Supplementary Material

Oxidative arylamination of 1,3-dinitrobenzene and 3-nitropyridine under anaerobic conditions: the dual role of the nitroarenes

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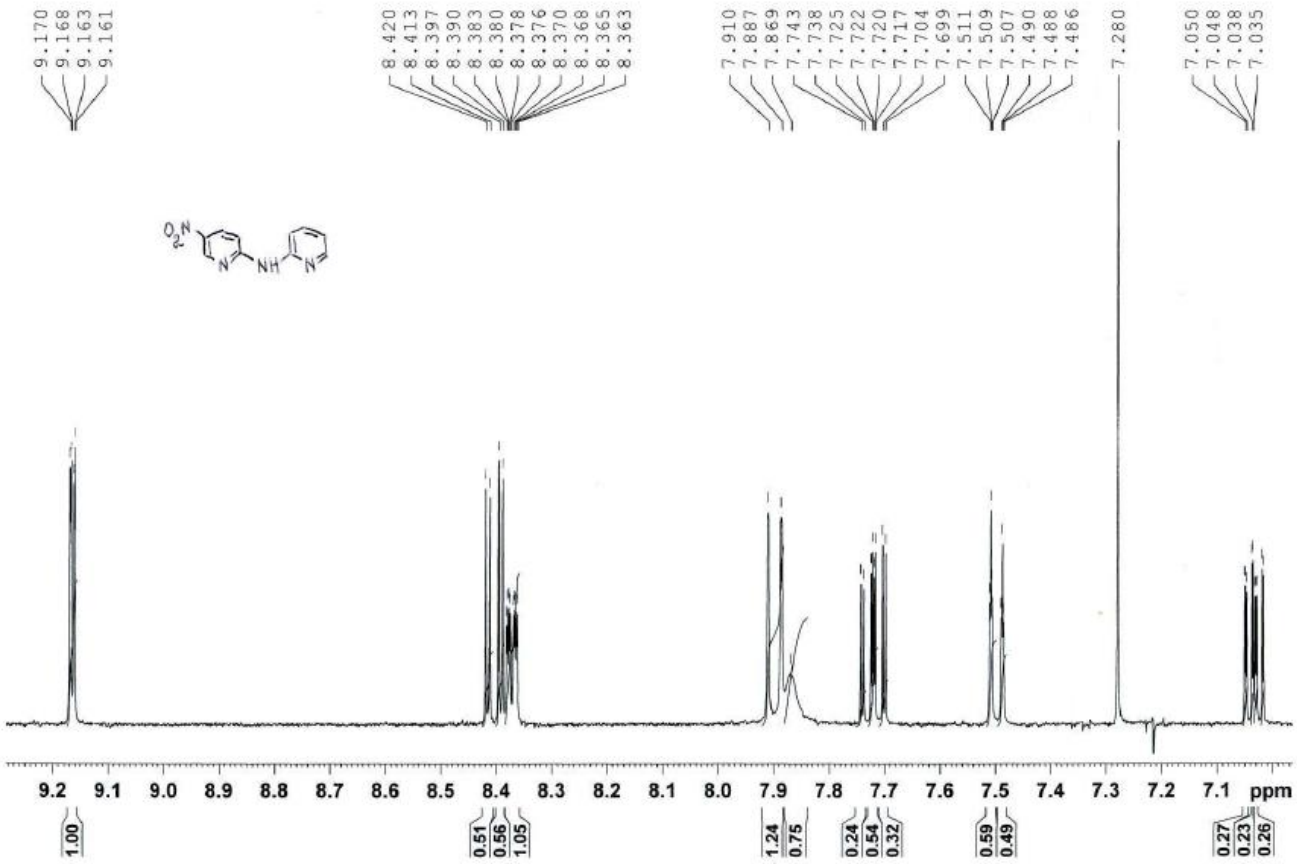

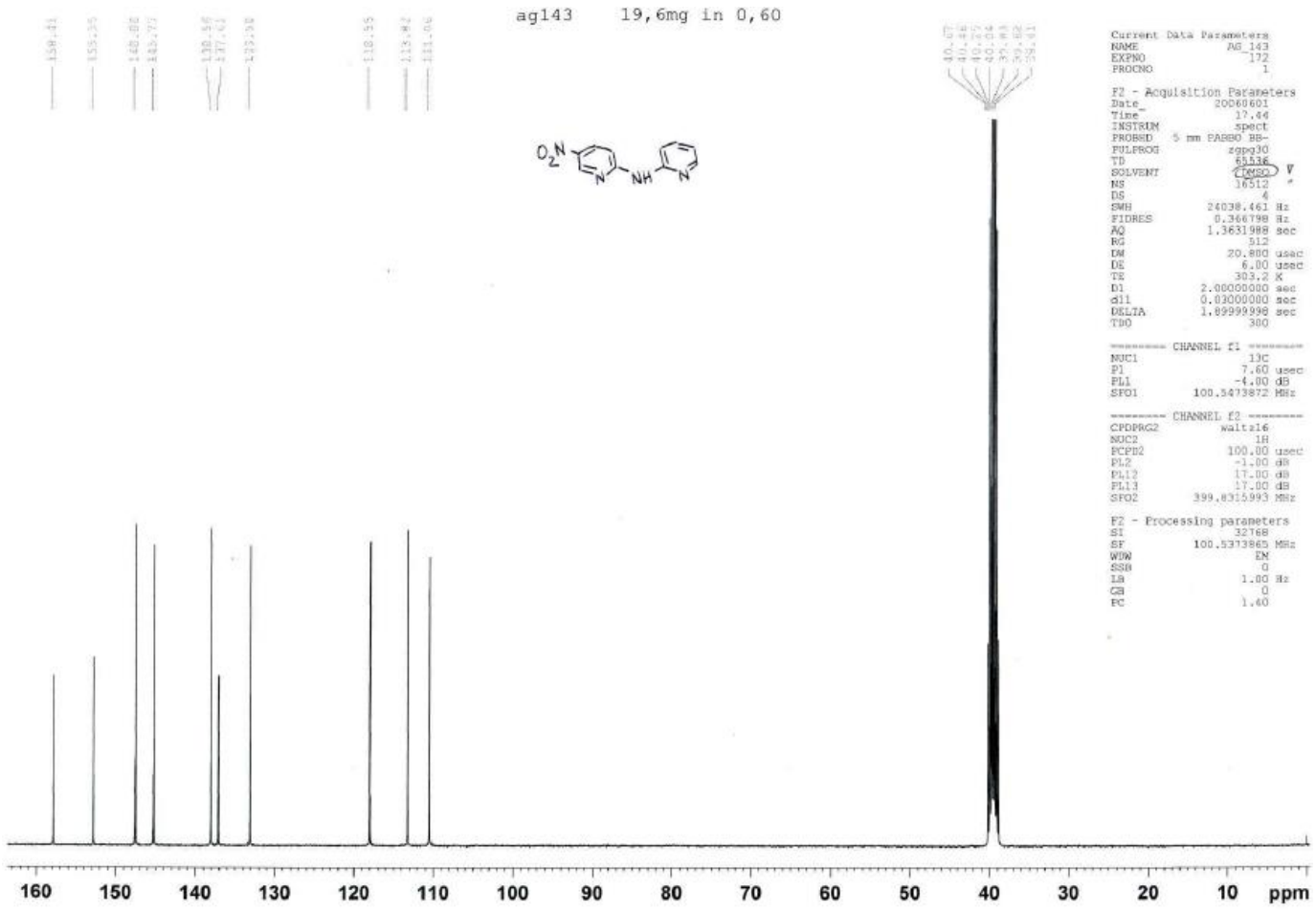

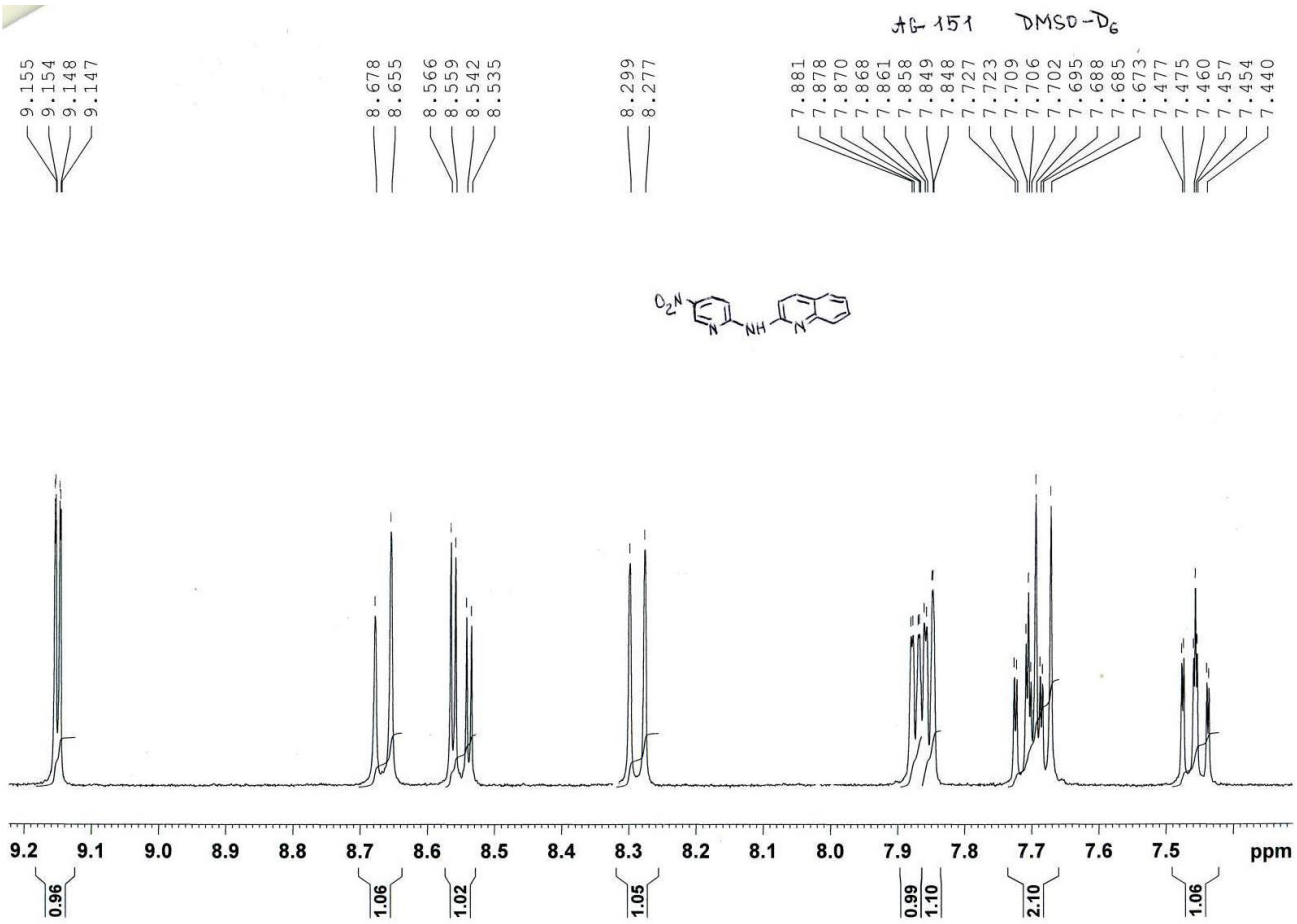

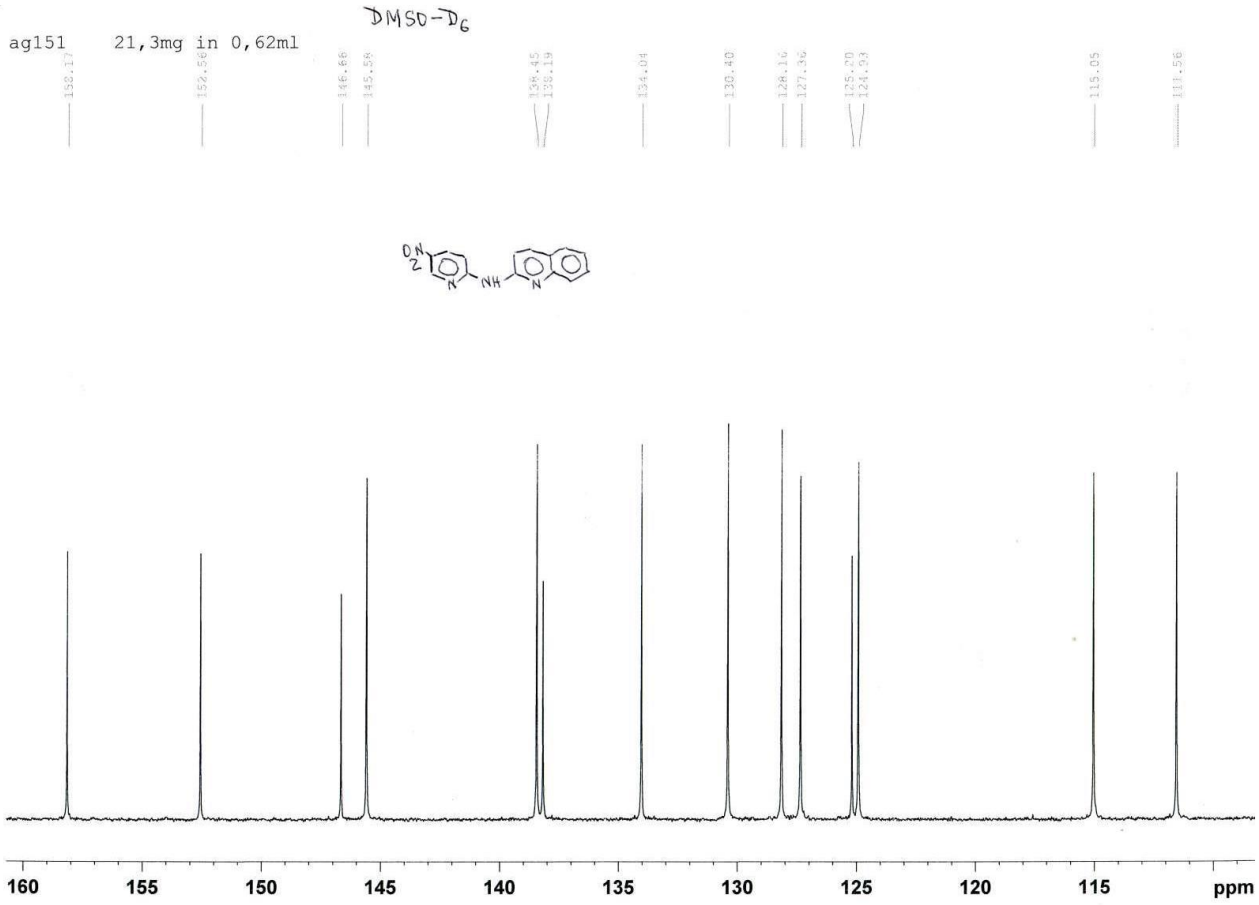

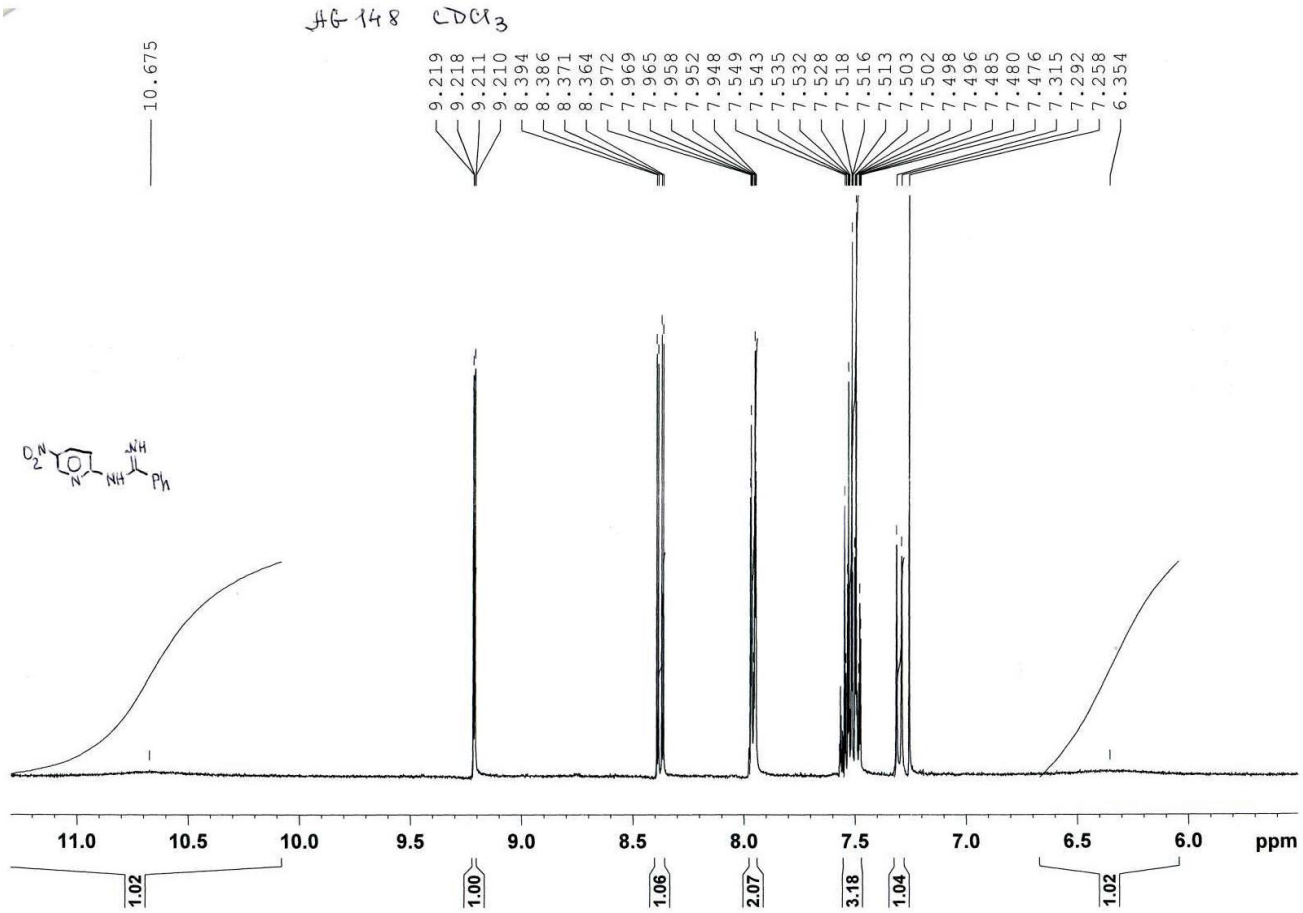

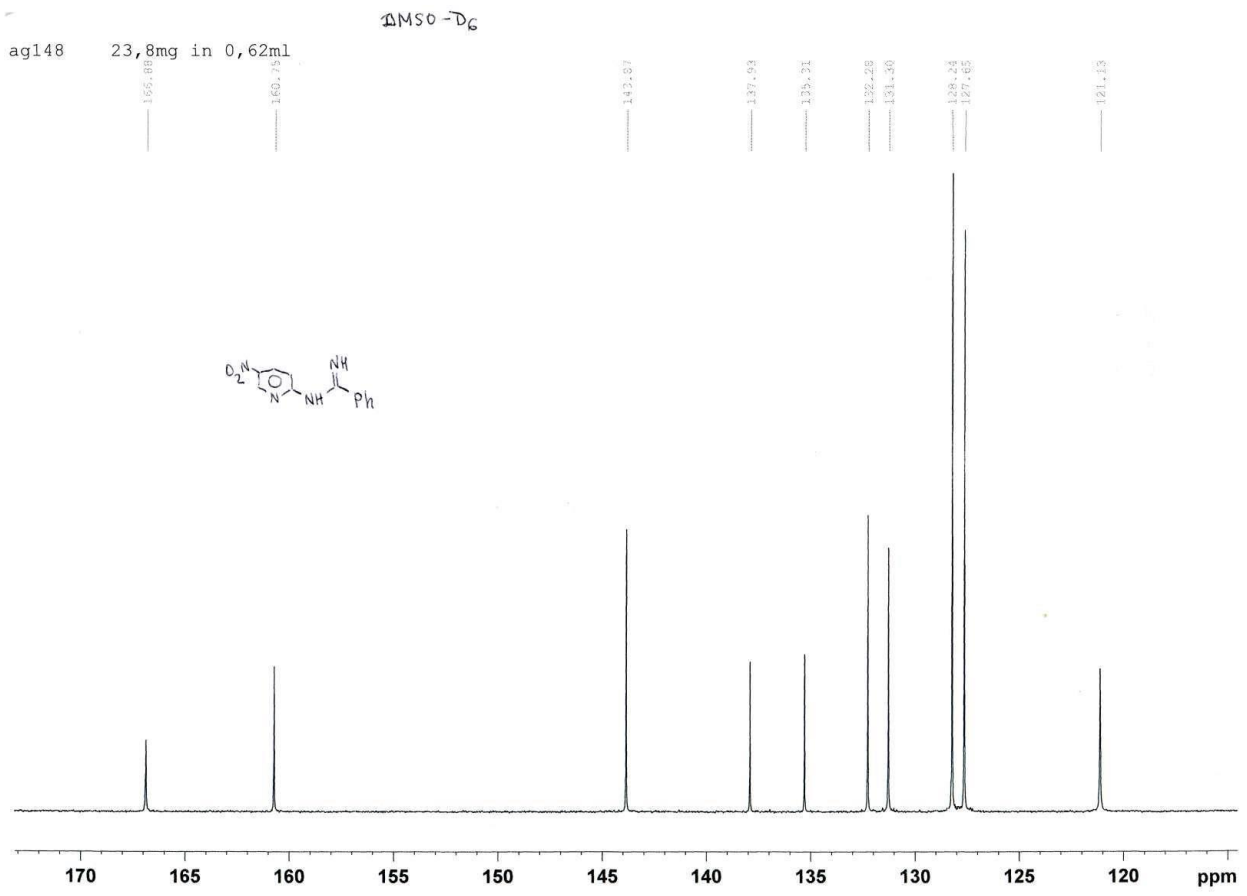

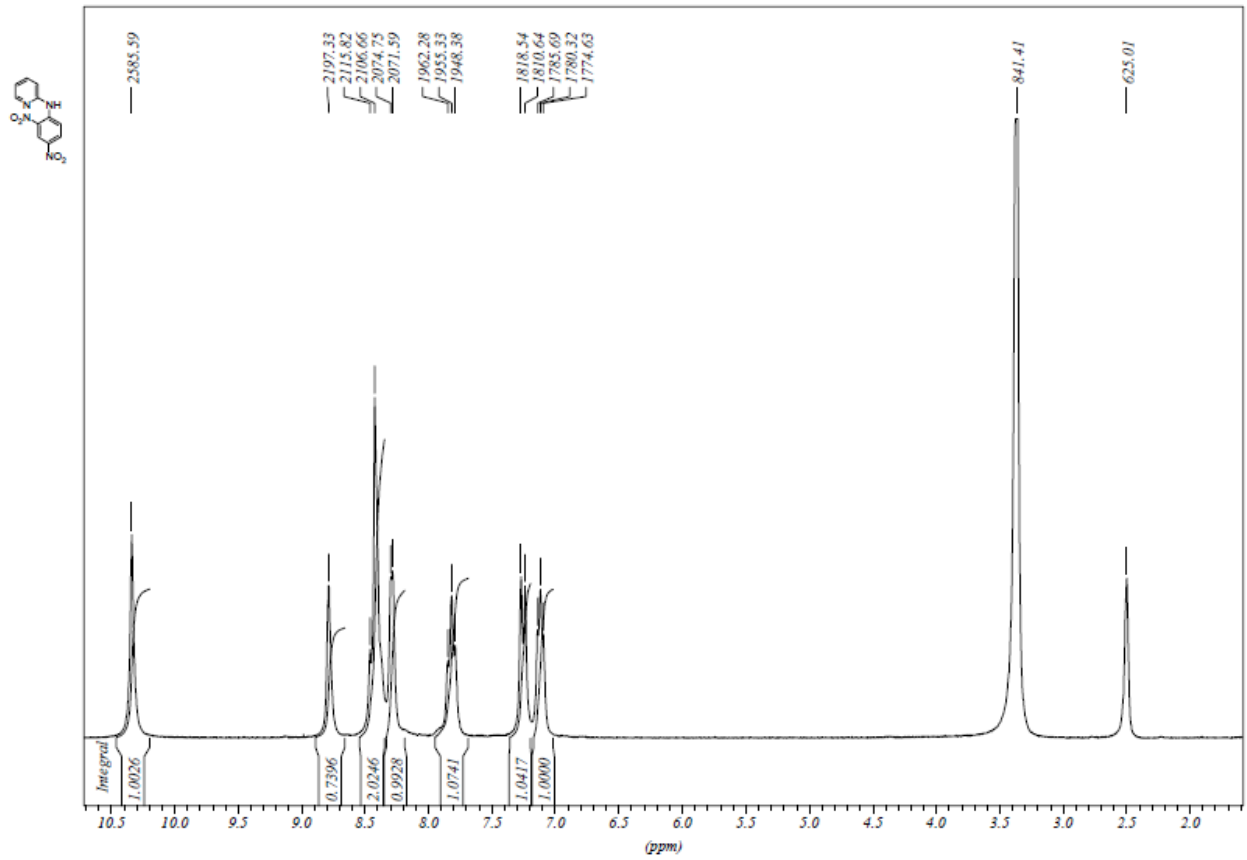

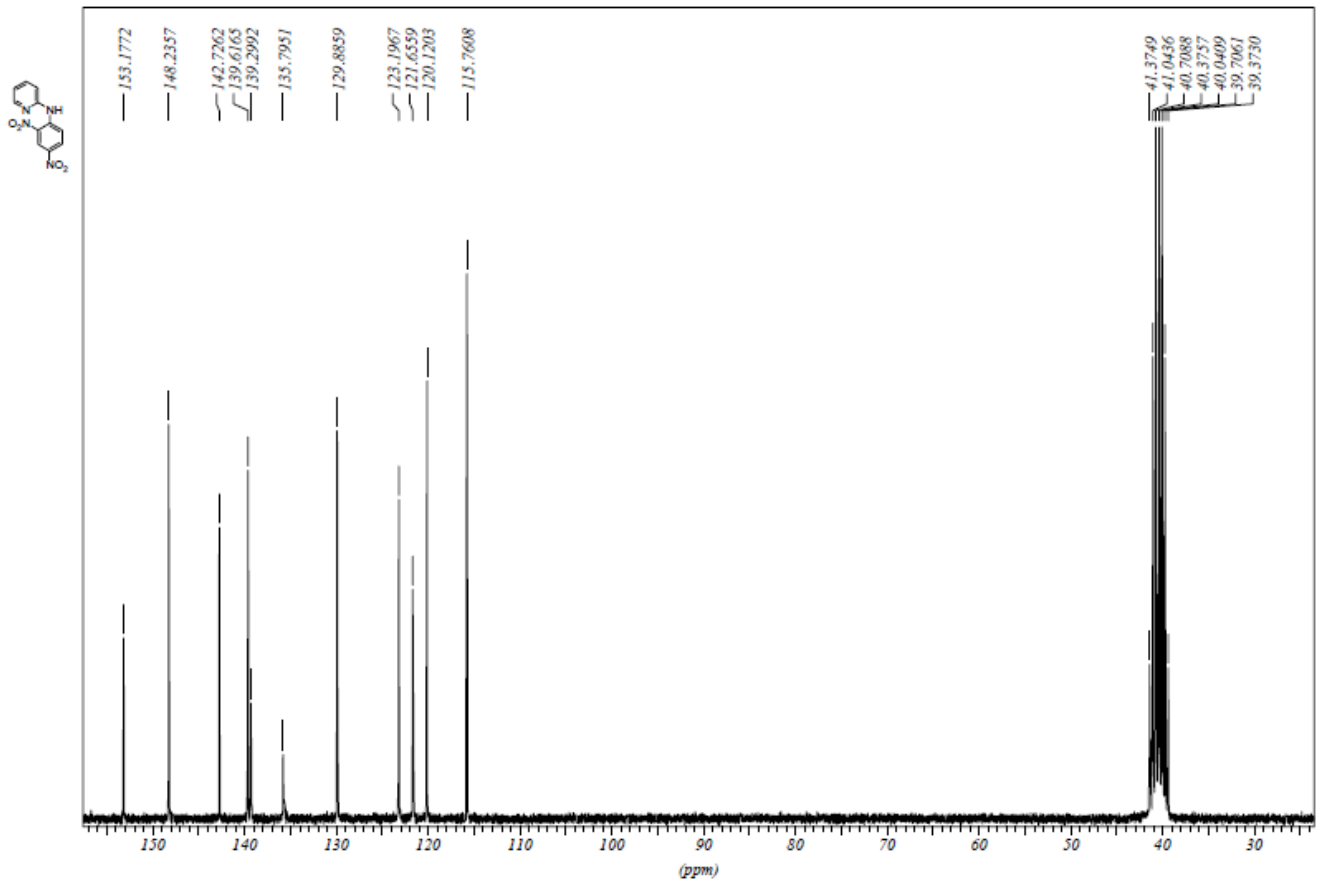

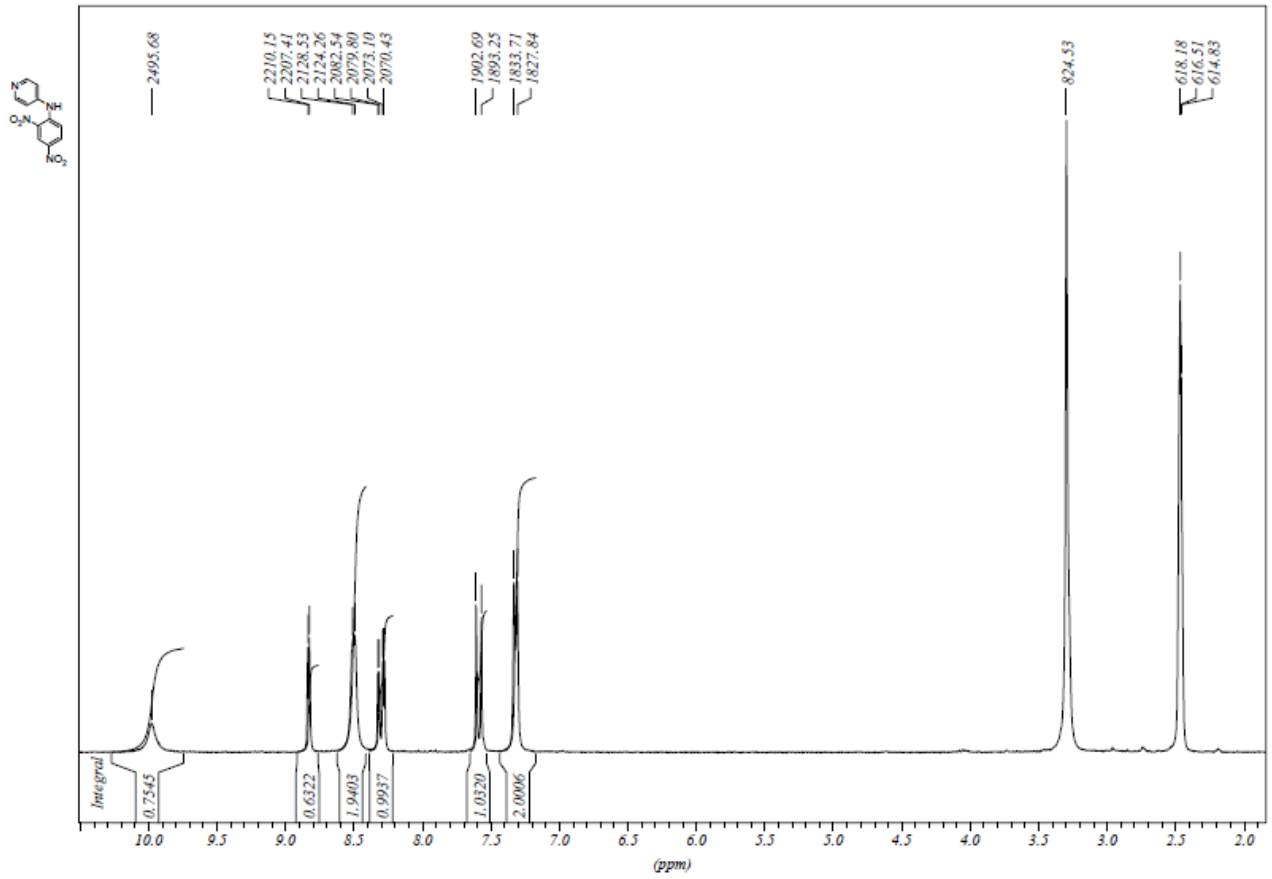

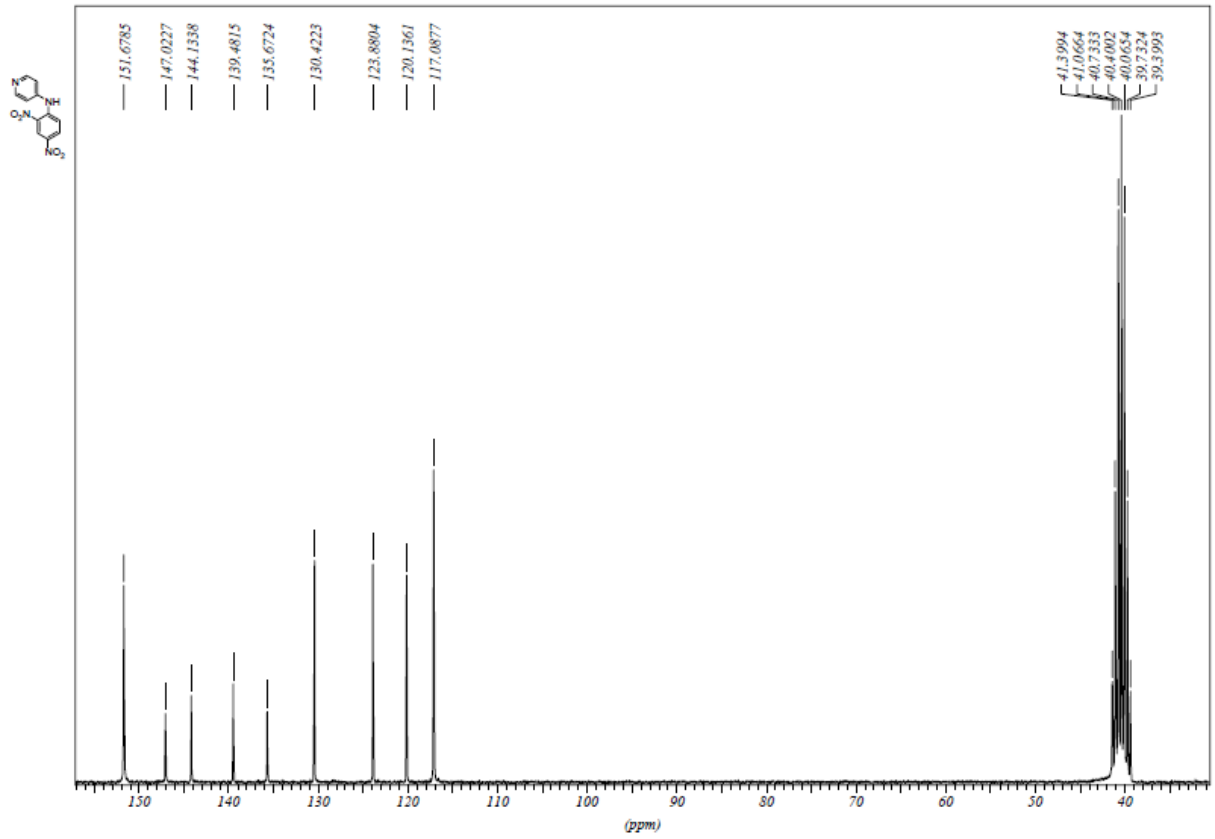

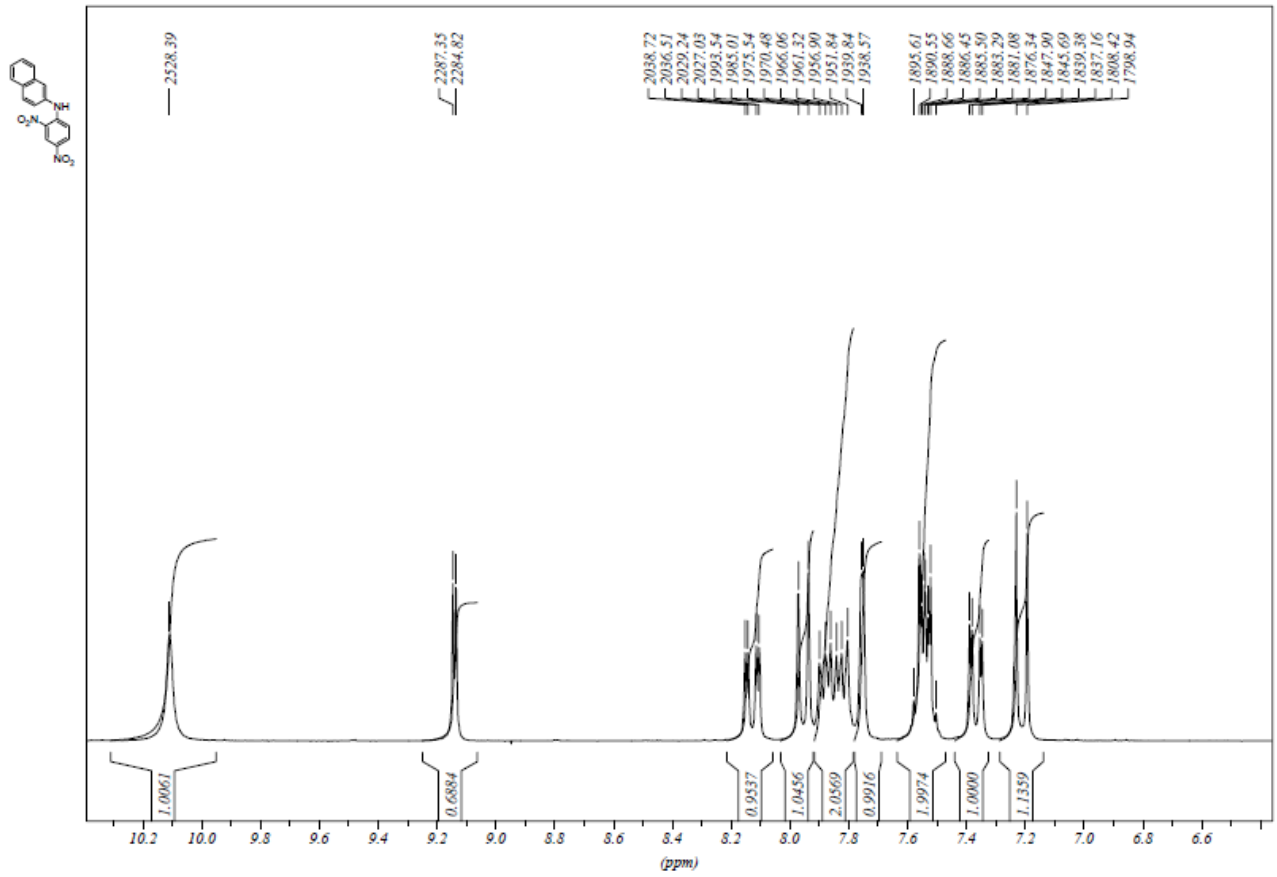

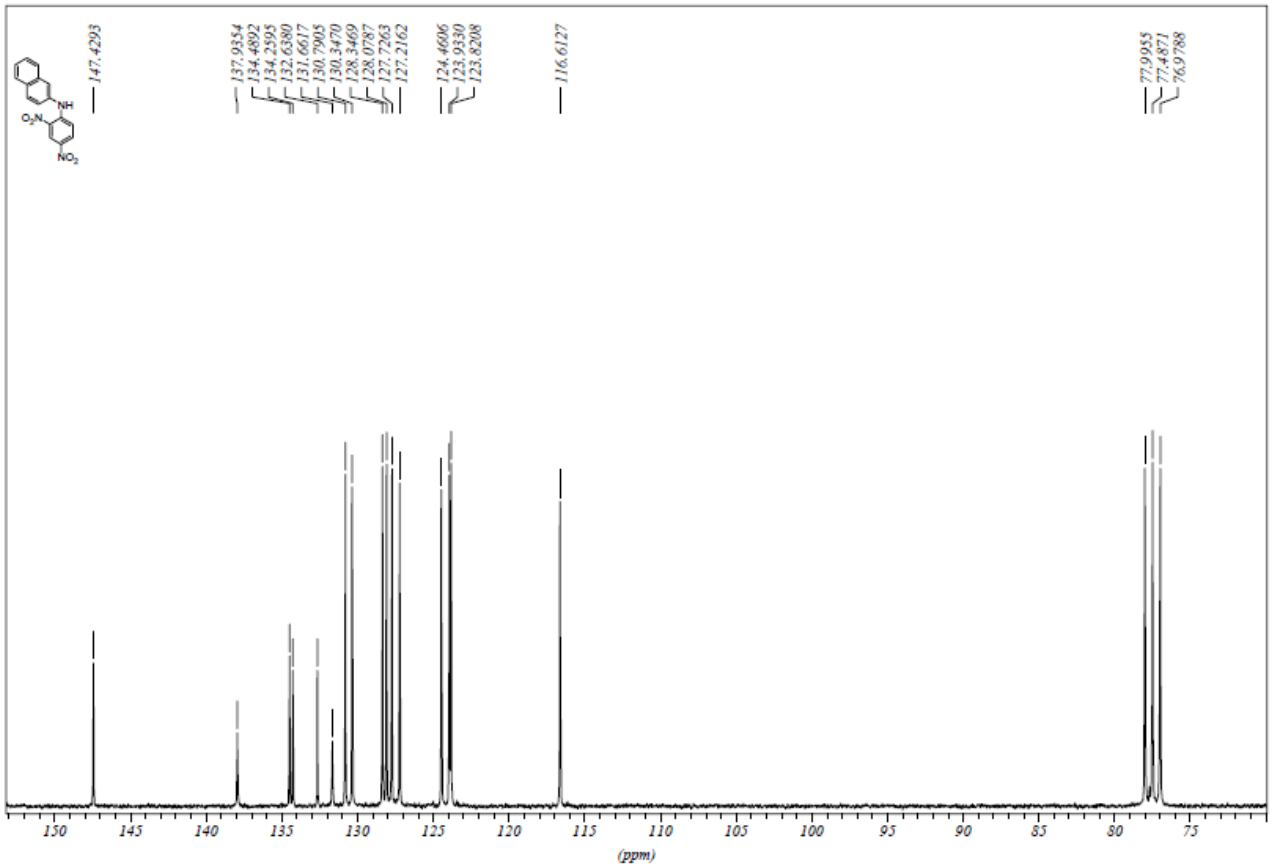

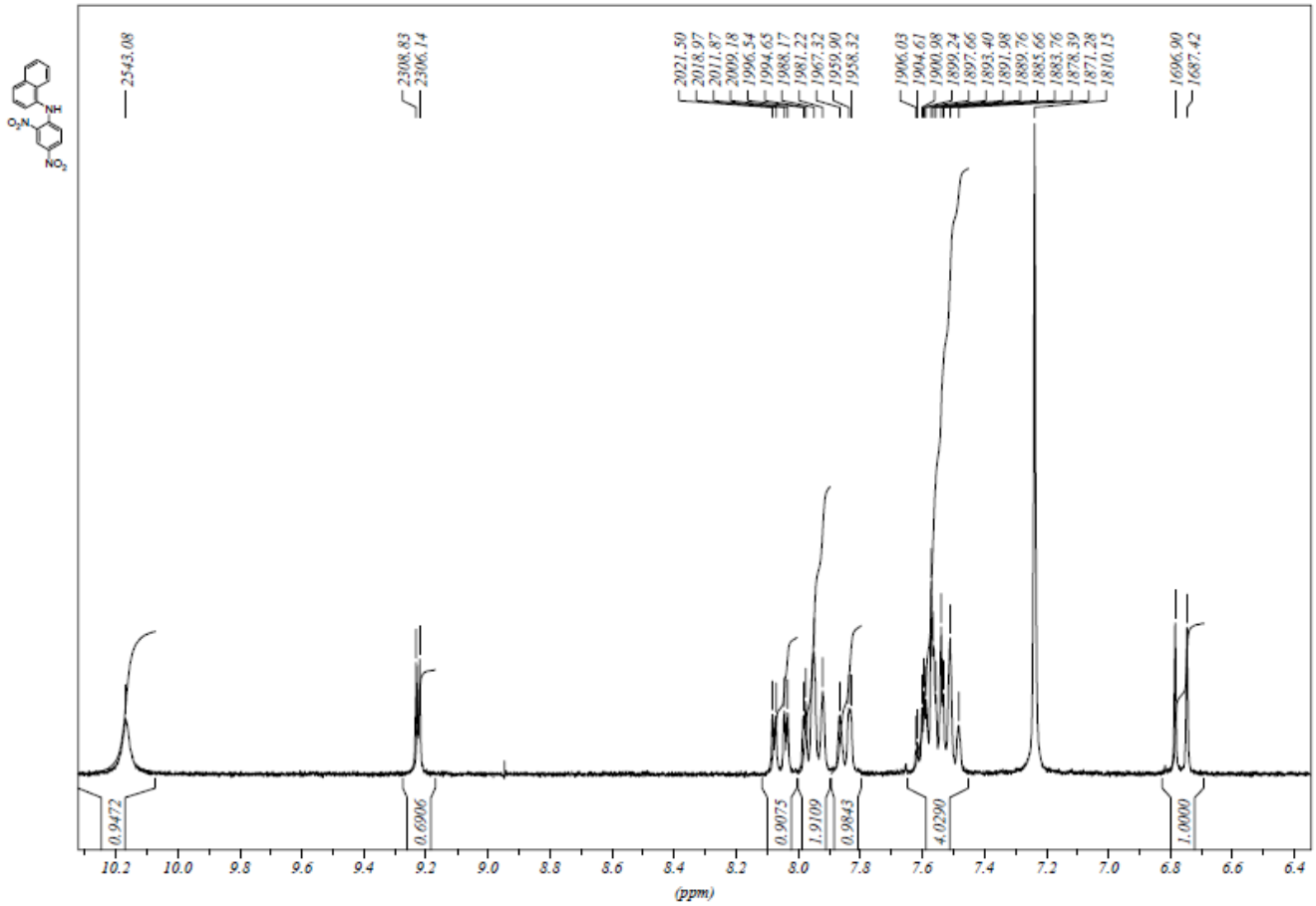

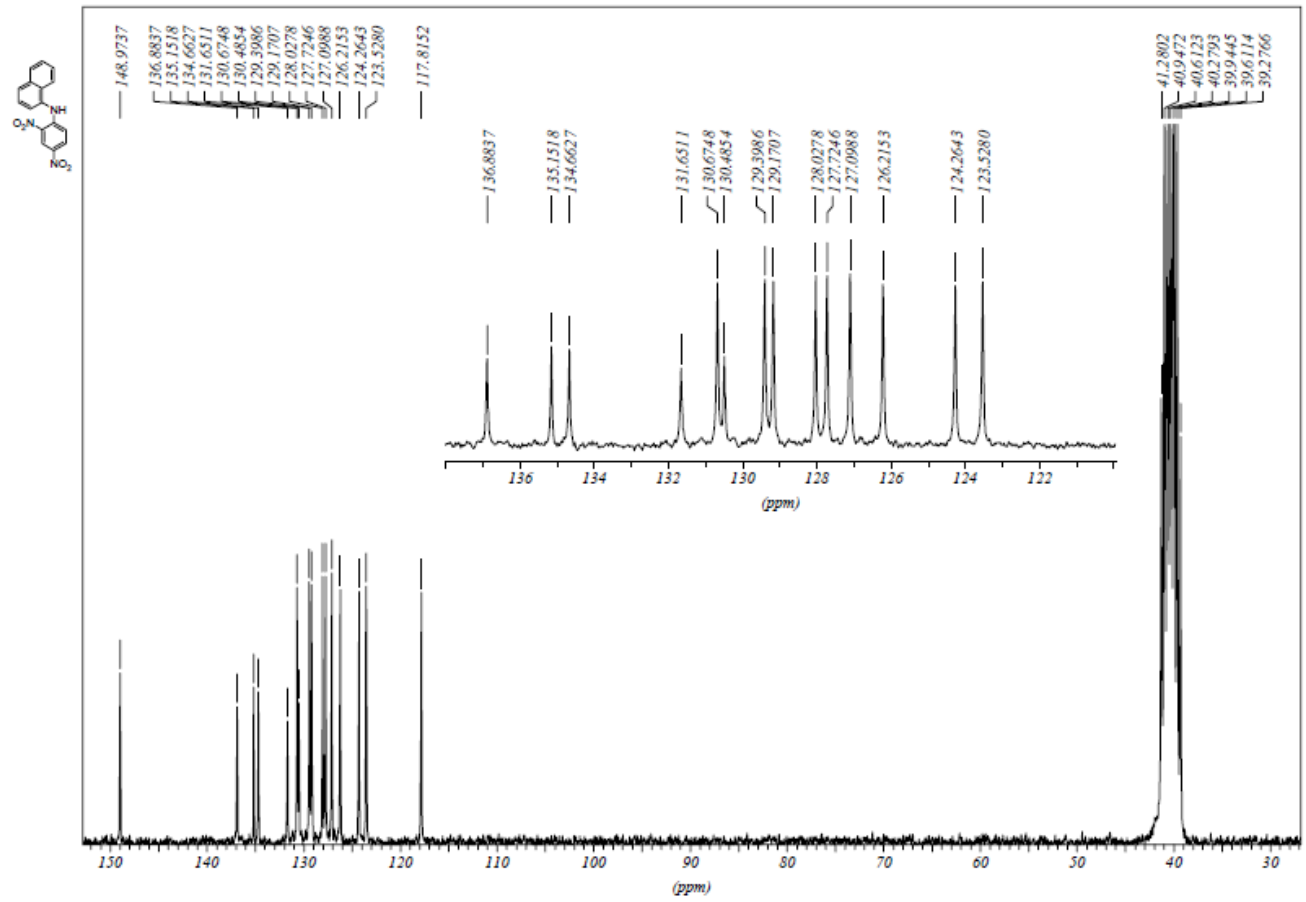

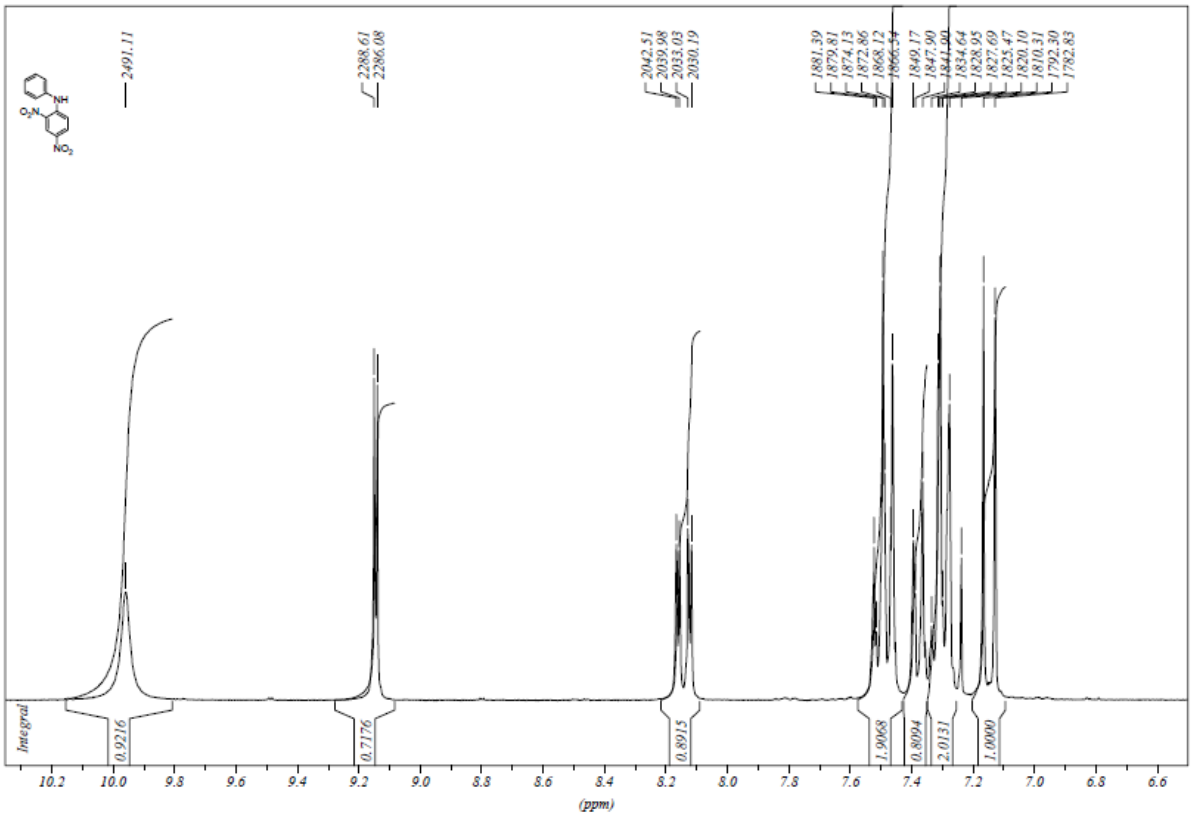

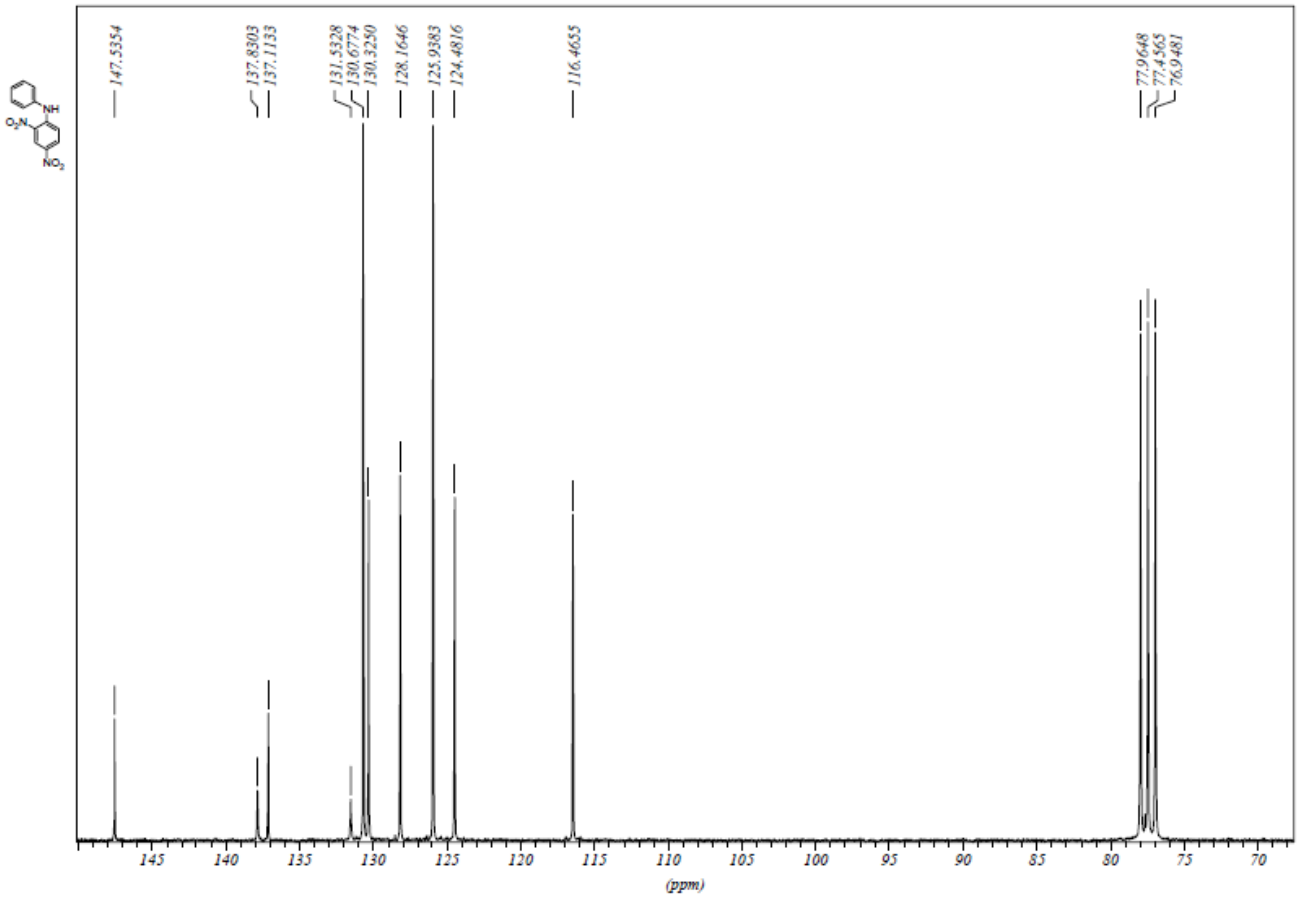

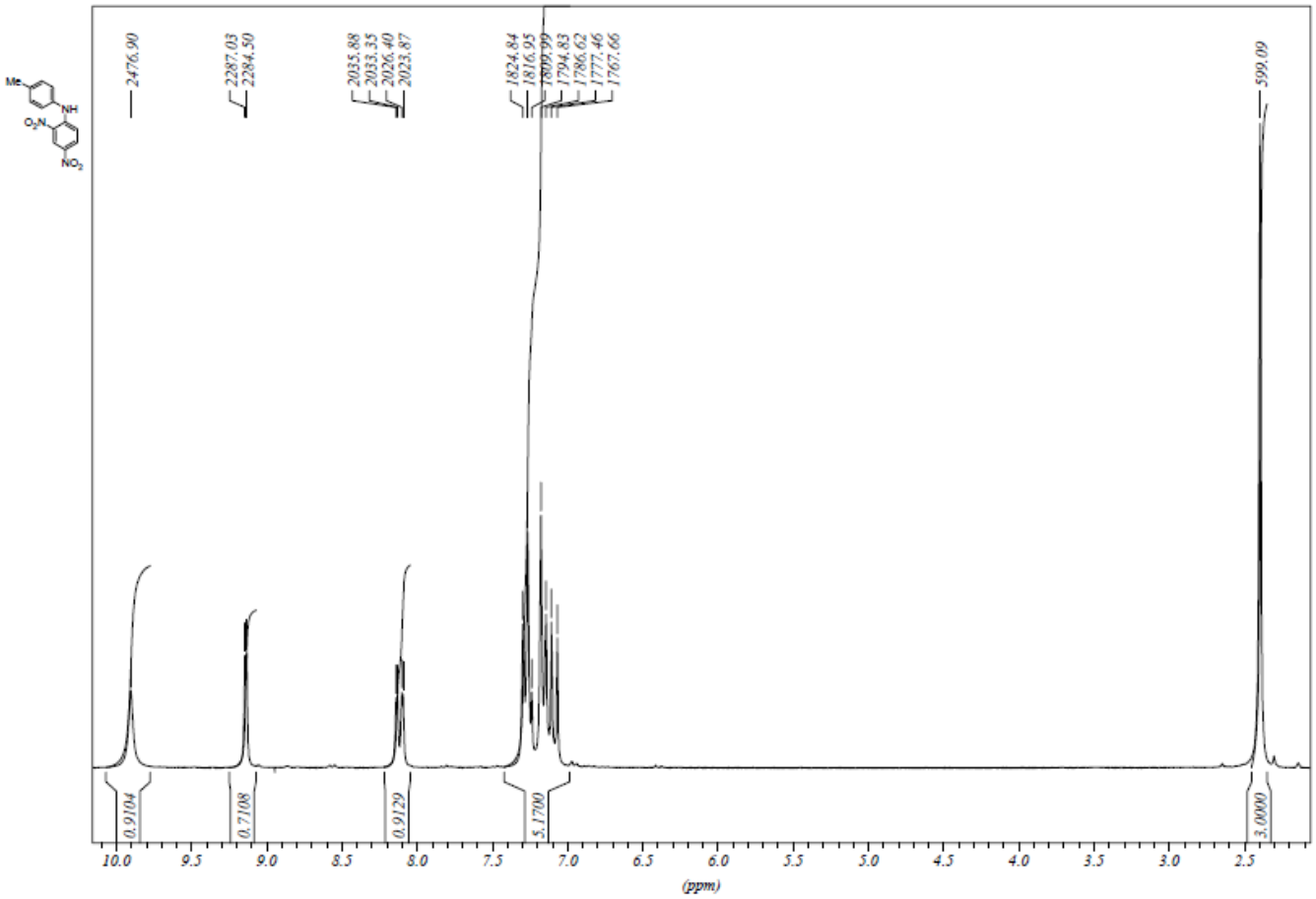

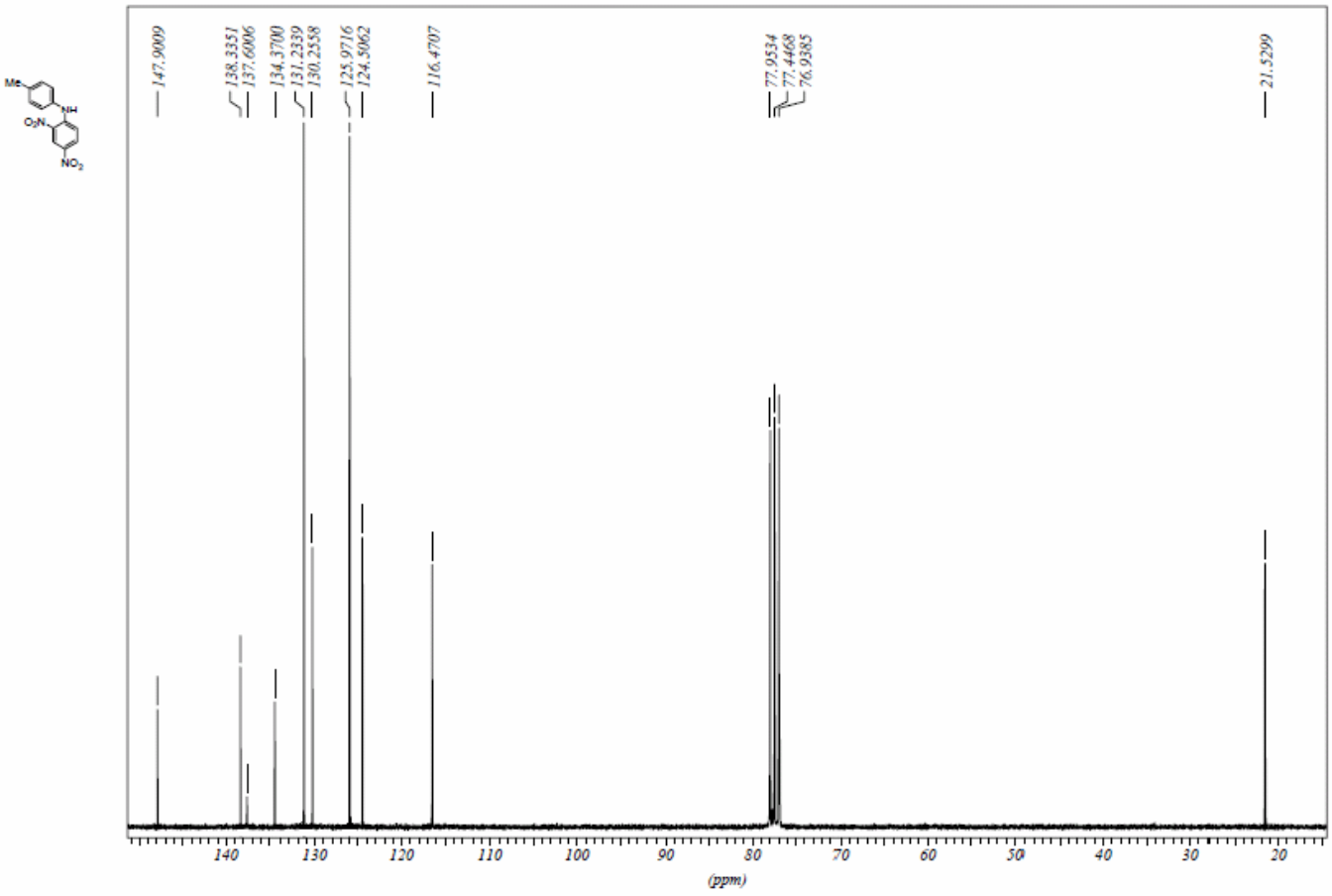

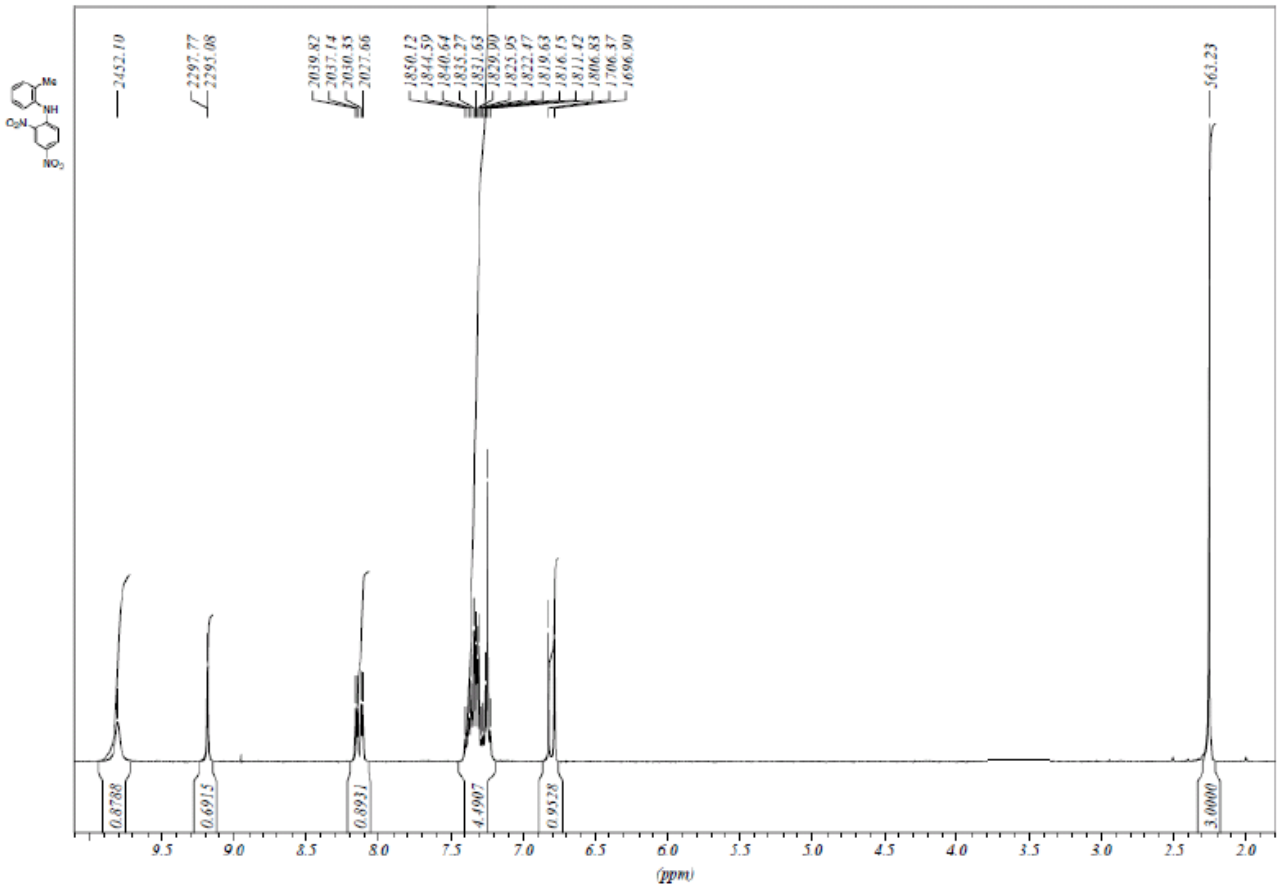

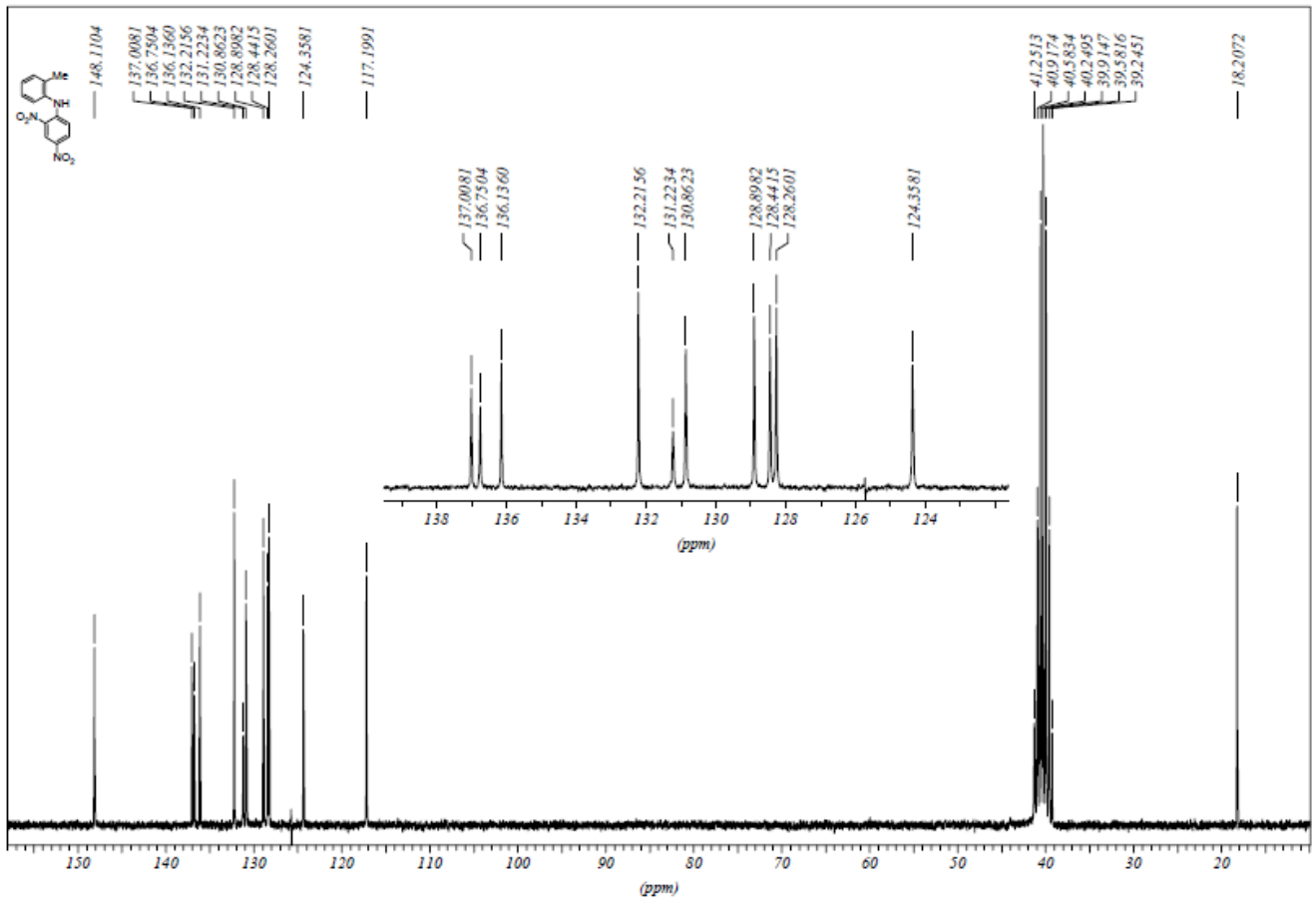

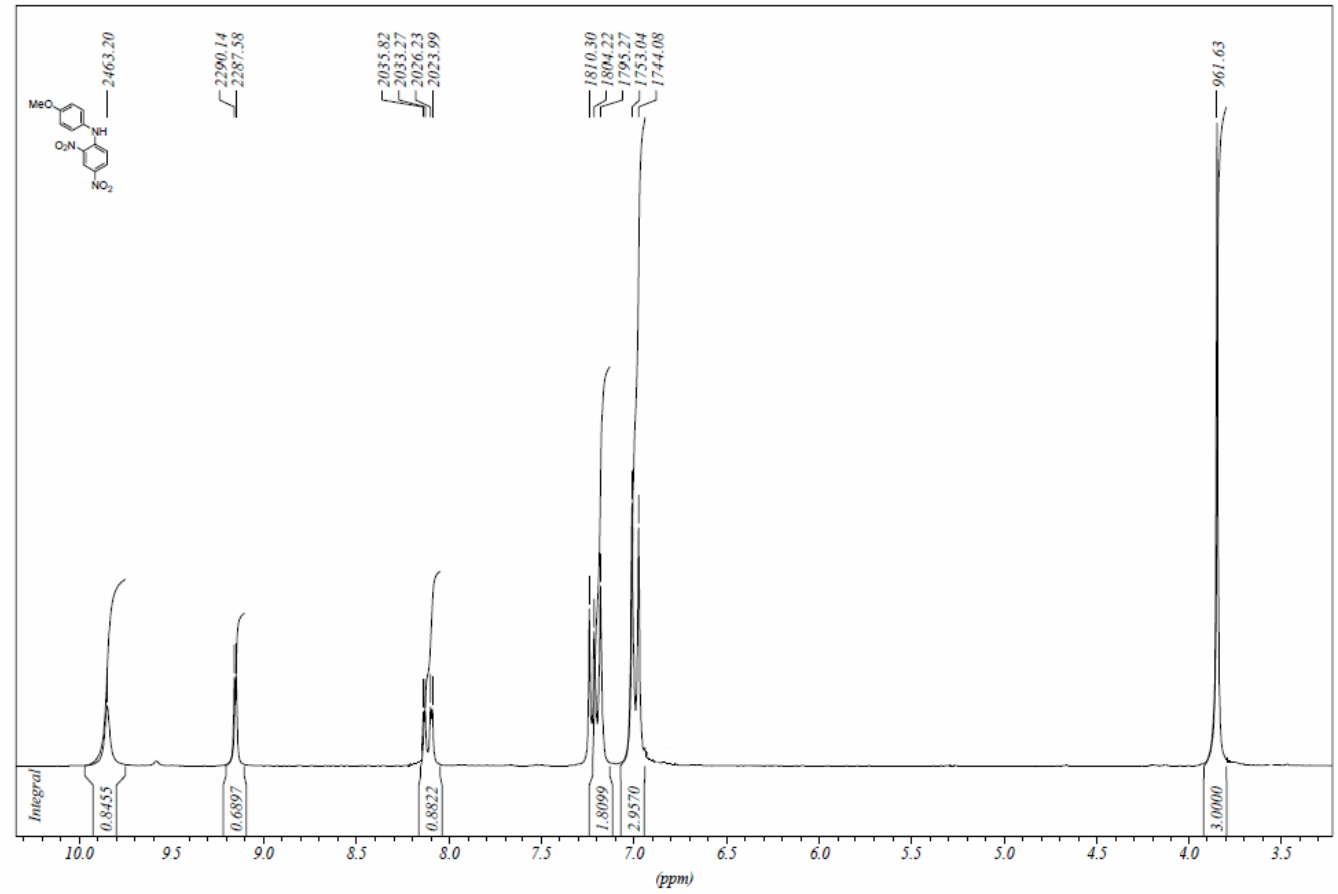

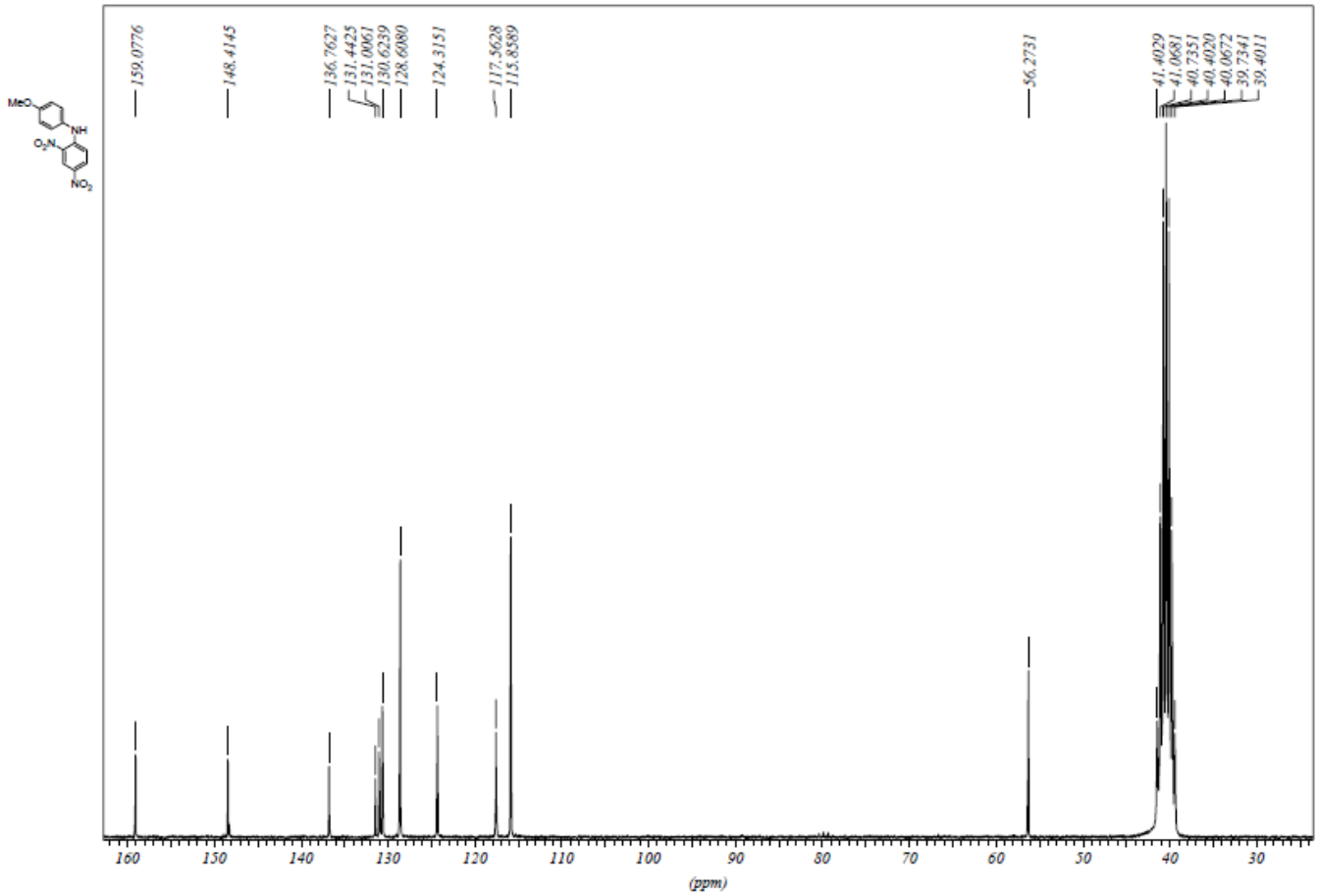

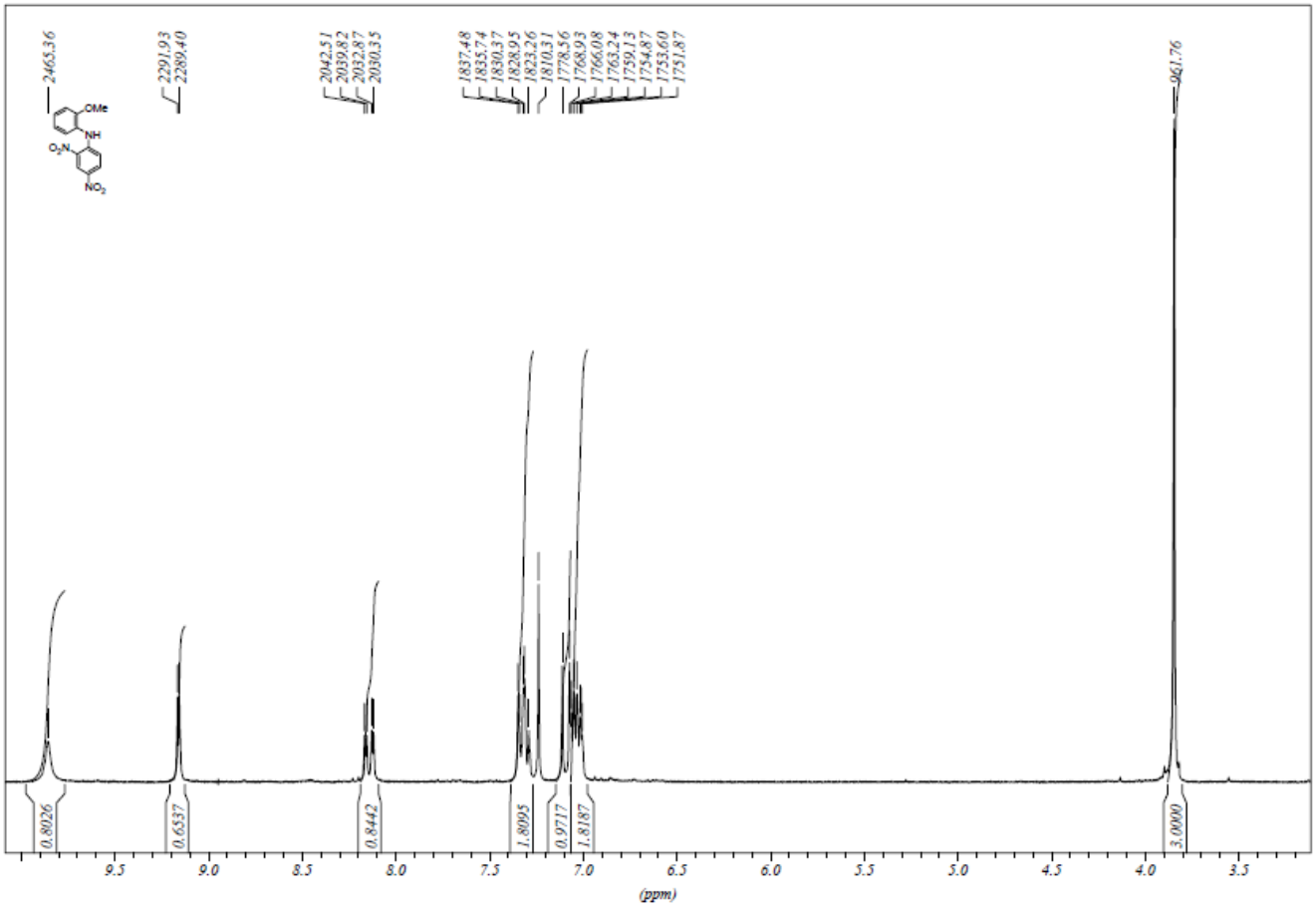

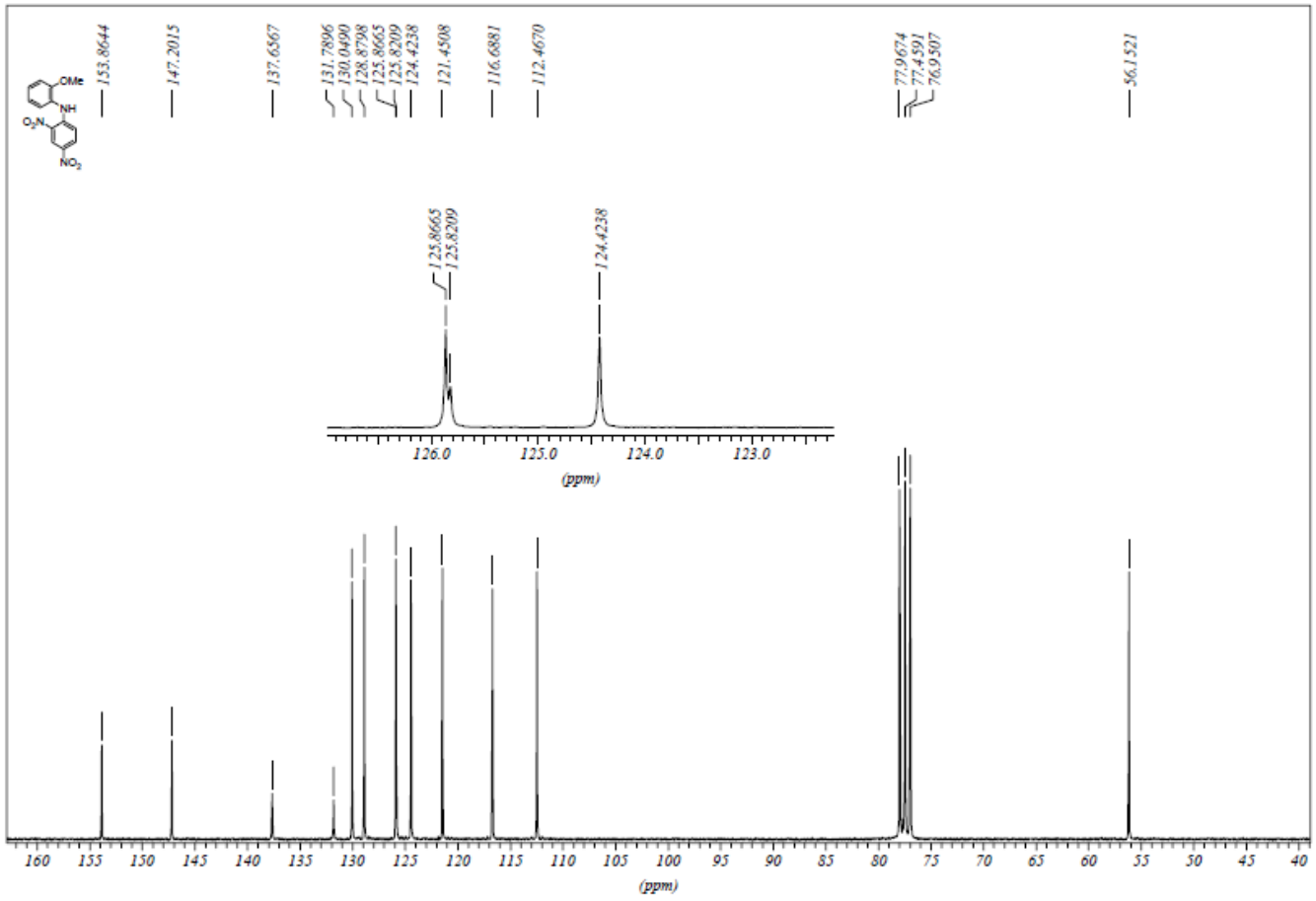

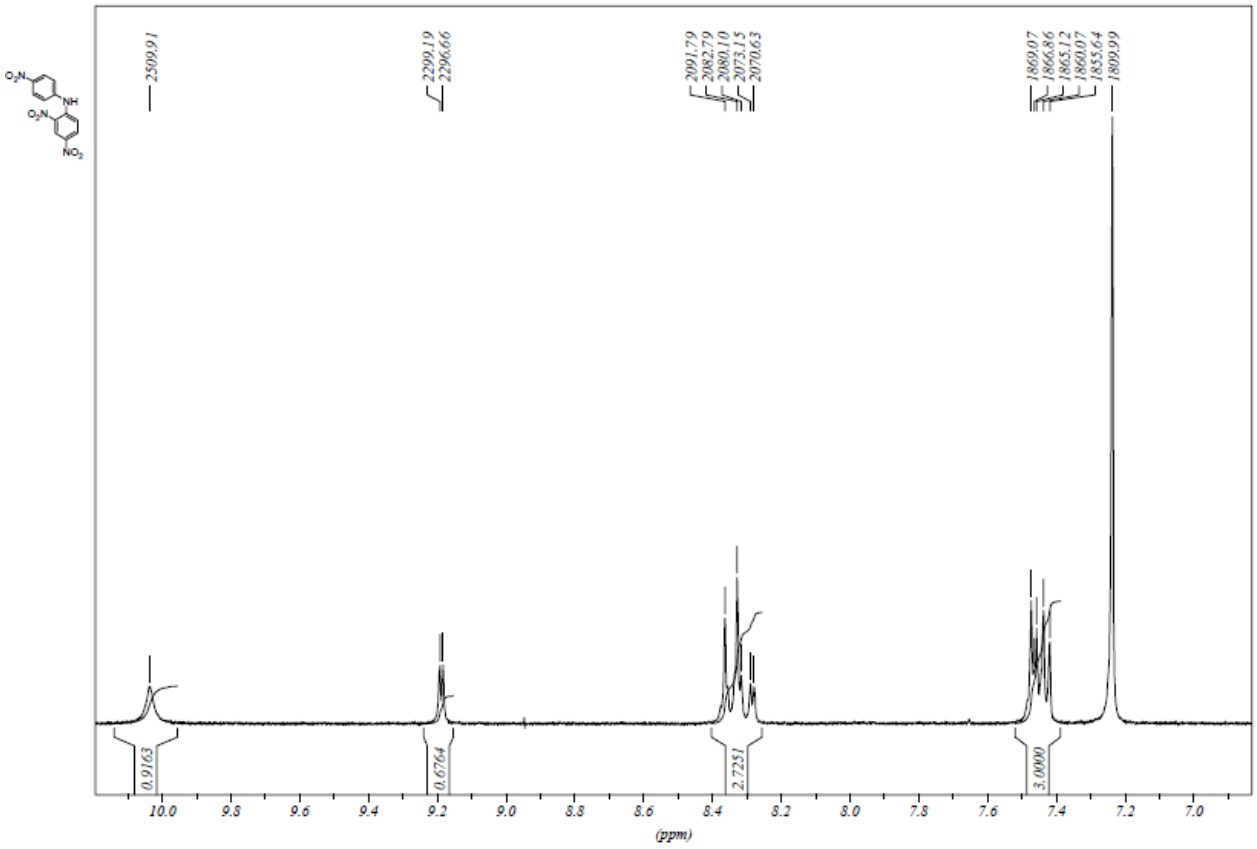

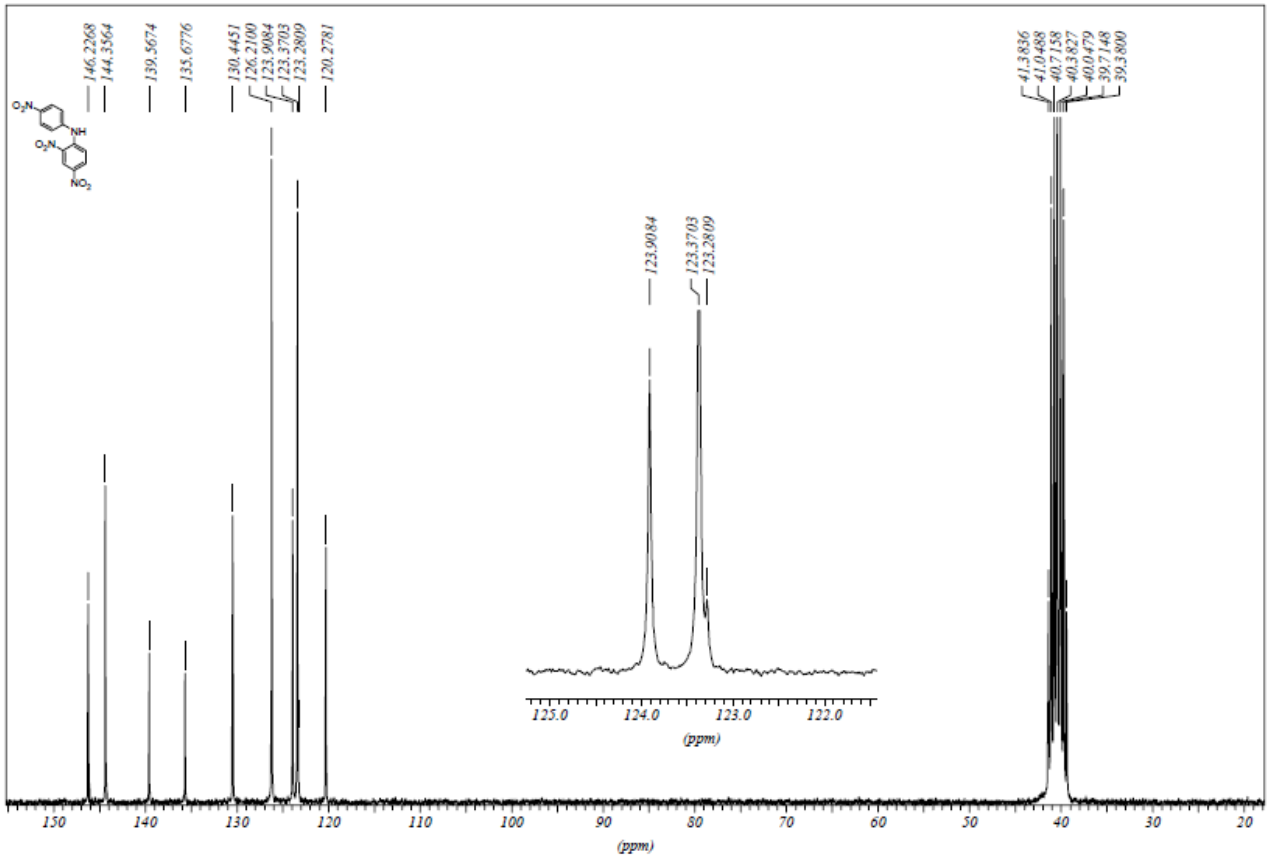

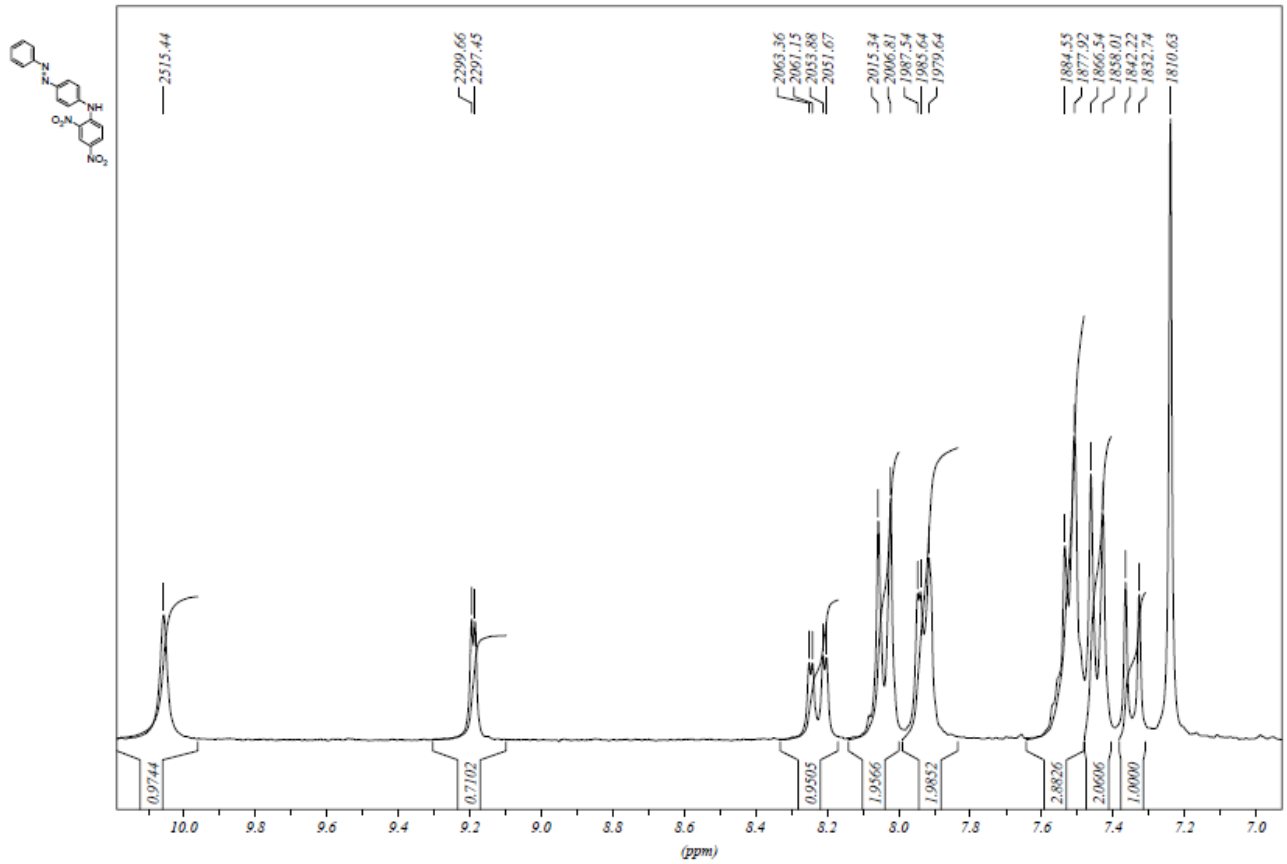

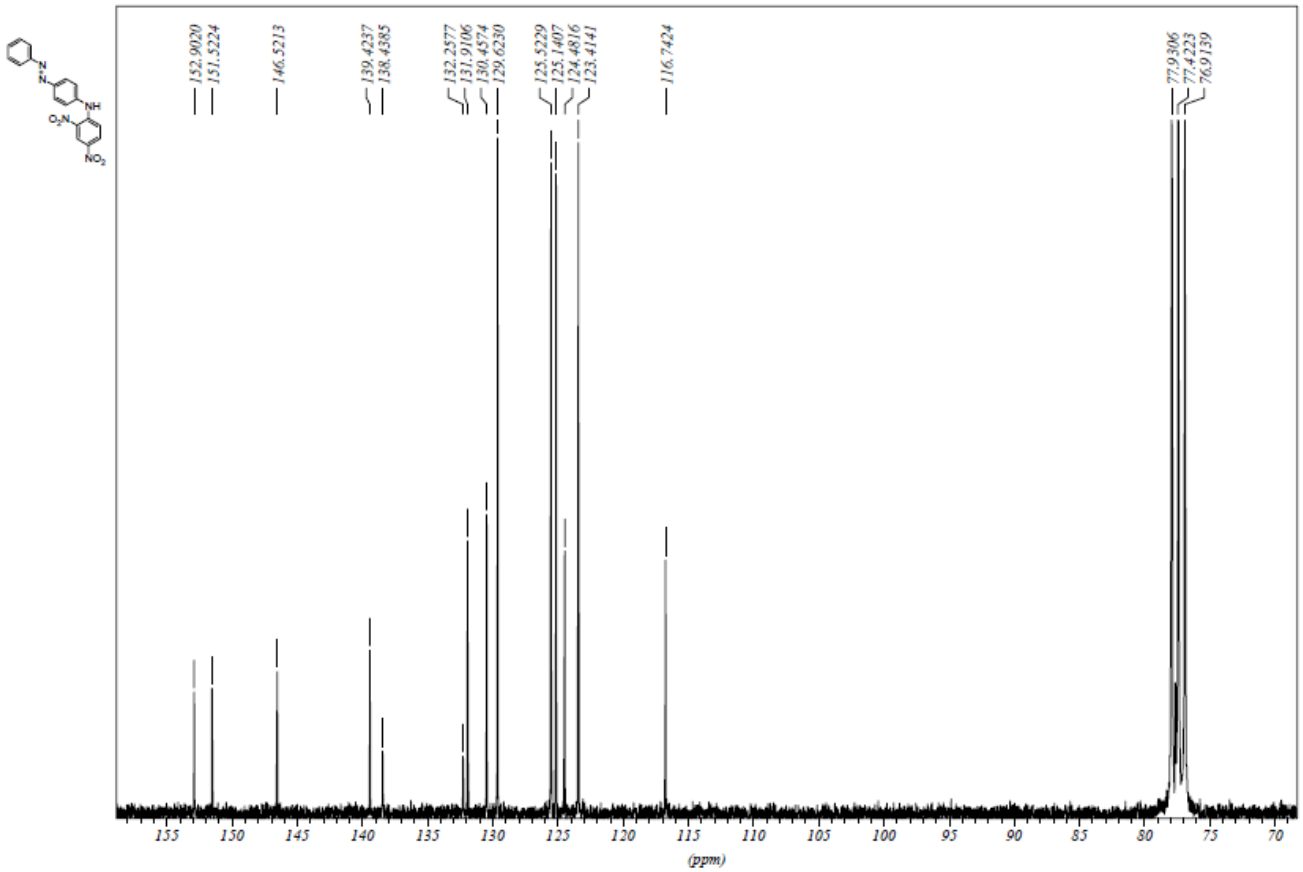

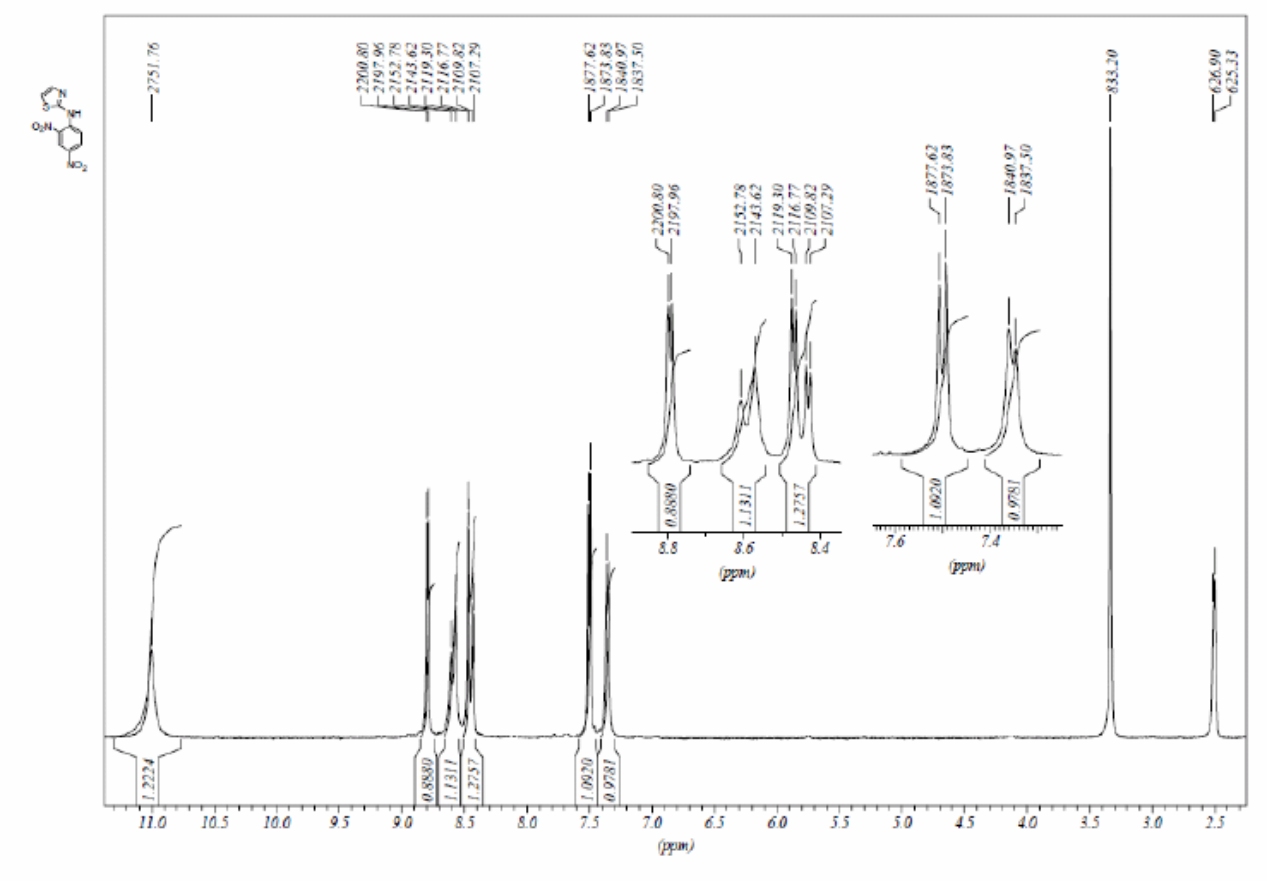

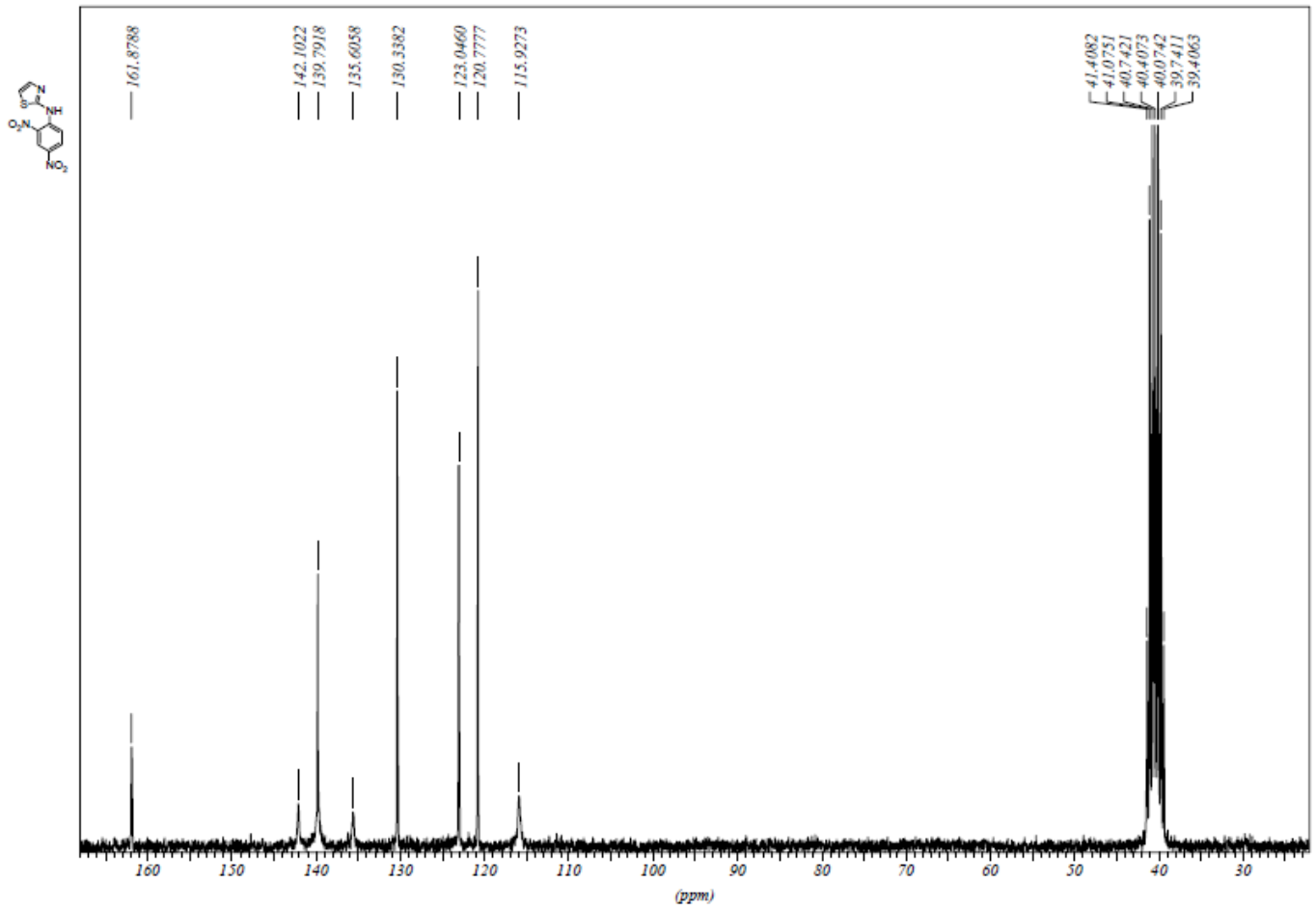

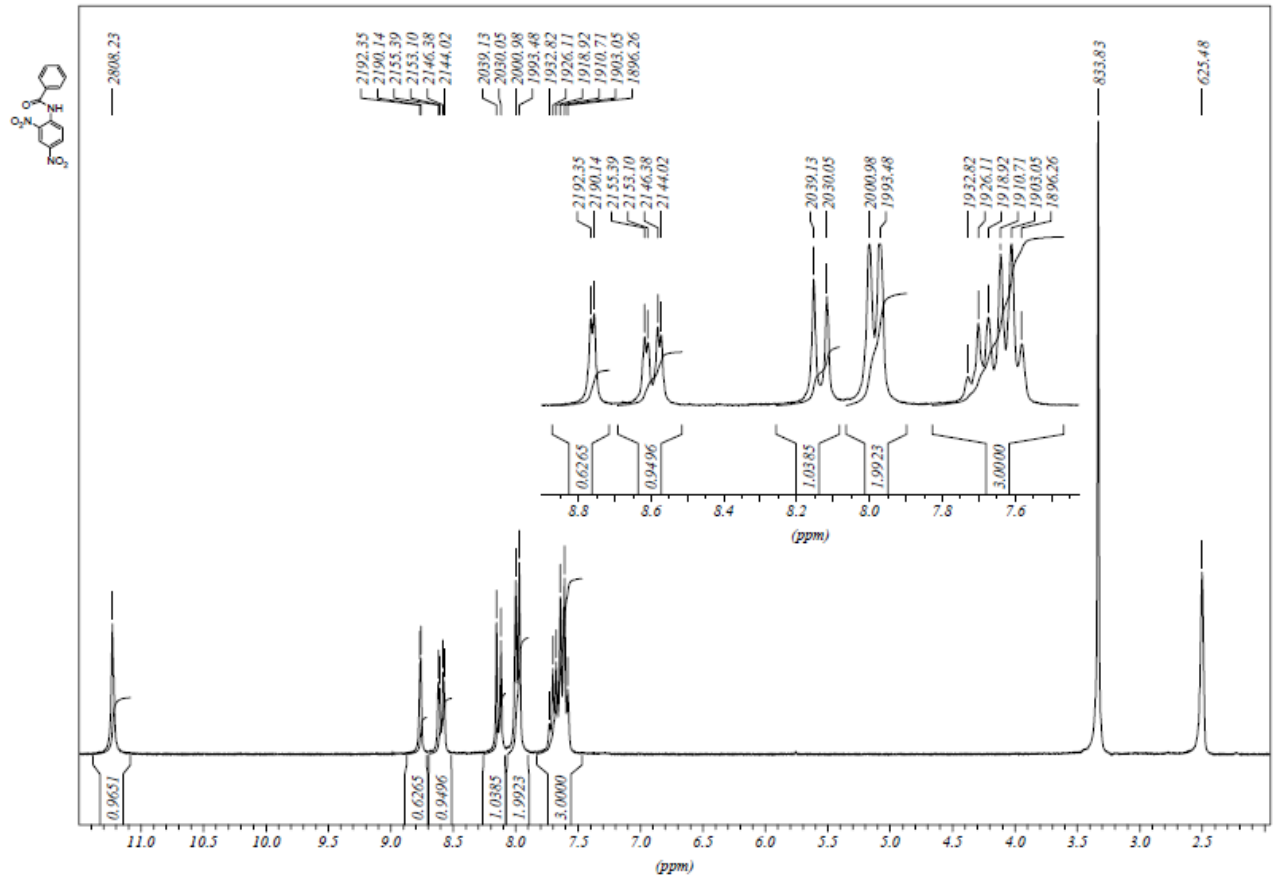

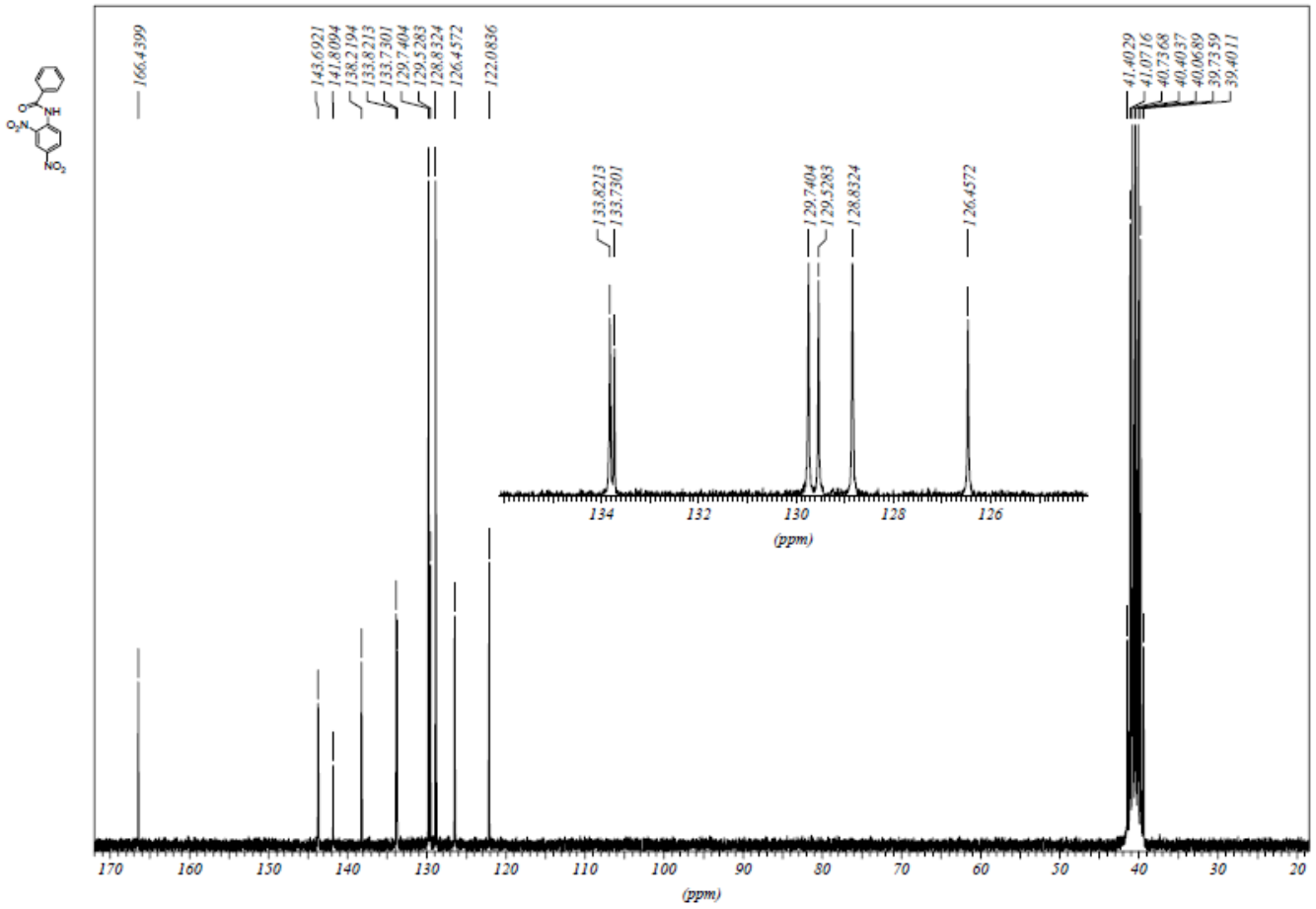

Table 1. Crystal data and structure refinement for N-(2,4-dinitrophenyl)pyridin-2-amine **4a** (C-80)

Identification code	fin
Empirical formula	C11 H8 N4 O4
Formula weight	260.21
Temperature	120(2) K
Wavelength	0.71073 A
Crystal system, space group	Triclinic, P -1
Unit cell dimensions	a = 9.8392(16) A alpha = 88.602(4) deg.
	b = 10.1624(16) A beta = 86.340(4) deg.
	c = 10.9012(18) A gamma = 87.703(4) deg.
Volume	1086.7(3) A^3
Z, Calculated density	4, 1.591 Mg/m^3
Absorption coefficient	0.125 mm^-1
F(000)	536
Crystal size	0.50 x 0.35 x 0.25 mm
Theta range for data collection	1.87 to 27.50 deg.
Limiting indices	-12<=h<=12, -13<=k<=13, -14<=l<=13
Reflections collected / unique	10765 / 4969 [R(int) = 0.0326]
Completeness to theta = 27.50	99.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.966 and 0.948
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4969 / 0 / 343
Goodness-of-fit on F^2	1.013
Final R indices [2975 rfls with I>2sigma(I)]	R1 = 0.0492, wR2 = 0.1031
R indices (all data)	R1 = 0.0777, wR2 = 0.1109
Largest diff. peak and hole	0.266 and -0.241 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for C-80.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
N(1')	3082(2)	5939(2)	1998(2)	27(1)
N(2')	-1286(2)	7927(2)	3349(2)	29(1)
N(3')	2171(2)	5808(2)	-490(1)	24(1)
N(4')	864(2)	6107(2)	-2228(2)	27(1)
O(1')	3304(2)	5855(2)	3094(1)	37(1)
O(2')	3968(1)	5645(2)	1175(1)	33(1)
O(3')	-2447(2)	8299(2)	3118(1)	36(1)
O(4')	-834(2)	7992(2)	4376(1)	41(1)
C(1')	1735(2)	6407(2)	1651(2)	23(1)
C(2')	887(2)	6916(2)	2593(2)	24(1)
C(3')	-397(2)	7387(2)	2349(2)	25(1)
C(4')	-856(2)	7348(2)	1169(2)	26(1)
C(5')	-13(2)	6840(2)	236(2)	26(1)
C(6')	1322(2)	6342(2)	428(2)	23(1)
C(7')	1989(2)	5644(2)	-1745(2)	24(1)
C(8')	3063(2)	4998(2)	-2423(2)	28(1)
C(9')	2951(2)	4867(2)	-3663(2)	31(1)
C(10')	1780(2)	5342(2)	-4183(2)	32(1)
C(11')	775(2)	5947(2)	-3437(2)	31(1)
O(1)	1110(1)	9162(1)	6738(1)	32(1)
O(2)	1802(2)	9037(2)	8578(1)	36(1)
O(3)	6079(2)	7244(2)	9500(1)	37(1)
O(4)	7628(1)	6784(1)	8049(1)	34(1)
N(1)	2011(2)	8931(2)	7462(2)	27(1)
N(2)	6475(2)	7189(2)	8407(2)	28(1)
N(3)	2897(2)	9014(2)	4857(1)	26(1)
N(4)	4222(2)	8800(2)	2979(2)	30(1)
C(1)	3369(2)	8493(2)	6981(2)	22(1)
C(2)	4245(2)	8053(2)	7858(2)	23(1)
C(3)	5546(2)	7635(2)	7484(2)	24(1)
C(4)	5991(2)	7660(2)	6242(2)	27(1)

C(5)	5118(2)	8102(2)	5378(2)	27(1)
C(6)	3774(2)	8543(2)	5704(2)	23(1)
C(7)	3081(2)	9216(2)	3582(2)	25(1)
C(8)	1996(2)	9846(2)	3009(2)	28(1)
C(9)	2096(2)	10019(2)	1755(2)	32(1)
C(10)	3278(2)	9582(2)	1106(2)	35(1)
C(11)	4305(2)	8999(2)	1751(2)	34(1)

Table 3. Bond lengths [A] and angles [deg] for C-80.

N(1')-O(1')	1.229(2)
N(1')-O(2')	1.244(2)
N(1')-C(1')	1.459(3)
N(2')-O(3')	1.229(2)
N(2')-O(4')	1.235(2)
N(2')-C(3')	1.457(2)
N(3')-C(6')	1.370(2)
N(3')-C(7')	1.405(2)
N(3')-H(3N')	0.8994
N(4')-C(7')	1.320(3)
N(4')-C(11')	1.341(2)
C(1')-C(2')	1.380(3)
C(1')-C(6')	1.422(3)
C(2')-C(3')	1.372(3)
C(2')-H(2'A)	0.9300
C(3')-C(4')	1.392(3)
C(4')-C(5')	1.369(3)
C(4')-H(4'A)	0.9300
C(5')-C(6')	1.415(3)
C(5')-H(5'A)	0.9300
C(7')-C(8')	1.401(3)
C(8')-C(9')	1.372(3)
C(8')-H(8'A)	0.9300
C(9')-C(10')	1.381(3)
C(9')-H(9'A)	0.9300
C(10')-C(11')	1.375(3)
C(10')-H(10A)	0.9300
C(11')-H(11A)	0.9300

O(1)-N(1)	1.236(2)
O(2)-N(1)	1.228(2)
O(3)-N(2)	1.231(2)
O(4)-N(2)	1.233(2)
N(1)-C(1)	1.460(2)
N(2)-C(3)	1.455(2)
N(3)-C(6)	1.369(2)
N(3)-C(7)	1.401(2)
N(3)-H(3N)	0.9013
N(4)-C(7)	1.324(2)
N(4)-C(11)	1.347(3)
C(1)-C(2)	1.381(3)
C(1)-C(6)	1.424(3)
C(2)-C(3)	1.372(3)
C(2)-H(2A)	0.9300
C(3)-C(4)	1.396(3)
C(4)-C(5)	1.370(3)
C(4)-H(4A)	0.9300
C(5)-C(6)	1.406(3)
C(5)-H(5A)	0.9300
C(7)-C(8)	1.396(3)
C(8)-C(9)	1.372(3)
C(8)-H(8A)	0.9300
C(9)-C(10)	1.385(3)
C(9)-H(9A)	0.9300
C(10)-C(11)	1.374(3)
C(10)-H(10B)	0.9300
C(11)-H(11B)	0.9300

O(1')-N(1')-O(2')	122.20(17)
O(1')-N(1')-C(1')	118.84(16)
O(2')-N(1')-C(1')	118.96(16)
O(3')-N(2')-O(4')	123.68(18)
O(3')-N(2')-C(3')	117.81(18)
O(4')-N(2')-C(3')	118.51(17)
C(6')-N(3')-C(7')	131.25(17)
C(6')-N(3')-H(3N')	111.6
C(7')-N(3')-H(3N')	117.1
C(7')-N(4')-C(11')	116.99(18)
C(2')-C(1')-C(6')	122.16(18)

C(2')-C(1')-N(1')	115.32(17)
C(6')-C(1')-N(1')	122.52(17)
C(3')-C(2')-C(1')	119.33(19)
C(3')-C(2')-H(2'A)	120.3
C(1')-C(2')-H(2'A)	120.3
C(2')-C(3')-C(4')	120.97(18)
C(2')-C(3')-N(2')	119.01(18)
C(4')-C(3')-N(2')	120.01(18)
C(5')-C(4')-C(3')	119.62(19)
C(5')-C(4')-H(4'A)	120.2
C(3')-C(4')-H(4'A)	120.2
C(4')-C(5')-C(6')	122.07(19)
C(4')-C(5')-H(5'A)	119.0
C(6')-C(5')-H(5'A)	119.0
N(3')-C(6')-C(5')	122.87(18)
N(3')-C(6')-C(1')	121.28(17)
C(5')-C(6')-C(1')	115.84(18)
N(4')-C(7')-C(8')	123.57(18)
N(4')-C(7')-N(3')	120.16(18)
C(8')-C(7')-N(3')	116.25(17)
C(9')-C(8')-C(7')	118.12(19)
C(9')-C(8')-H(8'A)	120.9
C(7')-C(8')-H(8'A)	120.9
C(8')-C(9')-C(10')	119.1(2)
C(8')-C(9')-H(9'A)	120.5
C(10')-C(9')-H(9'A)	120.5
C(11')-C(10')-C(9')	118.47(19)
C(11')-C(10')-H(10A)	120.8
C(9')-C(10')-H(10A)	120.8
N(4')-C(11')-C(10')	123.75(19)
N(4')-C(11')-H(11A)	118.1
C(10')-C(11')-H(11A)	118.1
O(2)-N(1)-O(1)	122.36(17)
O(2)-N(1)-C(1)	118.46(16)
O(1)-N(1)-C(1)	119.17(16)
O(3)-N(2)-O(4)	123.44(17)
O(3)-N(2)-C(3)	118.66(17)
O(4)-N(2)-C(3)	117.89(16)
C(6)-N(3)-C(7)	131.36(17)
C(6)-N(3)-H(3N)	112.5

C(7)-N(3)-H(3N)	116.0
C(7)-N(4)-C(11)	116.81(18)
C(2)-C(1)-C(6)	122.28(18)
C(2)-C(1)-N(1)	115.21(17)
C(6)-C(1)-N(1)	122.50(17)
C(3)-C(2)-C(1)	118.91(18)
C(3)-C(2)-H(2A)	120.5
C(1)-C(2)-H(2A)	120.5
C(2)-C(3)-C(4)	121.01(18)
C(2)-C(3)-N(2)	118.96(17)
C(4)-C(3)-N(2)	120.01(18)
C(5)-C(4)-C(3)	119.80(19)
C(5)-C(4)-H(4A)	120.1
C(3)-C(4)-H(4A)	120.1
C(4)-C(5)-C(6)	121.83(19)
C(4)-C(5)-H(5A)	119.1
C(6)-C(5)-H(5A)	119.1
N(3)-C(6)-C(5)	122.68(18)
N(3)-C(6)-C(1)	121.15(17)
C(5)-C(6)-C(1)	116.17(17)
N(4)-C(7)-C(8)	123.35(18)
N(4)-C(7)-N(3)	120.42(18)
C(8)-C(7)-N(3)	116.22(18)
C(9)-C(8)-C(7)	118.7(2)
C(9)-C(8)-H(8A)	120.6
C(7)-C(8)-H(8A)	120.6
C(8)-C(9)-C(10)	118.8(2)
C(8)-C(9)-H(9A)	120.6
C(10)-C(9)-H(9A)	120.6
C(11)-C(10)-C(9)	118.4(2)
C(11)-C(10)-H(10B)	120.8
C(9)-C(10)-H(10B)	120.8
N(4)-C(11)-C(10)	123.9(2)
N(4)-C(11)-H(11B)	118.1
C(10)-C(11)-H(11B)	118.1

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for C-80.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
N(1')	28(1)	27(1)	26(1)	-1(1)	-4(1)	-1(1)
N(2')	33(1)	24(1)	31(1)	0(1)	4(1)	0(1)
N(3')	24(1)	28(1)	21(1)	-1(1)	-3(1)	3(1)
N(4')	29(1)	29(1)	24(1)	1(1)	-3(1)	1(1)
O(1')	38(1)	49(1)	24(1)	1(1)	-9(1)	5(1)
O(2')	26(1)	45(1)	29(1)	-1(1)	-1(1)	4(1)
O(3')	29(1)	35(1)	42(1)	-3(1)	6(1)	2(1)
O(4')	52(1)	44(1)	25(1)	-2(1)	2(1)	7(1)
C(1')	24(1)	19(1)	25(1)	4(1)	-1(1)	-1(1)
C(2')	27(1)	21(1)	23(1)	2(1)	-1(1)	-4(1)
C(3')	29(1)	20(1)	25(1)	-1(1)	6(1)	-1(1)
C(4')	21(1)	26(1)	30(1)	3(1)	1(1)	1(1)
C(5')	26(1)	28(1)	24(1)	3(1)	-1(1)	0(1)
C(6')	26(1)	19(1)	24(1)	3(1)	0(1)	-2(1)
C(7')	27(1)	23(1)	22(1)	1(1)	0(1)	-5(1)
C(8')	26(1)	28(1)	29(1)	1(1)	0(1)	-3(1)
C(9')	35(1)	31(1)	28(1)	-5(1)	7(1)	-4(1)
C(10')	43(1)	34(1)	20(1)	2(1)	-1(1)	-5(1)
C(11')	35(1)	33(1)	26(1)	3(1)	-8(1)	1(1)
O(1)	24(1)	40(1)	31(1)	3(1)	-3(1)	4(1)
O(2)	34(1)	46(1)	26(1)	-5(1)	4(1)	5(1)
O(3)	42(1)	43(1)	25(1)	2(1)	-4(1)	7(1)
O(4)	26(1)	36(1)	38(1)	2(1)	-4(1)	6(1)
N(1)	27(1)	26(1)	27(1)	-2(1)	2(1)	0(1)
N(2)	30(1)	24(1)	29(1)	1(1)	-2(1)	1(1)
N(3)	22(1)	32(1)	24(1)	1(1)	1(1)	4(1)
N(4)	33(1)	31(1)	25(1)	-2(1)	1(1)	1(1)
C(1)	20(1)	20(1)	26(1)	-1(1)	3(1)	0(1)
C(2)	27(1)	20(1)	23(1)	-1(1)	1(1)	-2(1)
C(3)	24(1)	21(1)	27(1)	1(1)	-5(1)	-3(1)
C(4)	23(1)	27(1)	29(1)	-2(1)	0(1)	2(1)

C(5)	29(1)	28(1)	23(1)	-3(1)	1(1)	-1(1)
C(6)	25(1)	20(1)	25(1)	-1(1)	-3(1)	-1(1)
C(7)	30(1)	24(1)	22(1)	-1(1)	-2(1)	-4(1)
C(8)	27(1)	29(1)	29(1)	0(1)	-5(1)	-2(1)
C(9)	37(1)	29(1)	32(1)	3(1)	-12(1)	-5(1)
C(10)	51(2)	32(1)	22(1)	0(1)	-4(1)	-7(1)
C(11)	40(1)	34(1)	27(1)	-5(1)	4(1)	-1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for C-80.

	x	y	z	U(eq)
H(3N')	2989	5560	-219	29
H(2'A)	1181	6941	3386	28
H(4'A)	-1730	7666	1016	31
H(5'A)	-328	6820	-550	31
H(8'A)	3829	4667	-2045	33
H(9'A)	3655	4465	-4146	37
H(10A)	1673	5254	-5018	39
H(11A)	-12	6265	-3792	37
H(3N)	2080	9280	5205	31
H(2A)	3957	8039	8687	28
H(4A)	6876	7378	6002	32
H(5A)	5424	8110	4553	32
H(8A)	1220	10143	3469	34
H(9A)	1384	10423	1347	39
H(10B)	3374	9680	255	42
H(11B)	5105	8727	1313	41

Table 6. Torsion angles [deg] for C-80.

O(1')-N(1')-C(1')-C(2')	9.9(3)
O(2')-N(1')-C(1')-C(2')	-169.66(17)
O(1')-N(1')-C(1')-C(6')	-169.99(18)
O(2')-N(1')-C(1')-C(6')	10.4(3)
C(6')-C(1')-C(2')-C(3')	-0.4(3)
N(1')-C(1')-C(2')-C(3')	179.61(17)
C(1')-C(2')-C(3')-C(4')	0.5(3)
C(1')-C(2')-C(3')-N(2')	-179.87(17)
O(3')-N(2')-C(3')-C(2')	-177.45(17)
O(4')-N(2')-C(3')-C(2')	2.9(3)
O(3')-N(2')-C(3')-C(4')	2.2(3)
O(4')-N(2')-C(3')-C(4')	-177.42(18)
C(2')-C(3')-C(4')-C(5')	-0.4(3)

N(2')-C(3')-C(4')-C(5')	180.00(17)
C(3')-C(4')-C(5')-C(6')	0.2(3)
C(7')-N(3')-C(6')-C(5')	1.3(3)
C(7')-N(3')-C(6')-C(1')	-179.82(18)
C(4')-C(5')-C(6')-N(3')	178.76(18)
C(4')-C(5')-C(6')-C(1')	-0.1(3)
C(2')-C(1')-C(6')-N(3')	-178.65(18)
N(1')-C(1')-C(6')-N(3')	1.3(3)
C(2')-C(1')-C(6')-C(5')	0.3(3)
N(1')-C(1')-C(6')-C(5')	-179.80(17)
C(11')-N(4')-C(7')-C(8')	-0.6(3)
C(11')-N(4')-C(7')-N(3')	178.22(17)
C(6')-N(3')-C(7')-N(4')	3.7(3)
C(6')-N(3')-C(7')-C(8')	-177.39(19)
N(4')-C(7')-C(8')-C(9')	1.6(3)
N(3')-C(7')-C(8')-C(9')	-177.27(17)
C(7')-C(8')-C(9')-C(10')	-1.7(3)
C(8')-C(9')-C(10')-C(11')	1.0(3)
C(7')-N(4')-C(11')-C(10')	-0.3(3)
C(9')-C(10')-C(11')-N(4')	0.0(3)
O(2)-N(1)-C(1)-C(2)	9.1(2)
O(1)-N(1)-C(1)-C(2)	-169.86(17)
O(2)-N(1)-C(1)-C(6)	-169.55(18)
O(1)-N(1)-C(1)-C(6)	11.5(3)
C(6)-C(1)-C(2)-C(3)	-0.7(3)
N(1)-C(1)-C(2)-C(3)	-179.39(16)
C(1)-C(2)-C(3)-C(4)	0.5(3)
C(1)-C(2)-C(3)-N(2)	179.10(17)
O(3)-N(2)-C(3)-C(2)	-2.5(3)
O(4)-N(2)-C(3)-C(2)	178.04(17)
O(3)-N(2)-C(3)-C(4)	176.12(18)
O(4)-N(2)-C(3)-C(4)	-3.3(3)
C(2)-C(3)-C(4)-C(5)	-0.3(3)
N(2)-C(3)-C(4)-C(5)	-178.90(18)
C(3)-C(4)-C(5)-C(6)	0.3(3)
C(7)-N(3)-C(6)-C(5)	-2.6(3)
C(7)-N(3)-C(6)-C(1)	176.46(19)
C(4)-C(5)-C(6)-N(3)	178.56(18)
C(4)-C(5)-C(6)-C(1)	-0.5(3)
C(2)-C(1)-C(6)-N(3)	-178.37(18)

N(1)-C(1)-C(6)-N(3)	0.2(3)
C(2)-C(1)-C(6)-C(5)	0.7(3)
N(1)-C(1)-C(6)-C(5)	179.30(17)
C(11)-N(4)-C(7)-C(8)	-0.5(3)
C(11)-N(4)-C(7)-N(3)	178.22(18)
C(6)-N(3)-C(7)-N(4)	7.6(3)
C(6)-N(3)-C(7)-C(8)	-173.58(19)
N(4)-C(7)-C(8)-C(9)	1.6(3)
N(3)-C(7)-C(8)-C(9)	-177.22(18)
C(7)-C(8)-C(9)-C(10)	-1.0(3)
C(8)-C(9)-C(10)-C(11)	-0.4(3)
C(7)-N(4)-C(11)-C(10)	-1.1(3)
C(9)-C(10)-C(11)-N(4)	1.5(3)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for C-80 [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(3)-H(3B)...O(11)#1	0.88	2.26	3.101(5)	159
N(3')-H(3N')...O(2')#1	0.899	1.857	2.612(3)	140
N(3')-H(3N')...N(1')#1	0.899	2.464	2.918(3)	112
N(3)-H(3N)...O(1)#1	0.901	1.875	2.618(3)	138
N(3)-H(3N)...N(1)#1	0.901	2.474	2.917(3)	111

Symmetry transformations used to generate equivalent atoms:
#1 x,y,z

Table 1. Crystal data and structure refinement for 2,4-dinitro-N-(4-(phenyldiazenyl)phenyl)aniline **4k** (gull).

Identification code	mon
Empirical formula	C18 H13 N5 O4
Formula weight	363.33
Temperature	120(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P 21/n
Unit cell dimensions	a = 8.5347(9) A alpha = 90 deg.
	b = 16.2980(17) A beta = 96.204(2) deg.
	c = 11.6632(13) A gamma = 90 deg.
Volume	1612.8(3) A^3
Z, Calculated density	4, 1.496 Mg/m^3
Absorption coefficient	0.110 mm^-1
F(000)	752
Crystal size	0.55 x 0.45 x 0.35 mm
Theta range for data collection	2.16 to 27.00 deg.
Limiting indices	-10<=h<=10, -19<=k<=20, -14<=l<=14
Reflections collected / unique	11645 / 3503 [R(int) = 0.0388]
Completeness to theta = 27.00	99.5 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.962 and 0.942
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3503 / 0 / 248
Goodness-of-fit on F^2	1.008
Final R indices [3022 rfls with I>2sigma(I)]	R1 = 0.0509, wR2 = 0.1027
R indices (all data)	R1 = 0.0569, wR2 = 0.1057
Largest diff. peak and hole	0.358 and -0.289 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for gul1.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	2742(2)	1941(1)	3638(1)	29(1)
O(2)	2417(2)	3221(1)	4060(1)	32(1)
O(3)	4247(2)	5400(1)	1957(1)	37(1)
O(4)	6229(2)	5200(1)	973(1)	35(1)
N(1)	3002(2)	2681(1)	3498(1)	23(1)
N(2)	5172(2)	4946(1)	1516(1)	27(1)
N(3)	4699(2)	1535(1)	2146(1)	22(1)
N(4)	8069(2)	-1061(1)	395(1)	23(1)
N(5)	7530(2)	-1775(1)	499(1)	23(1)
C(1)	4791(2)	2359(1)	1975(1)	20(1)
C(2)	4002(2)	2934(1)	2628(1)	20(1)
C(3)	4121(2)	3775(1)	2474(1)	21(1)
C(4)	5022(2)	4064(1)	1653(1)	22(1)
C(5)	5763(2)	3532(1)	951(1)	23(1)
C(6)	5635(2)	2700(1)	1102(1)	22(1)
C(7)	5590(2)	897(1)	1720(1)	21(1)
C(8)	4893(2)	117(1)	1636(1)	23(1)
C(9)	5681(2)	-544(1)	1237(1)	23(1)
C(10)	7193(2)	-434(1)	893(1)	21(1)
C(11)	7893(2)	335(1)	992(1)	23(1)
C(12)	7125(2)	995(1)	1431(1)	23(1)
C(13)	8376(2)	-2394(1)	-47(1)	22(1)
C(14)	9311(2)	-2234(1)	-938(2)	25(1)
C(15)	10070(2)	-2881(1)	-1416(2)	27(1)
C(16)	9915(2)	-3678(1)	-1014(2)	29(1)
C(17)	8952(2)	-3835(1)	-153(2)	28(1)
C(18)	8167(2)	-3194(1)	321(2)	25(1)

Table 3. Bond lengths [A] and angles [deg] for gul1.

O(1)-N(1)	1.2405(19)
O(2)-N(1)	1.2344(19)

O(3)-N(2)	1.234(2)
O(4)-N(2)	1.229(2)
N(1)-C(2)	1.454(2)
N(2)-C(4)	1.454(2)
N(3)-C(1)	1.362(2)
N(3)-C(7)	1.409(2)
N(3)-H(3)	0.90(2)
N(4)-N(5)	1.261(2)
N(4)-C(10)	1.426(2)
N(5)-C(13)	1.430(2)
C(1)-C(2)	1.422(2)
C(1)-C(6)	1.422(2)
C(2)-C(3)	1.387(2)
C(3)-C(4)	1.375(2)
C(3)-H(3A)	0.9500
C(4)-C(5)	1.391(2)
C(5)-C(6)	1.373(2)
C(5)-H(5A)	0.9500
C(6)-H(6A)	0.9500
C(7)-C(12)	1.396(2)
C(7)-C(8)	1.403(2)
C(8)-C(9)	1.378(2)
C(8)-H(8A)	0.9500
C(9)-C(10)	1.403(2)
C(9)-H(9A)	0.9500
C(10)-C(11)	1.388(2)
C(11)-C(12)	1.386(2)
C(11)-H(11A)	0.9500
C(12)-H(12A)	0.9500
C(13)-C(18)	1.391(2)
C(13)-C(14)	1.402(2)
C(14)-C(15)	1.385(3)
C(14)-H(14A)	0.9500
C(15)-C(16)	1.393(3)
C(15)-H(15A)	0.9500
C(16)-C(17)	1.388(3)
C(16)-H(16A)	0.9500
C(17)-C(18)	1.388(2)
C(17)-H(17A)	0.9500
C(18)-H(18A)	0.9500

O(2)-N(1)-O(1)	122.26(14)
O(2)-N(1)-C(2)	118.04(14)
O(1)-N(1)-C(2)	119.68(14)
O(4)-N(2)-O(3)	123.44(16)
O(4)-N(2)-C(4)	117.89(15)
O(3)-N(2)-C(4)	118.67(15)
C(1)-N(3)-C(7)	129.32(15)
C(1)-N(3)-H(3)	113.9(15)
C(7)-N(3)-H(3)	116.8(15)
N(5)-N(4)-C(10)	114.12(14)
N(4)-N(5)-C(13)	113.59(14)
N(3)-C(1)-C(2)	122.23(15)
N(3)-C(1)-C(6)	122.15(15)
C(2)-C(1)-C(6)	115.58(15)
C(3)-C(2)-C(1)	122.42(15)
C(3)-C(2)-N(1)	115.30(14)
C(1)-C(2)-N(1)	122.28(15)
C(4)-C(3)-C(2)	118.91(16)
C(4)-C(3)-H(3A)	120.5
C(2)-C(3)-H(3A)	120.5
C(3)-C(4)-C(5)	121.35(16)
C(3)-C(4)-N(2)	118.58(15)
C(5)-C(4)-N(2)	120.06(15)
C(6)-C(5)-C(4)	119.52(15)
C(6)-C(5)-H(5A)	120.2
C(4)-C(5)-H(5A)	120.2
C(5)-C(6)-C(1)	122.07(16)
C(5)-C(6)-H(6A)	119.0
C(1)-C(6)-H(6A)	119.0
C(12)-C(7)-C(8)	119.18(15)
C(12)-C(7)-N(3)	123.89(16)
C(8)-C(7)-N(3)	116.88(15)
C(9)-C(8)-C(7)	120.94(15)
C(9)-C(8)-H(8A)	119.5
C(7)-C(8)-H(8A)	119.5
C(8)-C(9)-C(10)	119.61(16)
C(8)-C(9)-H(9A)	120.2
C(10)-C(9)-H(9A)	120.2
C(11)-C(10)-C(9)	119.50(15)

C(11)-C(10)-N(4)	116.16(15)
C(9)-C(10)-N(4)	124.29(15)
C(12)-C(11)-C(10)	120.96(15)
C(12)-C(11)-H(11A)	119.5
C(10)-C(11)-H(11A)	119.5
C(11)-C(12)-C(7)	119.67(16)
C(11)-C(12)-H(12A)	120.2
C(7)-C(12)-H(12A)	120.2
C(18)-C(13)-C(14)	120.38(16)
C(18)-C(13)-N(5)	115.86(15)
C(14)-C(13)-N(5)	123.69(16)
C(15)-C(14)-C(13)	118.99(17)
C(15)-C(14)-H(14A)	120.5
C(13)-C(14)-H(14A)	120.5
C(14)-C(15)-C(16)	120.63(17)
C(14)-C(15)-H(15A)	119.7
C(16)-C(15)-H(15A)	119.7
C(17)-C(16)-C(15)	120.07(17)
C(17)-C(16)-H(16A)	120.0
C(15)-C(16)-H(16A)	120.0
C(16)-C(17)-C(18)	119.85(18)
C(16)-C(17)-H(17A)	120.1
C(18)-C(17)-H(17A)	120.1
C(17)-C(18)-C(13)	119.98(16)
C(17)-C(18)-H(18A)	120.0
C(13)-C(18)-H(18A)	120.0

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for gul1.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	31(1)	27(1)	30(1)	3(1)	14(1)	-3(1)
O(2)	36(1)	33(1)	30(1)	-2(1)	18(1)	5(1)
O(3)	44(1)	25(1)	42(1)	0(1)	8(1)	8(1)
O(4)	41(1)	31(1)	35(1)	5(1)	10(1)	-11(1)

N(1)	21(1)	27(1)	21(1)	0(1)	6(1)	1(1)
N(2)	33(1)	23(1)	25(1)	2(1)	2(1)	-2(1)
N(3)	24(1)	20(1)	23(1)	1(1)	9(1)	1(1)
N(4)	23(1)	21(1)	26(1)	-1(1)	3(1)	1(1)
N(5)	24(1)	21(1)	25(1)	-1(1)	3(1)	0(1)
C(1)	19(1)	21(1)	20(1)	0(1)	3(1)	0(1)
C(2)	17(1)	23(1)	19(1)	0(1)	5(1)	-1(1)
C(3)	20(1)	23(1)	21(1)	-1(1)	3(1)	2(1)
C(4)	23(1)	19(1)	24(1)	1(1)	1(1)	0(1)
C(5)	21(1)	26(1)	22(1)	4(1)	6(1)	0(1)
C(6)	23(1)	24(1)	20(1)	0(1)	6(1)	2(1)
C(7)	24(1)	20(1)	19(1)	1(1)	5(1)	3(1)
C(8)	23(1)	24(1)	22(1)	2(1)	6(1)	-1(1)
C(9)	26(1)	20(1)	24(1)	1(1)	5(1)	-1(1)
C(10)	25(1)	19(1)	19(1)	0(1)	4(1)	2(1)
C(11)	21(1)	23(1)	24(1)	1(1)	5(1)	0(1)
C(12)	23(1)	20(1)	25(1)	-1(1)	4(1)	-1(1)
C(13)	20(1)	22(1)	24(1)	-4(1)	2(1)	0(1)
C(14)	22(1)	25(1)	27(1)	-3(1)	3(1)	-4(1)
C(15)	23(1)	33(1)	27(1)	-6(1)	6(1)	-4(1)
C(16)	26(1)	27(1)	34(1)	-10(1)	4(1)	2(1)
C(17)	29(1)	23(1)	31(1)	-3(1)	2(1)	1(1)
C(18)	24(1)	25(1)	25(1)	-2(1)	4(1)	-2(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for gul1.

	x	y	z	U(eq)
H(3)	3960(30)	1387(14)	2610(20)	41(6)
H(3A)	3589	4144	2929	26
H(5A)	6353	3743	371	27
H(6A)	6126	2341	608	27
H(8A)	3863	44	1857	27
H(9A)	5204	-1071	1195	27
H(11A)	8914	410	754	27
H(12A)	7639	1510	1534	27
H(14A)	9423	-1690	-1211	30
H(15A)	10702	-2779	-2023	33

H(16A)	10468	-4115	-1329	35
H(17A)	8831	-4381	111	33
H(18A)	7487	-3302	897	30

Table 6. Torsion angles [deg] for gul1.

C(10)-N(4)-N(5)-C(13)	177.44(13)
C(7)-N(3)-C(1)-C(2)	-168.53(16)
C(7)-N(3)-C(1)-C(6)	13.8(3)
N(3)-C(1)-C(2)-C(3)	178.49(16)
C(6)-C(1)-C(2)-C(3)	-3.7(2)
N(3)-C(1)-C(2)-N(1)	-2.1(2)
C(6)-C(1)-C(2)-N(1)	175.75(14)
O(2)-N(1)-C(2)-C(3)	-3.6(2)
O(1)-N(1)-C(2)-C(3)	175.14(15)
O(2)-N(1)-C(2)-C(1)	176.91(15)
O(1)-N(1)-C(2)-C(1)	-4.3(2)
C(1)-C(2)-C(3)-C(4)	0.6(2)
N(1)-C(2)-C(3)-C(4)	-178.83(14)
C(2)-C(3)-C(4)-C(5)	2.4(2)
C(2)-C(3)-C(4)-N(2)	-178.56(15)
O(4)-N(2)-C(4)-C(3)	164.50(15)
O(3)-N(2)-C(4)-C(3)	-14.8(2)
O(4)-N(2)-C(4)-C(5)	-16.4(2)
O(3)-N(2)-C(4)-C(5)	164.26(16)
C(3)-C(4)-C(5)-C(6)	-2.0(3)
N(2)-C(4)-C(5)-C(6)	178.89(15)
C(4)-C(5)-C(6)-C(1)	-1.3(3)
N(3)-C(1)-C(6)-C(5)	-178.16(16)
C(2)-C(1)-C(6)-C(5)	4.0(2)
C(1)-N(3)-C(7)-C(12)	27.2(3)
C(1)-N(3)-C(7)-C(8)	-155.54(17)
C(12)-C(7)-C(8)-C(9)	-2.1(2)
N(3)-C(7)-C(8)-C(9)	-179.48(15)
C(7)-C(8)-C(9)-C(10)	-1.0(3)
C(8)-C(9)-C(10)-C(11)	1.9(2)
C(8)-C(9)-C(10)-N(4)	-175.50(15)
N(5)-N(4)-C(10)-C(11)	168.51(15)
N(5)-N(4)-C(10)-C(9)	-14.0(2)

C(9)-C(10)-C(11)-C(12)	0.4(3)
N(4)-C(10)-C(11)-C(12)	177.98(15)
C(10)-C(11)-C(12)-C(7)	-3.5(3)
C(8)-C(7)-C(12)-C(11)	4.4(2)
N(3)-C(7)-C(12)-C(11)	-178.48(15)
N(4)-N(5)-C(13)-C(18)	161.11(15)
N(4)-N(5)-C(13)-C(14)	-21.8(2)
C(18)-C(13)-C(14)-C(15)	-2.4(2)
N(5)-C(13)-C(14)-C(15)	-179.37(15)
C(13)-C(14)-C(15)-C(16)	-0.3(3)
C(14)-C(15)-C(16)-C(17)	2.1(3)
C(15)-C(16)-C(17)-C(18)	-1.2(3)
C(16)-C(17)-C(18)-C(13)	-1.5(3)
C(14)-C(13)-C(18)-C(17)	3.4(3)
N(5)-C(13)-C(18)-C(17)	-179.45(15)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for gul1 [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(3)-H(3)...O(1)#1	0.90(2)	1.90(2)	2.624(2)	135.5(2)

Symmetry transformations used to generate equivalent atoms:
#1 x,y,z

