Supplementary Material

Convergent method for the determination of the absolute configurations of 2,3-dihydro-1*H*-inden-1-ols

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Table of Contents

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Data File:      C:\CHEM32\1\DATA\2019-12-13\2019-12-13 2019-12-13 16-27-
                25\4001_RAC_3_2.D
Sample Name:    4001_RAC
-------------------------------------------------------------------------

Acq. Operator   :  3_Krasnopolskaya                 Location:    Vial 91
Acq. Instrument :  Instrument 1
Injection Date  :  18:39:36    13.12.2019
Injection Volume:  5  mkl
Sample Info:       OJ-H, Hexane-IPA, 97-3, 1.0ml/min

-------------------------------------------------------------------------
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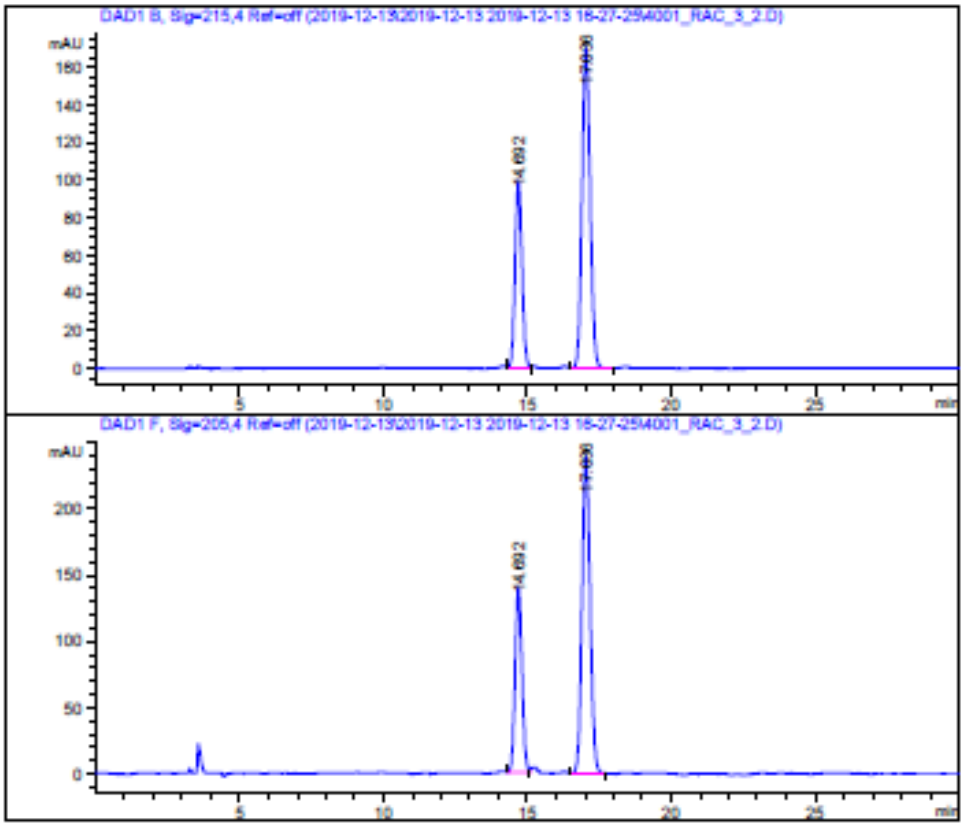


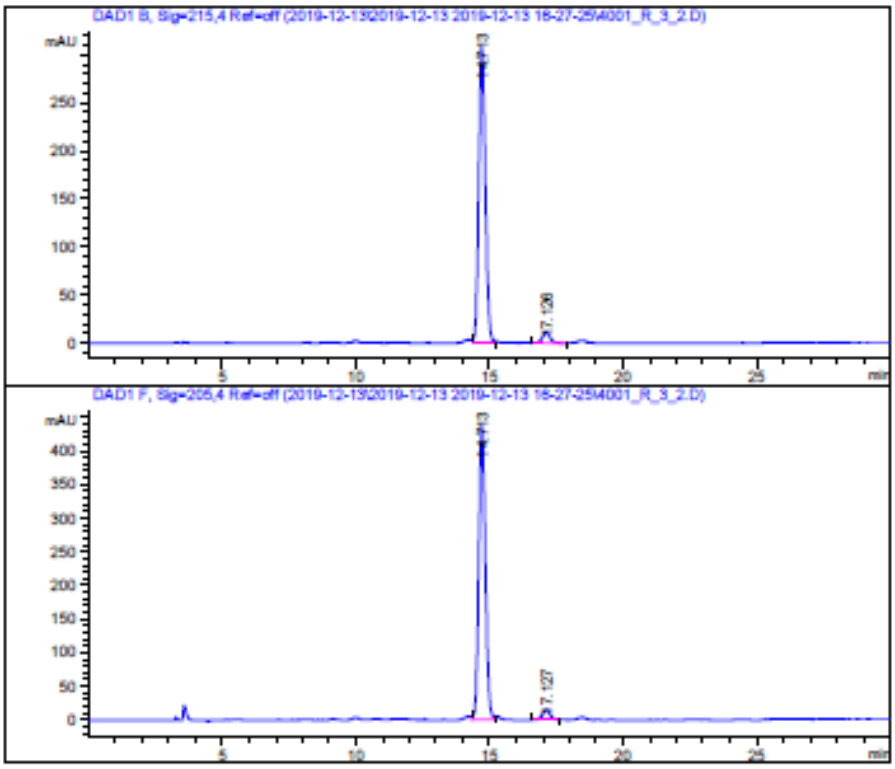

```
Signal:  DAD1 B, Sig=215,4 Ref=off
-------------------------------------------------------------------------
RetTime(min)  Area,%    Symm.     Resolution Selectivity
|-----------|--------|----------|----------|----------|
14.6922         33.31       0.910
17.0362         66.69       0.865     4.89         1.16
-------------------------------------------------------------------------

Signal:  DAD1 F, Sig=285,4 Ref=off
-------------------------------------------------------------------------
RetTime(min)  Area,%    Symm.     Resolution Selectivity
|-----------|--------|----------|----------|----------|
14.6922         33.04       0.903
17.0362         66.96       0.867     4.89         1.16
-------------------------------------------------------------------------
```

Chiral HPLC of (*Rac*)-4-fluoro-2,3-dihydro-1H-inden-1-ol

Data File: C:\CHEM32\1\DATA\2019-12-13\2019-12-13 2019-12-13 16-27-25\4001_R_3_2.D
Sample Name: EN300-89318

Acq. Operator : 3_Krasnopolskaya Location: Vial 92
Acq. Instrument : Instrument 1
Injection Date : 19:11:51 13.12.2019
Injection Volume: 5 mkl
Sample Info: OJ-H, Hexane-IPA, 97-3, 1.0ml/min



Signal: DAD1 B, Sig=215,4 Ref=off

RetTime(min)	Area,%	Symm.	Resolution	Selectivity
14.7132	96.12	0.838		
17.1262	3.88	0.956	5.01	1.16

Signal: DAD1 F, Sig=285,4 Ref=off

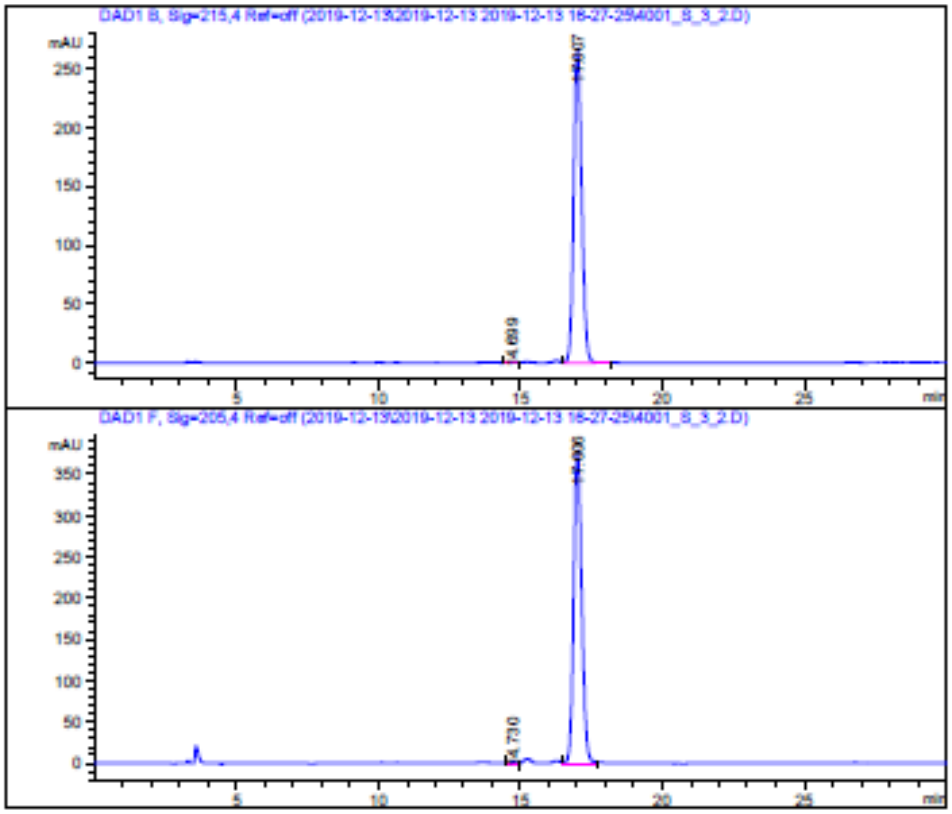
RetTime(min)	Area,%	Symm.	Resolution	Selectivity
14.7132	96.20	0.826		
17.1272	3.80	0.802	5.05	1.16

Instrument 1 16.12.2019 9:01:04 Page 1 of 1

Chiral HPLC of (*R*)-4-fluoro-2,3-dihydro-1H-inden-1-ol

```
Data File:      C:\CHEM32\1\DATA\2019-12-13\2019-12-13 2019-12-13 16-27-
                25\4001_S_3_2.D
Sample Name:    R1982471
```

```
Acq. Operator   :  3_Krasnopolskaya              Location:    Vial 93
Acq. Instrument :  Instrument 1
Injection Date  :  19:44:09    13.12.2019
Injection Volume:  5  mkl
Sample Info:       OJ-H, Hexane-IPA, 97-3, 1.0ml/min
```




```
Signal:  DAD1 B, Sig=215,4 Ref=off
```

RetTime(min)	Area,%	Symm.	Resolution	Selectivity
14.6992	0.23	0.875		
17.0072	99.77	0.820	4.83	1.16

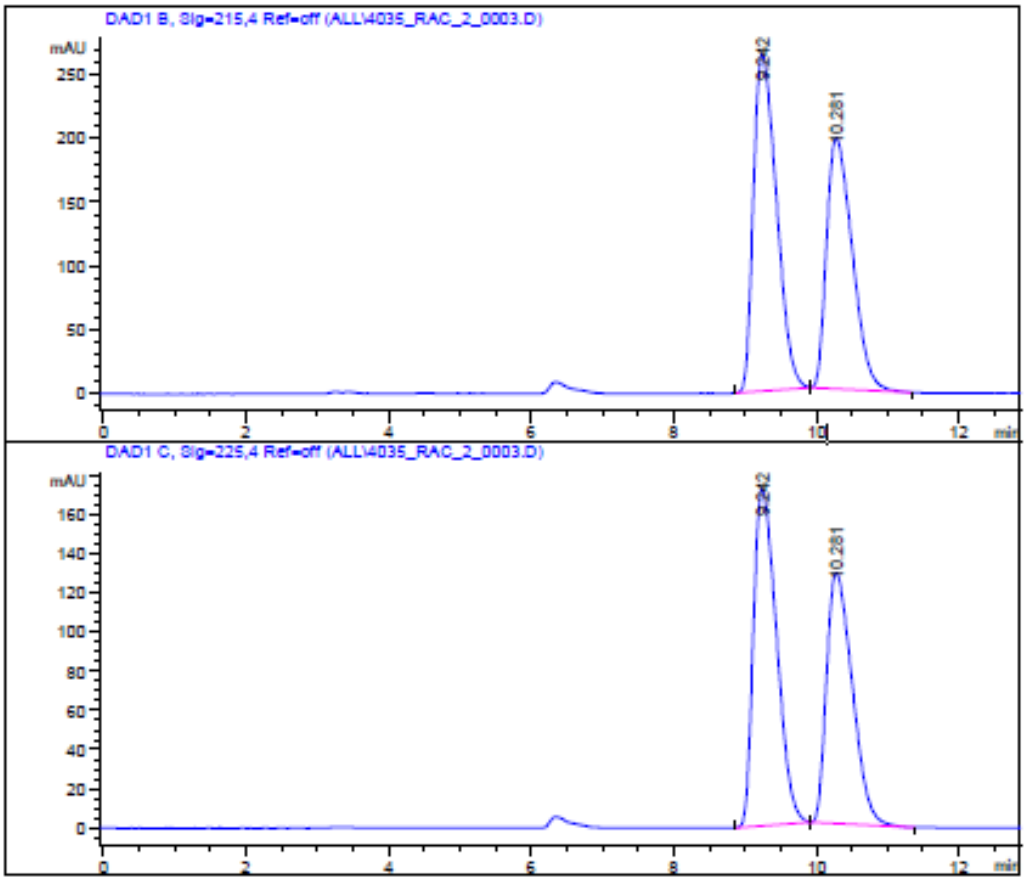
```
Signal:  DAD1 F, Sig=285,4 Ref=off
```

RetTime(min)	Area,%	Symm.	Resolution	Selectivity
14.7382	0.30	0.806		
17.0062	99.70	0.814	4.49	1.15

Chiral HPLC of (*S*)-4-fluoro-2,3-dihydro-1H-inden-1-ol

```
Sample Name:    4035_RAC_
================================================================================

Acq. Operator   :  2_Pugach                          Location:    Vial 68
Acq. Instrument :  Instrument 1
Injection Date  :  12:40:21    19-Dec-19
Injection Volume:  1  mkl
Sample Info:       OJ-H, Hexane-IPA-MeOH, 95-2.5-2.5,  1.0 ml/min
```



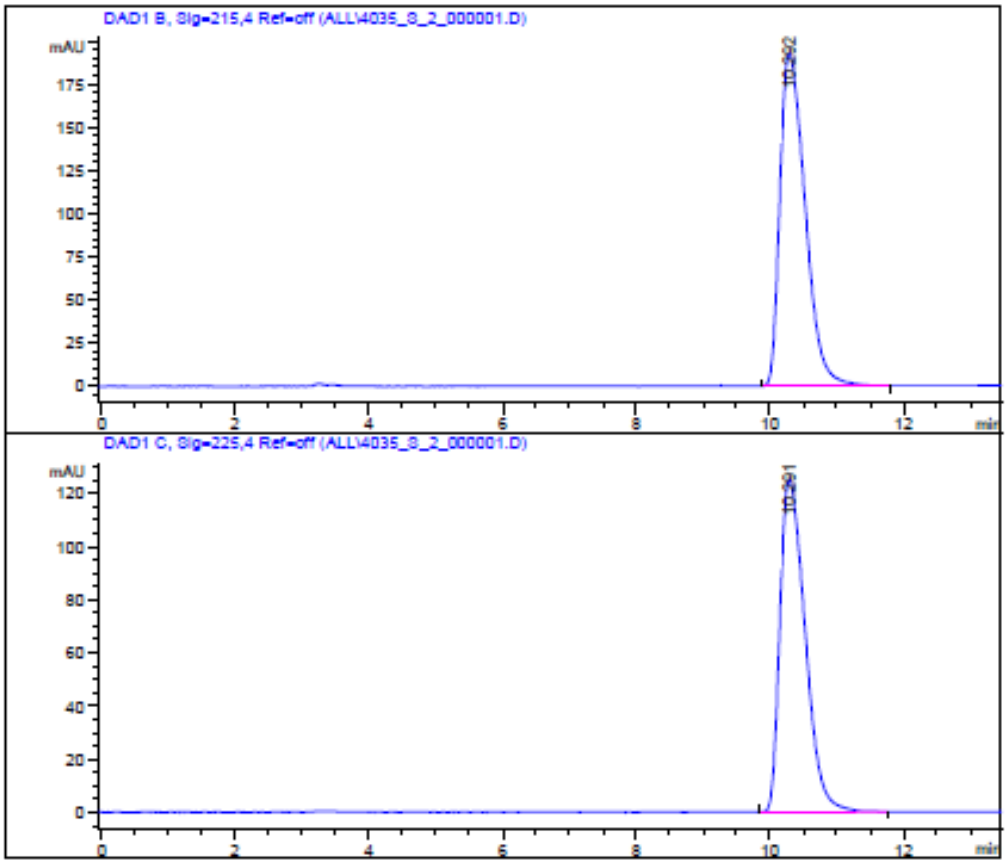

```
Signal:  DAD1 B, Sig=215,4 Ref=off
================================================================================
 RetTime(min)  Area,%     Symm.     Resolution Selectivity
|-------------|--------|----------|-----------|-----------|
 9.2422          54.64       0.582
 10.2812         45.36       0.573      1.60         1.17
================================================================================

Signal:  DAD1 C, Sig=225,4 Ref=off
================================================================================
 RetTime(min)  Area,%     Symm.     Resolution Selectivity
|-------------|--------|----------|-----------|-----------|
 9.2422          54.68       0.581
 10.2812         45.32       0.574      1.60         1.17
================================================================================
```

Chiral HPLC of rac- 4-chloro-2,3-dihydro-1H-inden-1-ol

Sample Name: 4035_S_

Acq. Operator : 2_Pugach Location: Vial 70
Acq. Instrument : Instrument 1
Injection Date : 13:09:56 19-Dec-19
Injection Volume: 1 mkl
Sample Info: OJ-H, Hexane-IPA-MeOH, 95-2.5-2.5, 1.0 ml/min



Signal: DAD1 B, Sig=215,4 Ref=off

RetTime(min)	Area,%	Symm.	Resolution	Selectivity
10.2922	100.00	0.555		

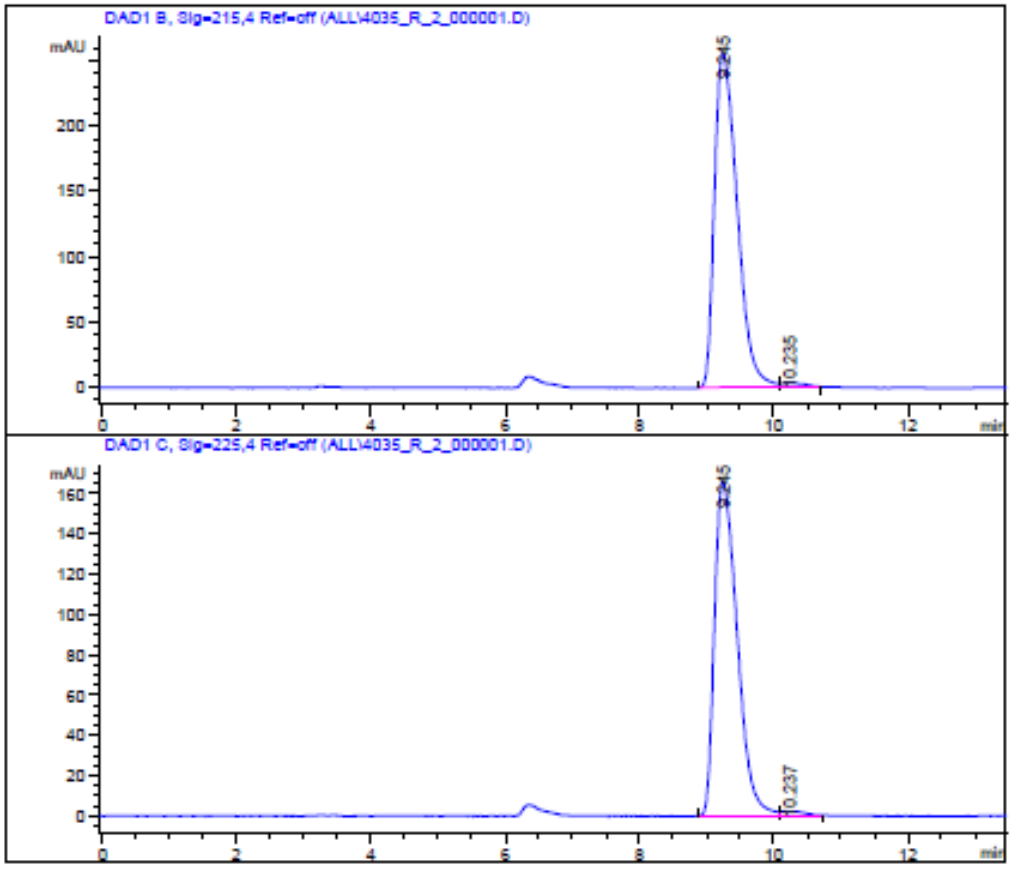
Signal: DAD1 C, Sig=225,4 Ref=off

RetTime(min)	Area,%	Symm.	Resolution	Selectivity
10.2912	100.00	0.554		

Chiral HPLC of (S)-4-bromo-2,3-dihydro-1H-inden-1-ol

Sample Name: 4035_R_

Acq. Operator : 2_Pugach Location: Vial 69
Acq. Instrument : Instrument 1
Injection Date : 12:54:54 19-Dec-19
Injection Volume: 1 mkl
Sample Info: OJ-H, Hexane-IPA-MeOH, 95-2.5-2.5, 1.0 ml/min



Signal: DAD1 B, Sig=215,4 Ref=off

RetTime(min)	Area,%	Symm.	Resolution	Selectivity
9.2452	98.78	0.575		
10.2352	1.22	0.520	1.53	1.16

Signal: DAD1 C, Sig=225,4 Ref=off

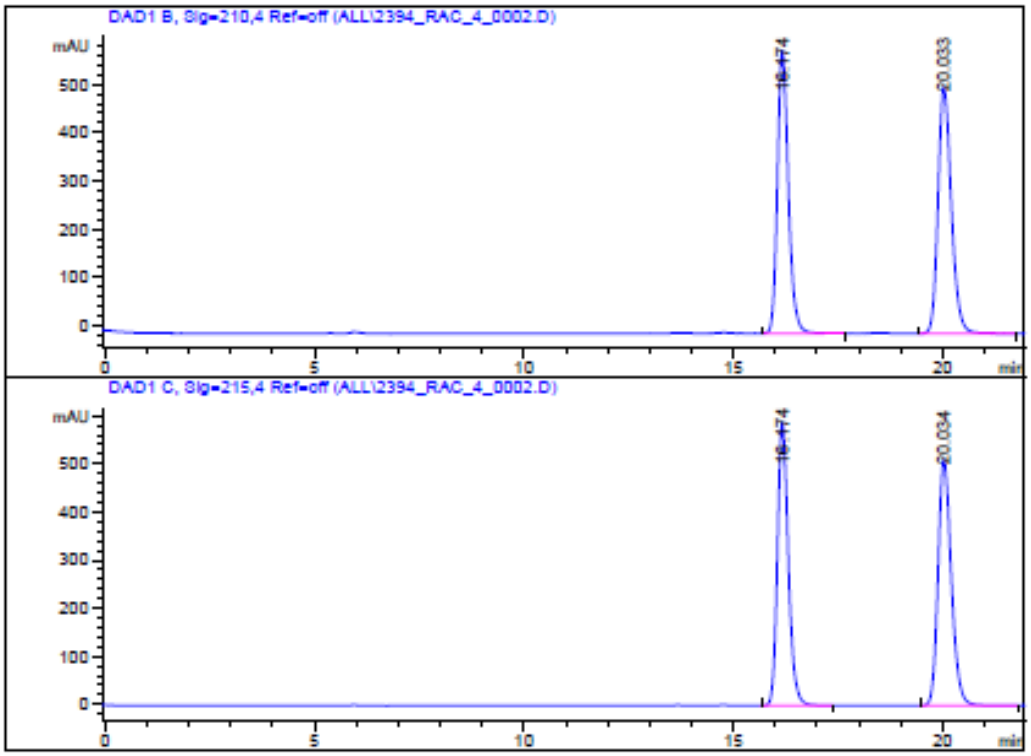
RetTime(min)	Area,%	Symm.	Resolution	Selectivity
9.2452	98.64	0.573		
10.2372	1.36	0.500	1.53	1.16

Chiral HPLC of (R)-4-bromo-2,3-dihydro-1H-inden-1-ol

```
Sample Name:    2394_RAC_
================================================================================

Acq. Operator   : 4_Vinogradska                    Location:   Vial 31
Acq. Instrument : Instrument 1
Injection Date  : 13:09:31   03.12.2018
Injection Volume: 1  mkl
Sample Info:      IA, Hexane-IPA-MeOH, 95-2.5-2.5, 0.6ml/min

================================================================================
```




```
Signal: DAD1 B, Sig=210,4 Ref=off
================================================================================
 RetTime(min) Area,%    Symm.    ResolutionSelectivity
|------------|--------|----------|----------|----------|
 16.1742       48.61      0.786
 20.0332       51.39      0.759    7.56       1.35
================================================================================


Signal: DAD1 C, Sig=215,4 Ref=off
================================================================================
 RetTime(min) Area,%    Symm.    ResolutionSelectivity
|------------|--------|----------|----------|----------|
 16.1742       48.67      0.788
 20.0342       51.33      0.769    7.53       1.35
================================================================================
```

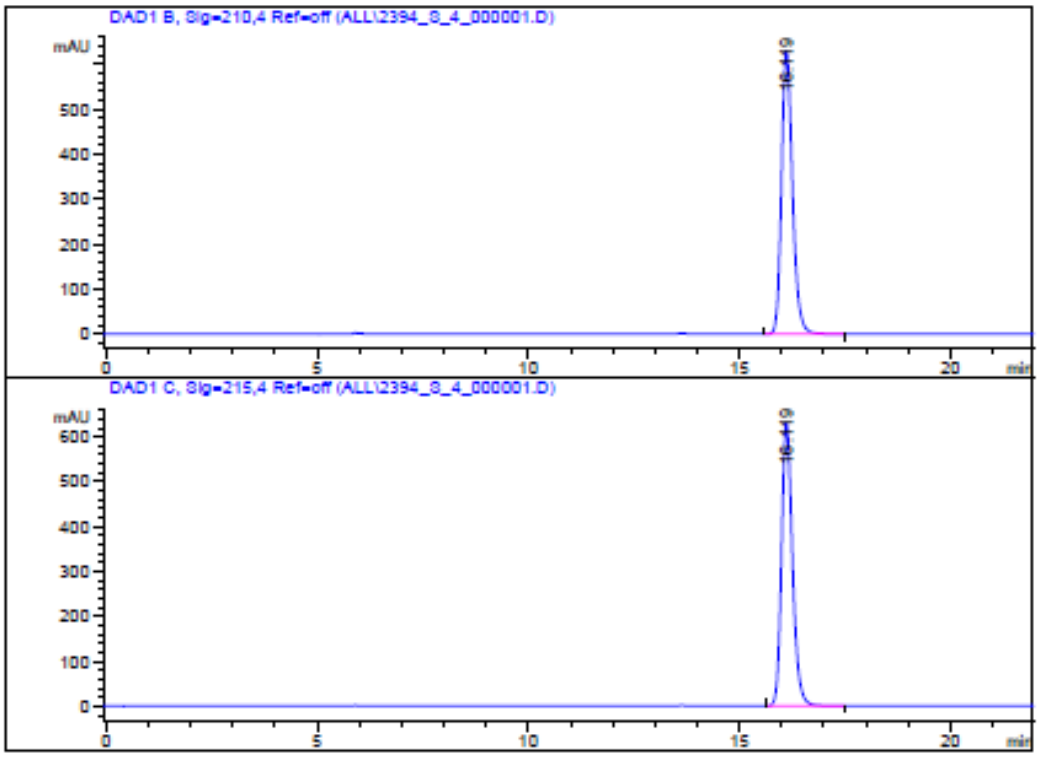
Chiral HPLC of (rac)-5-chloro-2,3-dihydro-1H-inden-1-ol

```
Data File:      C:\CHEM32\1\DATA\ALL\2394_S_4_000001.D

Sample Name:    R1986897
================================================================================

Acq. Operator   : 4_Vinogradska                    Location:   Vial 33
Acq. Instrument : Instrument 1
Injection Date  : 13:57:58   03.12.2018
Injection Volume: 1  mkl
Sample Info:      IA, Hexane-IPA-MeOH, 95-2.5-2.5, 0.6ml/min

================================================================================
```




```
Signal: DAD1 B, Sig=210,4 Ref=off
================================================================================
RetTime(min) Area,%    Symm.    ResolutionSelectivity
|------------|--------|----------|-----------|-----------|
16.1192       100.00     0.775
================================================================================

Signal: DAD1 C, Sig=215,4 Ref=off
================================================================================
RetTime(min) Area,%    Symm.    ResolutionSelectivity
|------------|--------|----------|-----------|-----------|
16.1192       100.00     0.782
================================================================================
```

Chiral HPLC of (S)- 5-chloro-2,3-dihydro-1H-inden-1-ol

```
Data File:      C:\CHEM32\1\DATA\ALL\2394_R_4_000001.D

Sample Name:    R1986898
================================================================================

Acq. Operator   : 4_Vinogradska                  Location:   Vial 32
Acq. Instrument : Instrument 1
Injection Date  : 13:33:45   03.12.2018
Injection Volume: 1  mkl
Sample Info:      IA, Hexane-IPA-MeOH, 95-2.5-2.5, 0.6ml/min

================================================================================
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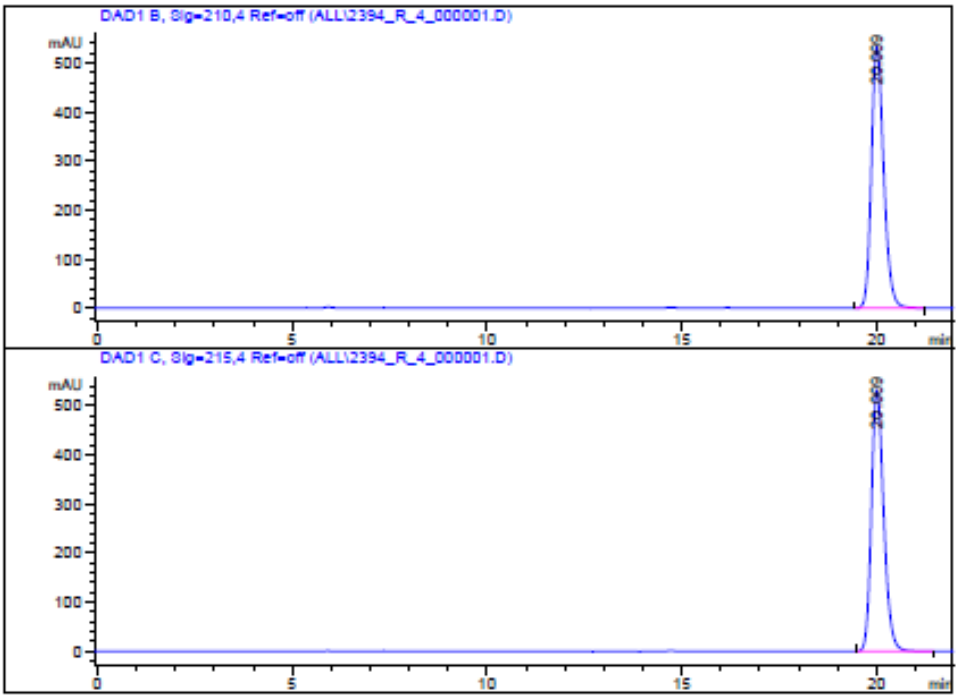
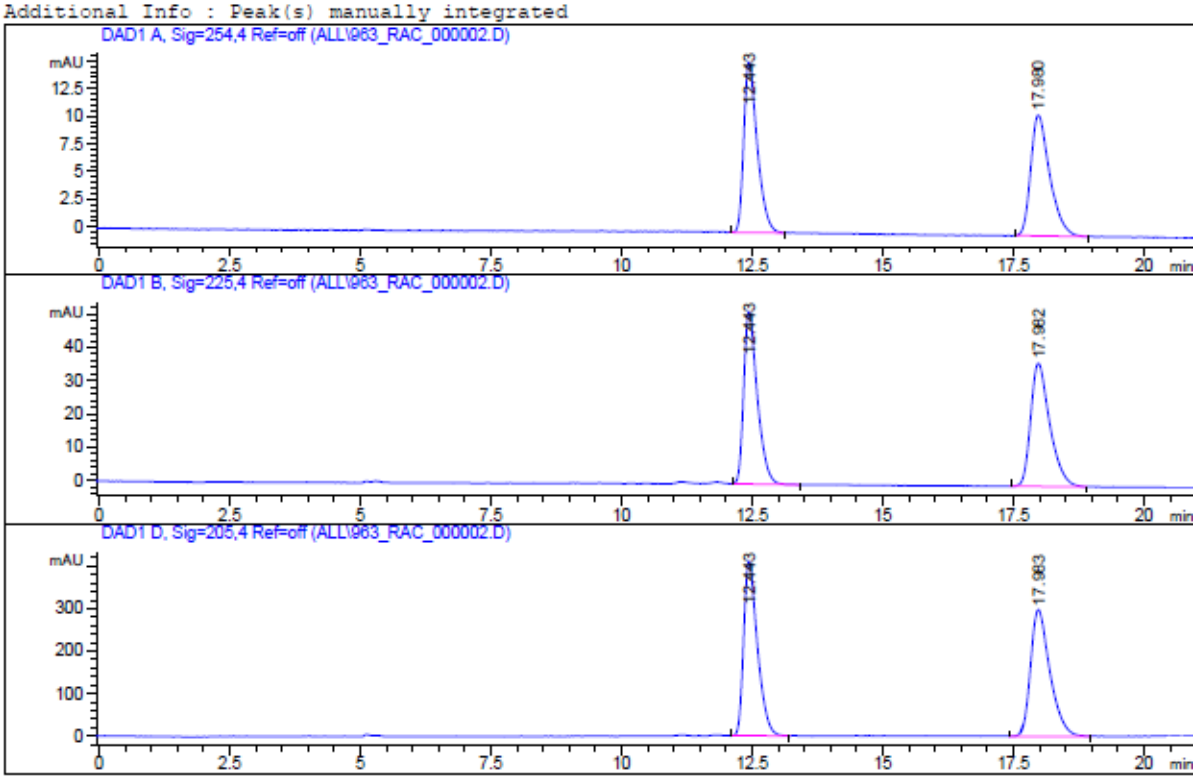



```
Signal: DAD1 B, Sig=210,4 Ref=off
================================================================================
RetTime (min) Area,%    Symm.   ResolutionSelectivity
|-------------|--------|---------|-----------|-----------|
20.0092       100.00     0.749
================================================================================

Signal: DAD1 C, Sig=215,4 Ref=off
================================================================================
RetTime (min) Area,%    Symm.   ResolutionSelectivity
|-------------|--------|---------|-----------|-----------|
20.0092       100.00     0.747
================================================================================
```

Chiral HPLC of (S)-5-chloro-2,3-dihydro-1H-inden-1-ol

Additional Info : Peak(s) manually integrated

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=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  12.443 BB    0.2665  275.02417   15.39033  49.9985
   2  17.980 BB    0.3769  275.04034   10.88141  50.0015
```
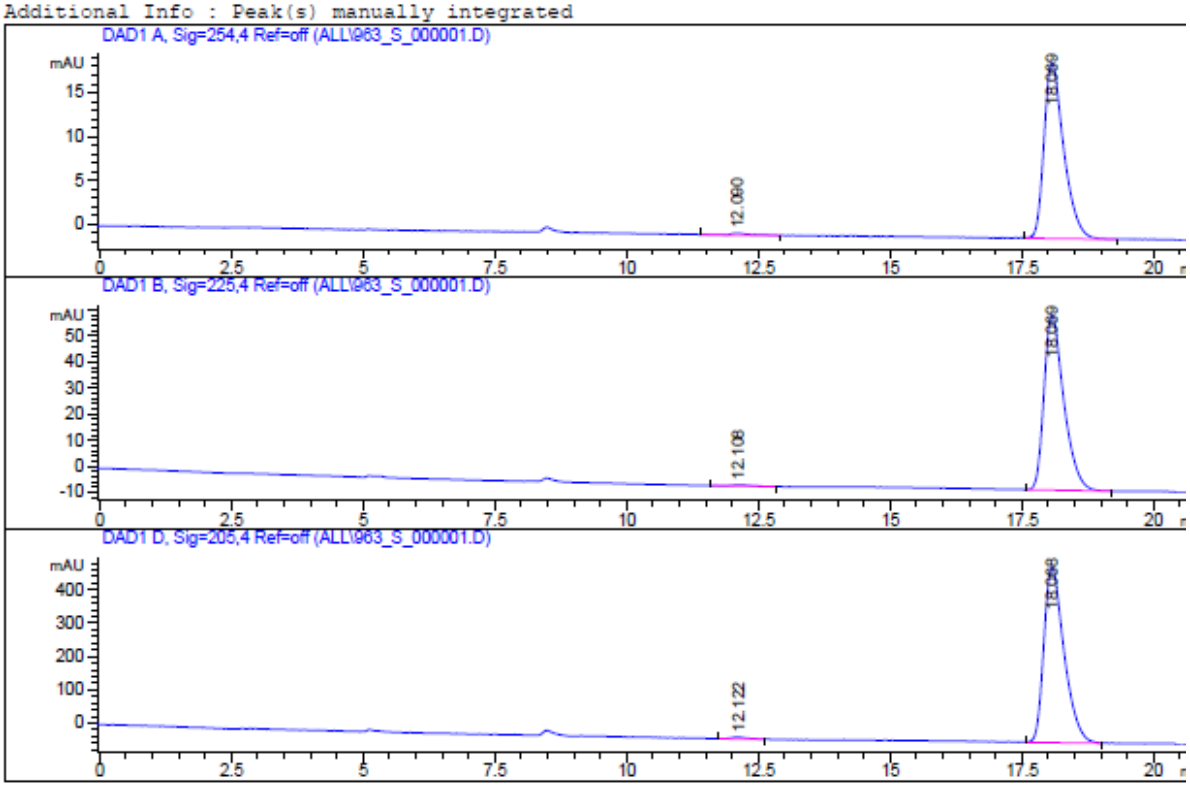

Chromatogram of racemic (*trans)*-2-bromo-2,3-dihydro-1H-inden-1-ol

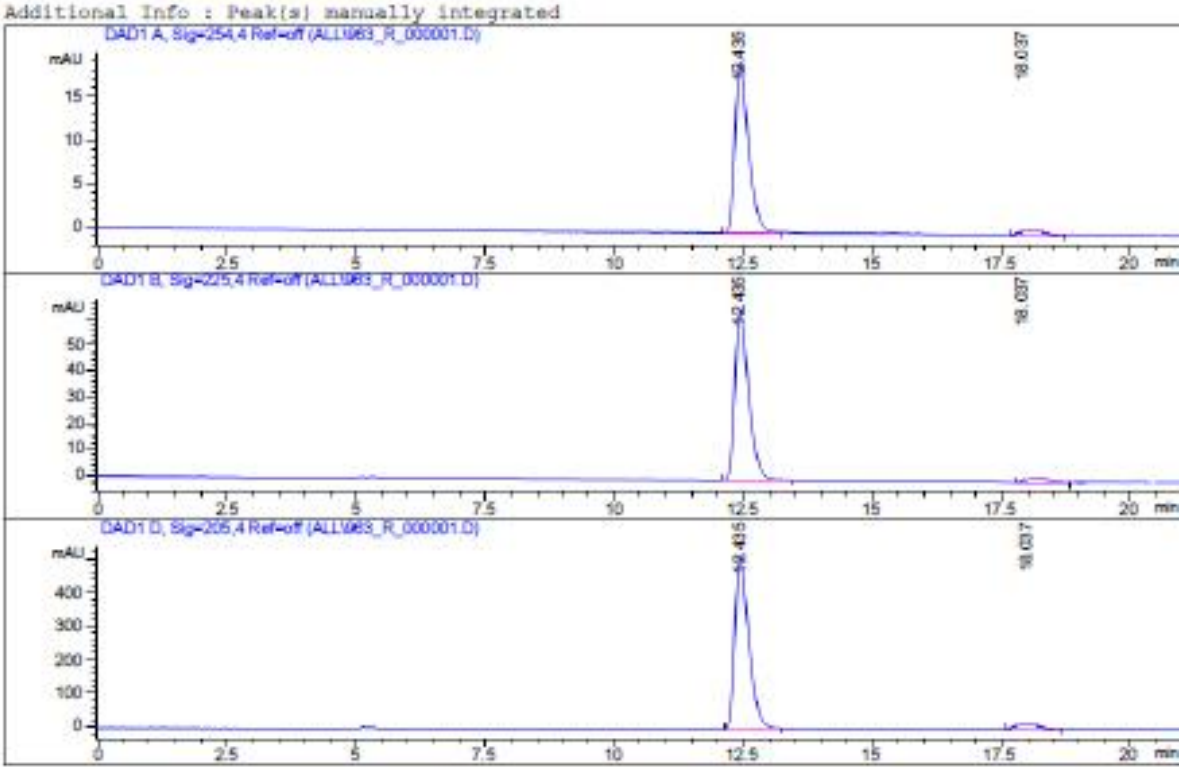
Additional Info : Peak(s) manually integrated




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  12.090 MM R  0.3881    5.17378 2.22200e-1   0.9834
   2  18.069 BB    0.3884  520.93622   20.09999  99.0166
```

Chromatogram of enantiopure (1*S*,2*R*)-2-bromo-2,3-dihydro-1H-inden-1-ol (chiral column Chiralcel OJ-H (250*4,6 mm, with Selector Celulose tris(4-methylbenzoate) coated on 5 mμ silicagel))

Additional Info : Peak(s) manually integrated



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.435	BB	0.2666	510.83340	20.0077	97.895
2	18.037	MM R	0.4508	10.76354	2.52522e-1	2.105

Chromatogram of enantiopure (1*R*,2*S*)-2-bromo-2,3-dihydro-1H-inden-1-ol (chiral column Chiralcel OJ-H (250*4,6 mm, with Selector Celulose tris(4-methylbenzoate) coated on 5 mμ silicagel))

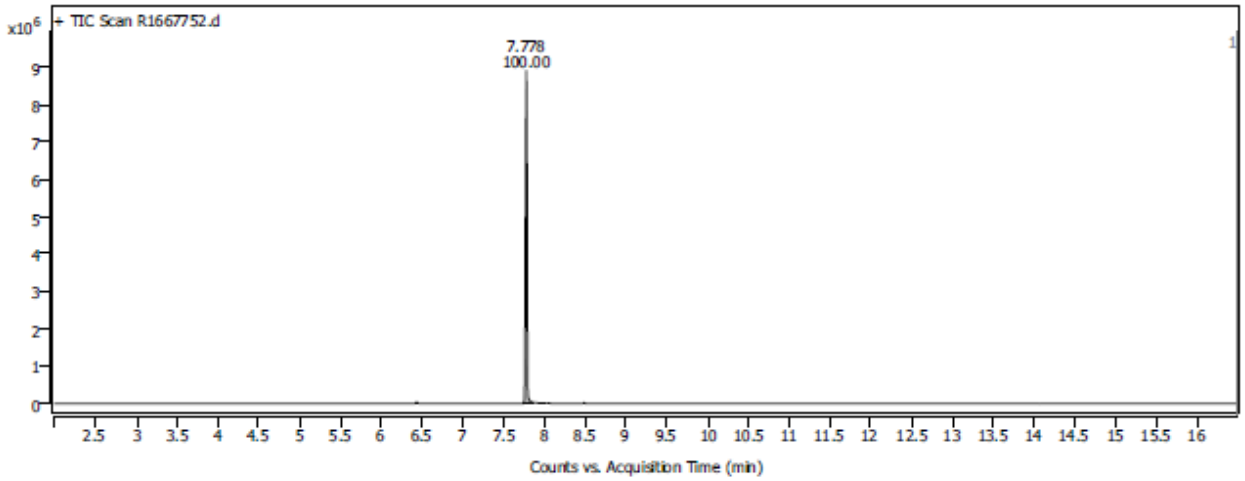
Analysis Report

Agilent | Trusted Answers

Sample Information

Name	R1667752	**Data File Path**	D:\MassHunter\GCMS\1\data\02_12\R1667752.D
Sample ID		**Acq. Time (Local)**	12-Feb-2022 51:13 (UTC+02:00)
Instrument	GCMS-5	**Method Path (Acq)**	D:\MassHunter\GCMS\1\methods\UNIV_ACN.M
Position	39		

Sample Chromatograms

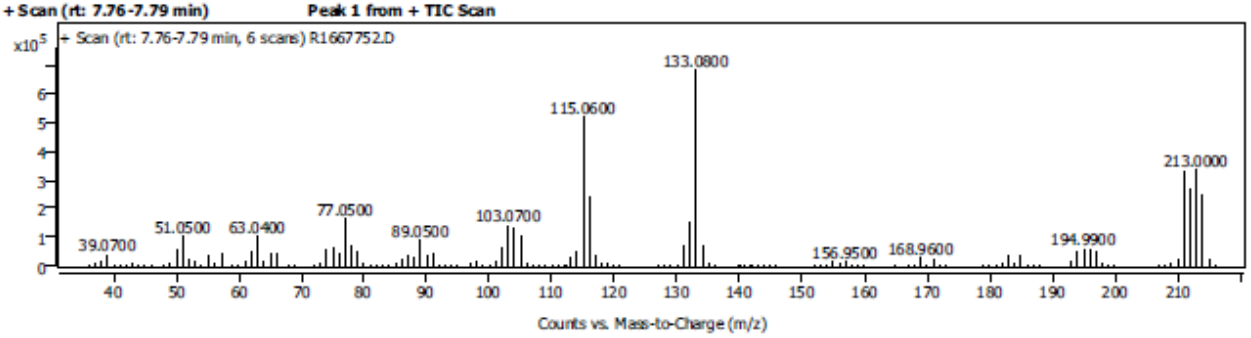


Chromatogram Peaks

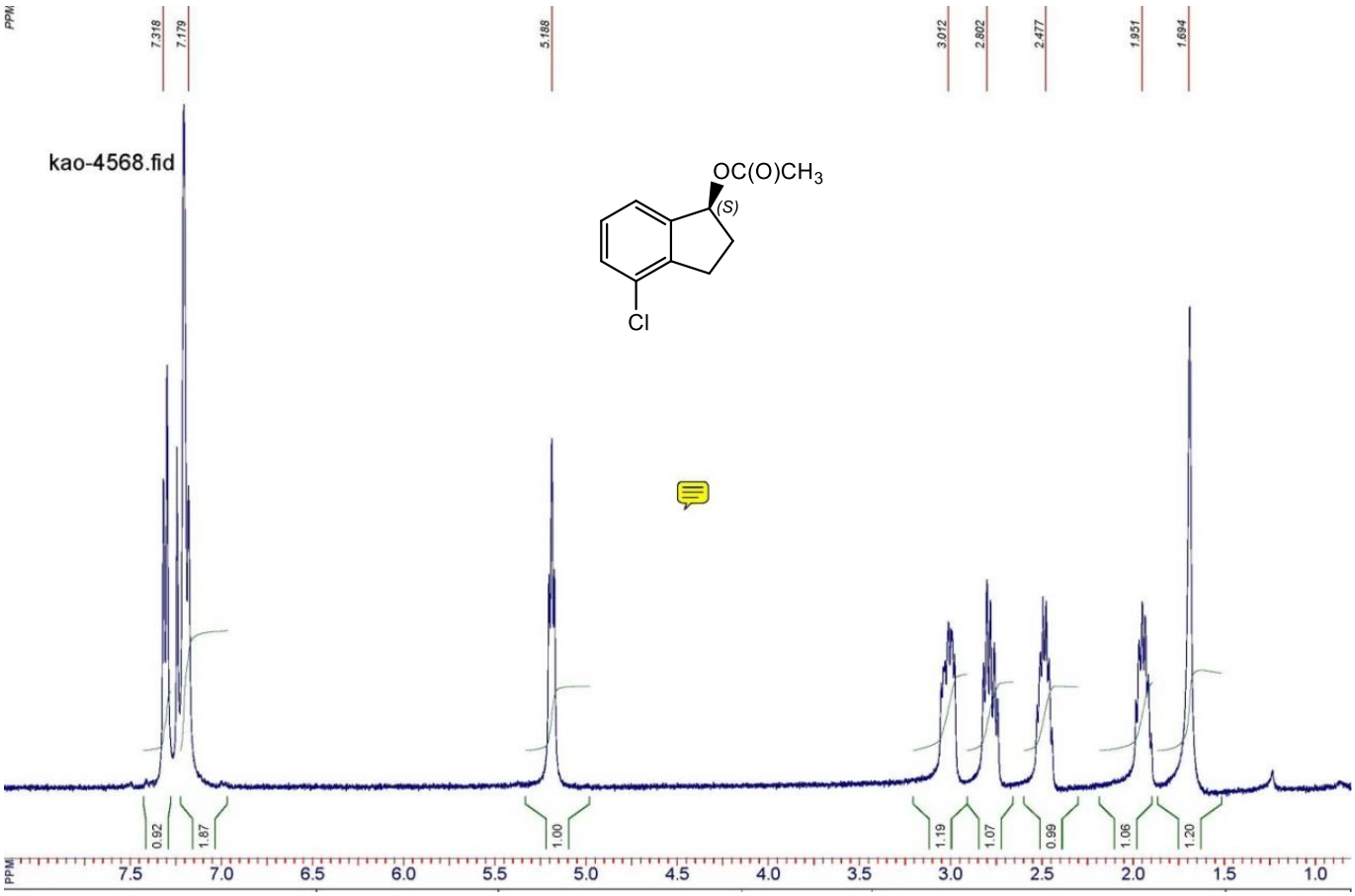
Peak	RT	Area	Area Sum %
1	7.778	13953682	100.00

Sample Spectra

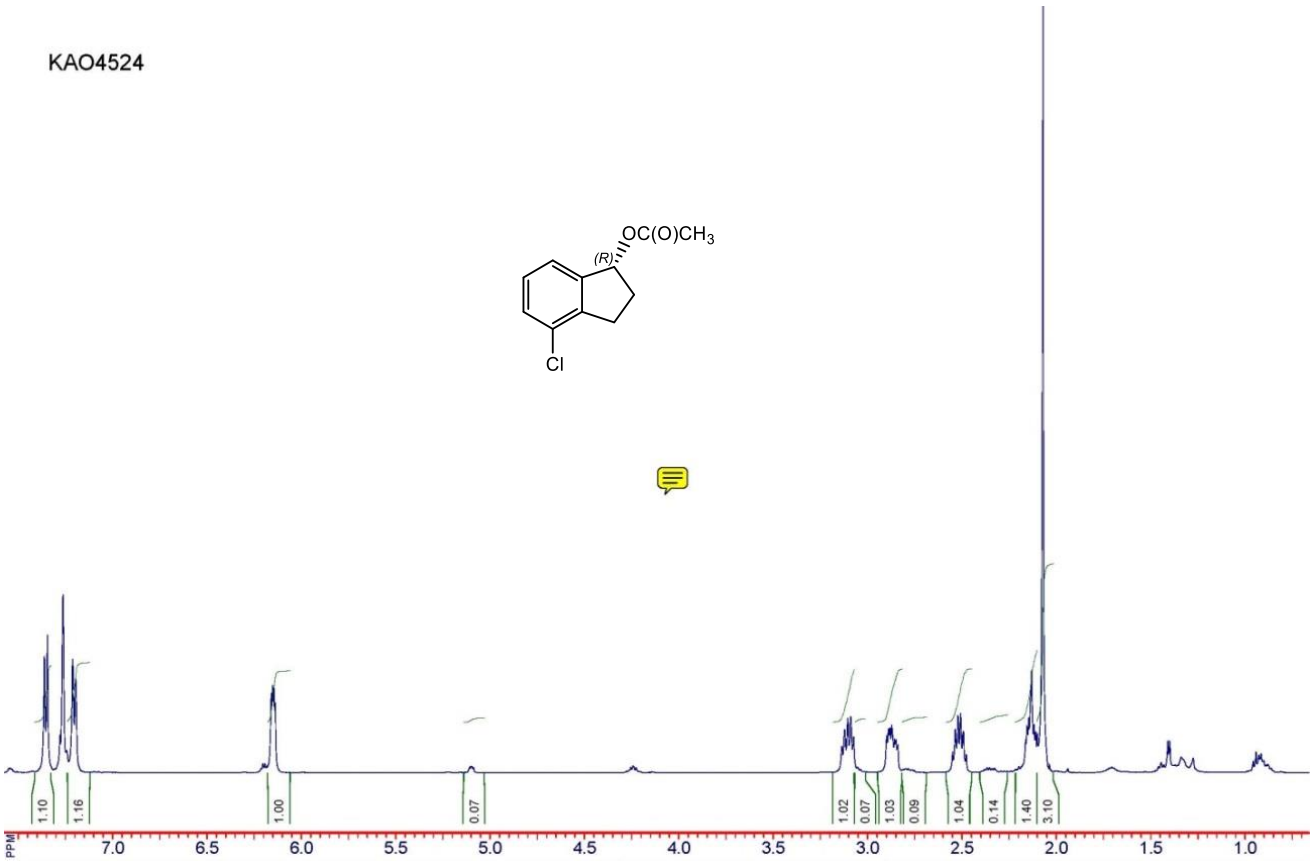
+ Scan (rt: 7.76-7.79 min) **Peak 1 from + TIC Scan**



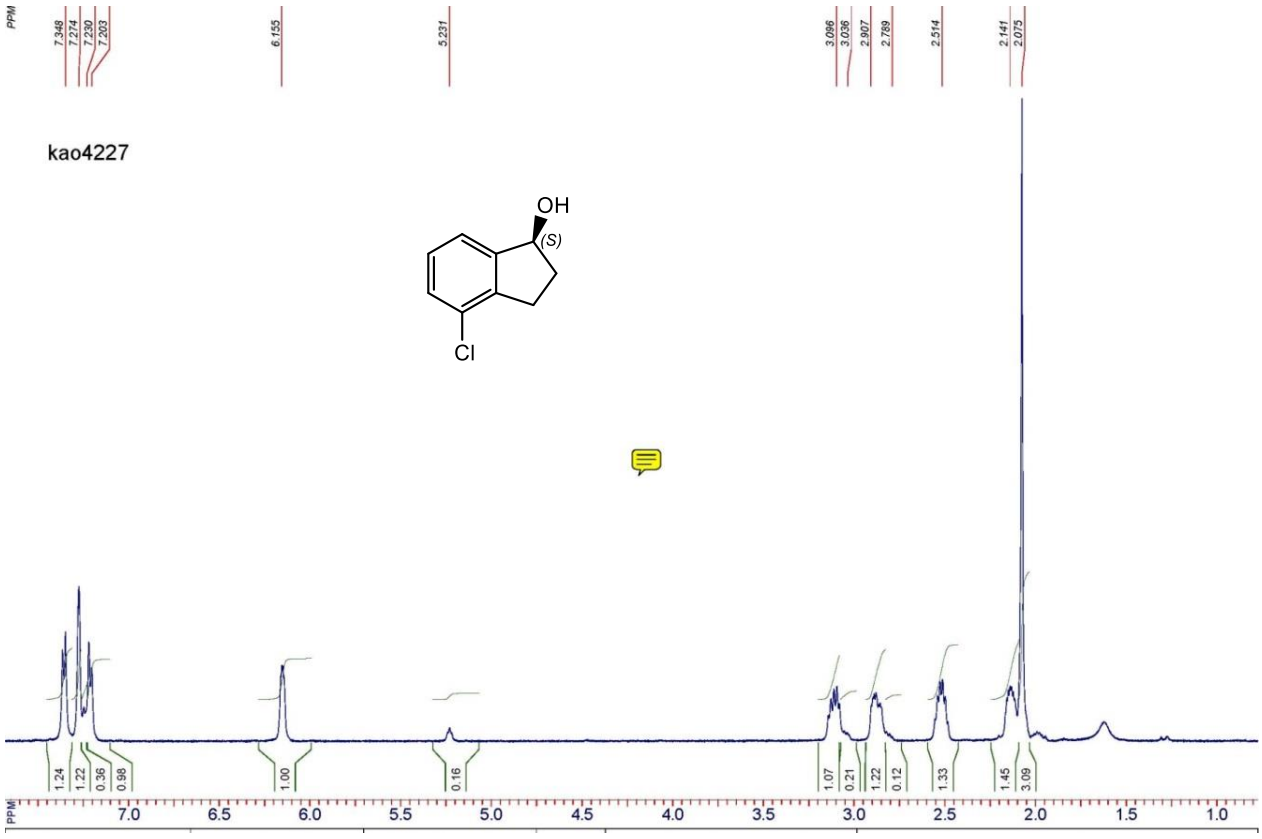
GCMS of (R) 4-bromo-2,3-dihydro-1H-inden-1-ol



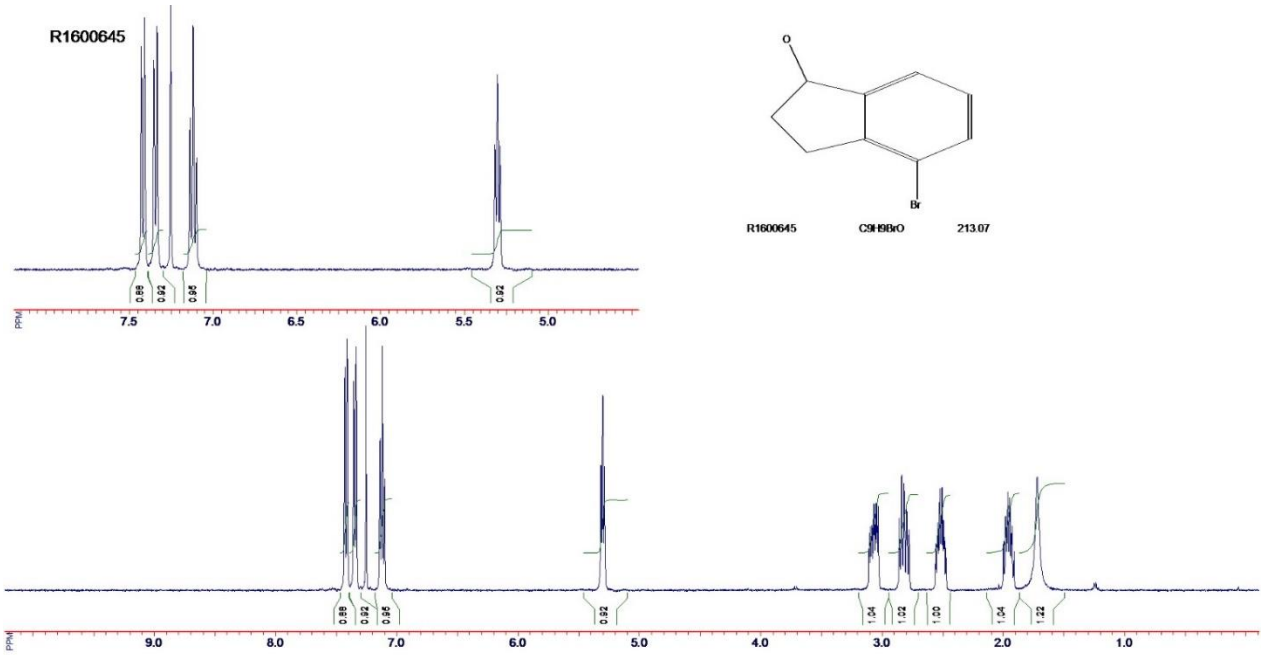
^{1}H NMR spectra of (*R*)-4-chloro-2,3-dihydro-1H-inden-1-ol resoved and crystallized



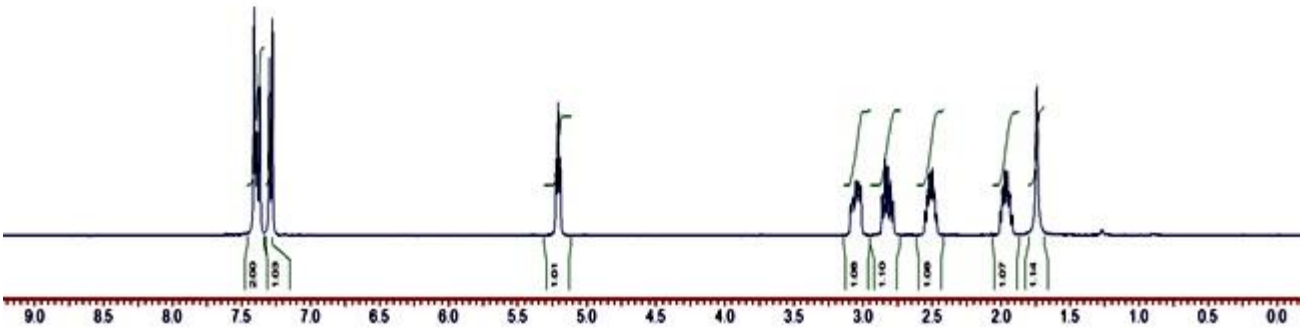
1H NMR spectra of (R)-4-chloro-2,3-dihydro-1H-inden-1-yl acetate



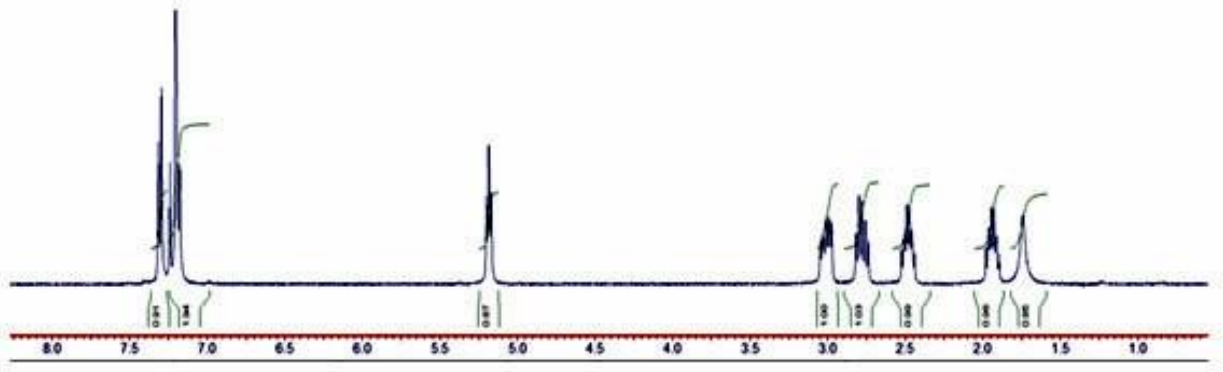
^{1}H NMR spectra of (S)-4-chloro-2,3-dihydro-1H-inden-1-yl acetate



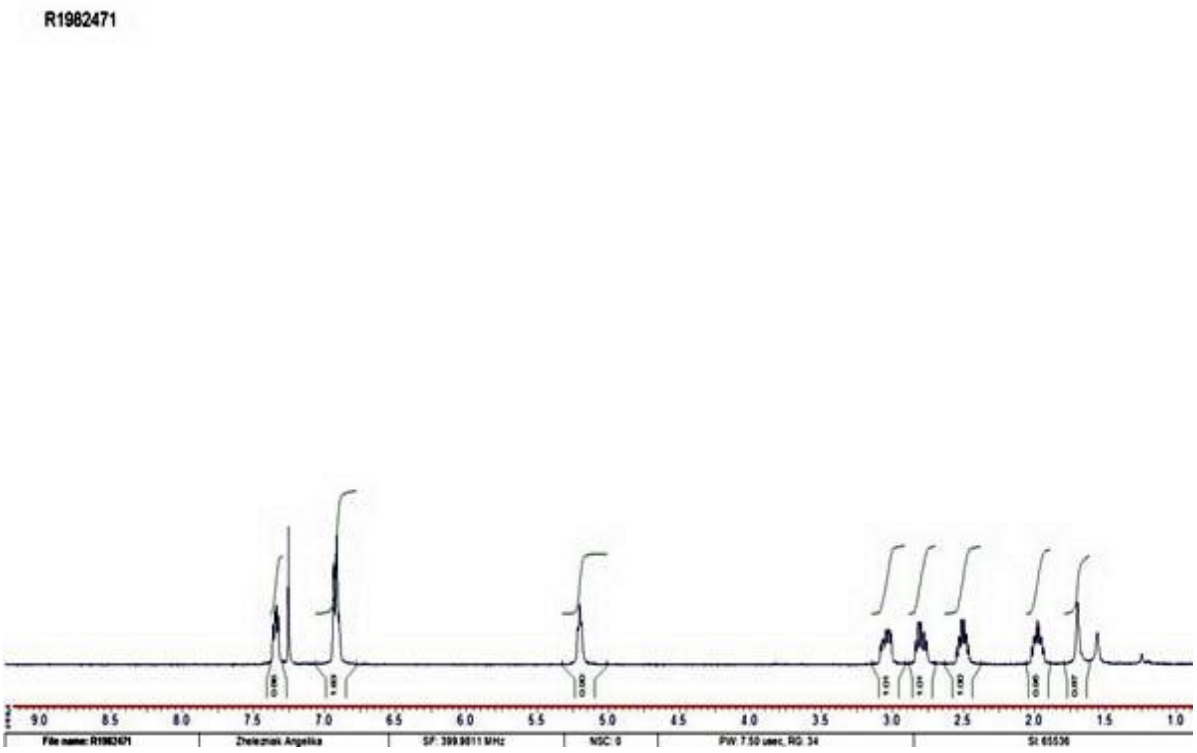
^{1}H NMR spectra of 1H NMR spectra of (*S*)-4-bromo-2,3-dihydro-1H-inden-1-ol



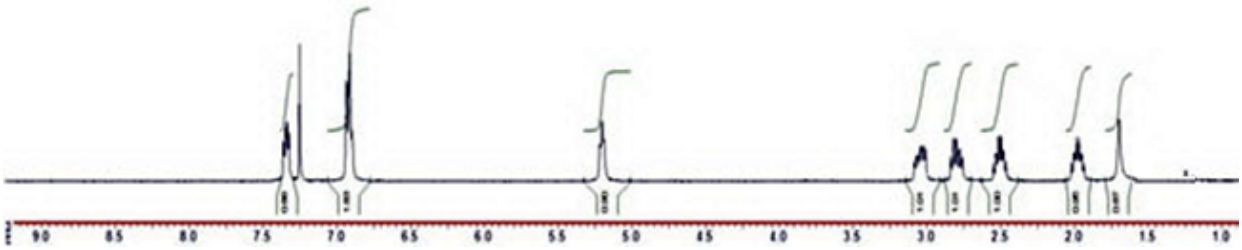
^{1}H NMR spectra of ^{1}H **NMR spectra of (S)-5-bromo-2,3-dihydro-1H-inden-1-ol**



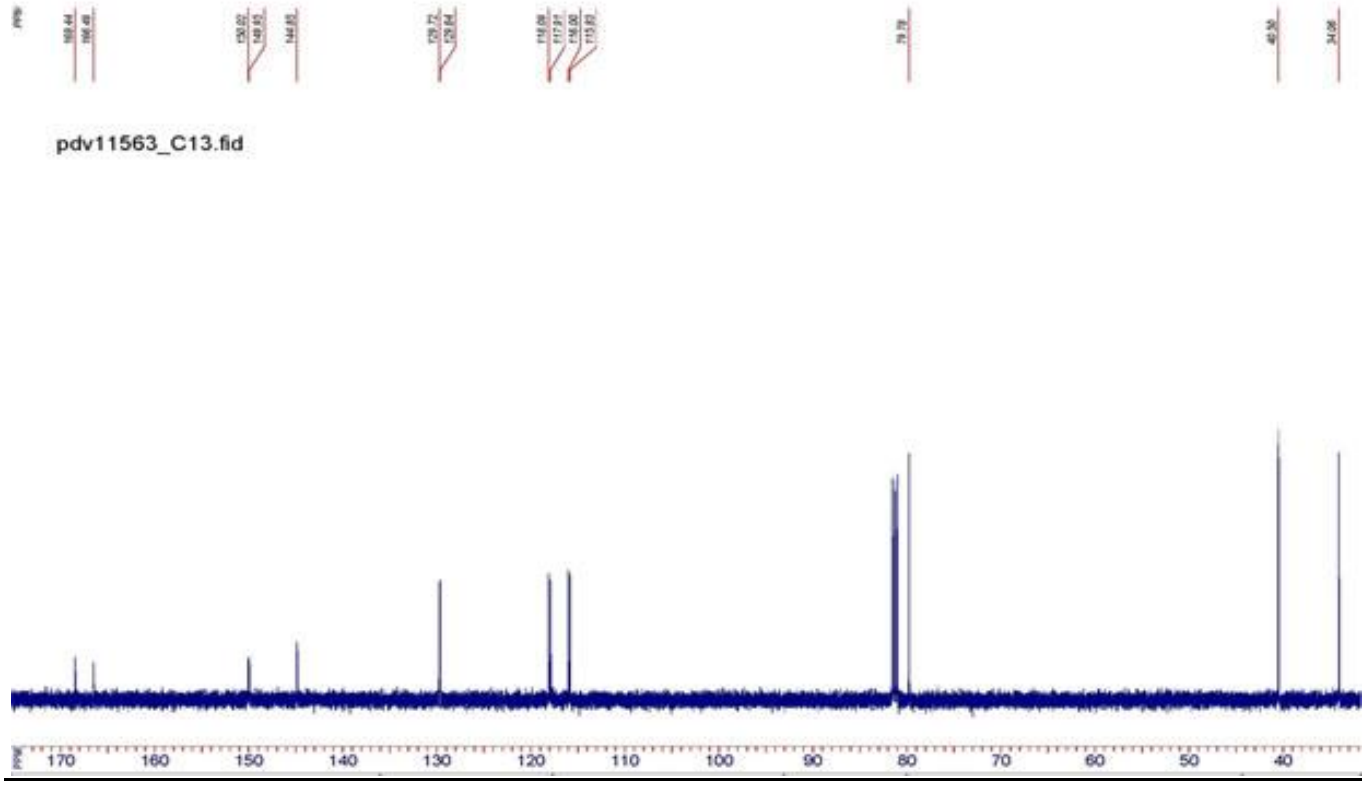
^{1}H NMR spectra of (R) 5-bromo-2,3-dihydro-1H-inden-1-ol



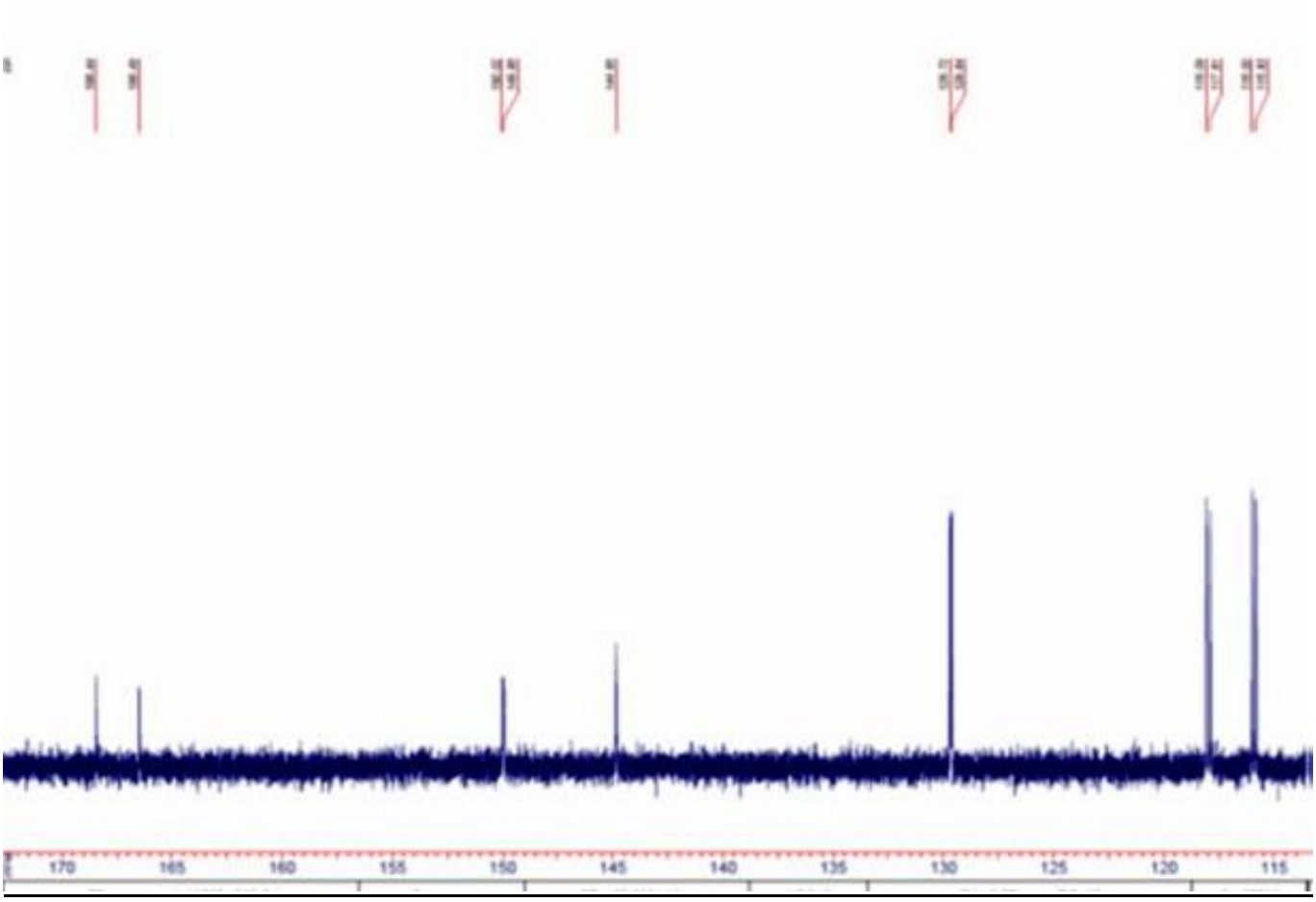
^{1}H NMR spectra of (S)-5-fluoro-2,3-dihydro-1H-inden-1-ol



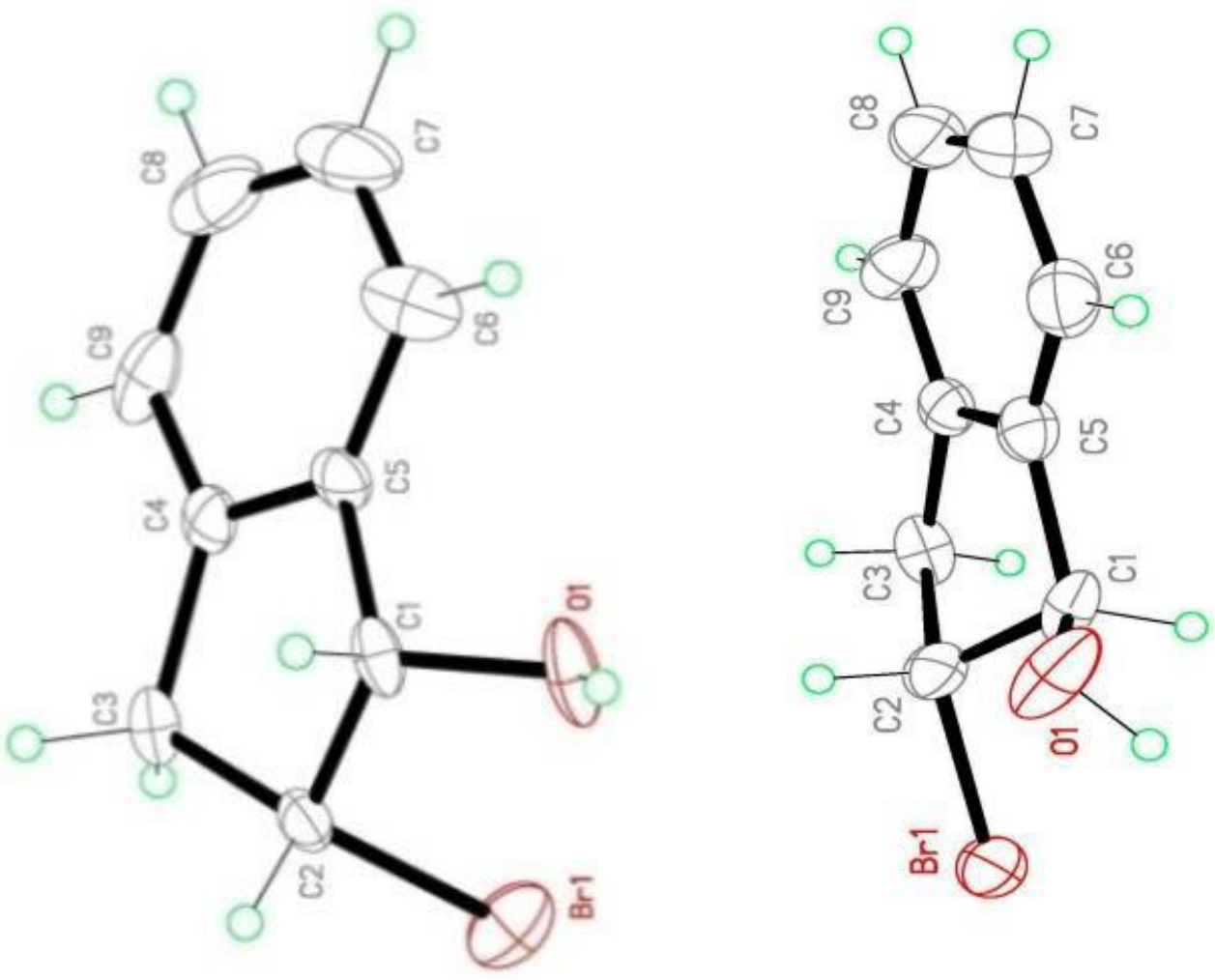
^{1}H NMR spectra of (R)-5-fluoro-2,3-dihydro-1H-inden-1-ol



^{13}C NMR spectra of (S)-4-fluor-2,3-dihydro-1H-inden-1-ol



^{13}C NMR spectra of (S)-5-fluoro-2,3-dihydro-1H-inden-1-ol (fragment)



A B

A) (1*S*,2*R*)-2-bromo-2,3-dihydro-1*H*-inden-1-ol; B) (1*R*,2*R*)-2-bromo-2,3-dihydro-1*H*-inden-1-ol [14]

