Supporting Information

Ring closing metathesis/fragmentation route to (Z)-configured medium ring cycloalkenes. Total synthesis of (±)-Periplanone C

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General experimental

All reactions were performed in dried glassware under an inert (Ar) atmosphere. Petroleum-ether refers to the fraction with distillation range 70-90 °C. All chromatographic separations¹ were performed on Silica, 10-18, 60A, ICN Biomedicals. Standard techniques were used for the purification of reagents and solvents.² If not otherwise stated, ^1H NMR spectra were recorded at 200 MHz, ^{13}C NMR at 50 MHz, for samples in deuterated chloroform. Chemical shifts are expressed in ppm using tetramethylsilane as internal standard, coupling constants (J) are in Hz (In addition to listed spectral data, scanned ^1H - and ^{13}C NMR spectra of all compounds can be found at the end of the supporting information). IR spectra are expressed in cm^{-1} . Melting points were determined on a Kofler hot-stage apparatus and are uncorrected. Compounds **1a**,³ **1b**,³ **1c**⁴ and **1d**⁵ were all prepared according to the literature procedure.³ Compounds **2a**,⁶ **2b** and **2c** were prepared according to the literature procedure.⁶

Ethyl 2-hydroxy-1,2-bis(2-propenyl)cyclopentanecarboxylate (2a**)**⁶

Following the previously described procedure,⁶ this compound was obtained in 84% yield, as a mixture of isomers in a *trans*:*cis* = 13.1:1 ratio.

Ethyl 2-hydroxy-1,2-bis(2-propenyl)cyclohexanecarboxylate (2b**)**

Following the previously described procedure,⁶ this compound was obtained in 71% yield, as a mixture of isomers in a *trans*:*cis* = 1.6:1 ratio.⁷

Physical data for **2b**: Light-yellow oil. Anal. calcd. for C₁₅H₂₄O₃: C, 71.39; H, 9.59; found: C, 71.64; H 9.87. IR (film) 3486, 3076, 2978, 2945, 2868, 1732, 1697, 1640, 1457, 1391, 1372. ¹H NMR (mixture of diastereoisomers): 5.47-6.04 (m, 2 x 2H); 5.10 (d, *J*=8.1, 4H); 5.00 (d, *J*=7.5, 4H); 4.30 (bs, 1H), 4.18 (q, *J*=7.1, 2H), 4.17 (q, *J*=7.1, 2H); 2.12-2.90 (m, 2 x 4H + 1H); 1.35-2.02 (m, 2 x 8H); 1.29 (t, *J*=7.1, 2 x 3H). ¹³C NMR (mixture of diastereoisomers): 177.0 (C); 176.8 (C); 134.5 (CH); 134.0 (CH); 133.7 (CH); 133.6 (CH); 118.2 (CH₂); 117.6 (CH₂); 117.5 (CH₂); 117.3 (CH₂); 74.4 (C); 74.2 (C); 60.7 (2 x CH₂); 54.2 (C); 53.9 (C); 40.4 (CH₂); 39.5 (CH₂); 37.5 (CH₂); 34.1 (CH₂); 33.9 (CH₂); 30.1 (CH₂); 30.0 (CH₂); 26.3 (CH₂); 21.6 (2 x CH₂); 21.4 (CH₂); 19.9 (CH₂); 14.2 (CH₃); 14.1 (CH₃).

Ethyl 2-hydroxy-1-(3-butenyl)-2-(2-propenyl)cyclohexanecarboxylate (2c**)**

Following the previously described procedure,⁶ this compound was obtained in 81% yield, as a mixture of isomers in a ratio *trans*:*cis* = 1.68:1.⁷

Physical data for **2c**: Colorless oil. IR (film) 3486, 3075, 2945, 2867, 1697, 1641, 1458, 1392, 1372, 1282. ¹H NMR (mixture of diastereoisomers): 5.13-5.69 (m, 2 x 2H); 4.92-5.12 (m, 2 x 4H); 4.21 (q, *J*=7.1, 2H); 4.19 (q, *J*=7.1, 2H); 3.27 (s, 1H); 1.37-2.65 (m, 2 x 14H + 1H); 1.30 (t, 2 x 3H, *J*=7.1). ¹³C NMR (mixture of diastereoisomers): 177.4 (2 x C); 138.6 (CH); 138.2 (CH); 134.1 (CH); 133.8 (CH); 117.6 (CH₂); 117.5 (CH₂); 114.7 (CH₂); 114.5 (CH₂); 74.7 (2 x C); 60.7 (2 x CH₂); 54.2 (C); 53.5 (C); 40.4 (CH₂); 39.3 (CH₂); 34.2 (CH₂); 32.4 (CH₂); 30.3 (CH₂); 30.0 (CH₂); 29.5 (CH₂); 28.8 (CH₂); 28.4 (2 x CH₂); 26.1 (CH₂); 21.8 (CH₂); 21.7 (CH₂); 20.1 (CH₂); 14.2 (CH₃); 14.1 (CH₃). HRMS (ESI) Calcd. for C₁₆H₂₆O₃Na: 289.1780; found: 289.1797.

Ethyl *trans*-bicyclo[4.3.0]non-3-en-6-ol-1-carboxylate (3a**)**

To a solution of compound **2a** (350 mg; 1.47 mmol) in dichloromethane (7.8 mL), previously deaerated in an ultrasonic bath under a stream of argon, was added catalyst

5b (9.4 mg; 11 μ mol; 0.75 mol %) and the reaction mixture was heated to reflux under an argon atmosphere. The progress of the reaction was monitored by TLC. Upon completion the solvent was removed under reduced pressure and the residue was purified, first by dry-flash chromatography on silica (eluent: petroleum-ether:ethyl acetate=9:1), then by low pressure liquid chromatography (eluent: toluene:ethyl acetate=97:3), to give 280 mg (91%) of the title compound **3a**.⁷

Physical data for **3a**: Colorless oil. Anal. calcd. for $C_{12}H_{18}O_3$: C, 68.54; H, 8.63; found: C, 68.51; H 8.91. IR (film): 3530, 3026, 2977, 2909, 1723, 1640, 1447, 1372, 1308. 1H NMR: 5.74-5.81 (m, 1H); 5.59-5.68 (m, 1H); 4.14 (q, $J=7.0$, 2H); 2.62-2.84 (m, 2H); 2.08-2.30 (m, 3H); 1.61-2.01 (m, 6H); 1.22 (t, $J=7.0$, 3H). ^{13}C NMR: 175.6 (C); 126.8 (CH); 125.2 (CH); 80.0 (C); 60.2 (CH_2); 55.9 (C); 35.6 (CH_2); 35.2 (CH_2); 33.7 (CH_2); 31.6 (CH_2); 20.6 (CH_2); 14.0 (CH_3).

Ethyl *cis*- and *trans*-bicyclo[5.4.0]undec-4-en-7-ol-1-carboxylate (3c cis**) and (**3c trans**)**

To a solution of compound **2c** (637 mg; 2.4 mmol) in deaerated dichloromethane (14.6 mL), was added catalyst **5a** (59.1 mg; 72 μ mol) and the reaction mixture was stirred at rt for 20 h, under an argon atmosphere. An additional amount of catalyst **5a** (40 mg; 48 μ mol) was added, and the reaction mixture was stirred at rt for an additional 6 h. Lead tetraacetate (200 mg; 0.45 mmol) was added and the mixture was stirred at rt for 7 h. The suspension was filtered through a short pad of silica, the pad was washed with dichloromethane and ethyl acetate, the combined organic extract

was concentrated under reduced pressure and purified by dry-flash chromatography (eluent: toluene:ethyl acetate=97:3) to give 443 mg (76%) of the title compound **3c** as a mixture of diastereoisomers. The diastereoisomers were separated by low pressure liquid chromatography (eluent: petroleum-ether:ethyl acetate=95:5), to give 192 mg of **3c cis** and 250 mg of **3c trans**.⁷

Physical data for **3c cis**: Colorless oil. Anal. calcd. for C₁₄H₂₂O₃: C, 70.56; H, 9.30; found: C, 70.63; H 9.57. IR (film): 3505, 3026, 2936, 2865, 1699, 1449, 1398, 1304. ¹H NMR: 5.78-5.91 (m, 1H); 5.58-5.70 (m, 1H); 4.75 (d, *J*=2.1, 1H); 4.20 (q, *J*=7.1, 2H); 2.81 (dd, 1H, *J*₁=14.9, *J*₂=3.7, 1H); 1.38-2.25 (m, 13H); 1.28 (t, *J*=7.1, 3H). ¹³C NMR: 178.8 (CH); 132.0 (CH); 128.0 (CH); 72.5 (C); 60.5 (CH₂); 55.2 (C); 39.5 (CH₂); 34.0 (CH₂); 33.4 (CH₂); 30.6 (CH₂); 24.0 (CH₂); 20.8 (2 x CH₂); 20.0 (CH₃).

Physical data for **3c trans**: Colorless oil. Anal. calcd. for C₁₄H₂₂O₃: C, 70.56; H, 9.30; found: C, 70.69; H 9.24. IR (film): 3559, 3022, 2934, 2860, 1724, 1449, 1366, 1312. ¹H NMR: 5.94-6.05 (m, 1H); 5.53-5.64 (m, 1H); 4.17 (q, *J* = 7.4, 2H); 3.29 (dt, *J*₁=15.8, *J*₂=2.9, 1H); 1.94-2.19 (m, 4H); 1.42-1.83 (m, 9H); 1.27 (t, *J*=7.0, 3H); 1.07-1.21 (m, 1H). ¹³C NMR: 175.2 (C); 134.6 (CH); 127.0 (CH); 70.5 (C); 59.9 (CH₂); 56.2 (C); 39.6 (CH₂); 36.5 (CH₂); 34.7 (CH₂); 32.5 (CH₂); 25.1 (CH₂); 22.7 (CH₂); 21.6 (CH₂); 14.2 (CH₃). HRMS (ESI) Calcd. for C₁₄H₂₂O₃Na: 261.1467; found: 261.1455.

Ethyl *cis*-12-hydroxy-bicyclo[10.4.0]hexadec-14-ene-1-carboxylate (**3d cis**)

According to the procedure for **3d trans** (given in the Experimental Section). Starting from 300 mg (0.893 mmol) of **2d cis** and 22 mg (26.8 μ mol) of **5a**, after purification of a crude product by flash-chromatography (eluent: petroleum-ether:ethyl acetate=975:25), 200 mg (72.7%) of the title compound **3d cis** was obtained.

Physical data for **3d cis**: Colorless oil. IR (film): 2924, 2864, 1701, 1195. ^1H NMR: 5.45-5.61 (m, 2H); 4.29 (s, 1H, OH); 4.16 (q, $J=7.1$, 2H); 2.55-2.71 (m, 1H); 2.27 (br. s, 2H); 1.08-2.10 (m, 24H). ^{13}C NMR: 178.0 (C); 125.8 (CH); 124.7 (CH); 74.7 (C); 60.6 (CH₂); 52.1 (C); 38.9 (CH₂); 34.9 (CH₂); 33.8 (CH₂); 30.0 (CH₂); 26.6 (CH₂); 25.7 (CH₂); 25.7 (CH₂); 23.0 (CH₂); 22.8 (CH₂); 22.8 (CH₂); 21.8 (CH₂); 20.9 (CH₂); 14.1 (CH₃). HRMS (ESI) Calcd. for C₁₉H₃₂O₃Na: 331.2249; found: 331.2241.

General procedure for the preparation of compounds 4a-c. *trans*-6-Hydroxymethylbicyclo[4.3.0]non-3-ene-1-ol (4a)

A solution of **3a** (280 mg; 1.33 mmol) in THF (10.2 mL) was added to a suspension of LiAlH₄ (300 mg; 8 mmol) in THF (10.2 mL), under an argon atmosphere, and the reaction mixture was heated to reflux for 2 h. Upon cooling, water (0.3 mL) was added, followed by 15% aqueous NaOH (0.3 mL) and water (0.9 mL). The mixture was filtered, the precipitate was washed with ethyl acetate, and the filtrate was extracted with ethyl acetate. The combined organic extract was dried over anh. MgSO₄ and concentrated under reduced pressure, to give 193 mg (87%) of the title compound **4a** as a white solid, which was recrystallized from dichloromethane/hexane.

Physical data for **4a**: White crystals, mp 98-103 °C. IR (KBr): 3408, 3371, 3288, 3271, 3031, 2948, 2889, 1641, 1500, 1032. ¹H NMR: 5.73-5.80 (m, 1H); 5.59-5.68 (m, 1H); 3.39 (d, *J*=10.1, 1H); 3.16 (d, *J*=10.1, 1H); 2.35-2.50 (m, 2H); 2.18-2.35 (m, 2H); 1.57-2.16 (m, 8H). ¹³C NMR: 127.1 (CH); 124.5 (CH); 79.3 (C); 63.8 (CH₂); 49.4 (C); 34.8 (CH₂); 34.7 (CH₂); 29.6 (CH₂); 29.5 (CH₂); 19.2 (CH₂). HRMS (ESI) Calcd. for C₁₀H₁₆O₂Na: 191.1048; found: 191.1049.

***cis*-6-Hydroxymethylbicyclo[4.4.0]dec-3-ene-1-ol (**4b cis**)**

According to the general procedure, starting from 181 mg (0.81 mmol) of **3b cis**, to give 147 mg (100%) of the title compound.⁷

Physical data for **4b cis**: Colorless oil. Anal. calcd. for C₁₁H₁₈O₂: C, 72.49; H, 9.95; found: C, 72.65; H 10.14. IR (film): 3304, 3026, 2939, 2863, 1449. ¹H NMR: 5.46-5.59 (m, 2H); 3.96 (d, *J*=10.8, 1H); 3.66 (bs, 2H); 3.37 (d, *J*=10.8, 1H); 1.90-2.50 (m, 4H); 1.34-1.81 (m, 7H), 1.05-1.13 (m 1H). ¹³C NMR: 124.1 (CH); 124.0 (CH); 74.1 (C); 69.7 (CH₂); 39.0 (C); 38.7 (CH₂); 34.5 (CH₂); 31.5 (CH₂); 28.6 (CH₂); 21.2 (CH₂); 20.6 (CH₂).

***trans*-6-Hydroxymethylbicyclo[4.4.0]dec-3-ene-1-ol (**4b trans**)**

According to the general procedure, starting from 166 mg (0.74 mmol) of **3b trans**. The crude product was purified by dry-flash chromatography (eluent: toluene:ethyl acetate=4:1) to give 134.5 mg (100%) of the title compound.⁷

Physical data for **4b trans**: White crystals, mp 83 °C. Anal. calcd. for C₁₁H₁₈O₂: C, 72.49; H, 9.95; found: C, 72.20; H 10.21. IR (KBr): 3380, 3364, 3024, 2935, 2863, 1652, 1451, 1411, 1297. ¹H NMR: 5.54-5.76 (m, 2H); 3.73 (dd, *J*₁=10.6, *J*₂=1.5, 1H); 3.47 (d, *J*=10.6, 1H); 2.16 (dd, *J*₁=18.0, *J*₂=4.8, 1H); 1.37-1.98 (m, 13H). ¹³C NMR: 126.7 (CH); 123.9 (CH); 70.2 (C); 61.8 (CH₂); 41.0 (C); 37.3 (CH₂); 33.5 (CH₂); 30.7 (CH₂); 27.7 (CH₂); 21.3 (CH₂); 21.0 (CH₂).

***cis*-7-Hydroxymethylbicyclo[5.4.0]undec-3-ene-1-ol (4c cis)**

According to the general procedure, starting from 180 mg (0.75 mmol) of **3c cis**. The crude product was purified by dry-flash chromatography (eluent: petroleum-ether:ethyl acetate=7:4) to give 146.4 mg (100%) of the title compound.⁷

Physical data for **4c cis**: White crystals, mp 58 °C. IR (film) 3351, 3327, 3025, 2930, 2863, 1656, 1449, 1310. ¹H NMR: 5.92 (ddt, *J*₁=10.5, *J*₂=6.1, *J*₃=1.8, 1H); 5.59 (ddt, *J*₁=10.5, *J*₂=6.1, *J*₃=1.8, 1H); 3.96 (d, *J*=11.2, 1H); 3.78 (d, *J*=11.2, 1H); 3.43 (bs, 1H); 3.16 (bs, 1H); 2.40-2.59 (m, 2H); 2.00-2.35 (m, 2H); 1.25-1.83 (m, 10H). ¹³C NMR: 134.1 (CH); 126.7 (CH); 77.7 (C); 69.0 (CH₂); 43.4 (C); 38.7 (CH₂); 35.9 (CH₂); 32.6 (2 x CH₂); 23.5 (CH₂); 22.2 (CH₂); 20.8 (CH₂). HRMS (ESI) Calcd. for C₁₂H₂₀O₂Na: 219.1361, found: 219.1359.

***trans*-7-Hydroxymethylbicyclo[5.4.0]undec-3-ene-1-ol (**4c trans**)**

According to the general procedure, starting from 200 mg (0.84 mmol) of **3c trans**. The crude product was purified by dry-flash chromatography (eluent: toluene:ethyl acetate=4:1), and then by low pressure liquid chromatography (eluent: petroleum-ether:ethyl acetate=4:1) to give 74.1 mg (45%) of the title compound.⁷

Physical data for **4c trans**: White solid, mp 87 °C. IR (KBr): 3427, 3068, 3016, 2932, 2861, 1485, 1450, 1369, 1277. ¹H NMR: 5.89-5.99 (d, 1H); 5.42-5.52 (m, 1H); 3.94 (d, *J*=11.0, 1H); 3.84 (d, *J*=11.0, 1H); 2.27-2.89 (m, 2H); 1.88-2.15 (m, 4 H); 1.41-1.58 (m, 10H). ¹³C NMR: 134.3 (CH); 125.6 (CH); 71.2 (C); 60.4 (CH₂); 45.4 (C); 39.6 (CH₂); 36.2 (CH₂); 29.9 (CH₂); 29.4 (CH₂); 24.2 (CH₂); 21.4 (CH₂); 21.0 (CH₂). HRMS (ESI) Calcd. for C₁₂H₂₀O₂Na: 219.1361, found: 219.1359.

(*Z*)-6-Methylenecyclodec-3-ene-1-one (7b**)**

According to the procedure for **7a** (given in the Experimental Section), starting from 120 mg (0.66 mmol) of **4b trans**. The fragmentation step was performed for 4 h at 60 °C. The crude product was purified by dry-flash chromatography (eluent: pentane:diethyl ether=9:1) to give 63 mg (58%) of the title compound **7b** (highly volatile).

Physical data for **7b**: Colorless oil. IR (film): 3072, 3021, 2939, 1705, 1644, 1462. ^1H NMR: 5.60-5.80 (m, 2H); 4.80 (s, 2H); 3.21 (d, $J=7.0$, 2H); 2.59 (d, $J=7.2$, 2H); 2.50-2.56 (m, 2H); 2.07 (t, $J=6.8$, 2H); 1.74-1.78 (m, 2H); 1.54-1.59 (m, 2H). ^{13}C NMR: 213.4 (C); 147.6 (C); 131.9 (CH); 123.9 (CH); 112.3 (CH₂); 42.4 (CH₂); 39.8 (CH₂); 34.2 (CH₂); 32.4 (CH₂); 25.4 (CH₂); 24.7 (CH₂). HRMS (ESI) Calcd. for C₁₁H₁₆ONa: 187.1099; found: 187.1099.

(Z)-7-Methylenecycloundec-3-ene-1-one (7c)

According to the procedure for **7a** (given in the Experimental Section), starting from 60 mg (0.306 mmol) of **4c trans**. The fragmentation step was performed for 4 h at 60 °C. The crude product was purified by dry-flash chromatography (eluent: pentane:diethyl ether=9:1) to give 41 mg (76%) of the title compound **7c** (highly volatile).

Physical data for **7c**: Light yellow liquid. IR (film): 3016, 2934, 2863, 1707, 1643, 1451. ^1H NMR: 5.48-5.64 (m, 2H), 4.86 (bs, 1H), 4.75 (d, $J=1.6$, 1H), 3.13 (d, $J=6.4$, 2H), 2.05-2.51 (m, 7H), 1.51-1.81 (m, 5H). ^{13}C NMR: 211.8 (C); 148.5 (C); 134.0 (CH); 122.2 (CH); 111.7 (CH₂); 41.9 (CH₂); 41.2 (CH₂); 36.7 (CH₂); 32.4 (CH₂); 26.5 (CH₂); 25.3 (CH₂); 23.4 (CH₂). HRMS (ESI) Calcd. for C₁₂H₁₈ONa: 201.1255; found: 201.1256.

Oxatricyclo[5.4.2.0^{1,7}]tridec-3-ene (**8c**)

According to the procedure for **8b** (given in the Experimental Section), starting from 146.4 mg (0.75 mmol) of **4c cis**. The crude product was purified by dry-flash chromatography (eluent: pentane:diethyl ether=9:1) to give 86.4 mg (65%) of the title compound **8c**.

Physical data for **8c**: Light yellow liquid. IR (film): 3015, 2922, 2863, 1462, 1269. ¹H NMR: 5.38-5.54 (m, 2H); 4.31 (bs, 2H); 3.25-3.35 (m, 2H); 1.25-2.00 (m, 8H); 2.65 (dd, *J*_I=10.4, *J*_I=6.2, 1H); 2.58 (dd, *J*=10.8, *J*_I=5.8, 1H); 2.22-2.28 (m, 2 H). ¹³C NMR: 130.6 (CH); 124.2 (CH); 90.9 (C); 76.4 (CH₂); 53.4 (C); 37.2 (CH₂); 33.4 (CH₂); 31.7 (CH₂); 29.4 (CH₂); 27.5 (CH₂); 19.6 (CH₂); 18.6 (CH₂). HRMS (ESI) Calcd. for C₁₂H₁₉O [M+H]⁺: 179.1436; found: 179.1422.

2-(Trimethylsilyloxy-2-butenyl)-cyclohexan-1-ones (**10 syn**) and (**10 anti**)

Crotonaldehyde (247 μ L; 3.0 mmol) and cyclohexanone trimethylsilyl enoether⁸ (1.72 mL; 9.0 mmol) were added to a solution of anhydrous lithium perchlorate⁹ (2.40 g; 22.6 mmol) in freshly distilled, dry nitromethane (3 mL). The reaction mixture was stirred at rt for 16 h, under an argon atmosphere (the reaction was monitored by TLC, eluent: 5% ethyl acetate in hexanes). Upon dilution of the reaction mixture with dichloromethane (100 mL), the organic solution was washed with ice-cold water (2 x

20 mL) and dried over anh. MgSO_4 . Concentration under reduced pressure afforded the crude product **10** as a 1:1 mixture of *syn* and *anti* isomers, as determined by GC analysis. Purification by dry-flash chromatography (eluent: 3% ethyl acetate in hexanes) afforded 255 mg (35%) of **10 syn** (eluted first), followed by 270 mg (38%) of **10 anti** and 30 mg (4%) of the mixture of isomers **10 syn** and **10 anti** that were not separated.

Physical data for **10 syn**: Colorless oil. IR (film): 3051, 2939, 1706, 1449, 1264, 1091, 910, 844, 734. ^1H NMR: 5.41-5.70 (m, 2H), 4.51 (t, $J=6.0$, 1H); 2.21-2.36 (m, 3H); 1.60-2.12 (m, 9H); 0.09 (s, 9H). ^{13}C NMR: 211.9 (C); 132.9 (CH); 125.6 (CH); 70.7 (CH); 57.1 (CH); 42.5 (CH_2); 28.3 (CH_2); 27.5 (CH_2); 24.2 (CH_2); 17.6 (CH_3); 0.3 (3 x CH_3).

Physical data for **10 anti**: Colorless oil. IR (film): 2942, 2863, 1707, 1659, 1450, 1260, 1058, 910, 845. ^1H NMR: 5.52-5.69 (m, 1H); 5.32-5.42 (m, 1H); 4.52 (t, $J=6.7$, 1H); 2.16-2.47 (m, 3H); 1.42-2.03 (m, 9H); 0.07 (s, 9H). ^{13}C NMR: 211.8 (C); 131.7 (CH); 127.0 (CH); 72.0 (CH); 57.3 (CH); 42.0 (CH_2); 28.9 (CH_2); 27.8 (CH_2); 24.0 (CH_2); 17.6 (CH_3); 0.3 (3 x CH_3).

Anal. Calcd. for $\text{C}_{13}\text{H}_{24}\text{O}_2\text{Si}$ (mixture of isomers): C, 64.95%; H, 10.06%. Found: C, 64.72%; H, 10.31%.

The relative stereochemistry of compound **10 syn** was determined by correlation with a known compound. Thus, treatment of **10 syn** with 5% HCl at rt, in a THF solution, for 5 min, effected the hydrolysis of trimethylsilyl group and afforded the known aldol whose structure was previously established.¹⁰

1-Allyl-2-(Trimethylsilyloxy-2-butenyl)-cyclohexan-1-ol (11 syn**)**

Zinc powder (180 mg; 2.75 mmol) was added to a solution of **10 syn** (220 mg; 0.92 mmol) and allyl bromide (237 μ L; 2.75 mmol) in DMF (1.2 mL) and the reaction mixture was stirred for 12 h under an argon atmosphere (the reaction can be monitored by TLC, eluent: 5% ethyl acetate in hexanes). The reaction mixture was poured into dichloromethane (100 mL), and the organic layer was washed with saturated aqueous NH_4Cl (3 x 15 mL), water (2 x 10 mL) and brine (10 mL), dried over anhydrous MgSO_4 and concentrated under reduced pressure. Purification by dry-flash chromatography (eluent: 5% ethyl acetate in hexanes) afforded 177 mg (68%) of the title compound **11 syn**, followed by 12 mg (4.6%) of the stereoisomer *epi*-**11 syn**.

Physical data for **11 syn**: Colorless oil. Anal. Calcd. for $\text{C}_{16}\text{H}_{30}\text{O}_2\text{Si}$: C, 68.03%; H, 10.70%. Found: C, 68.06%; H, 10.98%. IR (film): 3497, 2937, 2857, 1668, 1640, 1445, 1254, 1129, 1086, 1067, 987. ^1H NMR: 5.73-5.94 (m, 1H); 5.51-5.55 (m, 2H); 5.11 (d, $J=3.1$, 1H); 5.04 (br s, 1H); 4.72 (m, 1H); 4.27 (d, $J=2.2$, OH); 2.29-2.48 (m, 2H); 1.05-1.76 (m, 12H); 0.13 (s, 9H). ^{13}C NMR: 135.0 (CH); 132.8 (CH); 126.6 (CH); 117.2 (CH_2); 74.8 (CH); 73.3 (C); 47.6 (CH); 46.8 (CH_2); 36.7 (CH_2); 25.9 (CH_2); 21.2 (CH_2); 19.7 (CH_2); 17.5 (CH_3); 0.6 (3 x CH_3).

Physical data for *epi*-**11 syn**: Colorless oil. IR (film): 3438, 2932, 2860, 1669, 1639, 1451, 1252, 1153, 1092, 1035, 842. ^1H NMR: 5.83-6.03 (m, 1H); 5.49-5.67 (m, 2H); 5.02-5.12 (m, 2H); 4.33 (t, $J=5.5$, 1H); 3.20 (br s, 1H); 2.29-2.51 (m, 2H); 1.07-1.86 (m, 12H); 0.09 (s, 9H). ^{13}C NMR: 134.7 (CH); 133.8 (CH); 126.6 (CH); 117.3 (CH_2);

75.7 (CH); 74.6 (C); 52.8 (CH); 38.6 (CH₂); 37.7 (CH₂); 25.8 (CH₂); 25.4 (CH₂); 22.8 (CH₂); 17.6 (CH₃); 0.4 (3 x CH₃).

1-Allyl-2-(Trimethylsilyloxy-2-butenyl)-cyclohexan-1-ol (11 anti**)**

According to the procedure for **11 syn**. Starting from 237 mg (0.99 mmol) of **10 anti**, after purification by flash-chromatography (eluent: 3.8% ethyl acetate in hexanes), 168 mg (60%) of the title compound **11 anti** was obtained, followed by 25 mg (12.6%) of the desilylated derivative of **11 anti**.

Physical data for **11 anti**: Colorless oil. Anal. Calcd. for C₁₆H₃₀O₂Si: C, 68.03%; H, 10.70%. Found: C, 68.22%; H, 10.86%. IR (film): 3496, 2935, 2859, 1672, 1639, 1446, 1253, 1137, 1050, 976, 911, 876. ¹H NMR: 5.78-5.99 (m, 1H); 5.46-5.60 (m, 2H); 5.07 (br s, 1H); 4.98-5.04 (m, 1H); 4.35-4.41 (m, 1H); 3.96 (br s, OH); 2.40 (d, *J*=7.3, 2H); 1.70 (d, *J*=4.8, 3H); 1.27-1.62 (m, 9H); 0.11 (s, 9H). ¹³C NMR: 134.8 (CH); 134.3 (CH); 126.7 (CH); 117.1 (CH₂); 77.2 (CH); 73.8 (C); 47.6 (CH); 46.2 (CH₂); 36.4 (CH₂); 25.3 (CH₂); 24.0 (CH₂); 21.8 (CH₂); 17.5 (CH₃); 0.4 (3 x CH₃).

trans-Bicyclo[4.4.0]dec-3-ene-*cis*-1,5-diol (12 syn**)**

A deaerated solution of **11 syn** (150 mg; 0.53 mmol) and **5a** (15.3 mg; 18 μmol) in dichloromethane (2.8 mL) was stirred at rt for 24 h, under an argon atmosphere. The

solvent was removed under reduced pressure and the dark residue was purified by dry-flash chromatography (eluent: 5% ethyl acetate in hexanes), to afford 100 mg (78%) of the silylated derivative of **12-OTMS syn**, followed by 16.5 mg (12%) of the starting compound **11 syn** (the yield calculated on the basis of the converted starting compound is 89%).

Physical data for **12-OTMS syn**: Colorless, viscous oil. IR (film): 3478, 3030, 2933, 1656, 1445, 1388, 1254, 1127, 1068, 968, 906, 881. ¹H NMR: 5.59 (s, 2H); 3.94-4.01 (m, 1H); 1.94-2.17 (m, 2H); 1.15-1.92 (m, 10H); 0.10 (s, 9H). ¹³C NMR: 131.2 (CH); 124.5 (CH); 70.8 (C + CH); 47.2 (CH); 40.4 (CH₂); 39.3 (CH₂); 25.5 (CH₂); 24.9 (CH₂); 21.3 (CH₂); 0.1 (3 x CH₃).

To a solution of **12-OTMS syn** (92 mg; 0.38 mmol) in THF (4 mL) was added 5% aqueous HCl (130 µL; 0.173 mmol) and the reaction mixture was vigorously stirred at rt for 3-5 min. The mixture was diluted with dichloromethane (40 mL), washed successively with aqueous NaHCO₃ (5 mL), water (5 mL) and brine, and dried over anh. MgSO₄. Evaporation of solvent under reduced pressure, followed by purification of the residue by dry-flash chromatography (eluent: 30% ethyl acetate in hexanes) afforded 52 mg (80%) of the title compound **12 syn**.

Physical data for **12 syn**: Colorless film. IR (film): 3462-3365, 3027, 2927, 2855, 1655, 1446, 1385, 1225, 1119, 1033, 990, 928. ¹H NMR: 5.76 (d, *J*=10.3, 1H); 5.61-5.73 (m, 1H); 3.95-3.98 (m, 1H); 1.99-2.15 (m, 3H); 1.21-1.84 (m, 10H). ¹³C NMR: 130.7 (CH); 125.2 (CH); 70.7 (C); 70.4 (CH); 47.8 (CH); 40.3 (CH₂); 39.1 (CH₂); 25.4 (CH₂); 24.7 (CH₂); 21.2 (CH₂). HRMS (ESI) Calcd. for C₁₀H₁₆O₂Na: 191.1048; found: 191.1047.

***trans*-Bicyclo[4.4.0]dec-3-ene-*trans*-1,5-diol (**12 anti**)**

According to the procedure for **12-OTMS syn** and **12 syn**. Starting from 150 mg (0.53 mmol) of **11 anti**, after purification by dry-flash chromatography (eluent: 5% ethyl acetate in hexanes), 82 mg (64%) of the silylated derivative of **12-OTMS anti** was obtained.

Physical data for **12-OTMS anti**: Colorless needles, mp 69-71 °C. IR (KBr): 3474, 2929, 2861, 1655, 1424, 1323, 1253, 1151, 1029, 981, 920, 842. ^1H NMR: 5.71-5.87 (m, 2H); 4.26 (d, $J=2.0$, OH); 3.97-4.02 (m, 1H); 1.95-2.30 (m, 2H); 1.05-1.86 (m, 9H); 0.11 (s, 9H). ^{13}C NMR: 128.2 (CH); 127.9 (CH); 69.4 (C); 68.6 (CH); 43.4 (CH); 41.6 (CH₂); 38.9 (CH₂); 26.2 (CH₂); 24.6 (CH₂); 21.5 (CH₂); 0.1 (3 x CH₃).

Deprotection of **12-OTMS anti** (72 mg; 0.3 mmol) according to the procedure for the preparation of **12 syn**, followed by purification by dry-flash chromatography (eluent: 20% ethyl acetate in hexanes) afforded 43 mg (85%) of the title compound **12 anti**.

Physical data for **12 anti**: Colorless film. IR (film): 3367-3334, 2924, 2859, 1655, 1445, 1419, 1327, 1243, 1147, 1097, 988, 963. ^1H NMR: 5.93-6.03 (m, 1H); 5.75 (ddd, $J_1=2.8$, $J_2=4.7$, $J_3=10.0$, 1H); 3.92 (br s, 1H); 2.88 (br s, OH); 2.58 (br s, OH); 2.11-2.17 (m, 2H); 1.23-1.95 (m, 9H). ^{13}C NMR: 129.1 (CH); 126.7 (CH); 70.6 (C); 67.6 (CH); 43.2 (CH); 41.3 (CH₂); 39.5 (CH₂); 26.1 (CH₂); 24.0 (CH₂); 21.5 (CH₂). HRMS (ESI) Calcd. for C₁₀H₁₆O₂Na: 191.1048; found: 191.1047.

***trans*-1,5-Epoxy-bicyclo[4.4.0]dec-3-ene (**14**)**

A solution of **12 anti** (22 mg; 0.131 mmol), mesyl chloride (14.4 μ L; 0.191 mmol) and DMAP (3 mg; 25 μ mol) in pyridine (200 μ L) was stirred at rt under an argon atmosphere for 24 h. The reaction mixture was diluted with diethyl ether (15 mL) and concentrated aqueous NaHCO₃ (1 mL) and vigorously stirred for 10 min. The organic layer was washed successively with 1M aqueous HCl, saturated aqueous NaHCO₃ and water, dried over anhydrous MgSO₄ and concentrated under reduced pressure. Purification of the residue by column chromatography (eluent: 20% ethyl acetate in hexanes) afforded the title compound **14** (6 mg; 28%), followed by the recovered starting compound **12 anti** (7 mg; 34%; 42% calculated on the basis of recovered **12 anti**). The same compound was obtained when the procedure was performed with **12 syn**.

Physical data for **14**: Colorless film. IR (film): 3023, 2935, 2859, 1653, 1446, 1385, 1254, 1206, 1134, 1068, 983, 932. ¹H NMR: 5.80 (dt, $J_1=1.8$, $J_2=10.2$, 1H); 5.65-5.75 (m, 1H); 4.35 (ddt, $J_1=1.8$, $J_2=5.4$, $J_3=9.6$, 1H); 1.26-2.31 (m, 11H). ¹³C NMR: 129.4 (CH); 126.0 (CH); 70.8 (C); 61.5 (CH); 48.8 (CH); 40.1 (CH₂); 39.2 (CH₂); 25.6 (CH₂); 25.5 (CH₂); 21.3 (CH₂). HRMS (ESI) Calcd. for C₁₀H₁₄ONa: 173.0942; found: 173.0940.

1-Hydroxy-*trans*-bicyclo[4.4.0]dec-3-en-5-one (15)

PDC (244 mg; 0.65 mmol) was added in one portion to a solution of **12** *syn* (84 mg; 0.5 mmol) in dry dichloromethane (6.5 mL) and the reaction mixture was stirred for 45 min at rt. Dichloromethane was removed under reduced pressure, ethyl acetate (30 mL) was added and the resulting suspension was filtered through a short pad of celite (the celite pad was washed with two 15 mL portions of ethyl acetate). The combined ethyl acetate solution was concentrated under reduced pressure and the residue was purified by dry-flash chromatography to give 71 mg (86%) of the title compound **15**.

The same compound **15** was also obtained from **12** *anti*, using the identical experimental procedure, in 78% yield.

Physical data for **15**: Colorless crystals, mp 87-88 °C. Anal. Calcd. for C₁₀H₁₄O₂: C, 72.26%; H, 8.49%. Found: C, 72.01%; H, 8.80%. IR (KBr): 3368, 2931, 2855, 1666, 1447, 1419, 1395, 1305, 1269, 1232, 1141, 991. ¹H NMR: 6.81 (ddd, *J*₁=2.9, *J*₂=4.8, *J*₃=10.2, 1H); 6.09 (ddd, *J*₁=1.1, *J*₂=2.6, *J*₃=10.2, 1H); 2.42-2.54 (m, 2H); 2.36 (dd, *J*₁=3.7, *J*₂=4.2, 1H); 1.18-2.14 (m, 9H). ¹³C NMR: 199.5 (C); 144.6 (CH); 129.6 (CH); 73.4 (C); 53.6 (CH); 41.0 (CH₂); 38.9 (CH₂); 24.9 (CH₂); 20.7 (CH₂); 20.4 (CH₂).

The stereochemical configuration of **15** was established in a following way: a solution of **15** in methanol was hydrogenated under 1 at of hydrogen, in the presence of 10% Pd/C, at rt, for 45 min, to give a saturated hydroxyketone in a quantitative yield. This compound was recrystallized from ethyl acetate/hexane to give the crystalline material mp 104 °C (lit.¹¹ mp 104 °C) identical to the previously described compound,¹¹ thus proving the *trans*-decaline configuration of the bicyclic system in **15**.

Methyl 4-isopropyl-4-methoxybenzoate (**22**)¹²

To a refluxing solution of 3-isopropylanisole **21** (14.8 g; 98 mmol) in cyclohexane (100 mL) a solution of *n*-butyllithium in hexane (66.2 mL of 1.48 M solution; 98 mmol) was added dropwise. The reaction mixture was refluxed for 15 h, then allowed to cool to r. t. and transferred via cannula to a vigorously stirred suspension of dry ice (~100 g) in diethyl ether (350 mL). The resulting mixture was extracted with 3% aqueous NaOH (5 x 25 mL), the water phase was separated, acidified with HCl conc. (with cooling) and extracted with dichloromethane (4 x 20 mL). The organic extract was washed with water (2 x 10 mL), dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The crude residue (13.8 g) was dissolved in a mixture of toluene (40 mL) and methanol (30 mL), *p*-toluenesulfonic acid (1.5 g) was added and the mixture was refluxed in a Dean-Stark apparatus for 8 h with azeotropic removal of water. The mixture was cooled to r. t., concentrated under reduced pressure, dissolved in dichloromethane (100 mL), washed with water (5 x 10 mL),

saturated aqueous NaHCO_3 (3 x 10 mL), water (10 mL), dried over anhydrous Na_2SO_4 , filtered and concentrated under reduced pressure. Distillation under reduced pressure afforded the title compound **22** (9.15 g; 45%).

Physical data for **22**: Colorless liquid; E_2 147 °C. Anal. calcd. for $\text{C}_{12}\text{H}_{16}\text{O}_3$: C 69.21, H 7.74; found C 69.05, H 7.82. IR (film): 3055, 2963, 1728, 1609, 1461, 1415, 1294, 1251, 1188, 1148, 1090. ^1H NMR: 7.75 (d, $J=8.2$, 1H); 6.87-6.82 (m, 2H); 3.91 (s, 3H); 3.87 (s, 3H); 3.02-2.82 (hept, $J=6.9$, 1H); 1.26 (d, $J=7.0$, 6H). ^{13}C NMR: 166.5; 159.4; 155.4; 131.8; 118.1; 117.2; 110.2; 55.8; 51.7; 34.4; 23.6.

***trans*-1-Hydroxy-*cis*-6-hydroxymethyl-*r*-9-isopropyl-bicyclo[4.4.0]deca-3,7-diene
(26)**

To a suspension of lithium aluminum hydride (112 mg; 2.96 mmol) in THF (5.5 mL) was added dropwise a solution of **25** (148 mg; 0.592 mmol) and the reaction mixture was stirred for 15 h at room temperature. The mixture was cooled (4 °C) and ethyl acetate (1 mL) was slowly added, followed by saturated aqueous solution of K₂Na-tartrate (12 mL). The organic layer was separated, the aqueous layer was extracted with diethyl ether (4 x 25 mL), and the combined organic extract was washed with water, brine, dried over anhydrous MgSO_4 , filtered and concentrated under reduced pressure. Purification by dry-flash chromatography (eluent: petroleum-ether/acetone=85/15) afforded the title compound **26** (125.8 mg; 96%).

Physical data for **26**: White crystals, m.p. 70-76 °C. Anal. calcd. for C₁₄H₂₂O₂ (222.32): C 75.63, H 9.97; found: C 75.17, H 9.49. IR (film): 3401, 3024, 2954, 2920, 1647, 1465, 1438, 1372, 1073, 1002. ¹H NMR: 5.85 (dd, *J*₁=10.2, *J*₂=2.2, 1H); 5.76-5.52 (m, 2H); 5.41 (dd, *J*₁=10.2, *J*₂=2.6, 1H); 3.46 (s, 2H); 2.41-1.57 (m, 10H); 0.94 (d, *J*=3.6, 3H); 0.91 (d, *J*=3.6, 3H). ¹³C NMR: 132.2 (CH); 130.2 (CH); 126.1 (CH); 124.4 (CH); 70.6 (C); 67.5 (CH₂); 43.1 (C); 38.9 (CH); 36.3 (CH₂); 33.4 (CH₂); 31.4 (CH); 29.5 (CH₂); 19.5 (CH₃); 19.1 (CH₃).

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- ¹ For the description of the technique of dry-flash chromatography, see: a) Harwood, L. M. *Aldrichimica Acta* **1985**, 18, 25; b) *Vogel's Textbook of Practical Organic Chemistry*, Longman Scientific&Technical, 5th edition, London, 1989, p. 220; c) A recent account which includes some improvements of the separation technique: Pedersen, D. S.; Rosenbohm, C. *Synthesis* **2001**, 2431.
- ² Perrin, D. D.; Armarego, W. L. F. *Purification of Laboratory Chemicals*, 3rd edition, Pergamon Press, **1988**.
- ³ Aburel, P. S.; Romming, C.; Ma, K.; Undheim, K. *J. Chem. Soc. Perkin Trans. 1*, **2001**, 1458.
- ⁴ Belotti, D.; Cossy, J.; Pete, P.; Portella, C. *J. Org. Chem.* **1986**, 51, 4196.
- ⁵ Tsuji, J.; Yamada, T.; Shimizu, I. *J. Org. Chem.* **1980**, 45, 5209.
- ⁶ Molander, G. A.; Harris, C. R. *J. Am. Chem. Soc.* **1995**, 117, 3705.
- ⁷ The *cis/trans* stereochemistry of compounds **2**, **3** and **4** was assigned retrogradely, on the basis of the experiment displayed in Scheme 4 (see text), which allowed to assign *trans*-configuration to all synthetic predecessors of **7**, and *cis*-configuration to the predecessors of **8**.
- ⁸ Freshly prepared according to the procedure: Ito, Y.; Fujii, S.; Nakatsuka, M.; Kawamoto, F.; Saegusa, T. *Org. Synth. Coll. Vol. VI*, p. 327.
- ⁹ Lithium perchlorate was heated 3 h at 170-180 °C before the reaction.
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- ¹¹ Wharton, P. S.; Hiegel, G. A. *J. Org. Chem.* **1965**, 30, 3254.
- ¹² Shirley, D. A.; Harmon, T. E.; Cheng, C. F. *J. Organometal. Chem.* **1974**, 69, 323.

Scanned copies of ^1H and ^{13}C NMR spectra for all
compounds

Solvent: cdcl3
Ambient temperature
GENIINI-200 "nmf"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse arrayed
Acq. time 2.145 sec
Width 3401.7 Hz
Arrayed repetitions
OBSERVE H1 199.9710948 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Ft size 16384
Total time 1 minutes

2c

HM016 DF
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax, delay arrayed
1st pulse arrayed
2nd pulse arrayed
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827785 MHz
DECOUPLE H1, 199.8712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 59 minutes

2c

NO-III-1
 Solvent: cdcl₃
 Ambient temperature
 GENIINI-200 MHz
 PULSE SEQUENCE
 Relax. delay arrayed
 1st pulse arrayed
 2nd pulse 80.0 degrees
 Acq. time 3.541 sec
 Width 4608.0 Hz
 Arrayed repetitions
 OBSERVE H1, 139.9710953 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 32768
 Total time 38 minutes

2d cis

-1vent: cdc13
 Ambient temperature
 GENIINI-200 "nmr"
 PULSE SEQUENCE
 Relax. delay arrayed
 1st pulse arrayed
 2nd pulse 73.6 degrees
 Acq. time 1.067 sec
 Width 15000.0 Hz
 Arrayed repetitions
 OBSERVE C13, 50.2827782 MHz
 DECOUPLE H1, 199.9712807 MHz
 Power 0 dB
 continuously on
 VALT2-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 32768
 Total time 21 minutes

2d cis

Solvent: cdcl3
Ambient temperature
GENIINI-200 ¹HMR
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse arrayed
Acq. time 30.0 degrees
Width 3005.5 Hz
Arrayed repetitions
OBSERVE H1, 199.971058 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Ft size 32768
Total time 2 minutes

2d trans

Solvent: cdCl_3
 Ambient temperature
 GEMINI-200 "nmr"
 PULSE SEQUENCE
 Relax. delay arrayed
 1st pulse arrayed
 2nd pulse arrayed
 2nd pulse 73.6 degrees
 Acq. time 1.067 sec
 Width 15000.0 Hz
 Arrayed repetitions
 OBSERVE: Cl_3 , 50.282782 MHz
 DECOUPLE: H_1 , 199.9712607 MHz
 Power 0 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 32768
 Total time 24 minutes

3a

NH 007b
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse arrayed
Acq. time 122.7 degrees
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.282785 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
ON during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 12.4 hours

3a

MM015F1
Solvent: cdc13
Ambient temperature
GENIEMI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 2.149 sec
Width 3797.9 Hz
Arrayed repetitions
OBSERVE HI, 199.9710911 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 1 minutes

MM 015F1
 Solvent: cdcl3
 Ambient temperature
 GEMINI-200 "nmr"
 PULSE SEQUENCE: apt
 Relax. delay arrayed
 1st pulse arrayed
 2nd pulse 122.7 degrees
 Acq. time 2.00 sec
 Width 15000.0 Hz
 Arrayed repetitions
 OBSERVE G13, 50.282794 MHz
 DECOUPLE H1, 199.9712807 MHz
 Power 0 dB
 on during acquisition
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 2.0 Hz
 FT size 65536
 Total time 42 minutes

3b c/s

MM01SF2
Solvent: cdcl3
Ambient temperature
GENIINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 2.149 sec
Width 2986.1 Hz
Arrayed repetitions
OBSERVE H1, 199.9710945 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Ft size 16384
Total time 1 minutes

3b *trans*

Solvent: cdc13
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed Repetitions
OBSERVE C13, 50.282785 MHz
DECUPLE H1, 199.9712607 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 47 minutes

3b trans

NH023 F2
Solvent: cdc13
Ambient temperature
CEMNI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 2.074 sec
Width 3401.7 Hz
Arrayed repetitions
OBSERVE H1 159.8710870 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 1 minutes

3c cis

MH023 F2
 Solvent: cdcl3
 Ambient temperature
 GEMINI-200 "nmr"
 PULSE SEQUENCE: apt
 Relax. delay arrayed
 1st pulse arrayed
 2nd pulse 122.7 degrees
 Acq. time 2.000 sec
 Width 15000.0 Hz
 Arrayed repetitions
 OBSERVE C13, 50.2827817 MHz
 DECOUPLE H1, 199.3712807 MHz
 Power 0 dB
 on during acquisition
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 2.0 Hz
 FT size 65536
 Total time 16 minutes

3c c/s

HM029F1
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax: delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 2.073 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710820 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 1 minutes

3c trans

HM023F1
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13' 50.282794 MHz
DECUPLE H1' 100.621207 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 27 minutes

3c trans

Solvent: cdcl3
Ambient temperature
CEMNI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse arrayed
Acq. time 3.513 sec
Width 3015.1 Hz
Arrayed repetitions
OBSERVE H1, 199.971059 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Fi size 32768
Total time 2 minutes

3d cis

Solvent: cdc13
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 73.6 degrees
Acq. time 1.067 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.282773 MHz
DECUPLE H1, 199.9712807 MHz
Power 0 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Fit size 32768
Total time 52 minutes

3d cis

Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 3.543 sec
Width 2398.5 Hz
Arrayed repetitions
ORSERVE H1 188.9710952 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 2 minutes

Solvent: cdcl₃
Ambient temperature
GENI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 79.6 degrees
Acq. time 1.067 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.282782 MHz
DECOUPLE H1, 199.9712607 MHz
Power 0 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
Ft size 32768
Total time 65 minutes

NH022K
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 2.118 sec
Width 3392.0 Hz
Arrayed repetitions
OBSERVE H1 199.9710937 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 1 minutes

HM022K
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.282794 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 db
On during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 53 minutes

MM019F2
Solvent: cdcl3
Ambient temperature
GEMINI-200 ¹H NMR
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 54.0 degrees
Acq. time 2.145 sec
Width 3005.5 Hz
Arrayed repetitions
OBSERVE H1, 198.9710921 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Ft size 16384
Total time 1 minutes

4b *cis*

MM019F2
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827803 MHz
DECUPLE H1, 159.9712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FI size 65536
Total time 11 minutes

Solvent: cdc13
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 2.071 sec
Width 3005.5 Hz
Arrayed repetitions
OBSERVE H1 199.9710923 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 1 minutes

MM 046
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 12.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13' 50.282780 MHz
DECOUPLE H1, 199.9712607 MHz
Power 0 db
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FI size 65536
Total time 40 minutes

Solvent: cdcl3
Ambient temperature
GENMI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 80.0 degrees
Acq. time 2.078 sec
Width 3411.3 Hz
Arrayed repetitions
OBSERVE H1, 189.5710934 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 1 minutes

Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827803 MHz
DECOUPLE H1, 199.3712807 MHz
Power 0 db
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FI size 65536
Total time 31 minutes

Solvent: cdc13
 Ambient temperature
 GEMINI-200 100 MHz
 PULSE SEQUENCE
 Relax. delay arrayed
 1st pulse arrayed
 2nd pulse arrayed
 Acq. time 2.071 sec
 Width 3005.5 Hz
 Arrayed repetitions
 OBSERVE H1 199.8710965 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 16384
 Total time 1 minutes

7a

7a

MM045

Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"

PULSE SEQUENCE

Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 2.073 sec
Width 2801.4 Hz

Arrayed repetitions
OBSERVE H1: 159.9710923 MHz

DATA PROCESSING
Line broadening 0.2 Hz

FT size 16384
Total time 1 minute

7b

HM045

Solvent: cdc13
Ambient temperature
GEMINI-200 "nmr"

PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.00 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.282798 MHz
DECUPLE H1, 199.9712607 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FI size 65536
Total time 23 minutes

7b

Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse arrayed
Acq. time 2.070 sec
Width 3401.7 Hz
Arrayed repetitions
OBSERVE H1 189.9710953 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Ft size 16384
Total time 1 minutes

7c

Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt.
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.282785 MHz
DECOUPLE H1, 199.3712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 74 minutes

7C

MM 040
 Solvent: cdcl3
 Ambient temperature
 GEMINI-200 "nmr"
 PULSE SEQUENCE
 Relax. delay arrayed
 1st pulse arrayed
 2nd pulse 90.0 degrees
 Acq. time 2.070 sec
 Width 3015.1 Hz
 Arrayed repetitions
 OBSERVE H1: 139.9710948 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 16384
 Total time 1 minutes

8b

NM 040
Solvent: cdc13
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.00 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.282794 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Ft size 65536
Total time 44 minutes

8b

Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 2.070 sec
Width 3401.7 Hz
Arrayed repetitions
OBSERVE H1, 199.9710959 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 3 minutes

8c

MN053 a
Solvent: cdcl3
Ambient temperature
File: aptmm053a
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.282789 MHz
DECUPLE H1, 199.9712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 14.2 hours

8c

9

9

10 anti

Solvent: cdcl3
Ambient temperature
GENI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.00 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.262778 MHz
DECOUPLE H1, 100.628007 MHz
Power 0 db
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 41 minutes

10 anti

10 *syn*

Solvent: cdcl₃
 Ambient temperature
 GEMINI-200 "nmr"
 PULSE SEQUENCE: apt
 Relax. delay arrayed
 1st pulse arrayed
 2nd pulse 122.7 degrees
 Acq. time 2.000 sec
 Width 15000.0 Hz
 Arrayed repetitions
 OBSERVE C13, 50.2827780 MHz
 DECOUPLE H1, 199.9712807 MHz
 Power 0 db
 on during acquisition
 WALTZ-16 modulated
 DATA PROCESSING
 line broadening 1.0 Hz
 FT size 65536
 Total time 12.1 hours

10 syn

Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 3.543 sec
Width 2998.5 Hz
Arrayed repetitions
OBSERVE H1, 199.3710971 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 2 minutes

11 anti

Solvent: cdcl₃
 Ambient temperature
 GENIINI-200 "nmr"
 PULSE SEQUENCE: apt
 Relax. delay arrayed
 1st pulse arrayed
 2nd pulse 122.7 degrees
 Acq. time 2.000 sec
 Width 15000.0 Hz
 Arrayed repetitions
 OBSERVE C13, 50.282789 MHz
 DECOUPLE H1, 199.9712807 MHz
 Power 0 dB
 on during acquisition
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FI size 65536
 Total time 27 minutes

Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse arrayed
Acq. time 3.419 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710971 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 4 minutes

11 syn

Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse arrayed
Acq. time 122.7 degrees
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.282780 MHz
DECOUPLE H1, 199.8712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 11.0 hours

epi-11 *syn*

MET4-AB
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 12.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13' 50.282779 MHz
DECOUPLE H1' 199.9712607 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 96 minutes

epi-11 syn

MET AC
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nm"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 18.0 degrees
Acq. time 3.569 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1 199.9710968 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 2 minutes

12-OTMS syn

Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827821 MHz
DECUPLE H1, 199.9712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FI size 65536
Total time 9 minutes

12-OTMS syn

solvent: cdcl3
Ambient temperature
GENIHI-200 MHz
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse arrayed
Acq. time 3.551 sec
Width 4800.0 Hz
Arrayed repetitions
OBSERVE H1, 199.8710868 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Ft size 32768
Total time 2 minutes

12-OTMS anti

METAT B
 Solvent: cdc13
 Ambient temperature
 ORNINI-200 $^{\circ}\text{C}$
 PULSE SEQUENCE: ept
 Relax. delay arrayed
 1st pulse arrayed
 2nd pulse 122.7 degrees
 Acq. time 2.000 sec
 Width 15000.0 Hz
 Arrayed repetitions
 OBSERVE C13, 50.282780 MHz
 DECOUPLE H1, 100.62607 MHz
 Power 0 dB
 on during acquisition
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FI size 8536
 Total time 61 minutes

12-OTMS anti

ZTDIOL A
 Solvent: cdc13
 Ambient temperature
 GEMINI-200 "nmr"
 PULSE SEQUENCE
 Relax. delay arrayed
 1st pulse arrayed
 2nd pulse 90.0 degrees
 Acq. time 2.070 sec
 Width 4600.0 Hz
 Arrayed repetitions
 OBSERVE H1 159.3710962 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 32768
 Total time 6 minutes

583

ZT DIOL B

Solvent: cdcl3
 Ambient temperature
 GEMINI-200 "nmr"

PULSE SEQUENCE

Relax. delay arrayed
 1st pulse arrayed
 2nd pulse 90.0 degrees
 Acq. time 2.081 sec
 Width 3797.9 Hz

Arrived repetitions

OBSERVE H1, 199.3710834 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 16384

Total time 6 minutes

12 anti

Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.282798 MHz
DECUPLE N1, 159.9712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Ft size 65536
Total time 46 minutes

0X-3
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse arrayed
Acq. time 2.072 sec
Width 2995.8 Hz
Arrayed repetitions
OBSERVE H1, 199.8710967 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Ft size 16384
Total time 1 minutes

14

Solvent: cdc13
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.282771 MHz
DECUPLE H1, 199.9712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Ft size 65536
Total time 13.1 hours

Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr-"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.282785 MHz
DECUPLE H1, 199.5712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
Ft size 65536
Total time 12.5 hours

Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.378 sec
Width 3401.7 Hz
Arrayed repetitions
OBSERVE H1, 159.9710948 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
Total time 20 minutes

16

Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 73.6 degrees
Acq. time 1.067 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827800 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
Ft size 32768
Total time 15.1 hours

Ambient temperature
GEMINI-200 "hftmr"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 54.0 degrees
Acq. time 3.530 sec
Width 2987.4 Hz
32 repetitions
OBSERVE H1, 199.9750702 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 2 minutes

Solvent: cdc13
Ambient temperature
GEMINI-200 "hnmr"
PULSE SEQUENCE: apt
Relax. delay 4.000 sec
1st pulse 245.5 degrees
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
183 repetitions
OBSERVE C13, 50.2837664 MHz
DECUPLE H1, 199.9754738 MHz
Power 0 db
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 18 minutes

AI 22

Solvent: cdcl3
Ambient temperature
GEMINI-200 "hfrmr"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 90.0 degrees

Acq. time 3.477 sec

Width 2981.6 Hz

32 repetitions

OBSERVE H1, 199.9750703 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 32768

Total time 2 minutes

AI 22

Solvent: cdcl3
Ambient temperature
GEMINI-200 "Hnmr"
PULSE SEQUENCE: apt
Relax. delay 4.000 sec
1st pulse 245.5 degrees
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
736 repetitions
OBSERVE C13, 50.2837637 MHz
DECOUPLE H1, 199.9754738 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Ft size 65536
Total time 73 minutes

24

AI 24
Solvent: cdc13
Ambient temperature
GEMINI-200 "hfrmr"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 18.0 degrees
Acq. time 3.465 sec
Width 2367.4 Hz
32 repetitions
OBSERVE H1, 199.9750616 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 2 minutes

A124

Solvent: cdcl3
Ambient temperature
GEMINI-200 "hfrmr."

PULSE SEQUENCE: apt

Relax. delay 4.000 sec

1st pulse 245.5 degrees

2nd pulse 122.7 degrees

Acq. time 2.000 sec

Width 15000.0 Hz

520 repetitions

OBSERVE C13, 50.283715 MHz

DECUPLE C13, 50.283715 MHz

Power 0.000000

on during acquisition

WALTZ16 mode used

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 52 minutes

28

29

29

17 Periplanone C

17 Periplanone C