Structure-Reactivity Relationships of Zwitterionic 1,3-Diaza-Claisen Rearrangements

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Supporting Information

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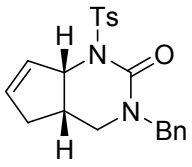
^{1}H and ^{13}C NMR-Spectra



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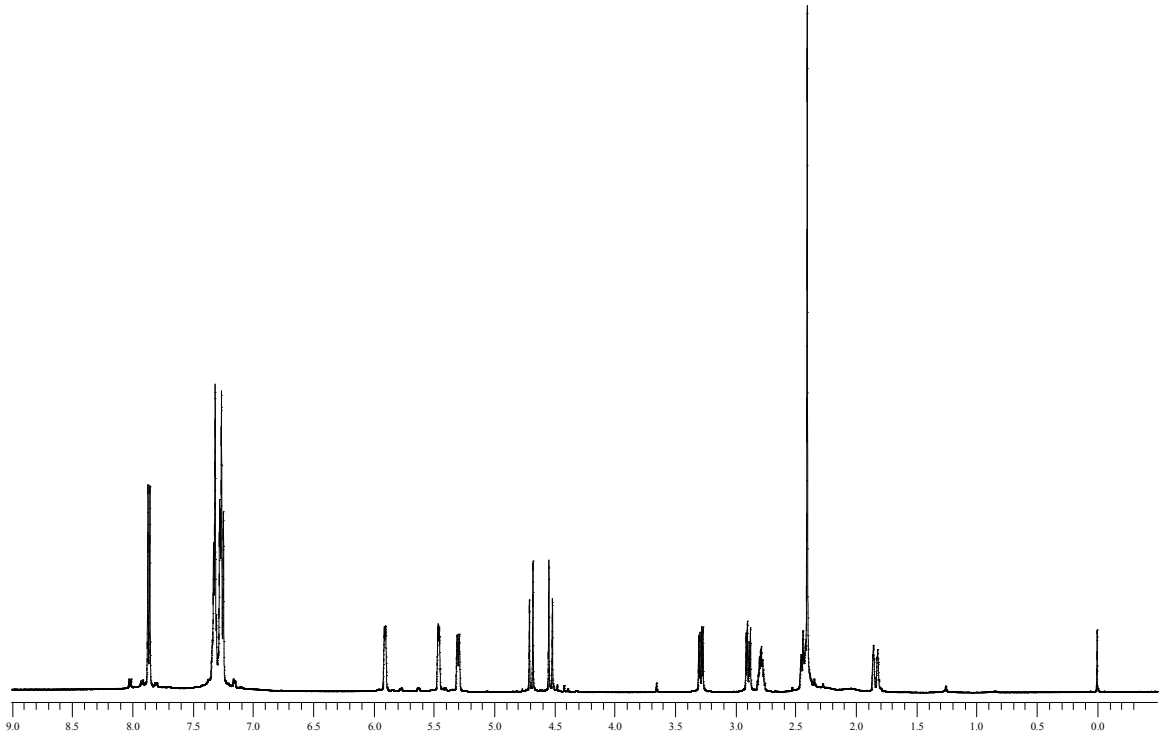

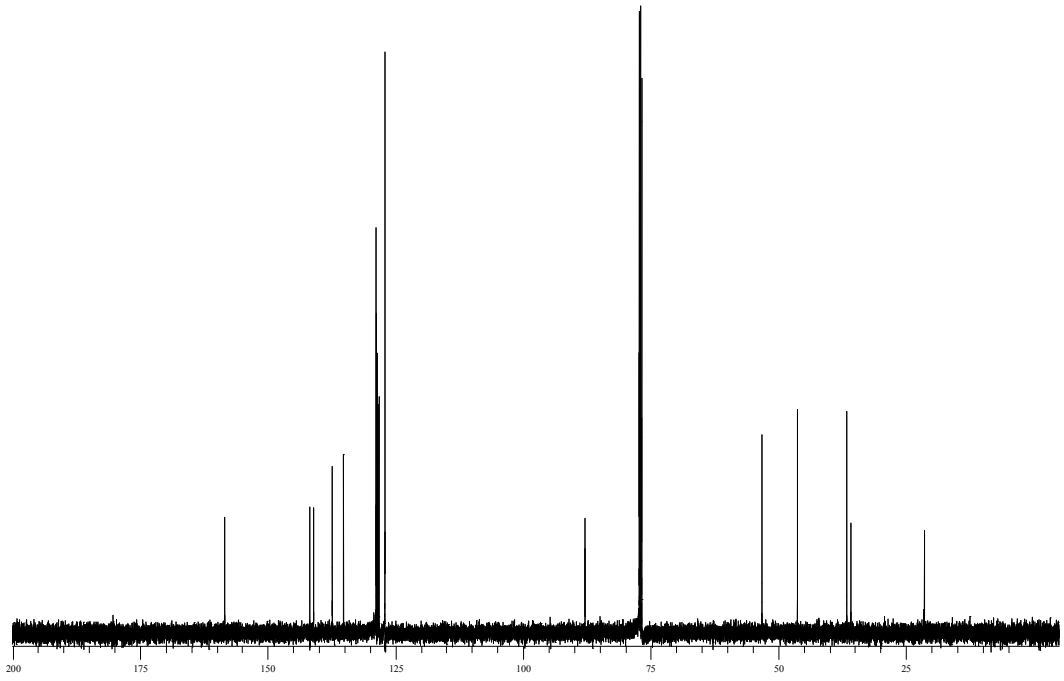

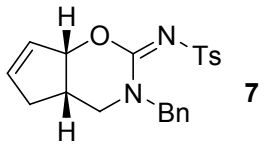
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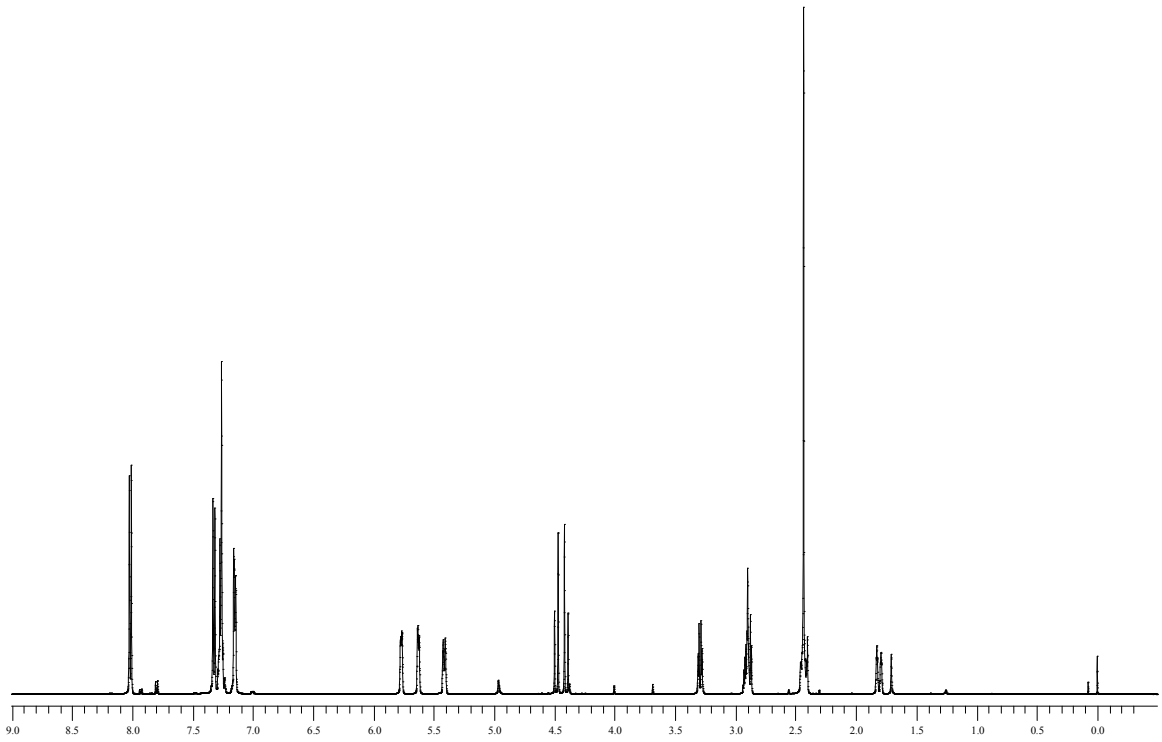
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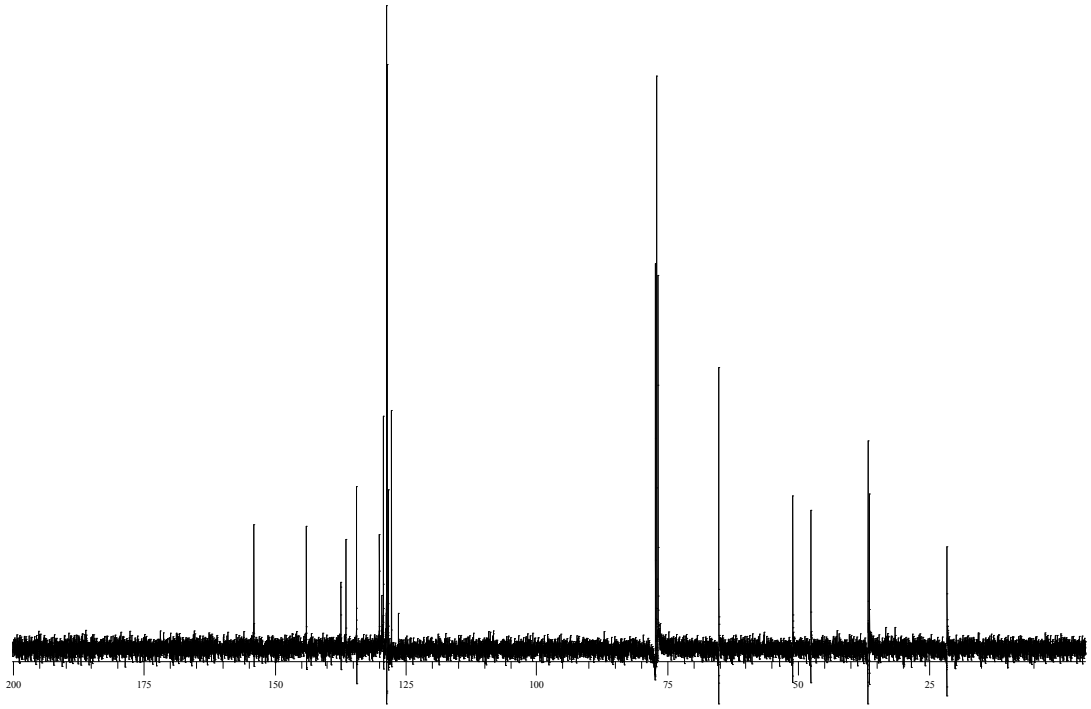
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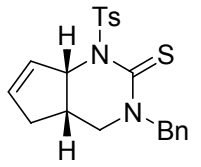
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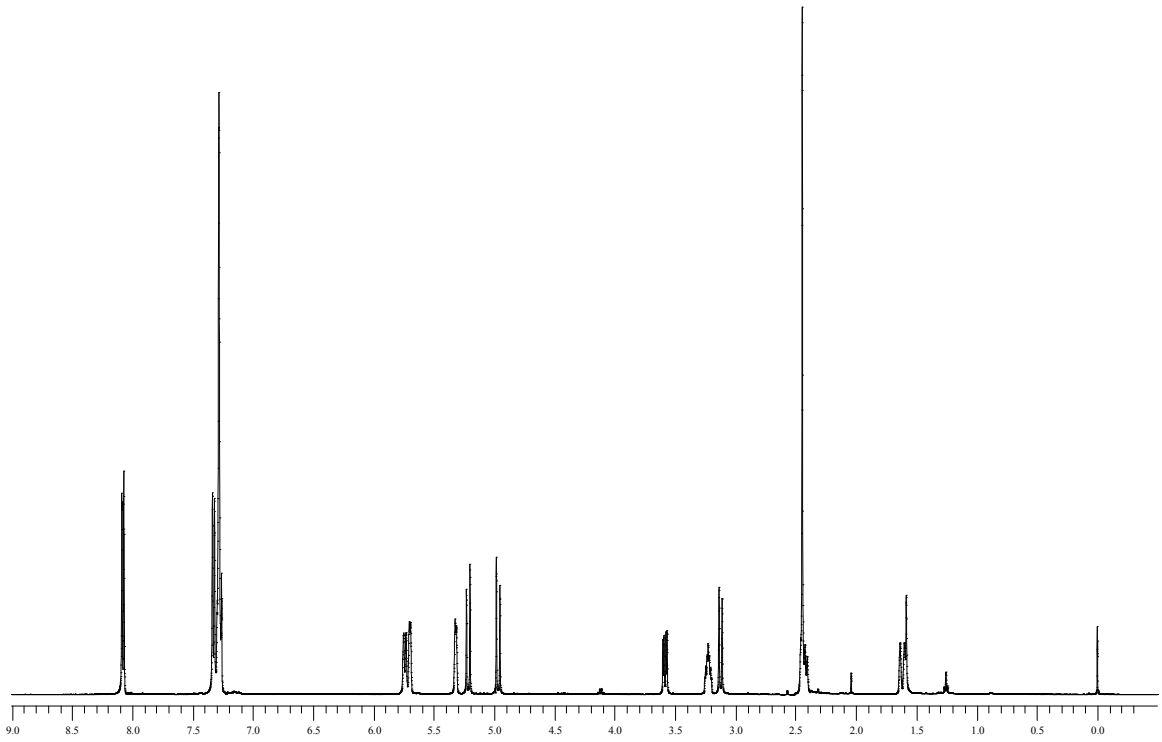
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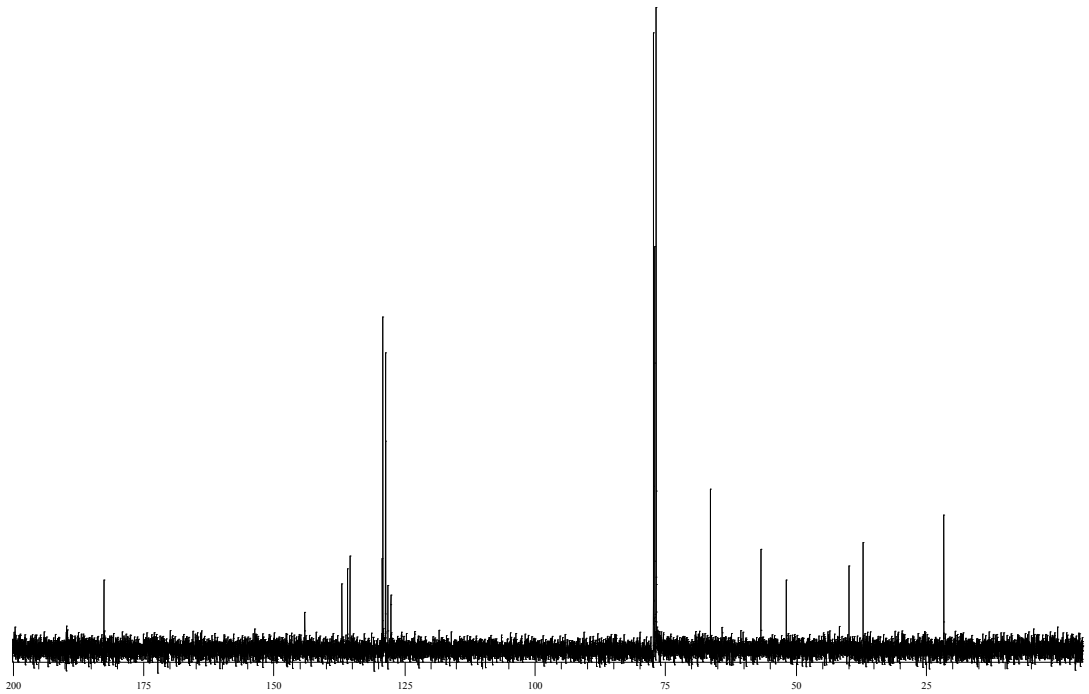
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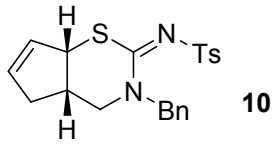
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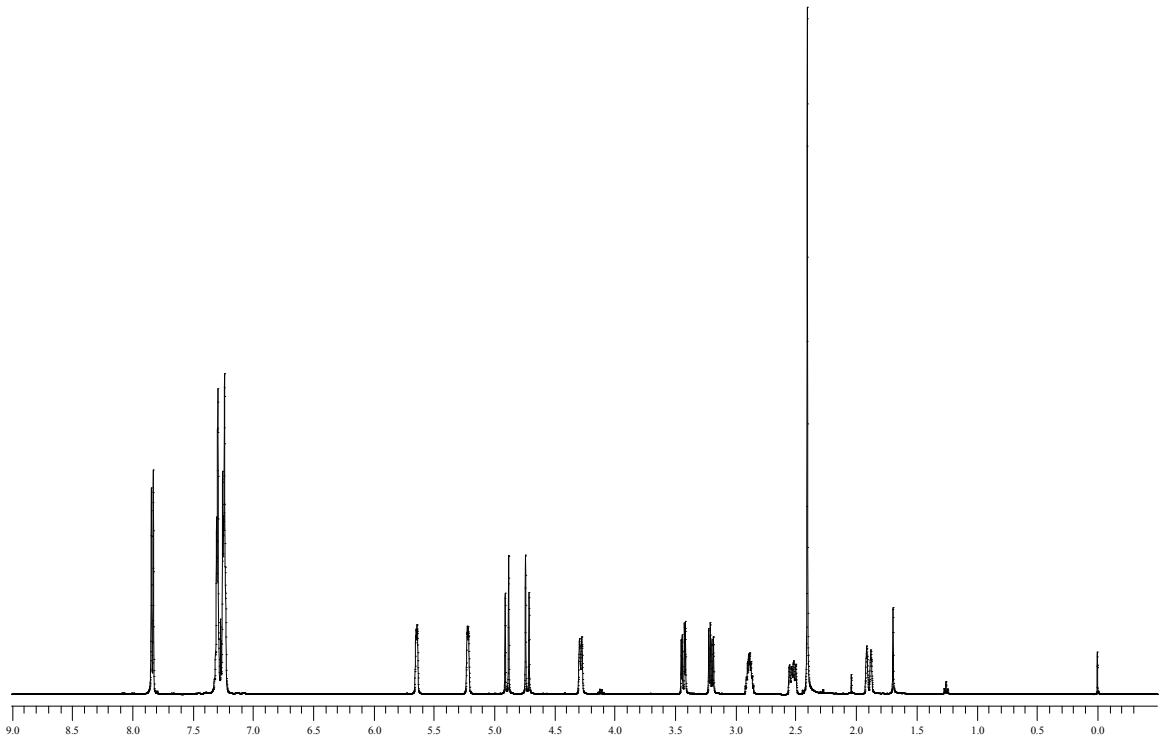
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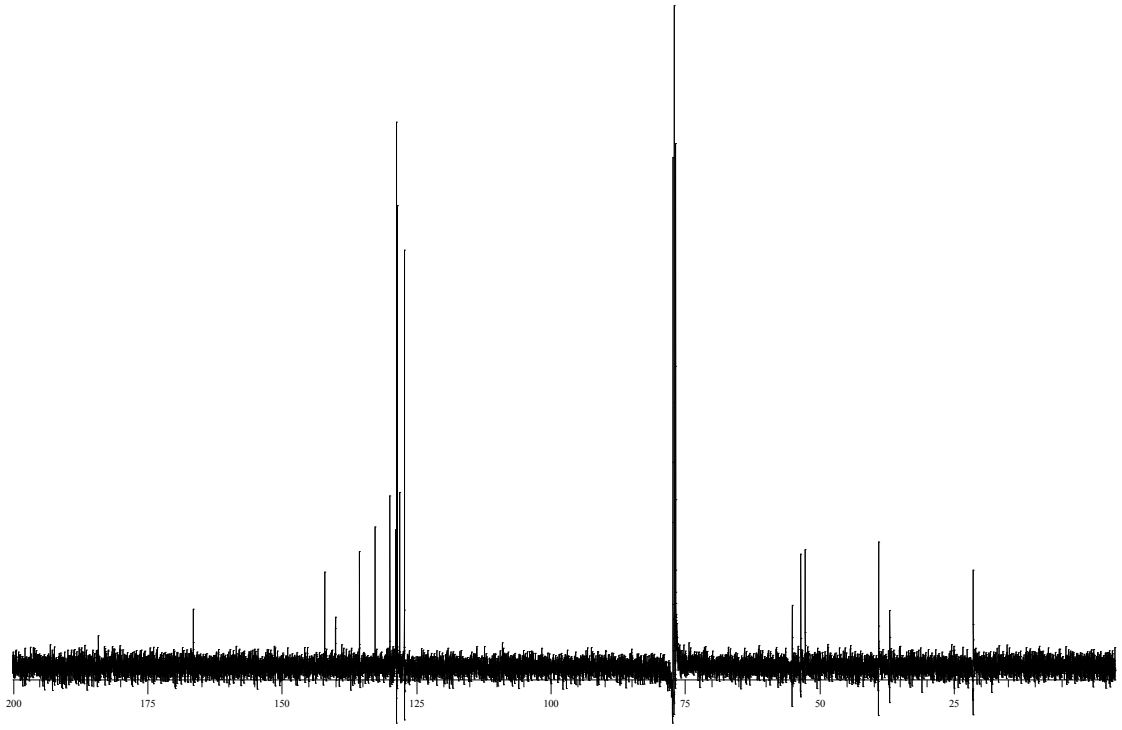
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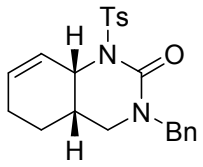
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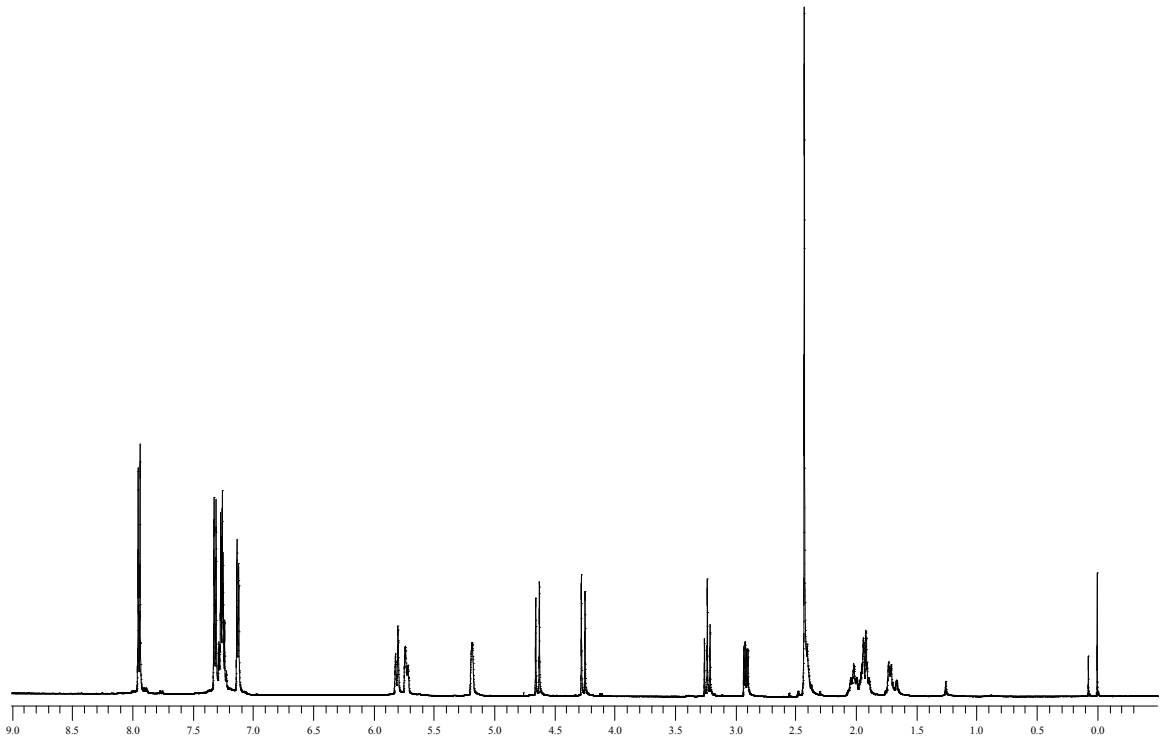
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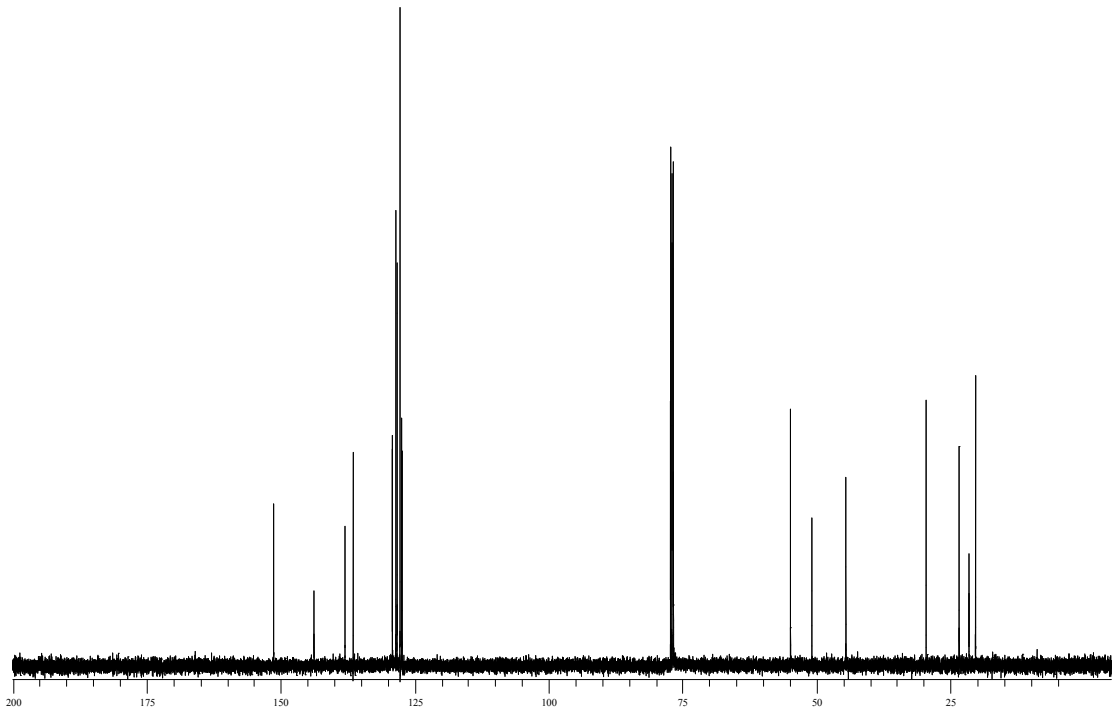
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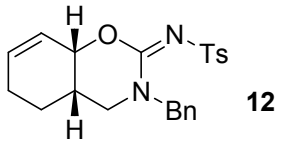

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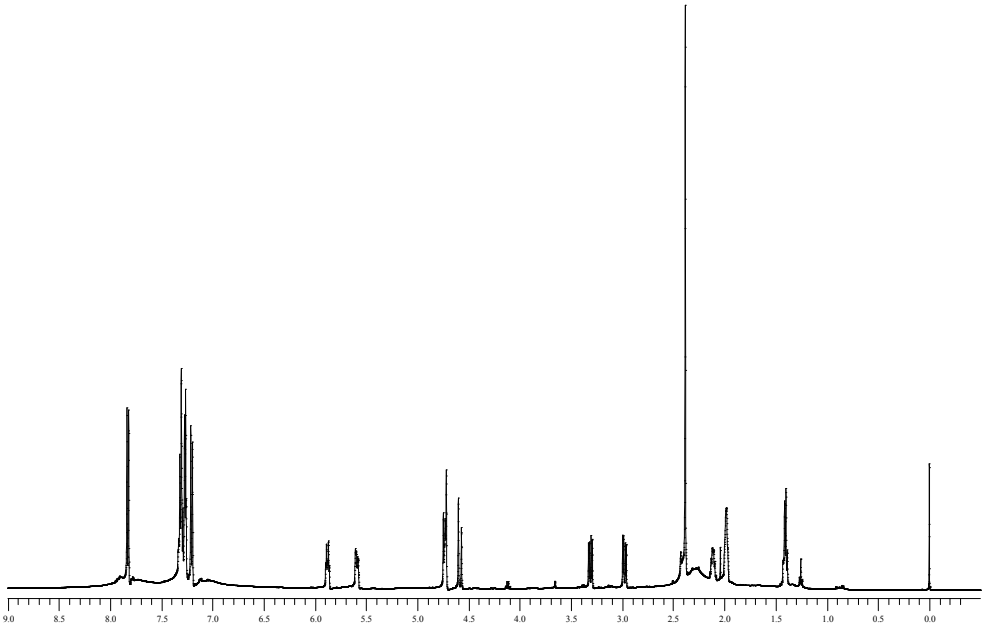

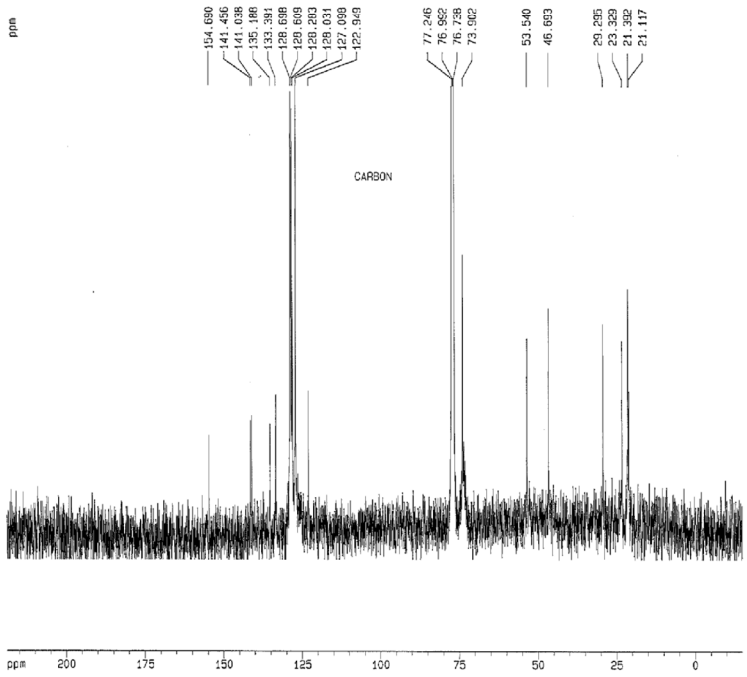


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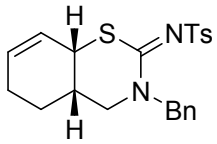
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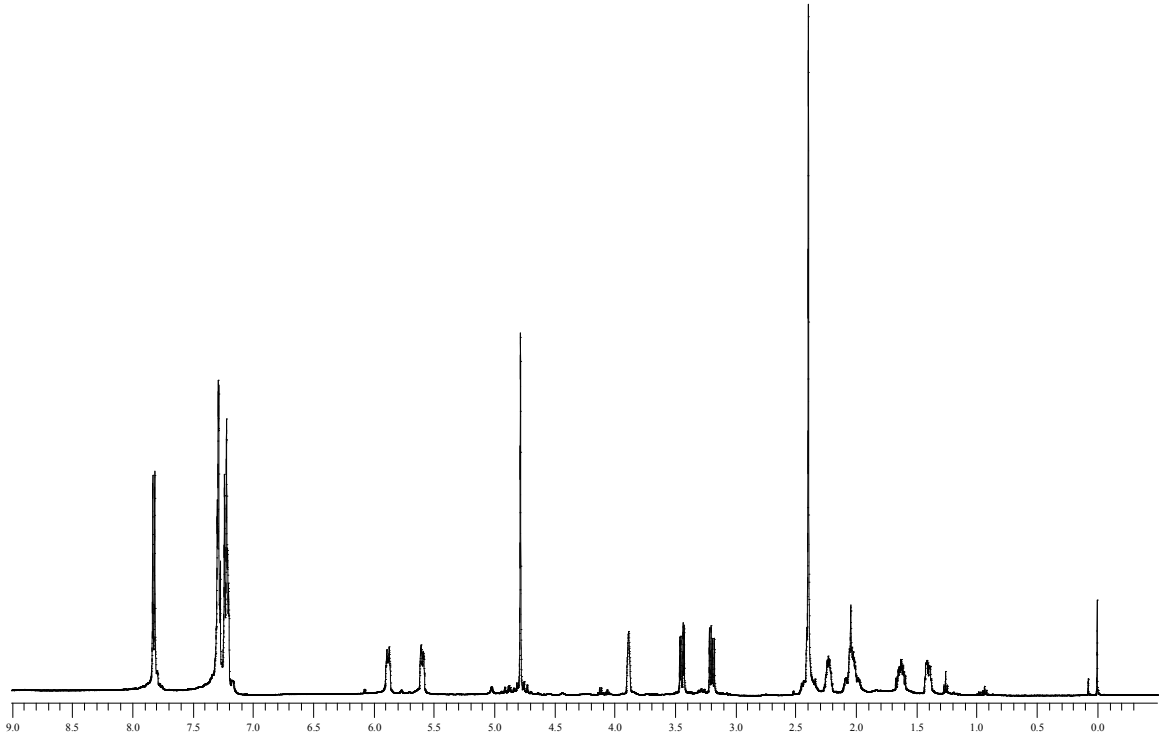
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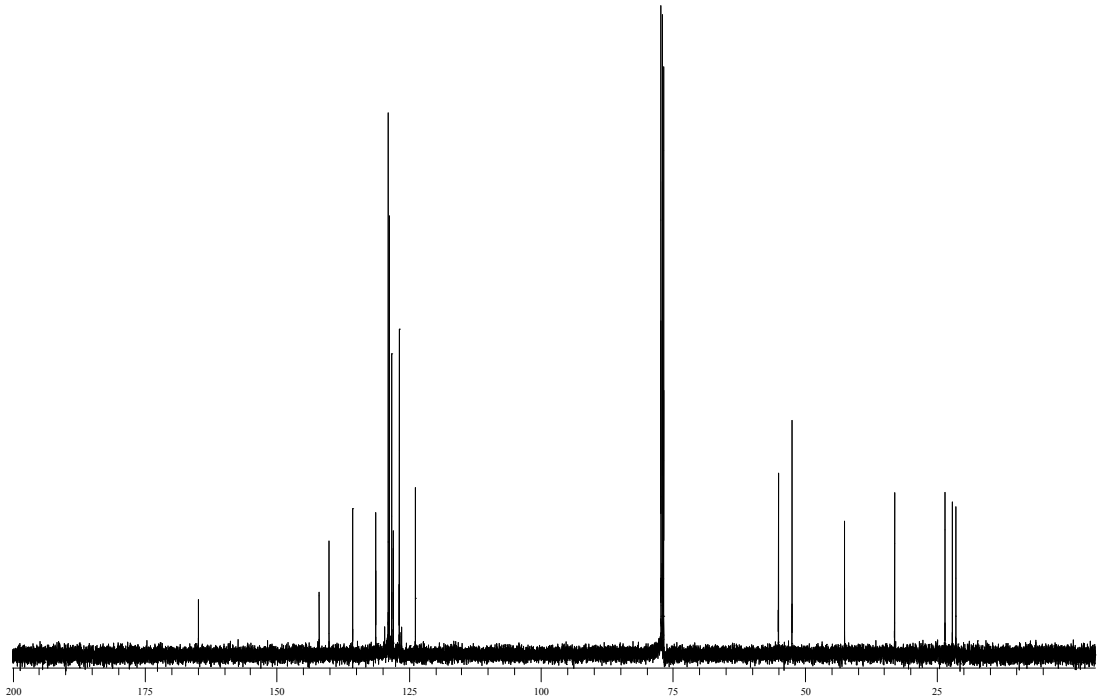
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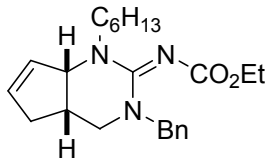
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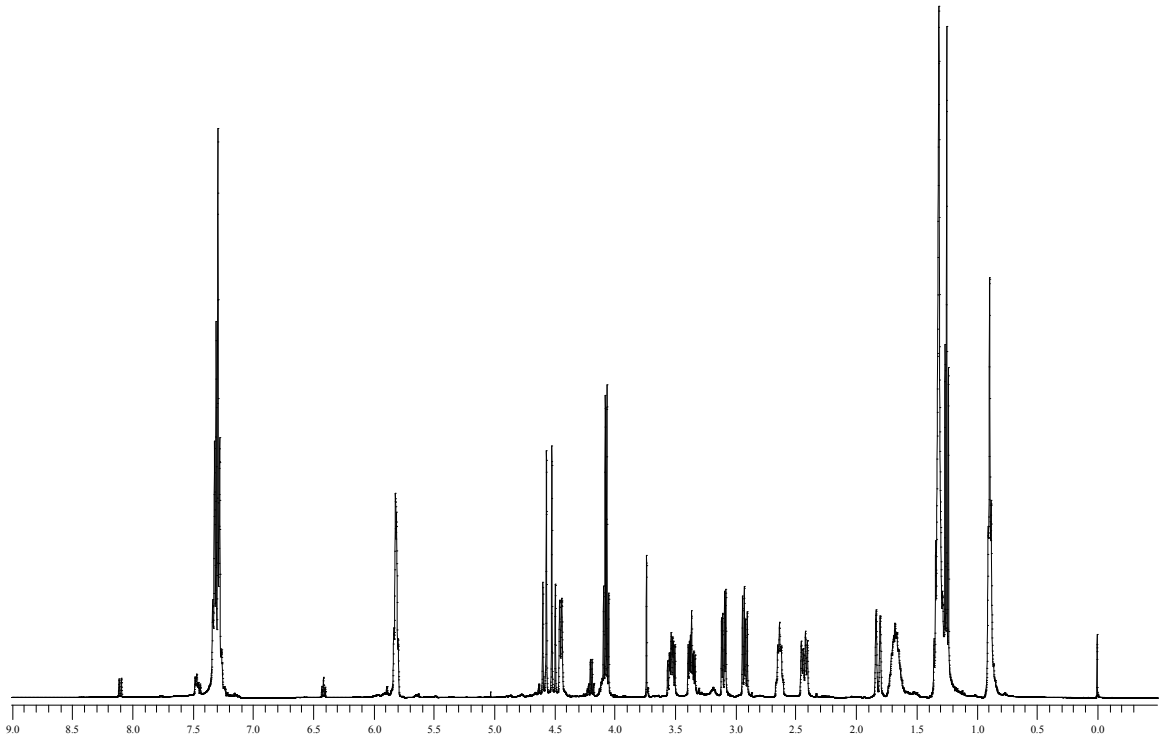
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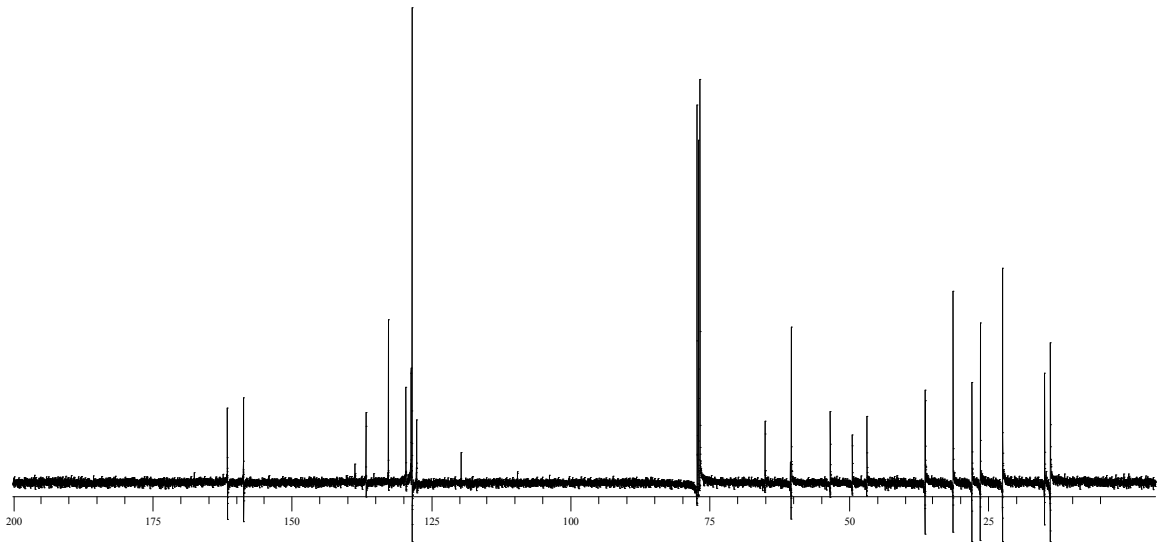
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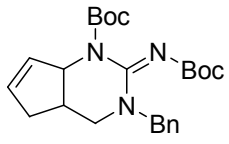
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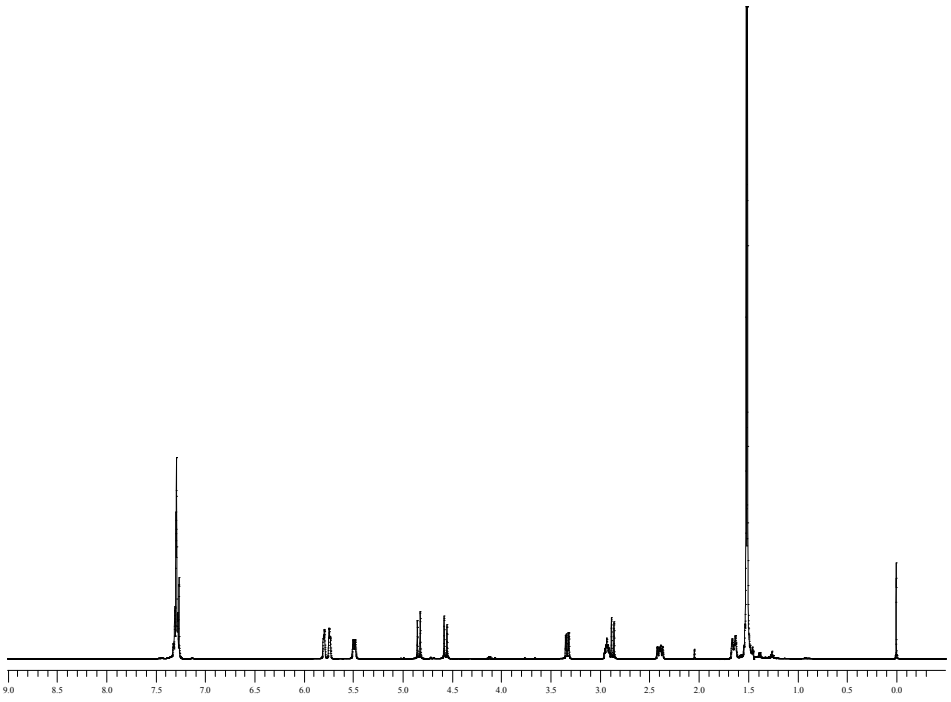
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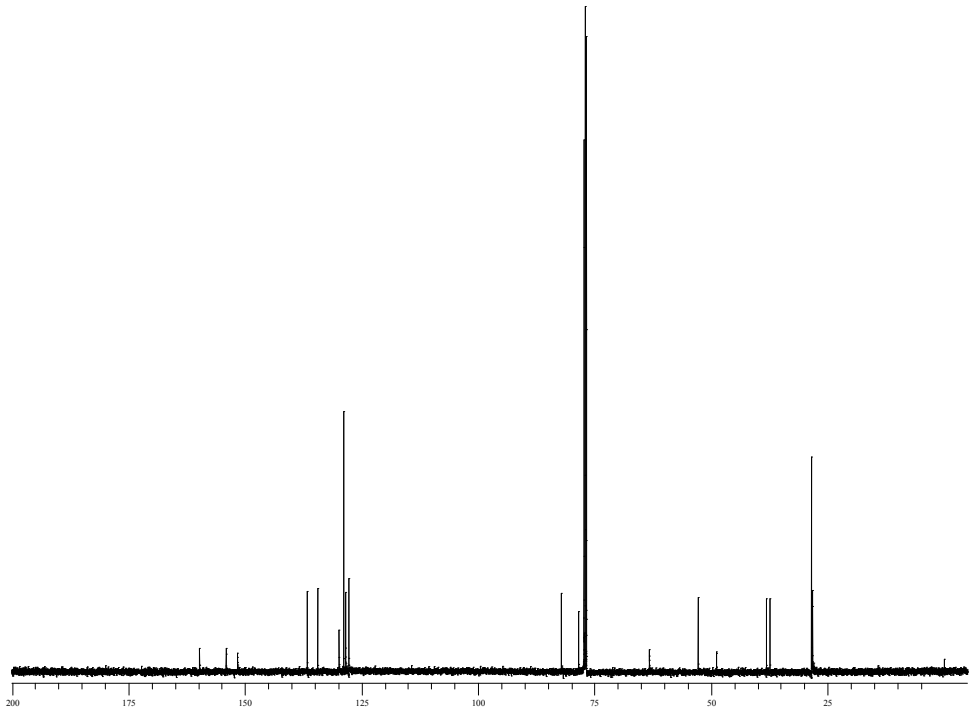
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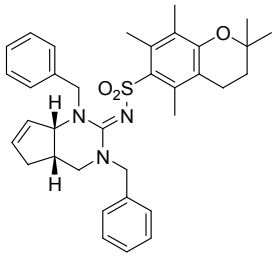

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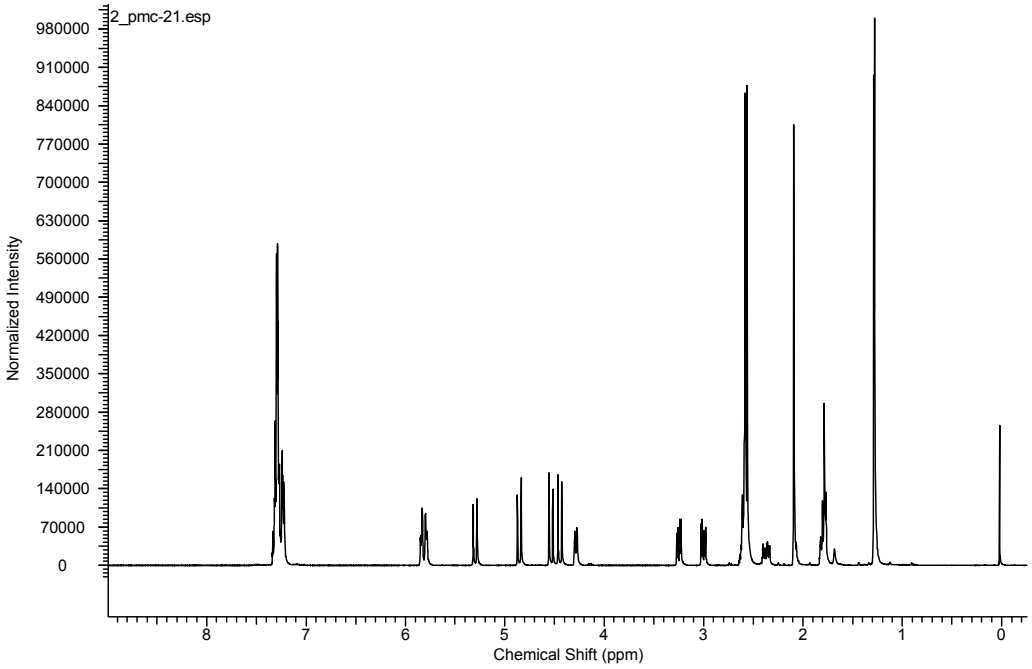
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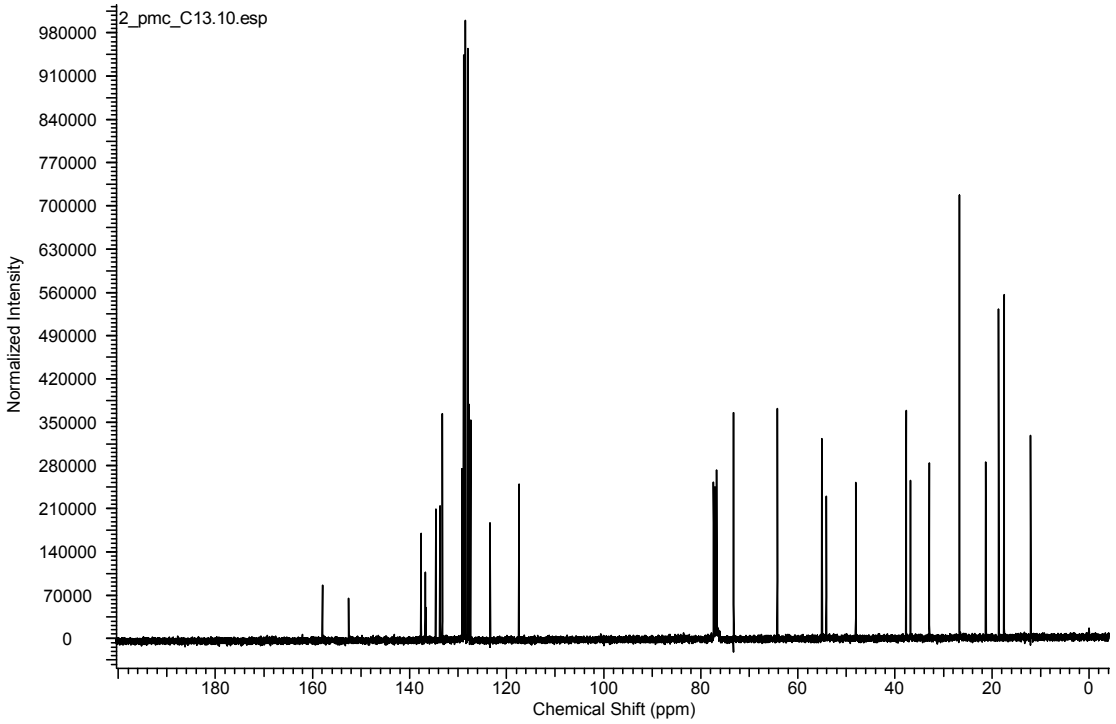
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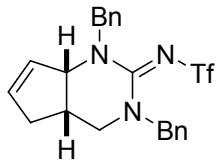
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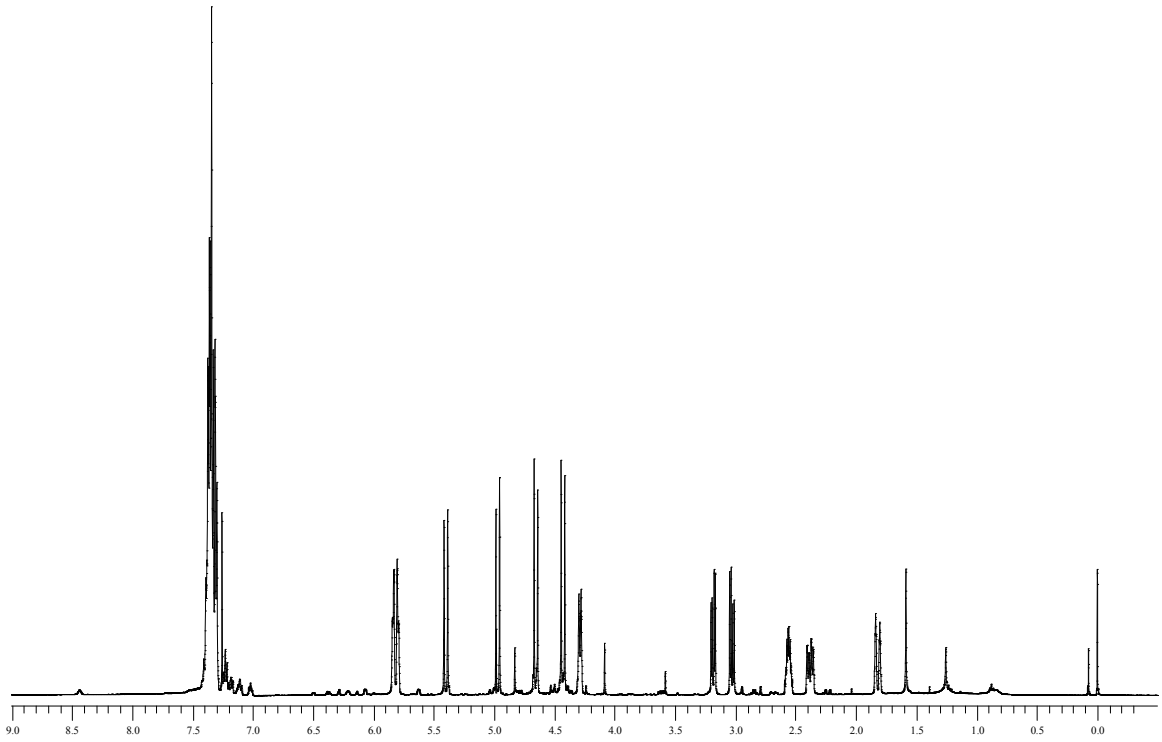
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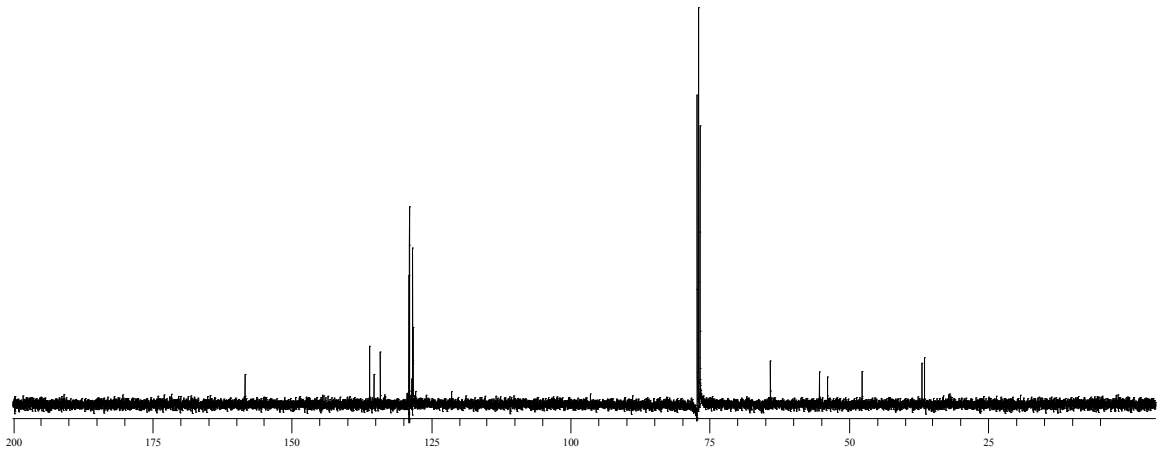
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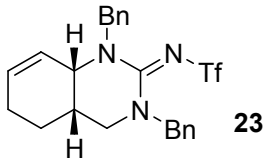
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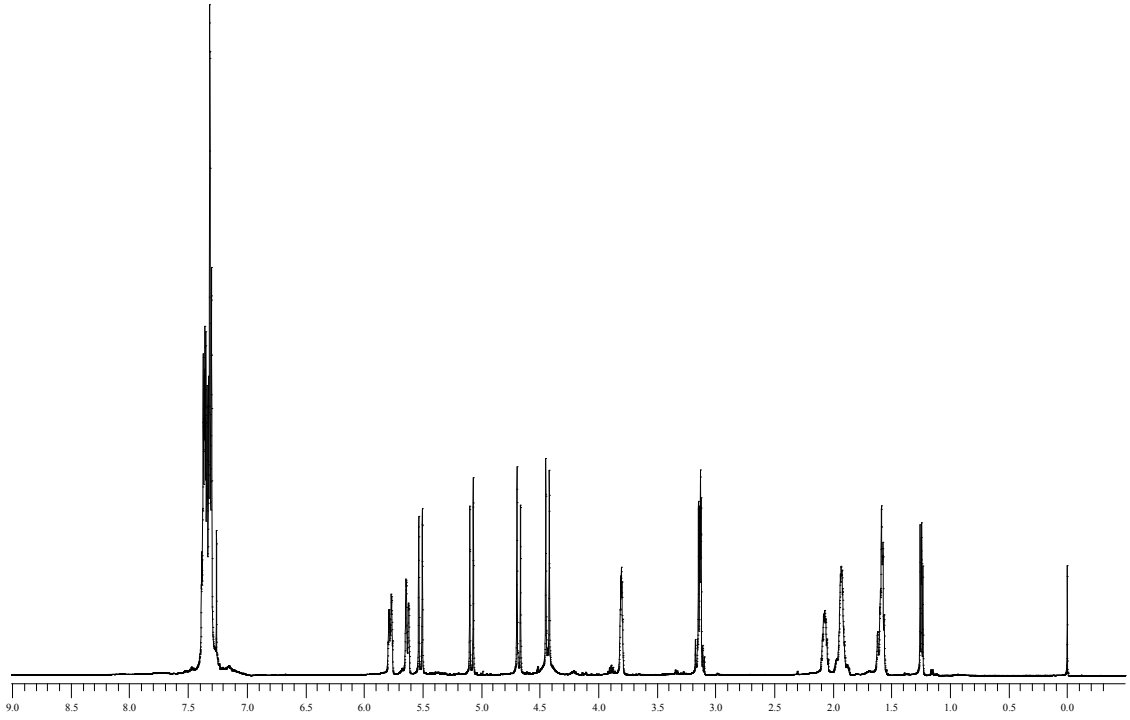
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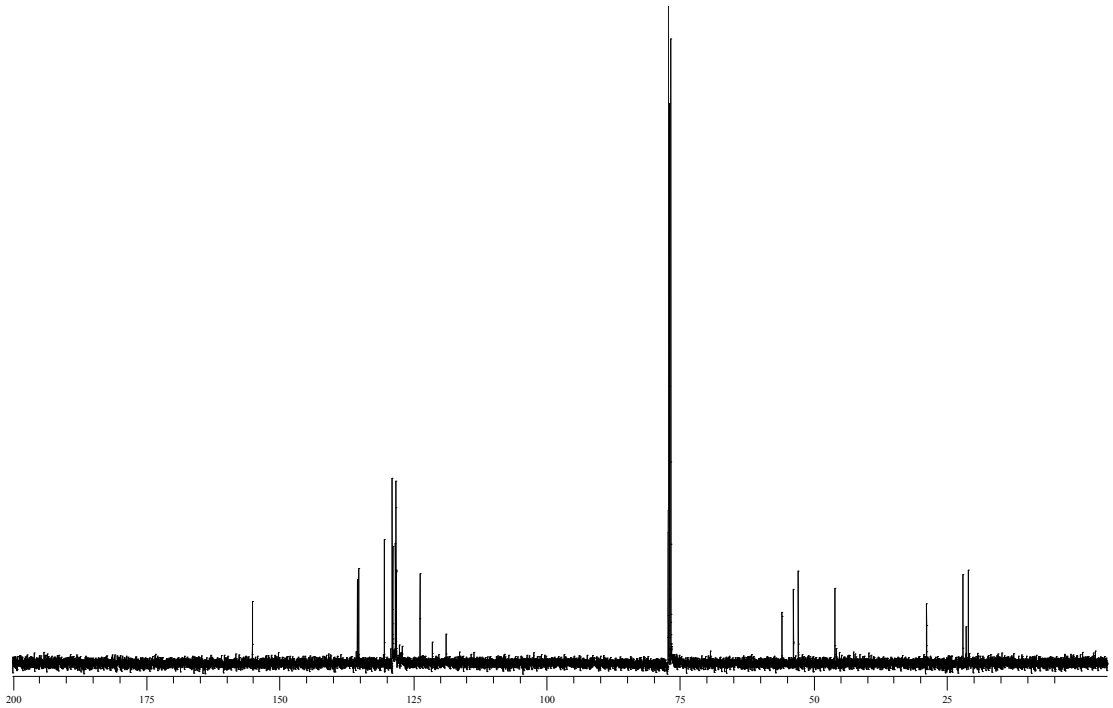
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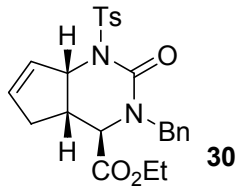
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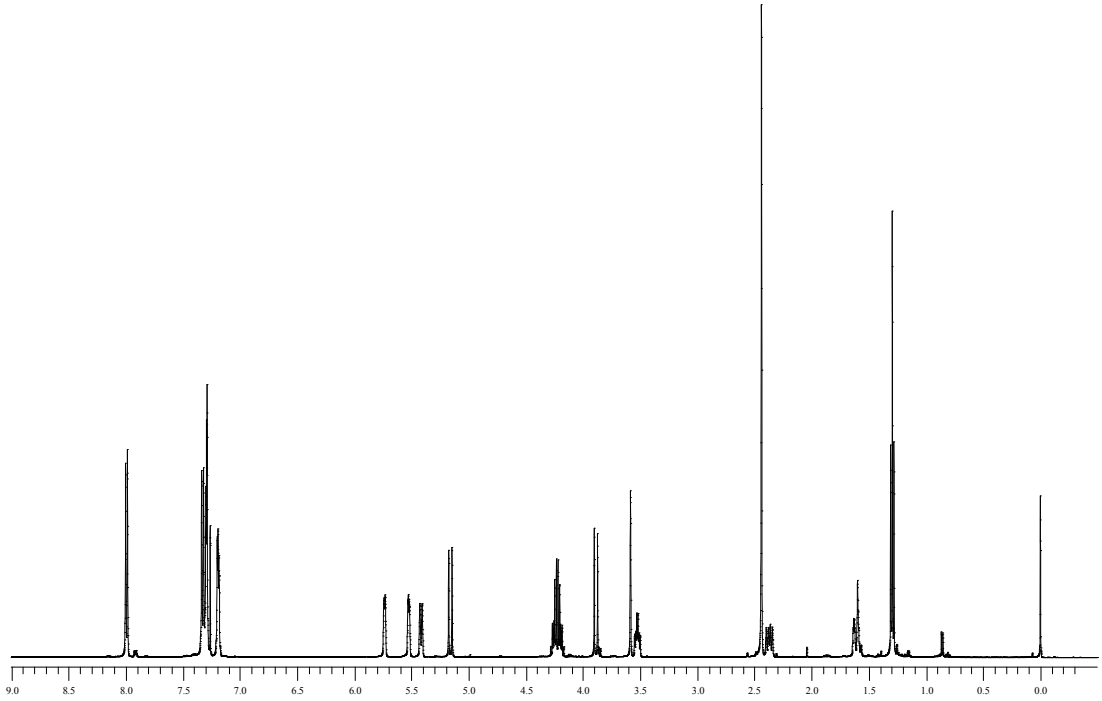
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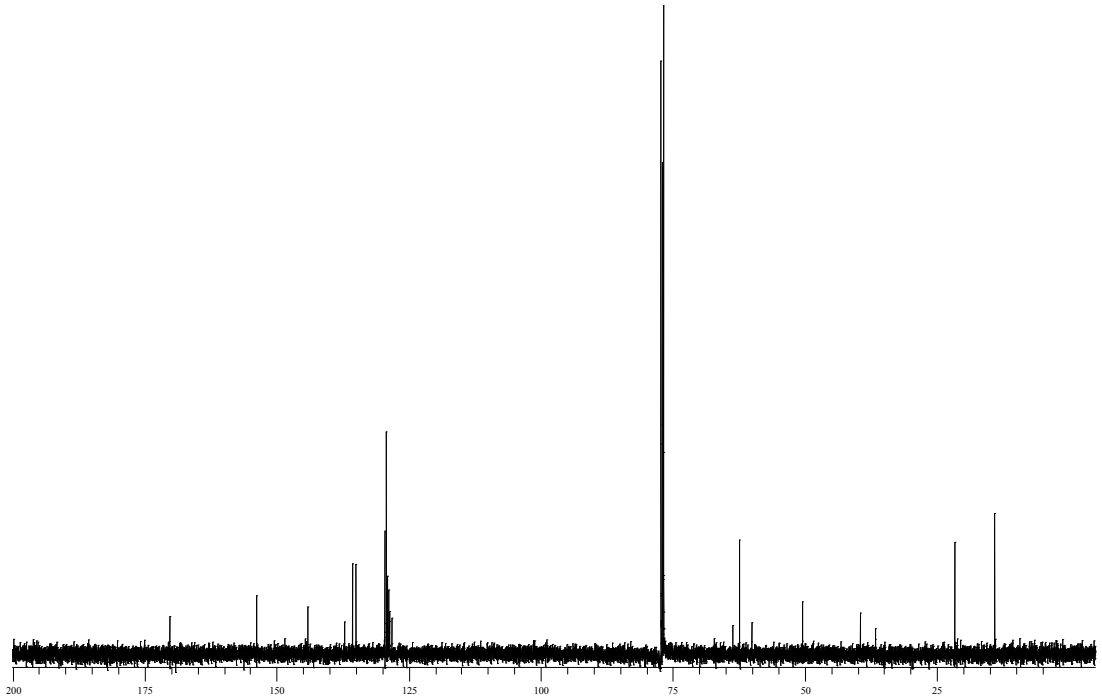
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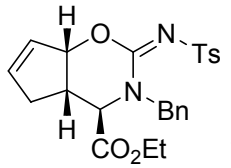
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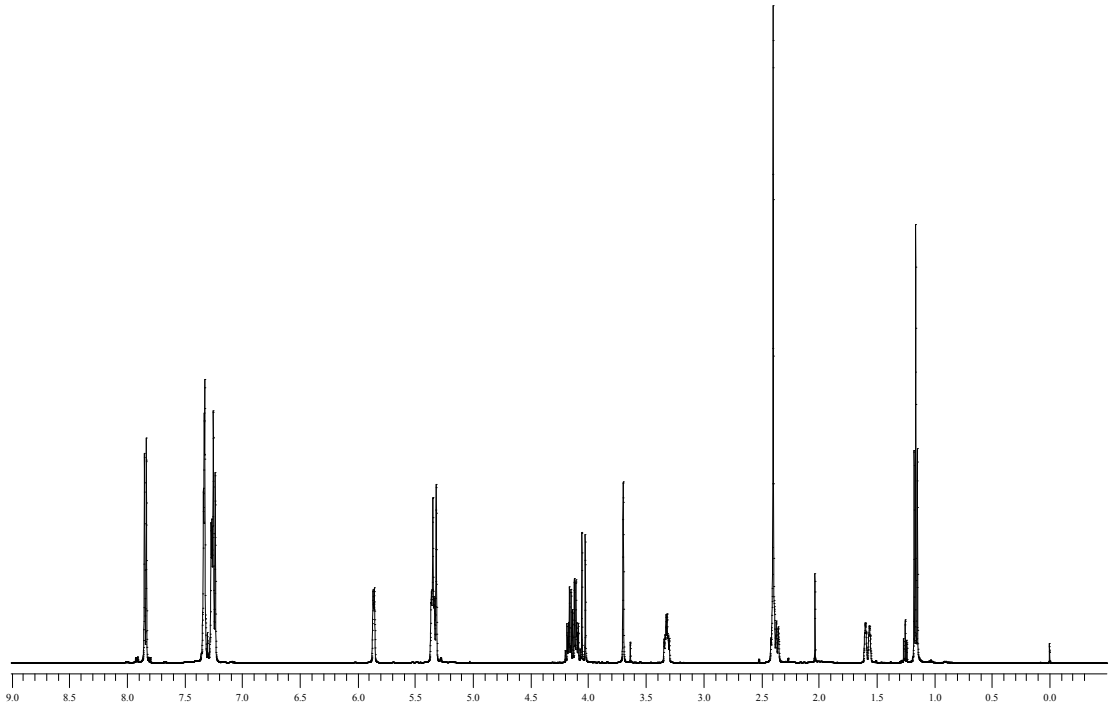
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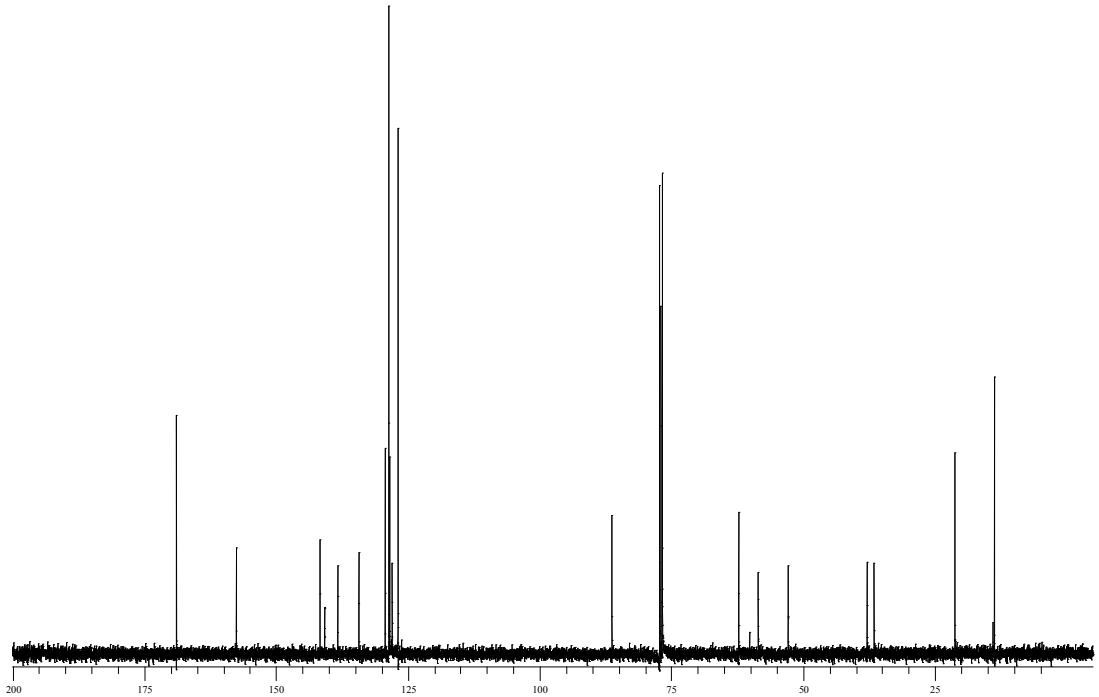
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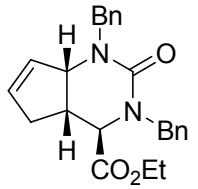
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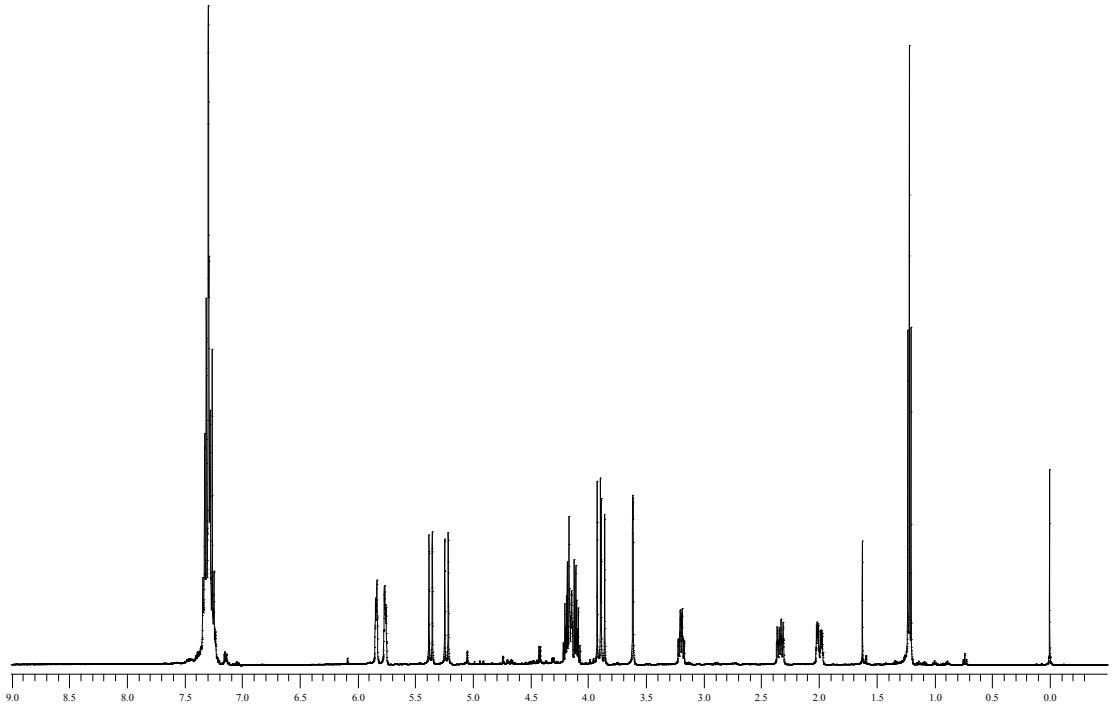
33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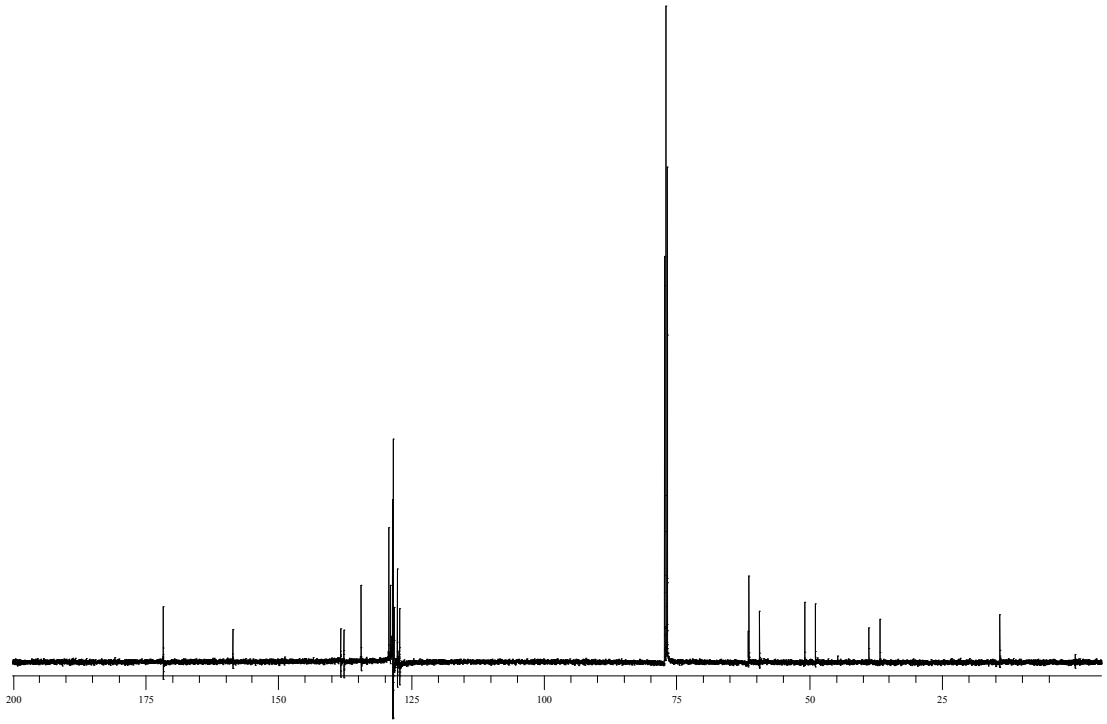
2.5
2.0
1.5
1.0
0.5
0.0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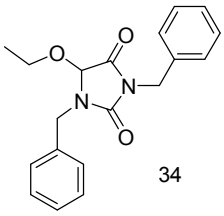
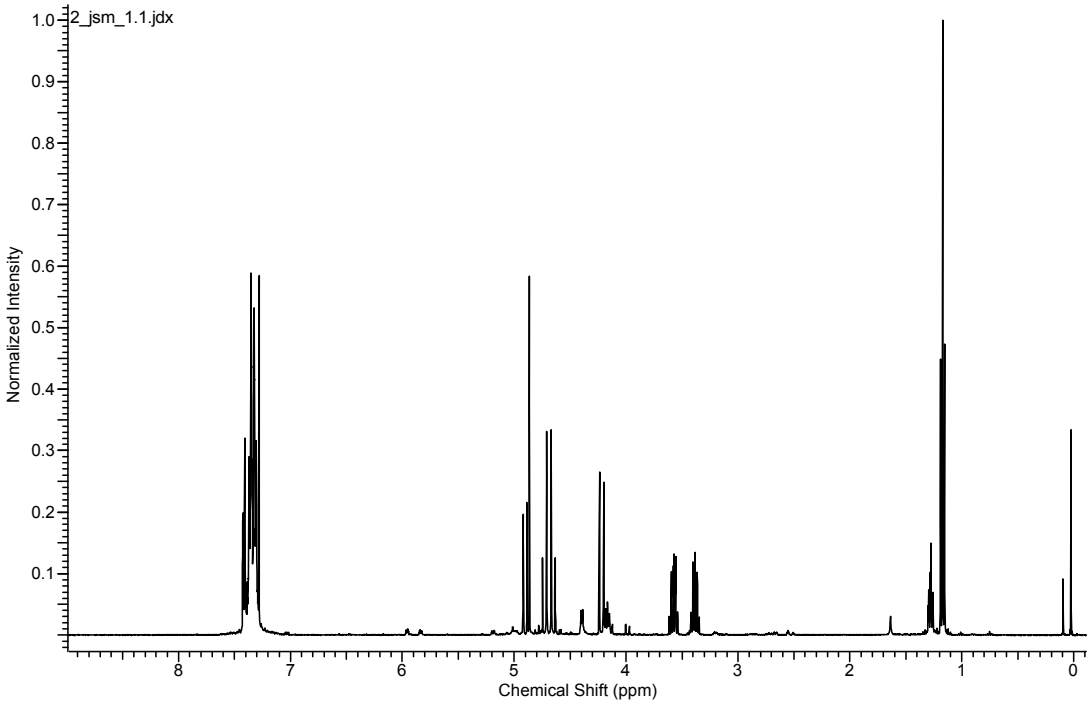
150
125
100
75
50
25


O
O
N
N
O
O

34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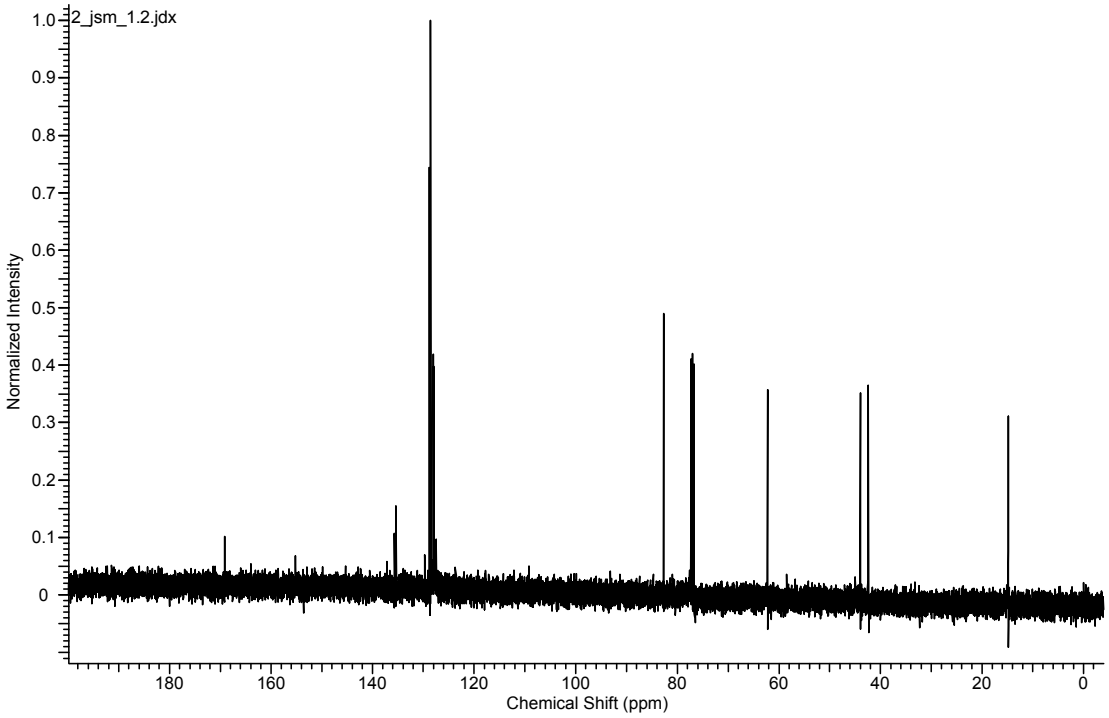
Normalized Intensity
Chemical Shift (ppm)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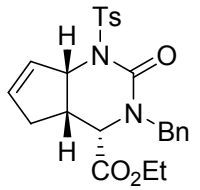
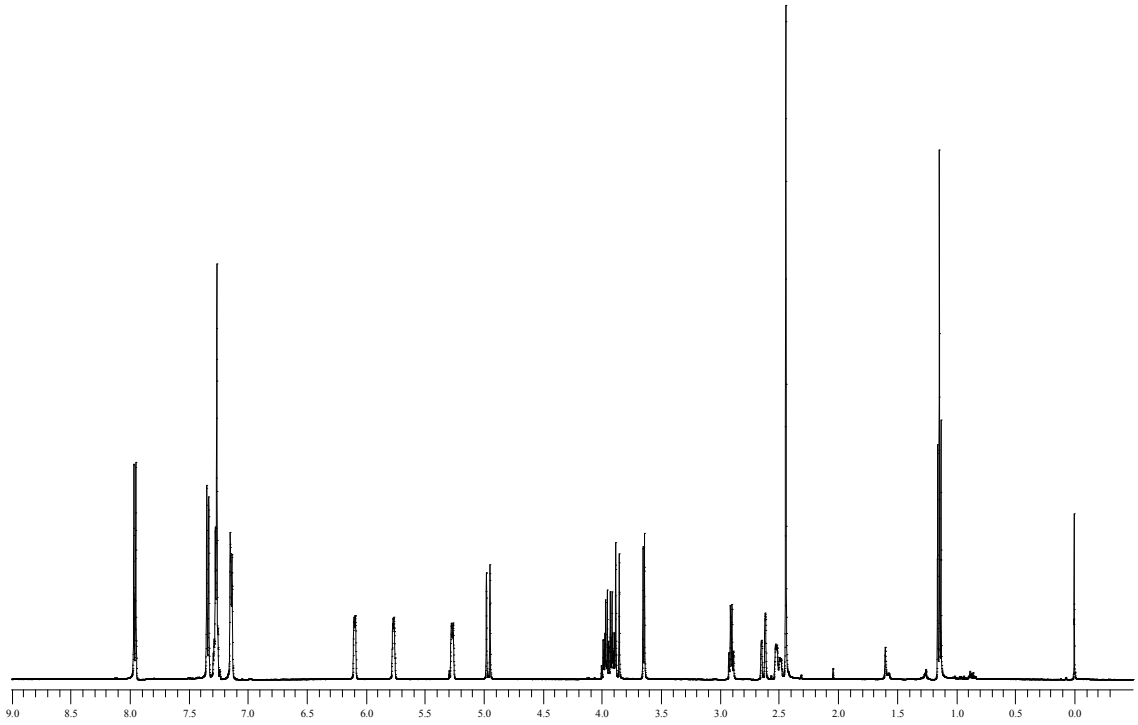

Ts
H
N
O
N
Bn
H
CO2Et

35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1.5
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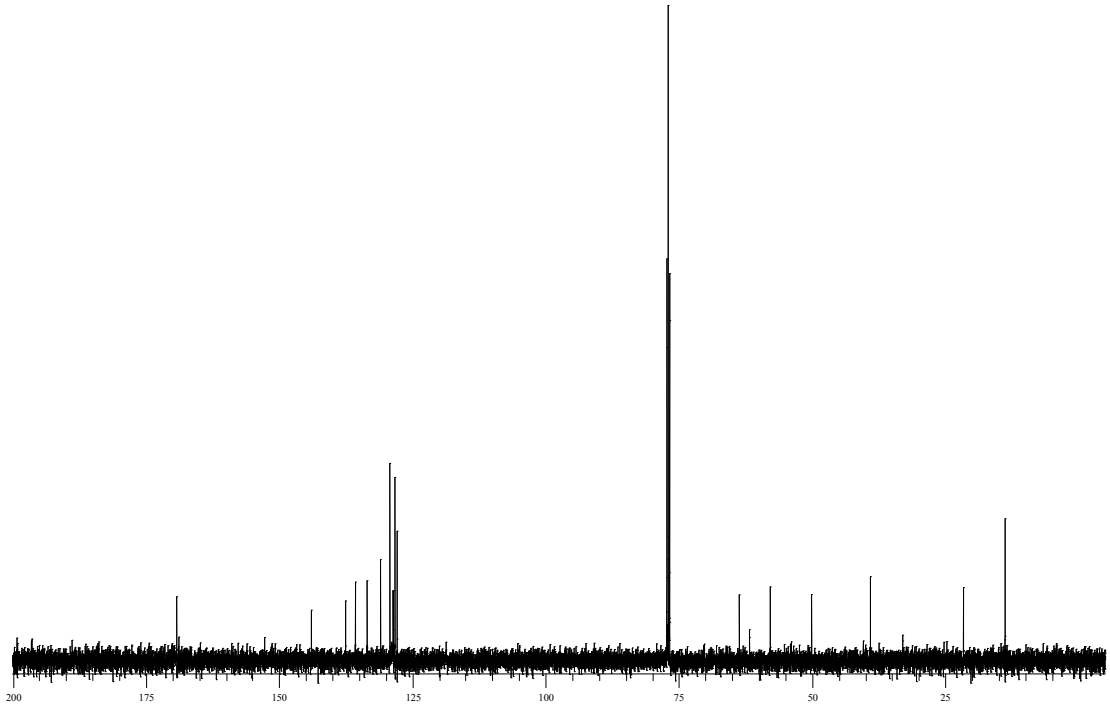
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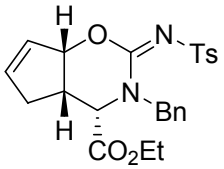
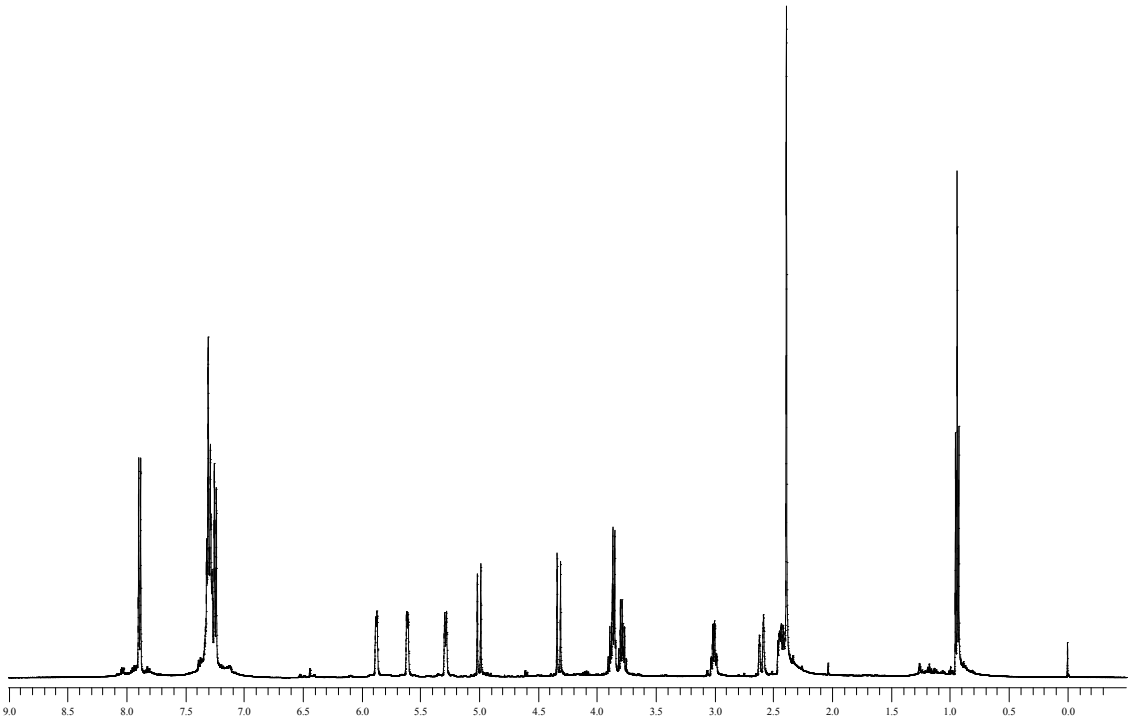
100
75
50
25


H
O
N
Ts
N
Bn
H
CO2Et

36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6.0
5.5
5.0
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4.0
3.5
3.0
2.5
2.0
1.5
1.0
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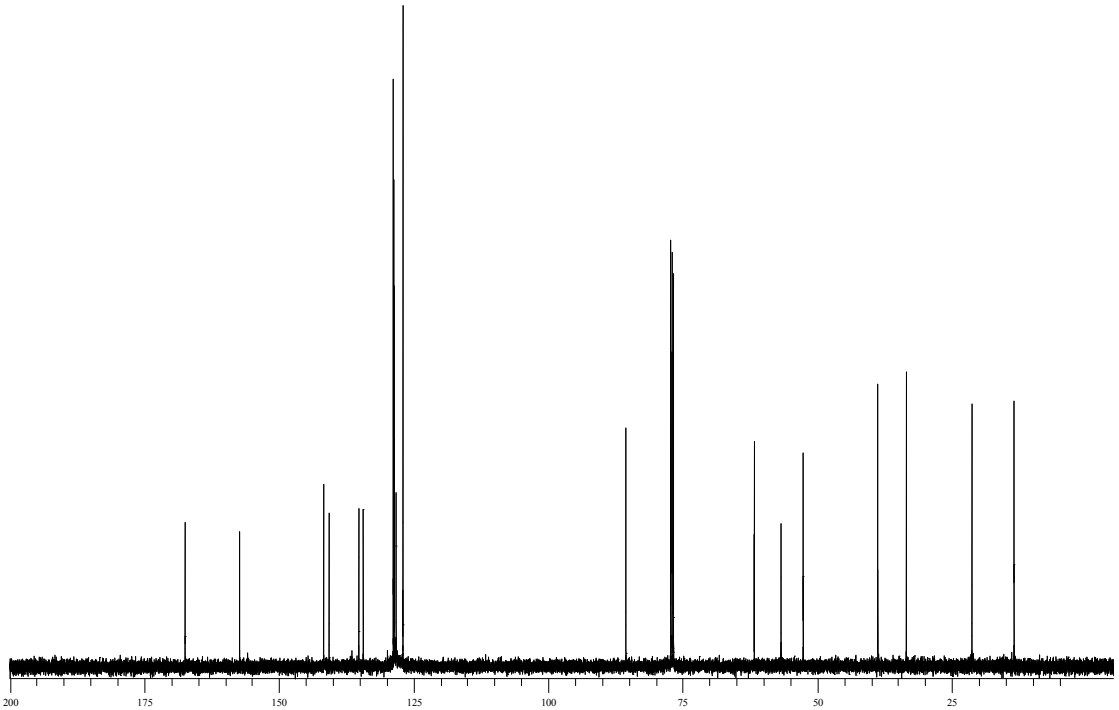
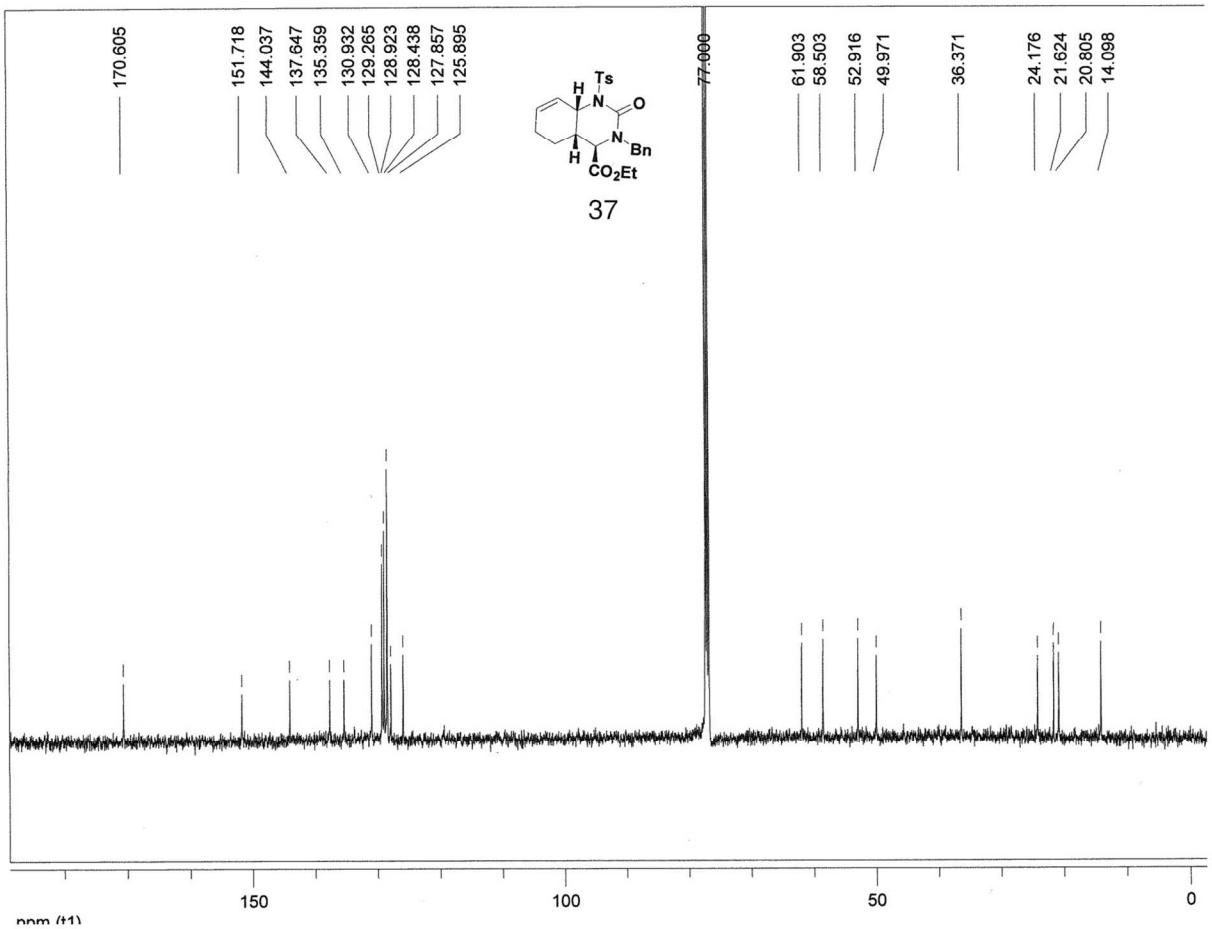
0.0


200
175
150
125
100
75
50
25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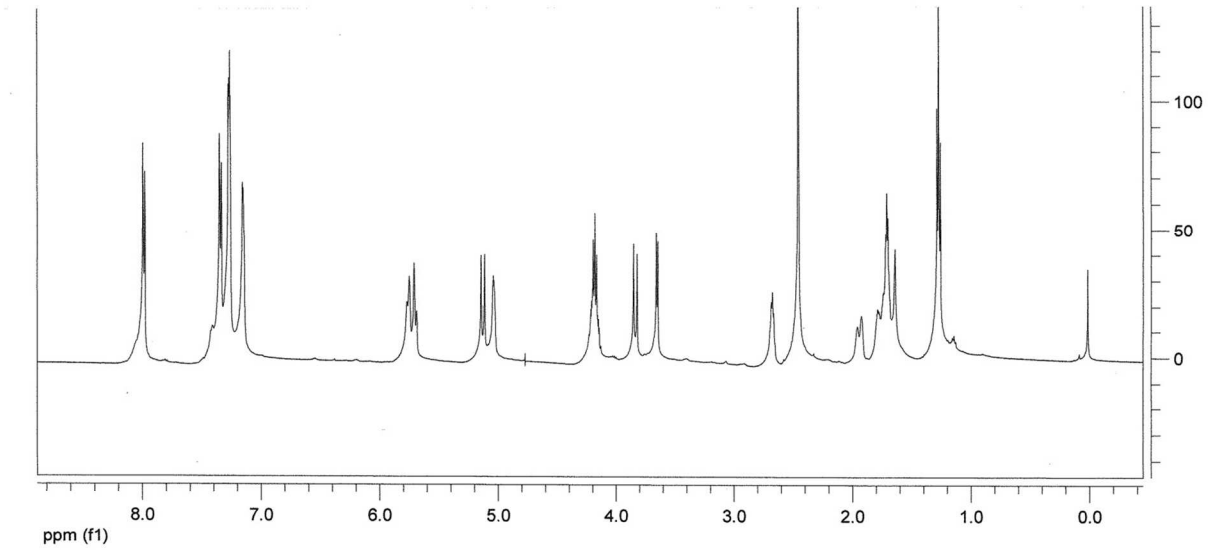
144.037
137.647
135.359
130.932
129.265
128.923
128.438
127.857
125.895
77.000
61.903
58.503
52.916
49.971
36.371
24.176
21.624
20.805
14.098
Ts
N
O
N
Bn
H
H
CO2Et
150
100
50
0
ppm (t1)

37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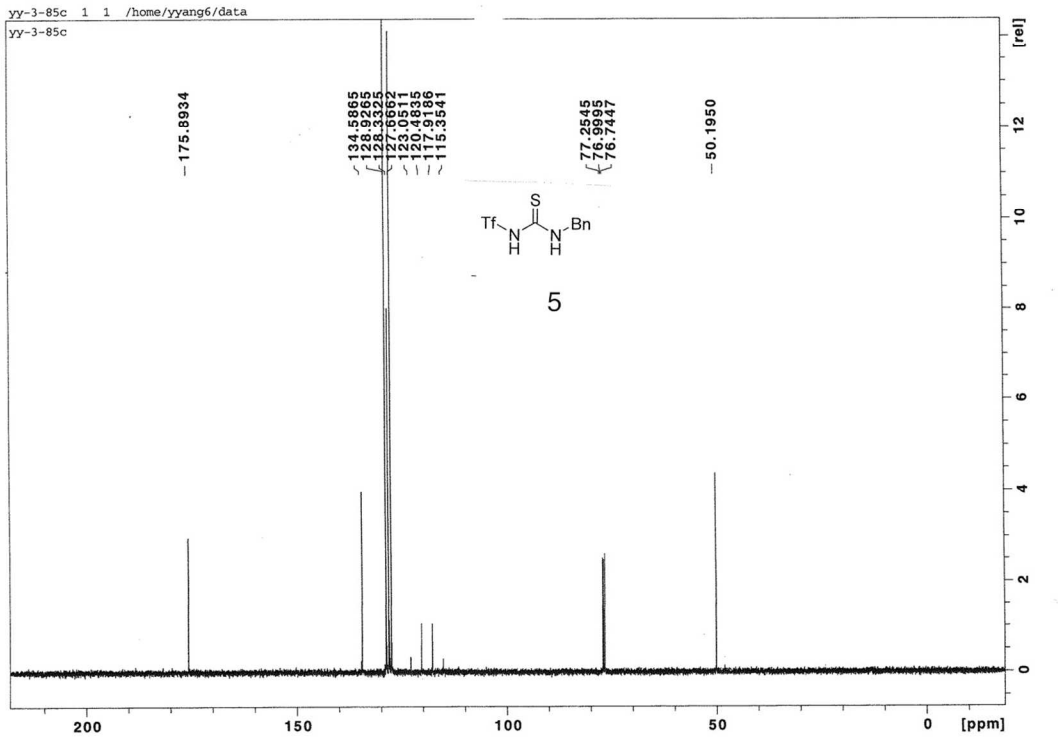
50
0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (f1)


yy-3-85c 1 1 /home/yyang6/data
yy-3-85c
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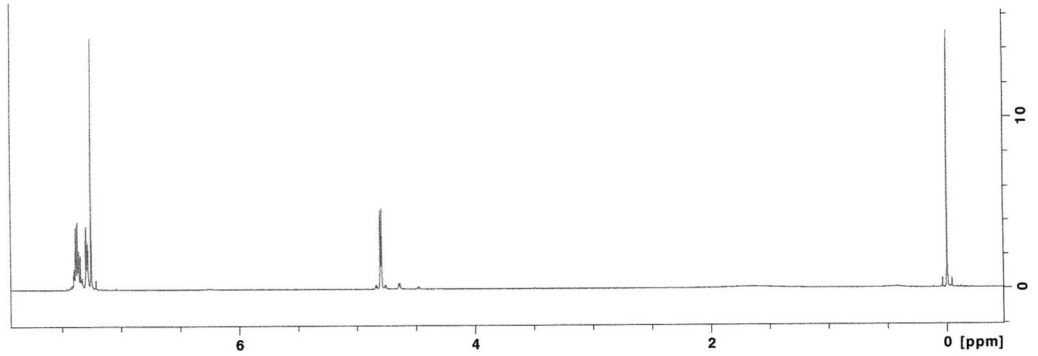
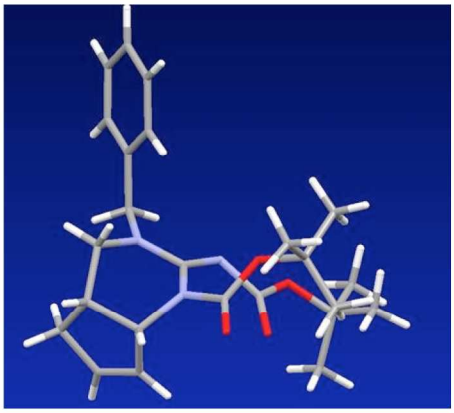
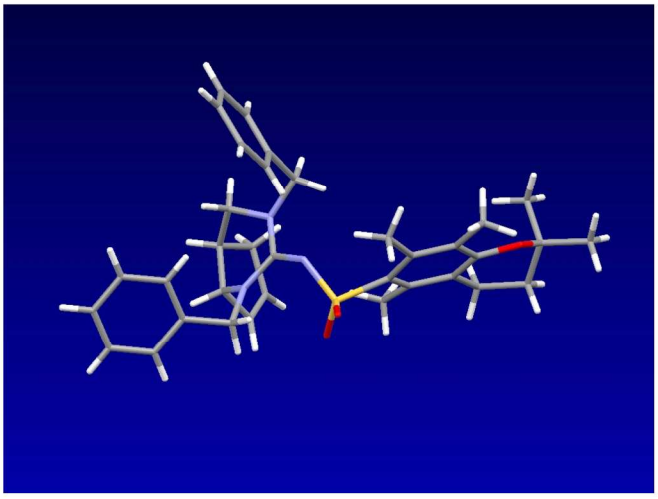
134.5865
134.0265
128.3325
127.6662
123.0511
120.4835
117.9186
115.3541
77.2545
76.9995
76.7447
50.1950
Tf
N
H
S
N
H
Bn
5
[rel]
[ppm]


[ppm]

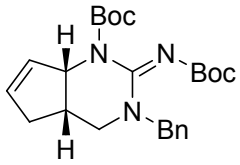
X-ray Figures





X-ray data for 19:

Crystallographic data (excluding structural factors) has been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC 800285



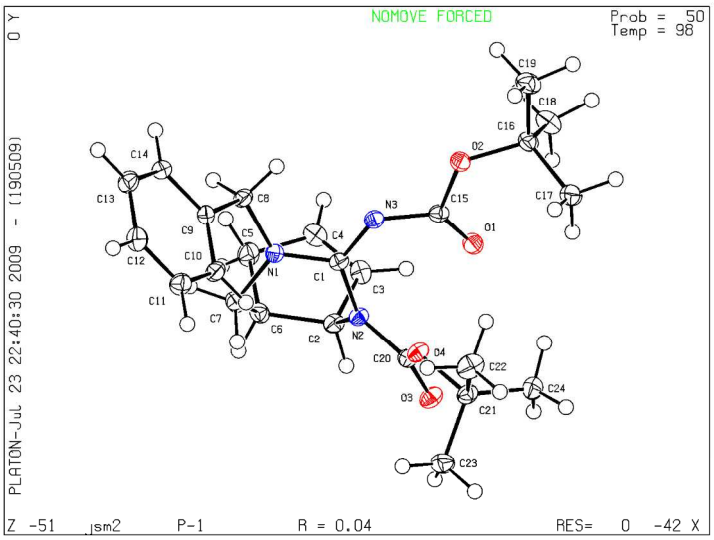


Table 1. Crystal data and structure refinement for **19**.

Identification code	jsm2 (Sample S-2)	
Empirical formula	$C_{24}\ H_{33}\ N_3\ O_4$	
Formula weight	427.53	
Temperature	98(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 9.7840(3) Å	α= 90.8517(3)°.
	b = 11.1537(3) Å	β= 98.4236(3)°.
	c = 11.4846(3) Å	γ = 112.3500(3)°.
Volume	1143.22(6) $Å^3$	
Z	2	
Density (calculated)	1.242 Mg/m^3	
Absorption coefficient	0.085 mm^{-1}	

F(000)	460
Crystal color	colorless
Crystal size	0.33 x 0.32 x 0.20 mm^3
Theta range for data collection	1.80 to 27.88°
Index ranges	$-12 \leq h \leq 12$, $-14 \leq k \leq 14$, $-15 \leq l \leq 15$
Reflections collected	13553
Independent reflections	5304 [R(int) = 0.0128]
Completeness to theta = 27.88°	97.4 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9832 and 0.9725
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5304 / 0 / 413
Goodness-of-fit on F^2	1.033
Final R indices [I>2sigma(I) = 4881 data]	R1 = 0.0353, wR2 = 0.0904
R indices (all data, 0.76Å)	R1 = 0.0378, wR2 = 0.0928
Extinction coefficient	0.0034(17)
Largest diff. peak and hole	0.349 and -0.203 $e.Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)

for **19**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	4786(1)	7062(1)	2442(1)	21(1)
O(2)	6834(1)	8952(1)	2677(1)	18(1)
O(3)	1151(1)	5712(1)	2784(1)	21(1)
O(4)	2725(1)	7687(1)	3684(1)	17(1)
N(1)	2733(1)	9376(1)	853(1)	16(1)
N(2)	2106(1)	7448(1)	1706(1)	15(1)
N(3)	4679(1)	9077(1)	2001(1)	15(1)
C(1)	3251(1)	8643(1)	1568(1)	14(1)
C(2)	1152(1)	6690(1)	601(1)	17(1)
C(3)	2035(1)	6217(1)	-128(1)	20(1)
C(4)	2072(1)	6683(1)	-1181(1)	21(1)
C(5)	1203(1)	7539(1)	-1387(1)	23(1)
C(6)	581(1)	7557(1)	-221(1)	19(1)
C(7)	1122(1)	8910(1)	403(1)	19(1)
C(8)	3726(1)	10634(1)	534(1)	18(1)
C(9)	3548(1)	11770(1)	1139(1)	16(1)

C(10)	2838(1)	11651(1)	2121(1)	19(1)
C(11)	2724(1)	12734(1)	2654(1)	21(1)
C(12)	3349(1)	13949(1)	2230(1)	21(1)
C(13)	4079(1)	14074(1)	1260(1)	21(1)
C(14)	4162(1)	12993(1)	708(1)	18(1)
C(15)	5338(1)	8234(1)	2392(1)	15(1)
C(16)	7877(1)	8346(1)	3137(1)	19(1)
C(17)	7527(1)	7808(1)	4317(1)	27(1)
C(18)	7870(1)	7311(1)	2253(1)	26(1)
C(19)	9376(1)	9505(1)	3306(1)	25(1)
C(20)	1960(1)	6840(1)	2757(1)	16(1)
C(21)	2696(1)	7238(1)	4898(1)	18(1)
C(22)	3832(1)	8433(1)	5646(1)	27(1)
C(23)	1132(1)	6911(1)	5192(1)	24(1)
C(24)	3187(1)	6096(1)	5018(1)	24(1)

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Table 3. Bond lengths [Å] and angles [°] for **19**.

O(1)-C(15)	1.2154(12)
O(2)-C(15)	1.3609(11)
O(2)-C(16)	1.4674(11)
O(3)-C(20)	1.2086(12)
O(4)-C(20)	1.3360(11)
O(4)-C(21)	1.4894(11)
N(1)-C(1)	1.3464(12)
N(1)-C(8)	1.4596(12)
N(1)-C(7)	1.4668(12)
N(2)-C(20)	1.3920(12)
N(2)-C(1)	1.4073(11)
N(2)-C(2)	1.4895(11)
N(3)-C(1)	1.3079(12)
N(3)-C(15)	1.3745(12)
C(2)-C(3)	1.5064(14)
C(2)-C(6)	1.5565(13)
C(2)-H(2A)	0.966(13)
C(3)-C(4)	1.3237(15)
C(3)-H(3A)	0.952(14)
C(4)-C(5)	1.5010(14)
C(4)-H(4A)	0.970(15)

C(5)-C(6)	1.5527(14)
C(5)-H(5A)	0.996(15)
C(5)-H(5B)	0.977(15)
C(6)-C(7)	1.5228(14)
C(6)-H(6A)	0.977(13)
C(7)-H(7A)	0.993(13)
C(7)-H(7B)	0.978(13)
C(8)-C(9)	1.5155(13)
C(8)-H(8A)	0.983(13)
C(8)-H(8B)	0.975(13)
C(9)-C(10)	1.3914(13)
C(9)-C(14)	1.3957(13)
C(10)-C(11)	1.3945(14)
C(10)-H(10A)	0.952(14)
C(11)-C(12)	1.3871(14)
C(11)-H(11A)	0.963(15)
C(12)-C(13)	1.3910(15)
C(12)-H(12A)	0.972(15)
C(13)-C(14)	1.3884(14)
C(13)-H(13A)	0.980(13)
C(14)-H(14A)	0.955(14)
C(16)-C(17)	1.5225(14)
C(16)-C(18)	1.5240(15)

C(16)-C(19)	1.5253(14)
C(17)-H(17A)	0.968(15)
C(17)-H(17B)	1.000(16)
C(17)-H(17C)	0.986(16)
C(18)-H(18A)	1.007(16)
C(18)-H(18B)	0.987(15)
C(18)-H(18C)	0.976(15)
C(19)-H(19A)	1.008(14)
C(19)-H(19B)	0.990(15)
C(19)-H(19C)	1.002(16)
C(21)-C(22)	1.5199(14)
C(21)-C(23)	1.5228(14)
C(21)-C(24)	1.5250(14)
C(22)-H(22A)	0.980(17)
C(22)-H(22B)	0.997(16)
C(22)-H(22C)	0.978(16)
C(23)-H(23A)	0.984(15)
C(23)-H(23B)	0.979(16)
C(23)-H(23C)	0.984(15)
C(24)-H(24A)	0.989(15)
C(24)-H(24B)	0.986(15)
C(24)-H(24C)	0.978(15)

C(15)-O(2)-C(16)	120.89(7)
C(20)-O(4)-C(21)	119.58(7)
C(1)-N(1)-C(8)	121.76(8)
C(1)-N(1)-C(7)	119.08(8)
C(8)-N(1)-C(7)	119.15(8)
C(20)-N(2)-C(1)	124.94(8)
C(20)-N(2)-C(2)	117.94(7)
C(1)-N(2)-C(2)	116.27(7)
C(1)-N(3)-C(15)	120.62(8)
N(3)-C(1)-N(1)	119.18(8)
N(3)-C(1)-N(2)	129.00(8)
N(1)-C(1)-N(2)	111.79(8)
N(2)-C(2)-C(3)	111.38(8)
N(2)-C(2)-C(6)	111.21(8)
C(3)-C(2)-C(6)	104.26(8)
N(2)-C(2)-H(2A)	105.2(8)
C(3)-C(2)-H(2A)	112.3(7)
C(6)-C(2)-H(2A)	112.6(8)
C(4)-C(3)-C(2)	112.41(9)
C(4)-C(3)-H(3A)	127.1(8)
C(2)-C(3)-H(3A)	120.5(8)
C(3)-C(4)-C(5)	112.98(9)
C(3)-C(4)-H(4A)	123.9(8)

C(5)-C(4)-H(4A)	123.1(8)
C(4)-C(5)-C(6)	104.29(8)
C(4)-C(5)-H(5A)	111.6(8)
C(6)-C(5)-H(5A)	112.3(8)
C(4)-C(5)-H(5B)	111.5(9)
C(6)-C(5)-H(5B)	111.2(9)
H(5A)-C(5)-H(5B)	106.1(12)
C(7)-C(6)-C(5)	113.51(8)
C(7)-C(6)-C(2)	108.63(8)
C(5)-C(6)-C(2)	106.06(8)
C(7)-C(6)-H(6A)	108.3(8)
C(5)-C(6)-H(6A)	110.5(8)
C(2)-C(6)-H(6A)	109.8(8)
N(1)-C(7)-C(6)	109.81(8)
N(1)-C(7)-H(7A)	106.9(8)
C(6)-C(7)-H(7A)	112.5(8)
N(1)-C(7)-H(7B)	108.8(7)
C(6)-C(7)-H(7B)	110.5(7)
H(7A)-C(7)-H(7B)	108.2(11)
N(1)-C(8)-C(9)	112.95(8)
N(1)-C(8)-H(8A)	107.5(8)
C(9)-C(8)-H(8A)	109.3(8)
N(1)-C(8)-H(8B)	107.1(7)

C(9)-C(8)-H(8B)	110.4(8)
H(8A)-C(8)-H(8B)	109.5(11)
C(10)-C(9)-C(14)	118.92(9)
C(10)-C(9)-C(8)	122.78(8)
C(14)-C(9)-C(8)	118.27(8)
C(9)-C(10)-C(11)	120.39(9)
C(9)-C(10)-H(10A)	120.2(8)
C(11)-C(10)-H(10A)	119.4(8)
C(12)-C(11)-C(10)	120.48(9)
C(12)-C(11)-H(11A)	120.1(8)
C(10)-C(11)-H(11A)	119.5(8)
C(11)-C(12)-C(13)	119.22(9)
C(11)-C(12)-H(12A)	120.1(9)
C(13)-C(12)-H(12A)	120.7(9)
C(14)-C(13)-C(12)	120.45(9)
C(14)-C(13)-H(13A)	121.1(8)
C(12)-C(13)-H(13A)	118.4(8)
C(13)-C(14)-C(9)	120.50(9)
C(13)-C(14)-H(14A)	120.8(8)
C(9)-C(14)-H(14A)	118.7(8)
O(1)-C(15)-O(2)	123.27(9)
O(1)-C(15)-N(3)	129.97(9)
O(2)-C(15)-N(3)	106.67(8)

O(2)-C(16)-C(17)	109.89(8)
O(2)-C(16)-C(18)	111.91(8)
C(17)-C(16)-C(18)	111.99(9)
O(2)-C(16)-C(19)	101.57(8)
C(17)-C(16)-C(19)	110.63(9)
C(18)-C(16)-C(19)	110.40(9)
C(16)-C(17)-H(17A)	111.6(9)
C(16)-C(17)-H(17B)	110.5(9)
H(17A)-C(17)-H(17B)	108.6(12)
C(16)-C(17)-H(17C)	109.8(9)
H(17A)-C(17)-H(17C)	107.2(12)
H(17B)-C(17)-H(17C)	109.0(12)
C(16)-C(18)-H(18A)	109.0(9)
C(16)-C(18)-H(18B)	110.4(9)
H(18A)-C(18)-H(18B)	108.3(12)
C(16)-C(18)-H(18C)	111.7(9)
H(18A)-C(18)-H(18C)	108.7(12)
H(18B)-C(18)-H(18C)	108.7(12)
C(16)-C(19)-H(19A)	110.0(8)
C(16)-C(19)-H(19B)	110.5(9)
H(19A)-C(19)-H(19B)	108.0(11)
C(16)-C(19)-H(19C)	110.0(9)
H(19A)-C(19)-H(19C)	109.6(12)

H(19B)-C(19)-H(19C)	108.7(12)
O(3)-C(20)-O(4)	126.78(9)
O(3)-C(20)-N(2)	122.10(9)
O(4)-C(20)-N(2)	110.93(8)
O(4)-C(21)-C(22)	102.27(8)
O(4)-C(21)-C(23)	108.65(8)
C(22)-C(21)-C(23)	111.07(9)
O(4)-C(21)-C(24)	111.31(8)
C(22)-C(21)-C(24)	110.73(9)
C(23)-C(21)-C(24)	112.36(9)
C(21)-C(22)-H(22A)	109.7(10)
C(21)-C(22)-H(22B)	110.0(9)
H(22A)-C(22)-H(22B)	109.7(13)
C(21)-C(22)-H(22C)	110.2(9)
H(22A)-C(22)-H(22C)	108.7(13)
H(22B)-C(22)-H(22C)	108.5(13)
C(21)-C(23)-H(23A)	111.4(8)
C(21)-C(23)-H(23B)	110.6(9)
H(23A)-C(23)-H(23B)	108.6(12)
C(21)-C(23)-H(23C)	109.3(9)
H(23A)-C(23)-H(23C)	108.9(12)
H(23B)-C(23)-H(23C)	107.8(12)
C(21)-C(24)-H(24A)	111.9(8)

C(21)-C(24)-H(24B)	108.4(9)
H(24A)-C(24)-H(24B)	108.9(12)
C(21)-C(24)-H(24C)	110.5(9)
H(24A)-C(24)-H(24C)	110.4(12)
H(24B)-C(24)-H(24C)	106.5(12)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for jsm2. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	17(1)	15(1)	29(1)	3(1)	3(1)	6(1)
O(2)	13(1)	19(1)	21(1)	3(1)	1(1)	4(1)
O(3)	22(1)	16(1)	20(1)	3(1)	3(1)	2(1)
O(4)	20(1)	16(1)	14(1)	1(1)	3(1)	4(1)
N(1)	16(1)	15(1)	18(1)	3(1)	4(1)	7(1)
N(2)	14(1)	14(1)	14(1)	1(1)	1(1)	2(1)
N(3)	15(1)	14(1)	16(1)	1(1)	3(1)	4(1)
C(1)	17(1)	13(1)	13(1)	0(1)	4(1)	5(1)
C(2)	15(1)	16(1)	16(1)	0(1)	0(1)	2(1)
C(3)	20(1)	16(1)	21(1)	-3(1)	-2(1)	7(1)
C(4)	20(1)	23(1)	20(1)	-4(1)	1(1)	8(1)
C(5)	27(1)	28(1)	16(1)	2(1)	3(1)	14(1)
C(6)	16(1)	23(1)	17(1)	1(1)	1(1)	8(1)
C(7)	17(1)	23(1)	20(1)	2(1)	3(1)	11(1)
C(8)	21(1)	15(1)	20(1)	6(1)	9(1)	9(1)
C(9)	16(1)	16(1)	17(1)	2(1)	2(1)	7(1)
C(10)	22(1)	17(1)	19(1)	5(1)	6(1)	8(1)
C(11)	25(1)	23(1)	17(1)	2(1)	6(1)	11(1)

C(12)	25(1)	18(1)	22(1)	-2(1)	2(1)	10(1)
C(13)	22(1)	15(1)	25(1)	4(1)	4(1)	6(1)
C(14)	18(1)	18(1)	20(1)	4(1)	6(1)	7(1)
C(15)	15(1)	18(1)	13(1)	0(1)	3(1)	4(1)
C(16)	14(1)	26(1)	18(1)	3(1)	2(1)	8(1)
C(17)	20(1)	42(1)	22(1)	12(1)	6(1)	14(1)
C(18)	20(1)	30(1)	29(1)	-2(1)	4(1)	11(1)
C(19)	16(1)	32(1)	24(1)	1(1)	1(1)	6(1)
C(20)	14(1)	16(1)	17(1)	1(1)	3(1)	6(1)
C(21)	20(1)	21(1)	13(1)	3(1)	4(1)	7(1)
C(22)	29(1)	28(1)	18(1)	-1(1)	1(1)	4(1)
C(23)	22(1)	29(1)	22(1)	4(1)	8(1)	11(1)
C(24)	25(1)	28(1)	22(1)	7(1)	6(1)	14(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **19**.

	x	y	z	U(eq)
H(2A)	346(14)	5983(12)	857(11)	19(3)
H(3A)	2501(15)	5654(13)	184(12)	27(3)
H(4A)	2591(16)	6486(14)	-1768(13)	30(3)
H(5A)	1847(16)	8425(14)	-1572(13)	32(4)
H(5B)	392(17)	7192(14)	-2060(13)	33(4)
H(6A)	-514(15)	7197(12)	-370(12)	23(3)
H(7A)	974(15)	9557(13)	-133(12)	23(3)
H(7B)	591(14)	8894(12)	1065(11)	19(3)
H(8A)	3493(15)	10653(13)	-326(12)	23(3)
H(8B)	4749(15)	10683(12)	747(11)	21(3)
H(10A)	2430(15)	10829(13)	2439(12)	24(3)
H(11A)	2221(16)	12634(14)	3329(13)	30(3)
H(12A)	3266(16)	14700(14)	2606(13)	31(4)
H(13A)	4524(15)	14939(13)	973(12)	24(3)
H(14A)	4644(15)	13070(13)	32(12)	23(3)
H(17A)	6592(17)	7057(15)	4223(13)	34(4)

H(17B)	7463(17)	8486(15)	4862(14)	37(4)
H(17C)	8321(17)	7527(14)	4685(13)	36(4)
H(18A)	8749(18)	7067(15)	2524(14)	38(4)
H(18B)	7968(16)	7652(14)	1467(14)	32(4)
H(18C)	6953(17)	6531(15)	2181(13)	34(4)
H(19A)	9555(16)	9902(14)	2531(13)	30(3)
H(19B)	9378(17)	10181(14)	3875(13)	34(4)
H(19C)	10207(18)	9220(14)	3615(13)	36(4)
H(22A)	3537(18)	9174(16)	5517(15)	42(4)
H(22B)	4846(18)	8642(14)	5434(13)	35(4)
H(22C)	3878(18)	8274(15)	6484(15)	40(4)
H(23A)	371(16)	6220(14)	4634(13)	29(3)
H(23B)	878(17)	7680(15)	5171(14)	38(4)
H(23C)	1103(16)	6618(14)	5996(13)	33(4)
H(24A)	2401(16)	5279(14)	4625(13)	31(4)
H(24B)	3409(17)	5982(14)	5865(14)	35(4)
H(24C)	4119(17)	6285(14)	4706(13)	33(4)

Table 6. Torsion angles [°] for jsm2.

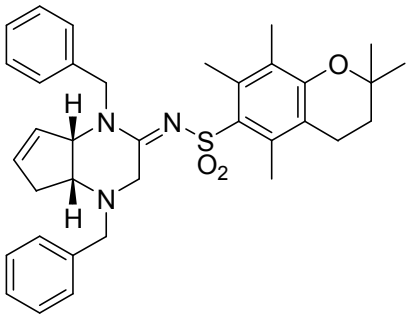
C(15)-N(3)-C(1)-N(1)	-158.27(8)
C(15)-N(3)-C(1)-N(2)	19.47(14)
C(8)-N(1)-C(1)-N(3)	0.92(13)
C(7)-N(1)-C(1)-N(3)	-177.76(8)
C(8)-N(1)-C(1)-N(2)	-177.19(8)
C(7)-N(1)-C(1)-N(2)	4.14(12)
C(20)-N(2)-C(1)-N(3)	40.96(14)
C(2)-N(2)-C(1)-N(3)	-128.17(10)
C(20)-N(2)-C(1)-N(1)	-141.17(9)
C(2)-N(2)-C(1)-N(1)	49.71(11)
C(20)-N(2)-C(2)-C(3)	-103.15(10)
C(1)-N(2)-C(2)-C(3)	66.76(10)
C(20)-N(2)-C(2)-C(6)	141.02(8)
C(1)-N(2)-C(2)-C(6)	-49.06(11)
N(2)-C(2)-C(3)-C(4)	-118.92(9)
C(6)-C(2)-C(3)-C(4)	1.10(11)
C(2)-C(3)-C(4)-C(5)	-1.10(12)
C(3)-C(4)-C(5)-C(6)	0.59(12)
C(4)-C(5)-C(6)-C(7)	119.31(9)
C(4)-C(5)-C(6)-C(2)	0.12(10)
N(2)-C(2)-C(6)-C(7)	-2.89(11)

C(3)-C(2)-C(6)-C(7)	-123.03(8)
N(2)-C(2)-C(6)-C(5)	119.46(9)
C(3)-C(2)-C(6)-C(5)	-0.68(10)
C(1)-N(1)-C(7)-C(6)	-54.96(11)
C(8)-N(1)-C(7)-C(6)	126.33(9)
C(5)-C(6)-C(7)-N(1)	-68.19(10)
C(2)-C(6)-C(7)-N(1)	49.52(10)
C(1)-N(1)-C(8)-C(9)	-107.18(10)
C(7)-N(1)-C(8)-C(9)	71.49(11)
N(1)-C(8)-C(9)-C(10)	16.67(13)
N(1)-C(8)-C(9)-C(14)	-165.23(8)
C(14)-C(9)-C(10)-C(11)	0.92(15)
C(8)-C(9)-C(10)-C(11)	179.01(9)
C(9)-C(10)-C(11)-C(12)	-1.56(16)
C(10)-C(11)-C(12)-C(13)	0.56(16)
C(11)-C(12)-C(13)-C(14)	1.07(15)
C(12)-C(13)-C(14)-C(9)	-1.72(15)
C(10)-C(9)-C(14)-C(13)	0.71(15)
C(8)-C(9)-C(14)-C(13)	-177.47(9)
C(16)-O(2)-C(15)-O(1)	-3.60(13)
C(16)-O(2)-C(15)-N(3)	179.58(7)
C(1)-N(3)-C(15)-O(1)	-2.36(15)
C(1)-N(3)-C(15)-O(2)	174.18(8)

C(15)-O(2)-C(16)-C(17)	-63.44(11)
C(15)-O(2)-C(16)-C(18)	61.63(11)
C(15)-O(2)-C(16)-C(19)	179.39(8)
C(21)-O(4)-C(20)-O(3)	3.91(14)
C(21)-O(4)-C(20)-N(2)	178.99(7)
C(1)-N(2)-C(20)-O(3)	-165.02(9)
C(2)-N(2)-C(20)-O(3)	3.93(13)
C(1)-N(2)-C(20)-O(4)	19.63(13)
C(2)-N(2)-C(20)-O(4)	-171.42(8)
C(20)-O(4)-C(21)-C(22)	173.33(8)
C(20)-O(4)-C(21)-C(23)	-69.18(11)
C(20)-O(4)-C(21)-C(24)	55.05(11)

X-ray data for 21:

Crystallographic data (excluding structural factors) has been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC 800286



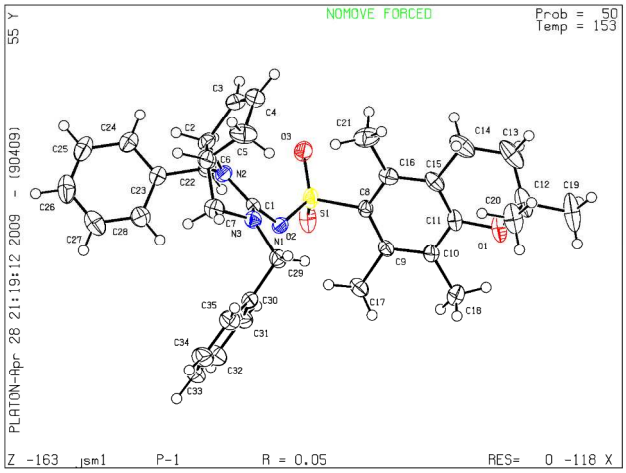


Table 1. Crystal data and structure refinement for **21**.

Identification code	jsm1	
Empirical formula	$C_{35}\ H_{41}\ N_3\ O_3\ S$	
Formula weight	583.77	
Temperature	153(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 9.705(2) Å	α= 103.347(3)°.
	b = 10.735(3) Å	β= 106.187(3)°.
	c = 15.984(4) Å	γ = 90.024(3)°.
Volume	1552.4(6) $Å^3$	

Z	2
Density (calculated)	1.249 Mg/m^3
Absorption coefficient	0.144 mm^{-1}
F(000)	624
Crystal color	colorless
Crystal size	0.20 x 0.14 x 0.12 mm^3
Theta range for data collection	1.95 to 26.37°
Index ranges	$-12 \le h \le 12$, $-13 \le k \le 13$, $-19 \le l \le 19$
Reflections collected	16698
Independent reflections	6312 [R(int) = 0.0371]
Completeness to theta = 26.37°	99.5 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9825 and 0.9714
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6312 / 0 / 384
Goodness-of-fit on F^2	1.037
Final R indices [I>2sigma(I) = 4437 data]	R1 = 0.0549, wR2 = 0.1303
R indices (all data, 0.80Å)	R1 = 0.0853, wR2 = 0.1445
Largest diff. peak and hole	0.411 and -0.425 $e.Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)

for **21**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	3448(1)	231(1)	3126(1)	32(1)
O(1)	542(2)	4598(2)	1918(1)	45(1)
O(2)	2228(2)	-627(2)	2978(1)	48(1)
O(3)	4275(2)	700(2)	4049(1)	51(1)
N(1)	4381(2)	-405(2)	2483(1)	30(1)
N(2)	6546(2)	-782(2)	3510(1)	28(1)
N(3)	6597(2)	-64(2)	2270(1)	29(1)
C(1)	5811(3)	-396(2)	2771(2)	26(1)
C(2)	8068(3)	-332(2)	3994(2)	33(1)
C(3)	8172(3)	880(3)	4710(2)	38(1)
C(4)	8778(3)	1850(3)	4535(2)	41(1)
C(5)	9193(4)	1490(3)	3678(2)	51(1)
C(6)	8902(3)	15(3)	3393(2)	37(1)
C(7)	8068(3)	-464(3)	2418(2)	36(1)
C(8)	2754(2)	1580(2)	2704(2)	24(1)
C(9)	1542(2)	1368(2)	1944(2)	25(1)

C(10)	813(3)	2406(2)	1711(2)	29(1)
C(11)	1341(3)	3643(2)	2208(2)	30(1)
C(12)	1202(3)	5893(3)	2211(2)	50(1)
C(13)	1931(4)	6150(3)	3212(2)	56(1)
C(14)	3110(3)	5238(3)	3434(2)	49(1)
C(15)	2596(3)	3873(2)	2921(2)	32(1)
C(16)	3356(3)	2828(2)	3158(2)	30(1)
C(17)	1089(3)	39(2)	1324(2)	34(1)
C(18)	-535(3)	2248(3)	923(2)	47(1)
C(19)	-64(4)	6718(3)	1978(3)	71(1)
C(20)	2235(4)	6015(3)	1671(2)	56(1)
C(21)	4824(3)	3118(3)	3838(2)	48(1)
C(22)	5868(3)	-1651(2)	3899(2)	31(1)
C(23)	6641(2)	-2873(2)	3852(2)	28(1)
C(24)	7374(3)	-3233(2)	4630(2)	34(1)
C(25)	8122(3)	-4333(3)	4588(2)	46(1)
C(26)	8146(3)	-5084(3)	3771(2)	52(1)
C(27)	7415(3)	-4749(3)	2990(2)	48(1)
C(28)	6656(3)	-3651(3)	3028(2)	37(1)
C(29)	5874(3)	136(2)	1378(2)	32(1)
C(30)	5669(3)	-1116(2)	661(2)	29(1)
C(31)	4576(3)	-2034(3)	543(2)	36(1)
C(32)	4431(3)	-3189(3)	-82(2)	43(1)

C(33)	5369(3)	-3443(3)	-603(2)	42(1)
C(34)	6438(3)	-2541(3)	-506(2)	41(1)
C(35)	6587(3)	-1382(3)	127(2)	36(1)

Table 3. Bond lengths [Å] and angles [°] for 21.

S(1)-O(2)	1.431(2)
S(1)-O(3)	1.440(2)
S(1)-N(1)	1.593(2)
S(1)-C(8)	1.790(2)
O(1)-C(11)	1.374(3)
O(1)-C(12)	1.447(3)
N(1)-C(1)	1.334(3)
N(2)-C(1)	1.354(3)
N(2)-C(22)	1.479(3)
N(2)-C(2)	1.484(3)
N(3)-C(1)	1.350(3)
N(3)-C(7)	1.461(3)
N(3)-C(29)	1.469(3)
C(2)-C(3)	1.504(4)
C(2)-C(6)	1.521(4)
C(3)-C(4)	1.317(4)
C(4)-C(5)	1.501(4)
C(5)-C(6)	1.545(4)

C(6)-C(7)	1.509(4)
C(8)-C(16)	1.406(3)
C(8)-C(9)	1.409(3)
C(9)-C(10)	1.388(3)
C(9)-C(17)	1.522(3)
C(10)-C(11)	1.394(3)
C(10)-C(18)	1.520(3)
C(11)-C(15)	1.392(4)
C(12)-C(13)	1.517(5)
C(12)-C(20)	1.517(4)
C(12)-C(19)	1.527(4)
C(13)-C(14)	1.526(5)
C(14)-C(15)	1.512(4)
C(15)-C(16)	1.409(4)
C(16)-C(21)	1.513(4)
C(22)-C(23)	1.508(3)
C(23)-C(24)	1.390(3)
C(23)-C(28)	1.392(4)
C(24)-C(25)	1.384(4)
C(25)-C(26)	1.373(5)
C(26)-C(27)	1.380(4)
C(27)-C(28)	1.389(4)
C(29)-C(30)	1.523(3)

C(30)-C(35)	1.384(3)
C(30)-C(31)	1.390(4)
C(31)-C(32)	1.380(4)
C(32)-C(33)	1.384(4)
C(33)-C(34)	1.370(4)
C(34)-C(35)	1.389(4)

O(2)-S(1)-O(3)	115.30(13)
O(2)-S(1)-N(1)	108.82(12)
O(3)-S(1)-N(1)	113.76(11)
O(2)-S(1)-C(8)	106.06(11)
O(3)-S(1)-C(8)	107.96(12)
N(1)-S(1)-C(8)	104.04(11)
C(11)-O(1)-C(12)	118.0(2)
C(1)-N(1)-S(1)	122.27(17)
C(1)-N(2)-C(22)	122.0(2)
C(1)-N(2)-C(2)	122.2(2)
C(22)-N(2)-C(2)	115.83(19)
C(1)-N(3)-C(7)	117.7(2)
C(1)-N(3)-C(29)	119.8(2)
C(7)-N(3)-C(29)	116.9(2)
N(1)-C(1)-N(3)	118.9(2)
N(1)-C(1)-N(2)	124.5(2)

N(3)-C(1)-N(2)	116.4(2)
N(2)-C(2)-C(3)	111.3(2)
N(2)-C(2)-C(6)	113.3(2)
C(3)-C(2)-C(6)	104.5(2)
C(4)-C(3)-C(2)	111.4(2)
C(3)-C(4)-C(5)	113.2(2)
C(4)-C(5)-C(6)	103.1(2)
C(7)-C(6)-C(2)	110.5(2)
C(7)-C(6)-C(5)	112.8(2)
C(2)-C(6)-C(5)	106.5(2)
N(3)-C(7)-C(6)	110.1(2)
C(16)-C(8)-C(9)	121.1(2)
C(16)-C(8)-S(1)	120.29(18)
C(9)-C(8)-S(1)	118.50(17)
C(10)-C(9)-C(8)	119.5(2)
C(10)-C(9)-C(17)	118.8(2)
C(8)-C(9)-C(17)	121.5(2)
C(9)-C(10)-C(11)	119.1(2)
C(9)-C(10)-C(18)	122.4(2)
C(11)-C(10)-C(18)	118.5(2)
O(1)-C(11)-C(15)	123.6(2)
O(1)-C(11)-C(10)	114.3(2)
C(15)-C(11)-C(10)	122.1(2)

O(1)-C(12)-C(13)	107.6(2)
O(1)-C(12)-C(20)	108.7(2)
C(13)-C(12)-C(20)	113.2(3)
O(1)-C(12)-C(19)	103.6(3)
C(13)-C(12)-C(19)	112.9(3)
C(20)-C(12)-C(19)	110.2(3)
C(12)-C(13)-C(14)	112.0(2)
C(15)-C(14)-C(13)	110.8(3)
C(11)-C(15)-C(16)	119.4(2)
C(11)-C(15)-C(14)	119.4(2)
C(16)-C(15)-C(14)	121.2(2)
C(8)-C(16)-C(15)	118.2(2)
C(8)-C(16)-C(21)	123.8(2)
C(15)-C(16)-C(21)	117.8(2)
N(2)-C(22)-C(23)	108.61(19)
C(24)-C(23)-C(28)	118.6(2)
C(24)-C(23)-C(22)	120.8(2)
C(28)-C(23)-C(22)	120.6(2)
C(25)-C(24)-C(23)	120.7(3)
C(26)-C(25)-C(24)	120.2(3)
C(25)-C(26)-C(27)	120.0(3)
C(26)-C(27)-C(28)	120.2(3)
C(27)-C(28)-C(23)	120.3(3)

N(3)-C(29)-C(30)	110.82(19)
C(35)-C(30)-C(31)	118.3(2)
C(35)-C(30)-C(29)	120.9(2)
C(31)-C(30)-C(29)	120.8(2)
C(32)-C(31)-C(30)	120.6(2)
C(31)-C(32)-C(33)	120.2(3)
C(34)-C(33)-C(32)	120.0(3)
C(33)-C(34)-C(35)	119.7(2)
C(30)-C(35)-C(34)	121.2(3)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **21**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	39(1)	36(1)	31(1)	15(1)	19(1)	19(1)
O(1)	45(1)	27(1)	71(1)	18(1)	24(1)	14(1)
O(2)	50(1)	40(1)	75(2)	31(1)	40(1)	20(1)
O(3)	73(1)	63(1)	23(1)	14(1)	19(1)	40(1)
N(1)	30(1)	40(1)	21(1)	6(1)	8(1)	16(1)
N(2)	27(1)	27(1)	26(1)	8(1)	1(1)	2(1)
N(3)	34(1)	28(1)	26(1)	7(1)	10(1)	5(1)
C(1)	31(1)	21(1)	22(1)	1(1)	6(1)	7(1)
C(2)	27(1)	32(1)	32(1)	9(1)	-3(1)	1(1)
C(3)	36(2)	42(2)	28(1)	3(1)	1(1)	5(1)
C(4)	41(2)	32(2)	40(2)	2(1)	2(1)	0(1)
C(5)	53(2)	47(2)	44(2)	-1(1)	7(1)	-19(2)
C(6)	27(1)	40(2)	39(2)	2(1)	7(1)	8(1)
C(7)	26(1)	43(2)	39(2)	5(1)	12(1)	1(1)
C(8)	23(1)	26(1)	26(1)	8(1)	11(1)	6(1)
C(9)	24(1)	23(1)	28(1)	2(1)	9(1)	3(1)
C(10)	26(1)	29(1)	30(1)	5(1)	7(1)	6(1)
C(11)	31(1)	25(1)	42(2)	12(1)	17(1)	9(1)

C(12)	54(2)	25(1)	83(2)	15(2)	39(2)	10(1)
C(13)	75(2)	23(1)	81(2)	-2(2)	51(2)	-5(2)
C(14)	55(2)	31(2)	58(2)	-6(1)	26(2)	-14(1)
C(15)	35(1)	22(1)	40(2)	-1(1)	18(1)	-4(1)
C(16)	27(1)	33(1)	30(1)	3(1)	11(1)	-1(1)
C(17)	31(1)	28(1)	38(2)	-2(1)	11(1)	0(1)
C(18)	43(2)	42(2)	46(2)	9(1)	-3(1)	15(1)
C(19)	69(2)	39(2)	139(4)	43(2)	66(2)	30(2)
C(20)	60(2)	42(2)	83(2)	25(2)	41(2)	17(2)
C(21)	31(2)	64(2)	42(2)	5(2)	4(1)	-8(1)
C(22)	32(1)	35(1)	27(1)	11(1)	10(1)	7(1)
C(23)	24(1)	30(1)	33(1)	12(1)	9(1)	1(1)
C(24)	27(1)	34(1)	40(2)	15(1)	4(1)	-4(1)
C(25)	31(2)	39(2)	63(2)	25(2)	-2(1)	-1(1)
C(26)	37(2)	31(2)	87(3)	19(2)	14(2)	5(1)
C(27)	49(2)	32(2)	64(2)	2(1)	25(2)	1(1)
C(28)	37(2)	35(2)	39(2)	9(1)	12(1)	2(1)
C(29)	44(2)	28(1)	29(1)	10(1)	14(1)	8(1)
C(30)	35(1)	29(1)	23(1)	9(1)	7(1)	7(1)
C(31)	38(2)	41(2)	33(1)	4(1)	17(1)	6(1)
C(32)	42(2)	39(2)	44(2)	-1(1)	16(1)	-1(1)
C(33)	45(2)	38(2)	36(2)	-4(1)	11(1)	10(1)
C(34)	40(2)	50(2)	33(2)	4(1)	17(1)	7(1)

C(35)	40(2)	39(2)	34(1)	10(1)	15(1)	2(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **21**.

	x	y	z	U(eq)
H(2A)	8556	-1015	4276	39
H(3A)	7844	938	5224	45
H(4A)	8936	2693	4913	49
H(5A)	10219	1740	3780	62
H(5B)	8595	1899	3219	62
H(6A)	9842	-392	3505	45
H(7A)	8545	-115	2040	43
H(7B)	8050	-1412	2244	43
H(13A)	1203	6054	3524	67
H(13B)	2355	7046	3434	67
H(14A)	3966	5502	3279	58
H(14B)	3391	5288	4087	58
H(17A)	741	113	701	50
H(17B)	319	-360	1482	50
H(17C)	1915	-492	1389	50
H(18A)	-1033	1406	817	71

H(18B)	-269	2310	385	71
H(18C)	-1174	2924	1058	71
H(19A)	-725	6631	2329	107
H(19B)	-572	6435	1337	107
H(19C)	292	7619	2118	107
H(20A)	3021	5454	1807	83
H(20B)	2628	6907	1828	83
H(20C)	1722	5762	1029	83
H(21A)	5409	2385	3741	72
H(21B)	4717	3279	4445	72
H(21C)	5296	3880	3768	72
H(22A)	4840	-1845	3560	37
H(22B)	5933	-1236	4532	37
H(24A)	7361	-2719	5198	41
H(25A)	8619	-4569	5124	55
H(26A)	8666	-5835	3744	62
H(27A)	7430	-5271	2426	58
H(28A)	6144	-3430	2488	44
H(29A)	6456	777	1240	39
H(29B)	4925	476	1375	39
H(31A)	3923	-1864	894	44
H(32A)	3685	-3812	-155	52
H(33A)	5271	-4242	-1029	50

H(34A)	7075	-2708	-869	49
H(35A)	7332	-761	194	44

Table 6. Torsion angles [°] for **21**.

O(2)-S(1)-N(1)-C(1)	-135.5(2)
O(3)-S(1)-N(1)-C(1)	-5.4(2)
C(8)-S(1)-N(1)-C(1)	111.8(2)
S(1)-N(1)-C(1)-N(3)	-133.5(2)
S(1)-N(1)-C(1)-N(2)	51.2(3)
C(7)-N(3)-C(1)-N(1)	-159.8(2)
C(29)-N(3)-C(1)-N(1)	-6.9(3)
C(7)-N(3)-C(1)-N(2)	15.9(3)
C(29)-N(3)-C(1)-N(2)	168.8(2)
C(22)-N(2)-C(1)-N(1)	21.3(3)
C(2)-N(2)-C(1)-N(1)	-157.0(2)
C(22)-N(2)-C(1)-N(3)	-154.1(2)
C(2)-N(2)-C(1)-N(3)	27.6(3)
C(1)-N(2)-C(2)-C(3)	89.5(3)
C(22)-N(2)-C(2)-C(3)	-88.9(3)
C(1)-N(2)-C(2)-C(6)	-27.9(3)
C(22)-N(2)-C(2)-C(6)	153.7(2)
N(2)-C(2)-C(3)-C(4)	-115.4(2)
C(6)-C(2)-C(3)-C(4)	7.2(3)
C(2)-C(3)-C(4)-C(5)	0.1(3)
C(3)-C(4)-C(5)-C(6)	-7.2(3)

N(2)-C(2)-C(6)-C(7)	-12.7(3)
C(3)-C(2)-C(6)-C(7)	-134.1(2)
N(2)-C(2)-C(6)-C(5)	110.0(2)
C(3)-C(2)-C(6)-C(5)	-11.3(3)
C(4)-C(5)-C(6)-C(7)	132.5(2)
C(4)-C(5)-C(6)-C(2)	11.2(3)
C(1)-N(3)-C(7)-C(6)	-55.7(3)
C(29)-N(3)-C(7)-C(6)	150.7(2)
C(2)-C(6)-C(7)-N(3)	50.4(3)
C(5)-C(6)-C(7)-N(3)	-68.7(3)
O(2)-S(1)-C(8)-C(16)	141.66(19)
O(3)-S(1)-C(8)-C(16)	17.5(2)
N(1)-S(1)-C(8)-C(16)	-103.6(2)
O(2)-S(1)-C(8)-C(9)	-34.3(2)
O(3)-S(1)-C(8)-C(9)	-158.45(18)
N(1)-S(1)-C(8)-C(9)	80.4(2)
C(16)-C(8)-C(9)-C(10)	-8.7(3)
S(1)-C(8)-C(9)-C(10)	167.22(18)
C(16)-C(8)-C(9)-C(17)	165.7(2)
S(1)-C(8)-C(9)-C(17)	-18.3(3)
C(8)-C(9)-C(10)-C(11)	3.3(3)
C(17)-C(9)-C(10)-C(11)	-171.3(2)
C(8)-C(9)-C(10)-C(18)	-177.7(2)

C(17)-C(9)-C(10)-C(18)	7.7(4)
C(12)-O(1)-C(11)-C(15)	15.8(4)
C(12)-O(1)-C(11)-C(10)	-162.7(2)
C(9)-C(10)-C(11)-O(1)	179.7(2)
C(18)-C(10)-C(11)-O(1)	0.6(3)
C(9)-C(10)-C(11)-C(15)	1.3(4)
C(18)-C(10)-C(11)-C(15)	-177.8(2)
C(11)-O(1)-C(12)-C(13)	-46.1(3)
C(11)-O(1)-C(12)-C(20)	76.8(3)
C(11)-O(1)-C(12)-C(19)	-166.0(2)
O(1)-C(12)-C(13)-C(14)	61.4(3)
C(20)-C(12)-C(13)-C(14)	-58.8(3)
C(19)-C(12)-C(13)-C(14)	175.2(2)
C(12)-C(13)-C(14)-C(15)	-45.3(3)
O(1)-C(11)-C(15)-C(16)	-178.8(2)
C(10)-C(11)-C(15)-C(16)	-0.5(4)
O(1)-C(11)-C(15)-C(14)	1.9(4)
C(10)-C(11)-C(15)-C(14)	-179.8(2)
C(13)-C(14)-C(15)-C(11)	13.7(4)
C(13)-C(14)-C(15)-C(16)	-165.6(2)
C(9)-C(8)-C(16)-C(15)	9.4(3)
S(1)-C(8)-C(16)-C(15)	-166.52(18)
C(9)-C(8)-C(16)-C(21)	-165.9(2)

S(1)-C(8)-C(16)-C(21)	18.2(3)
C(11)-C(15)-C(16)-C(8)	-4.7(3)
C(14)-C(15)-C(16)-C(8)	174.6(2)
C(11)-C(15)-C(16)-C(21)	170.9(2)
C(14)-C(15)-C(16)-C(21)	-9.9(4)
C(1)-N(2)-C(22)-C(23)	117.7(2)
C(2)-N(2)-C(22)-C(23)	-63.9(3)
N(2)-C(22)-C(23)-C(24)	117.0(2)
N(2)-C(22)-C(23)-C(28)	-61.8(3)
C(28)-C(23)-C(24)-C(25)	1.0(4)
C(22)-C(23)-C(24)-C(25)	-177.9(2)
C(23)-C(24)-C(25)-C(26)	-0.1(4)
C(24)-C(25)-C(26)-C(27)	-0.5(4)
C(25)-C(26)-C(27)-C(28)	0.2(4)
C(26)-C(27)-C(28)-C(23)	0.8(4)
C(24)-C(23)-C(28)-C(27)	-1.3(4)
C(22)-C(23)-C(28)-C(27)	177.5(2)
C(1)-N(3)-C(29)-C(30)	-88.9(3)
C(7)-N(3)-C(29)-C(30)	64.1(3)
N(3)-C(29)-C(30)-C(35)	-100.4(3)
N(3)-C(29)-C(30)-C(31)	78.0(3)
C(35)-C(30)-C(31)-C(32)	1.1(4)
C(29)-C(30)-C(31)-C(32)	-177.3(2)

C(30)-C(31)-C(32)-C(33)	-0.5(4)
C(31)-C(32)-C(33)-C(34)	-0.6(4)
C(32)-C(33)-C(34)-C(35)	0.9(4)
C(31)-C(30)-C(35)-C(34)	-0.8(4)
C(29)-C(30)-C(35)-C(34)	177.6(2)
C(33)-C(34)-C(35)-C(30)	-0.2(4)