SUPPORTING INFORMATION

Diastereoselective Intermolecular Ene Reactions: Synthesis of 4,5,6,7-Tetrahydro-1*H*-benzo[*d*]imidazoles

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ORTEP diagrams and notes for single crystal X-ray structures: 6b, 7c, 7d and 8a

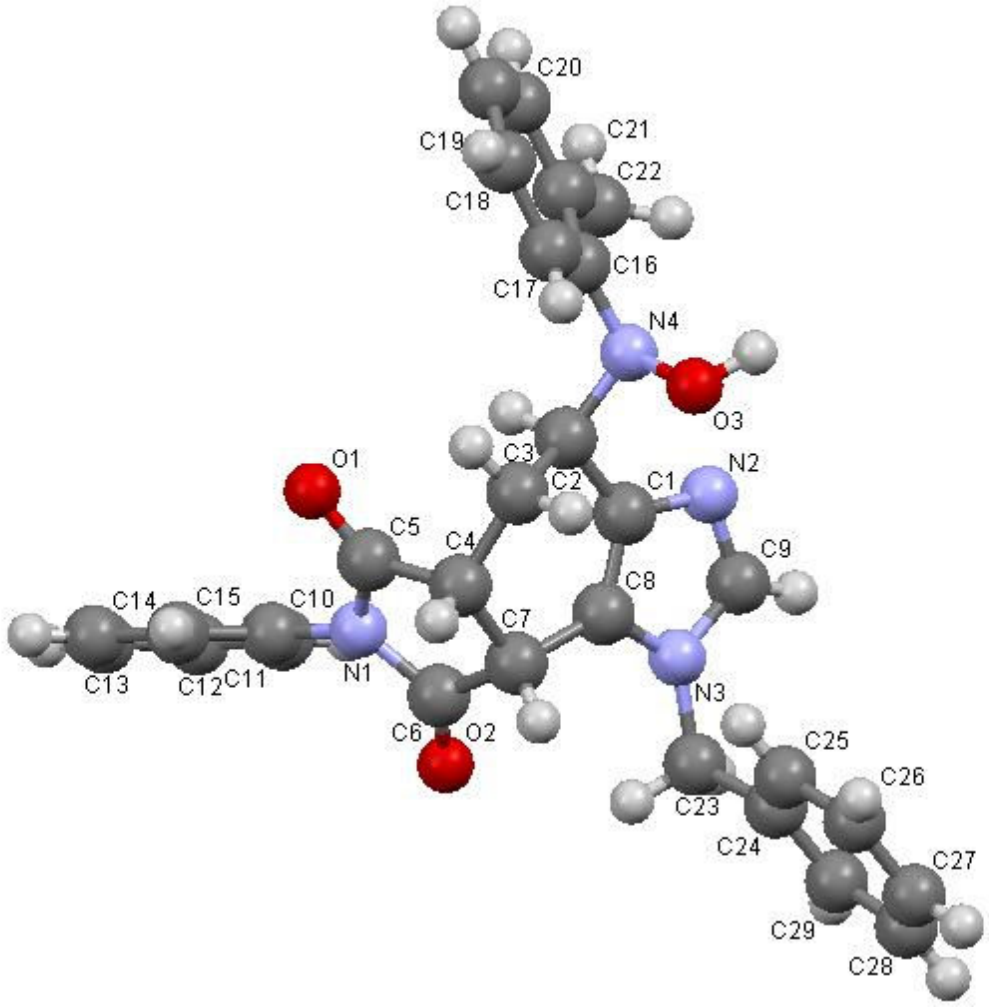
6b - (4*S**,5a*S**,8a*S**)-1-Benzyl-4-(hydroxy(*o*-tolyl)amino)-7-phenyl-5,5a,7,8a-tetrahydroimidazo[4,5-*e*]isoindole-6,8(1*H*,4*H*)-dione S3
7c - (*S**)-Ethyl 2-((4*S**,5a*S**,8a*S**)-1-benzyl-6,8-dioxo-7-phenyl-1,4,5,5a,6,7,8,8a-octahydroimidazo[4,5-*e*]isoindol-4-yl)-3,3,3-trifluoro-2-hydroxypropanoate S5
7d - (*S**)-Ethyl 2-((4*S**,5*S**,5a*S**,8a*S**)-1-benzyl-5-(((*tert*-butyldimethylsilyl)oxy)methyl)-6,8-dioxo-7-phenyl-1,4,5,5a,6,7,8,8a-octahydroimidazo[4,5-*e*]isoindol-4-yl)-3,3,3-trifluoro-2-hydroxypropanoate S7
8a - (*R**)-Ethyl 2-((4*S**,5a*S**,8a*S**)-1-benzyl-6,8-dioxo-7-phenyl-1,4,5,5a,6,7,8,8a-octahydroimidazo[4,5-*e*]isoindol-4-yl)-2-hydroxyacetate S9

^{1}H and ^{13}C NMR Spectra

^{1}H (5a) - (5a*S**,8a*S**)-1-Benzyl-7-phenyl-1,5,5a,8b-tetrahydroimidazo[4,5-*e*]isoindole-6,8(7*H*,8a*H*)-dione S11
^{13}C (5a) - (5aS*,8aS*)-1-Benzyl-7-phenyl-1,5,5a,8b-tetrahydroimidazo[4,5-*e*]isoindole-6,8(7*H*,8a*H*)-dione S12
^{1}H (6a) - (4*S**,5a*S**,8a*S**)-1-Benzyl-4-(hydroxy(phenyl)amino)-7-phenyl-5,5a,7,8a-tetrahydroimidazo[4,5-*e*]isoindole-6,8(1*H*,4*H*)-dione S13
^{13}C (6a) - (4*S**,5a*S**,8a*S**)-1-Benzyl-4-(hydroxy(phenyl)amino)-7-phenyl-5,5a,7,8a-tetrahydroimidazo[4,5-*e*]isoindole-6,8(1*H*,4*H*)-dione S14
^{1}H (6b) - (4*S**,5a*S**,8a*S**)-1-Benzyl-4-(hydroxy(*o*-tolyl)amino)-7-phenyl-5,5a,7,8a-tetrahydroimidazo[4,5-*e*]isoindole-6,8(1*H*,4*H*)-dione S15
^{13}C (6b) - (4*S**,5a*S**,8a*S**)-1-Benzyl-4-(hydroxy(*o*-tolyl)amino)-7-phenyl-5,5a,7,8a-tetrahydroimidazo[4,5-*e*]isoindole-6,8(1*H*,4*H*)-dione S16
^{1}H (6c) - (4*S**,5a*S**,8a*S**)-1-Benzyl-4-((2,6-dibromophenyl)(hydroxy)amino)-7-phenyl-5,5a,7,8a-tetrahydroimidazo[4,5-*e*]isoindole-6,8(1*H*,4*H*)-dione S17
^{13}C (6c) - (4*S**,5a*S**,8a*S**)-1-Benzyl-4-((2,6-dibromophenyl)(hydroxy)amino)-7-phenyl-5,5a,7,8a-tetrahydroimidazo[4,5-*e*]isoindole-6,8(1*H*,4*H*)-dione S18
^{1}H (6d) - (4*R**,5a*S**,8a*S**)-1-Benzyl-4,7-diphenyl-5,5a,7,8a-tetrahydroimidazo[4,5-*e*]isoindole-6,8(1*H*,4*H*)-dione S19
^{13}C (6d) - (4*R**,5a*S**,8a*S**)-1-Benzyl-4,7-diphenyl-5,5a,7,8a-tetrahydroimidazo[4,5-*e*]isoindole-6,8(1*H*,4*H*)-dione S20
^{1}H (6e) - (4*S**,5a*S**,8a*S**)-1-Benzyl-4-(3,5-dioxo-4-phenyl-1,2,4-triazolidin-1-yl)-7-phenyl-5,5a,7,8a-tetrahydroimidazo[4,5-*e*]isoindole-6,8(1*H*,4*H*)-dione S21
^{13}C (6e) - (4*S**,5a*S**,8a*S**)-1-Benzyl-4-(3,5-dioxo-4-phenyl-1,2,4-triazolidin-1-yl)-7-phenyl-5,5a,7,8a-tetrahydroimidazo[4,5-*e*]isoindole-6,8(1*H*,4*H*)-dione S22
^{1}H (6f) - Diethyl 2-((4*S**,5a*S**,8a*S**)-1-benzyl-6,8-dioxo-7-phenyl-1,4,5,5a,6,7,8,8a-octahydroimidazo[4,5-*e*]isoindol-4-yl)-2-hydroxymalonate S23
^{13}C (6f) - Diethyl 2-((4*S**,5a*S**,8a*S**)-1-benzyl-6,8-dioxo-7-phenyl-1,4,5,5a,6,7,8,8a-octahydroimidazo[4,5-*e*]isoindol-4-yl)-2-hydroxymalonate S24
^{1}H (5b) - (5*S**,5a*S**,8a*S**)-1-Benzyl-5-(((*tert*-butyldimethylsilyl)oxy)methyl)-7-phenyl-1,5,5a,8b-tetrahydroimidazo[4,5-e]isoindole-6,8(7*H*,8a*H*)-dione S25
^{13}C (5b) - (5*S**,5a*S**,8a*S**)-1-Benzyl-5-(((*tert*-butyldimethylsilyl)oxy)methyl)-7-phenyl-1,5,5a,8b-tetrahydroimidazo[4,5-e]isoindole-6,8(7*H*,8a*H*)-dione S26
^{1}H (6g) - (4*S**,5*R**,5a*S**,8a*S**)-1-Benzyl-5-(((*tert*-butyldimethylsilyl)oxy)methyl)-4-(3,5-dioxo-4-phenyl-1,2,4-triazolidin-1-yl)-7-phenyl-5,5a,7,8a-tetrahydroimidazo[4,5-*e*]isoindole-6,8(1H,4H)-dione S27
^{13}C (6g) - (4*S**,5*R**,5a*S**,8a*S**)-1-Benzyl-5-(((*tert*-butyldimethylsilyl)oxy)methyl)-4-(3,5-dioxo-4-phenyl-1,2,4-triazolidin-1-yl)-7-phenyl-5,5a,7,8a-tetrahydroimidazo[4,5-*e*]isoindole-6,8(1H,4H)-dione S28
^{1}H (6h) - (4*S**,5*R**,5a*S**,8a*S**)-Diethyl 2-(1-benzyl-5-(((*tert*-butyldimethylsilyl)oxy)methyl)-6,8-dioxo-7-phenyl-1,4,5,5a,6,7,8,8a-octahhydroimidazo[4,5-*e*]isoindol-4-yl)-2-hydroxymalonate S29
^{13}C (6h) -(4*S**,5*R**,5a*S**,8a*S**)-Diethyl 2-(1-benzyl-5-(((*tert*-butyldimethylsilyl)oxy)methyl)-6,8-dioxo-7-phenyl-1,4,5,5a,6,7,8,8a-octahhydroimidazo[4,5-*e*]isoindol-4-yl)-2-hydroxymalonate S30
^{1}H (7a) - (*S**)-Ethyl 2-((4*S**,5a*S**,8a*S**)-1-benzyl-6,8-dioxo-7-phenyl-1,4,5,5a,6,7,8,8a-octahydroimidazo[4,5-*e*]isoindol-4-yl)-2-hydroxyacetate S31

^{13}C (7a) - (*S**)-Ethyl 2-((4*S**,5a*S**,8a*S**)-1-benzyl-6,8-dioxo-7-phenyl-1,4,5,5a,6,7,8,8a-octahydroimidazo[4,5-*e*]isoindol-4-yl)-2-hydroxyacetate S32
^{1}H (8a) - (*R**)-Ethyl 2-((4*S**,5a*S**,8a*S**)-1-benzyl-6,8-dioxo-7-phenyl-1,4,5,5a,6,7,8,8a-octahydroimidazo[4,5-*e*]isoindol-4-yl)-2-hydroxyacetate S33
^{13}C (8a) - (*R**)-Ethyl 2-((4*S**,5a*S**,8a*S**)-1-benzyl-6,8-dioxo-7-phenyl-1,4,5,5a,6,7,8,8a-octahydroimidazo[4,5-*e*]isoindol-4-yl)-2-hydroxyacetate S34
^{1}H (7b) - Ethyl 2-((5*S**,5a*S**,8a*S**)-1-benzyl-5-(((*tert*-butyldimethylsilyl)oxy)methyl)-6,8-dioxo-7-phenyl-1,4,5,5a,6,7,8,8a-octahydroimidazo[4,5-*e*]isoindol-4-yl)-2-hydroxyacetate S35
^{13}C (7b) - Ethyl 2-((5*S**,5a*S**,8a*S**)-1-benzyl-5-(((*tert*-butyldimethylsilyl)oxy)methyl)-6,8-dioxo-7-phenyl-1,4,5,5a,6,7,8,8a-octahydroimidazo[4,5-*e*]isoindol-4-yl)-2-hydroxyacetate S36
^{1}H (7c) - (*S**)-Ethyl 2-((4*S**,5a*S**,8a*S**)-1-benzyl-6,8-dioxo-7-phenyl-1,4,5,5a,6,7,8,8a-octahydroimidazo[4,5-*e*]isoindol-4-yl)-3,3,3-trifluoro-2-hydroxypropanoate S37
^{13}C (7c) - (*S**)-Ethyl 2-((4*S**,5a*S**,8a*S**)-1-benzyl-6,8-dioxo-7-phenyl-1,4,5,5a,6,7,8,8a-octahydroimidazo[4,5-*e*]isoindol-4-yl)-3,3,3-trifluoro-2-hydroxypropanoate S38
^{1}H (7d) - (*S**)-Ethyl 2-((4*S**,5*S**,5a*S**,8a*S**)-1-benzyl-5-(((*tert*-butyldimethylsilyl)oxy)methyl)-6,8-dioxo-7-phenyl-1,4,5,5a,6,7,8,8a-octahydroimidazo[4,5-*e*]isoindol-4-yl)-3,3,3-trifluoro-2-hydroxypropanoate S39
^{13}C (7d) - (*S**)-Ethyl 2-((4*S**,5*S**,5a*S**,8a*S**)-1-benzyl-5-(((*tert*-butyldimethylsilyl)oxy)methyl)-6,8-dioxo-7-phenyl-1,4,5,5a,6,7,8,8a-octahydroimidazo[4,5-*e*]isoindol-4-yl)-3,3,3-trifluoro-2-hydroxypropanoate S40
^{1}H (8d) - (*R**)-Ethyl 2-((4*S**,5*S**,5a*S**,8a*S**)-1-benzyl-5-(((*tert*-butyldimethylsilyl)oxy)methyl)-6,8-dioxo-7-phenyl-1,4,5,5a,6,7,8,8a-octahydroimidazo[4,5-*e*]isoindol-4-yl)-3,3,3-trifluoro-2-hydroxypropanoate S41
^{13}C (8d) - (*R**)-Ethyl 2-((4*S**,5*S**,5a*S**,8a*S**)-1-benzyl-5-(((*tert*-butyldimethylsilyl)oxy)methyl)-6,8-dioxo-7-phenyl-1,4,5,5a,6,7,8,8a-octahydroimidazo[4,5-*e*]isoindol-4-yl)-3,3,3-trifluoro-2-hydroxypropanoate S42

Molecular structure of compound 6b

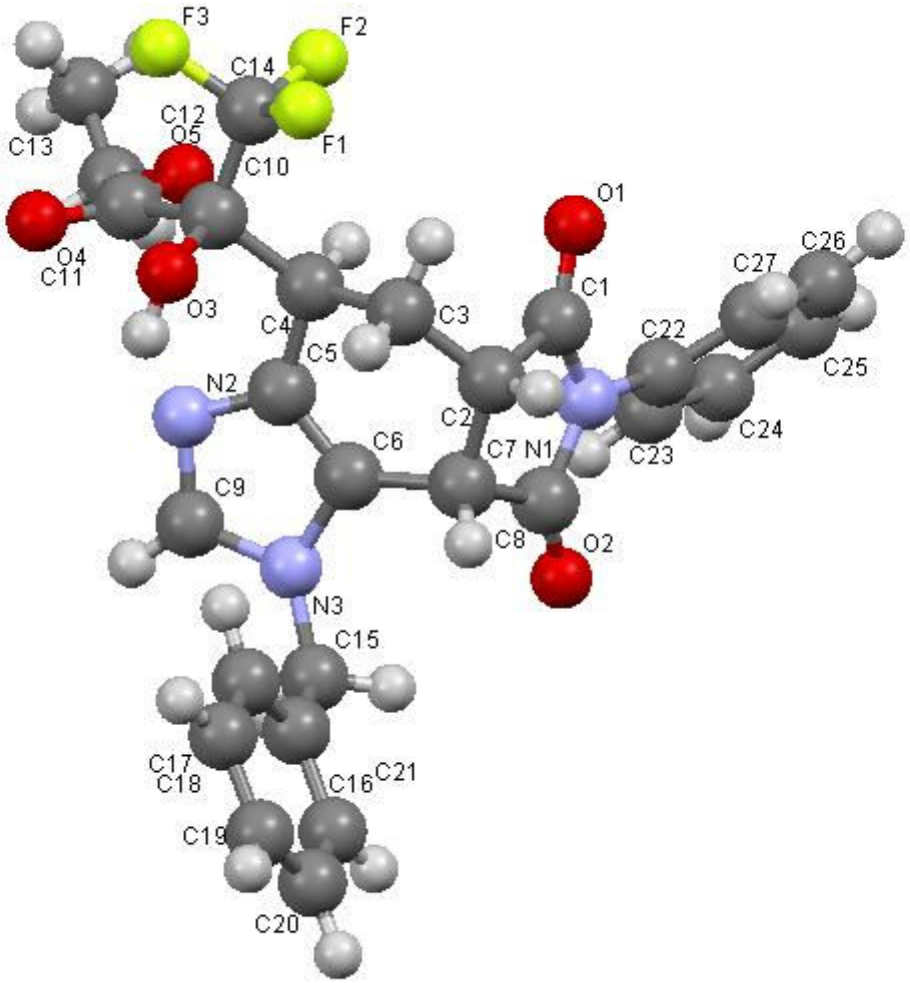


The benzyl group attached to N3 is disordered over three orientations, and the *o*-tolyl group attached to N4 is disordered over two orientations. The disorder was satisfactorily modelled with the aid of restraints. The small and weakly scattering crystal required synchrotron radiation for data collection. The largest residual electron density features may be due to disordered solvent, but it was not possible to develop a satisfactory model for this, and the contribution is very small in any case.

Table 1. Crystal data and structure refinement for **6b**.

Identification code	mjh71	
Chemical formula (moiety)	$C_{29}H_{26}N_4O_3$	
Chemical formula (total)	$C_{29}H_{26}N_4O_3$	
Formula weight	478.54	
Temperature	150(2) K	
Radiation, wavelength	synchrotron, 0.6889 Å	
Crystal system, space group	monoclinic, $P2_1/n$	
Unit cell parameters	a = 17.055(3) Å	α = 90°
	b = 7.5478(12) Å	β = 100.937(2)°
	c = 19.591(3) Å	γ = 90°
Cell volume	2476.1(7) $Å^3$	
Z	4	
Calculated density	1.284 g/cm^3	
Absorption coefficient μ	0.085 mm^{-1}	
F(000)	1008	
Crystal colour and size	yellow, 0.08 × 0.08 × 0.05 mm^3	
Reflections for cell refinement	9917 (θ range 2.4 to 27.5°)	
Data collection method	Crystal Logic diffractometer and Rigaku Saturn 724+ CCD thick-slice ω scans	
θ range for data collection	1.4 to 27.6°	
Index ranges	h –22 to 19, k –10 to 10, l –25 to 26	
Completeness to θ = 27.6°	97.6 %	
Reflections collected	24824	
Independent reflections	6127 (R_{int} = 0.0381)	
Reflections with $F^2>2\sigma$	4800	
Absorption correction	semi-empirical from equivalents	
Min. and max. transmission	0.993 and 0.996	
Structure solution	direct methods	
Refinement method	Full-matrix least-squares on F^2	
Weighting parameters a, b	0.0936, 1.0836	
Data / restraints / parameters	6127 / 818 / 523	
Final R indices [$F^2>2\sigma$]	R1 = 0.0614, wR2 = 0.1782	
R indices (all data)	R1 = 0.0742, wR2 = 0.1871	
Goodness-of-fit on F^2	1.043	
Extinction coefficient	0.054(6)	
Largest and mean shift/su	0.002 and 0.000	
Largest diff. peak and hole	1.04 and –0.19 e $Å^{-3}$	

Molecular structure of compound 7c



The crystal was very weakly scattering and, even with synchrotron radiation, gave only a poor quality data set. The structure is of relatively low precision, but serves the purpose of confirming the identity of the compound and the relative stereochemistry. Twinning is likely, but a simple twin law could not be found. There is no resolvable disorder in the structure.

Table 8. Crystal data and structure refinement for **7c.**

Identification code	mjh68	
Chemical formula (moiety)	$C_{27}H_{23}F_3N_3O_5$	
Chemical formula (total)	$C_{27}H_{23}F_3N_3O_5$	
Formula weight	526.48	
Temperature	120(2) K	
Radiation, wavelength	synchrotron, 0.6889 Å	
Crystal system, space group	monoclinic, $P2_1/c$	
Unit cell parameters	a = 10.988(10) Å	α = 90°
	b = 7.353(7) Å	β = 95.027(8)°
	c = 29.70(3) Å	γ = 90°
Cell volume	2390(4) $Å^3$	
Z	4	
Calculated density	1.463 g/cm^3	
Absorption coefficient μ	0.117 mm^{-1}	
F(000)	1092	
Crystal colour and size	colourless, 0.10 × 0.05 × 0.05 mm^3	
Reflections for cell refinement	4077 (θ range 2.3 to 25.5°)	
Data collection method	Crystal Logic diffractometer and Rigaku Saturn 724+ CCD thick-slice ω scans	
θ range for data collection	1.3 to 21.2°	
Index ranges	h −11 to 11, k −7 to 7, l −31 to 31	
Completeness to θ = 21.2°	99.9 %	
Reflections collected	21186	
Independent reflections	2913 (R_{int} = 0.1064)	
Reflections with $F^2>2\sigma$	1923	
Absorption correction	semi-empirical from equivalents	
Min. and max. transmission	0.988 and 0.994	
Structure solution	direct methods	
Refinement method	Full-matrix least-squares on F^2	
Weighting parameters a, b	0.2000, 0.0000	
Data / restraints / parameters	2913 / 396 / 344	
Final R indices [$F^2>2\sigma$]	R1 = 0.2297, wR2 = 0.6078	
R indices (all data)	R1 = 0.2693, wR2 = 0.6335	
Goodness-of-fit on F^2	2.681	
Extinction coefficient	0.20(5)	
Largest and mean shift/su	0.000 and 0.000	
Largest diff. peak and hole	0.59 and −0.46 e $Å^{-3}$	

Molecular structure of compound 7d

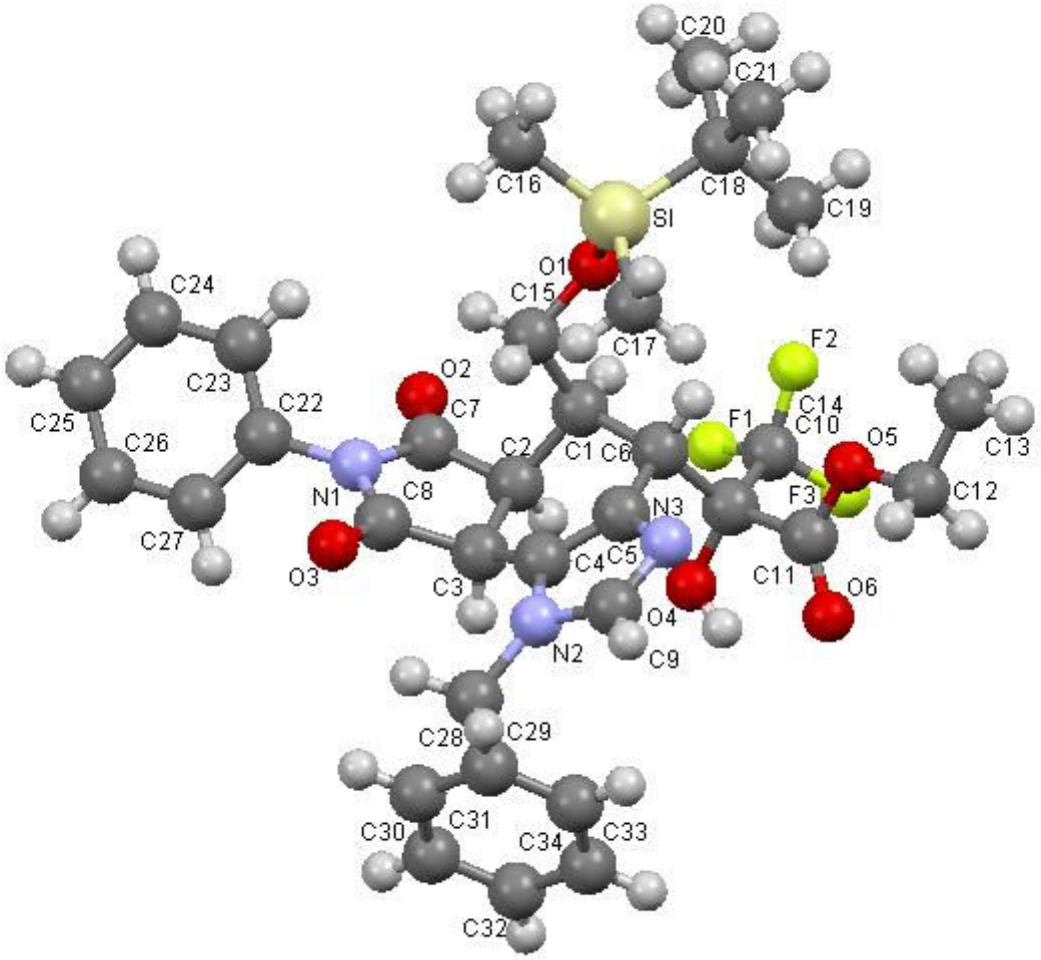


Table 15. Crystal data and structure refinement for **7d**.

Identification code	mjh74_compound_9d	
Chemical formula (moiety)	$C_{34}H_{40}F_3N_3O_6Si$	
Chemical formula (total)	$C_{34}H_{40}F_3N_3O_6Si$	
Formula weight	671.78	
Temperature	150(2) K	
Radiation, wavelength	MoKα, 0.71073 Å	
Crystal system, space group	triclinic, $P\bar{1}$	
Unit cell parameters	a = 10.8328(6) Å	α = 72.909(5)°
	b = 12.7806(8) Å	β = 79.316(4)°
	c = 13.3203(6) Å	γ = 71.622(6)°
Cell volume	1664.08(16) $Å^3$	
Z	2	
Calculated density	1.341 g/cm^3	
Absorption coefficient μ	0.136 mm^{-1}	
F(000)	708	
Crystal colour and size	yellow, 0.34 × 0.30 × 0.30 mm^3	
Reflections for cell refinement	6507 (θ range 3.0 to 28.5°)	
Data collection method	Oxford Diffraction Gemini A Ultra diffractometer	
	ω scans	
θ range for data collection	3.0 to 28.6°	
Index ranges	h –13 to 14, k –17 to 15, l –17 to 17	
Completeness to θ = 25.0°	99.8 %	
Reflections collected	15021	
Independent reflections	6980 (R_{int} = 0.0266)	
Reflections with $F^2>2\sigma$	5675	
Absorption correction	semi-empirical from equivalents	
Min. and max. transmission	0.955 and 0.960	
Structure solution	direct methods	
Refinement method	Full-matrix least-squares on F^2	
Weighting parameters a, b	0.0375, 0.8194	
Data / restraints / parameters	6980 / 0 / 435	
Final R indices [$F^2>2\sigma$]	R1 = 0.0410, wR2 = 0.0919	
R indices (all data)	R1 = 0.0544, wR2 = 0.1012	
Goodness-of-fit on F^2	1.031	
Extinction coefficient	0.0030(8)	
Largest and mean shift/su	0.001 and 0.000	
Largest diff. peak and hole	0.34 and –0.31 e $Å^{-3}$	

Molecular structure of compound 8a

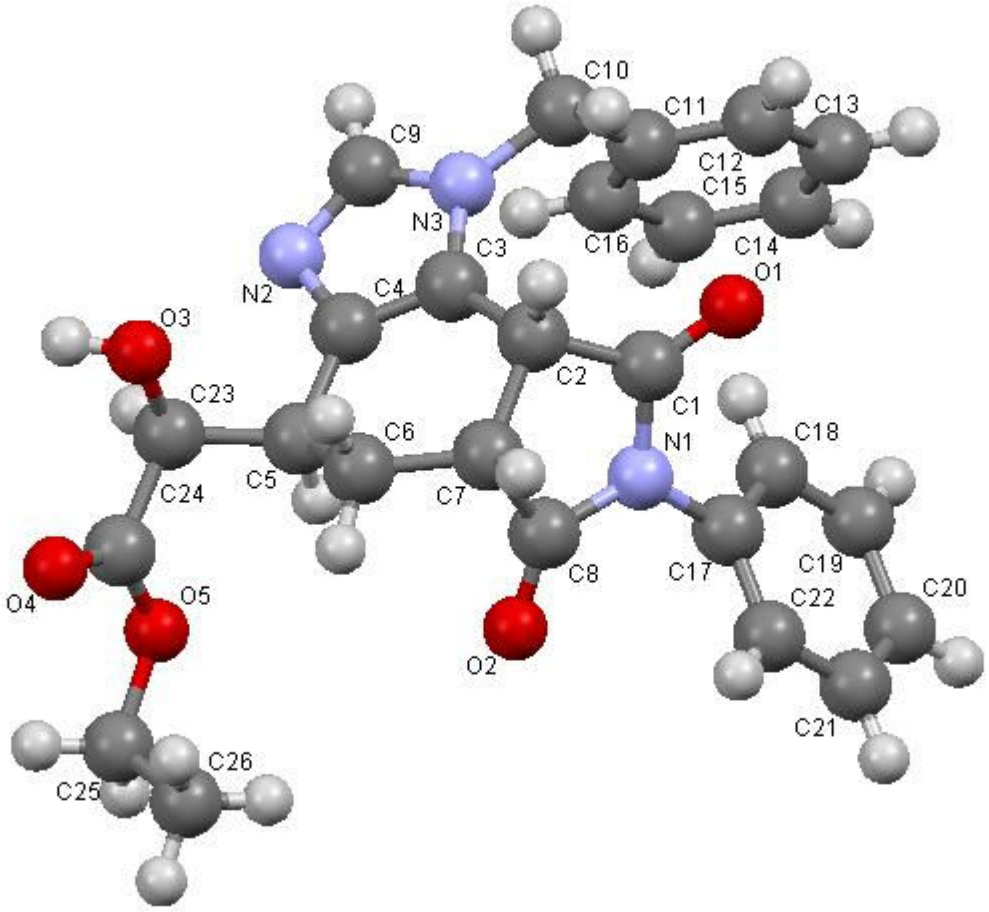
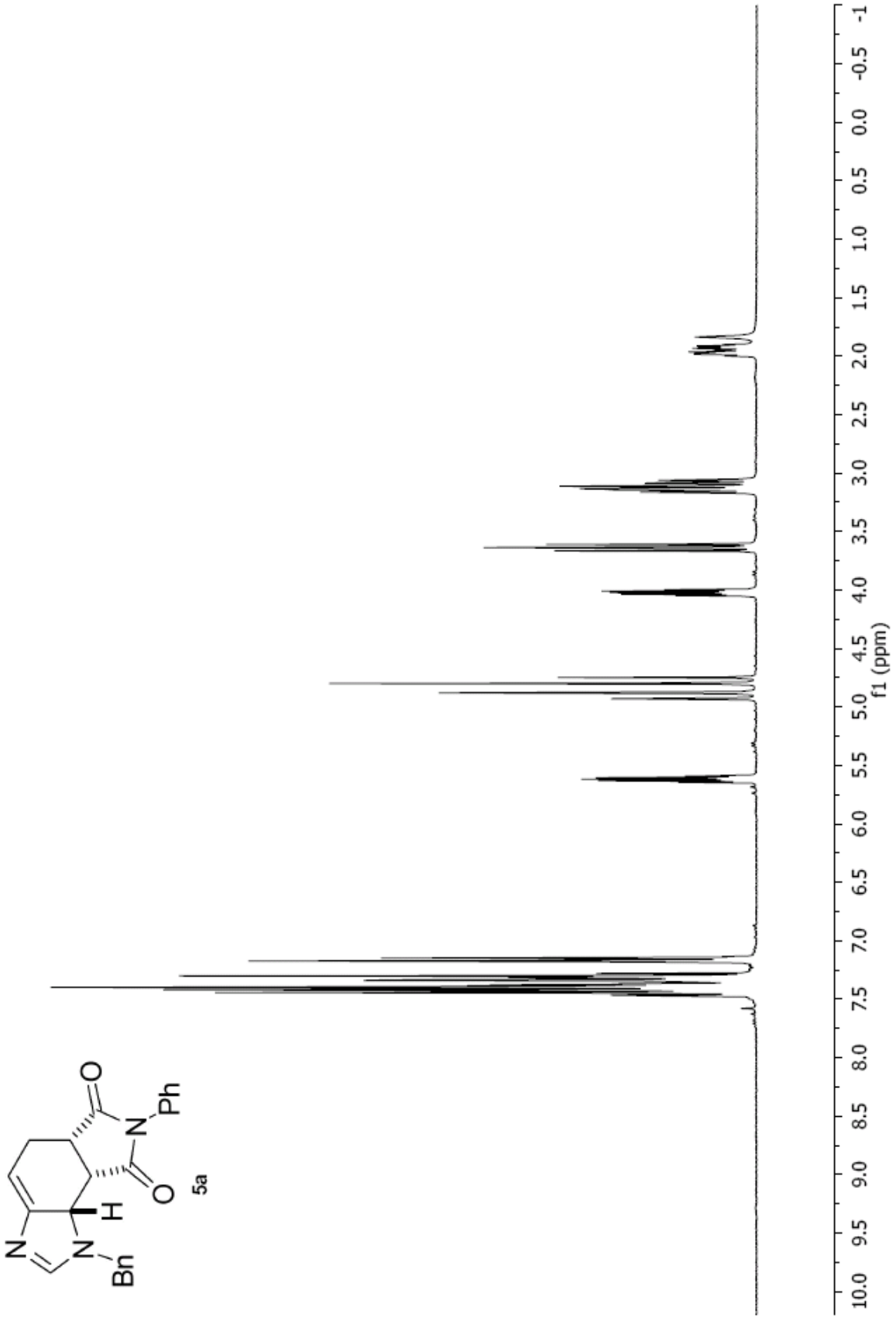
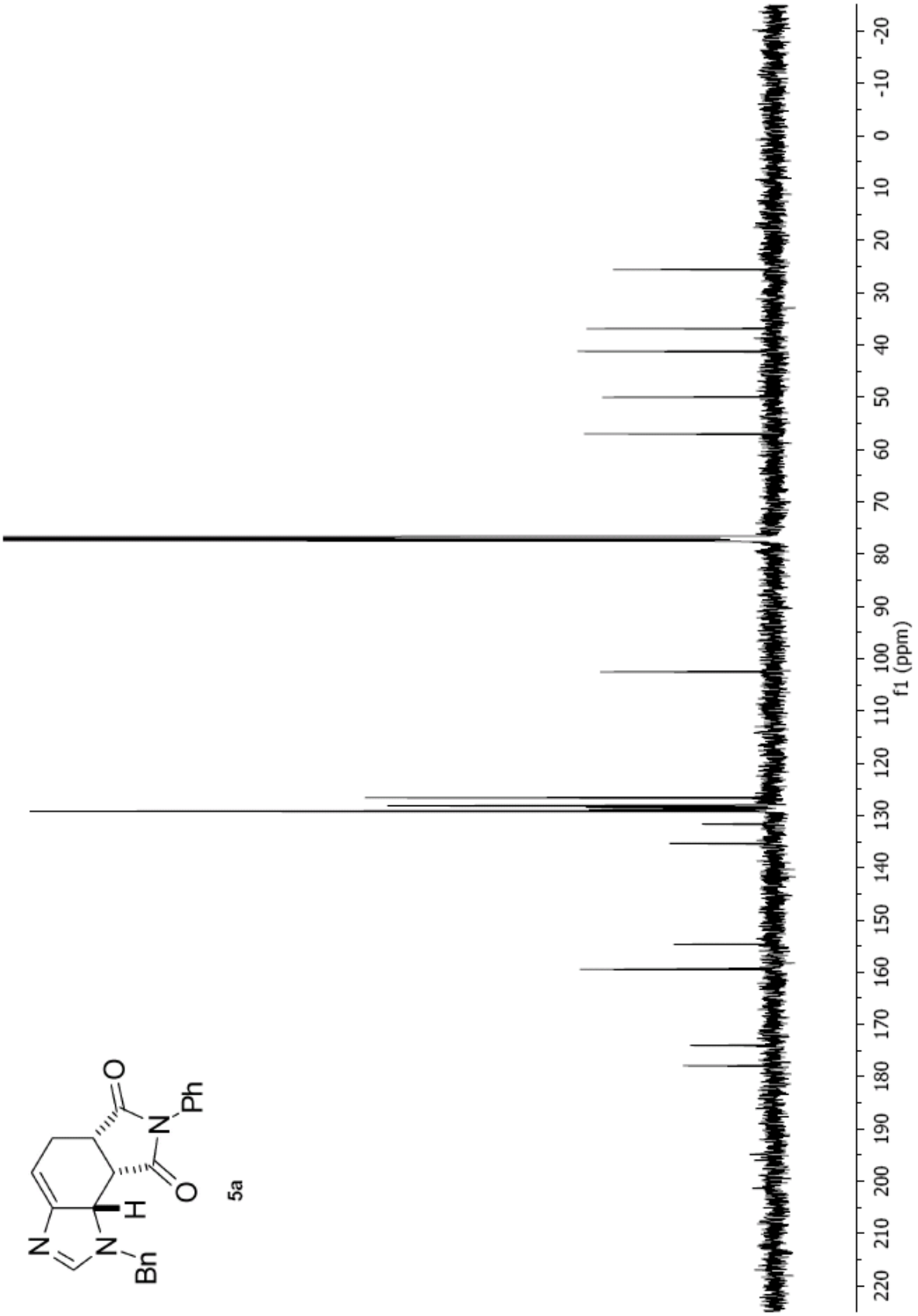
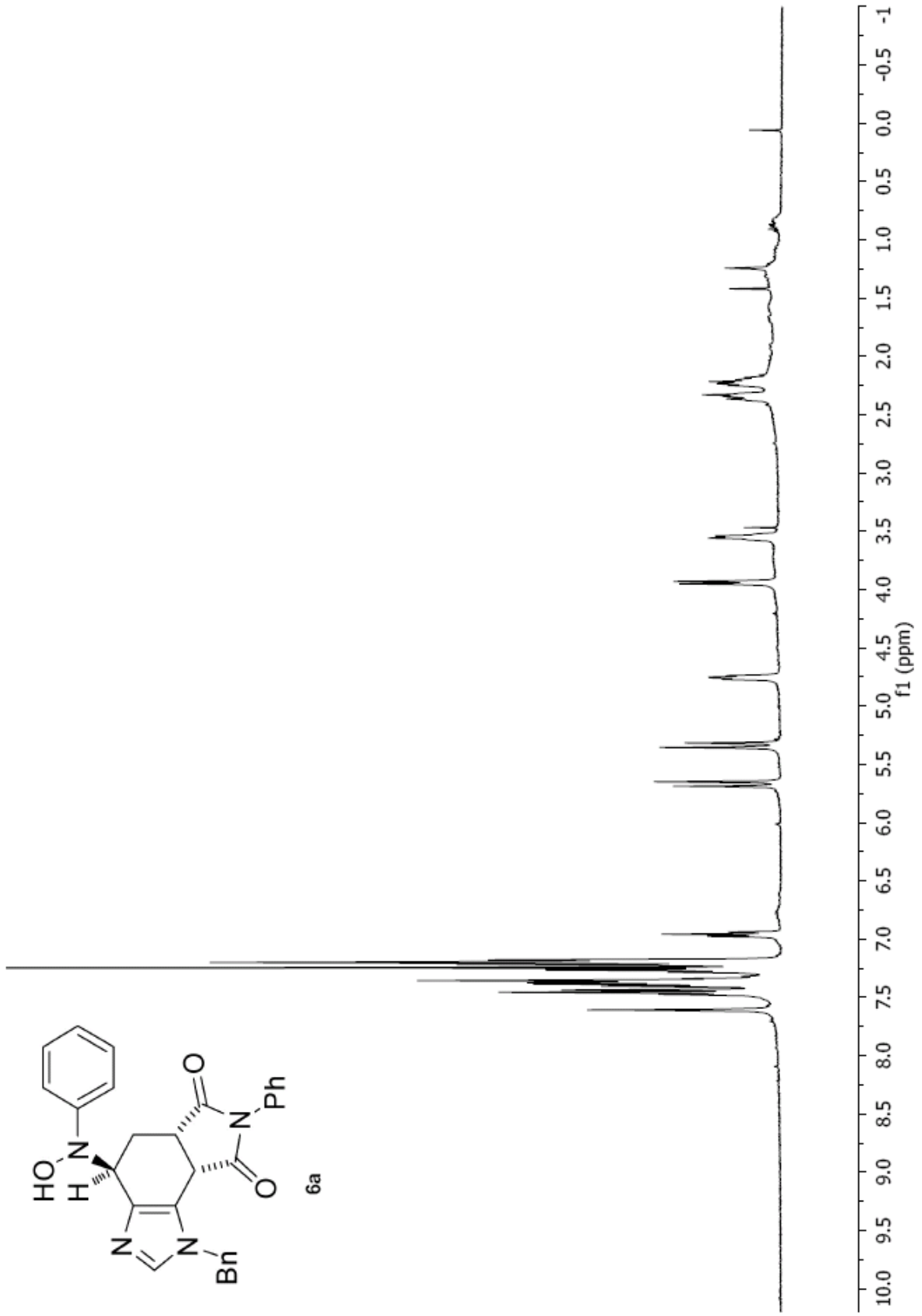


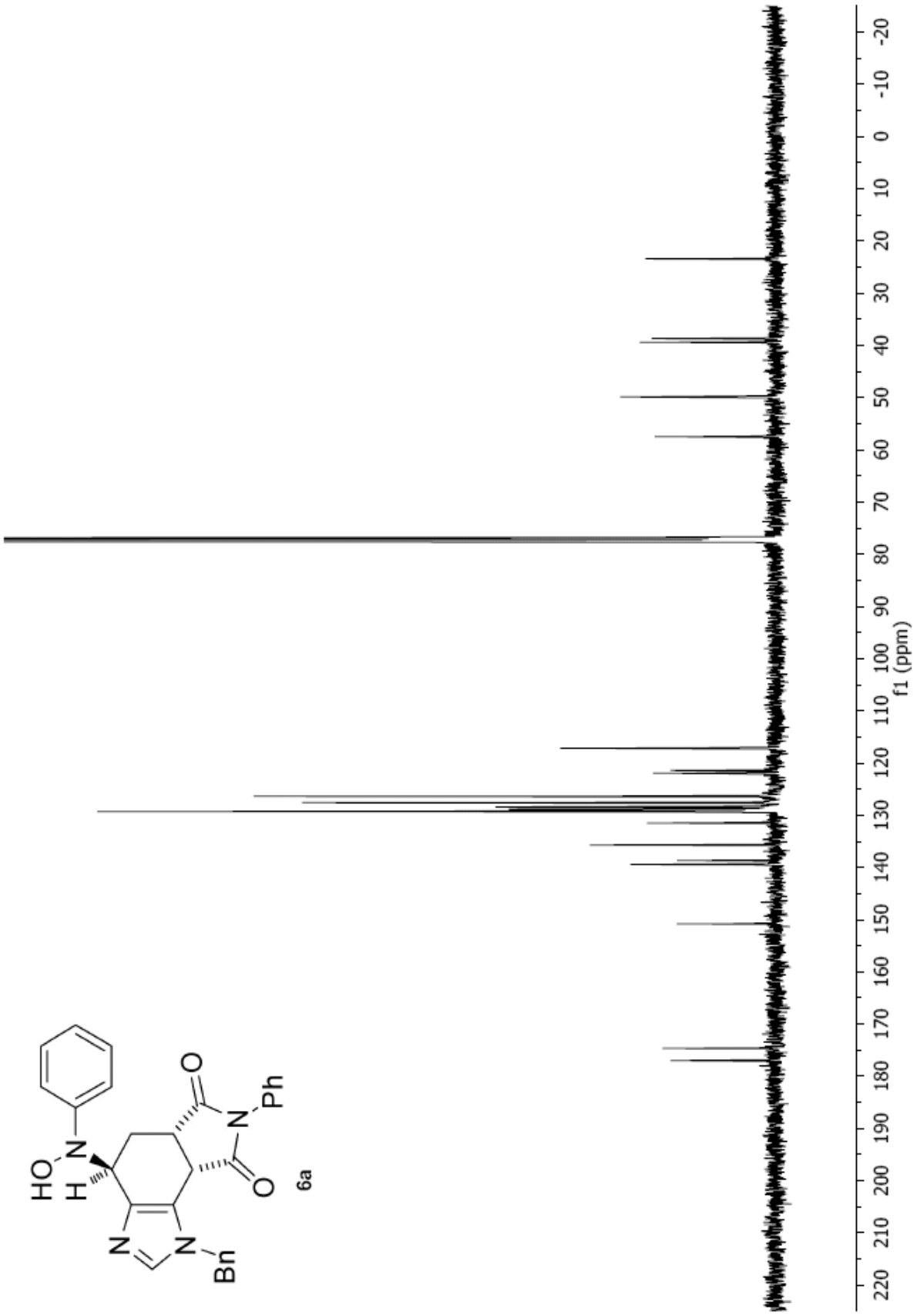
Table 22. Crystal data and structure refinement for **8a**.

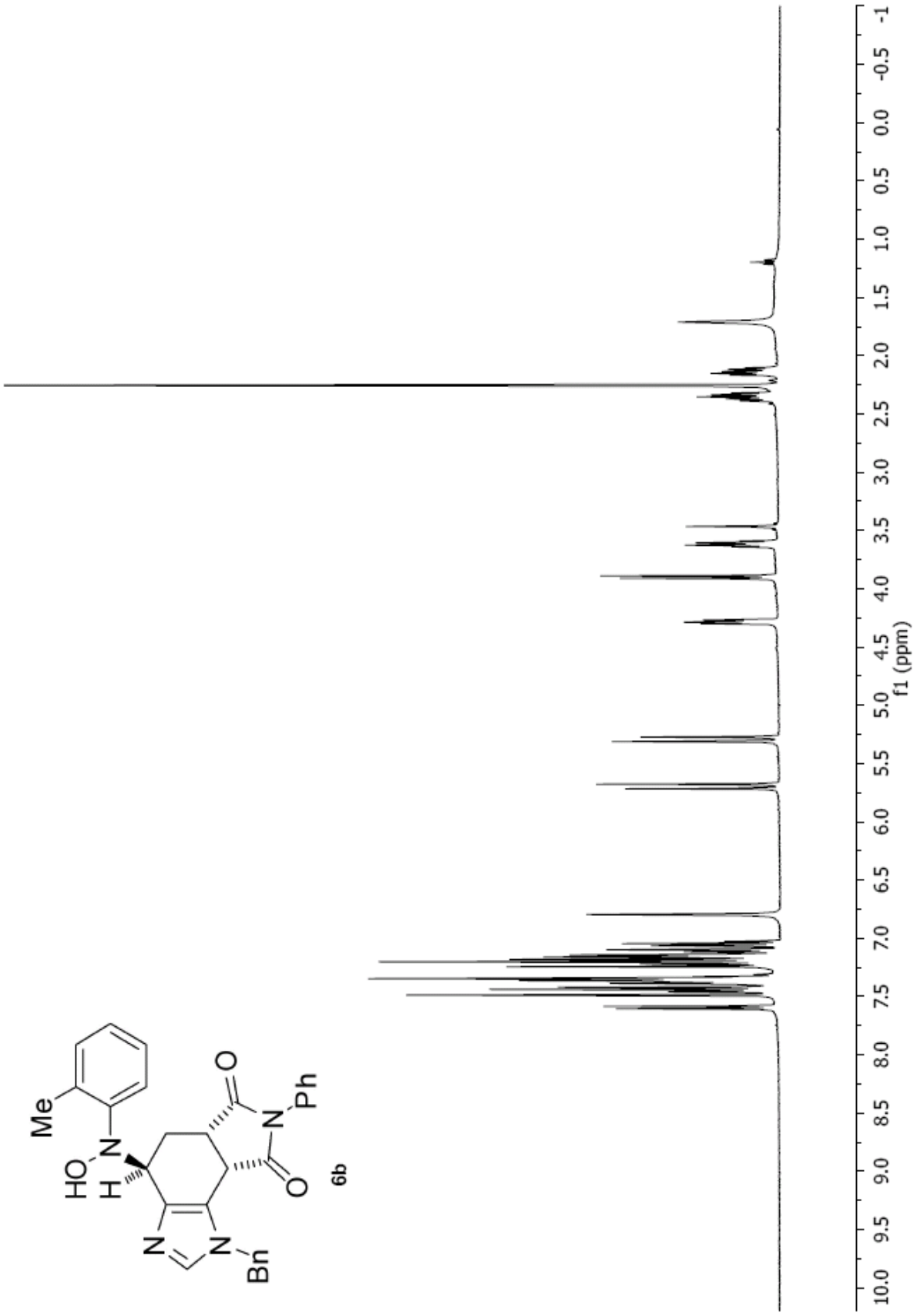
Identification code	mjh55	
Chemical formula (moiety)	$C_{26}H_{25}N_3O_5$	
Chemical formula (total)	$C_{26}H_{25}N_3O_5$	
Formula weight	459.49	
Temperature	150(2) K	
Radiation, wavelength	MoKα, 0.71073 Å	
Crystal system, space group	triclinic, $P\bar{1}$	
Unit cell parameters	a = 8.0807(3) Å	α = 79.423(3)°
	b = 11.5619(5) Å	β = 86.322(3)°
	c = 12.7860(5) Å	γ = 82.872(3)°
Cell volume	1164.15(8) $Å^3$	
Z	2	
Calculated density	1.311 g/cm^3	
Absorption coefficient μ	0.092 mm^{-1}	
F(000)	484	
Crystal colour and size	colourless, 0.34 × 0.30 × 0.30 mm^3	
Reflections for cell refinement	6804 (θ range 2.9 to 28.4°)	
Data collection method	Oxford Diffraction Gemini A Ultra diffractometer	
	ω scans	
θ range for data collection	2.9 to 28.5°	
Index ranges	h –9 to 10, k –11 to 15, l –14 to 16	
Completeness to θ = 26.0°	97.5 %	
Reflections collected	9971	
Independent reflections	4854 (R_{int} = 0.0172)	
Reflections with $F^2>2\sigma$	3963	
Absorption correction	semi-empirical from equivalents	
Min. and max. transmission	0.969 and 0.973	
Structure solution	direct methods	
Refinement method	Full-matrix least-squares on F^2	
Weighting parameters a, b	0.0619, 0.0759	
Data / restraints / parameters	4854 / 0 / 313	
Final R indices [$F^2>2\sigma$]	R1 = 0.0348, wR2 = 0.0996	
R indices (all data)	R1 = 0.0429, wR2 = 0.1020	
Goodness-of-fit on F^2	1.122	
Extinction coefficient	0.023(3)	
Largest and mean shift/su	0.001 and 0.000	
Largest diff. peak and hole	0.35 and –0.22 e $Å^{-3}$	

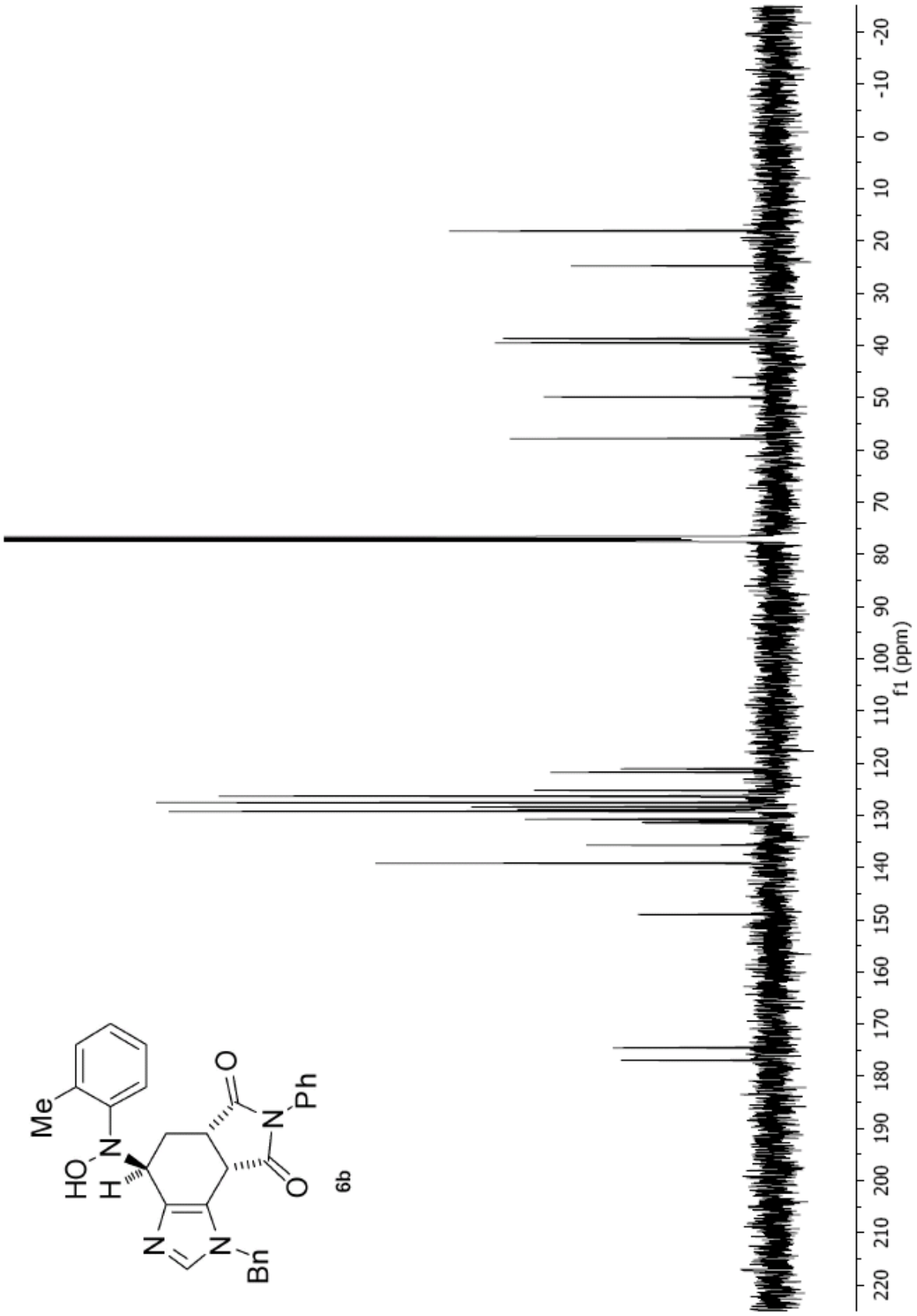


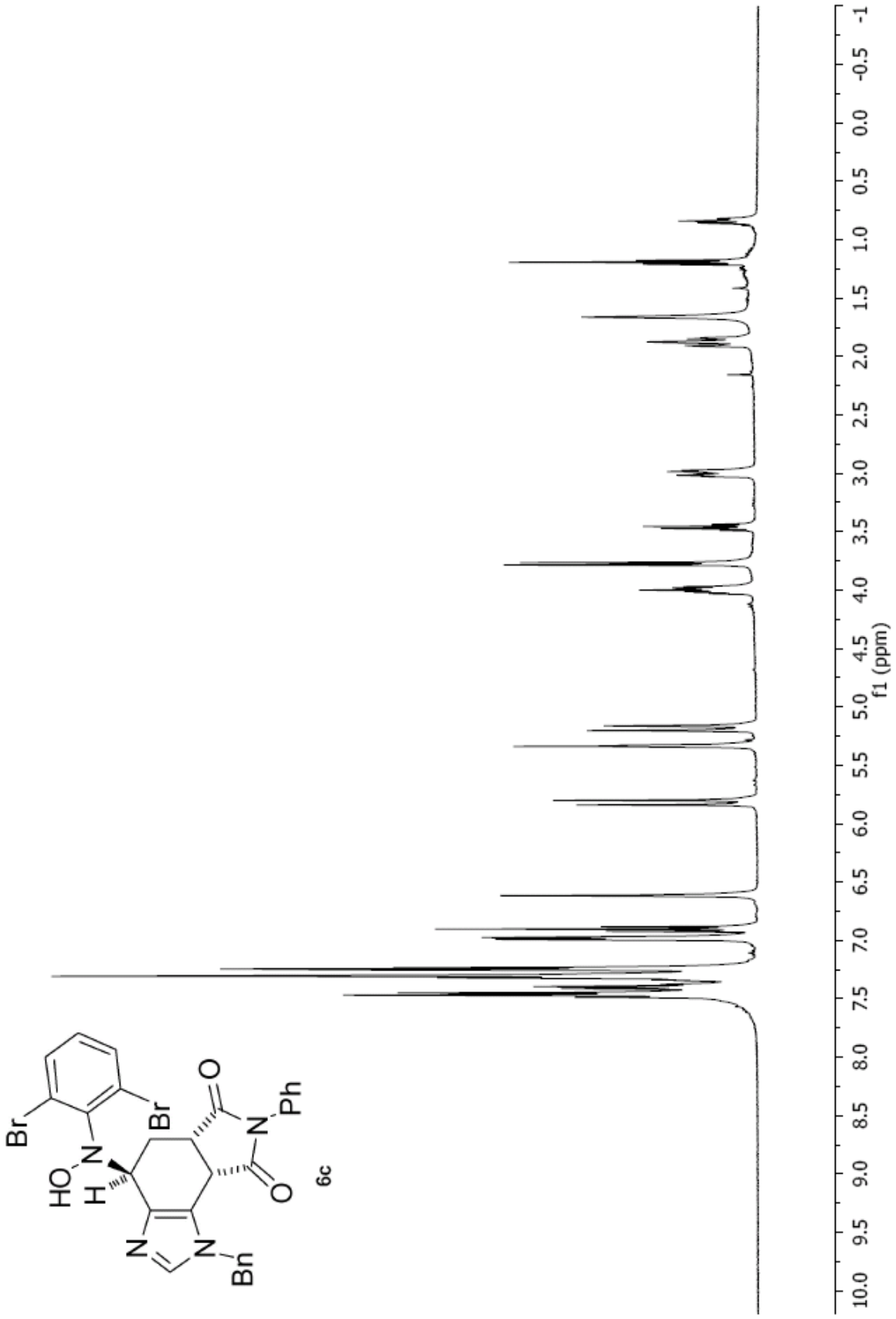


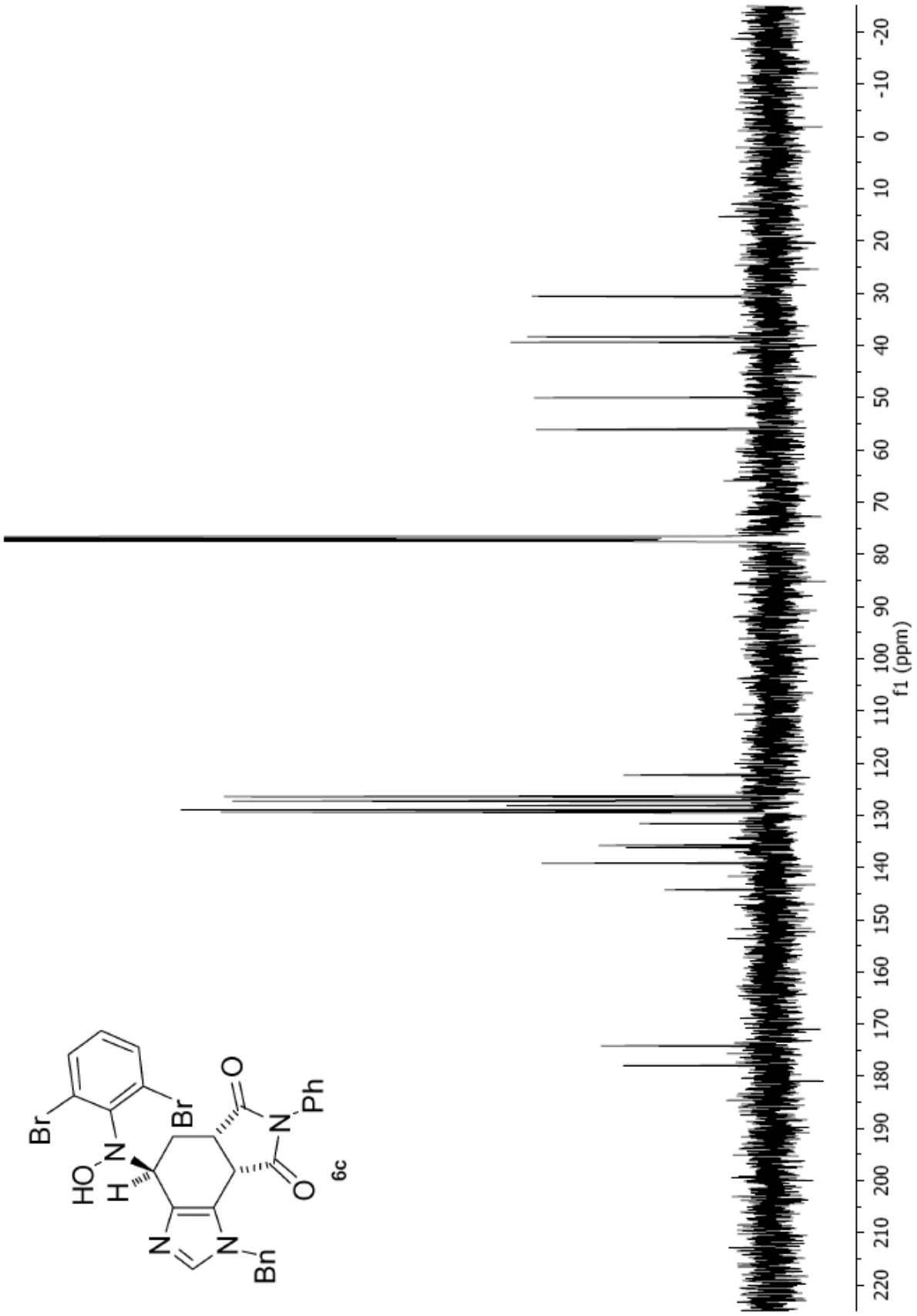


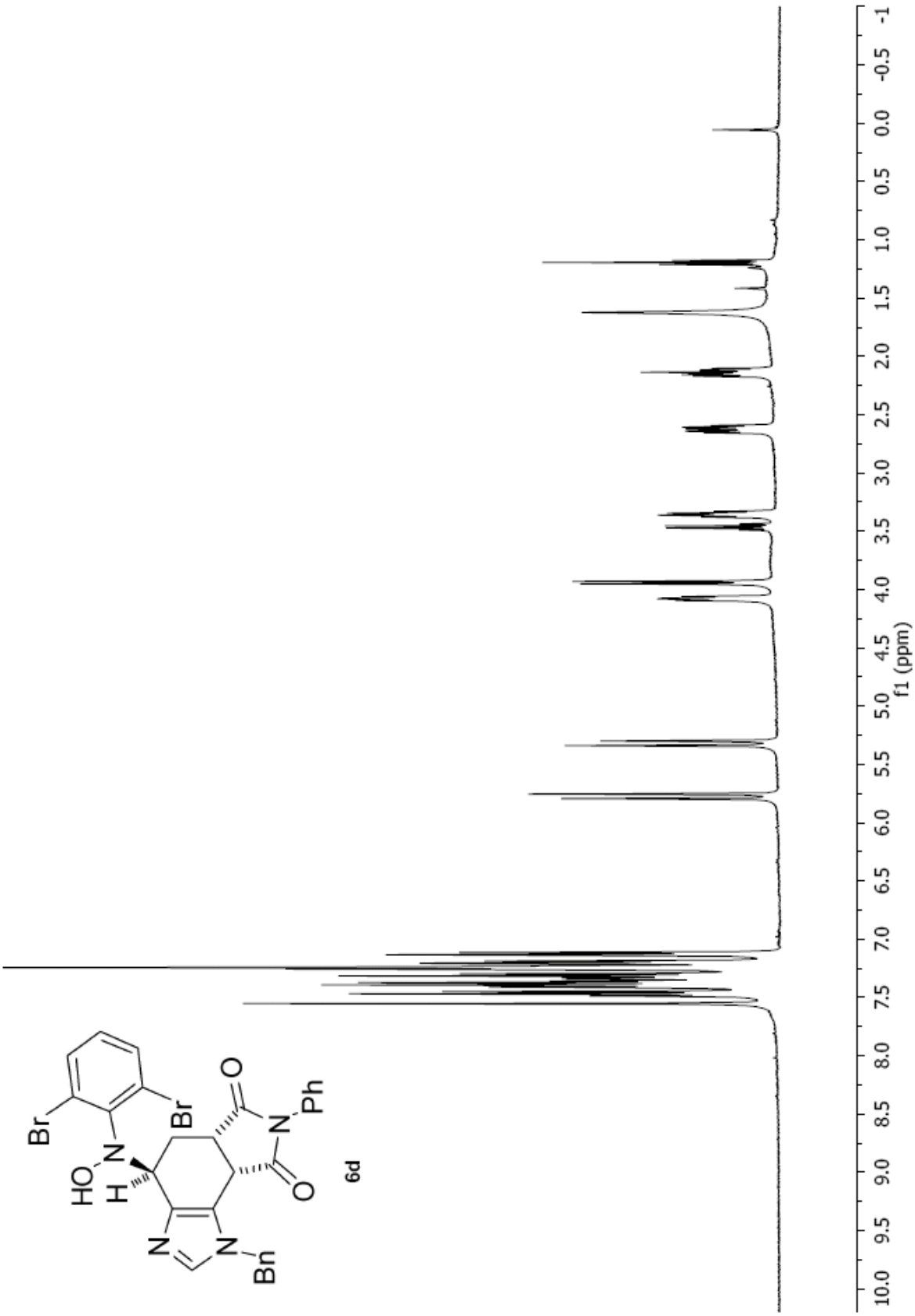


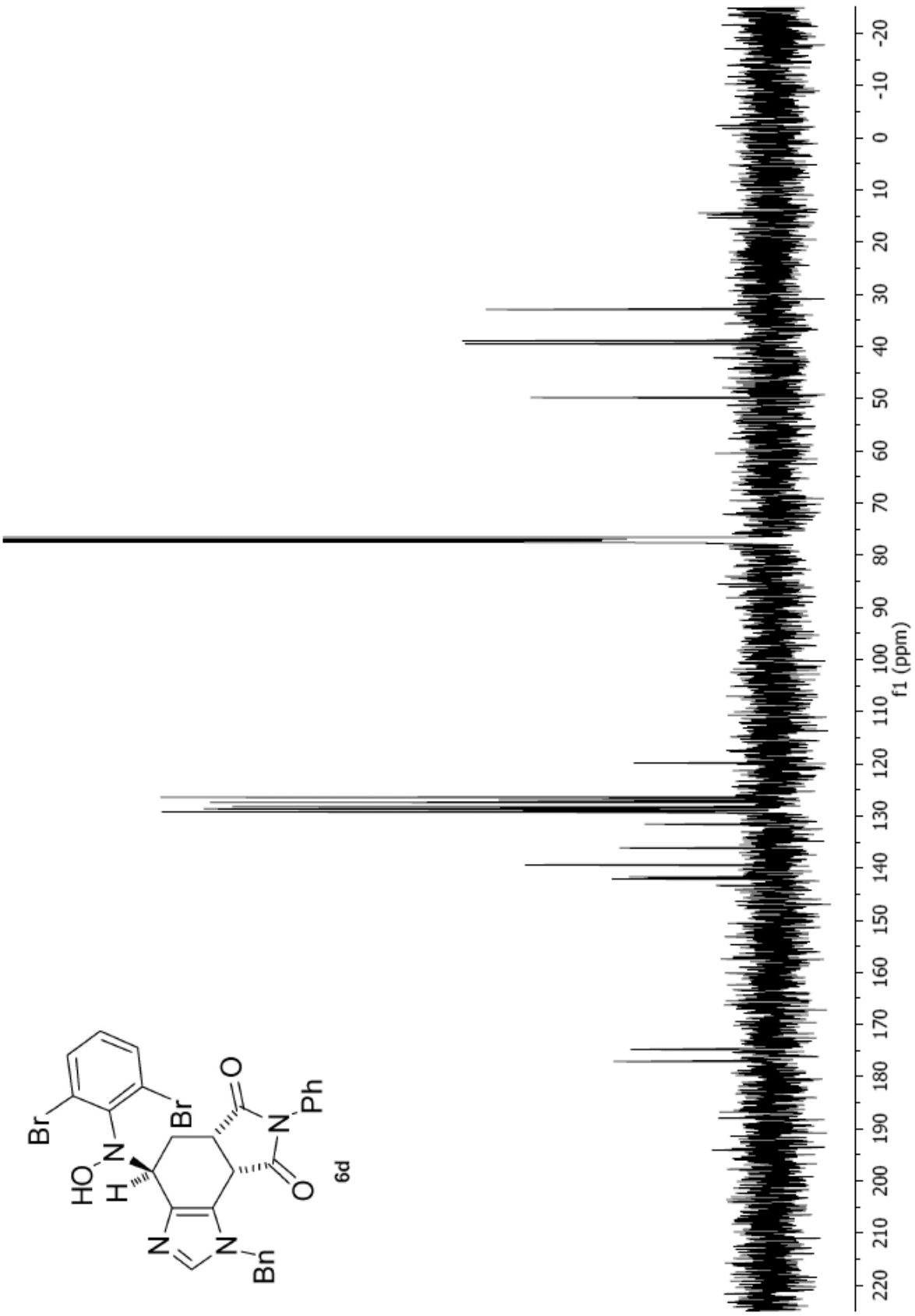


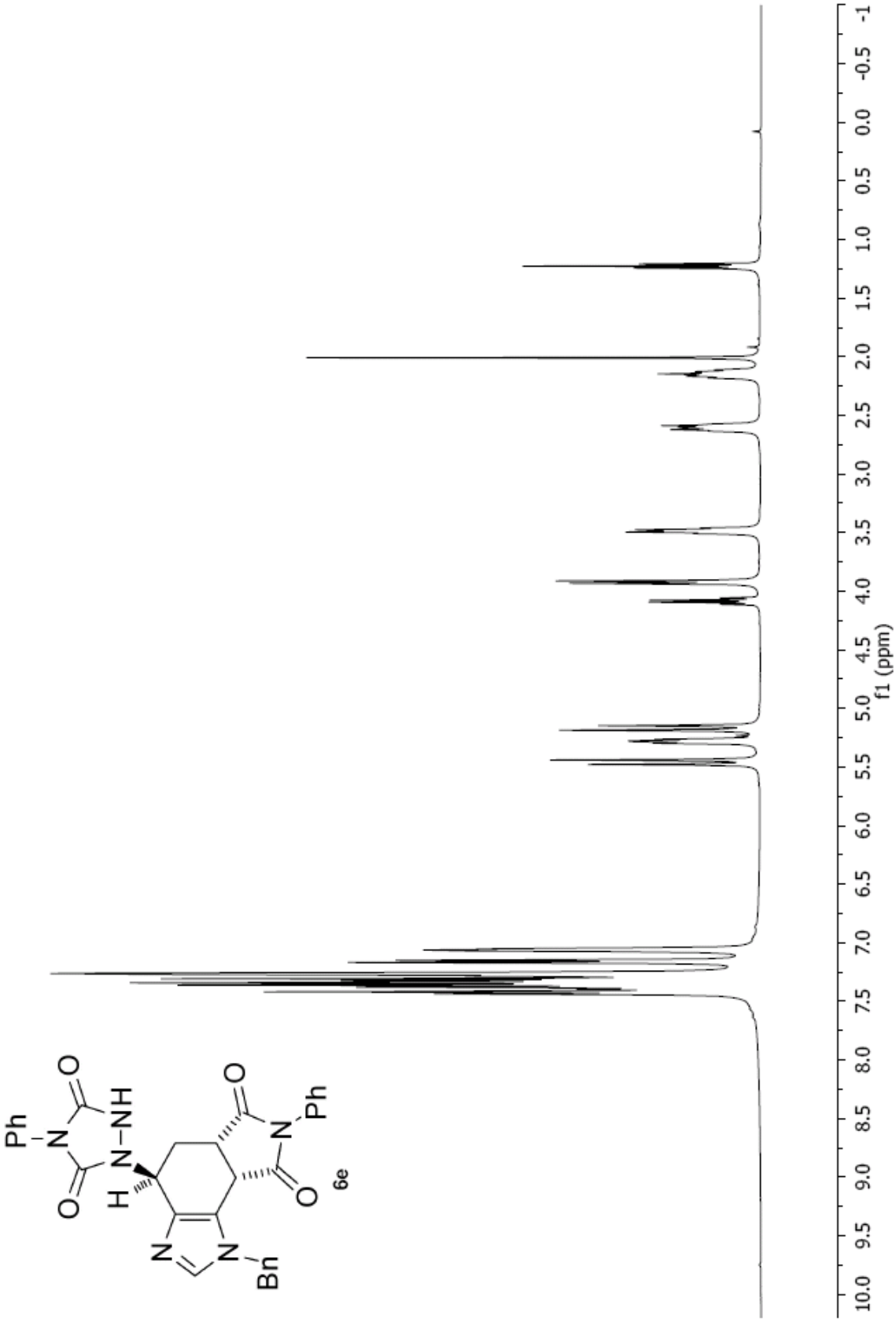


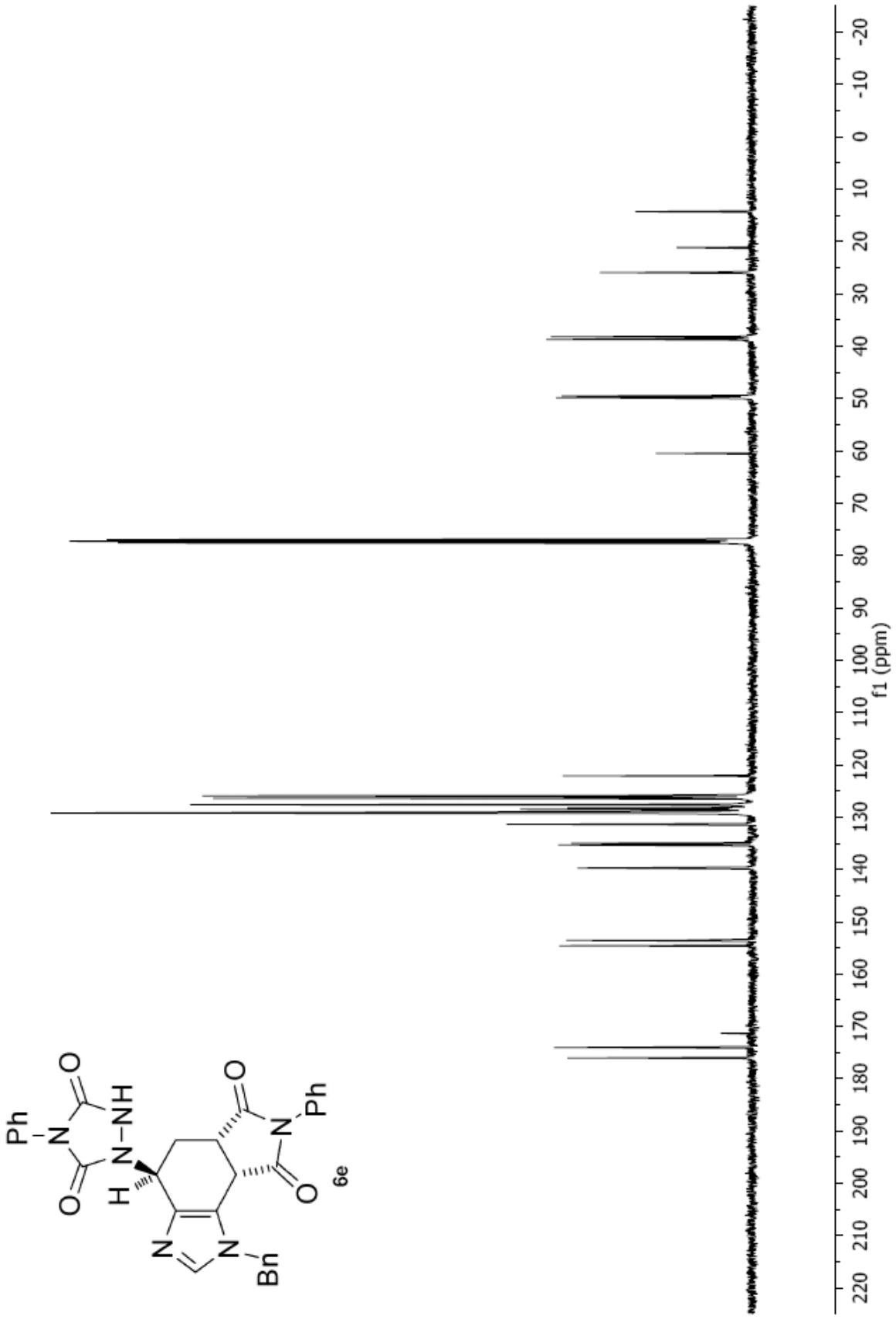


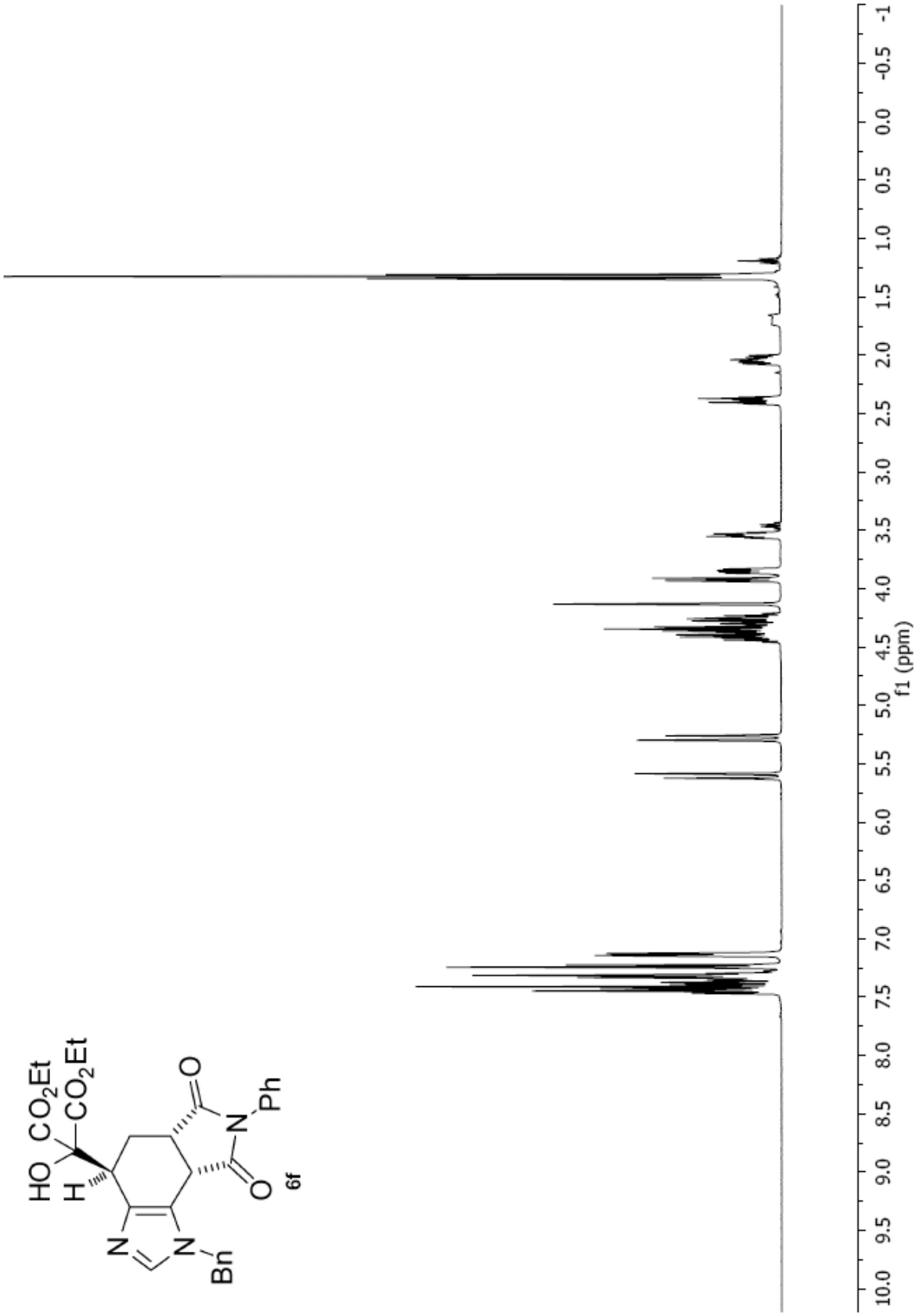


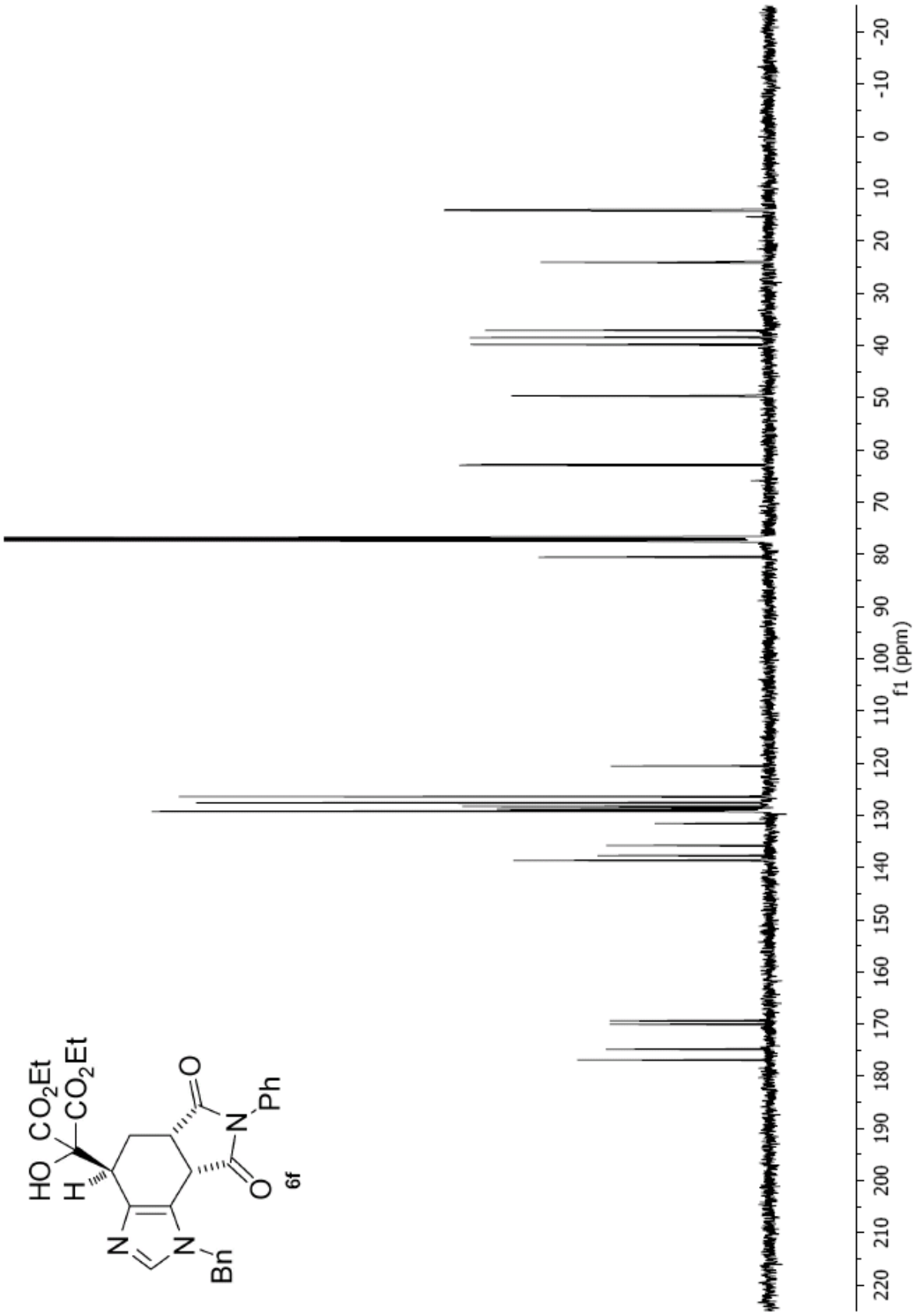



f1 (ppm)
-20
-10
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
220
Br
Br
HO
N
H
N
O
N
Ph
O
N
N
Bn
6d






HO
CO2Et
H
CO2Et
O
N
Ph
O
6f
N
N
Bn
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)


HO
CO2Et
H
CO2Et
O
N
Ph
O
N
N
Bn
6f
f1 (ppm)
-20
-10
0
10
20
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40
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80
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120
130
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150
160
170
180
190
200
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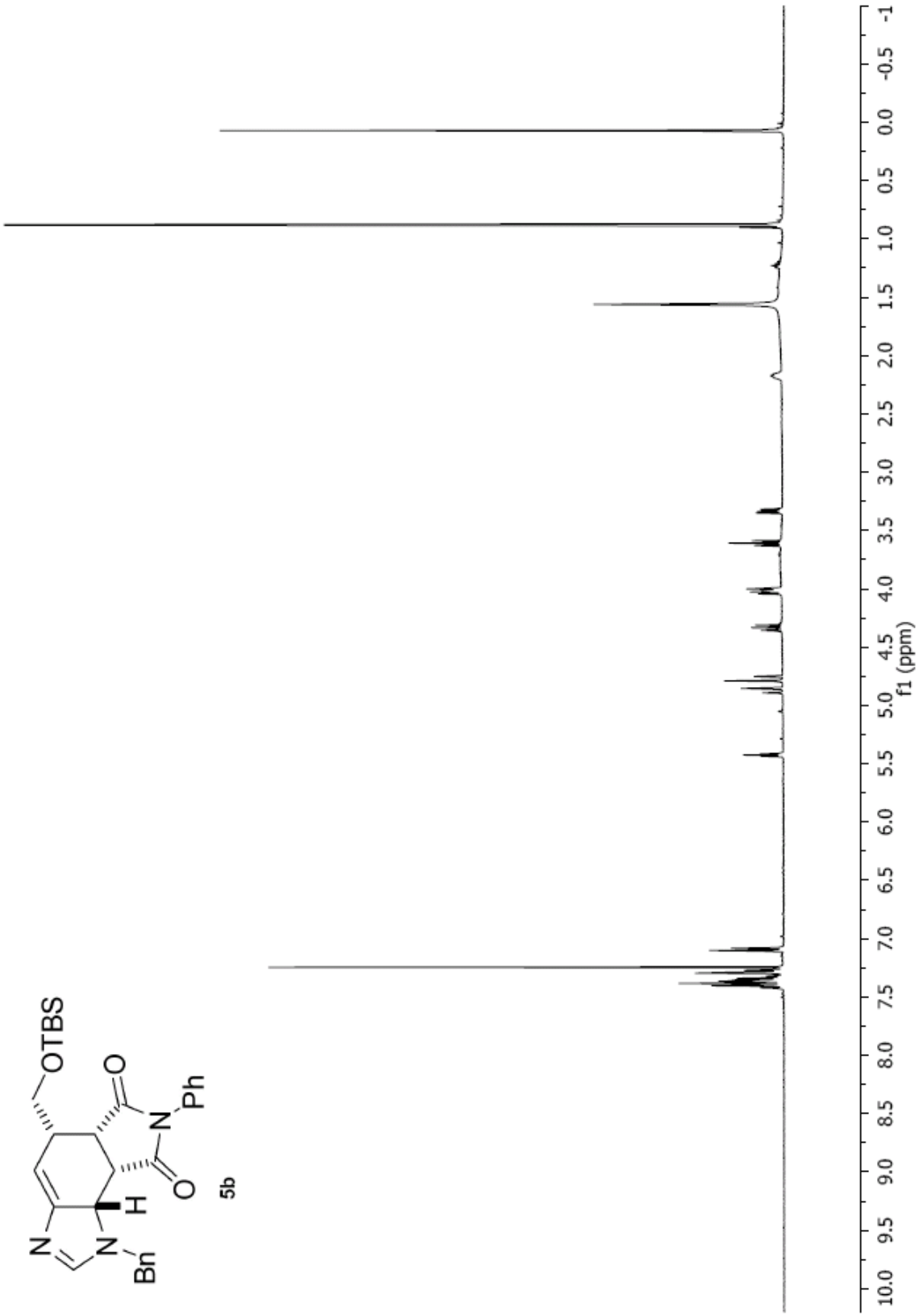
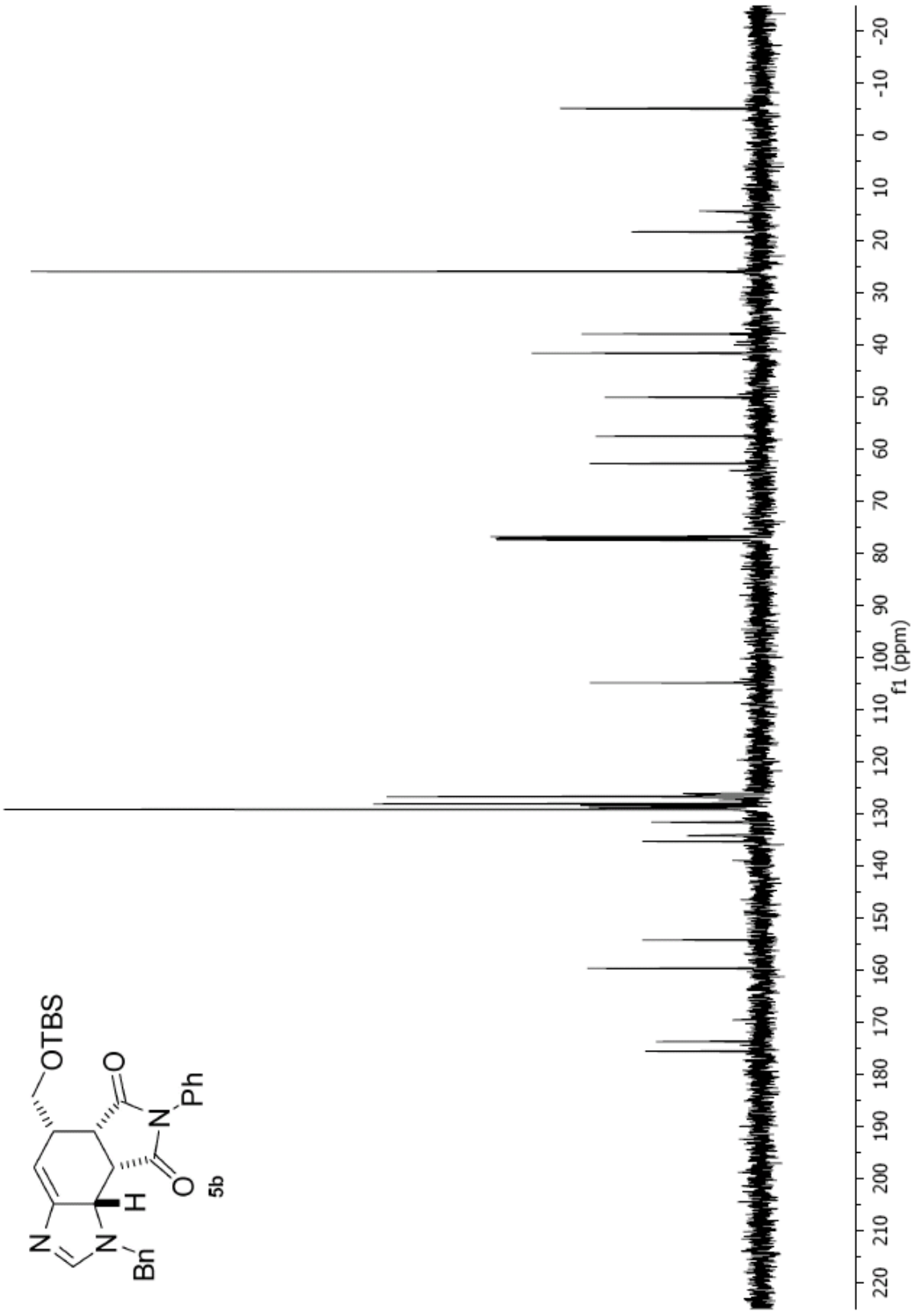
220




OTBS
O
N
Ph
O
H
N
N
Bn
5b
f1 (ppm)
-20
-10
0
10
20
30
40
50
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70
80
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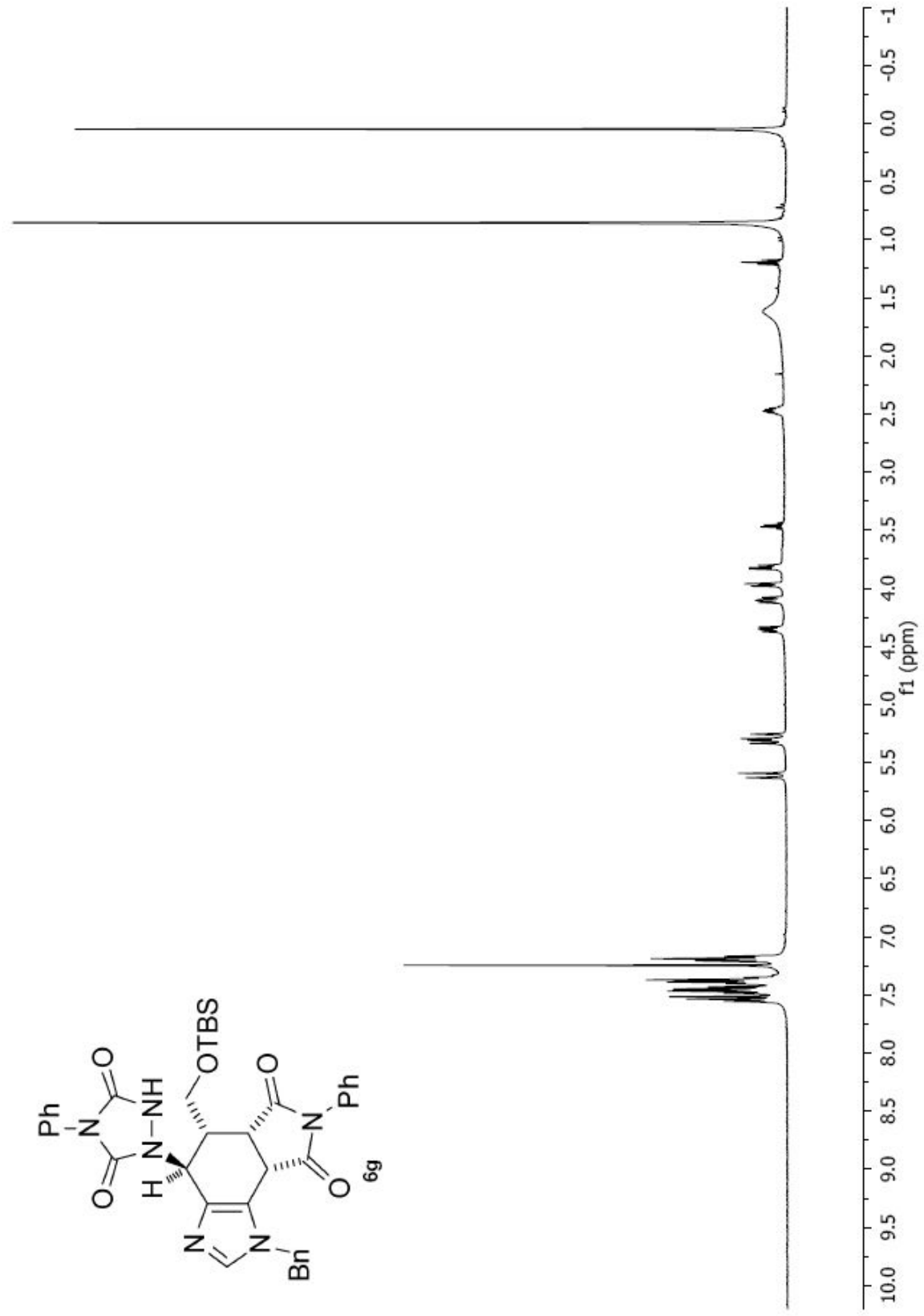
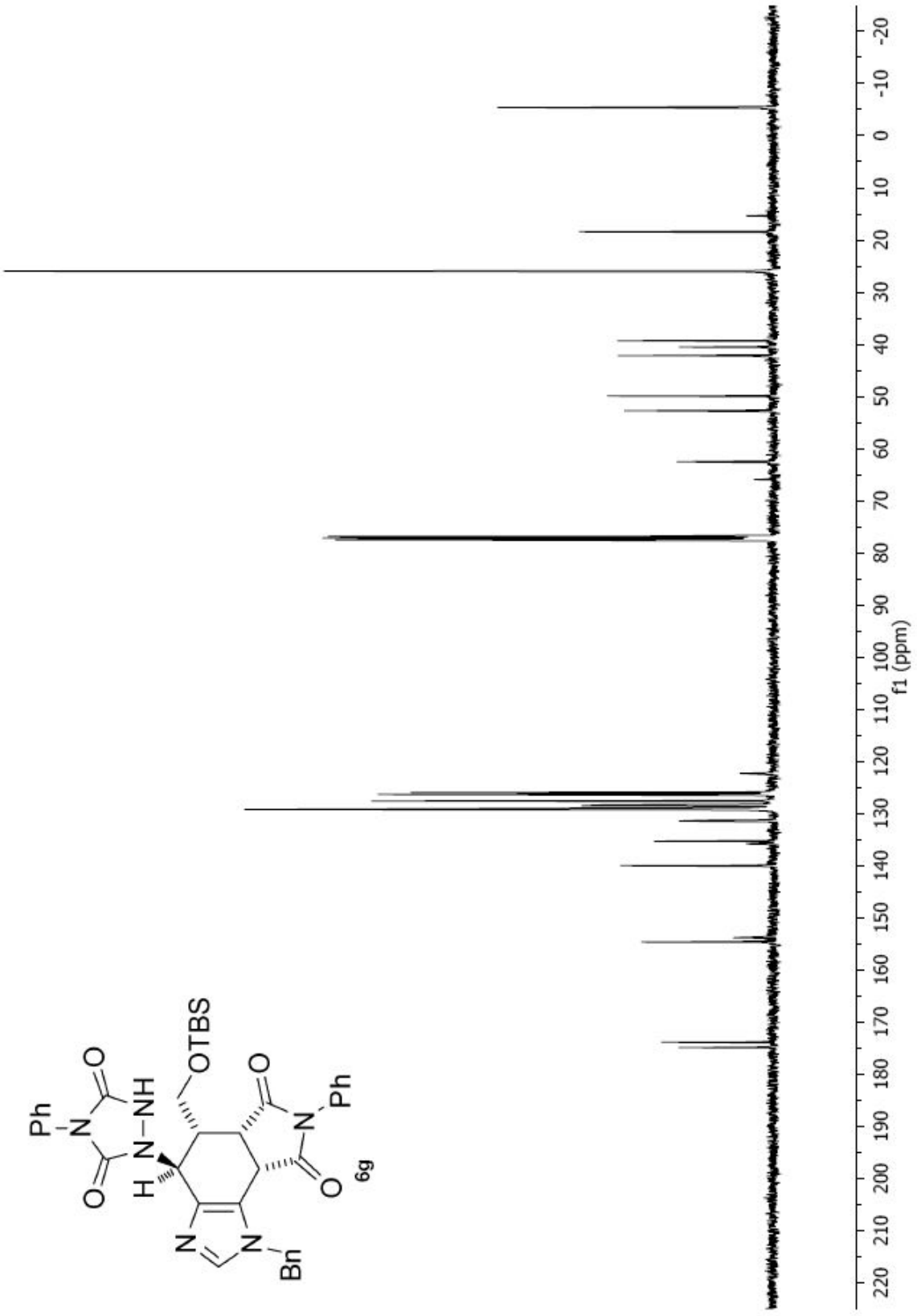

OTBS
Ph
N
O
H
N
NH
N
O
O
N
Ph
O
6g
N
N
Bn
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1
f1 (ppm)


OTBS
Ph
N
O
H
N
NH
O
N
Ph
O
6g
N
N
Bn
f1 (ppm)
-20
-10
0
10
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40
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70
80
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100
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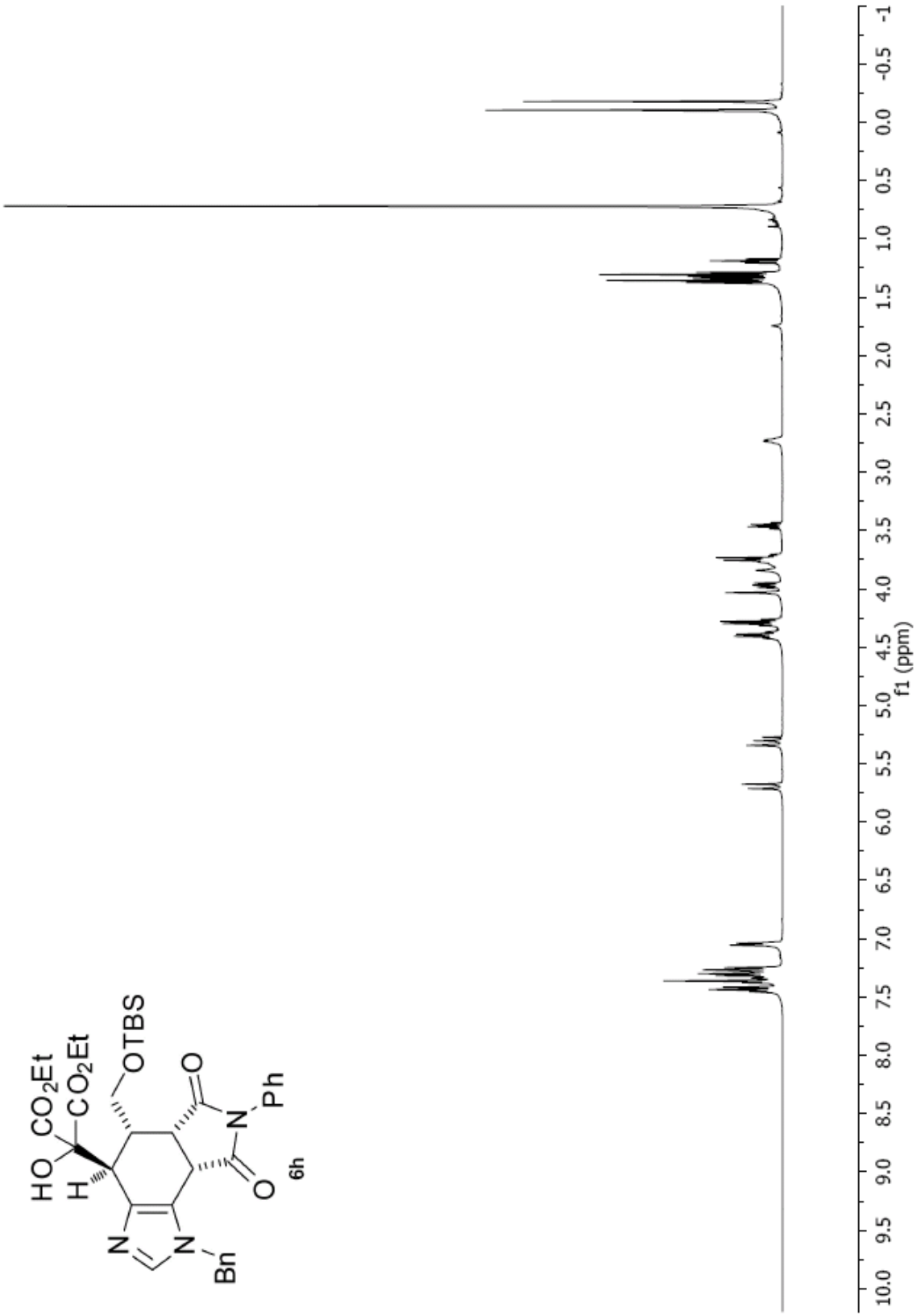
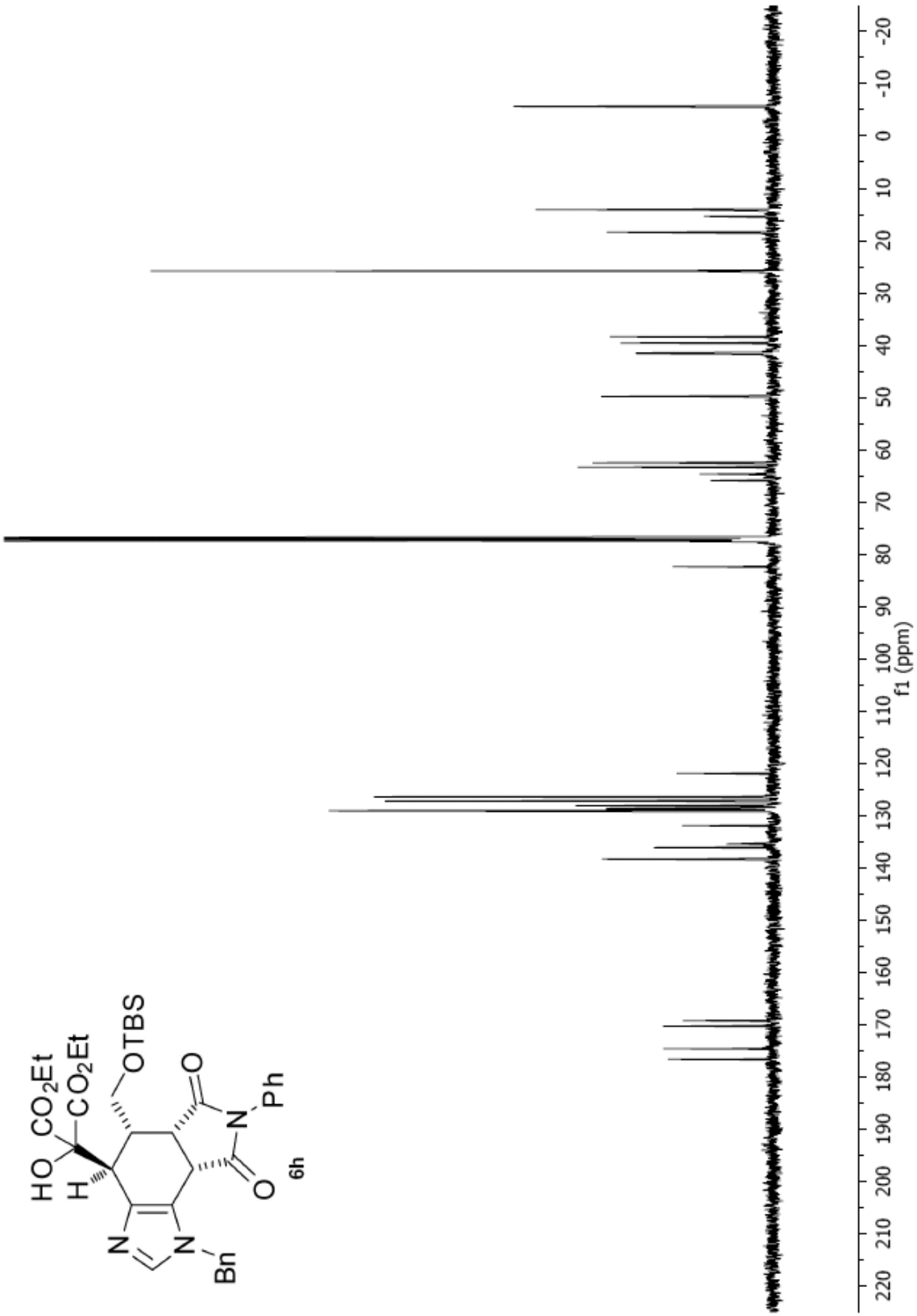
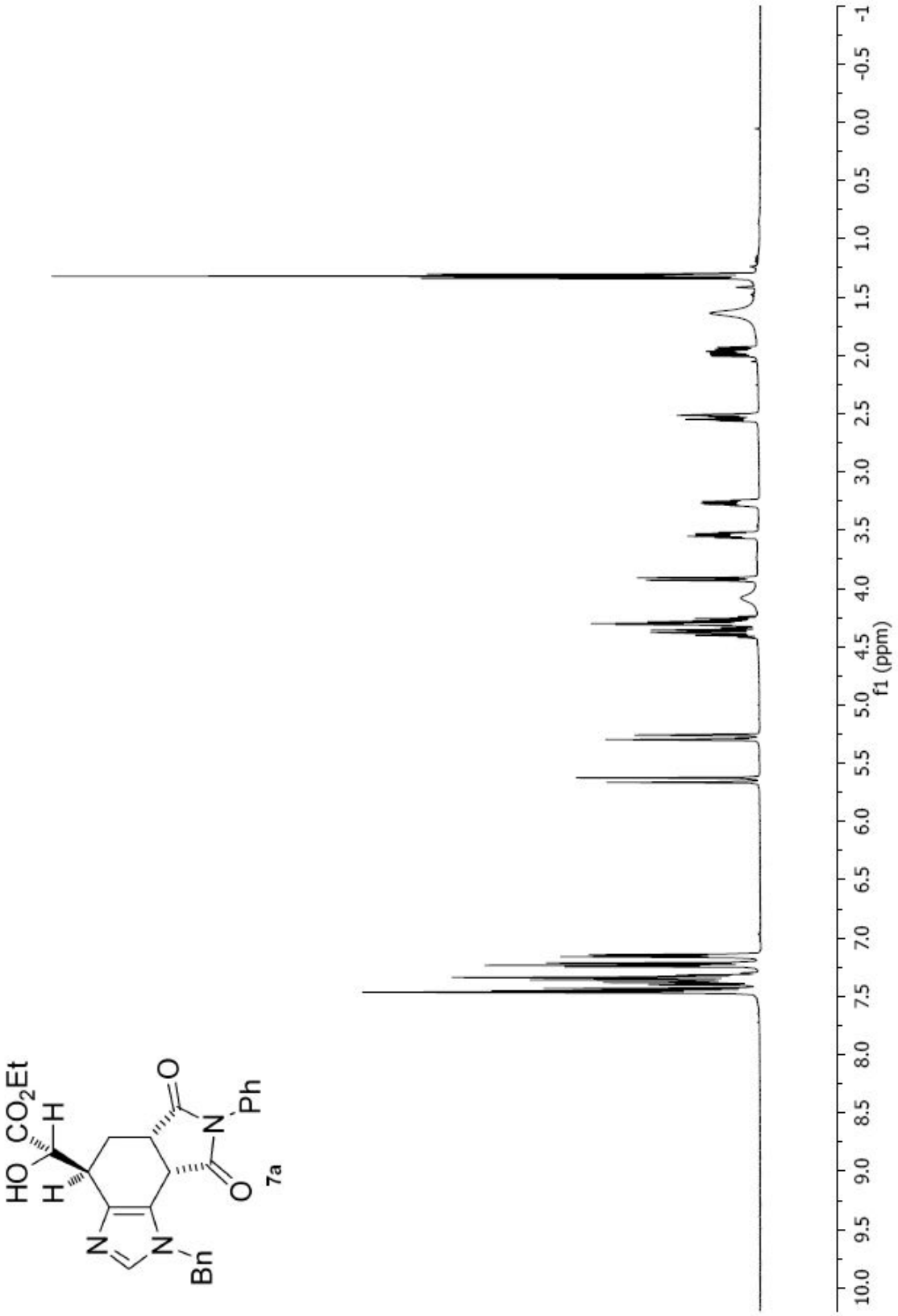
220




HO
CO2Et
CO2Et
H
OTBS
O
N
Ph
O
6h
N
N
Bn
f1 (ppm)


HO
CO2Et
H
H
O
N
Ph
O
N
N
Bn
7a
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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2.5
2.0
1.5
1.0
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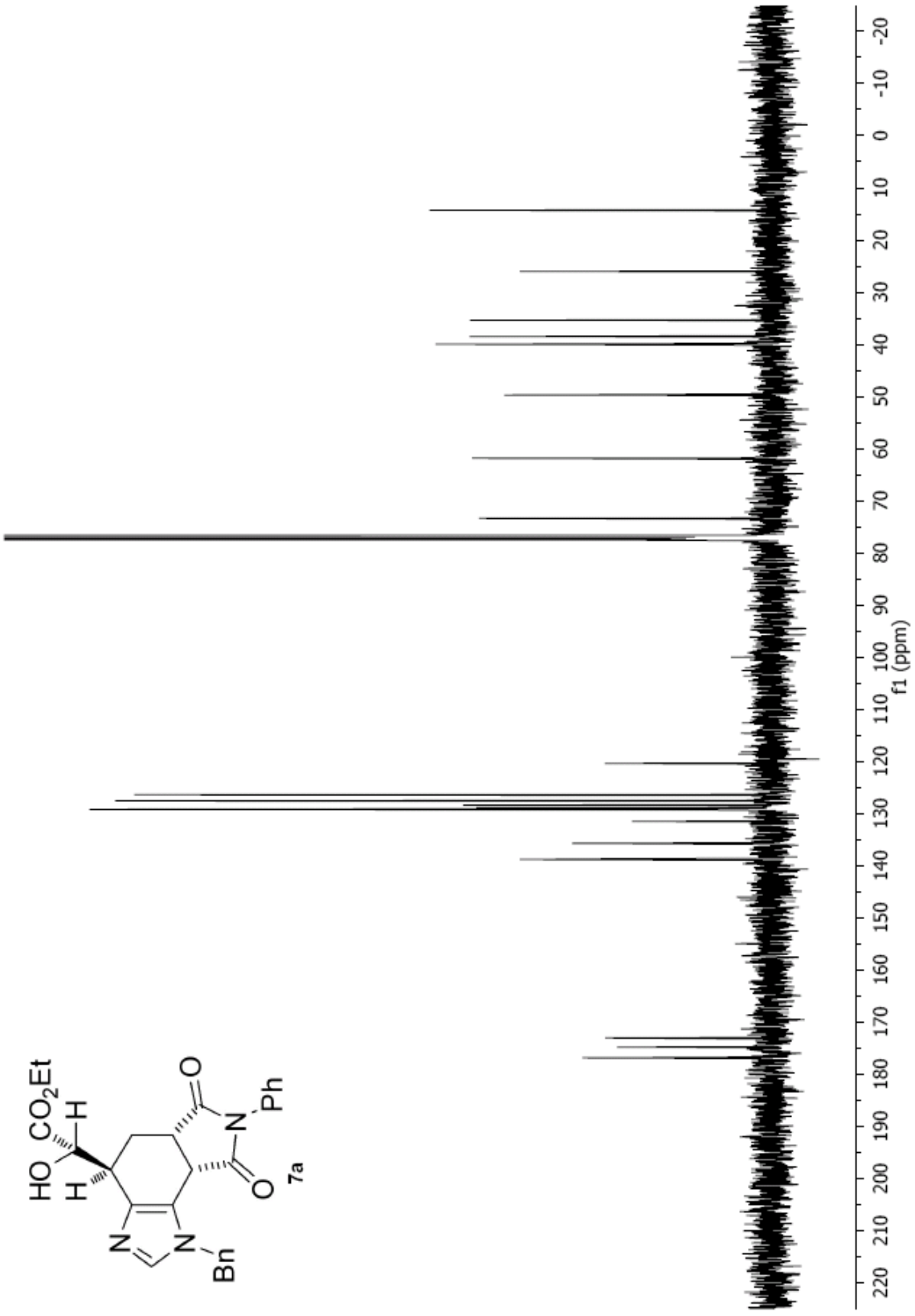
0.0
-0.5
-1
f1 (ppm)


HO
CO2Et
H
H
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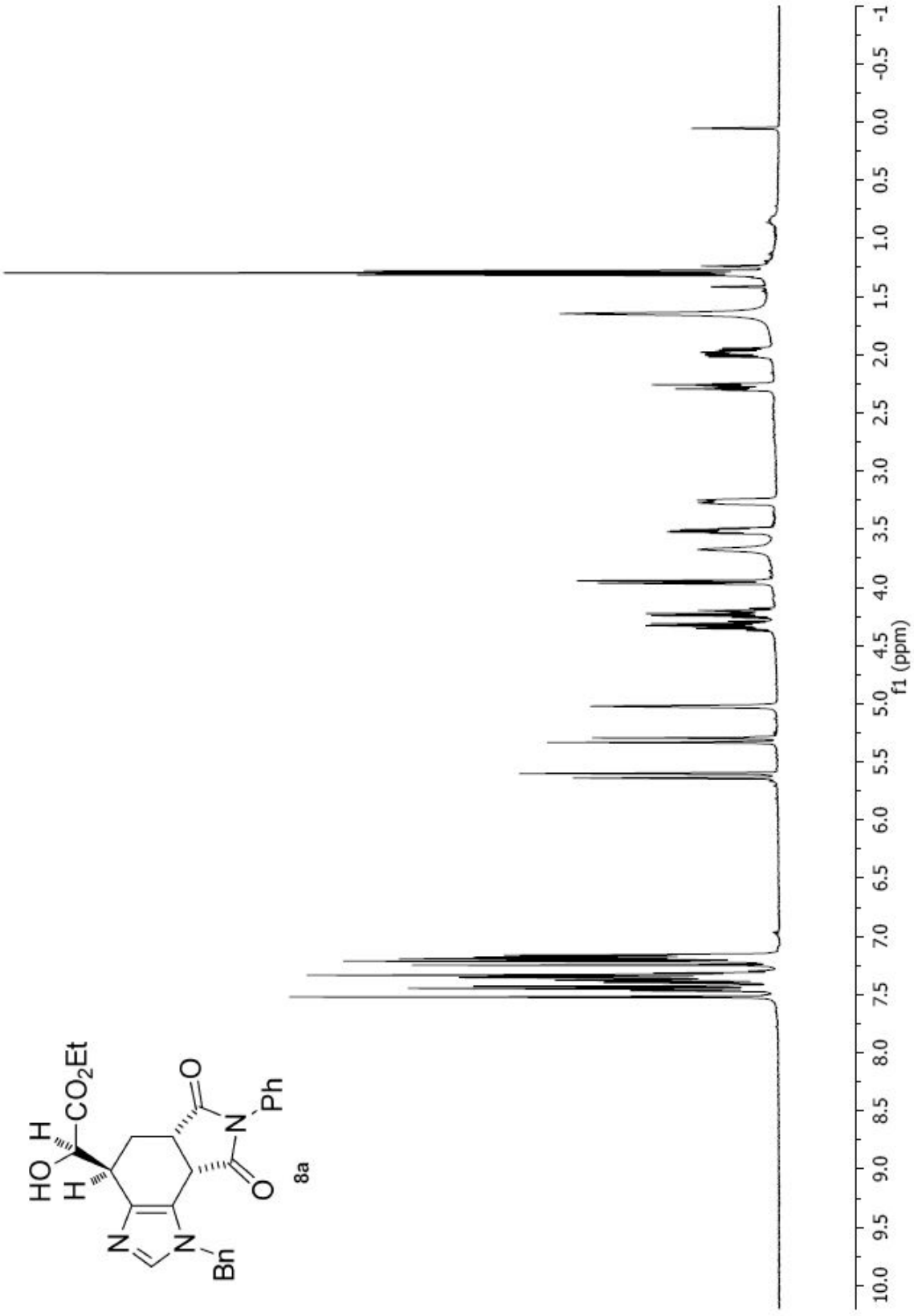
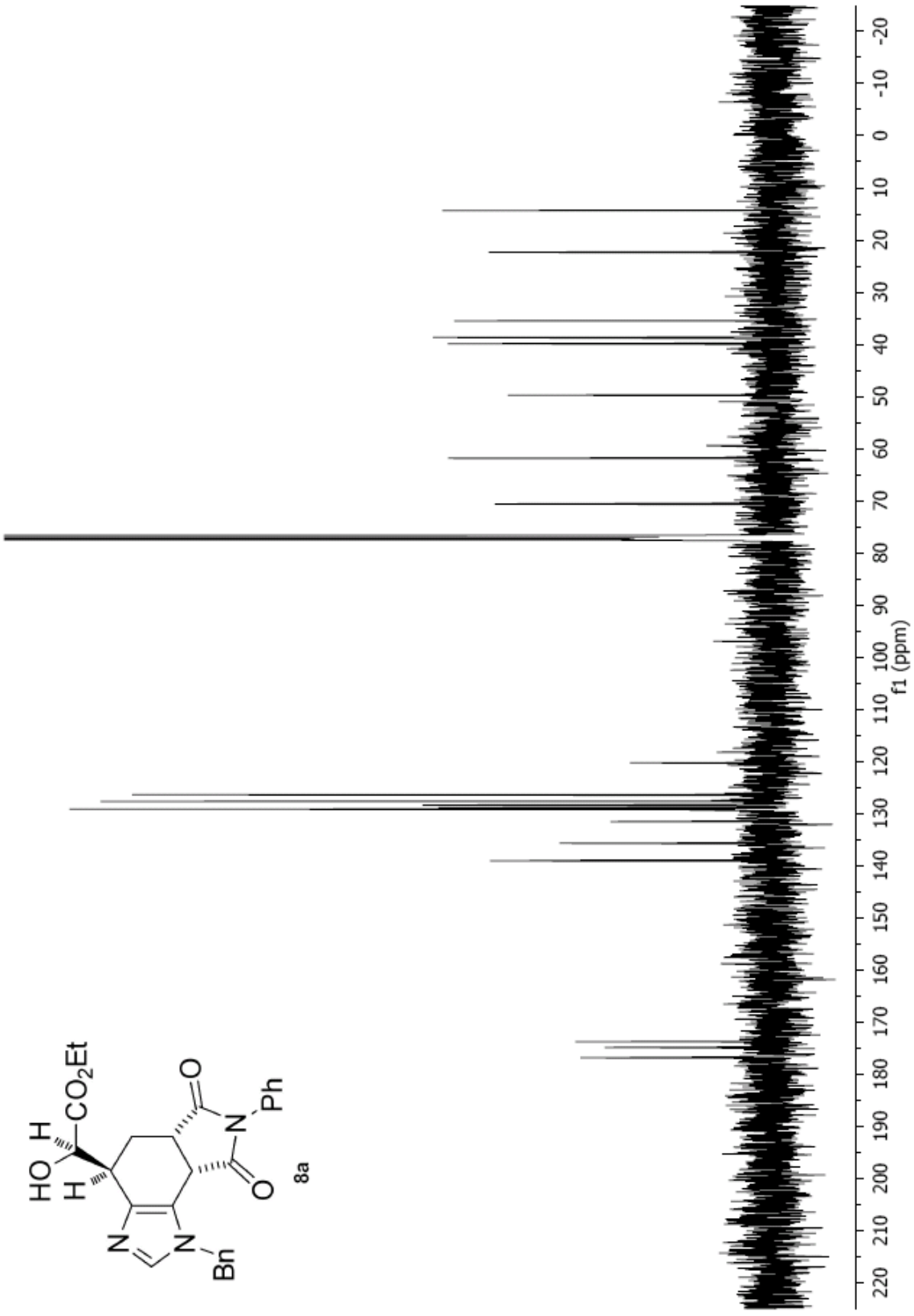
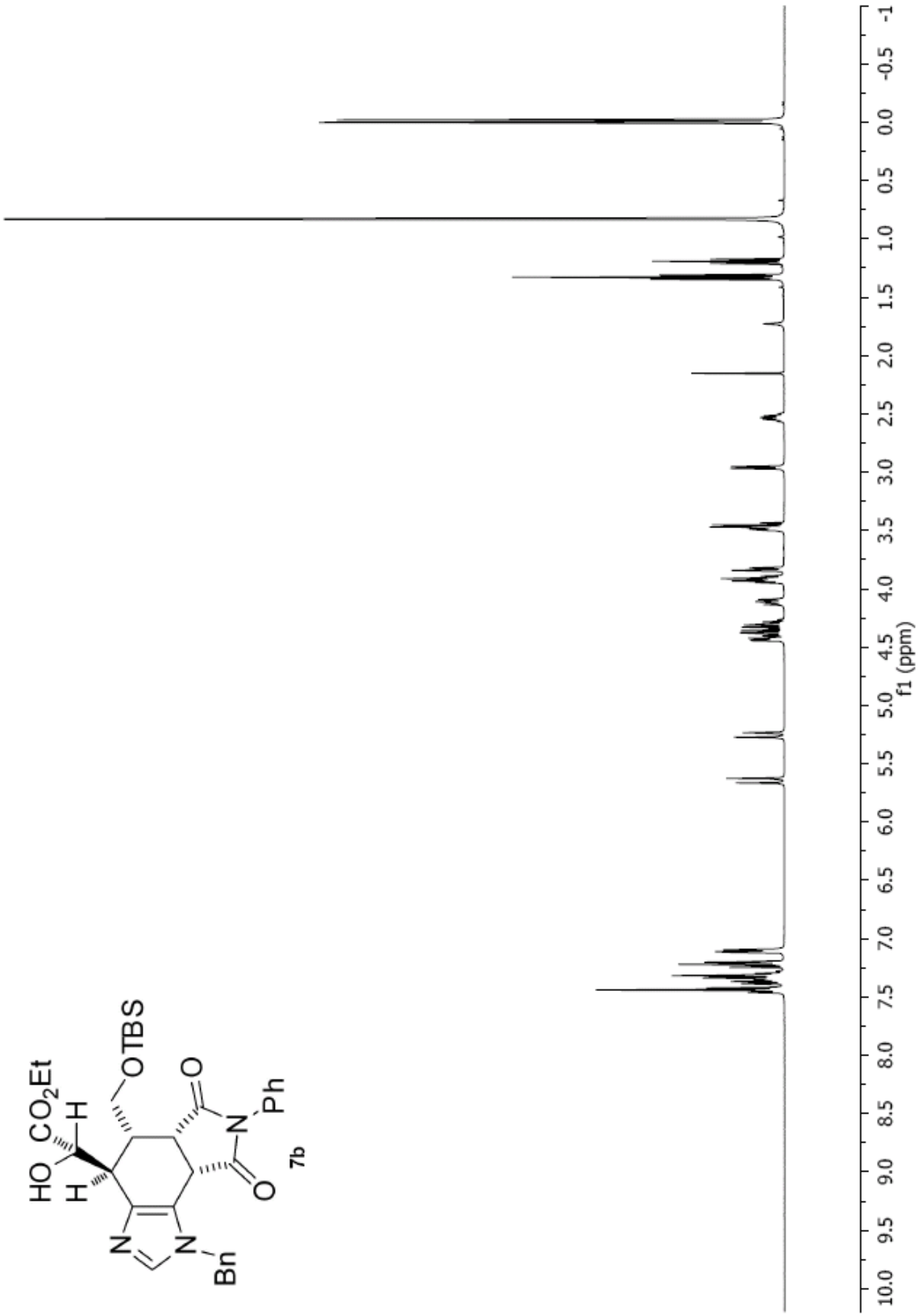
Bn
O
N
Ph
O
7a
f1 (ppm)



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H
OTBS
O
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Ph
O
N
N
Bn
7b
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9.0
8.5
8.0
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7.0
6.5
6.0
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5.0
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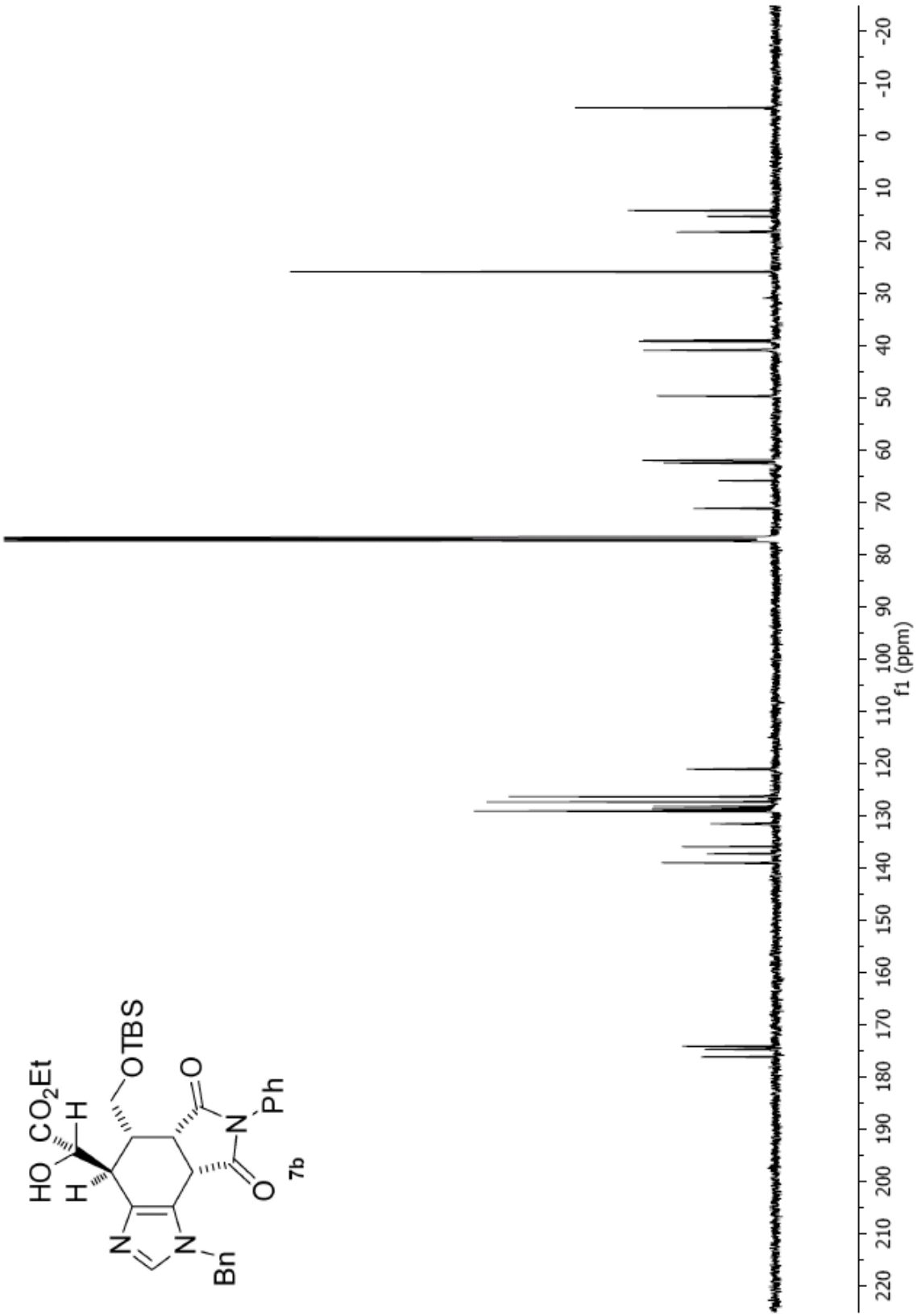
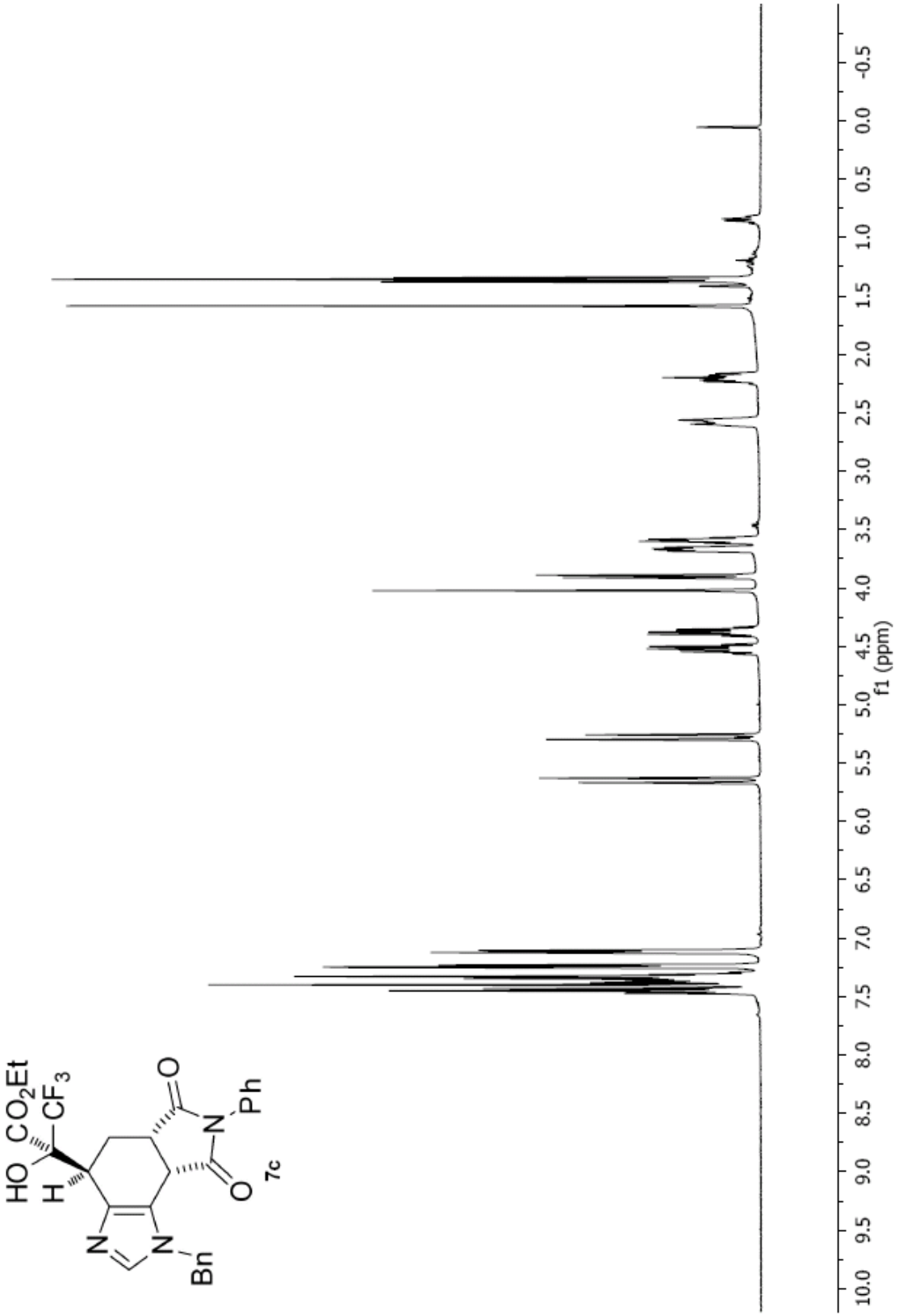
-0.5
-1
f1 (ppm)


HO
CO2Et
H
H
OTBS
O
N
Ph
O
N
N
Bn
7b
f1 (ppm)


HO
CO2Et
CF3
H
O
N
Ph
O
N
N
Bn
7c
10.0
9.5
9.0
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8.0
7.5
7.0
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6.0
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5.0
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3.0
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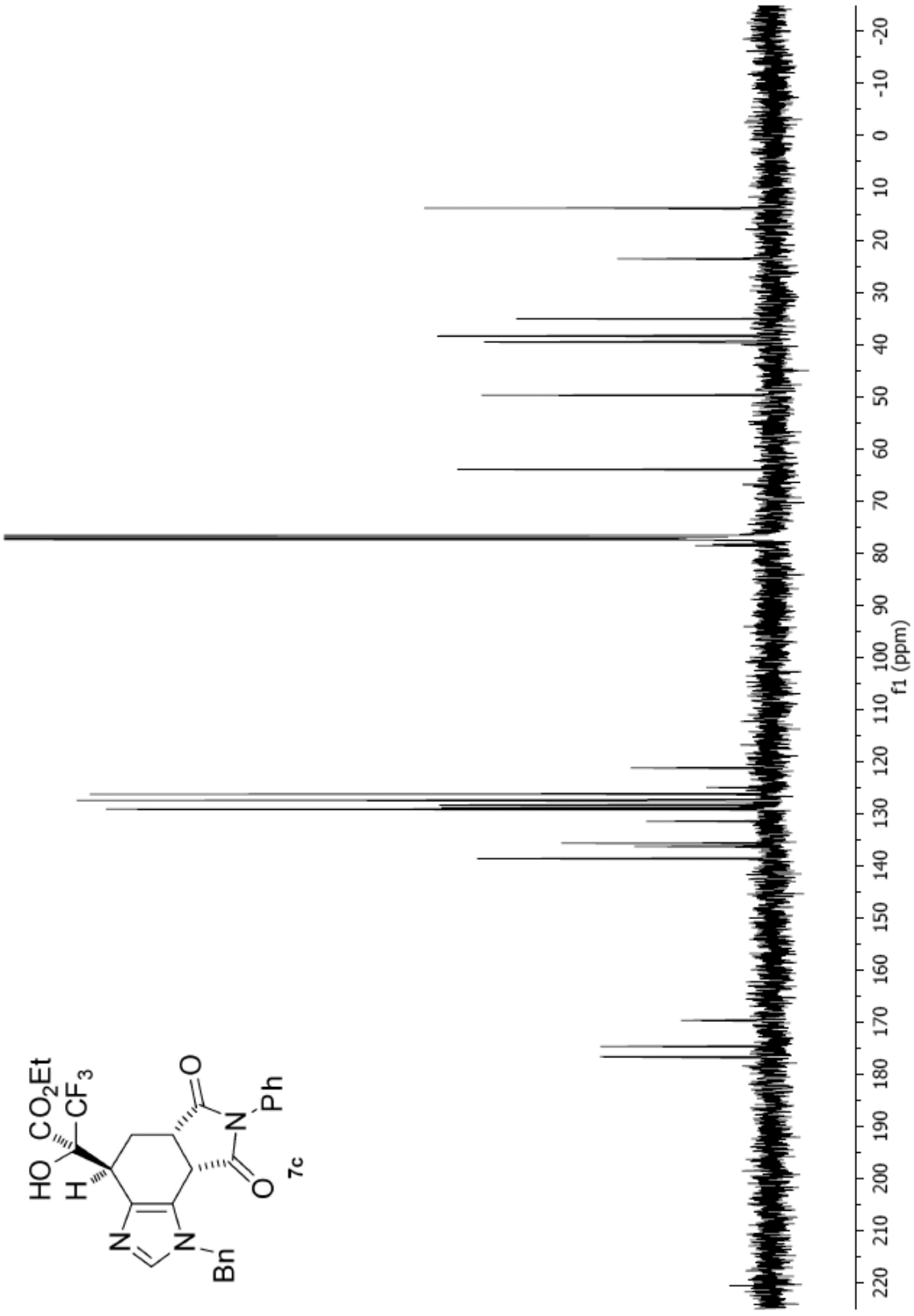
0.5
0.0
-0.5
f1 (ppm)


HO
CO2Et
CF3
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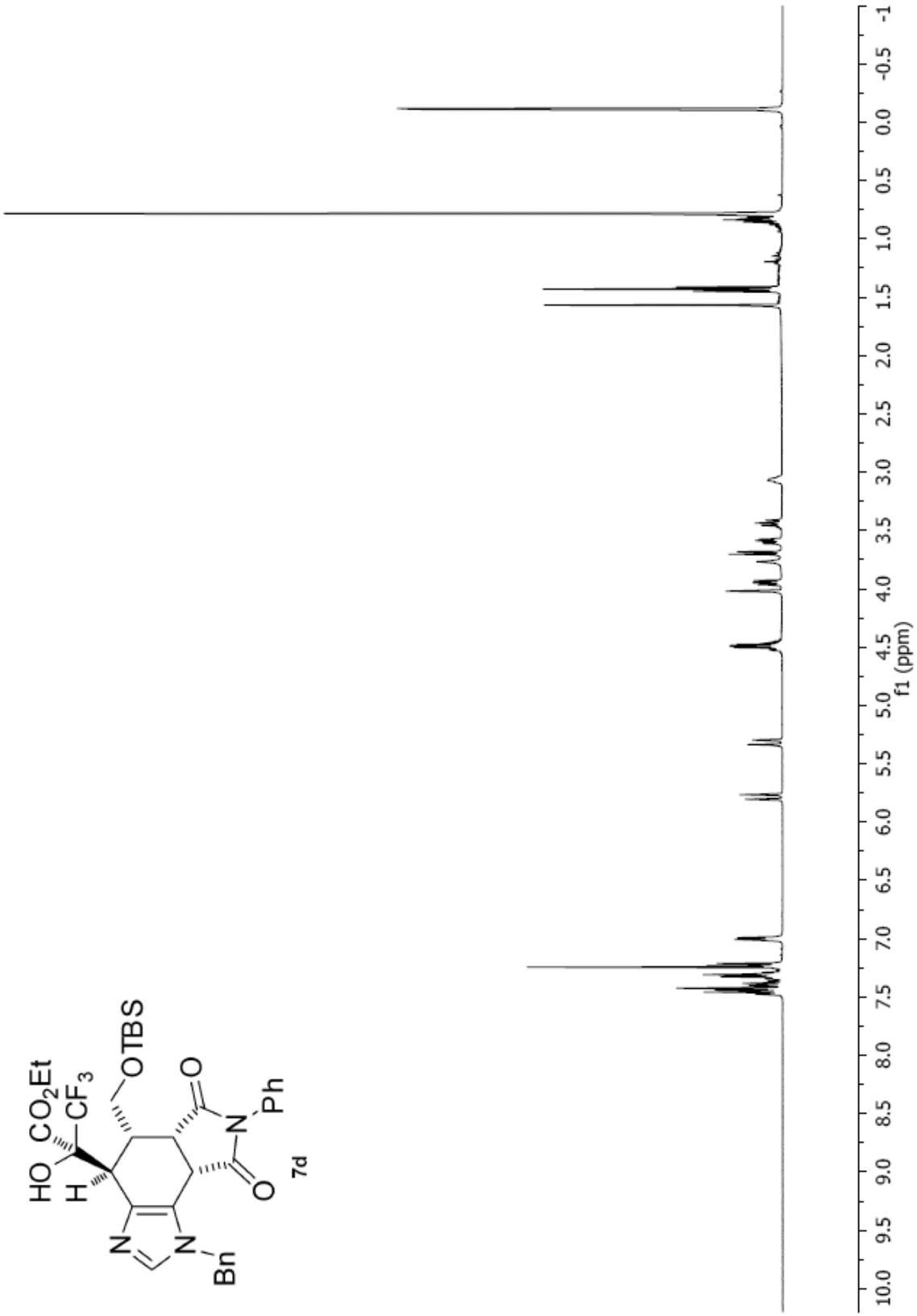
Bn
O
N
Ph
O
7c
f1 (ppm)


HO
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CF3
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OTBS
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Ph
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N
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Bn
7d
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0.5
0.0
-0.5
-1
f1 (ppm)