Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry


# Supporting Information-II

## Observation of Neighboring *ortho*-Hydroxyl Group Participation in Organocatalytic Asymmetric Sequential Michael-Lactonization Reactions: Synthesis of Highly Substituted Chiral Spirodihydrocoumarins

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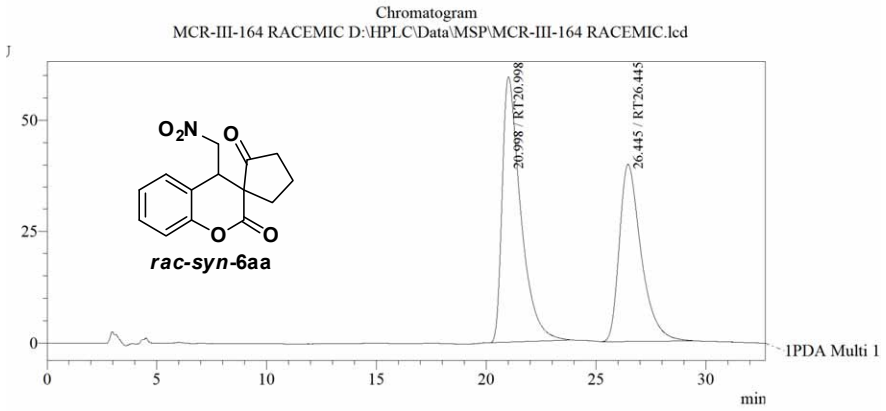
**General Methods:** The $^{1}$H NMR and $^{13}$C NMR spectra were recorded at 400 MHz and 100 MHz, respectively. The chemical shifts are reported in ppm downfield to TMS ($\delta = 0$) for $^{1}$H NMR and relative to the central $CDCl_3$ resonance ($\delta = 77.0$) for $^{13}$C NMR. *In the $^{13}$C NMR spectra, the nature of the carbons (C, CH, $CH_2$ or $CH_3$) was determined by recording the DEPT-135 experiment, and is given in parentheses.* The coupling constants *J* are given in Hz. Column chromatography was performed using Acme's silica gel (particle size 0.063-0.200 mm). High-resolution mass spectra were recorded on micromass ESI-TOF MS. GCMS mass spectrometry was performed on Shimadzu GCMS-QP2010 mass spectrometer. IR spectra were recorded on JASCO FT/IR-5300. Elemental analyses were recorded on a Thermo Finnigan Flash EA 1112 analyzer. Mass spectra were recorded on either VG7070H mass spectrometer using EI technique or Shimadzu-LCMS-2010 A mass spectrometer. The X-ray diffraction measurements were carried out at 298 K on an automated Enraf-Nonious MACH 3 diffractometer using graphite monochromated, Mo-Kα ($\lambda = 0.71073$ Å) radiation with CAD4 software or the X-ray intensity data were measured at 298 K on a Bruker SMART APEX CCD area detector system equipped with a graphite monochromator and a Mo-Kα fine-focus sealed tube ($\lambda = 0.71073$ Å). For thin-layer chromatography (TLC), silica gel plates Merck 60 F254 were used and compounds were visualized by

irradiation with UV light and/or by treatment with a solution of *p*-anisaldehyde (23 mL), conc. $H_2SO_4$ (35 mL), acetic acid (10 mL), and ethanol (900 mL) followed by heating.

The enantiomeric excess (*ee*) of the *M-L* products was determined by chiral stationary phase HPLC using a Daicel Chiralcel OD-H or OJ-H column and hexane/2-propanol as the eluent **(or)** using a Daicel Chiralpak AD-H or AS-H column and hexane/2-propanol as the eluent. Retention times and solvent ratios are indicated in the respective entries.

**RACEMIC 6aa:**



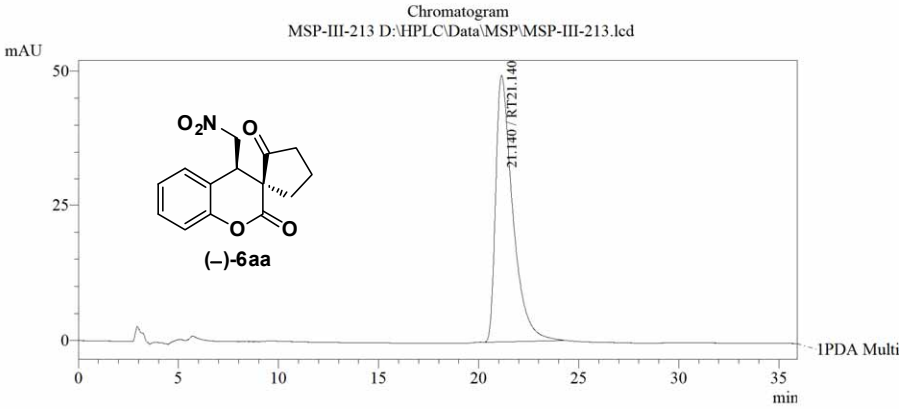

**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT20.998 | 20.998 | 3501206 | 59556 | 56.140 | 59.976 |
| 2 | RT26.445 | 26.445 | 2735356 | 39744 | 43.860 | 40.024 |
| Total | | | 6236562 | 99301 | 100.000 | 100.000 |

**CHIRAL-(–)-6aa (>99.9% *ee*):**



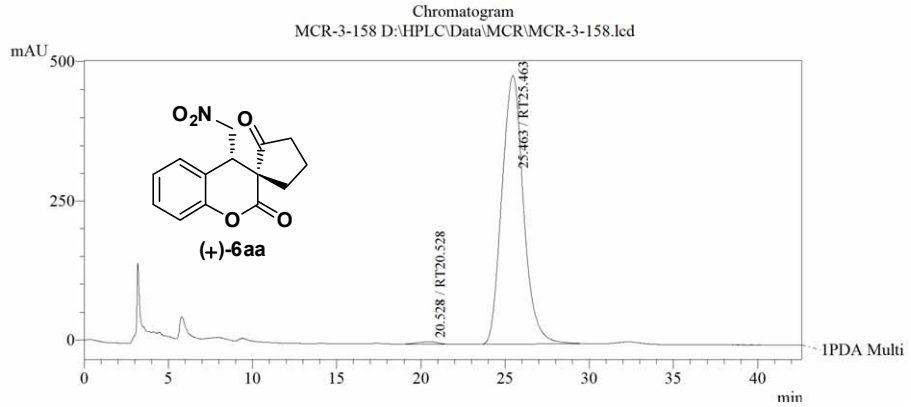

**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT21.140 | 21.140 | 2929263 | 49382 | 100.000 | 100.000 |
| Total | | | 2929263 | 49382 | 100.000 | 100.000 |

## CHIRAL-(+)-6aa (98% *ee*):



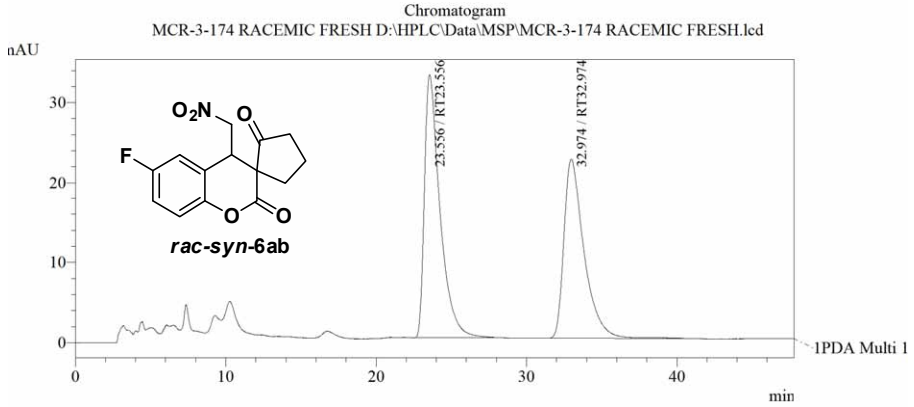

**Daicel ChiralCel OD-H, Hexane/ *i*-PrOH =80:20, Flow Rate 1.0 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT20.528 | 20.528 | 368565 | 4588 | 0.847 | 0.943 |
| 2 | RT25.463 | 25.463 | 43158451 | 482176 | 99.153 | 99.057 |
| Total | | | 43527017 | 486764 | 100.000 | 100.000 |

**RACEMIC 6ab:**



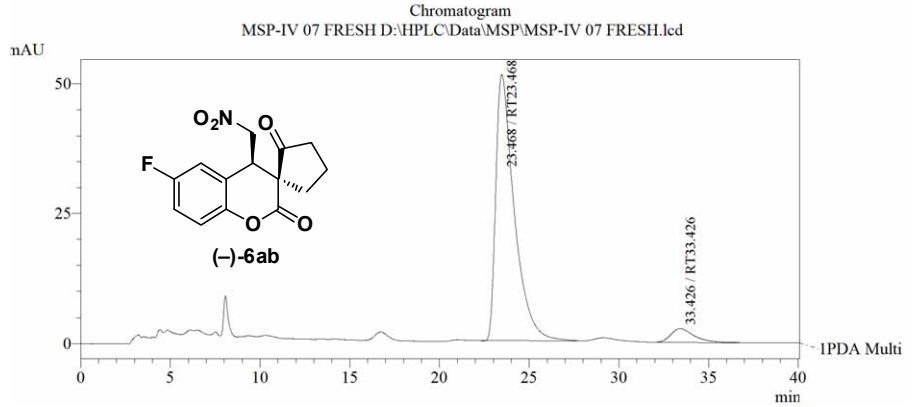

**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT23.556 | 23.556 | 2357276 | 32860 | 53.186 | 59.484 |
| 2 | RT32.974 | 32.974 | 2074868 | 22382 | 46.814 | 40.516 |
| Total | | | 4432144 | 55242 | 100.000 | 100.000 |

**CHIRAL-(–)-6ab (88% *ee*):**



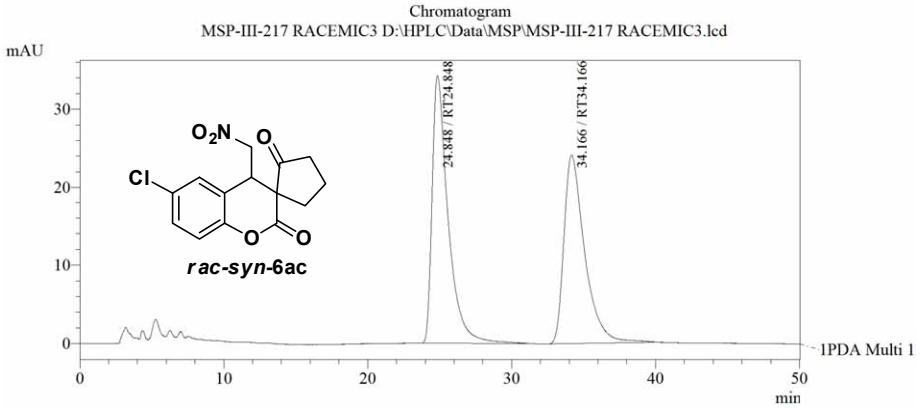

**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT23.468 | 23.468 | 3641664 | 51081 | 93.946 | 95.096 |
| 2 | RT33.426 | 33.426 | 234690 | 2634 | 6.054 | 4.904 |
| Total | | | 3876355 | 53715 | 100.000 | 100.000 |

**RACEMIC 6ac:**



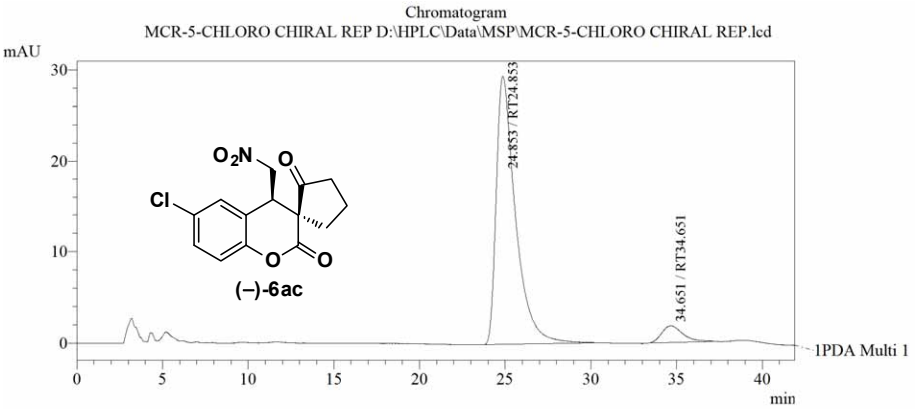

**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT24.848 | 24.848 | 2630053 | 34228 | 52.117 | 58.668 |
| 2 | RT34.166 | 34.166 | 2416371 | 24114 | 47.883 | 41.332 |
| Total | | | 5046423 | 58342 | 100.000 | 100.000 |

**CHIRAL-(–)-6ac (87% *ee*):**



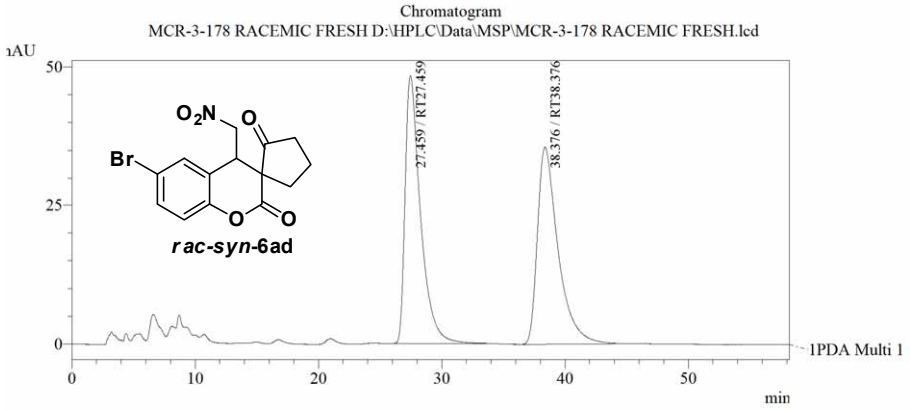

**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT24.853 | 24.853 | 2292412 | 29426 | 93.449 | 94.123 |
| 2 | RT34.651 | 34.651 | 160707 | 1837 | 6.551 | 5.877 |
| Total | | | 2453119 | 31264 | 100.000 | 100.000 |

**RACEMIC 6ad:**



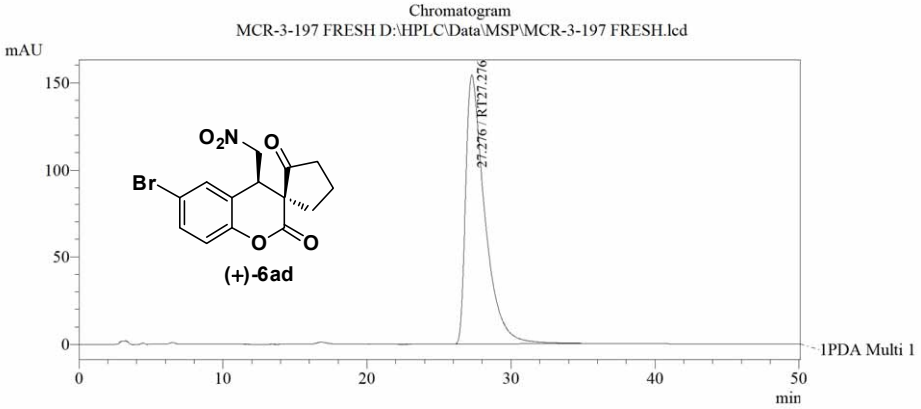

**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT27.459 | 27.459 | 4197176 | 48358 | 50.362 | 57.621 |
| 2 | RT38.376 | 38.376 | 4136819 | 35566 | 49.638 | 42.379 |
| Total | | | 8333995 | 83923 | 100.000 | 100.000 |

**CHIRAL-(+)-6ad (>99.9% *ee*):**



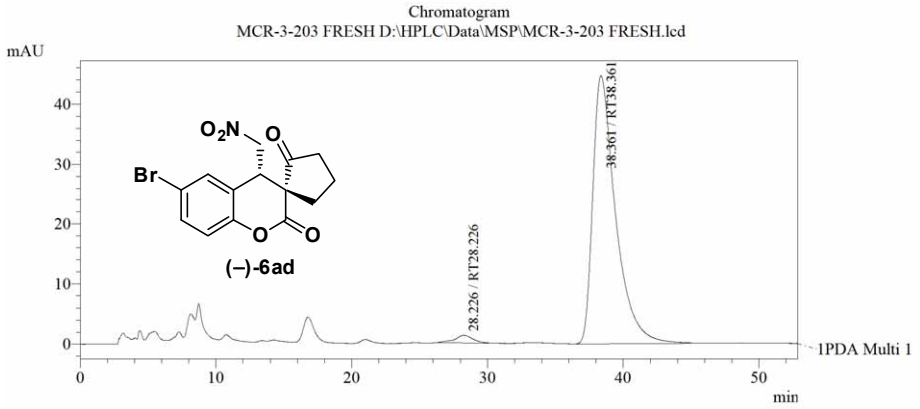

**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT27.276 | 27.276 | 14201683 | 154159 | 100.000 | 100.000 |
| Total | | | 14201683 | 154159 | 100.000 | 100.000 |

## CHIRAL-(–)-6ad (96% *ee*):



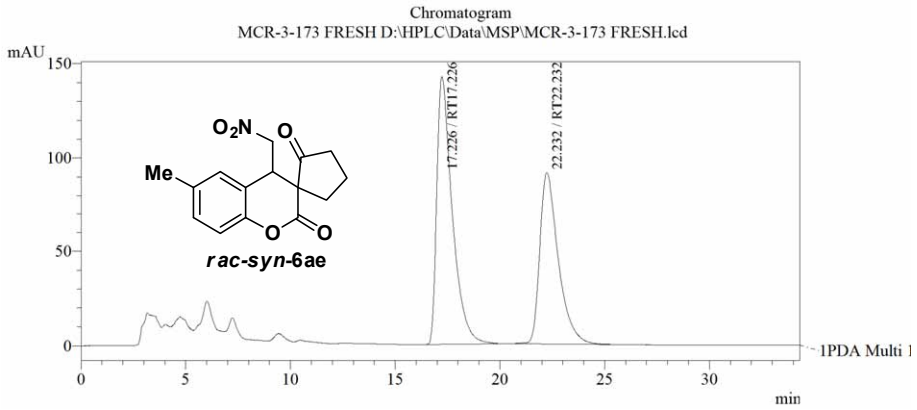

**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT28.226 | 28.226 | 118807 | 1275 | 2.239 | 2.782 |
| 2 | RT38.361 | 38.361 | 5187582 | 44550 | 97.761 | 97.218 |
| Total | | | 5306389 | 45825 | 100.000 | 100.000 |

**RACEMIC 6ae:**



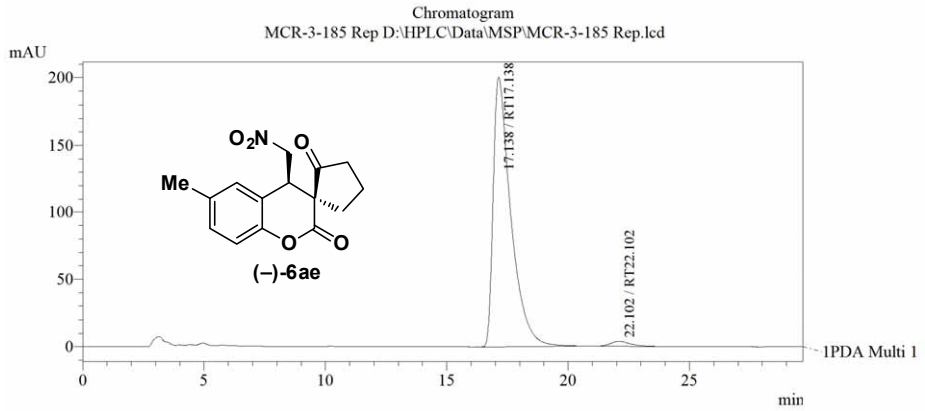

**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT17.226 | 17.226 | 7298464 | 142303 | 56.568 | 60.908 |
| 2 | RT22.232 | 22.232 | 5603728 | 91332 | 43.432 | 39.092 |
| Total | | | 12902192 | 233634 | 100.000 | 100.000 |

**CHIRAL-(–)-6ae (96% *ee*):**



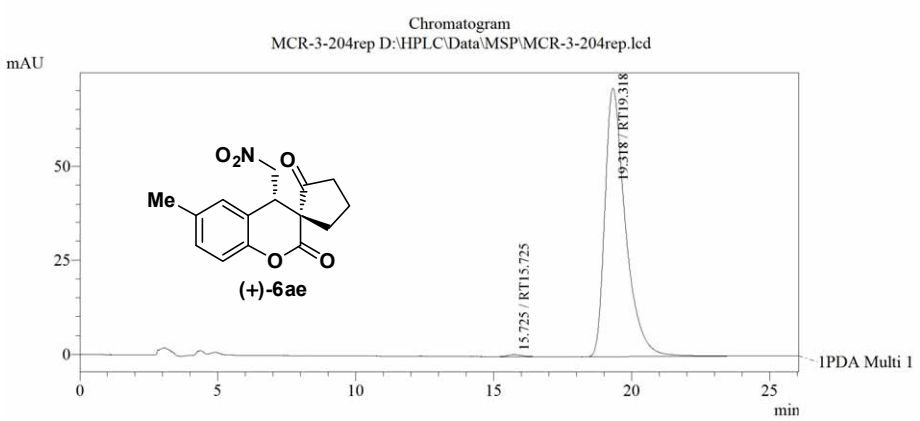

**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT17.138 | 17.138 | 10113386 | 200255 | 98.088 | 98.234 |
| 2 | RT22.102 | 22.102 | 197113 | 3599 | 1.912 | 1.766 |
| Total | | | 10310499 | 203855 | 100.000 | 100.000 |

## CHIRAL-(+)-6ae (99% *ee*):



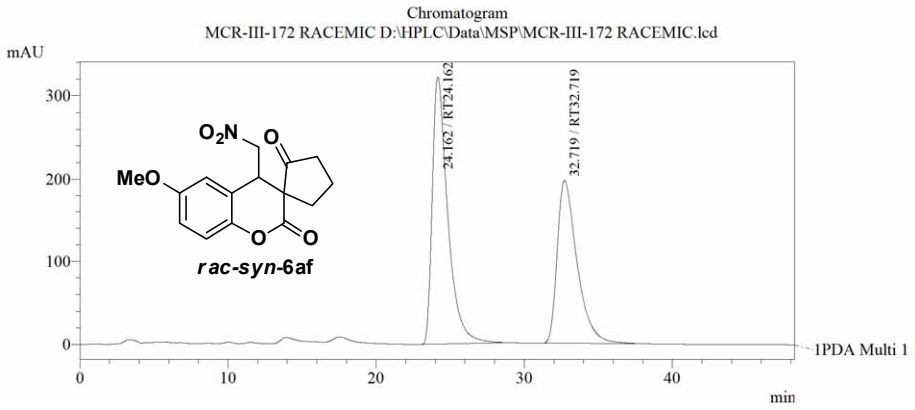

**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT15.725 | 15.725 | 19665 | 541 | 0.527 | 0.753 |
| 2 | RT19.318 | 19.318 | 3712561 | 71311 | 99.473 | 99.247 |
| Total | | | 3732226 | 71852 | 100.000 | 100.000 |

**RACEMIC 6af:**



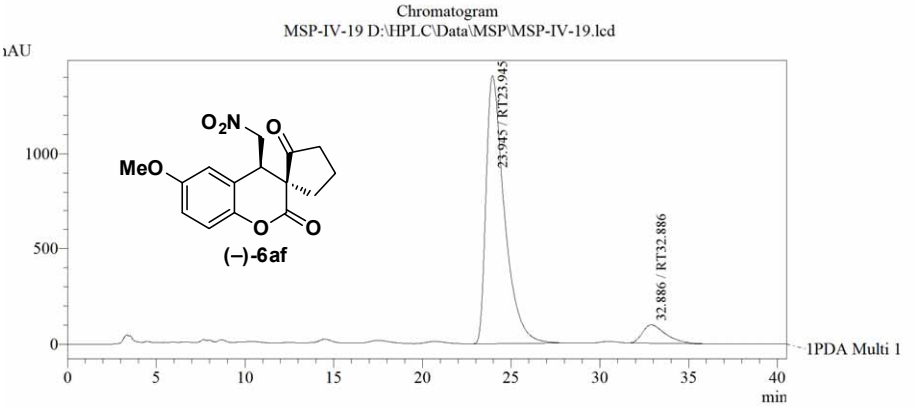

**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT24.162 | 24.162 | 23281427 | 321680 | 55.928 | 61.990 |
| 2 | RT32.719 | 32.719 | 18346355 | 197245 | 44.072 | 38.010 |
| Total | | | 41627782 | 518925 | 100.000 | 100.000 |

**CHIRAL-(–)-6af (84% *ee*):**



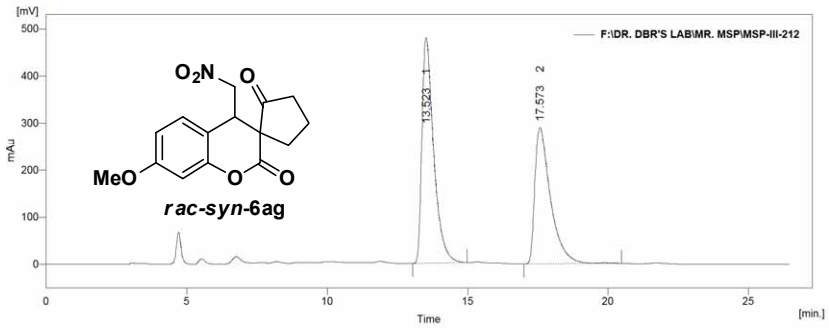

**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT23.945 | 23.945 | 100735105 | 1404811 | 92.060 | 93.602 |
| 2 | RT32.886 | 32.886 | 8688068 | 96018 | 7.940 | 6.398 |
| Total | | | 109423173 | 1500829 | 100.000 | 100.000 |

**RACEMIC 6ag:**

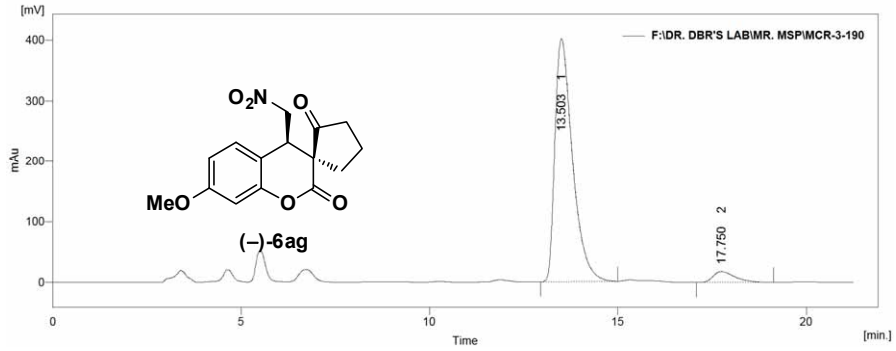



**Daicel Chiralpak AD-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm**

*Result Table (Uncal - F:\DR. DBR'S LAB\MR. MSP\MSP-III-212)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] |
|---|---|---|---|---|---|---|
| 1 | 13.523 | 9251.830 | 319.325 | 55.4 | 62.3 | 0.43 |
| 2 | 17.573 | 7457.533 | 193.278 | 44.6 | 37.7 | 0.57 |
| | Total | 16709.362 | 512.603 | 100.0 | 100.0 | |

**CHIRAL-(–)-6ag (90% *ee*):**

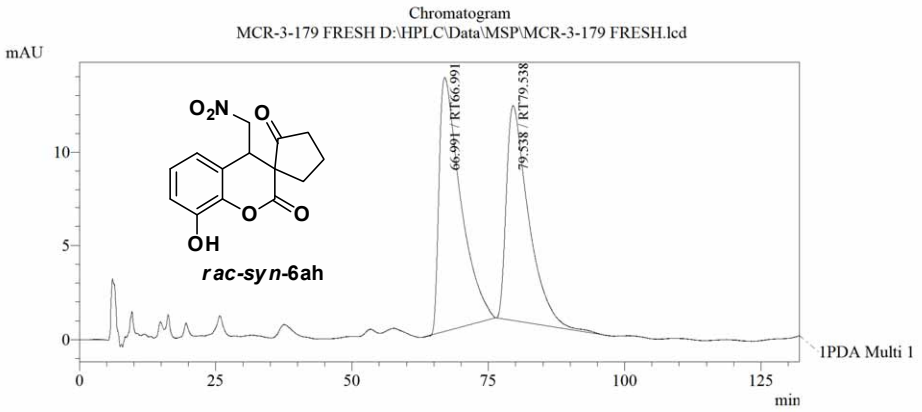



**Daicel Chiralpak AD-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm**

*Result Table (Uncal - F:\DR. DBR'S LAB\MR. MSP\MCR-3-190)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] |
|---|---|---|---|---|---|---|
| 1 | 13.503 | 8645.503 | 266.920 | 94.8 | 95.7 | 0.49 |
| 2 | 17.750 | 477.346 | 12.051 | 5.2 | 4.3 | 0.61 |
| | Total | 9122.850 | 278.971 | 100.0 | 100.0 | |

**RACEMIC 6ah:**



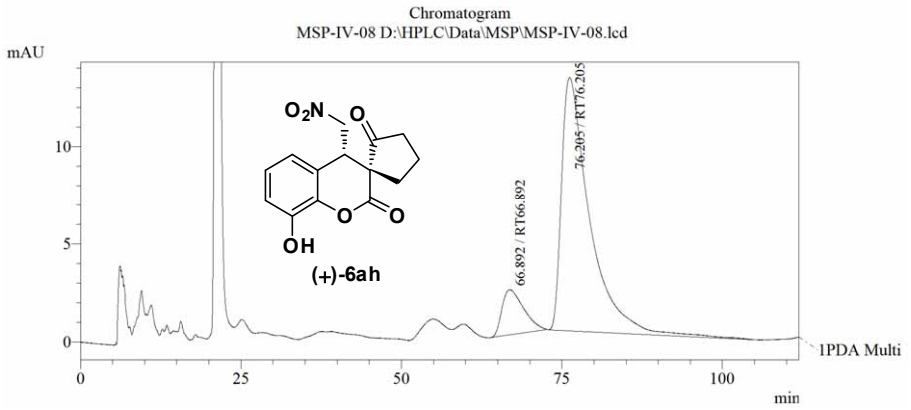

**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 90:10, Flow Rate 0.5 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT66.991 | 66.991 | 3711434 | 13509 | 53.294 | 54.074 |
| 2 | RT79.538 | 79.538 | 3252657 | 11473 | 46.706 | 45.926 |
| Total | | | 6964091 | 24982 | 100.000 | 100.000 |

**CHIRAL-(+)-6ah (76% *ee*):**



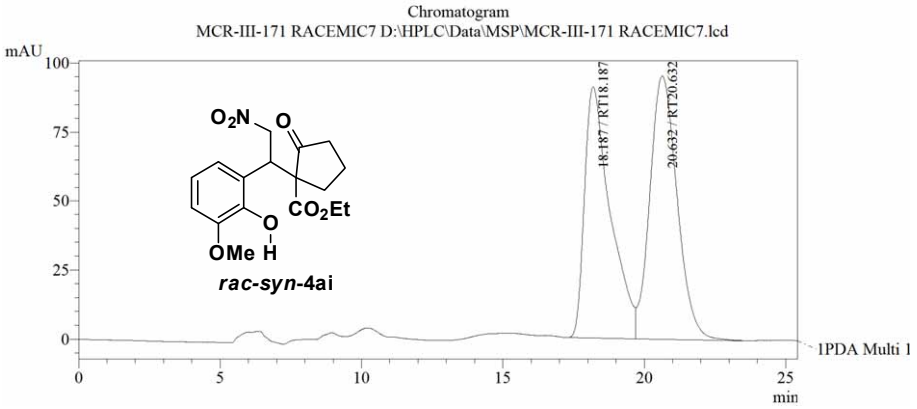

**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 90:10, Flow Rate 0.5 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT66.892 | 66.892 | 539714 | 2301 | 12.065 | 15.078 |
| 2 | RT76.205 | 76.205 | 3933701 | 12961 | 87.935 | 84.922 |
| Total | | | 4473415 | 15263 | 100.000 | 100.000 |

**RACEMIC 4ai:**



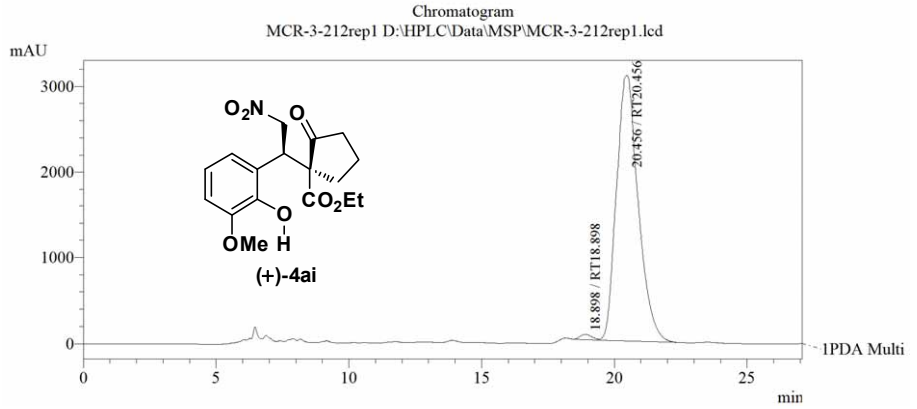

**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 0.5 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT18.187 | 18.187 | 5759669 | 90989 | 47.383 | 48.823 |
| 2 | RT20.632 | 20.632 | 6395860 | 95375 | 52.617 | 51.177 |
| Total | | | 12155529 | 186364 | 100.000 | 100.000 |

**CHIRAL-(+)-4ai (98% *ee*):**



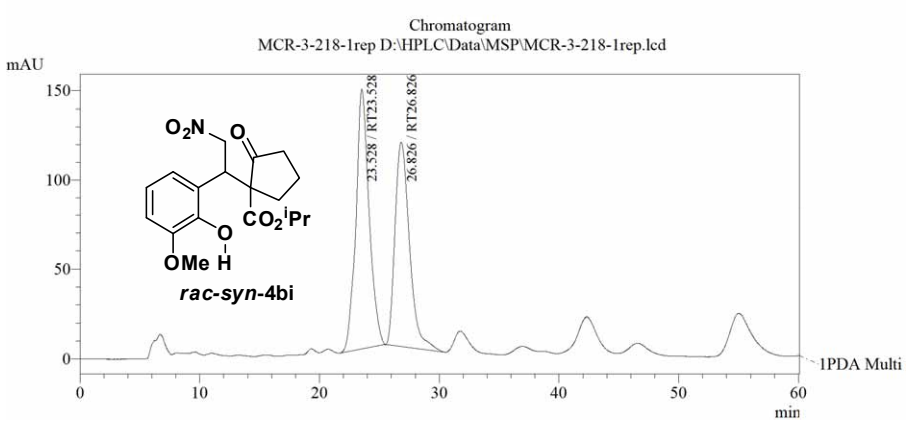

**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 0.5 mL/Min, 254 nm**

PeakTable

PDA Ch1 224nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT18.898 | 18.898 | 1818006 | 59729 | 1.022 | 1.894 |
| 2 | RT20.456 | 20.456 | 176033082 | 3093087 | 98.978 | 98.106 |
| Total | | | 177851088 | 3152816 | 100.000 | 100.000 |

**RACEMIC 4bi:**



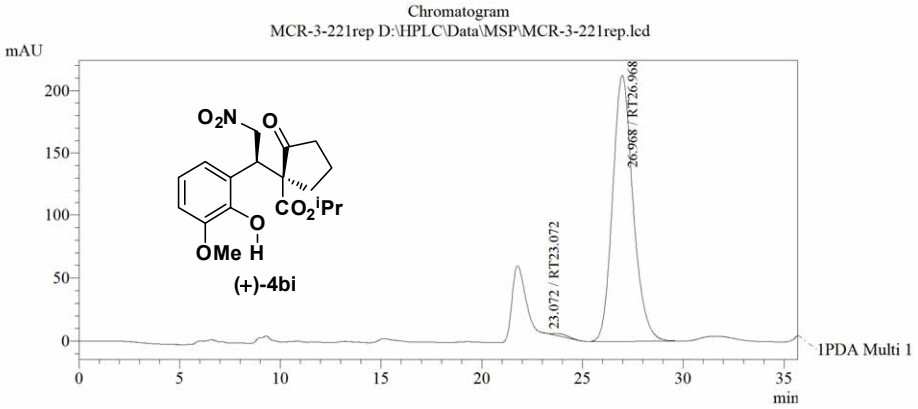

**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 90:10, Flow Rate 0.5 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT23.528 | 23.528 | 11116050 | 145147 | 53.639 | 55.924 |
| 2 | RT26.826 | 26.826 | 9607646 | 114395 | 46.361 | 44.076 |
| Total | | | 20723696 | 259542 | 100.000 | 100.000 |

**CHIRAL-(+)-4bi (99% *ee*):**



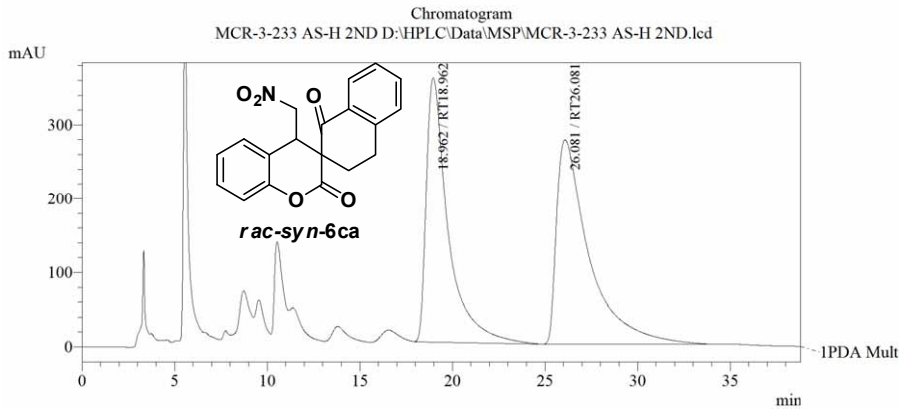

**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 90:10, Flow Rate 0.5 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT23.072 | 23.072 | 95860 | 1 | 0.637 | 0.000 |
| 2 | RT26.968 | 26.968 | 14958565 | 212148 | 99.363 | 100.000 |
| Total | | | 15054426 | 212149 | 100.000 | 100.000 |

**RACEMIC 6ca:**



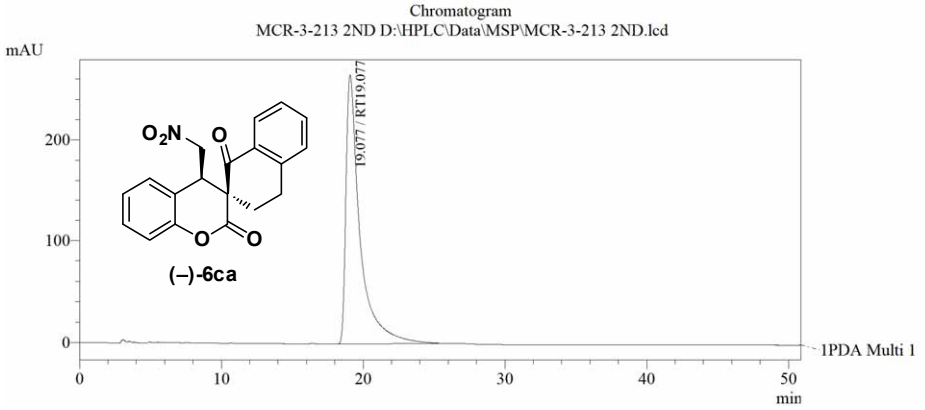

**Daicel Chiralpak AS-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT18.962 | 18.962 | 29179160 | 356817 | 46.417 | 56.361 |
| 2 | RT26.081 | 26.081 | 33683489 | 276270 | 53.583 | 43.639 |
| Total | | | 62862649 | 633087 | 100.000 | 100.000 |

**CHIRAL-(–)-6ca (>99.9% *ee*):**



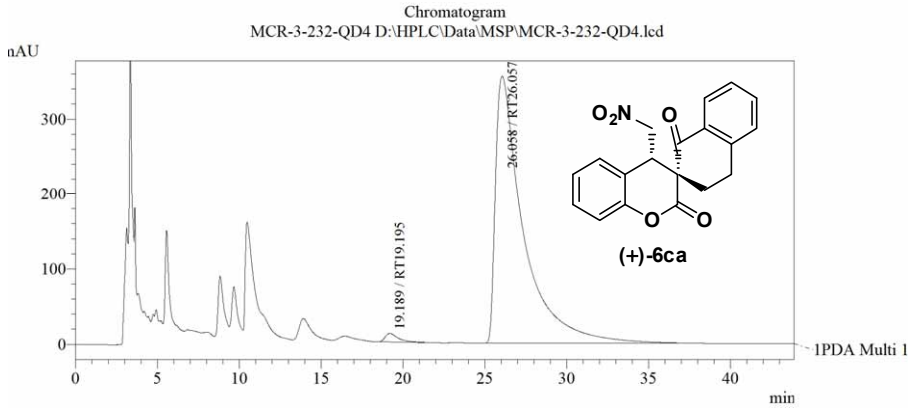

**Daicel Chiralpak AS-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT19.077 | 19.077 | 18307764 | 265383 | 100.000 | 100.000 |
| Total | | | 18307764 | 265383 | 100.000 | 100.000 |

## CHIRAL-(+)-6ca (97% *ee*):



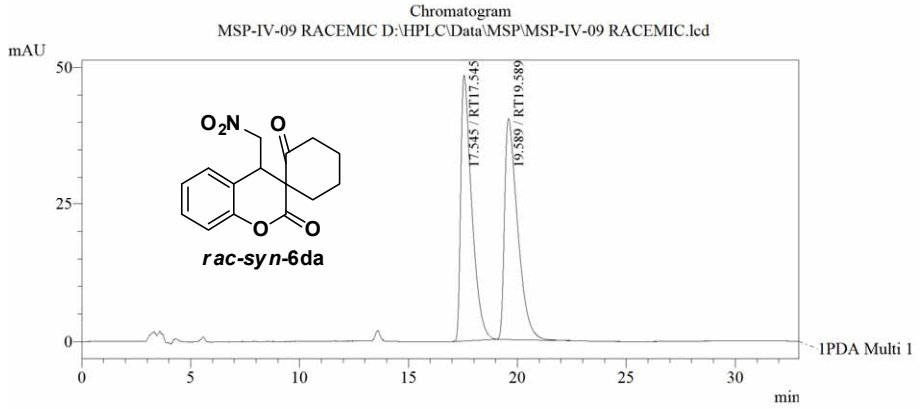

**Daicel Chiralpak AS-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm**

PeakTable

PDA Ch1 245nm 2nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT19.195 | 19.189 | 655697 | 11525 | 1.537 | 3.138 |
| 2 | RT26.057 | 26.058 | 42005311 | 355692 | 98.463 | 96.862 |
| Total | | | 42661008 | 367217 | 100.000 | 100.000 |

**RACEMIC 6da:**



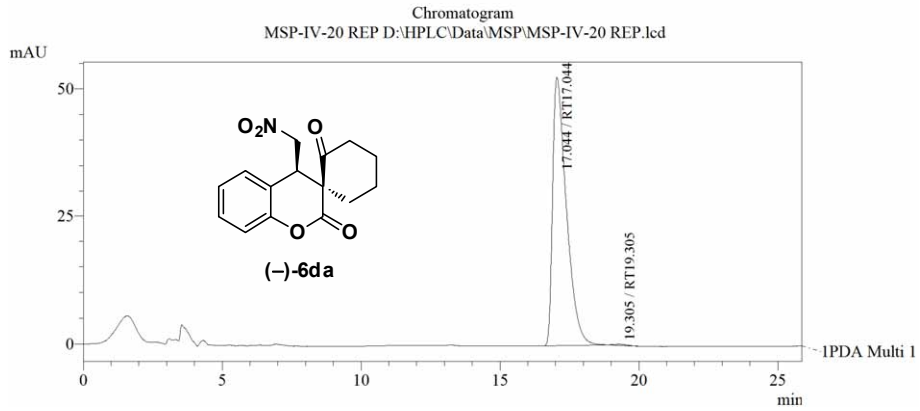

**Daicel Chiralcel OJ-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT17.545 | 17.545 | 1711125 | 48486 | 50.480 | 54.573 |
| 2 | RT19.589 | 19.589 | 1678561 | 40359 | 49.520 | 45.427 |
| Total | | | 3389686 | 88845 | 100.000 | 100.000 |

**CHIRAL-(–)-6da (99% *ee*):**



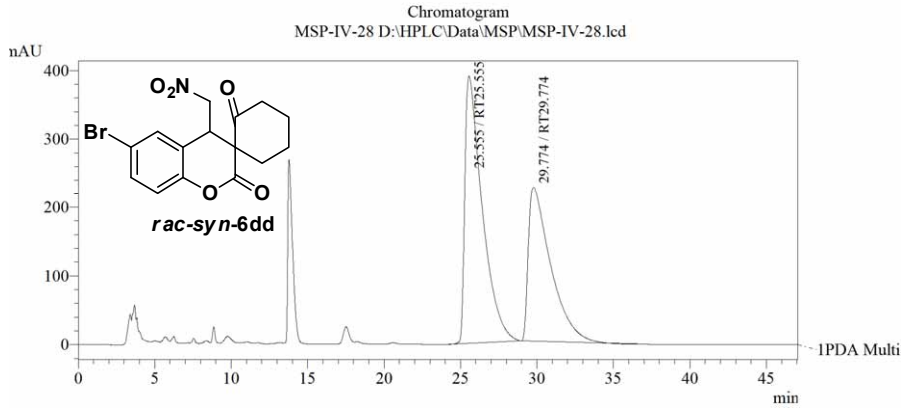

**Daicel Chiralcel OJ-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT17.044 | 17.044 | 1810745 | 52649 | 99.649 | 99.599 |
| 2 | RT19.305 | 19.305 | 6377 | 212 | 0.351 | 0.401 |
| Total | | | 1817123 | 52861 | 100.000 | 100.000 |

**RACEMIC 6dd:**



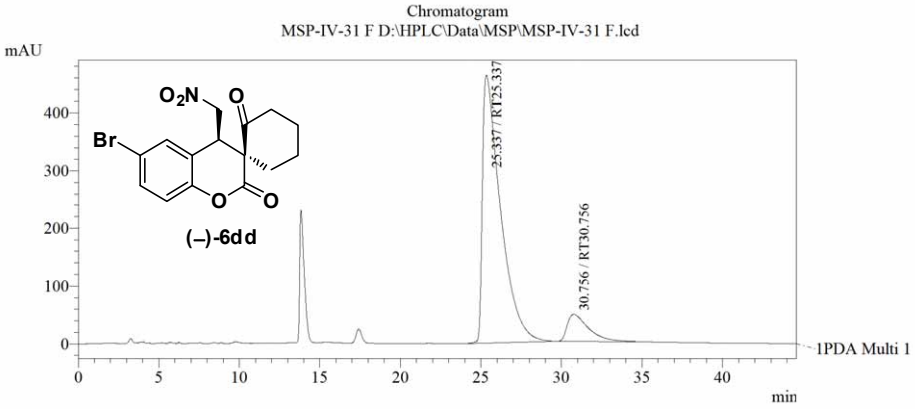

**Daicel Chiralcel OJ-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 240 nm**

PeakTable

PDA Ch1 240nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT25.555 | 25.555 | 30747712 | 389943 | 57.391 | 63.432 |
| 2 | RT29.774 | 29.774 | 22828368 | 224802 | 42.609 | 36.568 |
| Total | | | 53576080 | 614745 | 100.000 | 100.000 |

**CHIRAL-(–)-6dd (79% *ee*):**



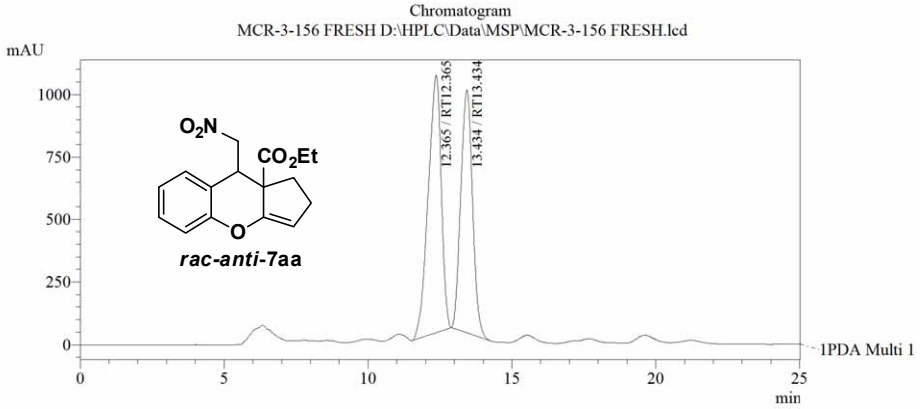

**Daicel Chiralcel OJ-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 240 nm**

PeakTable

PDA Ch1 240nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT25.337 | 25.337 | 36240663 | 463021 | 89.327 | 90.820 |
| 2 | RT30.756 | 30.756 | 4330297 | 46804 | 10.673 | 9.180 |
| Total | | | 40570961 | 509825 | 100.000 | 100.000 |

**RACEMIC 7aa:**



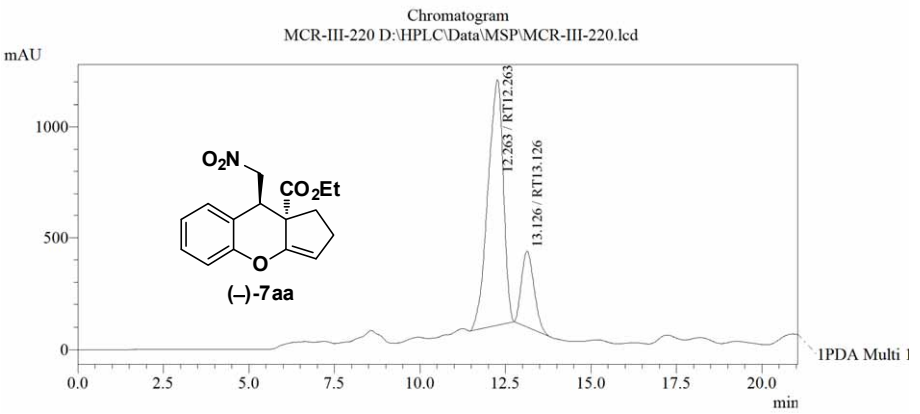

**Daicel Chiralpak AD-H, Hexane/ *i*-PrOH = 90:10, Flow Rate 0.5 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT12.365 | 12.365 | 32450863 | 1029173 | 53.993 | 51.388 |
| 2 | RT13.434 | 13.434 | 27650596 | 973569 | 46.007 | 48.612 |
| Total | | | 60101458 | 2002742 | 100.000 | 100.000 |

**CHIRAL-(–)-7aa (60% *ee*):**



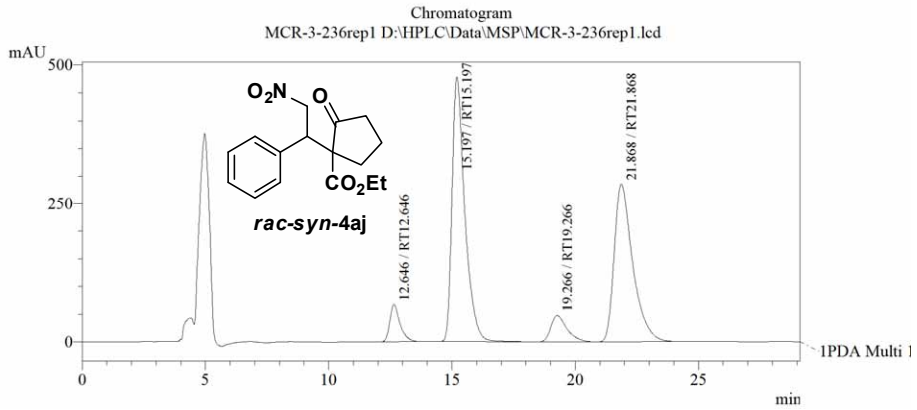

**Daicel Chiralpak AD-H, Hexane/ *i*-PrOH = 90:10, Flow Rate 0.5 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT12.263 | 12.263 | 34409809 | 1099945 | 79.912 | 76.473 |
| 2 | RT13.126 | 13.126 | 8649680 | 338399 | 20.088 | 23.527 |
| Total | | | 43059488 | 1438345 | 100.000 | 100.000 |

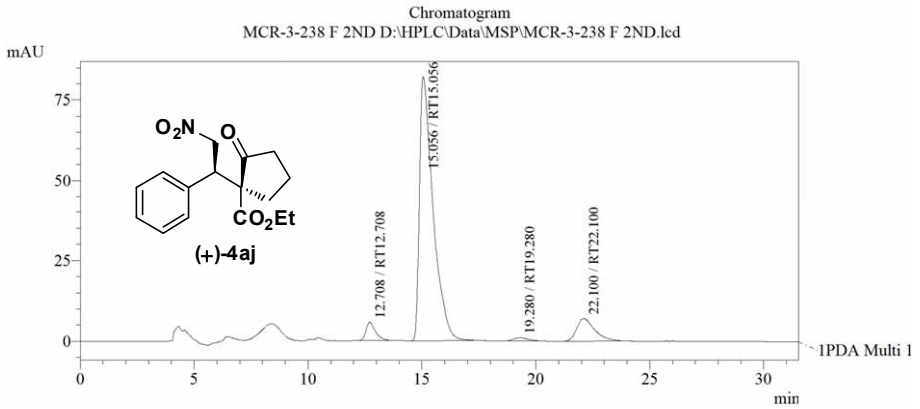
## RACEMIC 4aj:




**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 90:10, Flow Rate 0.7 mL/Min, 254 nm**

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT12.646 | 12.646 | 1949102 | 67023 | 5.380 | 7.634 |
| 2 | RT15.197 | 15.197 | 17148075 | 477420 | 47.334 | 54.381 |
| 3 | RT19.266 | 19.266 | 2182546 | 47920 | 6.025 | 5.458 |
| 4 | RT21.868 | 21.868 | 14948035 | 285547 | 41.261 | 32.526 |
| Total | | | 36227758 | 877910 | 100.000 | 100.000 |

## CHIRAL-(+)-4aj (for major isomer: 79% *ee*):



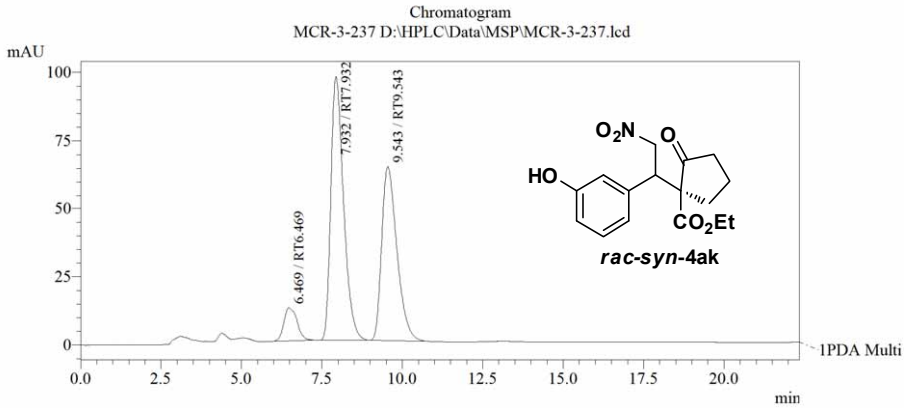

**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 90:10, Flow Rate 0.7 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT12.708 | 12.708 | 162742 | 5647 | 4.098 | 5.902 |
| 2 | RT15.056 | 15.056 | 3383508 | 82052 | 85.202 | 85.749 |
| 3 | RT19.280 | 19.280 | 42615 | 998 | 1.073 | 1.043 |
| 4 | RT22.100 | 22.100 | 382287 | 6992 | 9.627 | 7.307 |
| Total | | | 3971153 | 95689 | 100.000 | 100.000 |

## RACEMIC 4ak:



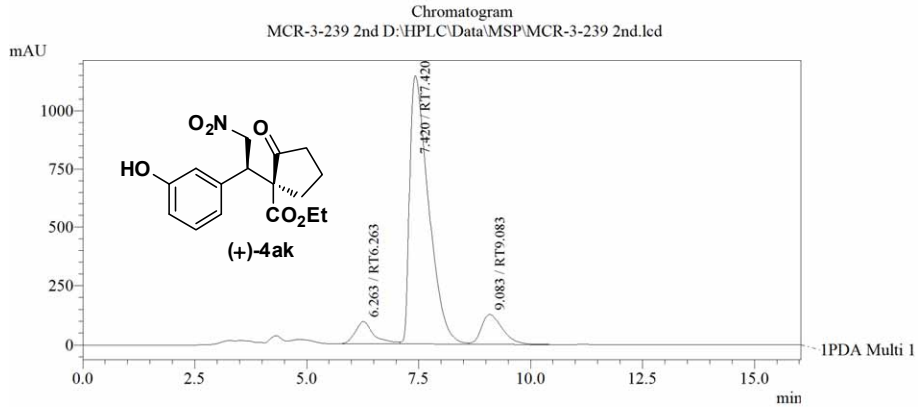

**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH =80:20, Flow Rate 1.0 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT6.469 | 6.469 | 342707 | 12091 | 6.716 | 7.002 |
| 2 | RT7.932 | 7.932 | 2654419 | 96594 | 52.020 | 55.935 |
| 3 | RT9.543 | 9.543 | 2105603 | 64005 | 41.264 | 37.064 |
| Total | | | 5102729 | 172690 | 100.000 | 100.000 |

## CHIRAL-(+)-4ak (for major isomer: 78% *ee*):




**Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm**

PeakTable

PDA Ch1 254nm 4nm

| Peak# | Name | Ret. Time | Area | Height | Area % | Height % |
|---|---|---|---|---|---|---|
| 1 | RT6.263 | 6.263 | 2732990 | 95423 | 6.751 | 6.977 |
| 2 | RT7.420 | 7.420 | 33606820 | 1145052 | 83.011 | 83.719 |
| 3 | RT9.083 | 9.083 | 4144923 | 127258 | 10.238 | 9.304 |
| Total | | | 40484734 | 1367734 | 100.000 | 100.000 |