Supporting information

Novel Catalytic Kinetic Resolution of Racemic Epoxides to Allylic Alcohols

Arnaud Gayet, Sophie Bertilsson and Pher G. Andersson*

Department of Organic Chemistry, Institute of Chemistry, Uppsala University, Box 531, S-751 21
Uppsala, Sweden.

All reactions were conducted under nitrogen or argon using oven-dried glassware (140 °C for at least 6h) and magnetic stirring. THF was freshly distilled from a deep-blue solution of sodium-benzophenone under nitrogen. DBU was distilled from powdered CaH₂ at reduced pressure and stored under argon over 3A molecular sieves. Flash chromatography was performed using Matrex silica gel 60A (37-70 µm). Analytical TLC was carried out utilizing 0.25 mm pre-coated plates from Macherey-Nagel SIL G-60 UV₂₅₄ 60-F and spots were visualized by the use of UV light and ethanolic phosphomolybdic acid, followed by heating. ¹H and ¹³C NMR spectra were recorded in CDCl₃ solutions at 400 and 100.4 MHz respectively. Chemical shifts for protons are reported using the residual CHCl₃ as internal reference (δ 7.26). Carbon shifts are referred to the resonance of CDCl₃ (δ 77.0). Enantiomeric determination was accomplished by analytical GC using a Chirasil Dex-CB column (25 m / 0.25 mm I.D.) and N₂ (12 psi) as the carrier gas.

General procedure for the kinetic resolution

n-BuLi (0.38 mmol, 1.6 M in hexane) was added dropwise over 5 min to a solution of (1*S*,3*R*,4*R*)-3-(pyrrolidiny)methyl-2-azabicyclo[2.2.1]heptane **1** (15 µmol), DIPA (0.36 mmol), and DBU (3.0 mmol) in THF (1.5 mL) at 0 °C. The resulting solution was stirred at 0 °C for 30 min and the racemic epoxide (0.30 mmol) in THF (1.0 mL) containing *n*-dodecane (ca 10 mg, as internal standard for GC analysis) was then added dropwise over a period of 5 min. The reaction mixture was stirred at 0 °C until about 45% or 55% conversion of the epoxide was reached (according to GC analysis). The reaction mixture was then partitioned between saturated aqueous NH₄Cl (5 mL) and Et₂O (10 mL). The phases were separated and the ether layer was washed with 10% aqueous citric acid (2 × 5 mL), water (5 mL), and brine (5 mL) and was dried (MgSO₄). The enantiomeric purity of the products was determined on the crude reaction mixture by chiral GC.

(1S,1R)-1-methylcyclohexene oxide (4) and (1R)-1-methylcyclohex-2-en-1-ol (5).

Racemic 1-methylcyclohexene oxide (40 mg, 0.36 mmol) was transformed to **4** and **5**. After 4h, a conversion of 43% was reached and the reaction quenched. Work-up and column chromatography (pentane/Et₂O:99/1 to 95/5) gave epoxide **4** (18 mg, 45%, 78% ee), and allylic alcohol **5** (15 mg, 30%, 96% ee), with spectroscopic properties identical to those reported¹. Similarly, **4** (12 mg, 38%, 87% ee), and **5** (16 mg, 40%, 94% ee) were isolated from racemic 1-methylcyclohexene oxide after 8h at 0 °C (52 % conversion).

4: *R*_f 0.63 (silica, pentane/EtOAc:4/1); GC (105 °C): *t*_R (*S*) 8.74 min, *t*_R (*R*) 9.09 min.

5: *R*_f 0.42 (silica, pentane/EtOAc:4/1); GC (105 °C): *t*_R (*S*) 12.97 min, *t*_R (*R*) 13.23 min.

Not fully characterized byproduct: (1*R*)-2-methylene-1-cyclohexanol; GC (105 °C isotherm): *t*_R (*S*) 14.79 min, (*R*) 14.97 min (11% yield at 64% conversion, 96% ee)¹.

(1S)-1-ethylcyclohexene oxide (6) and (1R)-1-ethylcyclohex-2-en-1-ol (7).

Racemic 1-ethylcyclohexene oxide² (40 mg, 0.32 mmol) was kinetically resolved into **6** and **7**. After 10h, 40% conversion was reached and the enantiopurities of the epoxide **6** (21 mg, 53%) and allylic alcohol **7** (12 mg, 31%)³ were determined to 30% ee and 90% ee, respectively. At 63% conversion (20h), enantioselectivities of 70% ee for **6** (12 mg, 32%) and 90% ee for **7** (19 mg, 47%) were obtained.

6: *R*_f 0.76 (silica, pentane/EtOAc:4/1); GC (110 °C): *t*_R (*S*) 5.31 min, *t*_R (*R*) 5.38 min.

7: *R*_f 0.36 (silica, pentane/EtOAc:4/1); GC (110 °C): *t*_R (*S*) 9.86 min, *t*_R (*R*) 10.20 min.

(1S,1R)-1-isopropylcyclohexene oxide (8) and (1R)-1-isopropylcyclohex-2-en-1-ol (9).

Racemic 1-isopropylcyclohexene oxide (40 mg, 0.29 mmol) was resolved to produce enantioenriched epoxide **8** and allylic alcohol **9**. At 46% conversion (7h) an optical purity of 73% ee was obtained for **8** (19 mg, 48%). At 52% conversion (10h), **8** was obtained in 92% ee (17 mg, 41%). The ee of the allylic alcohol **9** (17 mg, 41% at 46% conversion; 18 mg, 45% at 52% conversion) could not be determined successfully with the methods applied.

¹ Mori, K.; Hazra, B. G.; Pfeiffer, R. J.; Gupta, A. K.; Lindgren, B. S. *Tetrahedron* **1987**, 42, 2249-2254.

² Wenkert, E. et al. *Helv. Chem. Acta.* **1975**, 58, 1560-1567.

³ Alonso, E.; Diego, J.; Yus, M. *Tetrahedron* **1996**, 52, 14341-14348.

8: R_f 0.46 (silica, pentane/EtOAc:19/1); GC (100 °C): t_R (*S*) 8.30 min, t_R (*R*) 8.50 min. ^1H NMR δ 2.91 (1H, m), 1.97–1.88 (1H, m), 1.79–1.60 (1H, m), 1.72 (2H, t, J = 5.5 Hz), 1.52–1.31 (3H, m), 1.24–1.12 (2H, m), 0.95 (3H, d, J = 6.9 Hz), 0.91 (3H, d, J = 6.9 Hz); ^{13}C NMR δ 63.80, 58.64, 35.69, 25.40, 24.01, 20.80, 19.98, 18.53, 17.45. HRMS (CI^+) $\text{C}_9\text{H}_{16}\text{O}$ requires 140.1201, found 140.1201.

9: R_f 0.15 (silica, pentane/EtOAc:19/1); GC (100 °C): t_R (*S*) and (*R*) 21.88 min. ^1H NMR δ 5.88–5.82 (1H, m), 5.63 (1H, m), 2.09–1.85 (2H, m), 1.77–1.53 (5H, m), 0.95 (3H, d, J = 6.8 Hz), 0.89 (3H, d, J = 6.8 Hz); ^{13}C NMR δ 132.1, 130.6, 78.5, 37.5, 30.7, 25.3, 18.6, 17.5, and 16.4. HRMS (CI^+) $\text{C}_9\text{H}_{16}\text{O}$ requires 140.1201, found 140.1201.

(1*S*,2*R*)-1-*tert*-butylcyclohexene oxide (10) and (1*R*)-1-*tert*-butylcyclohex-2-en-1-ol (11).

Racemic 1-*tert*-butylcyclohexene oxide⁴ (40 mg, 0.26 mmol) was transformed to epoxide **10** and allylic alcohol **11**.⁵ After 17h a conversion of 44 % was reached and the reaction quenched. The enantiopurity was 64% ee for **10** (20 mg, 51%) and 99% ee for **11** (14 mg, 36%). At 58% conversion (26h) the enantiopurity of both **10** (14.4 mg, 36%) and **11** (16 mg, 40%) were 99% ee. ($t_{R,i.s.}$ 13.5 min)

10: R_f 0.71 (silica, pentane/EtOAc:4/1); GC (100 °C): t_R (*R*) 7.51 min, t_R (*S*) 7.59 min.

11: R_f 0.52 (silica, pentane/EtOAc:4/1); GC (100 °C): t_R (*R*) 18.34 min, t_R (*S*) 18.40 min.

Also, about 10% yield of 2-*tert*-Butylcyclohex-2-en-1-ol was obtained in the reaction.⁵

(1*R*,2*S*)-2-2-dimethylcyclohexene (12) oxide and (1*R*)-6-6-dimethylcyclohex-2-enol (13)

Racemic 2-2-dimethylcyclohexene oxide⁶ (45 mg, 0.36 mmol) was transformed to epoxide **12** and allylic alcohol⁷ **13**. After 18h a conversion of 52 % was reached and the reaction quenched. The enantiopurity was 99% ee for **12** (21 mg, 47%) and 94% ee for **13** (19 mg, 43%).

12: GC (100 °C): t_R (*S*) 4.22 min, t_R (*R*) 4.32 min.

13: GC (100 °C): t_R (*R*) 11.64, t_R (*S*) 12.17 min.

⁴ Wang, Z.-X.; Tu, Y.; Frohn, M.; Zhang, J.-R.; Shi, Y. *J. Am. Chem. Soc.* **1997**, *119*, 11224–11235.

⁵ Hudrlik, P. F.; Tafesse, L.; Hudrlik, A. M. *J. Am. Chem. Soc.* **1997**, *119*, 11689–11690.

⁶ Jacobson, E.N.; Deng, L.; Furakawa, Y.; Marines, L.E. *tetrahedron* **1994**, *50*, 423-433.

Preparation of (6,7)-epoxy-spiro[5,5]decane (14) (Standard experimental procedure for epoxidation)

In a 25 mL flask equipped with a magnetic stirrer, 1.03 g (7.56 mmol) of (6,7)spiro[4,5]dec-6-ene and 49 mg (0.2 mmol) of MTO were dissolved in 6 ml of CH₂Cl₂. To this solution 74 μ L (0.91 mmol) of pyridine were added followed by the dropwise addition of 1.28 ml (11.3 mmol) of 30% aq H₂O₂ (ca. 5min). During addition of the H₂O₂ solution the temperature was kept between 20-25°C by application of an external cooling bath. The reaction was stirred over night at R.T. and the aqueous phase was separated and discarded. The remaining H₂O₂ in the yellow organic phase was decomposed to O₂ and H₂O by stirring with a catalytic amount of MnO₂ (ca. 5mg). After the yellow color has completely disappeared the mixture was dried over Na₂SO₄, concentrated and purified by filtration through a short column of silica gel. The column is washed with pentane/EtOAc and concentration of the eluate affords 1 g (87%) of (6,7)-epoxy-spiro[5,5]decane⁸ as colorless oil

(6R,7S)-epoxy-spiro[5,5]decane (14) and (5S,6R)-spiro[4,5]dec-7-en-6-ol (15).

Racemic (6,7)-epoxy-spiro[5,5]decane (55 mg, 0.36 mmol) was transformed to epoxide **14** and allylic alcohol **15**. After 20h a conversion of 56% was reached and the reaction quenched. The enantiopurity was 93% ee for **14** (22 mg, 54%) and 90% for **15** (25 mg, 45%). (*t_R*.s. 5.58 min)

14: GC (120 °C): *t_R* (*S*) 9.35 min and (*R*) 9.42 min, ¹H NMR δ 3.22-3.16 (dt, 1H); 2.82-2.77 (dd, 1H); 1.91-1.04 (m, 14H) δ ; ¹³C NMR δ 60.39, 54.12, 41.82, 37.84, 36.32, 33.04, 25.19, 24.78, 24.18, 18.03. HRMS (CI⁺) C₁₀H₁₆O requires 152.1201, found 152.1202.

15: GC (120 °C): *t_R* (*R*) 20.01 min and (*S*) 20.38 min, ¹H NMR δ 5.84-5.23 (m, 2H), 3.81-3.71 (bs, 1H), 2.13-1.98 (m, 2H), 1.76-1.56 (m, 6H), 1.49-1.23 (m, 4H); ¹³C NMR δ 130.82, 129.46, 72.23, 45.95, 34.82, 33.98, 29.67, 25.40, 25.30, 23.75. HRMS (CI⁺) C₁₀H₁₆O requires 152.1201, found 152.1202.

(1S,2R)-1-tert-butylcyclopentene oxide (16) and (1R)-1-tert-butylcyclopent-2-en-1-ol (17).

⁷ Magnusson, M.; Thoren, S. *J. Org. Chem.* **1973**, 38, 1380-1384.

Racemic 1-*tert*-butylcyclopentene oxide⁹ (50 mg, 0.36 mmol) was transformed to epoxide **16** and allylic alcohol **17**. After 13h a conversion of 42 % was reached and the reaction quenched. The enantiopurity was 79% ee for **17** (20 mg, 37%), but the enantiomers of **16** (25 mg, 50%) could not be separated on the column. (*t_R*.s. 12.7 min)

16: GC (100 °C): *t_R* (*S*) and *t_R* (*R*) 6.85 min.

17: GC (100 °C): *t_R* (*S*) 15.28 *t_R* (*R*) 15.84 min. ¹H NMR δ 5.93-5.93 (1H, m), 5.80-5.76 (1H, m), 2.55-2.44 (1H, m), 3.30-2.16 (2H, m), 1.72-1.63 (1H,m), 0.96-0.90 (9H, s); ¹³C NMR δ 134.5, 134.4, 91.5, 37.16, 34.19, 31.80 and 25.58. HRMS (CI⁺) C₉H₁₆O requires 140.1201, found 140.1202.

Preparation of 1-*tert*-butylcycloheptene oxide (**18**).

1-*tert*-butylcycloheptene¹⁰ was epoxidised using the procedure describe above to afford 1-*tert*-butylcycloheptene oxide in 86% yield as a colorless oil.

(1*S*,2*R*)-1-*tert*-butylcycloheptene oxide (**18**) and (1*R*)-1-*tert*-butylcyclohept-2-en-1-ol (**19**).

Racemic 1-*tert*-butylcycloheptene oxide (60 mg, 0.36 mmol) was transformed to epoxide **18** and allylic alcohol **19**. After 9h a conversion of 41 % was reached and the reaction quenched. The enantiopurity was 94% ee for **19**(31 mg, **51%**), but the enantiomers of **19** (20 mg, 34%) could not be separated on the column. (*t_R*.s. 6.8 min)

18: GC (115 °C): *t_R* (*S*) and *t_R* (*R*) 10.95 min. ¹H NMR δ 3.17-3.12 (1H, t); 1.89-1.81 (4H, m); 1.58-1.17 (6H, m); 0.94-0.88 (9H, bs). ¹³C NMR δ 67.50, 58.85, 34.99, 31.75, 29.2, 29.09, 26.26, 25.13, 24.18. HRMS (CI⁺) C₁₁H₂₀O requires 168.1514, found 168.1514.

19: (115 °C): *t_R* (*S*) 21.34 min, *t_R* (*R*) 21.40 min. ¹H NMR δ 5.80-5.72 (1H, m), 5.63-5.58 (1H, m), 2.23-2.12 (1H, m), 2.10-2.01 (1H, m), 1.80-1.64 (5H, m), 1.60-1.49 (2H, m), 1.05-0.93 (9H, s) ; ¹³C NMR δ 134.74, 130.12, 95.68, 30.07, 31.29, 26.16, 25.24, 25.00 and 21.55. HRMS (CI⁺) C₁₁H₂₀O requires 168.1514, found 168.1515.

trans-4,5-dimethylcyclohexene oxide (**20**) and (1*S*,4*R*,5*R*)-*trans*-4,5-dimethylcyclohex-2-en-1-ol (**21**).

⁸ Krapcho, A.P.; Donn, R. *J Org. Chem.* **1965**, 30, 641.

⁹ Wang, Z.-X.; Tu, Y.; Frohn, M.; Zhang, J.-R.; Shi, Y. *J. Am. Chem. Soc.* **1997**, 119, 11224–11235.

Racemic *trans*-4,5-dimethylcyclohexene oxide (40 mg, 0.32 mmol)¹¹ was converted to the enantioenriched allylic alcohol **21**. A conversion of 50% was reached after 14h and **21** (40% ee) was isolated. The epoxide **20** was not successfully separated with the methods applied.

20: GC (100 °C): t_R (*S*) and (*R*) 6.11 min.

21: GC (100 °C): t_R (*S*) 10.83 min, t_R (*R*) 11.55 min. ¹H NMR δ 5.78–5.72 (1H, m), 5.64 (1H, dd, J = 9.9, 2.1 Hz), 4.13 (1H, m), 2.00–0.78 (4H, m), 1.01 (3H, d, J = 7.2 Hz), 0.97 (3H, d, J = 6.1 Hz). HRMS (CI⁺) C₈H₁₅O requires 126.1045, found 126.1046.

(1*S*,1*R*)-1-isopropylcyclohexene oxide (8).

¹⁰ Fadel, A.; Salaum, J. *Tetrahedron*, E.N., **1985**, 1267-1276.

¹¹ Gajewski, J.J.; Olson, F.P.; Wilcot, M.R. *J. Am. Chem. Soc.* **1996**, *118*, 299-306.

13C OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Temp: 25.0 C / 298.1 K
 UNIT: 400 "uppkems2"
 User: arnaud 2002
 Date: 11/11/02
 PULSE SEQUENCE: s2pul
 Relax. delay 1.000 sec
 Pulse: 12.4 degrees
 Width: 1.000 sec
 Mch: 25000.0 Hz
 288 repetitions
 OBSERVE CH: 100.57 MHz
 DECOUPLE 1: 399.95 MHz
 Power: 46 dB
 continuously on
 Waltz-16 modulated
 Spin Decoupling
 Data Processing
 Line broadening 1.0 Hz
 F2 size 65536
 Total time 10 minutes

s499p_f15-35
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp 25.0 C / 298.1 K
 UNITY-400 "uppkems2"
 User sophie
 Date Nov 11 1999
 PULSE SEQUENCE: s2pul
 Pulse 41.400 sec
 Acq time 3.744 sec
 Width 6000.6 Hz
 20 repetitions
 OBSERVE H1, 399.95 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536
 Total time 1 minute

(1R)-1-isopropylcyclohex-2-en-1-ol (9).

s670p_f9-20
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Temp: 25.0 C / 298.1 K
 UNITY-400 "uppkems2"
 User: sophie
 Date: Aug 10 2000
 PULSE SEQUENCE: s2pul
 Acquisition: 4 cycles
 Acquisition time: 3.74 sec
 Width: 600.6 Hz
 4 repetitions
 OBSERVE: H1, 399.95 MHz
 DATA PROCESSING
 Relaxing: 0.2 Hz
 FT size: 65536
 Total time: 1 minute

Et2O

(6,7)-epoxy-spiro[5,5]decane (14)

13C OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Temp: 25.0 C / 298.1 K
 UNPR-400 "updtmws2"
 User: arnaud 2002
 PULSE SEQUENCE: s2pul
 Relax. delay: 1.000 sec
 Pulse: 62.4 degrees
 Width: 1.000 sec
 Width: 2500.0 Hz
 384 repetitions
 OBSERVE C13, 100.57 MHz
 CHANNEL P1, 399.55 MHz
 Power 46 dB
 continuously on
 WALTZ-16 modulated
 Spin Echo on data
 DATA PROCESSING
 Line broadening 1.0 Hz
 Ft size 65536
 Total time 14 minutes

```

Pulse Sequence: s2pul
Solvent: GDC13
UNIT1: 398.1 K
UNIT2: 400 "uopkwmv2"
User: arnaud
Date: Apr 2 2002
PULSE SEQUENCE: s2pul
Acq time: 3.34 sec
Width: 6000.6 Hz
32 repetitions
OAT: 399.95 MHz
DATA PROCESSING
Ft. size: 65936
Total time: 2 minutes

```

(5S,6R)-spiro[4,5]dec-7-en-6-ol (15).

AC298_alcohol
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Temp: 25.0 C / 298.1 K
 UNIFY-400 "updkms2"
 User: Arnaud
 Date: 2001
 PULSE SEQUENCE: s2pul
 Pulse 1: 1.4 degrees
 Width: 600.6 Hz
 24 repetitions
 OBSERVE: H1MG 399.95 MHz
 P1: 12.00 sec
 Line broadening 0.2 Hz
 FT size 65536
 Total time 1 minutes

(1R)-1-*tert*-butylcyclopent-2-en-1-ol (17).

123_2_allilic_alcohol
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-2000 "gemini"
User: alan
Date: 12/27/2000
PULSE SEQUENCE: s2pul
Pulse: 28.8 degrees
Pulse width: 12.00 sec
Acq time: 250.00 Hz
40 repetitions
OBSERVE: H1
P1: 199.97 MHz
DATA PROCESSING
File: preceding 0.2 Hz
FT size: 16384
Total time: 2 minutes

+ pentane

(1S,2R)-1-*tert*-butylcycloheptene oxide (18)

STANDARD IN OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl₃ 298.1 K
 CW 2.0 "upkems2"
 UNIT-400
 User: arnaud
 Date: Mar 15 2002
 PULSE SEQUENCE: s2pul
 Pulse: 1.000000 sec
 Acq: 1.374556 sec
 Width: 6000.6 Hz
 32 repetitions
 OBSERVE: 399.95 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536
 Total time 2 minutes

3.5 3.0 2.5 2.0 1.5 1.0 ppm

1.00

4.51

5.46

1.65

9.34

(1R)-1-*tert*-butylcyclohept-2-en-1-ol (19).

AG126_alcohol
Pulse Sequence: s2pul
Solvent: CDCl₃ 298.1 K
Temp: 25.0 °C
UNITY: 400 "uppkems2"
User: arnaud
Date: Jan 14 2002
Pulse program: s2pul
Pulse 1: 12.000 sec
Acq. time: 3.744 sec
Width: 6000.6 Hz
Sweep rate: 1000.0 Hz/sec
OBSERVE: 1H
F2: 399.95 MHz
DATA PROCESSING
Fine broadening: 0.2 Hz
Phase: 0.00
Total time: 1 minutes

GSP GC chromatograms

Entry 1:

Print Date: Thu Dec 09 10:01:57 1999

Page 1 of 1

Title :
Run File : c:\star\chromatogram\sophie\s500\s439_443\s439_p1.run
Method File : c:\star\chromatogram\sophie\s500\s439_443\untitled.mth
Sample ID : s439p

Injection Date: 1999-08-24 17:05 Calculation Date: 1999-12-09 10:01

Operator :
Workstation :
Instrument : Varian Star #1
Channel : A = FID 0.1V
Detector Type: ADCB (10 Volts)
Bus Address : 16
Sample Rate : 10.00 Hz
Run Time : 44.002 min

** Star Chromatography Workstation Version 5.2 ** 00186-5E08-C93-22F8 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		4.6366	8.740	0.000	1743	BV	8.8	78% ee
2		37.7464	9.090	0.000	14193	VB	9.5	
3		29.5299	12.977	0.000	11104	BV	0.0	96% ee
4		0.6663	13.229	0.000	251	VB	9.7	
5		22.4221	14.786	0.000	8431	BV	0.0	64% ee
6		4.9986	14.996	0.000	1880	VB	11.5	
Totals:		99.9999		0.000	37602			

Total Unidentified Counts : 37602 counts

Entry 2

Print Date: Thu Dec 09 11:56:16 1999

Page 1 of 1

Title : ms1643_48h.run
Run File : c:\star\chromatogram\lmikael\1000-1200\ms1143-47ch\ms1143_48h.run
Method File : c:\star\chromatogram\sophie\~s500\s439_443\untitled.mth
Sample ID : Manual Sample

Injection Date: 1998-11-02 08:29 Calculation Date: 1999-12-09 11:55

Operator : Detector Type: ADCB (10 Volts)
Workstation: Bus Address : 16
Instrument : Varian Star #1 Sample Rate : 10.00 Hz
Channel : A = FID 0.1V Run Time : 17.602 min

** Star Chromatography Workstation Version 5.2 ** 00186-5E08-C93-22F8 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		19.2402	5.861	0.000	84322	BV	0.0	
2		1.3838	5.930	0.000	6065	VB	0.0	
3		0.4253	10.606	0.000	1864	BB	4.3	
4		22.9815	10.937	0.000	100718	BB	6.3	
5		6.2309	13.254	0.000	27307	BB	1.8	U
6		47.5561	13.579	0.000	208419	BV	0.0	
7		2.1823	13.648	0.000	9564	VB	0.8	
Totals:		100.0001		0.000	438259			

Status Codes:
U - User-defined peak endpoint(s)

Total Unidentified Counts : 438259 counts

Detected Peaks: 7 Rejected Peaks: 0 Identified Peaks: 0

Multiplier: 1 Divisor: 1 Unidentified Peak Factor: 0

Baseline Offset: 30850 microVolts

Offset (used): 17470 microVolts - monitored before this run

Peak not split: event not in an existing peak

Entry 3:

Title :
Run File : c:\star\chromatogram\sophie\s500\s477_78_80\s477_51.run
Method File : c:\star\chromatogram\lmikael\1400-1500\ms1409-1444\untitled.mth
Sample ID : s477_5

Injection Date: 1999-09-22 11:07 Calculation Date: 1999-12-09 19:37

Operator :
Workstation:
Instrument : Varian Star #1
Channel : A = FID 0.1V
Detector Type: ADCB (10 Volts)
Bus Address : 16
Sample Rate : 10.00 Hz
Run Time : 33.002 min

** Star Chromatography Workstation Version 5.2 ** 00186-5E08-C93-22F8 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		45.8229	5.273	0.000	8382	BV	2.3	} 29% ee
2		25.3709	5.341	0.000	4641	VB	2.7	
3		1.4657	9.871	0.000	268	BB	4.5	} 90% ee
4		27.3406	10.186	0.000	5001	BB	5.1	
Totals:		100.0001		0.000	18292			

Entry 4 (100°C):

Date: Thu Dec 16 13:34:00 1999 Page 1 of 2

Title :
Run File : c:\star\chromatogram\sophie\s600\s505_fin1.run
Method File : c:\star\chromatogram\lmikael\c-hex.mth
Sample ID : s505_fin

Injection Date: 1999-12-16 11:31 Calculation Date: 1999-12-16 13:33

Operator : Detector Type: ADCB (10 Volts)
Workstation: Bus Address : 16
Instrument : Varian Star #1 Sample Rate : 10.00 Hz
Channel : A = FID .5V Run Time : 22.400 min

** Star Chromatography Workstation Version 5.2 ** 00186-5E08-C93-22F8 *

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)
1		11.6534	6.666	0.000	55376	BV	4.1
2		2.1120	6.797	0.000	10036	VB	0.0
3		66.0404	13.307	0.000	313817	BB	10.2
4		0.7882	14.248	0.000	3746	BB	7.4
5		15.7241	14.746	0.000	74719	BB	11.9
6		3.6818	19.315	0.000	17496	BB	10.8
Totals:		99.9999		0.000	475190		

Status Codes:

Entry 5:

may 5

Print Date: Thu Jun 27 10:09:00 2002 Page 1 of 1

Title :
Run File : c:\star\sophie\s700\s669_s670\s670_61.run
Method File : c:\star\sophie\s700\s669_s670\untitled.mth
Sample ID : s670_6

Injection Date: 2000-08-09 10:52 Calculation Date: 2002-06-27 10:08

Operator :
Workstation: Detector Type: ADCB (10 Volts)
Bus Address : 18
Instrument : Varian Star #1 Sample Rate : 10.00 Hz
Channel : A = A 10 Run Time : 28.002 min

** Star Chromatography Workstation Version 5.2 ** 00186-5E08-C93-22F8 *

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result (%)	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)
1		13.1703	6.188	0.000	12683	BB	4.4
2		1.3525	8.374	0.000	1302	BV	0.0
3		42.2573	8.496	0.000	40692	VB	5.2
4		43.2199	21.882	0.000	41619	BB	13.5
Totals:		100.0000		0.000	96296		

Total Unidentified Counts : 96296 counts

Detected Peaks: 9 Rejected Peaks: 5

Entry 6:

Print Date: Thu Jun 27 10:05:41 2002

Page 1 of 1

Title :
Run File : c:\star\sophie\s700\s669_s670\s669_31.run
Method File : c:\star\sophie\s700\s669_s670\untitled.mth
Sample ID : s669_3

Injection Date: 2000-08-08 16:27 Calculation Date: 2002-06-27 10:05

Operator :
Workstation:
Instrument : Varian Star #1
Channel : A = A 10
Detector Type: ADCB (10 Volts)
Bus Address : 18
Sample Rate : 10.00 Hz
Run Time : 28.002 min

** Star Chromatography Workstation Version 5.2 ** 00186-5E08-C93-22F8 *

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)
1		7.5953	6.166	0.000	4017	BB	4.3
2		9.8379	8.342	0.000	5204	BV	0.0
3		45.6474	8.471	0.000	24145	VB	4.9
4		36.9195	21.924	0.000	19528	BB	13.2
Totals:		100.0001		0.000	52894		

Entry 7:

Chiral GCMS has been use here for the ee determination due to a better base line. The conversion has been determined using normal CSP.

Chromatogram Plot

File: c:\saturnws\data\ameud\524-529\525-end.sms
Sample: 525-End
Scan Range: 1 - 1415 Time Range: 0.00 - 21.98 min.

Operator: RA
Date: 2002-07-04 10:30

File: 04 Jul 2002 14:51:00

Sample Report for 525-end.sms

Sample ID: 525-End Operator: None
Instrument ID: Last Calibration: None
Acquisition Date: 2002-07-04 10:30 Data File: c:\... \525-end.sms
Calculation Date: 2002-07-04 14:50 Method: c:\... \525-end.sms
Inj. Sample Notes:

Target Compounds

None

Unidentified Peaks

Quan Ions:		Spectrum Match Type:				
RF Used:		Match Thresh:				
#	RT	Compound Name	Status	Area	Amount	Match
1	7.518	No Search	Unk.	877079	4.677	N/A
2	7.587	No Search	Unk.	6217663	33.152	N/A
3	7.593	No Search	Unk.	5183460	27.638	N/A
4	8.551	No Search	Unk.	2987144	15.927	N/A
5	18.341	No Search	Unk.	3489684	18.607	N/A

This chromatogram has been done at 80°C in order to obtain a better separation of the epoxide enantiomers.

Target Compounds
 None

Unidentified Peaks

Quan Ions:		RIC	Spectrum Match Type:		N/A	
RF Used:		1.000	Match Thresh:		N/A	
#	RT	Compound Name	Status	Area	Amount	Match
1	19.584	No Search	Unk.	1510151	10.660	N/A
2	19.855	No Search	Unk.	6528395	46.082	N/A
3	19.857	No Search	Unk.	2434029	17.181	N/A
4	22.541	No Search	Unk.	3694429	26.078	N/A

Entry 8:

Chiral GCMS has been use here for the ee determination due to a better base line. The conversion has been determined using normal CSP.

Target Compounds

None

Unidentified Peaks

Quan Ions: RIC
RF Used: 1.000

Spectrum Match Type: N/A
Match Thresh: N/A

#	RT	Compound Name	Status	Area	Amount	Match
1	7.602	No Search	Unk.	3003482	30.487	N/A
2	7.609	No Search	Unk.	3000245	30.454	N/A
3	18.310	No Search	Unk.	3847903	39.059	N/A

Entry 9:

Print Date: 05 Jul 2002 13:55:54

Chromatogram Plots

Plot 1: c:\saturnws\data\arnaud\524-529\526-527-ref.sms RIC all

Plot 2: c:\saturnws\data\arnaud\524-529\527-end.sms RIC all

Print Date: Thu Jul 04 15:07:56 2002

Page 1 of 1

Title :
Run File : c:\star\arnaud\ag524-529\ag526-end1.run
Method File : c:\star\arnaud\method\100'c.mth
Sample ID : AG526-end

Injection Date: 2002-07-04 14:03

Calculation Date: 2002-07-04 15:07

Operator : nono
Workstation: Detector Type: ADCB (10 Volts)
Instrument : Varian Star #1 Bus Address : 18
Channel : A = A 10 Sample Rate : 10.00 Hz
Run Time : 22.002 min

** Star Chromatography Workstation Version 5.2 ** 00186-5E08-C93-22F8 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result (%)	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		3.0263	11.639	0.000	6198	BP	0.0	
2		96.9738	12.169	0.000	198614	PB	13.8	
Totals:		100.0001		0.000	204812			

Entry 10:

Print Date: Fri Jul 05 13:11:56 2002

Page 1 of 1

Title :
Run File : c:\star\arnaud\524-529\529-ag529-f1001.run
Method File : c:\star\arnaud\method\120.c.mth
Sample ID : AG529-F

Injection Date: 2002-07-05 12:13 Calculation Date: 2002-07-05 13:11

Operator : nono
Workstation:
Instrument : Varian Star #1
Channel : A = A 10
Detector Type: ADCB (10 Volts)
Bus Address : 18
Sample Rate : 10.00 Hz
Run Time : 26.002 min

** Star Chromatography Workstation Version 5.2 ** 00186-5E08-C93-22F8 **

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	1/2 (sec)	Status Codes
1		28.4186	4.363	0.000	512398	BB	6.6	Internal Standard
2		1.3973	9.352	0.000	25194	BV	0.0	
3		35.6117	9.426	0.000	642092	VB	11.1	
4		1.9962	20.014	0.000	35992	BV	0.0	
5		32.5762	20.382	0.000	587361	VB	30.3	
Totals:		100.0000		0.000	1803037			

Entry 11:

Print Date: Thu Jun 27 14:51:04 2002

Page 1 of 1

Title :
Run File : c:\star\arnaud\tbucyclopentene oxide\ag128-20h1.run
Method File : c:\star\sophie\s700\s669_s670\untitled.mth
Sample ID : AG128-20h

Injection Date: 2000-07-26 10:37 Calculation Date: 2002-06-27 14:50

Operator :
Workstation:
Instrument : Varian Star #1
Channel : A = A 10
Detector Type: ADCB (10 Volts)
Bus Address : 18
Sample Rate : 10.00 Hz
Run Time : 18.002 min

** Star Chromatography Workstation Version 5.2 ** 00186-5E08-C93-22F8 *

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Width Sep. 1/2 Code (sec)
1		44.5600	6.852	0.000	41374	BB 5.3
2		0.3674	13.729	0.000	341	BB 2.4
3		10.6800	13.851	0.000	9916	BB 2.1
4		44.3926	13.966	0.000	41219	BB 2.2
Totals:		100.0000		0.000	92850	

Entry 12:

Print Date: Thu Jun 27 14:49:43 2002

Page 1 of 1

Title :
Run File : c:\star\arnaud\tbucyclopentene oxide\ag131-7h1.run
Method File : c:\star\sophie\s700\s669_s670\untitled.mth
Sample ID : AG131-7h

Injection Date: 2000-08-22 17:50 Calculation Date: 2002-06-27 14:49

Operator :
Workstation:
Instrument : Varian Star #1
Channel : A = A 10
Detector Type: ADCB (10 Volts)
Bus Address : 18
Sample Rate : 10.00 Hz
Run Time : 22.002 min

** Star Chromatography Workstation Version 5.2 ** 00186-5E08-C93-22F8 *

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)
1		48.3151	6.919	0.000	32749	BB	7.3
2		21.2406	12.716	0.000	14397	BB	8.3
3		3.0844	15.289	0.000	2091	BB	8.9
4		27.3599	15.842	0.000	18545	BB	10.0
Totals:		100.0000		0.000	67782		

Entry 13:

Entry: 13

Print Date: Thu Jun 27 10:34:42 2002

Page 1 of 1

Title :
Run File : c:\star\sophie\tbucycloheptene oxide\ag130-7h1.run
Method File : c:\star\sophie\s700\s669_s670\untitled.mth
Sample ID : AG130-7h

Injection Date: 2000-08-22 17:22 Calculation Date: 2002-06-27 10:34

Operator :
Workstation: Detector Type: ADCB (10 Volts)
Instrument : Varian Star #1 Bus Address : 18
Channel : A = A 10 Sample Rate : 10.00 Hz
Run Time : 24.002 min

** Star Chromatography Workstation Version 5.2 ** 00186-5E08-C93-22F8

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)
1		18.9565	6.934	0.000	5807	BB	4.5
2		36.8437	10.951	0.000	11286	BB	6.8
3		3.8810	21.372	0.000	1189	BV	0.0
4		40.3189	21.431	0.000	12351	VB	1.7
Totals:		100.0001		0.000	30633		

Entry 14:

Print Date: Thu Jun 27 10:30:11 2002

Page 1 of 1

Title :
Run File : c:\star\arnaud\tbucycloheptene oxide\ag119-36h1.run
Method File : c:\star\sophie\s700\s669_s670\untitled.mth
Sample ID : AG120-36h

Injection Date: 2000-07-19 10:29 Calculation Date: 2002-06-27 10:29

Operator :
Workstation:
Instrument : Varian Star #1
Channel : A = A 10
Detector Type: ADCB (10 Volts)
Bus Address : 18
Sample Rate : 10.00 Hz
Run Time : 24.002 min

** Star Chromatography Workstation Version 5.2 ** 00186-5E08-C93-22F8 *

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)
1		40.6308	6.869	0.000	21302	BB	4.3
2		31.2675	10.879	0.000	16393	BB	6.8
3		0.9113	20.868	0.000	478	BB	2.8
4		0.6939	21.344	0.000	364	BV	0.0
5		26.4966	21.401	0.000	13892	VB	1.9
Totals:		100.0001		0.000	52429		