

An Accelerated Intermolecular Rauhut–Currier Reaction Enables the Total Synthesis of (–)-Flueggenine C

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1. General Procedures

All reactions were performed in oven-dried or flame-dried round-bottomed flasks. Unless otherwise noted, the flasks were fitted with rubber septa and reactions were conducted under a positive pressure of argon. Stainless steel syringes or cannula were used to transfer air- and moisture-sensitive liquids. Flash column chromatography was performed as described by Still et al. using silica gel (60-Å pore size, 40–63 µm, 4-6% H₂O content, Merck).¹ Analytical thin-layer chromatography (TLC) was performed using glass plates pre-coated with 0.25 mm silica gel impregnated with a fluorescent indicator (254 nm). Thin layer chromatography plates were visualized by exposure to ultraviolet light, an aqueous solution of ceric ammonium molybdate (CAM) and/or a potassium permanganate (KMnO₄).

2. Materials and Instrumentations

Unless otherwise stated, all commercial reagents and solvents were used without additional purification with the following exceptions: dichloromethane and tetrahydrofuran were purchased from Merck and Daejung Inc., respectively and were purified by the method of Grubbs et al. under positive argon pressure.²

Proton and carbon nuclear magnetic resonance spectra were recorded with Bruker Ascend 400 (400 MHz), Bruker Avance III HD Nano bay (400 MHz), Agilent Technologies DD2 (600 MHz), Bruker AVANCE III HD (800MHz), or Bruker AVANCE III HD (900MHz) with ¹H-(¹³C/¹⁵N)Z-G cryogenic probe spectrometers. Proton nuclear magnetic resonance spectra are referenced from the residual protium in the NMR solvent (CDCl₃: δ 7.24 (CHCl₃)). Data are reported in the following manners: chemical shift in ppm [multiplicity (s = singlet, d = doublet, m = multiplet, app = apparent, br = broad), coupling constant(s) in Hertz, integration]. Carbon-13 nuclear magnetic resonance spectra are referenced from the carbon resonances of

the solvent (CDCl_3 : δ 77.23, CD_3OD : δ 49.15). Data are reported in the following manners: chemical shift in ppm. 800MHz and 900MHz NMR experiments were operated by E. H. Kim at Korea Basic Science Institute. High resolution mass spectra were obtained from KAIST Research Analysis Center (Daejeon) by using ESI method. Chiral HPLC analysis was performed on an Agilent Technologies 1100 Series system. Specific rotation $[\alpha]_D$ was obtained by JASCO P-1020 polarimeter.

3. Comprehensive List of Dimeric and Oligomeric Securinega Alkaloids

Figure S1. Structure of known dimeric securinega alkaloids.³

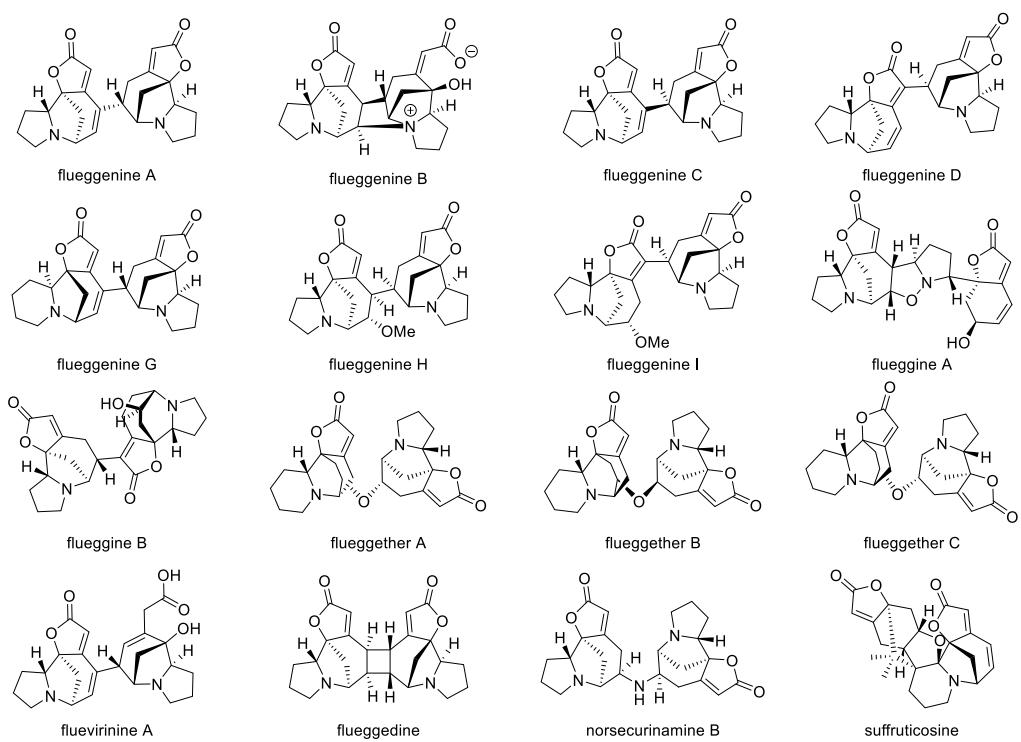


Figure S2. Structure of known trimeric securinega alkaloids.^{3,4}

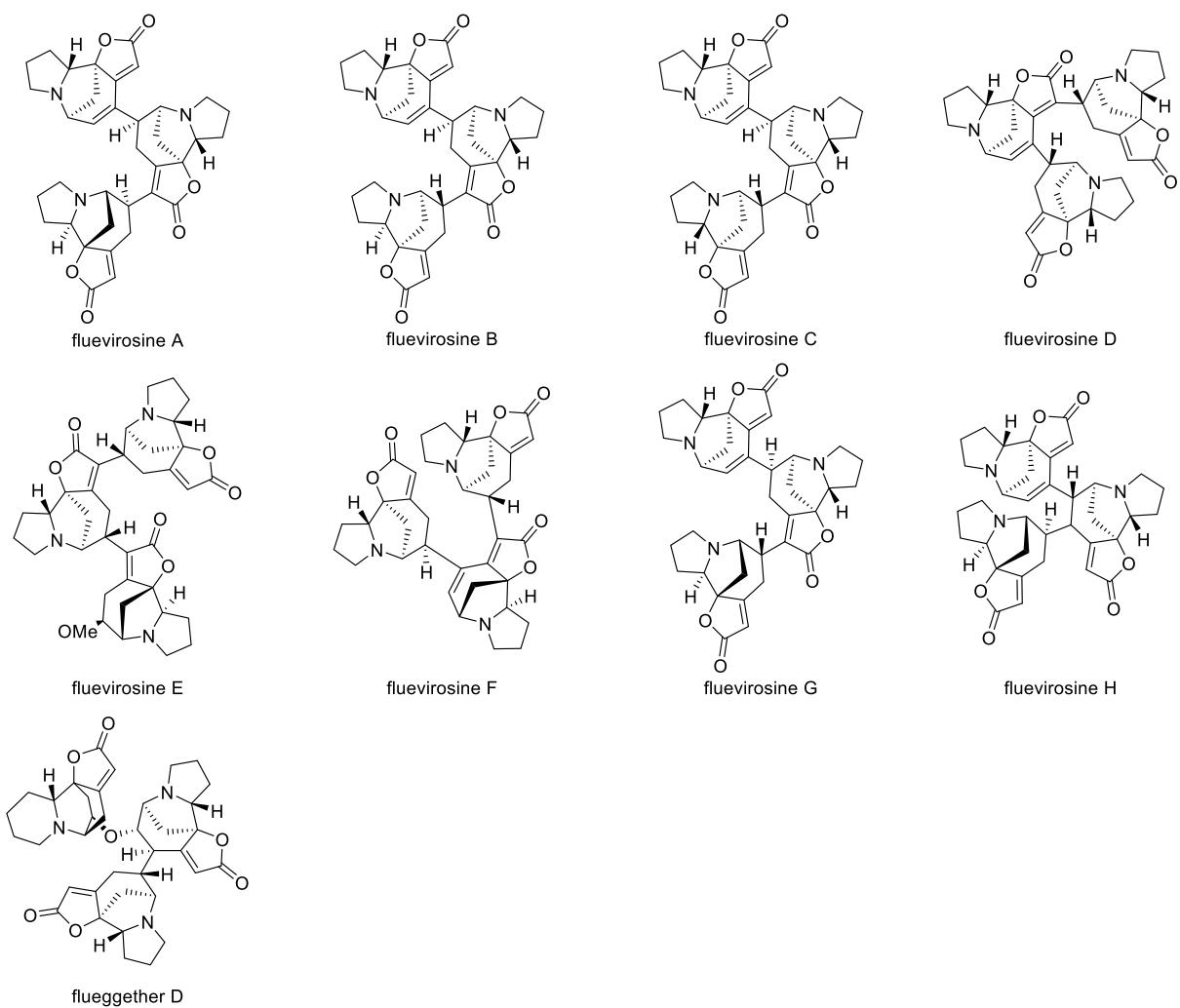
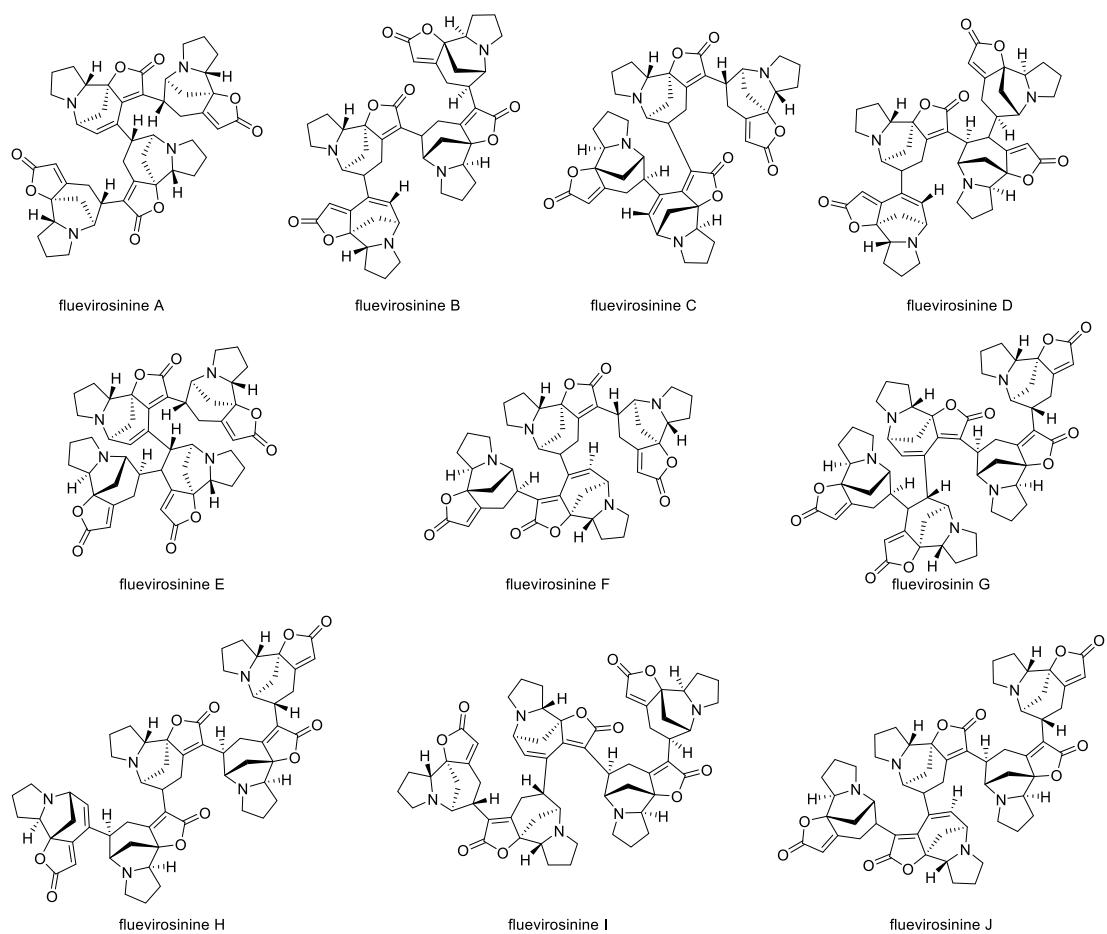
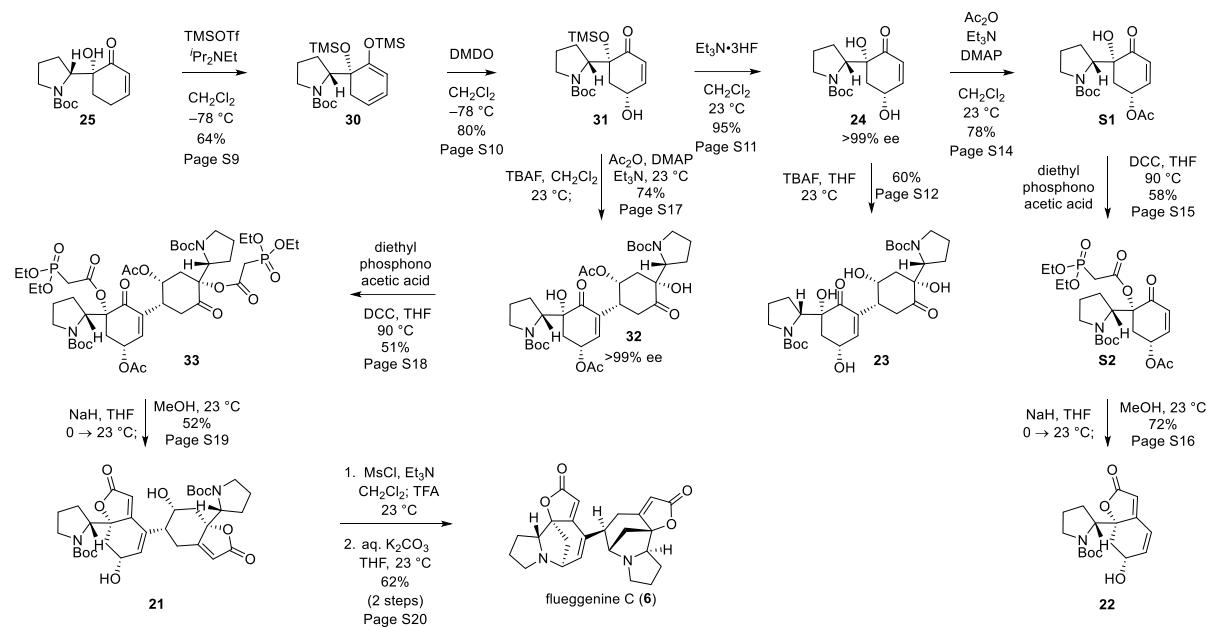


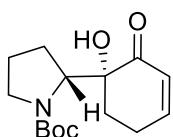
Figure S3. Structure of known tetrameric and pentameric securinega alkaloids.^{3,4}



4. Experimental Procedures and Physical Data for Newly Synthesized Compounds

Scheme S1. An overview of the synthetic pathways





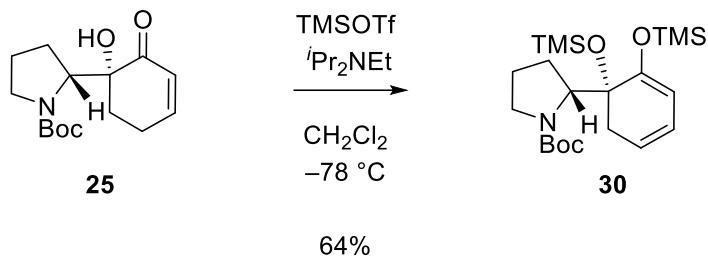
25

Table S1. Comparison of ^1H NMR spectroscopic data of compound 25

Jeon, S.; Han, S. ⁵ δ_1 (ppm ; multi, <i>J</i> in Hz)	Jiang, S. et al. ⁶ δ_2 (ppm ; multi, <i>J</i> in Hz)	deviation $\Delta\delta = \delta_1 - \delta_2$ (ppm)
6.99 – 6.93 (m, 1H)	7.02 – 6.99 (m, 1H)	-0.04
6.01 (d, 9.9, 1, 1H)	6.06 (d, 9.6, 1H)	-0.05
4.21 (s, 1H)	4.26 (s, 1H)	-0.06
3.96 (s, 1H)	4.00 (s, 1H)	-0.04
3.54 (brs, 1H)	3.60 (brs, 1H)	-0.06
3.36 – 3.24 (m, 2H)	3.37 – 3.32 (m, 2H)	-0.04
2.39 – 2.25 (m, 2H)	2.45 – 2.34 (m, 2H)	-0.07
1.97 – 1.88 (m, 1H)	1.98 – 1.96 (m, 1H)	-0.04
1.81 – 1.74 (m, 1H)	1.87 – 1.83 (m, 1H)	-0.07
1.67 (brs, 1H)	1.70 (brs, 1H)	-0.03
1.41 (s, 9H)	1.46 (s, 9H)	-0.05

Table S2. Comparison of ^{13}C NMR spectroscopic data of compound 25

Jeon, S.; Han, S. ⁷ δ_1 (ppm)	Jiang, S. et al. ⁶ δ_2 (ppm)	deviation $\Delta\delta = \delta_1 - \delta_2$ (ppm)
202.8	201.3	1.5
157.7	156.2	1.5
153.8	152.1	1.7
128.2	126.7	1.5
80.8	79.4	1.4
80.5	78.9	1.6
60.2	58.9	1.3
49.2	47.7	1.5
34.2	32.5	1.7
28.9 (3)	27.3	1.6
26.7	25.3	1.4
26.5	24.9	1.6
25.6	24.0	1.6



Tert-butyl (R)-2-((R)-1,2-bis(trimethylsilyl)oxy)cyclohexa-2,4-dien-1-yl)pyrroledine-1-carboxylate 30:

To a stirred solution of **25**⁶ (336 mg, 1.19 mmol, 1 equiv) and *N,N*-diisopropylethyl amine (1.072 mL, 5.97 mmol, 5.00 equiv) in CH₂Cl₂ (11 mL) at –78 °C was added trimethylsilyl trifluoromethanesulfonate (552 μL, 2.98 mmol, 2.50 equiv) dropwise under argon. After 1 h, saturated aqueous sodium bicarbonate solution (20 mL) was added to the reaction mixture, and the layers were separated. The aqueous layer was extracted with dichloromethane (3 × 20 mL), and the combined organic layers were dried over anhydrous sodium sulfate and concentrated under reduced pressure. The resulting crude residue was purified by flash column chromatography (silica gel: diam. 5cm, ht. 10 cm; eluent: ethyl acetate : hexanes = 1 : 20) to afford **30** (325 mg, 64%) as a colorless oil.

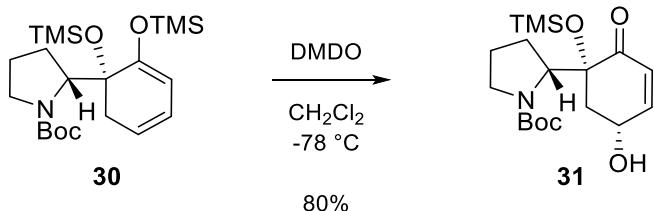
¹H NMR (400.1 MHz, CDCl₃): δ 5.69 (s, 1H), 5.53 (app d, 1H), 4.98 (app d, 1H), 4.10 (d, *J* = 40.7 Hz, 1H), 3.57 (app d, 1H), 3.24–3.12 (m, 1H), 2.51 (dd, *J* = 17.0, 5.4 Hz, 1H), 2.31 (d, *J* = 17.2 Hz, 1H), 2.16 (t, *J* = 9.1 Hz, 1H), 2.04–1.89 (m, 1H), 1.72–1.53 (m, 2H), 1.43 (s, 9H), 0.23 (s, 9H), 0.05 (s, 9H) (Mixture of rotamers).

¹³C NMR (100.6 MHz, CDCl₃): δ 155.9, 154.8, 122.7, 121.0, 101.7, 101.2, 79.5, 78.8, 61.2, 60.8, 47.4, 46.9, 36.8, 28.7 (3), 27.0, 26.6, 24.7, 23.7, 2.2 (3), 0.2 (3) (Mixture of rotamers).

HRMS (ESI): Calculated for C₂₁H₃₉NO₄Si₂ [M+Na]⁺: 448.2310, found: 448.2317.

[α]²⁰D: 105.5° (*c* = 1.0 in CH₂Cl₂).

TLC (ethyl acetate : hexanes = 1 : 20) *Rf*: 0.35 (CAM, KMnO₄, UV).



Tert-butyl (R)-2-((1*R*,5*R*)-5-hydroxy-2-oxo-1-((trimethylsilyl)oxy)cyclohex-3-en-1-yl)pyrrolidine-1-carboxylate 31:

To a stirred solution of **30** (404 mg, 0.950 mmol, 1 equiv) in CH₂Cl₂ (5 mL) at -78 °C was added freshly prepared 57.5 mM dimethyldioxirane solution⁸ (15.6 mL, 0.90 mmol, 0.95 equiv) in acetone under argon. After 30 min, the reaction mixture was concentrated under reduced pressure. The resulting crude residue was purified by flash column chromatography (silica gel: diam. 2.5cm, ht. 10 cm; eluent: ethyl acetate : hexanes = 1 : 2) to afford **31** (280 mg, 80%) as a colorless oil.

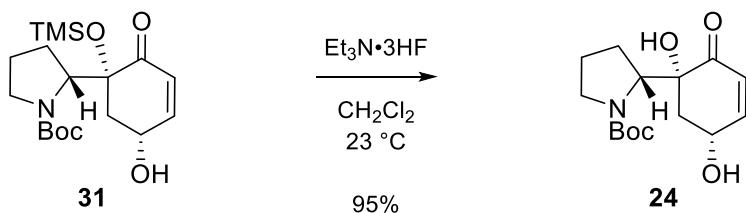
¹H NMR (400.1 MHz, CDCl₃): δ 6.84 (dt, *J* = 10.3, 2.0 Hz, 1H), 5.94 (dd, *J* = 10.4, 2.4 Hz, 1H), 5.42 (s, 1H), 4.02 (d, *J* = 7.7 Hz, 1H), 3.55 (d, *J* = 8.5 Hz, 1H), 3.31 (d, *J* = 6.1 Hz, 1H), 3.23 (ddd, *J* = 10.7, 8.9, 5.4 Hz, 1H), 2.71 – 2.53 (m, 1H), 2.07 – 1.78 (m, 3H), 1.74 – 1.48 (m, 2H), 1.44 (s, 9H), 0.07 (s, 9H).

^{13}C NMR (100.6 MHz, CDCl_3): δ 202.3, 156.3, 153.2, 127.7, 84.0, 79.8, 66.2, 60.1, 48.2, 45.0, 28.6 (3), 25.5, 24.6, 2.3 (3).

HRMS (ESI): Calculated for C₁₈H₃₁NO₅Si [M+Na]⁺: 392.1864, found: 392.1849.

$[\alpha]^{20}_{\text{D}} : 47.8^\circ$ ($c = 1.0$ in CH_2Cl_2).

TLC (ethyl acetate : hexanes = 1 : 3) R_f 0.24 (CAM, KMnO₄, UV).



Tert-butyl (R)-2-((1*R*,5*S*)-1,5-dihydroxy-2-oxocyclohex-3-en-1-yl)pyrrolidine-1-carboxylate 24:

To a stirred solution of **31** (160 mg, 0.43 mmol, 1 equiv) in CH₂Cl₂ (5 mL) at 23 °C was added triethylamine trihydrofluoride (705 µL, 4.32 mmol, 10.0 equiv) under argon. After 7 h, saturated aqueous sodium bicarbonate solution (10 mL) was added to the reaction mixture, and the layers were separated. The aqueous layer was extracted with dichloromethane (3 × 10 mL), and the combined organic layers were dried over anhydrous sodium sulfate and concentrated under reduced pressure. The resulting crude residue was purified by flash column chromatography (silica gel: diam. 2.5 cm, ht. 10 cm; eluent: ethyl acetate : hexanes = 1 : 1) to afford **24** (121 mg, 95%) as a colorless oil.

α',γ -dihydroxylated enone **24** was found to be higher than 99% ee by chiral HPLC analysis [CHIRALPAK IC 1 mL/min; 80% hexanes and 20% ethanol over 20 min; *t*_R = 8.3 min].

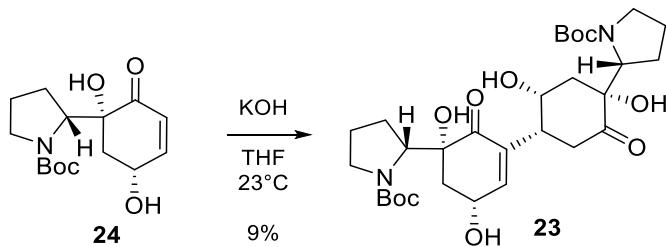
¹H NMR (400.1 MHz, CDCl₃): δ 6.91 (d, *J* = 10.2 Hz, 1H), 6.01 (d, *J* = 9.9 Hz, 1H), 5.17 (s, 1H), 4.38 (s, 1H), 4.00 (s, 1H), 3.54 (s, 1H), 3.44 – 3.26 (m, 2H), 2.60 (d, *J* = 8.5 Hz, 1H), 1.96–1.84 (m, 1H), 1.79 (dd, *J* = 12.9, 9.0 Hz, 1H), 1.74 – 1.58 (m, 3H), 1.40 (s, 9H).

¹³C NMR (100.6 MHz, CDCl₃): δ 200.9, 156.8, 153.7, 126.6, 80.3, 79.9, 65.6, 60.5, 48.2, 43.2, 28.6 (3), 26.2, 24.8.

HRMS (ESI): Calculated for C₁₅H₂₃NO₅ [M+Na]⁺: 320.1468, found: 320.1445.

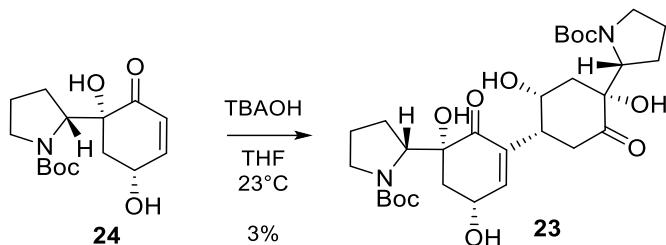
[*α*]²⁰_D: 47.4° (*c* = 0.3 in CH₂Cl₂).

TLC (ethyl acetate : hexanes = 1 : 1) *R*_f: 0.28 (CAM, KMnO₄, UV).



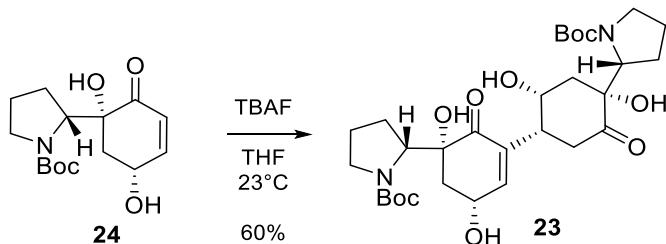
Di-*tert*-butyl 2,2'-(*(1'R,2'R,3R,4'R,5R)-2',3,4',5-tetrahydroxy-2,5'-dioxo-[1,1'-bi(cyclohexan)]-6-ene-3,4'-diyl*)(2*R,2'R*)-bis(pyrrolidine-1-carboxylate) 23:

To a solution of **24** (33 mg, 0.11 mmol, 1 equiv) in THF (1 mL) at 23 °C was added 2.0 M aqueous potassium hydroxide solution (55 µL, 0.11 mmol, 1.00 equiv) under argon. After 12 h, the reaction mixture was concentrated under reduced pressure. The resulting crude residue was purified by flash column chromatography (silica gel: diam. 2.5 cm, ht. 10 cm; eluent: ethyl acetate : hexanes = 2 : 1) to afford **23** (3 mg, 9%) as a colorless oil.



Di-*tert*-butyl 2,2'-(*(1'R,2'R,3R,4'R,5R)-2',3,4',5-tetrahydroxy-2,5'-dioxo-[1,1'-bi(cyclohexan)]-6-ene-3,4'-diyl*)(2*R,2'R*)-bis(pyrrolidine-1-carboxylate) 23:

To a solution of **24** (38 mg, 0.13 mmol, 1 equiv) in THF (1 mL) at 23 °C was added tetra-*n*-butylammonium hydroxide 40 wt% solution in H₂O (83 µL, 0.13 mmol, 1.00 equiv) under argon. After 1 h, the reaction mixture was concentrated under reduced pressure. The resulting crude residue was purified by flash column chromatography (silica gel: diam. 2.5 cm, ht. 10 cm; eluent: ethyl acetate : hexanes = 2 : 1) to afford **23** (1 mg, 3%) as a colorless oil.



Di-*tert*-butyl 2,2'-(*(1'R,2'R,3R,4'R,5R)-2',3,4',5-tetrahydroxy-2,5'-dioxo-[1,1'-bi(cyclohexan)]-6-ene-3,4'-diyl*)(2*R,2'R*)-bis(pyrrolidine-1-carboxylate) 23:

To a solution of **24** (25 mg, 0.08 mmol, 1 equiv) in THF (1 mL) at 23 °C was added tetra-*n*-butylammonium fluoride 1.0 M solution in THF (84 µL, 0.08 mmol, 1.00 equiv) under

argon. After 3 h, the reaction mixture was concentrated under reduced pressure. The resulting crude residue was purified by flash column chromatography (silica gel: diam. 2.5 cm, ht. 10 cm; eluent: ethyl acetate : hexanes = 2 : 1) to afford **23** (15 mg, 60%) as a colorless oil.

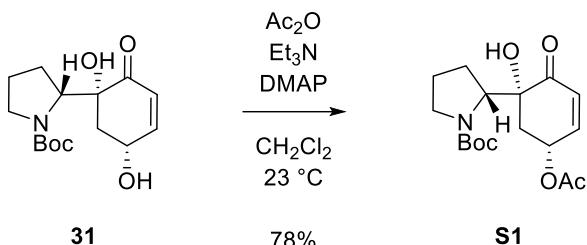
¹H NMR (400.1 MHz, CDCl₃ with 1% CD₃OD): δ 6.76 (s, 1H), 4.96 (s, 1H), 4.10 (s, 1H), 3.90 (dd, *J* = 7.1, 3.9 Hz, 2H), 3.69 (t, *J* = 9.3 Hz, 1H), 3.60 – 3.36 (m, 3H), 3.30 (ddd, *J* = 11.1, 8.0, 4.6 Hz, 1H), 3.01 (d, *J* = 14.4 Hz, 1H), 2.73 (d, *J* = 13.2 Hz, 1H), 2.23 (d, *J* = 14.3 Hz, 1H), 2.04 – 1.84 (m, 5H), 1.80 – 1.59 (m, 6H), 1.42 (s, 9H), 1.41 (s, 9H).

¹³C NMR (100.6 MHz, CDCl₃): δ 209.0, 201.3, 158.0, 156.5, 150.3, 135.6, 81.3, 81.2, 80.1, 79.6, 68.4, 64.7, 63.2, 61.2, 48.4, 48.3, 43.5, 42.7, 41.5, 37.3, 28.7, 28.6 (3), 28.6 (3), 26.1, 24.9, 24.6.

HRMS (ESI): Calculated for C₃₀H₄₆N₂O₁₀ [M+Na]⁺: 617.3045, found: 617.3060.

TLC (ethyl acetate : hexanes = 3 : 1) *Rf*: 0.20 (CAM, KMnO₄, UV).

[*α*]²⁰_D: -0.5° (*c* = 1.0 in CH₂Cl₂).



Tert-butyl (R)-2-((1*R*,5*R*)-5-acetoxy-1-hydroxy-2-oxocyclohex-3-en-1-yl)pyrrolidine-1-carboxylate S1:

To a stirred solution of **31** (352 mg, 1.18 mmol, 1 equiv) in CH_2Cl_2 (10 mL) at 23 °C was added triethylamine (985 μL , 7.10 mmol, 6.00 equiv), acetic anhydride (336 μL , 3.55 mmol, 3.00 equiv), and 4-dimethylaminopyridine (144 mg, 1.18 mmol, 1.00 equiv). After 1 h, saturated aqueous ammonium chloride solution (10 mL) was added and the layers were separated. The aqueous layer was extracted with dichloromethane (3×10 mL), and the combined organic layers were dried over anhydrous sodium sulfate and concentrated under reduced pressure. The resulting crude residue was purified by column chromatography (silica gel: diam. 4 cm, ht. 8 cm; eluent: ethyl acetate : hexanes = 3 : 7) to afford **S1** (314 mg, 78%) as a colorless oil.

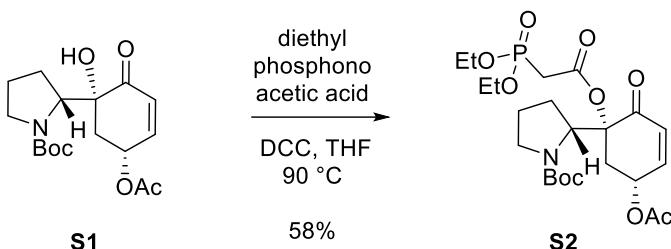
$^1\text{H NMR}$ (400.1 MHz, CDCl_3): δ 6.79 – 6.68 (m, 1H), 6.16 (dd, J = 10.4, 2.3 Hz, 1H), 6.11 – 5.57 (m, 1H), 4.25 – 3.75 (m, 2H), 3.65 – 3.25 (m, 2H), 2.85 – 2.68 (m, 1H), 2.04 (s, 3H), 2.02 (d, J = 2.3 Hz, 1H), 1.93 – 1.71 (m, 4H), 1.43 – 1.37 (m, 9H) (Mixture of rotamers).

$^{13}\text{C NMR}$ (100.6 MHz, CDCl_3): δ 201.0, 193.3, 170.2, 169.1, 156.1, 148.2, 129.1, 80.1, 78.7, 68.1, 61.8, 48.2, 39.0, 28.5, 26.0, 24.7, 21.7, 21.2 (Mixture of rotamers).

HRMS (ESI): Calculated for $\text{C}_{17}\text{H}_{25}\text{NO}_6$ $[\text{M}+\text{Na}]^+$: 362.1574, found: 344.1595.

$[\alpha]^{20}\text{D}$: 40.1° (c = 1.0 in CH_2Cl_2).

TLC (ethyl acetate : hexanes = 1 : 3) R_f : 0.23 (CAM, KMnO_4 , UV).



Tert-butyl (R)-2-((1R,5R)-5-acetoxy-1-(2-(diethoxyphosphoryl)acetoxy)-2-oxocyclohex-3-en-1-yl)pyrrolidine-1-carboxylate S2:

To a stirred solution of **S1** (314 mg, 0.93 mmol, 1 equiv) in THF (6 mL) at 90 °C was added a solution of *N,N'*-dicyclohexylcarbodiimide (1.91 g, 9.26 mmol, 10.0 equiv) in THF (4 mL) under argon. After 5 min, a solution of diethylphosphonoacetic acid (744 µL, 4.63 mmol, 5.00 equiv) in THF (4 mL) was added slowly via syringe pump over 1 h. After 30 min, the reaction mixture was filtered and concentrated under reduced pressure. The resulting crude residue was purified by column chromatography (silica gel: diam. 4 cm, ht. 8 cm; eluent: ethyl acetate : hexanes = 1 : 1 → 2 : 1) to afford **S2** (277 mg, 58%) as a colorless oil.

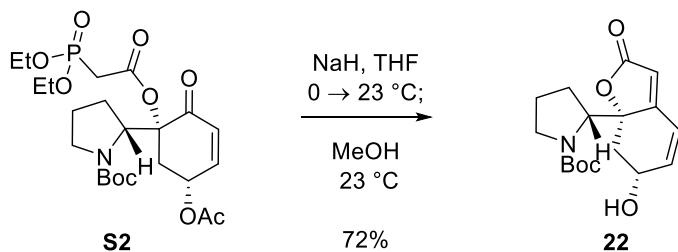
¹H NMR (400.1 MHz, CDCl₃): δ 6.80 (dt, *J* = 10.4, 1.9 Hz, 1H), 6.27 (s, 1H), 6.13 (dd, *J* = 10.4, 2.2 Hz, 1H), 4.33 – 4.25 (m, 1H), 4.23 – 4.15 (m, 4H), 3.80 – 3.60 (m, 1H), 3.40 (ddd, *J* = 11.0, 8.3, 5.1 Hz, 1H), 3.07 – 2.80 (m, 3H), 2.64 (dd, *J* = 12.5, 6.4 Hz, 1H), 2.29 – 2.18 (m, 1H), 2.15 – 2.05 (m, 4H), 1.90 – 1.75 (m, 2H), 1.46 (s, 9H), 1.36 (td, *J* = 7.1, 2.8 Hz, 6H) (Mixture of rotamers).

¹³C NMR (150.7 MHz, CDCl₃): δ 192.6, 170.0, 163.9, 156.1, 147.8, 129.5, 85.9, 80.5, 68.5, 62.9 (2), 60.1, 48.3, 35.6, 34.7, 34.2, 28.5 (3), 25.3, 24.8, 21.1 (2), 16.5 (Mixture of rotamers).

HRMS (ESI): Calculated for C₂₃H₃₆NO₁₀P [M+Na]⁺: 540.1969, found: 540.2020.

[α]²⁰D : 26.7° (*c* = 1.0 in CH₂Cl₂).

TLC (ethyl acetate : hexanes = 5 : 1) *Rf*: 0.35 (CAM, KMnO₄, UV).



Tert-butyl (R)-2-((6R,7aS)-6-hydroxy-2-oxo-6,7-dihydrobenzofuran-7a(2H)-yl)pyrrolidine-1-carboxylate 22:

To a stirred solution of **S2** (277 mg, 0.53 mmol, 1 equiv) in THF (10 mL) at 0 °C was added sodium hydride (60% dispersion in mineral oil, 65 mg, 1.60 mmol, 3.00 equiv) under argon and the reaction mixture was slowly warmed to 23 °C. After 30 min, MeOH (10 mL) was added to the reaction mixture. After 30 min, brine (20 mL) and ethyl acetate (20 mL) was added to the reaction mixture and the layers were separated. The aqueous layer was extracted with ethyl acetate (3 × 20 mL). The combined organic layers were dried over anhydrous sodium sulfate and concentrated under reduced pressure. The resulting crude residue was purified by flash column chromatography (silica gel: diam. 4 cm, ht. 10 cm; eluent: ethyl acetate : hexanes = 1 : 1) to afford **22** (124 mg, 72%) as a white crystalline solid.

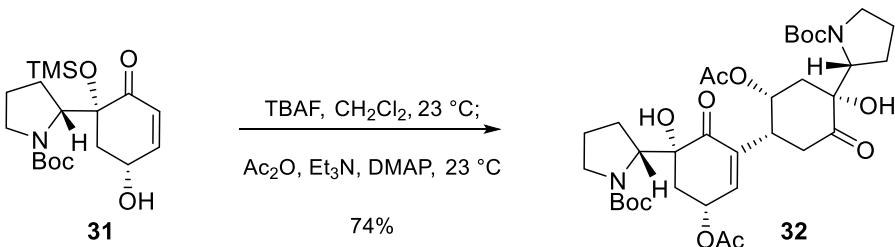
¹H NMR (400.1 MHz, CDCl₃): δ 6.50 (dd, *J* = 10.1, 2.3 Hz, 1H), 6.25 (ddd, *J* = 10.2, 2.5, 1.2 Hz, 1H), 5.82 (s, 1H), 5.30 (s, 1H), 4.16 – 4.02 (m, 1H), 3.56 (dd, *J* = 8.9, 8.3 Hz, 1H), 3.37 (ddd, *J* = 11.0, 8.8, 5.0 Hz, 1H), 3.03 – 2.76 (m, 2H), 2.01 – 1.52 (m, 5H), 1.44 (s, 9H).

¹³C NMR (100.6 MHz, CDCl₃): δ 172.6, 165.7, 156.5, 143.1, 120.0, 112.7, 90.4, 80.3, 66.3, 59.4, 47.5, 41.8, 28.6 (3), 25.3, 24.8.

HRMS (ESI): Calculated for C₁₇H₂₃NO₅ [M+Na]⁺: 344.1468, found: 344.1477.

[*a*]²⁰_D: 41.4° (*c* = 1.0 in CH₂Cl₂).

TLC (ethyl acetate : hexanes = 5 : 1) *Rf*: 0.6 (CAM, KMnO₄, UV).



Di-tert-butyl 2,2'-(*(1'R,2'R,3R,4'R,5R)-2',5-diacetoxy-3,4'-dihydroxy-2,5'-dioxo-[1,1'-bi(cyclohexan)]-6-ene-3,4'-diyl*)(2R,2'R)-bis(pyrrolidine-1-carboxylate) 32:

To a stirred solution of **31** (279 mg, 0.75 mmol, 1 equiv) in CH₂Cl₂ (7.5 mL) at 23 °C was added 1 M tetra-*n*-butylammonium fluoride solution in THF (755 µL, 0.75 mmol, 1.00 equiv) under argon. After 3 h, triethylamine (627 µL, 4.53 mmol, 6.00 equiv), acetic anhydride (214 µL, 2.26 mmol, 3.00 equiv), and 4-dimethylaminopyridine (92 mg, 0.75 mmol, 1.00 equiv) was added to the reaction mixture. After 1 h, saturated aqueous sodium bicarbonate solution (10 mL) was added to the reaction mixture, and the layers were separated. The aqueous layer was extracted with dichloromethane (3 × 20 mL), and the combined organic layers were dried over anhydrous sodium sulfate and concentrated under reduced pressure. The resulting crude residue was purified by flash column chromatography (silica gel: diam. 2.5cm, ht. 11 cm; eluent: ethyl acetate : hexanes = 1 : 1) to afford **32** (190 mg, 74%) as a colorless oil.

Acetylated dimer **32** was found to be higher than 99% ee by chiral HPLC analysis [CHIRALPAK IC 1 mL/min; 80% hexanes and 20% ethanol over 15 min; $t_R = 10.2$ min].

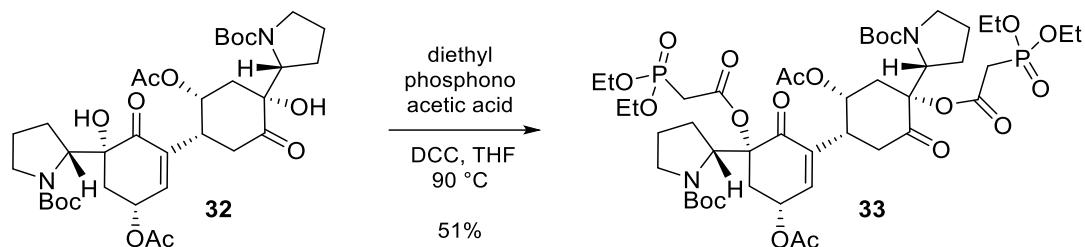
¹H NMR (400.1 MHz, CDCl₃): δ 6.45 (s, 1H), 6.00 (s, 1H), 5.52 (s, 1H), 5.42 (s, 1H), 4.08 (s, 1H), 3.88 (d, *J* = 5.4 Hz, 1H), 3.79 (s, 1H), 3.65 (s, 1H), 3.46 (s, 1H), 3.41 – 3.28 (m, 3H), 3.28 – 3.16 (m, 1H), 2.70 (dd, *J* = 12.9, 5.0 Hz, 1H), 2.28 – 2.11 (m, 3H), 2.04 (s, 3H), 2.01 (s, 3H), 1.98 – 1.54 (m, 9H), 1.42 (s, 9H), 1.40 (s, 9H).

¹³C NMR (150.7 MHz, CDCl₃): δ 209.4, 200.4, 170.1, 169.9, 157.4, 156.3, 144.4, 137.8, 80.6, 80.3, 79.8, 78.5, 69.4, 67.4, 62.5 (2), 48.5, 48.3, 48.2, 40.7, 39.4, 38.3, 37.3, 28.7 (3), 28.6 (3), 28.1, 26.2, 24.8, 21.2 (2).

HRMS (ESI): Calculated for $C_{34}H_{50}N_2O_{12}$ $[M+Na]^+$: 701.3256, found: 701.3255.

$[\alpha]^{20}_{D}: 4.5^\circ$ ($c = 0.5$ in CH_2Cl_2).

TLC (ethyl acetate : hexanes = 1 : 1) R_f: 0.25 (CAM, KMnO₄, UV).



Di-*tert*-butyl 2,2'-(1'R,2'R,3R,4'R,5R)-2',5-diacetoxy-3,4'-bis(2-(diethoxyphosphoryl)acetoxy)-2,5'-dioxo-[1,1'-bi(cyclohexan)]-6-ene-3,4'-diyl)(2R,2'R)-bis(pyrrolidine-1-carboxylate) 33:

To a stirred solution of **32** (80 mg, 0.12 mmol, 1 equiv) in THF (2 mL) at 90 °C was added a solution of *N,N'*-dicyclohexylcarbodiimide (486 mg, 2.4 mmol, 20.0 equiv) in THF (2 mL) under argon. After 5 min, a solution of diethylphosphonoacetic acid (190 µL, 1.2 mmol, 10.0 equiv) in THF (1 mL) was added slowly via syringe pump over 2 h. After 1 h, the reaction mixture was filtered and concentrated under reduced pressure. The resulting crude residue was purified by column chromatography (silica gel: diam. 2.5 cm, ht. 10 cm; eluent: ethyl acetate : hexanes = 5 : 1 → 40 : 1) to afford **33** (62 mg, 51%) as a colorless oil.

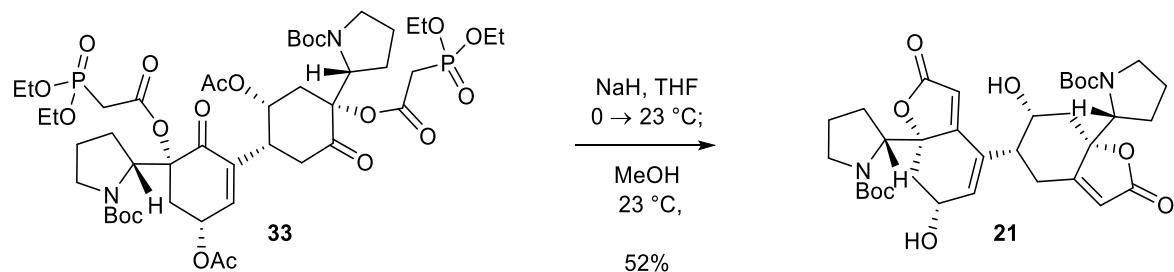
¹H NMR (400.1 MHz, CDCl₃): δ 6.74 (s, 1H), 6.27 (s, 1H), 5.57 (s, 1H), 4.49 (s, 1H), 4.21 – 4.10 (m, 9H), 3.82 – 3.54 (m, 3H), 3.39 – 3.27 (m, 2H), 3.00 – 2.76 (m, 5H), 2.72 – 2.62 (m, 2H), 2.58 – 2.46 (m, 2H), 2.16 – 1.89 (m, 10H), 1.86 – 1.57 (m, 5H), 1.43 (s, 9H), 1.41 (s, 9H), 1.32 (td, *J* = 7.1, 3.1 Hz, 12H) (Mixture of rotamers).

¹³C NMR (201.2 MHz, CDCl₃): δ 203.2, 191.9, 170.4, 170.0, 163.8, 163.6, 156.4, 156.2, 145.4, 137.6, 88.2, 86.1, 80.5, 80.3, 68.8, 67.5, 63.0 (2), 62.9 (3), 60.8, 60.3, 48.2, 48.1, 40.2, 36.1, 35.6, 35.5, 34.9, 34.8, 34.0, 33.7, 28.5 (6), 25.8, 25.5, 24.9 (2), 21.2 (2), 16.6 (4) (Mixture of rotamers).

HRMS (ESI): Calculated for C₄₆H₇₂N₂O₂₀P₂ [M+Na]⁺: 1057.4046, found: 1057.4046.

[*a*]²⁰_D: 37.4° (*c* = 0.3 in CH₂Cl₂).

TLC (ethyl acetate = 1) **R_f**: 0.1 (CAM, KMnO₄, UV).



Di-*tert*-butyl 2,2'-(*(5'R,6R,6'R,7aS,7'aS)-6,6'-dihydroxy-2,2'-dioxo-4',5',6,6',7,7'-hexahydro [4,5'-bibenzofuran]-7a,7'a(2H,2'H)-diyl*)(2*R,2'R*)-bis(pyrrolidine-1-carboxylate) **21:**

To a stirred solution of **33** (62 mg, 0.06 mmol, 1 equiv) in THF (3 mL) at 0 °C was added sodium hydride (60% dispersion in mineral oil, 24 mg, 0.60 mmol, 10.0 equiv) under argon and the reaction mixture was slowly warmed to 23 °C. After 1 h, MeOH (3 mL) was added to the reaction mixture. After 30 min, brine (10 mL) and ethyl acetate (10 mL) were added to the reaction mixture and the layers were separated. The aqueous layer was extracted with ethyl acetate (3 × 10 mL), and the combined organic layers were dried over anhydrous sodium sulfate and concentrated under reduced pressure. The resulting crude residue was purified by flash column chromatography (silica gel: diam. 2.5 cm, ht. 10 cm; eluent: ethyl acetate : hexanes = 3 : 1) to afford **21** (20 mg, 52%) as a colorless oil.

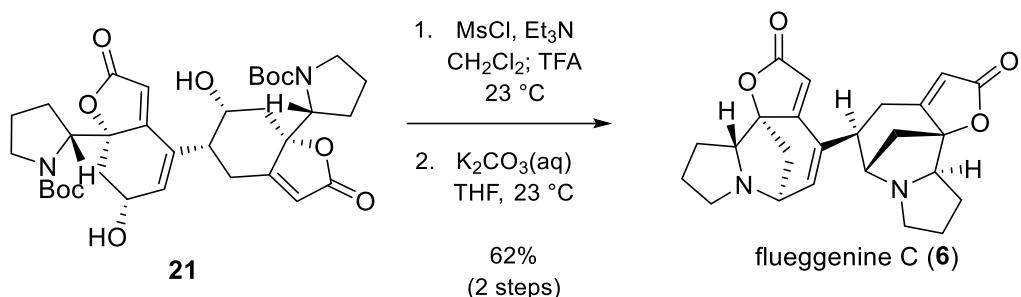
¹H NMR (400.1 MHz, CDCl₃): 6.25 (s, 1H), 6.00 (s, 1H), 5.78 (s, 1H), 5.21 (s, 1H), 4.75 (s, 1H), 4.34 (d, *J* = 8.2 Hz, 1H), 4.01 (d, *J* = 8.2 Hz, 1H), 3.62 – 3.51 (m, 2H), 3.44 – 3.35 (m, 2H), 3.31 (s, 1H), 2.95 – 2.85 (m, 1H), 2.77 (d, *J* = 15.1 Hz, 1H), 2.64 – 2.51 (m, 2H), 2.01 – 1.55 (m, 10H), 1.48 (s, 9H), 1.45 (s, 9H), 1.39 – 1.30 (m, 2H).

¹³C NMR (226.4 MHz, CDCl₃): δ 172.3, 171.9, 169.5, 168.4, 156.8, 156.4, 140.0, 130.5, 117.0, 112.8, 90.9, 90.8, 80.7, 80.4, 67.7, 66.1, 59.2, 58.8, 47.6, 47.4, 41.4, 41.0, 39.7, 28.9, 28.6 (6), 25.5, 25.0 (2), 24.8.

HRMS (ESI): Calculated for C₃₄H₄₆N₂O₁₀ [M+Na]⁺: 665.3045, found: 665.3088.

[*α*]²⁰_D : 140.5° (*c* = 0.1 in CH₂Cl₂).

TLC (ethyl acetate : hexanes = 6 : 1) *R*_f: 0.33 (CAM, KMnO₄, UV).



Flueggenine C (6):

To a stirred solution of **21** (10.9 mg, 0.017 mmol, 1 equiv) in CH₂Cl₂ (1 mL) at 0 °C was added triethylamine (19.1 µL, 0.137 mmol, 8.00 equiv), and methanesulfonyl chloride (5.3 µL, 0.068 mmol, 4.00 equiv). After 30 min, trifluoroacetic acid (1 mL) was added to the reaction mixture at 23 °C. After 30 min, the reaction mixture was concentrated under reduced pressure. The resulting crude was dissolved in THF (1 mL) and saturated aqueous potassium carbonate solution (1 mL) was added. The mixture was stirred at 23 °C. After 30 min, brine (10 mL) and ethyl acetate (10 mL) were added and the layers were separated. The aqueous layer was extracted with ethyl acetate (3 × 10 mL), and the combined organic layers were dried over anhydrous sodium sulfate and concentrated under reduced pressure. The resulting crude residue was purified by column chromatography (silica gel: diam. 1.5 cm, ht. 4 cm; eluent: methanol : dichloromethane = 1 : 15) to afford **6** (4.3 mg, 62%) as a white amorphous powder.

¹H NMR (800.2 MHz, CDCl₃)⁹: δ 6.44 (d, *J* = 6.6 Hz, 1H), 5.76 (s, 1H), 5.69 (d, *J* = 2.3 Hz, 1H), 3.65 – 3.62 (m, 1H), 3.40 (td, *J* = 6.6, 3.2 Hz, 1H), 3.28 (dt, *J* = 8.6, 3.7 Hz, 1H), 3.21 – 3.18 (m, 2H), 3.16 (t, *J* = 8.0 Hz, 1H), 3.08 (dt, *J* = 5.6, 2.4 Hz, 1H), 3.05 (dd, *J* = 8.5, 2.5 Hz, 1H), 3.02 (d, *J* = 16.8 Hz, 1H), 2.62 (td, *J* = 9.8, 5.5 Hz, 1H), 2.56 – 2.51 (m, 2H), 2.28 (dd, *J* = 11.5, 5.7 Hz, 1H), 2.03 – 1.96 (m, 3H), 1.96 – 1.90 (m, 1H), 1.83 – 1.72 (m, 4H), 1.58 (d, *J* = 10.6 Hz, 1H), 1.49 (d, *J* = 11.5 Hz, 1H).

¹³C NMR (201.1 MHz, CDCl₃): δ 173.6, 172.7, 172.3, 169.2, 140.2, 134.5, 131.1, 110.0, 107.4, 92.3, 92.2, 66.5, 65.4, 64.3, 59.6, 57.9, 55.5, 43.0, 35.7, 30.1, 29.9, 29.6, 29.1, 27.8, 27.0, 26.9.

HRMS (ESI): Calculated for C₂₄H₂₆N₂O₄ [M+Na]⁺: 429.1785, found: 429.1766.

$[\alpha]^{20}_{\text{D}}: -83^\circ$ ($c = 0.13$ in MeOH).

TLC (methanol : dichloromethane = 1 : 3) R_f: 0.2 (CAM, KMnO₄, UV).

5. Comparison of Spectroscopic Data of Natural and Synthetic Flueggene C

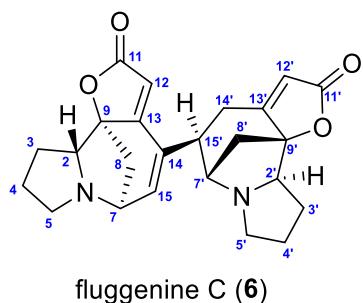


Table S3. Comparison of ^1H NMR spectroscopic data of natural and synthetic flueggene C (6**)**

position	natural 6 ^{3d} δ_1 (ppm ; multi, <i>J</i> in Hz)	synthetic 6 ⁹ δ_2 (ppm ; multi, <i>J</i> in Hz)	deviation $\Delta\delta = \delta_1 - \delta_2$ (ppm)
2	3.15 (dd, 8.7, 7.5)	3.16 (t, 8.0)	-0.01
3	a 1.97 (m) b 1.77 (m)	a 1.99 (m) b 1.78 (m)	-0.02 -0.01
4	a 1.98 (m) b 1.78 (m)	a 1.99 (m) b 1.78 (m)	-0.01 0
5	a 3.27 (m) b 2.52 (m)	a 3.28 (dt, 8.6, 3.7) b 2.54 (m)	-0.01 -0.02
7	3.63 (dd, 6.6, 4.6)	3.64 (m)	-0.01
8	2.55 (dd, 10.6, 4.6) 1.58 (d, 10.6)	2.54 (m) 1.58 (d, 10.6)	0.01 0
12	5.75 (s)	5.76 (s)	-0.01
15	6.43 (d, 6.5)	6.44 (d, 6.6)	-0.01
2'	3.18 (m)	3.19 (m)	-0.01
3'	a 1.92 (m) b 1.75 (m)	a 1.93 (m) b 1.77 (m)	-0.01 -0.02
4'	a 1.99 (m) b 1.76 (m)	a 1.99 (m) b 1.77 (m)	0 -0.01
5'	a 3.39 (m) b 2.61 (m)	a 3.40 (td, 6.6, 3.2) b 2.62 (td, 9.8, 5.5)	-0.01 -0.01
7'	3.07 (m)	3.08 (m)	-0.01
8'	a 2.28 (dd, 11.4, 5.7) b 1.48 (d, 11.4)	a 2.28 (dd, 11.5, 5.7) b 1.49 (d, 11.5)	0 -0.01
12'	5.69 (d, 1.8)	5.69 (d, 2.3)	0
14'	β 3.06 (ddd, 16.3, 7.9, 1.8) α 3.01 (d, 16.3)	β 3.06 (m) α 3.02 (d, 16.8)	0 -0.01
15'	3.17 (m)	3.19 (m)	-0.02

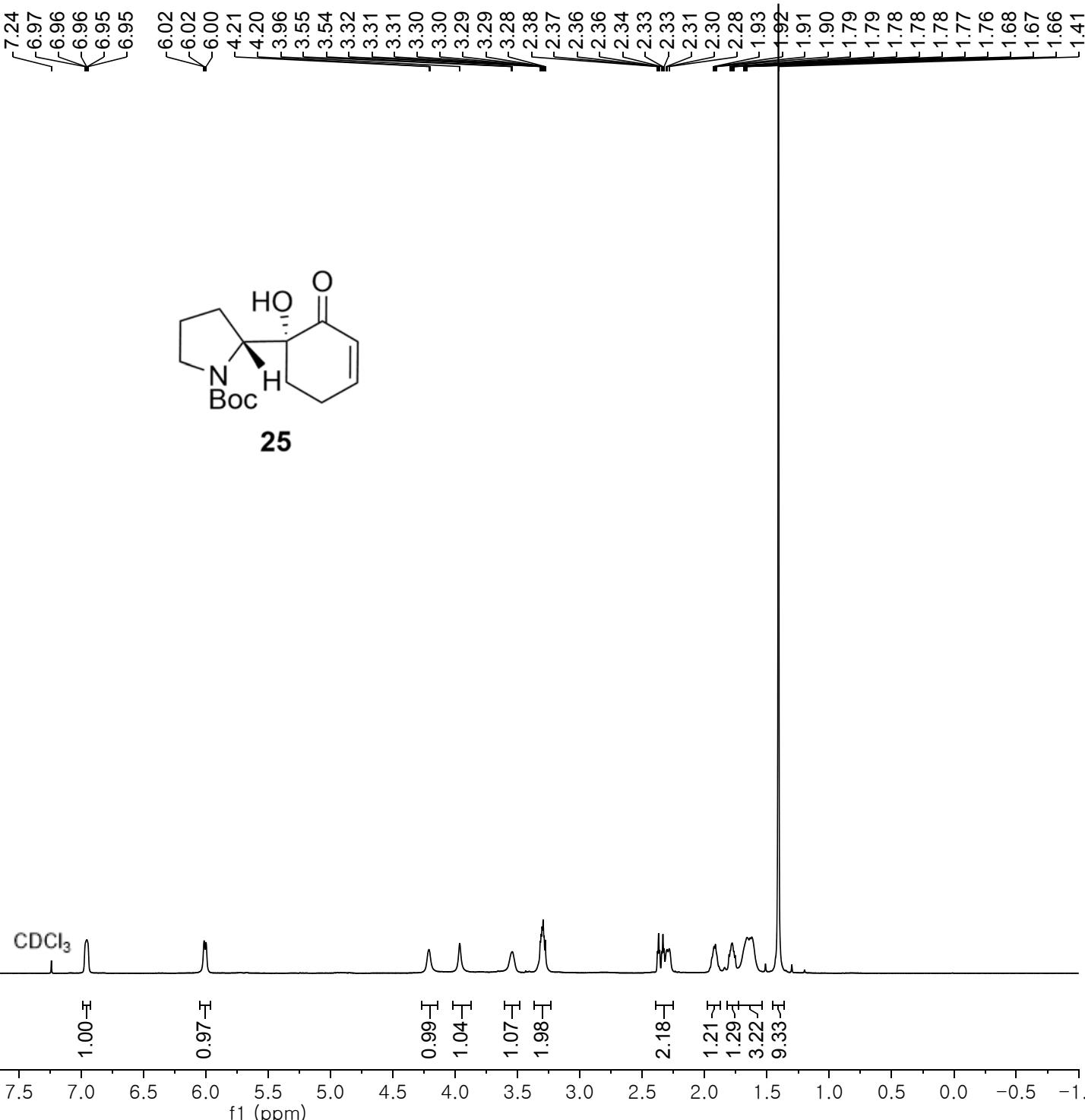
Table S4. Comparison of ^{13}C NMR spectroscopic data of natural and synthetic flueggene C (6)

position	natural 6 δ_1 (ppm)	synthetic 6 δ_2 (ppm)	deviation $\Delta\delta = \delta_1 - \delta_2$ (ppm)
2	65.4	65.4	0
3	29.6	29.6	0
4	27.0	27.0	0
5	55.4	55.5	-0.1
7	59.6	59.6	0
8	35.7	35.7	0
9	92.2	92.2	0
11	172.3	172.3	0
12	107.4	107.4	0
13	169.3	169.2	0.1
14	134.5	134.5	0
15	140.2	140.2	0
2'	66.4	66.5	-0.1
3'	29.1	29.1	0
4'	26.9	26.9	0
5'	57.9	57.9	0
7'	64.3	64.3	0
8'	30.1	30.1	0
9'	92.3	92.3	0
11'	172.7	172.7	0
12'	110.0	110.0	0
13'	173.6	173.6	0
14'	27.8	27.8	0
15'	42.9	43.0	-0.1

6. References

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- ² Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518.
- ³ (a) Gan, L. -S.; Fan, C. -Q.; Yang, S. -P.; Wu, Y.; Lin, L. -P.; Ding, J.; Yue, J. M. *Org. Lett.* **2006**, *8*, 2285. (b) Qin, S.; Liang, J. -Y.; Gu, Y. -C.; Guo Y. -W. *Tetrahedron Lett.* **2008**, *49*, 7066. (c) Zhao, B. X.; Wang, Y.; Zhang, D. M.; Jiang, R. W.; Wang, G. C.; Shi, J. M.; Huang, X. J.; Chen, W. M.; Che, C. T.; Ye, W. C. *Org. Lett.* **2011**, *13*, 3888. (d) Zhang, H.; Wei, W.; Yue, J. M. *Tetrahedron* **2013**, *69*, 3942. (e) Zhao, B. -X.; Wang, Y.; Li, C.; Wang, G. -C.; Huang, X. -J.; Fan, C. -L.; Li, Q.-M.; Zhu, H. -J.; Chen, W. -M.; Ye, W. -C. *Tetrahedron Lett.* **2013**, *54*, 4708. (f) Li, X. -H.; Cao, M. -M.; Zhang, Y.; Li, S. -L.; Di, Y. -T.; Hao, X. -J. *Tetrahedron Lett.* **2014**, *55*, 6101. (g) Zhang, H.; Zhang, C. -R.; Han, Y. -S.; Wainberg, M. A.; Yue, J. -M. *RSC Adv.* **2015**, *5*, 107045. (h) Zhang, H.; Han, Y. S.; Wainberg, M. A.; Yue, J. -M. *Tetrahedron* **2015**, *71*, 3671. (i) Zhang, H.; Zhu, K. -K.; Han, Y. S.; Luo, C.; Wainberg, M. A.; Yue, J. -M. *Org. Lett.* **2015**, *17*, 6274. (j) Zhang, H.; Han, Y. -S.; Wainberg, M. A.; Yue, J. -M. *Tetrahedron Lett.* **2016**, *57*, 1798.
- ⁴ Zhang, H.; Zhang, C. -R.; Zhu, K. -K.; Gao, A. -H.; Luo, C.; Li, J.; Yue, J. -M. *Org. Lett.* **2013**, *15*, 120.
- ⁵ NMR data is obtained in CDCl₃.
- ⁶ For the synthesis of compound **25**: Ma, N.; Yao, Y.; Zhao, B.-X.; Wang, Y.; Ye, W.-C.; Jiang, S. *Chem. Comm.* **2014**, *50*, 9284.
- ⁷ NMR data is obtained in CD₃OD.
- ⁸ Taber, D. F.; DeMatteo, P. W.; Hassan, R. A. *Org. Synth.* **2013**, *90*, 350.
- ⁹ Proton nuclear magnetic resonance spectra are referenced from the residual protium in the NMR solvent (CDCl₃: δ 7.26 (CHCl₃)) for direct comparison with the isolation paper.

7. Copies of ^1H NMR, ^{13}C NMR Spectra and HPLC Traces of New Compounds



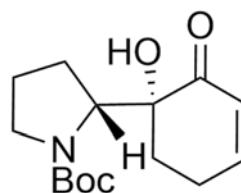
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—157.75
—153.82

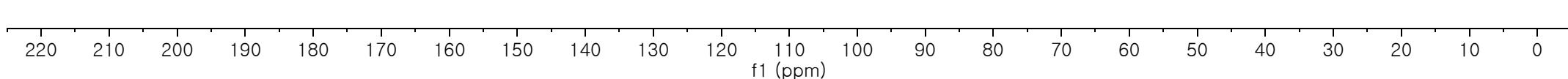
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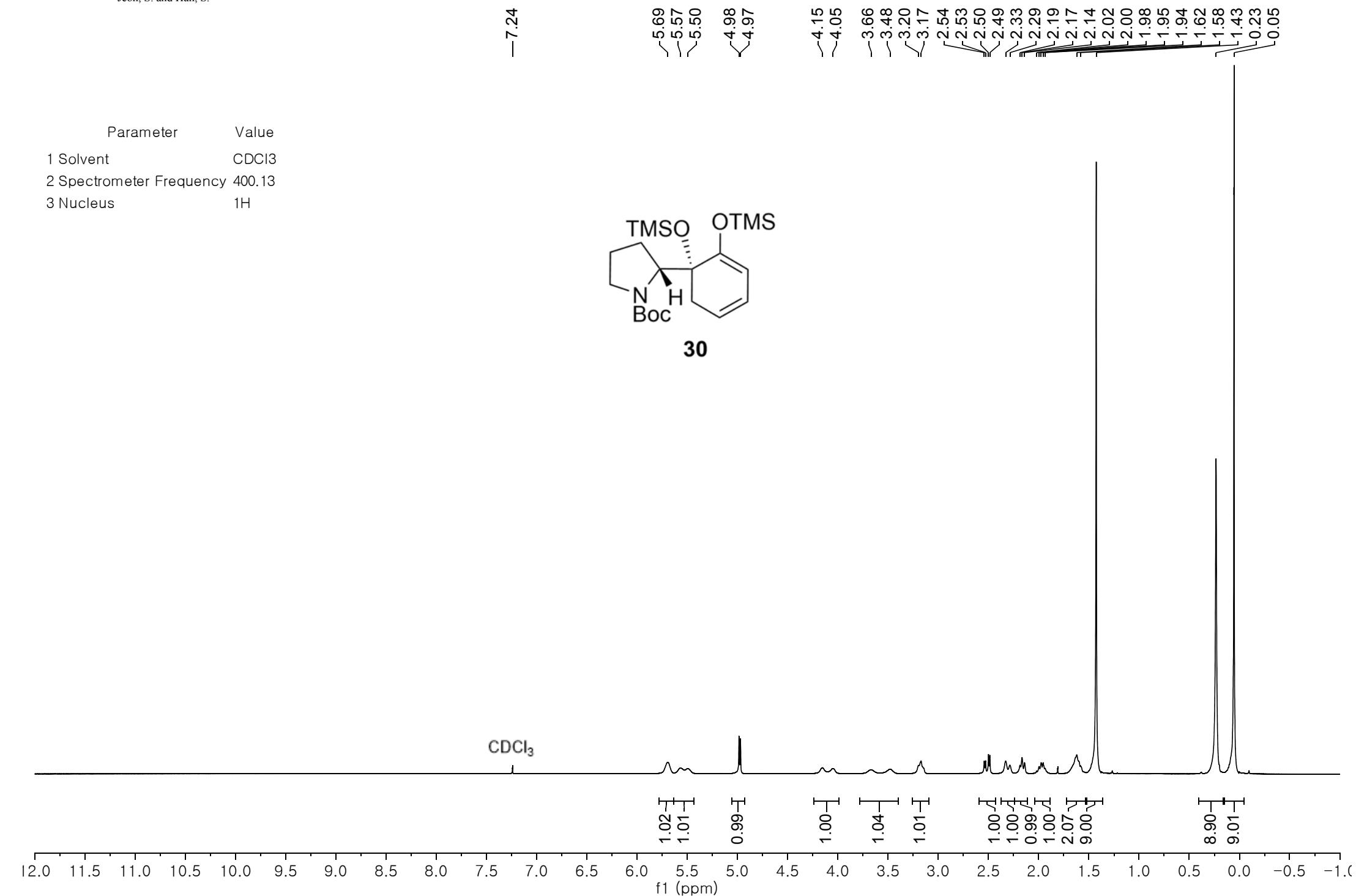
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80.53
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26.74
26.47
25.60

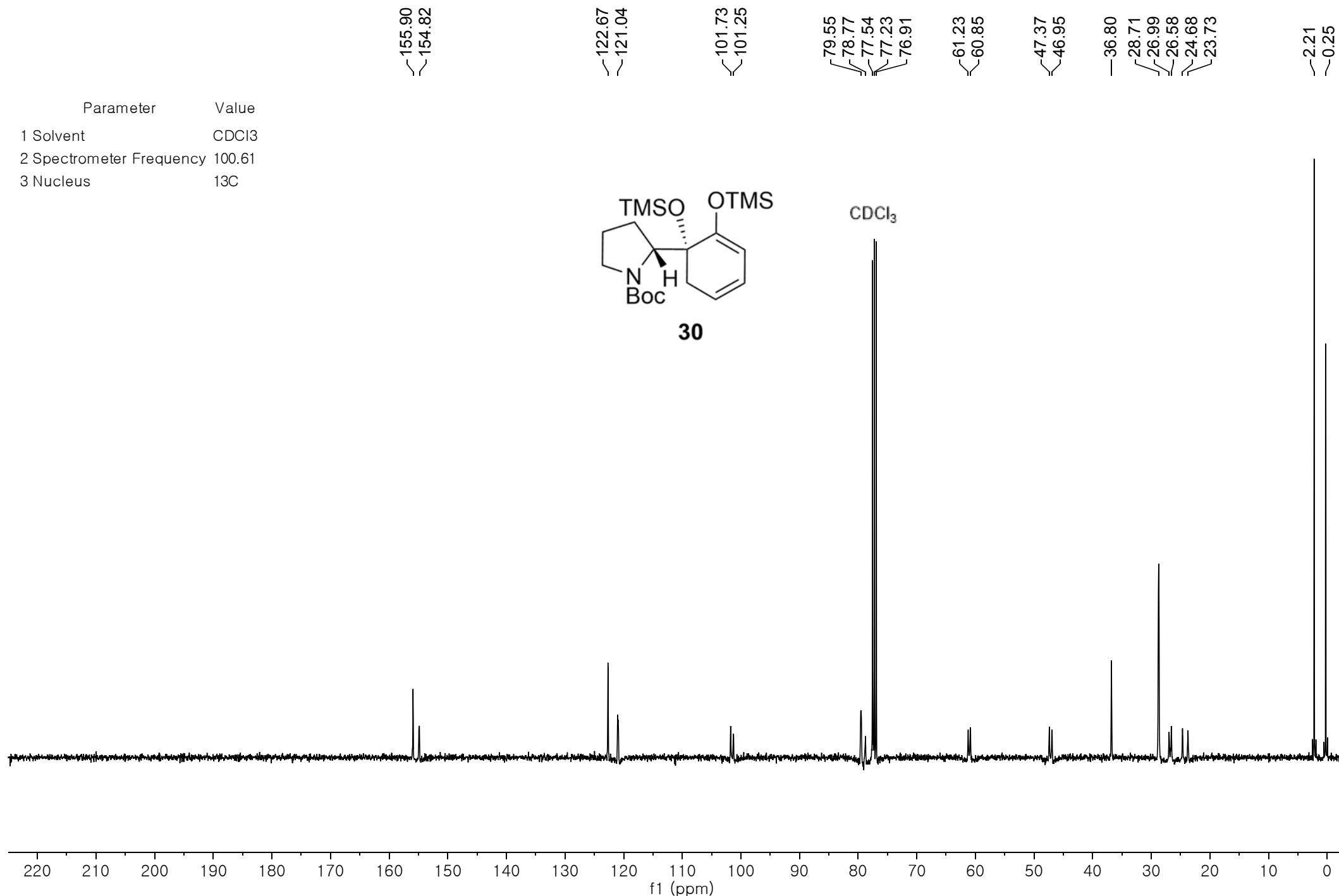
Parameter	Value
1 Solvent	MeOD
2 Spectrometer Frequency	100.62
3 Nucleus	¹³ C

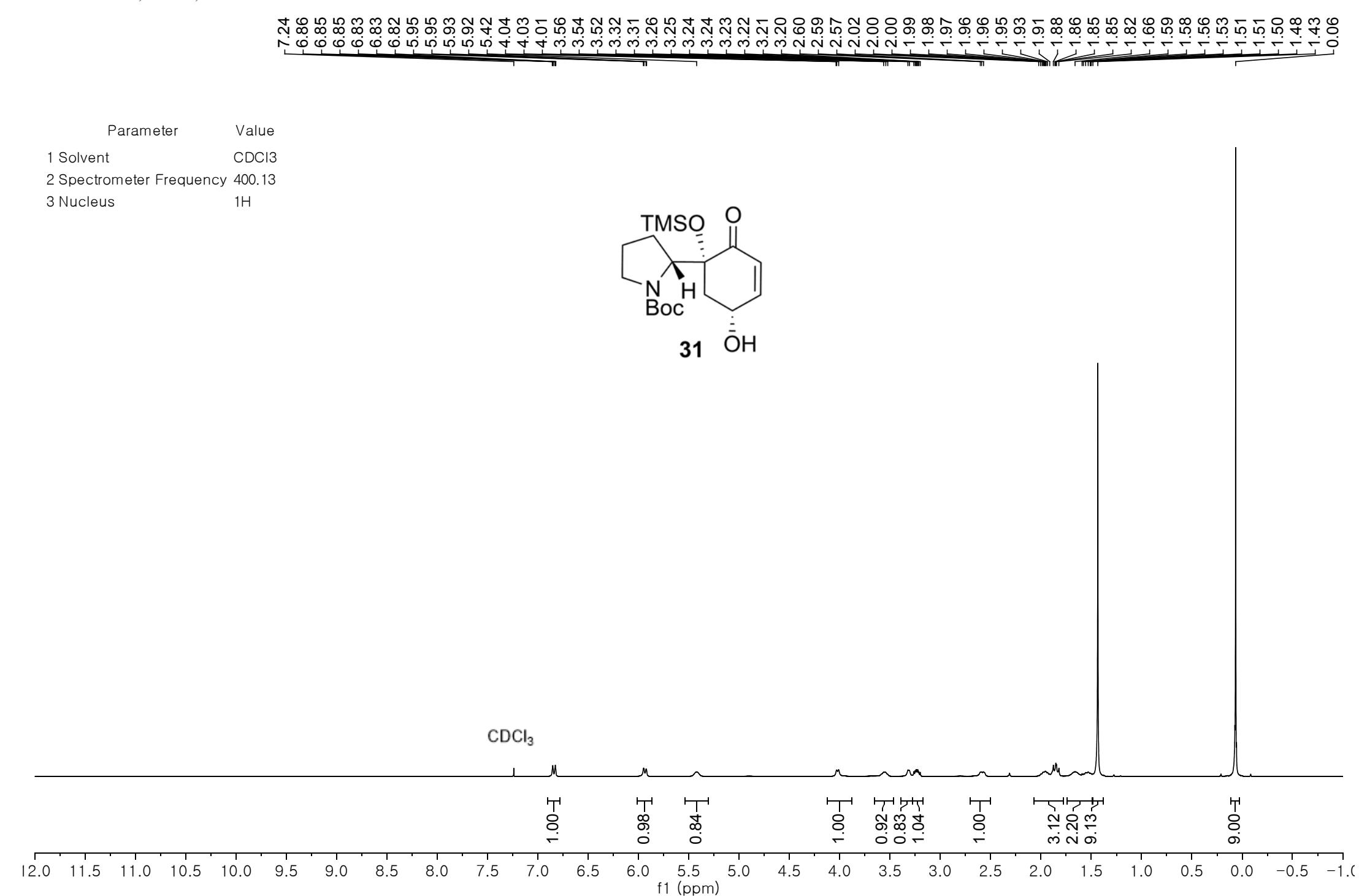


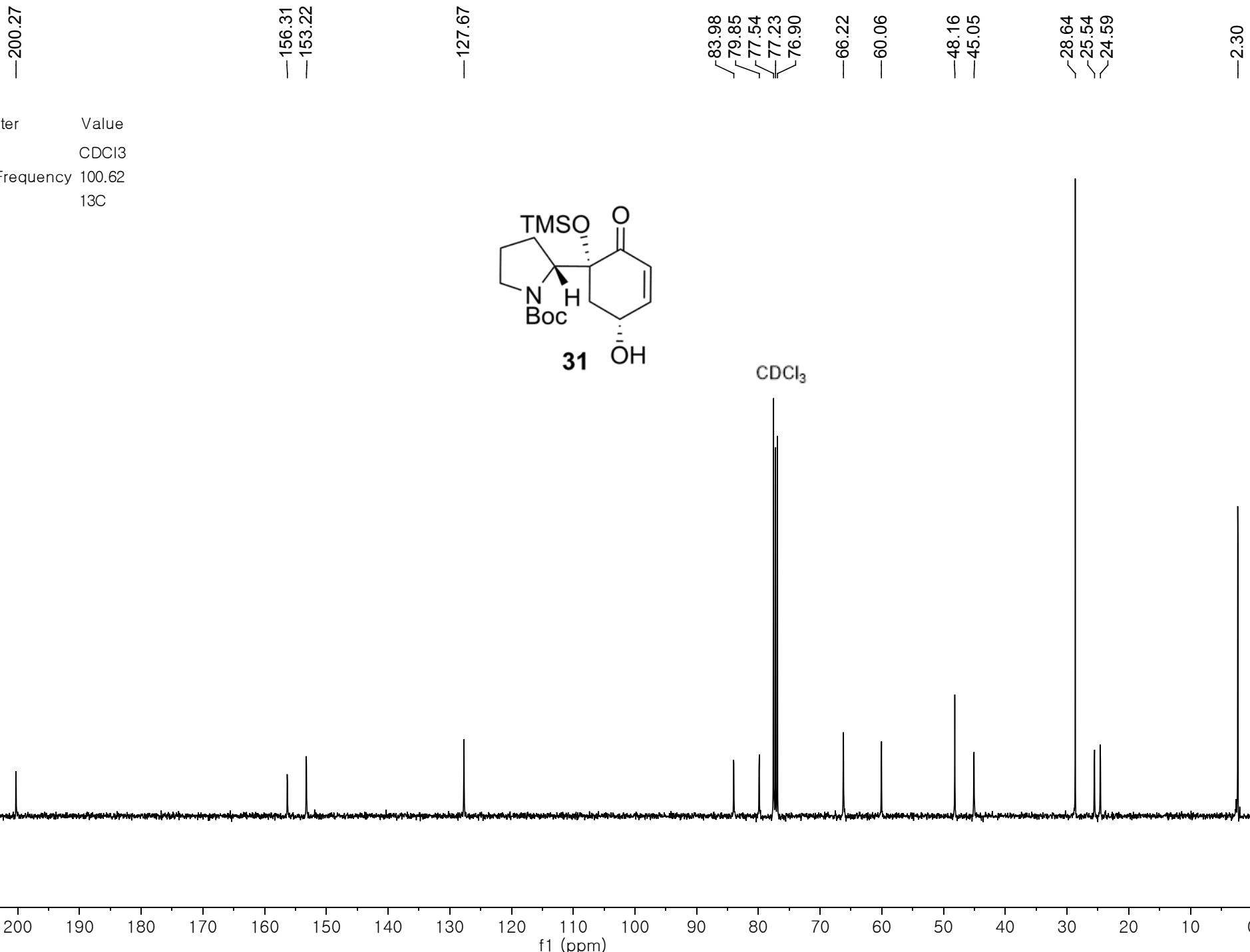
25

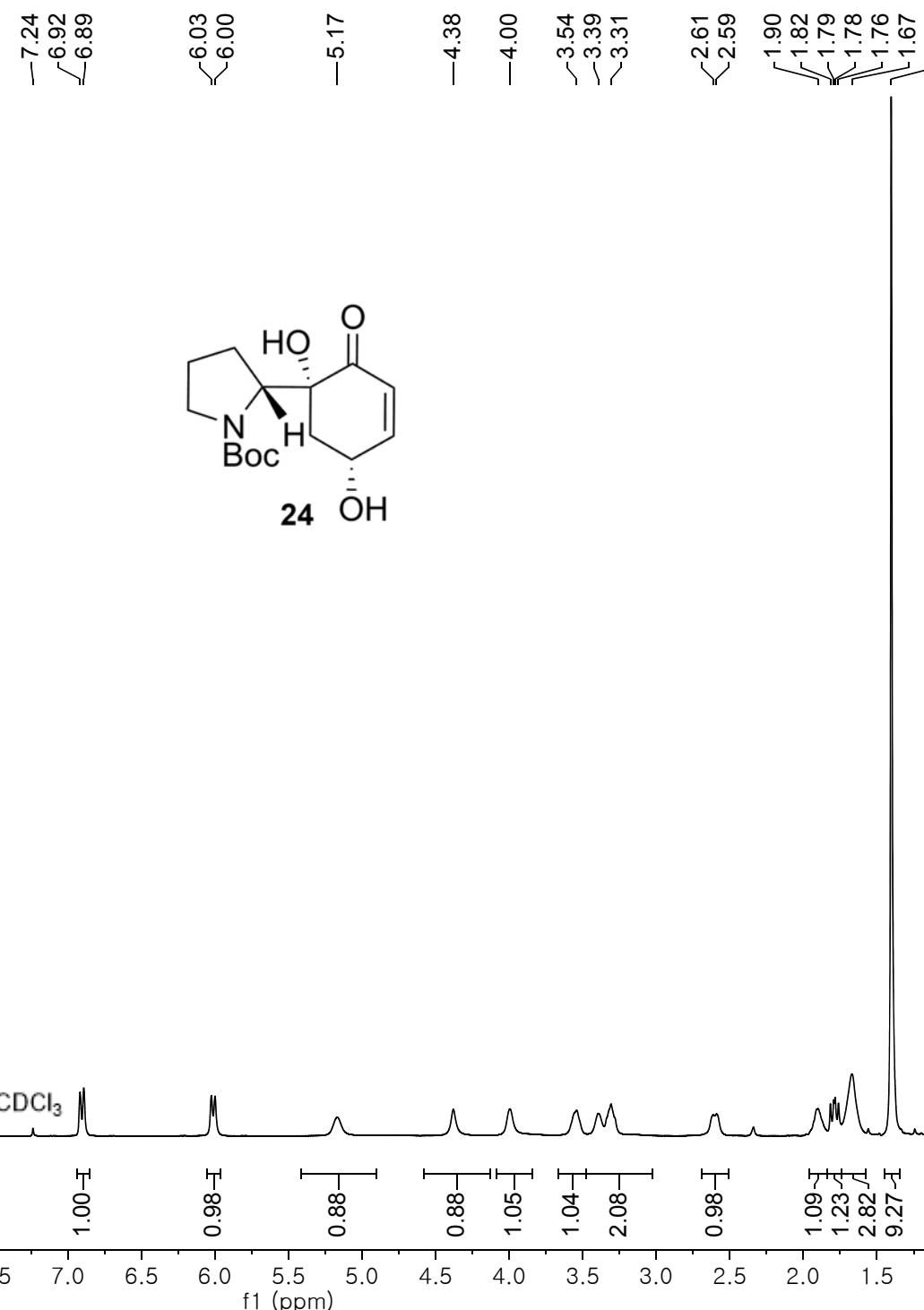


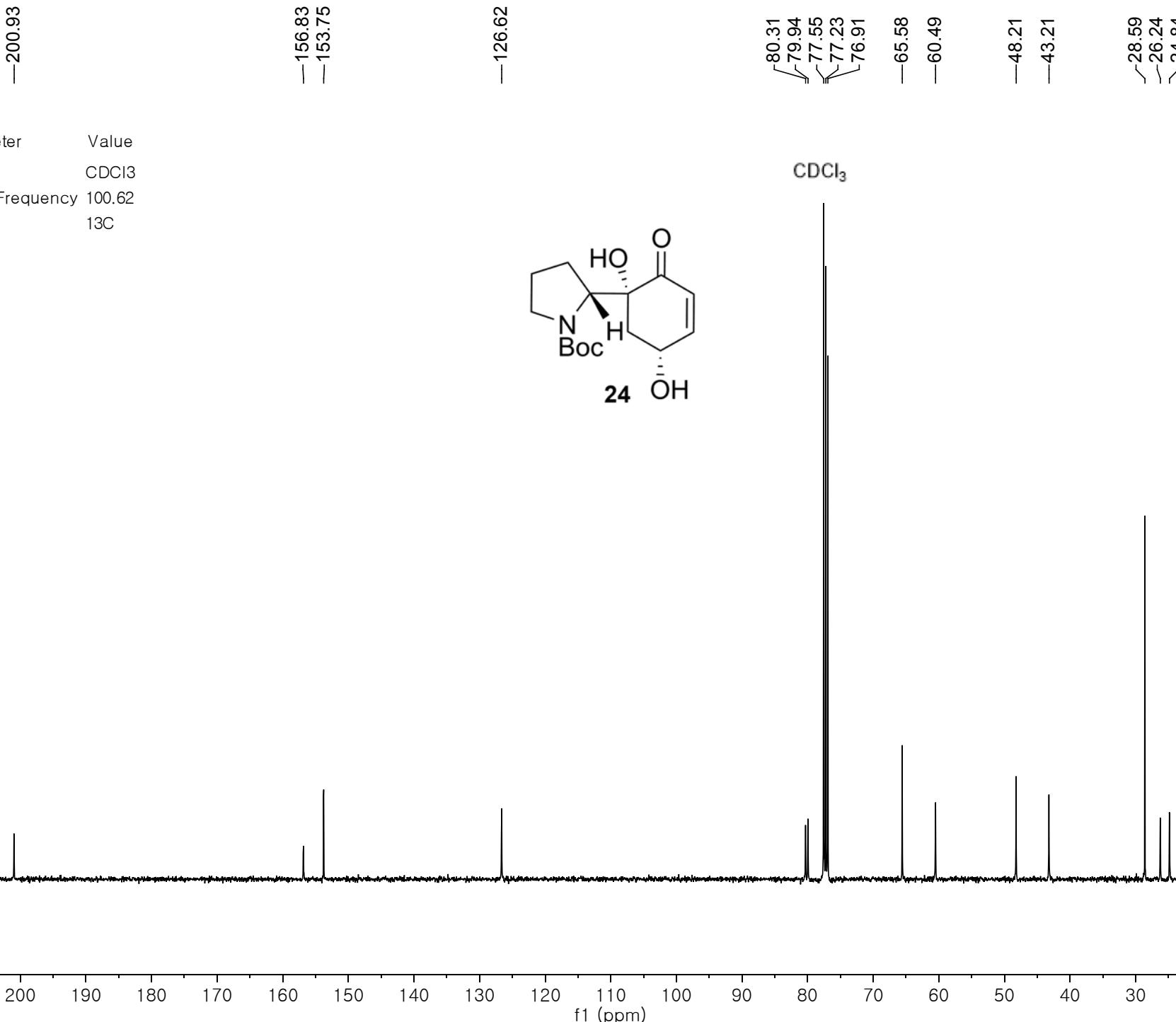






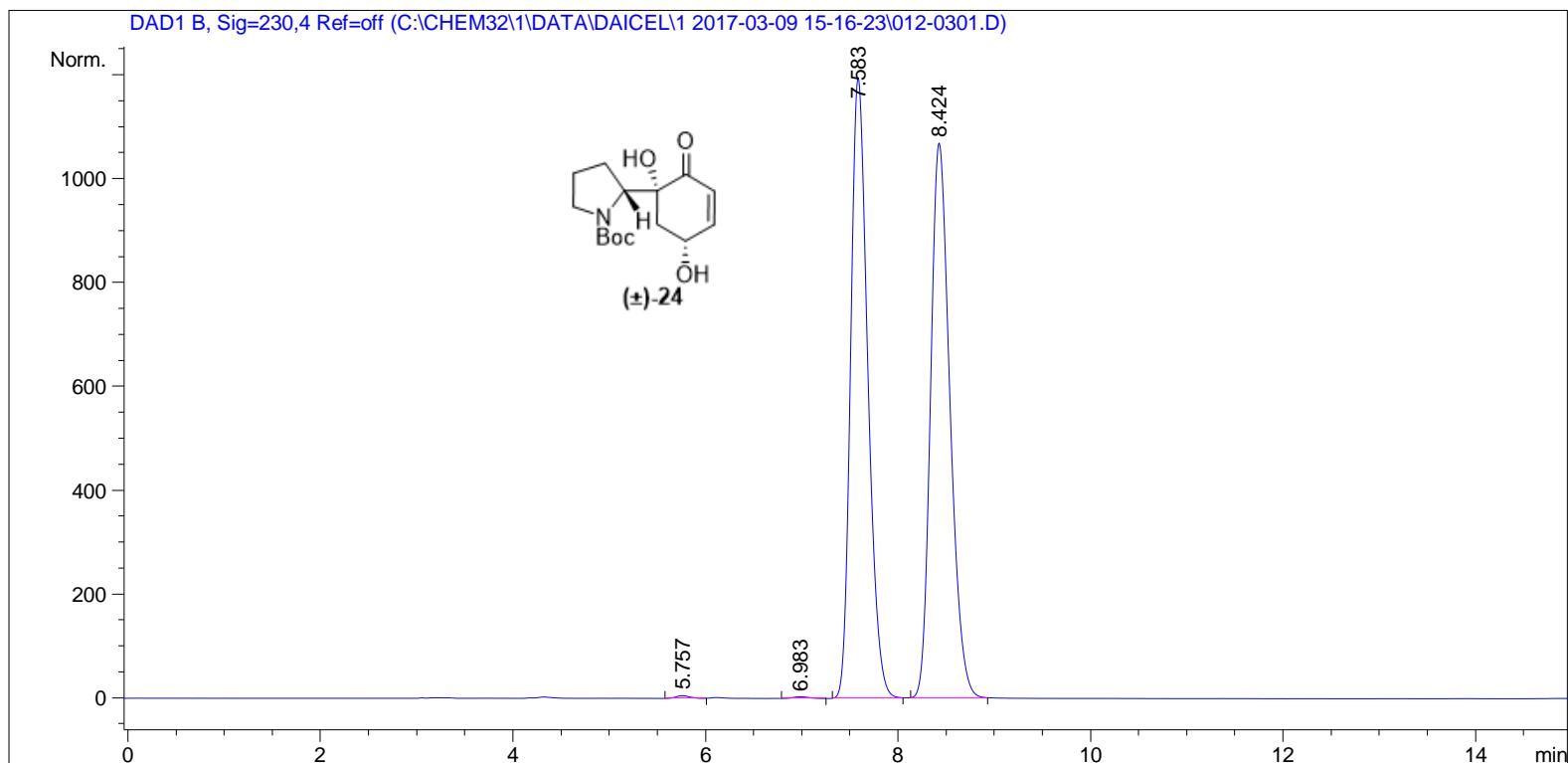






CHIRALPAK IC 1mL/min, 80:20=Hex:EtOH in 20min

=====
Acq. Operator : JH.Jin Seq. Line : 3
Acq. Instrument : Instrument 1 Location : Vial 12
Injection Date : 3/9/2017 3:55:23 PM Inj : 1
Inj Volume : 5 μ l
Acq. Method : C:\Chem32\1\DATA\DAICEL\1 2017-03-09 15-16-23\1.M
Last changed : 3/9/2017 3:54:08 PM by JH.Jin
(modified after loading)
Analysis Method : C:\CHEM32\1\DATA\DAICEL\1 2017-03-09 15-16-23\012-0301.D\DA.M (1.M)
Last changed : 3/9/2017 5:25:00 PM by JH.Jin
Sample Info : IC, Hex/EtOH=80/20



=====
Area Percent Report with Performance
=====

Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

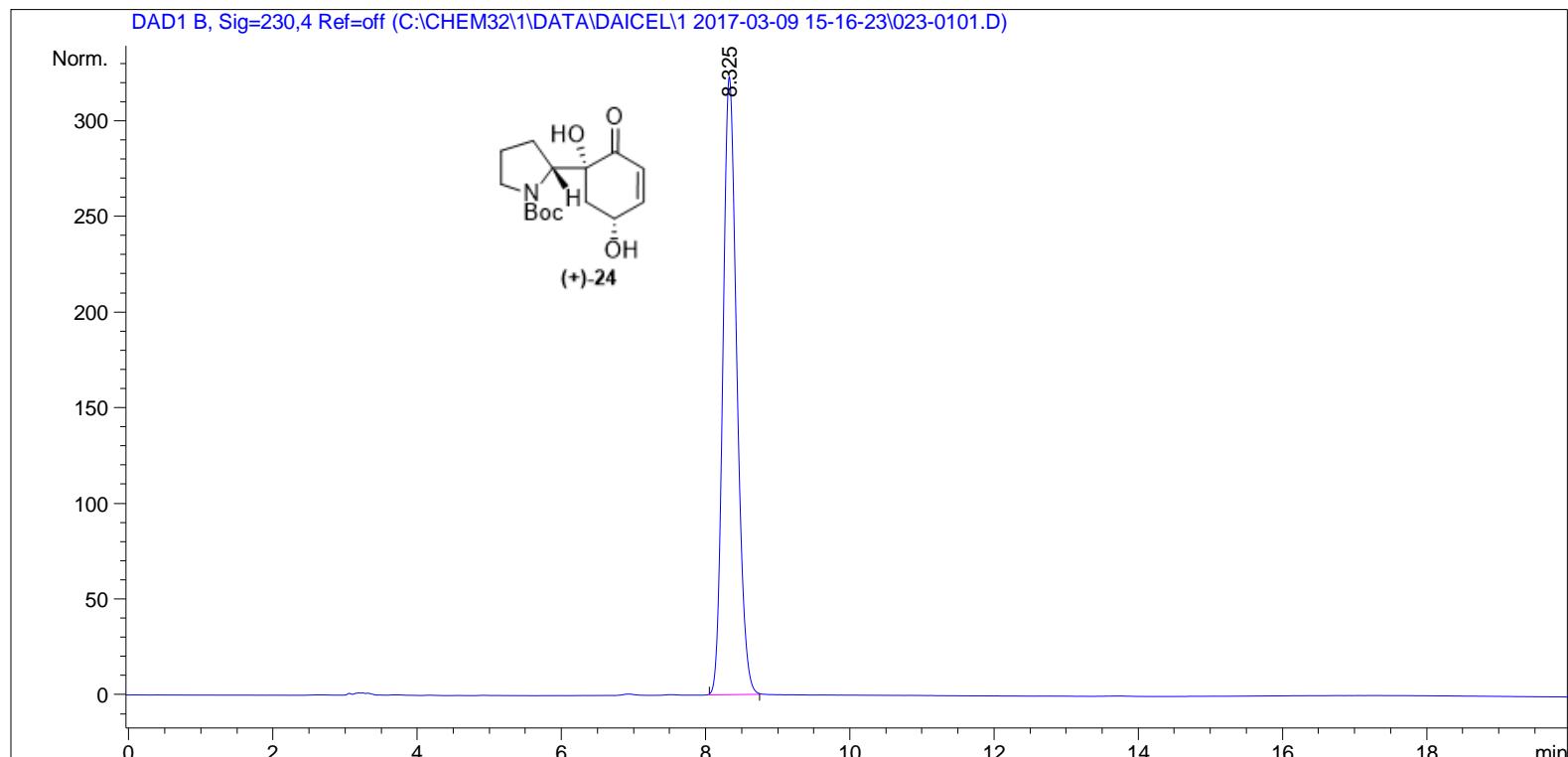
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RetTime	k'	Area	Height	Symm.	Width	Plates	Resol	Selectivity
[min]		[mAU*s]	[mAU]		[min]			
5.757	-	48.92553	5.11523	0.85	0.1500	8161	-	-
6.983	-	35.12663	3.15693	0.81	0.1700	9347	4.50	1.21
7.583	-	1.48401e4	1195.54419	0.73	0.1889	8929	1.97	1.09
8.424	-	1.49591e4	1069.42249	0.73	0.2111	8821	2.47	1.11

CHIRALPAK IC 1mL/min, 80:20=Hex:EtOH in 20min

=====

Acq. Operator : JH.Jin Seq. Line : 1
Acq. Instrument : Instrument 1 Location : Vial 23
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Inj Volume : 5 μ l
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Last changed : 3/9/2017 3:16:21 PM by JH.Jin
Analysis Method : C:\CHEM32\1\DATA\DAICEL\1 2017-03-09 15-16-23\023-0101.D\DA.M (1.M)
Last changed : 3/9/2017 5:25:28 PM by JH.Jin
Sample Info : IC, Hex/EtOH=80/20



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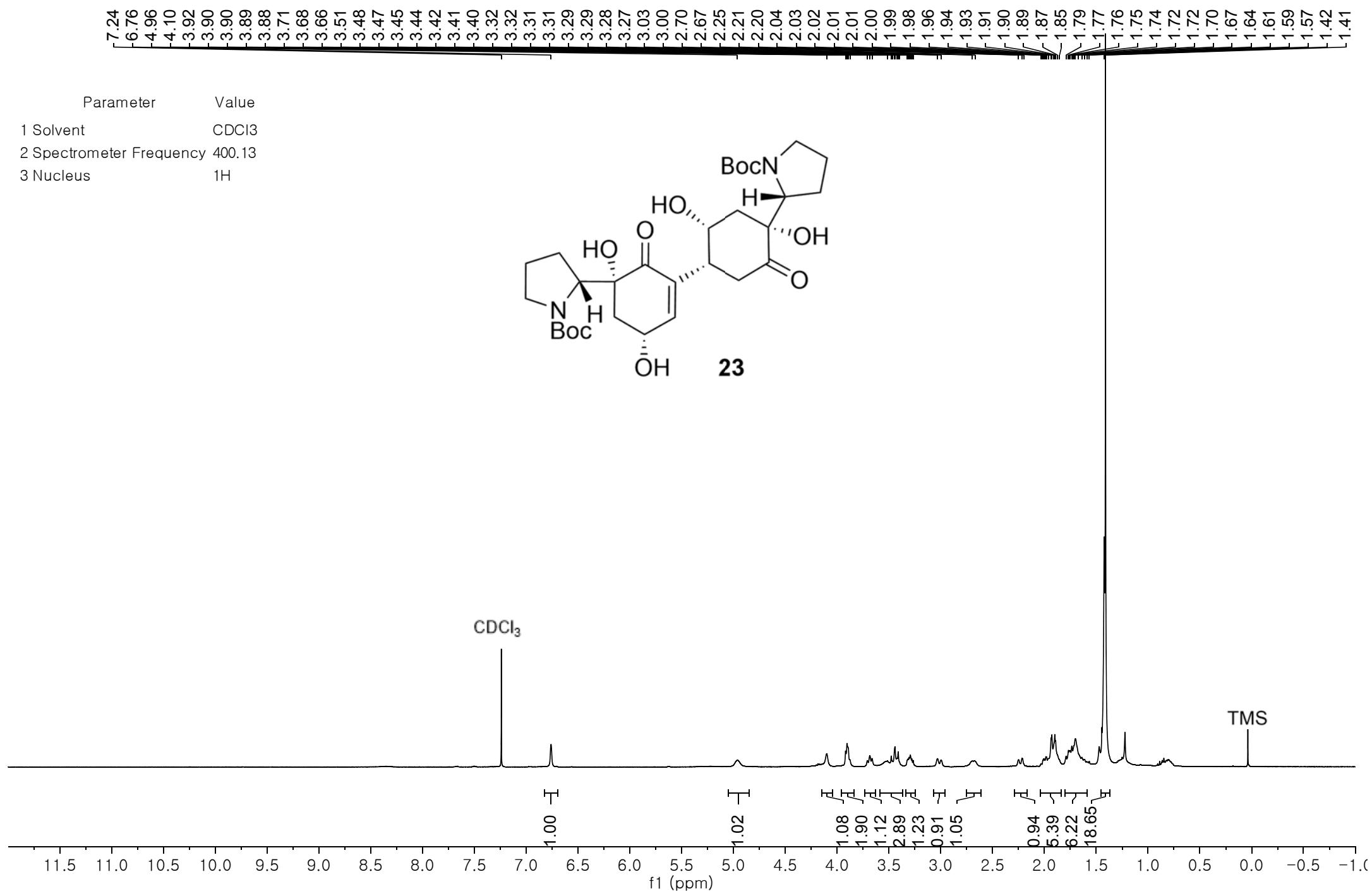
Area Percent Report with Performance

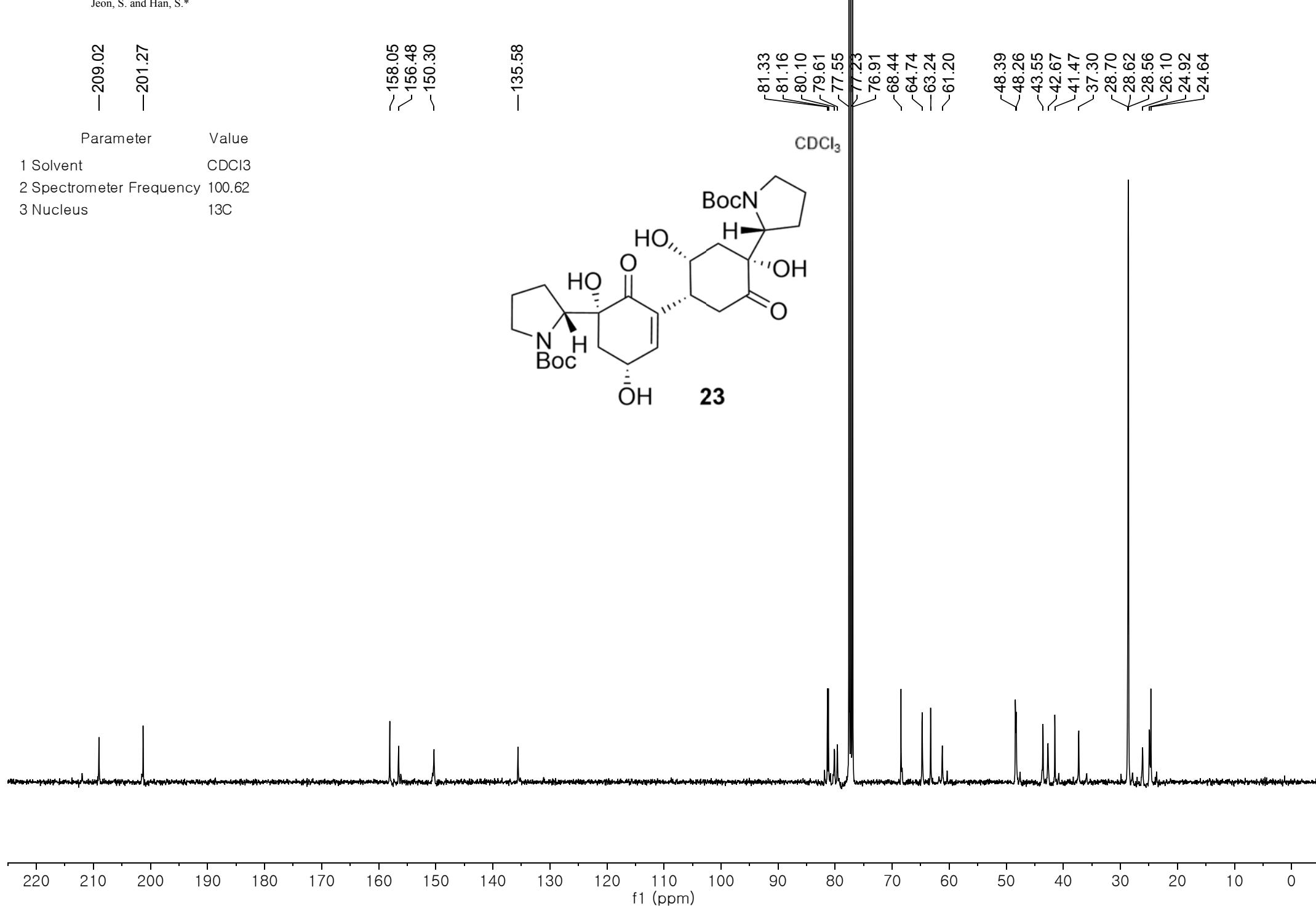
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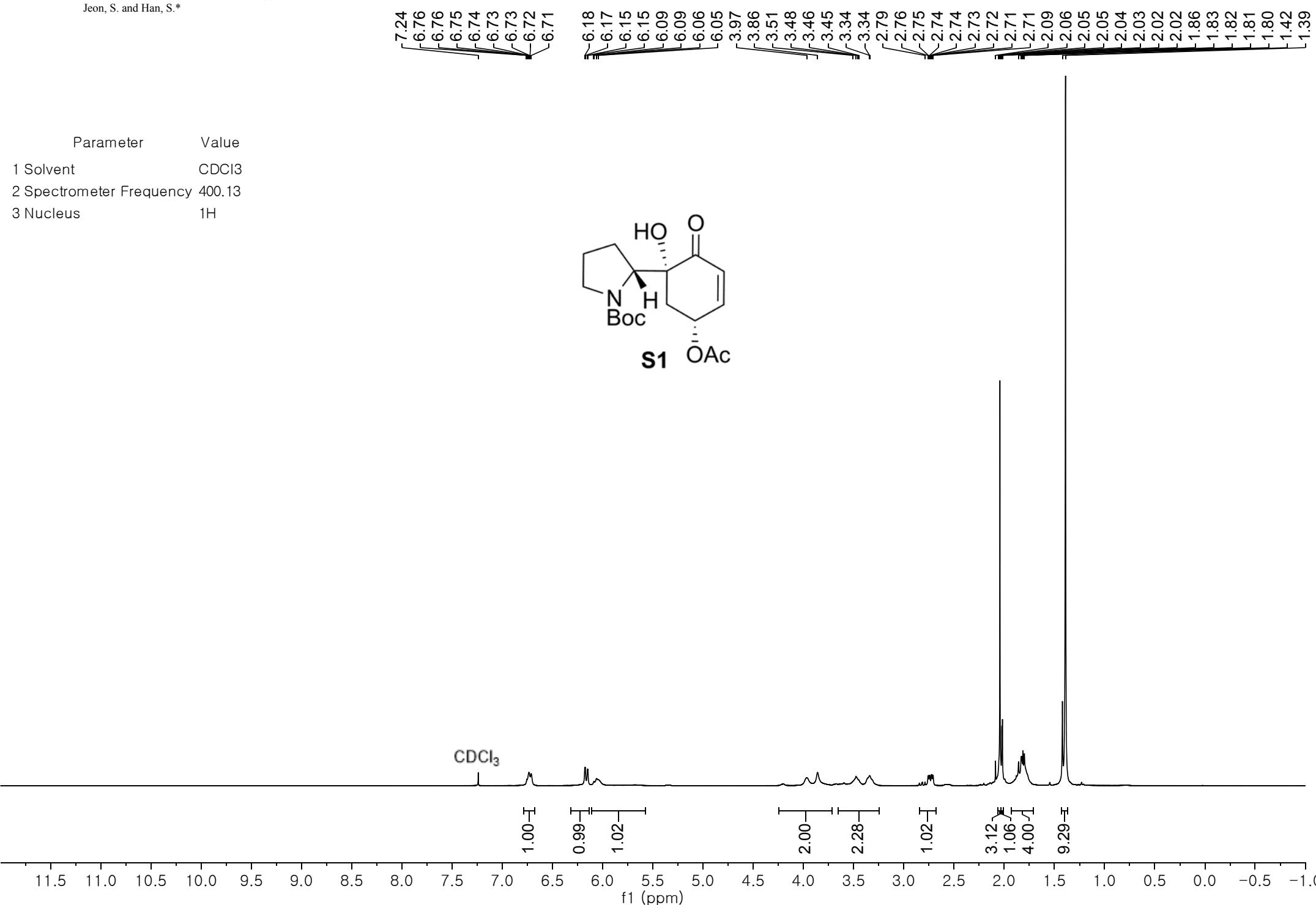
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

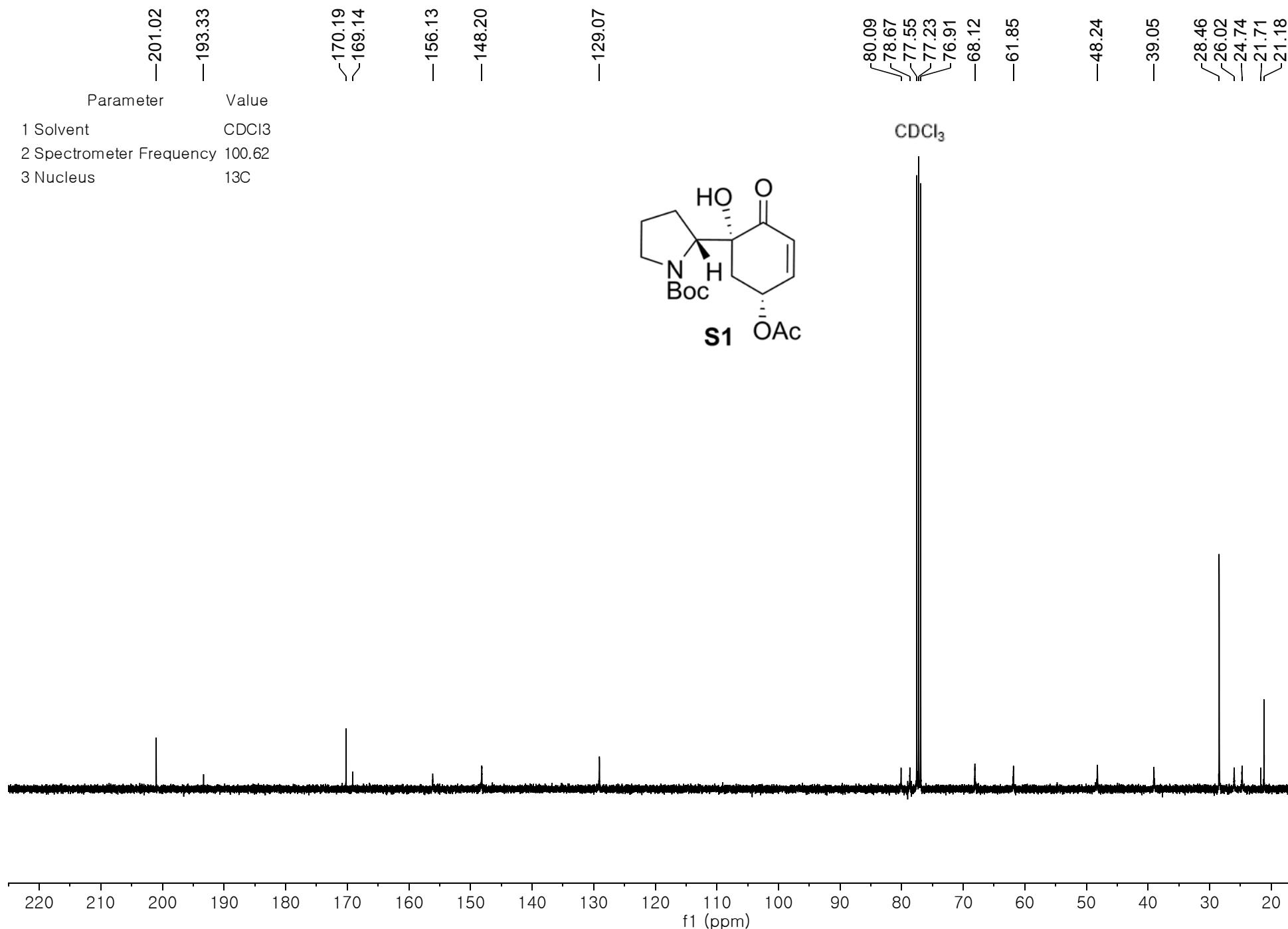
Signal 1: DAD1 B, Sig=230,4 Ref=off

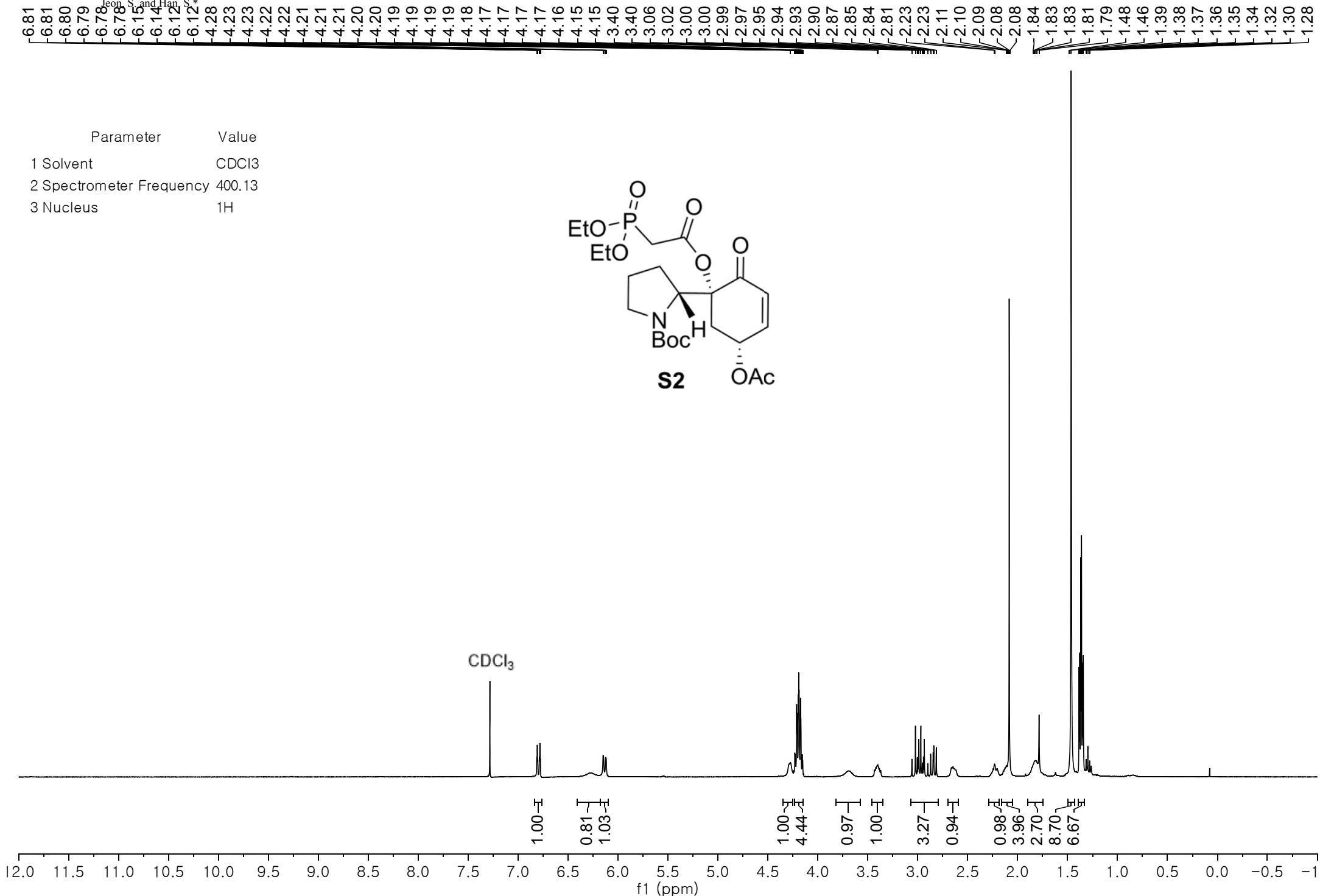
RetTime	k'	Area	Height	Symm.	Width	Plates	Resol	Selectivity
[min]		[mAU*s]	[mAU]		[min]			
8.325	-	4327.17725	323.11252	0.80	0.2044	9185	-	-

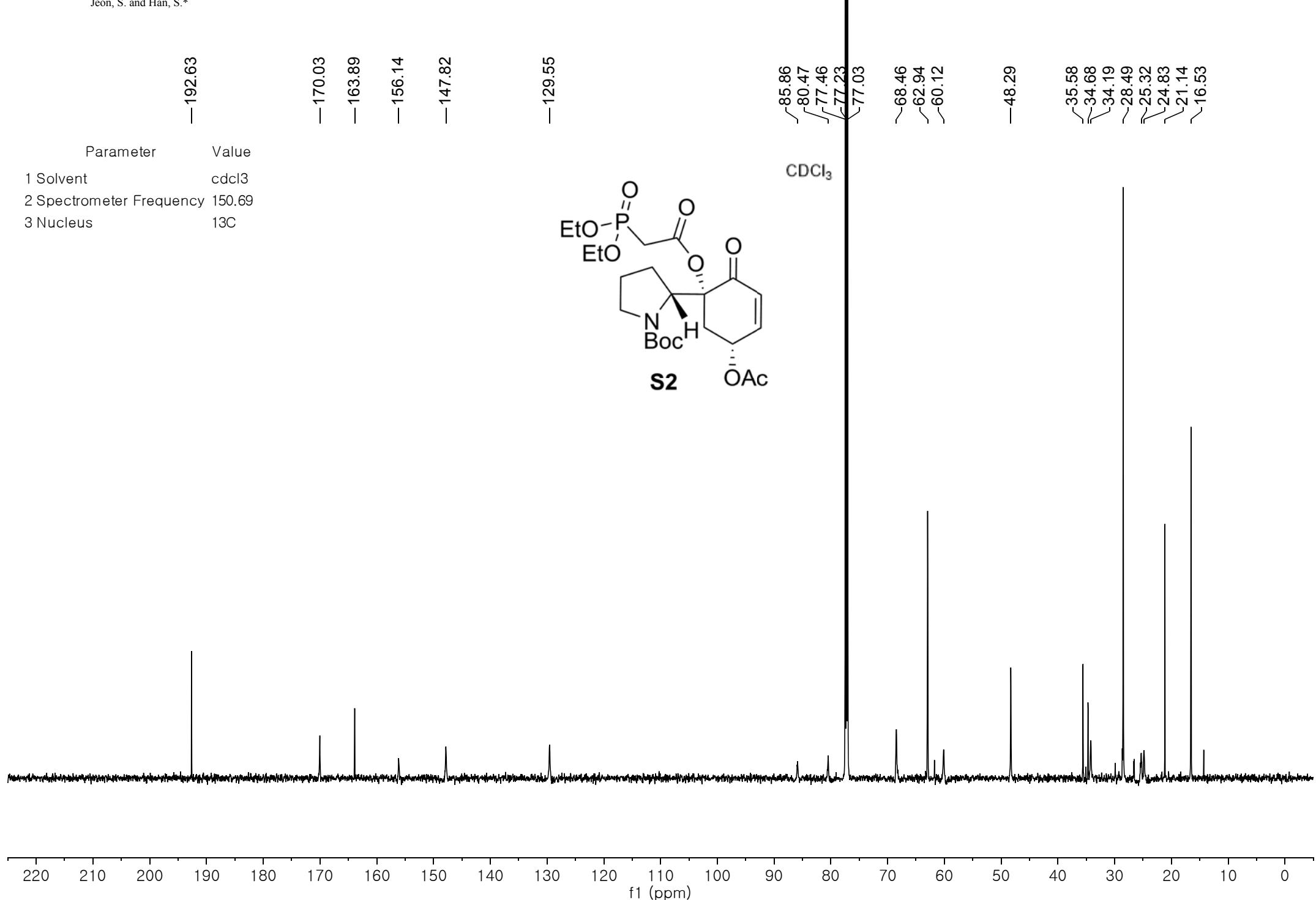


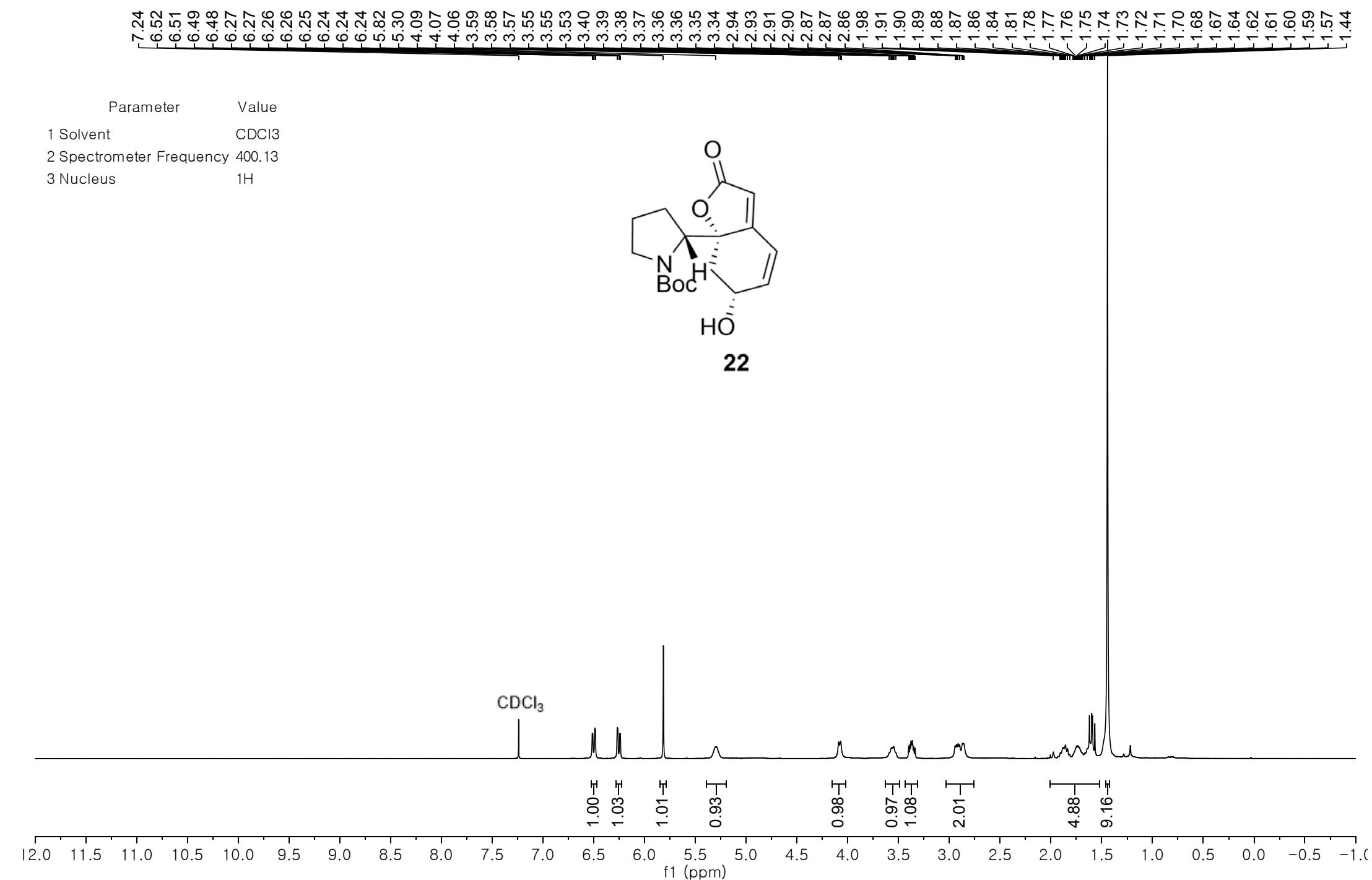


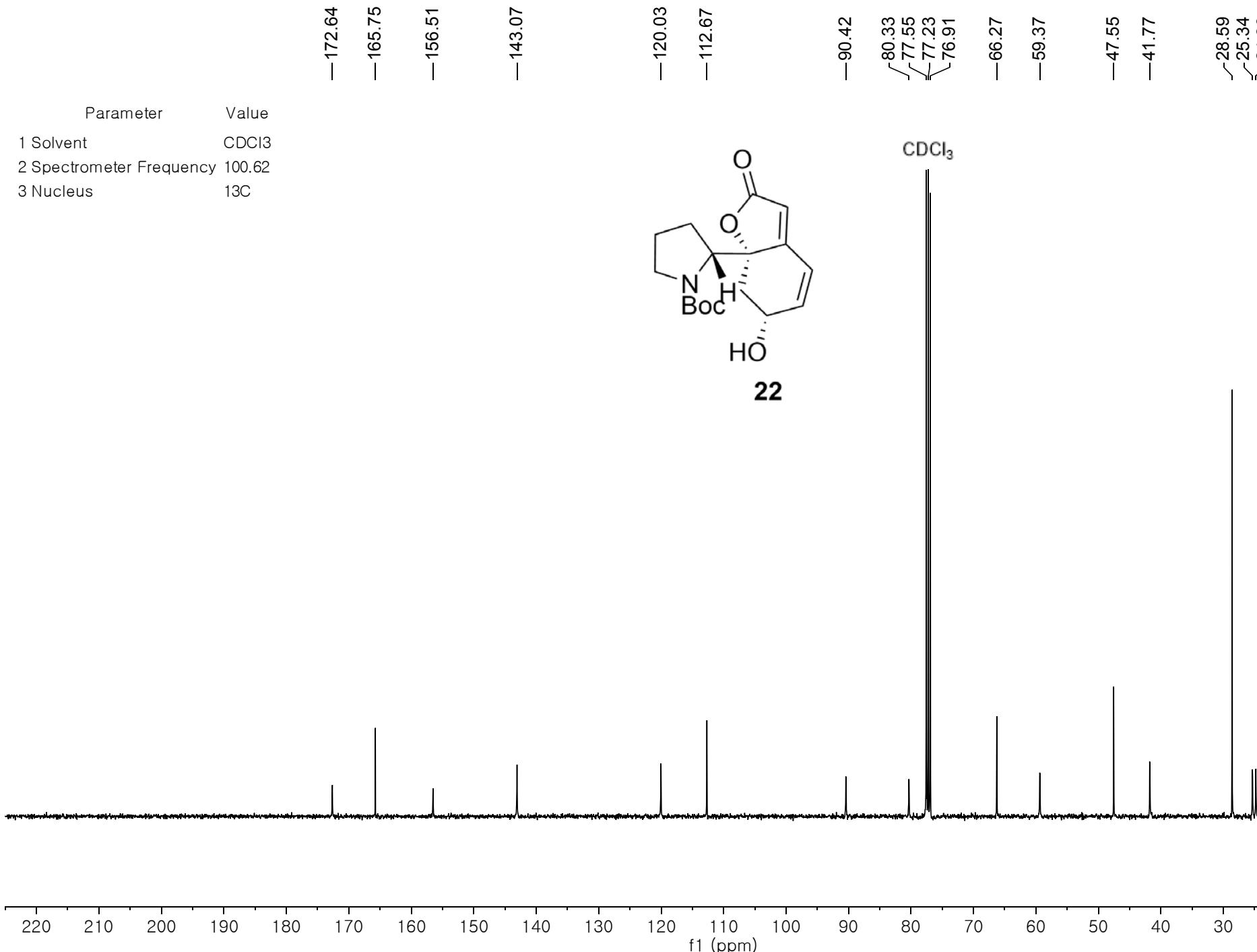


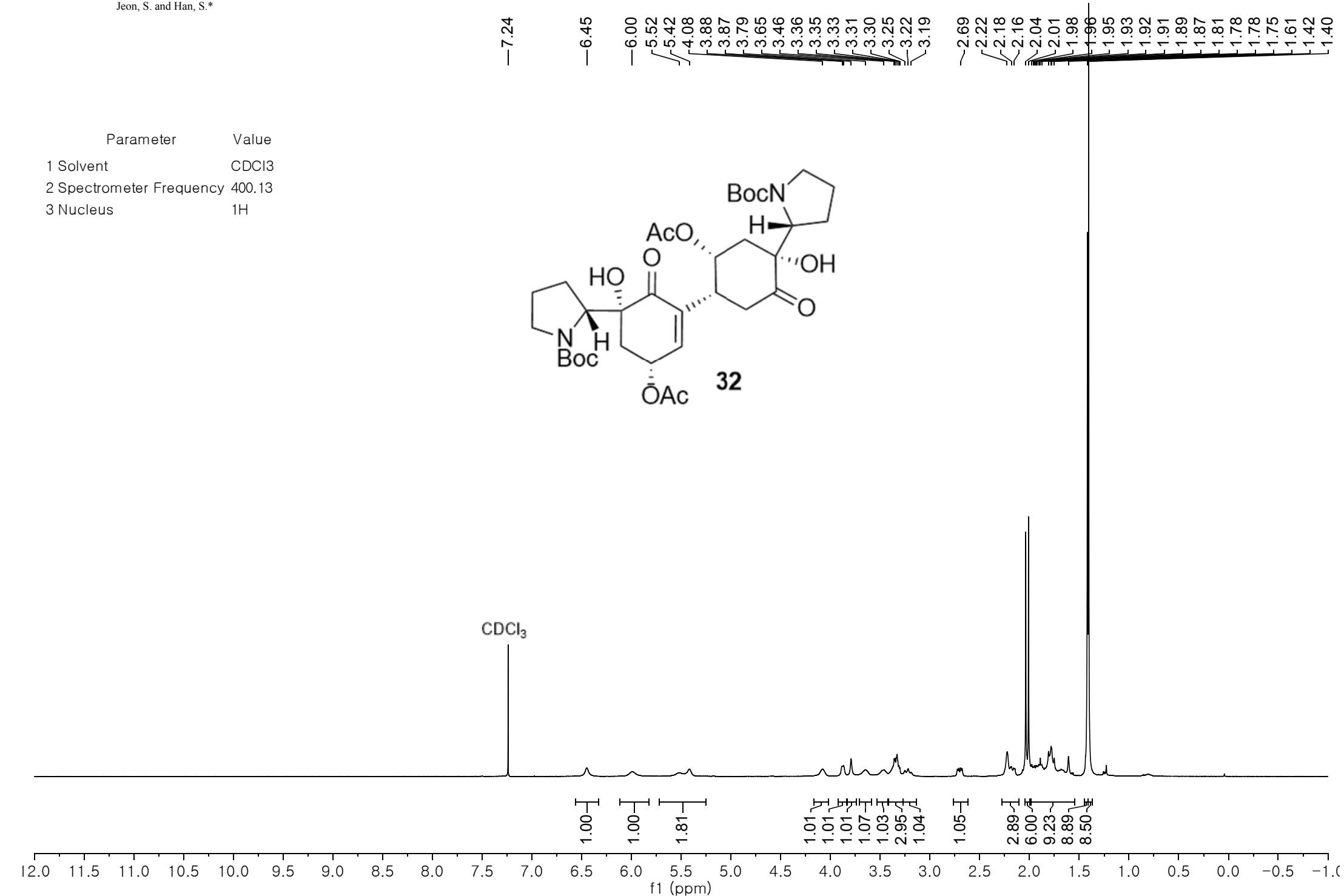


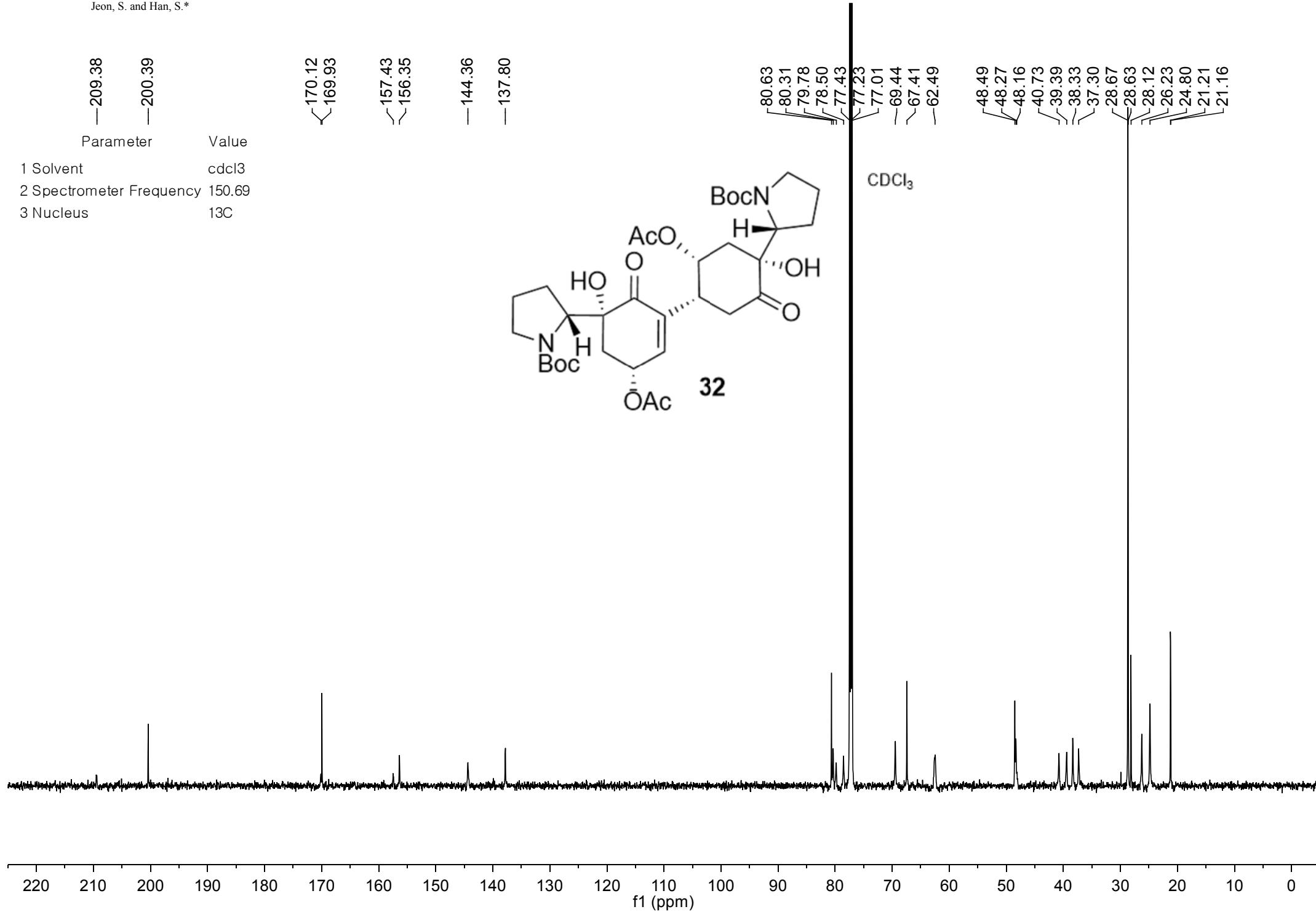












CHIRALPAK IC 1mL/min, 80:20=Hex:EtOH in 15min

=====
Acq. Operator : JH.Jin Seq. Line : 4

Acq. Instrument : Instrument 1 Location : Vial 13

Injection Date : 3/9/2017 4:11:40 PM Inj : 1

Inj Volume : 5 μ l

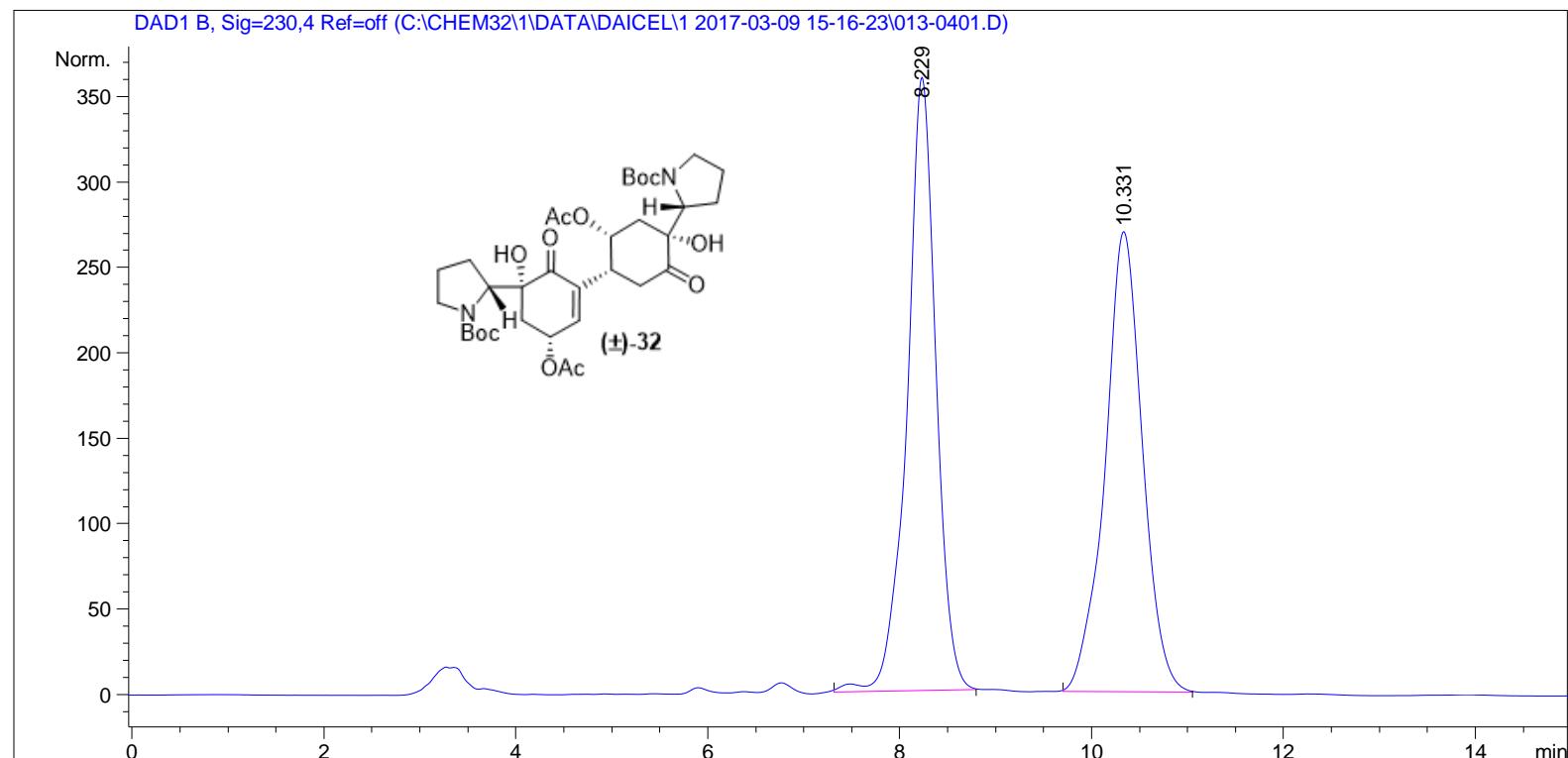
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Last changed : 3/9/2017 4:10:28 PM by JH.Jin
(modified after loading)

Analysis Method : C:\CHEM32\1\DATA\DAICEL\1 2017-03-09 15-16-23\013-0401.D\DA.M (1.M)

Last changed : 3/9/2017 5:25:39 PM by JH.Jin

Sample Info : IC, Hex/EtOH=80/20



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Area Percent Report with Performance
=====

Multiplier : 1.0000

Dilution : 1.0000

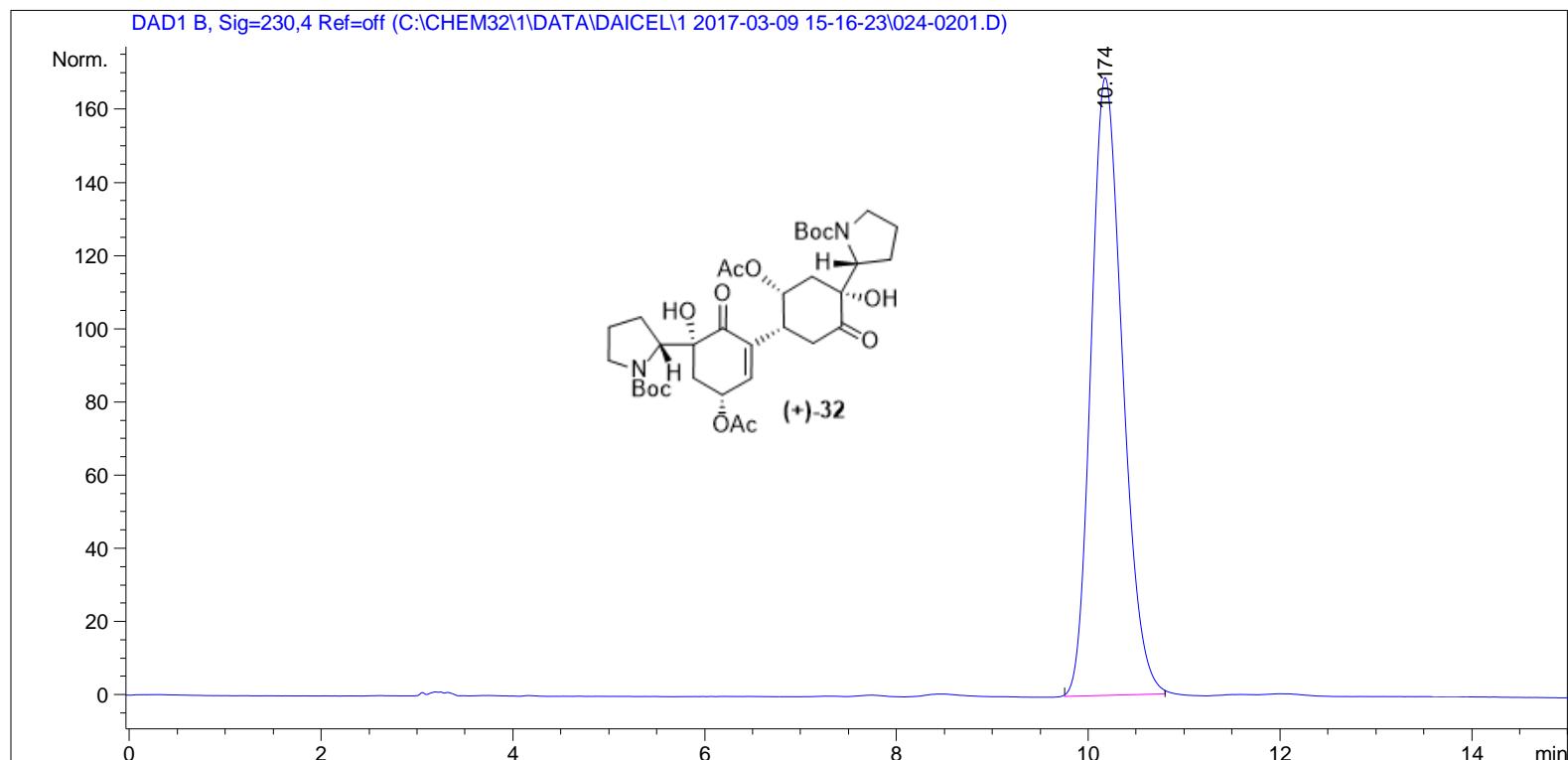
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=230,4 Ref=off

RetTime	k'	Area	Height	Symm.	Width	Plates	Resol	Selectivity
[min]		[mAU*s]	[mAU]		[min]			
8.229	-	7502.57275	358.97012	1.07	0.2933	4360	-	-
10.331	-	7286.40674	269.34409	0.98	0.3933	3822	3.60	1.26

CHIRALPAK IC 1mL/min, 80:20=Hex:EtOH in 15min

=====
Acq. Operator : JH.Jin Seq. Line : 2
Acq. Instrument : Instrument 1 Location : Vial 24
Injection Date : 3/9/2017 3:38:58 PM Inj : 1
Inj Volume : 5 μ l
Acq. Method : C:\Chem32\1\DATA\DAICEL\1 2017-03-09 15-16-23\1.M
Last changed : 3/9/2017 3:53:53 PM by JH.Jin
(modified after loading)
Analysis Method : C:\CHEM32\1\DATA\DAICEL\1 2017-03-09 15-16-23\024-0201.D\DA.M (1.M)
Last changed : 3/9/2017 5:26:05 PM by JH.Jin
Sample Info : IC, Hex/EtOH=80/20



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Area Percent Report with Performance
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Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=230,4 Ref=off

RetTime [min]	k' [min]	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates 4771	Resol -	Selectivity -
10.174	-	3851.31836	168.86855	0.77	0.3467	-	-	-

