Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Intermolecular binding preferences of haloethynyl halogen-bond donors as a function of molecular electrostatic potentials in a family of N-(pyridin-2-yl)amides

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1. X-ray crystallography results
2. CSD search on ethynyl halo compounds
3. ^{1}H and ^{13}C NMR data

1. X-ray crystallography data

Code	**Bz-act-Cl**	**Bz-act-Br**	**Bz-act-I(Form 1)**	**Bz-act-I(Form 2)**
Formula moiety	$C_{14}H_9ClN_2O$	$C_{14}H_9BrN_2O$	$C_{14}H_9IN_2O$	$C_{14}H_9IN_2O$
Empirical formula	$C_{14}H_9ClN_2O$	$C_{14}H_9BrN_2O$	$C_{14}H_9IN_2O$	$C_{14}H_9IN_2O$
Molecular weight	256.68	301.14	348.13	348.13
Color, Habit	Colorless, Plate	Light yellow, Plate	Yellow, Block	Colorless, Plate
Crystal system	Monoclinic	Monoclinic	Monoclinic	Monoclinic
Space group, Z	P_12_1/c_1, 4	P_12_1/c_1, 4	P_12_1/c_1, 4	P_12_1/c_1, 4
a, Å	5.10890(10)	5.19740(10)	4.08450(10)	5.25520(10)
b, Å	22.0087(4)	21.9187(5)	16.1979(4)	15.3923(3)
c, Å	10.5605(2)	10.6585(2)	19.1245(4)	16.4781(3)
α, º	90	90	90	90
β, º	99.571(2)	100.577(2)	95.784(2)	98.244(2)
γ, º	90	90	90	90
Volume, Å^3	1170.90(4)	1193.59(4)	1258.84(5)	1319.13(4)
Density, g/cm^3	1.456	1.676	1.837	1.753
T, ºK	100.00(10)	100.01(10)	293(2)	200.00(10)
Crystal size, min x mid x max	0.028 x 0.079 x 0.168	0.037 x 0.051 x 0.221	0.022 x 0.029 x 0.043	0.024 x 0.11 x 0.277
X-ray wavelength, Å	1.54184	1.54184	1.54184	0.71073
μ, mm^{-1}	2.786	4.596	19.896	2.416
Trans min / max	0.89283/ 1.00000	0.75134/ 1.00000	0.768/ 0.838	0.68484/ 1.00000
θ_{min}, º	4.017	4.034	3.584	2.498
θ_{max}, º	75.387	75.059	72.202	33.573
Reflections				
collected	8652	8888	8164	25130
independent	2241	2304	2361	4674
observed	2101	2181	2183	4037
R_{int}	0.0218	0.0302	0.0262	0.0239
Threshold expression	> 2σ(I)	> 2σ(I)	> 2σ(I)	> 2σ(I)
No. parameters	199	199	163	163
No. restraints	0	0	0	0
R_1 (observed)	0.0259	0.0244	0.0336	0.0261
wR_2 (all)	0.0683	0.0671	0.0828	0.0732
Goodness of fit (all)	1.063	1.058	1.110	1.078
ρ_{max}, ρ_{min}, e Å^{-3}	0.236, -0.209	0.337, -0.706	1.077, -0.817	1.385, -0.904
Completeness to 2θ limit	0.920	0.932	0.955	0.899

Code	**2act-Cl**	**2act-Br**	**3act-Cl**	**3act-I**
Formula moiety	$C_{13}H_8ClN_3O$	$C_{13}H_8BrN_3O$	$C_{13}H_8ClN_3O$	$C_{13}H_8IN_3O$
Empirical formula	$C_{13}H_8ClN_3O$	$C_{13}H_8BrN_3O$	$C_{13}H_8ClN_3O$	$C_{13}H_8IN_3O$
Molecular weight	257.67	302.13	257.67	349.12
Color, Habit	Colorless, Plate	Colorless, Needle	Yellow, needle	Yellow, Block
Crystal system	Monoclinic	Monoclinic	Monoclinic	Monoclinic
Space group, *Z*	P_12_1/c_1, 4	P_12_1/c_1, 4	P_12_1/c_1, 4	P_12_1/c_1, 4
a, Å	3.79810(10)	3.8797(2)	3.84600(10)	12.7284(3)
b, Å	11.5441(2)	21.3190(14)	9.8013(2)	6.7266(2)
c, Å	25.4972(4)	14.8922(9)	29.8606(6)	14.4741(4)
α, º	90	90	90	90
β, º	91.106(2)	95.148(5)	93.485(2)	104.558(3)
γ, º	90	90	90	90
Volume, Å^3	1117.73(4)	1226.79(13)	1123.54(4)	1199.47(6)
Density, g/cm^3	1.531	1.636	1.523	1.933
T, ºK	100.00(18)	180.00(10)	99.99(10)	293(2)
Crystal size, min x mid x max	0.154 x 0.295 x 0.518	0.032 x 0.046 x 0.143	0.011 x 0.036 x 0.106	0.041 x 0.065 x 0.095
X-ray wavelength, Å	1.54184	1.54184	1.54184	1.54184
μ, mm^{-1}	2.948	4.499	2.933	20.908
Trans min / max	0.62229/ 1.00000	0.70697/ 1.00000	0.64902/ 1.00000	0.41004/ 0.46137
θ_{min}, º	3.467	3.630	2.965	3.588
θ_{max}, º	77.261	77.529	77.222	67.057
Reflections				
collected	8420	7916	11353	8148
independent	2342	2542	2305	2136
observed	2199	2208	2235	1957
R_{int}	0.0273	0.0509	0.0245	0.0370
Threshold expression	> 2σ(*I*)	> 2σ(*I*)	> 2σ(*I*)	> 2σ(*I*)
No. parameters	163	163	163	163
No. restraints	0	0	0	0
R_1 (observed)	0.0385	0.0595	0.0416	0.0359
wR_2 (all)	0.1059	0.1540	1174	0.1013
Goodness of fit (all)	1.074	1.099	1.046	1.091
ρ_{max}, ρ_{min}, e Å^{-3}	0.607, -0.374	0.938, -1.707	0.538, -0.504	1.181, -1.580
Completeness to 2θ limit	0.984	0.981	0.969	0.999

Code	**4act-Cl**	**4act-Br**	**4act-I**
Formula moiety	$C_{13}H_8ClN_3O$	$C_{13}H_8BrN_3O$	$C_{13}H_8IN_3O$
Empirical formula	$C_{13}H_8ClN_3O$	$C_{13}H_8BrN_3O$	$C_{13}H_8IN_3O$
Molecular weight	257.67	302.13	349.12
Color, Habit	Colorless, Plate	Yellow, Irregular	Yellow, Block
Crystal system	Monoclinic	Triclinic	Monoclinic
Space group, *Z*	$P_12_1/c_{1,}4$	P -1, 2	C_1 2/c $_{1,}$ 8
a, Å	3.77350(10)	6.75090(10)	10.2100(2)
b, Å	10.1396(2)	9.2249(2)	13.0373(2)
c, Å	29.0994(6)	10.2422(2)	18.7180(3)
α, º	90	112.512(2)	90
β, º	90.876(2)	93.859(2)	103.054(2)
γ, º	90	97.720(2)	90
Volume, Å^3	1113.26(4)	578.97(2)	2427.18(7)
Density, g/cm^3	1.537	1.733	1.911
T, ºK	100.00(10)	99.99(10)	100.00(10)
Crystal size, min x mid x max	0.031 x 0.045 x 0.16	0.06 x 0.166 x 0.77	0.02 x 0.04 x 0.1
X-ray wavelength, Å	1.54184	1.54184	0.71073
μ, mm^{-1}	2.960	4.766	2.629
Trans min / max	0.87153 / 1.00000	0.82297/ 1.00000	0.46703/ 1.00000
θ_{min}, º	3.038	4.713	2.234
θ_{max}, º	76.514	74.953	33.419
Reflections			
collected	3178	8710	15079
independent	3178	2274	4104
observed	2965	2241	3962
R_{int}		0.0294	0.0742
Threshold expression	> 2σ(*I*)	> 2σ(*I*)	> 2σ(*I*)
No. parameters	165	163	164
No. restraints	0	0	0
R_1 (observed)	0.0348	0.0244	0.0348
wR_2 (all)	0.0910	0.0648	0.0973
Goodness of fit (all)	1.064	1.069	1.180
ρ_{max}, ρ_{min}, e Å^{-3}	0.329, -0.287	0.850, -0.482	1.842, -2.296
Completeness to 2θ limit	0.922	0.951	0.867

2. CSD search on ethynyl halo compounds

	Refcode	*Compound Name (L)*	Binding site		
			O	$N_{(pyr)}$	π
1	KORNOX	1-(6-bromohexa-1,3,5-triyn-1-yl)-4-nitrobenzene	1	na	0
2	KORPEP	1-(4-bromobuta-1,3-diyn-1-yl)-4-nitrobenzene	1	na	0
3	KORPOZ	1-[4-(6-bromohexa-1,3,5-triyn-1-yl)phenyl]ethan-1-one	1	na	0
4	KORQAM	1-[4-(4-bromobuta-1,3-diyn-1-yl)phenyl]ethan-1-one	1	na	0
5	BCACEN	Bromo-cyanoacetylene	na	1	0
6	BISBAL	1-(Bro[illegible]zene	na	na	1
7	KORPUF	3-[4-(bromoethynyl)phenyl]-4-hydroxypent-3-en-2-one	0	na	0
8	HEHTOF	1,3-bis(bromoethynyl)bicyclo[1.1.1]pentane	na	na	1
9	HEVWAI01	2-(Acetoxymethyl)-6-((5-bromopenta-2,4-diyn-1-yl)oxy)tetrahydro-2H-pyran-3,4,5-triyl triacetate	1	na	0
10	KAMXII	t-butyl (5-bromopenta-2,4-diyn-1-yl)carbamate	1	na	0
11	MIDVUT	{[4-(bromoethynyl)bicyclo[2.2.2]octan-1-yl]ethynyl}(trimethyl)silane	na	na	1
12	MIDWAA	1,4-bis(bromoethynyl)bicyclo[2.2.2]octane	na	na	1
13	NAMKER	6-(bromoethynyl)-5,6-dimethoxy-7-oxabicyclo[4.1.0]hept-3-en-2-one	1	na	0
14	NIDWAA	Diethyl 2,6-bis(bromoethynyl)-4,8-dimethoxy-1,2,3,5,6,7-hexahydro-s-indacene-2,6-dicarboxylate	1	na	0
15	PUWPON	6,13-bis(Bromoethynyl)-6,13-dimethoxy-6,13-dihydropentacene	0	na	1
16	QOFLOS	1,4-bis(Bromoethynyl)benzene	na	na	1
17	QOLMIN	1,3,5-tris(Bromoethynyl)benzene	na	na	1
18	RADCII	(E)-1,6-Dibromo-3,4-bis((tri-isopropylsilyl)ethynyl)hex-3-ene-1,5-diyne	na	na	0
19	RENQUX	1,6-Anhydro-4-C-(2-bromoethynyl)-4-deoxy-beta-D-glucopyranose	1	na	0
20	TOVNEZ	2-(bromoethynyl)-2-(hydroxymethyl)-6-methylenecyclohexanol	0	na	0
21	VEJQUX	2,3-bis(Bromoethynyl)-1,4-dimethoxybenzene	0	na	1

(1=interaction formed by halogen, na=binding site is not available in molecule, 0=interaction not formed by halogen)

a. Ethynylchloro

	Refcode	*Compound Name (L)*	Binding site		
			O	$N_{(pyr)}$	π
1	CIRJET	1-[4-(4-chlorobuta-1,3-diyn-1-yl)phenyl]ethan-1-one	1	na	0
2	CCACEN	Chloro-cyanoacetylene	na	1	0
3	CIPJAP	3-[4-(chloroethynyl)phenyl]-4-hydroxypent-3-en-2-one	0	na	0
4	CLPOBZ	1,4-bis(5-Chloro-pent-4-ynyloxy)benzene	0	na	1
5	ECOLOA	N-(chloroethynyl)-4-methyl-N-phenylbenzene-1-sulfonamide	1	na	0
6	HCLENE10	2alpha,17alpha-bis(2-Chloroethynyl)-A-nor-5alpha-estrane-2beta,17beta-diol methanol solvate	0	na	0
7	NICGEK	2-Chloroethynyl-adamantan-2-ol	0	na	0
8	NIDWII	(Z)-Diethyl 2,6-bis(chloroethynyl)-4,8-dimethoxy-1,2,3,5,6,7-hexahydro-s-indacene-2,6-dicarboxylate	1	na	0
9	UGEXUZ	(2R,4aS,10aR)-4a-Benzyl-2-(chloroethynyl)-1,2,3,4,4a,9,10,10a-octahydro-phenanthrene-2,7-diol	0	na	1
10	XAXNOG	t-butyl (5-chloropenta-2,4-diyn-1-yl)carbamate	1	na	0
		Total	4	1	2

		Total	9	1	8

b. Ethynylbromo

c. Ethynyl iodo

	Refcode	Compound Name (L)	Binding site			Other
			O	N	π	
1	KORNUD	1-(4-iodobuta-1,3-diyn-1-yl)-4-nitrobenzene	1	na	0	
2	KORPAL	4-(6-iodohexa-1,3,5-triyn-1-yl)benzonitrile	na	1	0	
3	KORPIT	1,2,3,4,5-pentafluoro-6-(6-iodohexa-1,3,5-triyn-1-yl)benzene	0	0	0	to flourine
4	KORQEQ	4-(8-iodoocta-1,3,5,7-tetrayn-1-yl)benzonitrile	na	1	0	
5	KORQIU	1-fluoro-4-(4-iodobuta-1,3-diyn-1-yl)benzene	0	0	0	to iodine
6	KORQOA	4-(4-iodobuta-1,3-diyn-1-yl)benzonitrile	0	1	0	
7	QOWQOL	1-ethynyl-3,3-bis(trifluoromethyl)-1,3-dihydro-1,2-benziodoxole	1	na	0	
8	AVIYEK	1,4-bis(iodoethynyl)bicyclo[2.2.2]octane	na	na	1	
9	BOBGUV	1,4-bis(Iodoethynyl)benzene	na	na	1	
10	CARGEJ	1-((Tri-isopropylsilyl)ethynyl)-1lambda$3!,2-benziodoxol-3(1H)-one	1	na	0	
11	CARGEJ03	1-{[tris(propan-2-yl)silyl]ethynyl}-1,2-benziodoxol-3(1H)-one	1	na	0	
12	CARGIN	1-((Trimethylsilyl)ethynyl)-1,2-benziodoxol-3(1H)-one	1	na	0	
13	CARGOT	1-((Triethylsilyl)ethynyl)-1,2-benziodoxol-3(1H)-one	1	na	0	
14	CARGUZ	1-((tert-Butyl(dimethyl)silyl)ethynyl)-1,2-benziodoxol-3(1H)-one	1	na	0	
15	CARHEK	5-Methyl-1-((triisopropylsilyl)ethynyl)-1,2-benziodoxol-3(1H)-one	1	na	0	
16	CARHIO	5,6-Dimethoxy-1-((triisopropylsilyl)ethynyl)-1,2-benziodoxol-3(1H)-one	1	na	0	
17	CARHOU	4-Methyl-1-((triisopropylsilyl)ethynyl)-1,2-benziodoxol-3(1H)-one	1	na	0	
18	CARHUA	7-Methyl-1-((triisopropylsilyl)ethynyl)-1,2-benziodoxol-3(1H)-one	1	na	0	
19	CARJAI01	1-(Phenylethynyl)-1lambda$3!,2-benziodoxol-3(1H)-one	1	na	0	
20	CIKZOO	1,4-bis{[4-(iodoethynyl)phenyl]ethynyl}bicyclo[2.2.2]octane	na	na	1	
21	CINWEE	4-hydroxy-3-[4-(iodoethynyl)phenyl]pent-3-en-2-one	1	na	0	
22	DAHVAM	t-butyl (4-(iodoethynyl)phenyl)carbamate	0	na	1	
23	DIACET	Di-iodoacetylene	na	na	1	
24	DOJPEA	2-{[(7,7-dimethyl-2-oxobicyclo[2.2.1]heptan-1-yl)methyl]sulfonyl}-1-{[tris(propan-2-yl)silyl]ethynyl}-1,2-dihydro-3H-1,2-benziodazol-3-one unknown solvate	1	na	0	
25	DOJPIE	2-[(4-methylphenyl)sulfonyl]-1-{[tris(propan-2-yl)silyl]ethynyl}-1,2-dihydro-3H-1,2-benziodazol-3-one	1	na	0	
26	EQUDIF	2,6-bis(iodoethynyl)pyridine	na	1	0	
27	FABWAJ	1-bromo-4-(iodoethynyl)benzene	na	na	1	
28	FABWOX	1-chloro-4-(iodoethynyl)benzene	na	na	1	
29	GUFBOY	11-((t-Butyldiphenylsiloxy)methyl)-2,7-bis(2-iodoethynyl)-1-methylidene-spiro(5.5)undecan-2-ol	1	na	0	
30	HEHTUL	1,3-bis(iodoethynyl)bicyclo[1.1.1]pentane	na	na	1	
31	HOWWUN	1-(iodoethynyl)-4-methoxybenzene	0	na	1	
32	HOWXAU	4-(iodoethynyl)benzonitrile	na	1	0	
33	HOWXEY	1,2-bis(iodoethynyl)-4,5-dimethoxybenzene	1	na	0	
34	HOWXIC	1-(4-(iodoethynyl)phenyl)ethanone	1	na	0	
35	IBUYAI	3-iodoprop-2-ynal	1	na	0	
36	IBUYOW	4-(iodoethynyl)benzaldehyde	1	na	0	
37	IBUYUC	2,3,5,6-tetrafluoro-4-(iodoethynyl)benzaldehyde	1	na	0	
38	ICACEN	Iodo-cyano-acetylene	na	1	0	
39	IFOGUH	1,3,5-Trifluoro-2,4,6-tris(iodoethynyl)benzene	na	na	0	iodine
40	IYAYUC	2-(7-Fluoro-3,4-dihydro-4-(3-iodoprop-2-ynyl)-3-oxo-2H-1,4-benzoxazin-6-yl)-4,5,6,7-tetrahydro-2H-isoindole-1,3-dione	1	na	0	
41	JOHGEU	3-iodoprop-2-yn-1-yl 2,4,5-trichlorophenyl ether	0	na	1	
42	JOHGEU01	3-iodoprop-2-yn-1-yl 2,4,5-trichlorophenyl ether	0	na	0	chloro
43	KIVBUP	2,6-bis(iodoethynyl)pyrazine	na	1	0	
44	LARKOH	(8-Methoxycyclooct-4-en-1-yl)-bis(iodo)-(iodoethynyl)-tellurium unknown solvate	na	na	na	Te
45	LUNKOW	1-(2,5-Dihydro-1H-pyrrol-1-yl)-3-iodoprop-2-yn-1-one	1	na	0	
46	LUNKUC	3-iodo-1-(piperidin-1-yl)prop-2-yn-1-one	1	na	0	
47	LUNLAJ	3-iodo-1-(morpholin-4-yl)prop-2-yn-1-one	1	na	0	

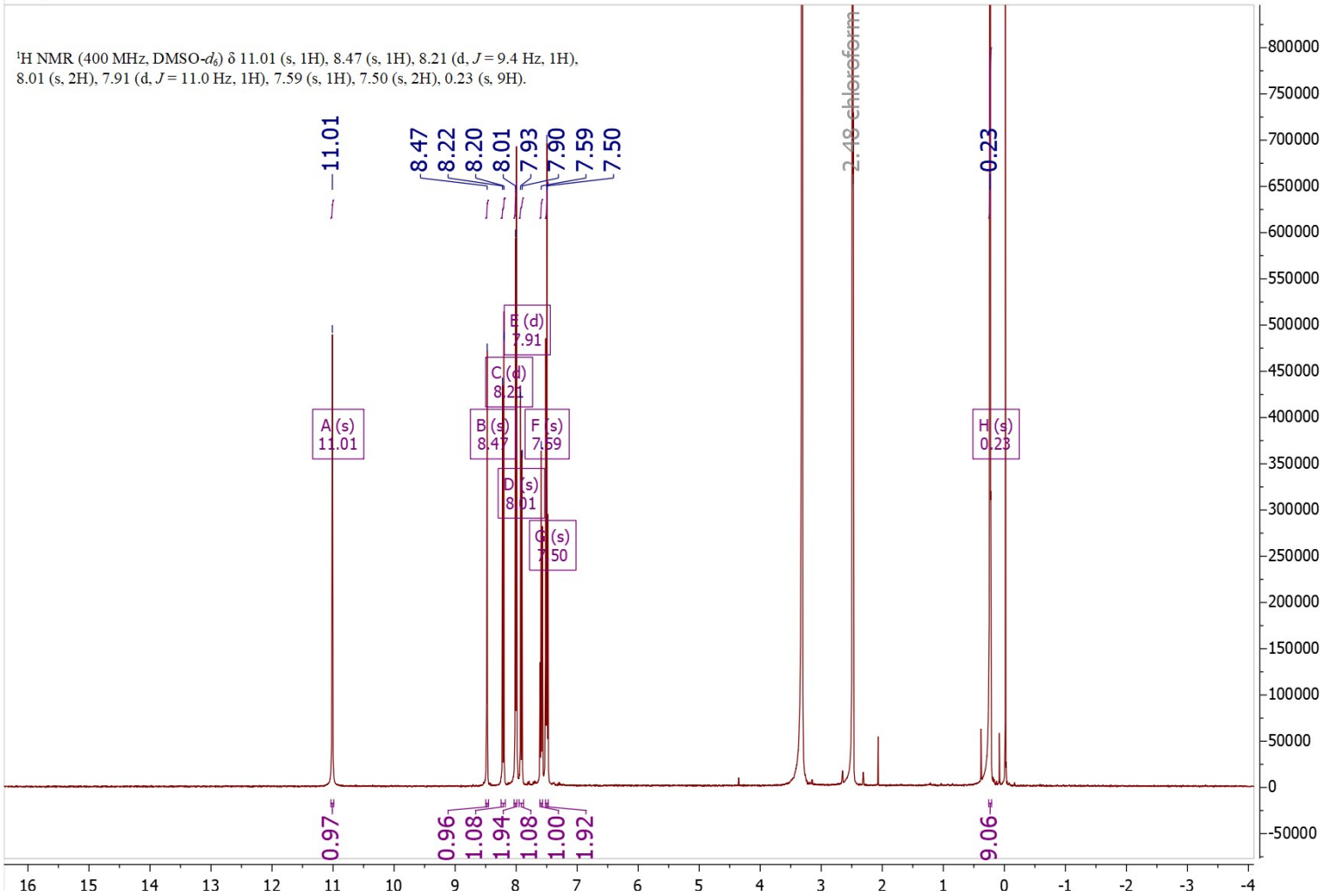
48	LUNLIR	3-iodo-N-methylprop-2-ynamide	1	na	0	
49	LUNLOX	3-iodo-1-(pyrrolidin-1-yl)prop-2-yn-1-one	1	na	0	
50	MIRPUB	3-(iodoethynyl)pyridine	na	1	0	
51	NEZTUJ	(2,2,2-trifluoroacetoxy)-((2-thienyl)ethynyl)-phenyl-iodane	1	na	0	
52	NEZVIZ	((4-Methoxyphenyl)ethynyl)-(phenyl)-(2,2,2-trifluoroacetoxy)-iodane	1	na	0	
53	NEZVOF	Mesityl-(phenylethynyl)-(2,2,2-trifluoroacetoxy)-iodane	1	na	0	
54	NEZVUL	(2-Methoxyphenyl)-(phenylethynyl)-(2,2,2-trifluoroacetoxy)-iodane	1	na	0	
55	NEZWAS	(4-Methoxyphenyl)-(phenylethynyl)-(2,2,2-trifluoroacetoxy)-iodane	1	na	0	
56	NEZWEW	(4-Chlorophenyl)-(phenylethynyl)-(2,2,2-trifluoroacetoxy)-iodane	1	na	0	
57	QUNPEW	5-(iodoethynyl)tricyclo[8.2.2.2$4,7!]hexadeca-1(12),4,6,10,13,15-hexaene	na	na	1	
58	REZQOF	2-(iodoethynyl)quinoline	na	1	0	
59	SEVVIB	2-(iodoethynyl)-1-methyl-1H-imidazole	na	1	0	
60	SEVVUN	1-hexyl-2-(iodoethynyl)-1H-imidazole	na	1	0	
61	SICFES	3-(iodoethynyl)benzoic acid	0	na	1	
62	SOKCUT	3-iodo-N-phenyl-N-[(trifluoromethyl)sulfonyl]prop-2-ynamide	1	na	0	
63	TADKAK	N-(3-Iodo-2-propynyl)-carbazole	na	na	1	
64	TEXTAU	1,4-bis(iodoethynyl)cyclohexane-1,4-diol	0	na	1	
65	TOYPUS	3-Iodo-2-propynyl-N-butylcarbamate	1	0	0	
66	UCUHUY	1-(iodoethynyl)-3,5-dinitrobenzene	1	na	0	
67	UYIRAX	2-amino-5-(iodoethynyl)pyrimidine	na	0	0	iodine
68	VIHFAU	1-(3-Iodopropargyl)imidazole	na	1	0	
69	WAPBUN	(4-(iodoethynyl)phenyl)(diphenyl)phosphine oxide	1	na	0	
70	XAXNUI	t-butyl (5-iodopenta-2,4-diyn-1-yl)carbamate	1	0	0	
71	MUFDAV	(R)-5-((2S,3S,4S,5R)-5-(3-iodoprop-2-yn-1-yl)-3-methyl-4-((trimethylsilyl)ethynyl)tetrahydro-furan-2-yl)-3-methylfuran-2(5H)-one unknown solvate	1	na	0	
72	MUFDEZ	(R)-5-((2R,3S,4R,5S)-5-(3-Iodoprop-2-yn-1-yl)-3-methyl-4-((trimethylsilyl)ethynyl)-tetrahydrofuran-2-yl)-3-methylfuran-2(5H)-one	1	na	0	
73	BULTEK	1-benzyl-3-(3-iodoprop-2-yn-1-yl)-3-methyl-1,3-dihydro-2H-indol-2-one	1	na	0	
74	CUPMEI	25,26,27,28-tetrakis[(3-iodoprop-2-yn-1-yl)oxy]pentacyclo[19.3.1.1$3,7!.1$9,13!.1$15,19!]octacosa-1(25),3(28),4,6,9(27),10,12,15(26),16,18,21,23-dodecaene	0	na	1	

3. ^{1}H and ^{13}C NMR data

3.1 N-(5-((trimethylsilyl)ethynyl)pyridin-2-yl)benzamide, **Bz-TMS**

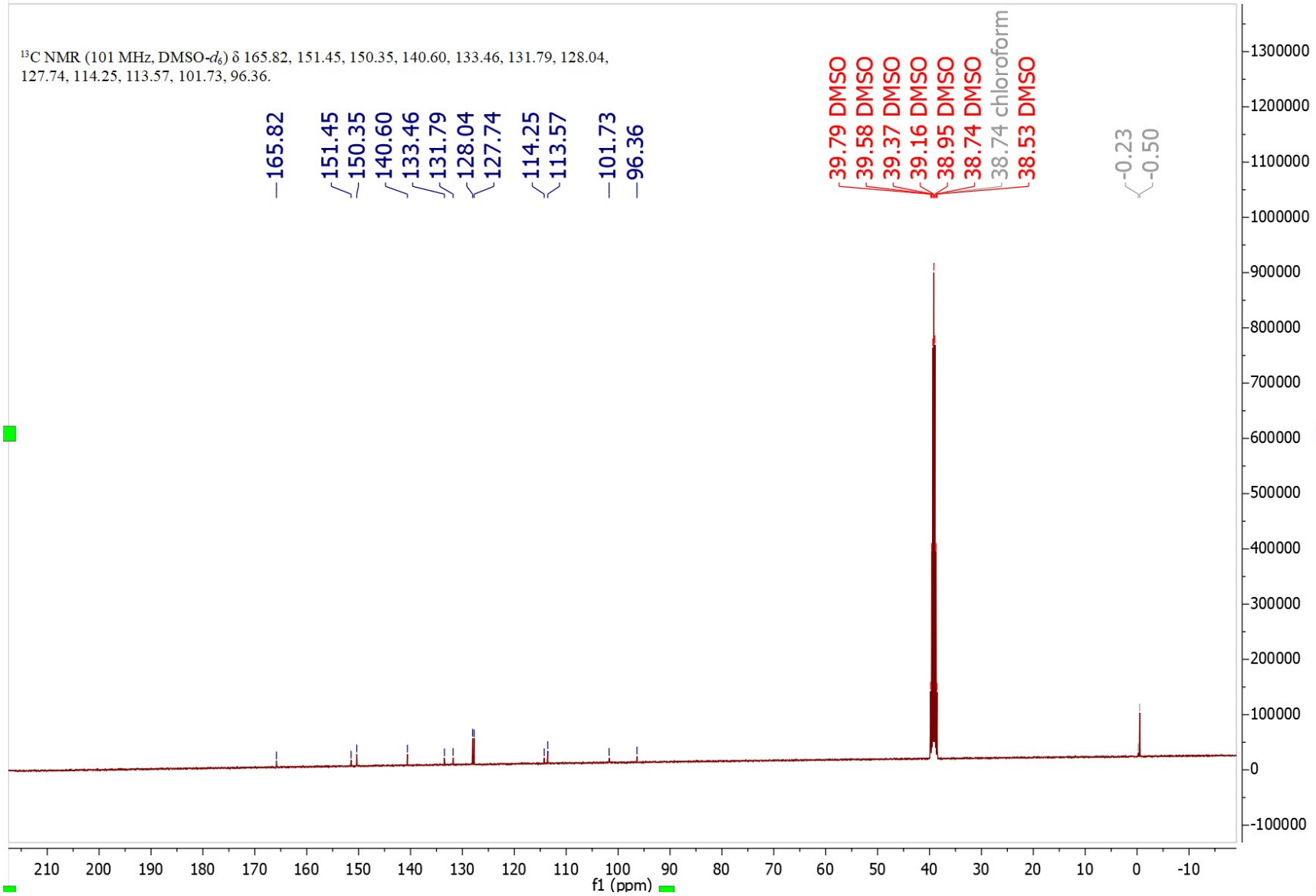
BZ-tms-h.10.fid —

^{1}H NMR (400 MHz, DMSO-d_6) δ 11.01 (s, 1H), 8.47 (s, 1H), 8.21 (d, J = 9.4 Hz, 1H), 8.01 (s, 2H), 7.91 (d, J = 11.0 Hz, 1H), 7.59 (s, 1H), 7.50 (s, 2H), 0.23 (s, 9H).

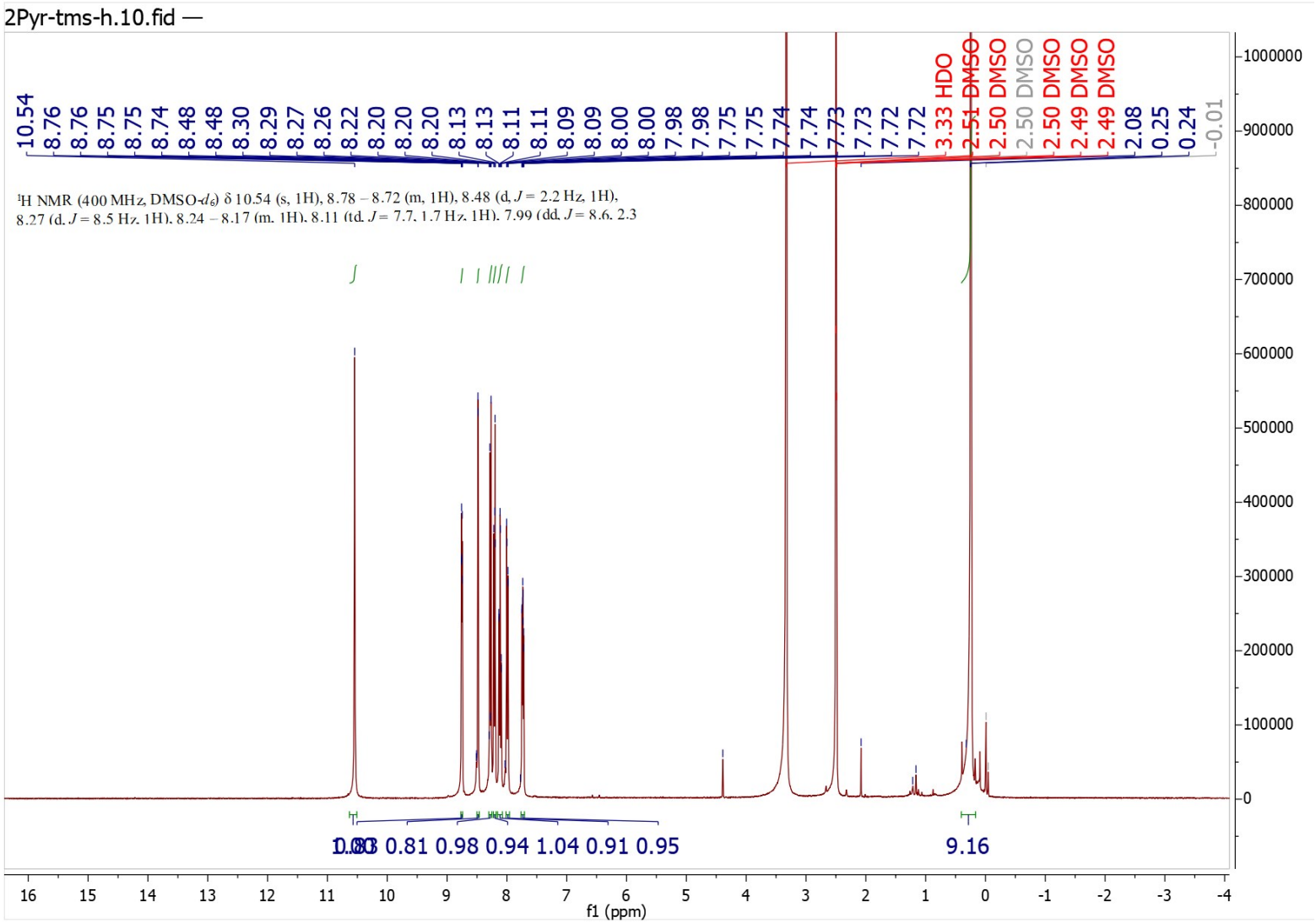


BZ-tms-c13.20.fid —

^{13}C NMR (101 MHz, DMSO-d_6) δ 165.82, 151.45, 150.35, 140.60, 133.46, 131.79, 128.04, 127.74, 114.25, 113.57, 101.73, 96.36.

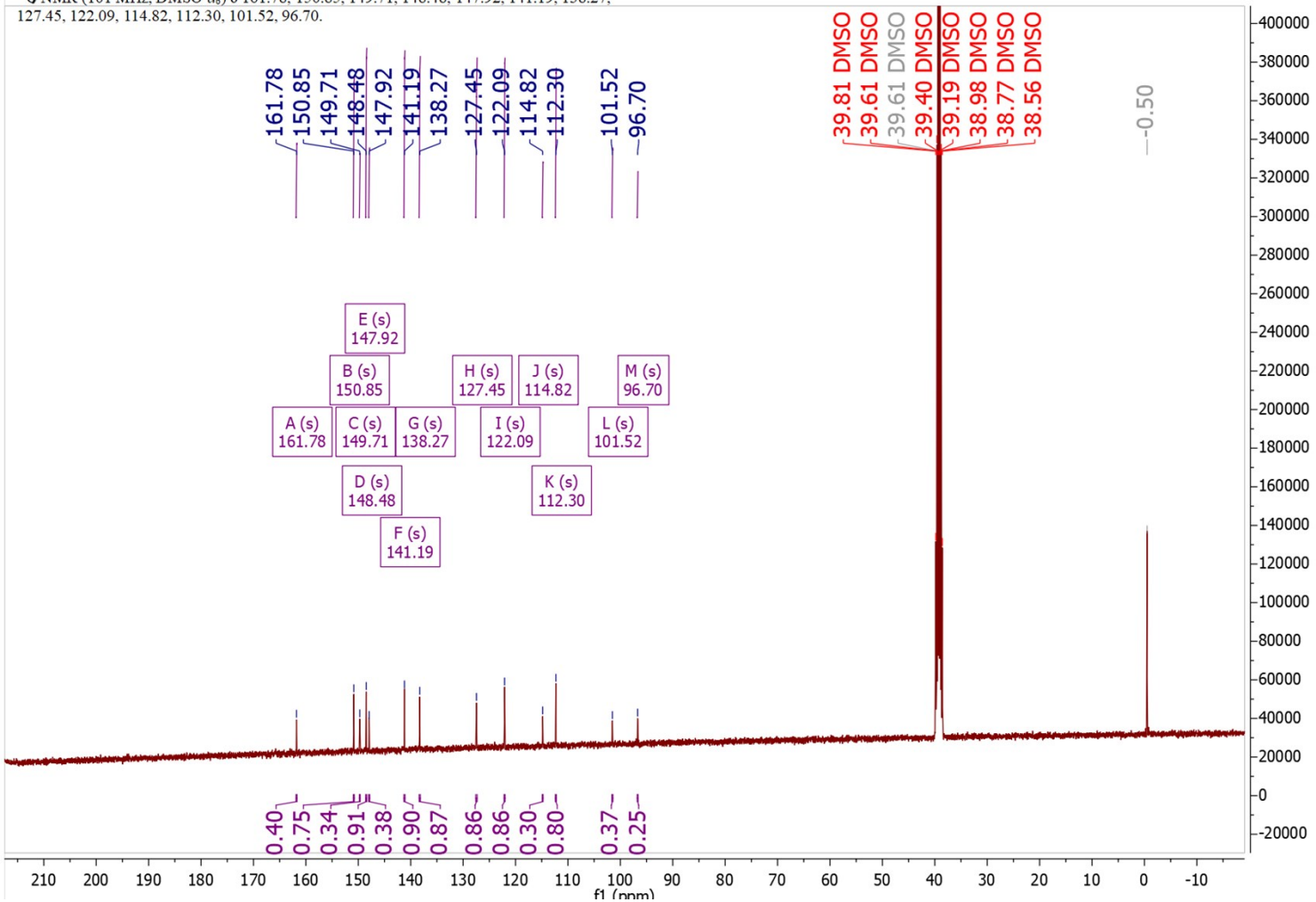


3.2 N-(5-((trimethylsilyl)ethynyl)pyridin-2-yl)picolinamide, **2Pyr-TMS**



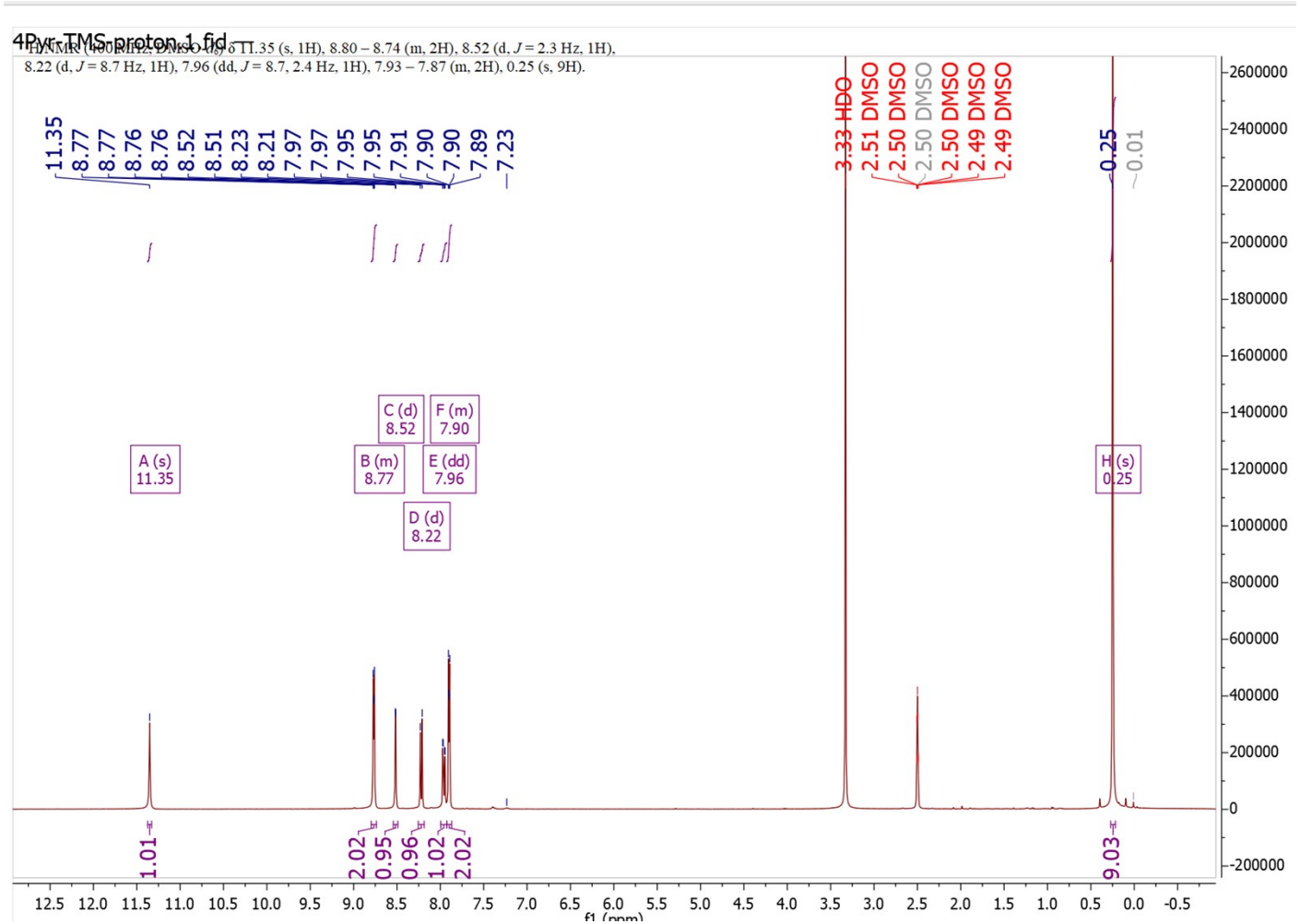
2Pyr-tms-c13 20.fid

^{13}C NMR (101 MHz, DMSO-d_6) δ 161.78, 150.85, 149.71, 148.48, 147.92, 141.19, 138.27, 127.45, 122.09, 114.82, 112.30, 101.52, 96.70.



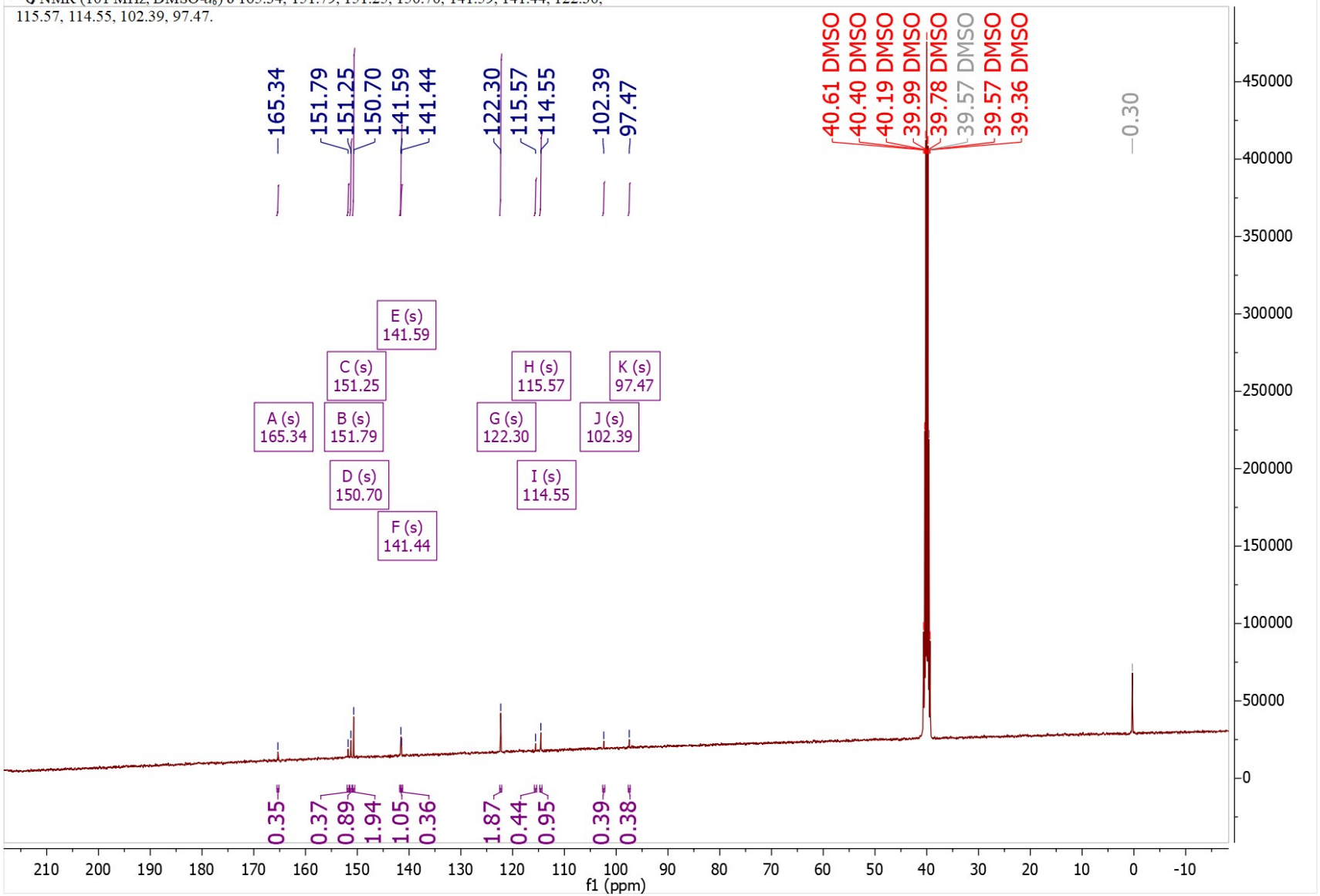
3.3 Synthesis of N-(5-((trimethylsilyl)ethynyl)pyridin-2-yl)nicotinamide, **3Pyr-TMS**

3.4 Synthesis of N-(5-((trimethylsilyl)ethynyl)pyridin-2-yl)isonicotinamide, **4Pyr-TMS**



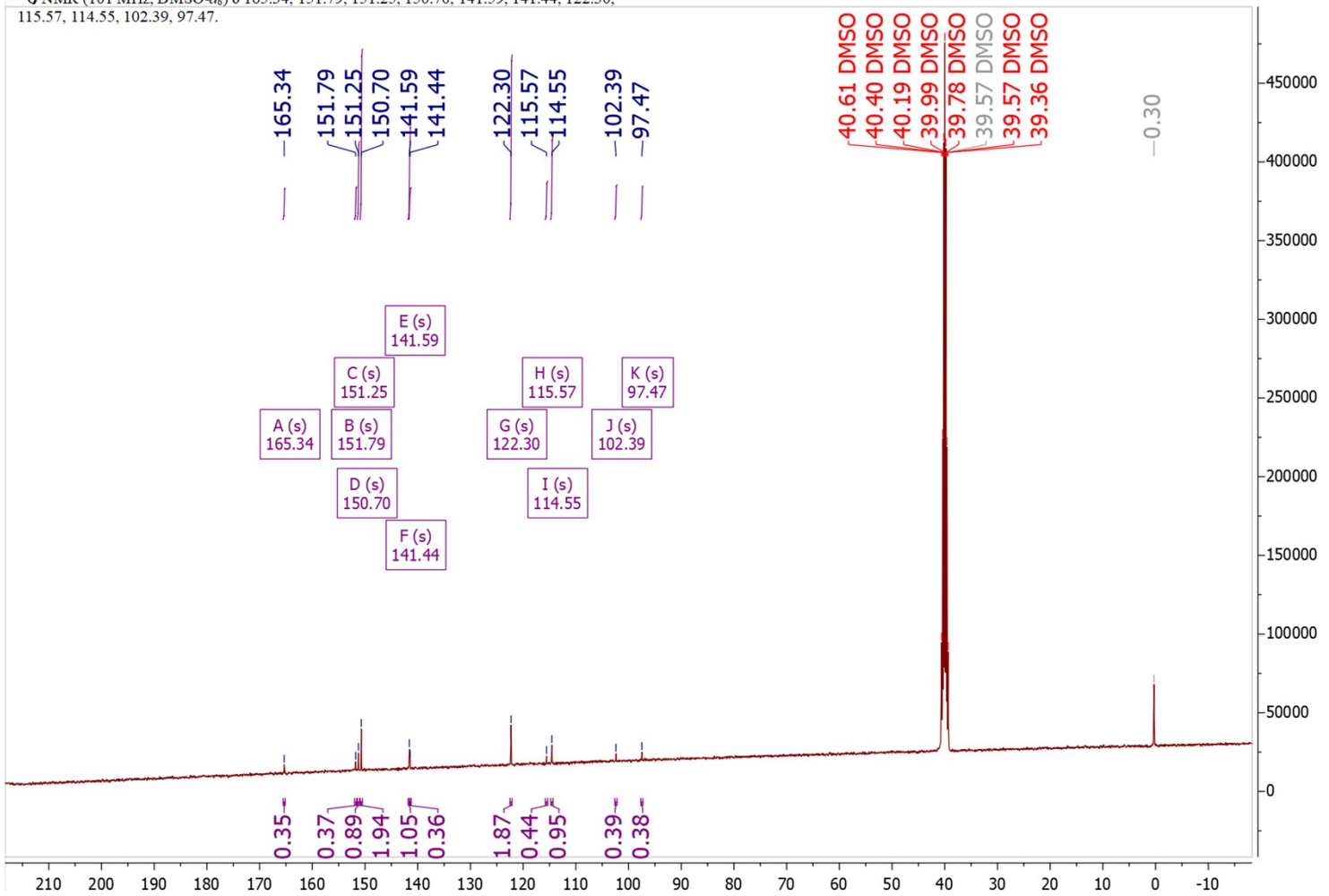
4Pyr-TMS-13C-real 1.fid

^{13}C NMR (101 MHz, DMSO-d_6) δ 165.34, 151.79, 151.25, 150.70, 141.59, 141.44, 122.30, 115.57, 114.55, 102.39, 97.47.

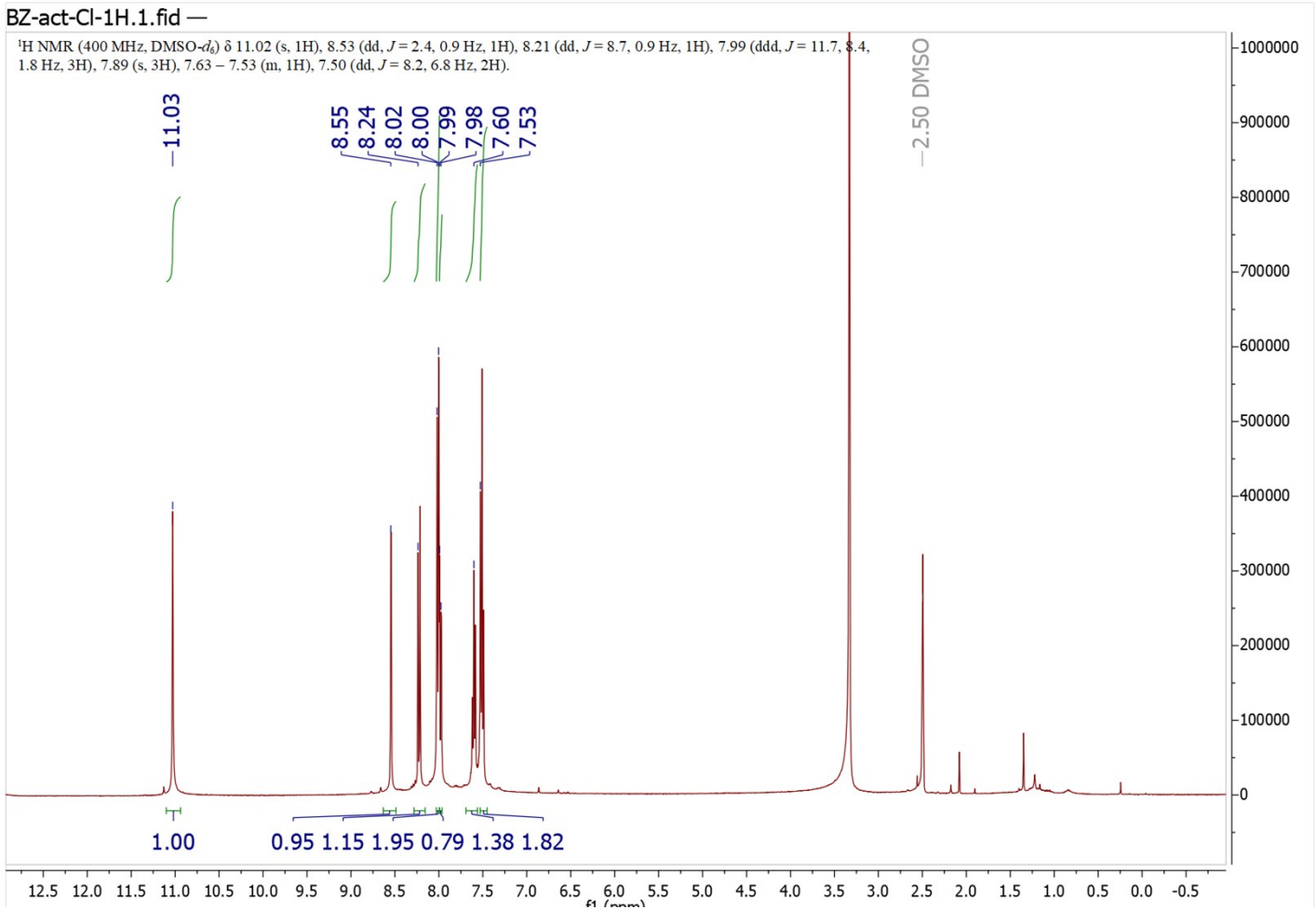


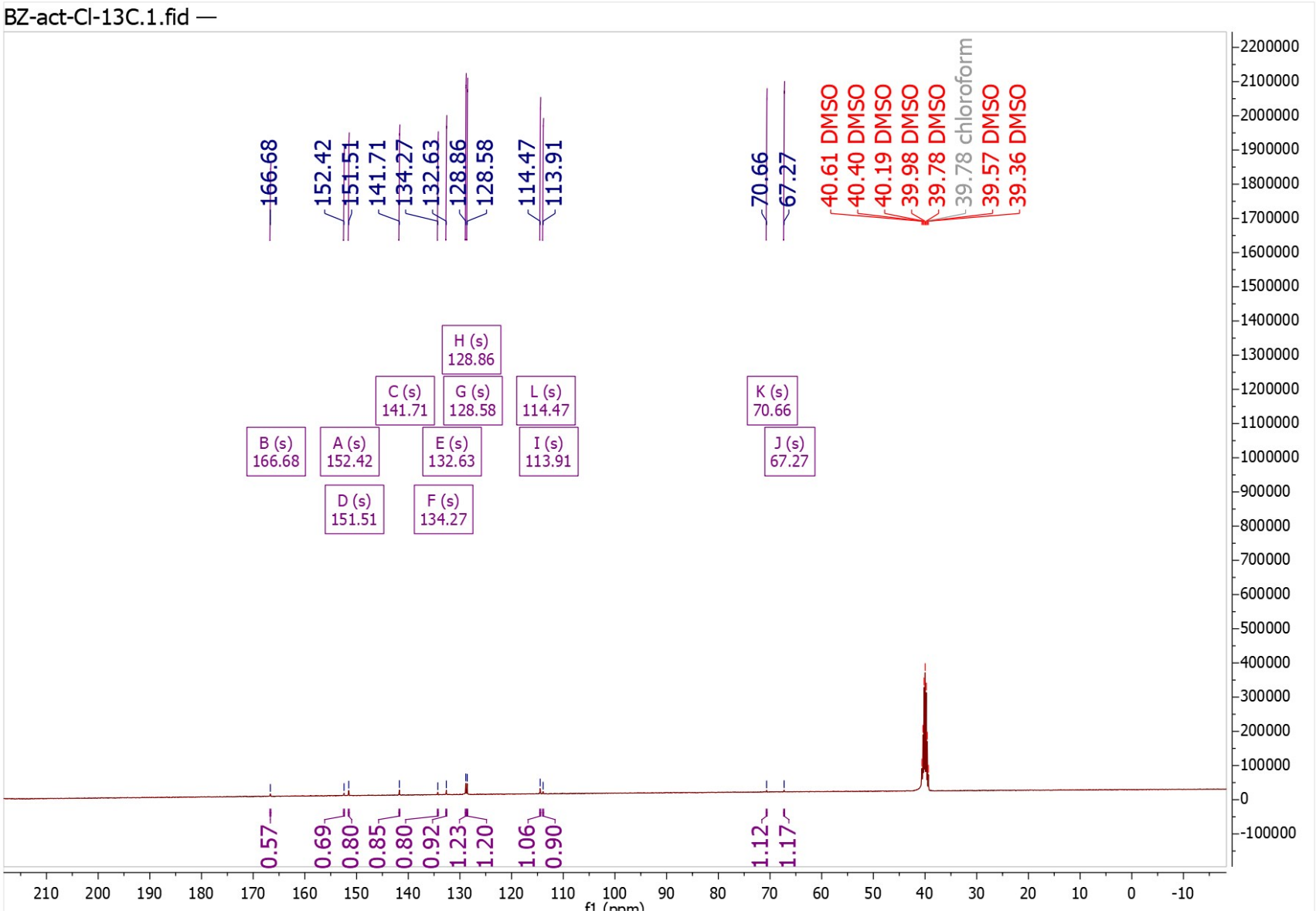
4Pyr_TMS_13C-real 1.fid

^{13}C NMR (101 MHz, DMSO-d_6) δ 165.34, 151.79, 151.25, 150.70, 141.59, 141.44, 122.30, 115.57, 114.55, 102.39, 97.47.

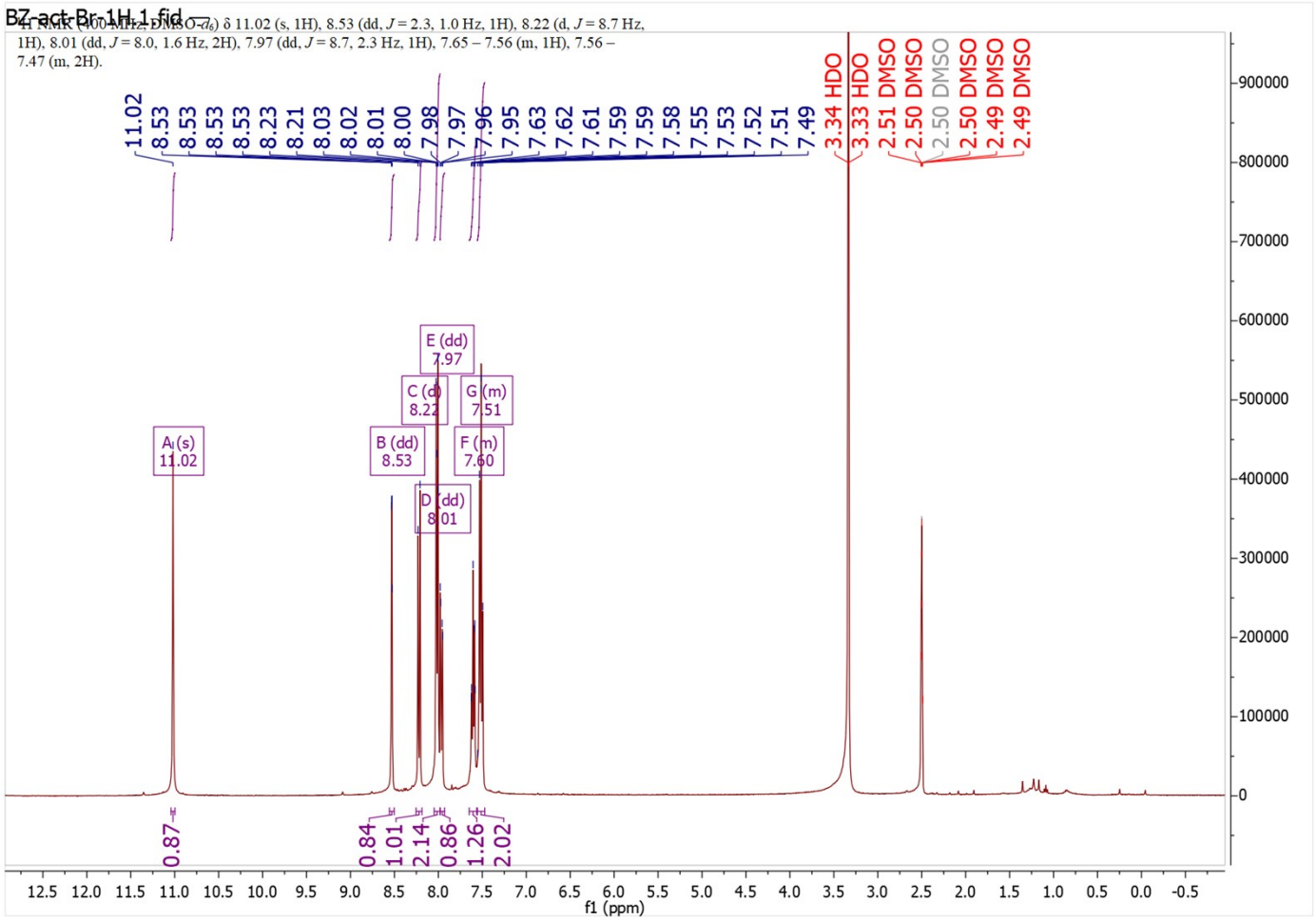


3.5 N-(5-(chloroethynyl)pyridin-2-yl)benzamide, **Bz-act-Cl**



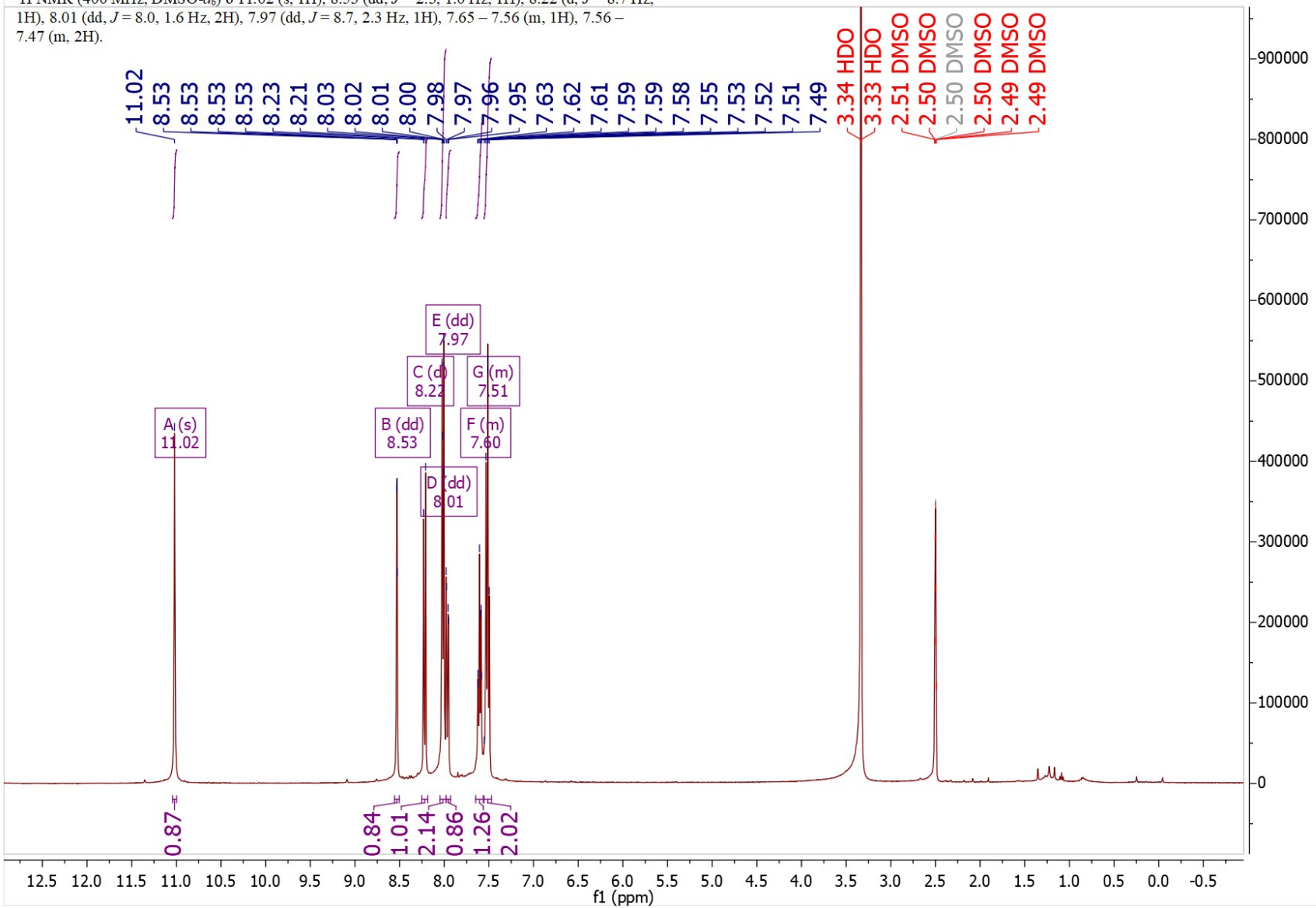

BZ-act-Cl-13C.1.fid
166.68
152.42
151.51
141.71
134.27
132.63
128.86
128.58
114.47
113.91
70.66
67.27
40.61 DMSO
40.40 DMSO
40.19 DMSO
39.98 DMSO
39.78 DMSO
39.78 chloroform
39.57 DMSO
39.36 DMSO
H (s) 128.86
C (s) 141.71
G (s) 128.58
L (s) 114.47
K (s) 70.66
B (s) 166.68
A (s) 152.42
E (s) 132.63
I (s) 113.91
J (s) 67.27
D (s) 151.51
F (s) 134.27
0.57
0.69
0.80
0.85
0.80
0.92
1.23
1.20
1.06
0.90
1.12
1.17
f1 (ppm)

3.6 N-(5-(bromoethynyl)pyridin-2-yl)benzamide, **Bz-act-Br**

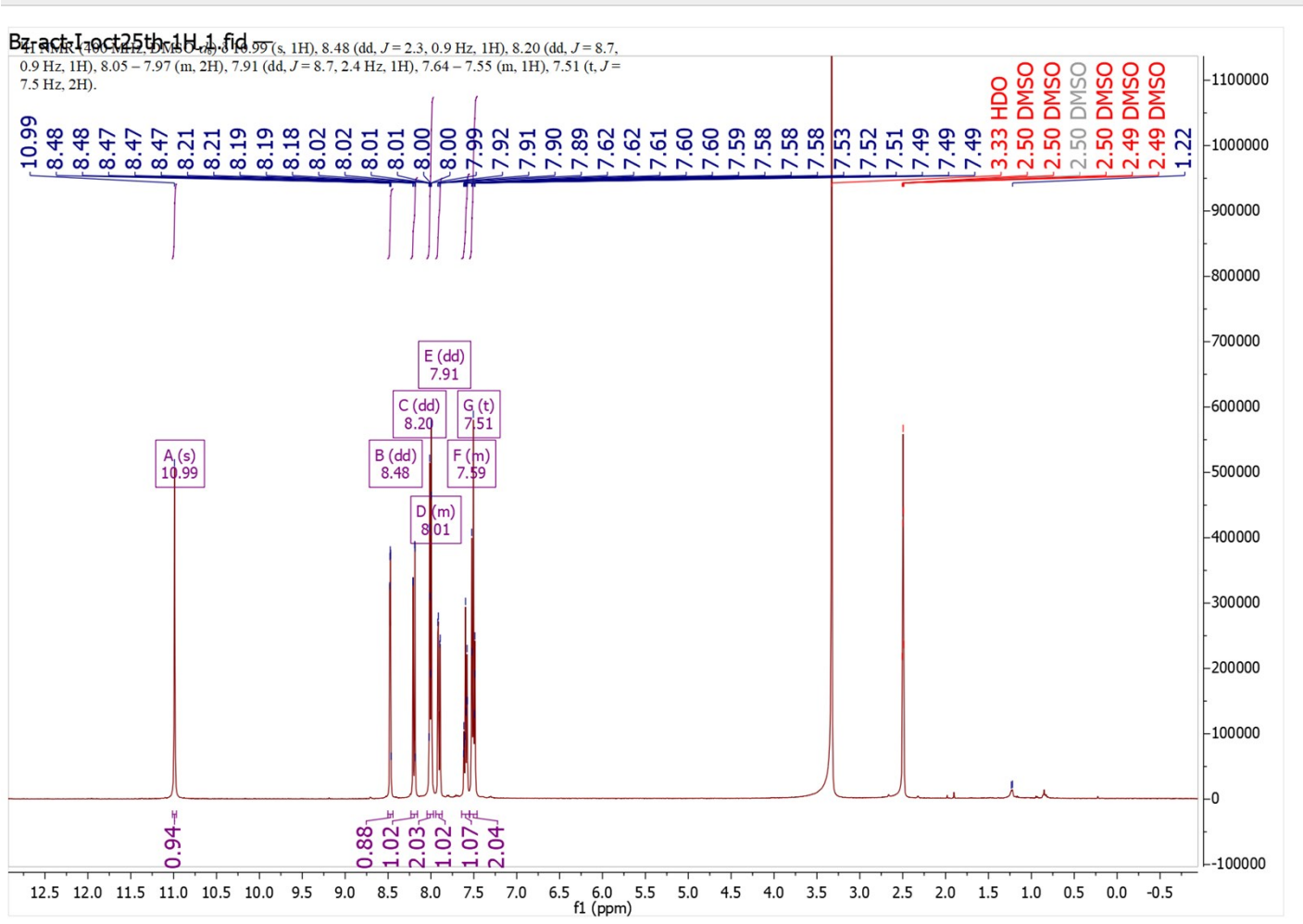


BZ-act-Br-1H-1.fid

^{1}H NMR (400 MHz, DMSO-d_6) δ 11.02 (s, 1H), 8.53 (dd, J = 2.3, 1.0 Hz, 1H), 8.22 (d, J = 8.7 Hz, 1H), 8.01 (dd, J = 8.0, 1.6 Hz, 2H), 7.97 (dd, J = 8.7, 2.3 Hz, 1H), 7.65 – 7.56 (m, 1H), 7.56 – 7.47 (m, 2H).

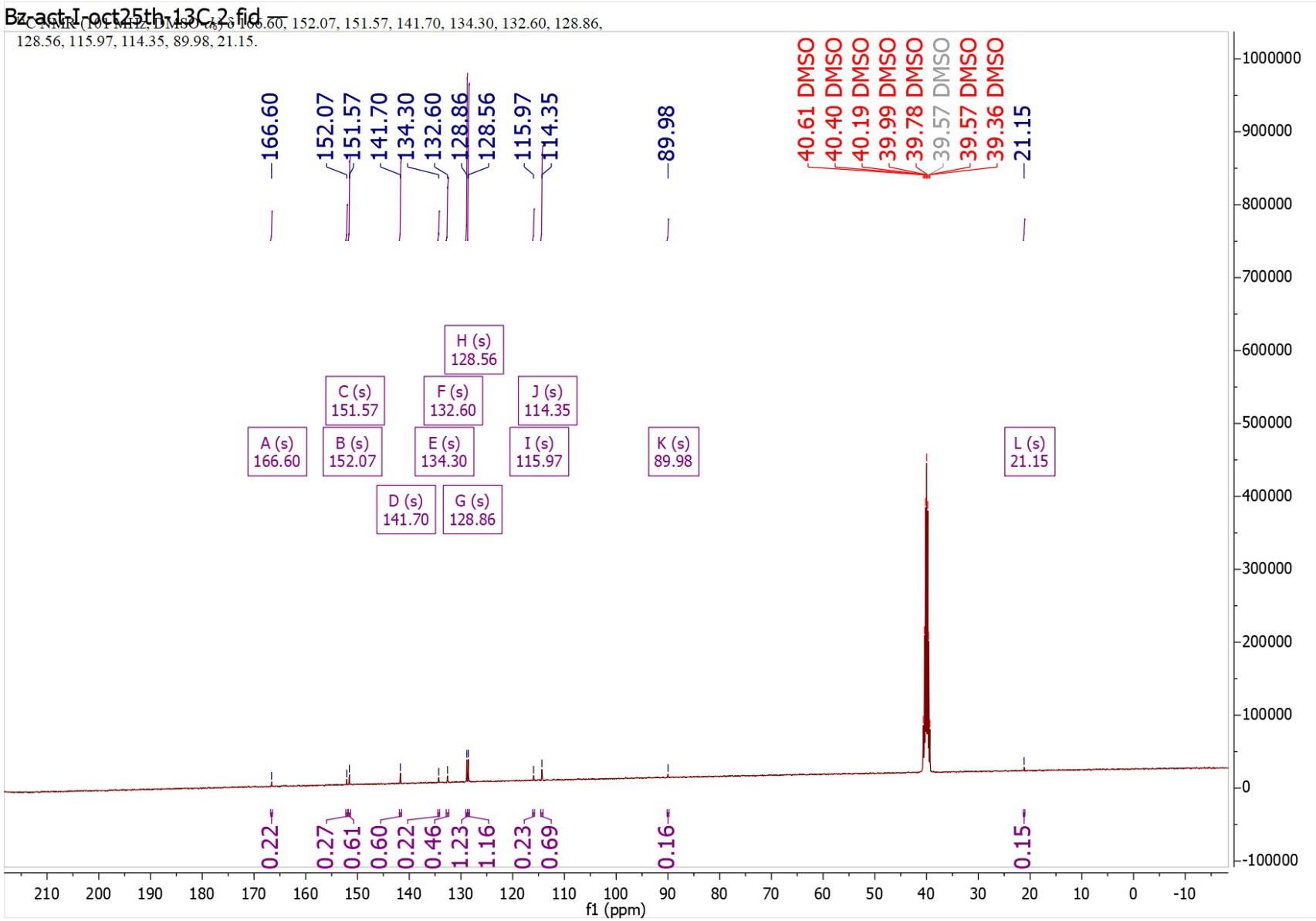


3.7 N-(5-(iodoethynyl)pyridin-2-yl)benzamide, **Bz-act-I**

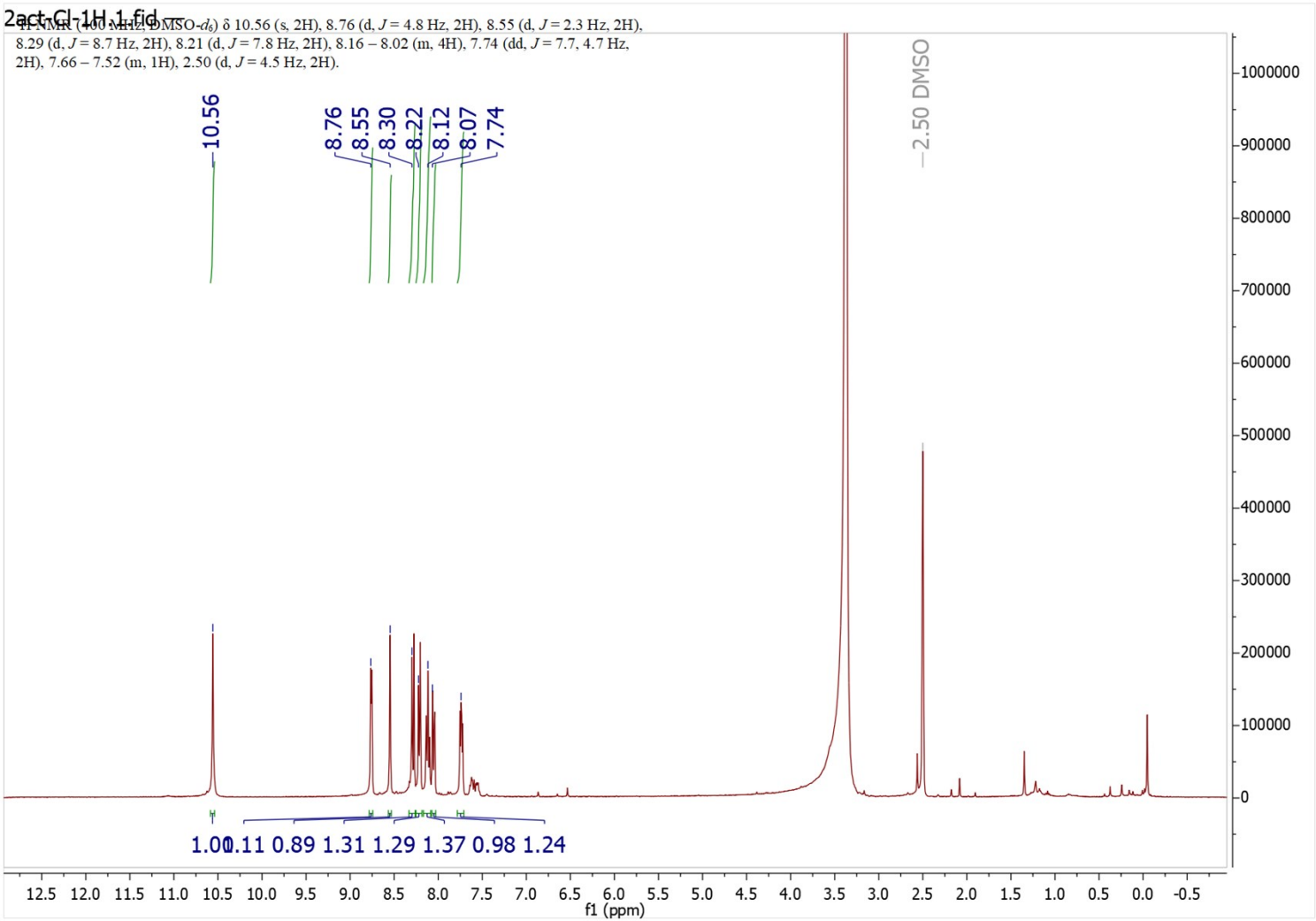


Bz-act-I-oct25th-13C-2.fid

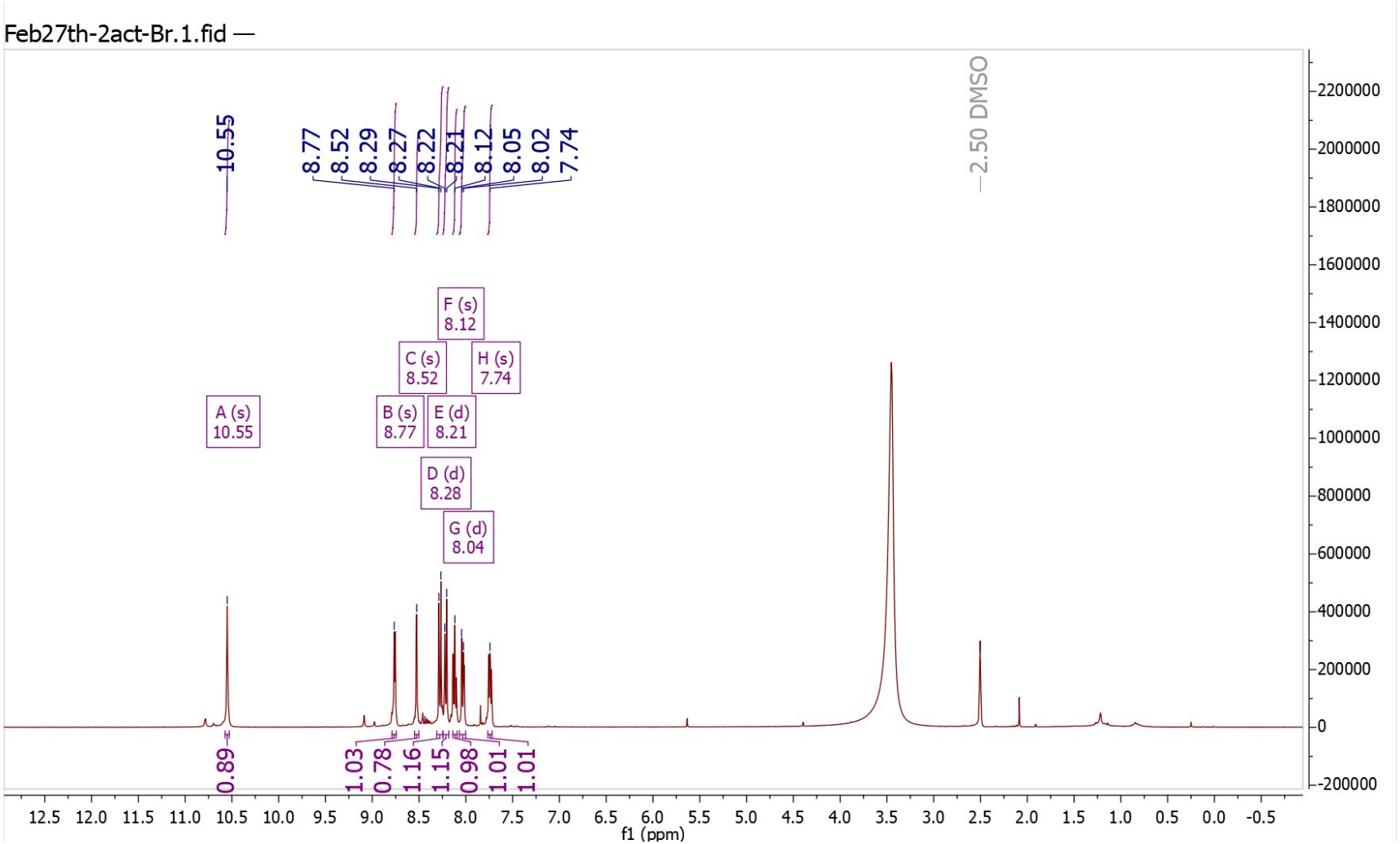
C NMR (101 MHz, DMSO-d_6) δ 166.60, 152.07, 151.57, 141.70, 134.30, 132.60, 128.86, 128.56, 115.97, 114.35, 89.98, 21.15.



3.8 N-(5-(chloroethynyl)pyridin-2-yl)picolinamide, **2act-Cl**

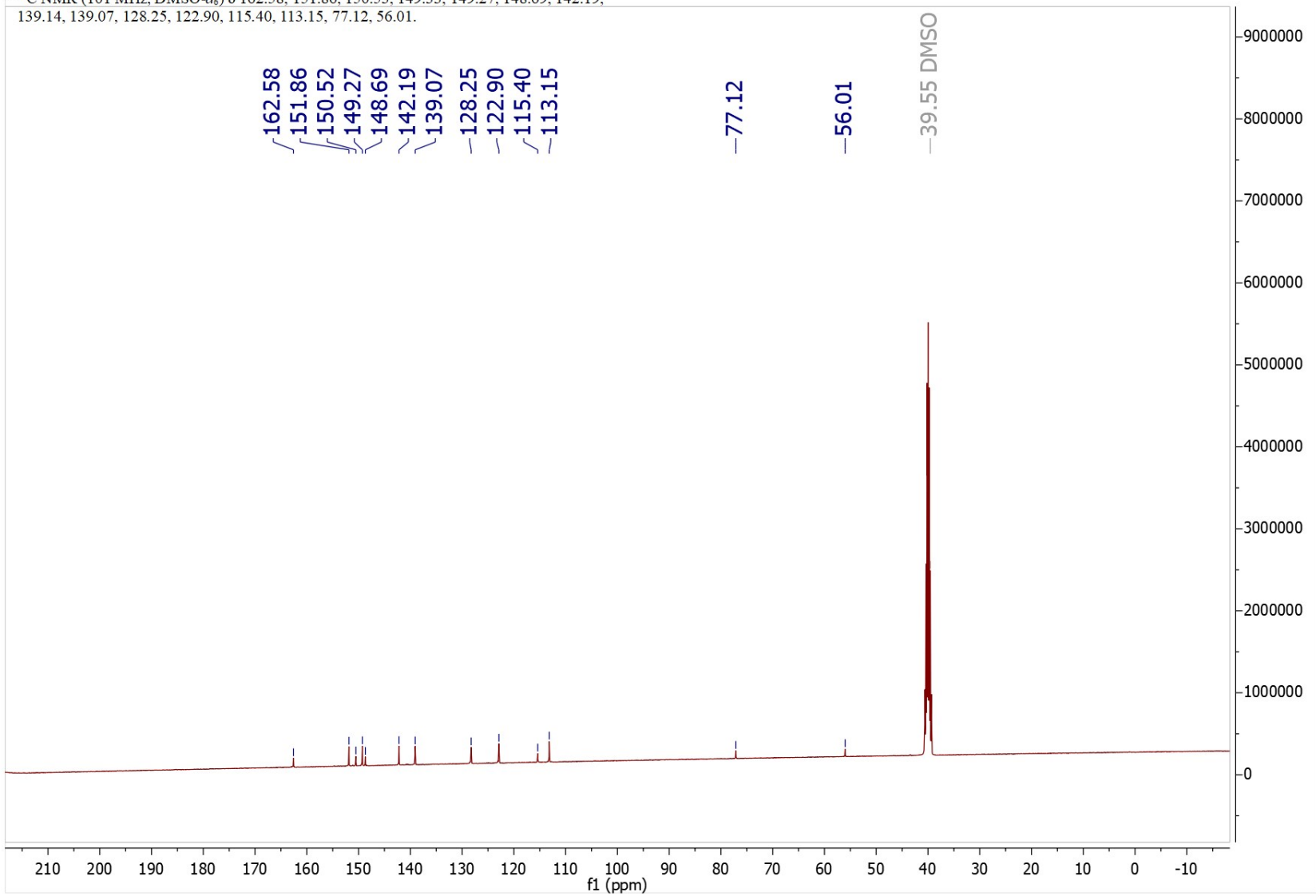


3.9 N-(5-(bromoethynyl)pyridin-2-yl)picolinamide, **2act-Br**

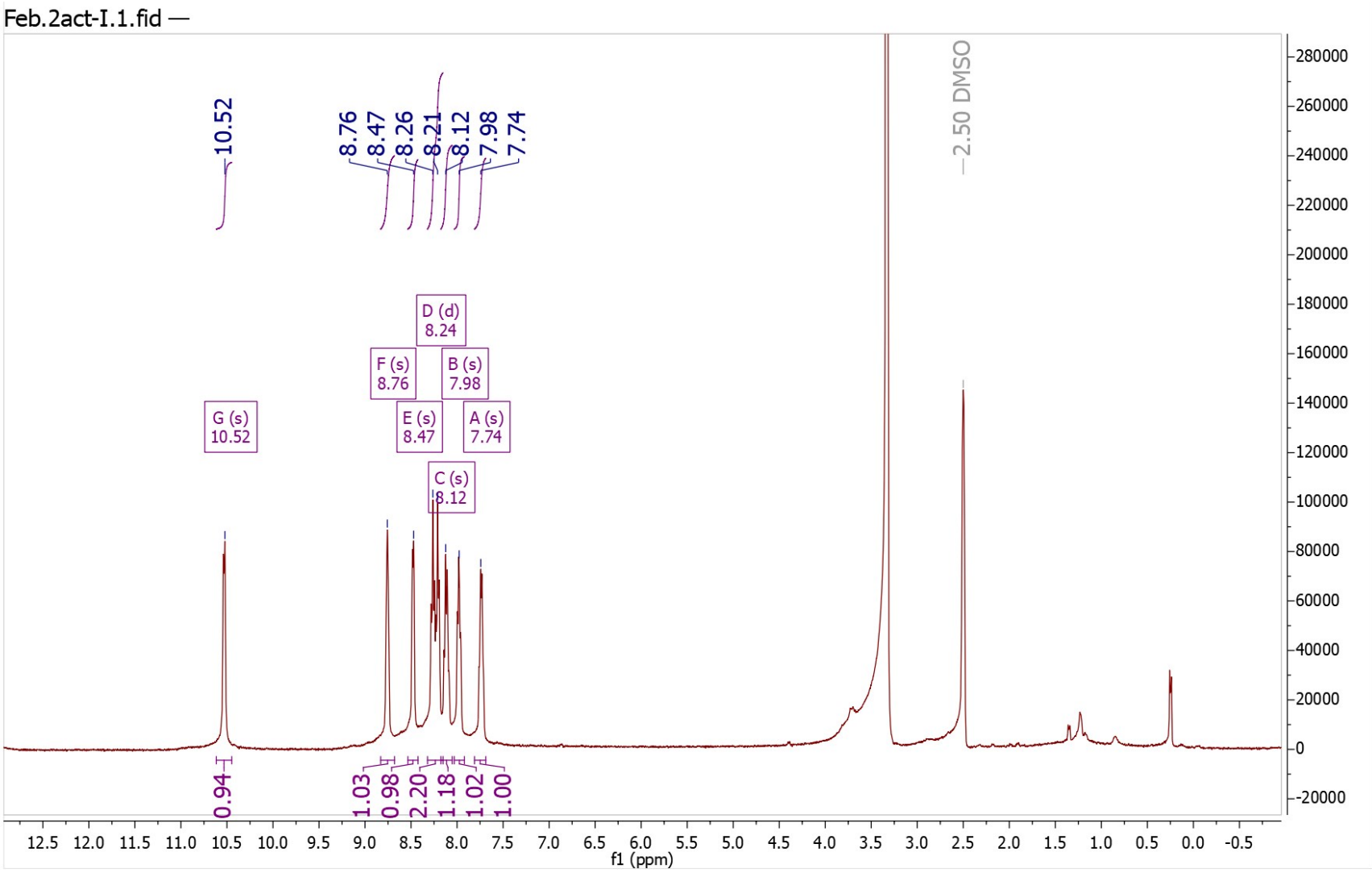


Feb27th_2act-Br_13C_1.fid

^{13}C NMR (101 MHz, DMSO-d_6) δ 162.58, 151.86, 150.53, 149.33, 149.27, 148.69, 142.19, 139.14, 139.07, 128.25, 122.90, 115.40, 113.15, 77.12, 56.01.

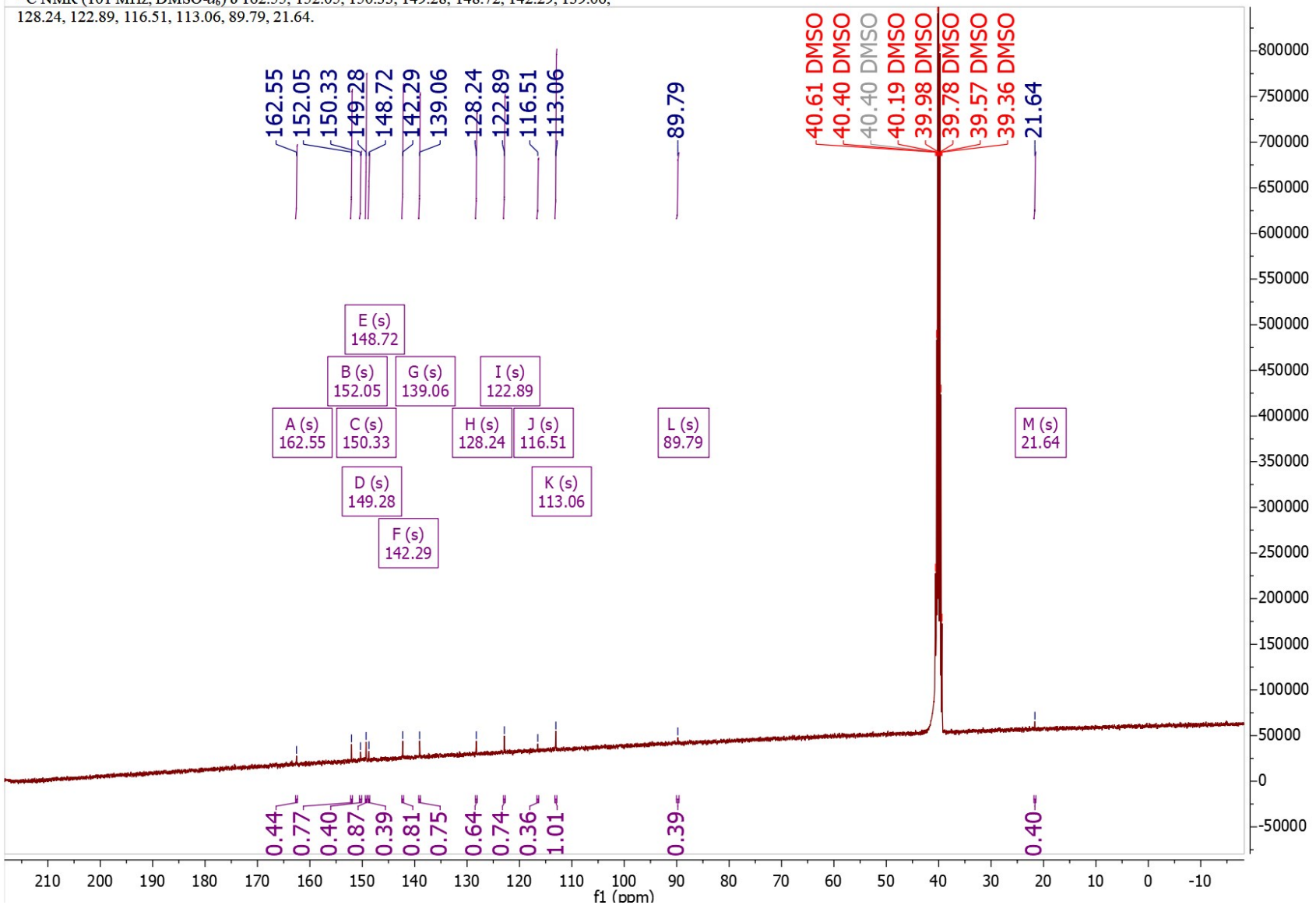


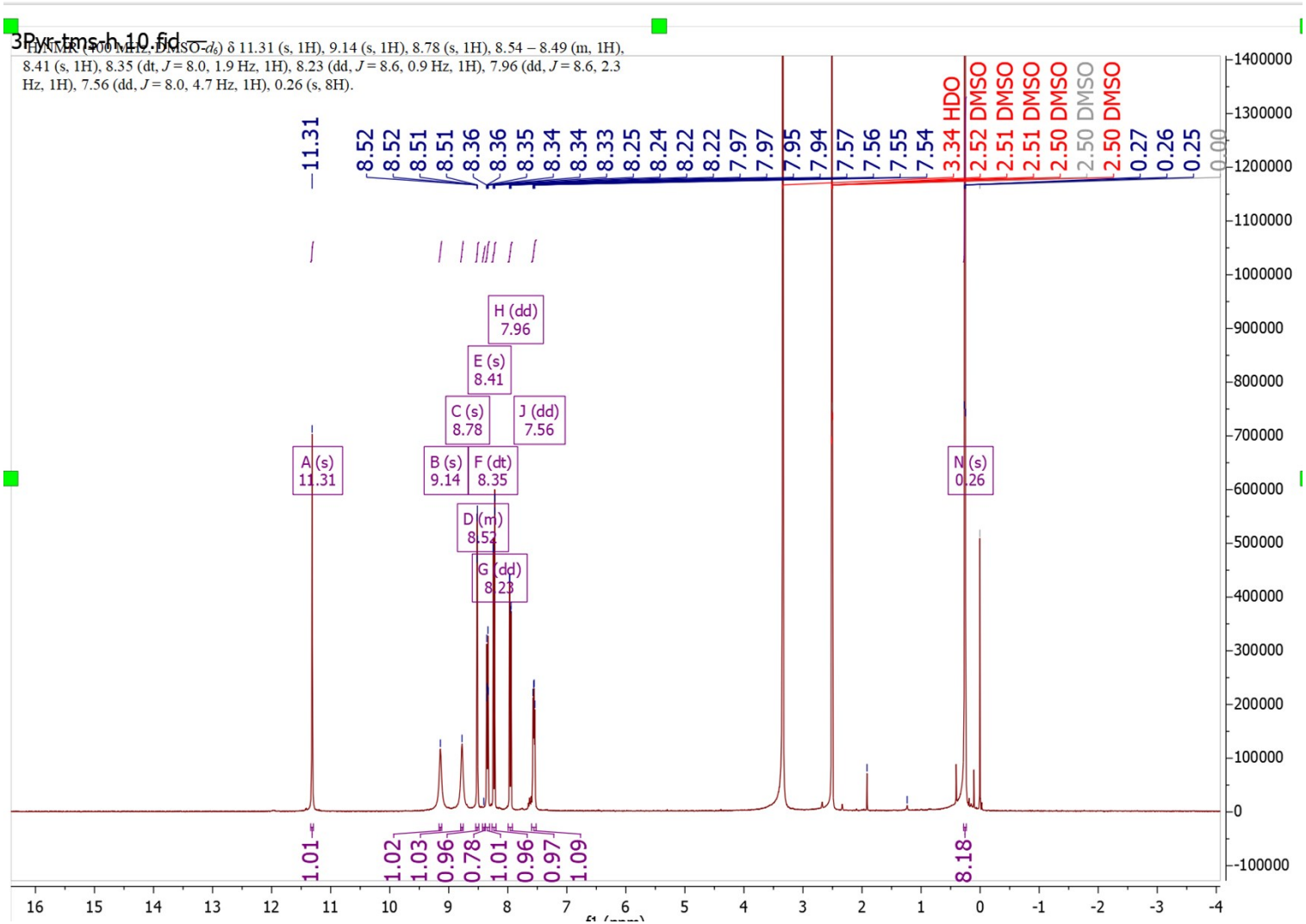
3.10 N-(5-(iodoethynyl)pyridin-2-yl)picolinamide, **2act-I**



Feb-2act-I-13c-20.fid

^{13}C NMR (101 MHz, DMSO-d_6) δ 162.55, 152.05, 150.33, 149.28, 148.72, 142.29, 139.06, 128.24, 122.89, 116.51, 113.06, 89.79, 21.64.

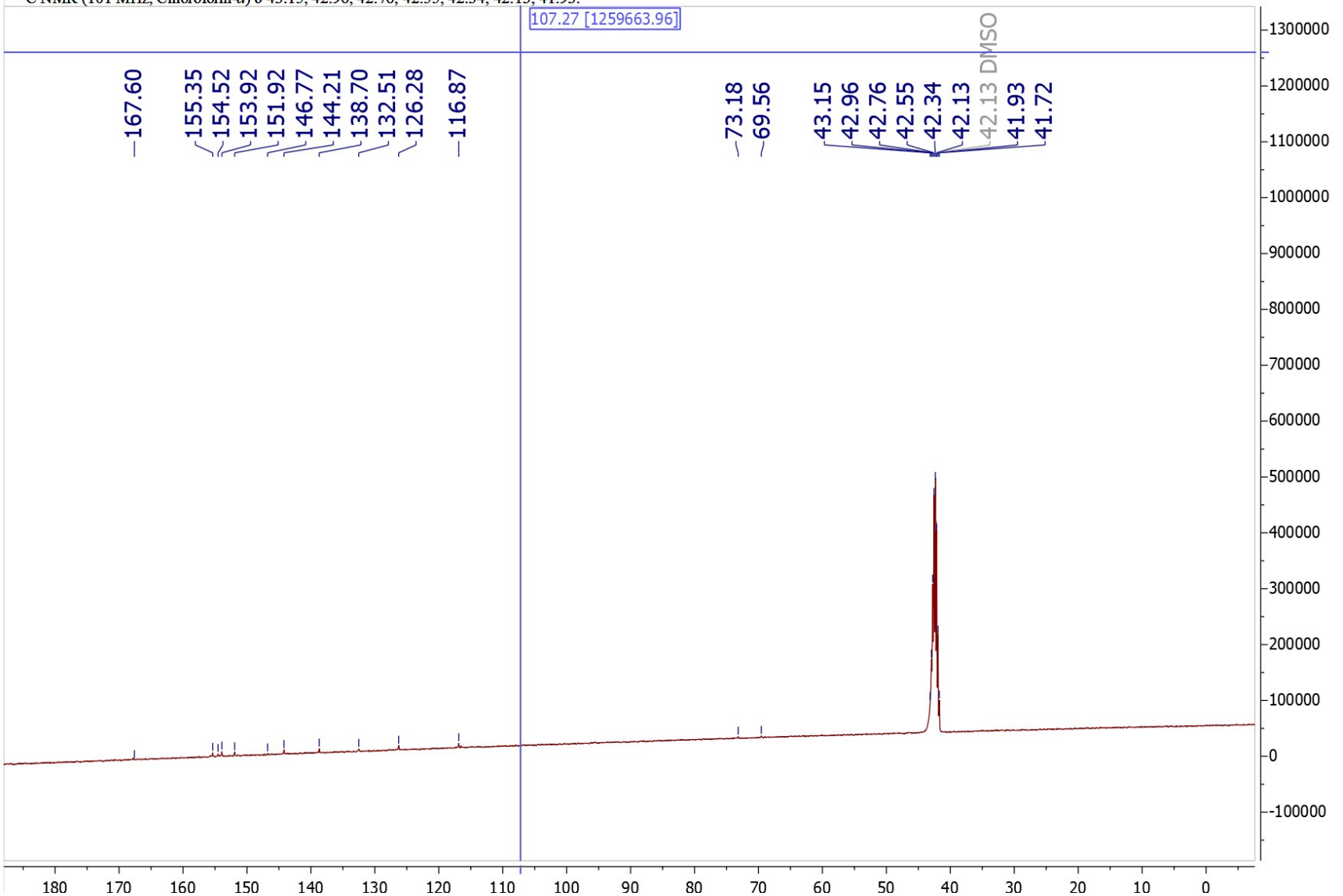




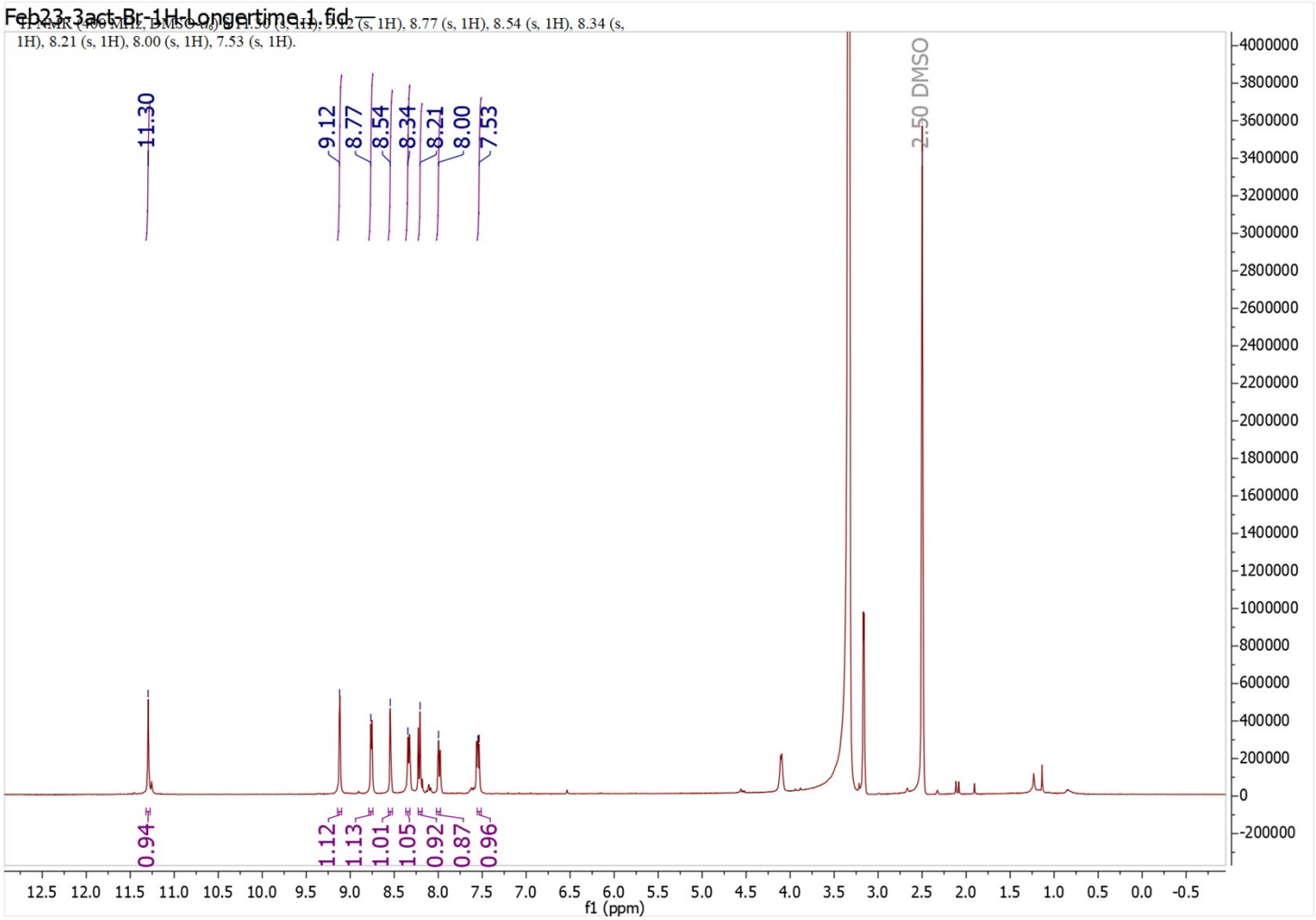
3.11 N-(5-(chloroethynyl)pyridin-2-yl)nicotinamide, **3act-Cl**

3act-Cl-13C-1.fid

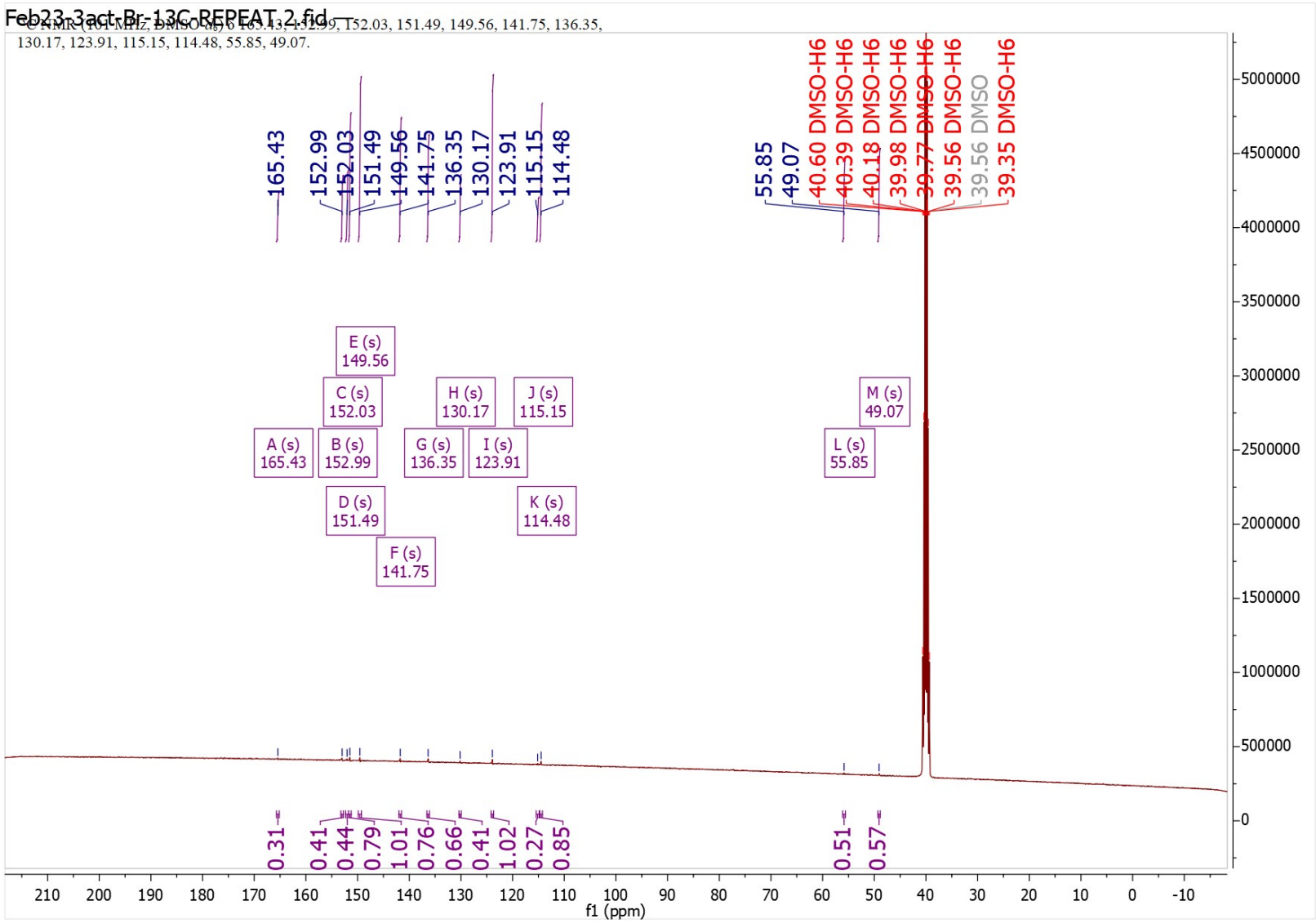
C NMR (101 MHz, Chloroform-*d*) δ 43.15, 42.96, 42.76, 42.55, 42.34, 42.13, 41.93.



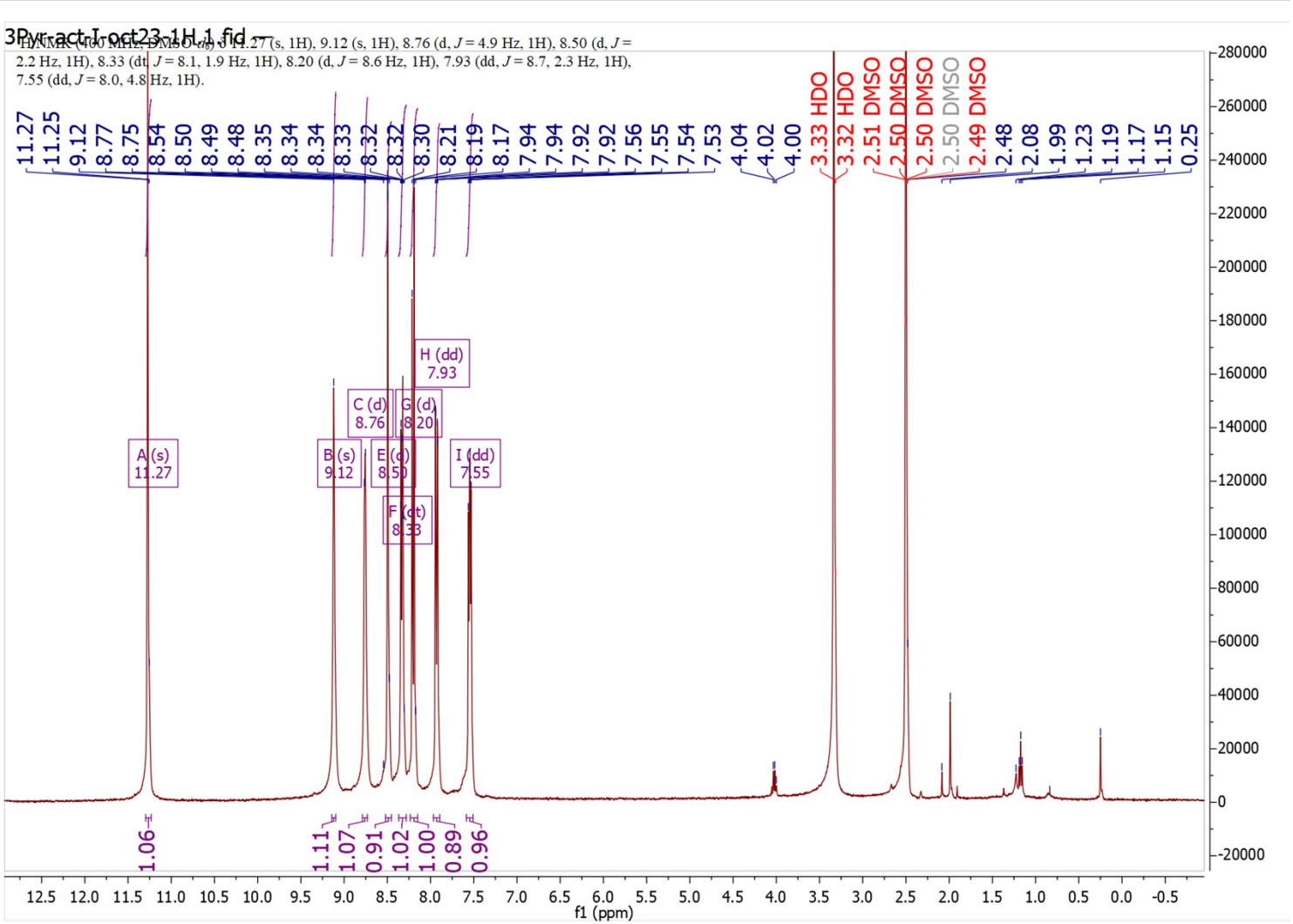
3.12 N-(5-(bromoethynyl)pyridin-2-yl)nicotinamide, **3act-Br**



Feb23-3act-Br-13C-REPEAT-2.fid

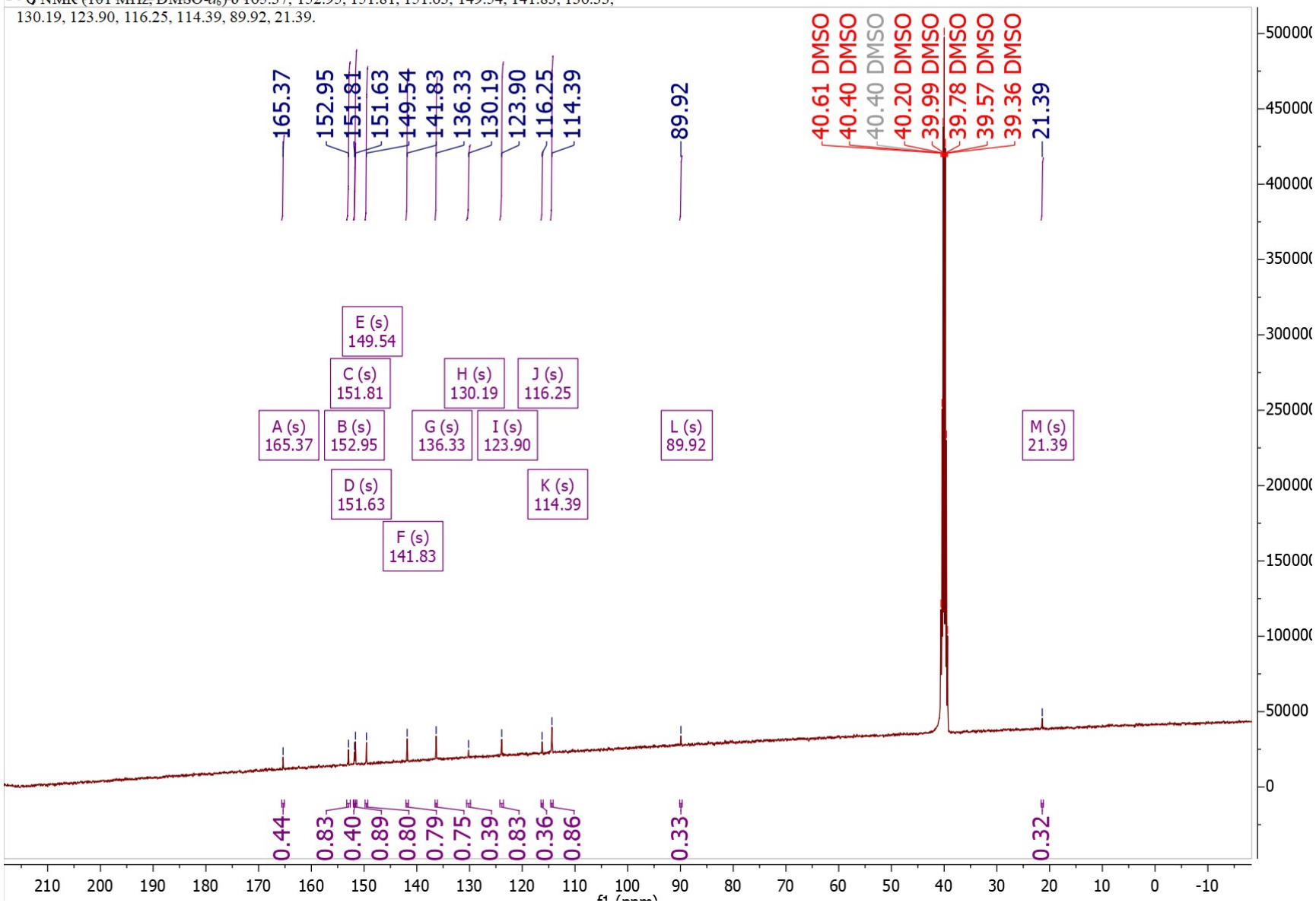


3.13 N-(5-(iodoethynyl)pyridin-2-yl)nicotinamide, **3act-I**

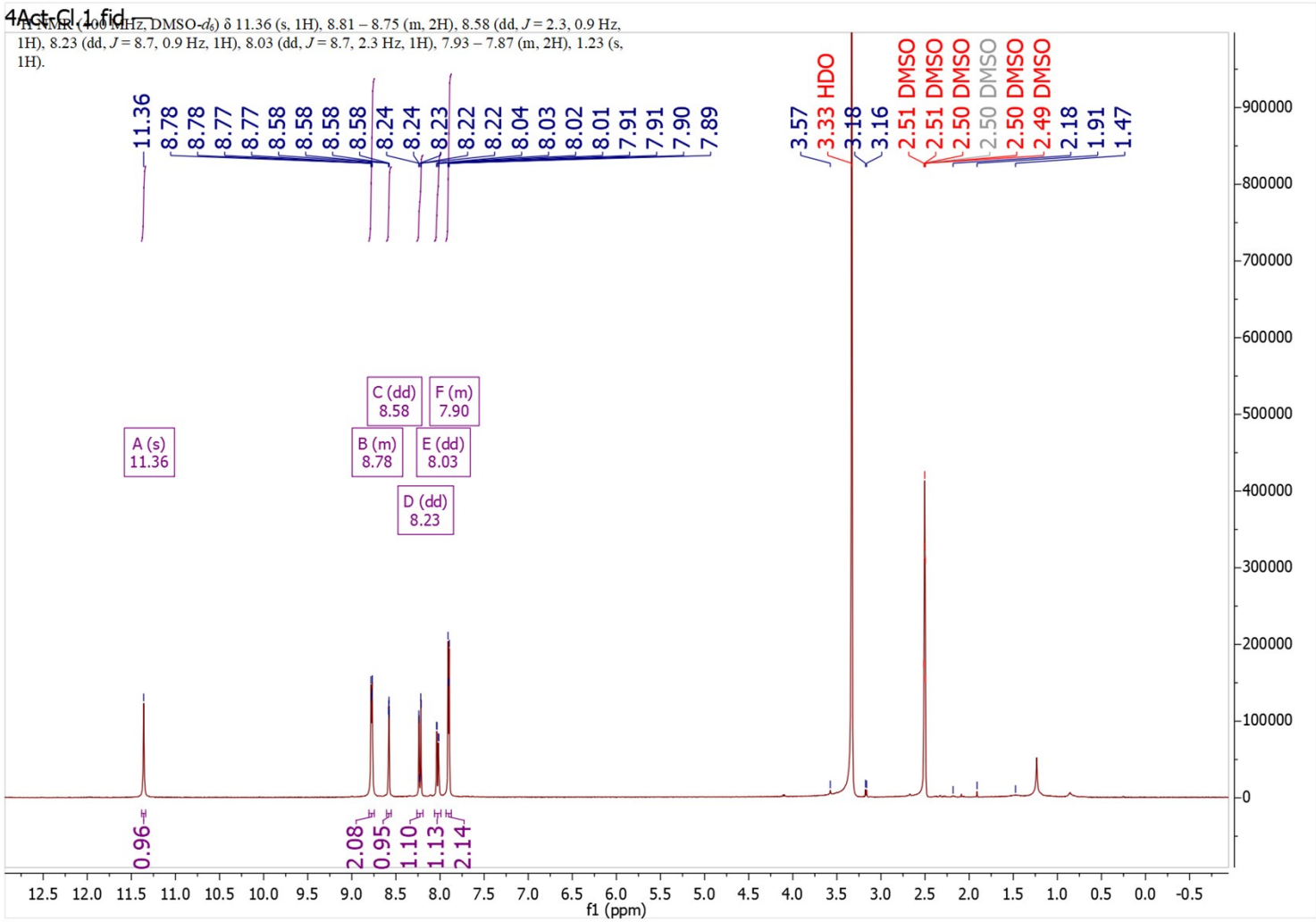


3Pyr-act-I-oct23-13C-1.fid —

^{13}C NMR (101 MHz, DMSO-d_6) δ 165.37, 152.95, 151.81, 151.63, 149.54, 141.83, 136.33, 130.19, 123.90, 116.25, 114.39, 89.92, 21.39.

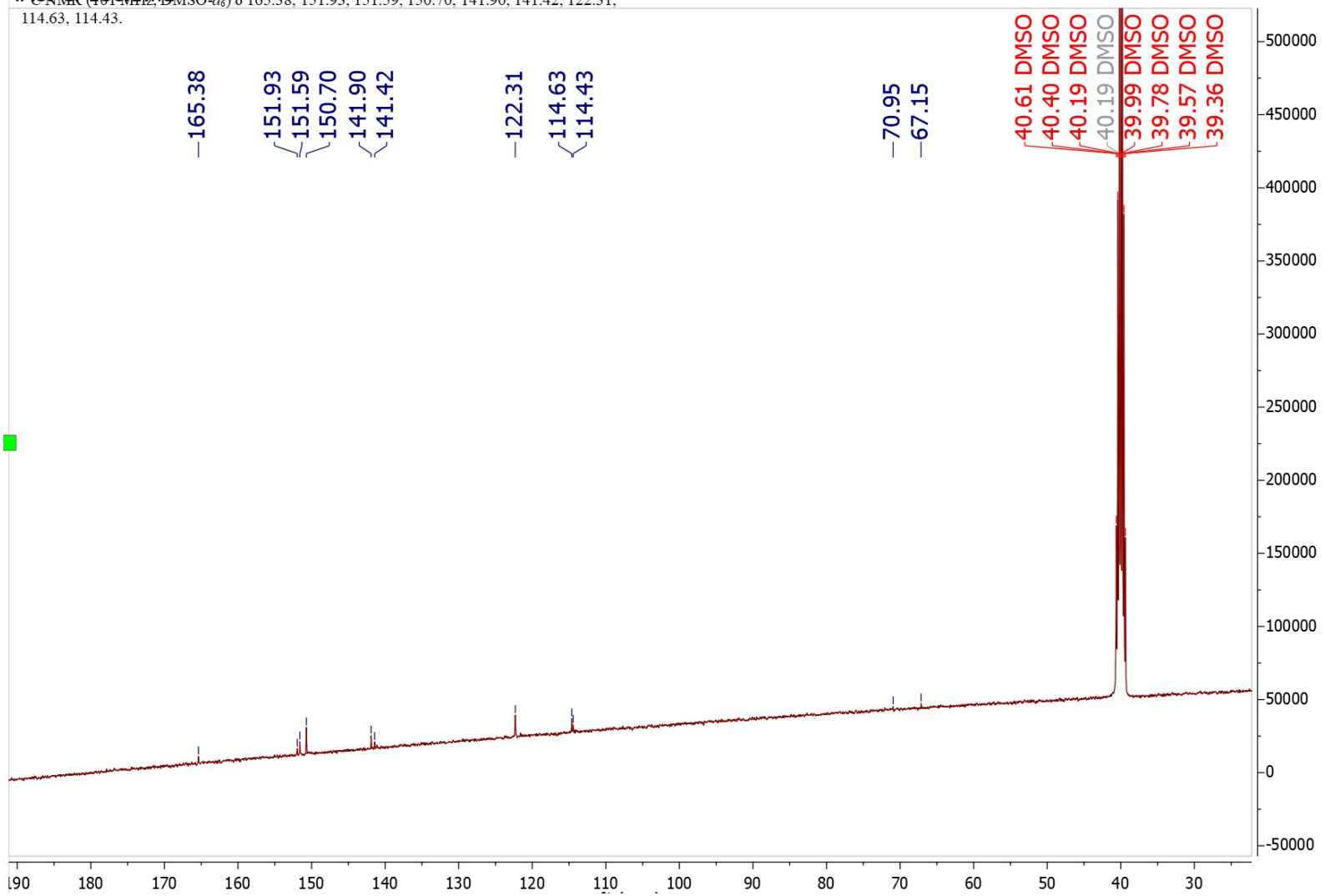


3.14 N-(5-(chloroethynyl)pyridin-2-yl)isonicotinamide, **4act-Cl**

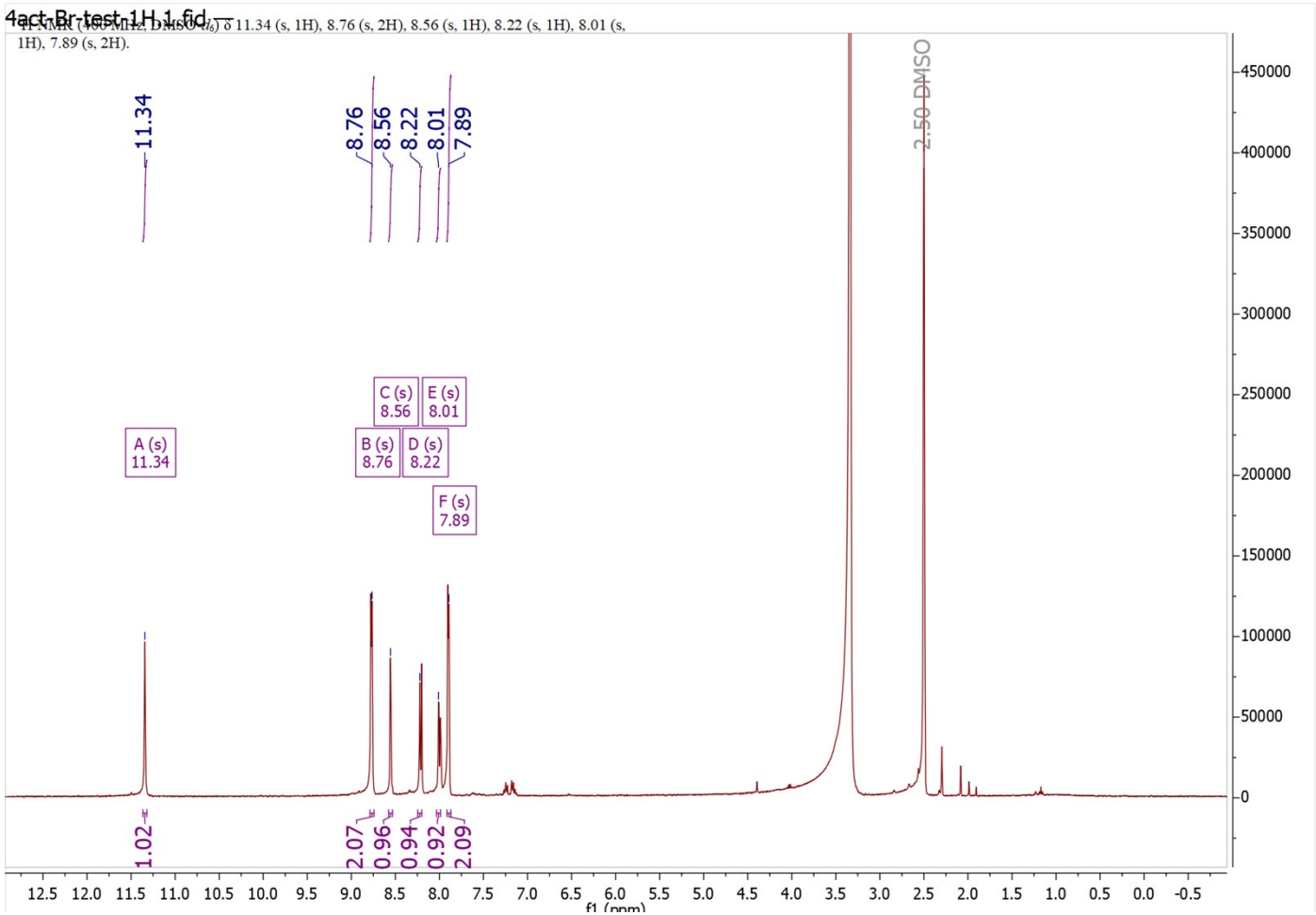


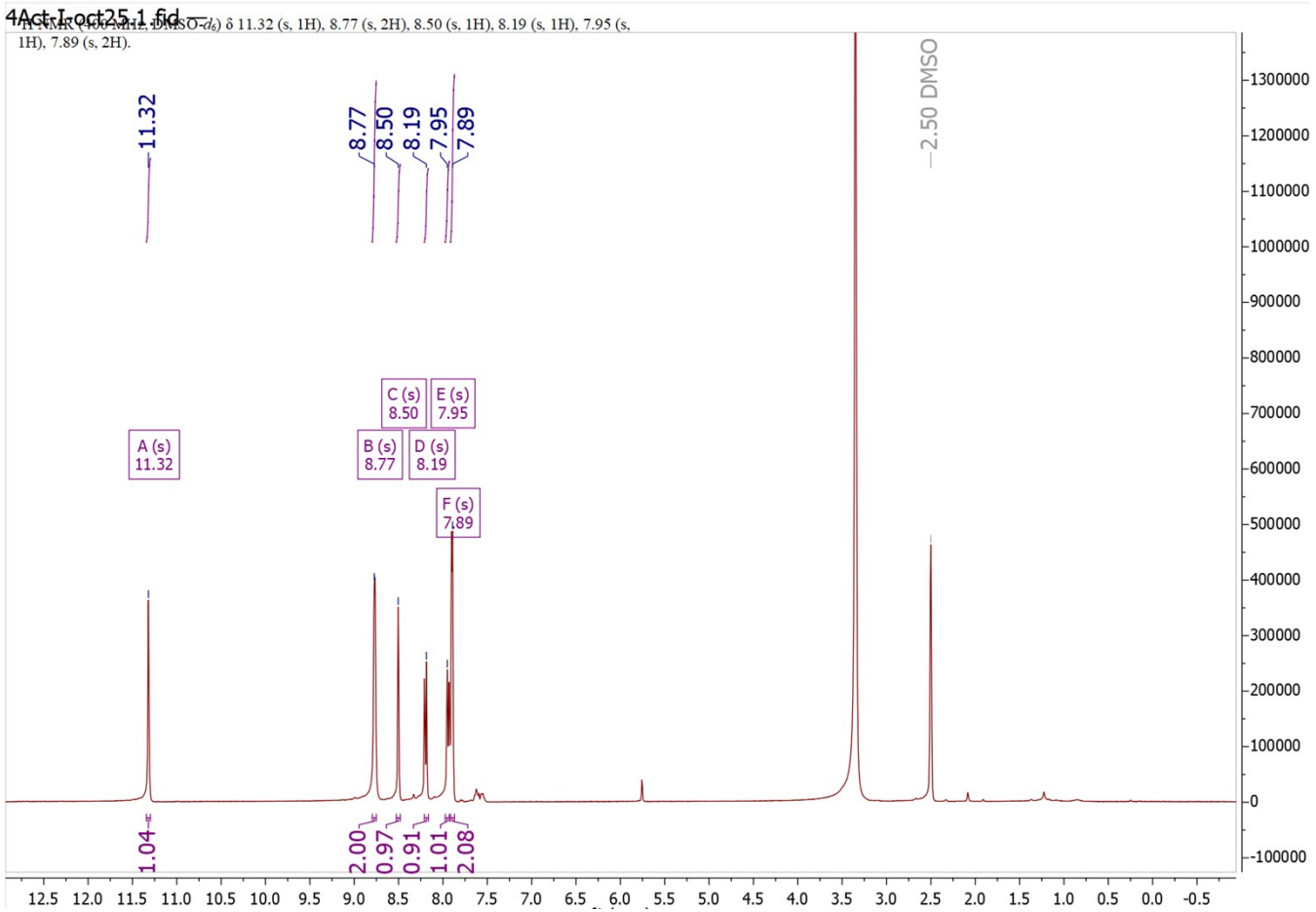
4Act-Cl-13c.1.fid

^{13}C NMR (101 MHz, DMSO-d_6) δ 165.38, 151.93, 151.59, 150.70, 141.90, 141.42, 122.31, 114.63, 114.43.



3.15 N-(5-(bromoethynyl)pyridin-2-yl)isonicotinamide, **4act-Br**





3.16 N-(5-(iodoethynyl)pyridin-2-yl)isonicotinamide, **4act-I**

4Act-I-oct25-13C-1.fid

^{13}C NMR (101 MHz, DMSO-d_6) δ 165.29, 151.65, 151.57, 150.66, 141.88, 141.49, 122.32, 116.47, 114.52, 89.88, 21.59.