

Enantioselective Synthesis of Dihydroquinazolinone derivatives Catalyzed by Chiral Organocatalyst

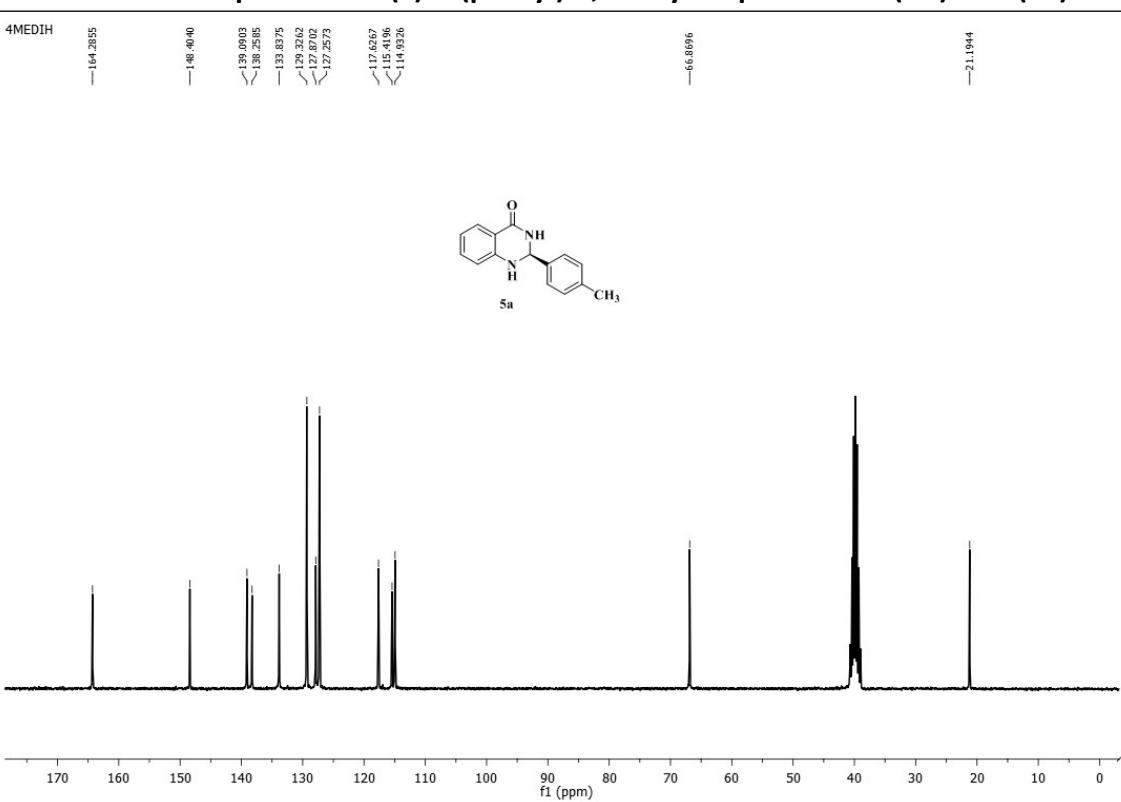
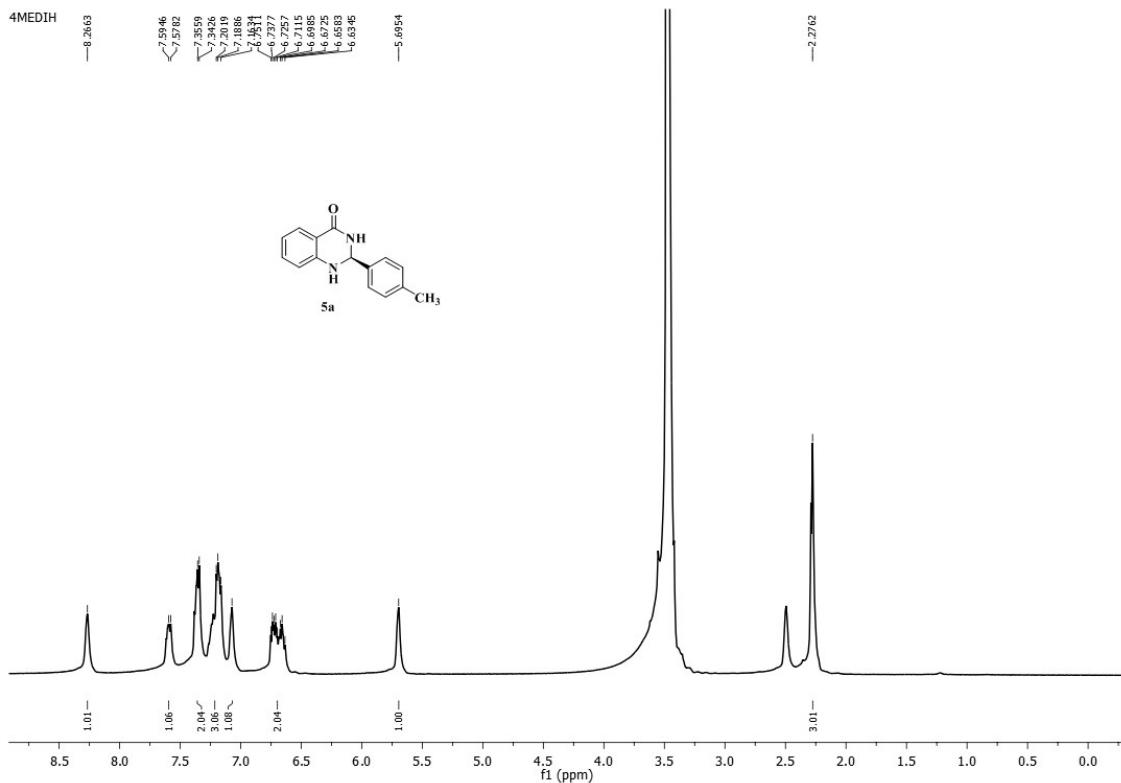
Ayyanar Siva*, Ponmuthu Kottala Vijaya, Madhappan Mariyappan, Veeramanoharan Ashokkumar, Velu Sadhasivam, Sankar Balakrishnan, Chithiraikumar Chinnadurai and Sepperumal Murugesan*

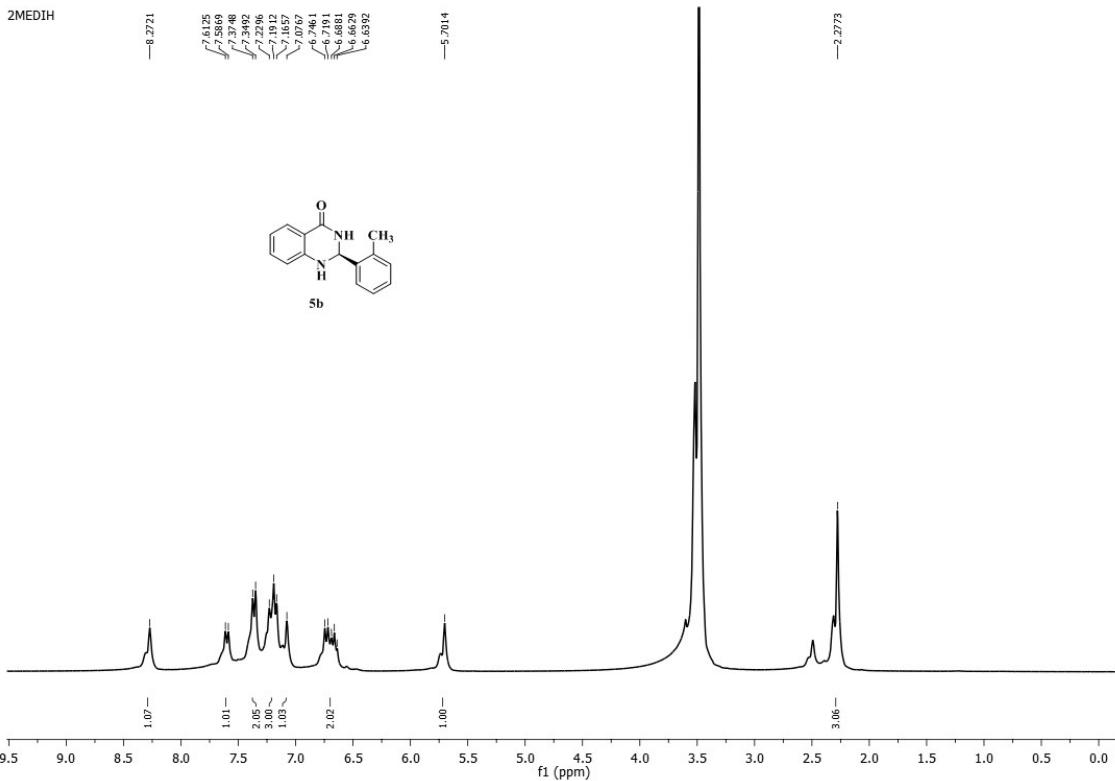
Supramolecular and Organometallic Chemistry Lab, Department of Inorganic Chemistry, School of Chemistry, Madurai Kamaraj University, Madurai-625 021, Tamilnadu, India.

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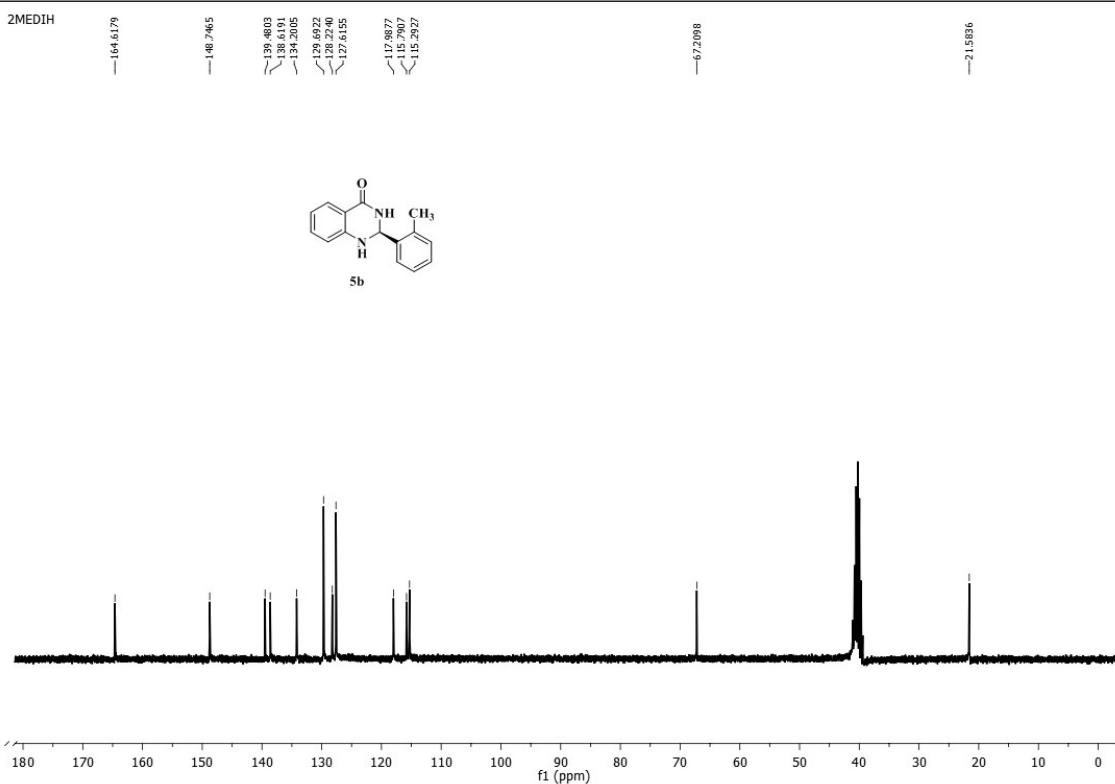
Content

| | Page.No |
|-------------------|----------------|
| NMR Spectra | 2-17 |
| HPLC Chromatogram | 18-41 |

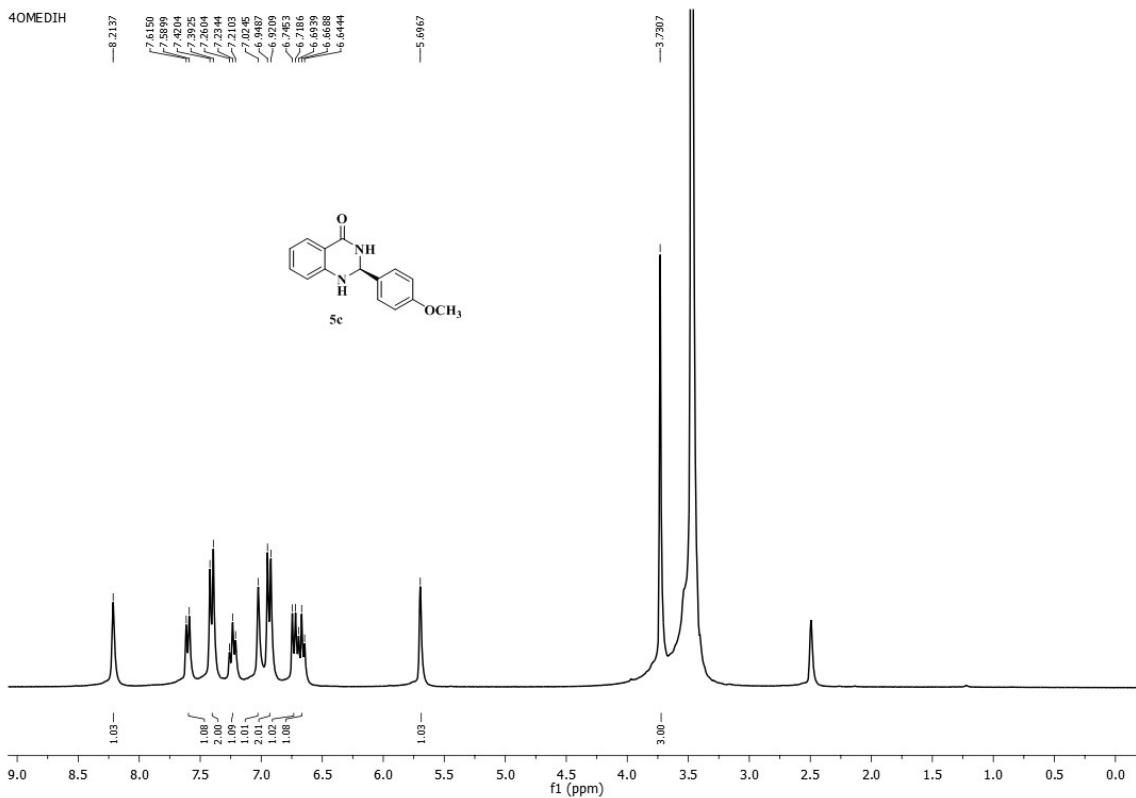




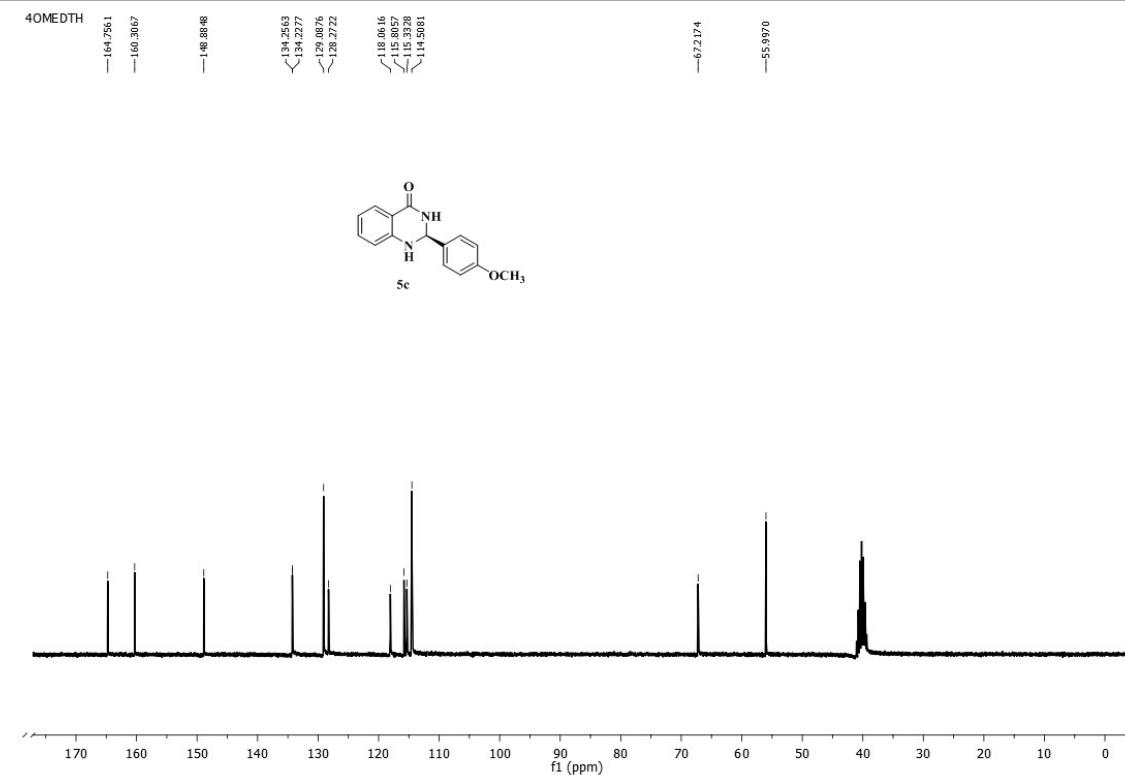
S 3: ^1H NMR Spectrum of (S)-2-(o-tolyl)-2,3-dihydroquinazolin-4(1H)-one (5b).



S 4: ^{13}C NMR Spectrum of (S)-2-(o-tolyl)-2,3-dihydroquinazolin-4(1H)-one (5b).



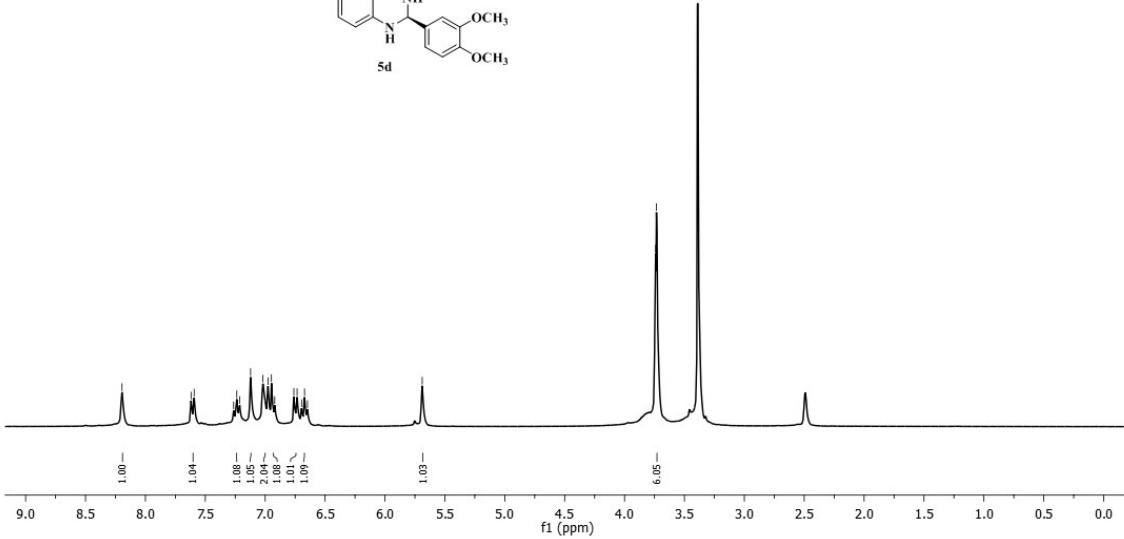
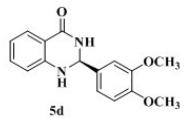
S 5: ^1H NMR Spectrum of (S)-2-(4-methoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one (5c).



S 6: ^{13}C NMR Spectrum of (S)-2-(4-methoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one (5c).

VDIH

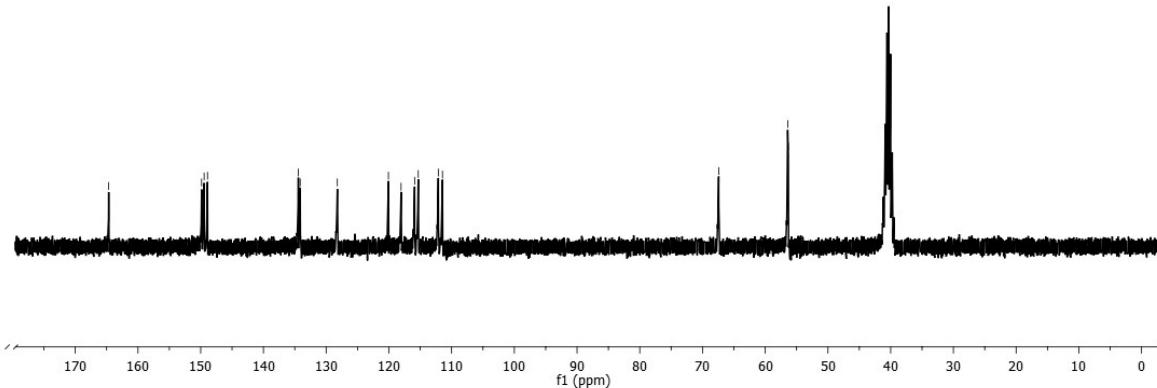
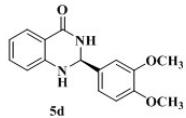
—8.1955
 $\text{---}^2\text{J}$ 6.189
 $\text{---}^2\text{J}$ 159.33
 $\text{---}^2\text{J}$ 26.97
 $\text{---}^2\text{J}$ 23.71
 $\text{---}^2\text{J}$ 2.28
 $\text{---}^2\text{J}$ 1.28
 $\text{---}^2\text{J}$ 0.93
 $\text{---}^4\text{J}$ 9.766
 $\text{---}^4\text{J}$ 9.79
 $\text{---}^4\text{J}$ 9.006
 $\text{---}^4\text{J}$ 8.766
 $\text{---}^4\text{J}$ 8.664
 $\text{---}^4\text{J}$ 6.716
 $\text{---}^4\text{J}$ 6.671
 $\text{---}^4\text{J}$ 5.6371
 $\text{---}^5\text{J}$ 6.671
 $\text{---}^5\text{J}$ 3.7336



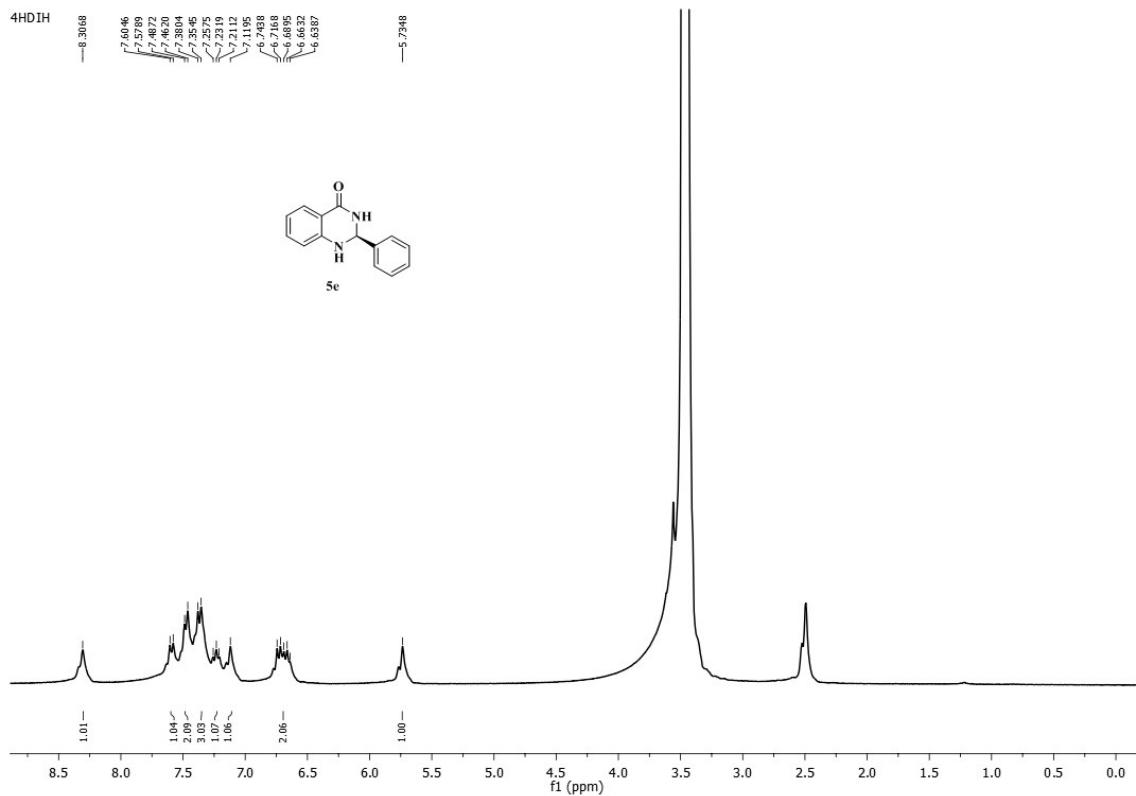
S 7: ^1H NMR Spectrum of (*S*)-2-(3,4-dimethoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one (5d).

VDIH

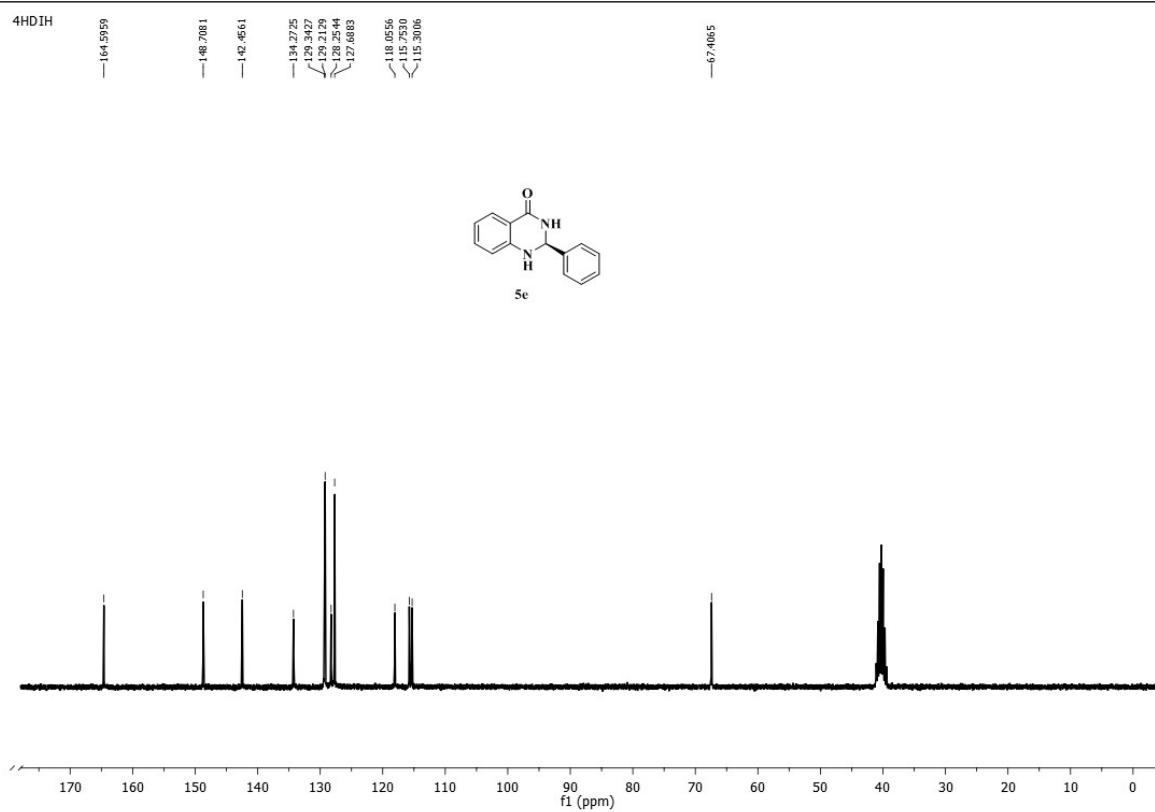
—164.6799
 $\text{---}^2\text{J}$ 149.8585
 $\text{---}^2\text{J}$ 149.4642
 $\text{---}^2\text{J}$ 148.9152
 $\text{---}^2\text{J}$ 134.4203
 $\text{---}^2\text{J}$ 134.1399
 $\text{---}^2\text{J}$ 128.2171
 $\text{---}^2\text{J}$ 120.0739
 $\text{---}^2\text{J}$ 118.0551
 $\text{---}^2\text{J}$ 115.8964
 $\text{---}^2\text{J}$ 115.3172
 $\text{---}^2\text{J}$ 112.1176
 $\text{---}^2\text{J}$ 111.4677
 $\text{---}^4\text{J}$ 67.4208
 $\text{---}^4\text{J}$ 56.4184
 $\text{---}^4\text{J}$ 56.3150



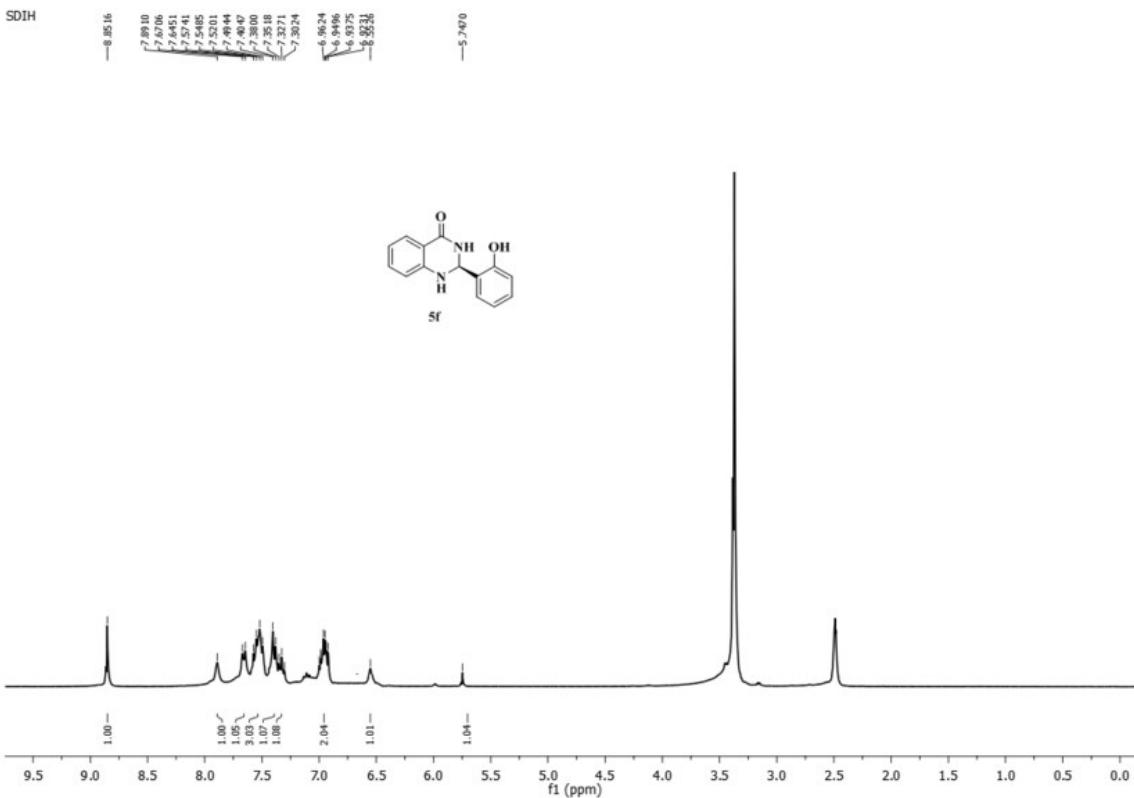
S 8: ^{13}C NMR Spectrum of (*S*)-2-(3,4-dimethoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one (5d).



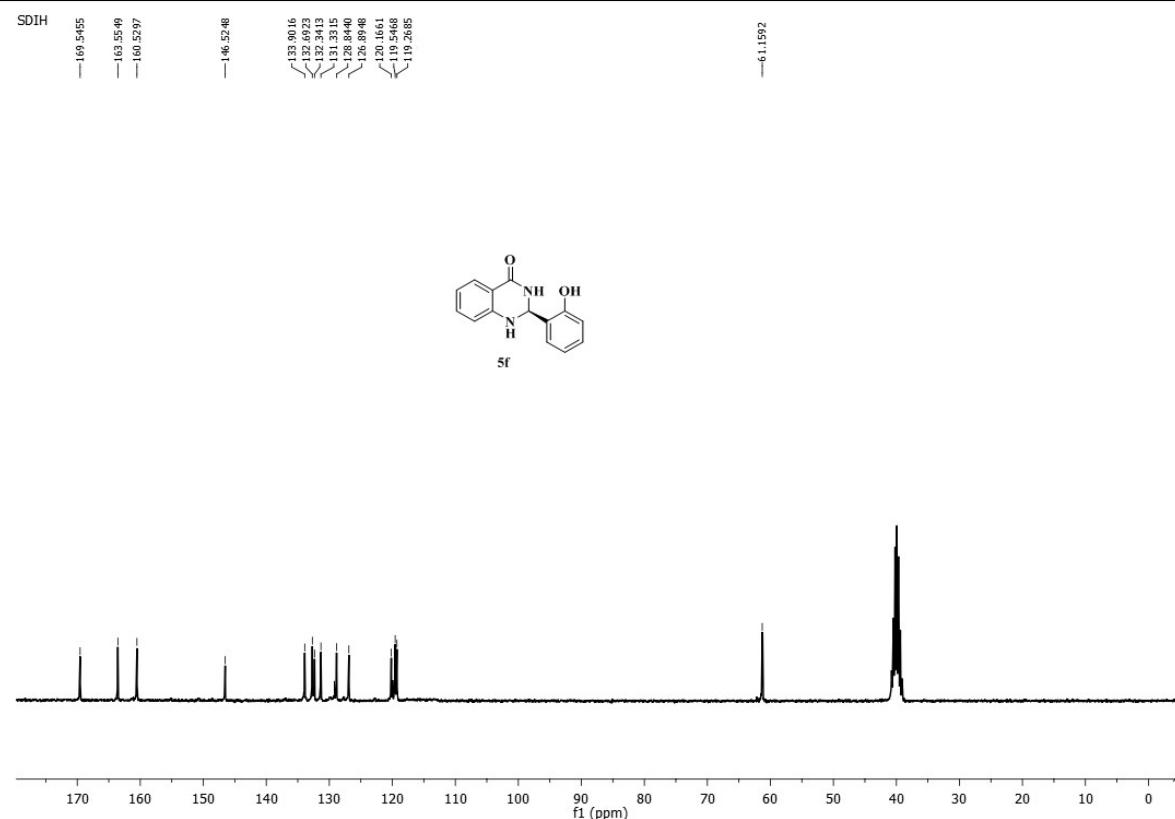
S 9: ^1H NMR Spectrum of (S)-2-phenyl-2,3-dihydroquinazolin-4(1H)-one (5e).



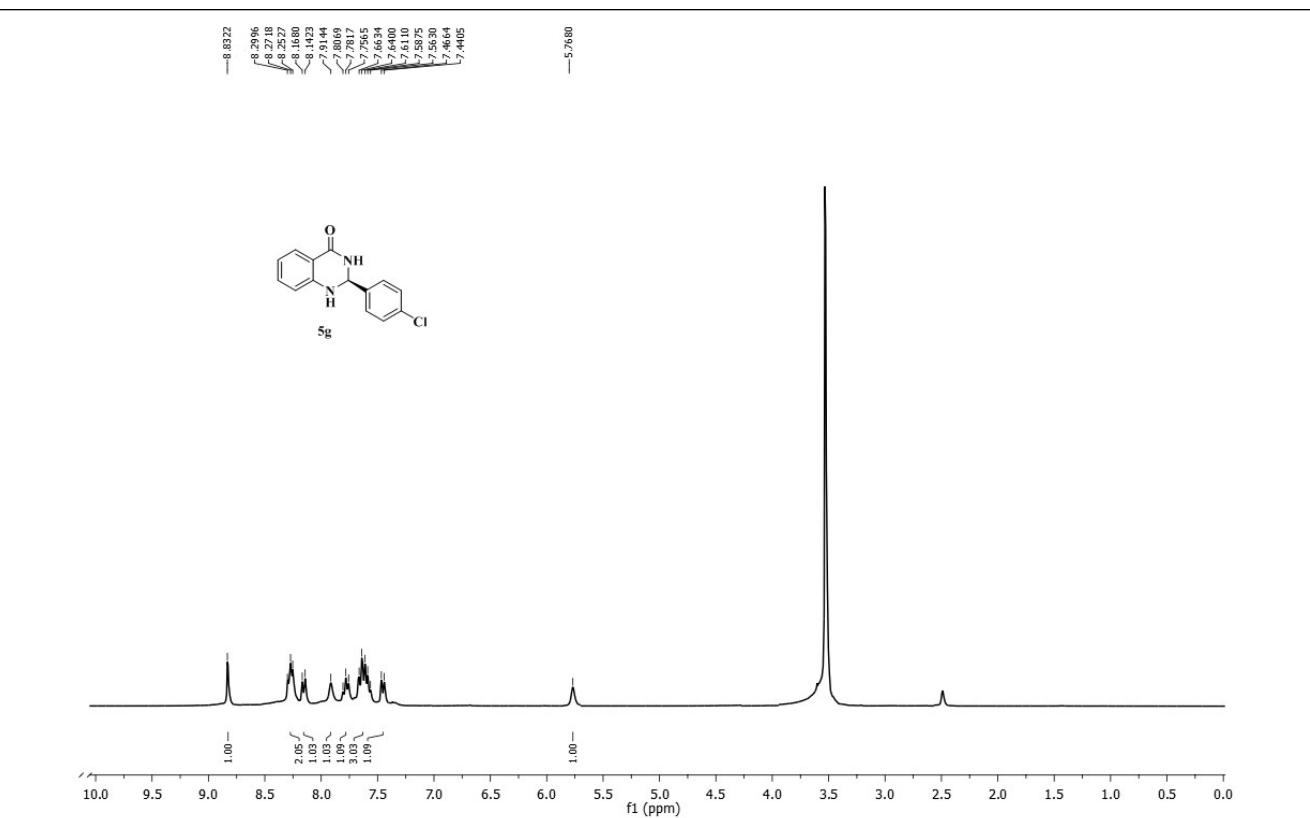
S 10: ^{13}C NMR Spectrum of (S)-2-phenyl-2,3-dihydroquinazolin-4(1H)-one (5e).



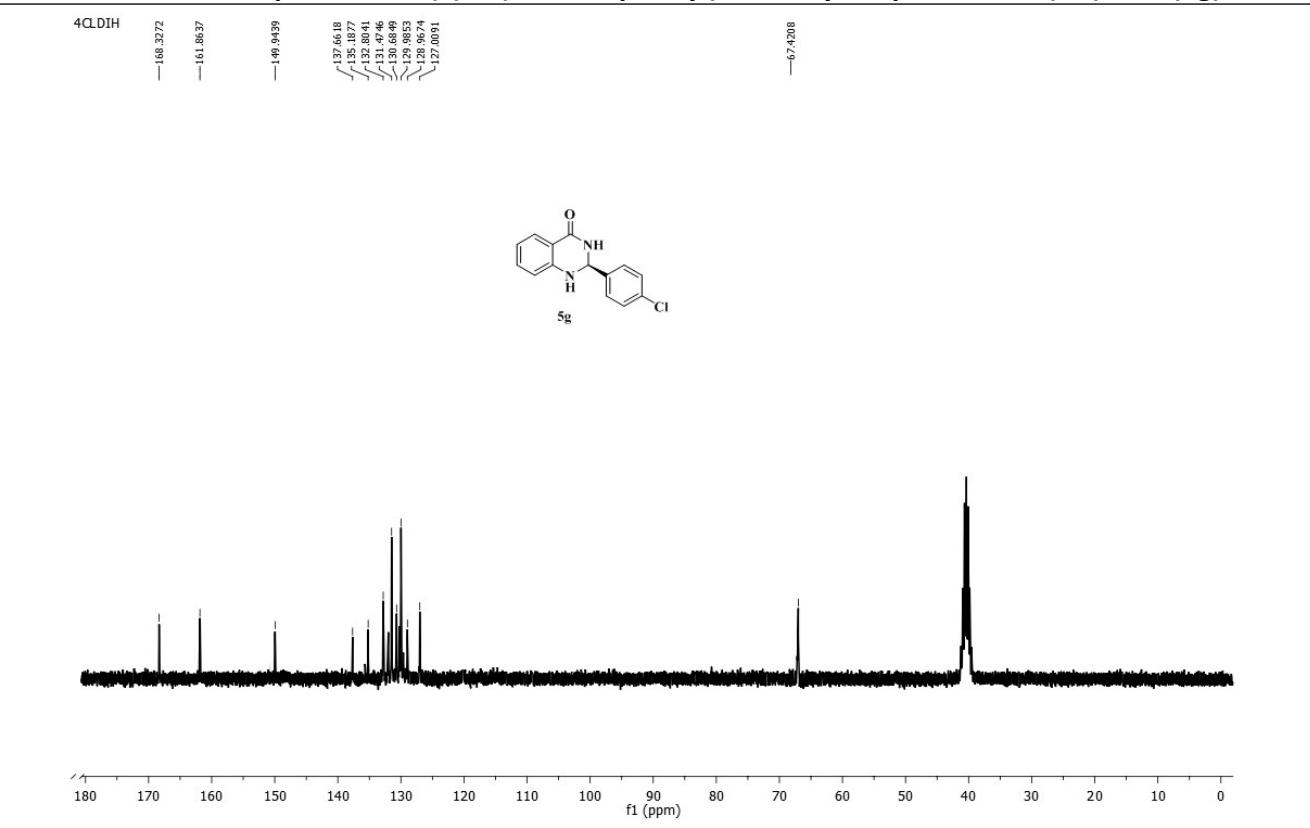
S 11: ^1H NMR Spectrum of (S)-2-(2-hydroxyphenyl)-2,3-dihydroquinazolin-4(1H)-one (5f).



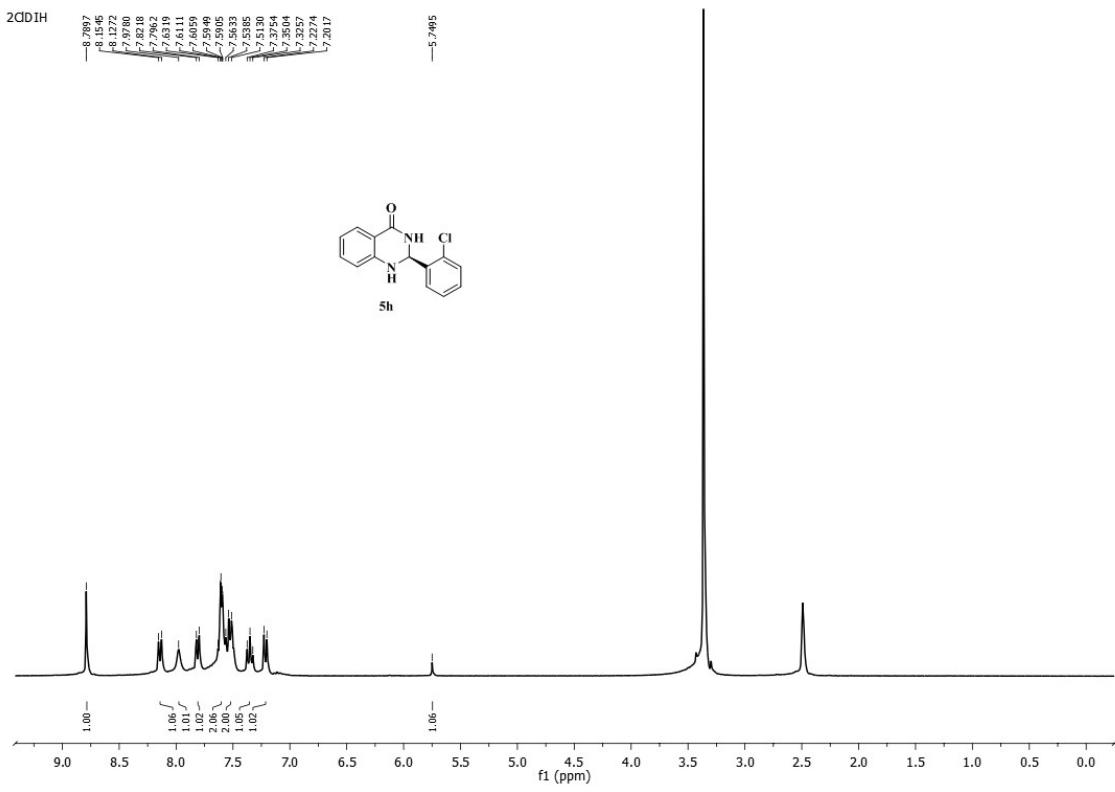
S 12: ^{13}C NMR Spectrum of (*S*)-2-(2-hydroxyphenyl)-2,3-dihydroquinazolin-4(1H)-one (5f).



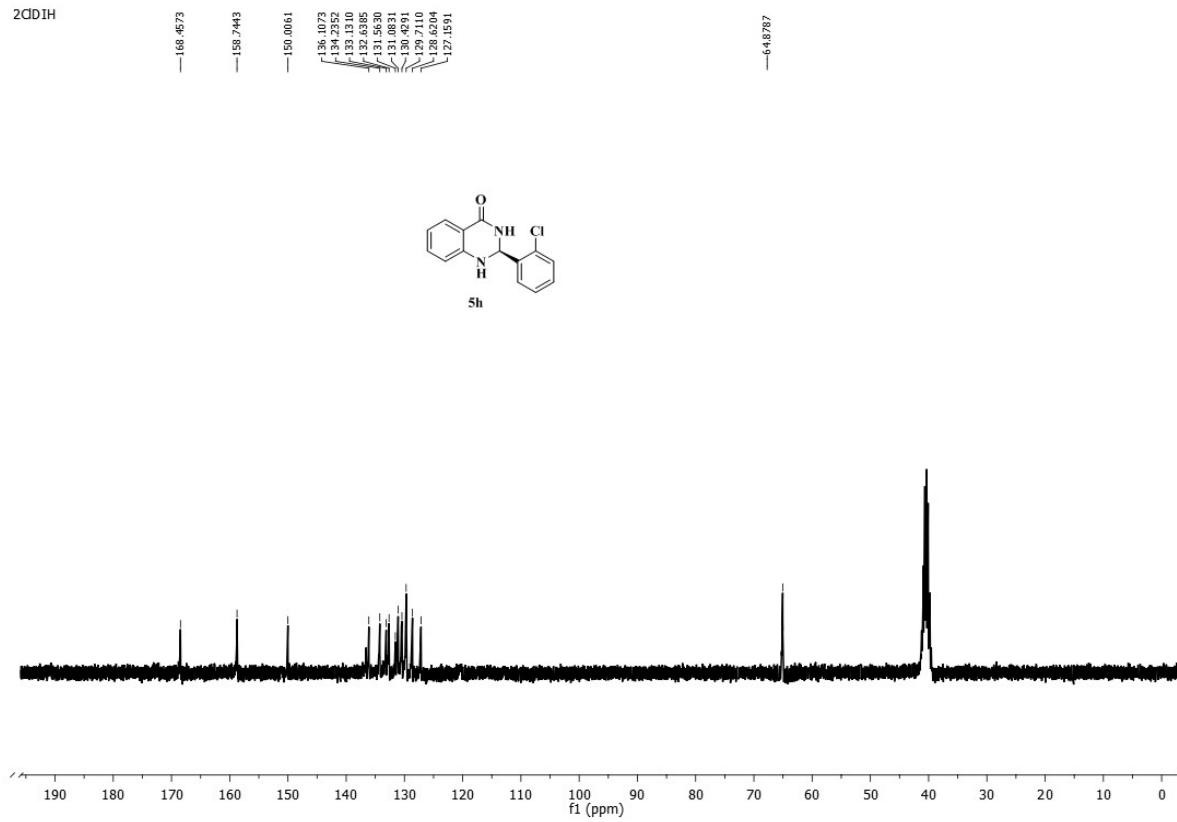
S 13: ¹H NMR Spectrum of (S)-2-(4-chlorophenyl)-2,3-dihydroquinazolin-4(1H)-one (5g).



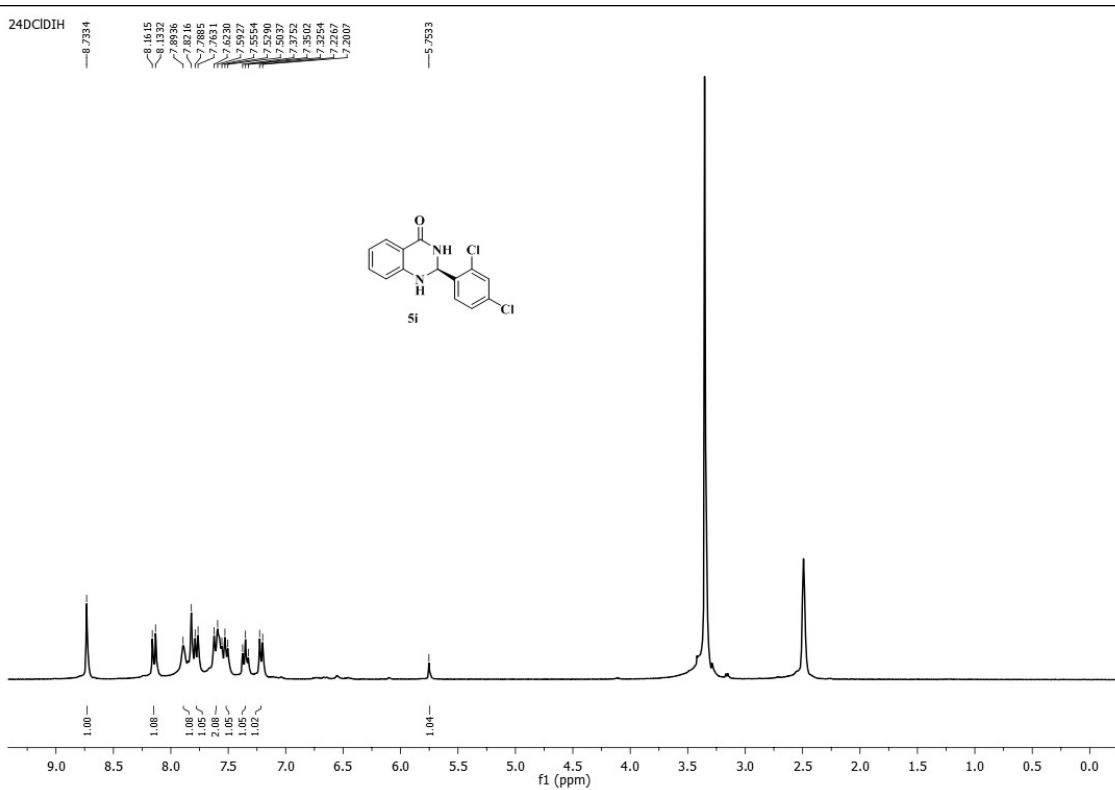
S 14: ¹³C NMR Spectrum of (S)-2-(4-chlorophenyl)-2,3-dihydroquinazolin-4(1H)-one (5g).



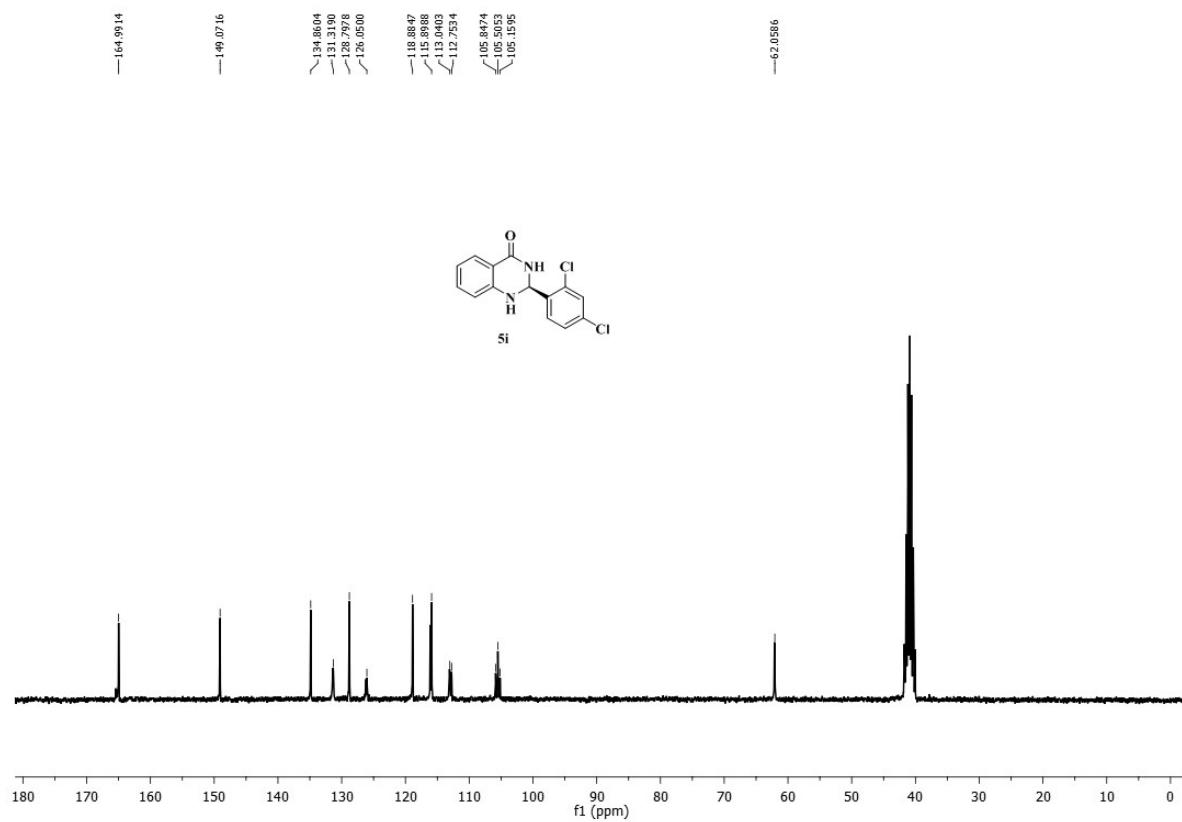
S 15: ^1H NMR Spectrum of (*S*)-2-(2-chlorophenyl)-2,3-dihydroquinazolin-4(1H)-one (5h).



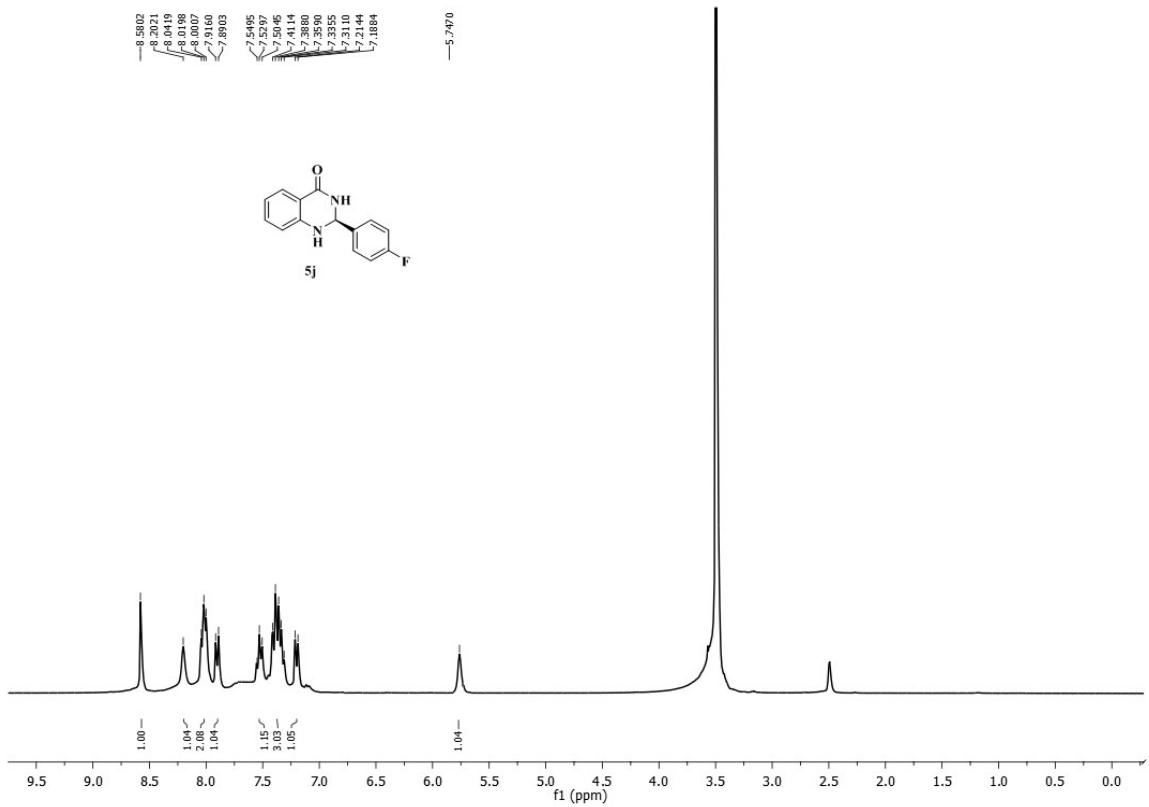
S 16: ^{13}C NMR Spectrum of (*S*)-2-(2-chlorophenyl)-2,3-dihydroquinazolin-4(1H)-one (5h).



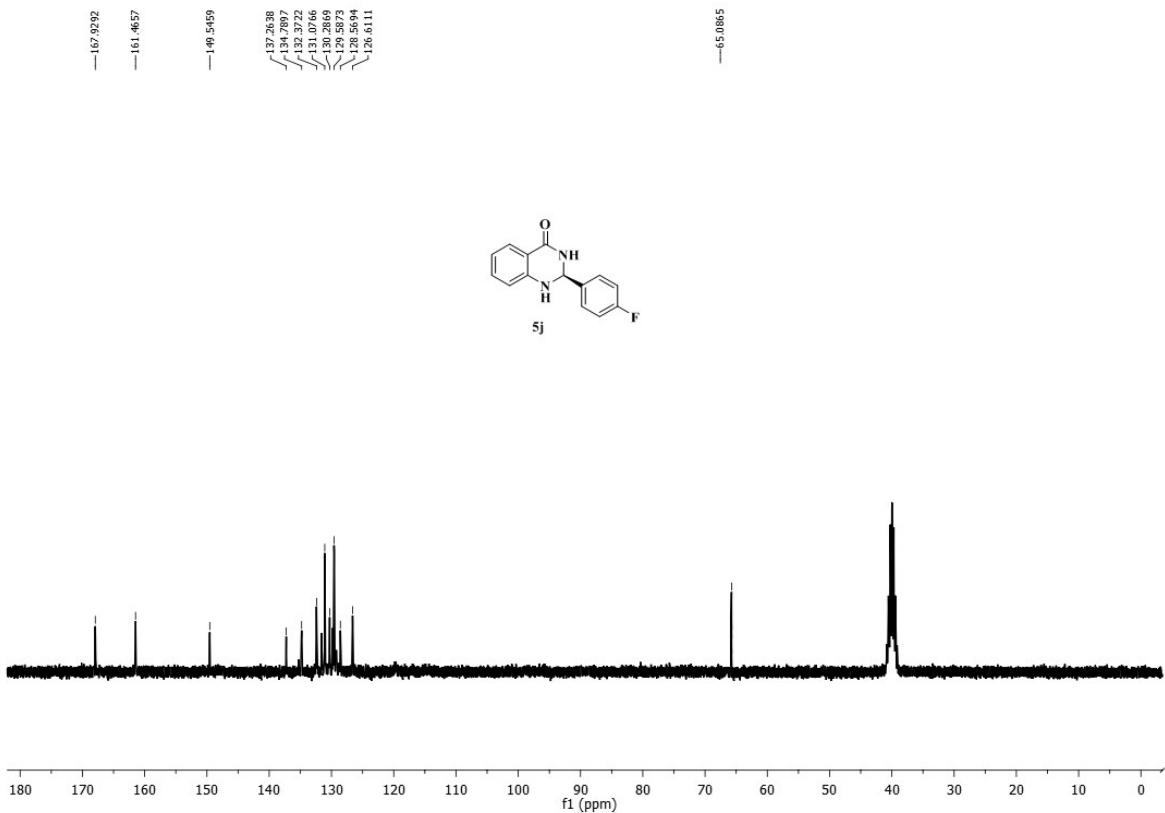
S 17: ^1H NMR Spectrum of (*S*)-2-(2,4-dichlorophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (5i).



S 18: ^{13}C NMR Spectrum of (*S*)-2-(2,4-dichlorophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (5i).

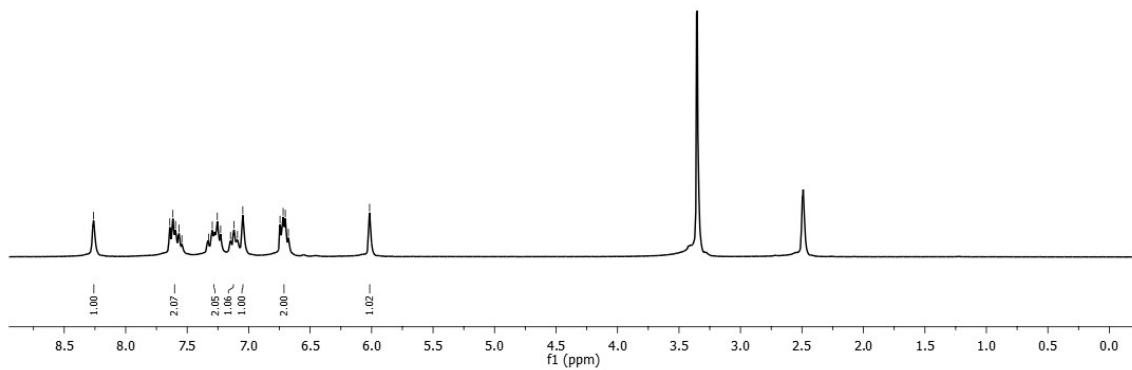
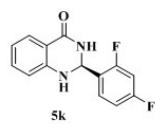


S 19: ^1H NMR Spectrum of (S)-2-(4-fluorophenyl)-2,3-dihydroquinazolin-4(1H)-one (5j).



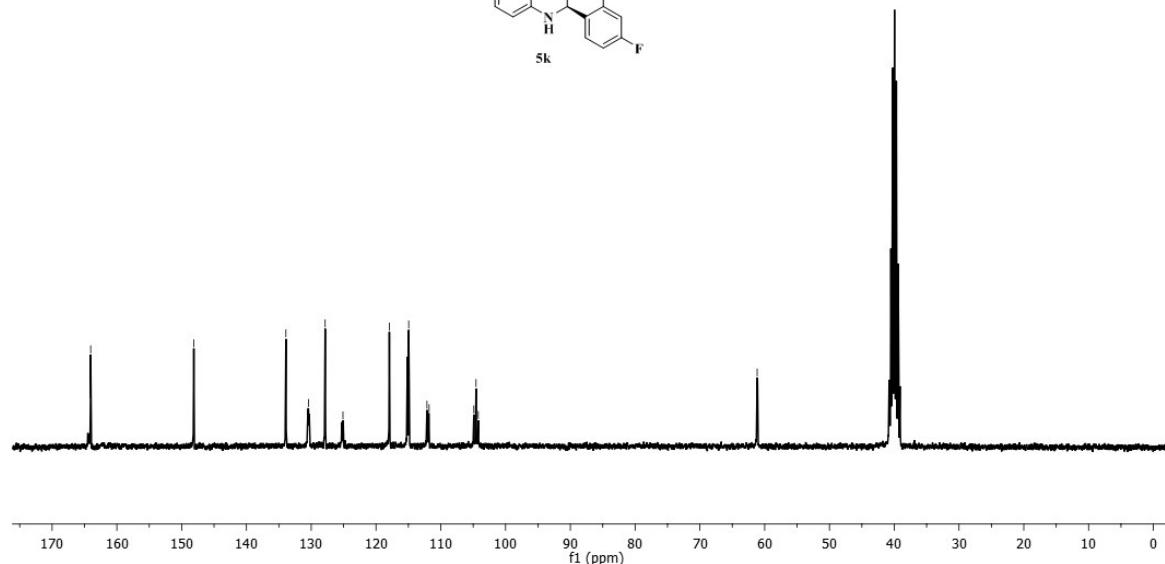
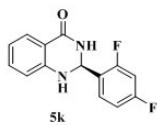
S 20: ^{13}C NMR Spectrum of (*S*)-2-(4-fluorophenyl)-2,3-dihydroquinazolin-4(1H)-one (5j).

—8.26¹³
—7.6113
—7.6160
—7.5946
—7.2961
—7.2957
—7.2949
—7.2277
—7.1475
—7.1194
—7.0909
—6.9326
—6.7196
—6.6767
—6.0162



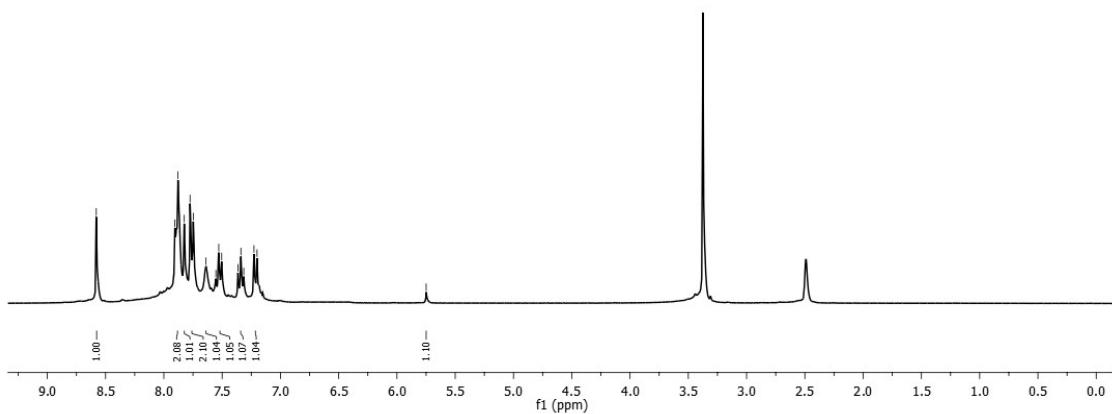
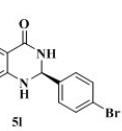
S 21: ^1H NMR Spectrum of (*S*)-2-(2,4-difluorophenyl)-2,3-dihydroquinazolin-4(1H)-one (5k).

—164.0424
—148.1226
—133.9114
—130.4511
—127.8688
—125.1010
—117.9357
—114.9498
—112.1356
—111.8044
—104.8984
—104.5563
—104.2105
—6.11592



S 22: ^{13}C NMR Spectrum of (*S*)-2-(2,4-difluorophenyl)-2,3-dihydroquinazolin-4(1H)-one (5k).

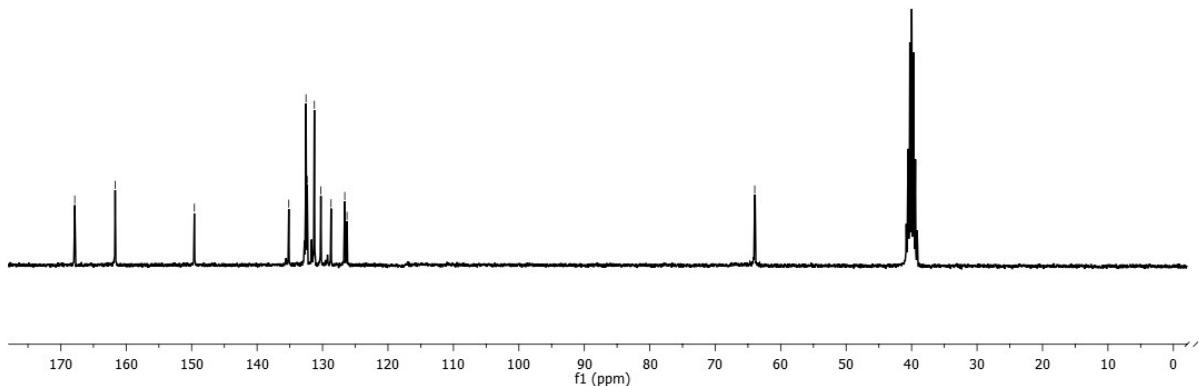
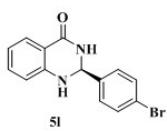
—8.5798
—8.5054
—7.8788
—7.8551
—7.7750
—7.7474
—7.6386
—7.5388
—7.5089
—7.3938
—7.3841
—7.3389
—7.3141
—7.2267
—7.2006



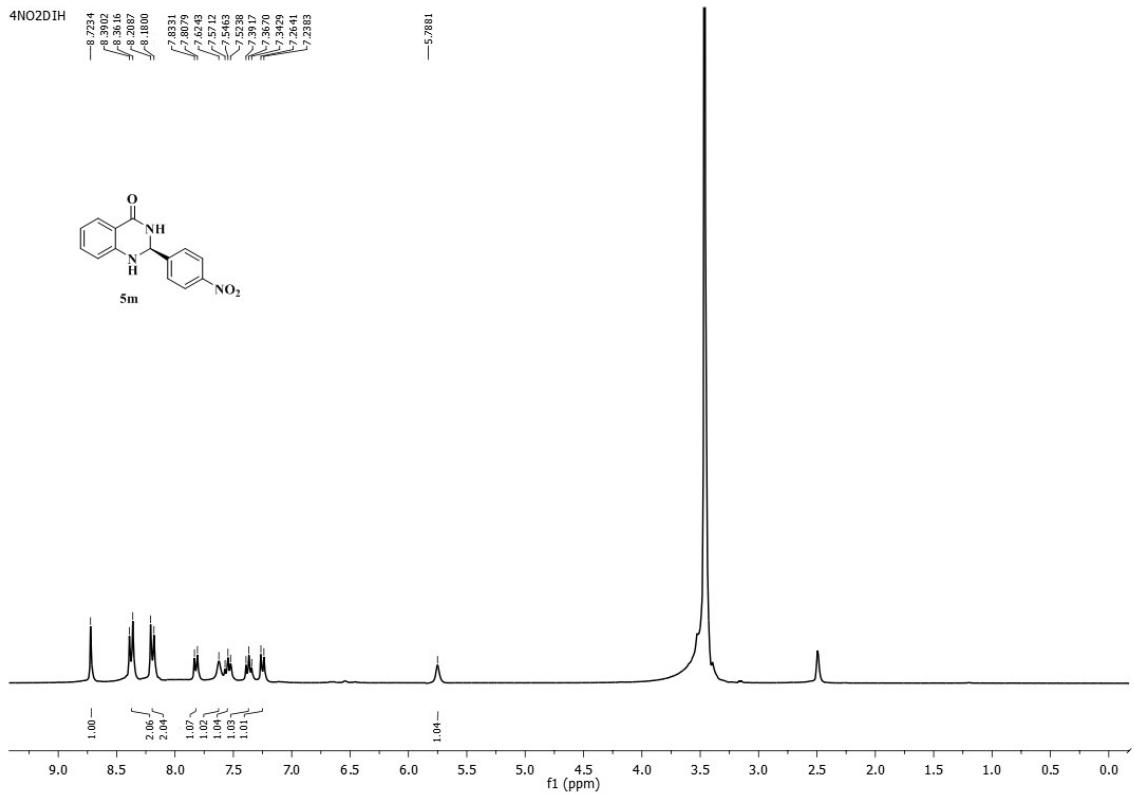
S 23: ^1H NMR Spectrum of (*S*)-2-(4-bromophenyl)-2,3-dihydroquinazolin-4(1H)-one (5l).

—167.8527
—161.6796
—149.5797
—135.1563
—132.5278
—132.3484
—131.2484
—130.2558
—126.6023
—126.2576

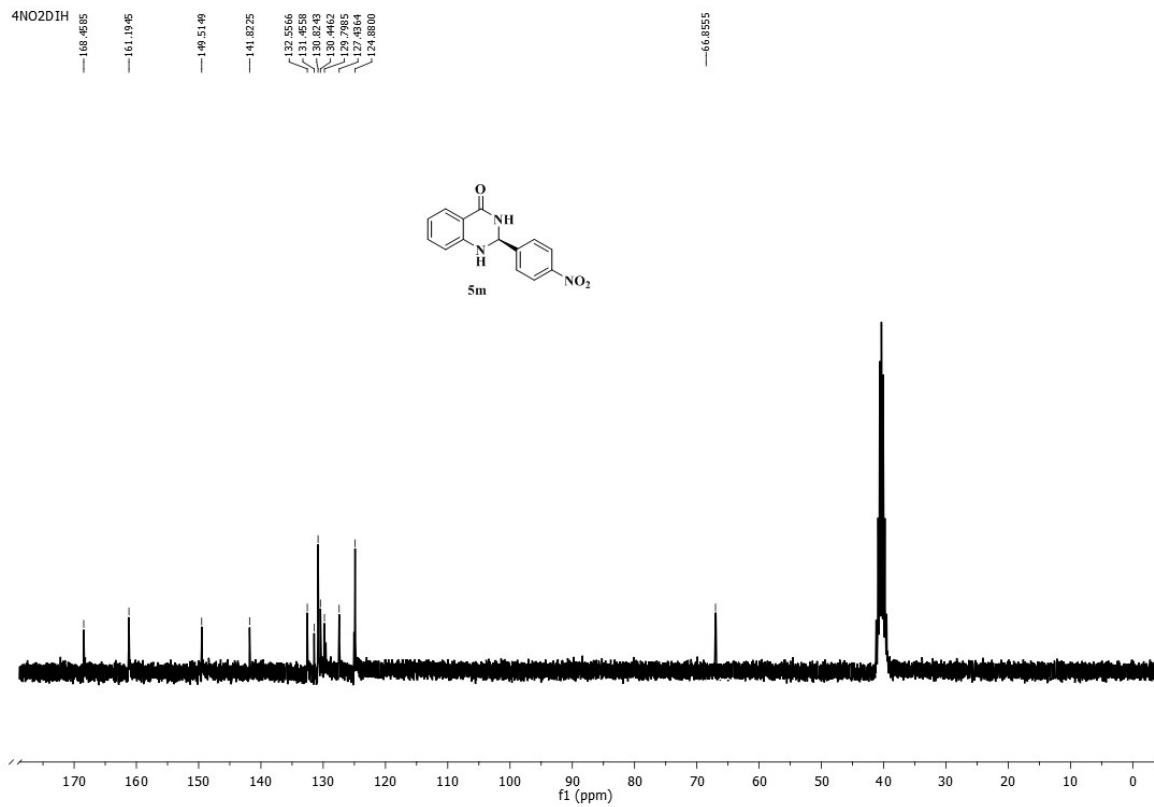
—64.1367



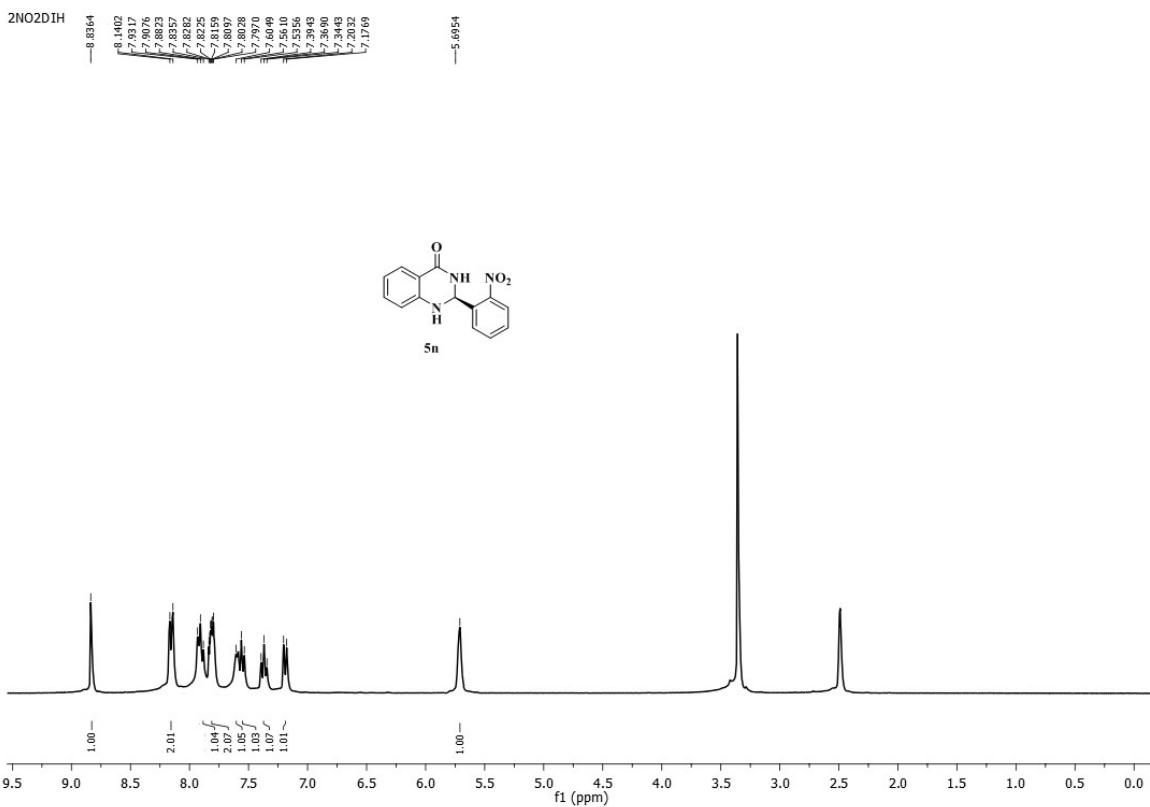
S 24: ^{13}C NMR Spectrum of (*S*)-2-(4-bromophenyl)-2,3-dihydroquinazolin-4(1H)-one (5l).



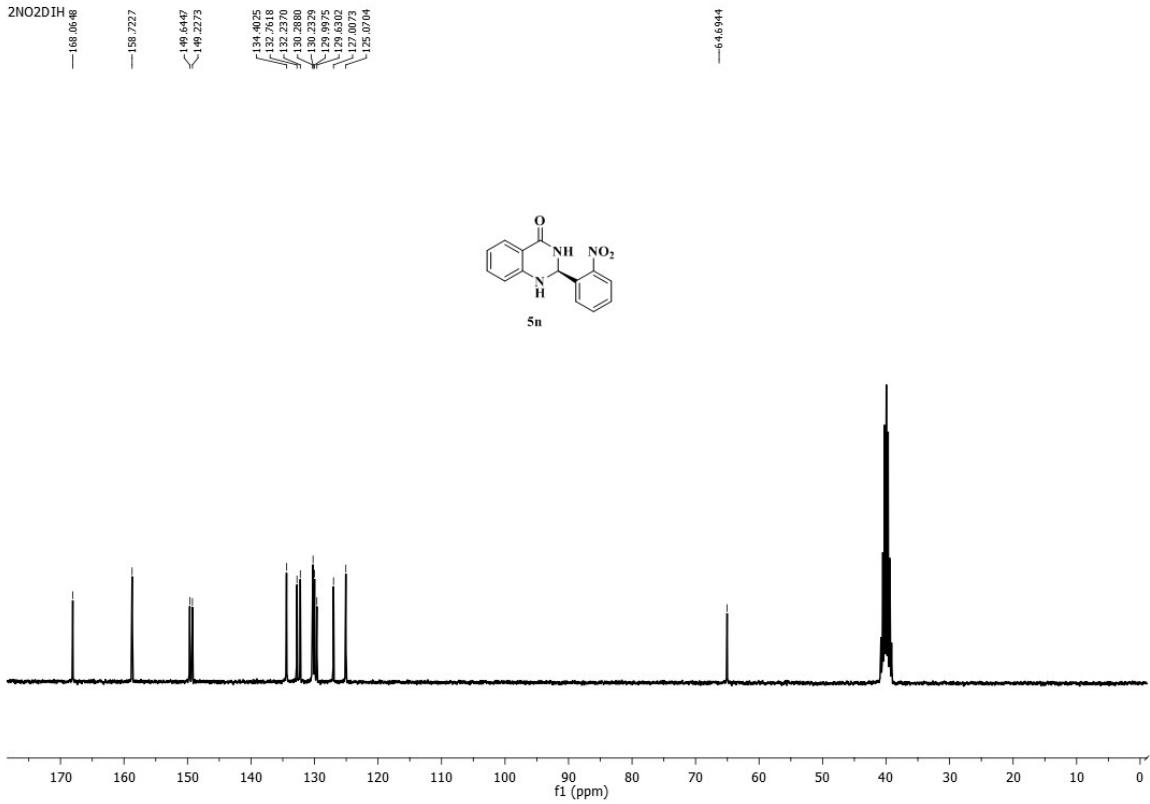
S 25: ¹H NMR Spectrum of (*S*)-2-(4-nitrophenyl)-2,3-dihydroquinazolin-4(1H)-one (5m).



S 26: ¹³C NMR Spectrum of (*S*)-2-(4-nitrophenyl)-2,3-dihydroquinazolin-4(1H)-one (5m).



S 27: ^1H NMR Spectrum of (S)-2-(2-nitrophenyl)-2,3-dihydroquinazolin-4(1H)-one (5n).

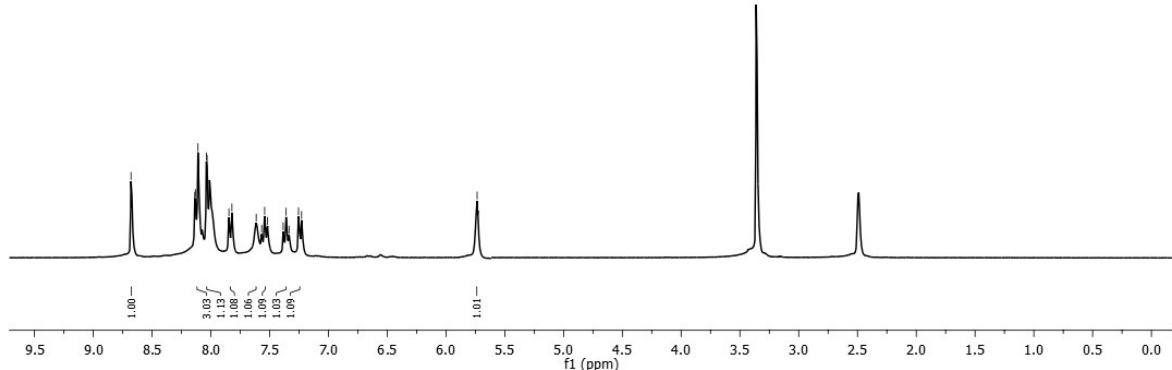
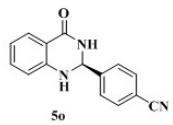


S 28: ^{13}C NMR Spectrum of (S)-2-(2-nitrophenyl)-2,3-dihydroquinazolin-4(1H)-one (5n).

4CNDIH

—8.6793
—8.1568
—8.1316
—8.1696
—8.1642
—8.0855
—8.0244
—7.8466
—7.8210
—7.6144
—7.5683
—7.5266
—7.5175
—7.3652
—7.3600
—7.3553
—7.2547
—7.2286

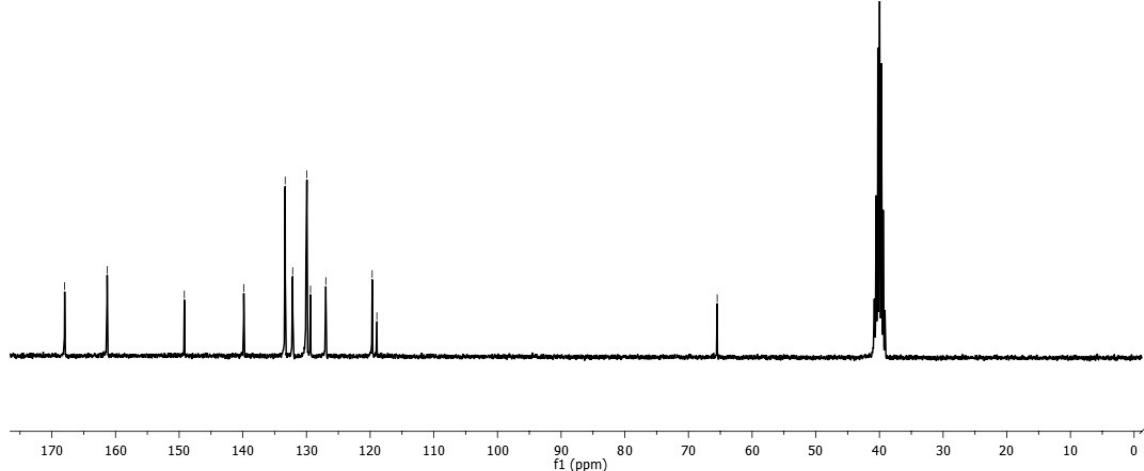
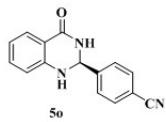
—5.7464

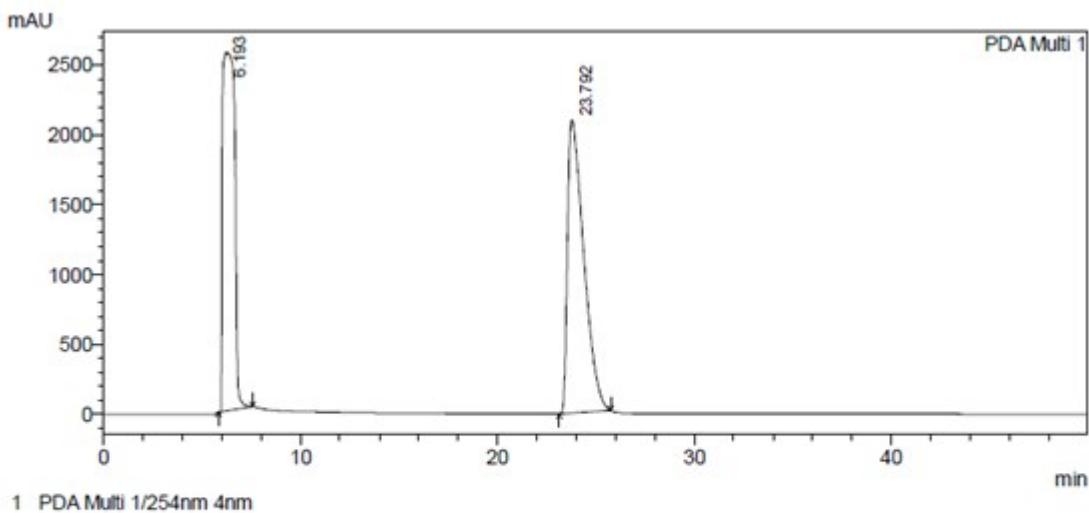
**S 29:** ^1H NMR Spectrum of (*S*)-4-(4-oxo-1,2,3,4-tetrahydroquinazolin-2-yl)benzonitrile (5o).

4CNDIH

—167.9838
—161.3042
—149.1839
—149.8461
—133.3485
—132.1728
—130.0854
—129.8396
—129.3672
—126.9639
—118.9474
—118.9475

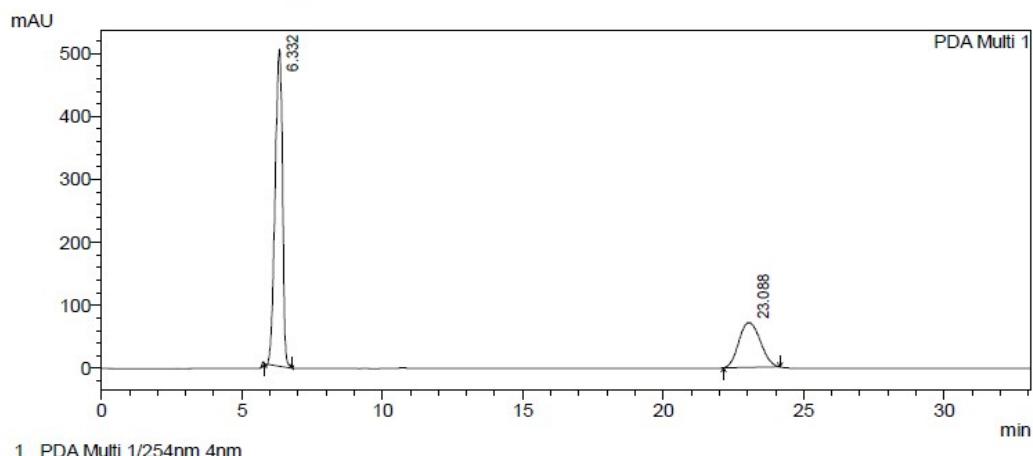
—65.2092

**S 30:** ^{13}C NMR Spectrum of (*S*)-4-(4-oxo-1,2,3,4-tetrahydroquinazolin-2-yl)benzonitrile (5o).



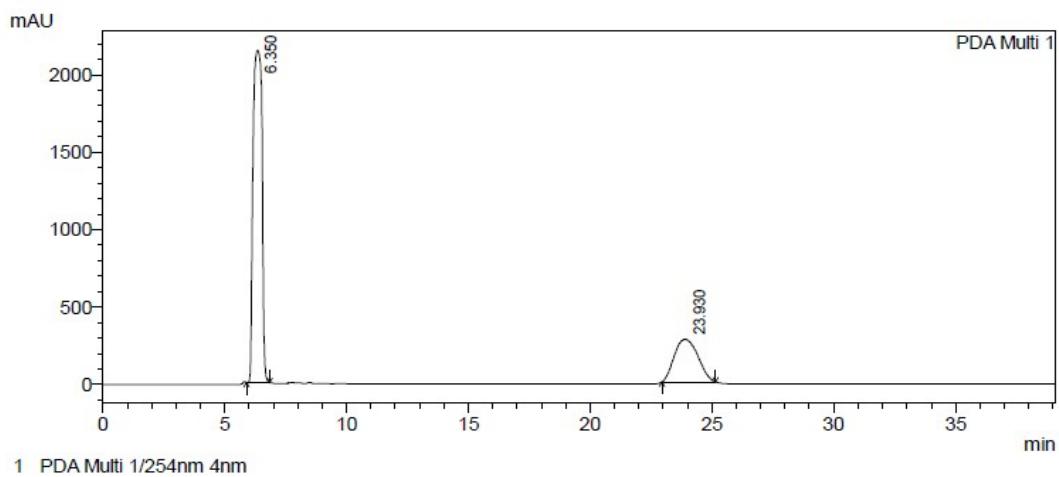
| PeakTable | | | | | |
|-----------|-----------|-----------|---------|---------|----------|
| Peak# | Ret. Time | Area | Height | Area % | Height % |
| 1 | 6.193 | 108356301 | 2567452 | 49.206 | 55.015 |
| 2 | 23.792 | 121180637 | 2099353 | 50.794 | 44.985 |
| Total | | 229536938 | 4666805 | 100.000 | 100.000 |

S 33: HPLC spectrum of Racemic Mixture (5a)

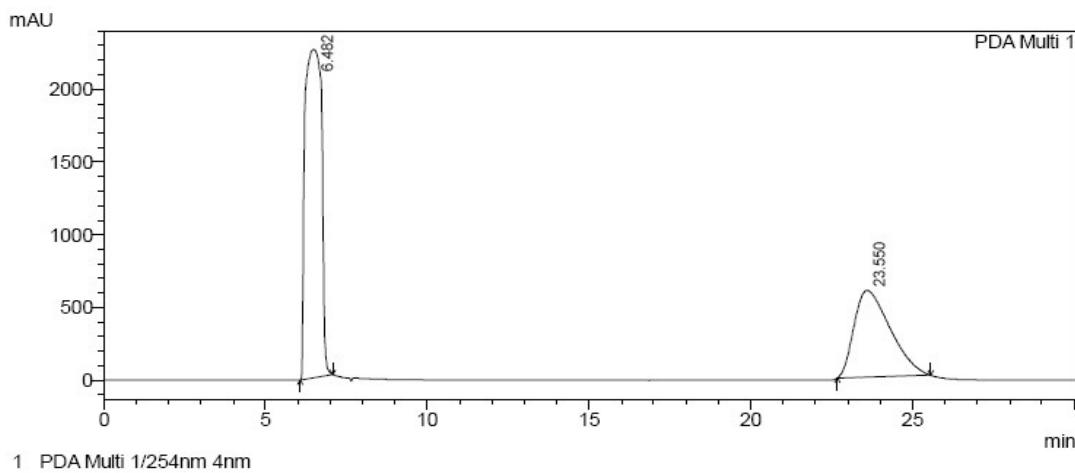


| PeakTable | | | | | |
|-----------|-----------|----------|--------|---------|----------|
| Peak# | Ret. Time | Area | Height | Area % | Height % |
| 1 | 6.332 | 9170207 | 502604 | 70.835 | 87.598 |
| 2 | 23.088 | 3775601 | 71160 | 29.165 | 12.402 |
| Total | | 12945808 | 573764 | 100.000 | 100.000 |

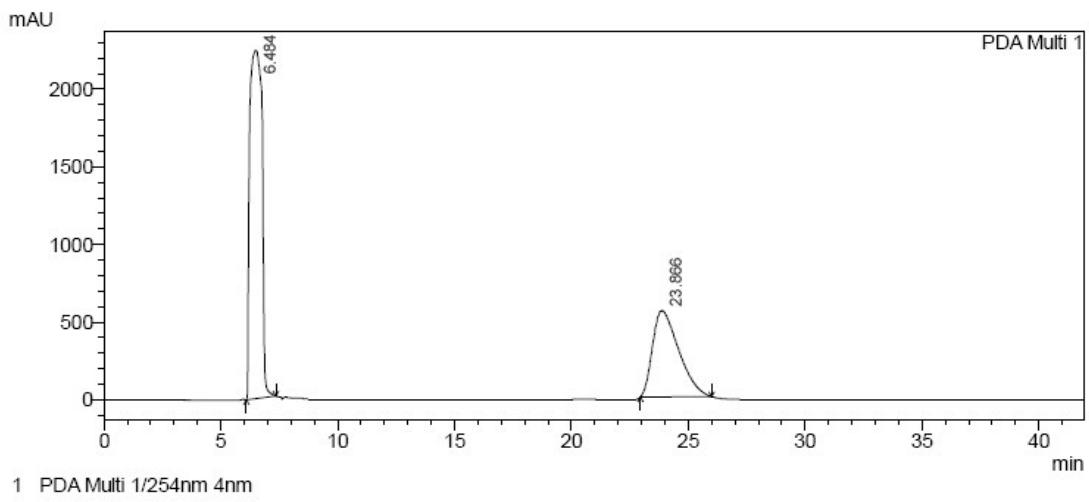
S 34: HPLC spectrum of Compound (5a) in presence of catalyst (1a) and Methanol in RT condition (Entry 1, Table 1).



S 35: HPLC spectrum of Compound (5a) in presence of catalyst (1b) and Methanol in RT condition (Entry 2, Table 1).



S 36: HPLC spectrum of Compound (5a) in presence of catalyst (1c) and Methanol in RT condition (Entry 3, Table 1).

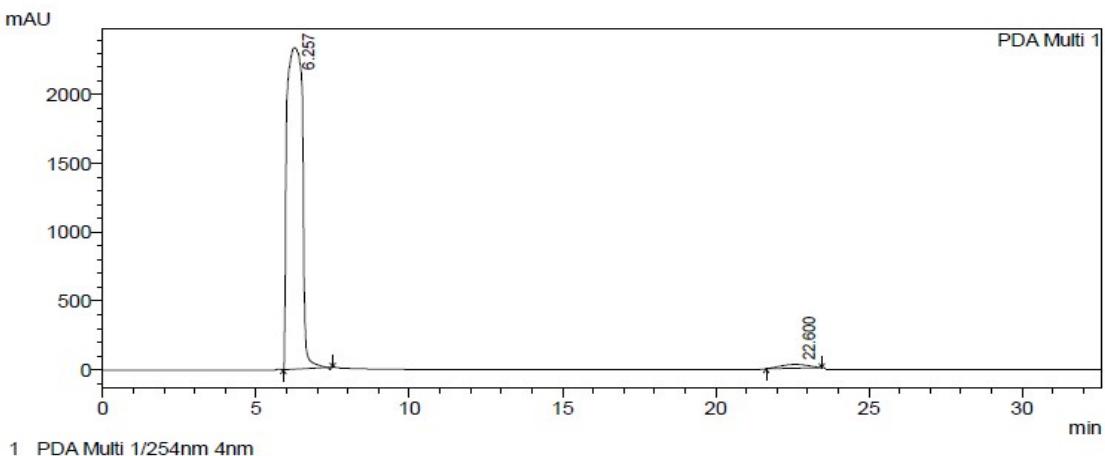


PeakTable

PDA Ch1 254nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|-----------|---------|---------|----------|
| 1 | 6.484 | 81332729 | 2240276 | 65.193 | 80.044 |
| 2 | 23.866 | 43424473 | 558526 | 34.807 | 19.956 |
| Total | | 124757202 | 2798802 | 100.000 | 100.000 |

S 37: HPLC spectrum of Compound (5a) in presence of catalyst (1d) and Methanol in RT condition (Entry 4, Table 1).

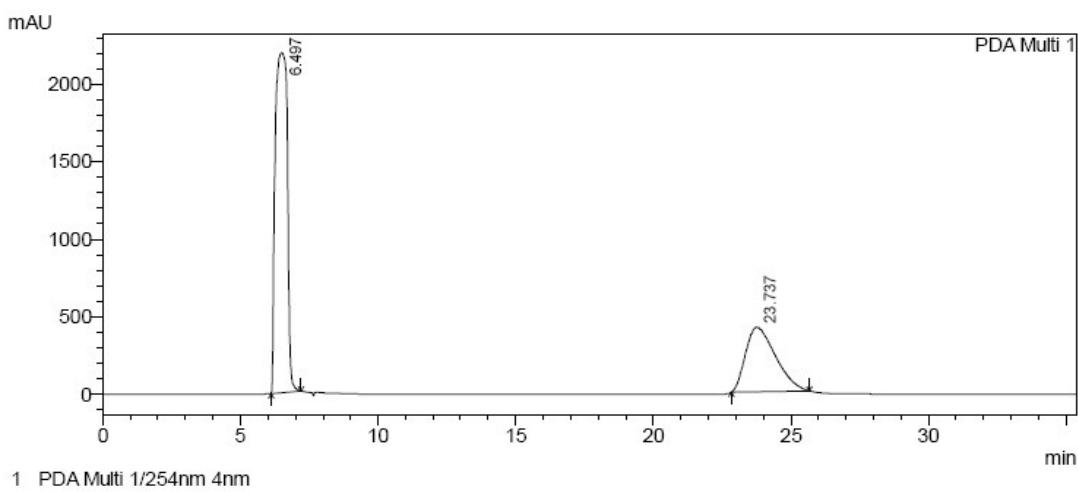


PeakTable

PDA Ch1 254nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|----------|---------|---------|----------|
| 1 | 6.257 | 80437817 | 2336764 | 97.848 | 98.805 |
| 2 | 22.600 | 1769313 | 28261 | 2.152 | 1.195 |
| Total | | 82207130 | 2365025 | 100.000 | 100.000 |

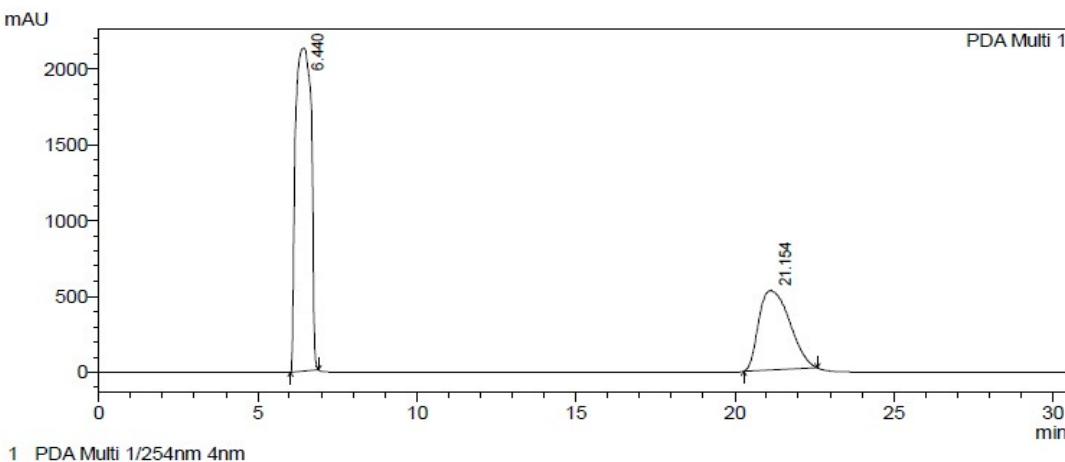
S 38: HPLC spectrum of Compound (5a) in presence of catalyst (2) and Methanol in RT condition (Entry 5, Table 1).



PDA Ch1 254nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|----------|---------|---------|----------|
| 1 | 6.497 | 67090226 | 2193852 | 68.115 | 84.041 |
| 2 | 23.737 | 31404660 | 416602 | 31.885 | 15.959 |
| Total | | 98494886 | 2610454 | 100.000 | 100.000 |

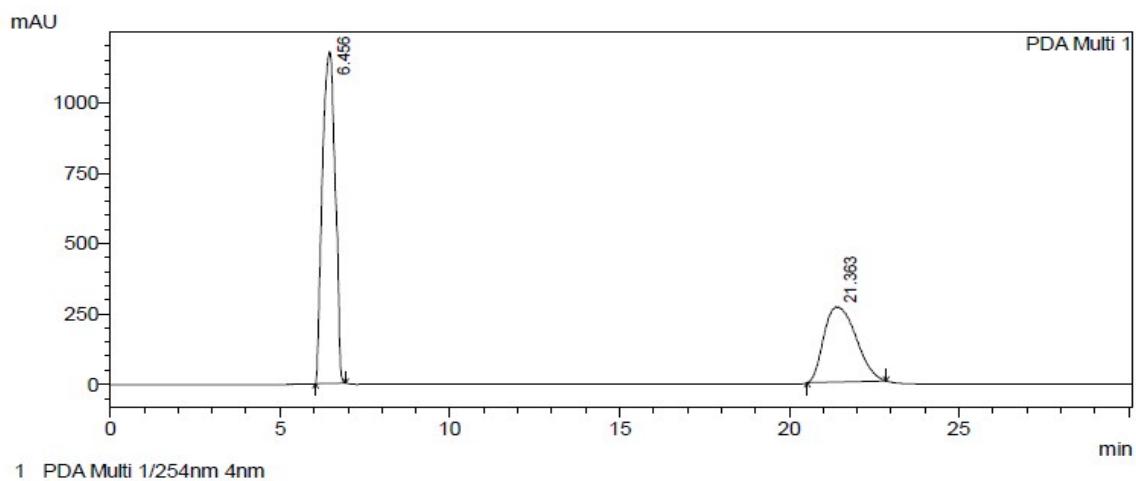
S 39: HPLC spectrum of Compound (5a) in presence of catalyst (2) and Chloroform in RT condition (Entry 1, Table 2).



PDA Ch1 254nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|-----------|---------|---------|----------|
| 1 | 6.440 | 71931661 | 2129986 | 66.938 | 80.222 |
| 2 | 21.154 | 35529201 | 525117 | 33.062 | 19.778 |
| Total | | 107460863 | 2655103 | 100.000 | 100.000 |

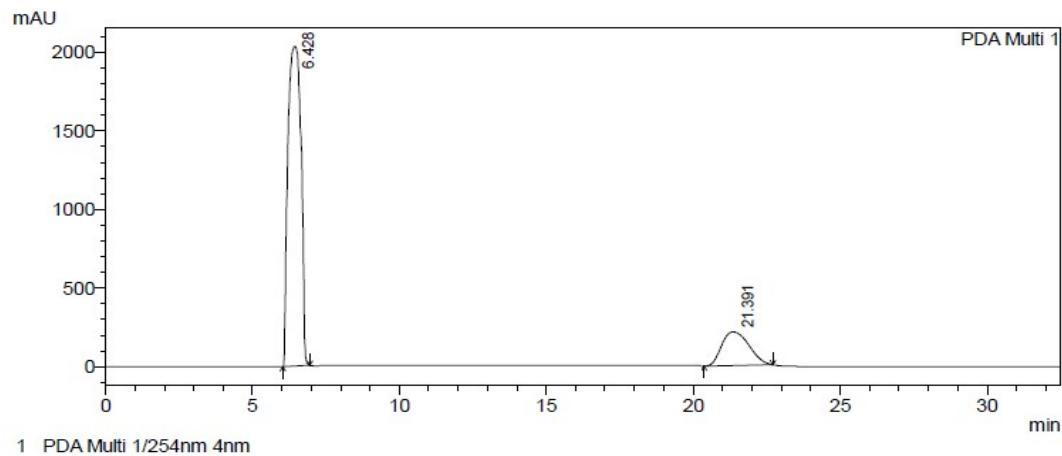
S 40: HPLC spectrum of Compound (5a) in presence of catalyst (2) and Acetone in RT condition (Entry 2, Table 2).



PDA Ch1 254nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|----------|---------|---------|----------|
| 1 | 6.456 | 30824209 | 1177051 | 63.585 | 81.493 |
| 2 | 21.363 | 17652729 | 267308 | 36.415 | 18.507 |
| Total | | 48476939 | 1444359 | 100.000 | 100.000 |

S 41: HPLC spectrum of Compound (5a) in presence of catalyst (2) and DCM in RT condition (Entry 3, Table 2).

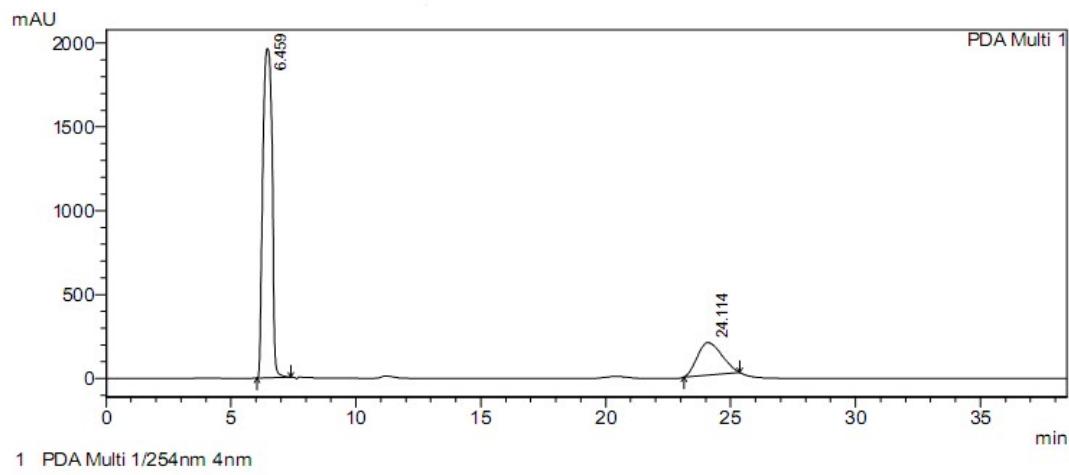


PDA Ch1 254nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|----------|---------|---------|----------|
| 1 | 6.428 | 62879476 | 2033093 | 81.778 | 90.363 |
| 2 | 21.391 | 14010710 | 216824 | 18.222 | 9.637 |
| Total | | 76890186 | 2249917 | 100.000 | 100.000 |

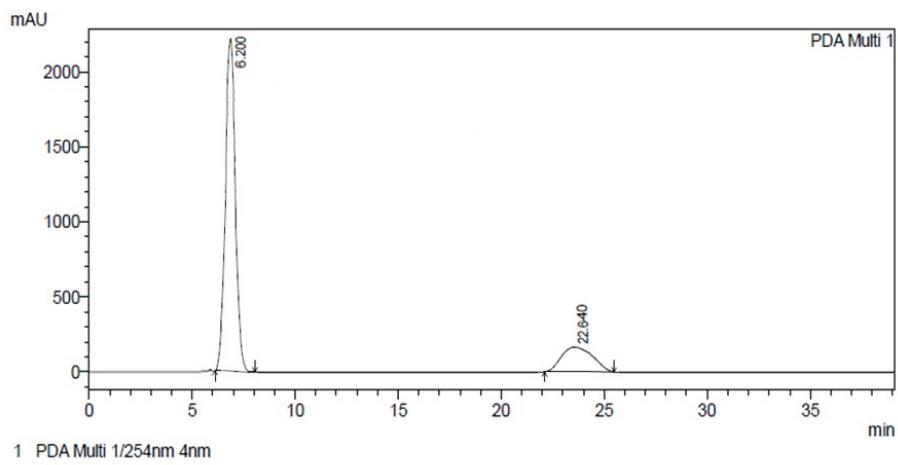
S 42: HPLC spectrum of Compound (5a) in presence of catalyst (2) and Ethanol in RT condition (Entry 4, Table 2).

S 43: HPLC spectrum of Compound (5a) in presence of catalyst (2) and Methanol in RT condition (Entry 5, Table 2).



| PDA Ch1 254nm 4nm | | | | | | |
|-------------------|-----------|----------|---------|---------|----------|--|
| Peak# | Ret. Time | Area | Height | Area % | Height % | |
| 1 | 6.459 | 50223635 | 1965629 | 79.318 | 90.986 | |
| 2 | 24.114 | 13095802 | 194738 | 20.682 | 9.014 | |
| Total | | 63319437 | 2160366 | 100.000 | 100.000 | |

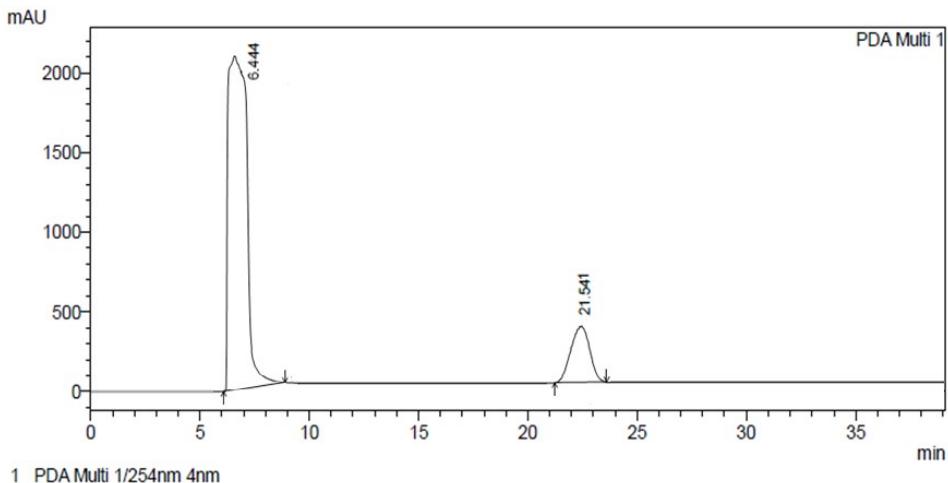
S 44: HPLC spectrum of Compound (5a) in presence of catalyst (2) and Methanol in Cold condition (0°C) (Entry 6, Table 2).



PDA Ch1 240nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|----------|---------|---------|----------|
| 1 | 6.200 | 14690360 | 1273254 | 91.885 | 96.459 |
| 2 | 22.640 | 1297389 | 19932 | 8.115 | 3.541 |
| Total | | 15987749 | 1293185 | 100.000 | 100.000 |

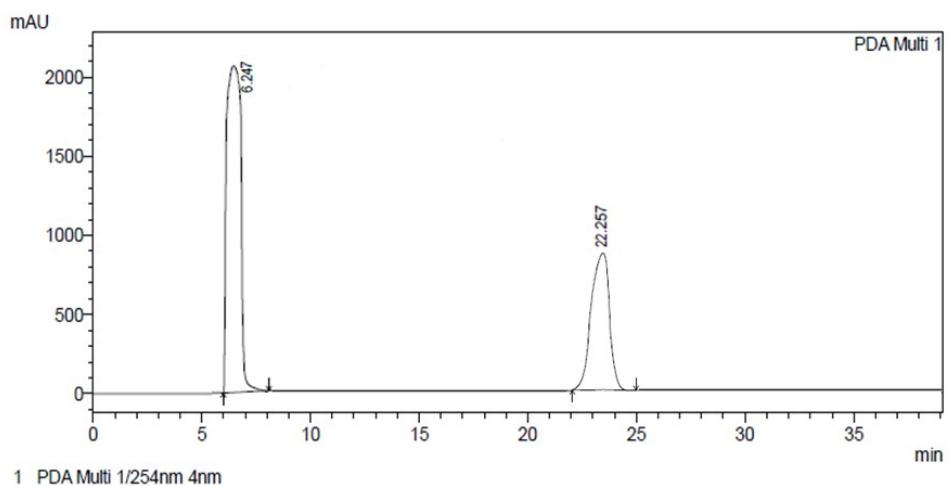
S 45: HPLC spectrum of Compound (5a) in presence of catalyst (2) and DMF in RT condition (Entry 7, Table 2).



PDA Ch1 254nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|----------|--------|---------|----------|
| 1 | 6.444 | 16288090 | 453694 | 93.044 | 88.009 |
| 2 | 21.541 | 1217689 | 61815 | 6.956 | 11.991 |
| Total | | 17505779 | 515509 | 100.000 | 100.000 |

S 46: HPLC spectrum of Compound (5a) in presence of catalyst (2) and DMSO in RT condition (Entry 8, Table 2).

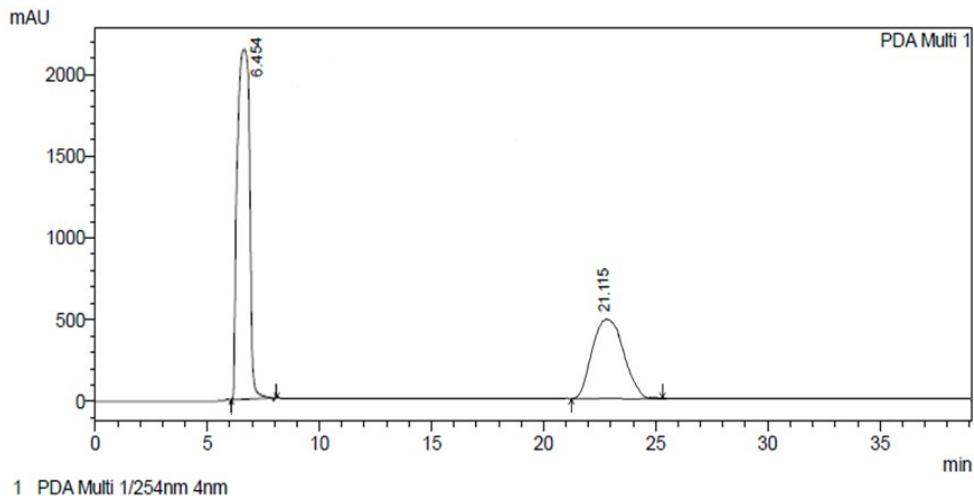


PeakTable

PDA Ch1 254nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|----------|--------|---------|----------|
| 1 | 6.247 | 8473498 | 108659 | 83.721 | 74.717 |
| 2 | 22.257 | 1647576 | 36770 | 16.279 | 25.283 |
| Total | | 10121074 | 145429 | 100.000 | 100.000 |

S 47: HPLC spectrum of Compound (5a) in presence of catalyst (2) and toluene in RT condition (Entry 9, Table 2).



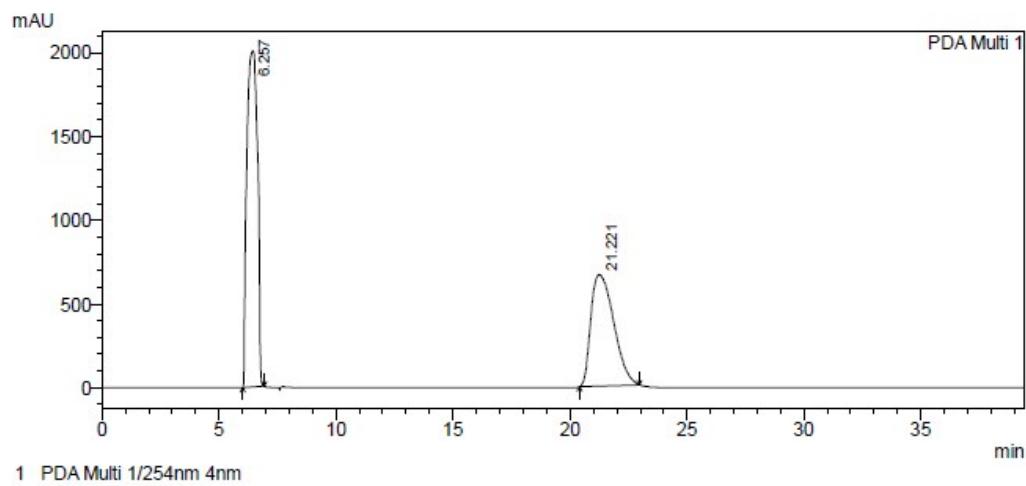
PeakTable

PDA Ch1 254nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|----------|--------|---------|----------|
| 1 | 6.454 | 59077266 | 753103 | 79.998 | 86.633 |
| 2 | 21.115 | 14771069 | 116201 | 20.002 | 13.367 |
| Total | | 73848335 | 869304 | 100.000 | 100.000 |

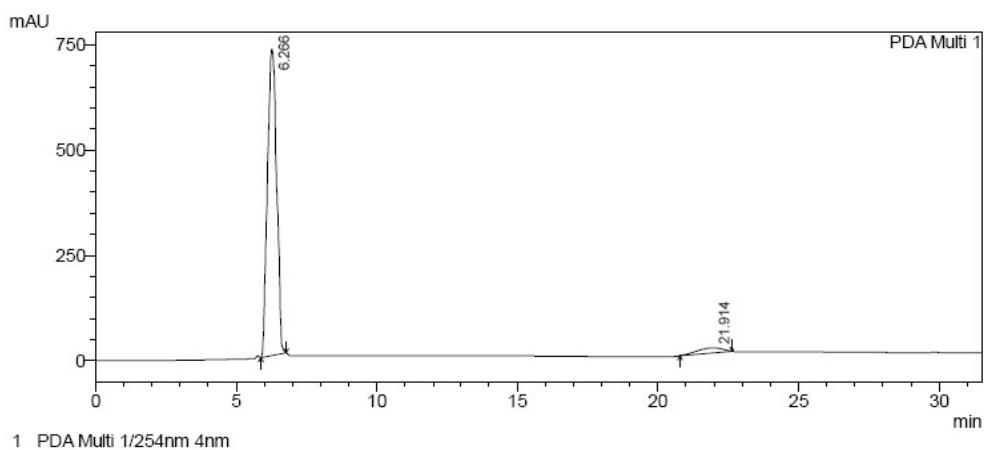
S 48: HPLC spectrum of Compound (5a) in presence of catalyst (2) and cyclohexane in RT condition (Entry 10, Table 2).

S 49: HPLC spectrum of Compound (5a) in presence of catalyst (2) and Methanol in RT condition (Entry 1, Table 3).



| PeakTable | | | | | | |
|-------------------|-----------|-----------|---------|---------|----------|--|
| PDA Ch1 254nm 4nm | | | | | | |
| Peak# | Ret. Time | Area | Height | Area % | Height % | |
| 1 | 6.257 | 63325278 | 2007387 | 50.272 | 75.038 | |
| 2 | 21.221 | 45346012 | 667764 | 49.728 | 24.962 | |
| Total | | 108671290 | 2675151 | 100.000 | 100.000 | |

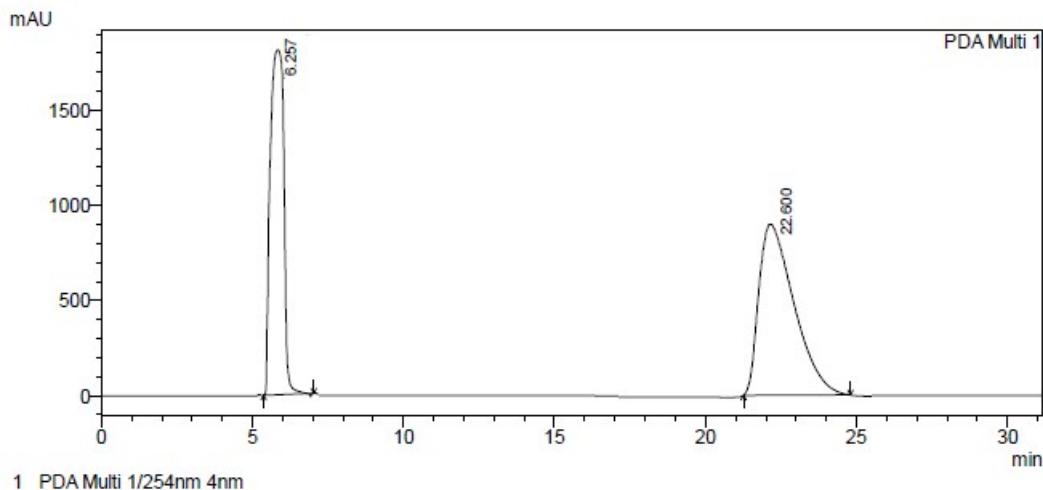
S 50: HPLC spectrum of Racemic Mixture 5b.



PeakTable

| PDA Ch1 254nm 4nm | | | | | |
|-------------------|-----------|----------|--------|---------|----------|
| Peak# | Ret. Time | Area | Height | Area % | Height % |
| 1 | 6.266 | 16549337 | 727187 | 95.540 | 98.337 |
| 2 | 21.914 | 772539 | 12294 | 4.460 | 1.663 |
| Total | | 17321876 | 739481 | 100.000 | 100.000 |

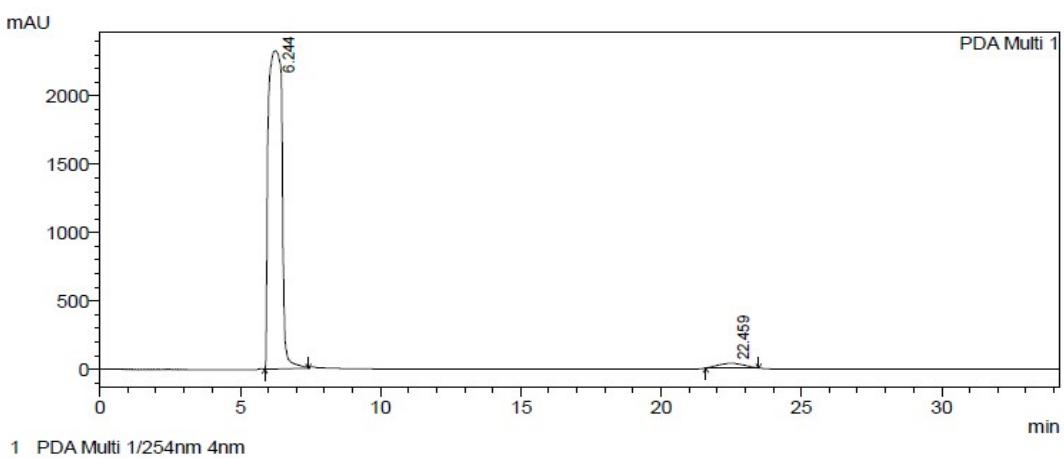
S 51: HPLC spectrum of Compound (5b) in presence of catalyst (2) and Methanol in RT condition (Entry 2, Table 3).



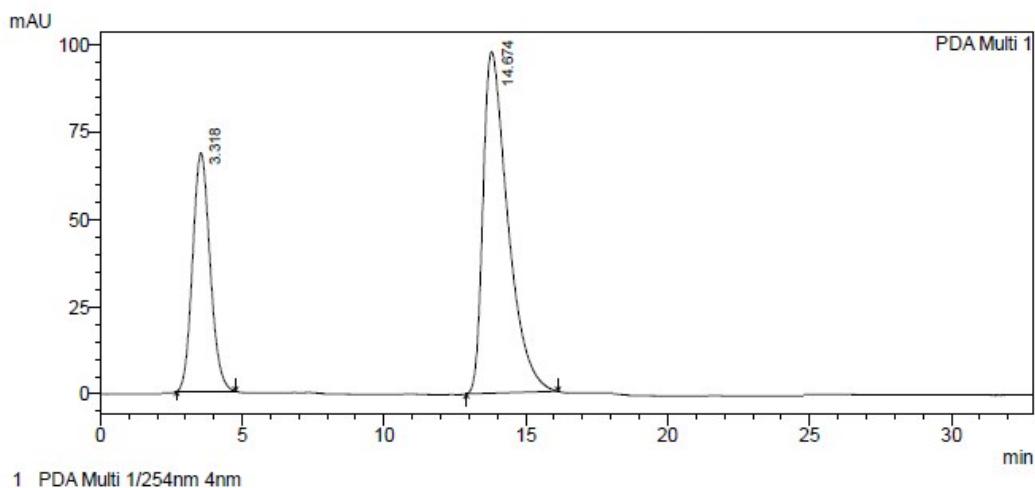
PeakTable

| PDA Ch1 254nm 4nm | | | | | |
|-------------------|-----------|-----------|---------|---------|----------|
| Peak# | Ret. Time | Area | Height | Area % | Height % |
| 1 | 6.257 | 56498188 | 1810690 | 49.654 | 66.834 |
| 2 | 22.600 | 72923638 | 898537 | 50.346 | 33.166 |
| Total | | 129421826 | 2709227 | 100.000 | 100.000 |

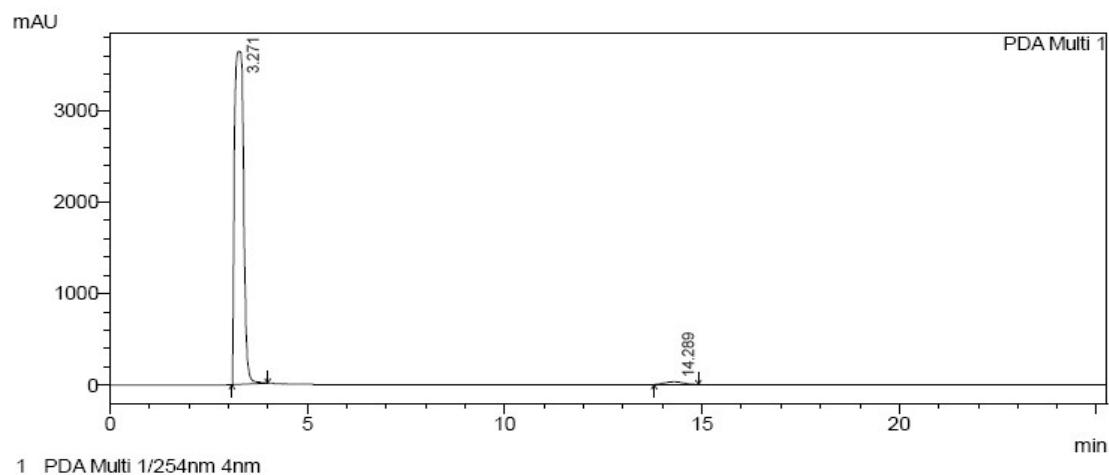
S 52: HPLC spectrum of Racemic Mixture 5c.



S 53: HPLC spectrum of Compound (5c) in presence of catalyst (2) and Methanol in RT condition (Entry 3, Table 3).



S 54: HPLC spectrum of Racemic Mixture 5d.

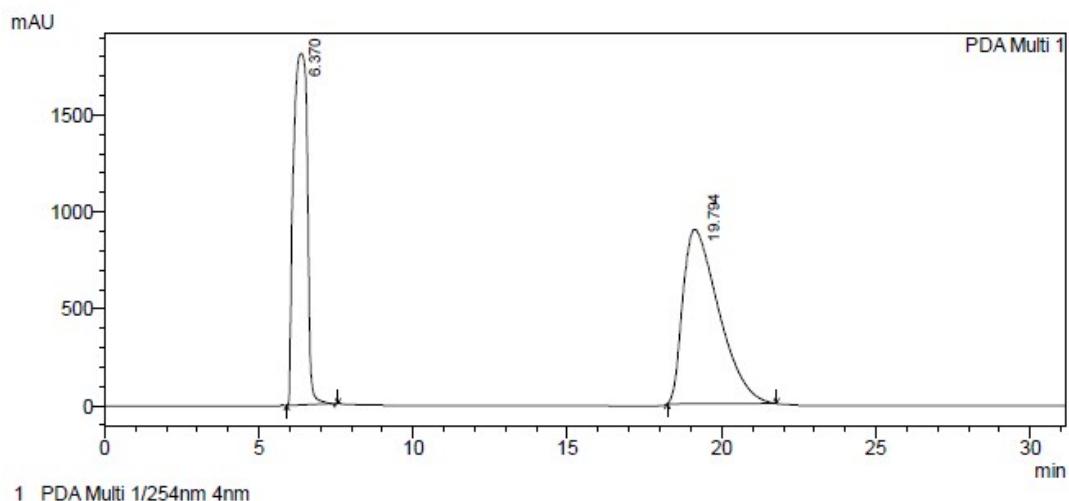


PDA Ch1 254nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|----------|---------|---------|----------|
| 1 | 3.271 | 58152507 | 3645163 | 98.299 | 99.178 |
| 2 | 14.289 | 1006096 | 30221 | 1.701 | 0.822 |
| Total | | 59158603 | 3675384 | 100.000 | 100.000 |

PeakTable

S 55: HPLC spectrum of Compound (5d) in presence of catalyst (2) and Methanol in RT condition (Entry 4, Table 3).

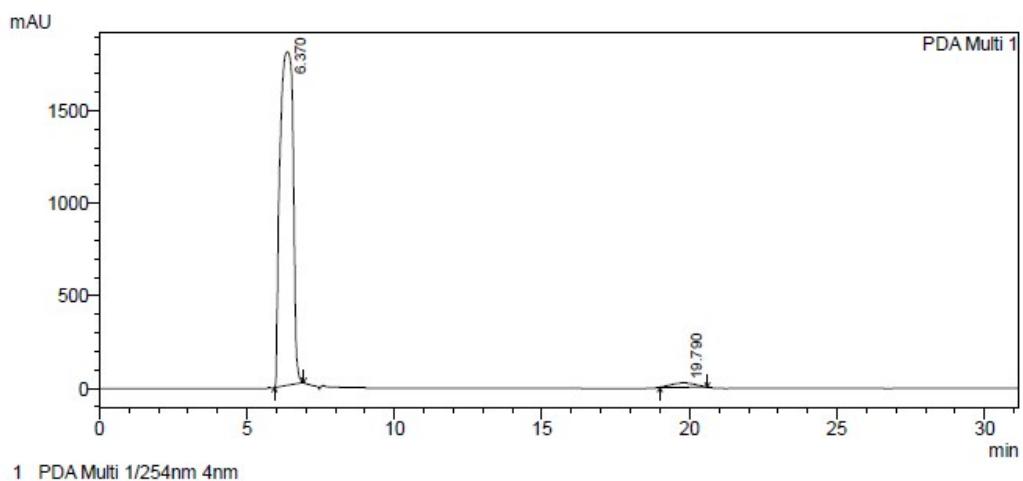


PDA Ch1 254nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|-----------|---------|---------|----------|
| 1 | 6.370 | 56498188 | 1810690 | 49.354 | 67.834 |
| 2 | 19.794 | 72923638 | 898537 | 50.646 | 32.166 |
| Total | | 129421826 | 2709227 | 100.000 | 100.000 |

PeakTable

S 56: HPLC spectrum of Racemic Mixture 5e.

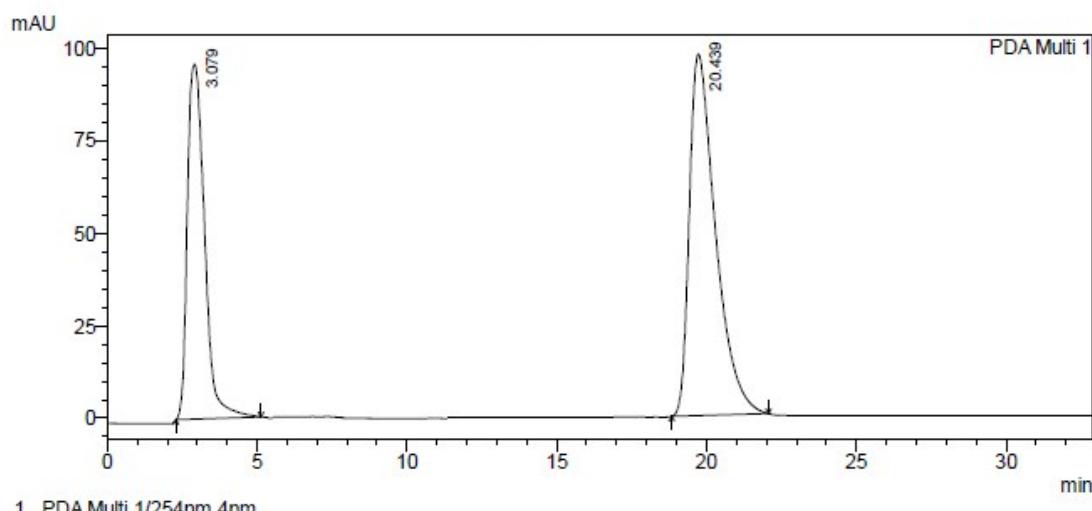


PeakTable

PDA.Ch1 254nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|----------|---------|---------|----------|
| 1 | 6.370 | 55537988 | 1799049 | 97.676 | 98.727 |
| 2 | 19.790 | 1321605 | 23206 | 2.324 | 1.273 |
| Total | | 56859593 | 1822256 | 100.000 | 100.000 |

S 57: HPLC spectrum of Compound (5e) in presence of catalyst (2) and Methanol in RT condition (Entry 5, Table 3).

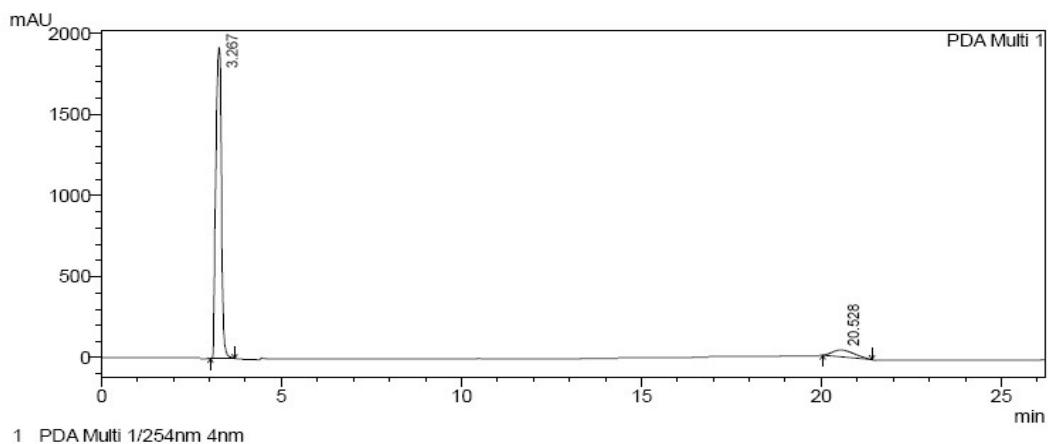


PeakTable

PDA.Ch1 254nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|---------|--------|---------|----------|
| 1 | 3.079 | 2921504 | 68179 | 49.200 | 49.114 |
| 2 | 20.439 | 5945230 | 97650 | 50.800 | 50.886 |
| Total | | 8866734 | 165829 | 100.000 | 100.000 |

S 58: HPLC spectrum of Racemic Mixture 5f.

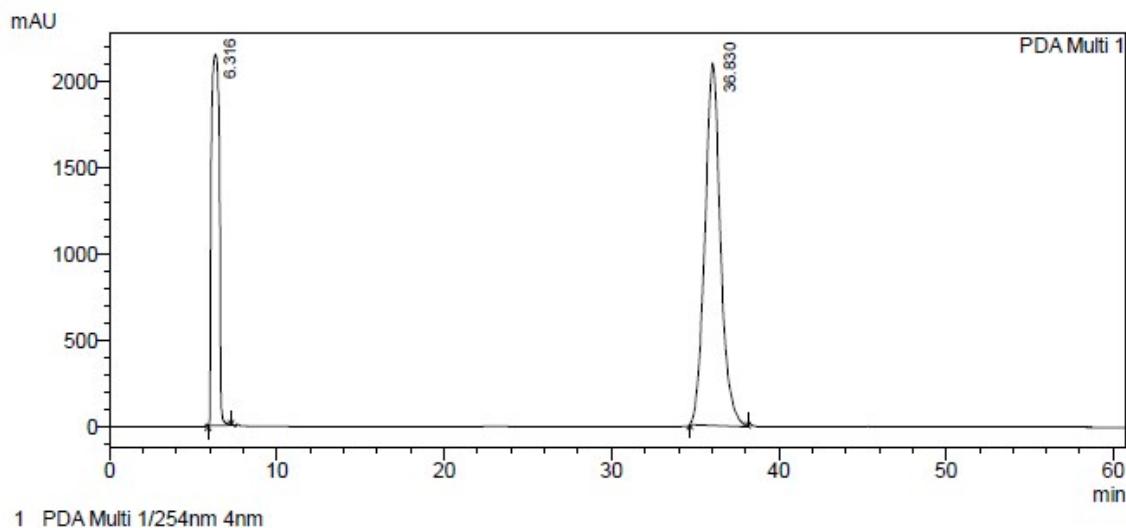


PeakTable

PDA Ch1 254nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|----------|---------|---------|----------|
| 1 | 3.267 | 20654317 | 1918320 | 92.007 | 97.886 |
| 2 | 20.528 | 1794330 | 41429 | 7.993 | 2.114 |
| Total | | 22448647 | 1959750 | 100.000 | 100.000 |

S 59: HPLC spectrum of Compound (5f) in presence of catalyst (2) and Methanol in RT condition (Entry 6, Table 3).

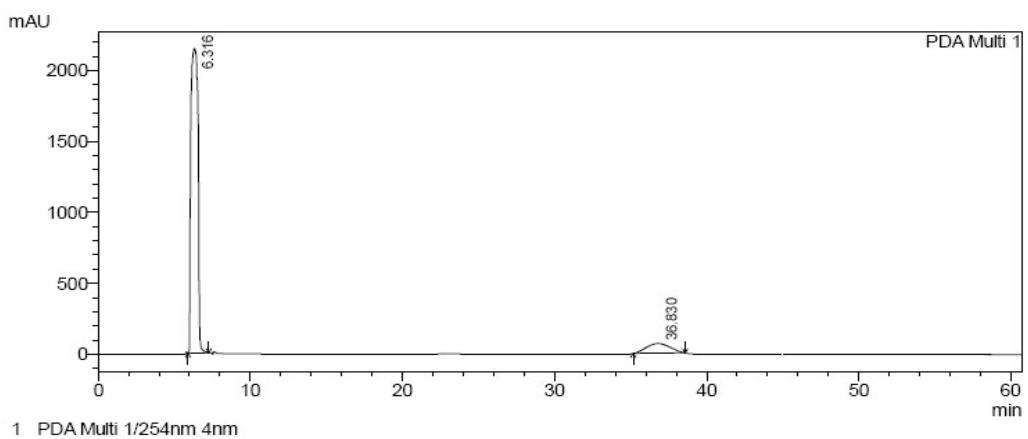


PeakTable

PDA Ch1 254nm 4nm

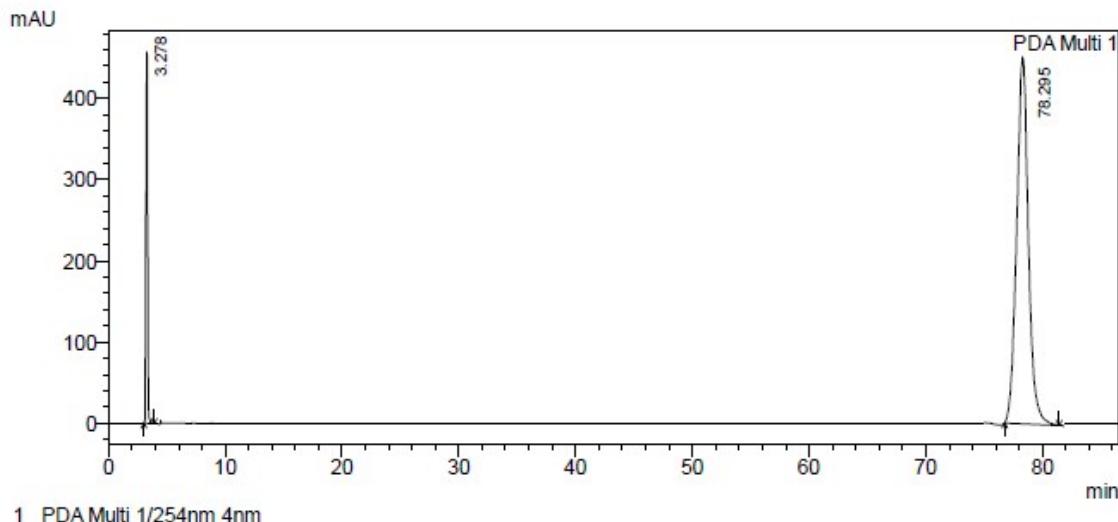
| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|----------|---------|---------|----------|
| 1 | 6.316 | 68898176 | 2146473 | 49.160 | 50.432 |
| 2 | 36.830 | 7519850 | 69181 | 50.840 | 49.568 |
| Total | | 76418027 | 2215654 | 100.000 | 100.000 |

S 60: HPLC spectrum of Racemic Mixture 5g.



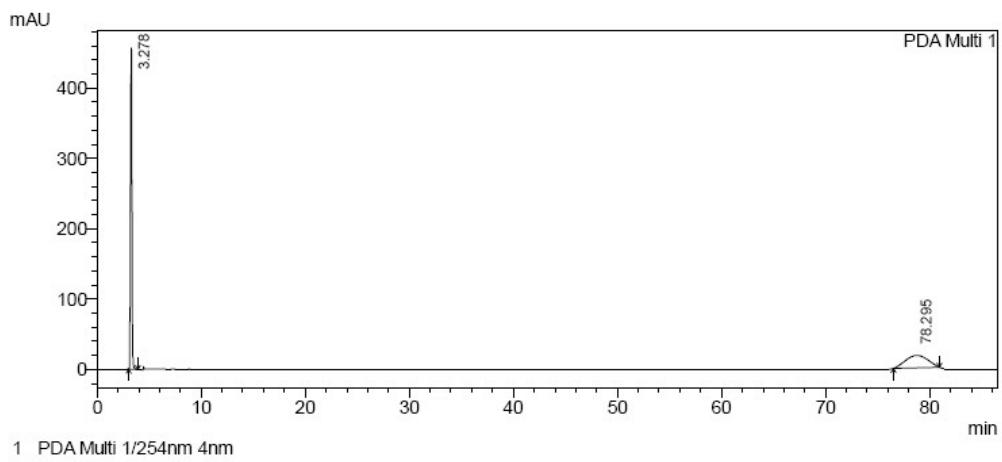
| PeakTable | | | | | |
|-----------|-----------|----------|---------|---------|----------|
| Peak# | Ret. Time | Area | Height | Area % | Height % |
| 1 | 6.316 | 68898176 | 2146473 | 90.160 | 96.878 |
| 2 | 36.830 | 7519850 | 69181 | 9.840 | 3.122 |
| Total | | 76418027 | 2215654 | 100.000 | 100.000 |

S 61: HPLC spectrum of Compound (5g) in presence of catalyst(2) and Methanol in RT condition (Entry 7, Table 3).



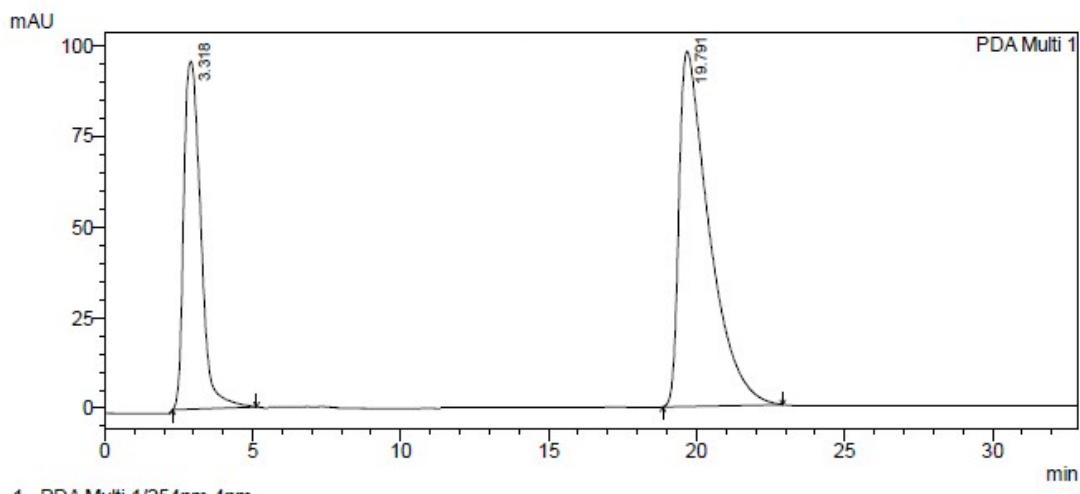
| PeakTable | | | | | |
|-----------|-----------|---------|--------|---------|----------|
| Peak# | Ret. Time | Area | Height | Area % | Height % |
| 1 | 3.278 | 1038116 | 77030 | 49.200 | 49.432 |
| 2 | 78.295 | 1421893 | 132360 | 50.800 | 50.568 |
| Total | | 2460009 | 209390 | 100.000 | 100.000 |

S 62: HPLC spectrum of Racemic Mixture 5h.



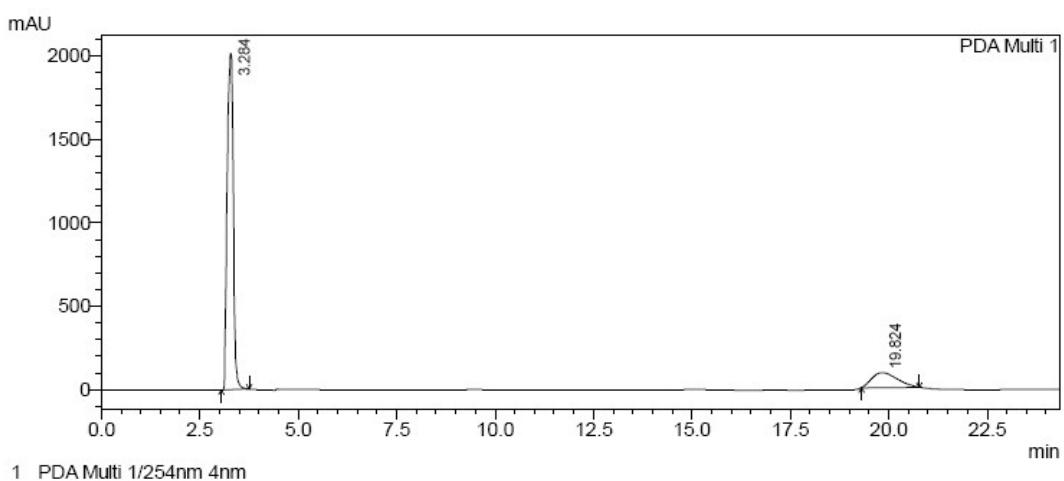
| PDA Ch1 254nm 4nm | | | | | |
|-------------------|-----------|---------|--------|---------|----------|
| Peak# | Ret. Time | Area | Height | Area % | Height % |
| 1 | 3.278 | 4572835 | 456401 | 97.406 | 99.782 |
| 2 | 78.295 | 121769 | 999 | 2.594 | 0.218 |
| Total | | 4694604 | 457400 | 100.000 | 100.000 |

S 63: HPLC spectrum of Compound (5h) in presence of catalyst (2) and Methanol in RT condition (Entry 8, Table 3).



| PDA Ch1 254nm 4nm | | | | | |
|-------------------|-----------|---------|--------|---------|----------|
| Peak# | Ret. Time | Area | Height | Area % | Height % |
| 1 | 3.318 | 2921504 | 68179 | 49.800 | 49.114 |
| 2 | 19.791 | 5945230 | 97650 | 50.200 | 50.886 |
| Total | | 8866734 | 165829 | 100.000 | 100.000 |

S 64: HPLC spectrum of Racemic Mixture 5i.

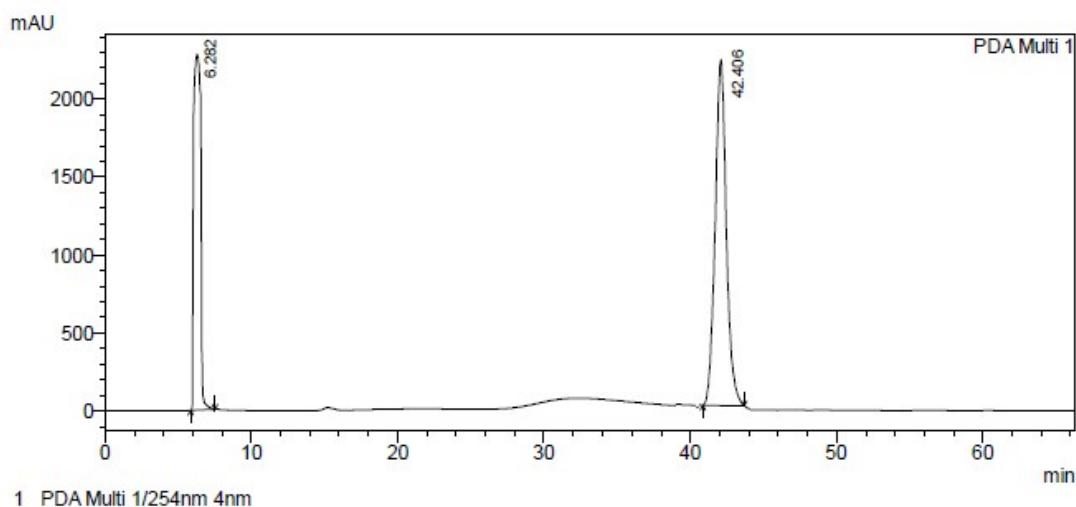


PeakTable

PDA Ch1 254nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|----------|---------|---------|----------|
| 1 | 3.284 | 21650659 | 2010549 | 84.480 | 95.751 |
| 2 | 19.824 | 3977410 | 89212 | 15.520 | 4.249 |
| Total | | 25628069 | 2099761 | 100.000 | 100.000 |

S 65: HPLC spectrum of Compound (5i) in presence of catalyst (2) and Methanol in RT condition (Entry 9, Table 3).

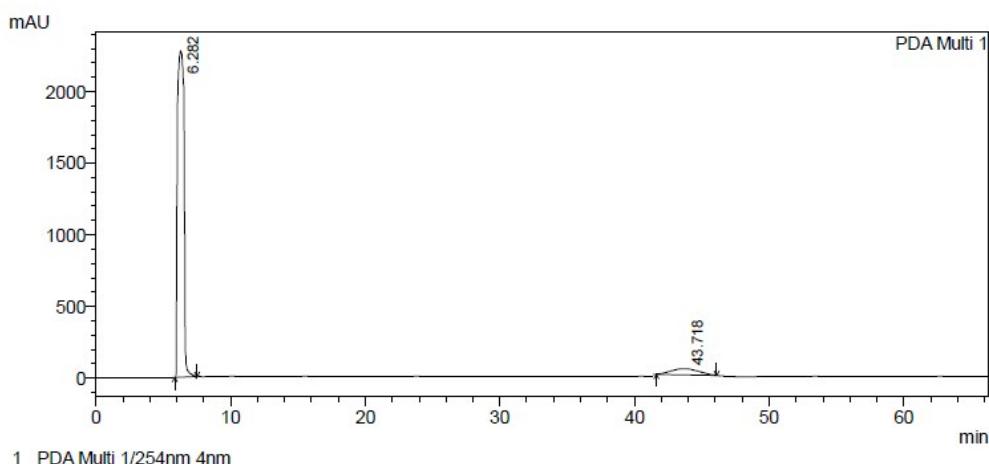


PeakTable

PDA Ch1 254nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|---------|--------|---------|----------|
| 1 | 6.282 | 884709 | 62400 | 49.017 | 49.432 |
| 2 | 42.406 | 996988 | 94308 | 50.983 | 50.568 |
| Total | | 1881697 | 156708 | 100.000 | 100.000 |

S 66: HPLC spectrum of Racemic Mixture 5j.

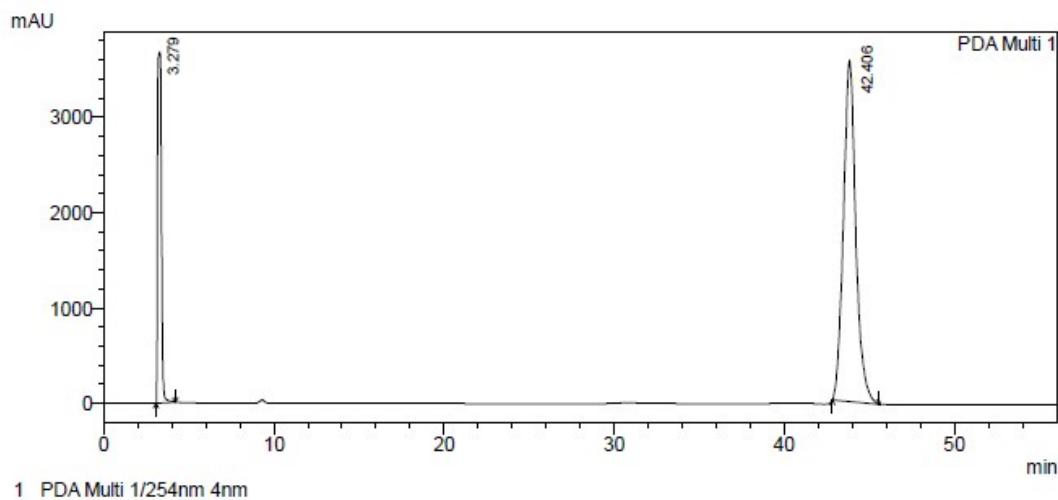


PeakTable

PDA Ch1 254nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|----------|---------|---------|----------|
| 1 | 6.282 | 76421011 | 2277298 | 92.640 | 98.177 |
| 2 | 43.718 | 6071399 | 42288 | 7.360 | 1.823 |
| Total | | 82492410 | 2319586 | 100.000 | 100.000 |

S 67: HPLC spectrum of Compound (5j) in presence of catalyst (2) and Methanol in RT condition (Entry 10, Table 3).

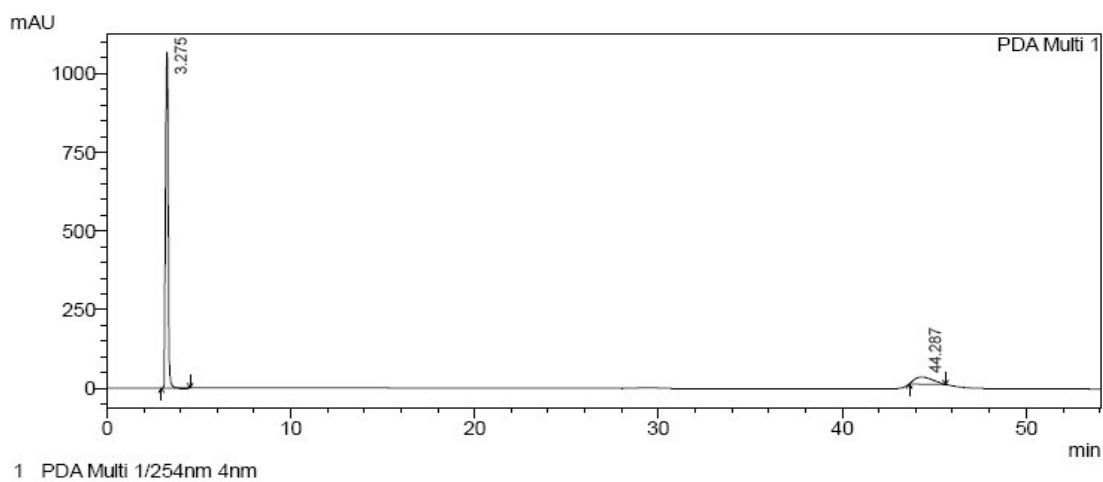


PeakTable

PDA Ch1 254nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|---------|--------|---------|----------|
| 1 | 3.279 | 1038116 | 77030 | 49.432 | 50.200 |
| 2 | 42.406 | 1421893 | 132360 | 50.568 | 49.800 |
| Total | | 2460009 | 209390 | 100.000 | 100.000 |

S 68: HPLC spectrum of Racemic Mixture 5k.

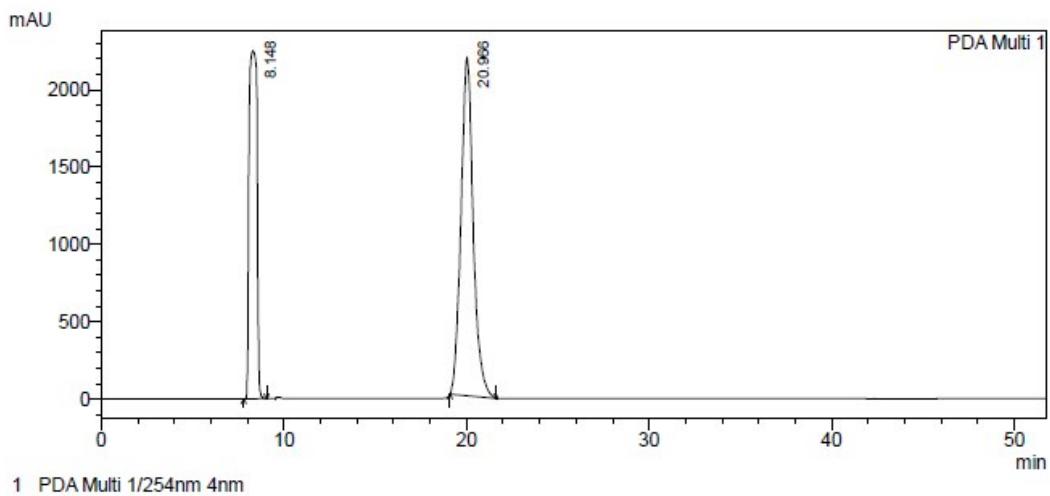


PDA Ch1 254nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|----------|---------|---------|----------|
| 1 | 3.275 | 10615892 | 1067485 | 87.388 | 97.957 |
| 2 | 44.287 | 1532149 | 22262 | 12.612 | 2.043 |
| Total | | 12148041 | 1089747 | 100.000 | 100.000 |

PeakTable

S 69: HPLC spectrum of Compound (5k) in presence of catalyst (2) and Methanol in RT condition (Entry 11, Table 3).

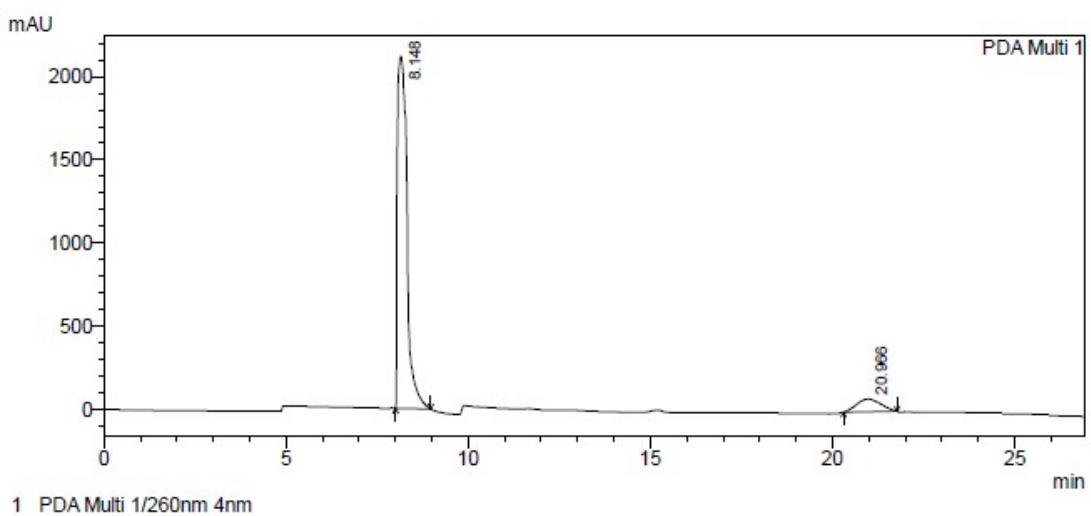


PDA Ch1 254nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|---------|--------|---------|----------|
| 1 | 8.148 | 1038116 | 77030 | 49.200 | 52.200 |
| 2 | 20.966 | 1421893 | 132360 | 50.800 | 47.800 |
| Total | | 2460009 | 209390 | 100.000 | 100.000 |

PeakTable

S 70: HPLC spectrum of Racemic Mixture 5l.

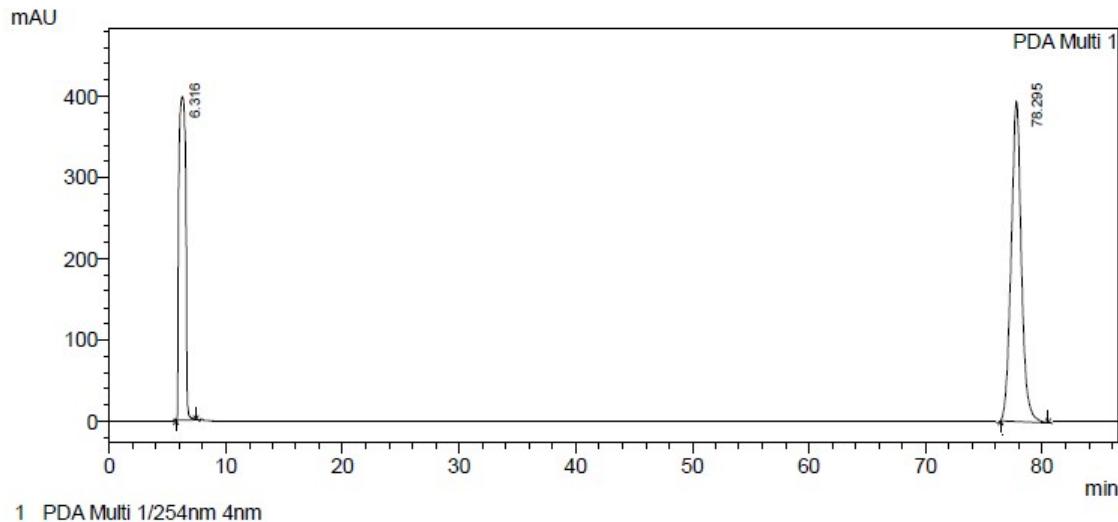


PeakTable

PDA Ch1 260nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|----------|---------|---------|----------|
| 1 | 8.148 | 38297910 | 2114859 | 91.489 | 96.497 |
| 2 | 20.966 | 3562699 | 76777 | 8.511 | 3.503 |
| Total | | 41860609 | 2191635 | 100.000 | 100.000 |

S 71: HPLC spectrum of Compound (5l) in presence of catalyst (2) and Methanol in RT condition (Entry 12, Table 3).

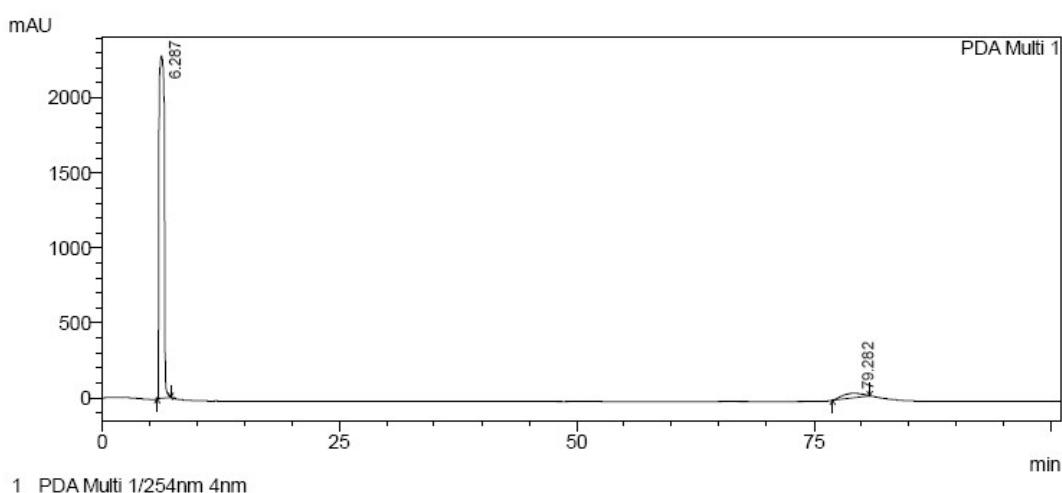


PeakTable

PDA Ch1 254nm 4nm

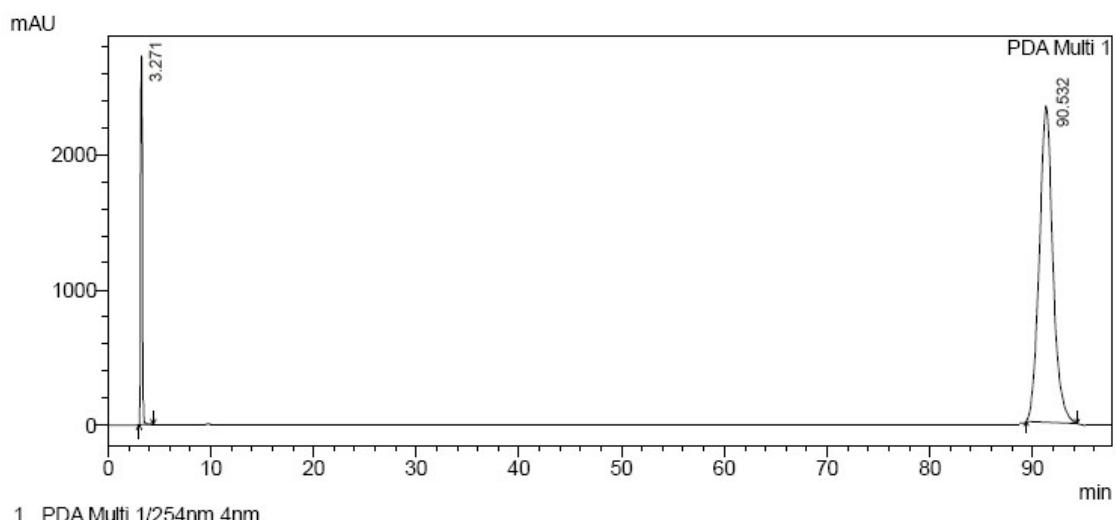
| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|---------|--------|---------|----------|
| 1 | 6.316 | 3004331 | 228818 | 49.625 | 49.432 |
| 2 | 78.295 | 3049796 | 274830 | 50.375 | 50.568 |
| Total | | 6054127 | 503648 | 100.000 | 100.000 |

S 72: HPLC spectrum of Racemic Mixture 5m.



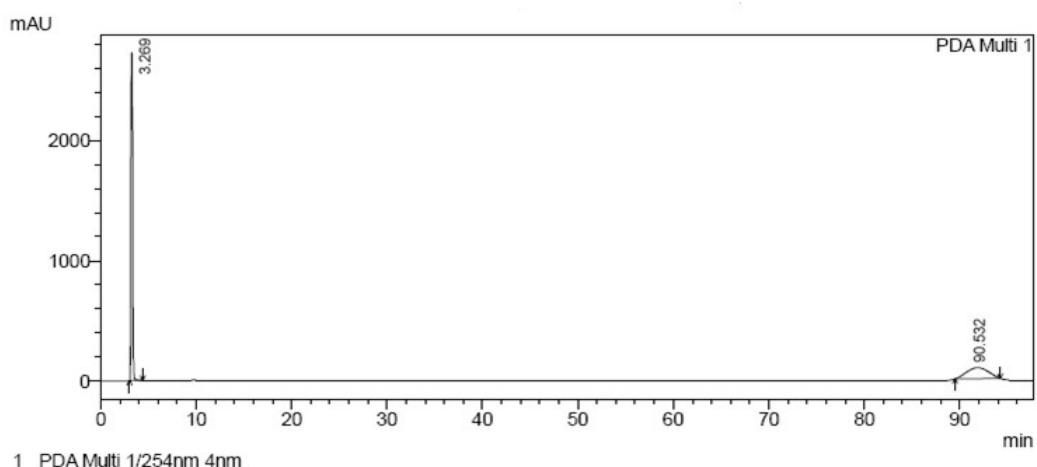
| PeakTable | | | | | |
|-------------------|-----------|----------|---------|---------|----------|
| PDA Ch1 254nm 4nm | | | | | |
| Peak# | Ret. Time | Area | Height | Area % | Height % |
| 1 | 6.287 | 82285871 | 2281070 | 95.054 | 98.738 |
| 2 | 79.282 | 4281483 | 29165 | 4.946 | 1.262 |
| Total | | 86567354 | 2310235 | 100.000 | 100.000 |

S 73: HPLC spectrum of Compound (5m) in presence of catalyst (2) and Methanol in RT condition (Entry 13, Table 3).



| PeakTable | | | | | |
|-------------------|-----------|---------|--------|---------|----------|
| PDA Ch1 254nm 4nm | | | | | |
| Peak# | Ret. Time | Area | Height | Area % | Height % |
| 1 | 3.271 | 3004331 | 228818 | 49.625 | 55.432 |
| 2 | 90.532 | 3049796 | 274830 | 50.375 | 44.568 |
| Total | | 6054127 | 503648 | 100.000 | 100.000 |

S 74: HPLC spectrum of Racemic Mixture 5n.

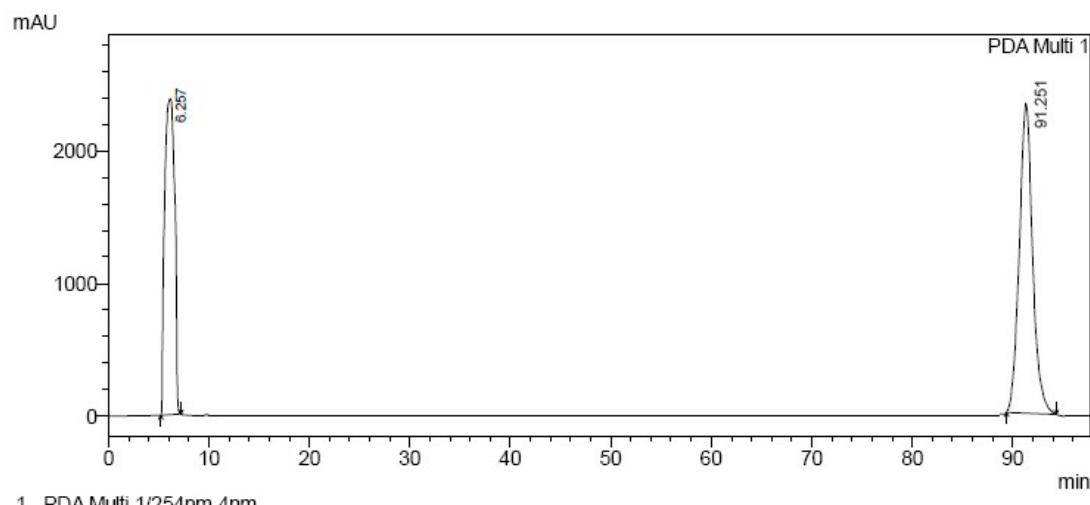


PeakTable

PDA Ch1 254nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|----------|---------|---------|----------|
| 1 | 3.269 | 34845989 | 2728383 | 97.394 | 99.777 |
| 2 | 90.532 | 932415 | 6093 | 2.606 | 0.223 |
| Total | | 35778404 | 2734476 | 100.000 | 100.000 |

S 75: HPLC spectrum of Compound (5n) in presence of catalyst (2) and Methanol in RT condition (Entry 14, Table 3).

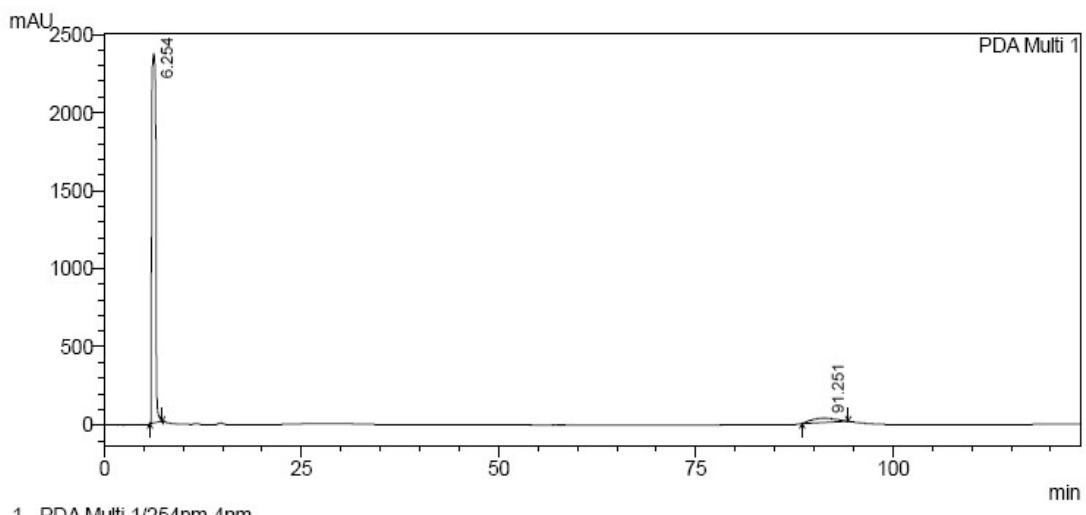


PeakTable

PDA Ch1 254nm 4nm

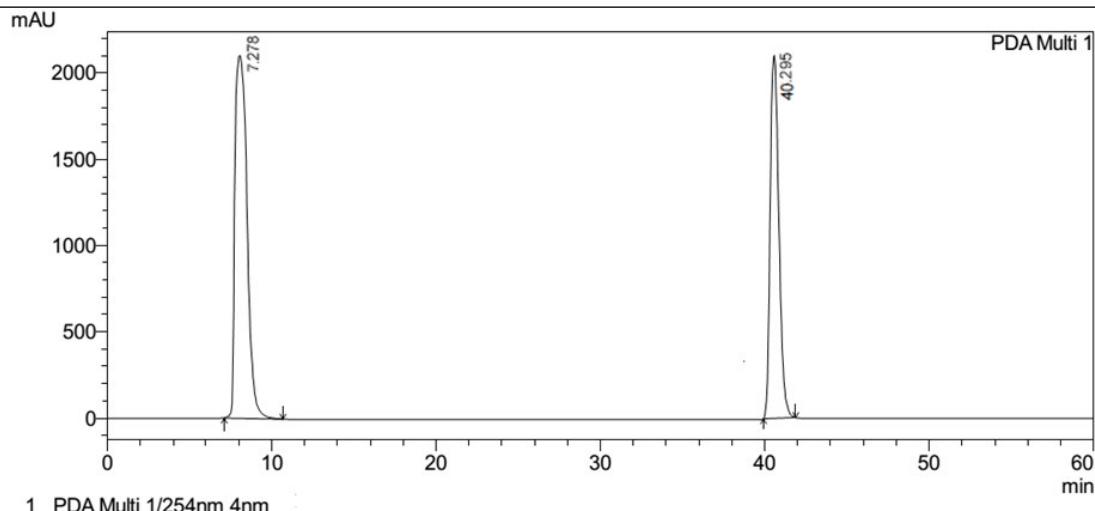
| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|---------|--------|---------|----------|
| 1 | 6.257 | 3004331 | 228818 | 49.625 | 49.432 |
| 2 | 91.251 | 3049796 | 274830 | 50.375 | 50.568 |
| Total | | 6054127 | 503648 | 100.000 | 100.000 |

S 76: HPLC spectrum of Racemic Mixture 5o.



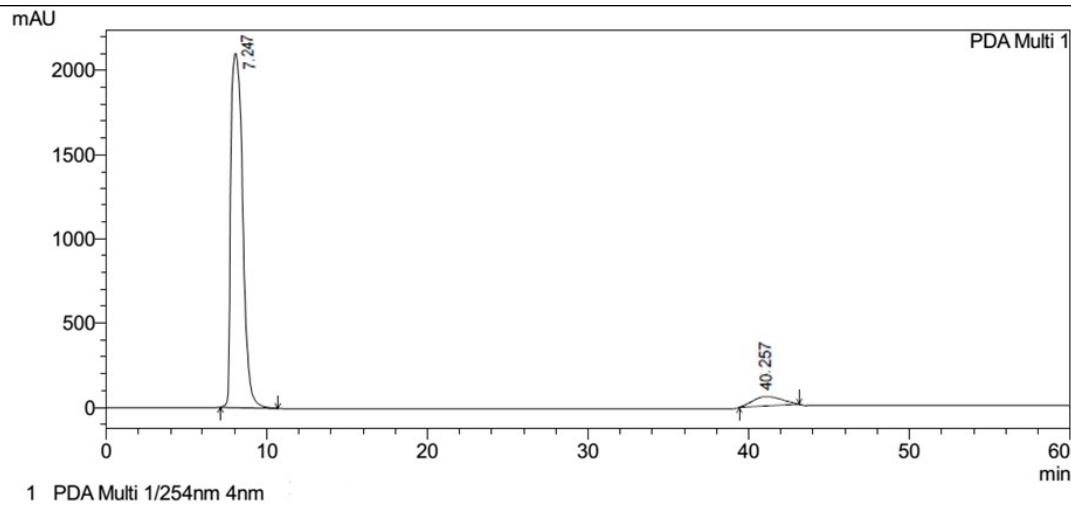
| PeakTable | | | | | |
|-------------------|-----------|----------|---------|---------|----------|
| PDA Ch1 254nm 4nm | | | | | |
| Peak# | Ret. Time | Area | Height | Area % | Height % |
| 1 | 6.254 | 82498582 | 2361240 | 93.413 | 98.828 |
| 2 | 91.251 | 5817610 | 27995 | 6.587 | 1.172 |
| Total | | 88316192 | 2389235 | 100.000 | 100.000 |

S 77: HPLC spectrum of Compound (5o) in presence of catalyst (2) and Methanol in RT condition (Entry 15, Table 3).



| PeakTable | | | | | |
|-------------------|-----------|-----------|---------|---------|----------|
| PDA Ch1 254nm 4nm | | | | | |
| Peak# | Ret. Time | Area | Height | Area % | Height % |
| 1 | 7.278 | 143622513 | 1483220 | 50.741 | 51.794 |
| 2 | 40.295 | 142638897 | 1365126 | 49.259 | 48.206 |
| Total | | 286261409 | 2848346 | 100.000 | 100.000 |

S 78: HPLC spectrum of Racemic Mixture 5p.



PeakTable

PDA Ch1 254nm 4nm

| Peak# | Ret. Time | Area | Height | Area % | Height % |
|-------|-----------|----------|---------|---------|----------|
| 1 | 7.247 | 76821577 | 1226754 | 93.021 | 95.364 |
| 2 | 40.257 | 5711609 | 146005 | 2.979 | 4.636 |
| Total | | 82533186 | 1372758 | 100.000 | 100.000 |

S 79: HPLC spectrum of Compound (5p) in presence of catalyst (2) and Methanol in RT condition (Entry 16, Table 3).