Convenient syntheses of 2-acylamino-4-halothiazoles and acylated derivatives using a versatile Boc-intermediate

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Department of Chemistry, University of Liverpool, Liverpool L69 7ZD, U.K.

Supporting Information

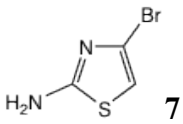
1. Photocopy 1H and 13C NMR spectra for compounds described,

2. Full Cif crystallographic data files for **13, 15** and **19**.

*

* To whom correspondence should be addressed: stachuls@liv.ac.uk or 0151-794-3482

1. Photocopy 1H (400 MHz) and 13C (100 MHz) NMR spectra for compounds described



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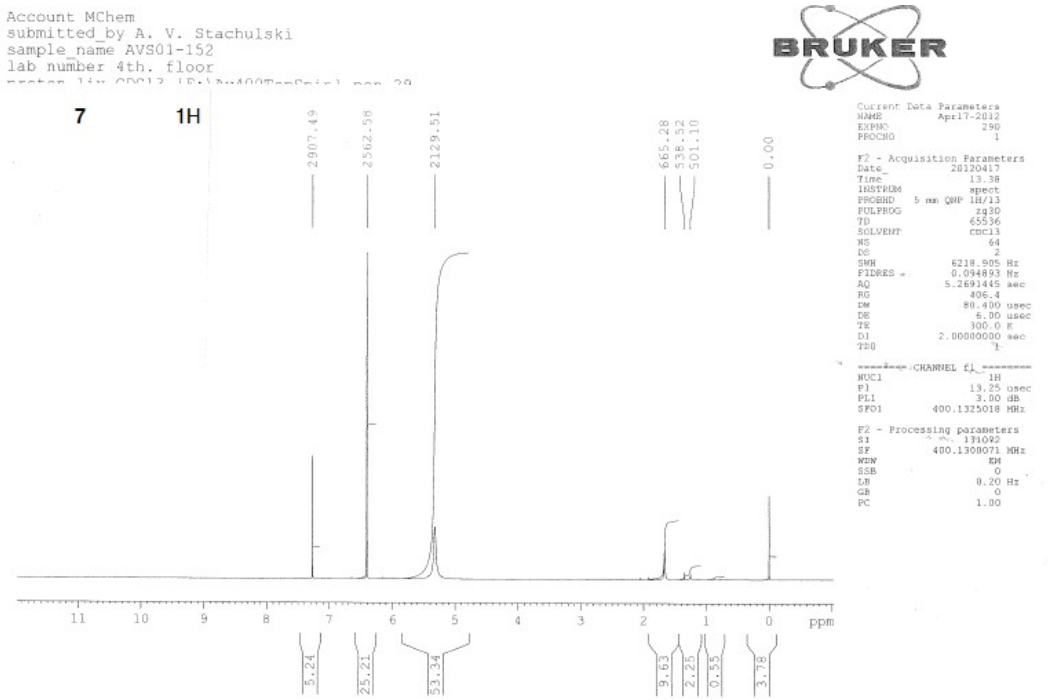

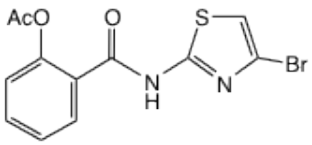

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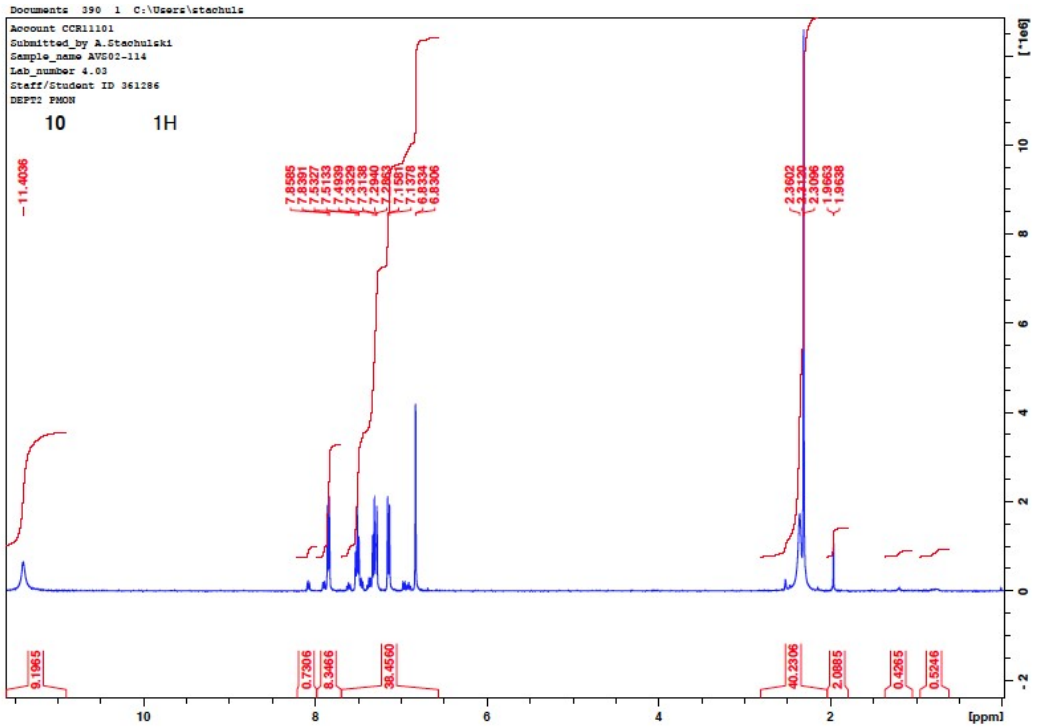

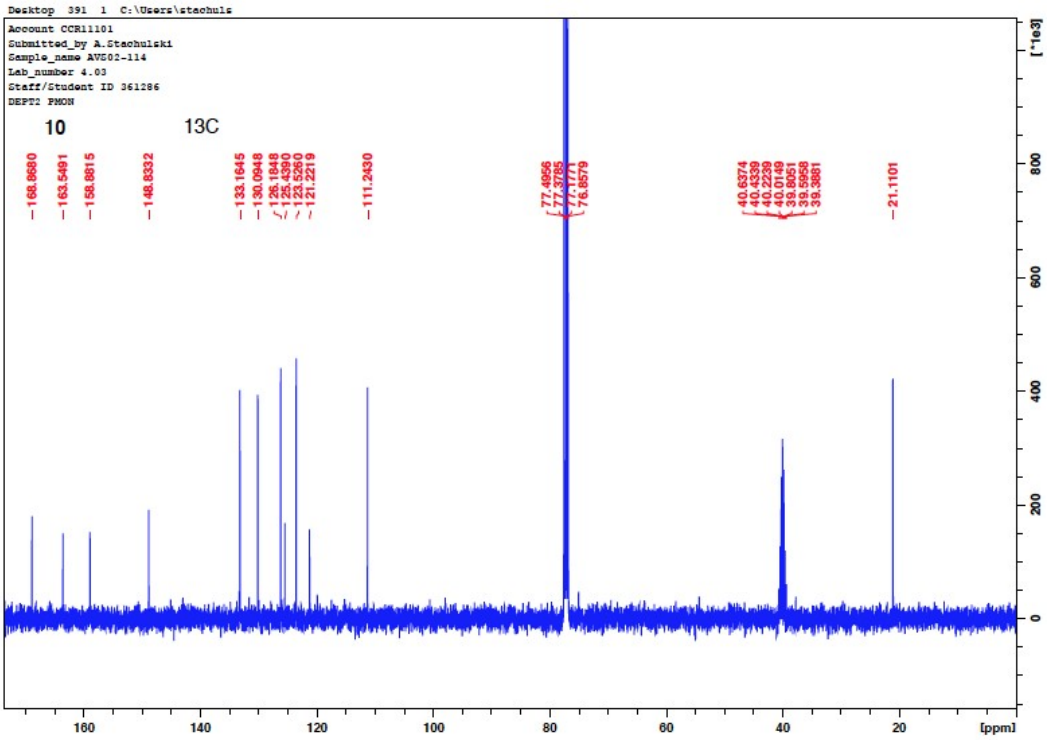

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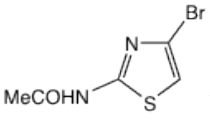

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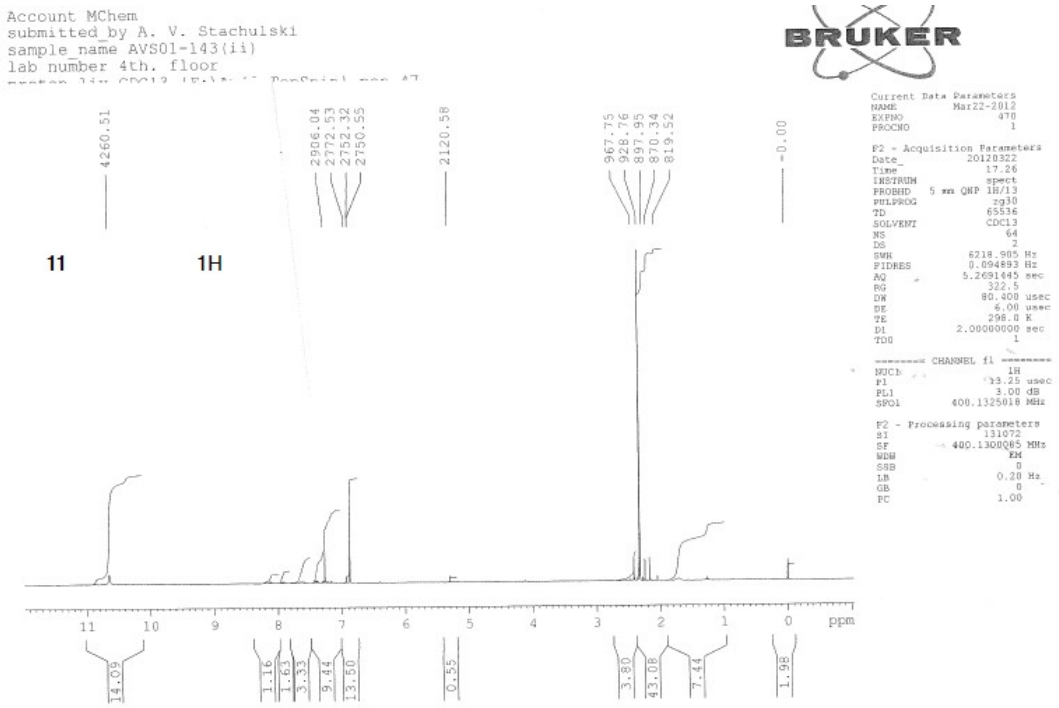

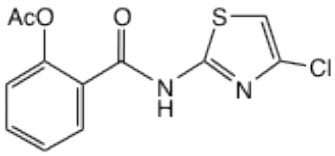
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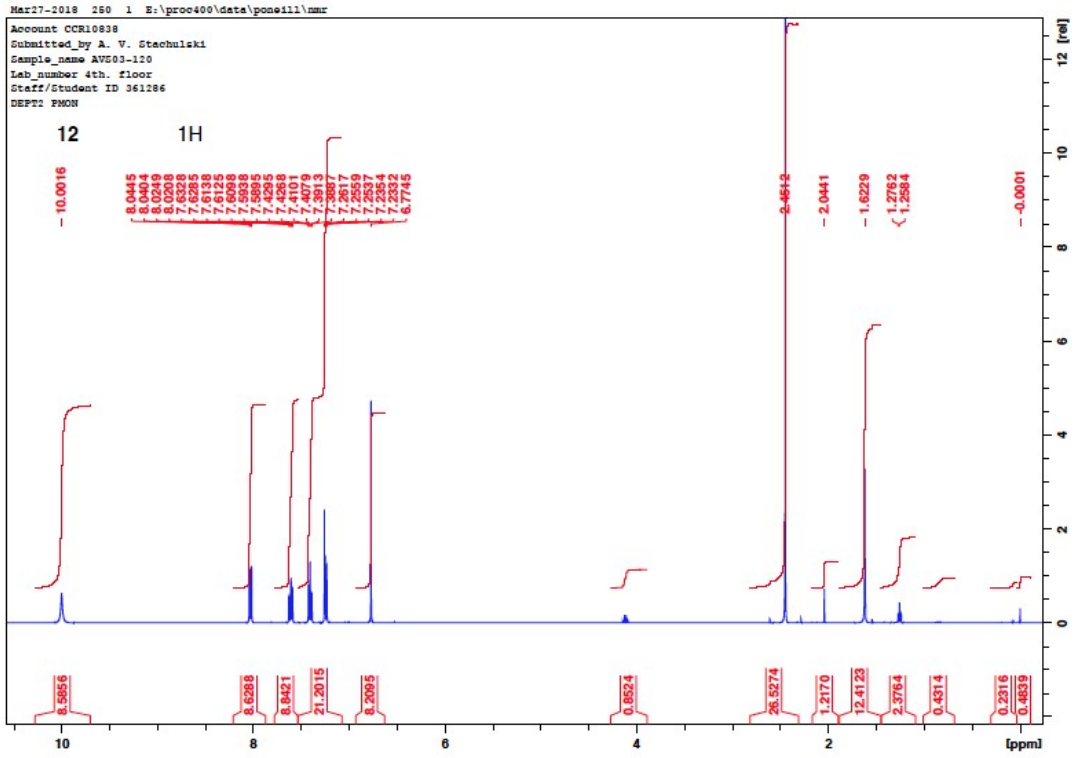
S
N
H
N
Cl

12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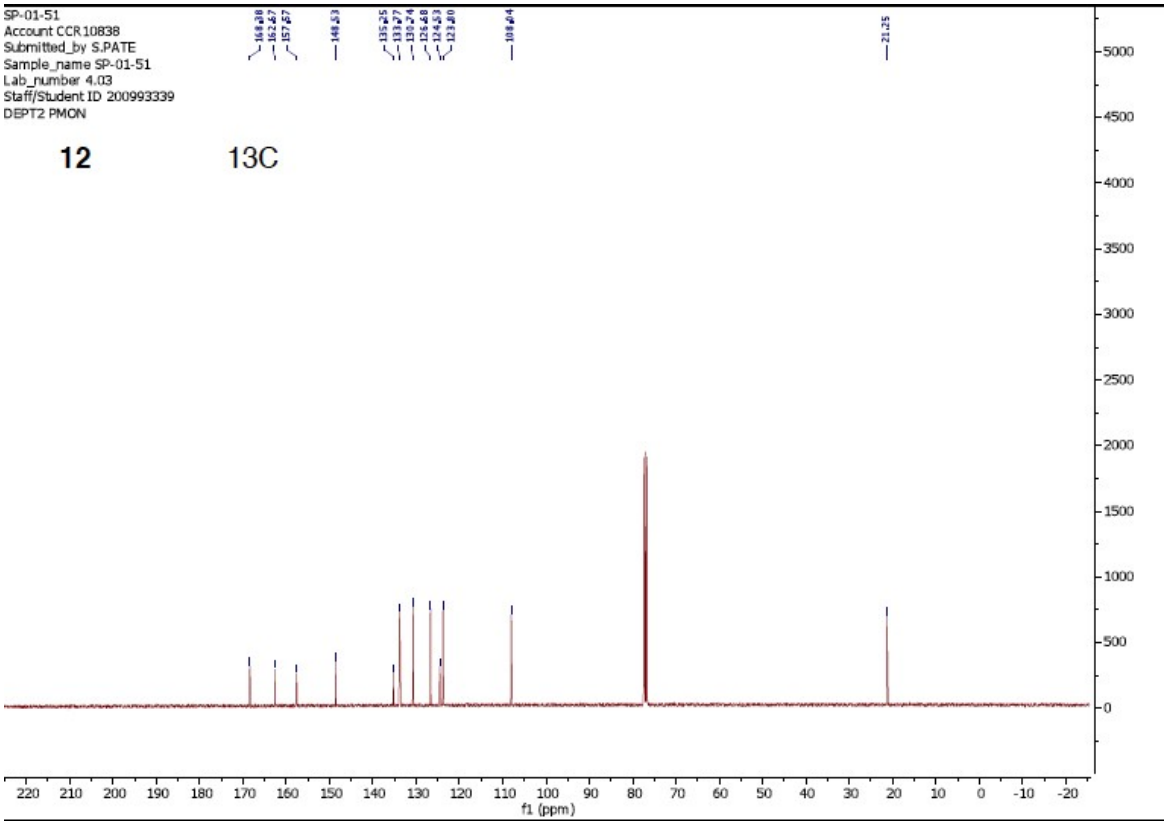
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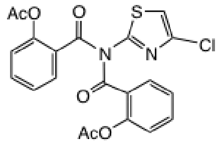
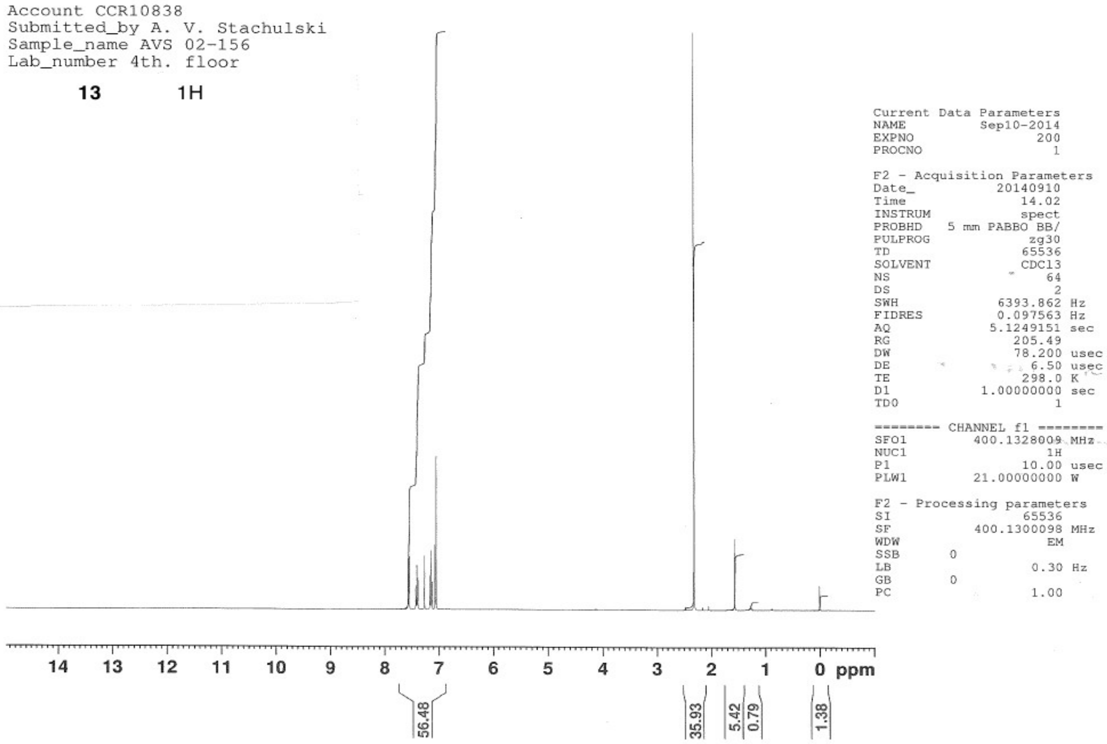


AcO
O
S
N
Cl
N
O
AcO

13



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NUC1 1H
P1 10.00 usec
PLW1 21.00000000 W
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SSB 0
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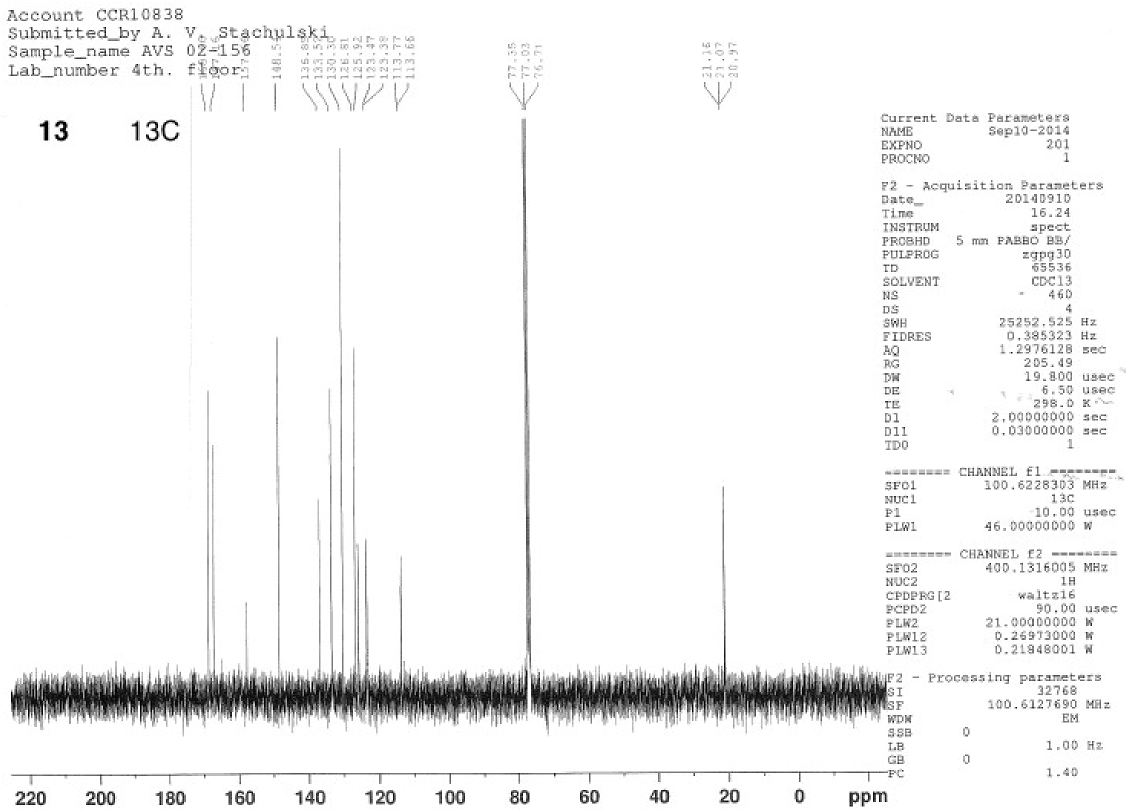
1.38



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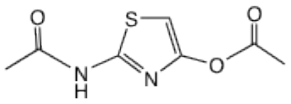

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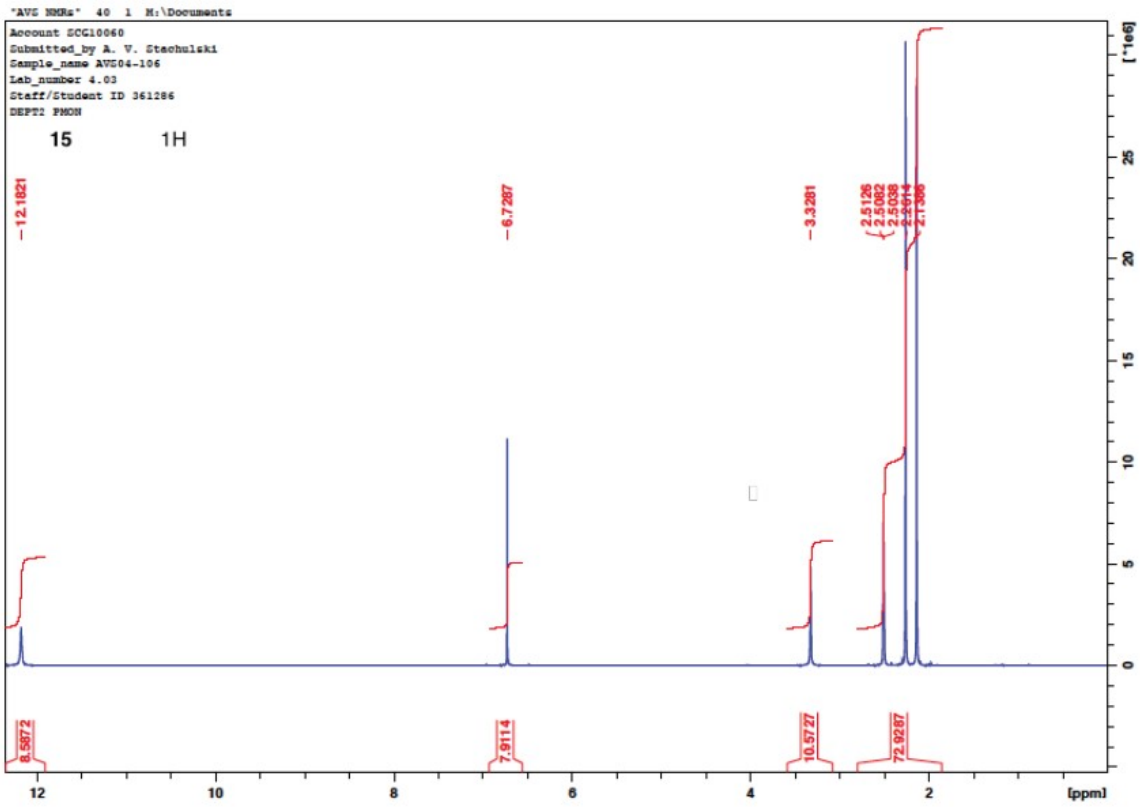
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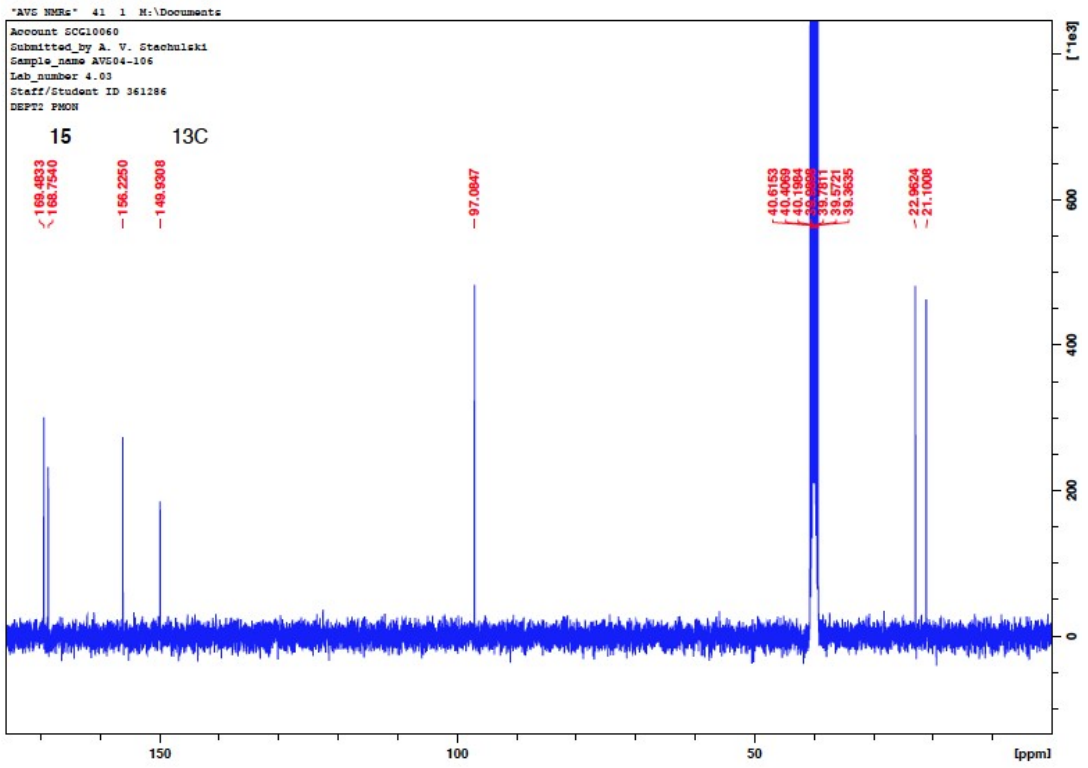
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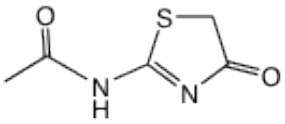

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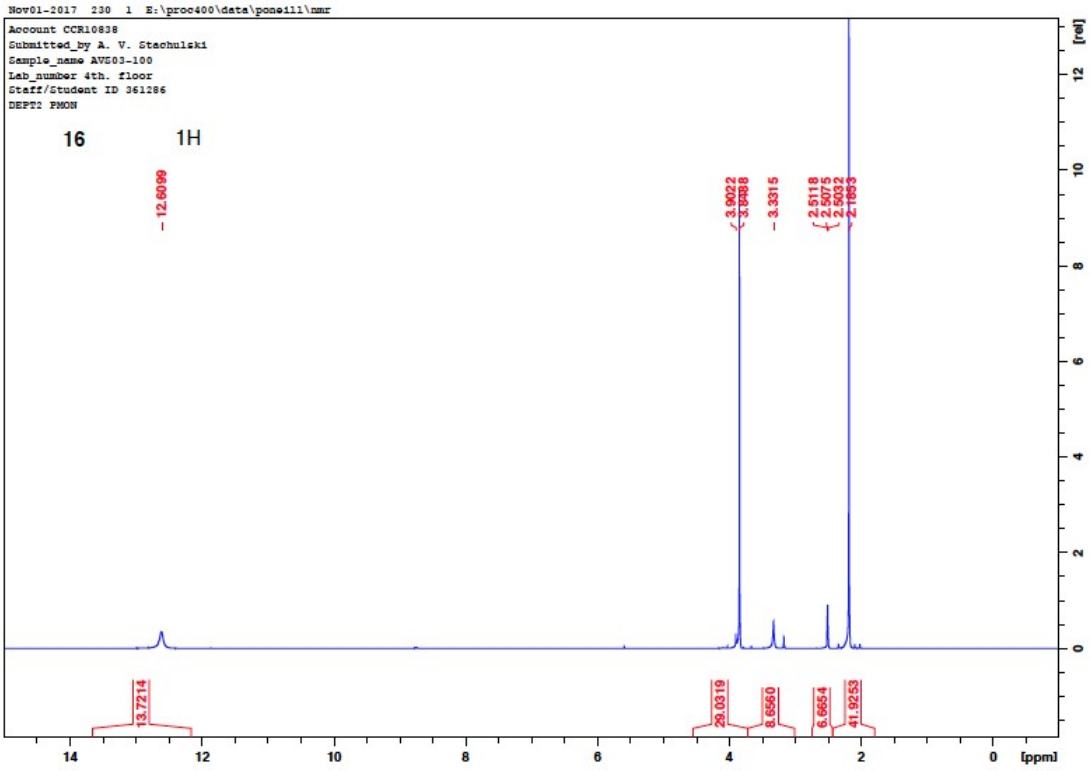
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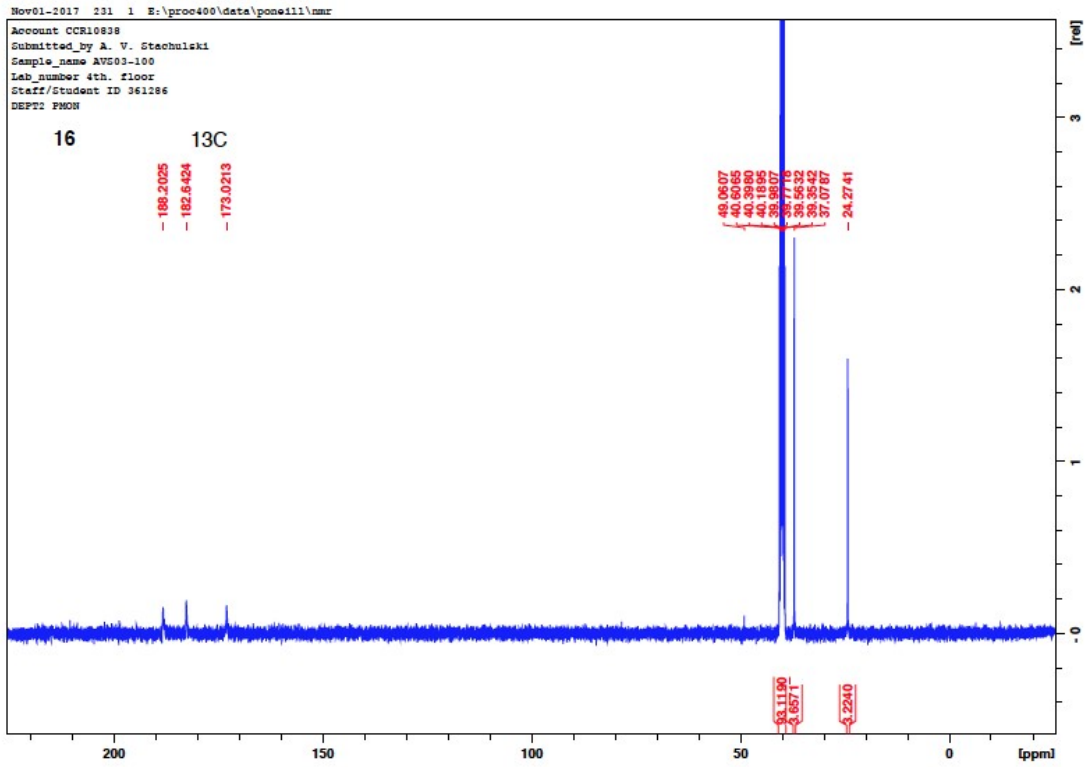
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13C
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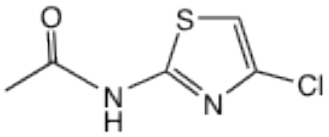
O
S
N
H
N
Cl

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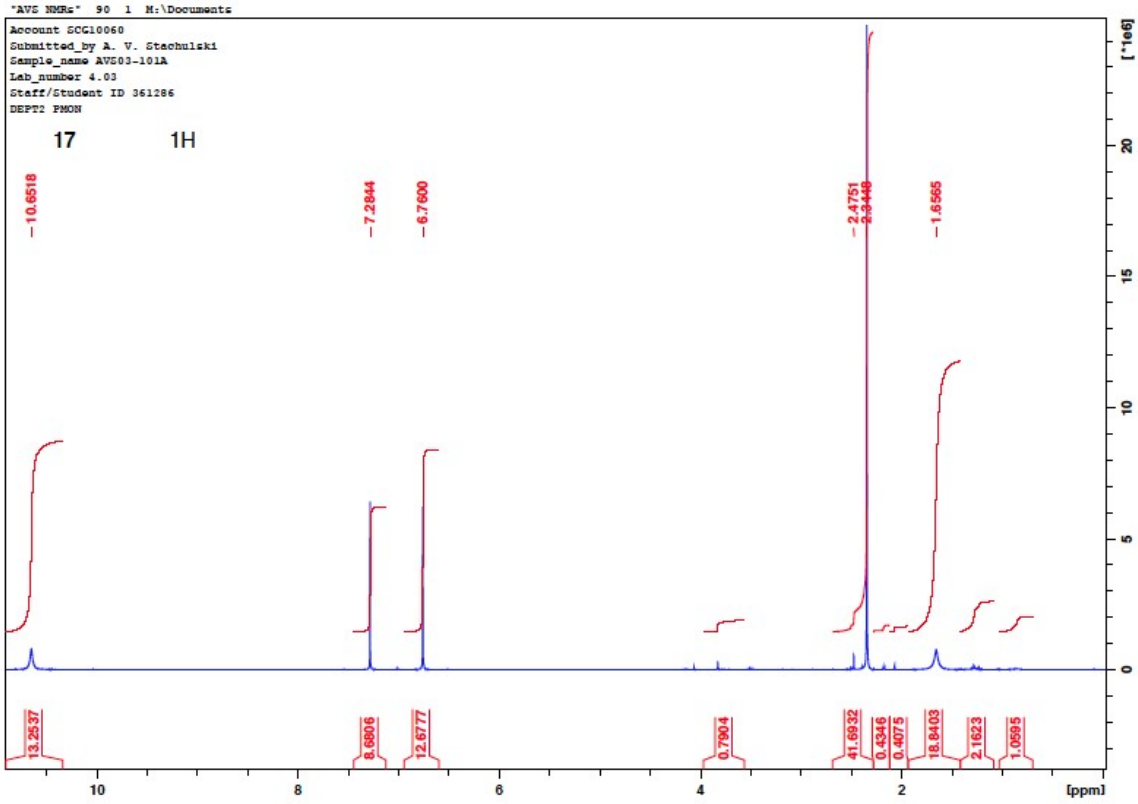
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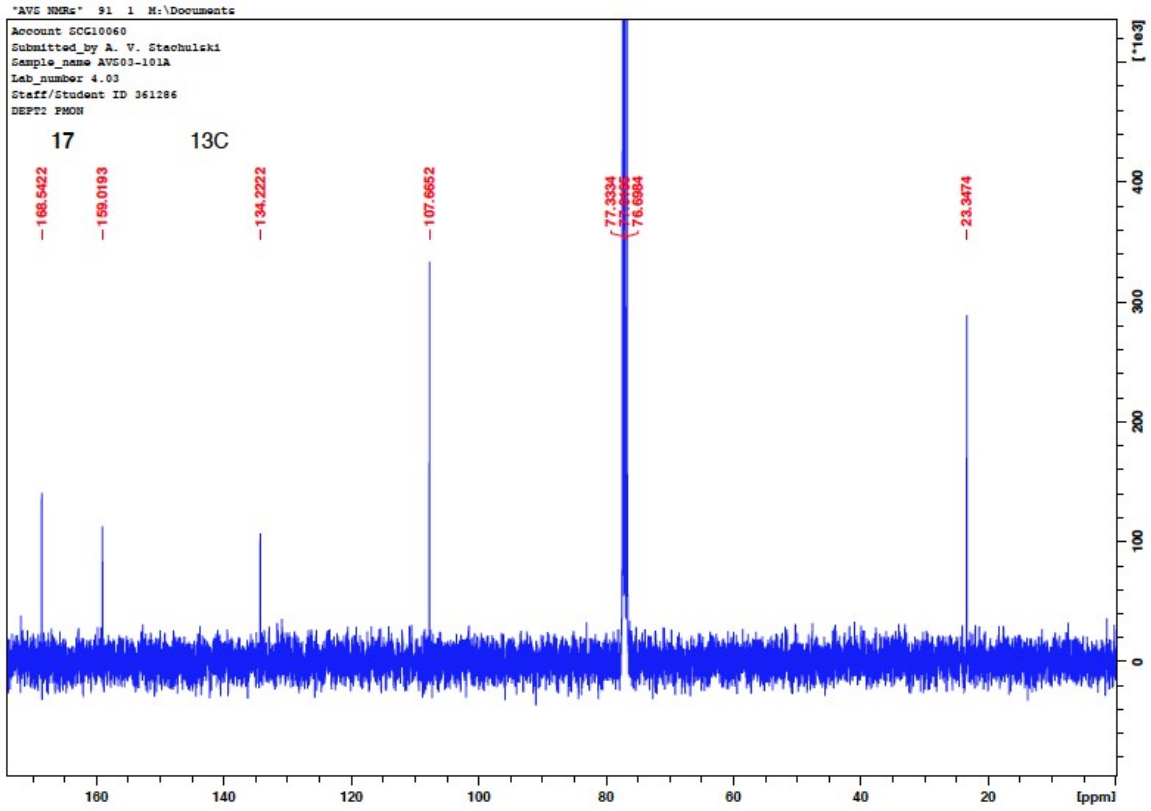
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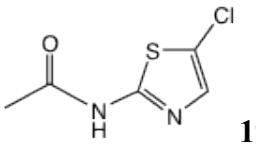
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S
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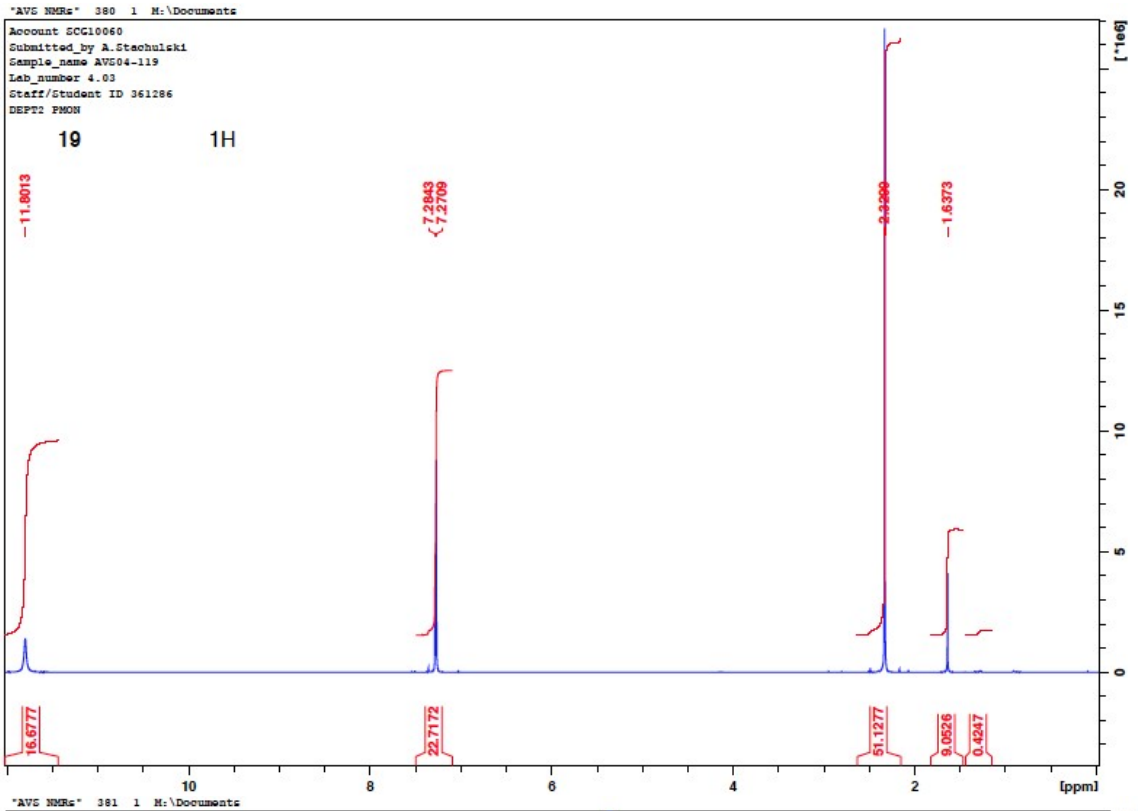
N
H
N

19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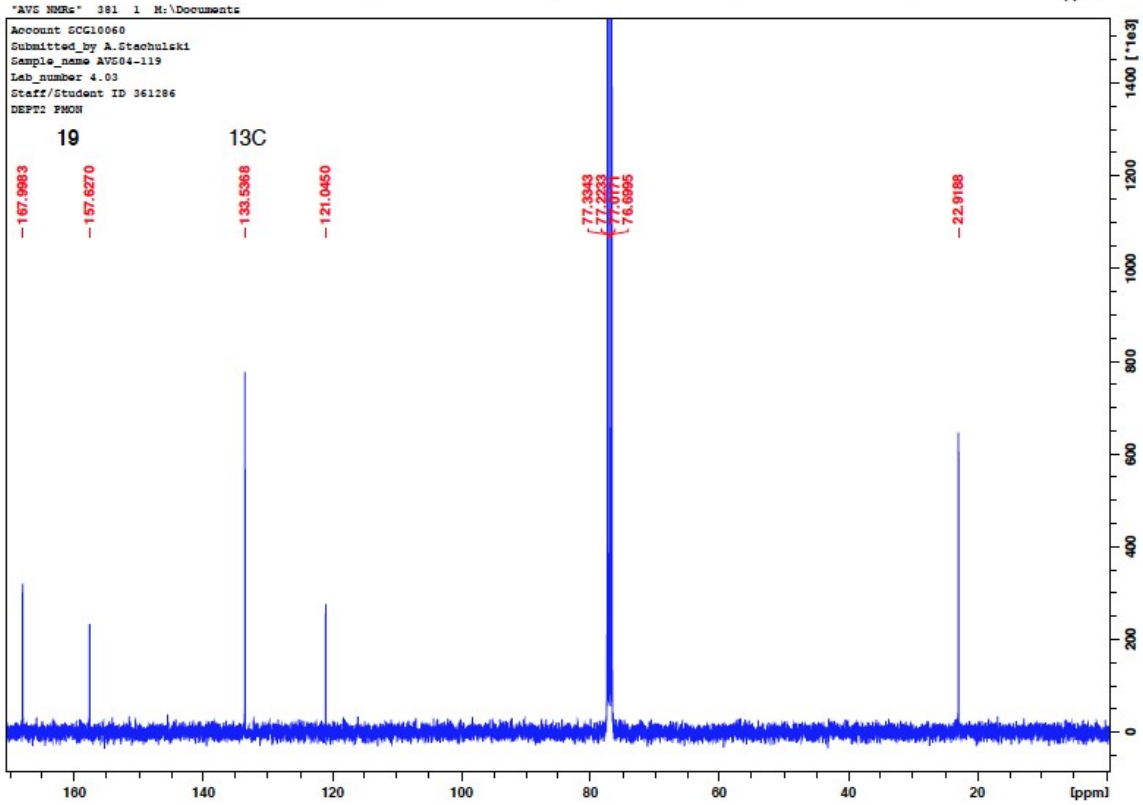
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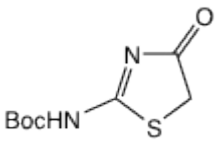
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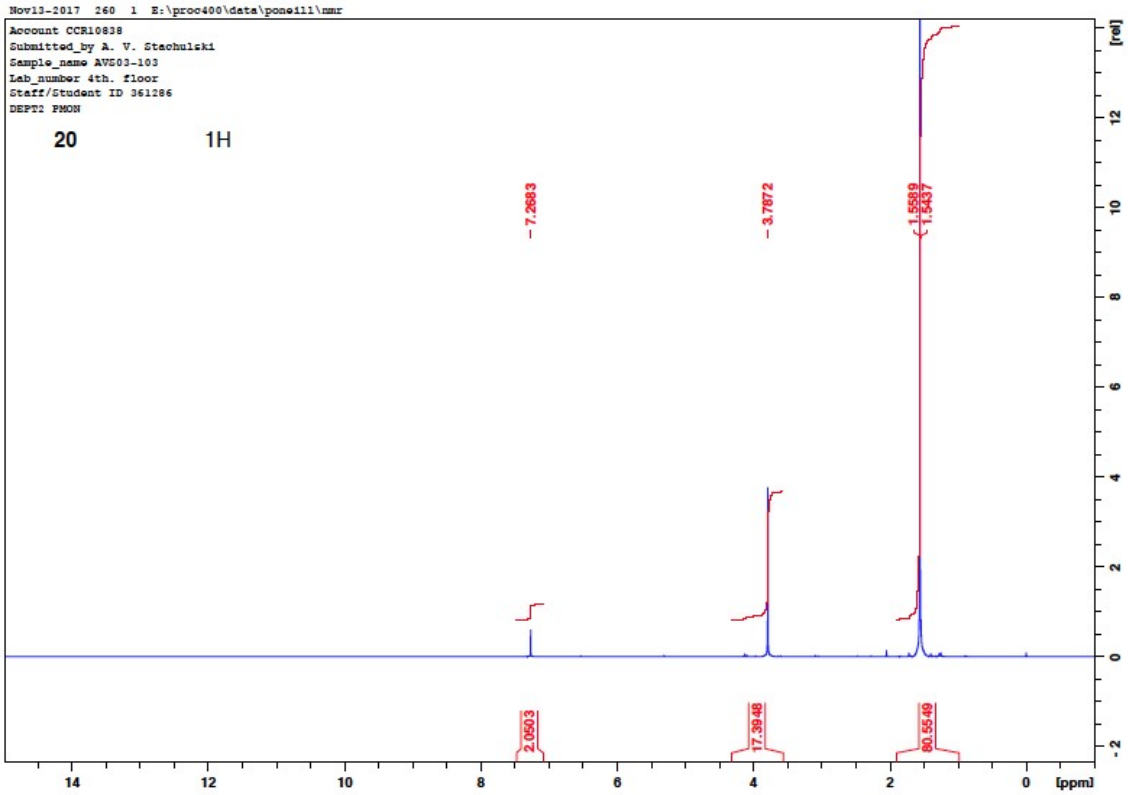
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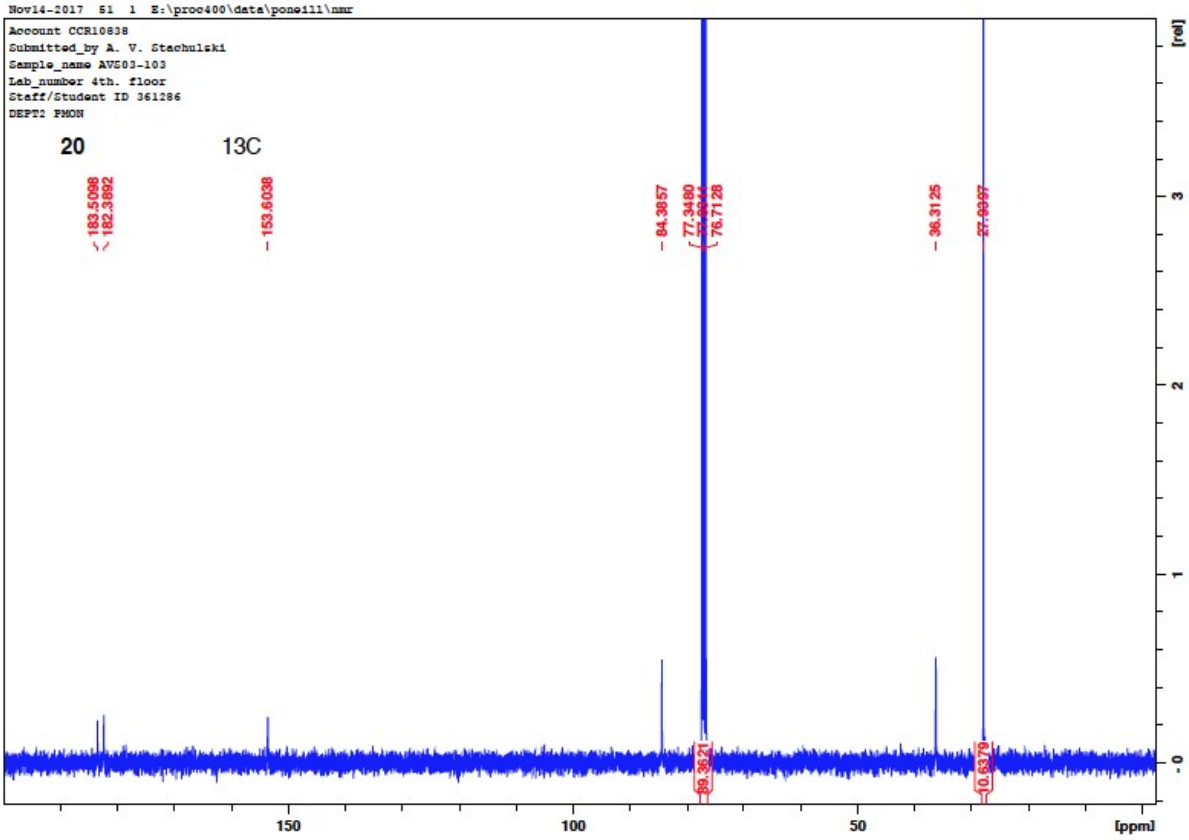
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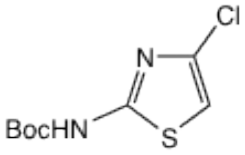
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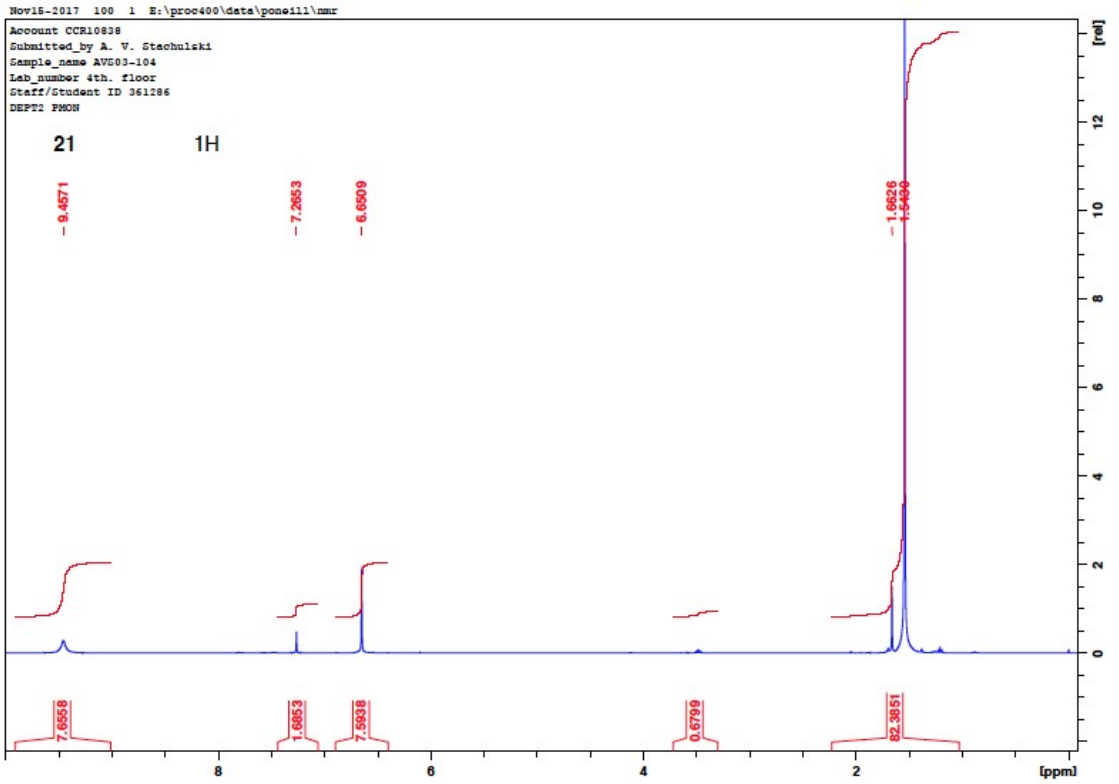
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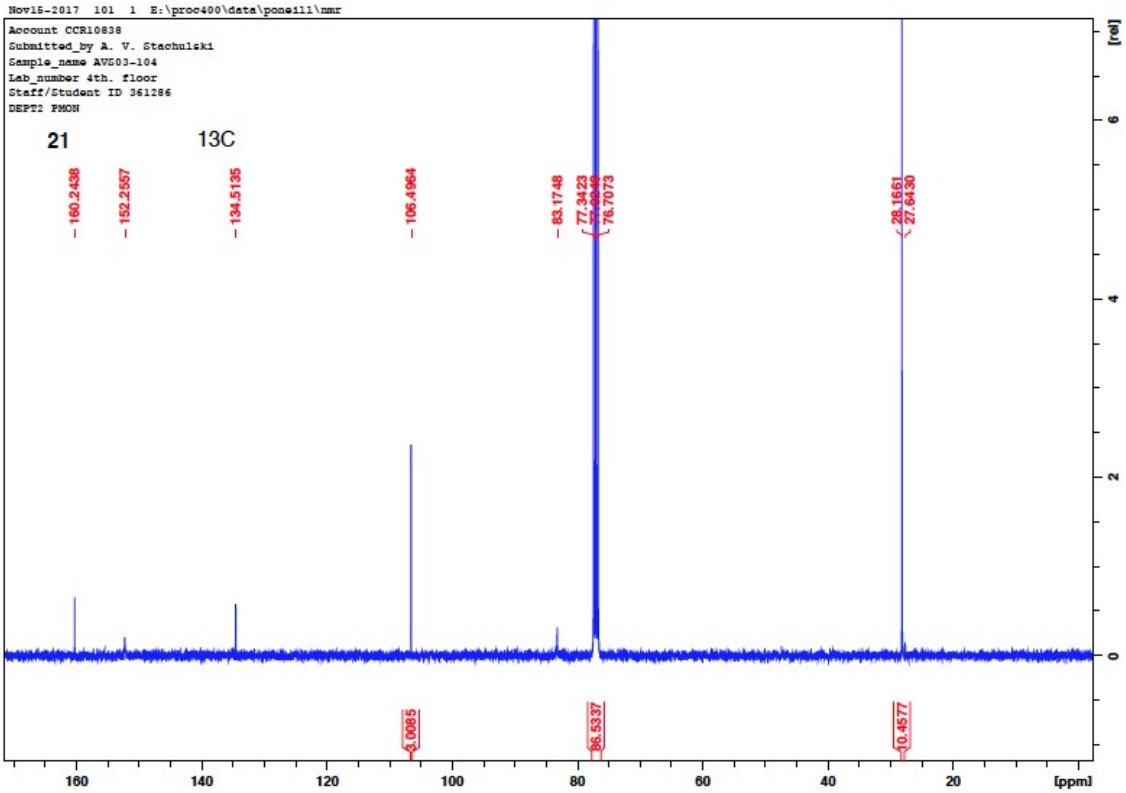
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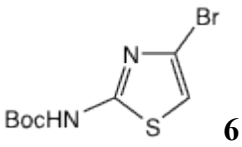
Br
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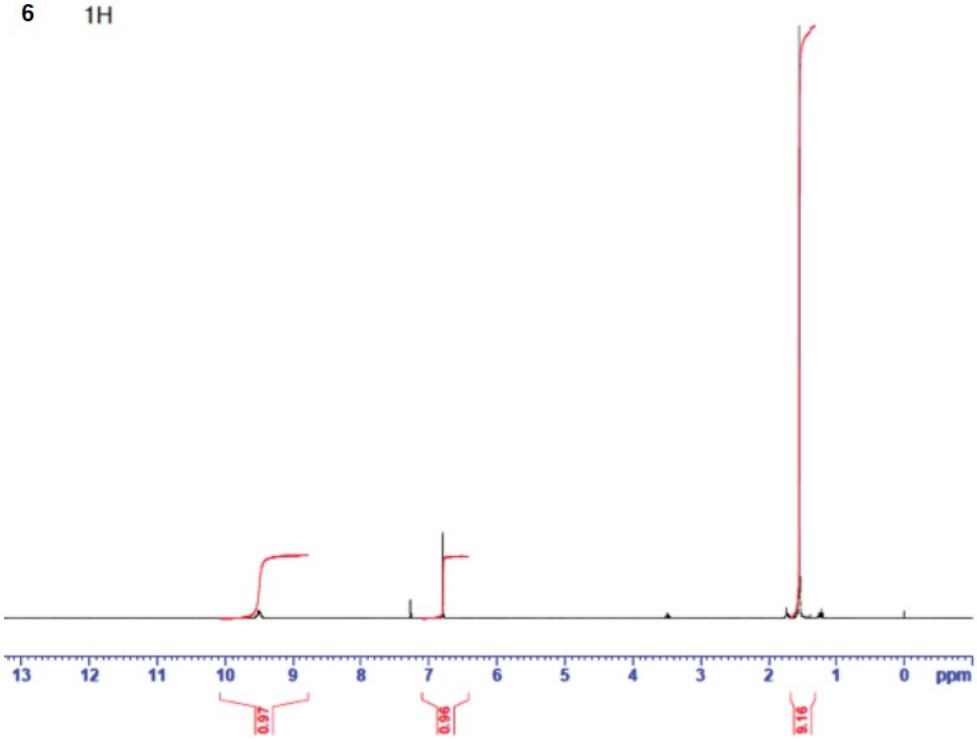
BocHN
S

6

6 1H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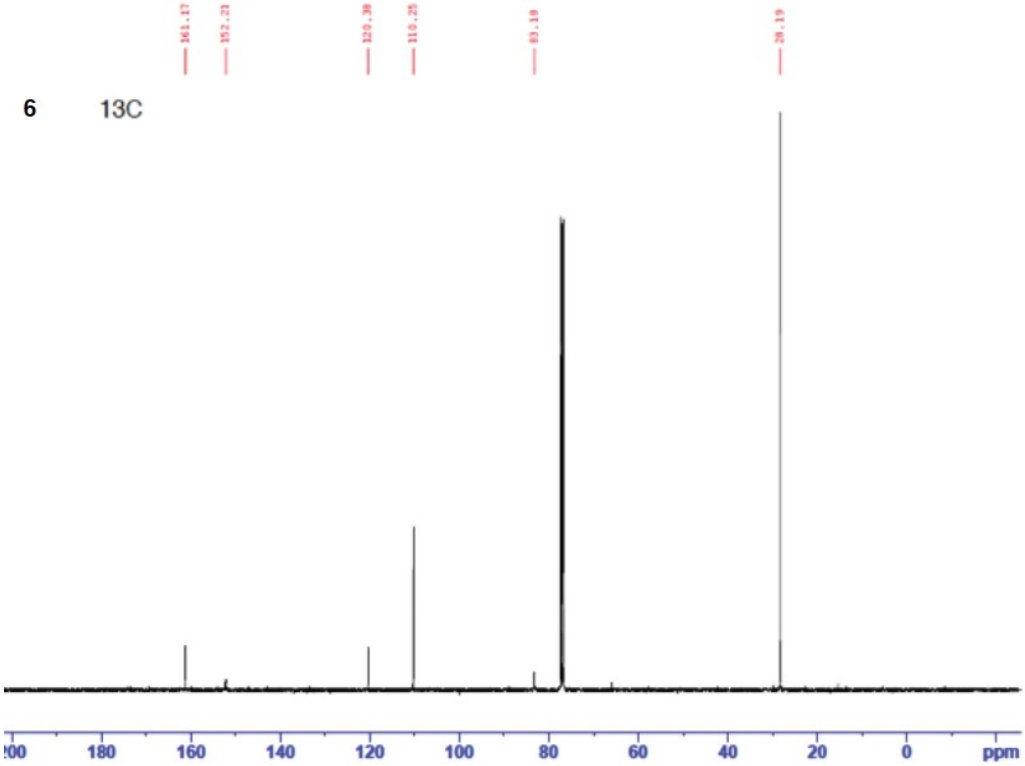
6 13C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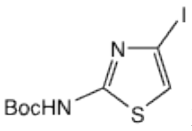
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BocHN
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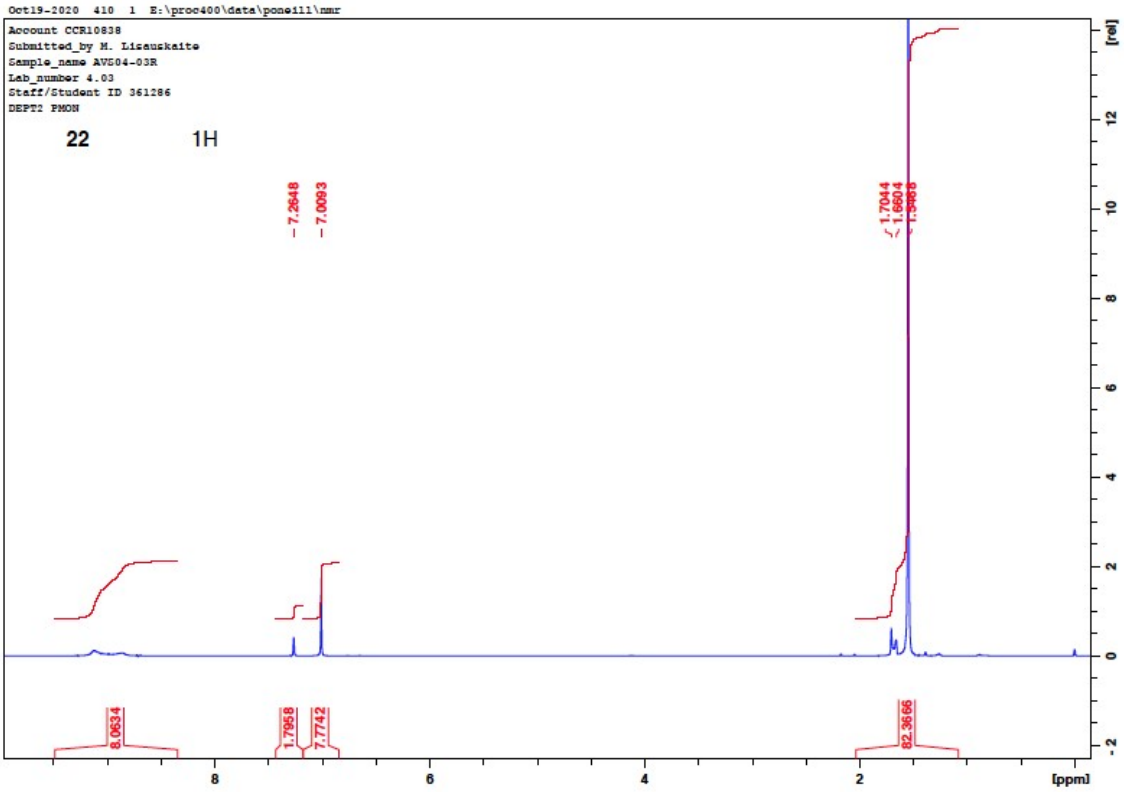
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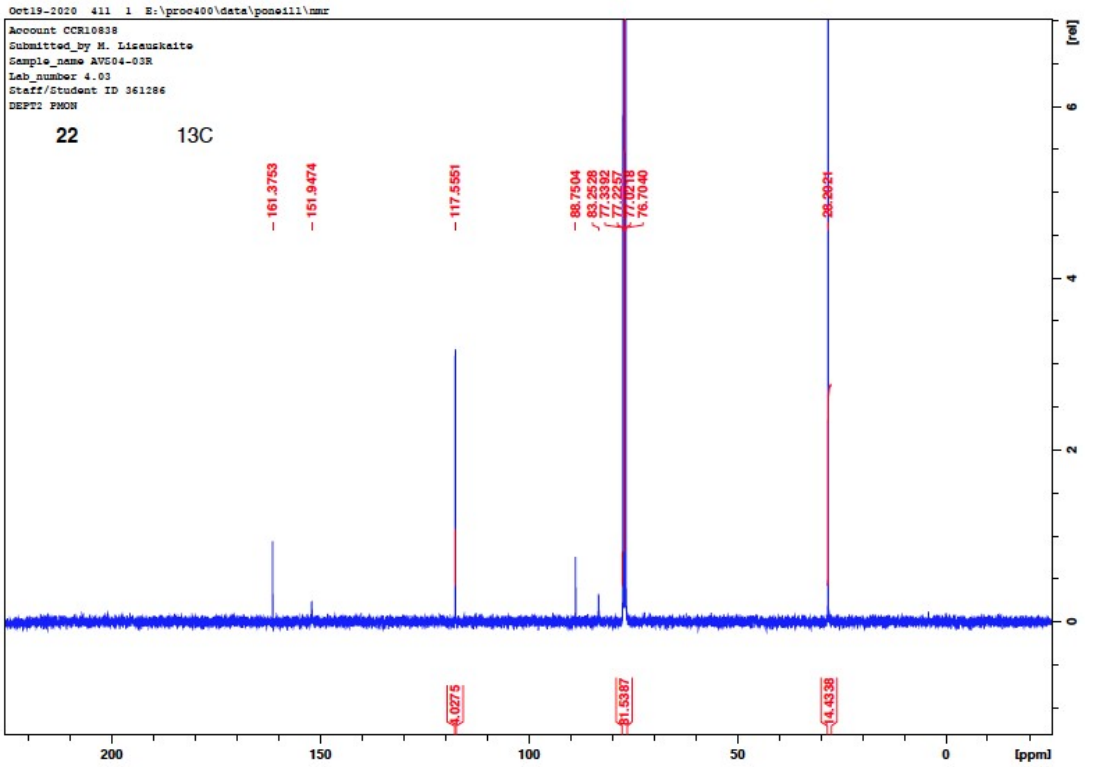
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Submitted_by M. Lisauskaite
Sample_name AVS04-03R
Lab_number 4.03
Staff/Student ID 361286
DEPT2 PMON
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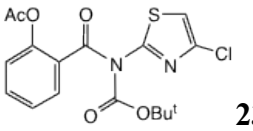
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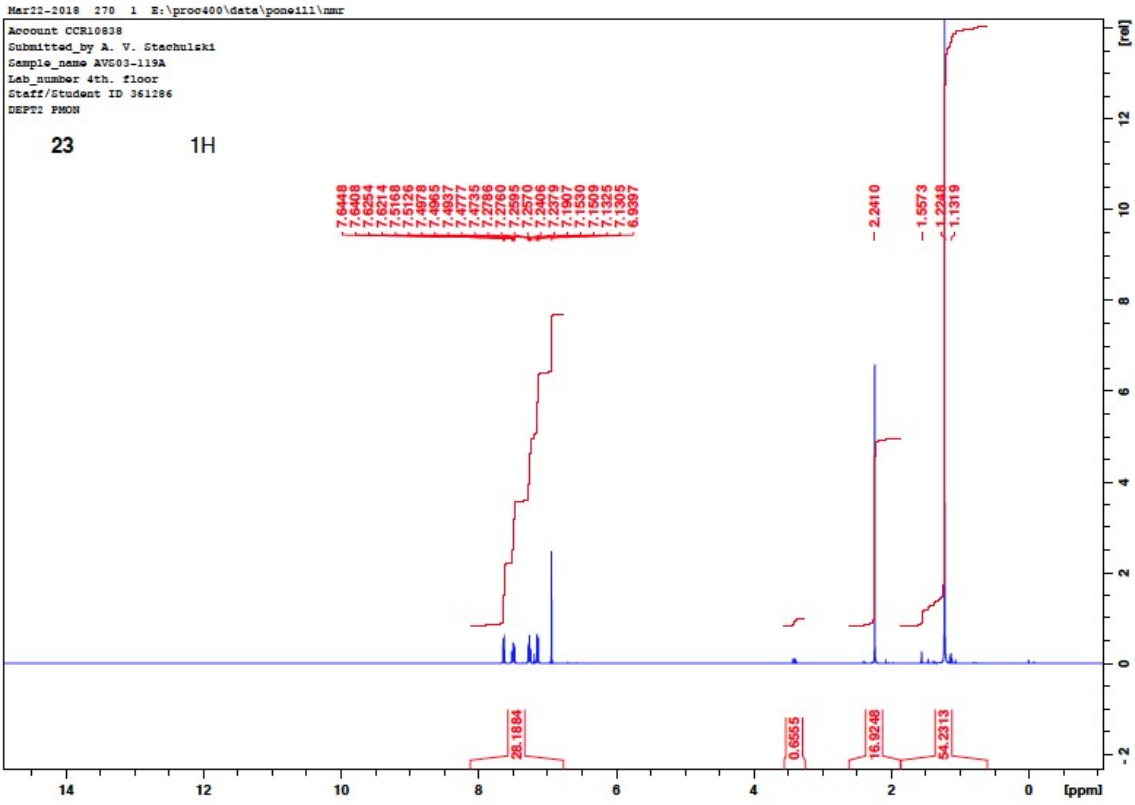
N
Cl
O
OBu^t

23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Submitted_by A. V. Stachulski
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Lab_number 4th. floor
Staff/Student ID 361286
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7.5168
7.5126
7.4978
7.4965
7.4937
7.4777
7.4735
7.2786
7.2760
7.2595
7.2570
7.2406
7.2379
7.1907
7.1530
7.1509
7.1325
7.1305
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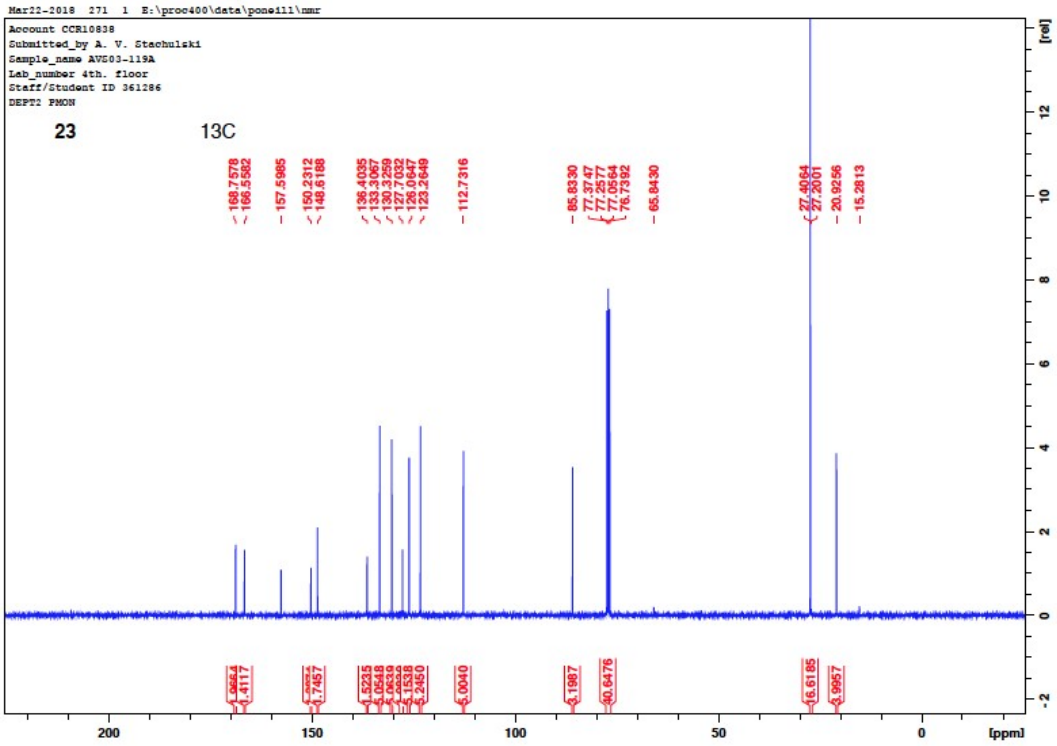
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Lab_number 4th. floor
Staff/Student ID 361286
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127.0647
123.2649
112.7316
85.8330
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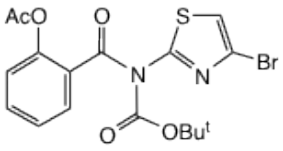
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Br
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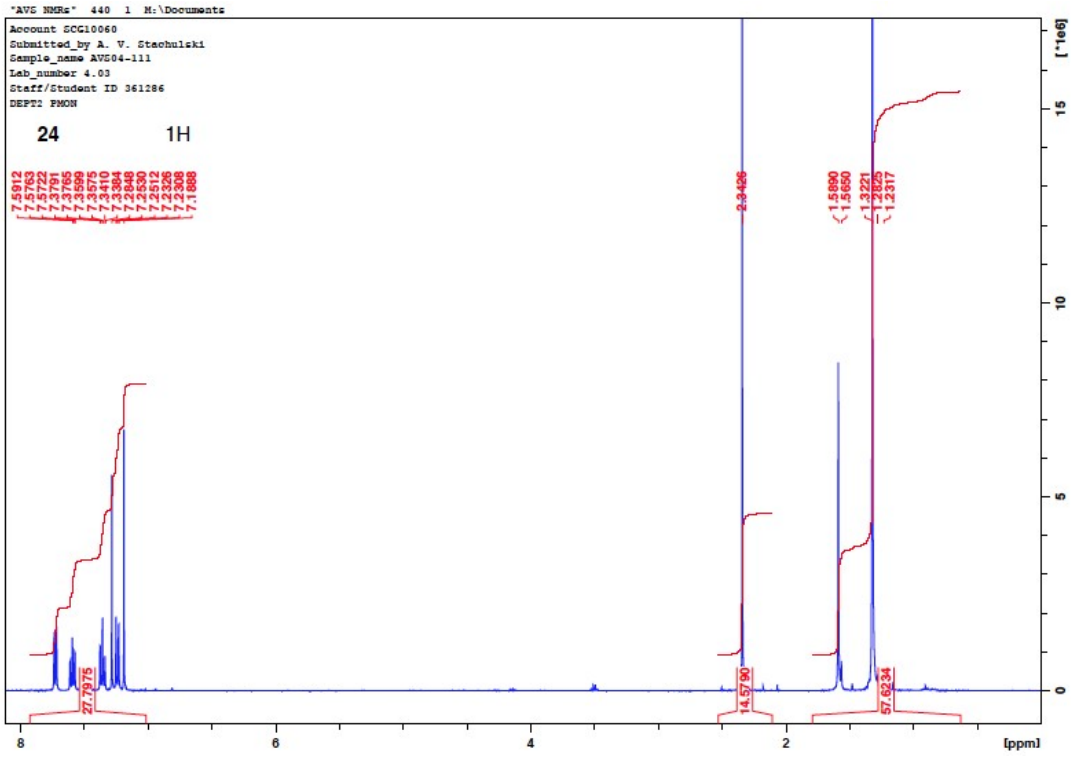
OBu^t

24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Staff/Student ID 361286
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7.3575
7.3410
7.3384
7.2848
7.2530
7.2512
7.2326
7.2308
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1.2826
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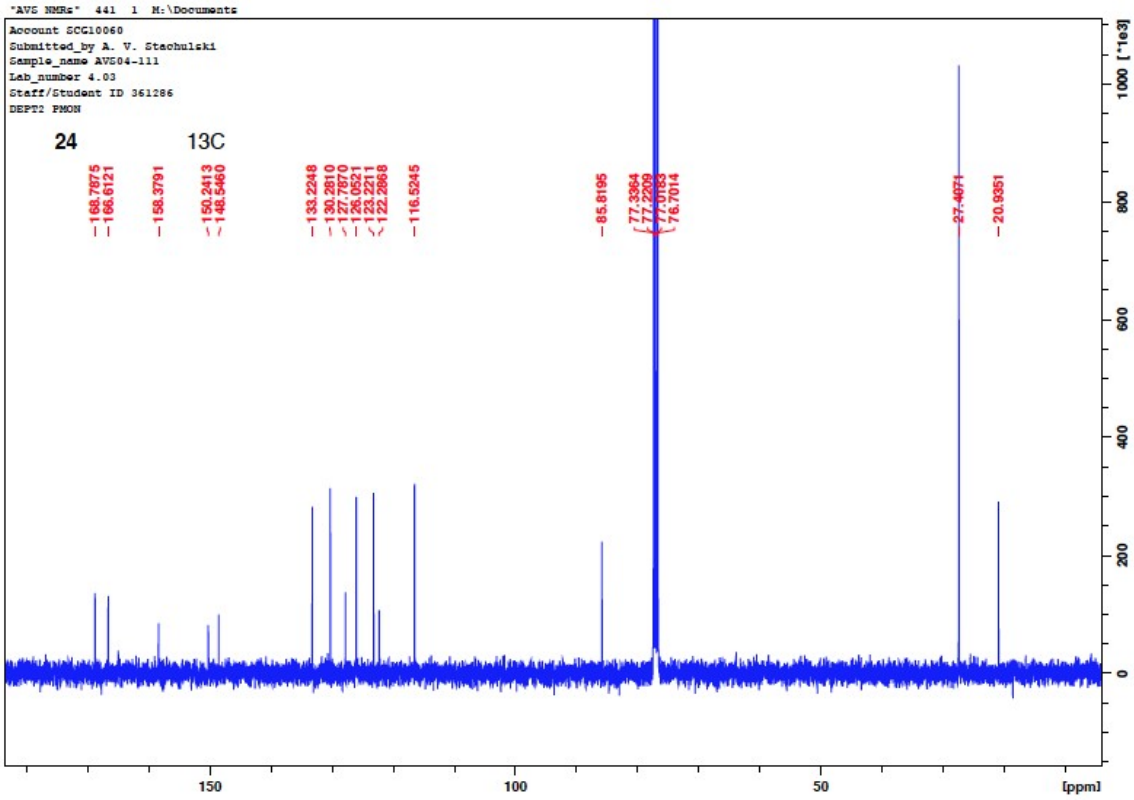
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Sample_name AVS04-111
Lab_number 4.03
Staff/Student ID 361286
DEPT2 PMON
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13C
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158.3791
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148.5460
133.2248
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127.7970
126.0521
123.2211
122.2868
116.5245
85.8195
77.3364
77.2209
77.0183
76.7014
27.4671
20.9351
[ppm]
[*1e3]

2. Full Cif crystallographic data files

Compound 13 CCDC 2362657.

Table 1 Crystal data and structure refinement for **13**.

Identification code	AVS_02_156
Empirical formula	$C_{41}H_{30}Cl_2N_4O_{12}S_2$
Formula weight	917.72
Temperature/K	150.01(10)
Crystal system	orthorhombic
Space group	*Pna2(1)*
a/Å	20.4035(6)
b/Å	8.6054(3)
c/Å	23.5849(6)
α/°	90.00
β/°	90.00
γ/°	90.00
Volume/Å^3	4141.0(2)
Z	4
ρ_{calc}g/cm^3	1.472
μ/mm^{-1}	0.327
F(000)	1888.0
Crystal size/mm^3	0.2 × 0.02 × 0.02
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	5.14 to 52.8
Index ranges	-25 ≤ h ≤ 25, -10 ≤ k ≤ 8, -29 ≤ l ≤ 29
Reflections collected	18121
Independent reflections	6797 [R_{int} = 0.0471, R_{sigma} = 0.0375]
Data/restraints/parameters	561/0/7
Goodness-of-fit on F^2	1.042
Final R indexes [I>=2σ (I)]	R_1 = 0.0441, wR_2 = 0.0999
Final R indexes [all data]	R_1 = 0.0584, wR_2 = 0.1082
Largest diff. peak/hole / e Å^{-3}	0.68/-0.50

Compound 15 CCDC 2330479.

Table 1 Crystal data and structure refinement for **15**.

Identification code	AVS04_106_auto

Empirical formula	$C_7H_8N_2O_3S$
Formula weight	200.21
Temperature/K	100.01(10)
Crystal system	orthorhombic
Space group	Pbcn
a/Å	22.162(3)
b/Å	7.1809(10)
c/Å	10.8505(15)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1726.8(4)
Z	8
ρ_{calc}g/cm^3	1.540
μ/mm^{-1}	0.349
F(000)	832.0
Crystal size/mm^3	0.22 × 0.05 × 0.02
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	5.964 to 60.416
Index ranges	-27 ≤ h ≤ 27, -7 ≤ k ≤ 8, -9 ≤ l ≤ 14
Reflections collected	7410
Independent reflections	2080 [R_{int} = 0.0205, R_{sigma} = 0.0205]
Data/restraints/parameters	2080/0/120
Goodness-of-fit on F^2	1.064
Final R indexes [I>=2σ (I)]	R_1 = 0.0306, wR_2 = 0.0833
Final R indexes [all data]	R_1 = 0.0349, wR_2 = 0.0867
Largest diff. peak/hole / e Å^{-3}	0.32/-0.28

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for **15**. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
S1	4888.7(2)	2475.9(4)	5969.1(3)	15.20(12)
O1	3769.9(4)	1186.3(14)	5314.5(8)	19.9(2)
O2	6326.8(4)	3863.7(14)	4148.0(8)	18.1(2)
O3	6808.2(5)	4590.6(17)	5910.2(9)	27.5(3)
N2	4383.6(5)	1802.6(16)	3708.2(9)	14.5(2)
N1	5384.8(5)	2740.6(15)	3839.4(10)	14.1(2)

C6	6789.8(6)	4604.9(18)	4811.2(12)	17.3(3)
C4	3846.7(6)	1246.3(18)	4202.4(12)	15.0(3)
C2	5798.7(5)	3277.9(18)	4697.0(11)	14.5(3)
C7	7244.0(6)	5435(2)	3963.6(13)	20.8(3)
C5	3372.4(6)	725(2)	3281.7(12)	20.5(3)
C1	4877.4(5)	2310.4(17)	4391.9(13)	13.2(3)
C3	5628.6(6)	3229.5(19)	5891.3(11)	16.1(3)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for **15**. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S1	14.51(19)	21.6(2)	9.54(17)	0.22(11)	1.14(10)	0.47(11)
O1	17.2(5)	28.9(5)	13.6(5)	3.0(4)	3.0(3)	-0.1(4)
O2	14.2(4)	26.9(5)	13.3(4)	-1.8(4)	0.5(3)	-5.0(4)
O3	21.4(5)	45.1(7)	15.9(5)	-0.7(4)	-3.6(4)	-8.1(4)
N2	12.6(5)	21.1(6)	9.7(5)	0.5(4)	0.3(4)	-0.8(4)
N1	12.6(5)	18.2(6)	11.5(5)	-0.8(4)	0.8(4)	-0.4(4)
C6	13.8(6)	19.2(7)	18.9(6)	-1.3(5)	-2.2(5)	0.1(4)
C4	13.3(6)	16.8(6)	15.0(6)	1.4(5)	1.2(4)	1.0(4)
C2	13.6(6)	16.2(6)	13.7(6)	-0.6(5)	0.7(4)	-0.5(4)
C7	14.8(6)	25.7(7)	21.9(7)	0.7(5)	0.0(5)	-3.5(5)
C5	13.9(6)	29.5(7)	18.1(6)	0.9(5)	-0.6(5)	-2.4(5)
C1	13.6(6)	15.1(6)	10.9(6)	0.5(4)	0.6(4)	1.2(4)
C3	15.6(6)	20.5(7)	12.2(6)	-0.2(5)	-0.6(4)	-0.7(5)

Table 4 Bond Lengths for **15**.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
S1	C1	1.7156(14)	N2	C1	1.3714(16)
S1	C3	1.7289(13)	N1	C2	1.3625(16)
O1	C4	1.2193(16)	N1	C1	1.3111(17)
O2	C6	1.3618(15)	C6	C7	1.4881(18)
O2	C2	1.3790(15)	C4	C5	1.4977(17)
O3	C6	1.1932(16)	C2	C3	1.3500(17)
N2	C4	1.3649(16)			

Table 5 Bond Angles for **15**.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C1	S1	C3	89.24(6)	N2	C4	C5	115.02(11)
C6	O2	C2	122.05(10)	N1	C2	O2	111.27(10)
C4	N2	C1	124.12(11)	C3	C2	O2	131.26(11)

C1	N1	C2	109.38(11)	C3	C2	N1	117.41(11)
O2	C6	C7	109.86(11)	N2	C1	S1	124.72(10)
O3	C6	O2	123.40(12)	N1	C1	S1	115.30(10)
O3	C6	C7	126.73(12)	N1	C1	N2	119.98(12)
O1	C4	N2	121.39(12)	C2	C3	S1	108.64(9)
O1	C4	C5	123.59(11)				

Table 6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for **15**.

Atom	***x***	***y***	***z***	**U(eq)**
H2	4414.91	1837.55	2900.05	17
H7A	7230.56	4788.6	3168.81	31
H7B	7647.5	5306.63	4323.17	31
H7C	7153.04	6758.01	3840.74	31
H5A	3221.94	-528.37	3464.69	31
H5B	3546.43	744.43	2451.54	31
H5C	3038.53	1617.02	3326.56	31
H3	5874.58	3555.2	6575.21	19

Compound 19 CCDC 2330480

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

Identification code	AVS_30_11_23_dry_auto
Empirical formula	$C_5H_5ClN_2OS$
Formula weight	176.62
Temperature/K	100.01(10)
Crystal system	triclinic
Space group	P-1
a/Å	5.0499(2)
b/Å	7.4591(3)
c/Å	9.4964(3)
α/°	90.462(3)
β/°	91.106(3)
γ/°	108.081(4)
Volume/Å^3	339.94(2)
Z	2
ρ_{calc}g/cm^3	1.725
μ/mm^{-1}	0.790
F(000)	180.0
Crystal size/mm^3	0.1 × 0.02 × 0.02
Radiation	Mo Kα (λ = 0.71073)

2Θ range for data collection/°	4.29 to 60.426
Index ranges	$-6 \le h \le 6$, $-10 \le k \le 10$, $-13 \le l \le 13$
Reflections collected	7149
Independent reflections	1682 [R_{int} = 0.0437, R_{sigma} = 0.0246]
Data/restraints/parameters	1682/0/92
Goodness-of-fit on F^2	1.067
Final R indexes [I>=2σ (I)]	R_1 = 0.0251, wR_2 = 0.0691
Final R indexes [all data]	R_1 = 0.0264, wR_2 = 0.0701
Largest diff. peak/hole / e Å^{-3}	0.46/-0.33

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for **19**. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*Z*	U(eq)
S1	2669.8(6)	1879.7(4)	5997.4(3)	11.08(10)
Cl1	1273.9(6)	2319.4(4)	9030.3(3)	14.75(10)
O1	2940.6(19)	1076.5(13)	3269.9(10)	18.4(2)
N2	6930(2)	3117.6(14)	4156.9(10)	11.1(2)
N1	7566(2)	4298.3(14)	6464.7(11)	12.3(2)
C3	5982(2)	3202.7(16)	5493.4(12)	10.0(2)
C2	6167(3)	4112.6(17)	7714.4(13)	12.9(2)
C4	5350(3)	2019.3(16)	3096.5(12)	12.2(2)
C5	6863(3)	2085.8(18)	1737.5(13)	15.4(2)
C1	3554(3)	2884.7(16)	7664.4(12)	12.1(2)

Table 3 Anisotropic Displacement Parameters (Å^2×10^3) for **19**. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S1	7.56(16)	11.78(16)	11.48(16)	-0.33(11)	2.27(11)	-0.64(11)
Cl1	12.32(17)	18.38(17)	11.78(16)	0.46(11)	5.50(11)	1.87(12)
O1	10.9(4)	20.2(5)	18.7(5)	-4.6(4)	1.2(3)	-2.9(4)
N2	7.1(5)	12.5(5)	11.3(5)	-0.4(4)	2.3(4)	-0.6(4)
N1	9.2(5)	13.1(5)	12.7(5)	-0.6(4)	1.4(4)	1.0(4)
C3	6.8(5)	10.3(5)	12.3(5)	1.0(4)	1.5(4)	1.5(4)
C2	11.4(6)	14.9(5)	12.1(5)	-0.9(4)	0.7(4)	3.4(4)
C4	11.3(6)	11.4(5)	13.2(5)	-0.9(4)	0.8(4)	2.4(4)
C5	14.7(6)	17.4(6)	12.3(5)	-2.5(4)	2.2(4)	2.5(5)
C1	11.6(5)	13.9(5)	10.9(5)	0.5(4)	2.6(4)	4.1(4)

Table 4 Bond Lengths for **19**.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
S1	C3	1.7362(12)	N2	C4	1.3681(15)
S1	C1	1.7359(12)	N1	C3	1.3086(15)
Cl1	C1	1.7180(12)	N1	C2	1.3789(15)
O1	C4	1.2169(16)	C2	C1	1.3522(18)
N2	C3	1.3730(15)	C4	C5	1.5058(17)

Table 5 Bond Angles for **19**.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C1	S1	C3	87.21(6)	O1	C4	N2	121.57(11)
C4	N2	C3	123.44(11)	O1	C4	C5	124.27(11)
C3	N1	C2	109.99(10)	N2	C4	C5	114.15(10)
N2	C3	S1	122.68(9)	Cl1	C1	S1	121.79(7)
N1	C3	S1	116.33(9)	C2	C1	S1	111.61(9)
N1	C3	N2	121.00(11)	C2	C1	Cl1	126.60(10)
C1	C2	N1	114.85(11)				

Table 6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for **19**.

Atom	*x*	*y*	*z*	U(eq)
H2	8643.35	3805.52	3972.81	13
H2A	6975.64	4794.88	8546.6	16
H5A	5652.16	1198.5	1053.63	23
H5B	8557.7	1737.16	1911.78	23
H5C	7365.39	3365.14	1362.57	23