Highly Diastereoselective Epoxidation of Allylically Substituted Cycloalkenes Catalyzed by Metalloporphyrins

Wing-Kei Chan, Peng Liu, Wing-Yiu Yu, Man-Kin Wong, and Chi-Ming Che**

Department of Chemistry and Open Laboratory of Chemical Biology of the Institute of Molecular Technology for Drug Discovery and Synthesis, The University of Hong Kong, Pokfulam Road, Hong Kong, China

cmche@hku.hk

SUPPORTING INFORMATION

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General Structures of Metalloporphyrin Catalysts

- 1:** $M = \text{Mn}^{3+}$; $X=\text{Cl}$; $Y=Z=\text{H}$ $\text{Mn}(\text{TDCPP})\text{Cl}$
2: $M = \text{Ru}^{2+}$; $X=\text{Cl}$; $Y=Z=\text{H}$ $\text{Ru}(\text{TDCPP})(\text{CO})$
 $M = \text{Mn}^{3+}$; $X=\text{CH}_3$; $Y=Z=\text{H}$ $\text{Mn}(\text{TMP})\text{Cl}$
 $M = \text{Mn}^{3+}$; $X=Y=Z=\text{F}$ $\text{Mn}(\text{TPFPP})\text{Cl}$
 $M = \text{Mn}^{3+}$; $X=Y=\text{H}$; $Z=\text{CH}_3$ $\text{Mn}(\text{TTP})\text{Cl}$

Control Experiments for Diastereoselective Epoxidation of **3d**, **3f** and **3h**

In table 1, it is noticed that high *trans*-selectivities yet moderate yields were obtained in the **1**-catalyzed epoxidation of **3d**, **3f**, and **3h**. To exclude the possibility that the high *trans*-selectivities might come from preferential decomposition of one of the diastereomeric epoxides during the reaction conditions, several control experiments have been performed. Epoxides **4d**, **4f**, and **4h** with known *trans*-selectivities (i.e., *trans*- : *cis*-epoxide ratio) were individually subjected to the **1**-catalyzed epoxidation reaction conditions. After the reactions, *trans*-selectivities of the recovered epoxides were determined by ^1H NMR. The results are summarized as follows:

entry	Epoxide (<i>trans</i> : <i>cis</i> ratio)	% of recovery ^b	Trans/ <i>cis</i> ratio of recovered epoxide ^b
1	4d (4.6:1)	84	4.8:1
2	4f (1.4:1)	92	1.4:1
3	4h (5:1)	74	4:1

^a All the epoxidation reactions were performed as follows: A solution of epoxide (0.25 mmol) and **1** (3 μmol) in CH_3CN (4 mL) was added a pre-mixed solution of 0.8 M aqueous NH_4HCO_3 (0.5 mL), CH_3CN (0.5 mL) and 35% H_2O_2 (0.10 mL) at room temperature. ^b Determined by ^1H NMR with internal standard.

As illustrated in the above table, no significant change on the *trans*-selectivities of the recovered epoxides **4d**, **4f** and **4h** was observed. These findings indicated that the high *trans*-selectivities observed in epoxidation of **3d**, **3f** and **3h** should not come from preferential decomposition of one of the diastereomeric epoxides.

Procedure for Control Experiment for Diastereoselective Epoxidation of **3d**

To a round-bottom flask containing Mn(TDCPP)(Cl) **1** (3.0 mg, 0.003 mmol) and **4d** (*trans/cis* ratio = 4.6:1) (88.0 mg, 0.25 mmol) in CH₃CN (4 mL) was added a premixed solution of 30% H₂O₂ (0.10 mL), aqueous NH₄HCO₃ (0.8 M, 0.5 mL) and CH₃CN (0.5 mL) via a syringe pump for 1.5 h at room temperature. After being stirred for 1 h, the reaction mixture was diluted with saturated aqueous Na₂S₂O₃ (1 mL) and extracted with n-hexane (4 × 20 mL). The combined organic layers were dried over anhydrous MgSO₄, filtered through a short pad of silica gel, and concentrated under reduced pressure. The % of recovery and ratio of *trans*-**4d** to *cis*-**4d** was determined to be 84% and 4.8:1 respectively, by ¹H NMR with internal standard.

Literature References for Cycloalkenes 3b–3d, 3f–3g, 3i, and 3l–3p

<p>3b</p>	<p>Pearson, A. J.; Hsu, S.-Y. <i>J. Org. Chem.</i> 1986, <i>51</i>, 2505.</p>
<p>3c</p>	<p>Corey, E. J.; Venkateswarlu, A. <i>J. Am. Chem. Soc.</i> 1972, <i>94</i>, 6190.</p> <p>TBDMS = <i>t</i>-Bu(CH₃)₂Si</p>
<p>3d</p>	<p>Detty, M. R.; Seidler, M. D. <i>J. Org. Chem.</i> 1981, <i>46</i>, 1283.</p> <p>TBDPS = <i>t</i>-Bu(Ph)₂Si</p>
<p>3f</p>	<p>Pearson, A. J.; Hsu, S.-Y. <i>J. Org. Chem.</i> 1986, <i>51</i>, 2505.</p>
<p>3g</p>	<p>Corey, E. J.; Venkateswarlu, A. <i>J. Am. Chem. Soc.</i> 1972, <i>94</i>, 6190.</p>
<p>3i</p>	<p>Davies, S. G.; Whitham, G. H. <i>J. Chem. Soc. Perkin Trans. 1</i> 1976, 2279.</p>
<p>3l</p>	<p>van Benthem, Rolf A. T. M.; Michels, J. J.; Hiemstra, H.; Nico Speckamp, W. <i>Synlett</i>. 1994, 368.</p>

<p>3m</p>	<p>Ahn, D.-R.; Mosimann, M.; Leumann, C. J. <i>J. Org. Chem.</i> 2003, 68, 7693.</p>
<p>3n</p>	<p>Crotti, P.; Di Bussolo, V.; Favero, L.; Macchia, F.; Pineschi, M. <i>Eur. J. Org. Chem.</i> 1998, 1675.</p>
<p>3o</p>	<p>Tachihara, T.; Kitahara, T. <i>Tetrahedron</i> 2003, 59, 1773.</p>
<p>3p</p>	<p>Curran, T. T.; Hay, D. A.; Koegel, C. P. <i>Tetrahedron</i> 1997, 53, 1983.</p>

Preparation of Cycloalkenes **3h**, **3j** and **3k**

Preparation of **3h**

A solution of 3-methyl-2-cyclohexen-1-ol (0.49 g, 5 mmol), TBDPSCl (1.01 g, 5.5 mmol), imidazole (0.5 g, 7.3 mmol) in anhydrous DMF (5 mL) was stirred at room temperature for 16 h. The mixture was diluted with EtOAc (50 mL), washed with 1 N HCl, saturated NaHCO₃ solution and brine, and concentrated under reduced pressure. The residue was purified by flash column chromatography (1% EtOAc in hexane) to afford alkene **3h** (1.4 g, 4.0 mmol, 80% yield). Colorless oil, analytical TLC (silica gel 60) (10% EA in hexane), R_f = 0.58; ¹H NMR (400 MHz, CDCl₃) δ 7.70–7.68 (m, 4H), 7.43–7.24 (m, 6H), 5.35 (m, 1H), 4.21 (m, 1H), 1.94–1.79 (m, 1H), 1.78–1.72 (m, 2H), 1.61 (s, 3H), 1.60–1.53 (m, 2H), 1.46–1.37 (m, 1H), 1.06 (s, 9H); ¹³C NMR (75.47 MHz, CDCl₃) δ 136.91, 135.87, 135.82, 134.79, 134.73, 129.41, 129.39, 127.44, 127.42, 125.32, 67.83, 31.93, 30.01, 27.05, 23.59, 19.58, 19.20; IR (KBr) 2931, 1472, 821 cm⁻¹; EIMS m/z 350 (M⁺), 293 (M⁺ - *t*C₄H₉); HRMS (EI) for C₂₃H₃₀OSi, calcd 350.2066, found 350.2062.

Preparation of **3j**

A solution of cyclohex-2-enecarboxylic acid (0.4 g, 3.2 mmol), cyclohexanol (0.635 g, 6.4 mmol), DMAP (0.195 g, 1.6 mmol), EDCI (0.92 g, 4.8 mmol) in anhydrous CH₂Cl₂ (10 mL) were stirred at room temperature for 6 h. The reaction mixture was diluted with CH₂Cl₂ (70 mL), washed with H₂O (2 × 10 mL), and dried over anhydrous Na₂SO₄. The reaction mixture was filtered, and concentrated under reduced pressure. The residue was purified by flash column chromatography (3% EtOAc in hexane) to afford alkene **3j** (0.5 g, 2.4 mmol, 75% yield). Colorless oil, analytical TLC (silica gel 60) (10% EA in hexane), R_f = 0.50; ¹H NMR (300 MHz,

CDCl₃) δ 5.85–5.74 (m, 2H), 4.81–4.77 (m, 1H), 3.06 (m, 1H), 2.02 (m, 2H), 1.92–1.64 (m, 7H), 1.60–1.26 (m, 7H); ¹³C NMR (75.47 MHz, CDCl₃) δ 174.00, 129.36, 124.65, 72.36, 41.39, 31.51, 25.43, 25.32, 24.67, 23.59, 20.80; IR (KBr) 1722 cm⁻¹; EIMS *m/z* 208 (M⁺); HRMS (EI) for C₁₃H₂₀O₂, calcd 208.1463, found 208.1443. (Synthesis of cyclohex-2-enecarboxylic acid, see: Davies, S. G.; Whitham, G. H. *J. Chem. Soc. Perkin Trans. 1* **1976**, 2279.)

Preparation of **3k**

A solution of cyclohex-2-enecarboxylic acid (0.48 g, 3.8 mmol), Benzhydrol (1.4 g, 7.6 mmol), DMAP (0.23 g, 1.9 mmol), EDCI (1.1 mg, 5.7 mmol) and anhydrous CH₂Cl₂ (10 mL) were stirred at room temperature for 6 h. The reaction mixture was diluted with CH₂Cl₂ (70 mL), washed with H₂O (2 × 10 mL), and over anhydrous Na₂SO₄. The reaction mixture was filtered, and concentrated under reduced pressure. The residue was purified by flash column chromatography (3% EtOAc in hexane) to afford alkene **3k** (0.77 g, 2.6 mmol, 69% yield). Colorless oil, analytical TLC (silica gel 60) (10% EA in hexane), R_f = 0.58; ¹H NMR (300 MHz, CDCl₃) δ 7.34–7.26 (m, 10H), 6.88 (s, 1H), 5.86–5.83 (m, 2H), 3.22–3.20 (m, 1H), 2.04–1.75 (m, 5H), 1.63–1.56 (m, 1H); ¹³C NMR (75.47 MHz, CDCl₃) δ 173.77, 140.85, 140.80, 130.25, 128.91, 128.26, 128.24, 127.49, 127.42, 124.50, 77.20, 41.72, 25.67, 25.07, 21.18; IR (KBr) 1715 cm⁻¹; EIMS *m/z* 292 (M⁺), 167 (M⁺ - C₇H₉O₂); HRMS (EI) for C₂₀H₂₀O₂, calcd 292.1463, found 292.1455.

Literature References for Epoxides 4a–4g, 4i and 4m–4p

<p><i>trans</i>-4a <i>cis</i>-4a</p>	<p>Murray, R. W.; Singh, M.; Williams, B. L.; Moncrieff, H. <i>M. J. Org. Chem.</i> 1996, 61, 1830.</p>
<p><i>trans</i>-4b <i>cis</i>-4b</p>	<p>Murray, R. W.; Singh, M.; Williams, B. L.; Moncrieff, H. <i>M. J. Org. Chem.</i> 1996, 61, 1830.</p>
<p><i>trans</i>-4c <i>cis</i>-4c</p>	<p>Kurihara, M.; Ito, S.; Tsutsumi, N.; Miyata, N. <i>Tetrahedron Lett.</i> 1994, 35, 1577.</p>
<p><i>trans</i>-4d <i>cis</i>-4d</p>	<p>Demay, S.; Kotschy, A.; Knochel, P. <i>Synthesis</i> 2001, 863.</p>
<p><i>trans</i>-4e <i>cis</i>-4e</p>	<p>Murray, R. W.; Singh, M.; Williams, B. L.; Moncrieff, H. <i>M. J. Org. Chem.</i> 1996, 61, 1830.</p>
<p><i>trans</i>-4f <i>cis</i>-4f</p>	<p>Murray, R. W.; Singh, M.; Williams, B. L.; Moncrieff, H. <i>M. J. Org. Chem.</i> 1996, 61, 1830.</p>

<p><i>trans</i>-4g <i>cis</i>-4g</p>	<p>Yang, D.; Jiao, G.-S.; Yip, Y.-C.; Wong, M.-K. <i>J. Org. Chem.</i> 1999, <i>64</i>, 1635.</p>
<p><i>trans</i>-4i <i>cis</i>-4i</p>	<p>Murray, R. W.; Singh, M.; Williams, B. L.; Moncrieff, H. M. <i>J. Org. Chem.</i> 1996, <i>61</i>, 1830.</p>
<p><i>trans</i>-4m <i>cis</i>-4m</p>	<p>Ahn, D.-R.; Mosimann, M.; Leumann, C. J. <i>J. Org. Chem.</i> 2003, <i>68</i>, 7693.</p>
<p><i>trans</i>-4n <i>cis</i>-4n</p>	<p>Crotti, P.; Di Bussolo, V.; Favero, L.; Macchia, F.; Pineschi, M. <i>Eur. J. Org. Chem.</i> 1998, 1675.</p>
<p><i>trans</i>-4o</p>	<p>Tachihara, T.; Kitahara, T. <i>Tetrahedron</i> 2003, <i>59</i>, 1773.</p>
<p><i>trans</i>-4p <i>cis</i>-4p</p>	<p><i>trans</i>-4p: Tanaka, H.; Yamada, H.; Matsuda, A.; Takahashi, T. <i>Synlett</i>. 1997, 381. <i>cis</i>-4p: Theil, F. <i>Tetrahedron: Asymmetry</i> 1995, <i>6</i>, 1693.</p>

Characterization Data of epoxide **4h**, and **4j-4l**

Colorless oil, analytical TLC (silica gel 60) (10% EA in hexane), *trans*-**4h** R_f = 0.28, *cis*-**4h** R_f = 0.25; ^1H NMR (400 MHz, CDCl_3) δ 7.96–7.66 (m, 4H), 7.45–7.34 (m, 6H), 4.02–3.96 (m, 1H), 2.93 (br s, $4/5 \times 1\text{H}$), 2.87 (br s, $1/5 \times 1\text{H}$), 1.85–1.35 (m, 5H), 1.29 (s, $4/5 \times 3\text{H}$), 1.26–1.13 (m, 5H), 1.22 (s, $1/5 \times 3\text{H}$), 1.09 (s, $4/5 \times 9\text{H}$), 1.08 (s, $1/5 \times 9\text{H}$); ^{13}C NMR (100.61 MHz, CDCl_3) δ 135.78, 135.70, 134.04, 133.86, 129.68, 129.65, 129.56, 127.62, 127.55, 127.52, 29.96, 29.39, 28.04, 27.66, 26.95, 26.89, 24.05, 23.36, 19.70, 19.17, 15.68; IR (KBr) 2933, 1472, 822 cm^{-1} ; EIMS m/z 366 (M^+), 309 ($\text{M}^+ - t\text{C}_4\text{H}_9$); HRMS (EI) for $\text{C}_{23}\text{H}_{30}\text{O}_2\text{Si}$, calcd 366.2015, found 366.2015.

Colorless oil, analytical TLC (silica gel 60) (10% EA in hexane), R_f = 0.31; ^1H NMR (400 MHz, CDCl_3) δ 4.86–4.80 (m, 1H), 3.41 (d, J = 3.9 Hz, 1H), 3.22–3.20 (m, 1H), 2.87–2.84 (dd, J = 8.6, 5.6 Hz, 1H), 2.08–2.03 (m, 1H), 1.85–1.69 (m, 6H), 1.57–1.25 (m, 9H); ^{13}C NMR (100.62 MHz, CDCl_3) δ 172.97, 72.80, 52.36, 52.24, 40.91, 31.49, 31.43, 25.32, 23.94, 23.78, 23.55, 23.53,

16.80; IR (KBr) 1728 cm^{-1} ; EIMS m/z 224 (M^+); HRMS (EI) for $\text{C}_{13}\text{H}_{20}\text{O}_3$, calcd 224.1412, found 224.14037.

Colorless oil, analytical TLC (silica gel 60) (10% EA in hexane), R_f = 0.25; ^1H NMR (400 MHz, CDCl_3) δ 4.88–4.84 (m, 1H), 3.45 (t, J = 3.5 Hz, 1H), 3.21–3.19 (m, 1H), 2.83–2.78 (m, 1H), 1.90–1.82 (m, 4H), 1.76–1.67 (m, 3H), 1.61–1.23 (m, 9H); ^{13}C NMR (100.62 MHz, CDCl_3) δ 172.24, 72.75, 52.26, 52.18, 41.13, 31.50, 31.47, 25.40, 23.53, 23.58, 23.35, 21.29, 18.91; IR (KBr) 1734 cm^{-1} ; EIMS m/z 125 ($\text{M}^+ - \text{C}_6\text{H}_{11}\text{O}$); HRMS (EI) for $\text{C}_7\text{H}_9\text{O}_2$, calcd 125.0603, found 125.0602.

Colorless oil, analytical TLC (silica gel 60) (20% EA in hexane), R_f = 0.38; ^1H NMR (300 MHz, CDCl_3) δ 7.35–7.26 (m, 10H), 6.92 (s, 1H), 3.45 (d, 3.5 Hz, 1H), 3.21–3.19 (m, 1H), 3.01 (dd, J = 8.8, 6.5 Hz, 1H), 2.08–2.01 (m, 1H), 1.91–1.87 (m, 1H), 1.86–1.70 (m, 1H), 1.49–1.33 (m, 3H); ^{13}C NMR (75.47 MHz, CDCl_3) δ 172.85, 140.44, 140.37, 128.97, 128.95, 128.43, 128.38, 127.46, 127.36, 77.60, 52.65, 52.56, 41.26, 24.29, 24.12, 17.17; IR (KBr) 1732 cm^{-1} ; EIMS m/z 308 (M^+), 183 ($\text{M}^+ - \text{C}_7\text{H}_9\text{O}_2$); HRMS (EI) for $\text{C}_{20}\text{H}_{20}\text{O}_3$, calcd 308.1412, found 308.1407.

Colorless oil, analytical TLC (silica gel 60) (20% EA in hexane), $R_f = 0.30$; ^1H NMR (300 MHz, CDCl_3) δ 7.39–7.23 (m, 10H), 6.94 (s, 1H), 3.54 (t, $J = 3.4$ Hz, 1H), 3.24–3.21 (m, 1H), 2.95–2.89 (m, 1H), 1.91–1.85 (m, 2H), 1.80–1.64 (m, 1H), 1.61–1.57 (m, 2H), 1.29–1.22 (m, 1H); ^{13}C NMR (75.47 MHz, CDCl_3) δ 171.79, 140.20, 140.13, 128.44, 128.40, 127.87, 127.71, 127.20, 126.82, 77.14, 52.12, 51.95, 41.27, 23.23, 21.32, 18.90; IR (KBr) 1738 cm^{-1} ; EIMS m/z 308 (M^+), 183 ($\text{M}^+ - \text{C}_7\text{H}_9\text{O}_2$); HRMS (EI) for $\text{C}_{20}\text{H}_{20}\text{O}_3$, calcd 308.1412, found 308.1407.

Colorless oil; analytical TLC (silica gel 60) (30% EA in hexane), $R_f = 0.67$; ^1H NMR (300 MHz, CDCl_3) δ 4.32 (dd, $J = 10.8, 6.3$ Hz, 1H), 3.29 (m, 1H), 3.22 (d, $J = 3.9$ Hz, 1H), 2.12–2.07 (m, 1H), 1.79–1.68 (m, 2H), 1.52 (s, 18H), 1.48–1.43 (m, 2H), 1.42–1.26 (m, 1H); ^{13}C NMR (75.47 MHz, CDCl_3) δ 152.97, 83.31, 57.54, 54.18, 53.25, 28.57, 25.90, 24.68, 16.81; IR (KBr) 1738, 1698 cm^{-1} ; EIMS m/z 257 ($\text{M}^+ + 1 - t\text{C}_4\text{H}_9$); HRMS (EI) for $\text{C}_{12}\text{H}_{19}\text{O}_5\text{N}$ ($\text{M}^+ + 1 - \text{C}_4\text{H}_9$), calcd 257.1263, found 257.1261.

GC spectra of Diastereomeric mixture of 4c

Table 1, entry 3

¹H NMR Spectra of Diastereomeric Mixtures of Epoxide 4

Table 1, entry 4

Table 1, entry 6

Table 1, entry 7

Table 1, entry 8

Table 1, entry 10

Table 1, entry 11

Table 1, entry 13

Table 1, entry 14

Table 3, entry 2

Table 3, entry 3

Table 3, entry 4

Table 3, entry 6

Current Data Parameters
Name 1d1-124
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 2003023
Time 15.06
INSTRUM a400
PROBHD 5 mm DPX 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
C13 27
NS 27
DS 4
SWH 8012.820 Hz
FIDRES 0.24452 Hz
AQ 2.0447731 sec
RG 655
DM 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====

NUC1 1H
P1 13.20 usec
PL1 -4.00 dB
SFO1 400.1324008 MHz

F2 - Processing parameters

SI 32768
SF 400.1300085 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10 NMR Plot Parameters

CK 29.00 cm
CY 2.33 cm
F1 10.250 ppm
F2 4.01250 Hz
F3 10.250 ppm
F4 -100.03 Hz
BPICM 0.42000 ppm/cm
HZCM 158.09460 Hz/cm

3 in CDCl₃

Current Data Parameters
NAME support15
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031024
Time 10.43
INSTRUM dpx300
PROBHD DUAL 5mm
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 794
DS 1
SWH 18832.393 Hz
FIDRES 0.574719 Hz
AQ 0.8700404 sec
RG 9195.2
DW 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 2.50000000 sec
C11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 7.00 usec
PL1 -6.00 dB
SF01 75.4760204 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 18.00 dB
SF02 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677495 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 25.00 cm
F1P 220.000 ppm
F1 16602.90 Hz
F2P -5.000 ppm
F2 -377.34 Hz
PCKM 9.00000 ppm/cm
HZCM 679.20978 Hz/cm

Current Data Parameters
 NAME jct-374
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20031020
 Time 21.57
 INSTRUM cpx300
 PROBHD DUAL 5mm

PULPROG zgpg
 TD 32768
 SOLVENT CDCl3
 NS 404
 DS 1
 SWH 18832.393 Hz
 FIDRES 0.574719 Hz
 AQ 0.8700404 sec
 RG 8192
 DW 26.350 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.50000000 sec
 d11 0.03000000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 7.00 usec
 PL1 -6.00 dB
 SFO1 75.4760204 MHz

===== CHANNEL f2 =====
 CDPORG2 Waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677190 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 25.00 cm
 F1P 220.000 ppm
 F1 16602.90 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 8.80000 ppm/cm
 HZCM 664.11591 Hz/cm

Current Data Parameters
NAME support13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20031023
Time 11:24
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 25
DS 4
SWH 8012.833 Hz
FIDRES 0.244833 Hz
AQ 2.044731 sec
RG 64
QM 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 13.00 usec
PL1 -4.00 dB
SFO1 400.1324099 MHz

F2 - Processing parameters

SI 32768
SF 400.1300031 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

10 NMR 0.01 Parameters

CX 25.00 cm
CY 4.29 cm
F1P 10.250 ppm
F1 4101.33 Hz
F2P -0.250 ppm
F2 0.13000 ppm/cm
NUC2 13C
HZCM 158.05460 Hz/cm

Current Data Parameters
NAME
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031023
Time 12:02
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TO 32768
SOLVENT CDCl3
NS 250
DS 4
SWH 8012.820 Hz
FIDRES 0.24432 Hz
AQ 2.044731 sec
RG 114
DM 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 13.20 usec
PL1 -4.00 dB
SFO1 400.1324008 MHz

F2 - Processing parameters
SI 32768
SF 400.130051 MHz
KOH
SOLVENT CDCl3
NS 250
DS 4
SWH 8012.820 Hz
FIDRES 0.24432 Hz
AQ 2.044731 sec
RG 114
DM 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

10 NMR D101 parameters
CY 25.00 cm
C1 1.11 cm
F1P 10.250 ppm
F1 4101.33 Hz
F2P -10.250 ppm
F2 -4101.33 Hz
PULPROG zgpg30
PC 1.00
H2CN 158.05460 Hz/cm

Current Data Parameters
NAME pc5-1491
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030912
Time 18.44
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 24
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 0.047721 sec
RG 327.5
DM 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 13.20 usec
PL1 -4.00 dB
SFO1 400.1324008 MHz

F2 - Processing parameters
SI 32768
SF 400.1301598 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
F1 - NMR plot parameters
CA 25.00 cm
CY 9.16 cm
F1P 10.250 ppm
F2P 4101.33 Hz
F3P 10.250 ppm
F2 -100.618 MHz
PPHM 0.42000 ppm/cm
HZCM 159.05460 Hz/cm

trans-4k in CDCl₃

Current Data Parameters
 NAME pcs-14712a
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030812
 Time 15.53
 INSTRUM gpc300
 PROBHD DUAL 5mm

PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 24
 DS 2
 SWH 4486.403 Hz
 FIDRES 0.137219 Hz
 AQ 3.6438315 sec
 RG 322.5
 DM 111.200 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

***** CHANNEL f1 *****
 NUC1 ¹H
 P1 9.00 usec
 PL1 -6.00 dB
 SF01 300.1315007 MHz

F2 - Processing parameters
 SI 32768
 SF 300.130088 MHz
 WDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 25.00 cm
 F1P 10.250 ppm
 F1 3076.33 Hz
 F2P -0.250 ppm
 F2 -75.03 Hz
 PPMCK 0.42000 ppm/cm
 HZCK 126.05460 Hz/cm

cis-4k in CDCl₃

Current Data Parameters
NAME pcd-107c
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030724
Time 15.14
INSTRUM cpx300
PROBHD DUAL 5mm
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 74
DS 2
SMH 4496.403 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 50.5
DM 111.200 usec
DE 5.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SF01 300.1315007 MHz

F2 - Processing parameters
SI 32768
SF 300.1299951 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 25.00 cm
FJP 10.250 ppm
F1 3076.33 Hz
F2P -0.250 ppm
F2 -75.03 Hz
PRGCM 0.42000 ppm/cm
HZCM 126.05460 Hz/cm

Report Data Parameters
NAME support1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030926
Time 19.18

INSTRUM av400
PULPROG zgpg30
PROBHD 5 mm QNP 1H/13

PLEXP0 3299C
TO 32788
SOLVENT CDCl3

NS 623
DS 4
SWH 25125.629 Hz

FIDRES 0.76573 Hz
AQ 0.652132 sec

RG 456.1
DN 19.980 usec

DE 6.00 usec
TE 300.0 K

D1 2.5000000 sec
d11 0.0300000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.10 usec
PL1 -5.00 dB
SFO1 100.628364 MHz

===== CHANNEL f2 =====
CPOPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 12.90 dB
SFO2 400.132007 MHz

F2 - Processing parameters
SI 32768
SF 100.612798 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10 MMR D10t parameters
CX 25.00 cm
CY 7.33 cm
F1P 220.000 DDM
F1 22134.81 Hz
F2P -5.000 DDM
F2 -303.06 Hz
PRGCM 9.00000 DDM/cm
HZCM 909.51501 Hz/cm

Current Data Parameters
NAME support-13
EXNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031023
Time 11:26
INSTRUM av400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 528
DS 0
SHH 26178.010 Hz
FIDRES 0.738889 Hz
AQ 0.6259189 sec
RG 328
BK 19.00 usec
DE 1.00 usec
TE 300.0 K
D1 2.5000000 sec
d11 0.0300000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.10 usec
PL1 -5.00 dB
SFO1 100.628364 MHz

===== CHANNEL f2 =====
COPROG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 12.90 dB
SFO2 400.132007 MHz

F2 - Processing parameters
SI 32768
SF 100.612722 MHz
WDW 4
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 25.00 cm
CY 8.21 cm
F1P 220.000 DDM
F1 22134.81 Hz
F2P -5.000 DDM
F2 -503.06 Hz
PNUC1 9.00000 DDM/cm
HZCM 905.51489 Hz/cm

trans-4j in CDCl₃

Parent Data Parameters
NAME Support 4
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031023

Time 12.04
INSTRUM spect
PROBHD 5 mm QNP
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 413
DS 0
SWH 26178.010 Hz
FIDRES 0.798889 Hz
AQ 0.6259188 sec
RG 2048
DM 19.100 usec
DE 6.00 usec
TE 300.0 K
D1 2.5000000 sec
d11 0.0300000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.10 usec
PL1 -5.00 dB
SFO1 100.6283664 MHz

===== CHANNEL f2 =====
CDDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 12.90 dB
SFO2 400.136007 MHz

F2 - Processing Parameters
SI 32768
SF 100.612714 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10 NMR plot Parameters
CX 25.00 cm
CY 6.48 cm
F1P 220.000 DPM
F1 2214.81 Hz
F2P 500.000 DPM
F2 -503.06 Hz
DPMCM 9.00000 DPM/cm
HZCM 905.51489 Hz/cm

500

Current Data Parameters
NAME PCS-14711
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030812
Time 12.44
INSTRUM gpc300
PROBHD DUAL 5mm

PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 1789

DS 1
SWH 18832.393 Hz
FIDRES 0.574719 Hz
AQ 0.8700404 sec

RG 8192
DM 26.550 usec
DE 6.00 usec

TE 300.0 K
D1 2.50000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====

NUC1 13C
P1 7.00 usec
PL1 -6.00 dB
SF01 75.4760204 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 18.00 dB
SF02 300.1312005 MHz

F2 - Processing parameters

SF 32768
SF 75.4677199 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10 NMR plot parameters

CX 25.00 cm
F1P 220.000 ppm
F1 16602.90 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 9.20000 ppm/cm
HZCM 694.30304 Hz/cm

trans-4k in CDCl₃

Current Data Parameters
NAME DIC5-12712
EXNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030812
Time 18.12
INSTRUM opx300
PROBHD DUAL 5mm

PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 712
DS 1
SWH 18832.393 Hz
FIDRES 0.574719 Hz
AQ 0.8700404 sec
RG 8192
DW 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 2.50000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 7.00 usec
PL1 -6.00 dB
SF01 75.4760204 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 18.00 dB
SF02 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.467548 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 25.00 cm
F1P 220.000 ppm
F1 16602.91 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCK 9.20000 ppm/cm
HZCM 684.30341 Hz/cm

cis-4k in CDCl₃

gent Data Parameters
NAME pcs-107c
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030724
Time 15.21
INSTRUM dpx300
PROBHD DUAL 5mm

PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 187

DS 1
SWH 18832.393 Hz
FIDRES 0.574719 Hz
AQ 0.8700404 sec

RG 8192
DM 26.350 usec
DE 6.00 usec
TE 300.0 K
D1 2.50000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====

NUC1 13C
P1 7.00 usec
PL1 -6.00 dB
SFO1 75.4750204 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters

SI 32768
SF 75.4677072 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 25.00 cm
F1P 220.000 ppm
F1 16602.90 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 9.20000 ppm/cm
HZCM 694.30292 Hz/cm