

Supporting Information-II

A general approach to high-yielding asymmetric synthesis of chiral 3-alkyl-4-nitromethylchromans *via* cascade Barbas- Michael and acetalization reactions

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R. Madhavachary

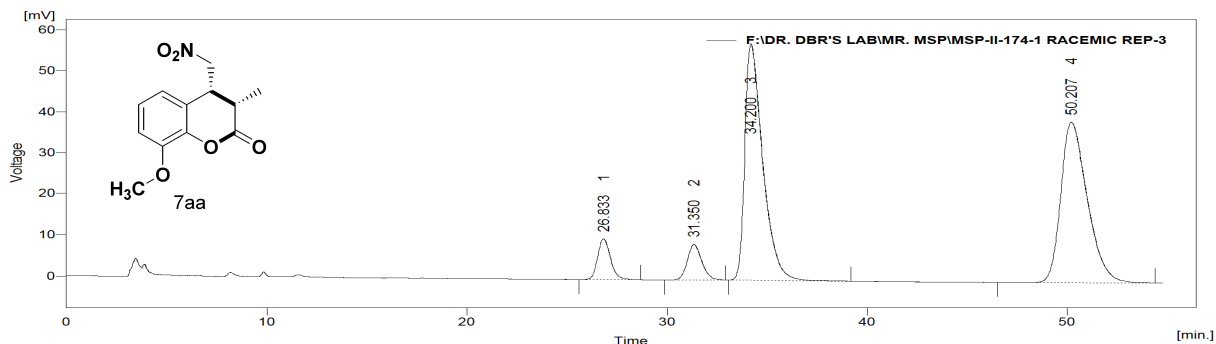
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General Methods: The ^1H NMR and ^{13}C NMR spectra were recorded at 400 MHz and 100 MHz, respectively. The chemical shifts are reported in ppm downfield to TMS ($\delta = 0$) for ^1H NMR and relative to the central CDCl_3 resonance ($\delta = 77.0$) 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- $\text{K}\alpha$ ($\lambda = 0.71073 \text{ \AA}$) 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- $\text{K}\alpha$ fine-focus sealed tube ($\lambda = 0.71073 \text{ \AA}$). 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. H₂SO₄ (35 mL), acetic acid (10 mL), and ethanol (900 mL) followed by heating.

The enantiomeric excess (*ee*) of the *BMA* products was determined by chiral stationary phase HPLC using a Daicel Chiralcel OD-H, Chiralcel OJ-H, Chiralpak AD-H, Chiralpak AS-H or Lux 5u Amylose-2 columns and hexane/2-propanol as the eluent. Retention times and solvent ratios are indicated in the respective entries.

RACEMIC 7aa:

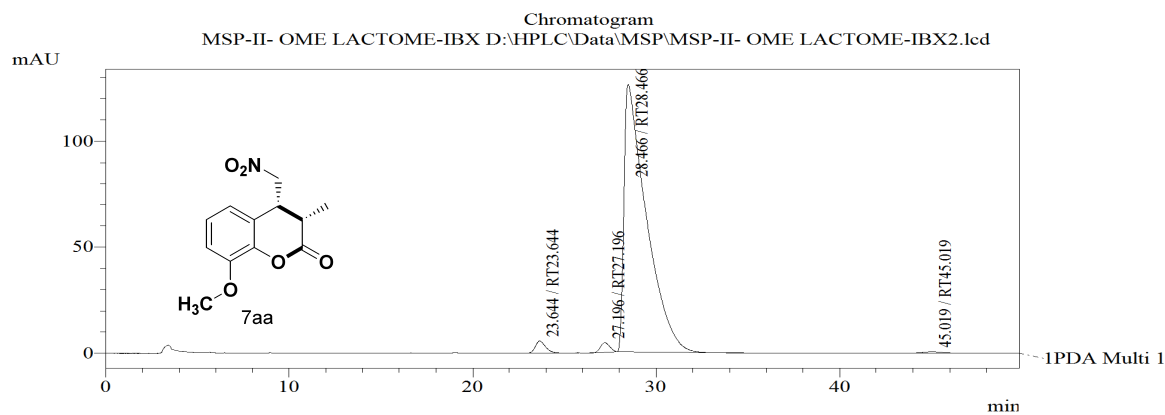


Daicel Chiralcel OJ-H, Hexane/ i-PrOH = 60:40, Flow Rate 1.0 mL/Min, 254 nm

Result Table (Uncal - F:\DR. DBR'S LAB\MR. MSP\MSP-II-174-1 RACEMIC REP-3)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	26.833	290.425	6.572	5.4	8.6	0.67
2	31.350	288.311	5.760	5.4	7.5	0.77
3	34.200	2377.838	38.388	44.5	50.0	0.92
4	50.207	2385.432	26.040	44.7	33.9	1.40
Total		5342.005	76.760	100.0	100.0	

CHIRAL 7aa (99% ee):

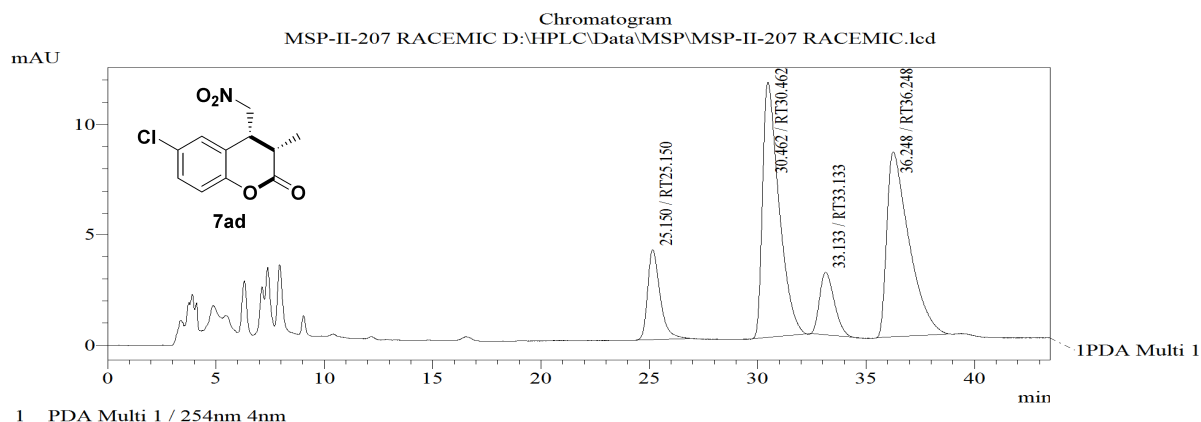


Daicel Chiralcel OJ-H, Hexane/ i-PrOH = 60:40, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT23.644	23.644	213663	5626	1.942	4.115
2	RT27.196	27.196	159136	4515	1.446	3.303
3	RT28.466	28.466	10582237	125950	96.162	92.123
4	RT45.019	45.019	49613	627	0.451	0.459
Total			11004650	136719	100.000	100.000

RACEMIC 7ad:

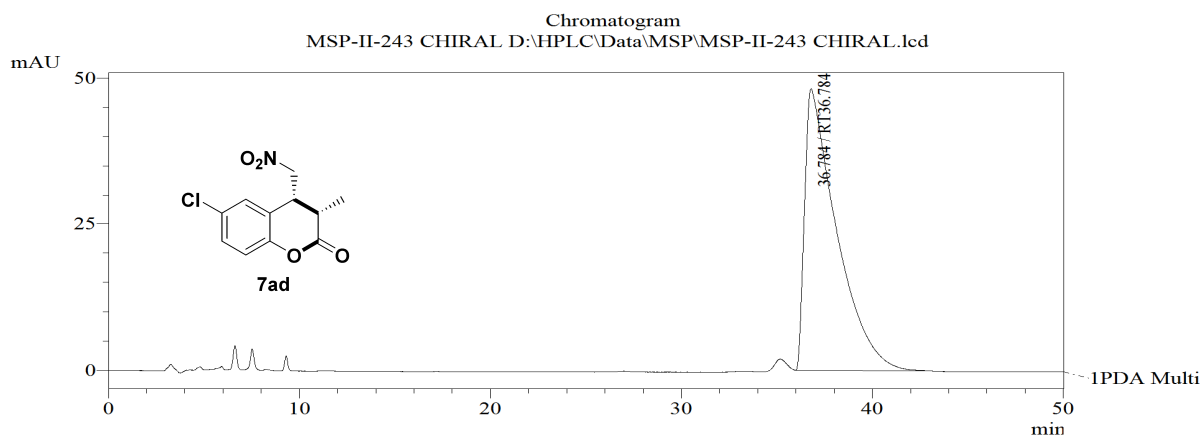


Daicel Chiralcel OJ-H, Hexane/ i-PrOH =75:25, Flow Rate 1.0 mL/Min, 254 nm

Peak table

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT25.150	25.150	163844	4076	10.879	15.188
2	RT30.462	30.462	609254	11537	40.454	42.994
3	RT33.133	33.133	133390	2837	8.857	10.574
4	RT36.248	36.248	599550	8384	39.810	31.245
Total			1506038	26834	100.000	100.000

CHIRAL 7ad (>99% ee):

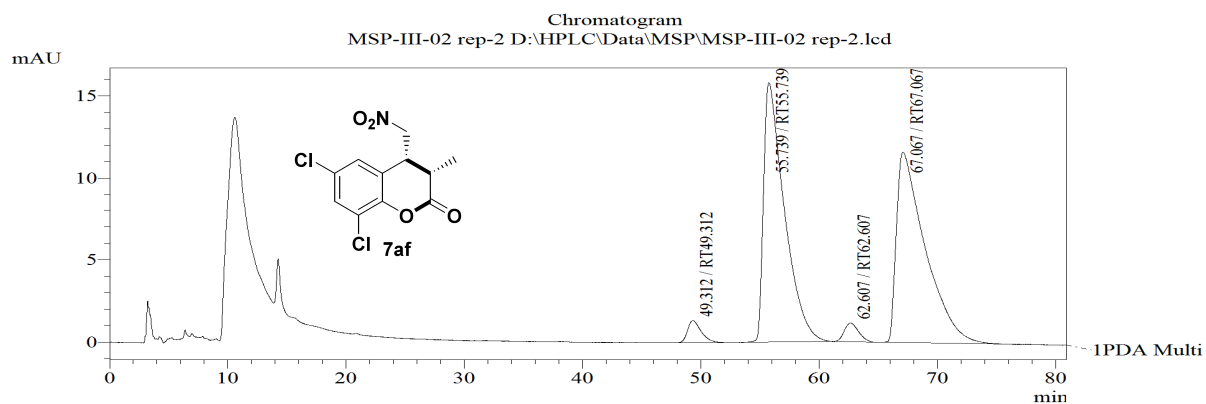


Daicel Chiralcel OJ-H, Hexane/ i-PrOH = 75:25, Flow Rate 1.0 mL/Min, 254 nm

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT36.784	36.784	5589141	48202	100.000	100.000
Total			5589141	48202	100.000	100.000

RACEMIC 7af:



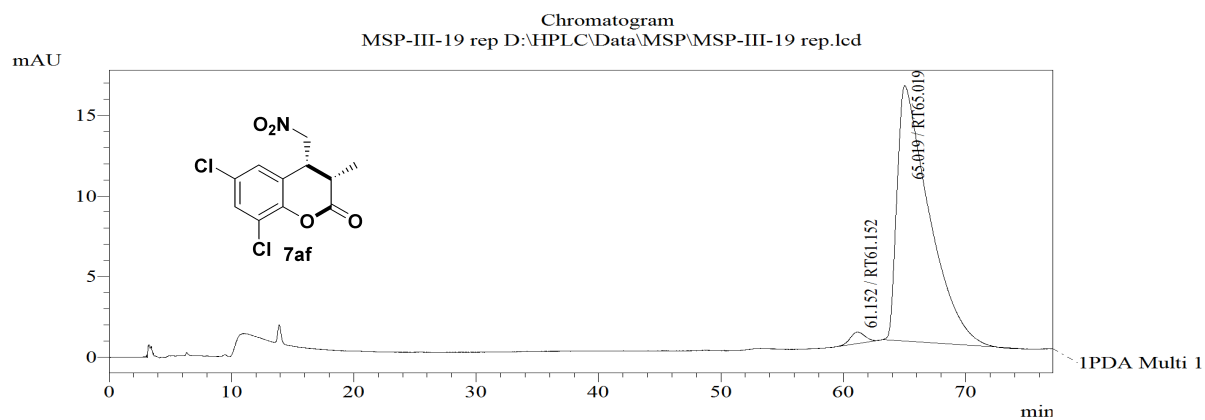
Daicel Chiralcel OJ-H, Hexane/ i- PrOH = 90:10, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT49.312	49.312	107600	1343	2.628	4.489
2	RT55.739	55.739	1933305	15801	47.211	52.823
3	RT62.607	62.607	96700	1157	2.361	3.866
4	RT67.067	67.067	1957429	11613	47.800	38.822
Total			4095033	29913	100.000	100.000

CHIRAL 7af (>99% ee):



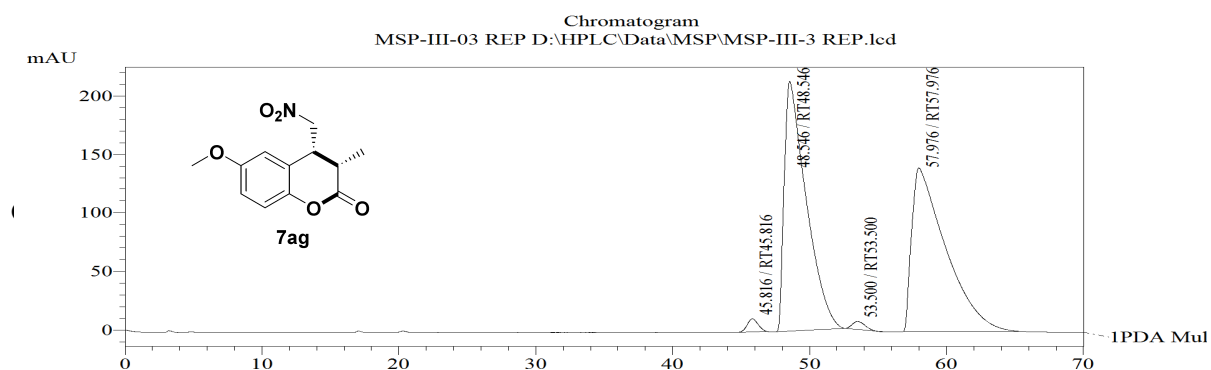
Daicel Chiralcel OJ-H, Hexane/ i- PrOH = 90:10, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT61.152	61.152	57124	714	1.954	4.319
2	RT65.019	65.019	2866434	15810	98.046	95.681
Total			2923558	16524	100.000	100.000

RACEMIC 7ag:



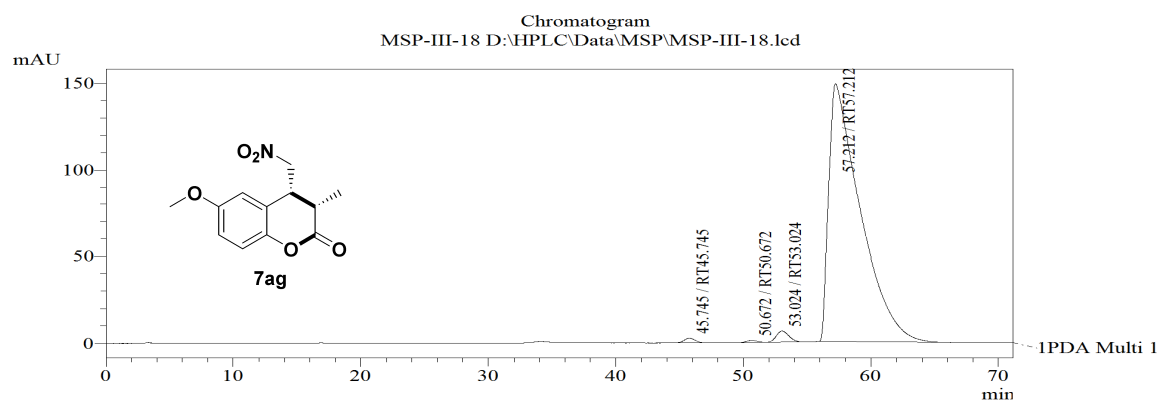
Daicel Chiralcel OJ-H, Hexane/ i- PrOH = 85:15, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT45.816	45.816	598484	11016	1.225	2.972
2	RT48.546	48.546	23759573	213146	48.637	57.494
3	RT53.500	53.500	436575	6798	0.894	1.834
4	RT57.976	57.976	24056143	139765	49.244	37.700
Total			48850775	370726	100.000	100.000

CHIRAL 7ag (99.7% ee):



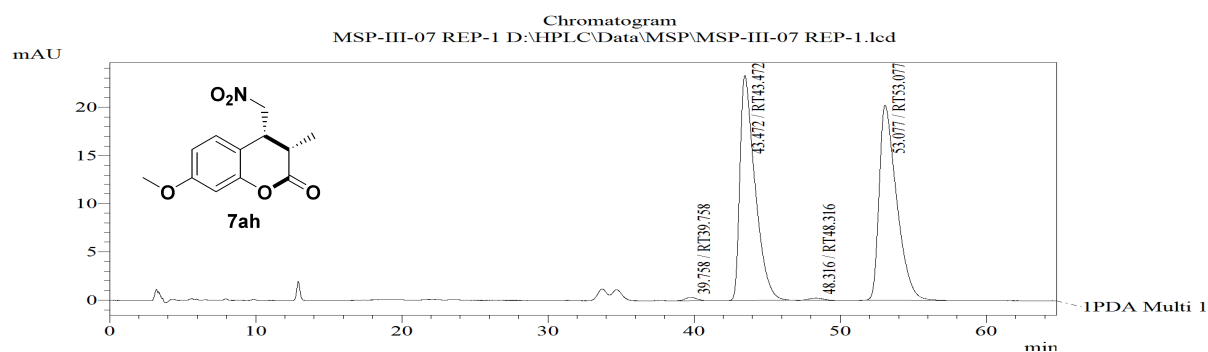
Daicel Chiralcel OJ-H, Hexane/ i- PrOH = 85:15, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT45.745	45.745	150552	2613	0.578	1.645
2	RT50.672	50.672	64342	1134	0.247	0.714
3	RT53.024	53.024	429868	6357	1.649	4.003
4	RT57.212	57.212	25424484	148721	97.527	93.638
Total			26069246	158824	100.000	100.000

RACEMIC 7ah:



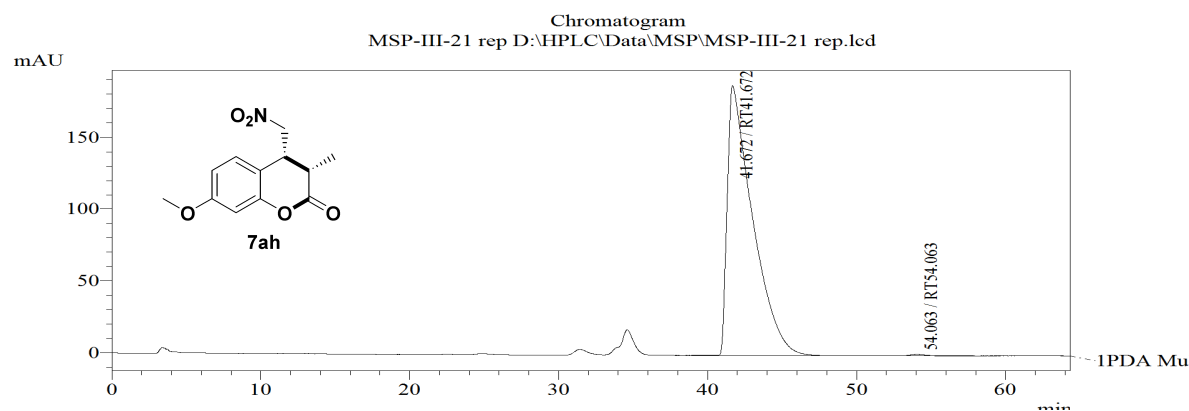
Daicel Chiralcel OJ-H, Hexane/ i-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm

Peak Table

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT39.758	39.758	18010	357	0.531	0.809
2	RT43.472	43.472	1670252	23285	49.267	52.766
3	RT48.316	48.316	16073	251	0.474	0.570
4	RT53.077	53.077	1685885	20235	49.728	45.855
Total			3390220	44129	100.000	100.000

CHIRAL 7ah (99% ee):



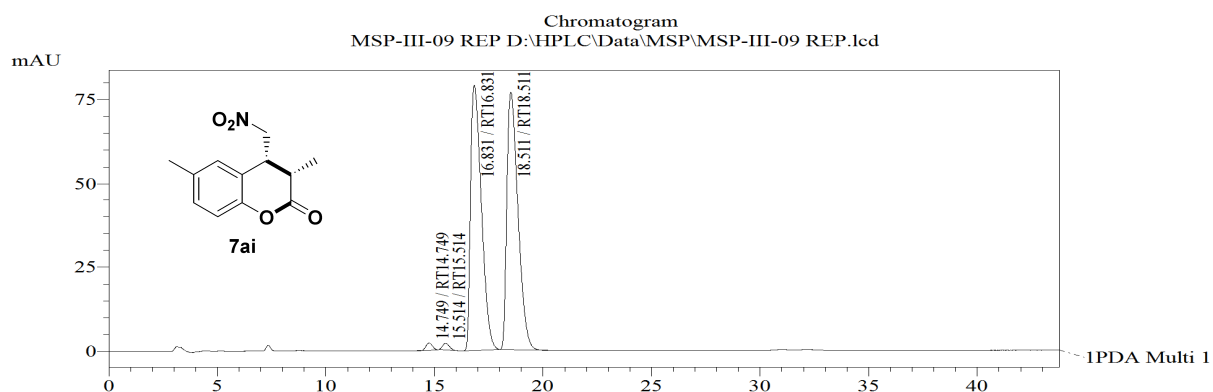
Daicel Chiralcel OJ-H, Hexane/ i-PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm

Peak Table

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT41.672	41.672	22151158	187523	99.620	99.467
2	RT54.063	54.063	84454	1005	0.380	0.533
Total			22235612	188528	100.000	100.000

RACEMIC 7ai:

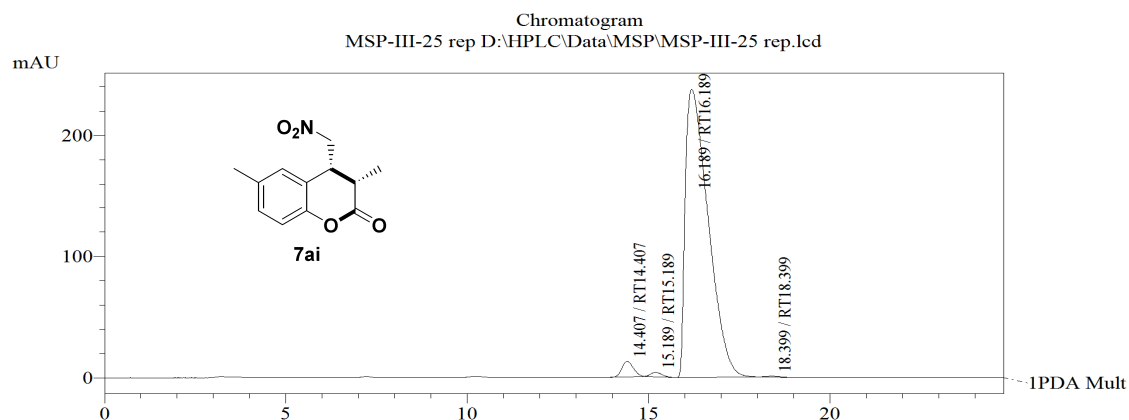


Daicel Chiralpak AS-H, Hexane/ i- PrOH = 85:15, Flow Rate 1.0 mL/Min, 254 nm

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT14.749	14.749	42568	2127	0.766	1.332
2	RT15.514	15.514	43780	2023	0.788	1.267
3	RT16.831	16.831	2735836	78910	49.214	49.402
4	RT18.511	18.511	2736860	76670	49.233	48.000
Total			5559044	159731	100.000	100.000

CHIRAL 7ai (99.6% ee):

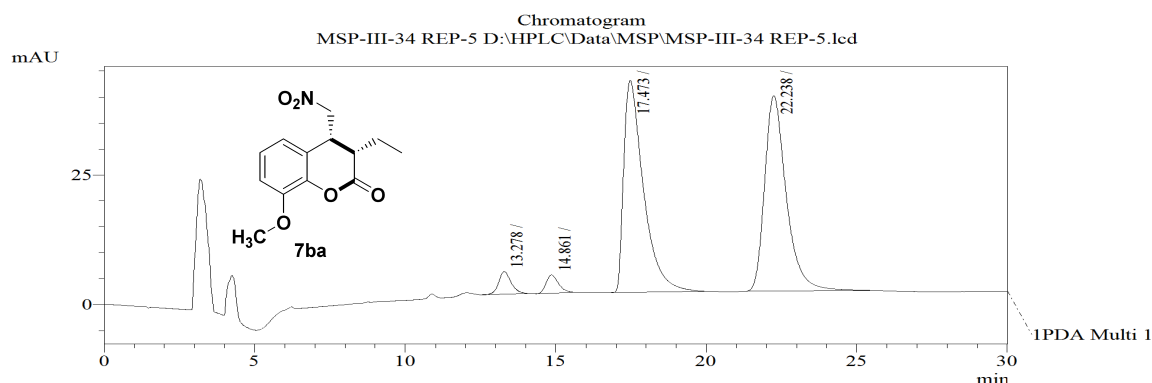


Daicel Chiralpak AS-H, Hexane/ i- PrOH = 85:15, Flow Rate 1.0 mL/Min, 254 nm

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT14.407	14.407	275116	12666	2.546	4.978
2	RT15.189	15.189	72384	3498	0.670	1.375
3	RT16.189	16.189	10439795	237545	96.628	93.363
4	RT18.399	18.399	16851	722	0.156	0.284
Total			10804145	254430	100.000	100.000

RACEMIC 7ba:

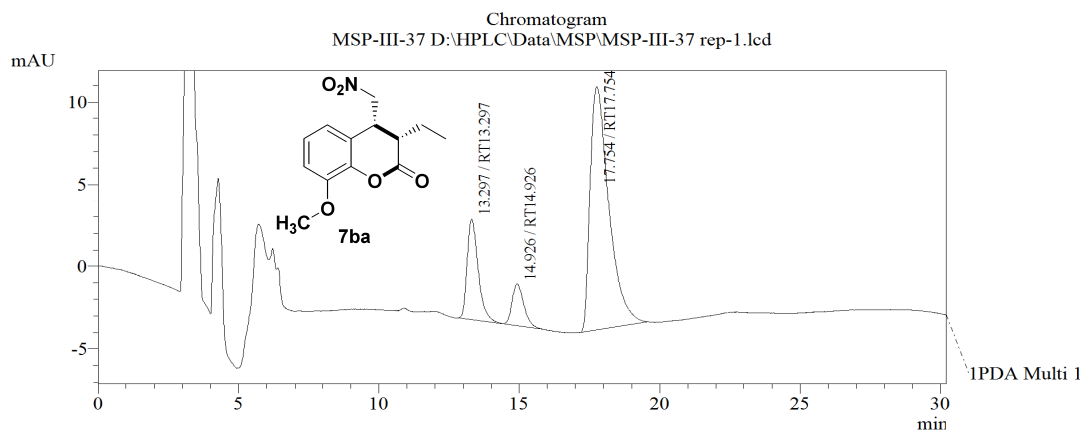


Lux 5u Amylose-2, Hexane/ i- PrOH = 75:25, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1		13.278	125351	4351	3.150	5.045
2		14.861	100186	3508	2.517	4.067
3		17.473	1868789	40748	46.959	47.248
4		22.238	1885324	37637	47.374	43.641
Total			3979650	86243	100.000	100.000

CHIRAL 7ba (>99.9% ee):

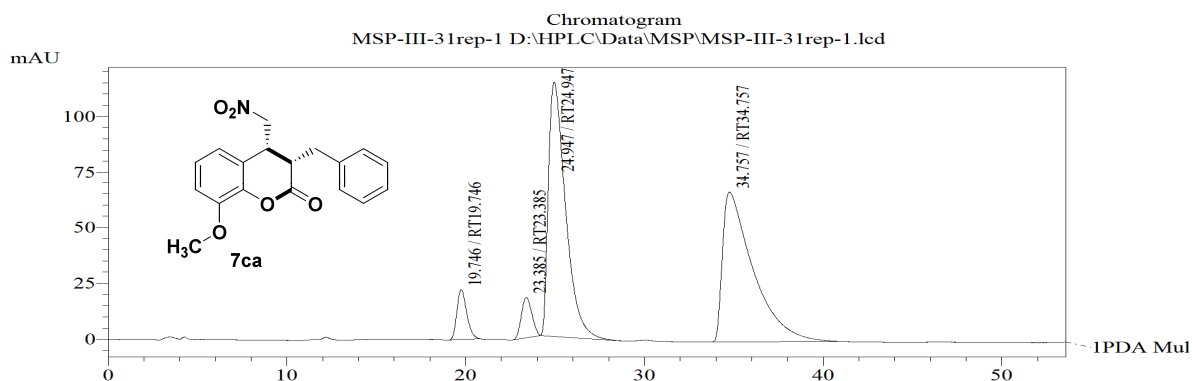


Lux 5u Amylose-2, Hexane/ i- PrOH = 75:25, Flow Rate 1.0 mL/Min, 254

PeakTable

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT13.297	13.297	164860	6060	17.845	25.968
2	RT14.926	14.926	72327	2511	7.829	10.760
3	RT17.754	17.754	686653	14766	74.326	63.272
Total			923840	23338	100.000	100.000

RACEMIC 7ca:

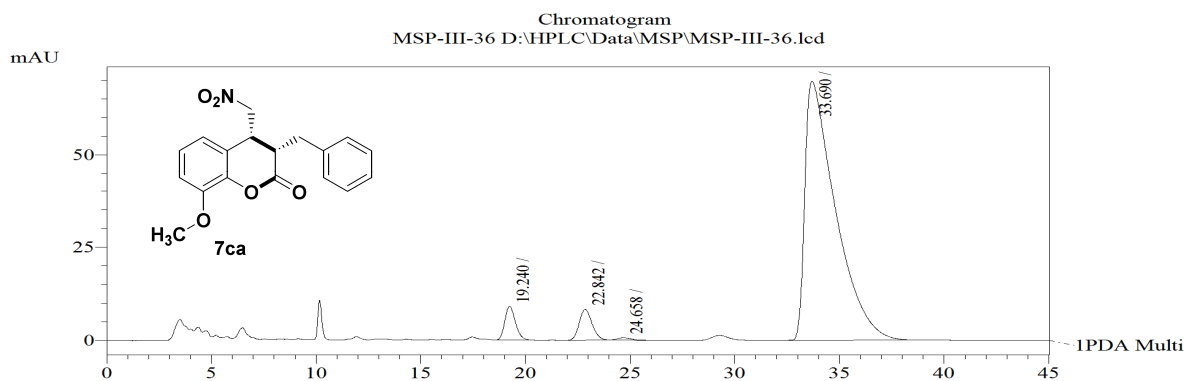


Daicel Chiralpak AS-H, Hexane/ i- PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT19.746	19.746	855164	22312	4.987	10.089
2	RT23.385	23.385	732489	17999	4.272	8.139
3	RT24.947	24.947	7589735	113938	44.263	51.520
4	RT34.757	34.757	7969496	66904	46.478	30.252
Total			17146884	221153	100.000	100.000

CHIRAL 7ca (99.3% ee):

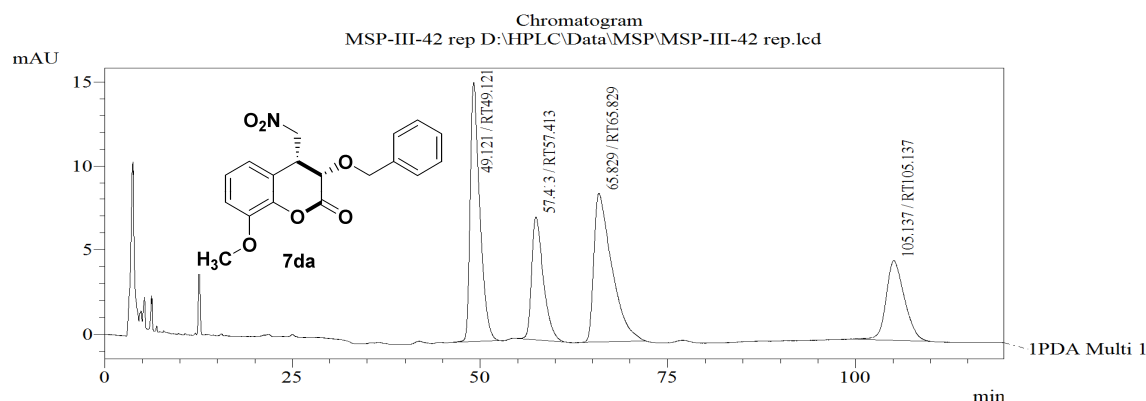


Daicel Chiralpak AS-H, Hexane/ i- PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1		19.240	317246	9037	4.097	10.327
2		22.842	349582	8247	4.514	9.424
3		24.658	26459	599	0.342	0.684
4		33.690	7050735	69625	91.047	79.564
Total			7744021	87508	100.000	100.000

RACEMIC 7da:

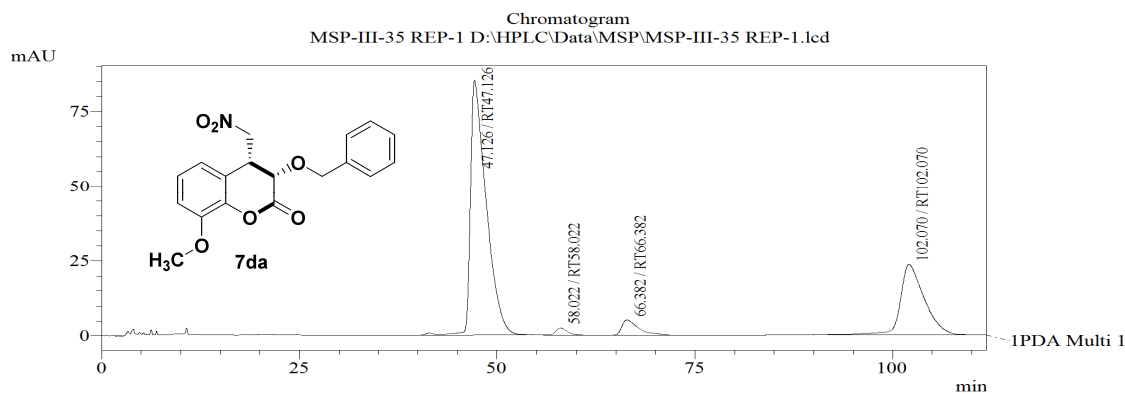


Daicel Chiralcel OJ-H, Hexane/ i- PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT49.121	49.121	1389850	15384	31.342	42.593
2	RT57.413	57.413	787553	7261	17.760	20.102
3	RT65.829	65.829	1449309	8759	32.683	24.250
4	RT105.137	105.137	807697	4716	18.214	13.056
Total			4434408	36119	100.000	100.000

CHIRAL 7da (87% ee):

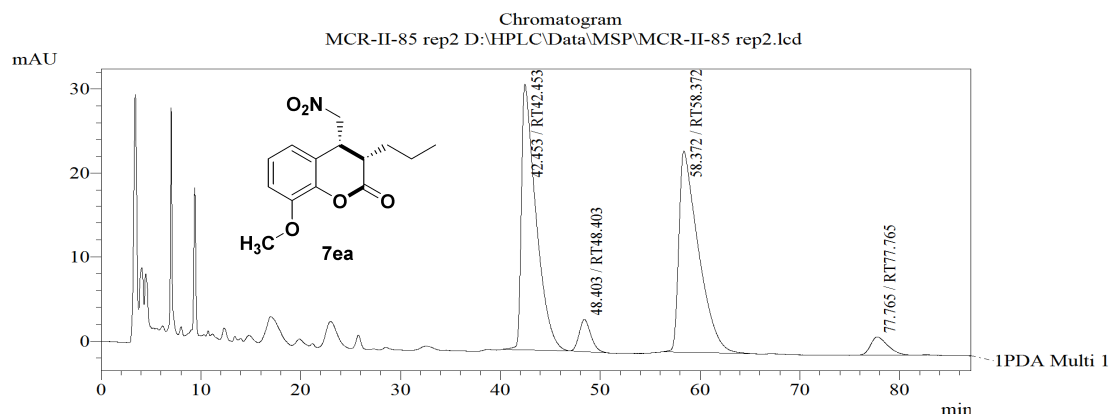


Daicel Chiralcel OJ-H, Hexane/ i- PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT47.126	47.126	11728860	85039	66.404	73.342
2	RT58.022	58.022	243370	2387	1.378	2.058
3	RT66.382	66.382	792703	5137	4.488	4.431
4	RT102.070	102.070	4897965	23385	27.730	20.168
Total			17662898	115948	100.000	100.000

RACEMIC 7ea:



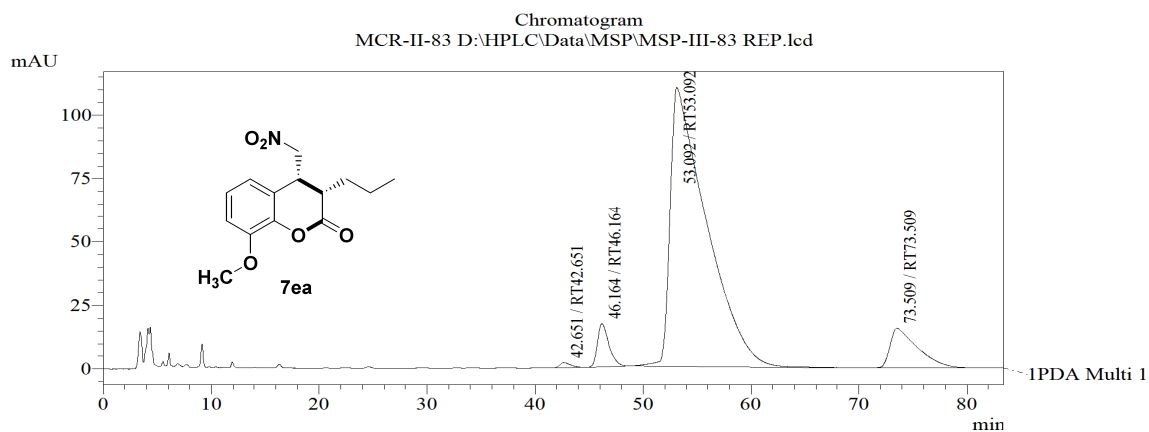
Daicel Chiralcel OJ-H, Hexane/ i- PrOH = 92:08, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT42.453	42.453	3204520	31604	45.618	51.345
2	RT48.403	48.403	309907	3821	4.412	6.208
3	RT58.372	58.372	3251225	23962	46.282	38.930
4	RT77.765	77.765	259092	2165	3.688	3.517
Total			7024745	61553	100.000	100.000

CHIRAL 7ea (99% ee):



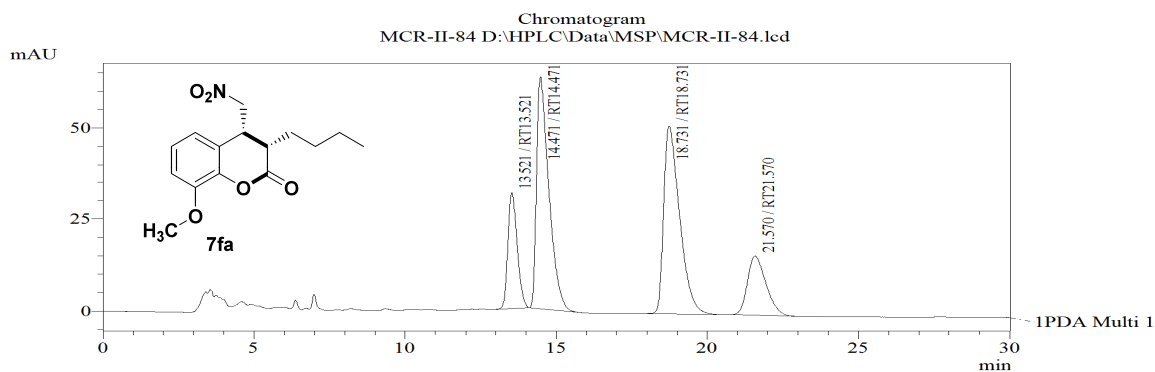
Daicel Chiralcel OJ-H, Hexane/ i- PrOH = 92:08, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT42.651	42.651	130466	1942	0.437	1.347
2	RT46.164	46.164	1260821	16994	4.226	11.785
3	RT53.092	53.092	25710301	109709	86.178	76.078
4	RT73.509	73.509	2732431	15560	9.159	10.790
Total			29834019	144205	100.000	100.000

RACEMIC 7fa:



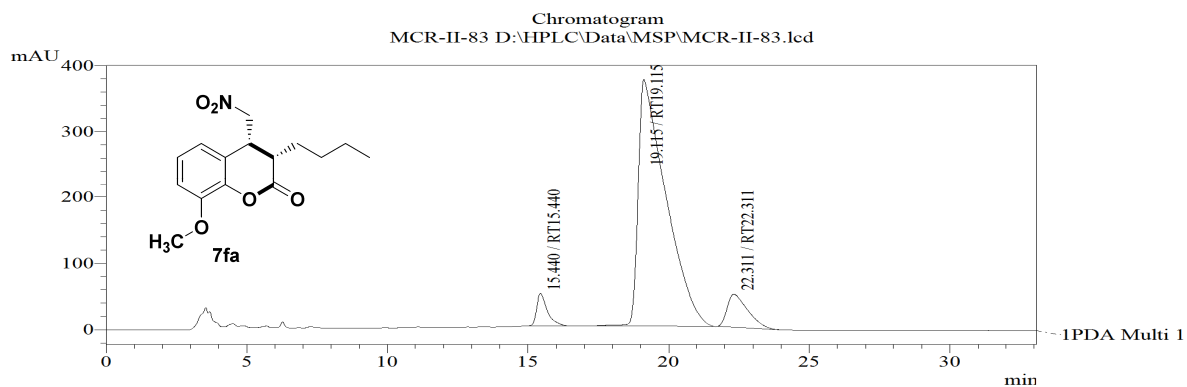
Daicel Chiralcel OJ-H, Hexane/ i-PrOH = 70:30, Flow Rate 1.0 mL/Min, 254 nm

PDA Ch1 254nm 4nm

PeakTable

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT13.521	13.521	678056	31485	13.472	19.418
2	RT14.471	14.471	1804639	63303	35.855	39.041
3	RT18.731	18.731	1860208	51238	36.959	31.600
4	RT21.570	21.570	690275	16119	13.714	9.941
Total			5033178	162145	100.000	100.000

CHIRAL 7fa (90% ee):



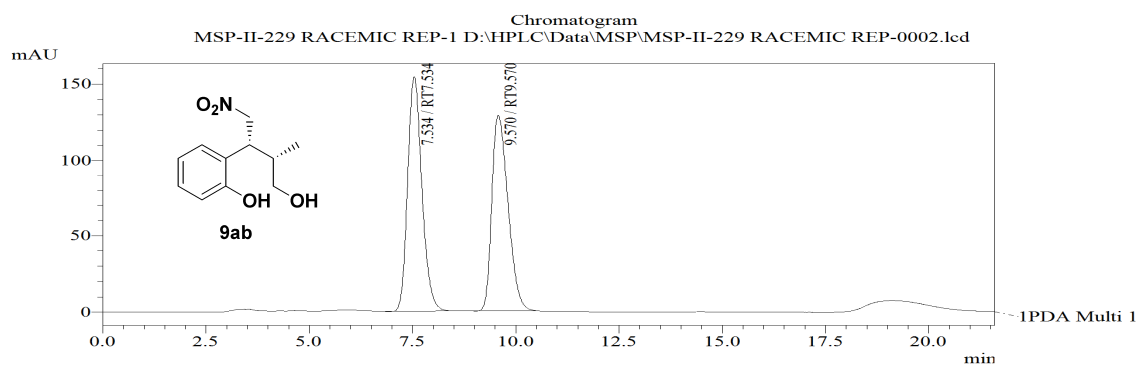
Daicel Chiralcel OJ-H, Hexane/ i-PrOH = 70:30, Flow Rate 1.0 mL/Min, 254 nm

PDA Ch1 254nm 4nm

PeakTable

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT15.440	15.440	1292964	48772	4.436	10.344
2	RT19.115	19.115	25347779	373417	86.966	79.196
3	RT22.311	22.311	2505977	49320	8.598	10.460
Total			29146720	471509	100.000	100.000

RACEMIC 9ab:



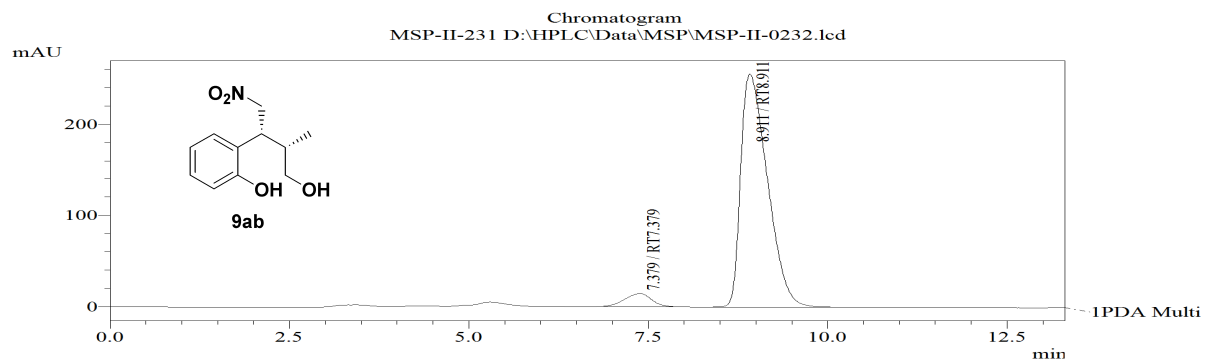
Daicel Chiralpak AS-H, Hexane/ i- PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT7.534	7.534	3704104	154363	51.259	54.476
2	RT9.570	9.570	3522185	128994	48.741	45.524
Total			7226289	283357	100.000	100.000

CHIRAL 9ab (89% ee):



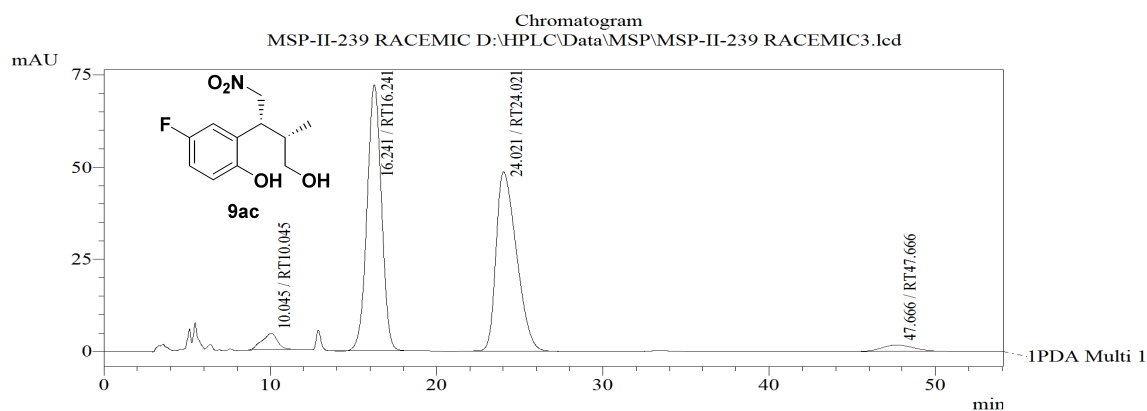
Daicel Chiralpak AS-H, Hexane/ i- PrOH = 80:20, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT7.379	7.379	360031	14132	5.158	5.241
2	RT8.911	8.911	6619846	255495	94.842	94.759
Total			6979876	269627	100.000	100.000

RACEMIC 9ac:



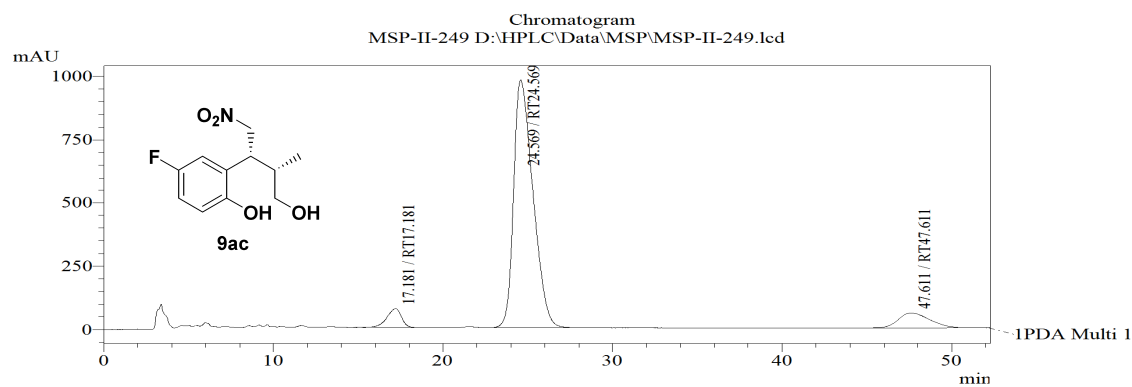
Daicel Chiralpak AS-H, Hexane/ i- PrOH = 90:10, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT10.045	10.045	283205	4366	3.153	3.442
2	RT16.241	16.241	4336633	72034	48.282	56.799
3	RT24.021	24.021	4129612	48682	45.977	38.386
4	RT47.666	47.666	232413	1741	2.588	1.373
Total			8981863	126824	100.000	100.000

CHIRAL 9ac (89% ee):



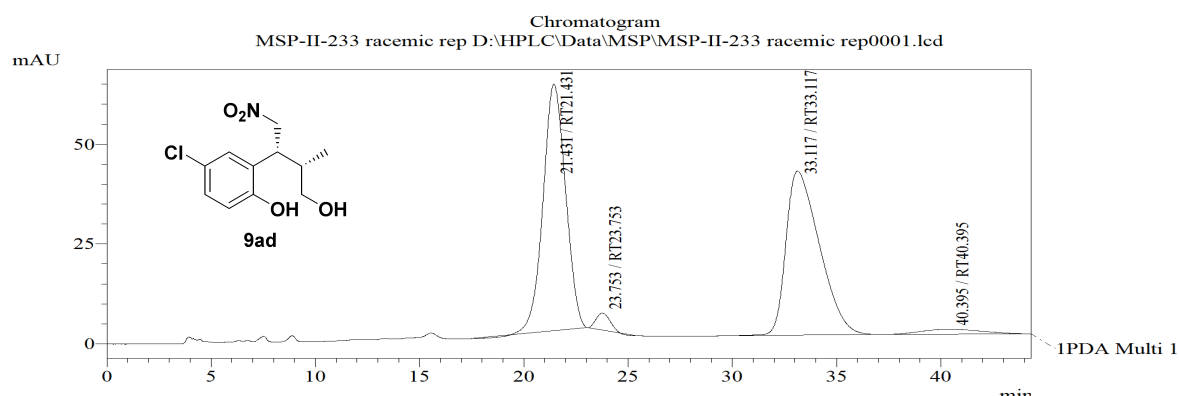
Daicel Chiralpak AS-H, Hexane/ i- PrOH = 90:10, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

PDA Ch1 200nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT17.181	17.181	4561958	75185	5.068	6.758
2	RT24.569	24.569	77929053	978058	86.566	87.907
3	RT47.611	47.611	7531388	59361	8.366	5.335
Total			90022399	1112604	100.000	100.000

RACEMIC 9ad:



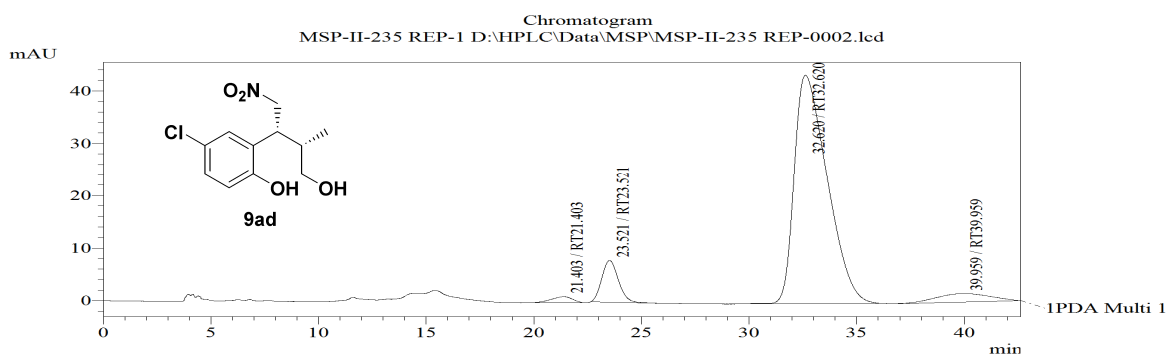
Daicel Chiralpak AS-H, Hexane/ i-PrOH = 90:10, Flow Rate 0.8 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT21.431	21.431	4633275	61640	48.045	56.935
2	RT23.753	23.753	193126	4239	2.003	3.915
3	RT33.117	33.117	4570137	41144	47.391	38.003
4	RT40.395	40.395	247030	1242	2.562	1.147
Total			9643567	108264	100.000	100.000

CHIRAL 9ad (96.8% ee):



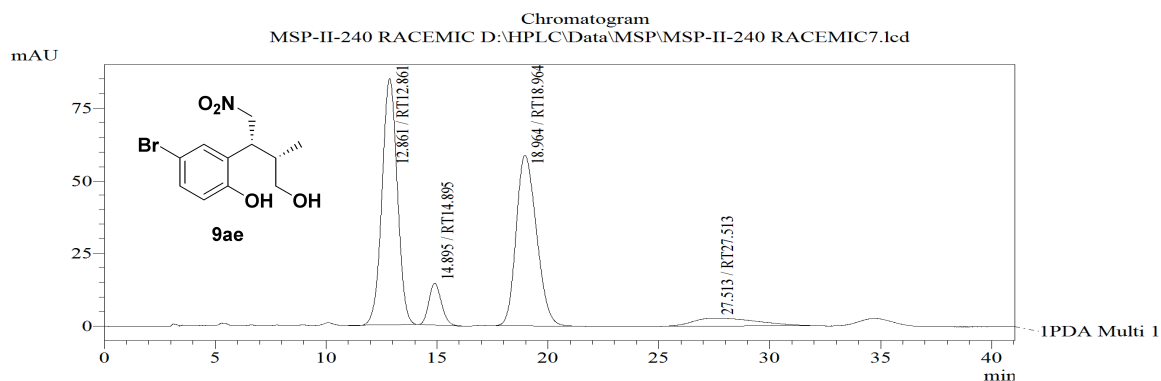
Daicel Chiralpak AS-H, Hexane/ i-PrOH = 90:10, Flow Rate 0.8 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT21.403	21.403	75310	1131	1.391	2.086
2	RT23.521	23.521	423480	7923	7.824	14.610
3	RT32.620	32.620	4636695	43576	85.662	80.354
4	RT39.959	39.959	277299	1600	5.123	2.950
Total			5412785	54230	100.000	100.000

RACEMIC 9ae:



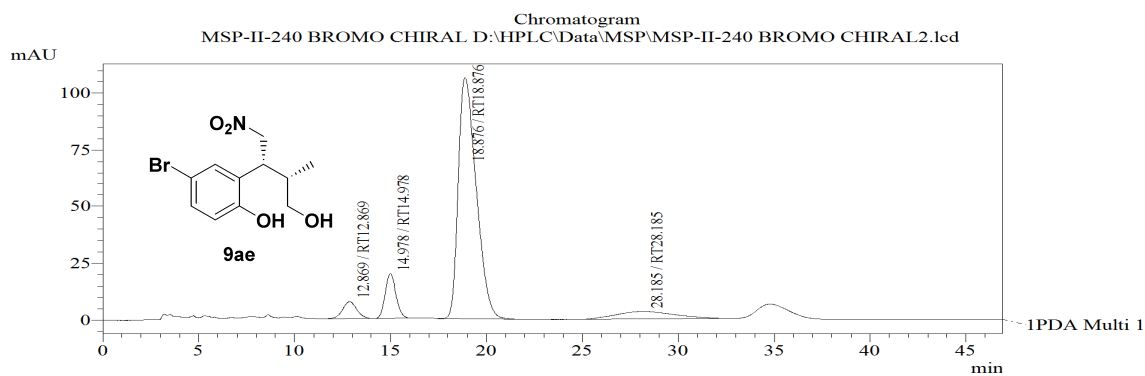
Daicel Chiralpak AS-H, Hexane/ i-PrOH = 88:12, Flow Rate 1.0 mL/Min, 254 nm

PDA Ch1 254nm 4nm

PeakTable

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT12.861	12.861	4055604	84679	45.630	52.767
2	RT14.895	14.895	561565	14299	6.318	8.911
3	RT18.964	18.964	3739635	58876	42.075	36.688
4	RT27.513	27.513	531268	2624	5.977	1.635
Total			8888072	160478	100.000	100.000

CHIRAL 9ae (89.5% ee):



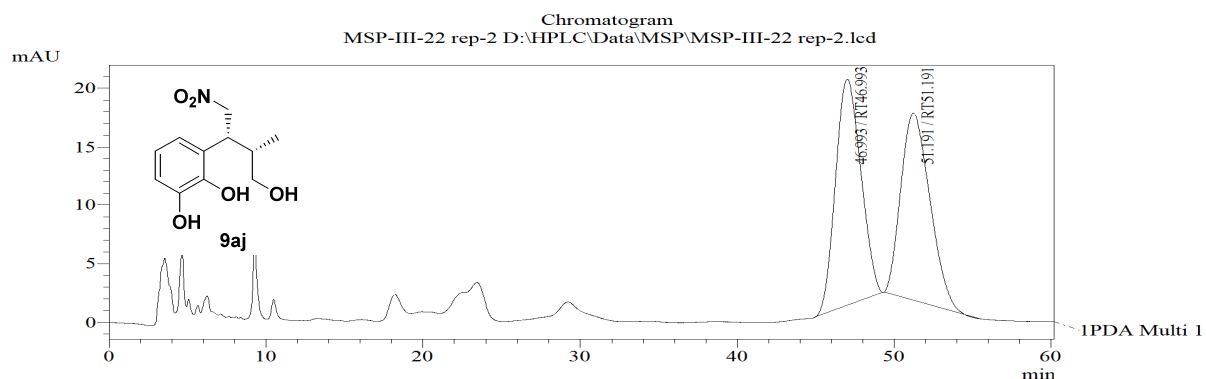
Daicel Chiralpak AS-H, Hexane/ i-PrOH = 88:12, Flow Rate 1.0 mL/Min, 254 nm

PDA Ch1 254nm 4nm

PeakTable

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT12.869	12.869	382904	7500	4.401	5.502
2	RT14.978	14.978	767479	19577	8.821	14.359
3	RT18.876	18.876	6888179	106113	79.165	77.833
4	RT28.185	28.185	662502	3144	7.614	2.306
Total			8701065	136334	100.000	100.000

RACEMIC 9aj:



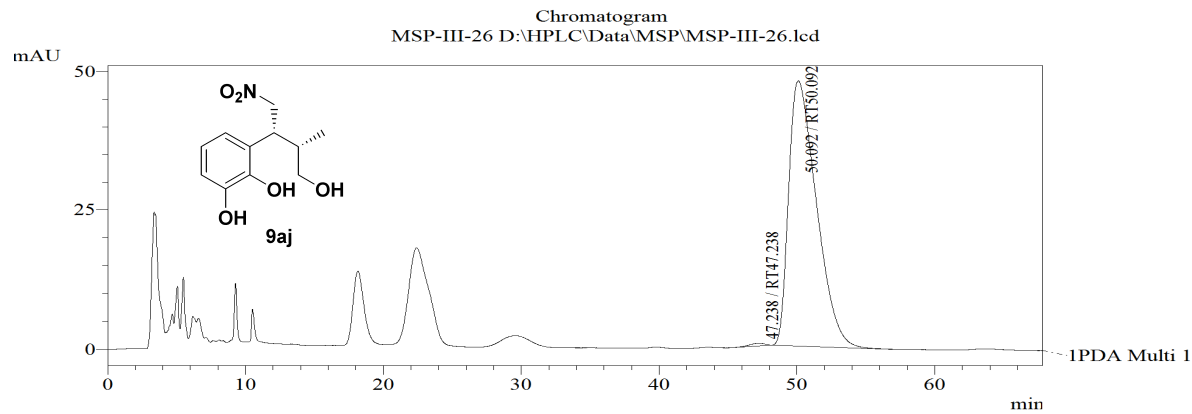
Daicel Chiralpak AS-H, Hexane/ i- PrOH = 92:08, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT46.993	46.993	2164025	19305	51.207	54.704
2	RT51.191	51.191	2061968	15985	48.793	45.296
Total			4225993	35290	100.000	100.000

CHIRAL 9aj (99% ee):



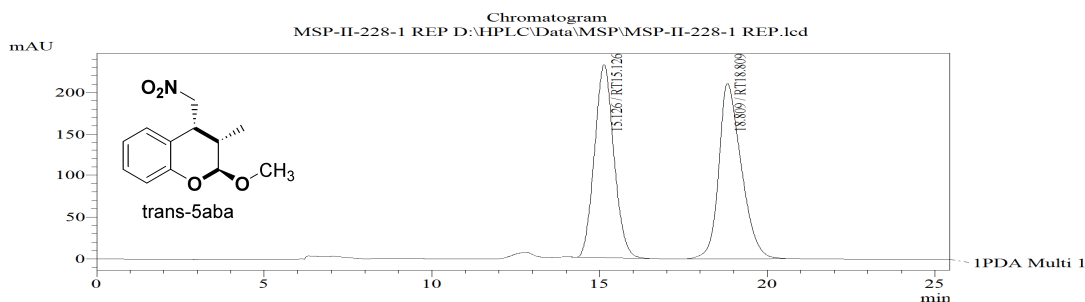
Daicel Chiralpak AS-H, Hexane/ i- PrOH = 92:08, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT47.238	47.238	32769	454	0.490	0.943
2	RT50.092	50.092	6661109	47691	99.510	99.057
Total			6693878	48145	100.000	100.000

RACEMIC *trans*-5aba:



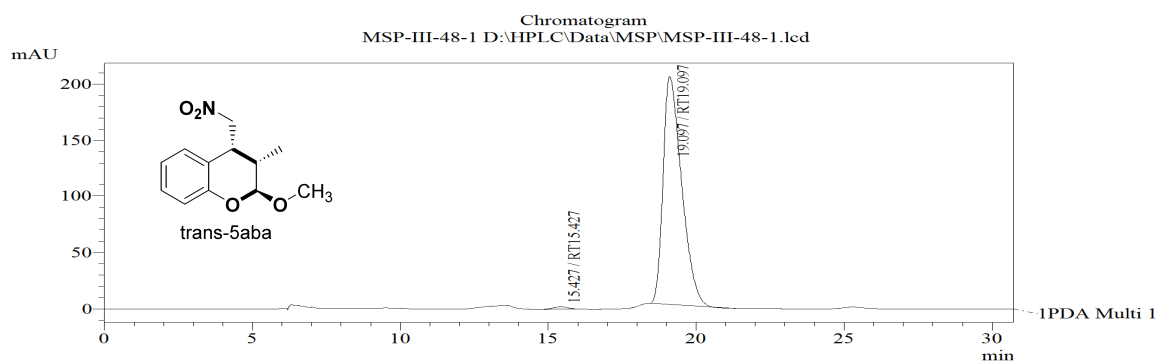
Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 98:02, Flow Rate 0.5 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT15.126	15.126	9309666	232022	48.586	52.356
2	RT18.809	18.809	9851554	211140	51.414	47.644
Total			19161220	443162	100.000	100.000

CHIRAL *trans*-5aba (98% *ee*):



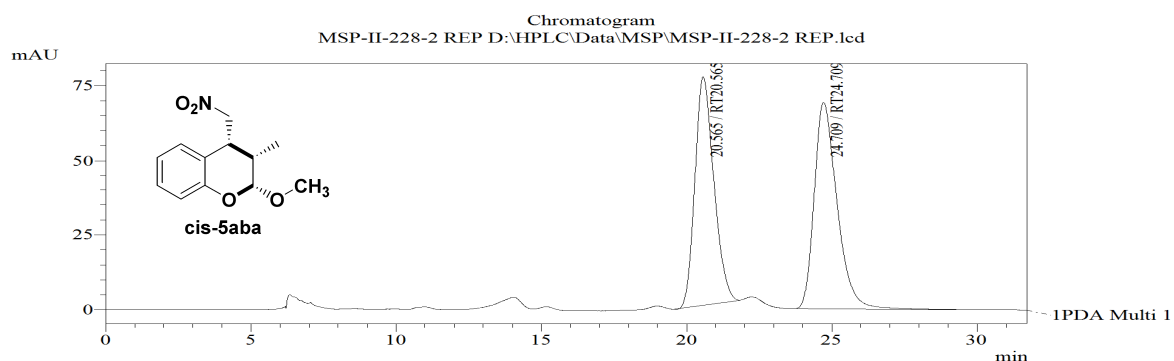
Daicel Chiralcel OD-H, Hexane/ *i*-PrOH = 98:02, Flow Rate 0.5 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT15.427	15.427	72418	2133	0.815	1.039
2	RT19.097	19.097	8810257	203067	99.185	98.961
Total			8882674	205200	100.000	100.000

RACEMIC *cis*-5aba:



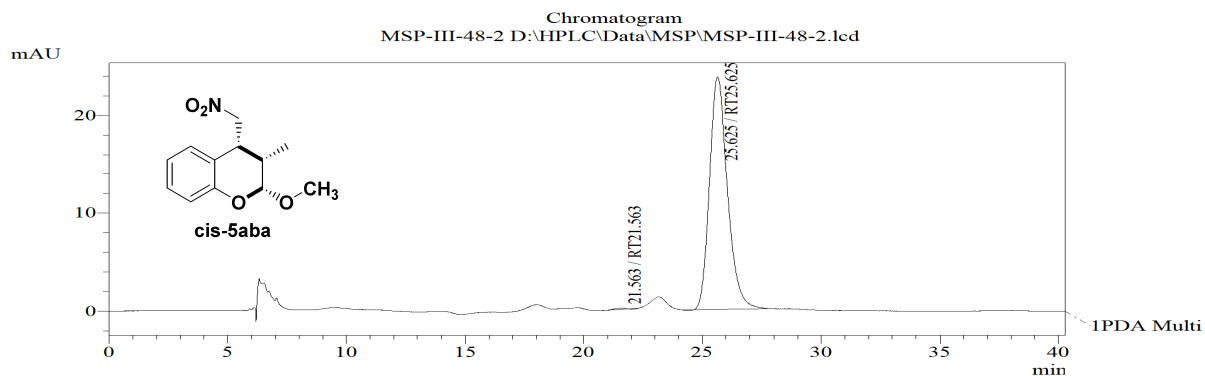
Daicel Chiralcel OD-H, Hexane/ i- PrOH = 98:02, Flow Rate 0.5 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT20.565	20.565	3480895	76370	47.990	52.498
2	RT24.709	24.709	3772431	69101	52.010	47.502
Total			7253326	145470	100.000	100.000

CHIRAL *cis*-5aba (99% *ee*):



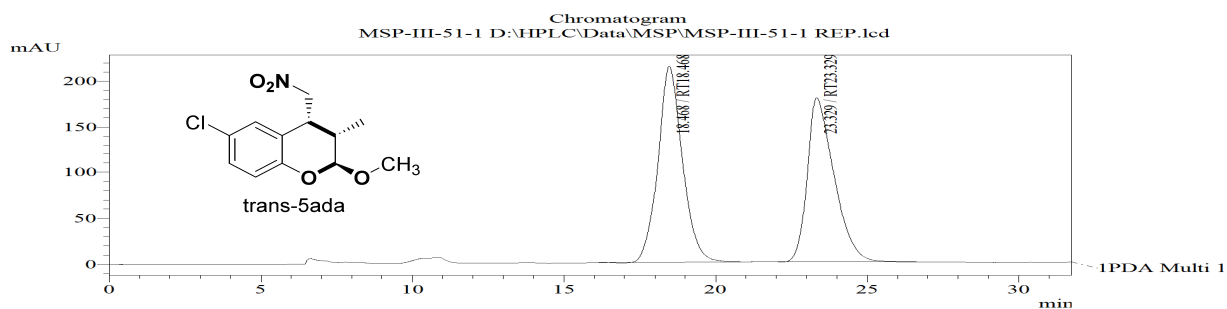
Daicel Chiralcel OD-H, Hexane/ i- PrOH = 98:02, Flow Rate 0.5 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT21.563	21.563	2657	141	0.210	0.589
2	RT25.625	25.625	1260675	23773	99.790	99.411
Total			1263332	23914	100.000	100.000

RACEMIC *trans*-5ada:



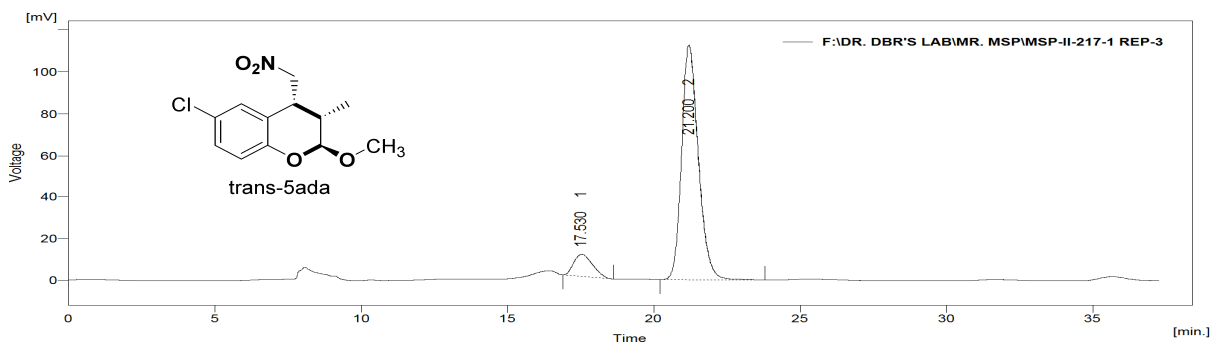
Daicel Chiralcel OD-H, Hexane/ i-PrOH = 99:01, Flow Rate 0.5 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT18.468	18.468	12011832	213840	52.377	54.441
2	RT23.329	23.329	10921717	178952	47.623	45.559
Total			22933549	392792	100.000	100.000

CHIRAL *trans*-5ada (80.7% ee):

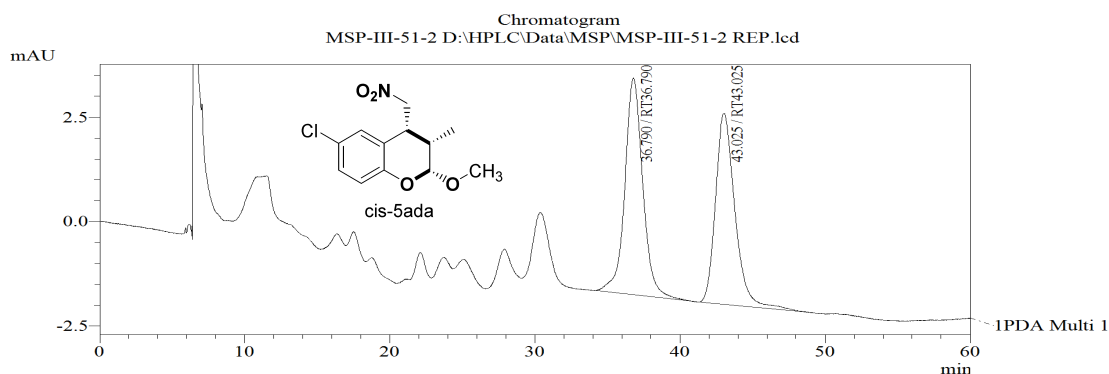


Daicel Chiralcel OD-H, Hexane/ i-PrOH = 99:01, Flow Rate 0.5 mL/Min, 254 nm

Result Table (Uncal - F:\DR. DBR'S LAB\MR. MSP\MSP-II-217-1 REP-3)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	17.530	319.160	7.109	9.7	8.7	0.71
2	21.200	2982.270	75.069	90.3	91.3	0.60
	Total	3301.430	82.178	100.0	100.0	

RACEMIC *cis*-5ada:



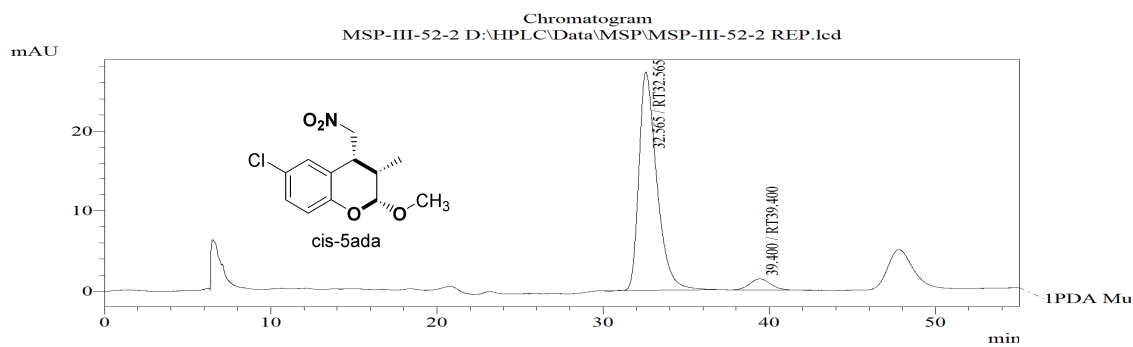
Daicel Chiralcel OD-H, Hexane/ i-PrOH = 99:01, Flow Rate 0.5 mL/Min, 254

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT36.790	36.790	442292	5173	51.077	53.074
2	RT43.025	43.025	423641	4574	48.923	46.926
Total			865934	9747	100.000	100.000

CHIRAL *cis*-5ada (90% *ee*):



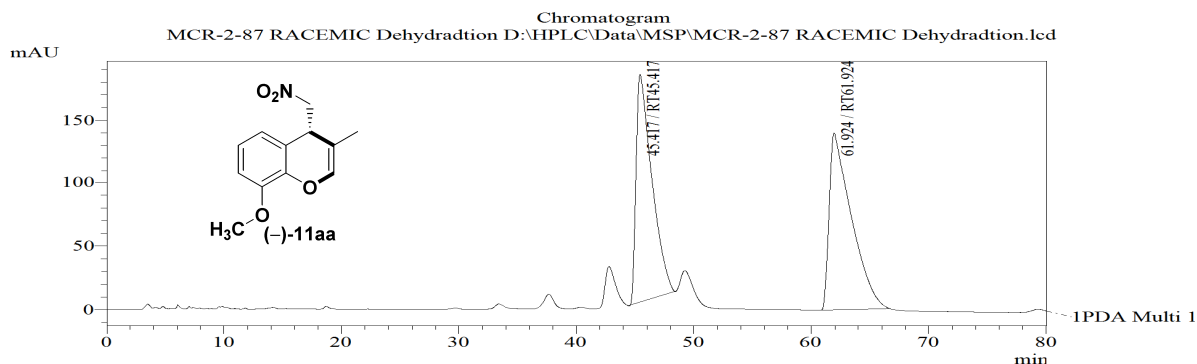
Daicel Chiralcel OD-H, Hexane/ i-PrOH = 99:01, Flow Rate 0.5 mL/Min, 254

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT32.565	32.565	2032991	27245	94.461	95.171
2	RT39.400	39.400	119213	1383	5.539	4.829
Total			2152204	28628	100.000	100.000

RACEMIC (-)-11aa:



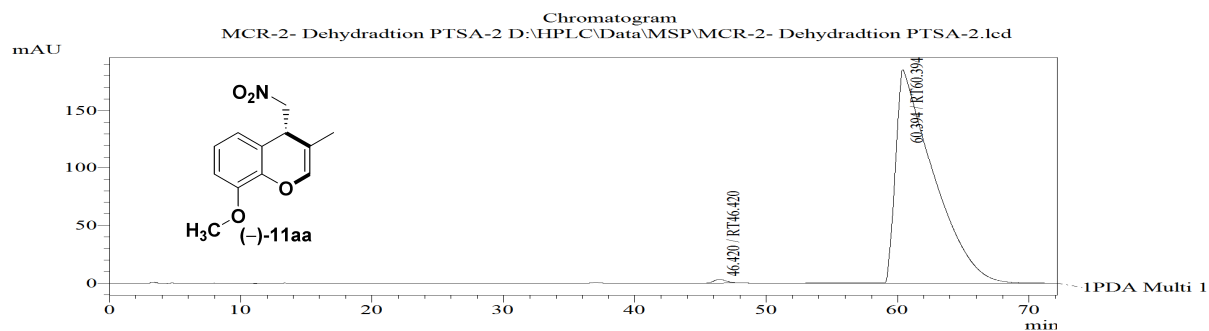
Daicel Chiralcel OJ-H, Hexane/ i-PrOH = 90:10, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT45.417	45.417	16692847	179878	47.442	56.261
2	RT61.924	61.924	18493279	139845	52.558	43.739
Total			35186126	319723	100.000	100.000

CHIRAL (-)-11aa (99% ee):



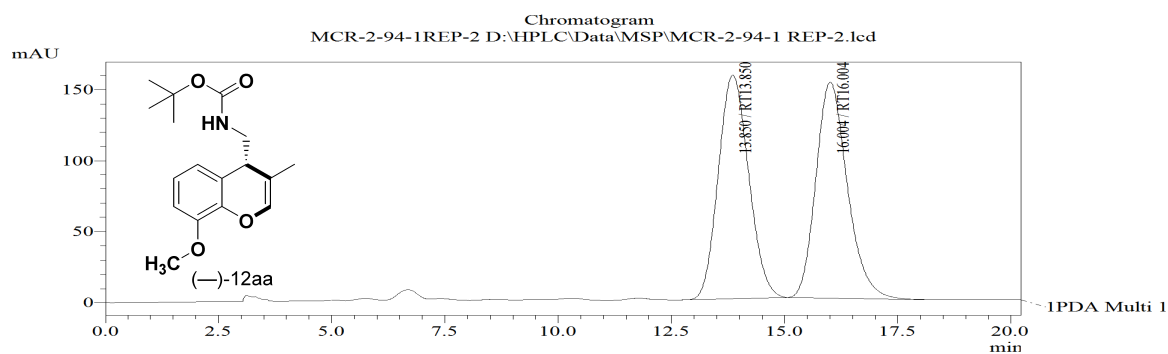
Daicel Chiralcel OJ-H, Hexane/ i-PrOH = 90:10, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT46.420	46.420	211455	3238	0.566	1.717
2	RT60.394	60.394	37171279	185326	99.434	98.283
Total			37382733	188564	100.000	100.000

RACEMIC (-)-12aa:



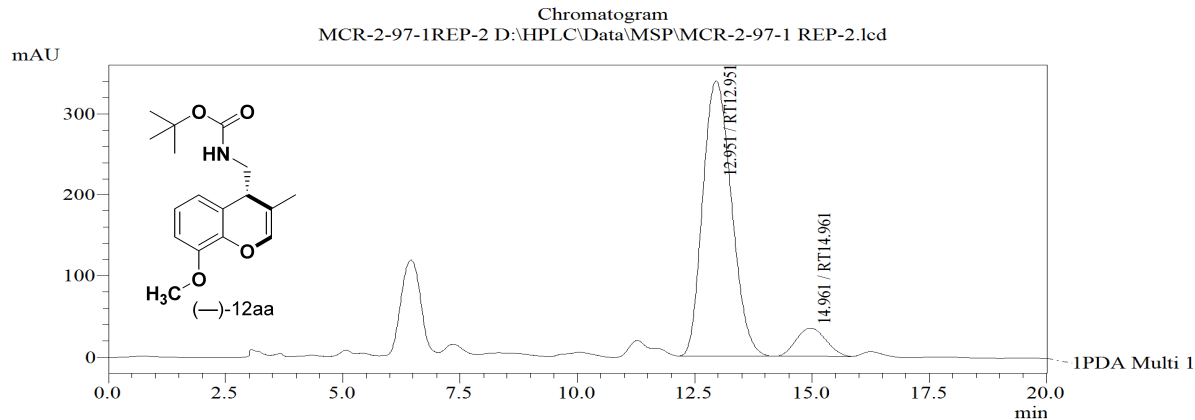
Daicel Chiralpak AD-H, Hexane/ i-PrOH = 97:03, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT13.850	13.850	7343422	157205	50.044	50.864
2	RT16.004	16.004	7330436	151866	49.956	49.136
Total			14673858	309071	100.000	100.000

CHIRAL (-)-12aa (81% ee):



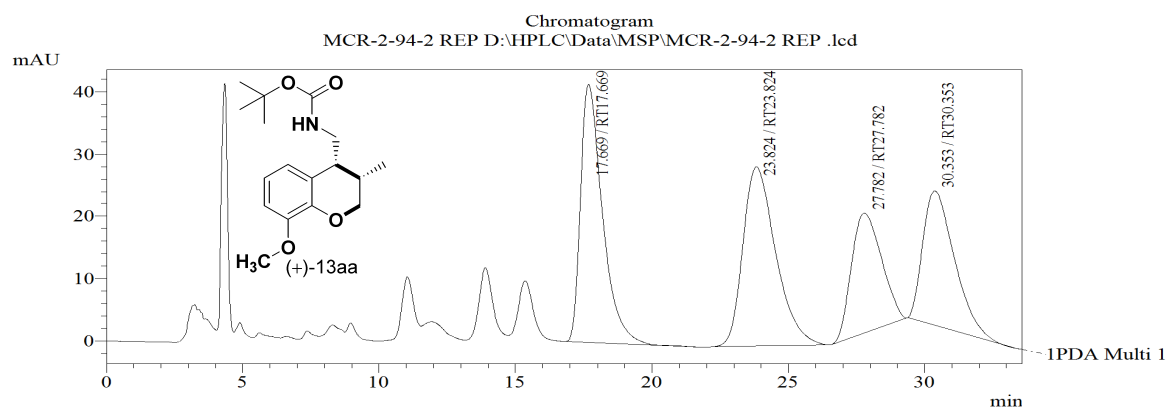
Daicel Chiralpak AD-H, Hexane/ i-PrOH = 97:03, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT12.951	12.951	14429772	339253	90.493	90.778
2	RT14.961	14.961	1515900	34462	9.507	9.222
Total			15945672	373715	100.000	100.000

RACEMIC (+)-13aa:



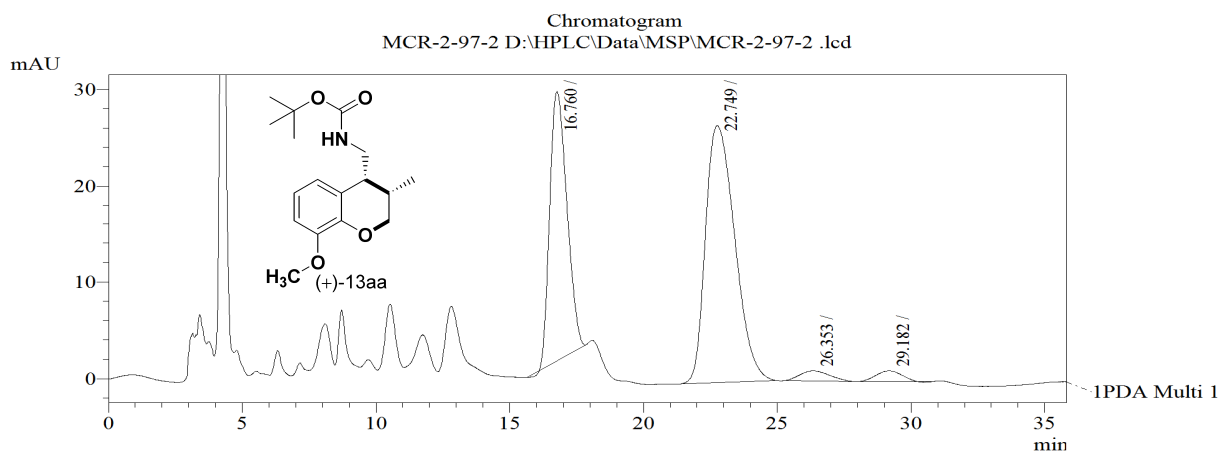
Daicel Chiralcel OD-H, Hexane/ i-PrOH = 95:05, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT17.669	17.669	2303365	41385	29.085	37.289
2	RT23.824	23.824	2404373	28825	30.361	25.972
3	RT27.782	27.782	1470317	19166	18.566	17.269
4	RT30.353	30.353	1741309	21608	21.988	19.470
Total			7919363	110984	100.000	100.000

CHIRAL (+)-13aa (93% ee):



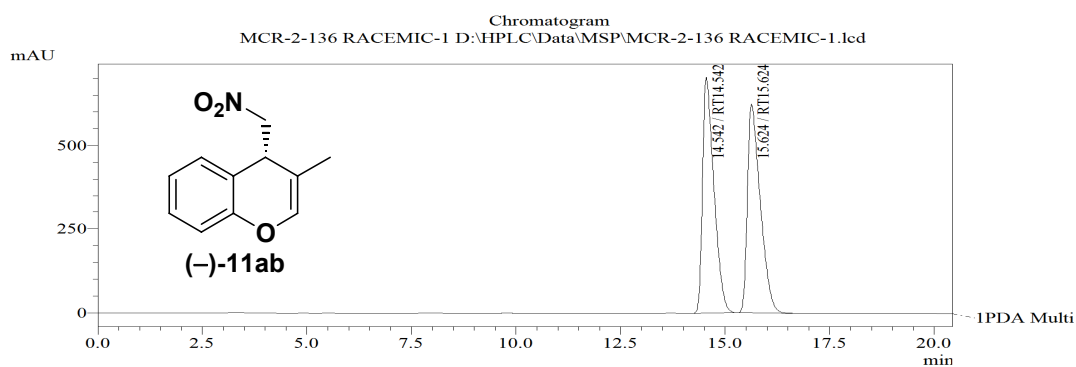
Daicel Chiralcel OD-H, Hexane/ i-PrOH = 95:05, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1		16.760	1245136	27943	36.280	49.223
2		22.749	2031344	26654	59.188	46.952
3		26.353	82373	1058	2.400	1.864
4		29.182	73139	1113	2.131	1.961
Total			3431991	56769	100.000	100.000

RACEMIC 11ab



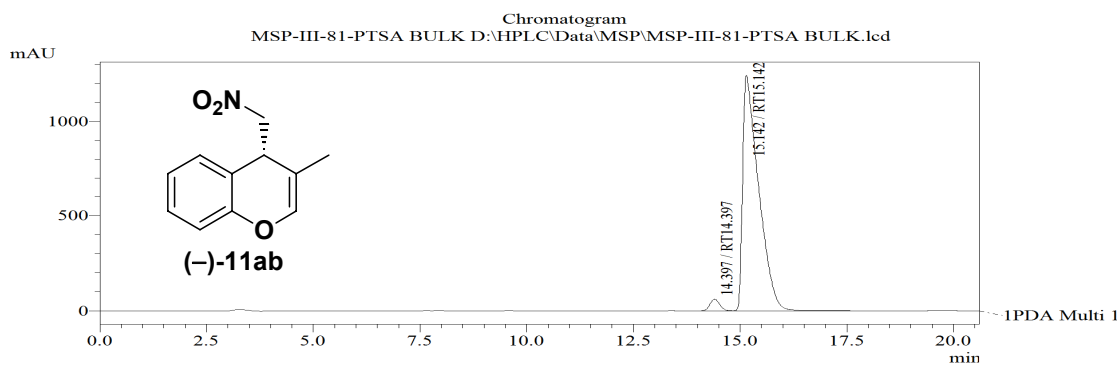
Column: Chiral Cel OJ-H, Hexane/ i-ProH = 90:10, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT14.542	14.542	13327387	703402	49.947	53.037
2	RT15.624	15.624	13355785	622840	50.053	46.963
Total			26683172	1326242	100.000	100.000

CHIRAL (-)-11ab (94% ee)



Column: Chiral Cel OJ-H, Hexane/ i-ProH = 90:10, Flow Rate 1.0 mL/Min, 254 nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Name	Ret. Time	Area	Height	Area %	Height %
1	RT14.397	14.397	985751	61310	2.975	4.699
2	RT15.142	15.142	32146132	1243370	97.025	95.301
Total			33131883	1304680	100.000	100.000