red solid (54 mg, 43% yield). The solid could not be characterized by conventional methods as it was not stable enough in solution to provide clean NMR spectra.

The structure was confirmed through x-ray crystallography. Crystals were grown by vapor diffusion with pentane and 2-methyltetrahydrofuran at -18ºC. The metal complex was dissolved in anhydrous 2-methyltetrahydrofuran in a glovebox, filtered through celite.

Stoichiometric Conjunctive Cross-Coupling Reactions.



In an argon filled glovebox, in an oven-dried 2-dram vial equipped with a magnetic stirbar, 1-octene (13.5 mg, 0.105 mmol, 1.05 equiv.) was added to a solution of 9-BBN in THF (0.21 mL, 0.5 M, 0.105 mmol, 1.05 equiv.) at 0º C. The reaction mixture was allowed to warm to room temperature and stir for an additional 3 hours before being cooled back to 0℃. Vinyllithium in THF (68 µL, 1.47 M, 0.1 mmol, 1.00 equiv.) was added to the reaction mixture which was then warmed to room temperature and stirred for 5 minutes**.** Phenyl(dipyridyl)nickel iodide **(34) (**41.9 mg, 0.1 mmol, 1.0 equiv.) was added to the vial which was then sealed with a septa-cap, brought outside and stirred for 8 hours at 60ºC. The reaction mixture was then cooled to 0℃, and 30% H_2O_2 (0.25 mL) were added along with 3 M NaOH (0.25 mL). The reaction mixture was allowed to warm up to room temperature and stirred for 3 hours, aq. $Na_2S_2O_3$ (0.5 mL) was then added to quench the reaction mixture. The aqueous phase was extracted with Et_2O (2 x 2 mL), followed by EtOAc (2 x 2 mL). The combined organic layer was dried over $MgSO_4$, filtered, concentrated under reduced pressure, and subsequently purified via silica gel column chromatography to provide ***1*** as a colorless oil (10.5 mg, 45% yield).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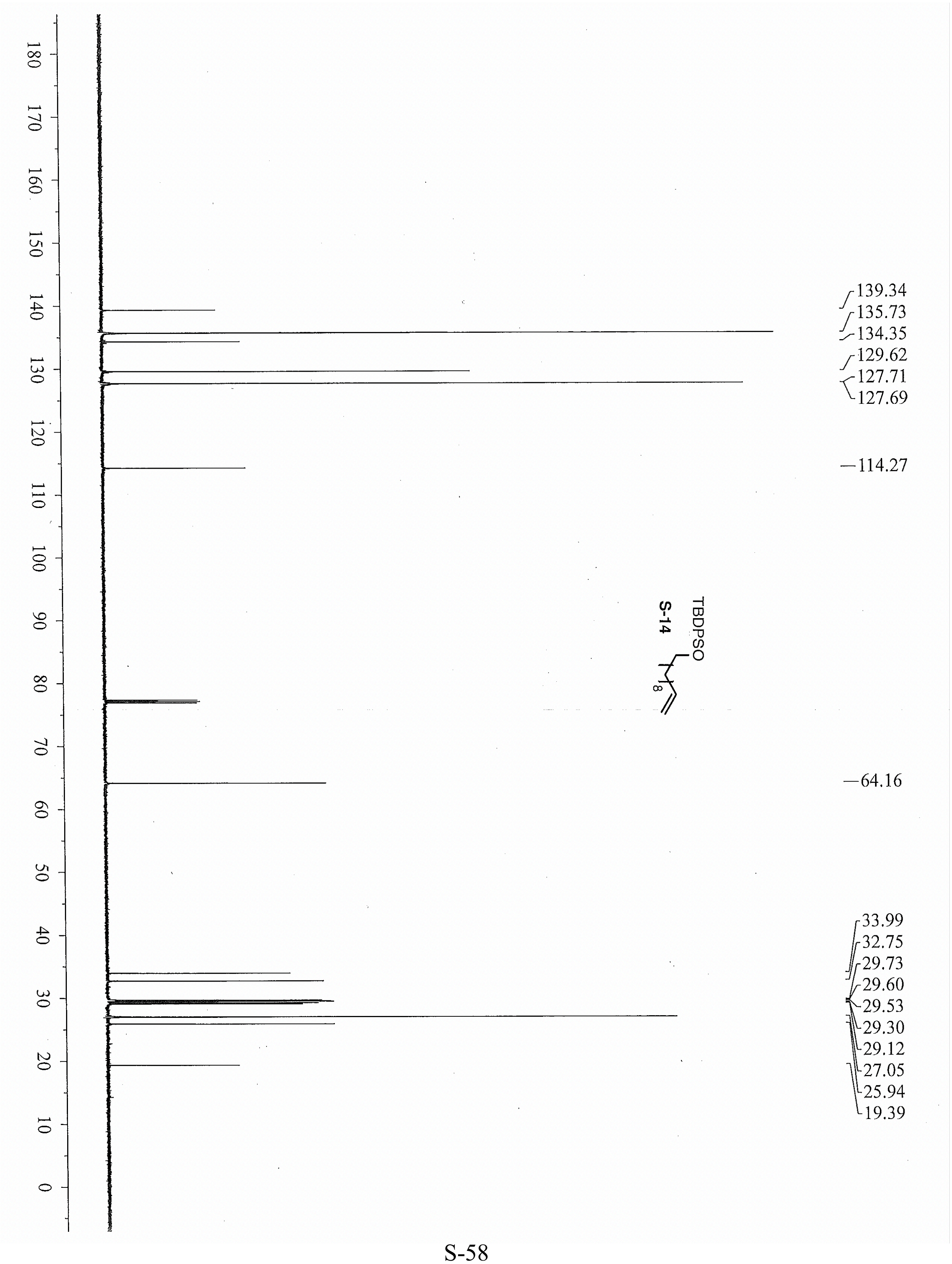
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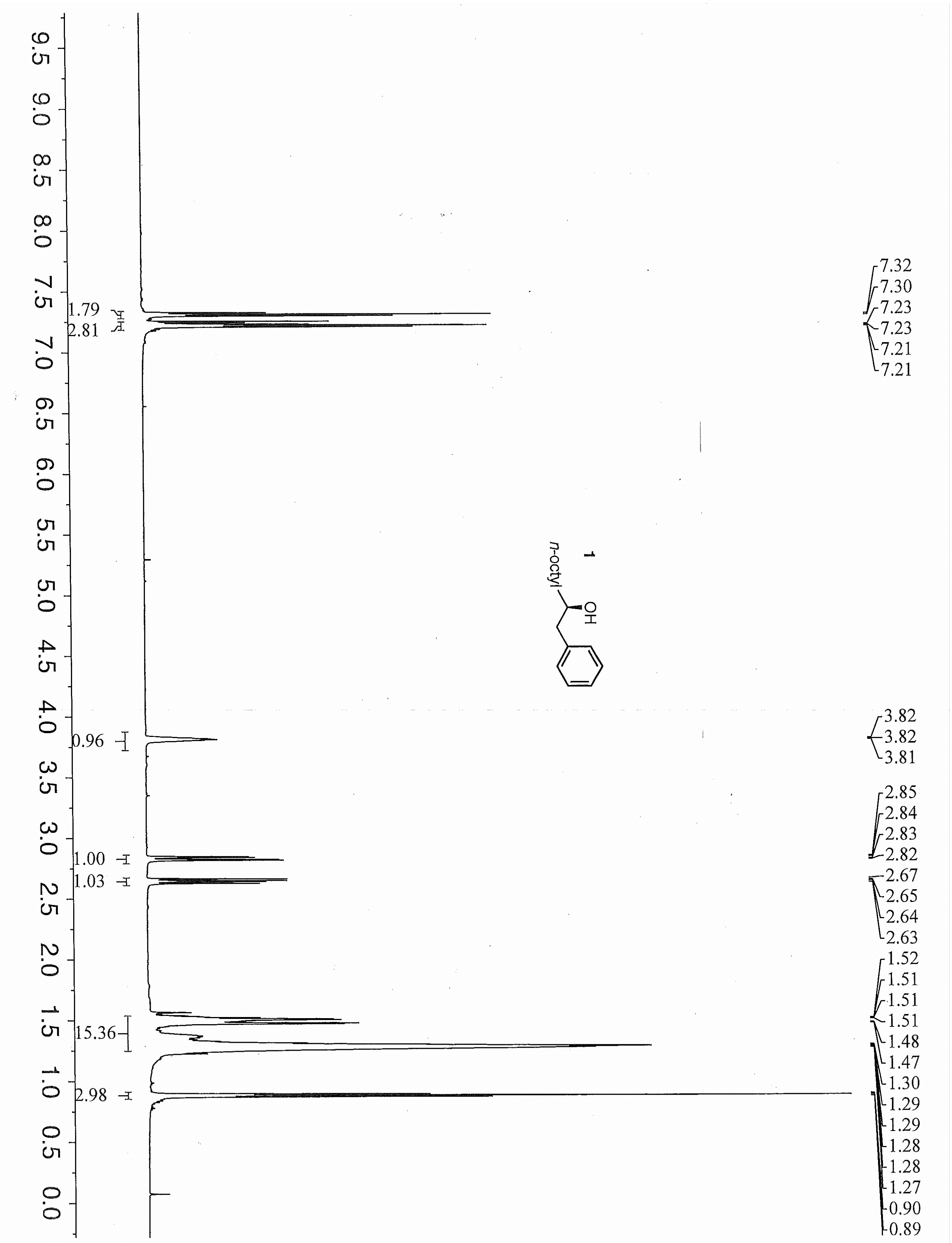
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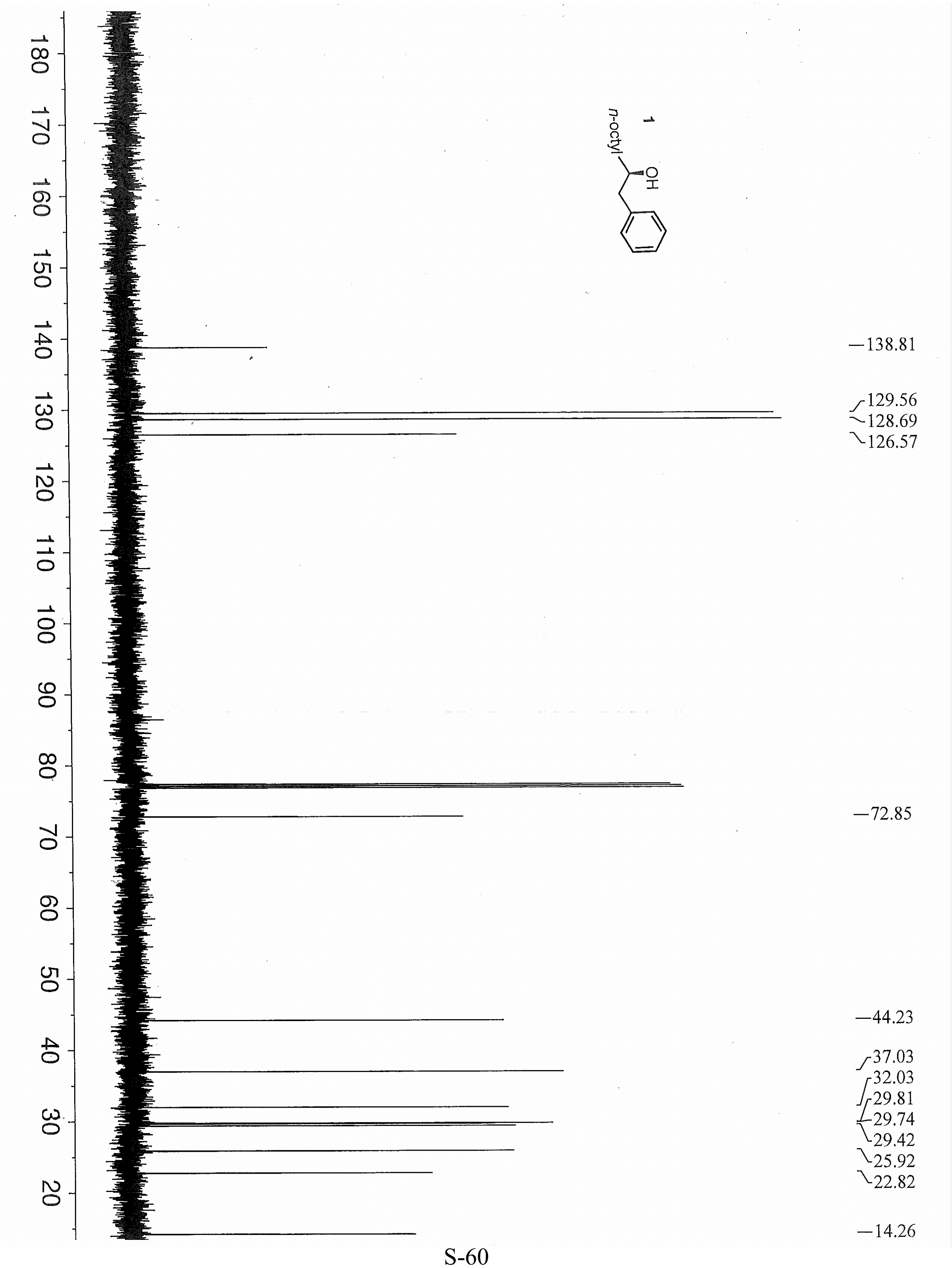
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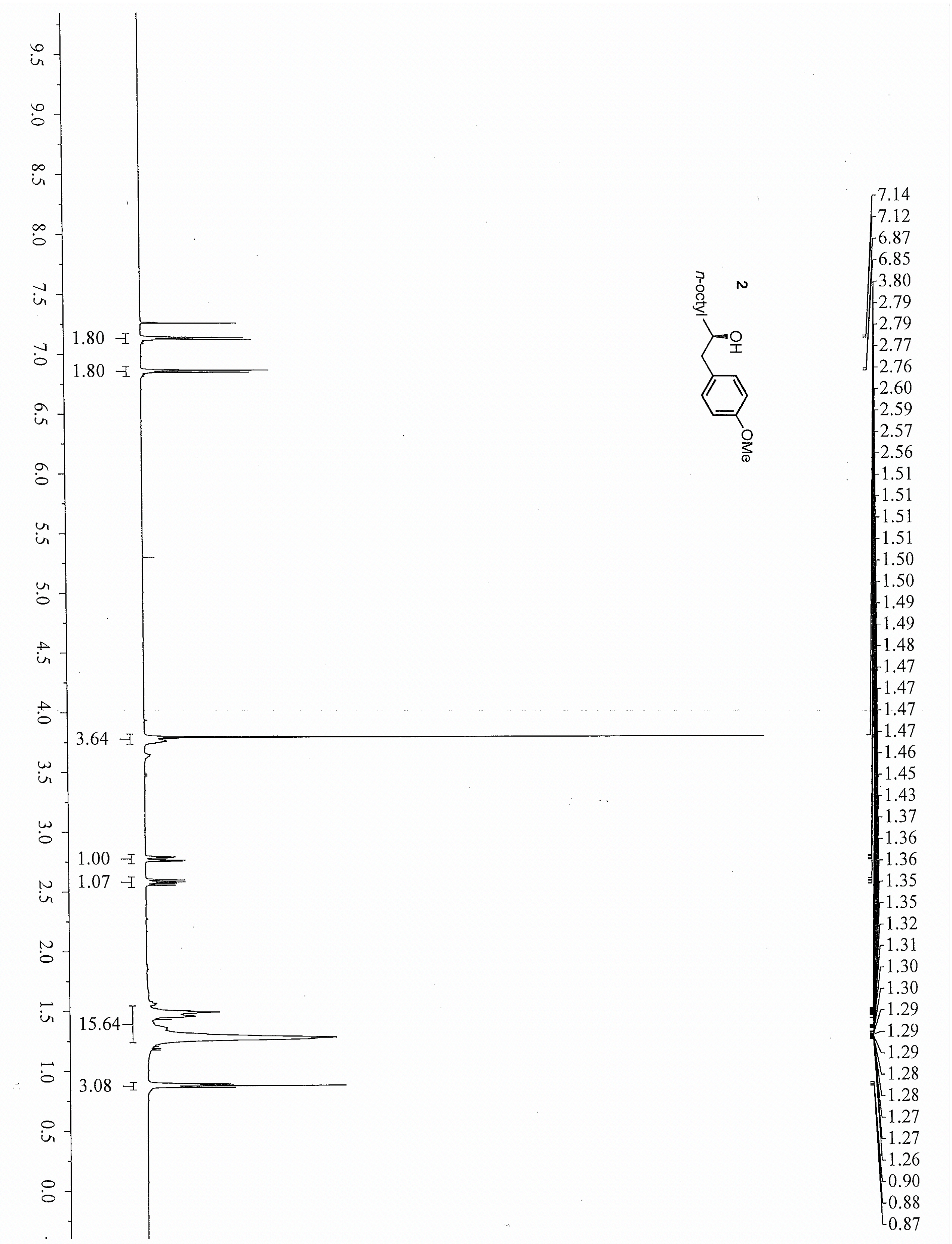
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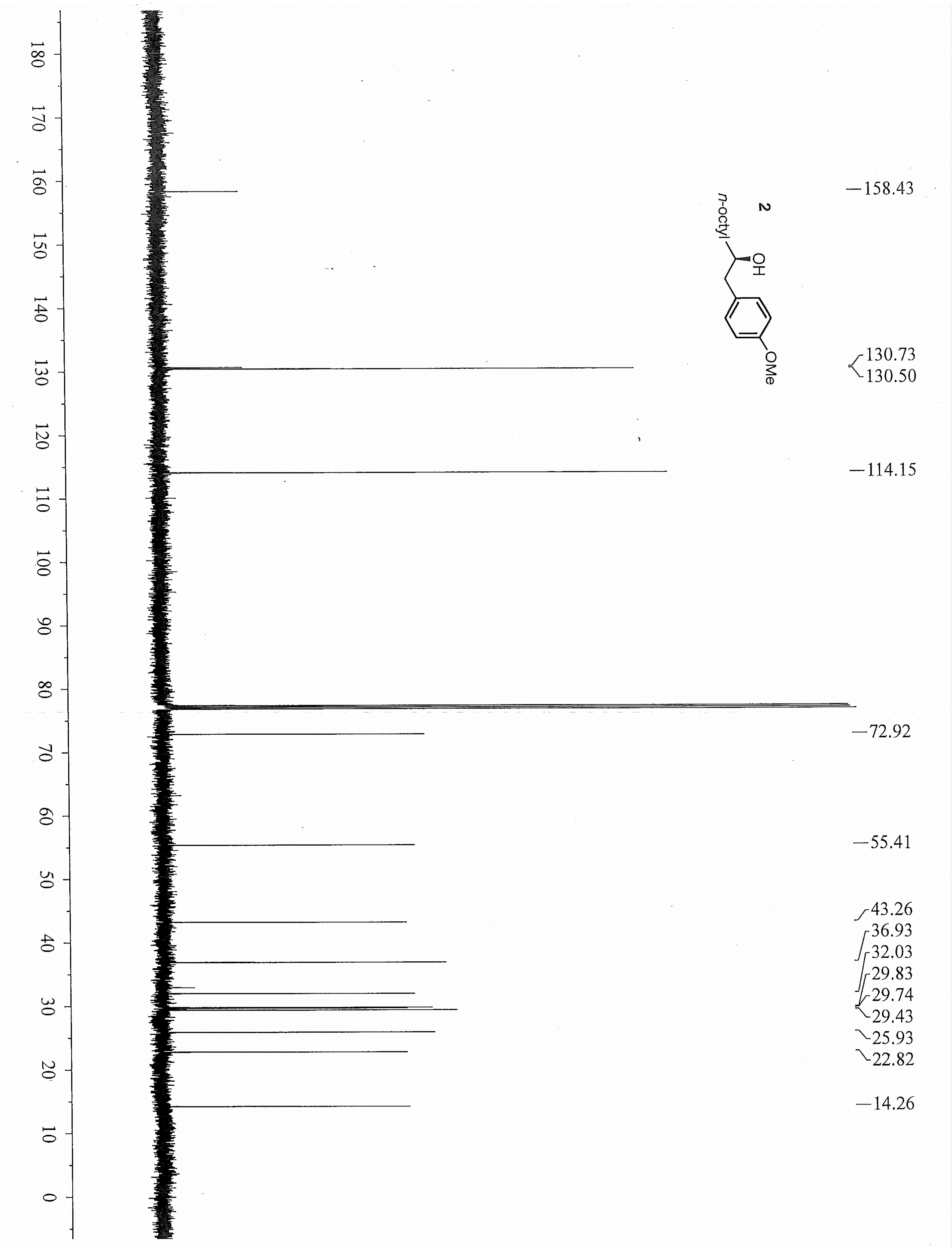
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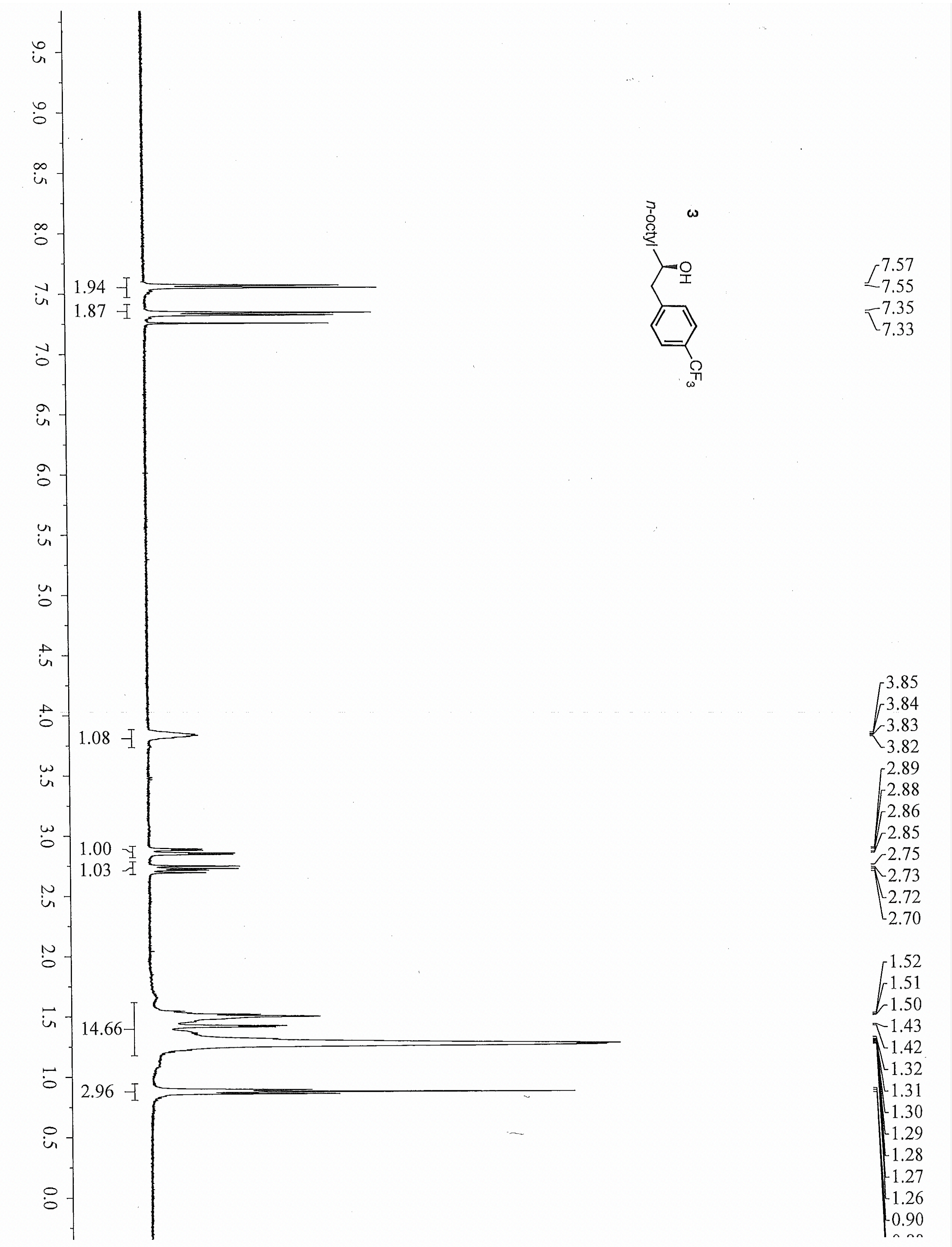
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1.51
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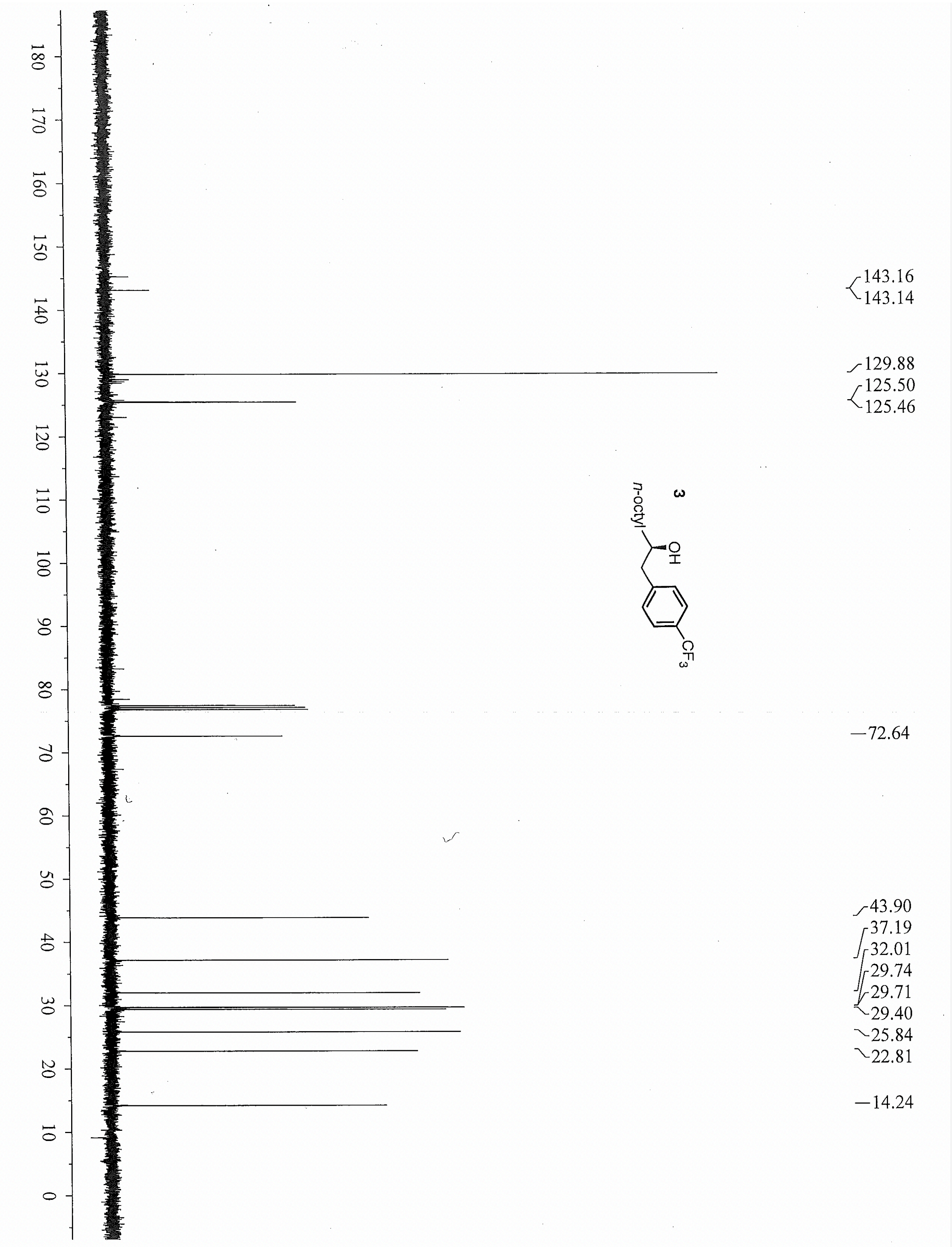
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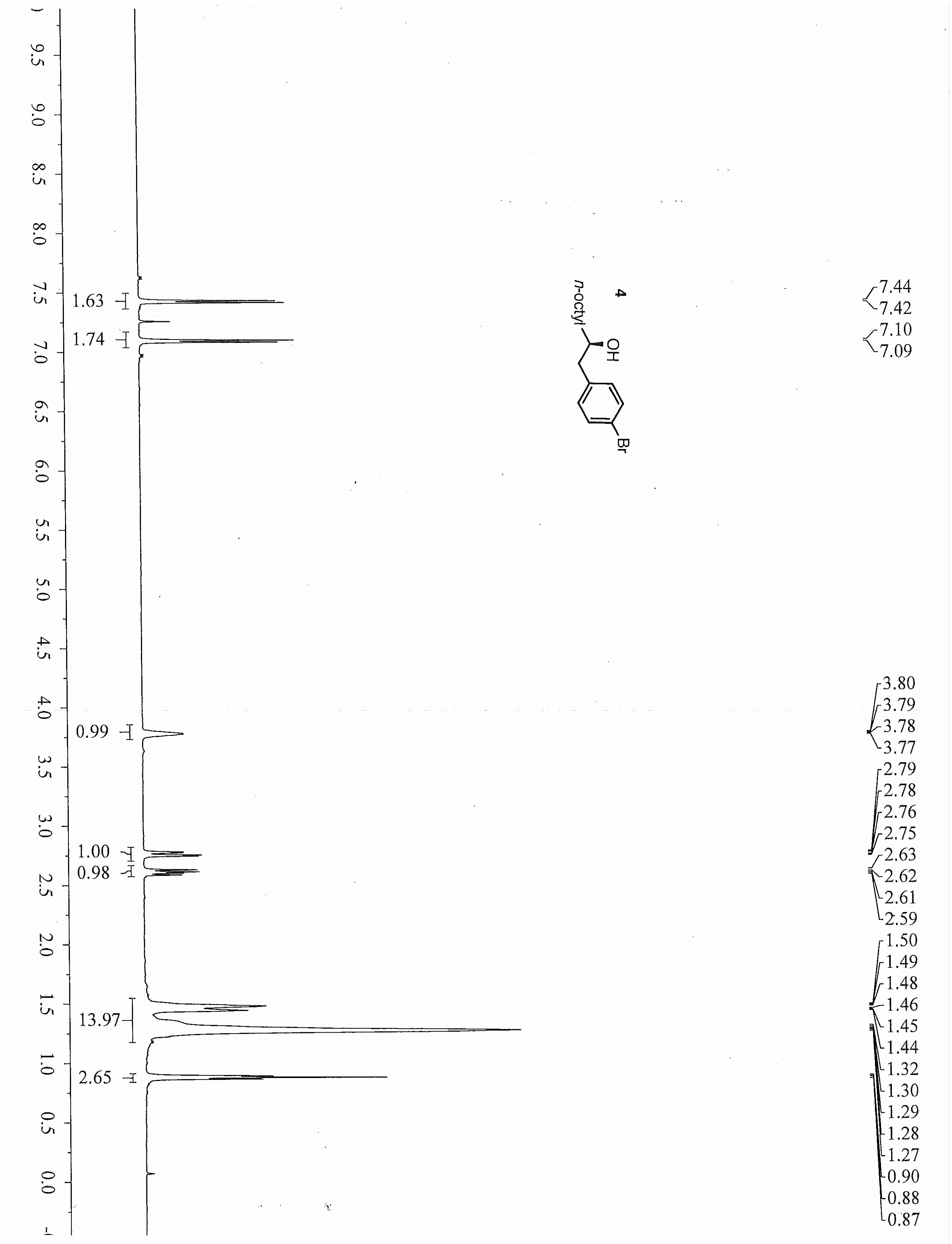
CF_3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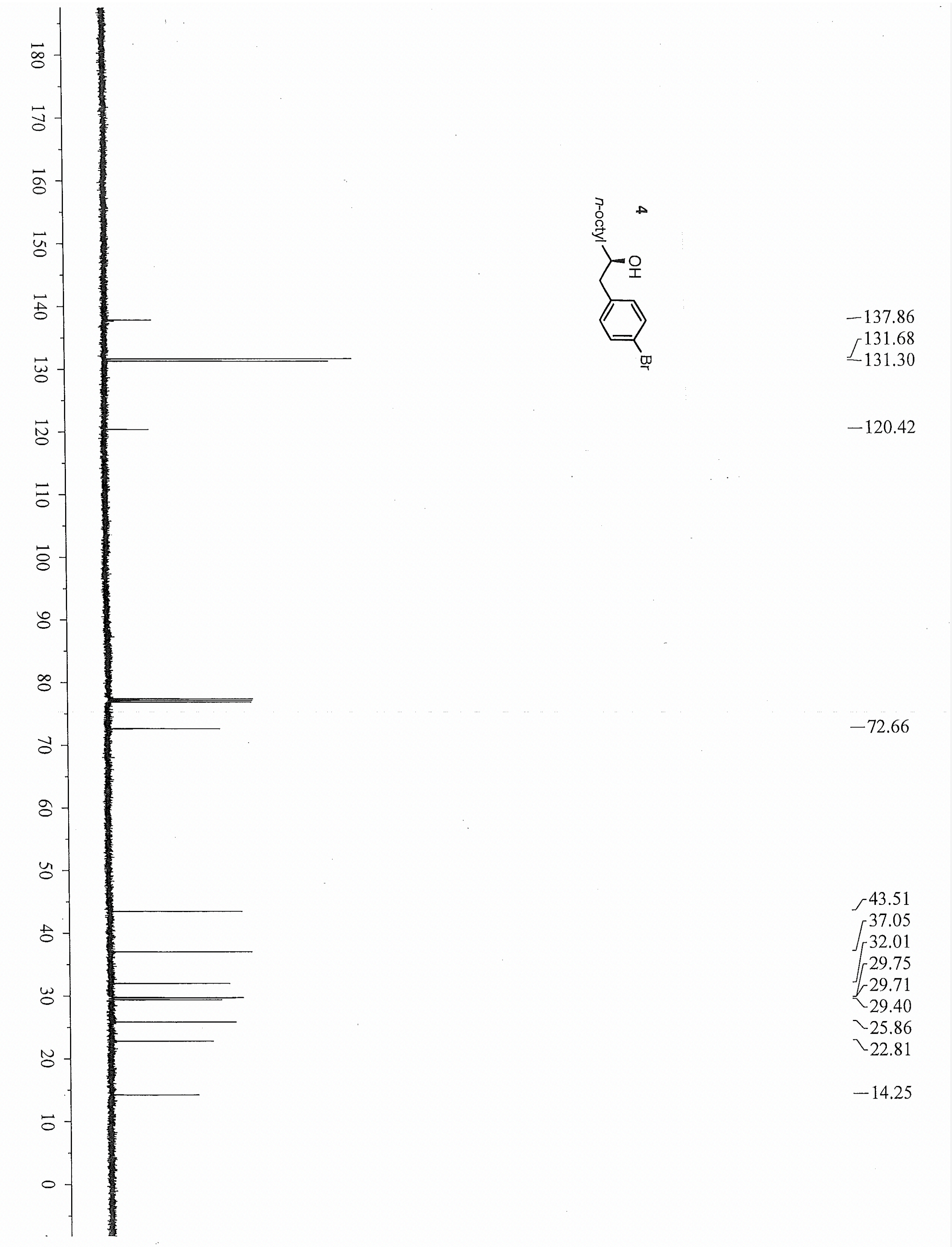
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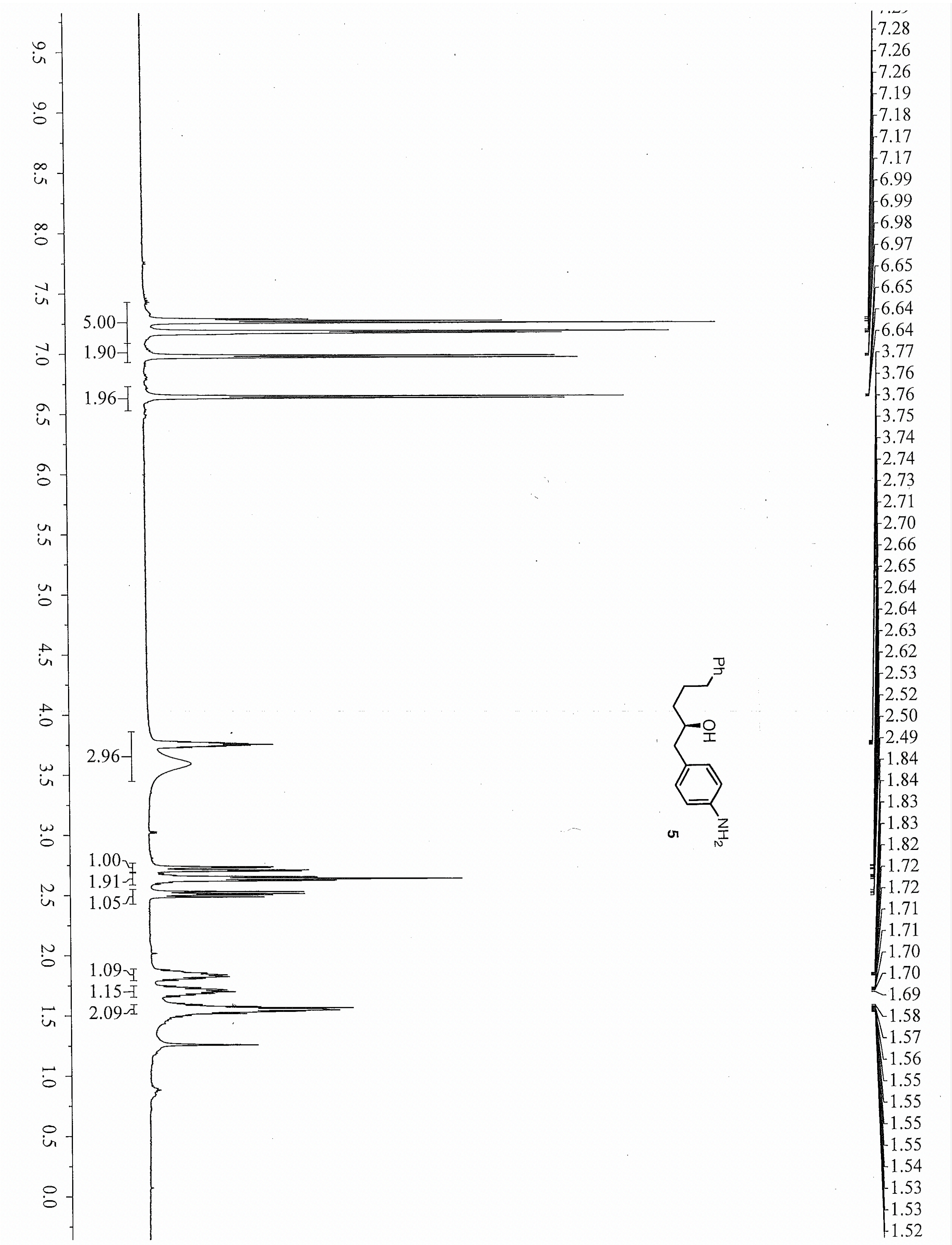
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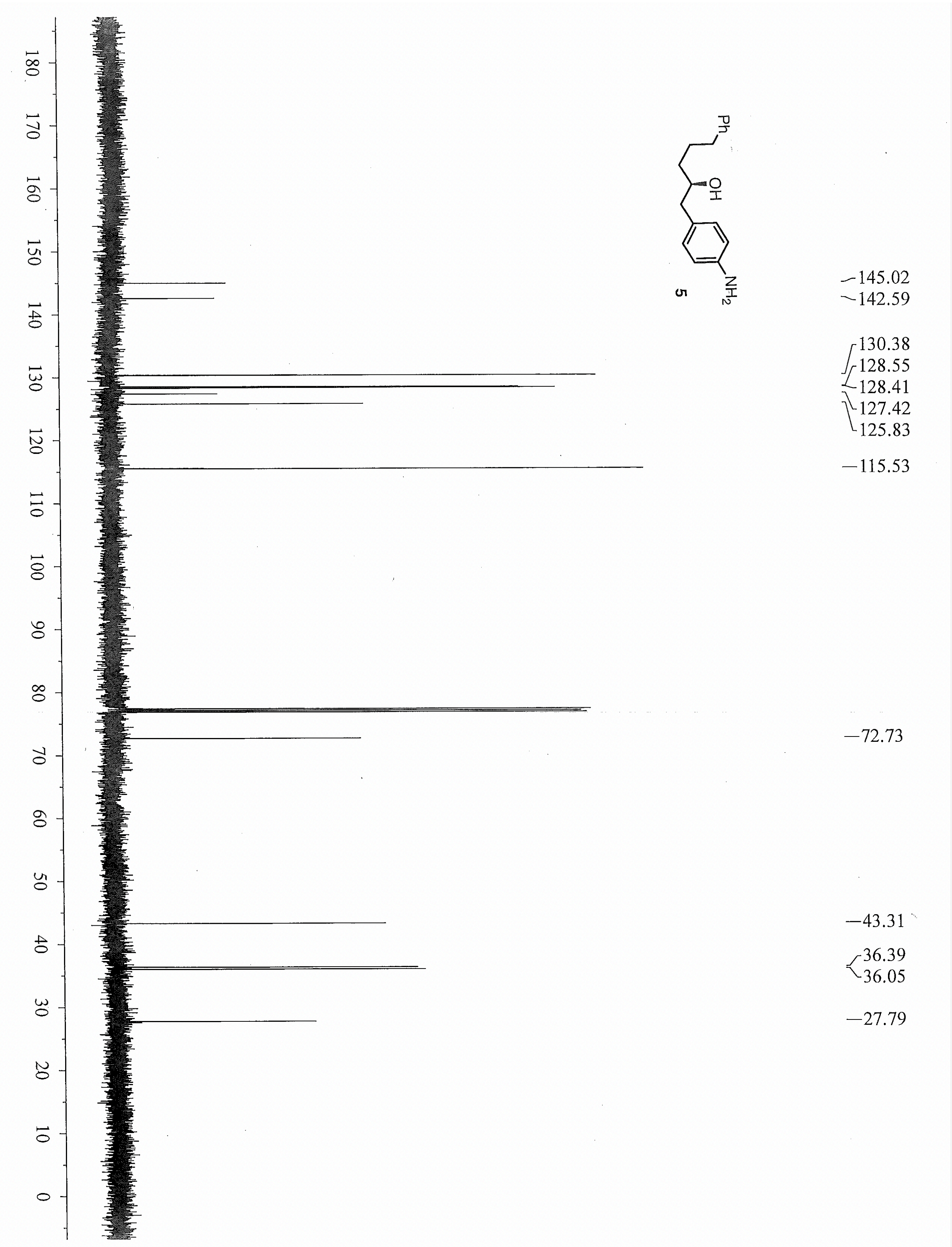
NH_2

5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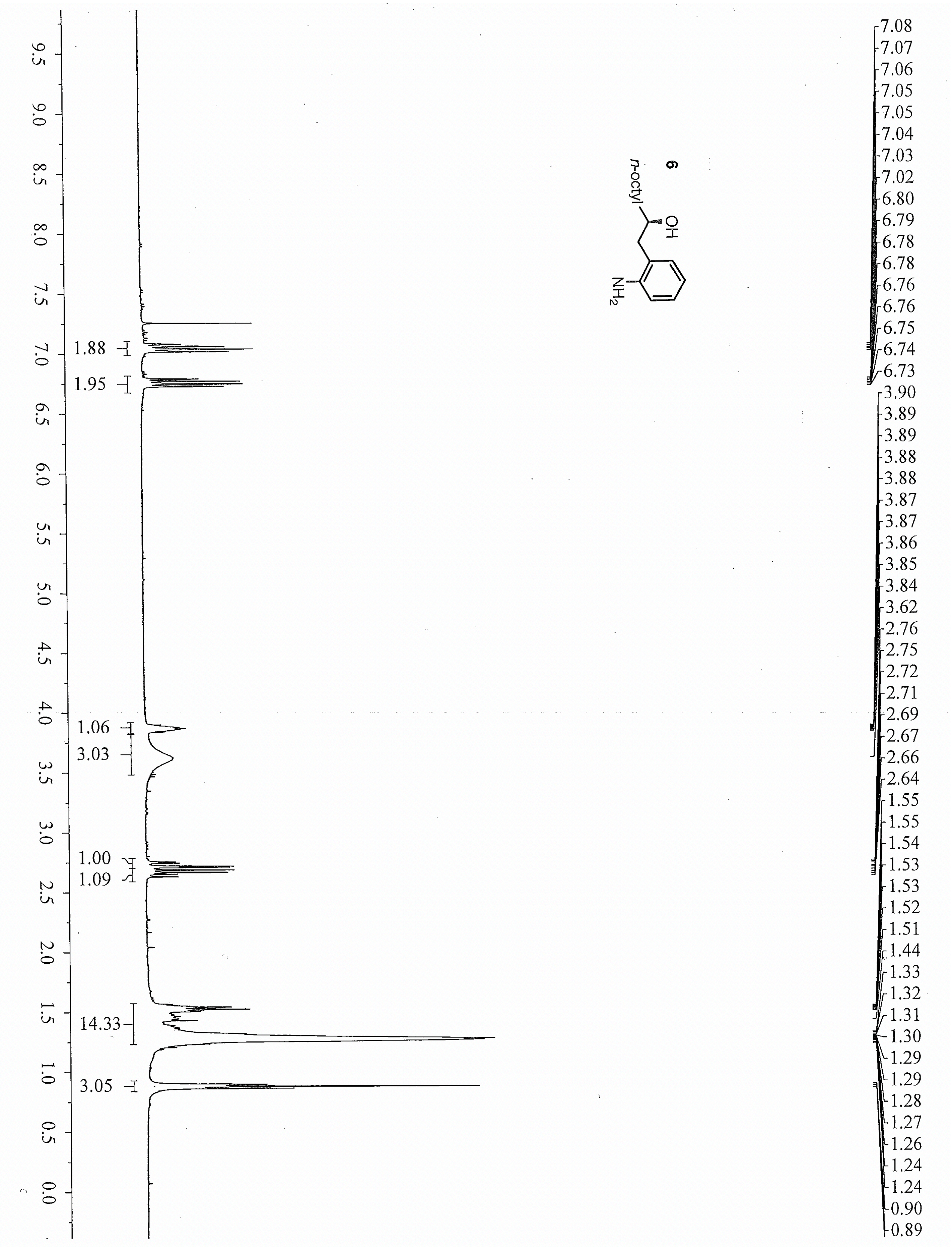
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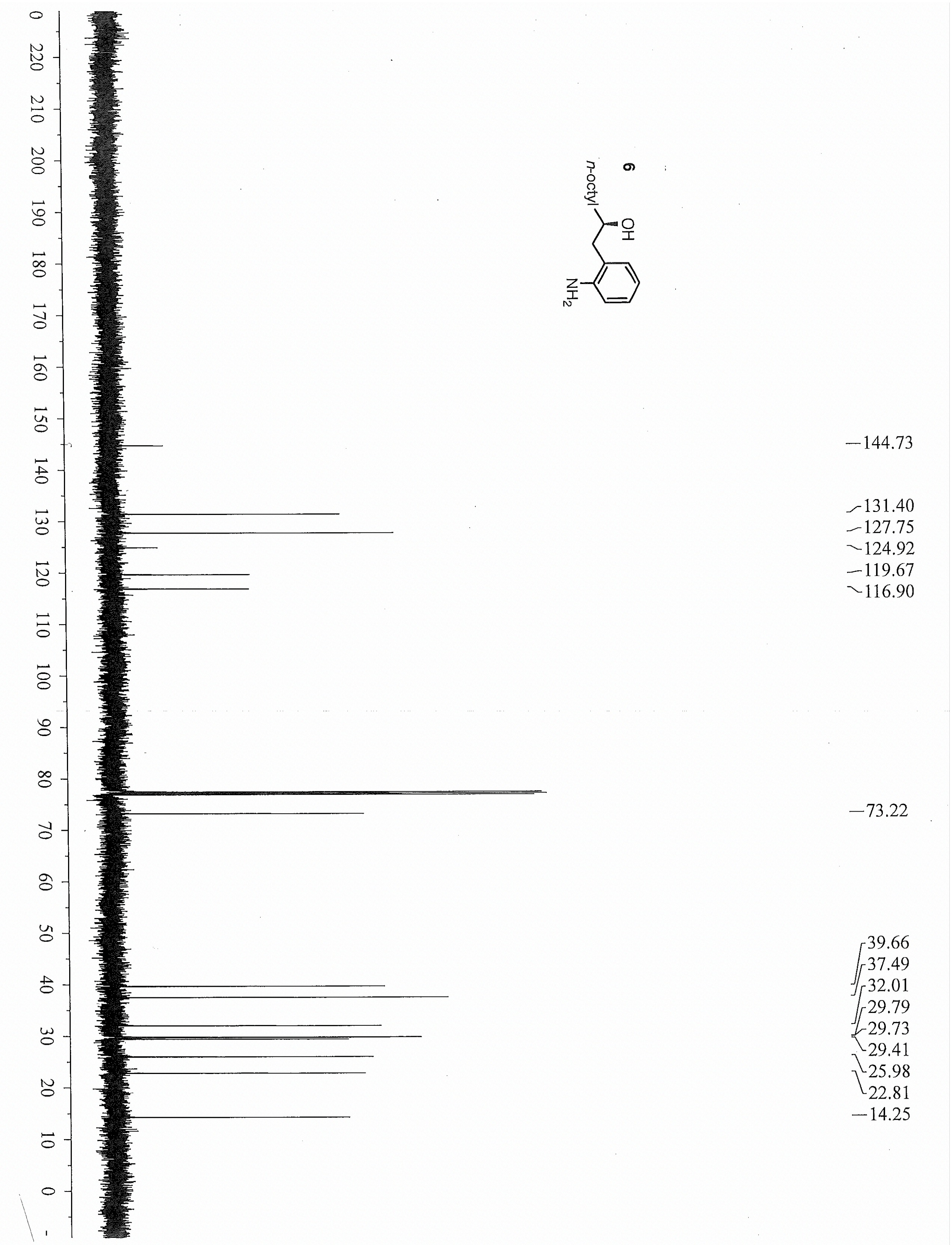
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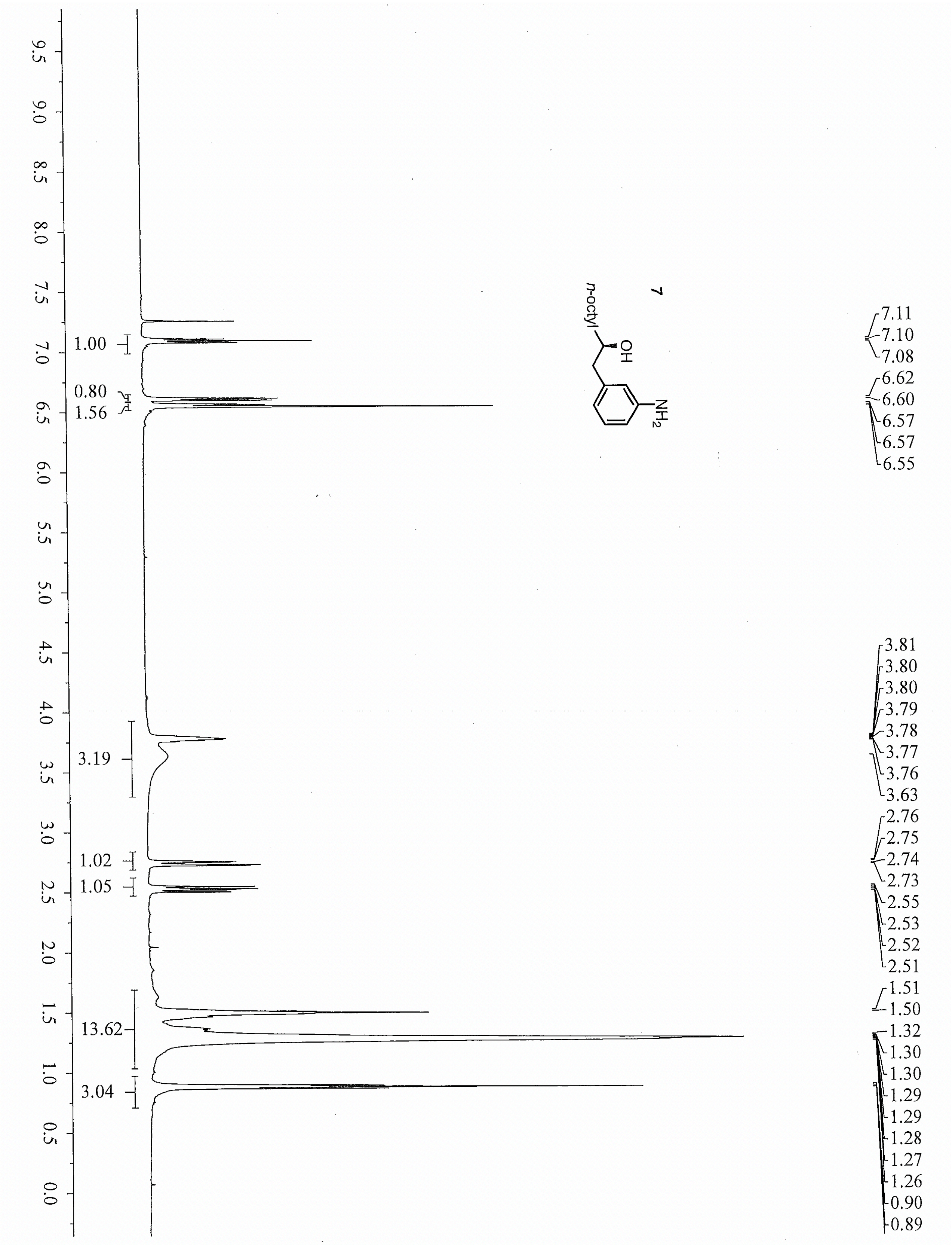
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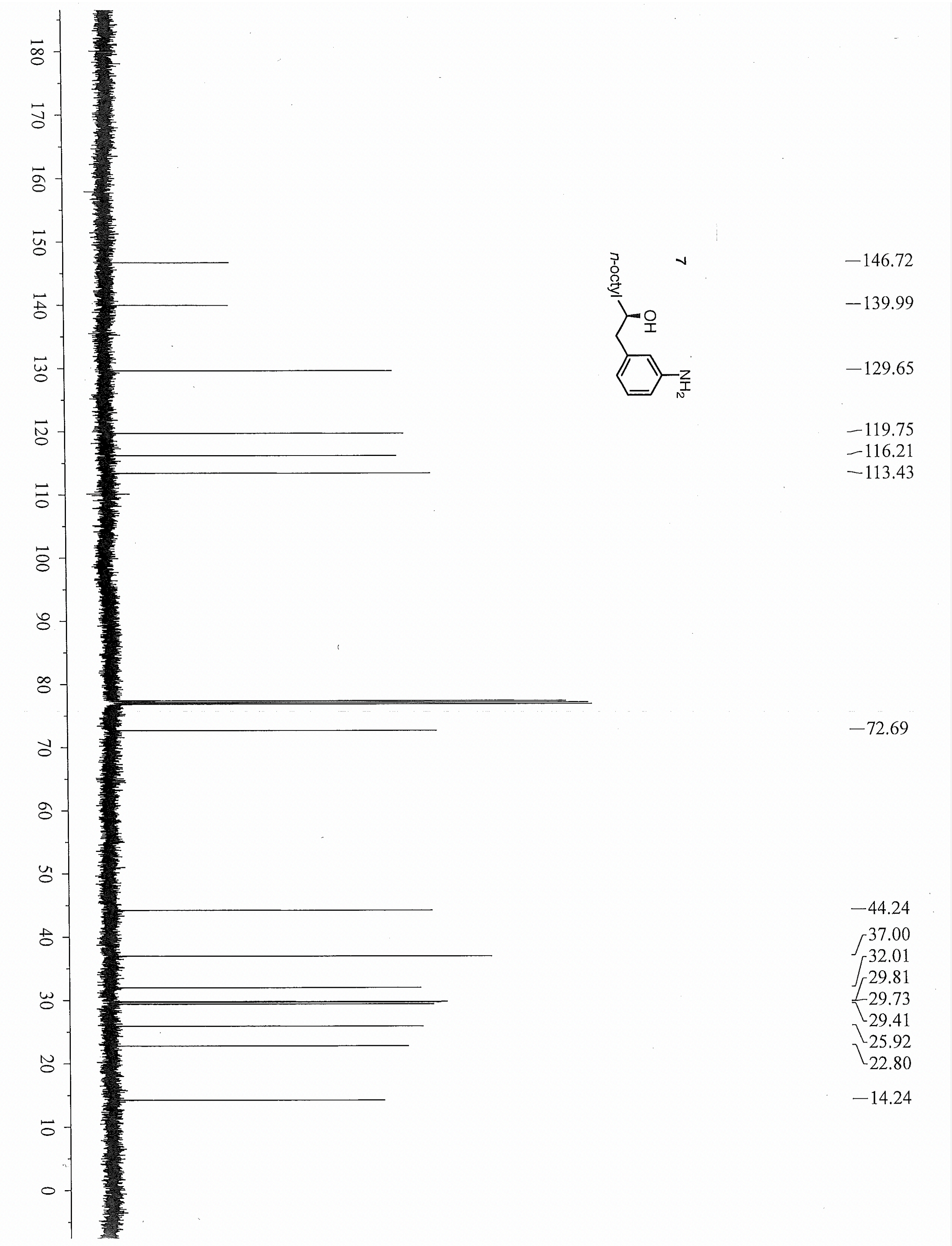
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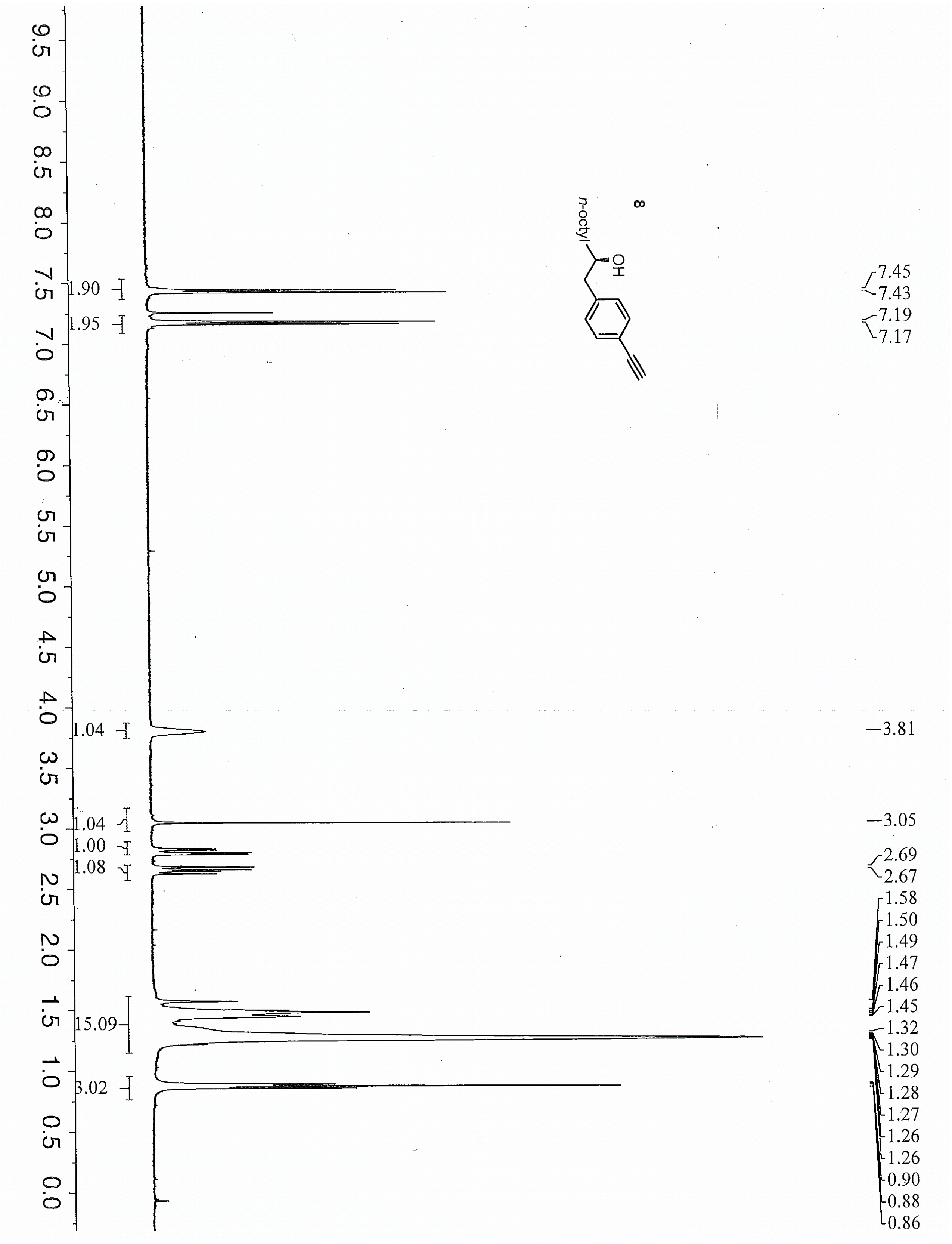
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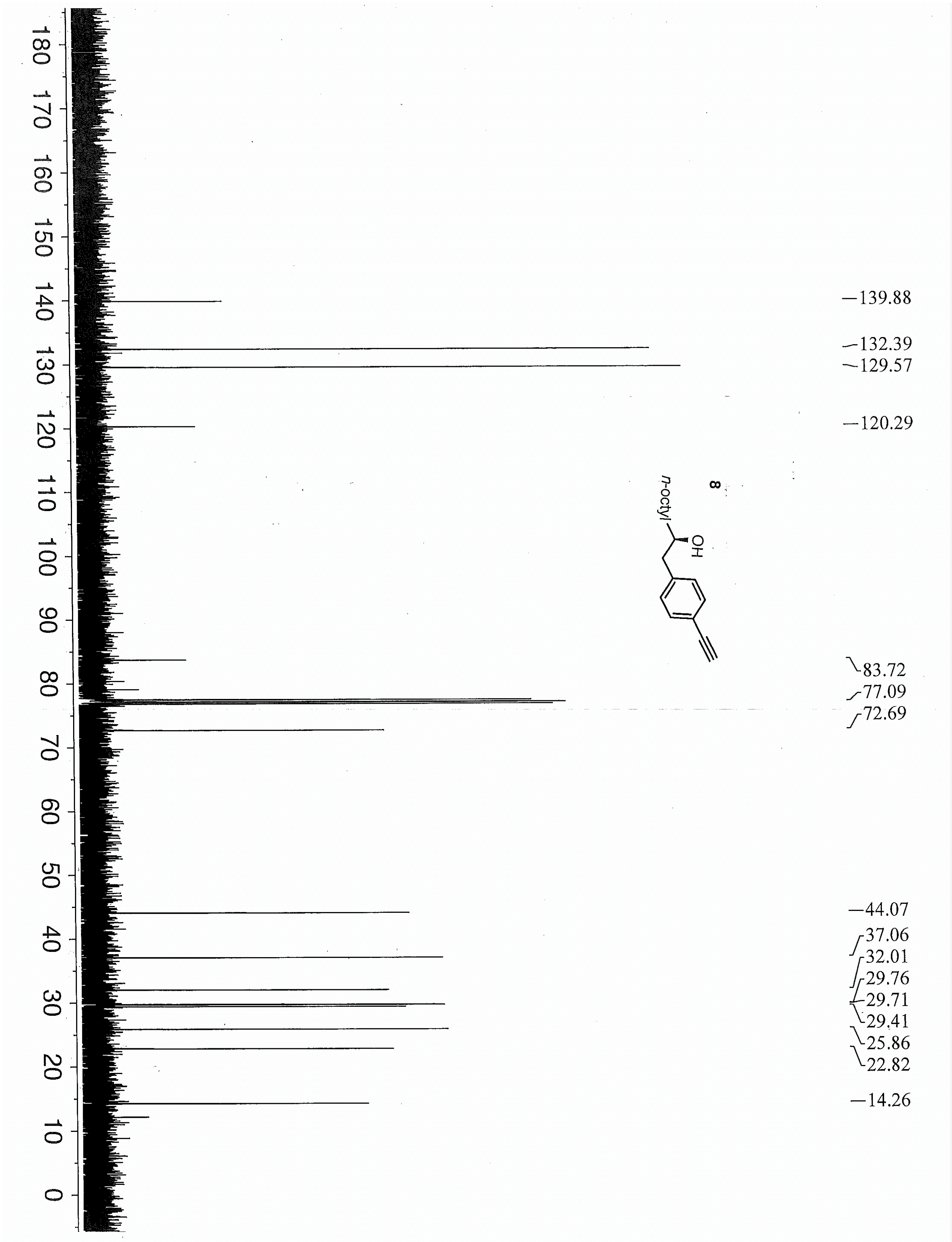
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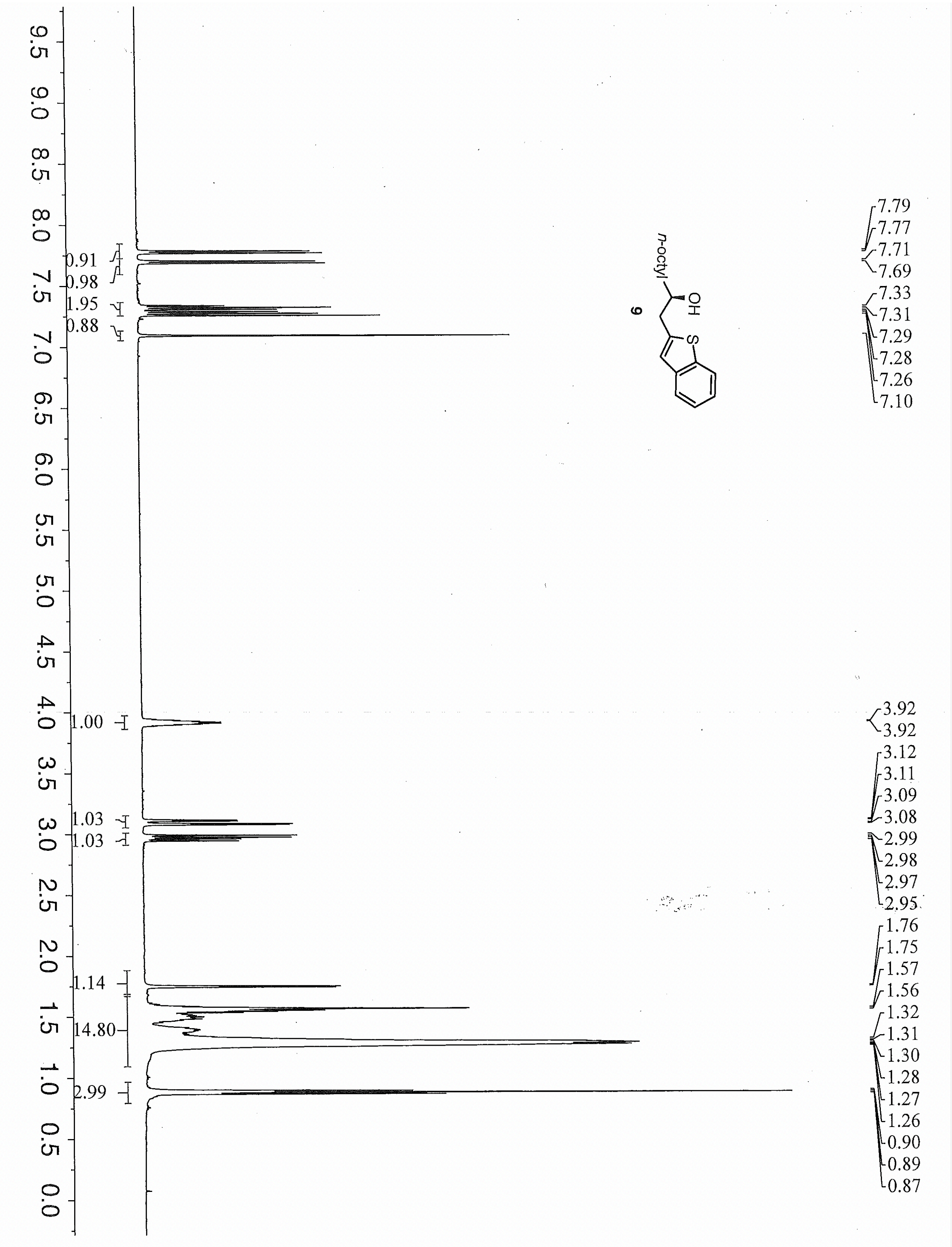
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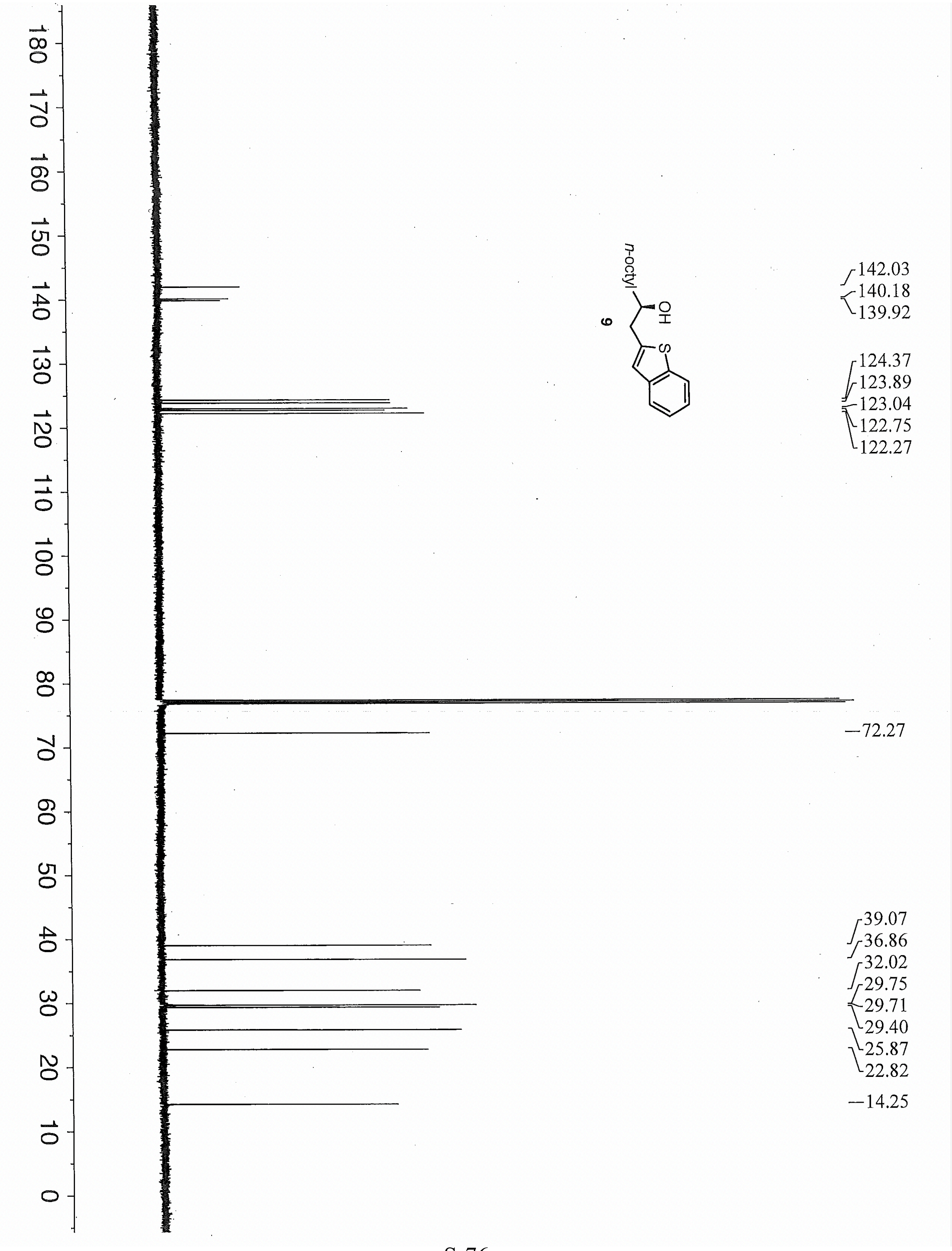

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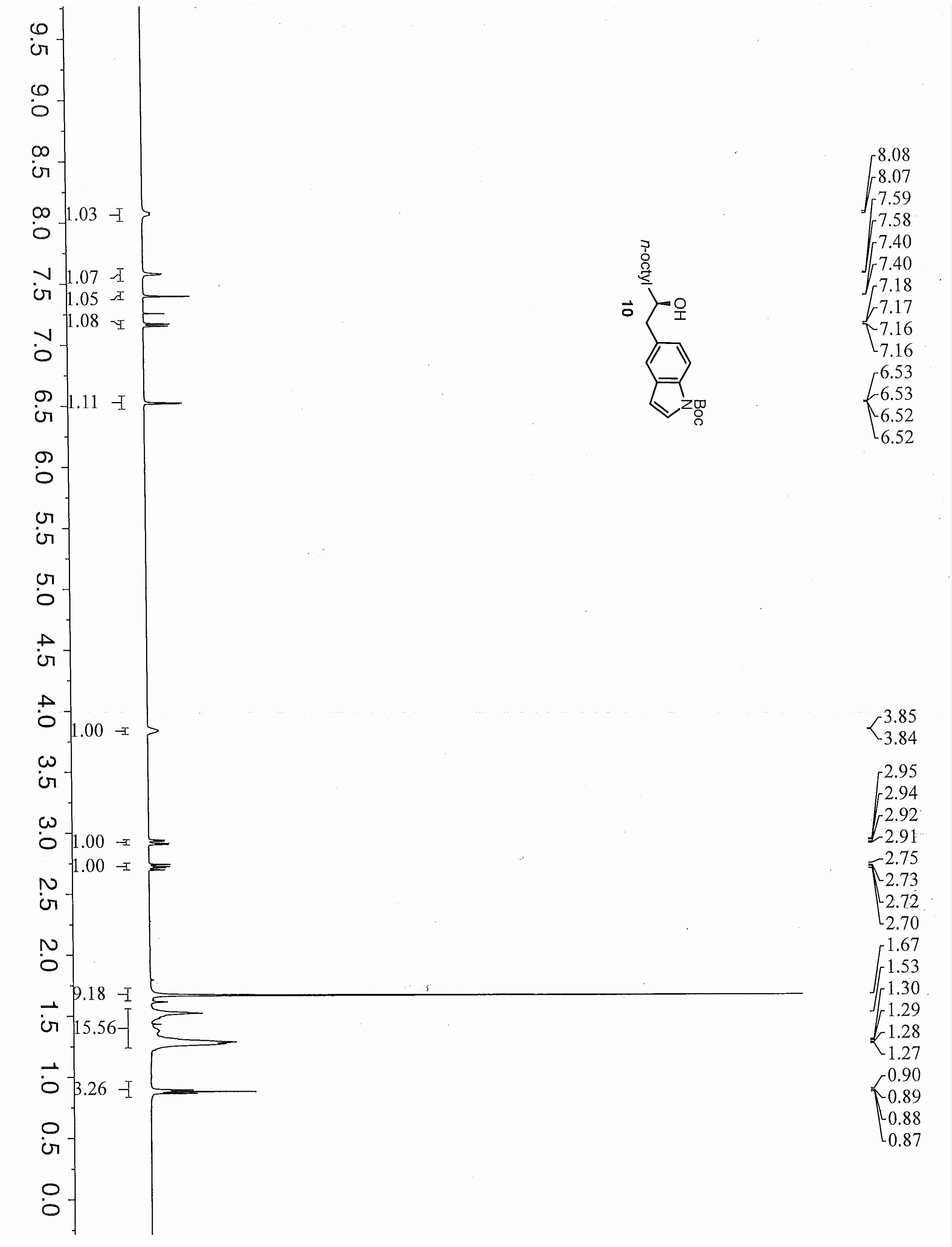
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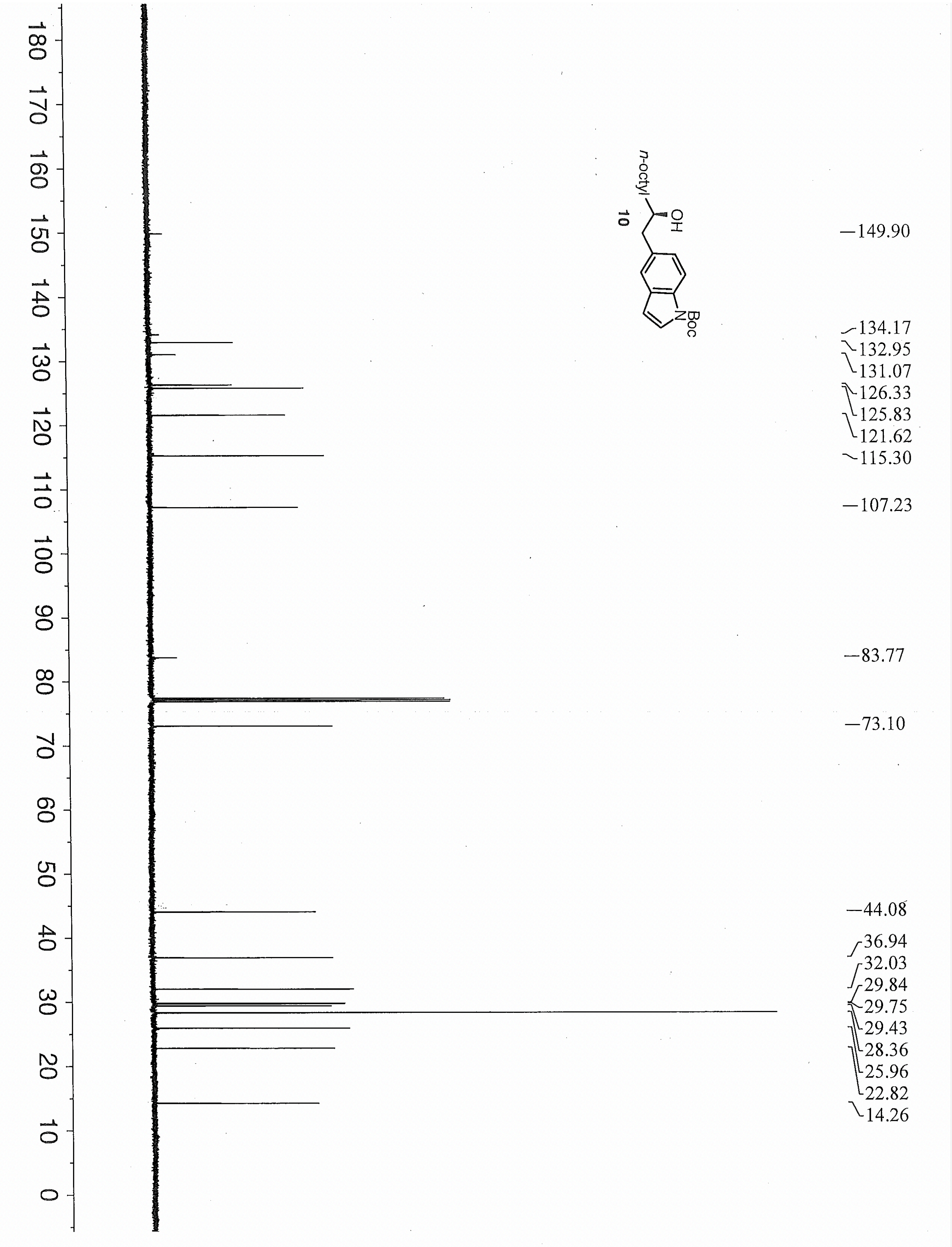
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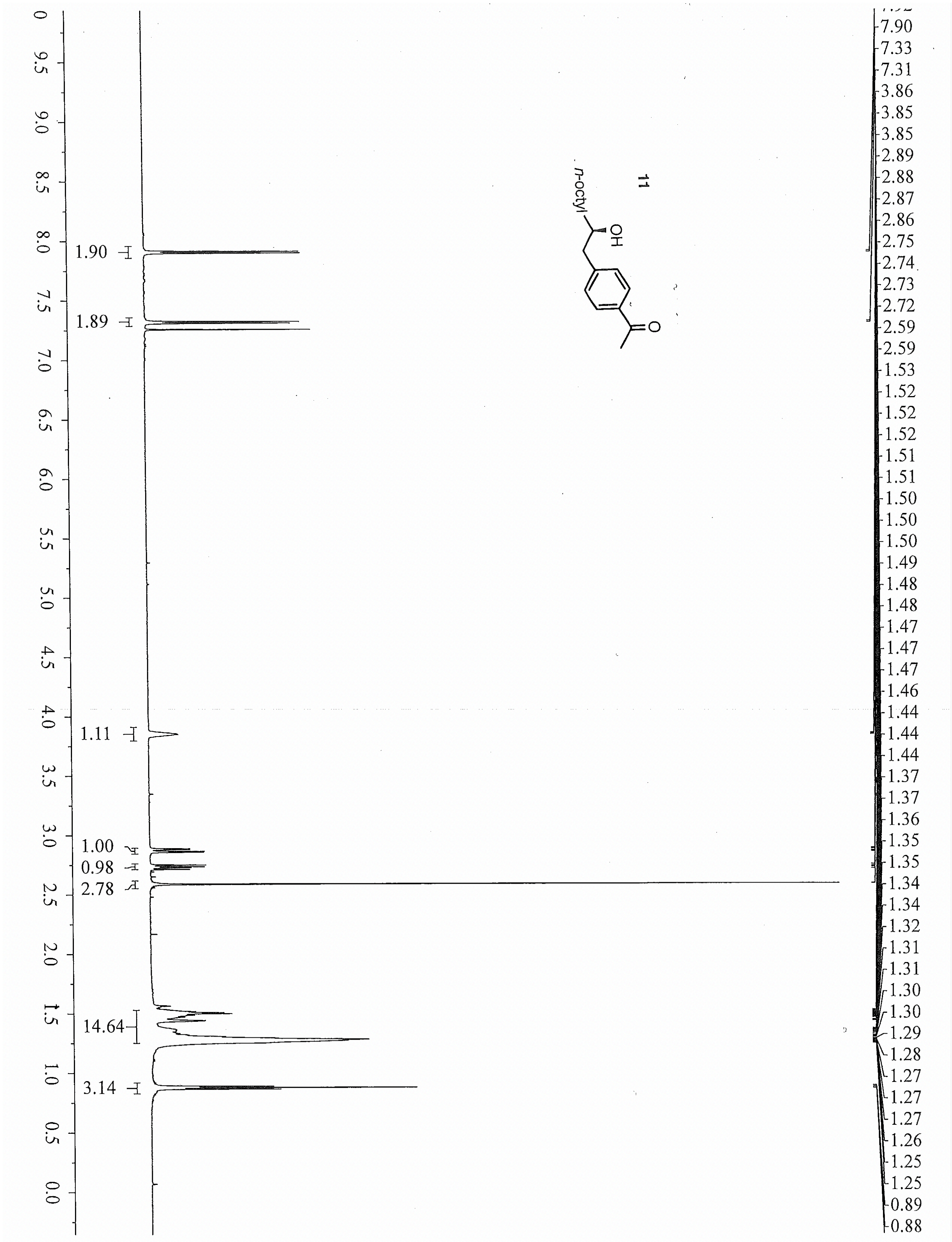
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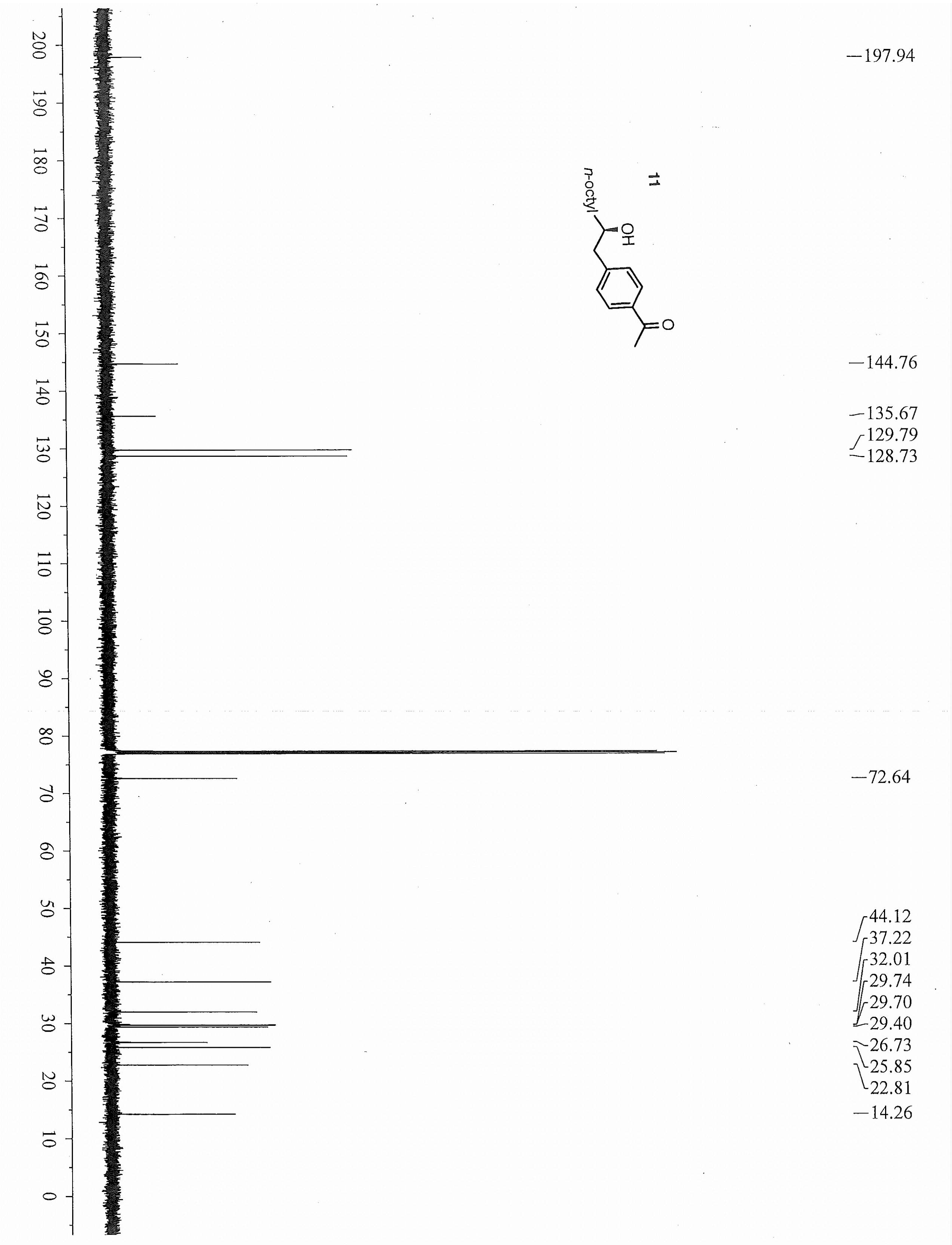
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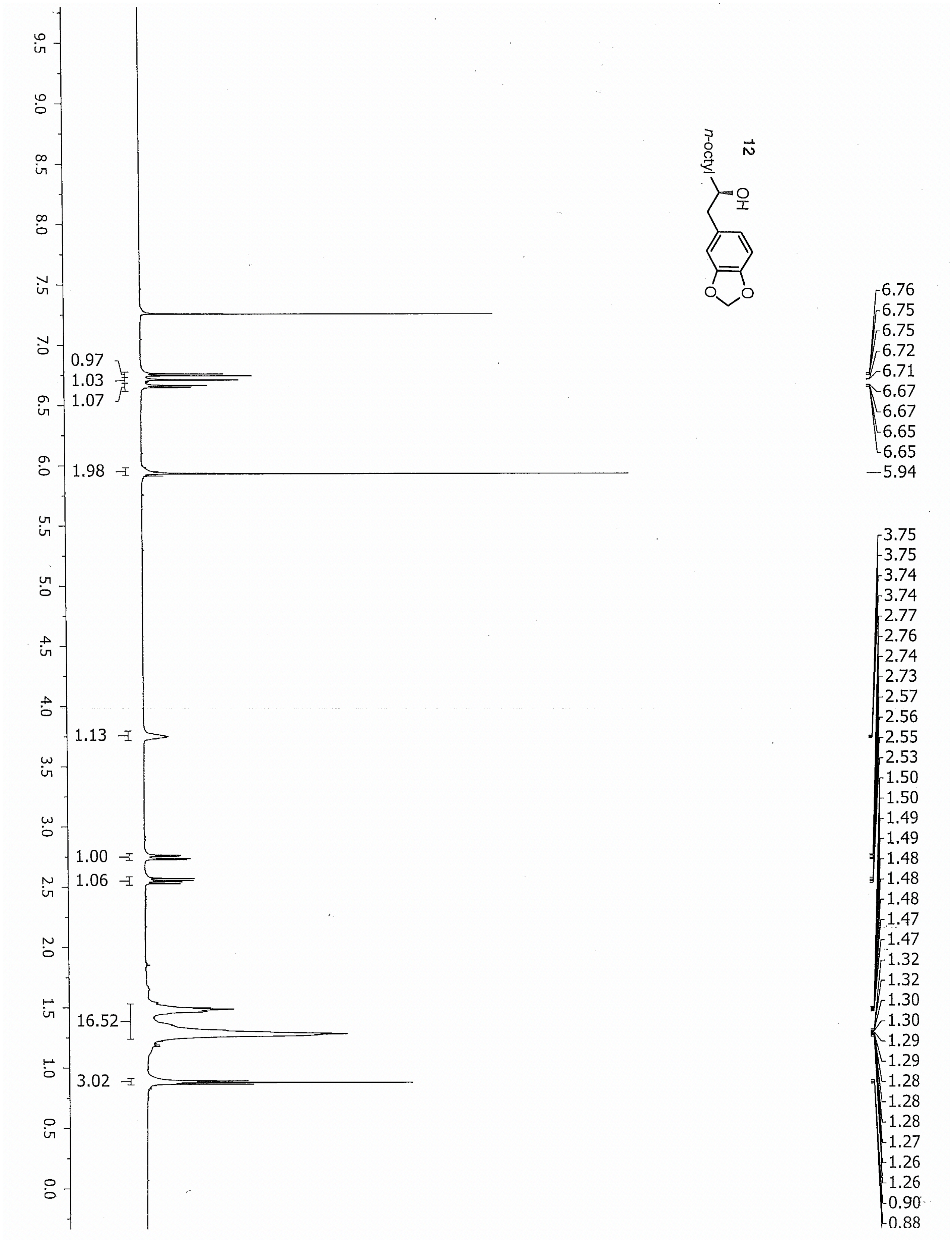
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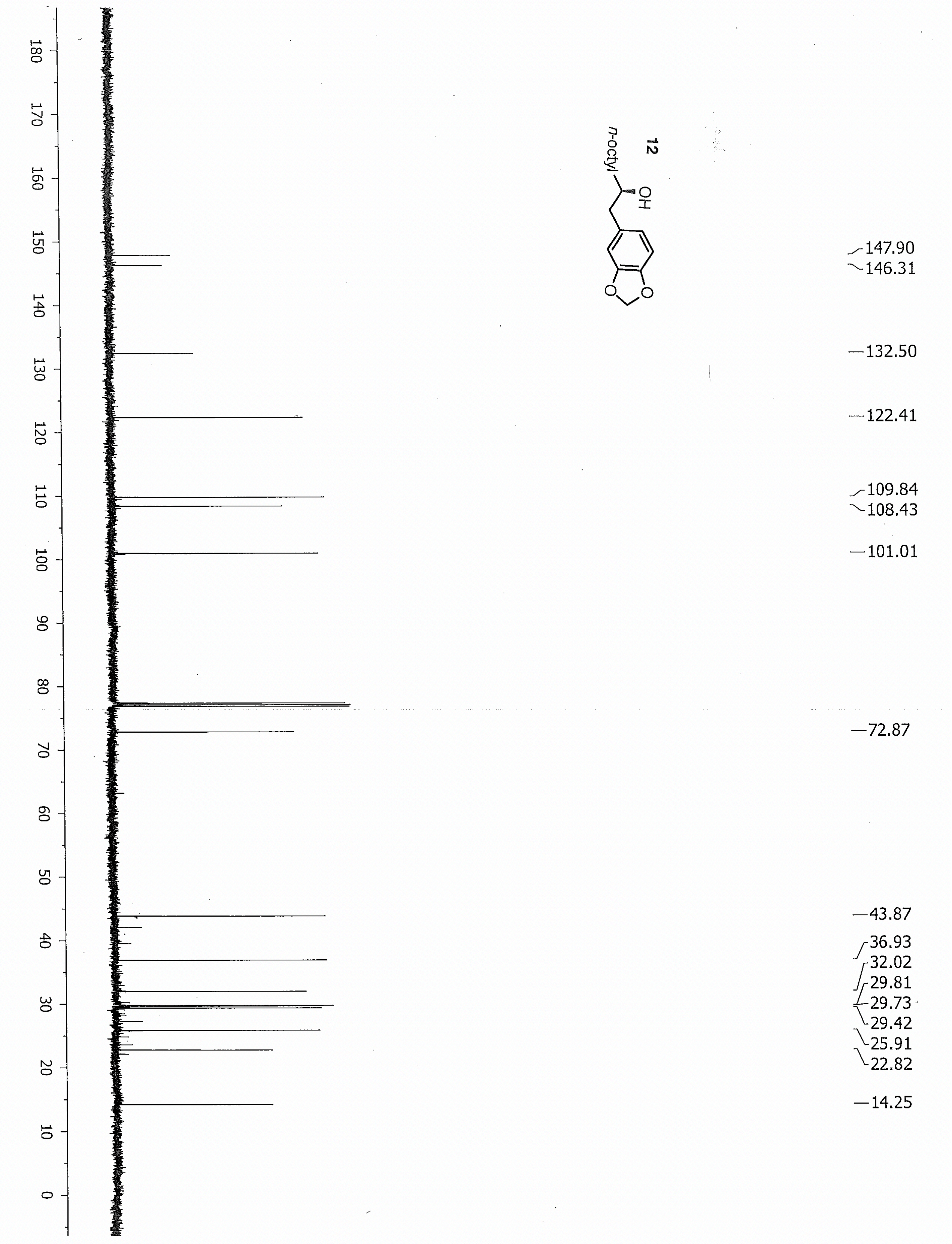
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22.82
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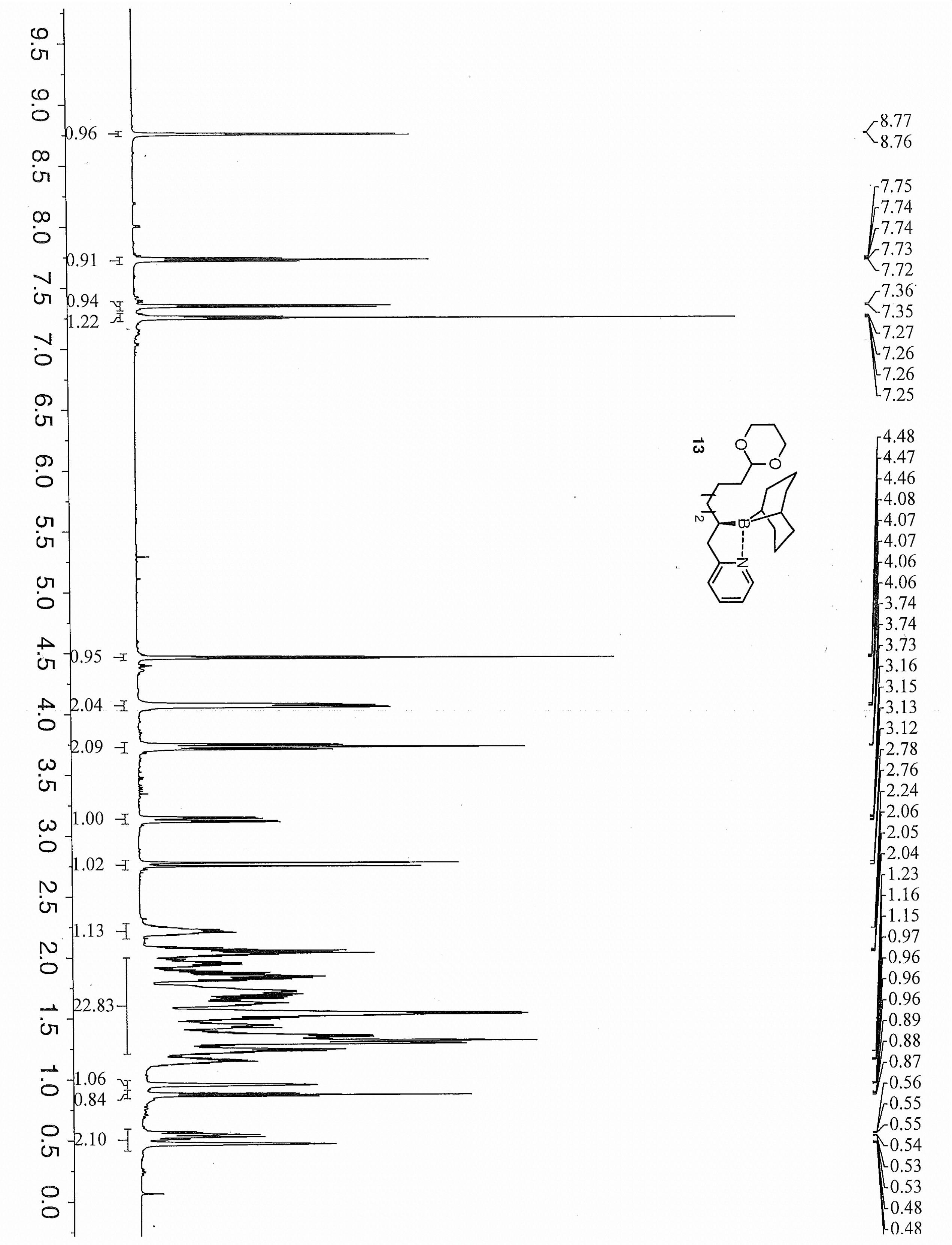
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1.13
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7.26
7.25
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4.06
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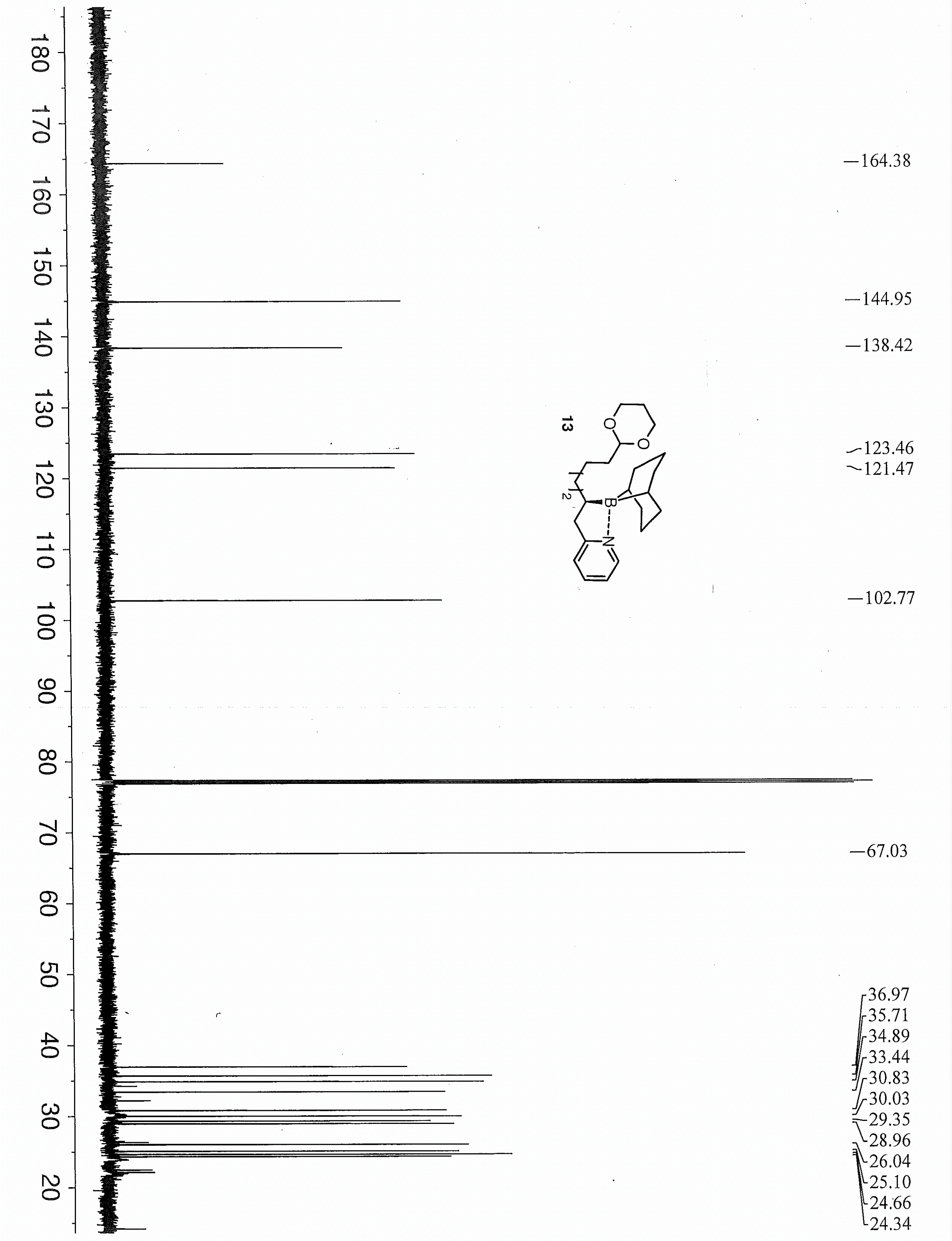
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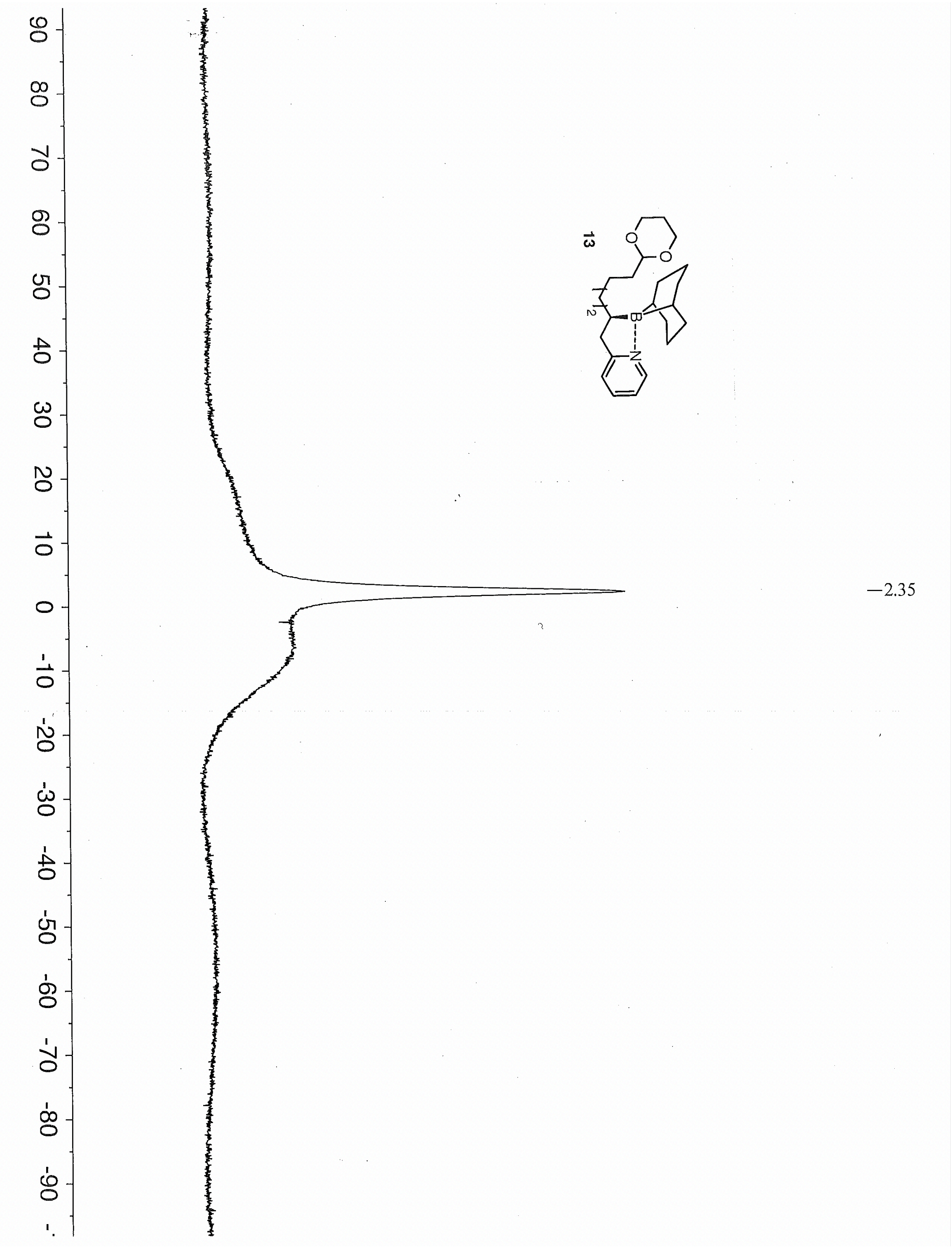
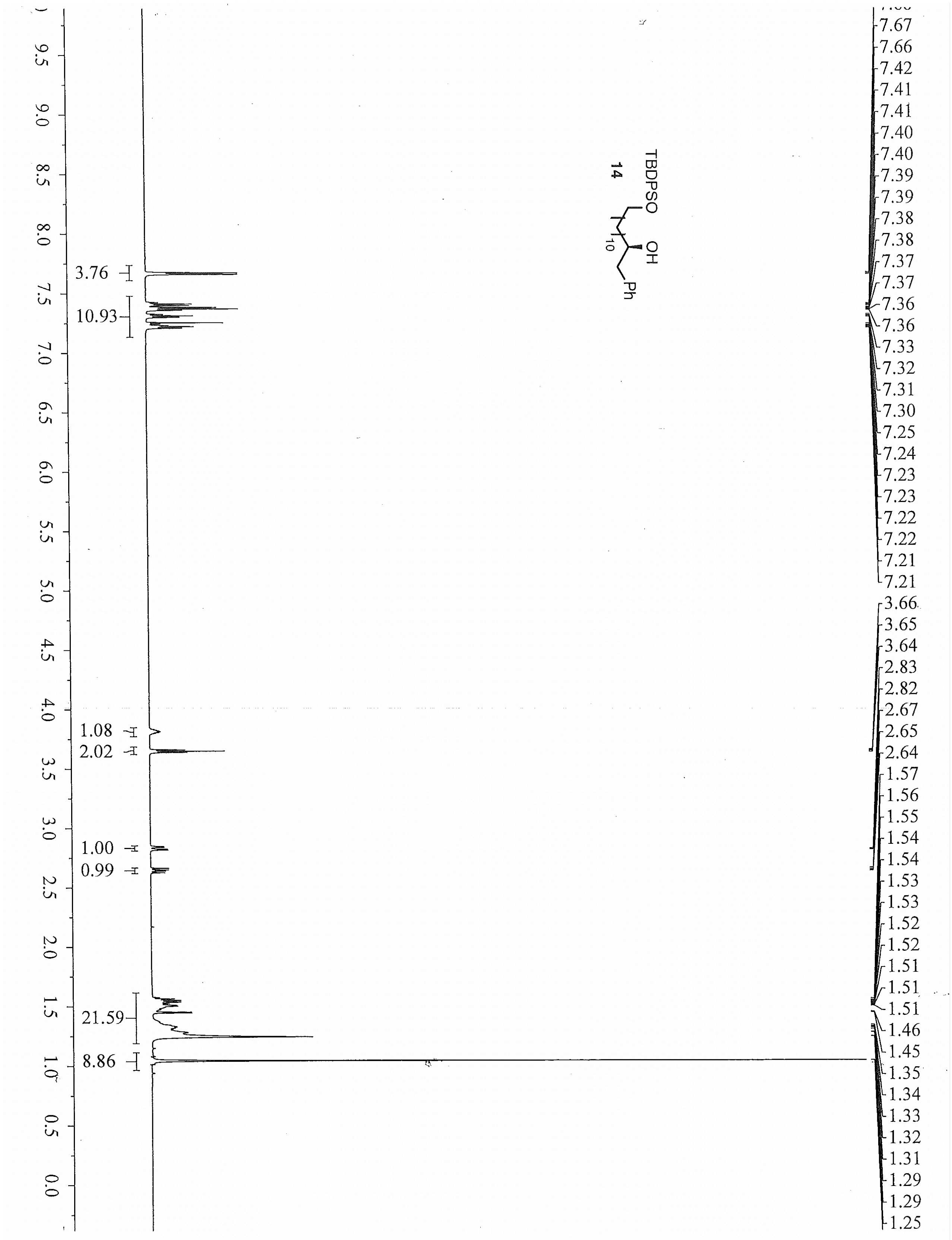
N
2

^{11}B NMR

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4.0
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2.0
1.5
1.0
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0.0
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10.93
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2.02
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1.52
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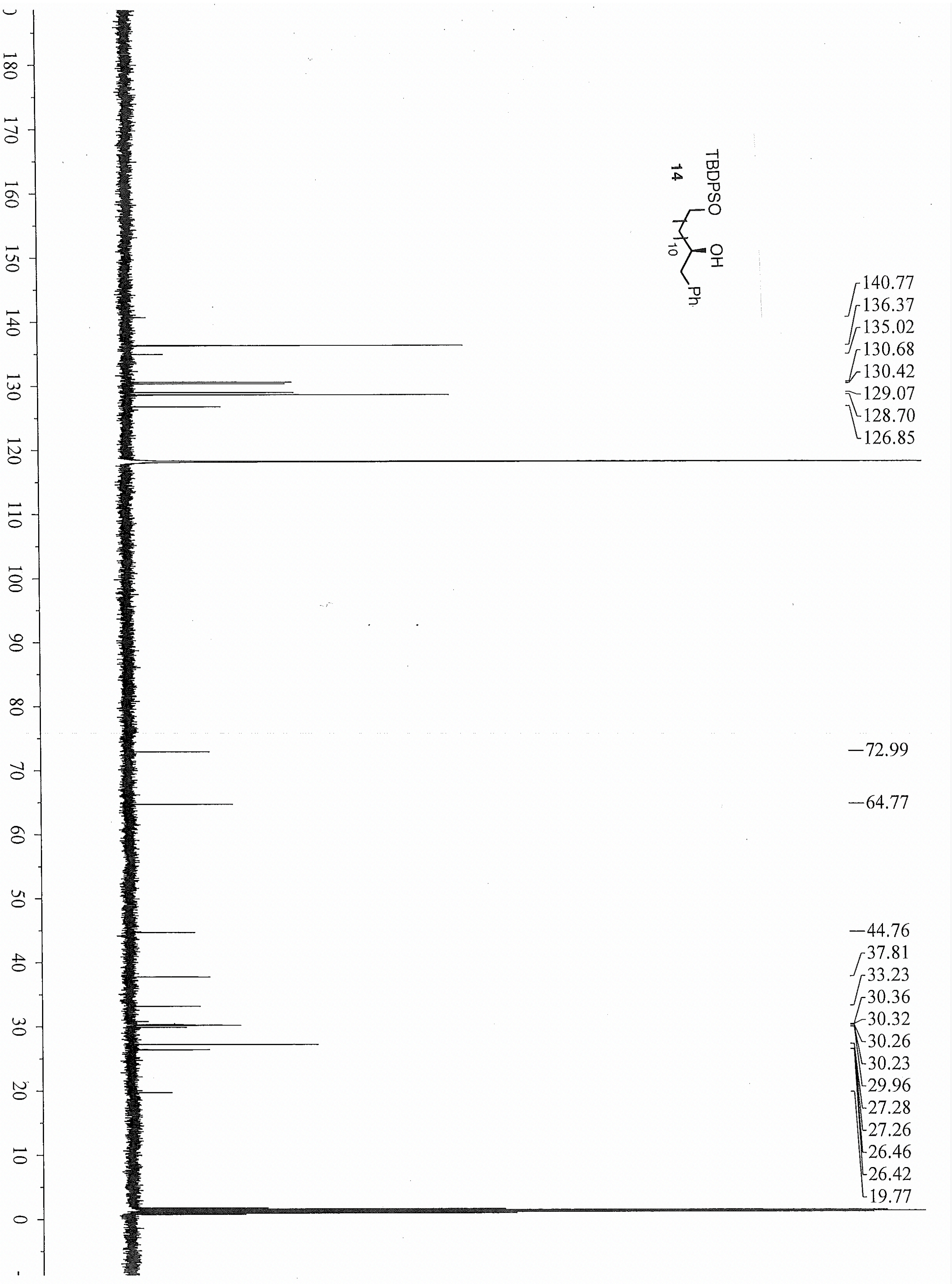
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44.76
37.81
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30.26
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27.28
27.26
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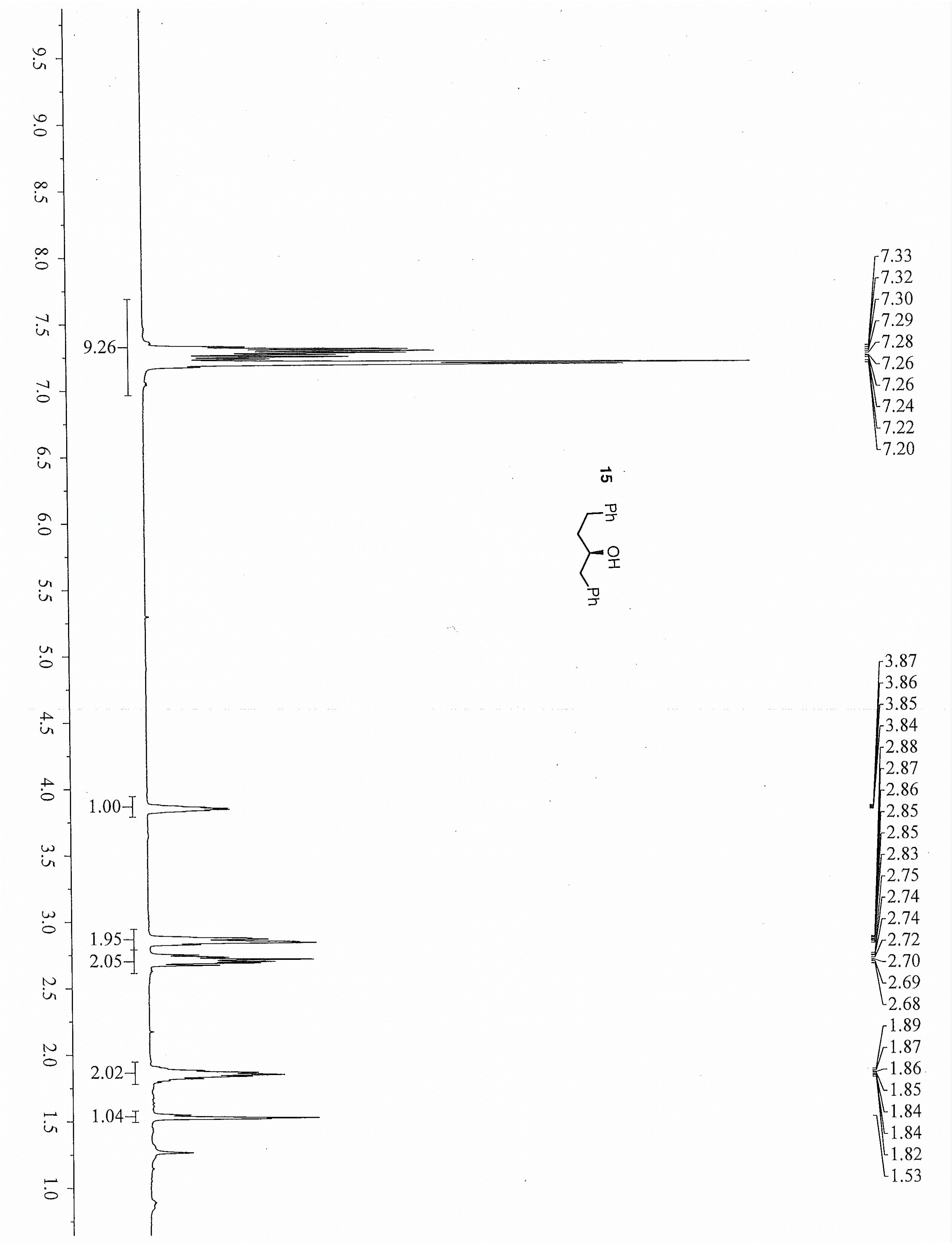
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7.20
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Ph
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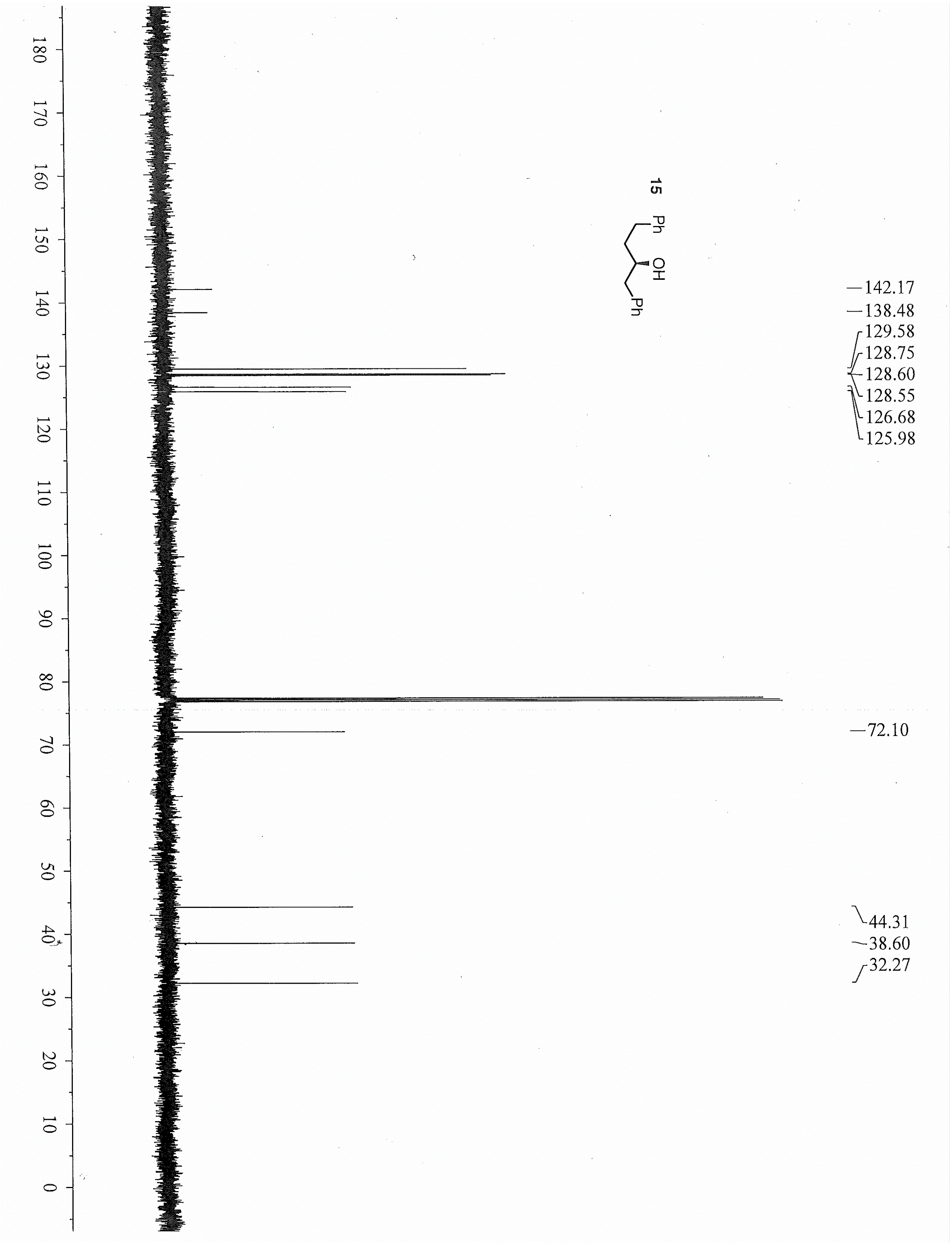
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38.60
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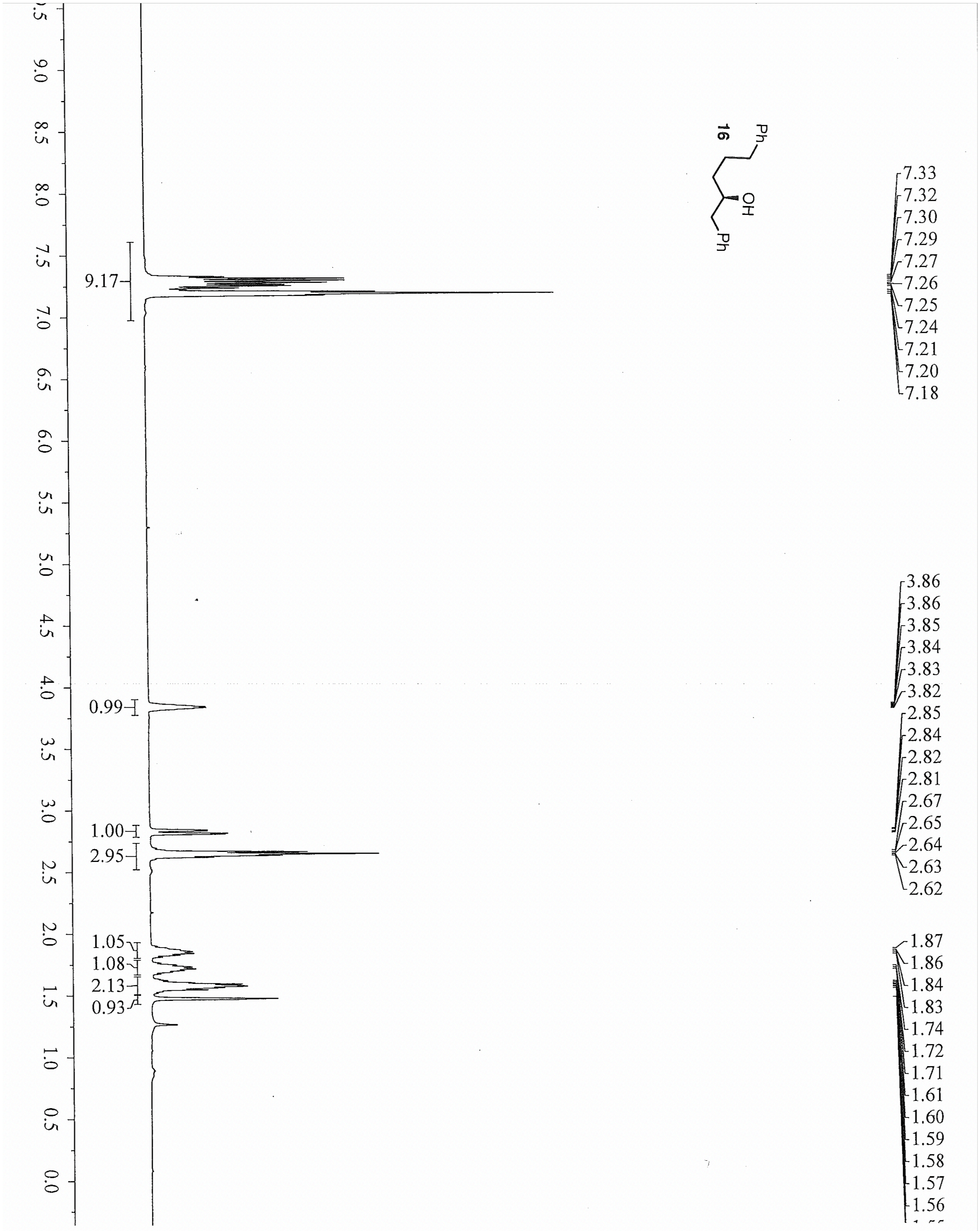
10
0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7.20
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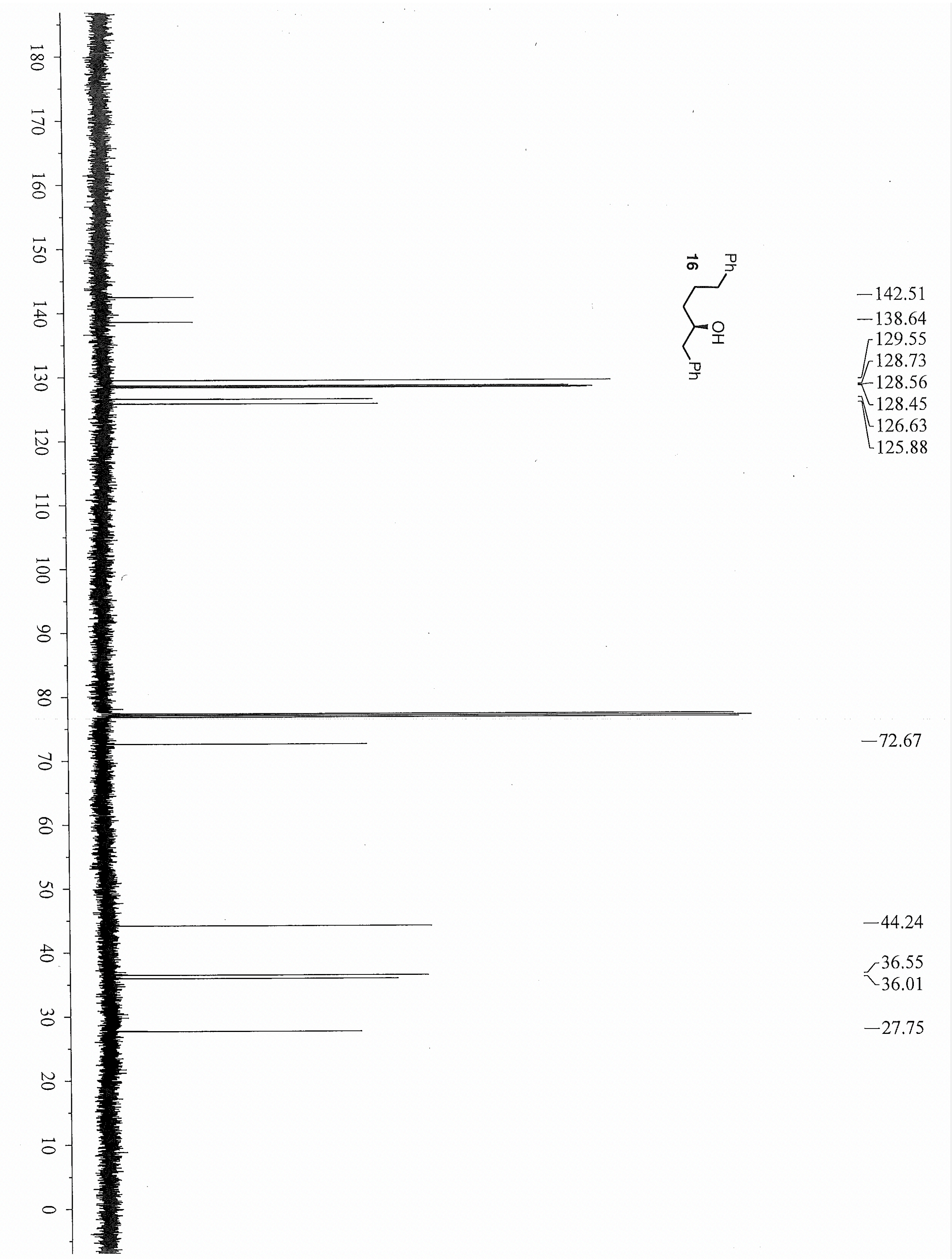
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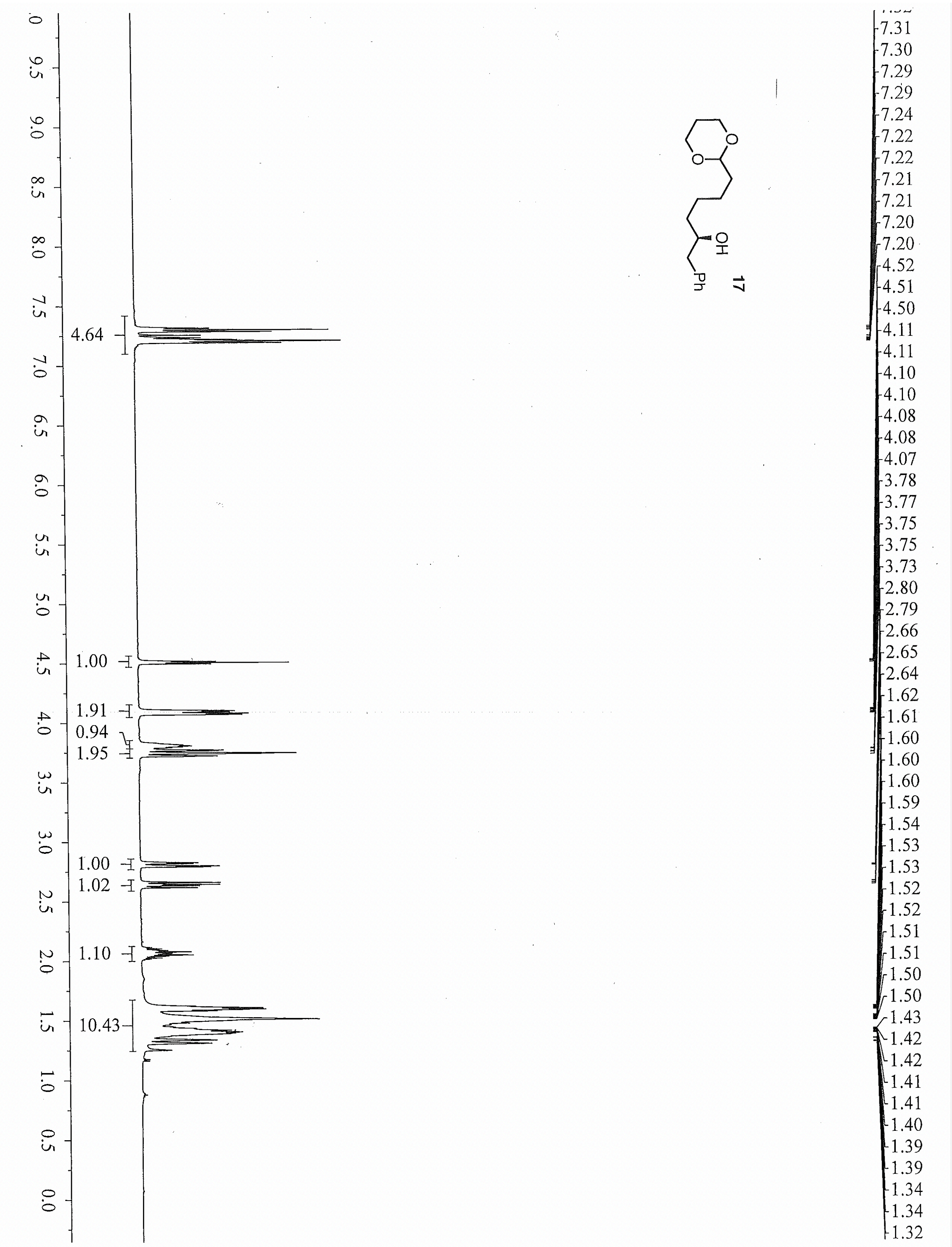
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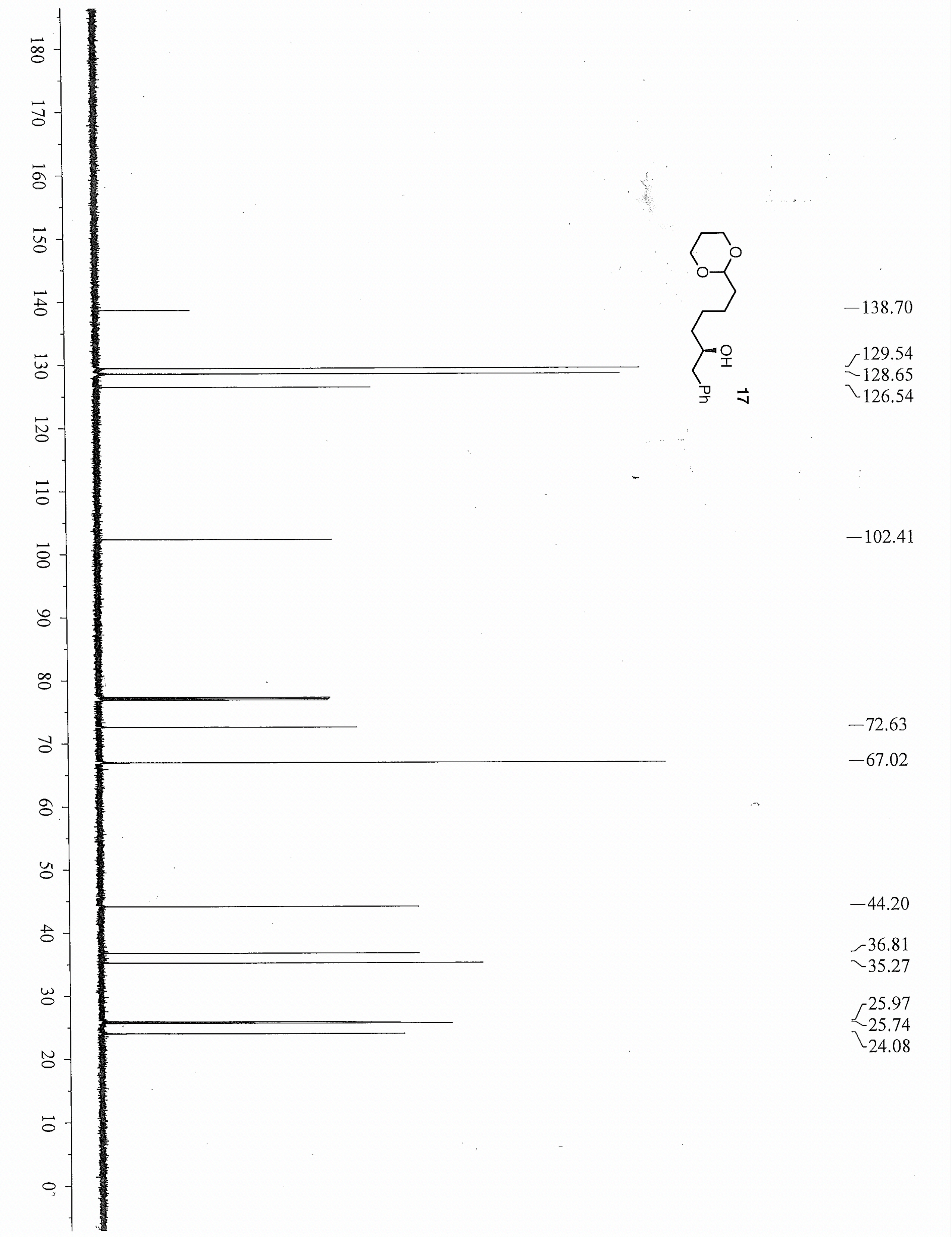
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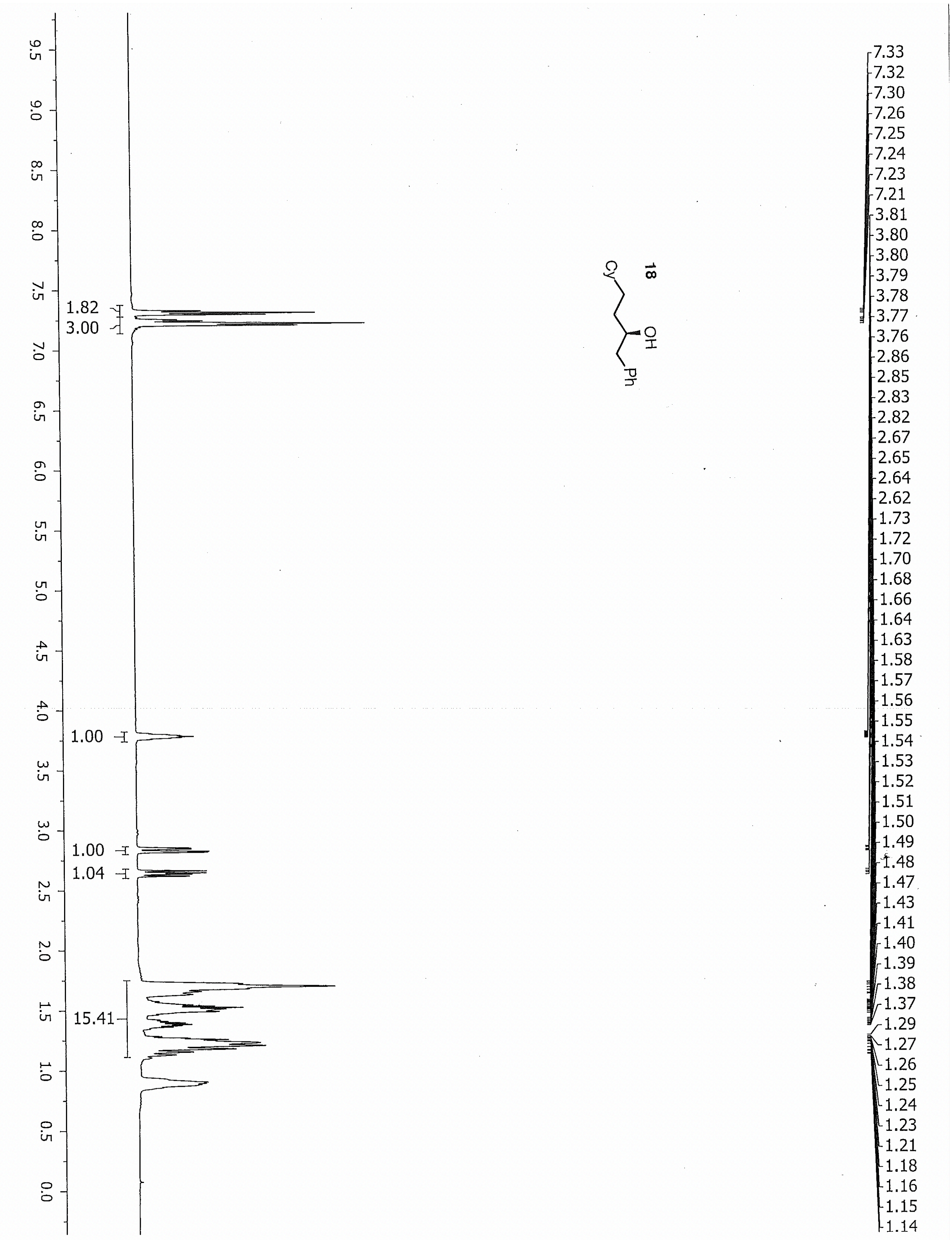
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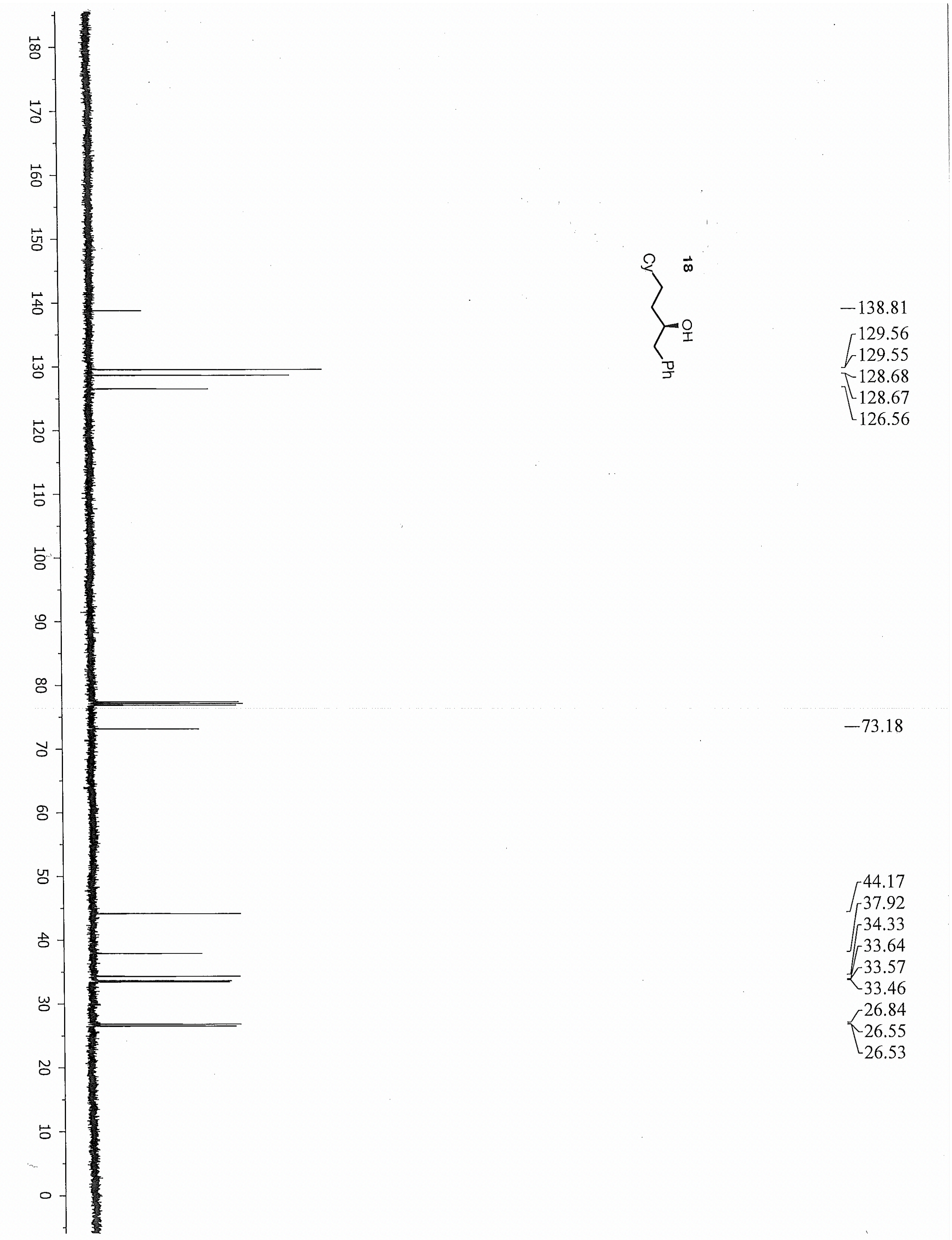
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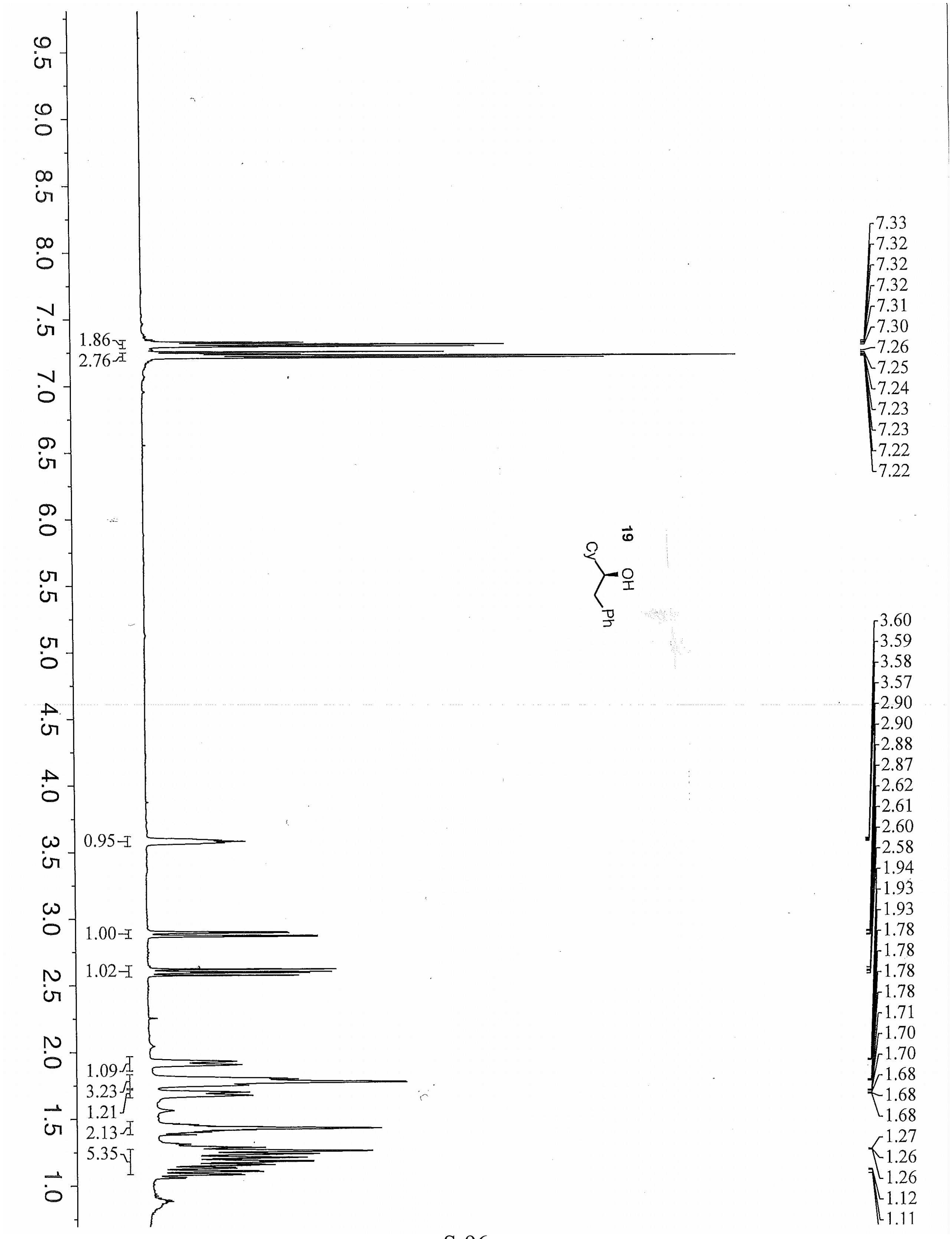
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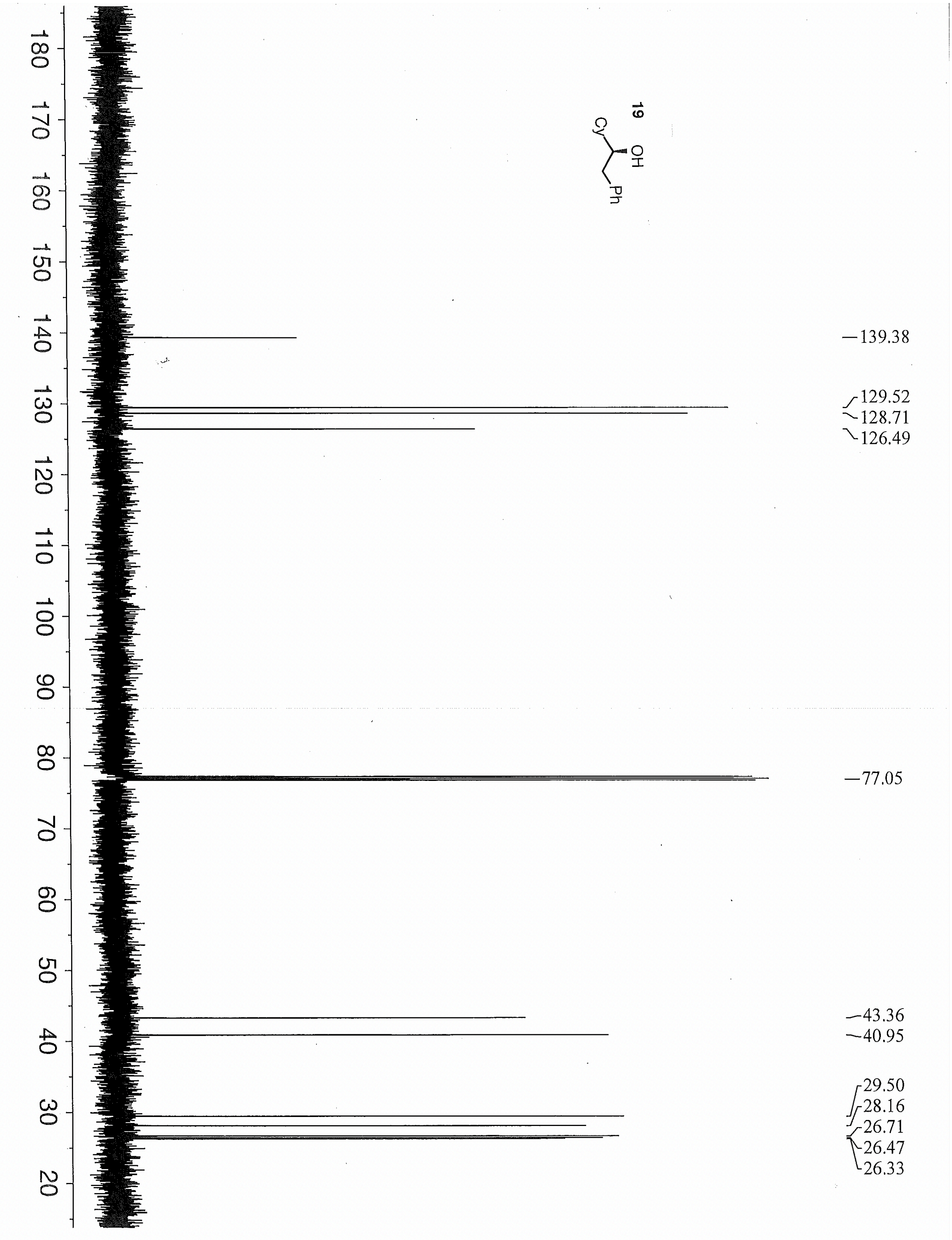
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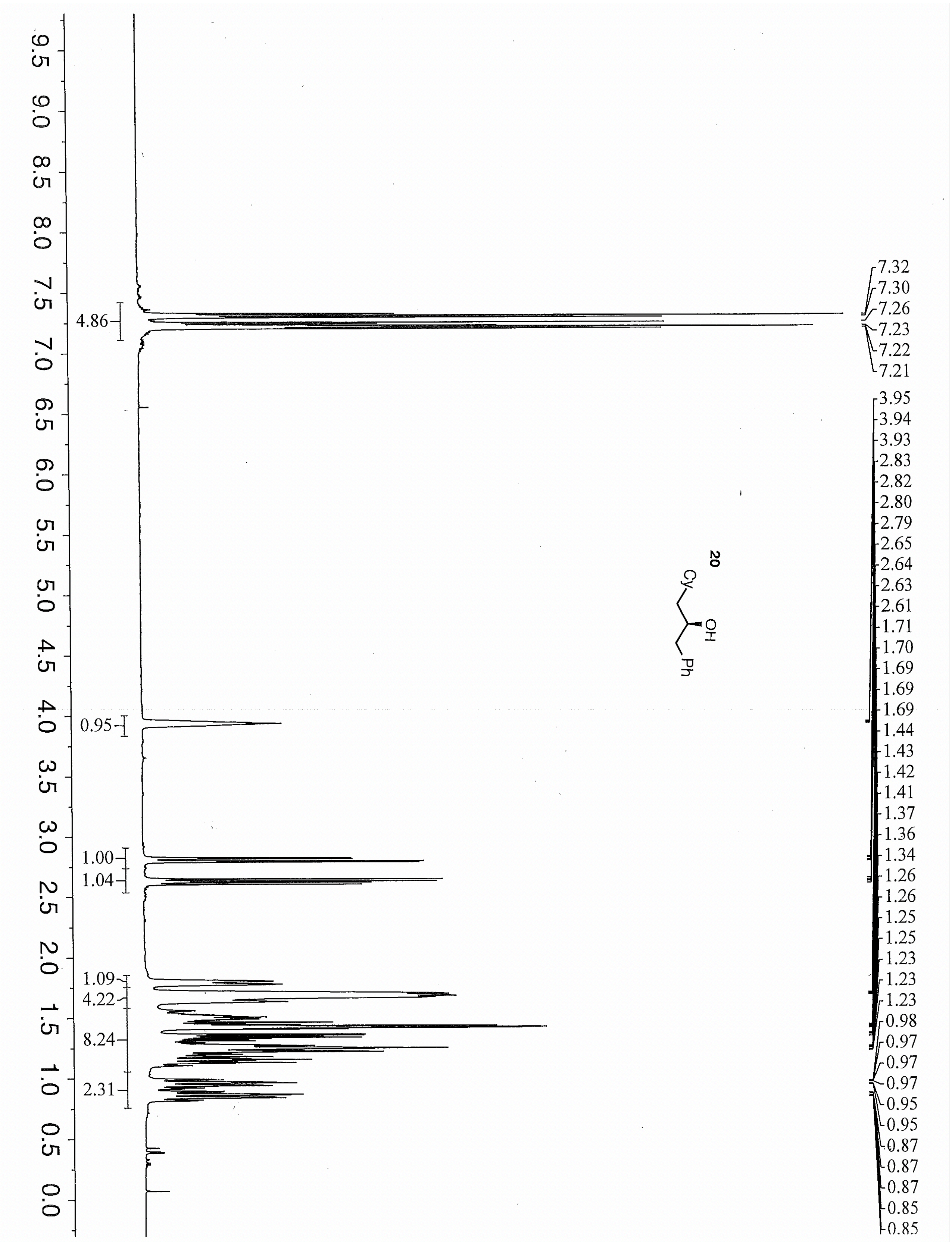
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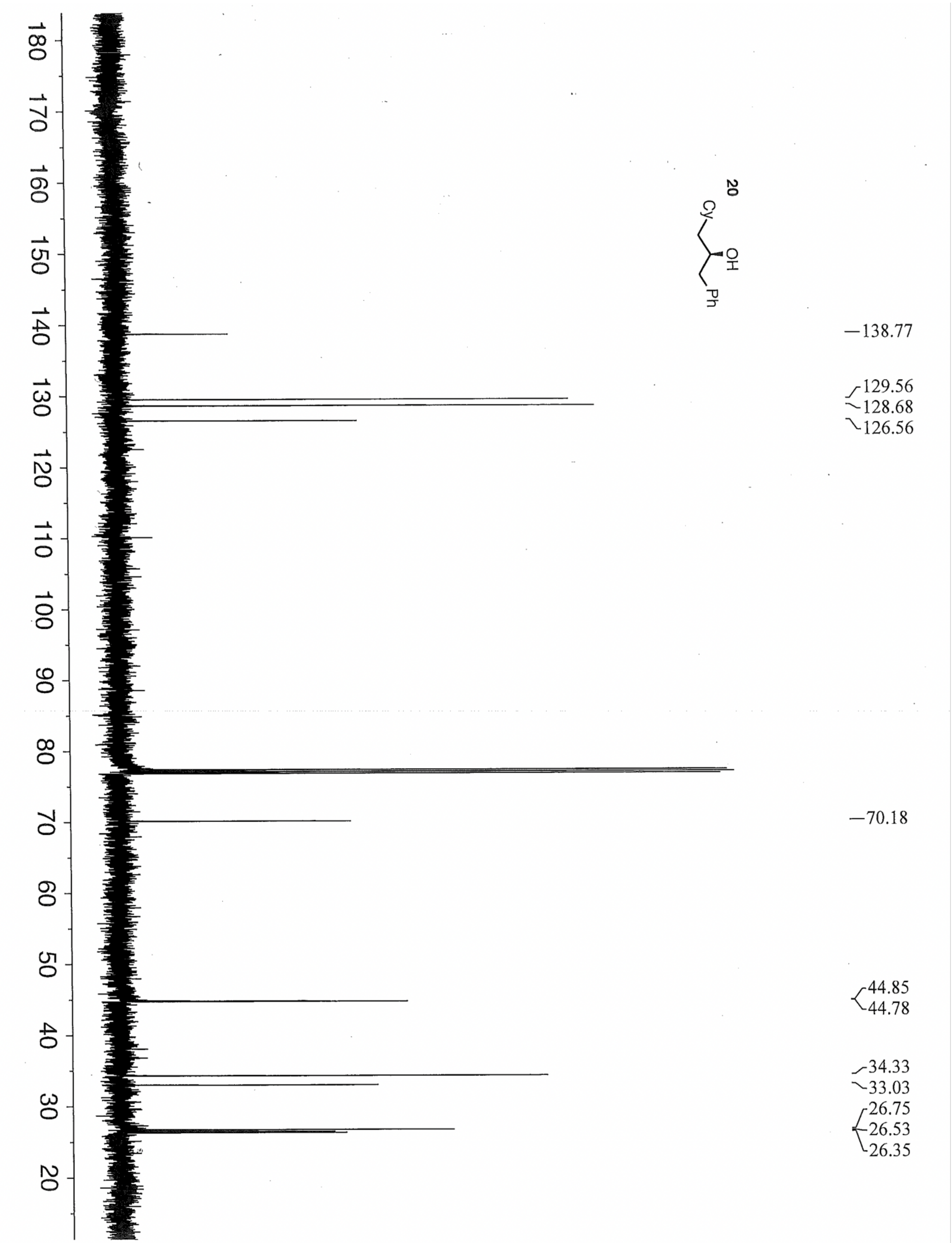
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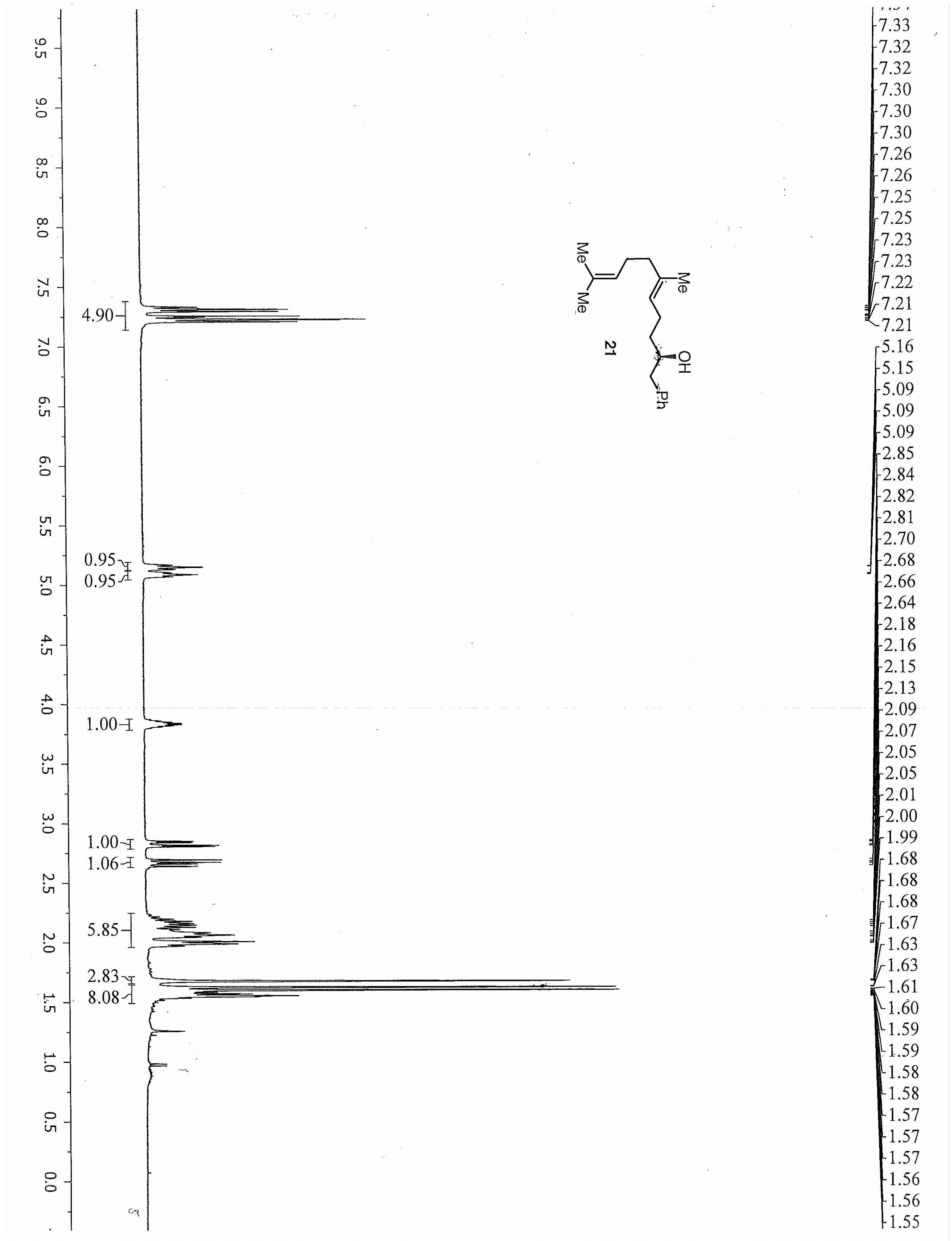
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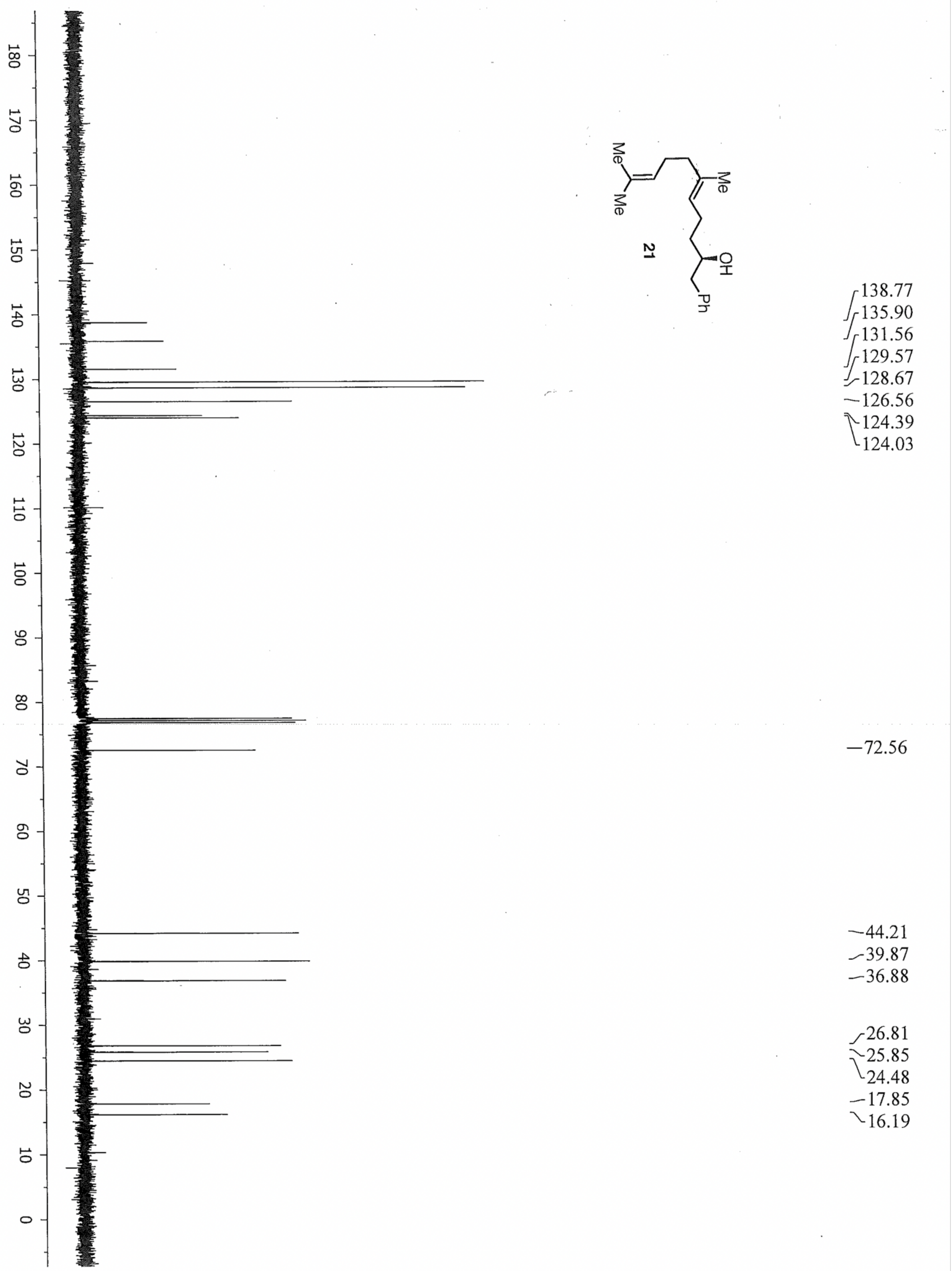

21

Me
Me
Me
OH
Ph
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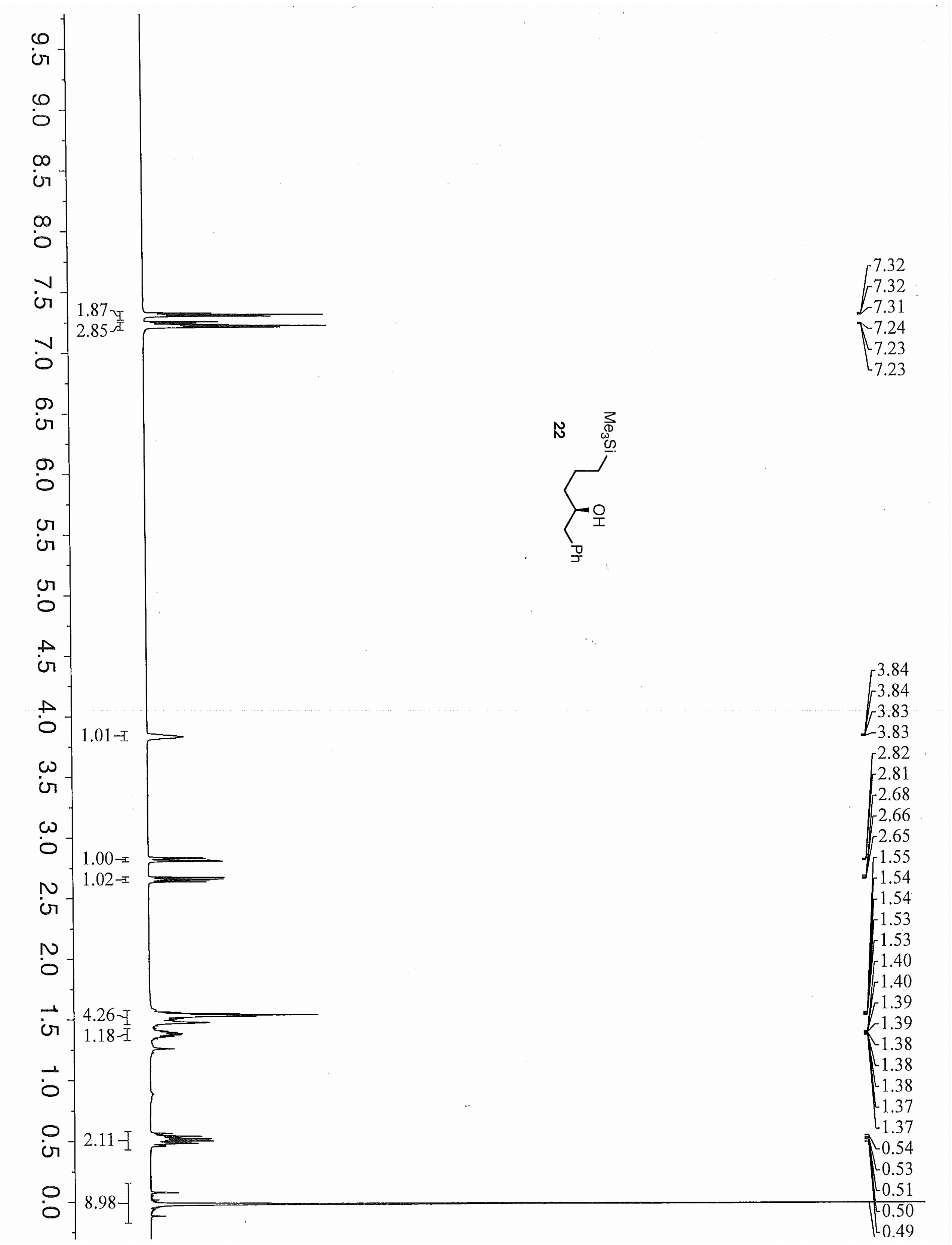

22

Me3Si
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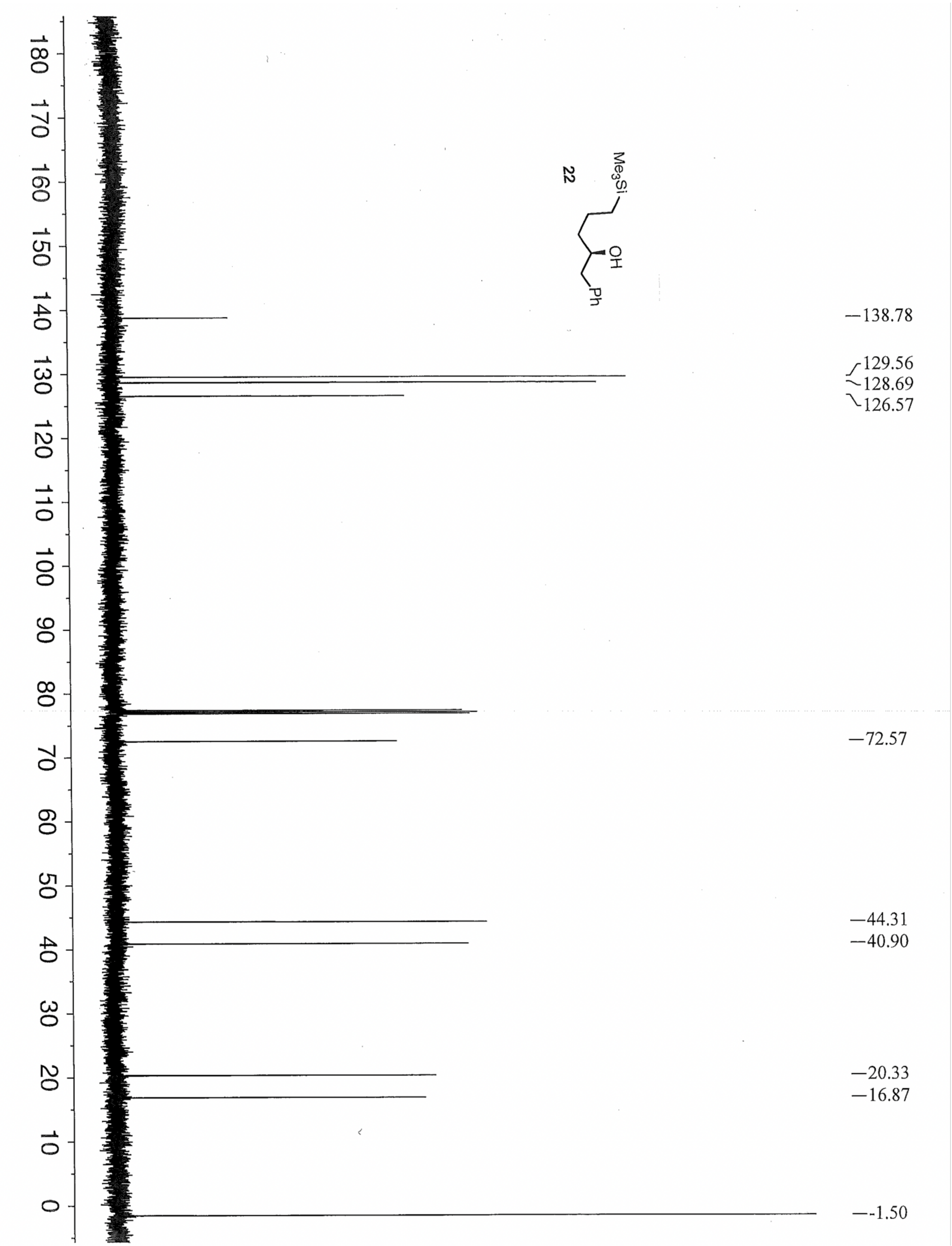
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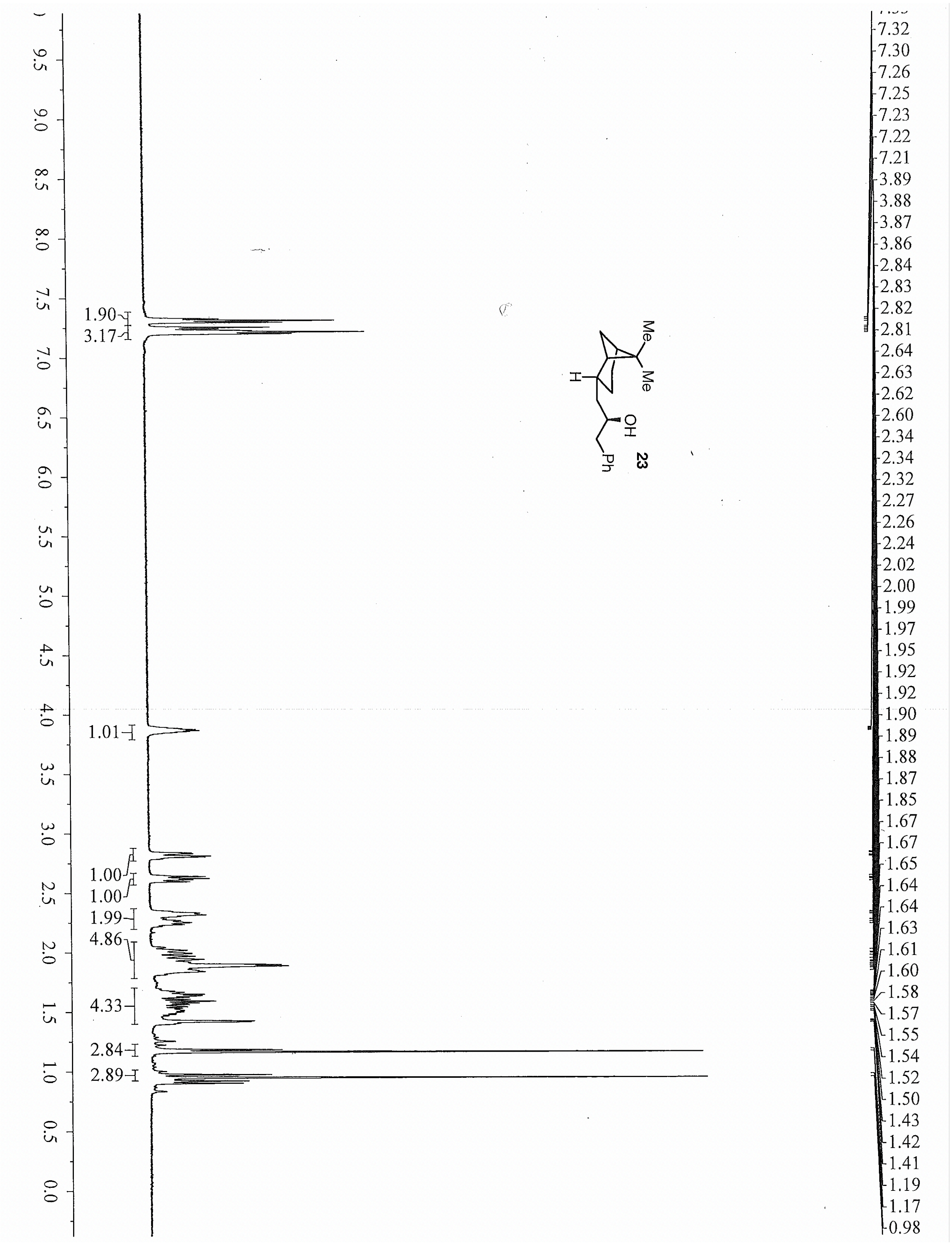
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Me
H
OH
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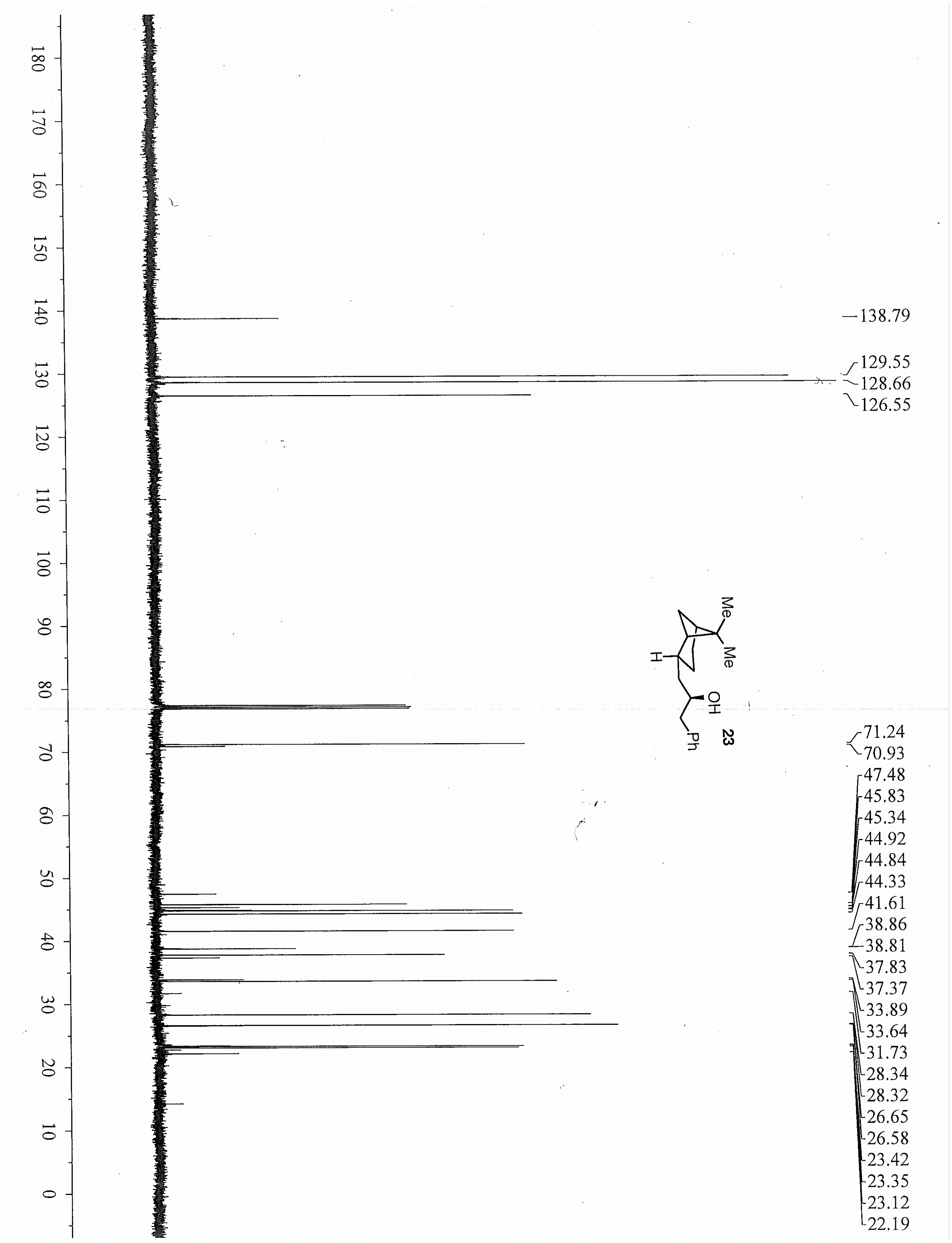
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Me
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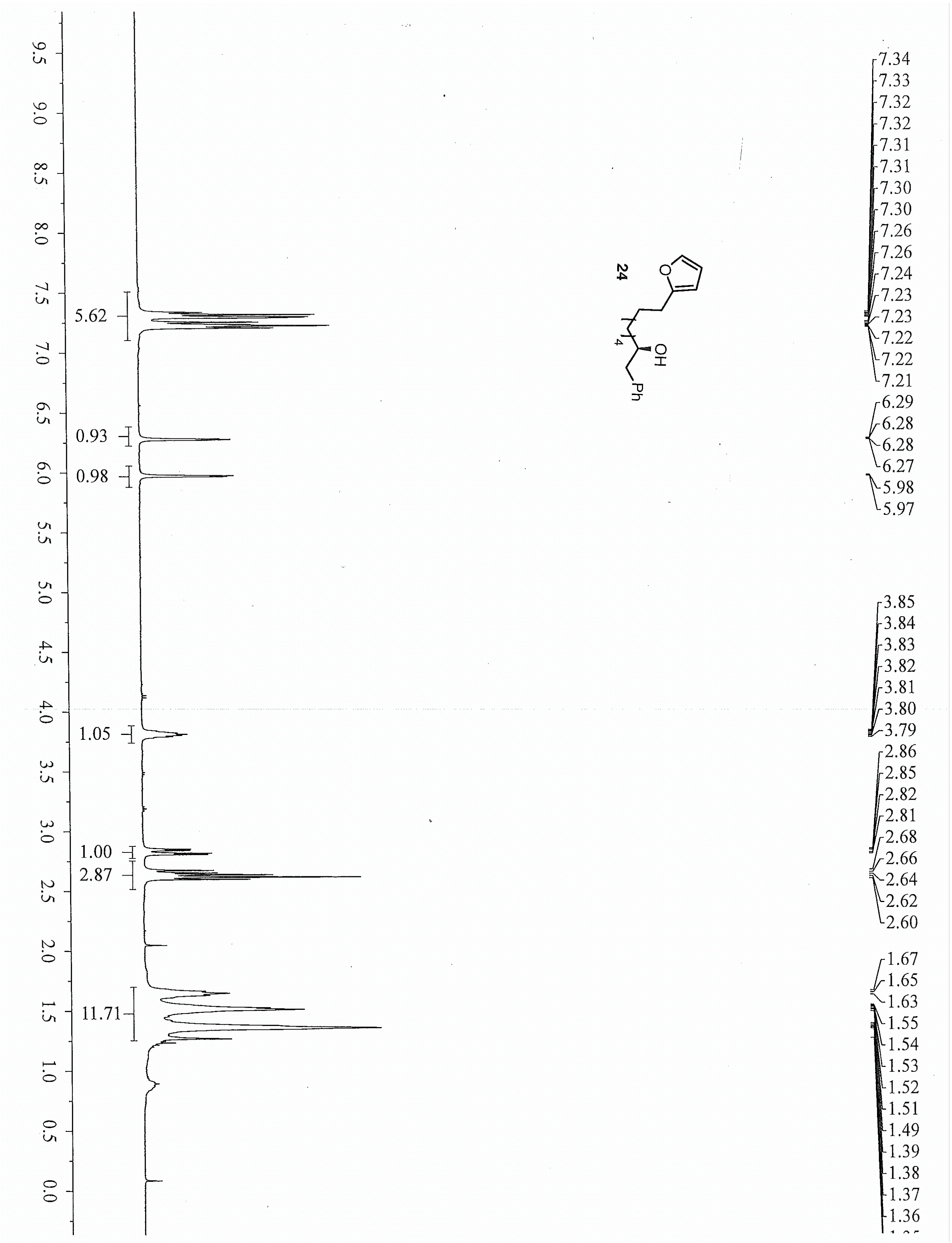
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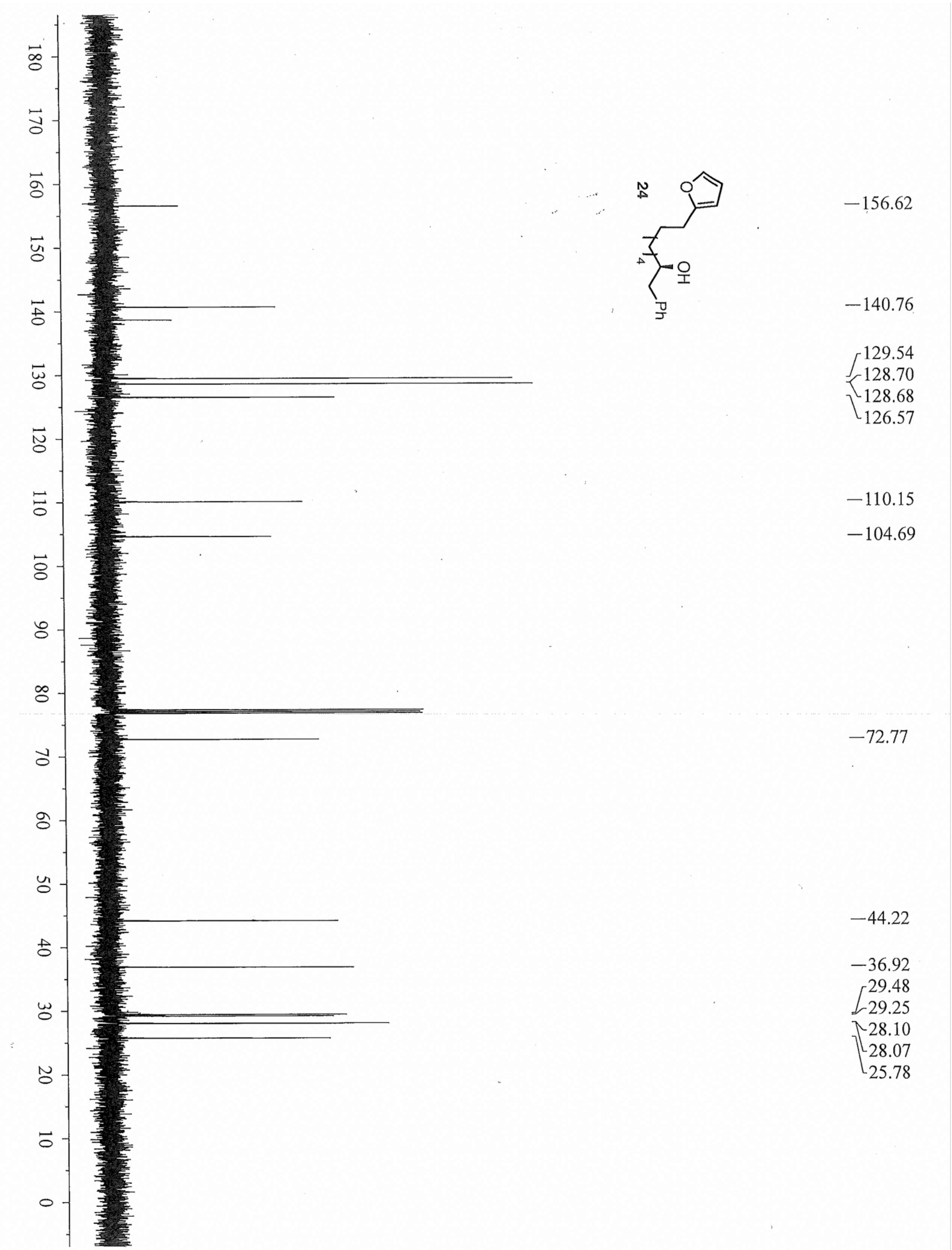
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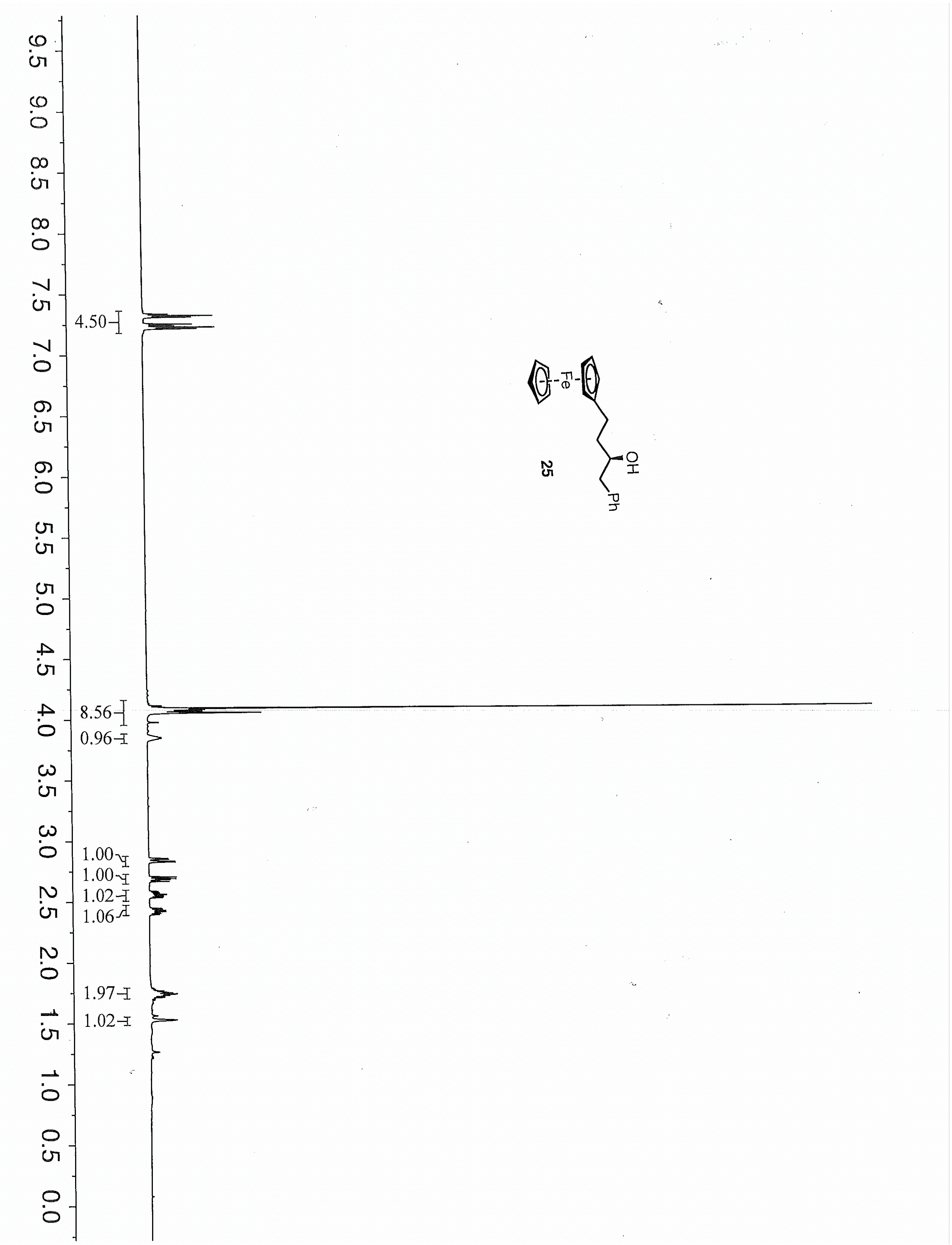
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Ph
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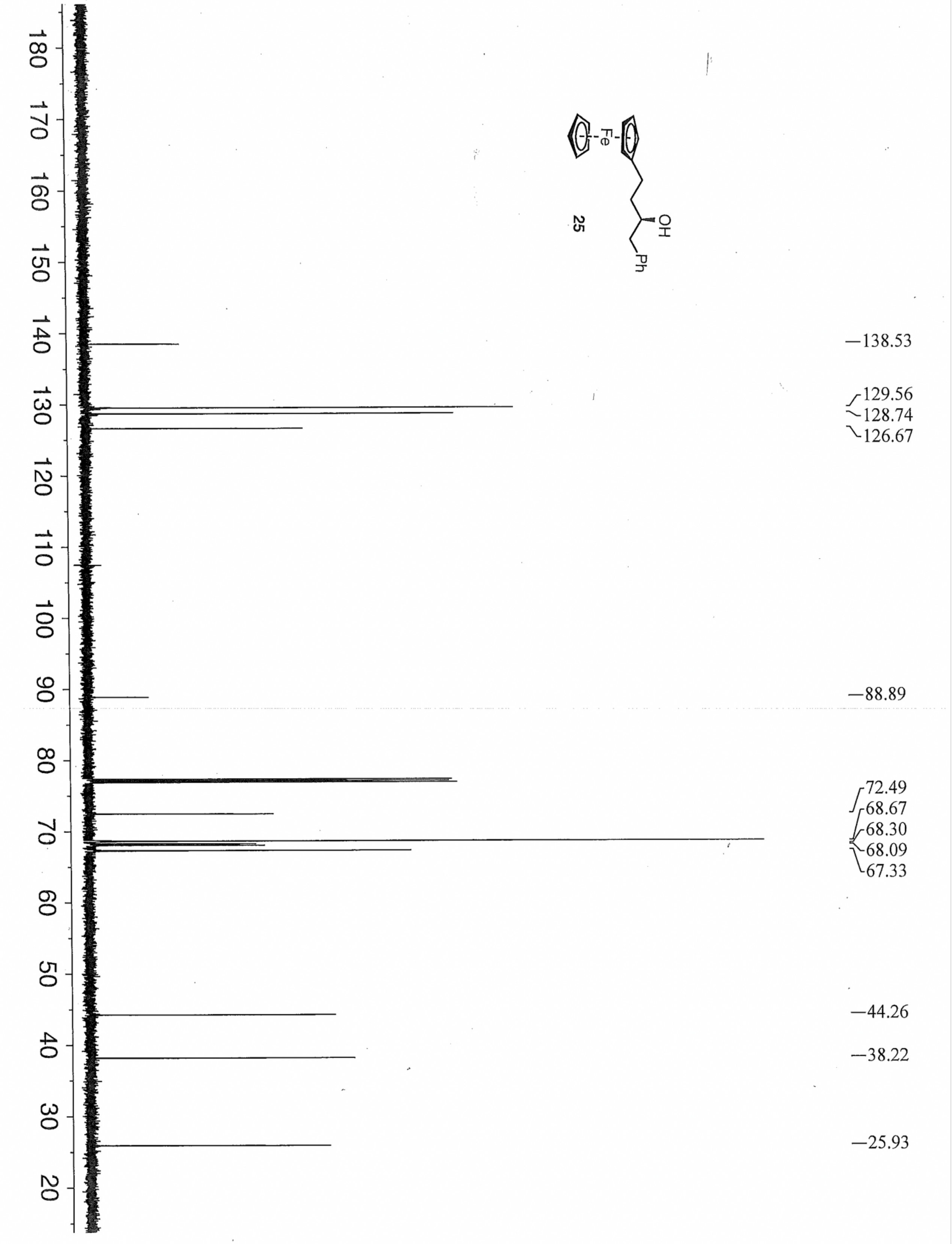
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44.26
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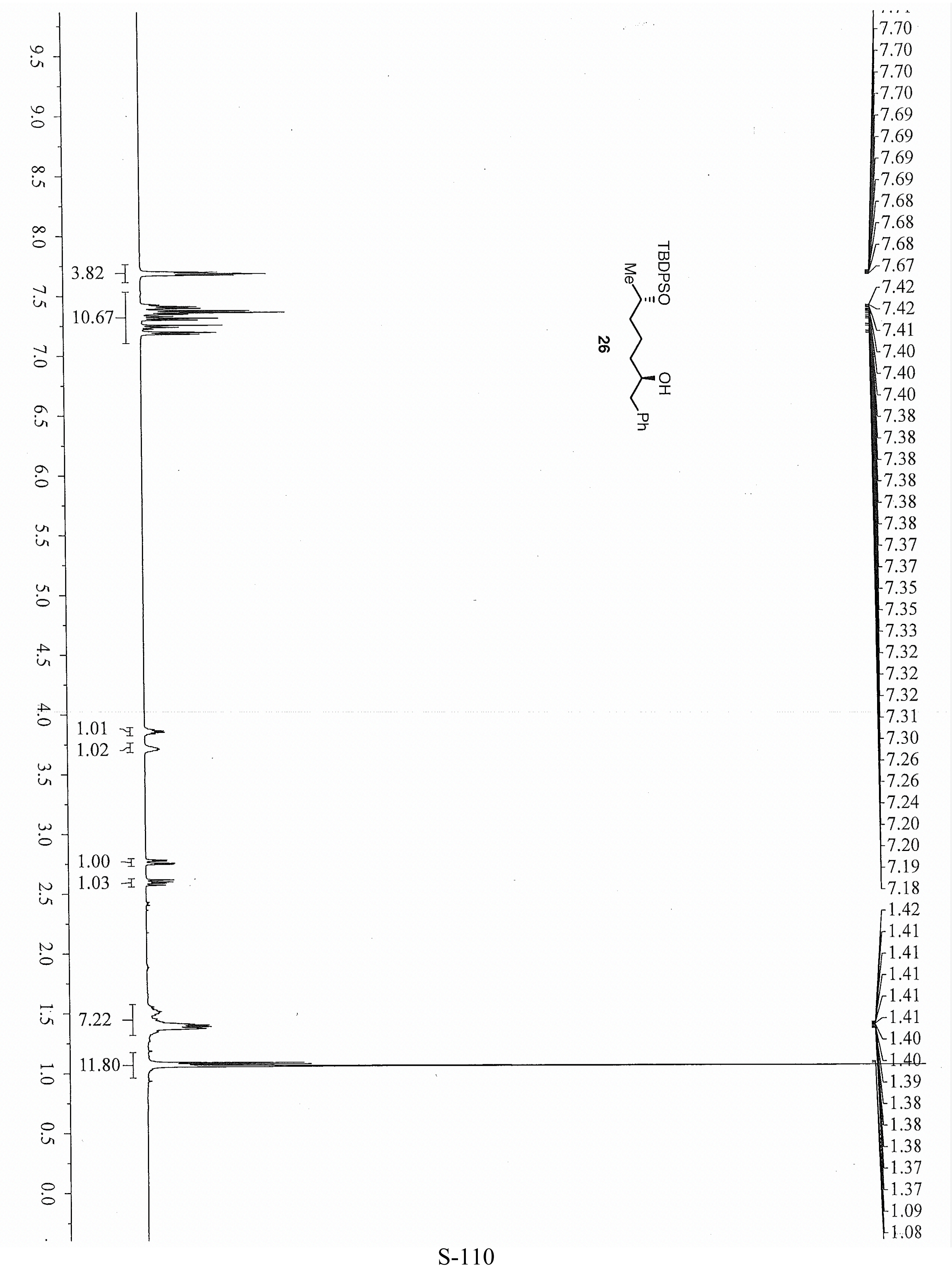
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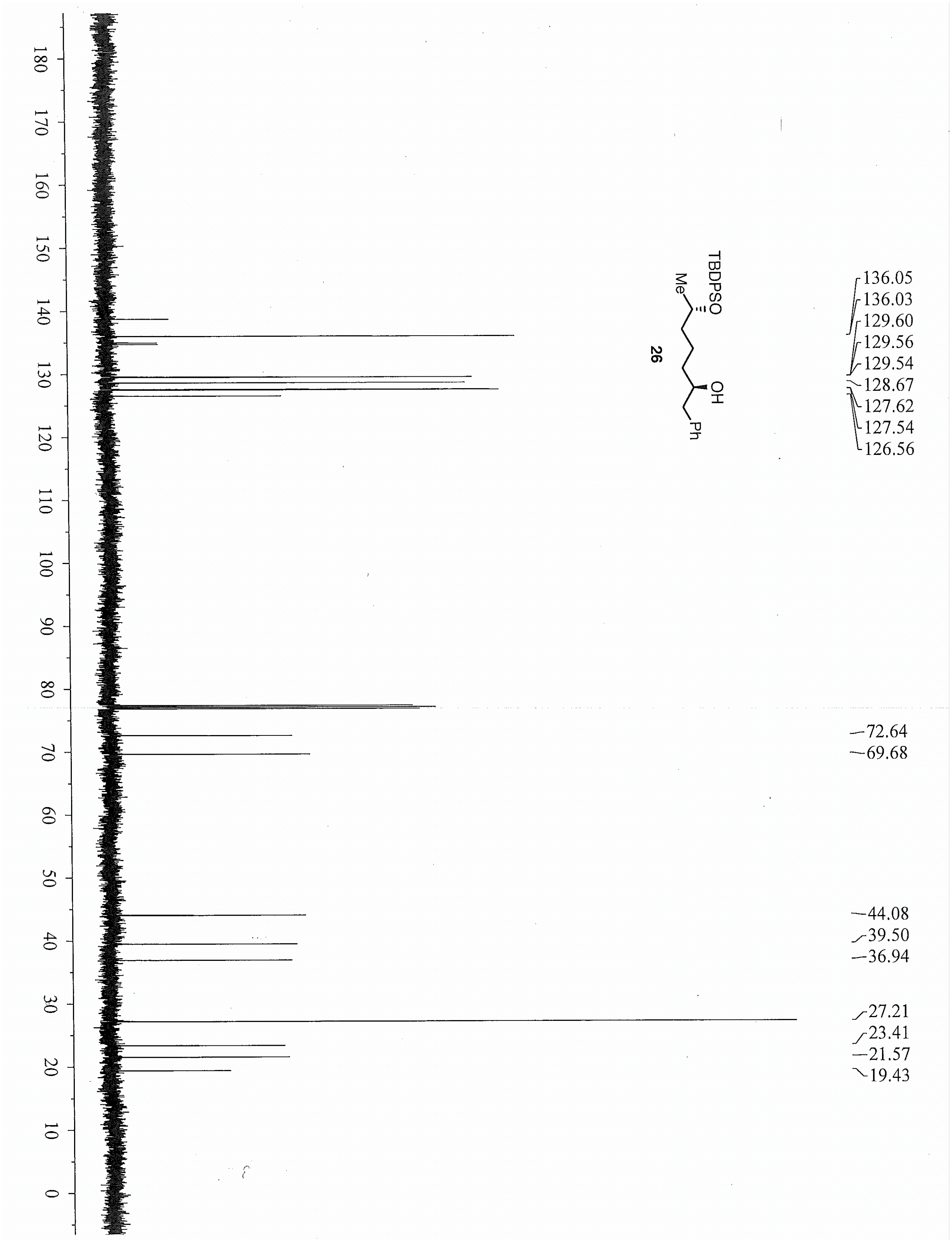
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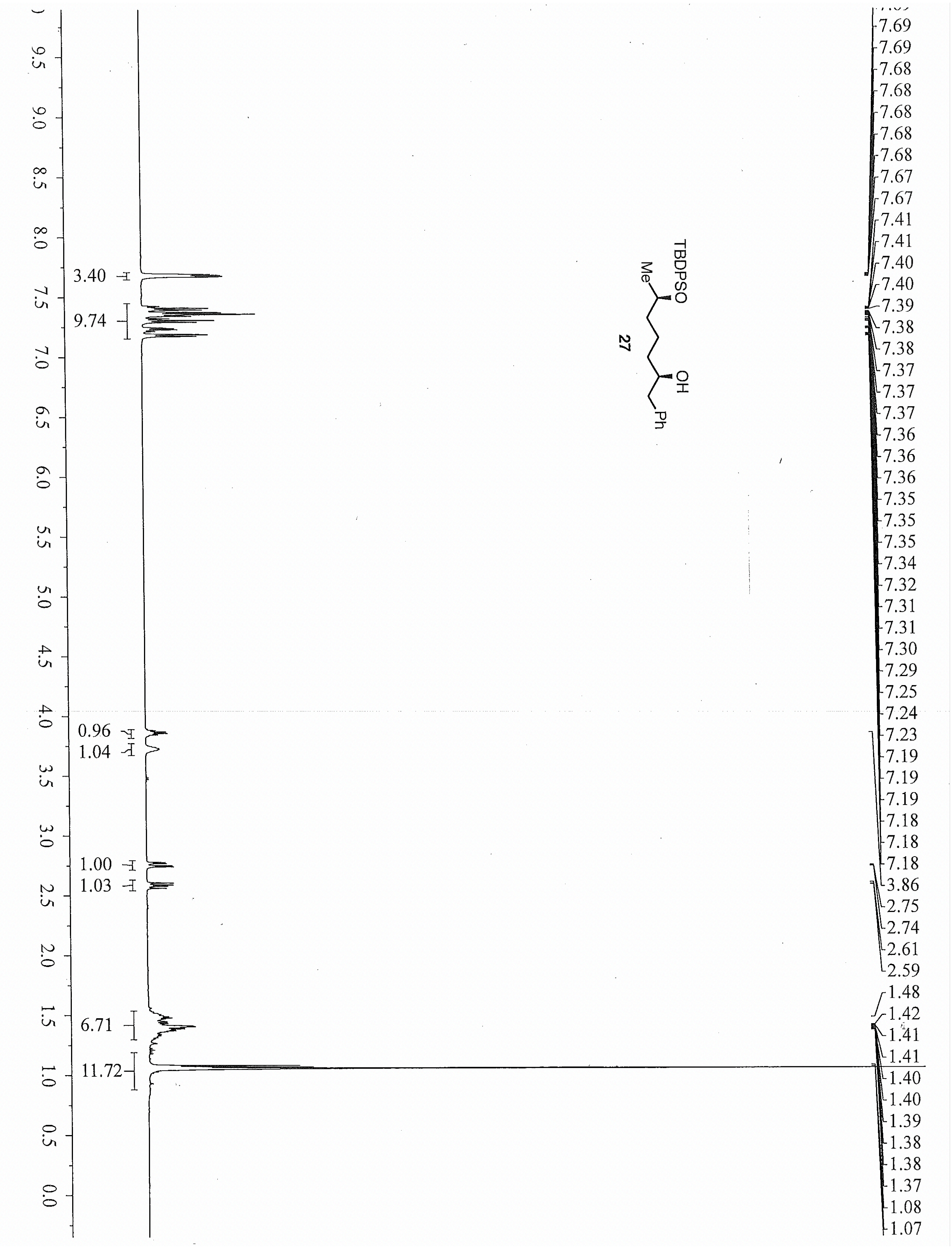
26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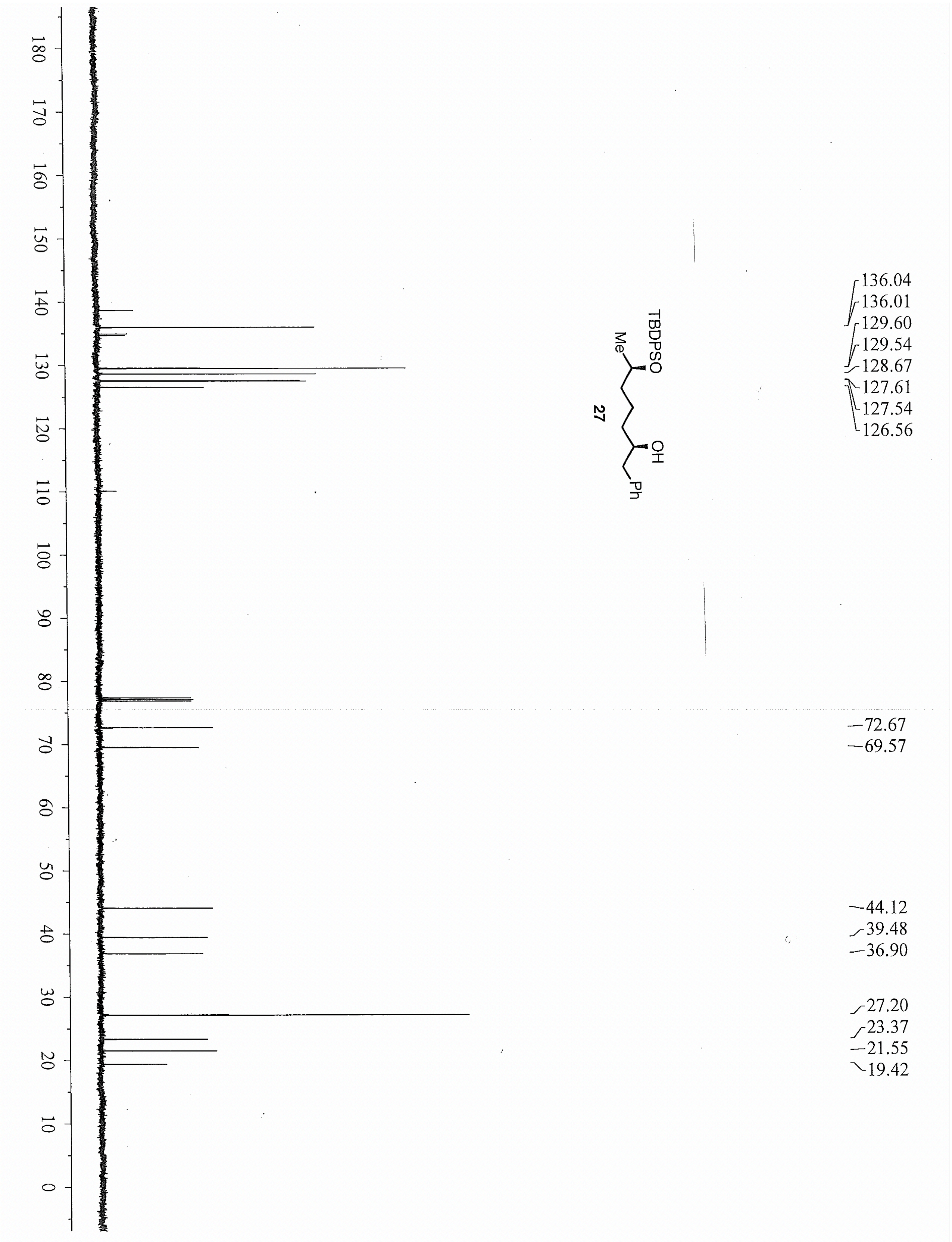
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21.55
19.42
TBDPSO
Me
OH
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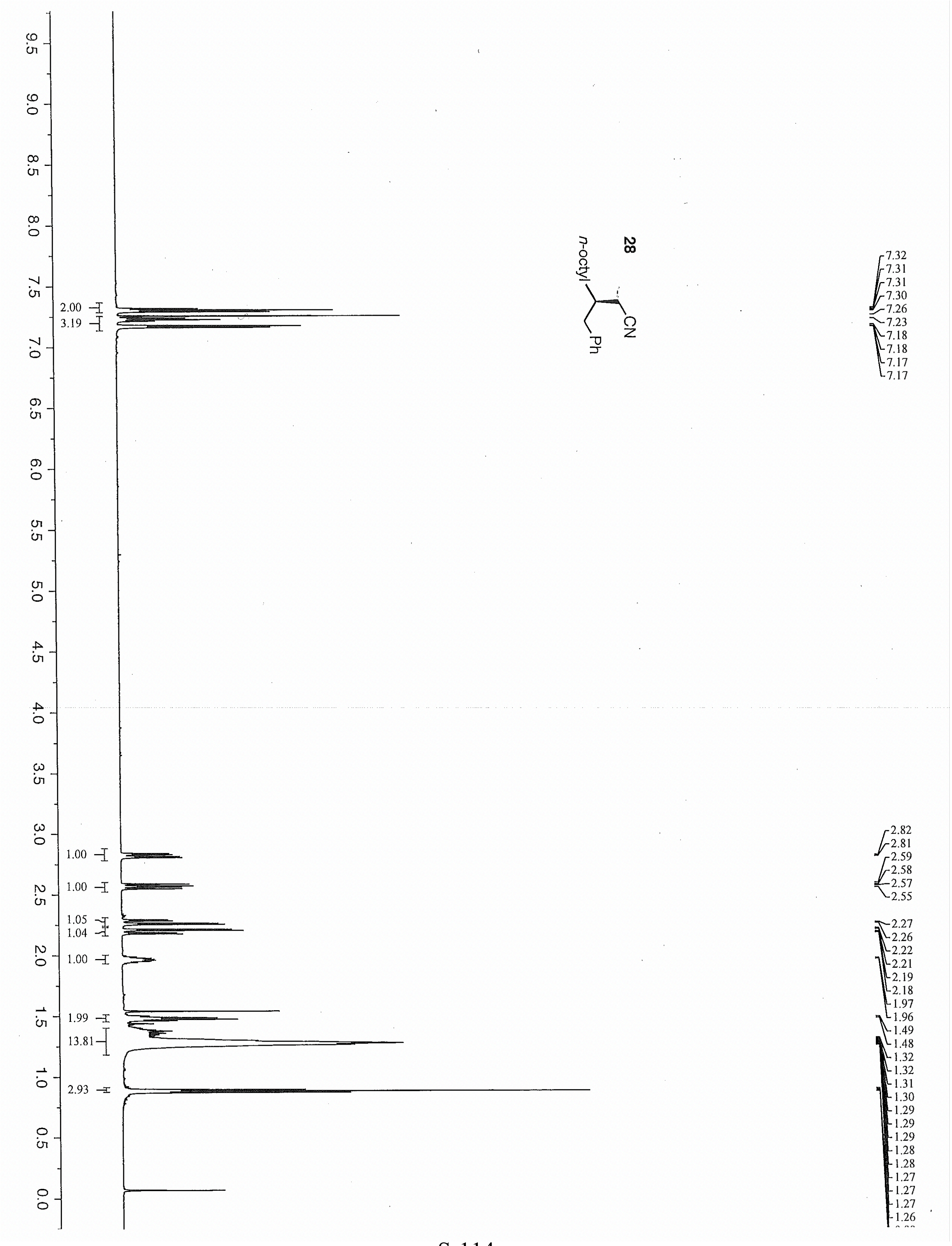
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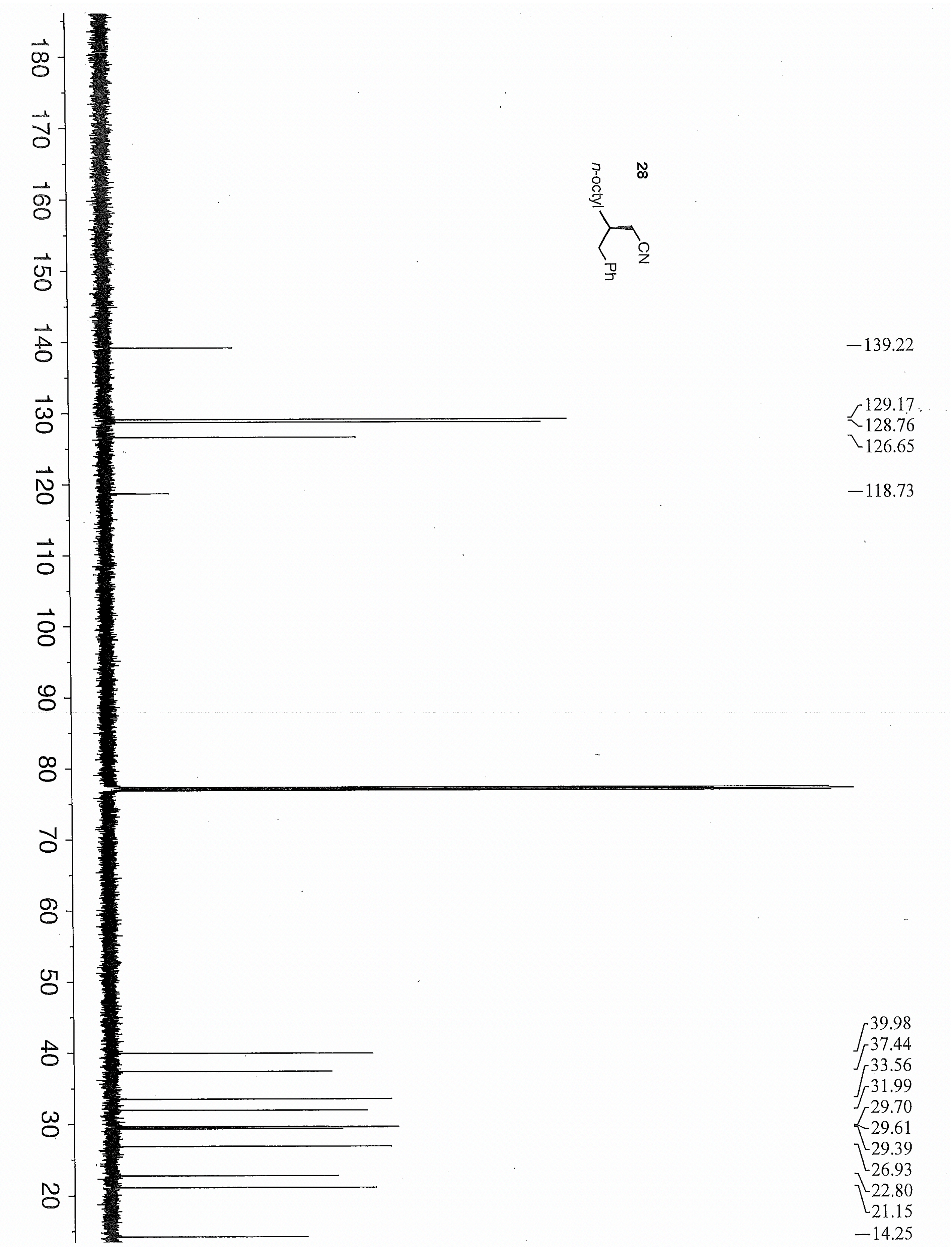

28

CN
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Ph
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30
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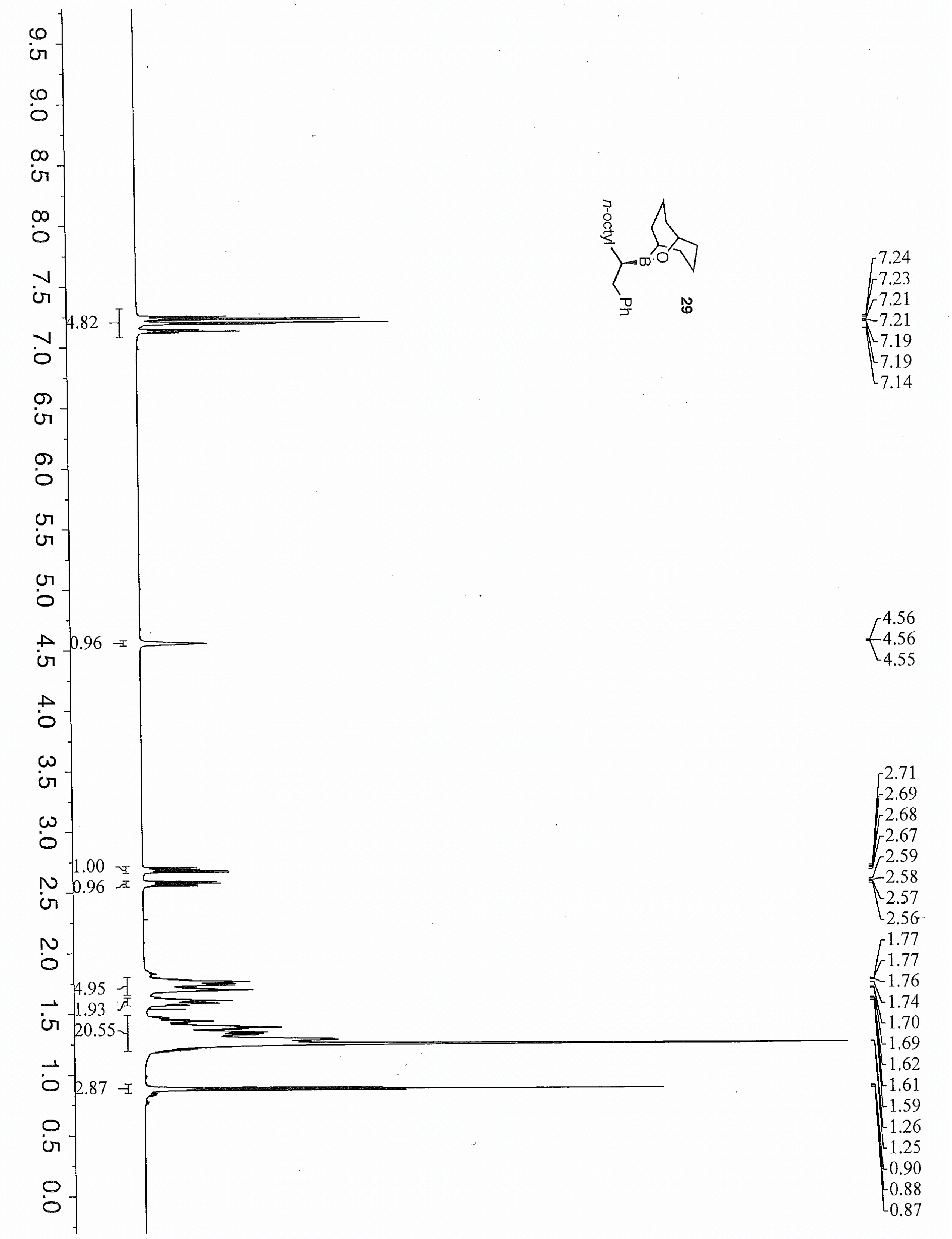
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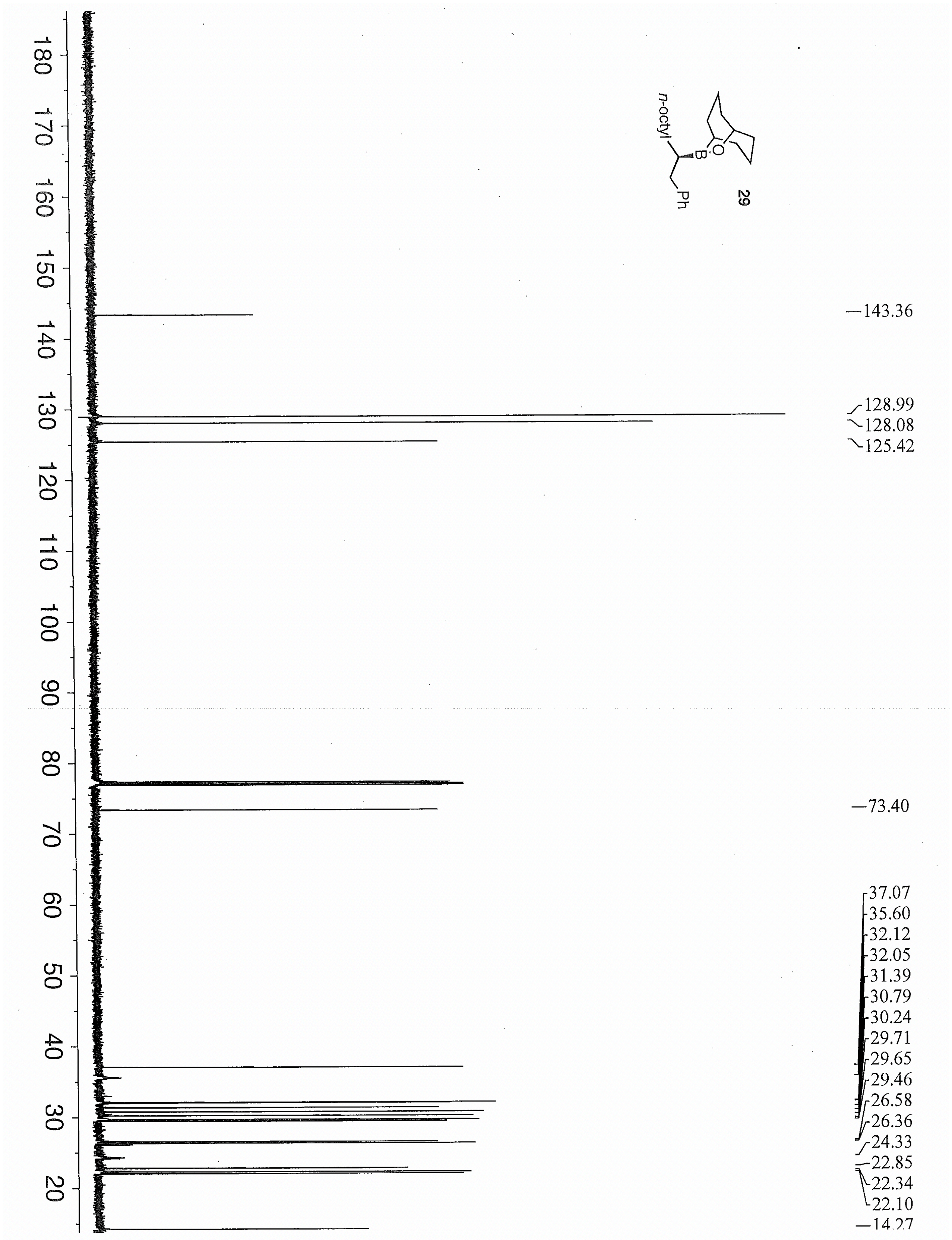
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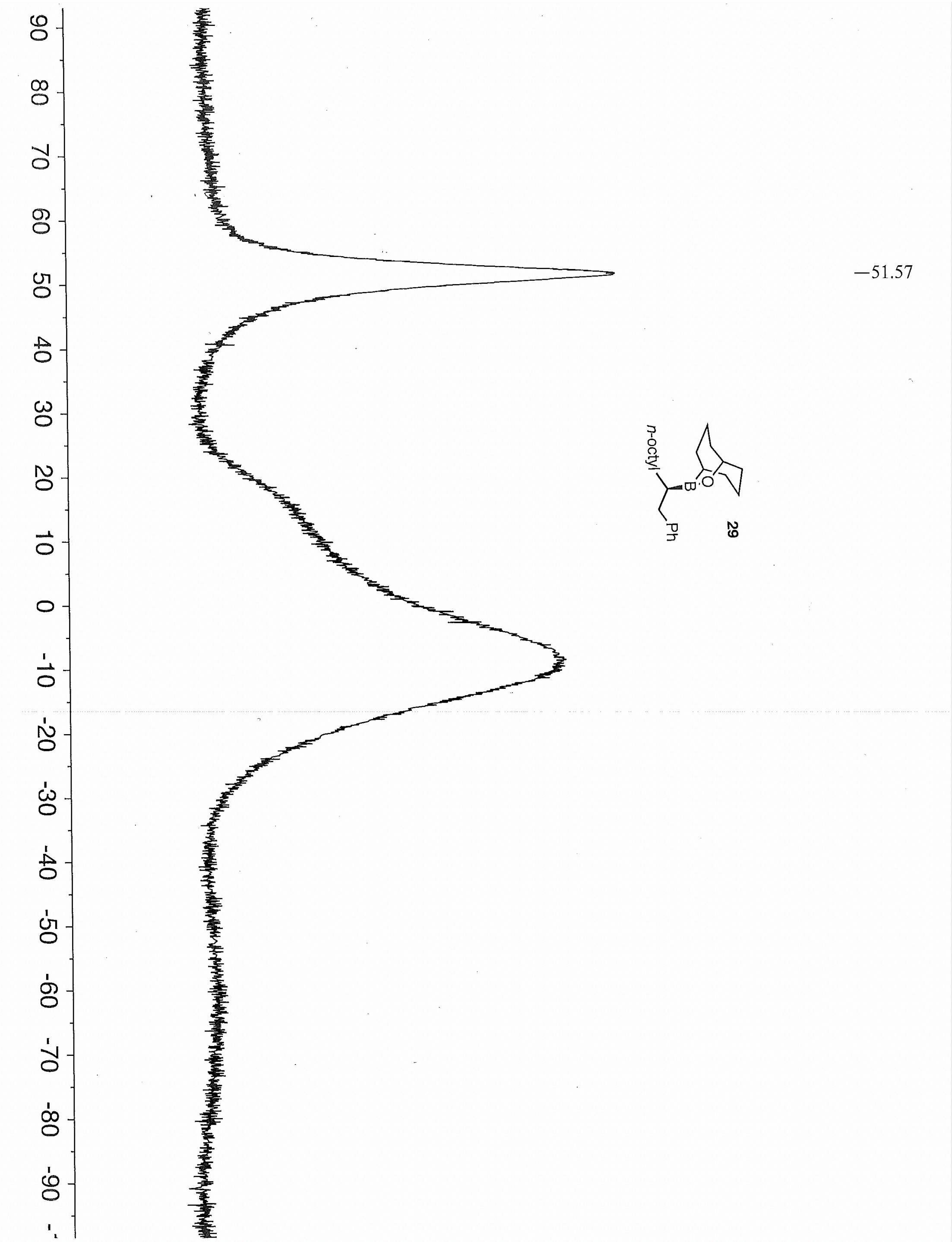
^{11}B NMR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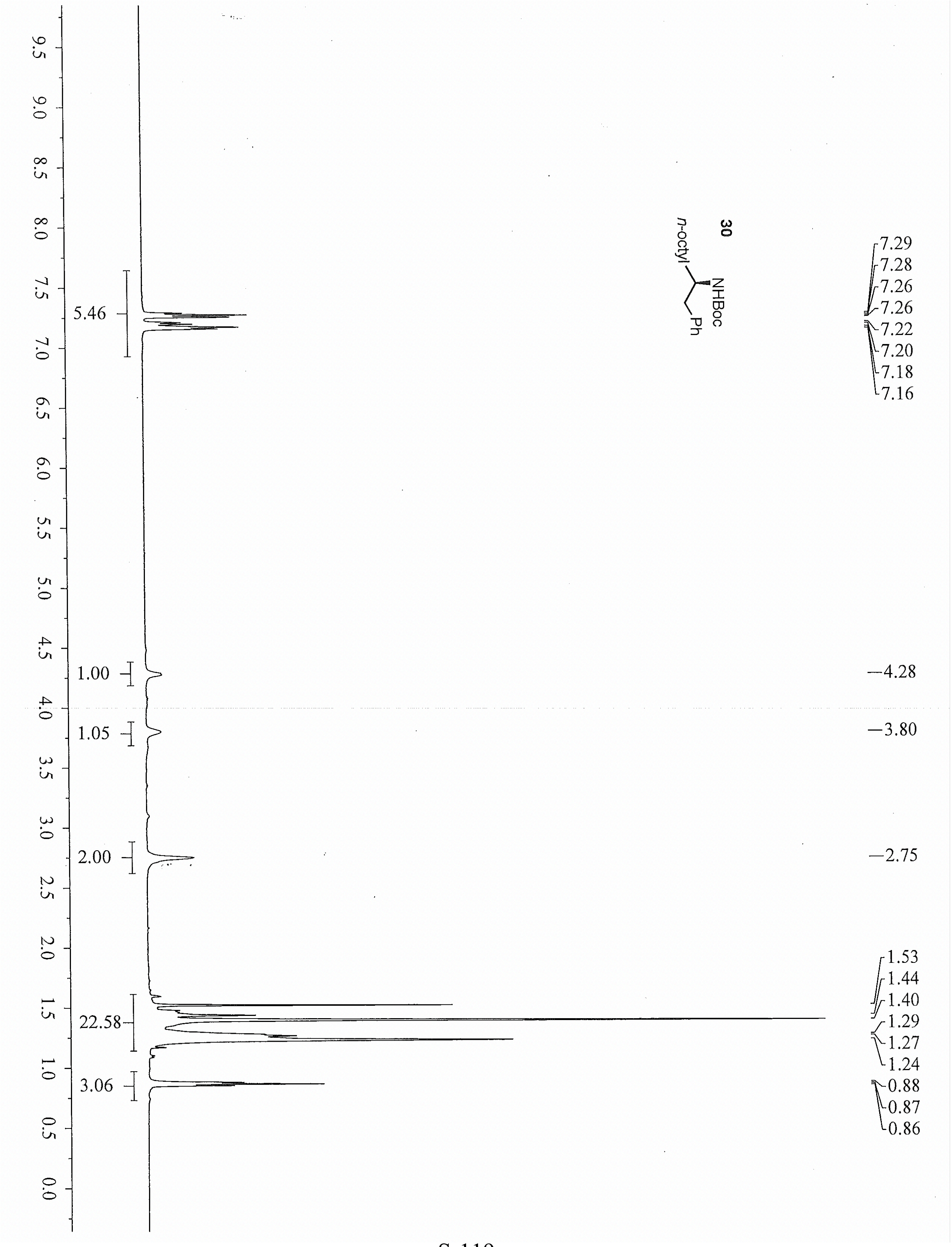
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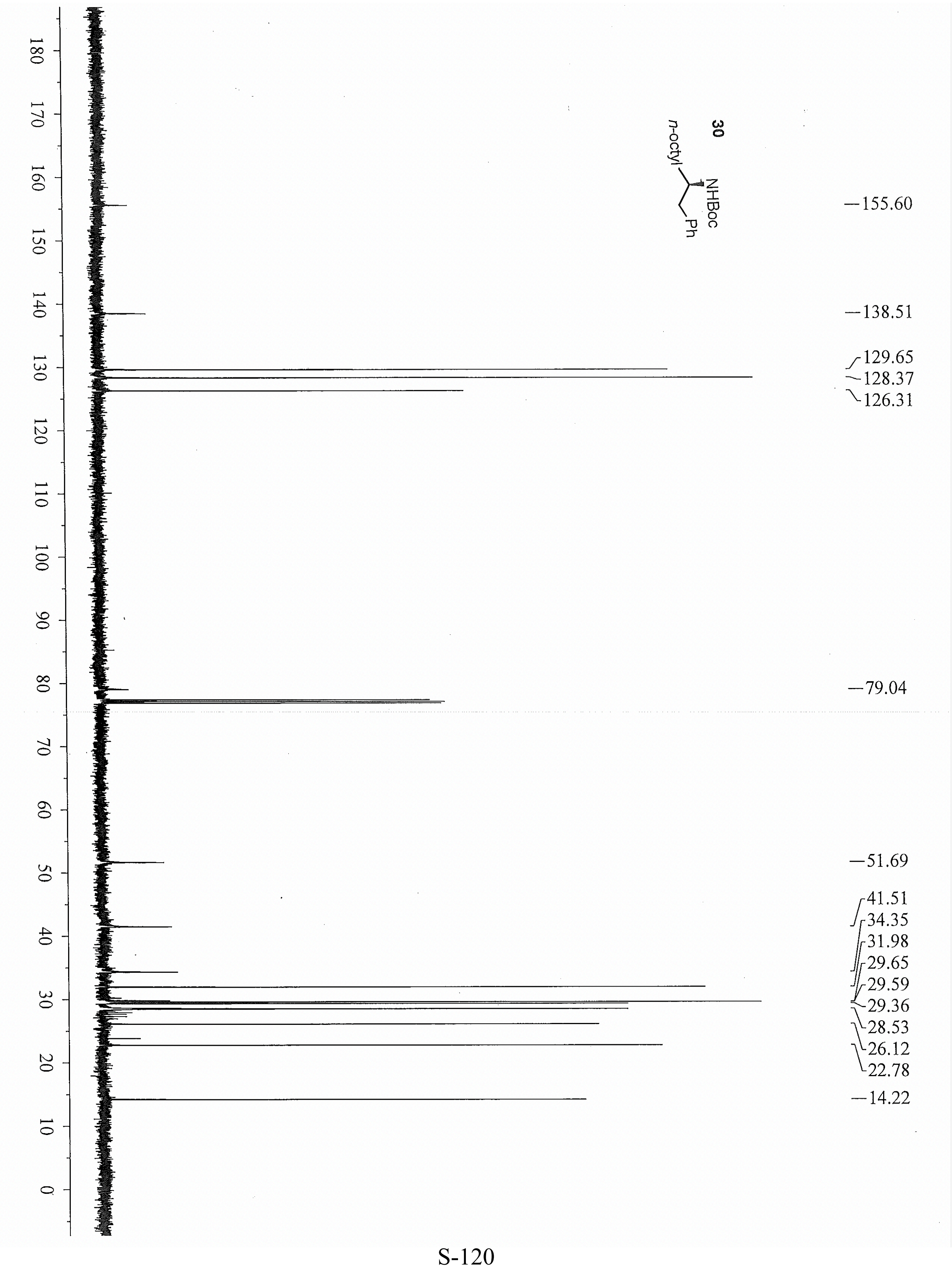
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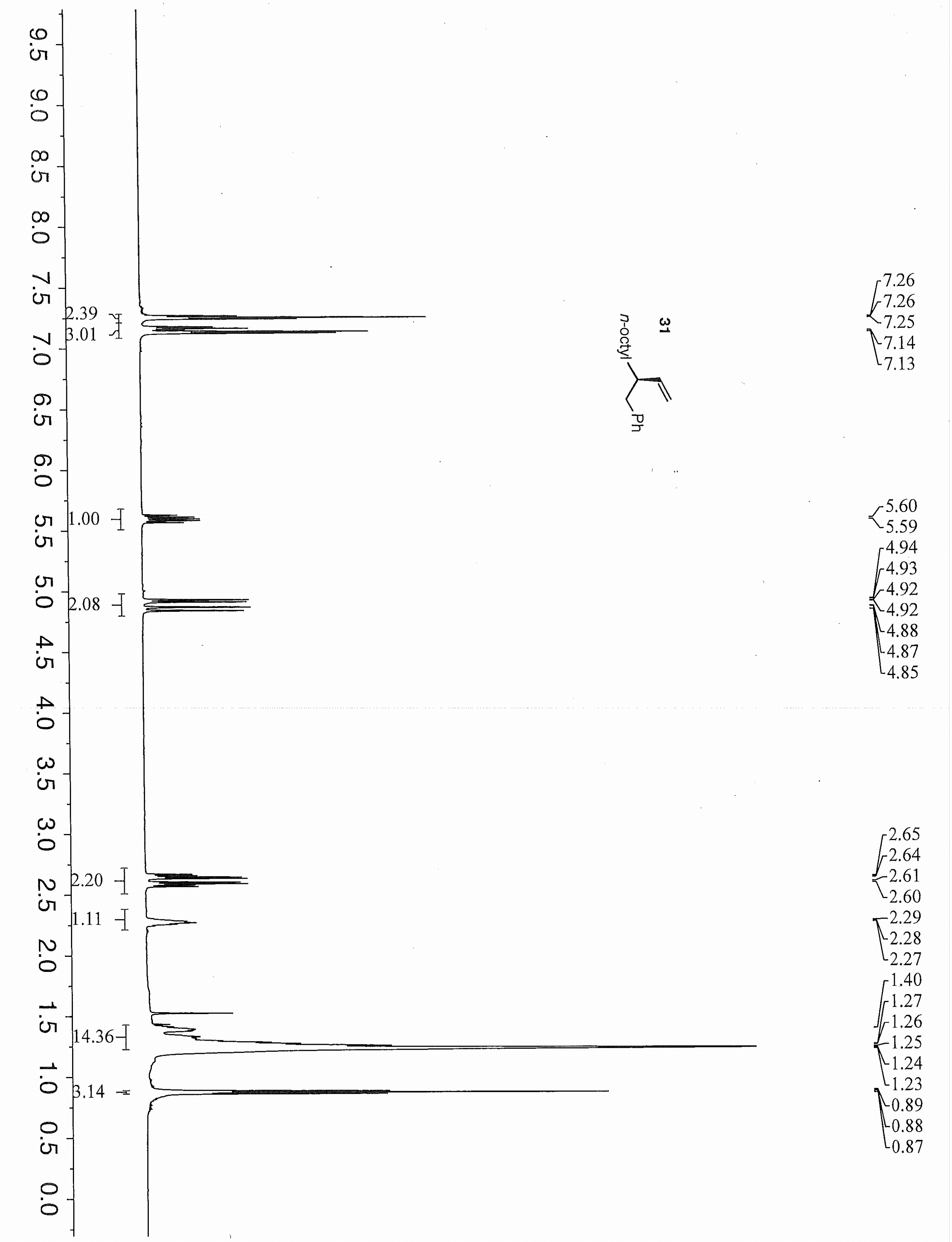

31

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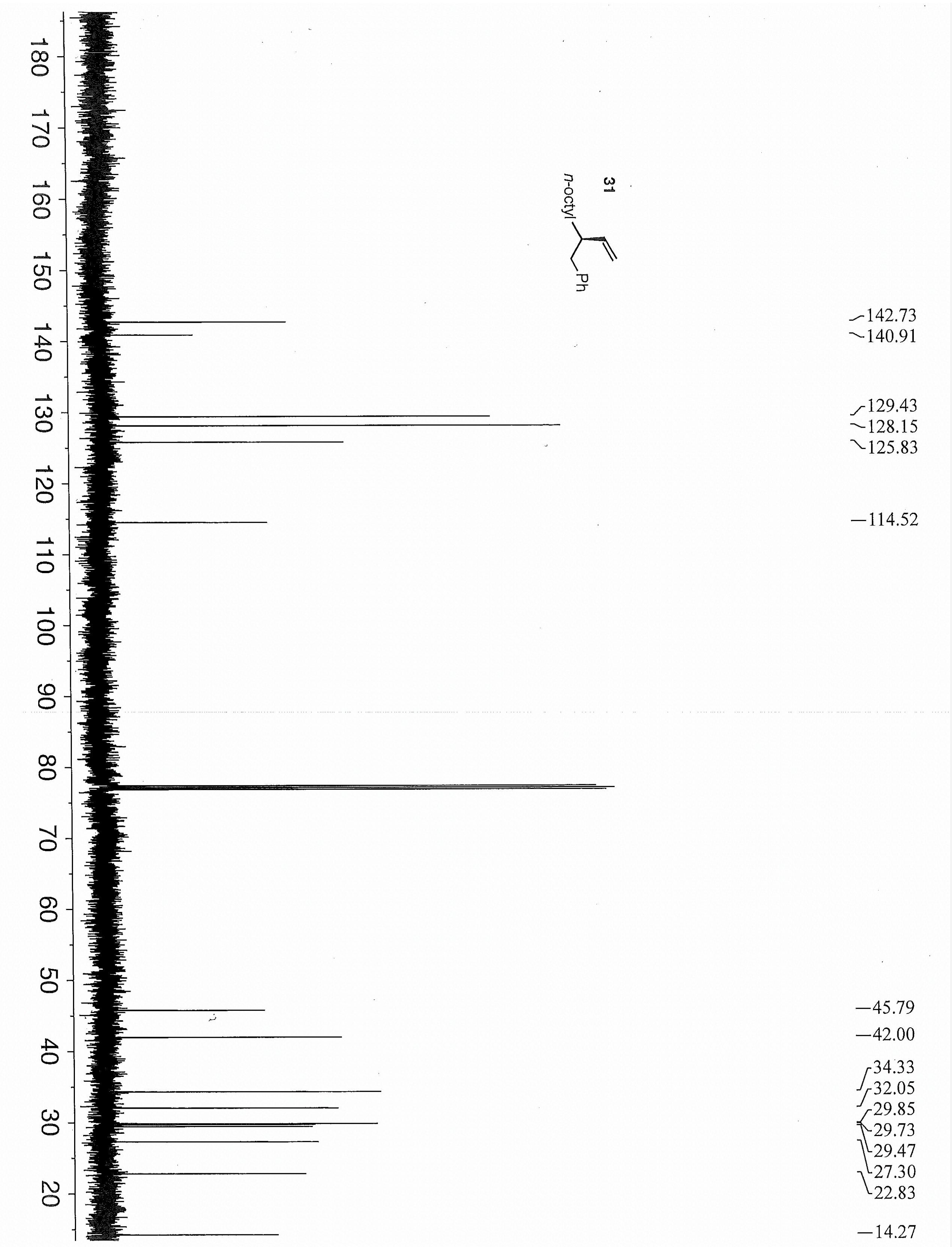
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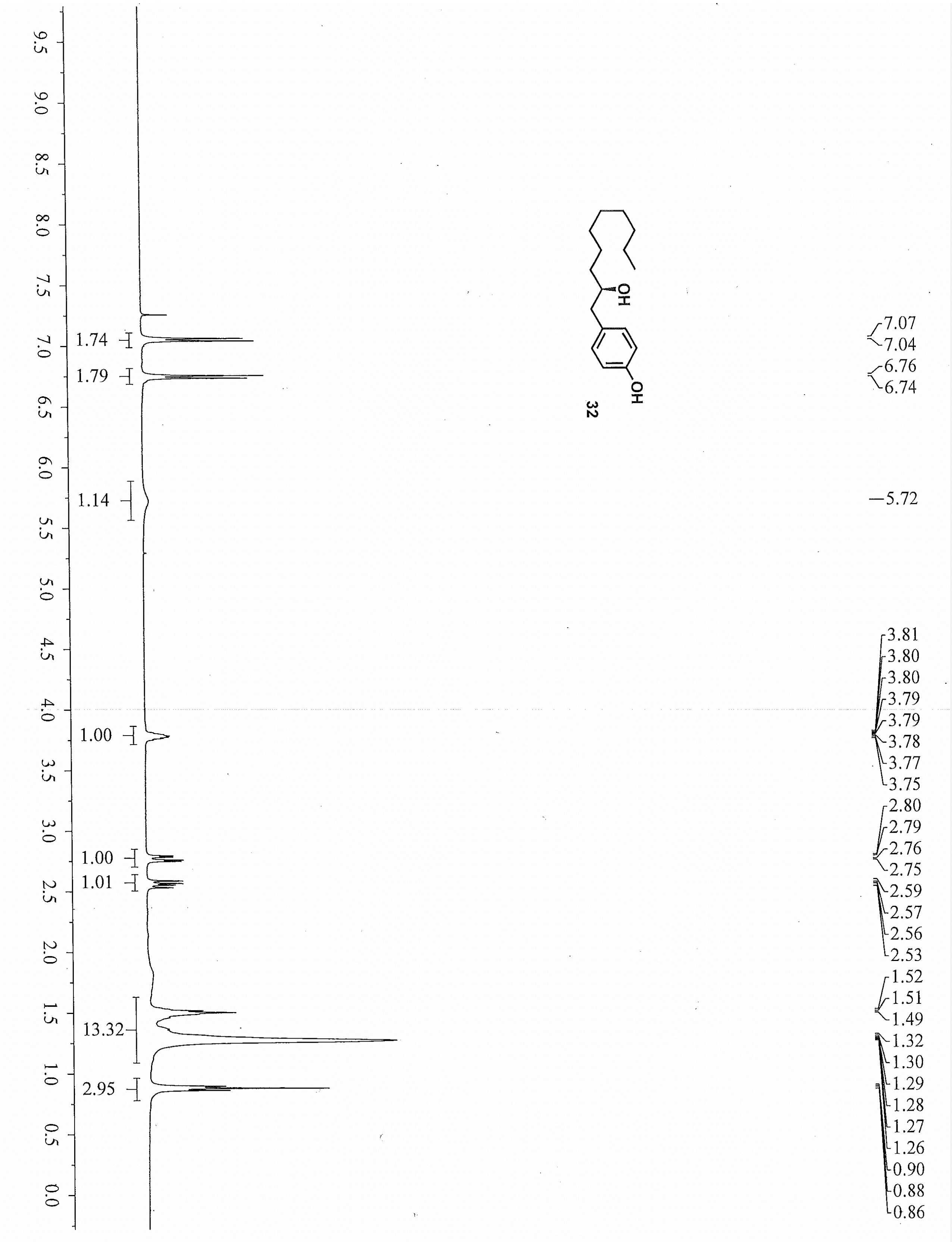

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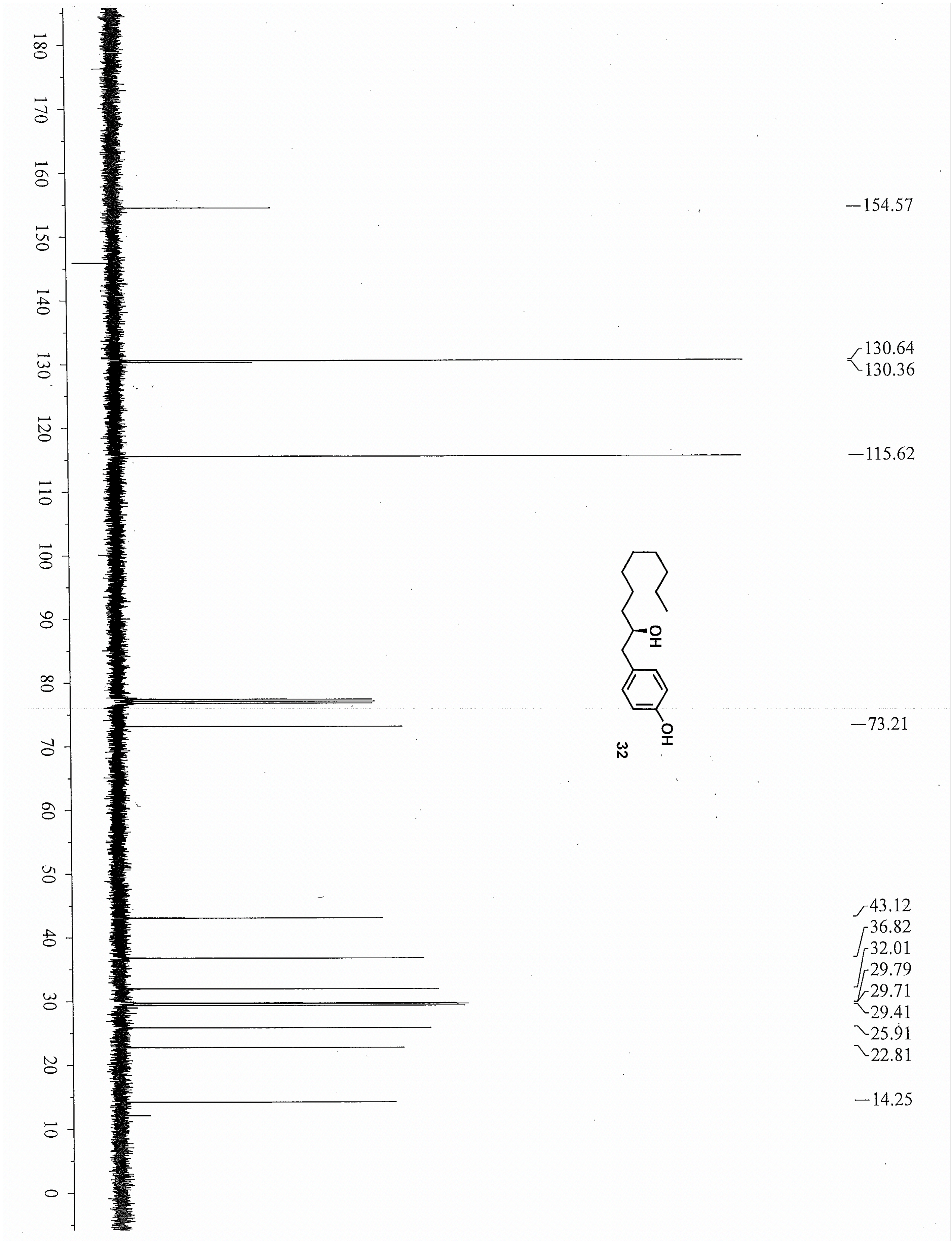
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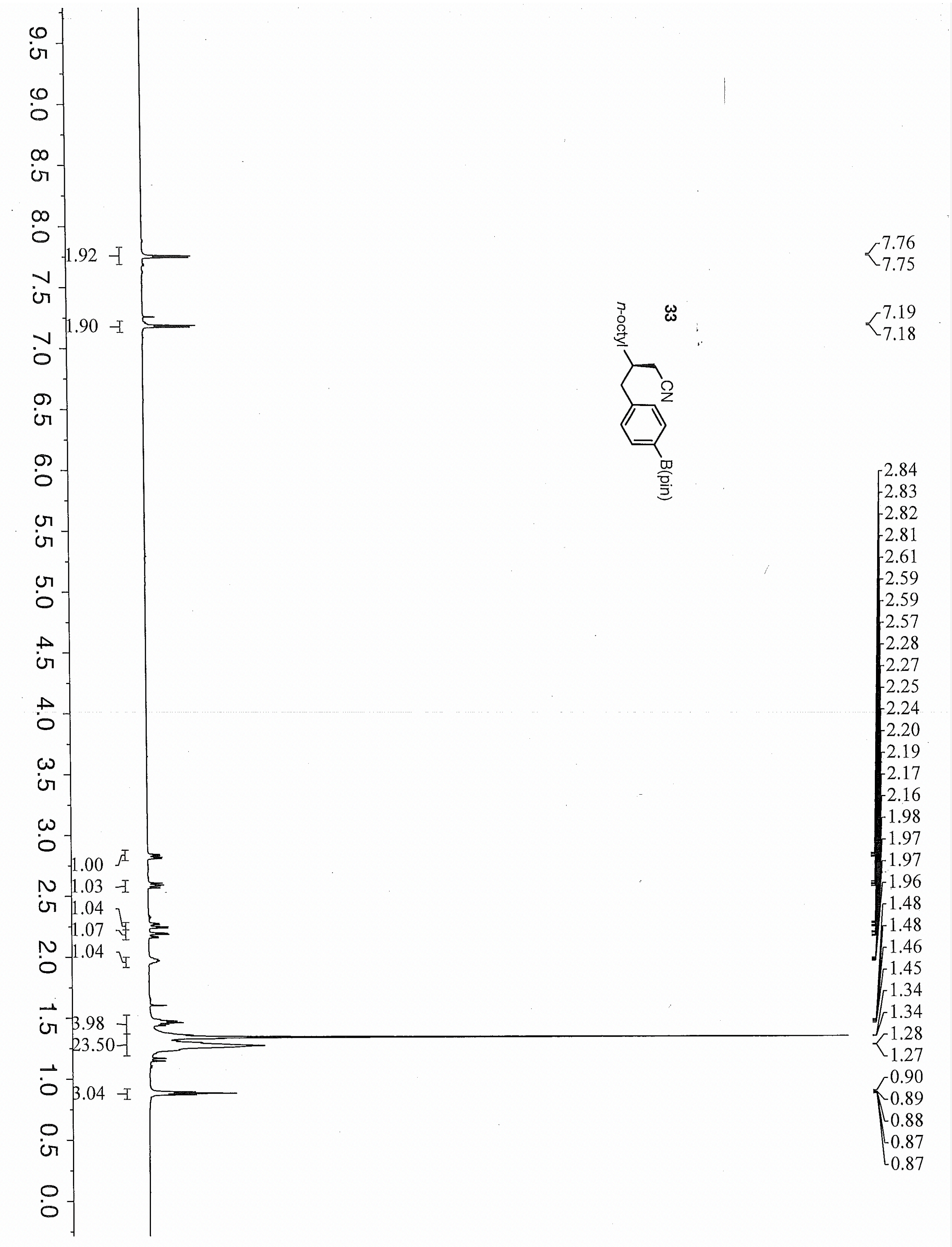
32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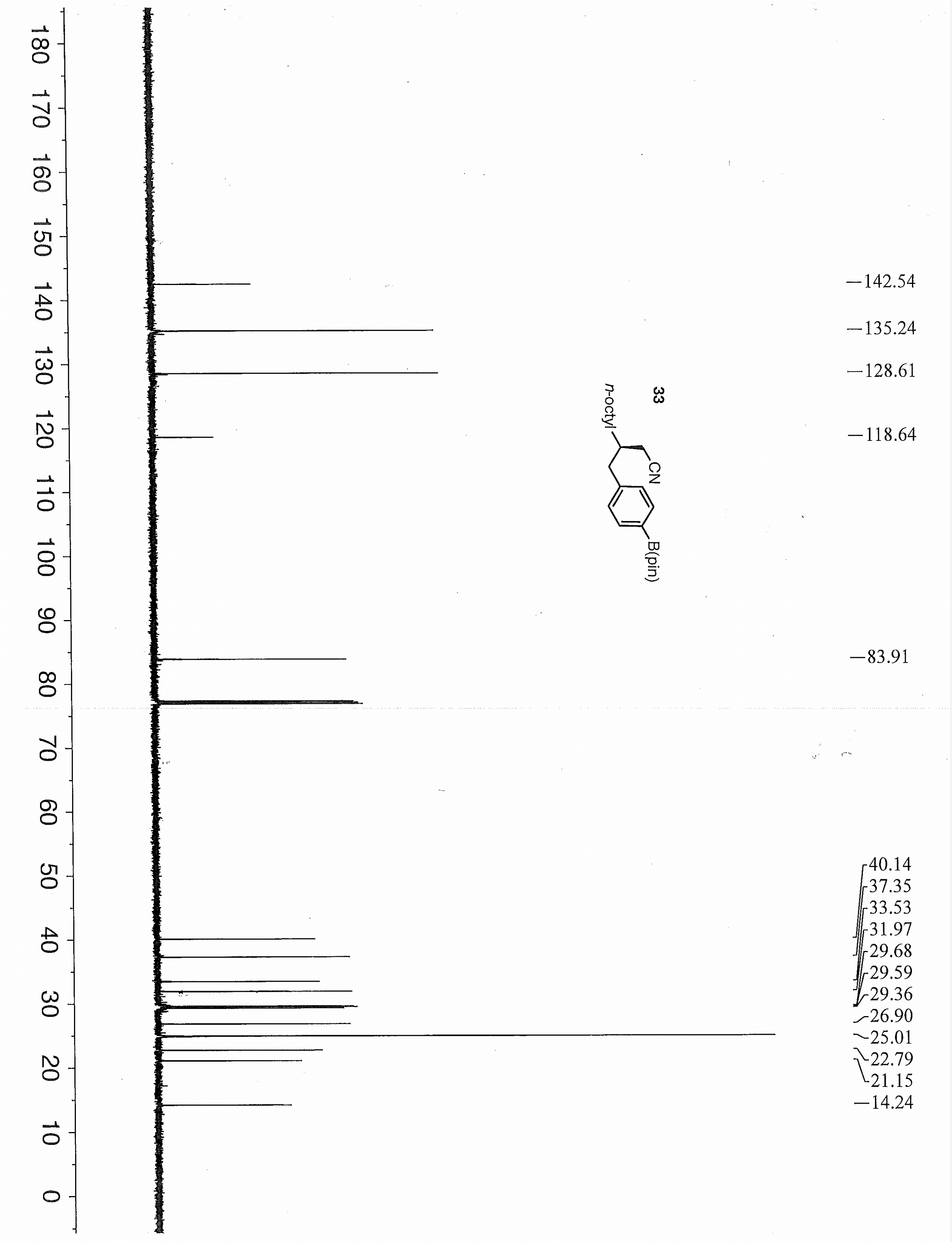

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90
80
70
60
50
40
30
20
10
0
n-octyl
CN
B(pin)
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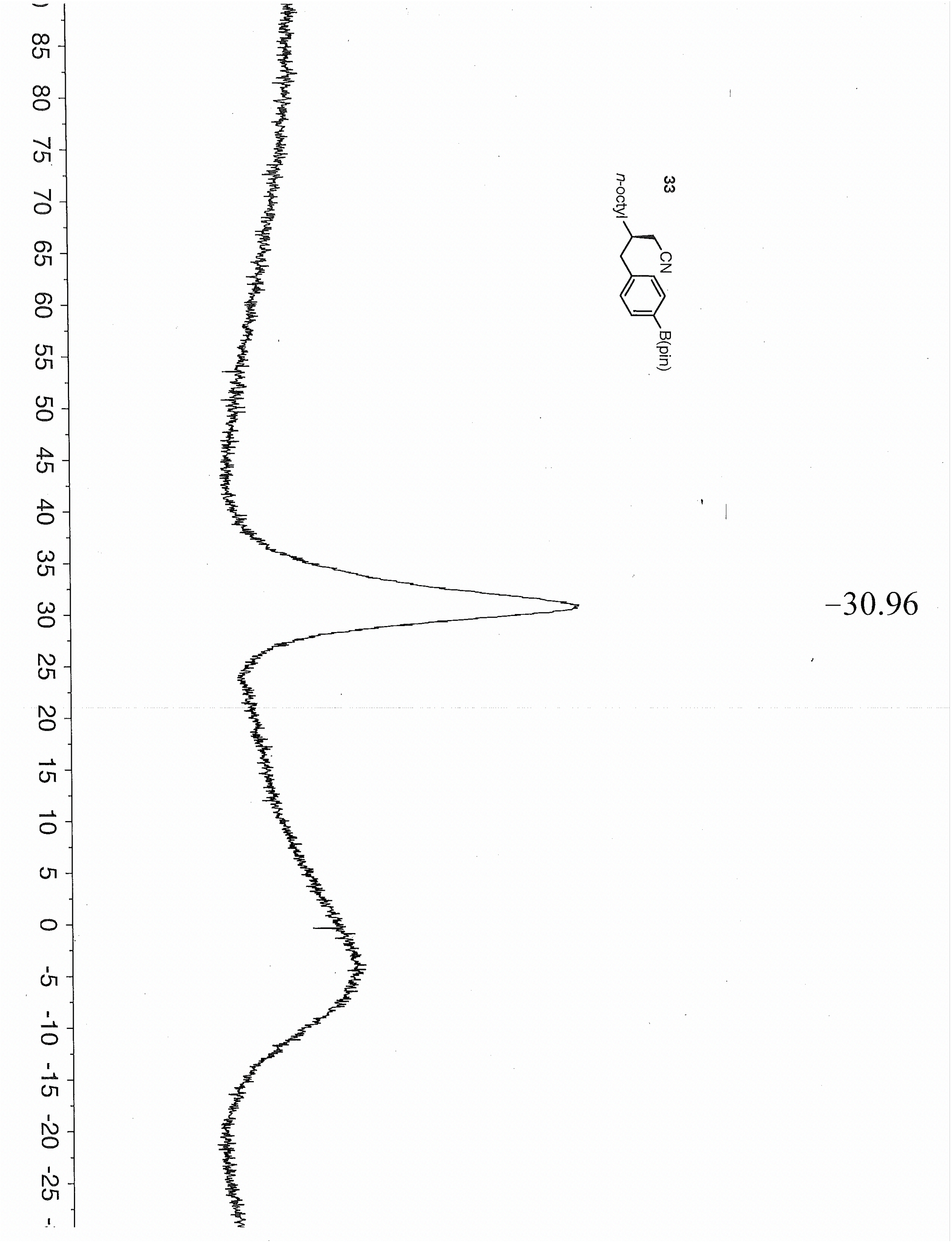
135.24
128.61
118.64
83.91
40.14
37.35
33.53
31.97
29.68
29.59
29.36
26.90
25.01
22.79
21.15
14.24

33

^{11}B NMR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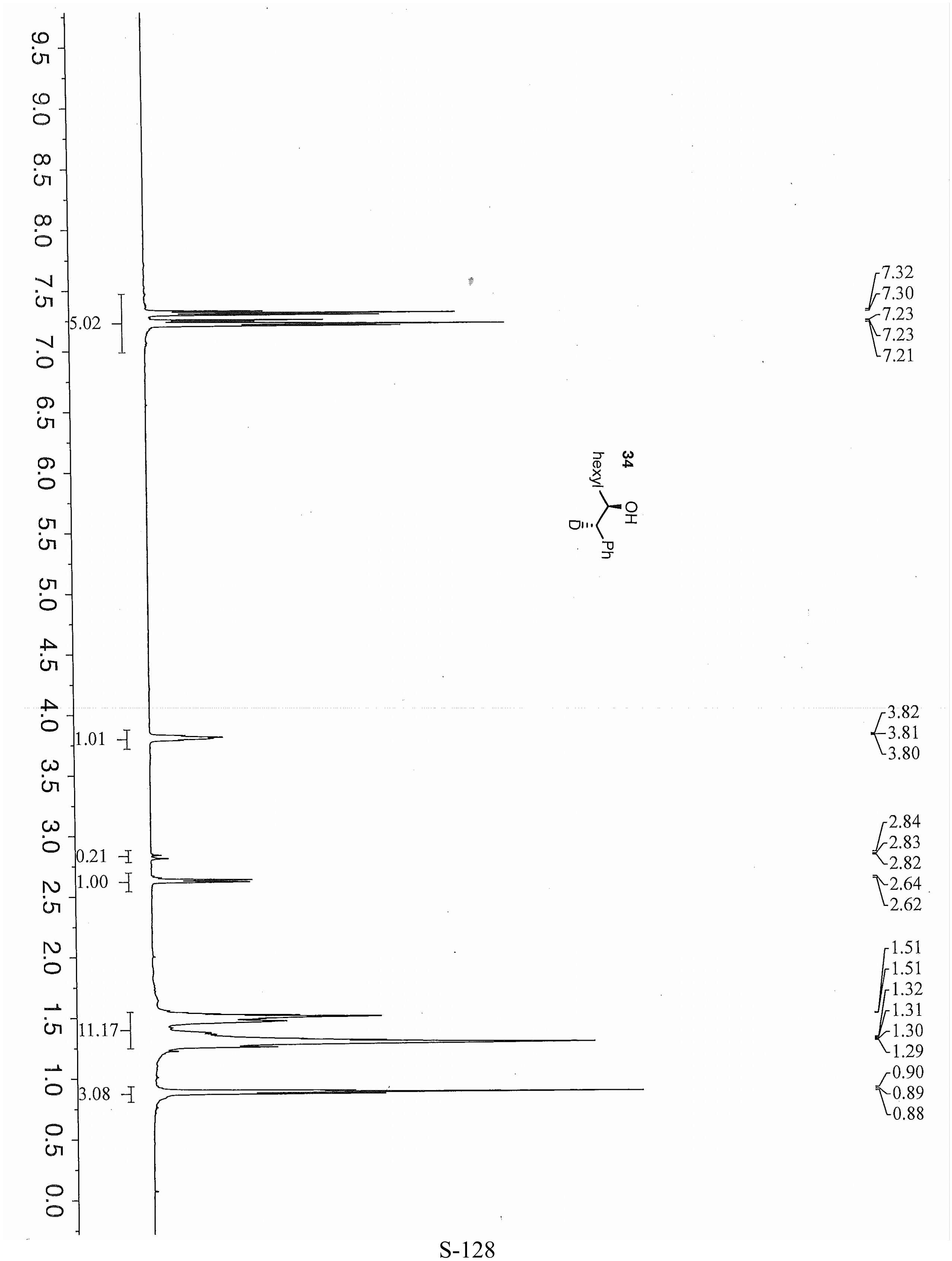
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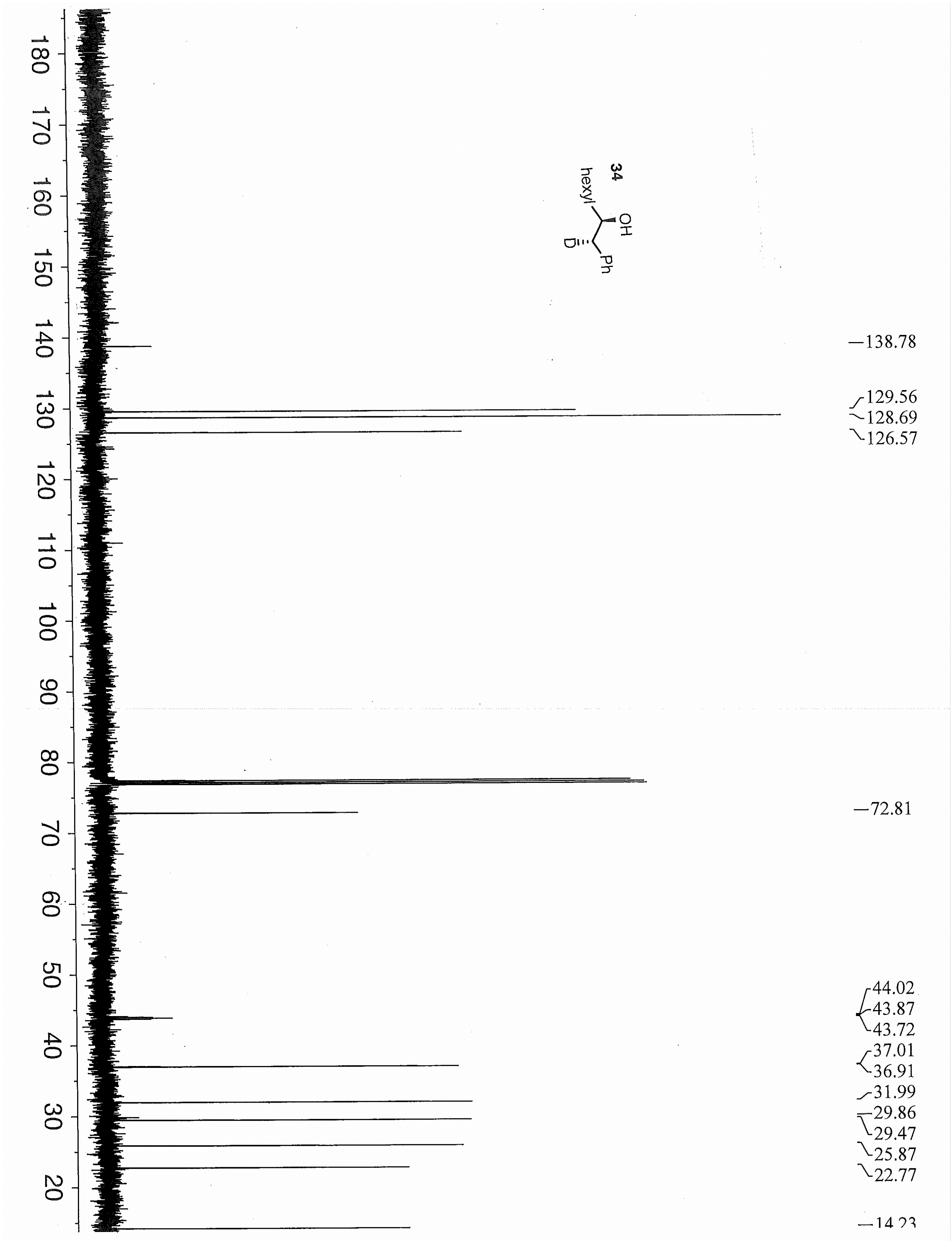
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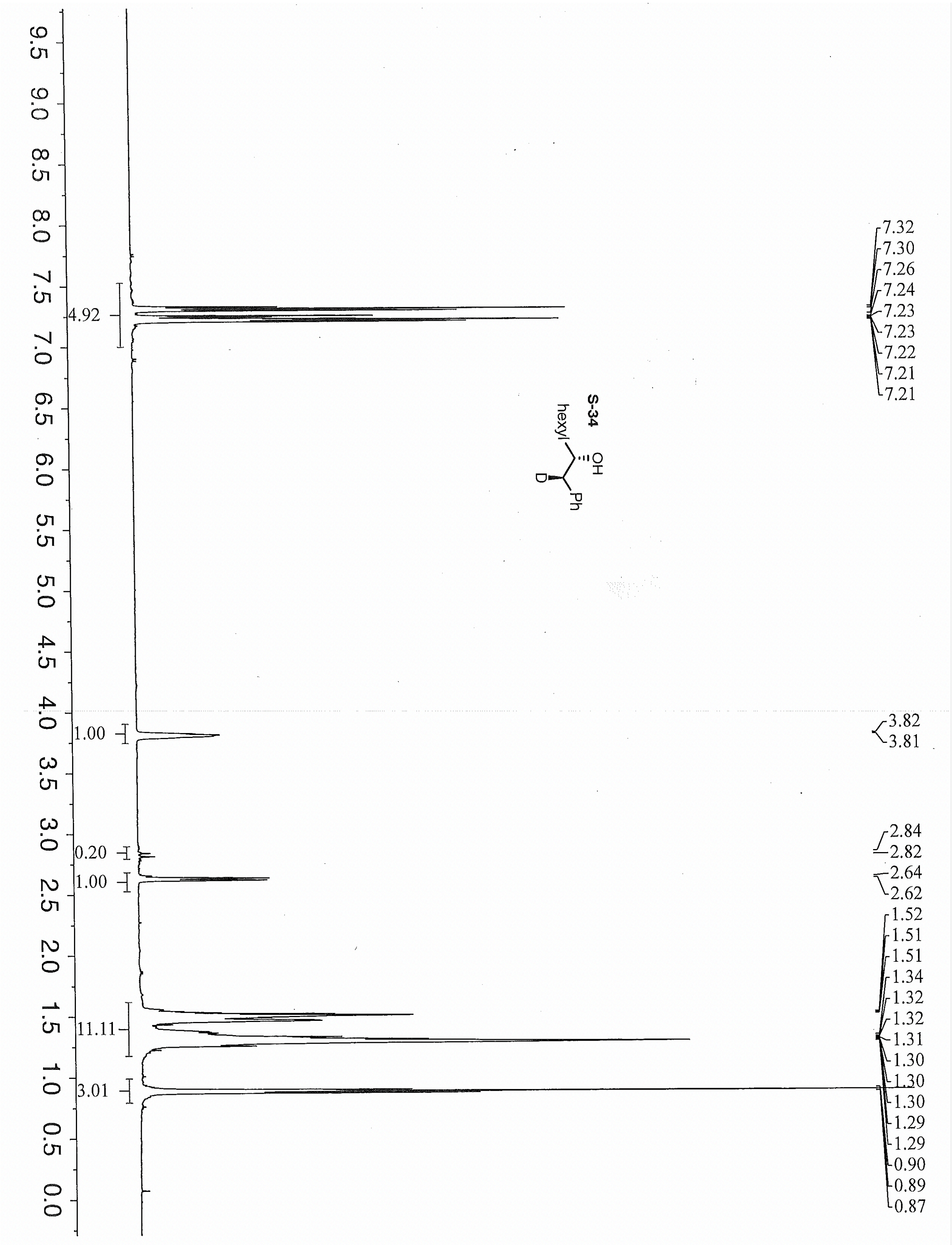
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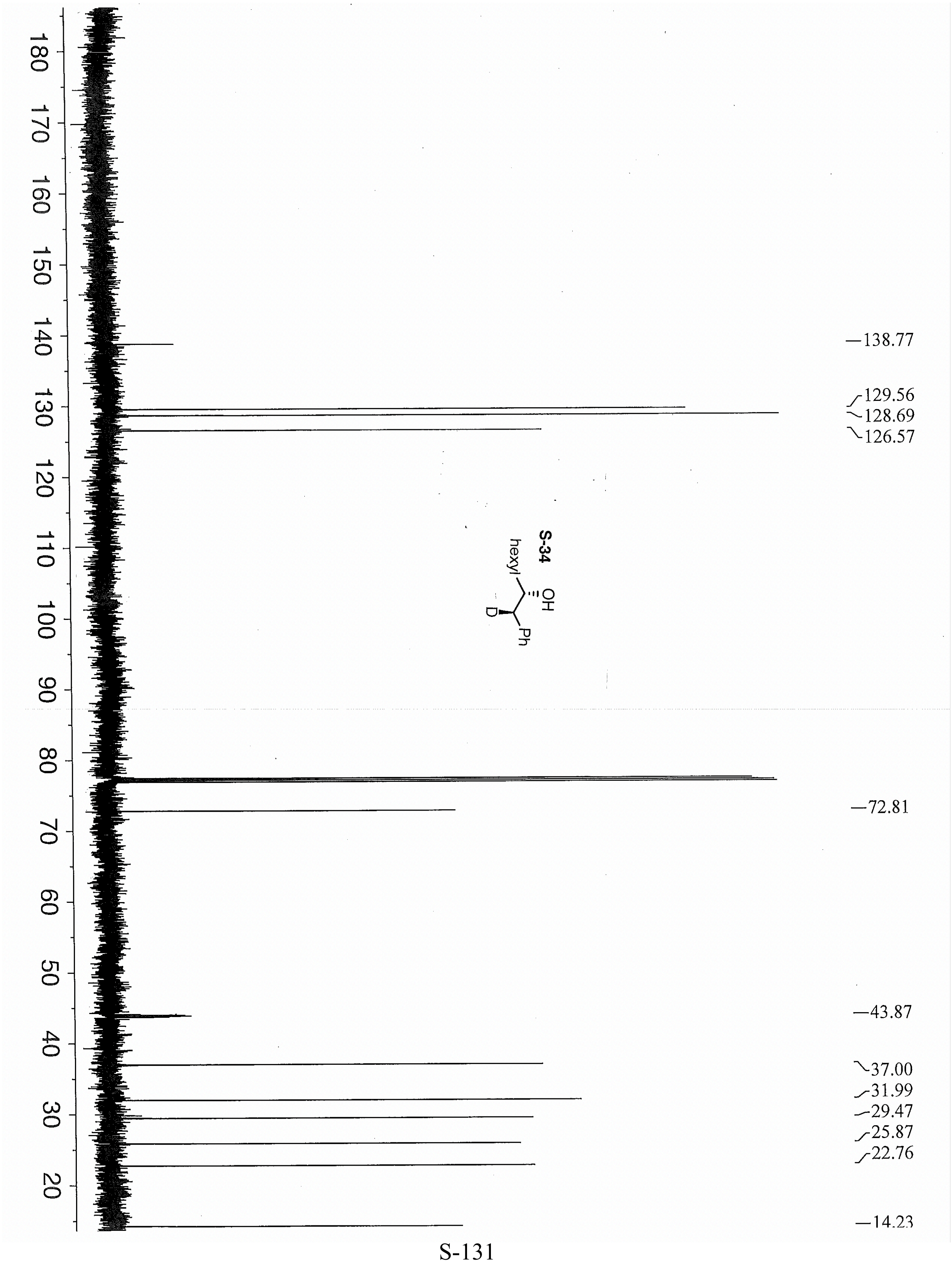
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Ph
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Crystallographic Data

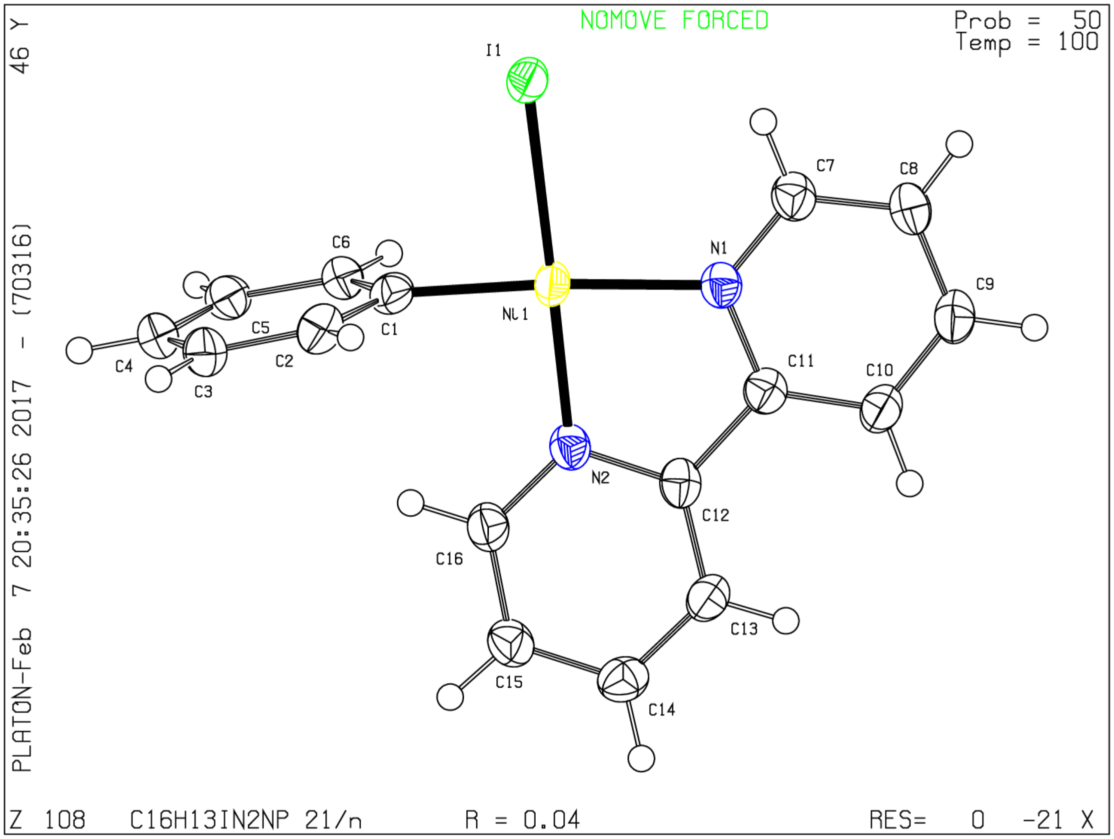


Table 1. Crystal data and structure refinement for C16h13IN2Ni.

Identification code	C16H13IN2Ni	
Empirical formula	C16 H13 I N2 Ni	
Formula weight	418.89	
Temperature	100(2) K	
Wavelength	1.54178 ≈	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 8.9583(9) ≈	α= 90∞.

	b = 16.2595(16) ≈	β= 107.788(3)∞.
	c = 10.4132(11) ≈	γ = 90∞.
Volume	1444.2(3) ≈^{3}	
Z	4	
Density (Calculated)	1.927 Mg/m^3	
Absorption coefficient	18.626 mm^{-1}	
F(000)	816	
Crystal size	0.410 x 0.220 x 0.140 mm^3	
Theta range for data collection	5.225 to 66.860∞.	
Index ranges	-10<=h<=10, -19<=k<=19, 0<=l<=12	
Reflections collected	2544	
Independent reflections	2544 [R(int) = 0.0732]	
Completeness to theta = 66.860∞	99.1 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.4660 and 0.1797	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2544 / 0 / 181	
Goodness-of-fit on F^2	1.100	
Final R indices [I>2sigma(I)]	R1 = 0.0409, wR2 = 0.1099	
R indices (all data)	R1 = 0.0411, wR2 = 0.1102	
Extinction coefficient	n/a	
Largest diff. peak and hole	1.123 and -1.325 e.≈$^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (≈^{2}x 10^3) for C16h13IN2Ni. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
I(1)	2706(1)	3600(1)	8208(1)	31(1)
Ni(1)	3008(1)	3743(1)	5916(1)	26(1)
N(1)	2306(4)	4899(2)	5566(3)	27(1)
N(2)	3570(4)	3887(2)	4279(3)	27(1)
C(1)	3468(5)	2608(3)	5986(4)	29(1)
C(2)	4982(5)	2279(3)	6452(4)	34(1)
C(3)	5260(7)	1451(3)	6270(6)	38(1)
C(4)	4020(6)	927(3)	5664(5)	38(1)
C(5)	2506(6)	1246(3)	5230(5)	33(1)
C(6)	2240(5)	2070(3)	5377(4)	30(1)
C(7)	1605(5)	5376(3)	6261(4)	31(1)
C(8)	1130(5)	6172(3)	5886(5)	32(1)
C(9)	1354(6)	6488(3)	4731(5)	34(1)
C(10)	2046(5)	6002(3)	3990(4)	31(1)
C(11)	2534(5)	5217(2)	4440(4)	28(1)
C(12)	3327(4)	4659(2)	3752(4)	27(1)
C(13)	3833(5)	4890(3)	2679(4)	31(1)
C(14)	4578(5)	4330(3)	2094(4)	33(1)
C(15)	4798(6)	3540(3)	2605(5)	33(1)
C(16)	4286(5)	3346(3)	3693(4)	32(1)

Table 3. Bond lengths [≈] and angles [∞] for C16h13IN2Ni.

I(1)-Ni(1)	2.4926(8)
Ni(1)-C(1)	1.887(4)
Ni(1)-N(2)	1.934(4)
Ni(1)-N(1)	1.981(3)
N(1)-C(7)	1.341(5)
N(1)-C(11)	1.352(5)
N(2)-C(16)	1.340(6)
N(2)-C(12)	1.360(5)
C(1)-C(6)	1.398(6)
C(1)-C(2)	1.399(6)
C(2)-C(3)	1.391(6)
C(2)-H(2)	0.9500
C(3)-C(4)	1.389(8)
C(3)-H(3)	0.9500
C(4)-C(5)	1.392(7)
C(4)-H(4)	0.9500
C(5)-C(6)	1.377(6)
C(5)-H(5)	0.9500
C(6)-H(6)	0.9500
C(7)-C(8)	1.381(6)
C(7)-H(7)	0.9500
C(8)-C(9)	1.378(7)
C(8)-H(8)	0.9500
C(9)-C(10)	1.376(6)
C(9)-H(9)	0.9500
C(10)-C(11)	1.383(6)
C(10)-H(10)	0.9500
C(11)-C(12)	1.468(6)
C(12)-C(13)	1.379(6)
C(13)-C(14)	1.376(6)
C(13)-H(13)	0.9500
C(14)-C(15)	1.382(6)

C(14)-H(14)	0.9500
C(15)-C(16)	1.382(7)
C(15)-H(15)	0.9500
C(16)-H(16)	0.9500
C(1)-Ni(1)-N(2)	92.30(16)
C(1)-Ni(1)-N(1)	171.31(16)
N(2)-Ni(1)-N(1)	82.84(14)
C(1)-Ni(1)-I(1)	87.61(12)
N(2)-Ni(1)-I(1)	171.36(11)
N(1)-Ni(1)-I(1)	98.28(10)
C(7)-N(1)-C(11)	117.8(4)
C(7)-N(1)-Ni(1)	128.6(3)
C(11)-N(1)-Ni(1)	113.6(3)
C(16)-N(2)-C(12)	117.5(4)
C(16)-N(2)-Ni(1)	127.7(3)
C(12)-N(2)-Ni(1)	114.5(3)
C(6)-C(1)-C(2)	117.6(4)
C(6)-C(1)-Ni(1)	117.5(3)
C(2)-C(1)-Ni(1)	124.4(3)
C(3)-C(2)-C(1)	121.1(4)
C(3)-C(2)-H(2)	119.4
C(1)-C(2)-H(2)	119.4
C(4)-C(3)-C(2)	120.2(5)
C(4)-C(3)-H(3)	119.9
C(2)-C(3)-H(3)	119.9
C(3)-C(4)-C(5)	118.9(4)
C(3)-C(4)-H(4)	120.5
C(5)-C(4)-H(4)	120.5
C(6)-C(5)-C(4)	120.7(5)
C(6)-C(5)-H(5)	119.7
C(4)-C(5)-H(5)	119.7
C(5)-C(6)-C(1)	121.3(4)
C(5)-C(6)-H(6)	119.3

C(1)-C(6)-H(6)	119.3
N(1)-C(7)-C(8)	122.9(4)
N(1)-C(7)-H(7)	118.5
C(8)-C(7)-H(7)	118.5
C(9)-C(8)-C(7)	118.8(4)
C(9)-C(8)-H(8)	120.6
C(7)-C(8)-H(8)	120.6
C(10)-C(9)-C(8)	119.1(4)
C(10)-C(9)-H(9)	120.5
C(8)-C(9)-H(9)	120.5
C(9)-C(10)-C(11)	119.3(4)
C(9)-C(10)-H(10)	120.3
C(11)-C(10)-H(10)	120.3
N(1)-C(11)-C(10)	122.1(4)
N(1)-C(11)-C(12)	114.2(4)
C(10)-C(11)-C(12)	123.7(4)
N(2)-C(12)-C(13)	121.7(4)
N(2)-C(12)-C(11)	114.5(4)
C(13)-C(12)-C(11)	123.7(4)
C(14)-C(13)-C(12)	120.2(4)
C(14)-C(13)-H(13)	119.9
C(12)-C(13)-H(13)	119.9
C(13)-C(14)-C(15)	118.4(4)
C(13)-C(14)-H(14)	120.8
C(15)-C(14)-H(14)	120.8
C(14)-C(15)-C(16)	118.9(4)
C(14)-C(15)-H(15)	120.5
C(16)-C(15)-H(15)	120.5
N(2)-C(16)-C(15)	123.2(4)
N(2)-C(16)-H(16)	118.4
C(15)-C(16)-H(16)	118.4

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\approx^2$x 10^3) for C16h13IN2Ni. The anisotropic displacement factor exponent takes the form: $-2\pi^2$[h^2 $a^{*2}U^{11}$ + ... + 2 h k a* b* U^{12}]

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
I(1)	43(1)	24(1)	26(1)	2(1)	11(1)	2(1)
Ni(1)	33(1)	18(1)	26(1)	1(1)	8(1)	2(1)
N(1)	28(2)	24(2)	27(2)	-1(1)	7(1)	0(1)
N(2)	30(2)	23(2)	26(2)	-2(1)	6(1)	1(1)
C(1)	38(2)	27(2)	23(2)	2(2)	11(2)	0(2)
C(2)	36(2)	31(2)	33(2)	5(2)	8(2)	0(2)
C(3)	43(3)	34(3)	39(3)	8(2)	17(2)	14(2)
C(4)	57(3)	25(2)	41(2)	1(2)	27(2)	7(2)
C(5)	48(3)	26(2)	31(2)	-5(2)	18(2)	-5(2)
C(6)	37(2)	24(2)	32(2)	-1(2)	16(2)	1(2)
C(7)	35(2)	27(2)	28(2)	-3(2)	6(2)	-1(2)
C(8)	35(2)	24(2)	36(2)	-5(2)	9(2)	1(2)
C(9)	40(2)	23(2)	37(3)	1(2)	9(2)	2(2)
C(10)	39(2)	25(2)	29(2)	4(2)	9(2)	-1(2)
C(11)	29(2)	26(2)	26(2)	-2(2)	6(2)	-2(2)
C(12)	27(2)	23(2)	27(2)	-4(2)	2(2)	-3(2)
C(13)	37(2)	26(2)	29(2)	4(2)	9(2)	-2(2)
C(14)	38(2)	35(2)	26(2)	-2(2)	9(2)	-5(2)
C(15)	39(2)	30(2)	29(2)	-5(2)	10(2)	1(2)
C(16)	39(2)	25(2)	31(2)	-1(2)	10(2)	3(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (≈^{2}x 10 ^{3})
for C16h13IN2Ni.

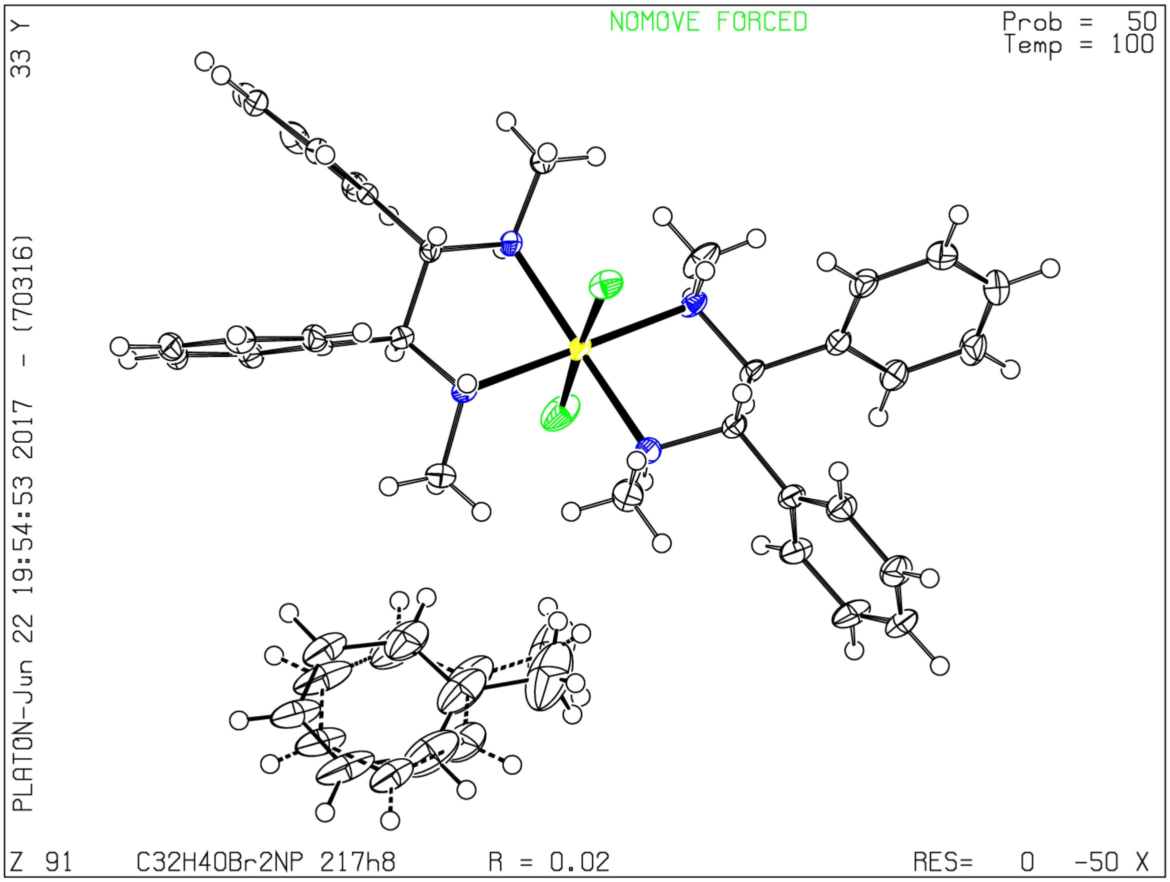
	x	y	z	U(eq)
H(2)	5835	2626	6900	40
H(3)	6301	1244	6561	45
H(4)	4202	360	5549	45
H(5)	1647	891	4829	40
H(6)	1199	2276	5057	36
H(7)	1428	5156	7047	37
H(8)	656	6496	6415	39
H(9)	1036	7033	4449	40
H(10)	2186	6203	3179	37
H(13)	3666	5436	2344	37
H(14)	4932	4484	1356	40
H(15)	5293	3137	2215	40
H(16)	4448	2802	4042	38

Table 6. Torsion angles [∞] for C16h13IN2Ni.

N(2)-Ni(1)-C(1)-C(6)	-92.5(3)
I(1)-Ni(1)-C(1)-C(6)	96.2(3)
N(2)-Ni(1)-C(1)-C(2)	79.1(4)
I(1)-Ni(1)-C(1)-C(2)	-92.3(3)
C(6)-C(1)-C(2)-C(3)	2.3(6)
Ni(1)-C(1)-C(2)-C(3)	-169.2(4)
C(1)-C(2)-C(3)-C(4)	-2.5(7)
C(2)-C(3)-C(4)-C(5)	0.8(7)
C(3)-C(4)-C(5)-C(6)	1.1(7)
C(4)-C(5)-C(6)-C(1)	-1.3(7)
C(2)-C(1)-C(6)-C(5)	-0.4(6)
Ni(1)-C(1)-C(6)-C(5)	171.7(3)
C(11)-N(1)-C(7)-C(8)	-0.8(6)
Ni(1)-N(1)-C(7)-C(8)	-178.0(3)
N(1)-C(7)-C(8)-C(9)	1.4(7)
C(7)-C(8)-C(9)-C(10)	-0.1(7)
C(8)-C(9)-C(10)-C(11)	-1.6(7)
C(7)-N(1)-C(11)-C(10)	-1.1(6)
Ni(1)-N(1)-C(11)-C(10)	176.6(3)
C(7)-N(1)-C(11)-C(12)	179.2(3)
Ni(1)-N(1)-C(11)-C(12)	-3.1(4)
C(9)-C(10)-C(11)-N(1)	2.3(6)
C(9)-C(10)-C(11)-C(12)	-178.0(4)
C(16)-N(2)-C(12)-C(13)	-1.8(6)
Ni(1)-N(2)-C(12)-C(13)	172.4(3)
C(16)-N(2)-C(12)-C(11)	179.8(4)
Ni(1)-N(2)-C(12)-C(11)	-6.0(4)
N(1)-C(11)-C(12)-N(2)	6.0(5)
C(10)-C(11)-C(12)-N(2)	-173.7(4)
N(1)-C(11)-C(12)-C(13)	-172.4(4)
C(10)-C(11)-C(12)-C(13)	7.9(6)
N(2)-C(12)-C(13)-C(14)	1.3(6)

C(11)-C(12)-C(13)-C(14)	179.5(4)
C(12)-C(13)-C(14)-C(15)	0.1(6)
C(13)-C(14)-C(15)-C(16)	-1.0(7)
C(12)-N(2)-C(16)-C(15)	0.9(6)
Ni(1)-N(2)-C(16)-C(15)	-172.4(3)
C(14)-C(15)-C(16)-N(2)	0.5(7)

Symmetry transformations used to generate equivalent atoms:



Crystal was obtained by vapor diffusion with pentane from a 1:1 metal to ligand solution in toluene. In an argon filled glove box Nickel(II) bromide 2-methoxyethyl ether complex (17.6 mg 0.05 mmol) and (1*S*,2*S*)-*N*,*N'*-Dimethyl-1,2-diphenyl-1,2-ethylenediamine (12.0 mg, 0.05 mmol) were placed in a 2-dram vial with 3 mL of toluene. After stirring at r.t. for 2 hours, the solution was filtered through an acro-disk syringe filter in a 1-dram vial which was then placed in scintillation vial filled with 2 mL of dry pentane. The vial was capped and kept at room temperature. Blue crystals were observable after 4 days.

Table 1. Crystal data and structure refinement for c32H40Br2N4Ni(C7H8).

Identification code	C32H40Br2N4Ni(C7H8)	
Empirical formula	C39 H48 Br2 N4 Ni	
Formula weight	791.34	
Temperature	100(2) K	
Wavelength	0.71073 ≈	
Crystal system	Monoclinic	
Space group	$P2_1$	
Unit cell dimensions	a = 8.9973(10) ≈	α= 90∞.
	b = 9.9788(11) ≈	β= 92.7026(16)∞.
	c = 20.810(2) ≈	γ = 90∞.
Volume	1866.3(4) ≈^{3}	
Z	2	
Density (calculated)	1.408 Mg/m^3	
Absorption coefficient	2.693 mm^{-1}	
F(000)	816	
Crystal size	0.380 x 0.350 x 0.260 mm^3	
Theta range for data collection	1.959 to 28.285∞.	
Index ranges	-11<=h<=11, -13<=k<=13, -27<=l<=27	
Reflections collected	30407	
Independent reflections	9256 [R(int) = 0.0241]	
Completeness to theta = 25.242∞	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7457 and 0.6609	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9256 / 110 / 451	
Goodness-of-fit on F^2	1.025	
Final R indices [I>2sigma(I)]	R1 = 0.0242, wR2 = 0.0548	
R indices (all data)	R1 = 0.0279, wR2 = 0.0562	
Absolute structure parameter	-0.006(2)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.500 and -0.330 e.≈$^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (≈^{2}x 10^3) for c32H40Br2N4Ni(C7H8). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	2855(1)	7770(1)	3215(1)	25(1)
Br(2)	1887(1)	3415(1)	1916(1)	38(1)
Ni(3)	2370(1)	5555(1)	2591(1)	18(1)
N(1)	1220(3)	6696(3)	1868(1)	20(1)
N(2)	4240(3)	5991(3)	2029(1)	20(1)
N(3)	3507(3)	4406(3)	3322(1)	26(1)
N(4)	506(3)	5174(2)	3172(1)	18(1)
C(1)	2213(3)	6917(3)	1328(1)	17(1)
C(2)	3818(3)	7126(3)	1591(1)	18(1)
C(3)	1702(3)	8061(3)	894(1)	19(1)
C(4)	1589(3)	7870(3)	229(1)	24(1)
C(5)	1082(4)	8902(3)	-173(2)	31(1)
C(6)	707(4)	10122(4)	82(2)	33(1)
C(7)	845(4)	10335(3)	741(2)	29(1)
C(8)	1349(3)	9302(3)	1139(1)	25(1)
C(9)	4871(3)	7300(3)	1048(1)	20(1)
C(10)	5684(3)	8472(3)	997(2)	26(1)
C(11)	6619(4)	8655(3)	490(2)	32(1)
C(12)	6734(4)	7678(4)	30(2)	35(1)
C(13)	5944(4)	6495(4)	82(2)	34(1)
C(14)	5021(3)	6302(3)	592(2)	26(1)
C(15)	5717(3)	6258(4)	2342(2)	30(1)
C(16)	-266(3)	6266(4)	1614(2)	35(1)
C(17)	2389(3)	3721(3)	3710(1)	19(1)
C(18)	1085(3)	4668(3)	3807(1)	16(1)
C(19)	3022(3)	3227(3)	4359(1)	17(1)
C(20)	2754(3)	1932(3)	4565(1)	20(1)
C(21)	3215(3)	1522(3)	5182(1)	23(1)
C(22)	3971(3)	2398(3)	5589(1)	24(1)

C(23)	4281(3)	3691(3)	5386(1)	23(1)
C(24)	3796(3)	4103(3)	4774(1)	21(1)
C(25)	-69(3)	4000(3)	4205(1)	16(1)
C(26)	-1039(3)	3034(3)	3943(1)	22(1)
C(27)	-2046(3)	2387(3)	4324(2)	26(1)
C(28)	-2079(3)	2707(3)	4976(2)	27(1)
C(29)	-1121(3)	3636(3)	5240(1)	25(1)
C(30)	-128(3)	4301(3)	4857(1)	20(1)
C(31)	4656(5)	3416(5)	3155(2)	60(1)
C(32)	-612(3)	6235(3)	3279(1)	25(1)
C(1S)	7142(12)	1400(40)	2086(18)	55(2)
C(2S)	6025(14)	747(16)	2409(8)	48(2)
C(3S)	6311(10)	-128(7)	2910(3)	41(1)
C(4S)	7779(9)	-402(7)	3093(4)	48(2)
C(5S)	8944(9)	166(9)	2791(4)	56(2)
C(6S)	8554(11)	1091(11)	2278(5)	65(2)
C(7S)	6740(20)	2360(20)	1561(10)	97(5)
C(1T)	6820(20)	1450(80)	2030(30)	55(2)
C(2T)	6080(30)	590(40)	2435(17)	65(2)
C(3T)	6882(18)	-201(18)	2896(9)	56(2)
C(4T)	8391(17)	-113(14)	2944(7)	48(2)
C(5T)	9146(14)	746(14)	2540(7)	41(1)
C(6T)	8326(17)	1507(16)	2097(7)	48(2)
C(7T)	6080(40)	2270(40)	1502(19)	97(5)

Table 3. Bond lengths [≈] and angles [∞] for c32H40Br2N4Ni(C7H8).

Br(1)-Ni(3)	2.5908(5)
Br(2)-Ni(3)	2.5810(6)
Ni(3)-N(1)	2.116(2)
Ni(3)-N(3)	2.128(3)
Ni(3)-N(2)	2.138(2)
Ni(3)-N(4)	2.148(2)
N(1)-C(16)	1.478(4)
N(1)-C(1)	1.485(3)
N(1)-H(1N)	0.87(2)
N(2)-C(15)	1.477(4)
N(2)-C(2)	1.491(4)
N(2)-H(2N)	0.86(2)
N(3)-C(31)	1.483(5)
N(3)-C(17)	1.486(4)
N(3)-H(3N)	0.86(2)
N(4)-C(32)	1.484(4)
N(4)-C(18)	1.484(3)
N(4)-H(4N)	0.85(2)
C(1)-C(3)	1.514(4)
C(1)-C(2)	1.534(4)
C(1)-H(1)	1.0000
C(2)-C(9)	1.519(4)
C(2)-H(2)	1.0000
C(3)-C(8)	1.382(4)
C(3)-C(4)	1.395(4)
C(4)-C(5)	1.390(4)
C(4)-H(4)	0.9500
C(5)-C(6)	1.377(5)
C(5)-H(5)	0.9500
C(6)-C(7)	1.387(4)
C(6)-H(6)	0.9500
C(7)-C(8)	1.385(4)

C(7)-H(7)	0.9500
C(8)-H(8)	0.9500
C(9)-C(10)	1.386(4)
C(9)-C(14)	1.386(4)
C(10)-C(11)	1.392(4)
C(10)-H(10)	0.9500
C(11)-C(12)	1.375(5)
C(11)-H(11)	0.9500
C(12)-C(13)	1.385(5)
C(12)-H(12)	0.9500
C(13)-C(14)	1.391(4)
C(13)-H(13)	0.9500
C(14)-H(14)	0.9500
C(15)-H(15A)	0.9800
C(15)-H(15B)	0.9800
C(15)-H(15C)	0.9800
C(16)-H(16A)	0.9800
C(16)-H(16B)	0.9800
C(16)-H(16C)	0.9800
C(17)-C(19)	1.523(4)
C(17)-C(18)	1.528(4)
C(17)-H(17)	1.0000
C(18)-C(25)	1.513(4)
C(18)-H(18)	1.0000
C(19)-C(20)	1.387(4)
C(19)-C(24)	1.392(4)
C(20)-C(21)	1.392(4)
C(20)-H(20)	0.9500
C(21)-C(22)	1.374(4)
C(21)-H(21)	0.9500
C(22)-C(23)	1.390(4)
C(22)-H(22)	0.9500
C(23)-C(24)	1.389(4)
C(23)-H(23)	0.9500

C(24)-H(24)	0.9500
C(25)-C(30)	1.391(4)
C(25)-C(26)	1.395(4)
C(26)-C(27)	1.391(4)
C(26)-H(26)	0.9500
C(27)-C(28)	1.394(4)
C(27)-H(27)	0.9500
C(28)-C(29)	1.364(5)
C(28)-H(28)	0.9500
C(29)-C(30)	1.393(4)
C(29)-H(29)	0.9500
C(30)-H(30)	0.9500
C(31)-H(31A)	0.9800
C(31)-H(31B)	0.9800
C(31)-H(31C)	0.9800
C(32)-H(32A)	0.9800
C(32)-H(32B)	0.9800
C(32)-H(32C)	0.9800
C(1S)-C(6S)	1.348(13)
C(1S)-C(2S)	1.394(11)
C(1S)-C(7S)	1.491(13)
C(2S)-C(3S)	1.375(11)
C(2S)-H(2S)	0.9500
C(3S)-C(4S)	1.384(10)
C(3S)-H(3S)	0.9500
C(4S)-C(5S)	1.369(9)
C(4S)-H(4S)	0.9500
C(5S)-C(6S)	1.442(12)
C(5S)-H(5S)	0.9500
C(6S)-H(6S)	0.9500
C(7S)-H(7S1)	0.9800
C(7S)-H(7S2)	0.9800
C(7S)-H(7S3)	0.9800
C(1T)-C(6T)	1.361(18)

C(1T)-C(2T)	1.394(18)
C(1T)-C(7T)	1.502(18)
C(2T)-C(3T)	1.414(19)
C(2T)-H(2T)	0.9500
C(3T)-C(4T)	1.359(16)
C(3T)-H(3T)	0.9500
C(4T)-C(5T)	1.398(16)
C(4T)-H(4T)	0.9500
C(5T)-C(6T)	1.382(17)
C(5T)-H(5T)	0.9500
C(6T)-H(6T)	0.9500
C(7T)-H(7T1)	0.9800
C(7T)-H(7T2)	0.9800
C(7T)-H(7T3)	0.9800
N(1)-Ni(3)-N(3)	179.47(10)
N(1)-Ni(3)-N(2)	82.69(9)
N(3)-Ni(3)-N(2)	97.84(9)
N(1)-Ni(3)-N(4)	97.30(9)
N(3)-Ni(3)-N(4)	82.17(9)
N(2)-Ni(3)-N(4)	178.16(11)
N(1)-Ni(3)-Br(2)	89.70(7)
N(3)-Ni(3)-Br(2)	90.35(9)
N(2)-Ni(3)-Br(2)	89.30(7)
N(4)-Ni(3)-Br(2)	92.55(7)
N(1)-Ni(3)-Br(1)	87.83(7)
N(3)-Ni(3)-Br(1)	92.12(9)
N(2)-Ni(3)-Br(1)	88.96(7)
N(4)-Ni(3)-Br(1)	89.20(7)
Br(2)-Ni(3)-Br(1)	177.144(18)
C(16)-N(1)-C(1)	109.7(2)
C(16)-N(1)-Ni(3)	120.1(2)
C(1)-N(1)-Ni(3)	109.06(17)
C(16)-N(1)-H(1N)	107(2)

C(1)-N(1)-H(1N)	107(2)
Ni(3)-N(1)-H(1N)	103(2)
C(15)-N(2)-C(2)	109.3(2)
C(15)-N(2)-Ni(3)	120.68(18)
C(2)-N(2)-Ni(3)	107.75(17)
C(15)-N(2)-H(2N)	111(2)
C(2)-N(2)-H(2N)	106(2)
Ni(3)-N(2)-H(2N)	101(2)
C(31)-N(3)-C(17)	108.5(3)
C(31)-N(3)-Ni(3)	120.6(2)
C(17)-N(3)-Ni(3)	108.76(17)
C(31)-N(3)-H(3N)	108(3)
C(17)-N(3)-H(3N)	110(3)
Ni(3)-N(3)-H(3N)	101(3)
C(32)-N(4)-C(18)	108.7(2)
C(32)-N(4)-Ni(3)	120.72(18)
C(18)-N(4)-Ni(3)	108.11(16)
C(32)-N(4)-H(4N)	109(2)
C(18)-N(4)-H(4N)	106(2)
Ni(3)-N(4)-H(4N)	104(2)
N(1)-C(1)-C(3)	112.8(2)
N(1)-C(1)-C(2)	109.9(2)
C(3)-C(1)-C(2)	111.4(2)
N(1)-C(1)-H(1)	107.5
C(3)-C(1)-H(1)	107.5
C(2)-C(1)-H(1)	107.5
N(2)-C(2)-C(9)	113.1(2)
N(2)-C(2)-C(1)	108.8(2)
C(9)-C(2)-C(1)	111.1(2)
N(2)-C(2)-H(2)	107.9
C(9)-C(2)-H(2)	107.9
C(1)-C(2)-H(2)	107.9
C(8)-C(3)-C(4)	118.7(3)
C(8)-C(3)-C(1)	121.6(2)

C(4)-C(3)-C(1)	119.7(3)
C(5)-C(4)-C(3)	120.2(3)
C(5)-C(4)-H(4)	119.9
C(3)-C(4)-H(4)	119.9
C(6)-C(5)-C(4)	120.1(3)
C(6)-C(5)-H(5)	119.9
C(4)-C(5)-H(5)	119.9
C(5)-C(6)-C(7)	120.3(3)
C(5)-C(6)-H(6)	119.9
C(7)-C(6)-H(6)	119.9
C(8)-C(7)-C(6)	119.3(3)
C(8)-C(7)-H(7)	120.4
C(6)-C(7)-H(7)	120.4
C(3)-C(8)-C(7)	121.4(3)
C(3)-C(8)-H(8)	119.3
C(7)-C(8)-H(8)	119.3
C(10)-C(9)-C(14)	119.1(3)
C(10)-C(9)-C(2)	120.2(3)
C(14)-C(9)-C(2)	120.8(3)
C(9)-C(10)-C(11)	120.5(3)
C(9)-C(10)-H(10)	119.7
C(11)-C(10)-H(10)	119.7
C(12)-C(11)-C(10)	120.2(3)
C(12)-C(11)-H(11)	119.9
C(10)-C(11)-H(11)	119.9
C(11)-C(12)-C(13)	119.6(3)
C(11)-C(12)-H(12)	120.2
C(13)-C(12)-H(12)	120.2
C(12)-C(13)-C(14)	120.3(3)
C(12)-C(13)-H(13)	119.8
C(14)-C(13)-H(13)	119.8
C(9)-C(14)-C(13)	120.2(3)
C(9)-C(14)-H(14)	119.9
C(13)-C(14)-H(14)	119.9

N(2)-C(15)-H(15A)	109.5
N(2)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
N(2)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
N(1)-C(16)-H(16A)	109.5
N(1)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
N(1)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5
N(3)-C(17)-C(19)	113.4(2)
N(3)-C(17)-C(18)	109.3(2)
C(19)-C(17)-C(18)	109.9(2)
N(3)-C(17)-H(17)	108.0
C(19)-C(17)-H(17)	108.0
C(18)-C(17)-H(17)	108.0
N(4)-C(18)-C(25)	114.6(2)
N(4)-C(18)-C(17)	109.4(2)
C(25)-C(18)-C(17)	110.5(2)
N(4)-C(18)-H(18)	107.3
C(25)-C(18)-H(18)	107.3
C(17)-C(18)-H(18)	107.3
C(20)-C(19)-C(24)	118.9(3)
C(20)-C(19)-C(17)	120.8(2)
C(24)-C(19)-C(17)	120.2(2)
C(19)-C(20)-C(21)	120.7(3)
C(19)-C(20)-H(20)	119.7
C(21)-C(20)-H(20)	119.7
C(22)-C(21)-C(20)	120.0(3)
C(22)-C(21)-H(21)	120.0
C(20)-C(21)-H(21)	120.0
C(21)-C(22)-C(23)	120.2(3)

C(21)-C(22)-H(22)	119.9
C(23)-C(22)-H(22)	119.9
C(24)-C(23)-C(22)	119.7(3)
C(24)-C(23)-H(23)	120.2
C(22)-C(23)-H(23)	120.2
C(23)-C(24)-C(19)	120.6(3)
C(23)-C(24)-H(24)	119.7
C(19)-C(24)-H(24)	119.7
C(30)-C(25)-C(26)	118.5(3)
C(30)-C(25)-C(18)	119.8(3)
C(26)-C(25)-C(18)	121.5(2)
C(27)-C(26)-C(25)	120.7(3)
C(27)-C(26)-H(26)	119.6
C(25)-C(26)-H(26)	119.6
C(26)-C(27)-C(28)	119.5(3)
C(26)-C(27)-H(27)	120.3
C(28)-C(27)-H(27)	120.3
C(29)-C(28)-C(27)	120.4(3)
C(29)-C(28)-H(28)	119.8
C(27)-C(28)-H(28)	119.8
C(28)-C(29)-C(30)	120.2(3)
C(28)-C(29)-H(29)	119.9
C(30)-C(29)-H(29)	119.9
C(25)-C(30)-C(29)	120.7(3)
C(25)-C(30)-H(30)	119.7
C(29)-C(30)-H(30)	119.7
N(3)-C(31)-H(31A)	109.5
N(3)-C(31)-H(31B)	109.5
H(31A)-C(31)-H(31B)	109.5
N(3)-C(31)-H(31C)	109.5
H(31A)-C(31)-H(31C)	109.5
H(31B)-C(31)-H(31C)	109.5
N(4)-C(32)-H(32A)	109.5
N(4)-C(32)-H(32B)	109.5

H(32A)-C(32)-H(32B)	109.5
N(4)-C(32)-H(32C)	109.5
H(32A)-C(32)-H(32C)	109.5
H(32B)-C(32)-H(32C)	109.5
C(6S)-C(1S)-C(2S)	116.4(11)
C(6S)-C(1S)-C(7S)	123.8(11)
C(2S)-C(1S)-C(7S)	119.9(11)
C(3S)-C(2S)-C(1S)	123.1(11)
C(3S)-C(2S)-H(2S)	118.4
C(1S)-C(2S)-H(2S)	118.4
C(2S)-C(3S)-C(4S)	118.4(8)
C(2S)-C(3S)-H(3S)	120.8
C(4S)-C(3S)-H(3S)	120.8
C(5S)-C(4S)-C(3S)	122.2(8)
C(5S)-C(4S)-H(4S)	118.9
C(3S)-C(4S)-H(4S)	118.9
C(4S)-C(5S)-C(6S)	116.1(8)
C(4S)-C(5S)-H(5S)	122.0
C(6S)-C(5S)-H(5S)	122.0
C(1S)-C(6S)-C(5S)	123.8(9)
C(1S)-C(6S)-H(6S)	118.1
C(5S)-C(6S)-H(6S)	118.1
C(1S)-C(7S)-H(7S1)	109.4
C(1S)-C(7S)-H(7S2)	109.5
H(7S1)-C(7S)-H(7S2)	109.5
C(1S)-C(7S)-H(7S3)	109.5
H(7S1)-C(7S)-H(7S3)	109.5
H(7S2)-C(7S)-H(7S3)	109.5
C(6T)-C(1T)-C(2T)	117.9(18)
C(6T)-C(1T)-C(7T)	117.0(19)
C(2T)-C(1T)-C(7T)	125(2)
C(1T)-C(2T)-C(3T)	120.6(19)
C(1T)-C(2T)-H(2T)	119.7
C(3T)-C(2T)-H(2T)	119.7

C(4T)-C(3T)-C(2T)	119.5(16)
C(4T)-C(3T)-H(3T)	120.2
C(2T)-C(3T)-H(3T)	120.2
C(3T)-C(4T)-C(5T)	120.5(14)
C(3T)-C(4T)-H(4T)	119.8
C(5T)-C(4T)-H(4T)	119.8
C(6T)-C(5T)-C(4T)	118.6(12)
C(6T)-C(5T)-H(5T)	120.7
C(4T)-C(5T)-H(5T)	120.7
C(1T)-C(6T)-C(5T)	122.9(15)
C(1T)-C(6T)-H(6T)	118.6
C(5T)-C(6T)-H(6T)	118.5
C(1T)-C(7T)-H(7T1)	109.6
C(1T)-C(7T)-H(7T2)	109.5
H(7T1)-C(7T)-H(7T2)	109.5
C(1T)-C(7T)-H(7T3)	109.4
H(7T1)-C(7T)-H(7T3)	109.5
H(7T2)-C(7T)-H(7T3)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (≈^{2}x 10^3) for c32H40Br2N4Ni(C7H8). The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	22(1)	37(1)	17(1)	-7(1)	-1(1)	-1(1)
Br(2)	50(1)	30(1)	35(1)	-10(1)	19(1)	-13(1)
Ni(3)	17(1)	25(1)	12(1)	2(1)	3(1)	1(1)
N(1)	14(1)	34(1)	11(1)	-1(1)	1(1)	-3(1)
N(2)	17(1)	24(1)	18(1)	1(1)	0(1)	0(1)
N(3)	18(1)	37(2)	24(1)	12(1)	8(1)	9(1)
N(4)	18(1)	24(1)	12(1)	3(1)	1(1)	3(1)
C(1)	16(1)	24(1)	11(1)	-1(1)	3(1)	-2(1)
C(2)	15(1)	22(1)	15(1)	1(1)	2(1)	-2(1)
C(3)	16(1)	26(2)	15(1)	2(1)	1(1)	-2(1)
C(4)	28(1)	28(2)	16(1)	-2(1)	1(1)	-2(1)
C(5)	41(2)	37(2)	15(1)	4(1)	-1(1)	2(2)
C(6)	38(2)	36(2)	26(2)	11(1)	1(1)	6(1)
C(7)	33(2)	27(2)	28(2)	0(1)	7(1)	5(1)
C(8)	28(2)	31(2)	15(1)	-2(1)	4(1)	1(1)
C(9)	16(1)	26(2)	18(1)	1(1)	4(1)	1(1)
C(10)	27(2)	24(1)	28(2)	0(1)	7(1)	-1(1)
C(11)	30(2)	26(2)	40(2)	5(1)	13(2)	-4(1)
C(12)	34(2)	40(2)	32(2)	6(2)	20(1)	3(2)
C(13)	35(2)	39(2)	29(2)	-9(1)	16(1)	-1(2)
C(14)	25(2)	28(2)	28(2)	-3(1)	9(1)	0(1)
C(15)	17(1)	43(2)	31(2)	9(1)	-3(1)	-2(1)
C(16)	17(2)	70(3)	18(2)	3(2)	0(1)	-12(2)
C(17)	18(1)	22(1)	17(1)	2(1)	4(1)	4(1)
C(18)	15(1)	20(1)	12(1)	2(1)	0(1)	0(1)
C(19)	13(1)	20(1)	17(1)	1(1)	2(1)	5(1)
C(20)	17(1)	20(1)	22(1)	1(1)	-2(1)	0(1)
C(21)	21(1)	21(1)	27(2)	8(1)	-1(1)	-1(1)
C(22)	18(1)	32(2)	21(1)	7(1)	-2(1)	4(1)

C(23)	20(1)	27(2)	22(1)	-4(1)	-2(1)	0(1)
C(24)	18(1)	21(1)	24(2)	4(1)	1(1)	0(1)
C(25)	14(1)	18(1)	16(1)	4(1)	2(1)	4(1)
C(26)	21(1)	24(2)	21(1)	2(1)	-2(1)	1(1)
C(27)	19(1)	21(2)	38(2)	6(1)	-4(1)	-2(1)
C(28)	21(1)	23(2)	38(2)	14(1)	11(1)	6(1)
C(29)	31(2)	23(2)	22(1)	6(1)	10(1)	7(1)
C(30)	21(1)	20(1)	18(1)	2(1)	3(1)	2(1)
C(31)	50(2)	78(3)	55(2)	39(2)	34(2)	45(2)
C(32)	21(1)	36(2)	18(1)	8(1)	4(1)	10(1)
C(1S)	81(5)	45(4)	42(6)	-24(3)	29(8)	-24(9)
C(2S)	66(4)	45(5)	34(3)	-17(3)	19(3)	-6(3)
C(3S)	48(3)	32(3)	47(3)	-13(2)	28(3)	-14(3)
C(4S)	41(5)	39(3)	68(4)	-17(3)	33(3)	-7(3)
C(5S)	46(4)	57(4)	70(4)	-33(3)	38(3)	-24(3)
C(6S)	74(5)	68(7)	57(6)	-31(4)	32(4)	-33(4)
C(7S)	179(17)	76(5)	37(5)	-11(4)	25(9)	-18(10)
C(1T)	81(5)	45(4)	42(6)	-24(3)	29(8)	-24(9)
C(2T)	74(5)	68(7)	57(6)	-31(4)	32(4)	-33(4)
C(3T)	46(4)	57(4)	70(4)	-33(3)	38(3)	-24(3)
C(4T)	41(5)	39(3)	68(4)	-17(3)	33(3)	-7(3)
C(5T)	48(3)	32(3)	47(3)	-13(2)	28(3)	-14(3)
C(6T)	66(4)	45(5)	34(3)	-17(3)	19(3)	-6(3)
C(7T)	179(17)	76(5)	37(5)	-11(4)	25(9)	-18(10)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (≈^{2}x 10 ^{3}) for c32H40Br2N4Ni(C7H8).

	x	y	z	U(eq)
H(1N)	1100(40)	7460(30)	2056(15)	23
H(2N)	4270(40)	5290(30)	1789(14)	23
H(3N)	3930(40)	5030(30)	3546(16)	32
H(4N)	70(30)	4510(30)	2990(15)	22
H(1)	2195	6082	1062	20
H(2)	3846	7968	1852	21
H(4)	1858	7034	51	29
H(5)	995	8765	-625	37
H(6)	352	10822	-194	40
H(7)	597	11180	917	35
H(8)	1454	9450	1590	29
H(10)	5604	9156	1312	31
H(11)	7179	9459	462	38
H(12)	7352	7813	-322	42
H(13)	6032	5812	-232	41
H(14)	4493	5484	628	32
H(15A)	5996	5513	2630	45
H(15B)	6455	6347	2013	45
H(15C)	5680	7090	2591	45
H(16A)	-917	6122	1971	53
H(16B)	-692	6962	1328	53
H(16C)	-172	5430	1372	53
H(17)	2005	2926	3460	23
H(18)	1481	5456	4057	19
H(20)	2251	1317	4282	24
H(21)	3007	639	5322	27
H(22)	4283	2118	6010	29
H(23)	4822	4290	5664	28

H(24)	3994	4991	4637	25
H(26)	-1013	2815	3500	27
H(27)	-2706	1731	4142	31
H(28)	-2773	2276	5237	33
H(29)	-1130	3831	5687	30
H(30)	516	4965	5042	23
H(31A)	5403	3853	2899	90
H(31B)	5138	3056	3551	90
H(31C)	4188	2683	2906	90
H(32A)	-1002	6576	2863	37
H(32B)	-1429	5860	3517	37
H(32C)	-142	6968	3527	37
H(2S)	5018	918	2276	57
H(3S)	5520	-536	3125	50
H(4S)	7986	-1004	3439	58
H(5S)	9949	-36	2913	68
H(6S)	9337	1510	2062	78
H(7S1)	7371	3162	1608	145
H(7S2)	5693	2624	1586	145
H(7S3)	6890	1943	1143	145
H(2T)	5022	526	2400	78
H(3T)	6373	-788	3170	68
H(4T)	8937	-639	3254	58
H(5T)	10201	804	2570	50
H(6T)	8840	2097	1826	57
H(7T1)	6286	3226	1583	145
H(7T2)	5004	2121	1491	145
H(7T3)	6473	2014	1089	145

Table 6. Torsion angles [∞] for c32H40Br2N4Ni(C7H8).

C(16)-N(1)-C(1)-C(3)	64.8(3)
Ni(3)-N(1)-C(1)-C(3)	-161.78(18)
C(16)-N(1)-C(1)-C(2)	-170.3(3)
Ni(3)-N(1)-C(1)-C(2)	-36.9(3)
C(15)-N(2)-C(2)-C(9)	62.4(3)
Ni(3)-N(2)-C(2)-C(9)	-164.73(19)
C(15)-N(2)-C(2)-C(1)	-173.6(2)
Ni(3)-N(2)-C(2)-C(1)	-40.7(2)
N(1)-C(1)-C(2)-N(2)	52.6(3)
C(3)-C(1)-C(2)-N(2)	178.3(2)
N(1)-C(1)-C(2)-C(9)	177.8(2)
C(3)-C(1)-C(2)-C(9)	-56.5(3)
N(1)-C(1)-C(3)-C(8)	49.9(4)
C(2)-C(1)-C(3)-C(8)	-74.2(3)
N(1)-C(1)-C(3)-C(4)	-130.5(3)
C(2)-C(1)-C(3)-C(4)	105.4(3)
C(8)-C(3)-C(4)-C(5)	-2.1(4)
C(1)-C(3)-C(4)-C(5)	178.3(3)
C(3)-C(4)-C(5)-C(6)	0.8(5)
C(4)-C(5)-C(6)-C(7)	0.6(5)
C(5)-C(6)-C(7)-C(8)	-0.7(5)
C(4)-C(3)-C(8)-C(7)	2.1(4)
C(1)-C(3)-C(8)-C(7)	-178.3(3)
C(6)-C(7)-C(8)-C(3)	-0.7(5)
N(2)-C(2)-C(9)-C(10)	-117.7(3)
C(1)-C(2)-C(9)-C(10)	119.6(3)
N(2)-C(2)-C(9)-C(14)	63.1(3)
C(1)-C(2)-C(9)-C(14)	-59.7(3)
C(14)-C(9)-C(10)-C(11)	1.2(5)
C(2)-C(9)-C(10)-C(11)	-178.1(3)
C(9)-C(10)-C(11)-C(12)	0.6(5)
C(10)-C(11)-C(12)-C(13)	-1.6(5)

C(11)-C(12)-C(13)-C(14)	0.9(5)
C(10)-C(9)-C(14)-C(13)	-1.9(5)
C(2)-C(9)-C(14)-C(13)	177.4(3)
C(12)-C(13)-C(14)-C(9)	0.8(5)
C(31)-N(3)-C(17)-C(19)	64.9(4)
Ni(3)-N(3)-C(17)-C(19)	-162.26(19)
C(31)-N(3)-C(17)-C(18)	-172.2(3)
Ni(3)-N(3)-C(17)-C(18)	-39.3(3)
C(32)-N(4)-C(18)-C(25)	63.2(3)
Ni(3)-N(4)-C(18)-C(25)	-164.10(18)
C(32)-N(4)-C(18)-C(17)	-172.1(2)
Ni(3)-N(4)-C(18)-C(17)	-39.3(2)
N(3)-C(17)-C(18)-N(4)	53.3(3)
C(19)-C(17)-C(18)-N(4)	178.3(2)
N(3)-C(17)-C(18)-C(25)	-179.6(2)
C(19)-C(17)-C(18)-C(25)	-54.6(3)
N(3)-C(17)-C(19)-C(20)	-131.9(3)
C(18)-C(17)-C(19)-C(20)	105.5(3)
N(3)-C(17)-C(19)-C(24)	52.5(3)
C(18)-C(17)-C(19)-C(24)	-70.1(3)
C(24)-C(19)-C(20)-C(21)	1.7(4)
C(17)-C(19)-C(20)-C(21)	-174.0(3)
C(19)-C(20)-C(21)-C(22)	-1.3(5)
C(20)-C(21)-C(22)-C(23)	-0.2(4)
C(21)-C(22)-C(23)-C(24)	1.3(4)
C(22)-C(23)-C(24)-C(19)	-1.0(4)
C(20)-C(19)-C(24)-C(23)	-0.5(4)
C(17)-C(19)-C(24)-C(23)	175.2(2)
N(4)-C(18)-C(25)-C(30)	-135.0(3)
C(17)-C(18)-C(25)-C(30)	100.8(3)
N(4)-C(18)-C(25)-C(26)	48.1(4)
C(17)-C(18)-C(25)-C(26)	-76.1(3)
C(30)-C(25)-C(26)-C(27)	0.1(4)
C(18)-C(25)-C(26)-C(27)	177.0(3)

C(25)-C(26)-C(27)-C(28)	-0.2(4)
C(26)-C(27)-C(28)-C(29)	-0.8(4)
C(27)-C(28)-C(29)-C(30)	1.8(4)
C(26)-C(25)-C(30)-C(29)	0.9(4)
C(18)-C(25)-C(30)-C(29)	-176.1(3)
C(28)-C(29)-C(30)-C(25)	-1.9(4)
C(6S)-C(1S)-C(2S)-C(3S)	2(6)
C(7S)-C(1S)-C(2S)-C(3S)	-178(3)
C(1S)-C(2S)-C(3S)-C(4S)	-2(4)
C(2S)-C(3S)-C(4S)-C(5S)	-0.1(14)
C(3S)-C(4S)-C(5S)-C(6S)	1.2(10)
C(2S)-C(1S)-C(6S)-C(5S)	-1(5)
C(7S)-C(1S)-C(6S)-C(5S)	179(3)
C(4S)-C(5S)-C(6S)-C(1S)	-1(3)
C(6T)-C(1T)-C(2T)-C(3T)	0(10)
C(7T)-C(1T)-C(2T)-C(3T)	177(6)
C(1T)-C(2T)-C(3T)-C(4T)	0(7)
C(2T)-C(3T)-C(4T)-C(5T)	0(3)
C(3T)-C(4T)-C(5T)-C(6T)	1(2)
C(2T)-C(1T)-C(6T)-C(5T)	0(10)
C(7T)-C(1T)-C(6T)-C(5T)	-176(4)
C(4T)-C(5T)-C(6T)-C(1T)	-1(5)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for c32H40Br2N4Ni(C7H8) [≈ and ∞].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1N)...Br(1)	0.87(2)	2.84(3)	3.283(2)	114(2)
N(2)-H(2N)...Br(2)	0.86(2)	2.86(3)	3.332(3)	116(3)
N(3)-H(3N)...Br(1)	0.86(2)	2.97(4)	3.413(3)	114(3)
N(4)-H(4N)...Br(2)	0.85(2)	3.04(3)	3.430(2)	111(3)

Symmetry transformations used to generate equivalent atoms: