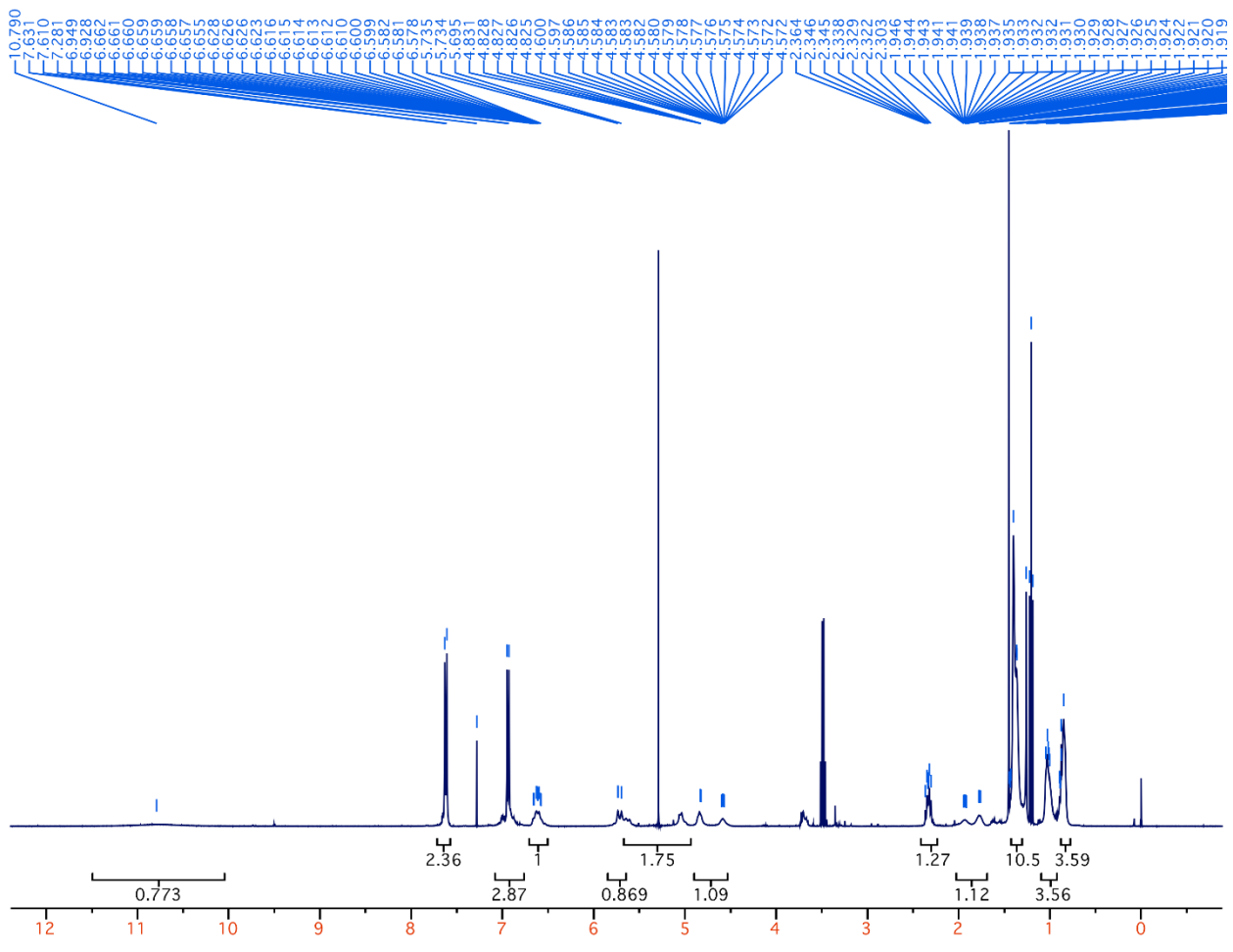
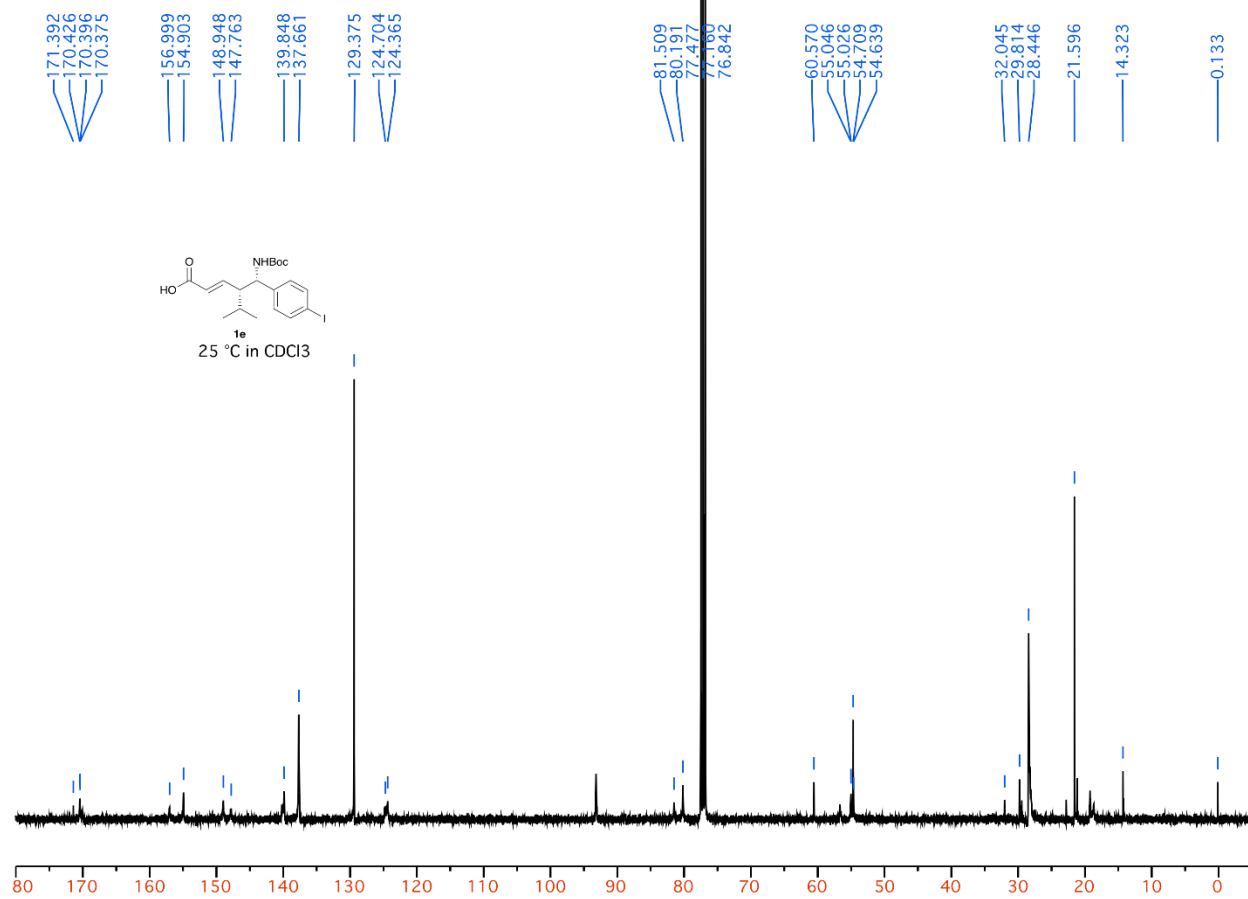
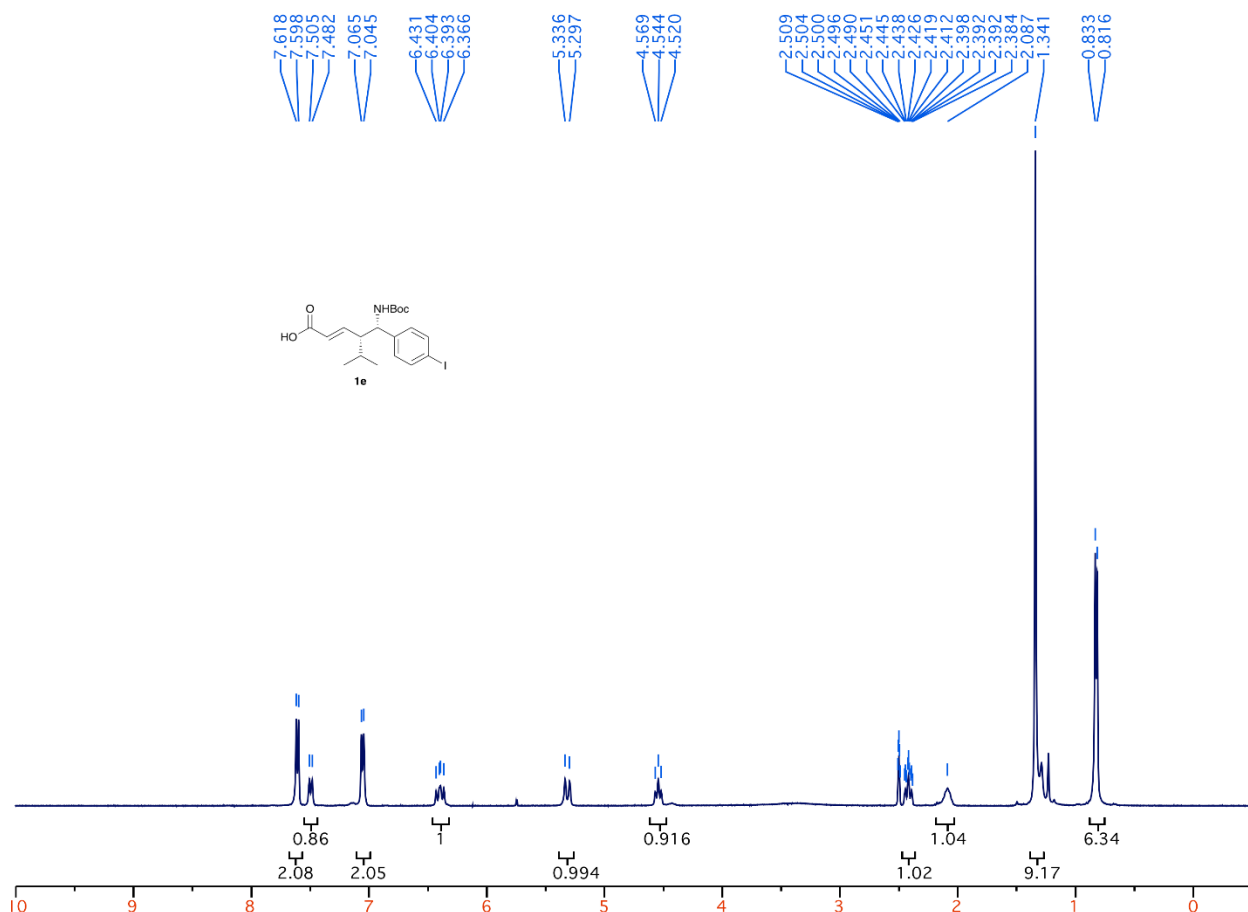


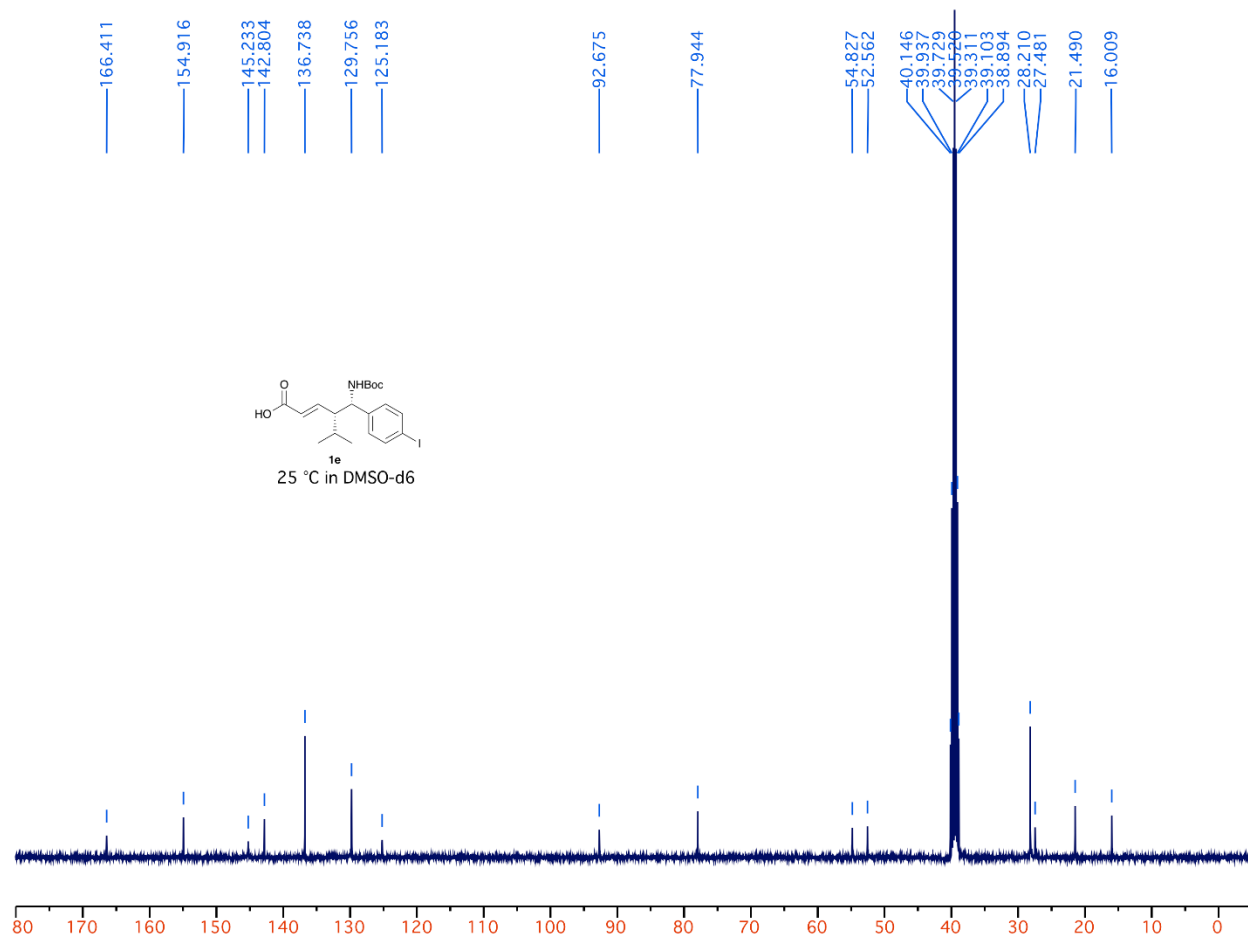
(Crude)

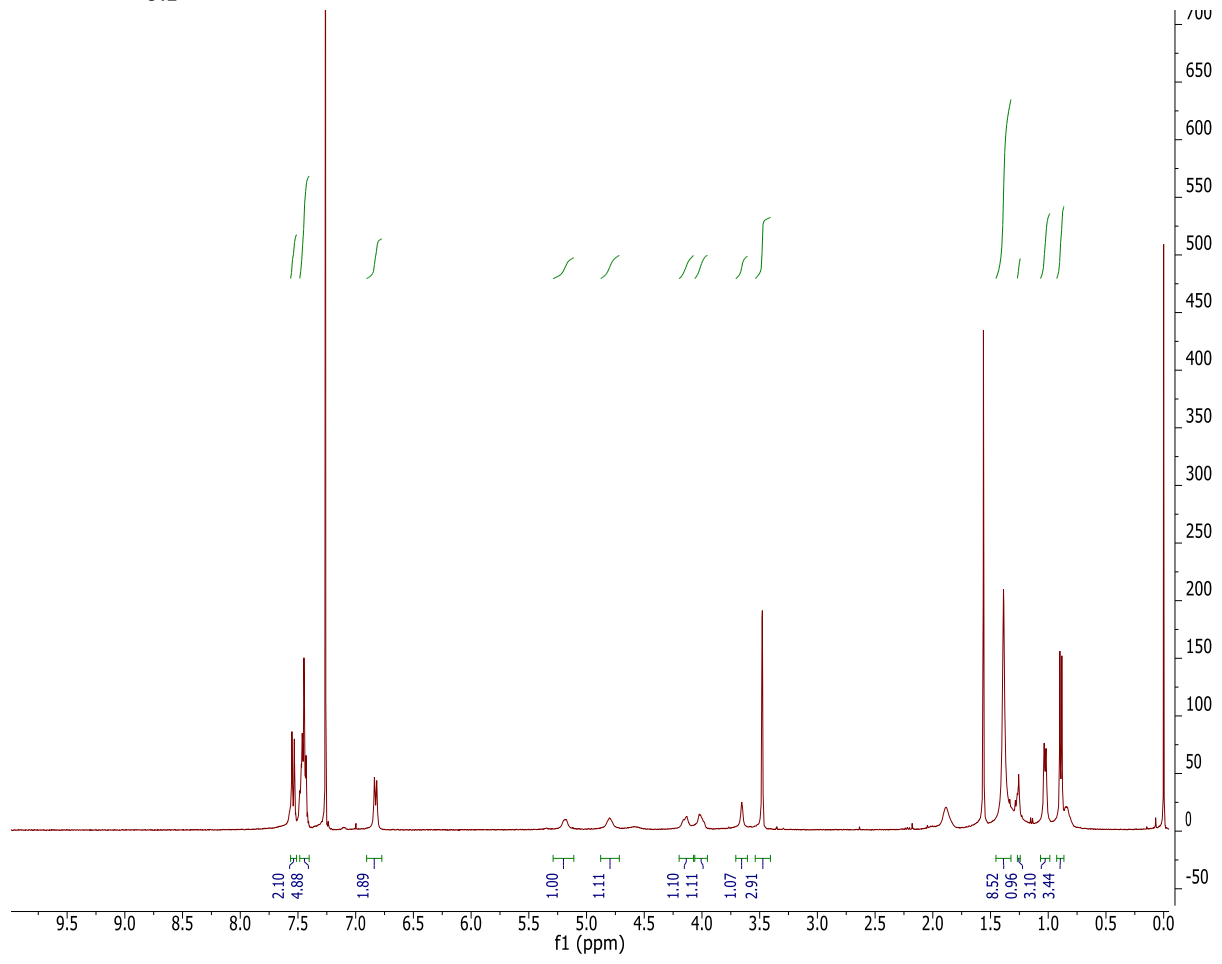
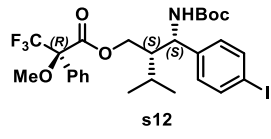


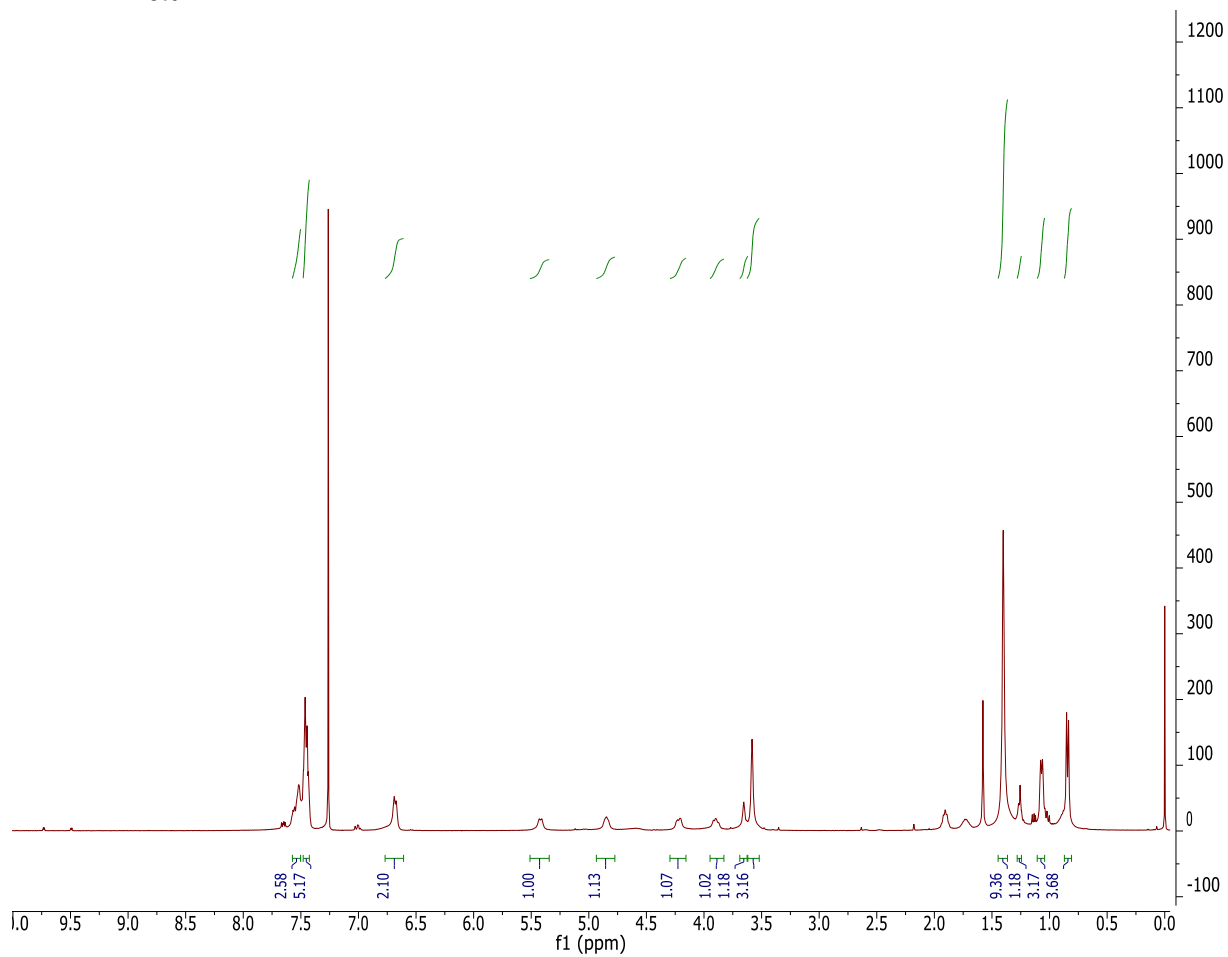
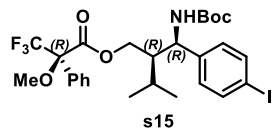


**1e** after purification

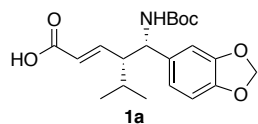








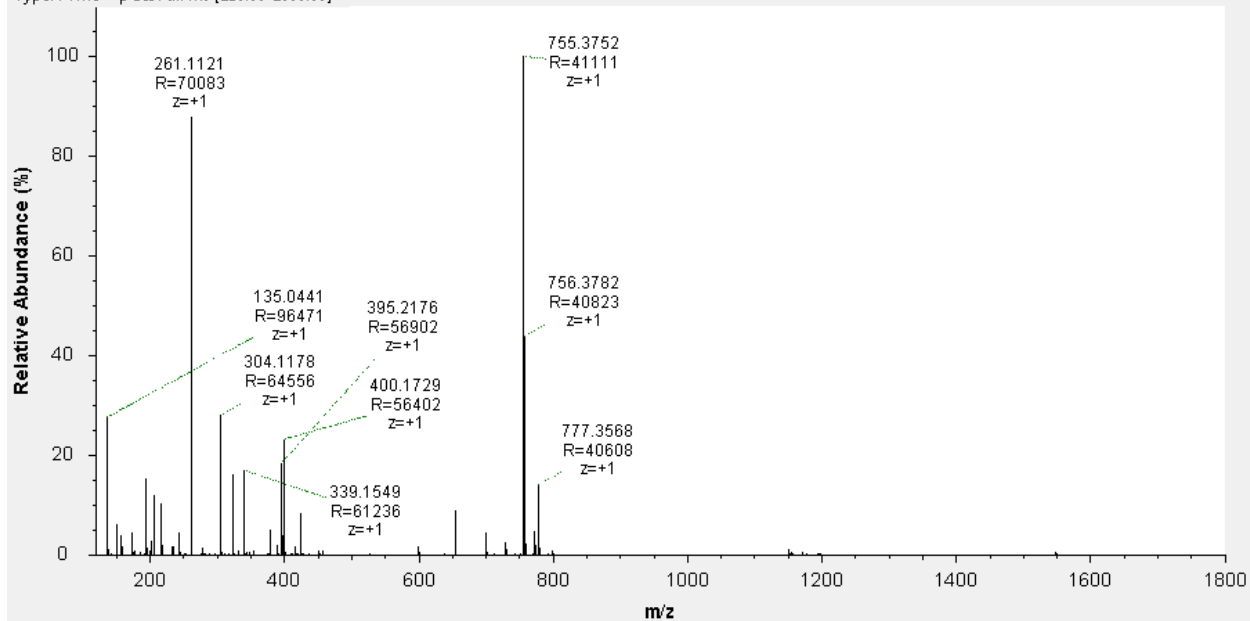
# High resolution mass spectra



Chemical Formula: C<sub>20</sub>H<sub>27</sub>NO<sub>6</sub>  
Exact Mass: 377.1838

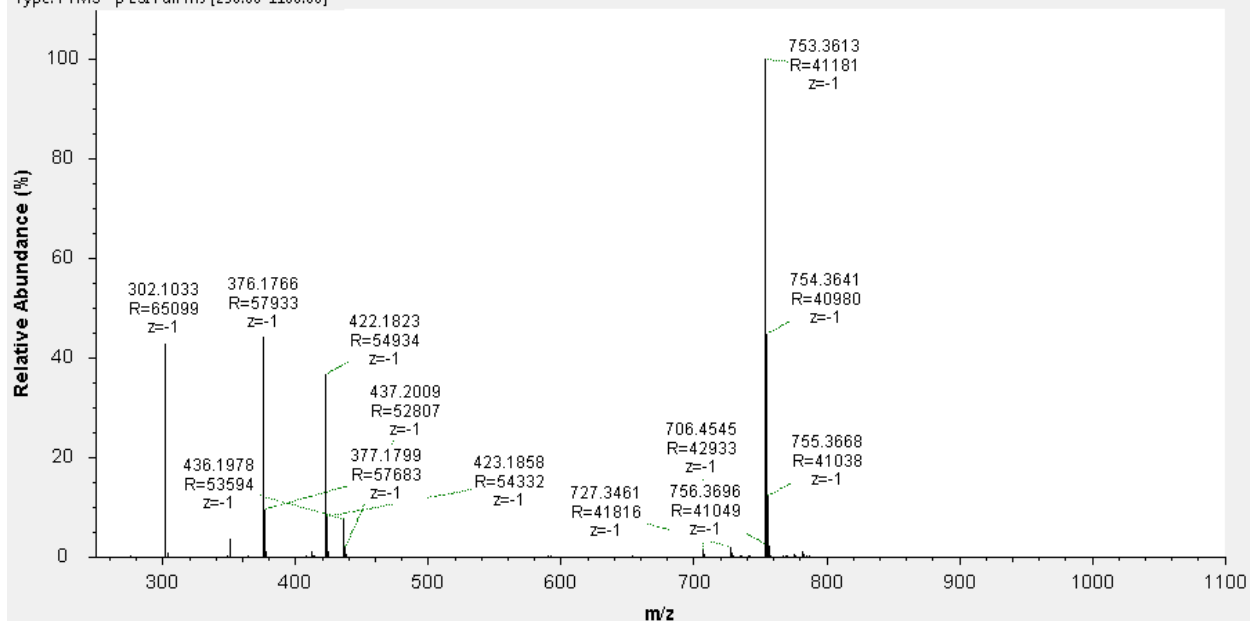
## Positive

Scan: #34035  $\mu$ S: 1 IT: 0.62 NL: 5.84E8  
Type: FTMS + p ESI Full ms [120.00-1800.00]

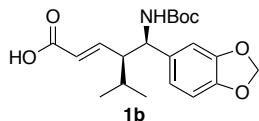


## Negative

Scan: #33488  $\mu$ S: 1 IT: 1.88 NL: 2.52E8  
Type: FTMS - p ESI Full ms [250.00-1100.00]



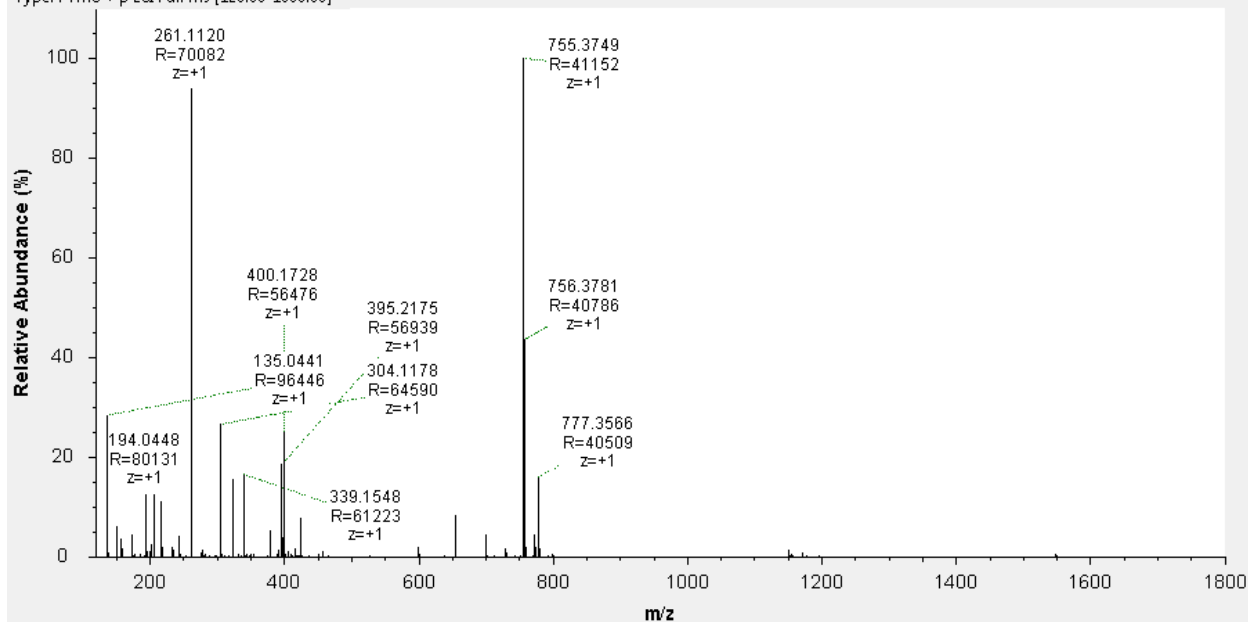
HRMS (ESI+) m/z: [2M+H]<sup>+</sup> Calcd for 755.3749; Found 755.3752. (ESI-) m/z:[M-H]<sup>-</sup> Calcd for 376.1765; Found 376.1766. [2M-H]<sup>-</sup> Calcd for 753.3603; Found 753.3613.



Chemical Formula:  $C_{20}H_{27}NO_6$   
 Exact Mass: 377.1838

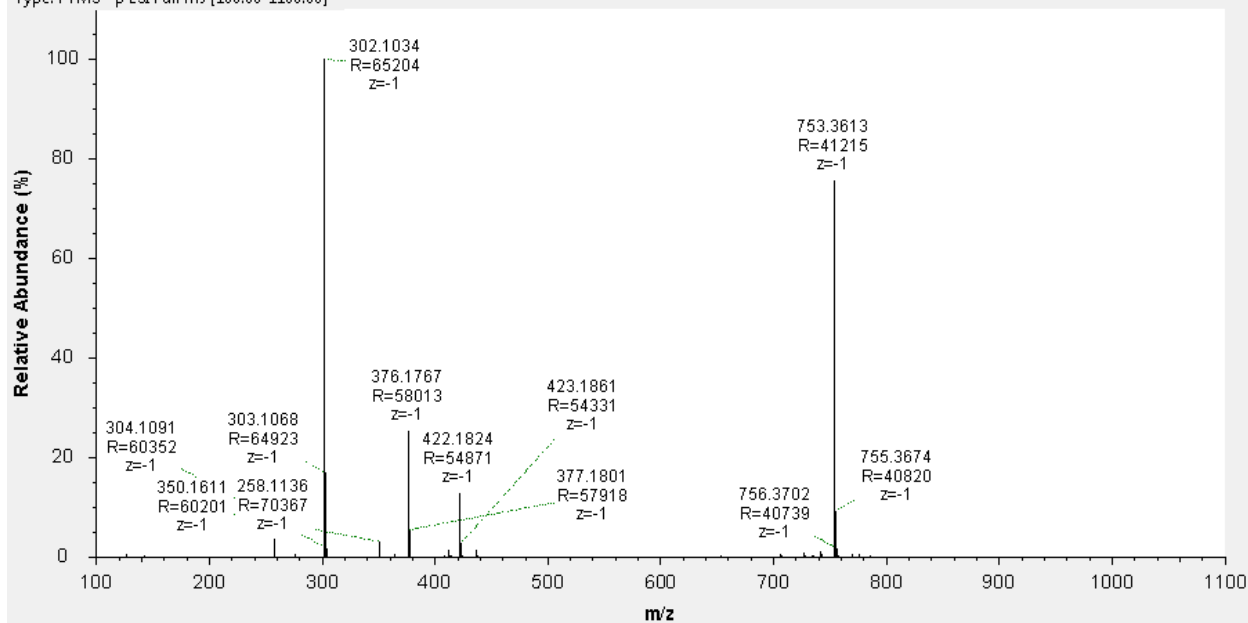
## Positive

Scan: #34256  $\mu$ S: 1 IT: 0.58 NL: 7.05E8  
 Type: FTMS + p ESI Full ms [120.00-1800.00]

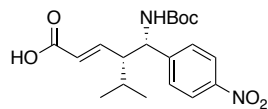


## Negative

Scan: #34617  $\mu$ S: 1 IT: 0.24 NL: 1.85E9  
 Type: FTMS - p ESI Full ms [100.00-1100.00]



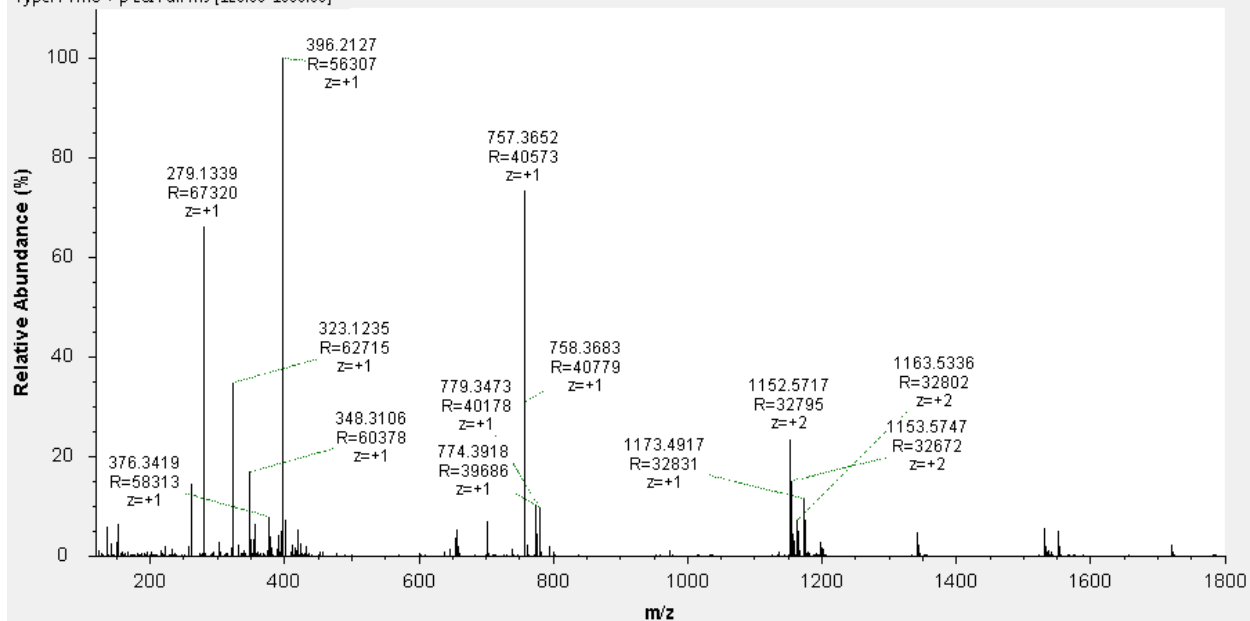
HRMS (ESI+) m/z:  $[2M+H]^+$  Calcd for 755.3749; Found 755.3749. (ESI-) m/z:  $[M-H]^-$  Calcd for 376.1765; Found 376.1767.  $[2M-H]^-$  Calcd for 753.3603; Found 753.3613.



**1c**  
 Chemical Formula: C<sub>19</sub>H<sub>26</sub>N<sub>2</sub>O<sub>6</sub>  
 Exact Mass: 378.1791

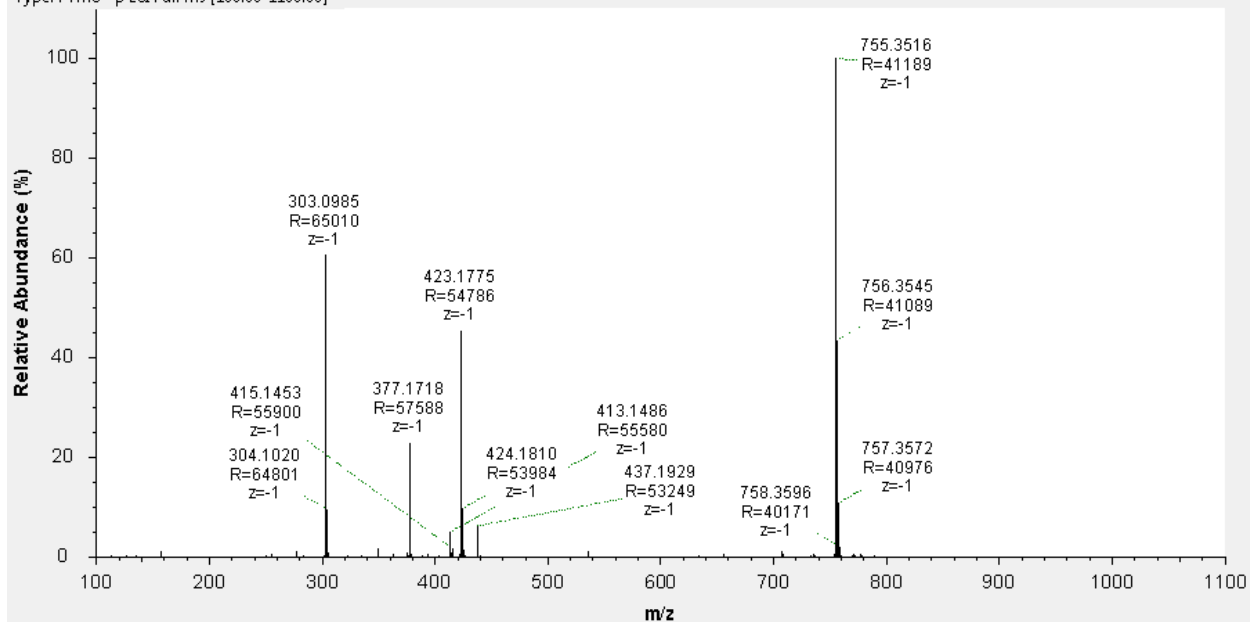
## Positive

Scan: #35168 μS: 1 IT: 7.63 NL: 2.63E7  
 Type: FTMS + p ESI Full ms [120.00-1800.00]

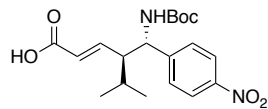


## Negative

Scan: #34885 μS: 1 IT: 1.65 NL: 2.74E8  
 Type: FTMS - p ESI Full ms [100.00-1100.00]



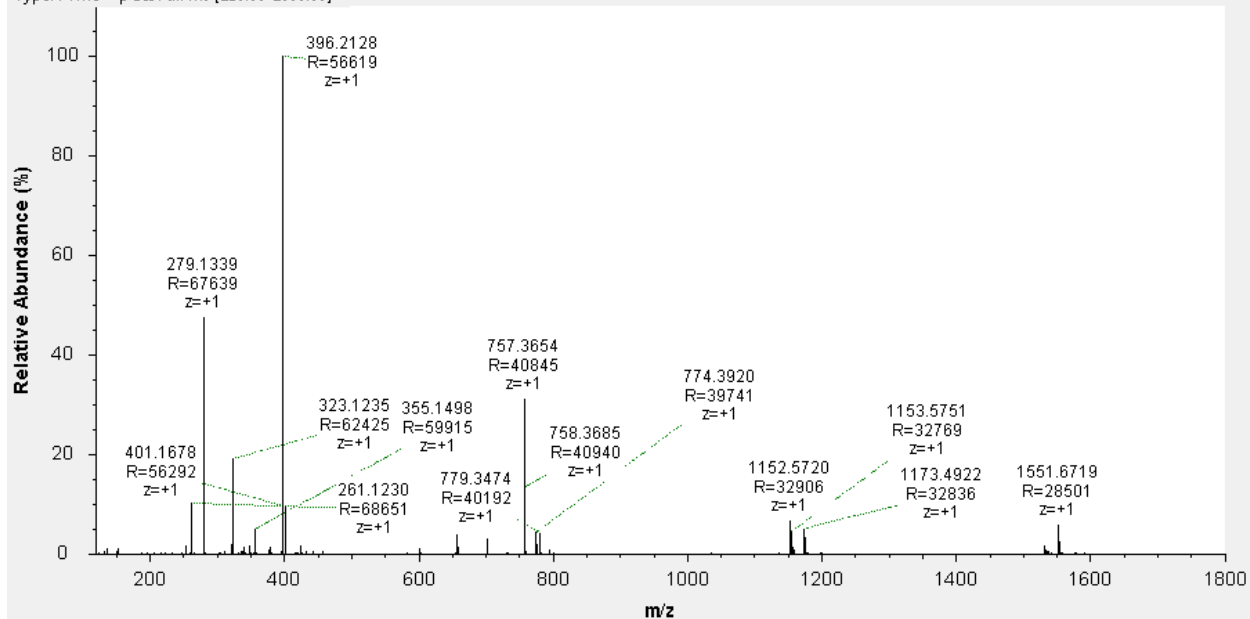
HRMS (ESI+) m/z: [2M+H]<sup>+</sup> Calcd for 757.3655; Found 757.3652. (ESI-) m/z:[M-H]<sup>-</sup>  
 Calcd for 377.1718; Found 376.1718. [2M-H]<sup>-</sup> Calcd for 755.3509; Found 755.3516.



**1d**  
 Chemical Formula: C<sub>19</sub>H<sub>26</sub>N<sub>2</sub>O<sub>6</sub>  
 Exact Mass: 378.1791

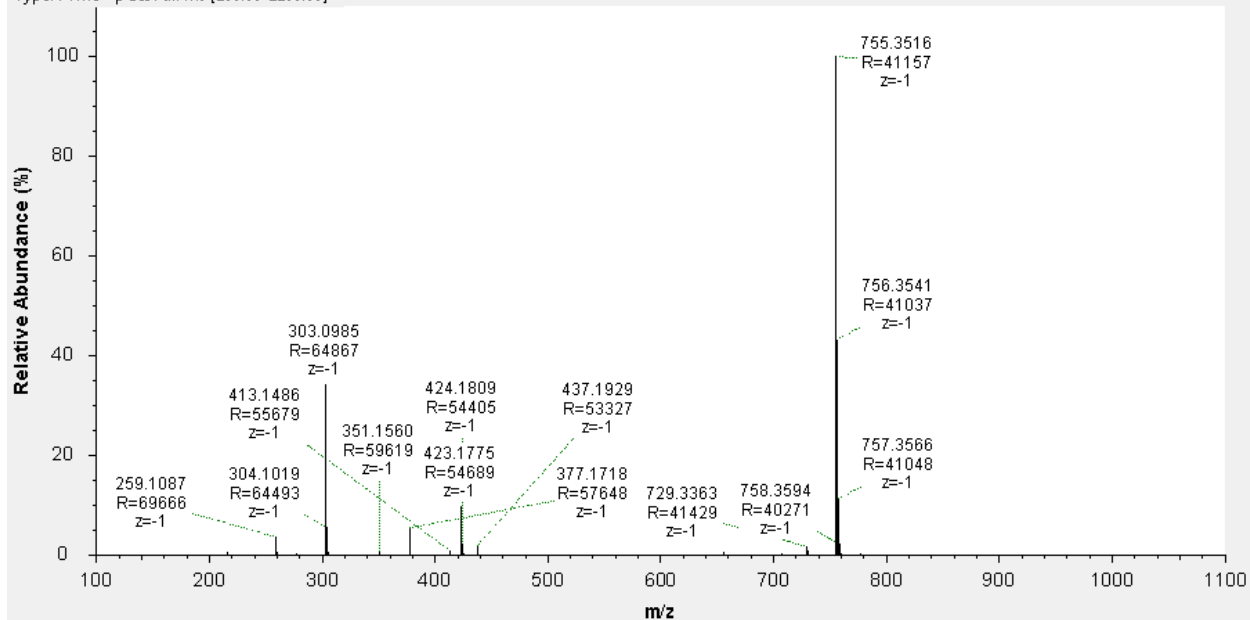
## Positive

Scan: #35520 μS: 1 IT: 4.33 NL: 9.77E7  
 Type: FTMS + p ESI Full ms [120.00-1800.00]



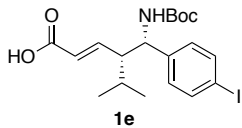
## Negative

Scan: #35713 μS: 1 IT: 0.16 NL: 5.43E9  
 Type: FTMS - p ESI Full ms [100.00-1100.00]



HRMS (ESI+) m/z: [2M+H]<sup>+</sup> Calcd for 757.3655; Found 757.3654. (ESI-) m/z:[M-H]<sup>-</sup> Calcd for 377.1718; Found 376.1718. [2M-H]<sup>-</sup> Calcd for 755.3509; Found 755.3516.

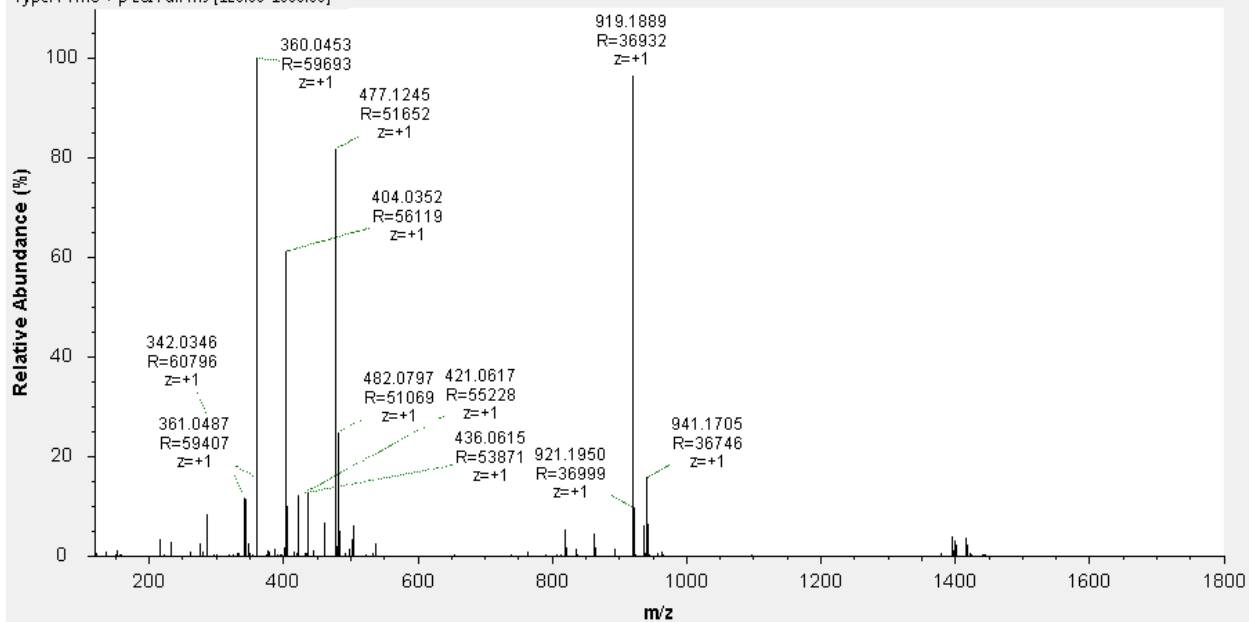




**1e**  
 Chemical Formula: C<sub>19</sub>H<sub>26</sub>INO<sub>4</sub>  
 Exact Mass: 459.0907

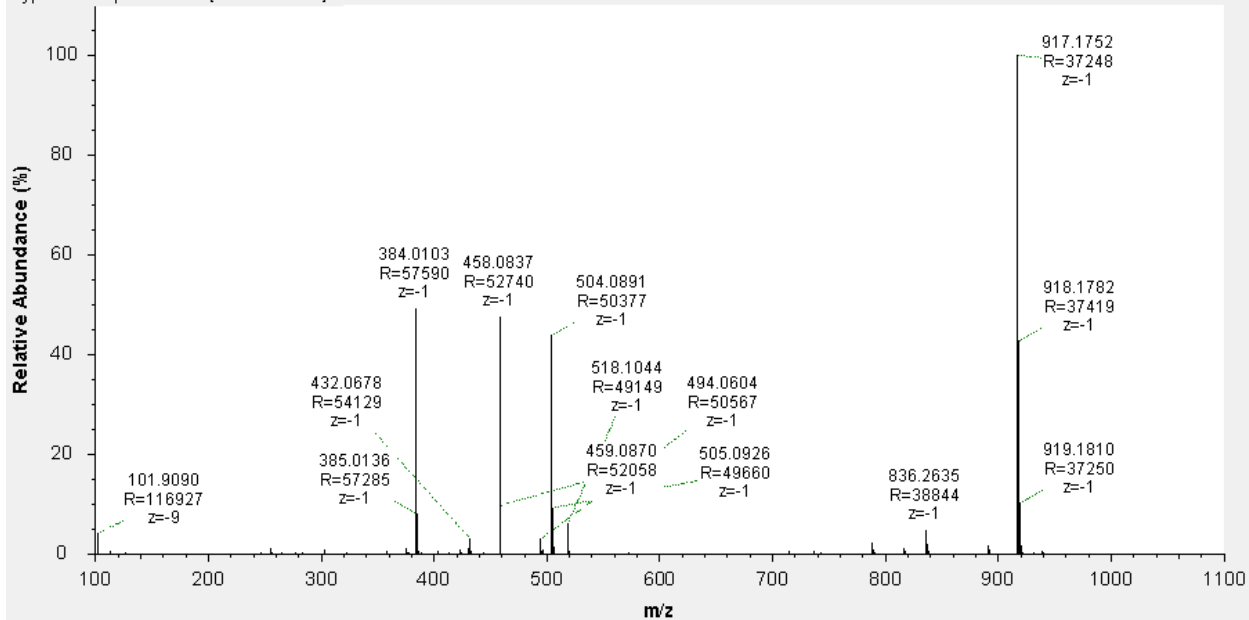
## Positive

Scan: #36308 μS: 1 IT: 4.08 NL: 7.43E7  
 Type: FTMS + p ESI Full ms [120.00-1800.00]

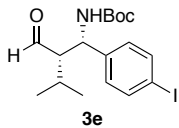


## Negative

Scan: #35896 μS: 1 IT: 2.59 NL: 1.89E8  
 Type: FTMS - p ESI Full ms [100.00-1100.00]



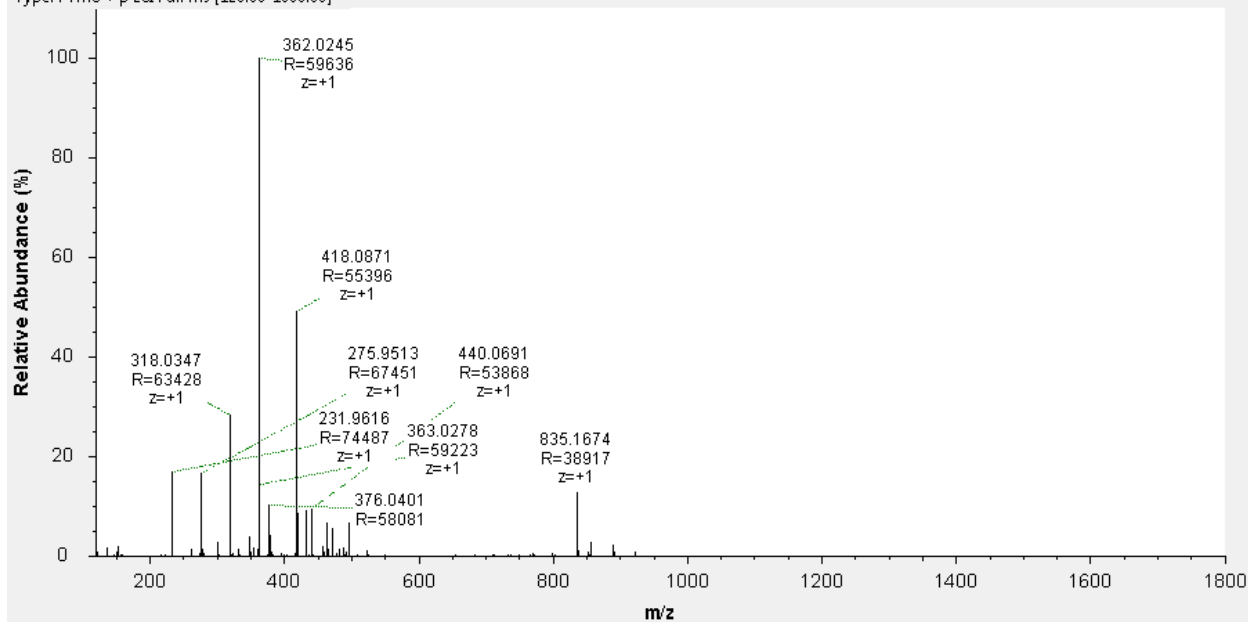
HRMS (ESI+) m/z: [2M+H]<sup>+</sup> Calcd for 919.1887; Found 919.1889. (ESI-) m/z:[M-H]<sup>-</sup>  
 Calcd for 458.0834; Found 458.0837. [2M-H]<sup>-</sup> Calcd for 917.1741; Found 917.1752.



Chemical Formula:  $C_{17}H_{24}INO_3$   
 Exact Mass: 417.0801

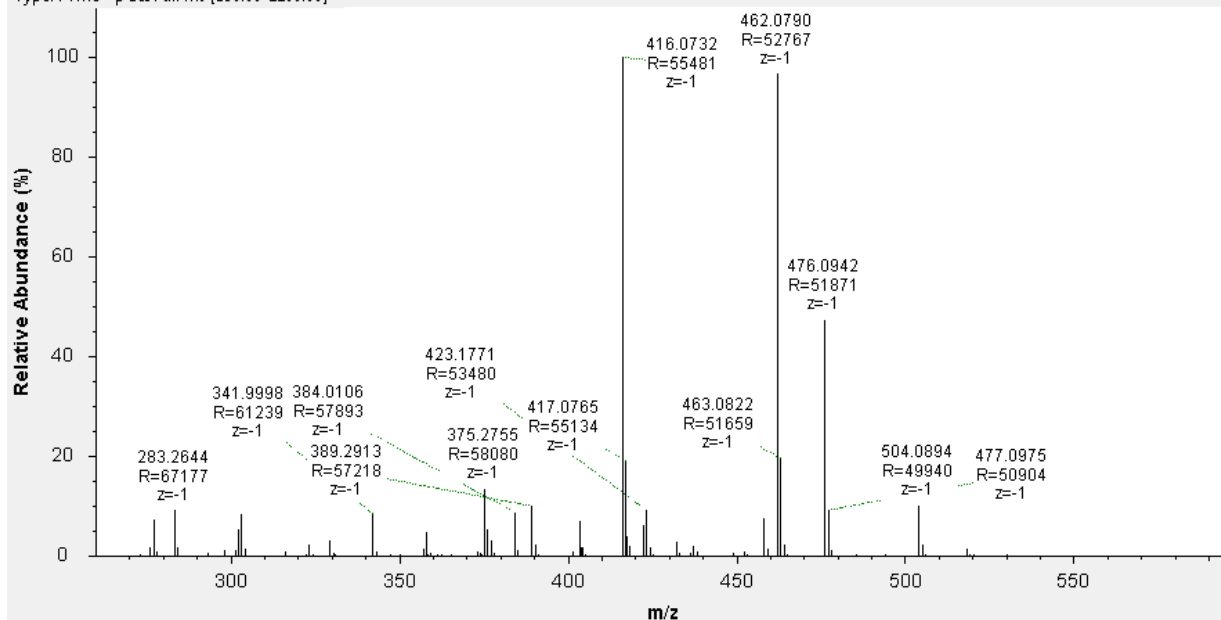
## Positive

Scan: #36494  $\mu$ S: 1 IT: 12.9 NL: 2.91E7  
 Type: FTMS + p ESI Full ms [120.00-1800.00]



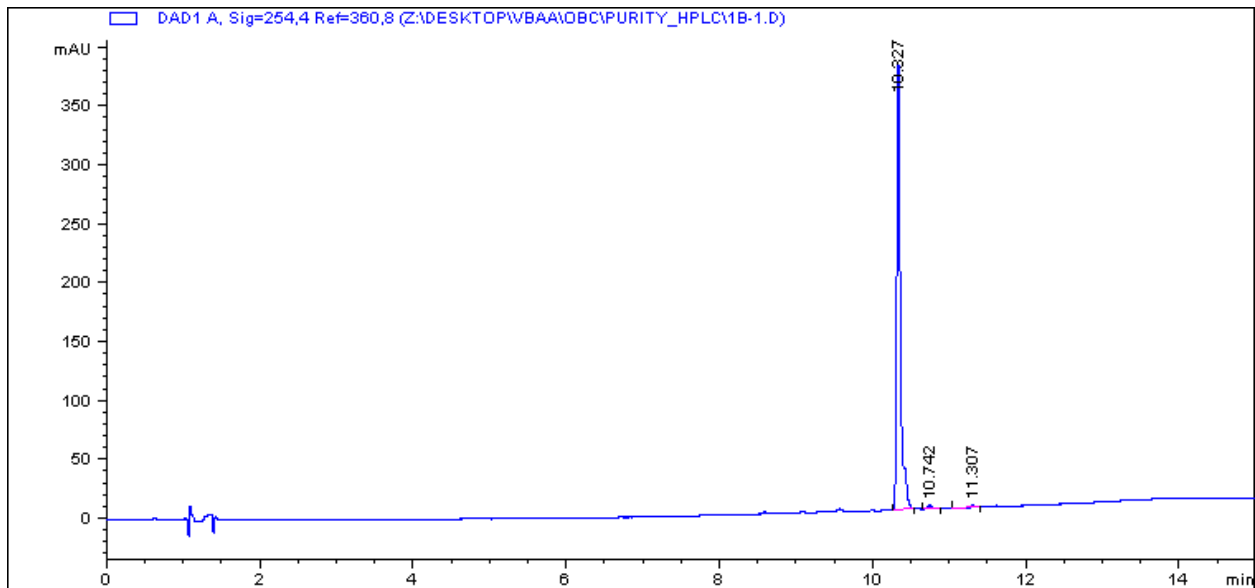
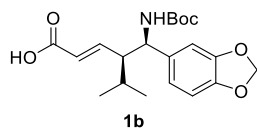
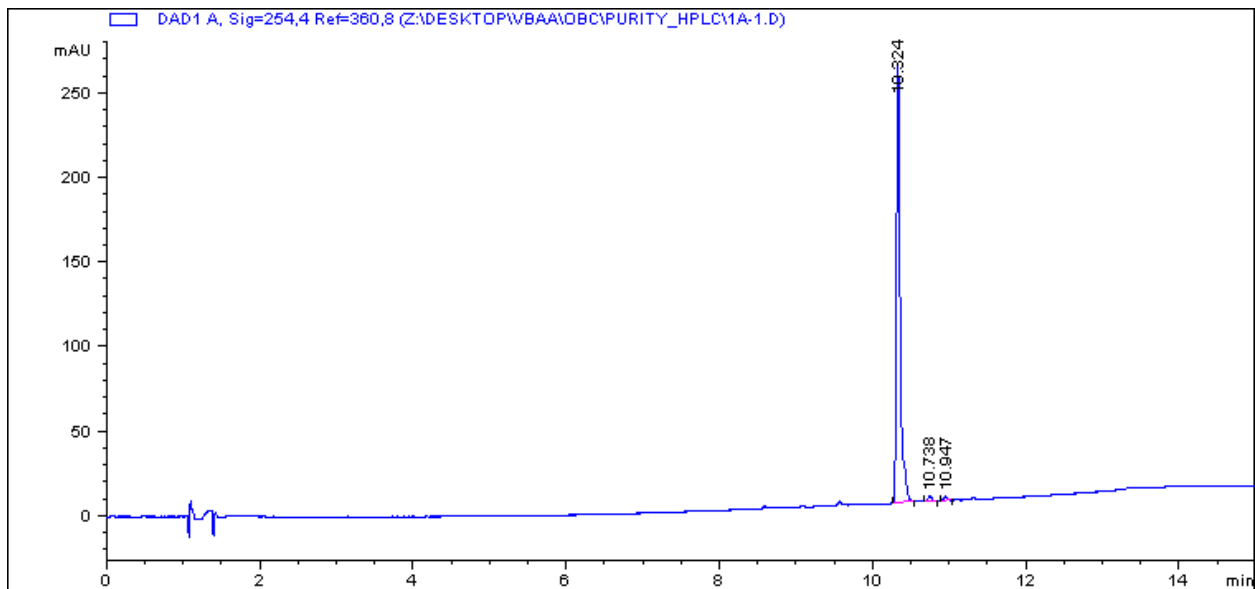
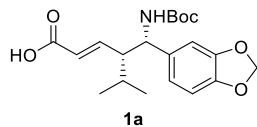
## Negative

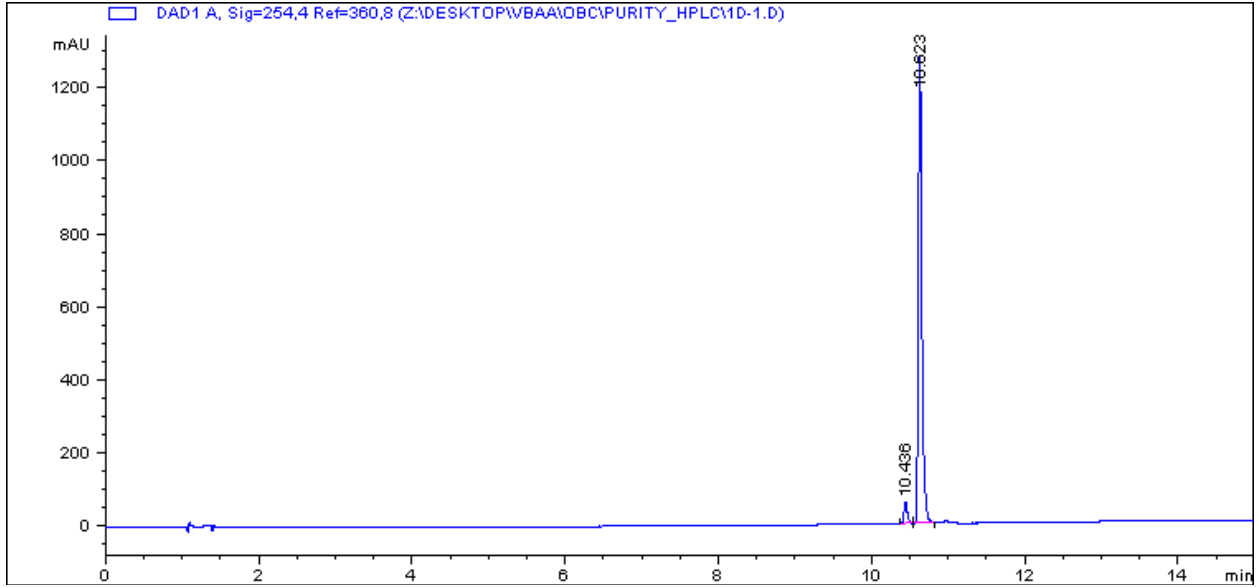
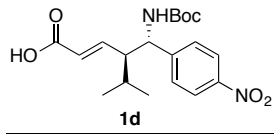
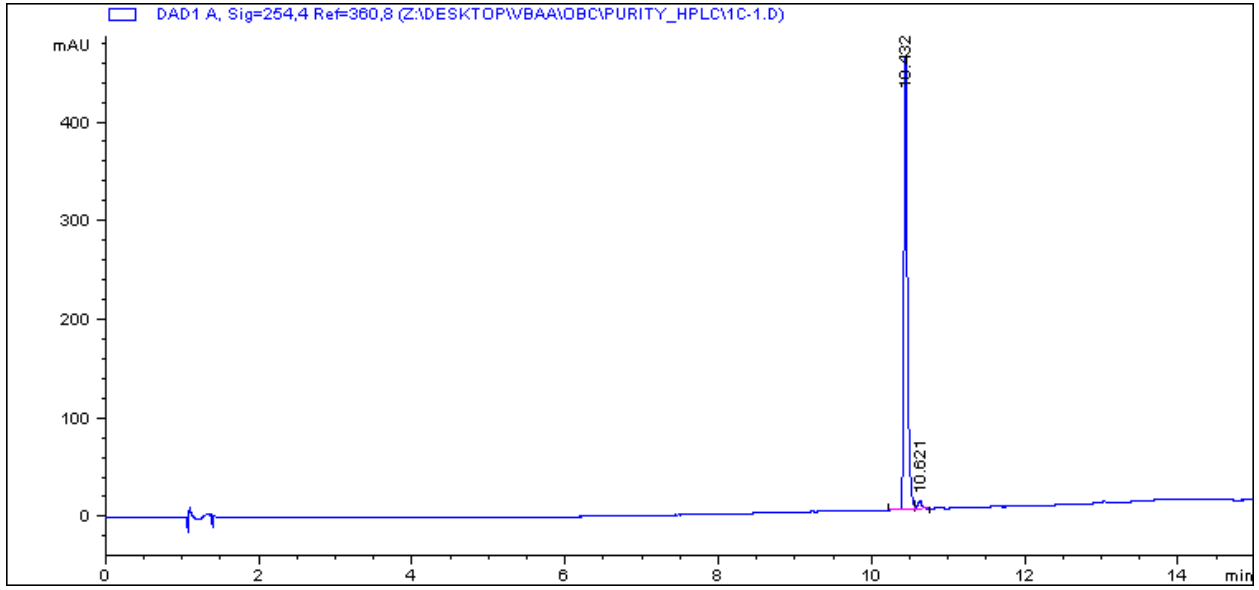
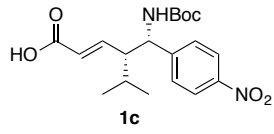
Scan: #33247  $\mu$ S: 1 IT: 4.00 NL: 1.01E8  
 Type: FTMS - p ESI Full ms [250.00-1100.00]

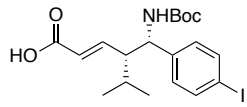


HRMS (ESI+) m/z:  $[M+H]^+$  Calcd for 418.0874; Found 418.0871.  $[2M+H]^+$  Calcd for 835.1675; Found 835.1674. (ESI-) m/z:  $[M-H]^-$  Calcd for 416.0728; Found 416.0732.  $[M+FA-H]^-$  Calcd for 462.0783; Found 462.0790.

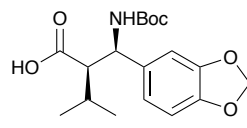
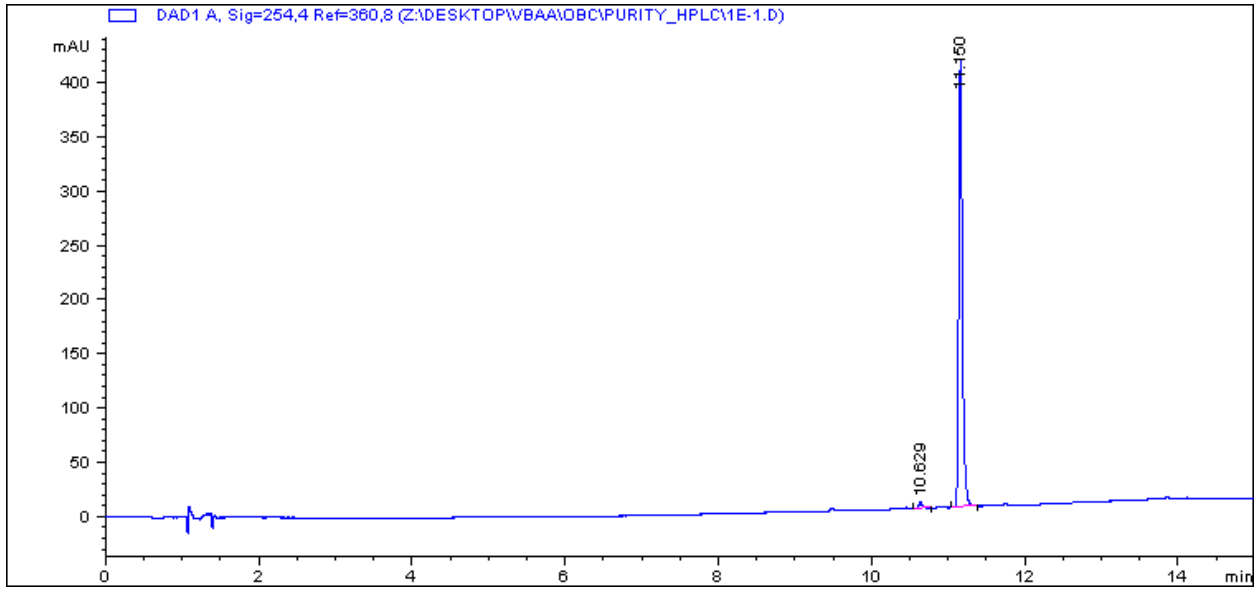
# HPLC Traces



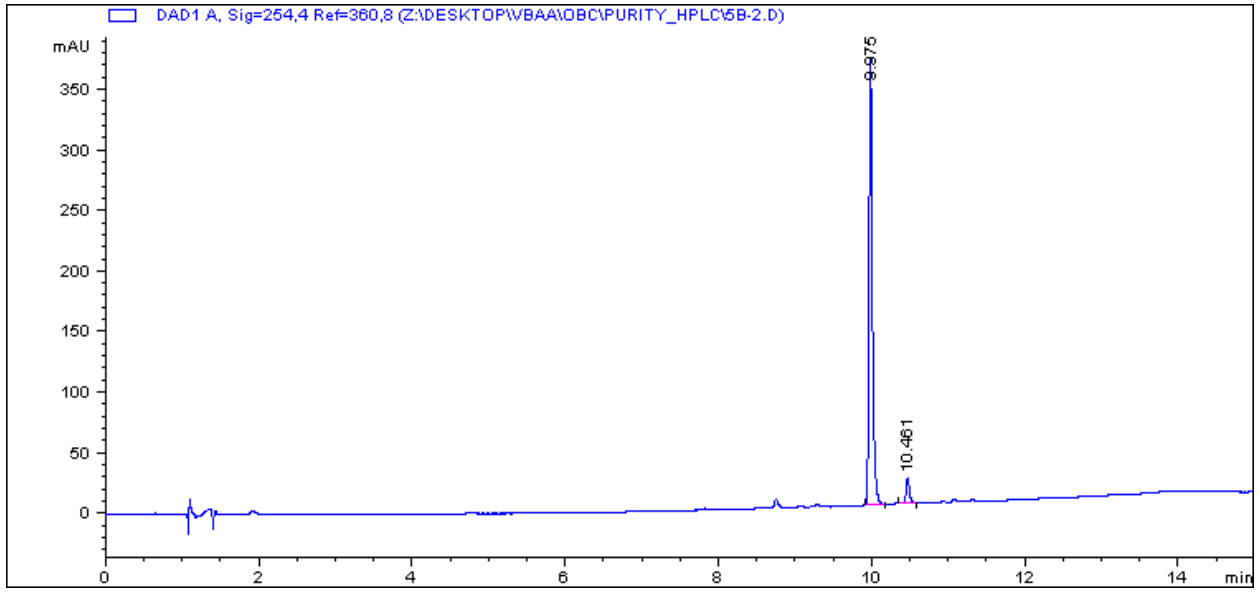




1e



5b



## References

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- (2) Huang, L.; Wulf, W. D. *J. Am. Chem. Soc.* **2011**, *133*, 8892.
- (3) Galzerano, P.; Agostino, D.; Bencivenni, G.; Sambri, L.; Bartoli, G.; Melchiorre, P. *Chem. – Eur. J.* **2010**, *16*, 6069.
- (4) Blanchette, M. A.; Choy, W.; Davis, J. T.; Essenfeld, A. P.; Masamune, S.; Roush, W. R.; Sakai, T. *Tetrahedron Lett.* **1984**, *25*, 2183.
- (5) Huang, N.; Siegel, M. M.; Kruppa, G. H.; Laukien, F. H. *J. Am. Soc. Mass Spectrom.* **1999**, *10*, 1166.