Supplementary Information

Synthesis of N-aryl derived formamides using triflic anhydride

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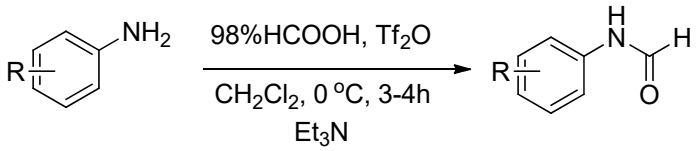
Contents

General Information

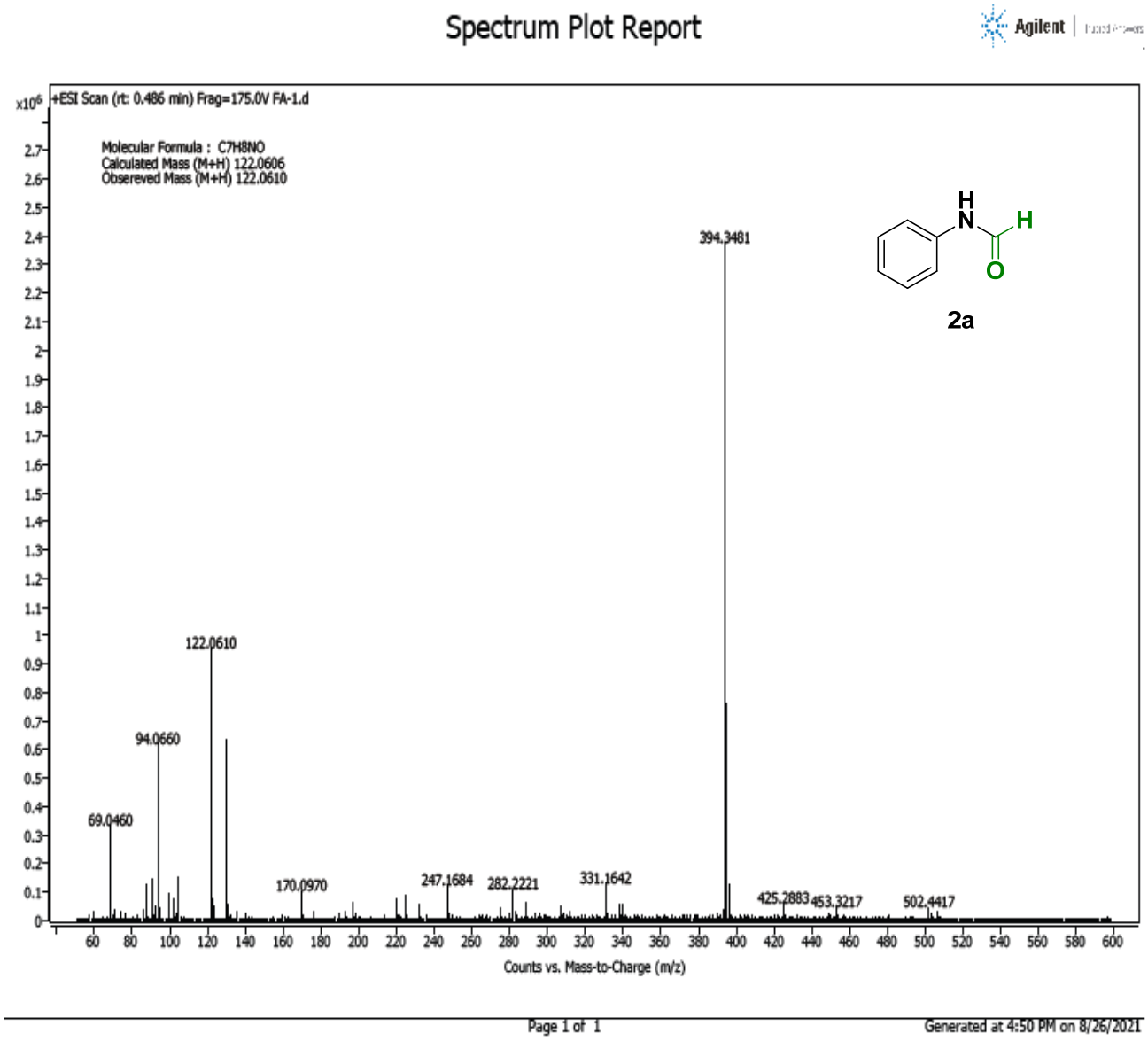
All commercially available reagents were used as received without further purification. The solvents were purified and dried by standard procedures prior to use. Reaction progress was monitored by MERCK thin layer chromatography (TLC) performed on aluminium plates coated with silica gel 60 F254. High 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

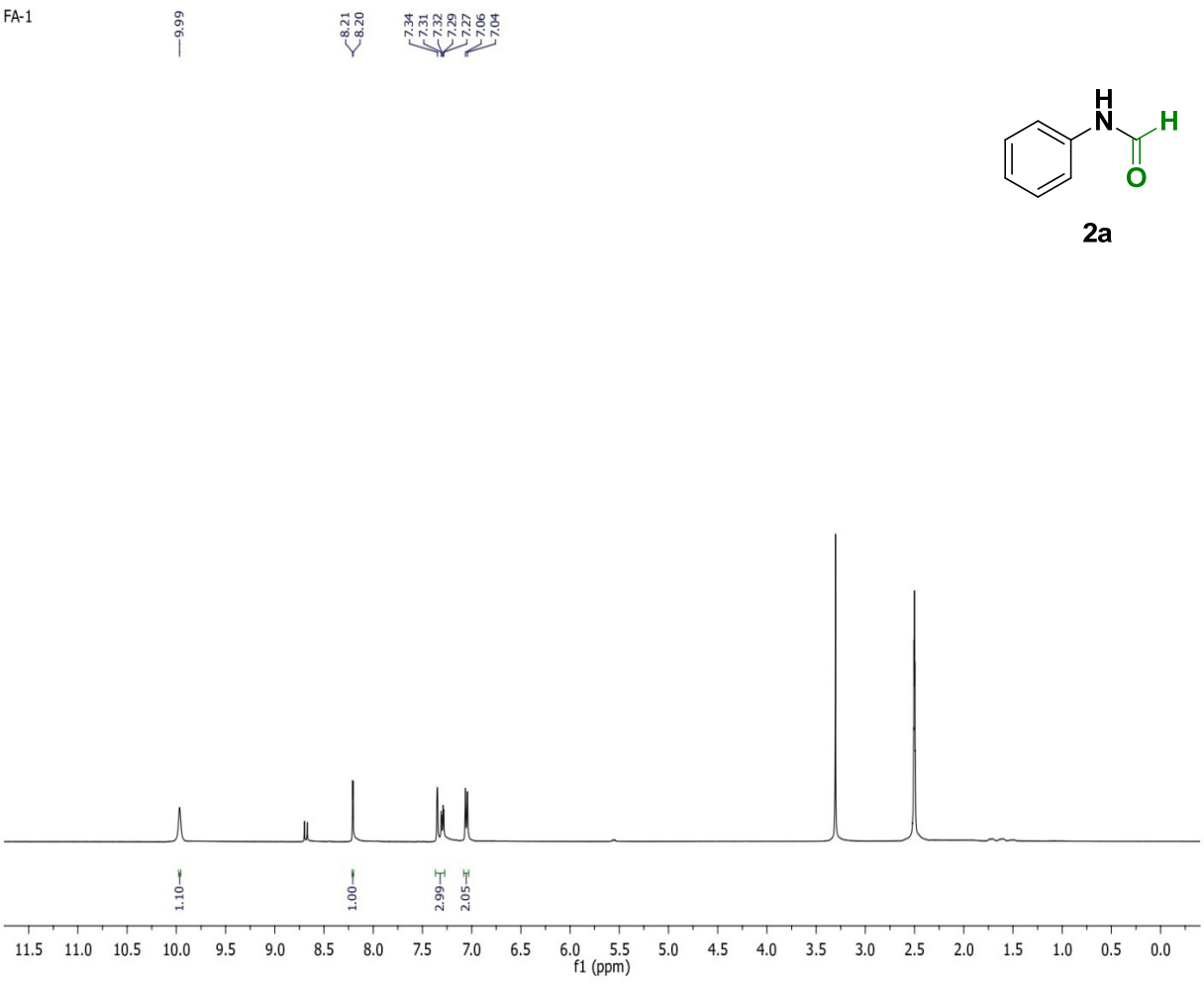


Scheme: Synthesis of formamides mediated by Tf_2O

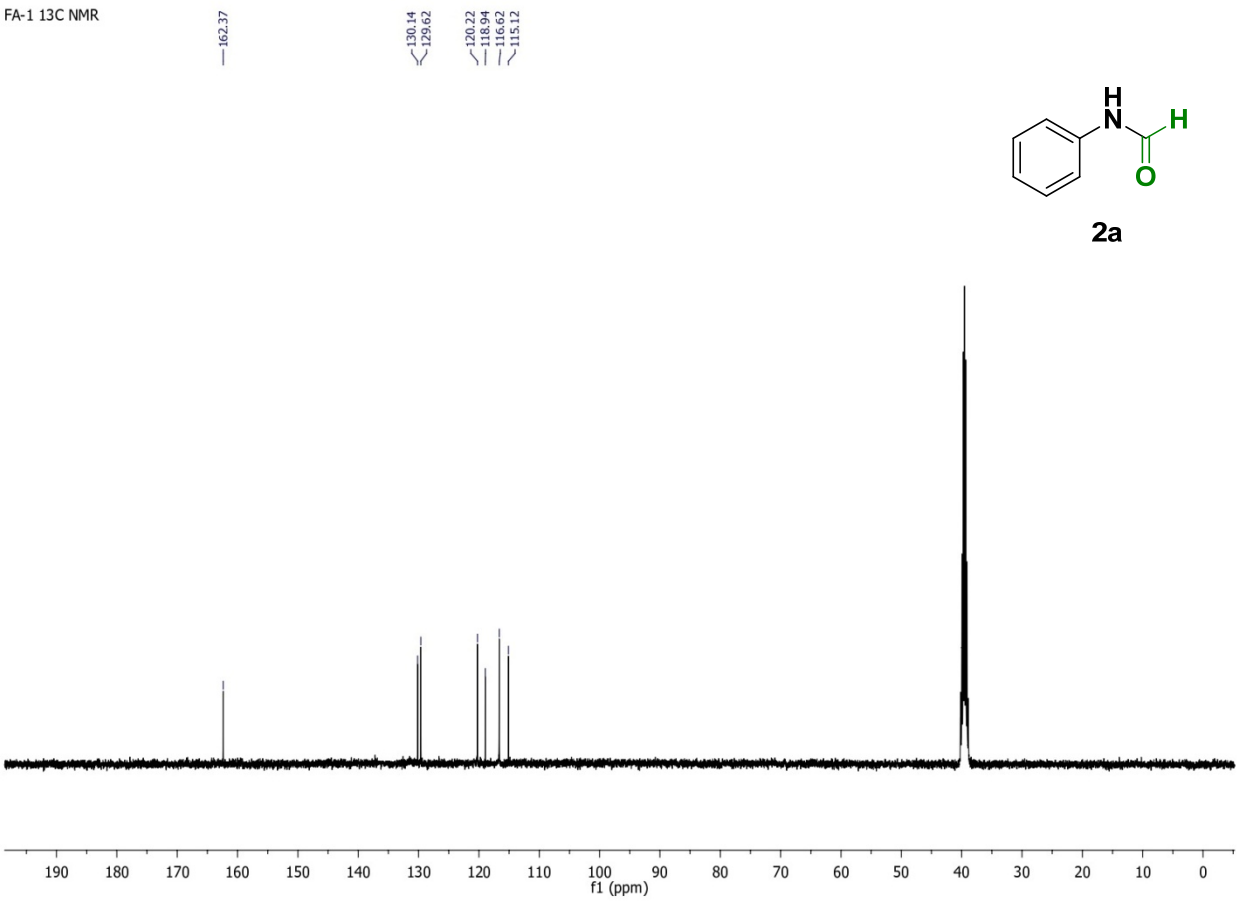
To a stirred solution of 98% Formic acid in DCM was initially cooled to 0 ˚C. Tf_2O (2.0 equiv) and Et_3N (2.0 equiv) were added. And the reaction mixture was stired for 15-20 min. Later, aniline (1.0 equiv) was added and the reaction mixture was stirred for another 4 hr. The course of the reaction was monitored by TLC. The solvent was vaccum-evaporated. The residue was diluted with ethyl acetate (EtOAc). The organic layer was washed with 10% HCl, followed by 10% Na_2CO_3 and then brine. The organic layer was dried over anhydrous sodium sulfate (Na_2SO_4), 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.



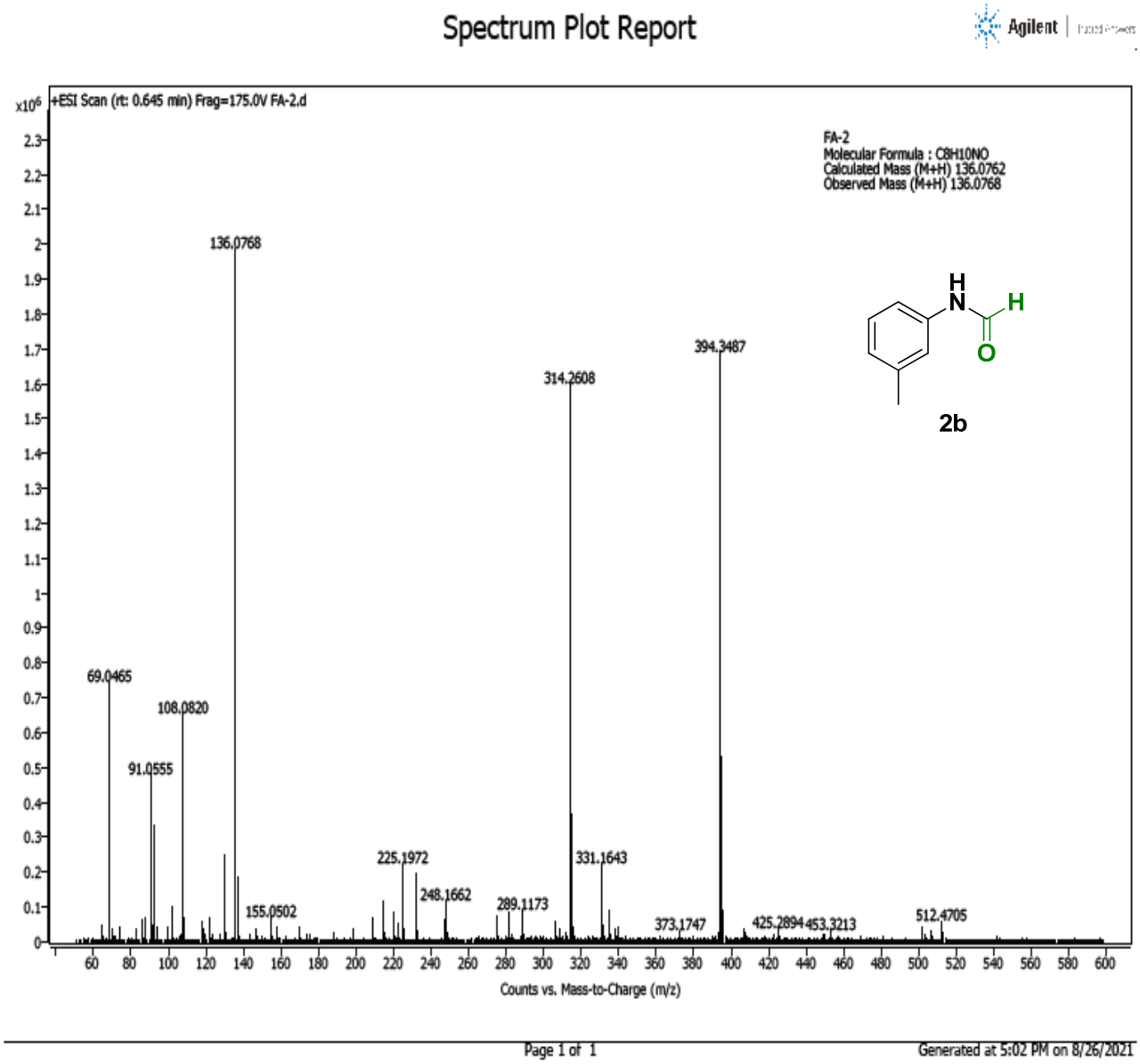
HRMS SPECTRUM OF COMPOUND 2a



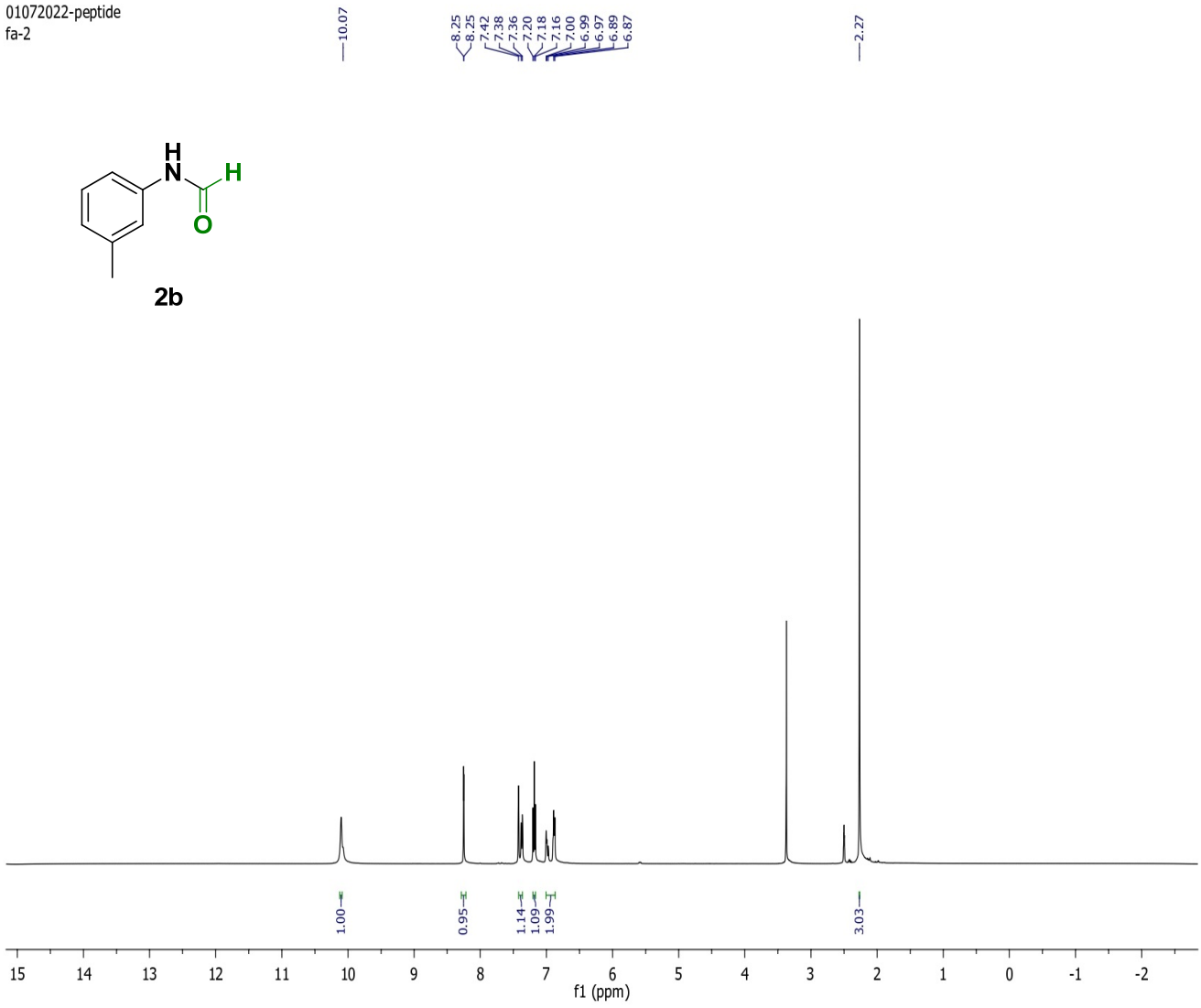
^{1}H NMR SPECTRUM OF COMPOUND 2a



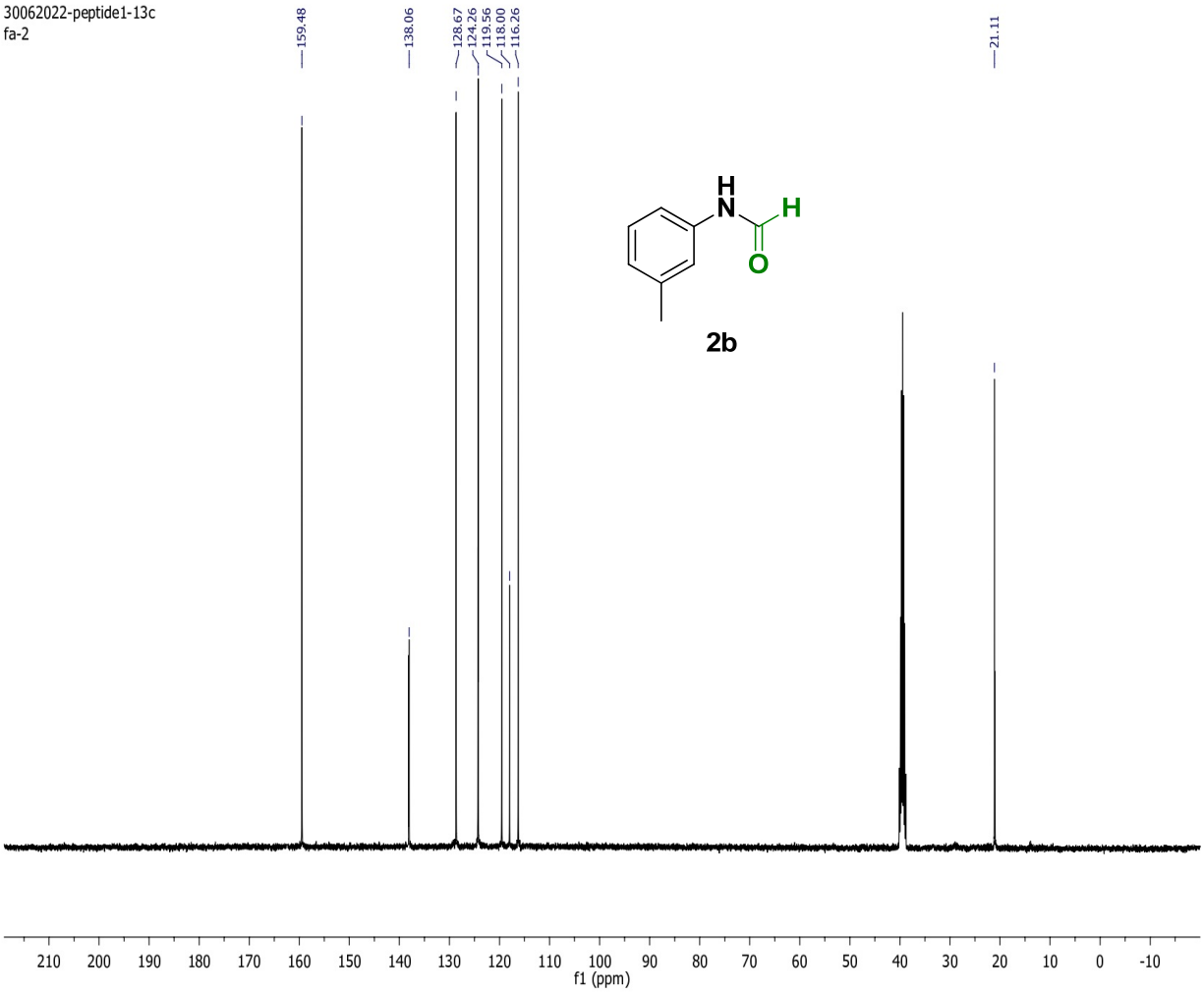
^{13}C NMR SPECTRUM OF COMPOUND 2a



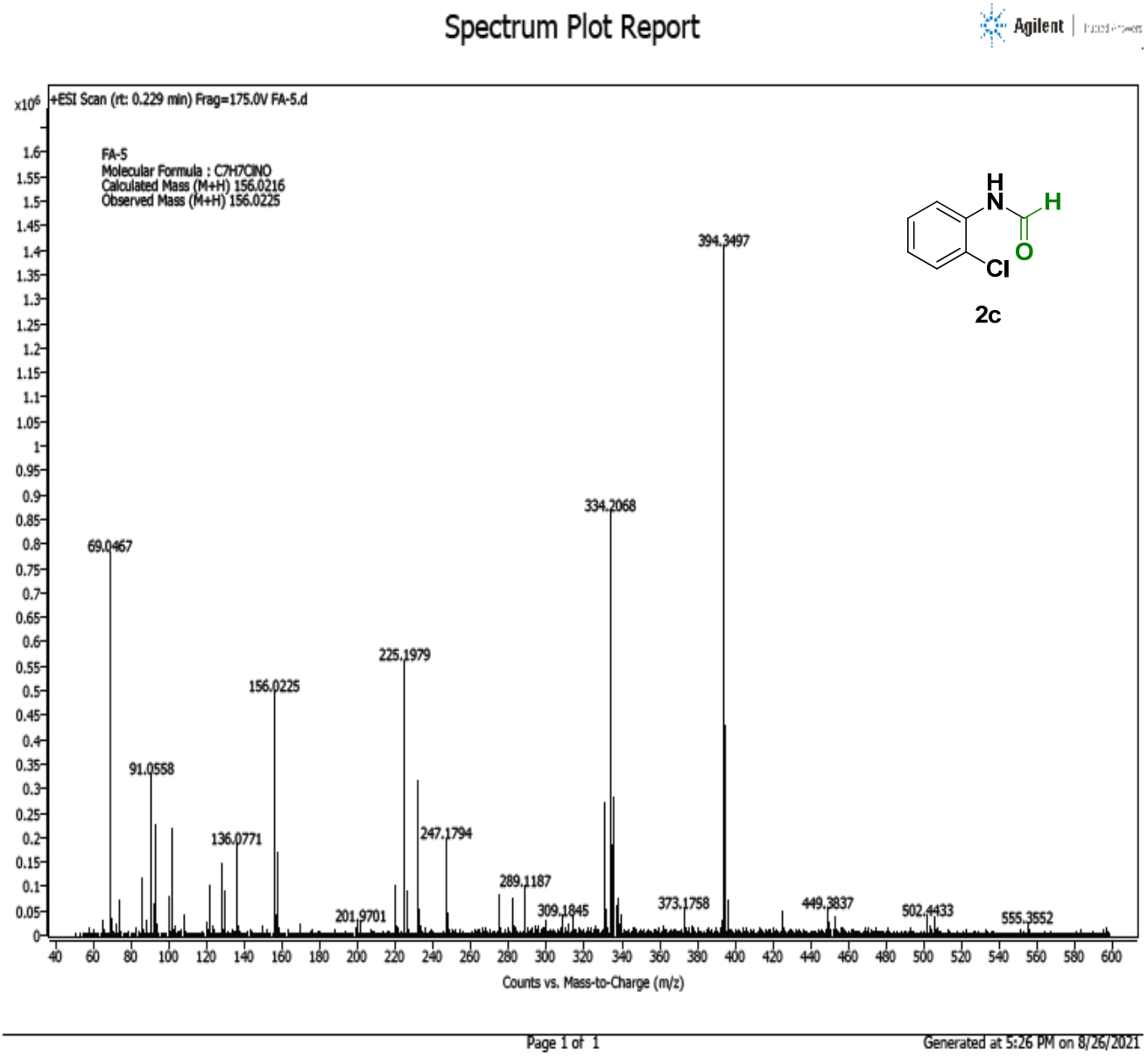
HRMS SPECTRUM OF COMPOUND 2b



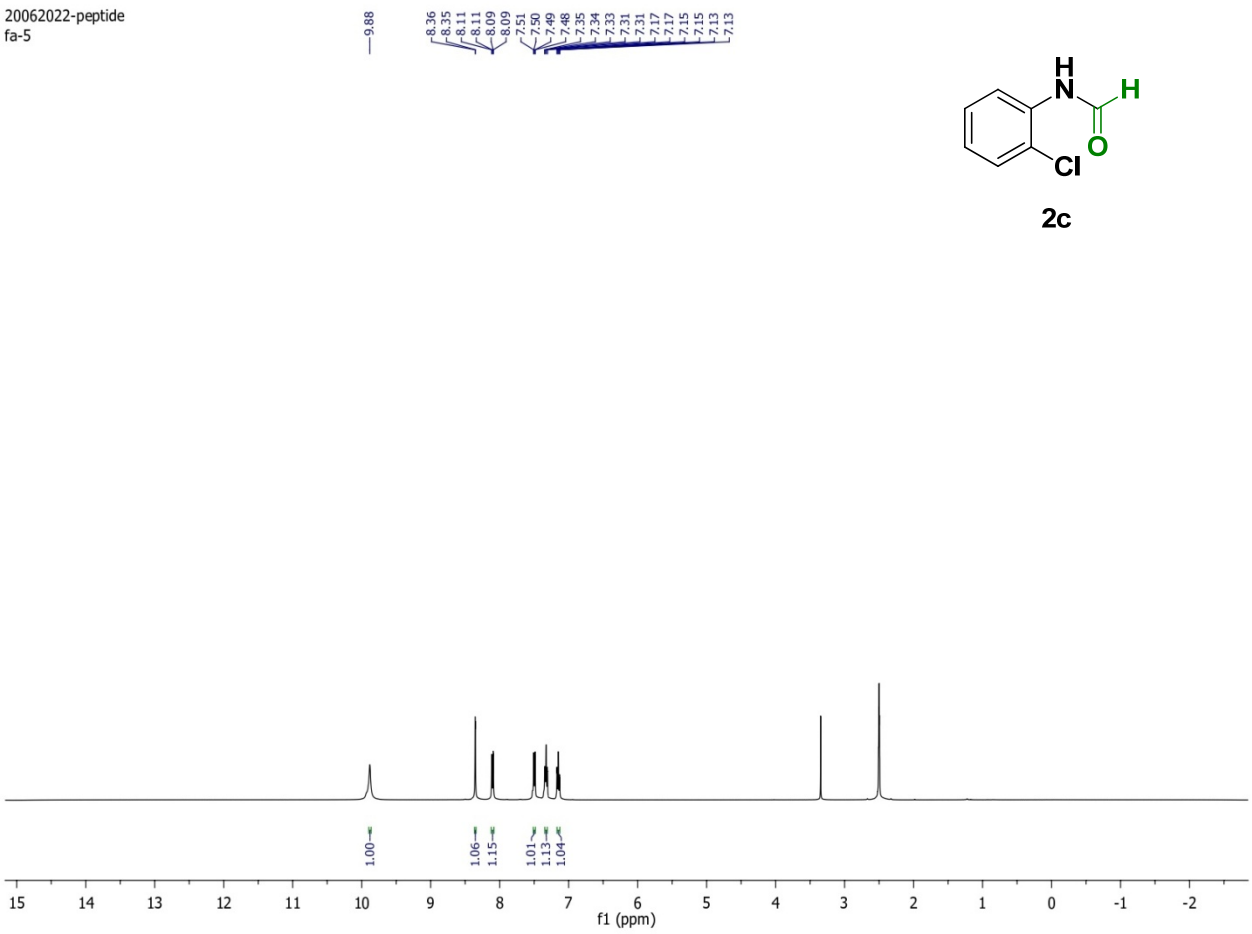
^{1}H NMR SPECTRUM OF COMPOUND 2b



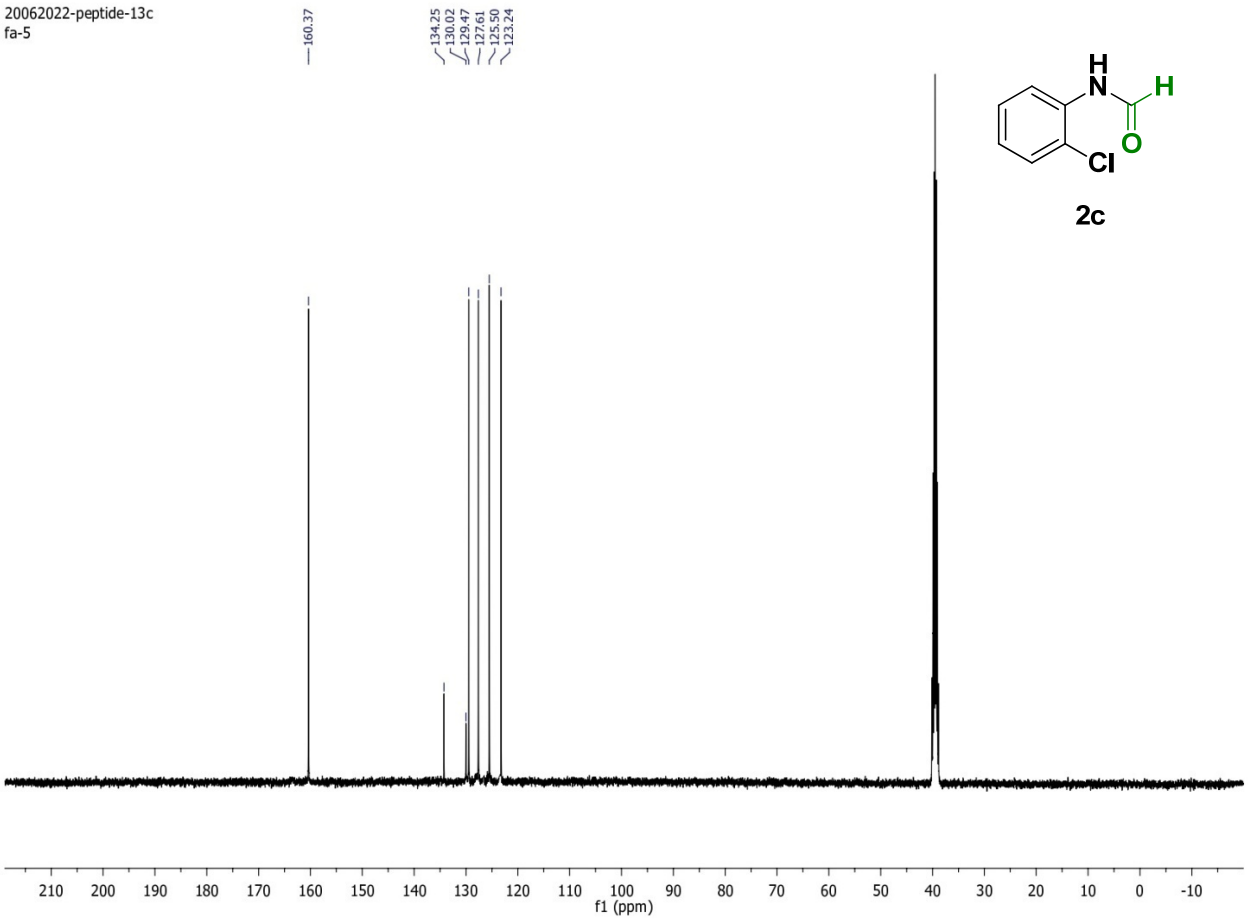
^{13}C NMR SPECTRUM OF COMPOUND 2b



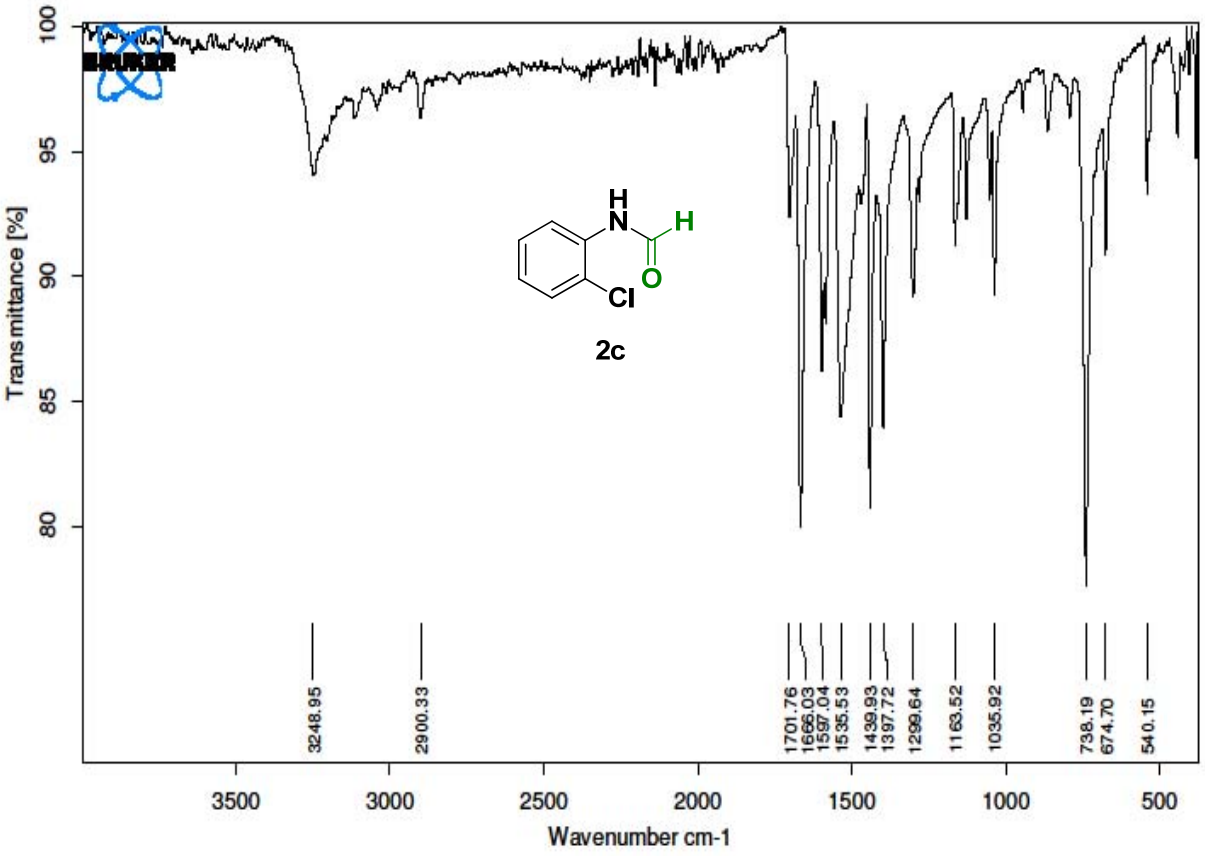
HRMS SPECTRUM OF COMPOUND 2c



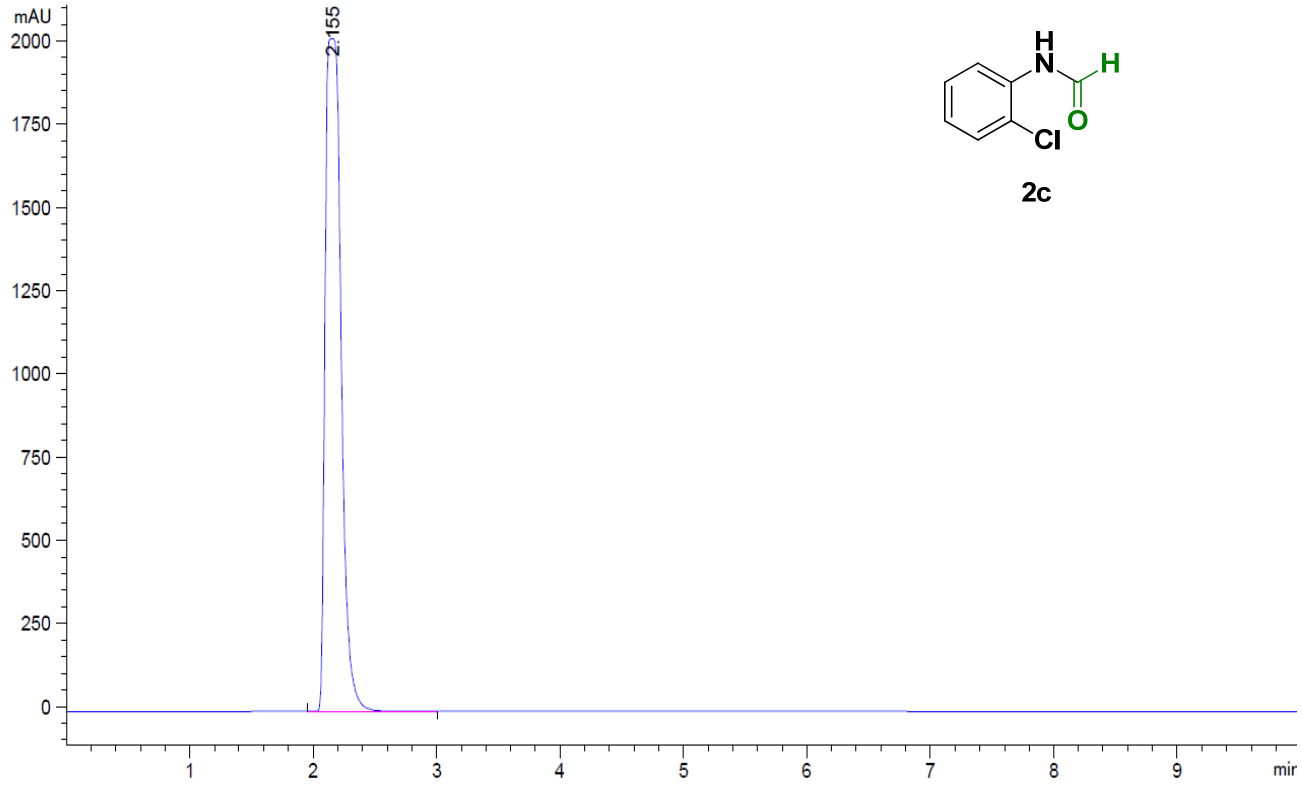
^{1}H NMR SPECTRUM OF COMPOUND 2c



^{13}C NMR SPECTRUM OF COMPOUND 2c



IR-SPECTRA OF COMPOUND 2c

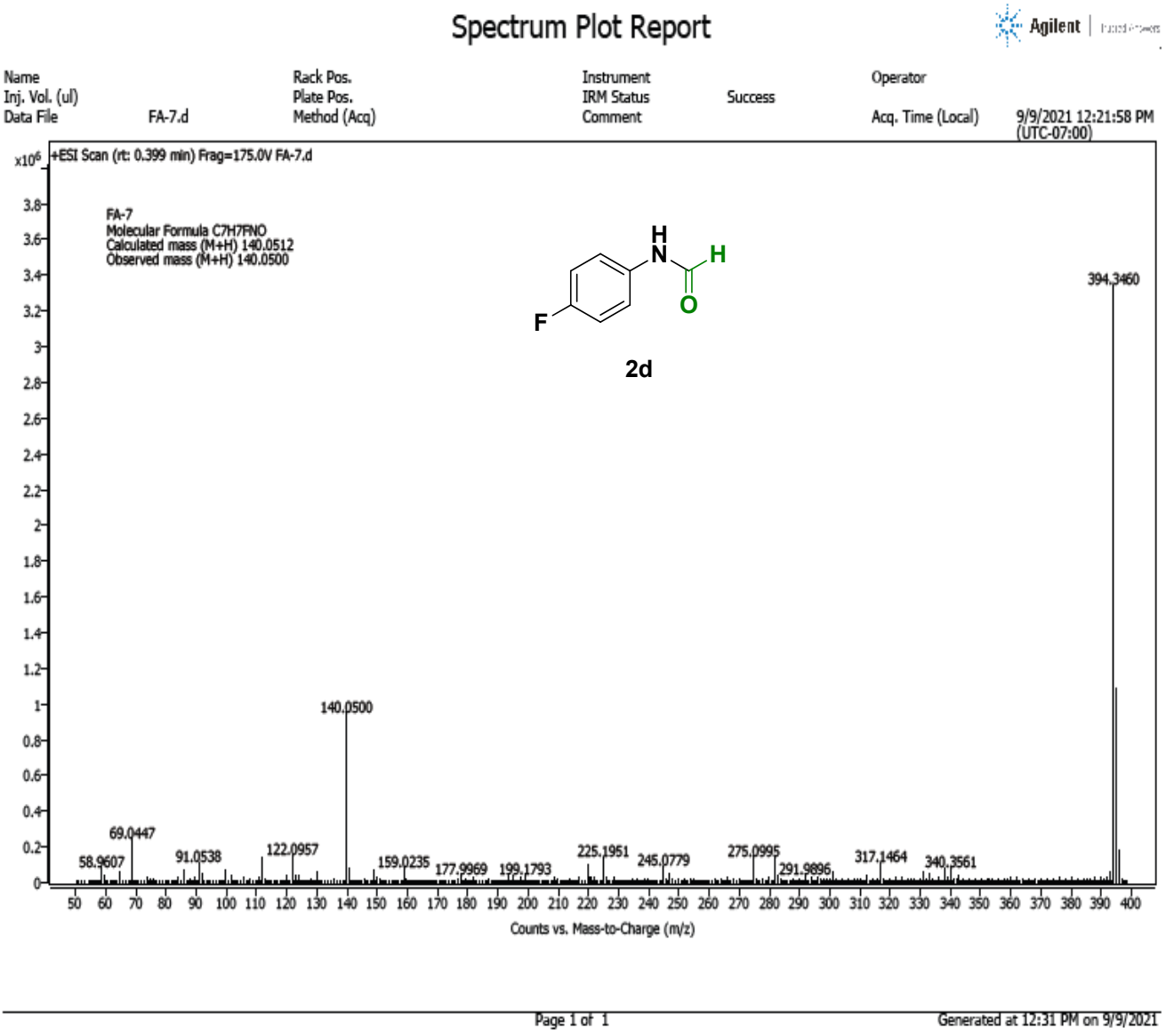


HPLC OF COMPOUND 2c

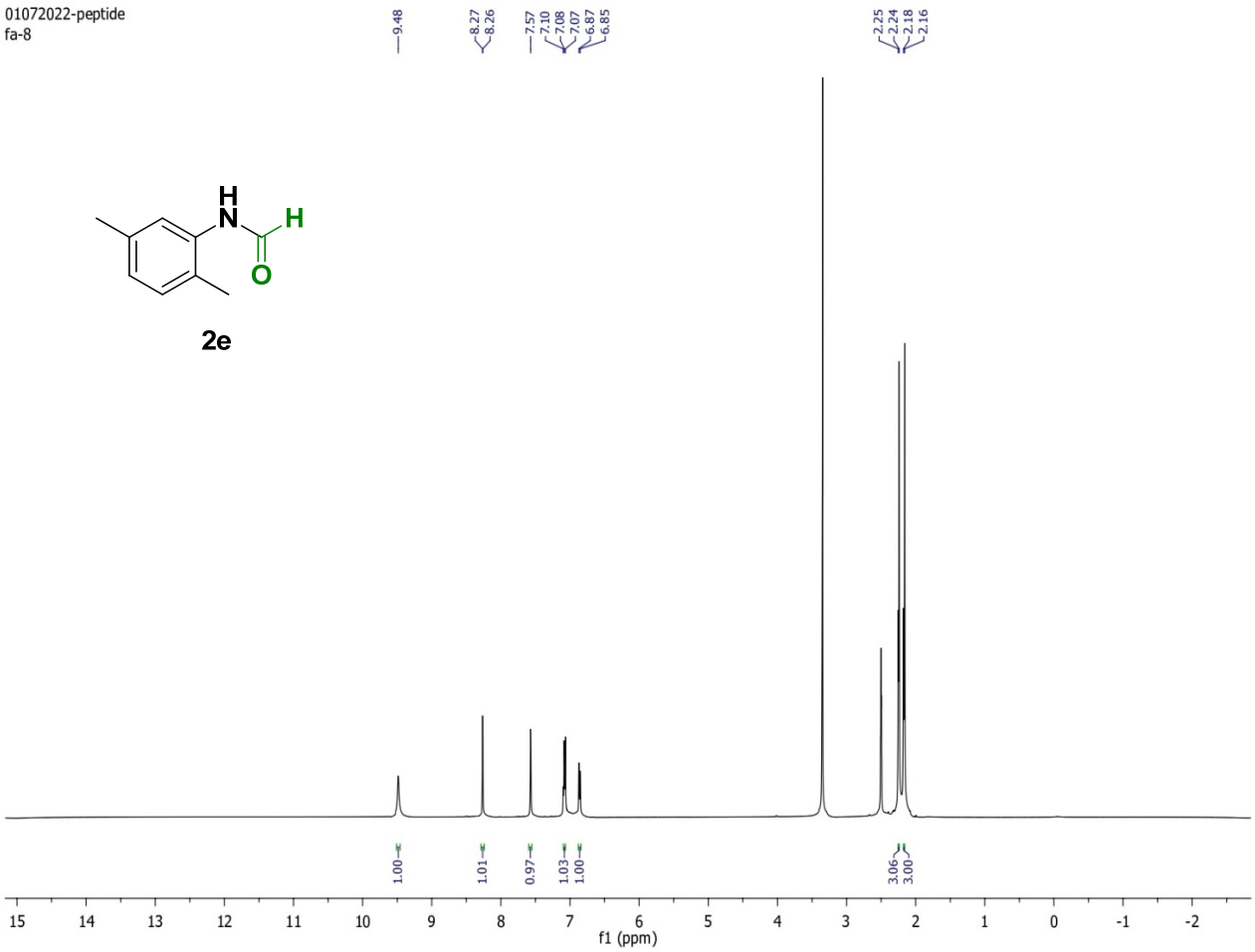
PEAK INFORMATION OF COMPOUND 2c

Peak #	Ret Time [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area %
1	2.155	0.1446	1.81149e4	2021.15210	100.0000

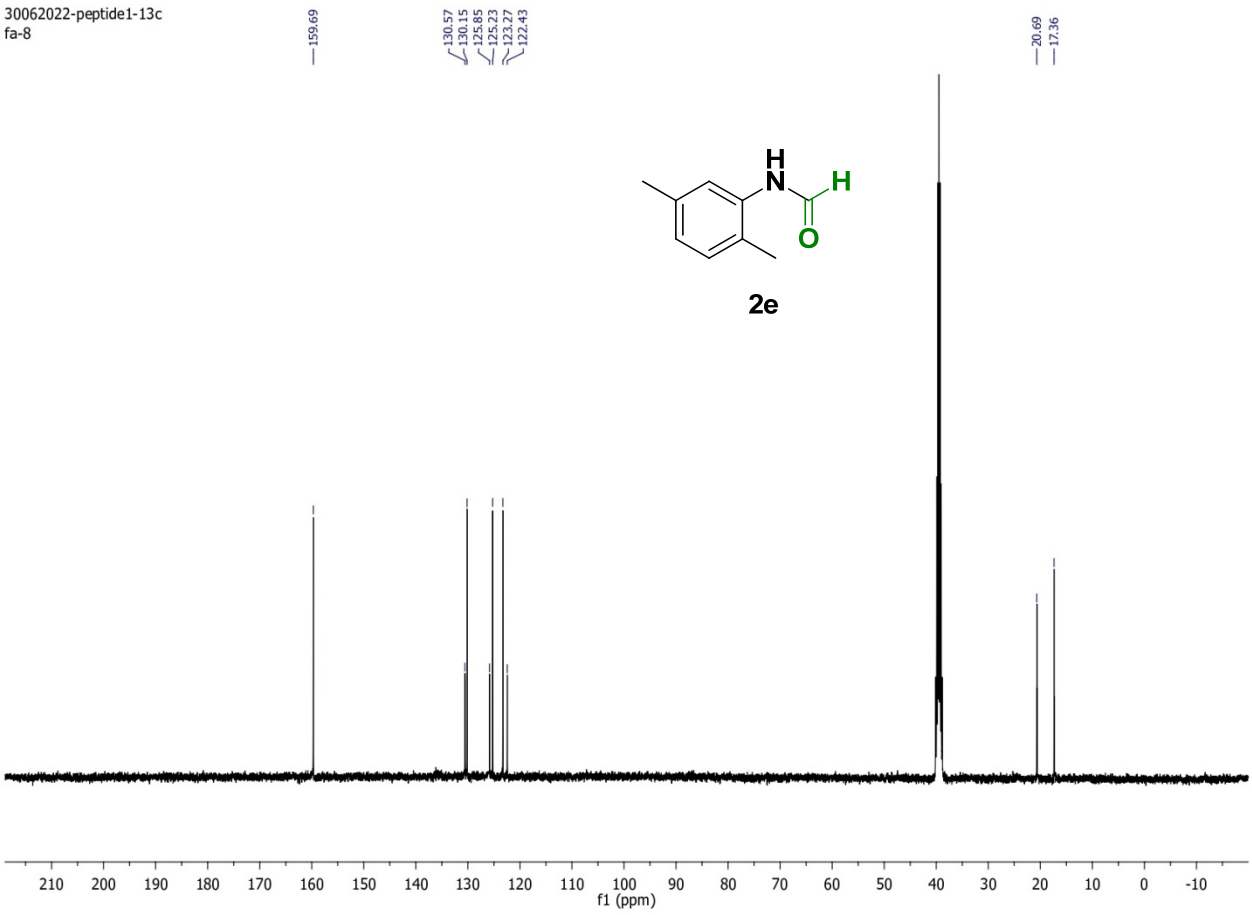
HPLC condition: RP-HPLC profile of 2c (method: water-acetonitrile (05-95%) in 10 min; detection wavelength at λ = 254nm; flow rate : 0.8 mL/min; column: Eclipse XDB-C18, pore size=5µm, diameter x length = 4.6 x 150mm.



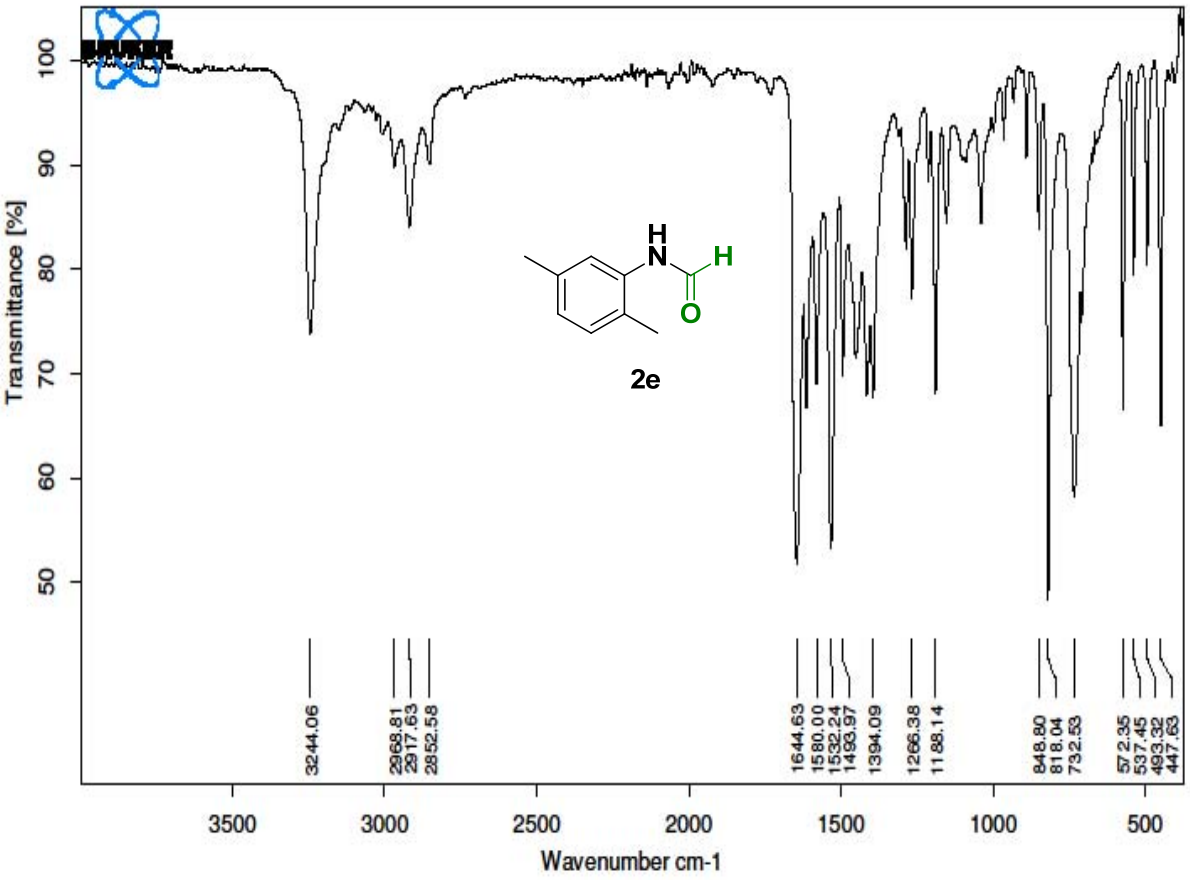
HRMS SPECTRUM OF COMPOUND 2d



^{1}H NMR SPECTRUM OF COMPOUND 2e



^{13}C NMR SPECTRUM OF COMPOUND 2e



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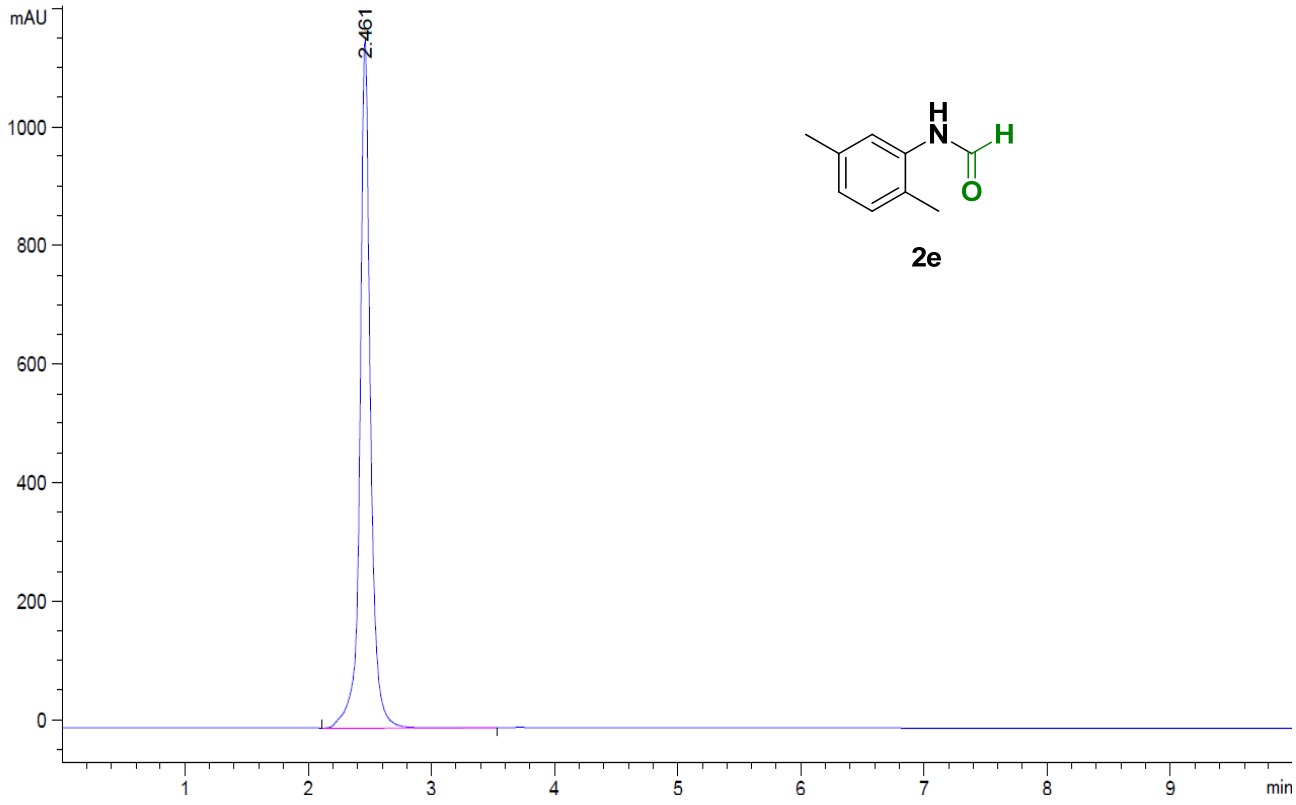
IR-SPECTRA OF COUMPOUND 2e



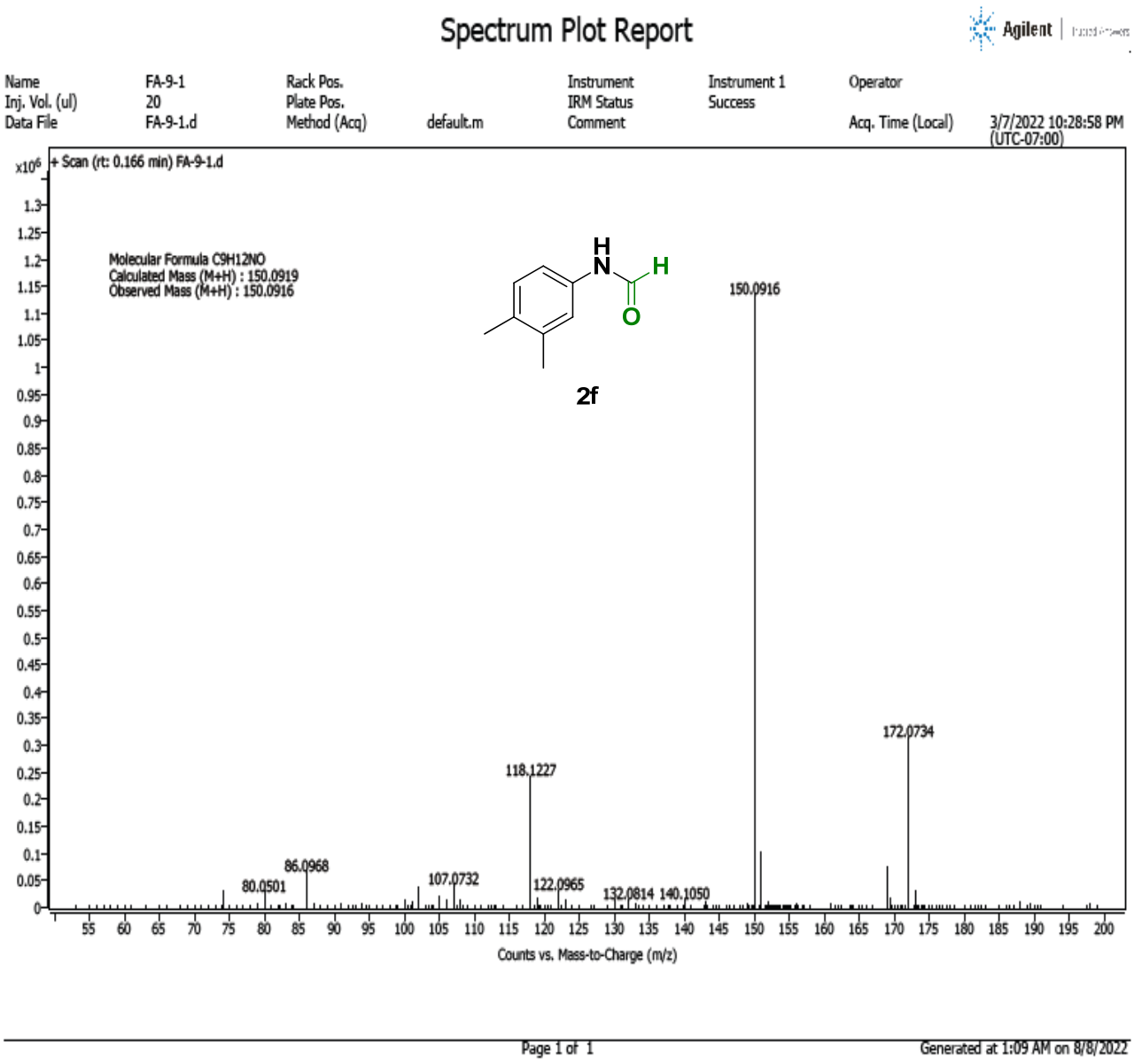
2e

HPLC OF COMPOUND 2e

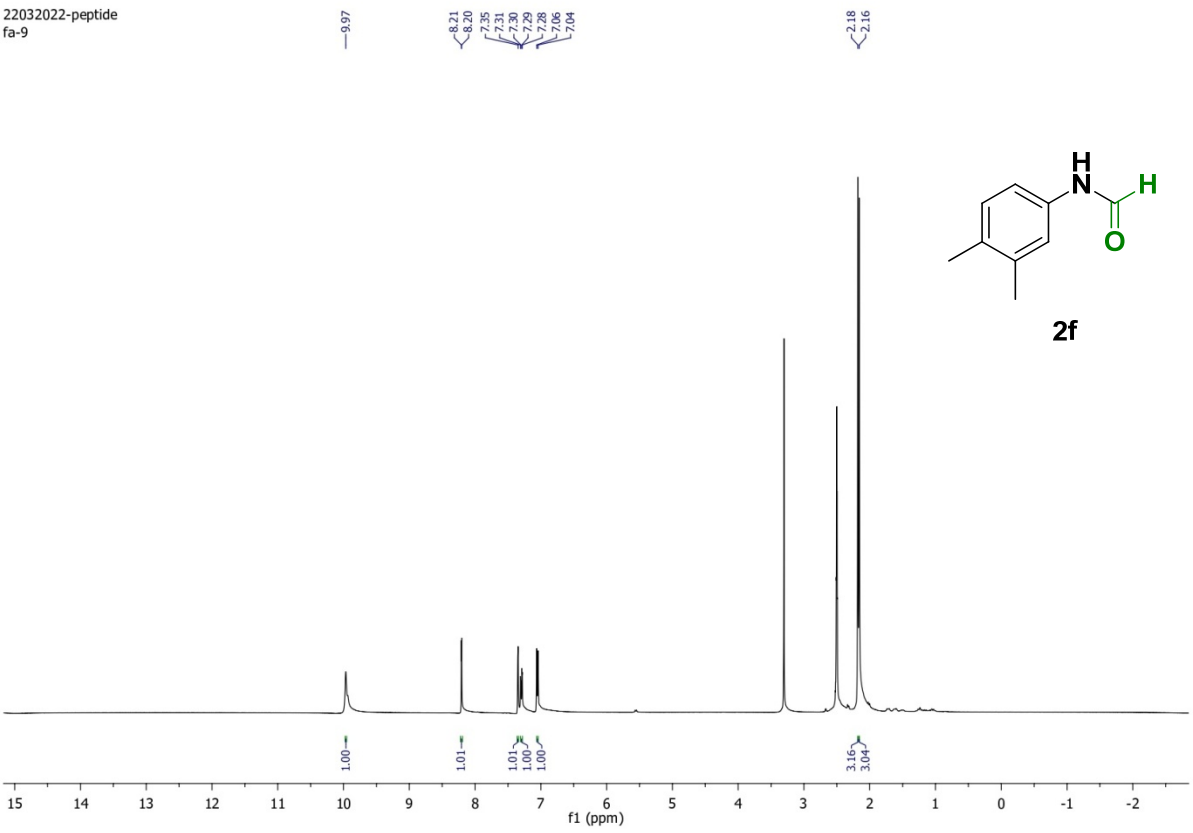
PEAK INFORMATION OF COMPOUND 2e

Peak #	Ret Time [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area %
1	2.461	0.0874	6917.81836	1162.57227	100.0000

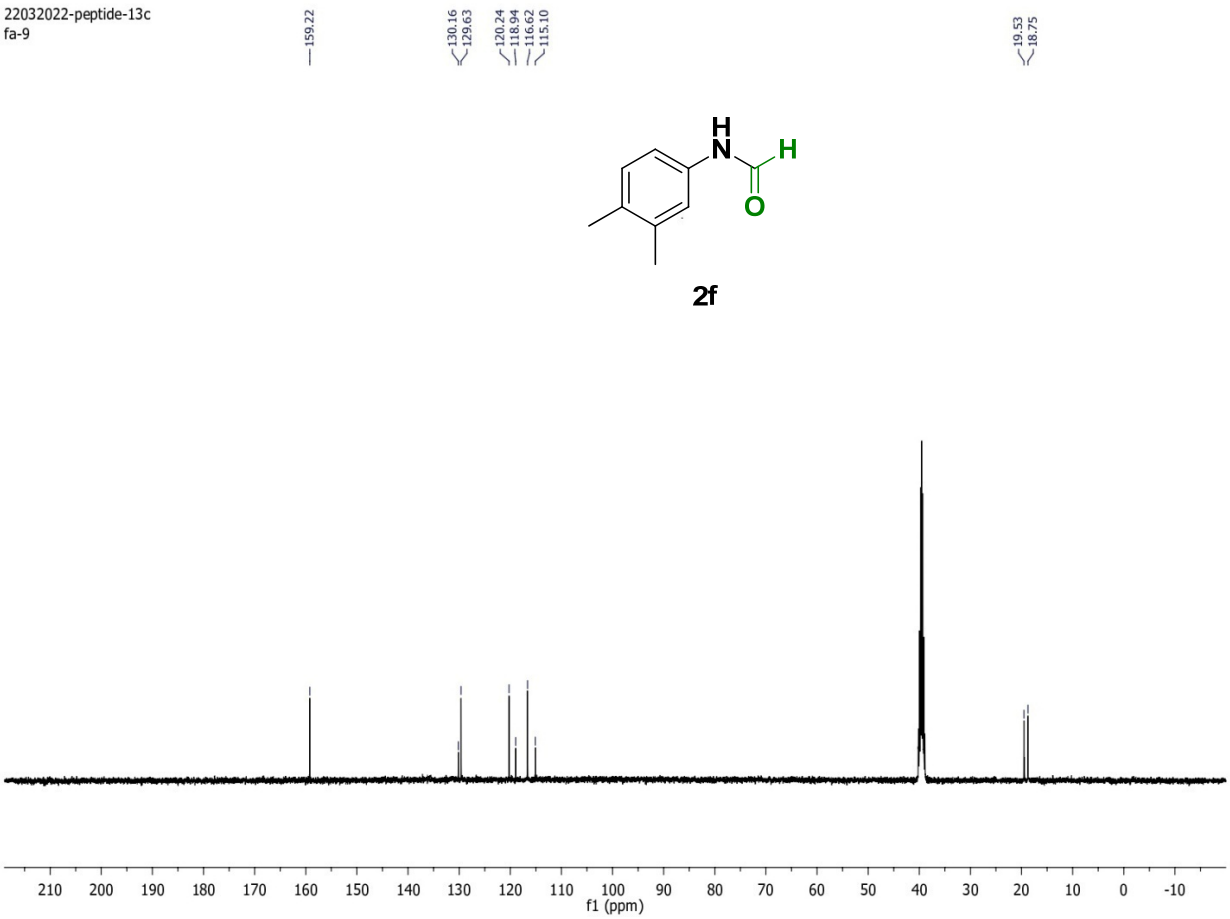
HPLC condition: RP-HPLC profile of 2e (method: water-acetonitrile (20-80%) in 10 min; detection wavelength at λ = 254nm; flow rate : 0.7 mL/min; column: Eclipse XDB-C18, pore size=5µm, diameter x length = 4.6 x 150mm.



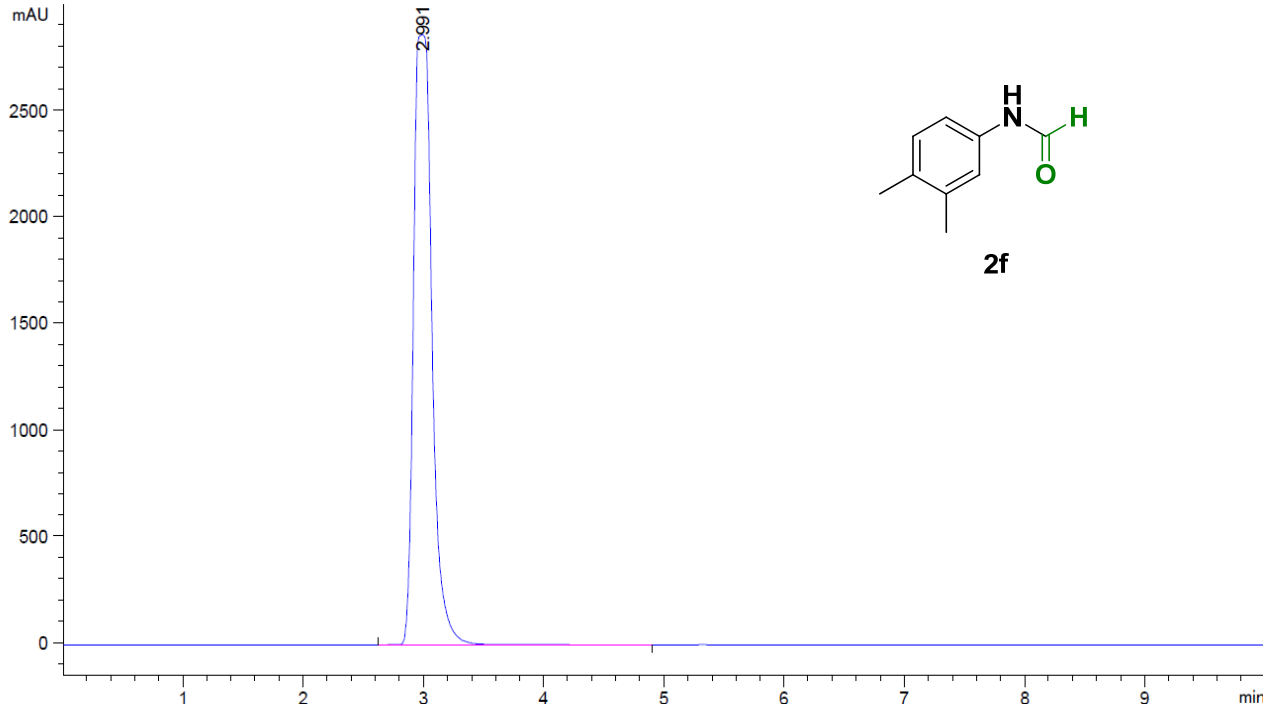
HRMS SPECTRUM OF COMPOUND 2f



^{1}H NMR SPECTRUM OF COMPOUND 2f



^{13}C NMR SPECTRUM OF COMPOUND 2f

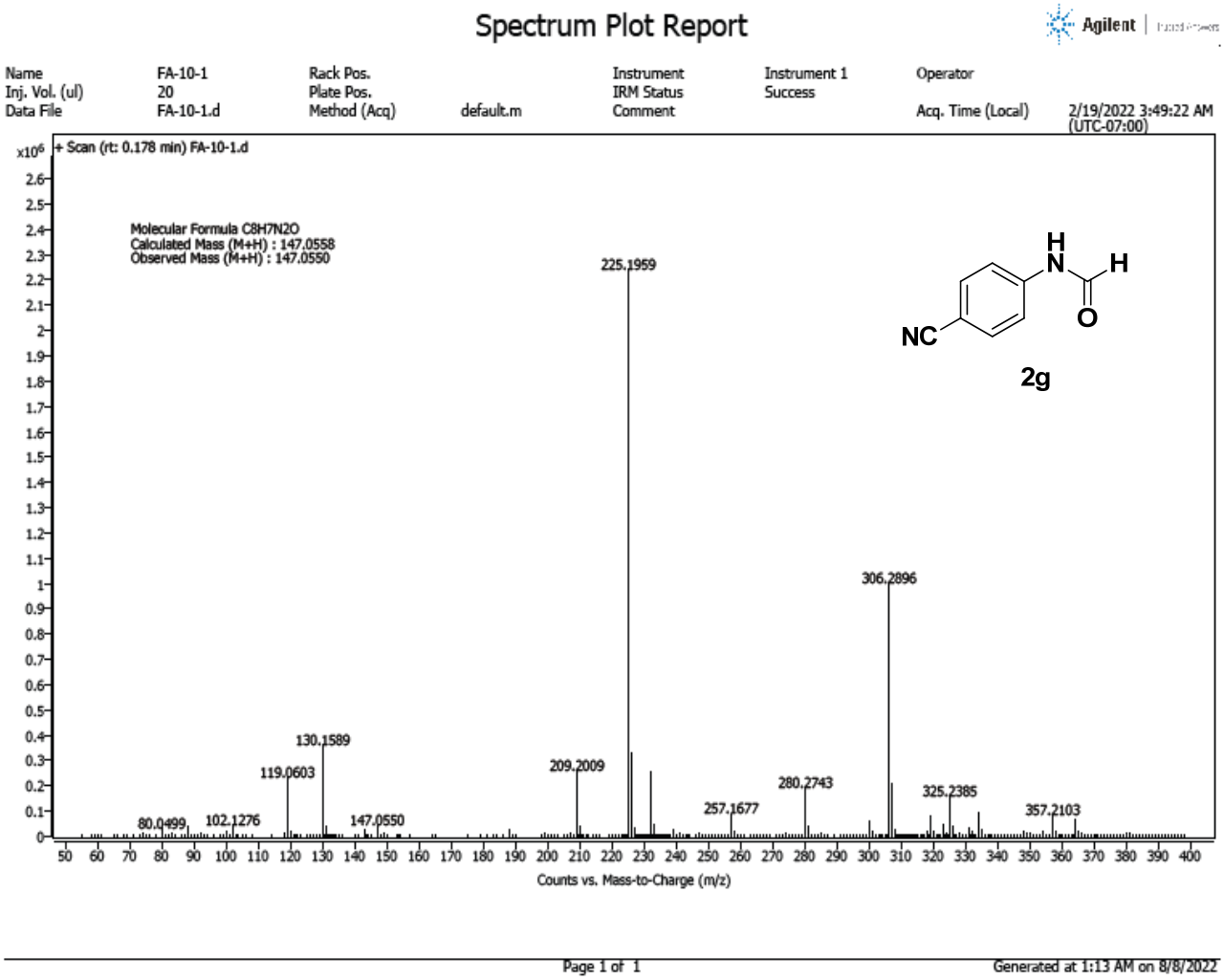


HPLC OF COMPOUND 2f

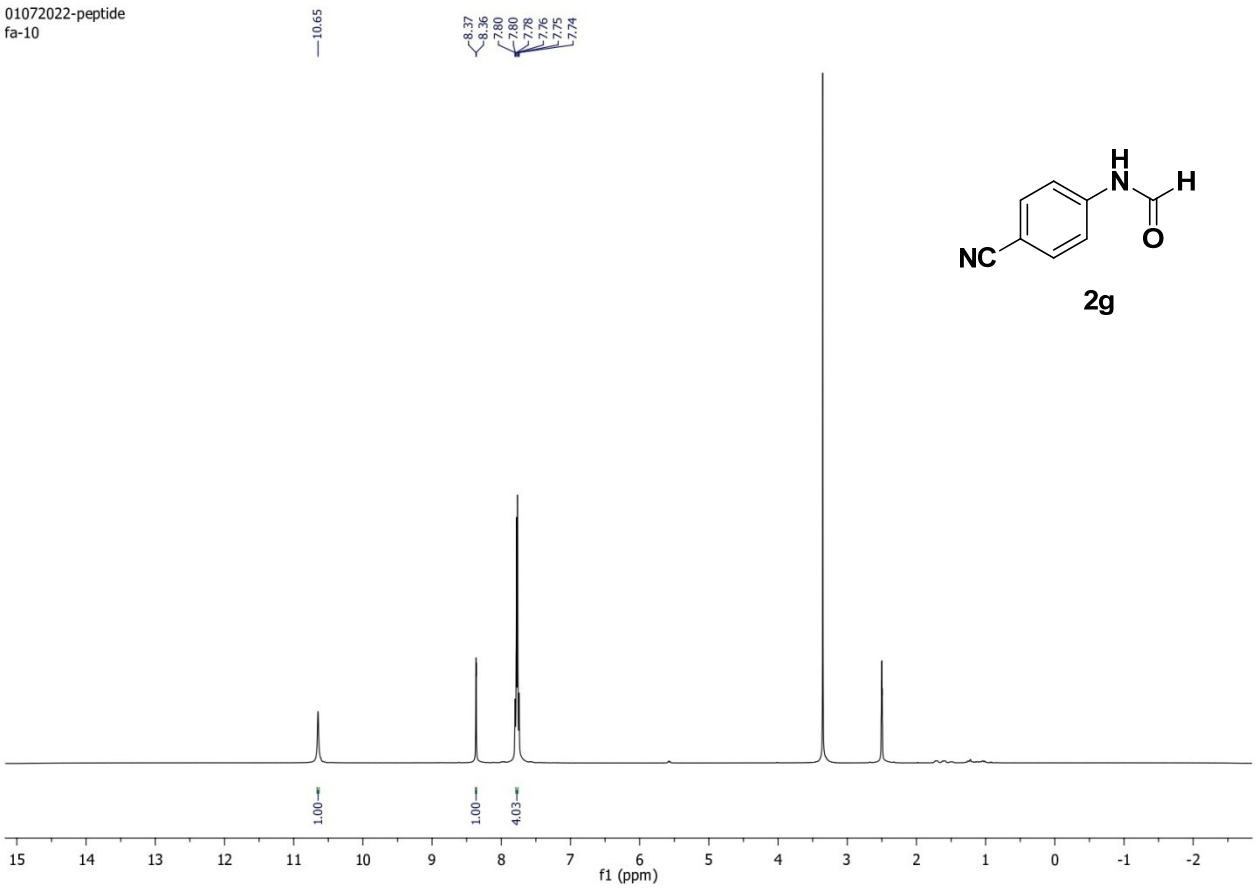
PEAK INFORMATION OF COMPOUND 2f

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

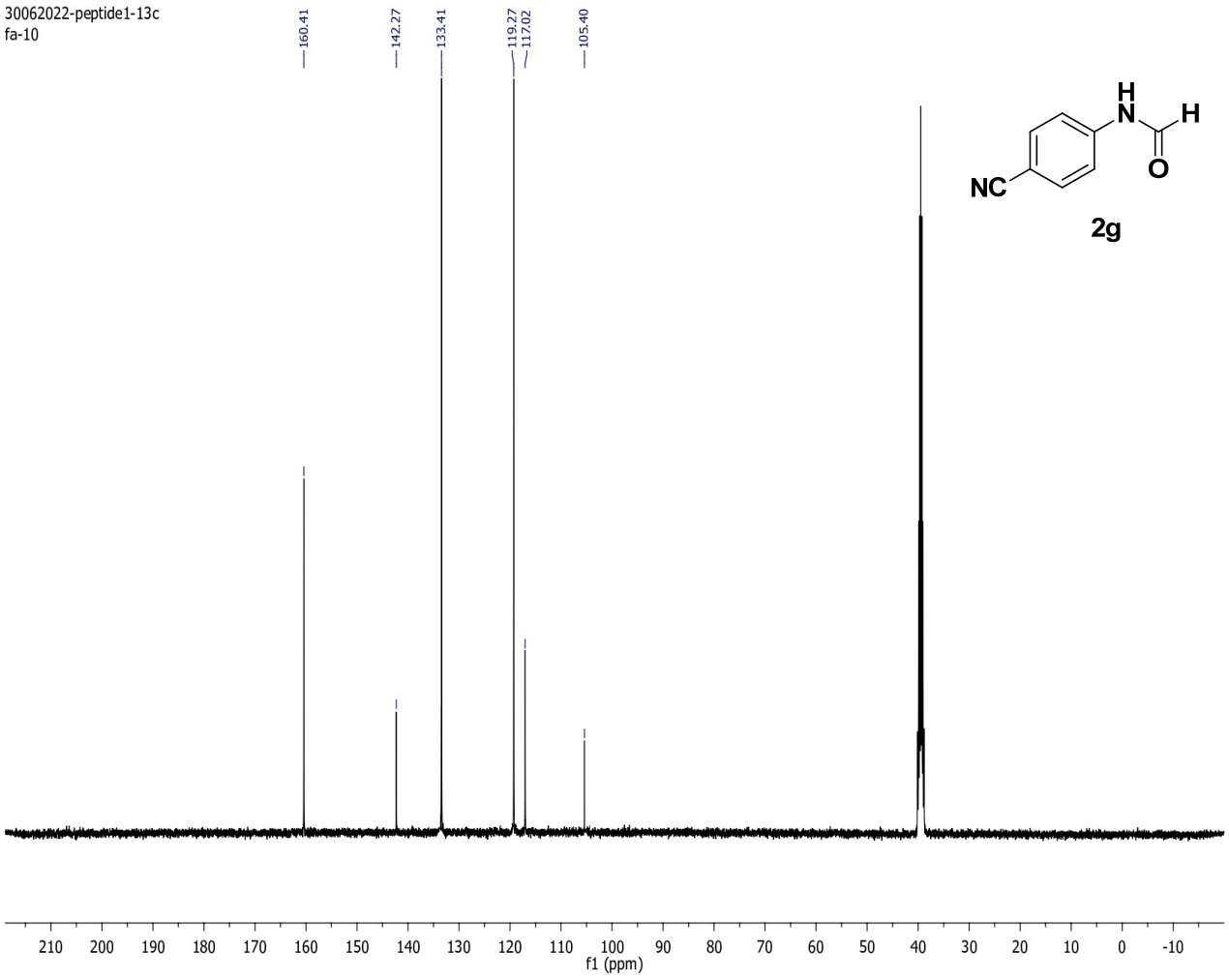
HPLC condition: RP-HPLC profile of 2f (method: water-acetonitrile (05-95%) in 10 min; detection wavelength at λ = 254nm; flow rate : 0.6 mL/min; column: Eclipse XDB-C18, pore size=5µm, diameter x length = 4.6 x 150mm.



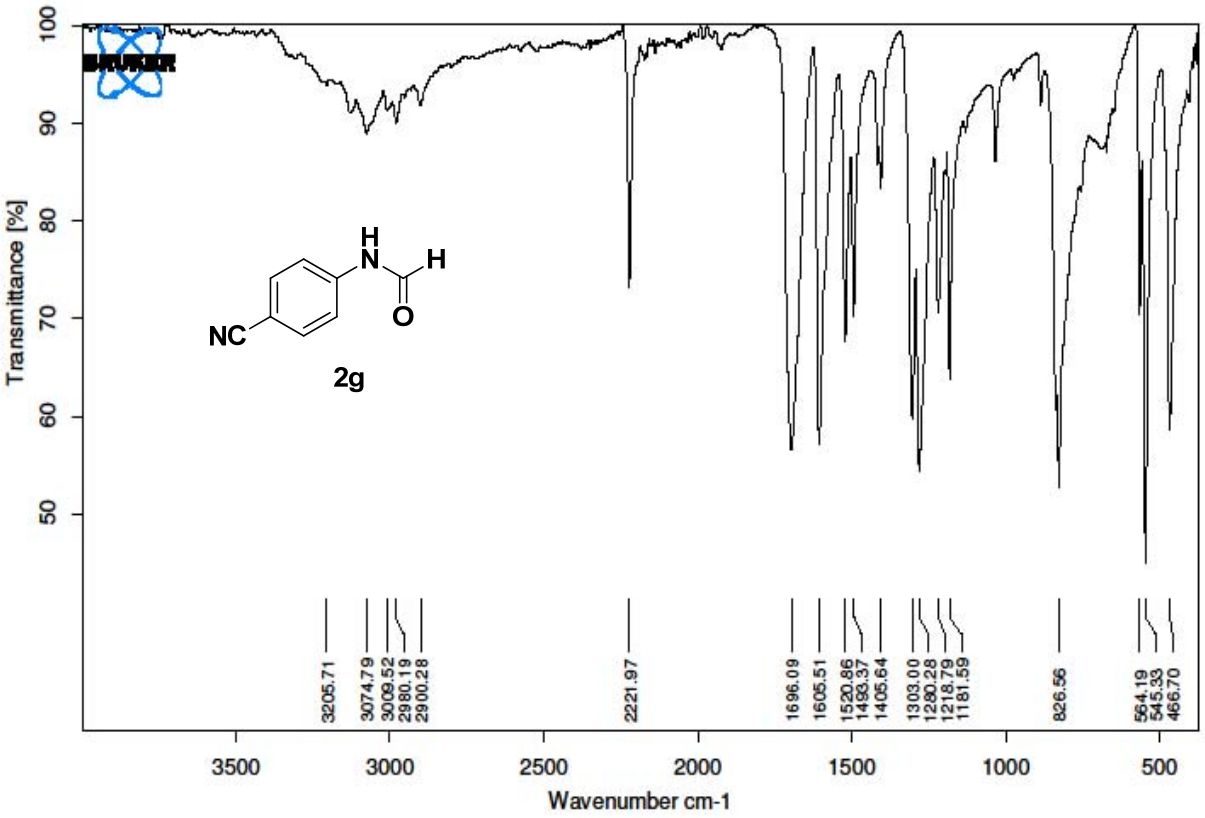
HRMS SPECTRUM OF COMPOUND 2g



^{1}H NMR SPECTRUM OF COMPOUND 2g



^{13}C NMR SPECTRUM OF COMPOUND 2g



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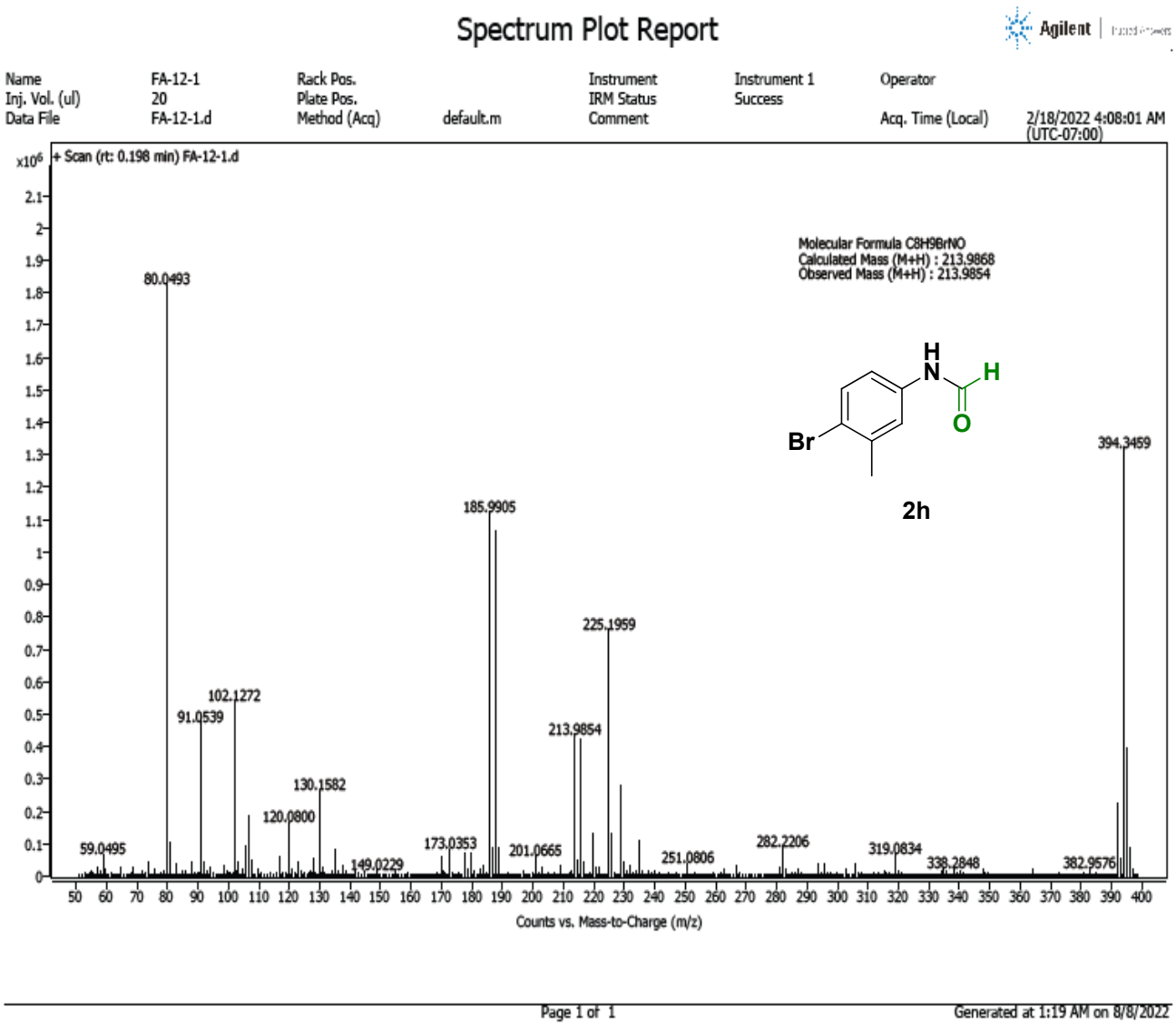
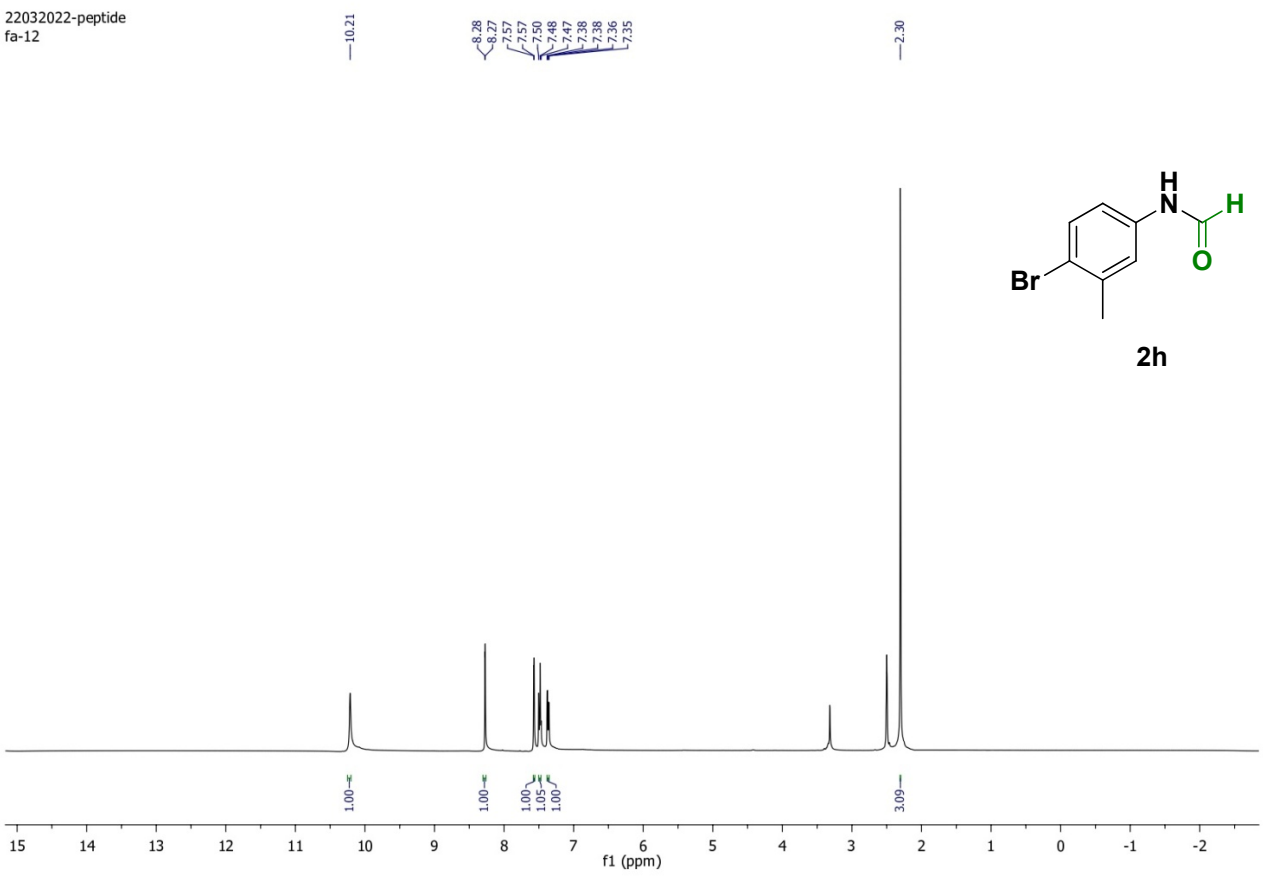
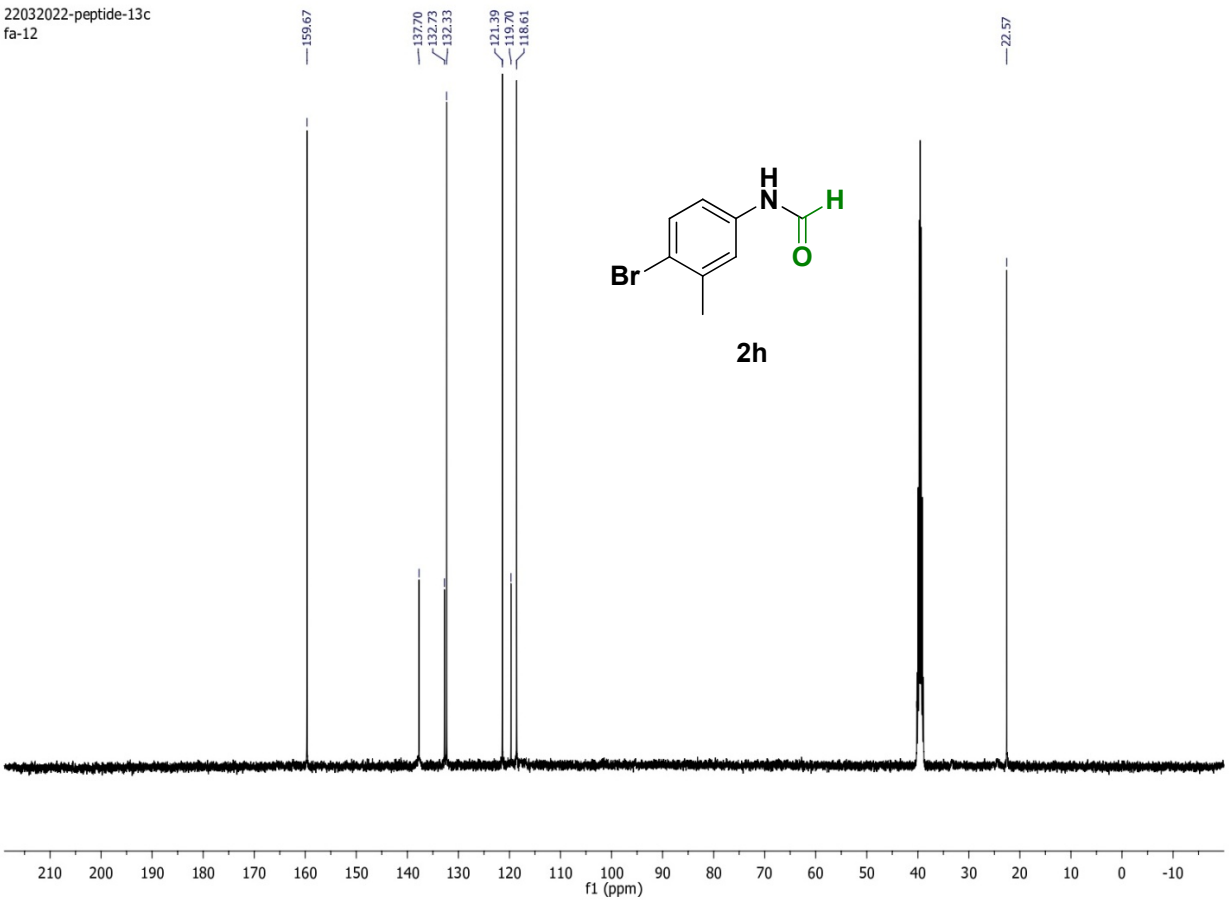
IR- SPECTRA OF COMPOUND 2g



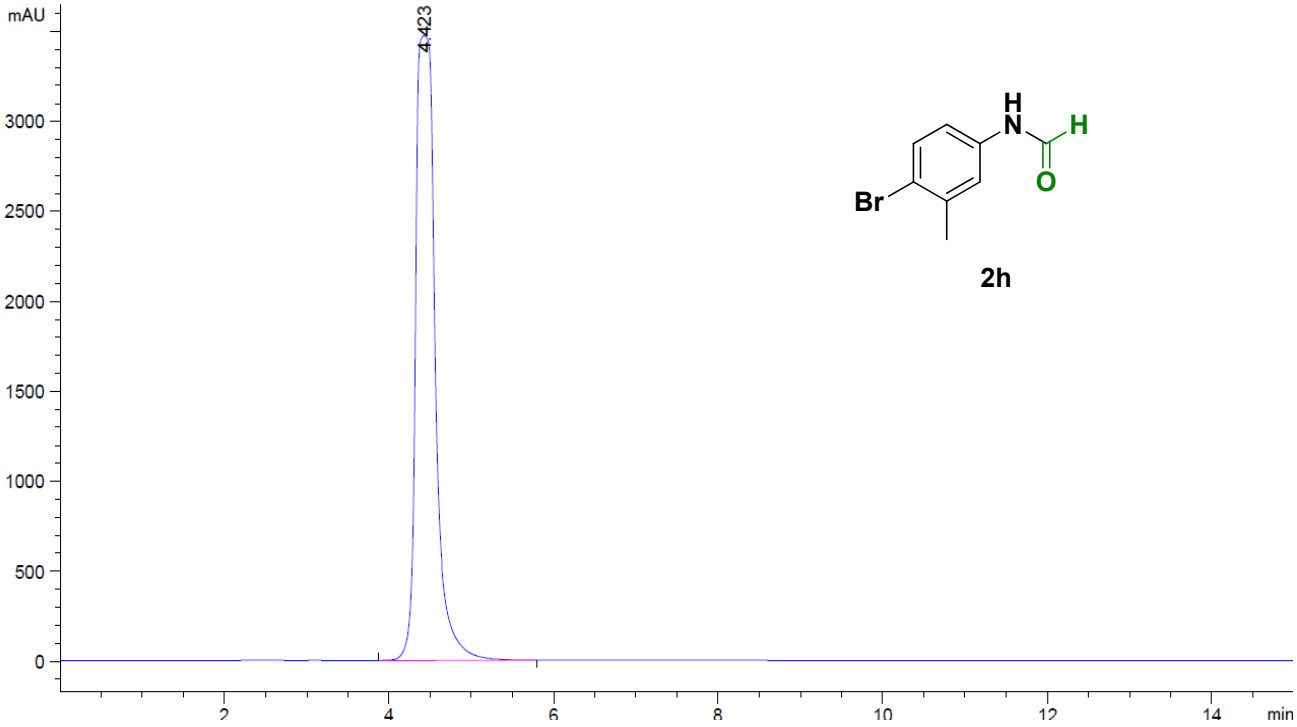
HRMS SPECTRUM OF COMPOUND 2h



^{1}H NMR SPECTRUM OF COMPOUND 2h



^{13}C NMR SPECTRUM OF COMPOUND 2h

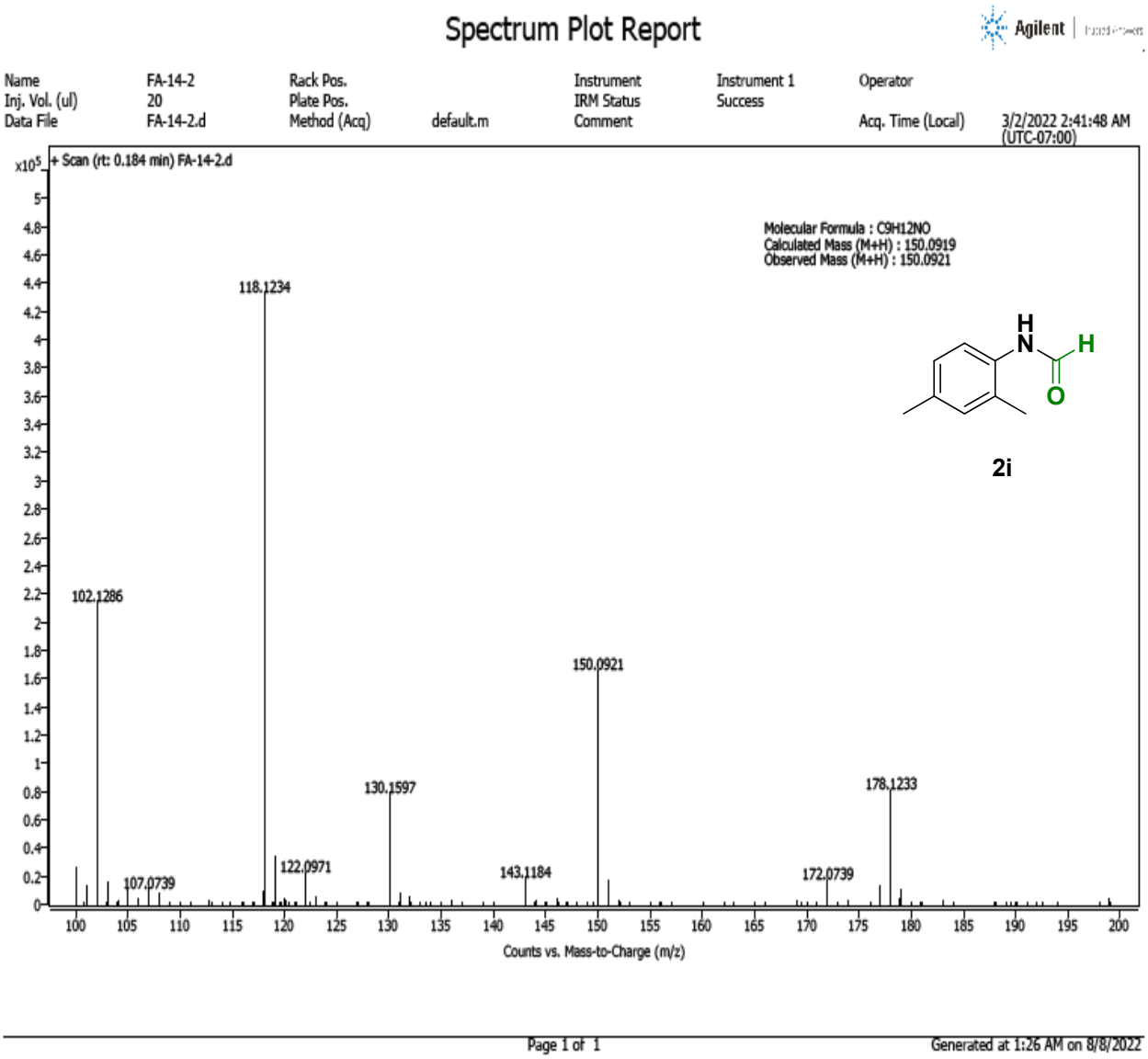


HPLC OF COMPOUND 2h

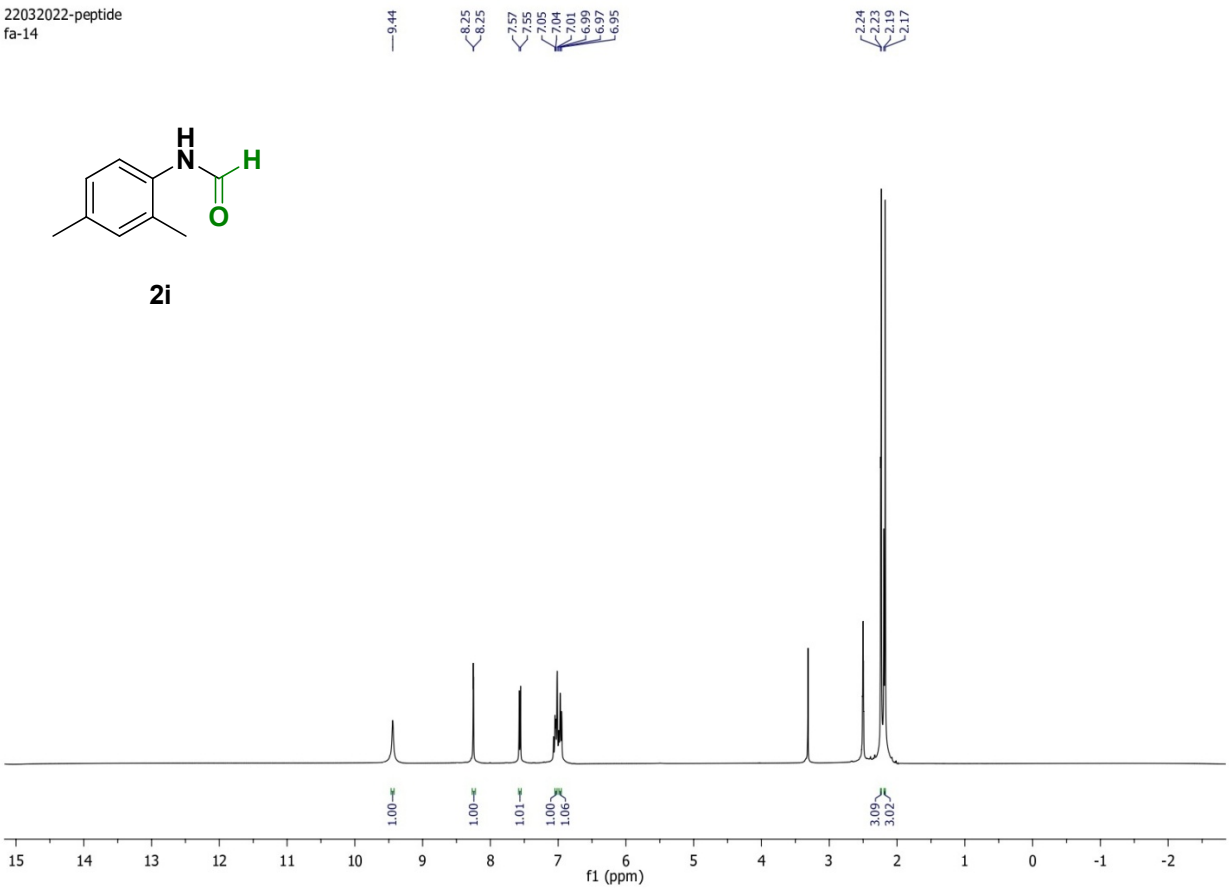
PEAK INFORMATION OF COMPOUND 2h

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

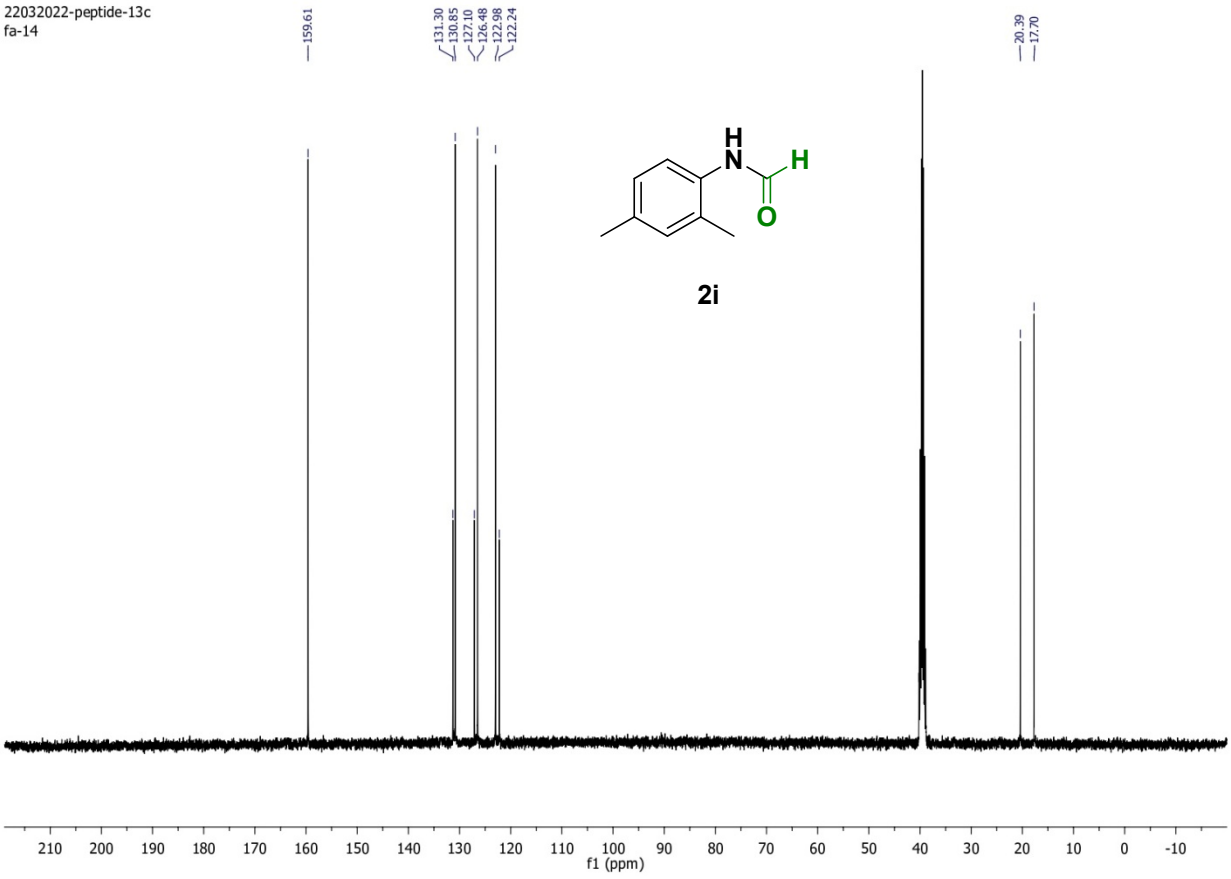
HPLC condition: RP-HPLC profile of 2h (method: water-acetonitrile (15-85%) in 15 min; detection wavelength at λ = 254nm; flow rate : 0.4 mL/min; column: Eclipse XDB-C18, pore size=5µm, diameter x length = 4.6 x 150mm.



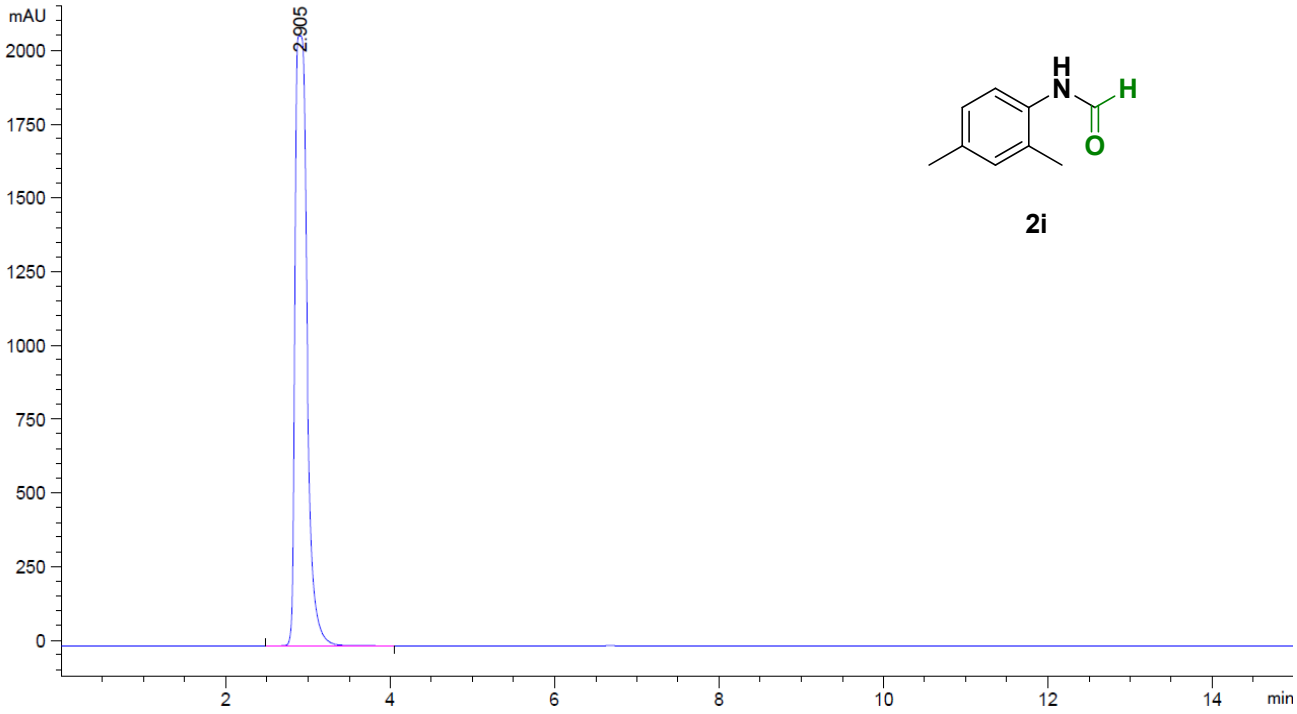
HRMS SPECTRUM OF COMPOUND 2i



^{1}H NMR SPECTRUM OF COMPOUND 2i



^{13}C NMR SPECTRUM OF COMPOUND 2i



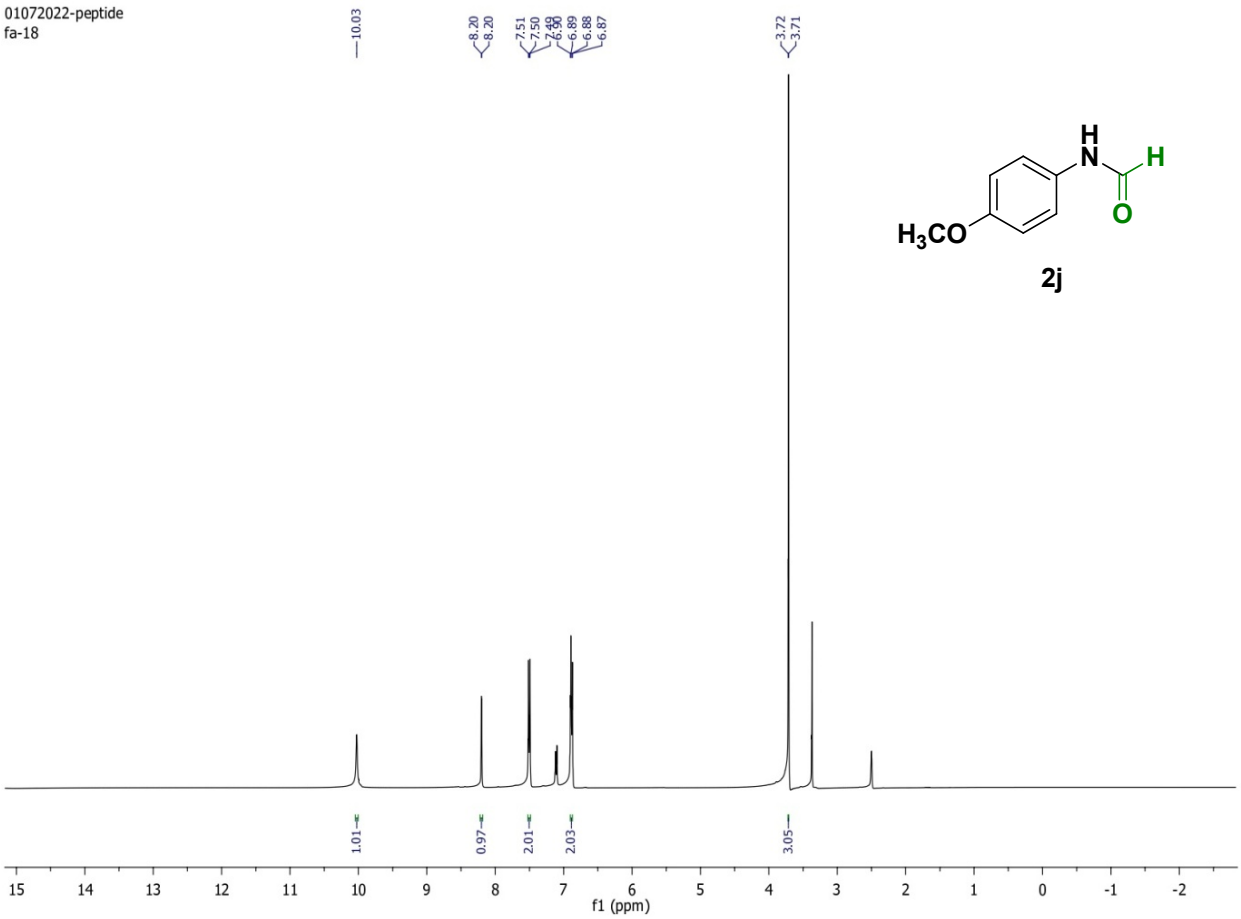
2i

HPLC OF COMPOUND 2i

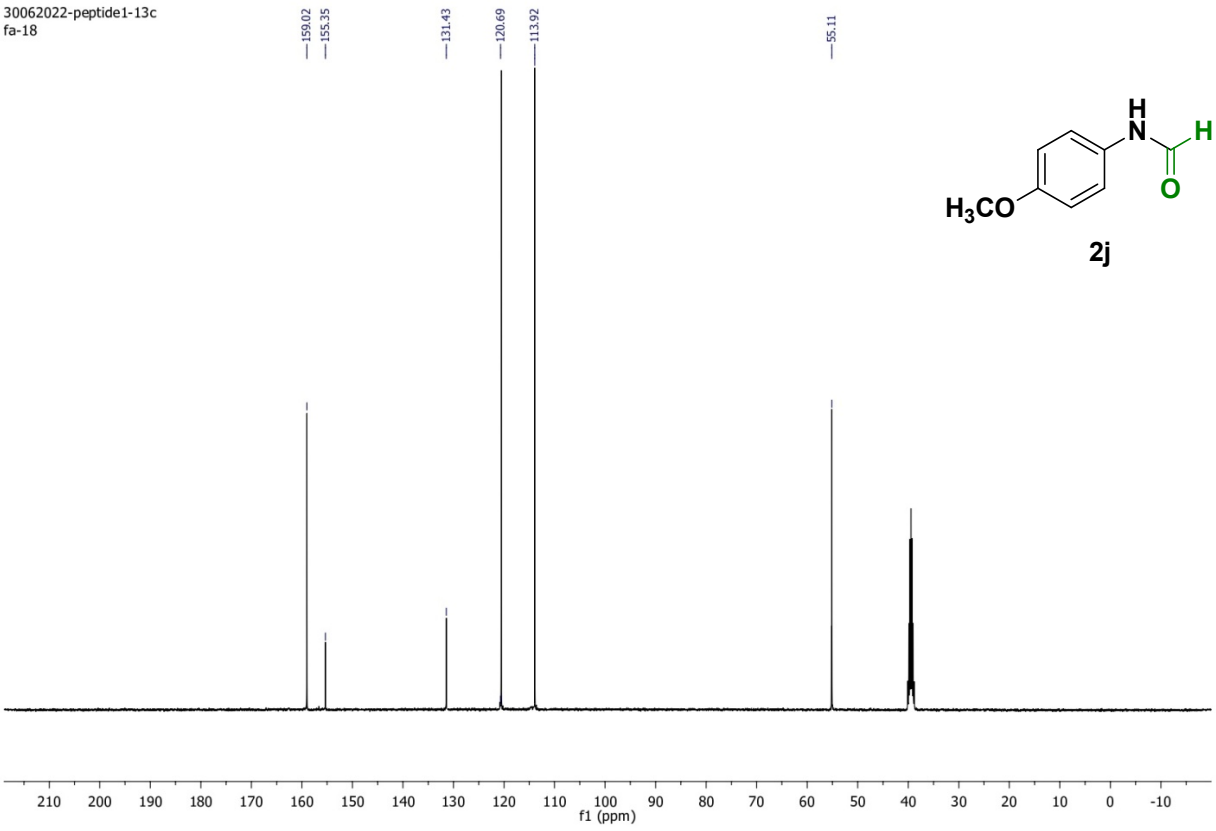
PEAK INFORMATION OF COMPOUND 2i

Peak #	Ret Time [min]	Width [min]	Area [mAU*S]	Height [mAU]	Area %
1	2.905	0.1599	2.08942e4	2067.22754	100.0000

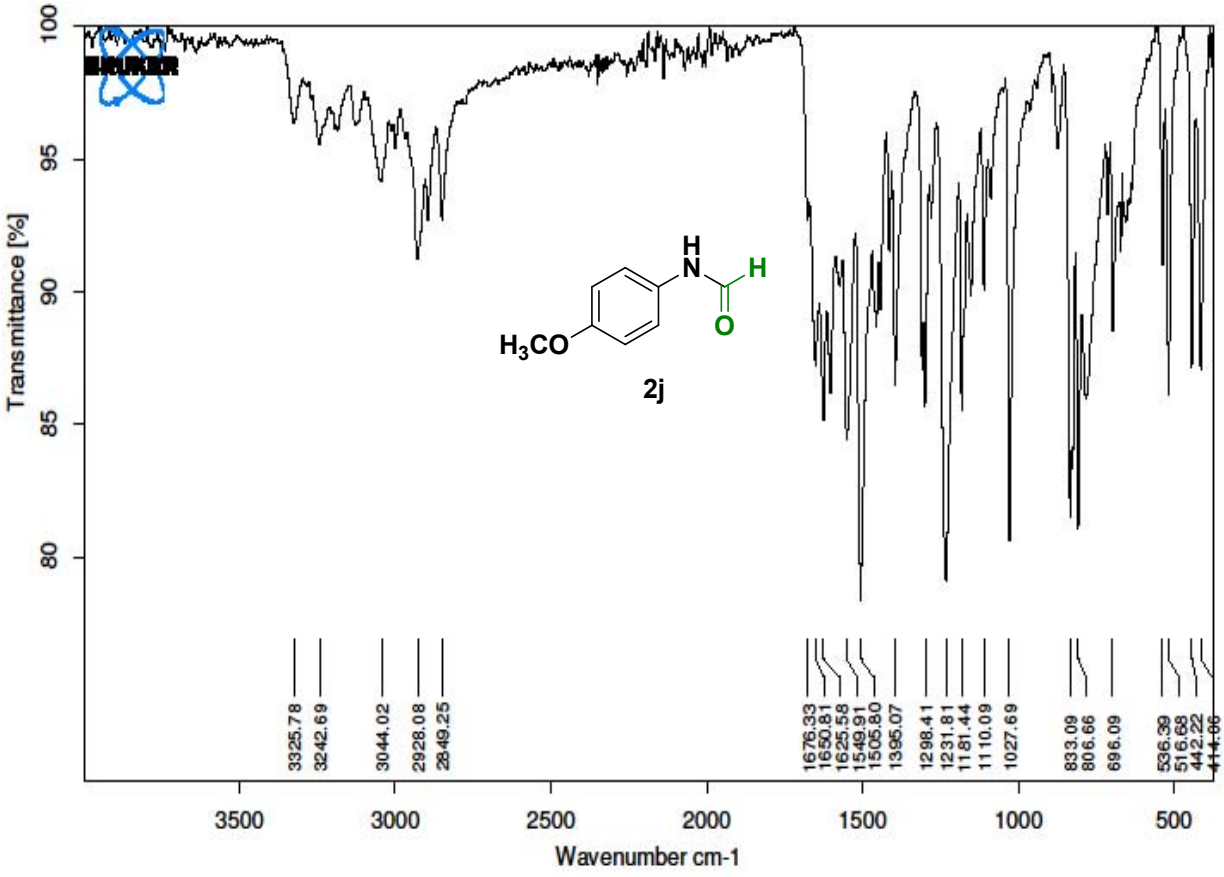
HPLC condition: RP-HPLC profile of 2i (method: water-acetonitrile (10-90%) in 15 min; detection wavelength at λ = 254nm; flow rate : 0.6 mL/min; column: Eclipse XDB-C18, pore size=5µm, diameter x length = 4.6 x 150mm.



^{1}H NMR SPECTRUM OF COMPOUND 2j



^{13}C NMR SPECTRUM OF COMPOUND 2j



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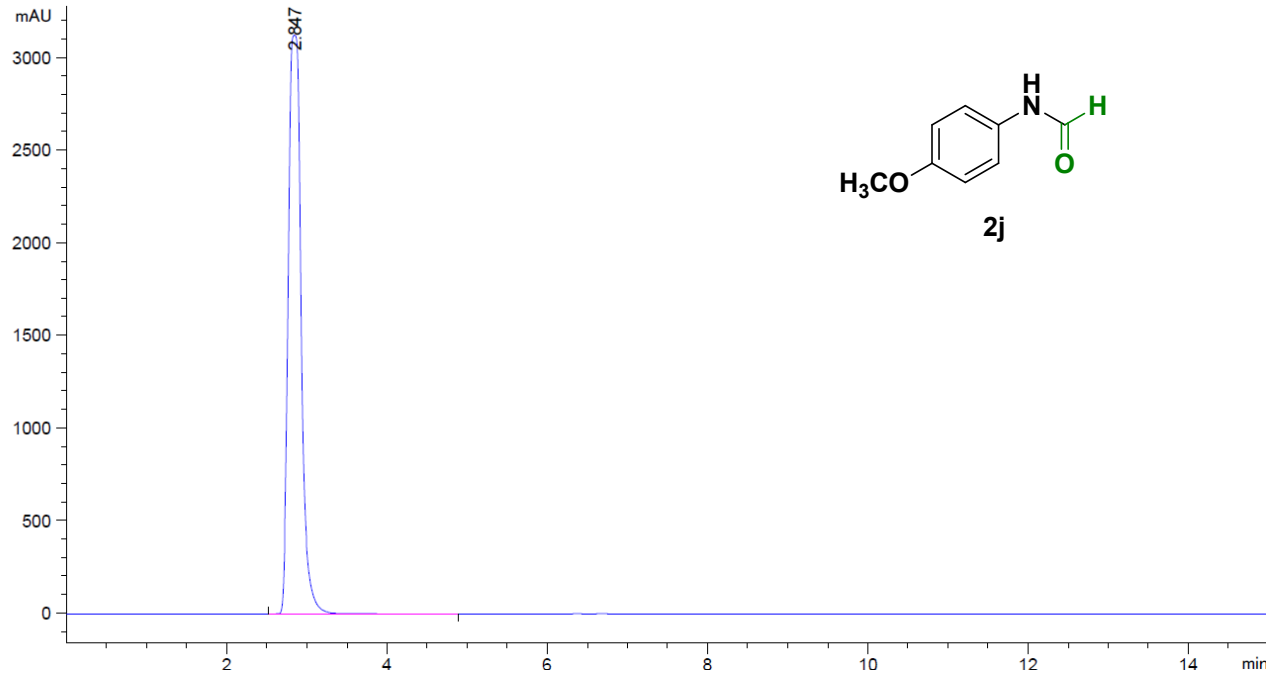
IR- SPECTRA OF COMPOUND 2j



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 λ = 254nm; flow rate : 0.6 mL/min; column: Eclipse XDB-C18, pore size=5µm, diameter x length = 4.6 x 150mm.