Concise Total Synthesis and Stereochemical Revision of (+)-Naseseazines A and B. Regioselective Arylative Dimerization of Diketopiperazine Alkaloids.

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General Procedures. All reactions were performed in oven-dried or flame-dried round-bottom flasks. The flasks were fitted with rubber septa and reactions were conducted under a positive pressure of argon. Gas-tight syringes with stainless steel needles or cannulae were used to transfer air- and moisture-sensitive liquids. Where necessary (so noted), reactions were performed in Schlenk tubes fitted with a PTFE stopcock. Flash column chromatography was performed as described by Still et al. using granular silica gel (60 Å pore size, 40–63 μm, 4–6% H_2O content, Zeochem).[1] Analytical thin layer chromatography (TLC) was performed using glass plates pre-coated with 0.25 mm 230–400 mesh silica gel impregnated with a fluorescent indicator (254 nm). TLC plates were visualized by exposure to short wave ultraviolet light (254 nm) and an aqueous solution of ceric ammonium molybdate (CAM) followed by heating on a hot plate (~250 ºC). Organic solutions were concentrated at 29–30 °C on rotary evaporators capable of achieving a minimum pressure of ~2 torr.

Materials. Commercial reagents and solvents were used as received with the following exceptions: dichloromethane, acetonitrile, tetrahydrofuran, methanol, pyridine, toluene, and triethylamine were purchased from J.T. Baker (Cycletainer™) and were purified by the method of Grubbs et al. under positive argon pressure.[2] Nitroethane was distilled over calcium hydride and stored over 4 Å molecular sieves. 1-Ethyl-3-(3-dimethylaminopropyl) carbodiimide hydrochloride was purchased from Oakwood Products, Inc.; *N*-hydroxybenzotriazole was purchased from Aroz Technologies, LLC; 6-bromo-3-formylindole was purchased from Frontier Scientific, Inc.; (+)-1,2-bis((2*S*,5*S*)-2,5-diethylphospholano)benzene(1,5-cyclooctadiene)rhodium(I) trifluoromethanesulfonate was purchased from Strem Chemicals, Inc.; and (±)-Boc-α-phosphonoglycine trimethyl ester as well as all amino acid derivatives were purchased from Chem-Impex International, Inc. All other solvents and chemicals were purchased from Sigma–Aldrich.

Instrumentation. Proton nuclear magnetic resonance (^{1}H NMR) spectra were recorded with Varian inverse probe INOVA-500 and Varian INOVA-500 spectrometers, are reported in parts per million on the δ scale, and are referenced from the residual protium in the NMR solvent ($CDCl_3$: δ 7.24 ($CHCl_3$), DMSO-d_6: δ 2.50 (DMSO-d_5)). Data are reported as follows: chemical shift [multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet), coupling constant(s) in Hertz, integration, assignment]. Carbon-13 nuclear magnetic resonance (^{13}C NMR) spectra were recorded with a Varian INOVA-500 spectrometer, are reported in parts per million on the δ scale, and are referenced from the carbon resonances of the solvent ($CDCl_3$: δ 77.23, DMSO-d_6: δ 39.51). Data are reported as follows: chemical shift (assignment). Fluorine-19 nuclear magnetic resonance (^{19}F NMR) spectra were recorded with a Varian Mercury 300 spectrometer, are reported in parts per million on the δ scale, and are referenced from the fluorine resonance of neat trichlorofluoromethane ($CFCl_3$: δ 0). Data are reported as follows: chemical shift (assignment). Infrared data (IR) were obtained with a Perkin-Elmer 2000 FTIR, and are reported as follows: frequency of absorption (cm^{-1}), intensity of absorption (s = strong, m = medium, w = weak, br = broad). Optical rotations were measured on a Jasco-1010 polarimeter. UV-Vis spectrophotometric data were acquired on a Varian Cary 50 Bio UV-Vis spectrophotometer. Preprativ HPLC was performed on a Waters system with the 1525 Binary HPLC Pump, 2489 UV/Vis Dtctor, 3100 Mass Detector, System Fluidics Organizer, and 2767 Sample Manager components. We are grateful to Dr. Li Li for obtaining the mass spectrometric data at the Department of Chemistry's Instrumentation Facility, Massachusetts Institute of Technology. High resolution mass spectra (HRMS) were recorded on a Bruker Daltonics APEXIV 4.7 Tesla FT-ICR-MS using either an electrospray (ESI) or direct analysis in real time (DART) ionization source.

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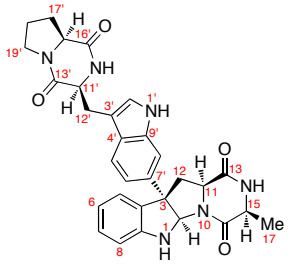
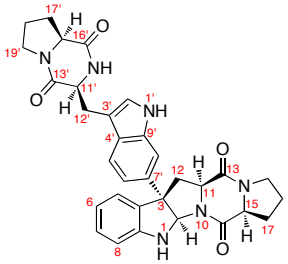
[1] Still, W. C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923.
[2] Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518.

Positional Numbering System. At least three numbering schemes for diketopiperazine alkaloids exist in the literature.[3] In assigning the ^{1}H and ^{13}C NMR data of all intermediates en route to our total syntheses of (+)-naseseazines A and B, we wished to employ a uniform numbering scheme. For ease of direct comparison, particularly between early intermediates and advanced compounds, the numbering scheme used by Barrow for (+)-WIN-64821 (using positional numbers 1–19) was optimal and is used in this document. This numbering system is also consistent with that employed in the isolation paper of (+)-naseseazines A and B.[4] In key instances, the products are accompanied by the numbering system as shown below.



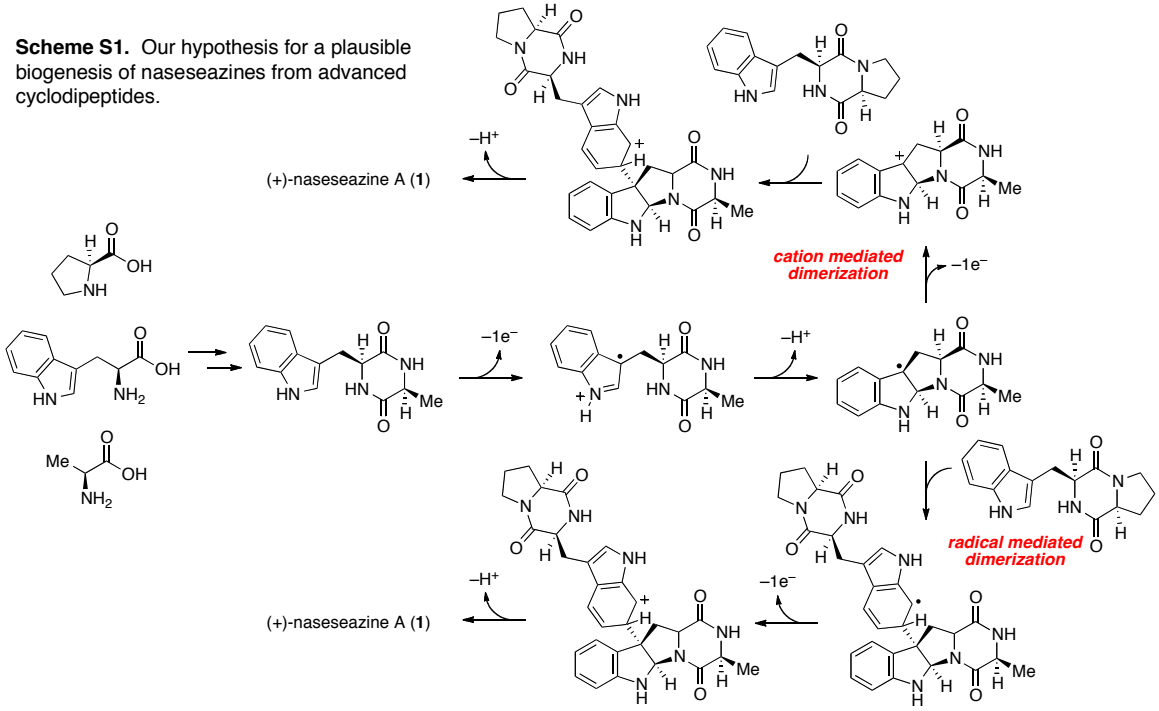
(+)-Naseseazine A (**1**)



(+)-Naseseazine B (**2**)

Biogenetic Hypothesis. We consider a biosynthetic hypothesis in which dimerization occurs at an advanced stage using well-elaborated tryptophan systems, such as cyclodipeptides, to be plausible.[5] Furthermore, a likely dimerization scheme would invoke a cationic mechanism (radical or non-radical) in contrast to that which is suggested in the isolation report.[4] Two contiguous or non-contiguous single-electron oxidation events of an indole core accompanied by a Friedel–Crafts addition of another tryptophan indole could give rise to a number of dimer cognates whose regioisomeric constitution would be controlled by the structure of an enzyme-binding pocket.

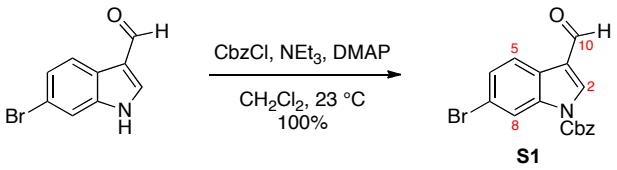
Scheme S1. Our hypothesis for a plausible biogenesis of naseseazines from advanced cyclodipeptides.



[3] (a) Von Hauser, D.; Weber, H. P.; Sigg, H. P. *Helv. Chim. Acta* **1970**, *53*, 1061. (b) Barrow, C. J.; Cai, P.; Snyder, J. K.; Sedlock, D. M.; Sun, H. H.; Cooper, R. *J. Org. Chem.* **1993**, *58*, 6016. (c) Springer, J. P.; Büchi, G.; Kobbe, B.; Demain, A. L.; Clardy, J. *Tetrahedron Lett.* **1977**, *28*, 2403.

[4] Raju, R.; Piggott, A. M.; Conte, M.; Aalbersberg, W. G. L.; Feussner, K.; Capon, R. J. *Org. Lett.* **2009**, *11*, 3862.

[5] For hypotheses on dimeric cyclotryptamine biosynthesis, see: (a) Woodward, R. B.; Yang, N. C.; Katz, T. J.; Clark, V. M.; Harley-Mason, J.; Ingleby, R. F. J.; Sheppard, N. *Proc. Chem. Soc.* **1960**, 76, and (b) Robinson, R.; Teuber, H. J. *Chem. Ind.* **1954**, 783. For labeling and biosynthetic studies, see: (c) Kirby, G. W.; Shah, S. W.; Herbert, E. J. *J. Chem. Soc. C* **1969**, 1916, (d) O'Donovan, D. G.; Keogh, M. F. *J. Chem. Soc. C* **1966**, 1570, and (e) Schutte, H. R.; Maier, B. *Arch. Pharm.* **1965**, *298*, 459. For reviews, see: (f) Kim, J.; Movassaghi, M. *Chem. Soc. Rev.* **2009**, *38*, 3035, (g) Schmidt, M. A.; Movassaghi, M. *Synlett* **2008**, 313, and (h) Steven, A.; Overman, L. E. *Angew. Chem. Int. Ed.* **2007**, *46*, 5488.

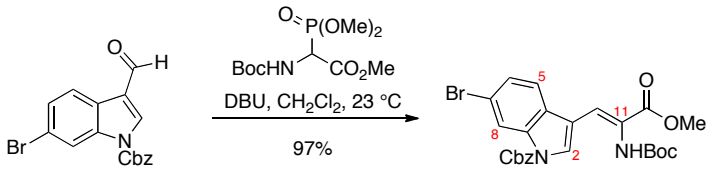


S1

Benzyl 6-bromo-3-formyl-1*H*-indole-1-carboxylate (S1):

Triethylamine (1.98 mL, 14.2 mmol, 1.50 equiv) was added via syringe to a solution of 6-bromo-3-formylindole (2.13 g, 9.49 mmol, 1 equiv) and 4-dimethylaminopyridine (116 mg, 949 μmol, 0.100 equiv) in dichloromethane (25 mL) at 23 °C. Benzyl chloroformate (1.74 mL, 12.3 mmol, 1.30 equiv) was added dropwise to the solution via syringe. After 1 h, another portion of benzyl chloroformate (267 μL, 1.90 mmol, 0.20 equiv) was added via syringe. After 40 min, the reaction mixture was diluted with dichloromethane (100 mL) and washed with saturated aqueous sodium bicarbonate solution (100 mL). The aqueous layer was further extracted with dichloromethane (2 × 25 mL). The combined organic layers were washed with aqueous hydrogen chloride (1 N, 100 mL) and the resulting aqueous layer was extracted with dichloromethane (2 × 25 mL). The combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure. Benzyl 6-bromo-3-formyl-1*H*-indole-1-carboxylate (**S1**) (3.40 g, 100%) was obtained as a white solid. Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

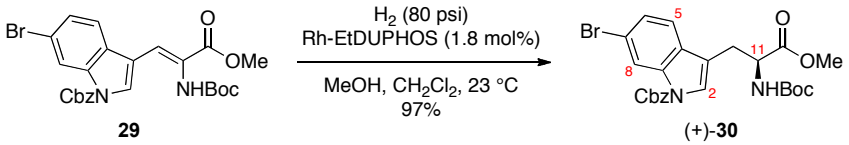
^{1}H NMR (500 MHz, $CDCl_3$, 20 °C):	δ 10.01 (s, 1H, C_{10}**H**), 8.35 (s, 1H, C_8**H**), 8.17 (s, 1H, C_2**H**), 8.09 (d, J = 8.4, 1H, C_5**H**), 7.50–7.38 (m, 5H, Ph_{Cbz}-**H**), 7.38–7.50 (m, 1H, C_6**H**), 5.48 (s, 2H, $Ph_{Cbz}C$**H**$_2$).
^{13}C NMR (125.8 MHz, $CDCl_3$, 20 °C):	δ 185.6 (**C**$_{10}$), 149.9 (**C**=O_{Cbz}), 136.7 (**C**$_9$), 136.1 (**C**$_2$), 134.2 (Ph_{Cbz}-*ipso*-**C**), 129.5 (Ph_{Cbz}-**C**), 129.2 (Ph_{Cbz}-**C**), 129.1 (Ph_{Cbz}-**C**), 128.5 (**C**$_6$), 125.0 (**C**$_4$), 123.5 (**C**$_5$), 122.0 (**C**$_3$), 120.3 (**C**$_7$), 118.5 (**C**$_8$), 70.3 (Ph_{Cbz}**C**H$_2$).
FTIR (thin film) cm^{-1}:	1753 (s), 1679 (s), 1343 (m), 1224 (s), 1092 (m).
HRMS (DART) (*m*/*z*):	calc'd for $C_{17}H_{13}BrNO_3$ $[M+H]^+$: 358.0073, found: 358.0077.
TLC (20% ethyl acetate in hexanes), R*f*:	0.47 (UV, CAM).



(*Z*)-Benzyl 6-bromo-3-(2-((*tert*-butoxycarbonyl)amino)-3-methoxy-3-oxoprop-1-en-1-yl)-1*H*-indole-1-carboxylate (29):

1,8-Diazabicycloundec-7-ene (1.70 mL, 11.4 mmol, 1.20 equiv) was added dropwise to a solution of carboxaldehyde **S1** (3.40 g, 9.49 mmol, 1 equiv) and (±)-Boc-α-phosphonoglycine trimethyl ester (3.39 g, 11.4 mmol, 1.20 equiv) in dichloromethane (32 mL) at 0 ºC. The ice-water bath was then removed and the reaction mixture was allowed to warm to 23 ºC. After 4 h, the reaction mixture was diluted with dichloromethane (100 mL) and washed with aqueous hydrogen chloride (1 N, 100 mL). The resulting aqueous layer was extracted with dichloromethane (2 × 25 mL). The combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure. The resulting oil was purified by flash column chromatography on silica gel (eluent: 20% ethyl acetate in hexanes) to afford the enamide **29** (4.85 g, 97%) as a beige foam. Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

^{1}H NMR (500 MHz, $CDCl_3$, 20 ºC):	δ 8.27 (br-s, 1H, C_8**H**), 7.74 (br-s, 1H, C_2**H**), 7.46–7.33 (m, 1H, C_{12}**H**), 7.46–7.33 (m, 1H, C_5**H**), 7.46–7.33 (m, 5H, Ph_{Cbz}-**H**), 7.30 (dd, *J* = 1.8, 8.4, 1H, C_6**H**), 6.44 (s, 1H, N_{10}**H**), 5.38 (s, 2H, $Ph_{Cbz}C$**H**$_2$), 3.82 (s, 3H, OC**H**$_3$), 1.35 (s, 9H, C(C**H**$_3$)$_3$).
^{13}C NMR (125.8 MHz, $CDCl_3$, 20 ºC):	δ 165.7 (**C**$_{13}$), 152.7 (**C**=O_{Boc}), 150.0 (**C**=O_{Cbz}), 135.4 (**C**$_9$), 134.5 (Ph_{Cbz}-*ipso*-**C**), 129.0 (Ph_{Cbz}-**C**), 128.8 (Ph_{Cbz}-**C**), 128.8 (Ph_{Cbz}-**C**), 128.3 (**C**$_{11}$), 127.1 (**C**$_2$), 126.7 (**C**$_6$), 124.2 (**C**$_4$), 121.4 (**C**$_{12}$), 120.2 (**C**$_5$), 118.9 (**C**$_7$), 118.3 (**C**$_8$), 114.8 (**C**$_3$), 81.0 (**C**$(CH_3)_3$), 69.5 (Ph_{Cbz}**C**H_2), 52.6 (O**C**H_3), 28.1 (C(**C**H_3)$_3$).
FTIR (thin film) cm^{-1}:	3324 (br, m), 2979 (m), 1723 (s), 1244 (s), 759 (m).
HRMS (DART) (*m*/*z*):	calc'd for $C_{25}H_{25}BrN_2O_6$ $[M]^+$: 528.0891, found: 528.0877.
TLC (20% ethyl acetate in hexanes), R*f*:	0.31 (UV, CAM).



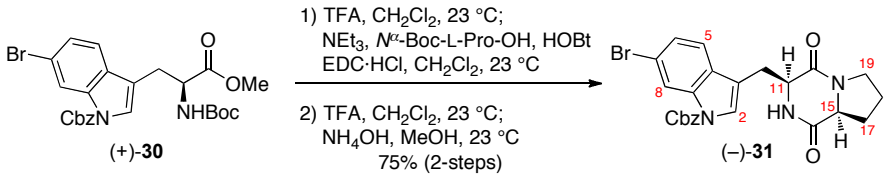
29 (+)-**30**

<u>N^{α}-Boc-N^{in}-Cbz-6-bromotryptophan methyl ester (+)-30:</u>

Dichloromethane (7.5 mL) and methanol (7.5 mL) were sequentially added via syringe to a Fischer-Porter tube charged with enamide **29** (4.00 g, 7.56 mmol, 1 equiv) and (+)-1,2-bis((2*S*,5*S*)-2,5-diethylphospholano)benzene(1,5-cyclooctadiene)rhodium(I) trifluoromethanesulfonate (100 mg, 138 μmol, 1.83 mol%). The reaction vessel was charged with hydrogen gas (80 psi) and then discharged (4 cycles). The vessel was charged a final time with hydrogen gas (80 psi) then sealed. After 9.5 h, the Fischer-Porter tube was depressurized and the reaction mixture was concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography on silica gel (eluent: 25% ethyl acetate in hexanes) to afford bromotryptophan derivative (+)-**30** (3.88 g, 96.6 %, >99% ee)[6] as a white foam. Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

^{1}H NMR (500 MHz, $CDCl_3$, 20 °C):	δ 8.35 (br-s, 1H, C_8**H**), 7.48–7.30 (m, 1H, C_2**H**), 7.48–7.30 (m, 1H, C_5**H**), 7.48–7.30 (m, 1H, C_6**H**), 7.48–7.30 (m, 5H, Ph_{Cbz}-**H**), 5.41 (app-s, 2H, $Ph_{Cbz}C$**H**$_2$), 5.11 (d, *J* = 7.8, 1H, N_{10}**H**), 4.60 (app-q, *J* = 5.8, 1H, C_{11}**H**), 3.64 (s, 3H, OC**H**$_3$), 3.21 (dd, *J* = 5.5, 14.8, 1H, C_{12}**H**$_a$), 3.10 (dd, *J* = 5.5, 14.8, 1H, C_{12}**H**$_b$), 1.38 (s, 9H, C(C**H**$_3$)$_3$).
^{13}C NMR (125.8 MHz, $CDCl_3$, 20 °C):	δ 172.3 (**C**$_{13}$), 155.2 (**C**=O_{Boc}), 150.4 (**C**=O_{Cbz}), 136.2 (**C**$_9$), 135.0 (Ph_{Cbz}-*ipso*-**C**), 129.6 (**C**$_4$), 129.0 (Ph_{Cbz}-**C**), 129.0 (Ph_{Cbz}-**C**), 128.7 (Ph_{Cbz}-**C**), 126.4 (**C**$_6$), 124.2 (**C**$_2$), 120.3 (**C**$_5$), 118.8 (**C**$_7$), 118.6 (**C**$_8$), 116.2 (**C**$_3$), 80.3 (**C**$(CH_3)_3$), 69.2 (Ph_{Cbz}**C**H_2), 53.7 (**C**$_{11}$), 52.6 (O**C**H_3), 28.4 (C(**C**$H_3)_3$), 27.9 (**C**$_{12}$).
FTIR (thin film) cm^{-1}:	3369 (m), 2978 (m), 1742 (s), 1715 (s), 1247 (s).
HRMS (DART) (*m*/*z*):	calc'd for $C_{25}H_{27}BrN_2O_6$ $[M]^+$: 530.1047, found: 530.1071.
$[\alpha]_D^{24}$:	+32 (*c* = 0.19, $CHCl_3$).
TLC (25% ethyl acetate in hexanes), R*f*:	0.37 (UV, CAM).

[6] In order to determine the enantiopurity, bromotryptophan derivative (+)-**30** was hydrogenated using palladium on carbon in acetic acid to remove the *N*-carboxybenzyl and aryl bromide functional groups. The product was then compared against commercially available enantioenriched *N*-Boc-L-Trp-OMe and *N*-Boc-D-Trp-OMe standards. Chiral HPLC analysis [Chiralpak IC column; 1.0 mL/min; 15% isopropanol in hexanes; t_R(*N*-Boc-L-Trp-OMe) = 10.6 min, t_R(*N*-Boc-D-Trp-OMe) = 13.5 min] showed the enantiopurity to be >99% ee.



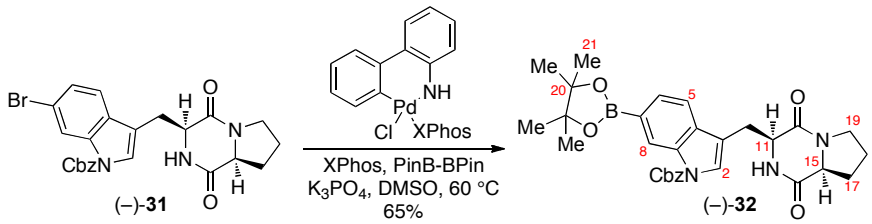
Bromoindole diketopiperazine (–)-31:

Trifluoroacetic acid (15 mL) was added via syringe to a solution of N^{α}-Boc-N^{in}-Cbz-L-6-bromotryptophan methyl ester ((+)-**30**) (3.83 g, 7.21 mmol, 1 equiv) in dichloromethane (50 mL) at 23 °C. After 1 h, the brown solution was concentrated under reduced pressure to afford a viscous brown residue, which was dissolved in dichloromethane (100 mL) and cooled to 0 °C in an ice-water bath. Triethylamine (4.52 mL, 32.5 mmol, 4.50 equiv) was added dropwise via syringe. *N*-hydroxybenzotriazole (1.46 g, 10.8 mmol, 1.50 equiv) and N^{α}-Boc-L-proline (3.10 g, 14.4 mmol, 2.00 equiv) were sequentially added to the solution. After 2 min, upon dissolution of the N^{α}-Boc-L-proline, 1-ethyl-3-(3-dimethylaminopropyl) carbodiimide hydrogen chloride (2.07 g, 10.8 mmol, 1.50 equiv) was added and the reaction mixture was allowed to warm to 23 °C. After 1 h, the reaction mixture was diluted with dichloromethane (60 mL) and washed with aqueous hydrogen chloride solution (1 N, 250 mL). The aqueous layer was extracted with dichloromethane (2 × 100 mL). The combined organic layers were washed with saturated aqueous sodium bicarbonate solution (250 mL) and the resulting aqueous layer was extracted with dichloromethane (2 × 25 mL). The combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure. The resulting off-white foam was then dissolved in dichloromethane (50 mL) and trifluoroacetic acid (15 mL) was added dropwise to the solution. After 1 h, the solution was concentrated under reduced pressure. The viscous residue was dissolved in methanol (200 mL) and cooled to 0 °C. Ammonium hydroxide (28–30% ammonia, 10 mL) was added dropwise and the reaction mixture was allowed to warm to 23 °C. After 22 h, the reaction mixture was cooled to –78 °C and filtered. The white precipitate was washed with methanol (2 × 50 mL), which had been cooled to 0 °C in an ice-water bath, and was dried under reduced pressure at 23 °C to afford diketopiperazine (–)-**31** (2.68 g, 74.9%, 2-steps) as a fluffy white solid. Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

^{1}H NMR (500 MHz, DMSO-d_6, 20 °C): δ 8.21 (s, 1H, C_8**H**), 8.13 (s, 1H, N_{10}**H**), 7.66 (d, J = 8.4, 1H, C_5**H**), 7.61 (s, 1H, C_2**H**), 7.53 (d, J = 6.8, 2H, Ph_{Cbz}-*o*-**H**), 7.47–7.37 (m, 1H, C_6**H**), 7.47–7.37 (m, 3H, Ph_{Cbz}-**H**), 5.46 (app-s, 2H, $Ph_{Cbz}C$**H**$_2$), 4.42 (app-t, J = 5.2, 1H, C_{11}**H**), 4.11 (app-t, J = 8.5, 1H, C_{15}**H**), 3.38–3.24 (m, 2H, C_{19}**H**), 3.17 (dd, J = 5.2, 15.3, 1H, C_{12}**H**$_a$), 3.05 (dd, J = 5.2, 15.3, 1H, C_{12}**H**$_b$), 2.10–2.00 (m, 1H, C_{17}**H**$_a$), 1.80–1.63 (m, 2H, C_{18}**H**), 1.61–1.47 (m, 1H, C_{17}**H**$_b$).

^{13}C NMR (125.8 MHz, DMSO-d_6, 20 °C): δ 169.3 (**C**$_{16}$), 165.1 (**C**$_{13}$), 149.9 (**C**=O_{Cbz}), 135.3 (**C**$_9$), 135.2 (Ph_{Cbz}-*ipso*-**C**), 129.6 (**C**$_4$), 128.7 (Ph_{Cbz}-**C**), 128.6 (Ph_{Cbz}-**C**), 128.4 (Ph_{Cbz}-**C**), 125.6 (**C**$_6$), 124.9 (**C**$_2$), 121.7 (**C**$_5$), 117.3 (**C**$_7$), 117.1 (**C**$_8$), 116.8 (**C**$_3$), 68.6 (Ph_{Cbz}**C**H$_2$), 58.5 (**C**$_{15}$), 54.3 (**C**$_{11}$), 44.7 (**C**$_{19}$), 27.8 (**C**$_{17}$), 24.8 (**C**$_{12}$), 22.0 (**C**$_{18}$).

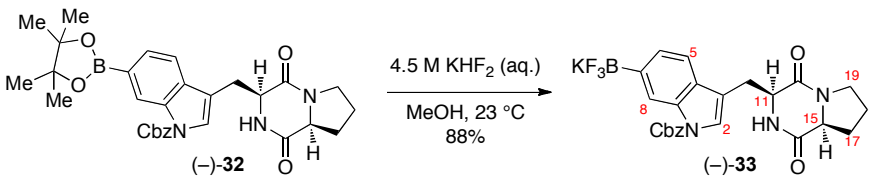
FTIR (thin film) cm^{-1}:	3584 (s), 1737 (s), 1668 (s), 1434 (m), 1247 (m), 1084 (w).
HRMS (DART) (*m*/*z*):	calc'd for $C_{24}H_{23}BrN_3O_4$ $[M+H]^+$: 496.0866, found: 496.0870.
$[\alpha]_D^{24}$:	–33 (*c* = 0.09, DMSO).
TLC (30% acetone in dichloromethane), R*f*:	0.31 (UV, CAM).



Pinacol boronic ester (–)-32:

A 25-mL round bottom flask was charged with diketopiperazine (–)-**31** (500 mg, 1.01 mmol, 1 equiv), aminobiphenyl(XPhos)palladium chloride precatalyst complex (39.6 mg, 50.4 μmol, 5.00 mol%), XPhos (72.2 mg, 151 μmol, 15.0 mol%), bis(pinacolato)diboron (769 mg, 3.03 mmol, 3.00 equiv), and tribasic potassium phosphate (643 mg, 3.03 mmol, 3.00 equiv). The flask was then evacuated and charged with argon (3 cycles). Dimethyl sulfoxide (10 mL) was introduced to the flask via syringe and the initial suspension was warmed to 60 ºC. After 2.5 h, the black solution was cooled to 23 ºC, diluted with ethyl acetate (125 mL), and washed with saturated aqueous sodium bicarbonate solution (250 mL). The resulting aqueous layer was extracted with ethyl acetate (2 × 125 mL), and the combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure. The crude reaction mixture was then purified by flash column chromatography on silica gel (eluent: 3% acetone in ethyl acetate) to afford pinacol boronic ester (–)-**32** (356 mg, 65.0%) as a white foam. Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

^{1}H NMR (500 MHz, $CDCl_3$, 20 ºC):	δ 8.61 (br-s, 1H, C_8**H**), 7.65 (d, *J* = 7.8, 1H, C_6**H**), 7.59 (s, 1H, C_2**H**), 7.51 (d, *J* = 7.9, 1H, C_5**H**), 7.42 (d, *J* = 7.1, 2H, Ph_{Cbz}-*o*-**H**), 7.36–7.26 (m, 3H, Ph_{Cbz}-**H**), 6.79 (s, 1H, N_{10}**H**), 5.35 (app-s, 2H, $Ph_{Cbz}C$**H**$_2$), 4.25 (app-d, *J* = 7.2, 1H, C_{11}**H**), 3.84 (app-t, *J* = 7.1, 1H, C_{15}**H**), 3.56 (dd, *J* = 2.7, 15.2, 1H, C_{12}**H**$_a$), 3.53–3.37 (m, 2H, C_{19}**H**), 2.90 (dd, *J* = 9.5, 15.3, 1H, C_{12}**H**$_b$), 2.17–2.05 (m, 1H, C_{17}**H**$_a$), 1.88–1.64 (m, 1H, C_{17}**H**$_b$), 1.88–1.64 (m, 2H, C_{18}**H**), 1.29 (s, 12H, $OC(C$**H**$_3)_{2pinacol}$).
^{13}C NMR (125.8 MHz, $CDCl_3$, 20 ºC):	δ 169.8 (**C**$_{16}$), 165.0 (**C**$_{13}$), 150.3 (**C**=O_{Cbz}), 135.3 (**C**$_9$), 135.0 (Ph_{Cbz}-*ipso*-**C**), 132.3 (**C**$_4$), 129.0 (**C**$_6$), 128.6 (Ph_{Cbz}-**C**), 128.6 (Ph_{Cbz}-**C**), 128.4 (Ph_{Cbz}-**C**), 125.4 (**C**$_2$), 125.4 (**C**$_7$), 121.8 (**C**$_8$), 118.2 (**C**$_5$), 116.1 (**C**$_3$), 83.7 (O**C**$(CH_3)_{2pinacol}$), 68.7 (Ph_{Cbz}**C**H_2), 58.9 (**C**$_{15}$), 54.3 (**C**$_{11}$), 45.3 (**C**$_{19}$), 28.0 (**C**$_{17}$), 26.1 (**C**$_{12}$), 24.8 (OC(**C**$H_3)_{2pinacol}$), 22.5 (**C**$_{18}$).
FTIR (thin film) cm^{-1}:	2978 (m), 1738 (s), 1673 (s), 1432 (s), 1354 (s).
HRMS (DART) (*m*/*z*):	calc'd for $C_{30}H_{35}BN_3O_6$ $[M+H]^+$: 544.2613, found: 544.2613.
$[\alpha]_D^{23}$:	–63 (*c* = 0.2, $CHCl_3$).
TLC (3% acetone in ethyl acetate), R*f*:	0.20 (UV, CAM).

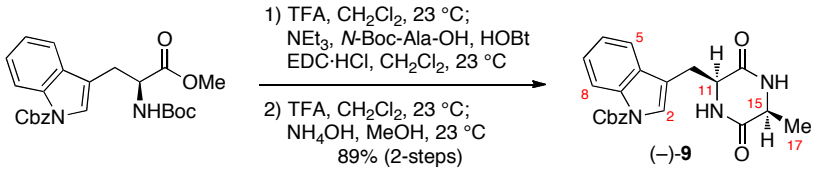


Potassium trifluoroborateindole (–)-33:

An aqueous solution of potassium hydrogen fluoride (4.5 M, 4.73 mL, 21.3 mmol, 20.0 equiv) was added dropwise to a solution of pinacol boronic ester (–)-**32** (578 mg, 1.06 mmol, 1 equiv) in methanol (15 mL) at 23 ºC. After 1.5 h, the turbid solution was concentrated to dryness under reduced pressure. The white solid residue was suspended in a dichloromethane and hexanes solution (1:1, 50 mL) and filtered over Celite to remove the pinacol byproduct. After further washing with a dichloromethane and hexanes solution (1:1, 50 mL), the solids were extracted with acetone (80 mL) and the filtrate was concentrated under reduced pressure to afford potassium 6-trifluoroborateindole (–)-**33** (489 mg, 87.8%) as a beige solid. Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

^{1}H NMR (500 MHz, acetone-d_6, 20 ºC):	δ 8.40 (s, 1H, C_8**H**), 7.54 (d, J = 7.3, 2H, Ph_{Cbz}-*o*-**H**), 7.52 (s, 1H, C_2**H**), 7.45 (d, J = 7.7, 1H, C_6**H**), 7.43–7.39 (m, 3H, Ph_{Cbz}-**H**), 7.36 (d, J = 7.5, 1H, C_5**H**), 6.79 (s, 1H, N_{10}**H**), 5.44 (app-s, 2H, $Ph_{Cbz}C$**H**$_2$), 4.41 (dd, J = 3.6, 8.3, 1H, C_{11}**H**), 4.10 (app-t, J = 8.1, 1H, C_{15}**H**), 3.54–3.43 (m, 1H, C_{12}**H**$_a$), 3.54–3.43 (m, 1H, C_{19}**H**$_a$), 3.42–3.33 (m, 1H, C_{19}**H**$_b$), 2.96 (dd, J = 8.3, 15.5, 1H, C_{12}**H**$_b$), 2.18–2.07 (m, 1H, C_{17}**H**$_a$), 1.85–1.70 (m, 1H, C_{17}**H**$_b$), 1.85–1.70 (m, 2H, C_{18}**H**).
^{13}C NMR (125.8 MHz, acetone-d_6, 20 ºC)[7]:	δ 170.1 (**C**$_{16}$), 166.1 (**C**$_{13}$), 151.3 (**C**$_{Cbz}$=O), 136.9 (Ph_{Cbz}-*ipso*-**C**), 136.5 (**C**$_9$), 129.3 (Ph_{Cbz}-**C**), 129.0 (Ph_{Cbz}-**C**), 129.0 (Ph_{Cbz}-**C**), 129.0 (**C**$_4$), 127.7 (**C**$_6$), 123.0 (**C**$_2$), 118.5 (**C**$_8$), 117.6 (**C**$_3$), 117.5 (**C**$_5$), 68.4 (Ph_{Cbz}**C**H$_2$), 59.5 (**C**$_{15}$), 55.2 (**C**$_{11}$), 45.6 (**C**$_{19}$), 28.7 (**C**$_{17}$), 26.6 (**C**$_{12}$), 23.0 (**C**$_{18}$).
^{19}F NMR (282.4 MHz, acetone-d_6, 20 ºC):	δ –141.0 (C_6B**F**$_3$K).
FTIR (thin film) cm^{-1}:	3583 (br, s), 1726 (m), 1666 (s), 1422 (m), 1255 (m).
HRMS (ESI) (*m*/*z*):	calc'd for $C_{24}H_{22}BF_3N_3O_4$ [M–K]$^-$: 484.1661, found: 484.1643.
$[\alpha]_D^{21}$:	–42 (*c* = 0.14, acetone).

[7] C_7 is not observed in the ^{13}C NMR spectrum. For other examples of unobservable carbon atoms *ipso* to the trifluoroborate functional group, see: Molander, G.; Argintaru, O. A.; Aron, I.; Dreher, S. D. *Org. Lett.* **2010**, *12*, 5783.



Alanine diketopiperazine (–)-(9):

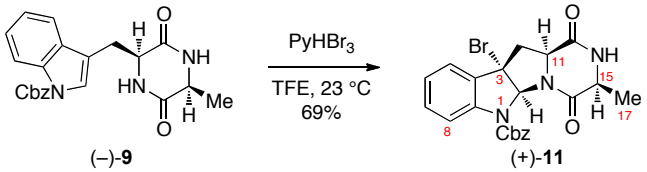
Trifluoroacetic acid (30 mL) was added via syringe to a solution of N^{α}-Boc-N^{in}-Cbz-L-tryptophan methyl ester[8] (9.0 g, 19.9 mmol, 1 equiv) in dichloromethane (100 mL) at 23 °C. After 1 h, the brown solution was concentrated under reduced pressure to afford a viscous brown residue, which was dissolved in dichloromethane (200 mL) and cooled to 0 °C in an ice-water bath. Triethylamine (12.5 mL, 89.5 mmol, 4.50 equiv) was added dropwise via syringe. *N*-hydroxybenzotriazole (4.03 g, 29.8 mmol, 1.50 equiv) and N^{α}-Boc-L-alanine (7.53 g, 39.8 mmol, 2.00 equiv) were sequentially added to the resulting solution. After 2 min, upon dissolution of the N^{α}-Boc-L-alanine, 1-ethyl-3-(3-dimethylaminopropyl) carbodiimide hydrogen chloride (5.72 g, 29.8 mmol, 1.50 equiv) was added and the reaction mixture was allowed to warm to 23 °C. After 13.5 h, aqueous hydrogen chloride solution (1 N, 500 mL) was added and the aqueous layer was extracted with dichloromethane (2 × 100 mL). The combined organic layers were washed with saturated aqueous sodium bicarbonate solution (500 mL) and the resulting aqueous layer was extracted with dichloromethane (2 × 100 mL). The combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure. The resulting off-white foam was then dissolved in dichloromethane (100 mL) and trifluoroacetic acid (30 mL) was added dropwise to the solution. After 2 h, the solution was concentrated under reduced pressure. The viscous residue was dissolved in methanol (300 mL) and cooled to 0 °C. Ammonium hydroxide (28–30% ammonia, 10.5 mL) was added dropwise and the reaction mixture was allowed to warm to 23 °C. After 23 h, the white precipitate was filtered and washed with methanol (2 × 50 mL), which had been cooled to 0 °C in an ice-water bath. The white powder was dried under reduced pressure at 23 °C to afford diketopiperazine (–)-**9** (6.7 g, 89%, 2-steps). Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

^{1}H NMR (500 MHz, DMSO-d_6, 20 °C): δ 8.17 (s, 1H, N_{14}**H**), 8.10 (s, 1H, N_{10}**H**), 8.06 (d, J = 8.1, 1H, C_8**H**), 7.67 (d, J = 7.8, 1H, C_5**H**), 7.51 (d, J = 6.3, 2H, Ph_{Cbz}-*o*-**H**), 7.51 (s, 1H, C_2**H**), 7.43 (app-t, J = 7.0, 2H, Ph_{Cbz}-*m*-**H**), 7.38 (t, J = 7.1, 1H, Ph_{Cbz}-*p*-**H**), 7.32 (app-t, J = 7.3, 1H, C_7**H**), 7.25 (app-t, J = 7.9, 1H, C_6**H**), 5.47 (app-s, 2H, $Ph_{Cbz}C$**H**$_2$), 4.22 (app-t, J = 4.3, 1H, C_{11}**H**), 3.71 (q, J = 7.0, 1H, C_{15}**H**), 3.20 (dd, J = 4.4, 14.7, 1H, C_{12}**H**$_a$), 3.03 (dd, J = 4.6, 14.6, 1H, C_{12}**H**$_b$), 0.66 (d, J = 7.0, 3H, C_{17}**H**).

^{13}C NMR (125.8 MHz, DMSO-d_6, 20 °C): δ 168.0 (**C**$_{16}$), 166.6 (**C**$_{13}$), 150.1 (**C**=O_{Cbz}), 135.4 (Ph_{Cbz}-*ipso*-**C**), 134.6 (**C**$_9$), 130.6 (**C**$_4$), 128.7 (Ph_{Cbz}-**C**), 128.5 (Ph_{Cbz}-**C**), 128.2 (Ph_{Cbz}-**C**), 124.6 (**C**$_7$), 124.3 (**C**$_2$), 122.8 (**C**$_6$), 120.0 (**C**$_5$), 116.2

[8] Prepared from commercially available N^{α}-Boc-tryptophan methyl ester in one step: Kiso, Y.; Inai, M.; Kitagawa, K.; Akita, T. *Chem. Lett.* **1983**, *5*, 739.

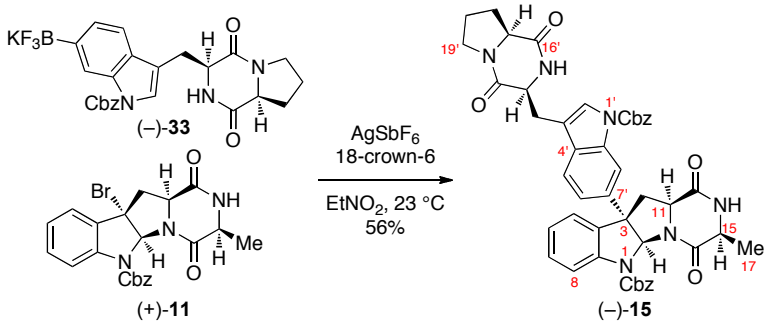
	(**C**$_3$), 114.4 (**C**$_8$), 68.3 (Ph$_{Cbz}$**C**H$_2$), 54.4 (**C**$_{11}$), 49.7 (**C**$_{15}$), 27.9 (**C**$_{12}$), 19.5 (**C**$_{17}$).
FTIR (thin film) cm^{-1}:	3584 (m), 3050 (br, s), 1739 (m), 1678 (s), 1453 (m), 1244 (m).
HRMS (DART) (*m*/*z*):	calc'd for $C_{22}H_{20}N_3O_4$ $[M-H]^-$: 390.1459, found: 390.1458.
$[\alpha]_D^{24}$:	–12 (*c* = 0.17, DMSO).
TLC (5% methanol in dichloromethane), R*f*:	0.59 (UV, CAM).



Tetracyclic bromide A (+)-11:

Pyridinium tribromide (4.24 g, 13.3 mmol, 2.00 equiv) was added to a suspension of diketopiperazine (–)-**9** (2.50 g, 6.62 mmol, 1 equiv) in 2,2,2-trifluoroethanol (132 mL) at 23 ºC. After 20 min, once the white solids have fully dissolved, a solution of saturated aqueous sodium thiosulfate (100 mL) was added. The reaction mixture was diluted with dichloromethane (200 mL) and washed with saturated aqueous sodium thiosulfate (200 mL). The resulting aqueous solution was further extracted with dichloromethane (2 × 150 mL) and the combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure. The crude reaction mixture was then purified by flash column chromatography on silica gel (eluent: 60% ethyl acetate in hexanes) to afford tetracyclic bromide (+)-**11** (2.08 g, 69.2%) as a white foam. Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

^{1}H NMR (500 MHz, $CDCl_3$, 20 ºC):	δ 7.65 (d, *J* = 8.1, 1H, C_8**H**), 7.44 (d, *J* = 7.2, 2H, Ph_{Cbz}-*o*-**H**), 7.40 (d, *J* = 7.6, 1H, C_5**H**), 7.37 (app-t, *J* = 7.0, 2H, Ph_{Cbz}-*m*-**H**), 7.32 (t, *J* = 7.2, 1H, Ph_{Cbz}-*p*-**H**), 7.24 (app-t, *J* = 7.6, 1H, C_7**H**), 7.06 (app-t, *J* = 7.5, 1H, C_6**H**), 6.32 (s, 1H, C_2**H**), 6.21 (s, 1H, N_{14}**H**), 5.42 (d, *J* = 12.2, 1H, $Ph_{Cbz}C$**H**$_a$), 5.32 (d, *J* = 12.2, 1H, $Ph_{Cbz}C$**H**$_b$), 4.32 (dd, *J* = 3.5, 10.1, 1H, C_{11}**H**), 3.93 (q, *J* = 6.7, 1H, C_{15}**H**), 3.69 (dd, *J* = 3.5, 14.1, 1H, C_{12}**H**$_a$), 3.06 (dd, *J* = 10.2, 14.2, 1H, C_{12}**H**$_b$), 1.29 (d, *J* = 6.8, 3H, C_{17}**H**).
^{13}C NMR (125.8 MHz, $CDCl_3$, 20 ºC):	δ 168.9 (**C**$_{13}$), 167.3 (**C**$_{16}$), 152.9 (**C**=O_{Cbz}), 139.4 (**C**$_9$), 135.8 (Ph_{Cbz}-*ipso*-**C**),), 132.7 (**C**$_4$), 130.9 (**C**$_7$), 128.6 (Ph_{Cbz}-**C**), 128.3 (Ph_{Cbz}-**C**), 128.2 (Ph_{Cbz}-**C**), 124.7 (**C**$_6$), 124.6 (**C**$_5$), 116.8 (**C**$_8$), 84.6 (**C**$_2$), 68.3 (Ph_{Cbz}**C**H_2), 60.3 (**C**$_3$), 58.0 (**C**$_{11}$), 51.4 (**C**$_{15}$), 37.6 (**C**$_{12}$), 14.8 (**C**$_{17}$).
FTIR (thin film) cm^{-1}:	3273 (br-m), 1720 (s), 1692 (s), 1408 (m), 752 (m).
HRMS (ESI) (*m*/*z*):	calc'd for $C_{22}H_{21}BrN_3O_4$ $[M+H]^+$: 470.0710, found 470.0692.
$[\alpha]_D^{25}$:	+71 (*c* = 0.09, $CHCl_3$).
TLC (60% ethyl acetate in hexanes), R*f*:	0.20 (UV, CAM).



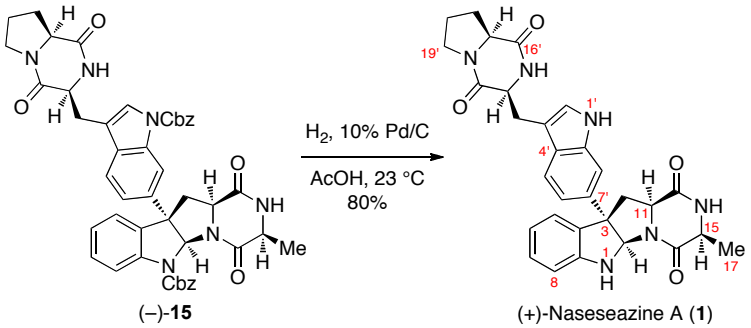
(–)-N^{in},$N^{in'}$-Dicarboxybenzyl Naseseazine A (15):

Silver(I) hexafluoroantimonate (73.1 mg, 213 μmol, 5.00 equiv) was added as a solid to a solution of tetracyclic bromide (+)-**11** (20.0 mg, 42.5 μmol, 1 equiv), potassium 6-trifluoroborateindole (–)-**33** (33.4 mg, 63.8 μmol, 1.50 equiv), and 18-crown-6 (56.2 mg, 213 μmol, 5.00 equiv) in nitroethane (2 mL) at 23 ºC. After 1 h, aqueous hydrogen chloride (2 N, 2 mL) was added. After stirring for 5 min, the reaction mixture was diluted with dichloromethane (60 mL) and washed with aqueous hydrogen chloride (2 N, 60 mL). The resulting aqueous layer was further extracted with dichloromethane (2 × 30 mL) and the combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography on silica gel (eluent: 8% methanol in ethyl acetate) to afford (–)-N^{in},$N^{in'}$-dicarboxybenzyl naseseazine A (**15**) (19.3 mg, 56.2%) as a white solid. Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

^{1}H NMR (500 MHz, $CDCl_3$, 20 ºC): δ 8.11 (br-s, 1H, $C_{8'}$**H**), 7.62 (d, J = 7.8, 1H, C_8**H**), 7.46 (s, 1H, $C_{2'}$**H**), 7.40 (d, J = 7.0, 2H, Ph_{Cbz}-*o*-**H**), 7.37–7.28 (m, 5H, Ph_{Cbz}-**H**), 7.37–7.28 (m, 1H, $C_{5'}$**H**), 7.28–7.20 (m, 3H, Ph_{Cbz}-**H**), 7.15(app-t, J = 7.6, 1H, C_7**H**), 7.05 (d, J = 7.2, 1H, C_5**H**), 6.93 (app-t, J = 7.4, 1H, C_6**H**), 6.90 (d, J = 8.4, 1H, $C_{6'}$**H**), 6.41 (s, 1H, $N_{10'}$**H**), 6.28 (s, 1H, N_{14}**H**), 6.28 (s, 1H, C_2**H**), 5.38–5.21 (m, 4H, $Ph_{Cbz}C$**H**$_2$), 4.29 (d, J = 9.5, 1H, $C_{11'}$**H**), 4.18 (br-s, 1H, C_{11}**H**), 4.01 (app-t, J = 7.5, 1H, $C_{15'}$**H**), 3.91 (q, J = 6.8, 1H, C_{15}**H**), 3.64–3.47 (m, 2H, $C_{19'}$**H**), 3.64–3.47 (m, 1H, $C_{12'}$**H**$_a$), 3.30 (d, J = 12.9, 1H, C_{12}**H**$_a$), 2.85 (dd, J = 10.7, 15.5, 1H, $C_{12'}$**H**$_b$), 2.81–2.70 (m, 1H, C_{12}**H**$_b$), 2.33–2.21 (m, 1H, $C_{17'}$**H**$_a$), 2.04–1.91 (m, 1H, $C_{17'}$**H**$_b$), 2.04–1.91 (m, 1H, $C_{18'}$**H**$_a$), 1.91–1.78 (m, 1H, $C_{18'}$**H**$_b$), 1.30 (d, J = 6.7, 3H, C_{17}**H**).

^{13}C NMR (125.8 MHz, $CDCl_3$, 20 ºC): δ 170.0 (**C**$_{16'}$), 169.6 (**C**$_{13}$), 167.7 (**C**$_{16}$), 165.2 (**C**$_{13'}$), 153.2 (**C**$_{Cbz}$=O), 150.6 (**C**$_{Cbz'}$=O), 140.0 (**C**$_9$), 139.3 (**C**$_{7'}$), 136.3 (Ph_{Cbz}-*ipso*-**C**), 135.0 (**C**$_4$), 135.0 (**C**$_{9'}$), 135.0 ($Ph_{Cbz'}$-*ipso*-**C**), 129.5 (**C**$_{4'}$), 129.2 (**C**$_7$), 129.1 (Ph_{Cbz}-**C**), 129.0 (Ph_{Cbz}-**C**), 129.0 (Ph_{Cbz}-**C**), 128.6 (Ph_{Cbz}-**C**), 128.2 (Ph_{Cbz}-**C**), 128.2 (Ph_{Cbz}-**C**), 125.3 (**C**$_5$), 124.7 (**C**$_{2'}$), 124.3 (**C**$_6$), 121.4 (**C**$_{6'}$), 119.5 (**C**$_{5'}$), 117.0 (**C**$_8$), 116.1 (**C**$_{3'}$), 113.2 (**C**$_{8'}$), 83.9 (**C**$_2$), 69.1 ($Ph_{Cbz'}$**C**H$_2$), 68.1 (Ph_{Cbz}**C**H$_2$), 59.3 (**C**$_{15'}$), 58.9

	(**C**$_3$), 58.5 (**C**$_{11}$), 54.2 (**C**$_{11'}$), 51.9 (**C**$_{15}$), 45.7 (**C**$_{19'}$), 35.4 (**C**$_{12}$), 28.4 (**C**$_{17'}$), 26.4 (**C**$_{12'}$), 22.8 (**C**$_{18'}$), 15.3 (**C**$_{17}$).
FTIR (thin film) cm^{-1}:	3226 (br, m), 1688 (s), 1399 (m), 1308 (m), 751 (m).
HRMS (DART) (*m*/*z*):	calc'd for $C_{46}H_{43}N_6O_8$ $[M+H]^+$: 807.3137 found: 807.3116.
$[\alpha]_D^{24}$:	–42 (*c* = 0.38, $CHCl_3$).
TLC (7.5% methanol in ethyl acetate), R*f*:	0.11 (UV, CAM).



(+)-Naseseazine A (1):

Palladium on charcoal (10% w/w, 1.0 mg, 905 nmol, 0.10 equiv) was added to a solution of (–)-N^{in},$N^{in'}$-dicarboxybenzyl naseseazine A (**15**) (7.3 mg, 9.05 μmol, 1 equiv) in acetic acid (1 mL) at 23 °C. A stream of hydrogen gas was passed through the solution for 2 min by discharge of a balloon equipped with a needle extending into the reaction mixture. After stirring the solution for 9 h under an atmosphere of hydrogen gas, the solution was filtered over Celite. The solids were further extracted with methanol and the combined filtrates were concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography on silica gel (eluent: 5% methanol, 45% tetrahydrofuran, 50% dichloromethane) to afford (+)-naseseazine A (**1**) (4.1 mg, 80%) as a white solid. Structural assignments were made using additional information from gCOSY, HSQC, HMBC, and ROESY experiments.

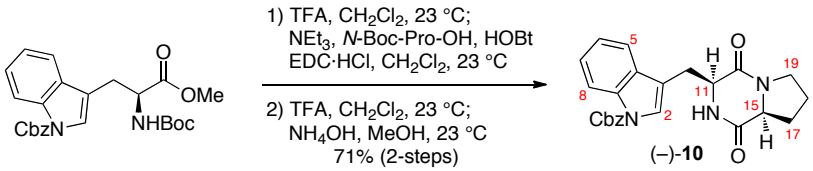
^{1}H NMR (500 MHz, methanol-d_4, 20 ºC): δ 7.57 (d, J = 8.4, 1H, $C_{5'}$**H**), 7.40 (d, J = 1.3, 1H, $C_{8'}$**H**), 7.12 (s, 1H, $C_{2'}$**H**), 7.05 (app-dt, J = 1.3, 7.9, 1H, C_7**H**), 7.03 (dd, J = 1.7, 8.4, 1H, $C_{6'}$**H**), 6.85 (dd, J = 0.6, 7.4, 1H, C_5**H**), 6.69 (d, J = 8.0, 1H, C_8**H**), 6.68 (app-dt, J = 0.9, 7.4, 1H, C_6**H**), 5.83 (s, 1H, C_2**H**), 4.65 (dd, J = 7.4, 9.2, 1H, C_{11}**H**), 4.39 (dt, J = 1.1, 4.7, 1H, $C_{11'}$**H**), 4.16 (dq, J = 1.4, 6.9, 1H, C_{15}**H**), 3.98 (ddd, J = 1.7, 6.3, 10.9, 1H, $C_{15'}$**H**), 3.42 (app-dt, J = 8.3, 11.7, 1H, $C_{19'}$**H**$_a$), 3.30–3.20 (m, 1H, $C_{19'}$**H**$_b$), 3.30–3.20 (m, 2H, $C_{12'}$**H**), 3.30–3.20 (m, 1H, C_{12}**H**$_a$), 2.59 (dd, J = 9.9, 13.6, 1H, C_{12}**H**$_b$), 1.96 (app-ddt, J = 2.0, 7.2, 12.5, 1H, $C_{17'}$**H**$_a$), 1.72–1.61 (m, 1H, $C_{18'}$**H**$_a$), 1.47–1.39 (m, 1H, $C_{18'}$**H**$_b$), 1.38 (d, J = 7.1, 3H, C_{17}**H**), 0.96–0.85 (m, 1H, $C_{17'}$**H**$_b$).

^{1}H NMR (500 MHz, DMSO-d_6, 20 ºC): δ 10.81 (s, 1H, $N_{1'}$**H**), 8.20 (s, 1H, N_{14}**H**), 7.71 (s, 1H, $N_{10'}$**H**), 7.56 (d, J = 8.4, 1H, $C_{5'}$**H**), 7.28 (s, 1H, $C_{8'}$**H**), 7.19 (d, J = 1.8, 1H, $C_{2'}$**H**), 7.04–6.95 (m, 1H, C_7**H**), 7.04–6.95 (m, 1H, $C_{6'}$**H**), 6.83 (d, J = 7.3, 1H, C_5**H**), 6.75 (d, J = 2.8, 1H, N_1**H**), 6.62 (d, J = 8.0, 1H, C_8**H**), 6.58 (app-t, J = 7.4, 1H, C_6**H**), 5.65 (d, J = 2.8, 1H, C_2**H**), 4.62 (app-t, J = 8.7, 1H, C_{11}**H**), 4.28 (app-t, J = 4.9, 1H, $C_{11'}$**H**), 4.14 (q, J = 6.5, 1H, C_{15}**H**), 4.06 (app-t, J = 7.8, 1H, $C_{15'}$**H**), 3.41–3.29 (m, 1H, $C_{19'}$**H**$_a$), 3.30–3.18 (m, 1H, $C_{19'}$**H**$_b$) 3.30–3.18 (m, 1H, $C_{12'}$**H**$_a$), 3.14–3.00 (m,

	1H, $C_{12}\mathbf{H}_a$), 3.14–3.00 (m, 1H, $C_{12'}\mathbf{H}_b$), 2.41 (dd, J = 10.0, 13.6, 1H, $C_{12}\mathbf{H}_b$), 2.03–1.94 (m, 1H, $C_{17'}\mathbf{H}_a$), 1.74–1.56 (m, 2H, $C_{18'}\mathbf{H}$), 1.47–1.36 (m, 1H, $C_{17'}\mathbf{H}_b$), 1.23 (d, J = 6.9, 3H, $C_{17}\mathbf{H}$).
^{13}C NMR (125.8 MHz, methanol-d_4, 20 ºC):	δ 172.7 ($\mathbf{C}_{13}$), 170.9 ($\mathbf{C}_{16}$), 170.9 ($\mathbf{C}_{16'}$), 167.5 ($\mathbf{C}_{13'}$), 149.3 ($\mathbf{C}_9$), 138.1 ($\mathbf{C}_{9'}$), 137.3 ($\mathbf{C}_{7'}$), 136.1 ($\mathbf{C}_4$), 129.6 ($\mathbf{C}_7$), 127.8 ($\mathbf{C}_{4'}$), 126.6 ($\mathbf{C}_{2'}$), 125.1 ($\mathbf{C}_5$), 120.6 ($\mathbf{C}_6$), 120.5 ($\mathbf{C}_{5'}$), 119.7 ($\mathbf{C}_{6'}$), 111.2 ($\mathbf{C}_8$), 110.4 ($\mathbf{C}_{8'}$), 109.7 ($\mathbf{C}_{3'}$), 87.3 ($\mathbf{C}_2$), 61.4 ($\mathbf{C}_3$), 60.5 ($\mathbf{C}_{11}$), 60.2 ($\mathbf{C}_{15'}$), 57.4 ($\mathbf{C}_{11'}$), 52.4 ($\mathbf{C}_{15}$), 46.1 ($\mathbf{C}_{19'}$), 39.9 ($\mathbf{C}_{12}$), 29.3 ($\mathbf{C}_{17'}$), 29.3 ($\mathbf{C}_{12'}$), 22.7 ($\mathbf{C}_{18'}$), 15.4 ($\mathbf{C}_{17}$).
^{13}C NMR (125.8 MHz, DMSO-d_6, 20 ºC):	δ 170.0 ($\mathbf{C}_{13}$), 169.1 ($\mathbf{C}_{16'}$), 168.6 ($\mathbf{C}_{16}$), 165.5 ($\mathbf{C}_{13'}$), 148.1 ($\mathbf{C}_9$), 135.9 ($\mathbf{C}_{9'}$), 135.7 ($\mathbf{C}_{7'}$), 134.4 ($\mathbf{C}_4$), 127.9 ($\mathbf{C}_7$), 126.1 ($\mathbf{C}_{4'}$), 125.0 ($\mathbf{C}_{2'}$), 123.6 ($\mathbf{C}_5$), 119.2 ($\mathbf{C}_{5'}$), 118.0 ($\mathbf{C}_6$), 117.8 ($\mathbf{C}_{6'}$), 109.3 ($\mathbf{C}_8$), 109.2 ($\mathbf{C}_{8'}$), 109.1 ($\mathbf{C}_{3'}$), 85.0 ($\mathbf{C}_2$), 59.3 ($\mathbf{C}_3$), 58.5 ($\mathbf{C}_{15'}$), 58.4 ($\mathbf{C}_{11}$), 55.2 ($\mathbf{C}_{11'}$), 50.4 ($\mathbf{C}_{15}$), 44.6 ($\mathbf{C}_{19'}$), 38.8 ($\mathbf{C}_{12}$), 27.7 ($\mathbf{C}_{17'}$), 25.7 ($\mathbf{C}_{12'}$), 21.9 ($\mathbf{C}_{18'}$), 14.8 ($\mathbf{C}_{17}$).
FTIR (thin film) cm^{-1}:	3584 (s), 3272 (br, s), 2924 (w), 1667 (s), 1453 (w), 1412 (m), 1346 (w), 1307 (w).
UV (CH_3OH) λ_{max} (log ε):	229 (4.67), 285 (3.91).
HRMS (DART) (*m*/*z*):	calc'd for $C_{30}H_{31}N_6O_4$ $[M+H]^+$: 539.2401, found: 539.2392.
$[\alpha]_D^{24}$:	+123 (c = 0.12, CH_3OH).[9]

TLC (5% methanol, 47.5% tetrahydrofuran, 47.5% dichloromethane), R*f*: 0.15 (UV, CAM).

[9] Literature value: $[\alpha]_D^{23}$ = +139 (c 0.10, CH_3OH), see Raju, R.; Piggott, A. M.; Conte, M.; Aalbersberg, W. G. L.; Feussner, K.; Capon, R. J. *Org. Lett.* **2009**, *11*, 3862.



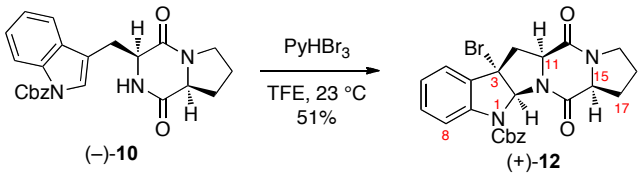
Proline diketopiperazine (–)-(10):

Trifluoroacetic acid (30 mL) was added via syringe to a solution of N^{α}-Boc-N^{in}-Cbz-L-tryptophan methyl ester[8] (7.10 g, 15.7 mmol, 1 equiv) in dichloromethane (100 mL) at 23 °C. After 1 h, the brown solution was concentrated under reduced pressure to afford a viscous brown residue, which was dissolved in dichloromethane (160 mL) and cooled to 0 °C in an ice-water bath. Triethylamine (9.85 mL, 70.7 mmol, 4.50 equiv) was added dropwise via syringe. *N*-hydroxybenzotriazole (3.18 g, 23.6 mmol, 1.50 equiv) and N^{α}-Boc-L-proline (6.76 g, 31.4 mmol, 2.00 equiv) were sequentially added to the solution. After 2 min, upon dissolution of the N^{α}-Boc-L-proline, 1-ethyl-3-(3-dimethylaminopropyl) carbodiimide hydrogen chloride (4.51 g, 23.6 mmol, 1.50 equiv) was added and the reaction mixture was allowed to warm to 23 °C. After 1 h and 45 min, another portion of 1-ethyl-3-(3-dimethylaminopropyl) carbodiimide hydrogen chloride (1.50 g, 7.85 mmol, 0.500 equiv) was added. After 1.5 h, aqueous hydrogen chloride solution (1 N, 500 mL) was added and the aqueous layer was extracted with dichloromethane (2 × 50 mL). The combined organic layers were washed with saturated aqueous sodium bicarbonate solution (500 mL) and the resulting aqueous layer was extracted with dichloromethane (2 × 50 mL). The combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure. The resulting off-white foam was then dissolved in dichloromethane (100 mL) and trifluoroacetic acid (30 mL) was added dropwise to the solution at 23 °C. After 1 h, the solution was concentrated under reduced pressure. The viscous residue was dissolved in methanol (400 mL) and cooled to 0 °C. Ammonium hydroxide (28–30% ammonia, 15 mL) was added dropwise and the reaction mixture was allowed to warm to 23 °C. After 14 h, another portion of ammonium hydroxide (28–30% ammonia, 10 mL) was added dropwise via syringe. After 23 h, the reaction mixture was cooled to –78 °C and filtered. The white precipitate was washed with methanol (2 × 50 mL), which had been cooled to 0 °C in an ice-water bath, and was dried under reduced pressure to afford diketopiperazine (–)-**10** (4.63 g, 70.6%, 2-steps) as a fine white powder. Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

^{1}H NMR (500 MHz, DMSO-d_6, 20 ºC): δ 8.11 (s, 1H, N_{10}**H**), 8.07 (d, J = 8.0, 1H, C_8**H**), 7.67 (d, J = 7.7, 2H, C_5**H**), 7.60 (s, 1H, C_2**H**), 7.52 (d, J = 7.7, 1H, Ph_{Cbz}-*o*-**H**), 7.46–7.37 (m, 3H, Ph_{Cbz}-**H**), 7.33 (app-dt, J = 0.9, 7.7, 1H, C_7**H**), 7.27 (app-dt, J = 0.9, 7.5, 1H, C_6**H**), 5.46 (s, 2H, $Ph_{Cbz}C$**H**$_2$), 4.42 (app-t, J = 5.2, 1H, C_{11}**H**), 4.12 (app-t, J = 8.2, 1H, C_{15}**H**), 3.40–3.26 (m, 2H, C_{19}**H**), 3.22 (dd, J = 4.2, 15.3, 1H, C_{12}**H**$_a$), 3.05 (dd, J = 5.6, 15.3, 1H, C_{12}**H**$_b$), 2.09–1.99 (m, 1H, C_{17}**H**$_a$), 1.78–1.62 (m, 2H, C_{18}**H**), 1.62–1.48 (m, 1H, C_{17}**H**$_b$).

^{13}C NMR (125.8 MHz, DMSO-d_6, 20 ºC): δ 169.3 (**C**$_{16}$), 165.2 (**C**$_{13}$), 150.1 (**C**$_{Cbz}$=O), 135.4 (Ph_{Cbz}-*ipso*-**C**), 134.6 (**C**$_9$), 130.4 (**C**$_4$), 128.6 (Ph_{Cbz}-**C**), 128.5 (Ph_{Cbz}-**C**), 128.3 (Ph_{Cbz}-**C**), 124.5 (**C**$_7$), 124.0 (**C**$_2$), 122.7 (**C**$_6$), 119.7 (**C**$_5$), 116.9

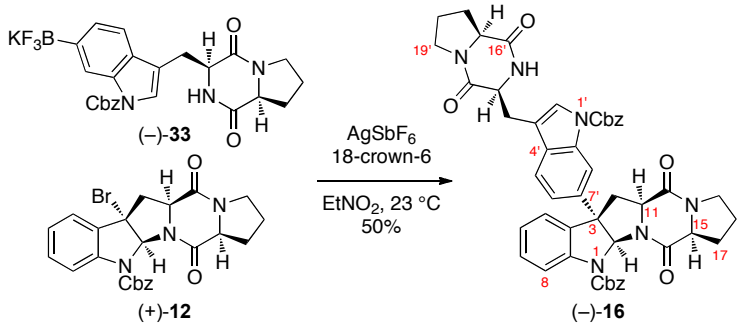
	(**C**$_3$), 114.5 (**C**$_8$), 68.2 (Ph$_{Cbz}$**C**H$_2$), 58.5 (**C**$_{15}$), 54.3 (**C**$_{11}$), 44.7 (**C**$_{19}$), 27.8 (**C**$_{17}$), 24.9 (**C**$_{12}$), 22.0 (**C**$_{18}$).
FTIR (thin film) cm^{-1}:	3233 (br, m), 1732 (s), 1667 (s), 1455 (m), 1399 (m), 1249 (m).
HRMS (DART) (*m*/*z*):	calc'd for $C_{24}H_{24}N_3O_4$ [M+H]$^+$: 418.1761, found: 418.1764.
$[\alpha]_D^{24}$:	–49 (*c* = 0.23, DMSO).
TLC (40% acetone in dichloromethane), R*f*:	0.45 (UV, CAM).



Tetracyclic bromide B (+)-12:

Pyridinium tribromide (461 mg, 1.44 mmol, 1.20 equiv) was added to a suspension of diketopiperazine (–)-**10** (500 mg, 1.20 mmol, 1 equiv) in 2,2,2-trifluoroethanol (10 mL) at 23 ºC. After 1 h, the reaction mixture was poured into a saturated aqueous sodium thiosulfate solution (60 mL) and extracted with dichloromethane (60 mL). The resulting aqueous layer was further extracted with dichloromethane (2 × 30 mL) and the combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography on silica gel (eluent: 75% ethyl acetate in hexanes) to afford tetracyclic bromide (+)-**12** (304 mg, 51.1%) as a beige foam. Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

^{1}H NMR (500 MHz, $CDCl_3$, 20 ºC):	δ 7.64 (d, *J* = 8.2, 1H, C_8**H**), 7.48–7.39 (m, 1H, C_5**H**), 7.48–7.39 (m, 2H, Ph_{Cbz}-*o*-**H**), 7.34 (app-t, *J* = 7.0, 2H, Ph_{Cbz}-*m*-**H**), 7.29 (t, *J* = 7.3, 1H, Ph_{Cbz}-*p*-**H**), 7.23 (app-t, *J* = 7.6, 1H, C_7**H**), 7.07 (app-t, *J* = 7.3, 1H, C_6**H**), 6.29 (s, 1H, C_2**H**), 5.41 (d, *J* = 12.2, 1H, $Ph_{Cbz}C\mathbf{H}_a$), 5.30 (d, *J* = 12.2, 1H, $Ph_{Cbz}C\mathbf{H}_b$), 4.35 (dd, *J* = 3.4, 14.3, 1H, $C_{12}\mathbf{H}_a$), 4.00 (app-t, *J* = 7.9, 1H, C_{15}**H**), 3.78 (dd, *J* = 3.4, 14.3, 1H, $C_{12}\mathbf{H}_a$), 3.37–3.27 (m, 1H, $C_{19}\mathbf{H}_a$), 3.18–3.07 (m, 1H, $C_{19}\mathbf{H}_b$), 3.03 (dd, *J* = 10.1, 14.0, 1H, $C_{12}\mathbf{H}_b$), 2.21–2.09 (m, 1H, $C_{17}\mathbf{H}_a$), 2.09–1.96 (m, 1H, $C_{17}\mathbf{H}_b$), 1.80–1.68 (m, 2H, C_{18}**H**).
^{13}C NMR (125.8 MHz, $CDCl_3$, 20 ºC):	δ 167.2 (**C**$_{13}$), 164.3 (**C**$_{16}$), 152.8 (**C**$_{Cbz}$=O), 139.4 (**C**$_9$), 135.8 (Ph_{Cbz}-*ipso*-**C**), 132.7 (**C**$_4$), 130.8 (**C**$_7$), 128.6 (Ph_{Cbz}-**C**), 128.3 (Ph_{Cbz}-**C**), 128.2 (Ph_{Cbz}-**C**), 124.9 (**C**$_5$), 124.7 (**C**$_6$), 116.6 (**C**$_8$), 84.6 (**C**$_2$), 68.3 ($Ph_{Cbz}\mathbf{C}H_2$), 60.6 (**C**$_3$), 60.5 (**C**$_{15}$), 59.2 (**C**$_{11}$), 45.1 (**C**$_{19}$), 37.5 (**C**$_{12}$), 27.3 (**C**$_{17}$), 23.3 (**C**$_{18}$).
FTIR (thin film) cm^{-1}:	1722 (s), 1679 (s), 1479 (m), 1410 (m), 751 (m).
HRMS (ESI) (*m*/*z*):	calc'd for $C_{24}H_{23}BrN_3O_4$ $[M+H]^+$: 496.0866, found: 496.0877.
$[\alpha]_D^{23}$:	+63 (*c* = 0.11, $CHCl_3$).
TLC (75% ethyl acetate in hexanes), R*f*:	0.18 (UV, CAM).



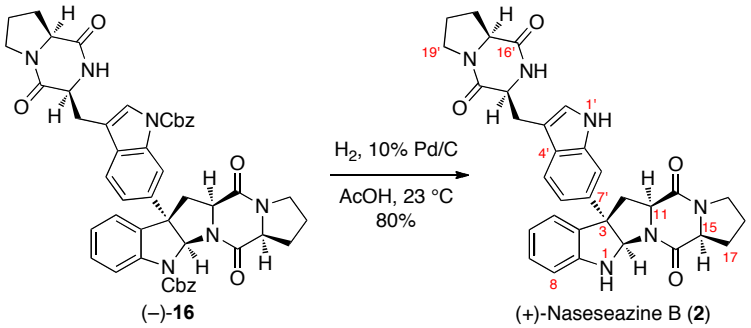
(–)-N^{in},$N^{in'}$-Dicarboxybenzyl Naseseazine B (16):

Silver(I) hexafluoroantimonate (69.2 mg, 202 μmol, 5.00 equiv) was added as a solid to a solution of tetracyclic bromide (+)-**12** (20.0 mg, 40.3 μmol, 1 equiv), potassium 6-trifluoroborateindole (–)-**33** (31.6 mg, 60.4 μmol, 1.50 equiv), and 18-crown-6 (53.3 mg, 202 μmol, 5.00 equiv) in nitroethane (2 mL) at 23 ºC. After 1 h, aqueous hydrogen chloride (2 N, 2 mL) was added. After stirring for 5 min, the reaction mixture was diluted with ethyl acetate (60 mL) and washed with brine (3 × 60 mL). The organic layer was dried over anhydrous sodium sulfate, was filtered, and was concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography on silica gel (eluent: gradient, 5% methanol in ethyl acetate→5% methanol in dichloromethane) to afford (–)-N^{in},$N^{in'}$-dicarboxybenzyl naseseazine B (**16**) (16.7 mg, 49.8%) as a white solid. Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

^{1}H NMR (500 MHz, $CDCl_3$, 20 ºC): δ 8.14 (br-s, 1H, $C_{8'}$**H**), 7.66 (d, J = 8.0, 1H, C_8**H**), 7.49 (s, 1H, $C_{2'}$**H**), 7.46–7.32 (m, 1H, $C_{5'}$**H**), 7.46–7.32 (m, 7H, Ph_{Cbz}-**H**), 7.32–7.25 (m, 1H, C_5**H**), 7.32–7.25 (m, 3H, Ph_{Cbz}-**H**), 7.18 (app-t, J = 7.5, 1H, C_7**H**), 7.07–6.97 (m, 1H, C_6**H**), 7.07–6.97 (m, 1H, $C_{6'}$**H**), 6.38 (s, 1H, C_2**H**), 5.82 (s, 1H, $N_{10'}$**H**), 5.41–5.22 (m, 4H, $Ph_{Cbz}C$**H**$_2$), 4.37 (d, J = 7.1, 1H, C_{11}**H**), 4.26 (dd, J = 2.3, 10.2, 1H, $C_{11'}$**H**), 4.04 (app-t, J = 8.0, 1H, C_{15}**H**), 4.00 (app-t, J = 7.9, 1H, $C_{15'}$**H**), 3.65–3.47 (m, 2H, $C_{19'}$**H**), 3.65–3.47 (m, 1H, C_{12}**H**$_a$), 3.65–3.47 (m, 1H, $C_{12'}$**H**$_a$), 3.41–3.31 (m, 1H, C_{19}**H**$_a$), 3.20–3.10 (m, 1H, C_{19}**H**$_b$), 2.91–2.78 (m, 1H, C_{12}**H**$_b$), 2.91–2.78 (m, 1H, $C_{12'}$**H**$_b$), 2.30–2.23 (m, 1H, $C_{17'}$**H**$_a$), 2.23–2.15 (m, 1H, C_{17}**H**$_a$), 2.14–2.04 (m, 1H, C_{17}**H**$_b$), 2.03–1.90 (m, 1H, $C_{17'}$**H**$_b$), 2.03–1.90 (m, 1H, $C_{18'}$**H**$_a$), 1.90–1.66 (m, 2H, C_{18}**H**), 1.90–1.66 (m, 1H, $C_{18'}$**H**$_b$).

^{13}C NMR (125.8 MHz, $CDCl_3$, 20 ºC): δ 169.5 (**C**$_{16'}$), 167.9 (**C**$_{16}$), 165.5 (**C**$_{13}$), 165.1 (**C**$_{13'}$), 153.2 (**C**$_{Cbz}$=O), 150.5 (**C**$_{Cbz'}$=O), 139.8 (**C**$_9$), 139.5 (**C**$_{7'}$), 136.4 (Ph_{Cbz}-*ipso*-**C**), 135.0 ($Ph_{Cbz'}$-*ipso*-**C**), 135.0 (**C**$_4$), 135.0 (**C**$_{9'}$), 129.1 (**C**$_7$), 129.1 (**C**$_{4'}$), 129.1 (Ph_{Cbz}-**C**), 129.0 (Ph_{Cbz}-**C**), 129.0 (Ph_{Cbz}-**C**), 128.6 (Ph_{Cbz}-**C**), 128.3 (Ph_{Cbz}-**C**), 128.2 (Ph_{Cbz}-**C**), 125.5 (**C**$_5$), 124.9 (**C**$_{2'}$), 124.4 (**C**$_6$), 121.4 (**C**$_{6'}$),

	119.5 ($\mathbf{C}_{5'}$), 116.8 ($\mathbf{C}_{8}$), 115.8 ($\mathbf{C}_{3'}$), 113.3 ($\mathbf{C}_{8'}$), 83.6 ($\mathbf{C}_{2}$), 69.3 ($Ph_{Cbz'}\mathbf{C}H_2$), 68.2 ($Ph_{Cbz}\mathbf{C}H_2$), 61.1 ($\mathbf{C}_{15}$), 59.7 ($\mathbf{C}_{11}$), 59.4 ($\mathbf{C}_{15'}$), 59.2 ($\mathbf{C}_{3}$), 54.2 ($\mathbf{C}_{11'}$), 45.7 ($\mathbf{C}_{19'}$), 45.2 ($\mathbf{C}_{19}$), 35.4 ($\mathbf{C}_{12}$), 28.5 ($\mathbf{C}_{17'}$), 27.6 ($\mathbf{C}_{17}$), 26.6 ($\mathbf{C}_{12'}$), 23.6 ($\mathbf{C}_{18}$), 22.8 ($\mathbf{C}_{18'}$).
FTIR (thin film) cm^{-1}:	1721 (s), 1679 (s), 1399 (s), 1249 (m), 751 (m).
HRMS (ESI) (*m*/*z*):	calc'd for $C_{48}H_{45}N_6O_8$ $[M+H]^+$: 833.3293, found: 833.3283.
$[\alpha]_D^{25}$:	–40 (*c* = 0.18, $CHCl_3$).
TLC (5% methanol in ethyl acetate), R*f*:	0.09 (UV, CAM).



(+)-Naseseazine B (2):

Palladium on charcoal (10% w/w, 2.1 mg, 1.98 μmol, 0.25 equiv) was added to a solution of dicarboxybenzyl naseseazine B (–)-(**16**) (6.6 mg, 7.92 μmol, 1 equiv) in acetic acid (1 mL) at 23 °C. A stream of hydrogen gas was passed through the solution for 2 min by discharge of a balloon equipped with a needle extending into the reaction mixture. After stirring the solution for 3.5 h under an atmosphere of hydrogen gas, the solution was filtered over Celite. The solids were further extracted with methanol and the combined filtrates were concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography on silica gel (eluent: 5% methanol, 45% tetrahydrofuran, 50% dichloromethane) to afford (+)-naseseazine B (**2**) (3.6 mg, 80%) as a white solid. Structural assignments were made using additional information from gCOSY, HSQC, HMBC, and ROESY experiments.

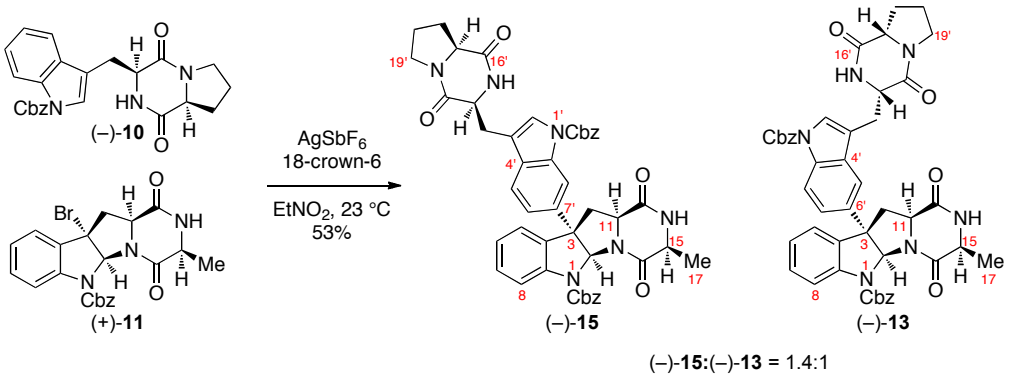
^{1}H NMR (500 MHz, methanol-d_4, 20 °C): δ 7.57 (d, J = 7.4, 1H, $C_{5'}$**H**), 7.41 (d, J = 1.3, 1H, $C_{8'}$**H**), 7.12 (s, 1H, $C_{2'}$**H**), 7.06 (app-dt, J = 1.2, 7.9, 1H, C_7**H**), 7.01 (dd, J = 1.6, 8.5, 1H, $C_{6'}$**H**), 6.83 (d, J = 7.2, 1H, C_5**H**), 6.69 (d, J = 7.7, 1H, C_8**H**), 6.68 (app-t, J = 7.3, 1H, C_6**H**), 5.84 (s, 1H, C_2**H**), 4.71 (app-t, J = 8.6, 1H, C_{11}**H**), 4.39 (app-t, J = 4.6, 1H, $C_{11'}$**H**), 4.29 (app-t, J = 7.7, 1H, C_{15}**H**), 3.98 (ddd, J = 1.4, 6.3, 10.7, 1H, $C_{15'}$**H**), 3.52–3.37 (m, 2H, C_{19}**H**), 3.52–3.37 (m, 1H, $C_{19'}$**H**$_a$), 3.35–3.21 (m, 2H, $C_{12'}$**H**), 3.35–3.21 (m, 1H, C_{12}**H**$_a$), 3.35–3.21 (m, 1H, $C_{19'}$**H**$_b$), 2.57 (dd, J = 10.2, 13.7, 1H, C_{12}**H**$_b$), 2.30–2.22 (m, 1H, C_{17}**H**$_a$), 2.15–2.04 (m, 1H, C_{17}**H**$_b$), 2.04–1.87 (m, 2H, C_{18}**H**), 2.04–1.87 (m, 1H, $C_{17'}$**H**$_a$), 1.73–1.60 (m, 1H, $C_{18'}$**H**$_a$), 1.49–1.40 (m, 1H, $C_{18'}$**H**$_b$), 0.97–0.87 (m, 1H, C_{17}**H**$_b$).

^{1}H NMR (500 MHz, DMSO-d_6, 20 °C): δ 10.81 (s, 1H, $N_{1'}$**H**), 7.71 (s, 1H, $N_{10'}$**H**), 7.57 (d, J = 8.4, 1H, $C_{5'}$**H**), 7.30 (d, J = 1.3, 1H, $C_{8'}$**H**), 7.19 (d, J = 2.2, 1H, $C_{2'}$**H**), 7.00 (app-dt, J = 1.3, 7.9, 1H, C_7**H**), 6.99 (d, J = 8.6, 1H, $C_{6'}$**H**), 6.82–6.76 (m, 1H, C_5**H**), 6.82–6.76 (m, 1H, N_1**H**), 6.60 (d, J = 7.6, 1H, C_8**H**), 6.58 (app-dt, J = 1.0, 7.5, 1H, C_6**H**), 5.68 (d, J = 3.2, 1H, C_2**H**), 4.72 (app-t, J = 8.2, 1H, C_{11}**H**), 4.35 (app-t, J = 7.9, 1H, C_{15}**H**), 4.29 (app-t, J = 5.1, 1H, $C_{11'}$**H**), 4.06 (app-t, J = 8.7, 1H, $C_{15'}$**H**), 3.41–3.30 (m, 2H, C_{19}**H**), 3.41–

	3.30 (m, 1H, $C_{19'}\mathbf{H}_a$), 3.29–3.24 (m, 1H, $C_{19'}\mathbf{H}_b$), 3.22 (dd, J = 4.6, 14.6, 1H, $C_{12'}\mathbf{H}_a$), 3.13 (dd, J = 7.4, 13.6, 1H, $C_{12}\mathbf{H}_a$), 3.05 (dd, J = 5.8, 14.8, 1H, $C_{12'}\mathbf{H}_b$), 2.36 (dd, J = 10.5, 13.6, 1H, $C_{12}\mathbf{H}_b$), 2.21–2.11 (m, 1H, $C_{17}\mathbf{H}_a$), 2.03–1.91 (m, 1H, $C_{17'}\mathbf{H}_a$), 2.03–1.91 (m, 1H, $C_{17}\mathbf{H}_b$), 1.91–1.77 (m, 2H, $C_{18}\mathbf{H}$), 1.75–1.56 (m, 2H, $C_{18'}\mathbf{H}$), 1.48–1.36 (m, 1H, $C_{17'}\mathbf{H}_b$).
^{13}C NMR (125.8 MHz, methanol-d_4, 20 ºC):	δ 170.9 ($\mathbf{C}_{16'}$), 170.3 ($\mathbf{C}_{16}$), 168.6 ($\mathbf{C}_{13}$), 167.5 ($\mathbf{C}_{13'}$), 149.3 ($\mathbf{C}_9$), 138.1 ($\mathbf{C}_{9'}$), 137.2 ($\mathbf{C}_{7'}$), 136.2 ($\mathbf{C}_4$), 129.6 ($\mathbf{C}_7$), 127.8 ($\mathbf{C}_{4'}$), 126.6 ($\mathbf{C}_{2'}$), 125.1 ($\mathbf{C}_5$), 120.6 ($\mathbf{C}_6$), 120.6 ($\mathbf{C}_{5'}$), 119.8 ($\mathbf{C}_{6'}$), 111.3 ($\mathbf{C}_8$), 110.5 ($\mathbf{C}_{8'}$), 109.8 ($\mathbf{C}_{3'}$), 87.1 ($\mathbf{C}_2$), 61.9 ($\mathbf{C}_{15}$), 61.9 ($\mathbf{C}_3$), 61.7 ($\mathbf{C}_{11}$), 60.2 ($\mathbf{C}_{15'}$), 57.3 ($\mathbf{C}_{11'}$), 46.4 ($\mathbf{C}_{19}$), 46.1 ($\mathbf{C}_{19'}$), 39.7 ($\mathbf{C}_{12}$), 29.3 ($\mathbf{C}_{17'}$), 29.3 ($\mathbf{C}_{12'}$), 28.6 ($\mathbf{C}_{17}$), 24.4 ($\mathbf{C}_{18}$), 22.7 ($\mathbf{C}_{18'}$).
^{13}C NMR (125.8 MHz, DMSO-d_6, 20 ºC):	δ 169.1 ($\mathbf{C}_{16'}$), 167.9 ($\mathbf{C}_{16}$), 165.9 ($\mathbf{C}_{13}$), 165.5 ($\mathbf{C}_{13'}$), 148.1 ($\mathbf{C}_9$), 135.9 ($\mathbf{C}_{9'}$), 135.6 ($\mathbf{C}_{7'}$), 134.6 ($\mathbf{C}_4$), 127.9 ($\mathbf{C}_7$), 126.1 ($\mathbf{C}_{4'}$), 125.0 ($\mathbf{C}_{2'}$), 123.4 ($\mathbf{C}_5$), 119.1 ($\mathbf{C}_{5'}$), 117.9 ($\mathbf{C}_6$), 117.8 ($\mathbf{C}_{6'}$), 109.2 ($\mathbf{C}_8$), 109.2 ($\mathbf{C}_{8'}$), 109.2 ($\mathbf{C}_{3'}$), 84.8 ($\mathbf{C}_2$), 59.9 ($\mathbf{C}_{15}$), 59.8 ($\mathbf{C}_3$), 59.5 ($\mathbf{C}_{11}$), 58.4 ($\mathbf{C}_{15'}$), 55.2 ($\mathbf{C}_{11'}$), 44.6 ($\mathbf{C}_{19}$), 44.6 ($\mathbf{C}_{19'}$), 38.7 ($\mathbf{C}_{12}$), 27.6 ($\mathbf{C}_{17'}$), 27.0 ($\mathbf{C}_{17}$), 25.6 ($\mathbf{C}_{12'}$), 22.9 ($\mathbf{C}_{18}$), 21.9 ($\mathbf{C}_{18'}$).
FTIR (thin film) cm^{-1}:	3295 (br, s), 2954 (w), 2882 (w), 1661 (s), 1426 (m), 1343 (w), 1312 (w), 1245 (w), 750 (m).
UV (CH_3OH) λ_{max} (log ε):	201 (4.95), 203 (4.98), 204 (4.92), 229 (4.67), 285 (3.92).
HRMS (DART) (*m*/*z*):	calc'd for $C_{32}H_{33}N_6O_4$ $[M+H]^+$: 565.2558, found: 565.2546.
$[\alpha]_D^{24}$:	+101 (c = 0.23, CH_3OH).[10]

TLC (5% methanol, 45% tetrahydrofuran, 50% dichloromethane), R*f*: 0.19 (UV, CAM).

[10] Literature value: $[\alpha]_D^{23}$ = +95 (c 0.08, CH_3OH), Raju, R.; Piggott, A. M.; Conte, M.; Aalbersberg, W. G. L.; Feussner, K.; Capon, R. J. *Org. Lett.* **2009**, *11*, 3862.



(–)-N^{in},$N^{in'}$-Dicarboxybenzyl *iso*-Naseseazine A (13):

Silver(I) hexafluoroantimonate (73.0 mg, 213 μmol, 5.00 equiv) was added as a solid to a solution of tetracyclic bromide (+)-**11** (20.0 mg, 42.5 μmol, 1 equiv) and proline diketopiperazine (–)-**10** (26.6 mg, 63.8 μmol, 1.50 equiv) in nitroethane (2 mL) at 23 ºC. After 2 h, the reaction mixture was diluted with dichloromethane (60 mL) and washed with brine (60 mL). The resulting aqueous layer was extracted with dichloromethane (2 × 30 mL) and the combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography on silica gel (eluent: gradient, 5% methanol in ethyl acetate→5% methanol in dichloromethane) to afford a regioisomeric mixture of (–)-N^{in},$N^{in'}$-dicarboxybenzyl naseseazine A (**15**) and (–)-N^{in},$N^{in'}$-dicarboxybenzyl *iso*-naseseazine A (**13**) (18.1 mg, 53%, (–)-**15**:(–)-**13**, 1.4:1) as a white solid.

Regioisomers (–)-**15** and (–)-**13** were separated for the purpose of full and independent characterization by preparative HPLC [Waters X-Bridge preparative HPLC column, C18, 5 μm, 19 × 250 mm; 20.0 mL/min; gradient, 55%→65% acetonitrile in water, 15 min; t_R((–)-**15**) = 4.6 min, t_R((–)-**13**) = 5.7 min]. Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

(–)-N^{in},$N^{in'}$-Dicarboxybenzyl *iso*-Naseseazine A (13):

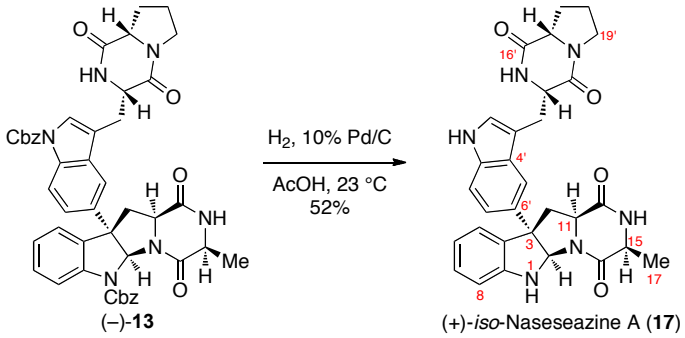
^{1}H NMR (500 MHz, $CDCl_3$, 20 ºC):

δ 8.03 (s, 1H, $C_{8'}$**H**), 7.65 (d, J = 8.2, 1H, C_8**H**), 7.49 (s, 1H, $C_{5'}$**H**), 7.43 (d, J = 7.8, 2H, Ph_{Cbz}-*o*-**H**), 7.41–7.25 (m, 8H, Ph_{Cbz}-**H**), 7.41–7.25 (m, 1H, $C_{2'}$**H**), 7.41–7.25 (m, 1H, C_5**H**), 7.18 (app-dt, J = 1.1, 8.0, 1H, C_7**H**), 7.13 (d, J = 8.9, 1H, $C_{7'}$**H**), 7.01 (app-dt, J = 0.8, 7.5, 1H, C_6**H**), 6.42 (s, 1H, C_2**H**), 5.85 (s, 1H, $N_{10'}$**H**), 5.81 (s, 1H, N_{14}**H**), 5.40 (s, 2H, $Ph_{Cbz'}C$**H**$_2$), 5.37 (d, J = 12.4, 1H, $Ph_{Cbz}C$**H**$_a$), 5.31 (d, J = 12.4, 1H, $Ph_{Cbz}C$**H**$_b$), 4.41 (dd, J = 3.5, 9.8, 1H, $C_{11'}$**H**), 4.23 (dd, J = 2.7, 9.4, 1H, $C_{11'}$**H**), 4.03–3.95 (m, 1H, C_{15}**H**), 4.03–3.95 (m, 1H, $C_{15'}$**H**), 3.59–3.38 (m, 1H, C_{12}**H**$_a$), 3.59–3.38 (m, 2H, $C_{19'}$**H**), 3.59–3.38 (m, 1H, $C_{12'}$**H**$_a$), 2.94 (dd, J = 10.0, 13.8, 1H, C_{12}**H**$_b$), 2.84 (dd, J = 9.9, 15.3, 1H, $C_{12'}$**H**$_b$), 2.30–2.19 (m, 1H, $C_{17'}$**H**$_a$), 1.94–1.77 (m, 2H, $C_{18'}$**H**), 1.94–1.77 (m, 1H, $C_{17'}$**H**$_b$), 1.33 (d, J = 6.9, 3H, C_{17}**H**).

^{13}C NMR (125.8 MHz, $CDCl_3$, 20 °C):	δ 169.5 ($\mathbf{C}_{16'}$), 169.5 ($\mathbf{C}_{13}$), 167.8 ($\mathbf{C}_{16}$), 165.0 ($\mathbf{C}_{13'}$), 153.3 ($\mathbf{C}_{Cbz}$=O), 150.5 ($\mathbf{C}_{Cbz'}$=O), 139.7 ($\mathbf{C}_{9}$), 137.2 ($\mathbf{C}_{6'}$), 136.3 ($\mathbf{C}_{Cbz'}$-*ipso*-**C**), 135.4 ($\mathbf{C}_{4}$), 135.1 ($\mathbf{C}_{Cbz}$-*ipso*-**C**), 134.9 ($\mathbf{C}_{9'}$), 130.2 ($\mathbf{C}_{4'}$), 129.2 ($\mathbf{C}_{7}$), 129.1 (Ph_{Cbz}-**C**), 129.0 (Ph_{Cbz}-**C**), 128.8 (Ph_{Cbz}-**C**), 128.7 (Ph_{Cbz}-**C**), 128.3 (Ph_{Cbz}-**C**), 128.3 (Ph_{Cbz}-**C**), 125.3 ($\mathbf{C}_{5'}$), 125.1 ($\mathbf{C}_{5}$), 124.5 ($\mathbf{C}_{6}$), 123.3 ($\mathbf{C}_{7'}$), 117.1 ($\mathbf{C}_{8}$), 116.3 ($\mathbf{C}_{8'}$), 116.0 ($\mathbf{C}_{3'}$), 115.9 ($\mathbf{C}_{2'}$), 83.6 ($\mathbf{C}_{2}$), 69.3 ($Ph_{Cbz'}\mathbf{C}H_2$), 68.2 ($Ph_{Cbz}\mathbf{C}H_2$), 59.3 ($\mathbf{C}_{15'}$), 58.6 ($\mathbf{C}_{11}$), 58.5 ($\mathbf{C}_{3}$), 54.2 ($\mathbf{C}_{11'}$), 52.0 ($\mathbf{C}_{15}$), 45.6 ($\mathbf{C}_{19'}$), 35.2 ($\mathbf{C}_{12}$), 28.4 ($\mathbf{C}_{17'}$), 26.8 ($\mathbf{C}_{12'}$), 22.7 ($\mathbf{C}_{18'}$), 15.3 ($\mathbf{C}_{17}$).
FTIR (thin film) cm^{-1}:	3251 (br, m), 1720 (s), 1688 (s), 1401 (m), 1310 (m), 751 (m).
HRMS (ESI) (*m*/*z*):	calc'd for $C_{46}H_{43}N_6O_8$ $[M+H]^+$: 807.3137 found: 807.3115.
$[\alpha]_D^{24}$:	–20 (*c* = 0.26, $CHCl_3$).
TLC (8% methanol in ethyl acetate), R*f*:	0.06 (UV, CAM).

(–)-N^{in},$N^{in'}$-Dicarboxybenzyl Naseseazine A (15):

Please see page S14 for the full characterization data for (–)-N^{in},$N^{in'}$-dicarboxybenzyl naseseazine A (**15**).



(+)-*iso*-Naseseazine A (17):

Palladium on charcoal (10% w/w, 6.0 mg, 5.67 μmol, 0.25 equiv) was added to a solution of (–)-N^{in},$N^{in'}$-dicarboxybenzyl *iso*-naseseazine A (**13**) (18.3 mg, 22.7 μmol, 1 equiv) in acetic acid (1 mL) at 23 °C. A stream of hydrogen gas was passed through the solution for 2 min by discharge of a balloon equipped with a needle extending into the reaction mixture. After stirring the solution for 20 h under an atmosphere of hydrogen gas, the solution was filtered over Celite. The solids were further extracted with methanol and the combined filtrates were concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography on silica gel (eluent: 7.5% methanol, 42.5% tetrahydrofuran, 50% dichloromethane) to afford (+)-*iso*-naseseazine A (**17**) (6.4 mg, 52.4%) as a white solid. Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

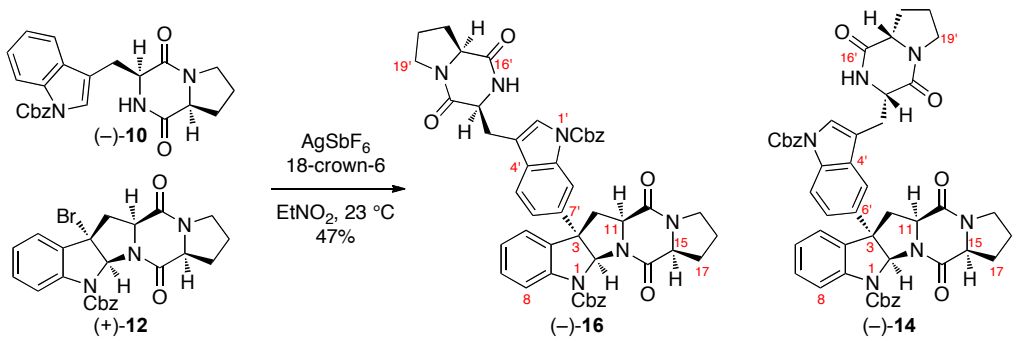
^{1}H NMR (500 MHz, methanol-d_4, 20 ºC): δ 7.71 (s, 1H, $C_{5'}$**H**), 7.32 (d, J = 8.6, 1H, $C_{8'}$**H**), 7.10 (s, 1H, $C_{2'}$**H**), 7.06 (app-dt, J = 1.0, 7.7, 1H, $C_{7'}$**H**), 7.01 (dd, J = 1.7, 8.6, 1H, $C_{7'}$**H**), 6.90 (d, J = 7.2, 1H, C_5**H**), 6.72–6.65 (m, 1H, C_8**H**), 6.72–6.65 (m, 1H, C_6**H**), 5.87 (s, 1H, C_2**H**), 4.97–4.78 (m, 1H, C_{11}**H**), 4.38 (app-t, J = 4.1, 1H, $C_{11'}$**H**), 4.23 (dq, J = 1.3, 7.0, 1H, C_{15}**H**), 3.88 (ddd, J = 1.3, 6.1, 11.0, 1H, $C_{15'}$**H**), 3.44 (dd, J = 4.6, 14.6, 1H, $C_{12'}$**H**$_a$), 3.34 (dd, J = 7.2, 13.6, 1H, C_{12}**H**$_a$), 3.25–3.15 (m, 1H, $C_{12'}$**H**$_b$), 3.25–3.15 (m, 1H, $C_{19'}$**H**$_a$), 3.06 (app-dt, J = 4.1, 10.3, 1H, $C_{19'}$**H**$_b$), 2.52 (dd, J = 10.7, 13.7, 1H, C_{12}**H**$_b$), 1.89–1.80 (m, 1H, $C_{17'}$**H**$_a$), 1.62–1.48 (m, 1H, $C_{18'}$**H**$_a$), 1.40 (d, J = 6.9, 3H, C_{17}**H**), 1.22–1.12 (m, 1H, $C_{18'}$**H**$_b$), 0.59–0.47 (m, 1H, $C_{17'}$**H**).

^{13}C NMR (125.8 MHz, methanol-d_4, 20 ºC): δ 173.1 (**C**$_{13}$), 171.0 (**C**$_{16}$), 170.2 (**C**$_{16'}$), 167.3 (**C**$_{13'}$), 149.3 (**C**$_9$), 136.8 (**C**$_{9'}$), 136.5 (**C**$_4$), 134.2 (**C**$_{6'}$), 129.5 (**C**$_7$), 128.6 (**C**$_{4'}$), 126.8 (**C**$_{2'}$), 125.1 (**C**$_5$), 123.2 (**C**$_{7'}$), 120.6 (**C**$_6$), 117.5 (**C**$_{5'}$), 113.0 (**C**$_{8'}$), 111.2 (**C**$_8$), 109.8 (**C**$_{3'}$), 87.3 (**C**$_2$), 61.5 (**C**$_3$), 60.8 (**C**$_{11}$), 60.1 (**C**$_{15'}$), 57.9 (**C**$_{11'}$), 52.4 (**C**$_{15}$), 45.9 (**C**$_{19'}$), 40.3 (**C**$_{12}$), 30.2 (**C**$_{12'}$), 29.2 (**C**$_{17'}$), 22.3 (**C**$_{18'}$), 15.3 (**C**$_{17}$).

FTIR (thin film) cm^{-1}: 3353 (br-s), 2924 (w), 1662 (s), 1466 (w), 1424 (m), 1345 (w), 1308 (m), 750 (m).

HRMS (DART) (*m*/*z*):	calc'd for $C_{30}H_{31}N_6O_4$ $[M+H]^+$: 539.2401, found: 539.2416.
$[\alpha]_D^{24}$:	+110 (*c* = 0.18, CH_3OH).

TLC (7.5% methanol, 42.5% tetrahydrofuran, 50% dichloromethane), R*f*: 0.23 (UV, CAM).



(–)-N^{in},$N^{in'}$-Dicarboxybenzyl *iso*-Naseseazine B (14):

Silver(I) hexafluoroantimonate (69.2 mg, 202 μmol, 5.00 equiv) was added as a solid to a solution of tetracyclic bromide (+)-**12** (20.0 mg, 40.3 μmol, 1 equiv) and proline diketopiperazine (–)-**10** (25.2 mg, 60.4 μmol, 1.50 equiv) in nitroethane (2 mL) at 23 ºC. After 2 h, the reaction mixture was diluted with dichloromethane (60 mL) and washed with brine (60 mL). The resulting aqueous layer was extracted with dichloromethane (2 × 30 mL) and the combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography on silica gel (eluent: gradient, 5% methanol in ethyl acetate→7.5% methanol in dichloromethane) to afford a regioisomeric mixture of (–)-N^{in},$N^{in'}$-dicarboxybenzyl naseseazine B (**16**) and (–)-N^{in},$N^{in'}$-dicarboxybenzyl *iso*-naseseazine B (**14**) (15.7 mg, 46.8%, (–)-**16**:(–)-**14**, 1.4:1) as a white solid.

Regioisomers (–)-**16** and (–)-**14** were separated for the purpose of full and independent characterization by preparative HPLC [Waters X-Bridge preparative HPLC column, C18, 5 μm, 19 × 250 mm; 20.0 mL/min; gradient, 55%→65% acetonitrile in water, 15 min; t_R((–)-**16**) = 5.5 min, t_R((–)-**14**) = 6.6 min]. Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

(–)-N^{in},$N^{in'}$-Dicarboxybenzyl *iso*-Naseseazine B (14):

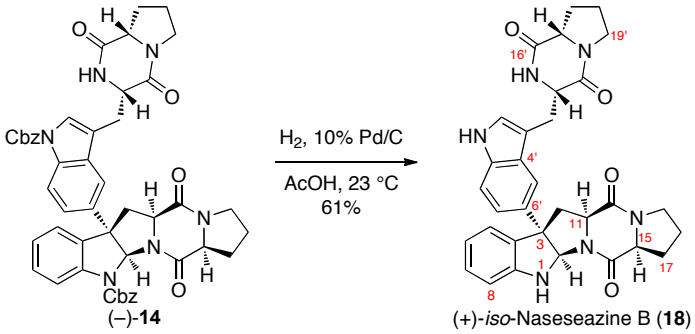
^{1}H NMR (500 MHz, $CDCl_3$, 20 ºC):

δ 8.04 (br-s, 1H, $C_{8'}$**H**), 7.65 (d, J = 8.0, 1H, C_8**H**), 7.48 (s, 1H, $C_{2'}$**H**), 7.43 (dd, J = 1.1, 7.6, 2H, Ph_{Cbz}-**H**), 7.41–7.34 (m, 5H, Ph_{Cbz}-**H**), 7.34–7.25 (m, 1H, C_5**H**), 7.34–7.25 (m, 1H, $C_{5'}$**H**), 7.34–7.25 (m, 3H, Ph_{Cbz}-**H**), 7.21–7.14 (m, 1H, C_7**H**), 7.21–7.14 (m, 1H, $C_{7'}$**H**), 7.04 (app-dt, J = 0.7, 7.5, 1H, C_6**H**), 6.42 (s, 1H, C_2**H**), 5.70 (s, 1H, $N_{10'}$**H**), 5.40 (s, 2H, $Ph_{Cbz'}C$**H**$_2$), 5.36 (d, J = 12.4, 1H, $Ph_{Cbz}C$**H**$_a$), 5.30 (d, J = 12.3, 1H, $Ph_{Cbz}C$**H**$_b$), 4.44 (dd, J = 3.5, 9.8, 1H, $C_{11'}$**H**), 4.23 (dd, J = 2.3, 9.6, 1H, $C_{11'}$**H**), 4.06 (app-t, J = 7.9, 1H, C_{15}**H**), 4.00 (app-t, J = 7.5, 1H, $C_{15'}$**H**), 3.60 (dd, J = 3.8, 13.9, 1H, C_{12}**H**$_a$), 3.58–3.45 (m, 2H, $C_{19'}$**H**), 3.43 (dd, J = 3.0, 15.2, 1H, $C_{12'}$**H**$_a$), 3.40–3.32 (m, 1H, C_{19}**H**$_a$), 3.20–3.11 (m, 1H, C_{19}**H**$_b$), 2.91 (dd, J = 10.0, 13.8, 1H, C_{12}**H**$_b$), 2.85 (dd, J = 10.1, 15.3, 1H, $C_{12'}$**H**$_b$), 2.32–2.16 (m, 1H, $C_{17'}$**H**$_a$), 2.32–2.16 (m, 1H, C_{17}**H**$_a$), 2.16–2.04 (m, 1H, C_{17}**H**$_b$), 1.98–1.65 (m,

	2H, C_{18}**H**), 1.98–1.65 (m, 2H, $C_{18'}$**H**), 1.98–1.65 (m, 1H, $C_{17'}$**H**$_b$).
^{13}C NMR (125.8 MHz, $CDCl_3$, 20 ºC):	δ 169.4 (**C**$_{16'}$), 168.0 (**C**$_{16}$), 165.5 (**C**$_{13}$), 164.9 (**C**$_{13'}$), 153.3 (**C**$_{Cbz}$=O), 150.5 (**C**$_{Cbz'}$=O), 139.7 (**C**$_9$), 137.3 (**C**$_{6'}$), 136.3 (Ph$_{Cbz}$-*ipso*-**C**), 135.3 (**C**$_4$), 135.0 (**C**$_{9'}$), 134.9 (Ph$_{Cbz'}$-*ipso*-**C**), 130.1 (**C**$_{4'}$), 129.1 (**C**$_7$), 129.1 (Ph$_{Cbz}$-**C**), 129.0 (Ph$_{Cbz}$-**C**), 128.8 (Ph$_{Cbz}$-**C**), 128.7 (Ph$_{Cbz}$-**C**), 128.2 (Ph$_{Cbz}$-**C**), 128.2 (Ph$_{Cbz}$-**C**), 125.3 (**C**$_5$), 125.2 (**C**$_{2'}$), 124.5 (**C**$_6$), 123.4 (**C**$_{7'}$), 116.8 (**C**$_8$), 116.3 (**C**$_{8'}$), 115.9 (**C**$_{5'}$), 115.9 (**C**$_{3'}$), 83.5 (**C**$_2$), 69.3 (Ph$_{Cbz'}$**C**H$_2$), 68.2 (Ph$_{Cbz}$**C**H$_2$), 61.1 (**C**$_{15}$), 59.7 (**C**$_{11}$), 59.3 (**C**$_{15'}$), 58.7 (**C**$_3$), 54.2 (**C**$_{11'}$), 45.6 (**C**$_{19'}$), 45.2 (**C**$_{19}$), 35.2 (**C**$_{12}$), 28.4 (**C**$_{17'}$), 27.6 (**C**$_{17}$), 26.8 (**C**$_{12'}$), 23.6 (**C**$_{18}$), 22.7 (**C**$_{18'}$).
FTIR (thin film) cm^{-1}:	3584 (m), 2954 (m), 1720 (s), 1677 (s), 1400 (s), 1254 (m), 750 (m).
HRMS (ESI) (*m*/*z*):	calc'd for $C_{48}H_{45}N_6O_8$ $[M+H]^+$: 833.3293, found: 833.3291.
$[\alpha]_D^{24}$:	–32 (*c* = 0.39, $CHCl_3$).
TLC (8% methanol in ethyl acetate), R*f*:	0.05 (UV, CAM).

(–)-*N*^{in},*N*$^{in'}$-Dicarboxybenzyl Naseseazine B (16):

Please see page S21 for the full characterization data for (–)-*N*^{in},*N*$^{in'}$-dicarboxybenzyl naseseazine B (**16**).



(+)-*iso*-Naseseazine B (18):

Palladium on charcoal (10% w/w, 4.4 mg, 4.11 μmol, 0.25 equiv) was added to a solution of (–)-N^{in},$N^{in'}$-dicarboxybenzyl *iso*-naseseazine B (**14**) (13.7 mg, 16.5 μmol, 1 equiv) in acetic acid (1 mL) at 23 °C. A stream of hydrogen gas was passed through the solution for 2 min by discharge of a balloon equipped with a needle extending into the reaction mixture. After stirring the solution for 18 h under an atmosphere of hydrogen gas, the solution was filtered over Celite. The solids were further extracted with methanol and the combined filtrates were concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography on silica gel (eluent: 7.5% methanol, 42.5% tetrahydrofuran, 50% dichloromethane) to afford (+)-*iso*-naseseazine B (**18**) (5.7 mg, 61.4%) as a white solid. Structural assignments were made using additional information from gCOSY, HSQC, HMBC, and nOe experiments.

^{1}H NMR (500 MHz, $CDCl_3$, 20 °C):

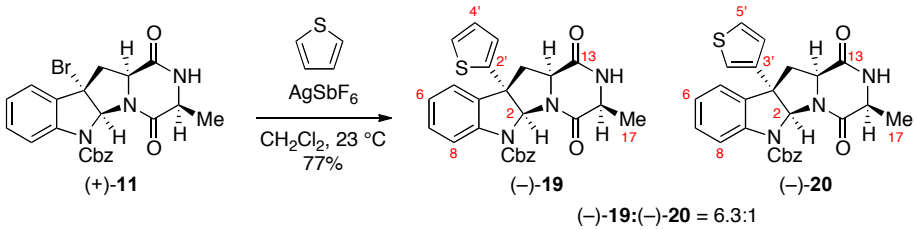
δ 8.28 (s, 1H, $N_{1'}$**H**), 7.59 (s, 1H, $C_{5'}$**H**), 7.33 (d, *J* = 8.6, 1H, $C_{8'}$**H**), 7.14–7.09 (m, 1H, $C_{2'}$**H**), 7.14–7.09 (m, 1H, $C_{7'}$**H**), 7.06 (app-dt, *J* = 0.9, 7.6, 1H, C_7**H**), 6.84 (d, *J* = 7.4, 1H, C_5**H**), 6.68 (app-t, *J* = 7.4, 1H, C_6**H**), 6.63 (d, *J* = 8.0, 1H, C_8**H**), 5.87 (d, *J* = 3.2, 1H, C_2**H**), 5.76 (s, 1H, $N_{10'}$**H**), 5.29 (d, *J* = 3.1, 1H, N_1**H**), 4.67 (dd, *J* = 7.3, 9.5, 1H, C_{11}**H**), 4.33 (dd, *J* = 3.0, 10.7, 1H, $C_{11'}$**H**), 4.18 (app-t, *J* = 8.2, 1H, C_{15}**H**), 4.08 (app-t, *J* = 7.8, 1H, $C_{15'}$**H**), 3.66 (dd, *J* = 3.5, 14.9, 1H, $C_{12'}$$\mathbf{H}_a$), 3.63–3.43 (m, 2H, C_{19}**H**), 3.63–3.43 (m, 2H, $C_{19'}$**H**), 3.31 (dd, *J* = 7.1, 13.9, 1H, $C_{12}$$\mathbf{H}_a$), 2.99 (dd, *J* = 10.5, 15.0, 1H, $C_{12'}$$\mathbf{H}_b$), 2.74 (dd, *J* = 10.5, 13.8, 1H, $C_{12}$$\mathbf{H}_b$), 2.36–2.22 (m, 1H, $C_{17'}$$\mathbf{H}_a$), 2.36–2.22 (m, 1H, $C_{17}$$\mathbf{H}_a$), 2.22–2.08 (m, 1H, $C_{17}$$\mathbf{H}_b$), 2.08–1.76 (m, 2H, C_{18}**H**), 2.08–1.76 (m, 2H, $C_{18'}$**H**), 2.08–1.76 (m, 1H, $C_{17'}$$\mathbf{H}_b$).

^{13}C NMR (125.8 MHz, $CDCl_3$, 20 °C):

δ 169.5 (**C**$_{16'}$), 168.2 (**C**$_{16}$), 166.2 (**C**$_{13}$), 165.4 (**C**$_{13'}$), 147.3 (**C**$_9$), 135.8 (**C**$_{9'}$), 134.7 (**C**$_4$), 134.3 (**C**$_{6'}$), 128.7 (**C**$_7$), 127.0 (**C**$_{4'}$), 124.4 (**C**$_{2'}$), 124.3 (**C**$_5$), 123.1 (**C**$_{7'}$), 119.9 (**C**$_6$), 115.9 (**C**$_{5'}$), 112.4 (**C**$_{8'}$), 110.3 (**C**$_{3'}$), 109.8 (**C**$_8$), 86.2 (**C**$_2$), 60.8 (**C**$_{11}$), 60.7 (**C**$_{15}$), 60.3 (**C**$_3$), 59.4 (**C**$_{15'}$), 55.2 (**C**$_{11'}$), 45.6 (**C**$_{19/19'}$), 45.4 (**C**$_{19/19'}$), 38.6 (**C**$_{12}$), 28.5 (**C**$_{17'}$), 27.8 (**C**$_{17}$), 27.1 (**C**$_{12'}$), 23.5 (**C**$_{18}$), 22.7 (**C**$_{18'}$).

FTIR (thin film) cm^{-1}:	3306 (br, s), 2925 (w), 1660 (s), 1428 (m), 1341 (w), 1312 (w), 1215 (w), 750 (m).
HRMS (ESI) (*m*/*z*):	calc'd for $C_{32}H_{33}N_6O_4$ $[M+H]^+$: 565.2558, found: 565.2544.
$[\alpha]_D^{24}$:	+43 (*c* = 0.15, $CHCl_3$).

TLC (5% methanol, 45% tetrahydrofuran, 50% dichloromethane), R*f*: 0.18 (UV, CAM).



3-(2-Thienyl)tetracycle (–)-19 and 3-(3-Thienyl)tetracycle (–)-20:

A solution of silver(I) hexafluoroantimonate (0.20 M, 425 µL, 85.1 µmol, 2.00 equiv) in dichloromethane was added via syringe to a solution of tetracyclic bromide (+)-**11** (20.0 mg, 42.5 µmol, 1 equiv) and thiophene (6.7 µL, 85 µmol, 2.0 equiv) in dichloromethane (2 mL) at 23 ºC. After 1 h, brine (200 µL) was added to the solution. The crude reaction mixture was purified by flash column chromatography on silica gel (eluent: 20% acetone in dichloromethane) to afford a mixture of regioisomeric 3-(thienyl)tetracycles (Run 1: 15.3 mg, 76.0%, (–)-**19**:(–)-**20**, 6.2:1; Run 2: 15.6 mg, 77.5%, (–)-**19**:(–)-**20**, 6.4:1) as a clear film.

Regioisomers (–)-**19** and (–)-**20** were separated for the purpose of full and independent characterization by preparative HPLC [Waters X-Bridge preparative HPLC column, C18, 5 µm, 19 × 250 mm; 20.0 mL/min; 50% acetonitrile in water; t_R((–)-**19**) = 8.4 min, t_R((–)-**20**) = 8.7 min]. Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

3-(2-Thienyl)tetracycle (–)-19:

^{1}H NMR (500 MHz, $CDCl_3$, 20 ºC): δ 7.68 (d, J = 8.3, 1H, C_8**H**), 7.38 (d, J = 7.9, 2H, Ph_{Cbz}-*o*-**H**), 7.35–7.27 (m, 3H, Ph_{Cbz}-**H**), 7.35–7.27 (m, 1H, C_5**H**), 7.24 (app-dt, J = 1.3, 7.8, 1H, C_7**H**), 7.17 (dd, J = 1.2, 5.1, 1H, $C_{5'}$**H**), 7.06 (app-dt, J = 1.0, 7.6, 1H, C_6**H**), 6.84 (dd, J = 3.6, 5.1, 1H, $C_{4'}$**H**), 6.63 (dd, J = 1.2, 3.6, 1H, $C_{3'}$**H**), 6.22 (s, 1H, C_2**H**), 5.46 (s, 1H, N_{14}**H**), 5.38 (d, J = 12.3, 1H, $Ph_{Cbz}C$**H**$_a$), 5.28 (d, J = 12.3, 1H, $Ph_{Cbz}C$**H**$_b$), 4.40 (dd, J = 3.3, 10.0, 1H, C_{11}**H**), 3.99 (q, J = 6.9, 1H, C_{15}**H**), 3.54 (dd, J = 3.5, 14.0, 1H, C_{12}**H**$_a$), 2.93 (dd, J = 10.1, 13.9, 1H, C_{12}**H**$_b$), 1.33 (d, J = 6.9, 3H, C_{17}**H**).

^{13}C NMR (125.8 MHz, $CDCl_3$, 20 ºC): δ 169.3 (**C**$_{13}$), 167.7 (**C**$_{16}$), 153.2 (**C**$_{Cbz}$=O), 145.3 (**C**$_{2'}$), 139.9 (**C**$_9$), 136.3 (Ph_{Cbz}-*ipso*-**C**), 134.6 (**C**$_4$), 129.6 (**C**$_7$), 128.7 (Ph_{Cbz}-**C**), 128.3 (Ph_{Cbz}-**C**), 128.2 (Ph_{Cbz}-**C**), 127.3 (**C**$_{4'}$), 125.3 (**C**$_{5'}$), 124.9 (**C**$_5$), 124.9 (**C**$_{3'}$), 124.4 (**C**$_6$), 117.1 (**C**$_8$), 83.9 (**C**$_2$), 68.1 (Ph_{Cbz}**C**H$_2$), 58.4 (**C**$_{11}$), 56.3 (**C**$_3$), 51.9 (**C**$_{15}$), 35.5 (**C**$_{12}$), 15.3 (**C**$_{17}$).

FTIR (thin film) cm^{-1}: 3260 (br, m), 1712 (s), 1689 (s), 1409 (m), 1311 (m).

HRMS (ESI) (*m*/*z*): calc'd for $C_{26}H_{24}N_3O_4S$ [M+H]$^+$: 474.1482, found: 474.1470.

$[\alpha]_D^{24}$: –35 (*c* = 0.33, $CHCl_3$).

TLC (ethyl acetate), R*f*: 0.47 (UV, CAM).

3-(3-Thienyl)tetracycle (−)-20:

^{1}H NMR (500 MHz, $CDCl_3$, 20 ºC): δ 7.66 (d, *J* = 8.1, 1H, C_8**H**), 7.37 (dd, *J* = 1.8, 8.3, 2H, Ph_{Cbz}-*o*-**H**), 7.35–7.30 (m, 3H, Ph_{Cbz}-**H**), 7.29 (d, *J* = 7.3, 1H, C_5**H**), 7.26 (dd, *J* = 3.0, 5.1, 1H, $C_{4'}$**H**), 7.21 (app-dt, *J* = 1.3, 7.8, 1H, C_7**H**), 7.05 (app-dt, *J* = 1.0, 7.5, 1H, C_6**H**), 6.90 (dd, *J* = 1.4, 5.1, 1H, $C_{5'}$**H**), 6.80 (dd, *J* = 1.4, 3.0, 1H, $C_{2'}$**H**), 6.24 (s, 1H, C_2**H**), 5.54 (s, 1H, N_{14}**H**), 5.40 (d, *J* = 12.4, 1H, $Ph_{Cbz}C$**H**$_a$), 5.28 (d, *J* = 12.3, 1H, $Ph_{Cbz}C$**H**$_b$), 4.37 (dd, *J* = 3.4, 10.1, 1H, C_{11}**H**), 3.99 (q, *J* = 6.9, 1H, C_{15}**H**), 3.47 (dd, *J* = 3.4, 13.8, 1H, C_{12}**H**$_a$), 2.84 (dd, *J* = 10.1, 13.9, 1H, C_{12}**H**$_b$), 1.33 (d, *J* = 6.9, 3H, C_{17}**H**).

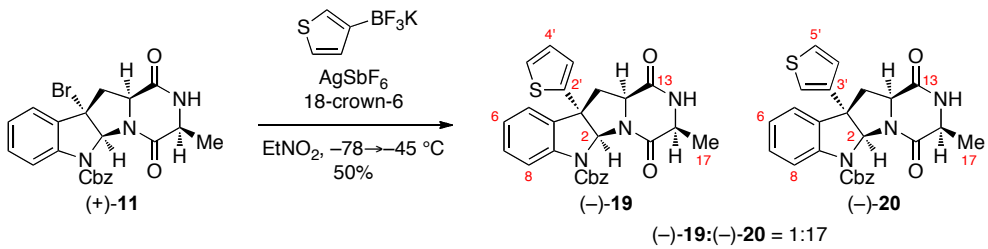
^{13}C NMR (125.8 MHz, $CDCl_3$, 20 ºC): δ 169.3 (**C**$_{13}$), 167.7 (**C**$_{16}$), 153.2 (**C**$_{Cbz}$=O), 142.3 (**C**$_{3'}$), 139.9 (**C**$_9$), 136.3 (Ph_{Cbz}-*ipso*-**C**), 134.8 (**C**$_4$), 129.3 (**C**$_7$), 128.7 (Ph_{Cbz}-**C**), 128.3 (Ph_{Cbz}-**C**), 128.3 (Ph_{Cbz}-**C**), 127.5 (**C**$_{4'}$), 125.5 (**C**$_{5'}$), 124.8 (**C**$_5$), 124.4 (**C**$_6$), 121.5 (**C**$_{2'}$), 117.1 (**C**$_8$), 83.0 (**C**$_2$), 68.2 (Ph_{Cbz}**C**H$_2$), 58.4 (**C**$_{11}$), 56.2 (**C**$_3$), 51.9 (**C**$_{15}$), 34.4 (**C**$_{12}$), 15.3 (**C**$_{17}$).

FTIR (thin film) cm^{-1}: 3268 (br, m), 1709 (s), 1689 (s), 1481 (m), 1409 (m).

HRMS (ESI) (*m*/*z*): calc'd for $C_{26}H_{24}N_3O_4S$ $[M+H]^+$: 474.1482, found: 474.1465.

$[\alpha]_D^{24}$: −17 (*c* = 0.21, $CHCl_3$).

TLC (ethyl acetate), R*f*: 0.43 (UV, CAM).



3-(2-Thienyl)tetracycle (–)-19 and 3-(3-Thienyl)tetracycle (–)-20:

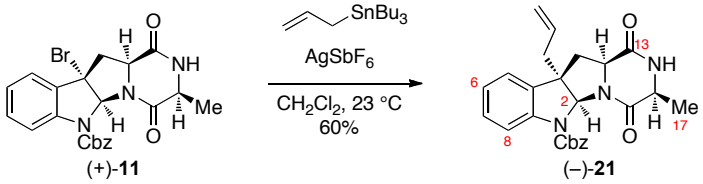
A Schlenk tube was charged with tetracyclic bromide (+)-**11** (20.0 mg, 42.5 μmol, 1 equiv), potassium 3-thiophenetrifluoroborate (16.2 mg, 85.1 μmol, 2.00 equiv), and 18-crown-6 (22.4 mg, 85.1 μmol, 2.00 equiv). Nitroethane (2 mL) was then introduced via syringe. Upon dissolution of all solid components, the reaction mixture was cooled to –78 °C. A solution of silver(I) hexafluoroantimonate (0.17 M, 500 μL, 85.1 μmol, 2.00 equiv) in nitroethane at –78 °C was then introduced to the Schlenk tube via cannula. The vessel was subsequently fitted with a PTFE screw cap, sealed, and introduced into a cold bath at –45 °C. After 12 h, aqueous hydrogen chloride (2 N, 4 mL) was added. The reaction mixture was diluted with ethyl acetate (60 mL) and washed with brine (3 × 60 mL). The organic layer was dried over anhydrous sodium sulfate, was filtered, and was concentrated under reduced pressure. The residue was diluted with dichloromethane (30 mL) and washed with brine (30 mL). The resulting aqueous layer was extracted with dichloromethane (2 × 30 mL). The combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography on silica gel (eluent: 90% ethyl acetate in hexanes) to afford a mixture of regioisomeric 3-(thienyl)tetracycles (Run 1: 10.4 mg, 51.6%, (–)-**19**:(–)-**20**, 1:17; Run 2: 9.5 mg, 47%, (–)-**19**:(–)-**20**, 1:17) as a clear film.

3-(2-Thienyl)tetracycle (–)-19:

Please see page S33 for the full characterization data for 3-(2-thienyl)tetracycle (–)-**19**.

3-(3-Thienyl)tetracycle (–)-20:

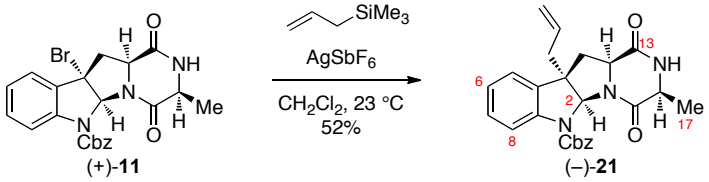
Please see page S34 for the full characterization data for 3-(3-thienyl)tetracycle (–)-**20**.



3-Allyltetracycle (–)-21:

A solution of silver(I) hexafluoroantimonate (0.20 M, 425 μL, 85.1 μmol, 2.00 equiv) in dichloromethane was added via syringe to a solution of tetracyclic bromide (+)-**11** (20.0 mg, 42.5 μmol, 1 equiv) and allyltributylstannane (26.1 μL, 85.1 μmol, 2.00 equiv) in dichloromethane (2 mL) at 23 ºC. After 1 h, brine (200 μL) was added to the solution. The crude reaction mixture was then purified by flash column chromatography on silica gel (eluent: 20% acetone in dichloromethane) to afford 3-allyl tetracycle (–)-**21** (Run 1: 11.1 mg, 60.5%; Run 2: 10.9 mg, 59.4%) as a clear film. Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

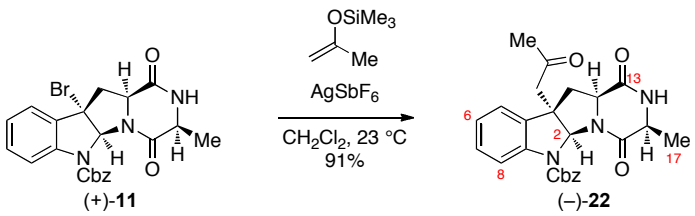
^{1}H NMR (500 MHz, $CDCl_3$, 20 ºC):	δ 7.61 (d, *J* = 7.0, 1H, C_8**H**), 7.42 (d, *J* = 8.3, 2H, Ph_{Cbz}-*o*-**H**), 7.37–7.27 (m, 3H, Ph_{Cbz}-**H**), 7.21–7.15 (m, 1H, C_7**H**), 7.21–7.15 (m, 1H, C_5**H**), 7.01 (app-dt, *J* = 0.9, 7.5, 1H, C_6**H**), 5.93 (s, 1H, C_2**H**), 5.84 (br-s, 1H, N_{14}**H**), 5.47–5.40 (m, 1H, CH_2**C**H=CH_2), 5.41 (d, *J* = 12.3, 1H, $Ph_{Cbz}C$**H**$_a$), 5.27 (d, *J* = 12.3, 1H, $Ph_{Cbz}C$**H**$_b$), 5.03–4.94 (m, 2H, CH_2CH=C**H**$_2$), 4.19 (dd, *J* = 3.9, 10.1, 1H, C_{11}**H**), 3.93 (q, *J* = 6.8, 1H, C_{15}**H**), 3.03 (dd, *J* = 4.0, 13.8, 1H, C_{12}**H**$_a$), 2.44–2.28 (m, 2H, C**H**$_2$CH=CH_2), 2.44–2.28 (m, 1H, C_{12}**H**$_b$), 1.30 (d, *J* = 6.8, 3H, C_{17}**H**).
^{13}C NMR (125.8 MHz, $CDCl_3$, 20 ºC):	δ 169.5 (**C**$_{13}$), 167.6 (**C**$_{16}$), 153.3 (**C**$_{Cbz}$=O), 140.3 (**C**$_9$), 136.4 (Ph_{Cbz}-*ipso*-**C**), 135.0 (**C**$_4$), 132.2 (CH_2**C**H=CH_2), 128.9 (**C**$_7$), 128.7 (Ph_{Cbz}-**C**), 128.4 (Ph_{Cbz}-**C**), 128.3 (Ph_{Cbz}-**C**), 124.2 (**C**$_5$), 124.1 (**C**$_6$), 119.9 (CH_2CH=**C**H_2), 116.8 (**C**$_8$), 81.1 (**C**$_2$), 68.1 (Ph_{Cbz}**C**H_2), 58.1 (**C**$_{11}$), 54.7 (**C**$_3$), 51.9 (**C**$_{15}$), 42.4 (**C**H_2CH=CH_2), 32.8 (**C**$_{12}$), 15.4 (**C**$_{17}$).
FTIR (thin film) cm^{-1}:	3264 (br, m), 1687 (s), 1481 (m), 1410 (m), 1328 (m).
HRMS (ESI) (*m*/*z*):	calc'd for $C_{25}H_{26}N_3O_4$ $[M+H]^+$: 432.1918, found: 432.1906.
$[\alpha]_D^{24}$:	–37 (*c* = 0.26, $CHCl_3$).
TLC (ethyl acetate), R*f*:	0.34 (UV, CAM).



3-Allyltetracycle (–)-21:

A solution of silver(I) hexafluoroantimonate (0.20 M, 425 μL, 85.1 μmol, 2.00 equiv) in dichloromethane was added via syringe to a solution of tetracyclic bromide (+)-**11** (20.0 mg, 42.5 μmol, 1 equiv) and allyltrimethylsilane (13.5 μL, 85.1 μmol, 2.00 equiv) in dichloromethane (2 mL) at 23 ºC. After 1 h, brine (200 μL) was added to the solution. The crude reaction mixture was purified by flash column chromatography on silica gel (eluent: 20% acetone in dichloromethane) to afford 3-allyl tetracycle (–)-**21** (Run 1: 9.5 mg, 52%; Run 2: 9.6 mg, 52%) as a clear film.

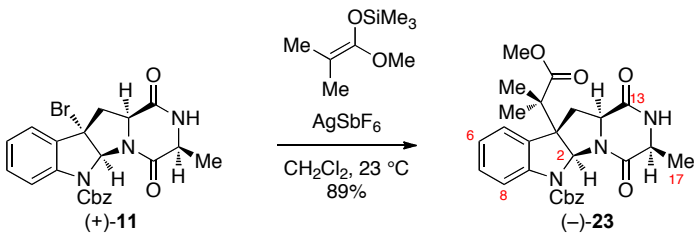
Please see page S36 for the full characterization data for 3-allyltetracycle (–)-**21**.



3-Acetonyltetracycle (–)-22:

A solution of silver(I) hexafluoroantimonate (0.20 M, 425 μL, 85.1 μmol, 2.00 equiv) in dichloromethane was added via syringe to a solution of tetracyclic bromide (+)-**11** (20.0 mg, 42.5 μmol, 1 equiv) and (isopropenyloxy)trimethylsilane (85% purity, 16.6 μL, 85.1 μmol, 2.00 equiv) in dichloromethane (2 mL) at 23 ºC. After 1 h, brine (200 μL) was added to the solution. The crude reaction mixture was purified by flash column chromatography on silica gel (eluent: 20% acetone in dichloromethane) to afford 3-acetonyltetracycle (–)-**22** (Run 1: 17.3 mg, 90.9%; Run 2: 17.2 mg, 90.4%) as a clear film. Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

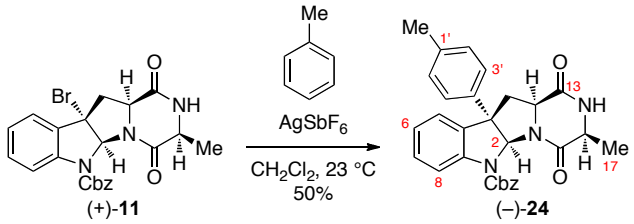
^{1}H NMR (500 MHz, $CDCl_3$, 20 ºC):	δ 7.60 (app-s, 1H, C_8**H**), 7.41 (d, *J* = 7.1, 2H, Ph_{Cbz}-*o*-**H**), 7.34 (app-t, *J* = 6.8, 2H, Ph_{Cbz}-*m*-**H**), 7.32–7.27 (m, 1H, Ph_{Cbz}-*p*-**H**), 7.19 (d, *J* = 7.5, 1H, C_5**H**), 7.16 (app-t, *J* = 7.8, 1H, C_7**H**), 6.99 (app-t, *J* = 7.5, 1H, C_6**H**), 6.29 (br-s, 1H, N_{14}**H**), 6.12 (s, 1H, C_2**H**), 5.39 (d, *J* = 12.3, 1H, $Ph_{Cbz}C$**H**$_a$), 5.27 (d, *J* = 12.3, 1H, $Ph_{Cbz}C$**H**$_b$), 4.42 (dd, *J* = 5.3, 9.8, 1H, C_{11}**H**), 3.97 (q, *J* = 6.7, 1H, C_{15}**H**), 2.98 (dd, *J* = 5.4, 13.5, 1H, C_{12}**H**$_a$), 2.90 (d, *J* = 18.4, 1H, C**H**$_2C$(=O)CH_3), 2.65 (d, *J* = 18.4, 1H, C**H**$_2C$(=O)CH_3), 2.52 (dd, *J* = 10.1, 13.5, 1H, C_{12}**H**$_b$), 2.04 (s, 3H, CH_2C(=O)C**H**$_3$), 1.31 (d, *J* = 6.8, 3H, C_{17}**H**).
^{13}C NMR (125.8 MHz, $CDCl_3$, 20 ºC):	δ 206.0 (CH_2**C**(=O)CH_3), 170.0 (**C**$_{13}$), 167.7 (**C**$_{16}$), 153.6 (**C**$_{Cbz}$=O), 139.8 (**C**$_9$), 136.2 (Ph_{Cbz}-*ipso*-**C**), 135.9 (**C**$_4$), 129.0 (**C**$_7$), 128.7 (Ph_{Cbz}-**C**), 128.5 (Ph_{Cbz}-**C**), 128.3 (Ph_{Cbz}-**C**), 124.4 (**C**$_6$), 124.3 (**C**$_5$), 116.9 (**C**$_8$), 80.5 (**C**$_2$), 68.2 (Ph_{Cbz}**C**H_2), 58.2 (**C**$_{11}$), 51.9 (**C**$_{15}$), 51.4 (**C**$_3$), 49.2 (**C**H_2C(=O)CH_3), 33.4 (**C**$_{12}$), 31.0 (CH_2C(=O)**C**H_3), 15.6 (**C**$_{17}$).
FTIR (thin film) cm^{-1}:	3271 (br, m), 1712 (s), 1688 (s), 1481(m), 1410 (m).
HRMS (ESI) (*m*/*z*):	calc'd for $C_{25}H_{26}N_3O_5$ $[M+H]^+$: 448.1867, found: 448.1846.
$[\alpha]_D^{24}$:	–59 (*c* = 0.52, $CHCl_3$).
TLC (ethyl acetate), R*f*:	0.23 (UV, CAM).



3-(Methyl-α,α-dimethylacetate)tetracycle (–)-23:

A solution of silver(I) hexafluoroantimonate (0.20 M, 425 μL, 85.1 μmol, 2.00 equiv) in dichloromethane was added via syringe to a solution of tetracyclic bromide (+)-**11** (20.0 mg, 42.5 μmol, 1 equiv) and methyl trimethysilyl dimethylketene acetal (17.2 μL, 85.1 μmol, 2.00 equiv) in dichloromethane (2 mL) at 23 ºC. After 1 h, brine (200 μL) was added to the solution. The crude reaction mixture was purified by flash column chromatography on silica gel (eluent: 20% acetone in dichloromethane) to afford 3-(methyl-α,α-dimethylacetate)tetracycle (–)-**23** (Run 1: 18.4 mg, 88.2%; Run 2: 18.8 mg, 89.9%) as a clear film. Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

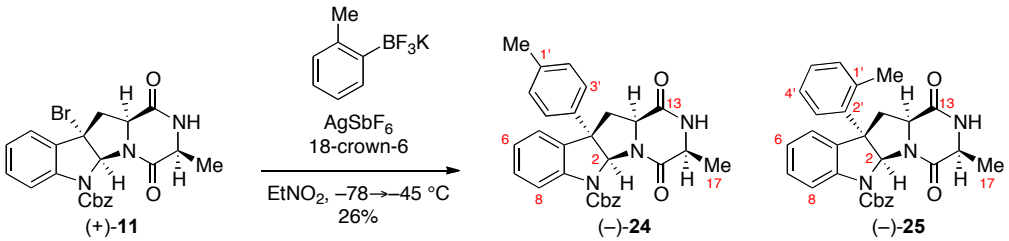
^{1}H NMR (500 MHz, $CDCl_3$, 20 ºC):	δ 7.69 (app-s, 1H, C_8**H**), 7.38 (m, 2H, Ph_{Cbz}-*o*-**H**), 7.32 (app-t, *J* = 7.5, 2H, Ph_{Cbz}-*m*-**H**), 7.29–7.25 (m, 1H, Ph_{Cbz}-*p*-**H**), 7.20 (app-t, *J* = 7.2, 1H, C_7**H**), 7.18 (d, *J* = 7.6, 1H, C_5**H**), 7.01 (app-dt, *J* = 0.9, 7.6, 1H, C_6**H**), 6.45 (s, 1H, C_2**H**), 6.17 (br-s, 1H, N_{14}**H**), 5.58–5.08 (br-m, 2H, $Ph_{Cbz}C$**H**$_2$), 4.38–4.27 (br-m, 1H, C_{11}**H**), 3.95 (q, *J* = 6.2, 1H, C_{15}**H**), 3.63 (s, 3H, OC**H**$_3$), 2.91–2.75 (br-m, 1H, C_{12}**H**$_a$), 2.71–2.57 (br-m, 1H, C_{12}**H**$_b$), 1.32 (d, *J* = 6.8, 3H, C_{17}**H**), 1.26 (s, 3H, C(C**H**$_3$)$_2$), 0.88 (s, 3H, C(C**H**$_3$)$_2$).
^{13}C NMR (125.8 MHz, $CDCl_3$, 20 ºC):	δ 176.6 (**C**O_2CH_3), 170.1 (**C**$_{13}$), 167.7 (**C**$_{16}$), 153.1 (**C**$_{Cbz}$=O), 141.2 (**C**$_9$), 136.5 (Ph_{Cbz}-*ipso*-**C**), 133.0 (**C**$_8$), 129.3 (**C**$_7$), 128.6 (Ph_{Cbz}-**C**), 128.2 (Ph_{Cbz}-**C**), 128.1 (Ph_{Cbz}-**C**), 126.3 (**C**$_5$), 123.9 (**C**$_6$), 117.2 (**C**$_4$), 80.3 (**C**$_2$), 68.1 (Ph_{Cbz}**C**H$_2$), 58.7 (**C**$_3$), 58.2 (**C**$_{11}$), 52.4 (O**C**H$_3$), 51.9 (**C**$_{15}$), 47.7 (**C**(CH$_3$)$_2$), 33.4 (**C**$_{12}$), 21.7 (C(**C**H$_3$)$_2$), 21.7 (C(**C**H$_3$)$_2$), 15.7 (**C**$_{17}$).
FTIR (thin film) cm^{-1}:	3266 (br, m), 1717 (s), 1688 (s), 1482 (m), 1410 (m).
HRMS (ESI) (*m*/*z*):	calc'd for $C_{27}H_{30}N_3O_6$ [M+H]$^+$: 492.2129, found: 492.2137.
$[\alpha]_D^{24}$:	–52 (*c* = 0.29, $CHCl_3$).
TLC (ethyl acetate), R*f*:	0.38 (UV, CAM).



3-(4-Tolyl)tetracycle (–)-24:

A solution of silver(I) hexafluoroantimonate (0.20 M, 425 μL, 85.1 μmol, 2.00 equiv) in dichloromethane was added via syringe to a solution of tetracyclic bromide (+)-**11** (20.0 mg, 42.5 μmol, 1 equiv) and toluene (9.1 μL, 85 μmol, 2.0 equiv) in dichloromethane (2 mL) at 23 ºC. After 1 h, brine (200 μL) was added to the solution. The crude reaction mixture was purified by flash column chromatography on silica gel (eluent: 20% acetone in dichloromethane) to afford 3-(4-tolyl)tetracycle (–)-**24** (Run 1: 11.1 mg, 54.2%; Run 2: 9.3 mg, 45%) as a clear film. Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

^{1}H NMR (500 MHz, $CDCl_3$, 20 ºC):	δ 7.64 (d, *J* = 7.9, 1H, C_8**H**), 7.37 (d, *J* = 7.5, 2H, Ph_{Cbz}-*o*-**H**), 7.35–7.26 (m, 3H, Ph_{Cbz}-**H**), 7.35–7.26 (m, 1H, C_5**H**), 7.18 (app-t, *J* = 7.9, 1H, C_7**H**), 7.07–7.00 (m, 2H, $C_{3'}$**H**), 7.07–7.00 (m, 2H, $C_{2'}$**H**), 7.07–7.00 (m, 1H, C_6**H**), 6.34 (s, 1H, C_2**H**), 5.85 (s, 1H, N_{14}**H**), 5.41 (d, *J* = 12.4, 1H, $Ph_{Cbz}C$**H**$_a$), 5.28 (d, *J* = 12.3, 1H, $Ph_{Cbz}C$**H**$_b$), 4.37 (dd, *J* = 3.4, 10.1, 1H, C_{11}**H**), 3.98 (q, *J* = 6.8, 1H, C_{15}**H**), 3.49 (dd, *J* = 3.4, 13.8, 1H, C_{12}**H**$_a$), 2.85 (dd, *J* = 10.3, 13.9, 1H, C_{12}**H**$_b$), 2.26 (s, 3H, ArC**H**$_3$), 1.33 (d, *J* = 6.8, 3H, C_{17}**H**).
^{13}C NMR (125.8 MHz, $CDCl_3$, 20 ºC):	δ 169.6 (**C**$_{13}$), 167.7 (**C**$_{16}$), 153.1 (**C**$_{Cbz}$=O), 139.8 (**C**$_9$), 138.8 (**C**$_{4'}$), 137.5 (**C**$_{1'}$), 136.3 (Ph_{Cbz}-*ipso*-**C**), 135.1 (**C**$_4$), 129.8 (**C**$_{2'}$), 129.1 (**C**$_7$), 128.7 (Ph_{Cbz}-**C**), 128.2 (Ph_{Cbz}-**C**), 128.2 (Ph_{Cbz}-**C**), 125.7 (**C**$_{3'}$), 125.1 (**C**$_5$), 124.3 (**C**$_6$), 117.0 (**C**$_8$), 83.3 (**C**$_2$), 68.1 (Ph_{Cbz}**C**H$_2$), 58.6 (**C**$_{11}$), 58.3 (**C**$_3$), 51.9 (**C**$_{15}$), 35.0 (**C**$_{12}$), 21.1 (Ar**C**H$_3$), 15.3 (**C**$_{17}$).
FTIR (thin film) cm^{-1}:	3268 (br, m), 1712 (s), 1689 (s), 1409 (m), 1313 (m).
HRMS (ESI) (*m*/*z*):	calc'd for $C_{29}H_{28}N_3O_4$ $[M+H]^+$: 482.2074, found: 482.2056.
$[\alpha]_D^{24}$:	–6.4 (*c* = 0.28, $CHCl_3$).
TLC (20% acetone in dichloromethane), R*f*:	0.22 (UV, CAM).



3-(4-Tolyl)tetracycle (–)-24 and 3-(2-Tolyl)tetracycle (–)-25:

A Schlenk tube was charged with tetracyclic bromide (+)-**11** (20.0 mg, 42.5 μmol, 1 equiv), potassium *o*-tolyltrifluoroborate (16.8 mg, 85.1 μmol, 2.00 equiv), and 18-crown-6 (22.4 mg, 85.1 μmol, 2.00 equiv). Nitroethane (2 mL) was then introduced via syringe. Upon dissolution of all solid components, the reaction mixture was cooled to –78 ºC. A solution of silver(I) hexafluoroantimonate (0.17 M, 500 μL, 85.1 μmol, 2.00 equiv) in nitroethane at –78 °C was then introduced to the Schlenk tube via cannula. The vessel was subsequently fitted with a PTFE screw cap, sealed, and introduced into a cold bath at –45 °C. After 12 h, aqueous hydrogen chloride (2 N, 4 mL) was added. The reaction mixture was diluted with ethyl acetate (60 mL) and washed with brine (3 × 60 mL). The organic layer was dried over anhydrous sodium sulfate, was filtered, and was concentrated under reduced pressure. The residue was diluted with dichloromethane (30 mL) and washed with brine (30 mL). The resulting aqueous layer was extracted with dichloromethane (2 × 30 mL). The combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography on silica gel (eluent: 90% ethyl acetate in hexanes) to afford a mixture of regioisomeric 3-(tolyl)tetracycles (Run 1: 5.1 mg, 25%, (–)-**24**:(–)-**25**, 1:4.2; Run 2: 5.4 mg, 26%, (–)-**24**:(–)-**25**, 1:3.7) as a clear film.

Regioisomers (–)-**24** and (–)-**25** were separated for the purpose of full and independent characterization by preparative HPLC [Waters X-Bridge preparative HPLC column, C18, 5 μm, 19 × 250 mm; 20.0 mL/min; 50% acetonitrile in water; t_R((–)-**25**) = 11.5 min, t_R((–)-**24**) = 12.4 min]. Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

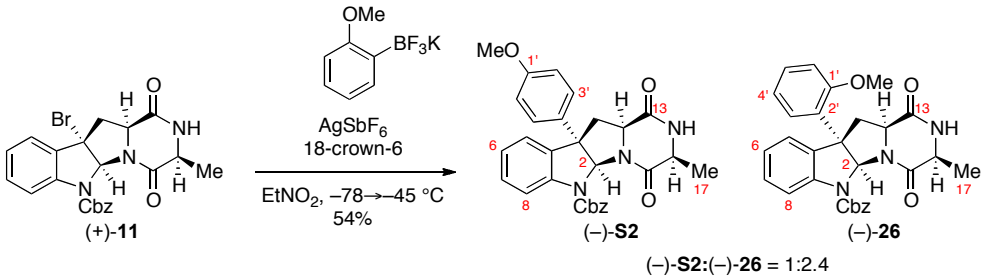
3-(4-Tolyl)tetracycle (–)-24:

Please see page S40 for the full characterization data for 3-(4-tolyl)tetracycle (–)-**24**.

3-(2-Tolyl)tetracycle (–)-25:

^{1}H NMR (500 MHz, $CDCl_3$, 20 ºC): δ 7.66 (d, J = 8.0, 1H, C_8**H**), 7.34–7.22 (m, 5H, Ph_{Cbz}-**H**), 7.34–7.22 (m, 1H, C_5**H**), 7.34–7.22 (C_7**H**), 7.19 (d, J = 7.4, 1H, C_6**H**), 7.16 (app-dt, J = 1.0, 7.4, 1H, $C_{5'}$**H**), 7.10 (app-dt, J = 1.0, 7.5, 1H, C_6**H**), 7.01 (app-dt, J = 1.2, 7.5, 1H, $C_{4'}$**H**), 6.85 (d, J = 7.8, 1H, $C_{3'}$**H**), 6.50 (s, 1H, C_2**H**), 5.62 (s, 1H, N_{14}**H**), 5.33 (d, J = 12.4, 1H, $Ph_{Cbz}C$**H**$_a$), 5.24 (d, J = 12.4, 1H, $Ph_{Cbz}C$**H**$_b$), 4.50 (dd, J = 5.7, 9.6, 1H, C_{11}**H**), 4.02 (q, J = 6.8, 1H, C_{15}**H**), 3.26 (dd, J = 5.8, 14.4, 1H, C_{12}**H**$_a$), 3.04 (dd, J = 9.7, 14.3, 1H, C_{12}**H**$_b$), 2.40 (s, 3H, ArC**H**$_3$), 1.34 (d, J = 6.9, 3H, C_{17}**H**).

^{13}C NMR (125.8 MHz, $CDCl_3$, 20 °C):	δ 169.6 (**C**$_{13}$), 168.0 (**C**$_{16}$), 153.3 (**C**$_{Cbz}$=O), 140.1 (**C**$_9$), 137.9 (**C**$_{2'}$), 136.3 (Ph$_{Cbz}$-*ipso*-**C**), 135.8 (**C**$_{1'}$), 135.5 (**C**$_4$), 133.2 (**C**$_{6'}$), 129.2 (**C**$_7$), 128.7 (Ph$_{Cbz}$-**C**), 128.2 (**C**$_{3'}$), 128.2 (**C**$_{5'}$), 128.2 (Ph$_{Cbz}$-**C**), 128.1 (Ph$_{Cbz}$-**C**), 126.4 (**C**$_{4'}$), 125.9 (**C**$_5$), 124.2 (**C**$_6$), 117.2 (**C**$_8$), 82.6 (**C**$_2$), 68.0 (Ph$_{Cbz}$**C**H$_2$), 59.1 (**C**$_3$), 59.0 (**C**$_{11}$), 52.1 (**C**$_{15}$), 36.0 (**C**$_{12}$), 21.9 (Ar**C**H$_3$), 15.5 (**C**$_{17}$).
FTIR (thin film) cm^{-1}:	1689 (s), 1481 (m), 1405 (m), 1329 (m), 751 (m).
HRMS (ESI) (*m*/*z*):	calc'd for $C_{29}H_{28}N_3O_4$ $[M+H]^+$: 482.2074, found: 482.2066.
$[\alpha]_D^{24}$:	–69 (*c* = 0.13, $CHCl_3$).
TLC (ethyl acetate), R*f*:	0.44 (UV, CAM).



3-(4-Methoxyphenyl)tetracycle (–)-S2 and 3-(2-Methoxyphenyl)tetracycle (–)-26:

A Schlenk tube was charged with tetracyclic bromide (+)-**11** (20.0 mg, 42.5 μmol, 1 equiv), potassium 2-methoxytrifluoroborate (18.2 mg, 85.1 μmol, 2.00 equiv), and 18-crown-6 (22.4 mg, 85.1 μmol, 2.00 equiv). Nitroethane (2 mL) was then introduced via syringe. Upon dissolution of all solid components, the reaction mixture was cooled to –78 ºC. A solution of silver(I) hexafluoroantimonate (0.17 M, 500 μL, 85.1 μmol, 2.00 equiv) in nitroethane at –78 °C was then introduced to the Schlenk tube via cannula. The vessel was subsequently fitted with a PTFE screw cap, sealed, and introduced into a cold bath at –45 °C. After 12 h, aqueous hydrogen chloride (2 N, 4 mL) was added. The reaction mixture was diluted with ethyl acetate (60 mL) and washed with brine (3 × 60 mL). The organic layer was dried over anhydrous sodium sulfate, was filtered, and was concentrated under reduced pressure. The residue was diluted with dichloromethane (30 mL) and washed with brine (30 mL). The resulting aqueous layer was extracted with dichloromethane (2 × 30 mL). The combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography on silica gel (eluent: 90% ethyl acetate in hexanes) to afford a mixture of regioisomeric 3-(methoxyphenyl)tetracycles (Run 1: 11.9 mg, 56.2%, (–)-**S2**:(–)-**26**, 1:2.2; Run 2: 11.1 mg, 52.5%, (–)-**S2**:(–)-**26**, 1:2.7) as a clear film.

Regioisomers (–)-**S2** and (–)-**26** were separated for the purpose of full and independent characterization by preparative HPLC [Waters X-Bridge preparative HPLC column, C18, 5 μm, 19 × 250 mm; 20.0 mL/min; gradient, 40%→60% acetonitrile in water, 20 min; t_R(**S2**) = 12.5 min, t_R(**26**) = 14.6 min]. Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

3-(4-Methoxyphenyl)tetracycle (–)-S2:

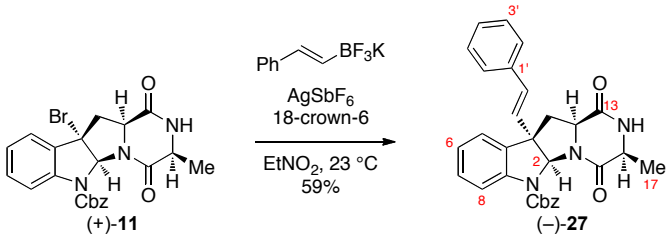
^{1}H NMR (500 MHz, $CDCl_3$, 20 ºC): δ 7.65 (d, J = 7.9, 1H, C_8**H**), 7.37 (dd, J = 1.1, 8.2, 2H, Ph_{Cbz}-*o*-**H**), 7.35–7.26 (m, 3H, Ph_{Cbz}-**H**), 7.35–7.26 (m, 1H, C_5**H**), 7.20 (app-dt, J = 1.2, 8.1, 1H, C_7**H**), 7.07–7.02 (m, 1H, C_6**H**), 7.07–7.02 (m, 2H, $C_{3'}$**H**), 6.76 (d, J = 8.9, 2H, $C_{2'}$**H**), 6.31 (s, 1H, C_2**H**), 5.42 (s, 1H, N_{14}**H**), 5.41 (d, J = 12.2, 1H, $Ph_{Cbz}C$**H**$_a$), 5.28 (d, J = 12.3, 1H, Ph_{Cbz}**H**$_b$), 4.38 (dd, J = 3.4, 10.1, 1H, C_{11}**H**), 4.00 (q, J = 6.8, 1H, C_{15}**H**), 3.73 (s, 3H, O**CH**$_3$), 3.50 (dd, J = 3.4, 13.9, 1H, C_{12}**H**$_a$), 2.84 (dd, J = 10.2, 13.9, 1H, C_{12}**H**$_b$), 1.33 (d, J = 6.8, 3H, C_{17}**H**).

^{13}C NMR (125.8 MHz, $CDCl_3$, 20 ºC): δ 169.3 (**C**$_{13}$), 167.7 (**C**$_{16}$), 159.0 (**C**$_{1'}$), 153.2 (**C**$_{Cbz}$=O), 139.8 (**C**$_9$), 136.4 (Ph_{Cbz}-*ipso*-**C**), 135.1 (**C**$_4$), 133.7 (**C**$_{4'}$), 129.1 (**C**$_7$), 128.7 (Ph_{Cbz}-**C**), 128.7 (Ph_{Cbz}-**C**), 128.3 (Ph_{Cbz}-**C**), 127.1 (**C**$_{3'}$), 125.2

	(**C**$_5$), 124.4 (**C**$_6$), 117.1 (**C**$_8$), 114.5 (**C**$_{2'}$), 83.5 (**C**$_2$), 68.1 (Ph$_{Cbz}$**C**H$_2$), 58.7 (**C**$_{11}$), 58.1 (**C**$_3$), 55.5 (O**C**H$_3$), 51.9 (**C**$_{15}$), 35.0 (**C**$_{12}$), 15.4 (**C**$_{17}$).
FTIR (thin film) cm^{-1}:	1709 (s), 1688 (s), 1409 (m), 1312 (m), 1254 (m).
HRMS (ESI) (*m*/*z*):	calc'd for $C_{29}H_{28}N_3O_5$ [M+H]$^+$: 498.2023, found: 498.2032.
$[\alpha]_D^{24}$:	–13 (*c* = 0.070, $CHCl_3$).
TLC (ethyl acetate), R*f*:	0.40 (UV, CAM).

3-(2-Methoxyphenyl)tetracycle (–)-26:

^{1}H NMR (500 MHz, $CDCl_3$, 20 ºC):	δ 7.61 (d, *J* = 7.5, 1H, C$_8$**H**), 7.36 (d, *J* = 7.1, 1H, C$_5$**H**), 7.32–7.20 (m, 1H, C$_7$**H**), 7.32–7.20 (m, 5H, Ph$_{Cbz}$-**H**), 7.32–7.20 (m, 1H, C$_{5'}$**H**), 7.13 (app-t, *J* = 7.5, 1H, C$_6$**H**), 6.92 (d, *J* = 8.0, 1H, C$_{6'}$**H**), 6.75 (app-t, *J* = 7.4, 1H, C$_{4'}$**H**), 6.66 (dd, *J* = 1.6, 7.8, 1H, C$_{3'}$**H**), 6.45 (s, 1H, C$_2$**H**), 5.90–5.75 (m, 1H, N$_{14}$**H**), 5.36 (d, *J* = 12.5, 1H, Ph$_{Cbz}$C**H**$_a$), 5.19 (d, *J* = 12.5, 1H, Ph$_{Cbz}$C**H**$_b$), 4.52 (dd, *J* = 5.8, 9.9, 1H, C$_{11}$**H**), 4.05 (q, *J* = 6.8, 1H, C$_{15}$**H**), 3.89 (s, 3H, OC**H**$_3$), 3.12 (dd, *J* = 5.8, 13.5, 1H, C$_{12}$**H**$_a$), 3.01 (dd, *J* = 10.1, 13.5, 1H, C$_{12}$**H**$_b$), 1.36 (d, *J* = 6.9, 3H, C$_{17}$**H**).
^{13}C NMR (125.8 MHz, $CDCl_3$, 20 ºC):	δ 170.1 (**C**$_{13}$), 167.6 (**C**$_{16}$), 156.8 (**C**$_{1'}$), 153.4 (**C**$_{Cbz}$=O), 140.5 (**C**$_9$), 136.4 (Ph$_{Cbz}$-*ipso*-**C**), 135.4 (**C**$_4$), 129.4 (**C**$_{5'}$), 129.2 (**C**$_{3'}$), 129.1 (**C**$_7$), 128.8 (**C**$_{2'}$), 128.6 (Ph$_{Cbz}$-**C**), 128.1 (Ph$_{Cbz}$-**C**), 127.9 (Ph$_{Cbz}$-**C**), 126.8 (**C**$_5$), 124.2 (**C**$_6$), 120.8 (**C**$_{4'}$), 117.5 (**C**$_8$), 111.5 (**C**$_{6'}$), 82.1 (**C**$_2$), 67.9 (Ph$_{Cbz}$**C**H$_2$), 58.7 (**C**$_{11}$), 56.8 (**C**$_3$), 55.6 (O**C**H$_3$), 52.0 (**C**$_{15}$), 36.3 (**C**$_{12}$), 15.9 (**C**$_{17}$).
FTIR (thin film) cm^{-1}:	1712 (s), 1688 (s), 1482 (m), 1409 (m), 1326 (m).
HRMS (ESI) (*m*/*z*):	calc'd for $C_{29}H_{28}N_3O_5$ [M+H]$^+$: 498.2023, found: 498.2033.
$[\alpha]_D^{24}$:	–152 (*c* = 0.15, $CHCl_3$).
TLC (ethyl acetate), R*f*:	0.33 (UV, CAM).



3-(*trans*-Styryl)tetracycle (–)-27:

A solution of silver(I) hexafluoroantimonate (0.20 M, 425 μL, 85.1 μmol, 2.00 equiv) in nitroethane was added via syringe to a solution of tetracyclic bromide (+)-**11** (20.0 mg, 42.5 μmol, 1 equiv), potassium *trans*-styryltrifluoroborate (17.9 mg, 85.1 μmol, 2.00 equiv), and 18-crown-6 (22.4 mg, 85.1 μmol, 2.00 equiv) in nitroethane (2 mL) at 23 ºC. After 1 h, aqueous hydrogen chloride (2 N, 4 mL) was added. The reaction mixture was diluted with ethyl acetate (60 mL) and washed with brine (3 × 60 mL). The organic layer was dried over anhydrous sodium sulfate, was filtered, and was concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography on silica gel (eluent: 20% acetone in dichloromethane). The fractions containing the desired product were combined and were concentrated under reduced pressure. The resulting residue was loaded onto a silica gel column and purified by flash column chromatography (eluent: 90% ethyl acetate in hexanes) to afford 3-(*trans*-styryl)tetracycle (–)-**27** (Run 1: 12.6 mg, 60.0%; Run 2: 12.0 mg, 57.2%) as a white foam. Structural assignments were made using additional information from gCOSY, HSQC, and HMBC experiments.

^{1}H NMR (500 MHz, $CDCl_3$, 20 ºC):	δ 7.68 (d, *J* = 8.0, 1H, C_8**H**), 7.41 (d, *J* = 7.1, 2H, Ph_{Cbz}-*o*-**H**), 7.33–7.18 (m, 1H, C_7**H**), 7.33–7.18 (m, 1H, C_5**H**), 7.33–7.18 (m, 8H, Ar**H**), 7.08 (app-t, *J* = 7.5, 1H, C_6**H**), 6.25 (d, *J* = 16.0, 1H, C**H**=CHPh), 6.09 (d, *J* = 14.4, 1H, CH=C**H**Ph), 6.07 (s, 1H, C_2**H**), 5.77 (br-s, 1H, N_{14}**H**), 5.39 (d, *J* = 12.3, 1H, Ph_{Cbz}C**H**$_a$), 5.29 (d, *J* = 12.3, 1H, Ph_{Cbz}C**H**$_b$), 4.31 (dd, *J* = 3.1, 10.0, 1H, C_{11}**H**), 3.97 (q, *J* = 6.8, 1H, C_{15}**H**), 3.28 (dd, *J* = 3.3, 13.9, 1H, C_{12}**H**$_a$), 2.63 (dd, *J* = 10.2, 13.8, 1H, C_{12}**H**$_b$), 1.32 (d, *J* = 6.8, 3H, C_{17}**H**).
^{13}C NMR (125.8 MHz, $CDCl_3$, 20 ºC):	δ 169.5 (**C**$_{13}$), 167.7 (**C**$_{16}$), 153.4 (**C**$_{Cbz}$=O), 140.4 (**C**$_9$), 136.3 (Ph_{Cbz}-*ipso*-**C**), 136.1 ($Ph_{styrene}$-*ipso*-**C**), 133.1 (**C**$_4$), 131.7 (CH=**C**HPh), 129.4 (**C**H=CHPh), 129.3 (Ph_{Cbz}-**C**), 128.8 (Ph_{Cbz}-**C**), 128.7 (Ph_{Cbz}-**C**), 128.4 (Ph_{Cbz}-**C**), 128.3 (Ph_{Cbz}-**C**), 128.2 (Ph_{Cbz}-**C**), 126.8 (**C**$_7$), 125.2 (**C**$_5$), 124.3 (**C**$_6$), 116.9 (**C**$_8$), 81.9 (**C**$_2$), 68.2 (Ph_{Cbz}**C**H$_2$), 58.3 (**C**$_{11}$), 57.2 (**C**$_3$), 51.9 (**C**$_{15}$), 33.3 (**C**$_{12}$), 15.3 (**C**$_{17}$).
FTIR (thin film) cm^{-1}:	3262 (br, m), 1712 (s), 1690 (s), 1409 (m), 1315 (m).
HRMS (ESI) (*m*/*z*):	calc'd for $C_{30}H_{28}N_3O_4$ $[M+H]^+$: 494.2074, found: 494.2084.
$[\alpha]_D^{24}$:	–23 (*c* = 0.081, $CHCl_3$).
TLC (ethyl acetate), R*f*:	0.44 (UV, CAM).

Table S1. Comparison of our data for (+)-Naseseazine A (1) with literature:

Assignment[11]	**Raju et al. Report**[4,12] (+)-Naseseazine A (**1**) ^{1}H NMR, 600 MHz, methanol-d_4	**This Work** (+)-Naseseazine A (**1**) ^{1}H NMR, 500 MHz, methanol-d_4, 20 ºC
N1	-	-
C2	5.83 (s)	5.83 (s)
C3	-	-
C4	-	-
C5	6.85 (d, J = 7.4)	6.85 (dd, J = 0.6, 7.4)
C6	6.67 (t, J = 7.5)	6.68 (app-dt, J = 0.9, 7.4)
C7	7.05 (t, J = 7.2)	7.05 (app-dt, J = 1.3, 7.9)
C8	6.69 (d, J = 7.6)	6.69 (d, J = 8.0)
C9	-	-
N10	-	-
C11	4.64 (dd, J = 7.4, 8.4)	4.65 (dd, J = 7.4, 9.2)
C12	3.26 (m) 2.59 (dd, J = 10.2, 13.7)	3.30−3.20 (m) 2.59 (dd, J = 9.9, 13.6)
C13	-	-
N14	-	-
C15	4.15 (q, J = 6.9)	4.16 (dq, J = 1.4, 6.9)
C16	-	-
C17	1.38 (d, J = 6.9)	1.38 (d, J = 7.1)
N1'	-	-
C2'	7.11 (s)	7.12 (s)
C3'	-	-
C4'	-	-
C5'	7.57 (d, J = 8.4)	7.57 (d, J = 8.4)
C6'	7.02 (d, J = 8.4)	7.03 (dd, J = 1.7, 8.4)
C7'	-	-
C8'	7.40 (s)	7.40 (d, J = 1.3)
C9'	-	-
N10'	-	-
C11'	4.39 (br-t, J = 4.5)	4.39 (app-dt, J = 1.1, 4.7)
C12'	3.30 (m) 3.28 (m)	3.30−3.20 (m)
C13'	-	-
N14'	-	-
C15'	3.97 (dd, J = 6.6, 10.8)	3.98 (ddd, J = 1.7, 6.3, 10.9)
C16'	-	-
C17'	1.97 (m) 0.93 (m)	1.96 (app-ddt, J = 2.0, 7.2, 12.5) 0.96−0.85 (m)
C18'	1.66 (m) 1.43 (m)	1.72−1.61 (m) 1.47−1.39 (m)
C19'	3.42 (dt, J = 8.1, 11.8) 3.24 (m)	3.42 (app-dt, J = 8.3, 11.7) 3.30−3.20 (m)

[11] Please see page S3 for the positional numbering system used in this report.
[12] The reference points for the residual protium and carbon resonances of the NMR solvent were not listed.

Table S2. Comparison of our data for (+)-Naseseazine A (1) with literature:

Assignment[11]	**Raju et al. Report**[4,12] (+)-Naseseazine A (**1**) ^{1}H NMR, 600 MHz, DMSO-d_6	**This Work** (+)-Naseseazine A (**1**) ^{1}H NMR, 500 MHz, DMSO-d_6, 20 ºC
N1	6.72 (d, J = 2.8)	6.75 (d, J = 2.8)
C2	5.65 (d, J = 2.8)	5.65 (d, J = 2.8)
C3	-	-
C4	-	-
C5	6.82 (d, J = 7.3)	6.83 (d, J = 7.3)
C6	6.57 (dt, J = 0.7, 7.5)	6.58 (app-t, J = 7.4)
C7	7.00 (t, J = 7.2)	7.04–6.95 (m)
C8	6.61 (d, J = 7.8)	6.62 (d, J = 8.0)
C9	-	-
N10	-	-
C11	4.60 (dd, J = 8.4, 9.9)	4.62 (app-t, J = 8.7)
C12	3.08 (dd, J = 7.6, 13.6) 2.41 (dd, J = 9.9, 13.6)	3.14–3.00 (m) 2.41 (dd, J = 10.0, 13.6)
C13	-	-
N14	8.17 (s)	8.20 (s)
C15	4.13 (q, J = 6.9)	4.14 (q, J = 6.5)
C16	-	-
C17	1.21 (d, J = 6.9)	1.23 (d, J = 6.9)
N1'	10.79 (d, J = 1.9)	10.81 (s)
C2'	7.17 (d, J = 2.2)	7.19 (d, J = 1.8)
C3'	-	-
C4'	-	-
C5'	7.55 (d, J = 8.3)	7.56 (d, J = 8.4)
C6'	6.98 (d, J = 8.2)	7.04–6.95 (m)
C7'	-	-
C8'	7.27 (d, J = 1.2)	7.28 (s)
C9'	-	-
N10'	7.68 (s)	7.71 (s)
C11'	4.27 (br-t, J = 5.2)	4.28 (app-t, J = 4.9)
C12'	3.21 (dd, J = 4.8, 15.0) 3.03 (dd, J = 5.8, 14.9)	3.30–3.18 (m) 3.14–3.00 (m)
C13'	-	-
N14'	-	-
C15'	4.06 (dd, J = 7.5, 10.0)	4.06 (app-t, J = 7.8)
C16'	-	-
C17'	1.96 (m) 1.40 (m)	2.03–1.94 (m) 1.47–1.36 (m)
C18'	1.67 (m) 1.61 (m)	1.74–1.56 (m)
C19'	3.34 (m) 3.25 (m)	3.41–3.29 (m) 3.30–3.18 (m)

Table S3. Comparison of our data for (+)-Naseseazine A (1) with literature:

Assignment[11]	**Raju et al. Report**[4,12] (+)-Naseseazine A (**1**) ^{13}C NMR, 151 MHz methanol-d_4	**This Work** (+)-Naseseazine A (**1**) ^{13}C NMR, 126 MHz methanol-d_4, 20 ºC	**Chemical Shift Difference** Δδ δ (this work) – δ (Raju et. al. report)
C2	87.1	87.3	0.2
C3	61.2	61.4	0.2
C4	135.8	136.1	0.3
C5	124.9	125.1	0.2
C6	120.2	120.6	0.4
C7	129.2	129.6	0.4
C8	110.9	111.2	0.3
C9	149.1	149.3	0.2
C11	60.2	60.5	0.3
C12	39.7	39.9	0.2
C13	172.6	172.7	0.1
C15	52.1	52.4	0.3
C16	170.6	170.9	0.3
C17	15.2	15.4	0.2
C2'	126.2	126.6	0.4
C3'	109.5	109.7	0.2
C4'	127.6	127.8	0.2
C5'	120.3	120.5	0.2
C6'	119.5	119.7	0.2
C7'	137.2	137.3	0.1
C8'	110.1	110.4	0.3
C9'	137.9	138.1	0.2
C11'	57.2	57.4	0.2
C12'	29.0	29.3	0.3
C13'	167.3	167.5	0.2
C15'	60.0	60.2	0.2
C16'	170.6	170.9	0.3
C17'	29.0	29.3	0.3
C18'	22.5	22.7	0.2
C19'	45.8	46.1	0.3

Table S4. Comparison of our data for (+)-Naseseazine B (2) with literature:

Assignment[11]	**Raju et al. Report**[4,12] (+)-Naseseazine B (**2**) ^{1}H NMR, 600 MHz, methanol-d_4	**This Work** (+)-Naseseazine B (**2**) ^{1}H NMR, 500 MHz, methanol-d_4, 20 ºC
N1	-	-
C2	5.85 (s)	5.84 (s)
C3	-	-
C4	-	-
C5	6.84 (dt, J = 0.9, 7.2)	6.83 (d, J = 7.2)
C6	6.68 (t, J = 7.6)	6.68 (app-t, J = 7.3)
C7	7.06 (dt, J = 1.3, 7.6)	7.06 (app-dt, J = 1.2, 7.9)
C8	6.69 (d, J = 7.6)	6.69 (d, J = 7.7)
C9	-	-
N10	-	-
C11	4.75 (dd, J = 8.7, 10.2)	4.71 (app-t, J = 8.6)
C12	3.27 (m) 2.59 (dd, J = 10.2, 13.8)	3.35−3.21 (m) 2.57 (dd, J = 10.2, 13.7)
C13	-	-
N14	-	-
C15	4.33 (dd, J = 7.1, 9.5)	4.29 (app-t, J = 7.7)
C16	-	-
C17	2.28 (m) 2.11 (m)	2.30−2.22 (m) 2.15−2.04 (m)
C18	2.00 (m) 1.95 (m)	2.04−1.87 (m) 2.04−1.87 (m)
C19	3.49 (m) 3.44 (m)	3.52−3.37 (m) 3.52−3.37 (m)
N1'	-	-
C2'	7.12 (s)	7.12 (s)
C3'	-	-
C4'	-	-
C5'	7.58 (d, J = 8.4)	7.57 (d, J = 7.4)
C6'	7.03 (dd, J = 1.8, 8.4)	7.01 (dd, J = 1.6, 8.5)
C7'	-	-
C8'	7.41 (d, j = 1.4)	7.41 (d, J =1.3)
C9'	-	-
N10'	-	-
C11'	4.40 (br-t, J = 4.7)	4.39 (app-t, J = 4.6)
C12'	3.32 (m) 3.28 (m)	3.35−3.21 (m) 3.35−3.21 (m)
C13'	-	-
N14'	-	-
C15'	3.99 (ddd, J = 1.6, 6.6, 11.4)	3.98 (ddd, J = 1.4, 6.3, 10.7)
C16'	-	-
C17'	1.97 (m) 0.92 (m)	2.04−1.87 (m) 0.97−0.87 (m)
C18'	1.67 (m) 1.44 (m)	1.73−1.60 (m) 1.49−1.40 (m)
C19'	3.43 (m) 3.24 (m)	3.52−3.37 (m) 3.35−3.21 (m)

Table S5. Comparison of our data for (+)-Naseseazine B (2) with literature:

Assignment[11]	**Raju et al. Report**[4,12] (+)-Naseseazine B (**2**) ^{1}H NMR, 600 MHz, DMSO-d_6	**This Work** (+)-Naseseazine B (**2**) ^{1}H NMR, 500 MHz, DMSO-d_6, 20 ºC
N1	6.75 (d, J = 3.2)	6.82–6.76 (m)
C2	5.65 (d, J = 3.2)	5.68 (d, J = 3.2)
C3	-	-
C4	-	-
C5	6.82 (d, J = 7.3)	6.82–6.76 (m)
C6	6.58 (dt, J = 0.9, 7.4)	6.58 (app-dt, J = 1.0, 7.5)
C7	7.00 (t, J = 7.2)	7.00 (app-dt, J = 1.3, 7.9)
C8	6.61 (d, J = 7.8)	6.60 (d, J = 7.6)
C9	-	-
N10	-	-
C11	4.71 (dd, J = 7.5, 10.5)	4.72 (app-t, J = 8.2)
C12	3.12 (dd, J = 7.5, 13.6) 2.36 (dd, J = 10.5, 13.6)	3.13 (dd, J = 7.4, 13.6) 2.36 (dd, J = 10.5, 13.6)
C13	-	-
N14	-	-
C15	4.34 (t, J = 8.0)	4.35 (app-t, J = 7.9)
C16	-	-
C17	2.15 (m) 1.95 (m)	2.21–2.11 (m) 2.03–1.91 (m)
C18	1.85 (m)	1.91–1.77 (m)
C19	3.34 (m)	3.41–3.30 (m)
N1'	10.80 (d, J = 1.8)	10.81 (s)
C2'	7.18 (d, J = 2.2)	7.19 (d, J = 2.2)
C3'	-	-
C4'	-	-
C5'	7.56 (d, J = 8.4)	7.57 (d, J = 8.4)
C6'	6.98 (dd, J = 1.5, 8.4)	6.99 (d, J = 8.6)
C7'	-	-
C8'	7.29 (d, J = 1.5)	7.30 (d, J = 1.3)
C9'	-	-
N10'	7.67 (s)	7.71 (s)
C11'	4.27 (br-t, J = 5.1)	4.29 (app-t, J = 5.1)
C12'	3.20 (dd, J = 4.5, 15.0) 3.04 (dd, J = 6.0, 15.0)	3.22 (dd, J = 4.6, 14.6) 3.05 (dd, J = 5.8, 14.8)
C13'	-	-
N14'	-	-
C15'	4.06 (dd, J = 7.6, 9.0)	4.06 (app-t, J = 8.7)
C16'	-	-
C17'	1.97 (m) 1.41 (m)	2.03–1.91 (m) 1.48–1.36 (m)
C18'	1.68 (m) 1.62 (m)	1.75–1.56 (m)
C19'	3.34 (m) 3.24 (m)	3.41–3.30 (m) 3.29–3.24 (m)

Table S6. Comparison of our data for (+)-Naseseazine B (2) with literature:

Assignment[11]	**Raju et al. Report**[4,12] (+)-Naseseazine B (**2**) ^{13}C NMR, 151 MHz methanol-d_4	**This Work** (+)-Naseseazine B (**2**) ^{13}C NMR, 126 MHz methanol-d_4, 20 ºC	**Chemical Shift Difference** Δδ δ (this work) – δ (Raju et. al. report)
C2	86.8	87.1	0.3
C3	61.7	61.9	0.2
C4	136.0	136.2	0.2
C5	124.8	125.1	0.3
C6	120.3	120.6	0.3
C7	129.1	129.6	0.5
C8	111.0	111.3	0.3
C9	149.0	149.3	0.3
C11	61.3	61.7	0.4
C12	39.5	39.7	0.2
C13	168.4	168.6	0.2
C15	61.8	61.9	0.1
C16	170.2	170.3	0.1
C17	28.3	28.6	0.3
C18	24.1	24.4	0.3
C19	45.9	46.4	0.5
C2'	126.1	126.6	0.5
C3'	109.5	109.8	0.3
C4'	127.6	127.8	0.2
C5'	120.3	120.6	0.3
C6'	119.4	119.8	0.4
C7'	136.9	137.2	0.3
C8'	110.3	110.5	0.2
C9'	137.9	138.1	0.2
C11'	57.0	57.3	0.3
C12'	29.2	29.3	0.1
C13'	167.3	167.5	0.2
C15'	59.9	60.2	0.3
C16'	170.7	170.9	0.2
C17'	29.1	29.3	0.2
C18'	22.4	22.7	0.3
C19'	45.8	46.1	0.3

Marfey's Analysis of (+)-Naseseazine B (2).

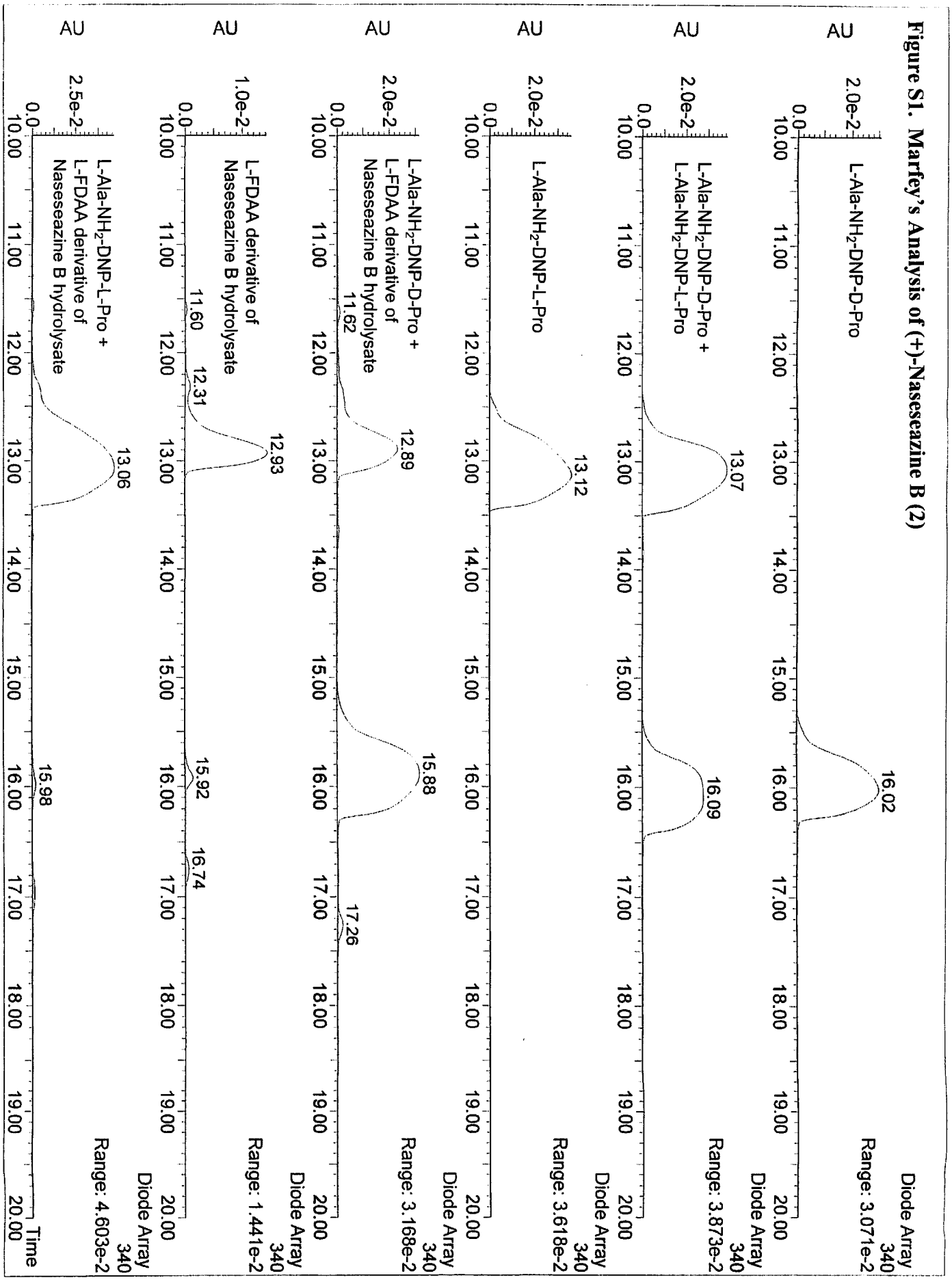
Marfey's analysis was performed on synthetic (+)-naseseazine B (**2**) using the method described in the isolation report.[4] (+)-Naseseazine B (**2**) (350 μg, 620 nmol, 1 equiv) was dissolved in aqueous hydrochloric acid (6 N, 1.40 mL) and heated to 100 °C in a sealed vial. After 12 h, the reaction mixture was allowed to cool to 23 °C then concentrated under reduced pressure. A solution of 1-fluoro-2,4-dinitrophenyl-5-L-alanine amide (L-FDAA) in acetone (1% w/v, 700 μL) and aqueous sodium bicarbonate (1 M, 140 μL) were sequentially added to the residue and the resulting solution was heated to 37 °C. After 1 h, the reaction mixture was allowed to cool to 23 °C and aqueous hydrochloric acid (1 M, 140 μL) was added. The solution was then diluted with acetonitrile (5.67 mL) and passed through a syringe filter (Acrodisc GHP, 13 mm diameter, 0.45 μm pore size). An aliquot of this solution (10 μL) was diluted in water (450 μL) and analyzed by HPLC.

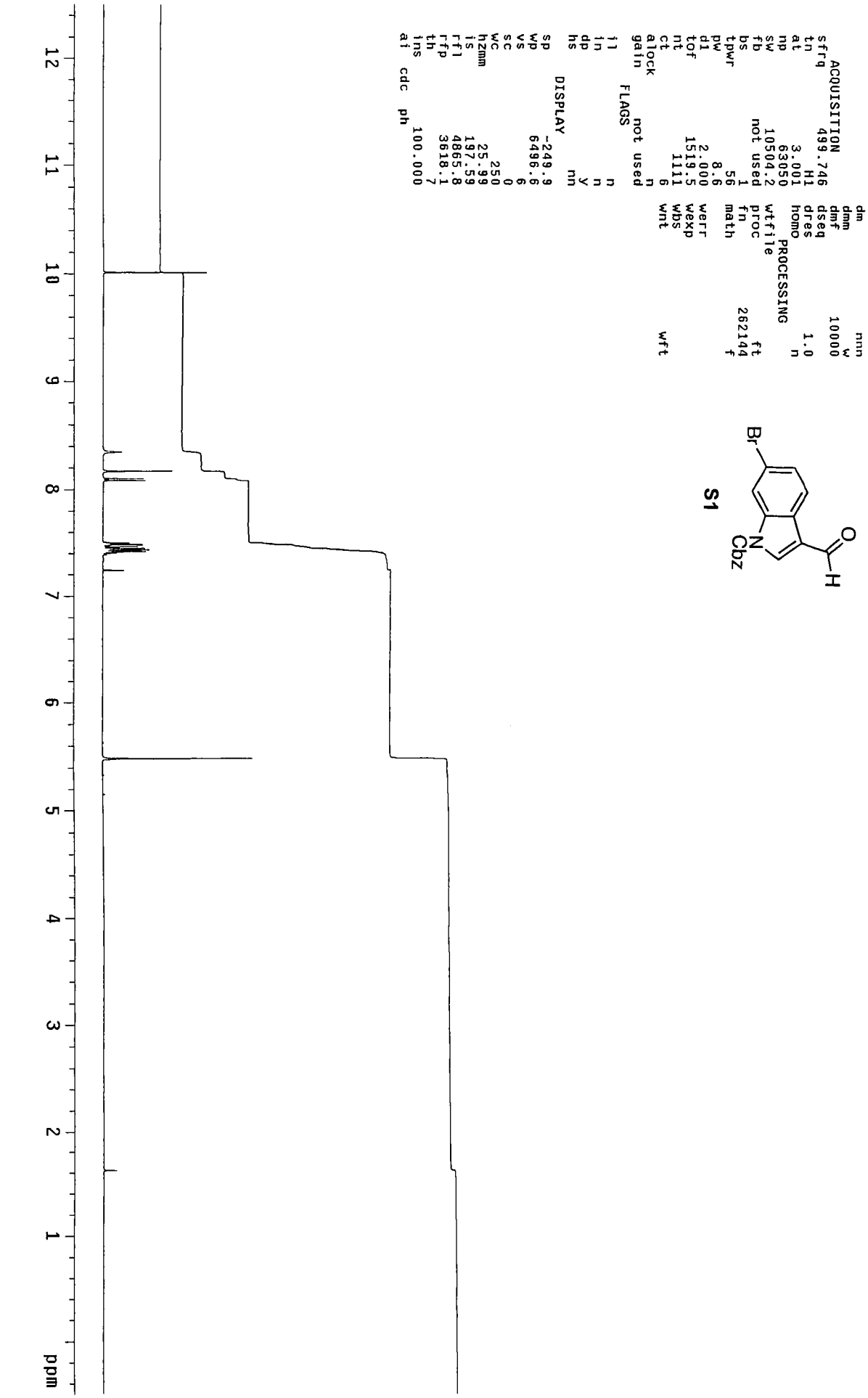
A standard solution of L-Ala-NH_2-DNP-L-Pro was prepared from L-proline. A solution of L-FDAA in acetone (1% w/v, 100 μL) and aqueous sodium bicarbonate (1 M, 20 μL) were sequentially added to an aqueous solution of L-proline (50 mM, 50 μL). The solution was then heated to 37 °C. After 1 h, the reaction mixture was allowed to cool to 23 °C and aqueous hydrochloric acid (1 M, 20 μL) was added. The solution was then diluted with isopropanol (810 μL) and passed through a syringe filter (Acrodisc GHP, 13 mm diameter, 0.45 μm pore size). An aliquot of this solution (1 μL) was diluted in water (450 μL) and analyzed by HPLC.

A standard solution of L-Ala-NH_2-DNP-D-Pro was prepared analogously from D-proline.

HPLC conditions: Waters X-Bridge analytical HPLC column, C18, 5 μm, 4.6 × 250 mm; 2.00 mL/min; gradient, 0%→30% acetonitrile in water, 30 min; t_R(L-Ala-NH_2-DNP-L-Pro) = 13.1 min, t_R(L-Ala-NH_2-DNP-D-Pro) = 16.0 min. HPLC traces were obtained using UV detection (340 nm) and product identity was further corroborated with HPLC traces of mass spectrometric data (ESI+). See Figure S1 for the HPLC traces of L-FDAA derived (+)-naseseazine B hydrolysate, the corresponding amino acid standards, and their co-injection. The HPLC analysis confirms the presence of L-proline residues in the synthetic samples of (+)-naseseazine B (**2**) and is consistent with our use of L-proline building blocks en route to the natural product. Marfey's analysis thus lends further support to our stereochemical reassignment of (+)-naseseazine B (**2**).

Figure S1. Marfey's Analysis of (+)-Naseseazine B (2)

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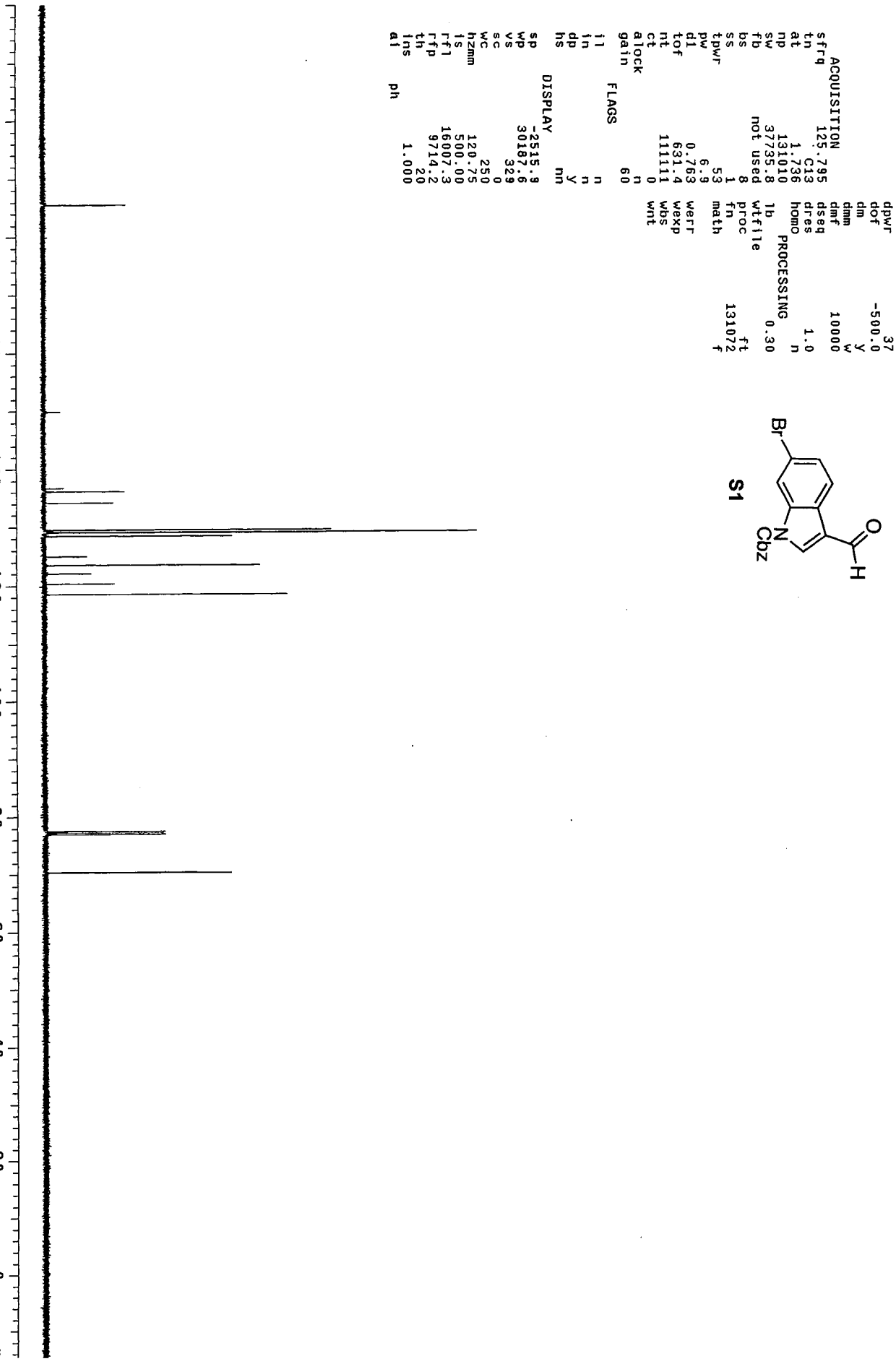
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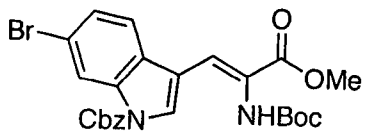

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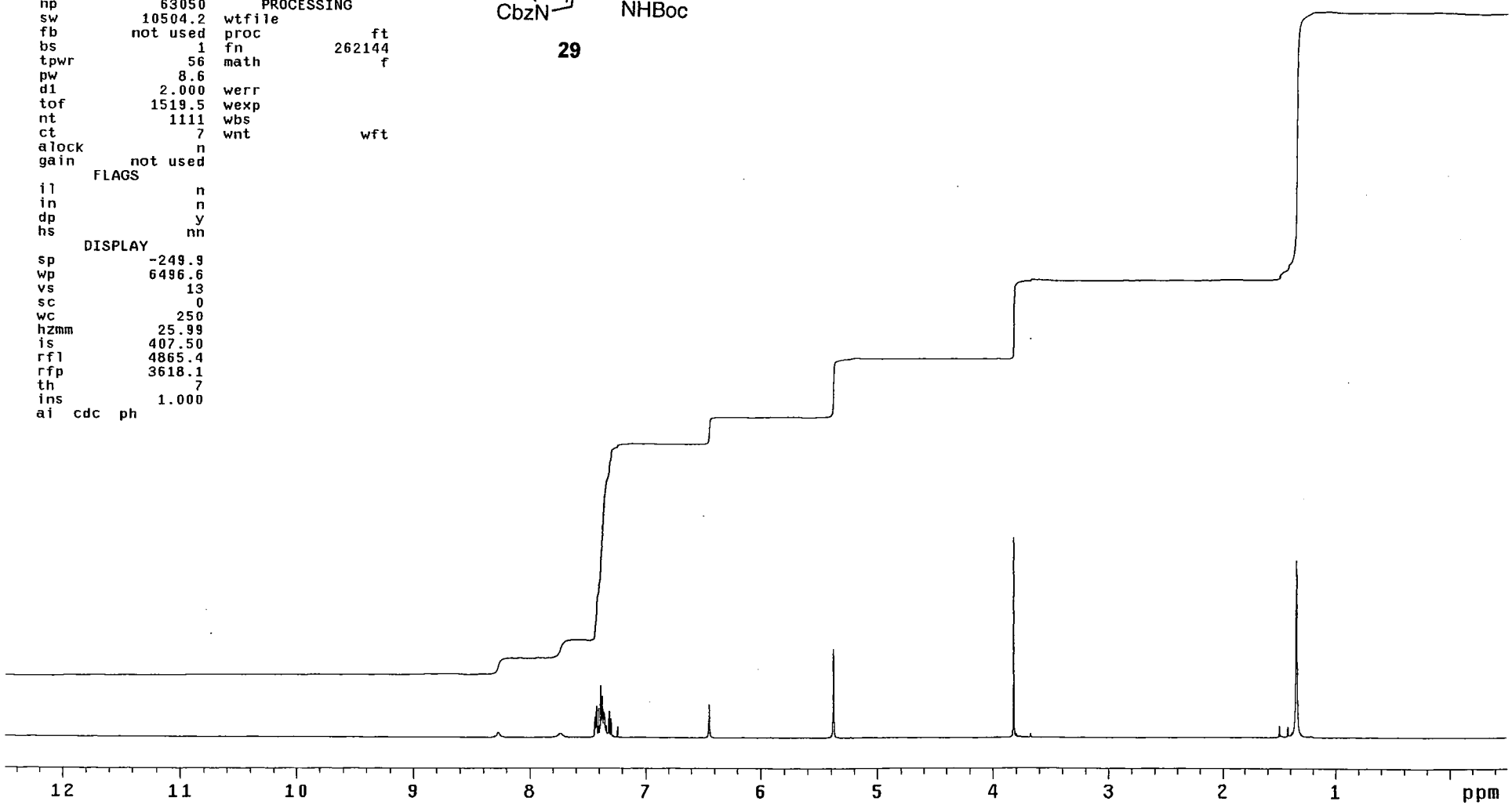

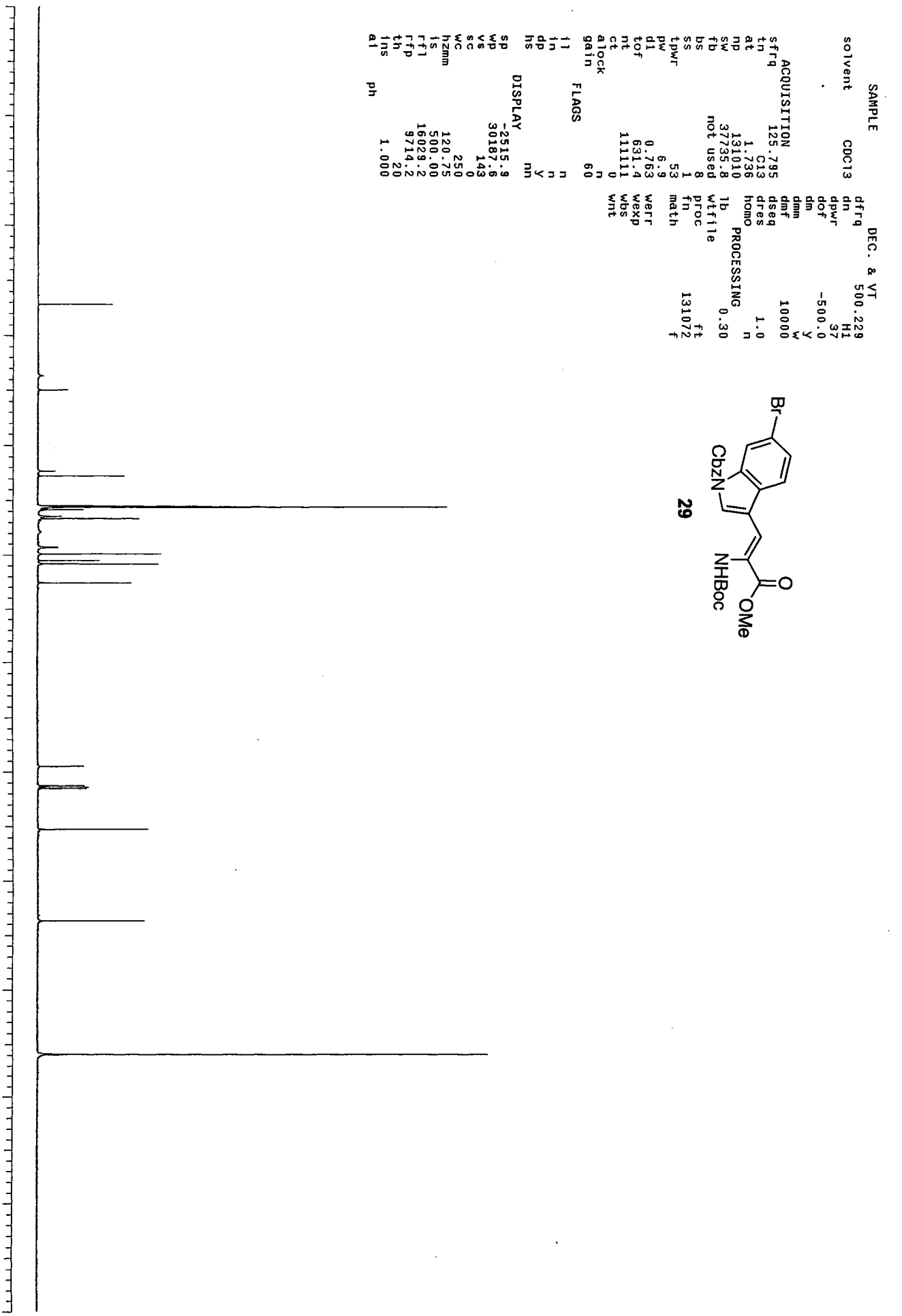
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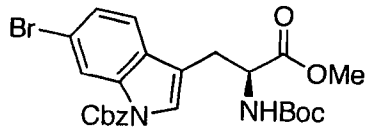
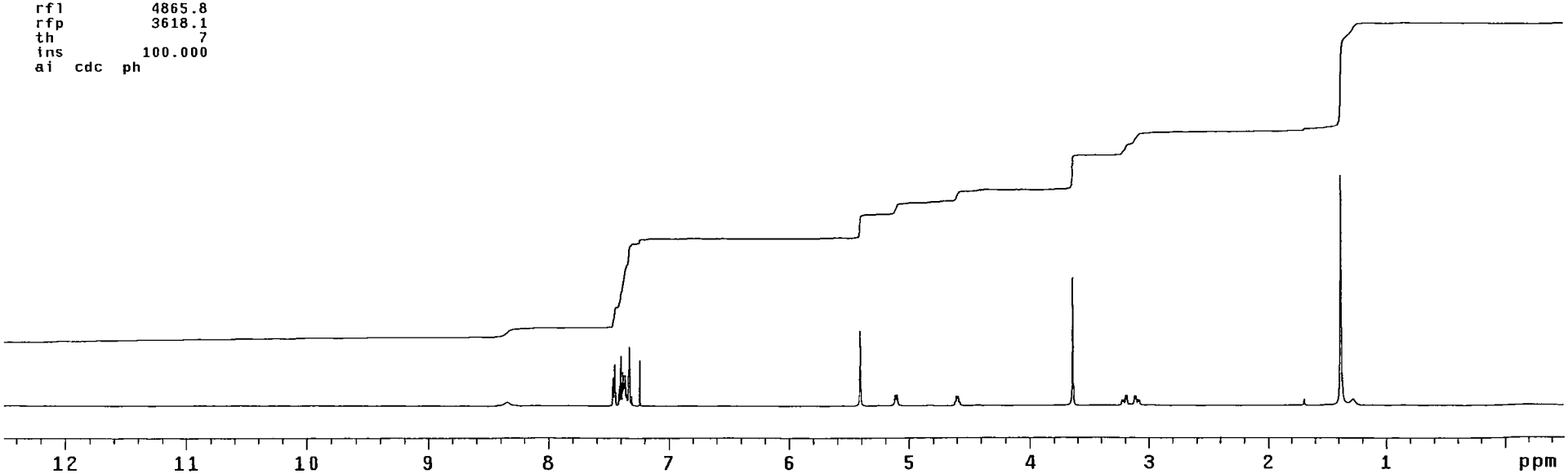
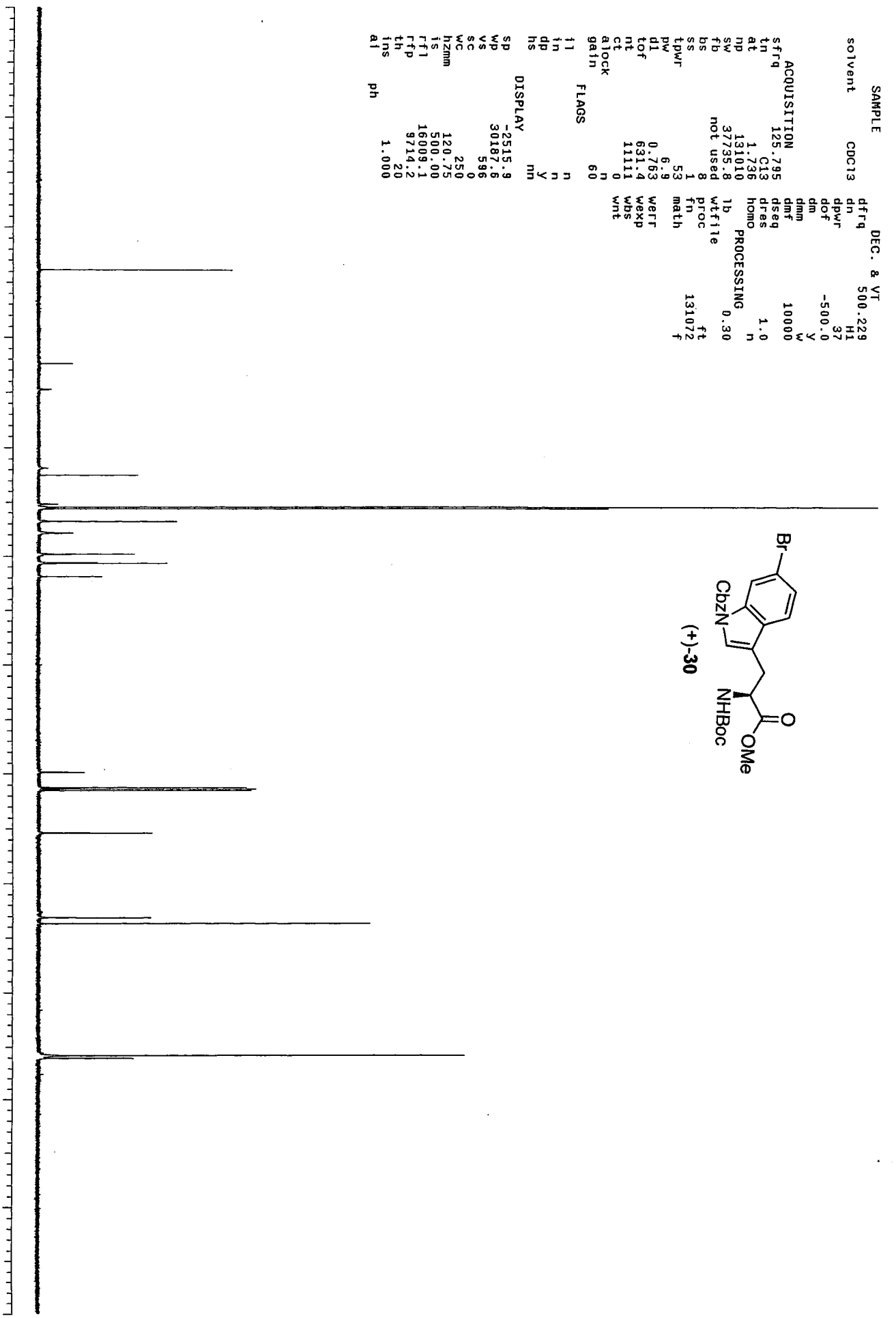
ppm

SAMPLE		DEC. & VT	
solvent	CDCl3	dfrq	125.672
		dn	C13
		dpwr	30
		dof	0
		dm	nnn
		dmm	w
ACQUISITION		dmf	10000
sfrq	499.746	dseq	
tn	H1	dres	1.0
at	3.001	homo	n
np	63050	PROCESSING	
sw	10504.2	wtfile	
fb	not used	proc	ft
bs	1	fn	262144
tpwr	56	math	f
pw	8.6		
d1	2.000	werr	
tof	1519.5	wexp	
nt	11111	wbs	
ct	8	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-249.9		
wp	6496.6		
vs	22		
sc	0		
wc	250		
hzmm	25.99		
is	257.77		
rfl	4865.8		
rfp	3618.1		
th	7		
ins	100.000		
ai cdc ph			

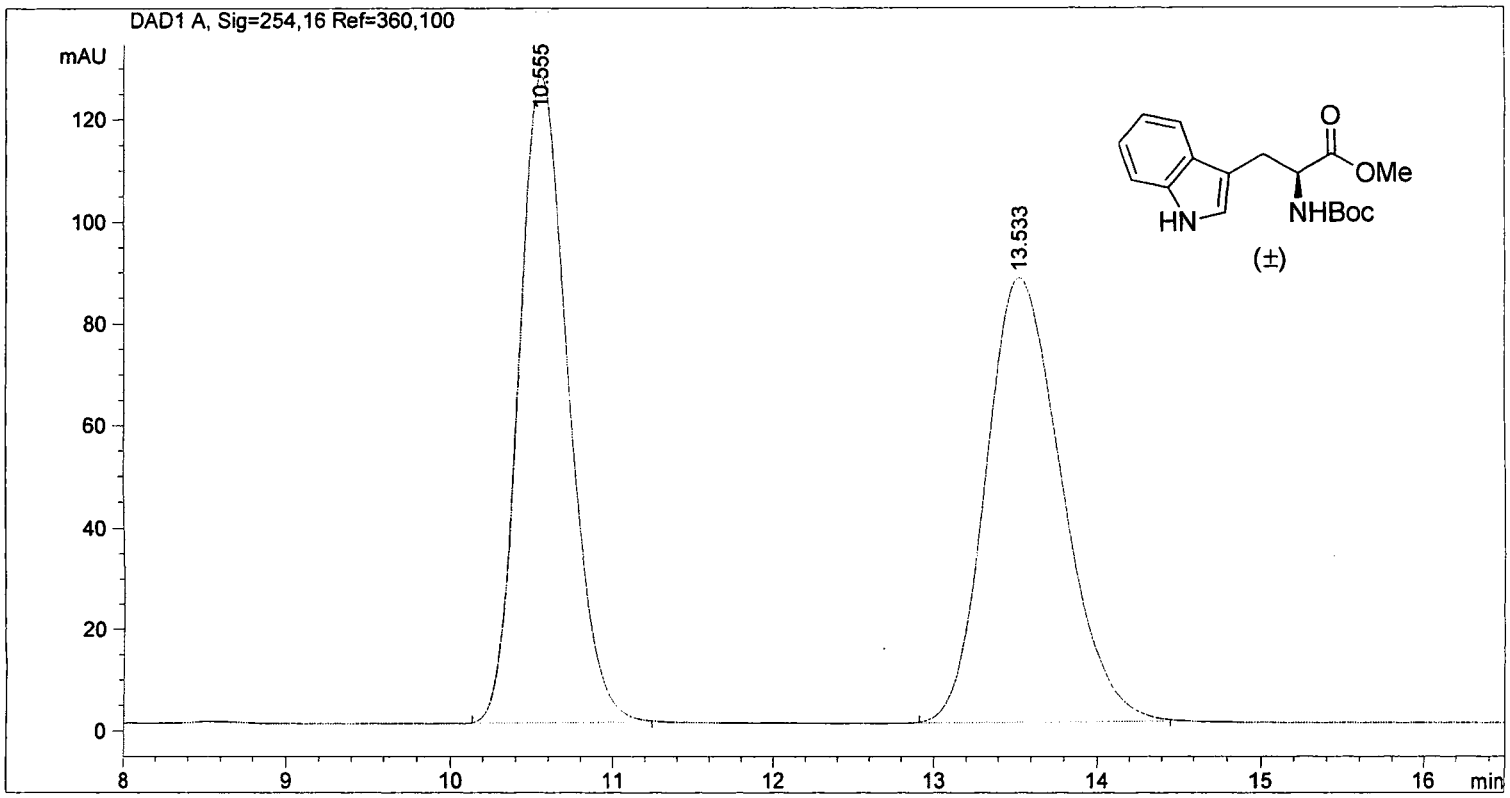


(+)-**30**



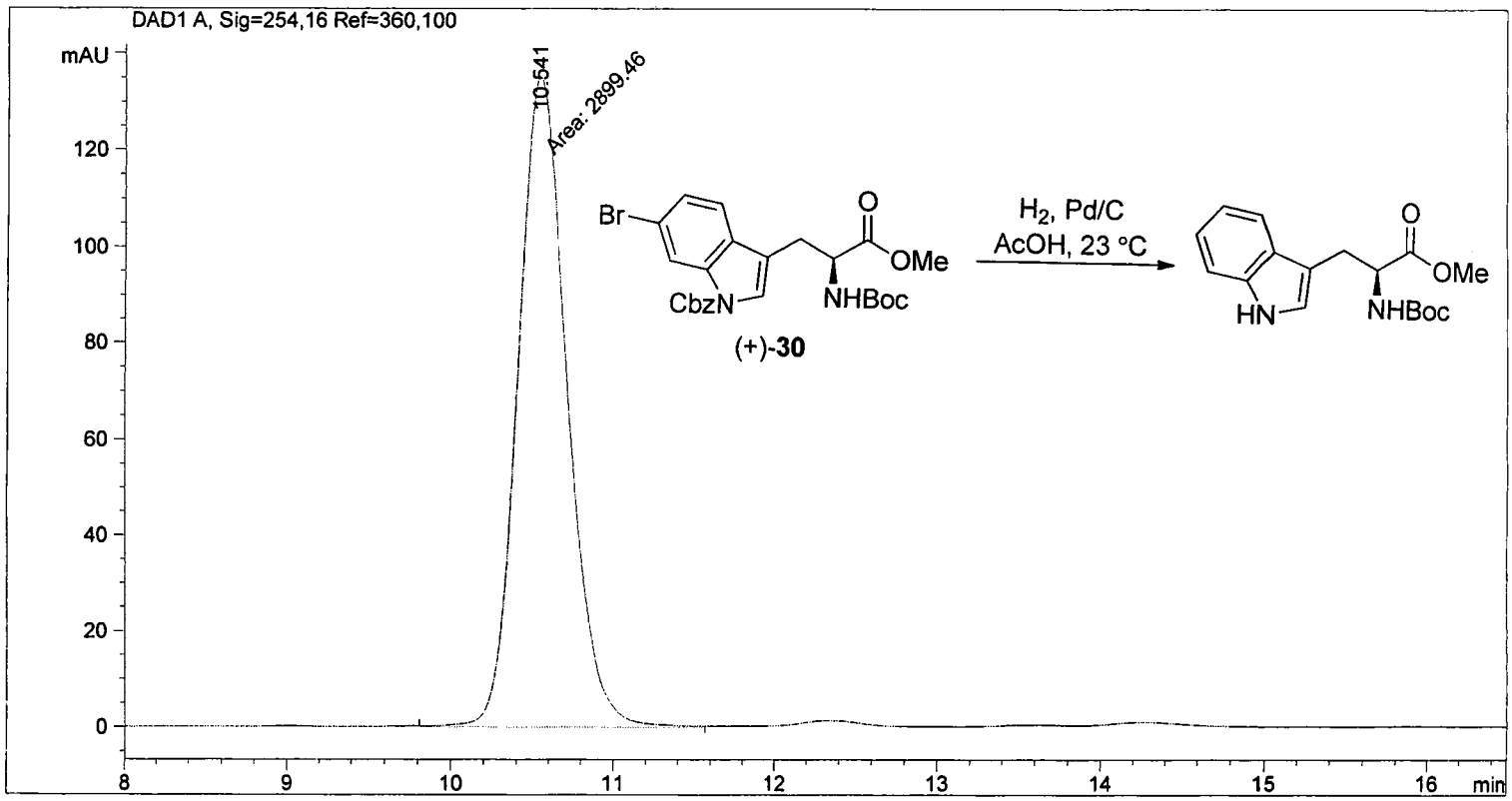

SAMPLE
solvent CDCl3
ACQUISITION
sfrq 125.795
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs 8
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 11111
ct 0
alock wnt
gain n
FLAGS 60
il n
in n
dp y
hs nn
DISPLAY
sp -2515.9
wp 30187.6
vs 596
sc 0
wc 250
hzmm 120.75
is 500.00
rfl 16009.1
rfp 9714.2
th 20
ins 1.000
al ph
DEC. & VT
dfrq 500.229
dn H1
dpwr 37
dof -500.0
dm y
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
lb 0.30
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
Br
CbzN
NHBoc
O
OMe
(+)-30
200
180
160
140
120
100
80
60
40
20
0
ppm

```
==========================================================================
Injection Date  :                                    Seq. Line :   1
Sample Name     :                                     Location :
Acq. Operator   :                                          Inj :   1
                                                   Inj Volume : 0 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 5 µl
Acq. Method     :
Last changed    :
Analysis Method :
Last changed    :
```

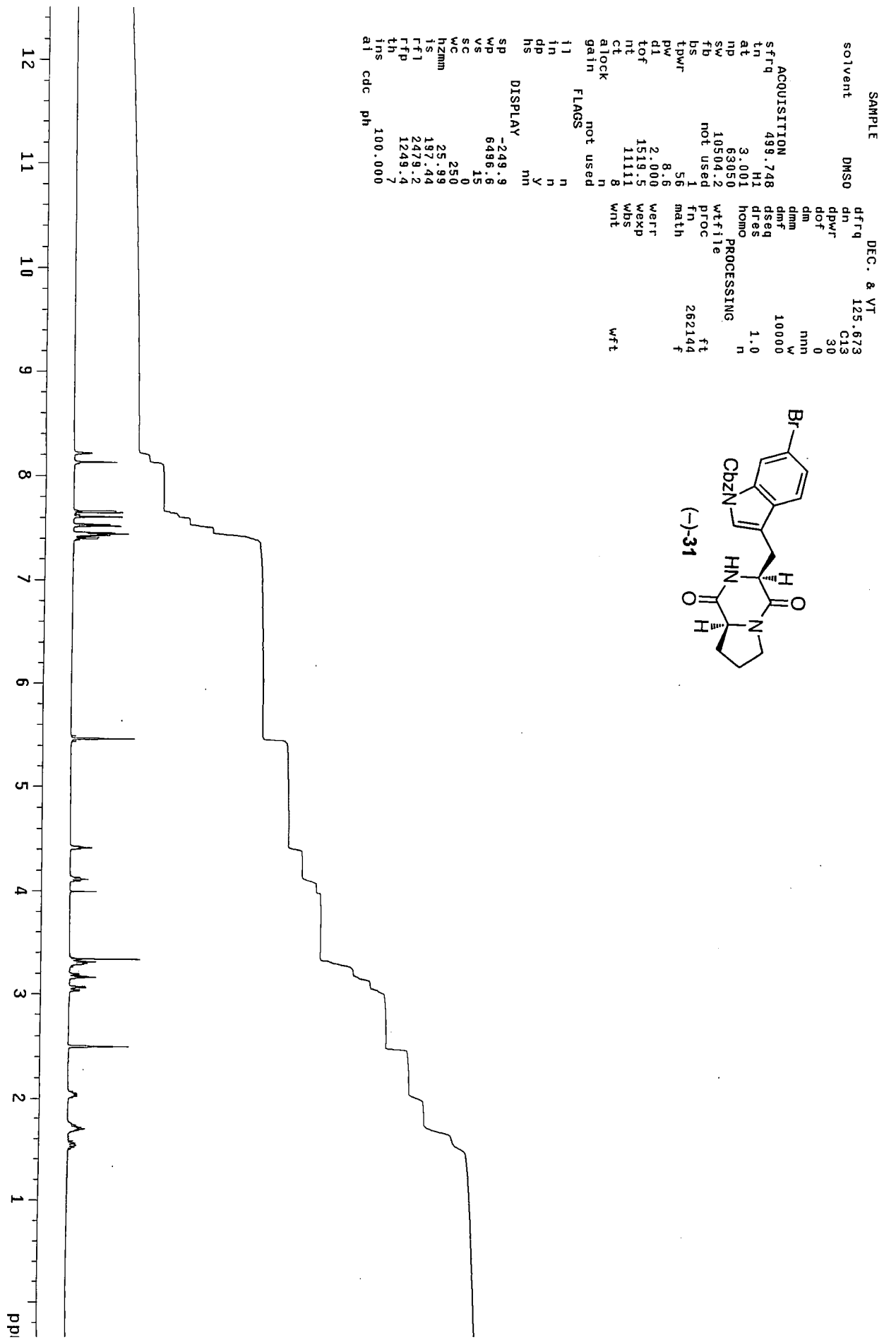


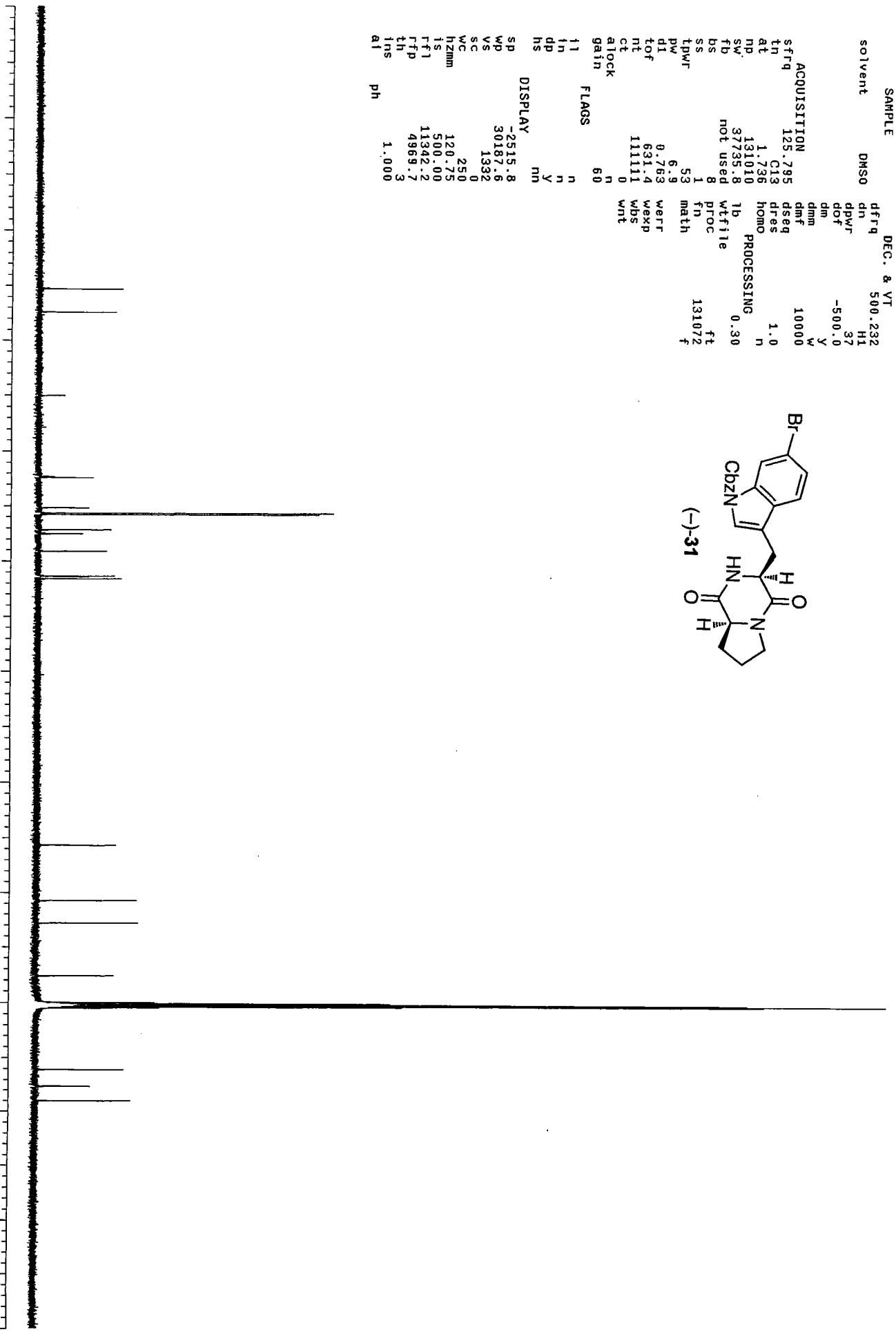

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.555 BB    0.3237 2673.67554  126.95678  48.7209
   2  13.533 BB    0.4994 2814.06738   87.21879  51.2791

Totals :                  5487.74292  214.17557

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

```
==========================================================================
Injection Date  :                                  Seq. Line :   1
Sample Name     :                                   Location :
Acq. Operator   :                                        Inj :   1
Acq. Method     :
Analysis Method :
Last changed    :
```

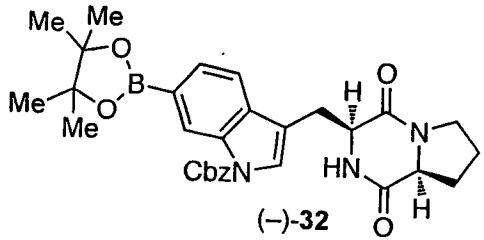



```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.541 MM    0.3577 2899.45532  135.09810 100.0000

Totals :                  2899.45532  135.09810

 Results obtained with enhanced integrator!
==========================================================================
                          *** End of Report ***
```

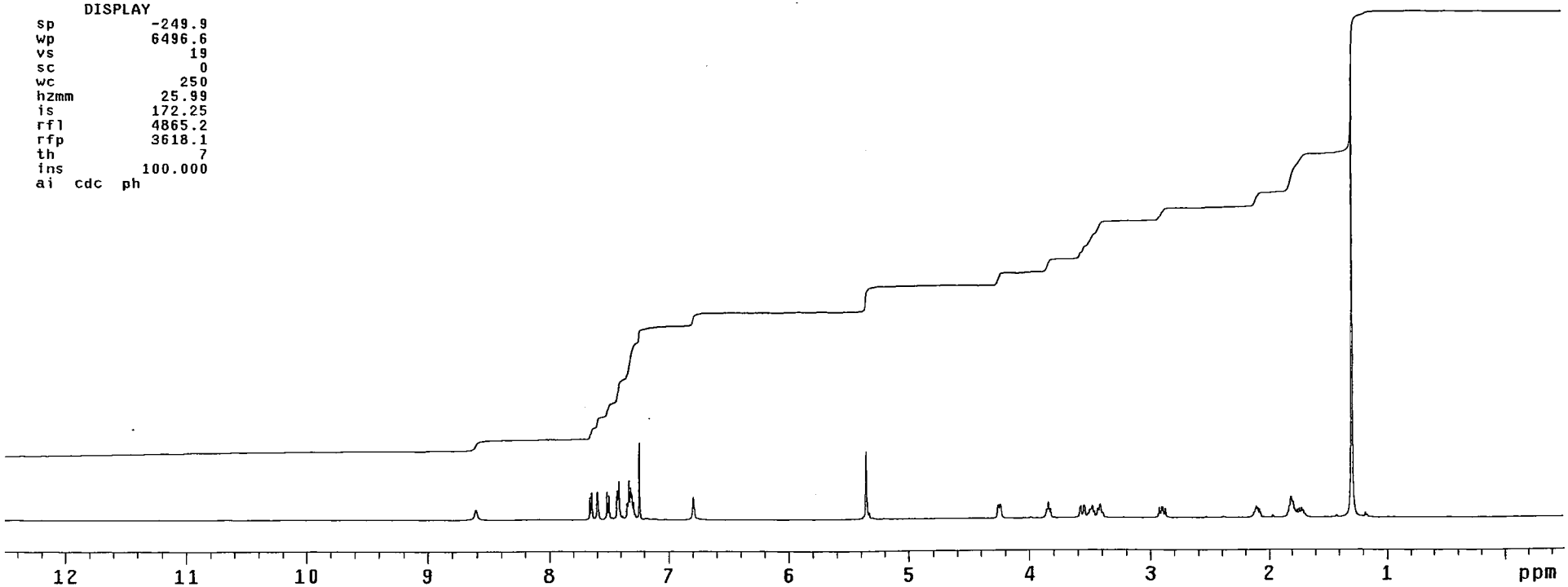

SAMPLE
solvent DMSO
ACQUISITION
sfrq 499.748
tn H1
at 3.001
np 63050
sw 10504.2
fb not used
bs 1
tpwr 56
pw 8.6
d1 2.000
tof 1519.5
nt 11111
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -249.9
wp 6496.6
vs 15
sc 0
wc 250
hzmm 25.99
is 197.44
rfl 2479.2
rfp 1249.4
th 7
ins 100.000
ai cdc ph
dfrq DEC. & VT
dn C13
dpwr 30
dof 0
dm nnn
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 262144
math f
wft
wnt
Br
CbzN
HN
H
O
N
O
H
(−)-31
12
11
10
9
8
7
6
5
4
3
2
1
ppm

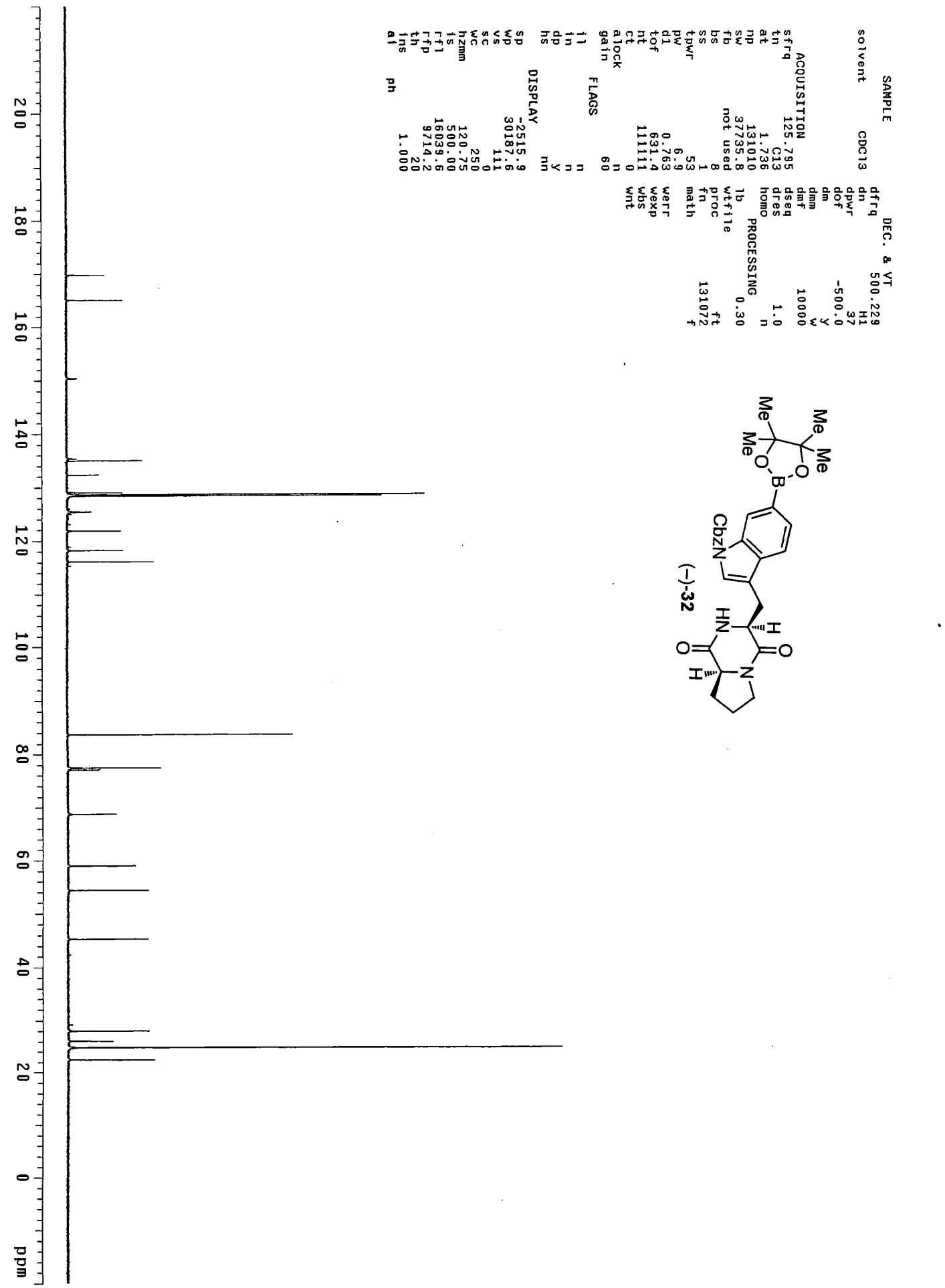

SAMPLE
solvent DMSO
ACQUISITION
sfrq 125.795
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 111111
ct 0
alock wnt
gain n 60
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -2515.8
wp 30187.6
vs 1332
sc 0
wc 250
hzmm 120.75
is 500.00
rfl 11342.2
rfp 4969.7
th 3
ins 1.000
al ph
dfrq DEC. & VT
dn H1
dpwr 37
dof -500.0
dm y
dmm y
dmf w
dseq 10000
dres 1.0
homo n
PROCESSING
lb 0.30
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
Br
CbzN
HN
O
H
O
H
N
(–)-31
200
180
160
140
120
100
80
60
40
20
0
ppm

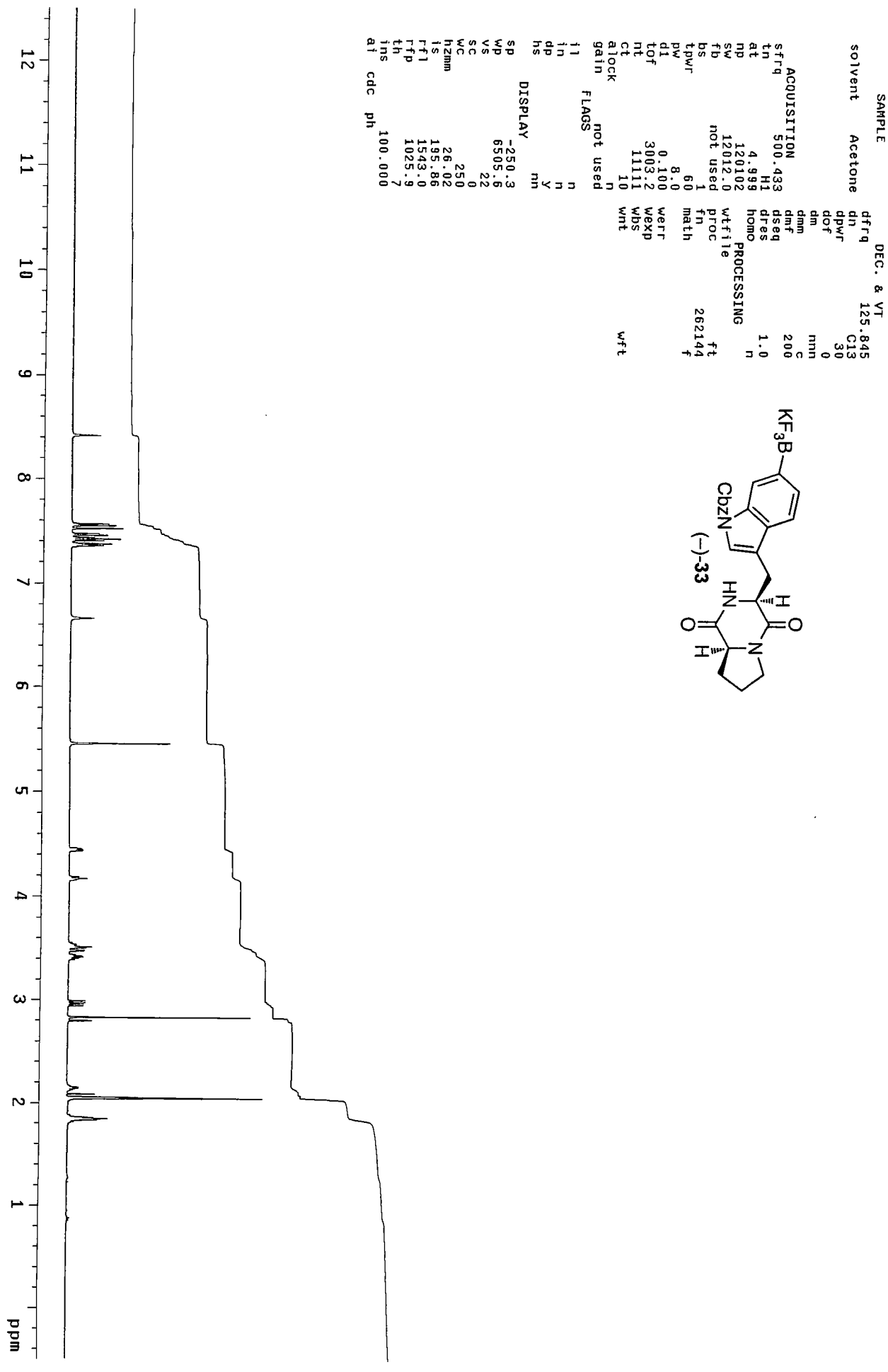
SAMPLE		DEC. & VT	
solvent	CDCl3	dfrq	125.672
		dn	C13
		dpwr	30
		dof	0
		dm	nnn
		dmm	w
ACQUISITION		dmf	10000
sfrq	499.746	dseq	
tn	H1	dres	1.0
at	3.001	homo	n
np	63050	PROCESSING	
sw	10504.2	wtfile	
fb	not used	proc	ft
bs	1	fn	262144
tpwr	56	math	f
pw	8.6		
d1	2.000	werr	
tof	1519.5	wexp	
nt	1111	wbs	
ct	10	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-249.9		
wp	6496.6		
vs	19		
sc	0		
wc	250		
hzmm	25.99		
is	172.25		
rfl	4865.2		
rfp	3618.1		
th	7		
ins	100.000		
ai cdc ph			

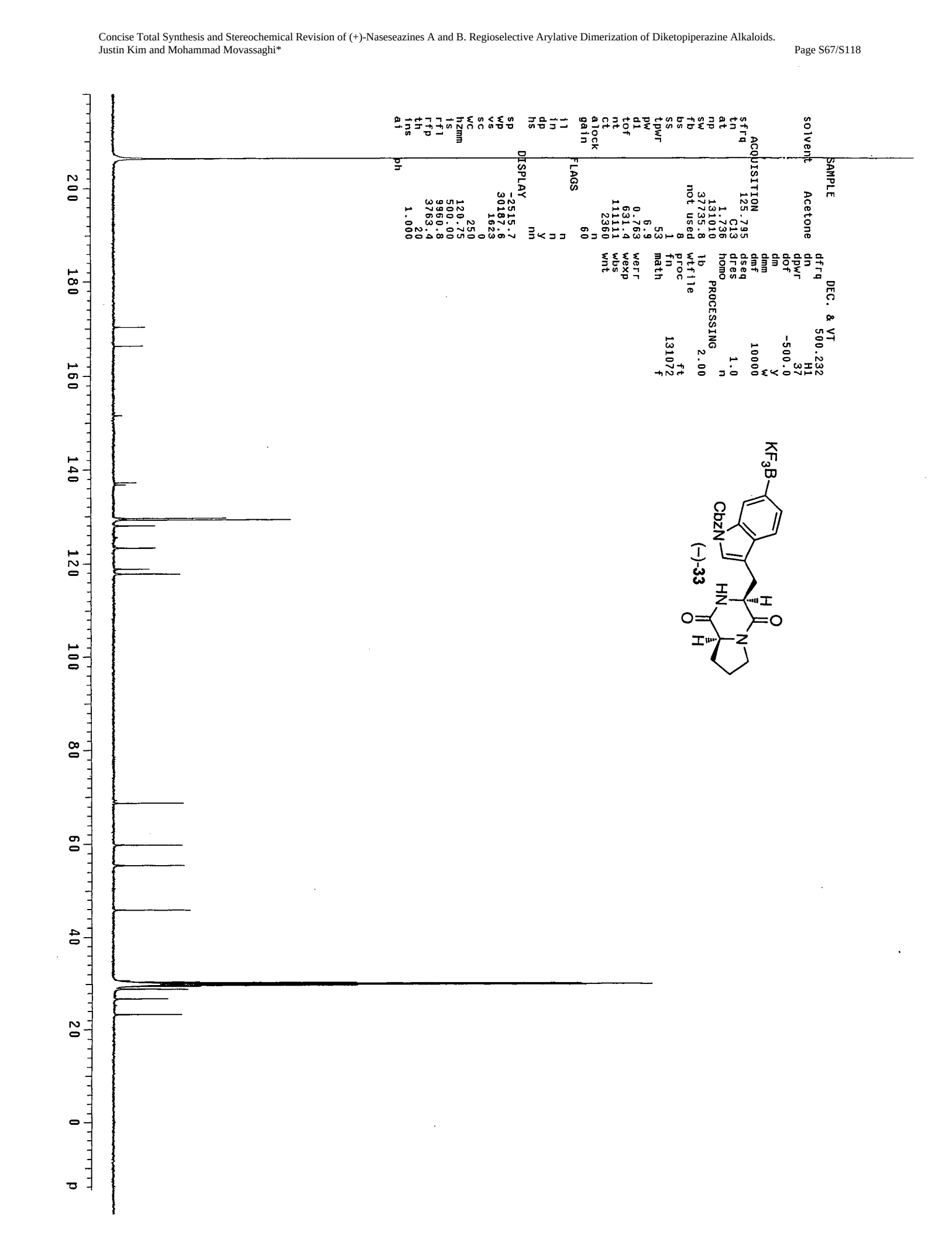


(–)-32

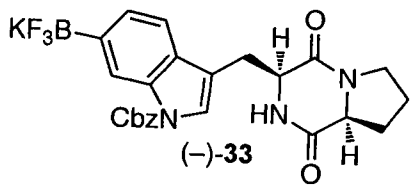



SAMPLE
solvent CDCl3
ACQUISITION
sfrq 125.795
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs 8
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 111111
ct 0
alock n
gain 60
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -2515.9
wp 30187.6
vs 111
sc 0
wc 250
hzmm 120.75
is 500.00
rfl 16039.6
rfp 9714.2
th 20
ins 1.000
ai ph
DEC. & VT
dfrq 500.229
dn H1
dpwr 37
dof -500.0
dm y
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
lb 0.30
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
Me
Me
Me
Me
O
B
O
CbzN
HN
H
O
O
N
H
(−)-32
200
180
160
140
120
100
80
60
40
20
0
ppm

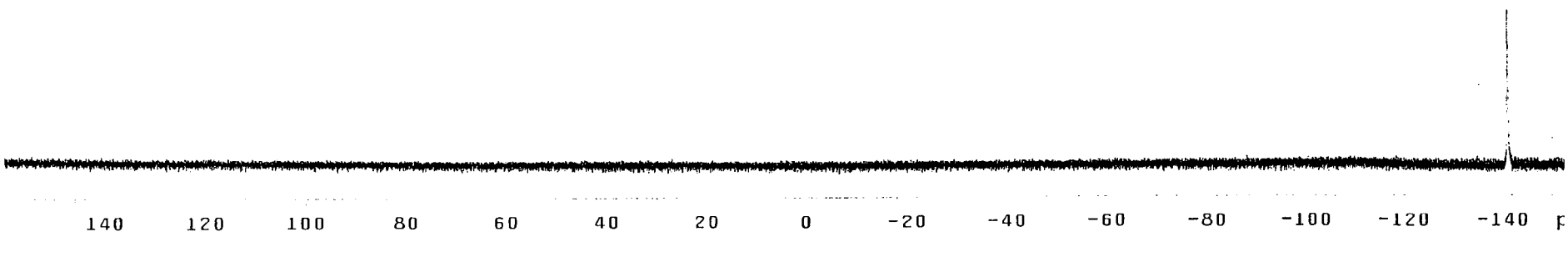

SAMPLE
solvent Acetone
ACQUISITION
sfrq 500.433
tn H1
at 4.999
np 120102
sw 12012.0
fb not used
bs 1
tpwr 60
pw 8.0
d1 0.100
tof 3003.2
nt 11111
ct 10
alock wft
gain n
FLAGS
not used
il n
in n
dp y
hs nn
DISPLAY
sp -250.3
wp 6505.6
vs 22
sc 0
wc 250
hzmm 26.02
is 195.86
rfl 1543.0
rfp 1025.9
th 7
ins 100.000
al cdc ph
DEC. & VT
dfrq 125.845
dn C13
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 262144
math f
wft
KF3B
CbzN
HN
H
O
N
(−)-33
12
11
10
9
8
7
6
5
4
3
2
1
ppm

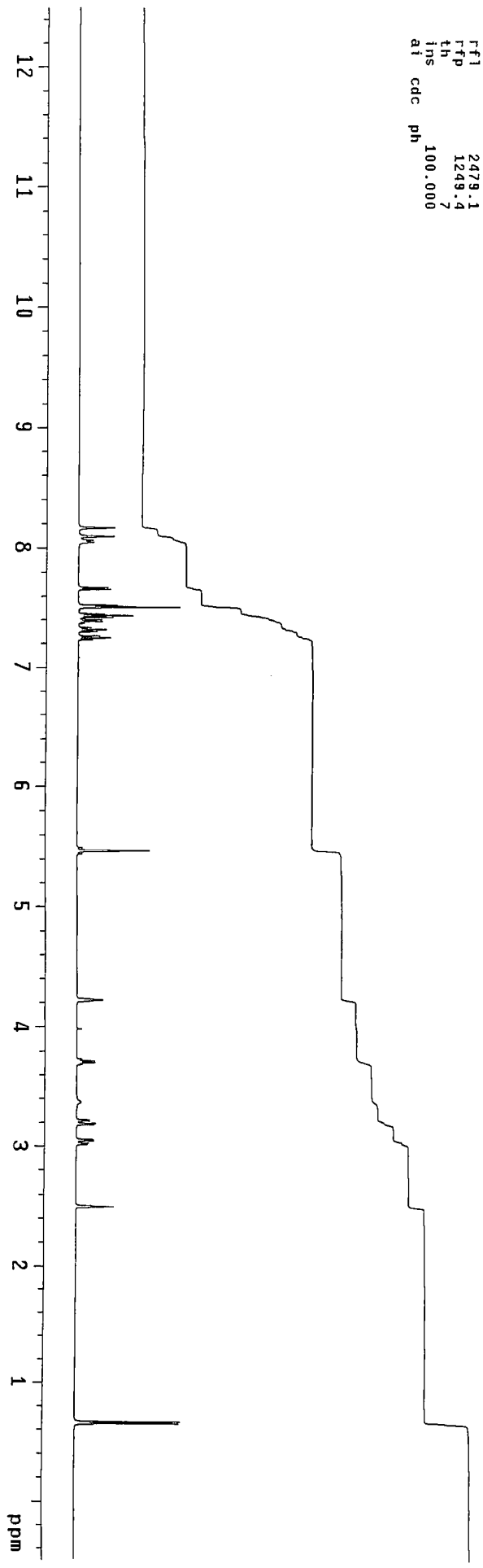

SAMPLE
solvent Acetone
ACQUISITION
sfrq 125.795
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs 8
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 111111
ct 2360
alock n
gain 60
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -2515.7
wp 30187.6
vs 1623
sc 0
wc 250
hzmm 120.75
is 500.00
rfl 9960.8
rfp 3763.4
th 20
ins 1.000
ai ph
DEC. & VT
dfrq 500.232
dn H1
dpwr 37
dof -500.0
dm y
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
lb 2.00
wtfile
proc ft
fn 131072
math f
wexp
wbs
wnt
KF3B
CbzN
HN
O
H
O
N
H
(−)-33
200
180
160
140
120
100
80
60
40
20
0
p

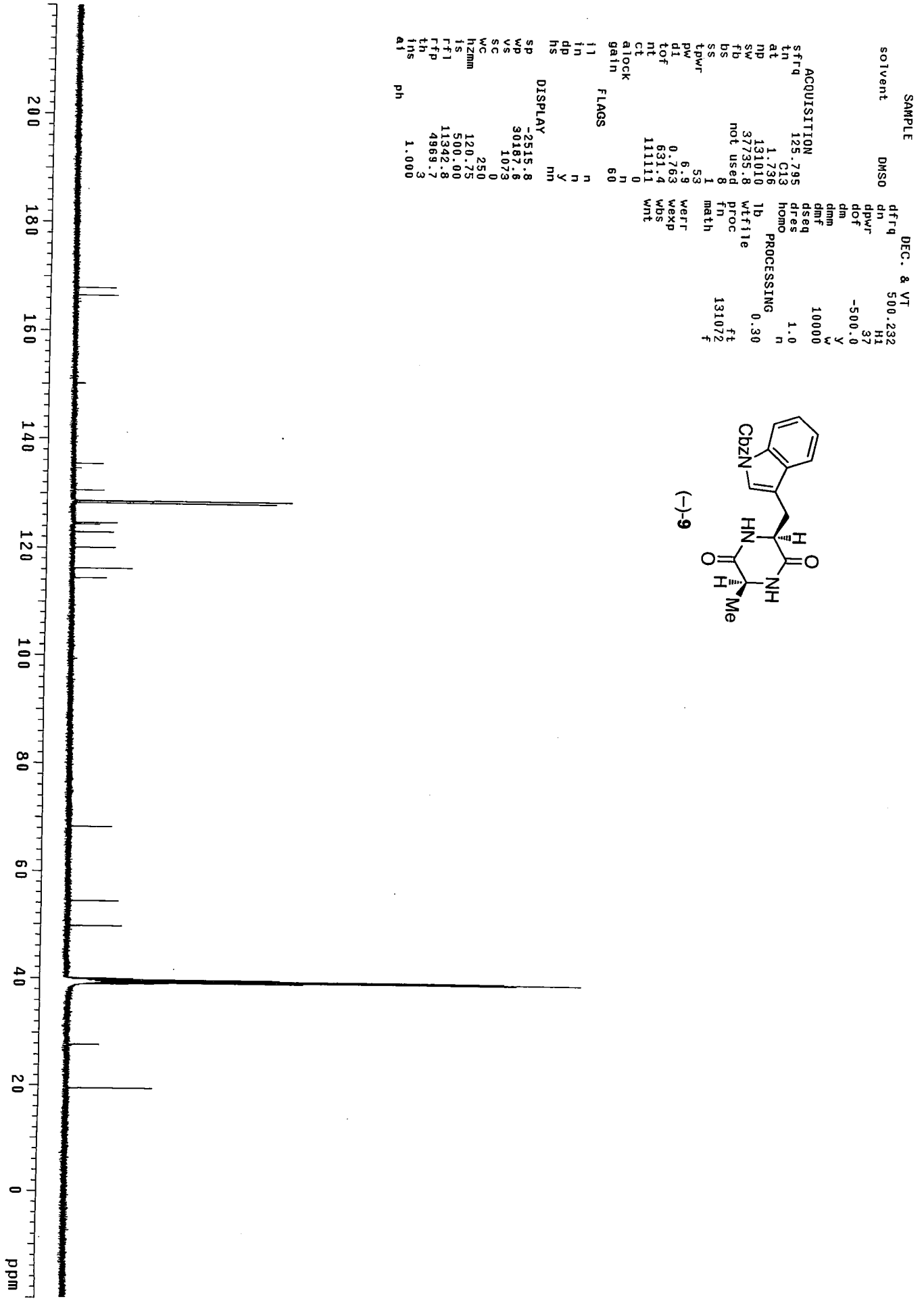
SAMPLE		DEC. & VT	
solvent	Acetone	dfrq	300.109
		dn	H1
		dpwr	30
		dof	0
		dm	nnn
		dmm	c
ACQUISITION		dmf	200
sfrq	282.383	PROCESSING	
tn	F19	lb	0.30
at	0.300	wtfile	
np	59906	proc	ft
sw	100000.0	fn	262144
fb	55000		
bs	16	werr	
tpwr	56	wexp	
pw	11.0	wbs	
d1	4.000	wnt	
tof	29637.2		
nt	111111		
ct	0		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-45181.3		
wp	90361.8		
vs	24		
sc	0		
wc	250		
hzmm	361.45		
is	500.00		
rfl	49424.0		
rfp	0		
th	20		
ins	100.000		
nm	ph		

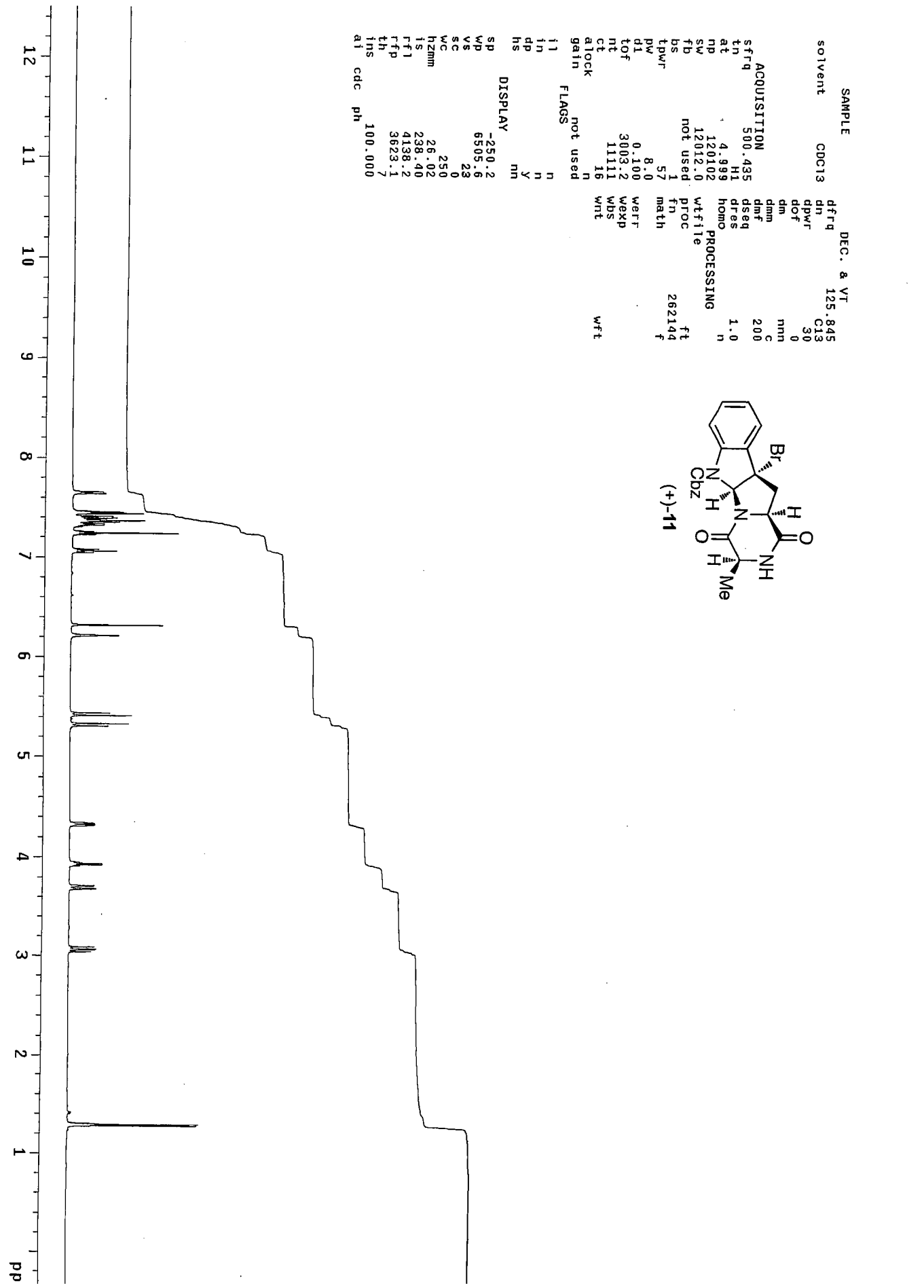


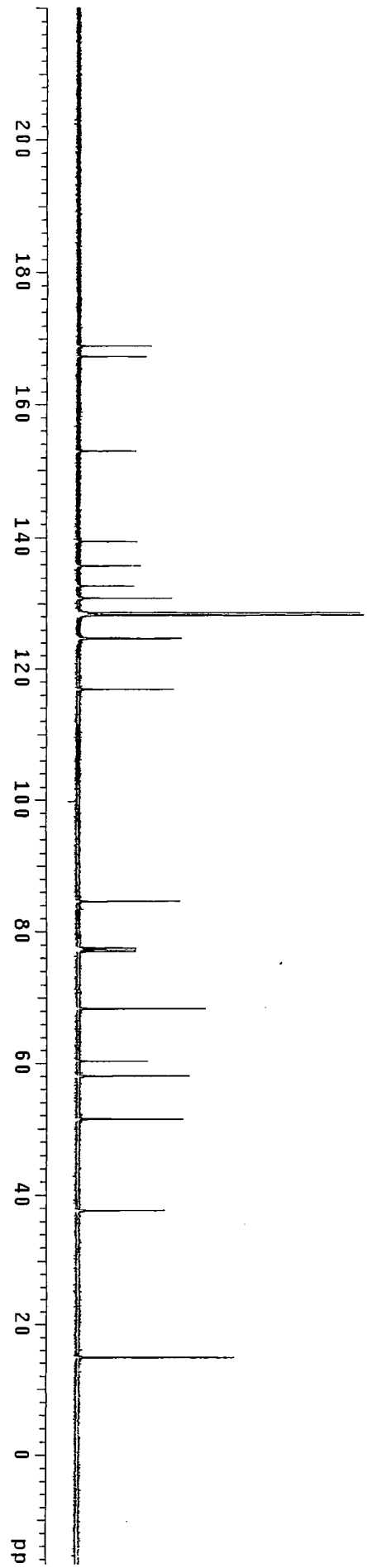
(–)-**33**



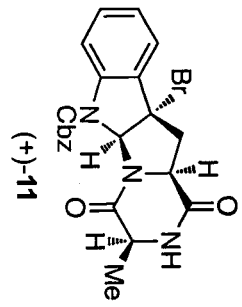

SAMPLE
solvent DMSO
ACQUISITION
sfrq 499.748
tn H1
at 3.001
np 63050
sw 10504.2
fb not used
bs 1
tpwr 56
pw 8.6
d1 2.000
tof 1519.5
nt 11111
ct 9
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -249.9
wp 6496.6
vs 15
sc 0
wc 250
hzmm 25.99
is 178.08
rfl 2479.1
rfp 1249.4
th 7
ins 100.000
ai cdc ph
DEC. & VT
dfrq 125.673
dn C13
dpwr 30
dof 0
dm nnn
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 262144
math f
werr
wexp
wbs
wnt wft
CbzN
HN
NH
O
H
Me
(−)-9
ppm


SAMPLE
solvent DMSO
ACQUISITION
sfrq 125.795
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs 8
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 111111
ct 0
alock n
gain 60
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -2515.8
wp 30187.6
vs 1073
sc 0
wc 250
hzmm 120.75
is 500.00
rfl 11342.8
rfp 4969.7
th 3
ins 1.000
ai ph
DEC. & VT
dfrq 500.232
dn H1
dpwr 37
dof -500.0
dm y
dmm y
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
lb 0.30
wtfile
proc ft
fn 131072
math f
wexp
wbs
wnt
CbzN
HN
NH
O
Me
(−)-9
200
180
160
140
120
100
80
60
40
20
0
ppm

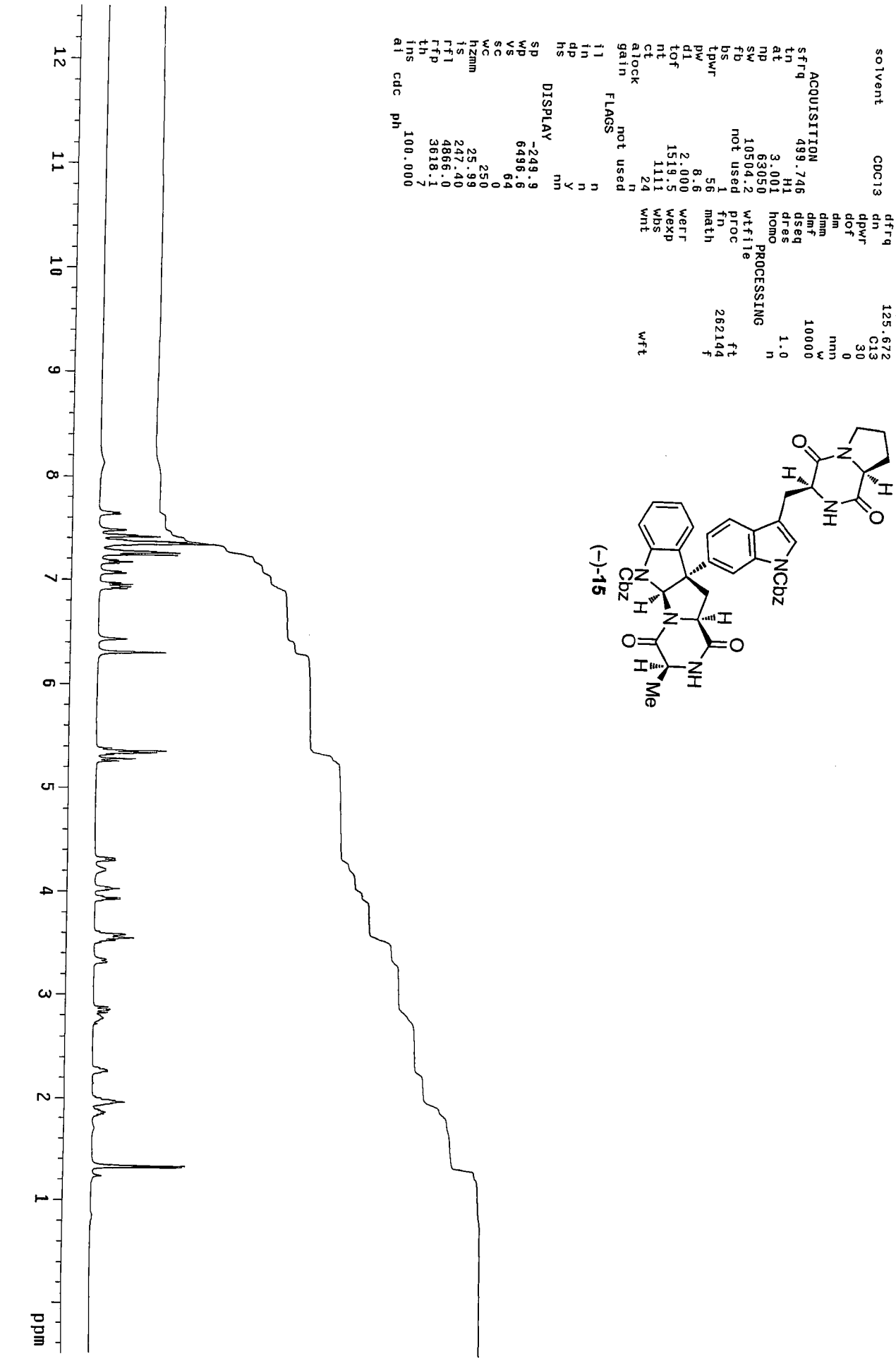

SAMPLE
solvent CDCl3
ACQUISITION
sfrq 500.435
tn H1
at 4.999
np 120102
sw 12012.0
fb not used
bs
tpwr 57
pw 8.0
d1 0.100
tof 3003.2
nt 11111
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 6505.6
vs 23
sc 0
wc 250
hzmm 26.02
is 238.40
rfl 4138.2
rfp 3623.1
th 7
ins
ai cdc ph 100.000
dfrq
dn
dpwr
dof
dm
dmm
dmf
dseq
dres
homo
PROCESSING
wtfile
proc
fn
math
werr
wexp
wbs
wnt
DEC. & VT
125.845
C13
30
0
nnn
c
200
1.0
n
ft
262144
f
wft
Br
N
Cbz
H
N
H
O
O
NH
Me
(+)-11
12
11
10
9
8
7
6
5
4
3
2
1
ppm

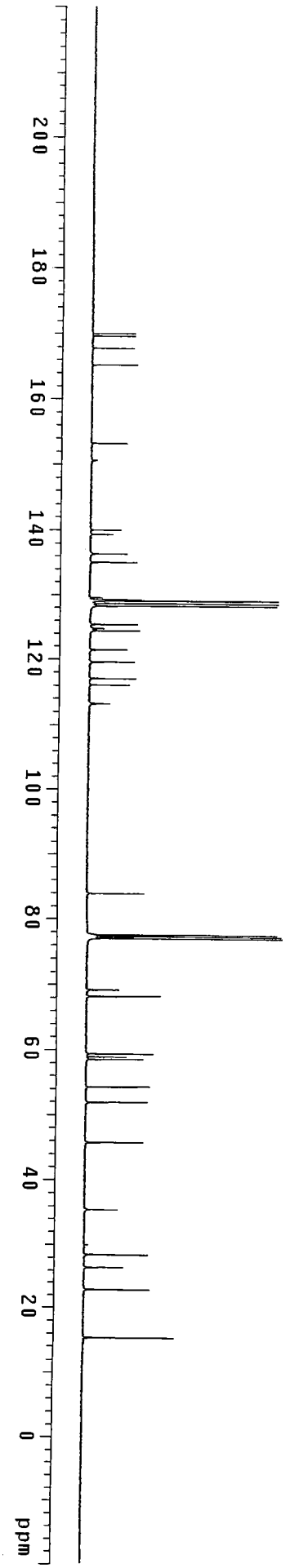



```
SAMPLE
solvent      CDCl3      dfrq      DEC. & VT
                        dn        500.229
                        dpwr      H1
                        dof       37
                        dm        -500.0
                        dmm       y
                        dmf       w
ACQUISITION             dseq      10000
sfrq      125.795       dres
tn            C13       homo      1.0
at          1.736                 n
np         131010     PROCESSING
sw        37735.8      lb        0.30
fb       not used      wtfile
bs              8      proc
ss              1      fn        ft
tpwr           53      math      131072
pw            6.9                f
d1          0.763     werr
tof         631.4     wexp
nt         111111     wbs
ct            128     wnt
alock           n
gain           60
          FLAGS
il              n
in              n
dp              y
hs             nn
          DISPLAY
sp        -2515.9
wp        30187.6
vs             209
sc               0
wc             250
hzmm        120.75
is          500.00
rfl        16032.7
rfp         9714.2
th              20
ins          1.000
al       ph
```

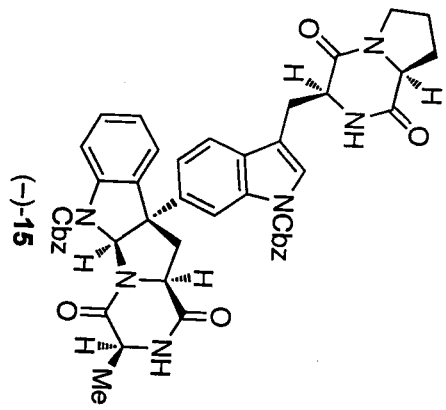


(+)-**11**

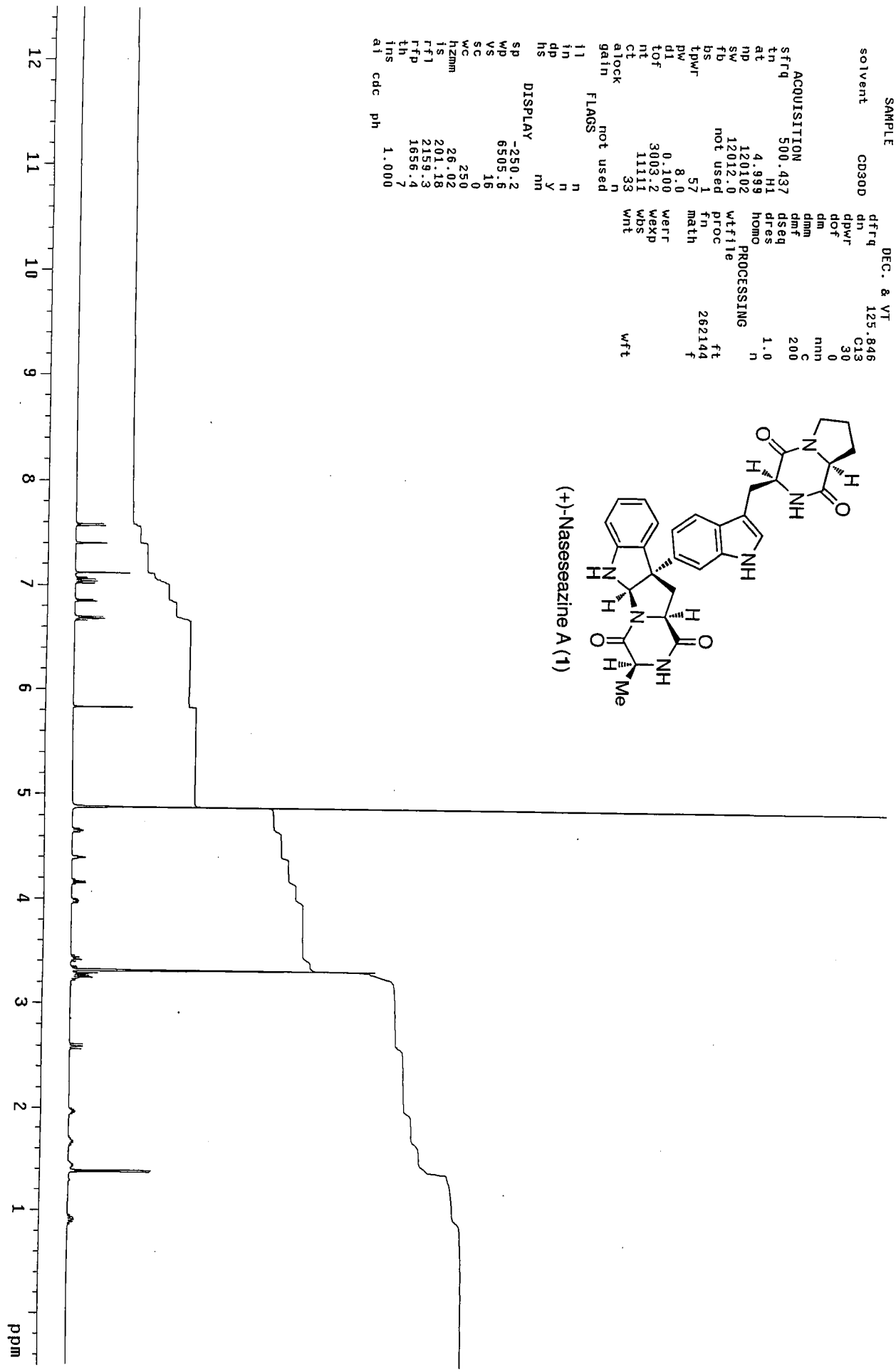

SAMPLE
solvent CDCl3
ACQUISITION
sfrq 499.746
tn H1
at 3.001
np 63050
sw 10504.2
fb not used
bs 1
tpwr 56
pw 8.6
d1 2.000
tof 1519.5
nt 1111
ct 24
alock n
gain n
FLAGS
not used
il n
in n
dp y
hs nn
DISPLAY
sp -249.9
wp 6496.6
vs 64
sc 0
wc 250
hzmm 25.99
is 247.40
rfl 4866.0
rfp 3618.1
th 7
ins
ai cdc ph 100.000
DEC. & VT
dfrq 125.672
dn C13
dpwr 30
dof 0
dm nnn
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 262144
math f
werr
wexp
wbs
wnt wft
(-)-15
NCbz
Cbz
Me
NH
ppm

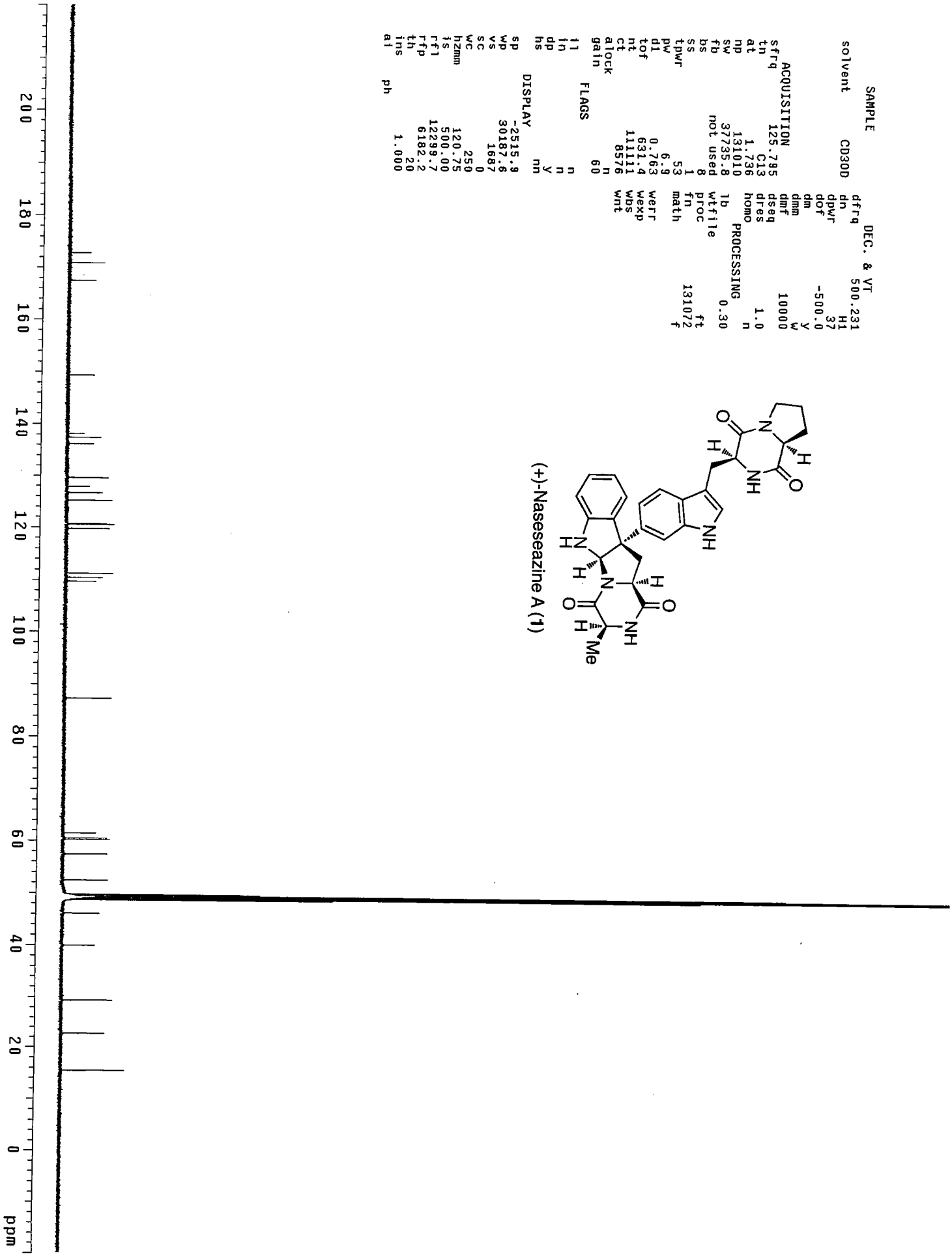



```
SAMPLE                          DEC. & VT
solvent      CDCl3          dfrq      500.229
                            dn           H1
                            dpwr         37
                            dof        -500.0
                            dm           y
ACQUISITION                 dmm          w
sfrq       125.795          dmf      10000
tn             C13          dseq         1.0
at           1.736          dres         n
np          131010          homo
sw        37735.8                PROCESSING
fb            not used      lb          1.00
bs                 8        wtfile
ss                 1        proc           ft
tpwr              53        fn        131072
pw               6.9        math           f
d1             0.763
tof            631.4        werr
nt       1.11111e+06        wexp
ct             10792        wbs
alock                       wnt
gain              60
                 FLAGS
                  n
                  n
                  y
                  nn
DISPLAY
sp          -2515.9
wp          30187.6
vs             1001
sc                0
wc              250
hzmm         120.75
is           500.00
rfl         16006.8
rfp          9714.2
th                20
ins            1.000
ai       ph
```



(−)-15


SAMPLE
solvent CD3OD
ACQUISITION
sfrq 500.437
tn H1
at 4.999
np 120102
sw 12012.0
fb not used
bs 1
tpwr 57
pw 8.0
d1 0.100
tof 3003.2
nt 11111
ct 33
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 6505.6
vs 16
sc 0
wc 250
hzmm 26.02
is 201.18
rfl 2159.3
rfp 1656.4
th 7
ins 1
ai cdc ph 1.000
DEC. & VT
dfrq 125.846
dn C13
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 262144
math f
werr
wexp
wbs
wnt wft
(+)-Naseseazine A (1)
ppm


SAMPLE
solvent CD3OD
ACQUISITION
sfrq 125.795
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs 8
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 111111
ct 8576
alock n
gain 60
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -2515.9
wp 30187.6
vs 1687
sc 0
wc 250
hzmm 120.75
is 500.00
rfl 12299.7
rfp 6182.2
th 20
ins 1.000
ai ph
DEC. & VT
dfrq 500.231
dn H1
dpwr 37
dof -500.0
dm y
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
lb 0.30
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
(+)-Naseseazine A (1)
200
180
160
140
120
100
80
60
40
20
0
ppm

SAMPLE	
solvent	DMSO

ACQUISITION	
sfrq	499.748
tn	H1
at	3.001
np	63050
sw	10504.2
fb	not used
bs	1
tpwr	56
pw	8.6
d1	2.000
tof	1519.5
nt	111111
ct	12
alock	n
gain	not used

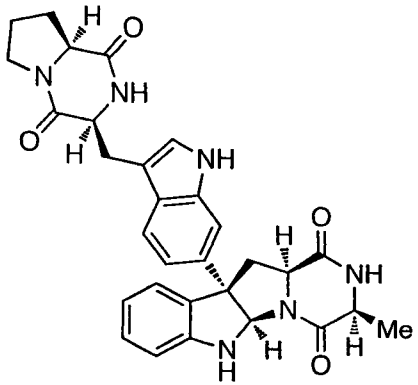
FLAGS	
il	n
in	n
dp	y
hs	nn

DISPLAY	
sp	-249.9
wp	6496.6
vs	10
sc	0
wc	250
hzmm	25.99
is	158.44
rfl	2478.9
rfp	1249.4
th	7
ins	100.000

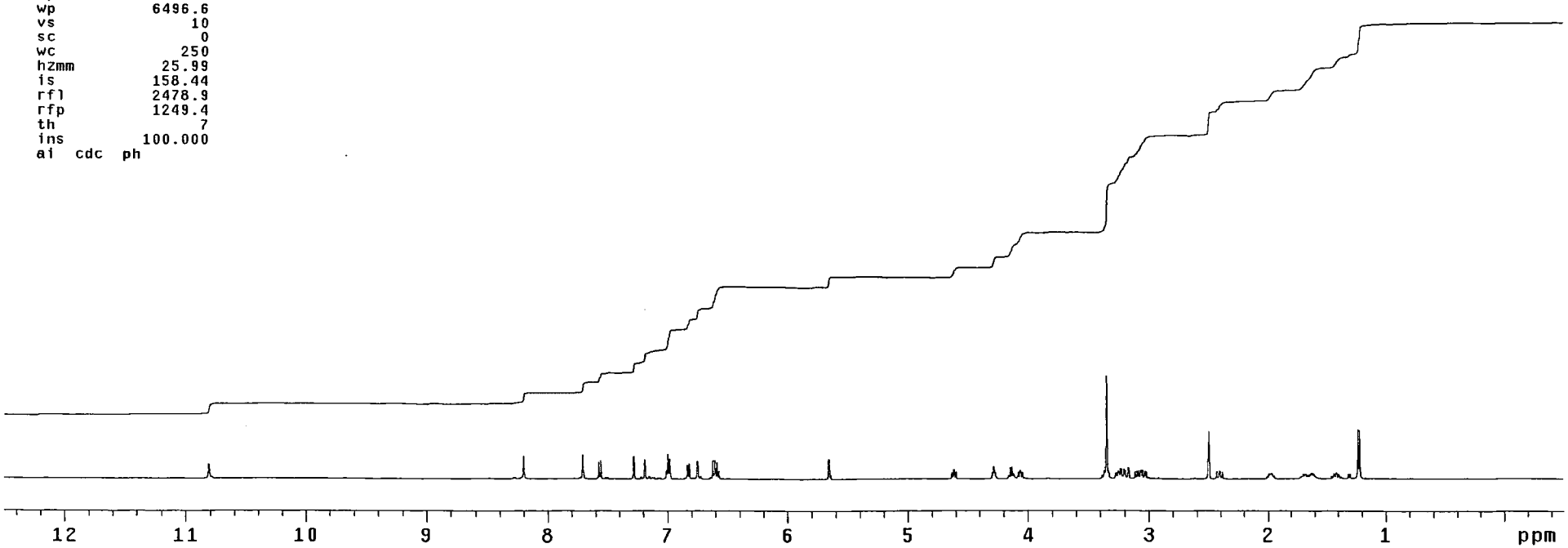
ai cdc ph

DEC. & VT	
dfrq	125.673
dn	C13
dpwr	30
dof	0
dm	nnn
dmm	w
dmf	10000
dseq	
dres	1.0
homo	n

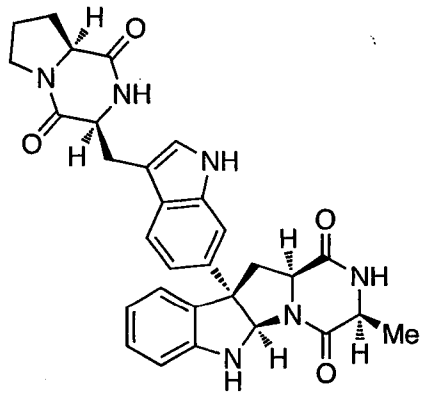
PROCESSING	
wtfile	
proc	ft
fn	262144
math	f
werr	
wexp	
wbs	
wnt	wft



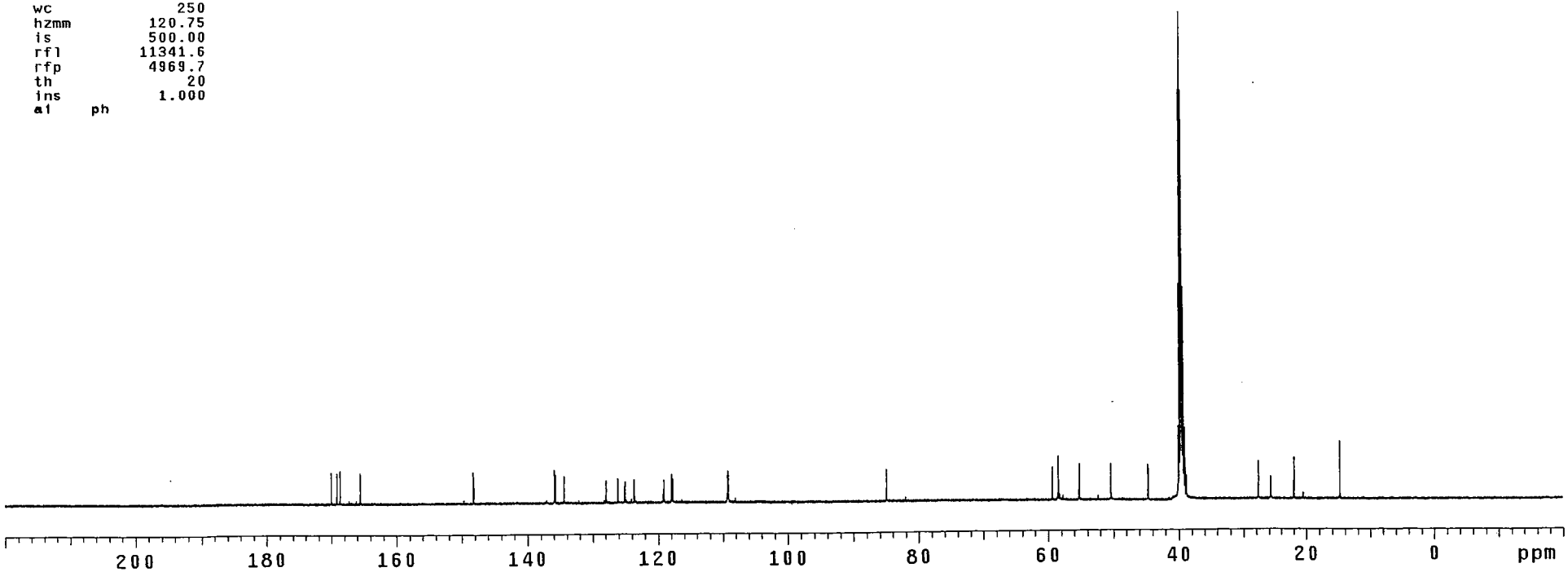
(+)-Naseseazine A (**1**)

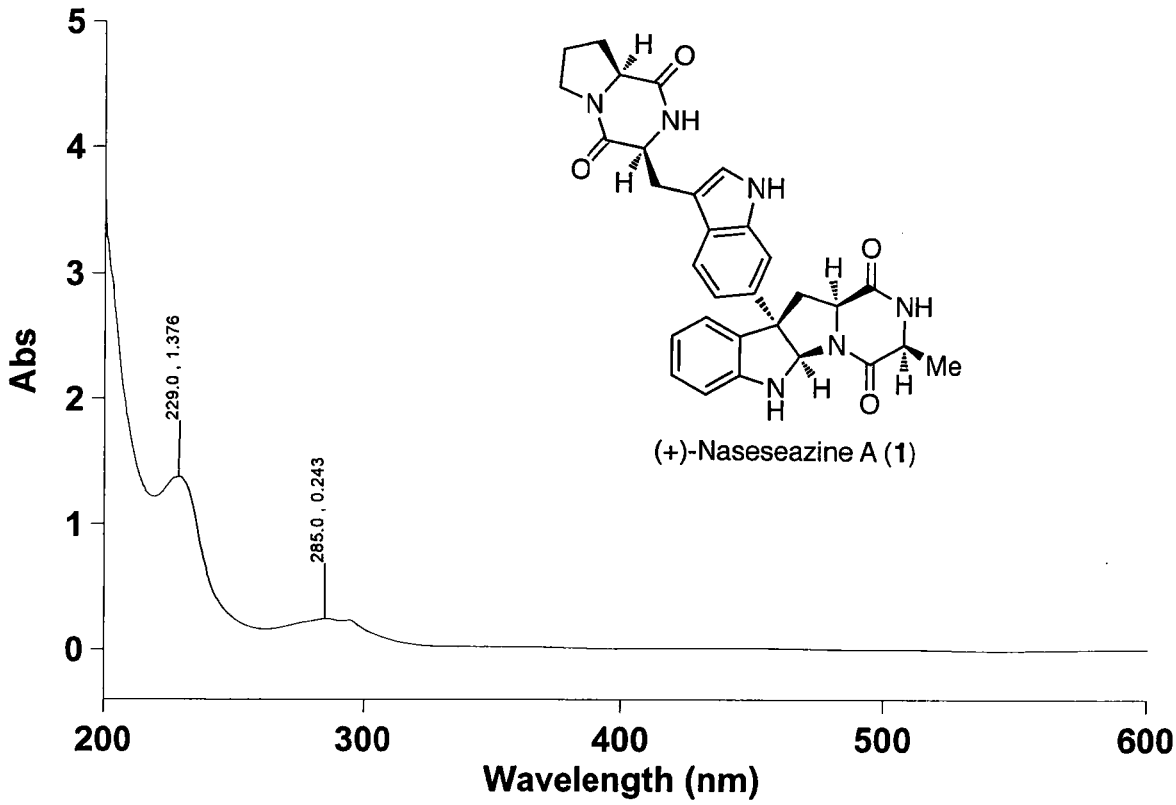


SAMPLE		DEC. & VT	
		dfrq	500.232
solvent	DMSO	dn	H1
		dpwr	37
		dof	-500.0
		dm	y
		dmm	w
ACQUISITION		dmf	10000
sfrq	125.795	dseq	
tn	C13	dres	1.0
at	1.736	homo	n
np	131010	PROCESSING	
sw	37735.8	lb	0.30
fb	not used	wtfile	
bs	8	proc	ft
ss	1	fn	131072
tpwr	53	math	f
pw	6.9		
d1	0.763	werr	
tof	631.4	wexp	
nt	1.11111e+06	wbs	
ct	14944	wnt	
alock	n		
gain	60		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-2515.8		
wp	30187.6		
vs	1144		
sc	0		
wc	250		
hzmm	120.75		
is	500.00		
rfl	11341.6		
rfp	4969.7		
th	20		
ins	1.000		
ai	ph		



(+)-Naseseazine A (**1**)



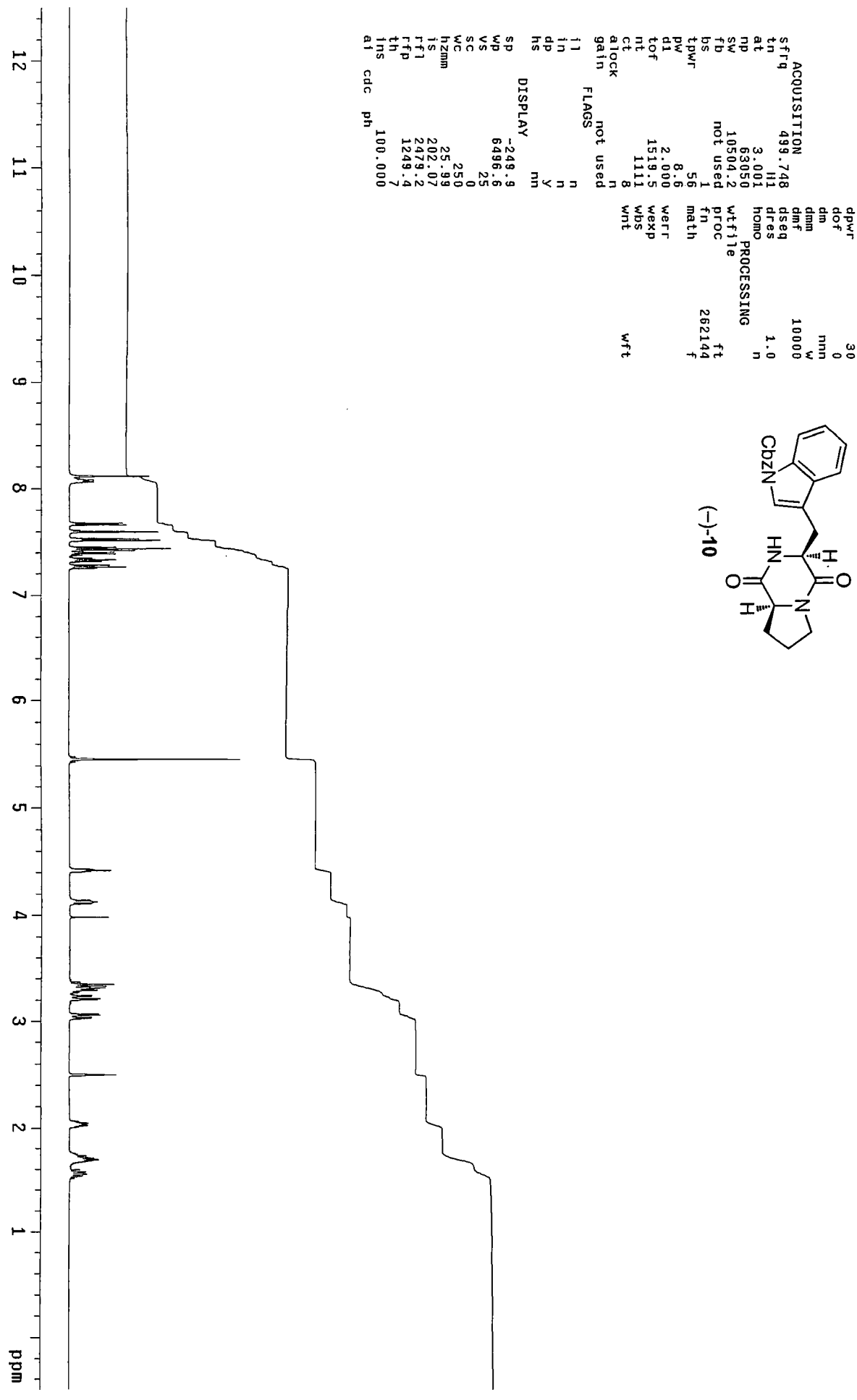


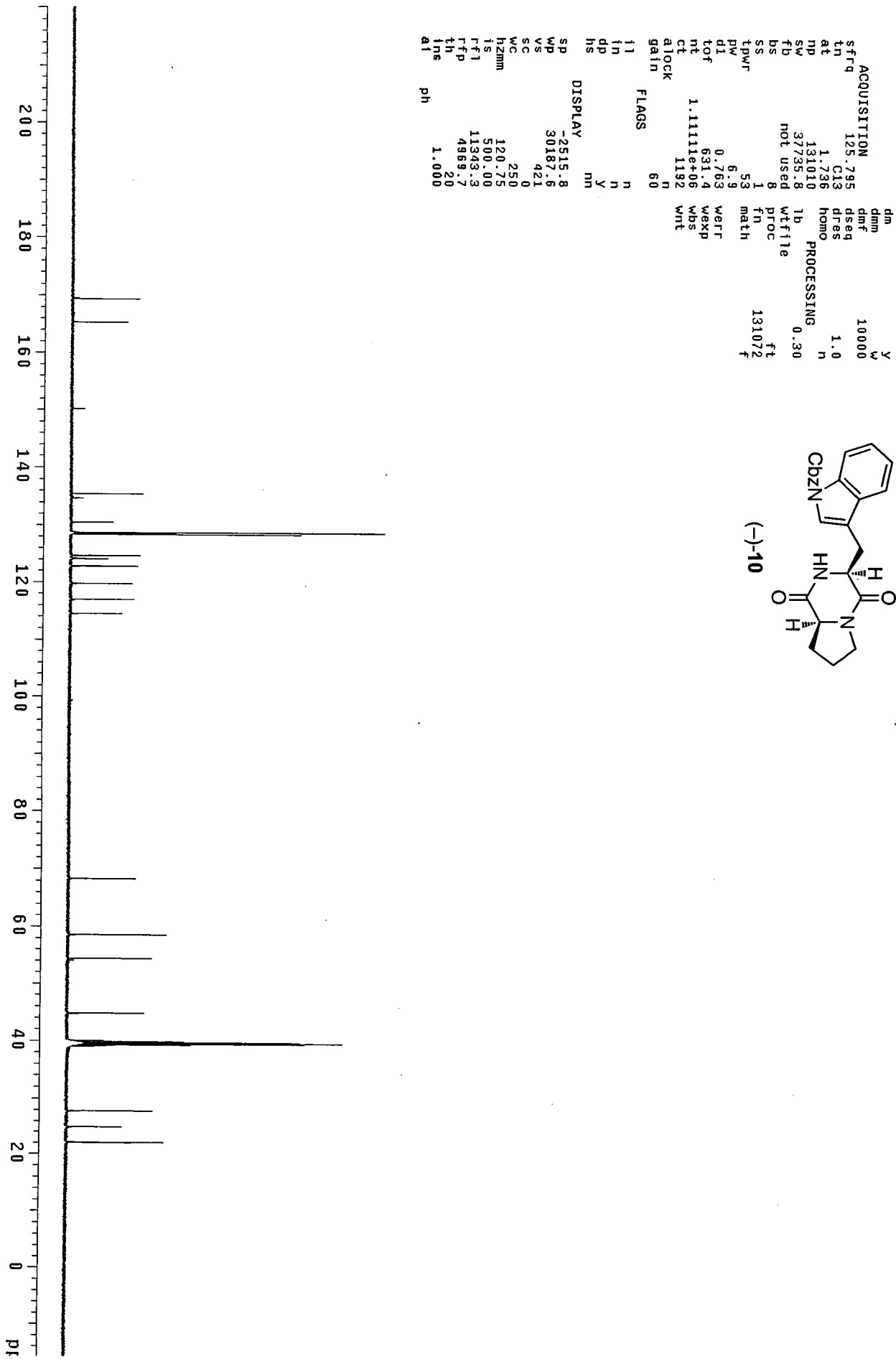
Scan Analysis Report

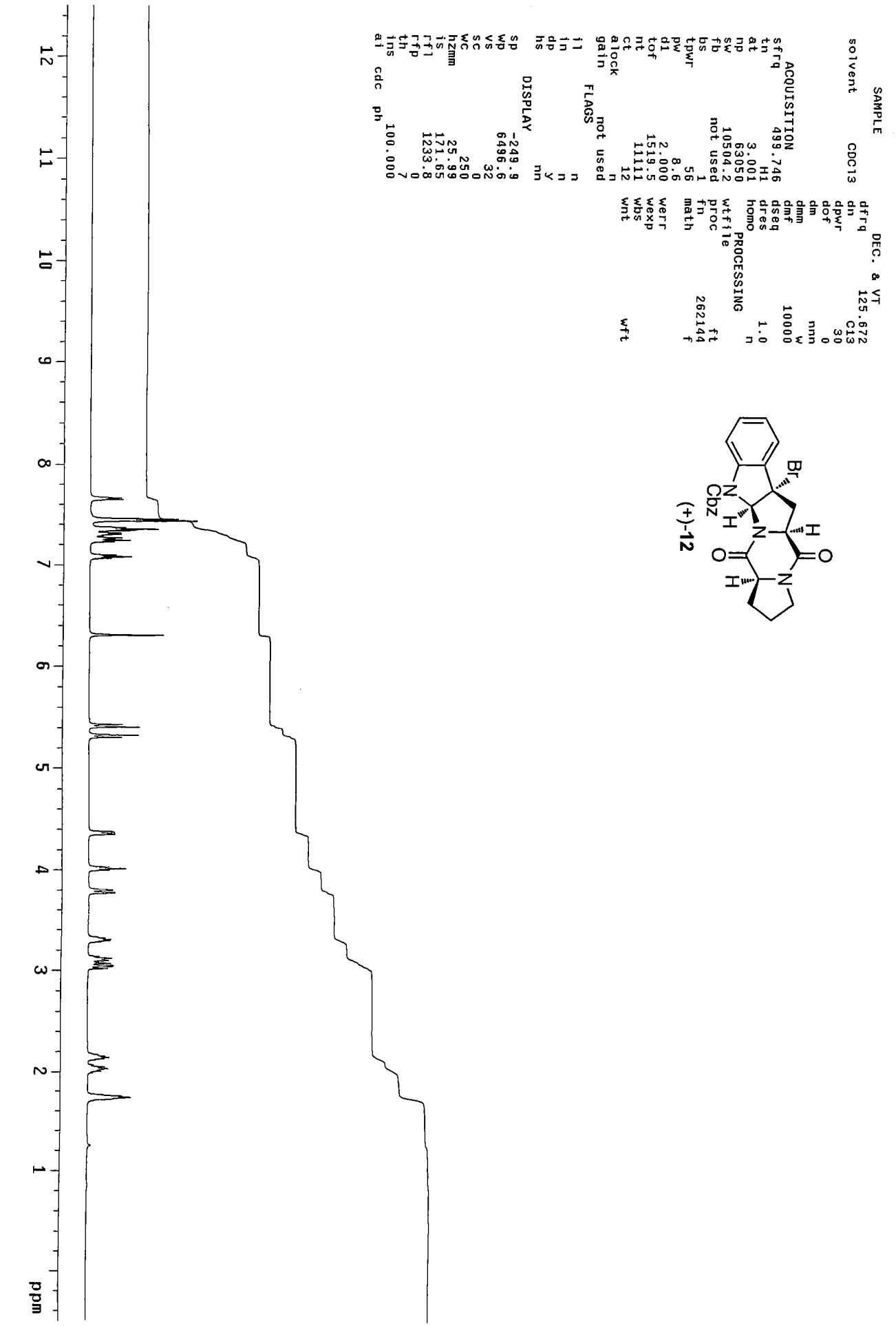
Sample Name: Naseseazine A

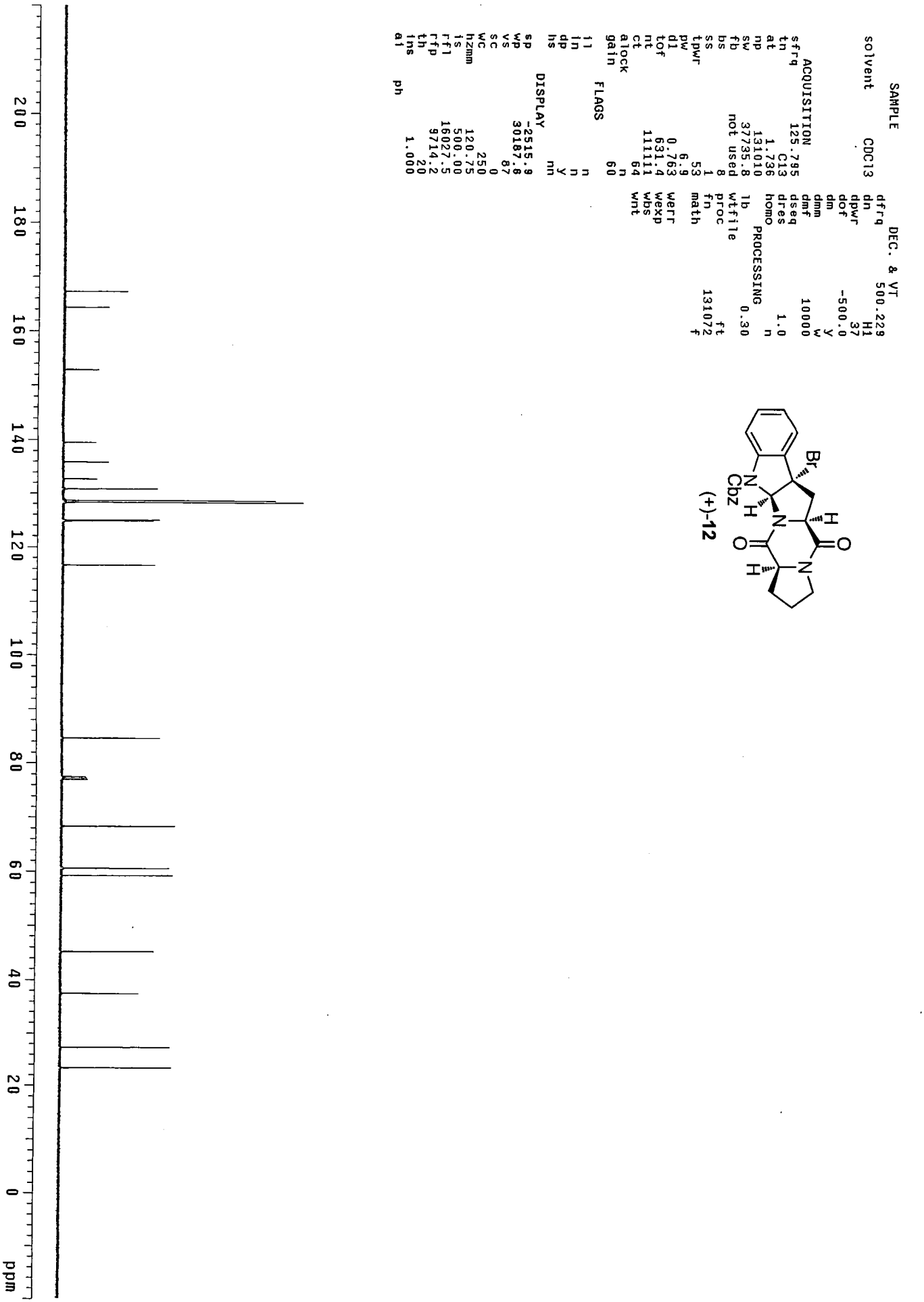
Peak Table
Peak Style Peaks
Peak Threshold 0.0100
Range 600.0nm to 200.0nm

Wavelength (nm)	Abs
285.0	0.243
229.0	1.376


SAMPLE
solvent DMSO
ACQUISITION
sfrq 499.748
tn H1
at 3.001
np 63050
sw 10504.2
fb not used
bs 1
tpwr 56
pw 8.6
d1 2.000
tof 1519.5
nt 1111
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -249.9
wp 6496.6
vs 25
sc 0
wc 250
hzmm 25.99
is 202.07
rfl 2479.2
rfp 1249.4
th 7
ins 100.000
ai cdc ph
dfrq DEC. & VT
dn 125.673
C13
dof 30
dm 0
nnn
w 10000
dmm
dmf
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 262144
math f
werr
wexp
wbs
wnt wft
CbzN
HN
O
O
H
H
O
N
(−)-10
12
11
10
9
8
7
6
5
4
3
2
1
ppm


SAMPLE
solvent DMSO
ACQUISITION
sfrq 125.795
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs 8
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 1.11111e+06
ct 1192
alock n
gain 60
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -2515.8
wp 30187.6
vs 421
sc 0
wc 250
hzmm 120.75
is 500.00
rfl 11343.3
rfp 4969.7
th 20
ins 1.000
ai ph
DEC. & VT
dfrq 500.232
dn H1
dpwr 37
dof -500.0
dm y
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
lb 0.30
wtfile
proc
ft
fn 131072
math f
wrr
wexp
wbs
wnt
CbzN
HN
H
O
O
H
N
(–)-10
200
180
160
140
120
100
80
60
40
20
0
pp


SAMPLE
solvent CDCl3
ACQUISITION
sfrq 499.746
tn H1
at 3.001
np 63050
sw 10504.2
fb not used
bs 1
tpwr 56
pw 8.6
d1 2.000
tof 1519.5
nt 11111
ct 12
alock n
gain n
FLAGS
not used
il n
in n
dp y
hs nn
DISPLAY
sp -249.9
wp 6496.6
vs 32
sc 0
wc 250
hzmm 25.99
is 171.65
rfl 1233.8
rfp 0
th 7
ins 100.000
ai cdc ph
DEC. & VT
dfrq 125.672
dn C13
dpwr 30
dof 0
dm nnn
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 262144
math f
wft
wnt
Br
N
Cbz
H
N
H
O
O
H
O
N
(+)-12
12
11
10
9
8
7
6
5
4
3
2
1
ppm


SAMPLE
solvent CDCl3
ACQUISITION
sfrq 125.795
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs 8
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 111111
ct 64
alock wnt
gain n
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -2515.9
wp 30187.6
vs 87
sc 0
wc 250
hzmm 120.75
is 500.00
rfl 16027.5
rfp 9714.2
th 20
ins 1.000
ph
dfrq
dn
dpwr
dof
dm
dmm
dmf
dseq
dres
homo
PROCESSING
lb
wtfile
proc
fn
math
werr
wexp
wbs
wnt
DEC. & VT
500.229
H1
37
-500.0
y
w
10000
1.0
n
0.30
ft
131072
f
Br
N
Cbz
H
N
H
O
O
H
N
(+)-12
200
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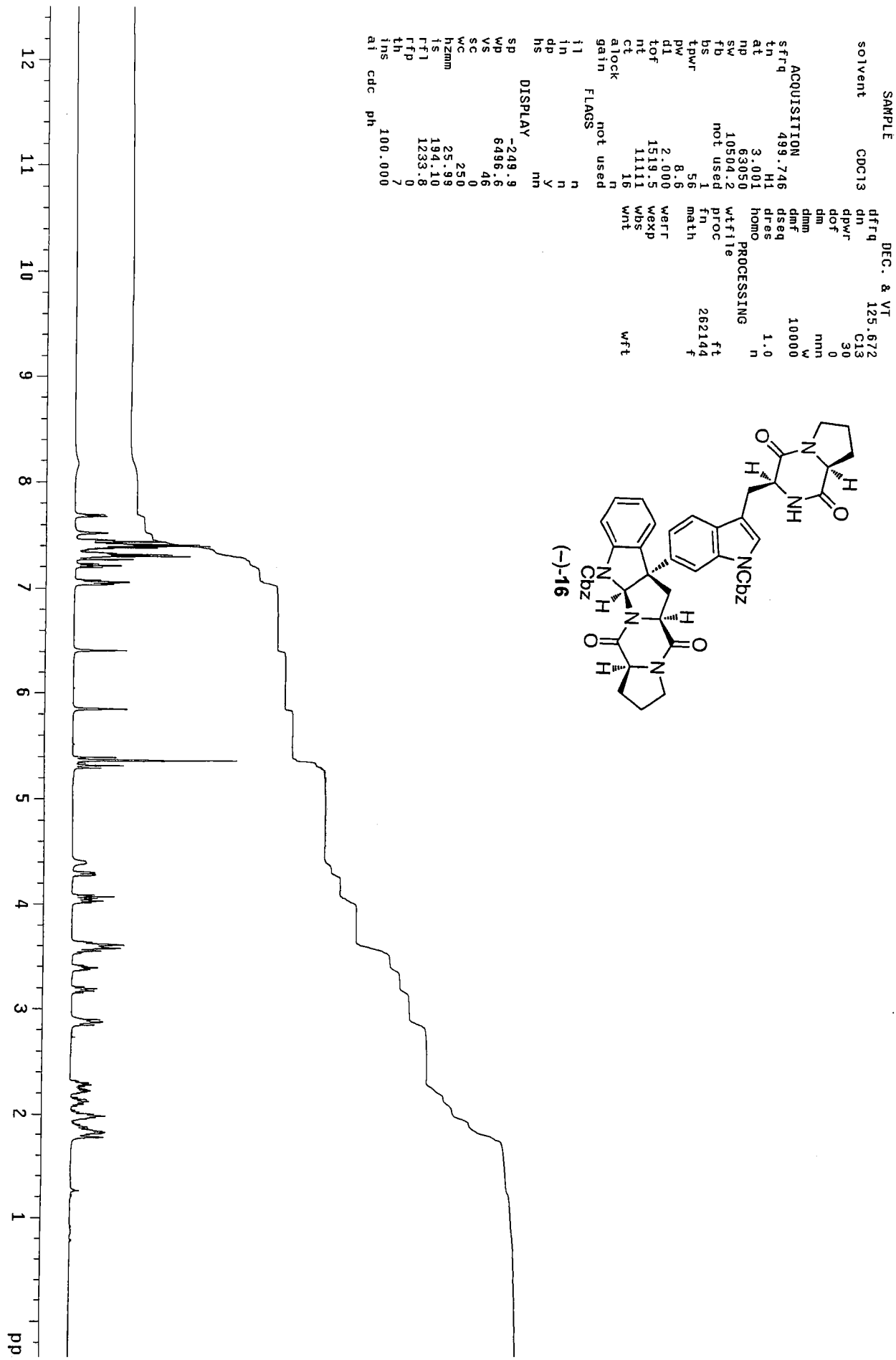
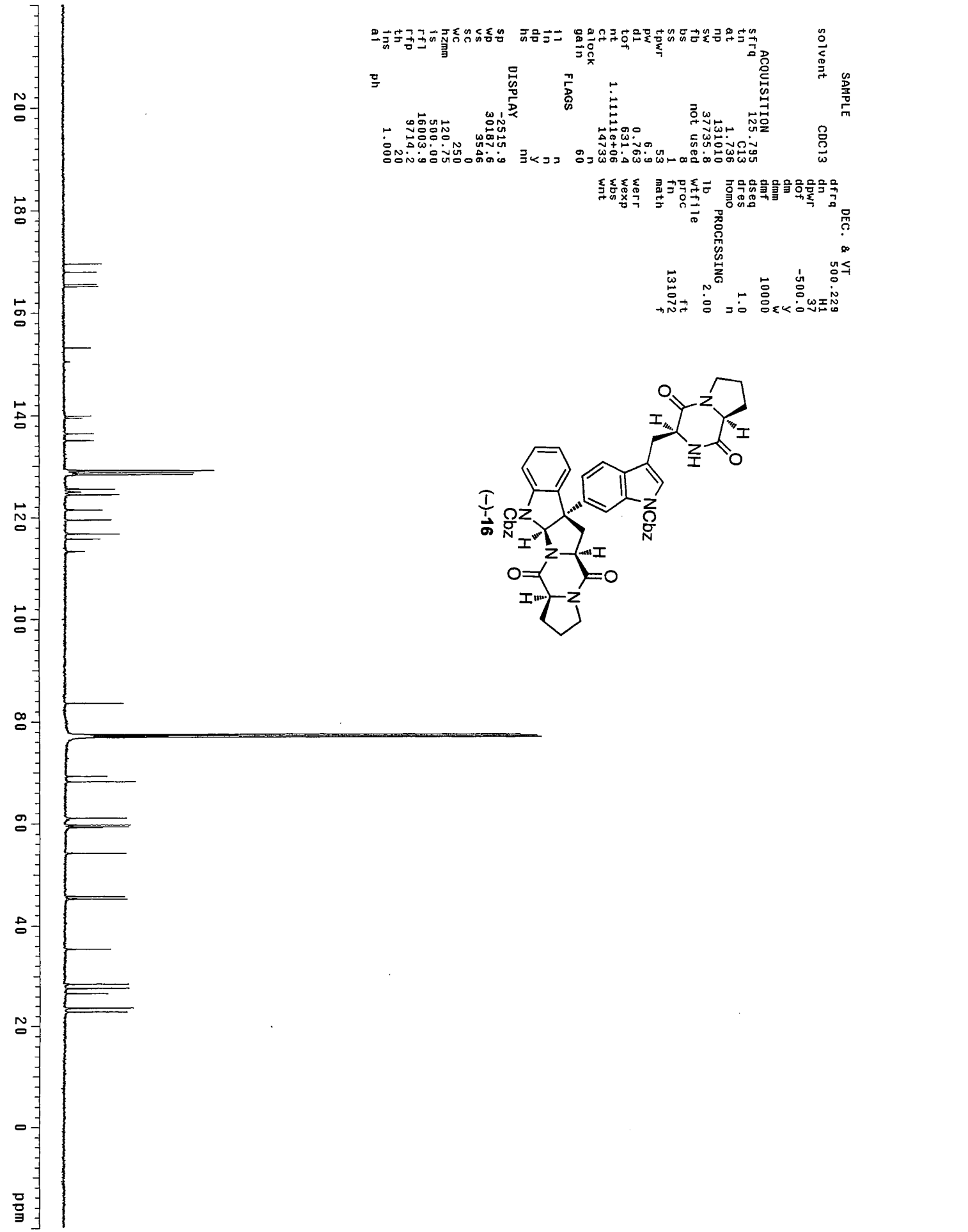
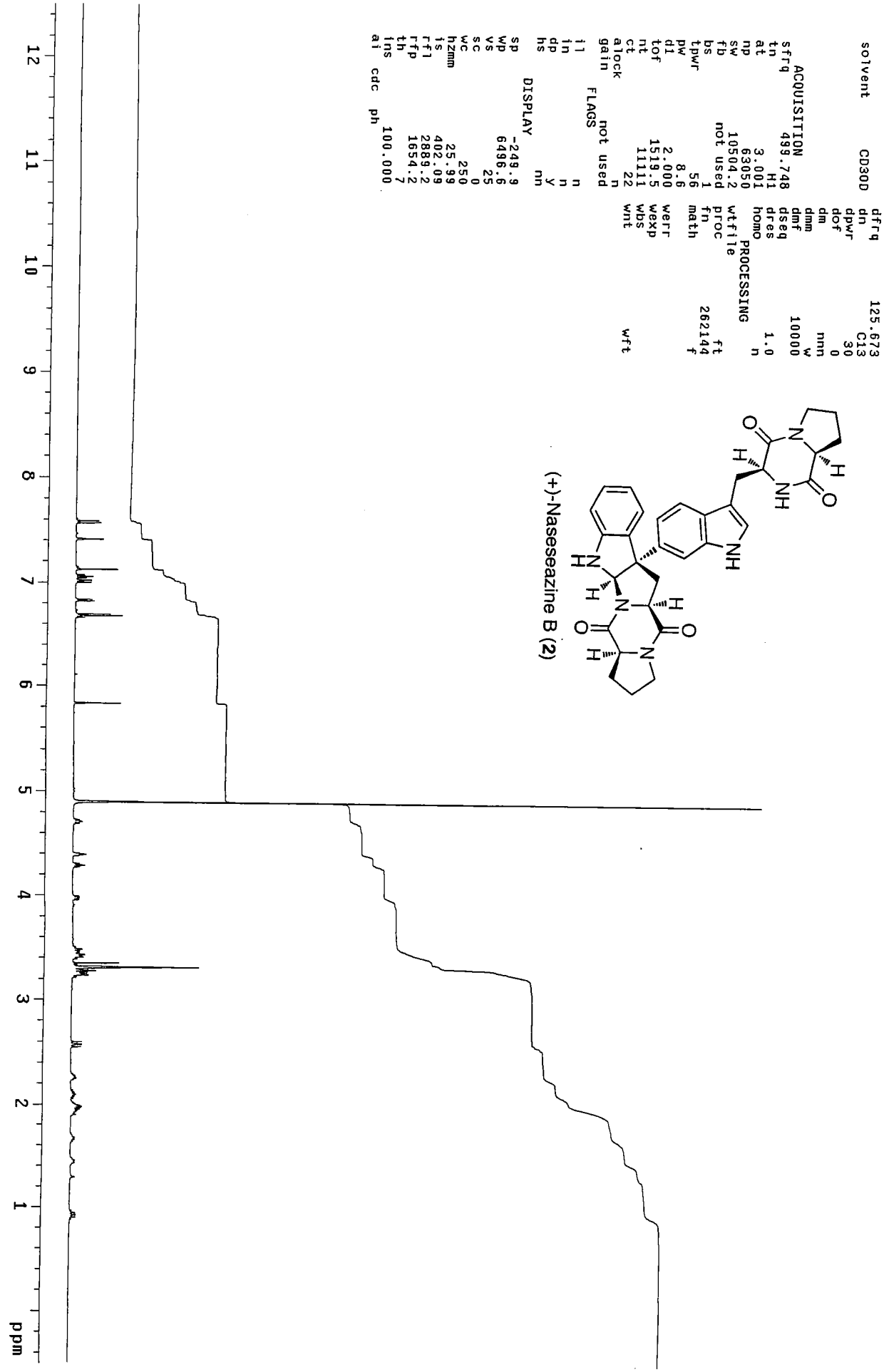
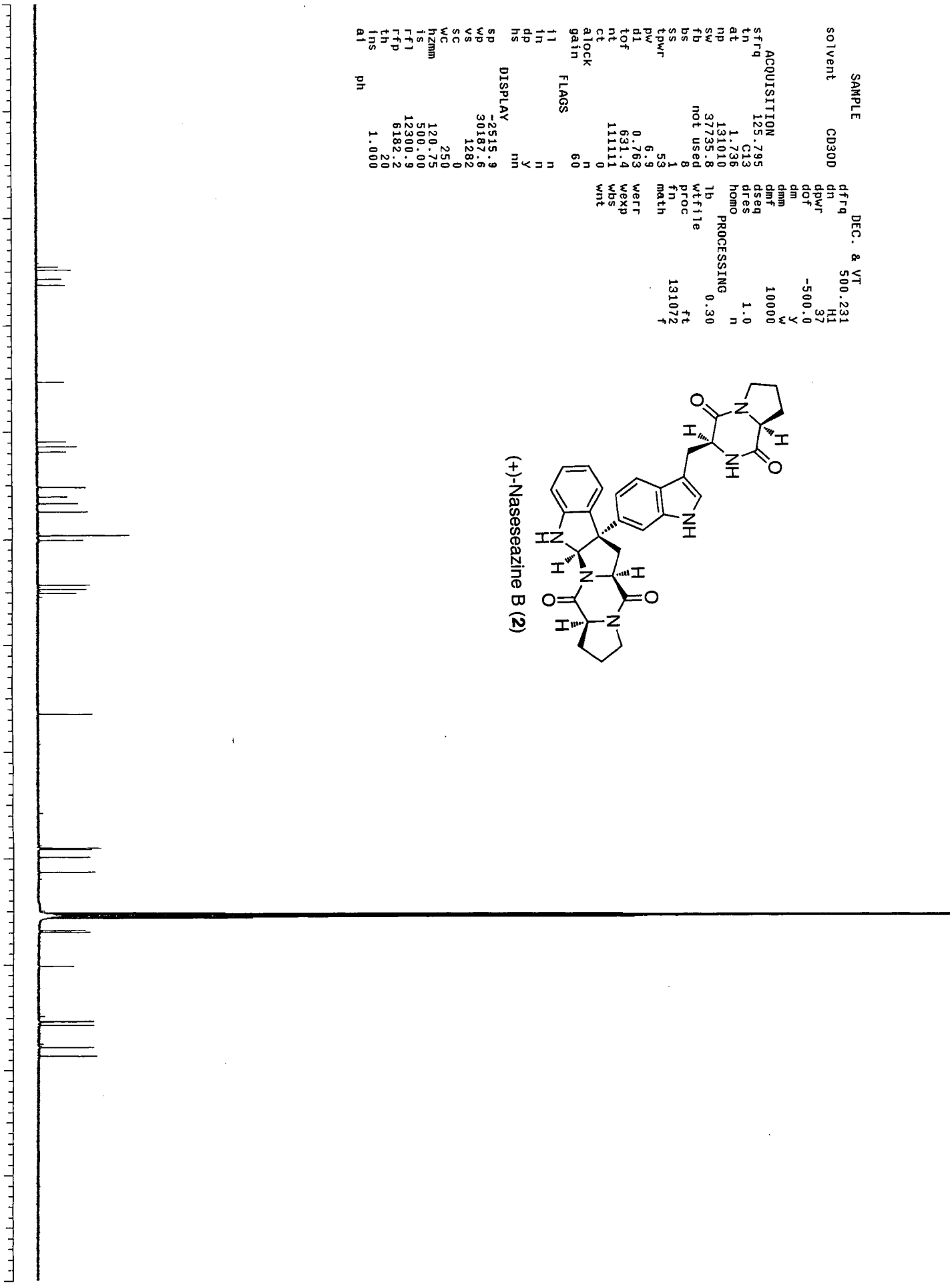
160
140
120
100
80
60
40
20
0
ppm

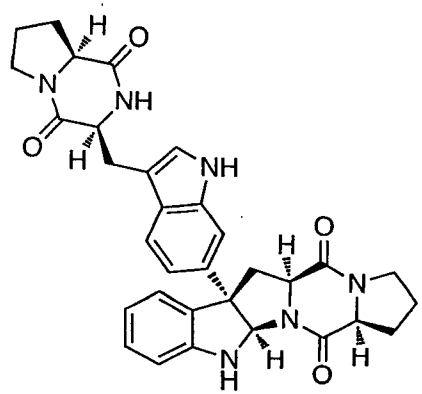

SAMPLE
solvent CDCl3
ACQUISITION
sfrq 499.746
tn H1
at 3.001
np 63050
sw 10504.2
fb not used
bs 1
tpwr 56
pw 8.6
d1 2.000
tof 1519.5
nt 11111
ct 16
alock wnt
gain n
FLAGS
not used
il n
in n
dp y
hs nn
DISPLAY
sp -249.9
wp 6496.6
vs 46
sc 0
wc 250
hzmm 25.99
is 194.10
rfl 1233.8
rfp 0
th 7
ins 100.000
al cdc ph
DEC. & VT
dfrq 125.672
dn C13
dpwr 30
dof 0
dm nnn
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 262144
math f
werr
wexp
wbs
wnt wft
(−)-16
NCbz
Cbz
NH
H
O
N
12
11
10
9
8
7
6
5
4
3
2
1
pp


SAMPLE
solvent CDCl3
ACQUISITION
sfrq 125.795
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 1.11111e+06
ct 14733
alock n
gain 60
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -2515.9
wp 30187.6
vs 3546
sc 0
wc 250
hzmm 120.75
is 500.00
rfl 16003.9
rfp 9714.2
th 20
ins 1.000
al
ph
DEC. & VT
dfrq 500.229
dn H1
dpwr 37
dof -500.0
dm y
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
lb 2.00
wtfile
proc
ft
fn 131072
math f
werr
wexp
wbs
wnt
NCbz
Cbz
(–)-16
200
180
160
140
120
100
80
60
40
20
0
ppm

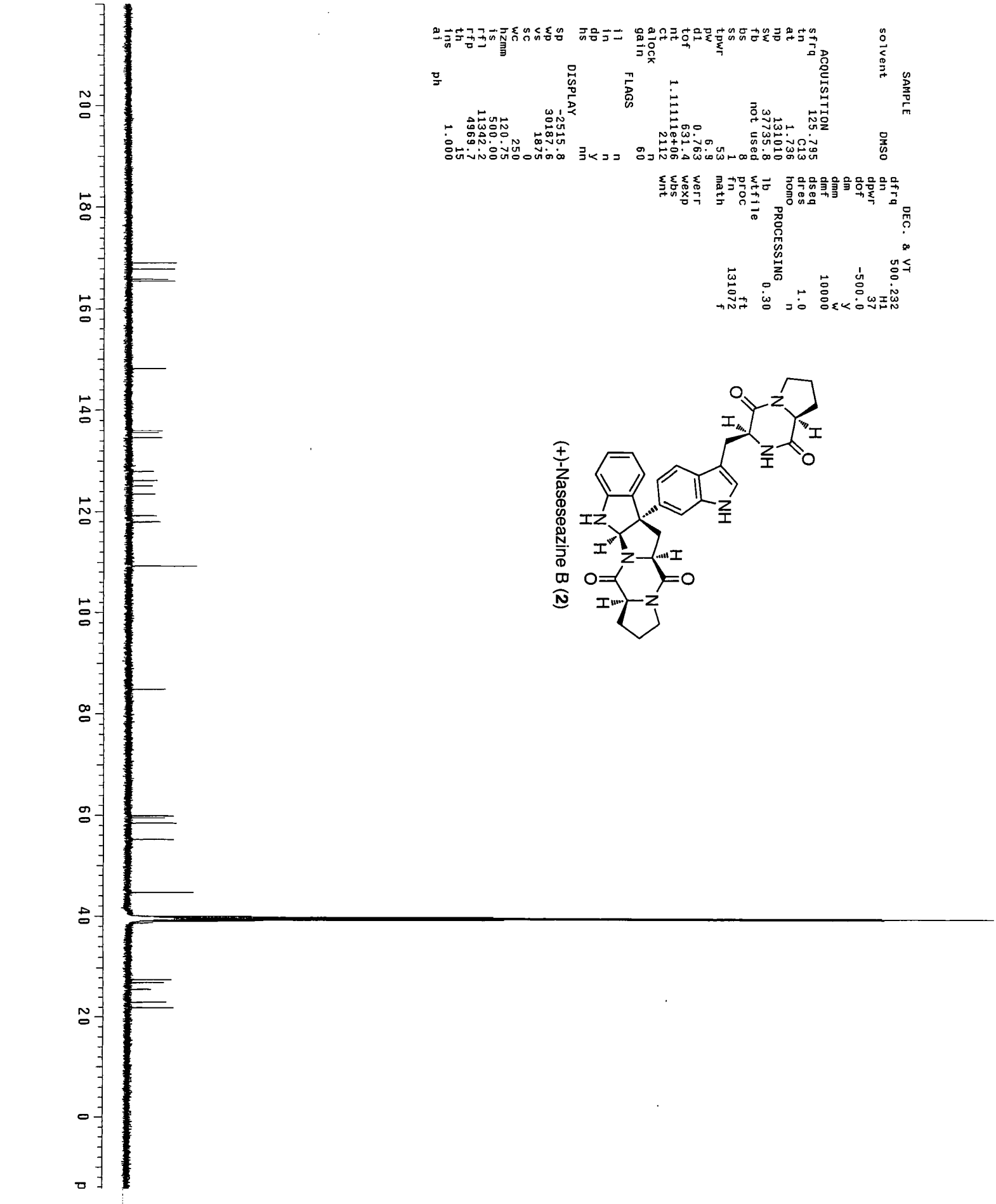

SAMPLE
solvent CD3OD
ACQUISITION
sfrq 499.748
tn H1
at 3.001
np 63050
sw 10504.2
fb not used
bs 1
tpwr 56
pw 8.6
d1 2.000
tof 1519.5
nt 11111
ct 22
alock n
gain n
FLAGS
not used
il n
in n
dp y
hs nn
DISPLAY
sp -249.9
wp 6496.6
vs 25
sc 0
wc 250
hzmm 25.99
is 402.09
rfl 2889.2
rfp 1654.2
th 7
ins 7
ai cdc ph 100.000
dfrq
dn
dpwr
dof
dm
dmm
dmf
dseq
dres
homo
PROCESSING
wtfile
proc
fn
math
werr
wexp
wbs
wnt
DEC. & VT 125.673
C13
30
0
nnn
w 10000
1.0
n
ft
262144
f
wft
(+)-Naseseazine B (2)
12
11
10
9
8
7
6
5
4
3
2
1
ppm

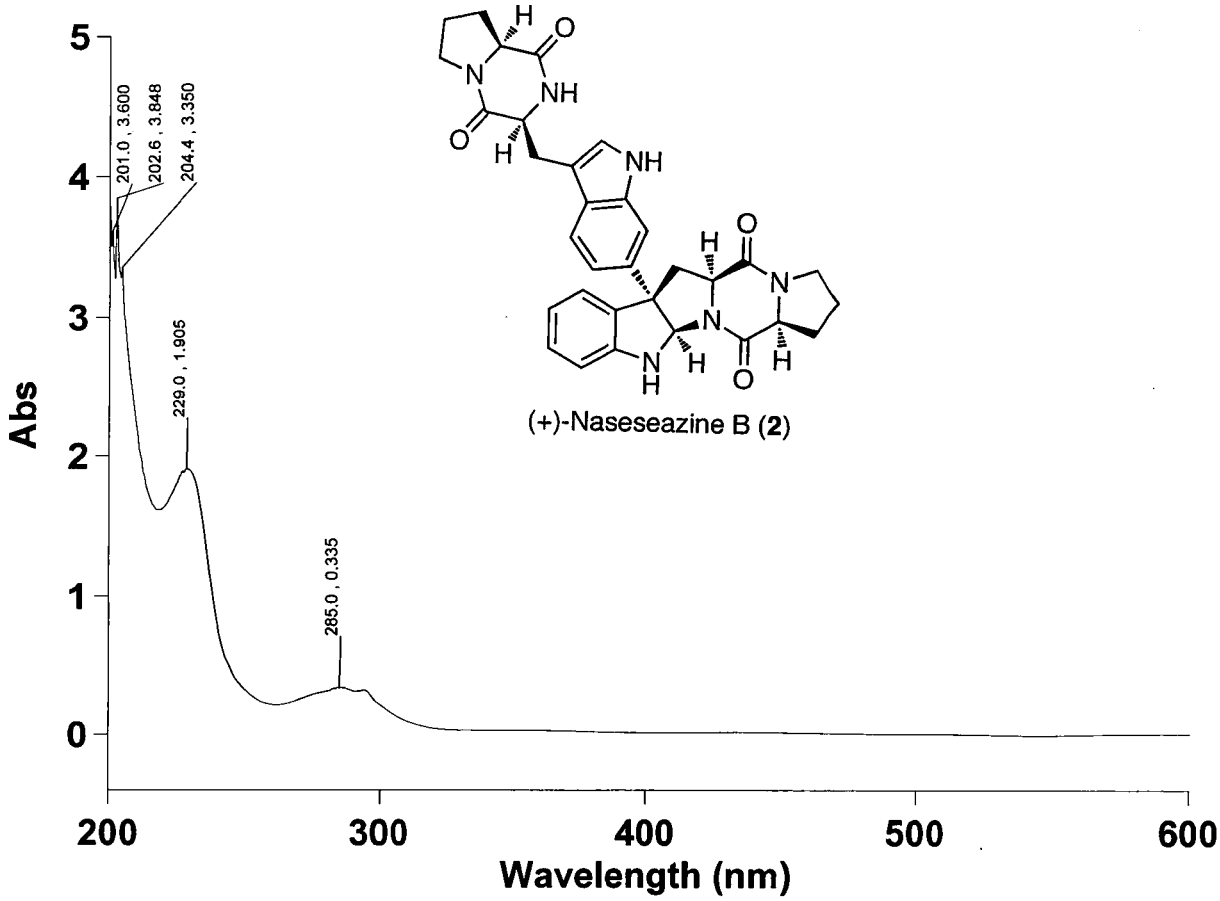

SAMPLE
solvent CD3OD
ACQUISITION
sfrq 125.795
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 111111
ct 0
alock n
gain 60
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -2515.9
wp 30187.6
vs 1282
sc 0
wc 250
hzmm 120.75
is 500.00
rfl 12300.9
rfp 6182.2
th 20
ins 1.000
ai ph
DEC. & VT
dfrq 500.231
dn H1
dpwr 37
dof -500.0
dm y
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
lb 0.30
wtfile
proc
fn 131072
math f
werr
wexp
wbs
wnt
(+)-Naseseazine B (2)
200
180
160
140
120
100
80
60
40
20
0
ppm

SAMPLE		DEC. & VT	
solvent	DMSO	dfrq	125.845
		dn	C13
		dpwr	30
		dof	0
		dm	nnn
		dmm	c
ACQUISITION		dmf	200
sfrq	500.433	dseq	
tn	H1	dres	1.0
at	4.999	homo	n
np	120102	PROCESSING	
sw	12012.0	wtfile	
fb	not used	proc	ft
bs	1	fn	262144
tpwr	60	math	f
pw	8.0		
d1	0.100	werr	
tof	3003.2	wexp	
nt	11111	wbs	
ct	10	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.2		
wp	6505.6		
vs	21		
sc	0		
wc	250		
hzmm	26.02		
is	370.90		
rfl	1762.8		
rfp	1251.1		
th	7		
ins	100.000		
ai cdc ph			



(+)-Naseseazine B (**2**)


SAMPLE
solvent DMSO
ACQUISITION
sfrq 125.795
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs 8
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 1.11111e+06
ct 2112
alock n
gain 60
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -2515.8
wp 30187.6
vs 1875
sc 0
wc 250
hzmm 120.75
is 500.00
rfl 11342.2
rfp 4969.7
th 15
ins 1.000
ai ph
DEC. & VT
dfrq 500.232
dn H1
dpwr 37
dof -500.0
dm y
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
lb 0.30
wtfile
proc
fn ft131072
math f
wbs
wnt
(+)-Naseseazine B (2)
200
180
160
140
120
100
80
60
40
20
0
p



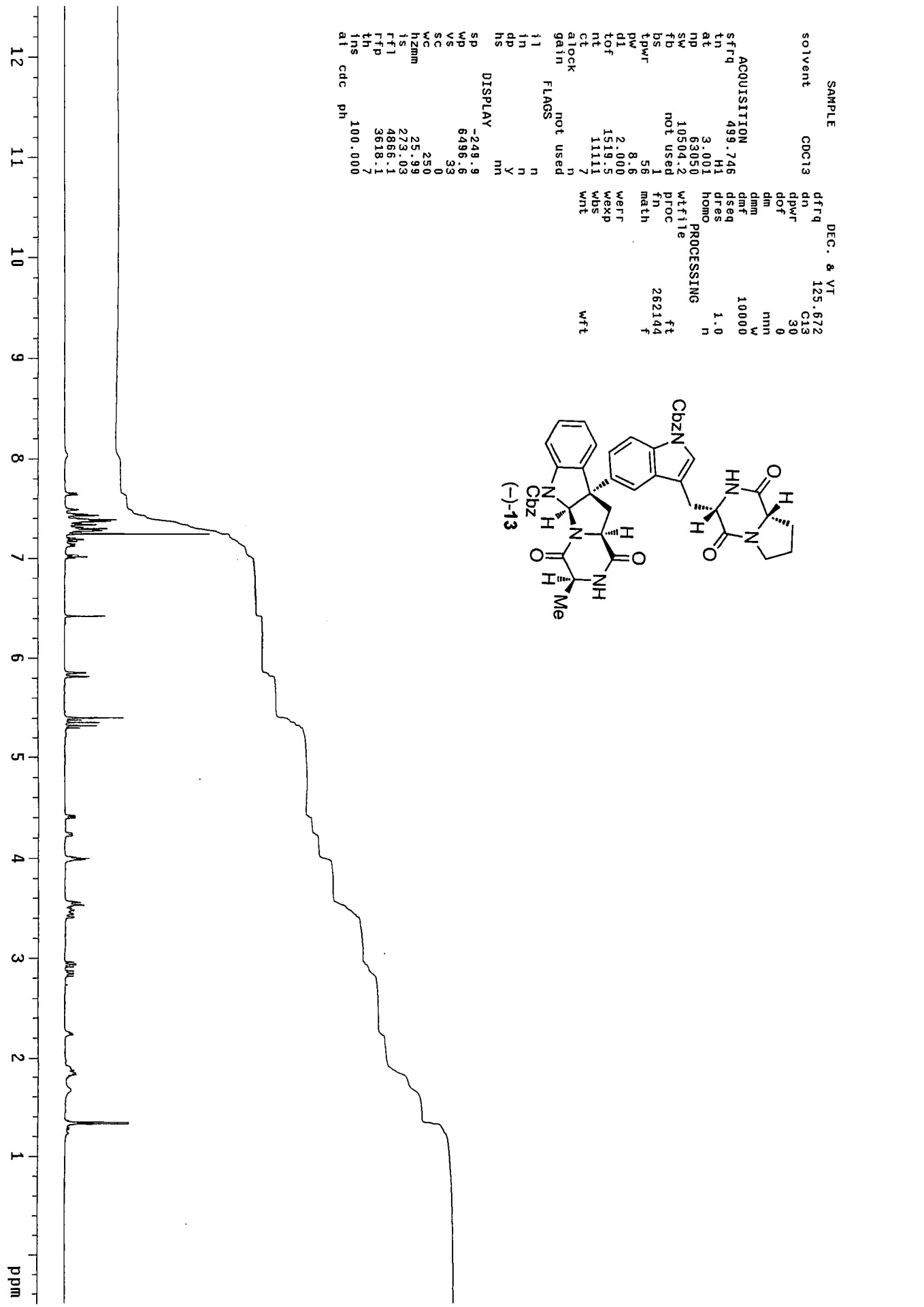
Scan Analysis Report

Sample Name: Naseseazine B

Peak Table

Peak Style	Peaks
Peak Threshold	0.0100
Range	600.0nm to 200.0nm

Wavelength (nm)	Abs
285.0	0.335
229.0	1.905
204.4	3.350
202.6	3.848
201.0	3.600


SAMPLE
solvent CDCl3
ACQUISITION
sfrq 499.746
tn H1
at 3.001
np 63050
sw 10504.2
fb not used
bs 1
tpwr 56
pw 8.6
d1 2.000
tof 1519.5
nt 11111
ct 7
alock n
gain n
FLAGS
not used
il n
in n
dp y
hs nn
DISPLAY
sp -249.9
wp 6496.6
vs 33
sc 0
wc 250
hzmm 25.99
is 273.03
rfl 4866.1
rfp 3618.1
th 7
ins 100.000
ai cdc ph
dfrq DEC. & VT
dn C13
dpwr 30
dof 0
dm nnn
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 262144
math f
werr
wexp
wbs
wnt wft
125.672
CbzN
HN
N
Cbz
NH
Me
(−)-13
12
11
10
9
8
7
6
5
4
3
2
1
ppm

SAMPLE	
solvent	CDCl3

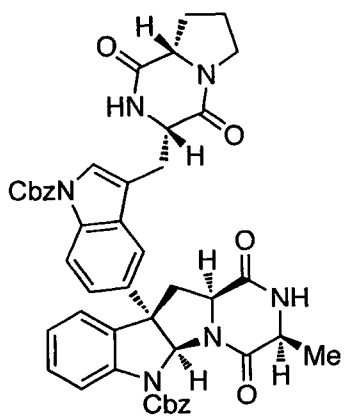
ACQUISITION	
sfrq	125.795
tn	C13
at	1.736
np	131010
sw	37735.8
fb	not used
bs	8
ss	1
tpwr	53
pw	6.9
d1	0.763
tof	631.4
nt	1.11111e+07
ct	1880
alock	n
gain	60

FLAGS	
il	n
in	n
dp	y
hs	nn

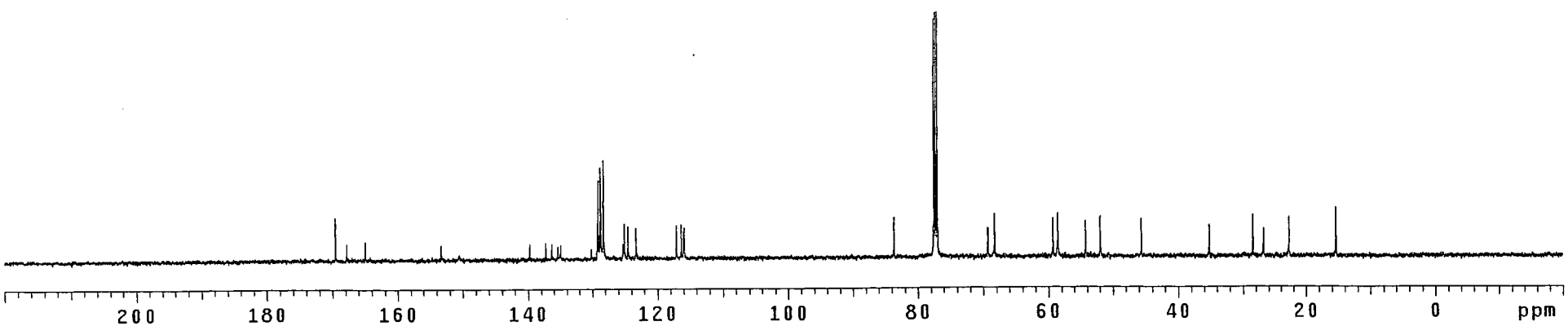
DISPLAY	
sp	-2515.9
wp	30187.6
vs	2083
sc	0
wc	250
hzmm	120.75
is	500.00
rfl	16003.9
rfp	9714.2
th	5
ins	1.000
ai	ph

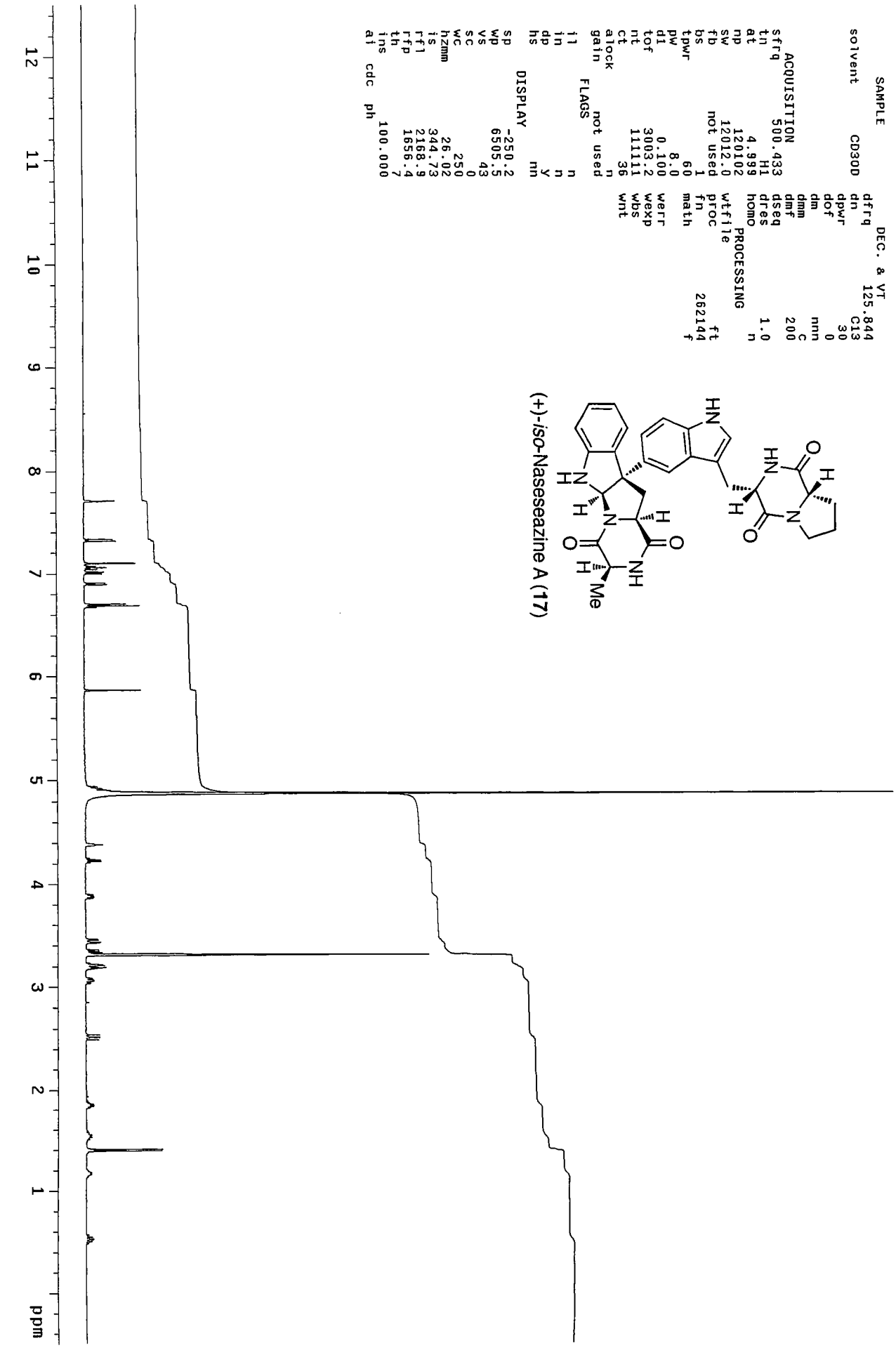
DEC. & VT	
dfrq	500.229
dn	H1
dpwr	37
dof	-500.0
dm	y
dmm	w
dmf	10000
dseq	
dres	1.0
homo	n

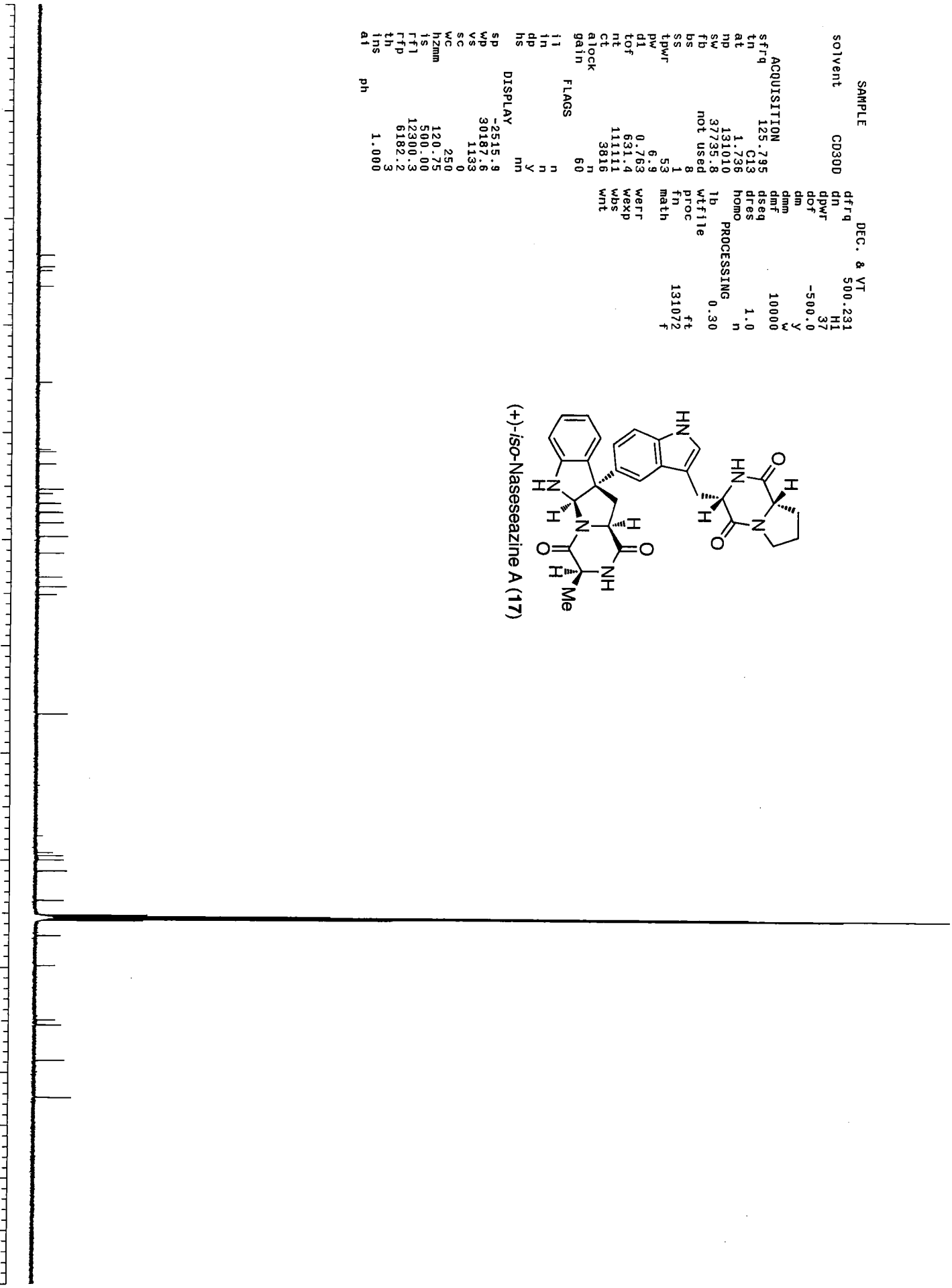
PROCESSING	
lb	2.00
wtfile	
proc	ft
fn	131072
math	f
werr	
wexp	
wbs	
wnt	

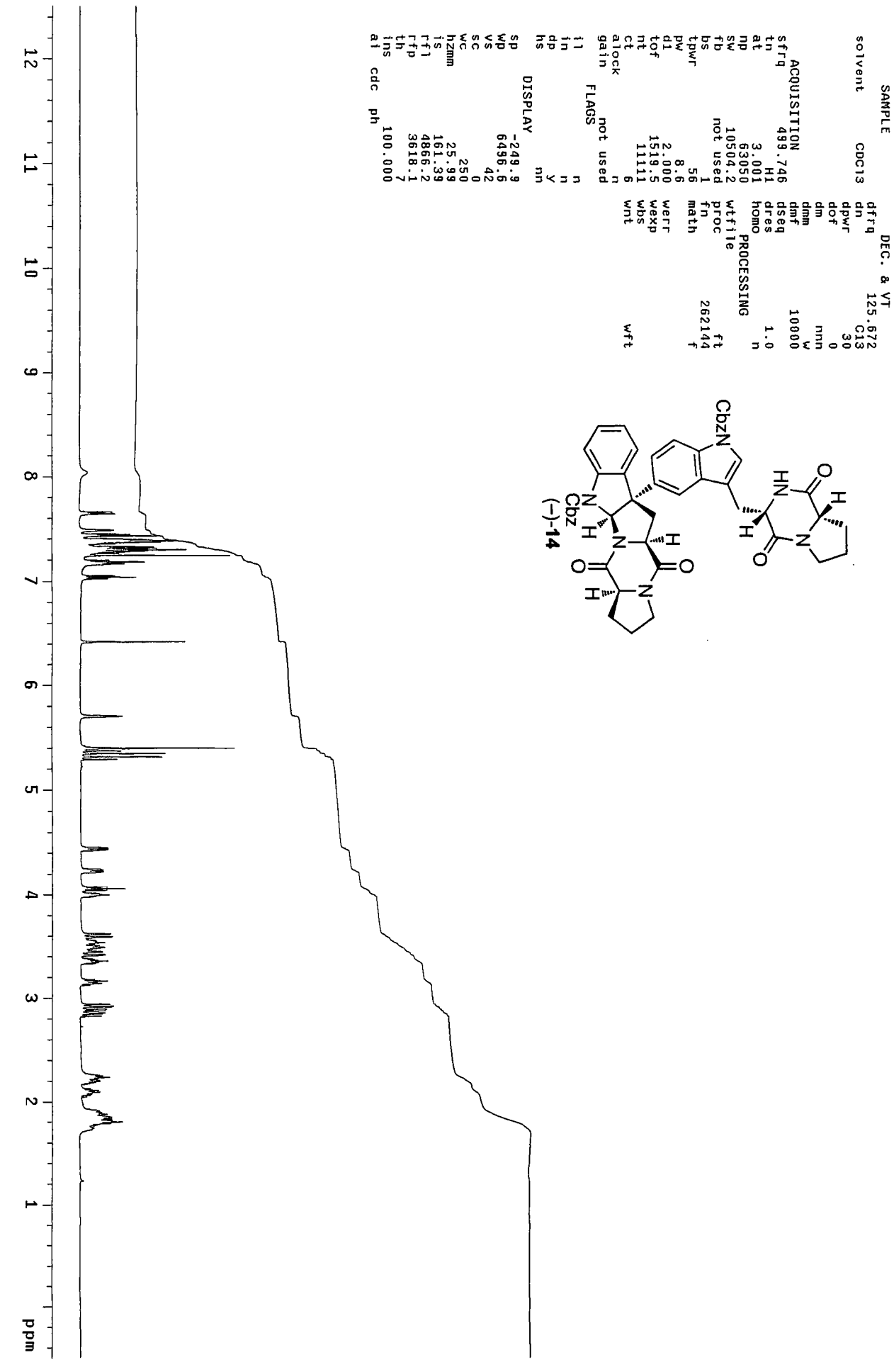


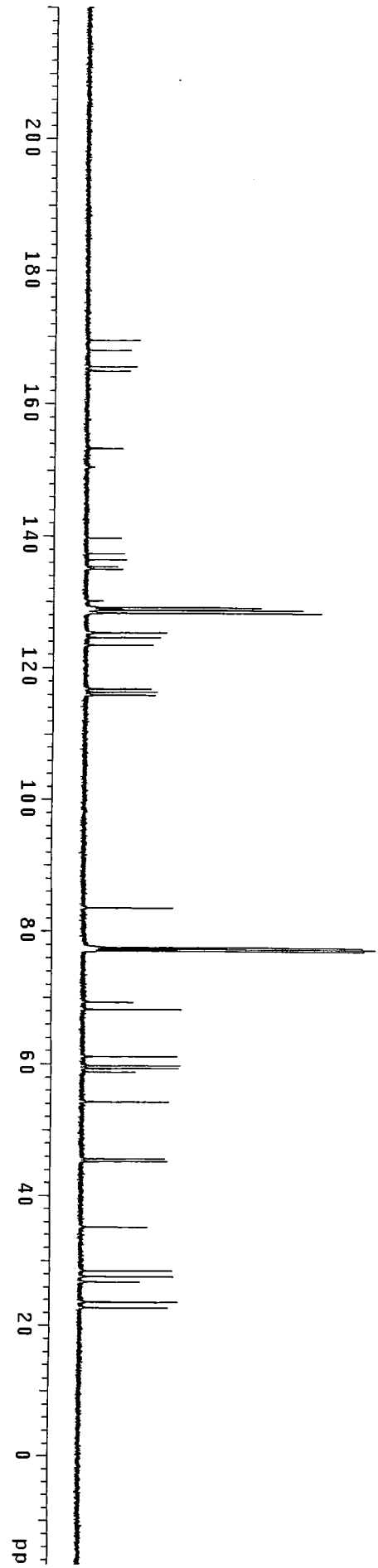
(–)-13



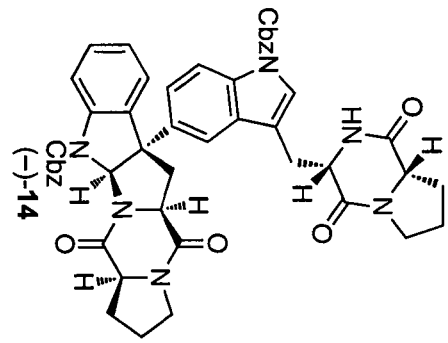

SAMPLE
solvent CD3OD
ACQUISITION
sfrq 500.433
tn H1
at 4.999
np 120102
sw 12012.0
fb not used
bs 1
tpwr 60
pw 8.0
d1 0.100
tof 3003.2
nt 111111
ct 36
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 6505.5
vs 43
sc 0
wc 250
hzmm 26.02
is 344.73
rfl 2168.9
rfp 1656.4
th 7
ins 100.000
ai cdc ph
dfrq
dn C13
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 262144
math f
werr
wexp
wbs
wnt
DEC. & VT
125.844
(+)-iso-Naseseazine A (17)
12
11
10
9
8
7
6
5
4
3
2
1
ppm

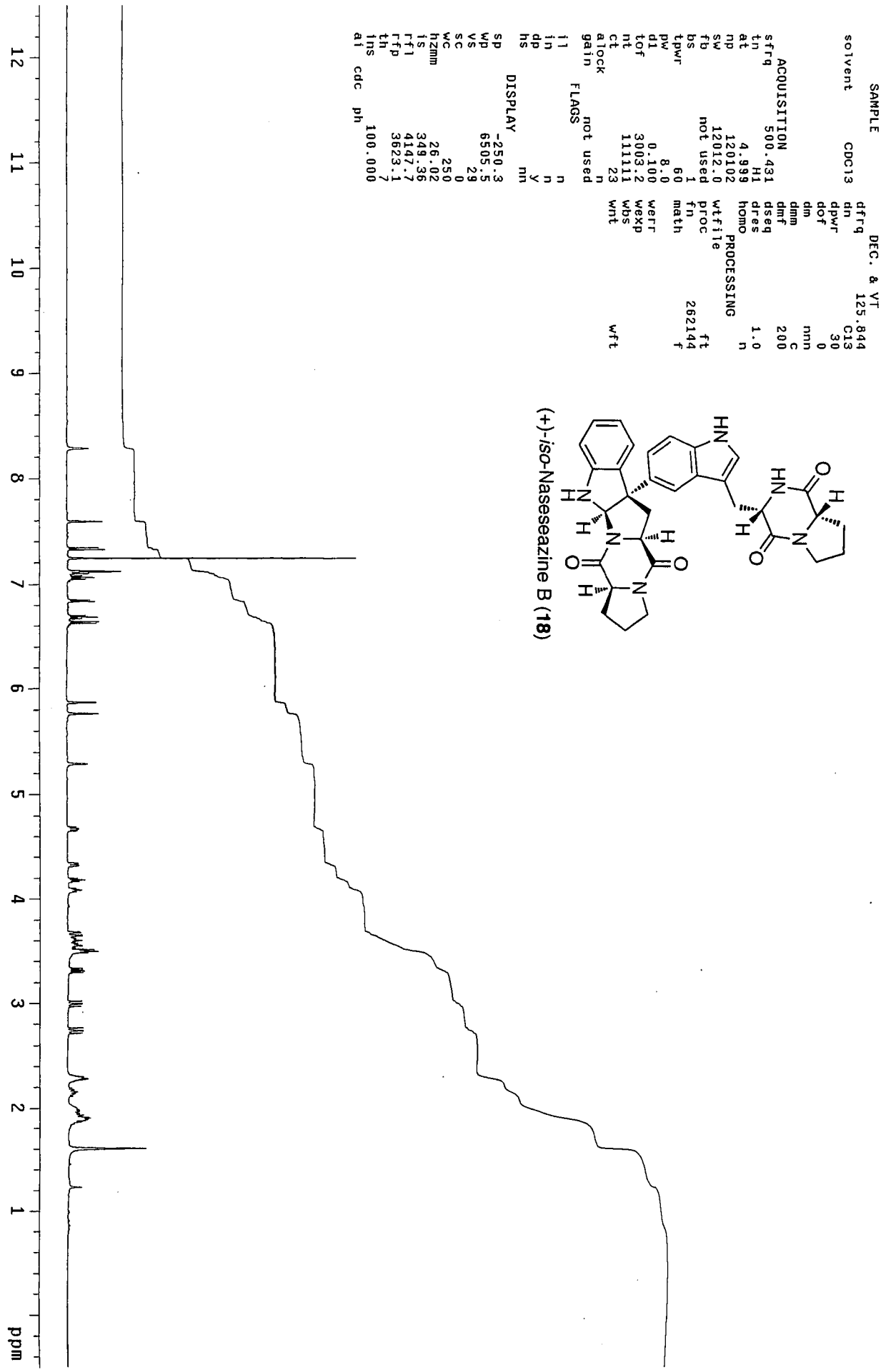

SAMPLE
solvent CD3OD
ACQUISITION
sfrq 125.795
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs 8
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 111111
ct 3816
alock n
gain 60
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -2515.9
wp 30187.6
vs 1133
sc 0
wc 250
hzmm 120.75
is 500.00
rfl 12300.3
rfp 6182.2
th 3
ins 1.000
ai ph
DEC. & VT
dfrq 500.231
dn H1
dpwr 37
dof -500.0
dm y
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
lb 0.30
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
HN
HN
O
H
N
O
H
N
H
N
H
O
H
O
NH
H
Me
(+)-iso-Naseseazine A (17)
200
180
160
140
120
100
80
60
40
20
0
ppm

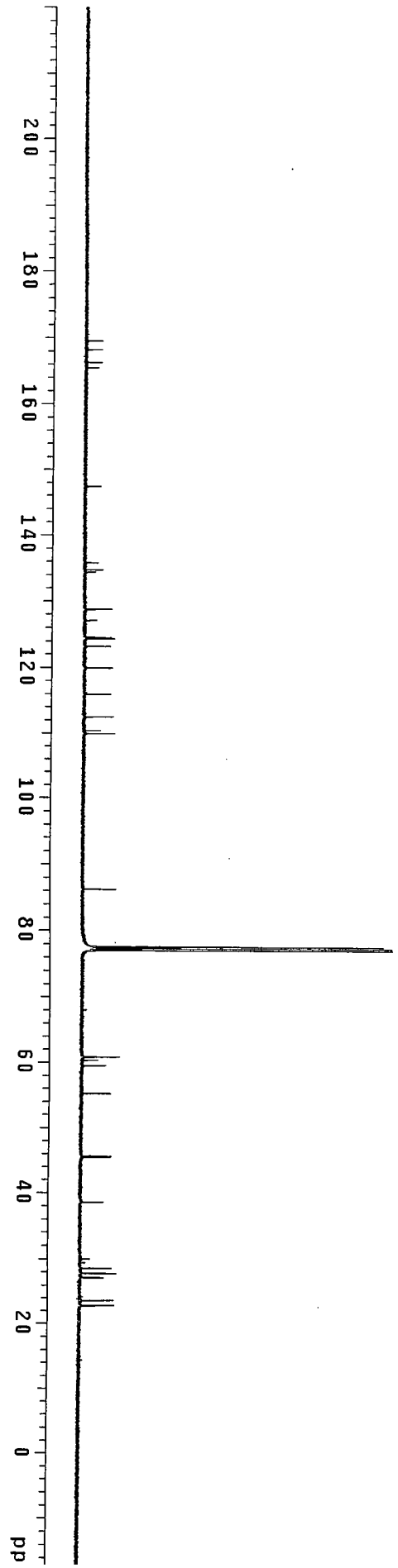

SAMPLE
solvent CDCl3
ACQUISITION
sfrq 499.746
tn H1
at 3.001
np 63050
sw 10504.2
fb
bs 1
tpwr 56
pw 8.6
d1 2.000
tof 1519.5
nt 11111
ct 6
alock wft
gain n
FLAGS
not used
il n
in n
dp y
hs nn
DISPLAY
sp -249.9
wp 6496.6
vs 42
sc 0
wc 250
hzmm 25.99
is 161.39
rfl 4866.2
rfp 3618.1
th 7
ins 100.000
al cdc ph
DEC. & VT
dfrq 125.672
dn C13
dpwr 30
dof 0
dm nnn
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 262144
math f
werr
wexp
wbs
wnt wft
CbzN
HN
NH
Cbz
(–)-14
12
11
10
9
8
7
6
5
4
3
2
1
ppm



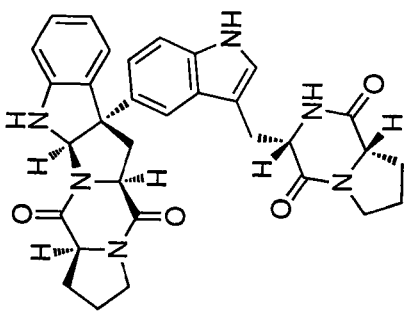

```
                SAMPLE                          DEC. & VT
solvent         CDCl3           dfrq            500.229
                                dn                  H1
                                dpwr                37
                                dof              -500.0
                                dm                   y
   ACQUISITION                  dmm                  w
sfrq          125.795           dmf              10000
tn                C13           dseq                 1
at              1.736           homo                 n
np             131010                PROCESSING
sw            37735.8           lb                1.00
fb       not used               wtfile
bs                8              proc                ft
ss                1              fn              131072
tpwr             53              math                f
pw              6.9
d1            0.763             werr
tof           631.4             wexp
nt           111111             wbs
ct             1992             wnt
alock             n
gain             60
                FLAGS
tl                n
in                n
dp                y
hs               nn
               DISPLAY
sp            -2515.9
wp            30187.6
vs               1495
sc                  0
wc                250
hzmm           120.75
is             500.00
rfl           16006.2
rfp            9714.2
th                 11
ins                11
al          1.000
   ph
```



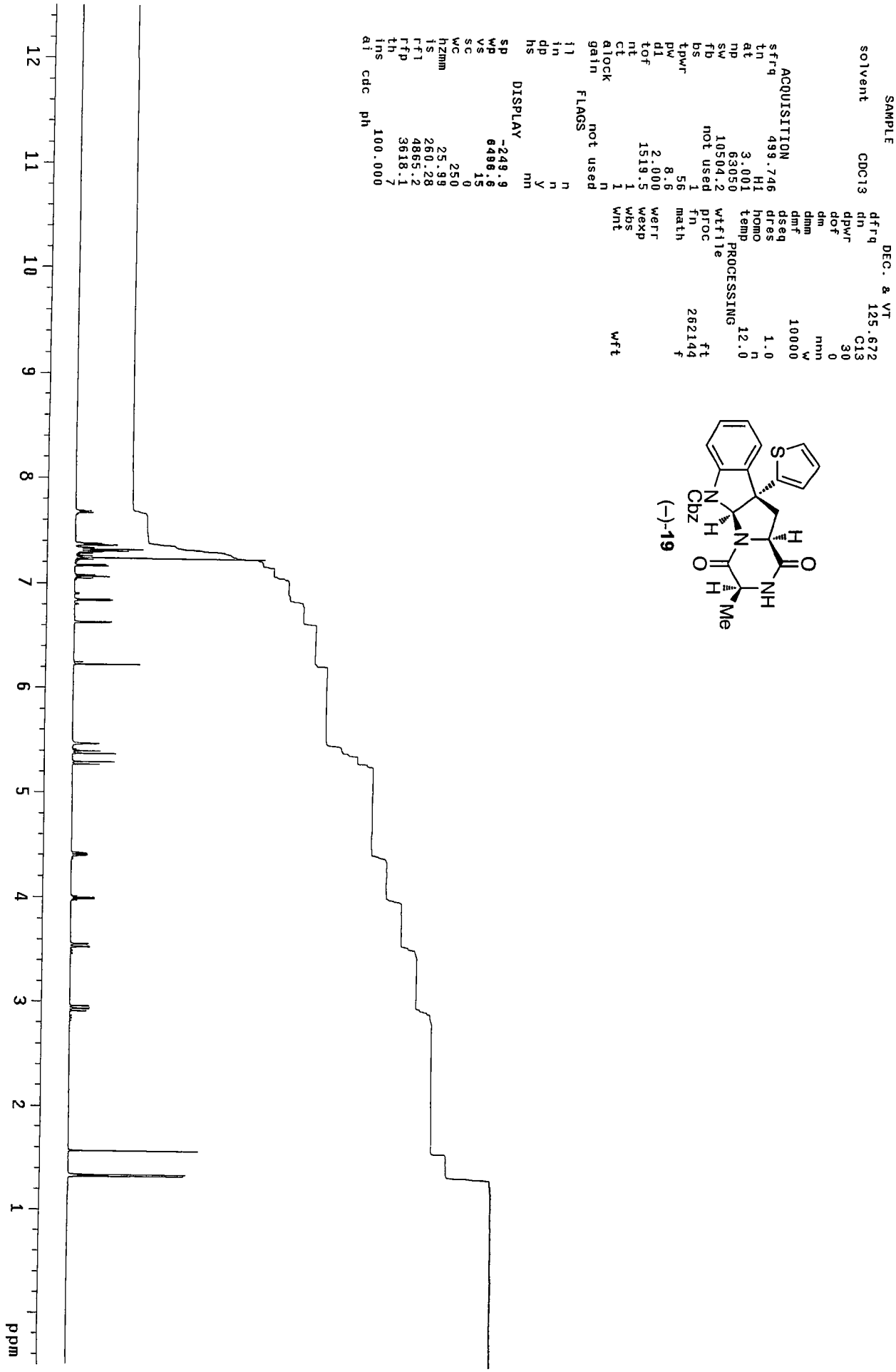

SAMPLE
solvent CDCl3
ACQUISITION
sfrq 500.431
tn H1
at 4.999
np 120102
sw 12012.0
fb not used
bs 1
tpwr 60
pw 8.0
d1 0.100
tof 3003.2
nt 111111
ct 23
alock n
gain n
FLAGS
not used
il n
in n
dp y
hs nn
DISPLAY
sp -250.3
wp 6505.5
vs 29
sc 0
wc 250
hzmm 26.02
is 349.36
rfl 4147.7
rfp 3823.1
th 7
ins 100.000
ai cdc ph
DEC. & VT
dfrq 125.844
dn C13
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 262144
math f
werr
wexp
wbs
wnt wft
(+)-iso-Naseseazine B (18)
12
11
10
9
8
7
6
5
4
3
2
1
ppm



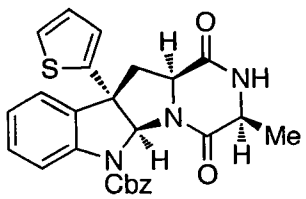

```
                SAMPLE                          DEC. & VT
solvent         CDCl3           dfrq        500.229
                                dn              H1
                                dpwr            37
                                dof          -500.0
                                dm              y
                                dmm             w
   ACQUISITION                  dmf         10000
sfrq          125.795           dseq
tn                C13           dres            1.0
at              1.736           homo            n
np             131010                PROCESSING
sw          37735.8             lb              0.30
fb          not used            wtfile
bs                8             proc
ss                1             fn          131072
tpwr             53             math            f
pw              6.9
d1            0.763             werr
tof           631.4             wexp
nt      1.11111e+07             wbs
ct            17520             wnt
alock             n
gain             60         FLAGS
                            il              n
                            in              n
                            dp              y
                            hs              nn
                       DISPLAY
                sp         -2515.9
                wp         30187.6
                vs            1792
                sc               0
                wc             250
                hzmm        120.75
                is             500.00
                rfl         16002.2
                rfp          9714.2
                th                8
                ins               8
                ai            1.000
                ph
```



(+)-*iso*-Naseseazine B (**18**)


SAMPLE
solvent CDCl3
ACQUISITION
sfrq 499.746
tn H1
at 3.001
np 63050
sw 10504.2
fb not used
bs 1
tpwr 56
pw 8.6
d1 2.000
tof 1519.5
nt 1
ct 1
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -249.9
wp 6406.6
vs 15
sc 0
wc 250
hzmm 25.99
is 260.28
rfl 4865.2
rfp 3618.1
th 7
ins
ai cdc ph 100.000
DEC. & VT
dfrq 125.672
dn C13
dpwr 30
dof 0
dm nnn
dmm w
dmf 10000
dseq
dres 1.0
homo n
temp 12.0
PROCESSING
wtfile
proc ft
fn 262144
math f
werr
wexp
wbs
wnt wft
S
N
Cbz
H
N
H
O
O
NH
H
Me
(−)-19
12
11
10
9
8
7
6
5
4
3
2
1
ppm

SAMPLE		DEC. & VT	
solvent	CDCl3	dfrq	500.229
		dn	H1
		dpwr	37
		dof	-500.0
		dm	y
		dmm	w
ACQUISITION		dmf	10000
sfrq	125.795	dseq	
tn	C13	dres	1.0
at	1.736	homo	n
np	131010	PROCESSING	
sw	37735.8	lb	0.30
fb	not used	wtfile	
bs	8	proc	ft
ss	1	fn	131072
tpwr	53	math	f
pw	6.9		
d1	0.763	werr	
tof	631.4	wexp	
nt	11111	wbs	
ct	128	wnt	
alock	n		
gain	60		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-2515.9		
wp	30187.6		
vs	330		
sc	0		
wc	250		
hzmm	120.75		
is	500.00		
rfl	16006.2		
rfp	9714.2		
th	20		
ins	1.000		
ai ph			



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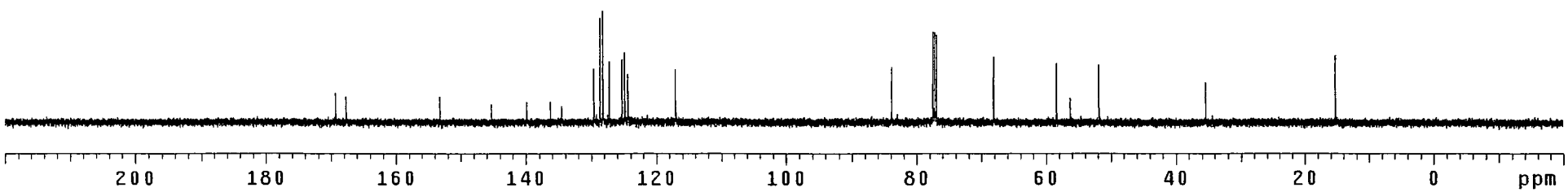
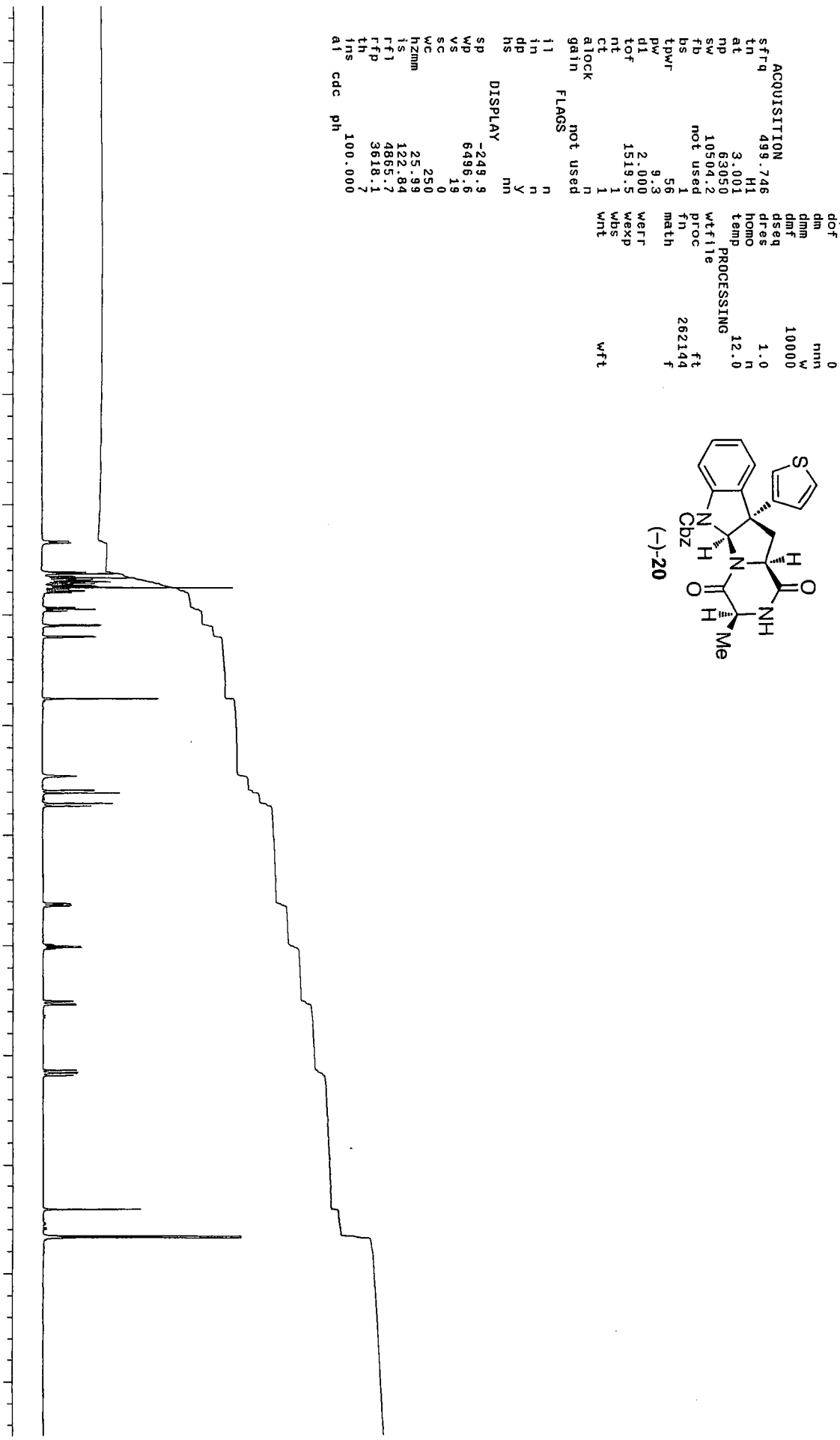
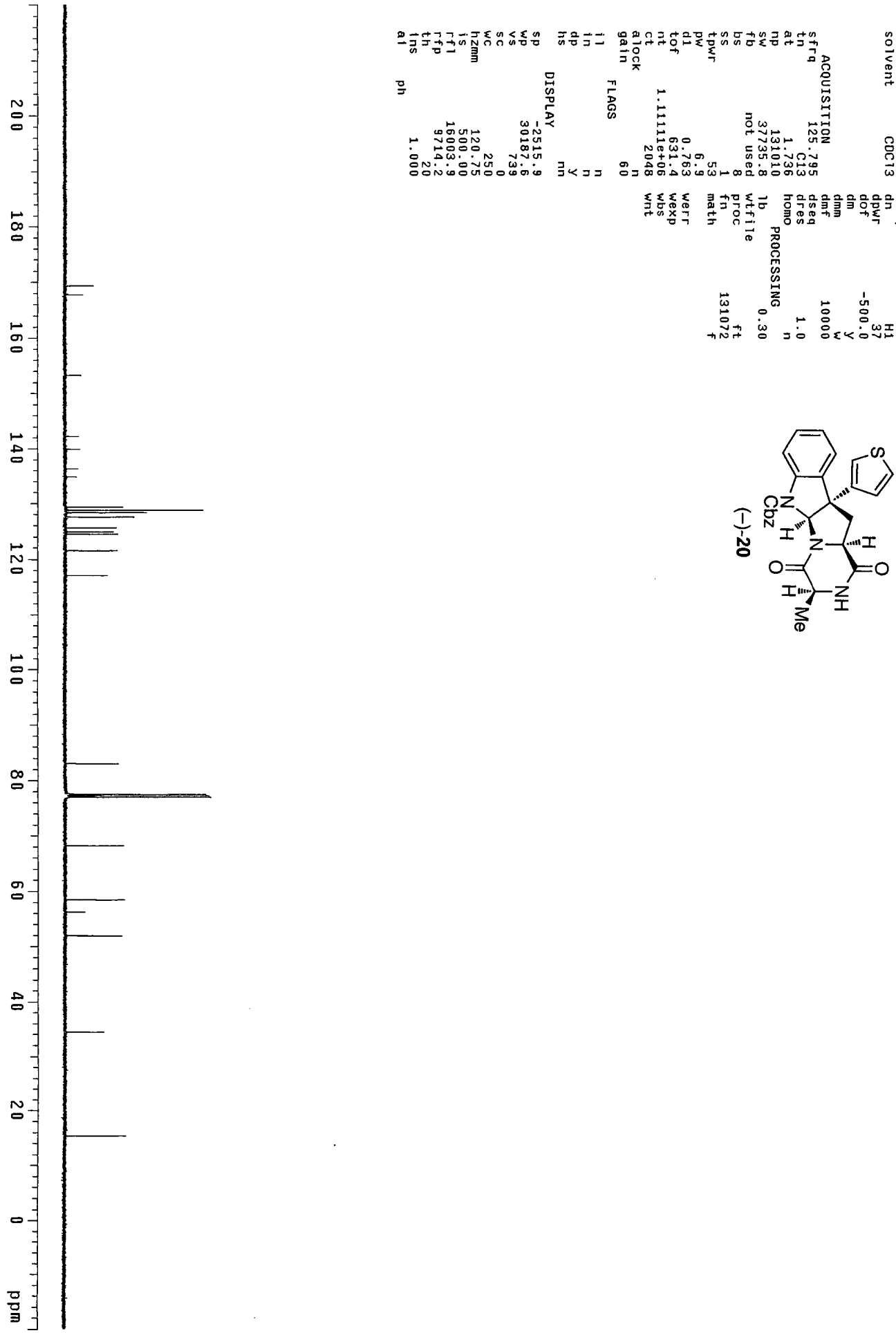
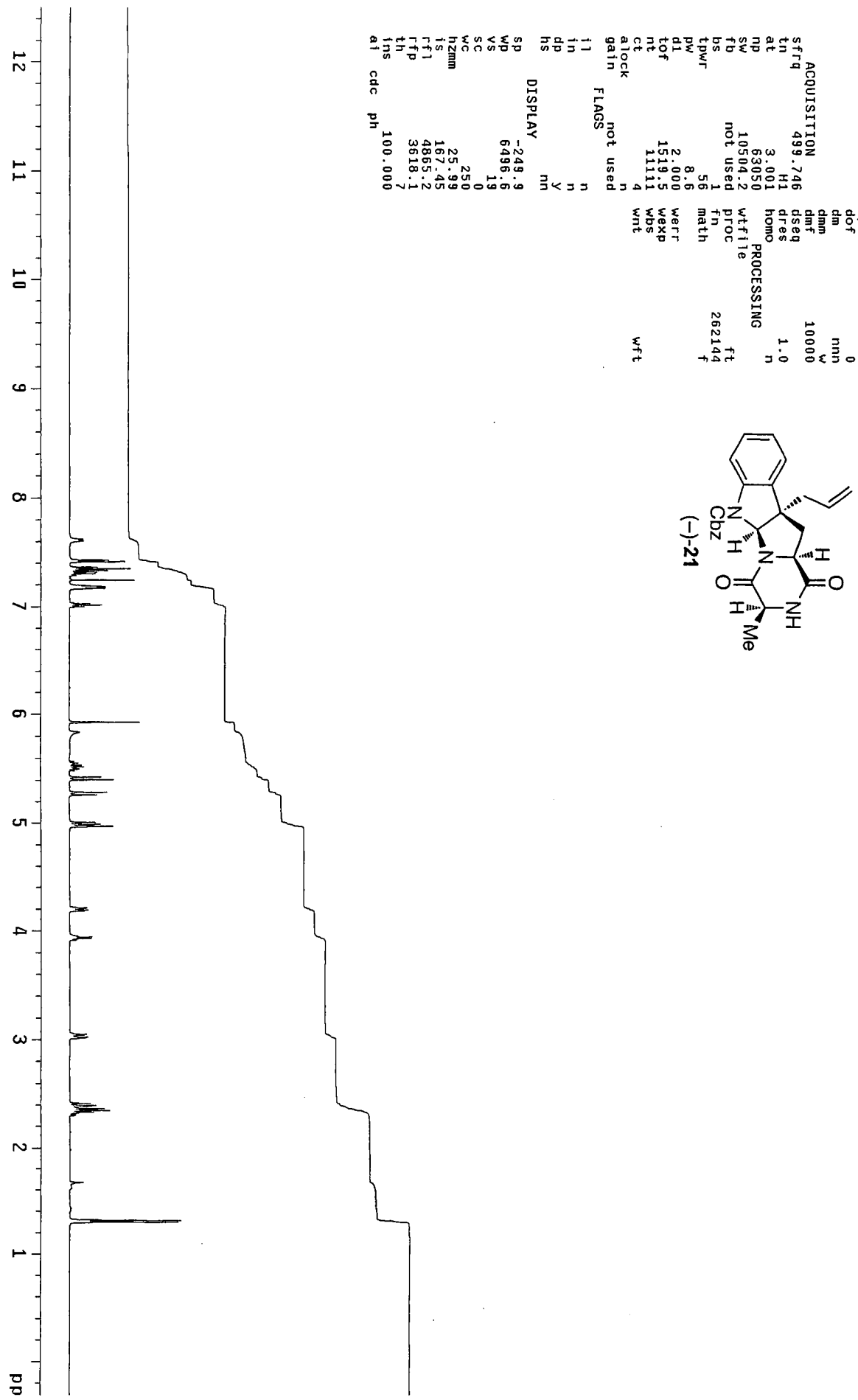
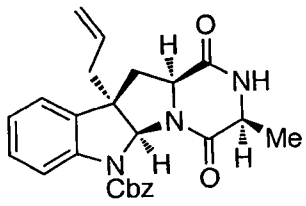



SAMPLE
solvent CDCl3
ACQUISITION
sfrq 499.746
tn H1
at 3.001
np 63050
sw 10504.2
fb not used
bs 1
tpwr 56
pw 9.3
d1 2.000
tof 1519.5
nt 1
ct 1
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -249.9
wp 6496.6
vs 19
sc 0
wc 250
hzmm 25.99
is 122.84
rfl 4865.7
rfp 3618.1
th 7
ins 100.000
al cdc ph
DEC. & VT
dfrq 125.672
dn C13
dpwr 30
dof 0
dm nnn
dmm w
dmf 10000
dseq
dres 1.0
homo n
temp 12.0
PROCESSING
wtfile
proc ft
fn 262144
math f
werr
wexp
wbs
wnt wft
(−)-20
Cbz
Me
ppm


SAMPLE
solvent CDCl3
ACQUISITION
sfrq 125.795
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs 8
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 1.11111e+06
ct 2048
alock wnt
gain n
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -2515.9
wp 30187.6
vs 739
sc 0
wc 250
hzmm 120.75
is 500.00
rfl 16003.9
rfp 9714.2
th 20
ins 1.000
ph
dfrq DEC. & VT
dn 500.229
dpwr H1
dof 37
dm -500.0
dmm y
dmf w
dseq 10000
dres 1.0
homo n
PROCESSING
lb 0.30
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
Cbz
(−)-20
Me
200
180
160
140
120
100
80
60
40
20
0
ppm


SAMPLE
solvent CDCl3
ACQUISITION
sfrq 499.746
tn H1
at 3.001
np 63050
sw 10504.2
fb not used
bs 1
tpwr 56
pw 8.6
d1 2.000
tof 1519.5
nt 11111
ct 4
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -249.9
wp 6496.6
vs 19
sc 0
wc 250
hzmm 25.99
is 167.45
rfl 4865.2
rfp 3618.1
th 7
ins 100.000
ai cdc ph
dfrq
dn
dpwr
dof
dm
dmm
dmf
dseq
dres
homo
PROCESSING
wtfile
proc
fn
math
werr
wexp
wbs
wnt
DEC. & VT
125.672
C13
30
0
nnn
w
10000
1.0
n
ft
262144
f
wft
N
Cbz
H
(–)-21
O
NH
Me

SAMPLE		DEC. & VT	
solvent	CDCl3	dfrq	500.229
		dn	H1
		dpwr	37
		dof	-500.0
		dm	y
		dmm	w
ACQUISITION		dmf	10000
sfrq	125.795	dseq	
tn	C13	dres	1.0
at	1.736	homo	n
np	131010	PROCESSING	
sw	37735.8	lb	0.30
fb	not used	wtfile	
bs	8	proc	ft
ss	1	fn	131072
tpwr	53	math	f
pw	6.9		
d1	0.763	werr	
tof	631.4	wexp	
nt	1.11111e+06	wbs	
ct	0	wnt	
alock	n		
gain	60		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-2515.9		
wp	30187.6		
vs	422		
sc	0		
wc	250		
hzmm	120.75		
is	500.00		
rfl	16005.0		
rfp	9714.2		
th	20		
ins	1.000		
al ph			



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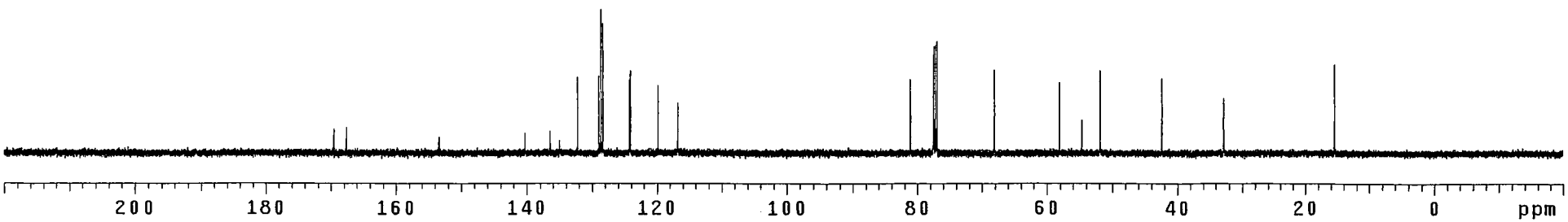
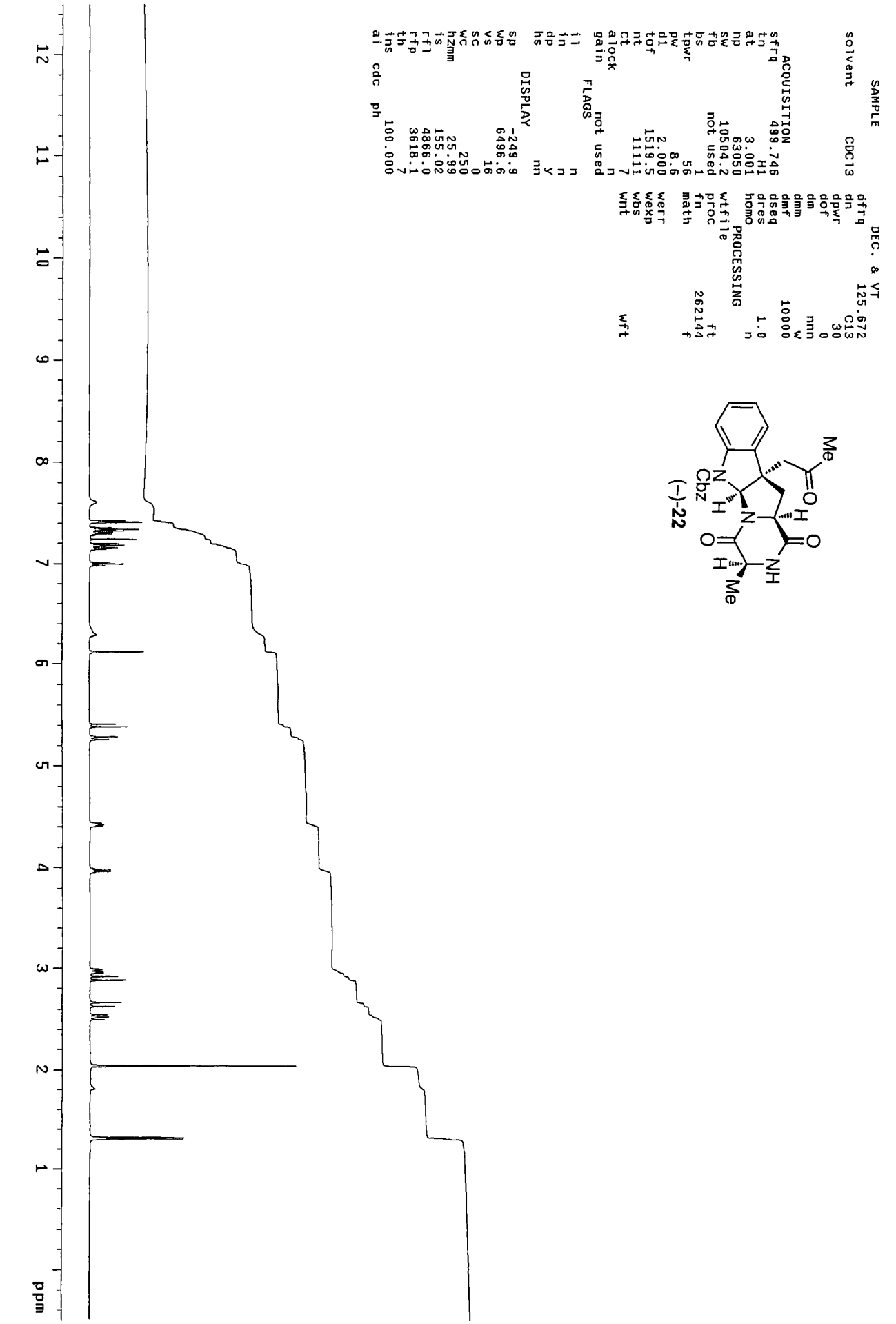



SAMPLE
solvent CDCl3
ACQUISITION
sfrq 499.746
tn H1
at 3.001
np 63050
sw 10504.2
fb not used
bs 1
tpwr 56
pw 8.6
d1 2.000
tof 1519.5
nt 11111
ct 7
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -249.9
wp 6496.6
vs 16
sc 0
wc 250
hzmm 25.99
is 155.02
rfl 4866.0
rfp 3618.1
th 7
ins
al cdc ph 100.000
DEC. & VT
dfrq 125.672
dn C13
dpwr 30
dof 0
dm nnn
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 262144
math f
werr
wexp
wbs
wnt wft
Me
O
H
N
Cbz
O
NH
Me
(−)-22
12
11
10
9
8
7
6
5
4
3
2
1
ppm

SAMPLE	
solvent	CDCl3

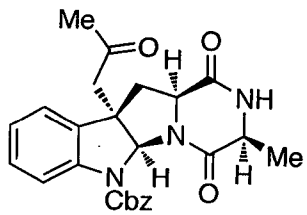
ACQUISITION	
sfrq	125.795
tn	C13
at	1.736
np	131010
sw	37735.8
fb	not used
bs	8
ss	1
tpwr	53
pw	6.9
d1	0.763
tof	631.4
nt	111111
ct	256
alock	n
gain	60

FLAGS	
il	n
in	n
dp	y
hs	nn

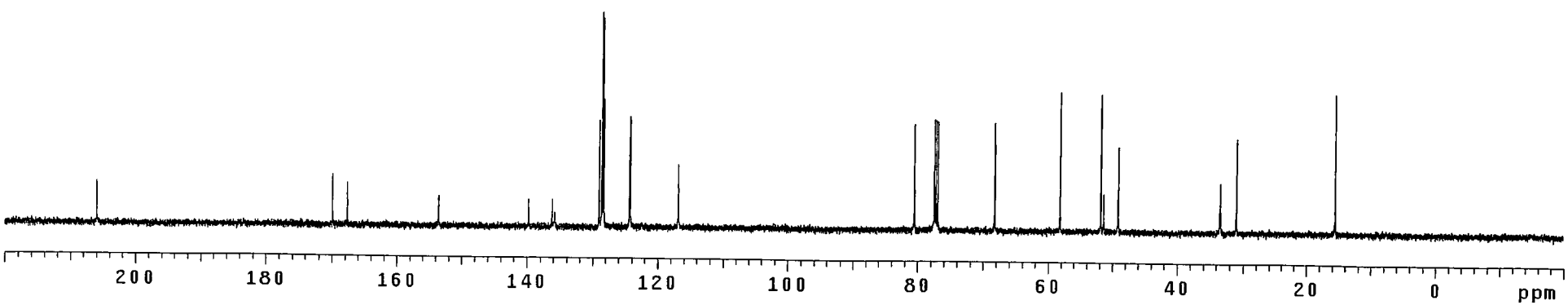
DISPLAY	
sp	-2515.9
wp	30187.6
vs	744
sc	0
wc	250
hzmm	120.75
is	500.00
rfl	16009.1
rfp	9714.2
th	4
ins	1.000
al	ph

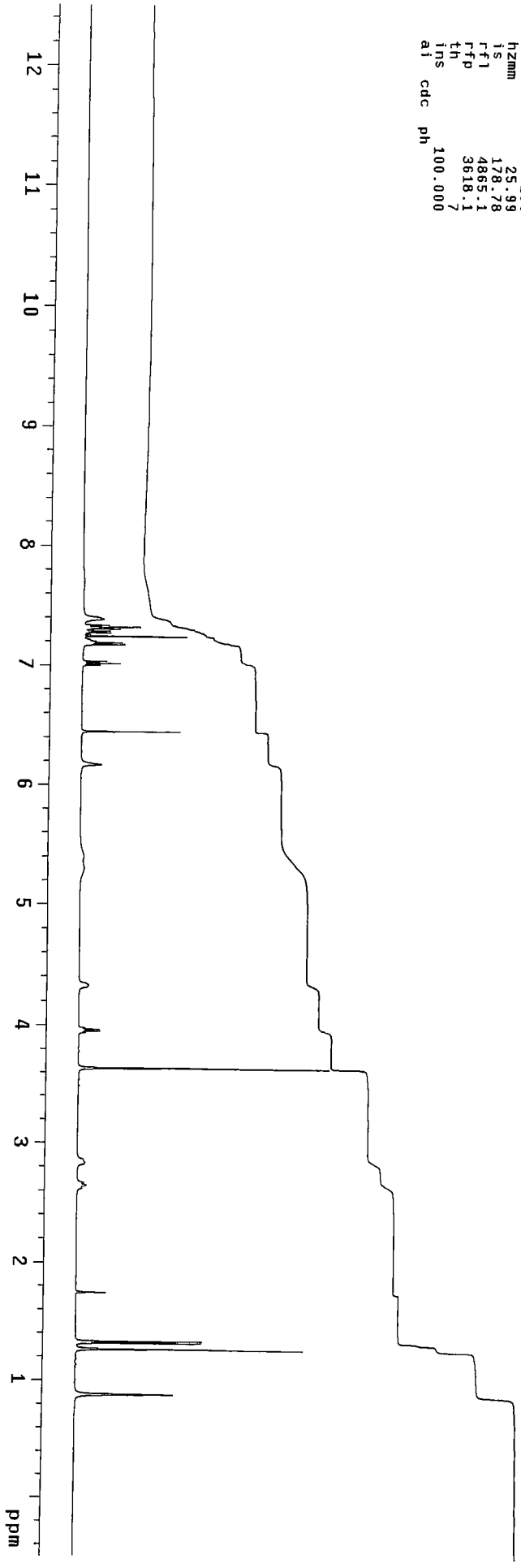
DEC. & VT	
dfrq	500.229
dn	H1
dpwr	37
dof	-500.0
dm	y
dmm	w
dmf	10000
dseq	
dres	1.0
homo	n

PROCESSING	
lb	1.00
wtfile	
proc	ft
fn	131072
math	f
werr	
wexp	
wbs	
wnt	

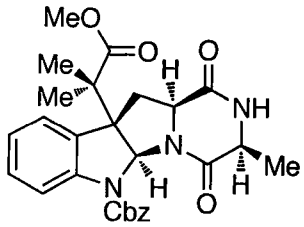


(–)-**22**

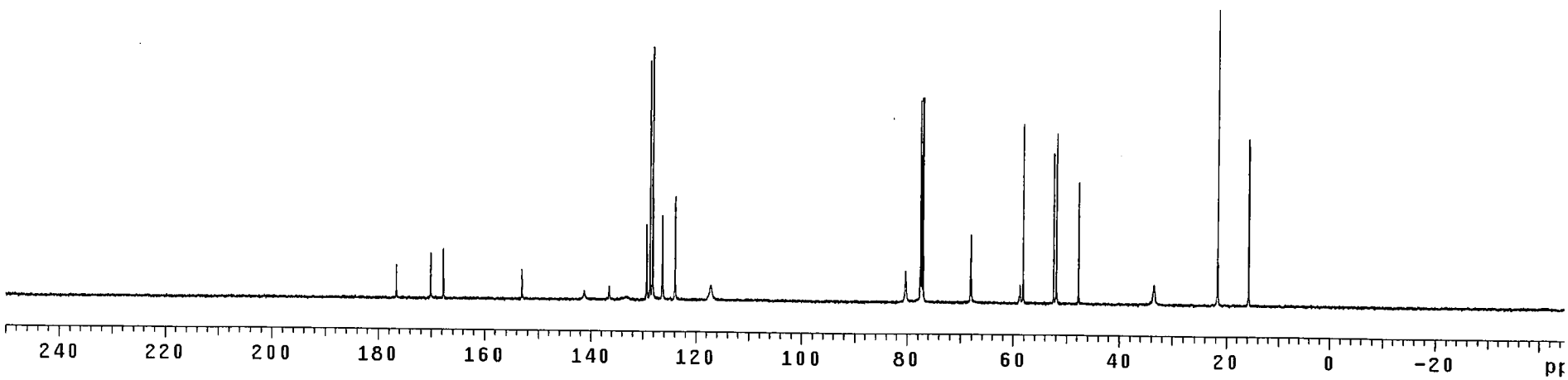


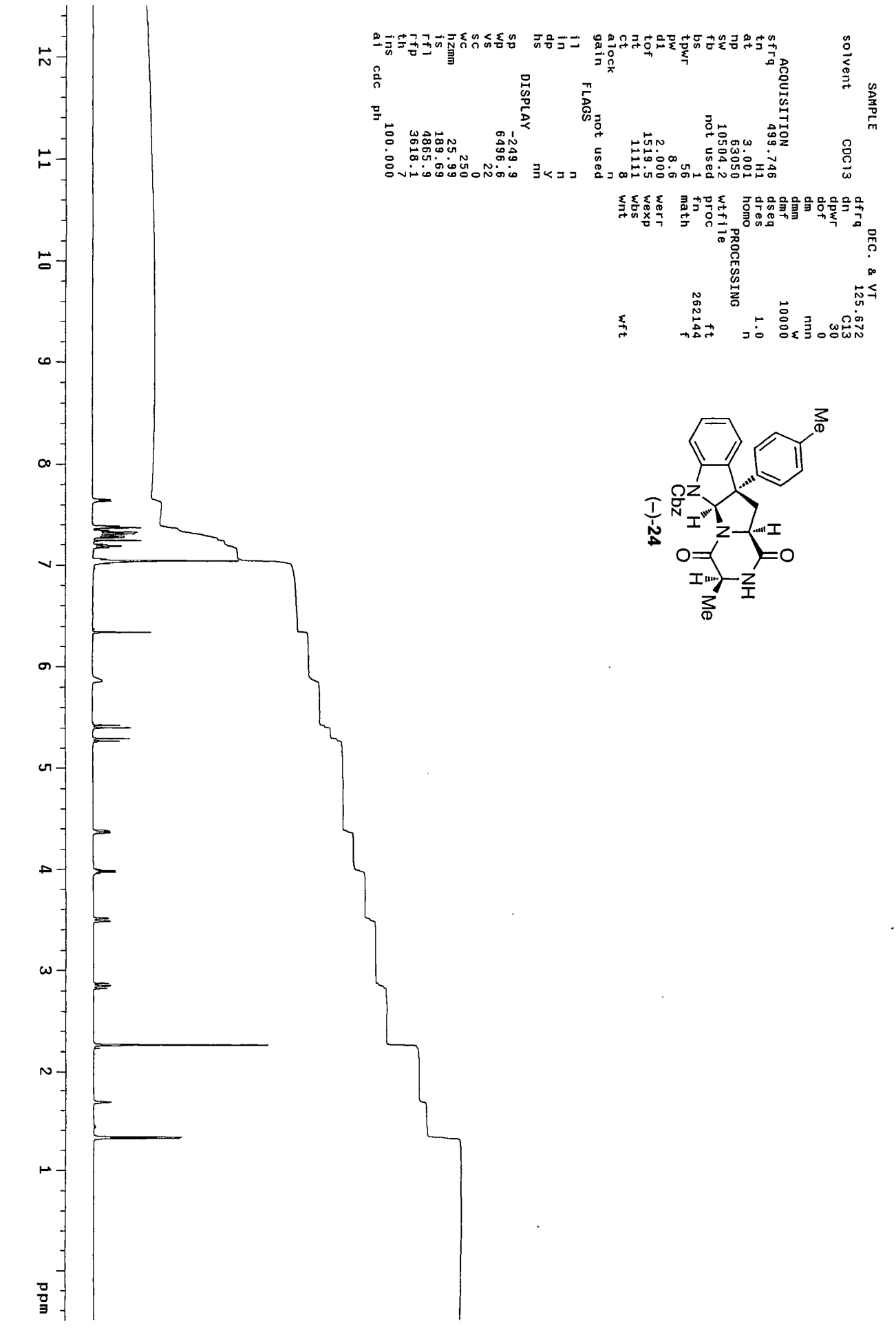

SAMPLE
solvent CDCl3
ACQUISITION
sfrq 499.746
tn H1
at 3.001
np 63050
sw 10504.2
fb
bs 1
tpwr 56
pw 8.6
d1 2.000
tof 1519.5
nt 11111
ct 10
alock n
gain
FLAGS
not used
i1 n
in n
dp y
hs nn
DISPLAY
sp -249.9
wp 6496.6
vs 19
sc 0
wc 250
hzmm 25.99
is 178.78
rfl 4865.1
rfp 3618.1
th 7
ins
ai cdc ph 100.000
DEC. & VT
dfrq 125.672
dn C13
dpwr 30
dof 0
dm nnn
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 262144
math f
werr
wexp
wbs
wnt wft
MeO
Me
Me
O
H
N
Cbz
H
N
O
H
O
NH
Me
(−)-23
12
11
10
9
8
7
6
5
4
3
2
1
ppm

SAMPLE		DEC. & VT	
solvent	CDCl3	dfrq	500.229
		dn	H1
		dpwr	37
		dof	-500.0
		dm	y
		dmm	w
ACQUISITION		dmf	10000
sfrq	125.795	dseq	
tn	C13	dres	1.0
at	1.736	homo	n
np	131010	PROCESSING	
sw	37735.8	lb	2.00
fb	not used	wtfile	
bs	8	proc	ft
ss	1	fn	131072
tpwr	53	math	f
pw	6.9		
d1	0.763	werr	
tof	631.4	wexp	
nt	111111	wbs	
ct	2800	wnt	
alock	n		
gain	60		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-6292.0		
wp	37735.3		
vs	1457		
sc	0		
wc	250		
hzmm	150.94		
is	500.00		
rfl	16006.8		
rfp	9714.2		
th	23		
ins	1.000		
ai ph			

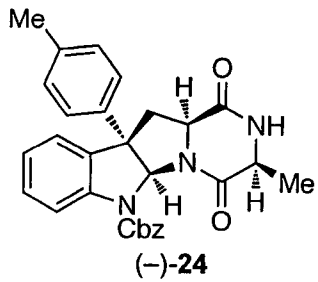


(–)-**23**

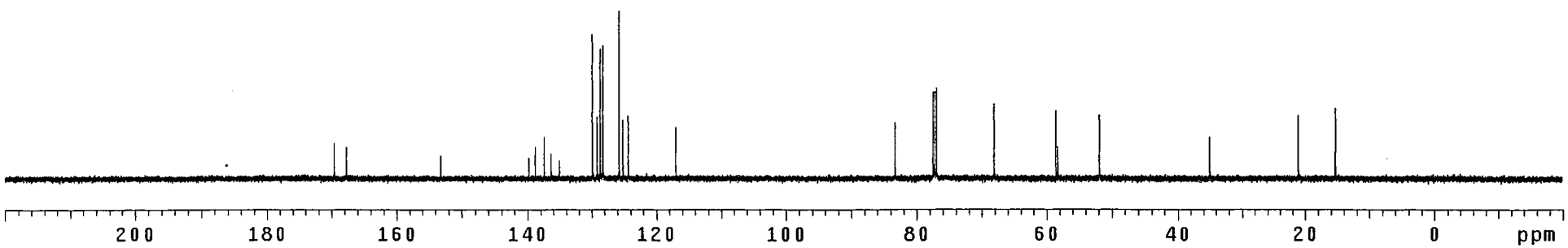


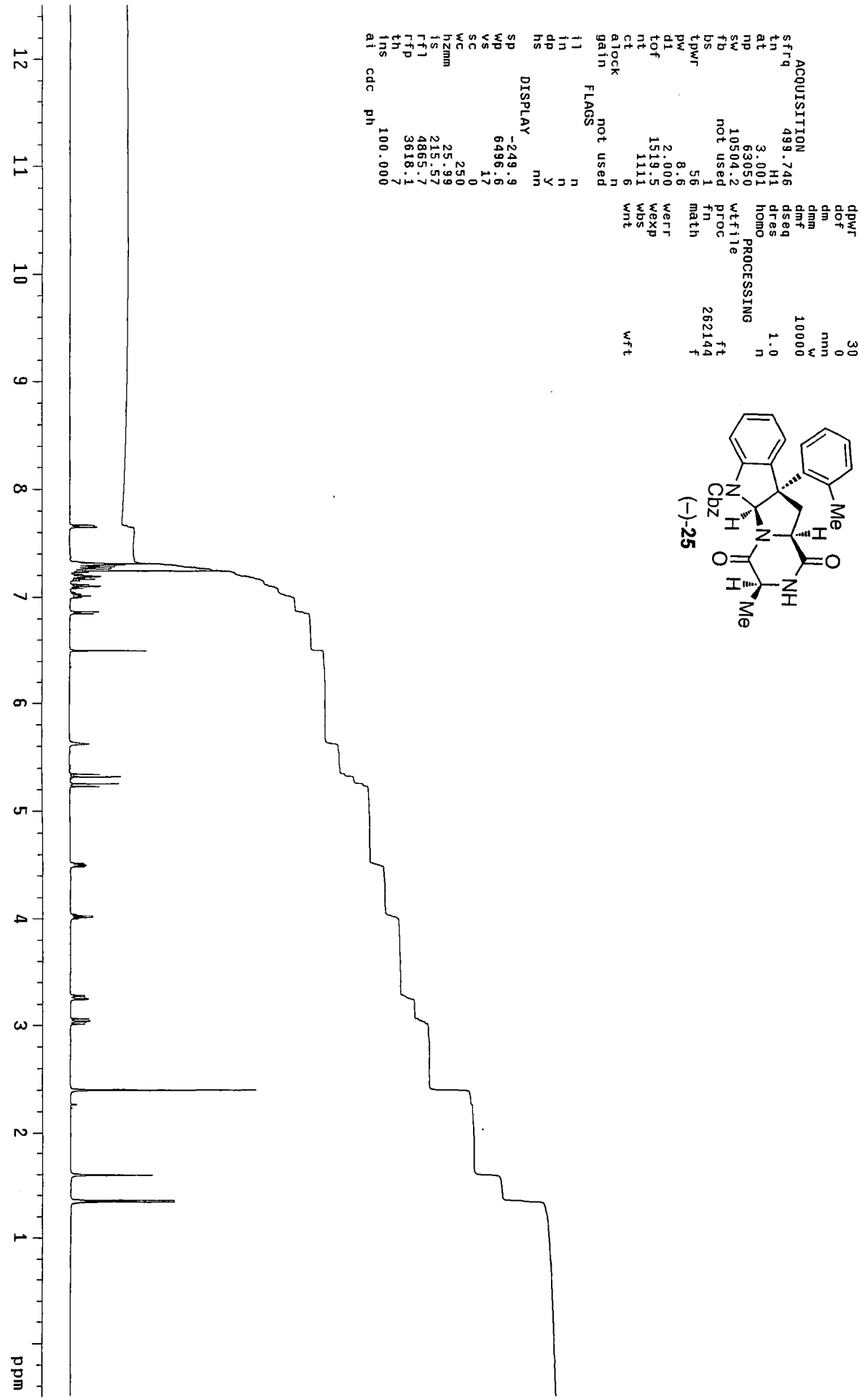

SAMPLE
solvent CDCl3
ACQUISITION
sfrq 499.746
tn H1
at 3.001
np 63050
sw 10504.2
fb not used
bs 1
tpwr 56
pw 8.6
d1 2.000
tof 1519.5
nt 11111
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -249.9
wp 6496.6
vs 22
sc 0
wc 250
hzmm 25.99
is 189.69
rfl 4865.9
rfp 3618.1
th 7
ins 100.000
ai cdc ph
DEC. & VT
dfrq 125.672
dn C13
dpwr 30
dof 0
dm nnn
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 262144
math f
werr
wexp
wbs
wnt wft
Me
N
Cbz
H
N
H
O
O
NH
H
Me
(−)-24
12
11
10
9
8
7
6
5
4
3
2
1
ppm

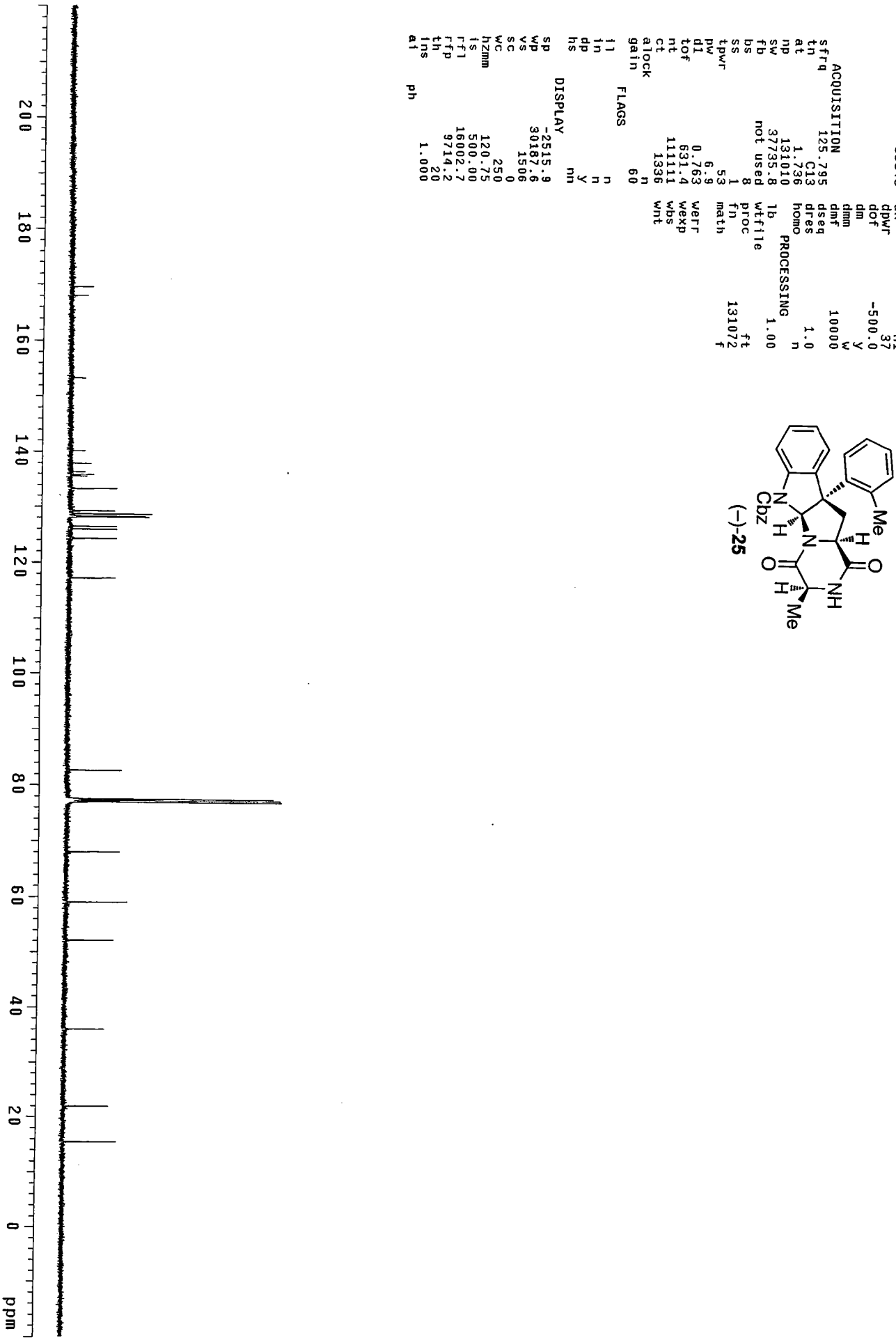
SAMPLE		DEC. & VT	
solvent	CDCl3	dfrq	500.229
		dn	H1
		dpwr	37
		dof	-500.0
		dm	y
		dmm	w
ACQUISITION		dmf	10000
sfrq	125.795	dseq	
tn	C13	dres	1.0
at	1.736	homo	n
np	131010	PROCESSING	
sw	37735.8	lb	0.30
fb	not used	wtfile	
bs	8	proc	ft
ss	1	fn	131072
tpwr	53	math	f
pw	6.9		
d1	0.763	werr	
tof	631.4	wexp	
nt	11111	wbs	
ct	192	wnt	
alock	n		
gain	60		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-2515.9		
wp	30187.6		
vs	301		
sc	0		
wc	250		
hzmm	120.75		
is	500.00		
rfl	16006.2		
rfp	9714.2		
th	20		
ins	1.000		
ai ph			

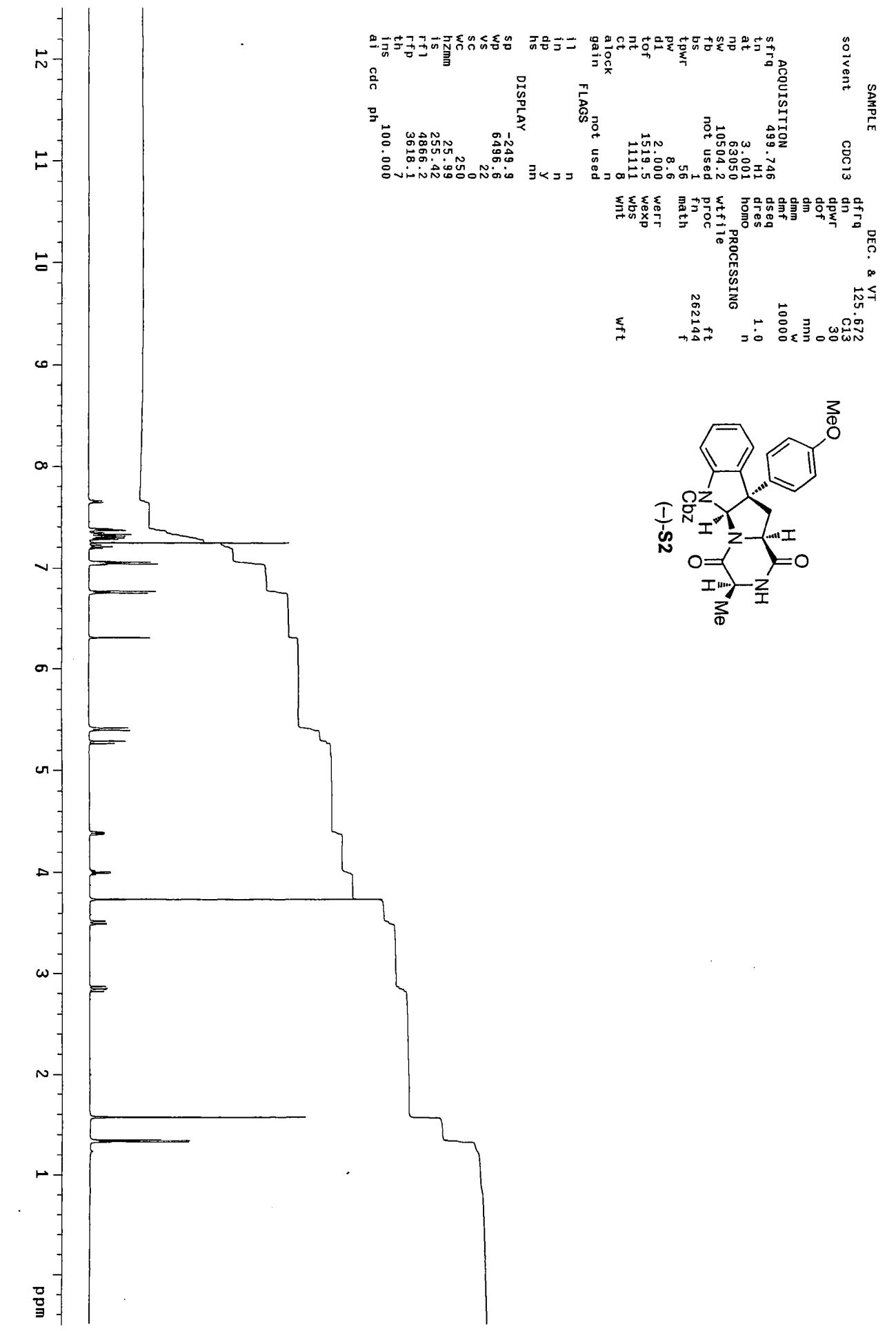


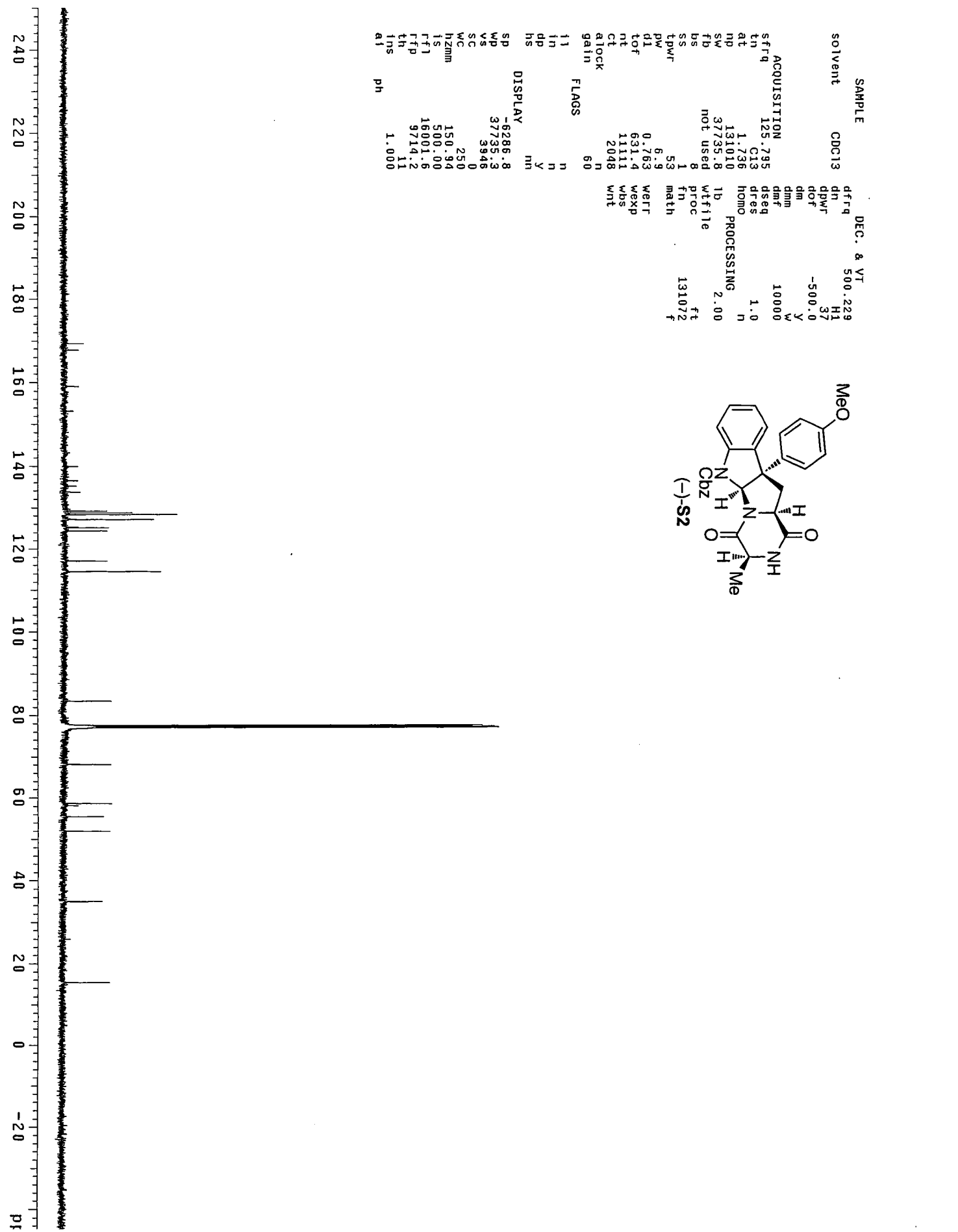
(–)-**24**

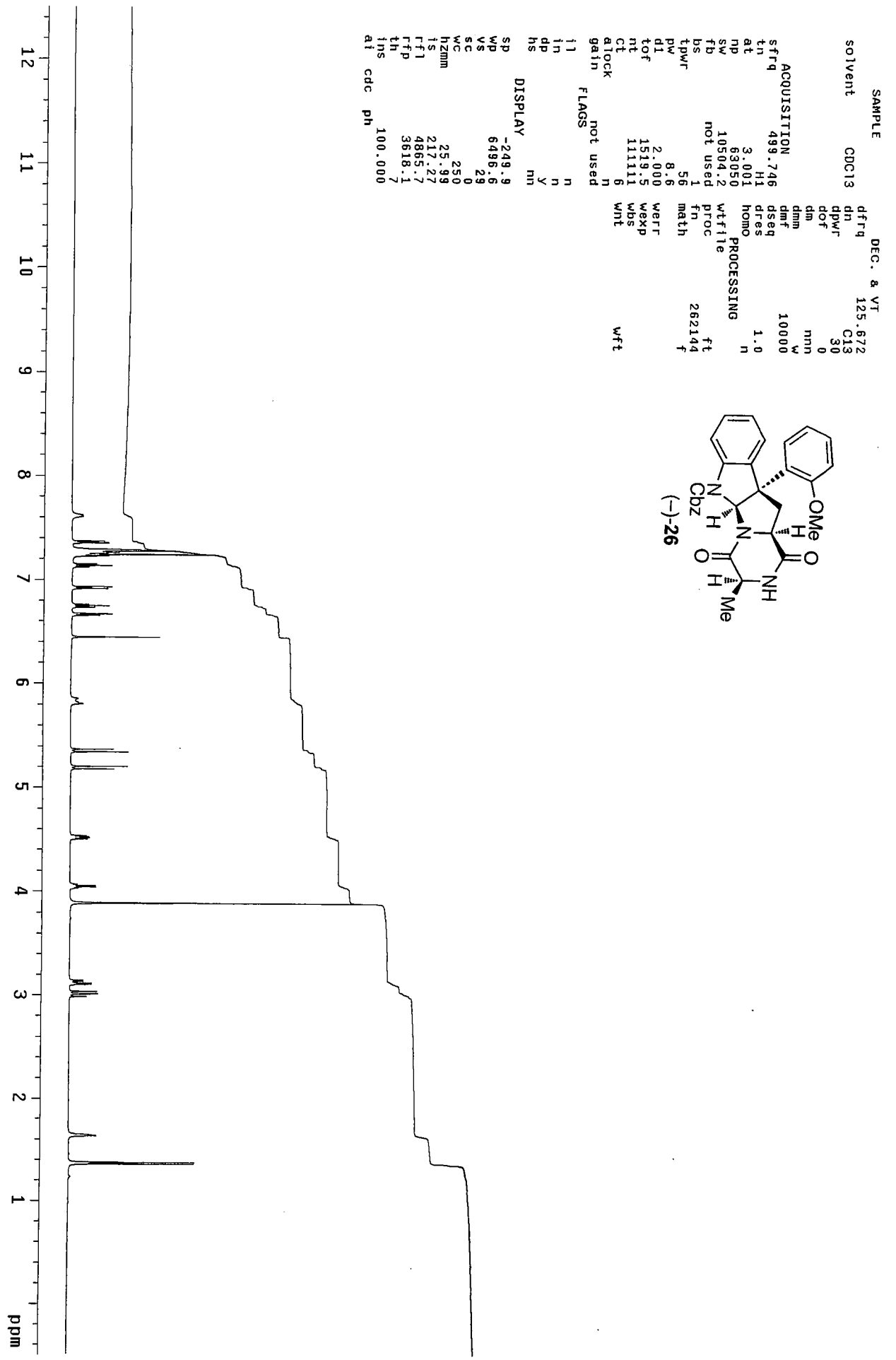


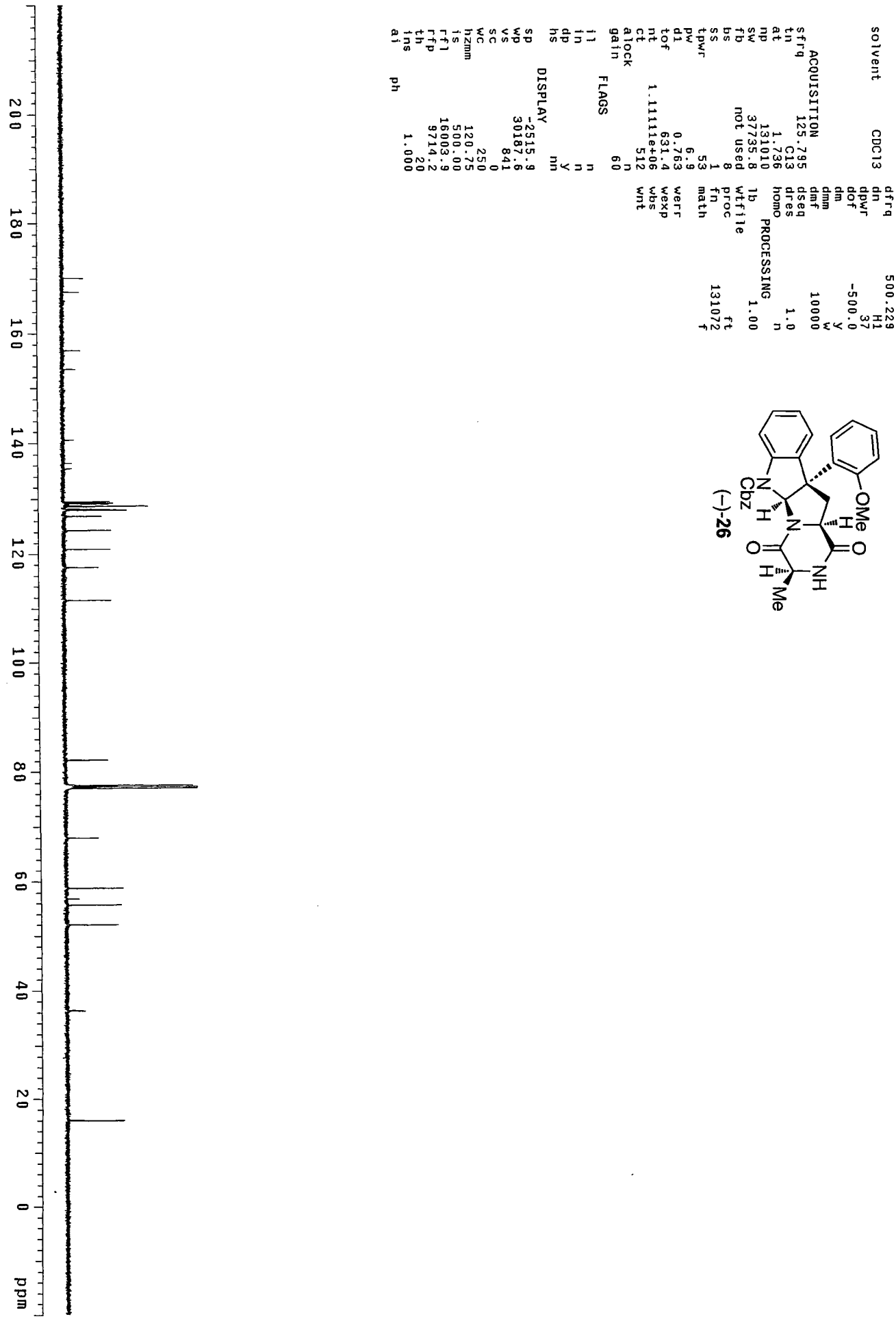

SAMPLE
solvent CDCl3
ACQUISITION
sfrq 499.746
tn H1
at 3.001
np 63350
sw 10504.2
fb not used
bs 1
tpwr 56
pw 8.6
d1 2.000
tof 1519.5
nt 1111
ct 6
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -249.9
wp 6496.6
vs 17
sc 0
wc 250
hzmm 25.99
is 215.57
rfl 4865.7
rfp 3618.1
th 7
ins 100.000
ai cdc ph
DEC. & VT
dfrq 125.672
dn C13
dpwr 30
dof 0
dm nnn
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 262144
math f
werr
wexp
wbs
wnt wft
Me
H
O
NH
Me
O
H
N
N
H
Cbz
(–)-25
12
11
10
9
8
7
6
5
4
3
2
1
ppm

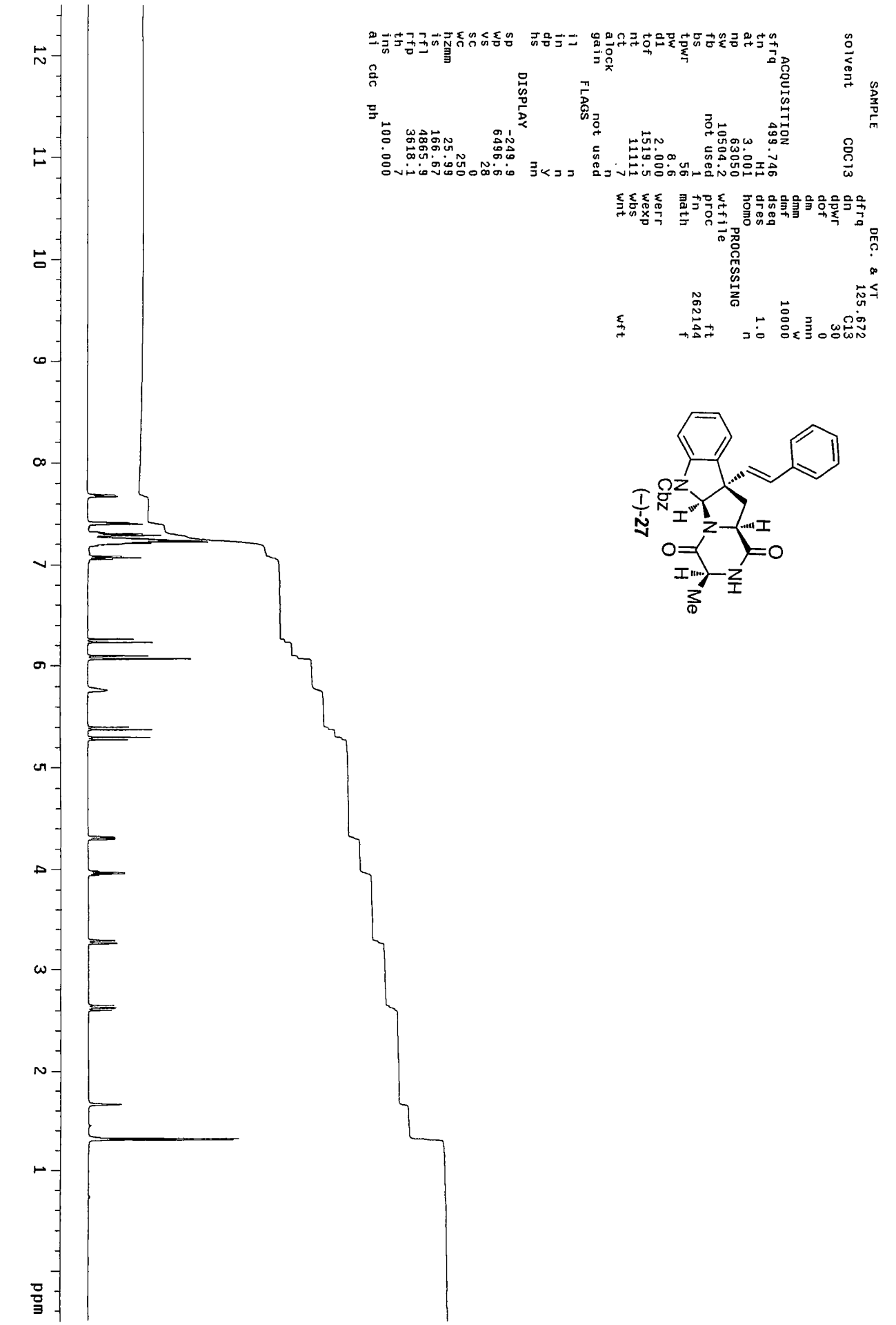

SAMPLE
solvent CDCl3
ACQUISITION
sfrq 125.795
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 111111
ct 1336
alock n
gain 60
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -2515.9
wp 30187.6
vs 1506
sc 0
wc 250
hzmm 120.75
is 500.00
rfl 16002.7
rfp 9714.2
th 20
ins 1.000
ai ph
DEC. & VT
dfrq 500.229
dn H1
dpwr 37
dof -500.0
dm y
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc
fn 131072
math f
wexp
wbs
wnt
Cbz
(−)-25
Me
NH
ppm
200
180
160
140
120
100
80
60
40
20
0


SAMPLE
solvent CDCl3
ACQUISITION
sfrq 499.746
tn H1
at 3.001
np 63050
sw 10504.2
fb not used
bs 1
tpwr 56
pw 8.6
d1 2.000
tof 1519.5
nt 11111
ct 8
alock n
gain n
FLAGS
not used
il n
in n
dp y
hs nn
DISPLAY
sp -249.9
wp 6496.6
vs 22
sc 0
wc 250
hzmm 25.99
is 255.42
rfl 4866.2
rfp 3618.1
th 7
ins 100.000
ai cdc ph
DEC. & VT
dfrq 125.672
dn C13
dpwr 30
dof 0
dm nnn
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 262144
math f
wnt wft
MeO
Cbz
(−)-S2
12
11
10
9
8
7
6
5
4
3
2
1
ppm

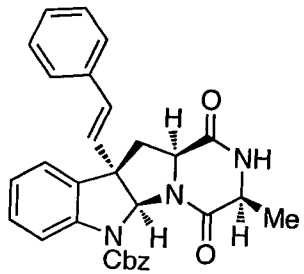

SAMPLE
solvent CDCl3
ACQUISITION
sfrq 125.795
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs 8
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 11111
ct 2048
alock n
gain 60
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -6286.8
wp 37735.3
vs 3946
sc 0
wc 250
hzmm 150.94
is 500.00
rfl 16001.6
rfp 9714.2
th 11
ins 1.000
ph
dfrq DEC. & VT
dn 500.229
dpwr H1
dof 37
dm -500.0
dmm y
dmf w
dseq 10000
dres 1.0
homo n
PROCESSING
lb 2.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
MeO
Cbz
(–)-S2
Me
NH
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
pp


SAMPLE
solvent CDCl3
ACQUISITION
sfrq 499.746
tn H1
at 3.001
np 63050
sw 10504.2
fb not used
bs 1
tpwr 56
pw 8.6
d1 2.000
tof 1519.5
nt 111111
ct 6
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -249.9
wp 6496.6
vs 29
sc 0
wc 250
hzmm 25.99
is 217.27
rfl 4865.7
rfp 3618.1
th 7
ins 100.000
ai cdc ph
dfrq DEC. & VT
dn 125.672
dpwr C13
dof 30
dm 0
dmm nnn
dmf w
dseq 10000
dres
homo 1.0
n
PROCESSING
wtfile
proc ft
fn 262144
math f
werr
wexp wft
wbs
wnt
OMe
NH
Me
Cbz
(−)-26
12
11
10
9
8
7
6
5
4
3
2
1
ppm


SAMPLE
solvent CDCl3
ACQUISITION
sfrq 125.795
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs 8
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 1.11111e+06
ct 512
alock wnt
gain n
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -2515.9
wp 30187.6
vs 841
sc 0
wc 250
hzmm 120.75
is 500.00
rfl 16003.9
rfp 9714.2
th 20
ins 1.000
ai ph
DEC. & VT
dfrq 500.229
dn H1
dpwr 37
dof -500.0
dm y
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc
ft
fn 131072
math f
werr
wexp
wbs
wnt
OMe
H
O
N
H
Cbz
N
O
NH
H
Me
(−)-26
200
180
160
140
120
100
80
60
40
20
0
ppm


SAMPLE
solvent CDCl3
ACQUISITION
sfrq 499.746
tn H1
at 3.001
np 63050
sw 10504.2
fb not used
bs 1
tpwr 56
pw 8.6
d1 2.000
tof 1519.5
nt 11111
ct 7
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -249.9
wp 6496.6
vs 28
sc 0
wc 250
hzmm 25.99
is 166.67
rfl 4865.9
rfp 3618.1
th 7
ins 100.000
ai cdc ph
DEC. & VT
dfrq 125.672
dn C13
dpwr 30
dof 0
dm nnn
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 262144
math f
werr
wexp
wbs
wnt wft
Cbz
(−)-27
Me
NH
O
H
N
ppm
12
11
10
9
8
7
6
5
4
3
2
1

SAMPLE		DEC. & VT	
solvent	CDCl3	dfrq	500.229
		dn	H1
		dpwr	37
		dof	-500.0
		dm	y
		dmm	w
ACQUISITION		dmf	10000
sfrq	125.795	dseq	
tn	C13	dres	1.0
at	1.736	homo	n
np	131010	PROCESSING	
sw	37735.8	lb	1.00
fb	not used	wtfile	
bs	8	proc	ft
ss	1	fn	131072
tpwr	53	math	f
pw	6.9		
d1	0.763	werr	
tof	631.4	wexp	
nt	11111	wbs	
ct	256	wnt	
alock	n		
gain	60		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-2515.9		
wp	30187.6		
vs	625		
sc	0		
wc	250		
hzmm	120.75		
is	500.00		
rfl	16005.6		
rfp	9714.2		
th	20		
ins	1.000		
ai	ph		



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