Organic Syntheses

Dirhodium (II) tetrakis[*N*-4-bromo-1,8-naphthoyl-(*S*)-*tert*-leucinate]

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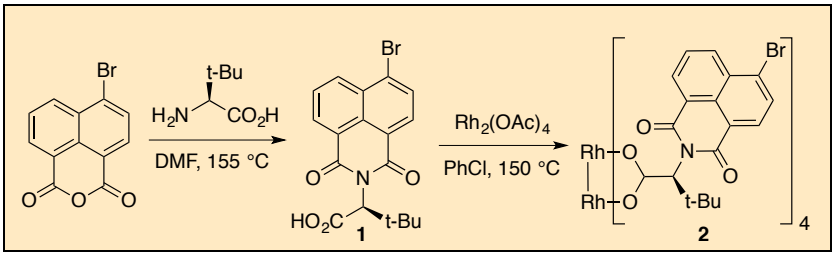
Checked by David Stephens and Richmond Sarpong



Procedure

A. *N-4-Bromo-1,8-naphtaloyl-(S)-tert-leucine, [(S)-4-Br-nttl] (**1**).* To a 100 mL three-necked round-bottomed flask (left neck with a nitrogen inlet, middle with a 20 cm reflux condenser with an outlet leading to a bubbler, and right with a thermometer) is added a 2 cm Teflon-coated magnetic stir bar, 4-bromo-1,8-naphthalic anhydride (3.26 g, 11.8 mmol, 1.00 equiv), L-*tert*-leucine (1.55 g, 11.8 mmol, 1.00 equiv) (Note 1) and dimethylformamide (DMF) (30 mL) (Note 2). The resulting brownish suspension is added to a preheated 160 °C oil bath, stirred (2000 rpm) at reflux (145–149 °C internal) for 2 h under continuous nitrogen atmosphere (Notes 3 and 4) (Figure 1). The reaction mixture is cooled to room temperature (Figure 2) and DMF is removed by short path distillation with the aid of high vacuum (80 °C oil bath, 100 to 80 mbar vacuum) (Note 5) until a brown-orange oil is obtained

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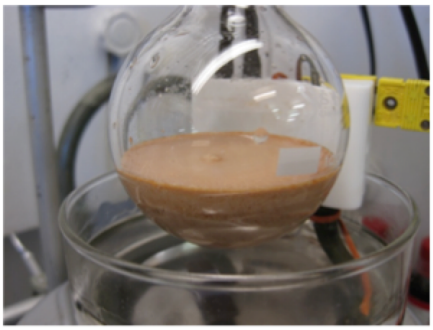
Figure 1. Glassware assembly for Step A



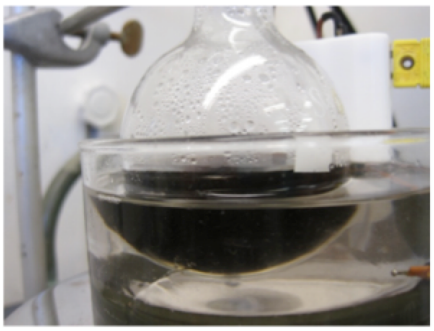


Figure 2. Reaction appearance before (left) and after (right) heating (photo provided by Authors, who performed the reaction in a one-necked flask)

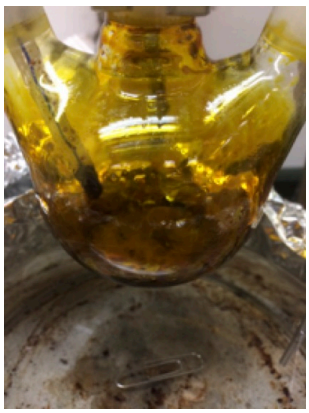


Figure 3. Crude brown oil after removal of DMF

(Figure 3). This residue is dissolved in ethyl acetate (150 mL) and transferred into a 500 mL separatory funnel. The organic layer is washed with distilled H_2O (3 x 150 mL) (Note 6), and a saturated aqueous NaCl solution (150 mL). The aqueous layers are combined, and extracted with ethyl acetate (2 x 75 mL). The combined organic layers are dried over anhydrous sodium sulfate (8-10 g) and filtered into a 500 mL round-bottomed flask. The sodium sulfate is washed with ethyl acetate (3 x 15 mL) into the round-bottomed flask, and the solution is concentrated under reduced pressure (250 to 120 mmHg, 40 °C) to yield a brown solid (Figure 4).

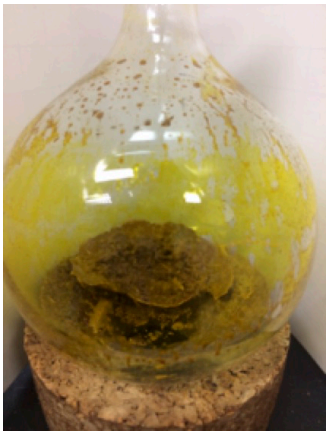


Figure 4. Crude *N*-4-Bromo-1,8-naphtaloyl-(*S*)-*tert*-leucine prior to chromatography

A dry pack of the crude product (Note 7) is charged on a column of silica gel (130 g) (Notes 8 and 9) and eluted with a mixture of ethyl acetate/hexanes (3:7) with 1% formic acid (Note 10). Fractions (25 mL fraction) of desired carboxylic acid are collected (Notes 11 and 12), combined and concentrated by rotary evaporation (275 to 120 mmHg, 40 °C) to afford a yellow solid (3.72 g). The product is transferred into a 100 mL round-bottomed flask. Dichloromethane (25 mL) and methanol (0.5 mL) are added. The resulting yellow mixture is heated at reflux until complete dissolution and then cooled to room temperature. The flask is then stored at 0–4 °C overnight to form pale yellow crystals. The crystals are filtered through filter paper with a 6.5 cm diameter Büchner funnel by suction, and

then washed with hexanes (15 mL) to yield 1.48 g of the desired product. The mother liquors are collected, concentrated and two additional recrystallizations and one precipitation processes are achieved (Notes 13 and 14) (Figure 5). The pale yellow solid is dried under high vacuum (0.5 mmHg, rt) for 16 h, affording a total of 3.35 g (8.61 mmol, 73% yield) of the desired product (Notes 15, 16, and 17).

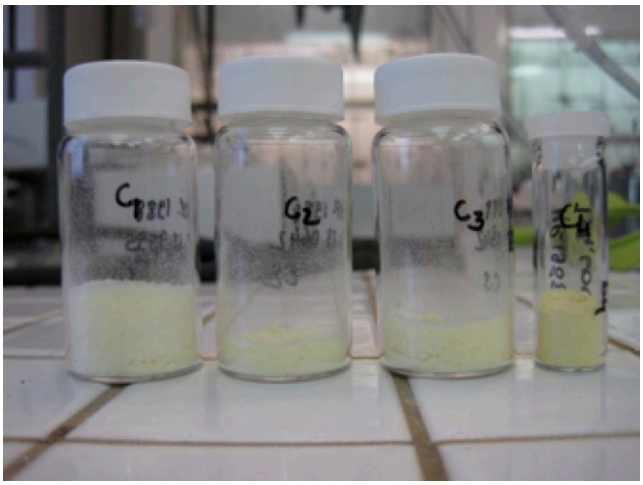


Figure 5. Fractions of carboxylic acid (*1*) after recrystallization

B. *Dirhodium (II) tetrakis[N-4-bromo-1,8-naphthoyl-(S)-tert-leucinate] [Rh_2{(S)-4-Br-nttl}$_4$] (**2**).* A 100 mL three-necked, round-bottomed flask (left neck with a nitrogen inlet, middle neck with a Soxhlet extractor (Note 18) attached to a bubbler, and the left with a thermometer) is equipped with a 2 cm Teflon-coated magnetic stir bar (Note 19). The flask is charged with rhodium (II) acetate (0.570 g, 1.28 mmol, 1.00 equiv) (Note 20), *N*-4-Bromo-1,8-naphthaloyl-(*S*)-*tert*-leucine (**1**) (3.00 g, 7.69 mmol, 6.00 equiv) and chlorobenzene (50 mL, 0.03 M) (Note 21).

The extractor body is filled with an oven-dried mixture of potassium carbonate (2 g) and sand (1 g) (Notes 22 and 23). The resulting mixture is stirred (2000 rpm) and heated at reflux under a continuous flow of nitrogen for 16 h (Notes 24 and 25) (Figure 6). The reaction is monitored by TLC (Note 26).

Figure 6. Glassware assembly for Step A

The reaction mixture is then cooled to room temperature, and the thermometer and the nitrogen inlet are replaced with glass stoppers. The chlorobenzene is removed by a short path distillation (Note 27), affording a dark green solid. The residue is dissolved in a minimum volume of diethyl ether (4-5 mL, Et_2O), which is then filtered through a pad of basic alumina (140 g) (Notes 28 and 29) to remove residual carboxylic acid and dark impurities, eluting with Et_2O (Note 30) (Figure 7).

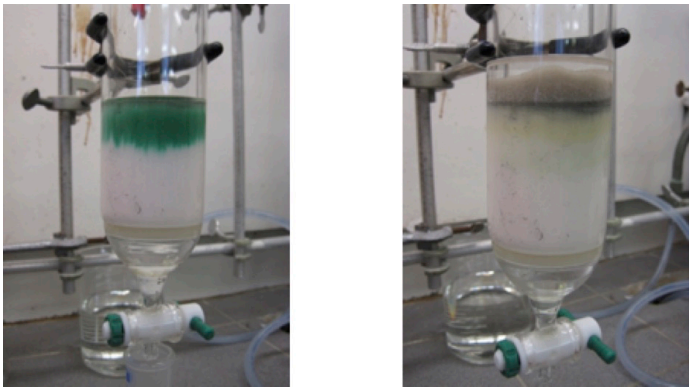


Figure 7. Initiation (left) and completion (right) of filtration on alumina

The filtrate is collected in 25 mL fractions and then transferred into a 500 mL round-bottomed flask. The green filtrate is evaporated to dryness using a 40 °C water bath, with the eventual application of a slight vacuum (650 mmHg). To the green solid is added diethyl ether (10 mL) and pentane (100 mL). The mixture is then gently shaken by hand until a mint green precipitate formed. The green precipitate is vacuum filtered through a filter paper using a 6.5 cm diameter Büchner funnel (Figure 8).

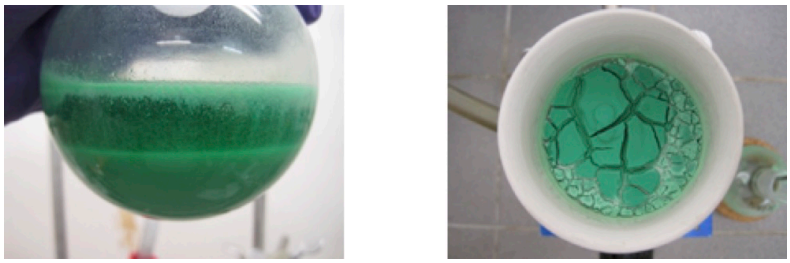


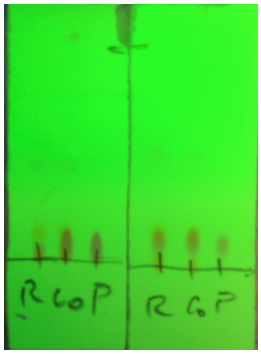
Figure 8. Precipitation in pentane (left) and filtration (right)

The solid is washed with pentane (10 mL) and dried 10-15 min by continued vacuum application. The green solid is carefully collected, affording 1.47 g of the desired catalyst. The mother liquors are collected into a 100 mL round-bottomed flask (Note 31), concentrated to dryness and the precipitation process is repeated (Note 32). The solids are combined and dried under high vacuum (0.5 mm Hg) at room temperature for 24 h (Note 33), affording a total of 2.26 g (1.26 mmol, 95% yield) of a pale green powder (Notes 34, 35, 36, 37, and 38).

Notes

1. 4-Bromo-1,8-naphtalic anhydride (95%) and neutral L-*tert*-leucine (99%, 99% ee) are purchased from Sigma-Aldrich Fine Chemicals Company Inc. and used as received.
2. The submitters used dimethylformamide (spectrograde) purchased from Caledon Company and used as received. The checkers used dimethylformamide (peptide grade) purchased from Acros Organics and used as received.

3. Residual solids in suspension are progressively solubilized during heating. If dark brownish mixture is not completely homogeneous when reflux temperature is reached, additional portions of methanol (50 μL) are added until all solids are dissolved.
4. The reaction is monitored by TLC analysis on silica gel using a mixture of EtOAc:hexanes (3:7) with 1% formic acid and visualized with UV light (254 nm) (R_f anhydride 0.6; R_f (*S*)-4-Br-nttl 0.2).
5. Alternatively, DMF can be removed by rotary evaporation.
6. An orange emulsion is formed (carboxylic acid in the interphase) on some occasions. The addition of a saturated aqueous solution of sodium chloride (10-15 mL) helps to separate the two layers.
7. Silica gel (8-9 g) is added to a solution of the crude product in dichloromethane (50 mL). The solvent is removed under reduced pressure to afford an orange-brown solid, which is dry loaded onto the column.
8. Silica gel F60 type 40–63 μm (230–400 mesh) was purchased from Silicycle Inc. and used as received.
9. The pad is a cylinder of 5.5 cm diameter and 13 cm of height.
10. Purification is followed by TLC analysis on silica gel using a mixture of EtOAc in hexanes (3:7) with 1% formic acid and visualization with UV light (254 nm) (R_f (*S*)-4-Br-nttl 0.2).



Both TLC plates spotted (from left to right) with the reaction mixture, co-spot, and the product. The left TLC is eluted with hexanes/EtOAc (7:3) with the product on the baseline. The right TLC plate is eluted with hexanes/EtOAc (7:3) with 1% formic acid to move the product off the baseline. Visualized using UV-light (254 nm). The anhydride starting material has $R_f = 0.6$ in this solvent system.

11. The desired carboxylic acid is typically obtained in fractions 16 to 58 (yielding 1.76 g). The impure fractions can be subjected to a second column for further purification (yielding 1.59 g).
12. Traces of residual UV-active impurities (R_f = 0.2) are removed during the recrystallization process.
13. After each filtration, the filter paper and the Büchner funnel are rinsed with dichloromethane and the mother liquors are concentrated under reduced pressure. The second recrystallization process uses dichloromethane (10 mL) and methanol (0.4 mL) at 0 °C for 2–3 h to furnish an additional 0.63 g of the desired product. The third recrystallization uses dichloromethane (6–8 mL) and methanol (0.2 mL) at 0 °C for 2–3 h to afford 0.65 g of desired product. For both recrystallizations, additional aliquats of MeOH (50 μL at a time) are added till the compound dissolves at reflux.
14. After the third recrystallization, the product is precipitated by concentrating the mother liquors and suspending the resulting yellow solid in a mixture of dichloromethane (5 mL) and hexanes (20 mL) and stirred for 20 min at room temperature. The pale yellow solid is filtered and washed with hexanes (5-8 mL), recovering 0.56 g of additional product All recrystallization solids show the same level of purity.
15. Dichloromethane can be encapsulated in crystals, and drying under high vacuum fails to remove it. Re-dissolution of the solid in dichloromethane and subsequent evaporation followed by drying under high vacuum typically yields crystals free of dichloromethane.
16. A second reaction on identical scale provided 3.27 g (71%) of the product, and a reaction performed on half-scale provided 1.67 g (73%) of the product.
17. Analytical data for *N*-4-bromo-1,8-naphthaloyl-(*S*)-*tert*-leucine: R_f 0.20 (EtOAc:hexanes (3:7) with 1% formic acid); ^{1}H NMR (600 MHz, $CDCl_3$, 298K, mixture of conformers) δ 1.19 (18H, s), 5.58 (2H, s), 7.85 (2H, apparent t, *J* = 7.8 Hz), 8.04 (2H, d, *J* = 7.8 Hz), 8.40 (1H, d, *J* = 8.4 Hz), 8.43 (1H, d, *J* = 8.4 Hz), 8.56 (2H, d, *J* = 8.4 Hz), 8.64 (1H, d, *J* = 7.2 Hz), 8.68 (1H, d, *J* = 7.2 Hz); ^{13}C NMR (151 MHz, $CDCl_3$, 298K, mixture of conformers) δ: 28.55, 36.10, 59.97, 121.77, 122.01, 122.65, 122.88, 128.24, 128.32, 128.97, 130.57, 130.63, 130.73, 131.25, 131.29, 131.76, 131.81 (br), 132.13 (br), 132.67 (br), 133.00 (br), 133.53, 133.62, 163.50 (br), 164.0 (br), 174.58; ^{1}H NMR (600 MHz, DMSO-d_6, 373K) δ : 1.16 (9H, s), 5.40 (1H, s), 8.00 (1H, apparent t, *J* = 7.8 Hz), 8.20 (1H, d, *J* = 7.8 Hz), 8.39 (1H, d, *J* = 7.8 Hz), 8.56 (1H, d, *J* = 8.4 Hz), 8.61 (1H, d, *J* = 7.8 Hz), 12.08 (1H, br s);

^{13}C NMR (151 MHz, DMSO-d_6, 373K) δ : 27.91, 34.81, 59.38, 121.24, 122.04, 127.83, 128.28, 128.90, 129.55, 130.96, 130.98 (br), 131.60 (br), 132.35, 162.66, 162.69, 168.30; IR (film): 751, 779, 1238, 1342, 1367, 1587, 1668, 1708, 2872, 2913 cm^{-1}; mp 233–235 °C, $[\alpha]_D^{21}$ –79.2 (*c* 1.02, $CHCl_3$); $[M + Na]^+$ calcd for $C_{18}H_{16}NNaO_4^+$: 412.0155; Found: 412.0157; Calcd for $C_{18}H_{16}BrNO_4$: C, 55.40; H, 4.13; N, 3.59; Found: C, 55.06; H, 4.26; N, 3.53.

18. A micro-size Soxhlet extraction apparatus (Chemglass, Inc.) consisting of the extractor (19/22 to inner joint and 14/20 lower inner joint) fitted with a small piece of cotton (2/2 cm) to cover the extractor body exit and an Allihn condenser with water circulation is used without further modification.
19. Glassware is oven-dried at 110 °C overnight.
20. Rhodium (II) acetate dimer is purchased from Pressure Chemical Company, stored, weighed in a glovebox under argon atmosphere and used without further purification. No detriment to the reaction was observed if the dimer is stored and weighed outside the glovebox.
21. The submitters used chlorobenzene (Laboratory grade) purchased from Caledon Company and used as received. The checkers used chlorobenzene (>98% GC analysis) purchased from TCI and used as received.
22. The submitters used potassium carbonate purchased from Caledon Company and used as received. The checkers used anhydrous potassium carbonate (99.8% purity) purchased from Fisher and used as received.
23. The K_2CO_3/sand mixture is covered with 2-4 mm of sand. The pad is then moistened with chlorobenzene (4 mL).
24. Oil bath temperature is 160-165 °C. A good reflux is needed to evacuate the maximum of acetic acid. The internal temperature of the reaction is between 135-140 °C.
25. The initially dark green heterogeneous mixture turns homogeneous during heating. Aluminum foil and cotton are used to insulate the extractor body of the Soxhlet apparatus.
26. The TLC plate is eluted with 1:1 hexanes/EtOAc. The product has R_f = 0.72; *N*-4-Bromo-1,8-naphtaloyl-(*S*)-*tert*-leucine (**1**) has R_f = 0.45.
27. Chlorobenzene can also be removed by rotary evaporation.
28. Brockmann I type, 58 Å pore size basic alumina was purchased from Sigma-Aldrich Fine Chemicals Company Inc. and used as received.
29. The pad is a cylinder of 5.5 cm diameter and 6.5 cm of height.

30. Approx. 1000 mL of Et_2O is needed. Migration of the catalyst on alumina is easily followed by the green color. The first colorless fractions are discarded (typically residual chlorobenzene). Only the green fractions are collected.
31. After each filtration process, the Büchner funnel and filter paper are rinsed with 5-10 mL of Et_2O to maximize the yield.
32. For subsequent precipitations, 1-3 mL of diethyl ether and 20-30 mL of pentane are used. Typically, four iterations of the precipitation process are necessary to maximize isolation of the catalyst with retention of purity through the crystallizations.
33. Aggregates are crushed 3-4 times during drying process with a spatula.
34. Two reactions performed on half scale provided 1.03 g (89%) and 1.04 g (90%) of the product, respectively.
35. Analytical data for Dirhodium (II) tetrakis[*N*-4-bromo-1,8-naphthoyl-(*S*)-*tert*-leucinate] [Rh_2{(*S*)-4-Br-nttl}$_4$]: Rf 0.72 (Hex/EtOAc); ^{1}H NMR (600 MHz, $CDCl_3$, mixture of conformers) δ: 1.16 (diethyl ether), 1.28* (36 H, s), 3.67 (diethyl ether), 5.76 – 5.83 (4H, m), 7.60–7.69 (2H, m), 7.78–7.85 (2H, m), 7.87–7.93 (2H, m), 8.07–8.12 (2H, m), 8.27–8.37 (6H, m), 8.49–8.60 (4H, m), 8.77–8.83 (2H, m), ^{13}C NMR (151 MHz, $CDCl_3$, mixture of conformers) δ: 15.19 (residual Et_2O), 28.94, 36.32, 62.23*, 66.23 (residual Et_2O) 122.43, 122.53, 123.26, 123.34, 127.75*, 128.75, 128.80, 128.91, 128.96, 129.54*, 129.84*, 130.31*, 130.69*, 131.12*, 131.69, 131.98*, 132.46*, 132.58*, 132.87, 133.34*, 162.67, 162.71, 164.23, 164.27, 164.30, 164.33, 187.33*. * Denotes that this chemical shift represents the center of multiple closely spaced chemical shifts arising from different conformers; IR (film): 749, 786, 1236, 1263, 1340, 1364, 1397, 1571, 1588, 1604, 1665, 1707, 2870, 2954, 2996 cm^{-1}; mp 260 °C (decomp); $[\alpha]_D^{21}$ +100.5 (*c* 0.25, $CHCl_3$); [M + Na]$^+$ calcd for $C_{72}H_{60}Br_4N_4NaO_{16}Rh_2$: 1780.8740; Found: 1780.8740. Calcd for $C_{76}H_{70}Br_4N_4O_{17}Rh_2$: C, 49.70; H, 3.84; N, 3.05; Found: C, 49.68; H, 3.89; N, 3.04.
36. Up to 2 equiv of Et_2O per molecule of rhodium dimer can be present and does not affect the reactivity of the catalyst.
37. More than one rotamer/conformer is observed by NMR. Although, a DOSY experiment suggested that all reported peaks belong to only one species, the resolution to only one rotamer/conformer cannot be achieved by performing variable temperature NMR experiments.
38. Traces of water (1.8 ppm) and chlorobenzene (7.21–7.35 ppm) are sometimes observed by ^{1}H NMR and do not affect the catalyst activity. Residual chlorobenzene can be removed by filtration of the catalyst on

silica gel, eluting with a mixture of Et_2O in pentane (2:8), collecting the green fractions.

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The procedures in *Organic Syntheses* are intended for use only by persons with proper training in experimental organic chemistry. All hazardous materials should be handled using the standard procedures for work with chemicals described in references such as "Prudent Practices in the Laboratory" (The National Academies Press, Washington, D.C., 2011; the full text can be accessed free of charge at http://www.nap.edu/catalog.php?record_id=12654). All chemical waste should be disposed of in accordance with local regulations. For general guidelines for the management of chemical waste, see Chapter 8 of Prudent Practices.

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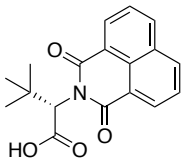
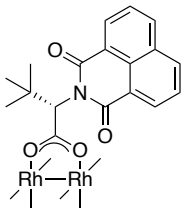
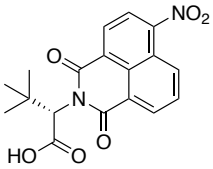
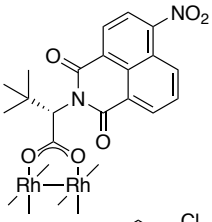
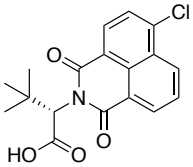
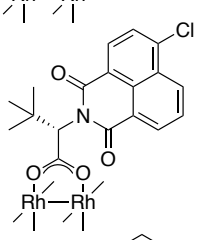
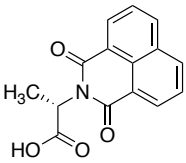
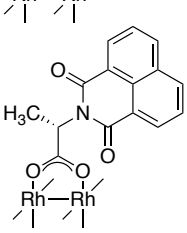
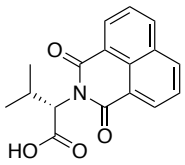
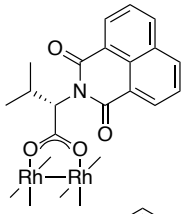
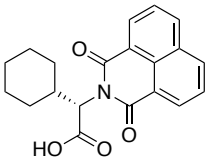
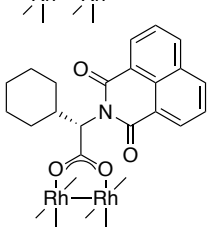
Discussion

Rhodium(II) carboxylate dimers are catalysts typically used in metal carbene[2] and nitrene chemistry.[3] Rhodium carbene precursors include diazo reagents[4] and *N*-sulfonyl-1,2,3-triazoles,[5] where as iminoiodinanes,[6] azides,[7]

and sulfonyloxycarbamates[8,9] are used to prepare rhodium nitrene species. Dirhodium carbene and nitrene species display high reactivity for carbon-carbon double or triple bonds as well as for C-H bonds.[2,3]

Naphthoyl amino acid-derived rhodium(II) dimers are suitable asymmetric catalysts to perform numerous stereoselective transformations, including cyclopropanation[10-21] and cyclopropenation,[22] C-H insertion,[8,9,23-28] aziridination,[8] thioether amination[29,30] and 1,3-dipolar cycloaddition with aldehydes and imines.[31] Naphthoyl *tert*-leucine-derived rhodium(II) dimers, namely $Rh_2\{(S)\text{-nttl}\}_4$ and $Rh_2\{(S)\text{-4-Br-nttl}\}_4$ are easily prepared from $Rh_2(OAc)_4$ and the corresponding naphtaloyl-(*S*)-*tert*-leucine derivative.[13] A slight excess of *N*-4-Bromo-1,8-naphtaloyl-(*S*)-*tert*-leucine (**1**) (1.5 equiv) is needed to favor ligand exchange and displace all the acetate ligands from the rhodium dimer. The naphtaloyl-(*S*)-*tert*-leucine derivatives are available from (*S*)-*tert*-leucine and the corresponding naphtaloyl anhydride.[32] The described procedure is applicable to other amino acids and various naphtaloyl anhydrides, as shown in Table 1.

Table 1. Synthesis of Various Rhodium(II) Dimers

ligand	yield (%)	catalyst	yield (%)
	85		95
	59		89
	57		93
	60		71
	82		92
	60		98

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1. Département de Chimie, Center for Green Chemistry and Catalysis, Université de Montréal, C.P. 6128, Succursale Centre-ville, Montréal, Québec, Canada H3C 3J7. This research was supported by NSERC (Canada), the Canadian Foundation for Innovation, the Canada Research Chair Program, the Université de Montréal and the Centre in Green Chemistry and Catalysis (CGCC).
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Appendix
Chemical Abstracts Nomenclature (Registry Number)

4-Bromo-1,8-naphtalic anhydride: 1*H*,3*H*-Naphtho[1,8-*cd*]pyran-1,3-dione, 6-bromo-; (81-86-7)

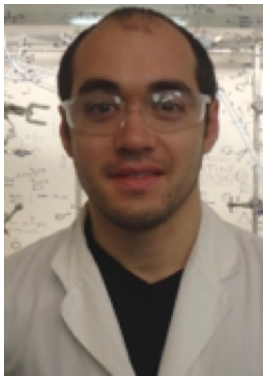
L-*tert*-Leucine: L-Valine, 3-methyl-; (20859-02-3)

N-4-Bromo-1,8-naphtaloyl-(*S*)-*tert*-leucine: 1*H*-Benz[*de*]isoquinoline-2(3*H*)-acetic acid, 6-bromo-α-(1,1-dimethylethyl)-1,3-dioxo-, (α*S*)-; (310874-15-8)

Rhodium (II) acetate: Rhodium, tetrakis[μ-(acetato-κ*O*:κ*O*')]di-, (*Rh-Rh*); (15956-28-2)

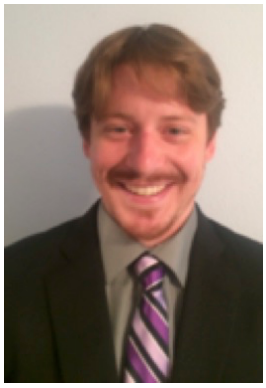
Dirhodium (II) tetrakis[*N*-4-bromo-1,8-naphthoyl-(*S*)-*tert*-leucinate] diethyl ether solvate: Rhodium, tetrakis[μ-[(α*S*)-6-bromo-α-(1,1-dimethylethyl)-1,3-dioxo-1*H*-benz[*de*]isoquinoline-2(3*H*)-acetato-κO^2:κ$O^{2'}$]]di-, (*Rh-Rh*); (802910-46-9)

Prof. Hélène Lebel obtained a B.S. degree from Université Laval (1993) and a Ph.D. from Université de Montréal (1998). She then joined the group of Eric N. Jacobsen at Harvard University as a NSERC Postdoctoral Fellow. She began her academic career at the Université de Montréal in 1999, under a NSERC University Faculty Award. She was promoted to the rank of Full Professor in 2010. Her research interests focus on the development of new synthetic methodologies in organic chemistry based on transition metal-catalyzed processes.



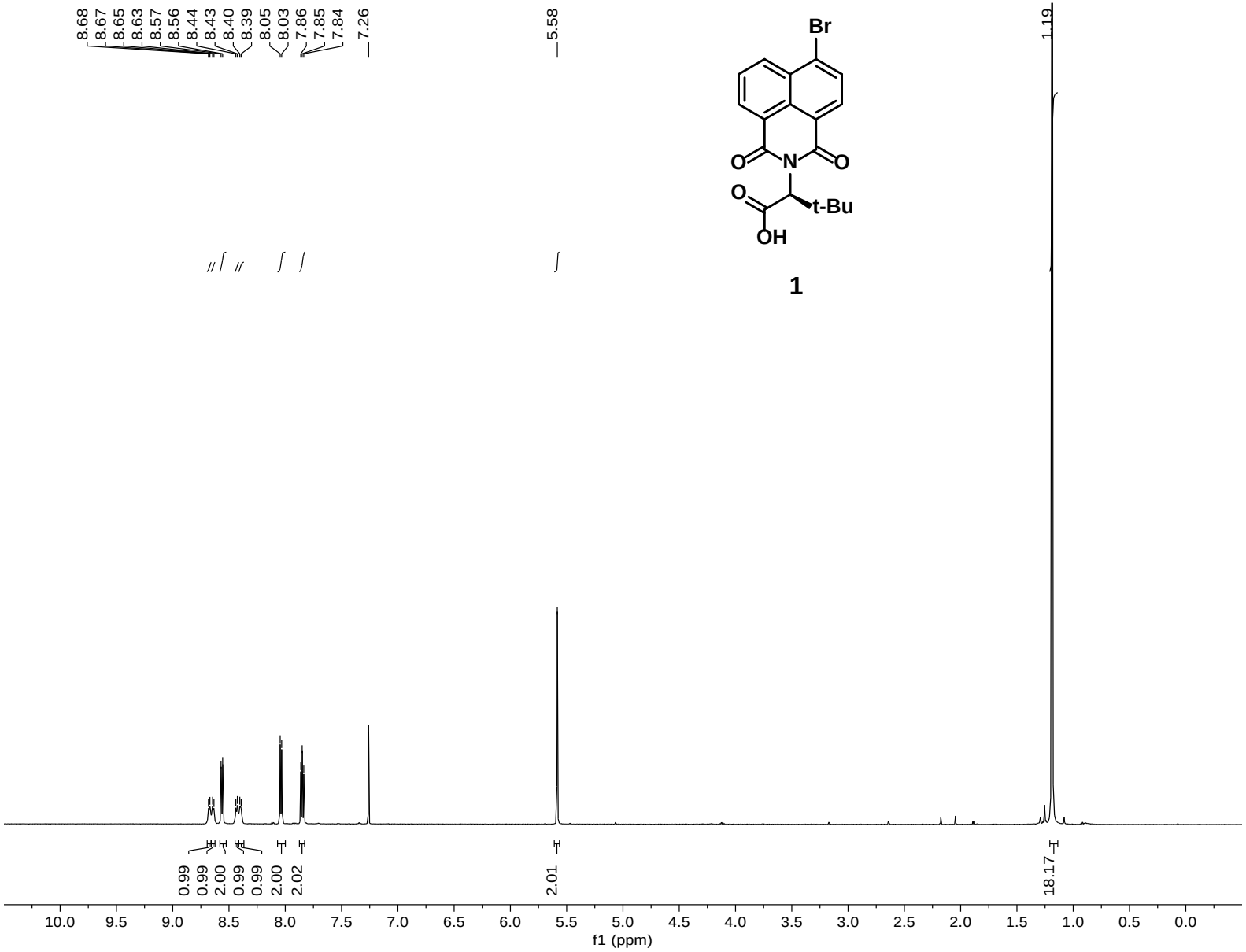
Henri Piras was born in Paris and raised in l'île de la Réunion, in France. He obtained in 2011, an Engineer degree in synthetic and industrial organic chemistry from the École National Supérieure de Chimie de Clermont-Ferrand, and a M.S. degree from Université Blaise-Pascal under the supervision of Prof. Yves Troin. Since January 2012, he has been a Ph.D. student with Prof. Hélène Lebel at Université de Montréal, working on the stereoselective synthesis of chiral sulfilimines and sulfoximines.

Johan Bartholoméüs was born and raised in Dunkerque, France. He received a Licence de chimie from Université du Littoral Côte d'Opale in Dunkerque in 2007 and a Master 1 in sciences from Université des Sciences et Technologies in Lille in 2008. He then completed a Master 2 in organic synthesis at Université de Bordeaux 1 under the supervision of Prof. Stéphane Quideaux. In September 2011, he joined the group of Prof. Hélène Lebel as a Ph.D. student and is currently writing his thesis on stereoselective amination of C-H bonds to synthesize propargylic amines.



Dr. David Stephens was born and raised in Austin, TX (USA). He received a Bachelor's of Science in Chemistry from St. Edward's University in 2011, and a Ph.D. in chemistry from the University of Texas at San Antonio under the guidance of Prof. Oleg V. Larionov in 2016. He is currently a postdoctoral researcher with Prof. Richmond Sarpong at UC Berkeley.

N-4-Bromo-1,8-naphtaloyl-(S)-tert-leucine (^{1}H NMR, 600 MHz, $CDCl_3$, 298K)



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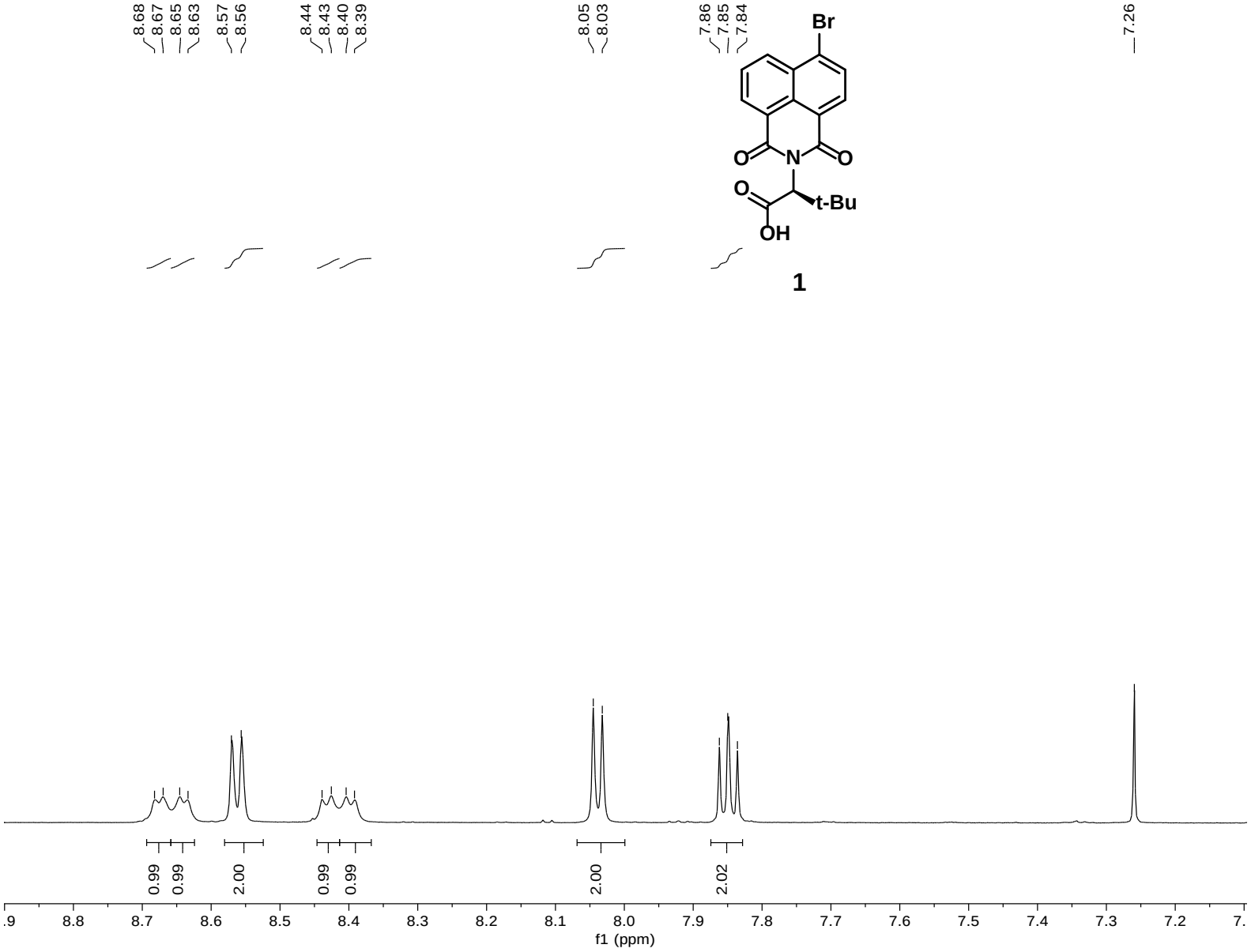

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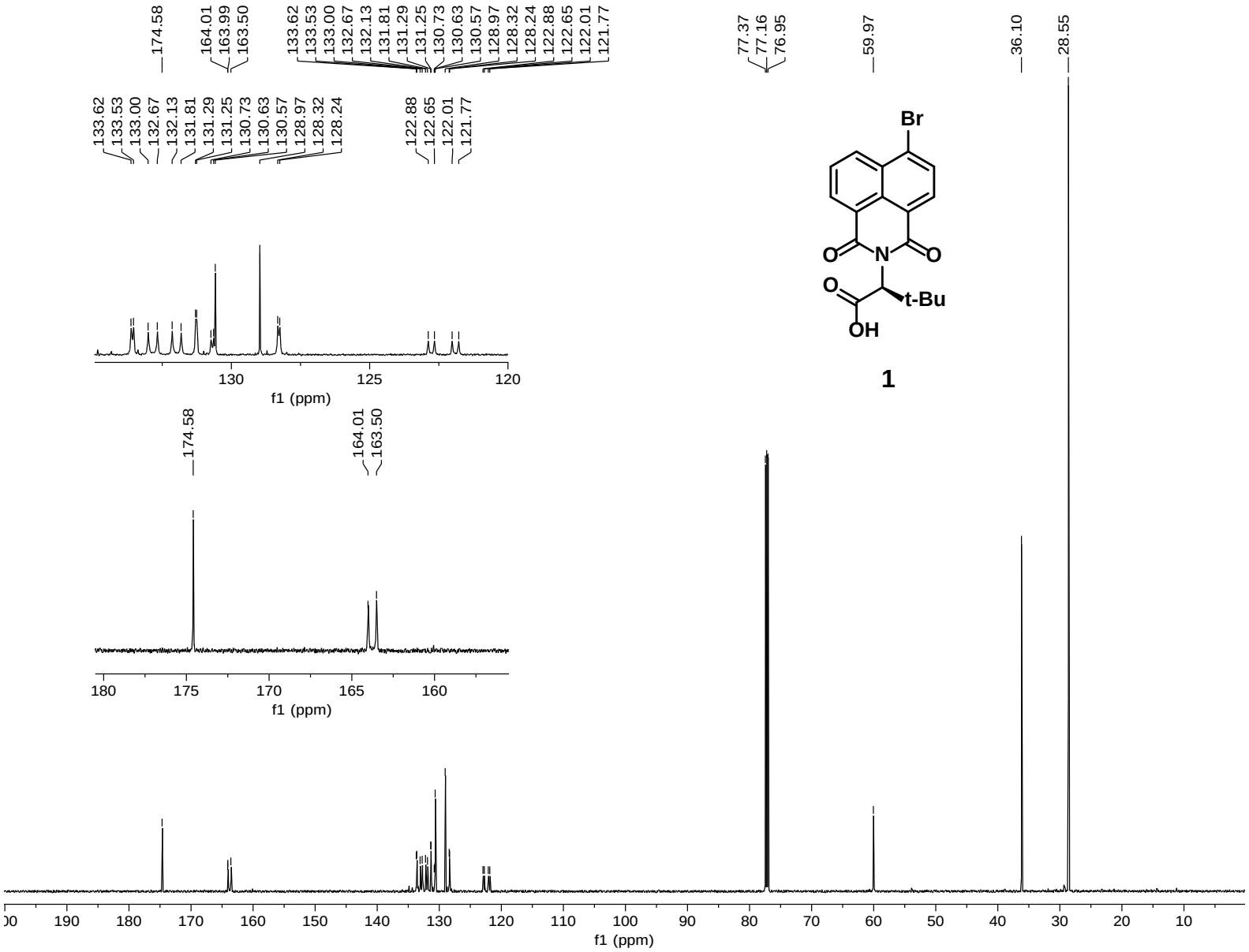
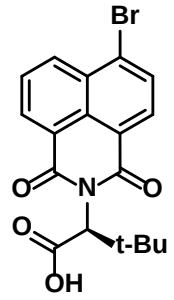
N-4-Bromo-1,8-naphtaloyl-(S)-tert-leucine (^{13}C NMR, 151 MHz, $CDCl_3$, 298K)



N-4-Bromo-1,8-naphtaloyl-(S)-tert-leucine (^{13}C NMR, 151 MHz, $CDCl_3$, 298K)

133.46
133.37
132.84
132.51
131.97
131.65
131.13
131.09
130.57
130.47
130.41
128.81
128.16
128.08
122.72
122.49
121.85
121.61



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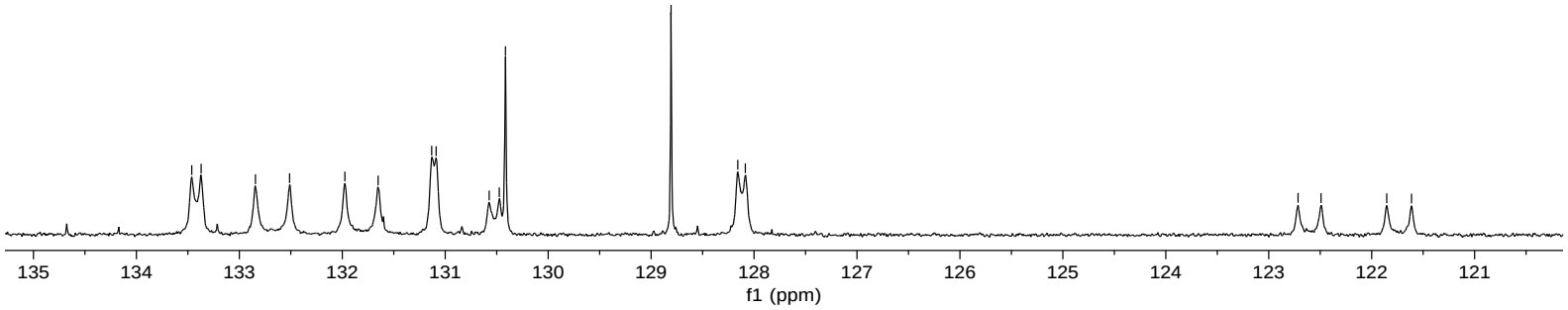

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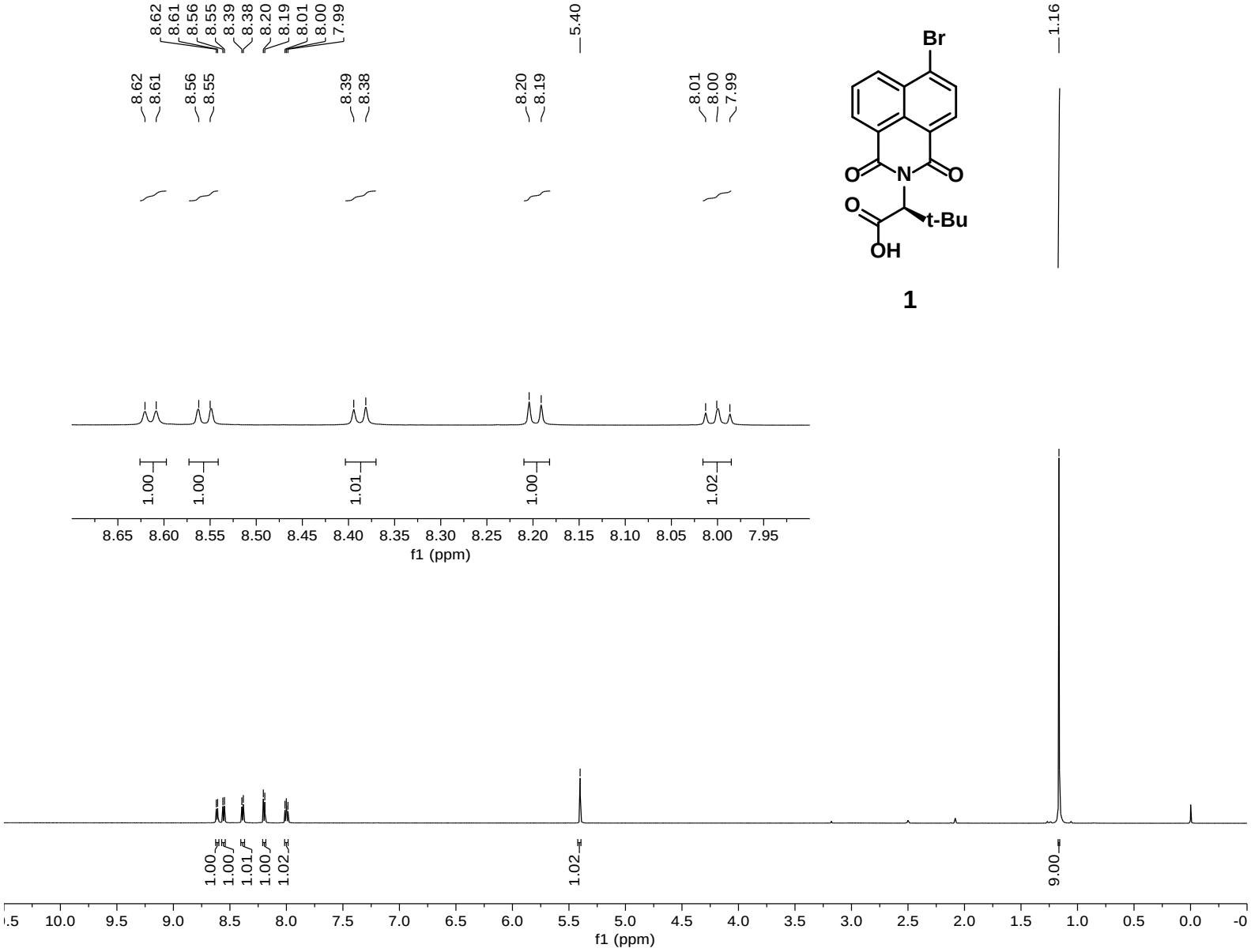

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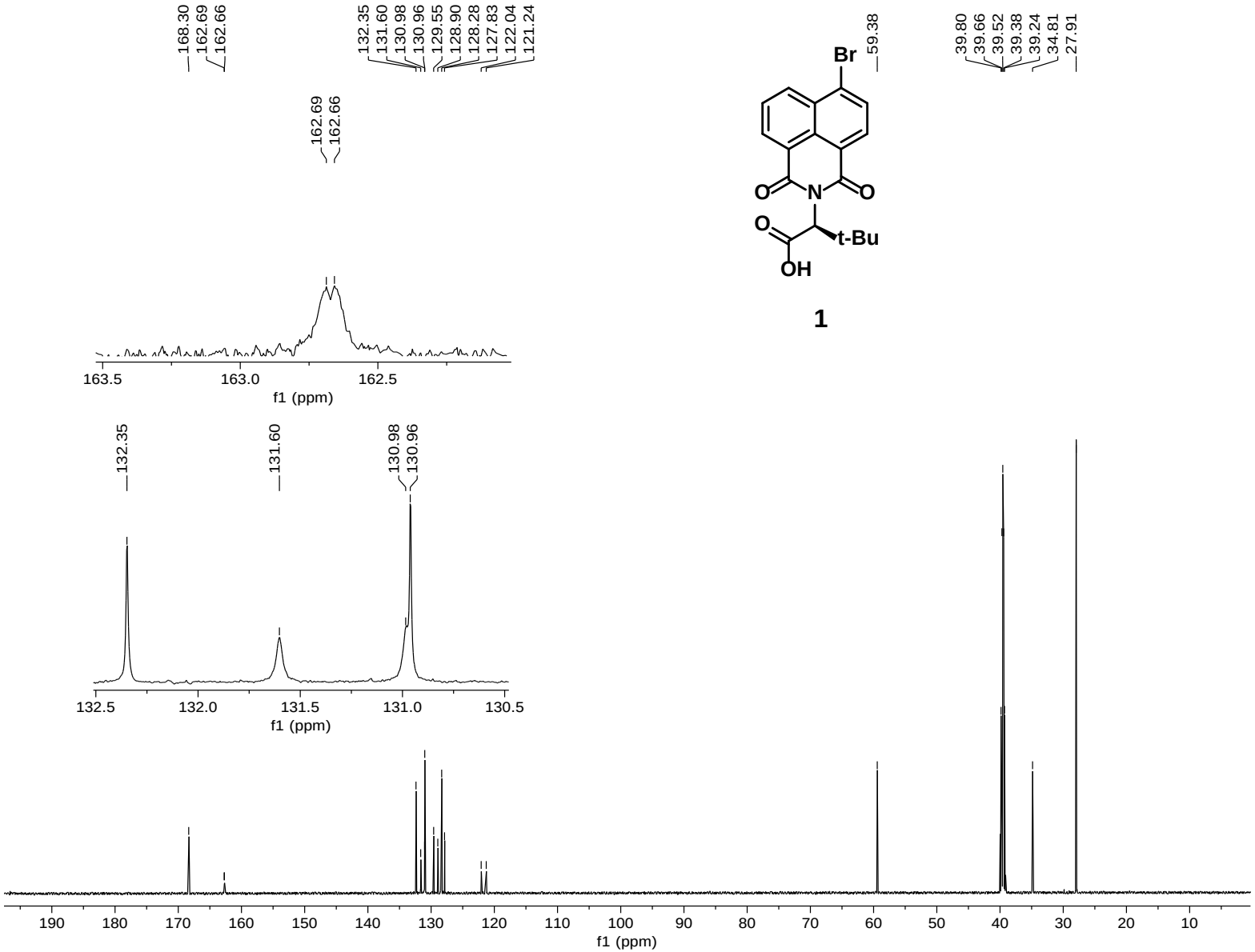
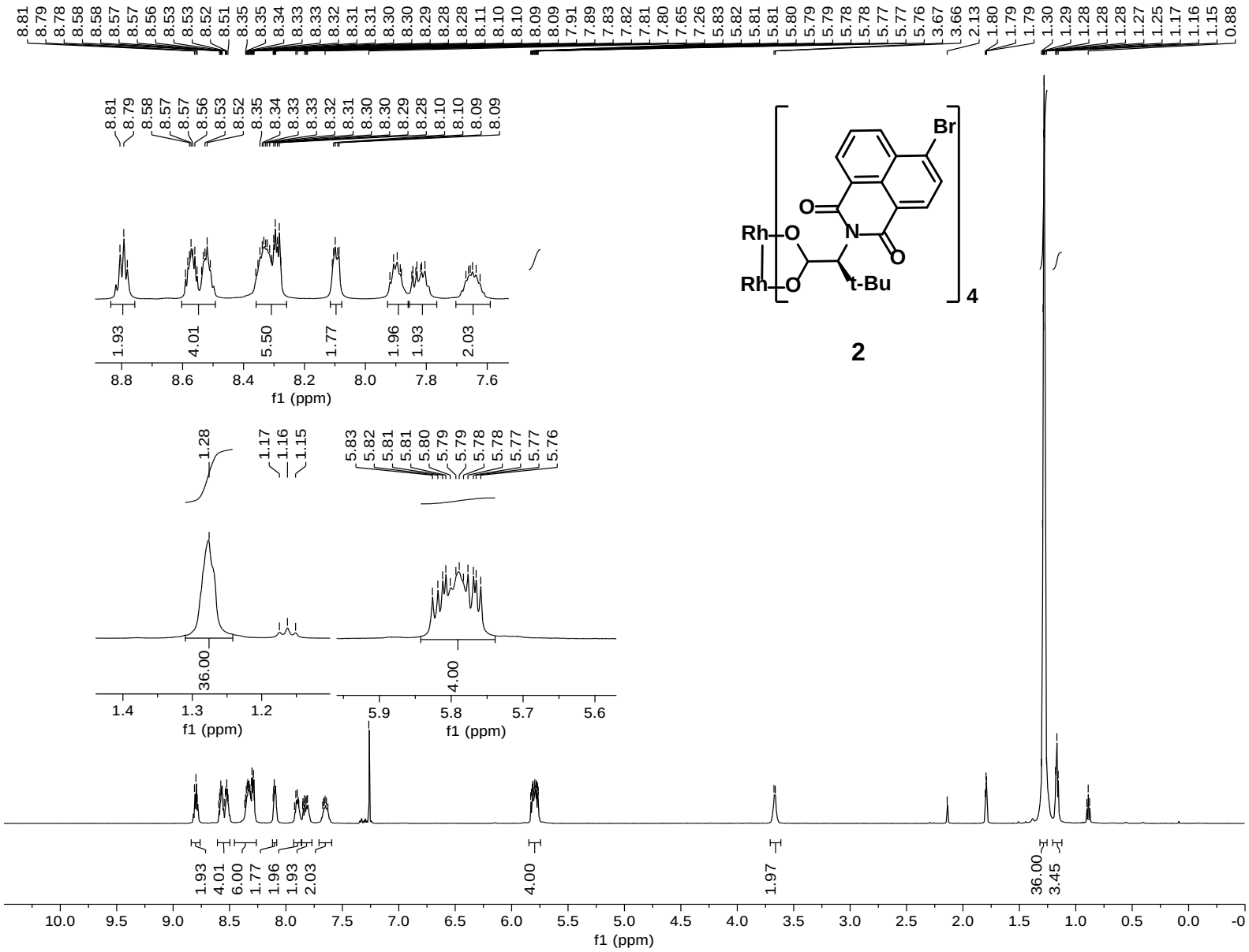
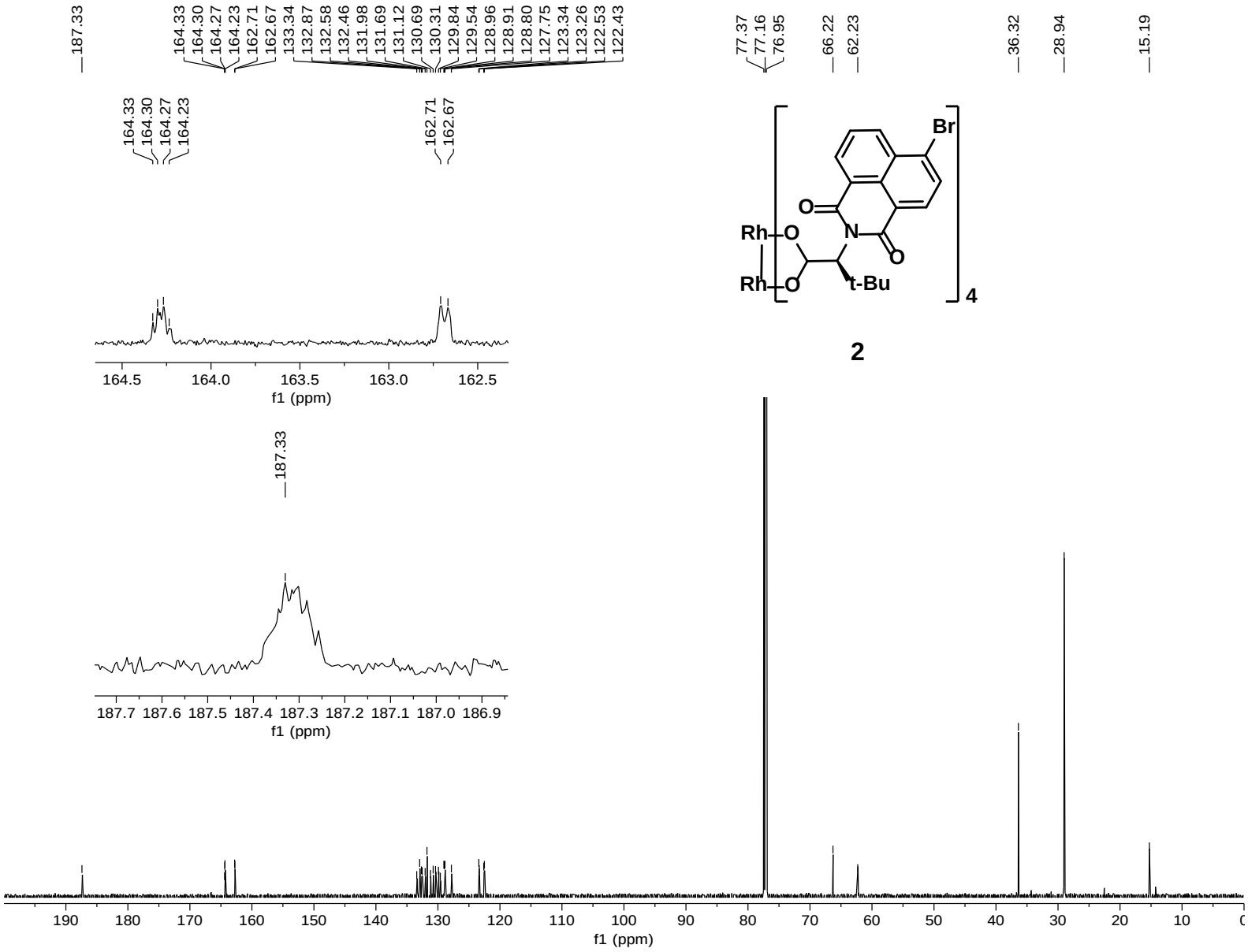


Dirhodium (II) tetrakis[*N*-4-bromo-1,8-naphthoyl-(*S*)-tert-leucinate] (^{1}H NMR, 600 MHz, $CDCl_3$, 298 K)



Dirhodium (II) tetrakis[*N*-4-bromo-1,8-naphthoyl-(*S*)-tert-leucinate] (^{13}C NMR, 151 MHz, $CDCl_3$, 298 K)



Dirhodium (II) tetrakis[*N*-4-bromo-1,8-naphthoyl-(*S*)-tert-leucinate] (^{13}C NMR, 151 MHz, $CDCl_3$, 298 K)

133.38
133.35
133.34
133.32
133.27
132.87
132.58
132.46
131.98
131.69
131.12
130.69
130.31
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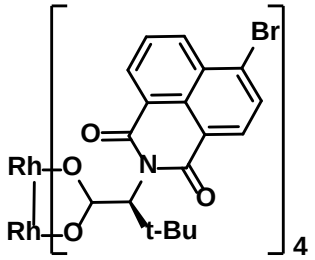
122.43



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