Michael Addition of Oxindoles to \( N-(2\text{-}tert\text{-}butyl\text{-}phenyl)\)maleimides: Efficient Desymmetrization for the Synthesis of Atropisomeric Succinimides with Quaternary and Tertiary Stereocenters

Nicola Di Iorio\(^a\), Lorenzo Soprani\(^a\), Simone Crotti\(^a\), Emanuela Marotta\(^a\), Andrea Mazzanti\(^a\), Paolo Righi\(^a\) and Giorgio Bencivenni\(^*\)\(^a\)

\(^a\)Department of Industrial Chemistry “Toso Montanari”, School of Science, University of Bologna, viale del Risorgimento 4, 40136, Bologna - Italy.

e-mail_giorgio.bencivenni2@unibo.it
Table of contests

- Crystal data for compound 3p  S3
- NMR traces  S4
- HPLC traces  S22
Crystal data for compound 3p

Molecular formula: C_{36}H_{40}N_{2}O_{5.5}Br; \( M_r = 668.61 \), monoclinic, space group \( C2 \), \( a = 18.3224(8) \), \( b = 14.0990(6) \), \( c = 15.04976(7) \) Å, \( \beta = 121.756(2) \); \( V = 3404.1(3) \) Å³, \( T = 298(2) \) K, \( Z = 4 \), \( \rho_e = 1.305 \) g cm⁻³, \( F(000) = 1396 \), graphite-monochromated Mo Kα radiation (\( \lambda = 0.71073 \) Å), \( \mu(\text{Mo}K\alpha) = 1.252 \) mm⁻¹, colourless brick (0.40 × 0.10 × 0.10 mm³), empirical absorption correction with SADABS (transmission factors: 0.634 – 0.885), 3600 frames, exposure time 20 s, \( 1.545 \leq \theta \leq 25.00 \), \(-21 \leq h \leq 21 \), \(-16 \leq k \leq 16 \), \(-18 \leq l \leq 18 \), 20183 reflections collected, 5903 independent reflections (\( R_{\text{int}} = 0.0403 \)), solution by intrinsic phasing method and subsequent Fourier syntheses, full-matrix least-squares on \( F_0^2 \) (SHELXL-2014/7), hydrogen atoms refined with a riding model, data / restraints / parameters = 5903/ 1/391, \( S(F^2) = 1.056 \), \( R(F) = 0.0763 \) and \( wR(F^2) = 0.1530 \) on all data, \( R(F) = 0.0473 \) and \( wR(F^2) = 0.1218 \) for 41100 reflections with \( I > 2\sigma (I) \), weighting scheme \( w = 1/[\sigma^2(F_o^2) + (0.0678P)^2 + 2.5392P] \) where \( P = (F_o^2 + 2F_c^2)/3 \), largest difference peak and hole 0.629 and –0.392 e Å⁻³. Flack parameter for \( R(C_1) \), \( S(C_0) \) absolute configuration: 0.033(5) using Parsons method [Parsons, S; Flack, H.D.; Wagner, T. Acta Cryst. 2013, B69, 249-259]. The unit cell contains a molecule of solvent (hexane) and a disordered water molecule located on a special point (0.5 site occupancy). The hydrogens of the water molecule cannot be experimentally located and the oxygen was isotropically refined. Crystallographic data have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-1511895. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).
NMR TRACES

Sample: NMR_518_pulito_H
File: np
Pulse Sequence: s1pul

Sample: NMR_518_pulito_C
File: sc
Pulse Sequence: s1pul
Sample: MDI 541 sulito H
File: mp
Pulse Sequence: alpul

Standard Carbon Parameters

Sample: MDI 541 sulito H
File: mp
Pulse Sequence: alpul
Sample: MDI_599_polyco_F
File: home/rosini/microcva/data/MDI/MDI 599 polyco_F.fid
Pulse Sequence: s2pol
Pulse Sequence: alpul

NMR Spectra Image
HPLC TRACES

Data File: S:\2\DATA\NDI\NDI_518_RAC_000.D
Sample Name: NDI_518_rac

Acq. Operator: Nico
Acq. Instrument: chiral
Injection Date: 10/01/2017 12:32:08
Inj Volume: 10.0 µl

Acq. Method: S:\2\METHODS\TMP.M
Last changed: 10/01/2017 12:30:07 by Nico
(modified after loading)
Analysis Method: S:\2\METHODS\NDI\NDI-381.M
Last changed: 10/01/2017 12:03:46 by Giada
(modified after loading)
Sample Info: NDI_518_rac; AD-H; Hex/IPA 90/10; 1 mL/min

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=360.100

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Type</th>
<th>Width [min]</th>
<th>Area [mAU*sec]</th>
<th>Height [mAU]</th>
<th>Area [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>VB</td>
<td>0.2717</td>
<td>2385.09351</td>
<td>139.36179</td>
<td>45.8334</td>
</tr>
<tr>
<td>2</td>
<td>VB</td>
<td>0.5714</td>
<td>2818.73438</td>
<td>69.75361</td>
<td>54.1666</td>
</tr>
</tbody>
</table>

Totals: 5203.82788, 209.11539

chiral 10/01/2017 13:01:19 Giada
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=360.100

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>18.992</td>
<td>BB</td>
<td>0.5693</td>
<td>1476.31018</td>
<td>37.82844</td>
<td>100.0000</td>
</tr>
</tbody>
</table>

Totals : 1476.31018 37.82844

*** End of Report ***

Instrument 1 28/10/2016 10:02:10

Page 1 of 1
Data File C:\CHEM32\1\DATA\NDI\NDI_513_514_000.D
Sample Name: NDI_513_514

Acq. Operator : Nico
Acq. Instrument : DAD1100
Injection Date : 12/07/2016 17:52.21
Injection Volume : 10 ul
Last changed : 12/07/2016 17:36.55 by Nico
(modified after loading)
Sample Info : NDI_513_514; AD-H; Hex/iPrOH 70/30; 0.8 mL/min

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

Signal 1, DAD1 A, Sig=254.4 Ref=360.100 (NDI/NDI_513_514_000.D)

Peak RetTime Type Width Area Height Area %
#   [min]   [min]  [mAU*s]  [mAU]   |
1   5.792 BB 0.1370 3307.30664 374.69611 52.7855
2   8.333 VB 0.2557 2958.25610 180.02423 47.2145

Total : 6265.56274 554.72034

*** end of report ***
Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1

Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig-254,4 Ref-360,100

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime [min]</th>
<th>Width [min]</th>
<th>Area [mAU*s]</th>
<th>Height [mAU]</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5.819</td>
<td>0.1425</td>
<td>29.11152</td>
<td>3.13148</td>
<td>1.1647</td>
</tr>
<tr>
<td>2</td>
<td>8.346</td>
<td>0.2594</td>
<td>2470.44800</td>
<td>149.02612</td>
<td>98.8353</td>
</tr>
</tbody>
</table>

Total : 2499.55952 152.13760

*** end of report ***
Data File: S:\2\DATA\NDI\NDI_561_562_001.D
Sample Name: NDI_561_562

Acq. Operator: Nico
Acq. Instrument: chiral
Injection Date: 21/10/2016 16:10:45
Inj Volume: 10.0 ul

Acq. Method: S:\2\METHODS\TMP.M
Last changed: 21/10/2016 16:00:59 by Nico
(modified after loading)

Analysis Method: S:\1\METHODS\TMP.M
Last changed: 21/10/2016 15:26:25 by Nico
(modified after loading)

Sample Info: NDI_561_562, AD-H, Hex/IPA 90/10, 1 mL/min

Additional Info: Peak(s) manually integrated

Area Percent Report

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>7.942</td>
<td>BB</td>
<td>0.2160</td>
<td>1493.30151</td>
<td>101.06313</td>
</tr>
<tr>
<td></td>
<td>11.297</td>
<td>VM</td>
<td>0.4999</td>
<td>2739.55607</td>
<td>31.34191</td>
</tr>
</tbody>
</table>

Totals: 3193.89758 152.40504

chiral 21/10/2016 16:27:51 Nico
Data File S:\\DATA\NDI\NDI_561_000000.D
Sample Name: NDI_561

Acq. Operator : Nico
Acq. Instrument : chiral
Injection Date : 21/10/2016 16:28:10
Inj Volume : 10.0 ul

Acq. Method : S:\\\2\METHODS\TMP.M
Last changed : 21/10/2016 16:26:36 by Nico
(modified after loading)

Analysis Method : S:\\\2\METHODS\TMP.W
Last changed : 21/10/2016 15:26:25 by Nico
(modified after loading)

Sample Info : NDI_561, AD-H, Hex/IPA 90/10, 1 mL/min

Additional Info : Peak(s) manually integrated

Additional Data:

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

<table>
<thead>
<tr>
<th>Peak</th>
<th>RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>8.003</td>
<td>BB</td>
<td>0.2097</td>
<td>39.73603</td>
<td>2.76448</td>
<td>0.9822</td>
</tr>
<tr>
<td>2</td>
<td>13.242</td>
<td>PK</td>
<td>0.5813</td>
<td>4005.62910</td>
<td>114.74101</td>
<td>29.0178</td>
</tr>
</tbody>
</table>

Totals : 4045.56513 117.50549

Page 1 of 1

chiral 21/10/2016 16:45:12 Nico
Data File: C:\CHEM32\DATA\NDI\NDI_509_510_002.D
Sample Name: NDI_509_510

Acq. Operator: Nico
Acq. Instrument: DAD100
Injection Date: 12/07/2016 14.07.50
Injection Volume: 10 ul

Acq. Method: C:\CHEM32\METHODS\TMP.M
Last changed: 12/07/2016 12.23.32 by Nico
(modified after loading)
Analysis Method: C:\\CHEM32\\METHODS\\TMD.W
Last changed: 11/07/2016 18.43.57 by Nico
Sample Info: NDI_509_510; AD-H; Hex/iPrOH 70/30; 0.8 mL/min

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1

Use Multiplier & Dilution Factor with ISTDs

Signal 1, DAD1 A, Sig=254.4 Ref=360.100 (NDI\NDI_509_510_002.D)

<table>
<thead>
<tr>
<th>Peak</th>
<th>RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>7.279</td>
<td>BB</td>
<td>0.1707</td>
<td>693.44891</td>
<td>62.84266</td>
<td>39.9129</td>
</tr>
<tr>
<td>2</td>
<td>21.322</td>
<td>BB</td>
<td>0.6449</td>
<td>1043.95496</td>
<td>22.85708</td>
<td>60.0871</td>
</tr>
</tbody>
</table>

Totals: 1737.40357 55.05974

*** end of report ***
Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1

Use Multiplier & Dilution Factor with ISTDs

Signal 1, DAD1 A, Sig=254.4 Ref=360.100

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime [min]</th>
<th>Width [min]</th>
<th>Area [mAU*s]</th>
<th>Height [mAU]</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>7.324 MM</td>
<td>0.2175</td>
<td>37.89753</td>
<td>2.90406</td>
<td>1.5436</td>
</tr>
<tr>
<td>2</td>
<td>21.311 MM</td>
<td>0.7794</td>
<td>2417.30957</td>
<td>51.69341</td>
<td>98.4564</td>
</tr>
</tbody>
</table>

Totals: 2455.20710 54.59747

*** End of report ***
Data File S:\2\DATA\NDI\NDI_537_538_000.D
Sample Name: NDI_537_538

Acq. Operator : Nico
Acq. Instrument : chiral
Location : Vial 36
Injection Date : 16/09/2016 16:00:06
Inj Volume : 10.0 µl
Acq. Method : S:\2\METHODS\TMP.M
Last changed : 16/09/2016 15:58:21 by Nico
(modified after loading)
Analysis Method : S:\2\METHODS\TMP.M
Last changed : 27/09/2016 18:34:41 by Nico
(modified after loading)
Sample Info : NDI_537_538, AD-H, Hex/IPA 80/20, 1 mL/min

Additional Info : Peak(s) manually integrated

Signal 1: DAD1 A, Sig=254.4 Ref=360,100

<p>| | | | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>#</td>
<td>Width</td>
<td>Area</td>
<td>Height</td>
<td>Area %</td>
</tr>
<tr>
<td>-----</td>
<td>-------</td>
<td>------</td>
<td>--------</td>
<td>-------</td>
</tr>
<tr>
<td>1</td>
<td>6.436</td>
<td>0.1763</td>
<td>3179.42334</td>
<td>280.28531</td>
</tr>
<tr>
<td>2</td>
<td>12.282</td>
<td>0.4831</td>
<td>5073.4276</td>
<td>57.68762</td>
</tr>
</tbody>
</table>

Totals : 6252.85034 377.97293

Area Percent Report

Use Multiplier & Dilution Factor with ISTDs
Data File C:\CHEM32\DATA\NDI\NDI_511_512_000.D
Sample Name: NDI_511_512

Acq. Operator : Nico
Acq. Instrument : DAD1100
Injection Date : 12/07/2016 14.35.03
Inj Volume : 10 ul

Acq. Method : C:\CHEM32\METHODS\MPM.M
Last changed : 12/07/2016 12.23.32 by Nico
(modified after loading)
Analysis Method : C:\CHEM32\METHODS\MPM.W
Last changed : 11/07/2016 18.43.57 by Nico
Sample Info : NDI_511_512; AD-H; Hex/iPrOH 70/30; 0.8 mL/min

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1

Use Multiplier & Dilution Factor with ISTDs

Signal 1, DAD1 A, Sig-254.4 Ref-360,100

<table>
<thead>
<tr>
<th>#</th>
<th>[min]</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5.440</td>
<td>BB</td>
<td>0.1249</td>
<td>3507.26685</td>
<td>430.95261</td>
<td>52.7955</td>
</tr>
<tr>
<td>2</td>
<td>8.520</td>
<td>VB</td>
<td>0.3088</td>
<td>3135.85181</td>
<td>158.53853</td>
<td>47.2045</td>
</tr>
</tbody>
</table>

Totals : 6643.11865 555.49113

*** end of report ***
Area Percent Report

Sorted By : Signal
Multiplier : 1.0000

Use Multiplier & Dilution Factor with ISTDs

---

Signal 1, DAD1 A, Sig=254.4 Ref=360,100

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5.439</td>
<td>MM</td>
<td>0.1238</td>
<td>21.52517</td>
<td>2.89436</td>
<td>0.9551</td>
</tr>
<tr>
<td>2</td>
<td>8.517</td>
<td>BB</td>
<td>0.3124</td>
<td>2232.26660</td>
<td>112.04066</td>
<td>99.0449</td>
</tr>
</tbody>
</table>

Totals : 2253.7577 114.23562

---

*** End of report ***
Data File S:\2\DATA\NDI\NDI_530_000008.D
Sample Name: NDI_530

Acq. Operator : Nico
Acq. Instrument : chiral
Injection Date : 29/09/2016 11:55:09
Injection Volume : 10.0 ul

Acq. Method : S:\2\METHOD\TMP.M
(modified after loading)

Analysis Method : S:\2\METHOD\TMP\W
Last changed : 27/09/2016 18:05:22 by Nico
Sample Info : NDI_530, IC, Hex/IPA 80/20, 1 mL/min

Additional Info : Peak(s) manually integrated

Area Percent Report

Sorted By : Signal
Multiplier: 1.0000
Dilution: 1
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254.4 Ref=360.100

Peak RetTime Type Width Area Height Area %
--- | --- | --- | --- | --- | ---
1 9.972 MM 0.4648 1669.59753 59.86451 62.7407
2 25.015 MM 1.1075 991.51086 14.92160 37.2593
Totals : 2661.10840 74.78611

*** End of Report ***
Data File S:\2\DATA\NDI\NDI_529_000008.D
Sample Name: NDI_529

Acq. Operator: Nico
Acq. Instrument: chiral
Inj. Volume: 10.0 ul
Injection Date: 29/09/2016 11:16:35
Injection Volume: 10.0 ul
Last changed: 29/09/2016 11:15:08 by Nico
(modified after loading)
Sample Info: NDI_529, IC, Hex/IPA 80/20, 1 mL/min

Additional Info: Peak(s) manually integrated

Area Percent Report

Signal: DAD1 A, Sig=254.4 Ref=360.100
Multiplier: 1
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISSTDs

Signal 1: DAD1 A, Sig=254.4 Ref=360.100

Peak RetTime Type Width Area Height Area %
# [min] [min] [mAU*sec] [mAU] 
1 9.911 MM 0.4812 4159.33252 144.05690 97.0091
2 25.088 MM 0.9271 128.23763 2.30541 2.9309

Totals: 4287.57014 146.36231

*** End of Report ***

chiral 29/09/2016 12:34:19 Nico
Data File S:\2\DATA\SC\SC_031_032_0004.D
Sample Name: SC_031_032

Acq. Operator : Simone
Acq. Instrument : chiral
Injection Date : 09/01/2017 15:29:58
Location : Vial 11
Inj Volume : 10.0 ul

Acq. Method : S:\2\METHODS\TMP.M
Last changed by : 09/01/2017 14:53:17 by Simone
(modified after loading)

Analysis Method : S:\2\METHODS\NN1\NN1-01.M
Last changed by : 09/01/2017 17:53:52 by Giada
(modified after loading)

Sample Info : SC_031_032; IC; Hex/IPA 80/20, 1 mL/min

Additional Info : Peak(s) manually integrated

---

Signal 1: DAD1 A, Sig=254.4 Ref=360.100
Use Multiplier & Dilution Factor with ISTDs

Multiplier: 1.0000
Dilution: 1.0000

Signal: DAD1 A, Sig=254.4 Ref=360.100
Multiplier & Dilution Factor with ISTDs

Peak RetTime Type Width Area Height Area %
-----|------|--------|--------|--------|-----|
1 11.986 BV 0.5244 1462.28839 42.18227 47.0401
2 24.672 DM 1.3154 1546.03790 16.69441 51.2739

Totals : 3108.19629 58.84667

---

chiral 09/01/2017 17:54:08 Giada

Page 1 of 1
Data File S:\2\DATA\SC\SC_028_029_0000.D
Sample Name: SC_028_029

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

Acq. Operator : Simone
Acq. Instrument : chiral Location : Vial 30
Injection Date : 19/10/2016 13:21:49 Inj Volume : 10.0 ul
Acq. Method : S:\2\METHODS\TMP.W Last changed : 19/10/2016 13:16:24 by Nico
Analysis Method : R:\\2\METHODS\TMP.W (modified after loading)
Last changed : 29/09/2016 12:38:01 by Nico
Sample Info : SC_028_029, AD-H, Hex/IPA 80/20, 1 mL/min

Additional Info : Peak(s) manually integrated

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

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

Sorted By : Signal
Multiplier : 1
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISSTDs

Signal 1: DAD1 A, Sig=254.4 Ref=360,100

Peak RetTime Type Width Area Height Area
# [min] [min] [mAU*a] [mAU] %
--- |--------|--------|--------|--------|--------|--------|
1 7.176 BB 0.1974 3966.94092 314.03513 56.2874
2 14.662 BB 0.5180 3080.71411 92.14939 43.7126

Totals : 7047.65503 406.18452

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

*** End of Report ***

chiral 19/10/2016 14:06:33 Simone
Data File S:\2\DATA\SC\SC_028_000000.D
Sample Name: SC_028

Acq. Operator : Simone
Acq. Instrument : chiral
Injection Date : 19/10/2016 13:58:56
Inj Volume : 10.0 µl

Acq. Method : S:\2\METHODS\TMP.W
Last changed : 19/10/2016 13:56:59 by Simone
(modified after loading)

Analysis Method : R:\2\METHODS\Cmpd Method 1
Last changed : 29/09/2016 12:38:01 by Nico

Sample Info : SC_028, AD-M, Hex/IPA 80/20, 1 mL/min

Additional Info : Peak(s) manually integrated

Area Percent Report

<table>
<thead>
<tr>
<th>Signal</th>
<th>Multiplier</th>
<th>Dilution</th>
<th>Use Multiplier &amp; Dilution Factor with IS Factor</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1</td>
<td>1.0000</td>
<td></td>
</tr>
</tbody>
</table>

Signal 1: DAD1 A, Sig=254.4 Ref=360,100

<table>
<thead>
<tr>
<th>Peak</th>
<th>RetTime</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>[min]</td>
<td>[min]</td>
<td>[mAU*a]</td>
<td>[mAU]</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>7.198</td>
<td>0.2046</td>
<td>126.9997</td>
<td>9.58648</td>
<td>1.9058</td>
</tr>
<tr>
<td>2</td>
<td>14.626</td>
<td>0.5304</td>
<td>6536.68750</td>
<td>192.36905</td>
<td>98.0942</td>
</tr>
</tbody>
</table>

Totals : 6663.68727 201.95553

*** End of Report ***

chiral 19/10/2016 14:50:27 Simone
Acq. Operator : Nico
Acq. Instrument : chiral
Injection Date : 16/09/2016 13:31:44
Inj Volume : 10.0 ul

Acq. Method : S:\2\METHODS\TMP.W
(modified after loading)
Analyzr Method : S:\2\METHODS\MPW.W
Last changed : 27/09/2016 17:14:38 by Nico
(modified after loading)
Sample Info : NDI_535_536, AD-H, Hex/IPA 80/20, 1 mL/min

Additional Info : Peak(s) manually integrated
**Data File:** S:\2\DATA\NDI\NDI_535_000000.D  
**Sample Name:** NDI_535

<table>
<thead>
<tr>
<th>Acq. Operator</th>
<th>Nico</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acq. Instrument</td>
<td>chiral</td>
</tr>
<tr>
<td>Location</td>
<td>Vial 35</td>
</tr>
<tr>
<td>Injection Date</td>
<td>16/09/2016 14:13:38</td>
</tr>
<tr>
<td>Inj Volume</td>
<td>10.0 µl</td>
</tr>
<tr>
<td>Acq. Method</td>
<td>S:\2\METHODS\TMP.W</td>
</tr>
<tr>
<td>Last changed</td>
<td>16/09/2016 14:12:37 by Nico</td>
</tr>
<tr>
<td>(modified after loading)</td>
<td></td>
</tr>
<tr>
<td>Analytical Method</td>
<td>S:\2\METHODS\TMP.W</td>
</tr>
<tr>
<td>Last changed</td>
<td>27/09/2016 18:30:38 by Nico</td>
</tr>
<tr>
<td>(modified after loading)</td>
<td></td>
</tr>
<tr>
<td>Sample Info</td>
<td>NDI_535, ADN, Hex/IPA 80/20, 1 mL/min</td>
</tr>
</tbody>
</table>

**Additional Info:** Peak(s) manually integrated

**Area Percent Report**

<table>
<thead>
<tr>
<th>Corrected By</th>
<th>Signal</th>
</tr>
</thead>
<tbody>
<tr>
<td>Multiplier:</td>
<td>1.0000</td>
</tr>
<tr>
<td>Dilution:</td>
<td>1.0000</td>
</tr>
</tbody>
</table>

Use Multiplier & Dilution Factor with ISTDs

**Signal 1: DAD1 A, Sig=254,4 Ref=360,100**

<table>
<thead>
<tr>
<th>Peak</th>
<th>RetTime</th>
<th>Type</th>
<th>Width [min]</th>
<th>Area [mAU*sec]</th>
<th>Height [mAU]</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5.308</td>
<td>BB</td>
<td>0.1755</td>
<td>22.28744</td>
<td>1.73878</td>
<td>0.9266</td>
</tr>
<tr>
<td>2</td>
<td>7.224</td>
<td>BR</td>
<td>0.2655</td>
<td>23.92.67294</td>
<td>117.36746</td>
<td>25.0754</td>
</tr>
</tbody>
</table>

**Totals:**

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>2405.16708</td>
</tr>
<tr>
<td></td>
<td>139.04624</td>
</tr>
</tbody>
</table>

**chiral 27/09/2016 18:31:00 Nico**
Data File S:\2\DATA\NDI\NDI_545_546_000.D
Sample Name: NDI_545_546

Additional Info: Peak(s) manually integrated

Area Percent Report

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type Width Area Height Area %

1  6.732  BB  0.1679  2712.30542  251.26443  49.4523
2 10.210  RM  0.3235  2772.35698  132.01585  50.5477

Totals:
5484.69238  383.07827

chiral 27/09/2016 11:44:28 Nico
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

<table>
<thead>
<tr>
<th>Peak</th>
<th>RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Height</th>
<th>Area</th>
<th>[mAU*s]</th>
<th>[mAU]</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5.681</td>
<td>BB</td>
<td>0.1881</td>
<td>3078.00293</td>
<td>236.02208</td>
<td>97.8101</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>6.203</td>
<td>RV</td>
<td>0.3174</td>
<td>69.21250</td>
<td>4.35401</td>
<td>2.1522</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Totals: 3146.91813 240.40609
Data File: S:\DATA\NDI\NDI_543_544_012.D
Sample Name: NDI_543

Acq. Operator: Nico
Acq. Instrument: chiral
Injection Date: 26/09/2016 16:38:12
Inj Volume: 10.0 µl
Acq. Method: S:\2\METHODS\TMP.M
Last Changed: 26/09/2016 16:35:35 by Nico

Analysis Method: S:\2\METHODS\NDI\NDI-301.M
Last changed: 10/01/2017 12:03:46 by Giada
Sample Info: NDI_543, AD-M, Hex/IPA 80/20, 0.8 mL/min

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=360,100

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime</th>
<th>Width</th>
<th>Area [mAU*s]</th>
<th>Height [mAU]</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>6.214 PM</td>
<td>0.2371 1277.71802</td>
<td>89.81195</td>
<td>53.9679</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>6.172 MM</td>
<td>0.1719 1089.83521</td>
<td>108.63887</td>
<td>46.0321</td>
<td></td>
</tr>
</tbody>
</table>

Totals: 2367.55322 195.45082

chiral 10/01/2017 12:16:09 Giada
Sample Name: NDI_543

Acq. Operator: Nico
Acq. Instrument: chiral
Injection Date: 26/09/2016 16:38:12
Injection Volume: 10.0 μl

Acq. Method: \S:\2\METHODS\TMP\W
Last changed: 26/09/2016 16:36:35 by Nico
(modified after loading)

Analysis Method: \S:\2\METHODS\TMP\W
Last changed: 27/09/2016 18:34:41 by Nico
(modified after loading)

Sample Info: NDI_543, AD-H, Hex/IPA 80/20, 0.8 mL/min

Additional Info: Peak(s) manually integrated

Signal 1: DAD1 A, Sig=254.4 Ref=360.100

Peak RetTime Type Width Area Height Area %
--- | ----- | ----- | -------- | ------ | ----- | ------ | -----
1  5.252 | BB | 0.1344 | 6.43026 | 6.92377e-1 | 0.1559
2  6.175 | BB | 0.3032 | 417.40527 | 206.02629 | 25.8441

Totals: 4123.83553 206.71867

Additional Info: Peak(s) manually integrated
Data File S:\2\DATA\NDI\NDI_541_542_005.D
Sample Name: NDI_541_542

================================================================================================
Acq. Operator : Nico  Location : Vial 40
Acq. Instrument : chiral  Inj Volume : 10.0 µl
Injection Date : 27/09/2016 14:42:31
Acq. Method : S:\2\METHODS\TMP.M
Last changed : 27/09/2016 14:29:44 by Nico
(modified after loading)
Analysis Method : S:\2\METHODS\TMP.M
Last changed : 27/09/2016 17:14:38 by Nico
(modified after loading)
Sample Info : NDI_541_542, AD-H, Hex/IPA 80/20, 0.8 mL/min

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-360.100

Peak RetTime Type Width Area Height Area %
--- | ------ | ------- | ----- | ------ | ------- | ----|
1  17.699  MP  0.9797  3583.09180   60.95808  39.8719
2  36.100  MM  2.6219  5403.41553   34.34755  60.1281

Totals : 8986.50732  95.30564

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

chiral 27/09/2016 17:28:13 Nico
Data File S:\2\DATA\NDI\NDI_541_000001.D
Sample Name: NDI_541

Acq. Operator : Nico
Acq. Instrument : chiral
Injection Date : 27/09/2016 15:27:16
Inj Volume : 10.0 µl

Acq. Method : S:\2\METHODS\TMP.M
Last changed : 27/09/2016 15:25:52 by Nico
(modified after loading)
Analysis Method : S:\2\METHODS\TMP.M
Last changed : 27/09/2016 17:14:38 by Nico
(modified after loading)
Sample Info : NDI_541, AD-H, Hex/IPA 80/20, 0.8 mL/min

Additional Info : Peak(s) manually integrated

![Chemical Structure](image)

---

**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=360,100**

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>17.792</td>
<td>MF</td>
<td>0.9772</td>
<td>695.98599</td>
<td>11.87059</td>
<td>7.7950</td>
</tr>
<tr>
<td>2</td>
<td>35.811</td>
<td>MM</td>
<td>2.6070</td>
<td>8228.86984</td>
<td>52.60294</td>
<td>92.2010</td>
</tr>
</tbody>
</table>

**Totals** : 8924.07983 64.47354
Data File S:\2\DATA\NDI\NDI_536_000000.D
Sample Name: NDI_536

Acq. Operator : Nico
Acq. Instrument : chiral
Injection Date : 16/09/2016 15:34:14
Inj Volume : 10.0 ul

Acq. Method : S:\2\METHODS\TMP.M
Last changed : 16/09/2016 15:32:53 by Nico
(modified after loading)

Analysis Method : S:\2\METHODS\TMP.M
Last changed : 27/09/2016 18:30:38 by Nico
(modified after loading)

Sample Info : NDI_536, AD-H, Hex/IPA 80/20, 1 mL/min

Additional Info : Peak(s) manually integrated

Signal 1: DAD1 A, Sig=254.4 Ref=360.100

<table>
<thead>
<tr>
<th>Peak</th>
<th>RetTime</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5.244</td>
<td>0.1394</td>
<td>2.237</td>
<td>0.00313</td>
<td>247.52386</td>
</tr>
<tr>
<td>2</td>
<td>7.257</td>
<td>0.2336</td>
<td>0.2336</td>
<td>0.2336</td>
<td>111.29333</td>
</tr>
</tbody>
</table>

Total: 2348.30506 253.96413

chiral 27/09/2016 18:32:25 Nico