Rhodium-Catalyzed Intramolecular Conjugate Addition of Vinylstannanes to 2,3-Dihydro-4-pyridones. An Efficient Route to Stereoselective Construction of Indolizidines

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General. Column chromatography was performed on silica gel, Merck grade 60 (230-400 mesh). TLC plates were visualized with UV and staining with phosphomolybdic acid. ^1H NMR spectra were recorded on a Bruker AM500 (500MHz) spectrometer and the chemical shifts are reported in ppm with TMS as an internal standard ($\delta = 0$ ppm). ^{13}C NMR spectra were recorded at 125 MHz on a Bruker AM500 spectrometer. The chemical shifts are reported in ppm relative to the center line of the triplet at 77.0 ppm for CDCl_3 . Infrared (IR) spectra were measured with a Perkin Elmer FT-IR-1600 infrared spectrophotometer. High resolution mass spectra were taken on a Mariner PerSeptive Biosystems mass spectrometer with time-of-flight (TOF) detector. Unless stated otherwise, all reagents and solvents were purchased from commercial sources and used without additional purification. The following known compounds were prepared by literature procedures: (Z)-3-(trimethylstannyl)-allylamine,¹ 1-methoxy-3-trimethylsilyloxy-1,3-butadiene,² $[\text{Rh}(\text{cod})(\text{MeCN})_2]\text{BF}_4$.³

General Procedure for the Synthesis of Imines with (Z)-3-(Trimethylstannyl)-allylamine

To a solution of (Z)-3-(trimethylstannyl)-allylamine (1 mmol) in 10 mL of CH_2Cl_2 was added 1 mmol of the corresponding aldehyde and 200 mg of activated 4 Å molecular sieves. The mixture was stirred for 12 h at ambient temperature and filtered through a Celite pad and the solvent was evaporated in vacuo. The resulting imines were directly used in the subsequent reactions without further purification.

General Procedure for the aza-Diels-Alder Reaction of Danishefsky's Diene with Imines

To a solution of 1.0 mmol of the respective imine in MeCN (10 mL) was added $\text{Yb}(\text{OTf})_3$ (0.1 mmol) followed by 1.2 equiv of the diene **4** (1.2 mmol). The reaction mixture was stirred for 6-12 h at room temperature. After addition of saturated NaHCO_3 (10 mL), the aqueous phase was extracted with CH_2Cl_2 (10 mL $\times$ 3), the combined organic phases were dried with MgSO_4 , and the solvent was evaporated in vacuo. The crude material was purified by flash chromatography on silica gel with Et_2O -hexane mixtures as eluents.

Analytical data for **2-phenyl-1-(3-trimethylstannanyl-allyl)-2,3-dihydro-1H-pyridin-4-one**: Chromatography (80% Et_2O /hexane) afforded 0.327 g (87%) of a yellow oil; 500-MHz ^1H NMR (CDCl_3) δ 7.35-7.23 (m, 5H), 7.16 (d, $J = 7.7$ Hz, 1 H), 6.40 (dq, $J = 12.8, 5.2$ Hz, 1 H), 6.13 (d, $J = 12.8$ Hz, 1 H), 5.03 (d, $J = 7.7$ Hz, 1 H), 4.58 (m, 1 H), 3.71 (ddd, $J = 14.8, 5.2, 1.6$ Hz, 1H), 3.55 (ddd, $J = 14.8, 7.9, 0.9$ Hz, 1H), 2.86 (dd, $J = 16.4, 7.0$ Hz, 1H), 2.64 (dd, $J = 16.4, 7.8$ Hz, 1H), 0.01 (s, 9H); 125-MHz ^{13}C NMR (CDCl_3) δ 190.2, 153.1, 142.1, 138.7, 136.1, 129.0, 128.2, 126.8, 99.0, 61.4, 57.3, 43.9, -8.7; IR (neat) cm^{-1} 1642, 1591; Anal. calcd for $\text{C}_{17}\text{H}_{23}\text{NOSn}$: C, 54.29; H, 6.16; N, 3.72. Found: C, 54.46; H, 6.10; N, 3.50.

Analytical data for **2-*p*-tolyl-1-(3-trimethylstannanyl-allyl)-2,3-dihydro-1H-pyridin-4-one**: Chromatography (80% Et₂O/hexane) afforded 0.359 g (92%) of a yellow oil; 500-MHz ¹H NMR (CDCl₃) δ 7.19-7.14 (m, 4H), 7.16 (d, J = 7.7 Hz, 1 H), 6.41 (dq, J = 12.8, 5.2 Hz, 1 H), 6.14 (d, J = 12.8 Hz, 1 H), 5.05 (d, J = 7.7 Hz, 1 H), 4.57 (dd, J = 8.6, 6.8 Hz, 1 H), 3.70 (ddd, J = 14.8, 5.2, 1.6 Hz, 1H), 3.55 (ddd, J = 14.8, 7.7, 1.1 Hz, 1H), 2.83 (dd, J = 16.4, 6.8 Hz, 1H), 2.67 (dd, J = 16.4, 8.6 Hz, 1H), 2.34 (s, 3H), 0.05 (s, 9H); 125-MHz ¹³C NMR (CDCl₃) δ 190.5, 153.2, 142.3, 138.1, 136.0, 135.7, 129.7, 126.8, 99.1, 61.5, 57.2, 44.2, 21.1, -8.7; IR (neat) cm⁻¹ 1645, 1591; HRMS calcd. for C₁₈H₂₅NOSn 391.09581, found 391.09414.

Analytical data for **2-(4-methoxy-phenyl)-1-(3-trimethylstannanyl-allyl)-2,3-dihydro-1H-pyridin-4-one**: Chromatography (70% Et₂O/hexane) afforded 0.345 g (85%) of a yellow oil; 500-MHz ¹H NMR (CDCl₃) δ 7.21 (d, 2H), 7.15 (d, J = 7.7 Hz, 1 H), 6.88 (d, 2H), 6.41 (dq, J = 12.8, 5.2 Hz, 1 H), 6.15 (d, J = 12.8 Hz, 1 H), 5.06 (d, J = 7.7 Hz, 1 H), 4.56 (dd, J = 8.3, 6.9 Hz, 1 H), 3.80 (s, 3H), 3.69 (ddd, J = 14.9, 5.3, 1.5 Hz, 1H), 3.55 (dd, J = 14.9, 7.7 Hz, 1H), 2.82 (dd, J = 16.4, 6.9 Hz, 1H), 2.67 (dd, J = 16.4, 8.4 Hz, 1H), 0.06 (s, 9H); 125-MHz ¹³C NMR (CDCl₃) δ 190.6, 159.6, 153.1, 142.3, 135.9, 130.8, 128.1, 114.5, 99.0, 61.2, 57.1, 55.3, 44.2, -8.7; IR (neat) cm⁻¹ 1647, 1576; HRMS calcd. for C₁₈H₂₅NO₂Sn 407.0907, found 407.0888.

Analytical data for **2-(4-chloro-phenyl)-1-(3-trimethylstannanyl-allyl)-2,3-dihydro-1H-pyridin-4-one**: Chromatography (80% Et₂O/hexane) afforded 0.369 g (90%) of a yellow oil; 500-MHz ¹H NMR (CDCl₃) δ 7.31 (m, 2H), 7.21 (m, 2H), 7.16 (d, J = 7.7 Hz, 1 H), 6.40 (dq, J = 12.8, 5.2 Hz, 1 H), 6.16 (d, J = 12.8 Hz, 1 H), 5.05 (d, J = 7.7 Hz, 1 H), 4.58 (t, J = 7.2 Hz, 1 H), 3.73 (ddd, J = 14.8, 5.2, 1.5 Hz, 1H), 3.55 (ddd, J = 14.8, 7.8, 0.9 Hz, 1H), 2.88 (dd, J = 16.4, 7.1 Hz, 1H), 2.59 (dd, J = 16.4, 7.3 Hz, 1H), 0.04 (s, 9H); 125-MHz ¹³C NMR (CDCl₃) δ 189.7, 152.9, 141.9, 137.2, 136.5, 134.1, 129.3, 128.2, 99.3, 60.7, 57.5, 43.7, -8.7; IR (neat) cm⁻¹ 1654, 1566; HRMS calcd. for C₁₇H₂₂NONaClSn (M+Na) 434.0304, found 434.0288.

Analytical data for **2-furan-2-yl-1-(3-trimethylstannanyl-allyl)-2,3-dihydro-1H-pyridin-4-one**: Chromatography (80% Et₂O/hexane) afforded 0.285 g (78%) of a yellow oil; 500-MHz ¹H NMR (CDCl₃) δ 7.40 (d, J = 2.0 Hz, 1H), 7.01 (d, J = 7.7 Hz, 1 H), 6.47 (dq, J = 12.9, 5.8 Hz, 1 H), 6.35 (dd, J = 2.0, 3.3 Hz, 1 H), 6.30 (d, J = 3.3 Hz, 1 H), 6.23 (d, J = 12.9 Hz, 1 H), 5.03 (d, J = 7.7 Hz, 1 H), 4.68 (dd, J = 6.8, 5.1 Hz, 1 H), 3.91-3.81 (m, 2H), 2.89 (dd, J = 16.4, 6.9 Hz, 1H), 2.76 (dd, J = 16.4, 5.1 Hz, 1H), 0.15 (s, 9H); 125-MHz ¹³C NMR (CDCl₃) δ 190.2, 151.6, 151.2, 142.5, 142.2, 136.6, 110.4, 108.5, 98.5, 58.3, 54.3, 39.9, -8.6; IR (neat) cm⁻¹ 1641, 1582; HRMS calcd. for C₁₅H₂₂NO₂Sn (M+H) 368.0667, found 368.0681.

Analytical data for **2-benzyloxymethyl-1-(3-trimethylstannanyl-allyl)-2,3-dihydro-1H-pyridin-4-one**: Chromatography (70% Et₂O/hexane) afforded 0.374 g (89%) of a yellow oil; 500-MHz ¹H NMR (CDCl₃) δ 7.36-7.25 (m, 5H), 6.97 (dd, J = 7.5, 1.1 Hz, 1 H), 6.44 (dq, J = 12.9, 5.9 Hz, 1 H), 6.16 (d, J = 12.9 Hz, 1 H), 4.89 (dd, J = 7.5, 0.9 Hz, 1 H), 4.49 (s, 2 H), 3.97-3.85 (m, 2 H), 3.83 (dd, J = 9.4, 7.9 Hz, 1 H), 3.78-3.72 (m, 1H), 3.46 (dd, J = 9.4, 5.1 Hz, 1H), 2.77 (dd, J = 16.7, 7.3 Hz, 1H), 2.36 (ddd, J = 16.7, 2.5, 1.0 Hz, 1 H), 0.18 (s, 9H); 125-MHz ¹³C NMR (CDCl₃) δ 190.1, 151.4, 142.9, 137.6, 136.2, 128.5, 127.8, 127.8, 98.1, 73.6, 68.5, 59.5, 56.5, 37.7, -8.6; IR (neat) cm⁻¹ 1642, 1586; HRMS calcd. for C₁₉H₂₇NO₂Sn 421.10638, found 421.10461.

Analytical data for **2-isopropyl-1-(3-trimethylstannanyl-allyl)-2,3-dihydro-1H-pyridin-4-one**: Chromatography (60% Et₂O/hexane) afforded 0.236 g (69%) of a yellow oil; 500-MHz ¹H NMR (CDCl₃) δ 7.01 (d, J = 7.4 Hz, 1 H), 6.45 (dq, J = 12.8, 5.4 Hz, 1 H), 6.19 (d, J = 12.8 Hz, 1 H), 4.89 (d, J = 7.4 Hz, 1 H), 3.87 (ddd, J = 15.0, 5.4, 1.5 Hz, 1 H), 3.79 (ddd, J = 15.0, 7.6, 1.0 Hz, 1 H), 3.26 (m, 1 H), 2.67 (dd, J = 16.6, 7.6 Hz, 1H), 2.43 (dd, J = 16.6, 3.9 Hz, 1H), 2.22 (m, 1H), 0.95 (d, J = 6.8 Hz, 6H) 0.25 (s, 9H); 125-MHz ¹³C NMR (CDCl₃) δ 191.1, 152.3, 142.9, 135.7, 97.6, 62.0, 59.0, 36.3, 29.0, 19.5, 17.7, -8.6; IR (neat) cm⁻¹ 1640, 1585; HRMS calcd. for C₁₄H₂₆NOSn (M + H) 344.1031, found 344.1045.

Analytical data for **2-cyclohexyl-1-(3-trimethylstannanyl-allyl)-2,3-dihydro-1H-pyridin-4-one**: Chromatography (50% Et₂O/hexane) afforded 0.275 g (72%) of a yellow oil; 500-MHz ¹H NMR (CDCl₃) δ 7.00 (d, J = 7.4 Hz, 1 H), 6.46 (dq, J = 12.6, 5.5 Hz, 1 H), 6.19 (d, J = 12.6 Hz, 1 H), 4.86 (d, J = 7.4 Hz, 1 H), 3.86 (ddd, J = 14.9, 5.5, 1.5 Hz, 1 H), 3.78 (ddd, J = 15.0, 8.6, 1.0 Hz, 1 H), 3.25 (m, 1 H), 2.70 (dd, J = 16.6, 7.8 Hz, 1H), 2.44 (dd, J = 16.6, 2.0 Hz, 1H), 1.85 (m, 1H), 1.80-1.54 (m, 6H), 1.25-1.10 (m, 4H) 0.25 (s, 9H); 125-MHz ¹³C NMR (CDCl₃) δ 191.0, 152.2, 142.9, 135.7, 97.4, 61.5, 59.3, 39.4, 37.0, 29.8, 28.5, 26.3, 26.1, -8.6; IR (neat) cm⁻¹ 1641, 1586; HRMS calcd. for C₁₇H₃₀NOSn (M + H) 384.13442, found 384.13354.

General Procedure for the Catalytic Intramolecular Conjugate Addition of Vinylstannanes to 2,3-Dihydro-4-pyridones. To a solution 0.5 mmol of the respective 2,3-dihydro-4-pyridone in 1,4-dioxane (4 mL) was added [RhCl(cod)]₂ (0.025 mmol). After stirring for 2-3 h at room temperature the reaction mixture was concentrated. The crude material was purified by flash chromatography on silica gel with Et₂O-hexane mixtures as eluents.

Analytical data for **5-phenyl-5,6,8,8a-tetrahydro-3H-indolizin-7-one**: Chromatography (30% Et₂O/hexane) afforded 0.089 g (84%) of a brown oil; 500-MHz ¹H NMR (CDCl₃) δ 7.41-7.25 (m, 5 H), 5.86 (dq, J = 6.2, 1.9 Hz, 1 H), 5.80 (dq, J = 6.2, 1.9 Hz, 1 H), 4.57 (m, 1 H), 4.15 (dd, J = 12.3, 2.9 Hz, 1 H), 3.77 (dq, J = 14.4, 1.9 Hz, 1 H), 3.41 (dddd, J = 14.4, 8.5, 4.4, 2.0 Hz, 1 H), 2.75 (m, 1H), 2.61-2.48 (m, 3 H); 125-MHz ¹³C NMR (CDCl₃) δ 209.8, 131.1, 130.1, 128.6, 127.6, 127.5, 126.9, 62.6, 62.5, 60.0, 48.7, 43.6; IR (neat) cm⁻¹ 2928, 1718; HRMS calcd. for C₁₄H₁₅NO 213.11536, found 213.11513.

Analytical data for **5-p-tolyl-5,6,8,8a-tetrahydro-3H-indolizin-7-one**: Chromatography (30% Et₂O/hexane) afforded 0.097 g (86%) of a brown oil; 500-MHz ¹H NMR (CDCl₃) δ 7.29-7.14 (m, 4 H), 5.85 (dq, J = 6.2, 1.9 Hz, 1 H), 5.79 (dq, J = 6.2, 2.1 Hz, 1 H), 4.55 (m, 1 H), 4.12 (dd, J = 12.2, 2.9 Hz, 1 H), 3.77 (dq, J = 14.5, 1.9 Hz, 1 H), 3.41 (dddd, J = 14.5, 8.6,

4.3, 2.1 Hz, 1 H), 2.72 (dd, $J = 17.4, 12.2$ Hz 1H), 2.57-2.48 (m, 3 H), 2.34 (s, 3 H); 125-MHz ^{13}C NMR (CDCl_3) δ 210.0, 140.1, 137.1, 130.1, 129.3, 127.7, 126.6, 62.4, 62.3, 60.0, 48.8, 43.7, 21.1; IR (neat) cm^{-1} 2920, 1716; HRMS calcd. for $\text{C}_{15}\text{H}_{17}\text{NO}$ 227.13101, found 213.13156.

Analytical data for **5-(4-methoxy-phenyl)-5,6,8,8a-tetrahydro-3H-indolizin-7-one**: Chromatography (30% Et_2O /hexane) afforded 0.099 g (82%) of a brown oil; 500-MHz ^1H NMR (CDCl_3) δ 7.32-6.86 (m, 4 H), 5.86 (m, 1 H), 5.79 (m, 1 H), 4.53 (m, 1 H), 4.11 (dd, $J = 12.1, 2.8$ Hz, 1 H), 3.81 (s, 3 H), 3.75 (dq, $J = 14.5, 2.0$ Hz, 1 H), 3.41 (dddd, $J = 14.5, 8.4, 4.2, 2.0$ Hz, 1 H), 2.71 (dd, $J = 17.3, 12.2$ Hz 1H), 2.58-2.46 (m, 3 H); 125-MHz ^{13}C NMR (CDCl_3) δ 210.1, 158.9, 135.1, 130.1, 127.9, 127.6, 114.1 62.4, 61.9, 60.0, 55.4, 48.8, 43.6; IR (neat) cm^{-1} 2943, 1717; HRMS calcd. for $\text{C}_{15}\text{H}_{17}\text{NO}_2$ 243.12593, found 243.12608.

Analytical data for **5-(4-chloro-phenyl)-5,6,8,8a-tetrahydro-3H-indolizin-7-one**: Chromatography (30% Et_2O /hexane) afforded 0.097 g (79%) of a brown oil; 500-MHz ^1H NMR (CDCl_3) δ 7.36-7.32 (m, 4 H), 5.86 (dq, $J = 5.9, 1.9$ Hz, 1 H), 5.79 (m, 1 H), 4.55 (m, 1 H), 4.13 (dd, $J = 12.3, 2.9$ Hz, 1 H), 3.76 (dq, $J = 14.3, 2.0$ Hz, 1 H), 3.37 (dddd, $J = 14.3, 8.6, 4.3, 2.0$ Hz, 1 H), 2.68 (dd, $J = 17.3, 12.3$ Hz 1H), 2.56-2.42 (m, 3 H); 125-MHz ^{13}C NMR (CDCl_3) δ 209.3, 141.6, 133.1, 130.1, 128.8, 128.2, 127.6, 62.5, 62.1, 60.0, 48.6, 43.6; IR (neat) cm^{-1} 1716; HRMS calcd. for $\text{C}_{14}\text{H}_{15}\text{ClNO}$ (M + H) 248.0837, found 248.0849.

Analytical data for **5-furan-2-yl-5,6,8,8a-tetrahydro-3H-indolizin-7-one**: Chromatography (50% Et_2O /hexane) afforded 0.087 g (86%) of a brown oil; 400-MHz ^1H NMR (CDCl_3) δ 7.38 (dd, $J = 1.8, 0.8$ Hz, 1 H), 6.32 (dd, $J = 3.3, 1.8$ Hz, 1 H), 6.19 (dt, $J = 3.3, 0.8$ Hz, 1 H), 5.89 (dq, $J = 6.0, 2.1$ Hz, 1 H), 5.78 (m, 1 H), 4.40 (dd, $J = 7.6, 5.1$ Hz, 1 H), 4.21 (m, 1 H), 3.85 (m, 1 H), 3.67 (m, 1 H), 2.85-2.72 (m, 2 H), 2.55 (ddd, $J = 15.5, 3.3, 0.8$ Hz, 1 H), 2.44 (dd, $J = 15.5, 11.9$ Hz, 1 H); 100-MHz ^{13}C NMR (CDCl_3) δ 208.6, 154.4, 142.2, 130.7, 127.9, 110.0, 107.1, 61.6, 58.1, 54.6, 44.8, 44.2; IR (neat) cm^{-1} 2922, 1716; HRMS calcd. for $\text{C}_{12}\text{H}_{13}\text{NO}_2$ 203.09463, found 203.09443.

Analytical data for **5-benzyloxymethyl-5,6,8,8a-tetrahydro-3H-indolizin-7-one**: Chromatography (30% Et₂O/hexane) afforded 0.115 g (90%) of a brown oil; 500-MHz ¹H NMR (CDCl₃) δ 7.37-7.26 (m, 5 H), 5.87 (dq, J = 6.2, 1.9 Hz, 1 H), 5.78 (dq, J = 6.2, 2.0 Hz, 1 H), 4.54 (s, 2 H), 4.38 (m, 1 H), 4.04 (dq, J = 14.0, 2.0 Hz, 1 H), 3.60 (m, 1 H), 3.55 (dd, J = 9.4, 4.6 Hz, 1 H), 3.44 (dd, J = 9.4, 5.9 Hz, 1H), 3.34 (m, 1 H), 2.62 (dd, J = 16.7, 3.8 Hz, 1 H), 2.50 (dd, J = 16.7, 10.1 Hz, 1 H), 2.41 (dd, J = 16.1, 3.3 Hz, 1 H), 2.31 (dd, J = 16.1, 12.0); 125-MHz ¹³C NMR (CDCl₃) δ 209.4, 138.0, 130.7, 128.4, 127.6, 127.5, 127.4, 73.4, 73.1, 62.6, 59.5, 57.8, 43.8, 42.3; IR (neat) cm⁻¹ 2928, 1722; HRMS calcd. for C₁₆H₁₉NO₂ 257.14158, found 257.14225.

Analytical data for **5-isopropyl-5,6,8,8a-tetrahydro-3H-indolizin-7-one**: Chromatography (30% Et₂O/hexane) afforded 0.072 g (80%) of a brown oil; 500-MHz ¹H NMR (CDCl₃) δ 5.88 (dq, J = 6.1, 1.8 Hz, 1 H), 5.78 (dq, J = 6.1, 1.9 Hz, 1 H), 4.34 (m, 1 H), 3.96 (dq, J = 13.9, 1.8 Hz, 1 H), 3.48 (dddd, J = 13.9, 8.6, 4.3, 2.1 Hz, 1 H), 2.92 (m, 1 H), 2.41-2.28 (m, 4 H), 1.84 (m, 1 H), 0.90 (d, J = 6.9, 6 H); 125-MHz ¹³C NMR (CDCl₃) δ 211.2, 130.8, 127.3, 63.5, 62.5, 59.5, 43.5, 39.4, 30.5, 16.0; IR (neat) cm⁻¹ 2957, 1721; HRMS calcd. for C₁₁H₁₇NO 179.13101, found 179.13041.

Analytical data for **5-cyclohexyl-5,6,8,8a-tetrahydro-3H-indolizin-7-one**: Chromatography (30% Et₂O/hexane) afforded 0.084 g (77%) of a brown oil; 500-MHz ¹H NMR (CDCl₃) δ 5.88 (dq, J = 6.2, 1.8 Hz, 1 H), 5.78 (dq, J = 6.2, 2.0 Hz, 1 H), 4.31 (m, 1 H), 3.96 (dq, J = 13.8, 1.8 Hz, 1 H), 3.52 (m, 1 H), 2.93 (m, 1 H), 2.42-2.40 (m, 2 H), 2.36-2.24 (m, 2 H), 1.90 (m, 1 H), 1.77 (m, 2 H), 1.67 (m, 2 H), 1.44 (m, 1 H), 1.28-1.07 (m, 3 H), 1.04-0.80 (m, 2 H); 125-MHz ¹³C NMR (CDCl₃) δ 211.2, 131.1, 127.4, 63.1, 62.4, 59.3, 43.8, 41.1, 40.5, 30.1, 27.4, 26.8, 26.7, 26.4; IR (neat) cm⁻¹ 2928, 1714; HRMS calcd. for C₁₄H₂₂NO (M+H) 220.16567, found 220.16545.

References

1. Corriu, R. J. P.; Geng, B.; Moreau, J. J. E. *J. Org. Chem.* **1993**, 58, 1443.
2. Hansson, L.; Carlson, R. *Acta Chem. Scand.* **1989**, 43, 188.
3. Green, M.; Kuc, T. A.; Taylor, S. H. *J. Chem. Soc. A* **1971**, 2334.

B. Furman
CHM MDZ 254
Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
File: chmdm2254
Mercury-400BB "SchliffSUN"

Relax. delay 1.000 sec
Pulse 33.3 degrees
Acq. time 4.736 sec
Width 4504.5 Hz
256 repetitions
OBSERVE H1, 400.0418311 MHz
DATA PROCESSING
FT size 65536

B. Furman
CHM MDZ 254
Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
File: chmdm2254c
Mercury-400BB "SchliffSUN"

Relax. delay 1.000 sec
Pulse 20.0 degrees
Acq. time 1.180 sec
Width 28248.6 Hz
17280 repetitions
OBSERVE C13, 100.5805998 MHz
DECOUPLE H1, 400.0440508 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536