Synthesis of functionalized polyhalogenated thiophene derivatives

Nikita A. Chaika¹, Kostiantyn V. Shvydenko²,
Tetiana I. Shvydenko², Aleksandr N. Kostyuk²*

¹ National Technical University of Ukraine "Igor Sikorsky Kyiv Polytechnic Institute",
37 Peremohy Ave., Kyiv 03056, Ukraine; e-mail: nikitachayka78@gmail.com

² Institute of Organic Chemistry, National Academy of Sciences of Ukraine,
5 Akademika Kukharya St., Kyiv 02094, Ukraine; e-mail: a.kostyuk@yahoo.com

Supplementary Information
<table>
<thead>
<tr>
<th>Compound #</th>
<th>Description</th>
<th>Page</th>
<th>Compound #</th>
<th>Description</th>
<th>Page</th>
<th>Compound #</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>2a</td>
<td>$^1$H NMR</td>
<td>S1</td>
<td>3a</td>
<td>APT</td>
<td>S21-S23</td>
<td>4a</td>
<td>APT</td>
<td>S43</td>
</tr>
<tr>
<td>2a</td>
<td>$^{13}$C NMR</td>
<td>S2</td>
<td>3b</td>
<td>$^1$H NMR</td>
<td>S24</td>
<td>4a</td>
<td>MS</td>
<td>S44-S45</td>
</tr>
<tr>
<td>2a</td>
<td>APT</td>
<td>S3</td>
<td>3b</td>
<td>$^{13}$C NMR</td>
<td>S25</td>
<td>4b</td>
<td>$^1$H NMR</td>
<td>S46</td>
</tr>
<tr>
<td>2a</td>
<td>MS</td>
<td>S4-S6</td>
<td>3b</td>
<td>APT</td>
<td>S26</td>
<td>4b</td>
<td>$^{13}$C NMR</td>
<td>S47</td>
</tr>
<tr>
<td>2b</td>
<td>$^1$H NMR</td>
<td>S7</td>
<td>3b</td>
<td>MS</td>
<td>S27-S28</td>
<td>4b</td>
<td>ATP</td>
<td>S48</td>
</tr>
<tr>
<td>2b</td>
<td>$^{13}$C NMR</td>
<td>S8</td>
<td>3c</td>
<td>$^1$H NMR</td>
<td>S29</td>
<td>4b</td>
<td>MS</td>
<td>S49-S51</td>
</tr>
<tr>
<td>2b</td>
<td>APT</td>
<td>S9</td>
<td>3c</td>
<td>$^{13}$C NMR</td>
<td>S30</td>
<td>4c</td>
<td>$^1$H NMR</td>
<td>S52</td>
</tr>
<tr>
<td>2b</td>
<td>MS</td>
<td>S10</td>
<td>3c</td>
<td>APT</td>
<td>S31</td>
<td>4c</td>
<td>$^{13}$C NMR</td>
<td>S53</td>
</tr>
<tr>
<td>2c</td>
<td>$^1$H NMR</td>
<td>S11</td>
<td>3c</td>
<td>MS</td>
<td>S32-S35</td>
<td>4c</td>
<td>APT</td>
<td>S54</td>
</tr>
<tr>
<td>2c</td>
<td>$^{13}$C NMR</td>
<td>S12</td>
<td>3d</td>
<td>$^1$H NMR</td>
<td>S36</td>
<td>4c</td>
<td>MS</td>
<td>S55-S58</td>
</tr>
<tr>
<td>2c</td>
<td>APT</td>
<td>S13</td>
<td>3d</td>
<td>$^{13}$C NMR</td>
<td>S37</td>
<td>4d</td>
<td>$^1$H NMR</td>
<td>S59</td>
</tr>
<tr>
<td>2c</td>
<td>MS</td>
<td>S14-S17</td>
<td>3d</td>
<td>APT</td>
<td>S38</td>
<td>4d</td>
<td>$^{13}$C NMR</td>
<td>S60</td>
</tr>
<tr>
<td>3a</td>
<td>$^1$H NMR</td>
<td>S18</td>
<td>3d</td>
<td>MS</td>
<td>S39-S40</td>
<td>4d</td>
<td>APT</td>
<td>S61</td>
</tr>
<tr>
<td>3a</td>
<td>$^{13}$C NMR</td>
<td>S19</td>
<td>4a</td>
<td>$^1$H NMR</td>
<td>S41</td>
<td>4d</td>
<td>MS</td>
<td>S62-S65</td>
</tr>
<tr>
<td>3a</td>
<td>APT</td>
<td>S20</td>
<td>4a</td>
<td>$^{13}$C NMR</td>
<td>S42</td>
<td>5a</td>
<td>$^1$H NMR</td>
<td>S66</td>
</tr>
<tr>
<td>3a</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>5d</td>
</tr>
<tr>
<td>Compound #</td>
<td>Description</td>
<td>Page</td>
<td>Compound #</td>
<td>Description</td>
<td>Page</td>
<td>Compound #</td>
<td>Description</td>
<td>Page</td>
</tr>
<tr>
<td>------------</td>
<td>-------------</td>
<td>------</td>
<td>------------</td>
<td>-------------</td>
<td>------</td>
<td>------------</td>
<td>-------------</td>
<td>------</td>
</tr>
<tr>
<td>6a</td>
<td>$^1$H NMR</td>
<td>S82</td>
<td>6c</td>
<td>MS</td>
<td>S99-102</td>
<td>7c</td>
<td>$^1$H NMR</td>
<td>S124</td>
</tr>
<tr>
<td>6a</td>
<td>$^{13}$C NMR</td>
<td>S83</td>
<td>6d</td>
<td>$^1$H NMR</td>
<td>S103</td>
<td>7c</td>
<td>$^{13}$C NMR</td>
<td>S125</td>
</tr>
<tr>
<td>6a</td>
<td>APT</td>
<td>S84</td>
<td>6d</td>
<td>$^{13}$C NMR</td>
<td>S104</td>
<td>7c</td>
<td>APT</td>
<td>S126</td>
</tr>
<tr>
<td>6a</td>
<td>$^{19}$F NMR</td>
<td>S85</td>
<td>6d</td>
<td>APT</td>
<td>S105</td>
<td>7c</td>
<td>MS</td>
<td>S127-129</td>
</tr>
<tr>
<td>6a</td>
<td>MS</td>
<td>S86-S88</td>
<td>6d</td>
<td>$^{19}$F NMR</td>
<td>S106</td>
<td>7d</td>
<td>$^1$H NMR</td>
<td>S130</td>
</tr>
<tr>
<td>6b</td>
<td>$^1$H NMR</td>
<td>S89</td>
<td>6d</td>
<td>MS</td>
<td>S107-108</td>
<td>7d</td>
<td>$^{13}$C NMR</td>
<td>S131</td>
</tr>
<tr>
<td>6b</td>
<td>$^{13}$C NMR</td>
<td>S90</td>
<td>7a</td>
<td>$^1$H NMR</td>
<td>S109</td>
<td>7d</td>
<td>APT</td>
<td>S132</td>
</tr>
<tr>
<td>6b</td>
<td>APT</td>
<td>S91</td>
<td>7a</td>
<td>$^{13}$C NMR</td>
<td>S110</td>
<td>7d</td>
<td>MS</td>
<td>S133-135</td>
</tr>
<tr>
<td>6b</td>
<td>$^{19}$F NMR</td>
<td>S92</td>
<td>7a</td>
<td>APT</td>
<td>S111</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>6b</td>
<td>MS</td>
<td>S93-S94</td>
<td>7a</td>
<td>MS</td>
<td>S112-115</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>6c</td>
<td>$^1$H NMR</td>
<td>S95</td>
<td>7b</td>
<td>$^1$H NMR</td>
<td>S116</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>6c</td>
<td>$^{13}$C NMR</td>
<td>S96</td>
<td>7b</td>
<td>$^{13}$C NMR</td>
<td>S117</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>6c</td>
<td>APT</td>
<td>S97</td>
<td>7b</td>
<td>APT</td>
<td>S118</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>6c</td>
<td>$^{19}$F NMR</td>
<td>S98</td>
<td>7b</td>
<td>MS</td>
<td>S119-123</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
PHts-46_C13apt.fid

Cl

I

2a

File name: PHts-46_C13apt.fid
Operator: 
SF: 150.8288 MHz
NSC: 0
PW: 4.63 usec, RG: 60
SI: 131072
Date: 18-Sep-2022
Solvent: cdcl3
SW: 39063 Hz
TE: 298 K
AQ: 1.23 sec, RD: 0.00 sec
Library Search Report

Data Path: C:\msdchem\1\data\09_16\nData File: IOH58207.D
Acq On: 16 Sep 2022 19:58
Operator:
Sample: IOH58207
Misc: CH3OH
ALS Vial: 62 Sample Multiplier: 1

Search Libraries: C:\Database\EMPTY.L  Minimum Quality: 0

Unknown Spectrum: Apex
Integration Events: ChemStation Integrator - autoint1.e

Abundance TIC: IOH58207.D\data.ms

Time—> 0 2.00 2.50 3.00 3.50 4.00 4.50 5.00 5.50 6.00 6.50 7.00 7.50 8.00 8.50 9.00 10.00 10.50 11.00 11.50 12.00 12.50 13.00 13.50 14.00 14.50 15.00 15.50 16.00

A 6.355 7.218

GCMS-3.M Sat Sep 17 09:22:15 2022 1
Unknown Spectrum based on Apex

Data File: \msdchem\1\data\09_16\IOH58207.D
Sample : IOH58207
Peak Number: 1 at 6.355 min Area: 68281633 Area % 98.97

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td>C:\Database\EMPTY.L</td>
<td>No hits were retrieved.</td>
<td></td>
</tr>
</tbody>
</table>
Unknown Spectrum based on Apex

Scan 940 (7.219 min): IOH58207.D\data.ms

Data File: C:\msdchem\1\data\09_16\IOH58207.D
Sample : IOH58207
Peak Number: 2 at 7.218 min Area: 708025 Area % 1.03

The 3 best hits from each library. 

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td>C:\Database\EMPTY.L</td>
<td>No hits were retrieved.</td>
<td></td>
</tr>
</tbody>
</table>

---

Abundance

Scan 940 (7.219 min): IOH58207.D\data.ms

m/z 287.80 100.00%
m/z 289.80 99.16%
m/z 243.90 94.97%
m/z 81.90 87.33%
m/z 44.00 57.49%
File name: phts47.fid  
Date: 15-Sep-2022  
Operator:  
Solvent: cdcl3  
SF: 399.9714 MHz  
SW: 8000 Hz  
NSC: 0  
TE: 298 K  
PW: 10.90 usec  
RG: 12  
AQ: 2.00 sec  
RD: 0.00 sec  
SI: 32768
PHts-47_C13

File name: PHts-47_C13
Operator: root
SF: 125.6681 MHz
NSC: 1731
PW: 0.00 usec, RG: 51200
AQ: 0.78 sec, RD: 0.00 sec
SI: 131072
Date: 17-Sep-2022
Solvent: DMSO
SW: 32680 Hz
TE: 683 K

2b
PHts-47_C13apt

File name: PHts-47_C13apt
Operator: root
Date: 17-Sep-2022
Solvent: DMSO

SF: 125.6681 MHz
NSC: 344
PW: 0.00 usec, RG: 51200
SI: 65536

SW: 32680 Hz
TE: 683 K
AQ: 1.57 sec, RD: 0.00 sec
MaxPeak: 100.00%
Ret_Time: 1.423 min

**Mol Wt**

<table>
<thead>
<tr>
<th>Exact Mass</th>
<th>#</th>
<th>Time</th>
<th>Area%</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1</td>
<td>1.423</td>
<td>100.00</td>
</tr>
</tbody>
</table>

---

**RT 1.449**

---

**DAD1 A, Sig=215,10 Ref=off (D:\WORK\D\09\09 22\L529554D\SAMPL010.D)**

---

**DAD1 B, Sig=254,10 Ref=off (D:\WORK\D\09\09 22\L529554D\SAMPL010.D)**

---

**MSD1 TIC, MS File (D:\WORK\D\09\09 22\L529554D\SAMPL010.D) API-ES, Scan, Frag: 120, "Pos"**

---

**MSD2 TIC, MS File (D:\WORK\D\09\09 22\L529554D\SAMPL010.D), Scan, Frag: 120, "Neg"**

---

**ADCT1 A, ADCT1 ELSD (D:\WORK\D\09\09 22\L529554D\SAMPL010.D)**

---

**MSD1 SPC, time=1.449 of D:\WORK\D\09\09 22\L529554D\SAMPL010.D API-ES, Scan, Frag: 120, "Pos"**

---

*Inj.Date 9/23/2022         K     P2-B-02  -SL-  Acq. Method C:\HPCHEM\*  ->

---

**IOH58211**

---

2b
File name: phts50.fid  
Date: 20-Sep-2022  
Operator:  
Solvent: cdcl3  
SF: 399.9714 MHz  
NSC: 0  
PW: 10.90 usec, RG: 24  
SW: 8000 Hz  
TE: 298 K  
AQ: 2.00 sec, RD: 0.00 sec  
SI: 32768
PHts-50-C13.fid

File name: PHts-50-C13.fid
Date: 21-Sep-2022
Operator:  
SR: 125.6921 MHz
NSC: 0
PW: 3.33 usec, RG: 60
AQ: 1.05 sec, RD: 0.00 sec
SI: 65536

Solvent: cdcl3
SW: 31250 Hz
TE: 298 K
Library Search Report

Data Path: D:\MassHunter\GCMS\1\data\09_09\nData File: IOH58204.D
Acq On: 09 Sep 2022 16:34
Operator:
Sample: IOH58204
Misc: CH3OH
ALS Vial: 131  Sample Multiplier: 1

Search Libraries: C:\Database\EMPTY.L  Minimum Quality: 0

Unknown Spectrum: Apex
Integration Events: ChemStation Integrator - autoint1.e

Abundance TIC: IOH58204.D\data.ms
Unknown Spectrum based on Apex

Scan 426 (4.302 min): IOH58204.D\data.ms

Data File: D:\MassHunter\GCMS1\data\09_09\IOH58204.D
Sample : IOH58204

Peak Number: 4 at 4.305 min Area: 49629932 Area % 94.62

The 3 best hits from each library.

Ref\# CAS\# Qual

C:\Database\EMPTY.L No hits were retrieved.
Library Search Report - ChemStation Integrator

Unknown Spectrum based on Apex

Scan 456 (4.479 min): IOH58204.D\data.ms

Abundance

Data File: D:\MassHunter\GCMS\1\data\09_09\IOH58204.D
Sample : IOH58204

Peak Number: 5 at 4.477 min Area: 702778 Area % 1.34

The 3 best hits from each library.

C:\Database\EMPTY.L No hits were retrieved.

Ref\# CAS\# Qual

m/z 197.90 100.00%
m/z 195.00 71.27%
m/z 117.00 37.80%
m/z 73.00 26.47%
m/z 199.80 25.76%
m/z 117.00 20.80%
m/z 98.90 15.08%
m/z 206.90 10.57%
m/z 162.90 9.95%
m/z 150.89 9.11%
m/z 100.89 9.11%
m/z 37.00 9.11%

m/z 197.90 100.00%
m/z 195.00 71.27%
m/z 117.00 37.80%
m/z 73.00 26.47%
m/z 199.80 25.76%
m/z 117.00 20.80%
m/z 98.90 15.08%
m/z 206.90 10.57%
m/z 162.90 9.95%
m/z 150.89 9.11%
m/z 100.89 9.11%
m/z 37.00 9.11%
Unknown Spectrum based on Apex

Scan 605 (5.359 min): IOH58204.D

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
</table>

No hits were retrieved.
Date: 31-Aug-2022 | Solvent: cdcl3 | SW: 8000 Hz | TE: 298 K | AQ: 2.00 sec, RD: 0.00 sec
Library Search Report

Data File : C:\HPCHEM\1\DATA\09_15\IOH58205.D
Acq On : 16 Sep 2022 1:15
Sample : IOH58205
Operator:
Inst : GC/MS Ins
Misc : CH3OH
Multiplier : 1.00
Sample Amount : 0.00
MS Integration Params: autoint1.e
Method : C:\HPCHEM\1\METHODS\UNIVERS.M (Chemstation Integrator)
Title :

![Graph showing abundance over time with peaks at 6.48, 7.12, and 7.68.]
Unknown Spectrum based on Apex

Peak Number: 1 at 6.48 min Area: 81760438 Area % 97.65

The 3 best hits from each library. Ref# CAS# Qual

C:\DATABASE\EMPTY.L No hits were retrieved.
Unknown Spectrum based on Apex

Peak Number: 2 at 7.13 min Area: 1799786 Area % 2.15

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

C:\DATABASE\EMPTY.L No hits were retrieved.
File name: PHTS-77.fid
Operator: 
Date: 31-Aug-2022
Solvent: cdcl3
SF: 399.9714 MHz
SW: 8000 Hz
NSC: 0
TE: 298 K
PW: 10.90 usec, RG: 20
AQ: 2.00 sec, RD: 0.00 sec
SI: 32768
Date: 02-Sep-2022  Solvent: cdcl3  SW: 37879 Hz  TE: 298 K  AQ: 0.87 sec, RD: 0.00 sec
File name: phts77_C13APT.fid
Date: 02-Sep-2022
Operator:
Solvent: cdcl3
SF: 150.8296 MHz
NSC: 0
PW: 4.63 usec, RG: 60
AQ: 1.27 sec, RD: 0.00 sec
SI: 131072
Data Path: C:\msdchem\1\data\09_08\nData File: IOH55256.D
Acq On: 8 Sep 2022 12:51
Operator:
Sample: IOH55256
Misc: CH3OH
ALS Vial: 2 Sample Multiplier: 1
Search Libraries: C:\\Database\\EMPTY.L
Minimum Quality: 0
Unknown Spectrum: Apex
Integration Events: ChemStation Integrator - autoint1.e

S27

Abundance

Time--->

GCMS-3.M Thu Sep 08 13:09:31 2022 1
Data File: C:\msdchem\1\data\data.ms
Sample : IOH55256
Peak Number: 1 at 9.208 min Area: 22608447 Area % 100.00

The 3 best hits from each library.

C:\Database\EMPTY.L No hits were retrieved.
File name: PHts-56.fid
Operator:
SF: 399.9714 MHz
NSC: 0
PW: 10.90 usec, RG: 24
SI: 32768
Date: 20-Jun-2022
Solvent: cdcl3
SW: 8000 Hz
TE: 298 K
AQ: 2.00 sec, RD: 0.00 sec
File name: PHTS-56_C13  
Operator: root  
SF: 125.6681 MHz  
NSC: 267  
PW: 0.00 usec, RG: 51200  
SI: 131072  

Date: 26-Jun-2022  
Solvent: CDCl3  
SW: 32680 Hz  
TE: 683 K  
AQ: 1.00 sec, RD: 0.00 sec
PHTS-56_C13APT

Cl
Br
CHO

3c

S31

File name: PHTS-56_C13APT
Operator: root
SF: 125.6681 MHz
NSC: 161
PW: 0.00 usec, RG: 51200
SI: 65536

Date: 26-Jun-2022
Solvent: CDCl3
SW: 32680 Hz
TE: 683 K
AQ: 1.00 sec, RD: 0.00 sec
Library Search Report

Data File : C:\HPCHEM\1\DATA\06_21\IOH55217.D
Acq On : 21 Jun 2022 16:57
Sample : IOH55217
Misc : CH3OH
MS Integration Params: autoint1.e
Method : C:\HPCHEM\1\METHODS\UNIVERS.M (Chemstation Integrator)
Title :
Unknown Spectrum based on Apex

Peak Number: 1 at 5.52 min Area: 43181334 Area % 97.11

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

C:\DATABASE\EMPTY.L No hits were retrieved.
Unknown Spectrum based on Apex

Peak Number: 2 at 5.79 min Area: 377658 Area % 0.85

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

C:\DATABASE\EMPTY.L No hits were retrieved.
Unknown Spectrum based on Apex

Peak Number: 4 at 6.24 min Area: 424379 Area % 0.95

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td>C:\DATABASE\EMPTY.L</td>
<td>No hits were retrieved.</td>
<td></td>
</tr>
</tbody>
</table>
File name: PHTS-64_C13APT.fid
Operator:               SF: 125.6919 MHz    NSC: 0    PW: 5.00 usec, RG: 60    SI: 131072
Date: 25-Jul-2022    Solvent: cdcl3     SW: 32895 Hz    TE: 298 K    AQ: 1.95 sec, RD: 0.00 sec    Automated Probe tuning parameter
Unknown Spectrum based on Apex

Data File: C:\msdchem\1\data\07_22\IOH55231.D\data.ms
Sample : IOH55231
Peak Number:  1     at      6.560 min  Area:  41194620  Area % 100.00

The 3 best hits from each library.          Ref\#    CAS\#      Qual
---------------------------------------------------------------------
C:\Database\EMPTY.L     No hits were retrieved.
PHTS-73.fid

File name: PHTS-73.fid
Date: 02-Sep-2022
Operator: Solvent: dmso
SF: 399.9733 MHz SW: 8000 Hz
NSC: 0 TE: 298 K
PW: 10.90 usec, RG: 24 AQ: 2.00 sec, RD: 0.00 sec
SI: 32768

4a

Cl

I

I

I

S41
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Date: 02-Sep-2022</td>
<td>Solvent: dmso</td>
<td>SW: 37879 Hz</td>
<td>TE: 298 K</td>
<td>AQ: 0.87 sec, RD: 0.00 sec</td>
<td></td>
</tr>
</tbody>
</table>
File name: PHTS-73_C13APT.fid
Date: 02-Sep-2022
Operator:  
SF: 150.8303 MHz  
NSC: 0  
PW: 4.63 usec, RG: 60  
SI: 131072
Solvent: dmso  
SW: 37879 Hz  
TE: 298 K  
AQ: 1.27 sec, RD: 0.00 sec
Data Path: D:\MassHunter\GCMS\1\data\09_05\nData File: IOH55251.D
Acq On: 05 Sep 2022 21:25
Operator:
Sample: IOH55251
Misc: CH3OH
ALS Vial: 34 Sample Multiplier: 1

Search Libraries: C:\Database\EMPTY.L Minimum Quality: 0

Unknown Spectrum: Apex
Integration Events: ChemStation Integrator - autoint1.e

Abundance

TIC: IOH55251.Didata.ms
8.156

GCMS-6.M Tue Sep 06 09:45:52 2022
Unknown Spectrum based on Apex

Scan 1107 (8.156 min): IOH55251.D\data.ms

Data File: D:\MassHunter\GCMS\1\data\09_05\IOH55251.D
Sample : IOH55251

Peak Number: 1 at 8.156 min Area: 47252677 Area % 100.00

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

C:\Database\EMPTY.L No hits were retrieved.
PHts74_C13.fid

![Chemical Structure](image)

**File name:** PHts74_C13.fid  **Operator:**

**SF:** 150.8304 MHz  **NSC:** 0  **PW:** 3.09 usec, **RG:** 60

**Date:** 15-Sep-2022  **Solvent:** dmso  **SW:** 37879 Hz  **TE:** 298 K  **AQ:** 1.69 sec, **RD:** 0.00 sec

**SI:** 131072
File name: PHts74_APTC13.fid
Date: 15-Sep-2022
Operator: 
Solvent: cdcl3
SF: 150.8288 MHz
SW: 39063 Hz
NSC: 0
TE: 298 K
PW: 4.63 usec, RG: 60
AQ: 1.23 sec, RD: 0.00 sec
SI: 131072
Data Path: D:\MassHunter\GCMS\1\data\08_19\nData File: IOH55244.D
Acq On: 19 Aug 2022 16:30
Operator:
Sample: IOH55244
Misc: CH3OH
ALS Vial: 41 Sample Multiplier: 1
Search Libraries: C:\Database\EMPTY.L
Minimum Quality: 0
Unknown Spectrum: Apex
Integration Events: ChemStation Integrator - autoint1.e

Abundance

TIC: IOH55244.D\data.ms

8.800
Unknown Spectrum based on Apex

Abundance

Scan 7 (1.662 min): IOH55244.D\data\ms

Data File: D:\MassHunter\GCMS\1\data\08_19\IOH55244.D
Sample: IOH55244

Peak Number: 1 at 1.660 min Area: 220042 Area % 1.57

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td>C:\Database\EMPTY.L</td>
<td>No hits were retrieved.</td>
<td></td>
</tr>
</tbody>
</table>
Unknown Spectrum based on Apex

Abundance

Scan 1216 (8.799 min): IOH55244.D\data.ms

Data File: D:\MassHunter\GCMS\data\08_19\IOH55244.D
Sample : IOH55244
Peak Number: 4 at 8.800 min Area: 13723802 Area % 97.94

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

C:\Database\EMPTY.L No hits were retrieved.
Library Search Report

Data Path: D:\MassHunter\GCMS\1\data\07_25\nData File: IOH55234.D
Acq On: 26 Jul 2022 00:22
Operator:
Sample: IOH55234
Misc: CH3OH
ALS Vial: 11 Sample Multiplier: 1

Search Libraries: C:\Database\EMPTY.L Minimum Quality: 0

Unknown Spectrum: Apex
Integration Events: ChemStation Integrator - autoint1.e

Abundance TIC: IOH55234.D\data.ms

GCMS - 5.M Tue Jul 26 12:19:36 2022 Page: 1
Unknown Spectrum based on Apex

Scan 978 (7.561 min): IOH55234.D\data.ms

Data File: D:\MassHunter\GCMS\data\07_25\IOH55234.D
Sample: IOH55234

Peak Number: 2 at 7.562 min Area: 53092891 Area % 95.36

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td>C:\Database\EMPTY.L</td>
<td>No hits were retrieved.</td>
<td></td>
</tr>
</tbody>
</table>
Unknown Spectrum based on Apex

Abundance

Scan 986 (7.608 min): IOH55234.D;data.ms

Data File: D:\MassHunter\GCMS\1\data\07_25\IOH55234.D
Sample : IOH55234

Peak Number: 3 at 7.609 min Area: 1056266 Area % 1.90

The 3 best hits from each library.

Ref\# CAS\# Qual

C:\Database\EMPTY.L No hits were retrieved.
Unknown Spectrum based on Apex

Scan 1100 (8.281 min): IOH55234.D\data.ms

Data File: D:\MassHunter\GCMS\1\data\07_25\IOH55234.D
Sample : IOH55234

Peak Number: 4 at 8.280 min Area: 1213038 Area % 2.18

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td>C:\Database\EMPTY.L</td>
<td>No hits were retrieved.</td>
<td></td>
</tr>
<tr>
<td>--------------------------</td>
<td>-----------</td>
<td>------------------</td>
</tr>
<tr>
<td>Date: 17-Jun-2022</td>
<td>Solvent: cdcl3</td>
<td>SW: 8000 Hz</td>
</tr>
</tbody>
</table>

PHTS-53-2.fid

![Chemical structure](image)

**4d**
PHTS-53-1_C13

File name: PHTS-53-1_C13
Operator: root
Date: 18-Jun-2022
Solvent: CDCl3
SF: 125.6681 MHz
SW: 32680 Hz
NSC: 1221
TE: 683 K
PW: 0.00 usec, RG: 51200
AQ: 0.78 sec, RD: 0.00 sec
SI: 131072
Library Search Report

Data File : C:\HPCHEM\1\DATA\06_20\IOH55833.D
Acq On : 21 Jun 2022 00:49
Sample : IOH55833
Inst : GC/MS Ins
Misc : CH3OH
Multiplr: 1.00
Sample Amount: 0.00

MS Integration Params: autoint1.e
Method : C:\HPCHEM\1\METHODS\UNIVERS.M (Chemstation Integrator)
Title :

![Graph](image-url)

0 200000 400000 600000 800000 1000000 1200000 1400000 1600000 1800000 2000000 2200000 2400000 2600000 2800000 3000000

0 1.50 2.00 2.50 3.00 3.50 4.00 4.50 5.00 5.50 6.00 6.50 7.00 7.50 8.00 8.50 9.00 9.50 10.00 10.50 11.00 11.50 12.00 12.50 13.00 13.50 14.00 14.50 15.00 15.50 16.00

TIC: IOH55833.D

Time-->
Unknown Spectrum based on Apex

Peak Number: 1 at 6.23 min Area: 52938401 Area % 98.49

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td>C:\DATABASE\EMPTY.L</td>
<td>No hits were retrieved.</td>
<td></td>
</tr>
</tbody>
</table>
Unknown Spectrum based on Apex

Peak Number: 4 at 7.72 min Area: 90659 Area % 0.17

The 3 best hits from each library. Ref# CAS# Qual

C:\DATABASE\EMPTY.L No hits were retrieved.
Unknown Spectrum based on Apex

Scan 733 (8.744 min): IOH55833.D

Peak Number: 5 at 8.74 min Area: 199965 Area % 0.37

The 3 best hits from each library. Ref# CAS# Qual

C:\DATABASE\EMPTY.L No hits were retrieved.
PHTS-60_C13

Cl

\[
\text{COOH}
\]

5a

File name: PHTS-60_C13
Operator: root
Date: 27-Jul-2022
SF: 125.6681 MHz
Solvent: DMSO
NSC: 675
PW: 0.00 usec, RG: 51200
TE: 683 K
AQ: 0.78 sec, RD: 0.00 sec
SI: 131072
File name: PHTS-60_C13APT

Operator: root
SF: 125.6681 MHz
NSC: 245
PW: 0.00 usec, RG: 51200
SI: 65536

Date: 27-Jul-2022
Solvent: DMSO
SW: 32680 Hz
TE: 683 K
AQ: 1.57 sec, RD: 0.00 sec
MaxPeak: 96.74%  
Ret_Time: 1.261 min

5a

Mol Wt
Exact Mass

<table>
<thead>
<tr>
<th>#</th>
<th>Time (min)</th>
<th>Area%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.825</td>
<td>1.04</td>
</tr>
<tr>
<td>2</td>
<td>1.261</td>
<td>96.74</td>
</tr>
<tr>
<td>3</td>
<td>1.297</td>
<td>2.22</td>
</tr>
</tbody>
</table>
phts59_C13.fid

Date: 22-Jul-2022 | Solvent: dmso | SW: 37879 Hz | TE: 298 K | AQ: 1.27 sec, RD: 0.00 sec
File name: phts59_C13APT.fid
Date: 22-Jul-2022
Operator: 
Solvent: dmso
SF: 150.8303 MHz
SW: 37879 Hz
NSC: 0
TE: 298 K
PW: 4.63 usec, RG: 60
AQ: 1.27 sec, RD: 0.00 sec
SI: 131072
File name: PHTS-61.fid
Date: 20-Jul-2022
Operator: Solvent: dmso
SF: 399.9733 MHz SW: 8000 Hz
NSC: 0 TE: 298 K
PW: 10.90 usec, RG: 24 AQ: 2.00 sec, RD: 0.00 sec
SI: 32768
**IOH55229**

MaxPeak: 100.00%
Ret_Time: 1.257 min

**Mol Wt** 0
**Exact Mass**

<table>
<thead>
<tr>
<th>#</th>
<th>Time</th>
<th>Area%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.257</td>
<td>100.00</td>
</tr>
</tbody>
</table>

**MSD1 SPC, time=1.321 of D:\DATA\0722\L508772D\SAMPL00025.D  ES-API, Scan, Frag: 100, *POS***
130.2 159.0 101.0
157.0

**MSD2 SPC, time=1.275 of D:\DATA\0722\L508772D\SAMPL00025.D  ES-API, Scan, Frag: 100, *NEG***
504.6 198.8 195.0

**Inj.Date 7/22/2022**
PHTS-63.fid

HBr\(-\text{COOH}

Br

Cl

5d

File name: PHTS-63.fid
Date: 20-Jul-2022
Operator:
Solvent: dmso
SF: 399.9733 MHz
NSC: 0
PW: 10.90 usec, RG: 24
SI: 32768
SW: 8000 Hz
TE: 298 K
AQ: 2.00 sec, RD: 0.00 sec
File name: phts63_C13.fid
Date: 22-Jul-2022
Operator: 
SF: 150.8304 MHz  
NSC: 0  
PW: 3.09 usec, RG: 60  
Si: 131072
Solvent: dmso  
SW: 37879 Hz  
TE: 298 K  
AQ: 1.27 sec, RD: 0.00 sec

Br
\[\text{COOH}\]

5d
Br\[\begin{array}{c}
\text{COOH} \\
\text{5d} \\
\text{Cl}
\end{array}\]
Max Peak: 97.15%
Ret Time: 1.168 min

Max Peak: 97.15%
Ret Time: 1.168 min

**Mol Wt**
**Exact Mass**

<table>
<thead>
<tr>
<th>#</th>
<th>Time</th>
<th>Area%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.155</td>
<td>2.85</td>
</tr>
<tr>
<td>2</td>
<td>1.168</td>
<td>97.15</td>
</tr>
</tbody>
</table>

**RT 1.182**

```
100  200  300  400  500
89.2 131.2 159.2 157.2 0
```

**RT 1.183**

```
100  200  300  400  500
195.0 199.0 238.8 242.8 0
```
PHTS-53.fid

Cl

\begin{align*}
\text{CH}_2
\end{align*}

I

6a

File name: PHTS-53.fid
Operator: 
SF: 399.9714 MHz
NSC: 0
PW: 10.90 usec, RG: 24
SI: 32768
Date: 17-Jun-2022
Solvent: cdcl3
SW: 8000 Hz
TE: 298 K
AQ: 2.00 sec, RD: 0.00 sec
File name: PHTS-53_C13
Operator: nmr
SF: 100.6128 MHz
NSC: 421
PW: 0.00 usec, RG: 2050
SI: 32768
Date: 17-Jun-2022
Solvent: CDCl3
SW: 26042 Hz
TE: 300 K
AQ: 0.98 sec, RD: 0.00 sec

S83
PHTS-53_C13APT

File name: PHTS-53_C13APT
Operator: root
Date: 18-Jun-2022
SF: 125.6681 MHz
Solvent: CDCl3
NSC: 574
SW: 32680 Hz
TE: 683 K
PW: 0.00 usec, RG: 51200
AQ: 1.00 sec, RD: 0.00 sec
SI: 65536
Data Path: D:\MassHunter\GCMS\1\Data\06_20\nData File: IOH55832.D
Acq On: 20 Jun 2022 21:58
Operator:
Sample: IOH55832
Misc: CH3OH
ALS Vial: 85  Sample Multiplier: 1

Search Libraries: C:\Database\EMPTY.L  Minimum Quality: 0

Unknown Spectrum: Apex
Integration Events: ChemStation Integrator - autoint1.e

TIC: IOH55832.D|data.ms
Unknown Spectrum based on Apex

Scan 725 (5.900 min): IOH55832.D\data.ms

Data File: D:\MassHunter\GCMS\data\06_20\IOH55832.D
Sample : IOH55832

Peak Number: 2 at 5.900 min Area: 3960527 Area % 97.60

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td>C:\Database\EMPTY.L</td>
<td>No hits were retrieved.</td>
<td></td>
</tr>
</tbody>
</table>
Unknown Spectrum based on Apex

Scan 760 (6.107 min): IOH55832.D\data.ms

---

Data File: D:\MassHunter\GCMS\1\data\06_20\IOH55832.D
Sample : IOH55832

Peak Number: 3 at 6.107 min Area: 866623 Area % 2.14

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

C:\Database\EMPTY.L No hits were retrieved.
File name: PHTS-52.fid
Operator: 
Date: 16-Jun-2022
Solvent: ccdl3
SF: 399.9714 MHz
SW: 8000 Hz
NSC: 0
TE: 298 K
PW: 10.90 usec, RG: 24
AQ: 2.00 sec, RD: 0.00 sec
SI: 32768
**File name:** PHTS-52_C13APT  
**Operator:** root  
**Date:** 18-Jun-2022  
**Solvent:** CDCl3  
**SF:** 125.6681 MHz  
**NSC:** 566  
**TE:** 683 K  
**PW:** 0.00 usec, **RG:** 51200  
**AQ:** 1.00 sec, **RD:** 0.00 sec  
**SI:** 65536

![Chemical Structure](image_url)

**PPT-52_C13APT**

<table>
<thead>
<tr>
<th>PPM</th>
<th>0</th>
<th>20</th>
<th>40</th>
<th>60</th>
<th>80</th>
<th>100</th>
<th>120</th>
<th>140</th>
<th>160</th>
<th>180</th>
<th>200</th>
<th>220</th>
<th>240</th>
</tr>
</thead>
</table>

**Remarks:**
- The chemical structure shown is that of compound 6b, featuring a bromine (Br) and an iodine (I) substituent.
- The NMR spectrum displays a signal at the 6b position.
<table>
<thead>
<tr>
<th>File name: PHts-52_F19</th>
<th>Operator: nmr</th>
<th>SF: 376.4986 MHz</th>
<th>NSC: 1</th>
<th>PW: 0.00 usec, RG: 2050</th>
<th>SI: 262144</th>
</tr>
</thead>
<tbody>
<tr>
<td>Date: 20-Jun-2022</td>
<td>Solvent: CDCl3</td>
<td>SW: 138889 Hz</td>
<td>TE: 300 K</td>
<td>AQ: 0.94 sec, RD: 0.00 sec</td>
<td>19F</td>
</tr>
</tbody>
</table>
Data Path: C:\msdchem\i\data\02_02\nData File: R2879836.D
Acq On: 2 Feb 2022 18:25
Operator:
Sample: R2879836
Misc: CH3CN
ALS Vial: 112 Sample Multiplier: 1
Search Libraries: C:\Database\EMPTY.L Minimum Quality: 0
Unknown Spectrum: Apex
Integration Events: ChemStation Integrator - autoint1.e

Abundance
3000000
2800000
2600000
2400000
2200000
2000000
1800000
1600000
1400000
1200000
1000000
800000
600000
400000
200000
0

Time→
2.50 3.00 3.50 4.00 4.50 5.00 5.50 6.00 6.50 7.00 7.50 8.00 8.50 9.00 9.50 10.00 10.50 11.00 11.50 12.00 12.50 13.00 13.50 14.00 14.50 15.00 15.50 16.00

TIC: R2879836.D\data.ms

GCMS-3.M Thu Feb 03 10:19:22 2022
**Unknown Spectrum based on Apex**

**Data File:** C:\msdchem\1\data\02_02\R2879836.D

**Sample:** R2879836

**Peak Number:** 3 at 7.472 min  Area: 38524026  Area % 99.02

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td>C:\Database\EMPTY.L</td>
<td>No hits were retrieved.</td>
<td></td>
</tr>
</tbody>
</table>

Data File: C:\msdchem\1\data\02_02\R2879836.D

Sample : R2879836

**Peak Number:** 3 at 7.472 min  Area: 38524026  Area % 99.02

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>m/z</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>339.9</td>
<td>100.00%</td>
</tr>
<tr>
<td>132.0</td>
<td>21.36%</td>
</tr>
<tr>
<td>212.9</td>
<td>18.55%</td>
</tr>
</tbody>
</table>

**Abundance Scan 904 (7.473 min):** R2879836.D\data.ms
PHTS-66.fid

File name: PHTS-66.fid
Operator: 
SF: 399.9714 MHz
NSC: 0
PW: 10.90 usec, RG: 24
SI: 32768
Date: 29-Jul-2022
Solvent: cdcl3
SW: 8000 Hz
TE: 298 K
AQ: 2.00 sec, RD: 0.00 sec
Library Search Report

Data File: C:\HPCHEM\1\DATA\08_02\H55241_1.D  Vial: 96
Acq On: 2 Aug 2022 16:39  Operator:
Sample: IOH55241  Inst: GC/MS Ins
Misc: CH3OH  Multiplr: 1.00
Sample Amount: 0.00

MS Integration Params: autoint1.e

Method: C:\HPCHEM\1\METHODS\UNIVERS.M (Chemstation Integrator)
Title:

Abundance

Time->

0 1.50
249 2.00
183 2.50
144 3.00
1.37

TIC: H55241_1.D

H55241_1.D UNIVERS.M  Wed Aug 03 10:35:26 2022
Unknown Spectrum based on Apex

Peak Number: 1 at 1.38 min Area: 250644 Area % 0.85

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td>C:\DATABASE\EMPTY.L</td>
<td>No hits were retrieved.</td>
<td></td>
</tr>
</tbody>
</table>
Unknown Spectrum based on Apex

Scan 19 (1.438 min): H55241_1.D

Peak Number: 2 at 1.44 min Area: 238405 Area % 0.81

The 3 best hits from each library.

Ref# CAS# Qual

C:\DATABASE\EMPTY.L No hits were retrieved.
Unknown Spectrum based on Apex

Peak Number: 3 at 1.83 min Area: 28908043 Area % 97.74

The 3 best hits from each library. Ref# CAS# Qual

C:\DATABASE\EMPTY.L No hits were retrieved.
File name: PHTS-68_C13APT.fid
Operator: 
Date: 25-Jul-2022
Solvent: cdcl3
SF: 150.8326 MHz
NSC: 0
PW: 4.63 usec, RG: 60
AQ: 0.87 sec, RD: 0.00 sec
SI: 131072
Library Search Report

Data Path: D:\MassHunter\GCMS\1\data\07_26\nData File: IOH55235.D
Acq On: 26 Jul 2022 16:17
Operator:
Sample: IOH55235
Misc: CH3OH
ALS Vial: 9 Sample Multiplier: 1

Search Libraries: C:\Database\EMPTY.L Minimum Quality: 0
Unknown Spectrum: Apex
Integration Events: ChemStation Integrator - autoint1.e

Abundance

Time--> Abundance TIC: IOH55235.D data.ms

S107

GCMS-5.M Tue Jul 26 17:03:44 2022
Data File: D:\MassHunter\GCMS\data\07_26\IOH55235.D
Sample : IOH55235

Peak Number: 2 at 5.009 min Area: 33221746 Area % 99.60

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

C:\Database\EMPTY.L No hits were retrieved.
PHTS-70.fid

![Diagram of chemical structure](image)

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Date: 15-Sep-2022</td>
<td>Solvent: cdc13</td>
<td>SW: 8000 Hz</td>
<td>TE: 298 K</td>
<td>AQ: 2.00 sec, RD: 0.00 sec</td>
<td></td>
</tr>
</tbody>
</table>
Data Path: C:\msdchem\1\data\09_16\nData File: IOH58206.D
Acq On: 16 Sep 2022 19:40
Operator:
Sample: IOH58206
Misc: CH3OH
ALS Vial: 61 Sample Multiplier: 1

Search Libraries: C:\Database\EMPTY.L
Minimum Quality: 0

Unknown Spectrum: Apex
Integration Events: ChemStation Integrator - autoint1.e

Abundance

Time→

GCMS-3.M Sat Sep 17 09:21:52 2022 1
Page: 1
Unknown Spectrum based on Apex

Data File: C:\msdchem\1\data\09_16\IOH58206.D
Sample : IOH58206
Peak Number: 2 at 8.476 min Area: 5139497 Area % 14.86

The 3 best hits from each library. Ref\# CAS\# Qual
---------------------------------------------------------------------
C:\Database\EMPTY.L No hits were retrieved.
Unknown Spectrum based on Apex

Data File: C:\msdchem\1\data\09_16\IOH58206.D
Sample : IOH58206

Peak Number:  3     at     8.738 min  Area:   27956078  Area % 80.85

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td>C:\Database\EMPTY.L</td>
<td>No hits were retrieved.</td>
<td></td>
</tr>
</tbody>
</table>
Unknown Spectrum based on Apex

Data File: C:\msdchem\1\data\09_16\IOH58206.D
Sample: IOH58206
Peak Number: 4 at 9.221 min Area: 110433  Area % 3.19

The 3 best hits from each library. Ref\#   CAS\#   Qual
---------------------------------------------------------------------
C:\Database\EMPTY.L   No hits were retrieved.
PHTS-71_C13.fid

![Chemical Structure](image)

---

**File name:** PHTS-71_C13.fid  
**Operator:**  
**SF:** 125.6921 MHz  
**NSC:** 0  
**PW:** 3.33 usec, **RG:** 60  
**SI:** 65536  
**Date:** 02-Sep-2022  
**Solvent:** cdcl3  
**SW:** 31250 Hz  
**TE:** 298 K  
**AQ:** 1.05 sec, **RD:** 0.00 sec
File name: PHTS-71_C13APT.fid
Operator: 
SF: 125.6919 MHz 
NSC: 0 
PW: 5.00 usec, RG: 60 
AQ: 1.95 sec, RD: 0.00 sec 
SI: 131072

Date: 02-Sep-2022 
Solvent: cdcl3 
SW: 32895 Hz 
TE: 298 K 

Automated Probe tuning parameter
Library Search Report

Data File : C:\HPCHEM\1\DATA\09_06\IOH55255.D
Acq On : 7 Sep 2022 4:41
Sample : IOH55255
Misc : CH3OH
MS Integration Params: autoint1.e
Method : C:\HPCHEM\1\METHODS\UNIVERS.M (Chemstation Integrator)
Title : S119

![Chemical Structure: 7b]
Unknown Spectrum based on Apex

Peak Number: 4 at 7.15 min Area: 971666 Area % 26.07

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

C:\DATABASE\EMPTY.L No hits were retrieved.
Unknown Spectrum based on Apex

Peak Number: 5 at 7.33 min Area: 25764661 Area % 69.13

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

C:\DATABASE\EMPTY.L No hits were retrieved.
Unknown Spectrum based on Apex

Peak Number: 6 at 7.81 min Area: 584624 Area % 1.57

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

C:\DATABASE\EMPTY.L No hits were retrieved.
Peak Number: 9 at 12.74 min Area: 490561 Area % 1.32

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td>C:\DATABASE\EMPTY.L</td>
<td>No hits were retrieved.</td>
<td></td>
</tr>
</tbody>
</table>
PHTS-69_C13

File name: PHTS-69_C13  Operator: root  SF: 125.6681 MHz  NSC: 500  PW: 0.00 usec, RG: 51200  SI: 131072
Date: 26-Jul-2022  Solvent: CDCl3  SW: 32680 Hz  TE: 683 K  AQ: 0.78 sec, RD: 0.00 sec
File name: PHTS-69_C13APT
Operator: root
Date: 26-Jul-2022
Solvent: CDCl3
SF: 125.6681 MHz
SW: 32680 Hz
NSC: 450
TE: 683 K
PW: 0.00 usec, RG: 51200
AQ: 0.78 sec, RD: 0.00 sec
SI: 65536
Library Search Report

Data Path: D:\MassHunter\GCMS\1\data\07_26\nData File: IOH55236.D
Acq On: 26 Jul 2022 14:37
Operator: 
Sample: IOH55236
Misc: CH3OH
ALS Vial: 6 Sample Multiplier: 1

Search Libraries: C:\Database\EMPTY.L Minimum Quality: 0

Unknown Spectrum: Apex
Integration Events: ChemStation Integrator - autoint1.e

Abundance

<table>
<thead>
<tr>
<th>Time</th>
<th>Abundance</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.375</td>
<td>6.586</td>
</tr>
<tr>
<td>6.798</td>
<td></td>
</tr>
<tr>
<td>7.176</td>
<td>7.393</td>
</tr>
<tr>
<td>12.605</td>
<td></td>
</tr>
</tbody>
</table>

GCMS-5.M Tue Jul 26 15:17:07 2022 Page: 1
Unknown Spectrum based on Apex

Data File: D:\MassHunter\GCMS\data\07_26\IOH55236.D
Sample: IOH55236

Peak Number: 3 at 6.586 min Area: 307957 Area % 1.67

The 3 best hits from each library.

Ref\# CAS\# Qual

C:\Database\EMPTY.L No hits were retrieved.
Unknown Spectrum based on Apex

Scan 849 (6.799 min): IOH55236.D.data.ms

Data File: D:\MassHunter\GCMS\data\07_26\IOH55236.D
Sample: IOH55236

Peak Number: 4 at 6.798 min Area: 17987116 Area % 97.54

The 3 best hits from each library.

| Ref# | CAS#  | Qual
|------|------|------
<p>| C:\Database\EMPTY.L | No hits were retrieved. |
|-----------------------|-----------|-----------------|--------|------------------------|---------|
| Date: 21-Jul-2022     | Solvent: cdc13 | SW: 8000 Hz     | TE: 298 K | AQ: 2.00 sec, RD: 0.00 sec |         |</p>
<table>
<thead>
<tr>
<th>PPM</th>
<th>138.39</th>
<th>129.32</th>
<th>127.69</th>
<th>109.90</th>
<th>77.00</th>
<th>39.78</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solvent: cdcl3</td>
<td>SW: 37879 Hz</td>
<td>TE: 298 K</td>
<td>AQ: 1.27 sec, RD: 0.00 sec</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Diagram:**

![Chemical structure](image)
Data Path: C:\msdchem\1\data\07_22\nData File: IOH55232.D
Acq On: 22 Jul 2022 19:47
Operator:
Sample: IOH55232
Misc: CH3OH
ALS Vial: 86 Sample Multiplier: 1
Search Libraries: C:\Database\EMPTY.L Minimum Quality: 0
Unknown Spectrum: Apex
Integration Events: ChemStation Integrator - autoint1.e

Abundance

Time--> 1.50 2.00 2.50 3.00 3.50 4.00 4.50 5.00 5.50 6.00 6.50 7.00 7.50 8.00 8.50 9.00 9.50 10.00 10.50 11.00 11.50 12.00 12.50 13.00 13.50 14.00 14.50 15.00 15.50 16.00


S133
Unknown Spectrum based on Apex

Data File: C:\msdchem\1\data\07_22\IOH55232.D
Sample: IOH55232
Peak Number: 4 at 6.860 min Area: 888443 Area % 1.17

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

C:\Database\EMPTY.L No hits were retrieved.
Unknown Spectrum based on Apex

Data File: C:\msdchem\1\data\07_22\IOH55232.D
Sample : IOH55232
Peak Number: 5 at 7.131 min Area: 7371961 Area % 97.48

The 3 best hits from each library.

<table>
<thead>
<tr>
<th>Ref#</th>
<th>CAS#</th>
<th>Qual</th>
</tr>
</thead>
<tbody>
<tr>
<td>C: Database\EMPTY.L</td>
<td>No hits were retrieved.</td>
<td></td>
</tr>
</tbody>
</table>