SUPPLEMENTARY INFORMATION

# A base-free hydroxylaminolysis protocol promoted by ZnO in deep eutectic solvents

Xinjie Liang, Bingqing Lv, Shitao Sun, Zhixuan Wu, Bin Lin, Xuefei Bao* and Guoliang Chen*

Key Laboratory of Key Laboratory of Structure-Based Drug Design & Discovery, Ministry of Education, Shenyang Pharmaceutical University, Shenyang 110016, China

* Corresponding author: spucgl@163.com (GL Chen); bxf551@163.com (XF Bao)

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## General Reaction Considerations

All reactions were performed under regular conditions unless otherwise stated. All reactions were magnetically stirred and elevated temperatures were reported as the temperature of the surrounding oil bath. Solvent evaporation was conducted by rotary evaporation at the appropriate temperature and pressure. All normal phase flash column chromatography was conducted using silica gel 200-300 mesh. All reported yields reflect spectroscopically ($^1$H-NMR) pure material unless otherwise stated.

## Materials

Unless stated otherwise, all starting materials, reagents, and solvents were purchased from commercial suppliers and were used without further purification.

## Analysis

Unless otherwise stated, all NMR data were acquired at ambient temperature. NMR solvents, dimethylsulfoxide-$d_6$ (DMSO-$d_6$), were purchased from Shanghai Meryer Chemical Tech Co Ltd. NMR spectra were recorded on Bruker Ascend 600 or Bruker AVIII 400. High-resolution mass spectrometry (HRMS) was conducted on an Agilent Accurate-Mass Q-TOF 6350 and LC-MS-ESI was recorded in Waters Quattro micro-API. Melting points were determined with a X-4 apparatus and were uncorrected.

## General procedure for the preparation of DESs.

A mixture of hydrogen-bond donor and hydrogen-bond acceptor, with the previously specified molar ratio, was added in a round bottom flask under an inert atmosphere. The mixture was stirred for 30 minutes in a T range between 65 and 80 °C obtaining the corresponding DES.

## General procedure for the synthesis of compound 2a to 2k, 2t-S, 2t-R from carboxylic esters

The carboxylic esters (1 mmol) and ZnO (1 mmol) was added to DES (ChCl : glycerol = 1 : 2) solvent (2 g) and stirred for 10 min. The hydroxylamine (50% aqueous solution) 2 mL was added slowly to the stirring solution at room temperature. Then the solution was allowed to stir for 12-36 h at room temperature. The crude mixture was diluted with an equal volume of water and filtered to obtain a complex of the product-zinc oxide. The complex was added to ethanol 20 mL containing oxalic acid (2 mmol.), stirred for 2 h, and filtered. The filtrate was evaporated and treated with water to obtain the target product. The crude products were purified by triturating in petroleum ether or diethyl ether.

## General procedure for the synthesis of compound 2l to 2o from carboxylic esters

The carboxylic esters (1 mmol) and ZnO (1 mmol) was added to DES (ChCl : glycerol = 1 : 2) solvent (2 g) and stirred for 10 min. The hydroxylamine (50% aqueous solution) 2 mL was added slowly to the stirring solution at room temperature. Then the solution was allowed to stir for 12-36 h at room temperature. The crude mixture was diluted with an equal volume of water and filtered to obtain a complex of the product-zinc oxide. The complex was added to ethanol 20 mL containing oxalic acid (2 mmol.), stirred for 2 h, and filtered. The filtrate was evaporated and treated with water to obtain the target product without further purification.

## General procedure for the synthesis of compound 2p to 2r from carboxylic esters

The carboxylic esters (0.5 mmol) and ZnO (1 mmol) was added to DES (ChCl : glycerol = 1 : 2) solvent (2 g) and stirred for 10 min. The hydroxylamine (50% aqueous solution) 2 mL was added slowly to the stirring solution at room temperature. Then the solution was allowed to stir for 24-36 h at 45 $^{o}$C. The crude mixture was diluted with an equal volume of water and filtered to obtain a complex of the product-zinc oxide. The complex was added to ethanol 20 mL containing oxalic acid (2 mmol.), stirred for 2 h, and filtered. The filtrate was evaporated and treated with water to obtain the target product. The crude products were purified by triturating in petroleum ether or diethyl ether.

## The procedure for the synthesis of compound 2n at gram-scale.

The carboxylic esters (1 g, 3.8 mmol) and ZnO (0.62 g, 7.6 mmol) was added to DES (ChCl : glycerol = 1 : 2) solvent (20 g) and stirred for 10 min. The hydroxylamine (50% aqueous solution) 20 mL was added slowly to the stirring solution at room temperature. Then the solution was allowed to stir

for 36 h at room temperature. The crude mixture was diluted with an equal volume of water and filtered to obtain a complex of the product-zinc oxide. The complex was added to ethanol 20 mL containing oxalic acid (2 mmol.), stirred for 2 h, and filtered. The filter cake needs to be treated again according to the same process. The filtrate was evaporated and treated with water to obtain the target product without further purification.

## The procedure for the synthesis of compound 2t-S according to Harris's method.

1,8-Diazabicyclo[5.4.0]undec-7-ene (0.46 g, 3 mmol) was added to a solution of **2t** (0.28 g, 1 mmol) in methanol (1 mL). The resulting solution was stirred at room temperature and hydroxylamine in aqueous solution 50% (0.60 ml, 10.14 mmol) was added. After 4 h the reaction was stopped and purified by column chromatography to afford **2t-S** as an off-white solid.

## Computational Methods

All computations were performed using density functional theory (DFT)[1] implemented in Gaussian 09 suite of program.[2] Geometry optimizations were performed in the gas phase with B3LYP[3,4] levels of theory and 6-31G(d) basis set was used for the geometry optimizations. Vibrational frequency calculations were conducted at the same level, to derive the thermochemistry correction term (G − E) as well as to confirm the stationary point was the minima point on the potential energy surface (no imaginary frequency). After geometry optimizations, the energies were re-evaluated under CAM-B3LYP[5] level with 6-311+G(d,p) for optimized structures. Solvation energy correction was carried out at the same level of the single point energy calculations using PCM continuum solvation model[6] (solvent = ethanol), and solution phase electronic energy ($E_{Sol}$) was evaluated. Final solution phase Gibbs free energy ($G_{Sol}$) were computed as follows:

$$G_{Sol} = E_{Sol} + (G - E)$$

$$\Delta G_{Sol} = \Sigma G_{Sol} \text{ for products} - \Sigma G_{Sol} \text{ for reactants}$$

**B3LYP Geometries for All the Optimized Compounds**

**1a**

$E_{Sol}$ = -1272.420265 hartree

$G_{sol}$ = -1,272.291849 hartree

Number of Imaginary Frequencies: 0

| | | | |
|---|---|---|---|
| C | 2.31111 | -1.58241 | -0.00004 |
| C | 3.34860 | -0.64691 | 0.00003 |
| C | 3.05874 | 0.72021 | 0.00006 |
| C | 1.73445 | 1.15050 | 0.00005 |
| C | 0.69084 | 0.21503 | -0.00001 |
| C | 0.98408 | -1.15580 | -0.00007 |
| H | 2.53650 | -2.64537 | -0.00007 |
| H | 4.38223 | -0.98300 | 0.00005 |
| H | 3.86513 | 1.44840 | 0.00011 |
| H | 1.48451 | 2.20637 | 0.00008 |
| H | 0.17381 | -1.87622 | -0.00013 |
| C | -0.70706 | 0.73458 | -0.00003 |
| O | -1.00035 | 1.91497 | -0.00004 |
| O | -1.62851 | -0.25562 | -0.00003 |
| C | -3.00915 | 0.17035 | -0.00005 |
| H | -3.18571 | 0.79321 | 0.88276 |
| H | -3.18572 | 0.79296 | -0.88303 |
| C | -3.87111 | -1.07822 | 0.00012 |
| H | -4.93021 | -0.79829 | 0.00003 |
| H | -3.67628 | -1.68851 | -0.88767 |
| H | -3.67634 | -1.68821 | 0.88811 |

**1a-1**

$E_{Sol}$ = -1272.420265 hartree

$G_{sol}$ = -1,272.291849 hartree

Number of Imaginary Frequencies: 0

| | | | |
|---|---|---|---|
| C | -3.13243 | -1.81872 | 0.78679 |
| C | -3.05250 | -2.55421 | -0.40033 |
| C | -2.06565 | -2.24295 | -1.33552 |
| C | -1.16521 | -1.20329 | -1.08705 |
| C | -1.23707 | -0.46519 | 0.09650 |
| C | -2.23285 | -0.78211 | 1.03046 |
| H | -3.89707 | -2.05776 | 1.52403 |
| H | -3.75167 | -3.36665 | -0.58960 |
| H | -1.99003 | -2.81331 | -2.25917 |
| H | -0.38838 | -0.95080 | -1.80078 |
| H | -2.28076 | -0.20966 | 1.95130 |
| C | -0.27462 | 0.72146 | 0.34069 |
| O | 0.68711 | 0.83299 | -0.57321 |
| O | -1.09380 | 1.92526 | 0.44128 |
| C | -1.53462 | 2.43564 | -0.80345 |
| H | -0.67745 | 2.59962 | -1.46716 |
| H | -2.20504 | 1.71566 | -1.30214 |
| C | -2.27497 | 3.73885 | -0.53351 |
| H | -2.63893 | 4.17849 | -1.47087 |
| H | -3.13359 | 3.56840 | 0.12618 |
| H | -1.61179 | 4.46213 | -0.04557 |
| N | 0.18959 | 0.66328 | 1.78749 |
| H | 0.45218 | 1.63160 | 1.97941 |
| O | 1.48757 | 0.02060 | 1.85138 |
| H | 1.32875 | -0.94888 | 1.70056 |
| Zn | 2.24289 | -0.26901 | -0.28083 |
| O | 1.79293 | -2.03016 | 0.23699 |
| O | 3.87614 | 0.33153 | -0.83158 |

**1a-2**

$E_{Sol}$ = -1272.420265 hartree

$G_{sol}$ = -1,272.291849 hartree

Number of Imaginary Frequencies: 0

| | | | |
|---|---|---|---|
| C | 2.64118 | 0.61216 | -1.26430 |
| C | 3.37676 | 0.81310 | -0.09063 |
| C | 2.78394 | 0.53895 | 1.14359 |
| C | 1.46878 | 0.06926 | 1.20059 |
| C | 0.72524 | -0.12852 | 0.03513 |
| C | 1.32759 | 0.14680 | -1.19934 |
| H | 3.09808 | 0.81791 | -2.23198 |
| H | 4.40282 | 1.17532 | -0.14082 |
| H | 3.34981 | 0.68786 | 2.06302 |
| H | 0.97736 | -0.16639 | 2.14045 |
| H | 0.75024 | -0.02272 | -2.10303 |

| | | | |
|---|---|---|---|
| C | -0.73924 | -0.61873 | 0.14964 |
| O | -1.11569 | -0.95031 | 1.34414 |
| O | -1.56333 | 0.46949 | -0.51173 |
| C | -1.94942 | 1.48642 | 0.37611 |
| H | -2.19593 | 1.04954 | 1.35315 |
| H | -1.12751 | 2.21139 | 0.54086 |
| C | -3.15449 | 2.21456 | -0.21310 |
| H | -3.48035 | 3.03578 | 0.44085 |
| H | -2.91348 | 2.63427 | -1.19769 |
| H | -3.99209 | 1.51890 | -0.34261 |
| N | -0.97265 | -1.71323 | -0.89564 |
| H | -1.98662 | -1.63540 | -1.04003 |
| O | -0.81388 | -2.95532 | -0.18001 |
| H | -1.01257 | -2.62876 | 0.74202 |

**2a**

$E_{Sol}$ = -1272.420265 hartree

$G_{sol}$ = -1,272.291849 hartree

Number of Imaginary Frequencies: 0

| | | | |
|---|---|---|---|
| C | 2.18700 | -1.31985 | 0.12886 |
| C | 2.99831 | -0.20155 | -0.07653 |
| C | 2.42352 | 1.06601 | -0.20117 |
| C | 1.04090 | 1.21581 | -0.12822 |
| C | 0.22151 | 0.09467 | 0.06123 |
| C | 0.80248 | -1.17471 | 0.20045 |
| H | 2.63309 | -2.30355 | 0.24533 |
| H | 4.07747 | -0.31676 | -0.13042 |
| H | 3.05425 | 1.93745 | -0.35295 |
| H | 0.57469 | 2.19176 | -0.21516 |
| H | 0.17737 | -2.04078 | 0.39892 |
| C | -1.24983 | 0.31217 | 0.11293 |
| O | -1.78154 | 1.39543 | 0.36722 |
| N | -2.04425 | -0.79323 | -0.10609 |
| H | -1.76961 | -1.44966 | -0.82848 |
| O | -3.41387 | -0.50593 | -0.23206 |
| H | -3.41751 | 0.46281 | -0.04112 |

ethanol

$E_{Sol}$ = -1272.420265 hartree

$G_{sol}$ = -1,272.291849 hartree

Number of Imaginary Frequencies: 0

| | | | |
|---|---|---|---|
| O | 1.15046 | -0.39972 | -0.00001 |
| C | 0.08705 | 0.54883 | -0.00008 |
| H | 0.13852 | 1.20105 | 0.88744 |

| | | | |
|---|---|---|---|
| H | 0.13843 | 1.20081 | -0.88778 |
| C | -1.22269 | -0.22199 | 0.00009 |
| H | -2.07541 | 0.46601 | 0.00003 |
| H | -1.28898 | -0.86085 | -0.88670 |
| H | -1.28890 | -0.86059 | 0.88707 |
| H | 1.98653 | 0.09035 | -0.00007 |

$ZnO_2$

$E_{Sol}$ = -1272.420265 hartree

$G_{sol}$ = -1,272.291849 hartree

Number of Imaginary Frequencies: 0

| | | | |
|---|---|---|---|
| Zn | 0.00000 | 0.00000 | 0.00000 |
| O | 0.00000 | 0.00000 | 1.70656 |
| O | 0.00000 | 0.00000 | -1.70656 |

hydroxylamine

$E_{Sol}$ = -1272.420265 hartree

$G_{sol}$ = -1,272.291849 hartree

Number of Imaginary Frequencies: 0

| | | | |
|---|---|---|---|
| N | -0.69132 | -0.00050 | 0.15726 |
| H | -1.03926 | 0.81199 | -0.35714 |
| H | -1.03973 | -0.80958 | -0.36220 |
| O | 0.72556 | 0.00049 | -0.14111 |
| H | 1.11373 | -0.00284 | 0.74735 |

**Reference**


(1) Parr, R. G.; Weitao, Y. *Density-Functional Theory of Atoms and Molecules*; Oxford University Press, 1995. https://doi.org/10.1093/oso/9780195092769.001.0001.

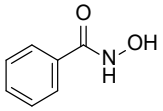
(2) Gaussian 09, Revision D.01, M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, B. Mennucci, G. A. Petersson, H. Nakatsuji, M. Caricato, X. Li, H. P. Hratchian, A. F. Izmaylov, J. Bloino, G. Zheng, J. L. Sonnenberg, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. Bearpark, J. J. Heyd, E. Brothers, K. N. Kudin, V. N. Staroverov, T. Keith, R. Kobayashi, J. Normand, K. Raghavachari, A. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, N. Rega, J. M. Millam, M. Klene, J. E. Knox, J. B. Cross, V. Bakken, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, R. L. Martin, K. Morokuma, V. G. Zakrzewski, G. A. Voth, P. Salvador, J. J. Dannenberg, S. Dapprich, A. D. Daniels, O. Farkas, J. B. Foresman, J. V. Ortiz, J. Cioslowski, and D. J. Fox, Gaussian, Inc., Wallingford CT, 2013.

(3) Lee, C.; Yang, W.; Parr, R. G. Development of the Colle-Salvetti Correlation-Energy Formula into a Functional of the Electron Density. *Phys. Rev. B* **1988**, *37* (2), 785–789. https://doi.org/10.1103/PhysRevB.37.785.

(4) Becke, A. D. Density-functional Thermochemistry. III. The Role of Exact Exchange. *J. Chem. Phys.* **1993**, *98* (7), 5648–5652. https://doi.org/10.1063/1.464913.

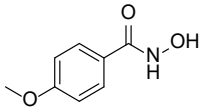
(5) Yanai, T.; Tew, D. P.; Handy, N. C. A New Hybrid Exchange–Correlation Functional Using the Coulomb-Attenuating Method (CAM-B3LYP). *Chem. Phys. Lett.* **2004**, *393* (1–3), 51–57. https://doi.org/10.1016/j.cplett.2004.06.011.
(6) Tomasi, J.; Mennucci, B.; Cammi, R. Quantum Mechanical Continuum Solvation Models. *Chem. Rev.* **2005**, *105* (8), 2999–3094. https://doi.org/10.1021/cr9904009.

**Hydroxamic Acids**



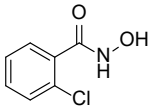
**2a**

Pinkish solid; $^{1}$H NMR (400 MHz, DMSO-$d_6$) δ 11.20 (s, 1H), 9.03 (s, 1H), 7.79-7.71 (m, 2H), 7.55-7.48 (m, 1H), 7.48-7.41 (m, 2H). $^{13}$C NMR (101 MHz, DMSO-$d_6$) δ 164.64, 133.25, 131.58, 128.84, 127.31. HRMS (ESI): m/z [M-H]$^-$ calculated for $C_7H_6O_2$: 136.0404; found: 136.0412. m.p. 104-106 °C.



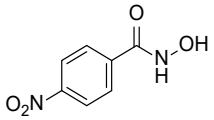
**2b**

Light brown solid, $^{1}$H NMR (400 MHz, DMSO-$d_6$) δ 11.05 (s, 1H), 8.88 (s, 1H), 7.73 (d, *J* = 8.8 Hz, 2H), 6.98 (d, *J* = 8.8 Hz, 2H), 3.80 (s, 3H). $^{13}$C NMR (101 MHz, DMSO-$d_6$) δ 164.52, 161.97, 129.10, 125.42, 114.09, 55.79. HRMS (ESI): m/z [M-H]$^-$ calculated for $C_8H_8NO_3$: 166.0510; found: 166.0526. m.p. 140-143 °C.



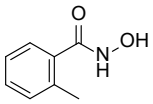
**2c**

White solid, $^{1}$H NMR (400 MHz, DMSO-$d_6$) δ 10.95 (s, 1H), 9.21 (s, 1H), 7.54-7.42 (m, 2H), 7.42-7.35 (m, 2H). $^{13}$C NMR (101 MHz, DMSO-$d_6$) δ 163.74, 135.10, 131.55, 131.08, 130.15, 129.82, 127.57. HRMS (ESI): m/z [M-H]$^-$ calculated for $C_7H_5ClNO_2$: 170.0014; found: 170.0012. m.p. 120-122 °C.



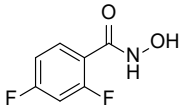
**2d**

Yellowish solid, $^{1}$H NMR (400 MHz, DMSO-$d_6$) δ 11.52 (s, 1H), 9.31 (s, 1H), 8.30 (d, *J* = 8.3 Hz, 2H), 7.99 (d, *J* = 8.3 Hz, 2H). $^{13}$C NMR (101 MHz, DMSO-$d_6$) δ 162.78, 149.44, 138.99, 128.87, 124.09. HRMS (ESI): m/z [M-H]$^-$ calculated for $C_7H_5N_2O_4$: 181.0255; found: 181.0256. m.p. 157-160 °C.



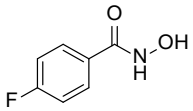
**2e**

White solid, $^{1}$H NMR (400 MHz, DMSO-$d_6$) δ 10.79 (s, 1H), 9.01 (s, 1H), 7.35-7.31 (m, 1H), 7.28-7.15 (m, 3H), 2.33 (s, 3H). $^{13}$C NMR (101 MHz, DMSO-$d_6$) δ 166.62, 136.17, 135.19, 130.89, 129.96, 127.80, 125.96, 19.65. HRMS (ESI): m/z [M-H]$^-$ calculated for $C_8H_8NO_2$: 150.0561; found: 150.0546. m.p. 116-120 °C.



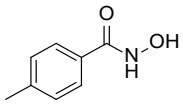
**2f**

White solid, $^{1}$H NMR (600 MHz, DMSO-$d_6$) δ 11.00 (s, 1H), 9.24 (s, 1H), 7.62 (q, *J* = 7.8 Hz, 1H), 7.38-7.32 (m, 1H), 7.20-7.14 (m, 1H). $^{13}$C NMR (151 MHz, DMSO-$d_6$) δ 164.51 (d, *J* = 12.5 Hz), 162.86 (d, *J* = 12.0 Hz), 160.91, 160.79 (d, *J* = 13.0 Hz), 159.12 (d, *J* = 13.0 Hz), 132.01 (d, *J* = 4.9 Hz), 131.94 (d, *J* = 4.7 Hz), 119.48 (d, *J* = 15.5 Hz), 112.21 (d, *J* = 20.1 Hz), 104.96 (t, *J* = 26.3 Hz). HRMS (ESI): m/z [M-H]$^-$ calculated for $C_7H_4F_2NO_2$: 172.0216; found: 172.0222. m.p. 142-146 °C.



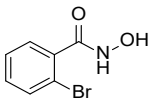
**2g**

Synthesized by following general procedure providing the product as an off-white solid in 90.1 % yield; $^{1}$H NMR (600 MHz, DMSO-$d_6$) δ 11.24 (s, 1H), 9.06 (s, 1H), 7.90 – 7.77 (m, 2H), 7.38 – 7.23 (m, 2H). $^{13}$C NMR (151 MHz, DMSO-$d_6$) δ 164.98, 163.61, 163.34, 129.87, 129.81, 129.64, 115.81, 115.66. HRMS (ESI): m/z [M-H]$^-$ calculated for $C_7H_5FNO_2$: 154.0310; found: 154.0307. m.p. 153-156 °C.



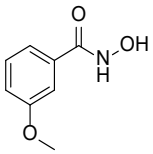
**2h**

White solid, $^{1}$H NMR (400 MHz, DMSO-$d_6$) δ 11.14 (s, 1H), 8.97 (s, 1H), 7.65 (d, *J* = 8.2 Hz, 2H), 7.25 (d, *J* = 7.9 Hz, 2H), 2.34 (s, 3H). $^{13}$C NMR (101 MHz, DMSO-$d_6$) δ 164.69, 141.44, 130.44, 129.35, 127.32, 21.40. HRMS (ESI): m/z [M-H]$^-$ calculated for $C_8H_8NO_2$: 150.0561; found: 150.0595. m.p. 136-138 °C.



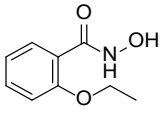
**2i**

White solid, $^{1}$H NMR (400 MHz, DMSO-$d_6$) δ 10.93 (s, 1H), 9.20 (s, 1H), 7.66 (dd, *J* = 8.1, 1.4 Hz, 1H), 7.48-7.33 (m, 3H). $^{13}$C NMR (101 MHz, DMSO-$d_6$) δ 164.60, 137.21, 133.28, 131.69, 129.84, 128.04, 120.21. HRMS (ESI): m/z [M-H]$^-$ calculated for $C_7H_5BrNO_2$: 213.9509, 215.9489; found: 213.9519, 215.9500. m.p. 161-164 °C.



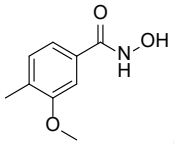
**2j**

Light brown solid, $^{1}$H NMR (600 MHz, DMSO-$d_6$) δ 11.20 (s, 1H), 9.03 (s, 1H), 7.38-7.27 (m, 3H), 7.08 (dd, *J* = 7.9, 1.5 Hz, 1H), 3.79 (s, 3H). $^{13}$C NMR (151 MHz, DMSO-$d_6$) δ 164.28, 159.52, 134.54, 129.93, 119.44, 117.34, 112.42, 55.60. HRMS (ESI): m/z [M-H]$^-$ calculated for $C_8H_8NO_3$: 166.0510; found: 166.0524. m.p. 86-88 °C.



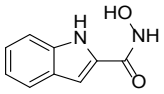
**2k**

Light brown solid, $^{1}$H NMR (400 MHz, DMSO-$d_6$) δ 10.48 (s, 1H), 9.07 (s, 1H), 7.53 (d, *J* = 7.2 Hz, 1H), 7.41 (t, *J* = 7.6 Hz, 1H), 7.08 (d, *J* = 8.3 Hz, 1H), 6.99 (t, *J* = 7.3 Hz, 1H), 4.12 (q, *J* = 6.9 Hz, 2H), 1.34 (t, *J* = 6.9 Hz, 3H). $^{13}$C NMR (101 MHz, DMSO-$d_6$) δ 163.83, 156.30, 132.15, 130.28, 123.32, 120.76, 113.25, 64.43, 14.96. HRMS (ESI): m/z [M-H]$^-$ calculated for $C_9H_{10}NO_3$: 180.0666; found: 180.0659. m.p. 108-110 °C.



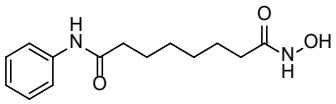
**2l**

White solid, $^{1}$H NMR (400 MHz, DMSO-$d_6$) δ 11.14 (s, 1H), 8.95 (s, 1H), 7.30 (d, *J* = 1.6 Hz, 1H), 7.26 (dd, *J* = 7.7, 1.6 Hz, 1H), 7.19 (d, *J* = 7.7 Hz, 1H), 3.82 (s, 3H), 2.17 (s, 3H). $^{13}$C NMR (101 MHz, DMSO-$d_6$) δ 164.58, 157.58, 132.15, 130.63, 129.57, 119.28, 109.07, 55.79, 16.44. HRMS (ESI): m/z [M-H]$^-$ calculated for $C_9H_{10}NO_3$: 180.0666; found: 180.0679. m.p. 147-150 °C.



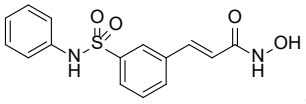
**2m**

Light brown solid, $^{1}$H NMR (600 MHz, DMSO-$d_6$) δ 11.62 (s, 1H), 11.24 (s, 1H), 9.12 (s, 1H), 7.59 (d, *J* = 8.0 Hz, 1H), 7.42 (d, *J* = 8.2 Hz, 1H), 7.17 (t, *J* = 7.6 Hz, 1H), 7.03 (t, *J* = 7.5 Hz, 1H), 6.98 (s, 1H). $^{13}$C NMR (151 MHz, DMSO-$d_6$) δ 160.00, 136.69, 129.93, 127.45, 123.53, 121.78, 120.13, 112.61, 102.00. HRMS (ESI): m/z [M-H]$^-$ calculated for $C_9H_7N_2O_2$: 175.0513; found: 175.0515. m.p. 131-134 °C.



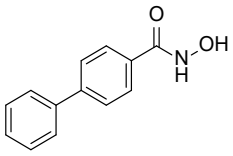
**2n**

Synthesized by following general procedure providing the product as a white solid in 90.5% yield; $^{1}$H NMR (400 MHz, DMSO-$d_6$) δ 10.33 (s, 1H), 9.84 (s, 1H), 8.66 (s, 1H), 7.58 (d, *J* = 7.3 Hz, 2H), 7.35-7.21 (m, 2H), 7.02 (t, *J* = 7.4 Hz, 1H), 2.29 (t, *J* = 7.5 Hz, 2H), 1.94 (t, *J* = 7.4 Hz, 2H), 1.57 (p, *J* = 7.2 Hz, 2H), 1.49 (p, *J* = 7.2 Hz, 2H), 1.36-1.20 (m, 4H). $^{13}$C NMR (101 MHz, DMSO-$d_6$) δ 171.69, 169.57, 139.81, 129.10, 123.38, 119.49, 36.84, 32.71, 28.88, 25.49.



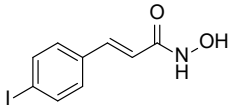
**2o**

Yellowish solid, $^{1}$H NMR (400 MHz, DMSO-$d_6$) δ 10.81 (s, 1H), 10.32 (s, 1H), 9.12 (s, 1H), 7.91 (s, 1H), 7.78 (d, *J* = 7.9 Hz, 1H), 7.70 (d, *J* = 8.1 Hz, 1H), 7.57 (t, *J* = 7.8 Hz, 1H), 7.46 (d, *J* = 15.9 Hz, 1H), 7.26-7.19 (m, 2H), 7.14 – 7.07 (m, 2H), 7.06-7.01 (m, 1H), 6.50 (d, *J* = 15.8 Hz, 1H). $^{13}$C NMR (101 MHz, DMSO-$d_6$) δ 162.51, 140.75, 137.98, 136.97, 136.36, 132.45, 130.49, 129.68, 127.53, 125.20, 124.80, 121.83, 120.83. HRMS (ESI): m/z [M-H]$^-$ calculated for $C_{15}H_{13}N_2O_4S$: 317.0602; found: 317.0620. m.p. 168-171 °C.



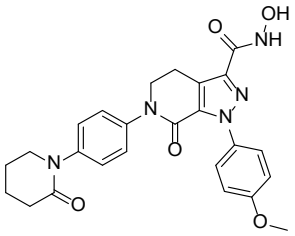
**2p**

White solid, $^{1}$H NMR (600 MHz, DMSO-$d_6$) δ 11.28 (s, 1H), 9.07 (s, 1H), 7.86 (d, *J* = 8.0 Hz, 2H), 7.76 (d, *J* = 8.0 Hz, 2H), 7.72 (d, *J* = 7.6 Hz, 2H), 7.49 (t, *J* = 7.6 Hz, 2H), 7.41 (t, *J* = 7.4 Hz, 1H). $^{13}$C NMR (151 MHz, DMSO-$d_6$) δ 164.27, 143.09, 139.57, 131.98, 129.43, 128.43, 127.91, 127.23, 127.00. HRMS (ESI): m/z [M-H]$^-$ calculated for $C_{13}H_{10}NO_2$: 212.0717; found: 212.0720. m.p. 186-190 °C.



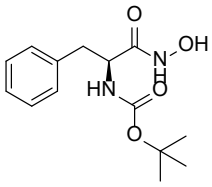
**2q**

Light brown solid, $^{1}$H NMR (400 MHz, DMSO-$d_6$) δ 10.76 (s, 1H), 9.05 (s, 1H), 7.77 (d, *J* = 8.1 Hz, 2H), 7.53-7.26 (m, 3H), 6.48 (d, *J* = 15.8 Hz, 1H). $^{13}$C NMR (101 MHz, DMSO-$d_6$) δ 162.84, 138.22, 137.73, 134.91, 129.89, 120.39, 96.38. HRMS (ESI): m/z [M-H]$^-$ calculated for $C_9H_7INO_2$: 287.9527; found: 287.9534. m.p. 189-192 °C.



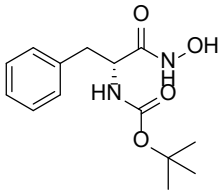
**2r**

Off-white solid, $^{1}$H NMR (400 MHz, DMSO-$d_6$) δ 11.13 (s, 1H), 9.03 (s, 1H), 7.50 (d, *J* = 8.9 Hz, 2H), 7.35 (d, *J* = 8.8 Hz, 2H), 7.27 (d, *J* = 8.9 Hz, 2H), 6.99 (d, *J* = 9.0 Hz, 2H), 4.06 (t, *J* = 6.6 Hz, 2H), 3.80 (s, 3H), 3.59 (t, *J* = 5.7 Hz, 2H), 3.18 (t, *J* = 6.6 Hz, 2H), 2.38 (t, *J* = 6.3 Hz, 2H), 1.90-1.78 (m, 4H). $^{13}$C NMR (101 MHz, DMSO-$d_6$) δ 169.33, 166.61, 159.60, 157.05, 141.89, 140.73, 140.29, 133.10, 133.00, 127.24, 126.81, 126.50, 125.50, 113.87, 55.95, 51.38, 51.30, 33.06, 23.47, 21.36, 21.30. HRMS (ESI): m/z $[M-H]^-$ calculated for $C_{25}H_{24}N_5O_5$: 474.1783; found: 474.1802. m.p. 133-136 °C.



**2t-s**

white solid, $^{1}$H NMR (400 MHz, DMSO-d6) δ 10.61 (d, J = 1.6 Hz, 1H), 8.83 (d, J = 1.6 Hz, 1H), 7.36–7.12 (m, 5H), 6.96 (d, J = 8.6 Hz, 1H), 4.07-4.01 (m, 1H), 2.91–2.71 (m, 2H), 1.31 (s, 9H). $^{13}$C NMR (101 MHz, DMSO-$d_6$) δ 168.88, 155.56, 138.52, 129.64, 128.49, 126.67, 78.38, 53.99, 38.17, 28.63, 28.31. MS (ESI): m/z $[M+Na]^+$ : 303.1. m.p. 136-138 °C.



**2t-r**

white solid, $^{1}$H NMR (400 MHz, DMSO-d6) δ 10.60 (s, 1H), 8.83 (s, 1H), 7.34 – 7.12 (m, 5H), 6.96 (d, J = 8.6 Hz, 1H), 4.07-4.01 (m, 1H), 2.92–2.70 (m, 2H), 1.31 (s, 9H). $^{13}$C NMR (101 MHz, DMSO-$d_6$) δ 168.88, 155.55, 138.52, 129.63, 128.49, 126.67, 78.38, 53.98, 38.18, 28.63, 28.32. MS (ESI): m/z $[M+Na]^+$ : 303.1. m.p. 140-143 °C.

**Figure S1**. HPLC spectrum of Vorinostat (1st run)

Column: Shimadzu Wondasil Superb C18 (5 µm; 150 mm × 4.6 mm),

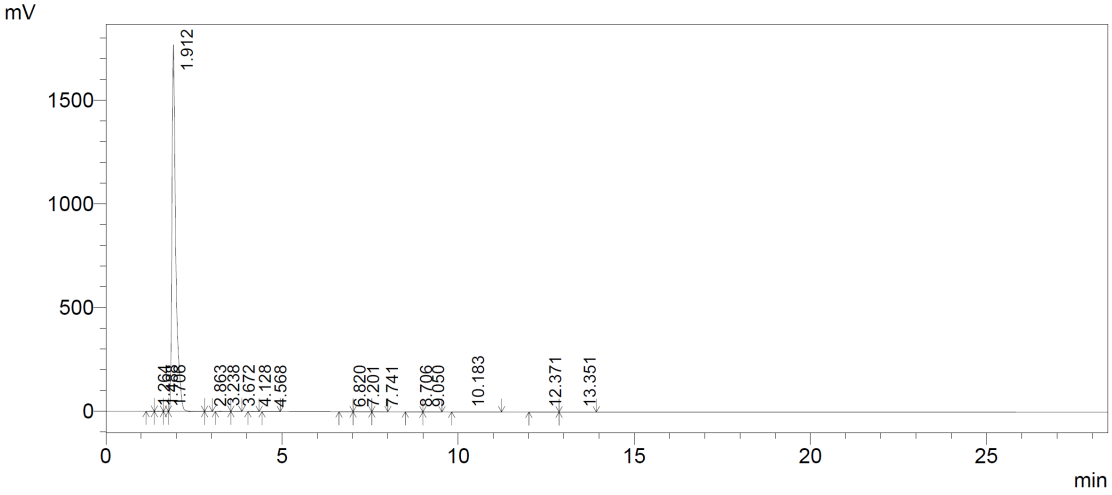
Flow Rate: 1 mL/min;

Oven Temp: 30 °C;

Inj. volume: 5 µL;

Detection: 241 nm

Eluent: 0.1 % phosphoric acid (aq): acetonitrile =60 : 40.




| peak# | retention time | peakarea | peak height | area % | height % |
|---|---|---|---|---|---|
| 1 | 1.264 | 10014 | 1440 | 0.074 | 0.081 |
| 2 | 1.488 | 14919 | 1368 | 0.110 | 0.077 |
| 3 | 1.706 | 7437 | 1191 | 0.055 | 0.067 |
| 4 | 1.912 | 13437975 | 1769273 | 99.137 | 99.454 |
| 5 | 2.863 | 1034 | 169 | 0.008 | 0.010 |
| 6 | 3.238 | 17490 | 1926 | 0.129 | 0.108 |
| 7 | 3.672 | 4921 | 588 | 0.036 | 0.033 |
| 8 | 4.128 | 2467 | 298 | 0.018 | 0.017 |
| 9 | 4.568 | 2664 | 246 | 0.020 | 0.014 |
| 10 | 6.820 | 2050 | 177 | 0.015 | 0.010 |
| 11 | 7.201 | 3800 | 278 | 0.028 | 0.016 |
| 12 | 7.741 | 3377 | 244 | 0.025 | 0.014 |
| 13 | 8.706 | 2220 | 94 | 0.016 | 0.005 |
| 14 | 9.050 | 2060 | 110 | 0.015 | 0.006 |
| 15 | 10.183 | 24986 | 966 | 0.184 | 0.054 |
| 16 | 12.371 | 7703 | 316 | 0.057 | 0.018 |
| 17 | 13.351 | 9845 | 308 | 0.073 | 0.017 |
| total | | 13554964 | 1778991 | 100.000 | 100.000 |

**Figure S2**. HPLC spectrum of Vorinostat (2nd run)

Column: Shimadzu Wondasil Superb C18 (5 µm; 150 mm × 4.6 mm),

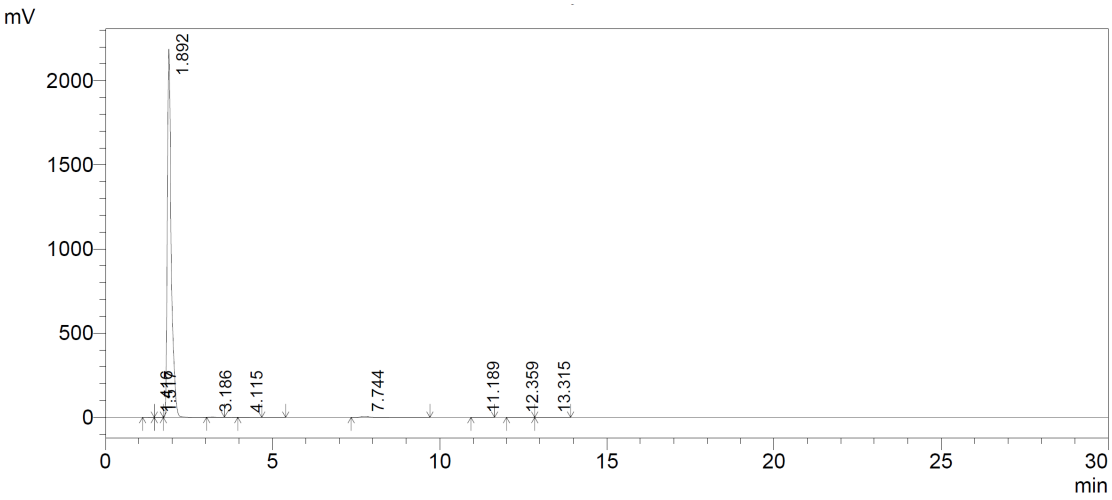
Flow Rate: 1 mL/min;

Oven Temp: 30 °C;

Inj. volume: 5 µL;

Detection: 241 nm

Eluent: 0.1 % phosphoric acid (aq): acetonitrile =60 : 40.




| peak# | retention time | peak area | peak height | area % | height % |
|---|---|---|---|---|---|
| 1 | 1.416 | 7400 | 869 | 0.041 | 0.040 |
| 2 | 1.517 | 10309 | 986 | 0.057 | 0.045 |
| 3 | 1.892 | 18010897 | 2187695 | 99.048 | 99.533 |
| 4 | 3.186 | 23899 | 2311 | 0.131 | 0.105 |
| 5 | 4.115 | 8937 | 731 | 0.049 | 0.033 |
| 6 | 7.744 | 104062 | 4692 | 0.572 | 0.213 |
| 7 | 11.189 | 2616 | 108 | 0.014 | 0.005 |
| 8 | 12.359 | 7130 | 279 | 0.039 | 0.013 |
| 9 | 13.315 | 8843 | 293 | 0.049 | 0.013 |
| total | | 18184093 | 2197964 | 100.000 | 100.000 |

**Figure S3**. HPLC spectrum of Vorinostat (3rd run)

Column: Shimadzu Wondasil Superb C18 (5 µm; 150 mm × 4.6 mm),

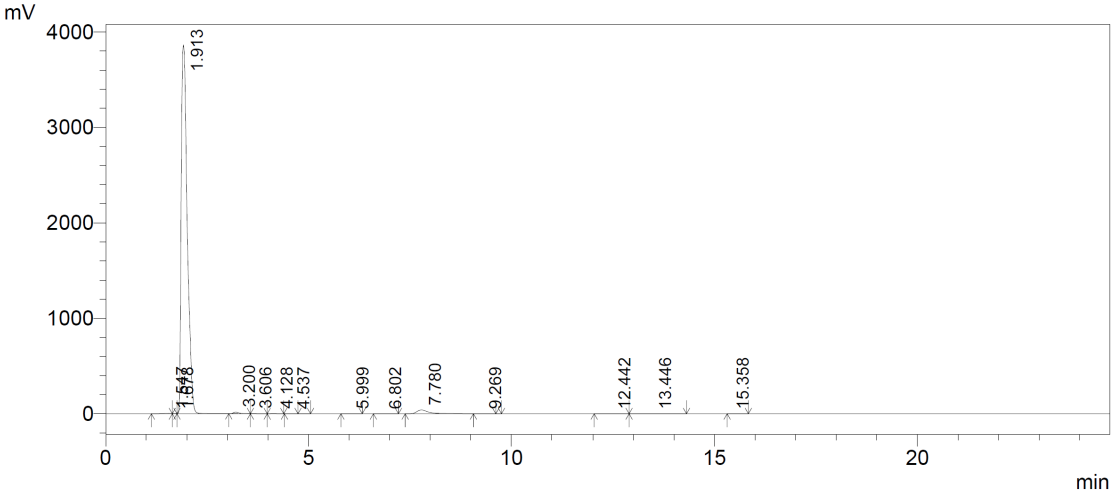
Flow Rate: 1 mL/min;

Oven Temp: 30 °C;

Inj. volume: 5 µL;

Detection: 241 nm

Eluent: 0.1 % phosphoric acid (aq): acetonitrile =60 : 40.




| peak# | retention time | peak area | peak height | area % | height % |
|---|---|---|---|---|---|
| 1 | 1.547 | 47007 | 3918 | 0.115 | 0.100 |
| 2 | 1.678 | 11396 | 1909 | 0.028 | 0.049 |
| 3 | 1.913 | 39857751 | 3865460 | 97.199 | 98.319 |
| 4 | 3.200 | 173687 | 17243 | 0.424 | 0.439 |
| 5 | 3.606 | 8270 | 1004 | 0.020 | 0.026 |
| 6 | 4.128 | 6479 | 669 | 0.016 | 0.017 |
| 7 | 4.537 | 2197 | 232 | 0.005 | 0.006 |
| 8 | 5.999 | 1532 | 126 | 0.004 | 0.003 |
| 9 | 6.802 | 1119 | 72 | 0.003 | 0.002 |
| 10 | 7.780 | 863668 | 39778 | 2.106 | 1.012 |
| 11 | 9.269 | 2192 | 135 | 0.005 | 0.003 |
| 12 | 12.442 | 8870 | 347 | 0.022 | 0.009 |
| 13 | 13.446 | 20558 | 585 | 0.050 | 0.015 |
| 14 | 15.358 | 1433 | 74 | 0.003 | 0.002 |
| total | | 41006159 | 3931554 | 100.000 | 100.000 |

**Figure S4**. HPLC spectrum of Vorinostat (4th run)

Column: Shimadzu Wondasil Superb C18 (5 µm; 150 mm × 4.6 mm),

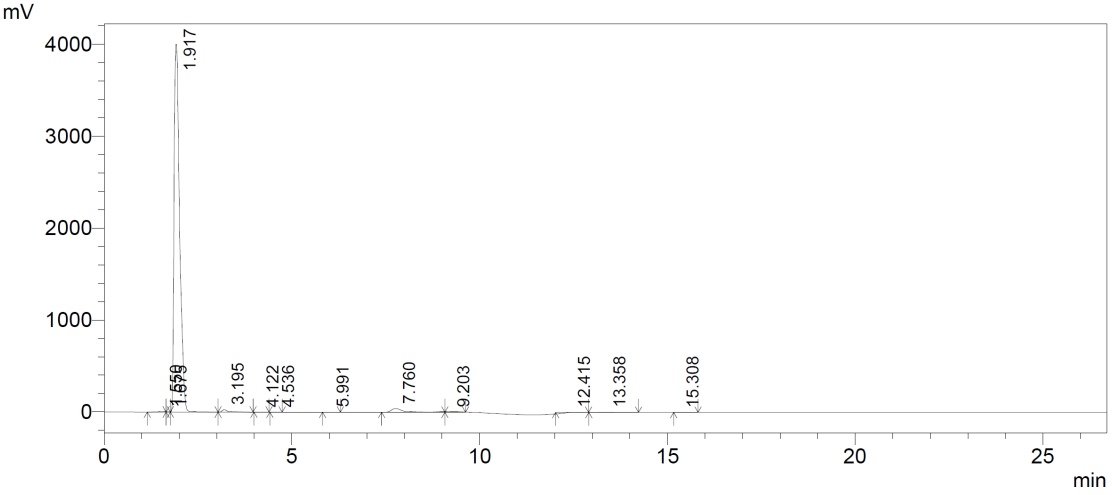
Flow Rate: 1 mL/min;

Oven Temp: 30 °C;

Inj. volume: 5 µL;

Detection: 241 nm

Eluent: 0.1 % phosphoric acid (aq): acetonitrile =60 : 40.




| peak# | retention time | peak area | peak height | area % | height % |
|---|---|---|---|---|---|
| 1 | 1.550 | 50740 | 4217 | 0.121 | 0.103 |
| 2 | 1.675 | 11181 | 1974 | 0.027 | 0.048 |
| 3 | 1.917 | 40823301 | 4001088 | 97.044 | 98.157 |
| 4 | 3.195 | 260487 | 25311 | 0.619 | 0.621 |
| 5 | 4.122 | 6647 | 702 | 0.016 | 0.017 |
| 6 | 4.536 | 2197 | 239 | 0.005 | 0.006 |
| 7 | 5.991 | 5027 | 412 | 0.012 | 0.010 |
| 8 | 7.760 | 874760 | 41125 | 2.079 | 1.009 |
| 9 | 9.203 | 1986 | 111 | 0.005 | 0.003 |
| 10 | 12.415 | 8505 | 352 | 0.020 | 0.009 |
| 11 | 13.358 | 20479 | 621 | 0.049 | 0.015 |
| 12 | 15.308 | 1650 | 64 | 0.004 | 0.002 |
| total | | 42066960 | 4076216 | 100.000 | 100.000 |

**Figure S5**. Chiral HPLC spectrum of **2t**

Column: CHIRALPAK®AD-H (4.6mm×250 mmL, Particle Size5μm)

Flow Rate: 0.6 mL/min;

Oven Temp: 30 °C;

Inj. volume: 1 μL;

Detection: 210 nm

Eluent: hexane: isopropanol = 96 : 4.

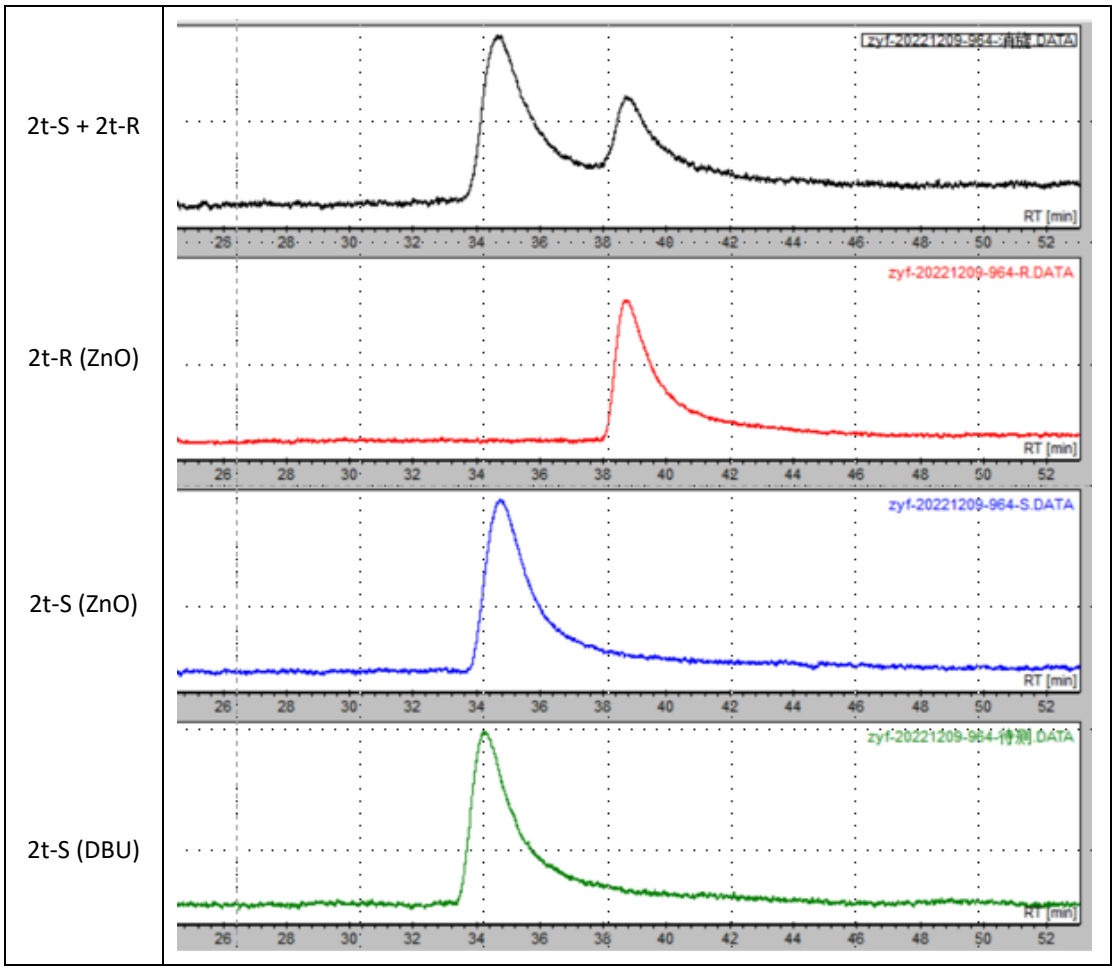
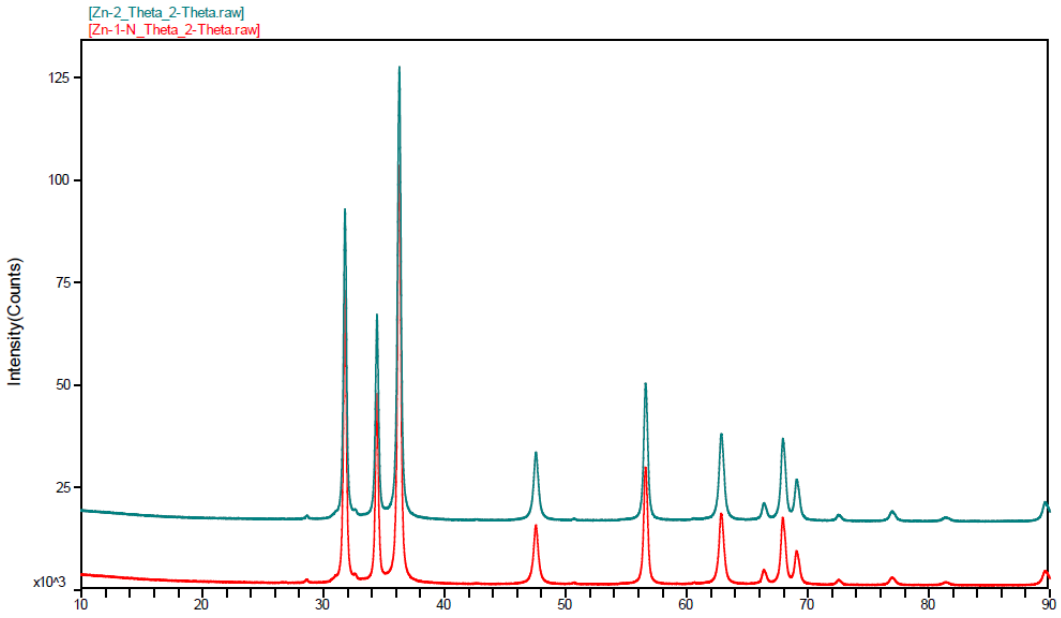
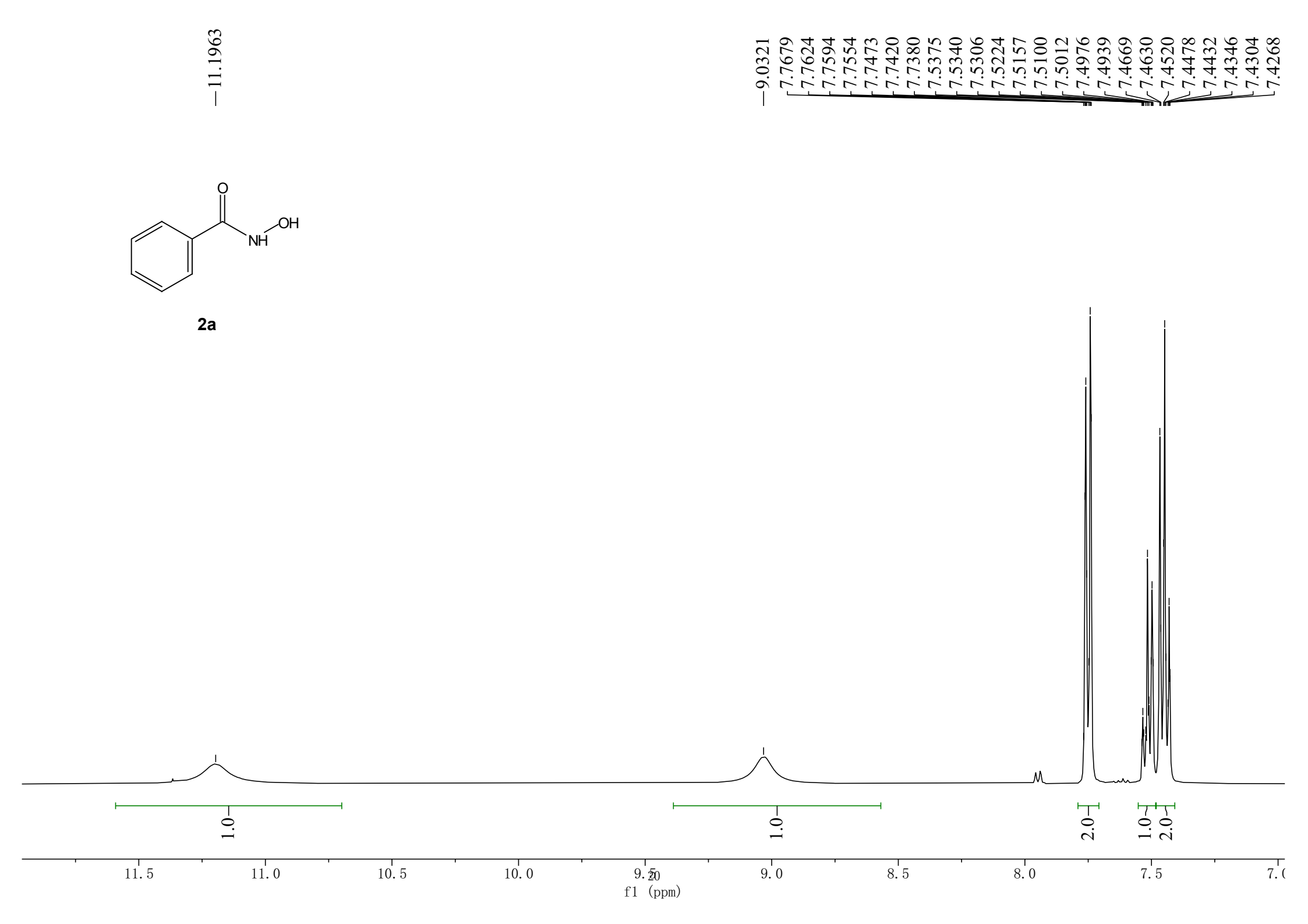
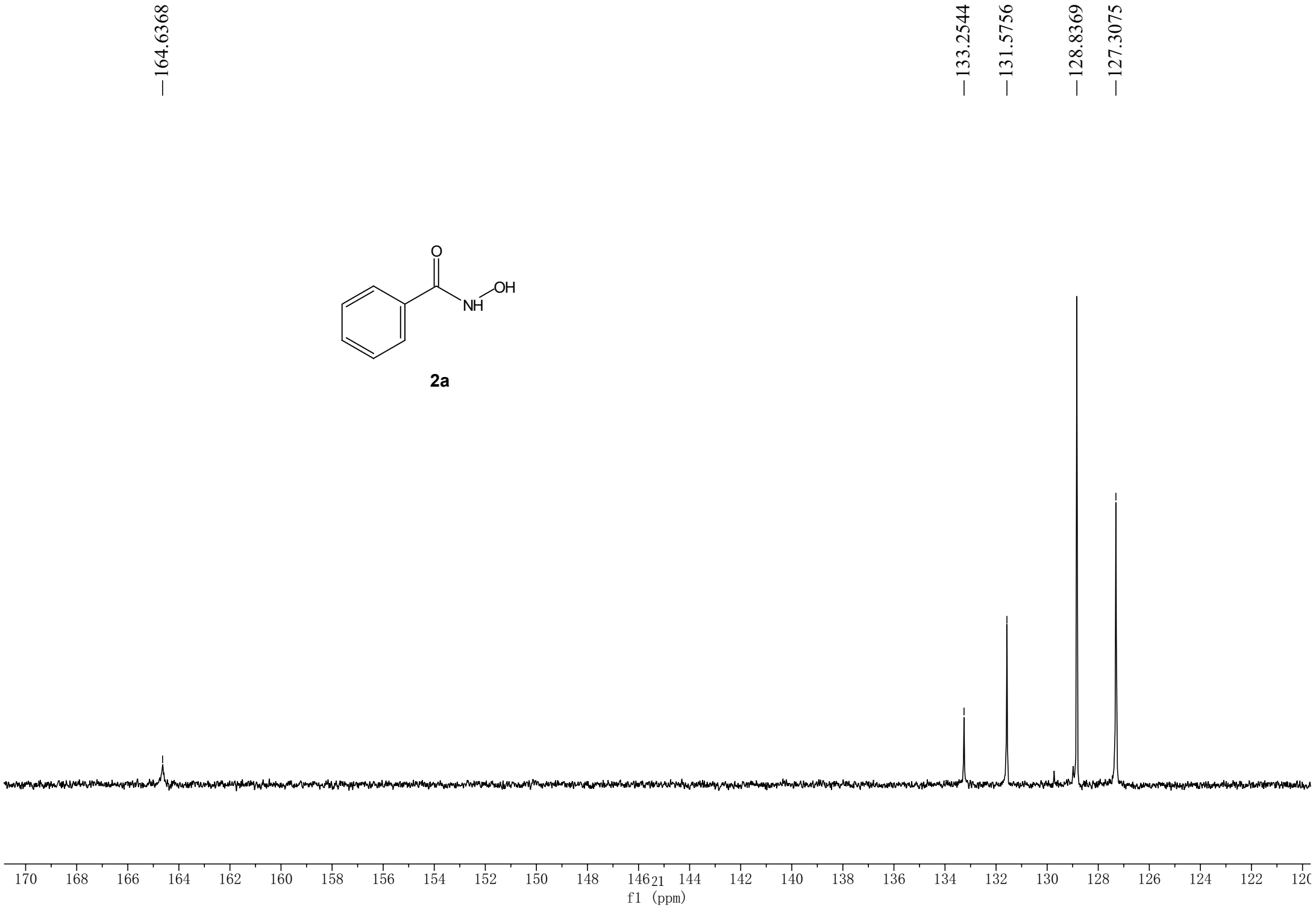


**Figure S6**. XRD spectrum of ZnO

Zn-1-N (Before use); Zn-2 (Recycle)




11.1963
9.0321
7.7679
7.7624
7.7594
7.7554
7.7473
7.7420
7.7380
7.5375
7.5340
7.5306
7.5224
7.5157
7.5100
7.5012
7.4976
7.4939
7.4669
7.4630
7.4520
7.4478
7.4432
7.4346
7.4304
7.4268
O
NH
OH
2a
1.0
1.0
2.0
1.0
2.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
f1 (ppm)

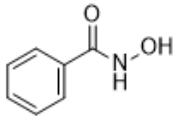

164.6368
133.2544
131.5756
128.8369
127.3075
O
NH
OH
2a
170
168
166
164
162
160
158
156
154
152
150
148
146
144
142
140
138
136
134
132
130
128
126
124
122
120
f1 (ppm)

# Qualitative Analysis Report

| | | | |
|---|---|---|---|
| **Data Filename** | H-0.d | **Sample Name** | H-0 |
| **Sample Type** | Sample | **Position** | P1-F8 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | FU100-1000.m | **Acquired Time** | 9/10/2021 9:05:34 PM |
| **IRM Calibration Status** | Success | **DA Method** | 20170311.m |
| **Comment** | | | |

**Sample Group** **Info.**

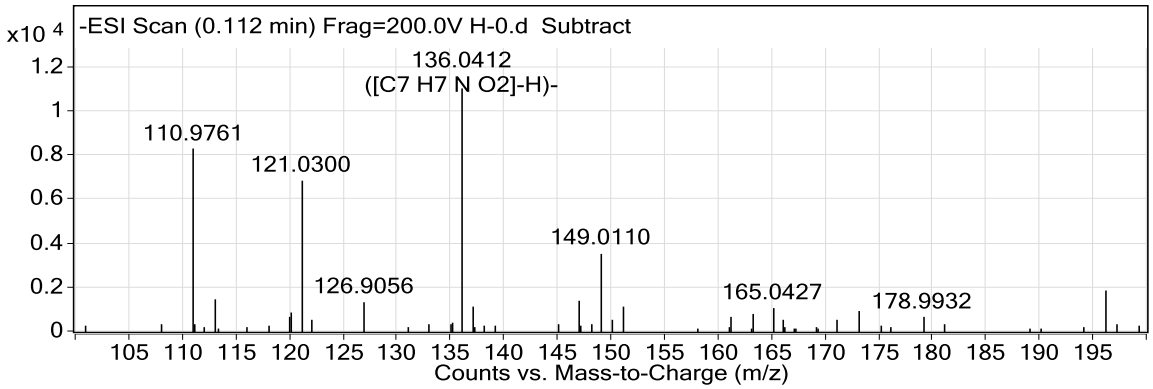
**Acquisition SW Version** 6200 series TOF/6500 series Q-TOF B.05.01 (B5125.2)




**2a**

## User Spectra

| Fragmentor Voltage | Collision Energy | Ionization Mode |
|---|---|---|
| 200 | 0 | ESI |




**Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 110.9761 | | 8326.06 | | |
| 121.03 | 1 | 6906.38 | | |
| 136.0412 | 1 | 11077.74 | C7 H7 N O2 | (M-H)- |
| 223.0301 | 1 | 5681 | | |
| 239.0846 | 1 | 16485.32 | | |
| 283.2657 | 1 | 4745.81 | | |
| 339.2348 | 1 | 30515.69 | | |
| 340.2387 | 1 | 7083.83 | | |
| 423.2053 | 1 | 5390.46 | | |
| 424.2862 | 1 | 4445.02 | | |

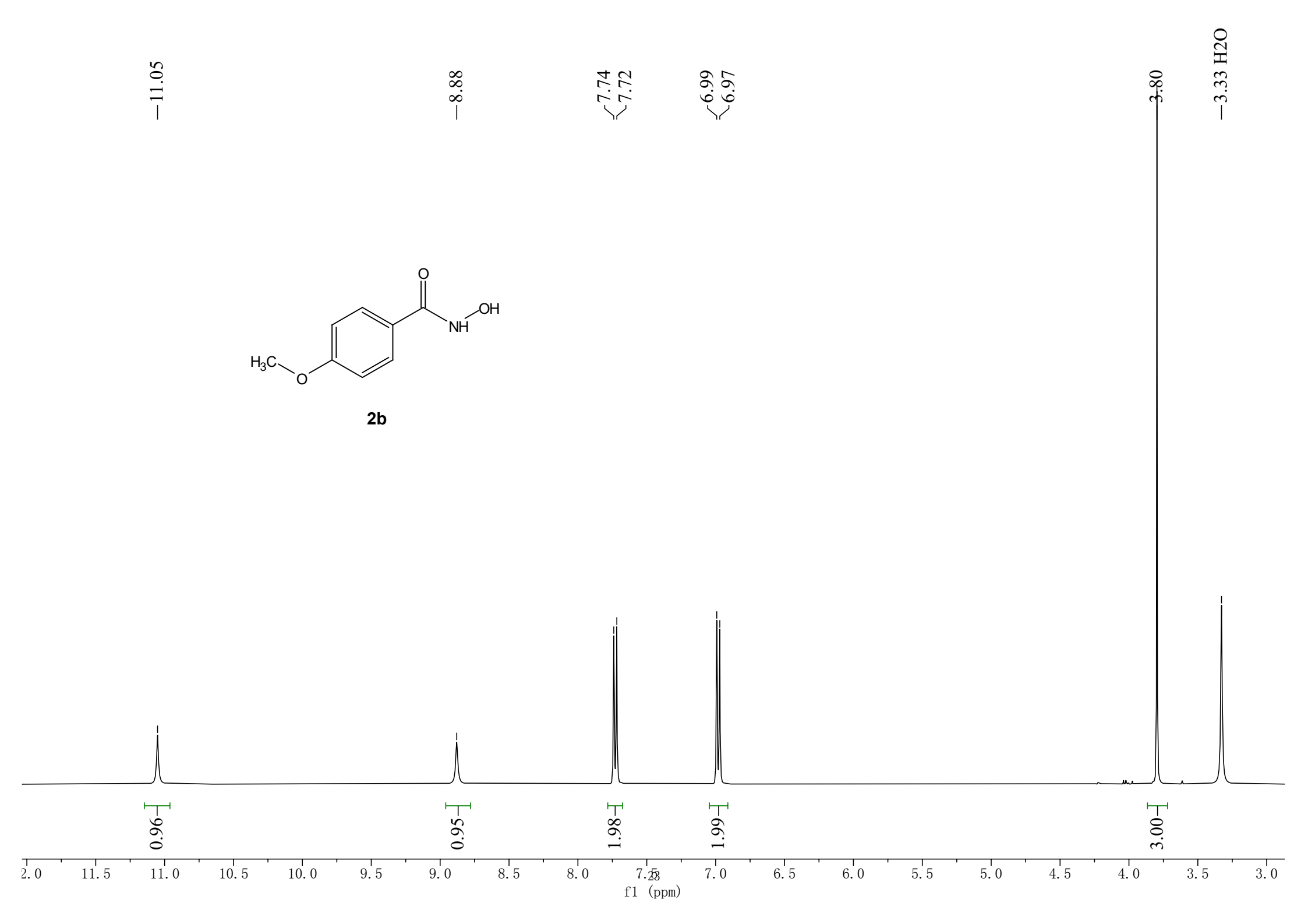
**Formula Calculator Element Limits**

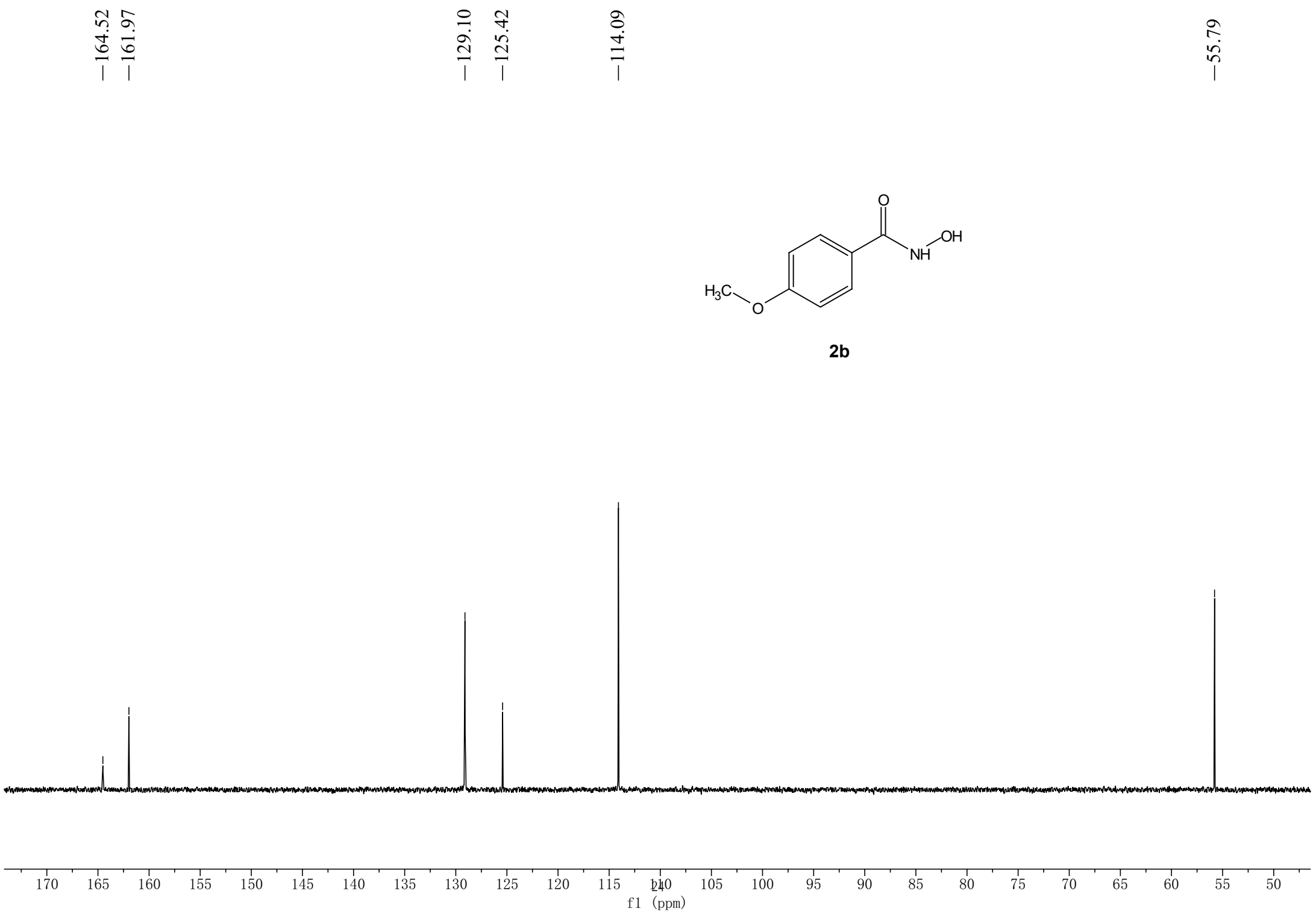
| Element | Min | Max |
|---|---|---|
| C | 3 | 60 |
| H | 0 | 120 |
| O | 0 | 30 |
| N | 0 | 30 |
| Cl | 0 | 0 |
| F | 0 | 0 |
| S | 0 | 0 |
| Br | 0 | 0 |

**Formula Calculator Results**

| Formula | Best | Mass | Tgt Mass | Diff (ppm) | Ion Species | Score |
|---|---|---|---|---|---|---|
| C7 H7 N O2 | TRUE | 137.0484 | 137.0477 | -5.26 | C7 H6 N O2 | 81.43 |

--- End Of Report ---


11.05
8.88
7.74
7.72
6.99
6.97
3.80
3.33 H2O
O
NH
OH
H3C
O
2b
0.96
0.95
1.98
1.99
3.00
f1 (ppm)

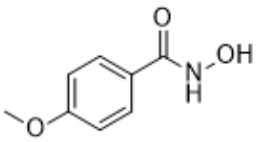

164.52
161.97
129.10
125.42
114.09
55.79
O
NH
OH
H3C
O
2b
170
165
160
155
150
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
55
50
f1 (ppm)

# Qualitative Analysis Report

| | | | |
|---|---|---|---|
| **Data Filename** | H-14.d | **Sample Name** | H-14 |
| **Sample Type** | Sample | **Position** | P1-E7 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | FU100-1000.m | **Acquired Time** | 9/10/2021 8:36:13 PM |
| **IRM Calibration Status** | Success | **DA Method** | 20170311.m |
| **Comment** | | | |

**Sample Group** **Info.**

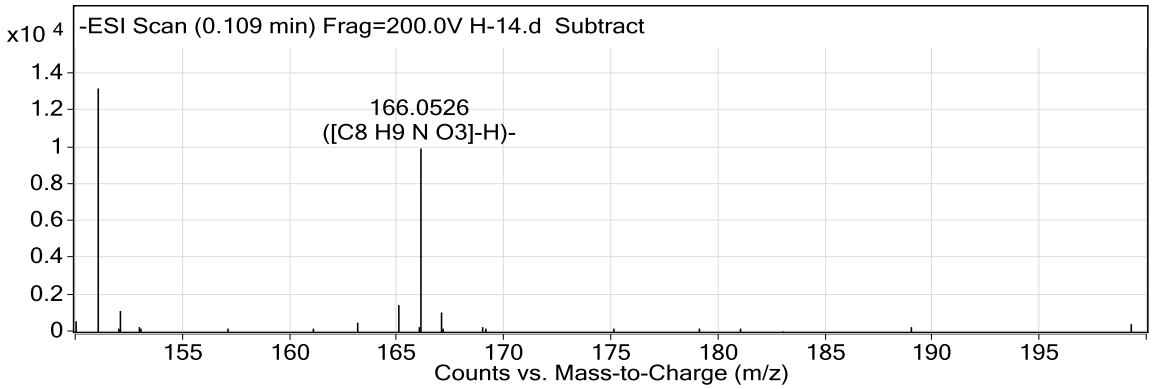
**Acquisition SW Version** 6200 series TOF/6500 series Q-TOF B.05.01 (B5125.2)



**2b**

## User Spectra

| Fragmentor Voltage | Collision Energy | Ionization Mode |
|---|---|---|
| 200 | 0 | ESI |




**Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 107.0384 | | 8023.11 | | |
| 108.0227 | | 7638.1 | | |
| 110.9762 | | 14154.65 | | |
| 121.03 | 1 | 17062.03 | | |
| 151.0294 | 1 | 13241.24 | | |
| 166.0526 | 1 | 9999.13 | C8 H9 N O3 | (M-H)- |
| 283.2657 | 1 | 6949.43 | | |
| 339.2341 | 1 | 31390.84 | | |
| 340.2364 | 1 | 7349.99 | | |
| 459.3275 | 1 | 8567.12 | | |

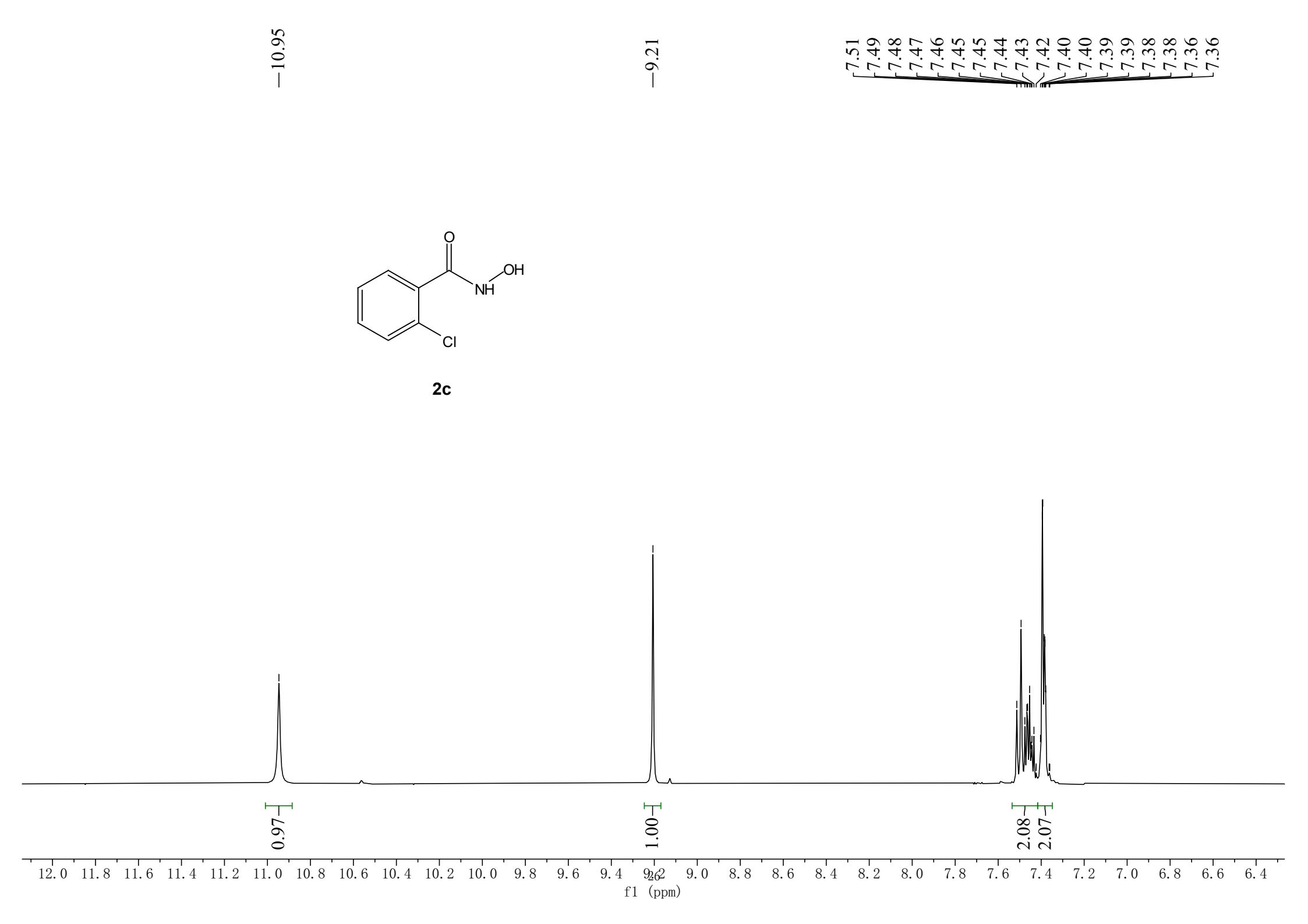
**Formula Calculator Element Limits**

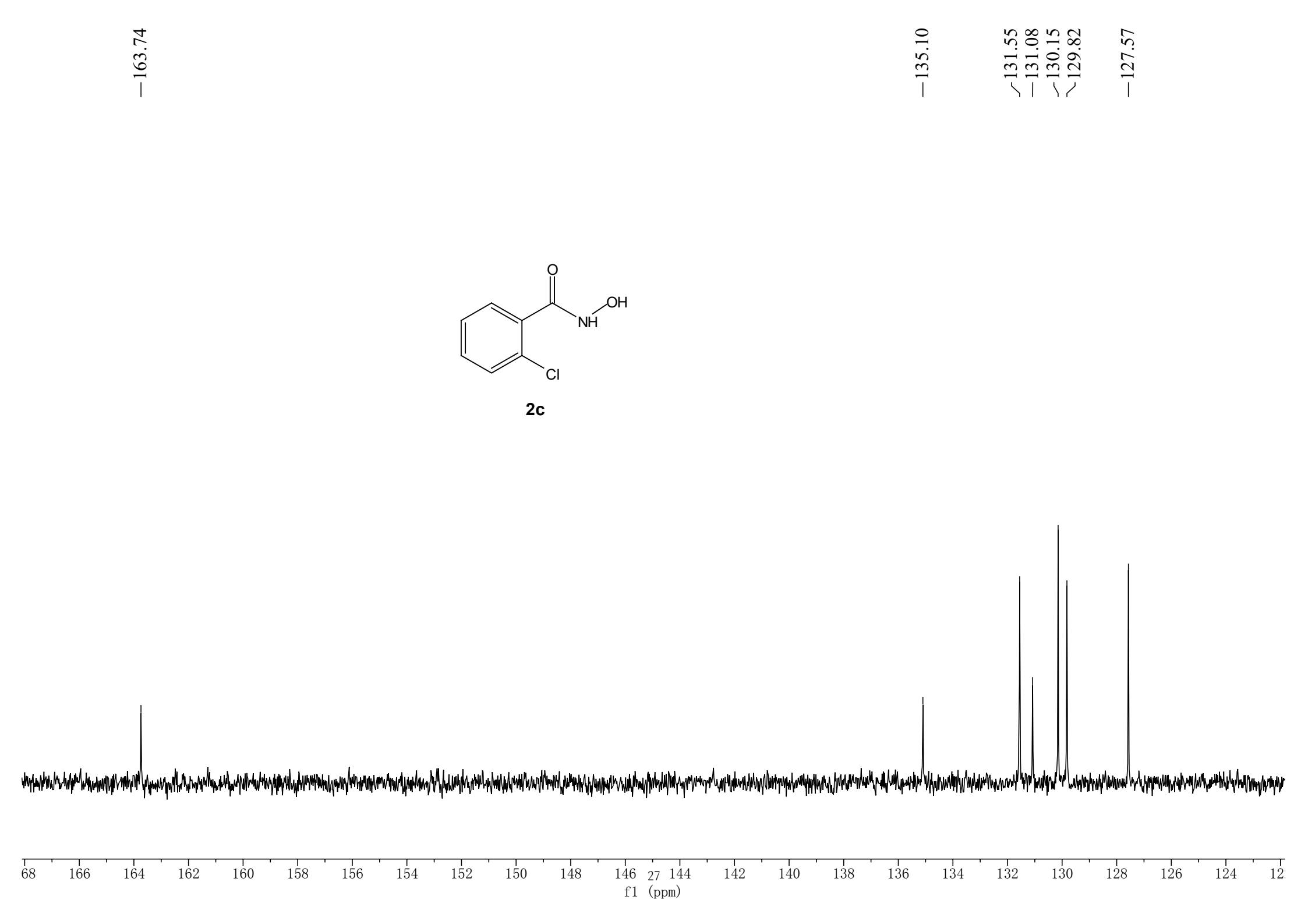
| Element | Min | Max |
|---|---|---|
| C | 3 | 60 |
| H | 0 | 120 |
| O | 0 | 30 |
| N | 0 | 30 |
| Cl | 0 | 0 |
| F | 0 | 0 |
| Br | 0 | 0 |

**Formula Calculator Results**

| Formula | Best | Mass | Tgt Mass | Diff (ppm) | Ion Species | Score |
|---|---|---|---|---|---|---|
| C8 H9 N O3 | TRUE | 167.0597 | 167.0582 | -8.51 | C8 H8 N O3 | 70.75 |

--- End Of Report ---


10.95
9.21
7.51
7.49
7.48
7.47
7.46
7.45
7.45
7.44
7.43
7.42
7.40
7.40
7.39
7.39
7.38
7.38
7.36
7.36
O
NH
OH
Cl
2c
0.97
1.00
2.08
2.07
12.0 11.8 11.6 11.4 11.2 11.0 10.8 10.6 10.4 10.2 10.0 9.8 9.6 9.4 9.2 9.0 8.8 8.6 8.4 8.2 8.0 7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4
f1 (ppm)

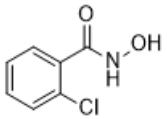

163.74
135.10
131.55
131.08
130.15
129.82
127.57
O
OH
NH
Cl
2c
68
166
164
162
160
158
156
154
152
150
148
146
144
142
140
138
136
134
132
130
128
126
124
f1 (ppm)

# Qualitative Analysis Report

| | | | |
|---|---|---|---|
| **Data Filename** | H-5.d | **Sample Name** | H-5 |
| **Sample Type** | Sample | **Position** | P1-D7 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | FU100-1000.m | **Acquired Time** | 9/10/2021 8:09:46 PM |
| **IRM Calibration Status** | Success | **DA Method** | 20170311.m |
| **Comment** | | | |

**Sample Group** **Info.**

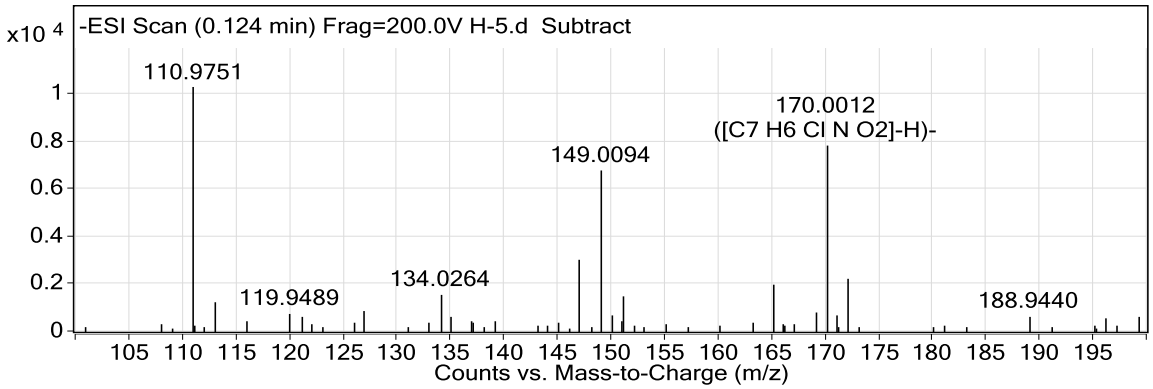
**Acquisition SW Version** 6200 series TOF/6500 series Q-TOF B.05.01 (B5125.2)



**2c**

## User Spectra

| Fragmentor Voltage | Collision Energy | Ionization Mode |
|---|---|---|
| 200 | 0 | ESI |




**Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 110.9751 | | 10296.62 | | |
| 149.0094 | 1 | 6810.69 | | |
| 170.0012 | 1 | 7862.36 | C7 H6 Cl N O2 | (M-H)- |
| 223.0281 | 1 | 9884.63 | | |
| 255.2315 | 1 | 8426.46 | | |
| 283.2633 | 1 | 7885.98 | | |
| 339.232 | 1 | 20531.96 | | |
| 340.2343 | 1 | 5633.36 | | |
| 423.2024 | 1 | 5422.42 | | |
| 486.1969 | 1 | 5105.35 | | |

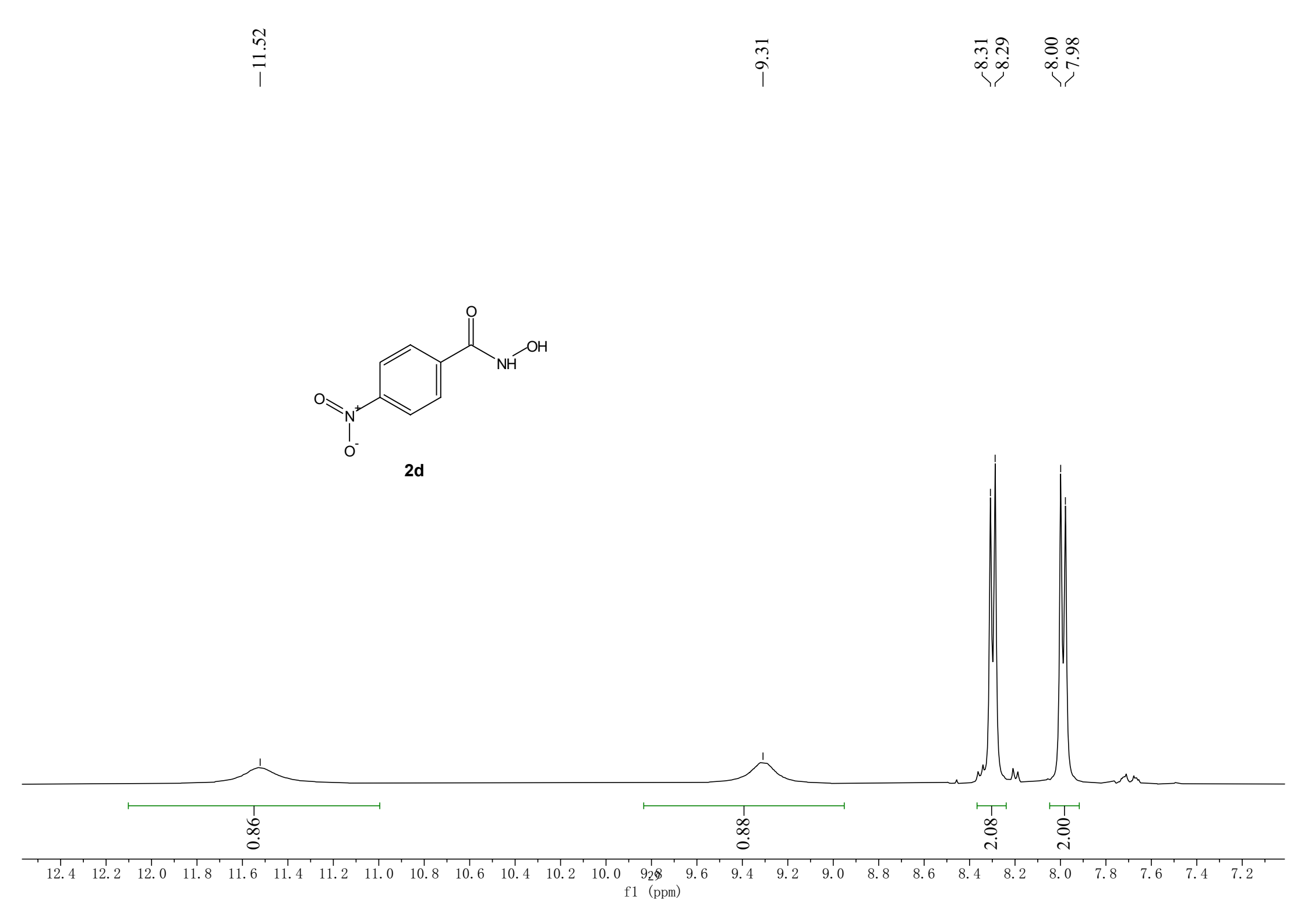
**Formula Calculator Element Limits**

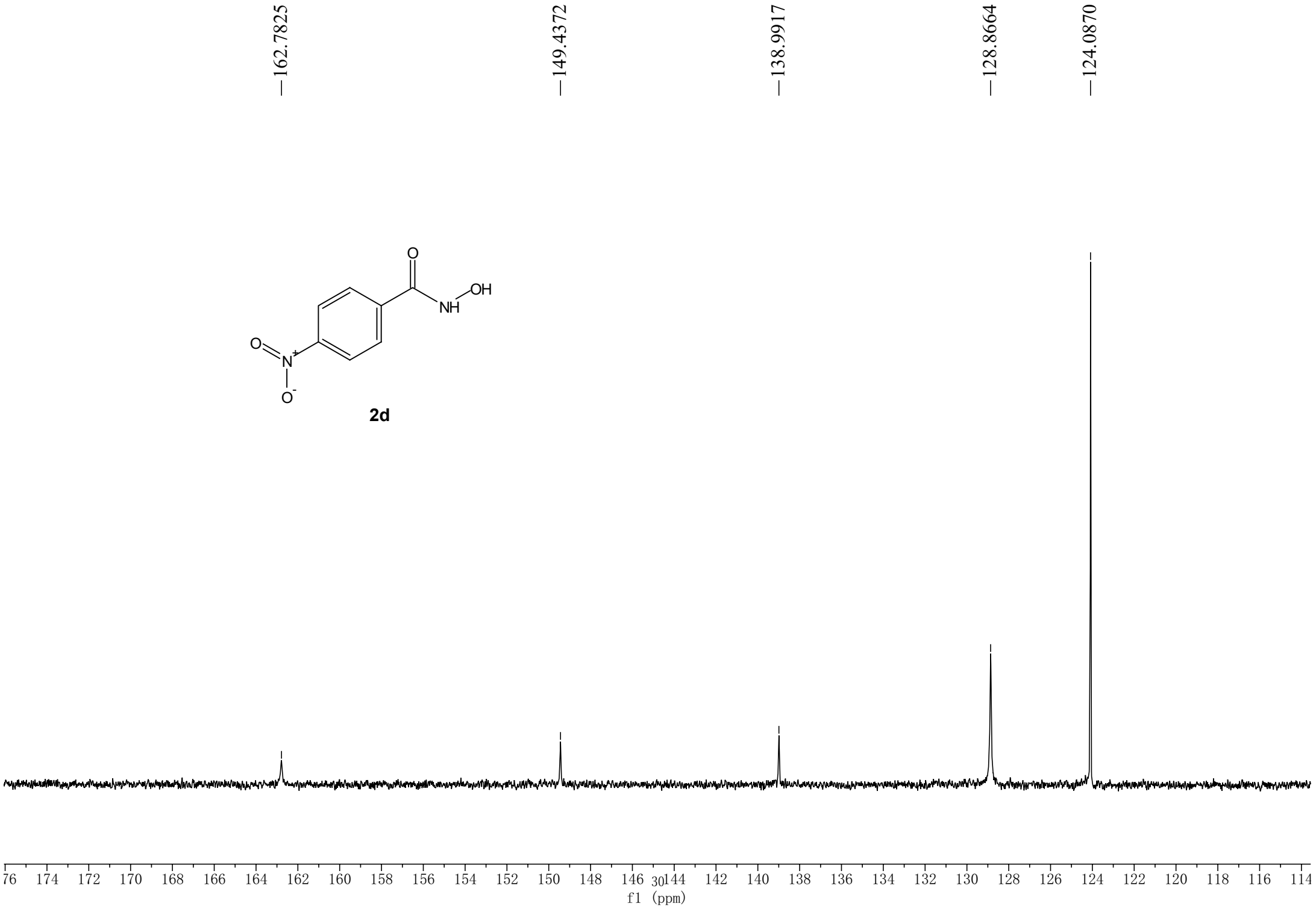
| Element | Min | Max |
|---|---|---|
| C | 3 | 60 |
| H | 0 | 120 |
| O | 0 | 30 |
| N | 0 | 30 |
| Cl | 1 | 1 |
| F | 0 | 0 |
| S | 0 | 0 |
| Br | 0 | 0 |

**Formula Calculator Results**

| Formula | Best | Mass | Tgt Mass | Diff (ppm) | Ion Species | Score |
|---|---|---|---|---|---|---|
| C7 H6 Cl N O2 | TRUE | 171.0084 | 171.0087 | 1.65 | C7 H5 Cl N O2 | 90.59 |

--- End Of Report ---

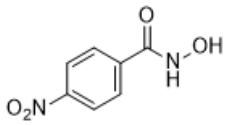

11.52
9.31
8.31
8.29
8.00
7.98
O
NH
OH
O
N+
O-
2d
0.86
0.88
2.08
2.00
12.4 12.2 12.0 11.8 11.6 11.4 11.2 11.0 10.8 10.6 10.4 10.2 10.0 9.8 9.6 9.4 9.2 9.0 8.8 8.6 8.4 8.2 8.0 7.8 7.6 7.4 7.2
f1 (ppm)


162.7825
149.4372
138.9917
128.8664
124.0870
O
NH
OH
O
N+
O-
2d
76 174 172 170 168 166 164 162 160 158 156 154 152 150 148 146 144 142 140 138 136 134 132 130 128 126 124 122 120 118 116 114
f1 (ppm)

# Qualitative Analysis Report

| | | | |
|---|---|---|---|
| **Data Filename** | H-3.d | **Sample Name** | H-3 |
| **Sample Type** | Sample | **Position** | P1-D5 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | FU100-1000.m | **Acquired Time** | 9/10/2021 8:03:49 PM |
| **IRM Calibration Status** | Success | **DA Method** | 20170311.m |
| **Comment** | | | |

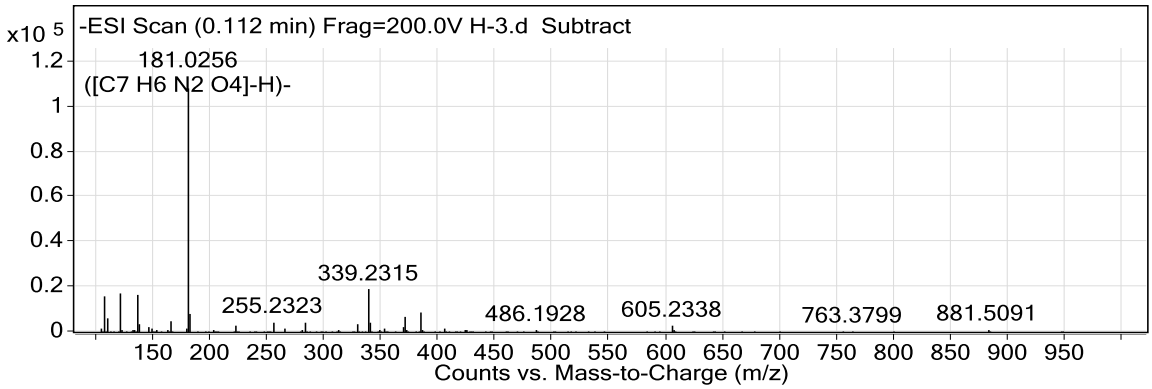
| | | | |
|---|---|---|---|
| **Sample Group** | | **Info.** | |
| **Acquisition SW Version** | 6200 series TOF/6500 series Q-TOF B.05.01 (B5125.2) | | |




**2d**

## User Spectra

| Fragmentor Voltage | Collision Energy | Ionization Mode |
|---|---|---|
| 200 | 0 | ESI |




**Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 107.0374 | | 16507.06 | | |
| 110.9754 | | 6768.81 | | |
| 122.0242 | 1 | 17808.98 | | |
| 137.0352 | | 16820.79 | | |
| 166.0154 | 1 | 4909.4 | | |
| 181.0256 | 1 | 108351.77 | C7 H6 N2 O4 | (M-H)- |
| 182.0285 | 1 | 8616.11 | C7 H6 N2 O4 | (M-H)- |
| 339.2315 | 1 | 19257.53 | | |
| 371.0425 | 1 | 7411.56 | | |
| 385.0388 | 1 | 9017.39 | | |

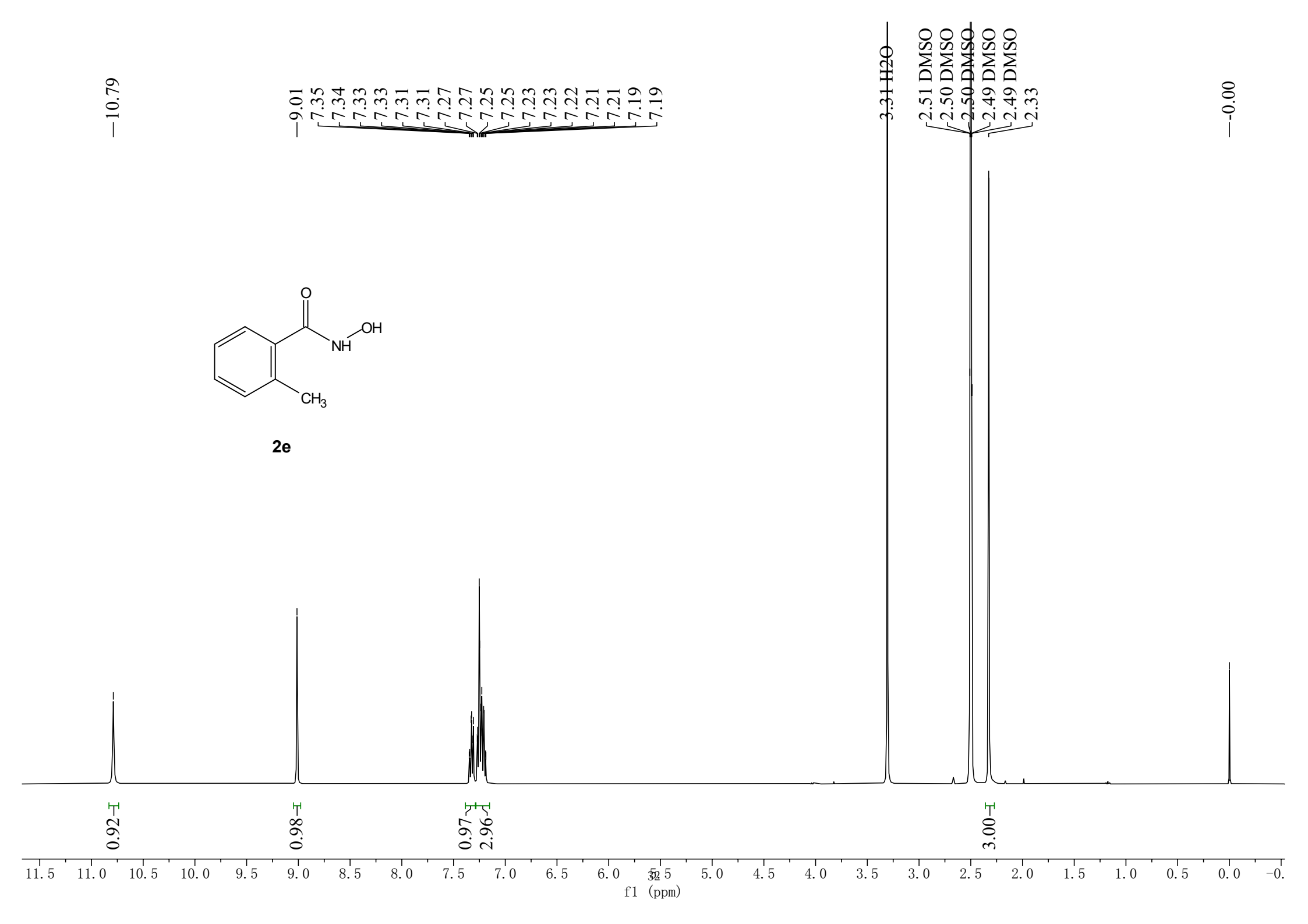
**Formula Calculator Element Limits**

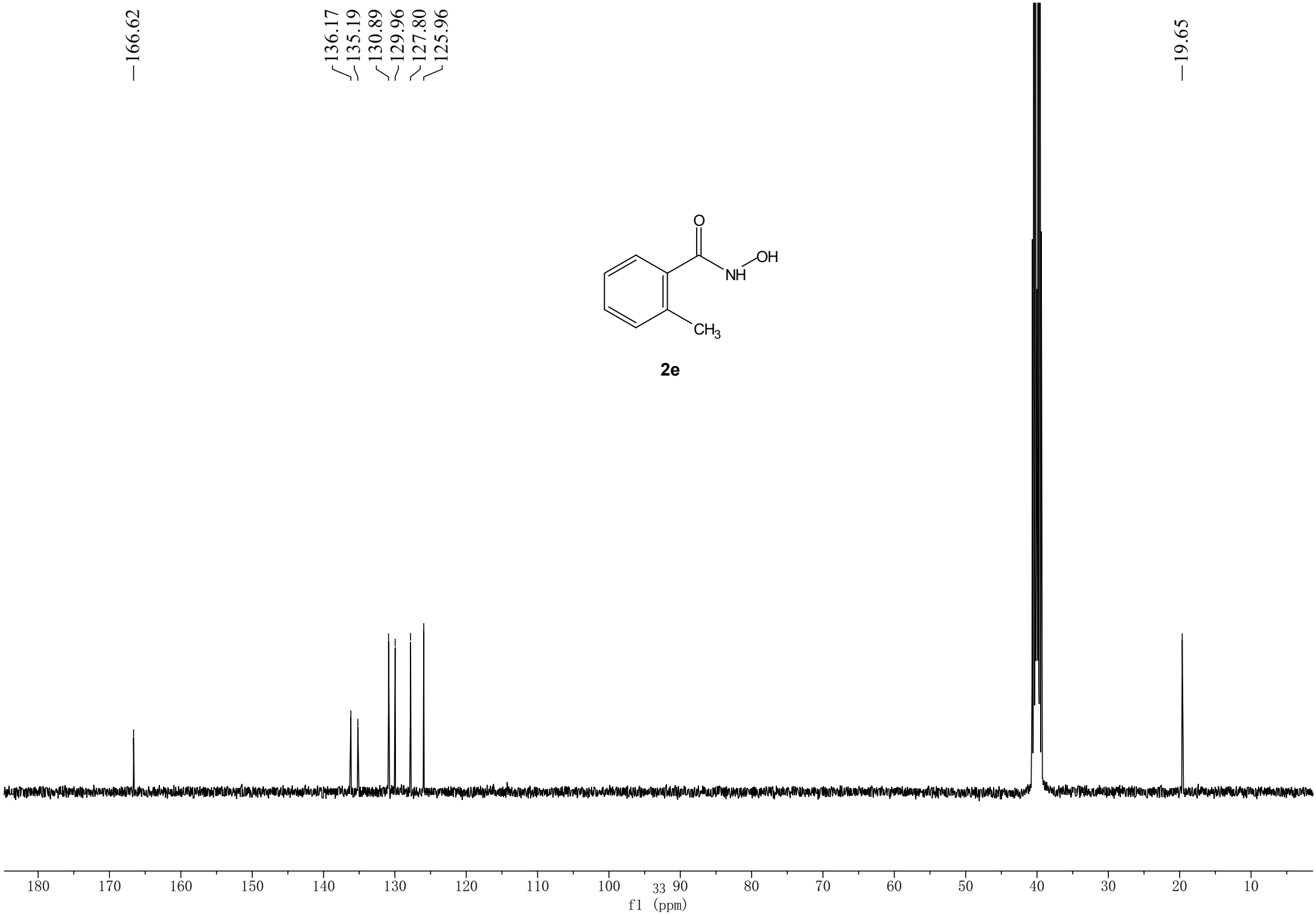
| Element | Min | Max |
|---|---|---|
| C | 3 | 60 |
| H | 0 | 120 |
| O | 0 | 30 |
| N | 0 | 30 |
| Cl | 0 | 0 |
| F | 0 | 0 |
| S | 0 | 0 |
| Br | 0 | 0 |

**Formula Calculator Results**

| Formula | Best | Mass | Tgt Mass | Diff (ppm) | Ion Species | Score |
|---|---|---|---|---|---|---|
| C7 H6 N2 O4 | TRUE | 182.0329 | 182.0328 | -0.79 | C7 H5 N2 O4 | 99.78 |

--- End Of Report ---


10.79
9.01
7.35
7.34
7.33
7.33
7.31
7.31
7.27
7.27
7.25
7.25
7.23
7.23
7.22
7.21
7.21
7.19
7.19
3.31 H2O
2.51 DMSO
2.50 DMSO
2.50 DMSO
2.49 DMSO
2.49 DMSO
2.33
-0.00
O
NH
OH
CH3
2e
0.92
0.98
0.97
2.96
3.00
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)

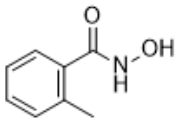

166.62
136.17
135.19
130.89
129.96
127.80
125.96
19.65
O
NH
OH
CH3
2e
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

# Qualitative Analysis Report

| | | | |
|---|---|---|---|
| **Data Filename** | H-2.d | **Sample Name** | H-2 |
| **Sample Type** | Sample | **Position** | P1-D4 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | FU100-1000.m | **Acquired Time** | 9/10/2021 8:00:53 PM |
| **IRM Calibration Status** | Success | **DA Method** | 20170311.m |
| **Comment** | | | |

**Sample Group** **Info.**

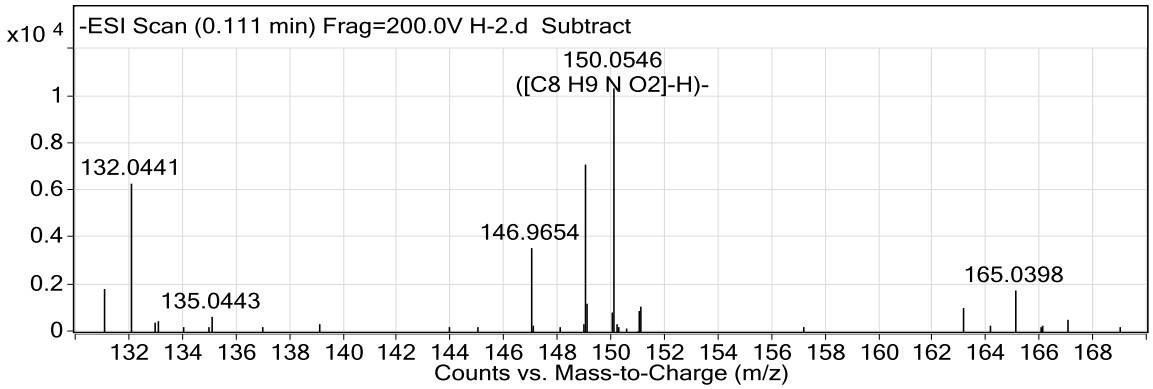
**Acquisition SW Version** 6200 series TOF/6500 series Q-TOF B.05.01 (B5125.2)




**2e**

## User Spectra

| Fragmentor Voltage | Collision Energy | Ionization Mode |
|---|---|---|
| 200 | 0 | ESI |




**Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 110.975 | | 8981.68 | | |
| 132.0441 | 1 | 6324.56 | | |
| 149.0093 | 1 | 7136.8 | | |
| 150.0546 | 1 | 10366.55 | C8 H9 N O2 | (M-H)- |
| 223.0276 | 1 | 12277.13 | | |
| 255.2311 | 1 | 10070.78 | | |
| 283.2625 | 1 | 11510.82 | | |
| 339.2305 | 1 | 71667.88 | | |
| 340.2335 | 1 | 16580.31 | | |
| 423.2006 | 1 | 5804.8 | | |

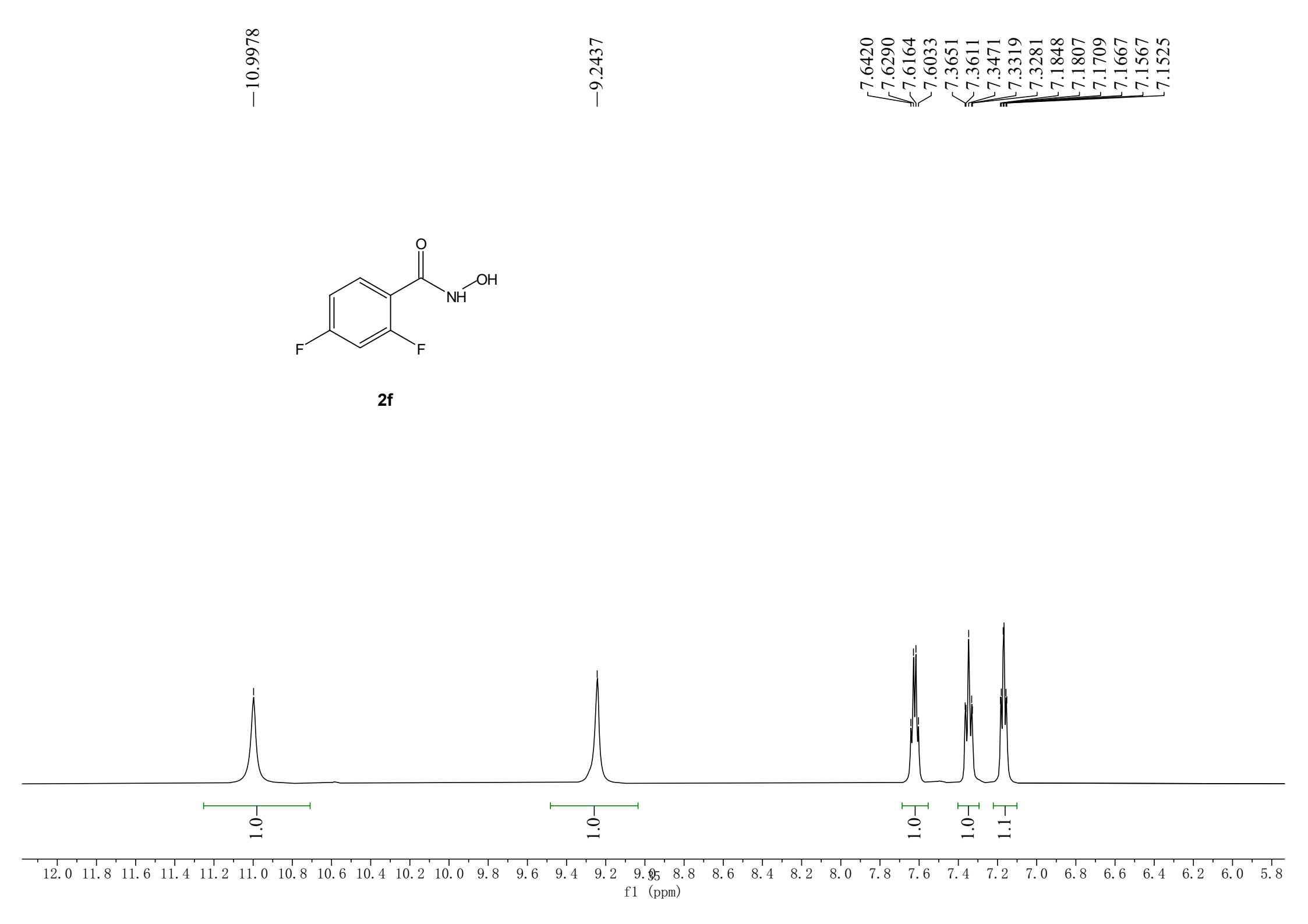
**Formula Calculator Element Limits**

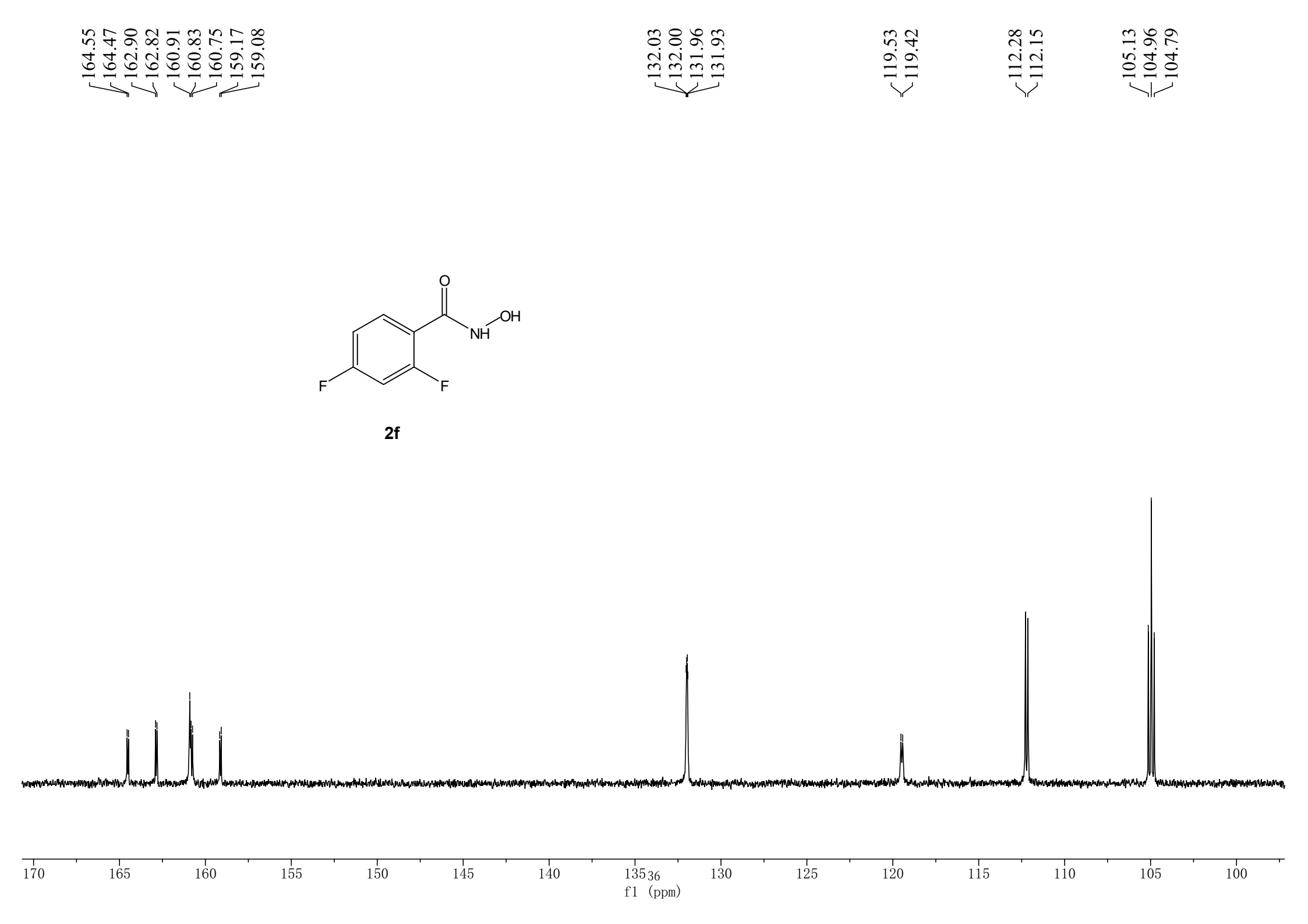
| Element | Min | Max |
|---|---|---|
| C | 3 | 60 |
| H | 0 | 120 |
| O | 0 | 30 |
| N | 0 | 30 |
| Cl | 0 | 0 |
| F | 0 | 0 |
| S | 0 | 0 |
| Br | 0 | 0 |

**Formula Calculator Results**

| Formula | Best | Mass | Tgt Mass | Diff (ppm) | Ion Species | Score |
|---|---|---|---|---|---|---|
| C8 H9 N O2 | TRUE | 151.0618 | 151.0633 | 9.96 | C8 H8 N O2 | 74.98 |

--- End Of Report ---


10.9978
9.2437
7.6420
7.6290
7.6164
7.6033
7.3651
7.3611
7.3471
7.3319
7.3281
7.1848
7.1807
7.1709
7.1667
7.1567
7.1525
O
NH
OH
F
F
2f
1.0
1.0
1.0
1.0
1.1
12.0 11.8 11.6 11.4 11.2 11.0 10.8 10.6 10.4 10.2 10.0 9.8 9.6 9.4 9.2 9.0 8.8 8.6 8.4 8.2 8.0 7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8
f1 (ppm)

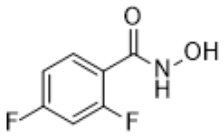

164.55
164.47
162.90
162.82
160.91
160.83
160.75
159.17
159.08
132.03
132.00
131.96
131.93
119.53
119.42
112.28
112.15
105.13
104.96
104.79
O
NH
OH
F
F
2f
170
165
160
155
150
145
140
135
130
125
120
115
110
105
100
f1 (ppm)

# Qualitative Analysis Report

| | | | |
|---|---|---|---|
| **Data Filename** | H-7.d | **Sample Name** | H-7 |
| **Sample Type** | Sample | **Position** | P1-D9 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | FU100-1000.m | **Acquired Time** | 9/10/2021 8:15:39 PM |
| **IRM Calibration Status** | Success | **DA Method** | 20170311.m |
| **Comment** | | | |

**Sample Group** **Info.**

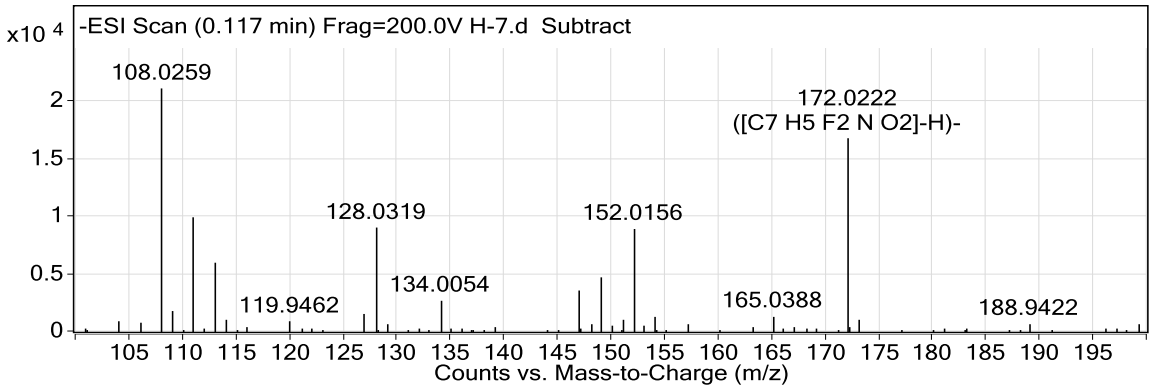
**Acquisition SW Version** 6200 series TOF/6500 series Q-TOF B.05.01 (B5125.2)




**2f**

## User Spectra

| Fragmentor Voltage | Collision Energy | Ionization Mode |
|---|---|---|
| 200 | 0 | ESI |




**Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 108.0259 | 1 | 21215.55 | | |
| 110.976 | | 10067.43 | | |
| 128.0319 | 1 | 9111.13 | | |
| 152.0156 | 1 | 9030.54 | | |
| 172.0222 | 1 | 16847.09 | C7 H5 F2 N O2 | (M-H)- |
| 223.0294 | 1 | 7639.98 | | |
| 255.2331 | 1 | 7922.67 | | |
| 283.2647 | 1 | 7155.33 | | |
| 339.2336 | 1 | 18010.52 | | |
| 423.2043 | 1 | 7402.89 | | |

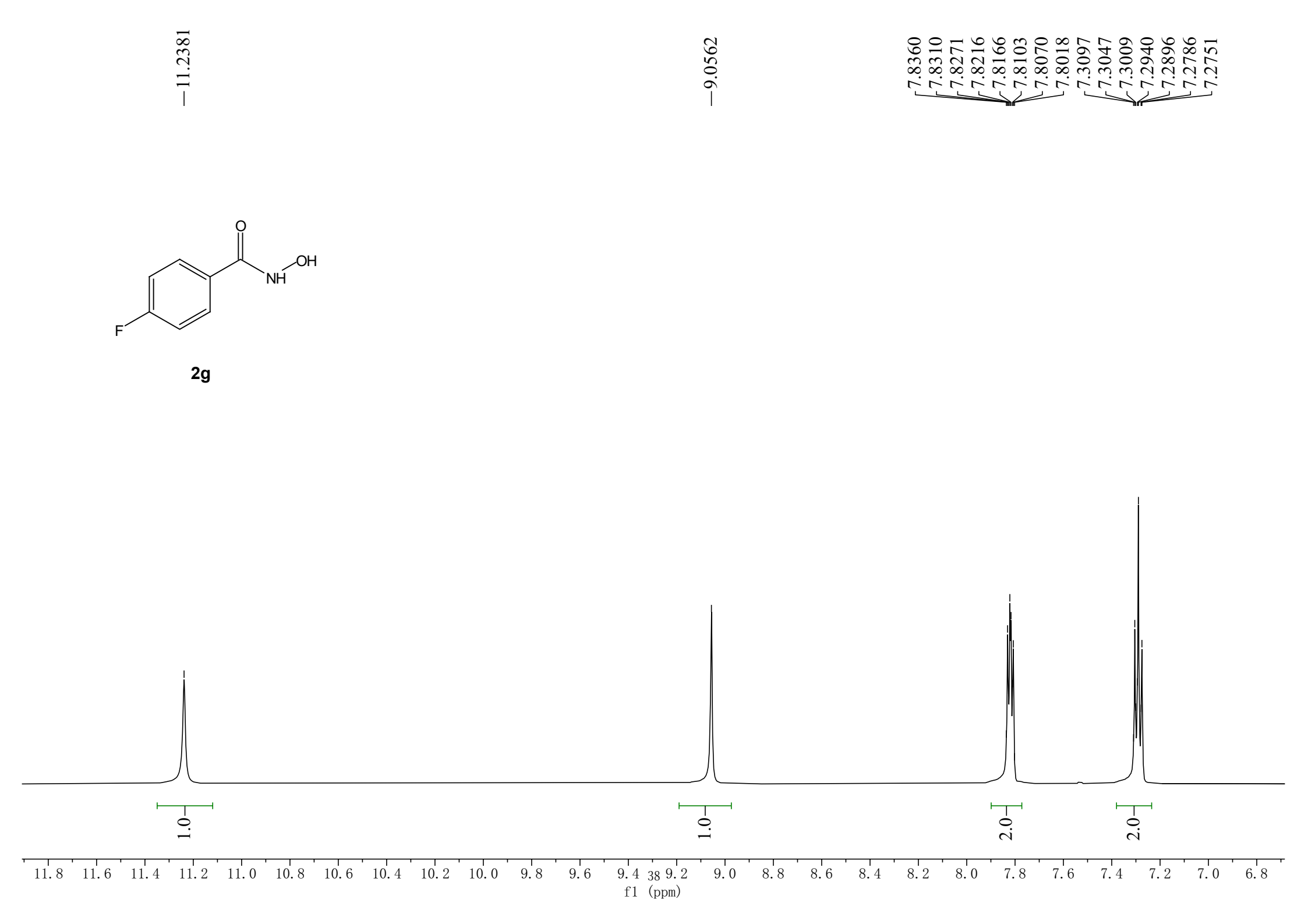
**Formula Calculator Element Limits**

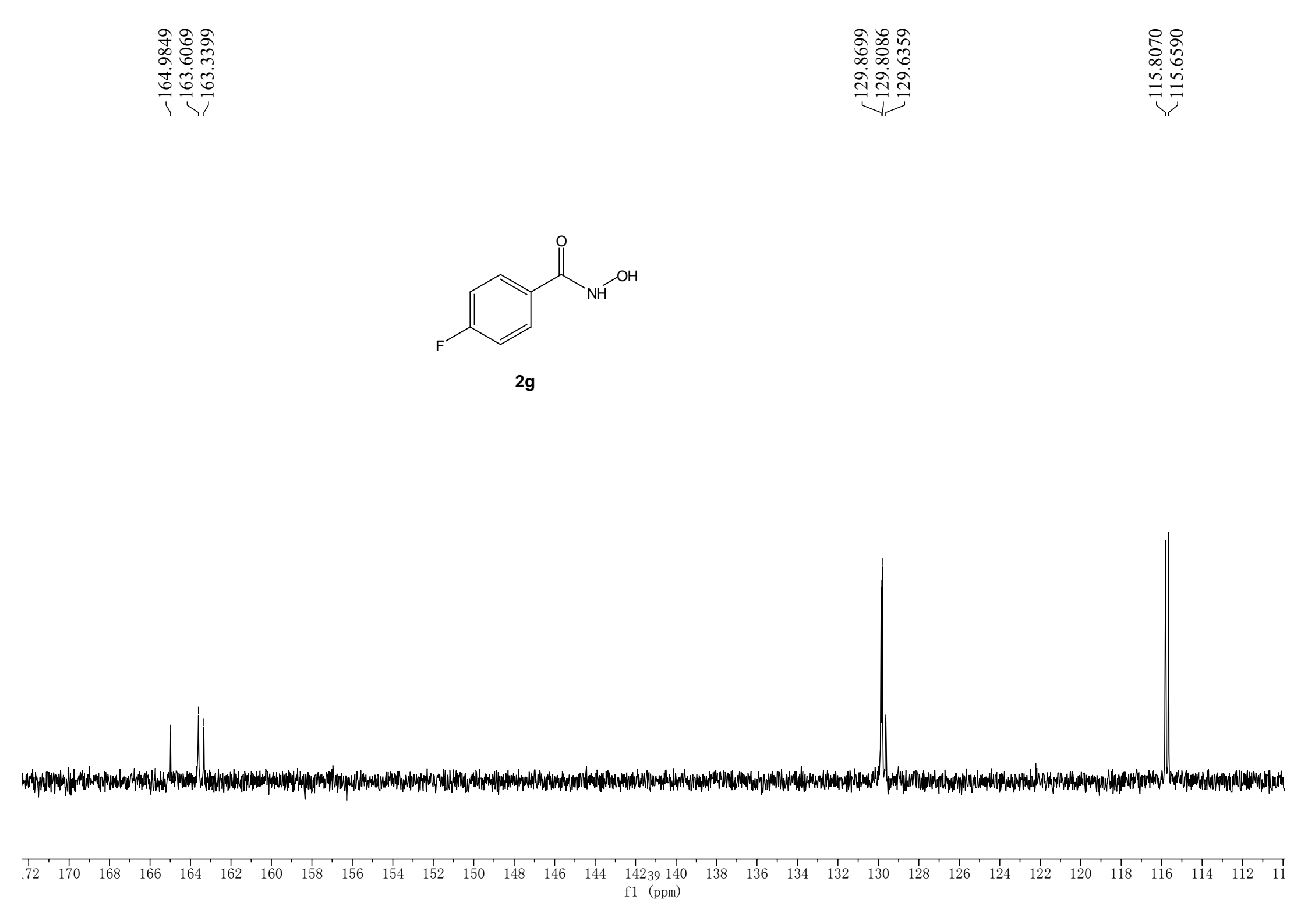
| Element | Min | Max |
|---|---|---|
| C | 3 | 60 |
| H | 0 | 120 |
| O | 0 | 30 |
| N | 0 | 30 |
| Cl | 0 | 0 |
| F | 2 | 2 |
| S | 0 | 0 |
| Br | 0 | 0 |

**Formula Calculator Results**

| Formula | Best | Mass | Tgt Mass | Diff (ppm) | Ion Species | Score |
|---|---|---|---|---|---|---|
| C7 H5 F2 N O2 | TRUE | 173.0295 | 173.0288 | -3.73 | C7 H4 F2 N O2 | 84.43 |

--- End Of Report ---


11.2381
9.0562
7.8360
7.8310
7.8271
7.8216
7.8166
7.8103
7.8070
7.8018
7.3097
7.3047
7.3009
7.2940
7.2896
7.2786
7.2751
O
NH
OH
F
2g
1.0
1.0
2.0
2.0
11.8 11.6 11.4 11.2 11.0 10.8 10.6 10.4 10.2 10.0 9.8 9.6 9.4 9.2 9.0 8.8 8.6 8.4 8.2 8.0 7.8 7.6 7.4 7.2 7.0 6.8
f1 (ppm)

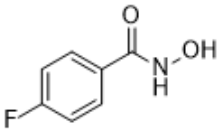

164.9849
163.6069
163.3399
129.8699
129.8086
129.6359
115.8070
115.6590
O
NH
OH
F
2g
172 170 168 166 164 162 160 158 156 154 152 150 148 146 144 142 140 138 136 134 132 130 128 126 124 122 120 118 116 114 112 11
f1 (ppm)

# Qualitative Analysis Report

| | | | |
|---|---|---|---|
| **Data Filename** | H-26.d | **Sample Name** | H-26 |
| **Sample Type** | Sample | **Position** | P1-C4 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | FU100-1000.m | **Acquired Time** | 9/24/2021 8:26:28 PM |
| **IRM Calibration Status** | Success | **DA Method** | 20170311.m |
| **Comment** | | | |

**Sample Group** **Info.**

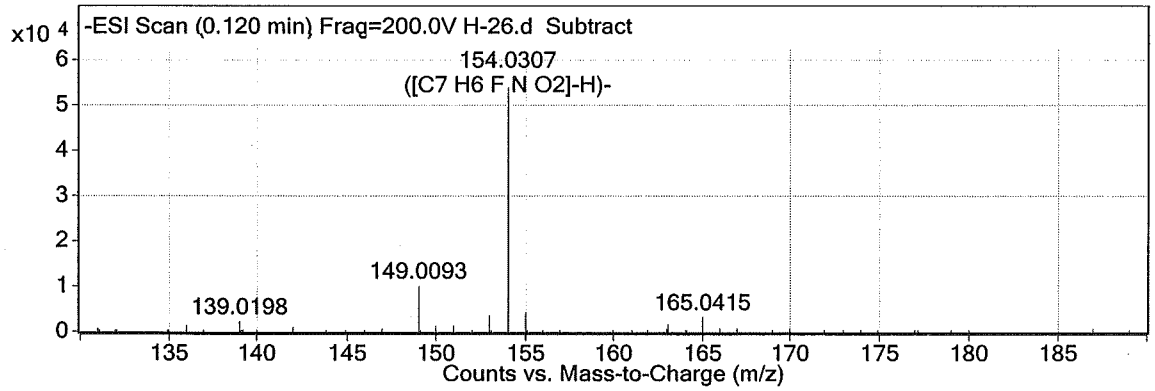
**Acquisition SW Version** 6200 series TOF/6500 series Q-TOF B.05.01 (B5125.2)




2g

## User Spectra

| Fragmentor Voltage | Collision Energy | Ionization Mode |
|---|---|---|
| 200 | 0 | ESI |




**Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 110.0404 | 1 | 27017.95 | | |
| 149.0093 | 1 | 10130.84 | | |
| 154.0307 | 1 | 53809.39 | C7 H6 F N O2 | (M-H)- |
| 223.0277 | 1 | 19997.59 | | |
| 225.0116 | 1 | 7004.46 | | |
| 339.231 | 1 | 76293.7 | | |
| 340.2341 | 1 | 17598.61 | | |
| 353.2092 | 1 | 7985.72 | | |
| 423.2011 | 1 | 8479.23 | | |
| 424.2834 | 1 | 16759 | | |

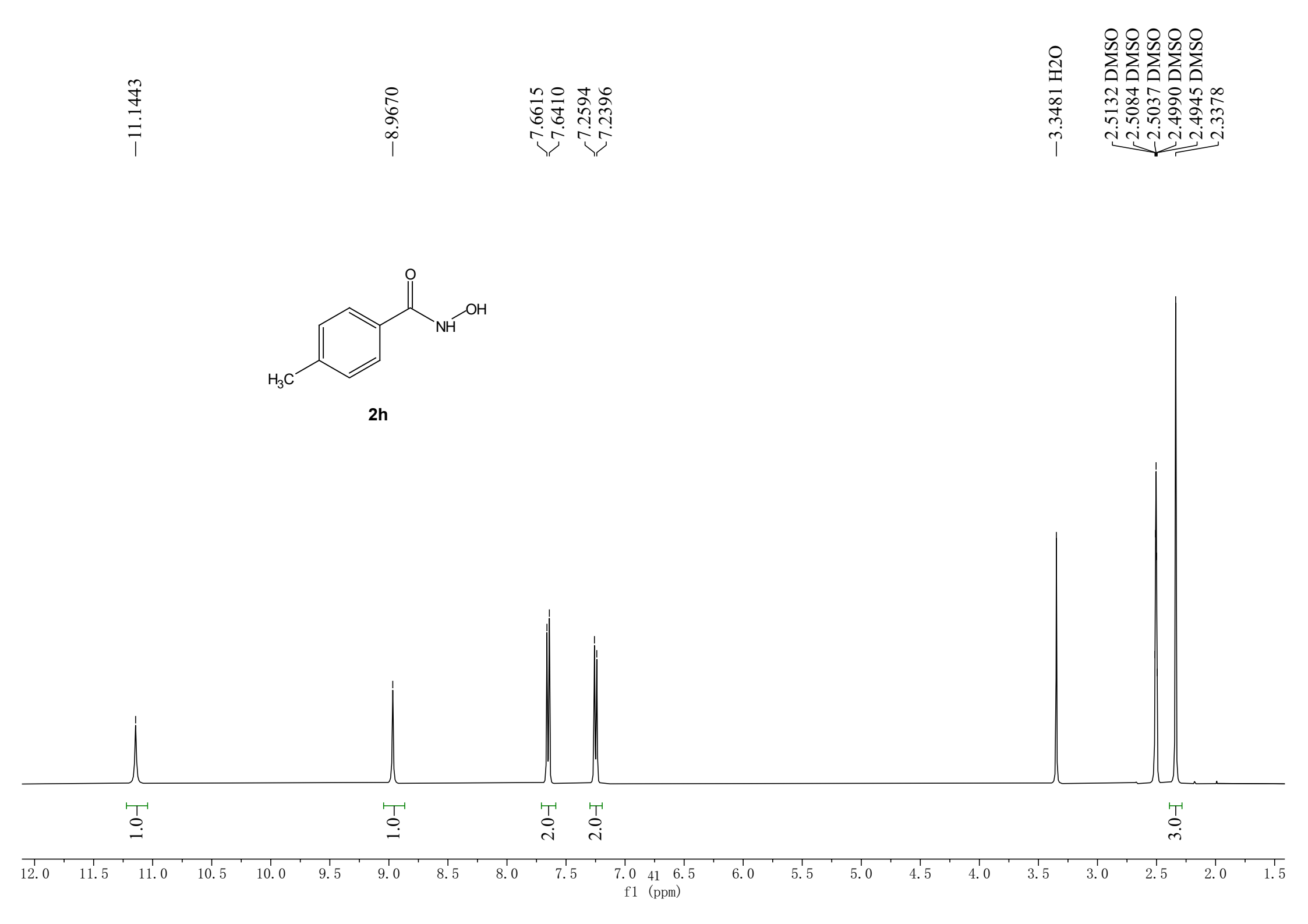
**Formula Calculator Element Limits**

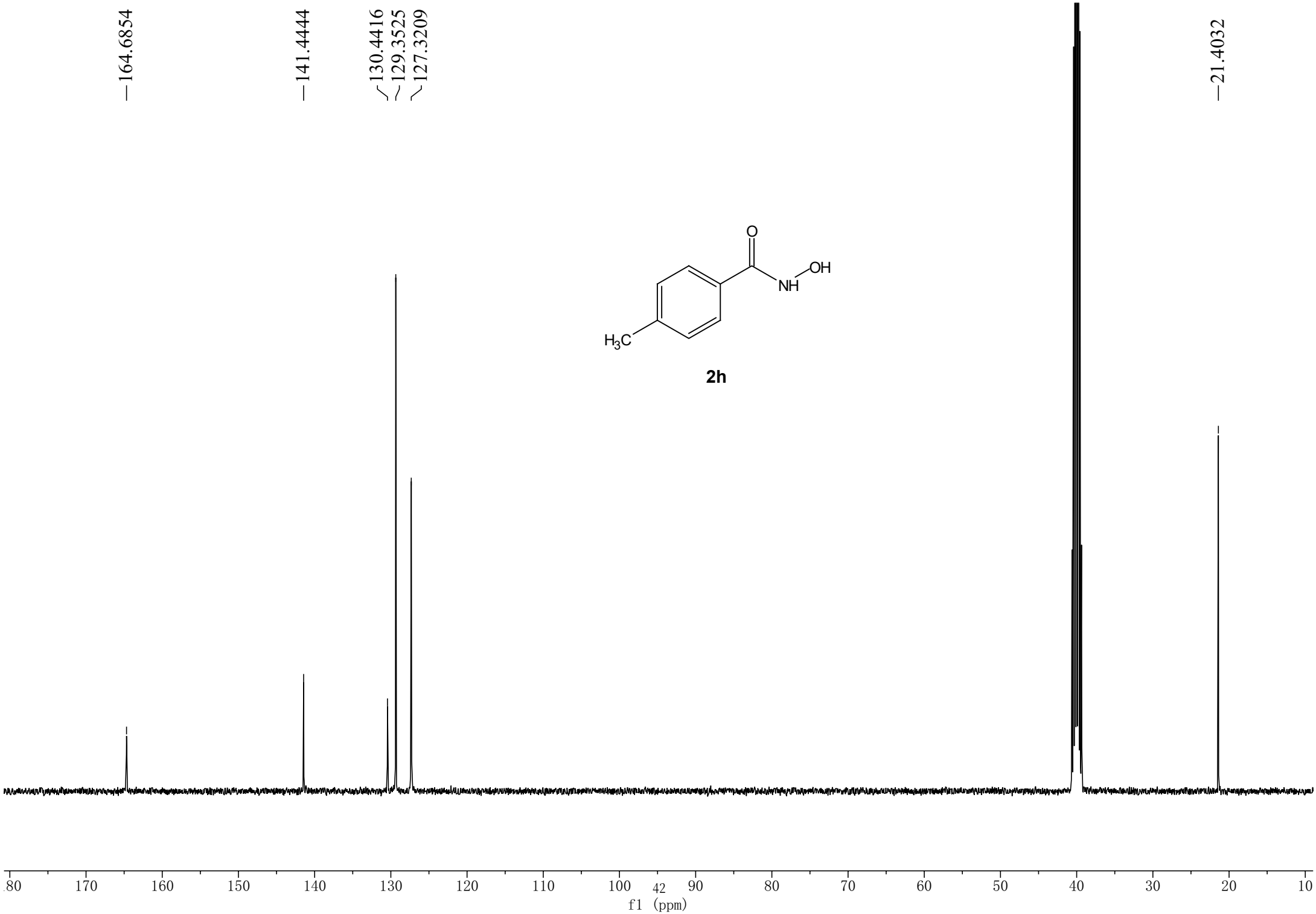
| Element | Min | Max |
|---|---|---|
| C | 3 | 60 |
| H | 0 | 120 |
| O | 0 | 30 |
| N | 0 | 30 |
| F | 1 | 1 |

**Formula Calculator Results**

| Formula | Best | Mass | Tgt Mass | Diff (ppm) | Ion Species | Score |
|---|---|---|---|---|---|---|
| C7 H6 F N O2 | TRUE | 155.0379 | 155.0383 | 2.24 | C7 H5 F N O2 | 98.6 |

--- End Of Report ---


11.1443
8.9670
7.6615
7.6410
7.2594
7.2396
3.3481 H2O
2.5132 DMSO
2.5084 DMSO
2.5037 DMSO
2.4990 DMSO
2.4945 DMSO
2.3378
O
NH
OH
H3C
2h
1.0
1.0
2.0
2.0
3.0
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5
f1 (ppm)

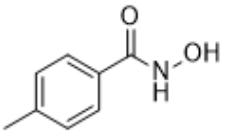

164.6854
141.4444
130.4416
129.3525
127.3209
21.4032
O
NH
OH
H3C
2h
80
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

# Qualitative Analysis Report

| | | | |
|---|---|---|---|
| **Data Filename** | H-27.d | **Sample Name** | H-27 |
| **Sample Type** | Sample | **Position** | P1-B1 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | FU100-1000.m | **Acquired Time** | 1/6/2022 10:06:48 AM |
| **IRM Calibration Status** | Success | **DA Method** | 20170311.m |
| **Comment** | | | |

**Sample Group** **Info.**

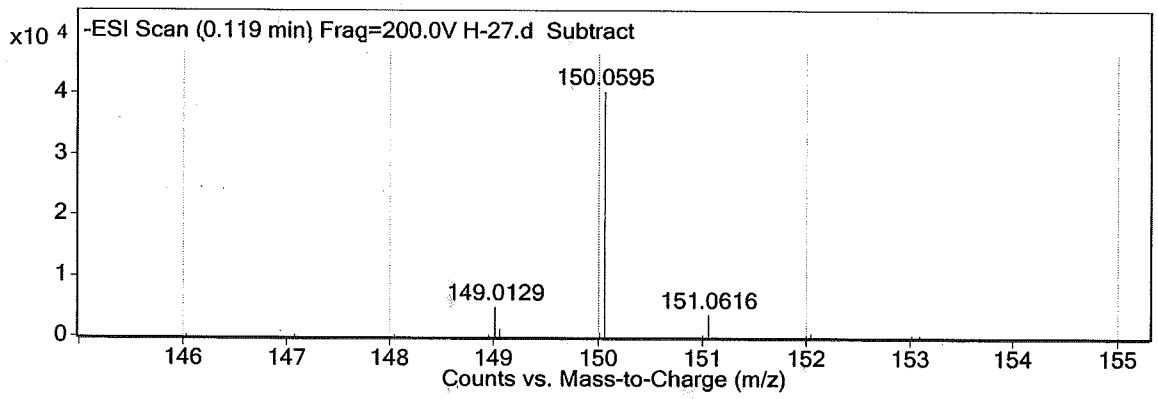
**Acquisition SW Version** 6200 series TOF/6500 series Q-TOF B.05.01 (B5125.2)




2h

## User Spectra

| Fragmentor Voltage | Collision Energy | Ionization Mode |
|---|---|---|
| 200 | 0 | ESI |



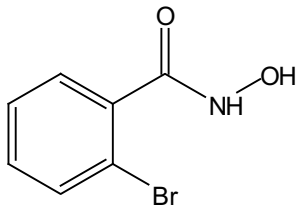

**Peak List**

| *m/z* | z | Abund |
|---|---|---|
| 106.0686 | 1 | 32083.81 |
| 150.0595 | 1 | 40359.3 |
| 339.2373 | 1 | 1012222 |
| 340.2406 | 1 | 238964.38 |
| 341.243 | 1 | 28280.03 |
| 353.2163 | 1 | 46349.3 |
| 369.2475 | 1 | 40906.86 |
| 415.2356 | 1 | 28253.72 |
| 424.2903 | 1 | 237418.89 |
| 425.2929 | 1 | 63363.42 |

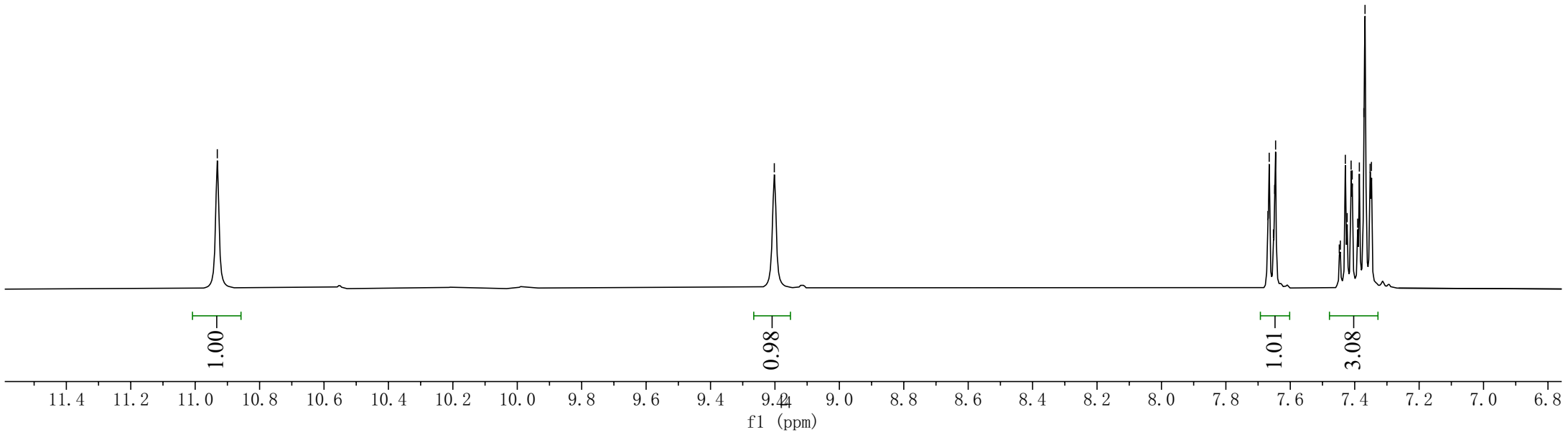
**Formula Calculator Element Limits**

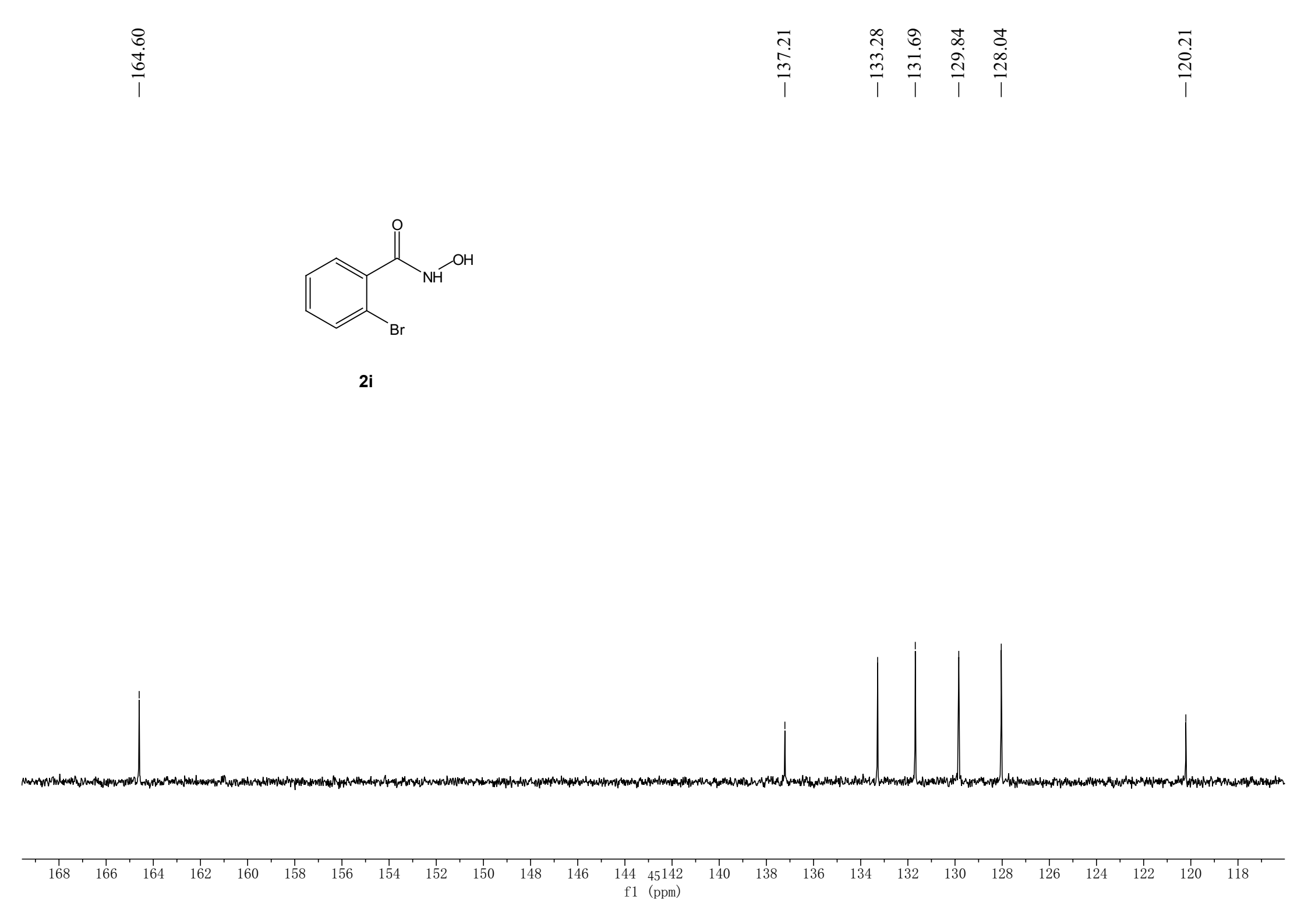
| Element | Min | Max |
|---|---|---|
| C | 3 | 60 |
| H | 0 | 120 |
| O | 0 | 30 |
| N | 0 | 30 |
| Br | 1 | 1 |

--- End Of Report ---


O
NH
OH
Br

**2i**


10.93
9.20
7.67
7.67
7.65
7.65
7.65
7.43
7.42
7.41
7.41
7.39
7.37
7.37
7.35
1.00
0.98
1.01
3.08
11.4
11.2
11.0
10.8
10.6
10.4
10.2
10.0
9.8
9.6
9.4
9.2
9.0
8.8
8.6
8.4
8.2
8.0
7.8
7.6
7.4
7.2
7.0
6.8
f1 (ppm)

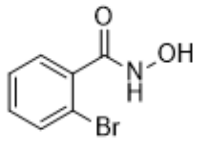

164.60
137.21
133.28
131.69
129.84
128.04
120.21
O
NH
OH
Br
2i
168
166
164
162
160
158
156
154
152
150
148
146
144
142
140
138
136
134
132
130
128
126
124
122
120
118
f1 (ppm)

# Qualitative Analysis Report

| | | | |
|---|---|---|---|
| **Data Filename** | H-15.d | **Sample Name** | H-15 |
| **Sample Type** | Sample | **Position** | P1-E8 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | FU100-1000.m | **Acquired Time** | 9/10/2021 8:39:09 PM |
| **IRM Calibration Status** | Success | **DA Method** | 20170311.m |
| **Comment** | | | |

**Sample Group** **Info.**

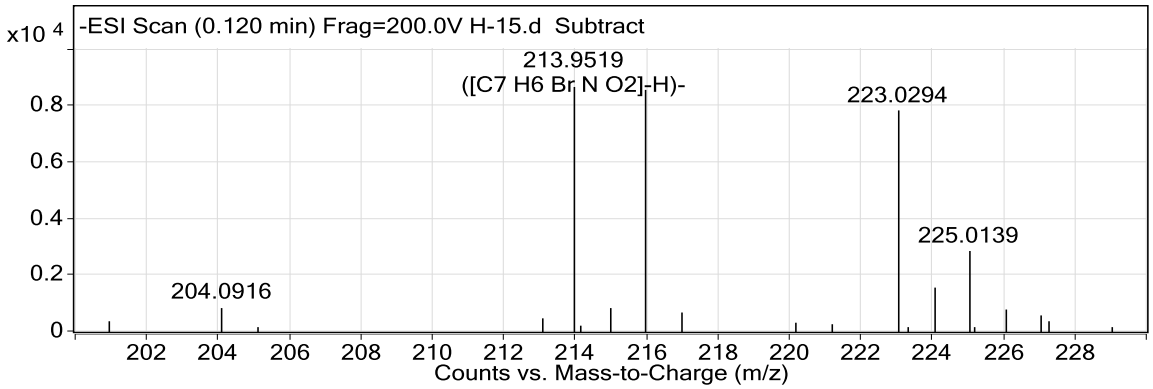
**Acquisition SW Version** 6200 series TOF/6500 series Q-TOF B.05.01 (B5125.2)




**2i**

## User Spectra

| Fragmentor Voltage | Collision Energy | Ionization Mode |
|---|---|---|
| 200 | 0 | ESI |




**Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 110.9762 | | 8395.81 | | |
| 213.9519 | 1 | 8680.39 | C7 H6 Br N O2 | (M-H)- |
| 215.95 | 1 | 8580.45 | C7 H6 Br N O2 | (M-H)- |
| 223.0294 | 1 | 7856.88 | | |
| 255.234 | 1 | 9644.34 | | |
| 283.2653 | 1 | 10117.29 | | |
| 339.234 | 1 | 80781.76 | | |
| 340.2366 | 1 | 19066.68 | | |
| 424.284 | 1 | 5476.01 | | |
| 459.3255 | 1 | 6645.28 | | |

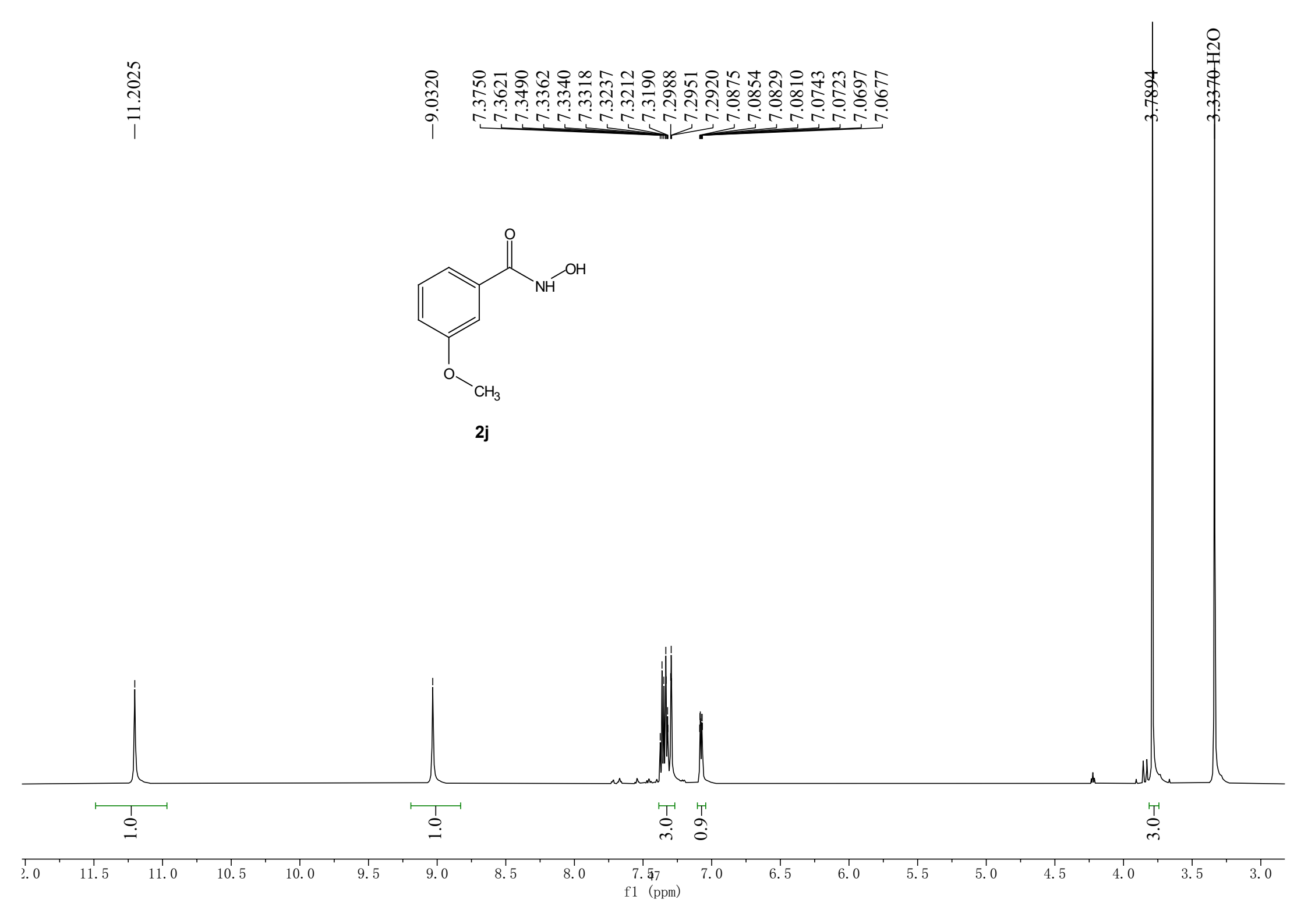
**Formula Calculator Element Limits**

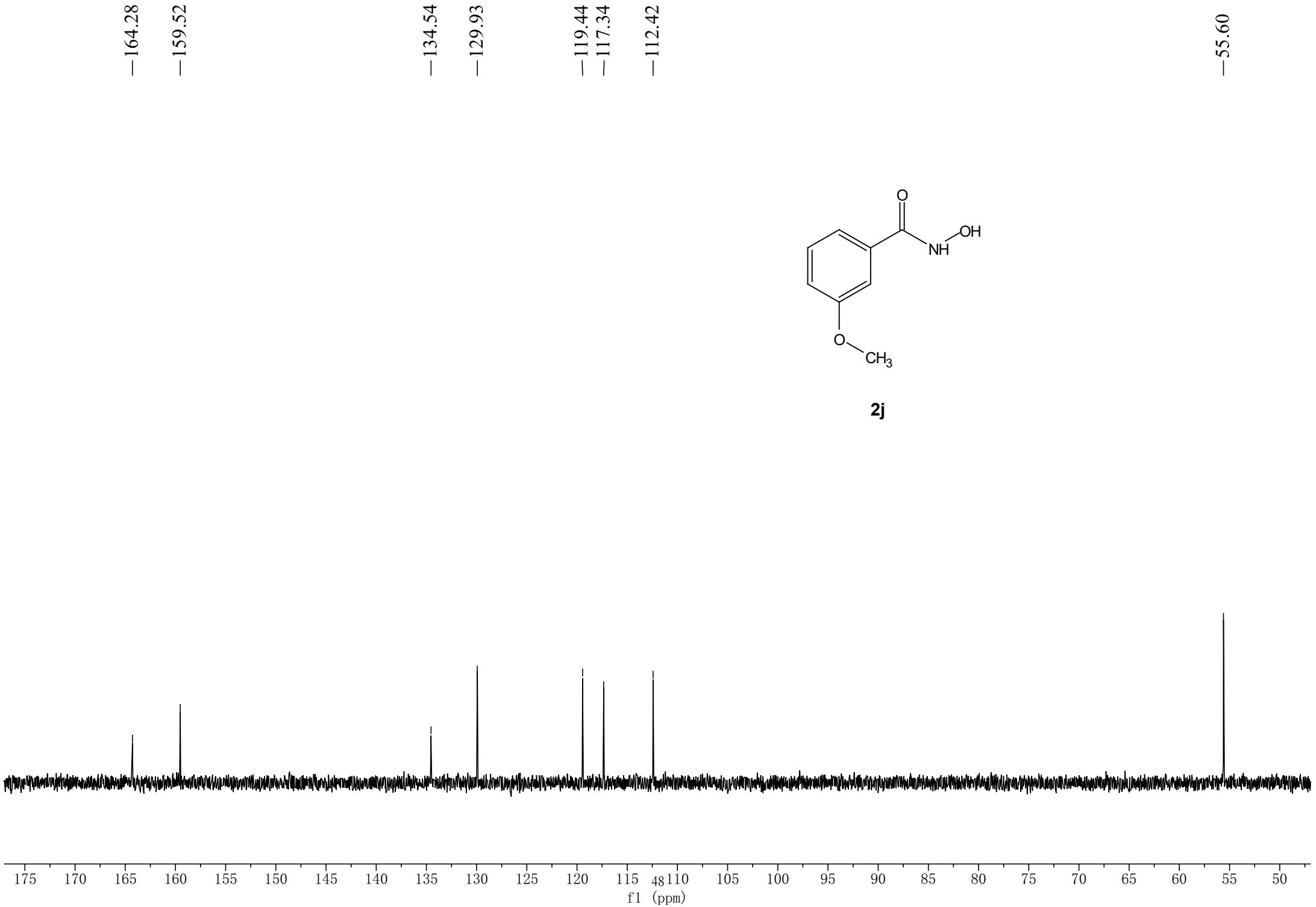
| Element | Min | Max |
|---|---|---|
| C | 3 | 60 |
| H | 0 | 120 |
| O | 0 | 30 |
| N | 0 | 30 |
| Cl | 0 | 0 |
| F | 0 | 0 |
| Br | 1 | 1 |

**Formula Calculator Results**

| Formula | Best | Mass | Tgt Mass | Diff (ppm) | Ion Species | Score |
|---|---|---|---|---|---|---|
| C7 H6 Br N O2 | TRUE | 214.9593 | 214.9582 | -5.26 | C7 H5 Br N O2 | 93.05 |

--- End Of Report ---


11.2025
9.0320
7.3750
7.3621
7.3490
7.3362
7.3340
7.3318
7.3237
7.3212
7.3190
7.2988
7.2951
7.2920
7.0875
7.0854
7.0829
7.0810
7.0743
7.0723
7.0697
7.0677
3.7894
3.3370
H2O
O
NH
OH
O
CH3
2j
1.0
1.0
3.0
0.9
3.0
f1 (ppm)

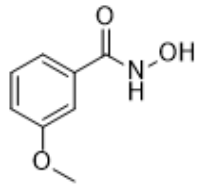

164.28
159.52
134.54
129.93
119.44
117.34
112.42
55.60
O
NH
OH
O
CH3
2j
175 170 165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50
f1 (ppm)

# Qualitative Analysis Report

| | | | |
|---|---|---|---|
| **Data Filename** | H-18.d | **Sample Name** | H-18 |
| **Sample Type** | Sample | **Position** | P1-F2 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | FU100-1000.m | **Acquired Time** | 9/10/2021 8:47:56 PM |
| **IRM Calibration Status** | Success | **DA Method** | 20170311.m |
| **Comment** | | | |

**Sample Group** **Info.**

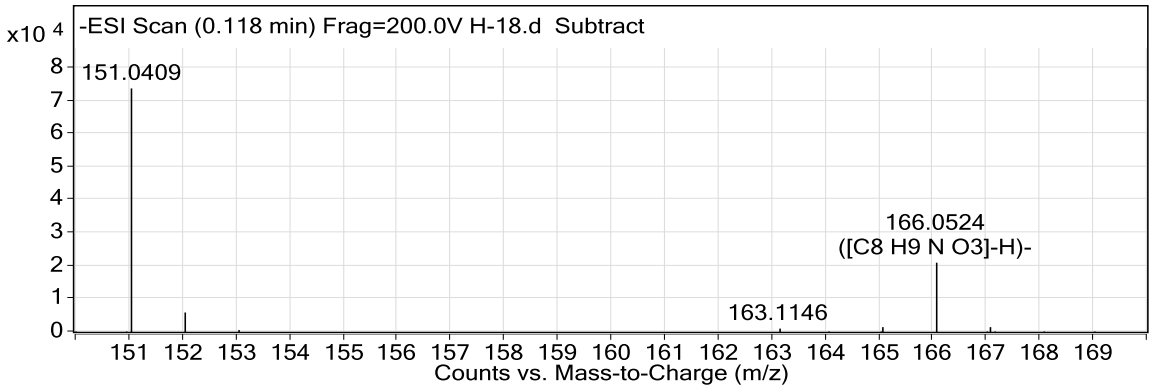
**Acquisition SW Version** 6200 series TOF/6500 series Q-TOF B.05.01 (B5125.2)



**2j**

## User Spectra

| Fragmentor Voltage | Collision Energy | Ionization Mode |
|---|---|---|
| 200 | 0 | ESI |




**Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 107.0429 | | 26161.12 | | |
| 108.0237 | 1 | 8583.72 | | |
| 110.9761 | | 8107.1 | | |
| 151.0409 | 1 | 73910.88 | | |
| 166.0524 | 1 | 21064.24 | C8 H9 N O3 | (M-H)- |
| 255.2341 | 1 | 9760.45 | | |
| 283.2659 | 1 | 9810.59 | | |
| 339.2342 | 1 | 75419.22 | | |
| 340.2372 | 1 | 18809.45 | | |
| 424.287 | 1 | 8763.27 | | |

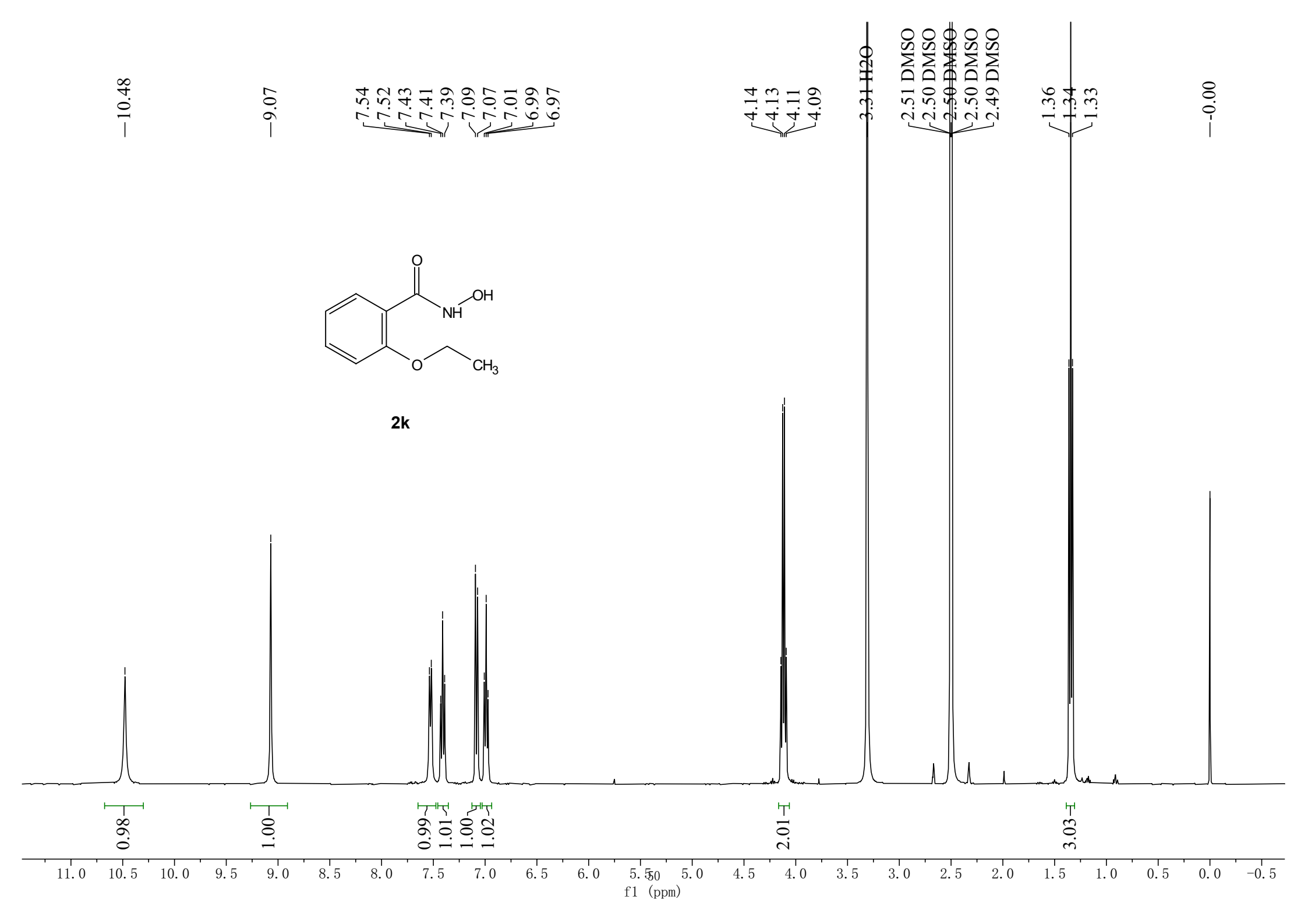
**Formula Calculator Element Limits**

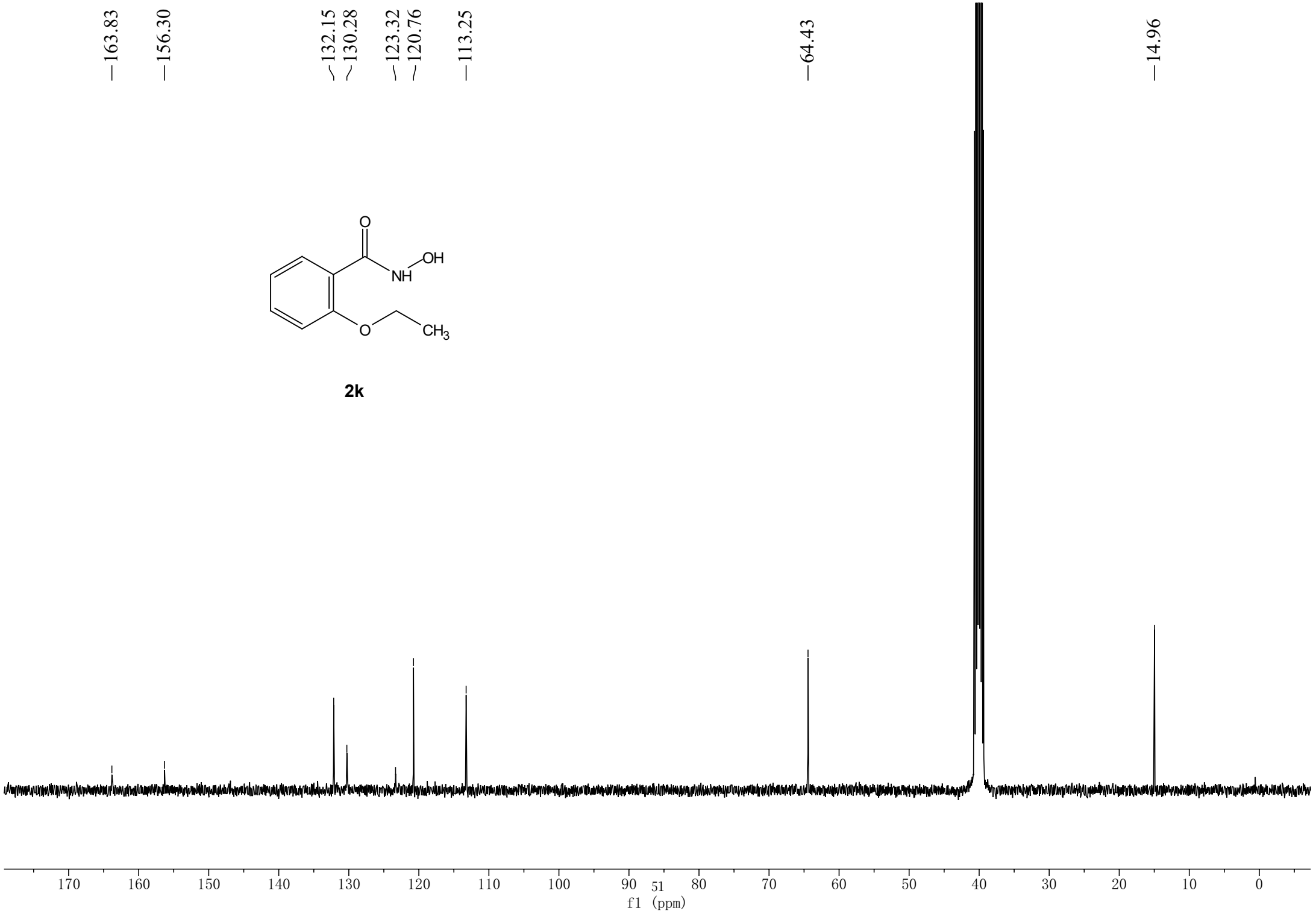
| Element | Min | Max |
|---|---|---|
| C | 3 | 60 |
| H | 0 | 120 |
| O | 0 | 30 |
| N | 0 | 30 |
| Cl | 0 | 0 |
| F | 0 | 0 |

**Formula Calculator Results**

| Formula | Best | Mass | Tgt Mass | Diff (ppm) | Ion Species | Score |
|---|---|---|---|---|---|---|
| C8 H9 N O3 | TRUE | 167.0597 | 167.0582 | -8.63 | C8 H8 N O3 | 87.85 |

--- End Of Report ---


10.48
9.07
7.54
7.52
7.43
7.41
7.39
7.09
7.07
7.01
6.99
6.97
4.14
4.13
4.11
4.09
3.31 H2O
2.51 DMSO
2.50 DMSO
2.50 DMSO
2.50 DMSO
2.49 DMSO
1.36
1.34
1.33
0.00
O
NH
OH
O
CH3
2k
0.98
1.00
0.99
1.01
1.00
1.02
2.01
3.03
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


163.83
156.30
132.15
130.28
123.32
120.76
113.25
64.43
14.96
O
NH
OH
O
CH3
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

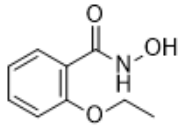
**2k**

# Qualitative Analysis Report

| | | | |
|---|---|---|---|
| **Data Filename** | H-1.d | **Sample Name** | H-1 |
| **Sample Type** | Sample | **Position** | P1-D3 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | FU100-1000.m | **Acquired Time** | 9/10/2021 7:57:54 PM |
| **IRM Calibration Status** | Success | **DA Method** | 20170311.m |
| **Comment** | | | |

**Sample Group** **Info.**

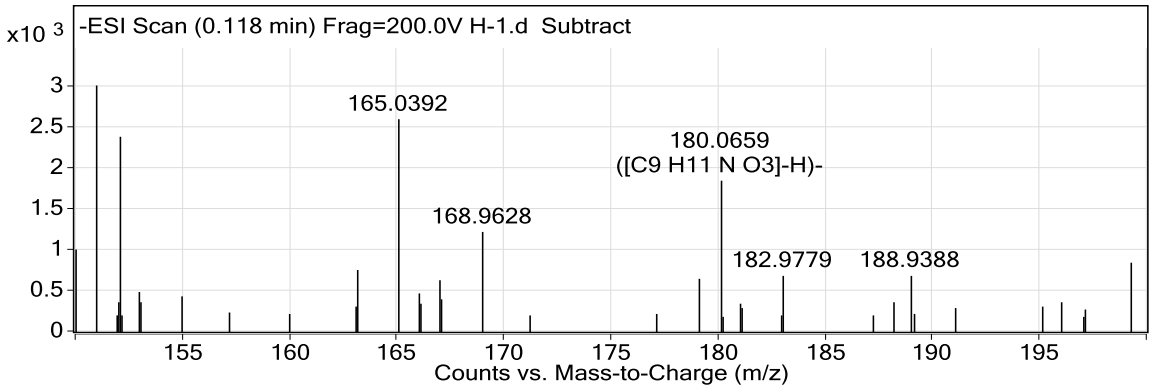
**Acquisition SW Version** 6200 series TOF/6500 series Q-TOF B.05.01 (B5125.2)



**2k**

## User Spectra

| Fragmentor Voltage | Collision Energy | Ionization Mode |
|---|---|---|
| 200 | 0 | ESI |




**Peak List**

| *m/z* | z | Abund |
|---|---|---|
| 110.9747 | | 16586.93 |
| 134.0236 | 1 | 10764.76 |
| 149.0082 | 1 | 8650.45 |
| 223.0281 | 1 | 12368.32 |
| 225.0085 | 1 | 7407.75 |
| 255.2313 | 1 | 14229.81 |
| 283.2622 | 1 | 14410.59 |
| 299.0249 | 1 | 5546.05 |
| 339.2306 | 1 | 52745.51 |
| 340.2325 | 1 | 12475.7 |

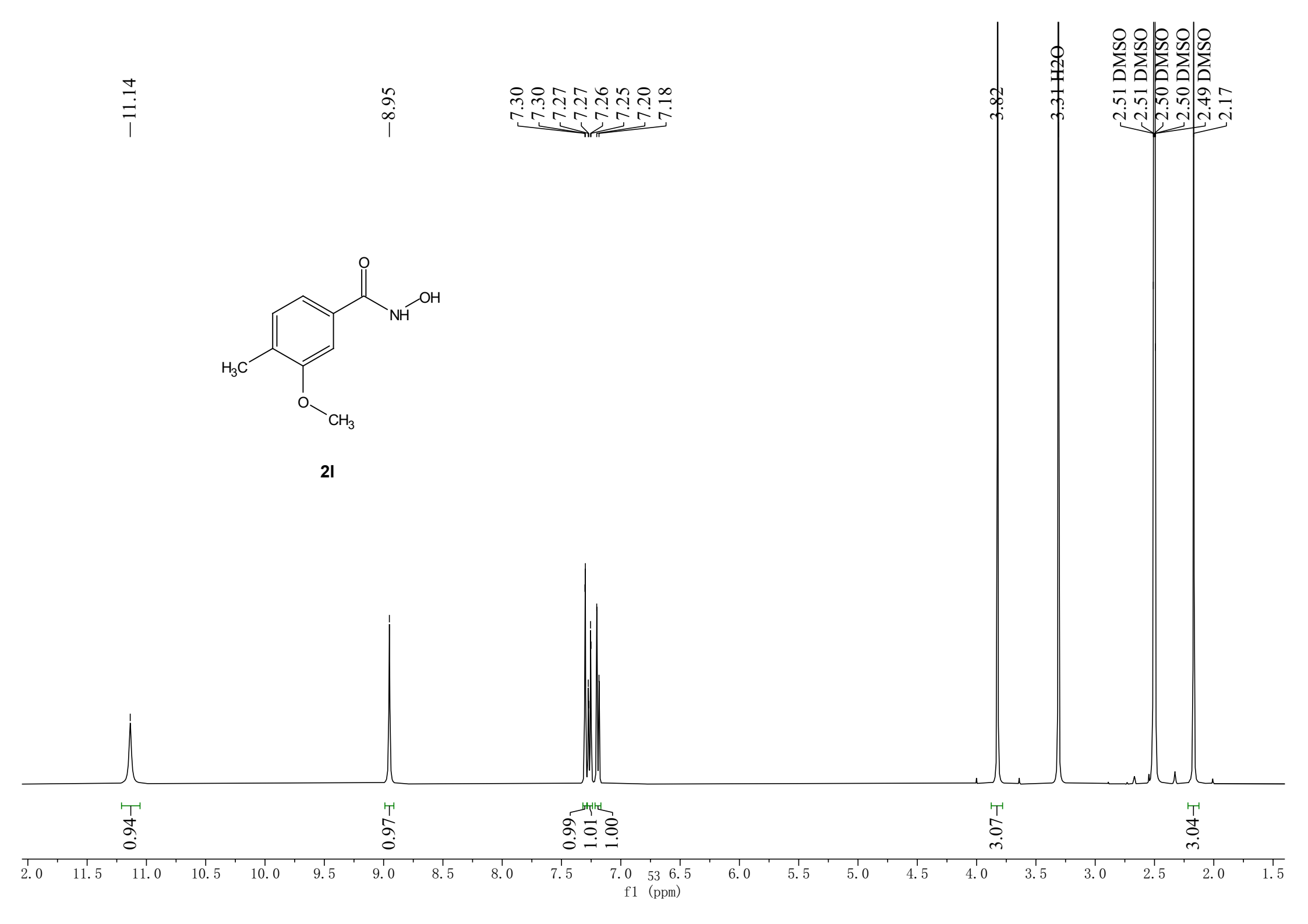
**Formula Calculator Element Limits**

| Element | Min | Max |
|---|---|---|
| C | 3 | 60 |
| H | 0 | 120 |
| O | 0 | 30 |
| N | 0 | 30 |
| Cl | 0 | 0 |
| F | 0 | 0 |
| S | 0 | 0 |
| Br | 0 | 0 |

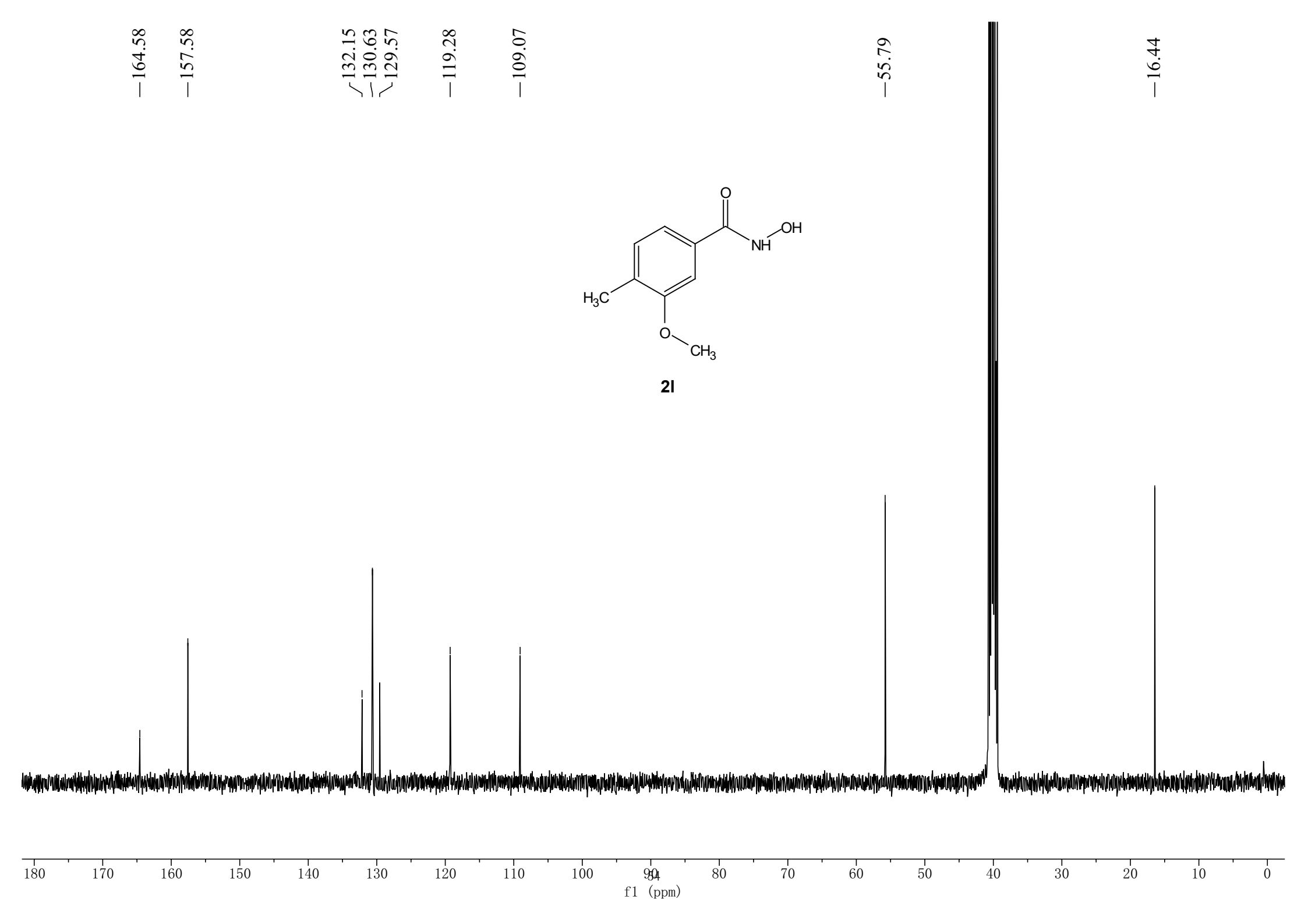
**Formula Calculator Results**

| Formula | Best | Mass | Tgt Mass | Diff (ppm) | Ion Species | Score |
|---|---|---|---|---|---|---|
| C9 H11 N O3 | TRUE | 181.0732 | 181.0739 | 3.92 | C9 H10 N O3 | 76.35 |

--- End Of Report ---


11.14
8.95
7.30
7.30
7.27
7.27
7.26
7.25
7.20
7.18
3.82
3.31 H2O
2.51 DMSO
2.51 DMSO
2.50 DMSO
2.50 DMSO
2.49 DMSO
2.17
O
NH
OH
H3C
O
CH3
0.94
0.97
0.99
1.01
1.00
3.07
3.04
f1 (ppm)

**2l**

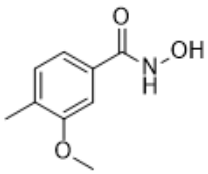

164.58
157.58
132.15
130.63
129.57
119.28
109.07
55.79
16.44
O
NH
OH
H3C
O
CH3
2l
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

# Qualitative Analysis Report

| | | | |
|---|---|---|---|
| **Data Filename** | H-11.d | **Sample Name** | H-11 |
| **Sample Type** | Sample | **Position** | P1-E4 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | FU100-1000.m | **Acquired Time** | 9/10/2021 8:27:24 PM |
| **IRM Calibration Status** | Success | **DA Method** | 20170311.m |
| **Comment** | | | |

**Sample Group** **Info.**

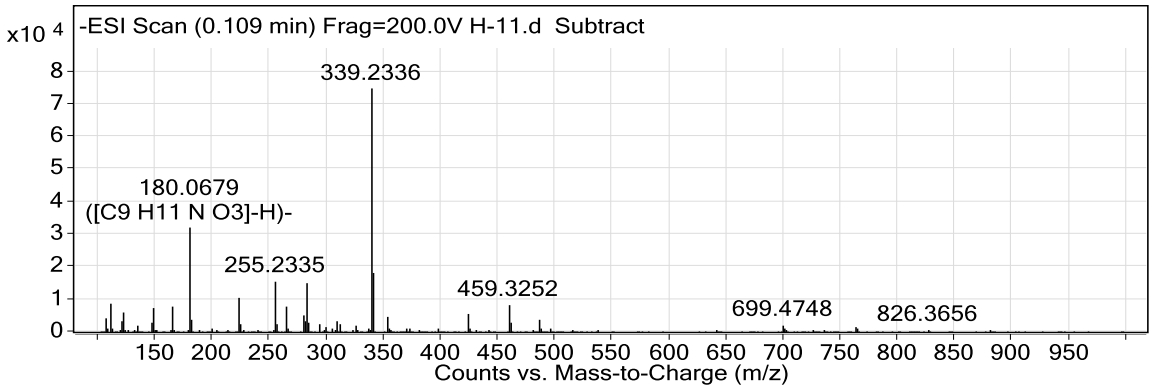
**Acquisition SW Version** 6200 series TOF/6500 series Q-TOF B.05.01 (B5125.2)



**2l**

## User Spectra

| Fragmentor Voltage | Collision Energy | Ionization Mode |
|---|---|---|
| 200 | 0 | ESI |




**Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 110.9757 | | 8873.31 | | |
| 165.045 | 1 | 8032.42 | | |
| 180.0679 | 1 | 32062.4 | C9 H11 N O3 | (M-H)- |
| 223.0288 | 1 | 10612.4 | | |
| 255.2335 | 1 | 15861.86 | | |
| 265.1485 | 1 | 8077.94 | | |
| 283.2653 | 1 | 15353.16 | | |
| 339.2336 | 1 | 74971.66 | | |
| 340.2355 | 1 | 18168.22 | | |
| 459.3252 | 1 | 8547.56 | | |

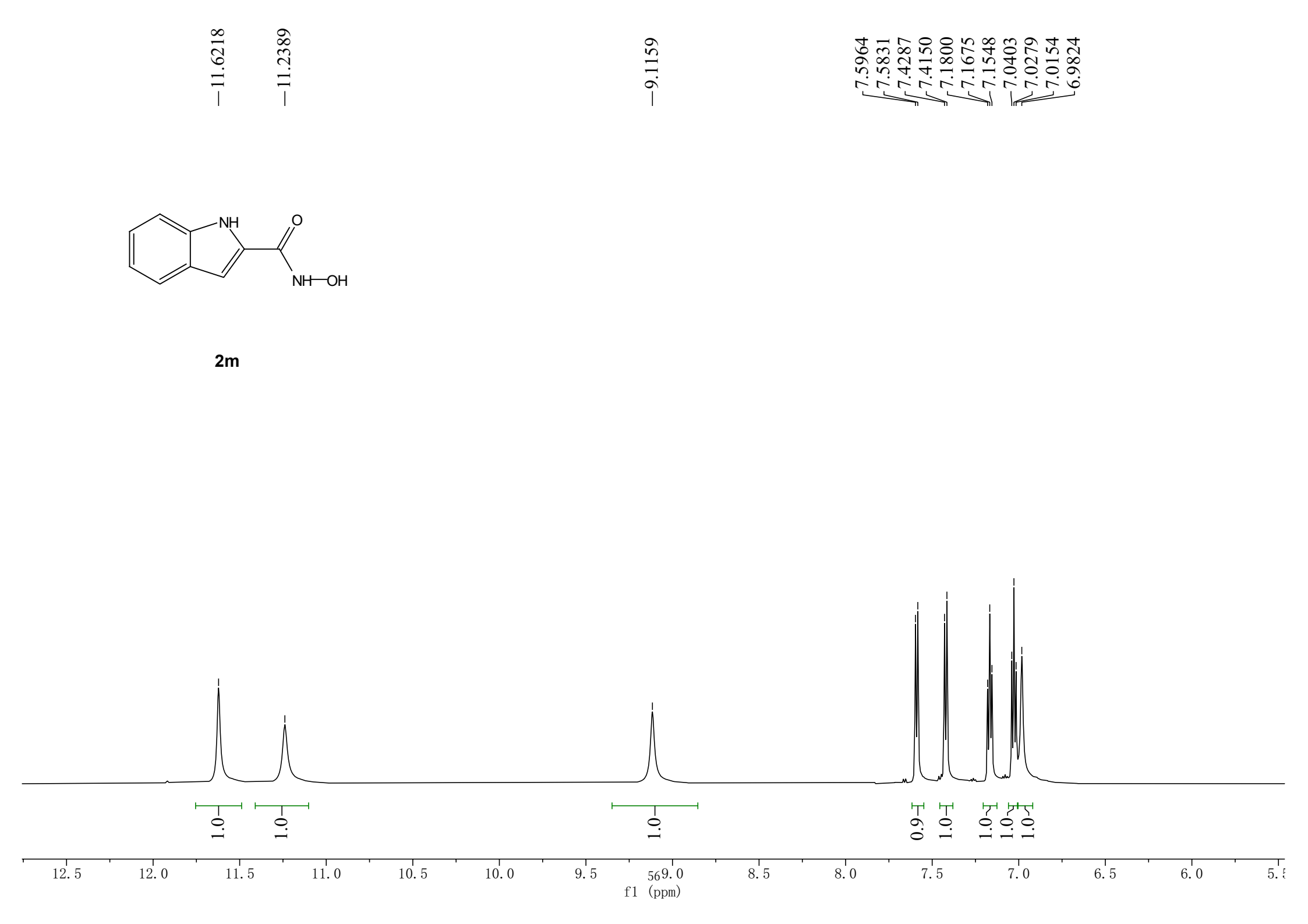
**Formula Calculator Element Limits**

| Element | Min | Max |
|---|---|---|
| C | 3 | 60 |
| H | 0 | 120 |
| O | 0 | 30 |
| N | 0 | 30 |
| Cl | 0 | 0 |
| F | 0 | 0 |
| Br | 0 | 0 |
| I | 0 | 0 |

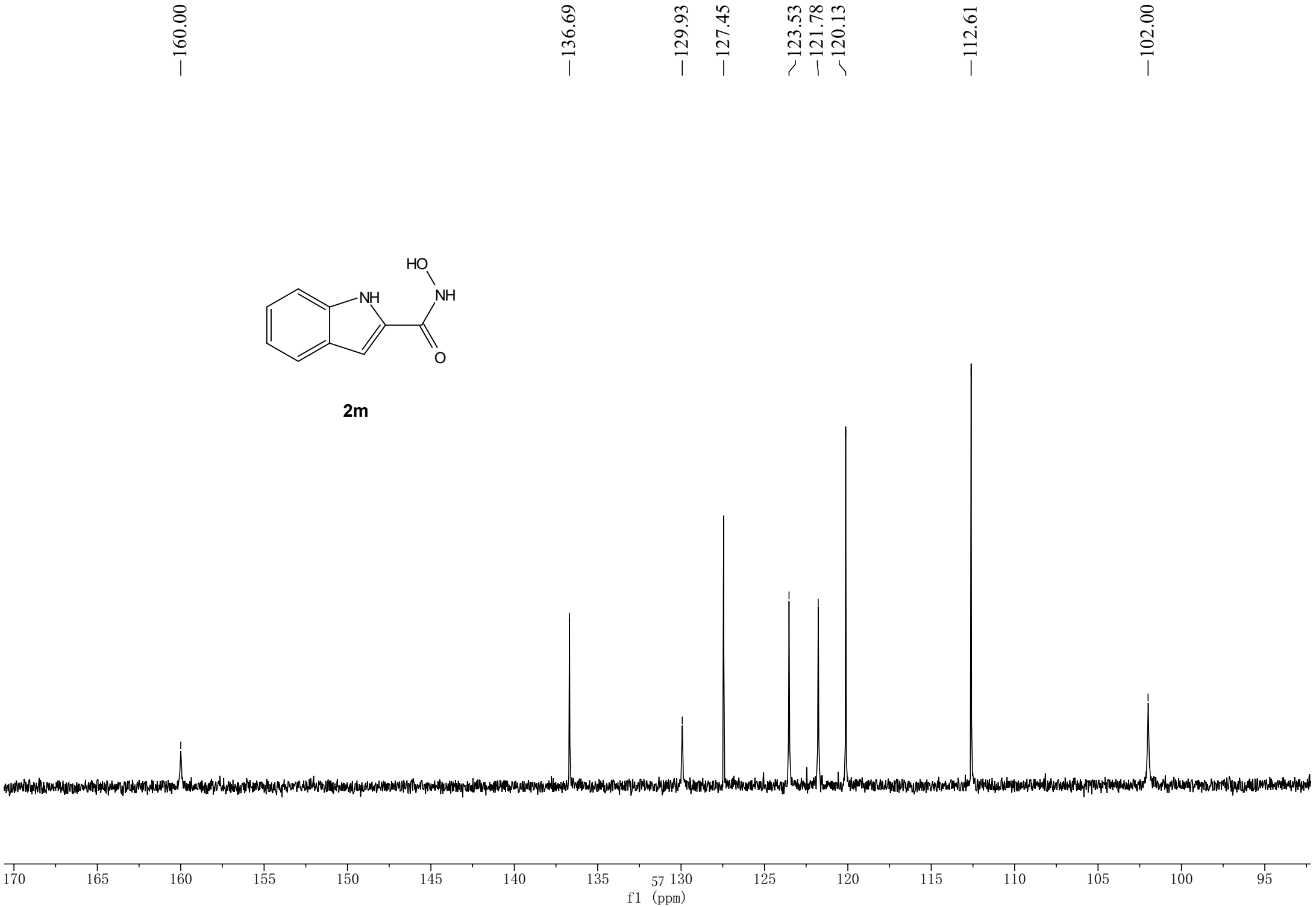
**Formula Calculator Results**

| Formula | Best | Mass | Tgt Mass | Diff (ppm) | Ion Species | Score |
|---|---|---|---|---|---|---|
| C9 H11 N O3 | TRUE | 181.0751 | 181.0739 | -6.91 | C9 H10 N O3 | 91.43 |

--- End Of Report ---


11.6218
11.2389
9.1159
7.5964
7.5831
7.4287
7.4150
7.1800
7.1675
7.1548
7.0403
7.0279
7.0154
6.9824
NH
O
NH—OH
1.0
1.0
1.0
0.9
1.0
1.0
1.0
1.0
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
f1 (ppm)

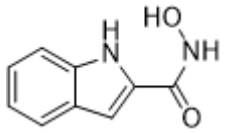
**2m**


160.00
136.69
129.93
127.45
123.53
121.78
120.13
112.61
102.00
HO
NH
NH
O
2m
170
165
160
155
150
145
140
135
130
125
120
115
110
105
100
95
f1 (ppm)

# Qualitative Analysis Report

| | | | |
|---|---|---|---|
| **Data Filename** | H-9.d | **Sample Name** | H-9 |
| **Sample Type** | Sample | **Position** | P1-E2 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | FU100-1000.m | **Acquired Time** | 9/10/2021 8:21:31 PM |
| **IRM Calibration Status** | Success | **DA Method** | 20170311.m |
| **Comment** | | | |

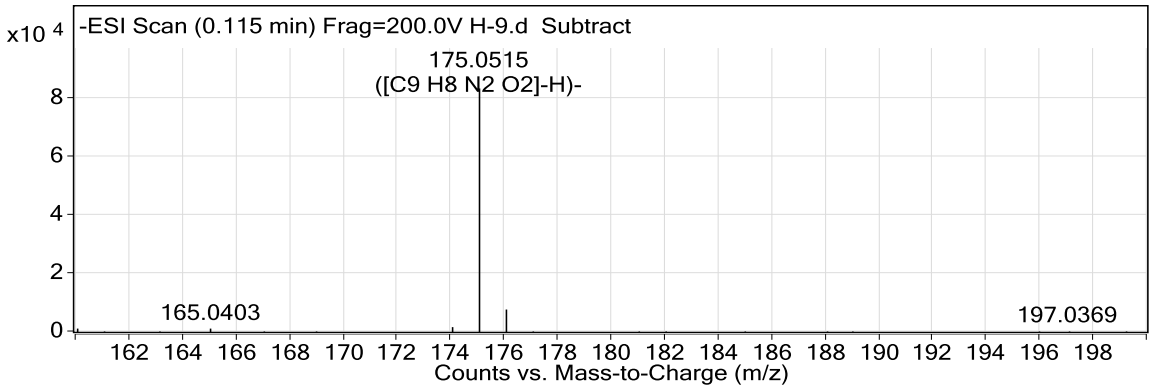
| | |
|---|---|
| **Sample Group** | **Info.** |
| **Acquisition SW Version** | 6200 series TOF/6500 series Q-TOF B.05.01 (B5125.2) |




**2m**

## User Spectra

| Fragmentor Voltage | Collision Energy | Ionization Mode |
|---|---|---|
| 200 | 0 | ESI |




**Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 110.975 | | 7256.83 | | |
| 115.0421 | | 11173.14 | | |
| 116.0495 | 1 | 11671.8 | | |
| 131.0387 | 1 | 15016.66 | | |
| 157.0411 | 1 | 291610.16 | | |
| 158.0433 | 1 | 25498.4 | | |
| 175.0515 | 1 | 83650.23 | C9 H8 N2 O2 | (M-H)- |
| 176.0542 | 1 | 7867.67 | C9 H8 N2 O2 | (M-H)- |
| 223.0285 | 1 | 6597.09 | | |
| 339.232 | 1 | 25715.18 | | |

**Formula Calculator Element Limits**

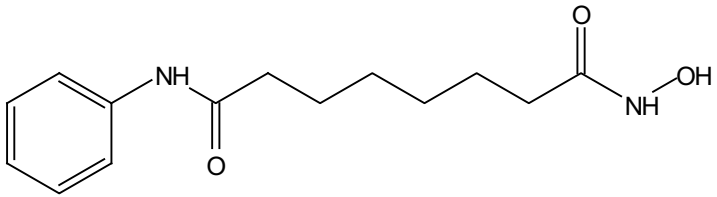
| Element | Min | Max |
|---|---|---|
| C | 3 | 60 |
| H | 0 | 120 |
| O | 0 | 30 |
| N | 0 | 30 |
| Cl | 0 | 0 |
| F | 0 | 0 |
| Br | 0 | 0 |
| I | 0 | 0 |

**Formula Calculator Results**

| Formula | Best | Mass | Tgt Mass | Diff (ppm) | Ion Species | Score |
|---|---|---|---|---|---|---|
| C9 H8 N2 O2 | TRUE | 176.0587 | 176.0586 | -0.96 | C9 H7 N2 O2 | 99.18 |

--- End Of Report ---

SAHA-0711.10.fid
1H NMR SAHA-0711 in DMSO



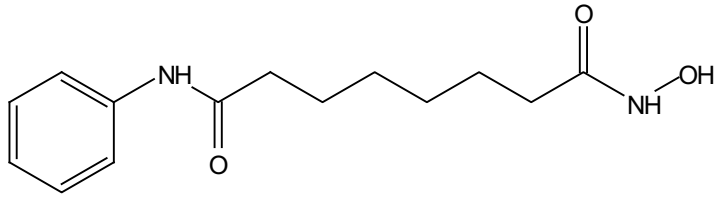

**2n**

f1 (ppm)

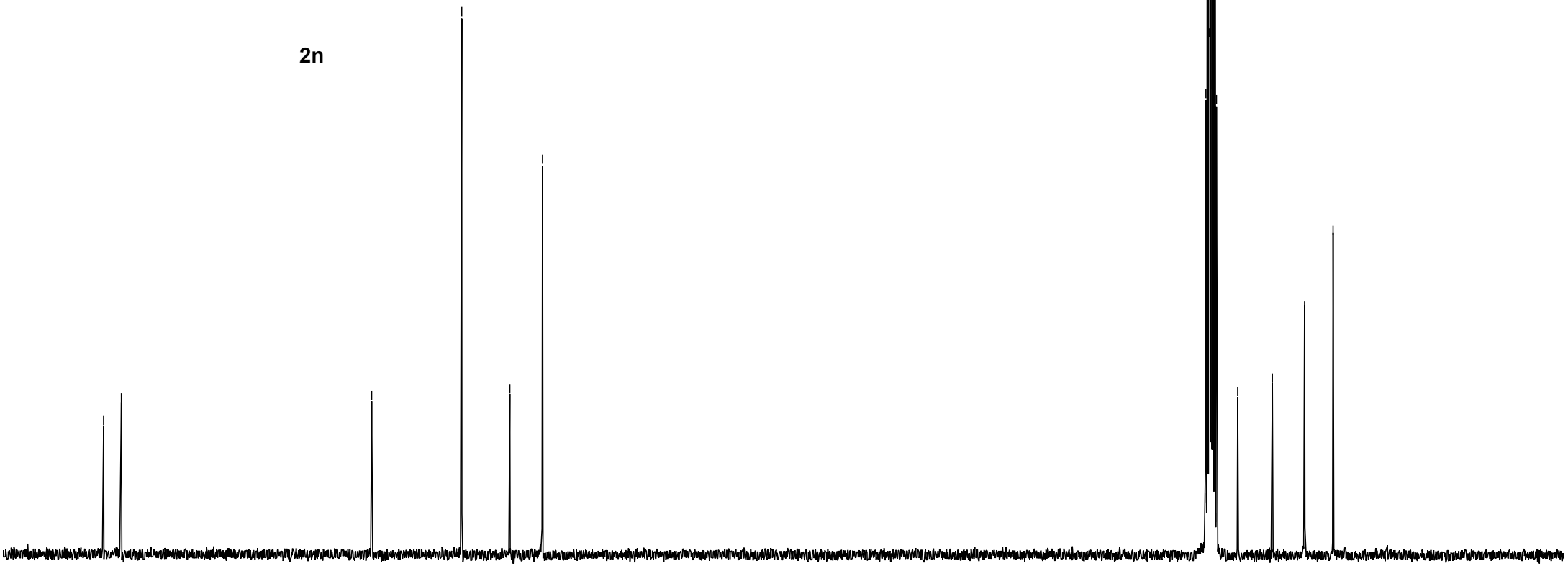
SAHA-0711.20.fid
13C NMR SAHA-0711 in DMSO

171.6861
169.5723
139.8100
129.0996
123.3792
119.4946

40.6626 DMSO
40.6112 DMSO
40.5461
40.4542 DMSO
40.4033 DMSO
40.2453 DMSO
40.1938 DMSO
40.0841
40.0366 DMSO
39.9862 DMSO
39.8606
39.7778 DMSO
39.6705
39.5696 DMSO
39.4509
39.3590 DMSO
36.8361
32.7128
28.8803
25.4949

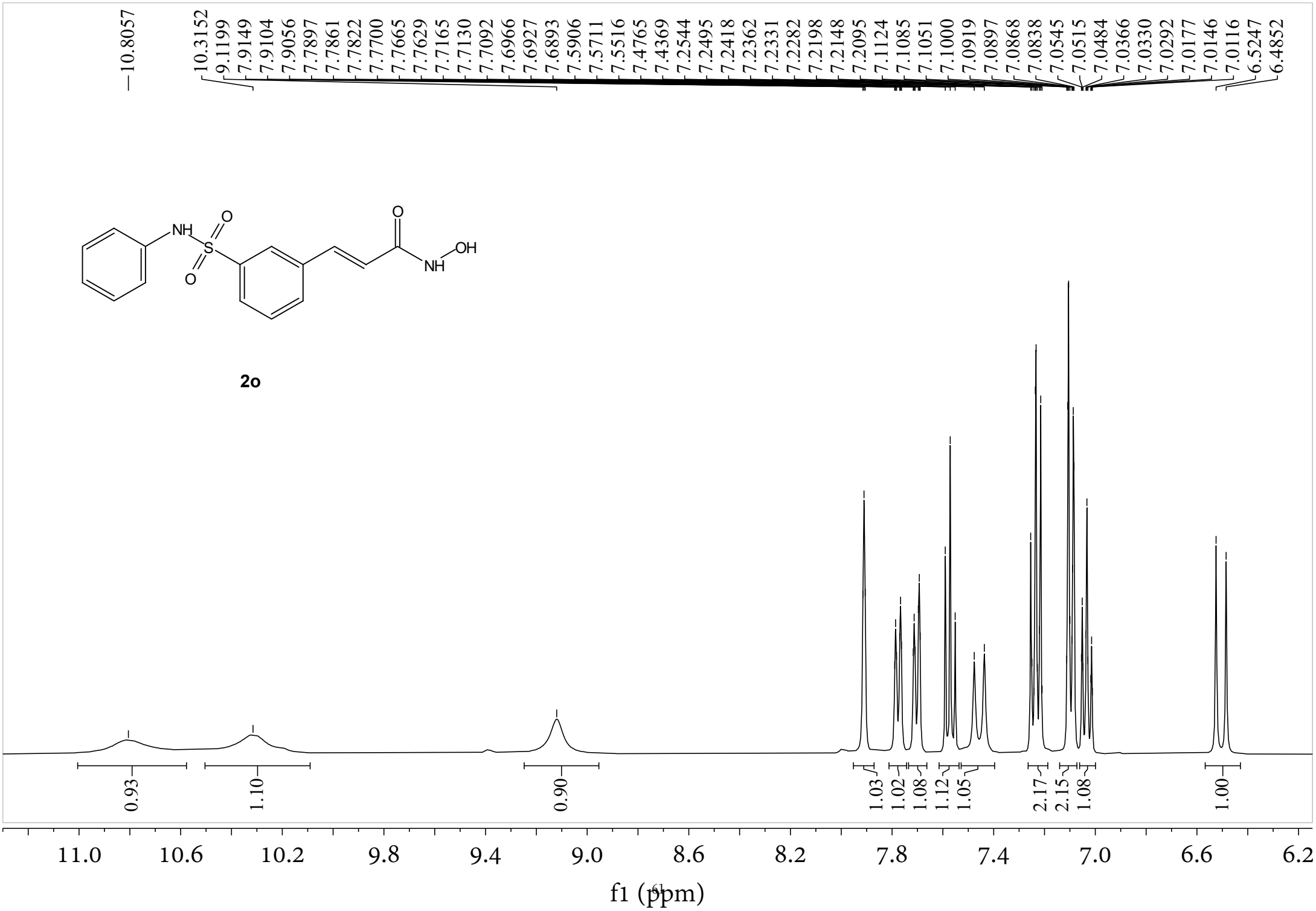



**2n**

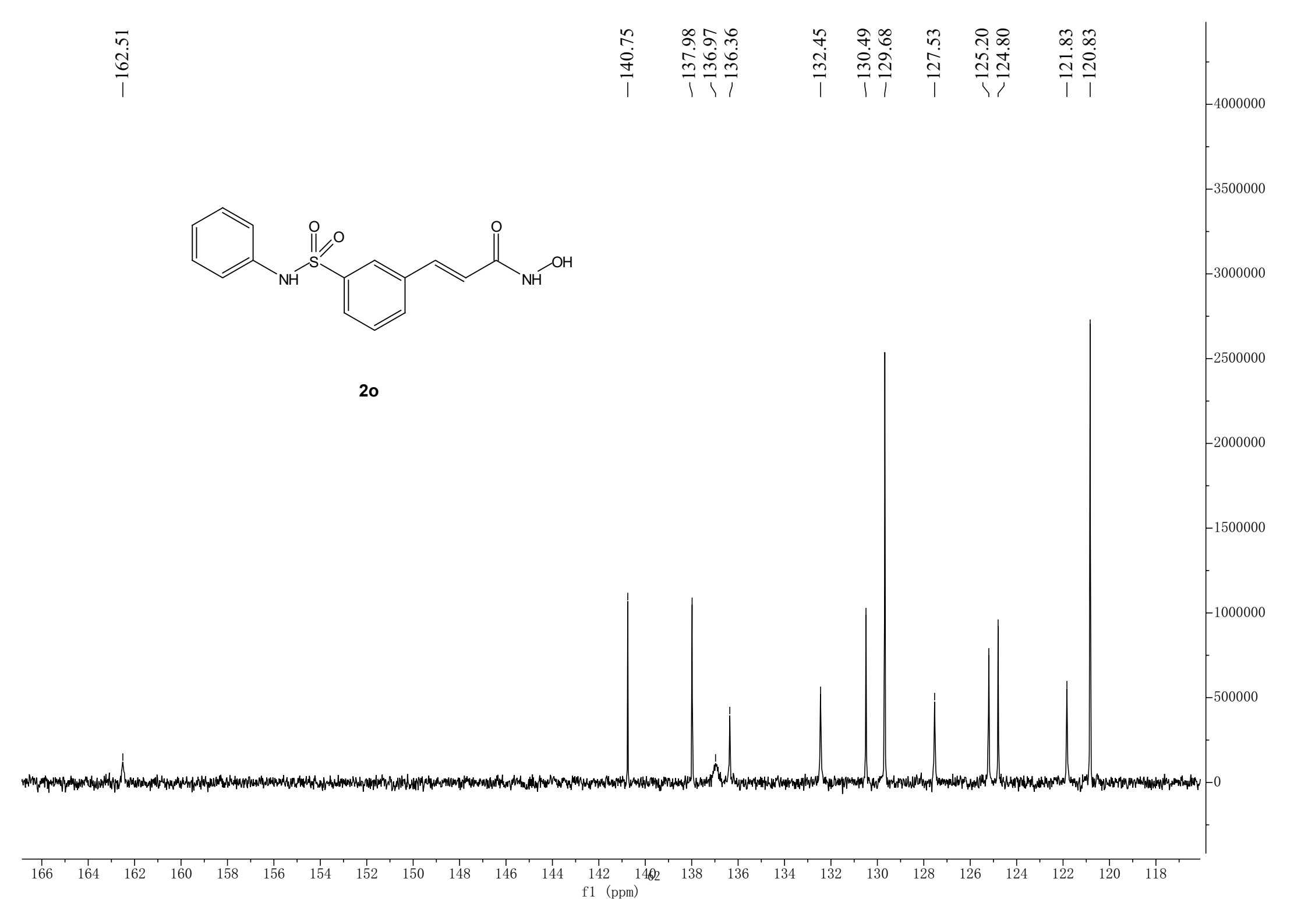


180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)


10.8057
10.3152
9.1199
7.9149
7.9104
7.9056
7.7897
7.7861
7.7822
7.7700
7.7665
7.7629
7.7165
7.7130
7.7092
7.6966
7.6927
7.6893
7.5906
7.5711
7.5516
7.4765
7.4369
7.2544
7.2495
7.2418
7.2362
7.2331
7.2282
7.2198
7.2148
7.2095
7.1124
7.1085
7.1051
7.1000
7.0919
7.0897
7.0868
7.0838
7.0545
7.0515
7.0484
7.0366
7.0330
7.0292
7.0177
7.0146
7.0116
6.5247
6.4852
NH
S
O
O
O
NH
OH
0.93
1.10
0.90
1.03
1.02
1.08
1.12
1.05
2.17
2.15
1.08
1.00
11.0
10.6
10.2
9.8
9.4
9.0
8.6
8.2
7.8
7.4
7.0
6.6
6.2
f1 (ppm)

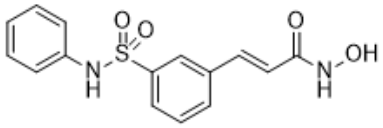
**2o**


162.51
140.75
137.98
136.97
136.36
132.45
130.49
129.68
127.53
125.20
124.80
121.83
120.83
O
O
S
NH
O
NH
OH
2o
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
166
164
162
160
158
156
154
152
150
148
146
144
142
140
138
136
134
132
130
128
126
124
122
120
118
f1 (ppm)

# Qualitative Analysis Report

| | | | |
|---|---|---|---|
| **Data Filename** | H-19.d | **Sample Name** | H-19 |
| **Sample Type** | Sample | **Position** | P1-F3 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | FU100-1000.m | **Acquired Time** | 9/10/2021 8:50:53 PM |
| **IRM Calibration Status** | Success | **DA Method** | 20170311.m |
| **Comment** | | | |

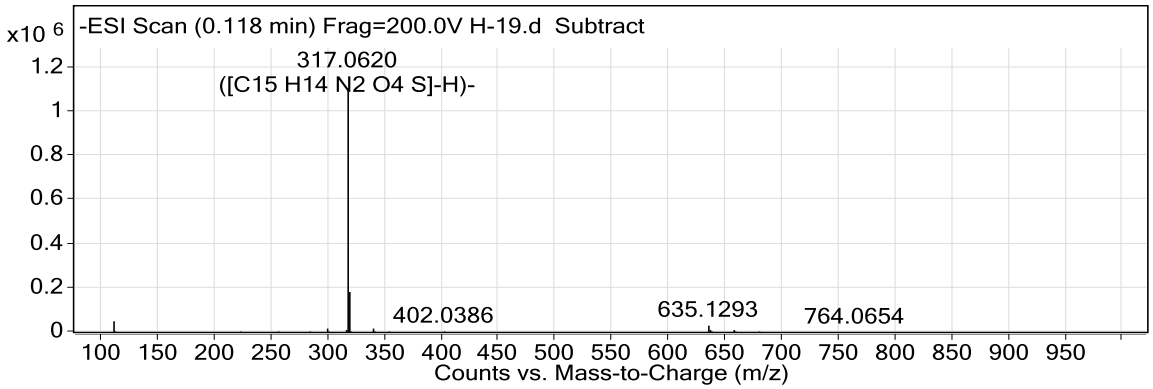
| | |
|---|---|
| **Sample Group** | **Info.** |
| **Acquisition SW Version** | 6200 series TOF/6500 series Q-TOF B.05.01 (B5125.2) |



**2o**

## User Spectra

| Fragmentor Voltage | Collision Energy | Ionization Mode |
|---|---|---|
| 200 | 0 | ESI |




**Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 110.9759 | 1 | 52656.79 | | |
| 299.05 | 1 | 22079.8 | | |
| 316.0645 | | 12206.38 | | |
| 317.062 | 1 | 1107429.13 | C15 H14 N2 O4 S | (M-H)- |
| 318.0648 | 1 | 182631.23 | C15 H14 N2 O4 S | (M-H)- |
| 319.0615 | 1 | 55347.61 | C15 H14 N2 O4 S | (M-H)- |
| 339.2344 | 1 | 17058.5 | | |
| 635.1293 | 1 | 31964.1 | | |
| 636.1315 | 1 | 11691.43 | | |
| 657.1118 | 1 | 13069.45 | | |

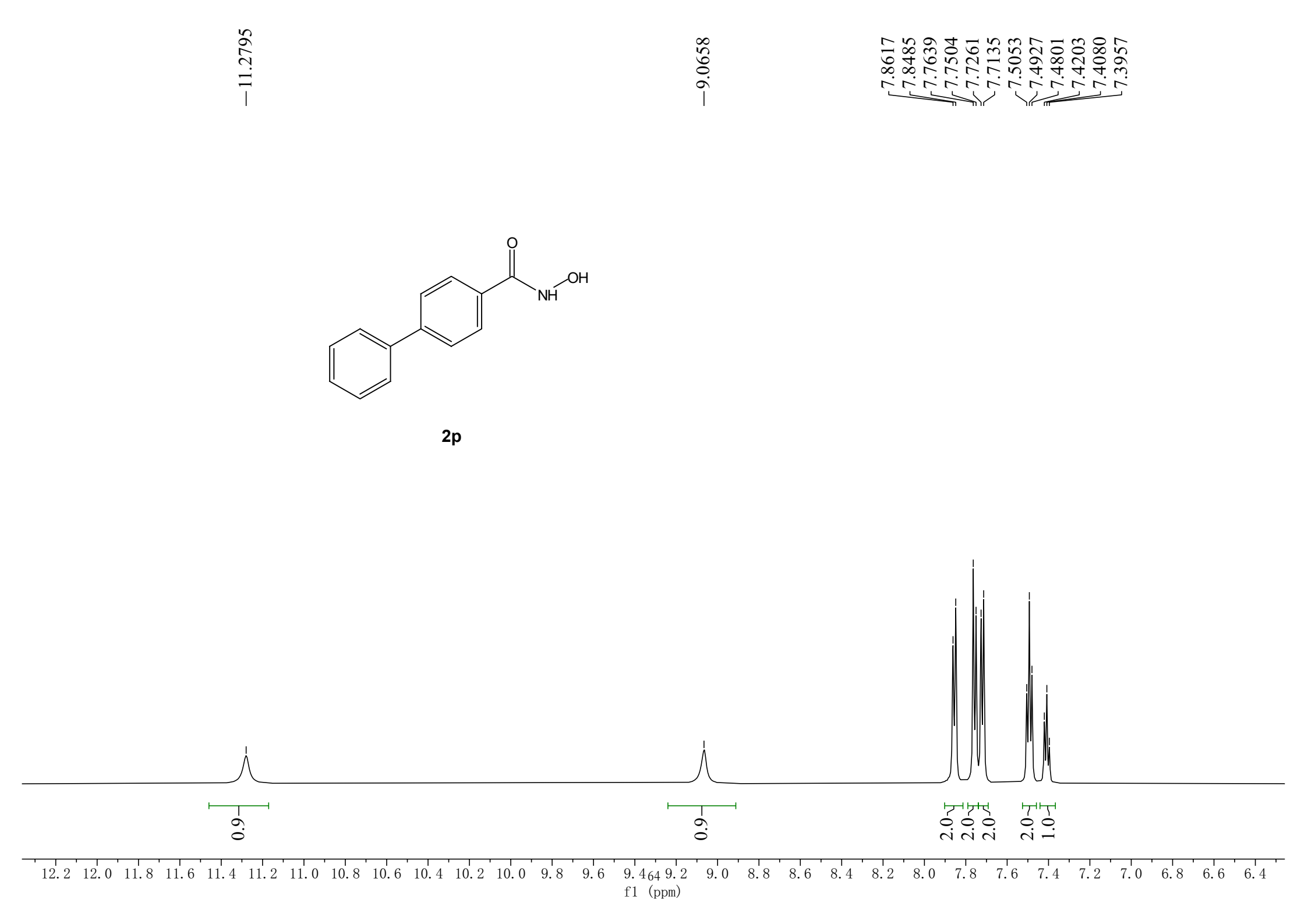
**Formula Calculator Element Limits**

| Element | Min | Max |
|---|---|---|
| C | 3 | 60 |
| H | 0 | 120 |
| O | 0 | 30 |
| N | 0 | 30 |
| Cl | 0 | 0 |
| F | 0 | 0 |
| S | 1 | 1 |

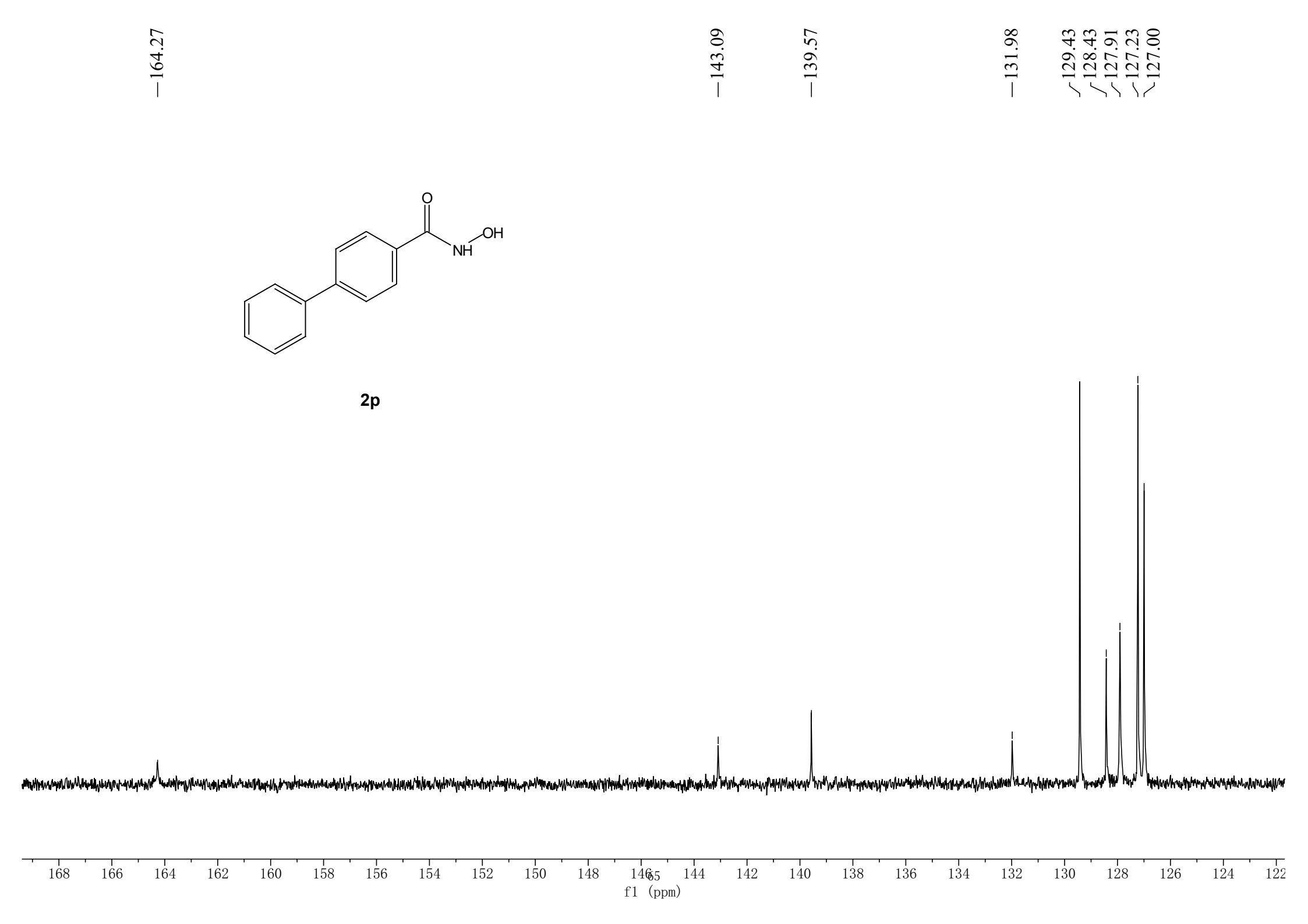
**Formula Calculator Results**

| Formula | Best | Mass | Tgt Mass | Diff (ppm) | Ion Species | Score |
|---|---|---|---|---|---|---|
| C15 H14 N2 O4 S | FALSE | 318.0693 | 318.0674 | -5.88 | C15 H13 N2 O4 S | 88.31 |

--- End Of Report ---


11.2795
9.0658
7.8617
7.8485
7.7639
7.7504
7.7261
7.7135
7.5053
7.4927
7.4801
7.4203
7.4080
7.3957
O
NH
OH
0.9
0.9
2.0
2.0
2.0
2.0
1.0
12.2 12.0 11.8 11.6 11.4 11.2 11.0 10.8 10.6 10.4 10.2 10.0 9.8 9.6 9.464 9.2 9.0 8.8 8.6 8.4 8.2 8.0 7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4
f1 (ppm)

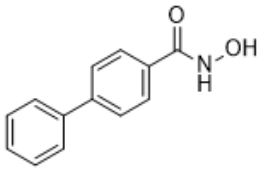
**2p**


164.27
143.09
139.57
131.98
129.43
128.43
127.91
127.23
127.00
O
NH
OH
2p
168
166
164
162
160
158
156
154
152
150
148
146
144
142
140
138
136
134
132
130
128
126
124
122
f1 (ppm)

# Qualitative Analysis Report

| | | | |
|---|---|---|---|
| **Data Filename** | H-6.d | **Sample Name** | H-6 |
| **Sample Type** | Sample | **Position** | P1-D8 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | FU100-1000.m | **Acquired Time** | 9/10/2021 8:12:42 PM |
| **IRM Calibration Status** | Success | **DA Method** | 20170311.m |
| **Comment** | | | |

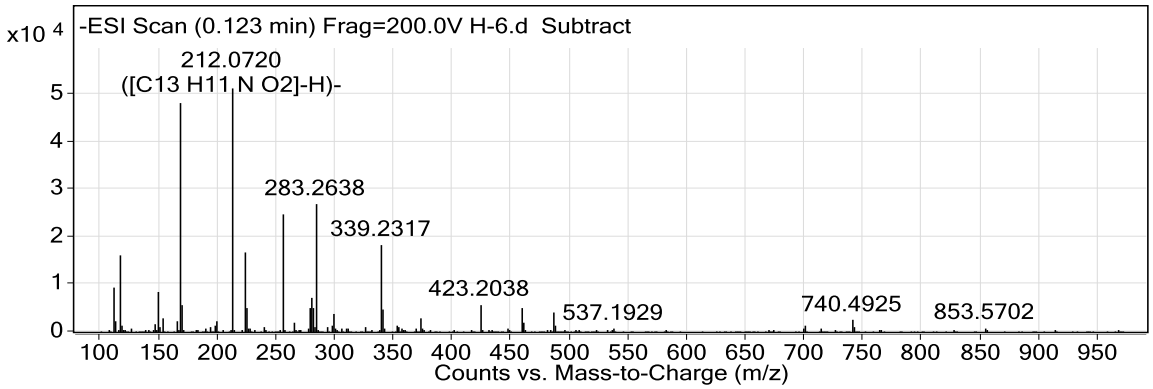
| | | |
|---|---|---|
| **Sample Group** | | **Info.** |
| **Acquisition SW Version** | 6200 series TOF/6500 series Q-TOF B.05.01 (B5125.2) | |




**2p**

## User Spectra

| Fragmentor Voltage | Collision Energy | Ionization Mode |
|---|---|---|
| 200 | 0 | ESI |




**Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 110.9753 | | 9583.14 | | |
| 117.0454 | 1 | 16289.46 | | |
| 149.0092 | 1 | 8452.67 | | |
| 168.0819 | 1 | 48064.19 | | |
| 212.072 | 1 | 51322.3 | C13 H11 N O2 | (M-H)- |
| 223.0286 | 1 | 16777.26 | | |
| 255.2332 | 1 | 24838.26 | | |
| 279.2324 | 1 | 7311.91 | | |
| 283.2638 | 1 | 27043.36 | | |
| 339.2317 | 1 | 18474.16 | | |

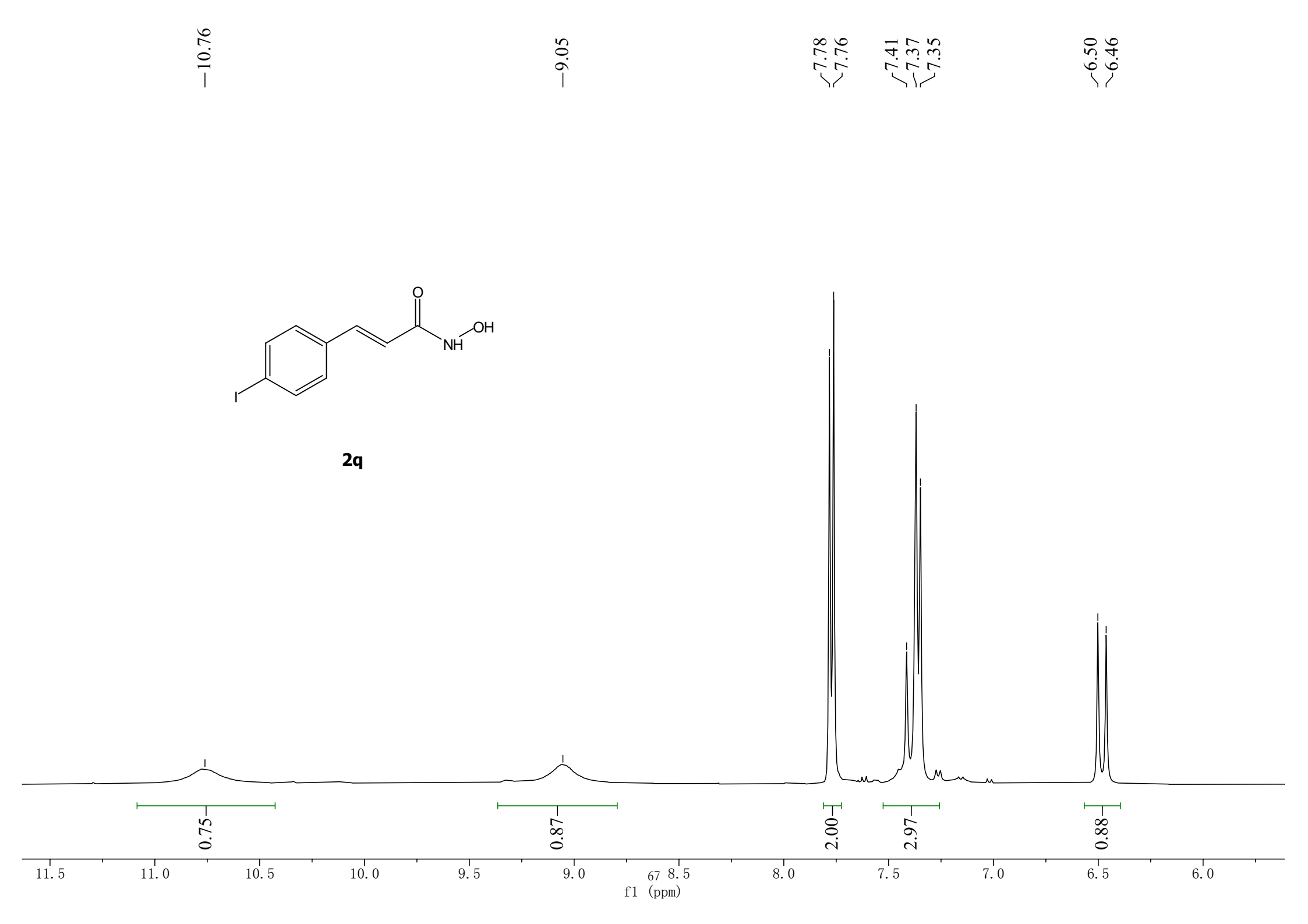
**Formula Calculator Element Limits**

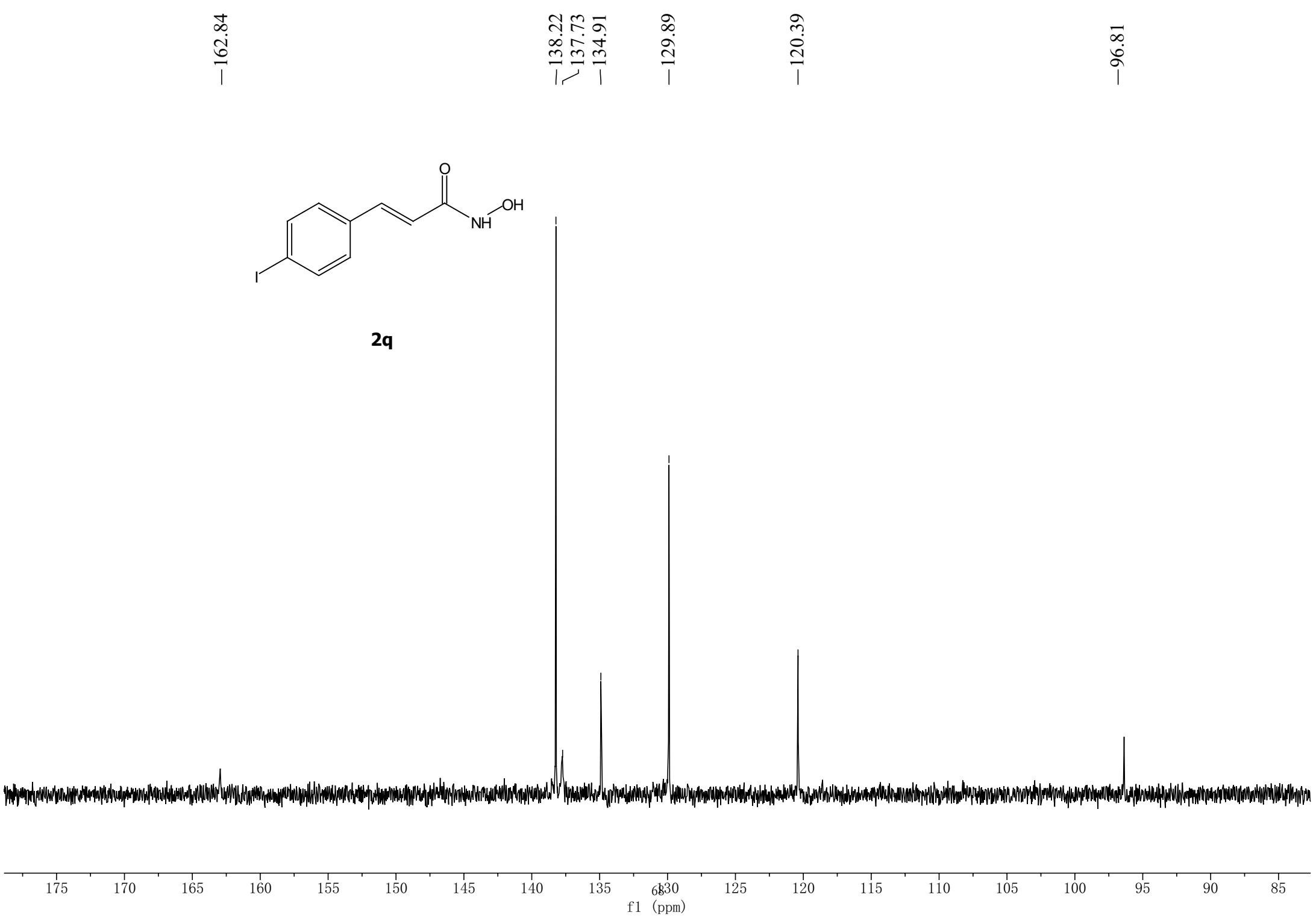
| Element | Min | Max |
|---|---|---|
| C | 3 | 60 |
| H | 0 | 120 |
| O | 0 | 30 |
| N | 0 | 30 |
| Cl | 0 | 0 |
| F | 0 | 0 |
| S | 0 | 0 |
| Br | 0 | 0 |

**Formula Calculator Results**

| Formula | Best | Mass | Tgt Mass | Diff (ppm) | Ion Species | Score |
|---|---|---|---|---|---|---|
| C13 H11 N O2 | TRUE | 213.0792 | 213.079 | -0.87 | C13 H10 N O2 | 97.91 |

--- End Of Report ---


10.76
9.05
7.78
7.76
7.41
7.37
7.35
6.50
6.46
O
NH
OH
I
2q
0.75
0.87
2.00
2.97
0.88
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
f1 (ppm)

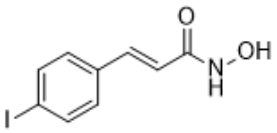

162.84
138.22
137.73
134.91
129.89
120.39
96.81
O
NH
OH
I
2q
175
170
165
160
155
150
145
140
135
130
125
120
115
110
105
100
95
90
85
f1 (ppm)

# Qualitative Analysis Report

| | | | |
|---|---|---|---|
| **Data Filename** | H-13.d | **Sample Name** | H-13 |
| **Sample Type** | Sample | **Position** | P1-E6 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | FU100-1000.m | **Acquired Time** | 9/10/2021 8:33:16 PM |
| **IRM Calibration Status** | Success | **DA Method** | 20170311.m |
| **Comment** | | | |

**Sample Group** **Info.**

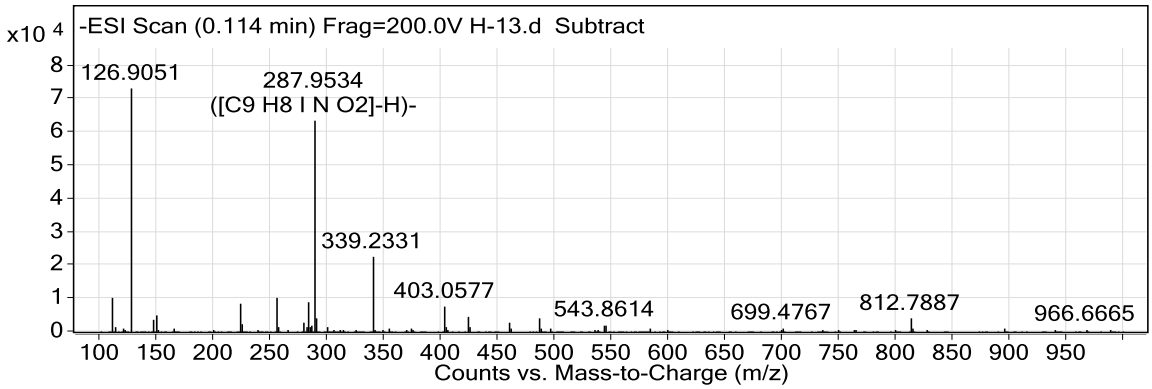
**Acquisition SW Version** 6200 series TOF/6500 series Q-TOF B.05.01 (B5125.2)




**2q**

## User Spectra

| Fragmentor Voltage | Collision Energy | Ionization Mode |
|---|---|---|
| 200 | 0 | ESI |




**Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 110.9761 | | 10625.62 | | |
| 126.9051 | | 73356 | | |
| 223.029 | 1 | 8786.01 | | |
| 255.2331 | 1 | 10344.08 | | |
| 283.2654 | 1 | 9097.98 | | |
| 287.9534 | 1 | 63628.27 | C9 H8 I N O2 | (M-H)- |
| 288.9557 | 1 | 6185.24 | C9 H8 I N O2 | (M-H)- |
| 339.2331 | 1 | 22716.29 | | |
| 340.2358 | 1 | 5732.13 | | |
| 403.0577 | 1 | 7799.41 | | |

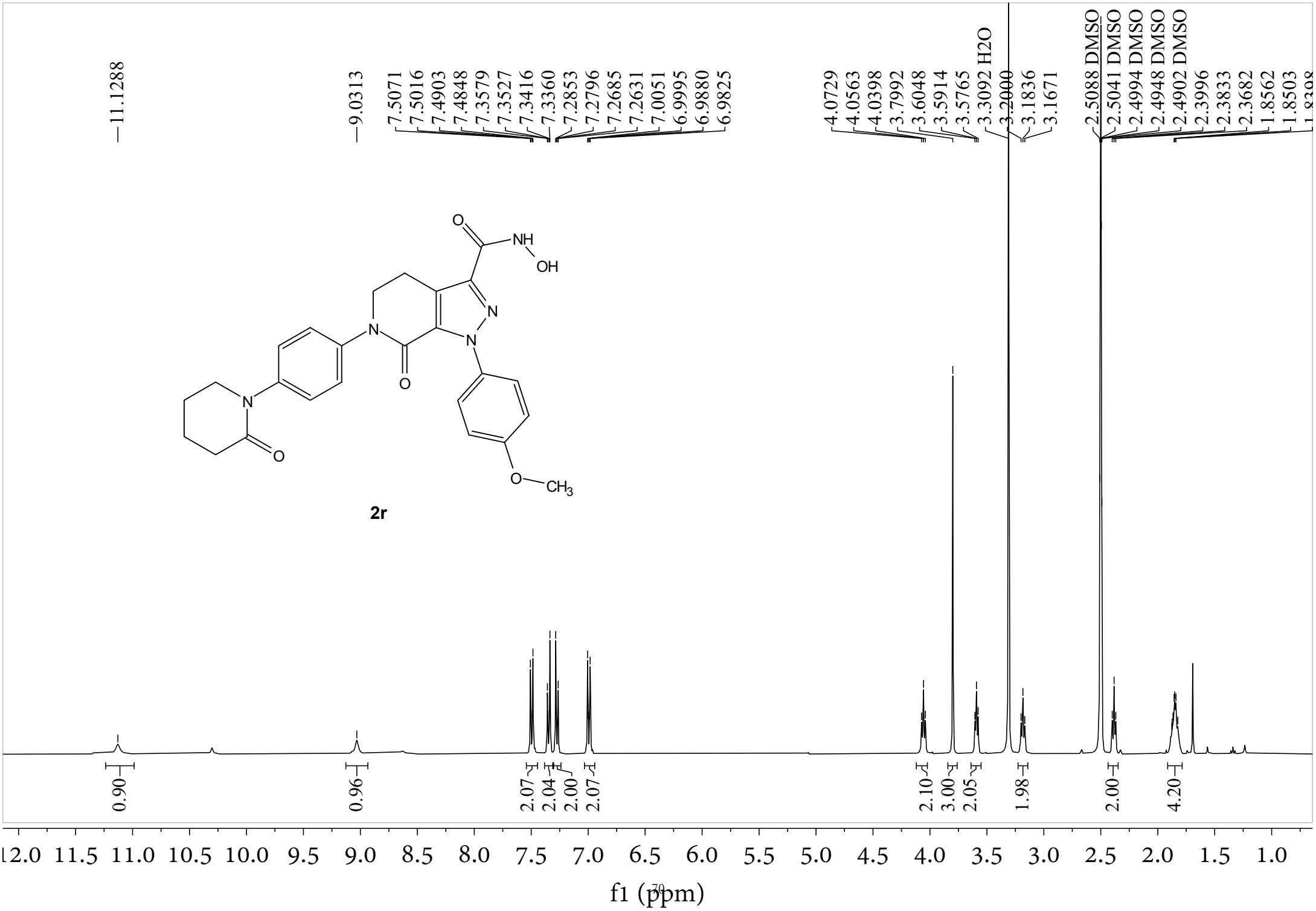
**Formula Calculator Element Limits**

| Element | Min | Max |
|---|---|---|
| C | 3 | 60 |
| H | 0 | 120 |
| O | 0 | 30 |
| N | 0 | 30 |
| Cl | 0 | 0 |
| F | 0 | 0 |
| Br | 0 | 0 |
| I | 1 | 1 |

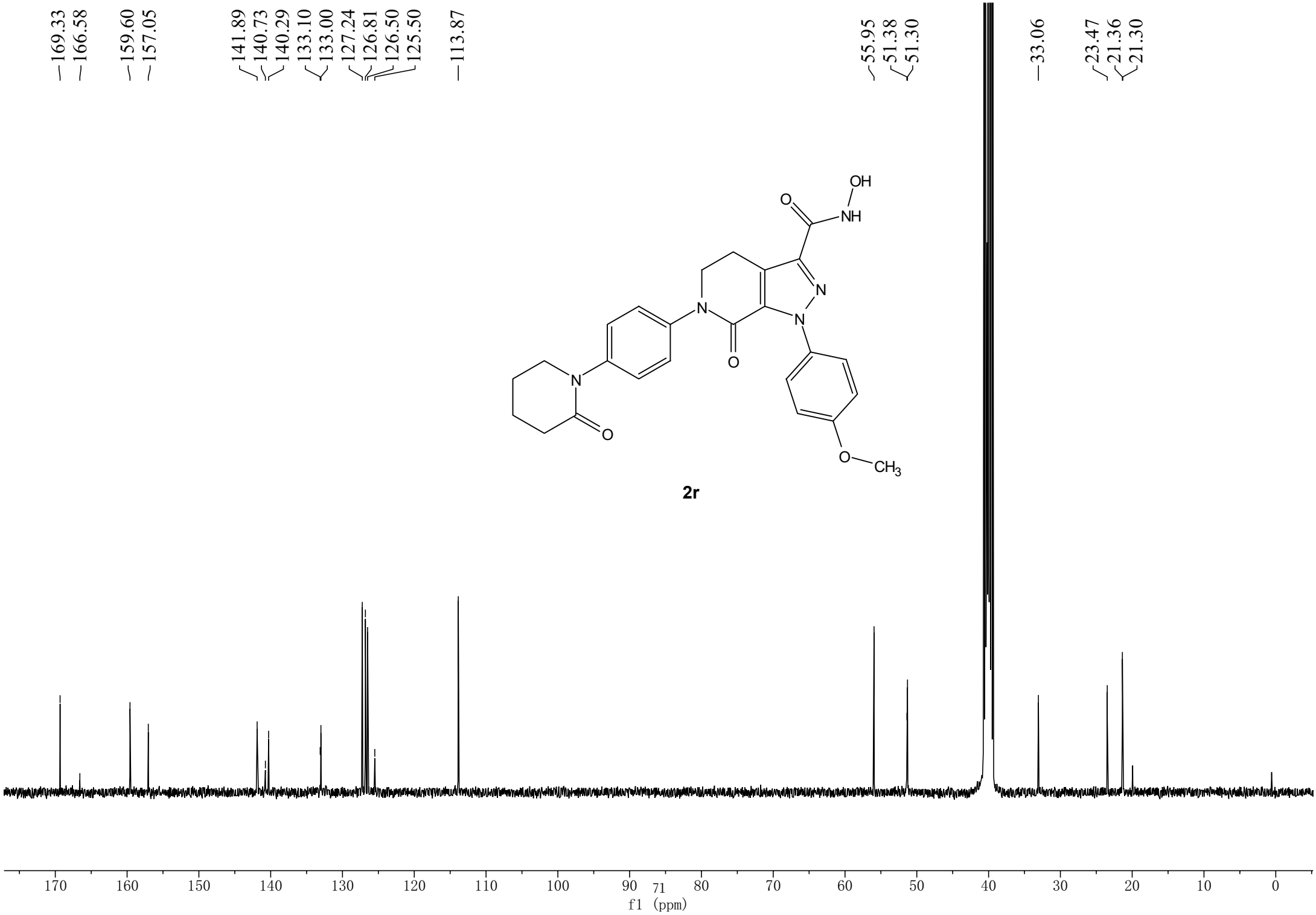
**Formula Calculator Results**

| Formula | Best | Mass | Tgt Mass | Diff (ppm) | Ion Species | Score |
|---|---|---|---|---|---|---|
| C9 H8 I N O2 | TRUE | 288.9606 | 288.96 | -2.1 | C9 H7 I N O2 | 85.37 |

--- End Of Report ---


11.1288
9.0313
7.5071
7.5016
7.4903
7.4848
7.3579
7.3527
7.3416
7.3360
7.2853
7.2796
7.2685
7.2631
7.0051
6.9995
6.9880
6.9825
4.0729
4.0563
4.0398
3.7992
3.6048
3.5914
3.5765
3.3092 H2O
3.2000
3.1836
3.1671
2.5088 DMSO
2.5041 DMSO
2.4994 DMSO
2.4948 DMSO
2.4902 DMSO
2.3996
2.3833
2.3682
1.8562
1.8503
0.90
0.96
2.07
2.04
2.00
2.07
2.10
3.00
2.05
1.98
2.00
4.20
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
f1 (ppm)

**2r**

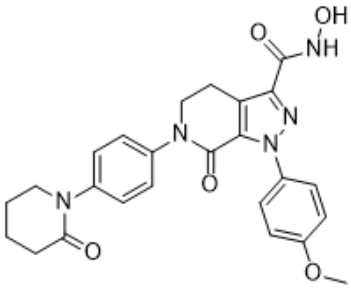

169.33
166.58
159.60
157.05
141.89
140.73
140.29
133.10
133.00
127.24
126.81
126.50
125.50
113.87
55.95
51.38
51.30
33.06
23.47
21.36
21.30
OH
O
NH
N
N
N
O
N
O
O
CH3
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

**2r**

# Qualitative Analysis Report

| | | | |
|---|---|---|---|
| **Data Filename** | H-22.d | **Sample Name** | H-22 |
| **Sample Type** | Sample | **Position** | P1-F6 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | FU100-1000.m | **Acquired Time** | 9/10/2021 8:59:44 PM |
| **IRM Calibration Status** | Success | **DA Method** | 20170311.m |
| **Comment** | | | |

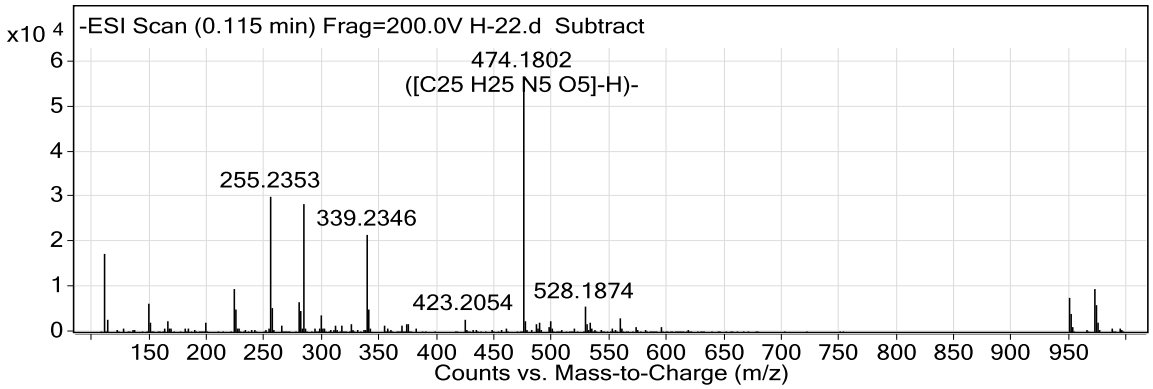
| | |
|---|---|
| **Sample Group** | **Info.** |
| **Acquisition SW Version** | 6200 series TOF/6500 series Q-TOF B.05.01 (B5125.2) |



**2r**

## User Spectra

| Fragmentor Voltage | Collision Energy | Ionization Mode |
|---|---|---|
| 200 | 0 | ESI |




**Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 110.9766 | | 17355.73 | | |
| 223.0308 | 1 | 9731.65 | | |
| 255.2353 | 1 | 30286.4 | | |
| 279.2348 | 1 | 6716.57 | | |
| 283.2662 | 1 | 28397.63 | | |
| 339.2346 | 1 | 21822.67 | | |
| 474.1802 | 1 | 54208.09 | C25 H25 N5 O5 | (M-H)- |
| 475.1833 | 1 | 14811.93 | C25 H25 N5 O5 | (M-H)- |
| 949.3683 | 1 | 7759.95 | | |
| 971.3494 | 1 | 9864.75 | | |

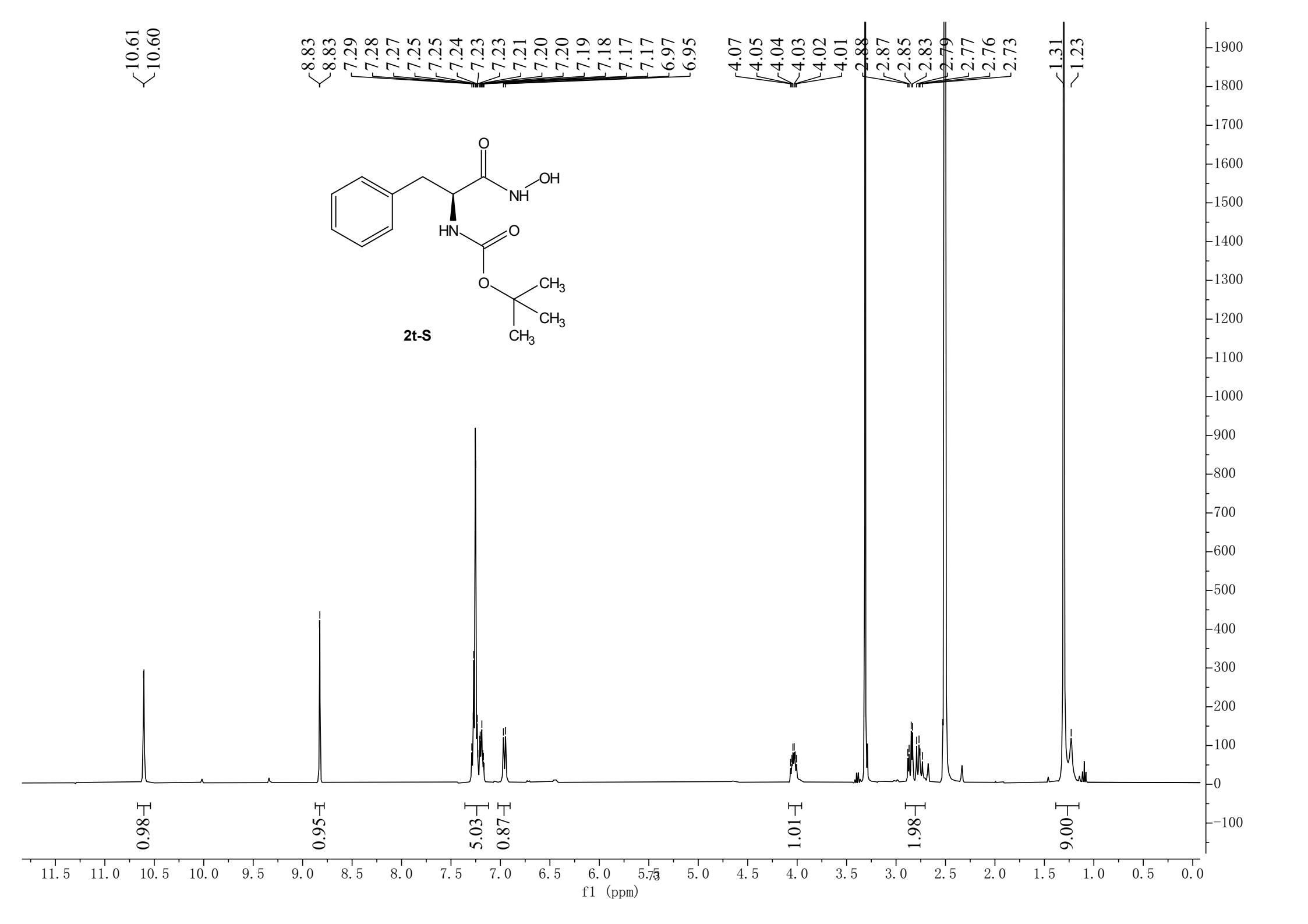
**Formula Calculator Element Limits**

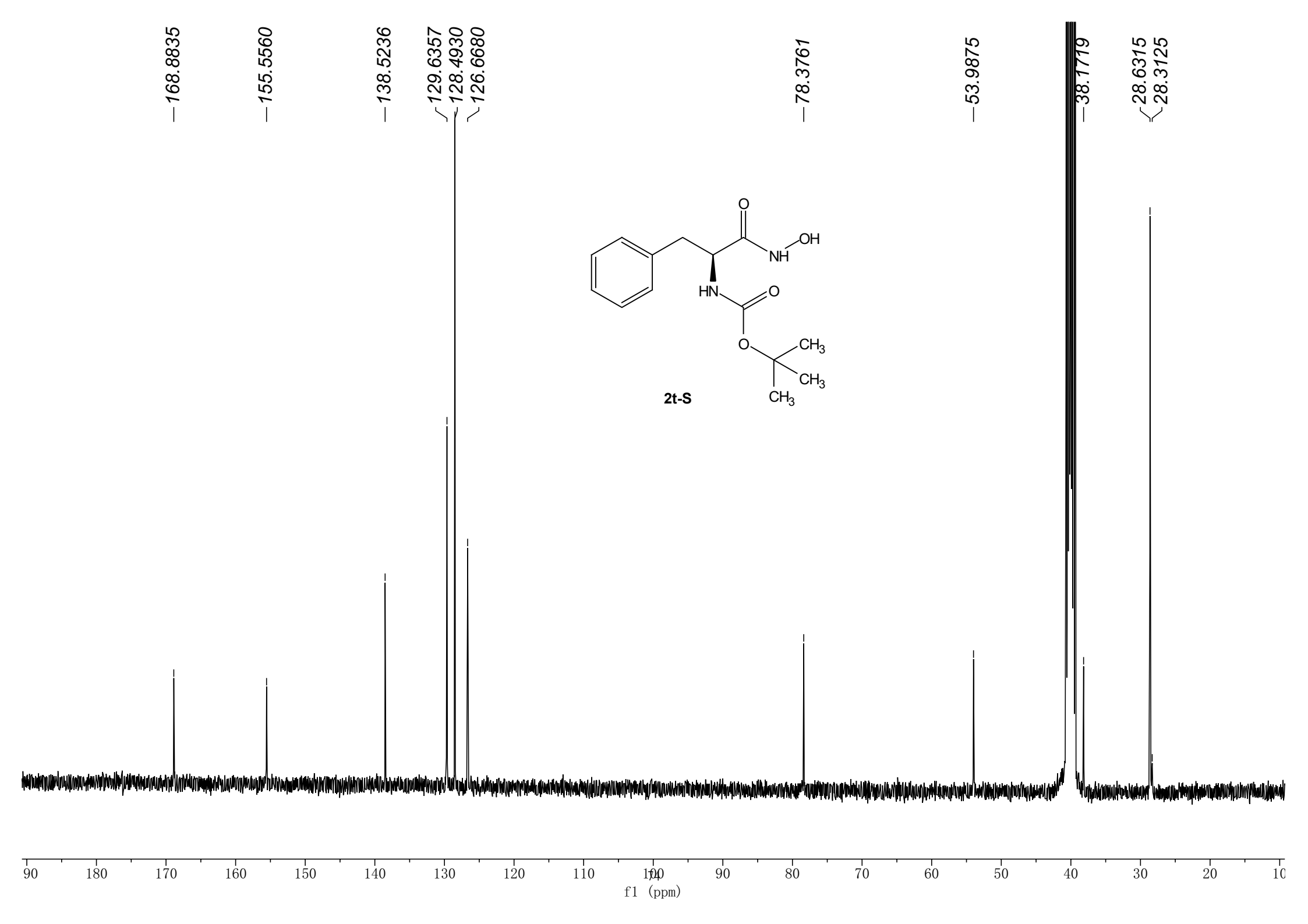
| Element | Min | Max |
|---|---|---|
| C | 3 | 60 |
| H | 0 | 120 |
| O | 0 | 30 |
| N | 0 | 30 |
| Cl | 0 | 0 |
| F | 0 | 0 |

**Formula Calculator Results**

| Formula | Best | Mass | Tgt Mass | Diff (ppm) | Ion Species | Score |
|---|---|---|---|---|---|---|
| C25 H25 N5 O5 | FALSE | 475.1875 | 475.1856 | -4.05 | C25 H24 N5 O5 | 91.69 |

--- End Of Report ---

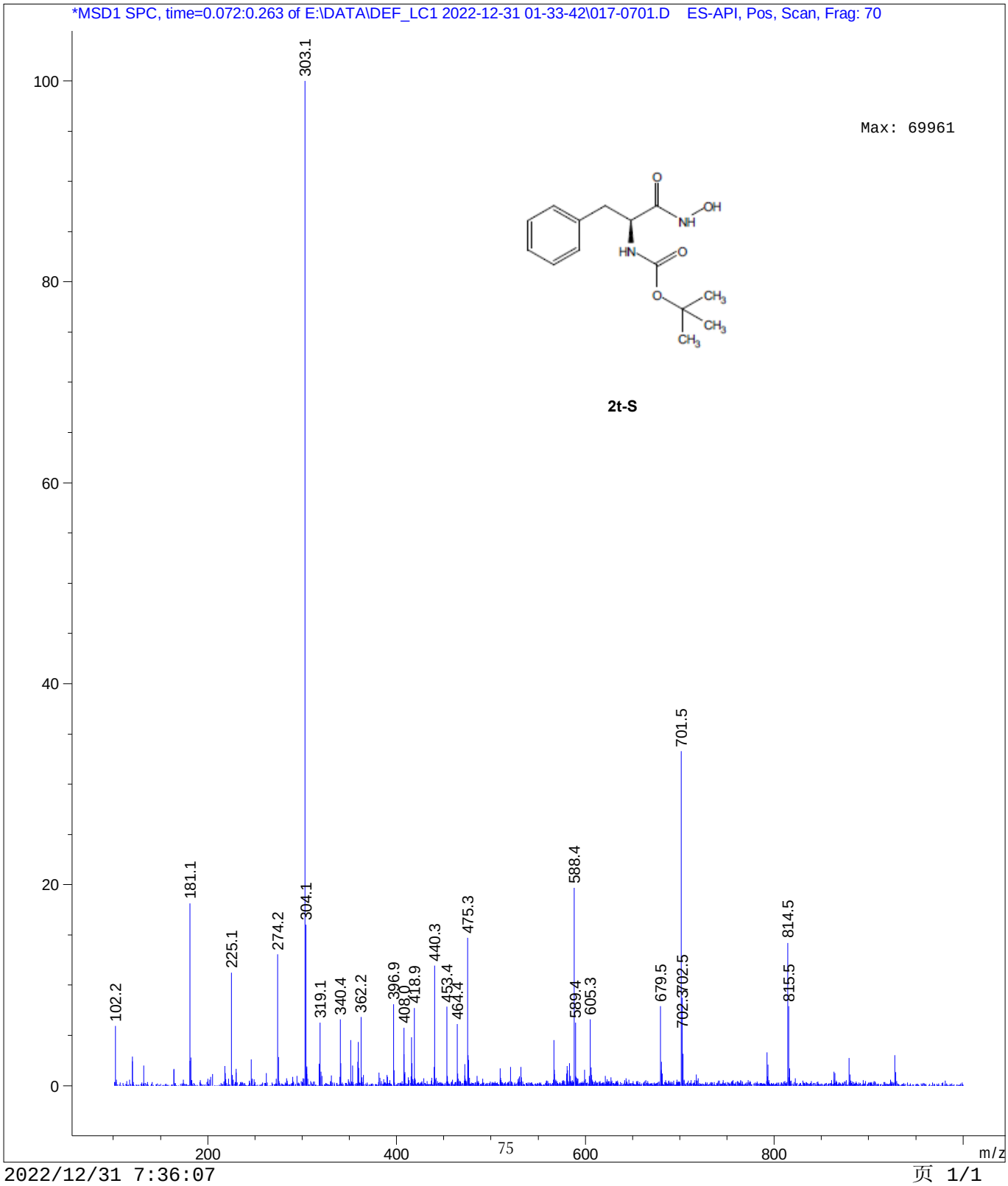

10.61
10.60
8.83
8.83
7.29
7.28
7.27
7.25
7.25
7.24
7.23
7.23
7.21
7.20
7.20
7.19
7.18
7.17
7.17
6.97
6.95
4.07
4.05
4.04
4.03
4.02
4.01
2.88
2.87
2.85
2.83
2.79
2.77
2.76
2.73
1.31
1.23
O
OH
NH
HN
O
O
CH3
CH3
CH3
2t-S
0.98
0.95
5.03
0.87
1.01
1.98
9.00
f1 (ppm)

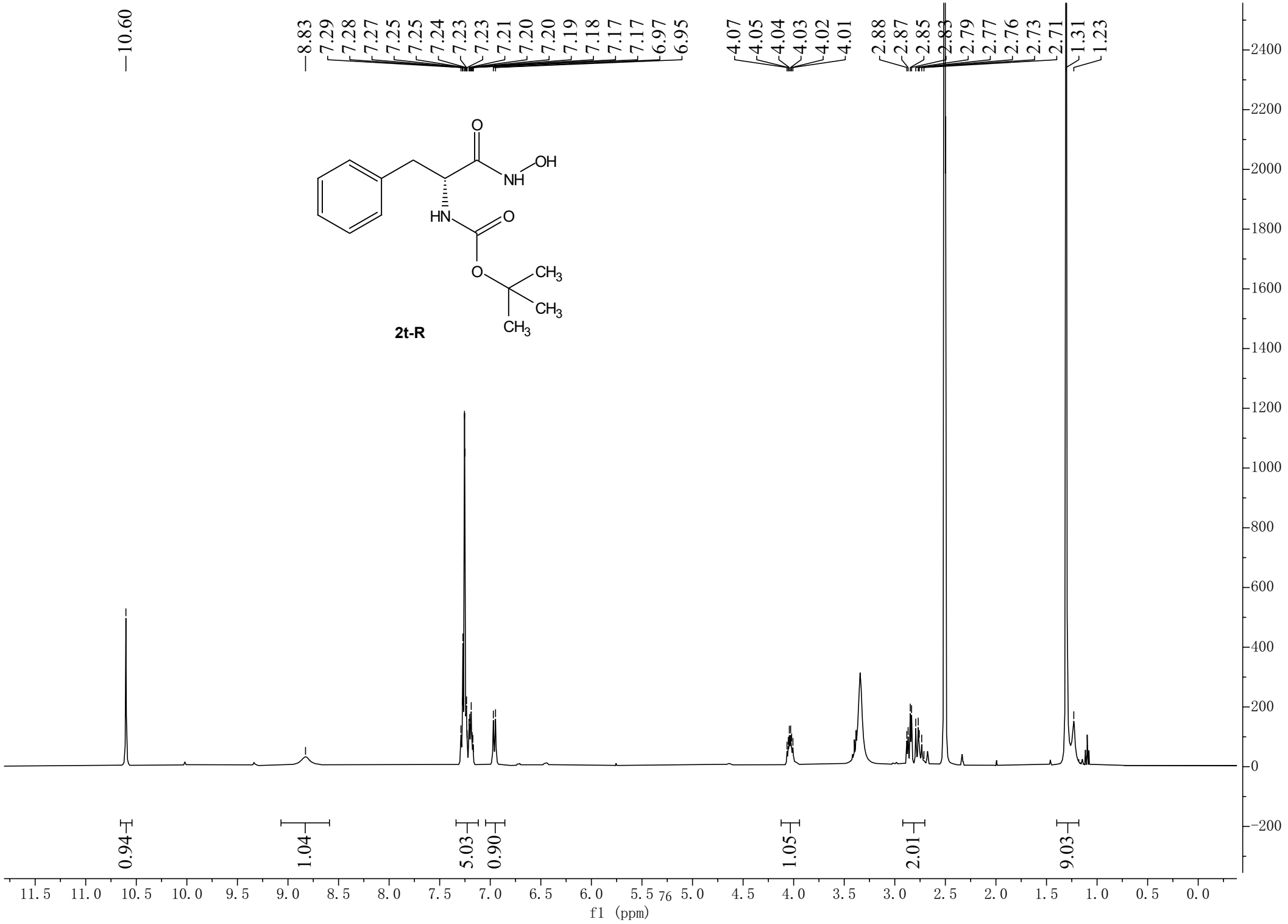

168.8835
155.5560
138.5236
129.6357
128.4930
126.6680
78.3761
53.9875
38.1719
28.6315
28.3125
O
OH
NH
HN
O
O
CH3
CH3
CH3
2t-S
90
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

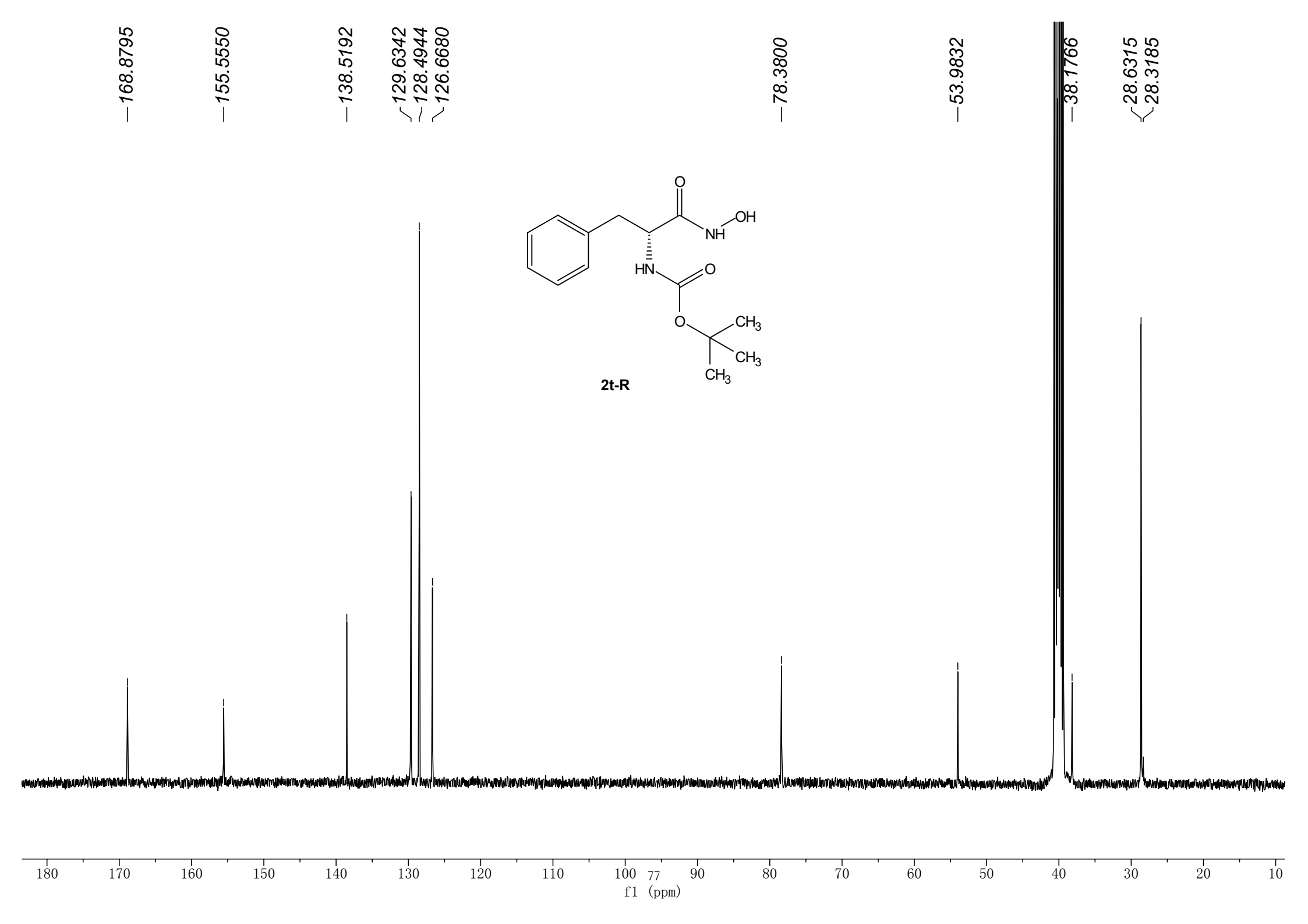
打印窗口 80: MS Spectrum
数据文件: : E:\DATA\DEF_LC1 2022-12-31 01-33-42\017-0701.D
样品名称 : PHE-NHOH-S

操作者 :
仪器 : 仪器 1
进样日期 : 2022/12/31 2:15:58
序列行 : 7
位置 : 样品瓶 17
进样次数 : 1
进样量 : 5.000 µl
采集方法 : E:\DATA\DEF_LC1 2022-12-31 01-33-42\100TO1000P_AND_N.M
最后修改 : 2021/4/17 14:18:09
分析方法 : C:\CHEM32\1\METHODS\100TO1000P_AND_N.M
最后修改 : 2022/12/1 16:12:04
(调用后修改)
方法信息 : 2080F

MS Spectrum




10.60
8.83
7.29
7.28
7.27
7.25
7.25
7.24
7.23
7.23
7.21
7.20
7.20
7.19
7.18
7.17
7.17
6.97
6.95
4.07
4.05
4.04
4.03
4.02
4.01
2.88
2.87
2.85
2.83
2.79
2.77
2.76
2.73
2.71
1.31
1.23
O
OH
NH
HN
O
O
CH3
CH3
CH3
2t-R
0.94
1.04
5.03
0.90
1.05
2.01
9.03
f1 (ppm)


168.8795
155.5550
138.5192
129.6342
128.4944
126.6680
78.3800
53.9832
38.1766
28.6315
28.3185
O
OH
NH
HN
O
O
CH3
CH3
CH3
2t-R
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
77
f1 (ppm)

打印窗口 80: MS Spectrum
数据文件: : E:\DATA\DEF_LC1 2022-12-31 01-33-42\018-0801.D
样品名称 : PHE-NHOH-R

操作者 : 序列行 : 8
仪器 : 仪器 1 位置 : 样品瓶 18
进样日期 : 2022/12/31 2:22:36 进样次数 : 1
进样量 : 5.000 µl
采集方法 : E:\DATA\DEF_LC1 2022-12-31 01-33-42\100TO1000P_AND_N.M
最后修改 : 2021/4/17 14:18:09
分析方法 : C:\CHEM32\1\METHODS\100TO1000P_AND_N.M
最后修改 : 2022/12/1 16:12:04
(调用后修改)
方法信息 : 2080F

MS Spectrum