

SUPPORTING INFORMATION

Silver Mediated Thio-Acetoxylation and TFA Triggered Cyclization of Amino Disulfides with Unactivated Alkenes: Synthesis of 3-Aryl/Alkyl-1,4-benzothiazines

Ch. Durga Prasad, Ajay Verma, Moh. Sattar, Sangit Kumar*

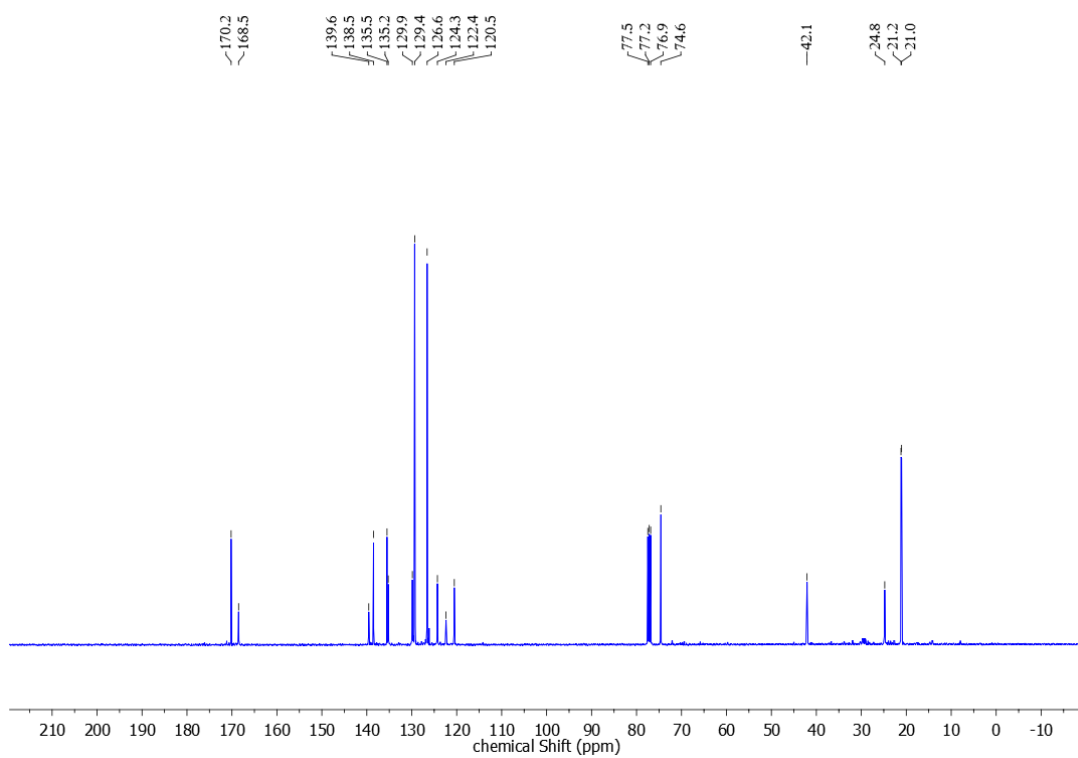
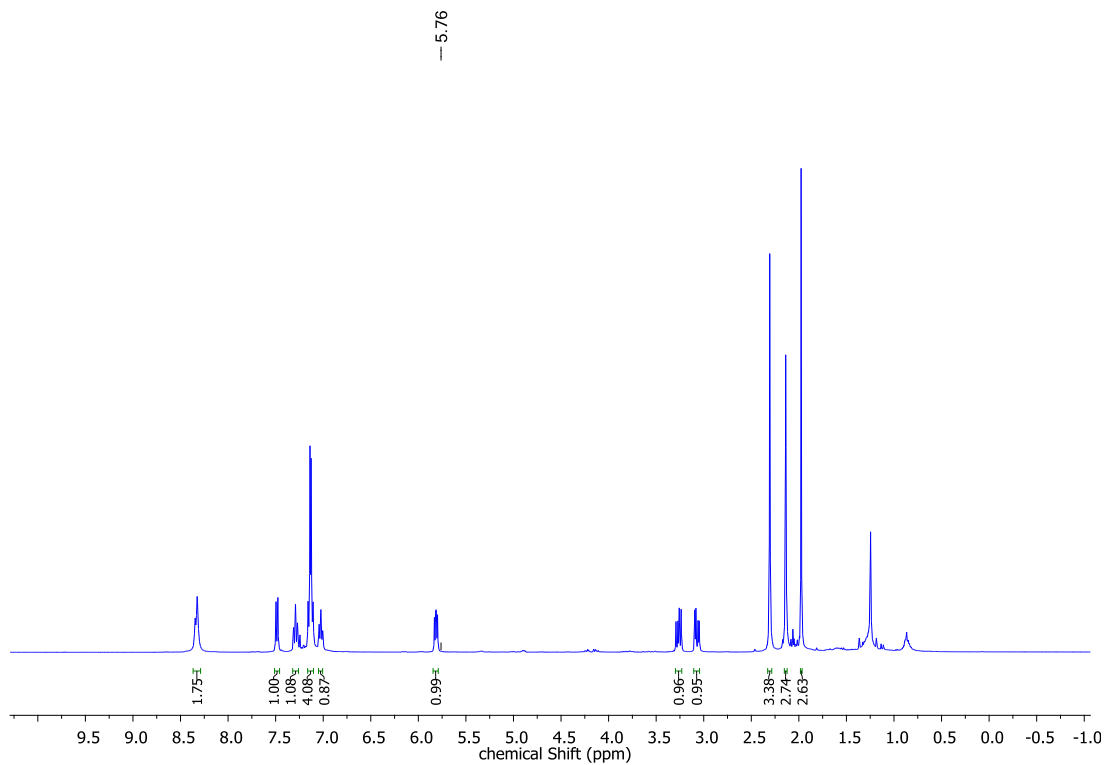
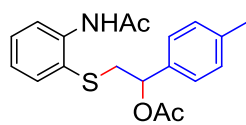
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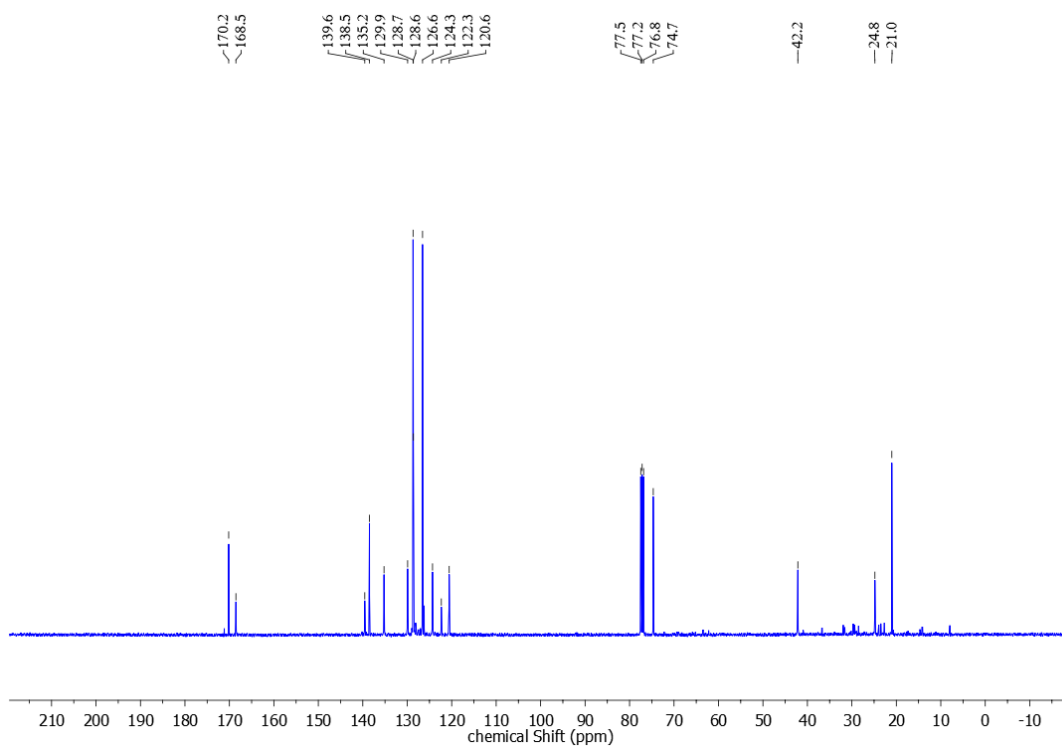
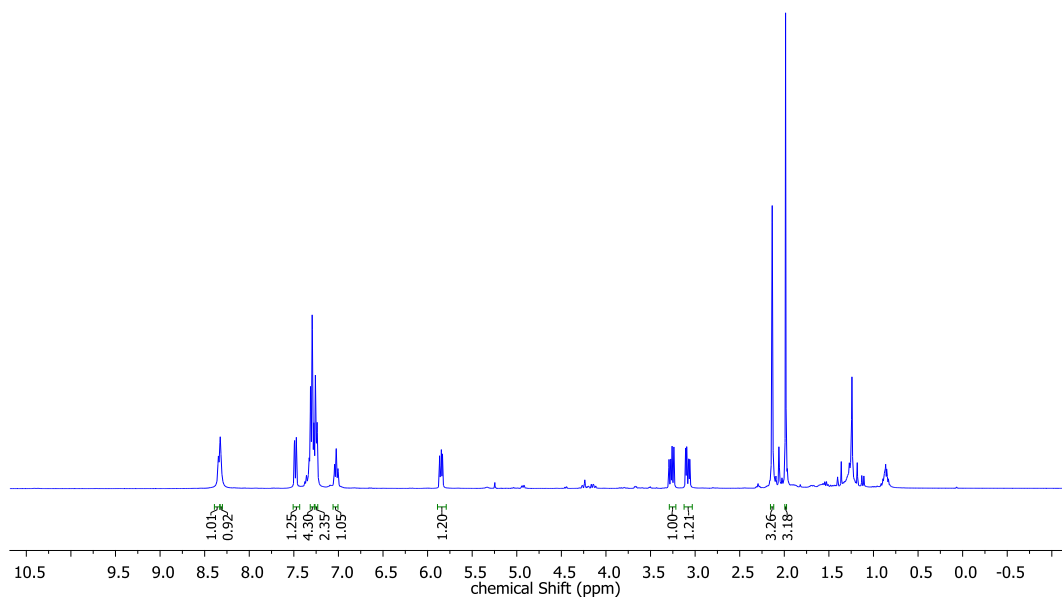
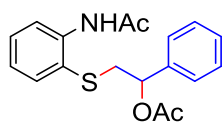
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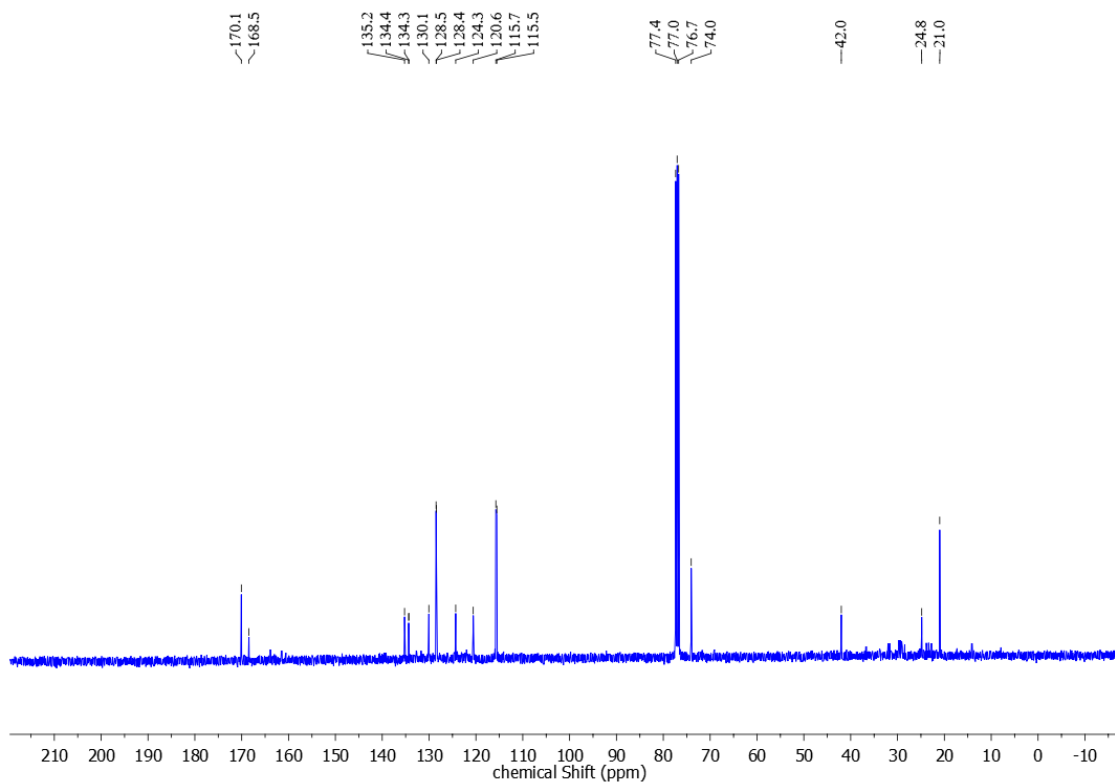
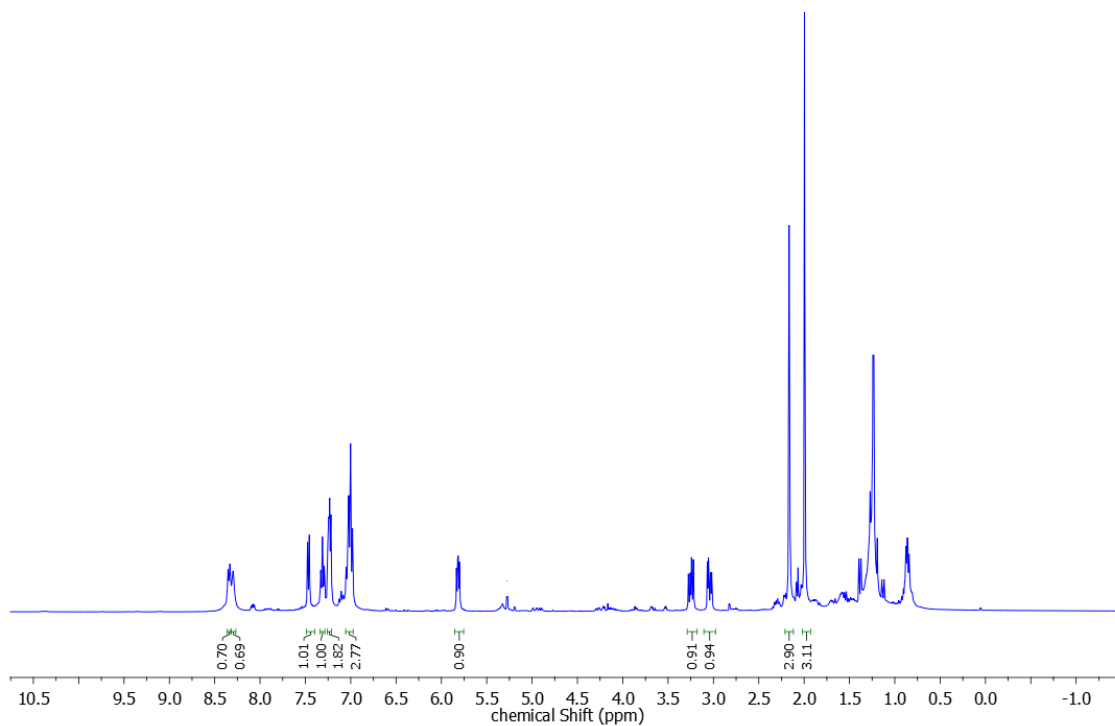
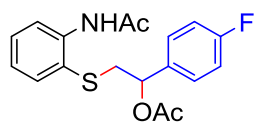
Spectral data of compound 1a



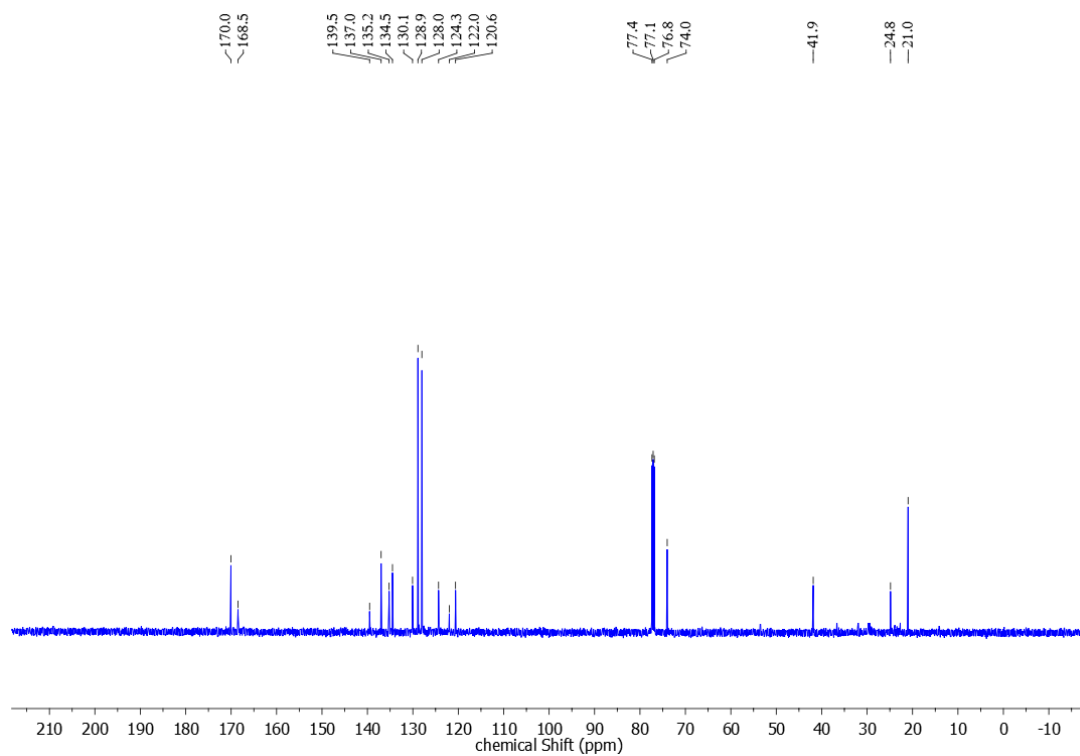
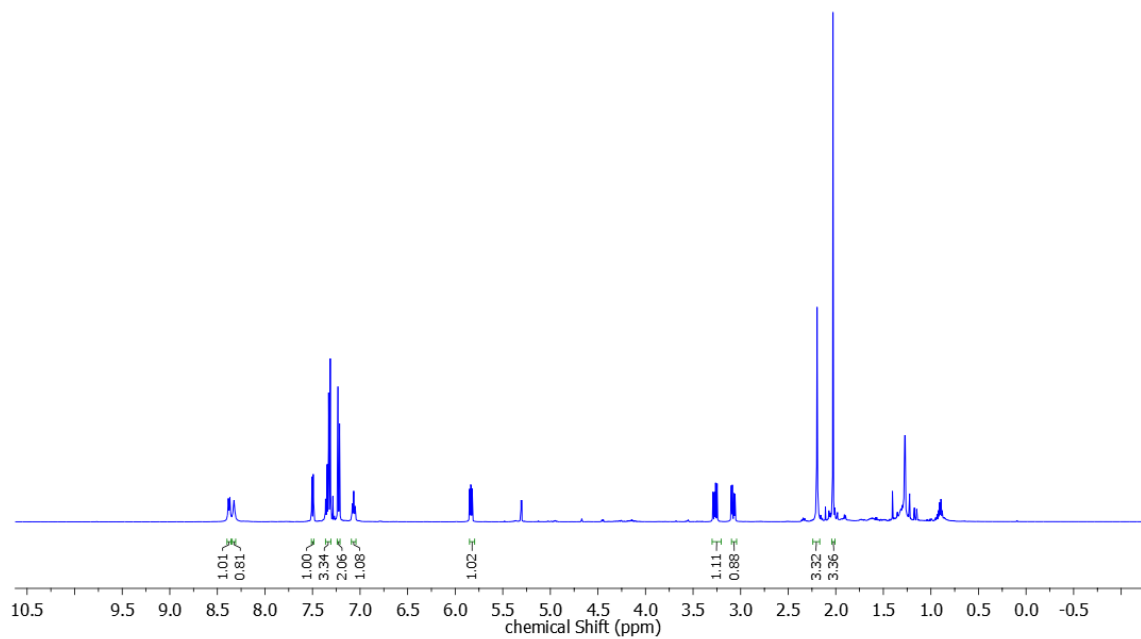
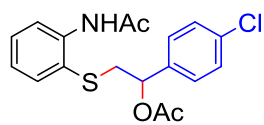
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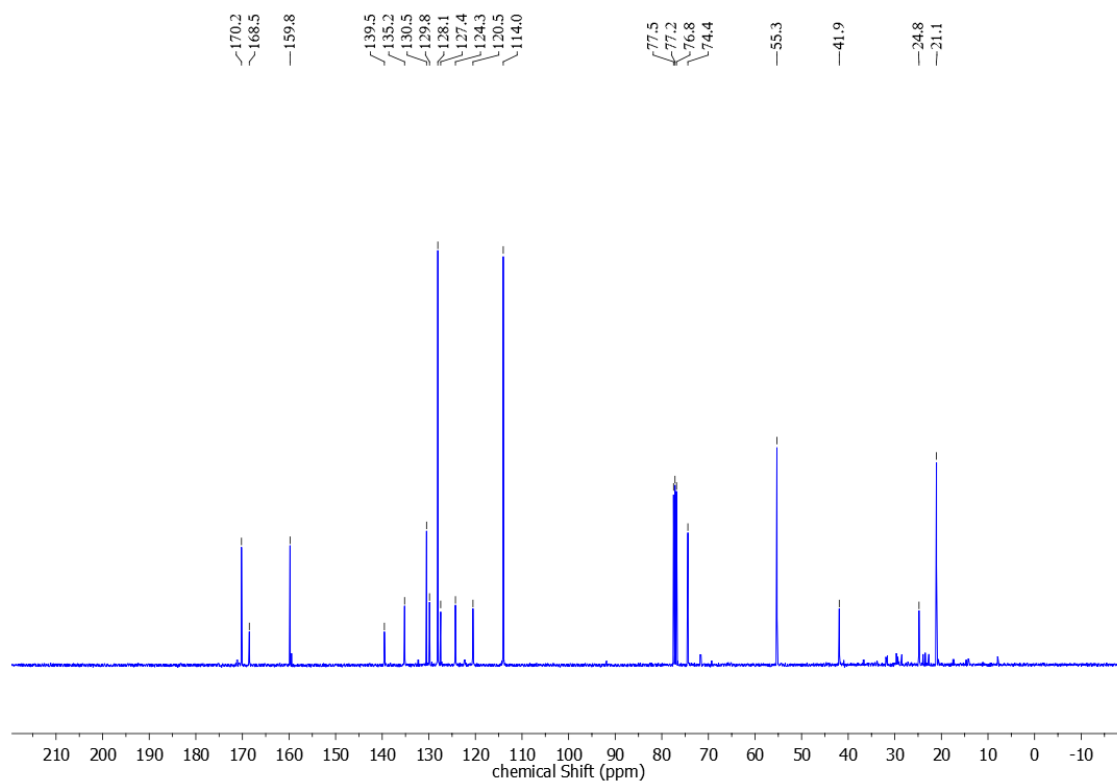
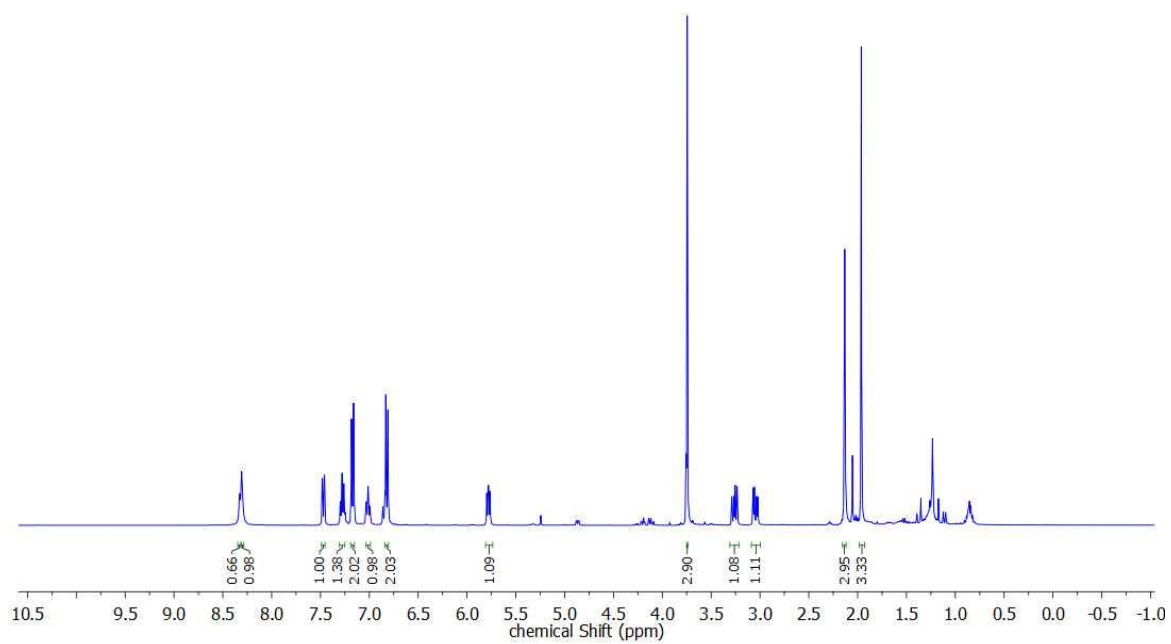
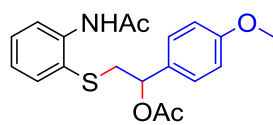
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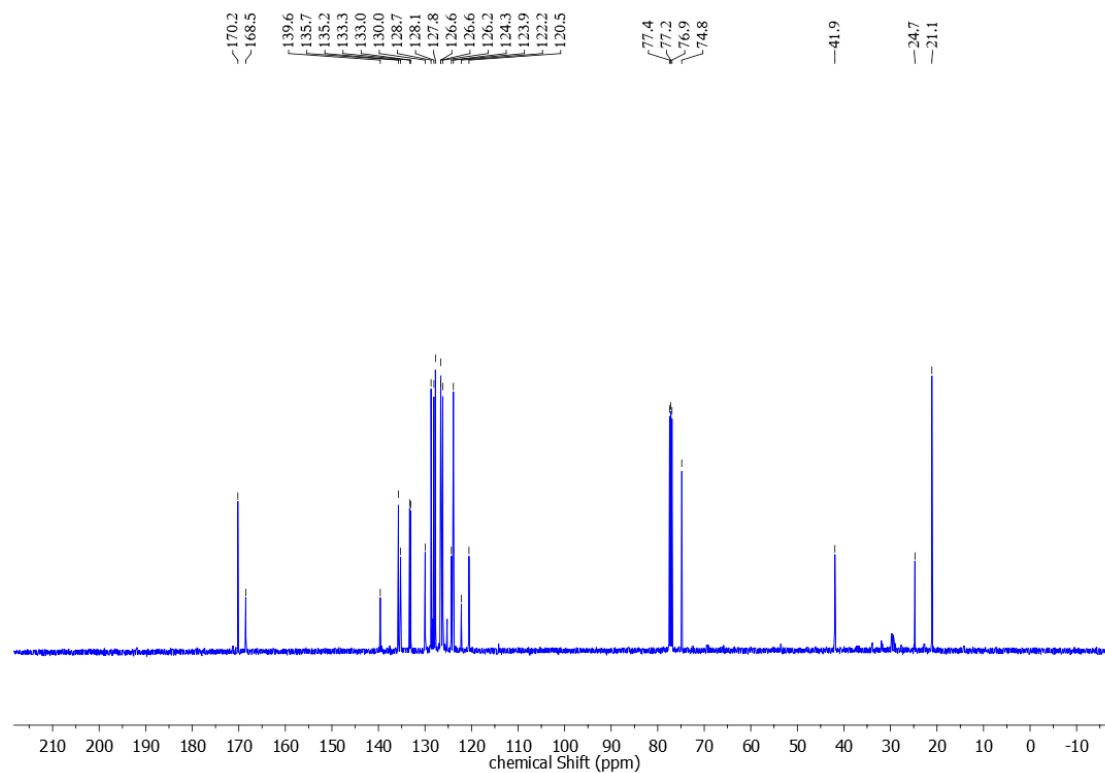
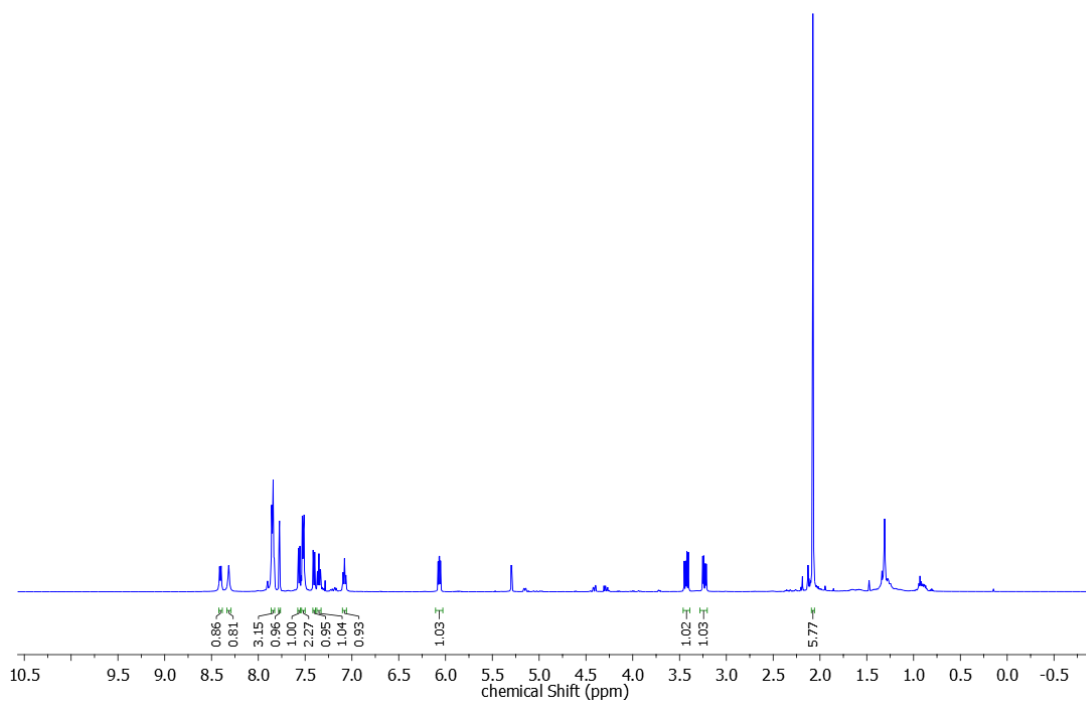
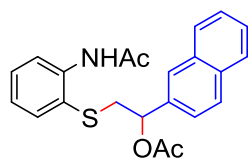
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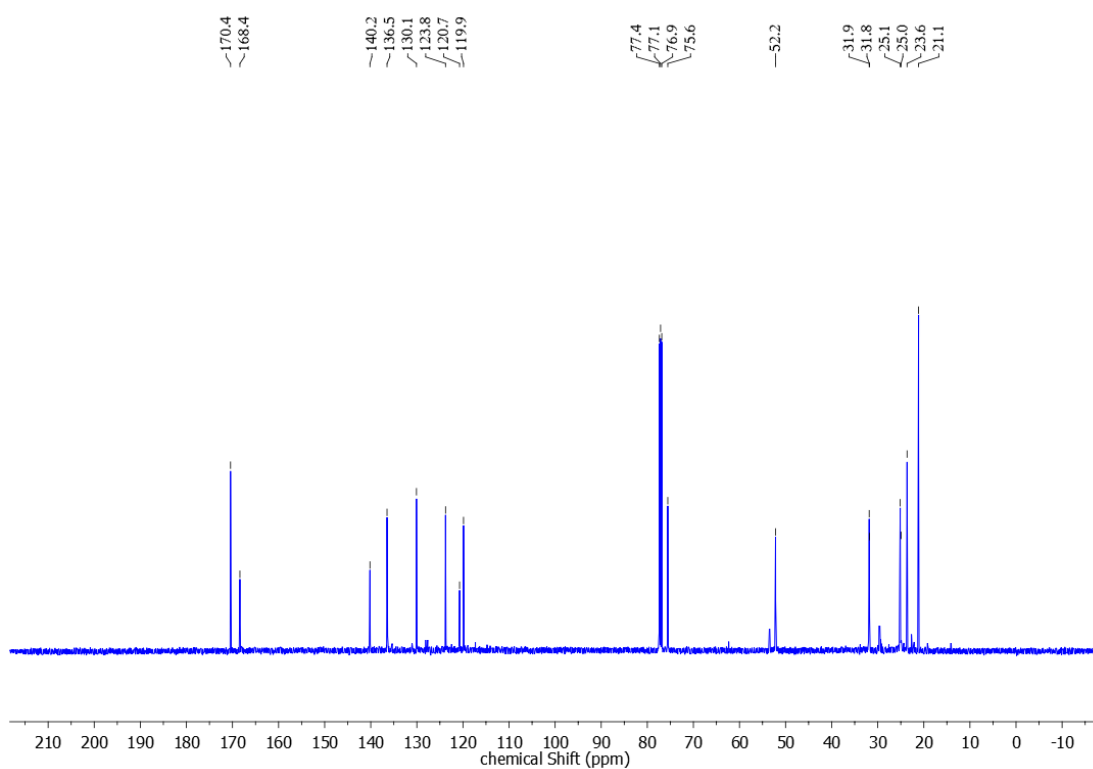
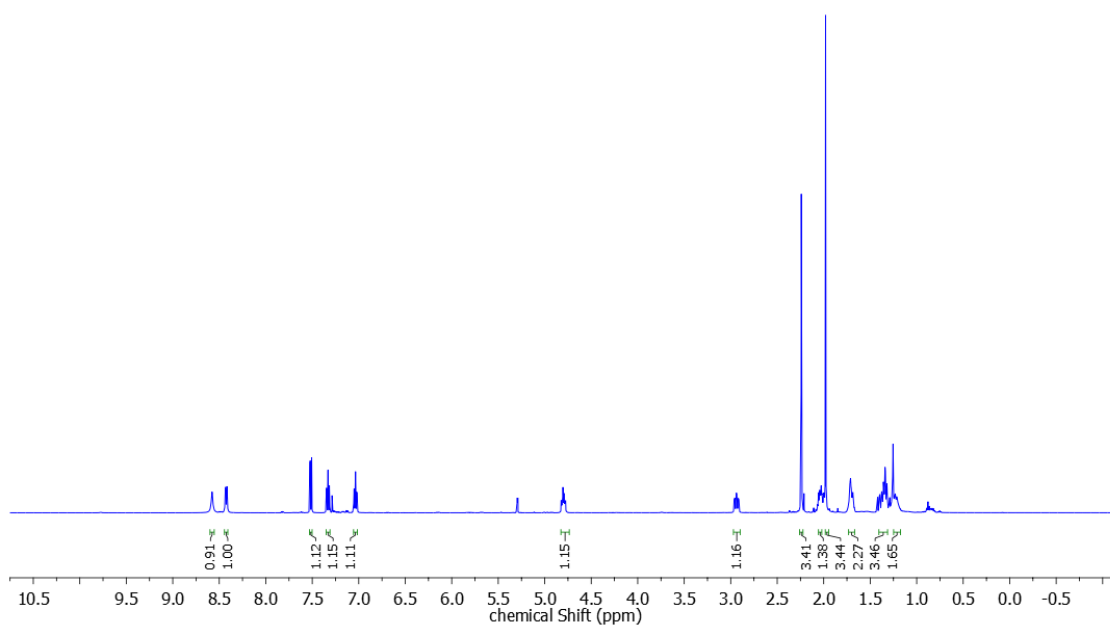
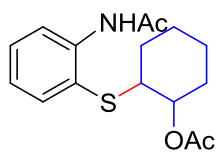
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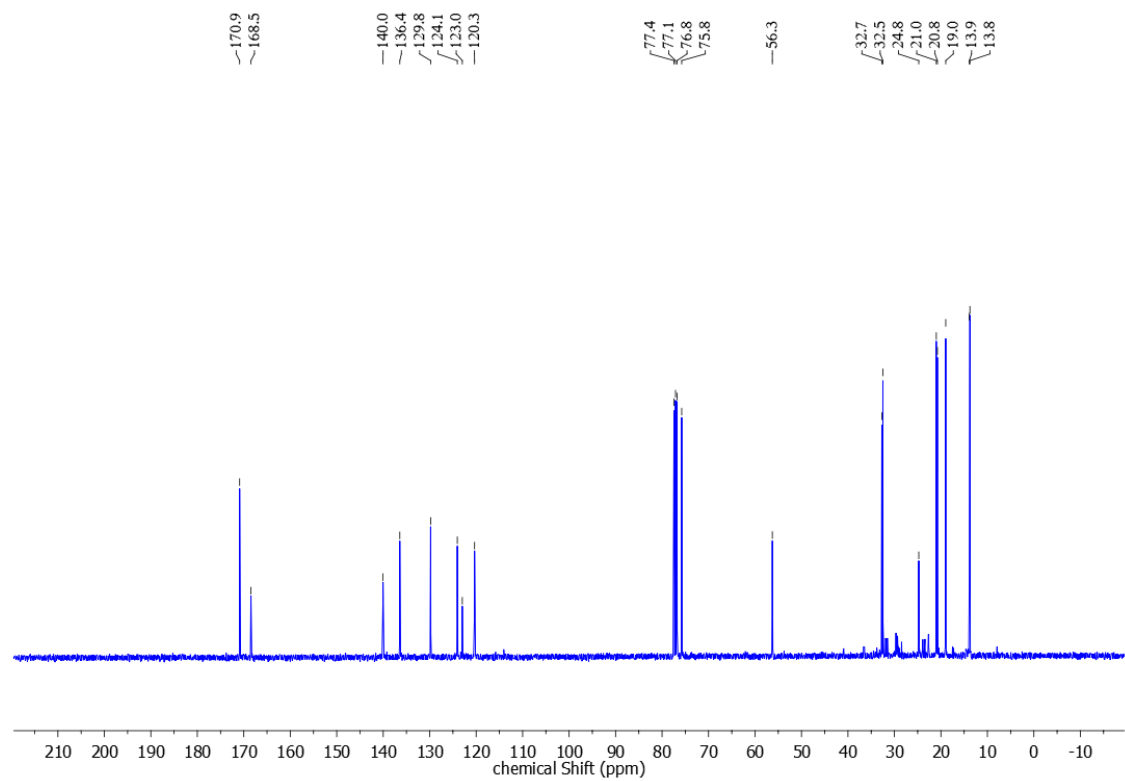
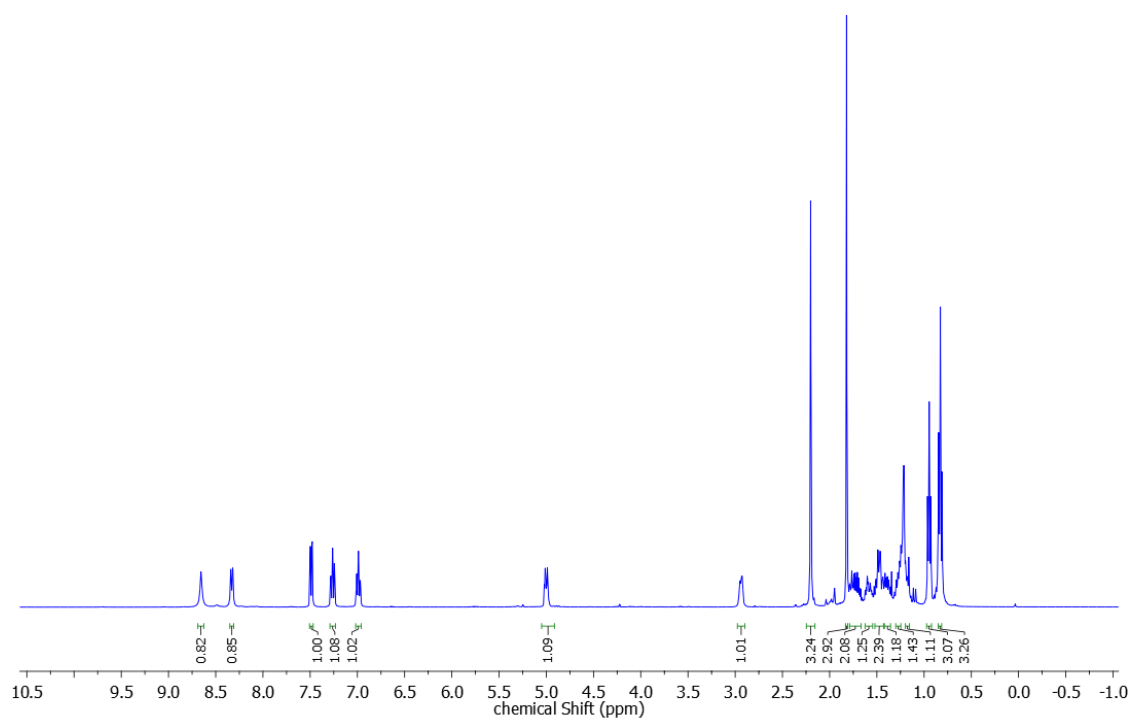
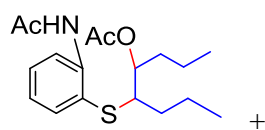
Spectral data of compound 1g



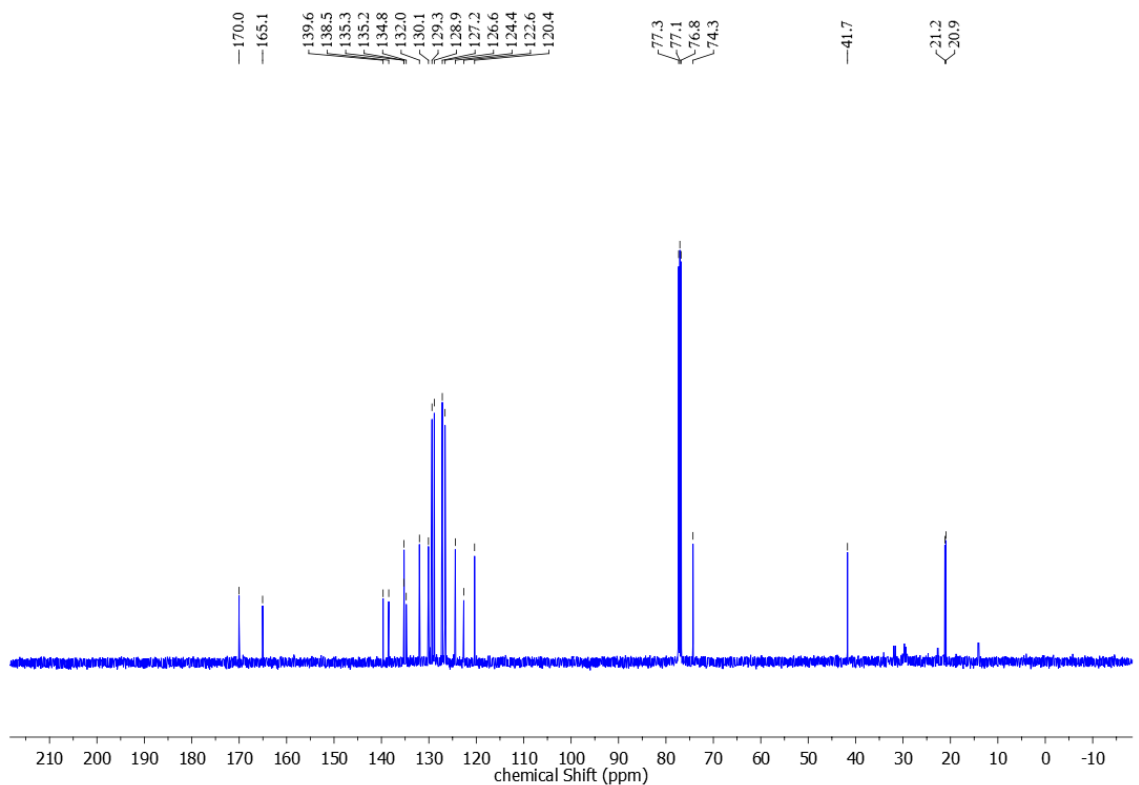
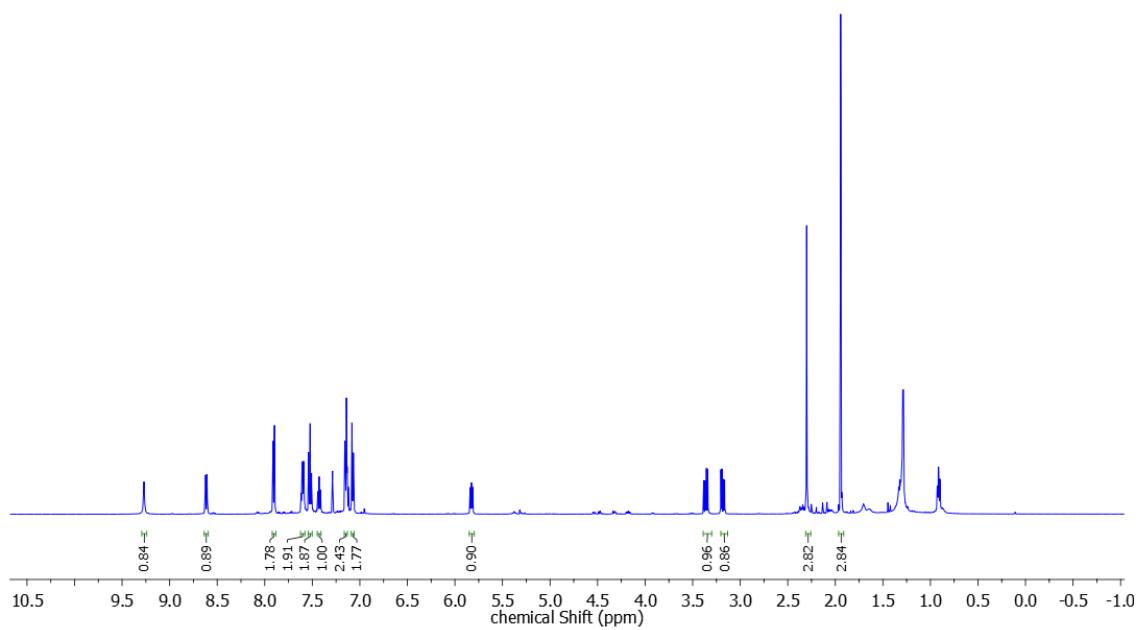
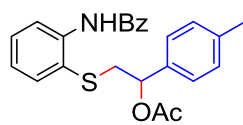
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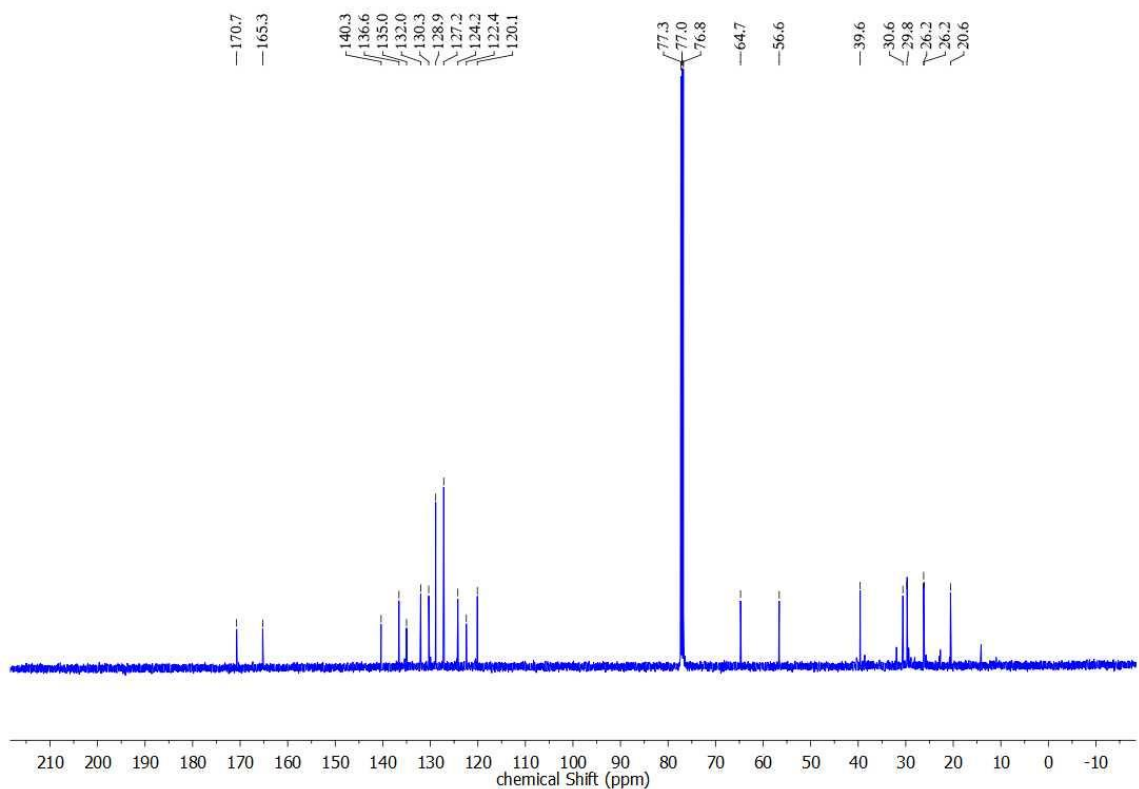
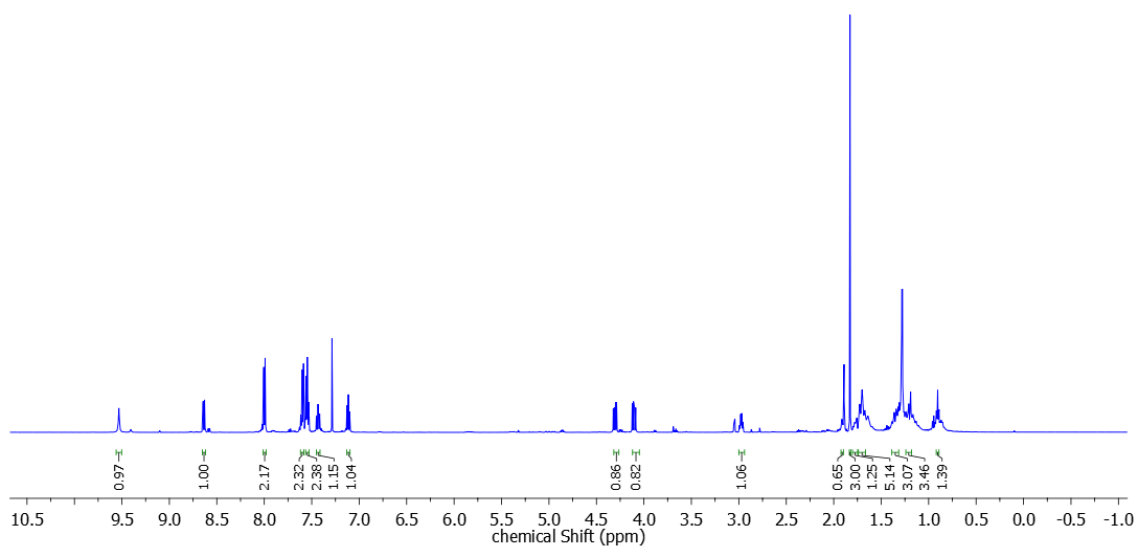
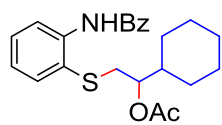
Spectral data of compound 1i



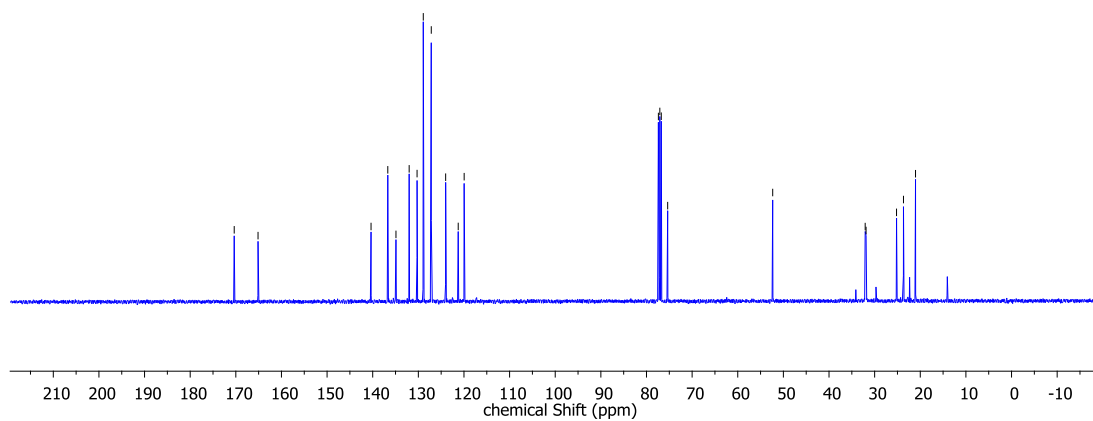
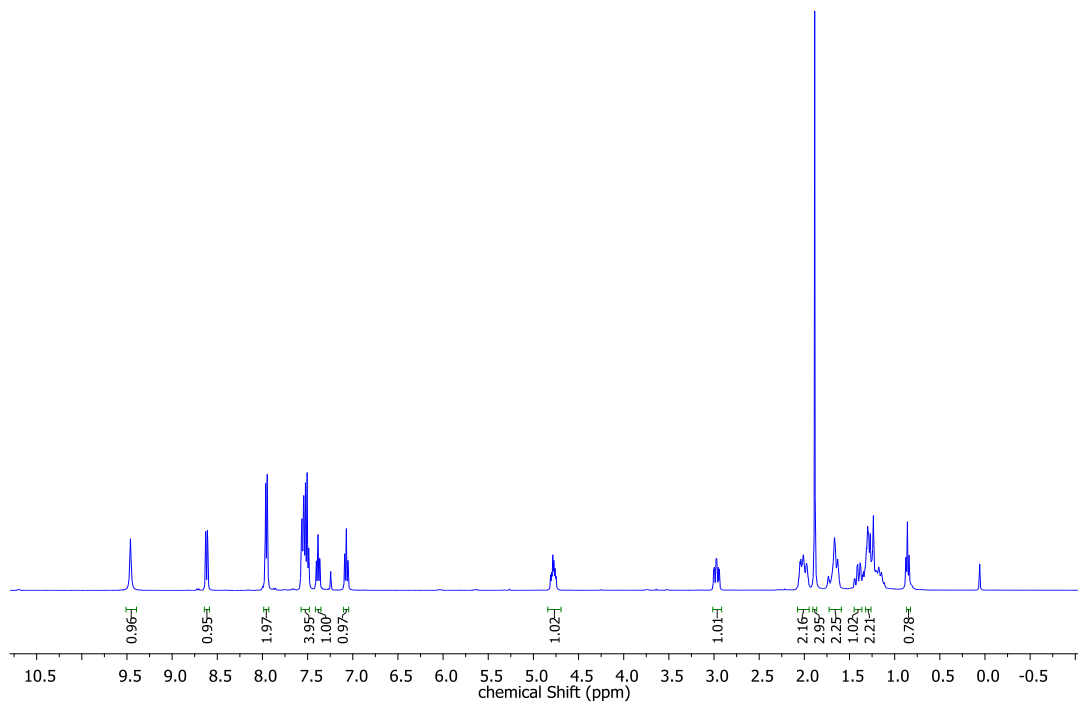
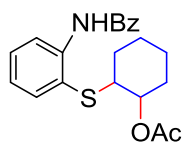
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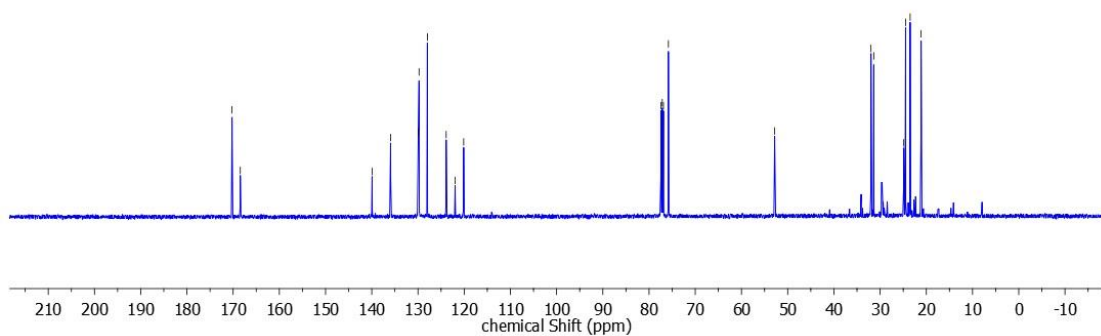
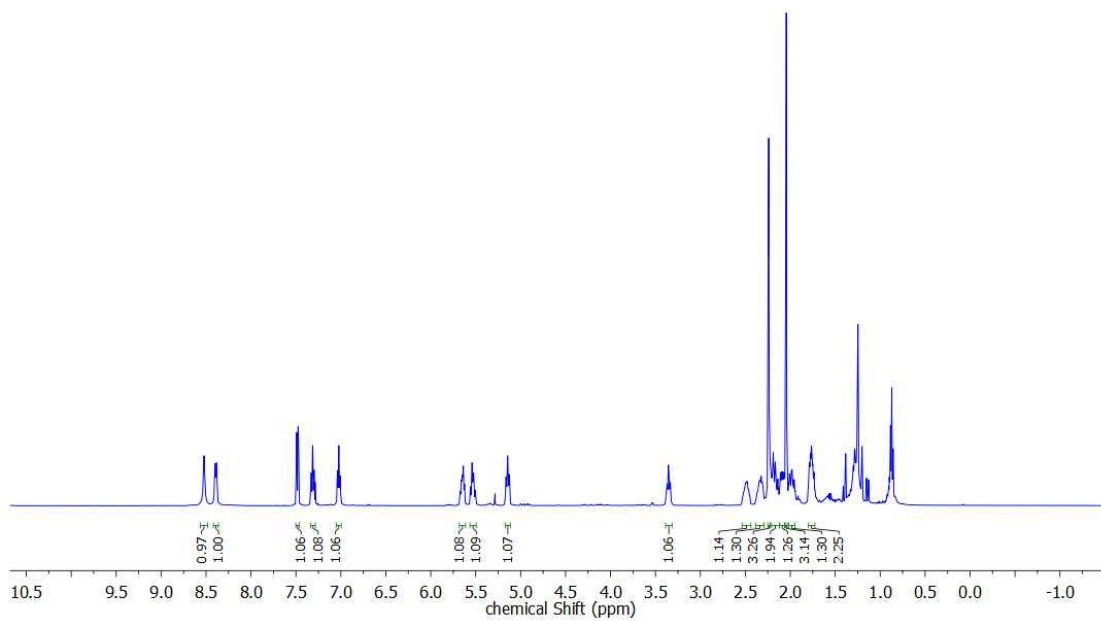
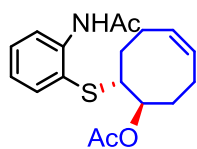
Spectral data of compound 1k



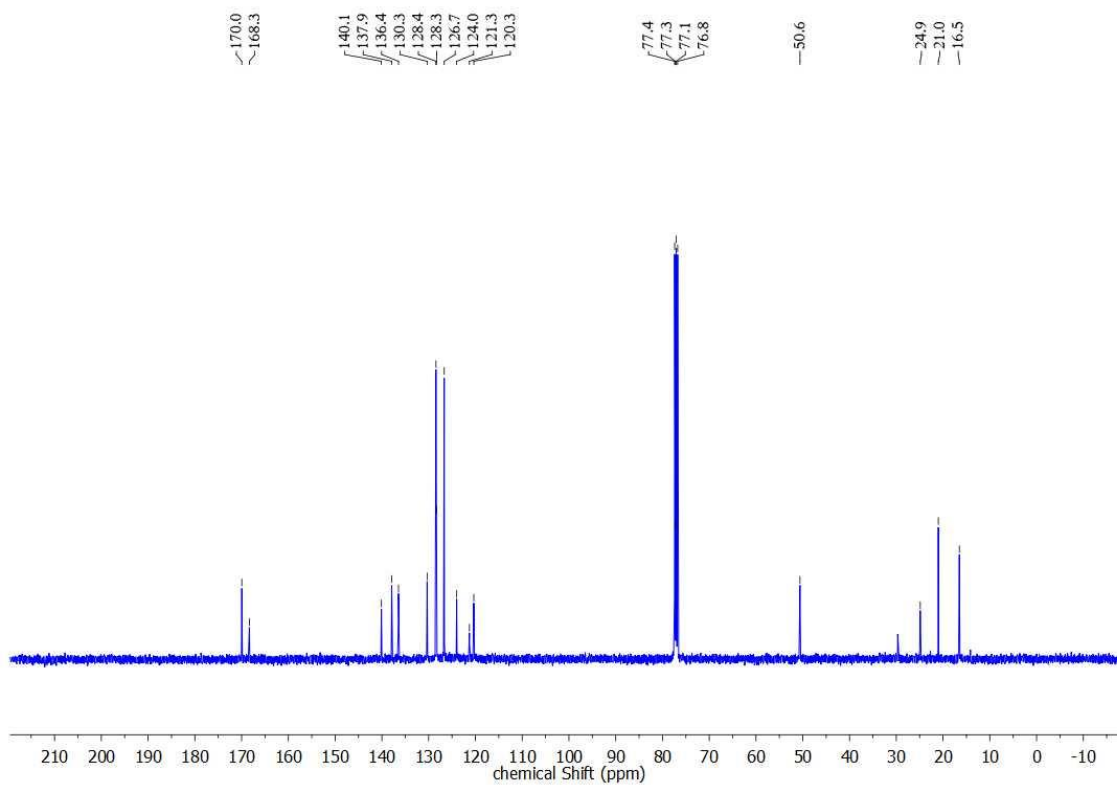
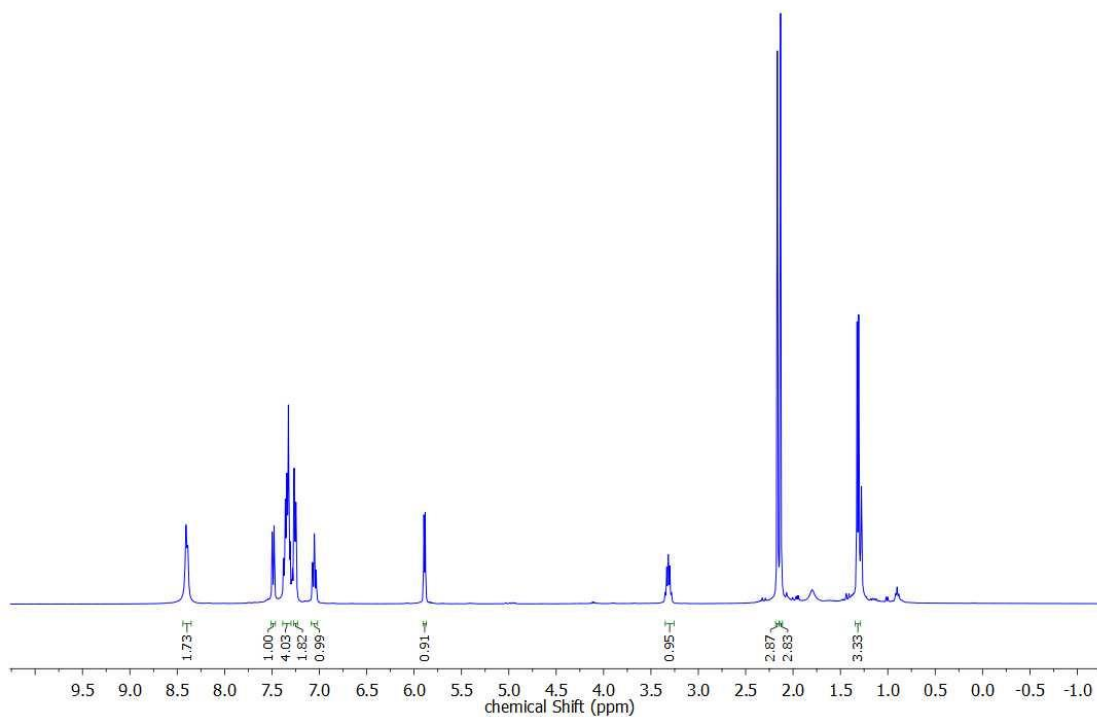
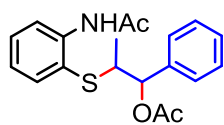
Spectral data of compound 11



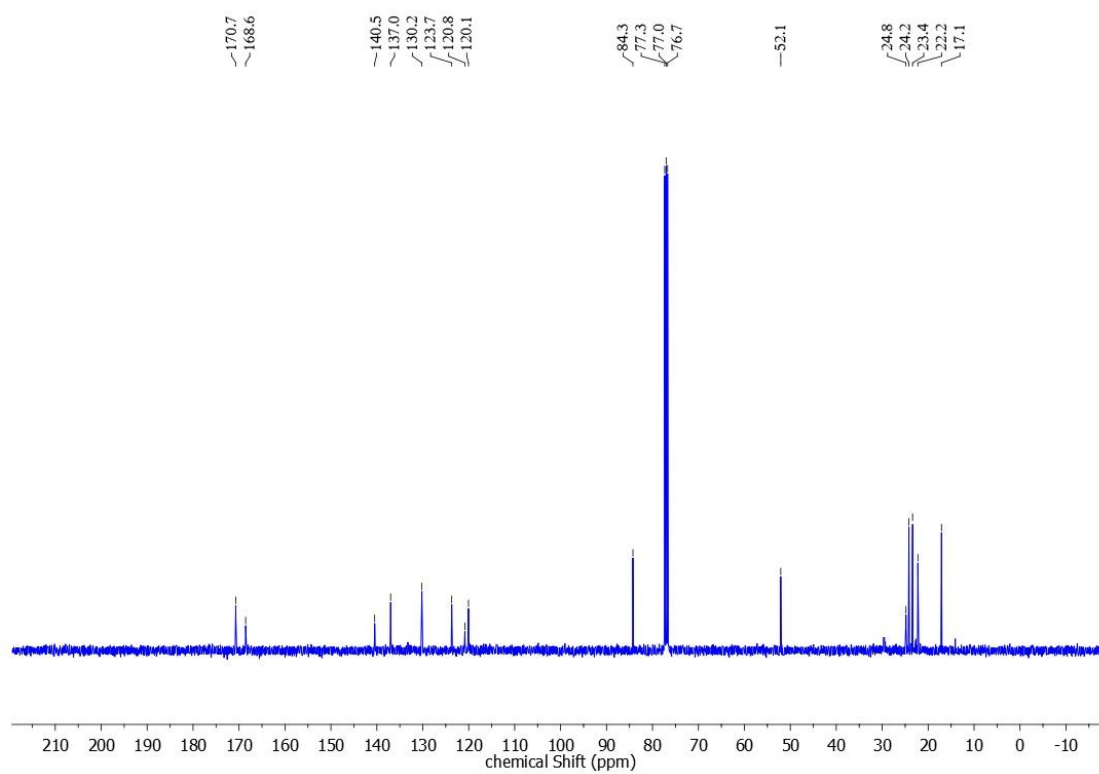
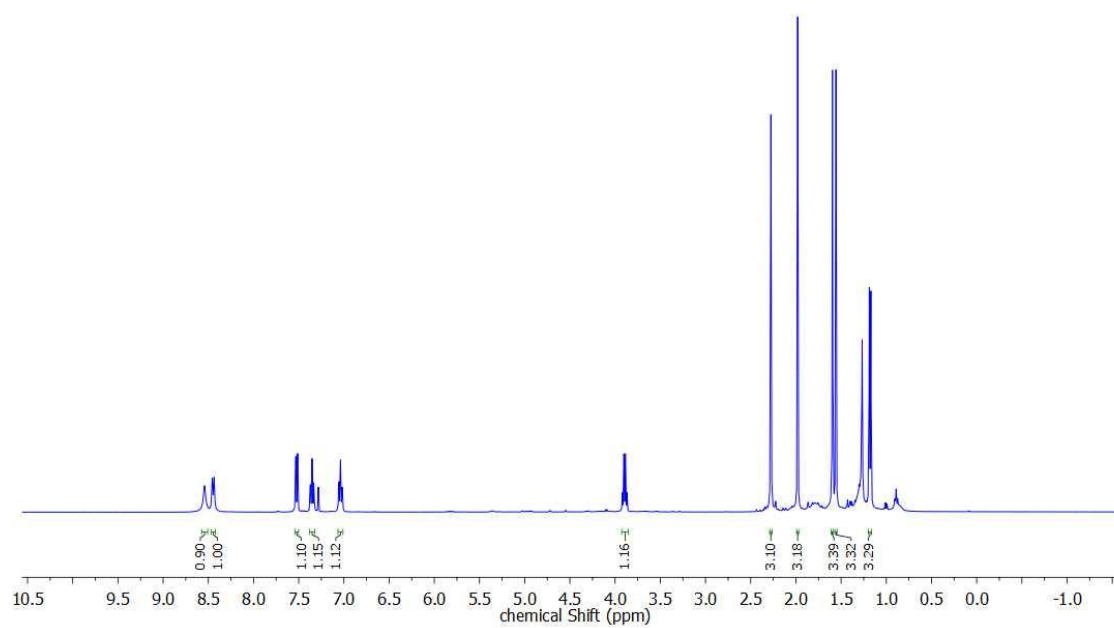
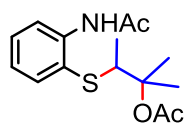
Spectral data of compound 1m



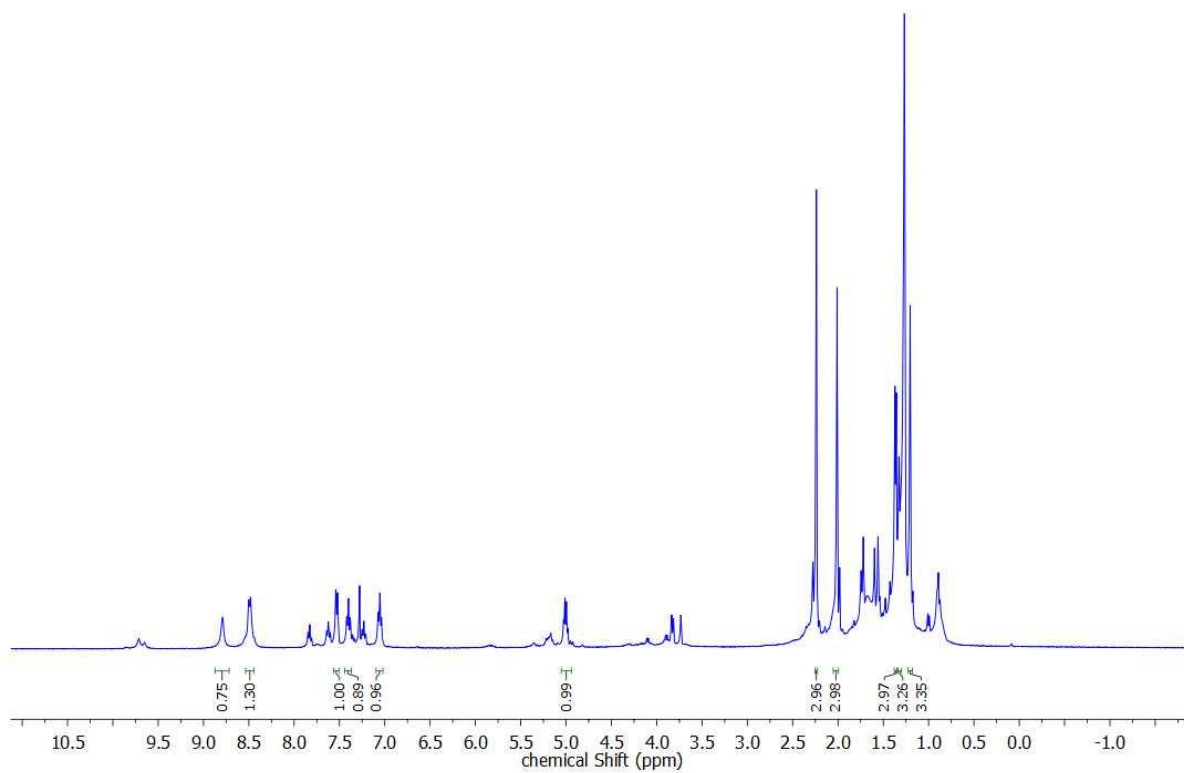
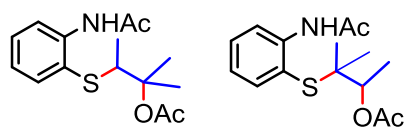
Spectral data of compound 1n



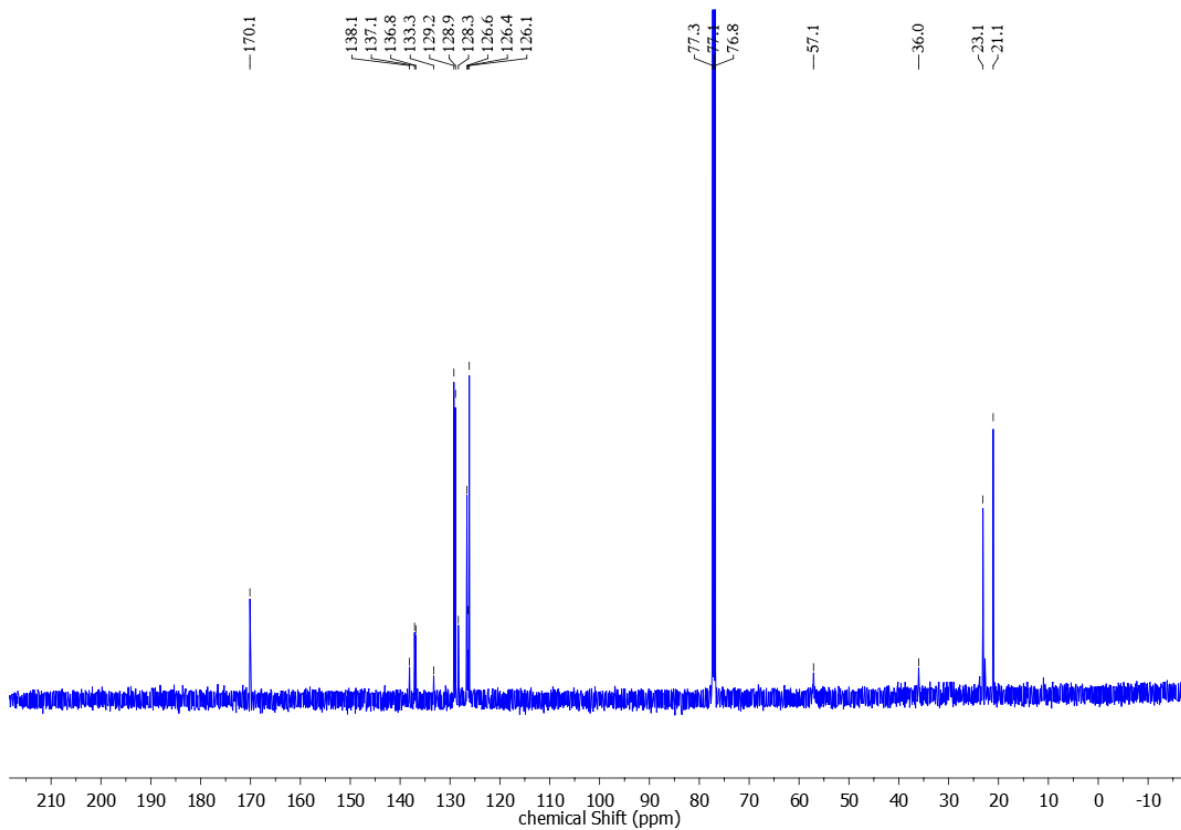
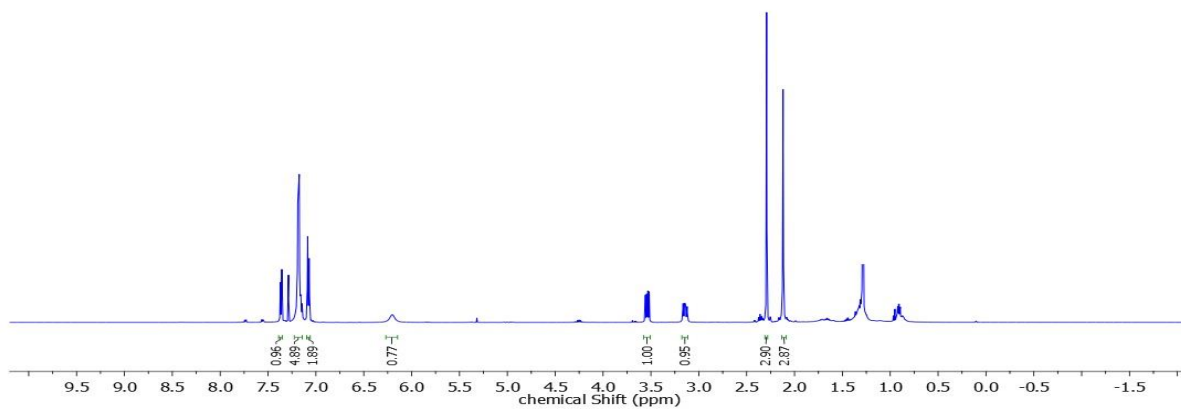
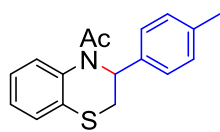
Spectral data of compound 1o



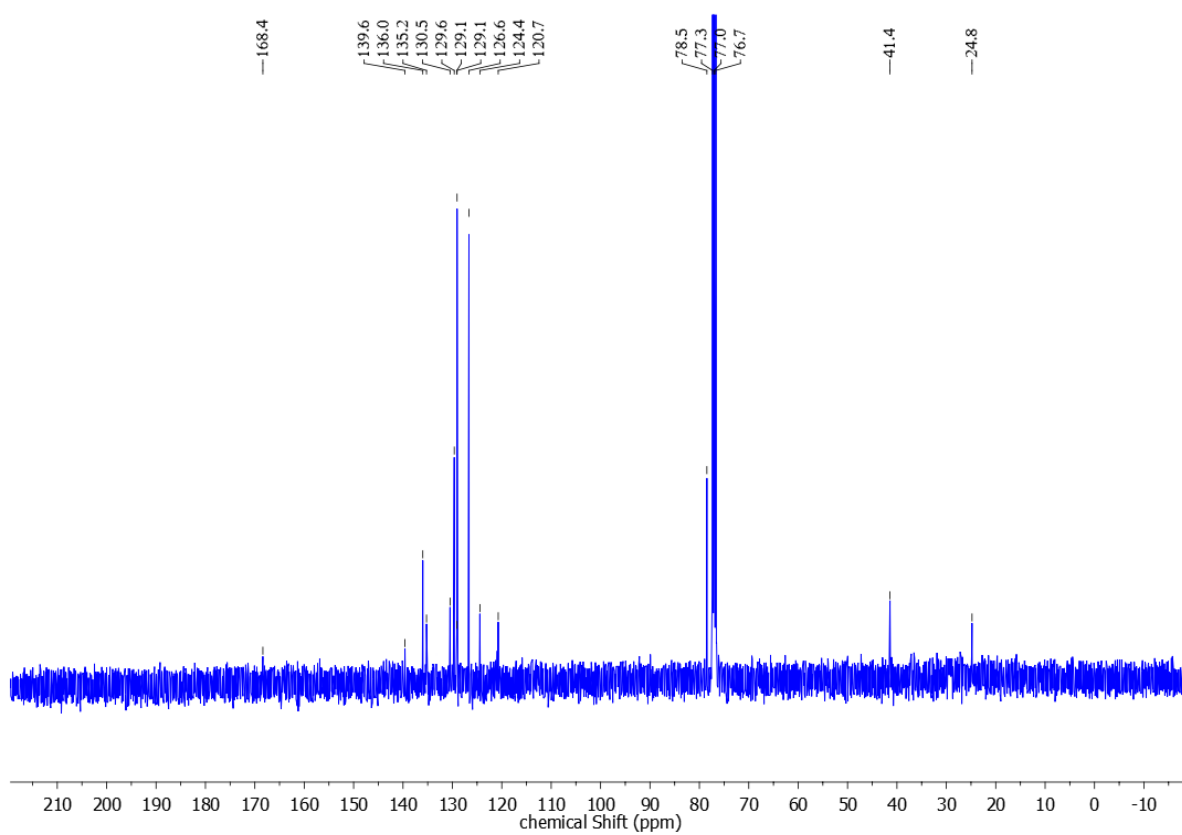
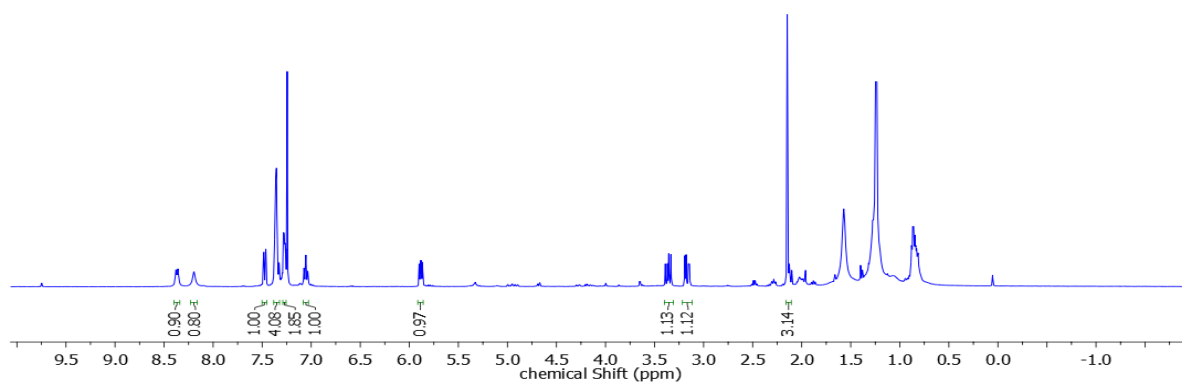
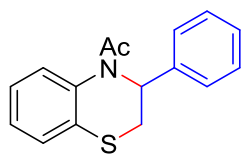
¹H-NMR of mixture of isomers



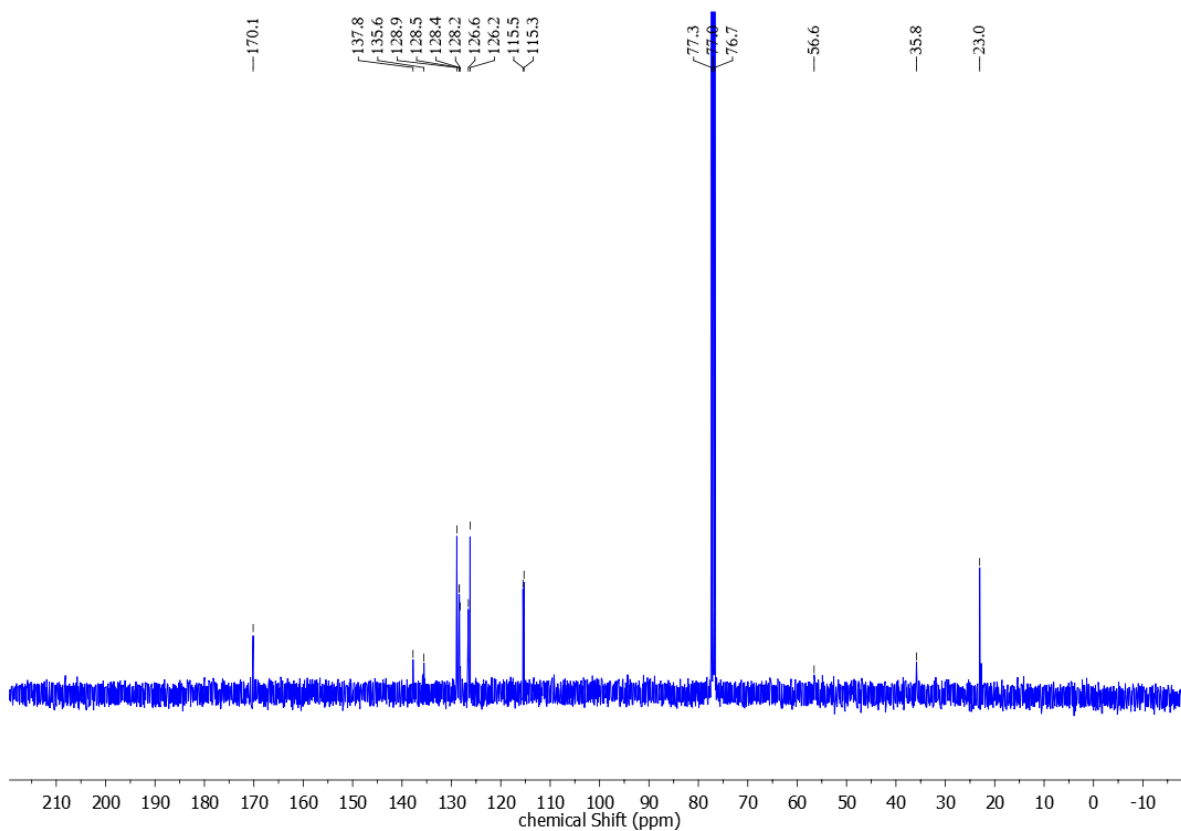
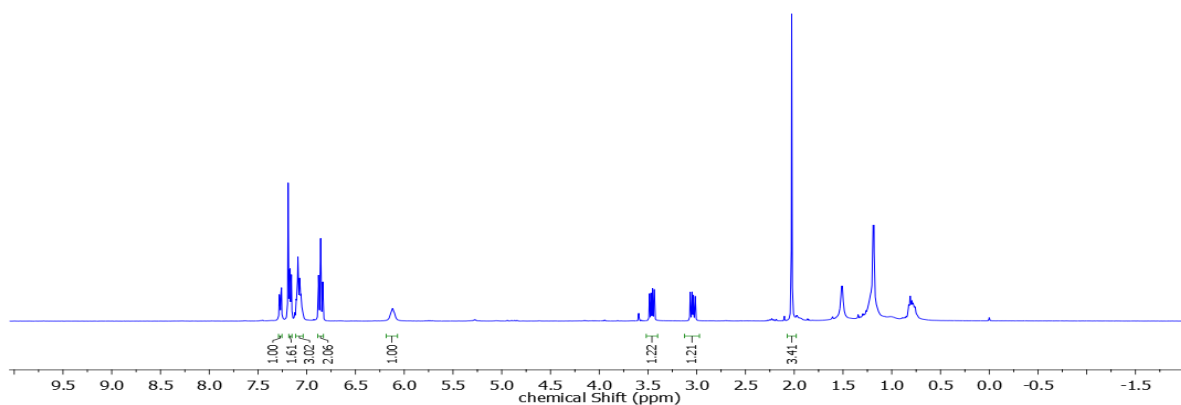
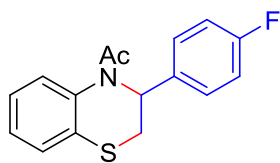
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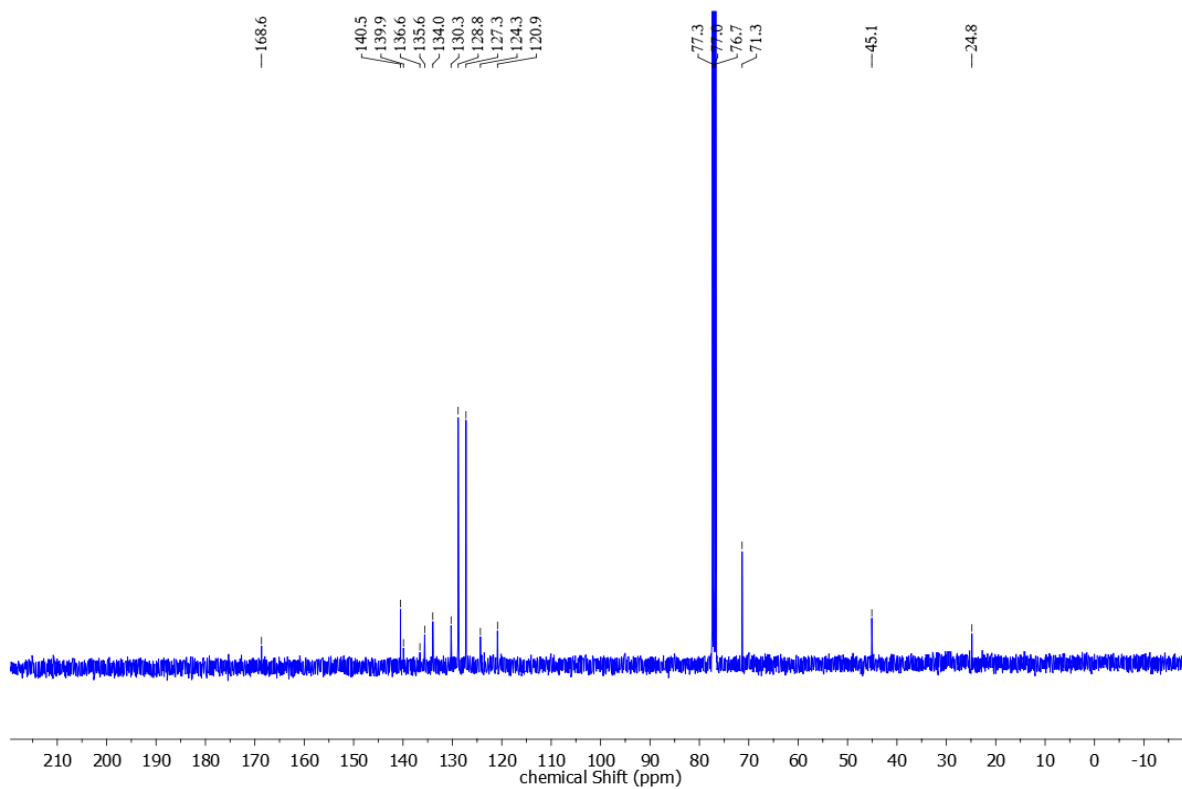
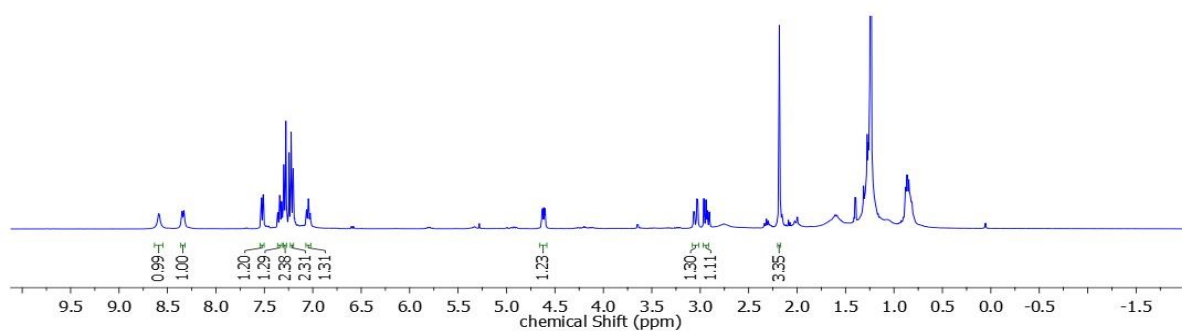
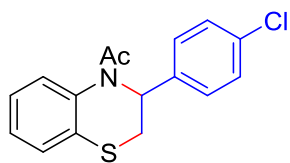
Spectral data of compound 2b



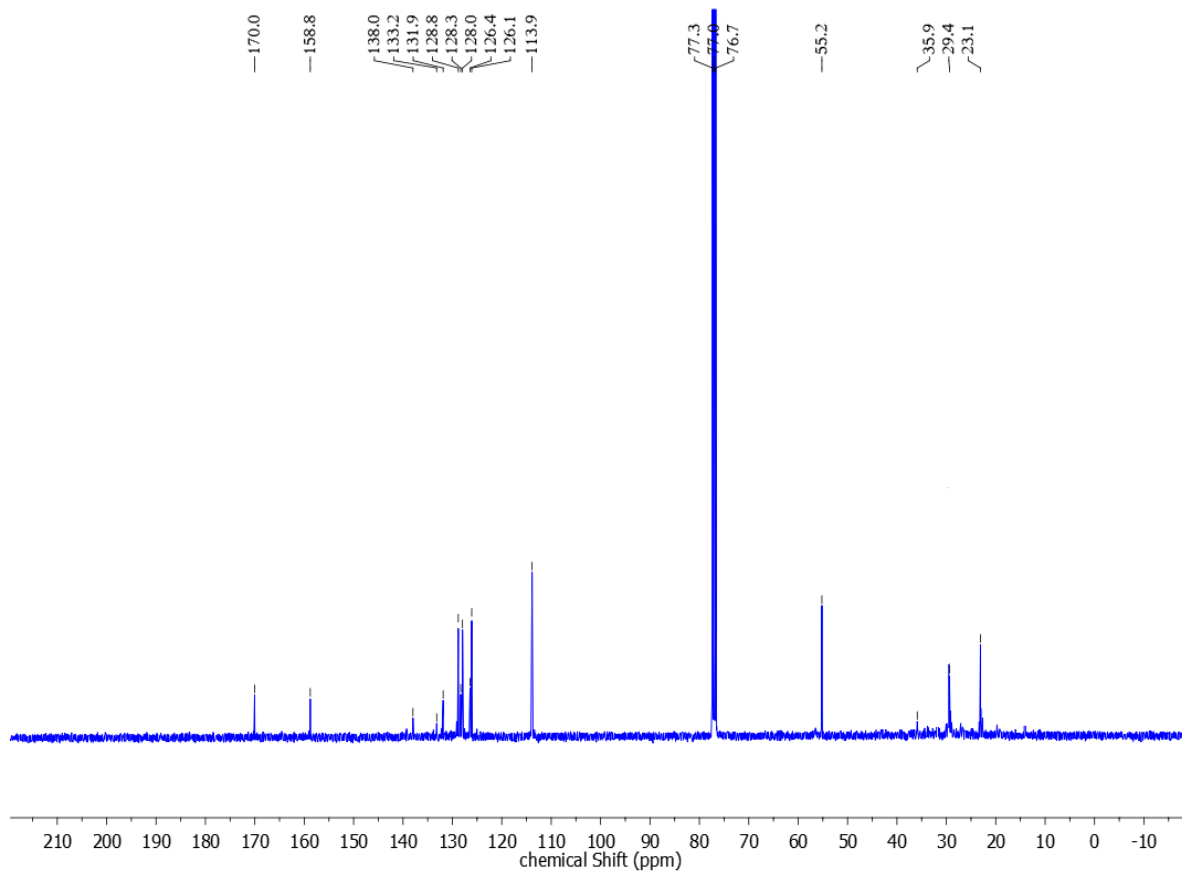
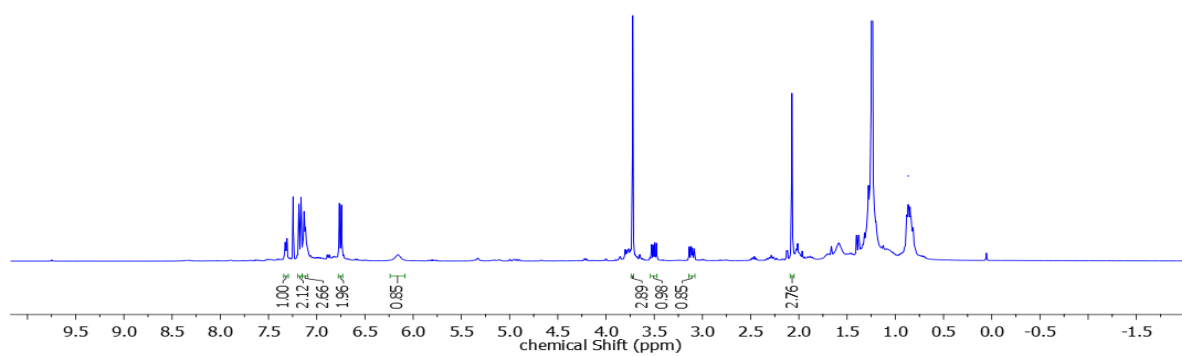
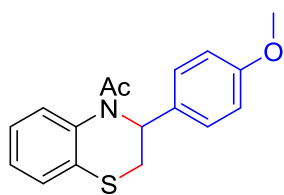
Spectral data of compound 2c



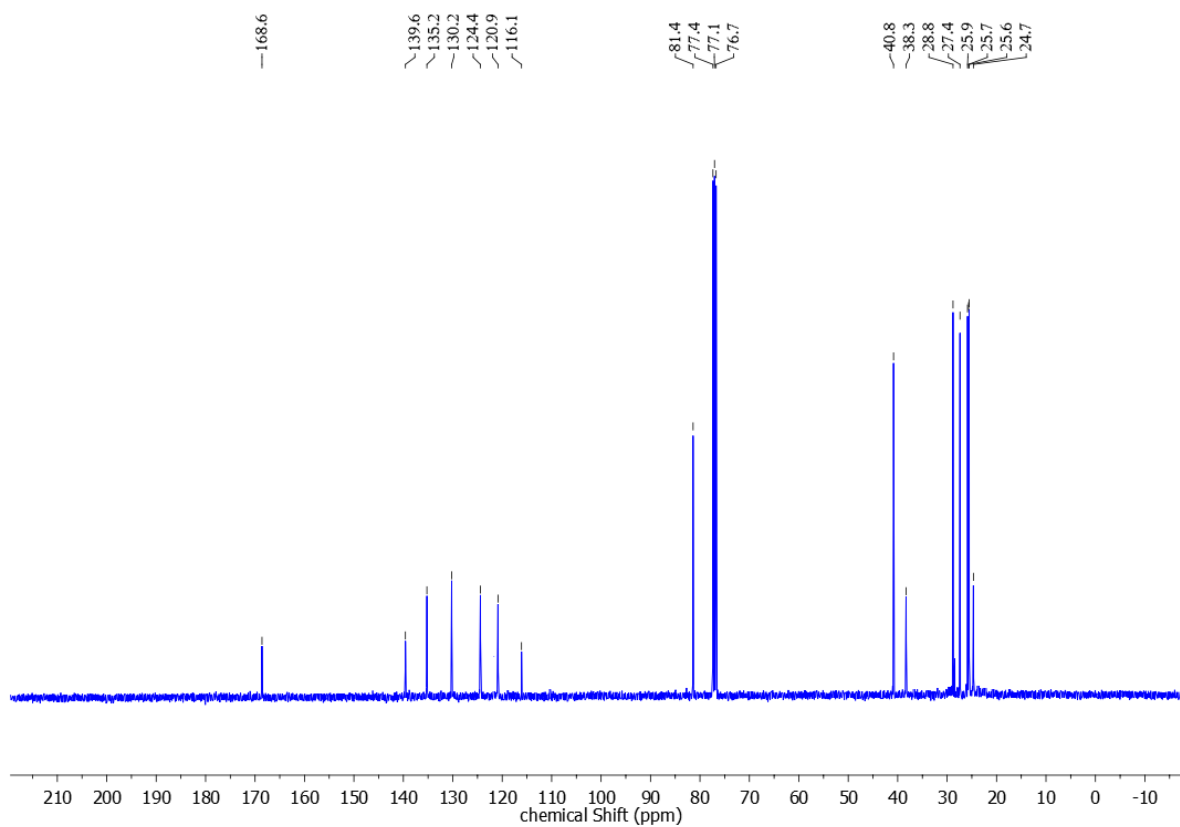
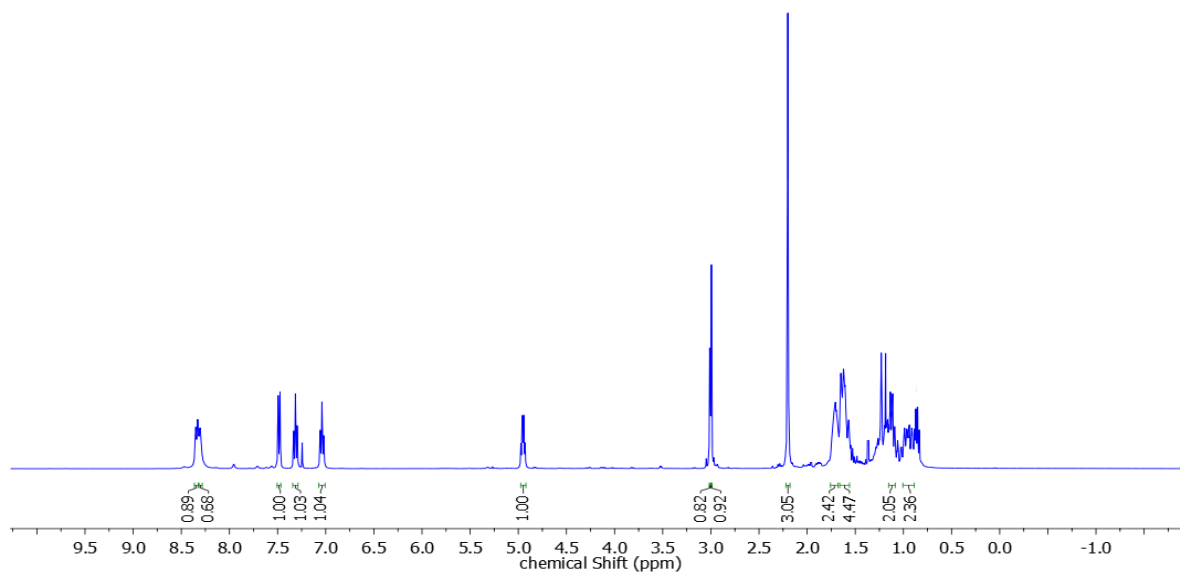
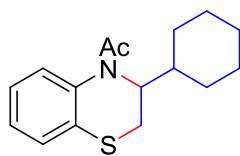
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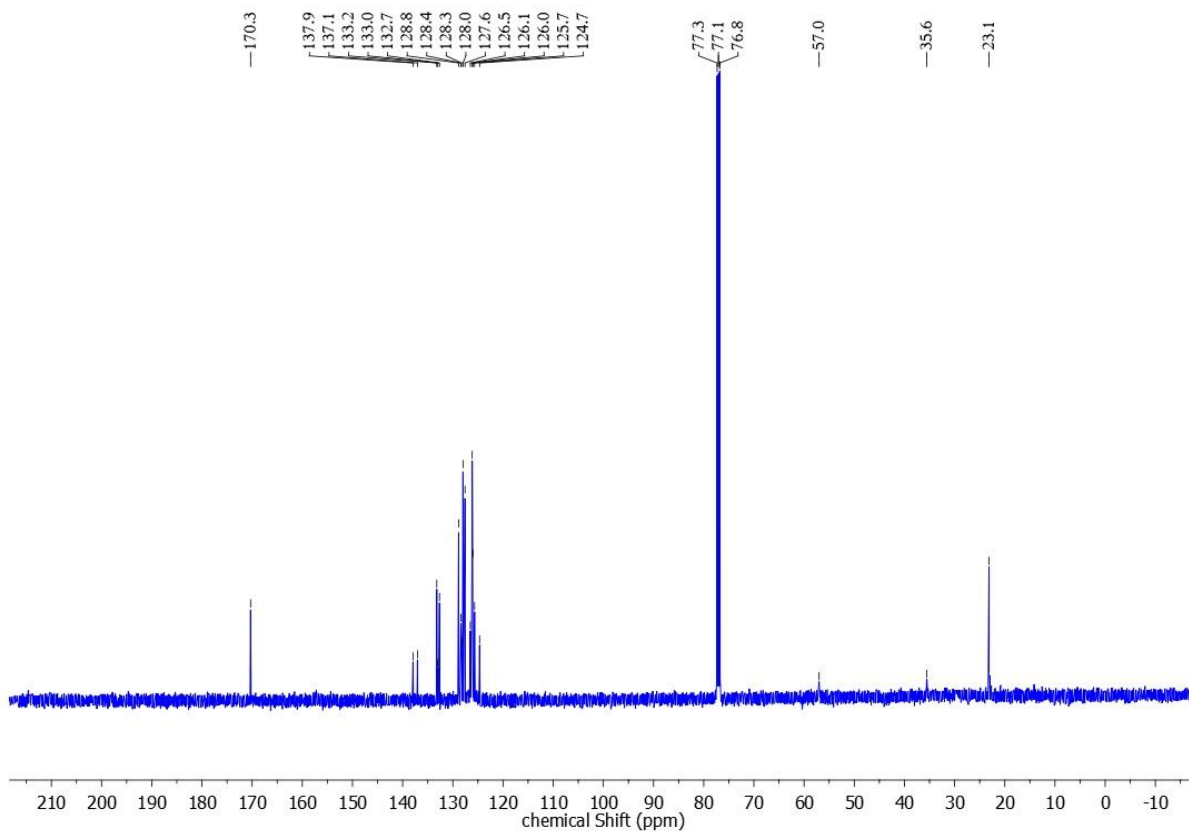
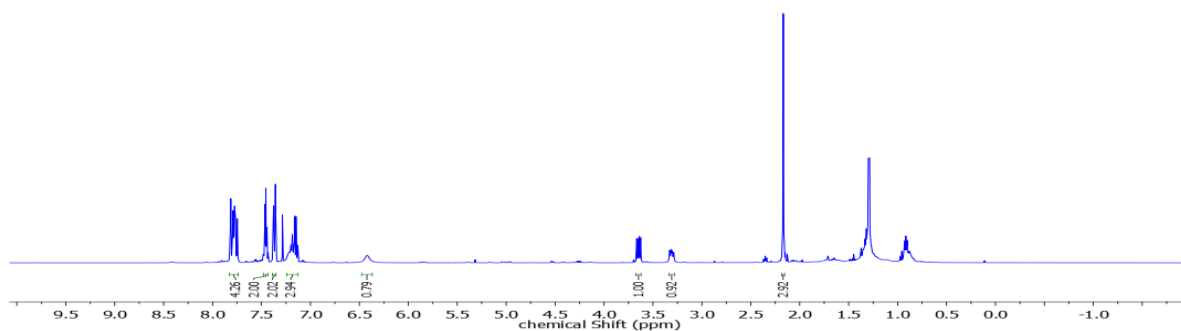
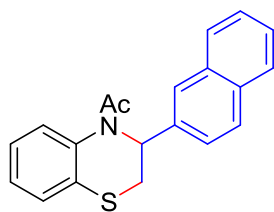
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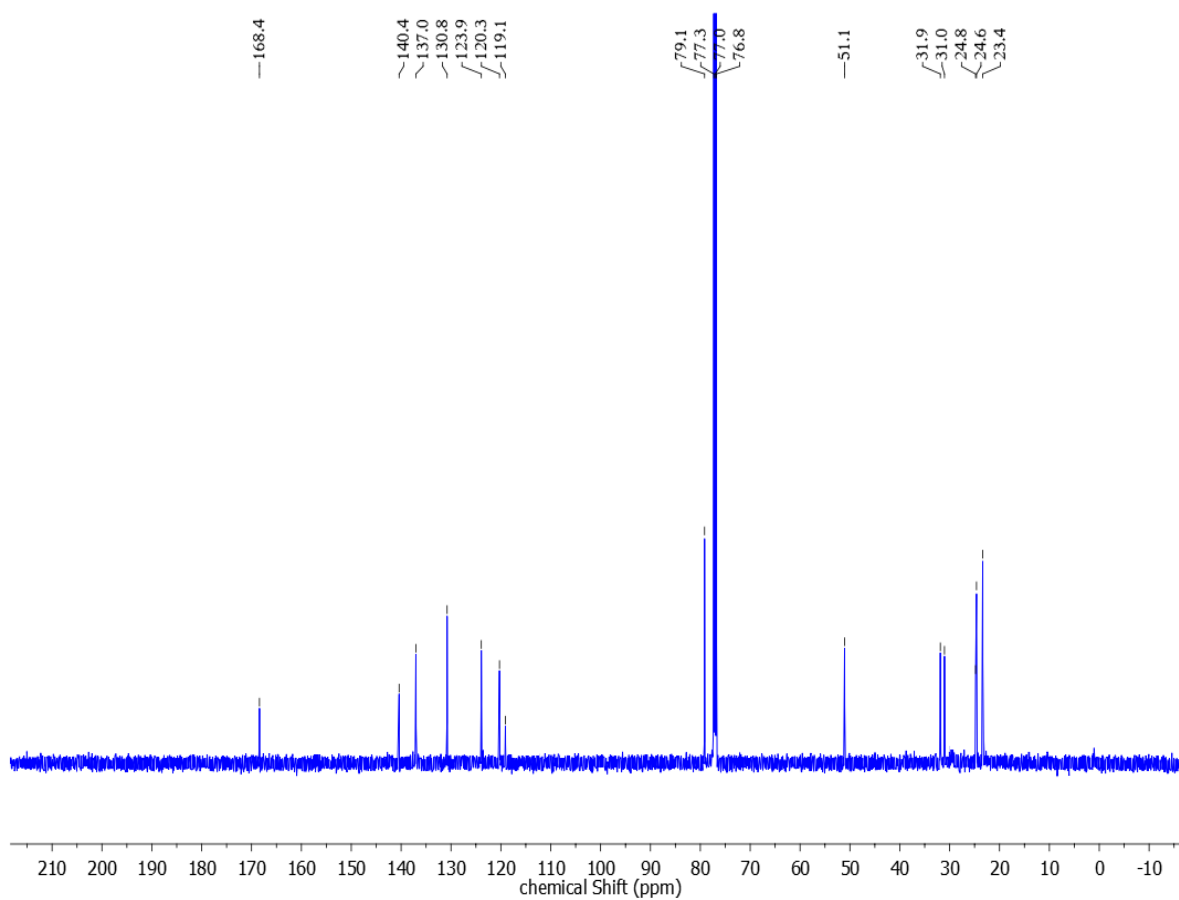
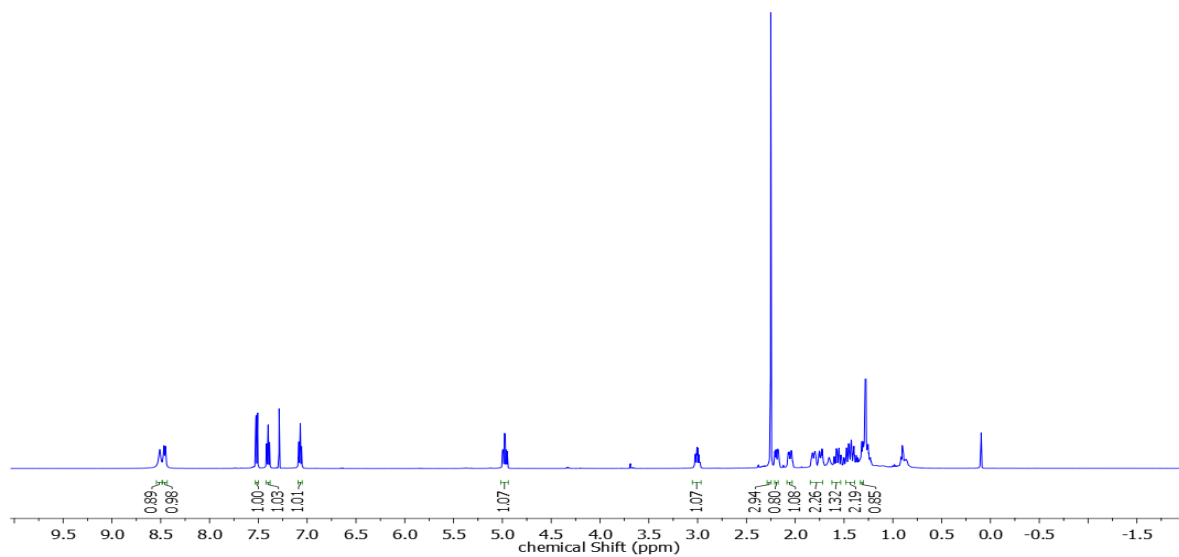
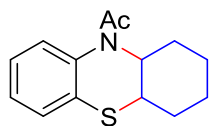
Spectral data of compound 2f



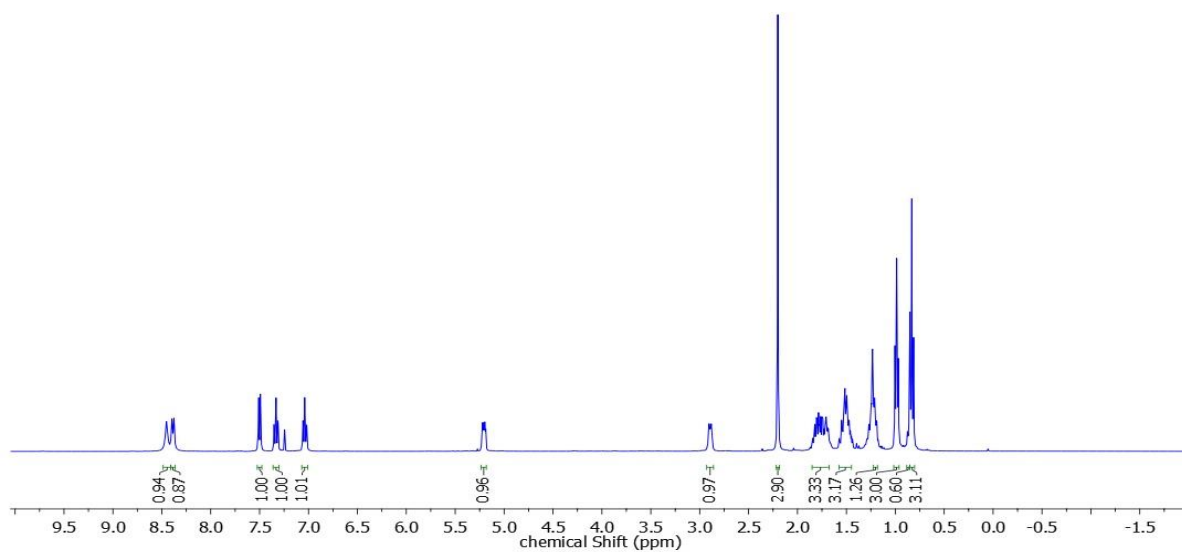
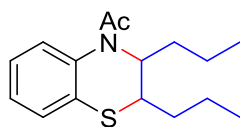
Spectral data of compound 2g



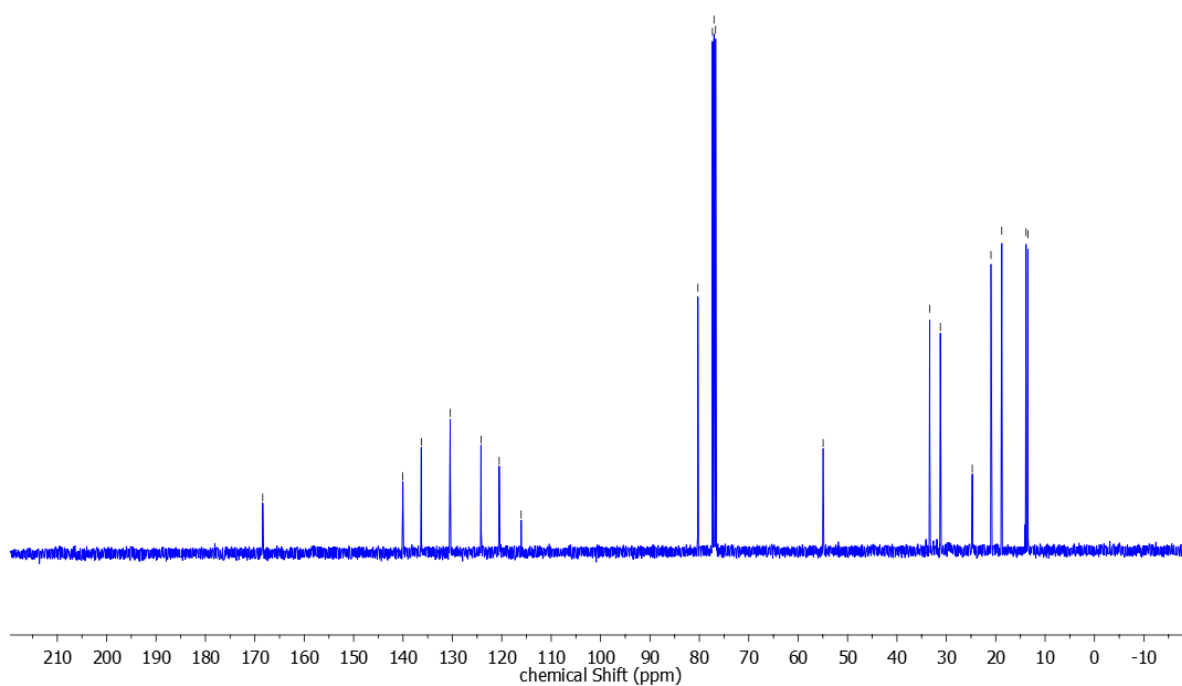
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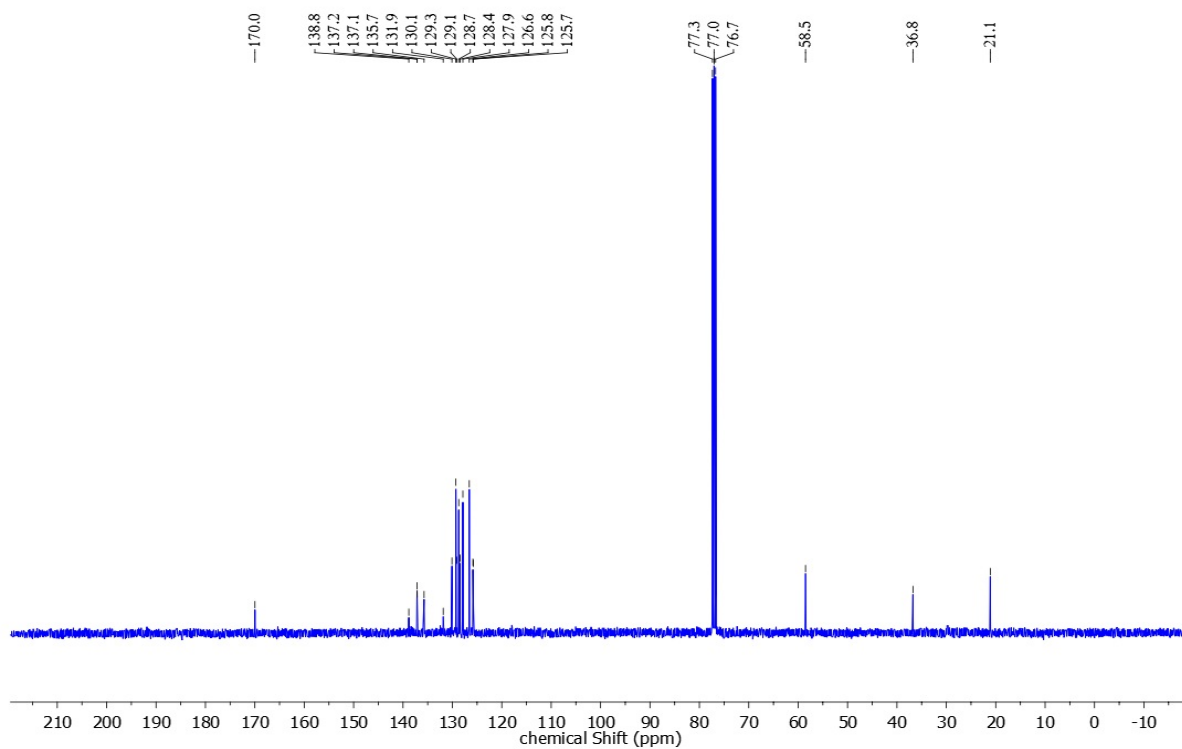
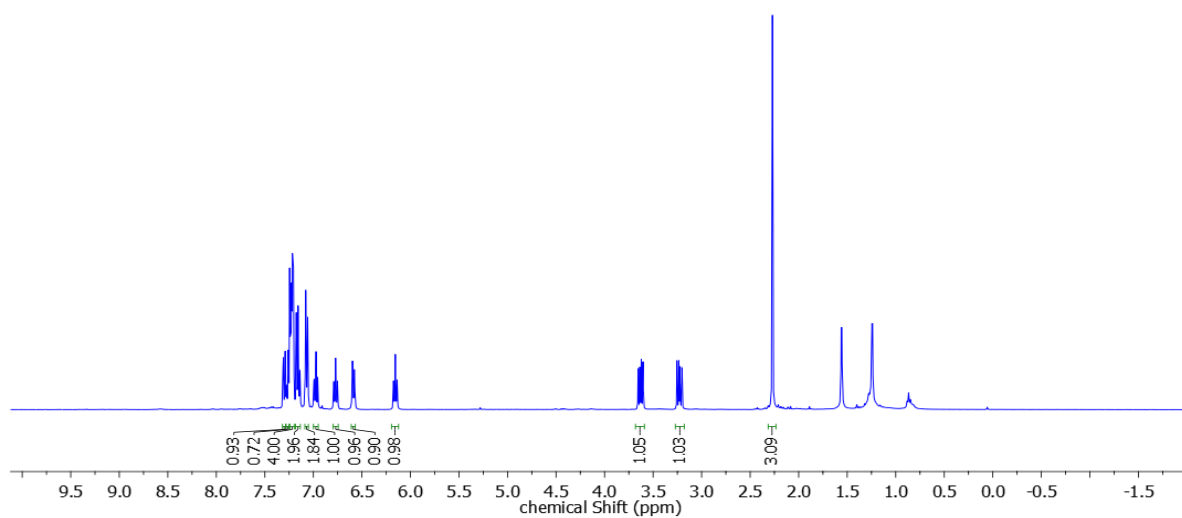
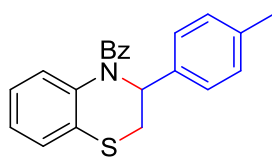
Spectral data of compound 2i



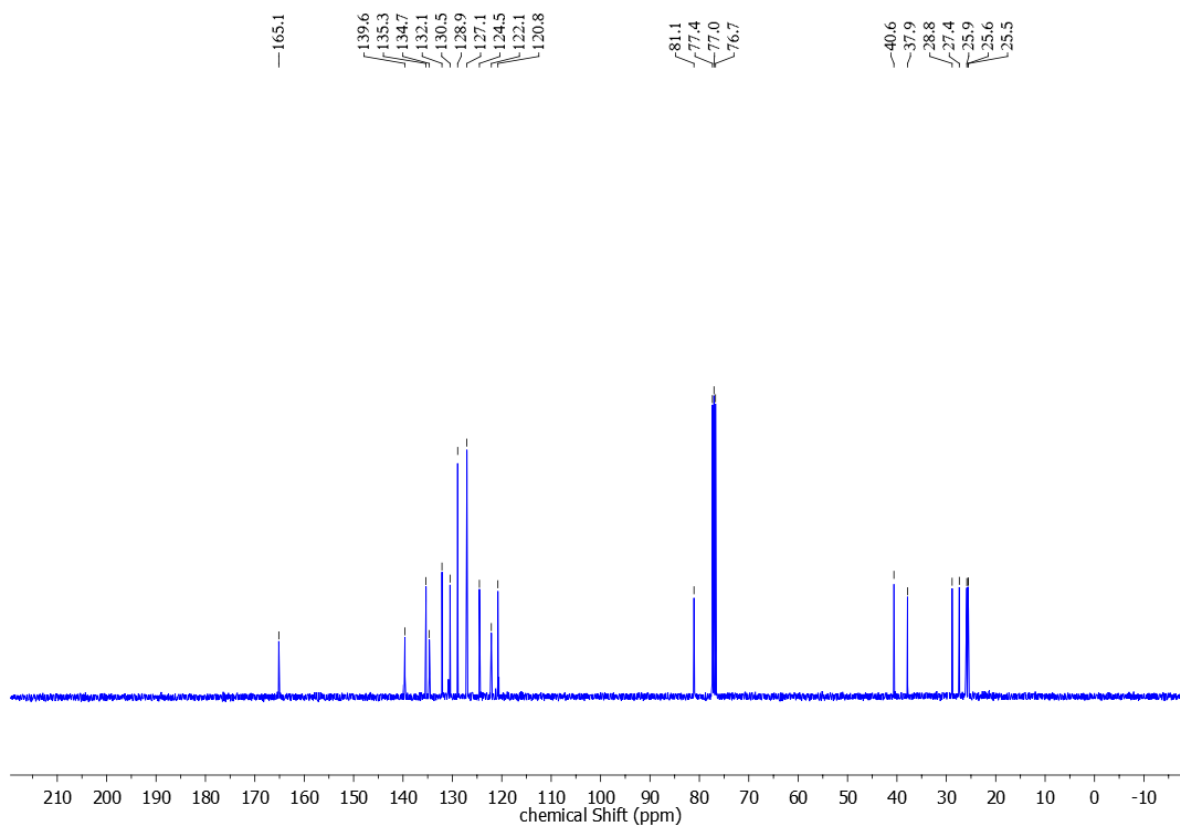
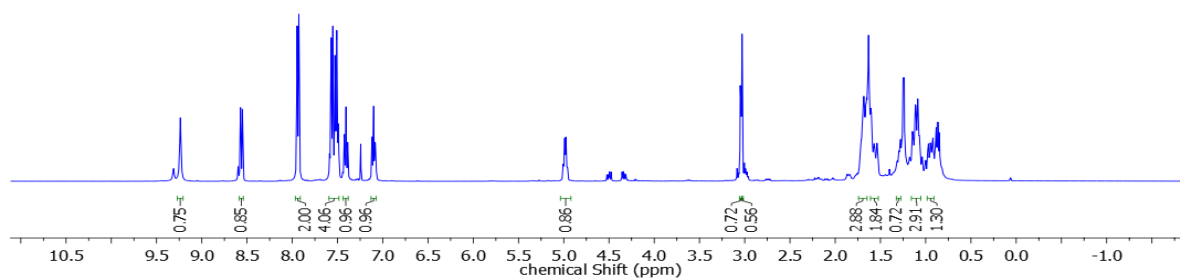
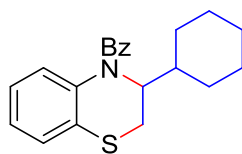
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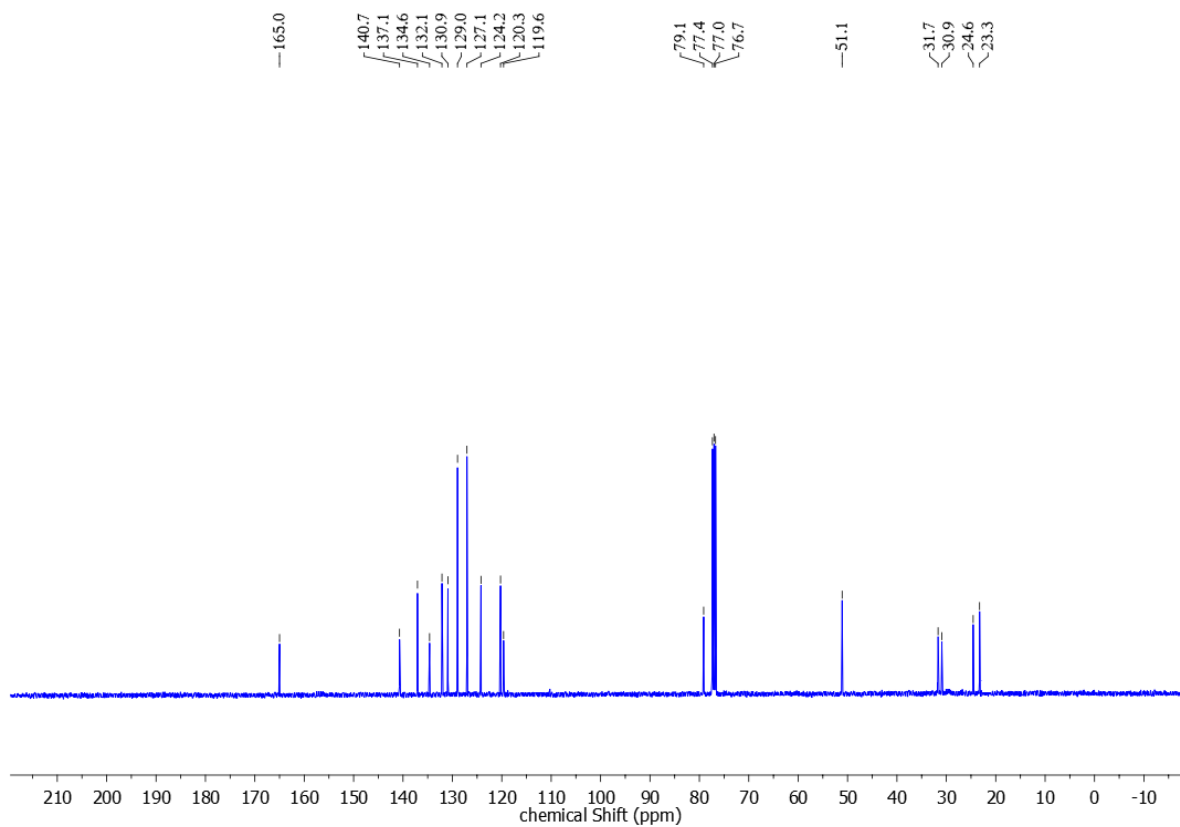
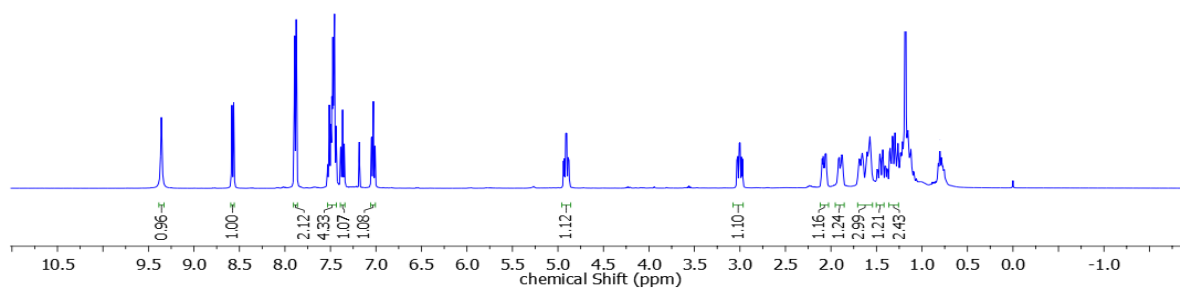
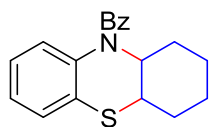
Spectral data of compound 2j



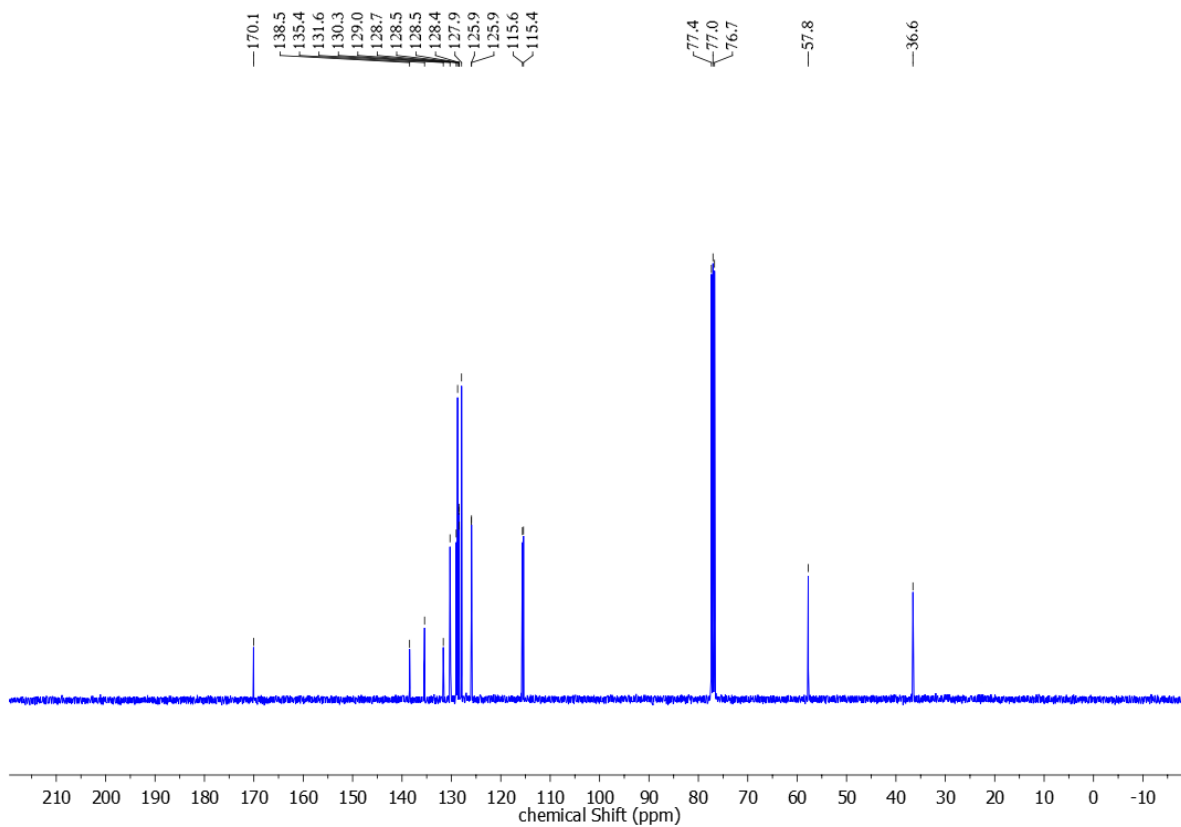
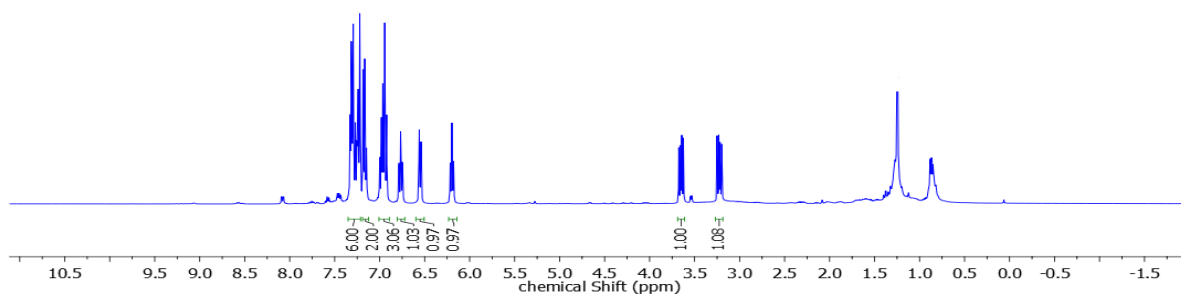
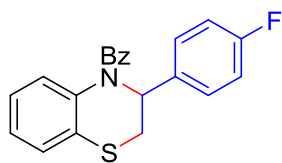
Spectral data of compound 2k



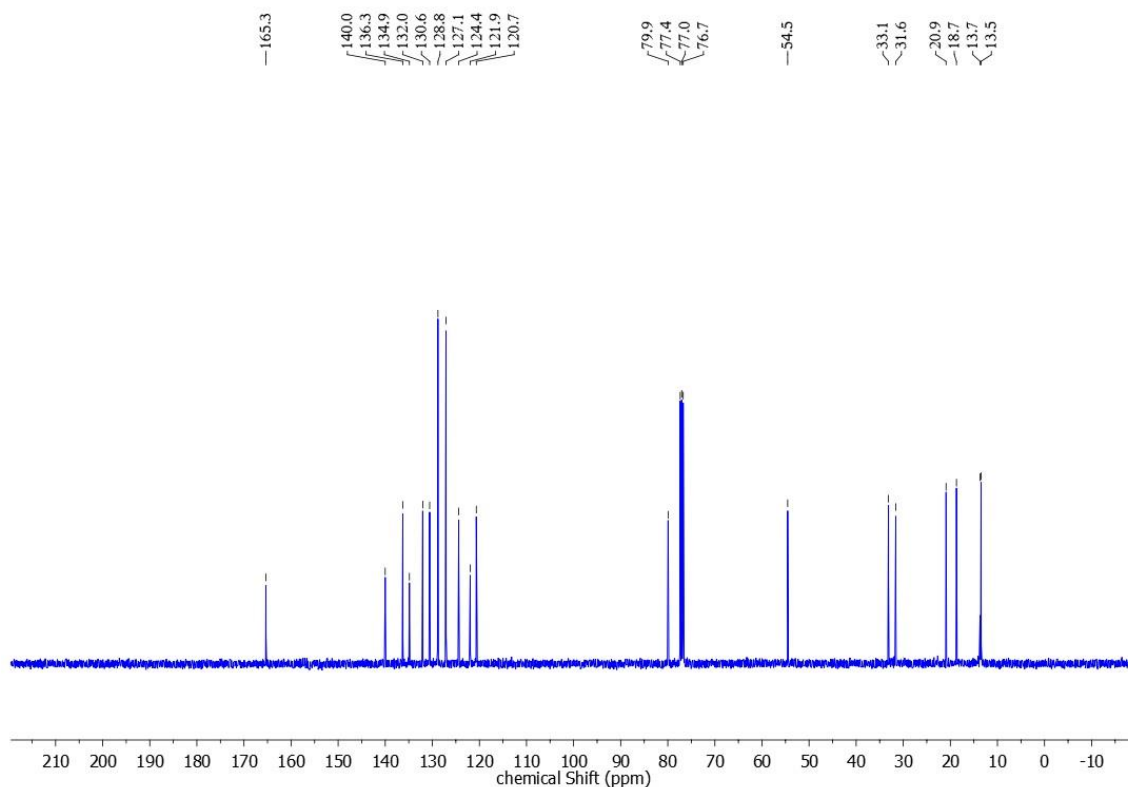
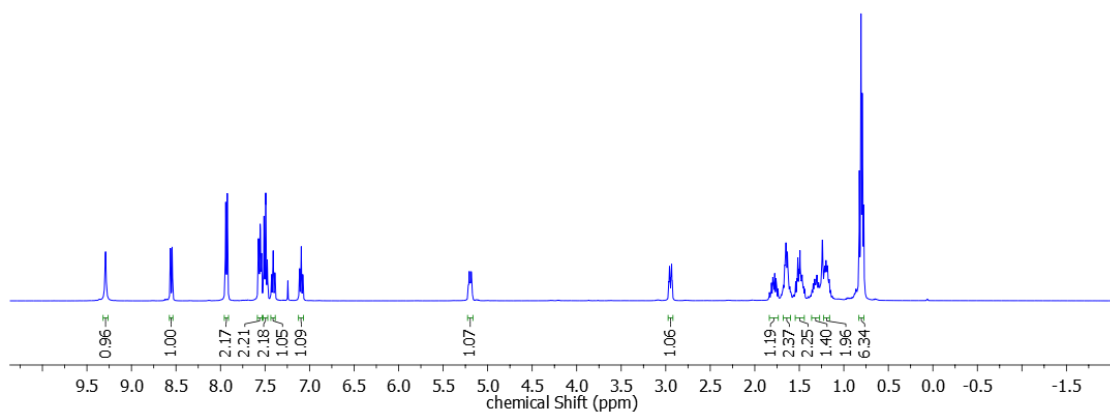
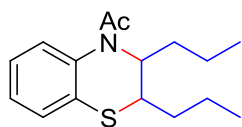
Spectral data of compound 21



Spectral data of compound 2m

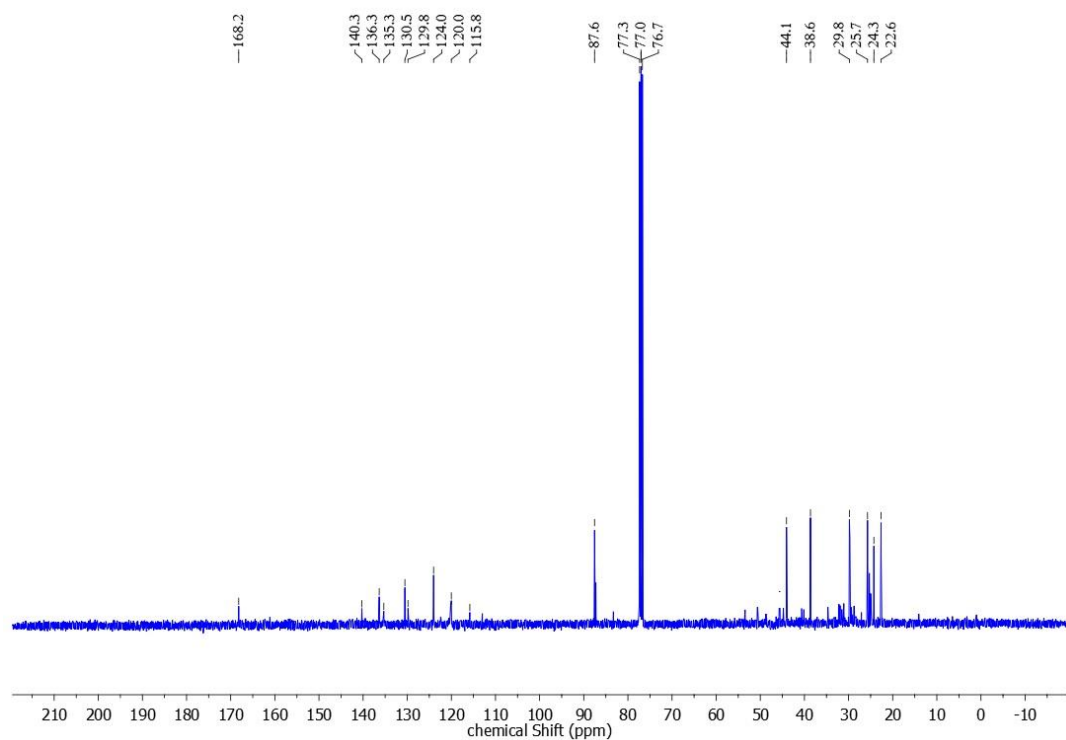
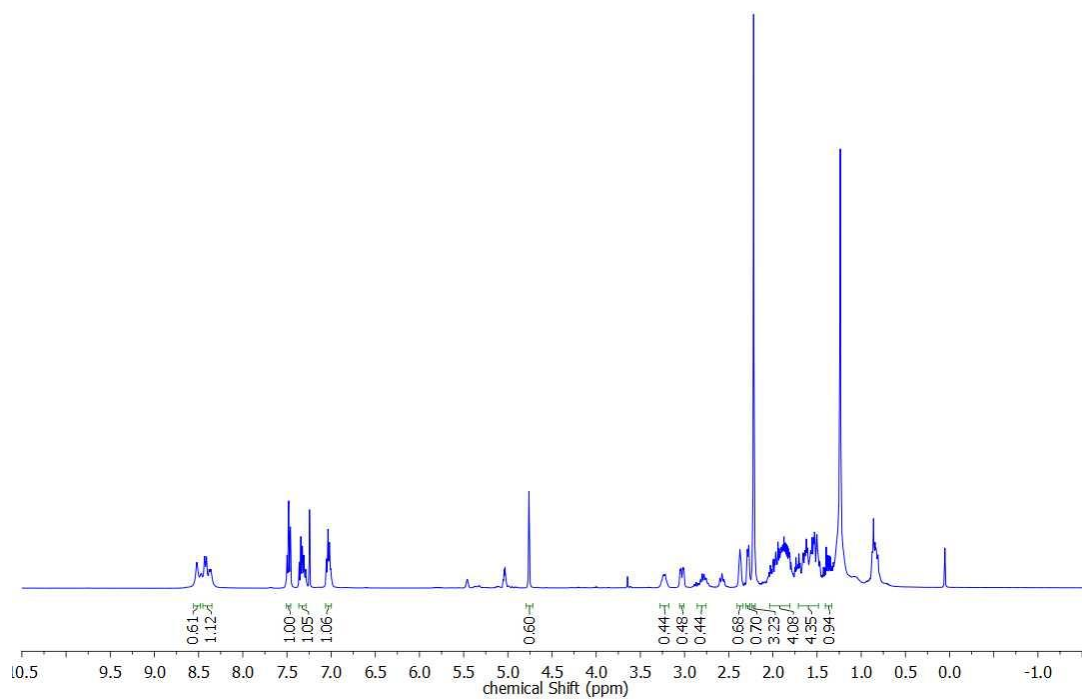
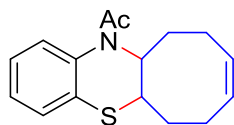


Spectral data of compound 2n

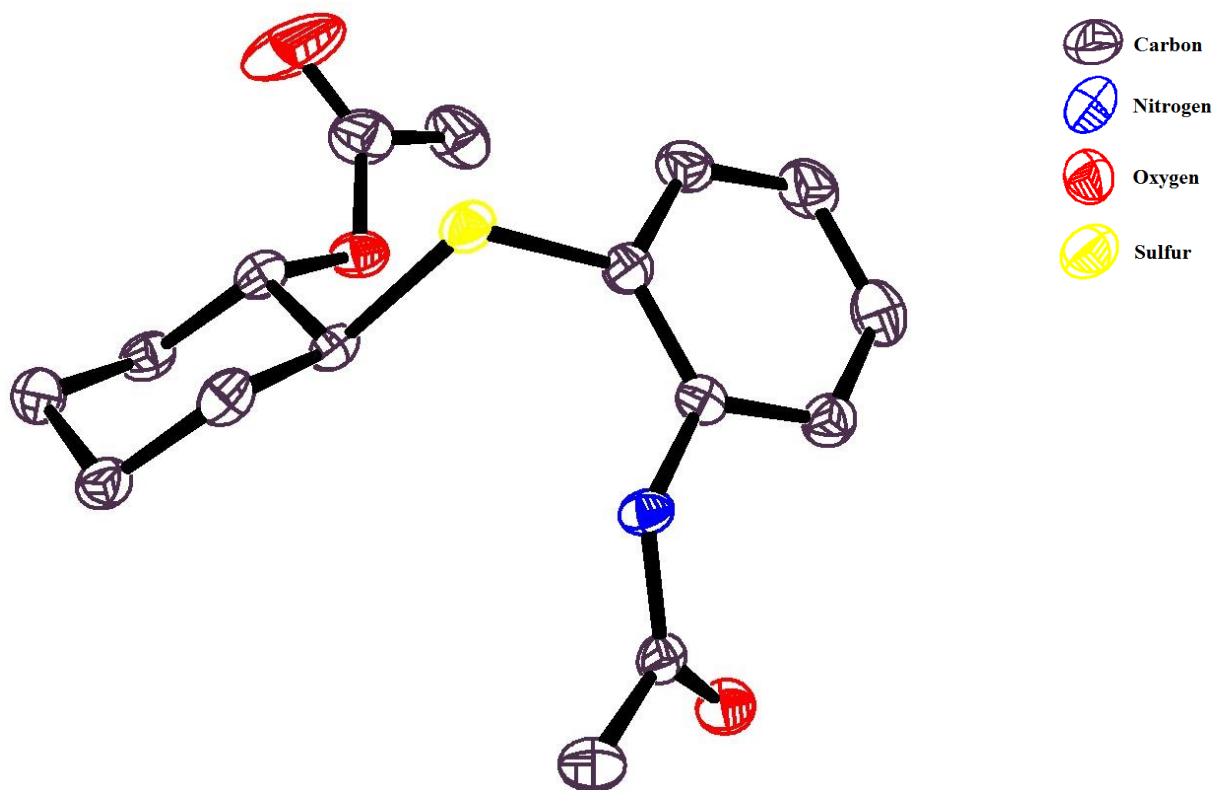


Spectral data of the inseparable mixture from 1m:

Probable compound:



Crystal structure of 1h with 50% ellipsoidal probability



Packing diagram for 1h

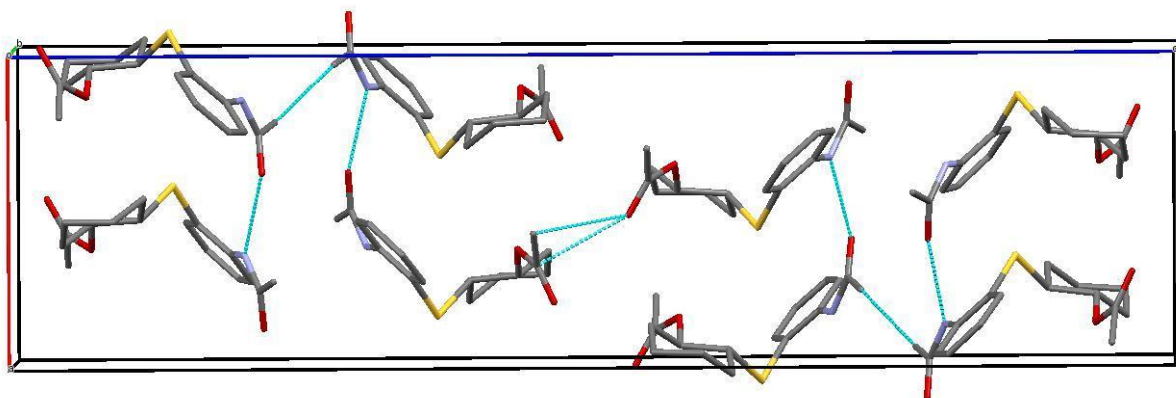


Table 1. Crystal data and structure refinement for 1h.

Identification code	DP-521 (CCDC No. 1409837)	
Empirical formula	C ₁₆ H ₂₁ N ₁ O ₃ S ₁	
Formula weight	307.40	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P n a 2 ₁	
Unit cell dimensions	a = 9.3777(6) Å	α = 90°.
	b = 9.5320(6) Å	β = 90°.
	c = 34.929(2) Å	γ = 90°.
Volume	3122.2(3) Å ³	
Z	8	
Density (calculated)	1.308 Mg/m ³	
Absorption coefficient	0.217 mm ⁻¹	
F(000)	1312	
Theta range for data collection	1.166 to 26.055°.	
Index ranges	-11 ≤ h ≤ 11, -10 ≤ k ≤ 11, -42 ≤ l ≤ 43	
Reflections collected	22493	
Independent reflections	6161 [R(int) = 0.0441]	
Completeness to theta = 25.242°	100.0 %	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	6161 / 1 / 383	
Goodness-of-fit on F ²	1.072	
Final R indices [I > 2σ(I)]	R1 = 0.0391, wR2 = 0.0849	
R indices (all data)	R1 = 0.0528, wR2 = 0.1055	
Absolute structure parameter	0.01(4)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.200 and -0.204 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 1h. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

–	x	y	z	U(eq)
S(1)	8515(1)	5843(1)	3629(1)	24(1)
S(2)	5739(1)	5875(1)	6379(1)	25(1)
O(5)	6379(3)	5355(3)	4319(1)	23(1)
O(2)	3703(3)	5480(3)	5670(1)	27(1)
O(4)	3962(3)	6745(3)	2858(1)	28(1)
O(1)	1202(3)	6809(3)	7160(1)	27(1)
O(6)	8034(4)	4128(3)	4641(1)	43(1)
N(1)	3536(3)	6561(3)	6995(1)	21(1)
N(2)	6304(4)	6504(3)	3015(1)	20(1)
C(24)	4578(4)	6736(4)	5690(1)	23(1)
C(15)	6880(5)	4192(4)	4498(1)	26(1)
C(31)	2414(4)	7290(4)	7132(1)	21(1)
C(2)	6313(4)	5091(4)	3134(1)	20(1)
C(7)	5171(4)	7237(5)	2876(1)	21(1)
C(23)	4697(4)	7168(4)	6106(1)	21(1)
C(25)	3911(5)	7852(5)	5445(1)	26(1)
C(1)	7266(4)	4665(4)	3421(1)	21(1)
O(3)	5270(5)	4449(4)	5284(1)	79(2)
C(18)	3521(4)	5155(4)	6870(1)	19(1)
C(28)	5420(5)	8590(4)	6147(1)	24(1)
C(17)	4459(4)	4718(5)	6578(1)	22(1)
C(22)	4472(5)	3320(4)	6468(1)	29(1)
C(20)	2646(5)	2781(5)	6920(2)	33(1)
C(32)	2755(5)	8762(4)	7258(1)	27(1)
C(9)	7417(4)	7083(4)	3901(1)	19(1)

C(19)	2609(5)	4176(4)	7037(1)	25(1)
C(10)	7252(4)	6604(4)	4317(1)	20(1)
C(14)	8126(5)	8527(4)	3880(1)	24(1)
C(13)	7378(5)	9611(4)	4131(1)	26(1)
C(3)	5418(5)	4104(4)	2967(1)	25(1)
C(5)	6436(5)	2281(5)	3355(2)	31(1)
C(26)	4700(5)	9237(4)	5478(1)	29(1)
C(27)	4721(5)	9708(5)	5894(1)	26(1)
C(8)	5502(5)	8695(4)	2747(1)	28(1)
C(6)	7324(5)	3257(4)	3524(1)	27(1)
C(12)	7298(5)	9108(4)	4544(1)	27(1)
C(4)	5481(5)	2712(5)	3077(2)	30(1)
C(11)	6531(5)	7705(5)	4565(2)	25(1)
C(29)	4169(6)	4418(5)	5453(2)	37(1)
C(21)	3574(5)	2345(5)	6636(2)	34(1)
C(16)	5812(5)	3036(5)	4476(2)	37(1)
C(30)	3124(6)	3213(5)	5453(2)	42(1)

Table 3. Selected bond lengths [\AA for 1h.

S1—C1	1.778 (4)	C32—H32A	0.96
S1—C9	1.834 (4)	C32—H32B	0.96
S2—C17	1.772 (4)	C32—H32C	0.96
S2—C23	1.839 (4)	C9—C14	1.531 (6)
O5—C15	1.355 (5)	C9—C10	1.531 (6)
O5—C10	1.445 (5)	C9—H9	0.98
O2—C29	1.338 (5)	C19—H19	0.93
O2—C24	1.453 (5)	C10—C11	1.519 (6)
O4—C7	1.229 (5)	C10—H10	0.98
O1—C31	1.229 (5)	C14—C13	1.526 (6)

O6—C15	1.195 (5)	C14—H14A	0.97
N1—C31	1.349 (5)	C14—H14B	0.97
N1—C18	1.409 (5)	C13—C12	1.524 (6)
N1—H1	0.86	C13—H13A	0.97
N2—C7	1.360 (5)	C13—H13B	0.97
N2—C2	1.409 (5)	C3—C4	1.382 (6)
N2—H2	0.86	C3—H3	0.93
C24—C25	1.501 (6)	C5—C6	1.381 (7)
C24—C23	1.516 (6)	C5—C4	1.384 (7)
C24—H24	0.98	C5—H5	0.93
C15—C16	1.491 (6)	C26—C27	1.520 (6)
C31—C32	1.505 (6)	C26—H26A	0.97
C2—C3	1.390 (6)	C26—H26B	0.97
C2—C1	1.403 (6)	C27—H27A	0.97
C7—C8	1.494 (6)	C27—H27B	0.97
C23—C28	1.523 (6)	C8—H8A	0.96
C23—H23	0.98	C8—H8B	0.96
C25—C26	1.518 (6)	C8—H8C	0.96
C25—H25A	0.97	C6—H6	0.93
C25—H25B	0.97	C12—C11	1.521 (6)
C1—C6	1.391 (6)	C12—H12A	0.97
O3—C29	1.189 (6)	C12—H12B	0.97
C18—C19	1.394 (6)	C4—H4	0.93
C18—C17	1.409 (6)	C11—H11A	0.97
C28—C27	1.531 (6)	C11—H11B	0.97
C28—H28A	0.97	C29—C30	1.510 (7)
C28—H28B	0.97	C21—H21	0.93
C17—C22	1.387 (6)	C16—H16A	0.96
C22—C21	1.384 (6)	C16—H16B	0.96
C22—H22	0.93	C16—H16C	0.96

C20—C21	1.384 (7)	C30—H30A	0.96
C20—C19	1.392 (6)	C30—H30B	0.96
C20—H20	0.93	C30—H30C	0.96

Table 4. Selected bond angles [°] for 1h.

C1—S1—C9	104.41 (19)	O5—C10—C9	107.9 (3)
C17—S2—C23	105.10 (19)	C11—C10—C9	112.4 (4)
C15—O5—C10	118.7 (3)	O5—C10—H10	109.4
C29—O2—C24	117.7 (4)	C11—C10—H10	109.4
C31—N1—C18	126.3 (3)	C9—C10—H10	109.4
C31—N1—H1	116.9	C13—C14—C9	112.4 (4)
C18—N1—H1	116.9	C13—C14—H14A	109.1
C7—N2—C2	126.9 (3)	C9—C14—H14A	109.1
C7—N2—H2	116.5	C13—C14—H14B	109.1
C2—N2—H2	116.5	C9—C14—H14B	109.1
O2—C24—C25	108.8 (3)	H14A—C14—H14B	107.8
O2—C24—C23	108.1 (3)	C12—C13—C14	110.7 (4)
C25—C24—C23	112.6 (4)	C12—C13—H13A	109.5
O2—C24—H24	109.1	C14—C13—H13A	109.5
C25—C24—H24	109.1	C12—C13—H13B	109.5
C23—C24—H24	109.1	C14—C13—H13B	109.5
O6—C15—O5	123.3 (4)	H13A—C13—H13B	108.1
O6—C15—C16	126.3 (4)	C4—C3—C2	120.5 (4)
O5—C15—C16	110.4 (4)	C4—C3—H3	119.8
O1—C31—N1	123.9 (4)	C2—C3—H3	119.8
O1—C31—C32	121.3 (4)	C6—C5—C4	119.4 (4)
N1—C31—C32	114.7 (4)	C6—C5—H5	120.3
C3—C2—C1	119.3 (4)	C4—C5—H5	120.3
C3—C2—N2	121.3 (4)	C25—C26—C27	109.6 (4)
C1—C2—N2	119.4 (4)	C25—C26—H26A	109.7
O4—C7—N2	122.9 (4)	C27—C26—H26A	109.7

O4—C7—C8	122.1 (4)	C25—C26—H26B	109.7
N2—C7—C8	115.1 (4)	C27—C26—H26B	109.7
C24—C23—C28	111.4 (4)	H26A—C26—H26B	108.2
C24—C23—S2	110.8 (3)	C26—C27—C28	110.5 (4)
C28—C23—S2	108.2 (3)	C26—C27—H27A	109.5
C24—C23—H23	108.8	C28—C27—H27A	109.5
C28—C23—H23	108.8	C26—C27—H27B	109.5
S2—C23—H23	108.8	C28—C27—H27B	109.5
C24—C25—C26	111.8 (4)	H27A—C27—H27B	108.1
C24—C25—H25A	109.3	C7—C8—H8A	109.5
C26—C25—H25A	109.3	C7—C8—H8B	109.5
C24—C25—H25B	109.3	H8A—C8—H8B	109.5
C26—C25—H25B	109.3	C7—C8—H8C	109.5
H25A—C25—H25B	107.9	H8A—C8—H8C	109.5
C6—C1—C2	119.3 (4)	H8B—C8—H8C	109.5
C6—C1—S1	118.6 (3)	C5—C6—C1	121.0 (4)
C2—C1—S1	121.9 (3)	C5—C6—H6	119.5
C19—C18—C17	119.2 (4)	C1—C6—H6	119.5
C19—C18—N1	120.8 (4)	C11—C12—C13	110.2 (4)
C17—C18—N1	119.9 (4)	C11—C12—H12A	109.6
C23—C28—C27	112.1 (4)	C13—C12—H12A	109.6
C23—C28—H28A	109.2	C11—C12—H12B	109.6
C27—C28—H28A	109.2	C13—C12—H12B	109.6
C23—C28—H28B	109.2	H12A—C12—H12B	108.1
C27—C28—H28B	109.2	C3—C4—C5	120.5 (4)
H28A—C28—H28B	107.9	C3—C4—H4	119.7
C22—C17—C18	119.3 (4)	C5—C4—H4	119.7
C22—C17—S2	118.9 (3)	C10—C11—C12	111.7 (4)
C18—C17—S2	121.5 (3)	C10—C11—H11A	109.3
C21—C22—C17	121.5 (4)	C12—C11—H11A	109.3

C21—C22—H22	119.2	C10—C11—H11B	109.3
C17—C22—H22	119.2	C12—C11—H11B	109.3
C21—C20—C19	120.9 (4)	H11A—C11—H11B	107.9
C21—C20—H20	119.6	O3—C29—O2	123.1 (5)
C19—C20—H20	119.6	O3—C29—C30	125.6 (5)
C31—C32—H32A	109.5	O2—C29—C30	111.3 (4)
C31—C32—H32B	109.5	C20—C21—C22	118.9 (4)
H32A—C32—H32B	109.5	C20—C21—H21	120.5
C31—C32—H32C	109.5	C22—C21—H21	120.5
H32A—C32—H32C	109.5	C15—C16—H16A	109.5
H32B—C32—H32C	109.5	C15—C16—H16B	109.5
C14—C9—C10	111.0 (4)	H16A—C16—H16B	109.5
C14—C9—S1	108.1 (3)	C15—C16—H16C	109.5
C10—C9—S1	110.9 (3)	H16A—C16—H16C	109.5
C14—C9—H9	108.9	H16B—C16—H16C	109.5
C10—C9—H9	108.9	C29—C30—H30A	109.5
S1—C9—H9	108.9	C29—C30—H30B	109.5
C20—C19—C18	120.1 (4)	H30A—C30—H30B	109.5
C20—C19—H19	120	C29—C30—H30C	109.5
C18—C19—H19	120	H30A—C30—H30C	109.5
O5—C10—C11	108.3 (3)	H30B—C30—H30C	109.5

Table 5. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **1h**. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^* 2U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U ₁₁	U ₂₂	U ₃₃	U ₂₃	U ₁₃	U ₁₂
$\overline{S(1)}$	18(1)	31(1)	22(1)	-3(1)	-1(1)	3(1)
S(2)	18(1)	34(1)	23(1)	2(1)	1(1)	3(1)
O(5)	24(2)	21(2)	24(2)	4(1)	-2(1)	-2(1)
O(2)	26(2)	26(2)	29(2)	-9(1)	1(1)	0(1)

O(4)	20(2)	30(2)	35(2)	-1(1)	-2(2)	2(1)
O(1)	18(2)	32(2)	32(2)	2(1)	1(1)	1(1)
O(6)	36(2)	41(2)	52(2)	16(2)	-7(2)	9(2)
N(1)	15(2)	22(2)	24(2)	-3(2)	3(2)	-1(2)
N(2)	16(2)	22(2)	22(2)	0(2)	-2(2)	-2(2)
C(24)	18(2)	29(2)	21(3)	-4(2)	0(2)	-1(2)
C(15)	35(3)	25(2)	17(2)	2(2)	3(2)	8(2)
C(31)	20(2)	27(2)	15(3)	2(2)	-2(2)	4(2)
C(2)	21(2)	18(2)	20(2)	-2(2)	2(2)	1(2)
C(7)	20(2)	25(2)	17(3)	-1(2)	2(2)	1(2)
C(23)	17(2)	29(2)	17(2)	-1(2)	3(2)	1(2)
C(25)	24(2)	35(2)	18(3)	-4(2)	-1(2)	3(2)
C(1)	19(2)	26(2)	17(2)	-3(2)	1(2)	6(2)
O(3)	68(3)	72(3)	97(4)	-57(3)	44(3)	-16(2)
C(18)	18(2)	21(2)	20(2)	-1(2)	-5(2)	1(2)
C(28)	24(2)	30(2)	18(2)	-6(2)	0(2)	-5(2)
C(17)	20(2)	28(2)	19(2)	2(2)	-2(2)	3(2)
C(22)	36(3)	26(2)	25(3)	-3(2)	-1(2)	7(2)
C(20)	36(3)	26(2)	36(3)	5(2)	-3(2)	-6(2)
C(32)	26(2)	27(2)	28(3)	-7(2)	-1(2)	2(2)
C(9)	15(2)	25(2)	17(2)	-1(2)	1(2)	1(2)
C(19)	26(2)	28(2)	22(3)	-1(2)	0(2)	1(2)
C(10)	19(2)	22(2)	20(2)	0(2)	-5(2)	-1(2)
C(14)	24(2)	29(2)	18(2)	4(2)	-4(2)	-4(2)
C(13)	29(2)	20(2)	30(3)	1(2)	-4(2)	0(2)
C(3)	25(2)	28(2)	22(3)	-4(2)	-3(2)	1(2)
C(5)	45(3)	21(2)	28(3)	3(2)	7(2)	1(2)
C(26)	33(3)	30(2)	25(3)	2(2)	1(2)	3(2)
C(27)	29(2)	25(2)	25(3)	-5(2)	5(2)	-1(2)

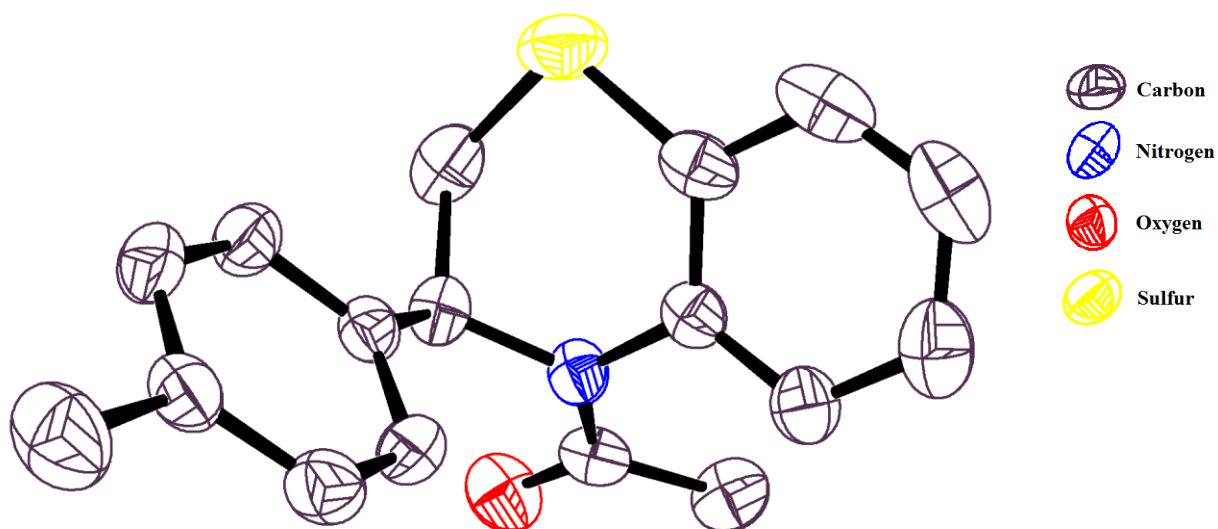
C(8)	26(2)	28(2)	29(3)	5(2)	1(2)	6(2)
C(6)	36(3)	27(2)	19(3)	1(2)	1(2)	7(2)
C(12)	35(3)	25(2)	21(3)	-5(2)	-5(2)	-1(2)
C(4)	35(2)	24(2)	33(3)	-8(2)	5(2)	-4(2)
C(11)	25(2)	30(2)	20(3)	-3(2)	-3(2)	-2(2)
C(29)	44(3)	34(3)	32(3)	-11(2)	-1(3)	4(2)
C(21)	49(3)	22(2)	30(3)	-5(2)	-2(2)	2(2)
C(16)	52(3)	22(2)	37(3)	3(2)	10(3)	-2(2)
C(30)	57(3)	27(3)	42(3)	-7(2)	-8(3)	1(2)

Table 6. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 1h.

	x	y	z	U(eq)
H(1)	4338	6996	6982	25
H(2)	7098	6950	3032	24
H(24)	5531	6522	5591	27
H(23)	3737	7225	6216	25
H(25A)	2927	7984	5522	31
H(25B)	3915	7549	5180	31
H(28A)	6417	8502	6077	29
H(28B)	5373	8887	6412	29
H(22)	5098	3031	6278	35
H(20)	2038	2133	7034	39
H(32A)	2051	9395	7159	40
H(32B)	3679	9023	7164	40
H(32C)	2753	8806	7533	40
H(9)	6471	7138	3783	23
H(19)	1976	4455	7227	30

H(10)	8193	6385	4423	24
H(14A)	9113	8445	3960	28
H(14B)	8119	8849	3616	28
H(13A)	7896	10492	4120	32
H(13B)	6423	9774	4034	32
H(3)	4772	4382	2779	30
H(5)	6478	1343	3428	38
H(26A)	4230	9941	5323	35
H(26B)	5668	9129	5386	35
H(27A)	3753	9875	5981	32
H(27B)	5247	10581	5916	32
H(8A)	5551	8717	2472	41
H(8B)	6402	8984	2852	41
H(8C)	4767	9321	2833	41
H(6)	7971	2967	3710	32
H(12A)	8254	9012	4647	33
H(12B)	6792	9795	4698	33
H(4)	4877	2061	2963	37
H(11A)	6515	7384	4829	30
H(11B)	5552	7824	4481	30
H(21)	3595	1411	6559	40
H(16A)	6212	2198	4585	55
H(16B)	5564	2868	4214	55
H(16C)	4973	3297	4617	55
H(30A)	3360	2574	5250	63
H(30B)	2177	3568	5414	63
H(30C)	3170	2733	5694	63

Crystal Structure of 2a



Packing diagram of 2a

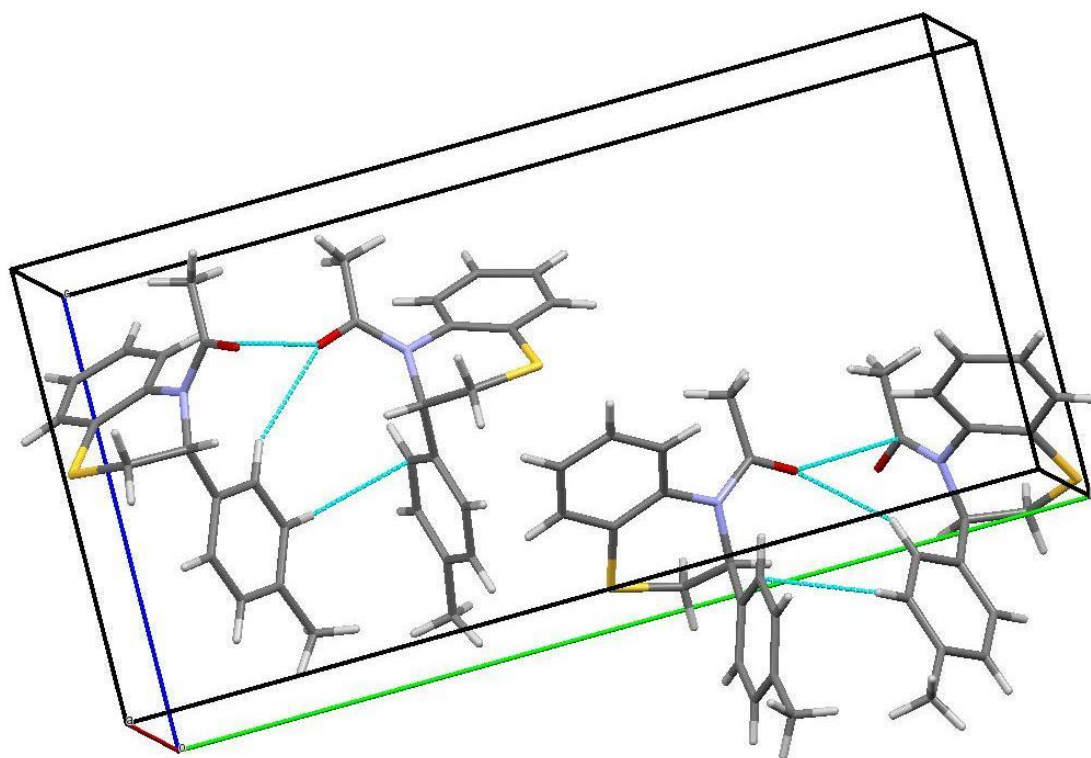


Table 7. Crystal data and structure refinement for 2a

Identification code	DP_548 (CCDC No. 1409829)	
Empirical formula	C ₁₇ H ₁₇ N O S	
Formula weight	283.37	
Temperature	301(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P n a 21	
Unit cell dimensions	a = 6.2101(4) Å	α = 90°.
	b = 21.6012(15) Å	β = 90°.
	c = 10.6587(8) Å	γ = 90°.
Volume	1429.82(17) Å ³	
Z	4	
Density (calculated)	1.316 Mg/m ³	
Absorption coefficient	0.221 mm ⁻¹	
F(000)	600	
Theta range for data collection	2.131 to 25.053°.	
Index ranges	-7 ≤ h ≤ 7, -25 ≤ k ≤ 25, -12 ≤ l ≤ 12	
Reflections collected	16452	
Independent reflections	2542 [R(int) = 0.0617]	
Completeness to theta = 25.242°	98.0 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	2542 / 1 / 183	
Goodness-of-fit on F ²	1.050	
Final R indices [I > 2σ(I)]	R1 = 0.0357, wR2 = 0.0789	
R indices (all data)	R1 = 0.0499, wR2 = 0.0847	
Absolute structure parameter	-0.02(5)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.160 and -0.130 e.Å ⁻³	

Table 8. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 2a. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
S(1)	4919(2)	4917(1)	5436(1)	62(1)
N(1)	4454(4)	3584(1)	6541(2)	38(1)
O(1)	6832(4)	2821(1)	6955(2)	59(1)
C(16)	5484(6)	3181(2)	7338(3)	41(1)
C(6)	2876(5)	4028(1)	6934(3)	36(1)
C(13)	294(5)	3083(2)	3547(3)	42(1)
C(10)	3916(5)	3754(2)	3054(3)	44(1)
C(9)	3595(5)	3553(1)	4262(3)	35(1)
C(12)	647(5)	3278(1)	2331(3)	40(1)
C(8)	5296(5)	3644(1)	5263(3)	40(1)
C(5)	1165(5)	3854(2)	7684(3)	45(1)
C(11)	2488(6)	3618(2)	2098(3)	47(1)
C(14)	1724(5)	3217(2)	4499(3)	41(1)
C(1)	3015(5)	4641(1)	6516(3)	43(1)
C(4)	-279(6)	4281(2)	8129(4)	58(1)
C(3)	-111(6)	4889(2)	7772(4)	61(1)
C(7)	6568(5)	4245(2)	5179(3)	53(1)
C(2)	1472(6)	5060(2)	6948(4)	58(1)
C(17)	4964(6)	3198(2)	8716(3)	54(1)
C(15)	-904(7)	3119(2)	1287(4)	59(1)

Table 9. Selected bond lengths [\AA] for 2a.

S1—C1	1.754 (4)	C8—H8	0.98
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S1—C7	1.797 (4)	C5—C4	1.371 (5)
N1—C16	1.375 (4)	C5—H5	0.93
N1—C6	1.434 (4)	C11—H11	0.93
N1—C8	1.465 (5)	C14—H14	0.93
O1—C16	1.214 (4)	C1—C2	1.396 (5)
C16—C17	1.504 (5)	C4—C3	1.370 (6)
C6—C5	1.382 (4)	C4—H4	0.93
C6—C1	1.398 (4)	C3—C2	1.370 (6)
C13—C14	1.379 (5)	C3—H3	0.93
C13—C12	1.380 (5)	C7—H7A	0.97
C13—H13	0.93	C7—H7B	0.97
C10—C9	1.375 (5)	C2—H2	0.93
C10—C11	1.383 (5)	C17—H17A	0.96
C10—H10	0.93	C17—H17B	0.96
C9—C14	1.393 (4)	C17—H17C	0.96
C9—C8	1.515 (4)	C15—H15A	0.96
C12—C11	1.382 (5)	C15—H15B	0.96
C12—C15	1.512 (5)	C15—H15C	0.96
C8—C7	1.522 (4)		

Table 10. Selected bond angles [°] for 2a.

C1—S1—C7	102.12 (15)	C13—C14—C9	120.8 (3)
C16—N1—C6	124.1 (3)	C13—C14—H14	119.6
C16—N1—C8	117.7 (3)	C9—C14—H14	119.6
C6—N1—C8	117.1 (2)	C2—C1—C6	117.8 (3)
O1—C16—N1	121.3 (3)	C2—C1—S1	117.3 (3)
O1—C16—C17	119.4 (3)	C6—C1—S1	124.9 (3)
N1—C16—C17	119.3 (3)	C3—C4—C5	119.9 (4)
C5—C6—C1	119.3 (3)	C3—C4—H4	120

C5—C6—N1	120.8 (3)	C5—C4—H4	120
C1—C6—N1	119.9 (3)	C2—C3—C4	119.4 (3)
C14—C13—C12	121.7 (3)	C2—C3—H3	120.3
C14—C13—H13	119.2	C4—C3—H3	120.3
C12—C13—H13	119.2	C8—C7—S1	112.5 (2)
C9—C10—C11	122.0 (3)	C8—C7—H7A	109.1
C9—C10—H10	119	S1—C7—H7A	109.1
C11—C10—H10	119	C8—C7—H7B	109.1
C10—C9—C14	117.2 (3)	S1—C7—H7B	109.1
C10—C9—C8	121.1 (3)	H7A—C7—H7B	107.8
C14—C9—C8	121.5 (3)	C3—C2—C1	121.9 (3)
C13—C12—C11	117.5 (3)	C3—C2—H2	119
C13—C12—C15	121.4 (3)	C1—C2—H2	119
C11—C12—C15	121.0 (3)	C16—C17—H17A	109.5
N1—C8—C9	113.2 (2)	C16—C17—H17B	109.5
N1—C8—C7	108.4 (3)	H17A—C17—H17B	109.5
C9—C8—C7	115.6 (3)	C16—C17—H17C	109.5
N1—C8—H8	106.3	H17A—C17—H17C	109.5
C9—C8—H8	106.3	H17B—C17—H17C	109.5
C7—C8—H8	106.3	C12—C15—H15A	109.5
C4—C5—C6	121.3 (3)	C12—C15—H15B	109.5
C4—C5—H5	119.4	H15A—C15—H15B	109.5
C6—C5—H5	119.4	C12—C15—H15C	109.5
C12—C11—C10	120.8 (3)	H15A—C15—H15C	109.5
C12—C11—H11	119.6	H15B—C15—H15C	109.5
C10—C11—H11	119.6		

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

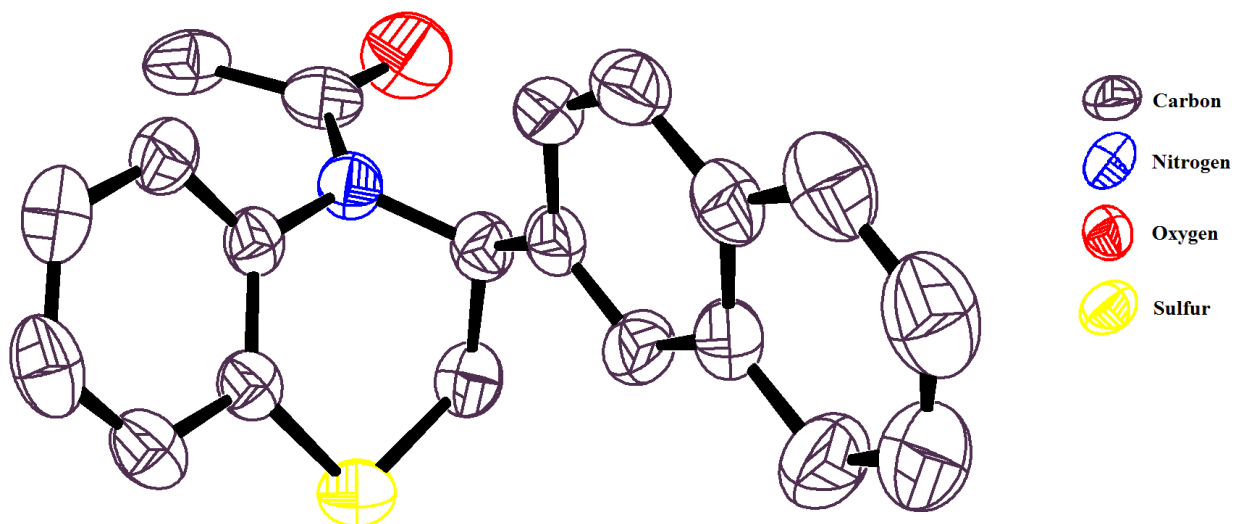
	U11	U22	U33	U23	U13	U12
S(1)	79(1)	45(1)	62(1)	7(1)	-4(1)	-19(1)
N(1)	42(1)	40(2)	32(1)	0(1)	-4(1)	7(1)
O(1)	59(1)	54(1)	64(2)	-1(1)	-14(1)	21(1)
C(16)	41(2)	36(2)	46(2)	2(2)	-14(2)	-1(2)
C(6)	39(2)	37(2)	33(2)	-3(1)	-8(2)	1(1)
C(13)	39(2)	40(2)	47(2)	-4(2)	0(2)	-7(1)
C(10)	47(2)	49(2)	36(2)	-1(2)	2(2)	-12(2)
C(9)	37(2)	33(2)	34(2)	-3(1)	-1(1)	5(1)
C(12)	48(2)	35(2)	37(2)	-7(2)	-5(2)	3(2)
C(8)	36(2)	48(2)	36(2)	1(2)	0(2)	4(1)
C(5)	43(2)	49(2)	42(2)	-5(2)	-2(2)	2(2)
C(11)	62(2)	51(2)	28(2)	0(2)	2(2)	-8(2)
C(14)	50(2)	40(2)	33(2)	5(1)	2(2)	-2(2)
C(1)	45(2)	37(2)	47(2)	-6(2)	-16(2)	-3(2)
C(4)	45(2)	75(3)	53(2)	-14(2)	1(2)	4(2)
C(3)	53(2)	60(3)	68(3)	-26(2)	-10(2)	16(2)
C(7)	45(2)	73(2)	42(2)	1(2)	-6(2)	-18(2)
C(2)	66(2)	37(2)	70(3)	-7(2)	-25(2)	6(2)
C(17)	56(2)	64(2)	42(2)	13(2)	-15(2)	1(2)
C(15)	67(2)	57(2)	51(2)	-9(2)	-20(2)	0(2)

Table 12. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for XXXX

	x	y	z	U(eq)
H(13)	-940	2856	3729	50

H(10)	5132	3990	2873	53
H(8)	6340	3308	5154	48
H(5)	989	3439	7891	54
H(11)	2769	3757	1288	57
H(14)	1436	3080	5310	49
H(4)	-1370	4159	8673	69
H(3)	-1065	5182	8087	73
H(7A)	7228	4274	4356	64
H(7B)	7713	4238	5798	64
H(2)	1521	5467	6667	69
H(17A)	3740	2939	8879	81
H(17B)	4643	3615	8961	81
H(17C)	6178	3050	9186	81
H(15A)	-2334	3239	1525	88
H(15B)	-869	2681	1135	88
H(15C)	-493	3335	537	88

Crystal Structure of 2g with 50% ellipsoidal probability



Packing diagram of 2g

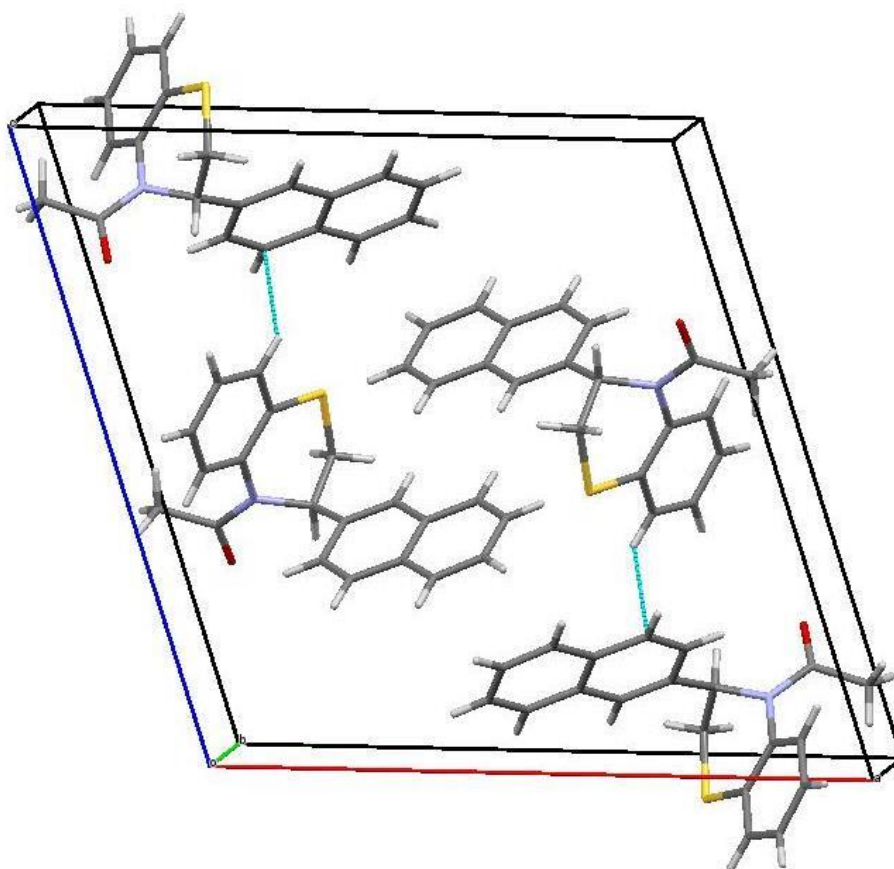


Table 13. Crystal data and structure refinement for 2g

Identification code	DP_547 (CCDC No. 1409828)
Empirical formula	C ₂₀ H ₁₇ N O S

Formula weight	319.40	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 2 ₁ /c	
Unit cell dimensions	a = 16.3601(8) Å	α = 90°.
	b = 6.1956(3) Å	β = 108.141(2)°.
	c = 16.4686(8) Å	γ = 90°.
Volume	1586.29(13) Å ³	
Z	4	
Density (calculated)	1.337 Mg/m ³	
Absorption coefficient	0.208 mm ⁻¹	
F(000)	672	
Theta range for data collection	1.310 to 25.717°.	
Index ranges	-19 ≤ h ≤ 19, -7 ≤ k ≤ 7, -19 ≤ l ≤ 20	
Reflections collected	14231	
Independent reflections	2997 [R(int) = 0.0178]	
Completeness to theta = 25.242°	99.1 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	2997 / 0 / 209	
Goodness-of-fit on F ²	1.038	
Final R indices [I > 2σ(I)]	R1 = 0.0352, wR2 = 0.0921	
R indices (all data)	R1 = 0.0419, wR2 = 0.0972	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.159 and -0.190 e.Å ⁻³	

Table 14. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 2g. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	7081(1)	-337(1)	4458(1)	55(1)
N(1)	8571(1)	541(2)	6119(1)	45(1)
O(1)	9328(1)	-1585(3)	7189(1)	89(1)
C(1)	8494(1)	2038(2)	5447(1)	40(1)
C(6)	7834(1)	1776(2)	4674(1)	42(1)
C(9)	7246(1)	1736(2)	6399(1)	44(1)
C(2)	9025(1)	3838(3)	5565(1)	49(1)
C(5)	7755(1)	3276(3)	4023(1)	52(1)
C(18)	7688(1)	3369(3)	6964(1)	50(1)
C(8)	7754(1)	-164(2)	6231(1)	47(1)
C(10)	6373(1)	1875(3)	6074(1)	53(1)
C(17)	7259(1)	5016(3)	7192(1)	54(1)
C(19)	9318(1)	-443(3)	6586(1)	54(1)
C(11)	5907(1)	3580(3)	6294(1)	52(1)
C(7)	7298(1)	-1634(3)	5488(1)	53(1)
C(16)	6357(1)	5191(3)	6864(1)	50(1)
C(20)	10116(1)	-149(3)	6330(1)	62(1)
C(3)	8946(1)	5276(3)	4906(1)	57(1)
C(4)	8317(1)	4975(3)	4135(1)	61(1)
C(15)	5884(1)	6892(3)	7076(1)	67(1)
C(12)	5005(1)	3748(4)	5951(1)	77(1)
C(14)	5016(2)	6997(4)	6726(1)	79(1)
C(13)	4575(1)	5414(4)	6161(2)	84(1)

Table 15. Selected bond lengths [\AA] for 2g.

S1—C6	1.7555 (15)	C17—C16	1.409 (2)
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S1—C7	1.8098 (17)	C17—H17	0.93
N1—C19	1.368 (2)	C19—C20	1.502 (2)
N1—C1	1.4185 (18)	C11—C12	1.409 (2)
N1—C8	1.4722 (18)	C11—C16	1.411 (2)
O1—C19	1.215 (2)	C7—H7A	0.97
C1—C2	1.390 (2)	C7—H7B	0.97
C1—C6	1.400 (2)	C16—C15	1.413 (2)
C6—C5	1.393 (2)	C20—H20A	0.96
C9—C10	1.363 (2)	C20—H20B	0.96
C9—C18	1.412 (2)	C20—H20C	0.96
C9—C8	1.515 (2)	C3—C4	1.377 (3)
C2—C3	1.377 (2)	C3—H3	0.93
C2—H2	0.93	C4—H4	0.93
C5—C4	1.372 (2)	C15—C14	1.358 (3)
C5—H5	0.93	C15—H15	0.93
C18—C17	1.356 (2)	C12—C13	1.354 (3)
C18—H18	0.93	C12—H12	0.93
C8—C7	1.521 (2)	C14—C13	1.389 (3)
C8—H8	0.98	C14—H14	0.93
C10—C11	1.414 (2)	C13—H13	0.93
C10—H10	0.93		

Table 16. Selected bond angles [°] for 2g.

C6—S1—C7	102.78 (7)	C12—C11—C16	118.58 (16)
C19—N1—C1	125.21 (13)	C12—C11—C10	122.22 (16)
C19—N1—C8	118.97 (13)	C16—C11—C10	119.19 (15)
C1—N1—C8	115.38 (11)	C8—C7—S1	113.06 (11)
C2—C1—C6	119.46 (13)	C8—C7—H7A	109

C2—C1—N1	121.07 (13)	S1—C7—H7A	109
C6—C1—N1	119.31 (13)	C8—C7—H7B	109
C5—C6—C1	118.85 (14)	S1—C7—H7B	109
C5—C6—S1	116.78 (12)	H7A—C7—H7B	107.8
C1—C6—S1	124.37 (11)	C17—C16—C11	117.87 (15)
C10—C9—C18	118.24 (14)	C17—C16—C15	123.44 (16)
C10—C9—C8	123.02 (14)	C11—C16—C15	118.69 (16)
C18—C9—C8	118.60 (13)	C19—C20—H20A	109.5
C3—C2—C1	120.51 (15)	C19—C20—H20B	109.5
C3—C2—H2	119.7	H20A—C20—H20B	109.5
C1—C2—H2	119.7	C19—C20—H20C	109.5
C4—C5—C6	120.75 (16)	H20A—C20—H20C	109.5
C4—C5—H5	119.6	H20B—C20—H20C	109.5
C6—C5—H5	119.6	C4—C3—C2	119.95 (16)
C17—C18—C9	121.18 (15)	C4—C3—H3	120
C17—C18—H18	119.4	C2—C3—H3	120
C9—C18—H18	119.4	C5—C4—C3	120.30 (15)
N1—C8—C9	111.35 (12)	C5—C4—H4	119.9
N1—C8—C7	108.25 (12)	C3—C4—H4	119.9
C9—C8—C7	116.93 (13)	C14—C15—C16	120.74 (19)
N1—C8—H8	106.6	C14—C15—H15	119.6

C9—C8—H8	106.6	C16—C15—H15	119.6
C7—C8—H8	106.6	C13—C12—C11	121.1 (2)
C9—C10—C11	121.97 (15)	C13—C12—H12	119.5
C9—C10—H10	119	C11—C12—H12	119.5
C11—C10—H10	119	C15—C14—C13	120.42 (19)
C18—C17—C16	121.53 (15)	C15—C14—H14	119.8
C18—C17—H17	119.2	C13—C14—H14	119.8
C16—C17—H17	119.2	C12—C13—C14	120.5 (2)
O1—C19—N1	120.10 (16)	C12—C13—H13	119.8
O1—C19—C20	120.55 (15)	C14—C13—H13	119.8
N1—C19—C20	119.33 (15)		

Table 17. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **2g**. The anisotropic displacement factor exponent takes the form: $-2p^2[h^2 a^* 2U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
S(1)	55(1)	55(1)	51(1)	-6(1)	10(1)	-3(1)
N(1)	42(1)	49(1)	44(1)	9(1)	12(1)	5(1)
O(1)	82(1)	95(1)	75(1)	42(1)	5(1)	14(1)
C(1)	38(1)	43(1)	42(1)	4(1)	17(1)	8(1)
C(6)	42(1)	43(1)	43(1)	0(1)	17(1)	10(1)
C(9)	49(1)	47(1)	40(1)	4(1)	20(1)	-5(1)
C(2)	42(1)	51(1)	56(1)	-1(1)	20(1)	2(1)
C(5)	59(1)	57(1)	43(1)	6(1)	20(1)	18(1)
C(18)	48(1)	61(1)	39(1)	1(1)	13(1)	-8(1)
C(8)	50(1)	47(1)	46(1)	9(1)	17(1)	-1(1)

C(10)	48(1)	56(1)	58(1)	-12(1)	20(1)	-10(1)
C(17)	66(1)	53(1)	43(1)	-6(1)	18(1)	-13(1)
C(19)	55(1)	50(1)	46(1)	3(1)	1(1)	9(1)
C(11)	51(1)	59(1)	51(1)	-2(1)	24(1)	-4(1)
C(7)	57(1)	43(1)	64(1)	0(1)	24(1)	-2(1)
C(16)	65(1)	49(1)	44(1)	6(1)	26(1)	-2(1)
C(20)	43(1)	65(1)	67(1)	-13(1)	2(1)	15(1)
C(3)	60(1)	48(1)	76(1)	5(1)	39(1)	1(1)
C(4)	77(1)	53(1)	64(1)	19(1)	40(1)	17(1)
C(15)	90(1)	55(1)	65(1)	-1(1)	38(1)	4(1)
C(12)	52(1)	94(2)	88(1)	-21(1)	25(1)	1(1)
C(14)	92(2)	76(1)	83(1)	6(1)	45(1)	26(1)
C(13)	64(1)	108(2)	87(2)	-5(1)	32(1)	19(1)

Table 18. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 2g

	x	y	z	U(eq)
H(2)	9437	4075	6092	58
H(5)	7316	3124	3508	63
H(18)	8285	3315	7183	60
H(8)	7904	-1060	6747	57
H(10)	6075	816	5697	64
H(17)	7567	6054	7573	65
H(7A)	6759	-2114	5557	64
H(7B)	7652	-2901	5503	64
H(20A)	10463	974	6669	93
H(20B)	9957	241	5737	93
H(20C)	10436	-1473	6420	93
H(3)	9317	6448	4983	69

H(4)	8273	5927	3686	73
H(15)	6171	7951	7459	80
H(12)	4701	2696	5574	93
H(14)	4714	8137	6865	95
H(13)	3981	5500	5926	101
