Concise Total Synthesis of (±)-Marcfortine B

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Supporting Information

Complete reference 2a:

Lee, B. H.; Clothier, M. F.; Dutton, F. E.; Nelson, S. J.; Johnson, S. S.; Thompson, D. P.; Geary, T. G.; Whaley, H. D.; Haber, C. L.; Marshall, V. P.; Kornis, G. I.; McNally, P. L.; Ciadella, J. I.; Martin, D. G.; Bowman, J. W.; Baker, C. A.; Coscarelli, E. M.; Alexander-Bowman, S. J.; Davis, J. P.; Zinser, E. W.; Wiley, V.; Lipton, M. F.; Mauragis, M. A. *Curr. Top. Med. Chem.* **2002**, 2, 779.

Experimental

General

All reactions were carried out under an atmosphere of nitrogen or argon in oven-dried glassware with magnetic stirring, unless otherwise indicated. Solvents were from J. C. Meyer's Solvent Purification System. All other reagents were used as obtained unless otherwise noted. Flash Chromatography was performed with EM Science silica gel (0.040-0.063 μm grade). Analytical thin-layer chromatography was performed with commercial glass plates coated with 0.25 mm silica gel (E. Merck, Kieselgel 60 F254). Melting points were obtained on a Thomas-Hoover apparatus in open capillary tubes and are uncorrected. Proton nuclear magnetic resonance (¹H-NMR) data were acquired on a Mercury 400 (400 MHz) or on a Varian Unity Inova-500 (500 MHz) spectrometer. Chemical shifts are reported in delta (δ) units, in parts per million (ppm) downfield from tetramethylsilane. Splitting patterns are designated as s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet, br, broad. Carbon-13 nuclear magnetic resonance (¹³C-NMR) data were acquired at 100 MHz on a Mercury 400 or at 125 MHz on a Varian Unity Inova 500 spectrometer. Chemical shifts are reported in ppm relative to the center line of a triplet at 77.0 ppm for chloroform-*d*. Infrared (IR) data were recorded as films on sodium chloride plates or as potassium bromide (KBr) pellets on a Perkin-Elmer Paragon 500 FT-IR spectrometer. Absorbance frequencies are reported in reciprocal centimeters (cm⁻¹). Elemental analyses (Anal.) were performed by M.-H.-W. Laboratories of Phoenix, AZ.

3-Hydroxy pipercolinic amide hydrobromide (2): 3-Hydroxypicolinic amide (978 mg, 7.08 mmol) was dissolved in 50 mL hot MeOH and acidified to pH 2 with 48% HBr (0.8 mL) and subsequently concentrated *in vacuo*. The residue was suspended in 25 mL water, transferred to a high pressure hydrogenation apparatus and charged with 300 mg 5% Rh/Al₂O₃. The apparatus was sealed, purged 3 times with Nitrogen, pressurized with 100 bar Hydrogen and heated to 70°C for 18 h in an oil bath. After cooling to rt, the pressure was released and the reaction mixture was filtered. The filter cake was washed twice with 10 mL water. The filtrate was evaporated, suspended in MeOH and concentrated *in vacuo*, yielding 1.59 g (quant.) of the piperidine hydrobromide salt **2** as white solid.

2: mp: 235-240°C. **IR** (KBr): $\tilde{\nu}$ = 3384, 3059, 1717, 1609, 1564, 1381, 1138, 1059, 994, 939, 903 cm⁻¹. **¹H-NMR** (400 MHz, D₂O): δ 4.31 (s, 1H), 3.85 (s, 1H), 3.25 (dd, *J* = 12.6 Hz, *J* = 2.4 Hz, 1H), 2.85 (td, *J* = 12.6 Hz, *J* = 3.2 Hz, 1H), 1.88-1.53 (m, 4H) ppm. **¹³C-NMR** (100 MHz, D₂O): δ 170.9, 64.7, 60.7, 43.7, 28.6, 16.2 ppm. **Anal:** calc'd. for C₆H₁₃BrN₂O₂: C 32.02, H 5.82, N 12.45. Found: C 32.30, H 5.75, N 12.28.

***N*-(*tert*-butyloxycarbonyl)-3-isopropylidene-6,7-dimethoxy-indoline-2-one (6):** A solution of 6,7-dimethoxyoxindole **5** (1.70 g, 8.80 mmol) in 100 mL of acetone was treated with concentrated hydrochloric acid (7 mL), and was stirred at r.t. for 48 h. The suspension was concentrated in *vacuo* and the residue was taken up in ethyl acetate. The organic layer was washed with water and brine and dried (MgSO₄). Evaporation of the solvent gave the desired 3-isopropylidene-6,7-dimethoxy-indoline-2-one (**6**) as an amorphous orange powder, 1.43 g (6.13 mmol, 70%).

mp. 188°C. **IR** (NaCl, film): $\tilde{\nu}$ = 3131, 2995, 2832, 2786, 1688, 1506, 1454, 1346, 1278, 1221, 1093, 1039, 986 cm⁻¹. **¹H-NMR** (300 MHz, CDCl₃): δ 8.26 (s, br., 1H), 7.15 (d, *J* = 8.6 Hz, 1H), 6.51 (d, *J* = 8.6 Hz, 1H), 3.86 (s, 6H), 2.55 (s, 3H), 2.28 (s, 3H) ppm. **¹³C-NMR** (75 MHz, CDCl₃): δ 169.4, 152.9, 152.1, 133.1, 132.2, 122.9, 119.3, 118.8, 104.9, 60.8, 55.9, 25.0, 22.6 ppm.

An ice-cold solution of 3-isopropylidene-6,7-dimethoxy-indoline-2-one (5.43 g, 23.28 mmol) in 130 mL of CH₂Cl₂ was treated with (Boc)₂O (7.62 g, 34.92 mmol), triethylamine (3.57 mL, 25.61 mmol), and finally with DMAP (569 mg, 4.66 mmol). The solution was then allowed to warm to r.t., and was stirred for additional 12 h. After quenching with water, the reaction mixture was extracted with EtOAc. The organic phase was washed with water and brine, dried (MgSO₄), and the solvent was evaporated in *vacuo*. Recrystallization of the resulting orange solid from diethyl Et₂O/petrol ether yielded 6.59 g (19.79 mmol, 85%) of **6** as orange crystals.

6: mp 117°C. **IR** (NaCl, film): $\tilde{\nu}$ = 2981, 2940, 2840, 1760, 1715, 1616, 1505, 1455, 1368, 1300, 1265, 1196, 1152, 1122, 1069, 1022, 1004, 954, 849 cm⁻¹. **¹H-NMR** (500 MHz, CDCl₃): δ 7.22 (d, *J* = 8.6 Hz, 1H), 6.63 (d, *J* = 8.6 Hz, 1H), 3.89 (s, 3H), 3.82 (s, 3H), 2.56 (s, 3H), 2.32 (s, 3H), 1.63 (s, 9H) ppm. **¹³C-NMR** (125 MHz, CDCl₃): δ 166.5, 154.1, 152.9, 150.1, 134.1, 131.9, 121.8, 119.1, 118.7, 106.5, 84.4, 60.4, 56.0, 27.6, 25.3, 23.2 ppm. **Anal:** Calc'd for C₁₈H₂₃NO₅: C, 64.85; H, 6.95; N, 4.20. Found: C, 64.66; H, 7.03; N, 4.23.

Carboxylic acid 7: A solution of palladium acetate (892 mg, 3.96 mmol) in 500 mL dry toluene at r.t. was treated with triisopropyl phosphite (6.82 mL, 27.6 mmol). After 15 min *N*-(*tert*-butyloxycarbonyl)-3-isopropylidene-6,7-dimethoxy-indoline-2-one (**6**) (26.3 g, 78.9 mmol) and methyl 3-(trimethylsilyl)-2-(trimethylsilylmethyl)-allyl carbonate (**3**) (43.6 g, 157.8 mmol) were added and the reaction mixture was heated to 110°C for 1.5 h (preheated oilbath). After cooling to r.t. the reaction mixture was concentrated in *vacuo*, and the resulting residue was chromatographed on silica gel (CH₂Cl₂/MeOH, 20:1 → 10:1) to afford **7** as a 1:1 mixture of the two diastereomers as faint red oil, which was used directly in the next step.

7: **IR** (NaCl, film): $\tilde{\nu}$ = 2981, 2942, 1759, 1742, 1624, 1503, 1462, 1371, 1261, 1150 cm⁻¹. **¹H-NMR** (500 MHz, CDCl₃): δ 6.87 (d, *J* = 8.3 Hz, 1H), 6.87 (d, *J* = 8.3 Hz, 1H), 6.64 (d, *J* = 8.3 Hz, 1H), 6.63 (d, *J* = 8.3 Hz, 1H), 5.35–5.20 (m, 4H), 4.28 (s, br., 1H), 3.88 (s, 3H), 3.87 (s, 3H), 3.83 (s, 3H), 3.83 (s, 3H), 3.72 (s, br., 1H), 3.10 (d, *J* = 16.4 Hz, 1H), 2.91 (d, *J* = 17.6 Hz, 1H), 2.79 (d, *J* = 17.6 Hz, 1H), 2.72 (d, *J* = 16.4 Hz, 1H), 1.60 (s, 9H), 1.59 (s, 9H), 1.14 (s, 3H), 1.09 (s, 3H), 1.03 (s, 3H) ppm. **¹³C-NMR** (75 MHz, CDCl₃): δ : 178.5, 177.9, 176.9, 176.1, 153.5, 153.3, 149.2, 144.2, 143.3, 134.5, 134.4, 133.7, 132.6, 124.4, 120.8, 120.5, 119.1, 113.1, 111.8, 107.1, 106.6, 85.1, 84.8, 60.4, 60.3, 60.1, 60.0, 56.8, 56.0, 50.0, 49.1, 40.9, 39.4, 27.5, 27.4 ppm. **HRMS** (EI): Calc'd for C₂₃H₂₉NO₇⁺ [M]⁺: 431.1944. Found: 431.1939.

Methyl ester 8: A solution of the above carboxylic acid **7** in 1.5 L acetone was treated with 14.2 g (103 mmol) K_2CO_3 and 10.6 mL (112 mmol) dimethyl sulfate. The reaction mixture was stirred at 40 °C for 2 h, cooled to r.t. and concentrated in *vacuo*. The residue was redissolved in 600 mL water and extracted into CH_2Cl_2 (3x 600 mL). The combined organic layers were dried ($MgSO_4$) and evaporated. The residue was purified *via* chromatography on silica gel (petrol ether/EtOAc 4:1) to give 32.8 g (93% over 2 steps) of **8** as a 1:1 mixture of the two diastereomers.

IR (NaCl, film): $\tilde{\nu}$ = 2981.1, 2947.0, 1759.2, 1740.2, 1622.8, 1503.0, 1462.0, 1370.6, 1261.2, 1234.9, 1194.9, 1056.2, 1117.4, 1022.1 cm^{-1} . **1H -NMR** (500 MHz, $CDCl_3$, *trans*-isomer): δ 6.87 (d, J = 8.4 Hz, 1H), 6.64 (d, J = 8.4 Hz, 1H), 5.24-5.22 (m, 1H), 5.17-5.15 (m, 1H), 4.24 (s, br., 1H), 3.88 (s, 3H), 3.84 (s, 3H), 3.74 (s, 3H), 2.93-2.86 (m, 1H), 2.80 (d, J = 18.1 Hz, 1H), 1.60 (s, 9H), 1.02 (s, 3H), 0.98 (s, 3H) ppm. **1H -NMR** (500 MHz, $CDCl_3$, *cis*-isomer): δ 6.84 (d, J = 8.3 Hz, 1H), 6.60 (d, J = 8.3 Hz, 1H), 5.34-5.30 (m, 1H), 5.20-5.17 (m, 1H), 3.87 (s, 3H), 3.83 (s, 3H), 3.75 (s, 3H), 3.70-3.68 (m, 1H), 3.33-3.28 (m, 1H), 2.53 (d, J = 16.7 Hz, 1H), 1.61 (s, 9H), 1.13 (s, 3H), 0.79 (s, 3H) ppm. **^{13}C -NMR** (125 MHz, $CDCl_3$, *trans*-isomer): δ 178.6, 172.6, 153.5, 149.3, 144.0, 134.5, 133.8, 120.8, 120.8, 111.3, 106.6, 84.8, 60.4, 60.0, 57.1, 56.0, 51.5, 49.0, 39.5, 27.6, 21.9, 21.6 ppm. **^{13}C -NMR** (125 MHz, $CDCl_3$, *cis*-isomer): δ 175.5, 172.1, 153.1, 149.5, 144.6, 134.5, 132.4, 126.0, 118.4, 112.6, 106.9, 84.7, 59.6, 58.8, 58.7, 56.0, 51.7, 50.3, 40.3, 27.5, 23.0, 21.1 ppm. **HRMS** (EI): Calc'd for $C_{24}H_{31}NO_7$ $[M]^+$: 445.2101. Found: 445.2103.

Allylic alcohol 9: A solution of 12.13 g (27.23 mmol) of the methyl esters **8** in 750 mL dry CH_2Cl_2 was treated with (13.43 g, 54.45 mmol, 70%, 2 equiv.) *m*CPBA at 0°C. The reaction mixture was allowed to warm to r.t. and stirred over night. The reaction was quenched with sat. aq. Na_2SO_3 (400 mL). After vigorous stirring for 30 min, the aqueous layer was separated and extracted with CH_2Cl_2 (3x 300 mL). The combined organic phases were dried ($MgSO_4$) and concentrated in *vacuo*. Chromatography on silica gel (petrol ether/EtOAc 2:1) yielded the diastereomeric epoxides as a complex mixture (11.27 g, 24.42 mmol, 90 %).

To an ice-cold solution of the epoxides (1.09 g, 2.37 mmol) in 5 mL of dry THF was added DBU (0.54 mL, 3.55 mmol). The mixture was stirred at r.t. for 1 day. The mixture was diluted with diethyl ether, washed with 1N $KHSO_4$, sat. $NaHCO_3$, brine, dried ($MgSO_4$) and evaporated. Chromatography of the residue on silica gel (petrol ether/EtOAc 1:1) yielded 550 mg, (50%) of **9** as colourless foam. The recovered starting material (483 mg, 44 %) was subjected to DBU again to give additional allylic alcohol **9** (233 mg, 22 %). The total yield of **9** after two iterations was 783 mg (72 %).

9: IR (NaCl, film): $\tilde{\nu}$ = 3515.8, 2979.7, 2940.4, 1763.8, 1736.6, 1709.4, 1623.4, 1503.1, 1462.4, 1369.5, 1268.3, 1215.8, 1148.2 cm^{-1} . **1H -NMR** (500 MHz, $CDCl_3$): δ 6.94 (d, J = 8.3 Hz, 1H), 6.62 (d, J = 8.3 Hz, 1H), 4.60-4.46 (m, 2H), 3.87 (s, 3H), 3.83 (s, 3H), 3.77 (s, 3H), 3.06 (d, J = 17.9 Hz, 1H), 2.93 (s, br., 1H), 2.86 (d, J = 17.9 Hz, 1H), 1.59 (s, 9H), 1.22 (s, 3H), 1.09 (s, 3H) ppm. **^{13}C -NMR** (125 MHz, $CDCl_3$): δ 177.3, 166.0, 155.3, 153.3, 149.4, 134.5, 134.4, 133.2, 122.7, 119.9, 106.9, 84.7, 60.4, 60.3, 59.9, 56.0, 53.6, 51.5, 42.3, 27.5, 24.2, 22.1 ppm. **Anal:** Calc'd. for $C_{24}H_{31}NO_8$: C 62.46, H 6.77, N 3.04. Found: C 62.70, H 6.77, N 3.04.

α,β -Unsaturated amide 10: To an ice-cold solution of **9** (10.8 g, 23.5 mmol) and NEt₃ (3.7 mL, 25.9 mmol) in 200 mL dry CH₂Cl₂ was added 2.00 mL (2.96 g, 25.9 mmol) methanesulfonylchloride. The mixture was stirred for 10 min at 0°C and then diluted with 600 mL Et₂O, washed with sat. NaHCO₃ and brine, dried (MgSO₄) and evaporated. The residue was taken up in 30 mL dry DMSO and added to a solution of 10.53 g (47 mmol) piperidine hydrobromide **2** and 14.0 mL (98 mmol) NEt₃ in 120 mL dry DMSO. The mixture was stirred at r.t. over night, diluted with 800 mL EtOAc, washed with water and brine, dried (MgSO₄) and evaporated. Column chromatography on silica gel (petrol ether/EtOAc 1:1 -> CH₂Cl₂/MeOH 20:1) gave the unstable tertiary amine which was immediately dissolved in 150 mL dry CH₂Cl₂ and cooled to 0°C. 4.0 mL (28.2 mmol) NEt₃ and 2.00 mL (2.96 g, 25.9 mmol) methanesulfonylchloride were added. After 10 min, 19 mL DBU were added, the reaction was brought back to r.t. and stirred for 2 h. The mixture was extracted with 600 mL EtOAc, washed with water and brine, dried (MgSO₄) and evaporated. The residue was chromatographed on silica gel (CH₂Cl₂/MeOH 20:1) yielding 11.28 g (84% over 3 steps) of the α,β -unsaturated amide **10** as pale yellow foam.

10: mp: 81-83°C. **IR** (KBr): $\tilde{\nu}$ = 3458, 3354, 2934, 1761, 1713, 1681, 1624, 1502, 1462, 1435, 1369, 1267, 1209, 1148, 1114, 1063, 1020, 916, 845, 798, 732 cm⁻¹. **¹H-NMR** (500 MHz, CDCl₃): δ 6.96 (d, J = 8.5 Hz, 1H), 6.65 (d, J = 8.5 Hz, 1H), 6.56 (br, 1H), 6.04 (t, J = 3.1 Hz, 1H), 5.28 (br, 1H), 4.01 (d, J = 16.2 Hz, 1H), 3.91 (s, 3H), 3.85 (s, 3H), 3.77 (s, 3H), 3.66 (d, J = 16.2 Hz, 1H), 3.15 (d, J = 17.0 Hz, 1H), 3.07-3.02 (m, 1H), 2.94-2.89 (m, 1H), 2.82 (d, J = 17.0 Hz, 1H), 2.19-2.14 (m, 2H), 1.73-1.67 (m, 2H), 1.61 (s, 9H), 1.24 (s, 3H), 1.14 (s, 3H) ppm. **¹³C-NMR** (125 MHz, CDCl₃): δ 177.6, 168.6, 166.1, 153.6, 149.6, 142.3, 136.7, 134.8, 133.5, 122.6, 120.2, 118.0, 107.1, 85.0, 60.6, 60.0, 56.2, 54.2, 51.8, 51.6, 47.6, 42.6, 27.8, 24.6, 23.4, 22.1, 17.3 ppm. **HRMS** (EI): Calc'd for C₃₀H₃₉N₃O₈⁺ [M]⁺: 569.2737. Found: 569.2738.

Oxindole (11): To a vigorously stirred solution of 11.28 g (19.8 mmol) Boc-protected oxindole **10** in 150 mL EtOAc was added 32 mL SnCl₄ (1M solution in CH₂Cl₂). After 3 min, the reaction was quenched with 150 mL 10% aq. KF solution and extracted twice with 400 mL EtOAc. The combined organic phases were washed with brine, dried (MgSO₄) and evaporated. The residue was chromatographed on silica gel (CH₂Cl₂/MeOH 20:1) and yielded 8.28 g (89%) of oxindole **11** as pale yellow foam.

11: **IR** (NaCl, film): $\tilde{\nu}$ = 3469, 3207, 2936, 2828, 1704, 1668, 1633, 1505, 1463, 1434, 1337, 1271, 1217, 1176, 1105, 1085, 1059, 733 cm⁻¹. **¹H-NMR** (500 MHz, CDCl₃): δ 9.85 (br, 1H), 7.05 (br, 1H), 6.91, 6.71 (br, 1H), 6.51 (d, J = 8.3 Hz, 1H), 6.08 (t, J = 3.7 Hz, 1H), 4.16 (d, J = 17.6 Hz, 1H), 3.87 (s, 3H), 3.84 (s, 3H), 3.71 (s, 3H), 3.60 (d, J = 17.6 Hz, 1H), 3.06 (d, J = 17.2 Hz, 1H), 2.92-2.83 (m, 2H), 2.83 (d, J = 17.2 Hz, 1H), 2.13-2.08 (m, 2H), 1.68-1.59 (m, 2H), 1.19 (s, 3H), 1.16 (s, 3H) ppm. **¹³C-NMR** (125 MHz, CDCl₃): δ 182.2, 168.7, 165.6, 152.7, 151.5, 141.9, 135.4, 135.0, 132.9, 122.5, 120.5, 117.5, 105.0, 60.8, 60.2, 55.8, 52.7, 52.6, 51.1, 48.5, 41.6, 24.3, 23.1, 21.4, 17.0 ppm. **HRMS** (EI): Calc'd for C₂₅H₃₁N₃O₆⁺ [M]⁺: 469.2213. Found: 469.2224.

Oxindole 13: To a 0°C cold solution of amide **11** (140 mg, 0.3 mmol, azeotropically dried twice with toluene) in 6 mL dry THF was added a 1.2 mL (2.2 equiv.) KHMDS in THF (freshly prepared, 0.55 M). After 1 h at 0°C, the cooling bath was removed and the mixture stirred for 15 h at r.t.. The reaction was quenched with 0.1 mL water and poured in 60 mL CH₂Cl₂/MeOH 10:1, dried (MgSO₄) and evaporated. The residue was washed with methanol (3x 2 mL) and acetone (2x 1 mL) to give pure **13** (140 mg, quant.) as colorless amorphous powder.

13: mp 250°C (dec.). **IR** (NaCl, film): $\tilde{\nu}$ = 3257, 3142, 2951, 2921, 2852, 1731, 1698, 1662, 1633, 1615, 1505, 1456, 1392, 1345, 1269, 1259, 1224, 1196, 1171, 1135, 1109, 1087, 796 cm⁻¹. **¹H-NMR** (500 MHz, CDCl₃): δ 8.17 (s, 1H), 7.31 (s, 1H), 6.82 (d, J = 8.4 Hz, 1H), 6.53 (d, J = 8.4 Hz, 1H), 6.01 (dd, J = 4.8, J = 3.7 Hz, 1H), 3.89 (s, 3H), 3.87 (s, 3H), 3.84 (s, 1H), 3.61 (s, 3H), 3.27 (d, J = 11.7 Hz, 1H), 3.03-2.98 (m, 1H), 2.94 (d, J = 11.2 Hz, 1H), 2.73 (td, J = 10.7, J = 2.8 Hz, 1H), 2.36 (d, J = 15.1 Hz, 1H), 2.21-2.12 (m, 3H), 1.88-1.79 (m, 2H), 1.34 (s, 3H), 0.88 (s, 3H) ppm. **¹³C-NMR** (125 MHz, DMSO, 50°C): δ 182.5, 170.8, 158.8, 152.3, 135.9, 135.5, 132.4, 121.2, 120.3, 106.0, 105.0, 61.1, 60.6, 60.1, 59.6, 55.6, 55.1, 50.8, 48.6, 47.8, 44.6, 22.7, 21.5, 20.7, 20.5 ppm. **Anal:** Calc'd for C₂₅H₃₁N₃O₆: C, 63.95; H, 6.65; N, 8.95. Found: C, 63.97; H, 6.47; N, 8.83.

PMB-protected oxindole 14: To a suspension of oxindole **13** (2.40 g, 5.11 mmol), 16.8 g (88 mmol) cesium carbonate and 1.90 g (5.15 mmol) tetrabutylammonium iodide in 1.8 L acetone was added 4.00 g (25.6 mmol) *p*-methoxybenzylchloride. The mixture was refluxed for 1 h, cooled, filtered and evaporated. The residue was purified by chromatography on silica gel (petrol ether/EtOAc 1:1 → EtOAc) and gave 2.85 g (95%) of the protected oxindole **14** as colorless gum.

14: **IR** (NaCl, film): $\tilde{\nu}$ = 3352, 2948, 2837, 1732, 1693, 1668, 1621, 1513, 1503, 1462, 1380, 1347, 1268, 1248, 1216, 1199, 1175, 1137, 1105, 1033, 981, 908, 732 cm⁻¹. **¹H-NMR** (500 MHz, CDCl₃): δ 7.31 (br, 1H), 7.26 (d, J = 8.5 Hz, 2H), 6.85 (d, J = 8.3 Hz, 1H), 6.82 (d, J = 8.5 Hz, 2H), 6.55 (d, J = 8.3 Hz, 1H), 5.99 (t, J = 4.9 Hz, 1H), 5.07 (d, J = 14.3 Hz, 1H), 5.02 (d, J = 14.3 Hz, 1H), 3.89 (s, 1H), 3.85 (s, 3H), 3.76 (s, 3H), 3.62 (s, 3H), 3.61 (s, 3H), 3.27 (d, J = 11.1 Hz, 1H), 3.03-2.98 (m, 1H), 2.93 (d, J = 11.1 Hz, 1H), 2.73 (td, J = 10.6 Hz, J = 2.6 Hz, 1H), 2.37, 2.19-2.14 (m, 2H), 2.07 (d, J = 14.9 Hz, 1H), 1.90-1.75 (m, 2H), 1.30 (s, 3H), 0.74 (s, 3H) ppm. **¹³C-NMR** (125 MHz, CDCl₃): δ 181.2, 171.9, 160.7, 158.6, 153.7, 136.1, 135.6, 134.0, 129.5, 128.6, 120.9, 120.4, 113.7, 108.6, 104.8, 61.8, 61.3, 59.8, 59.6, 56.2, 55.8, 55.1, 51.3, 49.4, 49.2, 46.6, 44.6, 23.1, 22.3, 21.5, 21.3 ppm. **HRMS** (EI): Calc'd for C₃₃H₃₉N₃O₇⁺ [M]⁺: 589.2788. Found: 589.2792.

Xanthate ester 15: Methyl ester **14** (2.80g, 4.75 mmol) was dried azeotropically with toluene (3x), dissolved in 120 mL dry CH_2Cl_2 and cooled to 0°C . 23.5 mL of a 1M solution of DIBAL in toluene (5 equiv.) was added slowly and the mixture was stirred for 15 min. 300 mL of a sat. aq. Rochelle salt solution was added and stirred vigorously for 1.5 h. The mixture was extracted twice with 400 mL EtOAc and the combined organic phases were washed with brine, dried (MgSO_4) and evaporated. The residue was chromatographed on silica gel ($\text{CH}_2\text{Cl}_2/\text{MeOH}$ 20:1) yielding 2.29 g (86%) of the primary alcohol as colourless foam which was directly used for the next step.

To a -78°C cold mixture of 2.29 g (4.08 mmol) of the primary alcohol in 80 mL dry THF and 6.3 mL carbondisulfide was added slowly 5.4 mL of a freshly prepared KHMDS-solution in THF (0.90M). After 15 min, the cooling bath was removed and the reaction mixture warmed up and stirred 1 h at r.t. 3.6 mL methyl iodide was added and stirring was continued for an additional hour. After quenching with water, the mixture was extracted with CH_2Cl_2 . The organic phase was dried (MgSO_4) and concentrated in *vacuo*. The residue was chromatographed on silica gel ($\text{CH}_2\text{Cl}_2 \rightarrow \text{CH}_2\text{Cl}_2/\text{acetone}$ 10:1) yielding 2.00 g (75%) of the xanthate ester **15** as yellow foam.

15: IR (NaCl, film): $\tilde{\nu} = 3351, 2933, 2835, 1694, 1667, 1620, 1513, 1462, 1380, 1349, 1306, 1267, 1246, 1215, 1176, 1105, 1067, 987 \text{ cm}^{-1}$. **$^1\text{H-NMR}$** (500 MHz, CDCl_3): δ 7.27 (d, $J = 8.8 \text{ Hz}$, 2H), 7.22 (br, 1H), 6.83 (d, $J = 8.8 \text{ Hz}$, 2H), 6.81 (d, $J = 8.4 \text{ Hz}$, 1H), 6.54 (d, $J = 8.4 \text{ Hz}$, 1H), 6.03 (t, $J = 4.3 \text{ Hz}$, 1H), 5.06 (s, 2H), 4.92 (dd, $J = 11.4 \text{ Hz}$, $J = 8.7 \text{ Hz}$, 1H), 4.55 (dd, $J = 11.4 \text{ Hz}$, $J = 5.0 \text{ Hz}$, 1H), 3.86 (s, 3H), 3.77 (s, 3H), 3.63 (s, 3H), 3.46 (dd, $J = 8.6 \text{ Hz}$, $J = 5.0 \text{ Hz}$, 1H), 3.08 (d, $J = 11.2 \text{ Hz}$, 1H), 3.01-2.96 (m, 2H), 2.85-2.79 (m, 1H), 2.51 (s, 3H), 2.32 (d, $J = 15.0 \text{ Hz}$, 1H), 2.20-2.15 (m, 2H), 2.14 (d, $J = 15.0 \text{ Hz}$, 1H), 1.95-1.80 (m, 2H), 1.04 (s, 3H), 0.80 (s, 3H) ppm. **$^{13}\text{C-NMR}$** (100 MHz, CDCl_3): δ 213.0, 181.6, 160.9, 158.9, 154.0, 136.3, 129.8, 128.8, 121.1, 120.7, 114.0, 109.7, 105.2, 94.6, 71.5, 68.2, 61.5, 60.6, 59.7, 56.1, 55.4, 54.7, 50.5, 48.6, 47.7, 44.9, 25.8, 23.2, 22.7, 21.9, 21.0, 18.7 ppm. **HRMS** (EI): Calc'd for $\text{C}_{34}\text{H}_{41}\text{N}_3\text{O}_6\text{S}_2^+ [\text{M}]^+$: 651.2437. Found: 651.2425.

Cyclized compound 19: A solution of xanthate ester **15** (200 mg, 0.307 mmol) in 350 mL dry benzene was degassed for 15 min with Argon. 8 μL (30 μmol) tributylstannane and 60 mg (0.368 mmol) AIBN were added and the mixture was immersed in a preheated oilbath. When the solvent began to reflux, a mixture of 8 μL (30 μmol) tributylstannane and 24 mg (0.150 mmol) AIBN in 50 mL benzene was added slowly over 1.5 h. The reaction was cooled after 2 h and evaporated. The residue was purified by chromatography on silica gel ($\text{CH}_2\text{Cl}_2/\text{acetone}$ 10:1 $\rightarrow$ $\text{CH}_2\text{Cl}_2/\text{MeOH}$ 20:1) yielding 101 mg (61%) of the cyclized compound **19** as colourless glass.

19: IR (NaCl, film): $\tilde{\nu} = 3324, 2933, 2836, 1691, 1673, 1616, 1513, 1500, 1462, 1378, 1344, 1266, 1247, 1176, 1118, 1104, 1036, 982, 901, 809, 733 \text{ cm}^{-1}$. **$^1\text{H-NMR}$** (500 MHz, CDCl_3): δ 7.25 (d, $J = 8.8 \text{ Hz}$, 2H), 6.87 (d, $J = 8.2 \text{ Hz}$, 1H), 6.80 (d, $J = 8.8 \text{ Hz}$, 2H), 6.60 (br, 1H), 5.53 (d, $J = 8.2 \text{ Hz}$, 1H), 5.96 (ddd, $J = 10.0 \text{ Hz}$, $J = 4.7 \text{ Hz}$, $J = 2.6 \text{ Hz}$, 1H), 5.76 (ddd, $J = 10.0 \text{ Hz}$, $J = 2.4 \text{ Hz}$, $J = 1.6 \text{ Hz}$, 1H), 5.07 (d, $J = 14.9 \text{ Hz}$, 1H), 4.98 (d, $J = 14.9 \text{ Hz}$, 1H), 3.86 (s, 3H), 3.76 (s, 3H), 3.71 (br d, $J = 10.6 \text{ Hz}$, 1H),

3.60 (s, 3H), 3.25 (ddd, $J = 10.5$ Hz, $J = 10.4$ Hz, $J = 1.6$ Hz, 1H), 2.72 (dd, $J = 8.4$ Hz, $J = 3.6$ Hz, 2H), 2.64 (dd, $J = 10.8$ Hz, $J = 1.7$ Hz, 1H), 2.42-2.32 (m, 1H), 2.22 (d, $J = 15.2$ Hz, 1H), 2.11-2.04 (m, 1H), 1.98 (d, $J = 15.2$ Hz, 1H), 1.85-1.68 (m, 2H), 1.07 (s, 3H), 0.70 (s, 3H) ppm. $^{13}\text{C-NMR}$ (125 MHz, CDCl_3) δ : 181.6, 175.5, 158.6, 153.5, 135.5, 133.8, 129.8, 128.7, 128.6, 127.6, 127.5, 122.8, 120.5, 113.7, 113.7, 113.6, 104.8, 61.7, 61.6, 61.3, 61.1, 60.8, 55.8, 55.1, 54.1, 49.2, 46.7, 44.6, 40.8, 29.8, 26.4, 24.0, 20.7 ppm. **HRMS** (EI): Calc'd for $\text{C}_{32}\text{H}_{37}\text{N}_3\text{O}_5^+ [\text{M}]^+$: 543.2733. Found: 543.2727.

Saturated hexacycle 20: Alkene **19** (843 mg, 1.55 mmol) was dissolved in 20 mL dry CH_2Cl_2 and transferred to a high pressure hydrogenation apparatus. 185 mg (0.23 mmol) Crabtree's catalyst were added, the apparatus purged with Nitrogen and pressurized with 50 bar Hydrogen. After 5 h at r.t., the pressure was released and the solvent evaporated. The residue was chromatographed twice on silica gel ($\text{CH}_2\text{Cl}_2/\text{acetone}$ 10:1 $\rightarrow$ $\text{CH}_2\text{Cl}_2/\text{MeOH}$ 20:1 and EtOAc/MeOH 10:1) to yield 753 mg (89%) of the reduced compound **19** as colourless foam.

19: **IR** (NaCl, film): $\tilde{\nu} = 3292, 3203, 2936, 2869, 2836, 1673, 1616, 1513, 1499, 1463, 1377, 1344, 1268, 1247, 1176, 1118, 1035, 909, 732 \text{ cm}^{-1}$. $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ 7.21 (d, $J = 8.7$ Hz, 2H), 6.84 (d, $J = 8.3$ Hz, 1H), 6.81 (br, 1H), 6.77 (d, $J = 8.7$ Hz, 2H), 6.50 (d, $J = 8.3$ Hz, 1H), 5.03 (d, $J = 14.9$ Hz, 1H), 4.93 (d, $J = 14.9$ Hz, 1H), 3.81 (s, 3H), 3.70 (s, 3H), 3.69 (d, $J = 10.7$ Hz, 1H), 3.57 (s, 3H), 3.12 (t, $J = 10.1$ Hz, 1H), 2.64 (d, $J = 11.5$ Hz, 1H), 2.44-2.35 (m, 2H), 2.19 (d, $J = 15.1$ Hz, 1H), 2.07 (d, $J = 13.2$ Hz, 1H), 1.93 (d, $J = 15.1$ Hz, 1H), 1.81-1.72 (m, 1H), 1.62 (dd, $J = 10.9$ Hz, $J = 3.9$ Hz, 1H), 1.57-1.47 (m, 3H), 1.29 (td, $J = 16.2$ Hz, $J = 3.2$ Hz, 1H), 1.02 (s, 3H), 0.64 (s, 3H) ppm. $^{13}\text{C-NMR}$ (100 MHz, CDCl_3): δ 181.6, 176.8, 158.4, 153.4, 135.4, 133.7, 129.7, 128.5, 122.6, 120.4, 113.5, 104.7, 61.3, 61.1, 61.1, 61.0, 61.0, 55.7, 55.0, 54.4, 54.0, 46.5, 44.5, 40.4, 31.6, 30.8, 25.7, 23.8, 20.6, 20.4 ppm. **HRMS** (EI): Calc'd for $\text{C}_{32}\text{H}_{39}\text{N}_3\text{O}_5^+ [\text{M}]^+$: 545.2890. Found: 545.2881.

Oxindole 21: Trifluoroacetic acid (30 mL) was added to a mixture of 480 mg (0.88 mmol) oxindole **20** and 3 mL anisole. The reaction was refluxed for 35 h under Nitrogen, cooled and evaporated. The residue taken up in 60 mL CH_2Cl_2 , washed with sat. aq. NaHCO_3 , dried (MgSO_4) and evaporated. The residue was chromatographed on silica gel ($\text{CH}_2\text{Cl}_2 \rightarrow \text{CH}_2\text{Cl}_2/\text{acetone}$ 6:1 $\rightarrow \text{CH}_2\text{Cl}_2/\text{MeOH}$ 20:1) giving 341 mg (91%) of the deprotected oxindole **21** as colorless solid, which was recrystallized from $\text{EtOAc}/\text{CH}_2\text{Cl}_2/\text{MeOH}$ 10:2:1.

21: mp: 290°C (dec.). **IR** (NaCl, film): $\tilde{\nu} = 3207, 2940, 2868, 1688, 1661, 1634, 1506, 1463, 1344, 1276, 1228, 1197, 1097, 1088, 728 \text{ cm}^{-1}$. $^1\text{H-NMR}$ (500 MHz, CDCl_3): δ 8.16 (br, 1H), 6.98 (br, 1H), 6.84 (d, $J = 8.3$ Hz, 1H), 6.52 (d, $J = 8.3$ Hz, 1H), 3.90 (s, 3H), 3.87 (s, 3H), 3.71 (d, $J = 10.8$ Hz, 1H), 3.08 (ddd, $J = 10.5$ Hz, $J = 10.4$ Hz, $J = 1.4$ Hz, 1H), 2.68 (d, $J = 11.5$ Hz, 1H), 2.46 (dd, $J = 11.3$ Hz, $J = 1.7$ Hz, 2H), 2.26 (d, $J = 15.3$ Hz, 1H), 2.15-2.04 (m, 1H), 1.95 (d, $J = 15.3$ Hz, 1H), 1.86-1.76 (m, 1H), 1.72-1.52 (m, 5H), 1.34 (ddd, $J = 13.3$ Hz, $J = 13.2$ Hz, $J = 4.3$ Hz, 1H), 1.10 (s, 3H), 0.82 (s, 3H). ppm. $^{13}\text{C-NMR}$

(100 MHz, CDCl₃): δ 182.9, 177.3, 152.6, 135.2, 132.6, 122.8, 120.8, 105.0, 62.7, 61.3, 61.2, 60.9, 55.9, 54.5, 54.2, 46.1, 40.6, 31.7, 30.9, 25.8, 23.8, 20.7, 20.4 ppm. **HRMS** (EI): Calc'd for C₂₄H₃₁N₃O₄⁺ [M]⁺: 425.2315. Found: 425.2995.

Prenylated compound 22: To an ice-cold solution of oxindole **21** (103 mg, 0.24 mmol) in 5 mL dry CH₂Cl₂ was added slowly 1.45 mL boron tribromide (1 M in CH₂Cl₂). The resulting suspension was stirred vigorously 3 h at 0°C, before it was quenched with 5 mL MeOH and stirred for 10 min at the same temperature. All volatiles were removed and the residue was redissolved in 5 mL MeOH. 5 mL Water were added and the mixture was concentrated again in *vacuo*. To the residue which was suspended in 20 mL CH₂Cl₂/MeOH 5:1, were added 1.5 g polymer bound diethanolamine (DEAM-PS, 0.9 mmol/g)¹ and the mixture was gently shaken for 30 min. The resin was filtered off, washed five times with 5 mL CH₂Cl₂/MeOH 5:1. The combined washings were concentrated and yielded 97 mg (quant.) of the crude catechol which was used without further purification. Catechol (97 mg, 0.24 mmol), cesium carbonate (236 mg, 0.73 mmol) and potassium iodide (80 mg, 0.49 mmol) were suspended in 10 mL acetone. 72 mg (0.49 mmol) prenyl bromide followed by 1 mL water were added and the suspension stirred for 30 min. The reaction mixture was poured into 200 mL EtOAc and washed twice with 20 mL water and subsequently with brine. The organic layer was dried (Na₂SO₄) and concentrated in *vacuo*. The residue was purified by chromatography on silica gel (CH₂Cl₂/MeOH 20:1) yielding 70 mg of the desired monoprenylated compound **22** (62% over both steps) as faint pink foam.

22: IR (NaCl, film): $\tilde{\nu}$ = 3234, 2933, 2872, 1702, 1694, 1651, 1633, 1505, 1463, 1335, 1287, 1196, 1112, 1055, 911, 805, 732 cm⁻¹. **¹H-NMR** (500 MHz, CDCl₃): δ 9.51 (br, 1H), 8.04 (br, 1H), 6.77 (d, *J* = 8.2 Hz, 1H), 6.55 (d, *J* = 8.2 Hz, 1H), 5.58-5.52 (m, 1H), 4.58 (d, *J* = 7.4 Hz, 2H), 3.71 (br d, *J* = 11.2 Hz, 1H), 3.12 (t, *J* = 11.2 Hz, 1H), 2.69 (br d, *J* = 11.2 Hz, 1H), 2.49-2.32 (m, 3H), 2.11 (d, *J* = 12.1 Hz, 1H), 1.92 (d, *J* = 15.1 Hz, 1H), 1.89-1.54 (m, 6H), 1.77 (s, 3H), 1.63 (s, 3H), 1.41-1.29 (m, 1H), 1.12 (s, 3H), 0.81 (s, 3H) ppm. **¹³C-NMR** (125 MHz, CDCl₃): δ 183.8, 177.5, 149.5, 140.2, 134.9, 130.1, 122.0, 121.5, 119.6, 108.6, 69.9, 62.5, 61.2, 61.1, 60.7, 54.5, 54.3, 45.9, 40.9, 31.5, 30.8, 25.8, 25.7, 23.8, 20.6, 20.5, 18.0 ppm. **HRMS** (EI): Calc'd for C₂₇H₃₅N₃O₄⁺ [M]⁺: 465.2628. Found: 465.2604.

Marcfortine B (1): Freshly purified *m*CPBA (25.6 mg, 0.15 mmol) was dissolved in 1 mL CHCl₃ and cooled to 0°C. Alkene **22** (20.0 mg, 43 μ mol) was dissolved in 1 mL CHCl₃ (0.5 mL additional CHCl₃ to rinse) and added to the *m*CPBA solution. To mixture was slowly warmed to r.t. over night. 1 mL of 10% aq. NaHSO₃ was added to reduce the formed *N*-oxide and the mixture was stirred for 30 min vigorously.

¹ M. Gravel, K. A. Thompson, M. Zak, C. Bérubé, D. G. Hall *J. Org. Chem.* **2002**, 67, 3-15.

The solution was extracted 5 times with 7 mL EtOAc. The combined organic phases were washed twice with 3 mL sat. aq. NaHCO₃. The aqueous washes were back extracted twice with 7 mL EtOAc. The combined organic phases were washed with 5 mL brine, dried over Na₂SO₄ and concentrated in *vacuo* and used immediately for the next step.

Neat Tin tetrachloride (60 µL, 512 µmol) was added dropwise to 6 mL dry dioxane. When the turbid solution cleared up, a solution of the crude epoxides in 1.5 mL dry THF was added and the reaction mixture was stirred for 20 min. The reaction was quenched with 10 mL 10% aq. KF-solution and extracted twice with 50 mL EtOAc. The combined organic layers were washed with brine, dried (Na₂SO₄) and evaporated. The residue was filtered over silica gel (CH₂Cl₂/MeOH 20:1) and used directly for the next step.

A solution of 90 mg (222 µmol) methyltriphenoxyposphonium iodide (thoroughly washed with dry ether before use) in 1 mL freshly distilled dry DMPU was added to the above cyclised compound. The mixture was stirred under exclusion of light for 24 h at r.t.. After quenching the reaction with 1 mL methanol, 50 mL EtOAc were added and the solution washed with 10 mL sat. aq. Na₂S₂O₃, 3 times with 10 mL water and 10 mL brine. The organic phase was dried (Na₂SO₄), evaporated and purified by silica gel chromatography (CH₂Cl₂ -> CH₂Cl₂/MeOH 20:1). The fractions containing **1** were purified further by preparative TLC (CH₂Cl₂/MeOH: 25:1) and finally crystallized from EtOAc/CH₂Cl₂ (5:1) to yield 8.4 mg (42% over 3 steps) of synthetic marcfortine B (**1**).

1: mp 220°C (dec.). **IR** (NaCl, film): $\tilde{\nu}$ = 3232, 1702, 1670, 1633, 1592, 1503, 1467, 1382, 1366, 1331, 1289, 1233, 1200, 1146, 1120, 1049 cm⁻¹. **¹H-NMR** (500 MHz, DMSO): δ 10.54 (s, 1H), 8.51 (s, 1H), 6.98 (d, *J* = 8.1 Hz, 1H), 6.60 (d, *J* = 8.1 Hz, 1H), 6.35 (d, *J* = 7.6 Hz, 1H), 4.97 (d, *J* = 7.6 Hz, 1H), 3.61 (d, *J* = 11.3 Hz, 1H), 2.81 (t, *J* = 10.5 Hz, 1H), 2.54 (d, *J* = 11.7 Hz, 1H), 2.28 (d, *J* = 14.9 Hz, 1H), 2.23-2.12 (m, 2H), 1.90 (d, *J* = 12.7 Hz, 1H), 1.85 (d, *J* = 14.9 Hz, 1H), 1.72-1.60 (m, 1H), 1.54 (dd, *J* = 12.4, 11.3 Hz, 1H), 1.48-1.39 (m, 3H), 1.36 (s, 3H), 1.34 (s, 3H), 1.23-1.13 (m, 1H), 0.96 (s, 3H), 0.69 (s, 3H) ppm. **¹³C-NMR** (125 MHz, DMSO): δ 182.2, 173.9, 145.6, 138.9, 135.0, 133.5, 125.8, 120.9, 116.1, 115.1, 79.2, 61.9, 61.0, 59.8, 59.7, 53.3, 45.3, 38.3, 31.0, 30.7, 29.5, 29.1, 25.4, 24.1, 20.5, 20.3 ppm.

Comparison of the ^{13}C -NMR spectra of synthetic and natural marcfortine B measured on the same 400Mhz spectrometer in DMSO

Synthetic ($\pm$)-marcfortine B	Natural (-)-marcfortine B	Δ ppm
182.19	182.19	0
173.89	173.89	0
145.58	145.57	-0.01
138.93	138.93	0
134.96	134.96	0
133.46	133.45	-0.01
125.79	125.79	0
120.90	120.89	-0.01
116.08	116.08	0
115.08	115.07	-0.01
79.22	79.22	0
61.87	61.87	0
61.02	61.02	0
59.78	59.78	0
59.74	59.74	0
53.79	53.79	0
53.31	53.31	0
45.34	45.34	0
38.34	38.32	-0.02
30.99	31.00	+0.01
30.74	30.74	0
29.51	29.51	0
29.13	29.13	0
25.44	25.45	+0.01
24.06	24.06	0
20.53	20.53	0
20.30	20.30	0

Natural (-)-Marcfortine B

Synthetic (±)-Marcfortine B

S13

HB-11-17
Pulse Sequence: s2pu1
Solvent: CDCl3
Acq. time 4.555 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7485710 MHz
DATA PROCESSING
F1 size 131072
Total time 2 min, 8 sec

HB-11-17-13C
Pulse Sequence: s2pu1
Solvent: CDCl3
Acq. time 1.555 sec
Width 31421.8 Hz
655 repetitions
OBSERVE C13, 125.6618804 MHz
DECOUPLE H1, 499.7505695 MHz
Power 42.00
WALTZ16 irradiated
DATA PROCESSING
Line broadening 2.0 Hz
F1 size 131072
Total time 55604 hr, 37 min, 4 sec

S14

H0-II-31-mix
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 INOVA-500 "q1500"
 Pulse: 60.0 degrees
 Acq. time: 4.000 sec
 Width: 8000.0 Hz
 32 repetitions
 OBSERVE: H1, 299.7485617 MHz
 DATA PROCESSING
 FT size: 131072
 Total time: 2 min, 8 sec

H0-II-31-mix-13C
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 User: 1-15-87
 INOVA-500 "q1500"
 Relax. delay: 0.500 sec
 Pulse: 33.3 degrees
 Acq. time: 1.500 sec
 Width: 31401.0 Hz
 33 repetitions
 OBSERVE: C13, 125.661852 MHz
 DECOUPLE: H1, 499.7505605 MHz
 Power: 42 dB
 CONTINUOUSLY ON
 WALTZ16-SCULPTED
 DATA PROCESSING
 FT size: 131072
 Total time: 55804 hr, 37 min, 4 sec

S16

HS-V-20
Pulse Sequence: zgpg30
Solvent: CDCl3
Ambient temperature
INNOVA-500 "400.130"
Pulse: 60.0 degrees
Acq. time: 1.000 sec
Width: 8000.0 Hz
276 repetitions
OBSERVE H1: 499.7485689 MHz
DATA PROCESSING
F1 size: 131072
Total time: 2 min, 8 sec

HS-V-20-130
Pulse Sequence: zgpg30
Solvent: CDCl3
Ambient temperature
User: J-15-07
INNOVA-500 "400.130"
Relax. delay: 0.500 sec
Pulse: 33.3 degrees
Acq. time: 1.500 sec
Width: 31421.8 Hz
276 repetitions
OBSERVE C13: 125.6618823 MHz
NUC1: 13C-125.6618823 MHz
Power: 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening: 2.0 Hz
F1 size: 131072
Total time: 55.64 hr, 37 min, 4 sec

S23