

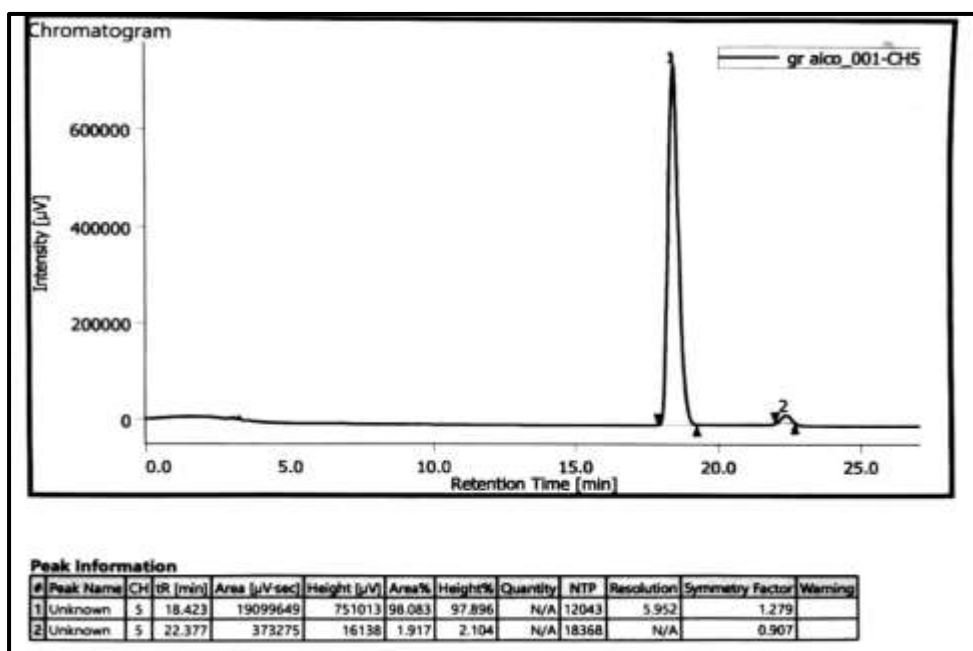
## Supporting information for

### One-pot kinetic resolution-Mitsunobu reaction to access optically pure compounds, silver salts in aid of substitution protocol

Hiten B. Raval and Ashutosh V. Bedekar\*

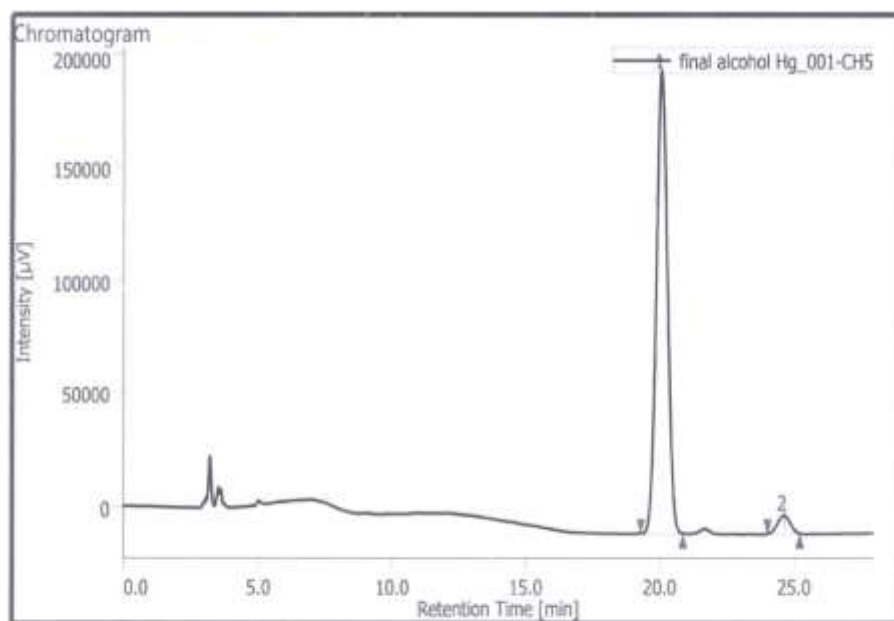
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HPLC (*R*) 1-(3-nitrophenyl)ethan-1-ol for CH<sub>3</sub>COOAg



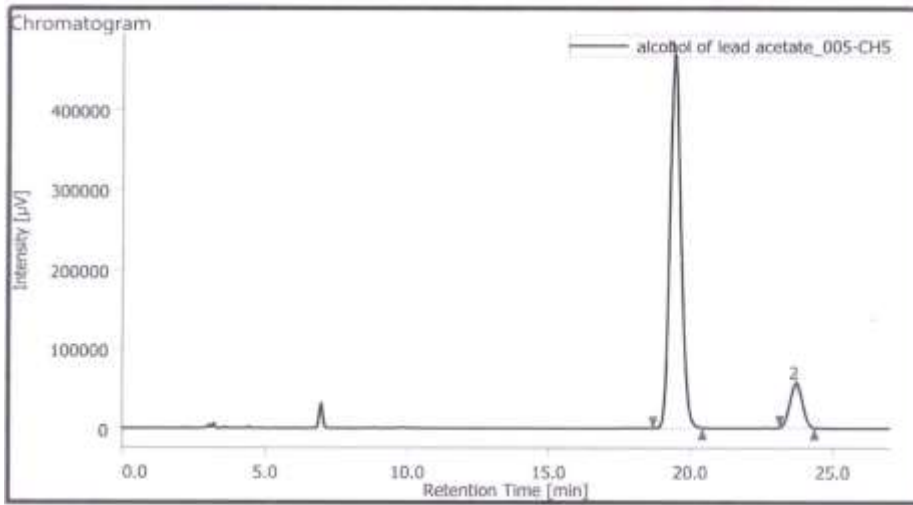
HPLC Condition for compound 1-(3-nitrophenyl)ethan-1-ol<sup>b</sup> Determined by HPLC analysis (chiral column Amylose, 1ml/min IPA (5.0%) in hexane; 18.42 min for (*R*)-1 and 22.37 min for (*S*)-1)

HPLC (*R*) 1-(3-nitrophenyl)ethan-1-ol for Hg(OAc)<sub>2</sub>



Peak Information												
#	Peak Name	CH	TR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	20.043	5844907	204532	95.683	96.203	N/A	11178	5.562	1.070	
2	Unknown	5	24.613	263703	8072	4.317	3.797	N/A	12255	N/A	0.962	

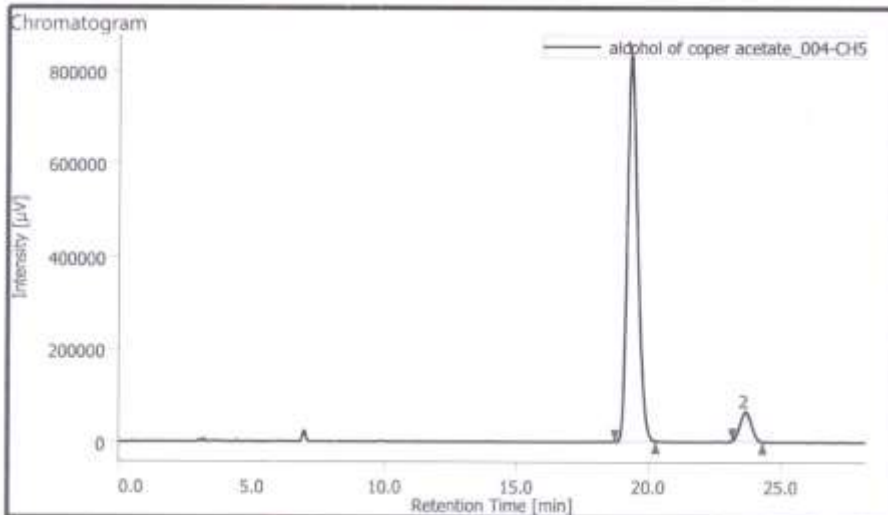
HPLC (R) 1-(3-nitrophenyl)ethan-1-ol for Pb(OAc)<sub>2</sub>



Peak Information

#	Peak Name	CH	RT [min]	Area [µV·sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	19.413	12624275	469662	88.066	89.353	N/A	12144	5.612	1.196	
2	Unknown	5	23.667	1710704	33990	11.934	10.647	N/A	13278	N/A	1.020	

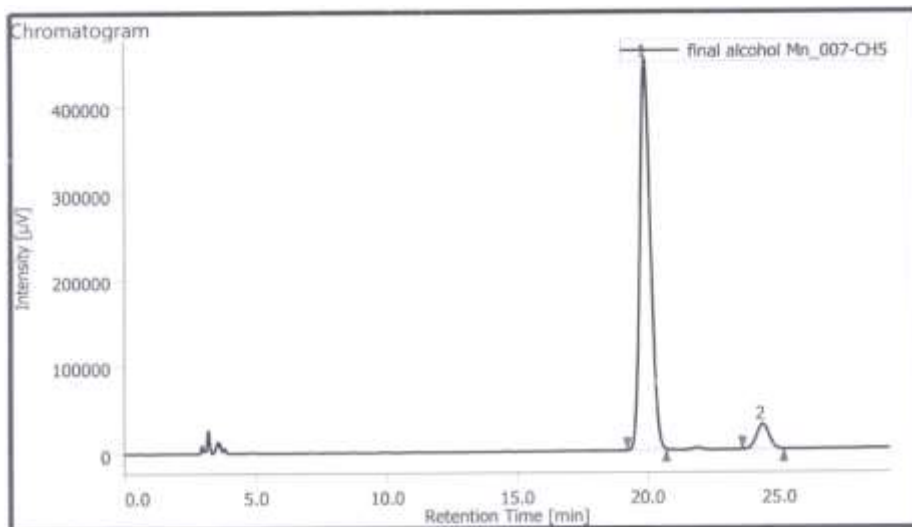
HPLC (R) 1-(3-nitrophenyl)ethan-1-ol for Cu(OAc)<sub>2</sub>



Peak Information

#	Peak Name	CH	RT [min]	Area [µV·sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	19.323	22798679	833114	92.441	92.978	N/A	11593	5.700	1.331	
2	Unknown	5	23.647	1864222	62920	7.559	7.022	N/A	13867	N/A	1.054	

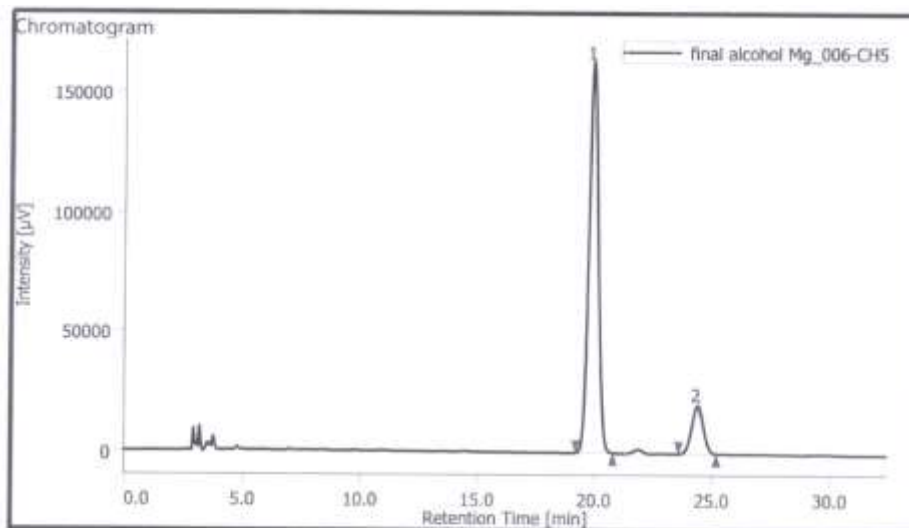
HPLC (R) 1-(3-nitrophenyl)ethan-1-ol for Mn(OAc)<sub>2</sub>



Peak Information

#	Peak Name	CH	TR [min]	Area [µV·sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	19.870	12642180	451131	93.109	94.148	N/A	11618	5.543	1.238	
2	Unknown	S	24.353	935691	28039	6.891	5.852	N/A	12124	N/A	1.014	

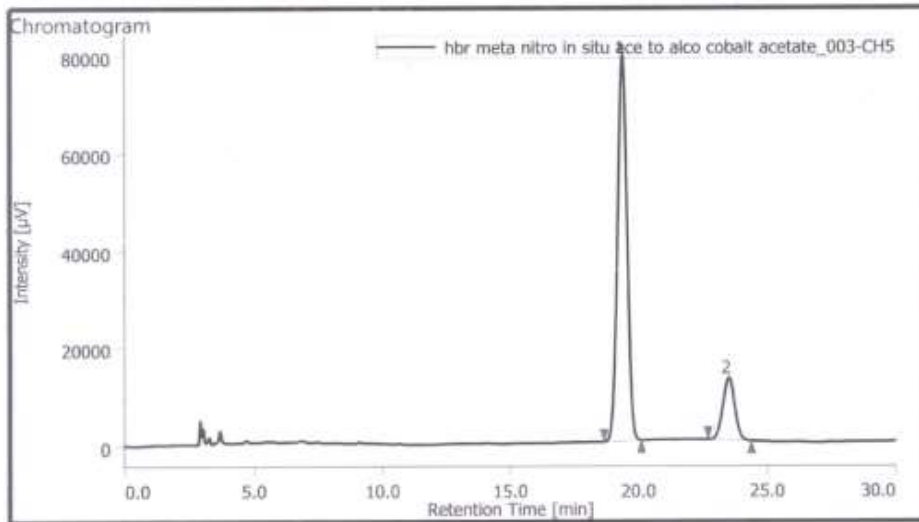
HPLC (R) 1-(3-nitrophenyl)ethan-1-ol for Mg(OAc)<sub>2</sub>



Peak Information

#	Peak Name	CH	TR [min]	Area [µV·sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	19.903	4529994	163717	86.951	89.163	N/A	11864	5.470	1.066	
2	Unknown	S	24.373	679652	19899	13.049	10.837	N/A	11536	N/A	1.004	

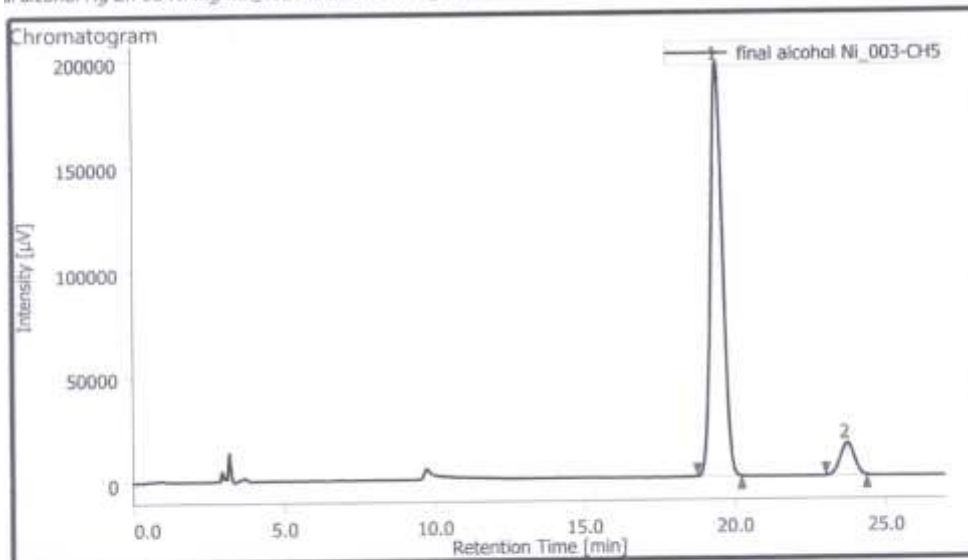
HPLC (R) 1-(3-nitrophenyl)ethan-1-ol for Co(OAc)<sub>2</sub>



Peak Information

#	Peak Name	CH	tR [min]	Area [μV-sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	19.373	2043977	79136	83.453	86.215	N/A	12821	5.399	0.993	
2	Unknown	S	23.497	405275	12653	16.547	13.785	N/A	12306	N/A	1.000	

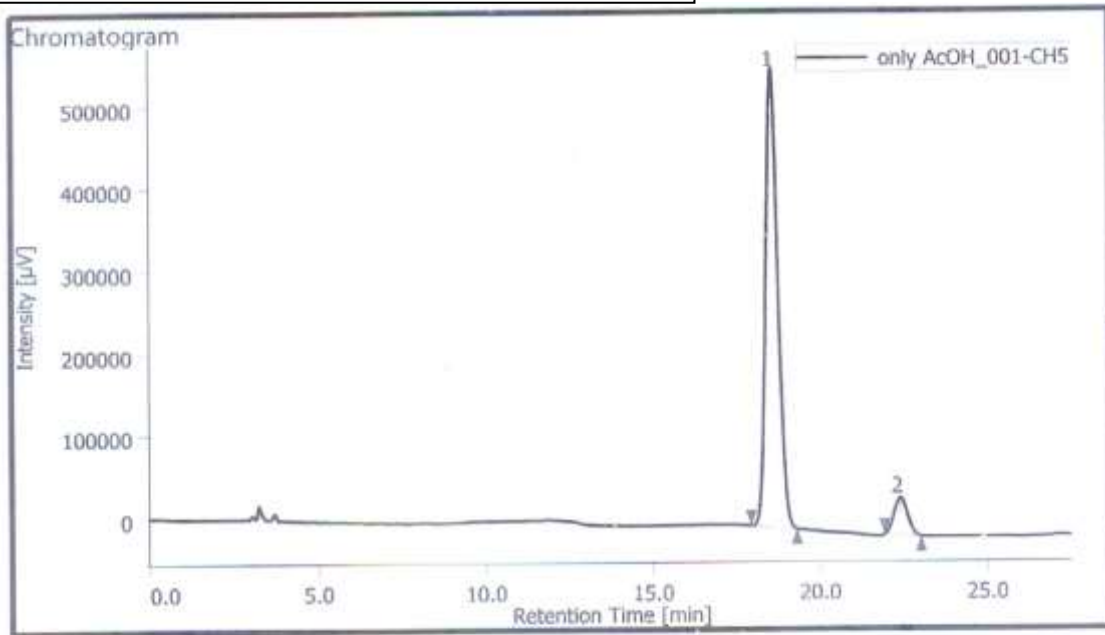
HPLC (R) 1-(3-nitrophenyl)ethan-1-ol for Ni(OAc)<sub>2</sub>



Peak Information

#	Peak Name	CH	tR [min]	Area [μV-sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	19.420	5380817	196443	91.651	92.914	N/A	11456	5.355	1.055	
2	Unknown	S	23.697	490175	14983	8.349	7.086	N/A	11717	N/A	0.984	

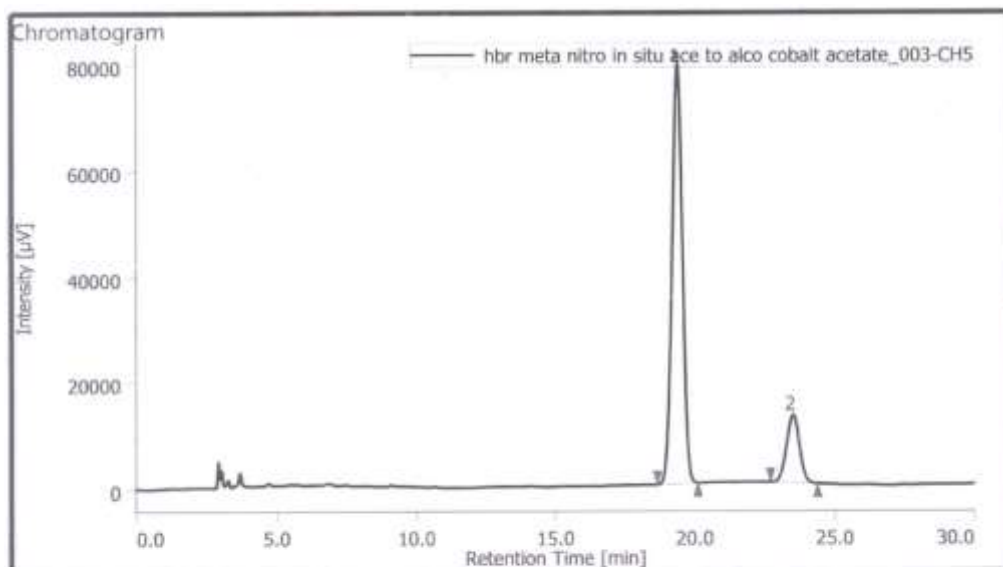
HPLC (R) 1-(3-nitrophenyl)ethan-1-ol for CH<sub>3</sub>COONa



Peak Information

#	Peak Name	CH	tR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	18.553	14009885	557357	91.836	92.676	N/A	12485	5.373	1.178	
2	Unknown	5	22.387	1245444	44044	8.164	7.324	N/A	13650	N/A	1.078	

HPLC (R) 1-(3-nitrophenyl)ethan-1-ol(Co(OAc)2/AgNO3:2.0/0.0)

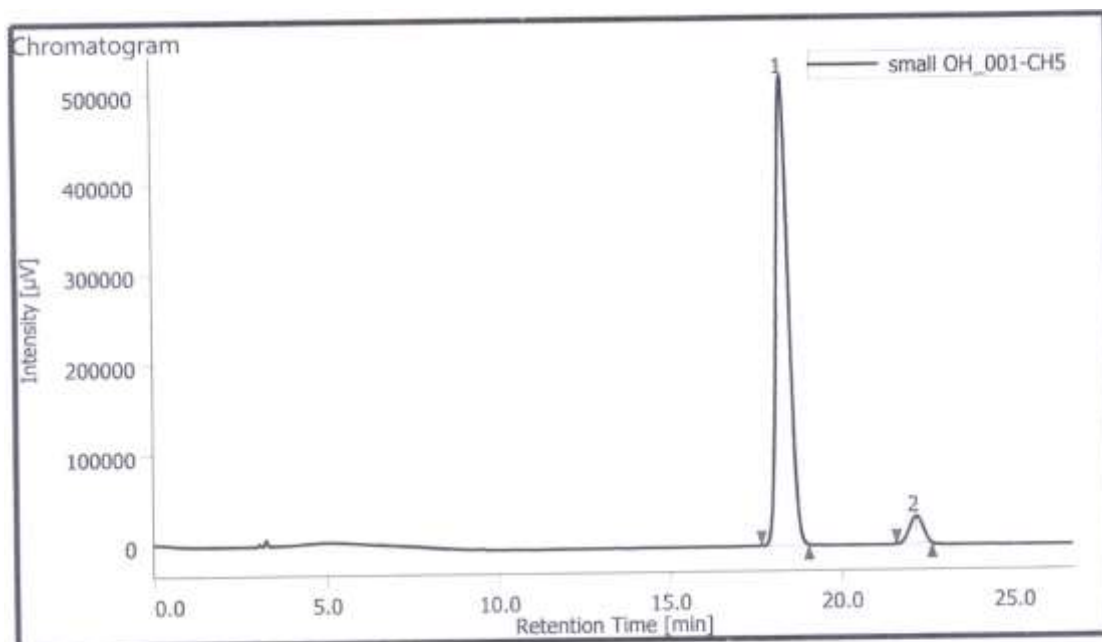


Peak Information

#	Peak Name	CH	tR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	18.373	2043977	79136	83.453	86.215	N/A	12821	5.399	0.993	
2	Unknown	S	23.497	405275	12653	16.547	13.785	N/A	12306	N/A	1.000	

HPLC Condition for compound 1-(3-nitrophenyl)ethan-1-ol<sup>b</sup> Determined by HPLC analysis (chiral column Amylose, 1ml/min IPA (5.0%) in hexane; 18.42 min for (R)-1 and 22.37 min for (S)-1)

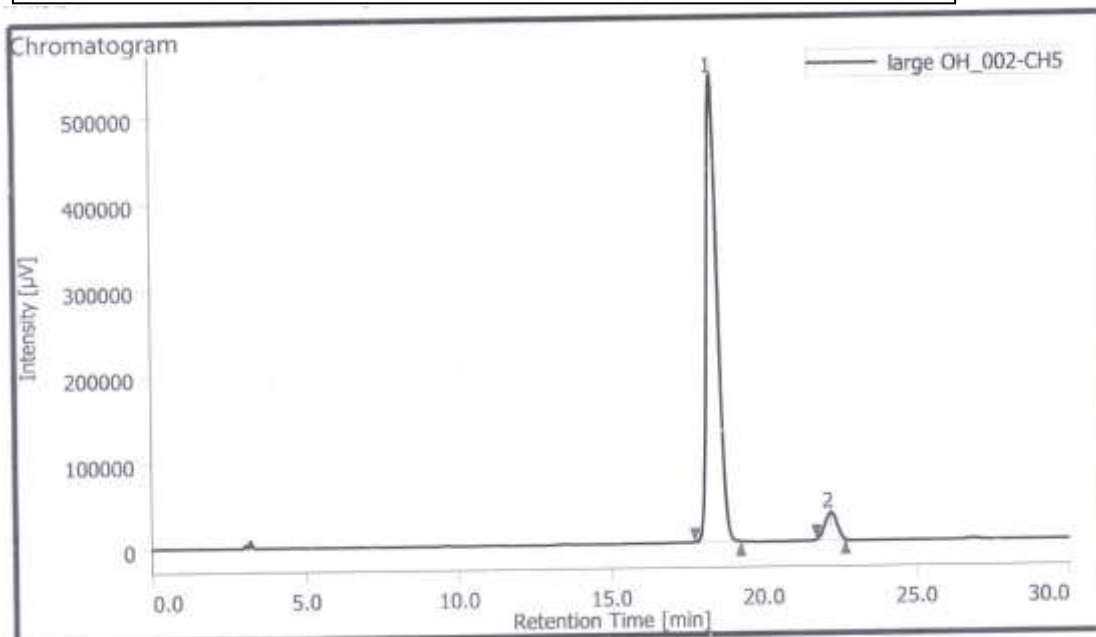
HPLC (R) 1-(3-nitrophenyl)ethan-1-ol(Co(OAc)2/AgNO3:1.75/0.25)



Peak Information

#	Peak Name	CH	tR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	18.297	12968278	522047	93.944	94.551	N/A	12403	5.453	1.164	
2	Unknown	S	22.140	835998	30086	6.056	5.449	N/A	13713	N/A	0.936	

HPLC (R) 1-(3-nitrophenyl)ethan-1-ol(Co(OAc)2/AgNO3:1.50/0.50)

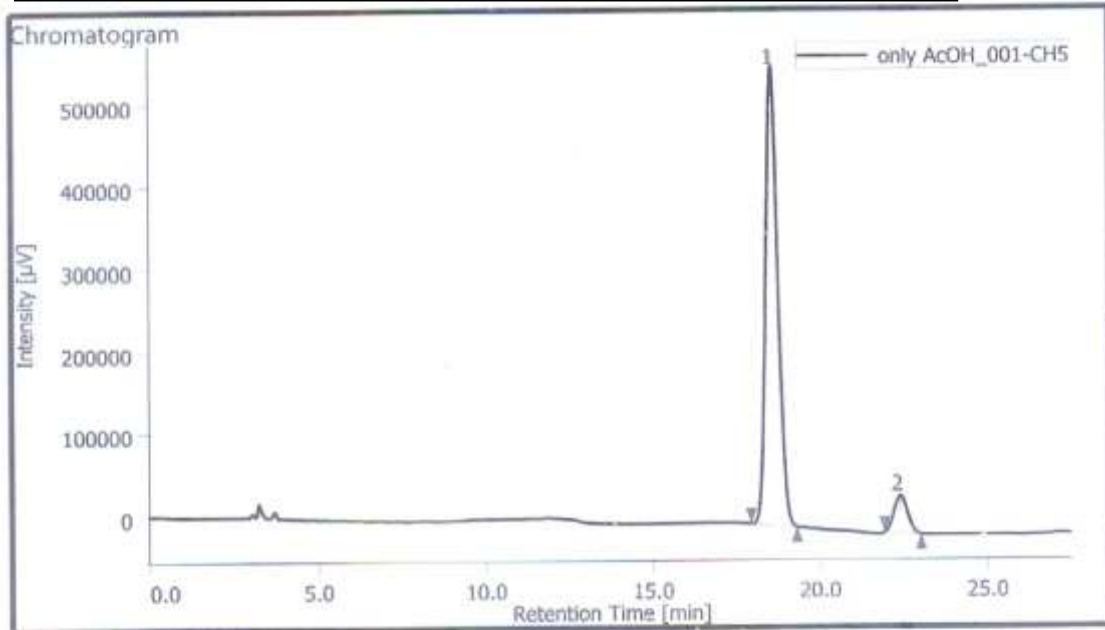


**Peak Information**

#	Peak Name	CH	tR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	18.370	13382699	540080	94.067	94.488	N/A	12606	5.534	1.157	
2	Unknown	5	22.203	844056	31504	5.933	5.512	N/A	14619	N/A	0.984	



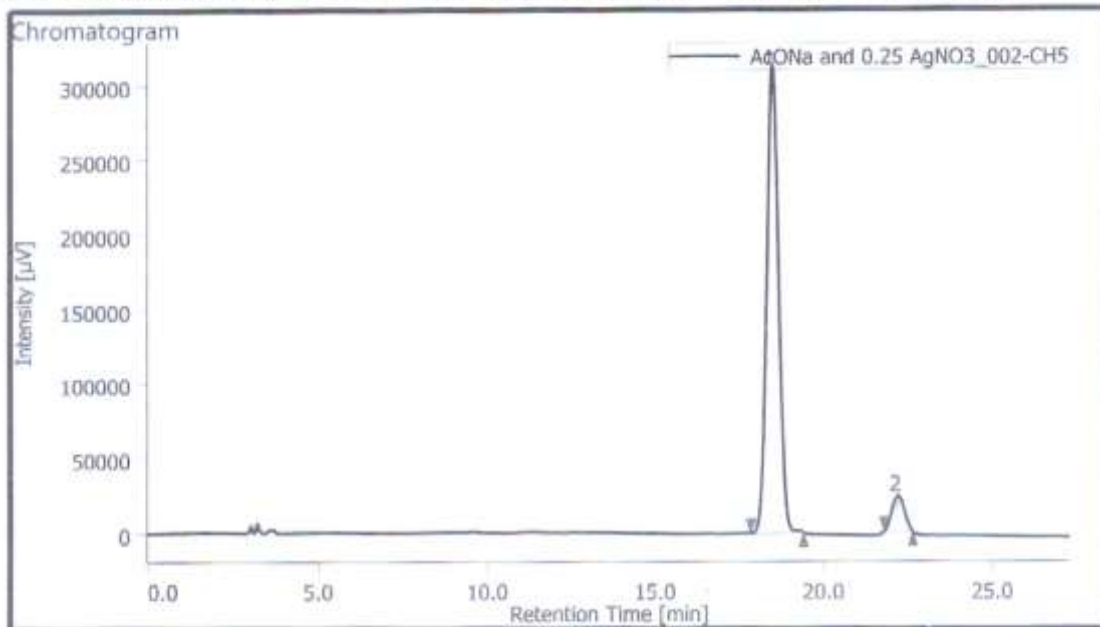
HPLC (R) 1-(3-nitrophenyl)ethan-1-ol(CH<sub>3</sub>COONa/AgNO<sub>3</sub>:2.0/0.0)



**Peak Information**

#	Peak Name	CH	tR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	18.553	14009885	557357	91.836	92.676	N/A	12485	5.373	1.178	
2	Unknown	5	22.387	1245444	44044	8.164	7.324	N/A	13650	N/A	1.078	

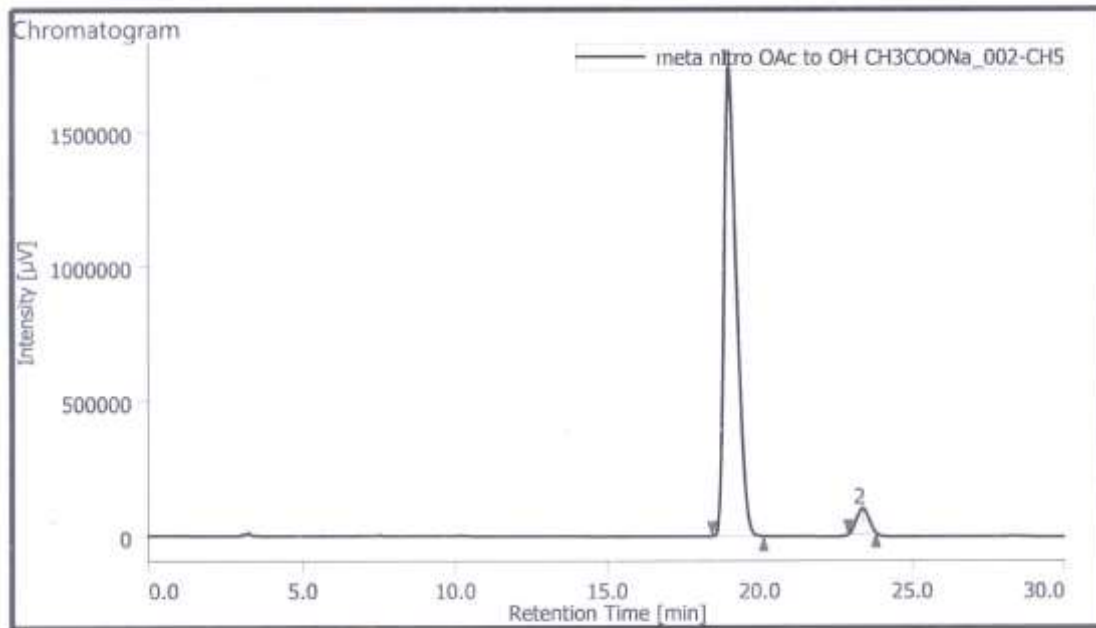
HPLC (R) 1-(3-nitrophenyl)ethan-1-ol(CH<sub>3</sub>COONa/AgNO<sub>3</sub>:1.75/0.25)



**Peak Information**

#	Peak Name	CH	tR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	18.473	7615426	312724	92.575	92.986	N/A	13234	5.479	1.079	
2	Unknown	5	22.197	610796	23587	7.425	7.014	N/A	15189	N/A	1.016	

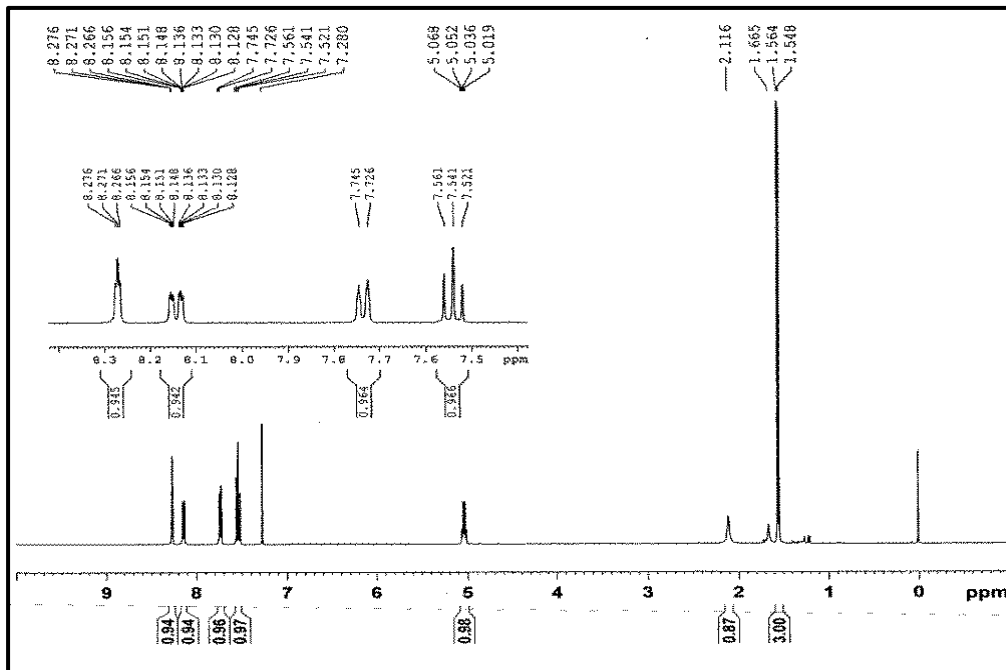
HPLC (R) 1-(3-nitrophenyl)ethan-1-ol(CH3COONa/AgNO3:1.50/0.50)



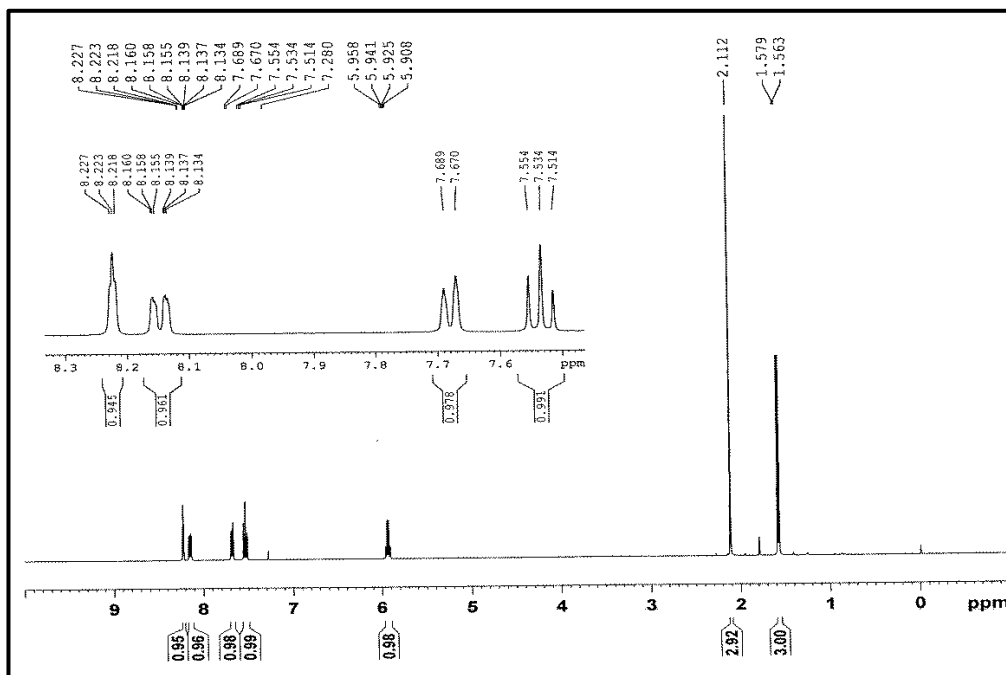
Peak Information

#	Peak Name	CH	tR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	18.973	49362935	1739983	95.074	94.987	N/A	10273	5.764	1.529	
2	Unknown	5	23.357	2557572	91823	4.926	5.013	N/A	14490	N/A	0.977	

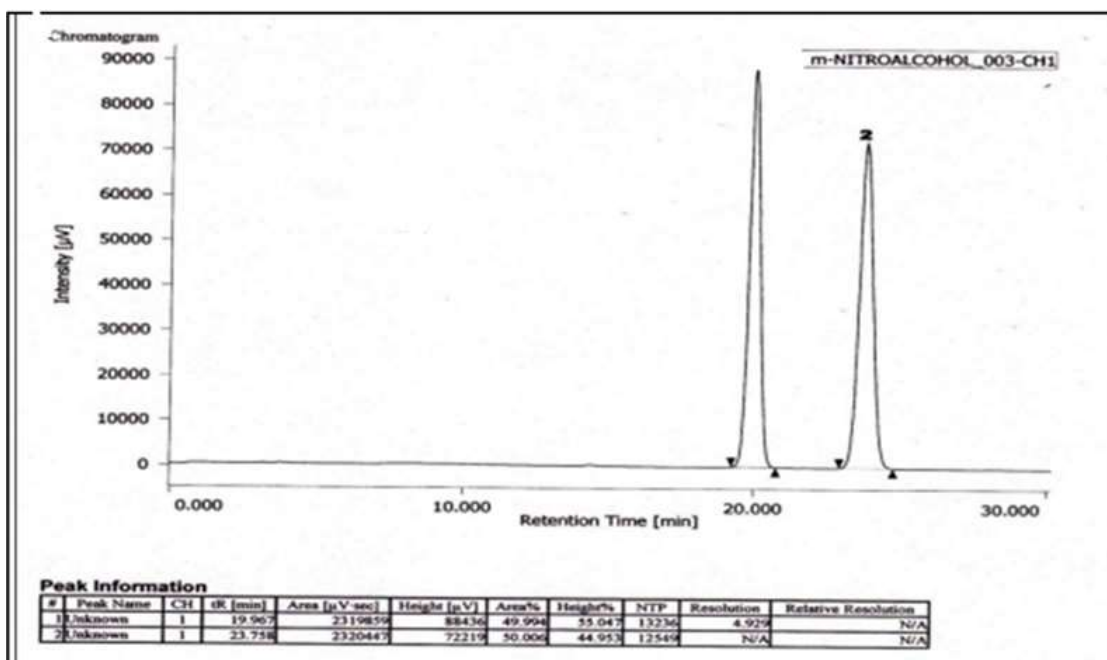
NMR 1-(3-nitrophenyl)ethan-1-ol



NMR 1-(3-nitrophenyl)ethyl acetate

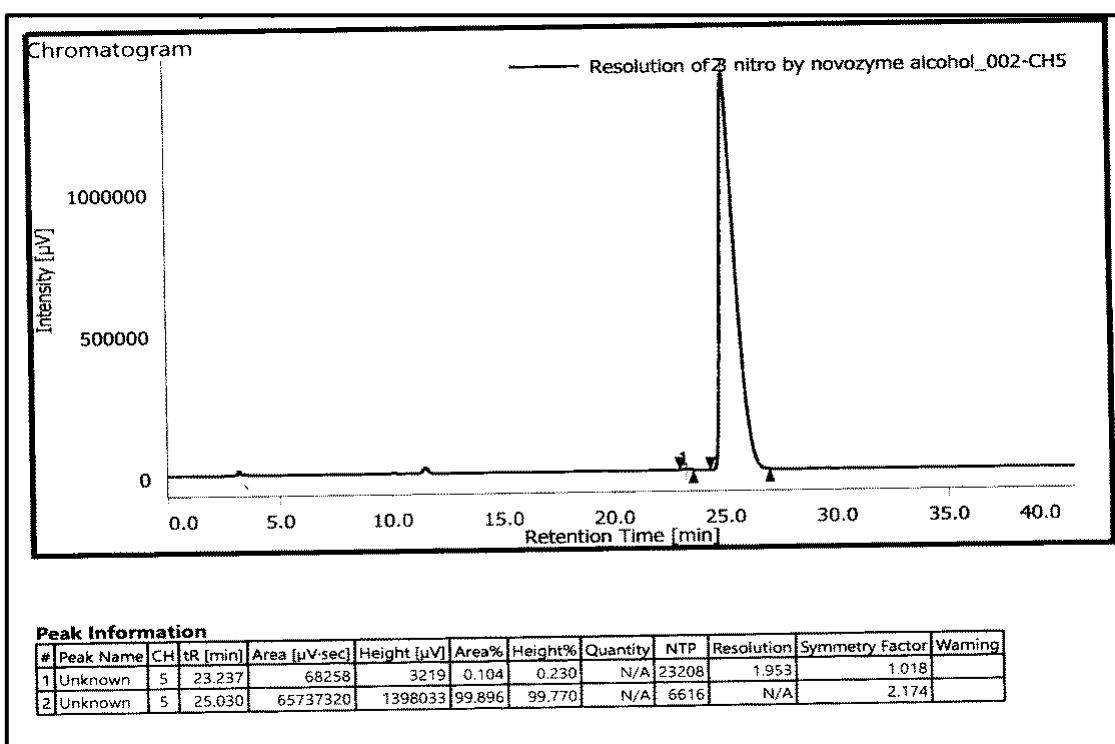


Racemic 1-(3-nitrophenyl)ethan-1-ol

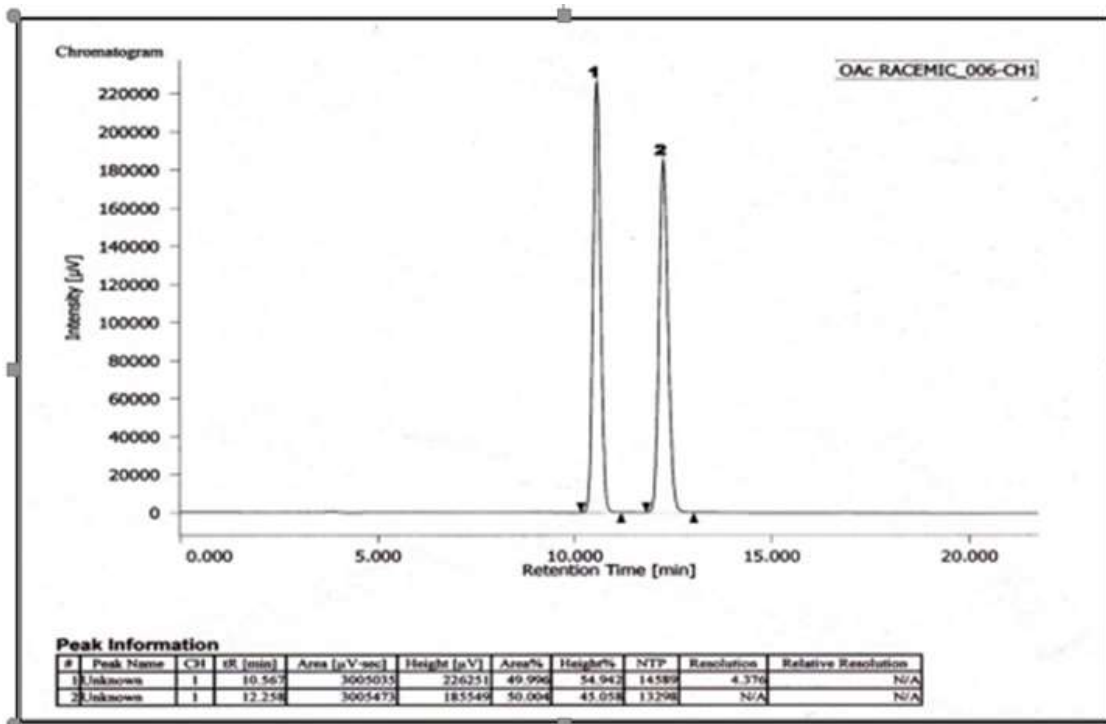


HPLC Condition for compound 1-(3-nitrophenyl)ethan-1-ol<sup>b</sup> Determined by HPLC analysis (chiral column Amylose, 1ml/min IPA (5.0%) in hexane; 18.42 min for (R)-1 and 22.37 min for (S)-1)

(S) 1-(3-nitrophenyl)ethan-1-ol

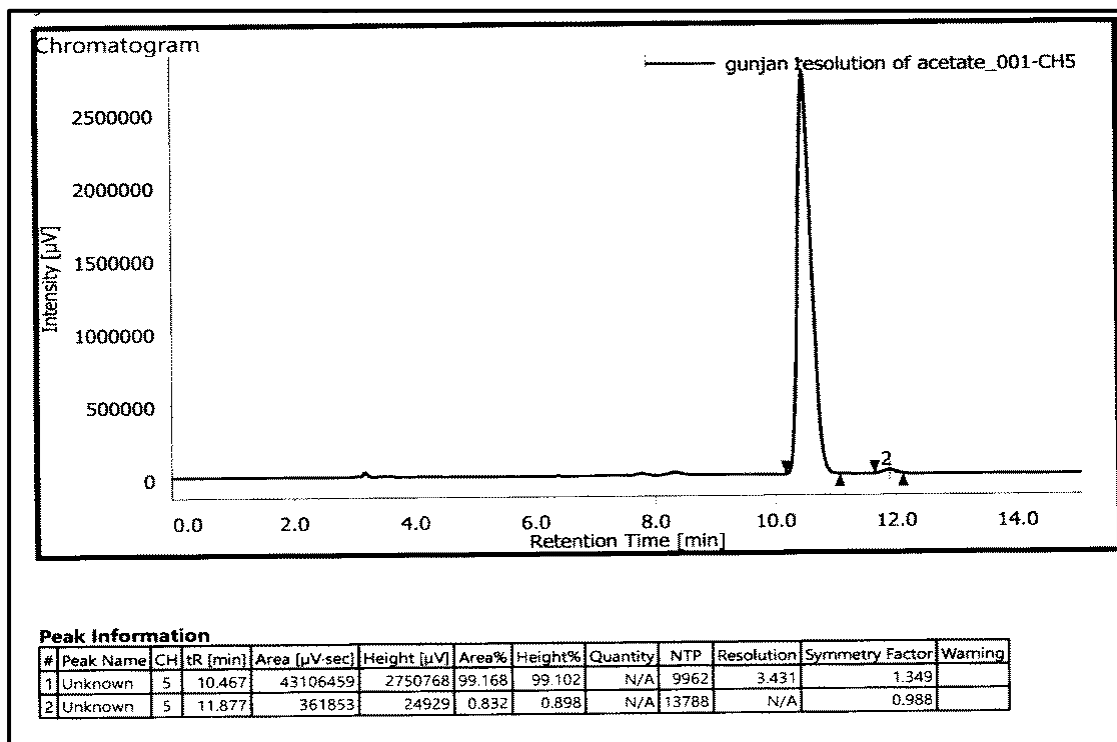


Racemic 1-(3-nitrophenyl)ethyl acetate

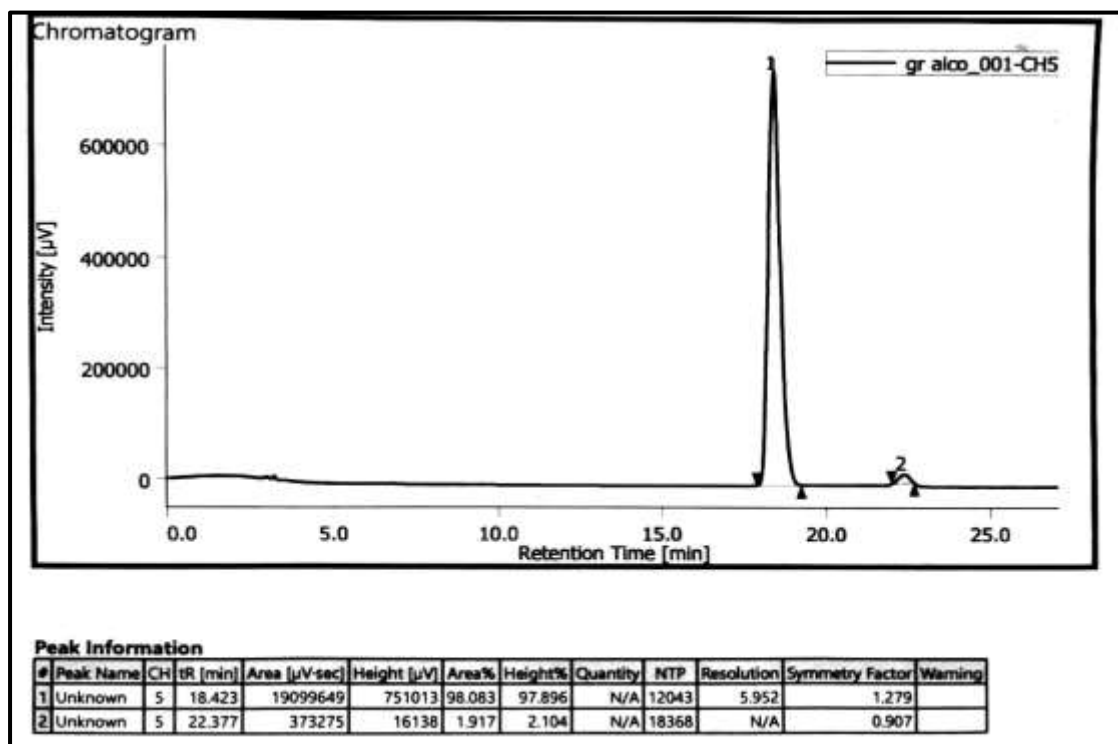


HPLC Condition for compound 1-(3-nitrophenyl)ethyl acetate Determined by HPLC analysis (chiral column Amylose, 1ml/min IPA (5.0%) in hexane; 11.23 min for (*R*)-**2** and 12.67 min for (*S*)-**2**)

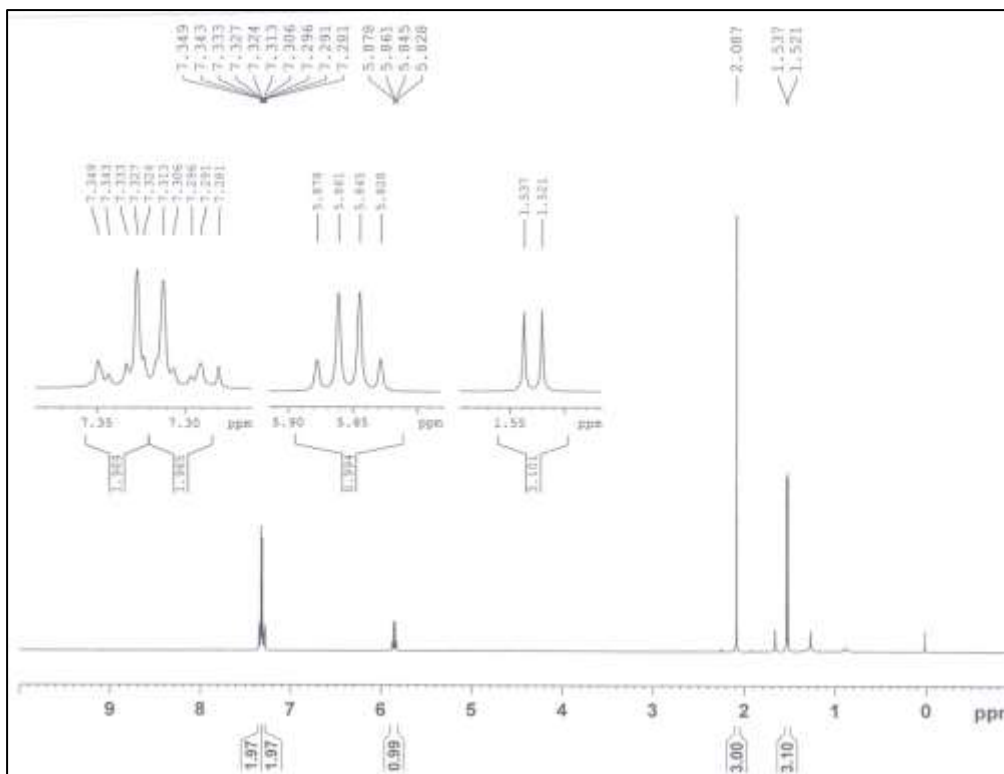
(*R*) 1-(3-nitrophenyl)ethyl acetate



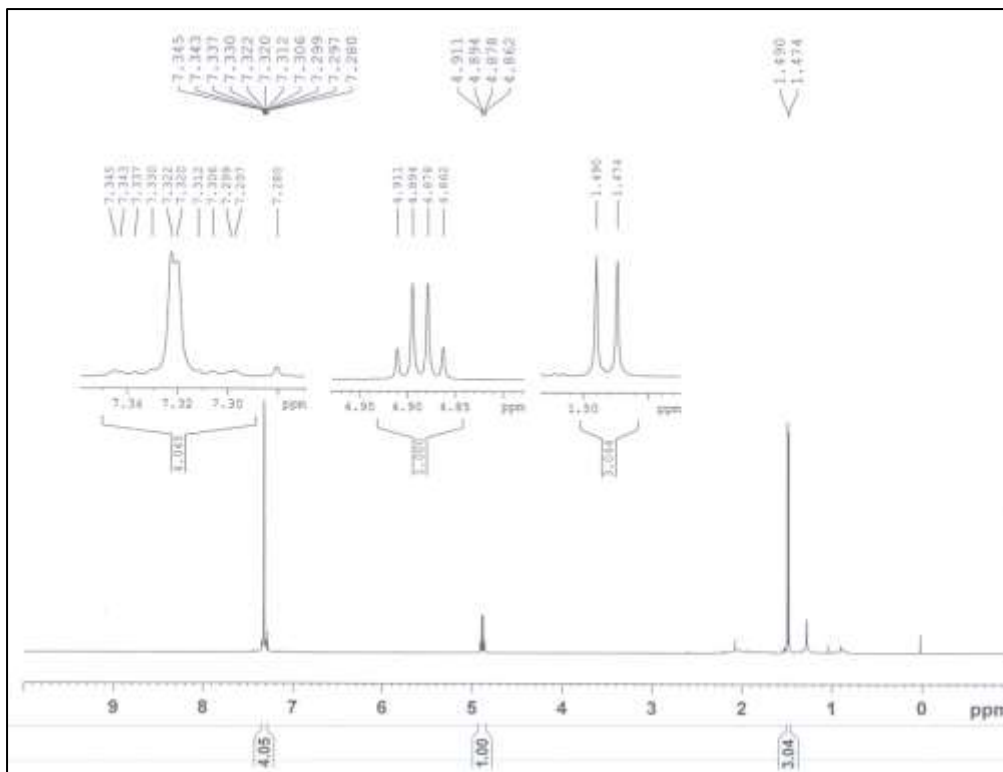
(R) 1-(3-nitrophenyl)ethan-1-ol after mitsunobu



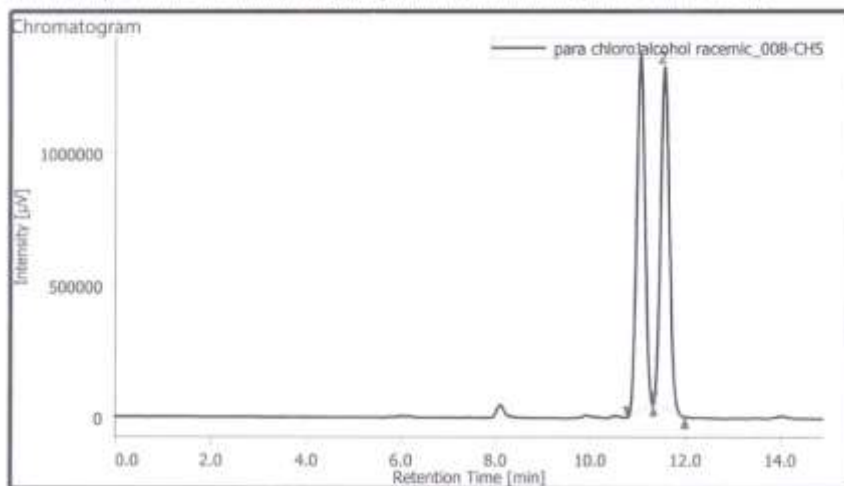
<sup>1</sup>H NMR of 1-(4-chlorophenyl)ethyl acetate



<sup>1</sup>H NMR of 1-(4-chlorophenyl)ethan-1-ol



## HPLC of racemic 1-(4-chlorophenyl)ethan-1-ol

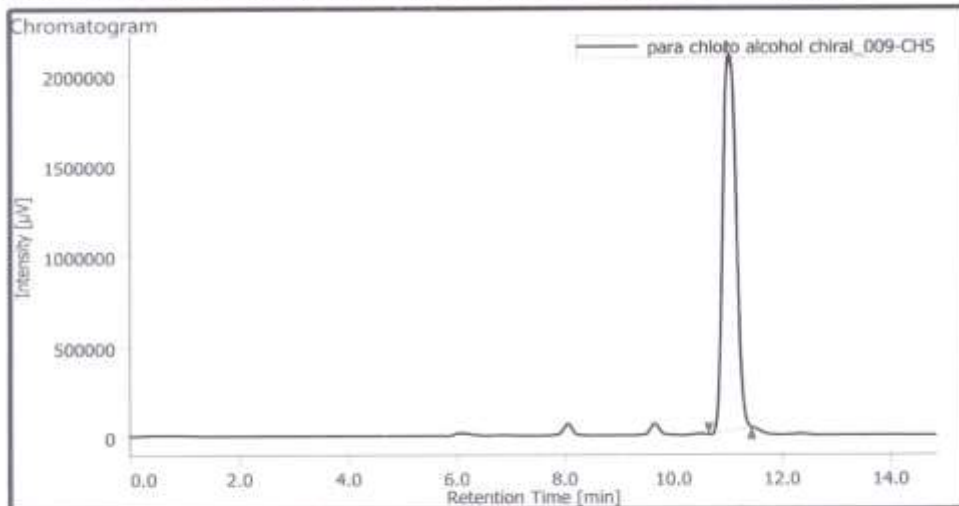


### Peak Information

#	Peak Name	CH	tr [min]	Area [µVsec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	11.050	16235399	1345199	49.632	51.009	N/A	18718	1.521	1.053	
2	Unknown	S	11.553	16476054	1290000	50.368	48.991	N/A	18430	N/A	1.134	

HPLC Condition for compound 1-(4-chlorophenyl)ethan-1-ol Determined by HPLC analysis (chiral column OD-H, 0.5ml/min IPA (10.0%) in hexane 11.05 min for (*R*)-**3** and 11.53 min for (*S*)-**3**)

## HPLC of (*S*) 1-(4-chlorophenyl)ethan-1-ol

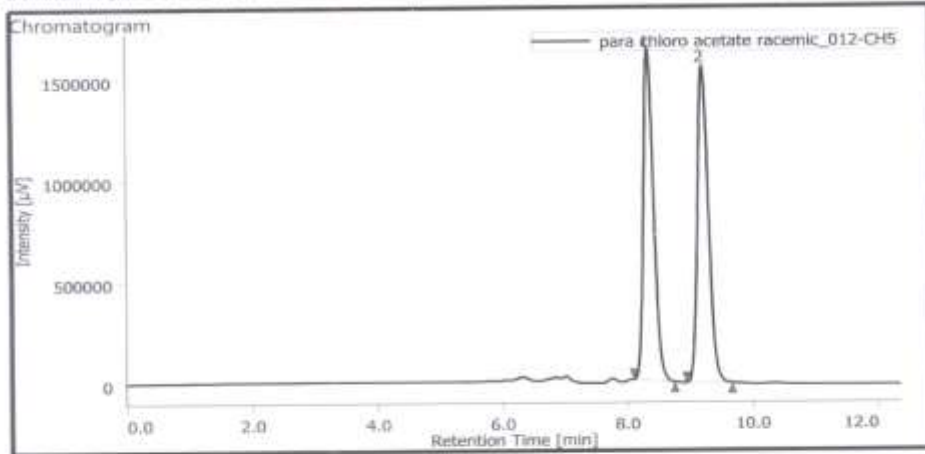


### Peak Information

#	Peak Name	CH	tr [min]	Area [µVsec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	11.030	35469976	2078323	100.000	100.000	N/A	9134	N/A	1.102	



HPLC of Racemic 1-(4-chlorophenyl)ethyl acetate

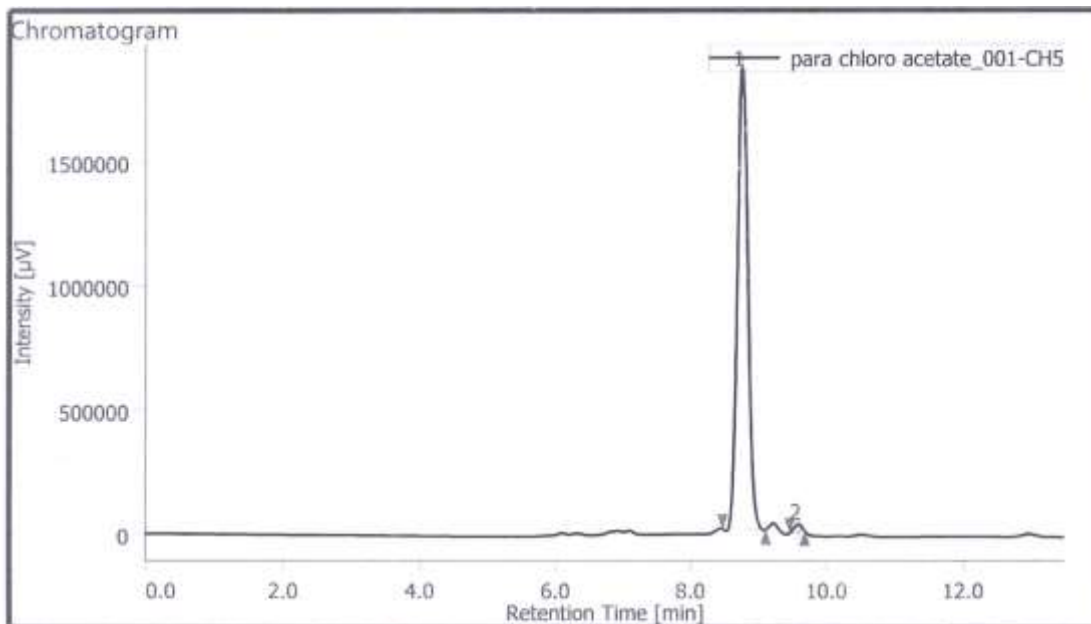


Peak Information

#	Peak Name	CH	TR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	8.343	18488171	1635014	49.324	51.159	N/A	12798	2.857	1.145	
2	Unknown	S	9.210	18995098	1160906	50.676	48.841	N/A	13449	N/A	1.130	

HPLC Condition for compound 1-(4-chlorophenyl)ethyl acetate Determined by HPLC analysis (chiral column Amylose, IPA 0.5ml/min (10.0%) in hexane; 8.34 min for (*R*)-4 and 9.20 min for (*S*)-4)

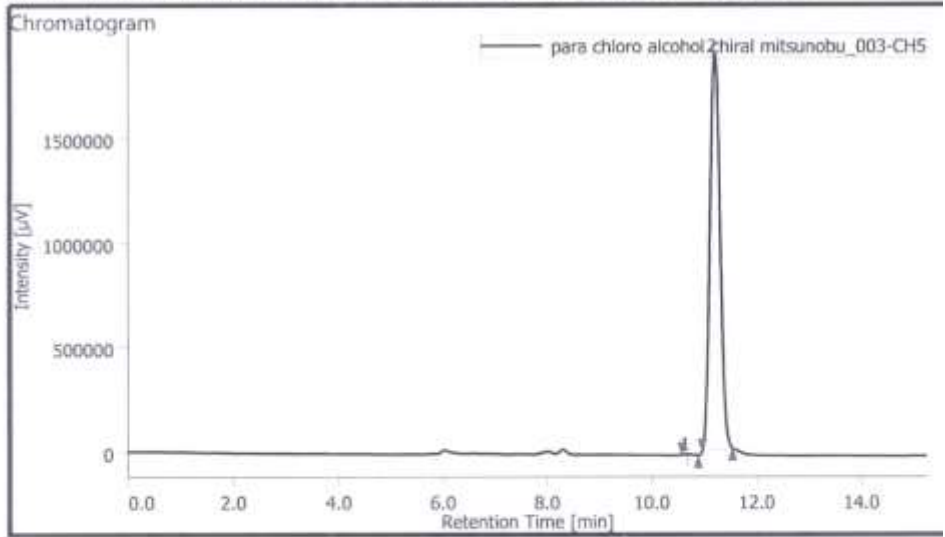
HPLC of (*R*) 1-(4-chlorophenyl)ethyl acetate



Peak Information

#	Peak Name	CH	TR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	8.747	20124635	1859258	98.903	98.433	N/A	14970	3.336	1.178	
2	Unknown	S	9.583	223115	29602	1.097	1.567	N/A	31191	N/A	0.865	

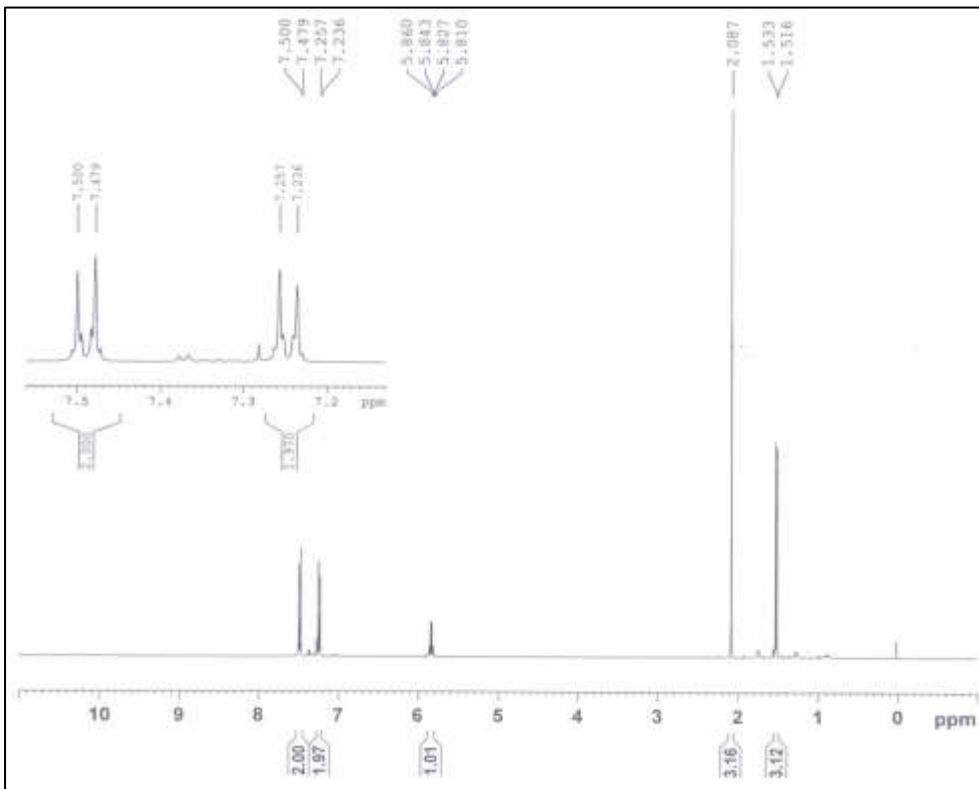
HPLC of (R) 1-(4-chlorophenyl)ethan-1-ol after mitsunobu



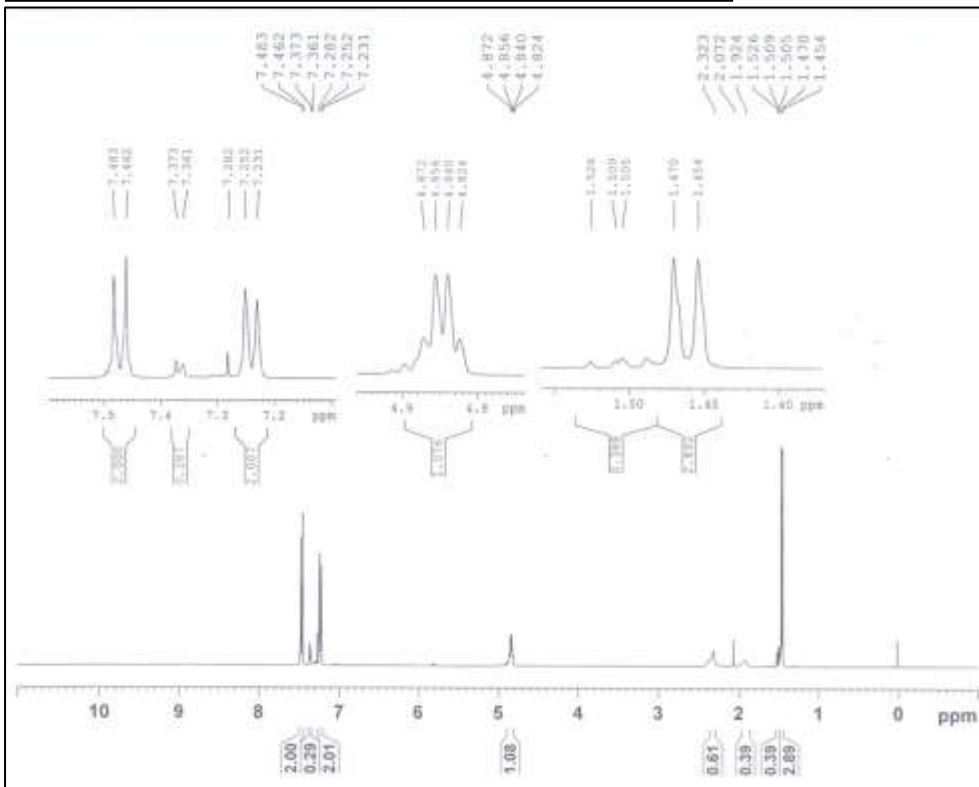
Peak Information

#	Peak Name	CH	RT [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	10.673	28618	3235	0.112	0.171	N/A	28432	1.703	1.201	
2	Unknown	S	11.193	25413159	1892494	99.888	99.829	N/A	15549	N/A	1.114	

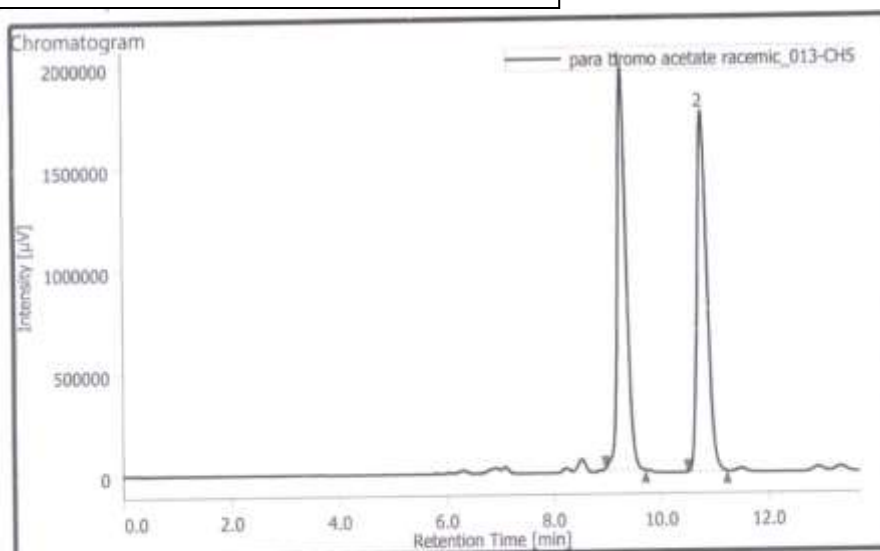
<sup>1</sup>H NMR of 1-(4-bromophenyl)ethan-1-ol



<sup>1</sup>H NMR of 1-(4-bromophenyl)ethyl acetate



### HPLC of 1-(4-bromophenyl)ethyl acetate

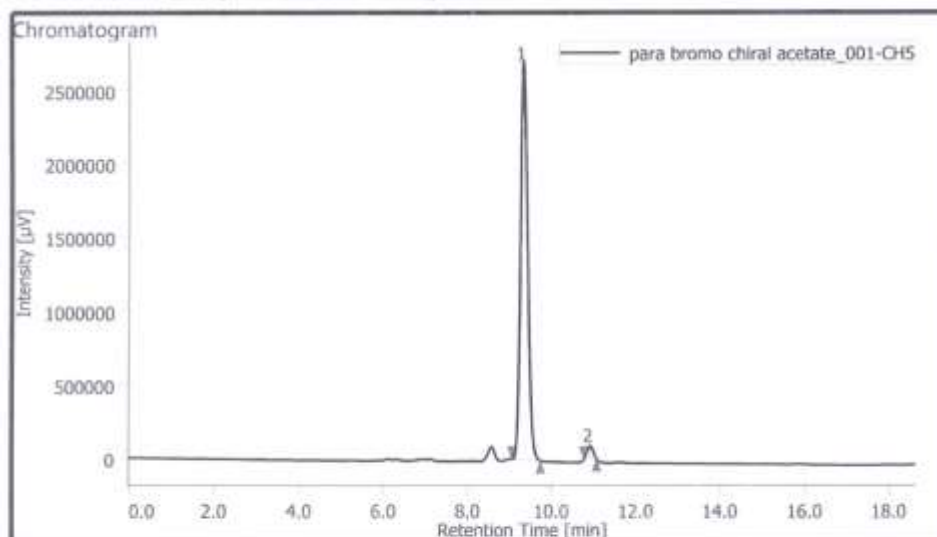


#### Peak Information

#	Peak Name	CH	tR [min]	Area [µV·sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	9.287	22299548	1962149	49.691	52.707	N/A	16590	4.790	1.130	
2	Unknown	S	10.770	32577311	1760591	50.309	47.293	N/A	16773	N/A	1.199	

HPLC Condition for compound 1-(4-bromophenyl)ethyl acetate Determined by HPLC analysis (chiral column Amylose, 0.5ml/min IPA (10.0%) in hexane; 9.28 min for (*R*)-6 and 10.77 min for (*S*)-6

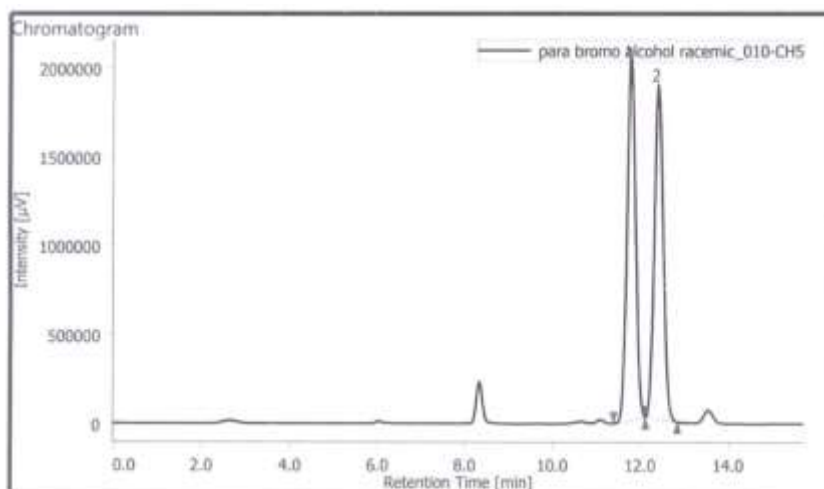
### HPLC of (*R*) 1-(4-bromophenyl)ethyl acetate



#### Peak Information

#	Peak Name	CH	tR [min]	Area [µV·sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	9.330	31956428	2702481	97.366	96.913	N/A	14719	5.363	1.219	
2	Unknown	S	10.920	864578	86085	2.634	3.087	N/A	23173	N/A	0.969	

HPLC of 1-(4-bromophenyl)ethan-1-ol

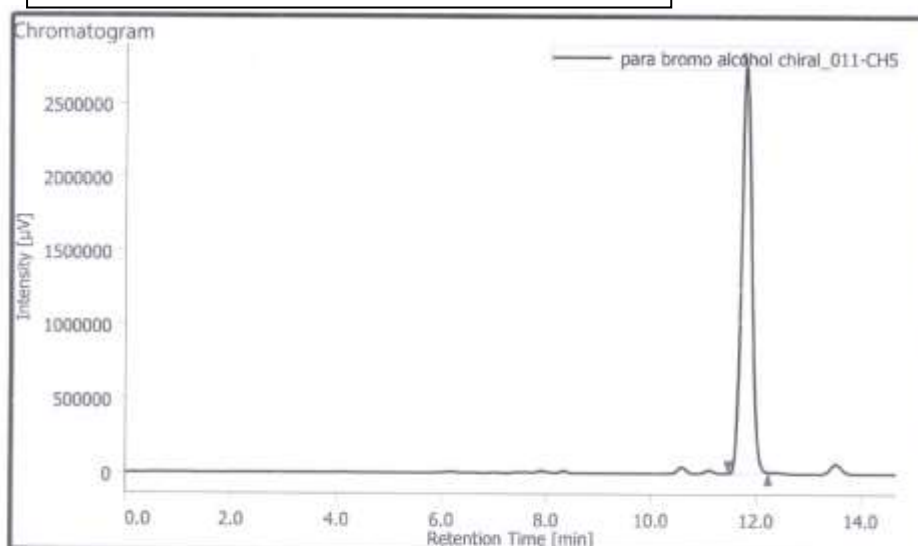


Peak Information

#	Peak Name	CH	tR [min]	Area [µVsec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	11.773	27387943	2030146	50.863	51.978	N/A	17503	1.708	1.083	
2	Unknown	S	12.393	26458181	1875631	49.137	48.022	N/A	17748	N/A	1.121	

HPLC Condition for compound 1-(4-bromophenyl)ethan-1-ol Determined by HPLC analysis (chiral column OD-H, 0.5ml/min IPA (10.0%) in hexane; 11.77 min for (*R*)-**5** and 12.39 min for (*S*)-**5**)

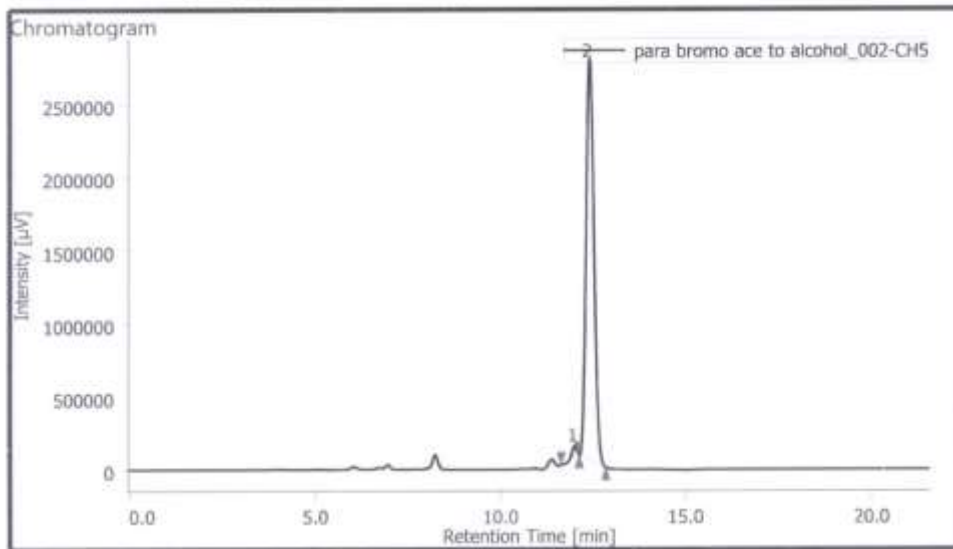
HPLC of (*S*) 1-(4-bromophenyl)ethan-1-ol



Peak Information

#	Peak Name	CH	tR [min]	Area [µVsec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	11.783	36958061	2744524	100.000	100.000	N/A	17760	N/A	1.106	

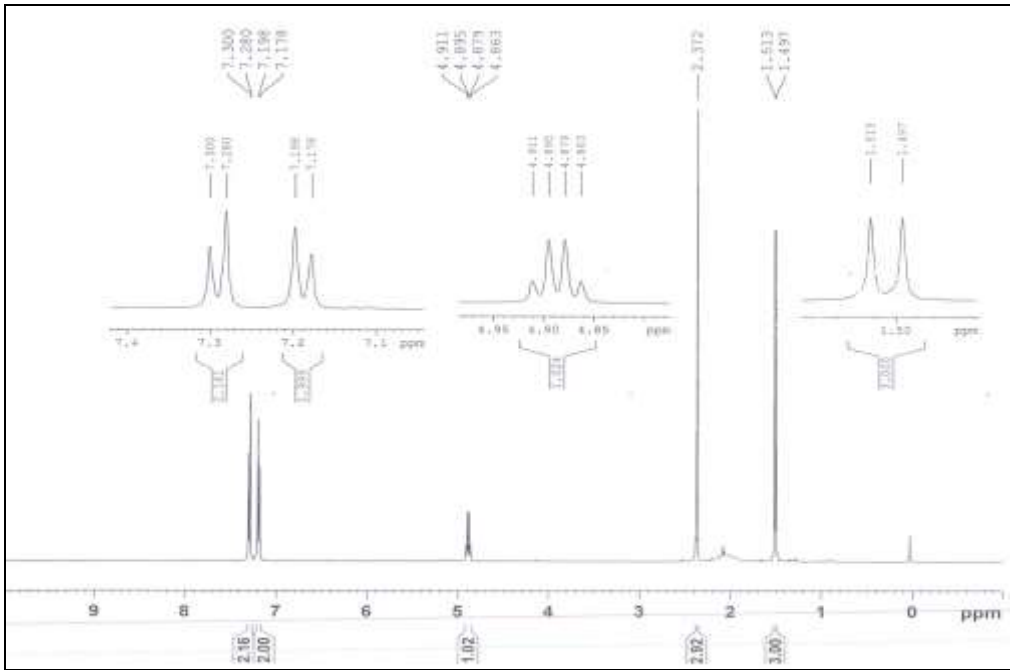
HPLC of (*R*) 1-(4-bromophenyl)ethan-1-ol after mitsunobu



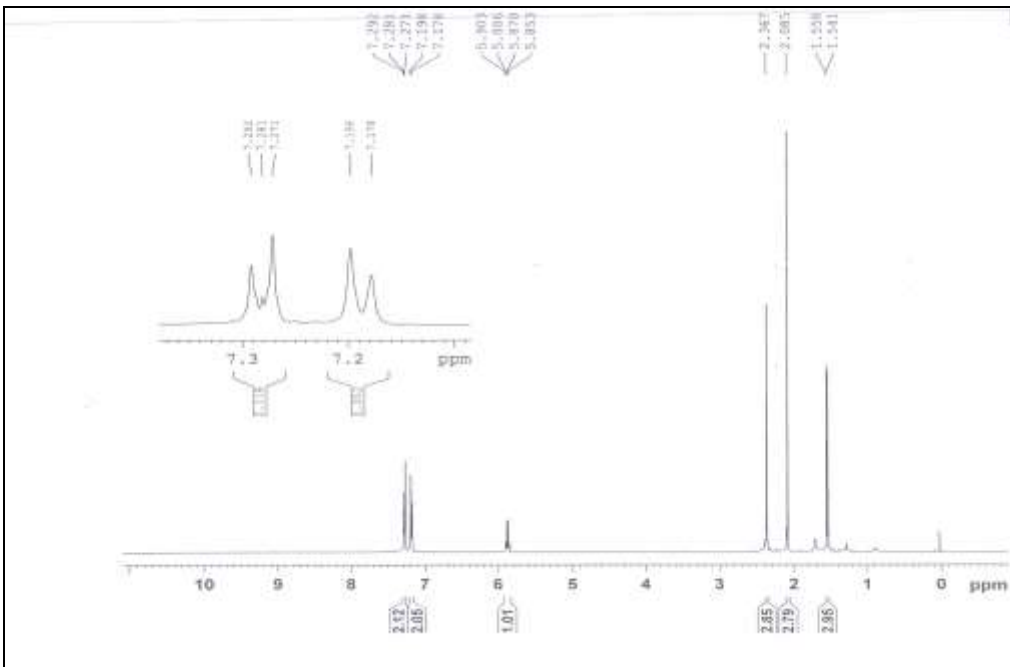
Peak Information

#	Peak Name	CH	tR [min]	Area [µV·sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	12.007	763814	81596	1.843	2.869	N/A	32896	1.215	0.838	
2	Unknown	5	12.407	40691051	2742474	98.157	97.111	N/A	15756	N/A	1.157	

<sup>1</sup>H NMR of 1-(p-tolyl)ethan-1-ol

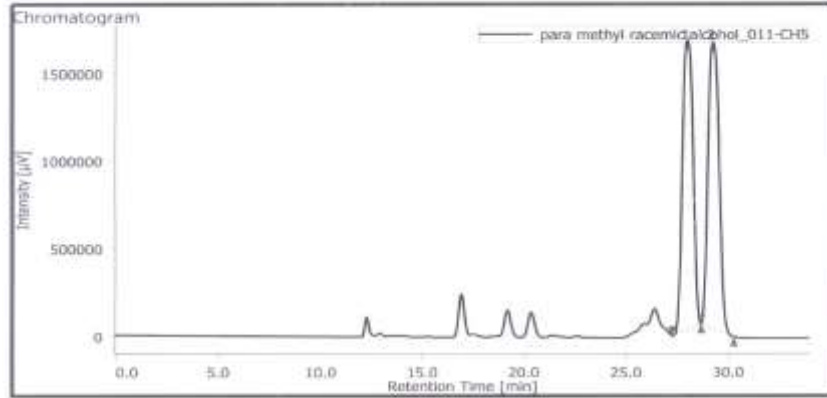


<sup>1</sup>H NMR of 1-(p-tolyl)ethyl acetate



## HPLC of Racemic 1-(p-tolyl)ethan-1-ol

para methyl and para bromo, 0701 para methyl racemic alcohol, 011 01-07-2019 16:15:01



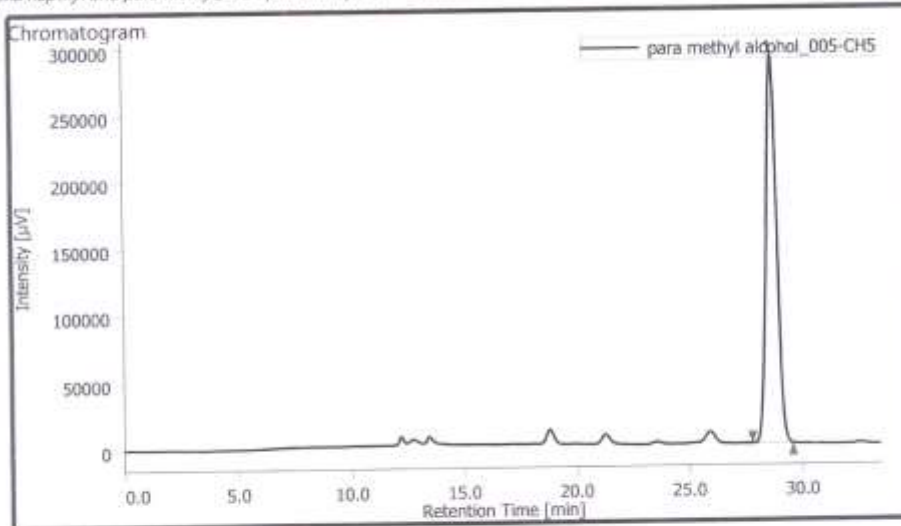
### Peak Information

#	Peak Name	CH	TR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	27.987	33324754	1544421	45.070	50.254	N/A	12628	1.128	1.044	
2	Unknown	S	29.233	61573086	1627809	50.930	49.746	N/A	12685	N/A	1.126	

HPLC Condition for compound 1-(p-tolyl)ethan-1-ol Determined by HPLC analysis (chiral column IE, 0.25ml/min IPA (5.0%) in hexane; 27.98 min for (R)-7 and 29.23 min for (S)-7)

## HPLC (S) 1-(p-tolyl)ethan-1-ol

alpha naphthyl and para methyl, 0724 para methyl alcohol, 005 24-07-2019 17:30:28

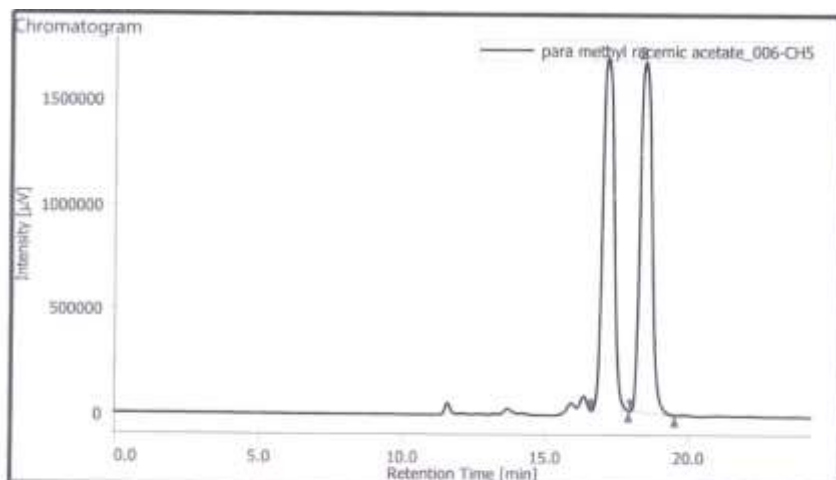


### Peak Information

#	Peak Name	CH	TR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	28.663	9456494	290024	100.000	100.000	N/A	17621	N/A	1.052	



### HPLC of Racemic 1-(p-tolyl)ethyl acetate

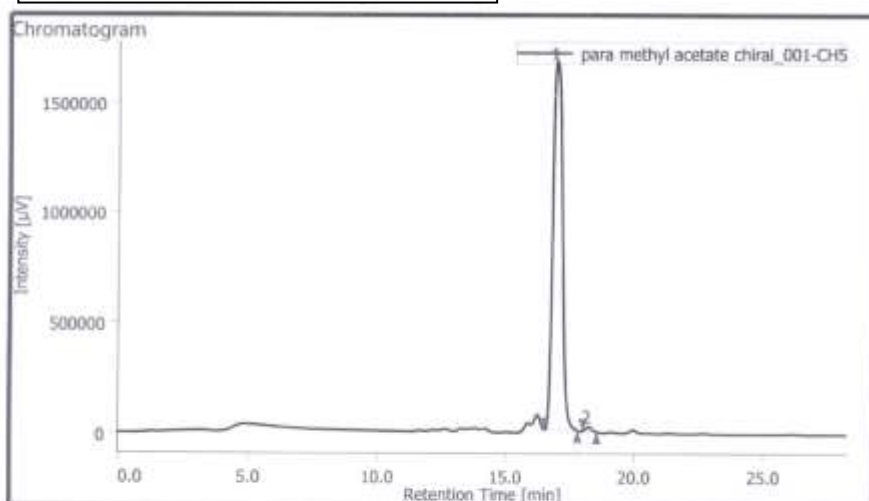


#### Peak Information

#	Peak Name	CH	RT [min]	Area [uV-sec]	Height [uV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	17.120	48375292	1481123	48.727	50.165	N/A	8572	1.751	1.172	
2	Unknown	S	18.423	46884083	1670066	50.273	49.835	N/A	9581	N/A	1.346	

HPLC Condition for compound 1-(p-tolyl)ethyl acetate Determined by HPLC analysis (chiral column Amylose, 1ml/min IPA (20.0%) in hexane; 4.003 min for (*R*)-**8** and 4.25 min for (*S*)-**8**)

### HPLC of (*R*) 1-(p-tolyl)ethyl acetate

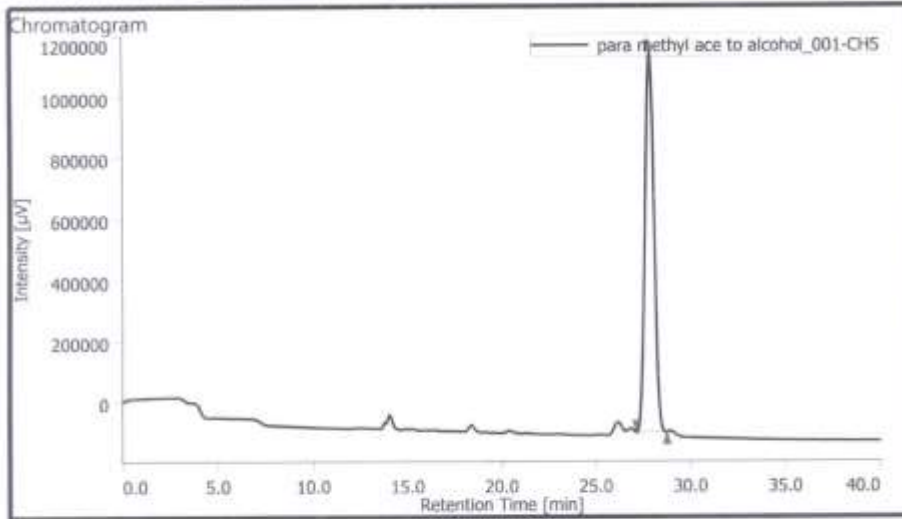


#### Peak Information

#	Peak Name	CH	RT [min]	Area [uV-sec]	Height [uV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	16.980	43121062	1671463	99.175	98.761	N/A	9534	2.136	1.161	
2	Unknown	S	18.233	358680	20979	0.825	1.239	N/A	22993	N/A	1.153	

# HPLC (R) 1-(p-tolyl)ethan-1-ol after misunobu

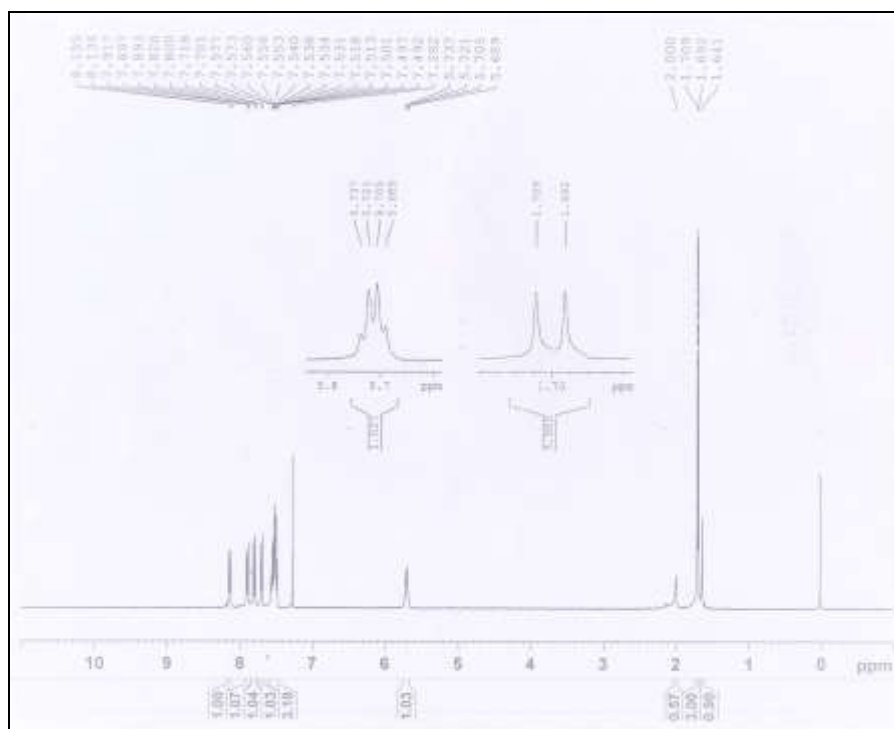
para methyl ace to alcohol\_0913 para methyl ace to alcohol\_001 13-09-2019 10:30:19



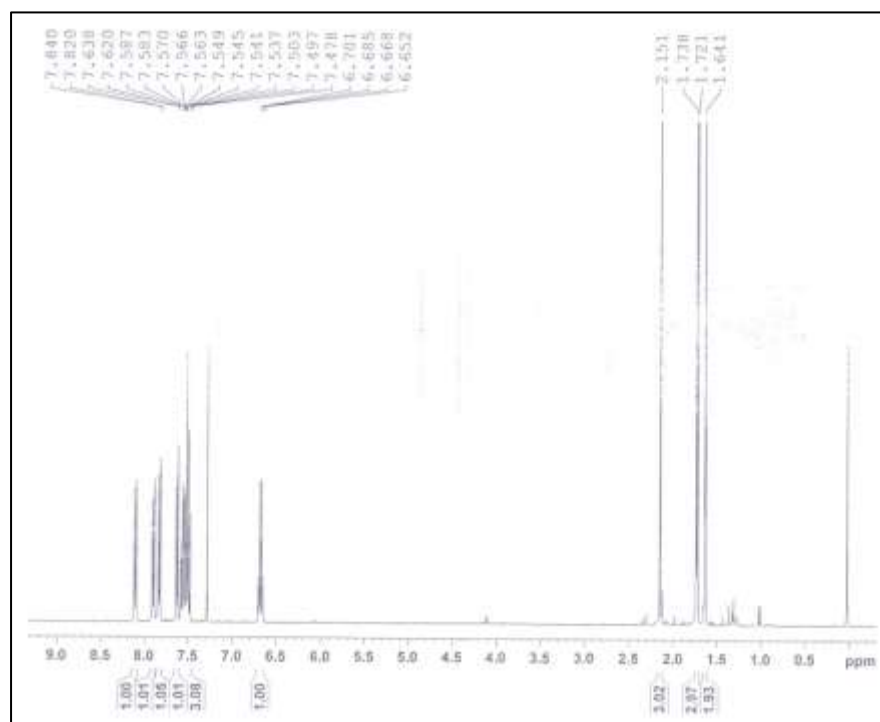
## Peak Information

#	Peak Name	CH	IR [min]	Area [µVsec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	27.840	41456791	1241815	100.000	100.000	N/A	15297	N/A	1.059	

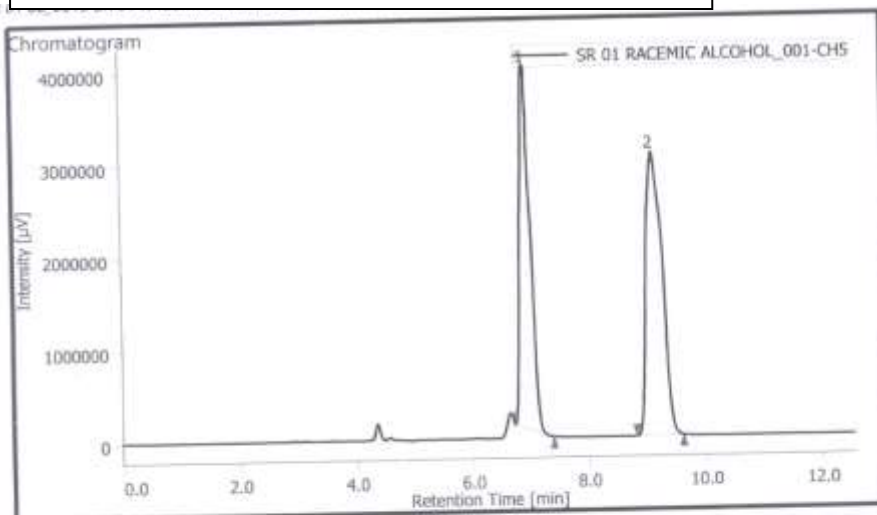
$^1\text{H}$  NMR of 1-(naphthalen-1-yl)ethan-1-ol



$^1\text{H}$  NMR of 1-(naphthalen-1-yl)ethyl acetate



### HPLC of Racemic 1-(naphthalen-1-yl)ethan-1-ol

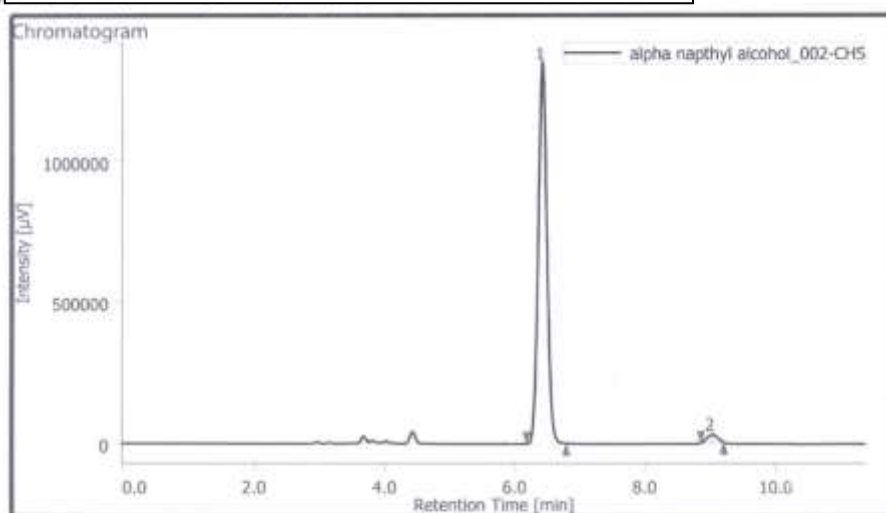


#### Peak Information

#	Peak Name	Ch	IR [min]	Area [µV·sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	6.913	48517814	3933567	45.422	56.202	N/A	5853	4.875	1.273	
2	Unknown	5	9.113	58298850	3063352	54.578	43.798	N/A	4348	N/A	1.254	

HPLC Condition for compound 1-(naphthalen-1-yl)ethan-1-ol Determined by HPLC analysis (chiral column OD-H, 1ml/min IPA (20.0%) in hexane; 6.91 min for (*R*)-**9** and 9.11 min for (*S*)-**9**)

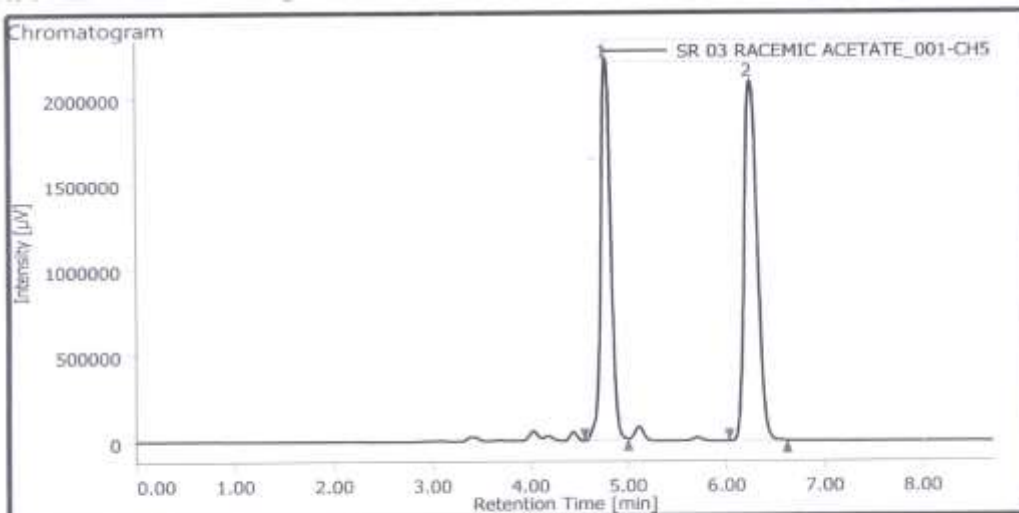
### HPLC of (*S*) 1-(naphthalen-1-yl)ethan-1-ol



#### Peak Information

#	Peak Name	Ch	IR [min]	Area [µV·sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	6.420	12472895	1351921	97.317	97.979	N/A	11480	9.311	1.106	
2	Unknown	5	9.023	317575	27860	2.483	2.021	N/A	12657	N/A	1.006	

### HPLC of Racemic 1-(naphthalen-1-yl)ethyl acetate

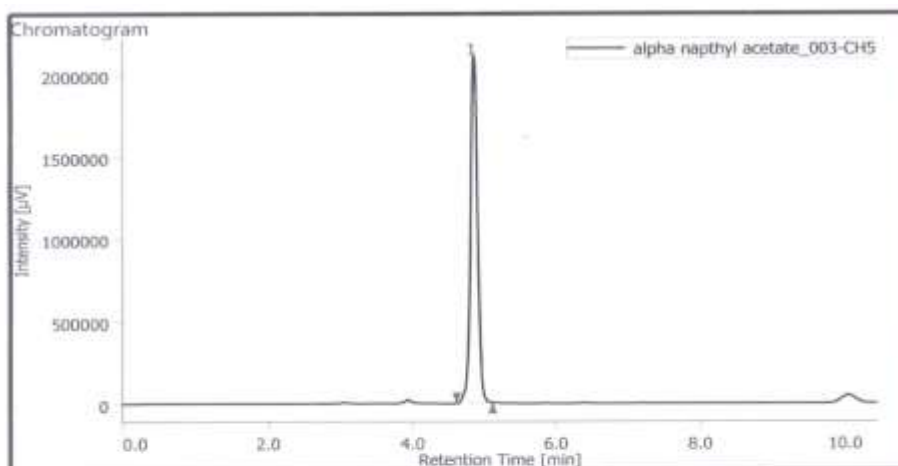


#### Peak Information

#	Peak Name	CH	TR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	4.773	15687702	2212312	44.953	51.362	N/A	11397	7.040	1.040	
2	Unknown	S	6.250	19210335	2094995	55.047	48.638	N/A	10692	N/A	1.144	

HPLC Condition for compound 1-(naphthalen-1-yl)ethyl acetate Determined by HPLC analysis (chiral column OD-H, 1ml/min IPA (10.0%) in hexane; 4.77 min for (*R*)-**10** and 6.20 min for (*S*)-**10**)

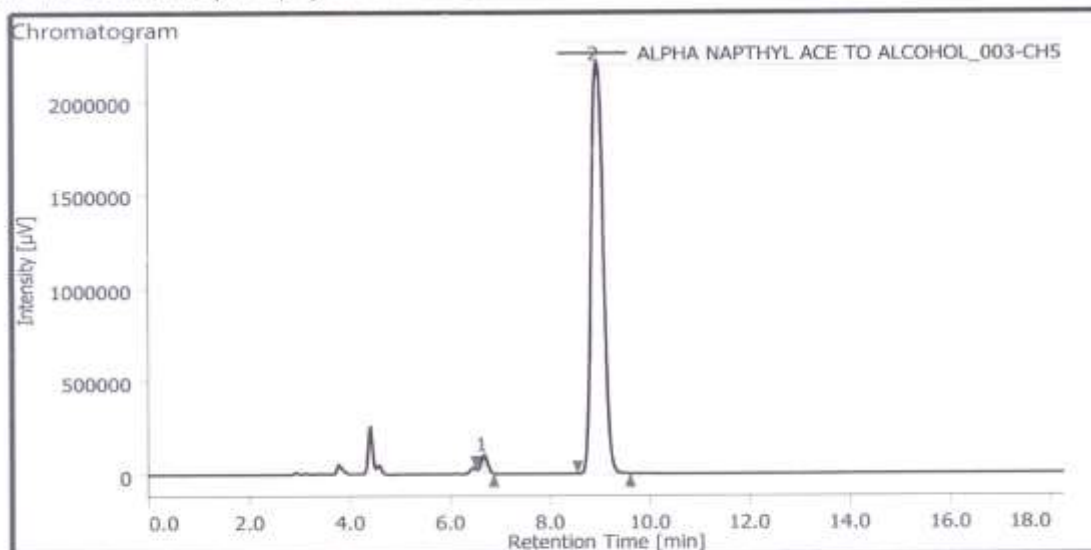
### HPLC of (*R*) 1-(naphthalen-1-yl)ethyl acetate



#### Peak Information

#	Peak Name	CH	TR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	4.833	14607551	2106189	100.000	100.000	N/A	13399	N/A	1.031	

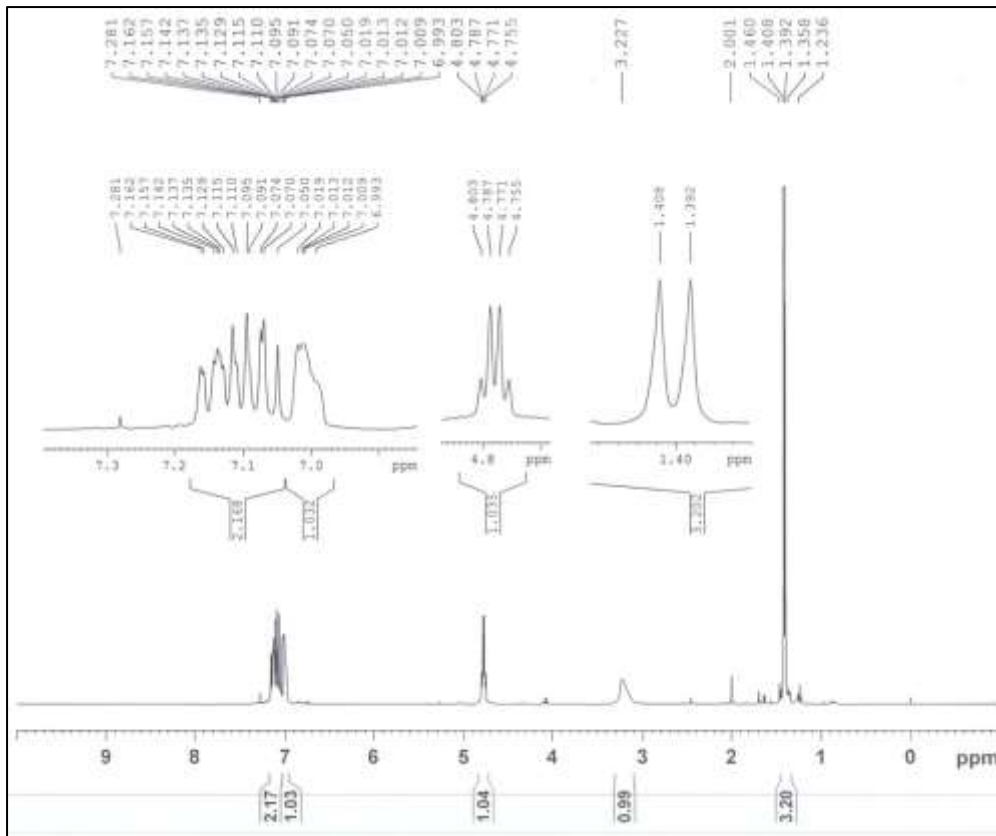
HPLC of (R) 1-(naphthalen-1-yl)ethan-1-ol after mitsunobu



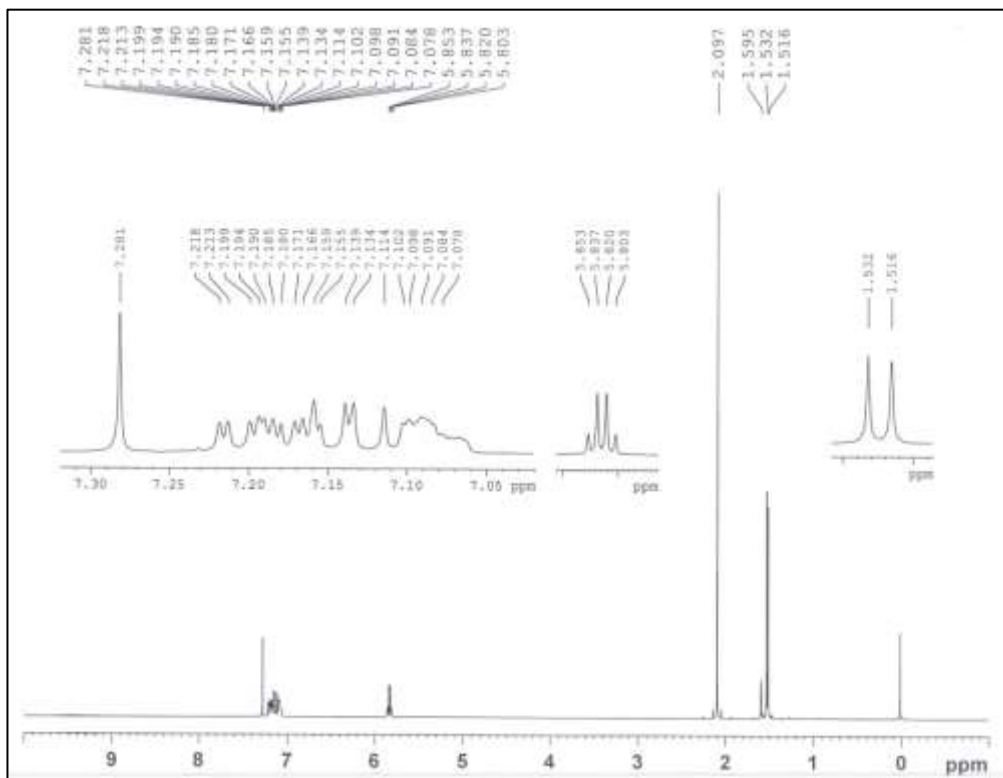
Peak Information

#	Peak Name	Ch	tR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	6.677	746834	86271	1.985	3.769	N/A	12734	6.620	1.144	
2	Unknown	5	8.943	36884906	2202476	98.015	96.231	N/A	6322	N/A	1.164	

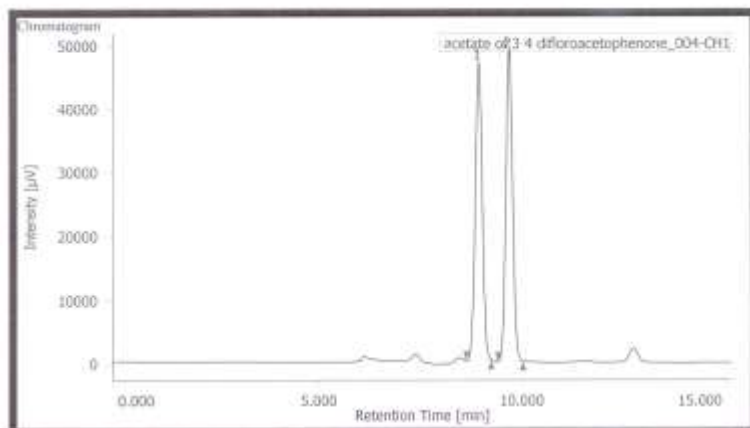
$^1\text{H}$  NMR of 1-(3,4-difluorophenyl)ethan-1-ol



$^1\text{H}$  NMR of 1-(3,4-difluorophenyl)ethyl acetate



## HPLC of Racemic 1-(3,4-difluorophenyl)ethan-1-ol

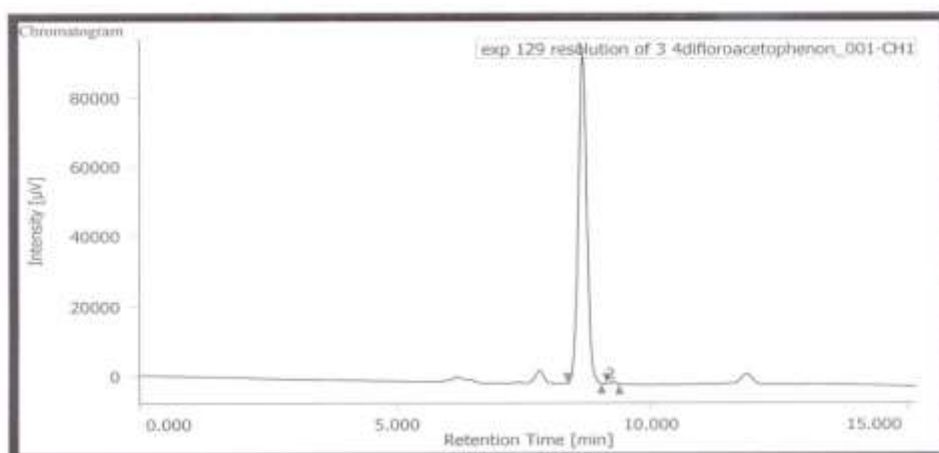


### Peak Information

#	Peak Name	CH	RT [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	1	8.956	560730	46842	49.865	48.874	N/A	13899	1.411	0.072	
2	Unknown	1	9.602	561732	49004	58.135	51.125	N/A	16219	N/A	1.054	

HPLC Condition for compound 1-(3,4-difluorophenyl)ethan-1-ol Determined by HPLC analysis (chiral column Amylose, 0.25ml/min IPA (7.0%) in hexane; 8.9 min for (*R*)-**11** and 9.6 min for (*S*)-**11**)

## HPLC of (*S*) 1-(3,4-difluorophenyl)ethan-1-ol

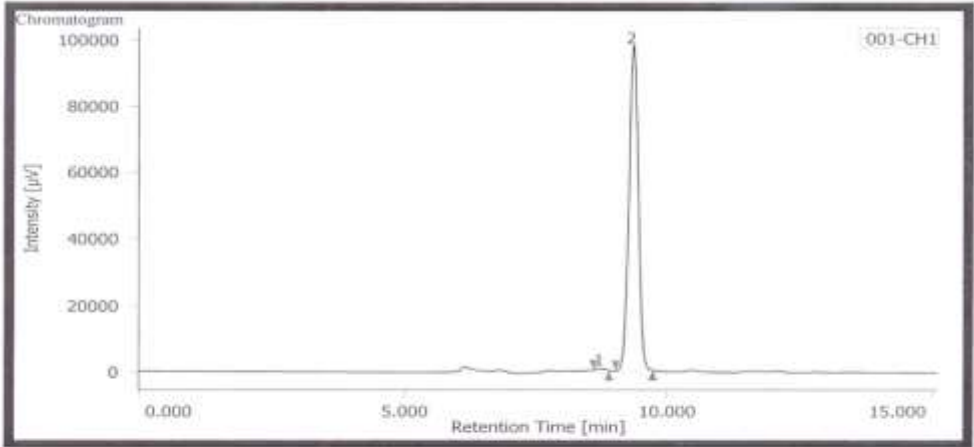


### Peak Information

#	Peak Name	CH	RT [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	1	8.658	1090906	84074	99.644	99.508	N/A	13183	1.184	1.005	
2	Unknown	1	9.250	3931	465	0.354	0.492	N/A	23520	N/A	1.004	



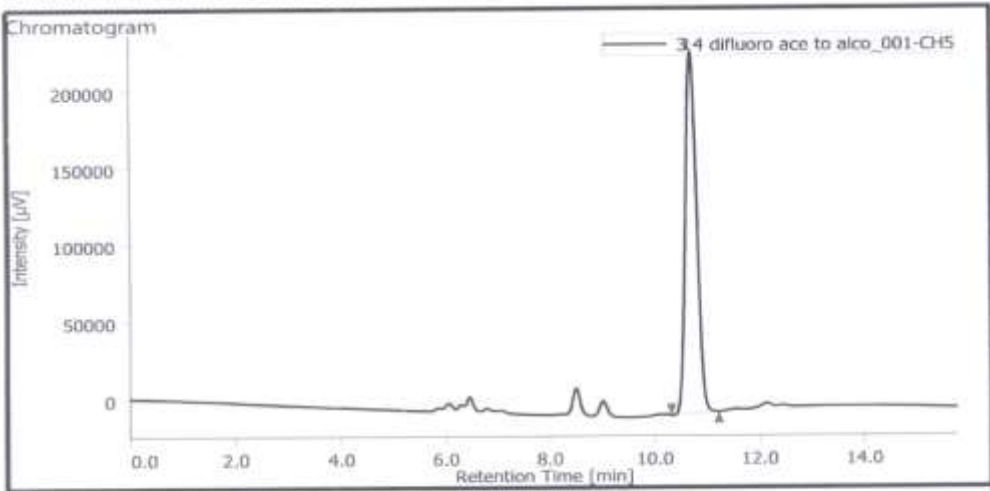
HPLC of (R) 1-(3,4-difluorophenyl)ethan-1-ol



**Peak Information**

#	Peak Name	CH	RT [min]	Area [µV·sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	1	8.756	5133	531	0.426	0.529	N/A	16203	2.018	0.970	
2	Unknown	1	9.358	1216705	98037	99.588	99.461	N/A	12806	N/A	1.017	

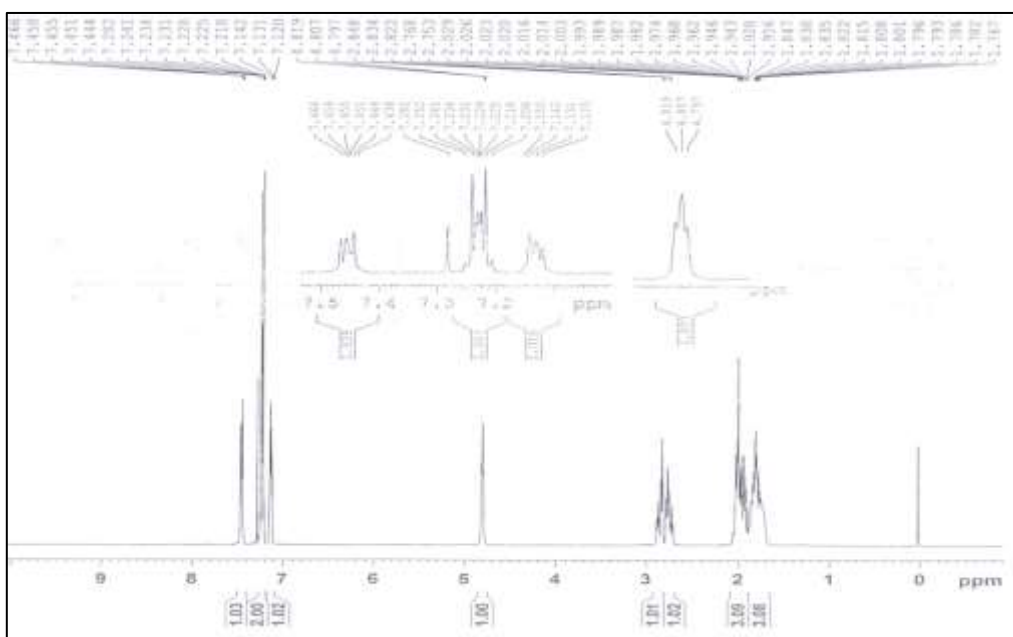
HPLC of (R) 1-(3,4-difluorophenyl)ethan-1-ol after mitsunobu



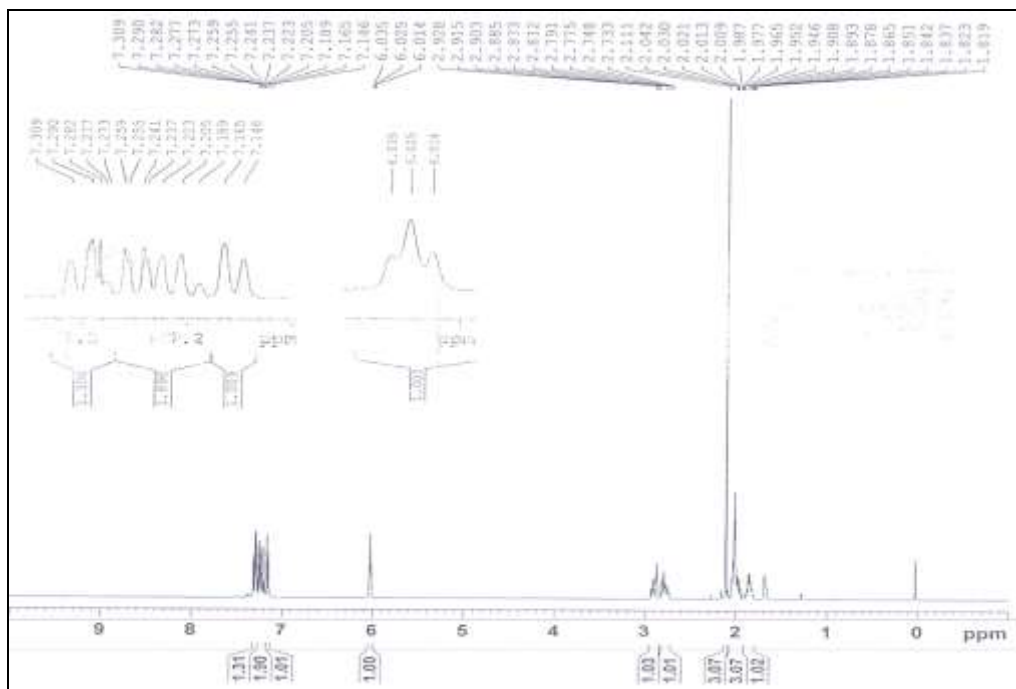
**Peak Information**

#	Peak Name	CH	RT [min]	Area [µV·sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	10.700	3515098	234164	100.000	100.000	N/A	11219	N/A	1.090	

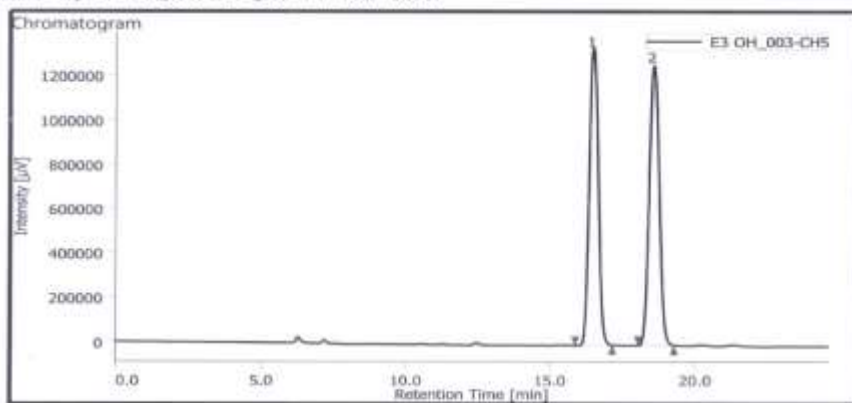
<sup>1</sup>H NMR of 1,2,3,4-tetrahydronaphthalen-1-ol



<sup>1</sup>H NMR of 1,2,3,4-tetrahydronaphthalen-1-yl acetate



### HPLC of Racemic 1,2,3,4-tetrahydronaphthalen-1-ol

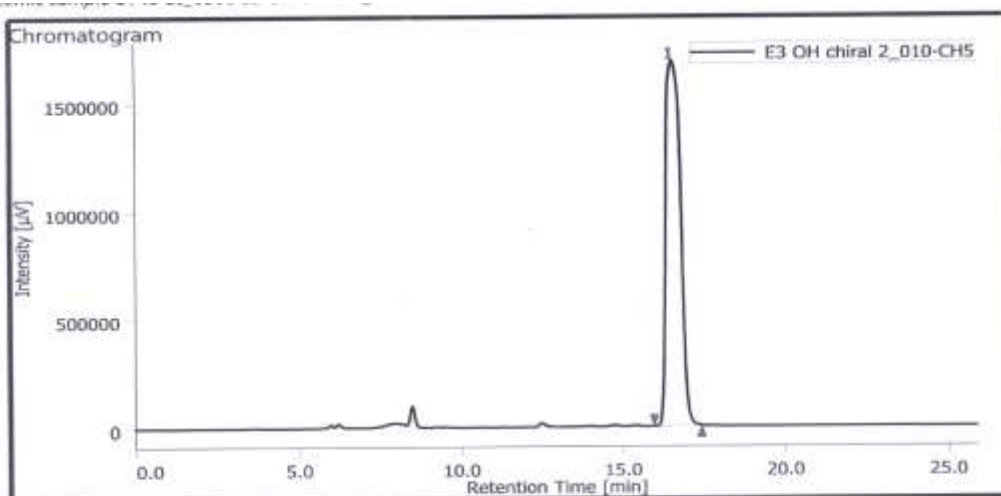


#### Peak Information

#	Peak Name	CH	TR [min]	Area [µV.sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	16.477	29187139	1337870	49.109	51.513	N/A	12594	3.369	1.126	
2	Unknown	5	18.560	30247365	1299270	50.892	48.487	N/A	13234	N/A	1.116	

HPLC Condition for compound 1,2,3,4-tetrahydronaphthalen-1-ol Determined by HPLC analysis (chiral column OD-H,0.5ml/ml IPA (5.0%) in hexane; 16.47 min for (*R*)-**15** and 18.56 min for (*S*)-**15**)

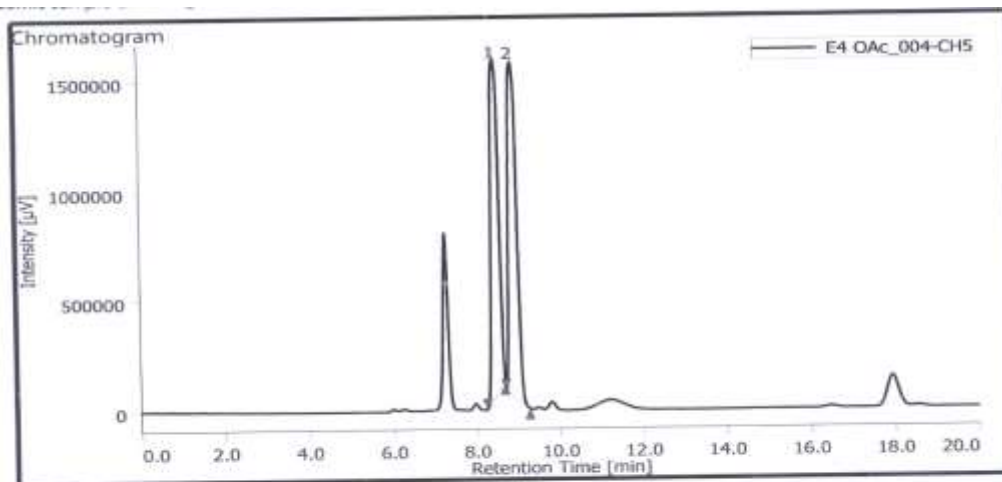
### HPLC of (*S*) 1,2,3,4-tetrahydronaphthalen-1-ol



#### Peak Information

#	Peak Name	CH	TR [min]	Area [µV.sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry factor	Warning
1	Unknown	5	16.530	52695577	1684081	100.000	100.000	N/A	5789	N/A	1.248	

## HPLC of Racemic 1,2,3,4-tetrahydronaphthalen-1-yl acetate

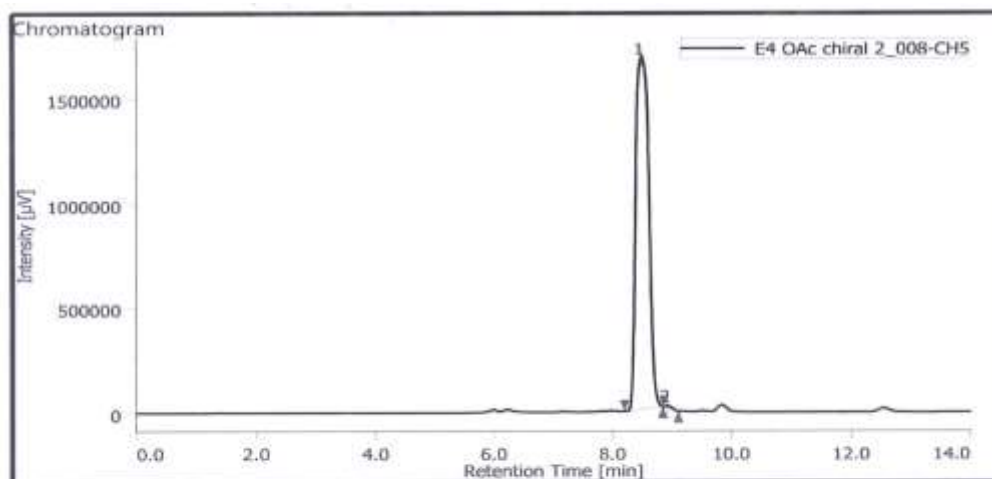


### Peak Information

#	Peak Name	CH	tr [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	8.470	19994974	1528313	48.835	50.205	N/A	8652	1.139	1.050	
2	Unknown	5	8.893	20948855	1515857	51.165	49.795	N/A	8712	N/A	1.157	

HPLC Condition for compound 1,2,3,4-tetrahydronaphthalen-1-yl acetate<sup>b</sup> Determined by HPLC analysis (chiral column Amylose 0.5ml/min, IPA (5.0%) in hexane; 8.47 min for (*R*)-**16** and 8.89 min for (*S*)-**16**)

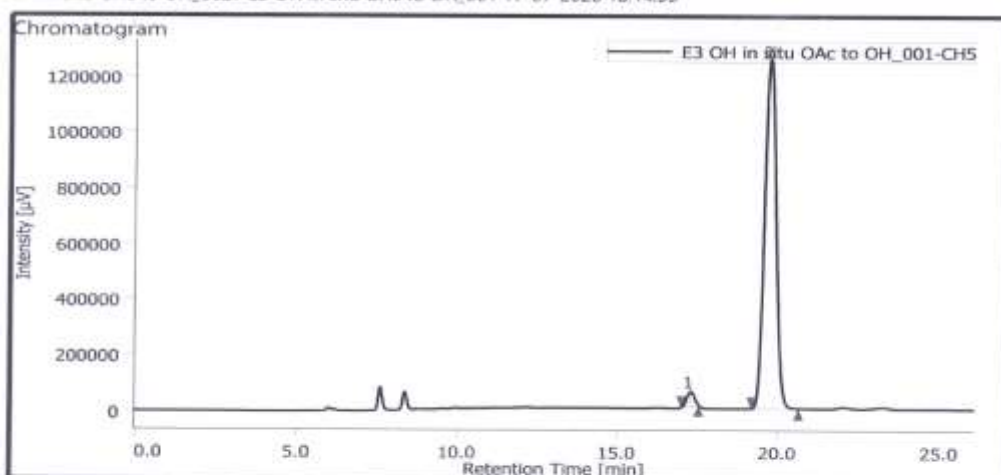
## HPLC of (*R*) 1,2,3,4-tetrahydronaphthalen-1-yl acetate



### Peak Information

#	Peak Name	CH	tr [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	8.490	25641833	1681781	99.719	99.418	N/A	6460	1.381	1.182	
2	Unknown	5	8.920	72356	9846	0.281	0.582	N/A	31190	N/A	1.510	

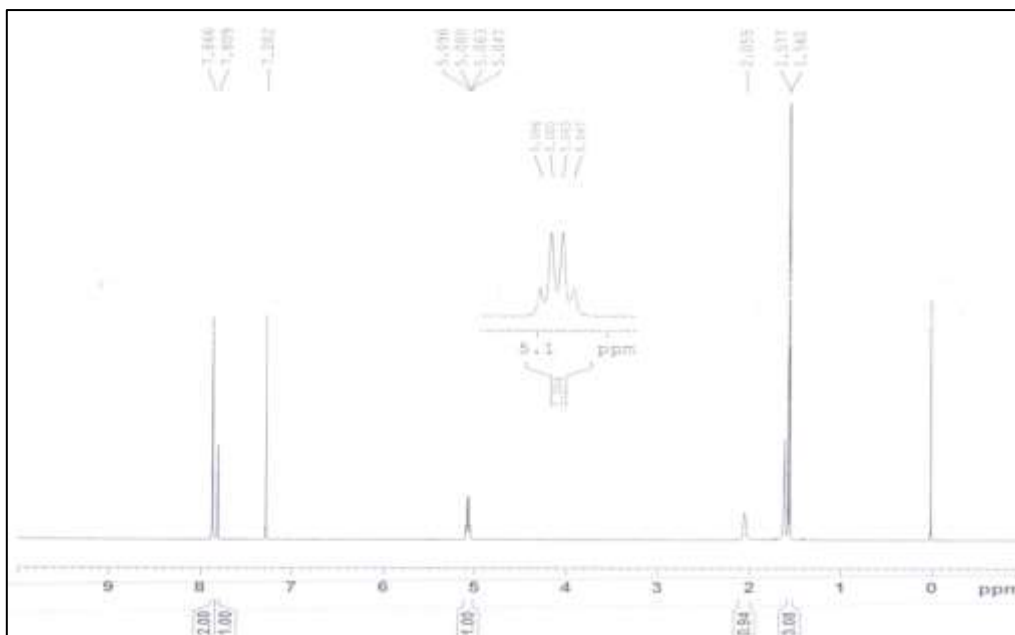
HPLC (R) 1,2,3,4-tetrahydronaphthalen-1-ol after mitsunobu



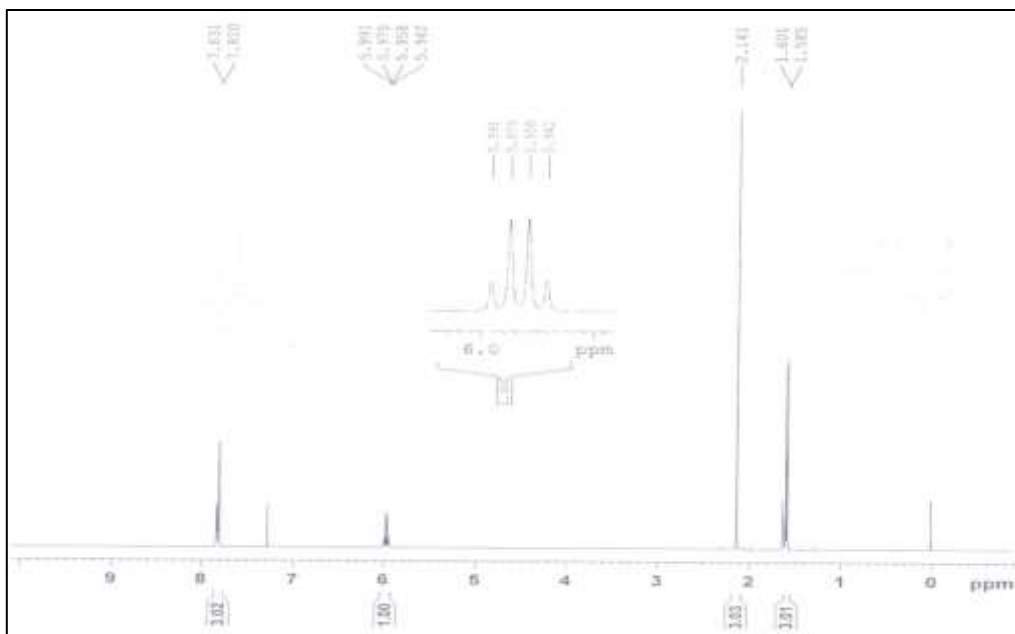
Peak Information

#	Peak Name	CH	TR [min]	Area [µV·sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	17.800	838201	50933	2.624	3.916	N/A	22667	4.302	0.935	
2	Unknown	5	19.720	31102045	1249648	97.376	96.084	N/A	13927	N/A	1.115	

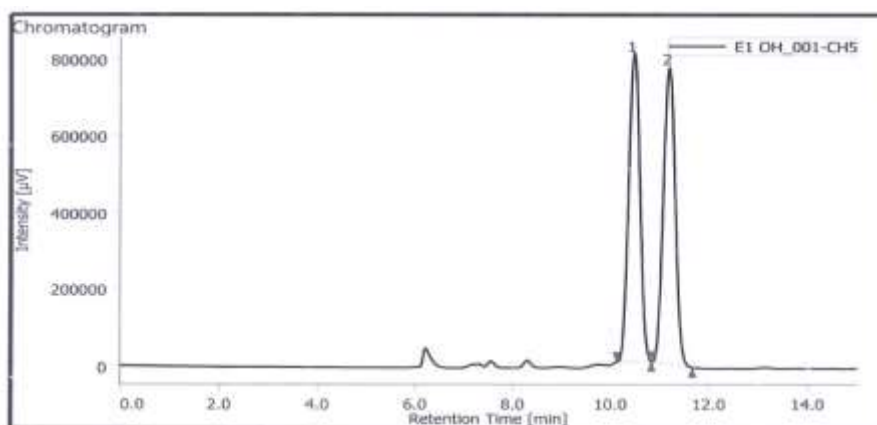
<sup>1</sup>H NMR of 1-(3,5-bis(trifluoromethyl)phenyl)ethan-1-ol



<sup>1</sup>H NMR of 1-(3,5-bis(trifluoromethyl)phenyl)ethyl acetate



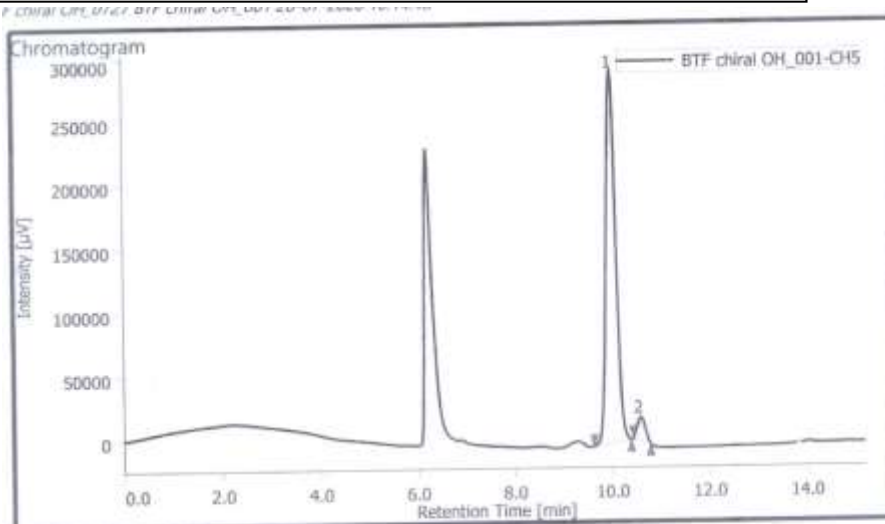
### HPLC of Racemic 1-(3,5-bis(trifluoromethyl)phenyl)ethan-1-ol



#	Peak Name	CH	TR [min]	Area [µV·sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	10.467	12880591	802697	50.003	51.064	N/A	9242	1.603	1.061	
2	Unknown	S	11.177	12878815	769248	48.997	48.936	N/A	9746	N/A	1.080	

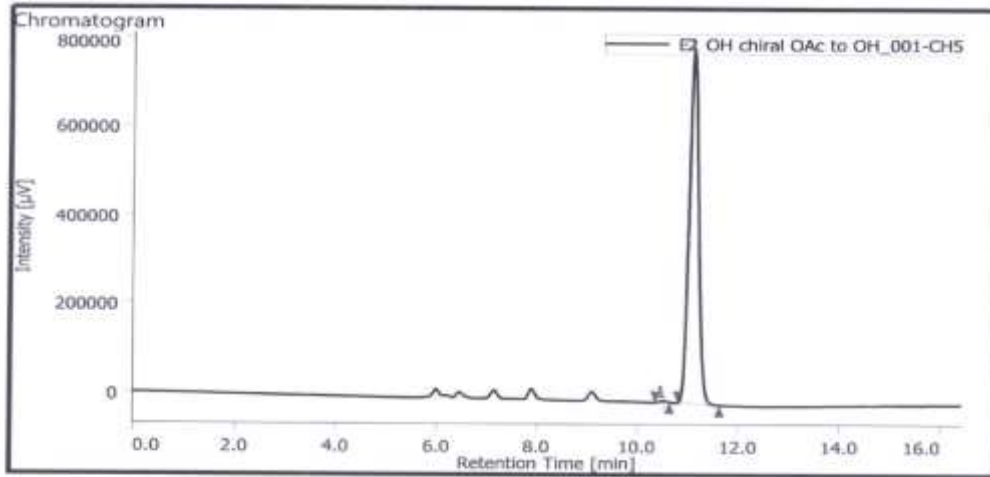
HPLC Condition for compound 1-(3,5-bis(trifluoromethyl)phenyl)ethan-1-ol<sup>b</sup> Determined by HPLC analysis (chiral column OD-H 0.5ml/min, IPA (5.0%) in hexane; 10.46 min for (*R*)-**13** and 11.17 min for (*S*)-**13**)

### HPLC of (*S*) 1-(3,5-bis(trifluoromethyl)phenyl)ethan-1-ol



#	Peak Name	CH	TR [min]	Area [µV·sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	9.997	4046572	290729	95.103	94.138	N/A	11883	1.688	1.051	
2	Unknown	S	10.577	208377	18224	4.897	5.861	N/A	17244	N/A	1.120	

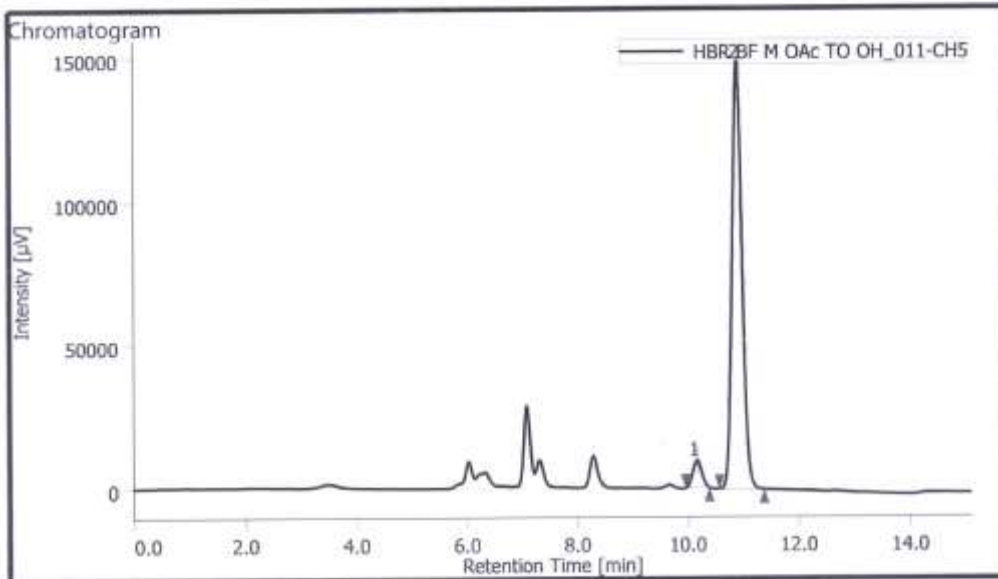
HPLC of (R) 1-(3,5-bis(trifluoromethyl)phenyl)ethan-1-ol.



Peak Information

#	Peak Name	CH	TR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	10.517	29271	3232	0.281	0.408	N/A	26714	1.889	0.882	
2	Unknown	S	11.087	10386013	789864	99.719	99.592	N/A	16270	N/A	1.103	

HPLC of (R) 1-(3,5-bis(trifluoromethyl)phenyl)ethan-1-ol after mitsunobu

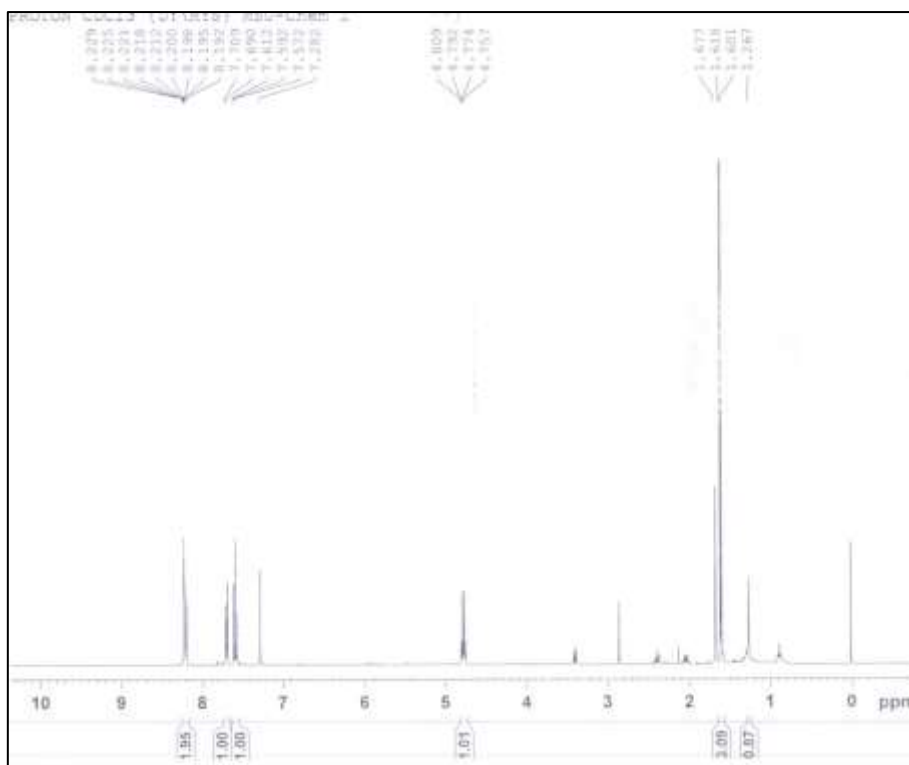


Peak Information

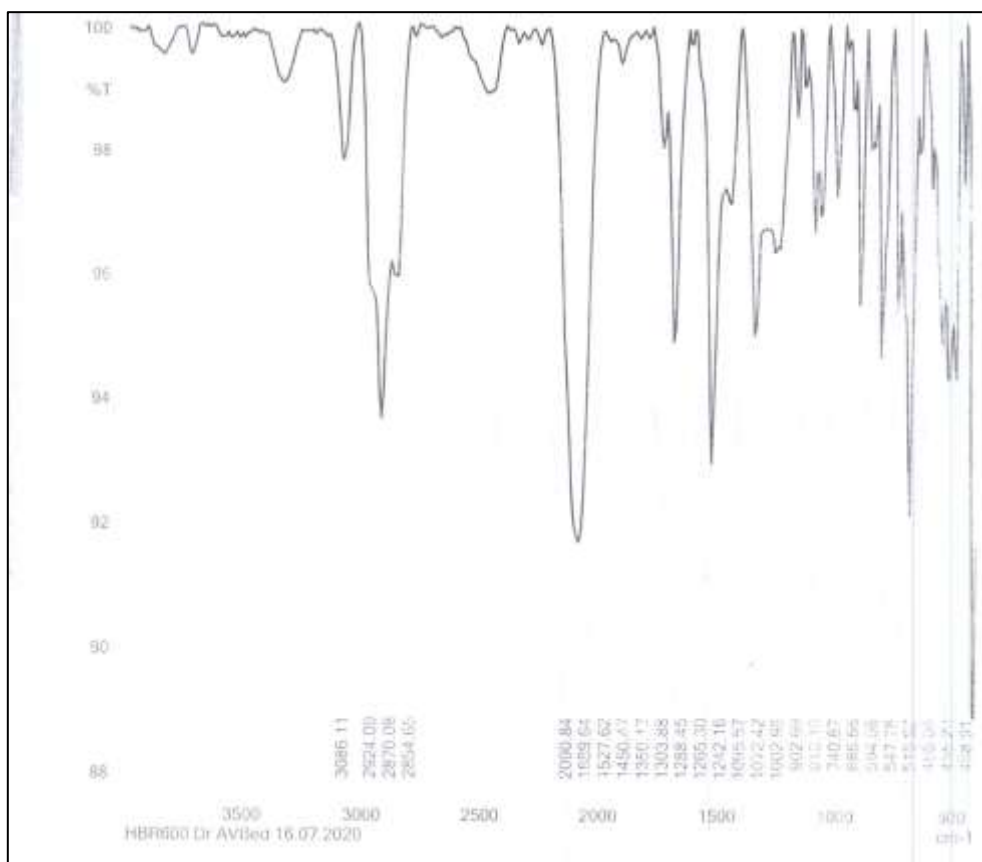
#	Peak Name	CH	TR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	10.157	107929	9536	5.460	6.017	N/A	17708	2.316	1.047	
2	Unknown	S	10.890	1868941	148943	94.540	93.983	N/A	17462	N/A	1.088	



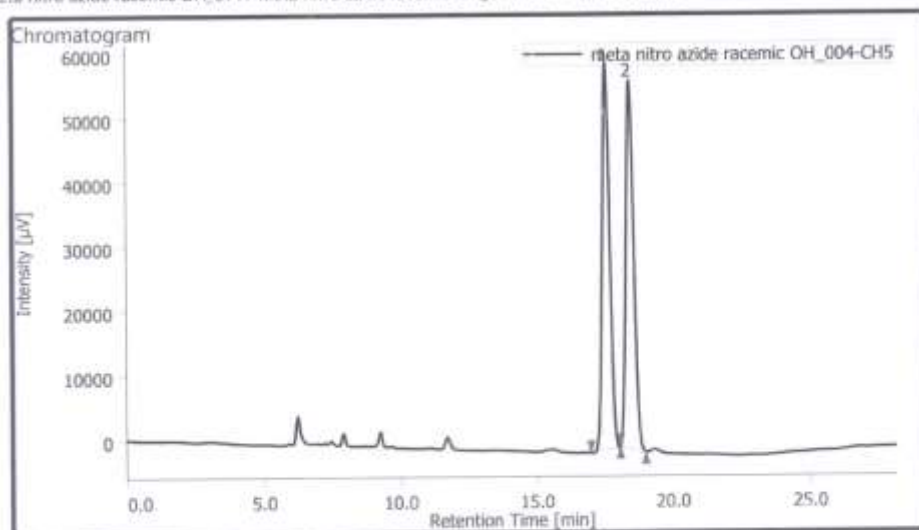
<sup>1</sup>H NMR 1-(1-azidoethyl)-3-nitrobenzene



IR 1-(1-azidoethyl)-3-nitrobenzene



HPLC racemic 1-(1-azidoethyl)-3-nitrobenzene

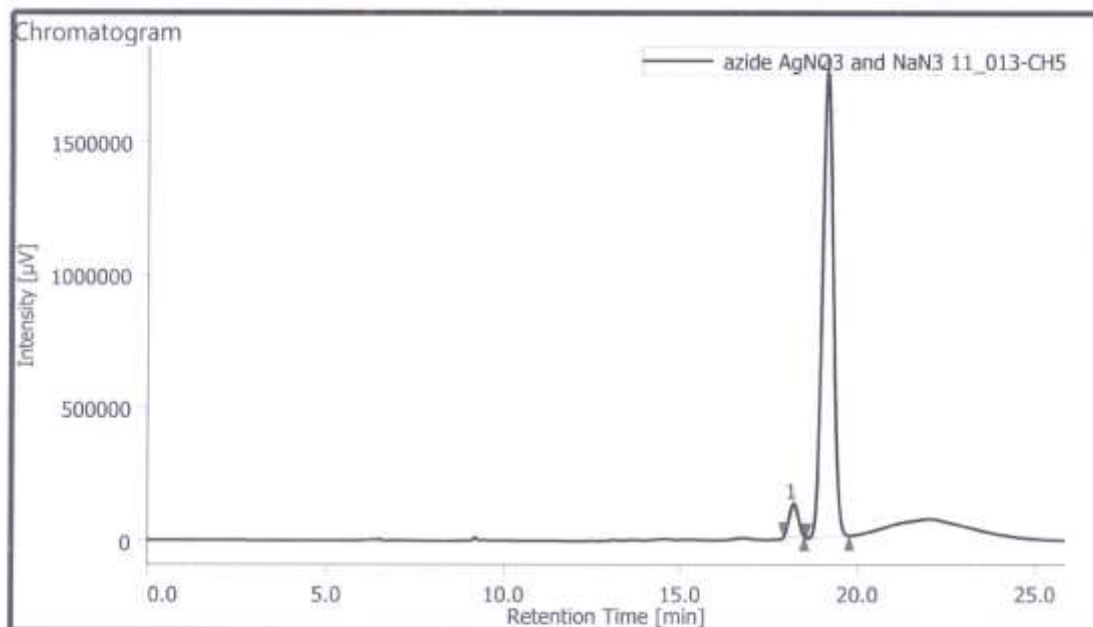


Peak Information

#	Peak Name	CH	tR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	17.550	1145231	60020	50.091	51.258	N/A	19049	1.682	1.062	
2	Unknown	5	18.423	1141063	57072	49.909	48.742	N/A	19134	N/A	1.093	

HPLC Condition for compound 1-(1-azidoethyl)-3-nitrobenzene<sup>b</sup>Determined by HPLC analysis (chiral column OD-H 0.5ml/min, IPA (5.0%) in hexane; 10.46 min for (R)-**17** and 11.17 min for (S)-**17**)

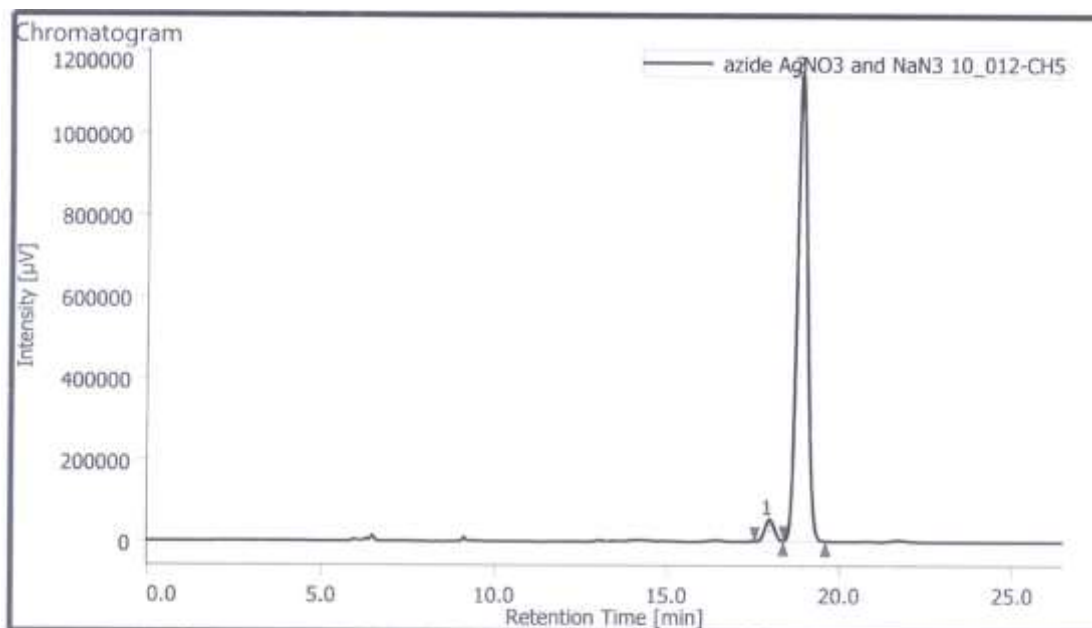
HPLC chiral (R) 1-(1-azidoethyl)-3-nitrobenzene (NaN<sub>3</sub>/AgNO<sub>3</sub>:2.0/0.0)



Peak Information

#	Peak Name	CH	tR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	18.197	2202454	124710	5.571	6.662	N/A	22085	1.781	1.021	
2	Unknown	5	19.137	37332581	1747195	94.429	93.338	N/A	18106	N/A	1.053	

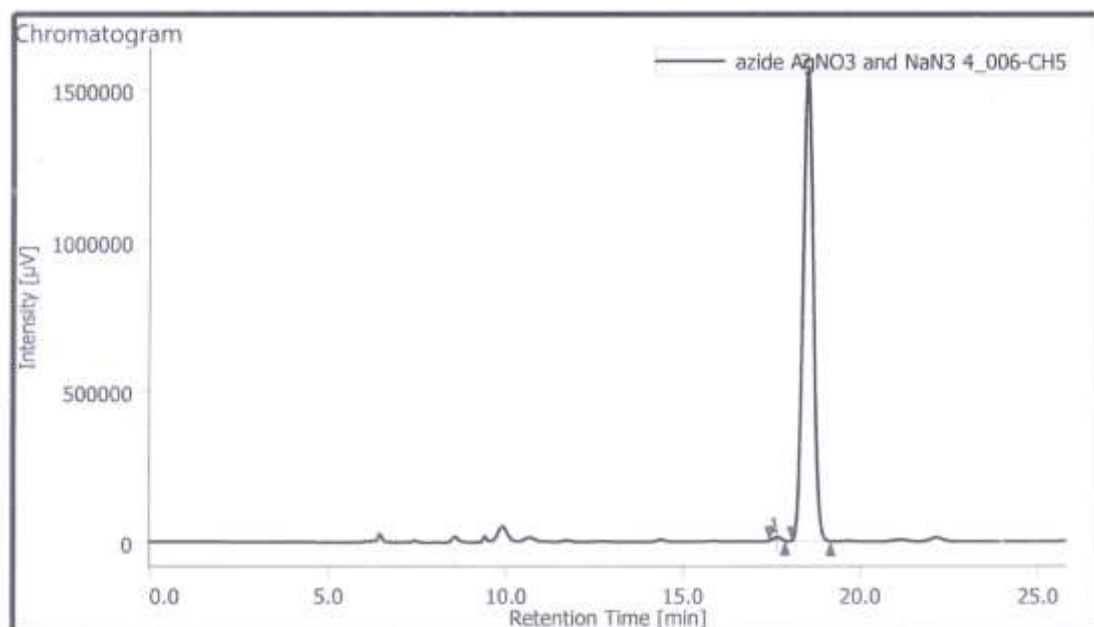
HPLC chiral (*R*)-1-(1-azidoethyl)-3-nitrobenzene (NaN<sub>3</sub>/AgNO<sub>3</sub>:1.75/0.25)



Peak Information

#	Peak Name	CH	tR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	17.967	1005149	53164	4.045	4.437	N/A	19973	1.734	1.003	
2	Unknown	5	18.883	23846105	1145036	95.955	95.563	N/A	18761	N/A	1.047	

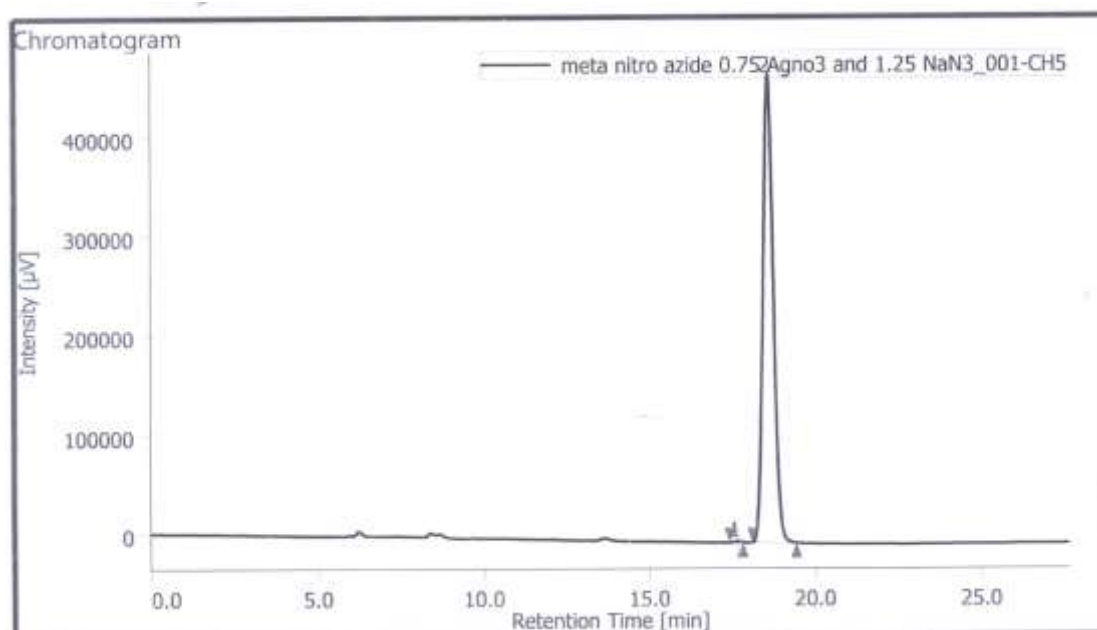
HPLC chiral (*R*)-1-(1-azidoethyl)-3-nitrobenzene (NaN<sub>3</sub>/AgNO<sub>3</sub>:1.50/0.50)



Peak Information

#	Peak Name	CH	tR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	17.650	172864	11727	0.548	0.748	N/A	28082	1.815	1.009	
2	Unknown	5	18.517	31393449	1556648	99.452	99.252	N/A	19094	N/A	1.049	

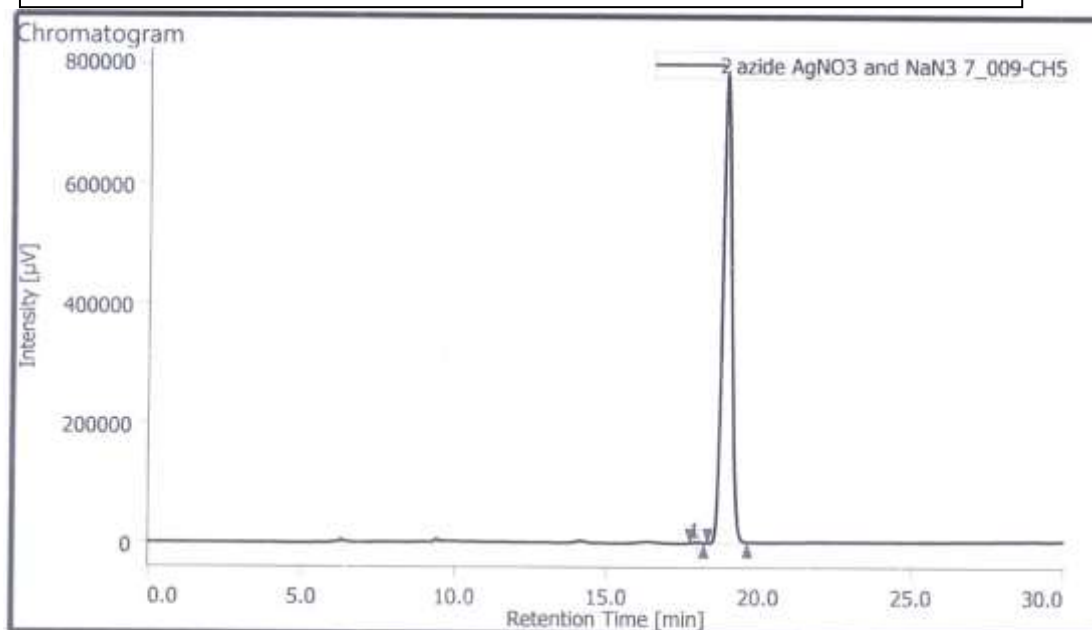
HPLC chiral (*R*) 1-(1-azidoethyl)-3-nitrobenzene (NaN<sub>3</sub>/AgNO<sub>3</sub>:1.25/0.75)



Peak Information

#	Peak Name	CH	tR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	17.653	8198	635	0.081	0.135	N/A	36661	1.997	0.847	
2	Unknown	5	18.587	10160469	470563	99.919	99.865	N/A	17120	N/A	1.099	

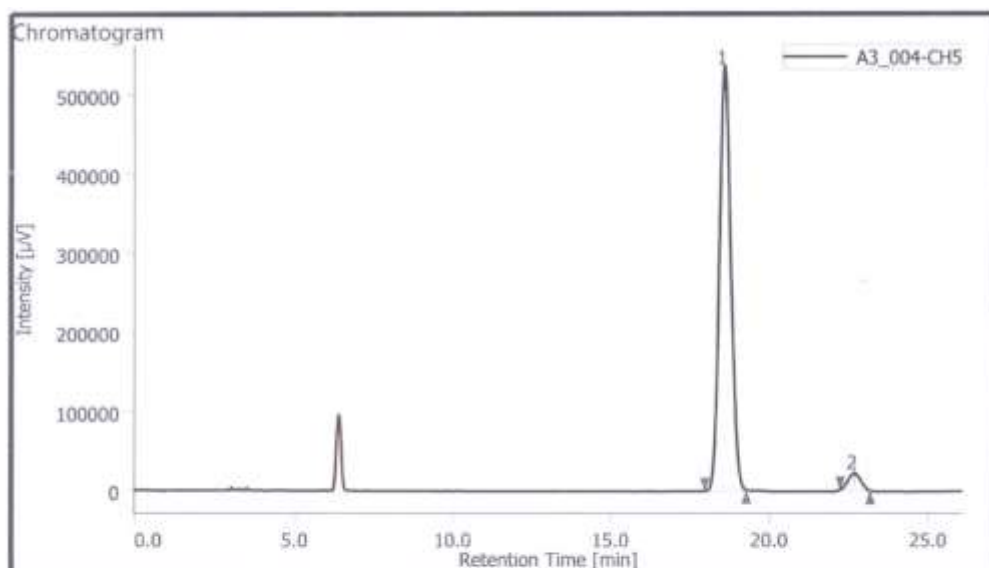
HPLC chiral (*R*) 1-(1-azidoethyl)-3-nitrobenzene (NaN<sub>3</sub>/AgNO<sub>3</sub>:2.0/0.75)



Peak Information

#	Peak Name	CH	tR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	17.997	23141	1502	0.142	0.191	N/A	26840	1.870	0.927	
2	Unknown	5	18.920	16303509	783218	99.858	99.809	N/A	18866	N/A	1.059	

HPLC (*R*) 1-(3-nitrophenyl)ethan-1-ol (NaN3/AgNO3:2.0/0.0)

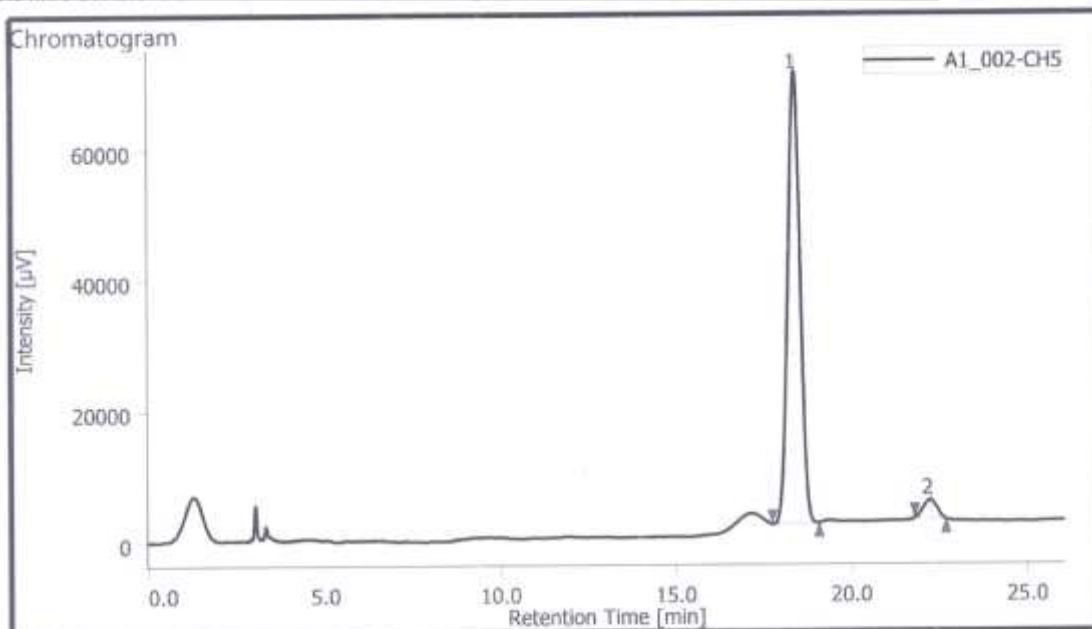


Peak Information

#	Peak Name	CH	tR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	18.587	13456708	535620	95.746	96.144	N/A	12497	5.694	1.139	
2	Unknown	S	22.660	597605	21483	4.252	3.856	N/A	13874	N/A	1.034	

HPLC Condition for compound 1-(3-nitrophenyl)ethan-1-ol<sup>b</sup> Determined by HPLC analysis (chiral column Amylose, 1ml/min IPA (5.0%) in hexane; 18.42 min for (*R*)-**1** and 22.37 min for (*S*)-**1**)

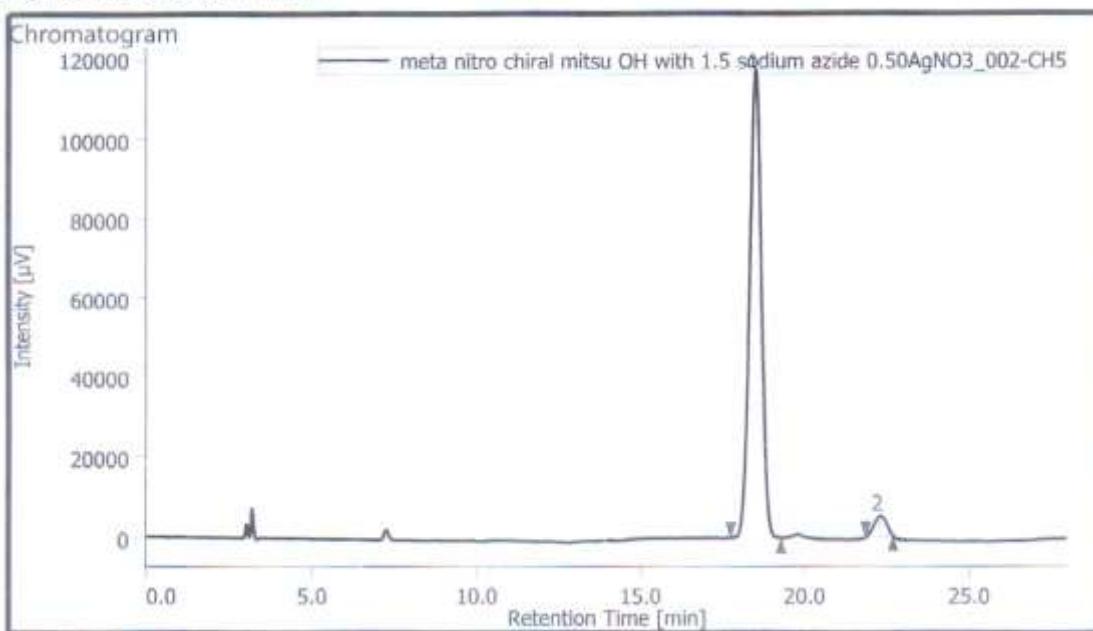
HPLC (*R*) 1-(3-nitrophenyl)ethan-1-ol (NaN3/AgNO3:1.75/0.25)



Peak Information

#	Peak Name	CH	tR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	S	18.380	1678417	68890	95.597	95.989	N/A	12808	5.514	0.981	
2	Unknown	S	22.230	77298	2876	4.403	4.011	N/A	14031	N/A	0.968	

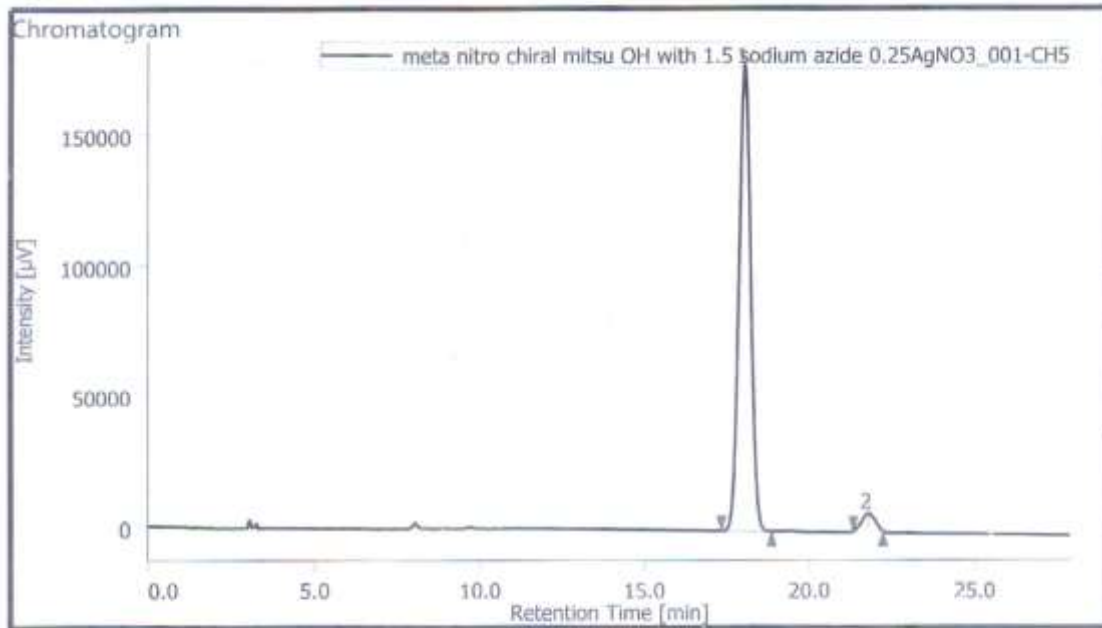
HPLC (R) 1-(3-nitrophenyl)ethan-1-ol (NaN<sub>3</sub>/AgNO<sub>3</sub>:1.50/0.50)



Peak Information

#	Peak Name	CH	tR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	18.483	2914862	117425	95.736	95.959	N/A	12639	5.462	1.016	
2	Unknown	5	22.280	129821	4945	4.264	4.041	N/A	14649	N/A	0.952	

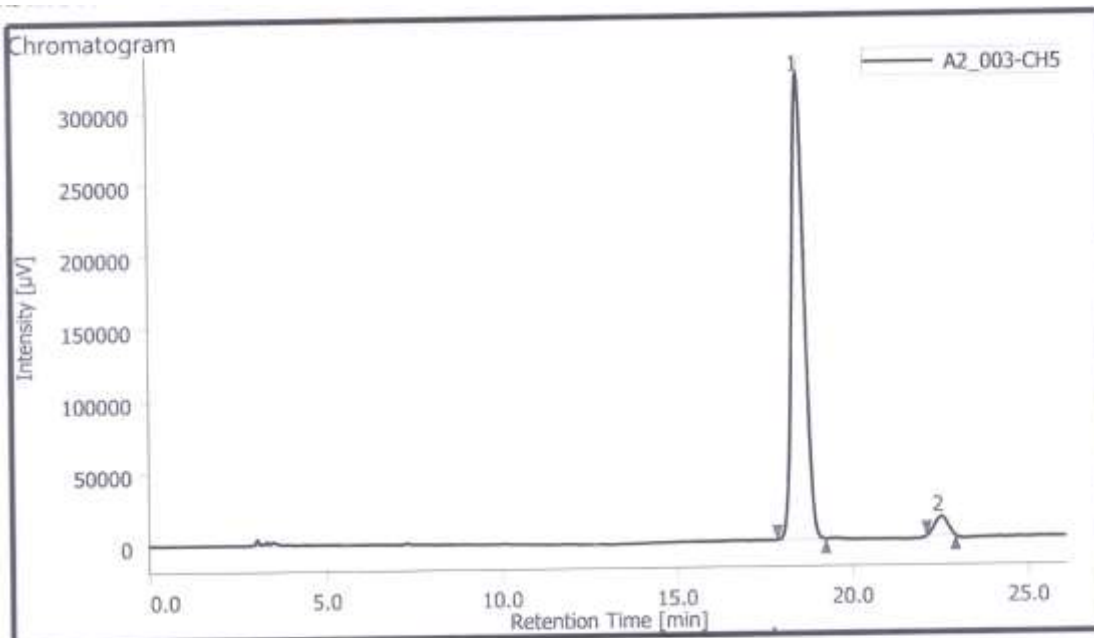
HPLC (R) 1-(3-nitrophenyl)ethan-1-ol (NaN<sub>3</sub>/AgNO<sub>3</sub>:1.25/0.75)



Peak Information

#	Peak Name	CH	tR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	18.037	4406564	178458	96.081	96.465	N/A	12134	5.307	1.034	
2	Unknown	5	21.793	179759	6539	3.919	3.535	N/A	12996	N/A	0.940	

HPLC (R) 1-(3-nitrophenyl)ethan-1-ol (NaN<sub>3</sub>/AgNO<sub>3</sub>:2.0/0.75)



Peak Information

#	Peak Name	CH	tR [min]	Area [µV-sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	5	18.493	7953367	322782	95.972	96.122	N/A	12869	5.871	1.061	
2	Unknown	5	22.503	333772	13024	4.028	3.878	N/A	15733	N/A	0.992	

