Supporting Information

Synthesis of 3-Trifluoromethyl-1,2,4-triazolines and 1,2,4-Triazoles by Tandem Addition/Cyclization of Trifluoromethyl *N*-Acylhydrazones with Cyanamide

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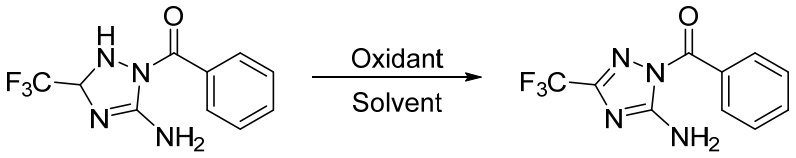
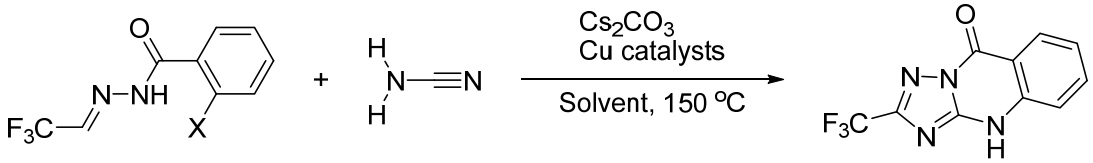
Table S1. Reaction optimization of the synthesis of trifluoromethylated 1,2,4-triazoles **3a**[a]



Entry	Oxidant	Time (h)	Yield[b] (%)
1	NIS	1.5	83
2	NBS	1.5	84
3	TCCA	1	6
4	TBHP	2	34
5	BPO	2	15
6	$CuCl_2$	5	trace

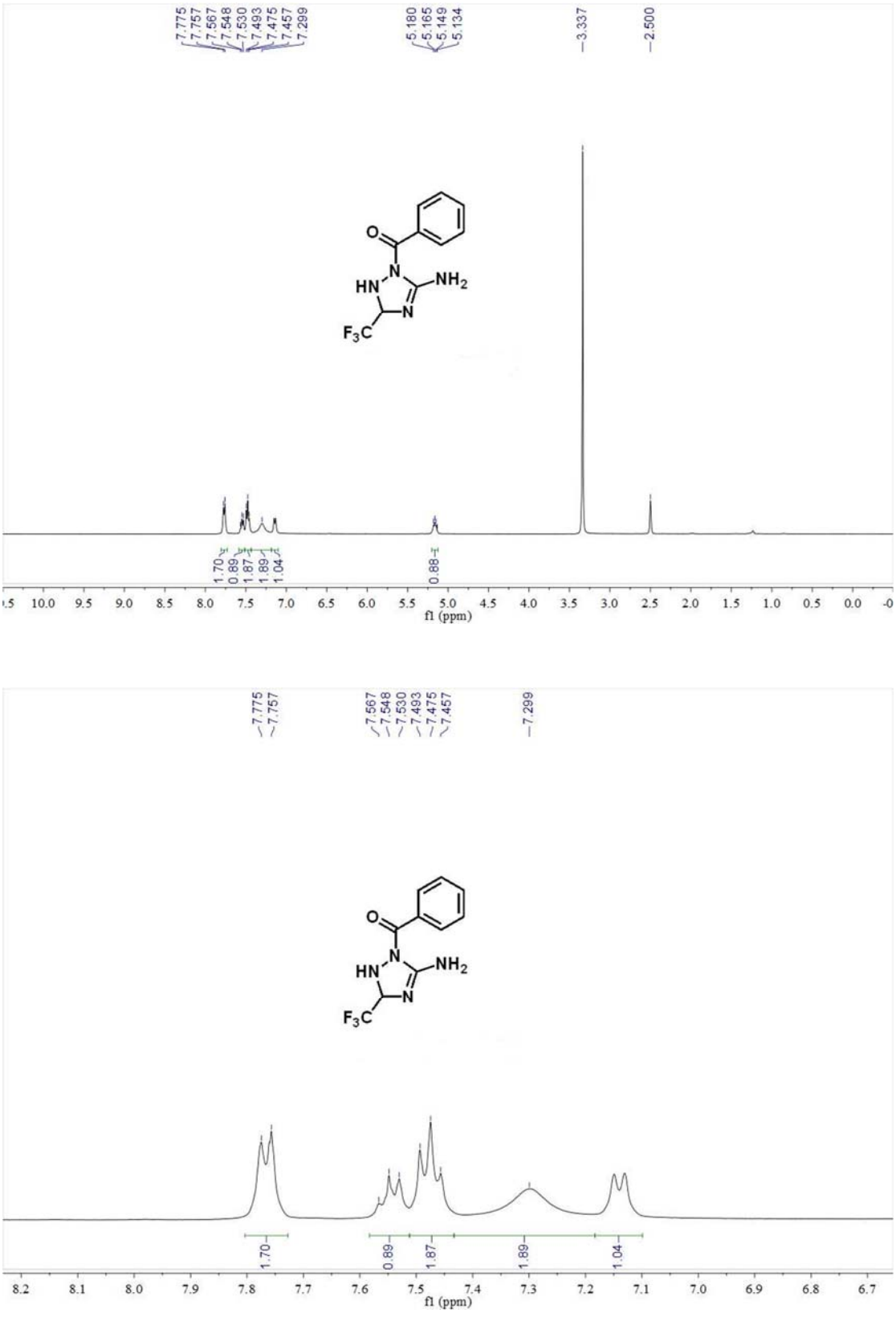
[a]Reaction conditions: All reactions were carried out by using 0.2 mmol of **2a** and 3.0 mmol of oxidant in 2 mL of CH_2Cl_2 at 40 °C. [b]Isolated yields.

Table S2. Reaction optimization of the synthesis of 3-trifluoromethyl-[1,2,4]triazolo[1,5-a]pyrimidines[a]

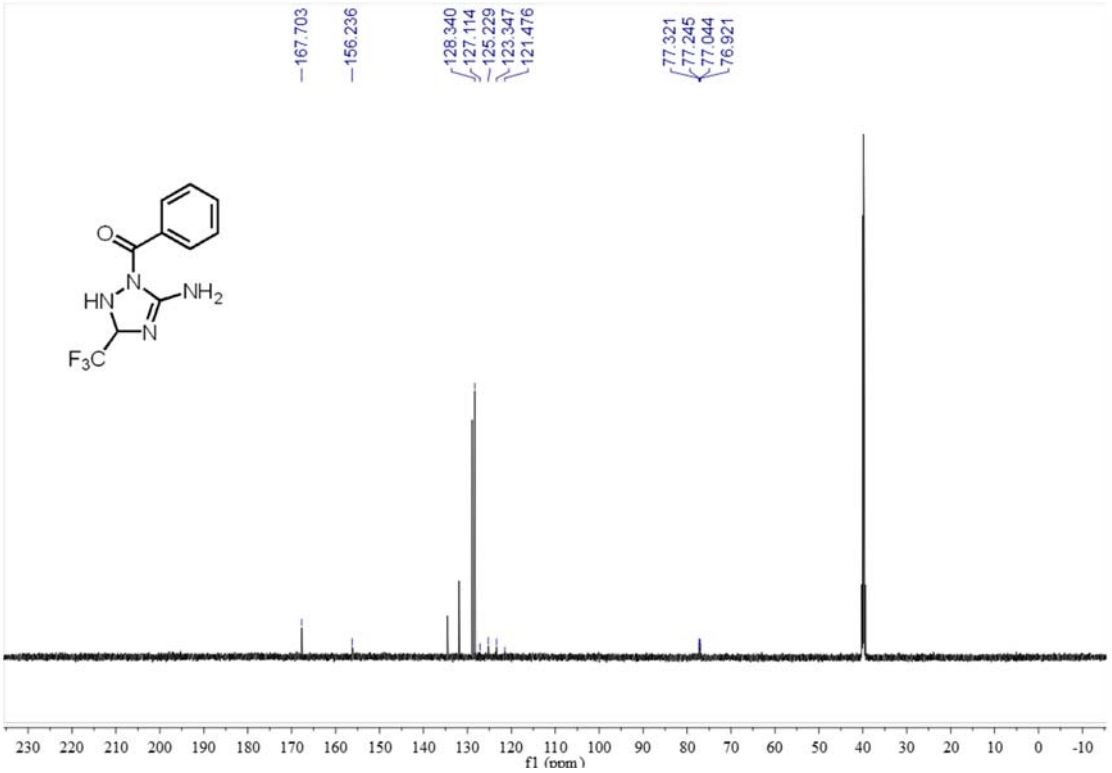


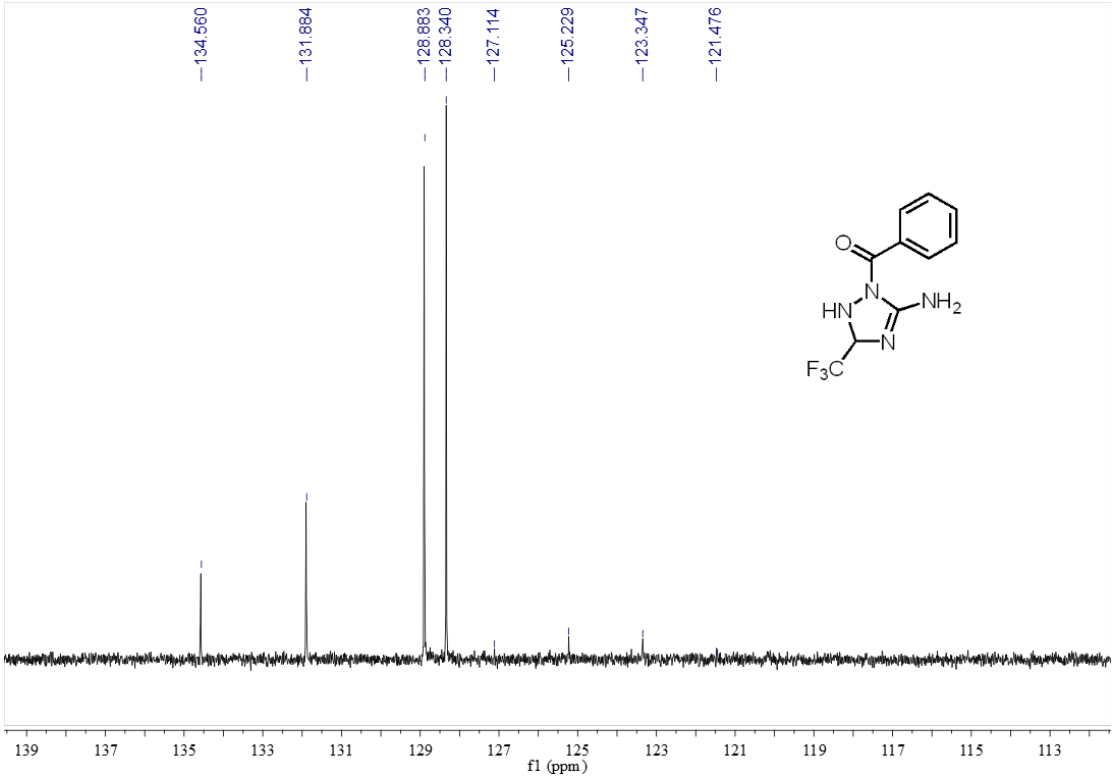
Entry	Cu catalysts	Solvent	Time (h)	Yield[b] (%)
1	CuI	DMSO	2.5	20
2	CuI	DMF	0.5	78
3	$CuCl_2$	DMF	0.5	75
4	$CuBr_2$	DMF	2	41
5	$Cu(OAc)_2$	DMF	2	63
6	CuI	DMF	1.5	71
7	Cu powder	DMF	9	NR

[a]Reaction conditions: All reactions were carried out by using 0.2 mmol of **1a´**, 2.5 equiv. of cyanamide, 2.5 equiv. of Cs_2CO_3 and 0.15 equiv. of Cu catalysts in 2 mL of solvent at 150 °C under air atmosphere. [b]Isolated yields.

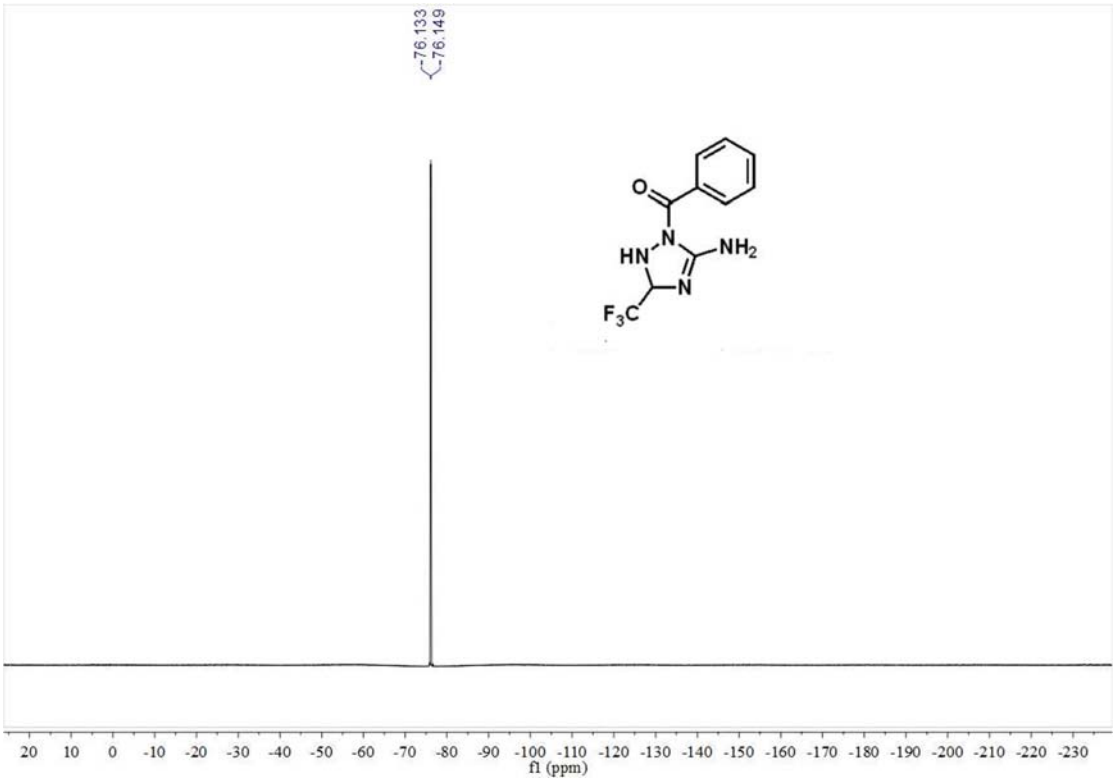


^{1}H NMR (400 MHz) spectrum of **2a** in DMSO-d_6

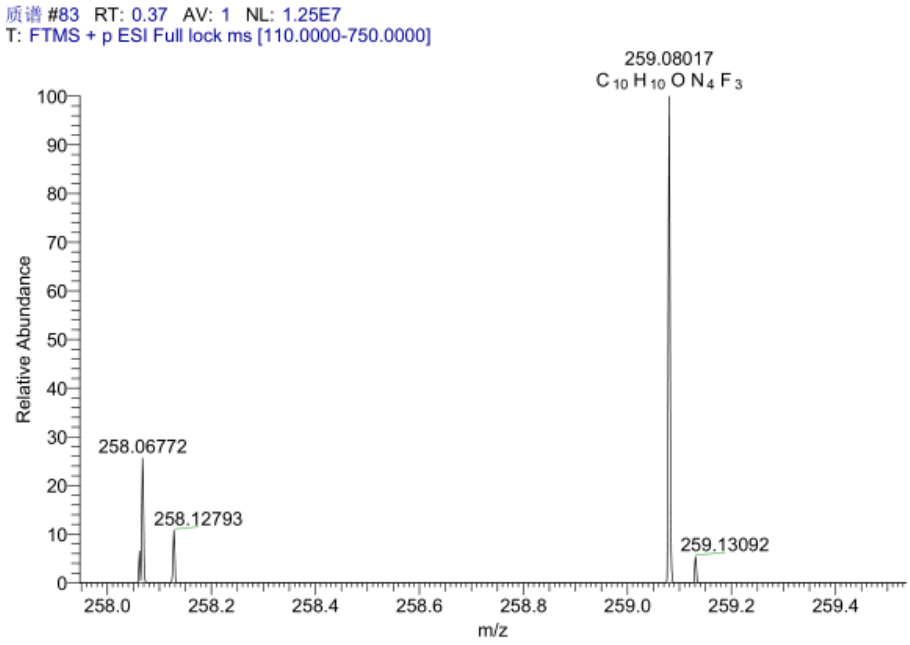




$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **2a** in DMSO-*d*$_6$



^{19}F NMR (376 MHz) spectrum of **2a** in DMSO-d_6



质谱#83 RT: 0.37
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m/z= 257.94692-259.53593

m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	Composition
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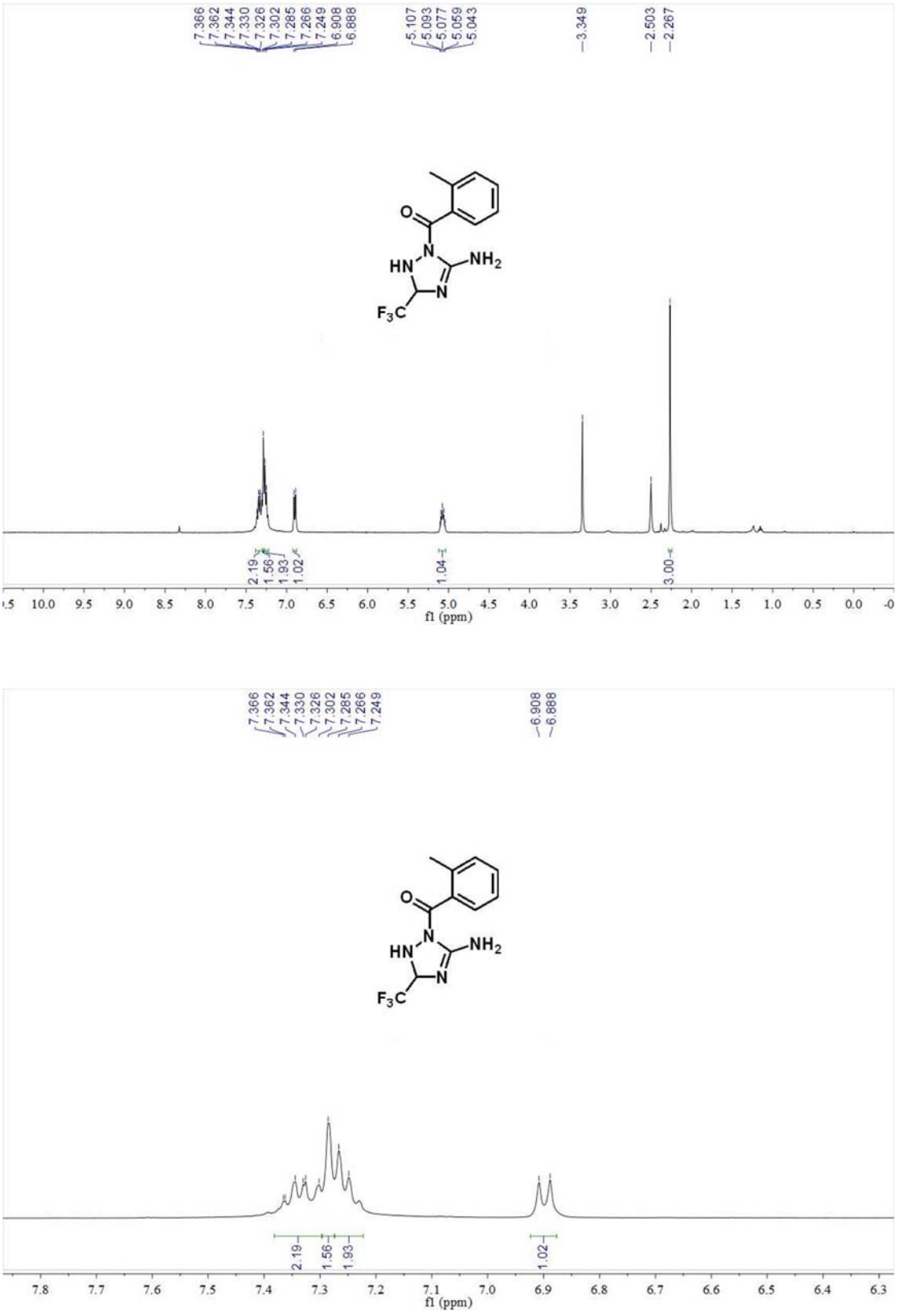
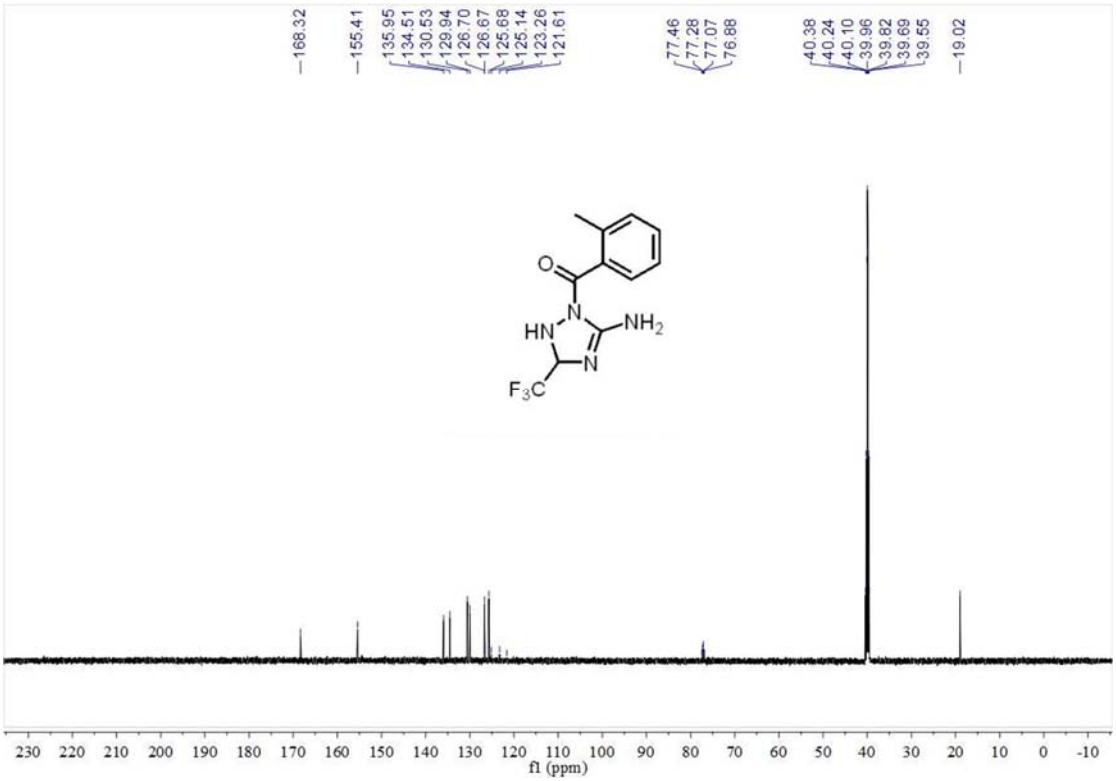
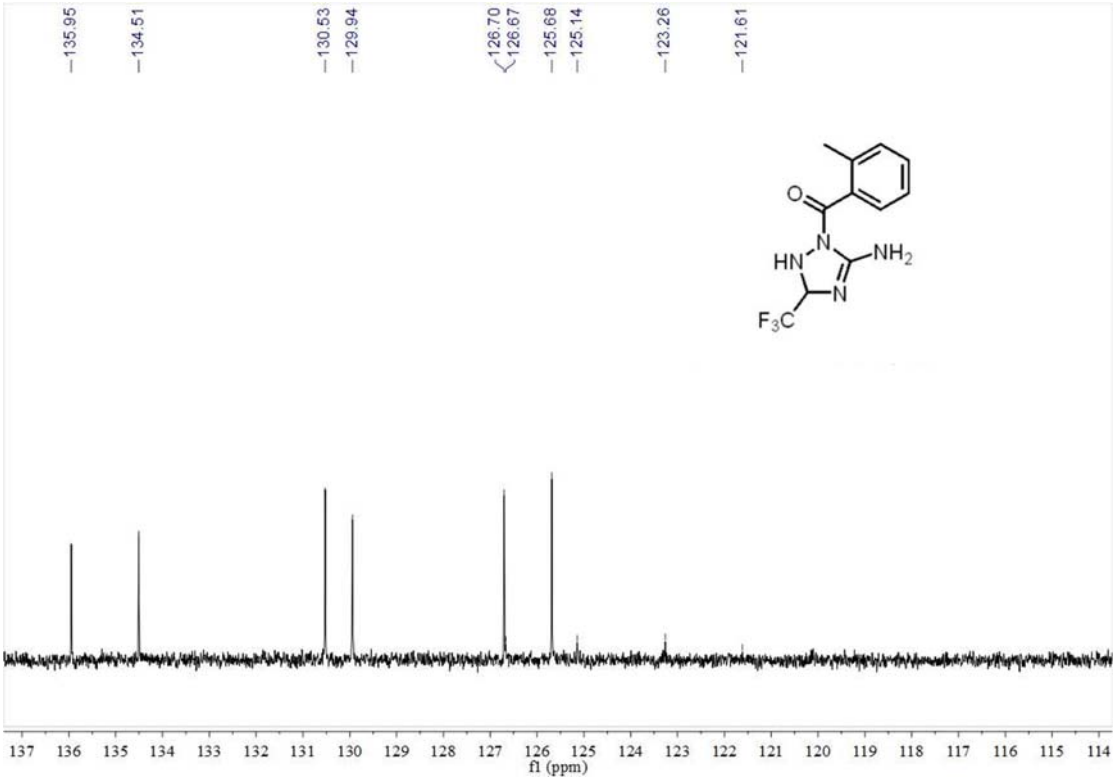
258.06772	3263975.3	26.16			
258.12793	1340575.1	10.74			
259.08017	12477085.0	100.00	259.08012	0.05	$C_{10}H_{10}ON_4F_3$
259.13092	677639.9	5.43			

HRMS (ESI) copy of compound **2a**

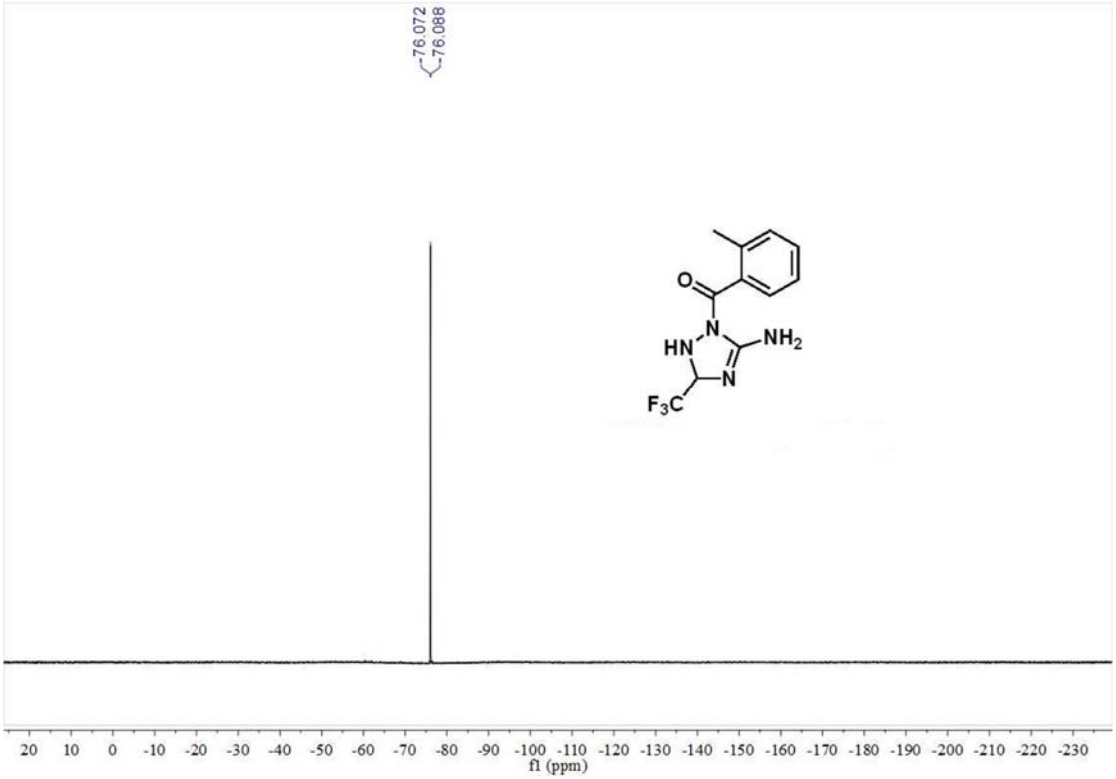


[1]H NMR (400 MHz) spectrum of **2b** in DMSO-d_6





$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **2b** in DMSO-d_6



^{19}F NMR (376 MHz) spectrum of **2b** in DMSO-*d*$_6$

Mass Spectrum SmartFormula Report

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Sample Name	1b	Instrument / Ser#	micrOTOF-Q 20453
Comment			

Acquisition Parameter

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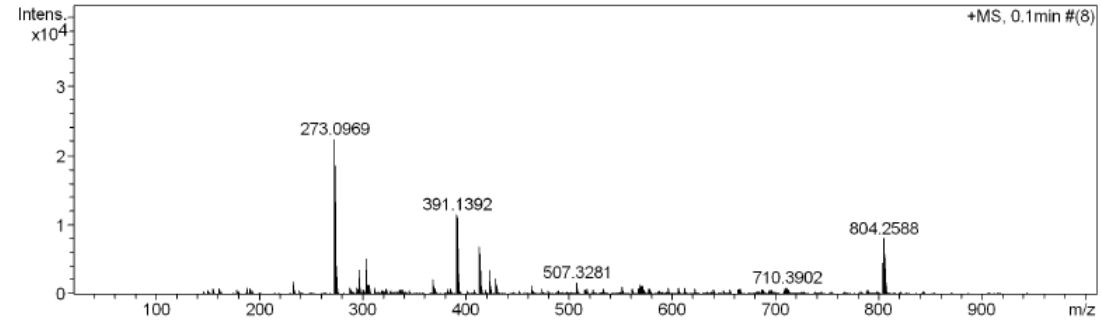
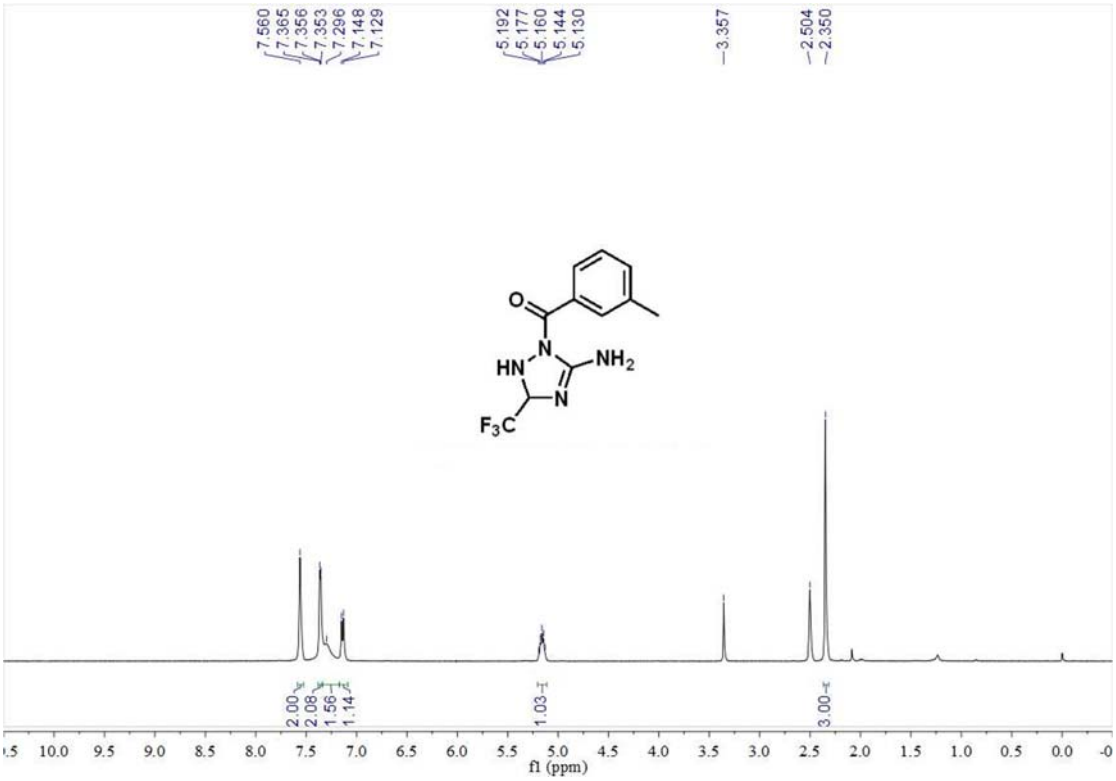
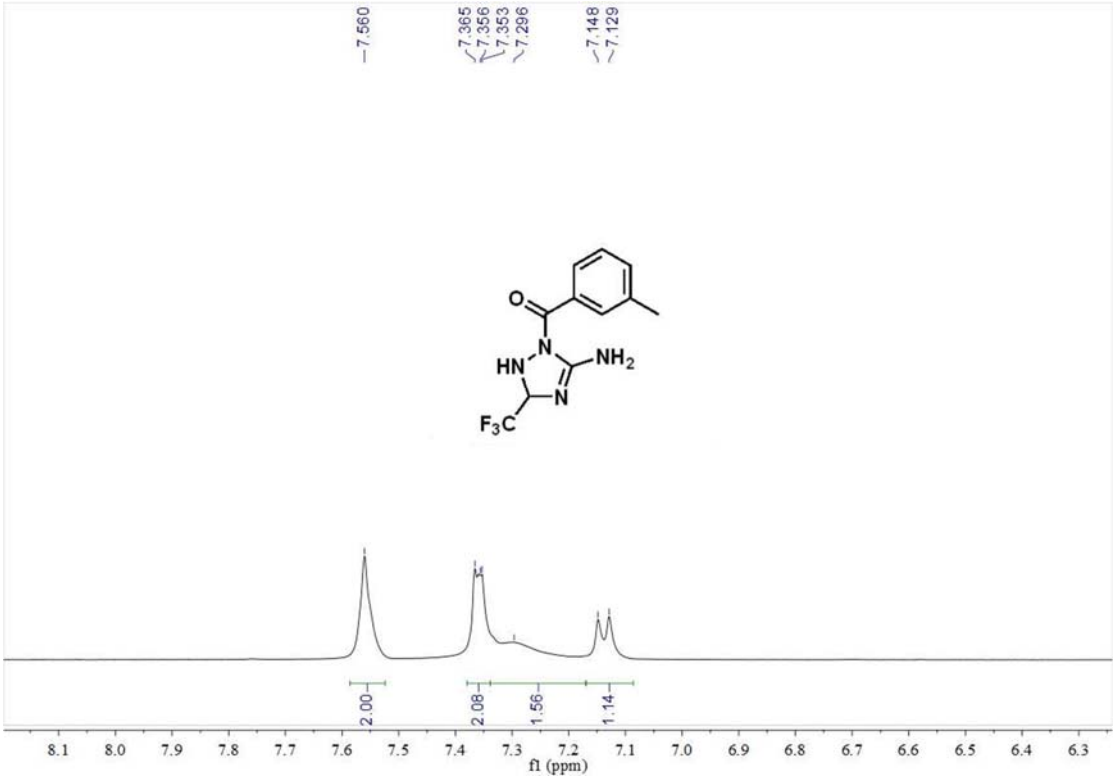


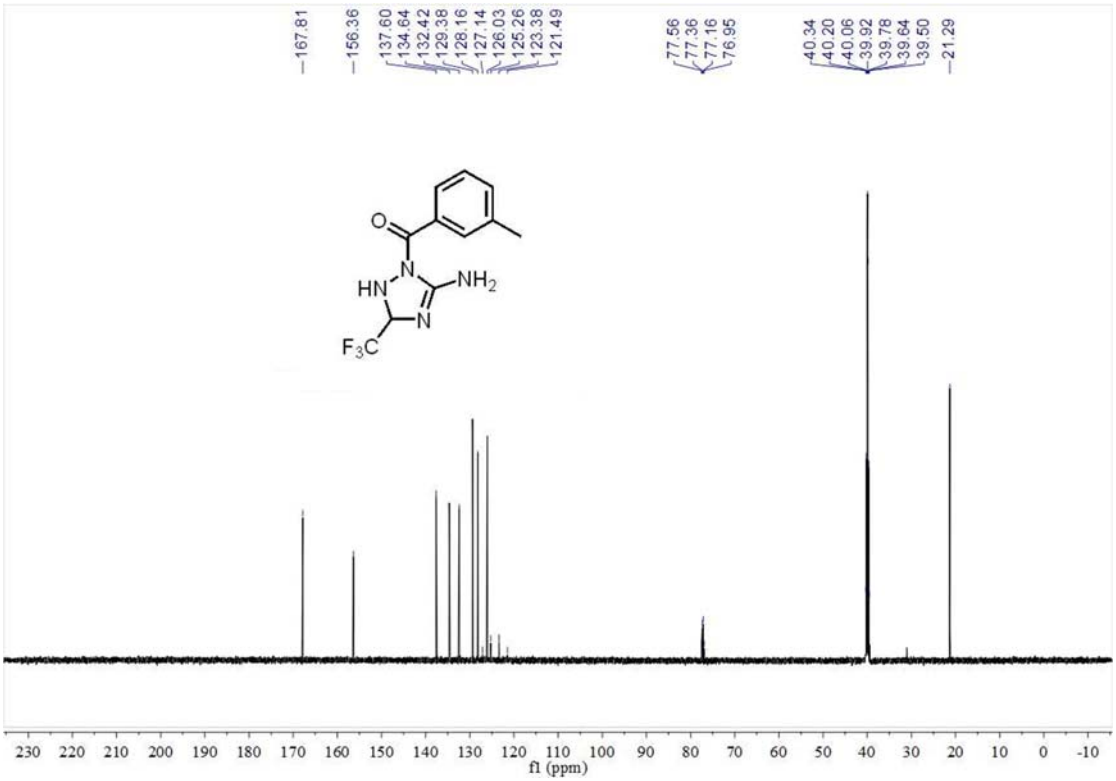
Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma	Std I	Std Mean m/z	Std I VarNorm	Std m/z Diff	Std Comb Dev
273.0969	1	C 11 H 12 F 3 N 4 O	273.0958	-4.1	-9.3	6.5	ok	even	417.3	488.3	2.9	199.5	2.7	842.7

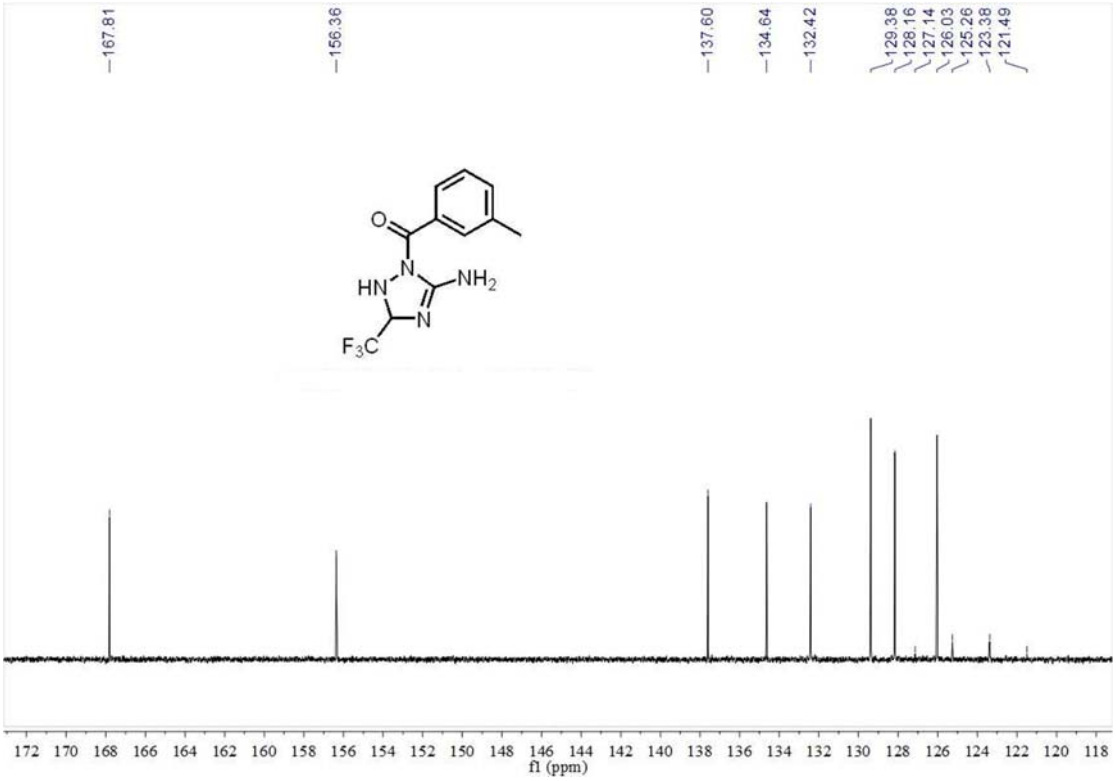
HRMS (ESI) copy of compound **2b**



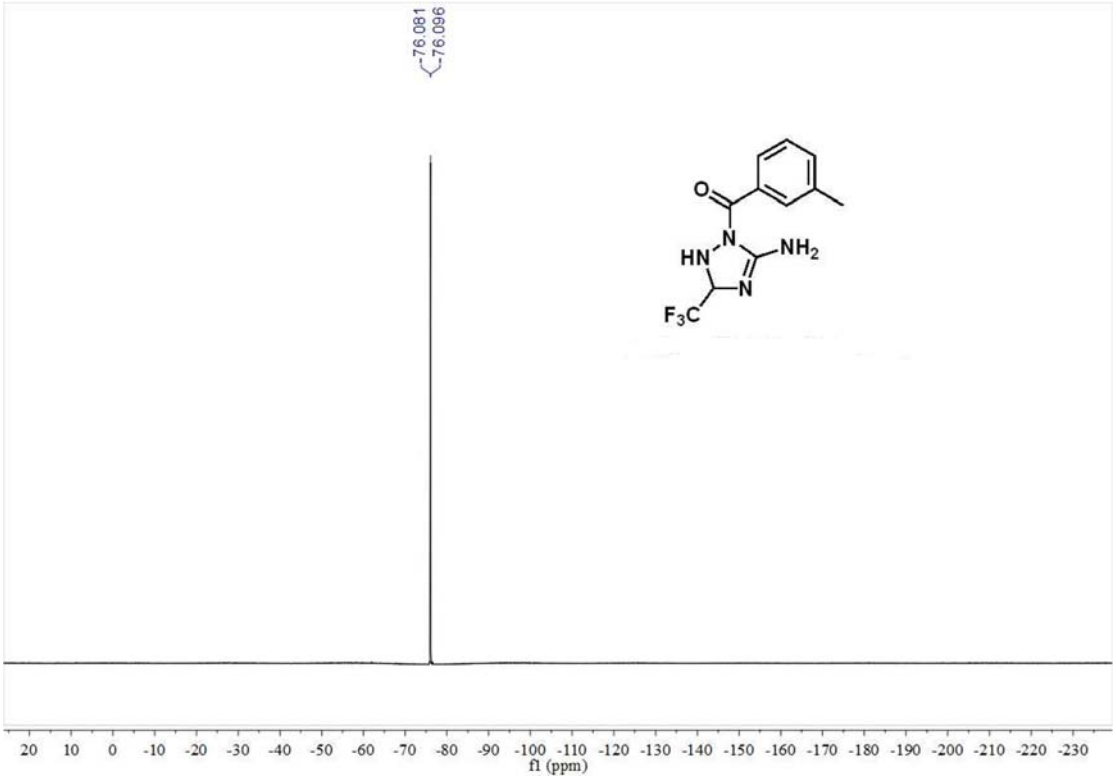


^{1}H NMR (400 MHz) spectrum of **2c** in DMSO-*d*$_6$





$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **2c** in DMSO-d_6



^{19}F NMR (376 MHz) spectrum of **2c** in DMSO-d_6

Mass Spectrum SmartFormula Report

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Sample Name	1c	Instrument / Ser#	micrOTOF-Q 20453
Comment			

Acquisition Parameter

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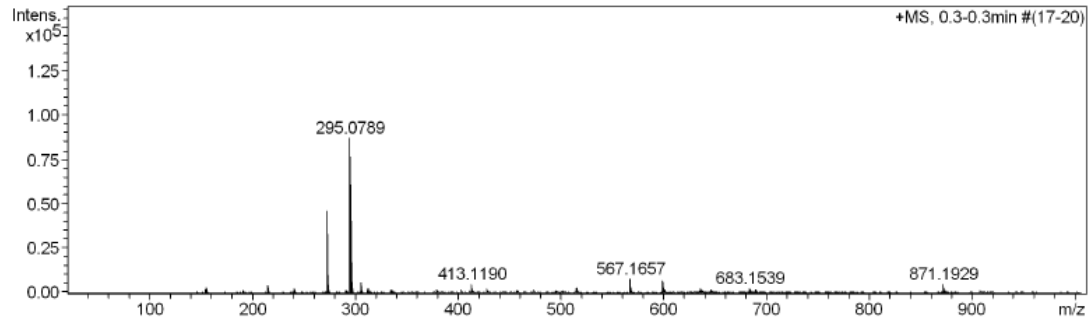

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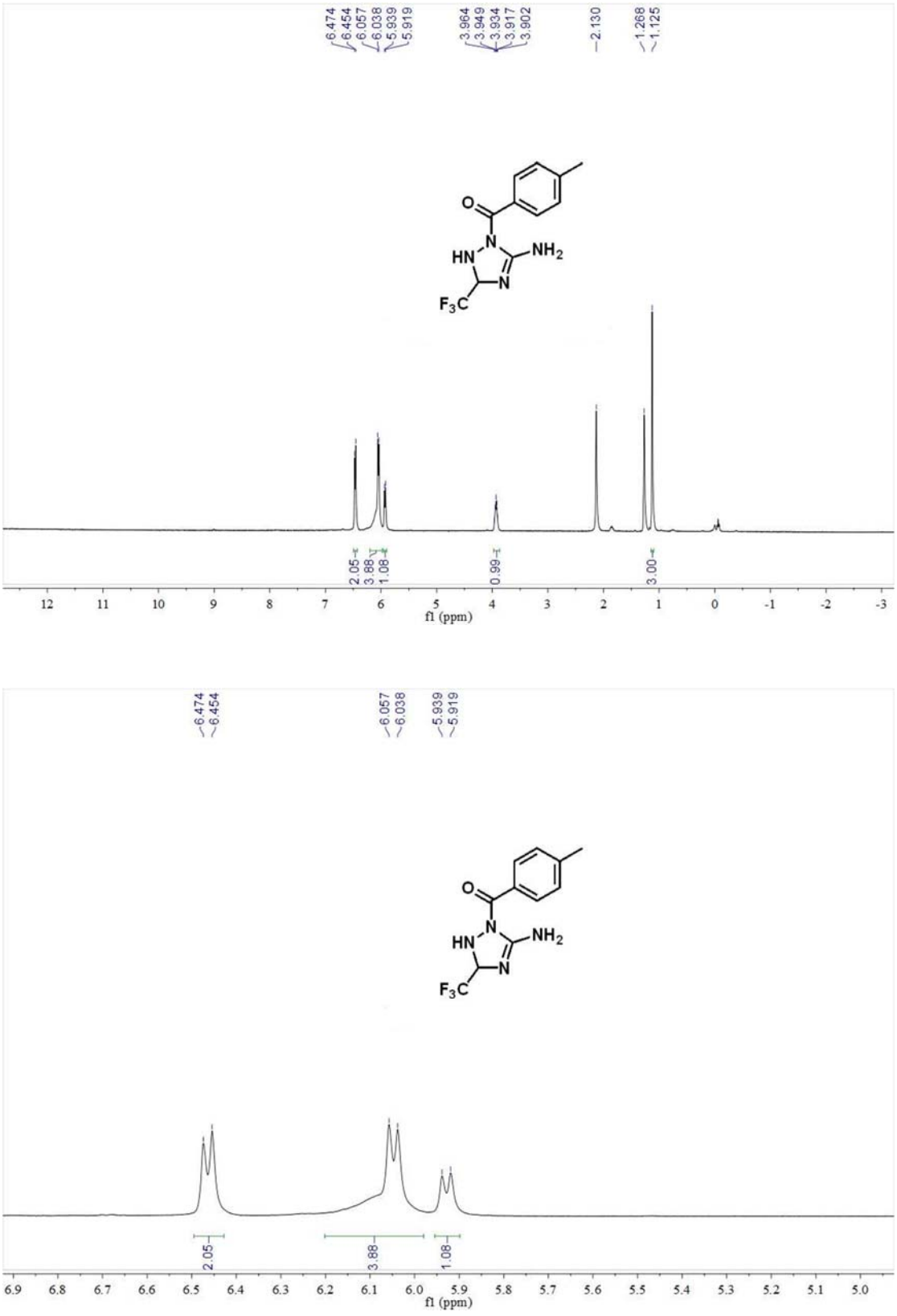
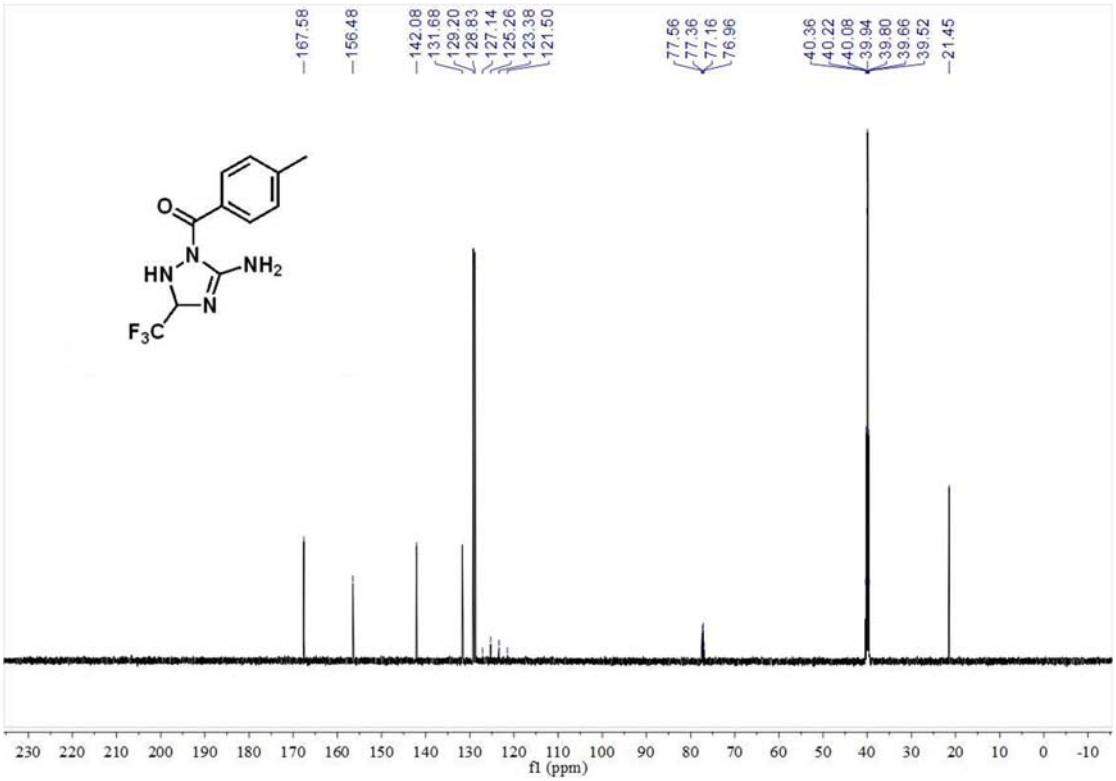
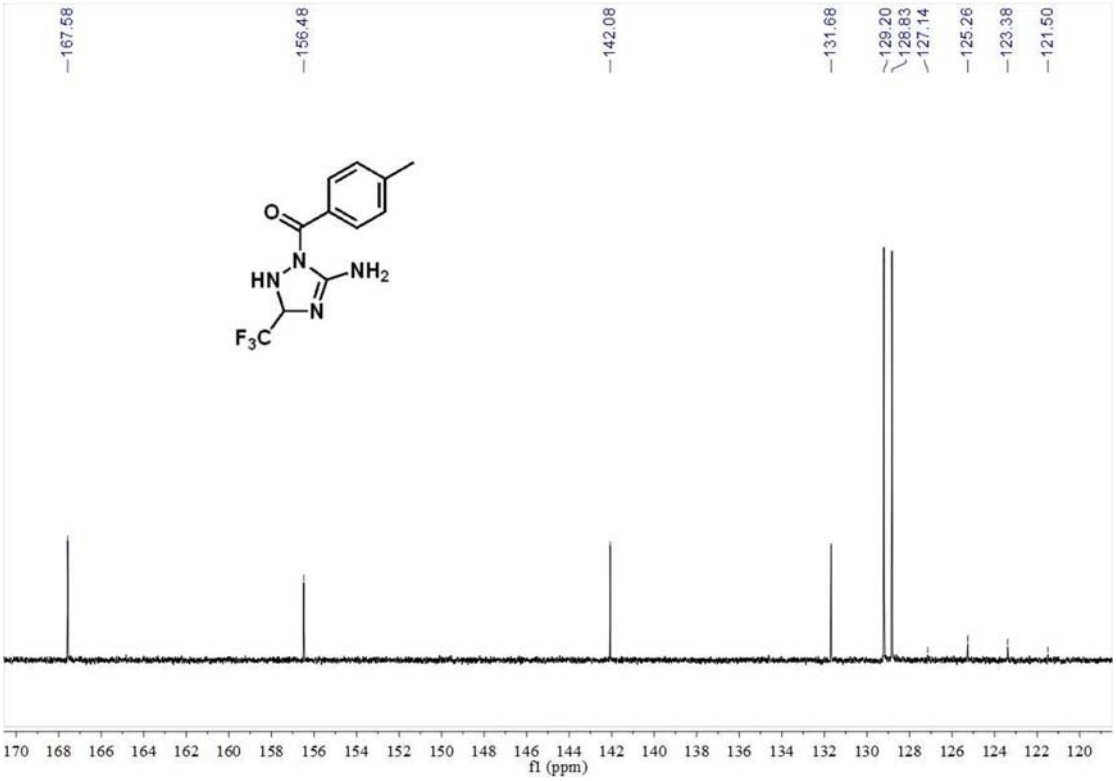
273.0967	1	C 11 H 12 F 3 N 4 O	273.0958	-3.4	-3.4	6.5	ok	even	5.9	13.3	1.0	6.2	0.4	842.7
295.0789	1	C 11 H 11 F 3 N 4 Na O	295.0777	-3.9	-3.1	6.5	ok	even	4.7	7.1	1.1	4.7	2.1	842.7

HRMS (ESI) copy of compound **2c**

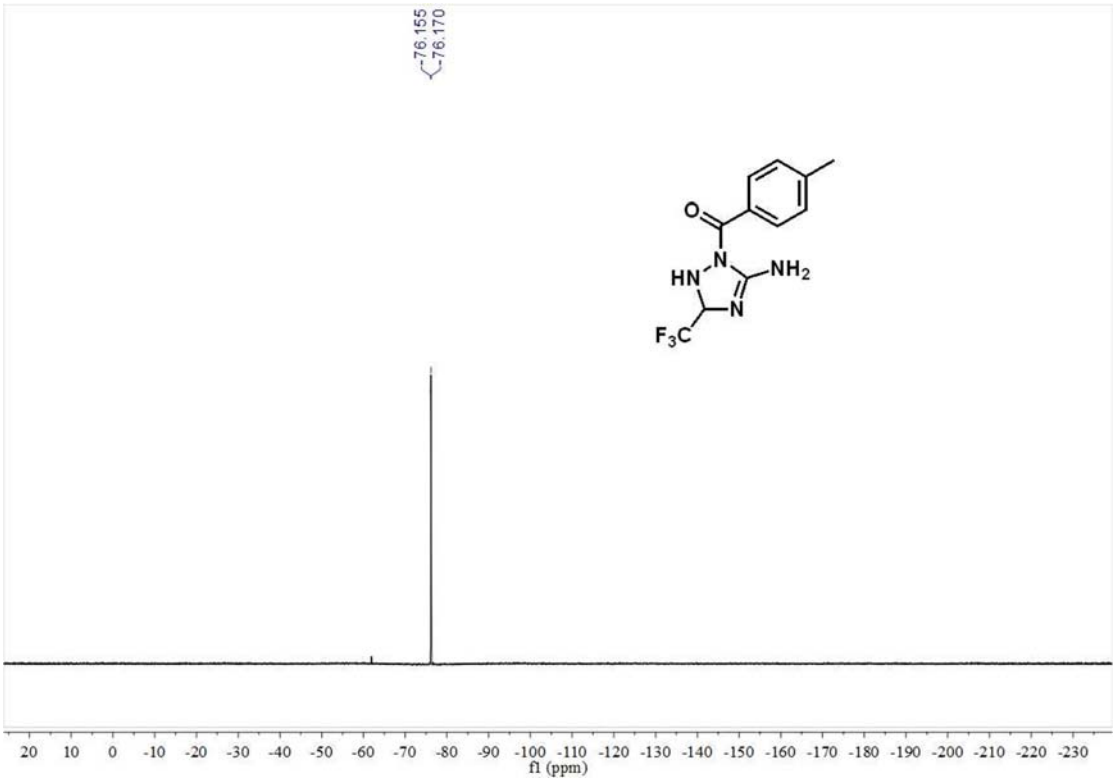


[1]H NMR (400 MHz) spectrum of **2d** in DMSO-d_6





$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **2d** in DMSO-d_6



^{19}F NMR (376 MHz) spectrum of **2d** in DMSO-*d*$_6$

Mass Spectrum SmartFormula Report

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Sample Name	1d	Instrument / Ser#	micrOTOF-Q 20453
Comment			

Acquisition Parameter

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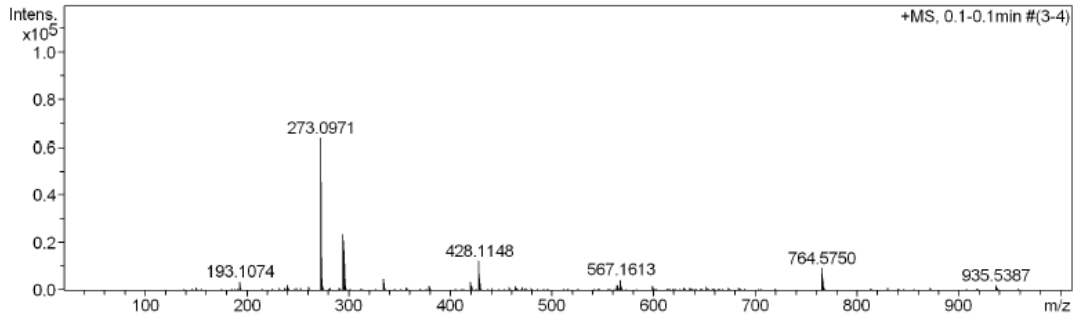

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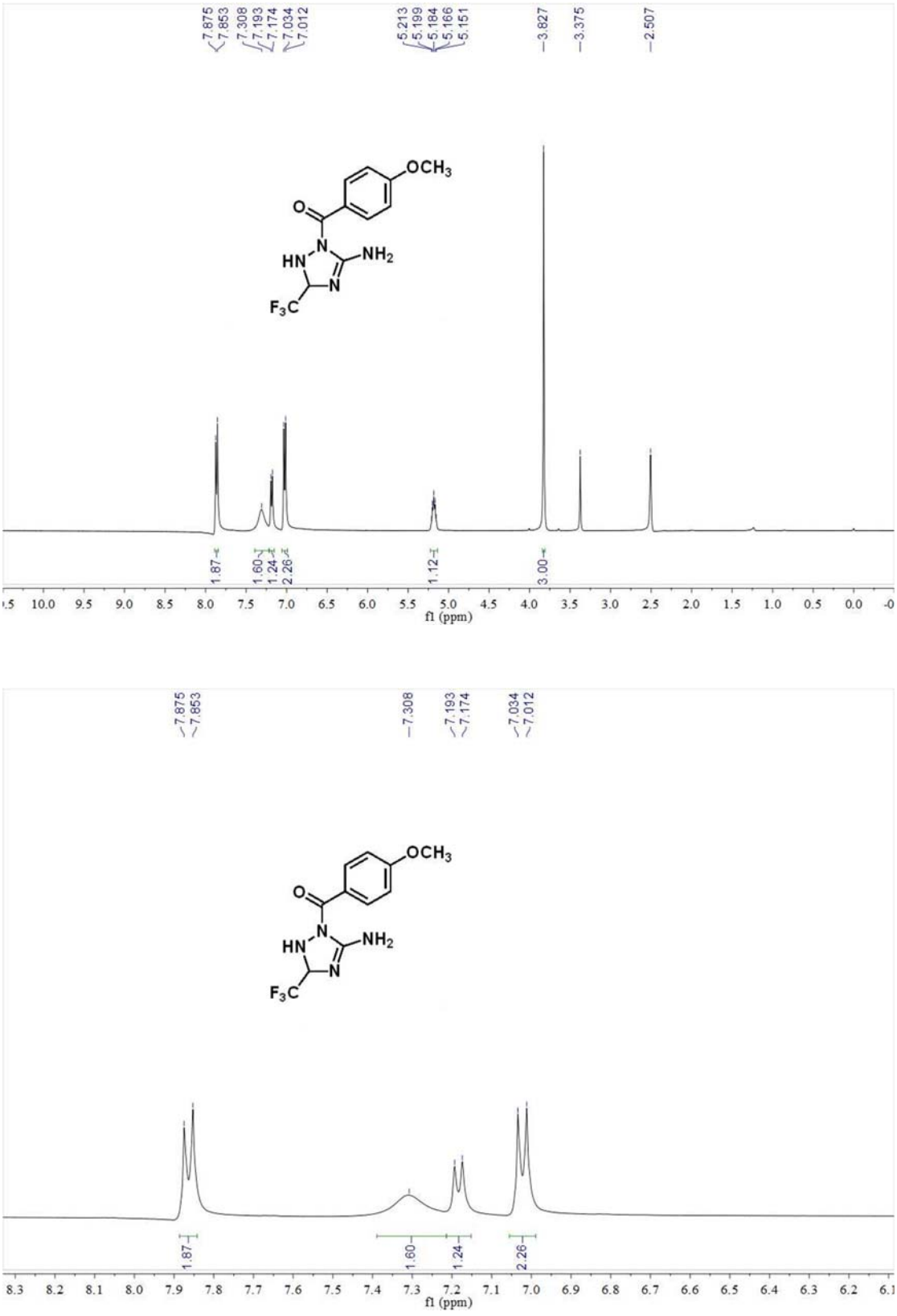
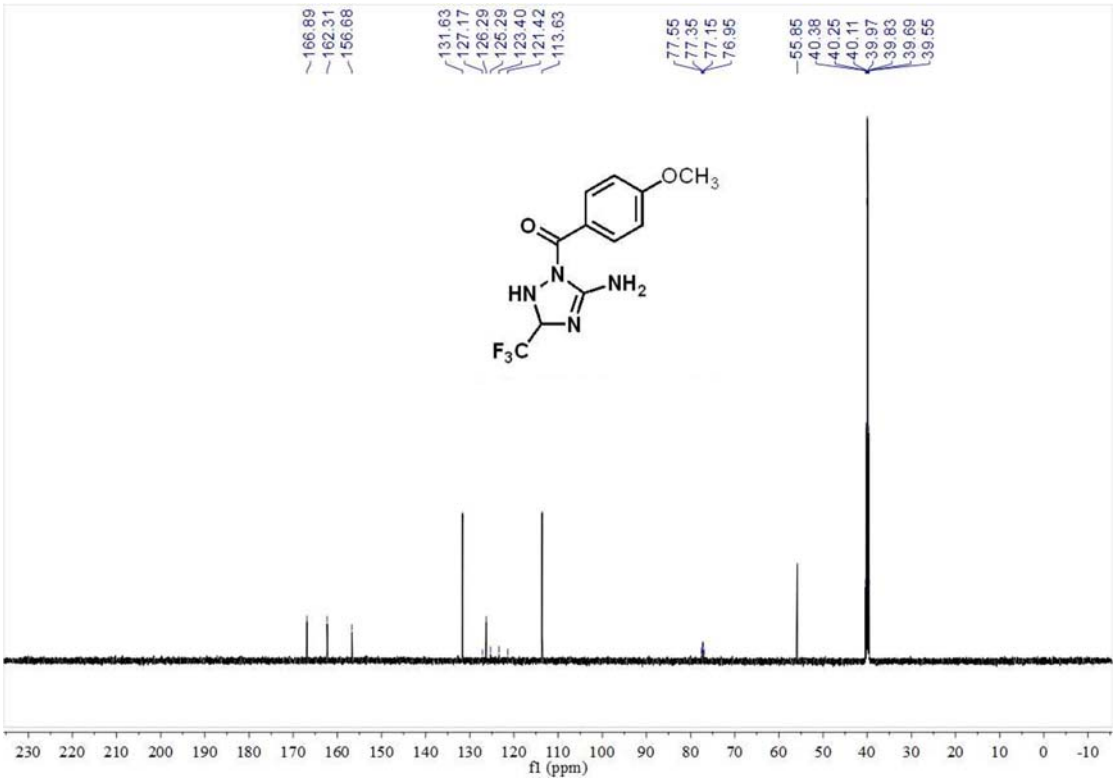
273.0971	1	C 11 H 12 F 3 N 4 O	273.0958	-4.9	-4.8	6.5	ok	even	11.6	21.7	1.3	10.3	0.4	842.7

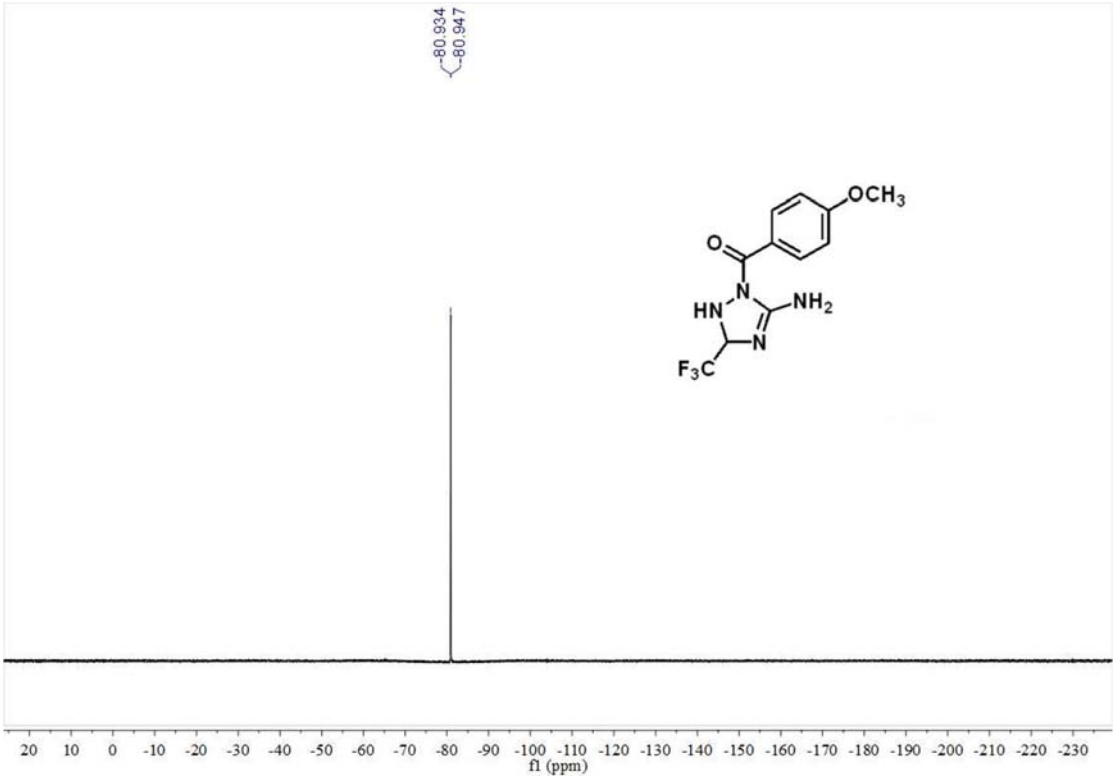
NMR copies of compound **2d**



^{1}H NMR (400 MHz) spectrum of **2e** in DMSO-d_6



$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **2e** in DMSO-*d*$_6$



^{19}F NMR (376 MHz) spectrum of **2e** in DMSO-*d*$_6$

Mass Spectrum SmartFormula Report

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Sample Name	1e	Instrument / Ser#	micrOTOF-Q 20453
Comment			

Acquisition Parameter

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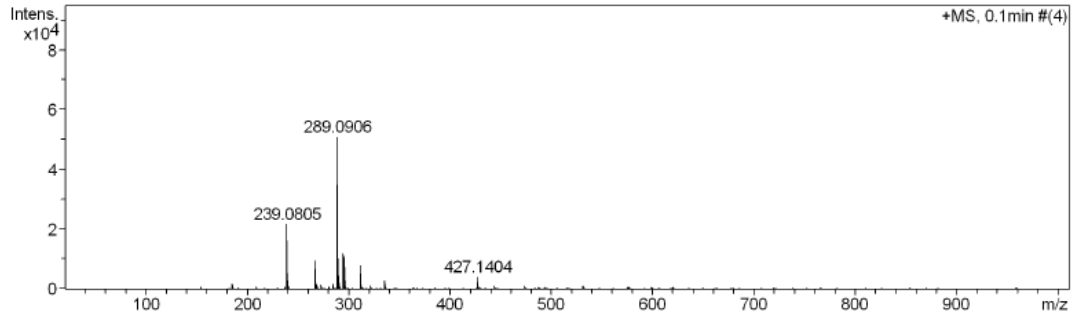
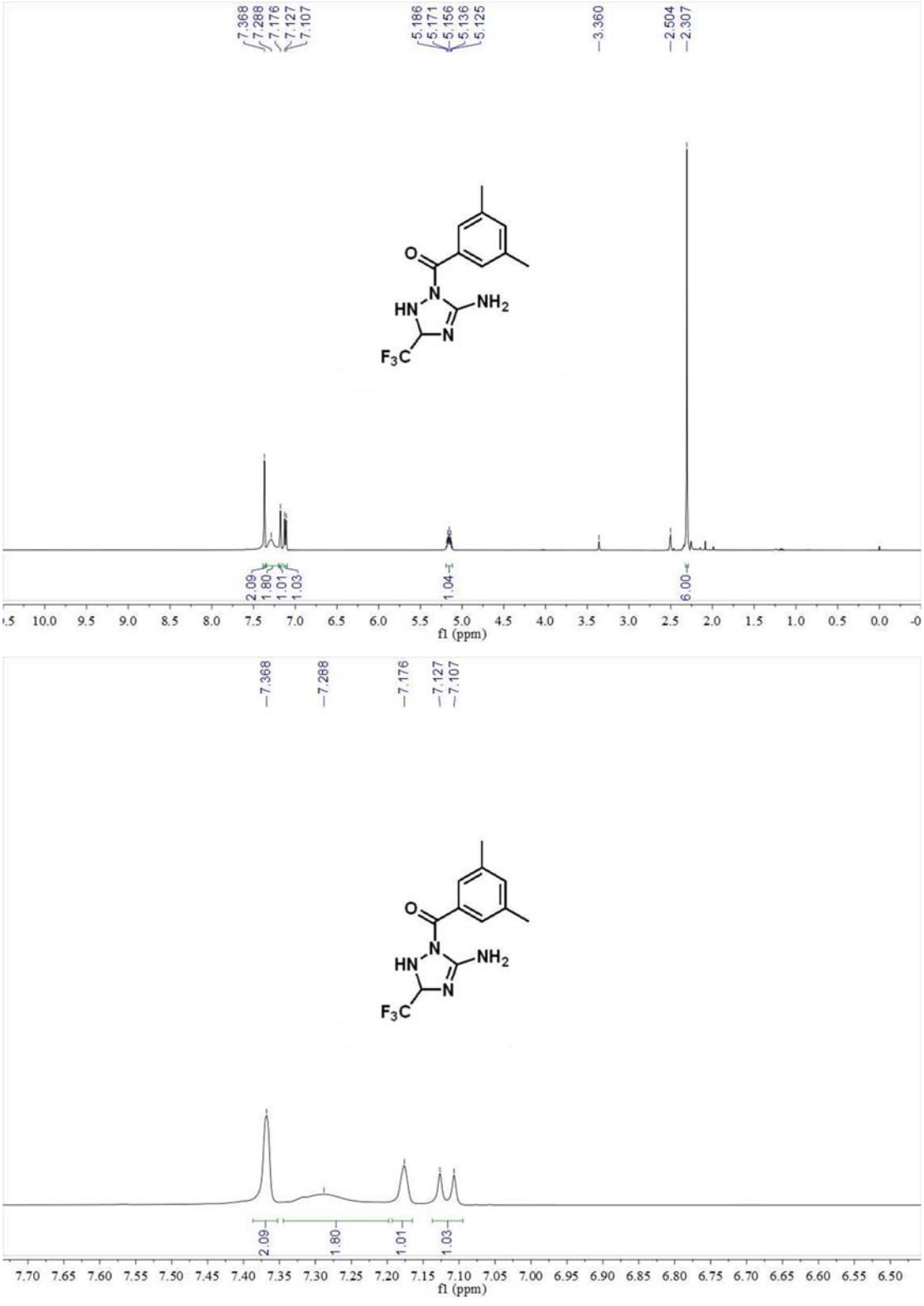
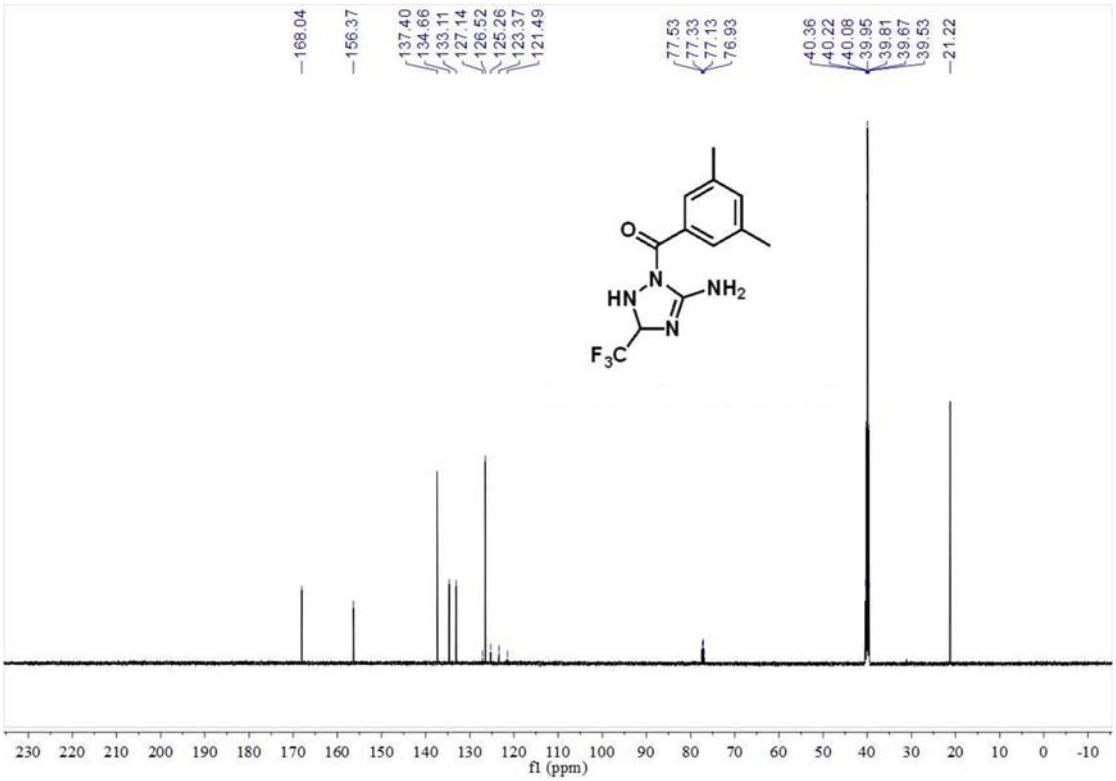


Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma	Std I	Std Mean m/z	Std I Var Norm	Std m/z Diff	Std Comb Dev
289.0906	1	C 11 H 12 F 3 N 4 O 2	289.0907	0.4	1.2	6.5	ok	even	10.9	19.3	0.8	11.0	2.2	842.7

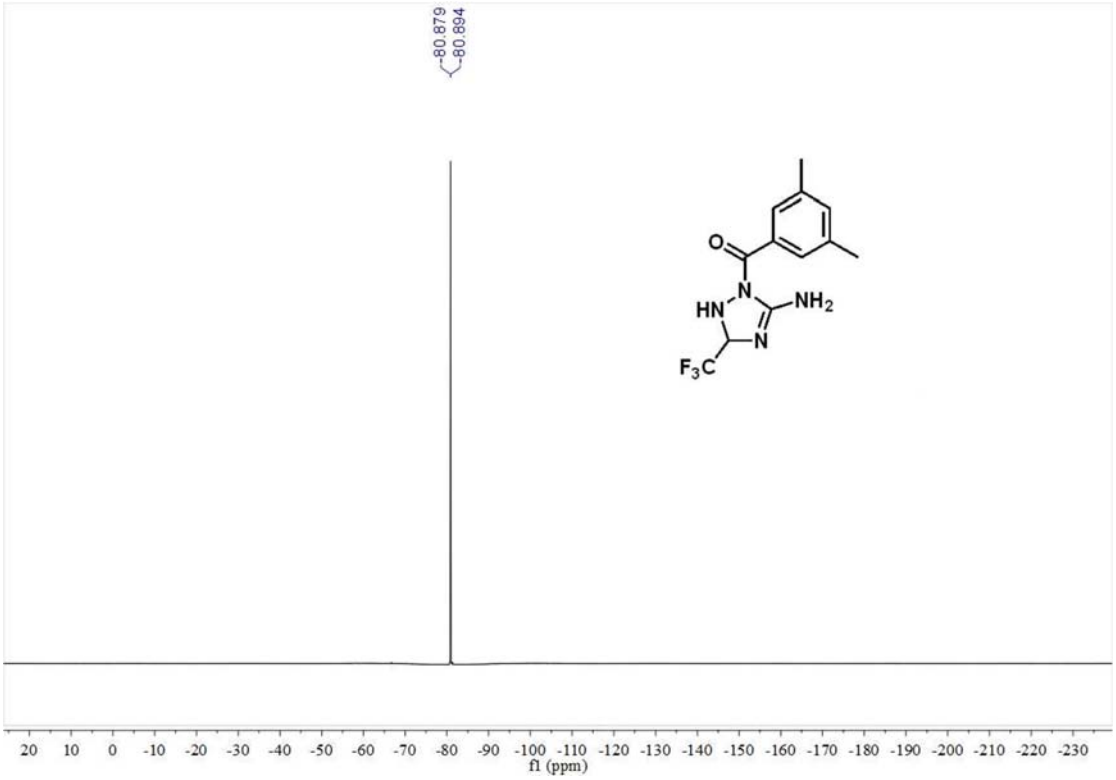
HRMS (ESI) copy of compound **2e**



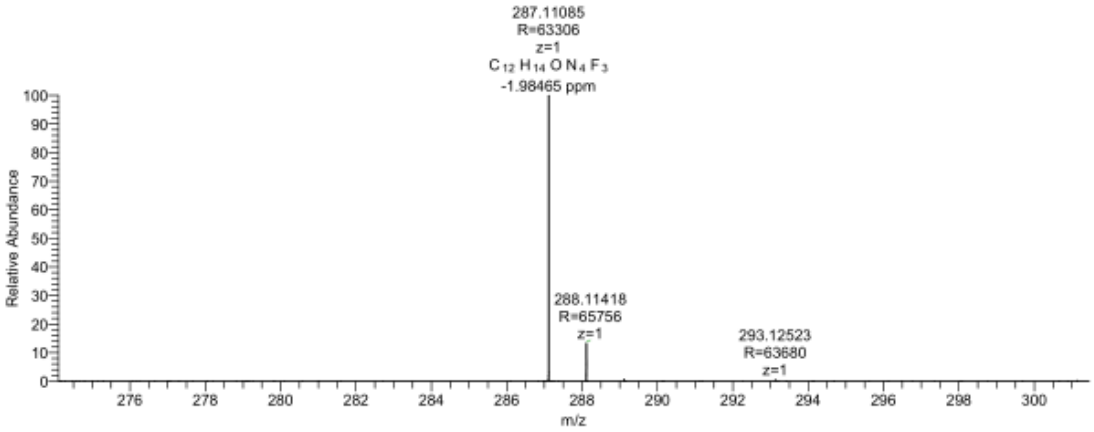
^{1}H NMR (400 MHz) spectrum of **2f** in DMSO-*d*$_6$



$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **2f** in DMSO-*d*$_6$



^{19}F NMR (376 MHz) spectrum of **2f** in DMSO-*d*$_6$



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288.11418	321198240.0	12.74	65755.63	1.00		
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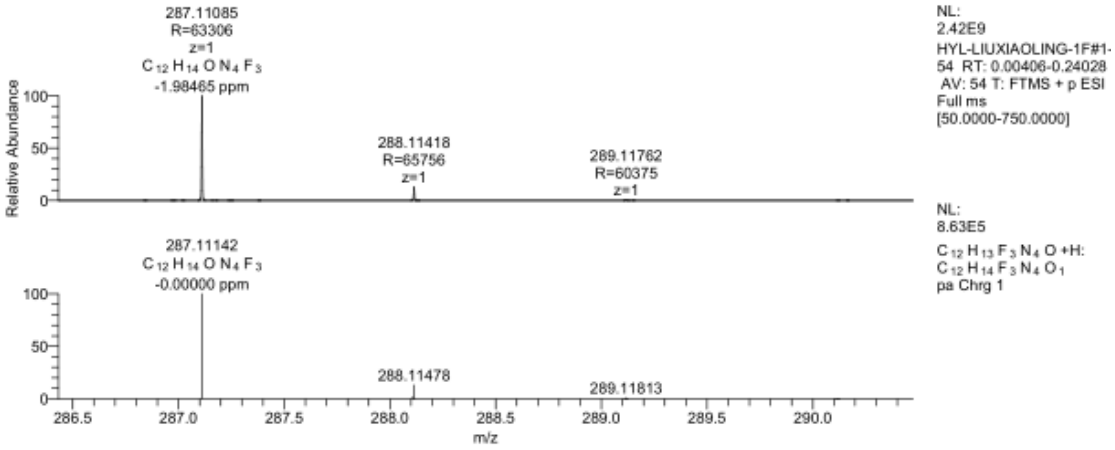
293.12523	13266798.0	0.53	63680.04	1.00		



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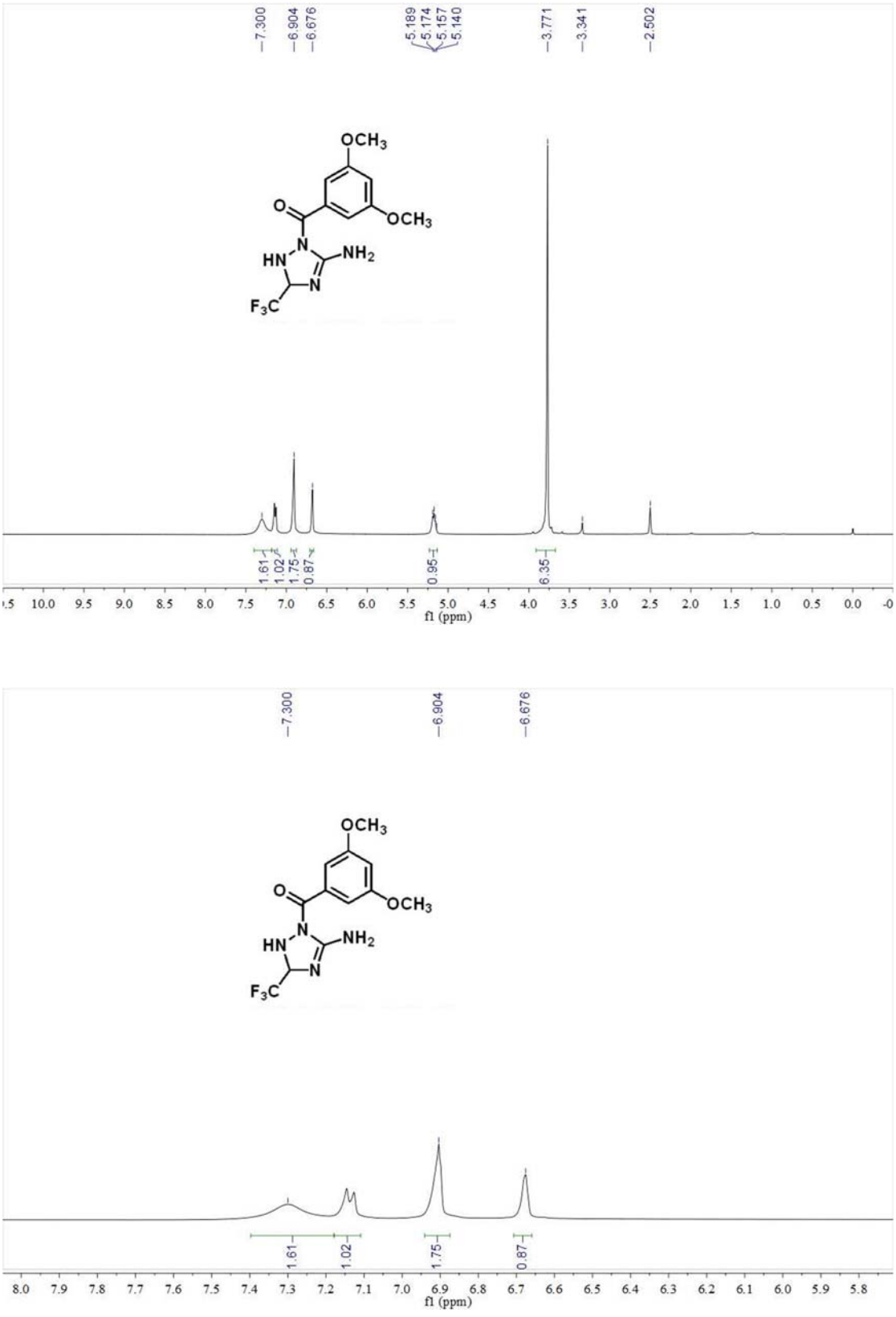
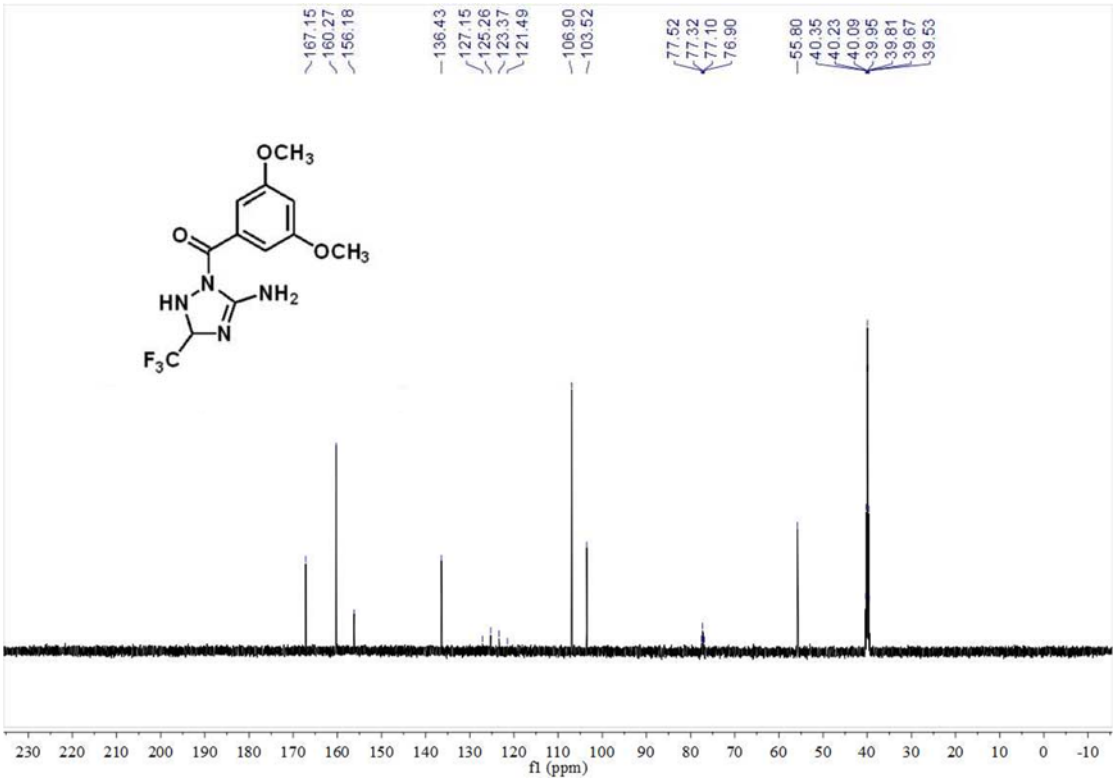
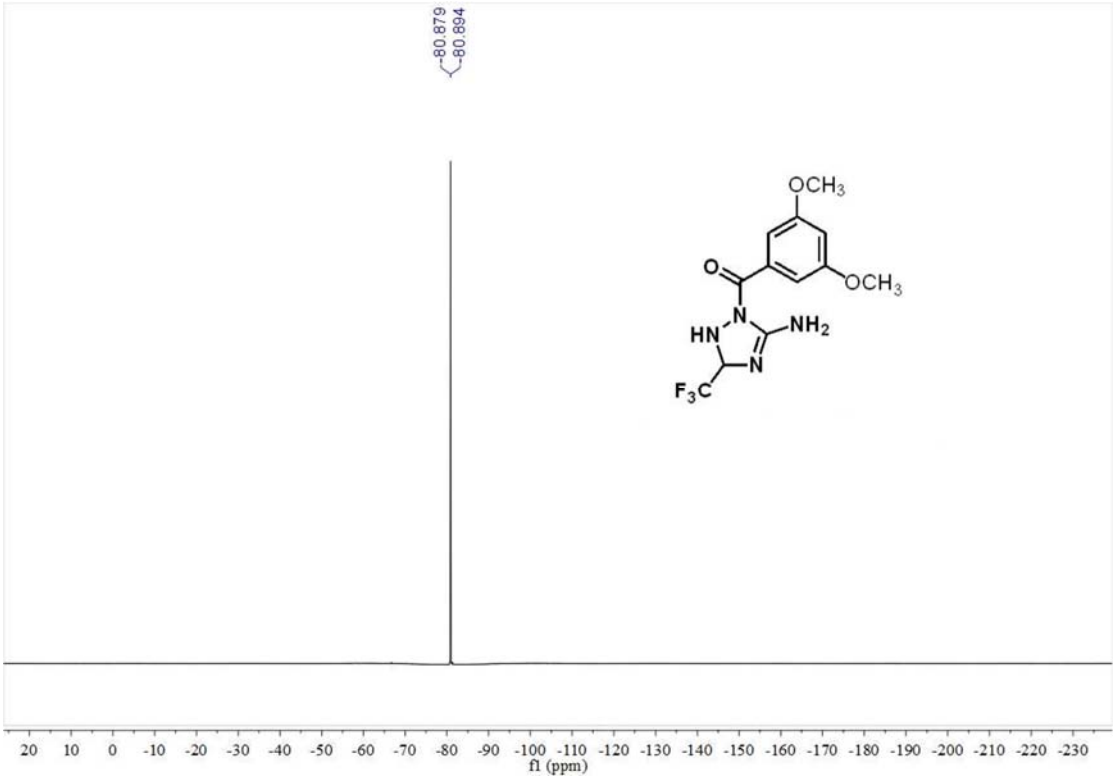


^{1}H NMR (400 MHz) spectrum of **2g** in DMSO-d_6



$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **2g** in DMSO-*d*$_6$



^{19}F NMR (376 MHz) spectrum of **2g** in DMSO-*d*$_6$

Mass Spectrum SmartFormula Report

Analysis Info

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Comment			

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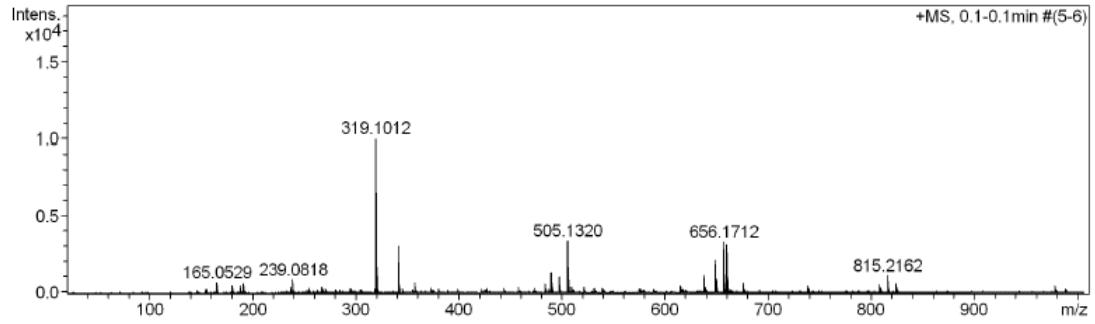
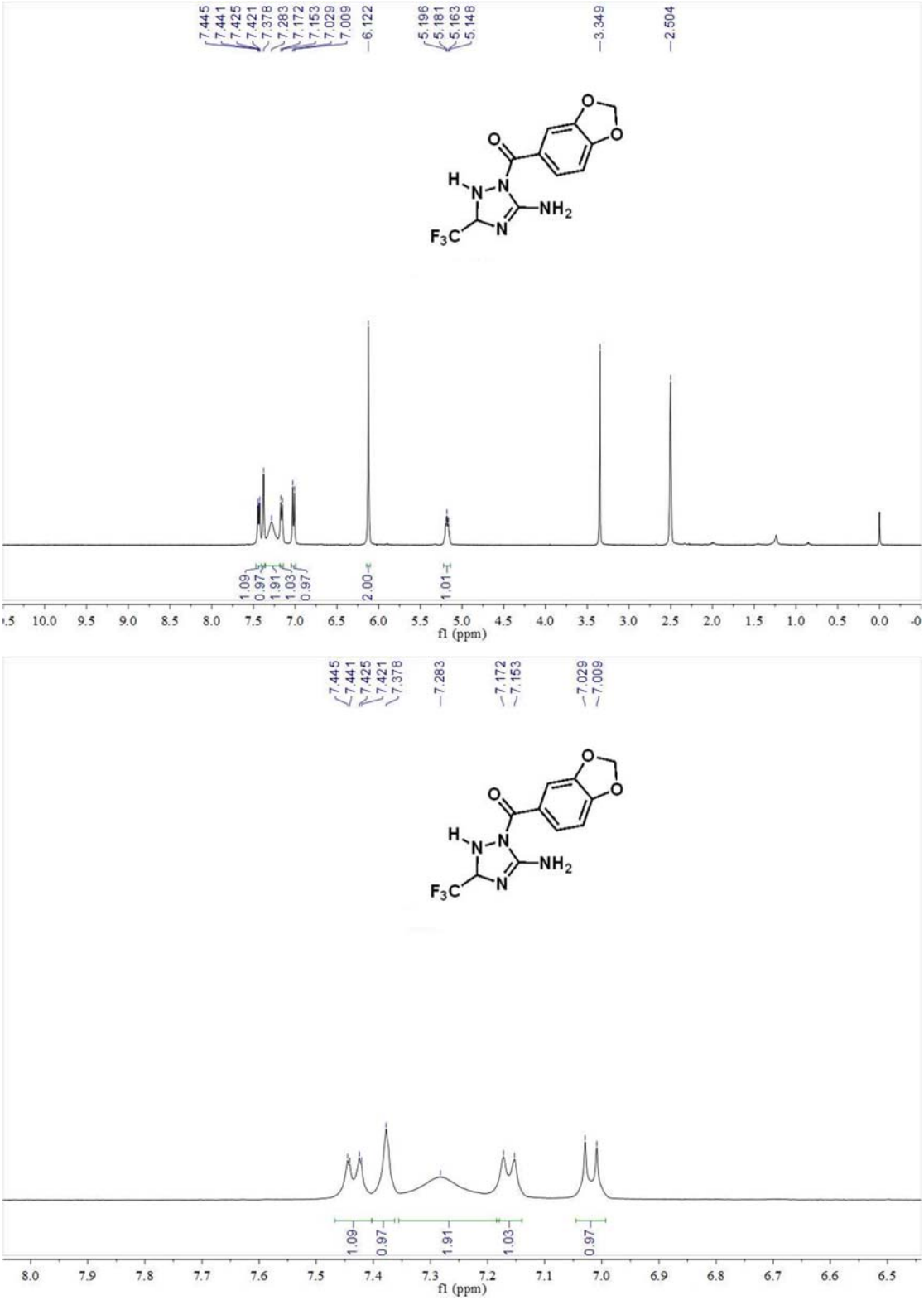
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste

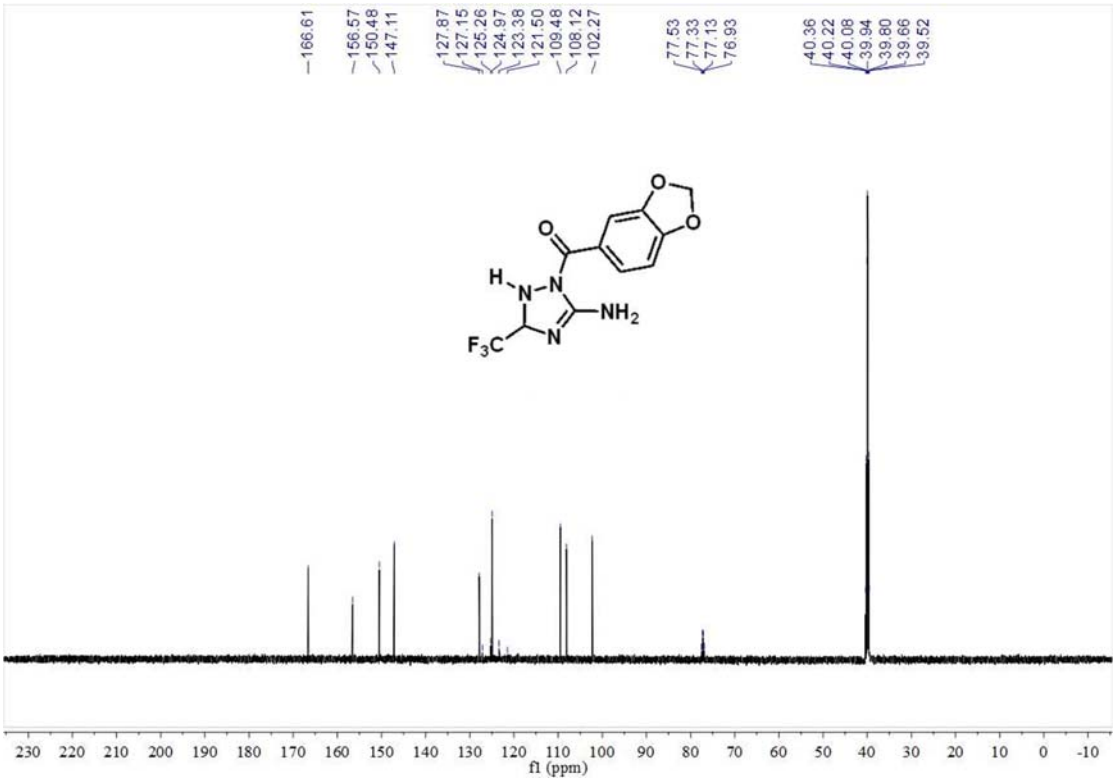


Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	e¡¥ Conf	mSigma	Std I	Std Mean m/z	Std I Var Norm	Std m/z Diff	Std Comb Dev
319.1012	1	C 12 H 14 F 3 N 4 O 3	319.1013	0.3	0.7	6.5	ok	even	12.5	24.3	0.5	9.9	1.1	842.7

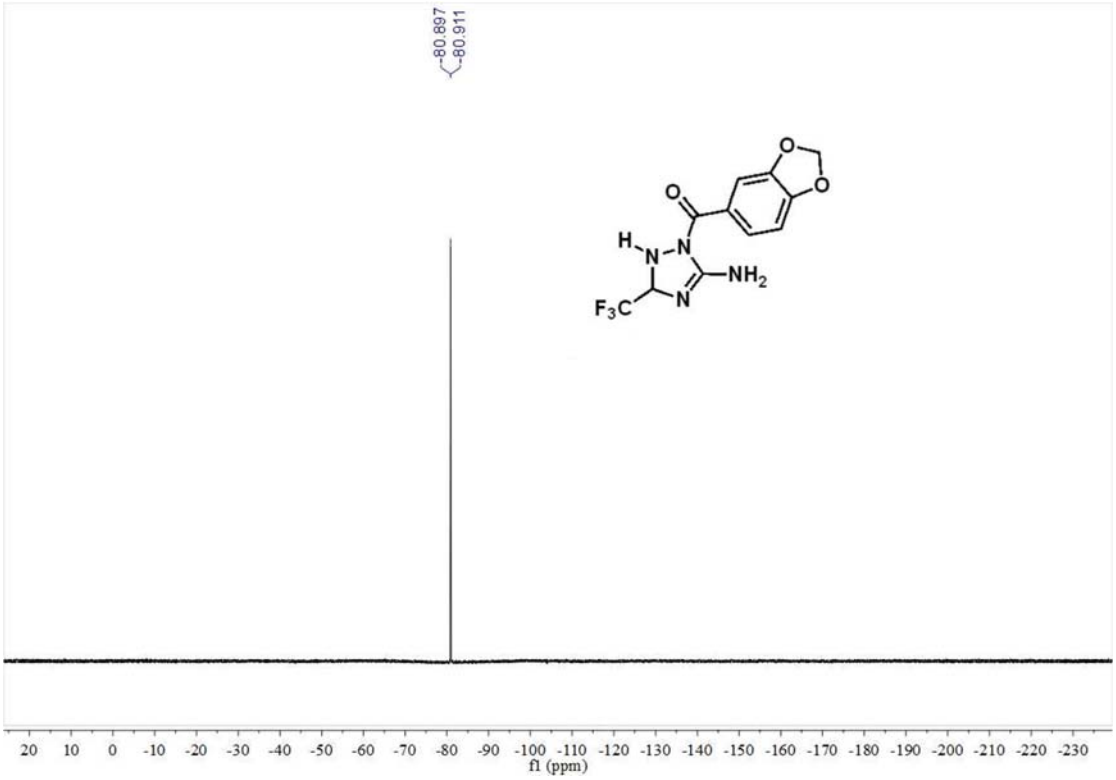
HRMS (ESI) copy of compound **2g**



^{1}H NMR (400 MHz) spectrum of **2h** in DMSO-*d*$_6$



$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **2h** in DMSO-*d*$_6$



^{19}F NMR (376 MHz) spectrum of **2h** in DMSO-*d*$_6$

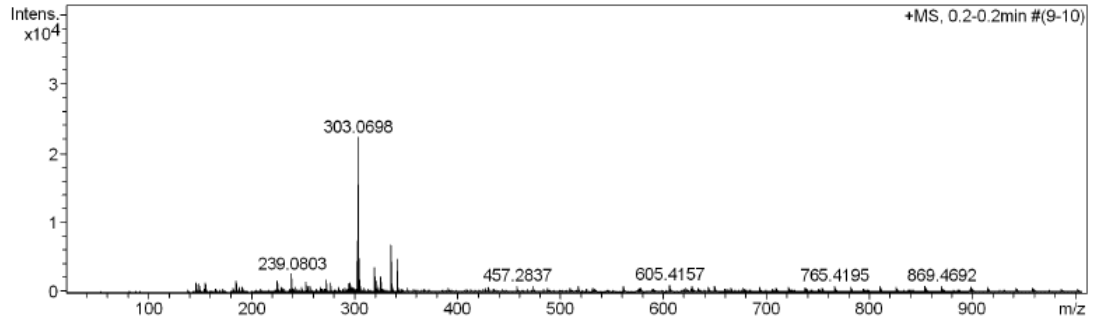
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\user\liuxiaoling20210623-7.d	Acquisition Date	2021-6-23 10:43:42
Method	tune_low.m	Operator	BDAL@DE
Sample Name	1h	Instrument / Ser#	micrOTOF-Q 20453
Comment			

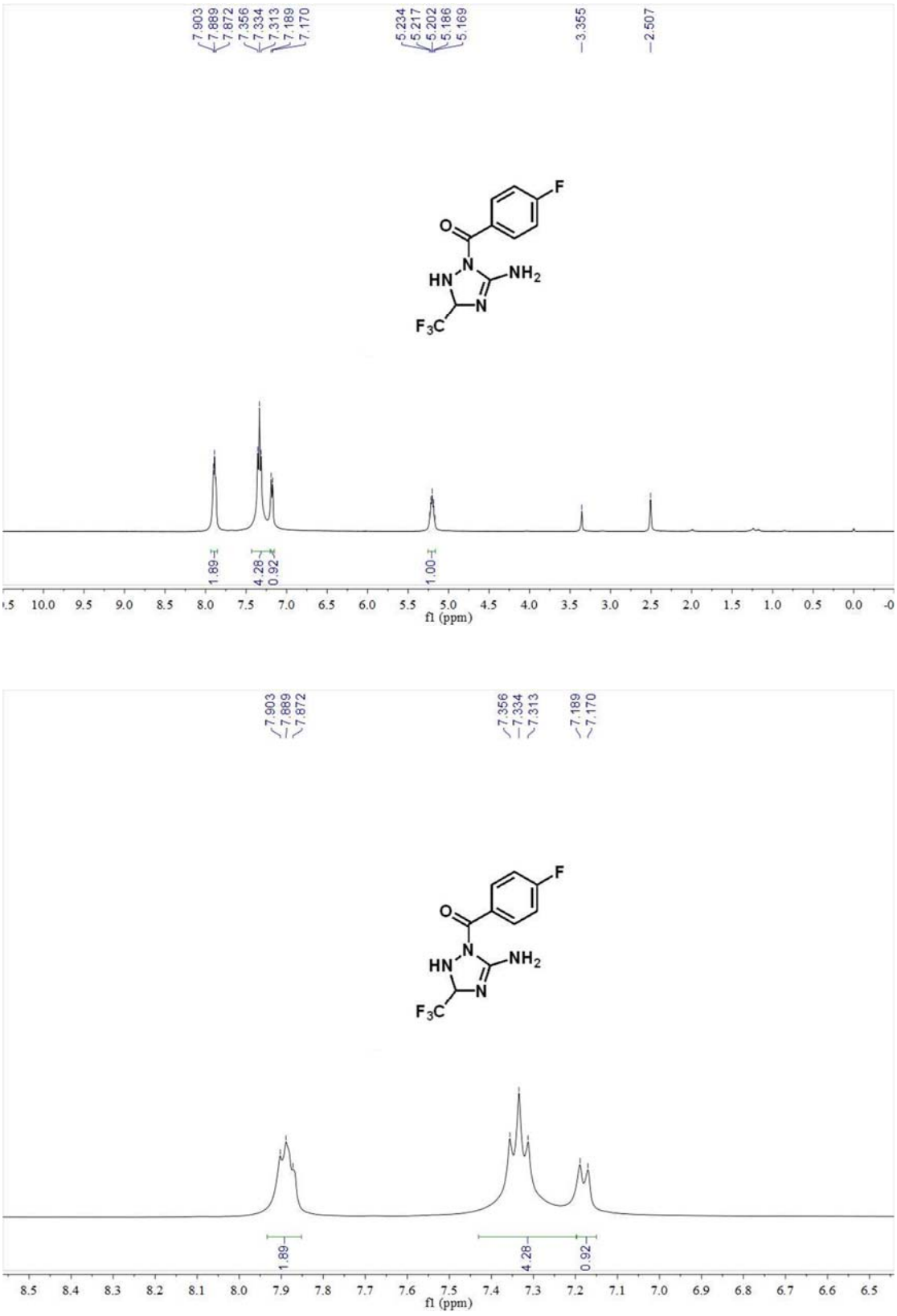
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 jãC
Scan Begin	21 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste

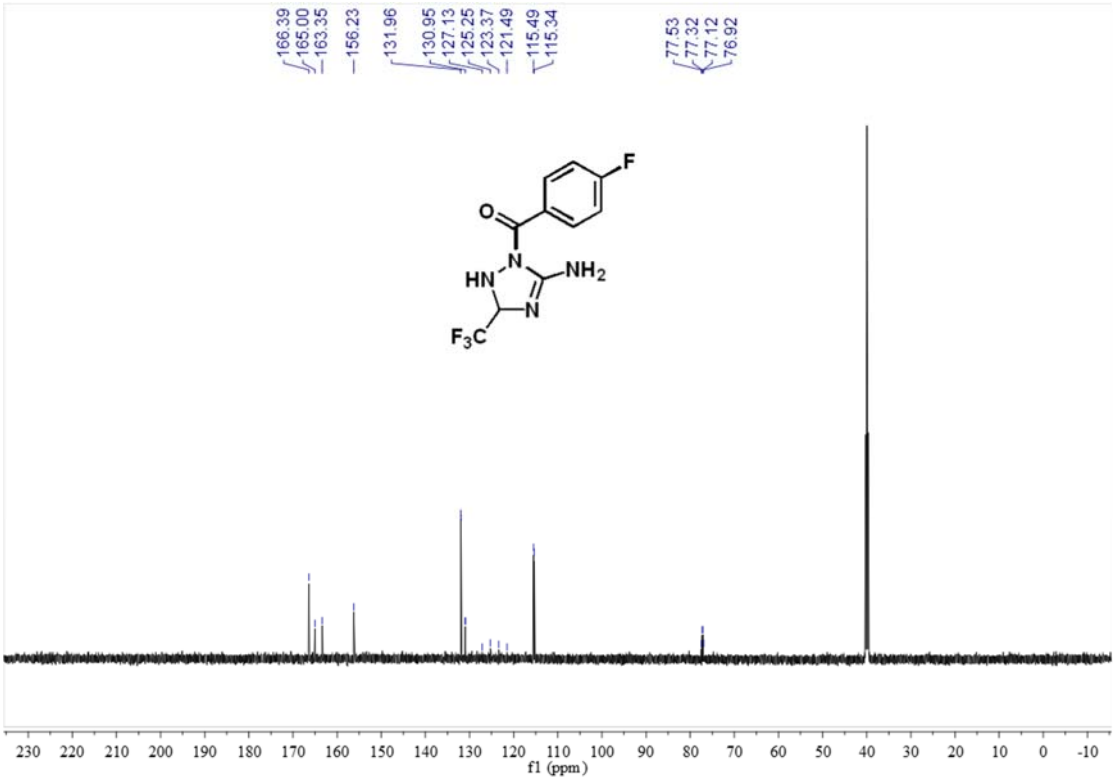


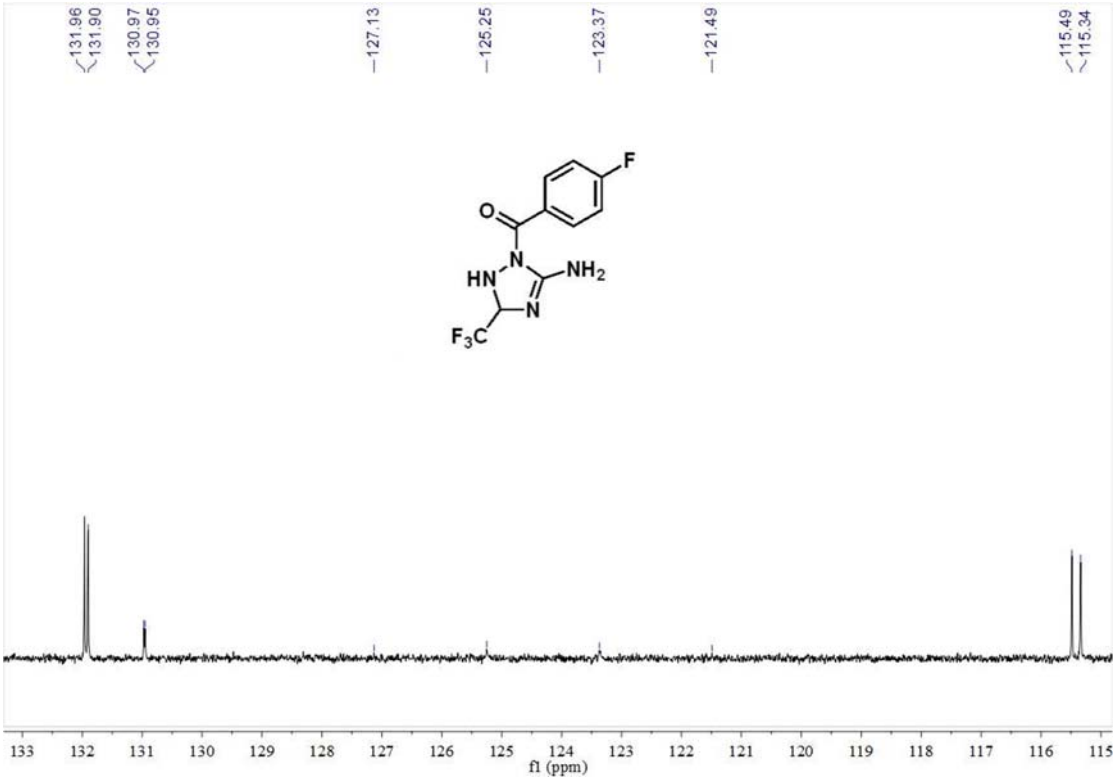
Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	e¡¥ Conf	mSigma	Std I	Std Mean m/z	Std I Var Norm	Std m/z Diff	Std Comb Dev
303.0698	1	C 11 H 10 F 3 N 4 O 3	303.0700	0.6	-0.7	7.5	ok	even	16.9	27.5	1.9	19.4	0.2	842.7

HRMS (ESI) copy of compound **2h**

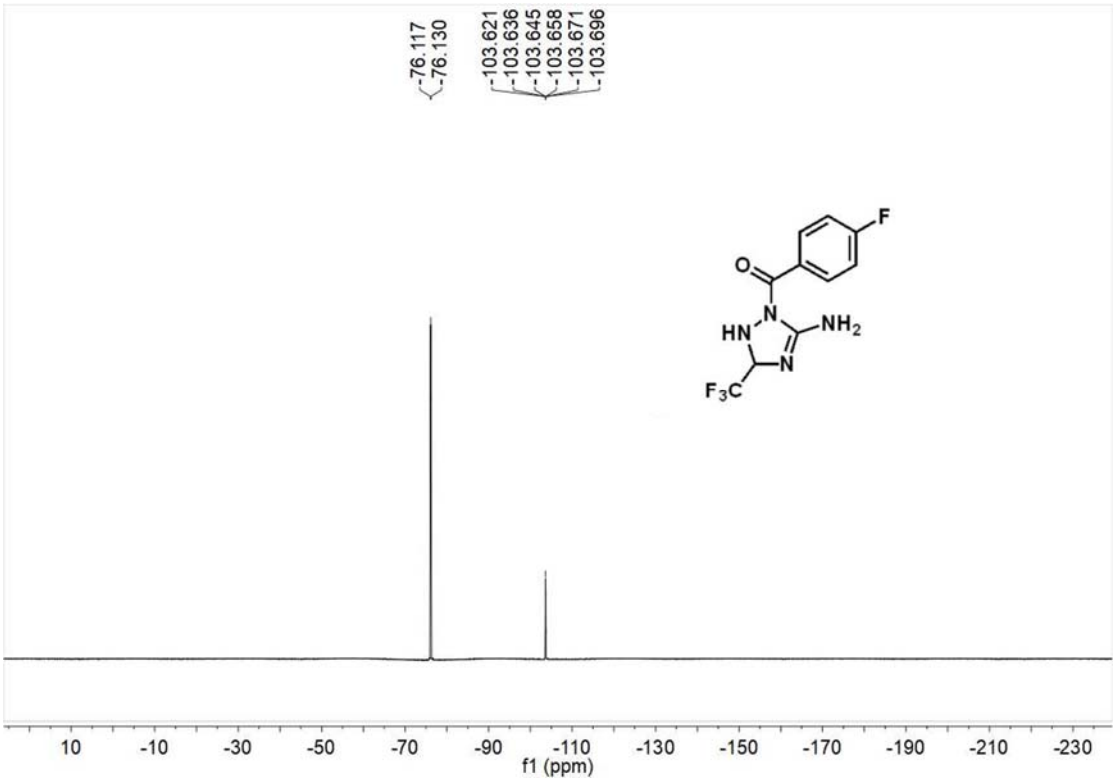


[1]H NMR (400 MHz) spectrum of **2i** in DMSO-*d*$_6$

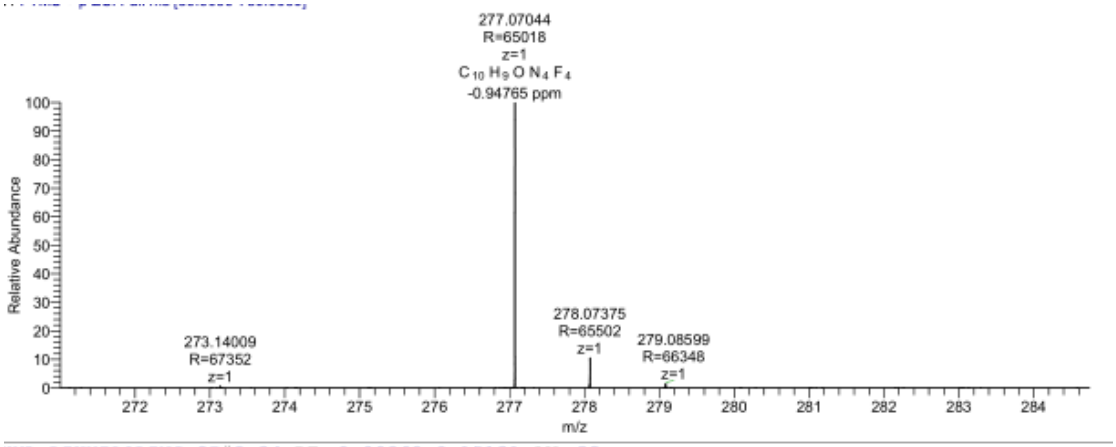




$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **2i** in DMSO-*d*$_6$

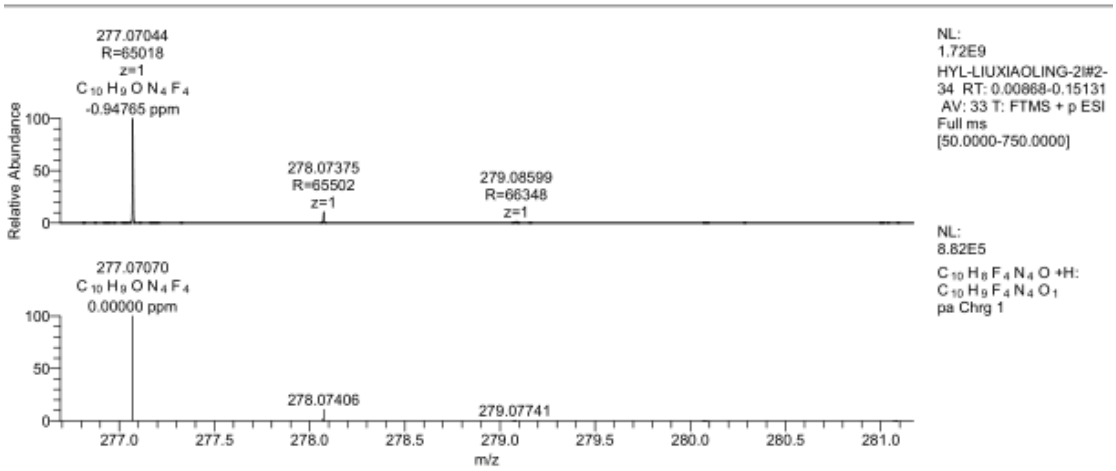


^{19}F NMR (376 MHz) spectrum of **2i** in DMSO-d_6

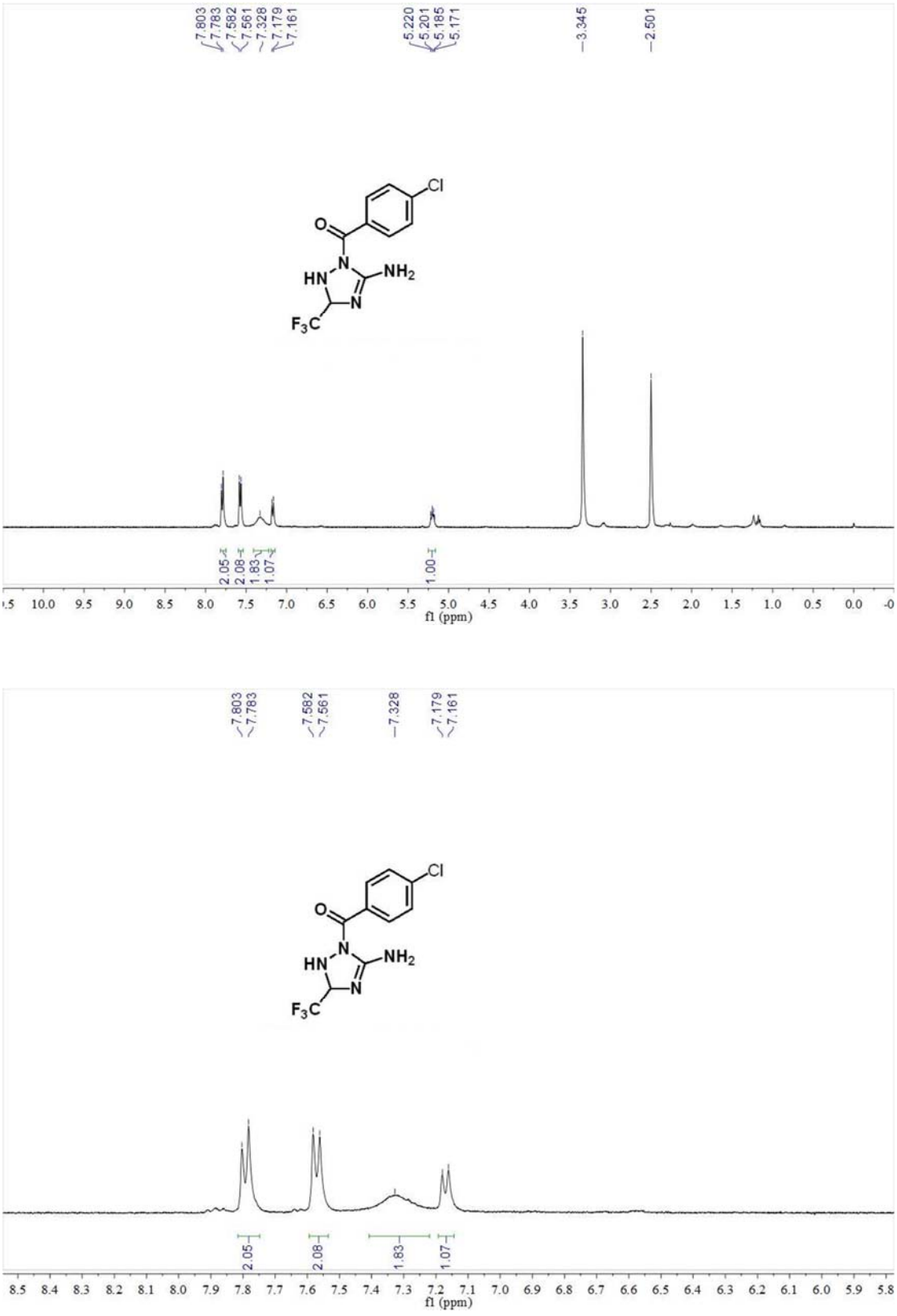


HYL-LIUXIAOLING-2I#2-34 RT: 0.00868-0.15131 AV: 33
T: FTMS + p ESI Full ms [50.0000-750.0000]
m/z= 271.00250-284.73960

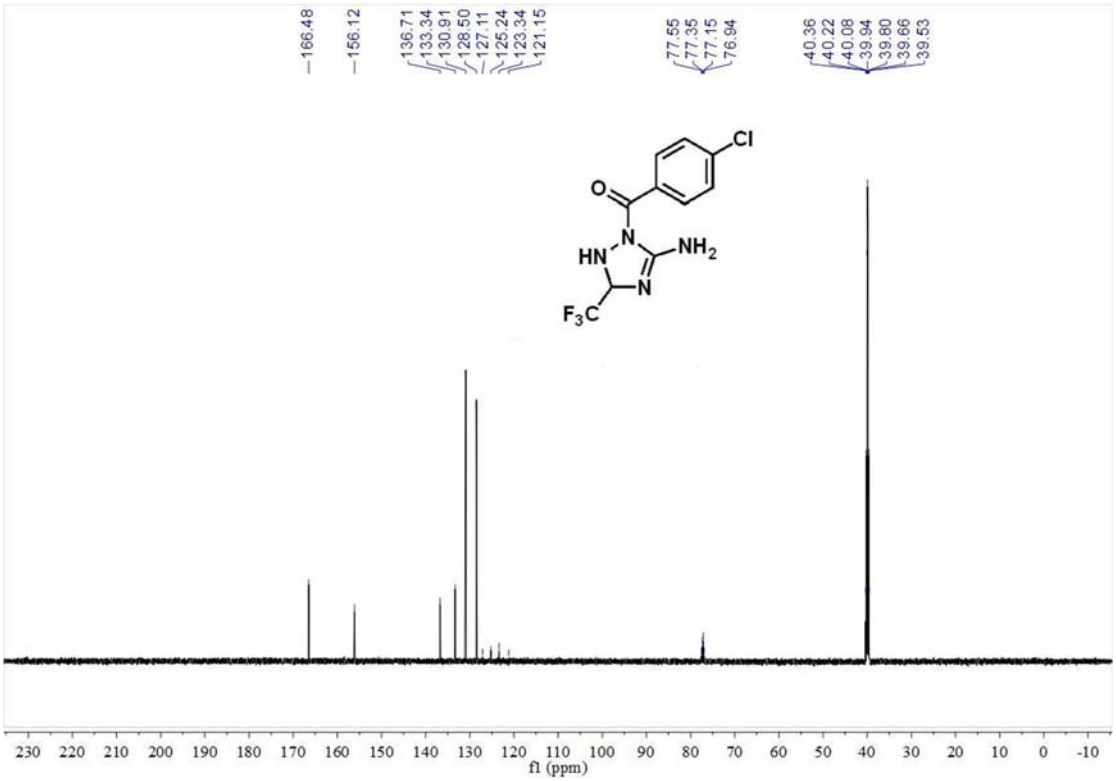
m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
273.14009	11125195.0	0.63	67352.11	1.00		
277.07044	1753931392.0	100.00	65017.52	1.00	-0.95	$C_{10}H_9ON_4F_4$
278.06746	18703808.0	1.07	68185.45	1.00		
278.07375	185596080.0	10.58	65501.78	1.00		
279.08599	25655622.0	1.46	66348.16	1.00		



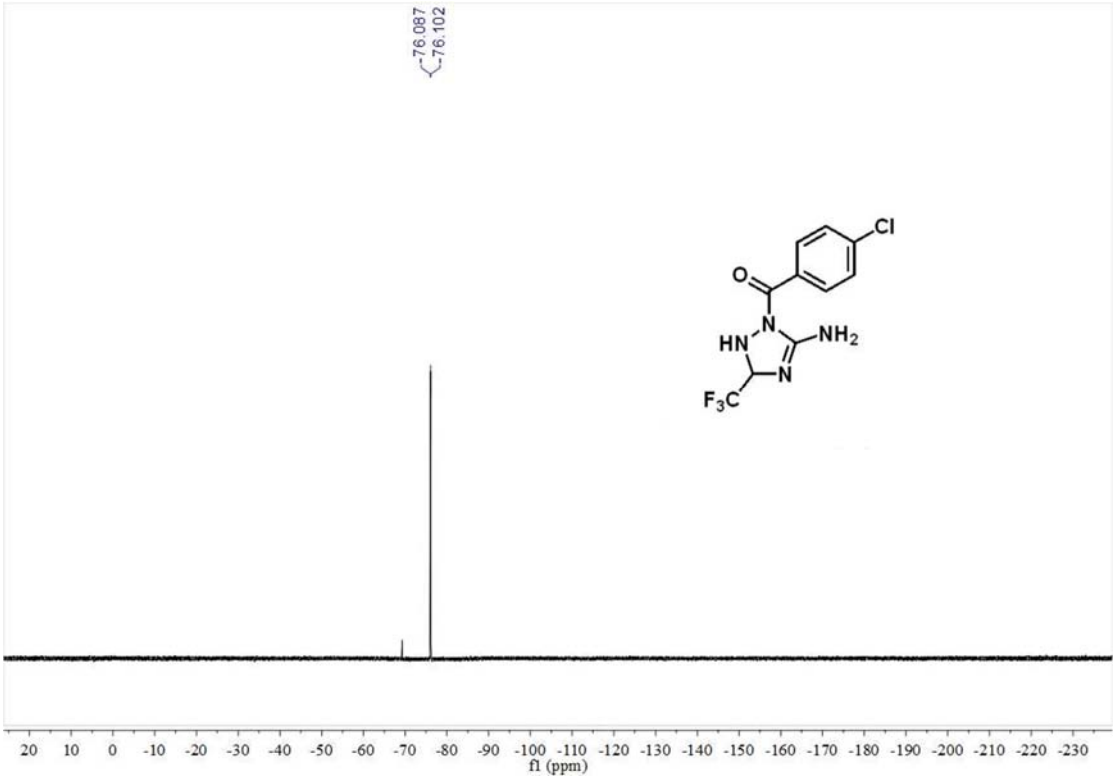
HRMS (ESI) copy of compound **2i**



[1]H NMR (400 MHz) spectrum of **2j** in DMSO-*d*$_6$



$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **2j** in DMSO-*d*$_6$



^{19}F NMR (376 MHz) spectrum of **2j** in DMSO-*d*$_6$

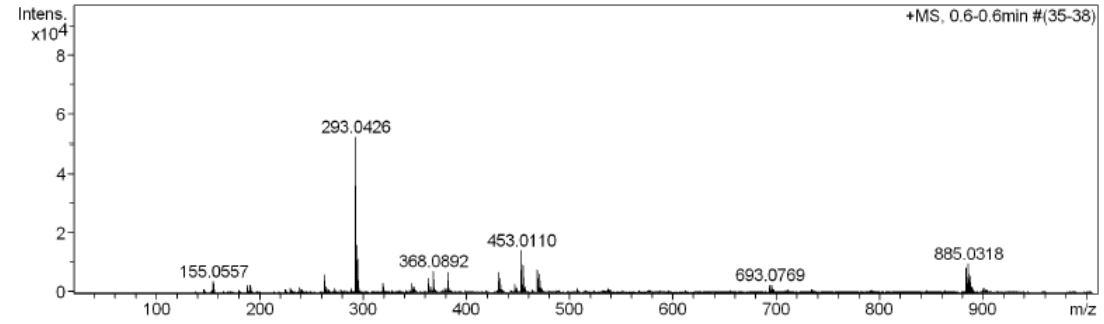
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\user\liuxiaoling20210623-9.d	Acquisition Date	2021-6-23 10:48:53
Method	tune_low.m	Operator	BDAL@DE
Sample Name	1j	Instrument / Ser#	micrOTOF-Q 20453
Comment			

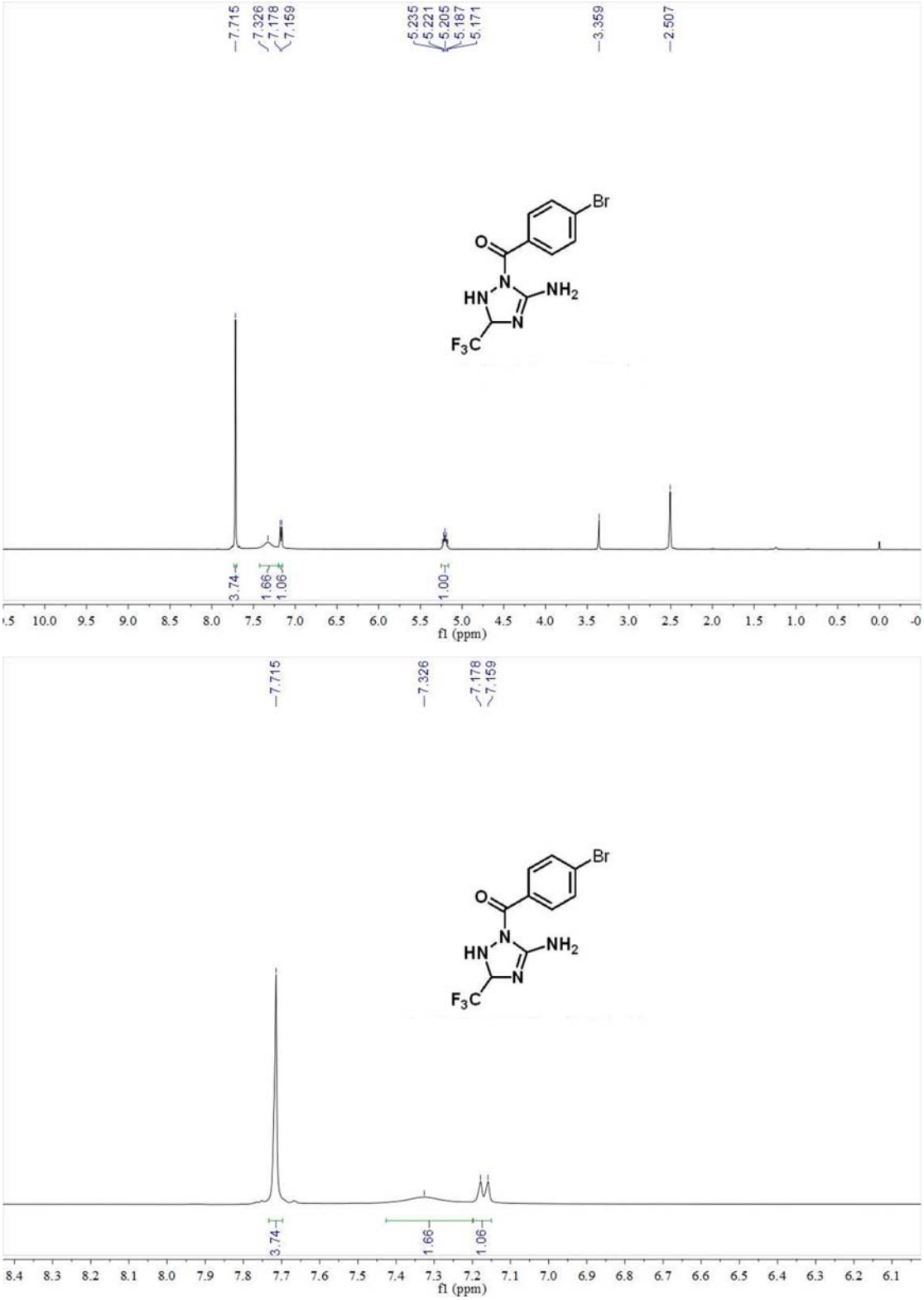
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 ¡ãC
Scan Begin	21 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste

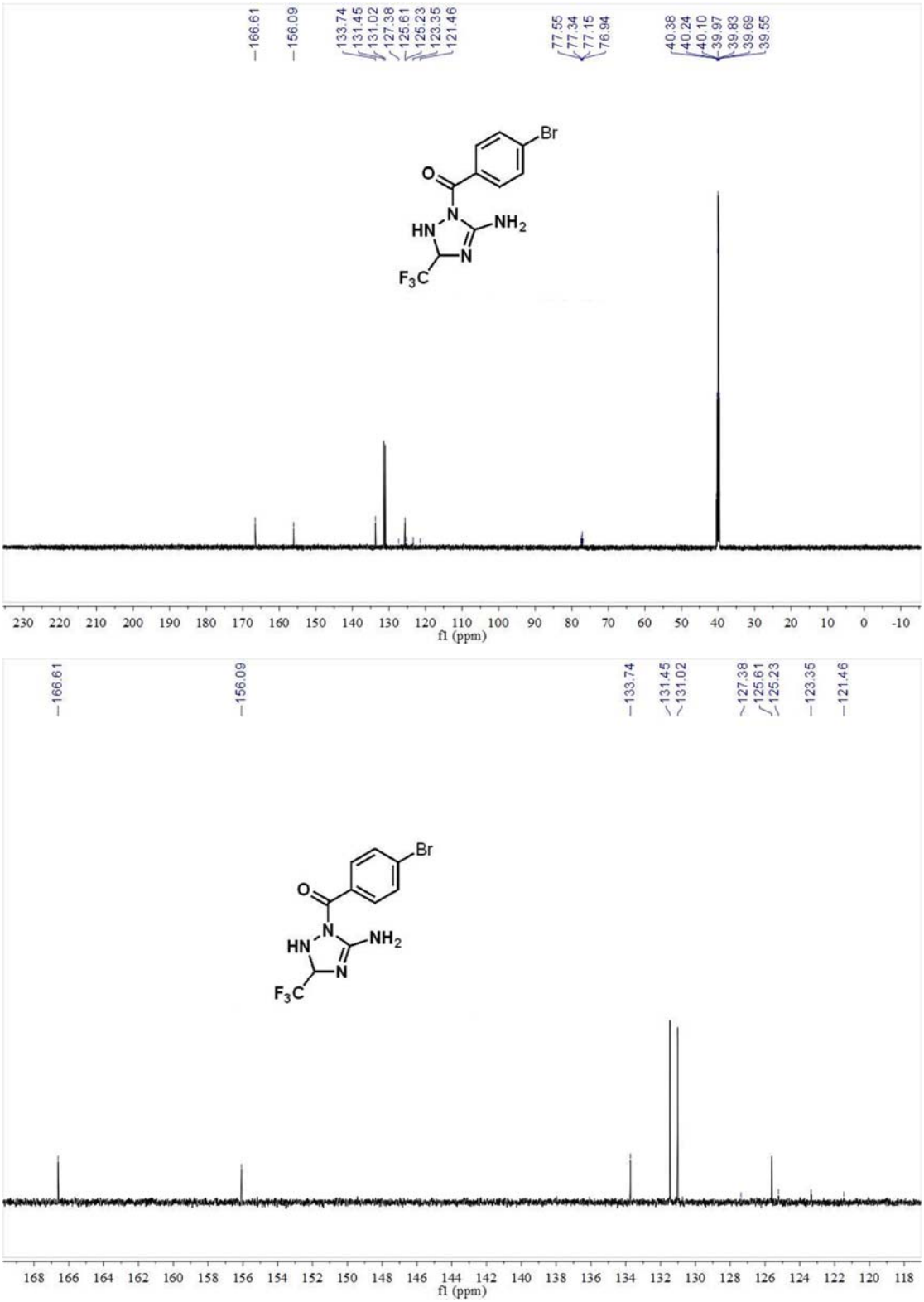


Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	ej¥Conf	mSigma	Std I	Std Mean m/z	Std I VarNorm	Std m/z Diff	Std Comb Dev
293.0426	1	C 10 H 9 Cl F 3 N 4 O	293.0411	-5.0	-5.5	6.5	ok	even	11.1	14.6	1.7	6.3	0.8	842.7

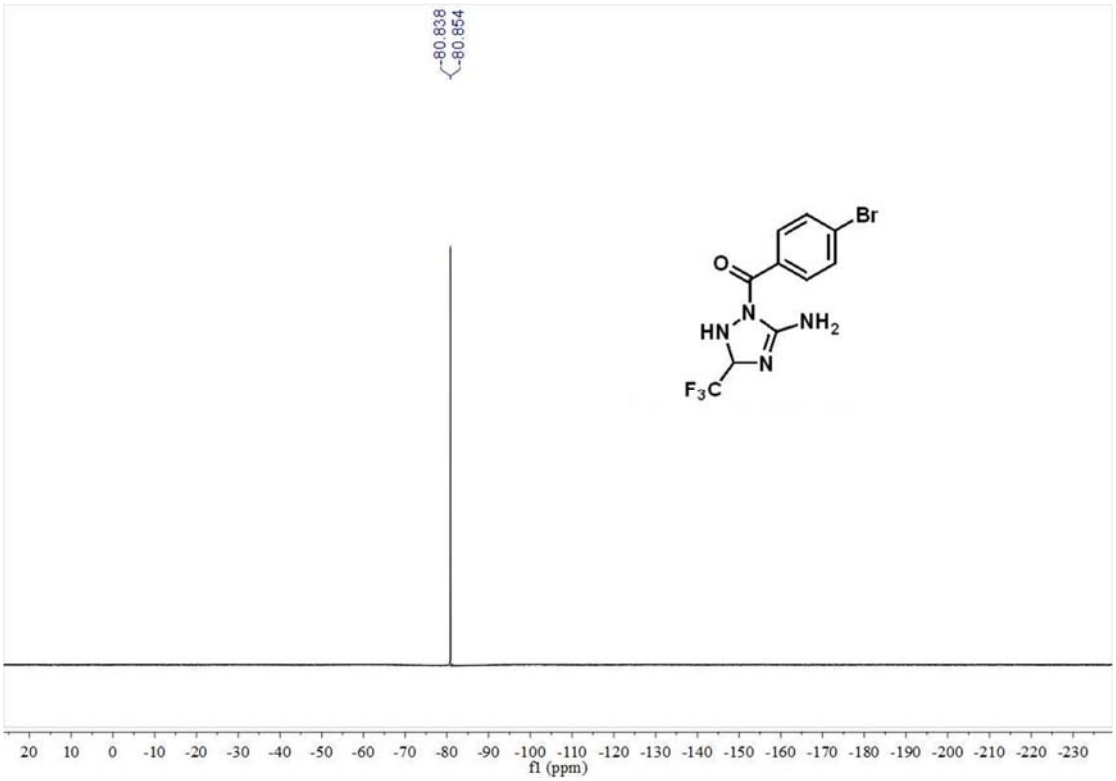
HRMS (ESI) copy of compound **2j**



[1]H NMR (400 MHz) spectrum of **2k** in DMSO-*d*$_6$



$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **2k** in DMSO-*d*$_6$



^{19}F NMR (376 MHz) spectrum of **2k** in DMSO-*d*$_6$

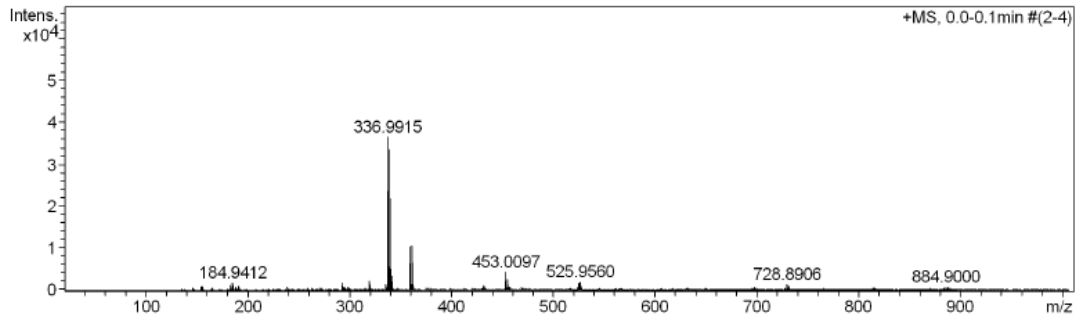
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\user\liuxiaoling20210623-10.d	Acquisition Date	2021-6-23 10:51:29
Method	tune_low.m	Operator	BDAL@DE
Sample Name	1k	Instrument / Ser#	micrOTOF-Q 20453
Comment			

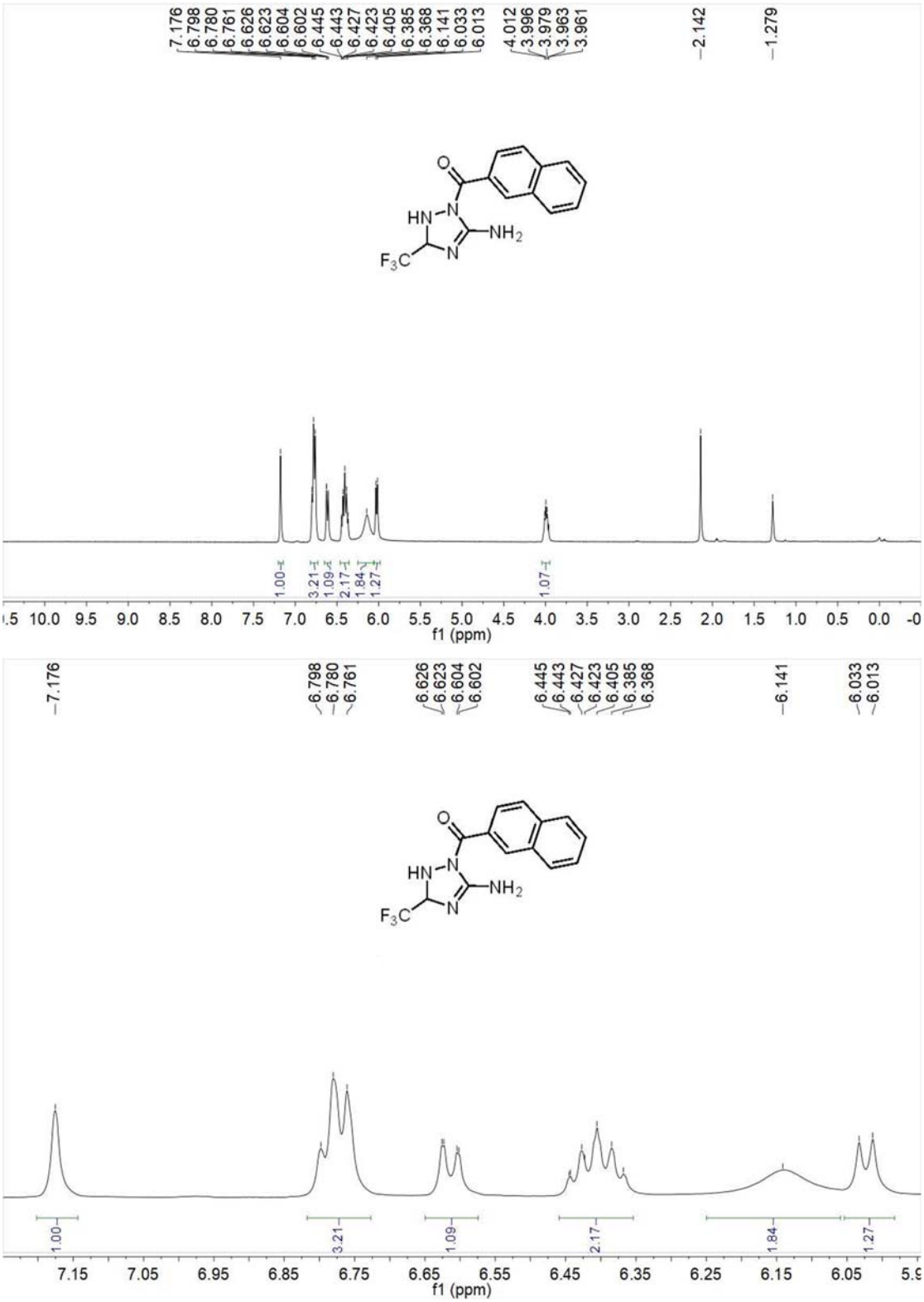
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 ¡ãC
Scan Begin	21 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste

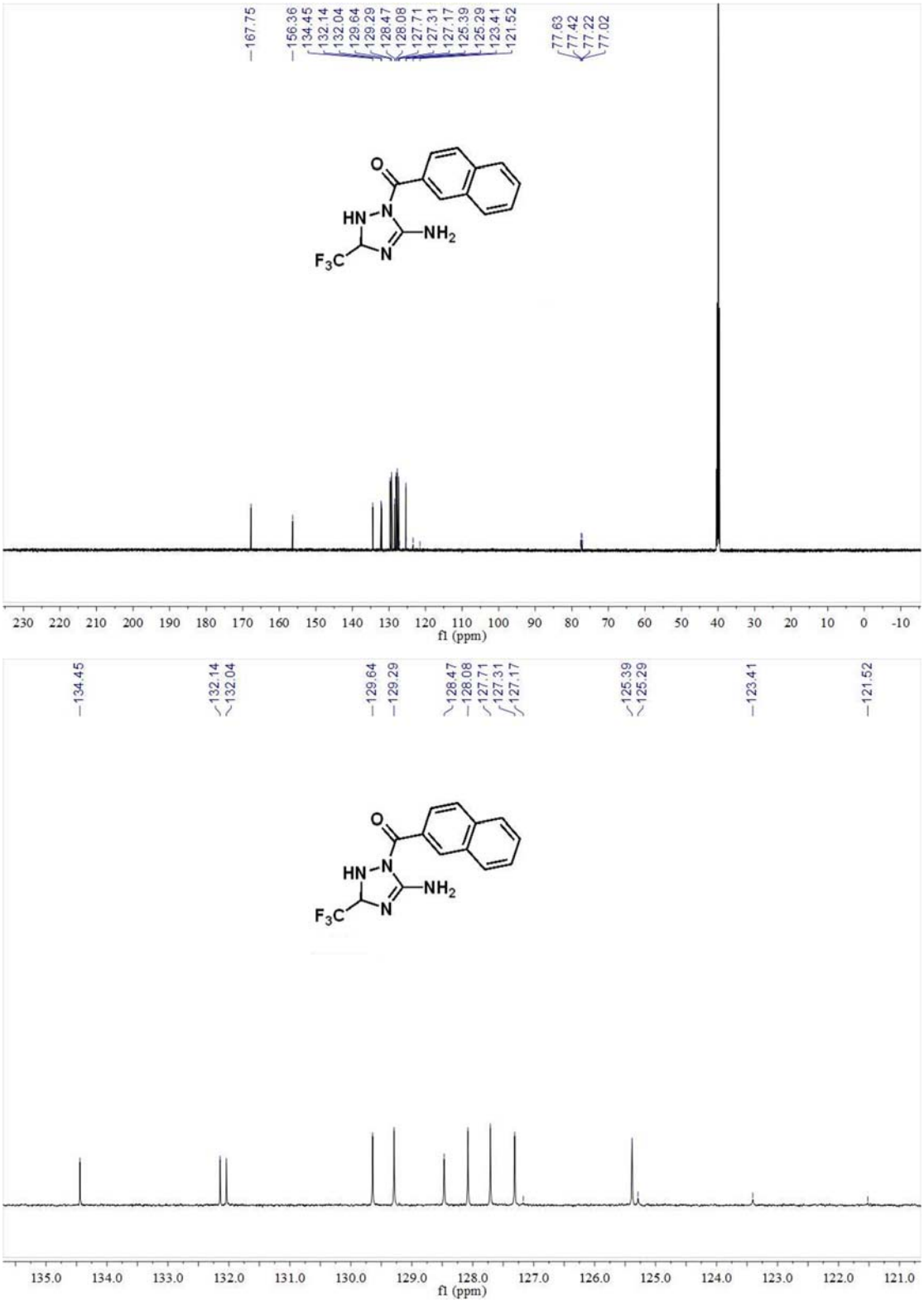


Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma	Std I	Std Mean m/z	Std I VarNorm	Std m/z Diff	Std Comb Dev
336.9915	1	C 10 H 9 Br F 3 N 4 O	336.9906	-2.5	-2.6	6.5	ok	even	24.1	18.2	1.0	5.4	1.0	842.7

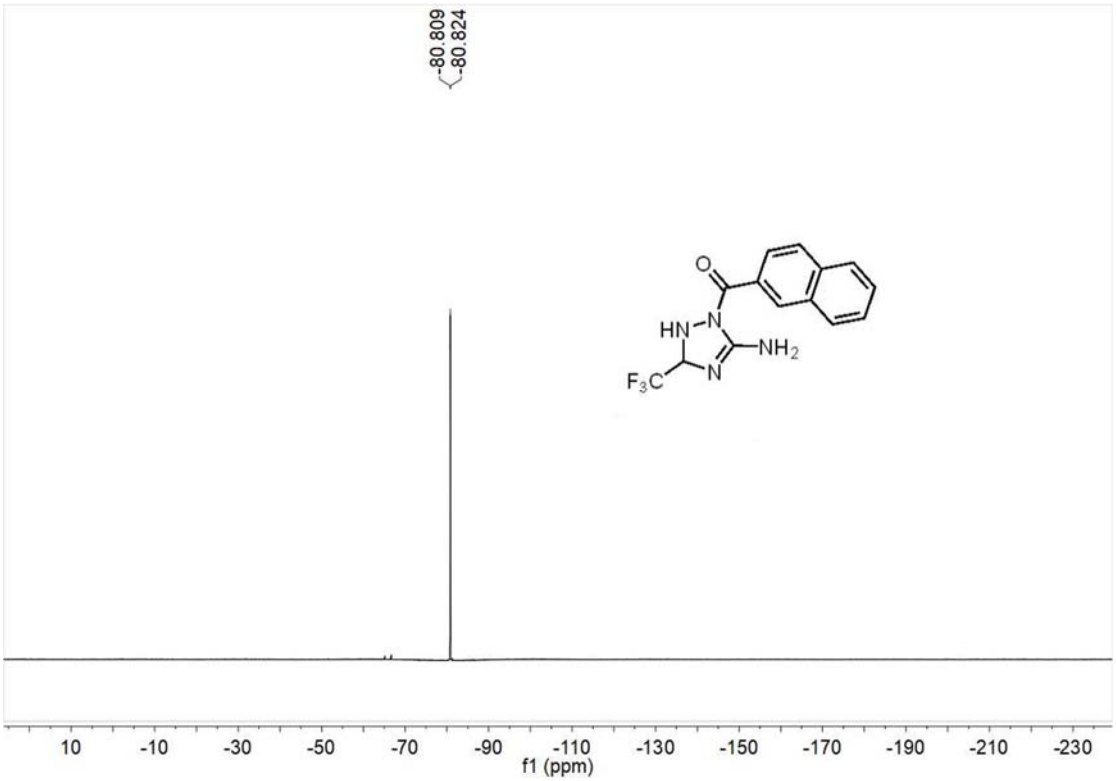
HRMS (ESI) copy of compound **2k**



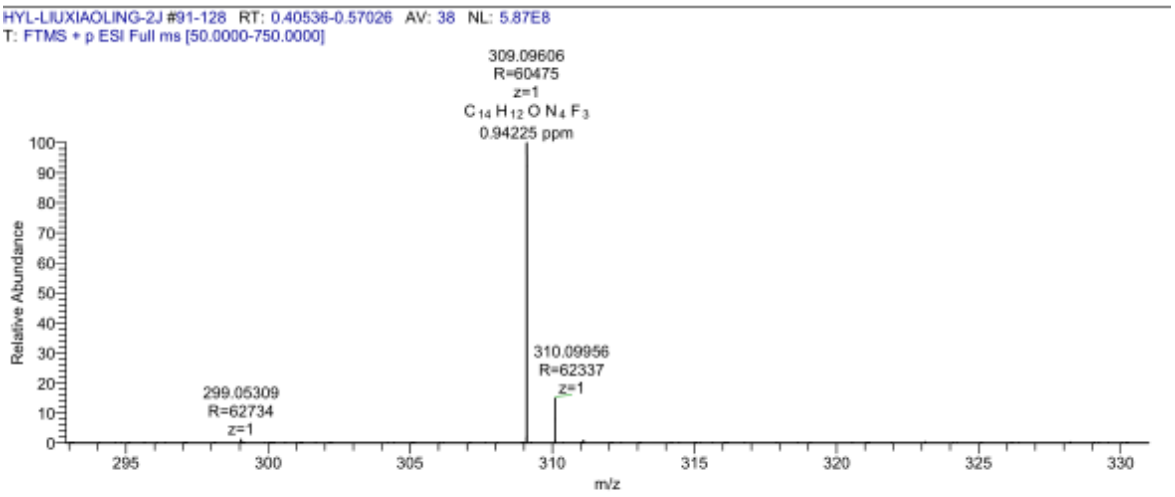
[1]H NMR (400 MHz) spectrum of **2o** in DMSO-*d6*



$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **2o** in DMSO-*d*$_6$

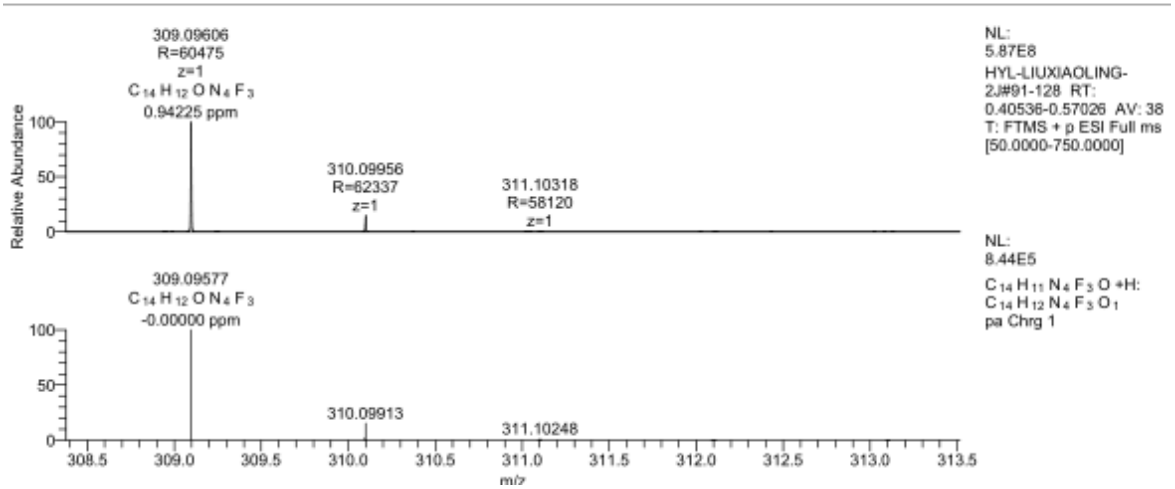


^{19}F NMR (376 MHz) spectrum of **2o** in DMSO-d_6

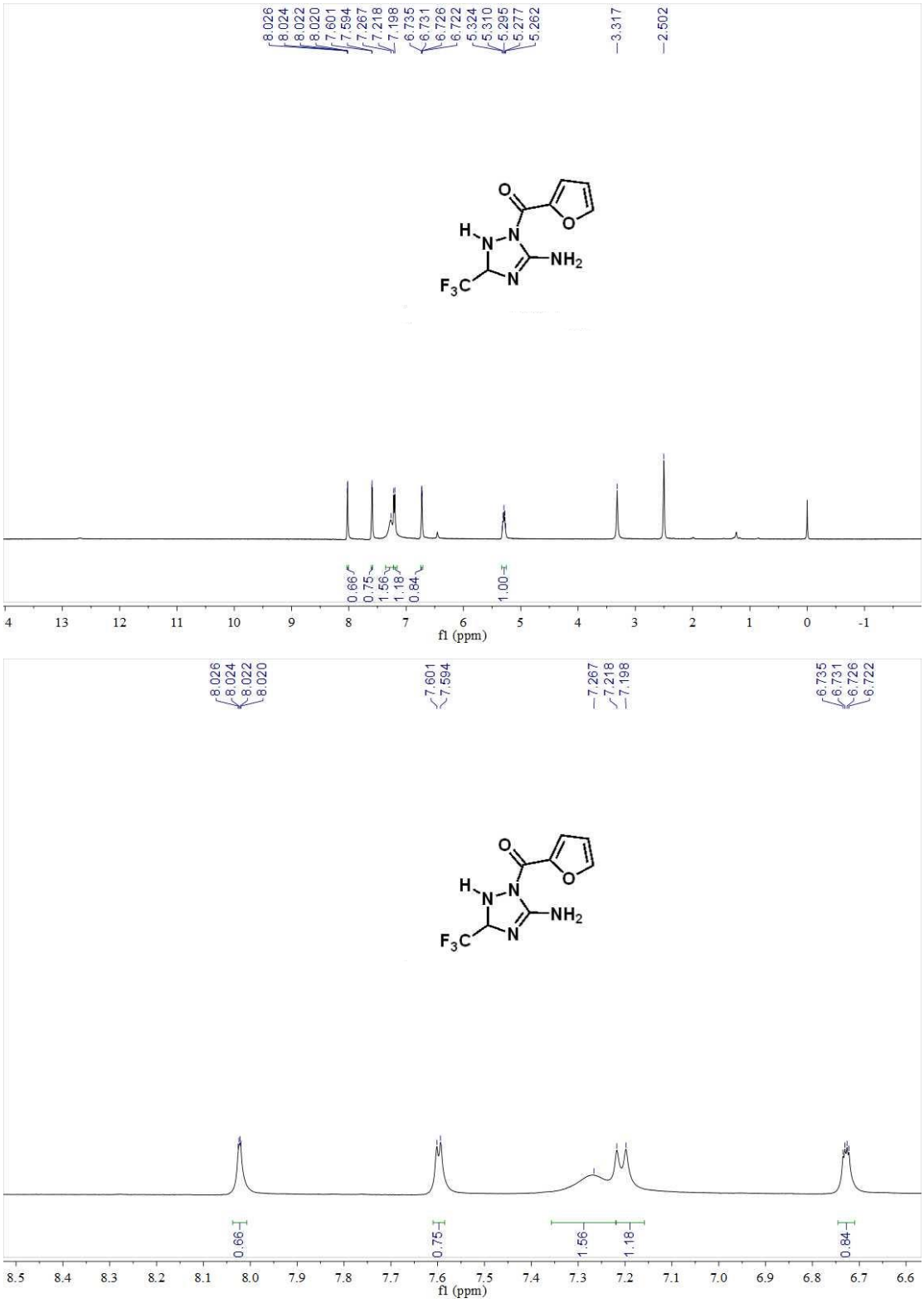


HYL-LIUXIAOLING-2J#91-128 RT: 0.40536-0.57026 AV: 38
T: FTMS + p ESI Full ms [50.0000-750.0000]
m/z= 292.88689-330.98834

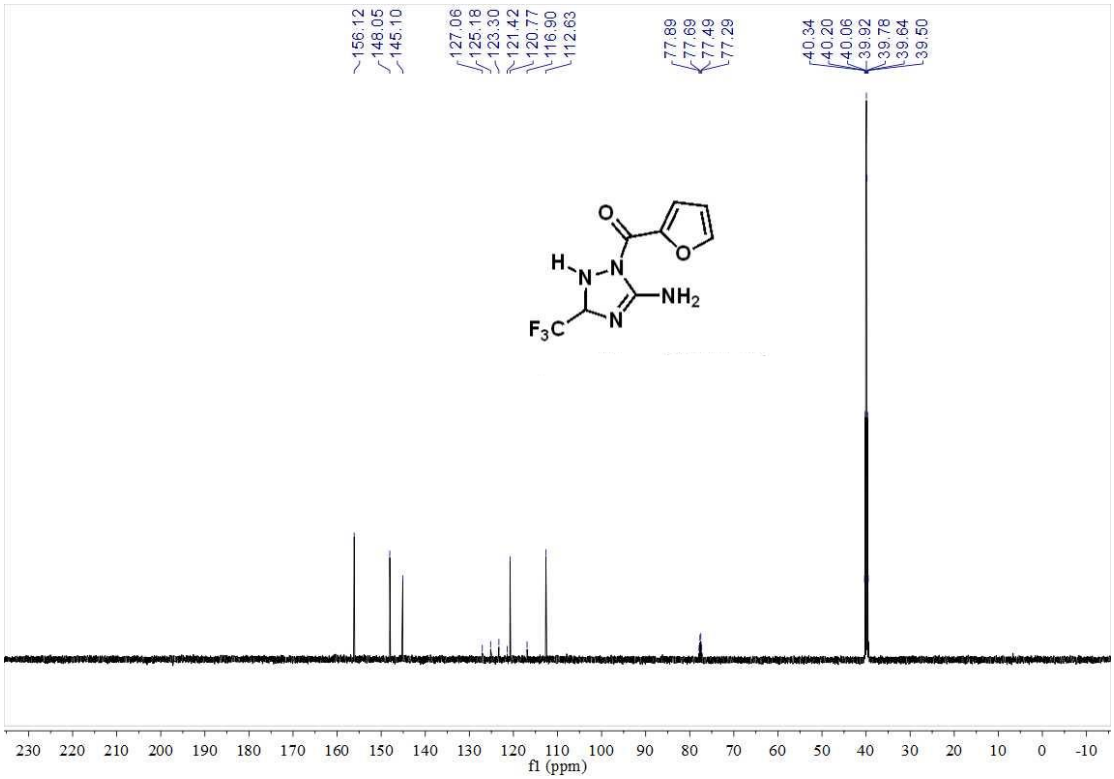
m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
299.05309	7544616.0	1.27	62733.59	1.00		
309.09606	593948288.0	100.00	60474.58	1.00	0.94	$C_{14}H_{12}ON_4F_3$
310.09956	88484088.0	14.90	62337.25	1.00		
311.10318	5990641.5	1.01	58119.59	1.00		
323.11169	2707042.3	0.46	61122.30	1.00		



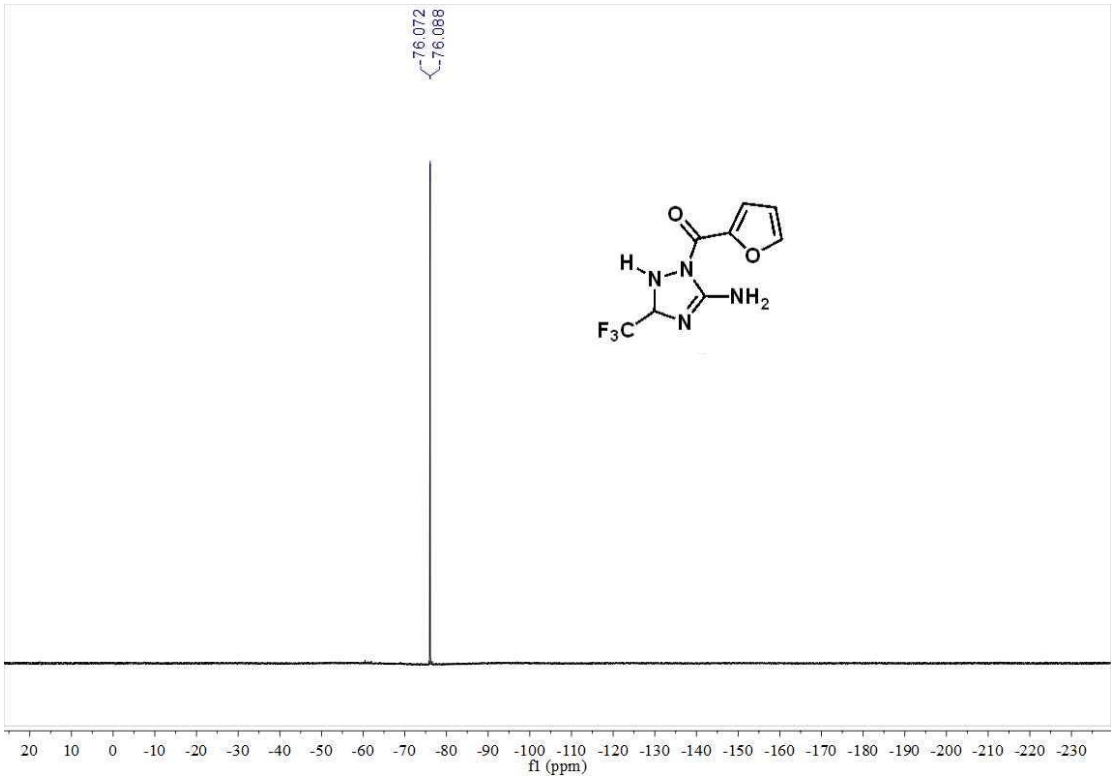
HRMS (ESI) copy of compound **2o**



^{1}H NMR (400 MHz) spectrum of **2p** in DMSO-*d*$_6$



$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **2p** in DMSO-*d*$_6$



^{19}F NMR (376 MHz) spectrum of **2p** in DMSO-*d*$_6$

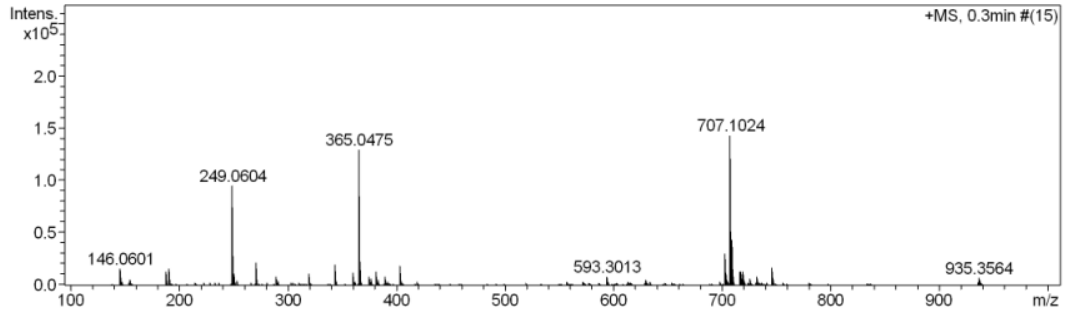
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\user\liuxiaoling20211124-1.d	Acquisition Date	2021-11-24 10:55:44
Method	tune_low.m	Operator	BDAL@DE
Sample Name	2p	Instrument / Ser#	micrOTOF-Q 20453
Comment			

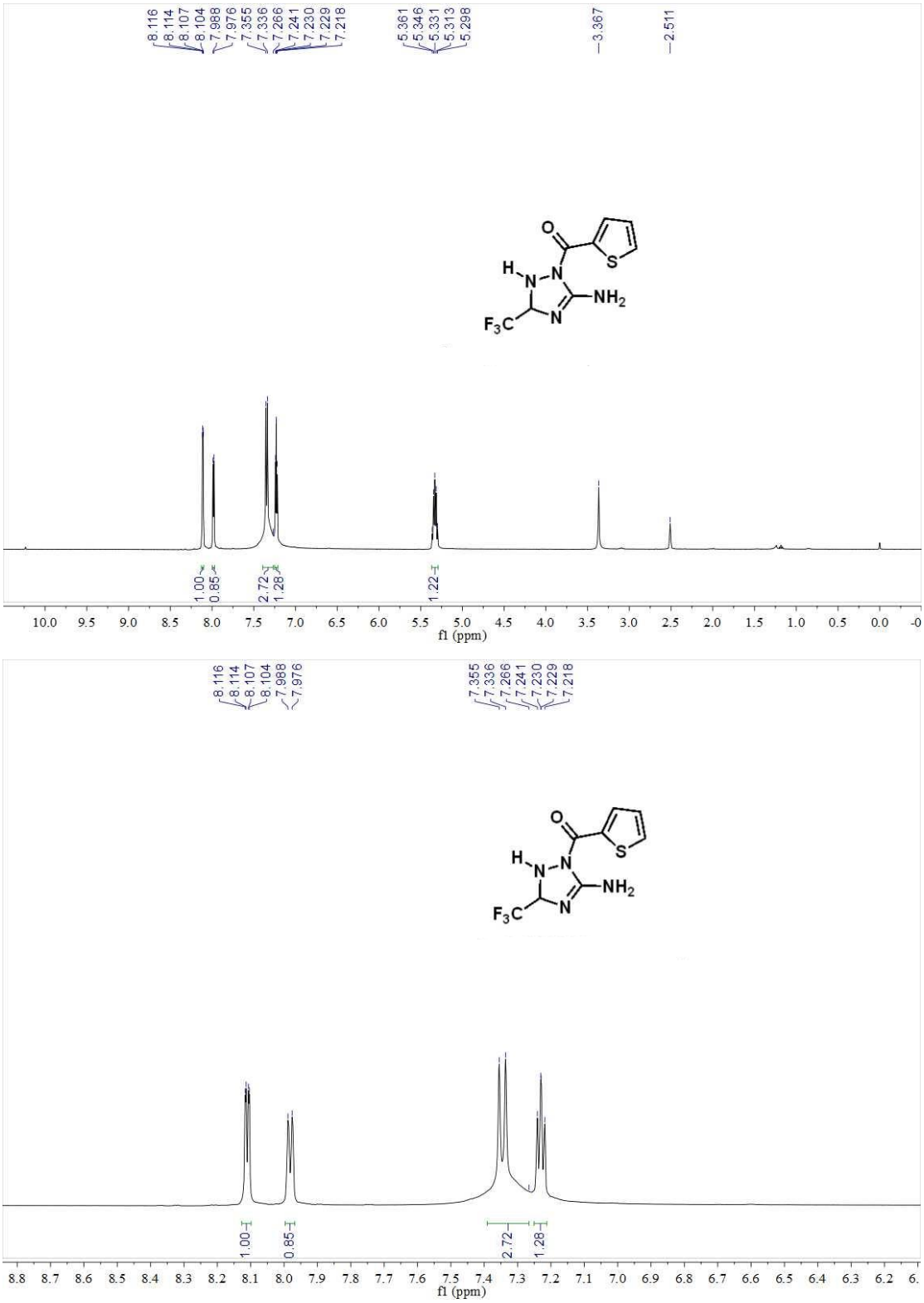
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 ¡ãC
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste

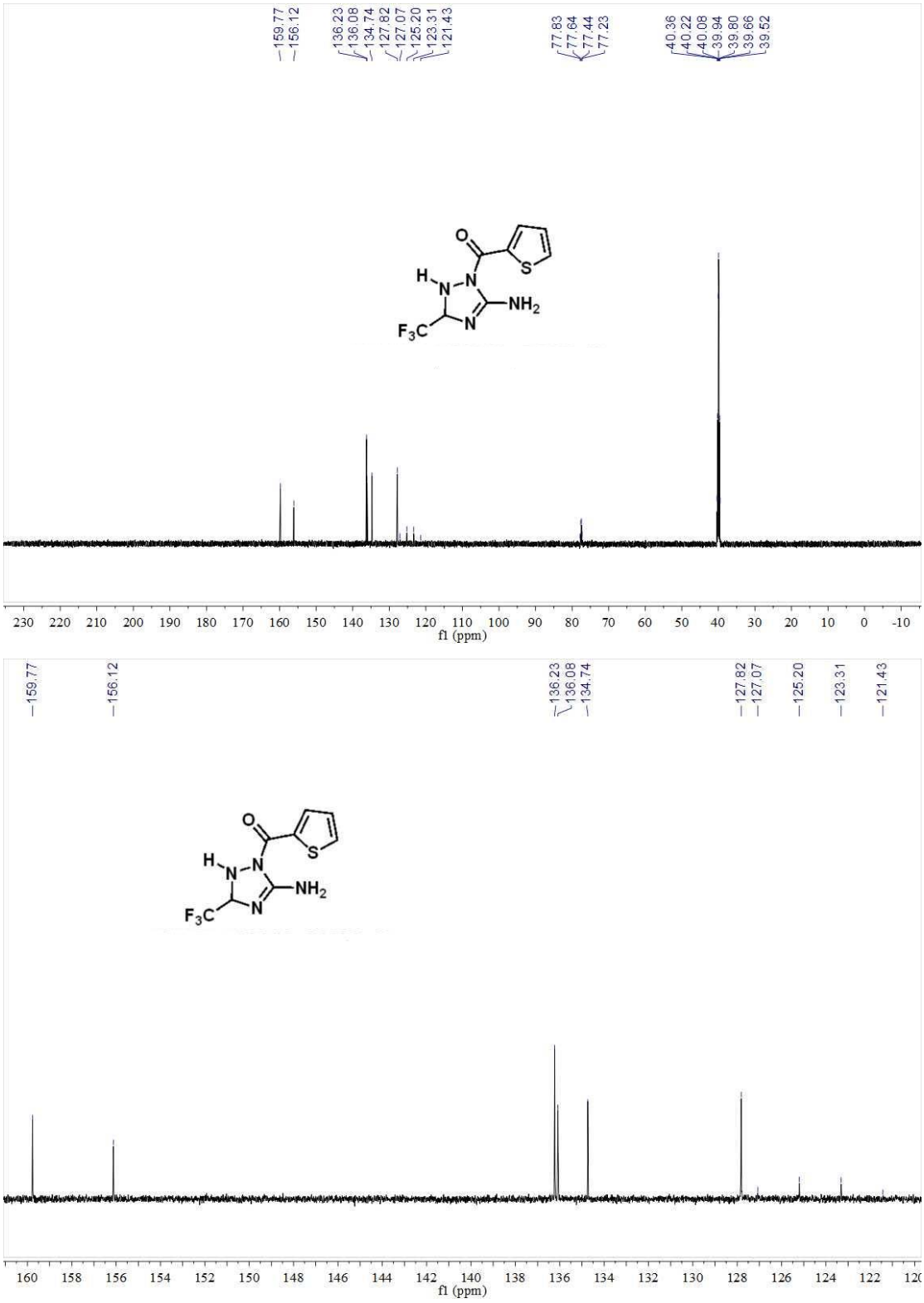


Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	e¡¥ Conf	mSigma	Std I	Std Mean m/z	Std I Var Norm	Std m/z Diff	Std Comb Dev
249.0604	1	C 8 H 8 F 3 N 4 O 2	249.0594	-4.3	-3.4	5.5	ok	even	6.0	11.4	1.1	5.9	2.2	842.7

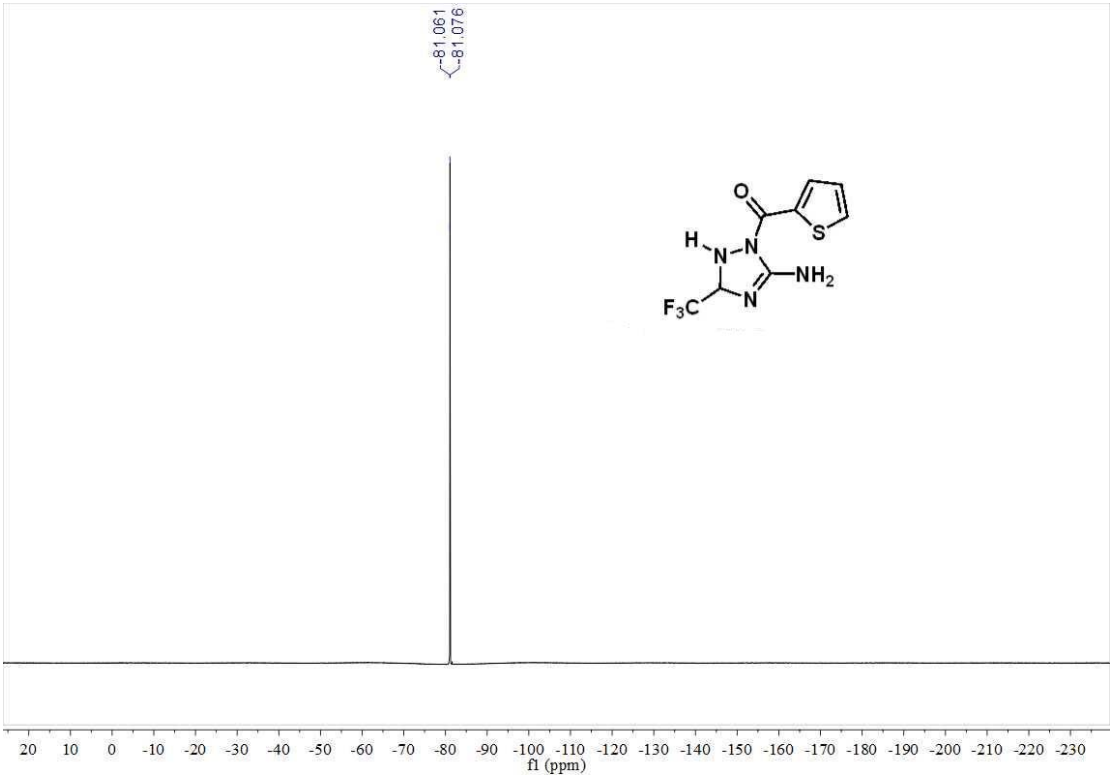
HRMS (ESI) copy of compound **2p**



^{1}H NMR (400 MHz) spectrum of **2q** in DMSO-d_6



$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **2q** in DMSO-*d*$_6$



^{19}F NMR (376 MHz) spectrum of **2q** in DMSO-*d*$_6$

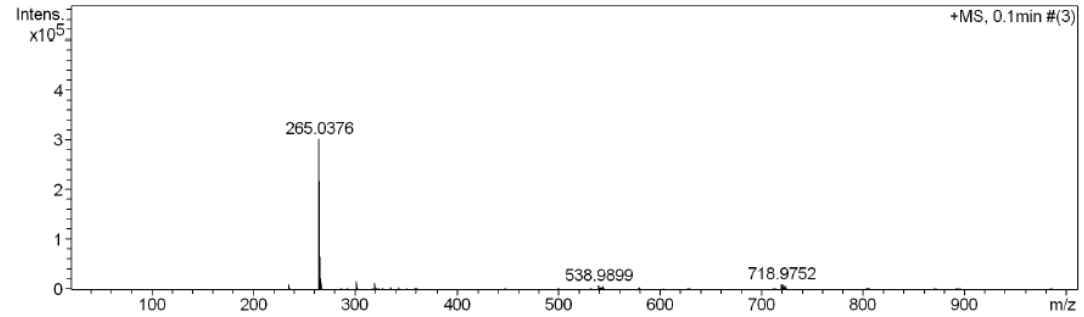
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\user\liuxiaoling20210623-12.d	Acquisition Date	2021-6-23 10:56:06
Method	tune_low.m	Operator	BDAL@DE
Sample Name	1m	Instrument / Ser#	micrOTOF-Q 20453
Comment			

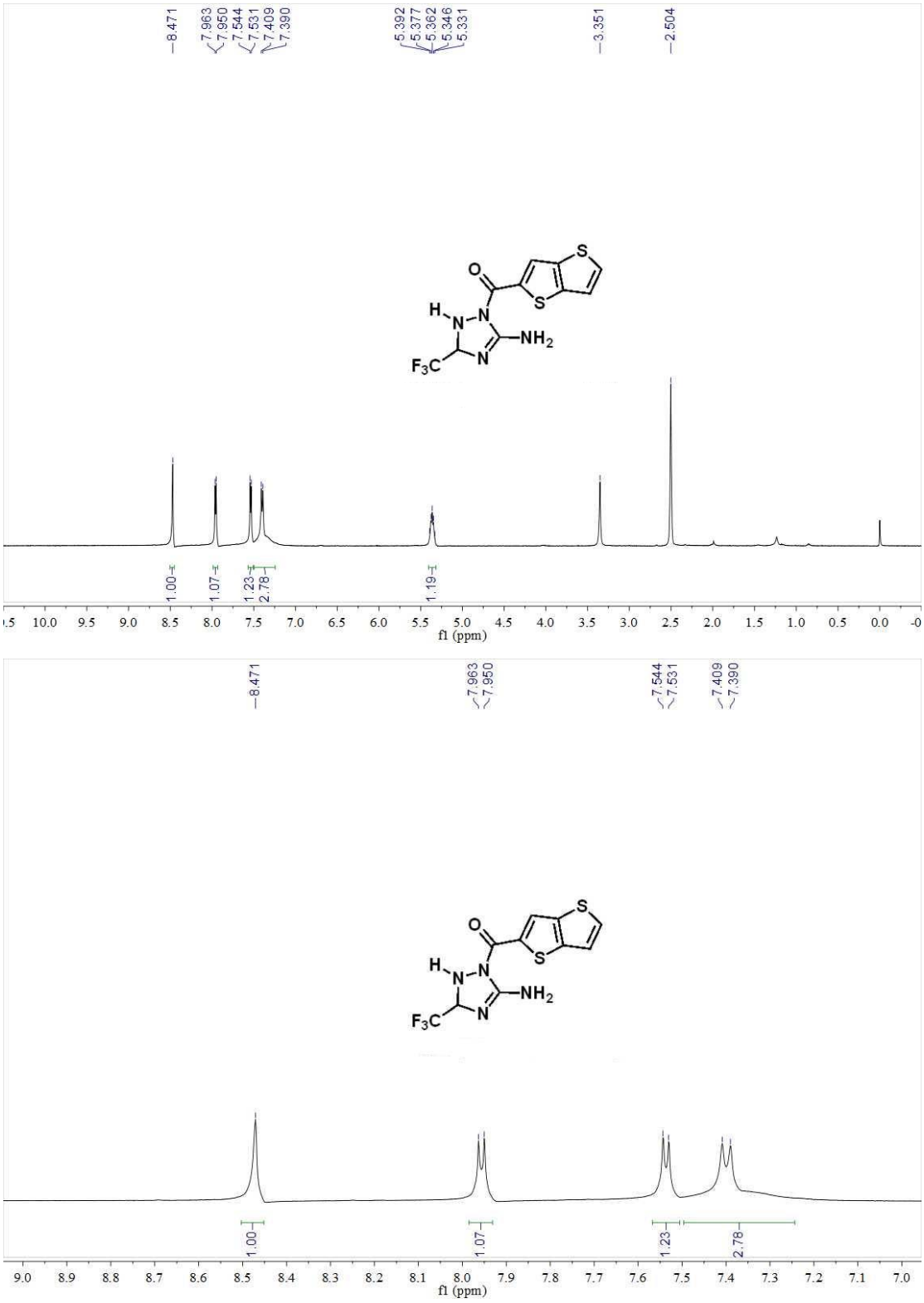
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 ¡ãC
Scan Begin	21 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste

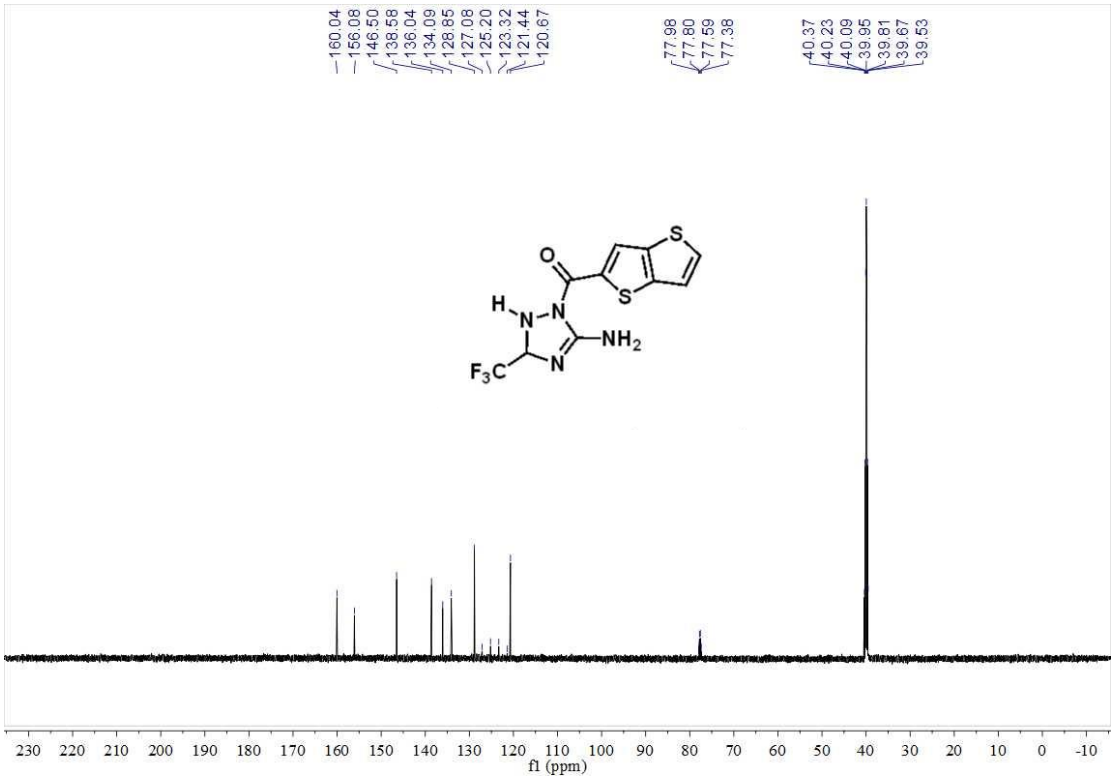


Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	ej¥Conf	mSigma	Std I	Std Mean m/z	Std I Var Norm	Std m/z Diff	Std Comb Dev
265.0376	1	C 8 H 8 F 3 N 4 O S	265.0365	-4.2	-3.5	5.5	ok	even	5.3	11.8	1.1	4.7	1.6	842.7

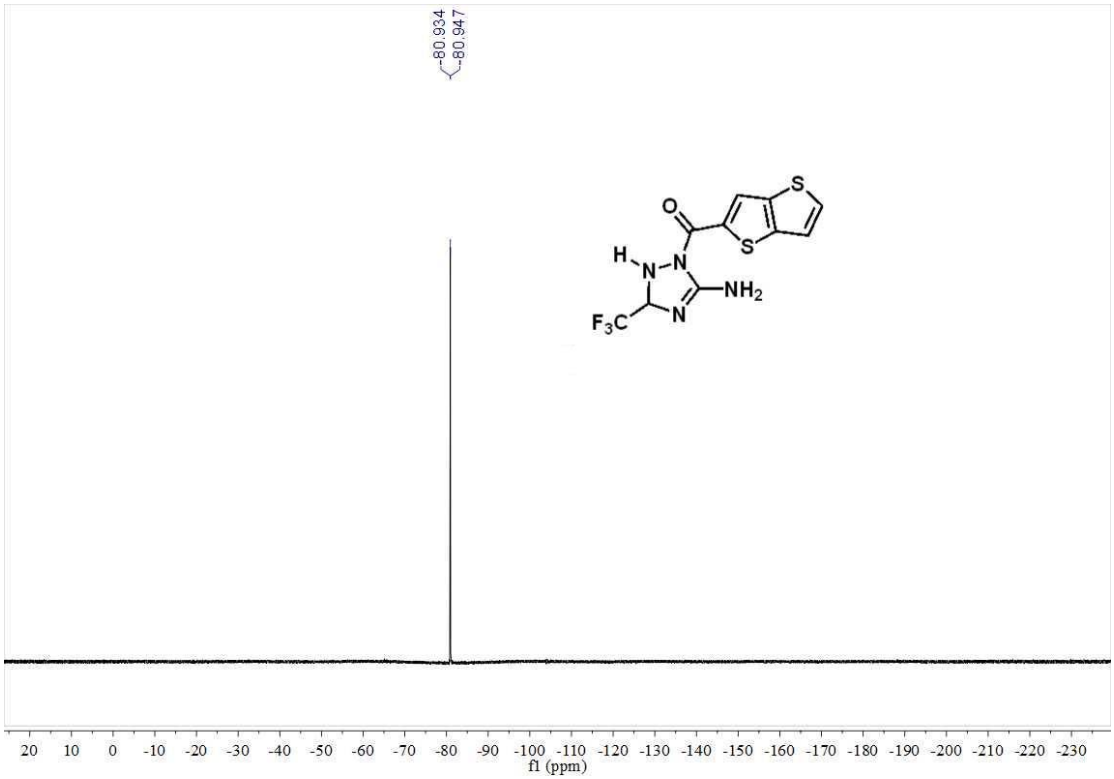
HRMS (ESI) copy of compound **2q**



^{1}H NMR (400 MHz) spectrum of **2r** in DMSO-*d*$_6$



$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **2r** in DMSO-*d*$_6$



^{19}F NMR (376 MHz) spectrum of **2r** in DMSO-*d*$_6$

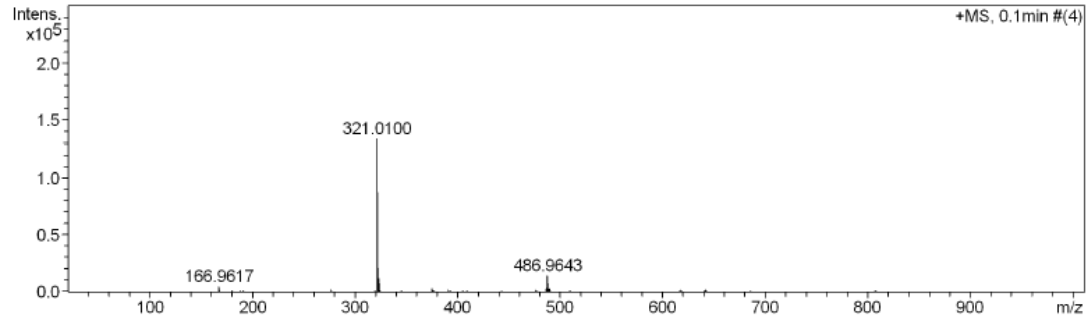
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\user\liuxiaoling20210623-13.d	Acquisition Date	2021-6-23 11:00:31
Method	tune_low.m		
Sample Name	1n	Operator	BDAL@DE
Comment		Instrument / Ser#	micrOTOF-Q 20453

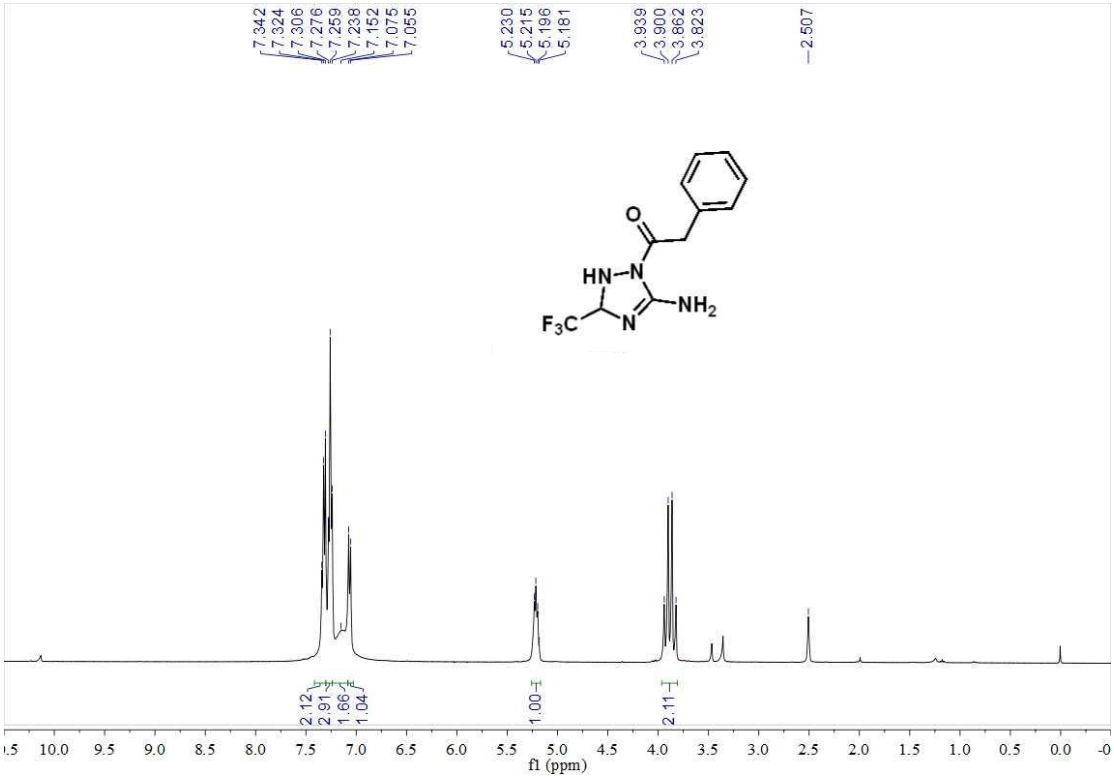
Acquisition Parameter

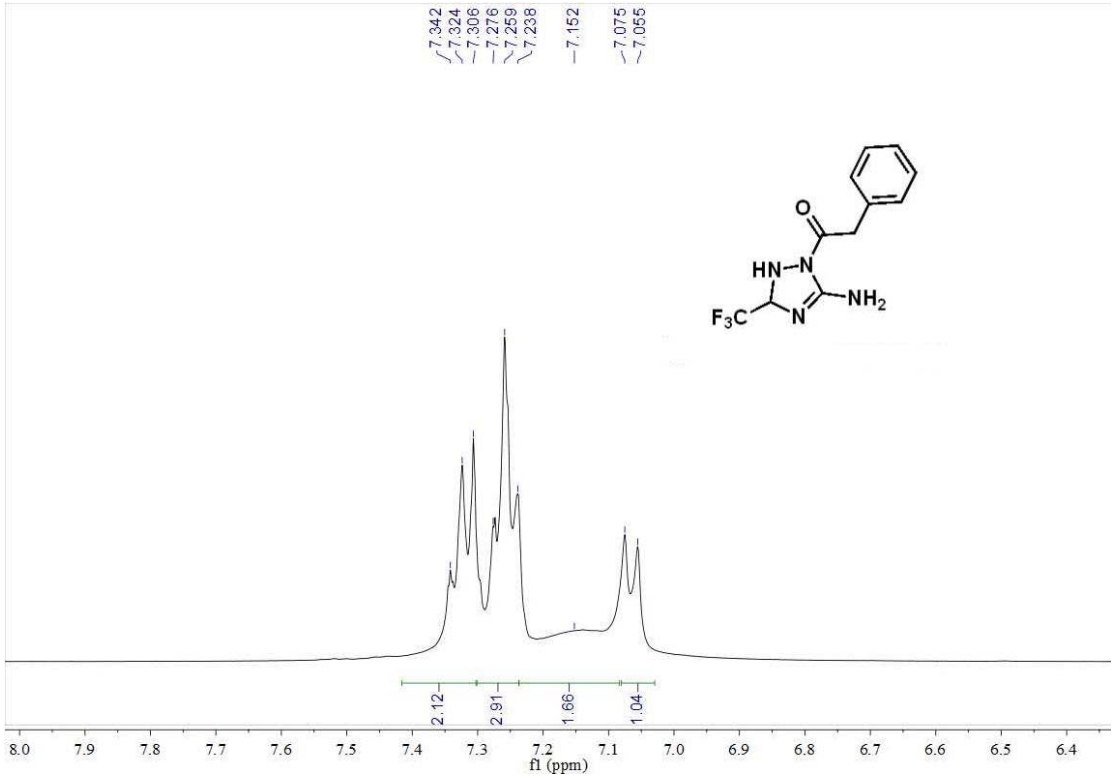
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 ¡ãC
Scan Begin	21 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste



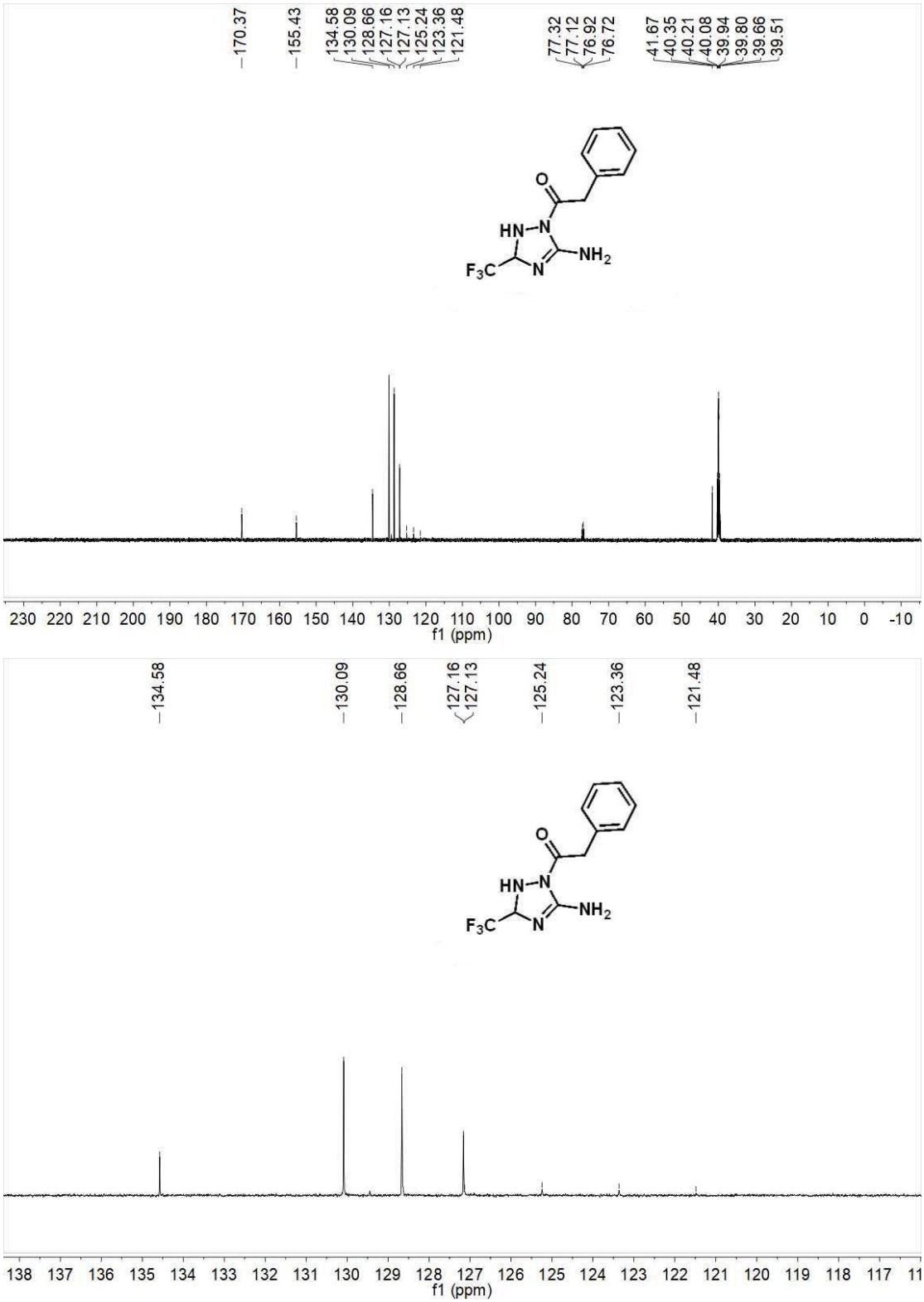
Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	ej¥ Conf	mSigma	Std I	Std Mean m/z	Std I VarNorm	Std m/z Diff	Std Comb Dev
321.0100	1	C 10 H 8 F 3 N 4 O S 2	321.0086	-4.4	-4.2	7.5	ok	even	8.7	17.6	1.4	8.4	1.1	842.7

HRMS (ESI) copy of compound **2r**

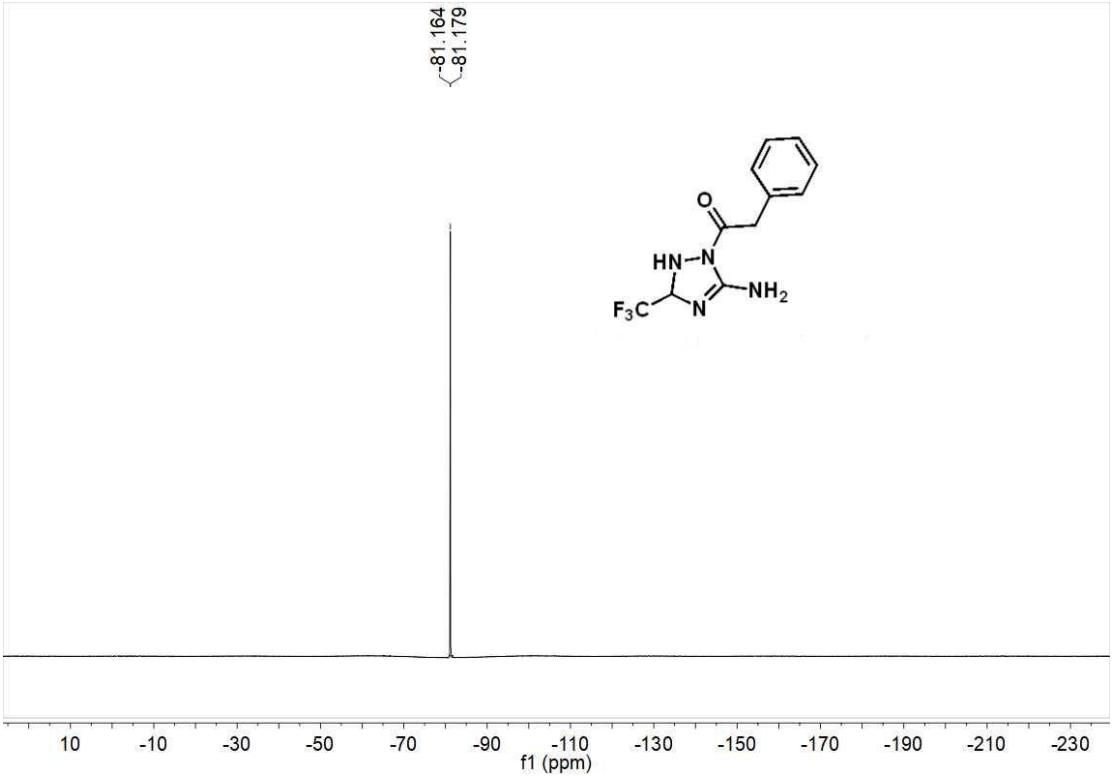




^{1}H NMR (400 MHz) spectrum of **2t** in DMSO-*d*$_{6}$



$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **2t** in DMSO-*d6*



^{19}F NMR (376 MHz) spectrum of **2t** in DMSO-d_6

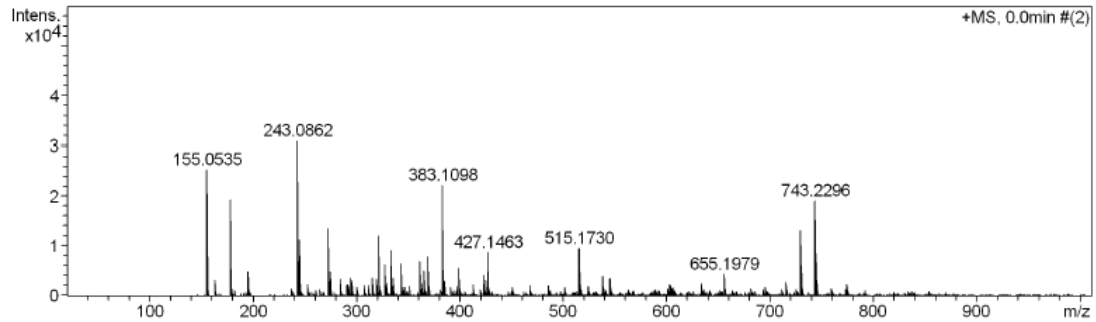
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\user\liuxiaoling20210623-14.d	Acquisition Date	2021-6-23 11:02:47
Method	tune_low.m	Operator	BDAL@DE
Sample Name	1o	Instrument / Ser#	micrOTOF-Q 20453
Comment			

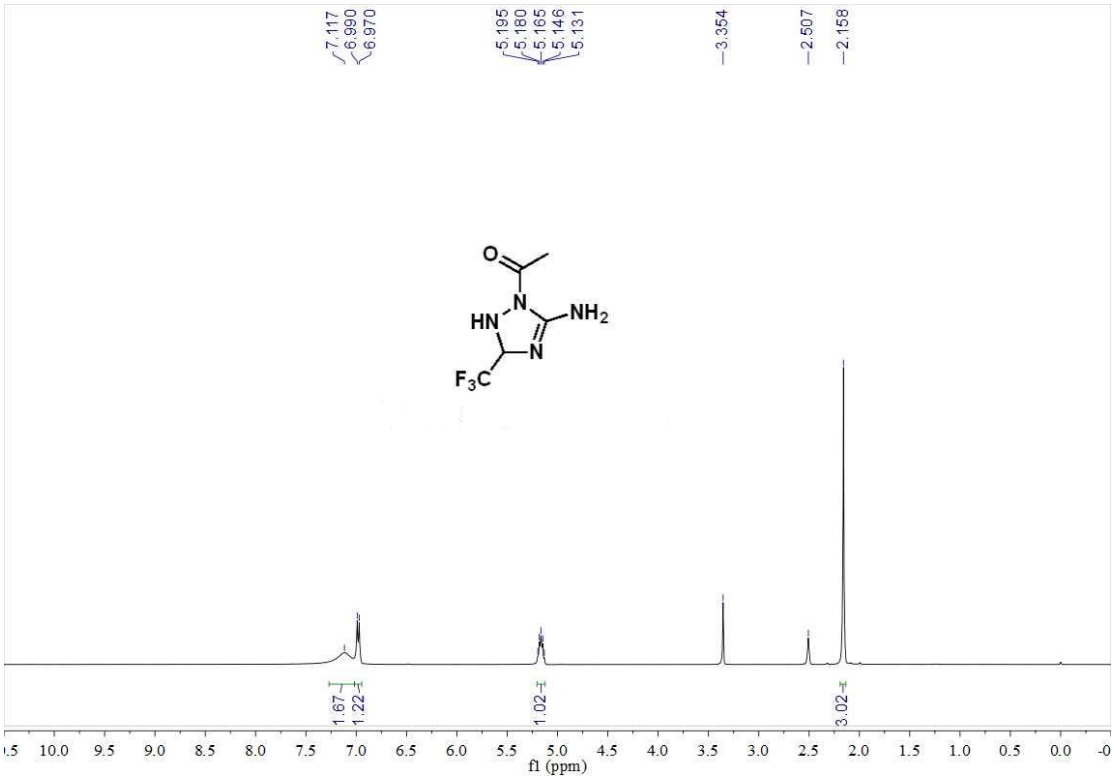
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 ¡ãC
Scan Begin	21 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste

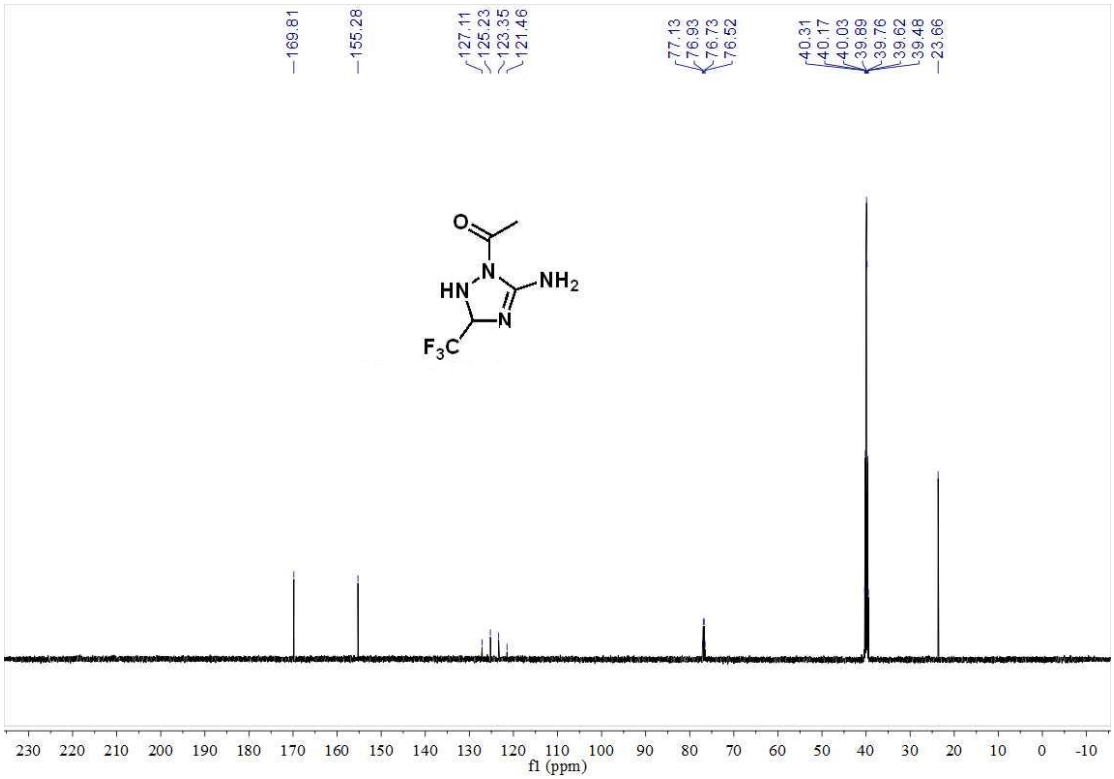


Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	ej¥Conf	mSigma	Std I	Std Mean m/z	Std I VarNorm	Std m/z Diff	Std Comb Dev
273.0966	1	C 11 H 12 F 3 N 4 O	273.0958	-2.9	-11.2	6.5	ok	even	214.3	382.3	4.9	229.0	0.8	842.7

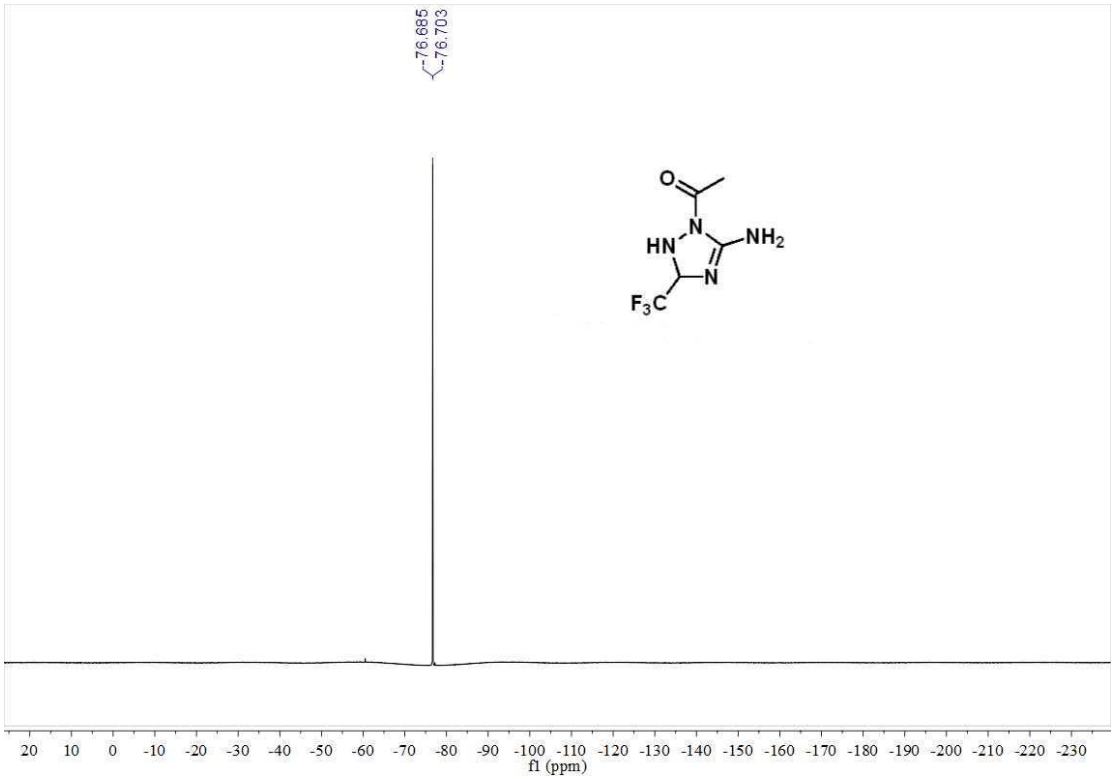
HRMS (ESI) copy of compound **2t**



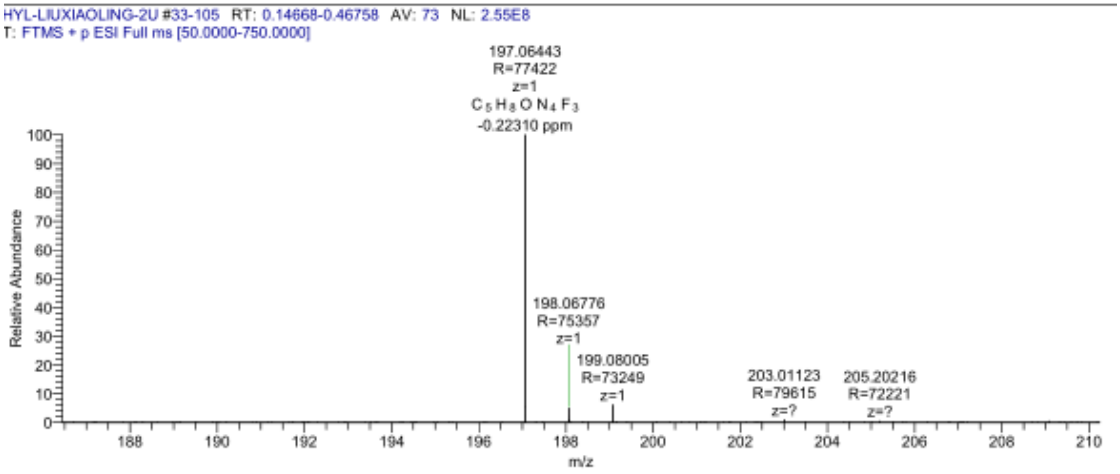
^{1}H NMR (400 MHz) spectrum of **2u** in DMSO-*d*$_6$



^{13}C{^{1}H} NMR (150 MHz) spectrum of **2u** in DMSO-*d*$_6$

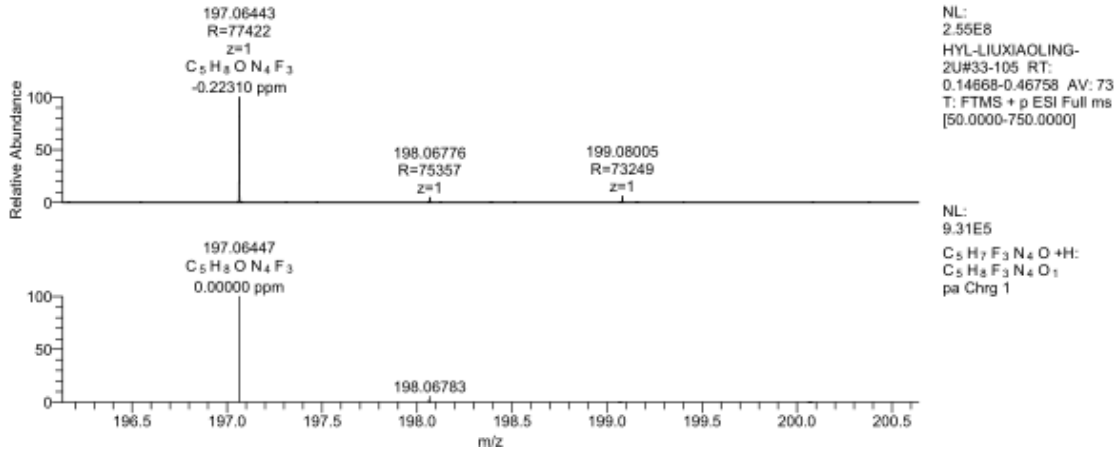


^{19}F NMR (376 MHz) spectrum of **2u** in DMSO-d_6

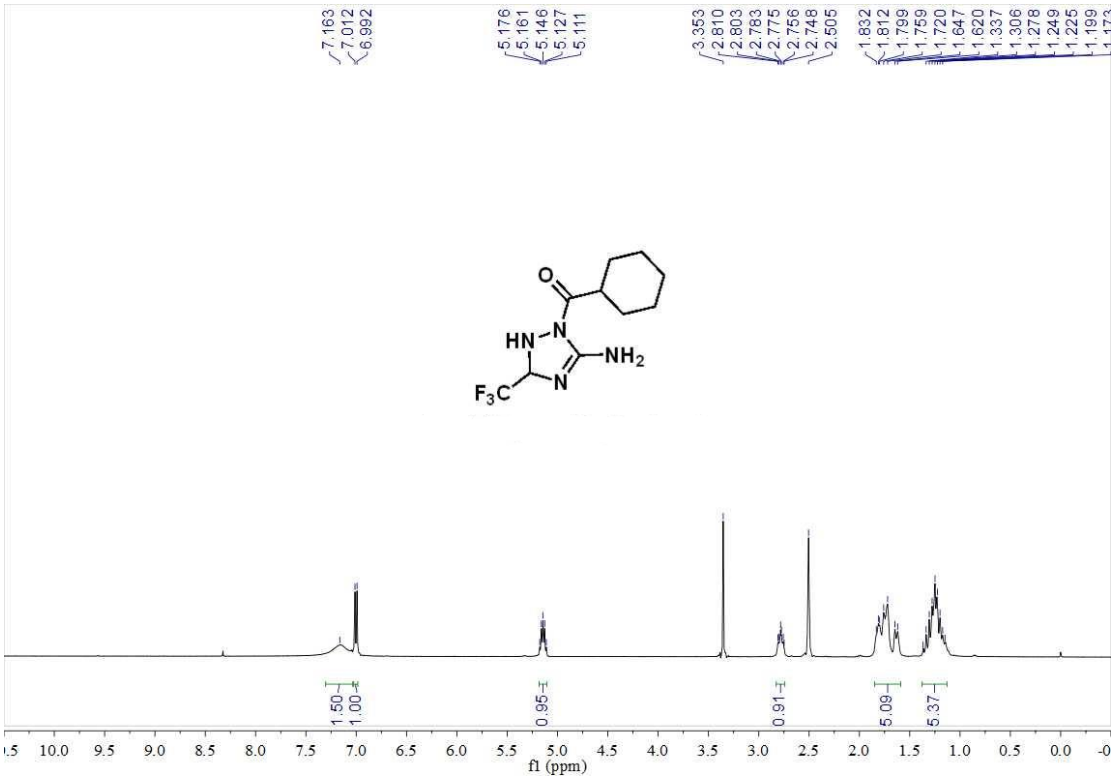


HYL-LIUXIAOLING-2U#33-105 RT: 0.14668-0.46758 AV: 73
T: FTMS + p ESI Full ms [50.0000-750.0000]
m/z= 186.44893-210.24046

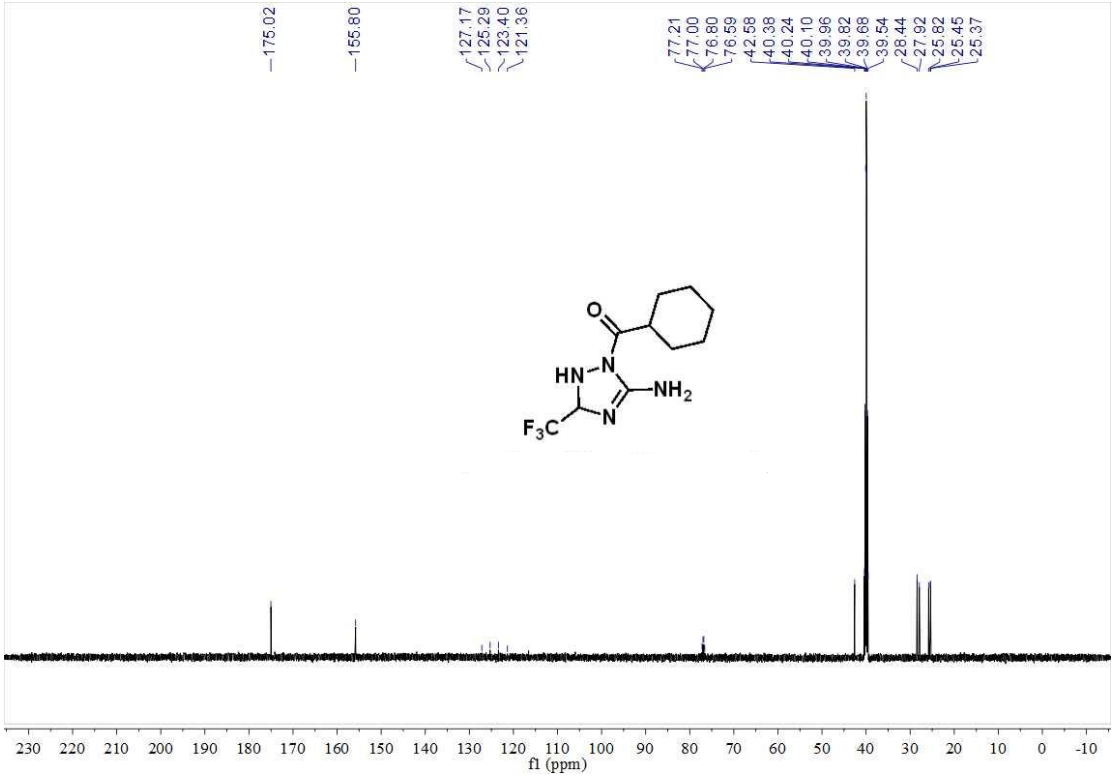
m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
197.06443	254637136.0	100.00	77422.06	1.00	-0.22	$C_5H_8ON_4F_3$
198.06143	3118452.5	1.22	75035.34	1.00		
198.06776	12910066.0	5.07	75356.79	1.00		
199.08005	16234504.0	6.38	73248.86	1.00		
203.01123	2156633.5	0.85	79615.36	0.00		

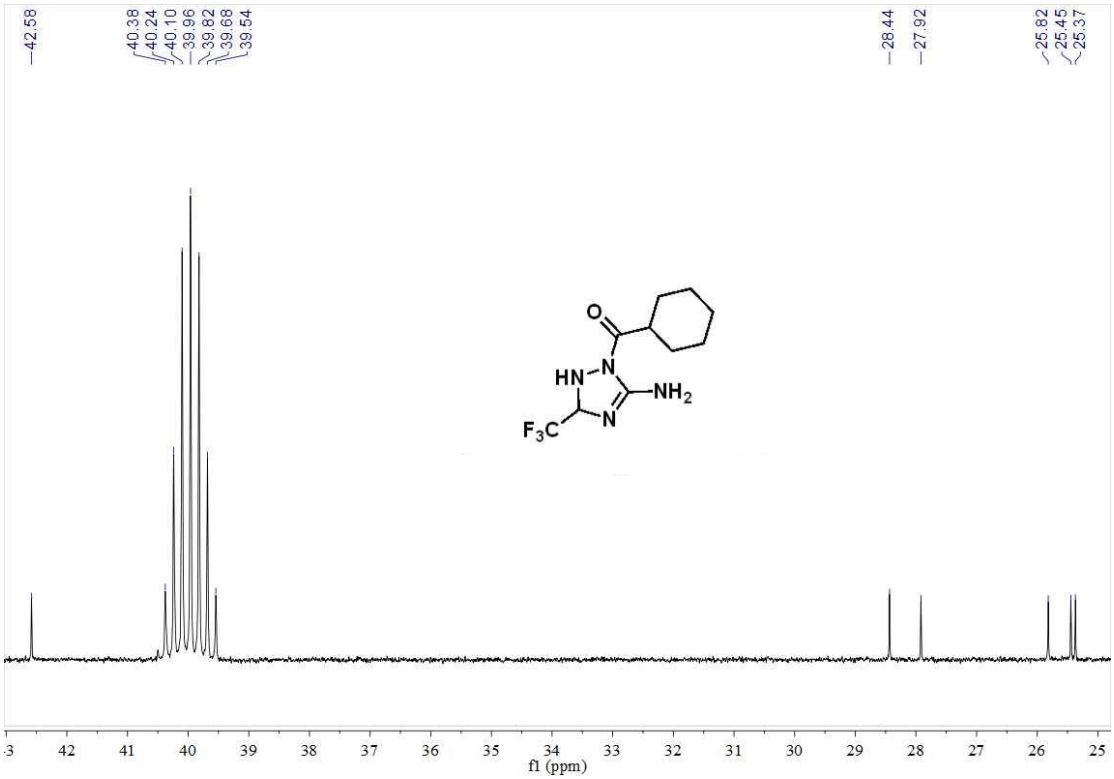


HRMS (ESI) copy of compound **2u**

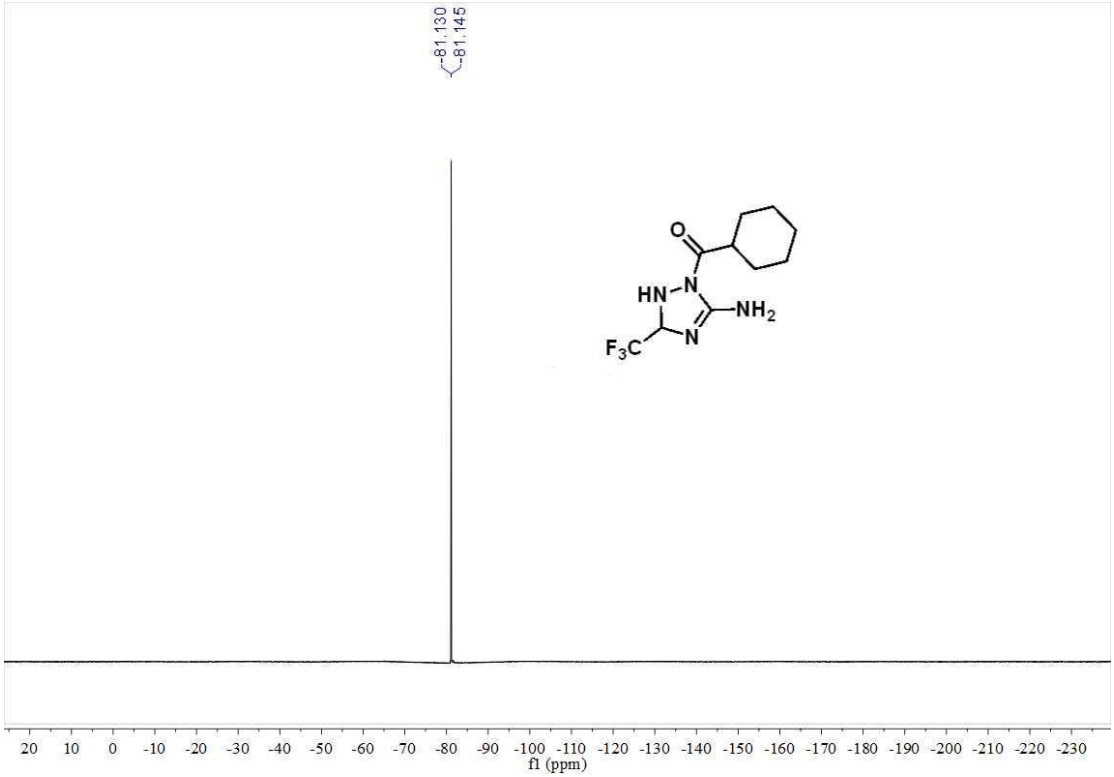


^{1}H NMR (400 MHz) spectrum of **2v** in DMSO-*d*$_6$





$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **2v** in DMSO-*d*$_6$



^{19}F NMR (376 MHz) spectrum of **2v** in DMSO-*d*$_6$

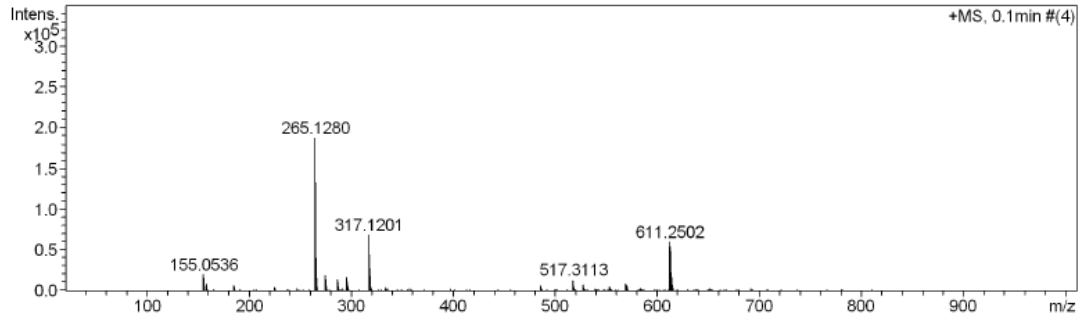
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\user\liuxoiaoling0210623-17.d	Acquisition Date	2021-6-23 11:15:06
Method	tune_low.m	Operator	BDAL@DE
Sample Name	1r	Instrument / Ser#	micrOTOF-Q 20453
Comment			

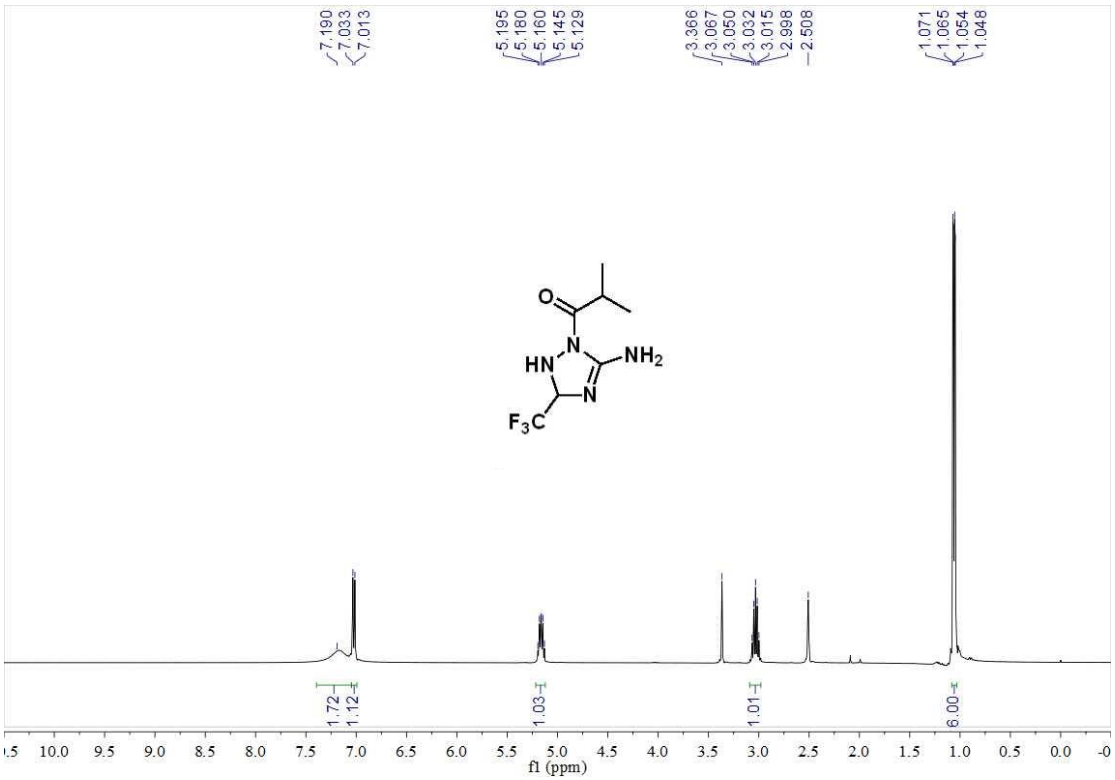
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 ¡ãC
Scan Begin	21 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste

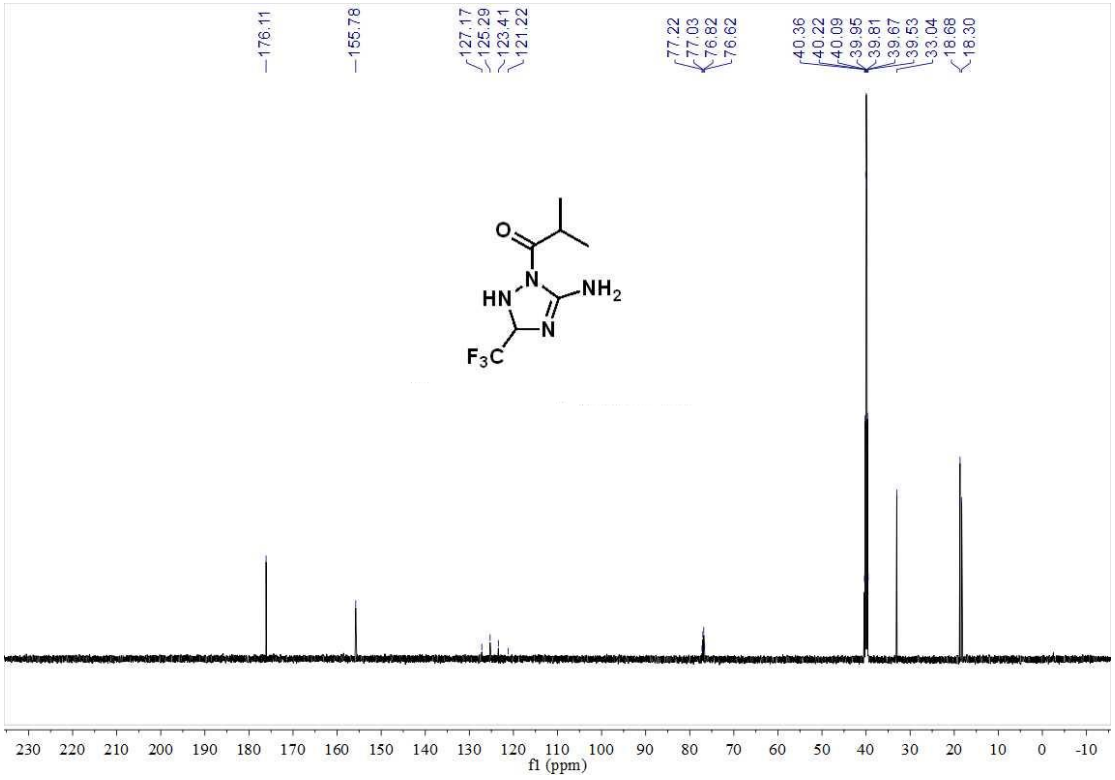


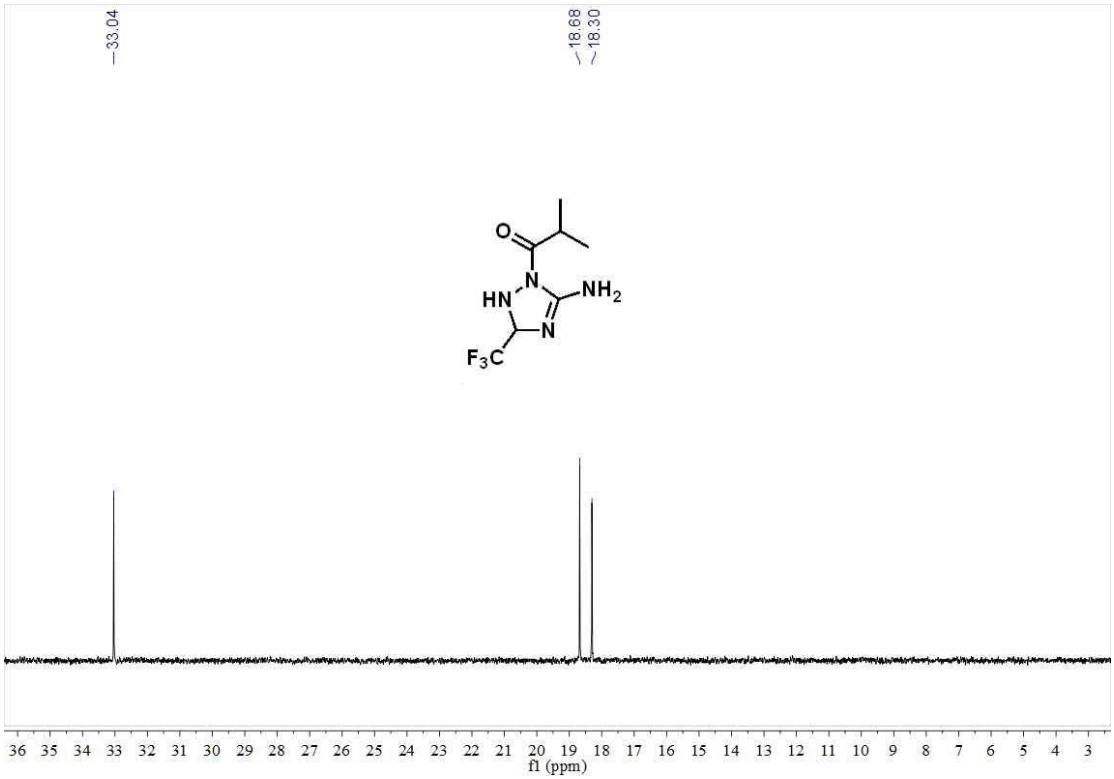
Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma	Std I	Std Mean m/z	Std I Var Norm	Std m/z Diff	Std Comb Dev
265.1280	1	C 10 H 16 F 3 N 4 O	265.1271	-3.6	-2.9	3.5	ok	even	4.2	6.8	0.9	5.1	1.8	842.7

HRMS (ESI) copy of compound **2v**

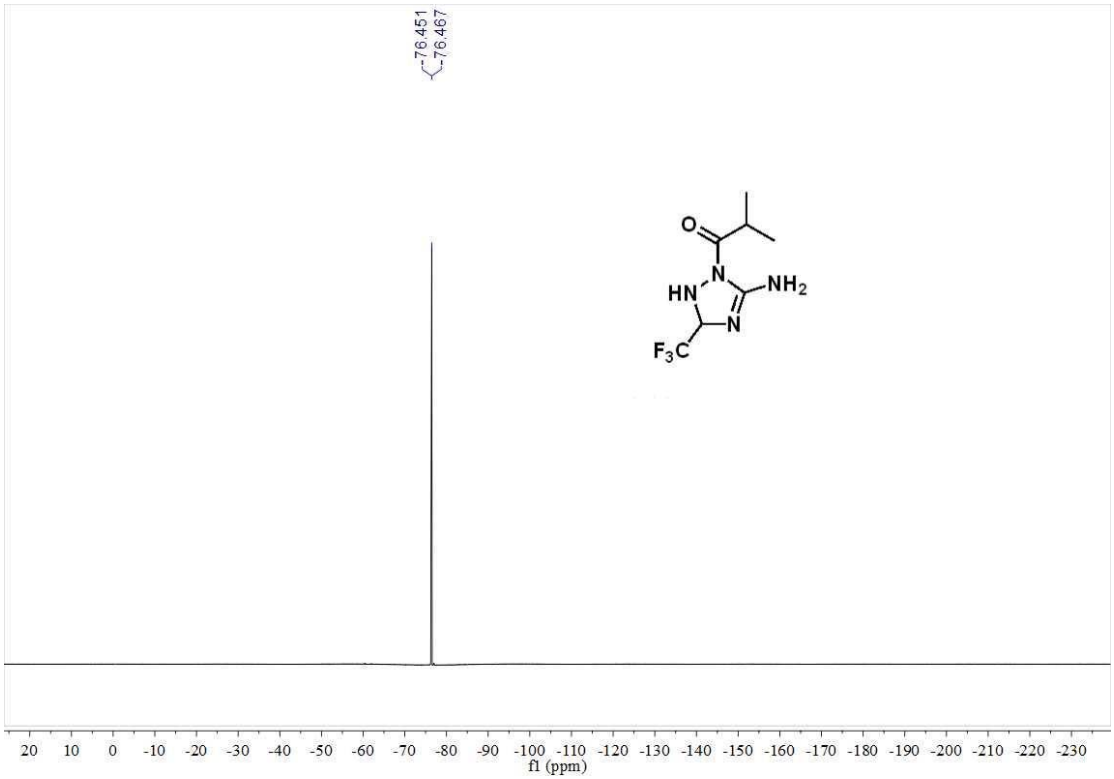


^{1}H NMR (400 MHz) spectrum of **2w** in DMSO-*d*$_6$





$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **2w** in DMSO-*d*$_6$



^{19}F NMR (376 MHz) spectrum of **2w** in DMSO-*d*$_6$

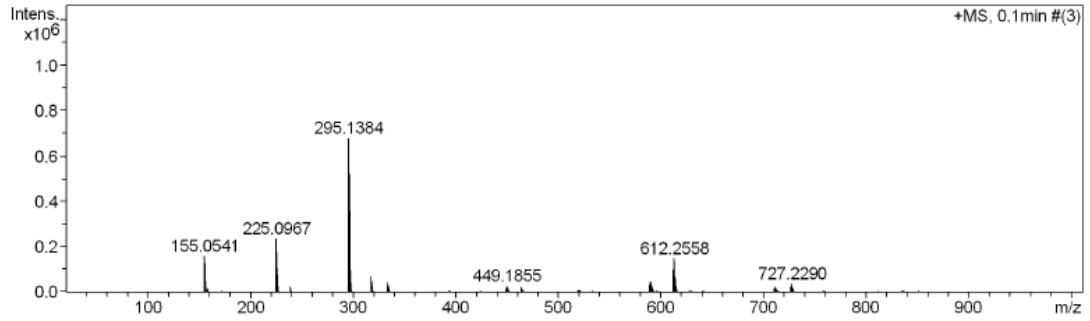
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\user\liuxoiaoling0210623-16.d	Acquisition Date	2021-6-23 11:13:00
Method	tune_low.m	Operator	BDAL@DE
Sample Name	1q	Instrument / Ser#	micrOTOF-Q 20453
Comment			

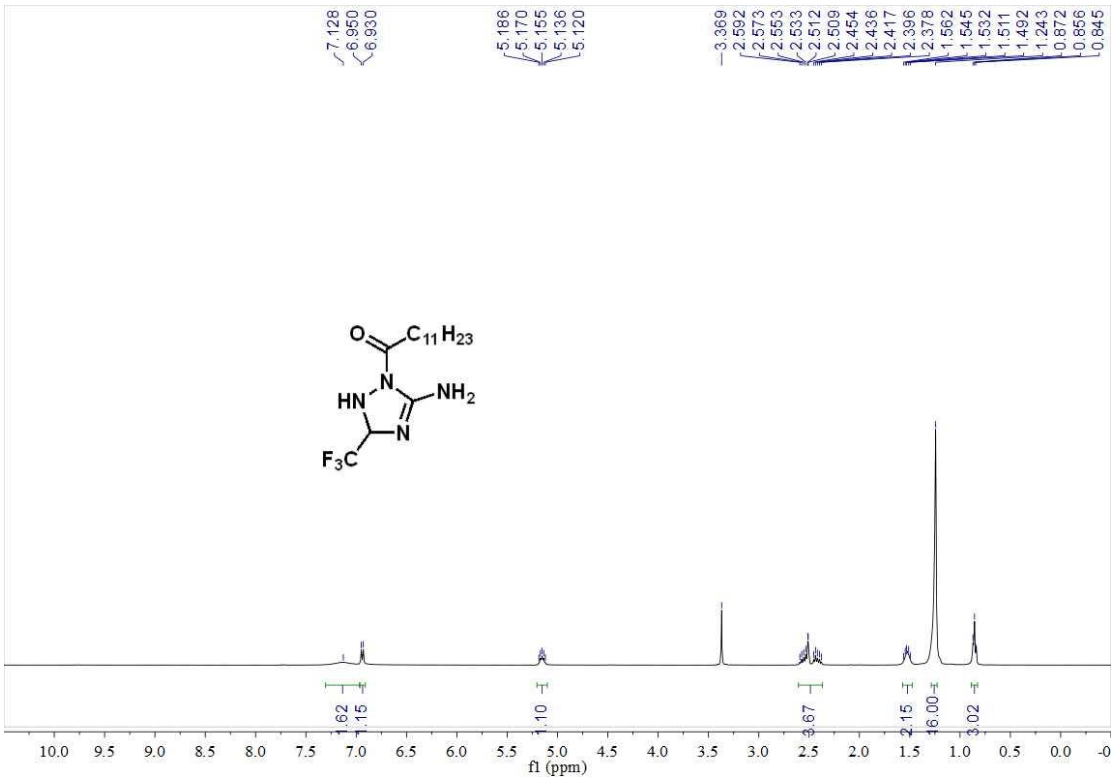
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 ¡ãC
Scan Begin	21 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste

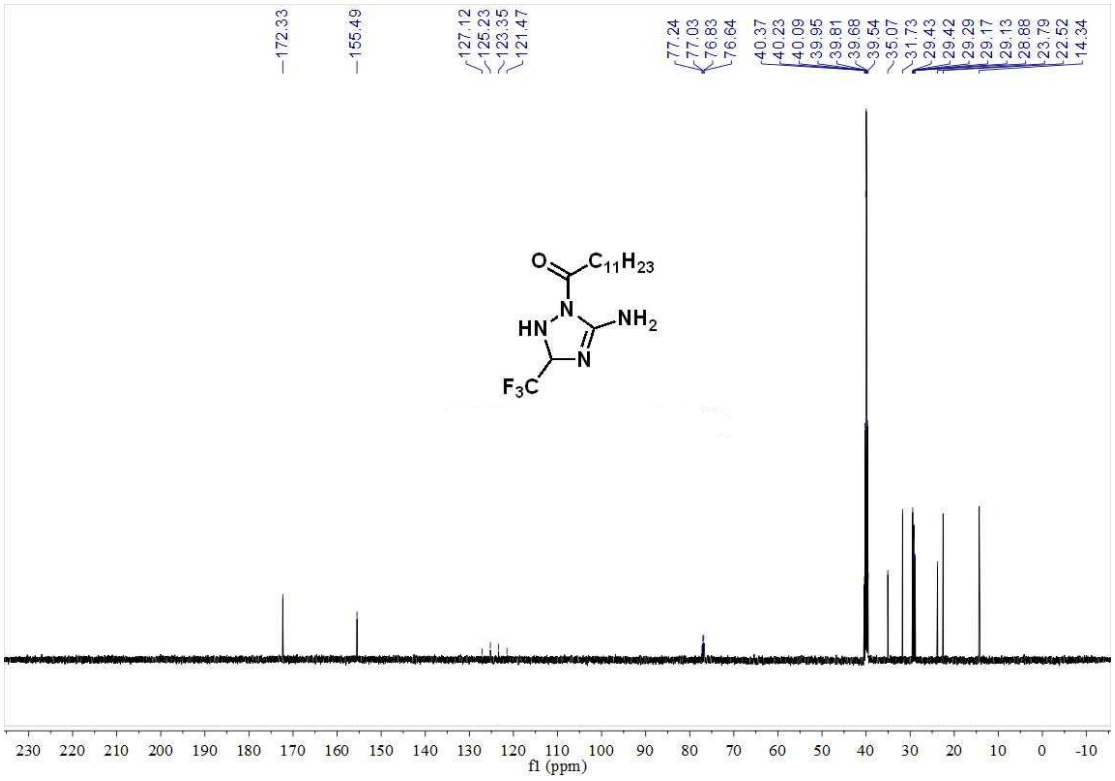


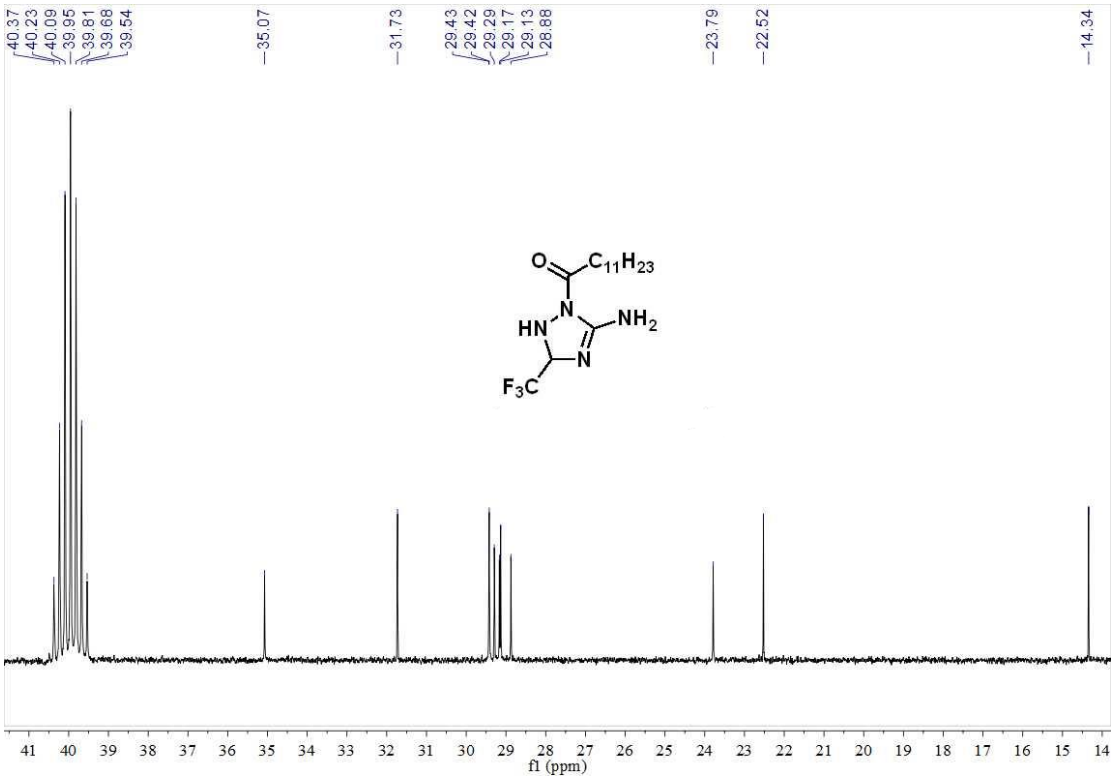
Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma	Std I	Std Mean m/z	Std I VarNorm	Std m/z Diff	Std Comb Dev
225.0967	1	C 7 H 12 F 3 N 4 O	225.0958	-4.3	-7.5	2.5	ok	even	204.9	327.9	2.0	138.9	2.1	842.7

HRMS (ESI) copy of compound **2w**

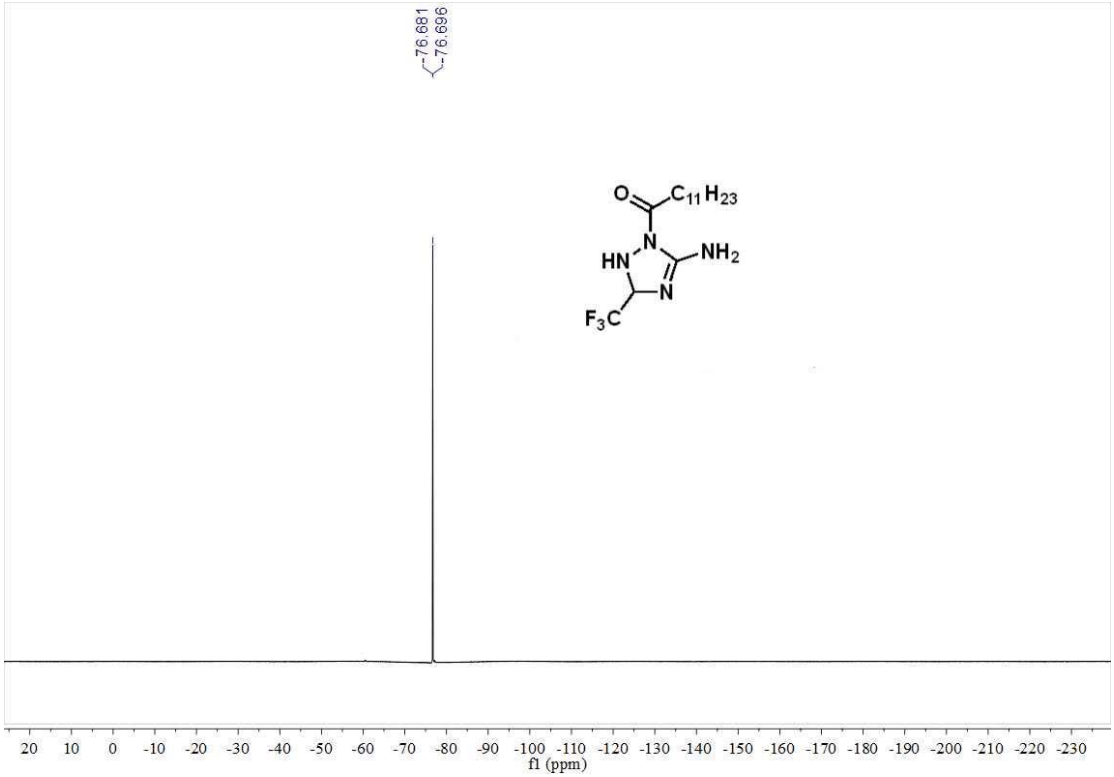


^{1}H NMR (400 MHz) spectrum of **2x** in DMSO-d6





$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **2x** in DMSO-*d*$_6$



^{19}F NMR (376 MHz) spectrum of **2x** in DMSO-*d*$_6$

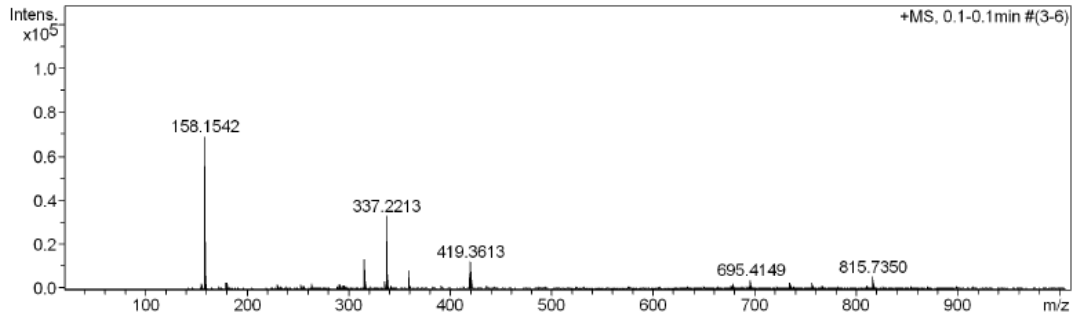
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\user\liulixoling20210623-15.d	Acquisition Date	2021-6-23 11:08:02
Method	tune_low.m	Operator	BDAL@DE
Sample Name	1p	Instrument / Ser#	micrOTOF-Q 20453
Comment			

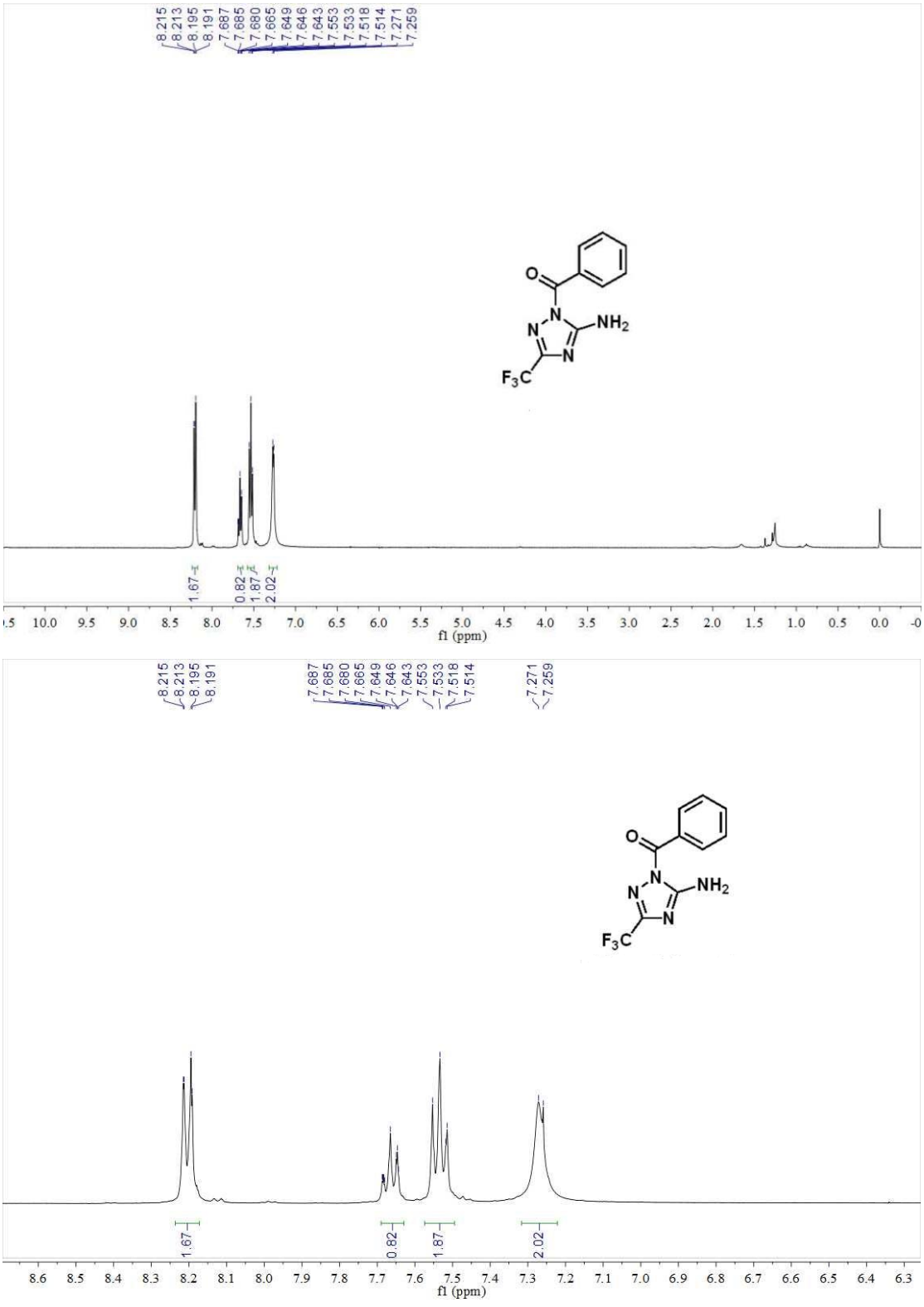
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 ¡ãC
Scan Begin	21 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste

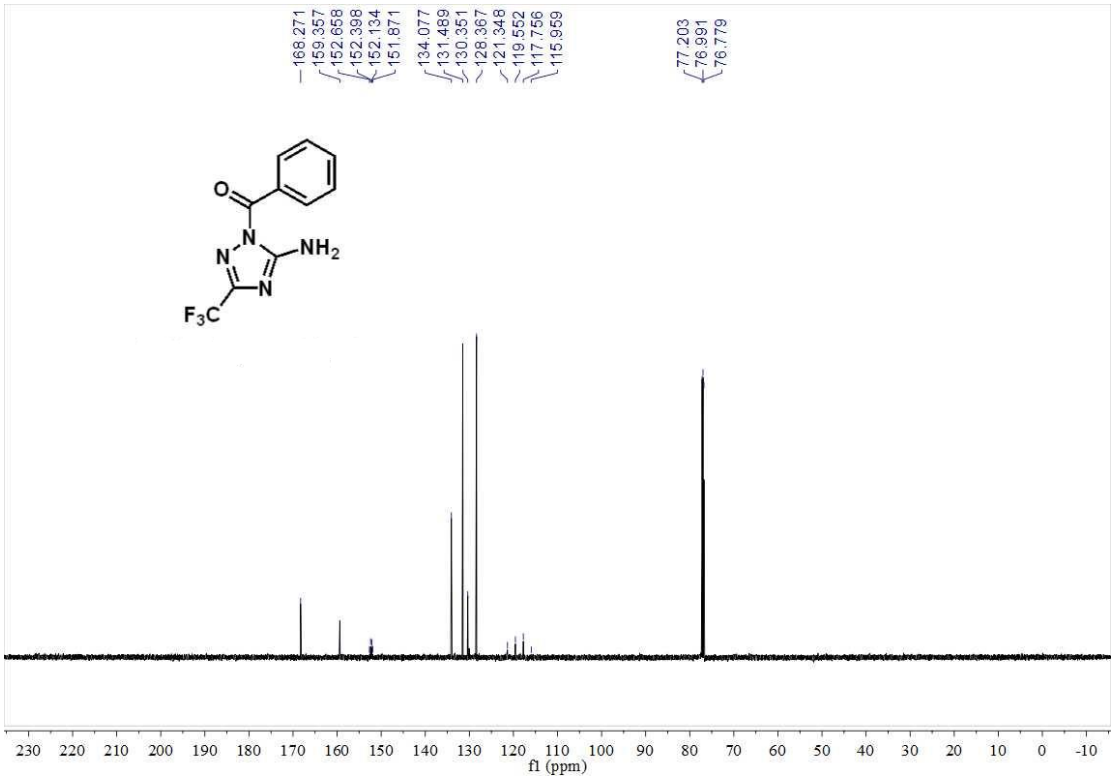


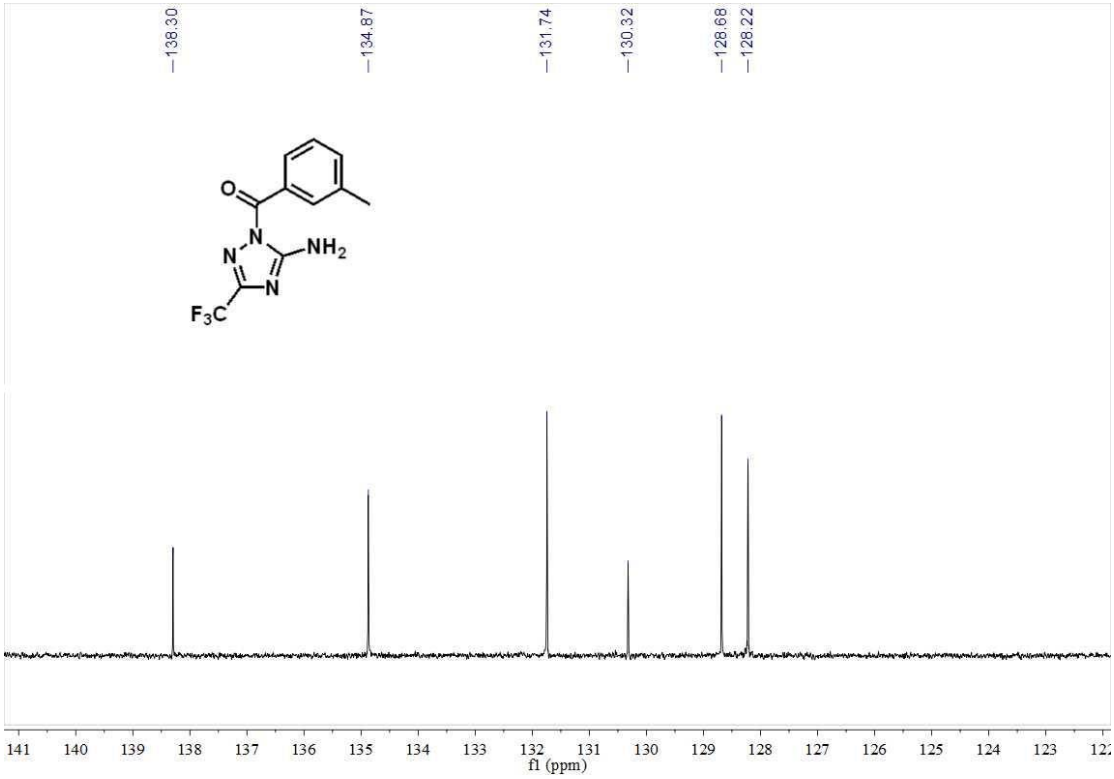
Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	e¡¥ Conf	mSigma	Std I	Std Mean m/z	Std I Var Norm	Std m/z Diff	Std Comb Dev
337.2213	1	C 15 H 28 F 3 N 4 O	337.2210	-0.8	-0.9	2.5	ok	even	12.4	23.7	0.5	9.7	0.4	842.7

HRMS (ESI) copy of compound **2x**

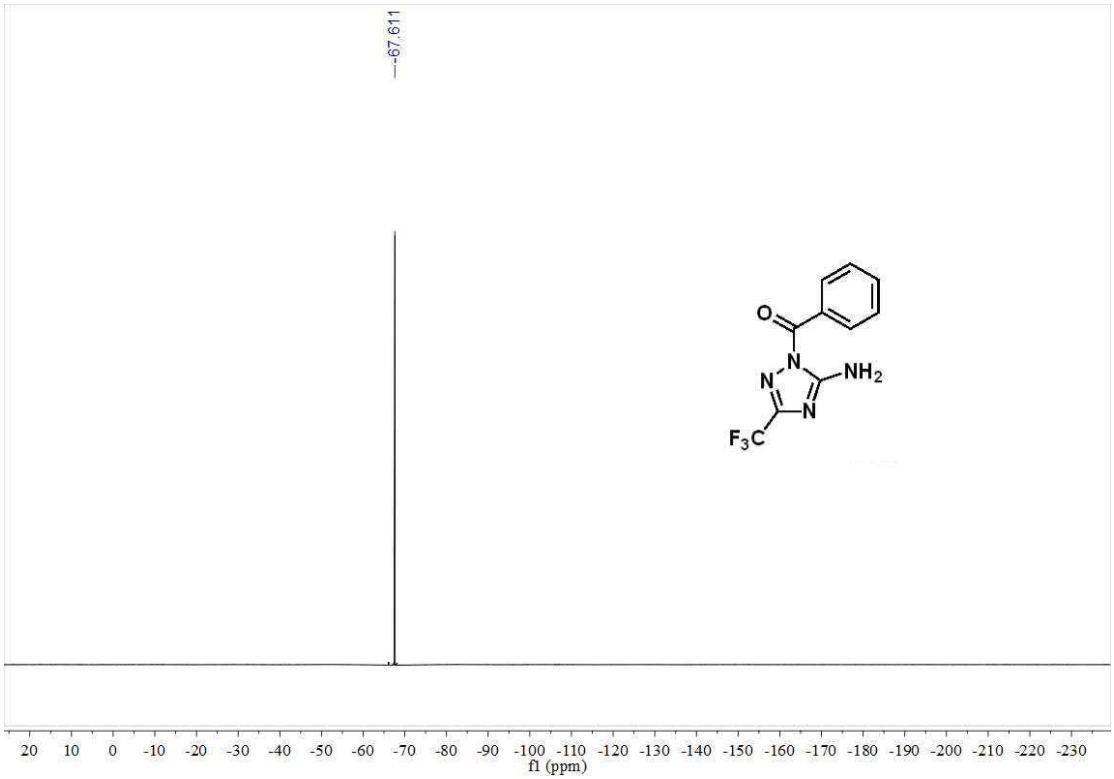


^{1}H NMR (400 MHz) spectrum of **3a** in $CDCl_3$

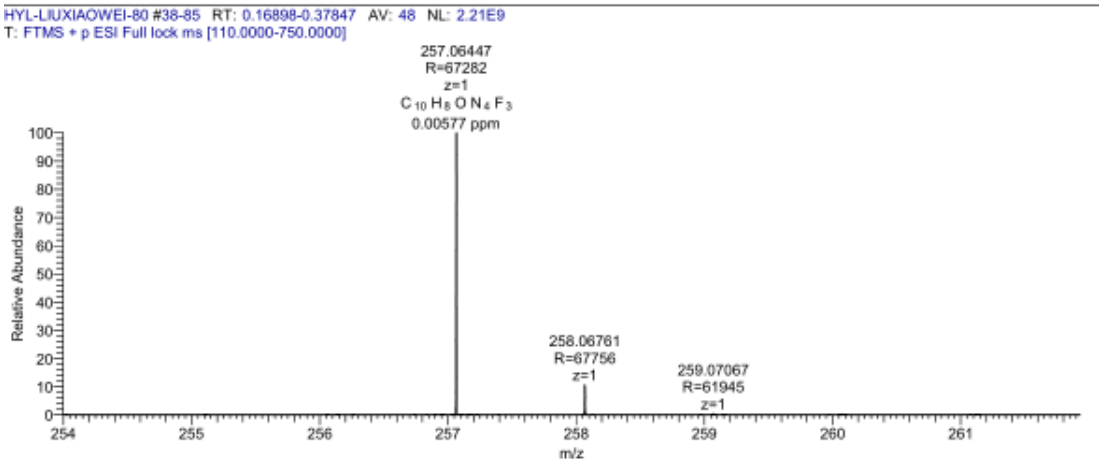




$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **3a** in $CDCl_3$

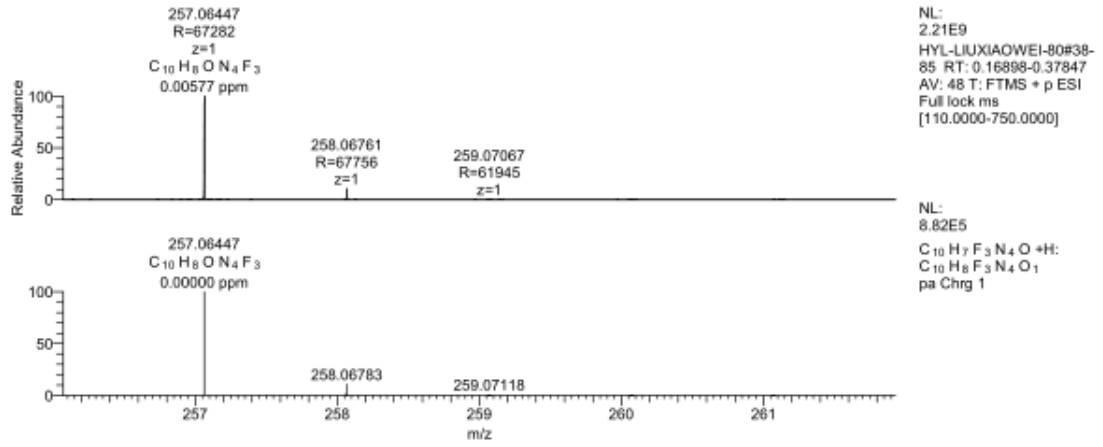


^{19}F NMR (376 MHz) spectrum of **3a** in $CDCl_3$

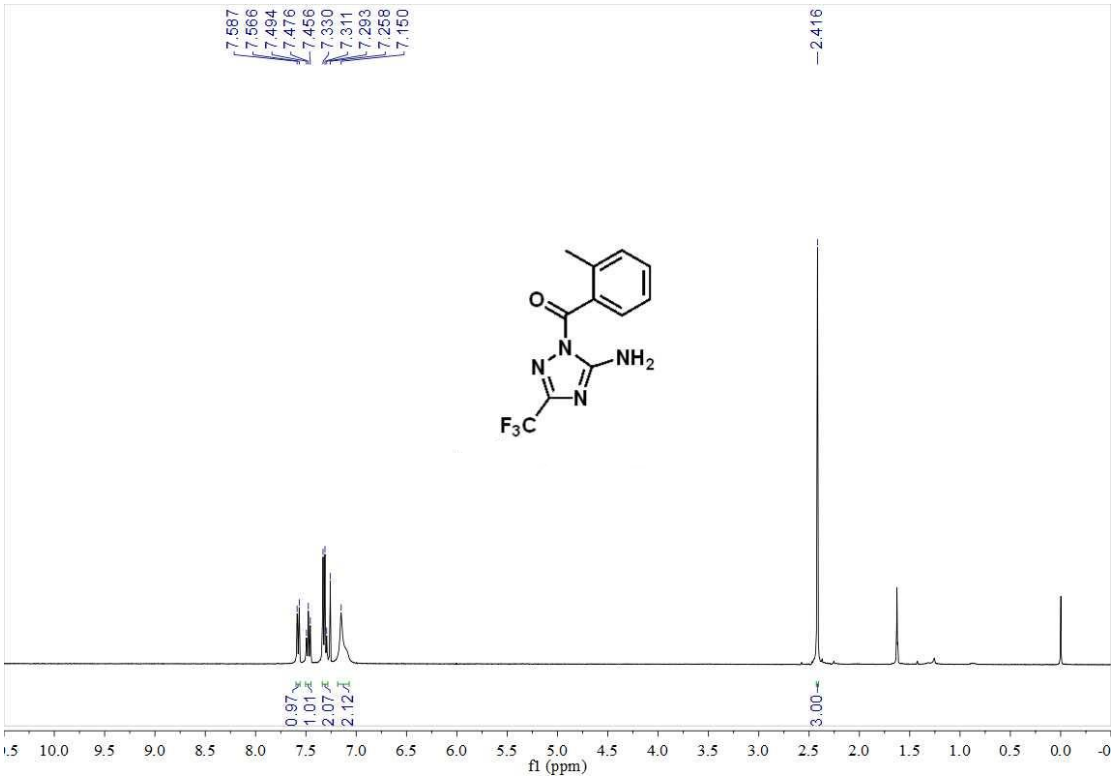


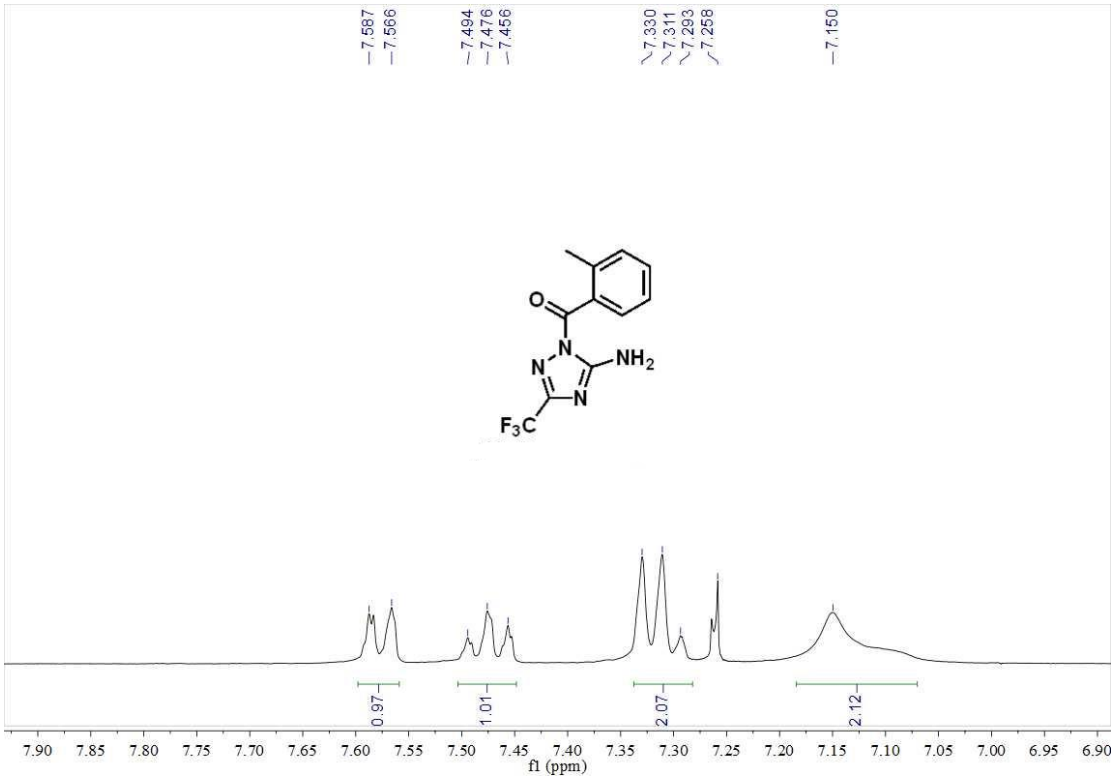
HYL-LIUXIAOWEI-80#38-85 RT: 0.16898-0.37847 AV: 48
T: FTMS + p ESI Full lock ms [110.0000-750.0000]
m/z= 253.99792-261.92977

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
257.06447	2242674944.0	100.00	67281.80	1.00	0.01	$C_{10}H_8ON_4F_3$
258.06157	26372470.0	1.18	75170.33	1.00		
258.06761	237931792.0	10.61	67756.16	1.00		
259.07067	11116520.0	0.50	61944.76	1.00		
260.07371	9195582.0	0.41	67951.39	1.00		

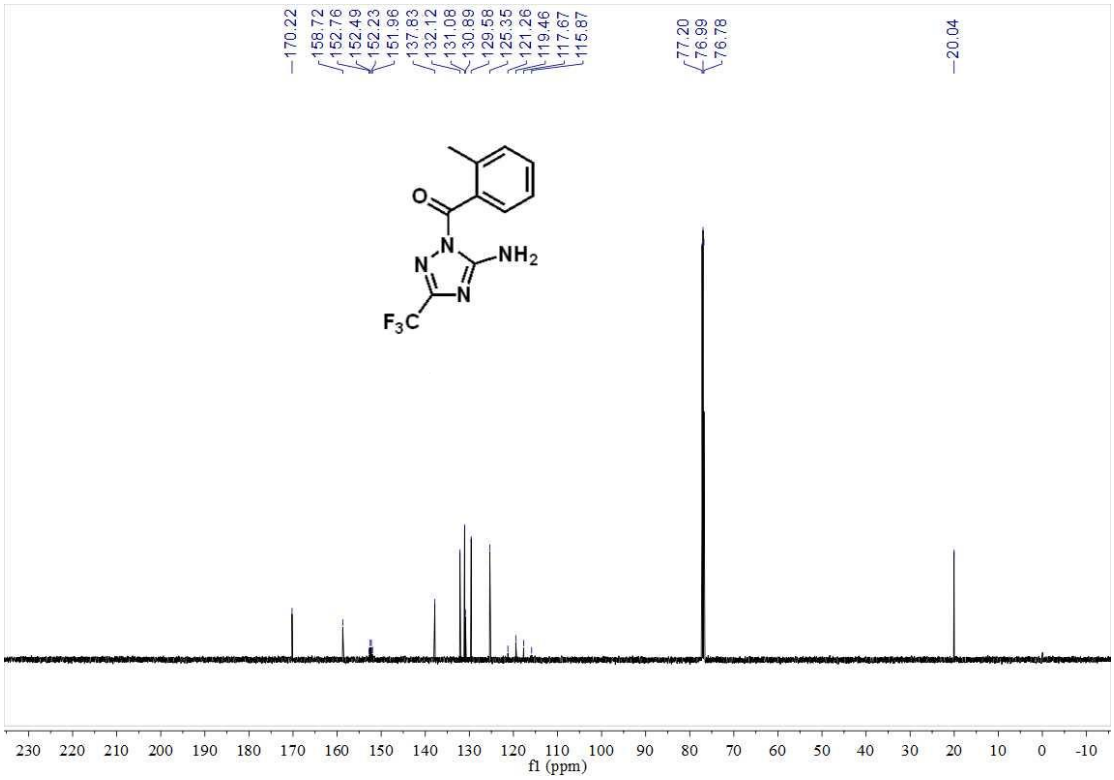


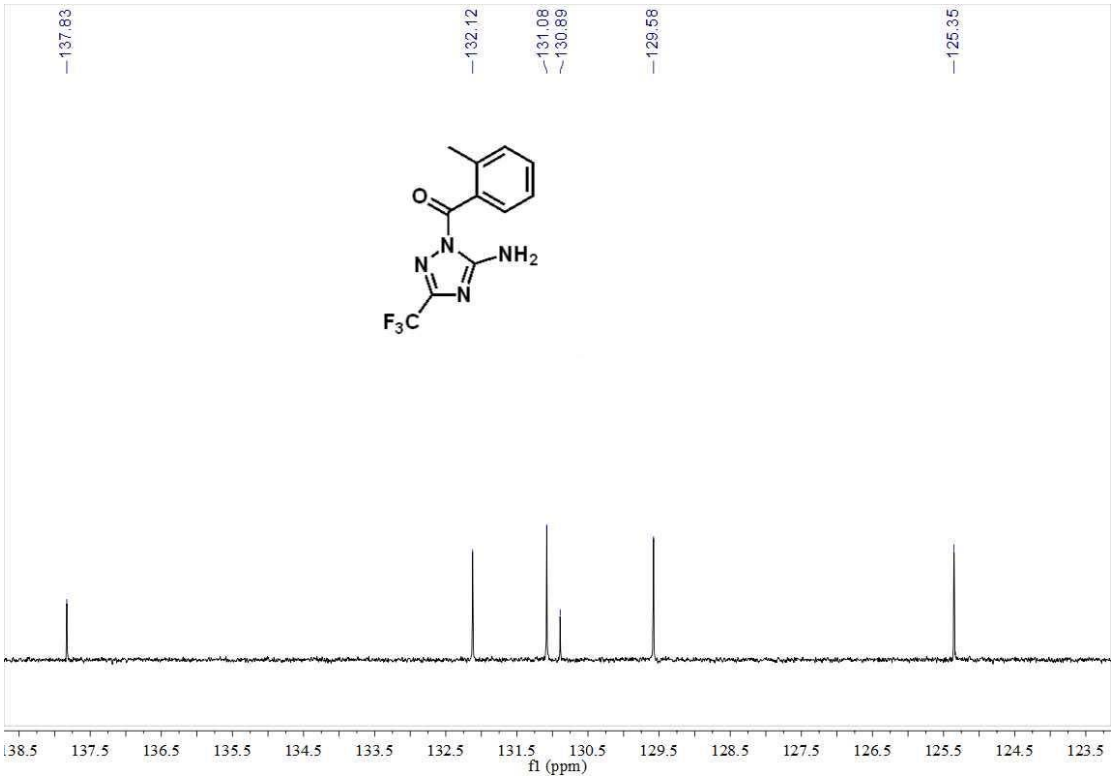
HRMS (ESI) copy of compound **3a**



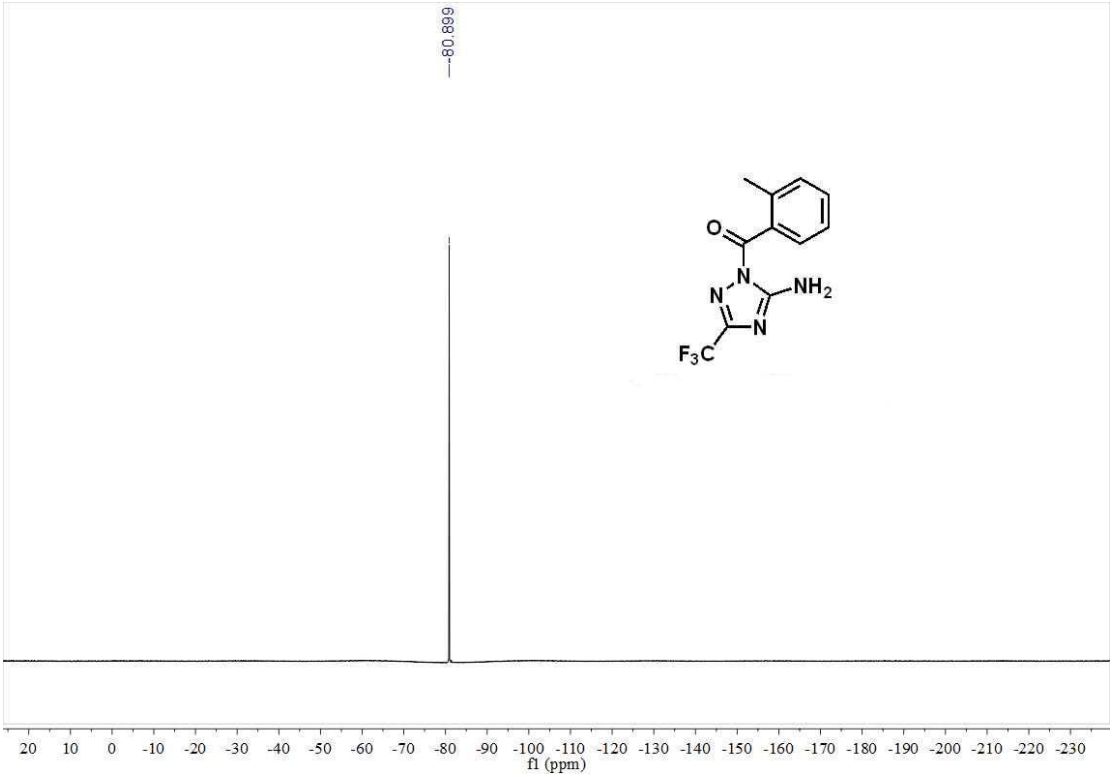


^{1}H NMR (400 MHz) spectrum of **3b** in $CDCl_3$





$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **3b** in $CDCl_3$



^{19}F NMR (376 MHz) spectrum of **3b** in $CDCl_3$

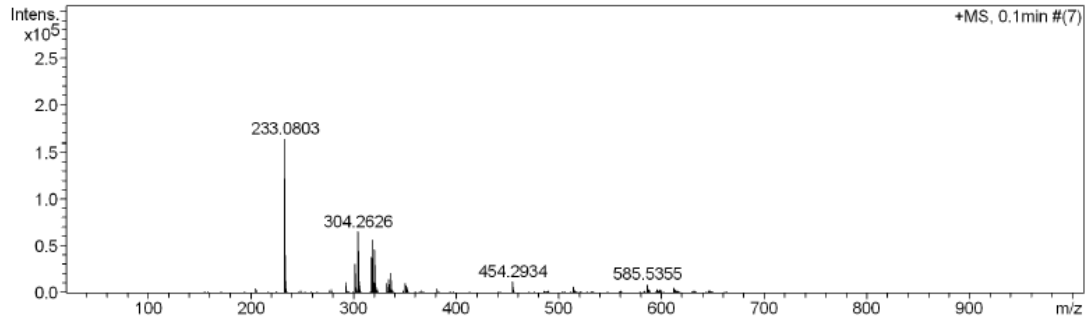
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\user\liuxiaoling0210623-18.d	Acquisition Date	2021-6-23 11:17:32
Method	tune_low.m		
Sample Name	2b	Operator	BDAL@DE
Comment		Instrument / Ser#	micrOTOF-Q 20453

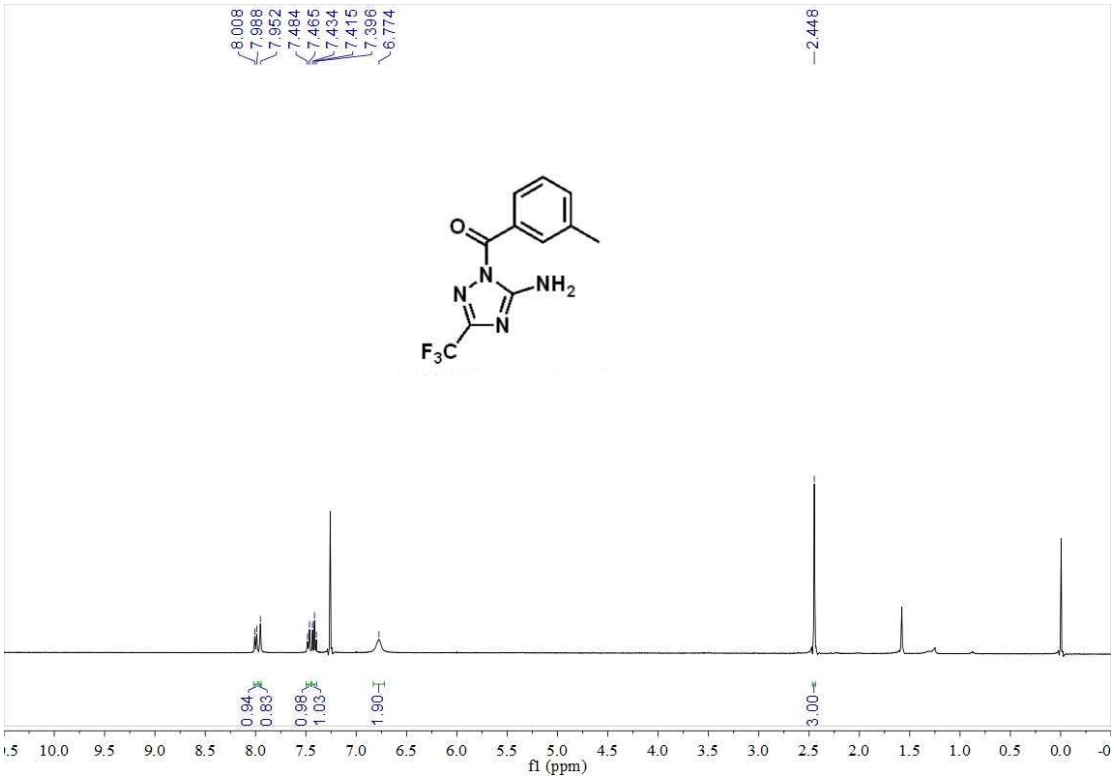
Acquisition Parameter

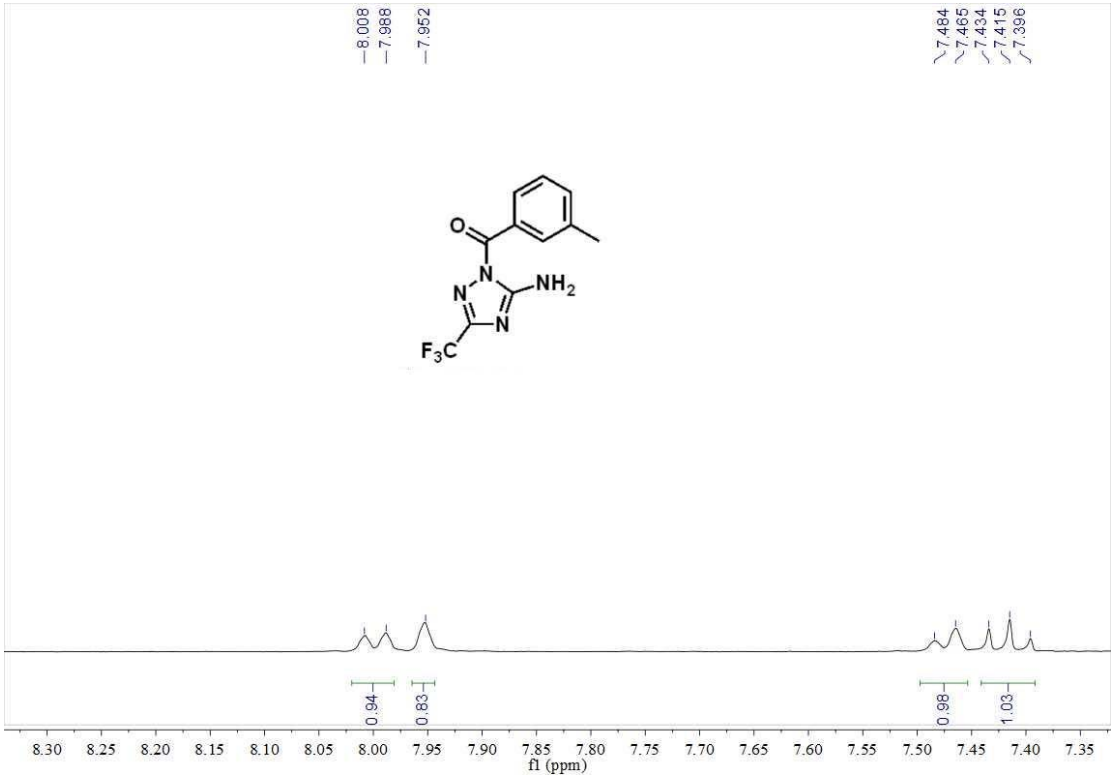
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 ¡ãC
Scan Begin	21 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste



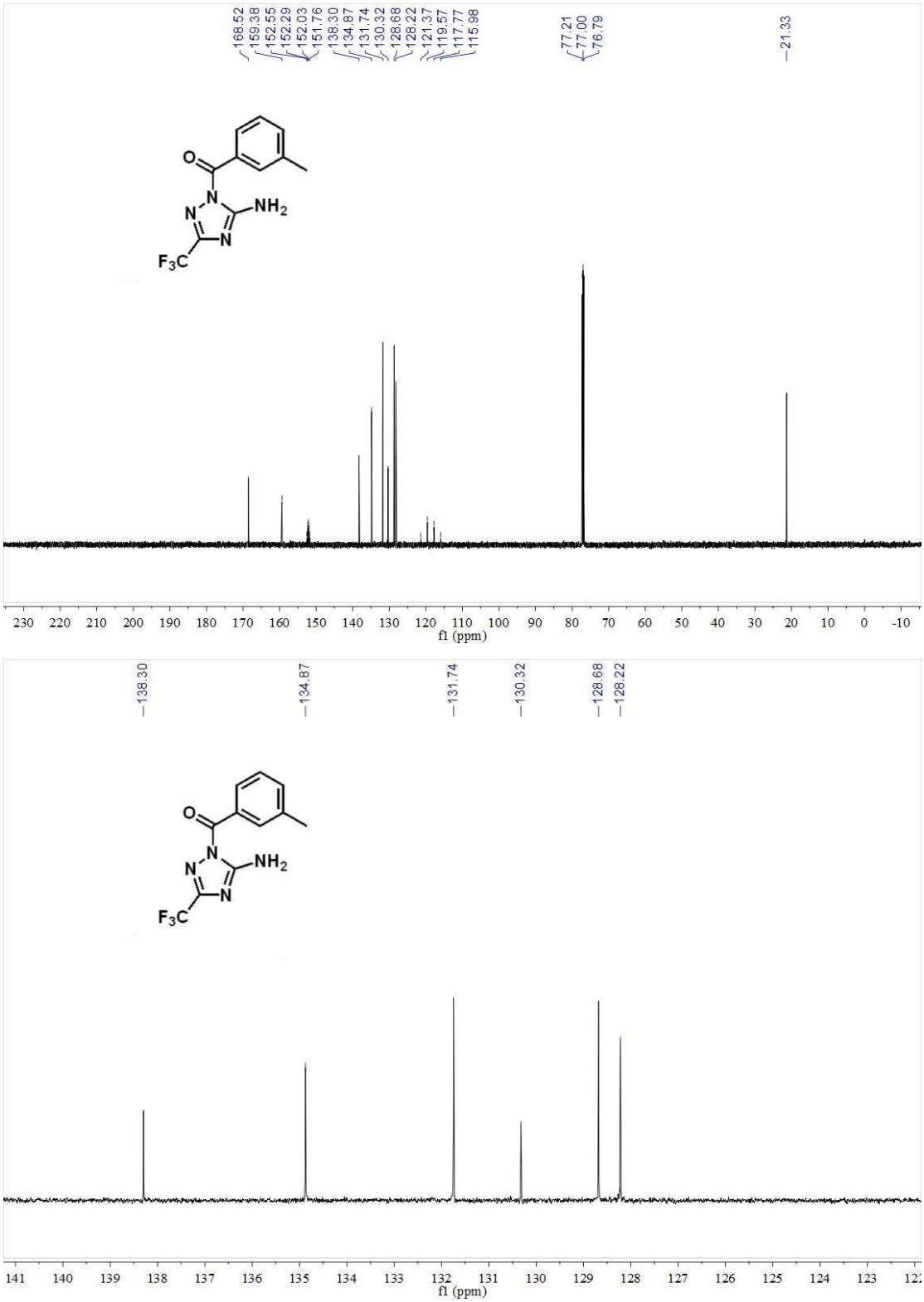
Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma	Std I	Std Mean m/z	Std I Var Norm	Std m/z Diff	Std Comb Dev
293.0628	1	C 11 H 9 F 3 N 4 Na O	293.0621	-2.4	-10.6	7.5	ok	even	88.8	161.9	7.5	99.7	1.2	842.7

HRMS (ESI) copy of compound **3b**

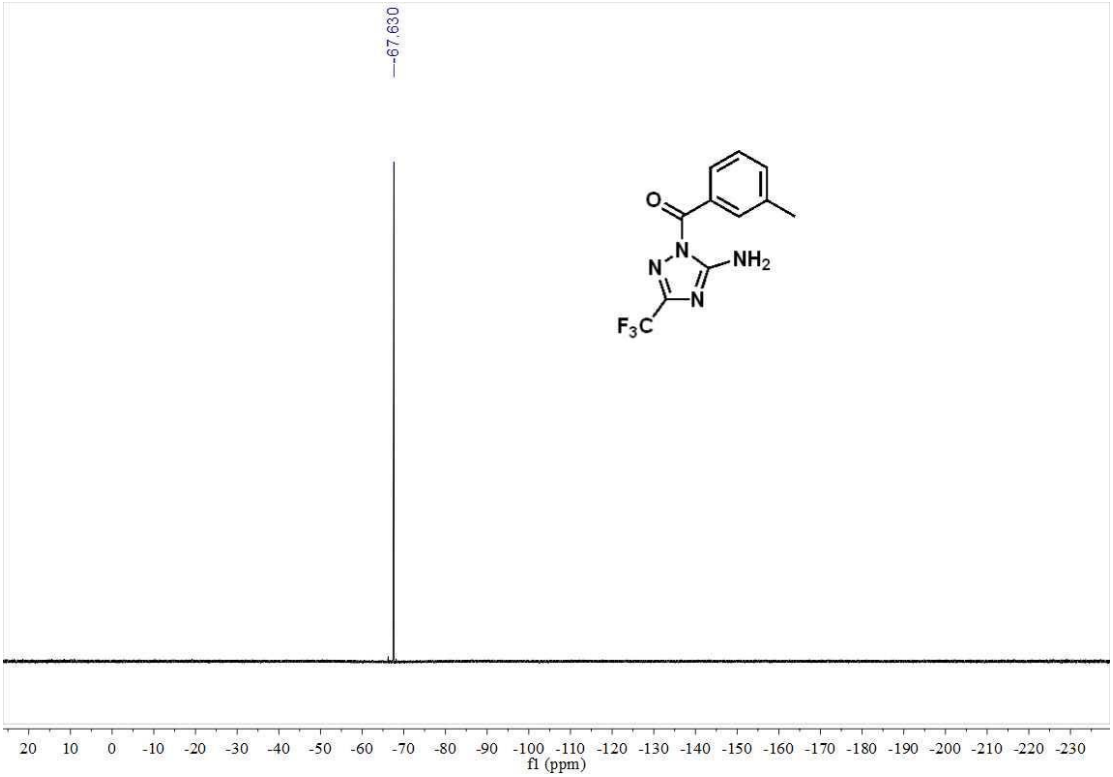




^{1}H NMR (400 MHz) spectrum of **3c** in $CDCl_3$



$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **3c** in $CDCl_3$



^{19}F NMR (376 MHz) spectrum of **3c** in $CDCl_3$

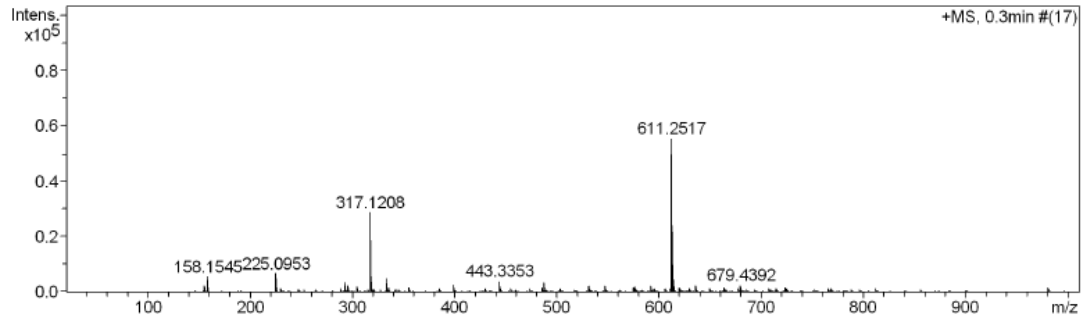
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\user\liuxiaoling0210623-19.d	Acquisition Date	2021-6-23 11:19:53
Method	tune_low.m		
Sample Name	2c	Operator	BDAL@DE
Comment		Instrument / Ser#	micrOTOF-Q 20453

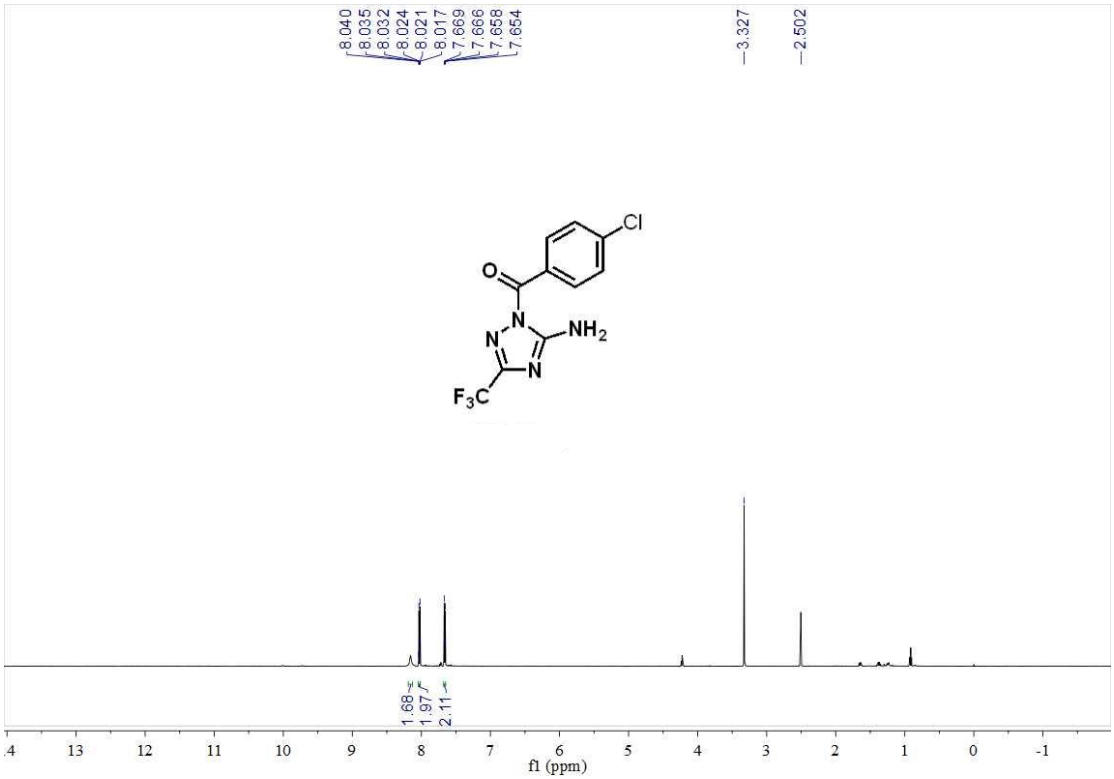
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 ¡ãC
Scan Begin	21 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste

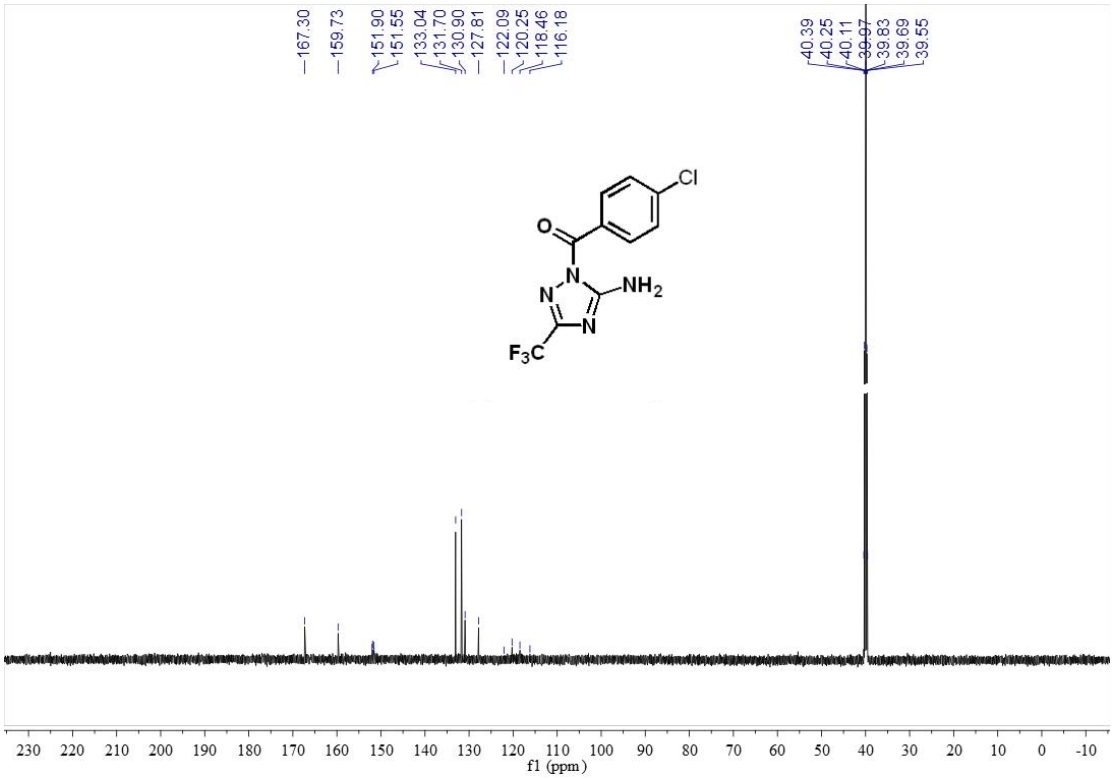


Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	ej¥ Conf	mSigma	Std I	Std Mean m/z	Std I Var Norm	Std m/z Diff	Std Comb Dev
293.0632	1	C 11 H 9 F 3 N 4 Na O	293.0621	-4.0	-4.0	7.5	ok	even	77.4	133.7	1.2	67.4	1.2	842.7

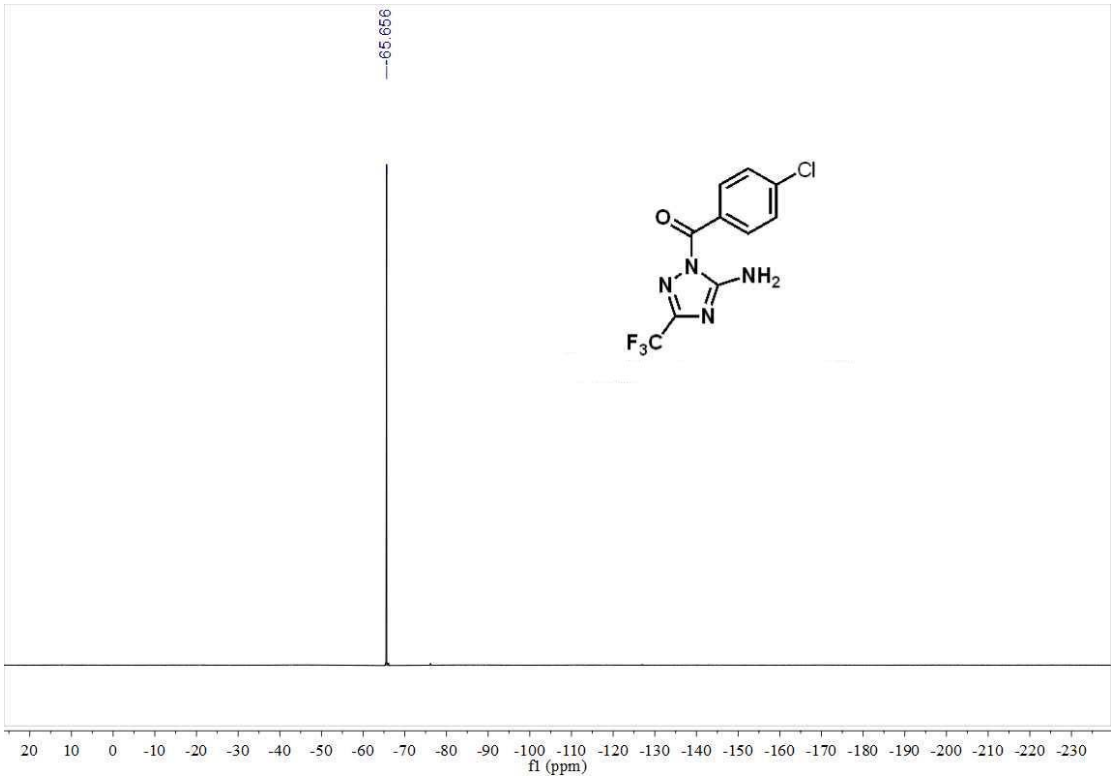
HRMS (ESI) copy of compound **3c**



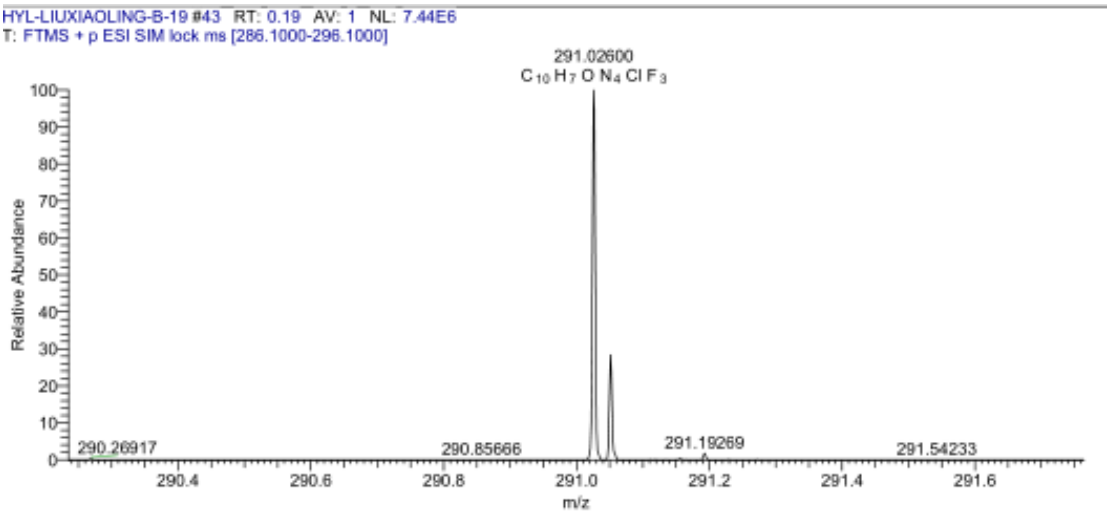
^{1}H NMR (400 MHz) spectrum of **3d** in DMSO-*d*$_6$



^{13}C{^{1}H} NMR (150 MHz) spectrum of **3d** in DMSO-*d*$_6$

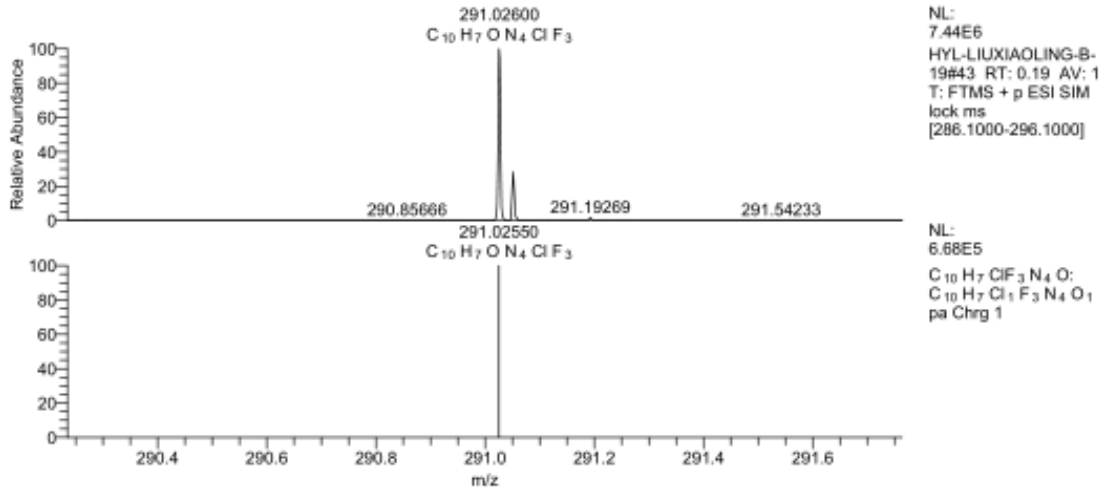


^{19}F NMR (376 MHz) spectrum of **3d** in DMSO-d_6

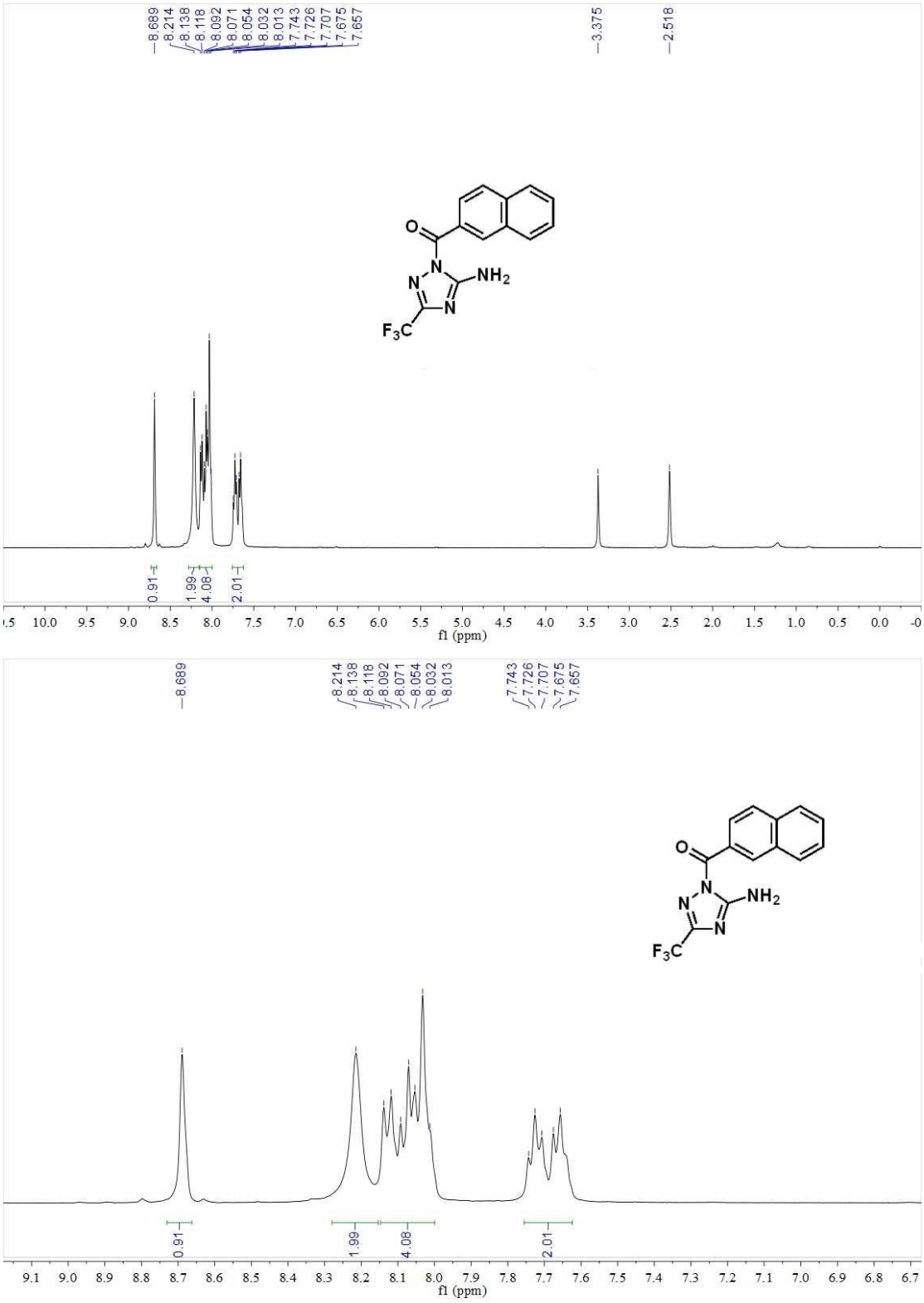


HYL-LIUXIAOLING-B-19#43 RT: 0.19
T: FTMS + p ESI SIM lock ms [286.1000-296.1000]
m/z= 290.23481-291.76399

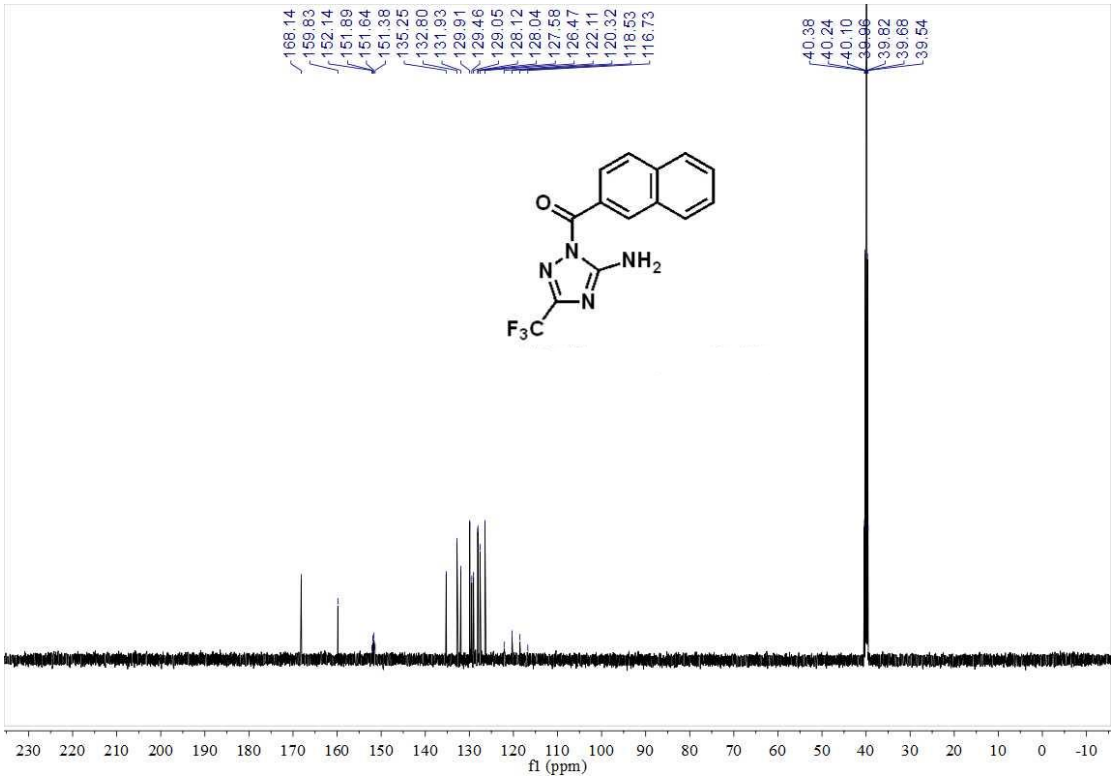
m/z	Intensity	Relative	Delta (ppm)	Composition
290.26917	27174.2	0.35		
291.02600	7778287.0	100.00	0.50	$C_{10}H_7ON_4ClF_3$
291.05139	2135044.3	27.45	25.89	$C_{10}H_7ON_4ClF_3$
291.15604	25543.4	0.33	130.54	$C_{10}H_7ON_4ClF_3$
291.19269	133103.1	1.71	167.19	$C_{10}H_7ON_4ClF_3$

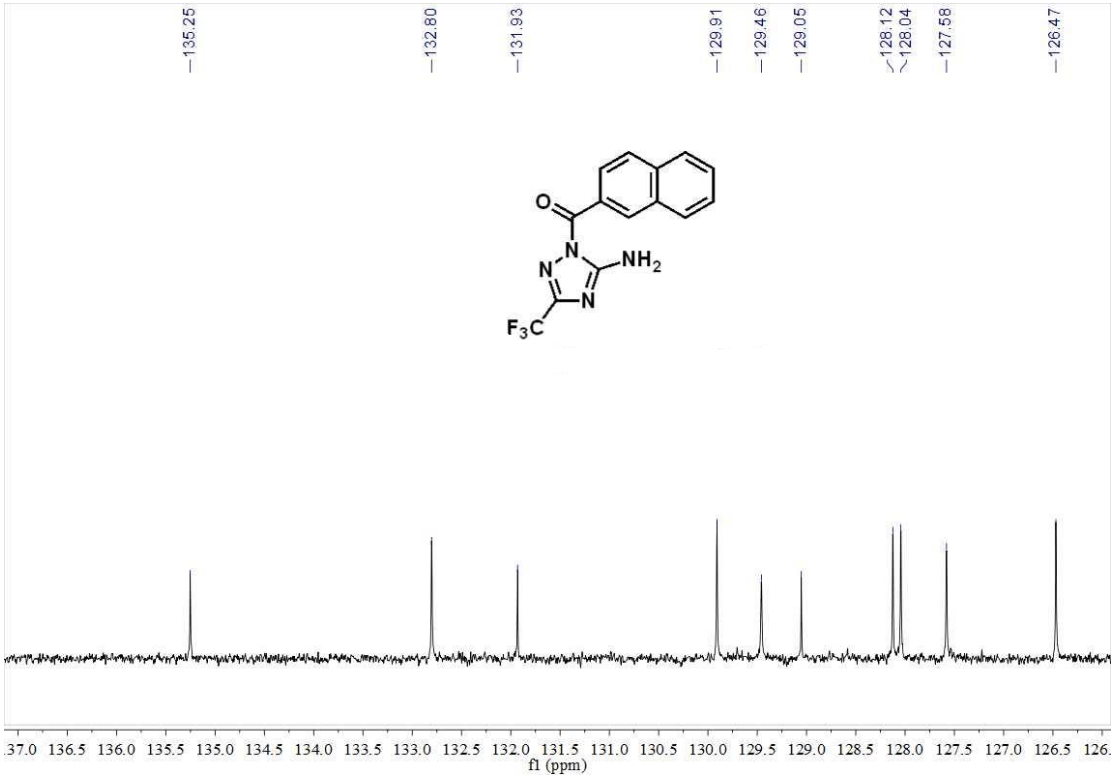


HRMS (ESI) copy of compound **3d**

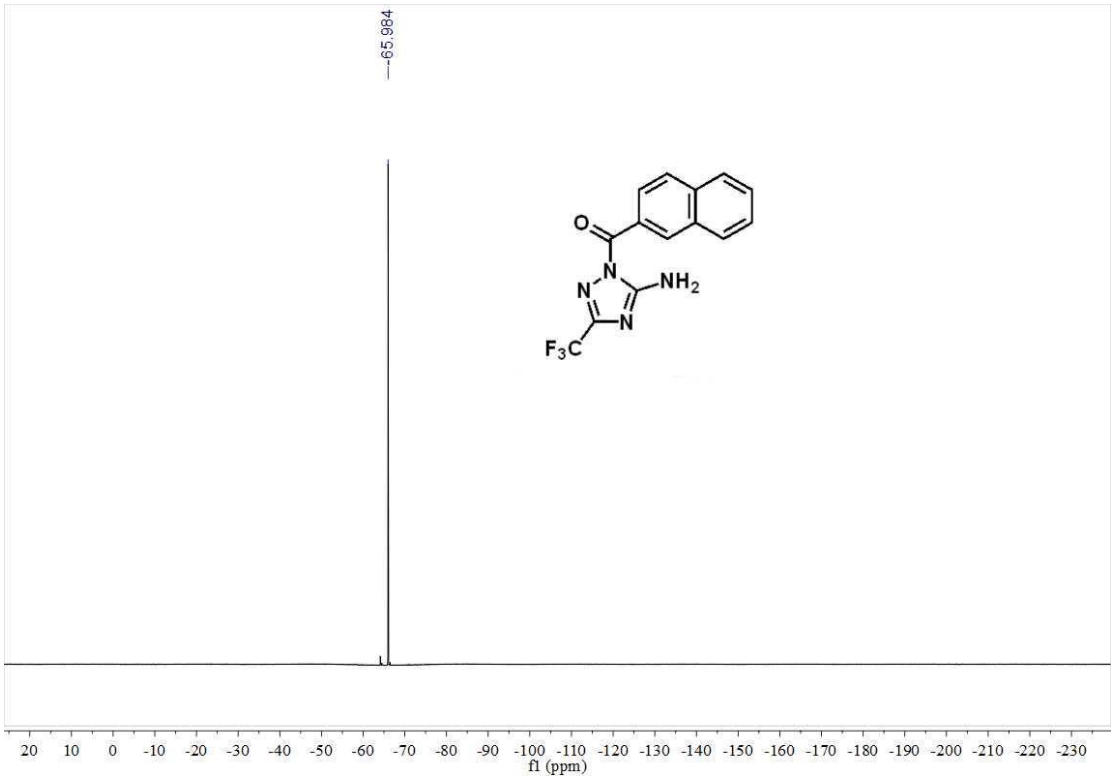


^{1}H NMR (400 MHz) spectrum of **3e** in DMSO-*d*$_6$

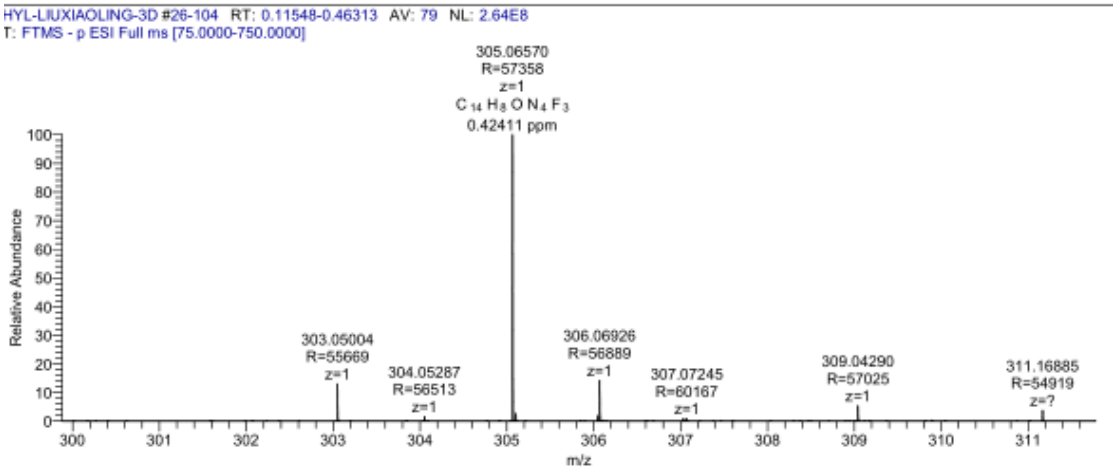




$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **3e** in DMSO-*d*$_6$

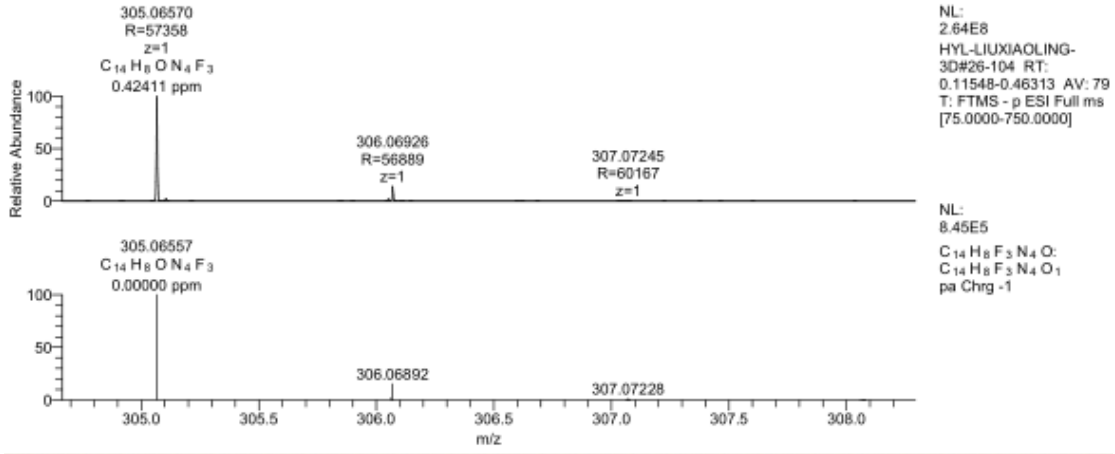


^{19}F NMR (376 MHz) spectrum of **3e** in DMSO-d_6

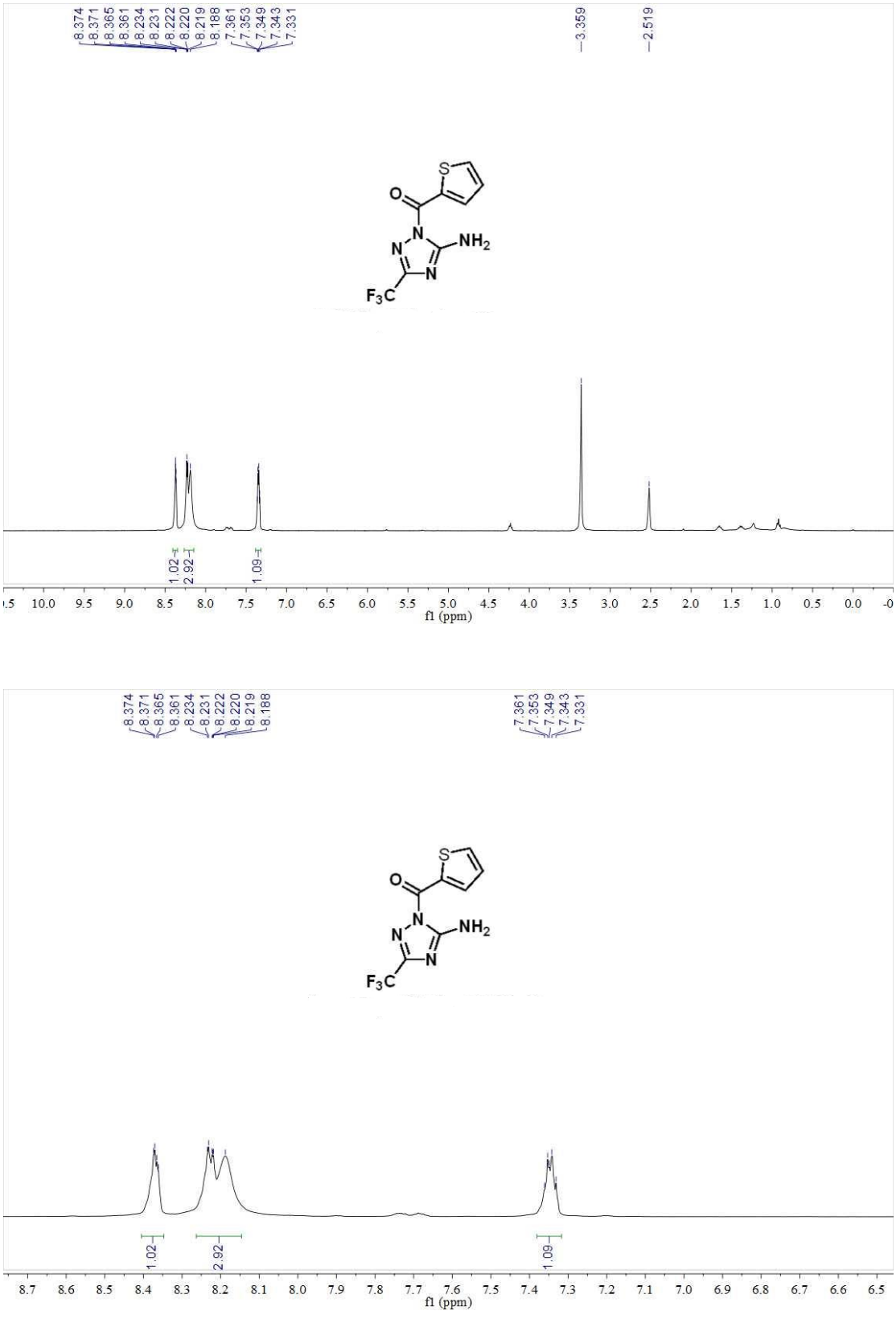


HYL-LIUXIAOLING-3D#26-104 RT: 0.11548-0.46313 AV: 79
T: FTMS - p ESI Full ms [75.0000-750.0000]
m/z= 299.88065-311.77811

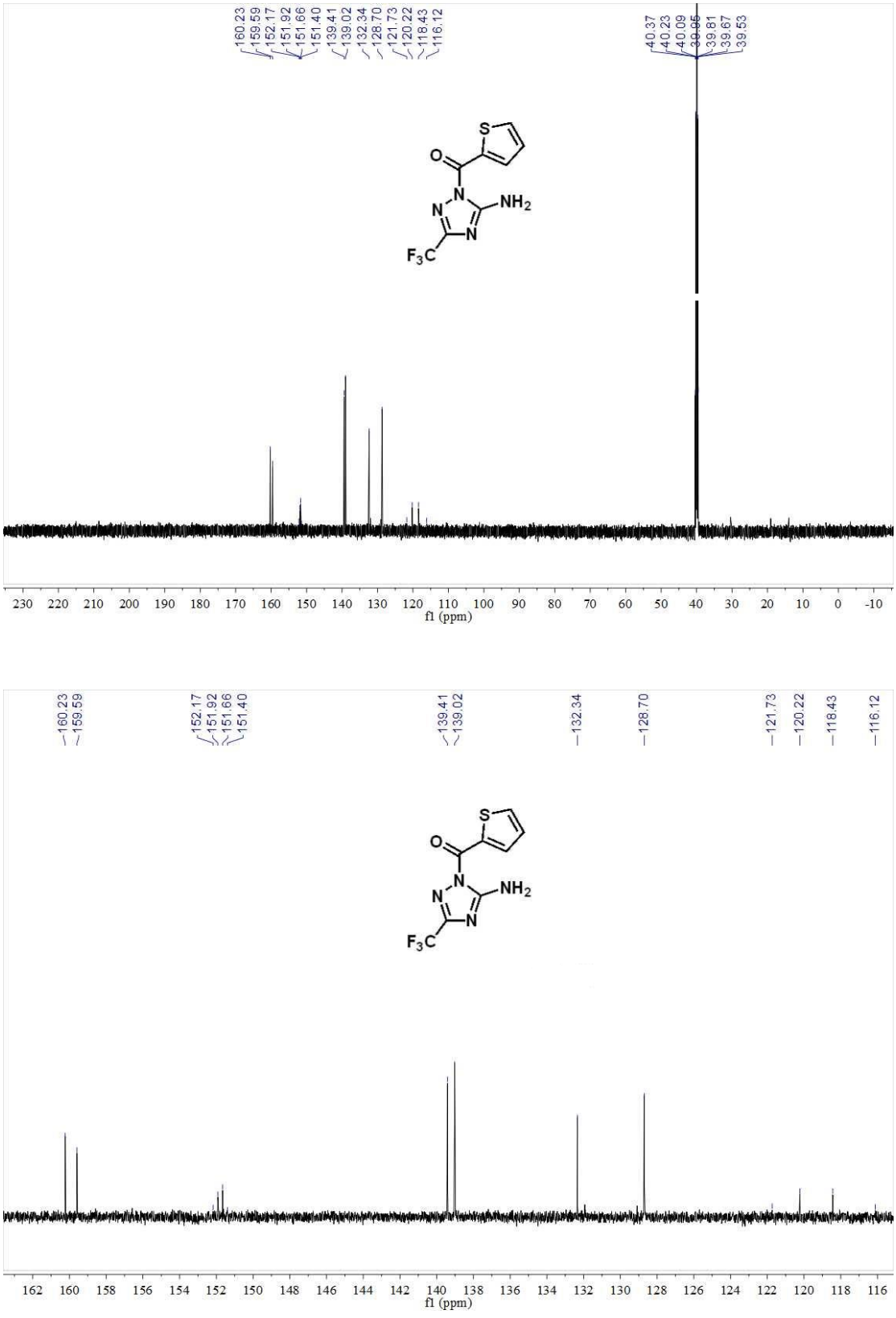
m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
303.05004	35508560.0	13.41	55668.67	1.00		
305.06570	264871696.0	100.00	57357.68	1.00	0.42	$C_{14}H_8ON_4F_3$
306.06926	38607104.0	14.58	56889.27	1.00		
309.04290	14462588.0	5.46	57024.52	1.00		
311.16885	10251565.0	3.87	54919.10	0.00		



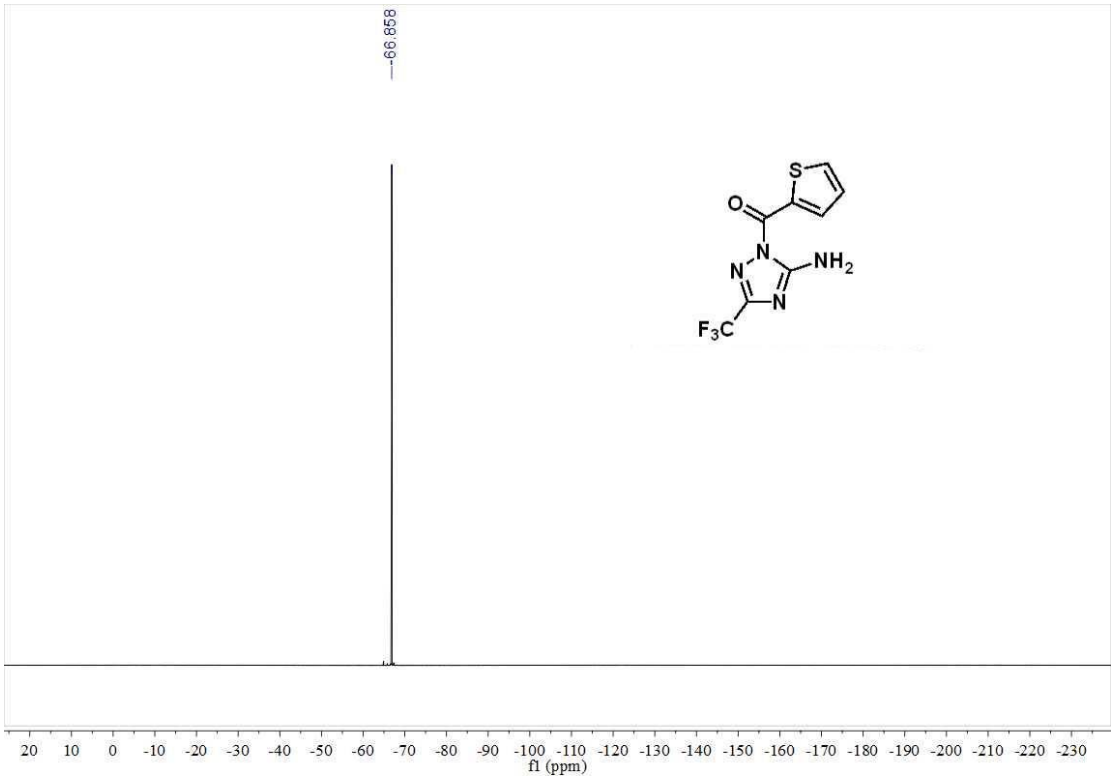
HRMS (ESI) copy of compound **3e**



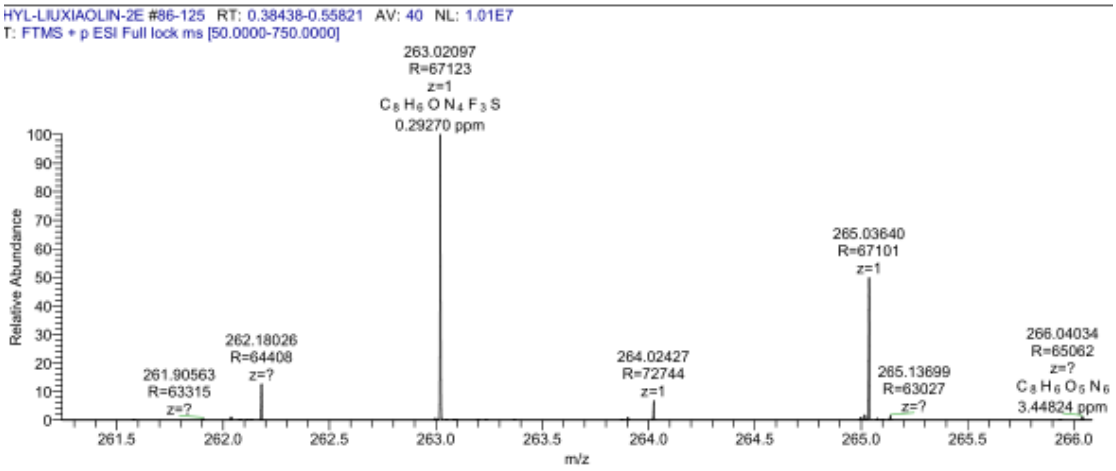
^{1}H NMR (400 MHz) spectrum of **3f** in DMSO-d_6



$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **3f** in DMSO-*d*$_6$

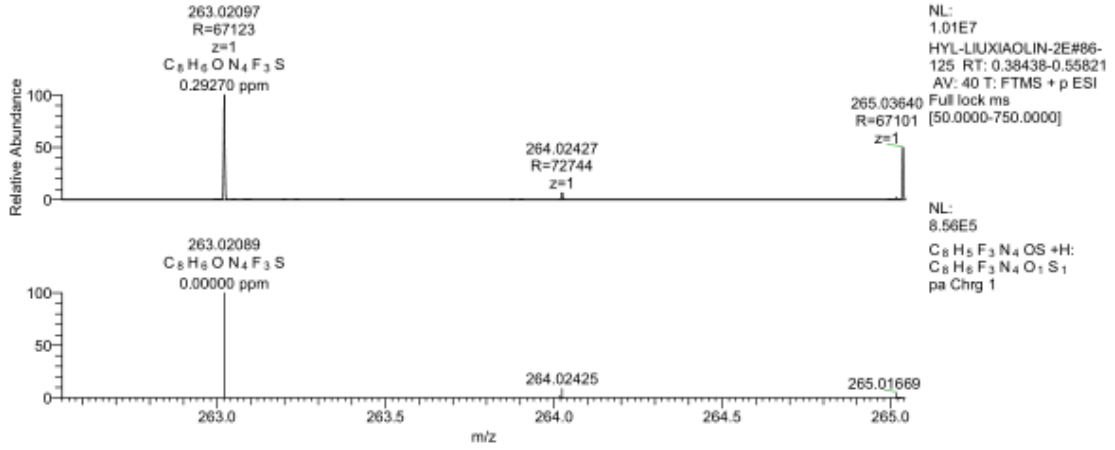


^{19}F NMR (376 MHz) spectrum of **3f** in DMSO-d_6

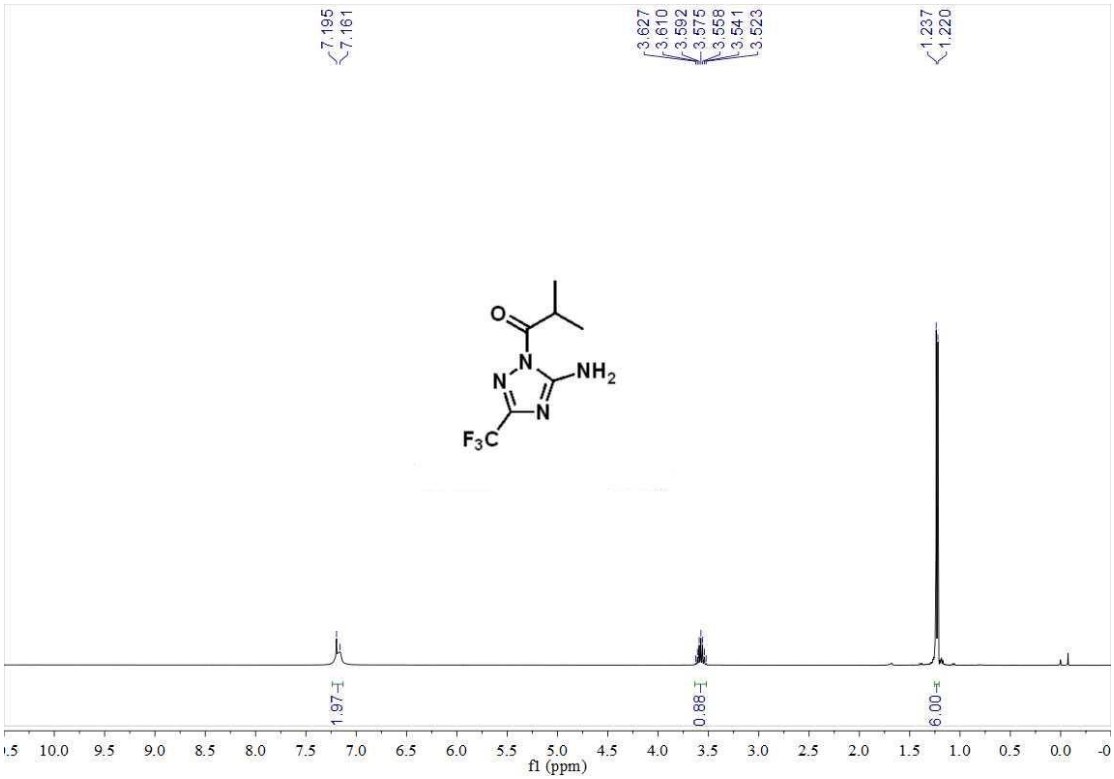


HYL-LIUXIAOLIN-2E#86-125 RT: 0.38438-0.55821 AV: 40
T: FTMS + p ESI Full lock ms [50.0000-750.0000]
m/z= 261.24253-266.08455

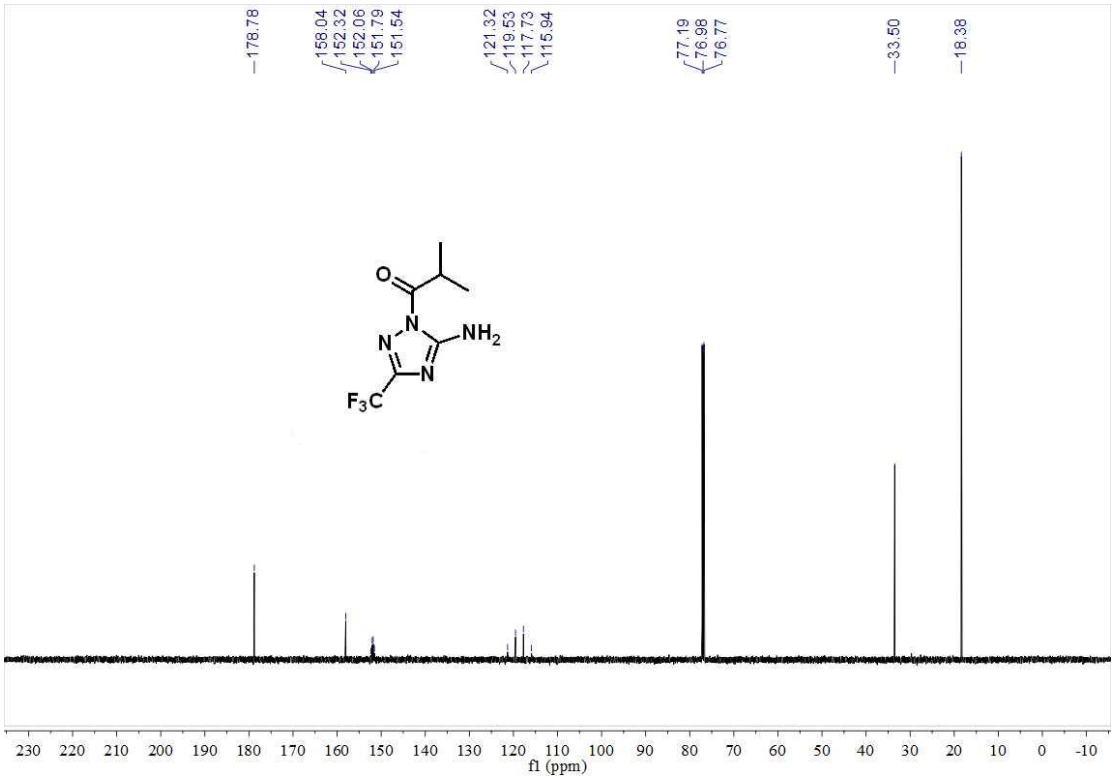
m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
262.18026	1298712.9	12.81	64408.20	0.00		
263.02097	10140636.0	100.00	67123.41	1.00	0.29	C8 H6 O N4 F3 S
264.02427	688226.6	6.79	72743.89	1.00		
265.01644	185254.6	1.83	63834.33	1.00		
265.03640	5049564.0	49.80	67100.92	1.00		



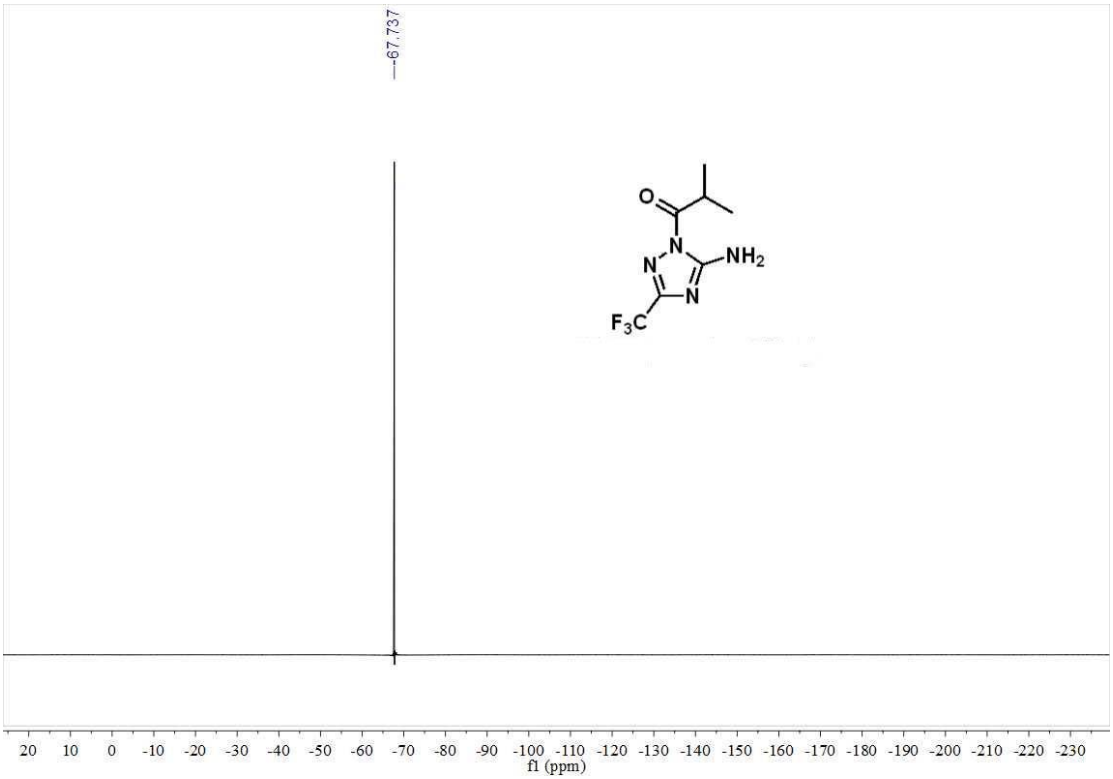
HRMS (ESI) copy of compound **3f**



^{1}H NMR (400 MHz) spectrum of **3g** in $CDCl_3$



^{13}C{^{1}H} NMR (150 MHz) spectrum of **3g** in $CDCl_3$



^{19}F NMR (376 MHz) spectrum of **3g** in $CDCl_3$

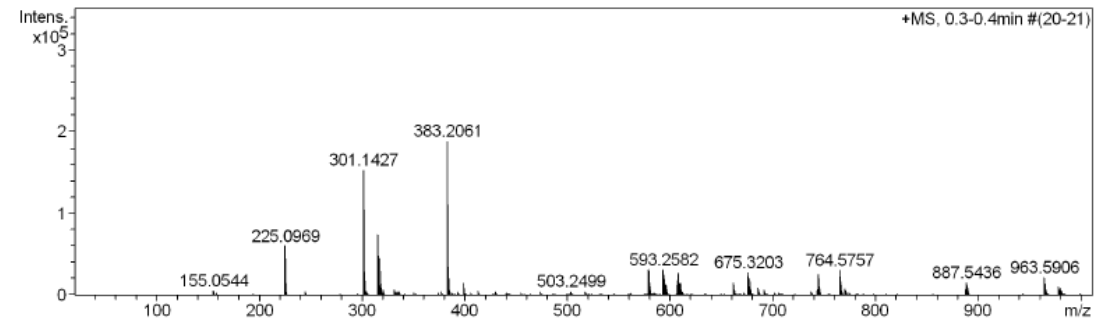
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\user\liuxiaoling20210623-21.d	Acquisition Date	2021-6-23 11:29:03
Method	tune_low.m	Operator	BDAL@DE
Sample Name	2f	Instrument / Ser#	micrOTOF-Q 20453
Comment			

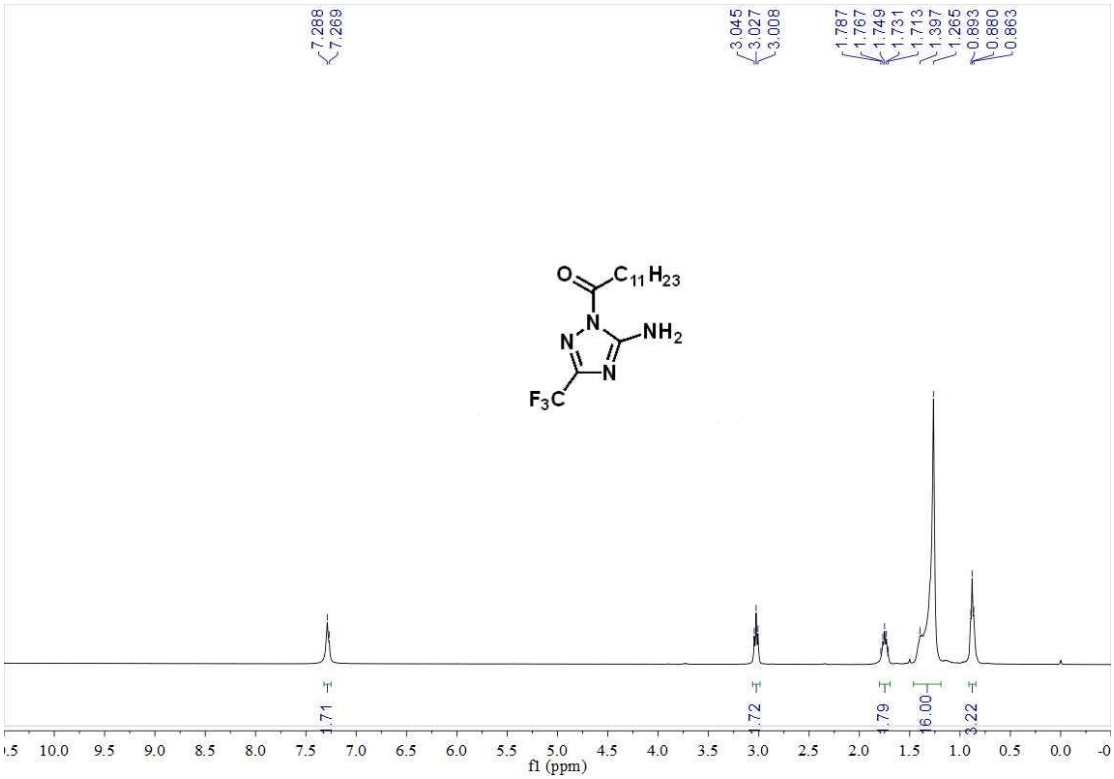
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 ¡ãC
Scan Begin	21 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste

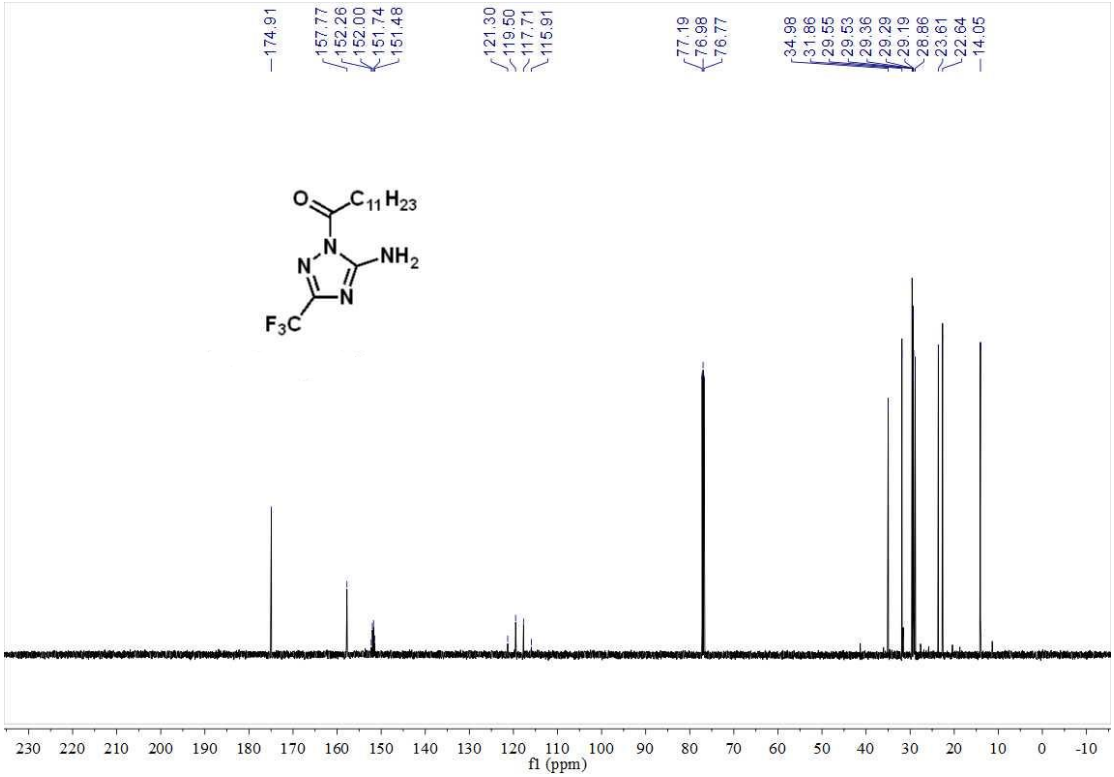


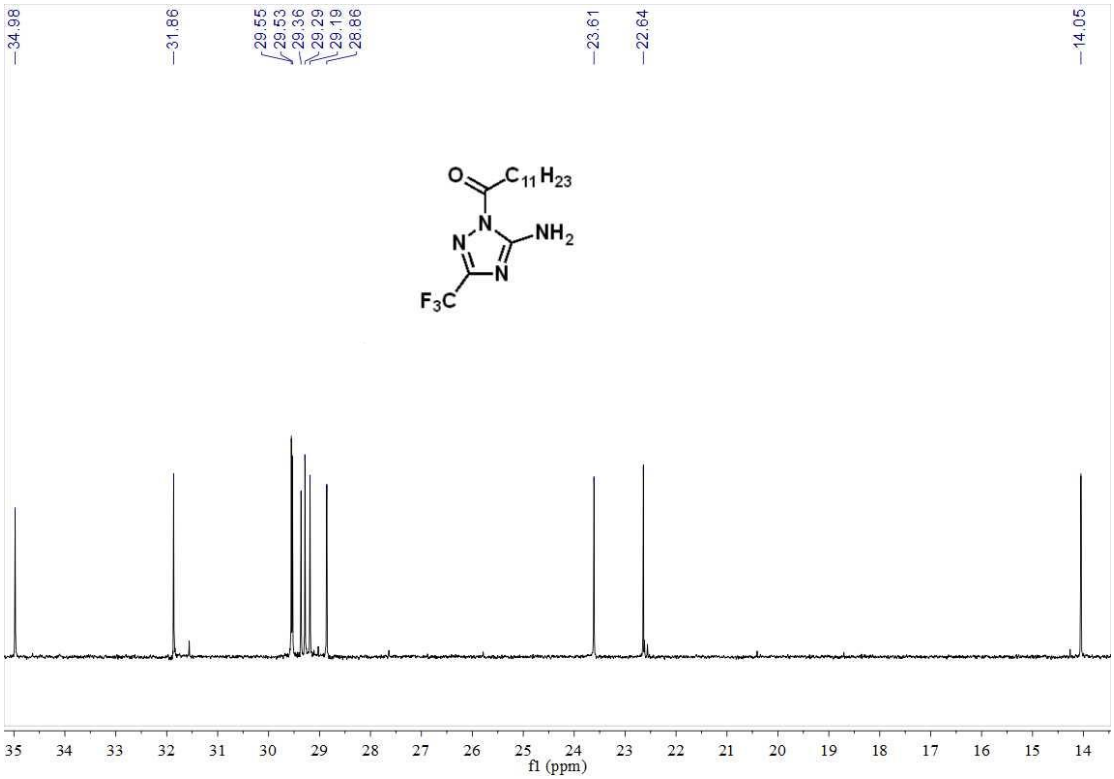
Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma	Std I	Std Mean m/z	Std I Var Norm	Std m/z Diff	Std Comb Dev
245.0630	1	C 7 H 9 F 3 N 4 Na O	245.0621	-3.9	-4.6	3.5	ok	even	8.9	18.1	1.3	7.9	1.8	842.7

HRMS (ESI) copy of compound **3g**

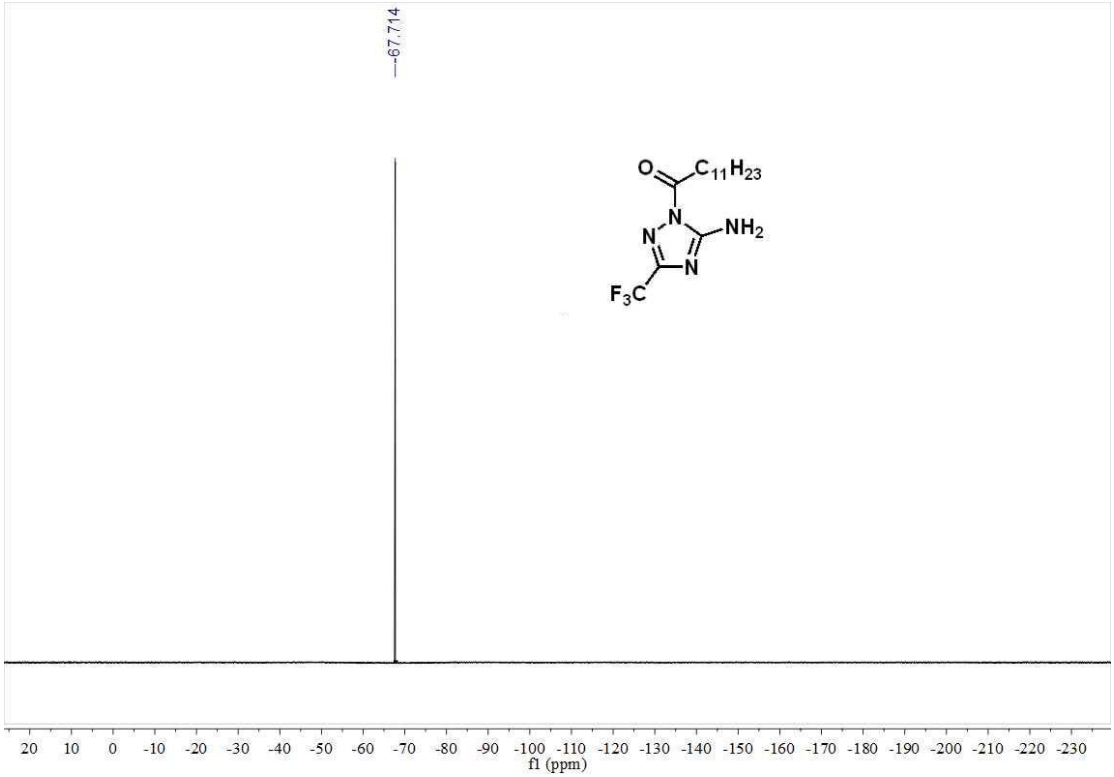


^{1}H NMR (400 MHz) spectrum of **3h** in $CDCl_3$

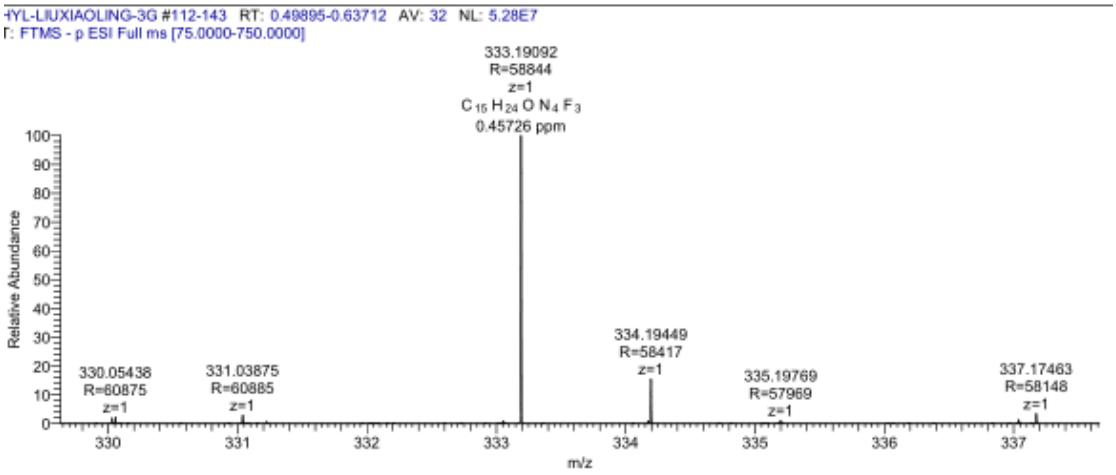




$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **3h** in $CDCl_3$

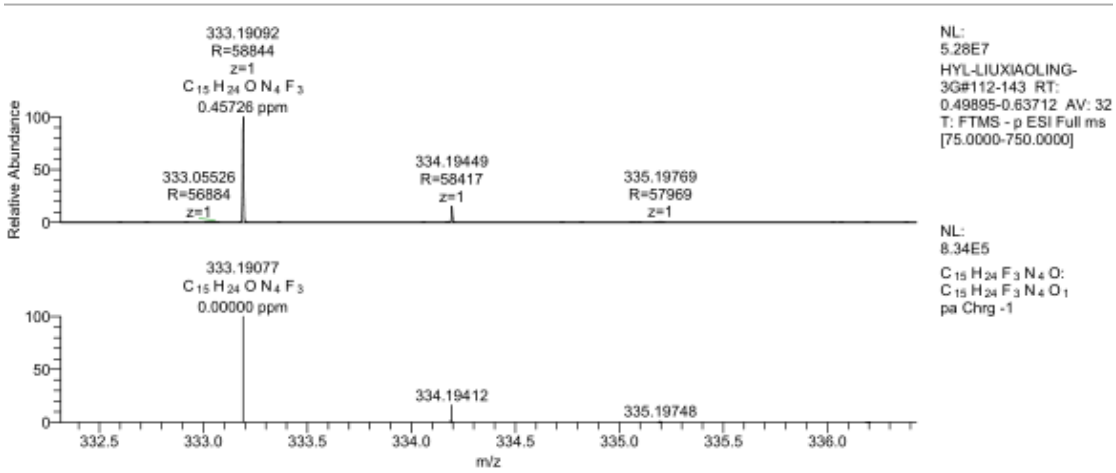


^{19}F NMR (376 MHz) spectrum of **3h** in $CDCl_3$

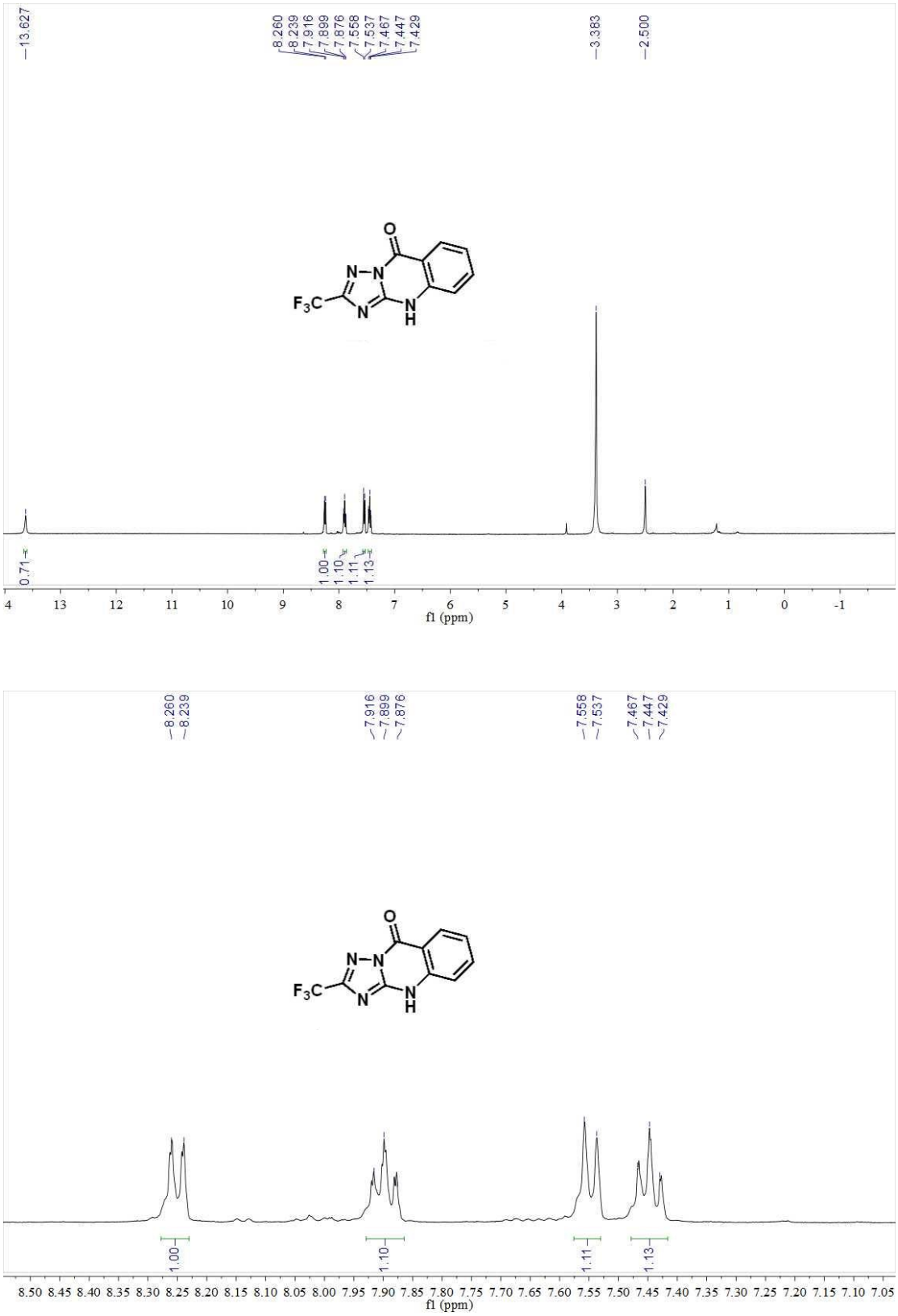


HYL-LIUXIAOLING-3G#112-143 RT: 0.49895-0.63712 AV: 32
T: FTMS - p ESI Full ms [75.0000-750.0000]
m/z= 329.63135-337.66149

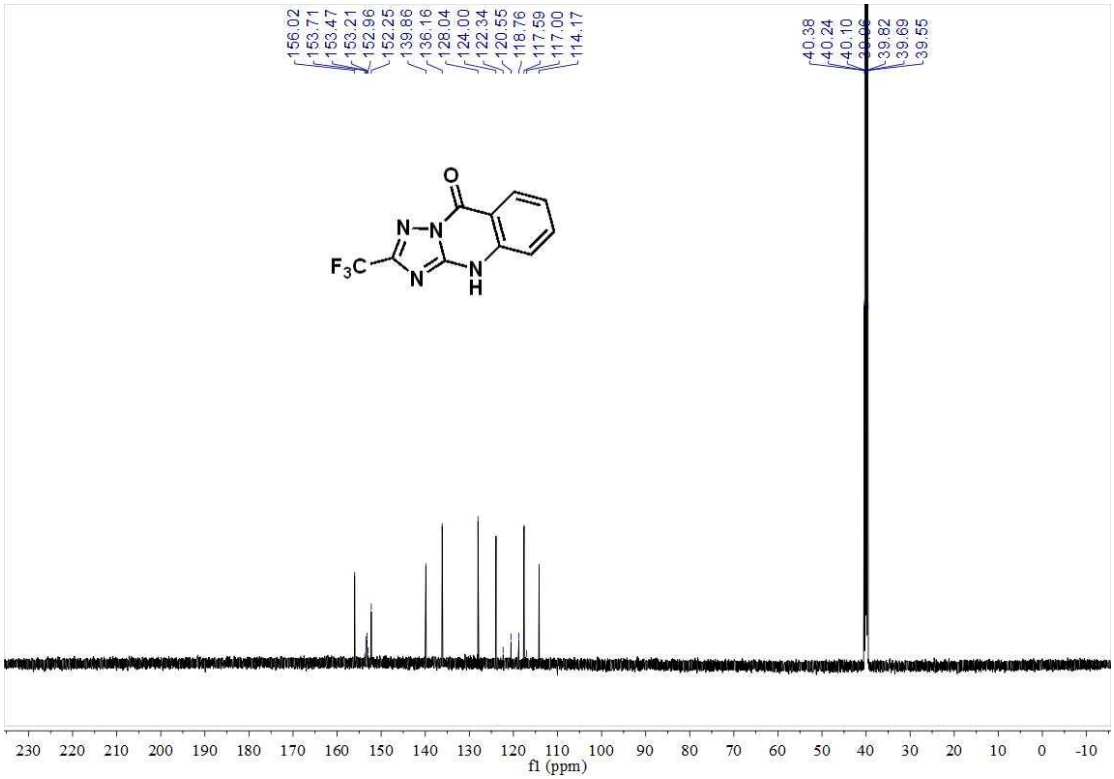
m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
330.02926	1039746.8	1.96	60163.28	1.00		
330.05438	1191047.8	2.25	60875.16	1.00		
331.03875	1577355.9	2.98	60884.51	1.00		
333.19092	52984556.0	100.00	58844.30	1.00	0.46	$C_{15}H_{24}ON_4F_3$
334.19449	8366770.0	15.79	58417.41	1.00		
337.03914	728693.9	1.38	60737.91	1.00		
337.17463	1868253.5	3.53	58148.49	1.00		



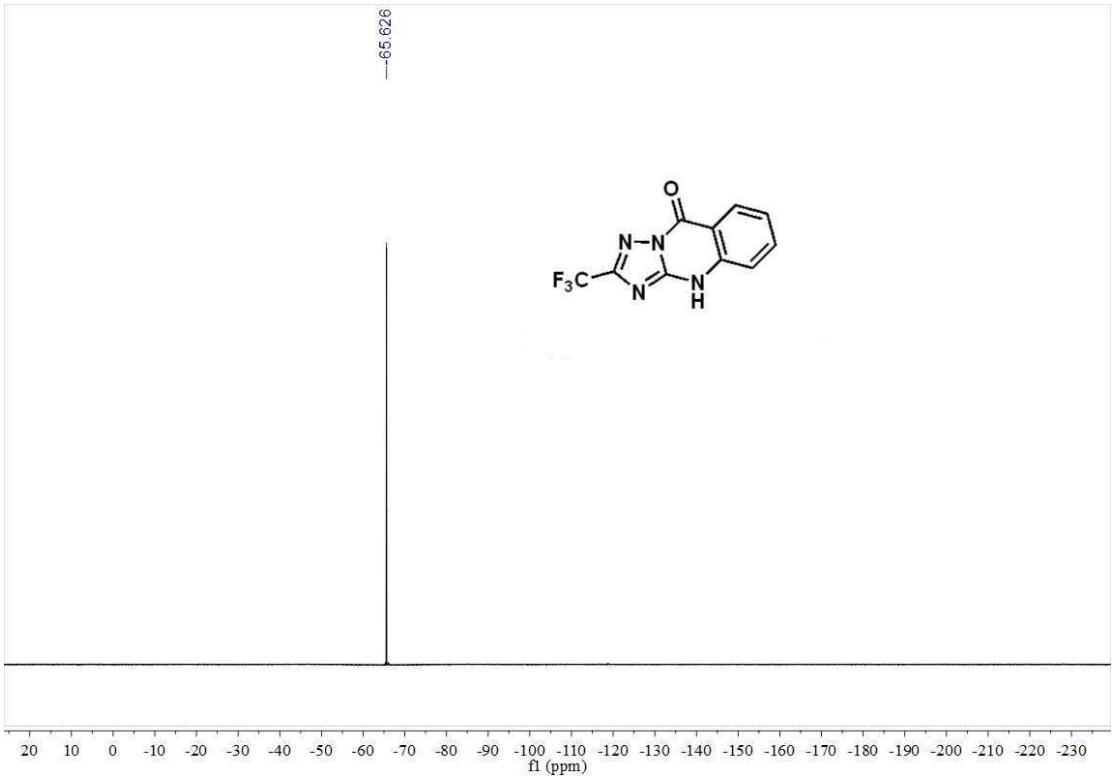
HRMS (ESI) copy of compound **3h**



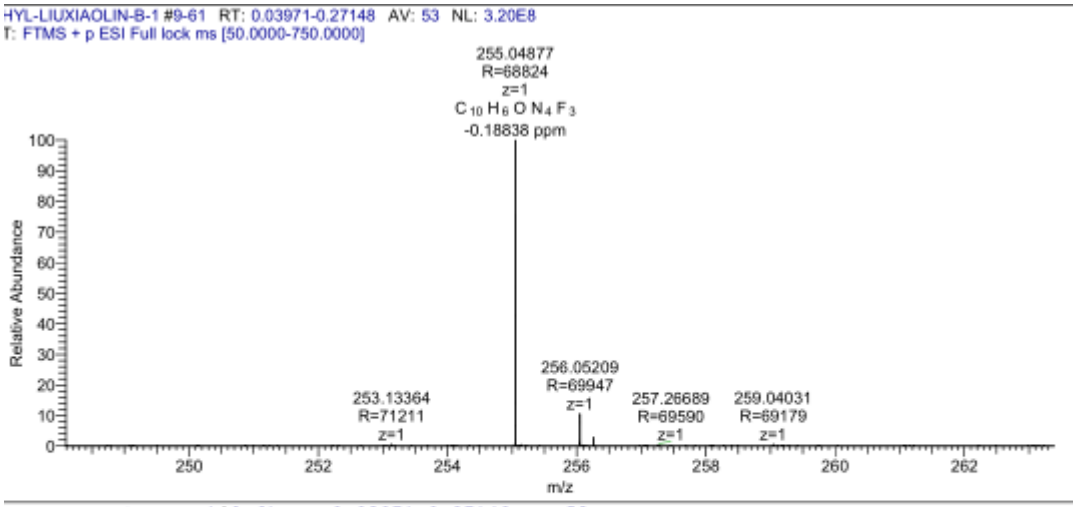
^{1}H NMR (400 MHz) spectrum of **4a** in DMSO-d_6



$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **4a** in DMSO-*d*$_6$

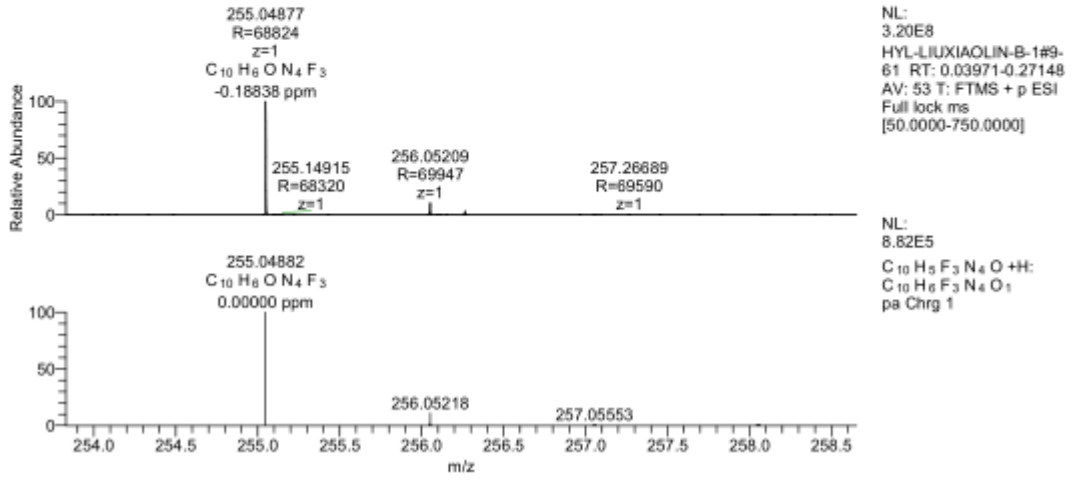


^{19}F NMR (376 MHz) spectrum of **4a** in DMSO-*d*$_6$

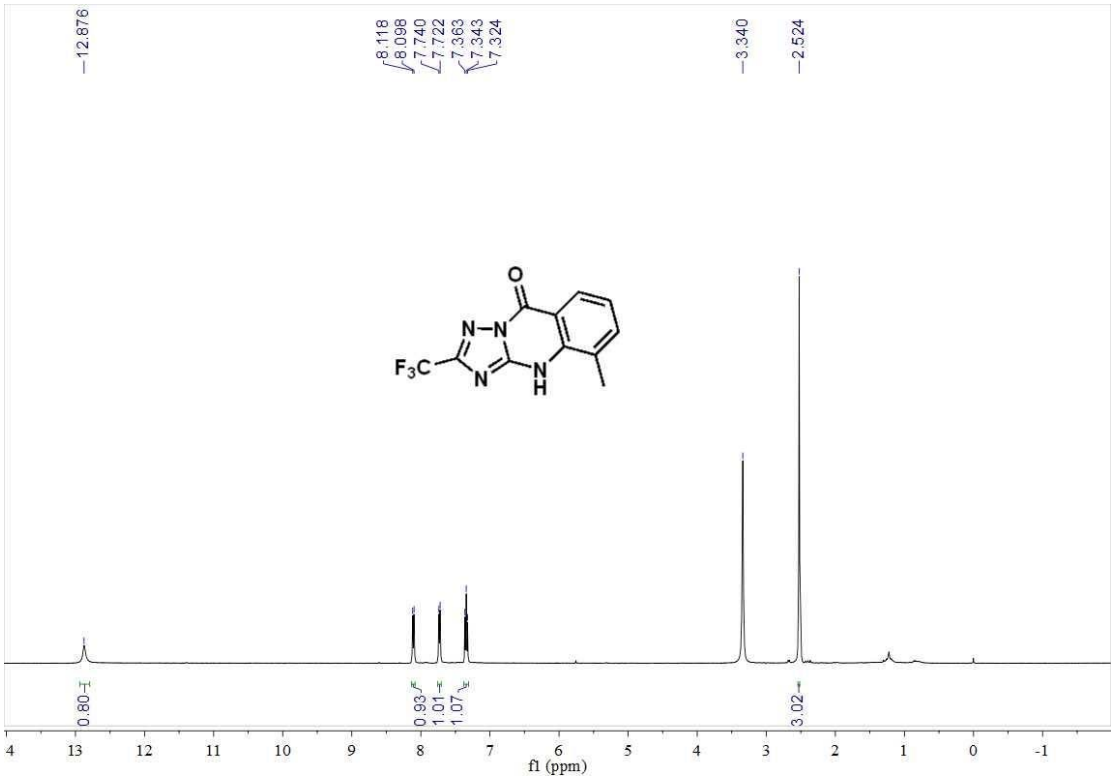


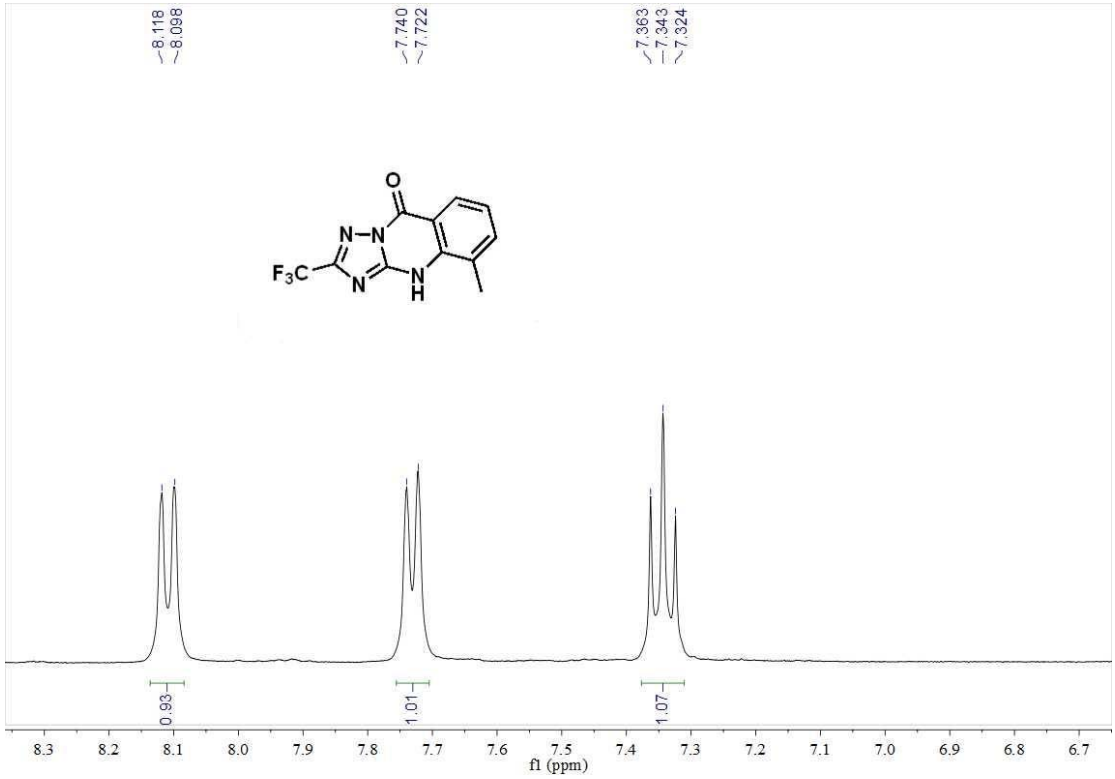
HYL-LIUXIAOLIN-B-1#9-61 RT: 0.03971-0.27148 AV: 53
T: FTMS + p ESI Full lock ms [50.0000-750.0000]
m/z= 248.09235-263.39129

m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
253.13364	2034478.1	0.63	71211.41	1.00		
255.04877	320960128.0	100.00	68824.11	1.00	-0.19	$C_{10}H_6ON_4F_3$
256.04580	4043985.5	1.26	83455.97	1.00		
256.05209	33908984.0	10.56	69947.47	1.00		
256.26342	9716543.0	3.03	67628.91	1.00		

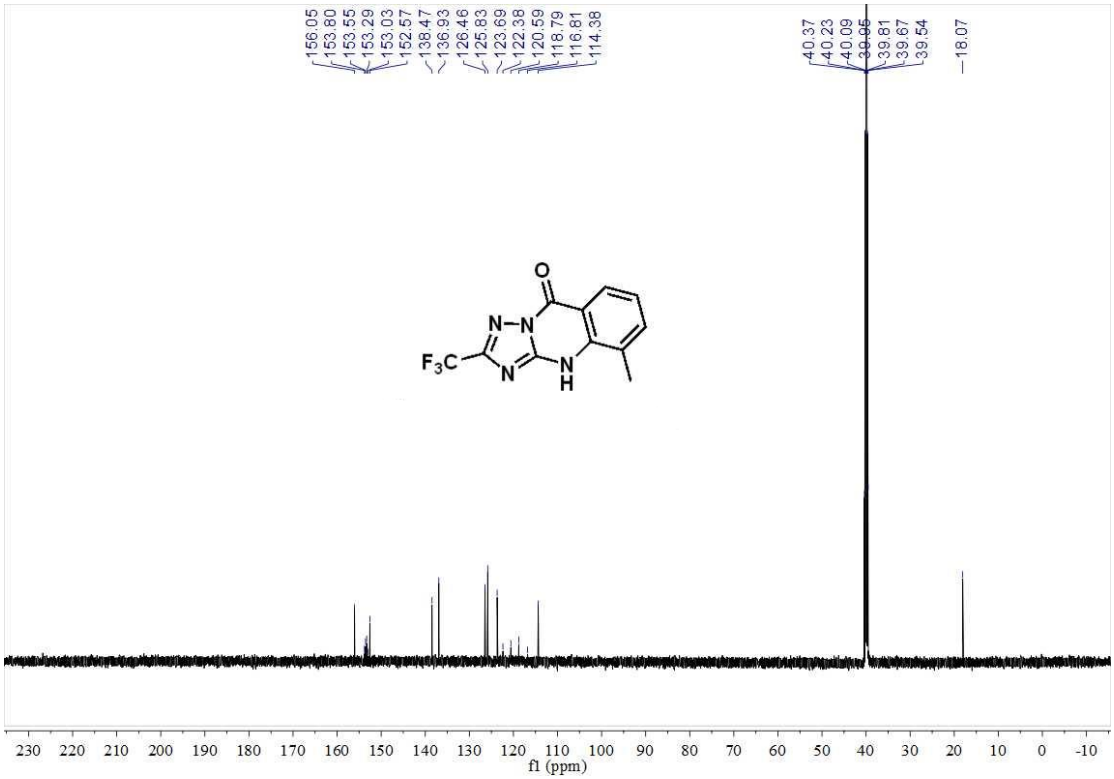


HRMS (ESI) copy of compound **4a**

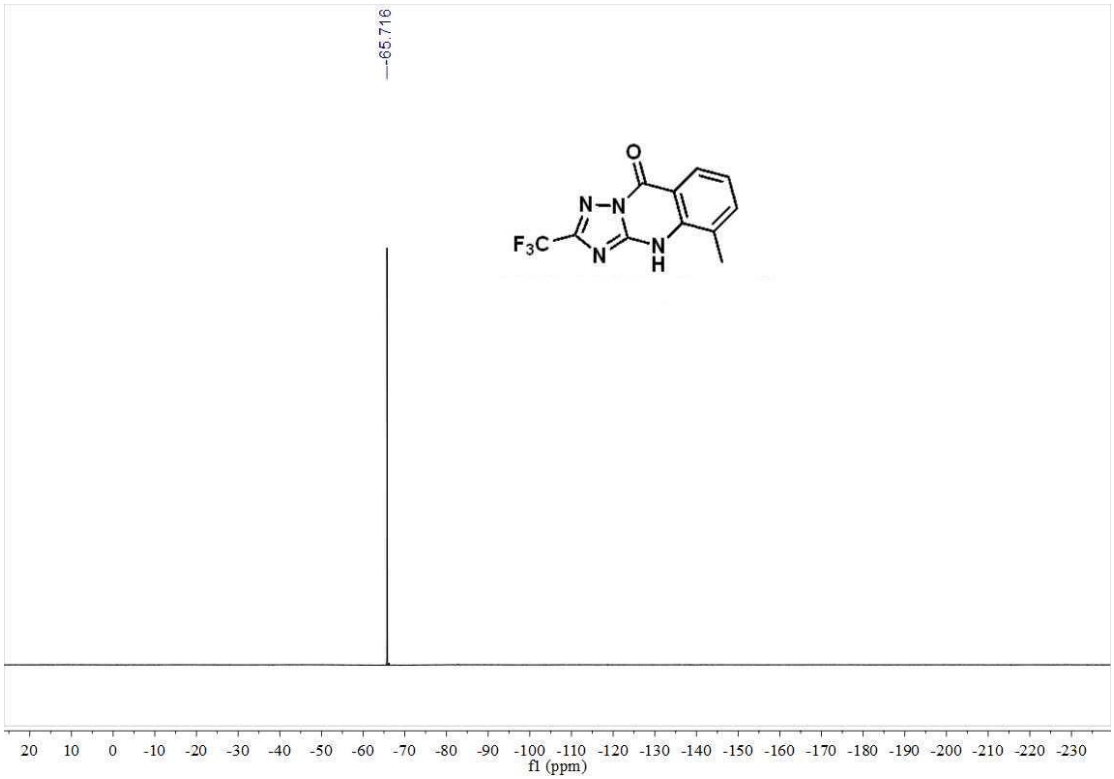




^{1}H NMR (400 MHz) spectrum of **4b** in DMSO-d_6



$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **4b** in DMSO-*d*$_6$



^{19}F NMR (376 MHz) spectrum of **4b** in DMSO-*d*$_6$

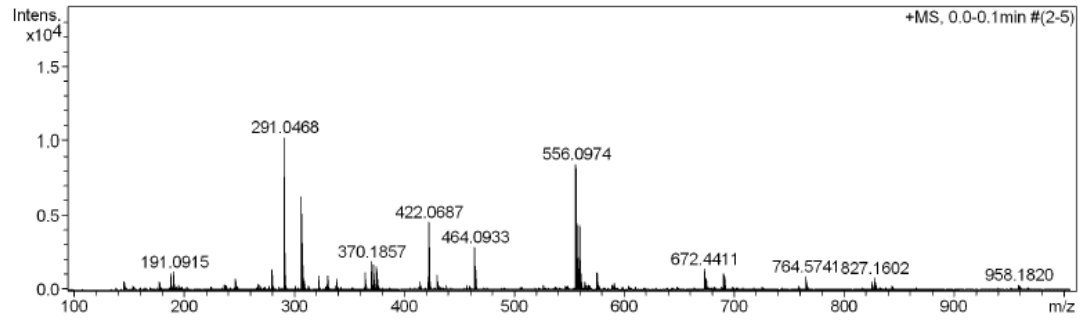
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\user\liuxiaoling20211124-6.d	Acquisition Date	2021-11-24 11:08:44
Method	tune_low.m	Operator	BDAL@DE
Sample Name	4d	Instrument / Ser#	micrOTOF-Q 20453
Comment			

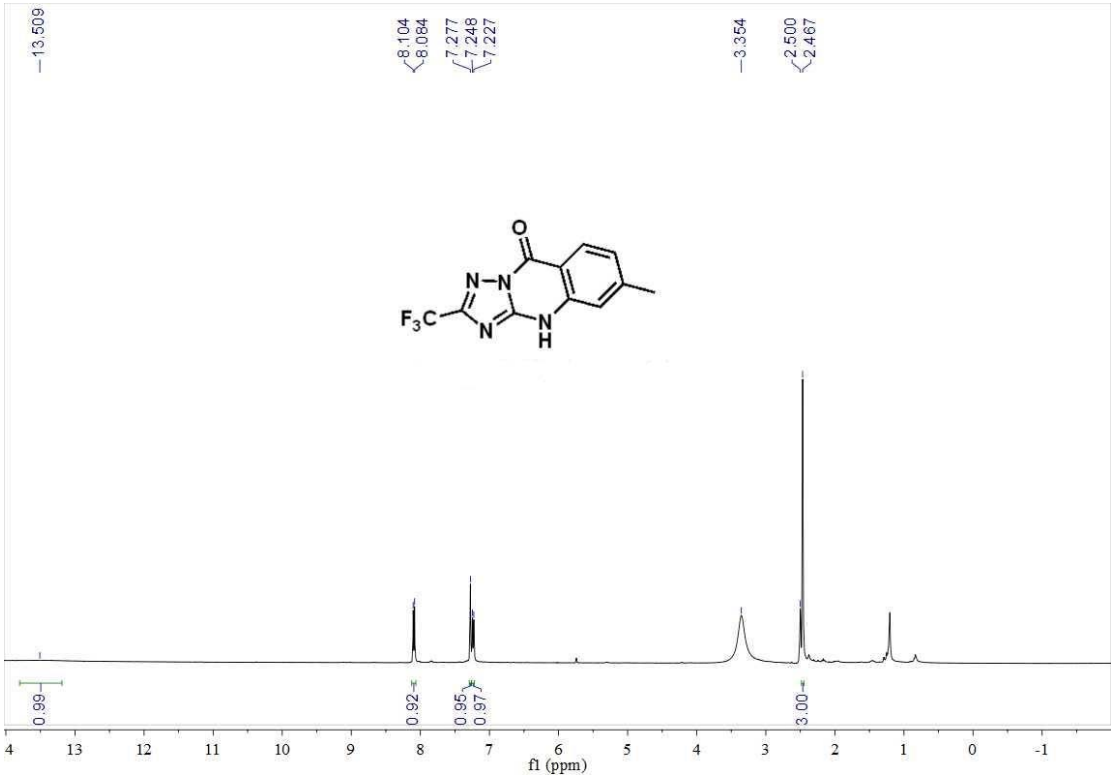
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 ¡ãC
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste

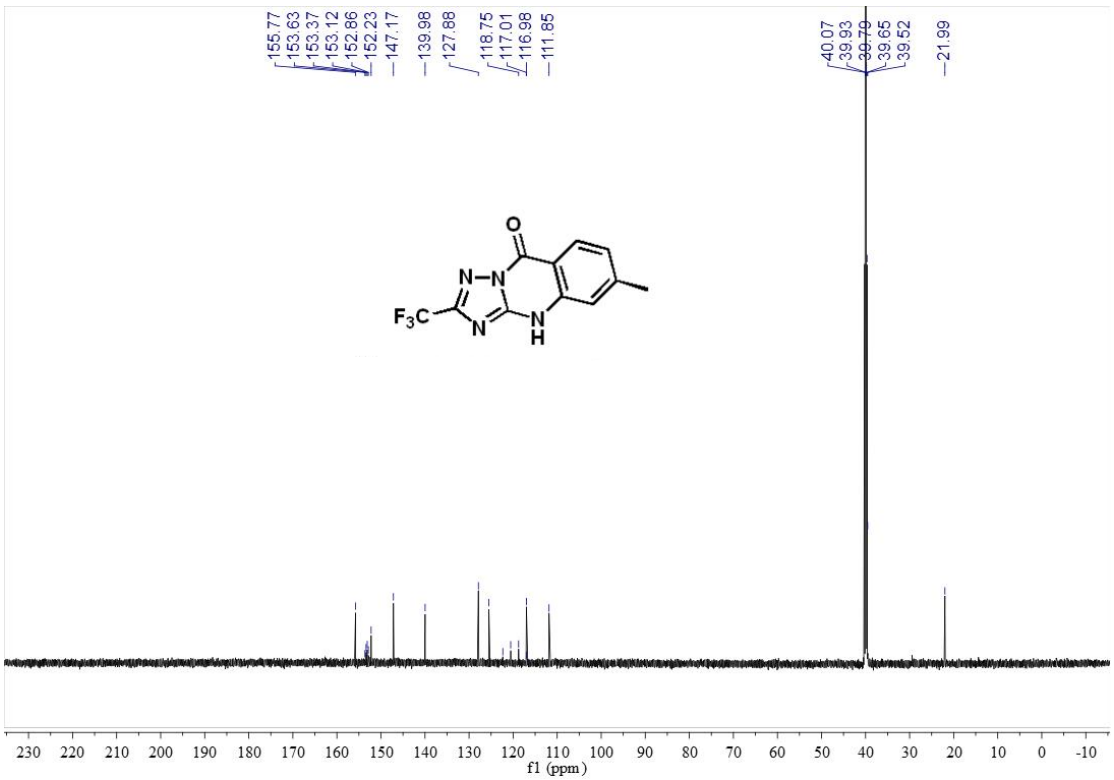


Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	e¡¥ Conf	mSigma	Std I	Std Mean m/z	Std I Var Norm	Std m/z Diff	Std Comb Dev
291.0468	1	C 11 H 7 F 3 N 4 Na O	291.0464	-1.4	-0.9	8.5	ok	even	5.4	8.7	0.5	6.1	1.4	842.7

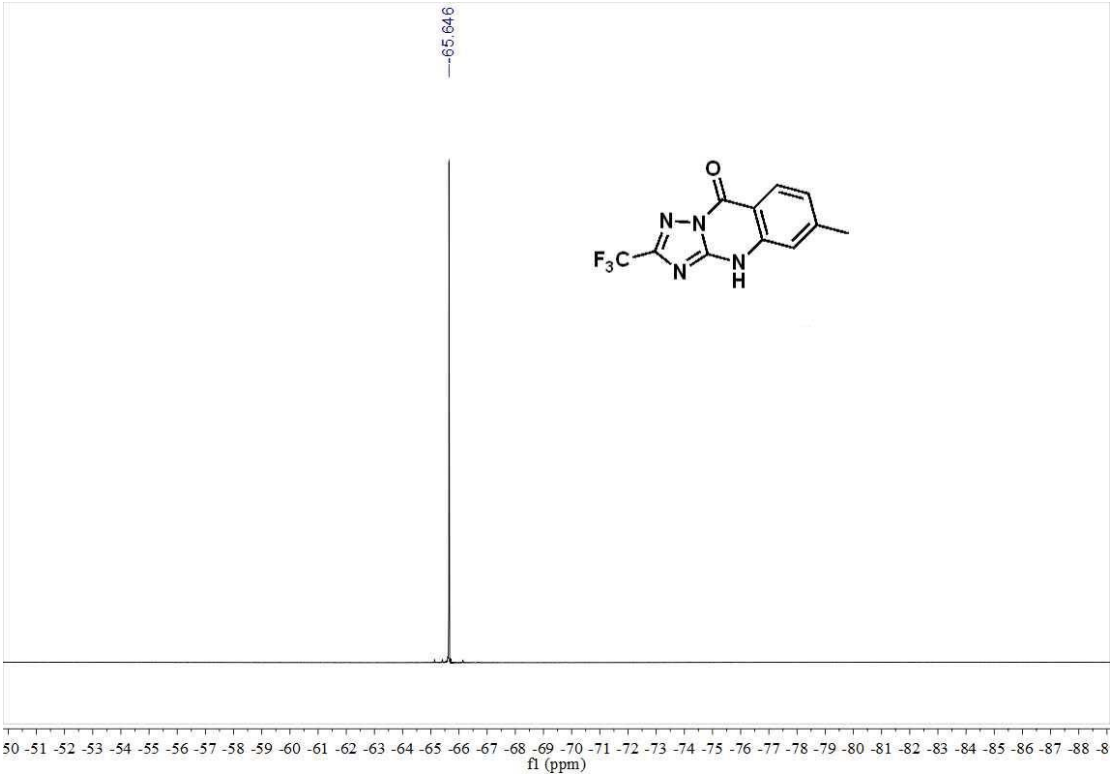
HRMS (ESI) copy of compound **4b**



^{1}H NMR (400 MHz) spectrum of **4c** in DMSO-*d*$_6$



^{13}C{^{1}H} NMR (150 MHz) spectrum of **4c** in DMSO-*d*$_6$



^{19}F NMR (376 MHz) spectrum of **4c** in DMSO-d6

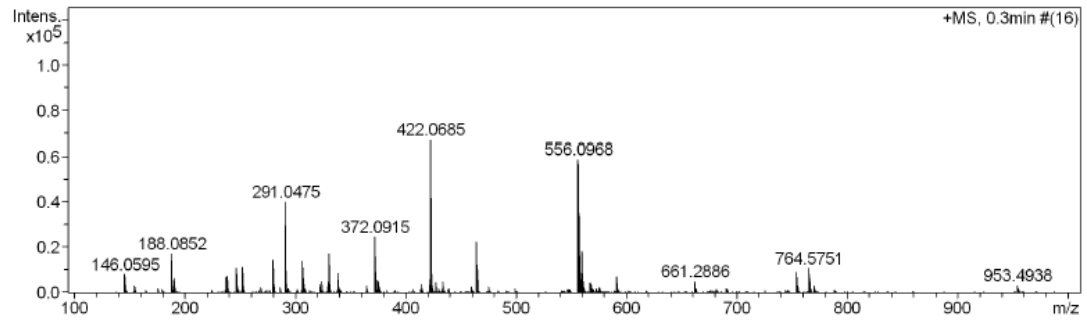
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\user\liuxiaoling20211124-7.d	Acquisition Date	2021-11-24 11:10:34
Method	tune_low.m		
Sample Name	4e	Operator	BDAL@DE
Comment		Instrument / Ser#	micrOTOF-Q 20453

Acquisition Parameter

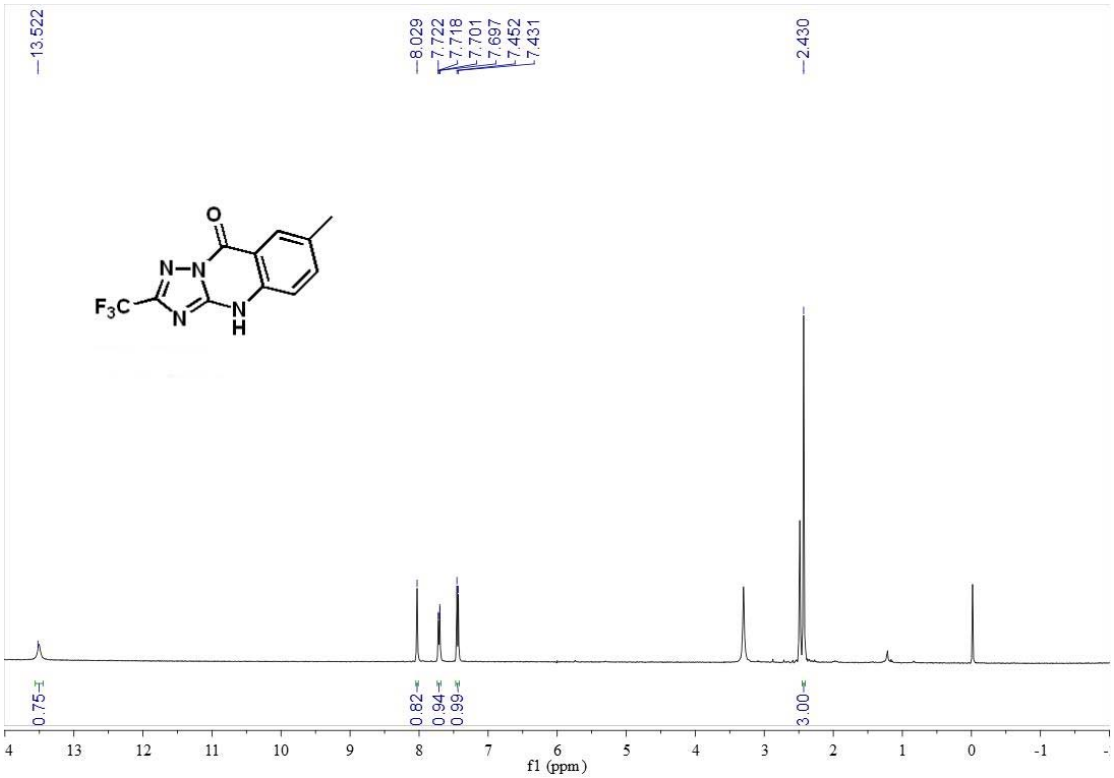
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 ¡ãC
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste



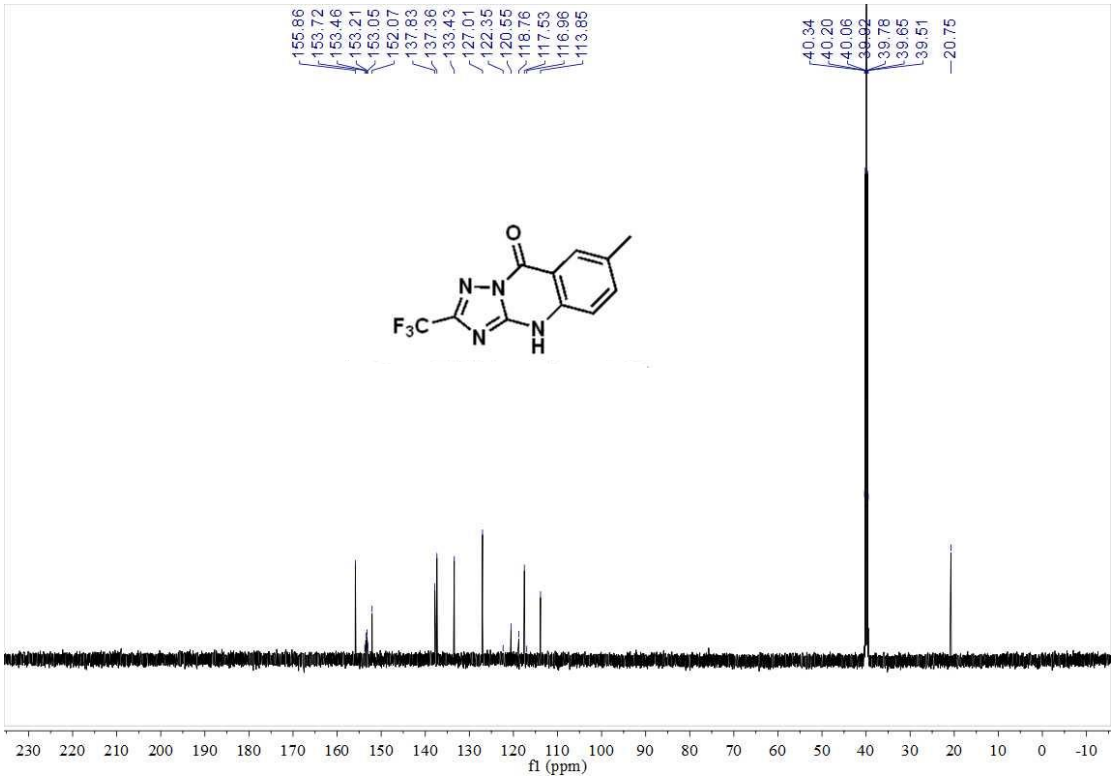
Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	e¡¥Conf	mSigma	Std I	Std Mean m/z	Std I Var Norm	Std m/z Diff	Std Comb Dev
291.0475	1	C 11 H 7 F 3 N 4 Na O	291.0464	-3.8	-3.3	8.5	ok	even	5.3	8.6	1.0	6.1	1.1	842.7

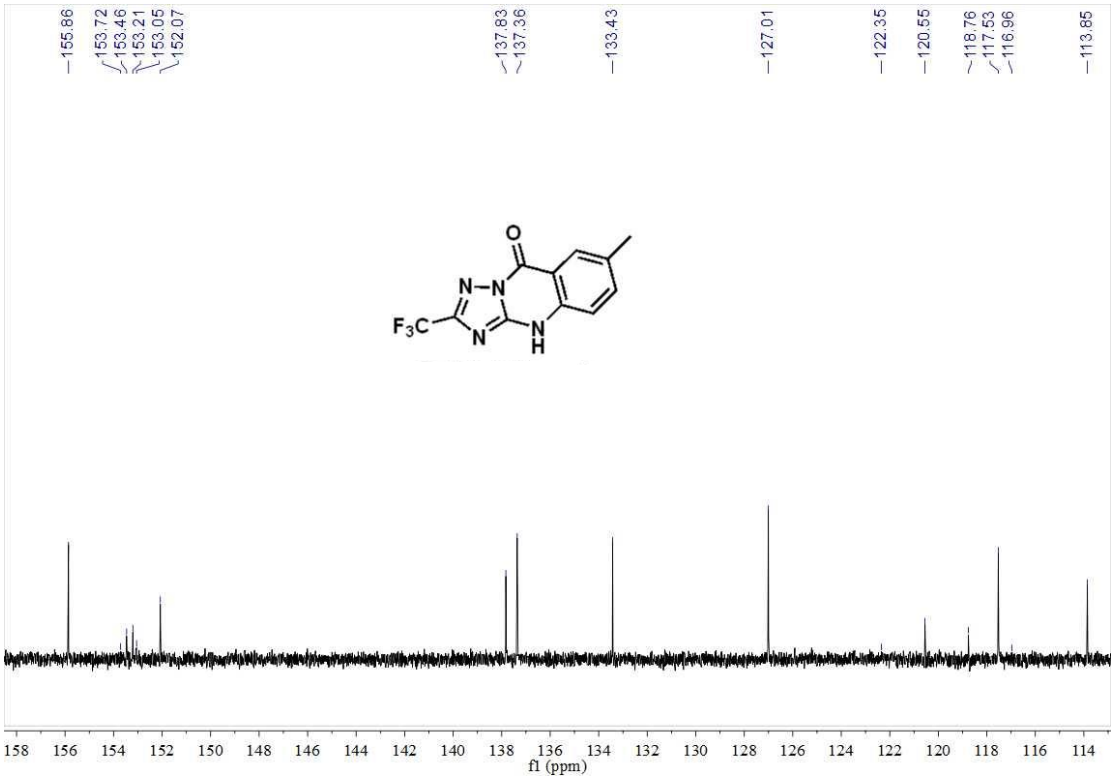
NMR copies of compound **4c**

:

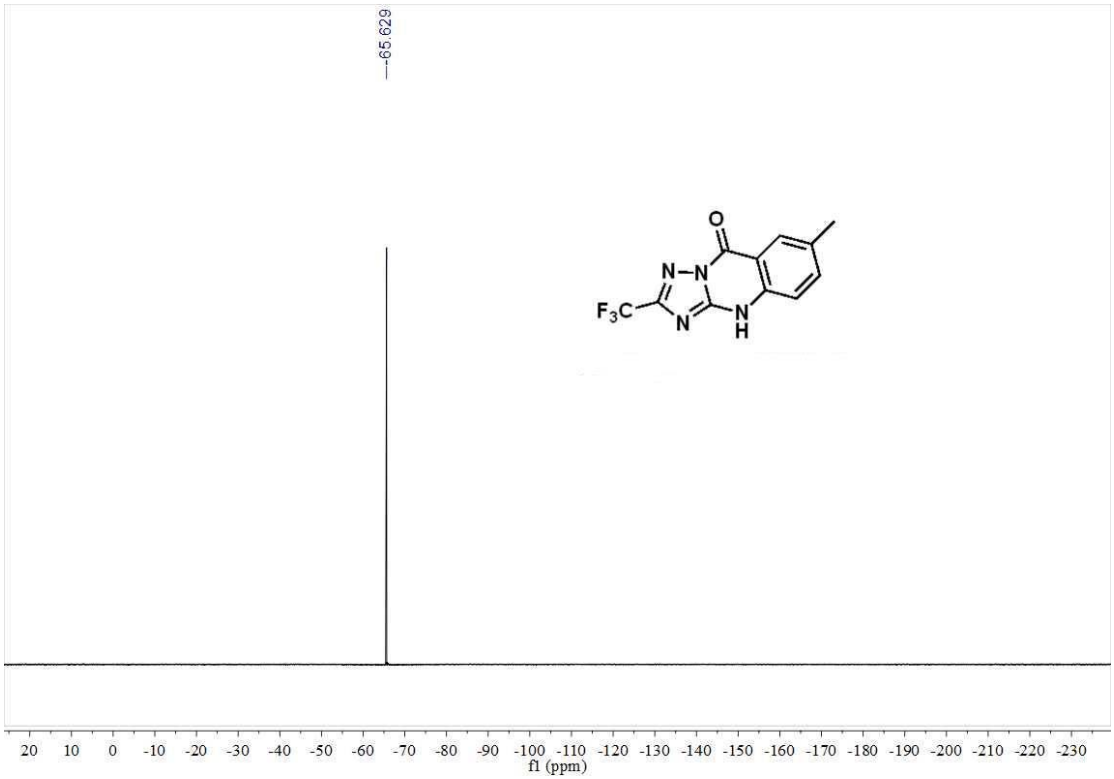


^{1}H NMR (400 MHz) spectrum of **4d** in DMSO-d_6





$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **4d** in DMSO-*d*$_6$



^{19}F NMR (376 MHz) spectrum of **4d** in DMSO-*d*$_6$

Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\user\liuxiaoling20211124-8.d	Acquisition Date	2021-11-24 11:11:39
Method	tune_low.m	Operator	BDAL@DE
Sample Name	4f	Instrument / Ser#	micrOTOF-Q 20453
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 ¡ãC
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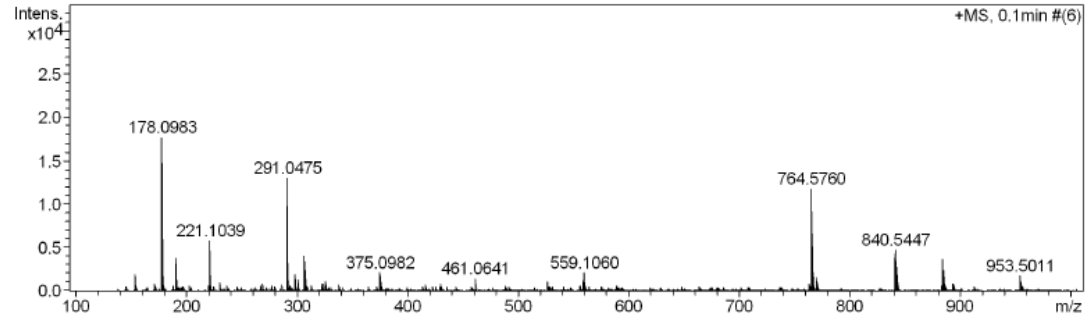
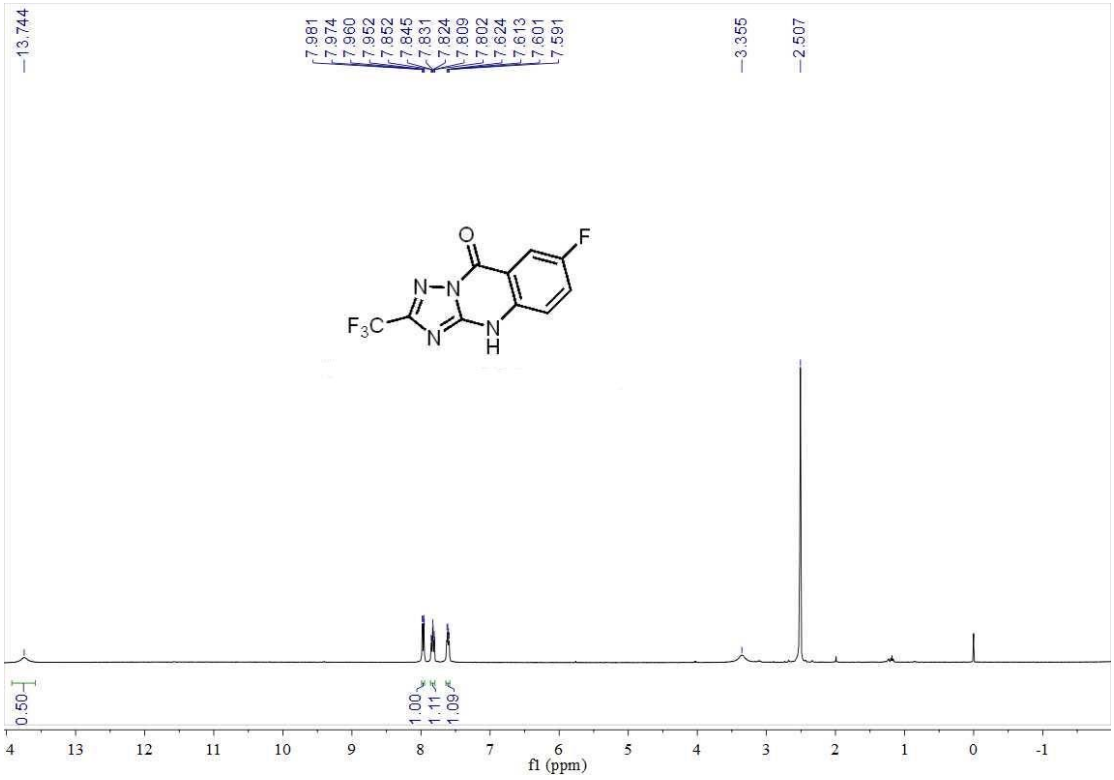
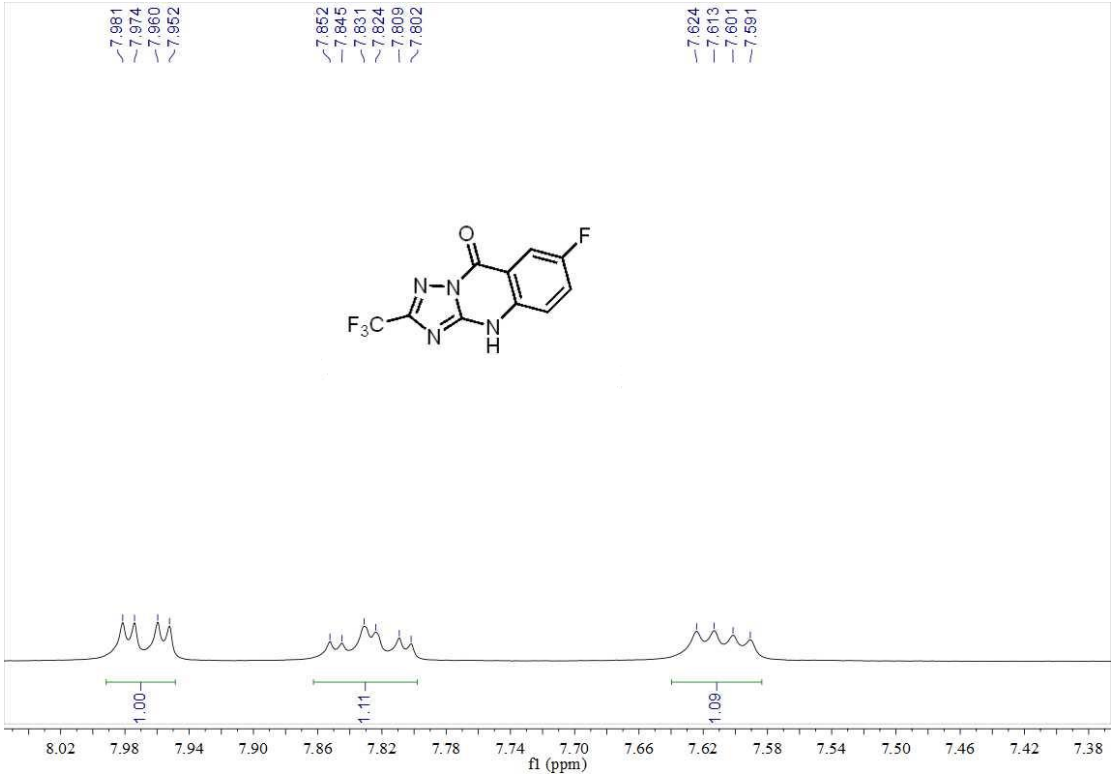
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste



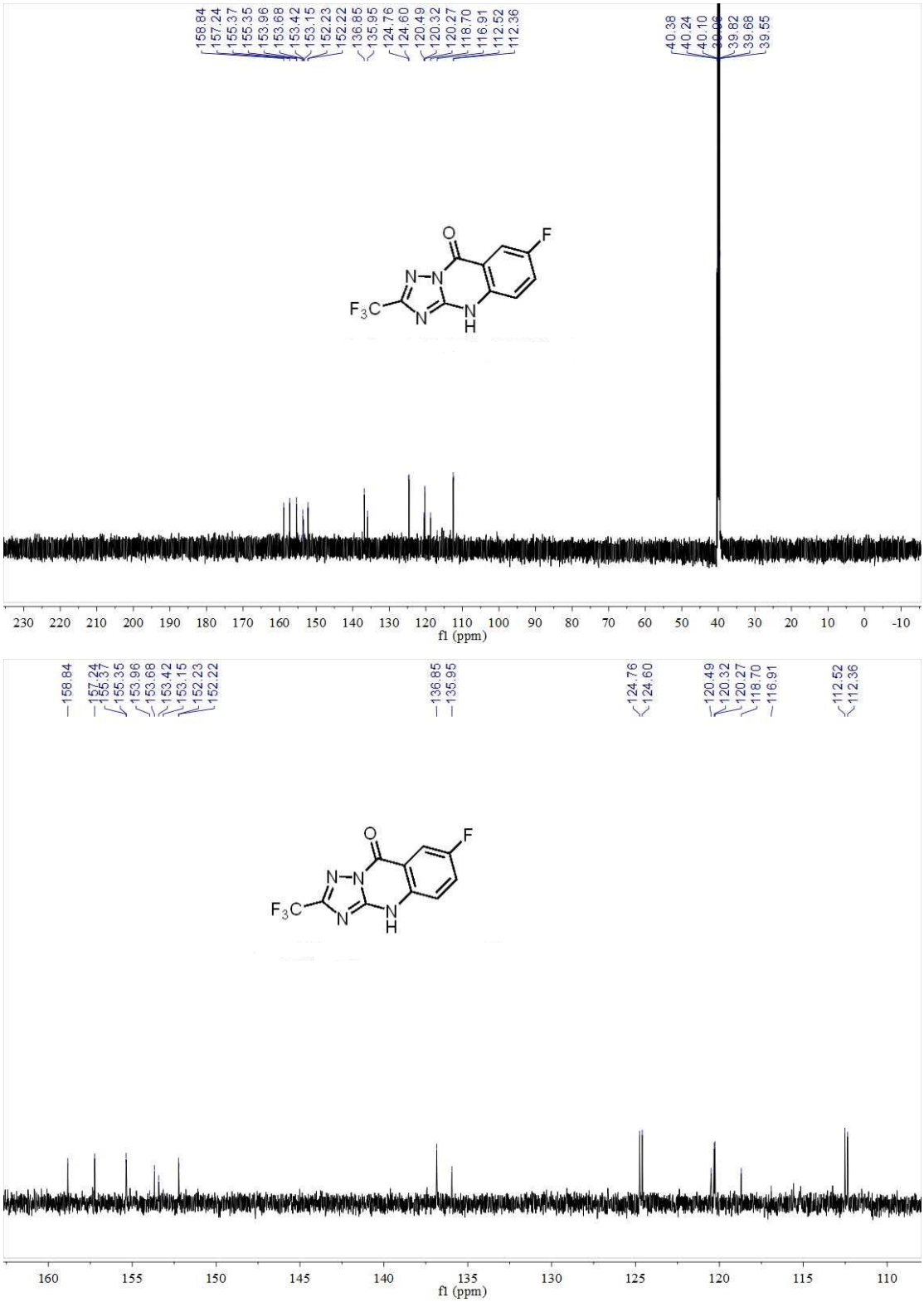
Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	e¡¥ Conf	mSigma	Std I	Std Mean m/z	Std I Var Norm	Std m/z Diff	Std Comb Dev
291.0475	1	C 11 H 7 F 3 N 4 Na O	291.0464	-3.6	-6.5	8.5	ok	even	4.8	7.4	3.0	5.8	7.1	842.7

NMR copies of compound **4d**

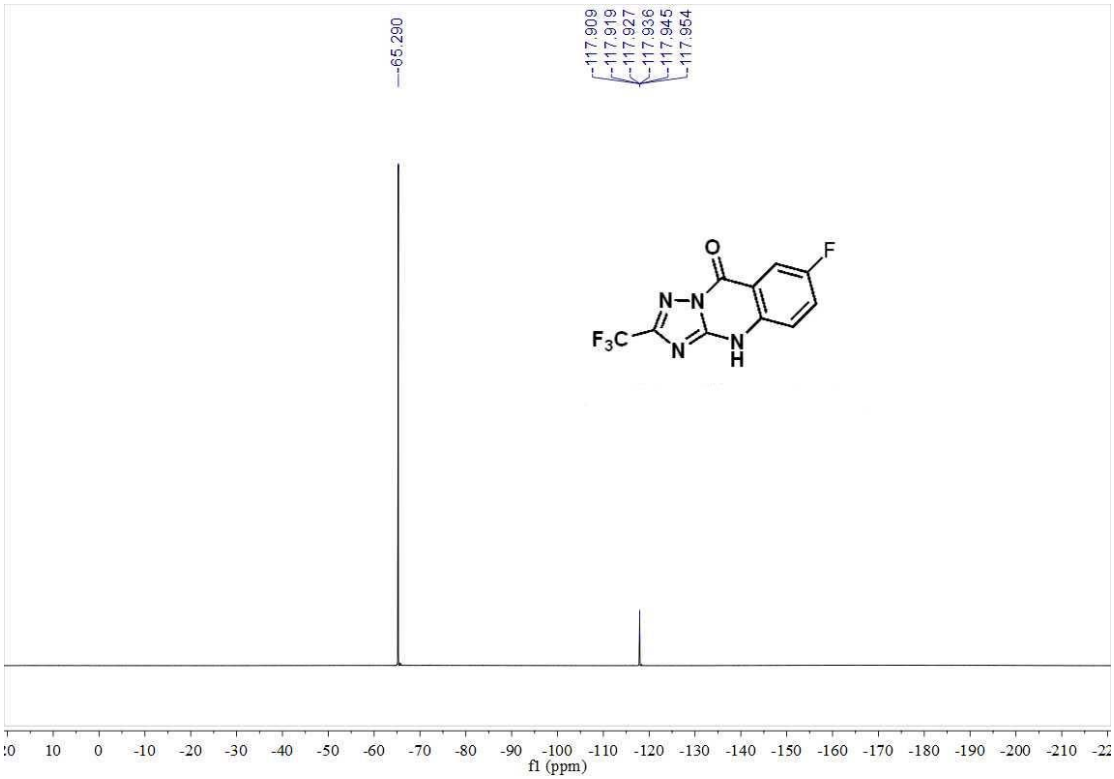




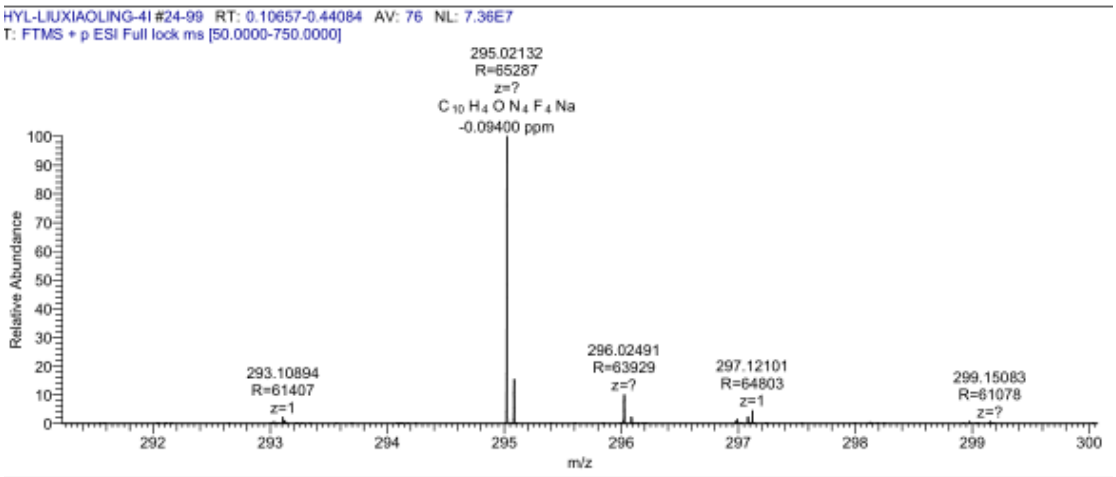
^{1}H NMR (400 MHz) spectrum of **4e** in DMSO-*d*$_6$



$^{13}C\{^{1}H\}$ NMR (150 MHz) spectrum of **4e** in DMSO-*d*$_6$

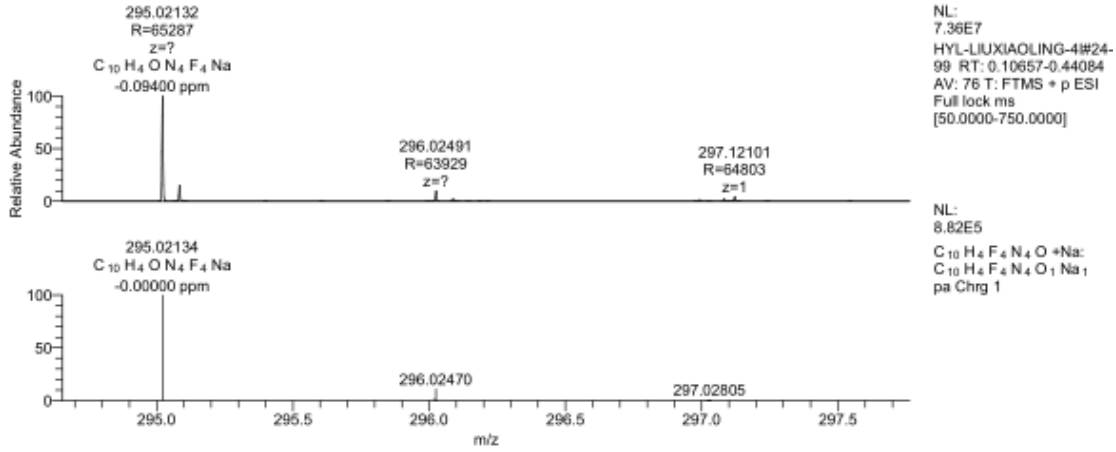


^{19}F NMR (376 MHz) spectrum of **4e** in DMSO-d_6

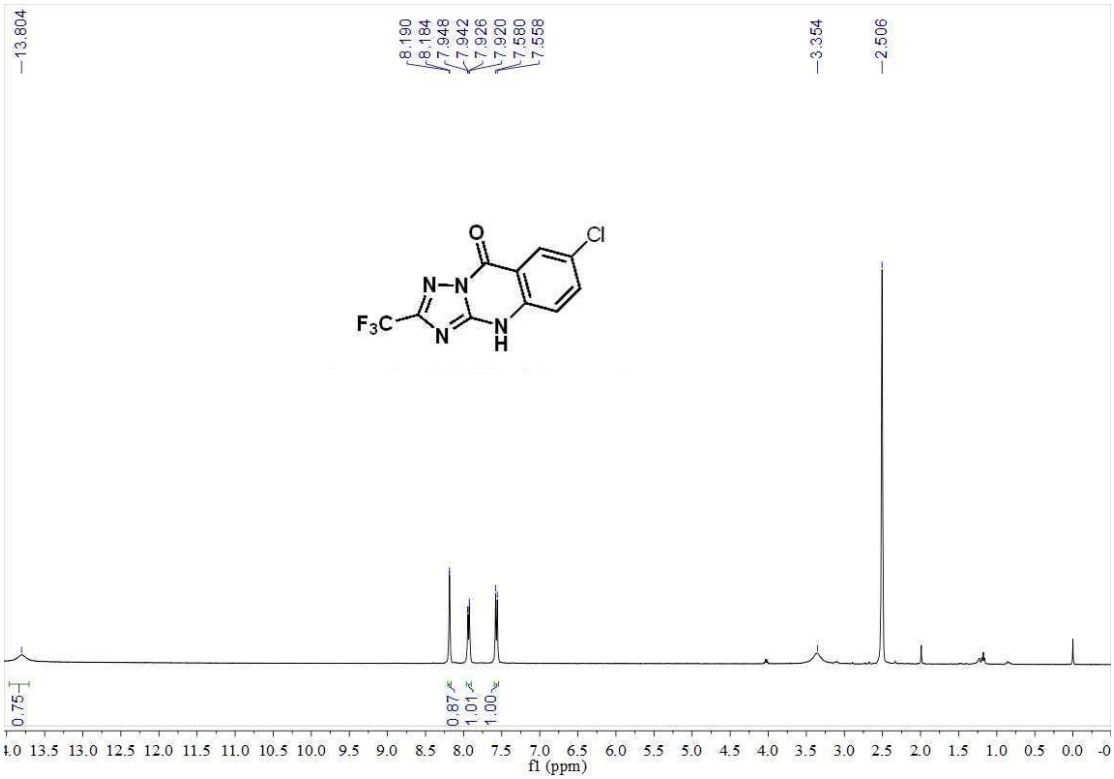


HYL-LIUXIAOLING-4I#24-99 RT: 0.10657-0.44084 AV: 76
T: FTMS + p ESI Full lock ms [50.0000-750.0000]
m/z= 291.22275-300.06994

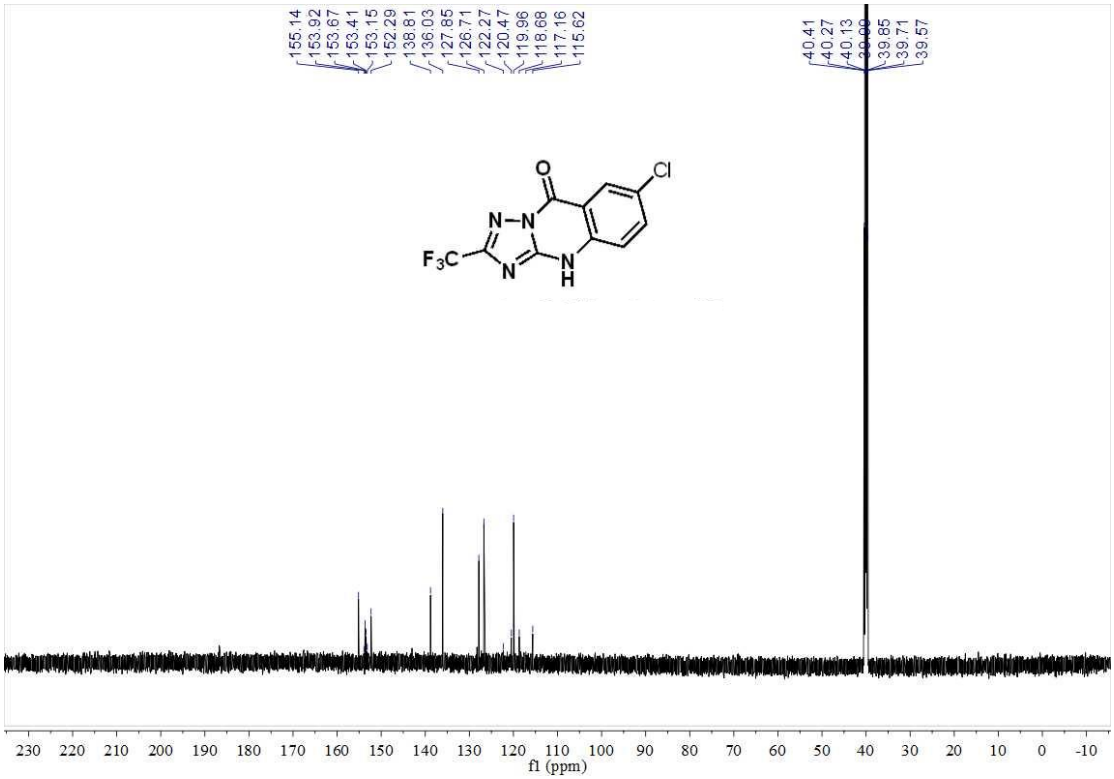
m/z	Intensity	Relative	Resolution	Charge	Delta (ppm)	Composition
295.02132	73694120.0	100.00	65287.43	0.00	-0.09	$C_{10}H_4ON_4F_4Na$
295.08428	11588387.0	15.72	62423.29	0.00		
296.02491	7437359.0	10.09	63929.43	0.00		
297.08228	1808216.0	2.45	68745.58	0.00		
297.12101	3358921.8	4.56	64802.61	1.00		



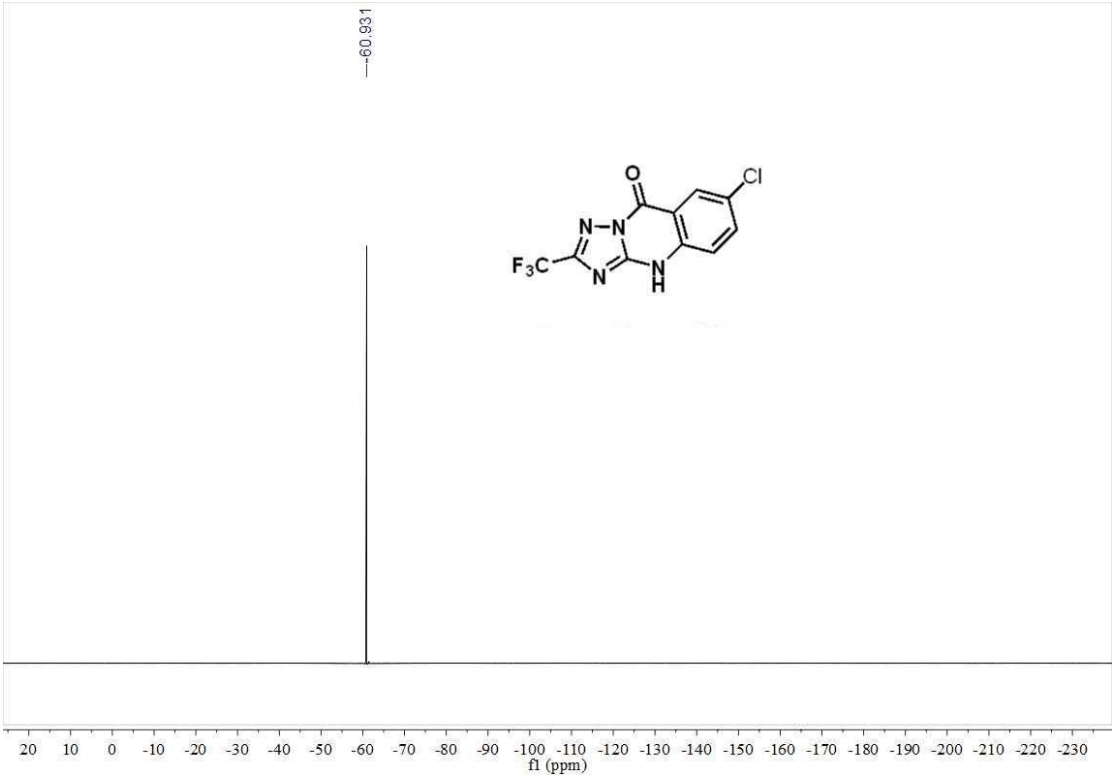
HRMS (ESI) copy of compound **4e**



^{1}H NMR (400 MHz) spectrum of **4f** in DMSO-*d*$_6$



^{13}C{^{1}H} NMR (150 MHz) spectrum of **4f** in DMSO-*d*$_6$



^{19}F NMR (376 MHz) spectrum of **4f** in DMSO-d_6

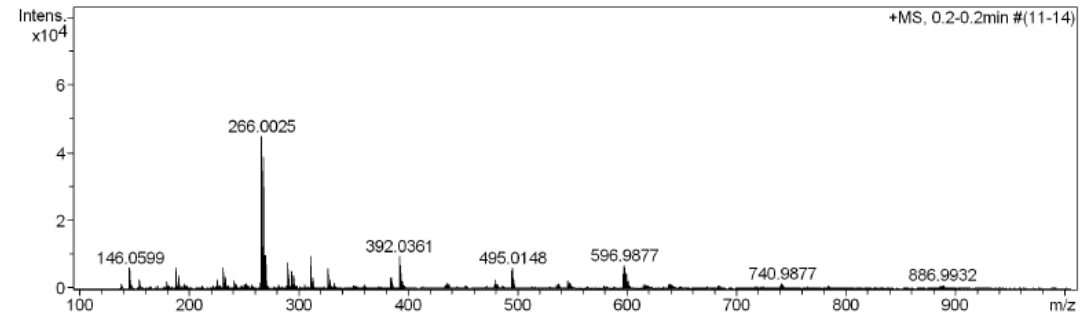
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\user\liuxiaoling20211124-9.d	Acquisition Date	2021-11-24 11:14:23
Method	tune_low.m		
Sample Name	4h	Operator	BDAL@DE
Comment		Instrument / Ser#	micrOTOF-Q 20453

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 ¡ãC
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste



Meas. m/z	#	Formula	m/z	err [ppm]	Mean err [ppm]	rdb	N-Rule	e¡¥Conf	m Sigma	Stdl	Std Mean m/z	Std l VarNorm	Std m/z Diff	Std Comb Dev
310.9925	1	C 10 H 4 Cl F 3 N 4 Na O	310.9918	-2.1	-1.5	8.5	ok	even	7.7	12.5	1.5	5.0	2.7	842.7

HRMS (ESI) copy of compound **4f**

X-ray Crystallographic Data of Compound 2w

Thermal ellipsoids are set at a 50% probability level. Crystal data have been deposited to CCDC, number 2090350.

Crystallization Details

The obtained compound **2w** (20 mg) was dissolved in MeOH (0.1 mL) in a NMR tube at room temperature. Then CH_2Cl_2 (3 mL) was added to the solution slowly along the tube wall, resulting in a two-phase mixture. The colorless crystal of **2w** was formed after the two-phase mixture has diffused.

Experimental

A suitable crystal was selected and placed on a ROD, Synergy Custom system, HyPixdiffractometer. The crystal was kept at 149.98(10) K during data collection. Using Olex2[1], the structure was solved with the SHELXT[2] structure solution program using Intrinsic Phasingand refined with the SHELXL[3] refiement package using Least Squares minimisation.

Crystal structure determination

Crystal Data for $C_7H_{11}F_3N_4O$ (M = 224.20 g/mol): triclinic, space group P-1 (no. 2), a = 5.74722(9) Å, b = 7.76991(12) Å, c = 10.83508(18) Å, α = 81.6145(13) , β = 89.3269(13)°, γ = 86.1828(13)°, V = 477.609(13) Å^3, Z = 2, T = 149.98(10) K, μ (Cu Kα) = 1.309 mm^{-1}, *Dcalc* = 1.559 g/cm^3, 10721 reflections measured (11.536° ≤ 2Θ ≤ 153.942°), 1882 unique (R_{int} = 0.0242, R_{sigma} = 0.0109) which were used in all calculations. The final R_1 was 0.0374 (I > 2σ(I)) and wR_2 was 0.0960 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.

Details:

1. Fixed Uiso

At 1.2 times of:

All C(H) groups

2.a Ternary CH refined with riding coordinates:

C10(H10)

3.b Aromatic/amide H refined with riding coordinates:

C2(H2), C5(H5), C6(H6), C8(H8), C9(H9), C13(H13), C14(H14), C15(H15),

C16(H16), C17(H17)

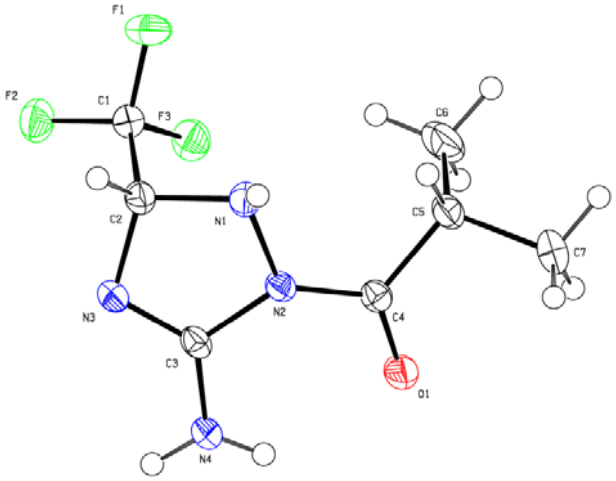


Table S3 Crystallographic Data of Compound 2w

Empirical formula	$C_7H_{11}F_3N_4O$
Formula weight	224.20
Temperature/K	149.98(10)
Crystal system	triclinic
Space group	P-1
a/Å	5.74722(9)
b/Å	7.76991(12)
c/Å	10.83508(18)
α/°	81.6145(13)
β/°	89.3269(13)
γ/°	86.1828(13)
Volume/Å^3	477.609(13)
Z	2
ρ_{calc}g/cm^3	1.559
μ/mm^{-1}	1.309
F(000)	232.0
Crystal size/mm^3	0.18 × 0.15 × 0.12
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	11.536 to 153.942
Index ranges	$-6 \le h \le 7$, $-9 \le k \le 9$, $-13 \le l \le 12$
Reflections collected	10721
Independent reflections	1882 [Rint = 0.0242, Rsigma = 0.0109]
Data/restraints/parameters	1882/0/146
Goodness-of-fit on F^2	1.089
Final R indexes [I>=2σ (I)]	R1 = 0.0374, wR2 =0.0957
Final R indexes [all data]	R1 = 0.0380, wR2 = 0.0960
Largest diff. peak/hole / e Å^{-3}	0.34/-0.36

Table S4 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for Compound 3g. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	x	y	z	U(eq)
$F^{(1)}$	8160.2(17)	1183.6(12)	9357.9(8)	37.3(3)
$F^{(2)}$	7271.7(16)	-1018.2(11)	8499.9(8)	32.9(2)
$F^{(3)}$	4670.6(14)	1051.3(11)	8683.3(7)	30.0(2)
$O^{(1)}$	1308.4(16)	5059.1(13)	6035.8(9)	26.6(2)
$N^{(1)}$	6987.1(18)	3544.8(14)	7165.2(10)	21.5(3)
$N^{(2)}$	4864.1(18)	3766.1(13)	6460.8(10)	18.3(2)
$N^{(3)}$	6391.3(19)	1087.8(14)	6170.7(10)	21.2(3)
$N^{(4)}$	3075(2)	2220.0(15)	5008.6(11)	24.3(3)
$C^{(1)}$	6935(2)	709.8(17)	8438.5(13)	24.0(3)
$C^{(2)}$	7641(2)	1679.2(16)	7174.6(12)	20.9(3)
$C^{(3)}$	4749(2)	2280.8(16)	5842.0(11)	19.0(3)
$C^{(4)}$	3108(2)	4970.3(16)	6651.2(11)	18.7(3)
$C^{(5)}$	3427(2)	6132.2(17)	7633.8(13)	24.1(3)
$C^{(6)}$	2464(4)	5210(2)	8860.9(14)	43.6(4)
$C^{(7)}$	2138(3)	7899.4(18)	7254.9(14)	30.1(3)

Table S5 Anisotropic Displacement Parameters ($Å^2×10^3$) for Compound 3g. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\cdots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
$F^{(1)}$	48.1(6)	40.2(5)	24.2(5)	-6.5(4)	-13.5(4)	-0.4(4)
$F^{(2)}$	43.0(5)	22.1(4)	32.0(5)	-1.6(3)	1.7(4)	4.6(3)
$F^{(3)}$	30.9(4)	32.1(5)	25.8(4)	-2.9(3)	7.4(3)	2.1(3)
$O^{(1)}$	22.8(5)	29.2(5)	29.6(5)	-12.2(4)	-6.5(4)	3.3(4)
$N^{(1)}$	17.9(5)	20.1(5)	27.5(6)	-6.2(4)	-4.1(4)	-1.0(4)
$N^{(2)}$	18.7(5)	18.6(5)	18.6(5)	-5.7(4)	-1.2(4)	-1.5(4)
$N^{(3)}$	25.7(6)	20.5(5)	17.9(5)	-6.1(4)	0.3(4)	0.6(4)
$N^{(4)}$	29.0(6)	21.5(6)	23.8(6)	-9.6(4)	-6.1(5)	3.2(5)
$C^{(1)}$	26.7(7)	23.0(6)	22.5(7)	-6.1(5)	-3.4(5)	3.8(5)
$C^{(2)}$	19.7(6)	21.3(6)	22.5(6)	-6.5(5)	-0.5(5)	1.3(5)
$C^{(3)}$	23.5(6)	18.3(6)	15.9(6)	-4.6(4)	3.9(5)	-2.5(5)
$C^{(4)}$	19.9(6)	18.4(6)	18.1(6)	-3.3(4)	2.0(5)	-2.5(4)
$C^{(5)}$	23.7(6)	24.2(6)	26.9(7)	-11.9(5)	-2.2(5)	0.0(5)
$C^{(6)}$	74.3(13)	36.2(9)	20.9(8)	-9.8(6)	3.2(7)	3.1(8)
$C^{(7)}$	33.3(7)	23.5(7)	35.2(8)	-12.0(6)	4.1(6)	2.0(6)

Table S6 Bond Lengths for Compound 2w.

Atom	Atom	Length/Å
$F^{(1)}$	$C^{(1)}$	1.3347(15)
$F^{(2)}$	$C^{(1)}$	1.3357(15)
$F^{(3)}$	$C^{(1)}$	1.3422(16)
$O^{(1)}$	$C^{(4)}$	1.2298(16)
$N^{(1)}$	$N^{(2)}$	1.4340(14)
$N^{(1)}$	$C^{(2)}$	1.4716(16)
$N^{(2)}$	$C^{(3)}$	1.4222(15)

Atom	Atom	Length/Å
$N^{(3)}$	$C^{(2)}$	1.4554(16)
$N^{(3)}$	$C^{(3)}$	1.2930(17)
$N^{(4)}$	$C^{(3)}$	1.3350(17)
$C^{(1)}$	$C^{(2)}$	1.5277(19)
$C^{(4)}$	$C^{(5)}$	1.5130(17)
$C^{(5)}$	$C^{(6)}$	1.532(2)
$C^{(5)}$	$C^{(7)}$	1.5200(19)

Table S7 Bond Angles for Compound 2w.

Atom	Atom	Atom	Angle/°
$N^{(2)}$	$N^{(1)}$	$C^{(2)}$	101.91(9)
$C^{(3)}$	$N^{(2)}$	$N^{(1)}$	107.36(9)
$C^{(4)}$	$N^{(2)}$	$N^{(1)}$	122.60(10)
$C^{(4)}$	$N^{(2)}$	$C^{(3)}$	128.06(11)
$C^{(3)}$	$N^{(3)}$	$C^{(2)}$	105.90(10)
$F^{(1)}$	$C^{(1)}$	$F^{(2)}$	107.59(11)
$F^{(1)}$	$C^{(1)}$	$F^{(3)}$	107.34(11)
$F^{(1)}$	$C^{(1)}$	$C^{(2)}$	110.89(11)
$F^{(2)}$	$C^{(1)}$	$F^{(3)}$	106.87(11)
$F^{(2)}$	$C^{(1)}$	$C^{(2)}$	112.42(11)
$F^{(3)}$	$C^{(1)}$	$C^{(2)}$	111.48(11)
$N^{(1)}$	$C^{(2)}$	$C^{(1)}$	107.85(10)

Atom	Atom	Atom	Angle/°
$N^{(3)}$	$C^{(2)}$	$N^{(1)}$	108.16(10)
$N^{(3)}$	$C^{(2)}$	$C^{(1)}$	110.61(10)
$N^{(3)}$	$C^{(3)}$	$N^{(2)}$	113.16(11)
$N^{(3)}$	$C^{(3)}$	$N^{(4)}$	126.38(12)
$N^{(4)}$	$C^{(3)}$	$N^{(2)}$	120.46(11)
$N^{(4)}$	$C^{(3)}$	$N^{(2)}$	120.46(11)
$N^{(4)}$	$C^{(3)}$	$N^{(2)}$	120.46(11)
$N^{(2)}$	$C^{(4)}$	$C^{(5)}$	118.66(11)
$C^{(4)}$	$C^{(5)}$	$C^{(6)}$	107.18(11)
$C^{(4)}$	$C^{(5)}$	$C^{(7)}$	110.03(11)
$C^{(7)}$	$C^{(5)}$	$C^{(6)}$	111.01(12)

Table S8 Torsion Angles for Compound 2w.

A	B	C	D	Angle/°
$F^{(1)}$	$C^{(1)}$	$C^{(2)}$	$N^{(1)}$	-65.01(13)
$F^{(1)}$	$C^{(1)}$	$C^{(2)}$	$N^{(3)}$	176.90(10)
$F^{(2)}$	$C^{(1)}$	$C^{(2)}$	$N^{(1)}$	174.51(10)
$F^{(2)}$	$C^{(1)}$	$C^{(2)}$	$N^{(3)}$	56.41(14)
$F^{(3)}$	$C^{(1)}$	$C^{(2)}$	$N^{(1)}$	54.53(14)
$F^{(3)}$	$C^{(1)}$	$C^{(2)}$	$N^{(3)}$	-63.56(14)
$O^{(1)}$	$C^{(4)}$	$C^{(5)}$	$C^{(6)}$	88.08(16)
$O^{(1)}$	$C^{(4)}$	$C^{(5)}$	$C^{(7)}$	-32.72(17)
$N^{(1)}$	$N^{(2)}$	$C^{(3)}$	$N^{(3)}$	6.79(14)
$N^{(1)}$	$N^{(2)}$	$C^{(3)}$	$N^{(4)}$	-172.73(11)
$N^{(1)}$	$N^{(2)}$	$C^{(4)}$	$O^{(1)}$	-178.11(11)
$N^{(1)}$	$N^{(2)}$	$C^{(4)}$	$C^{(5)}$	0.76(17)
$N^{(2)}$	$N^{(1)}$	$C^{(2)}$	$N^{(3)}$	18.84(12)

A	B	C	D	Angle/°
$N^{(2)}$	$N^{(1)}$	$C^{(2)}$	$C^{(1)}$	-100.81
$N^{(2)}$	$C^{(4)}$	$C^{(5)}$	$C^{(6)}$	-90.75(15)
$N^{(2)}$	$C^{(4)}$	$C^{(5)}$	$C^{(7)}$	148.44(12)
$C^{(2)}$	$N^{(1)}$	$N^{(2)}$	$C^{(3)}$	-15.38(12)
$C^{(2)}$	$N^{(1)}$	$N^{(2)}$	$C^{(4)}$	149.80(11)
$C^{(2)}$	$N^{(3)}$	$C^{(3)}$	$N^{(2)}$	5.50(14)
$C^{(2)}$	$N^{(3)}$	$C^{(3)}$	$N^{(4)}$	-175.01(12)
$C^{(3)}$	$N^{(2)}$	$C^{(4)}$	$O^{(1)}$	-16.17(19)
$C^{(3)}$	$N^{(2)}$	$C^{(4)}$	$C^{(5)}$	162.70(12)
$C^{(3)}$	$N^{(3)}$	$C^{(2)}$	$N^{(1)}$	-15.57(13)
$C^{(3)}$	$N^{(3)}$	$C^{(2)}$	$C^{(1)}$	102.33(12)
$C^{(4)}$	$N^{(2)}$	$C^{(3)}$	$N^{(3)}$	-157.33(12)
$C^{(4)}$	$N^{(2)}$	$C^{(3)}$	$N^{(4)}$	23.15(19)

Table S9 Hydrogen Atom Coordinates ($Å \times 10^4$) and Isotropic Displacement Parameters ($Å^2 \times 10^3$) for Compound 2w.

Atom	x	y	z	U(eq)
$H^{(1)}$	8064.77	4271.4	6913.06	26
$H^{(4A)}$	2010(30)	3010(20)	4910(16)	26(4)
$H^{(4B)}$	3010(30)	1240(30)	4676(18)	33(5)
$H^{(2)}$	9360.96	1496.85	7048.24	25
$H^{(5)}$	5125.33	6288.54	7730.64	29
$H^{(6A)}$	826.75	4986.46	8748.85	65
$H^{(6B)}$	2577.71	5950.91	9513.18	65
$H^{(6C)}$	3369.98	4101.1	9105.85	65
$H^{(7A)}$	2762.02	8451.91	6459.87	45
$H^{(7B)}$	2346.88	8640.47	7898.42	45
$H^{(7C)}$	473.66	7745.15	7162.18	45