

Supplementary Information

Synthesis of N-aryl derived formamides using triflic anhydride

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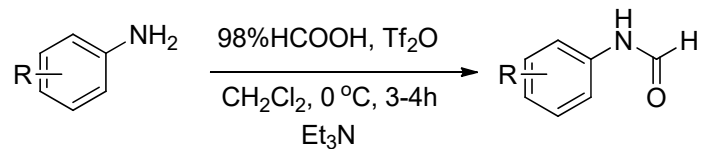
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General Information

All commercially available reagents were used as received without further purification. The solvents were purified and dried by standard procedures prior to use. Reaction progress was monitored by MERCK thin layer chromatography (TLC) performed on aluminium plates coated with silica gel 60 F254. High resolution mass spectra were recorded on a Micromass Q-TOF micromass spectrometer using electron spray ionization mode. ¹H NMR and ¹³C NMR spectra were recorded on a Bruker AMX 400 MHz and 100 MHz spectrometer, respectively in DMSO-*d*₆ using TMS as internal standard. Chemical shifts (δ) for ¹H and ¹³C are given in ppm and coupling constants (J) quoted in Hz. ¹H NMR splitting patterns were designated as s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad signal. The RP-HPLC analysis of compounds was carried out by using an Agilent instrument at λ = 254 nm; column: Eclipse XDB-C18, pore size=5μm, diameter x length = 4.6 x 150mm. For purification of products, column chromatography was performed on silica gel (100-200 mesh) using ethyl acetate and hexane mixture as eluent. Evaporation of solvents was performed under reduced pressure with a Büchi rotary evaporator. Melting points were determined in an open capillary using VEEGO, model: VMP-DS.

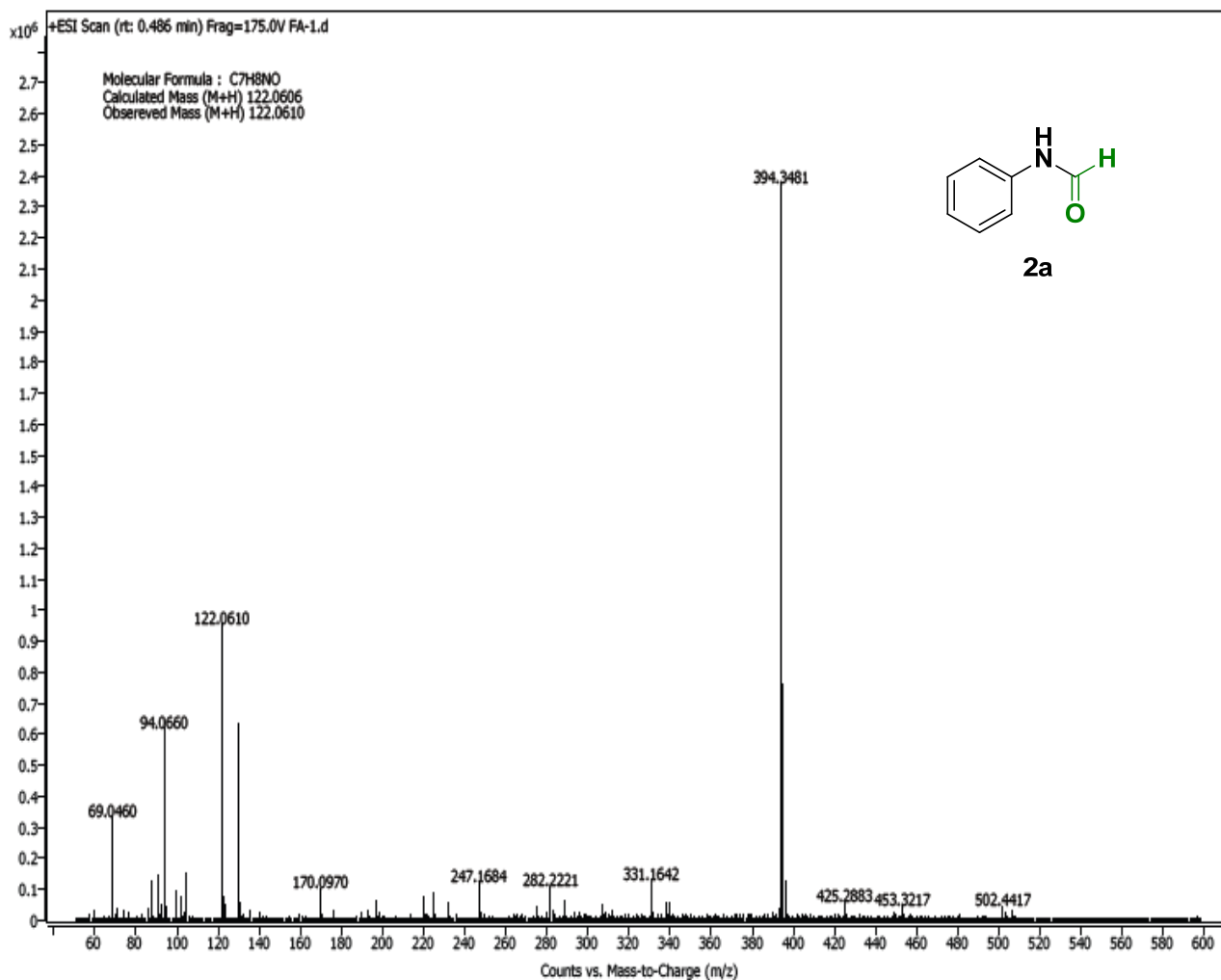
General procedure for formamide



Scheme: Synthesis of formamides mediated by Tf₂O

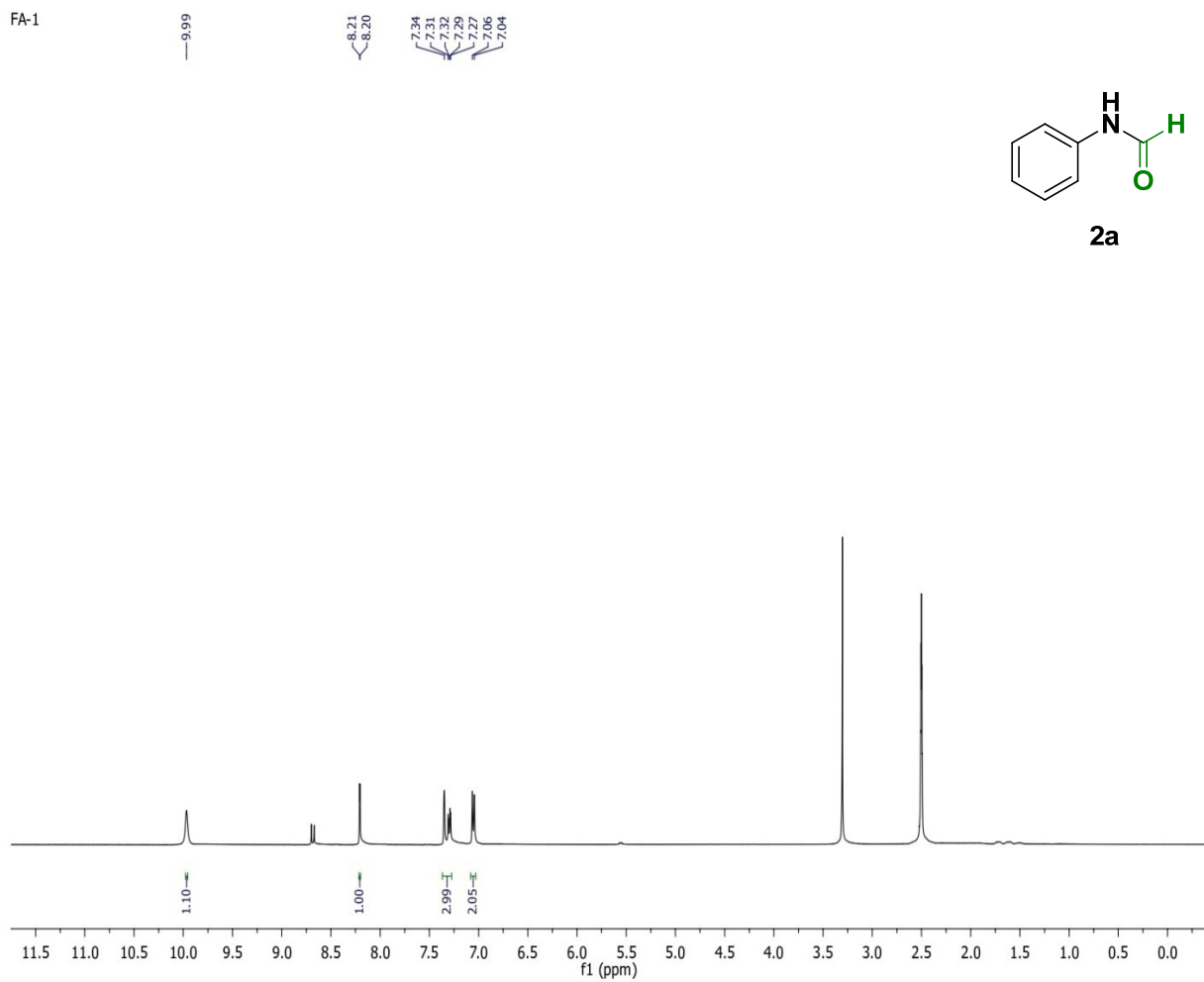
To a stirred solution of 98% Formic acid in DCM was initially cooled to 0 °C. Tf₂O (2.0 equiv) and Et₃N (2.0 equiv) were added. And the reaction mixture was stirred for 15-20 min. Later, aniline (1.0 equiv) was added and the reaction mixture was stirred for another 4 hr. The course of the reaction was monitored by TLC. The solvent was vacuum-evaporated. The residue was diluted with ethyl acetate (EtOAc). The organic layer was washed with 10% HCl, followed by 10% Na₂CO₃ and then brine. The organic layer was dried over anhydrous sodium sulfate (Na₂SO₄), filtered and concentrated under reduced pressure to obtain the desired product. The residue was purified by column chromatography (silica gel 100-200 mesh) with 3:7 ethyl acetate and hexane to afford corresponding formamide.

Spectrum Plot Report



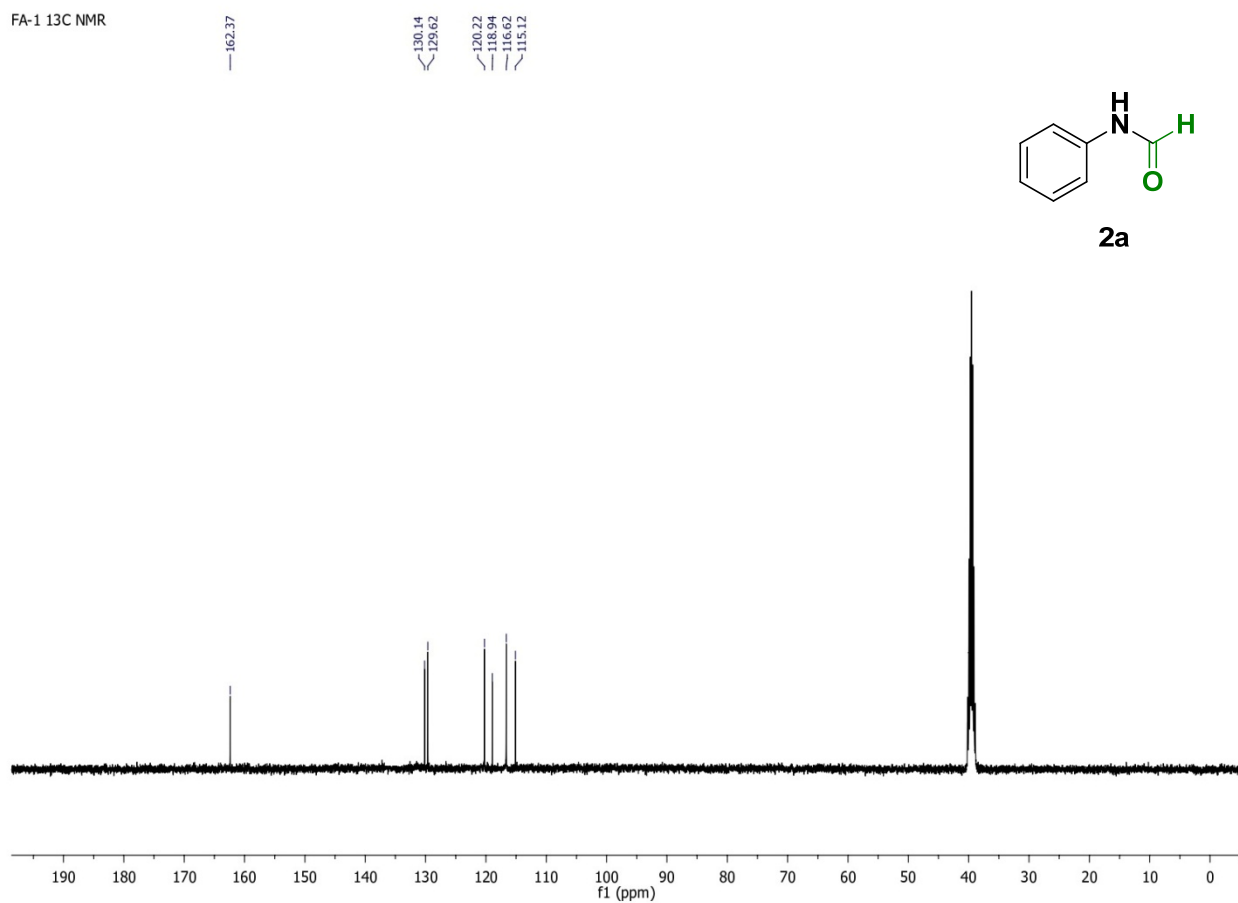
HRMS SPECTRUM OF COMPOUND 2a

FA-1



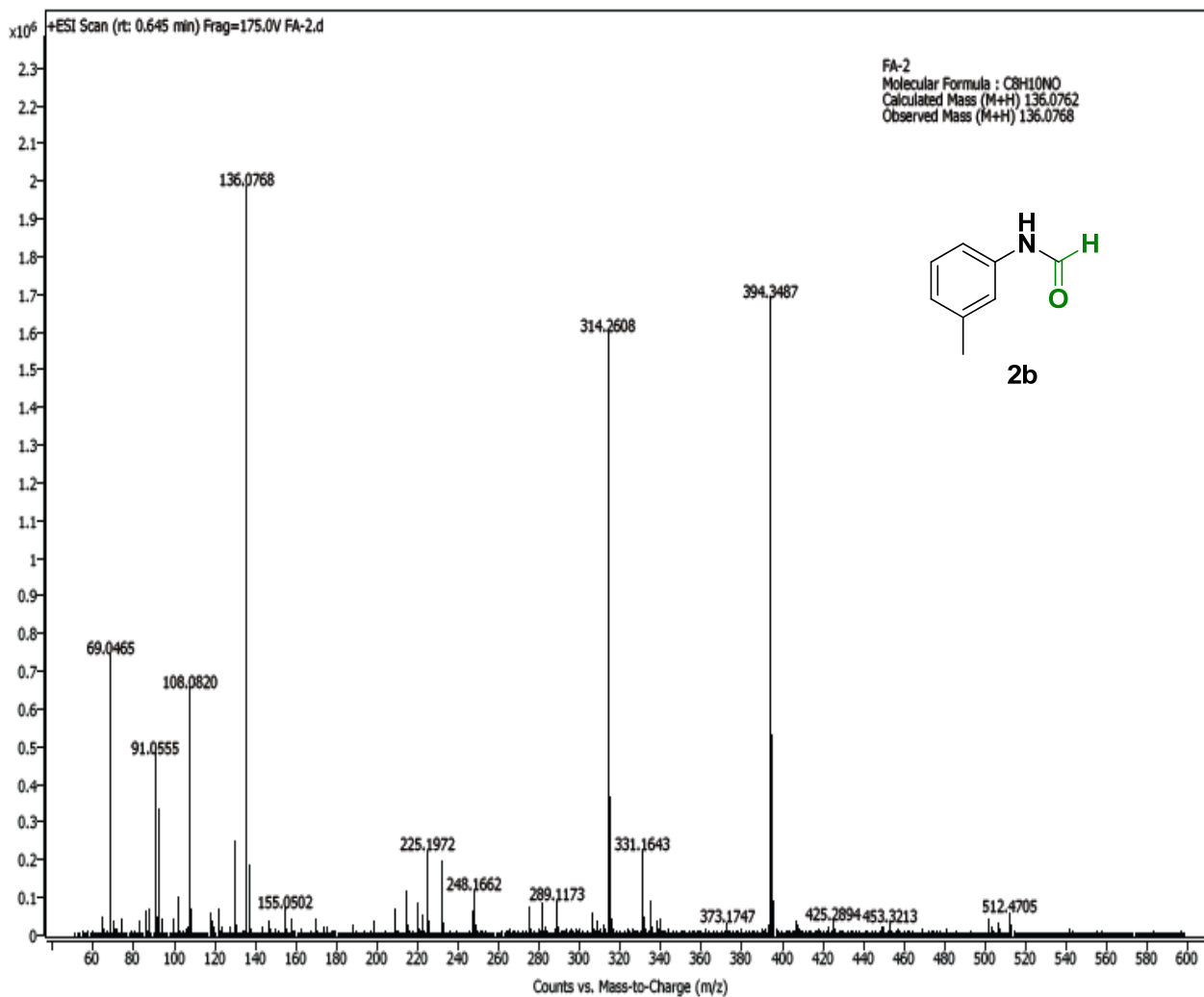
¹H NMR SPECTRUM OF COMPOUND 2a

FA-1 13C NMR



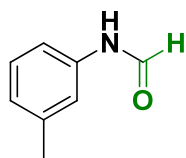
¹³C NMR SPECTRUM OF COMPOUND 2a

Spectrum Plot Report

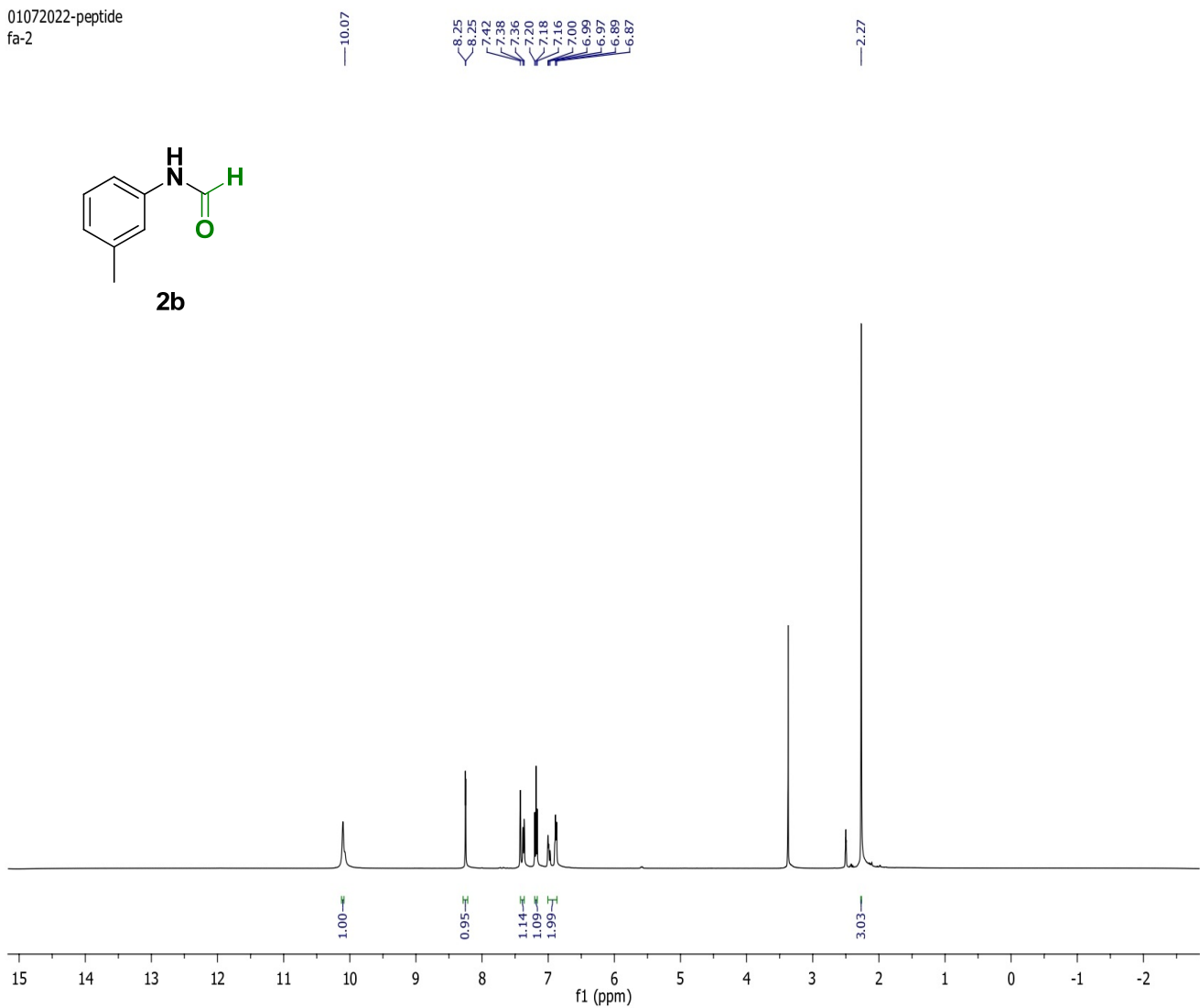


HRMS SPECTRUM OF COMPOUND 2b

01072022-peptide
fa-2

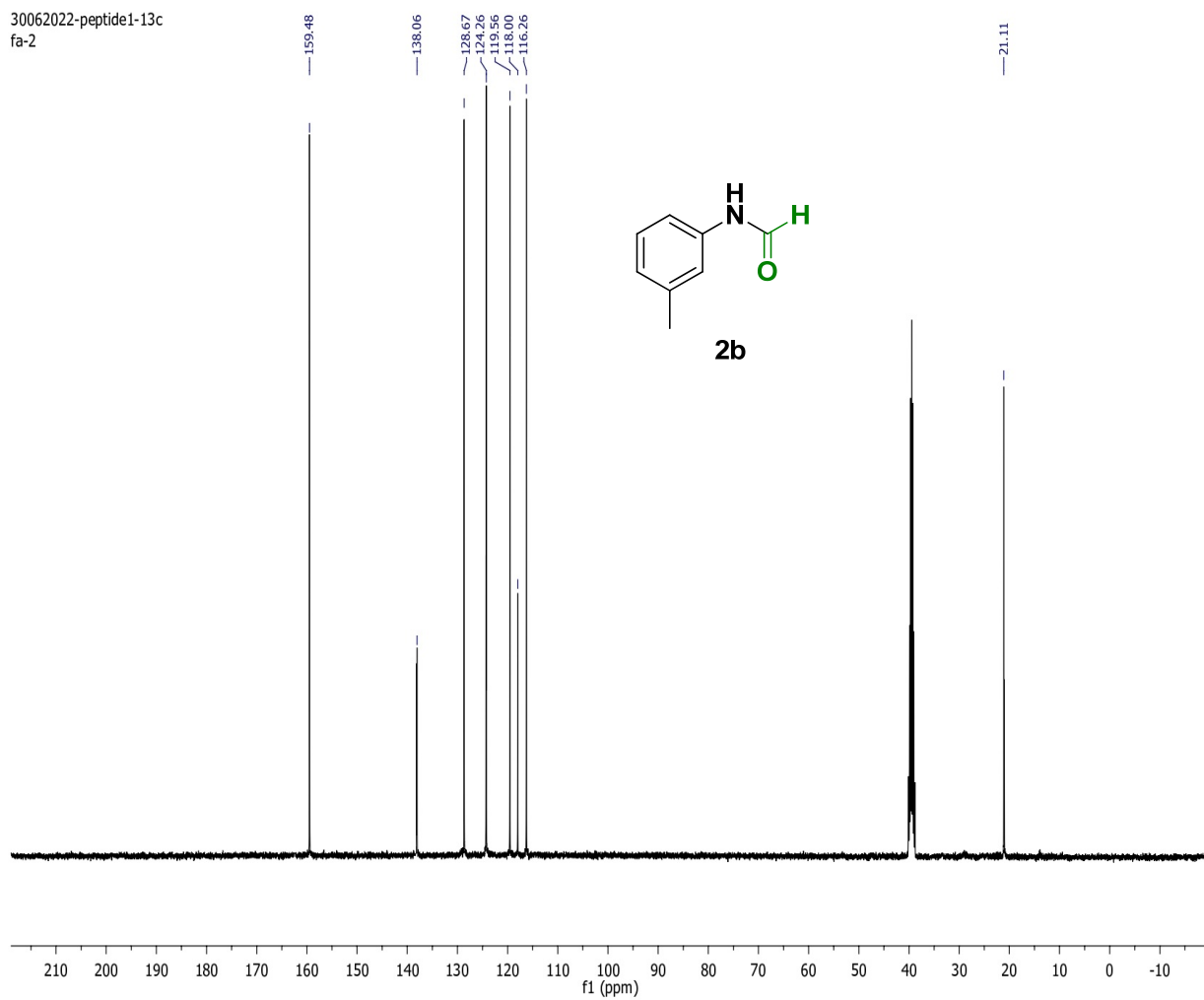


2b



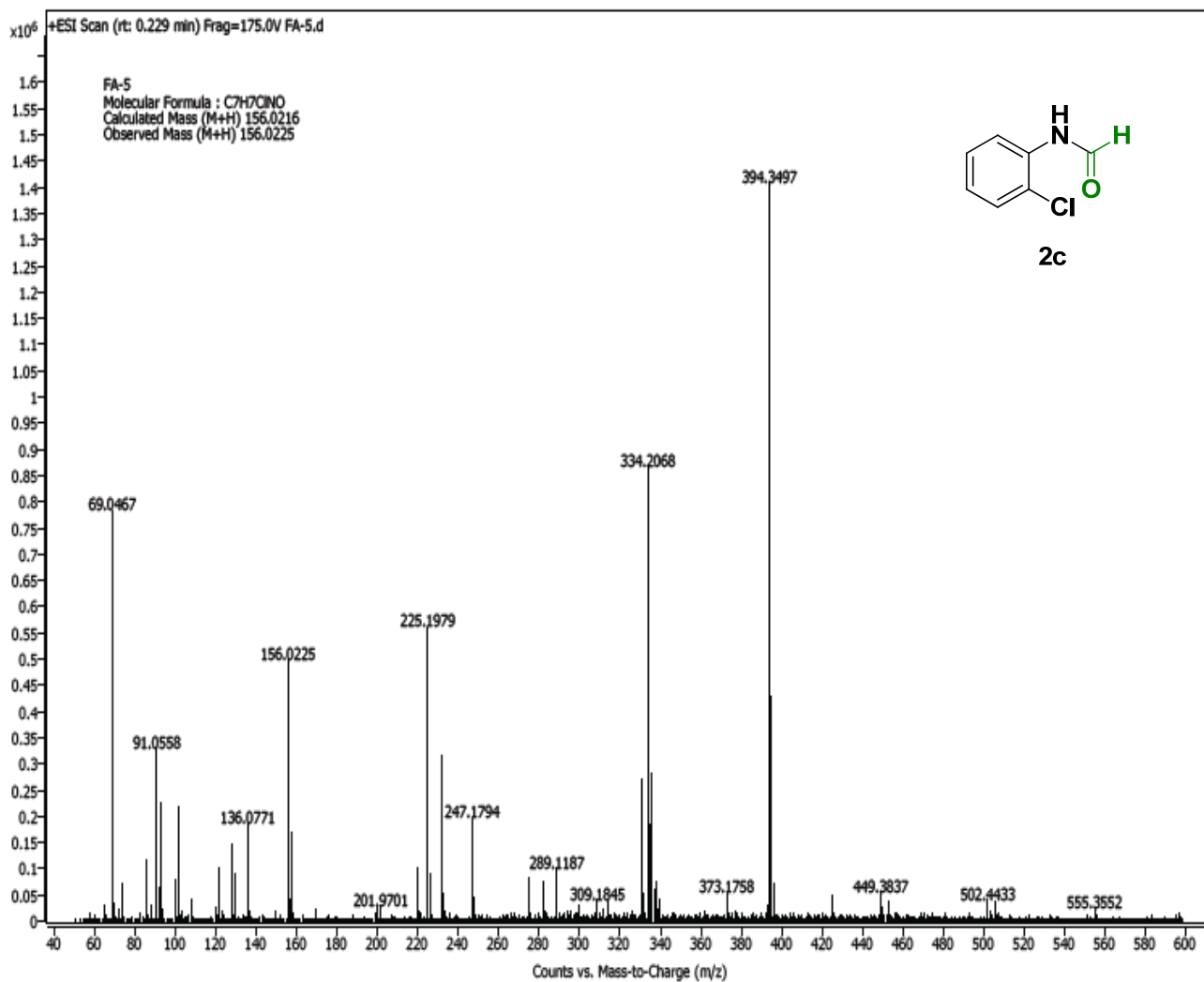
¹H NMR SPECTRUM OF COMPOUND 2b

30062022-peptide1-13c
fa-2



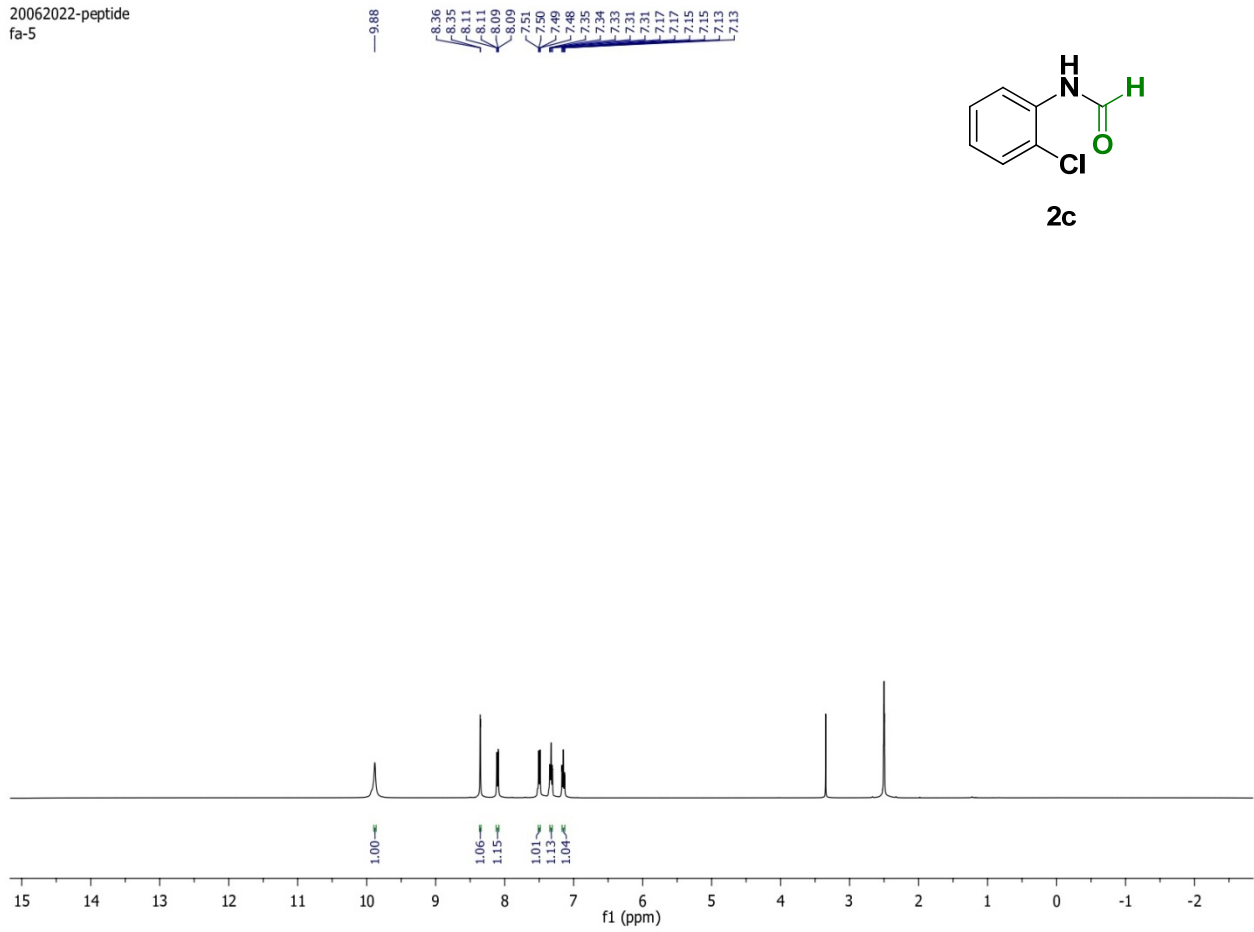
¹³C NMR SPECTRUM OF COMPOUND 2b

Spectrum Plot Report

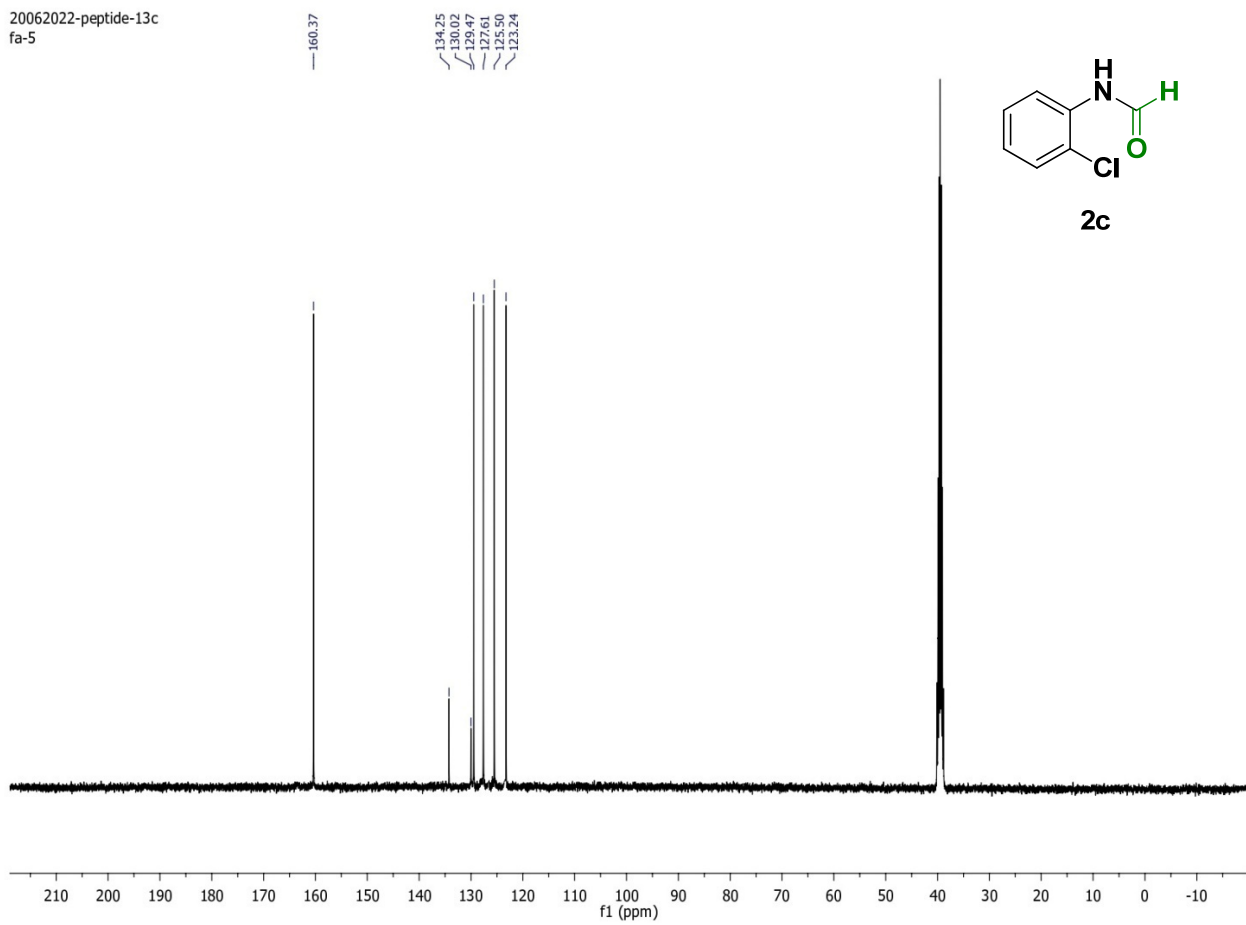


HRMS SPECTRUM OF COMPOUND 2c

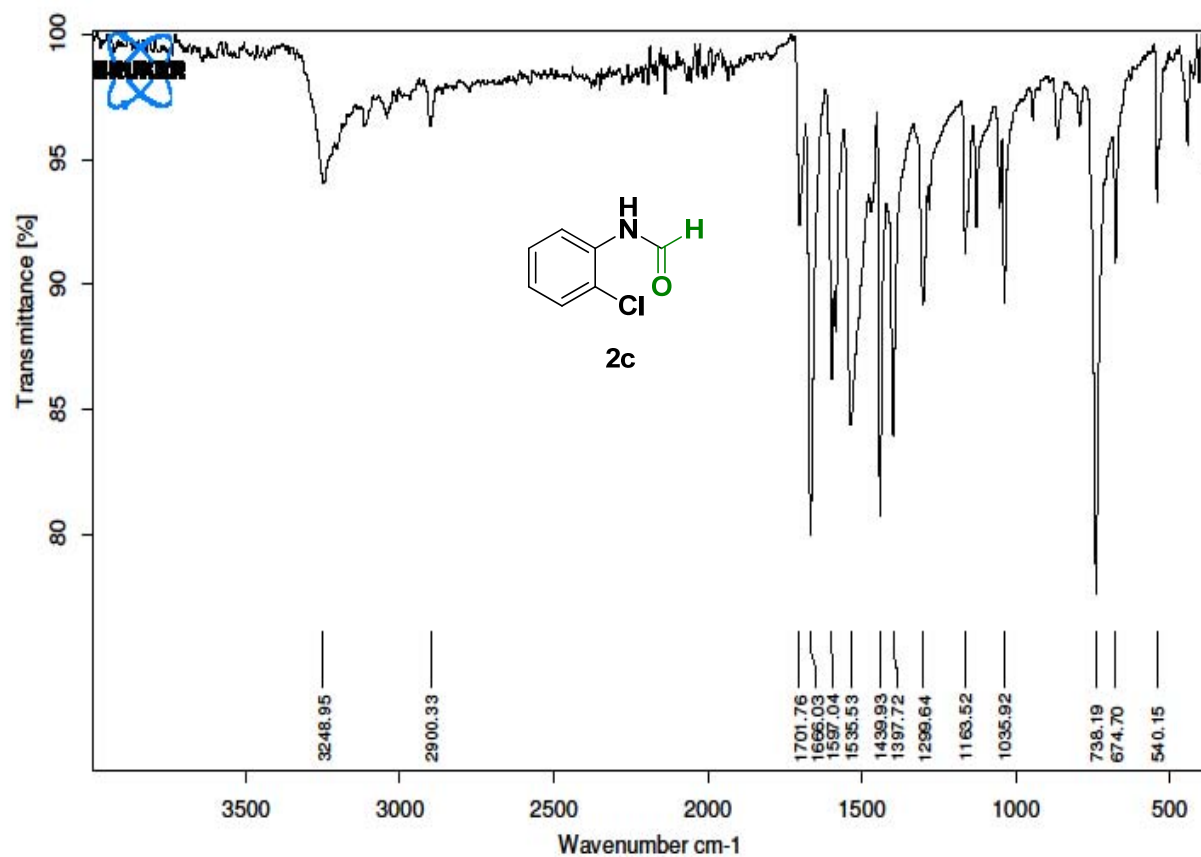
20062022-peptide
fa-5



20062022-peptide-13c
fa-5

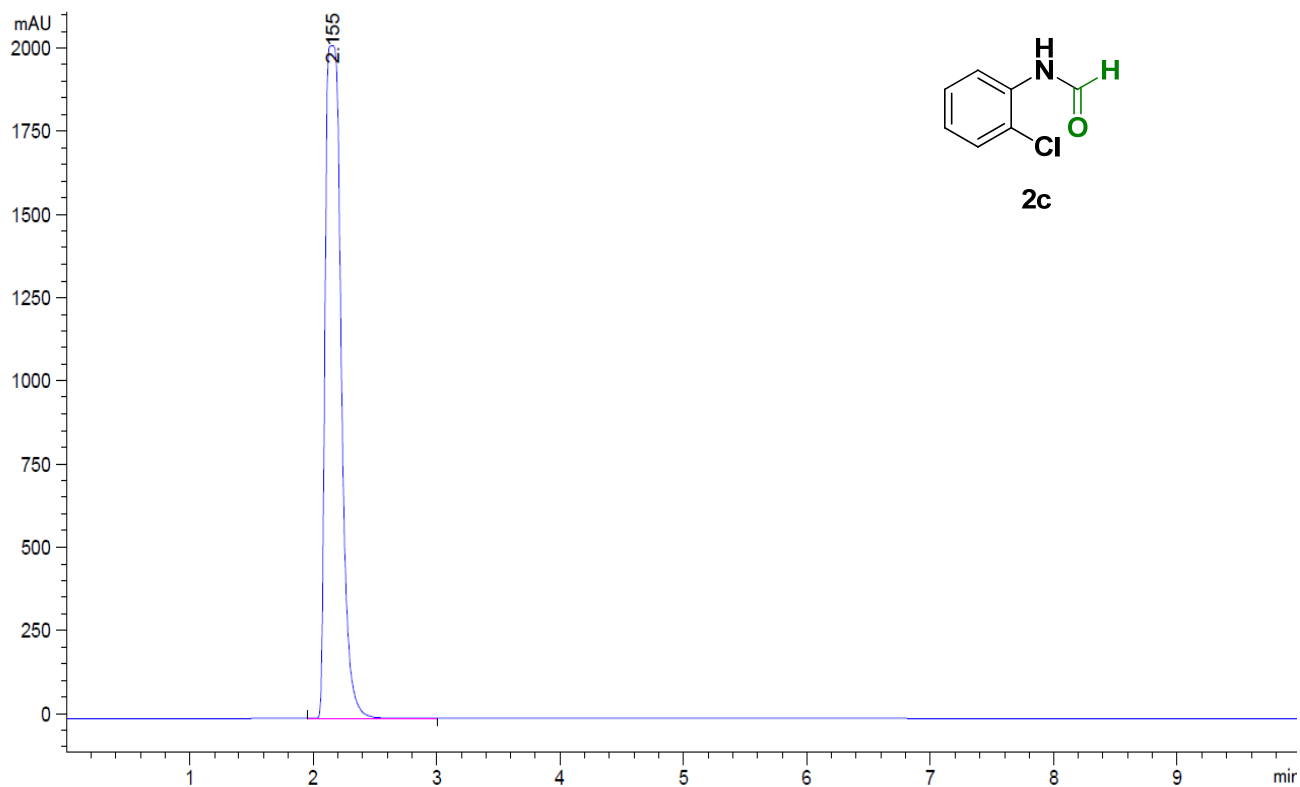


¹³C NMR SPECTRUM OF COMPOUND 2c



E:\WS\Chetan\FA-5.1	FA-5	Instrument type and / or accessory	27/08/2022
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IR-SPECTRA OF COMPOUND 2c



HPLC OF COMPOUND 2c

PEAK INFORMATION OF COMPOUND 2c

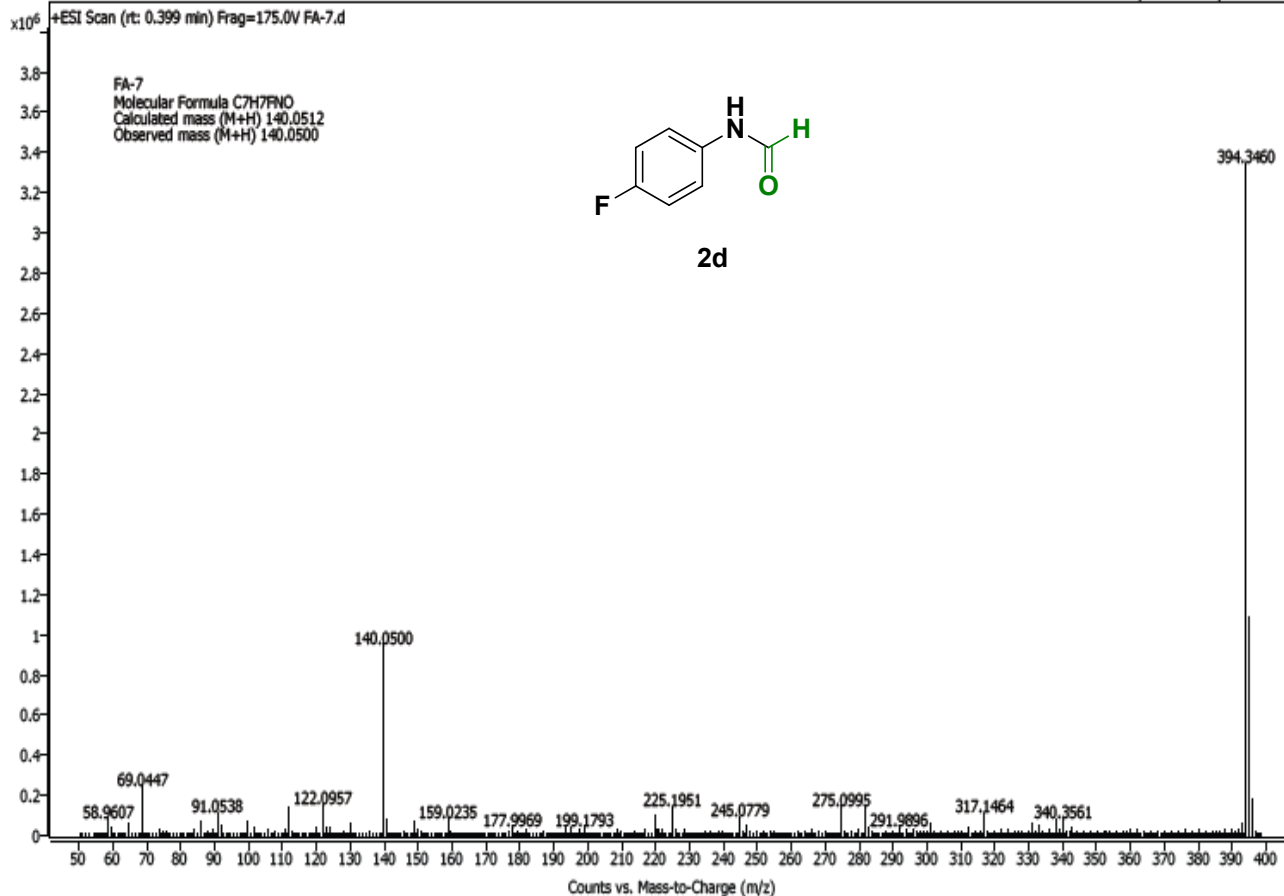
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Spectrum Plot Report

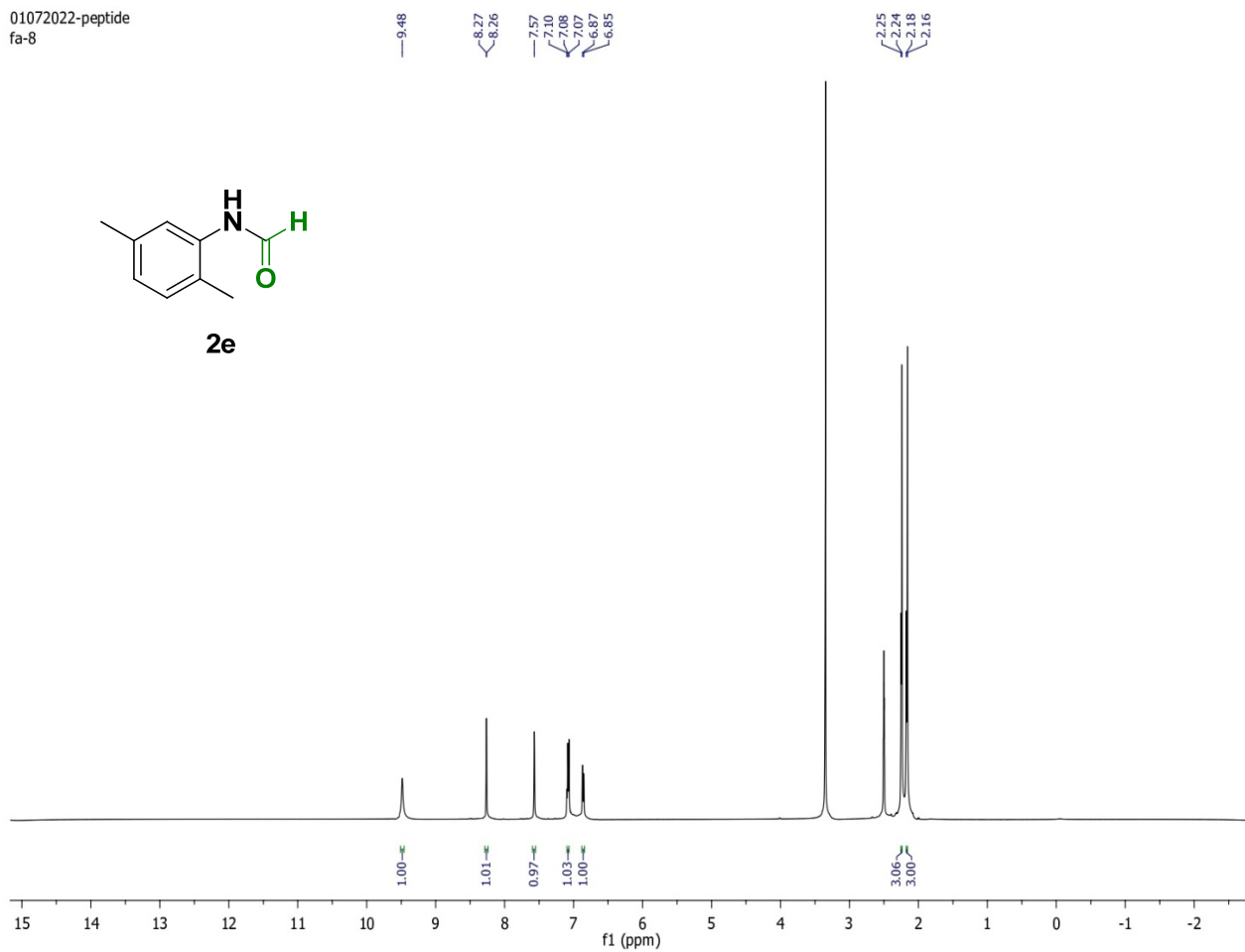


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		Comment	Acq. Time (Local)
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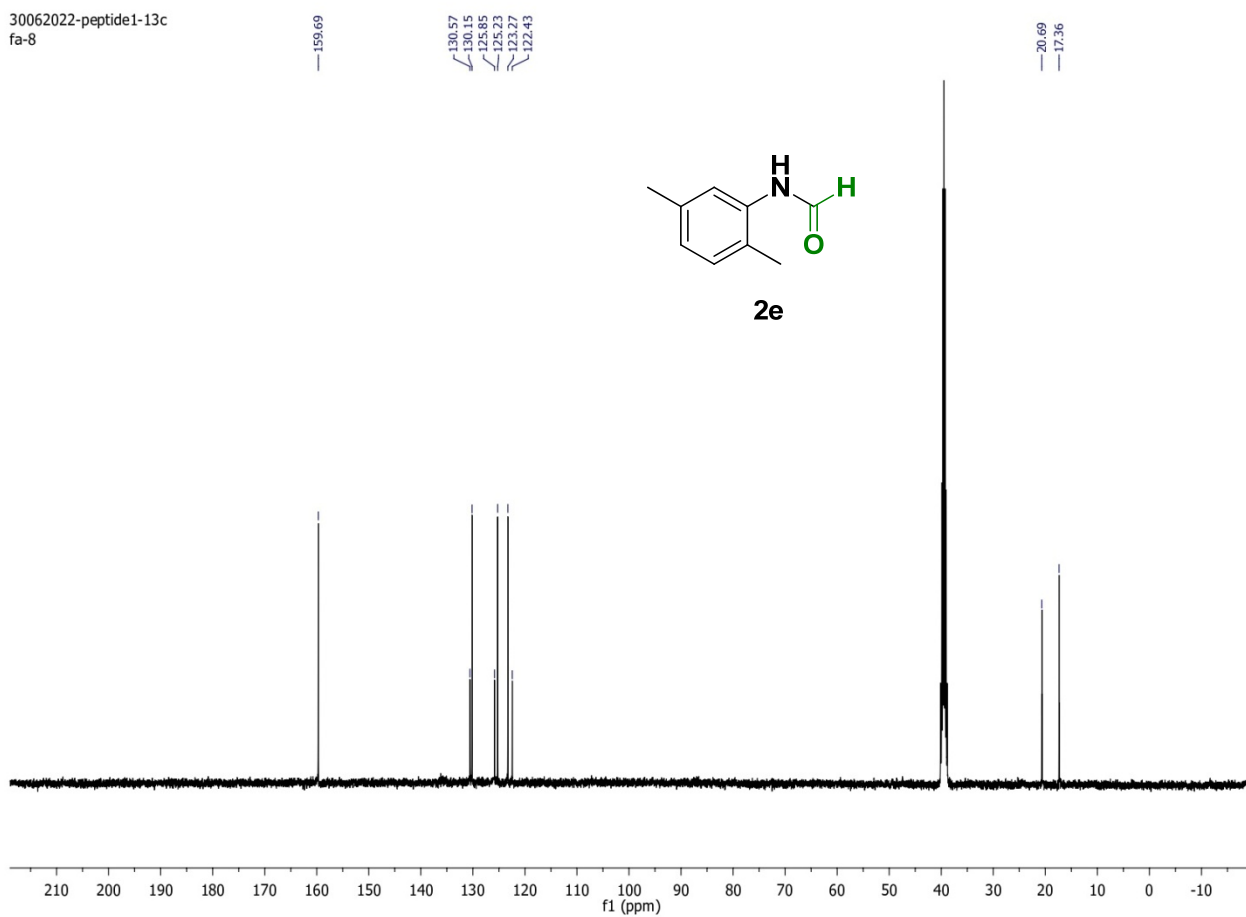
HRMS SPECTRUM OF COMPOUND 2d

01072022-peptide
fa-8

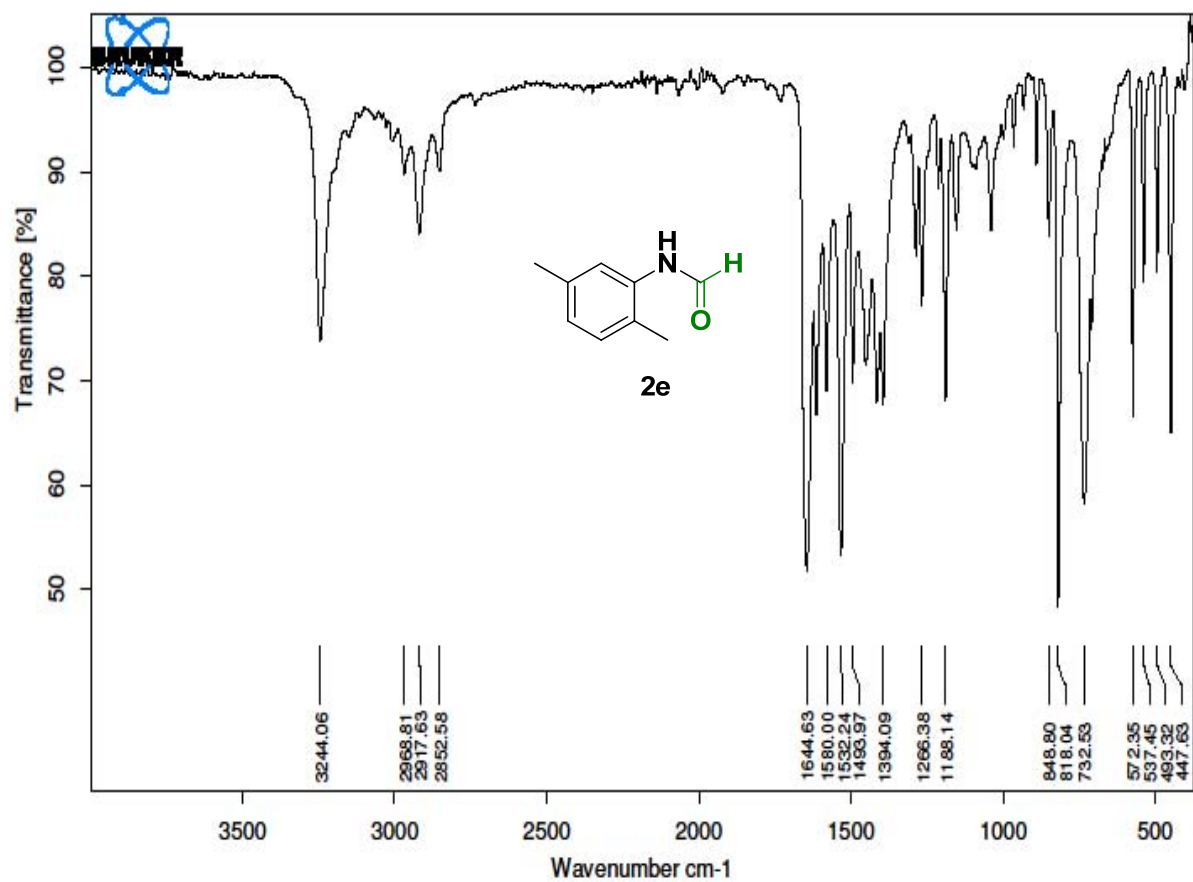


¹H NMR SPECTRUM OF COMPOUND 2e

30062022-peptide1-13c
fa-8

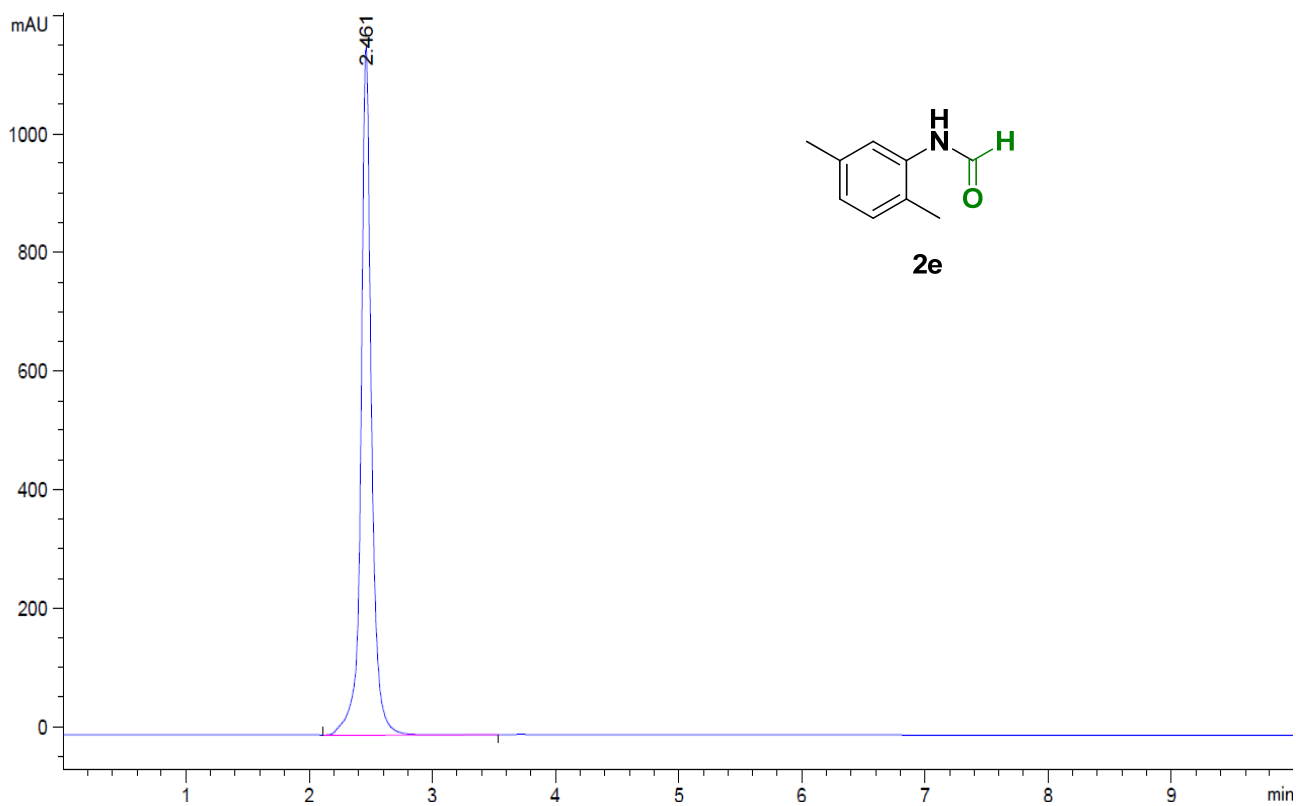


^{13}C NMR SPECTRUM OF COMPOUND 2e



E:\WS\Chetani\FA-5.7	FA-8	Instrument type and / or accessory	27/08/2022
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IR-SPECTRA OF COUMPOUND 2e



HPLC OF COMPOUND 2e

PEAK INFORMATION OF COMPOUND 2e

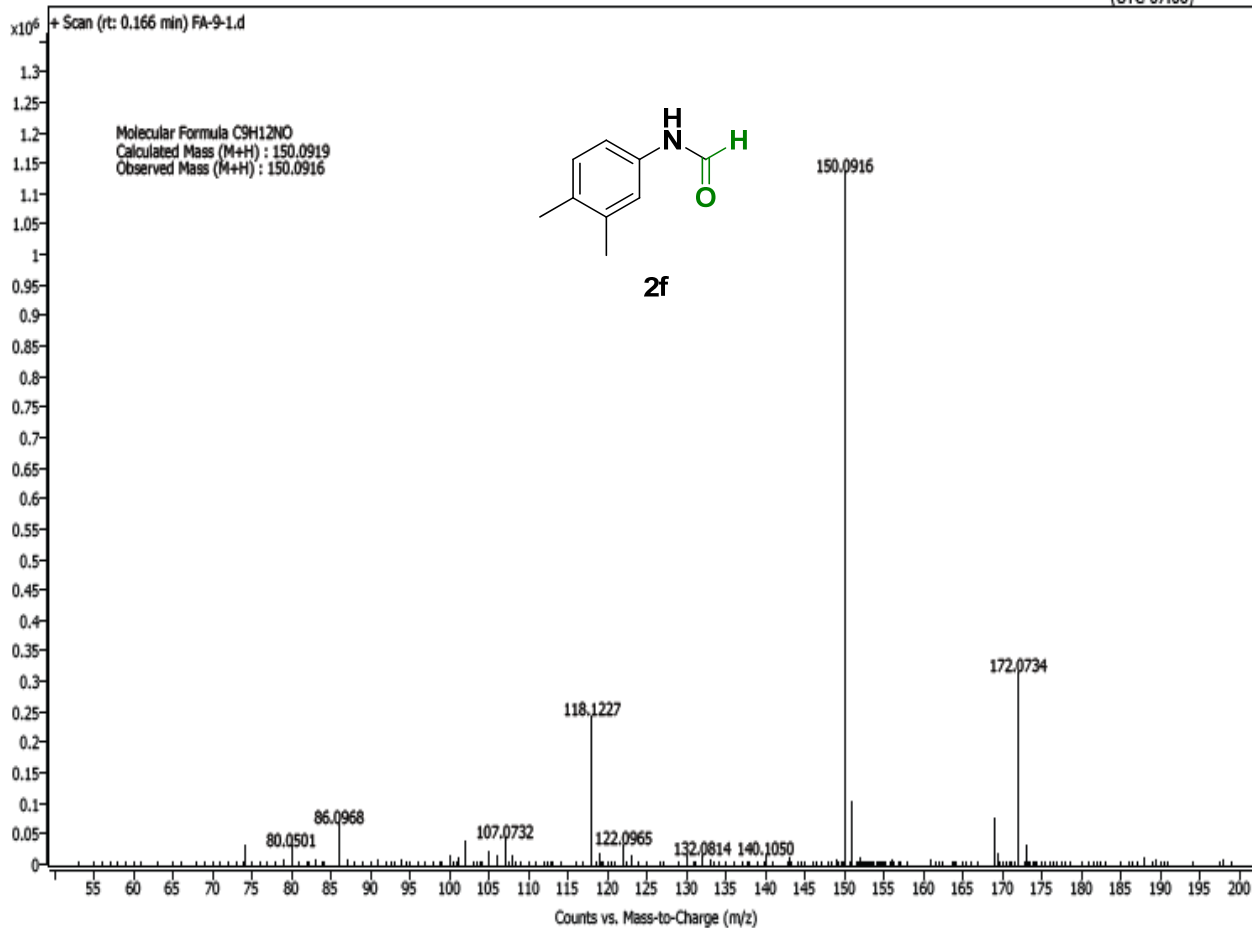
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1	2.461	0.0874	6917.81836	1162.57227	100.0000

HPLC condition: RP-HPLC profile of 2e (method: water-acetonitrile (20-80%) in 10 min; detection wavelength at $\lambda = 254\text{nm}$; flow rate : 0.7 mL/min; column: Eclipse XDB-C18, pore size=5 μm , diameter x length = 4.6 x 150mm).

Spectrum Plot Report

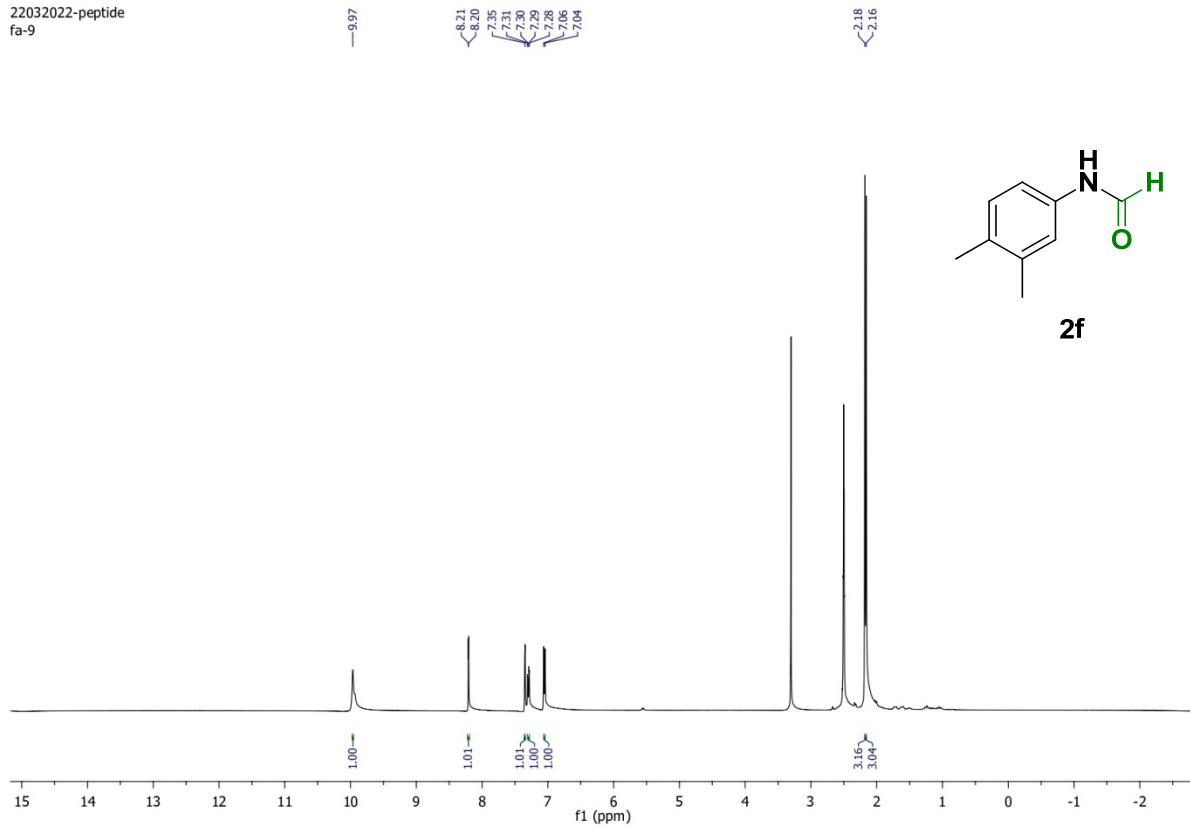


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HRMS SPECTRUM OF COMPOUND 2f

22032022-peptide
fa-9



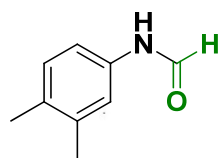
¹H NMR SPECTRUM OF COMPOUND 2f

22032022-peptide-13c
fa-9

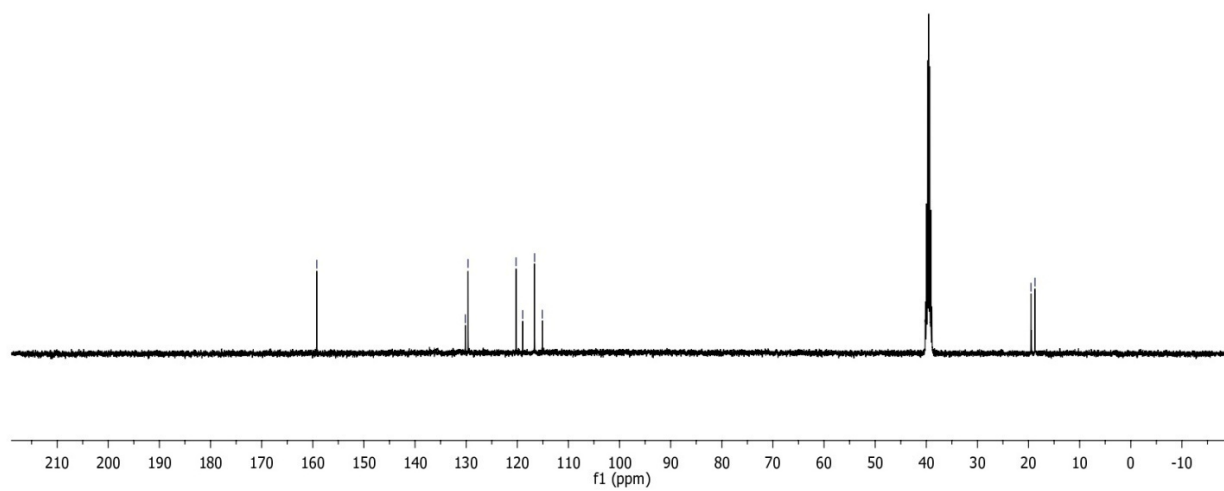
159.22

130.16
129.63
120.74
118.94
116.62
115.10

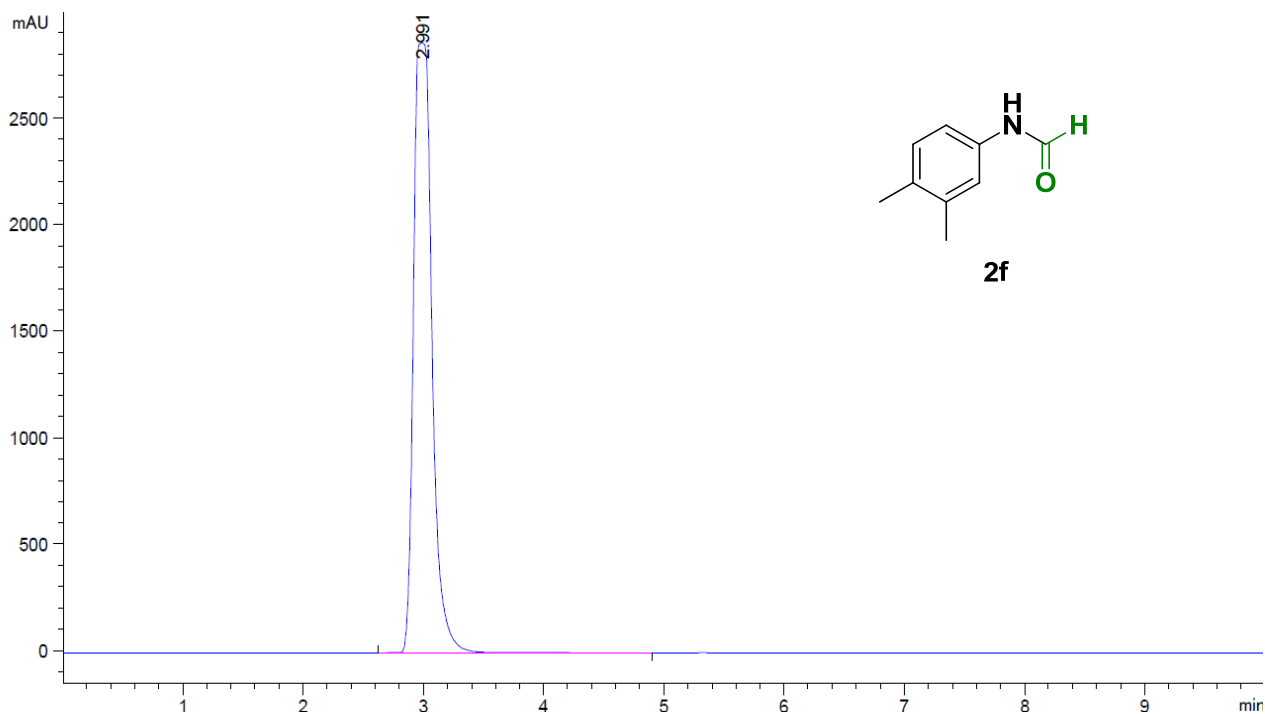
19.53
18.75



2f



¹³C NMR SPECTRUM OF COMPOUND 2f



HPLC OF COMPOUND 2f

PEAK INFORMATION OF COMPOUND 2f

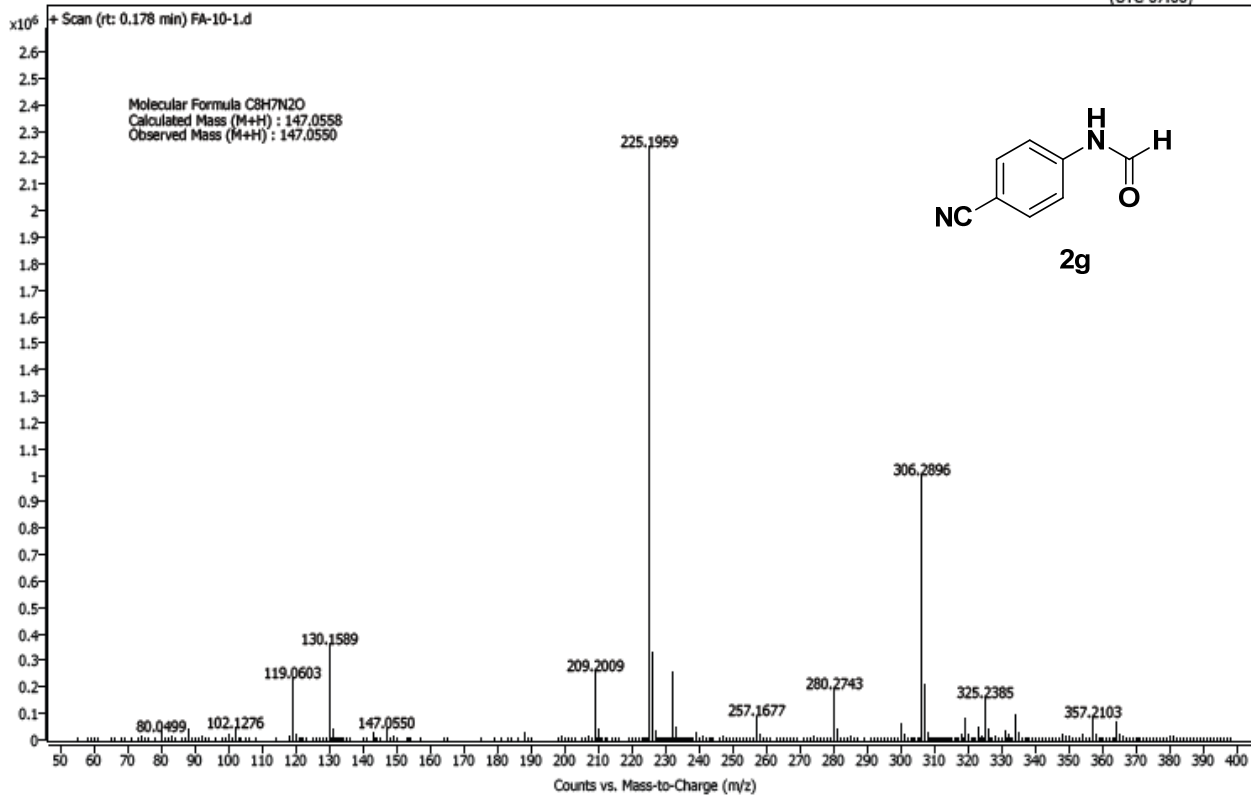
Peak #	Ret Time [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area %
1	2.991	0.1689	3.06458e4	2863.43213	100.0000

HPLC condition: RP-HPLC profile of 2f (method: water-acetonitrile (05-95%) in 10 min; detection wavelength at $\lambda = 254\text{nm}$; flow rate : 0.6 mL/min; column: Eclipse XDB-C18, pore size=5 μm , diameter x length = 4.6 x 150mm).

Spectrum Plot Report

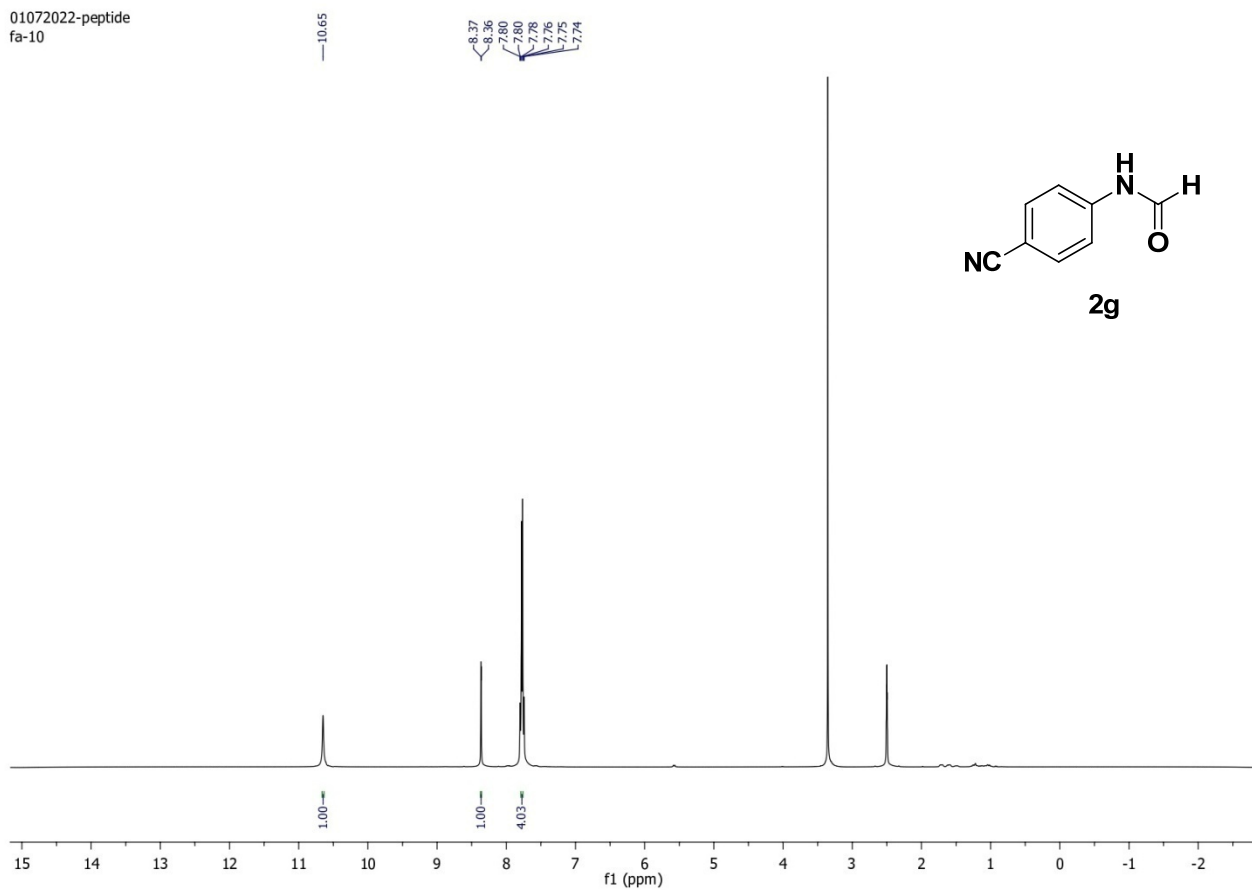


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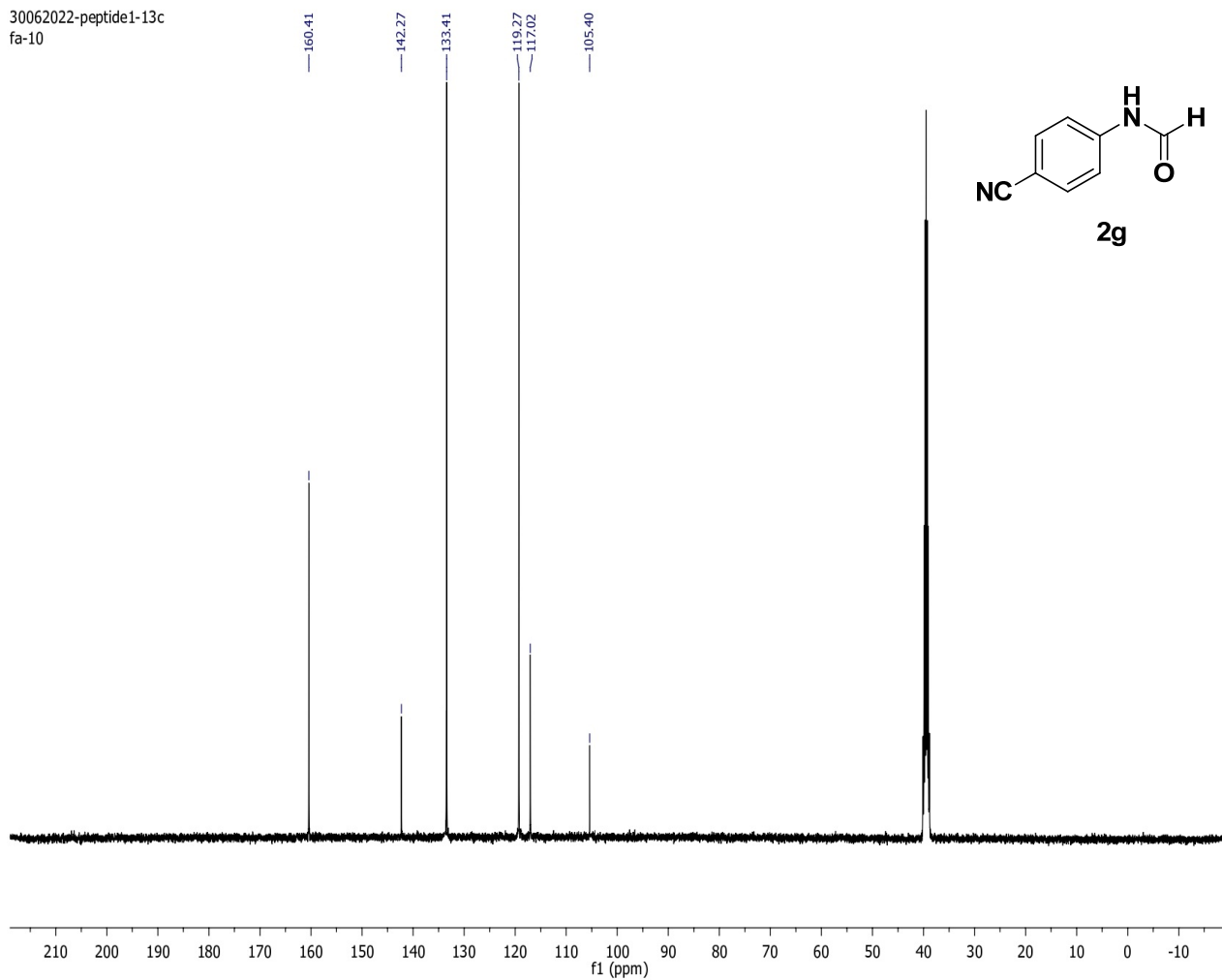


HRMS SPECTRUM OF COMPOUND 2g

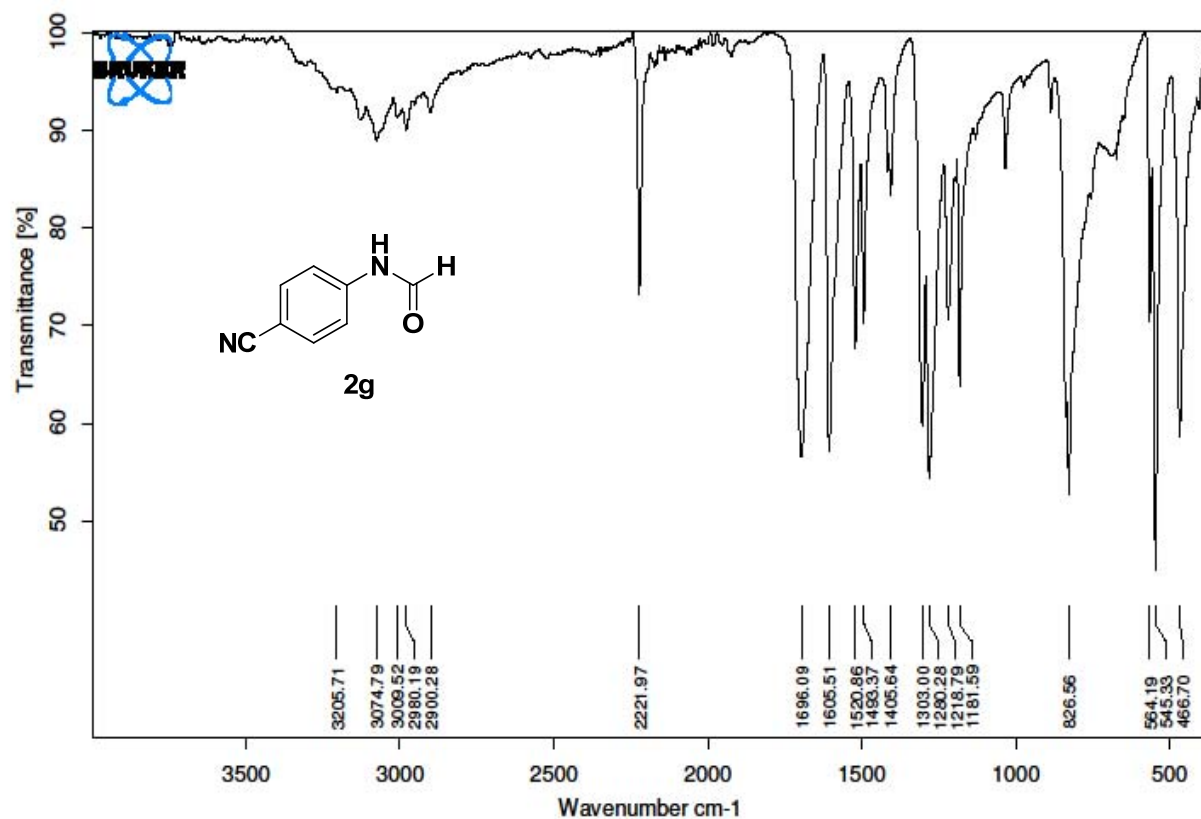
01072022-peptide
fa-10



30062022-peptide1-13c
fa-10



¹³C NMR SPECTRUM OF COMPOUND 2g



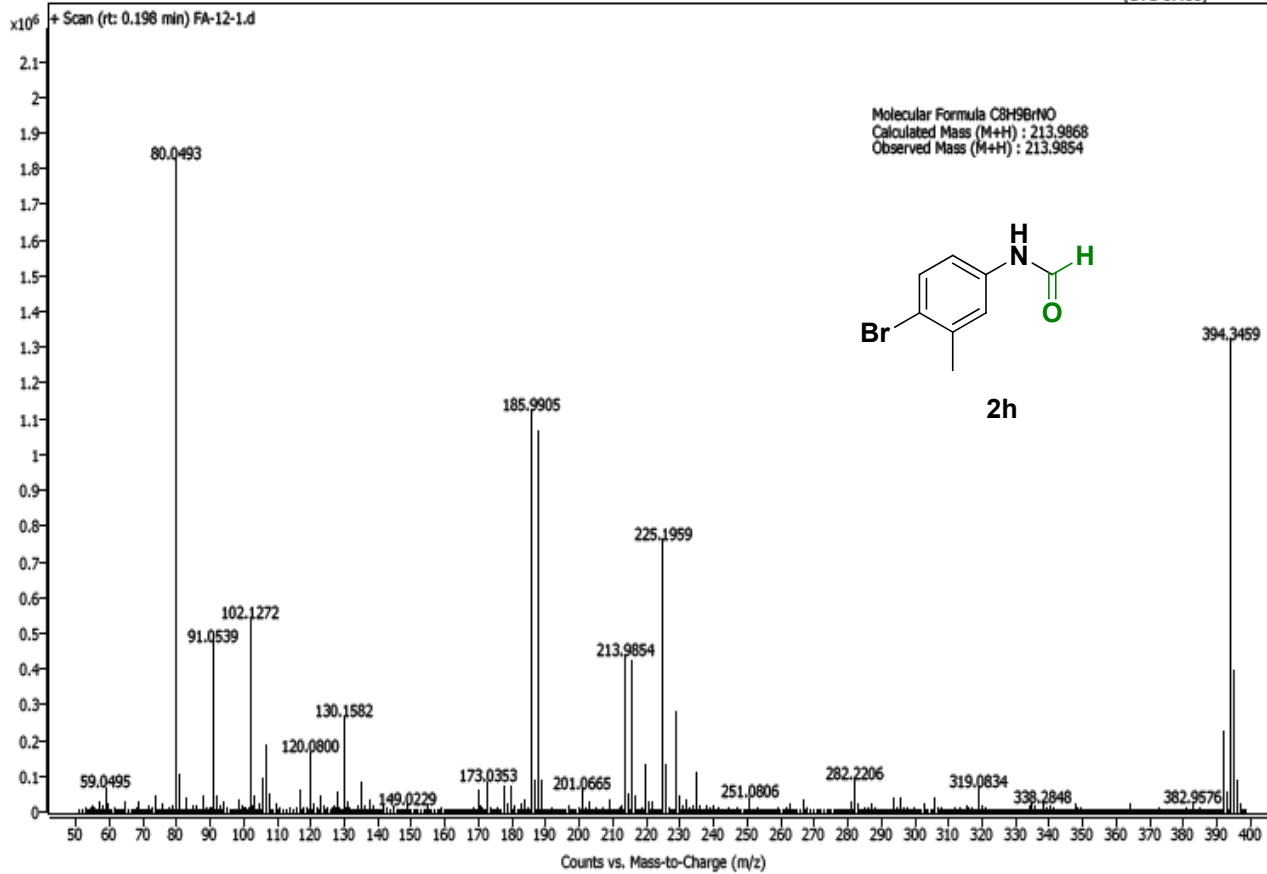
E:\WS\Chetani\FA-5.6	FA-10	Instrument type and / or accessory	27/08/2022
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IR- SPECTRA OF COMPOUND 2g

Spectrum Plot Report

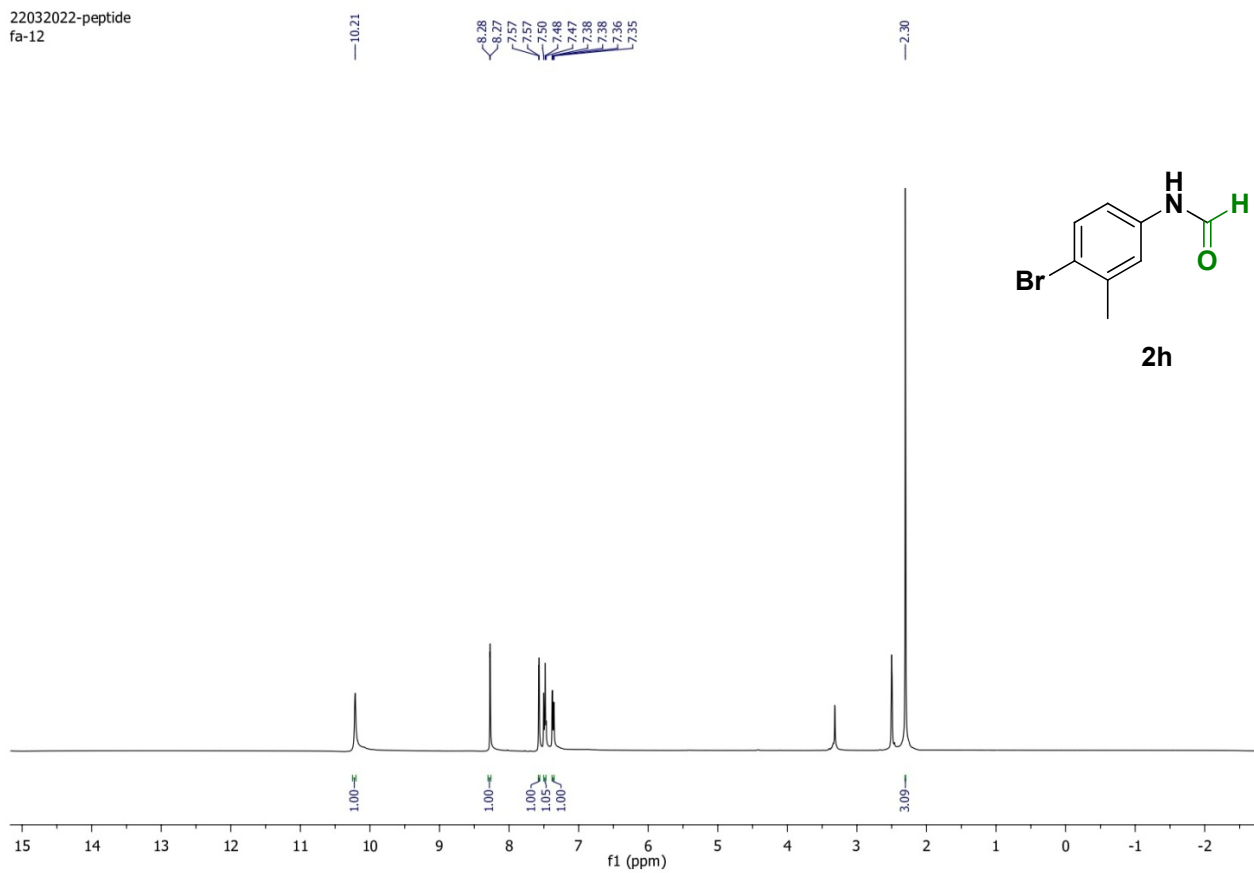


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Data File	FA-12-1.d	Method (Acq)	default.m	Comment		(UTC-07:00)	

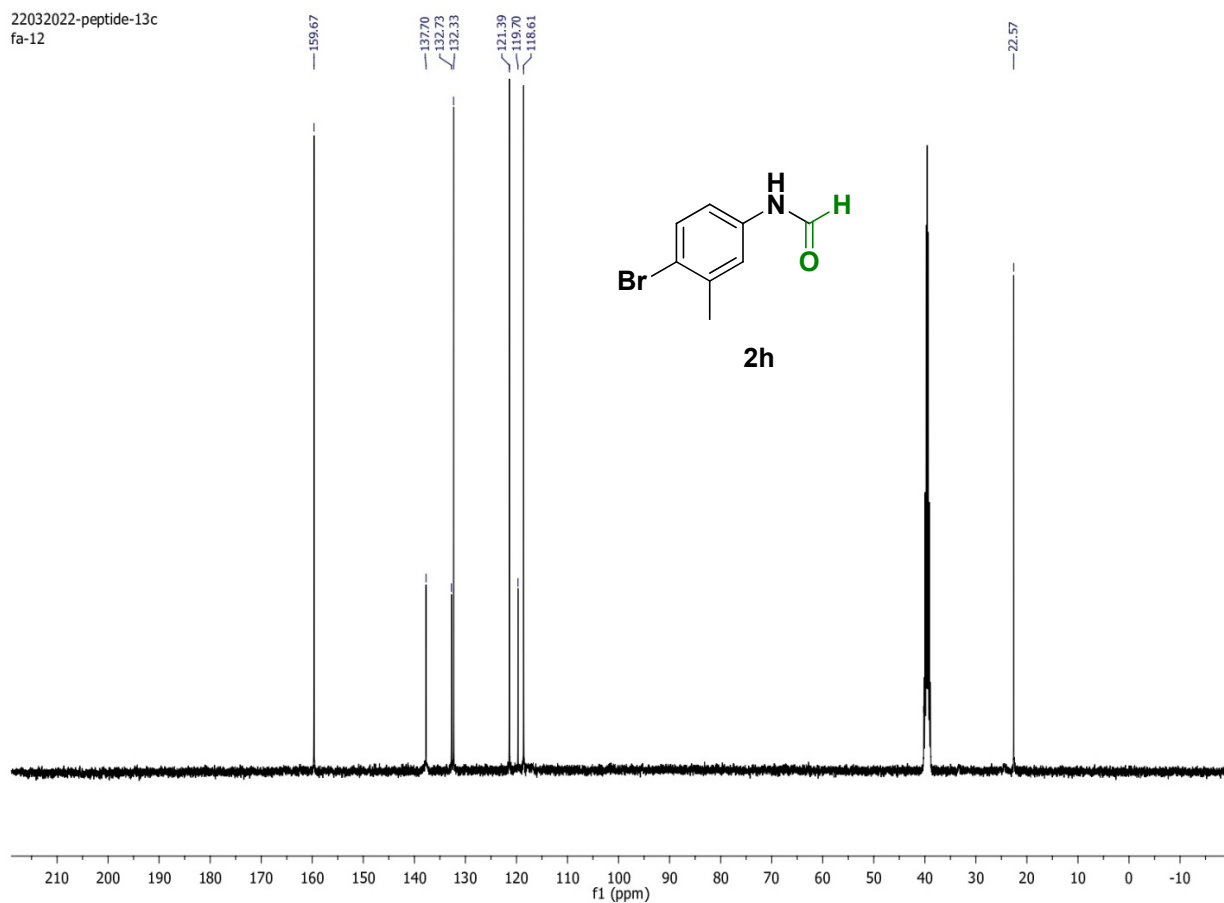


HRMS SPECTRUM OF COMPOUND 2h

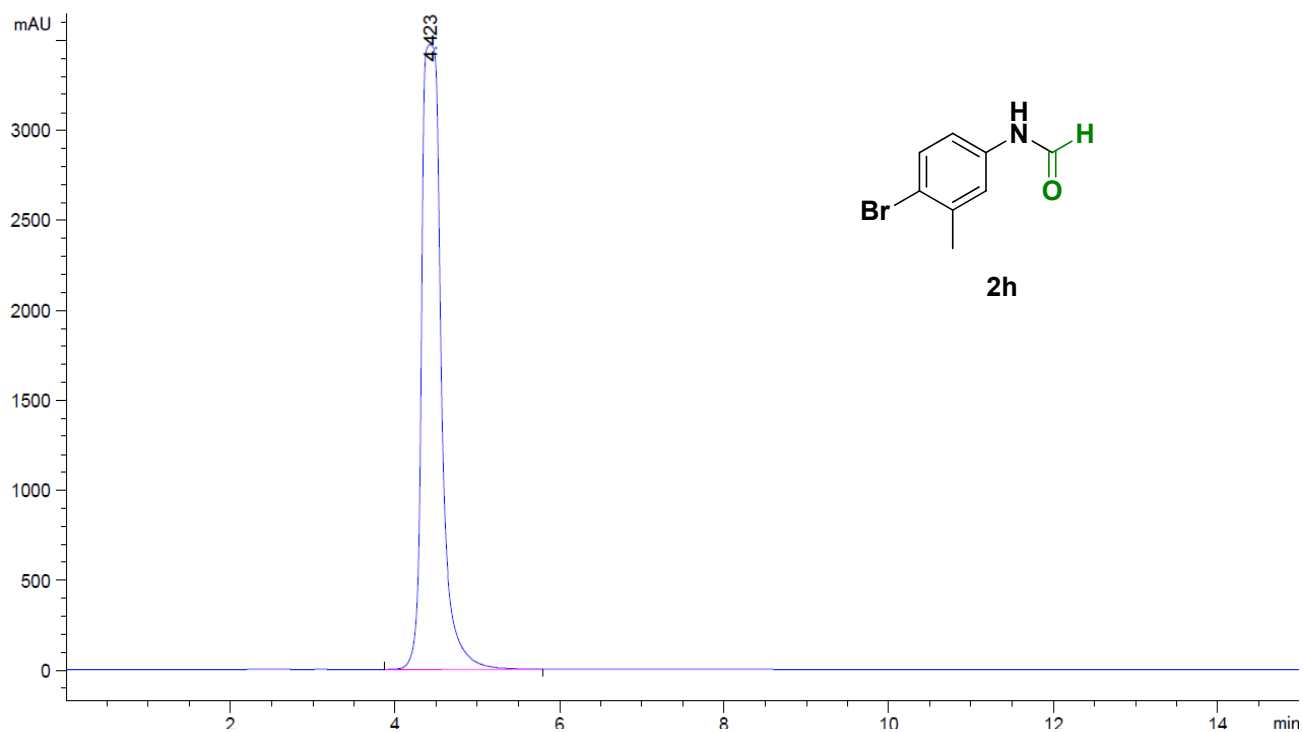
22032022-peptide
fa-12



22032022-peptide-13c
fa-12



¹³C NMR SPECTRUM OF COMPOUND 2h



HPLC OF COMPOUND 2h

PEAK INFORMATION OF COMPOUND 2h

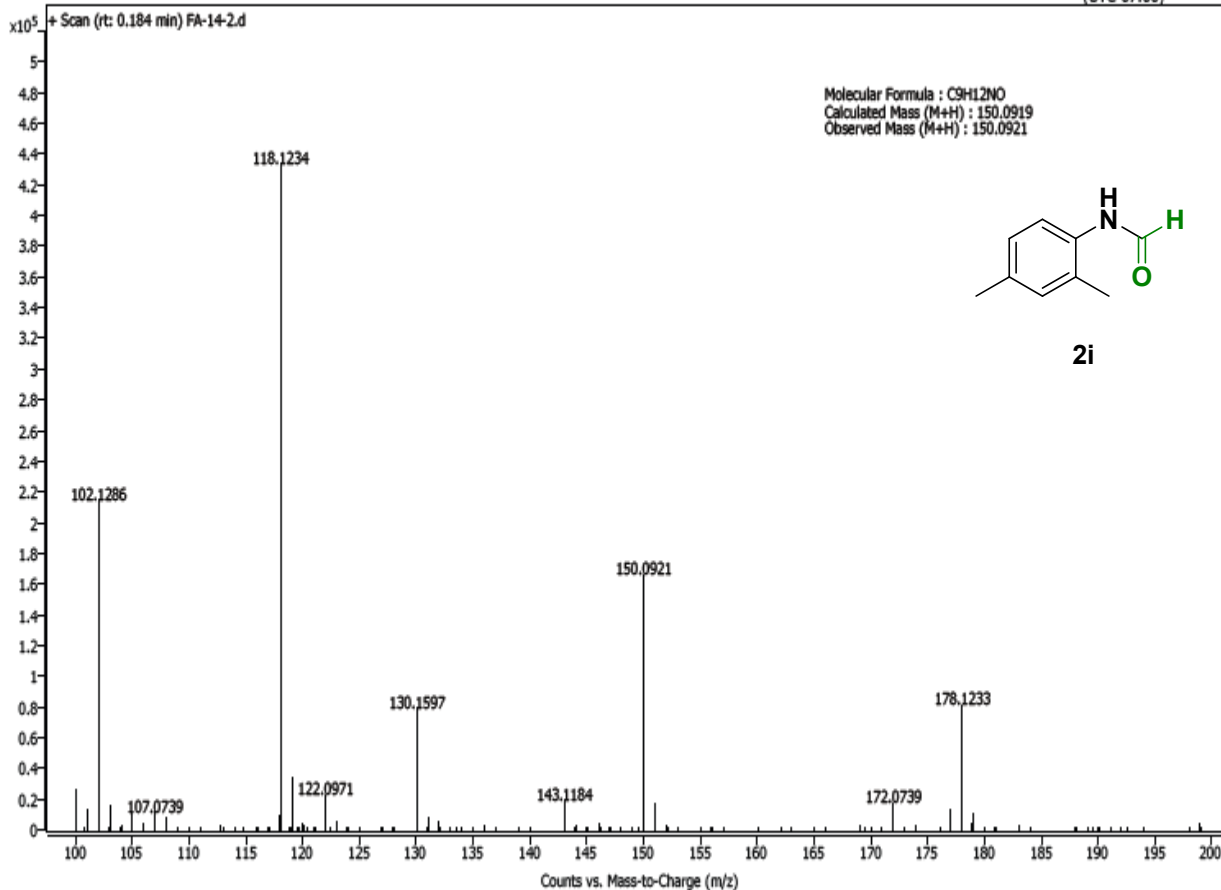
Peak #	Ret Time [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area %
1	4.423	0.2255	5.75809e4	3472.37183	100.0000

HPLC condition: RP-HPLC profile of 2h (method: water-acetonitrile (15-85%) in 15 min; detection wavelength at $\lambda = 254\text{nm}$; flow rate : 0.4 mL/min; column: Eclipse XDB-C18, pore size=5 μm , diameter x length = 4.6 x 150mm).

Spectrum Plot Report

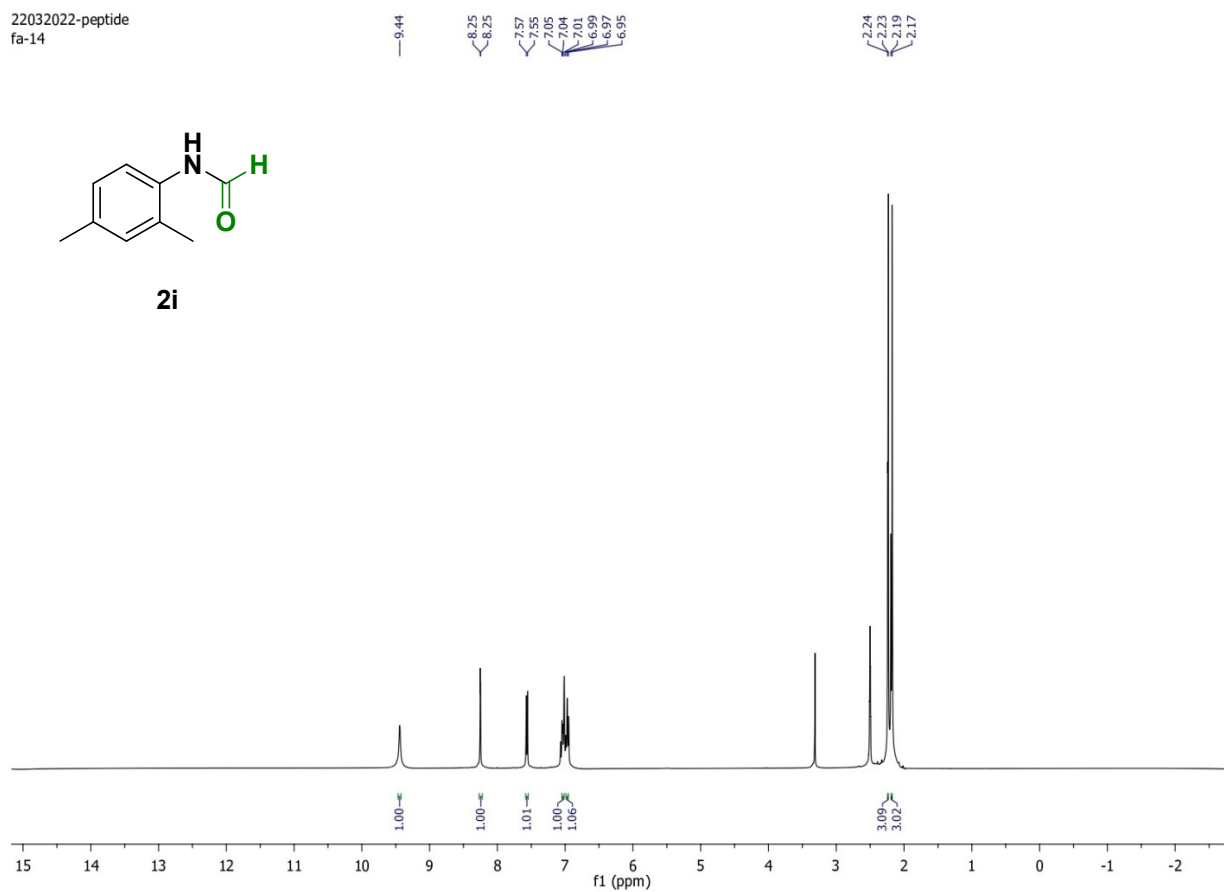


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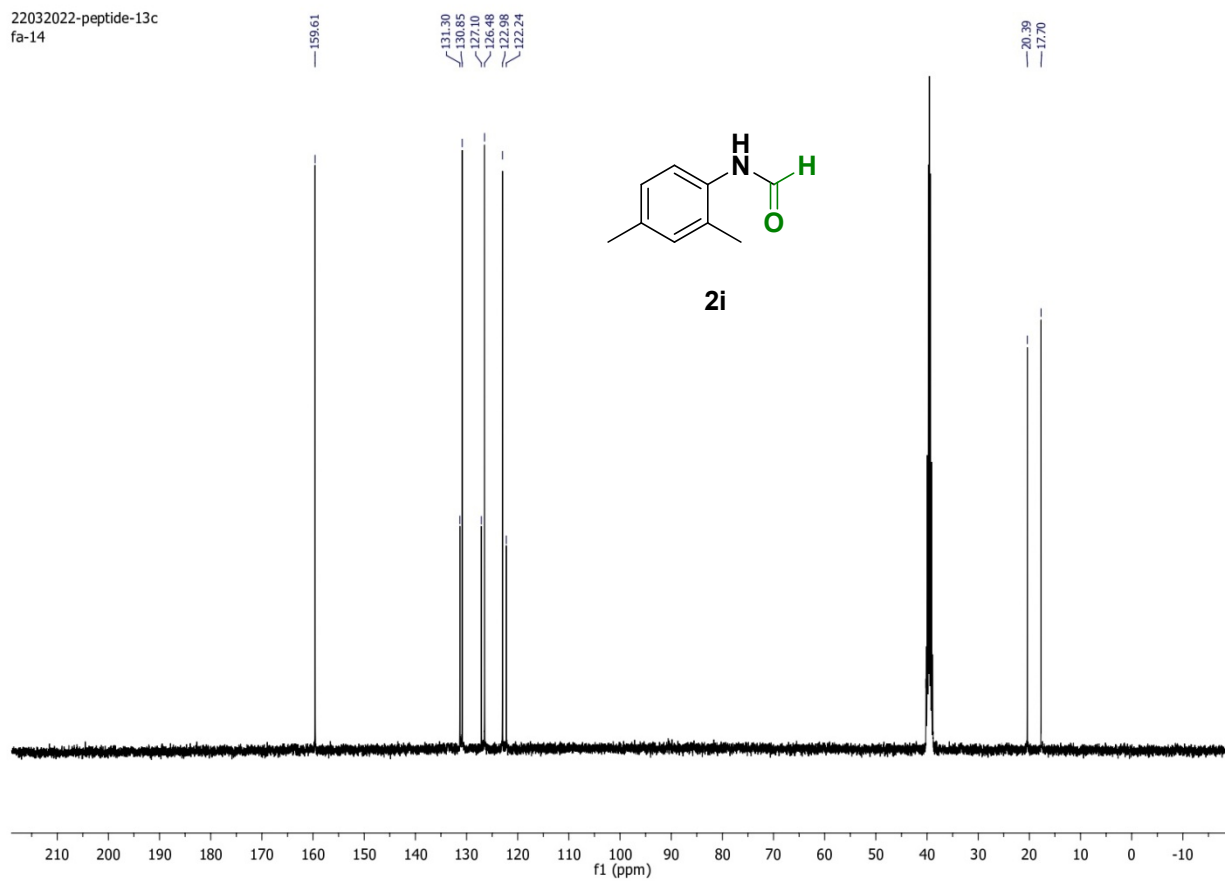
HRMS SPECTRUM OF COMPOUND 2i

22032022-peptide
fa-14

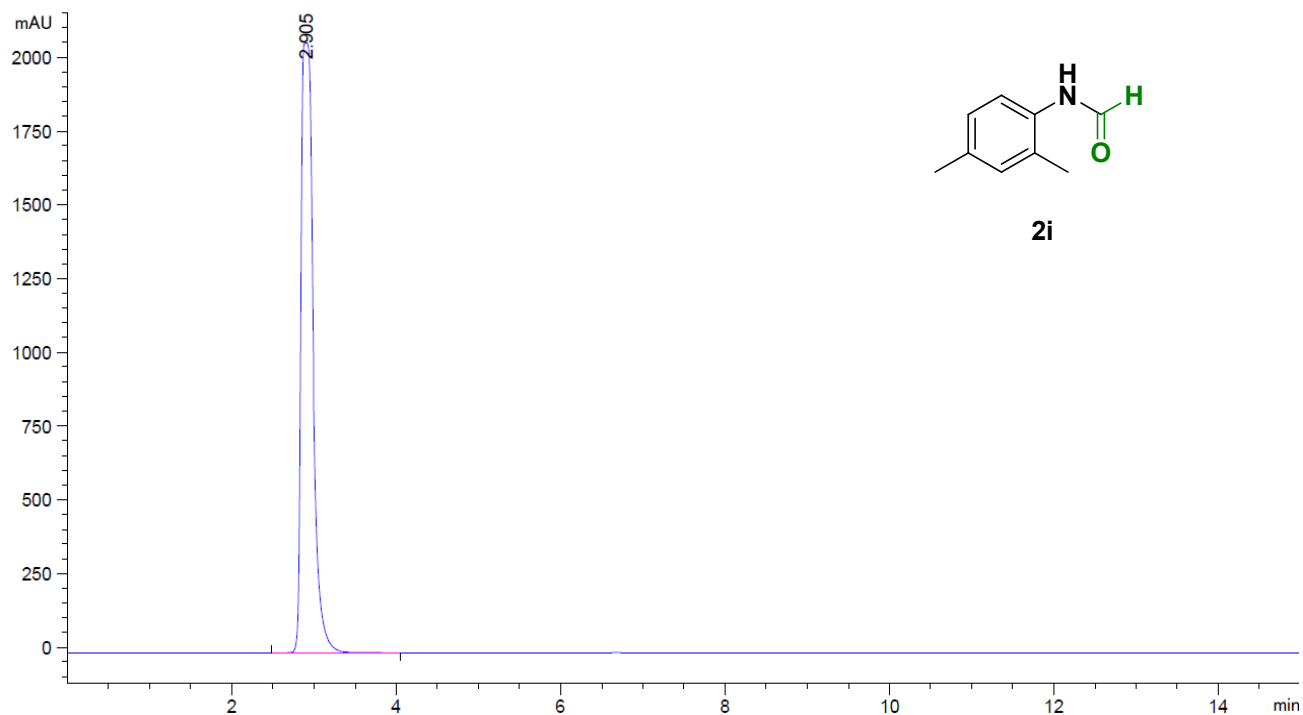


¹H NMR SPECTRUM OF COMPOUND 2i

22032022-peptide-13c
fa-14



¹³C NMR SPECTRUM OF COMPOUND 2i



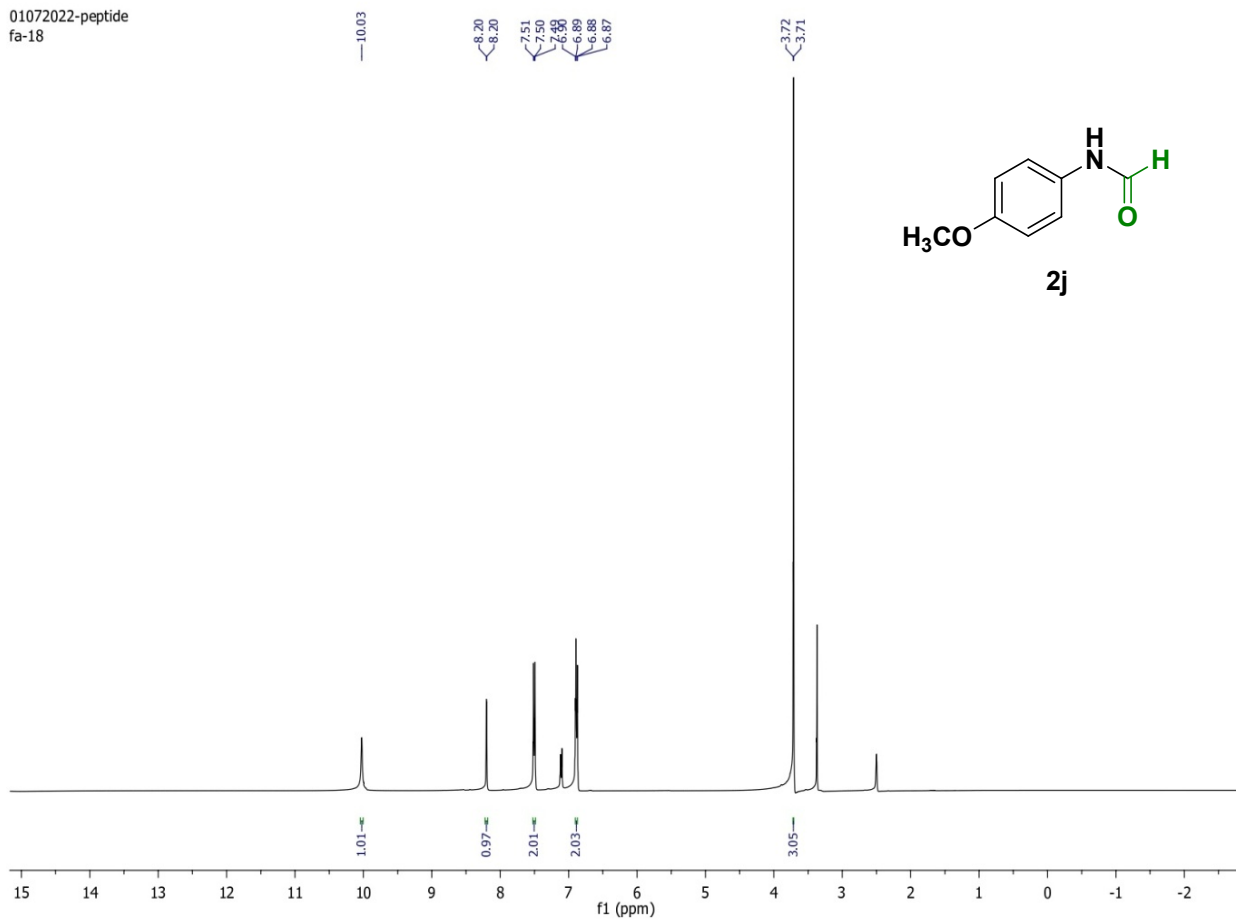
HPLC OF COMPOUND 2i

PEAK INFORMATION OF COMPOUND 2i

Peak #	Ret Time [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area %
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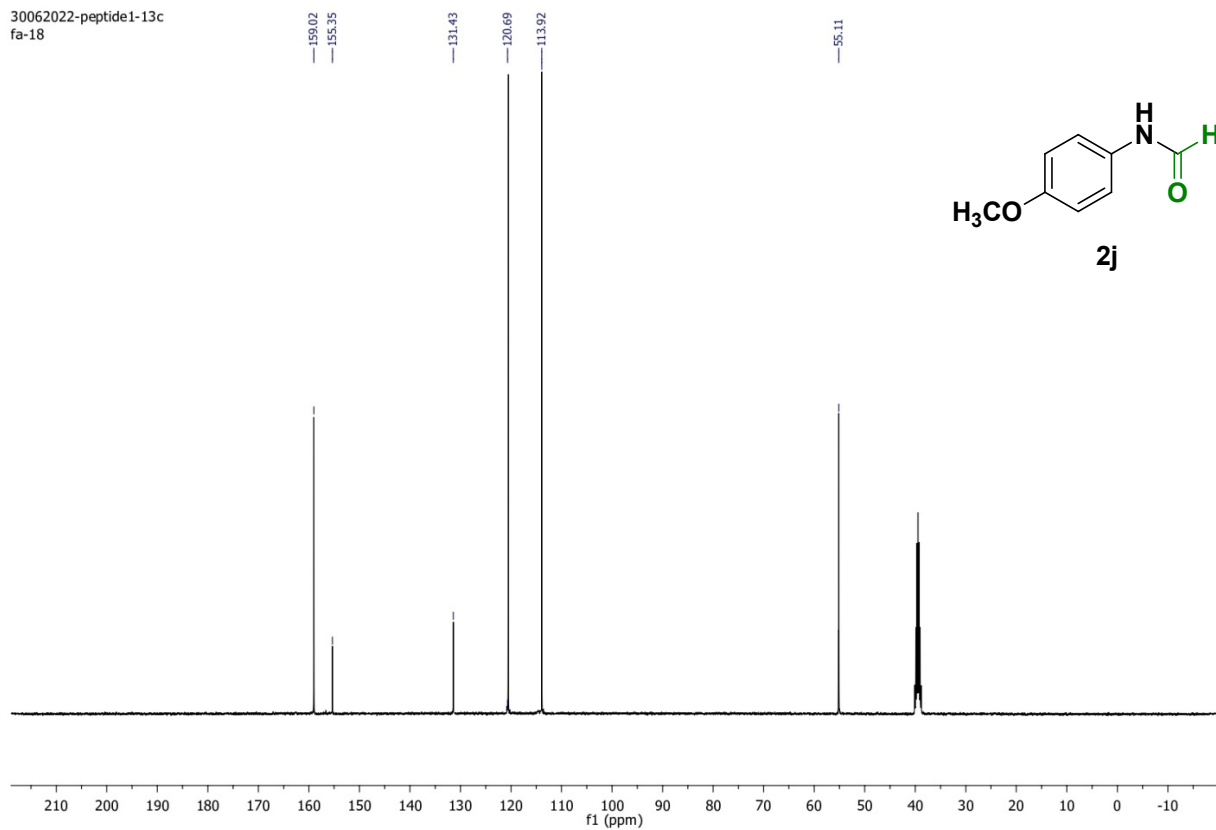
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01072022-peptide
fa-18

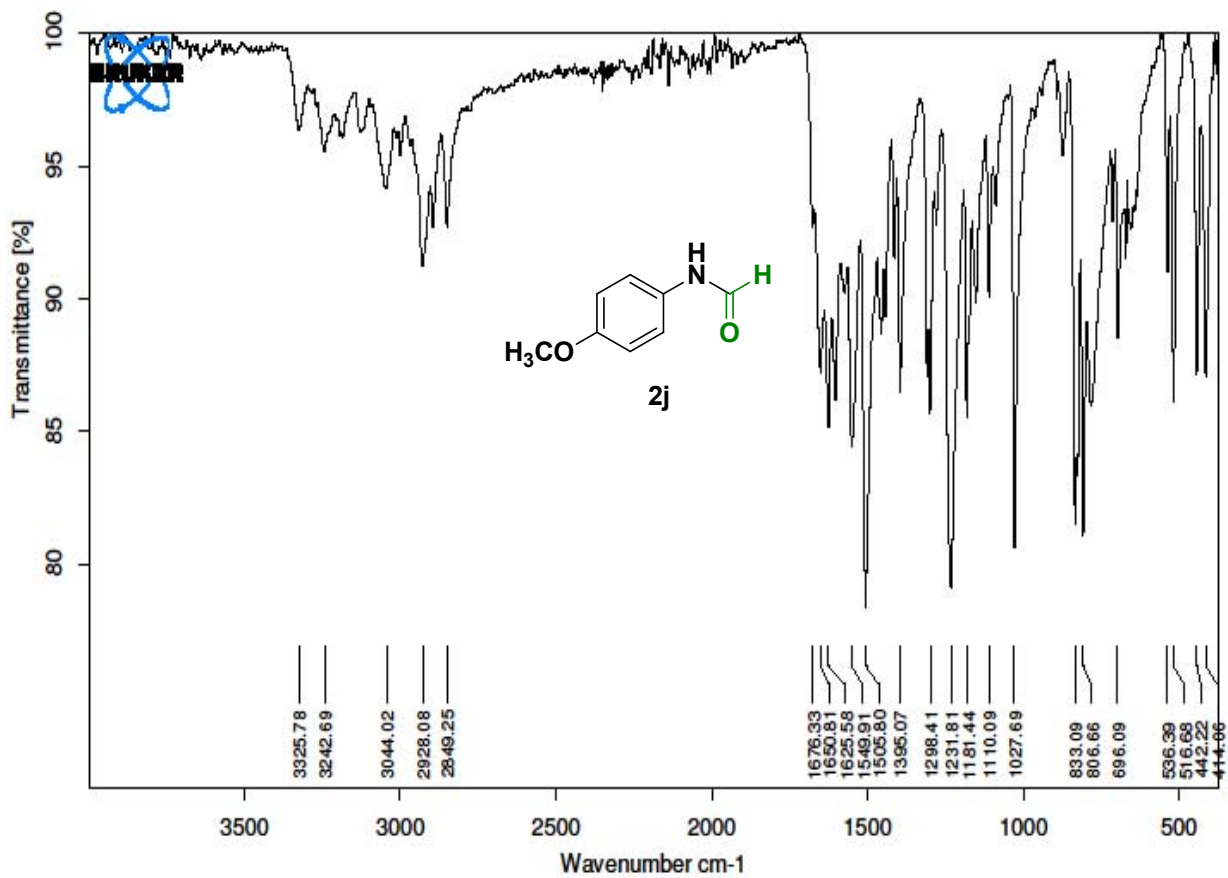


¹H NMR SPECTRUM OF COMPOUND 2j

30062022-peptide1-13c
fa-18

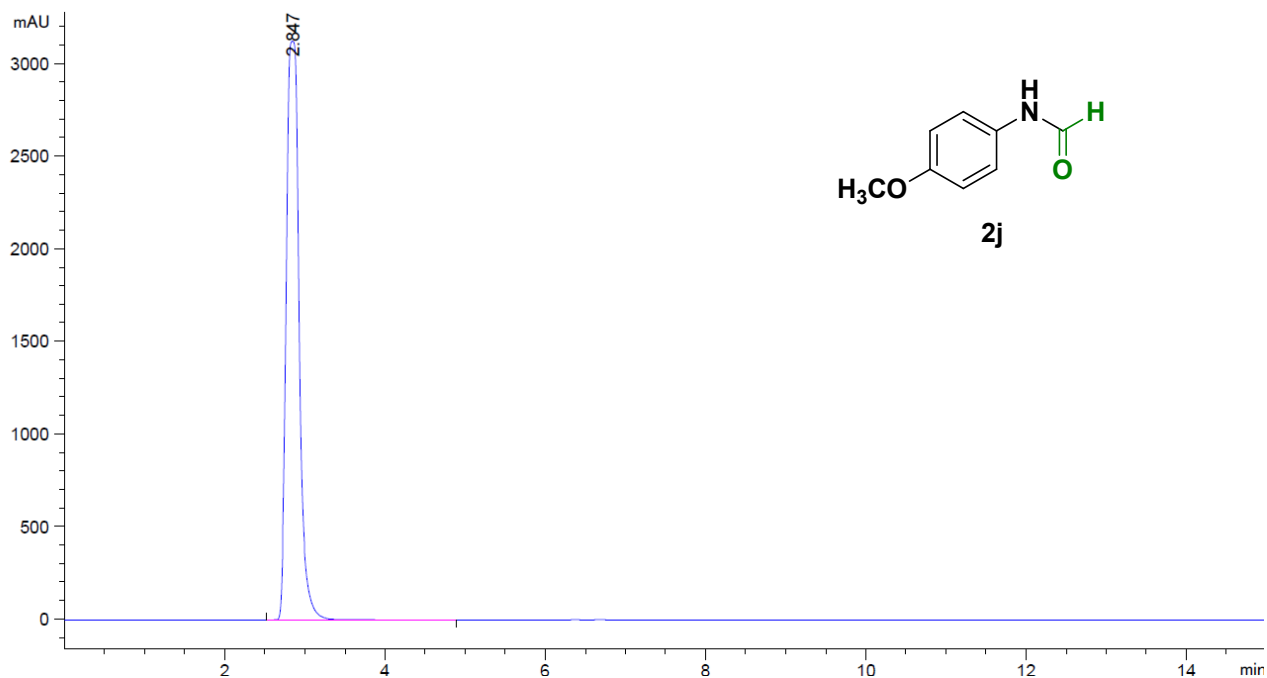


¹³C NMR SPECTRUM OF COMPOUND 2j



E:\WS\Chetan\FA-5.3	FA-18	Instrument type and / or accessory	27/08/2022
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IR- SPECTRA OF COMPOUND 2j



HPLC OF COMPOUND 2j

PEAK INFORMATION OF COMPOUND 2j

Peak #	Ret Time [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area %
1	2.847	0.1699	3.42507e4	3125.20752	100.0000

HPLC condition: RP-HPLC profile of 2j (method: water-acetonitrile (10-90%) in 15 min; detection wavelength at $\lambda = 254\text{nm}$; flow rate : 0.6 mL/min; column: Eclipse XDB-C18, pore size=5 μm , diameter x length = 4.6 x 150mm).