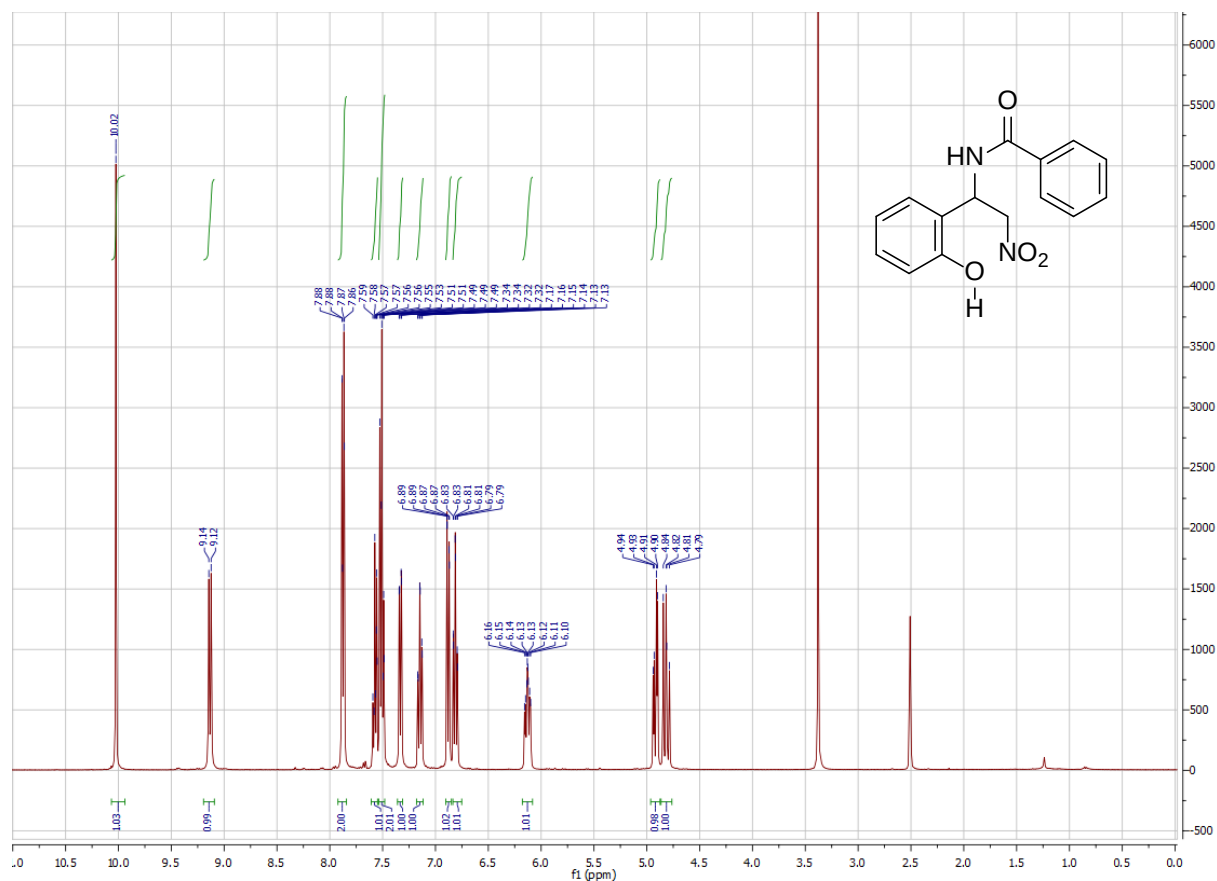
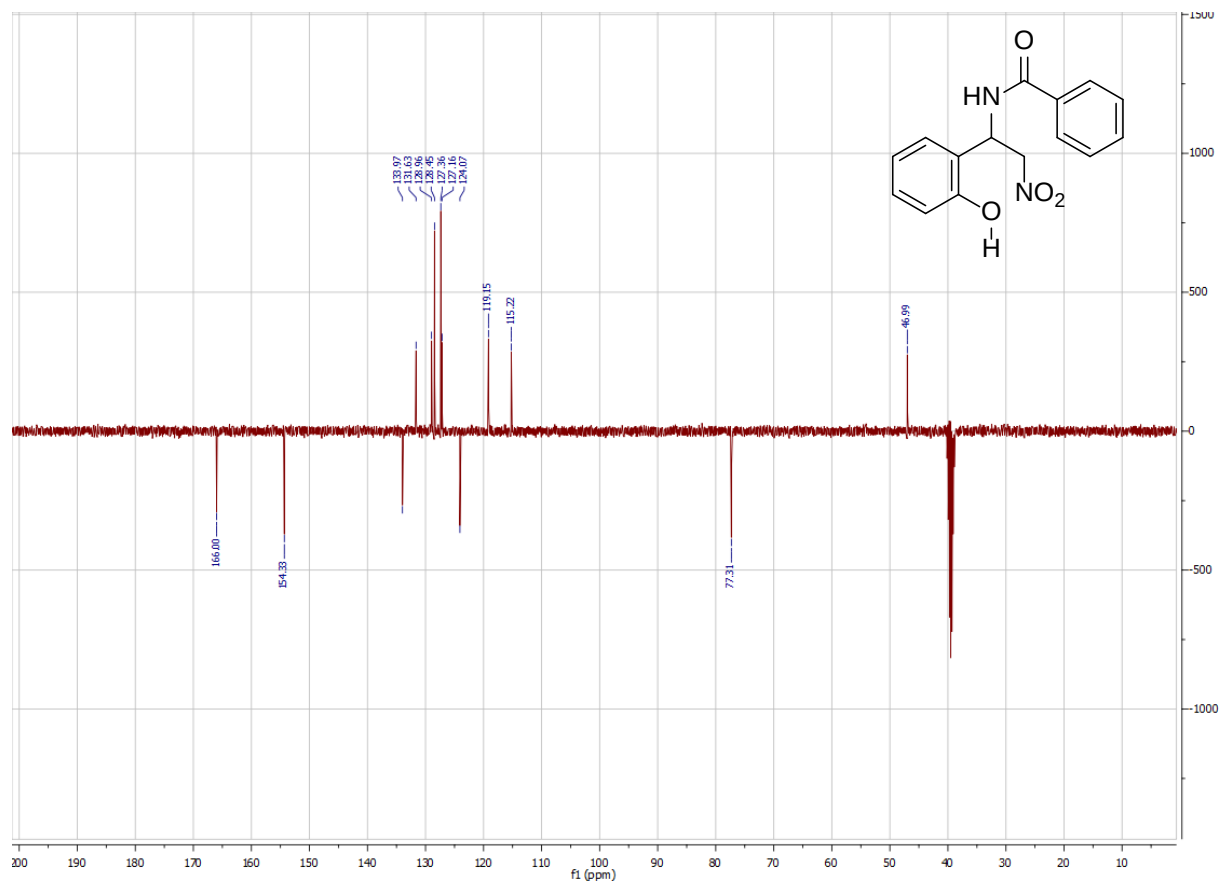


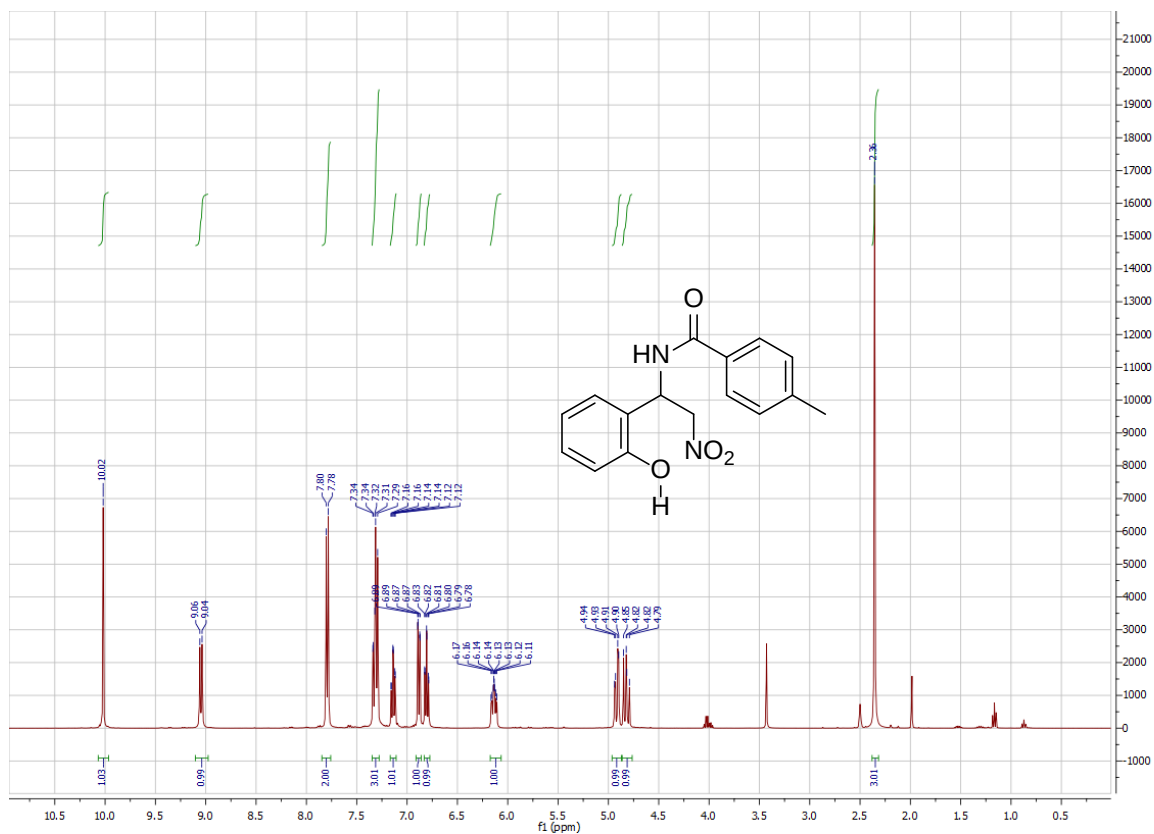
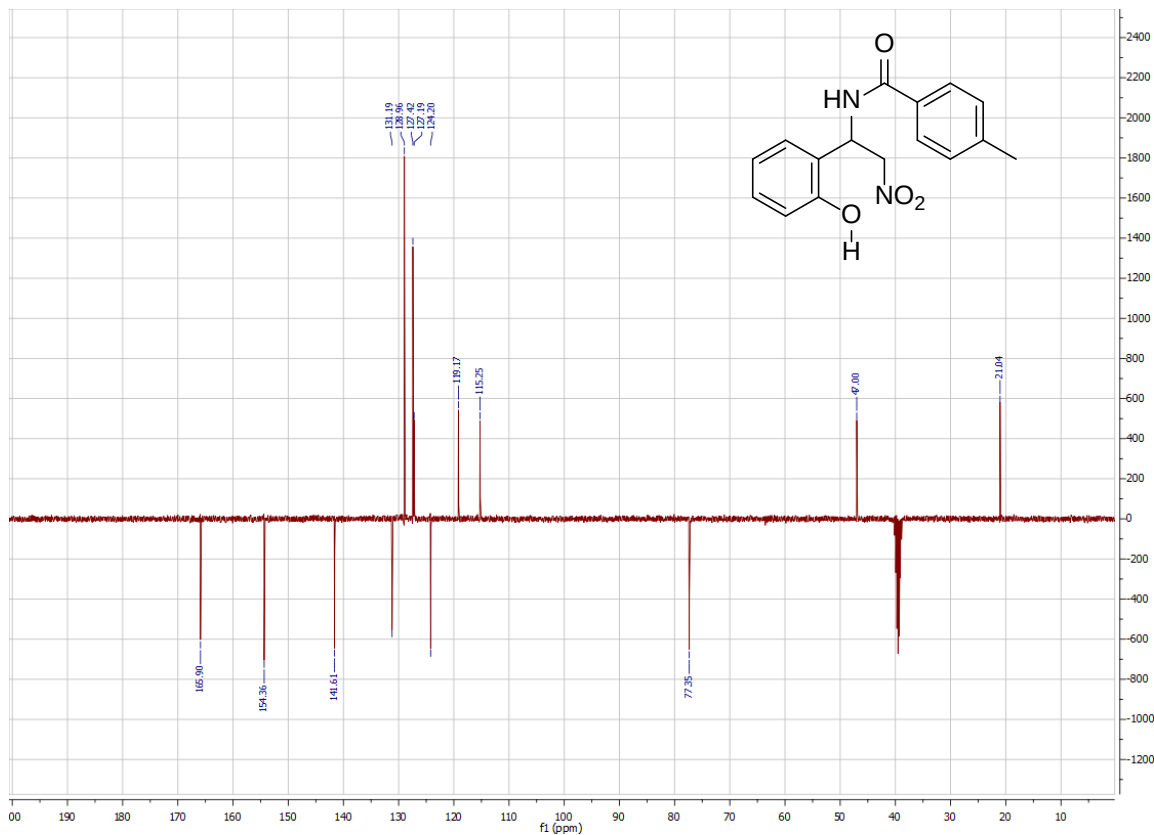
¹H NMR (400 MHz, DMSO-*d*₆) for *N*-(1-(2-hydroxyphenyl)-2-nitroethyl)benzamide (3a):



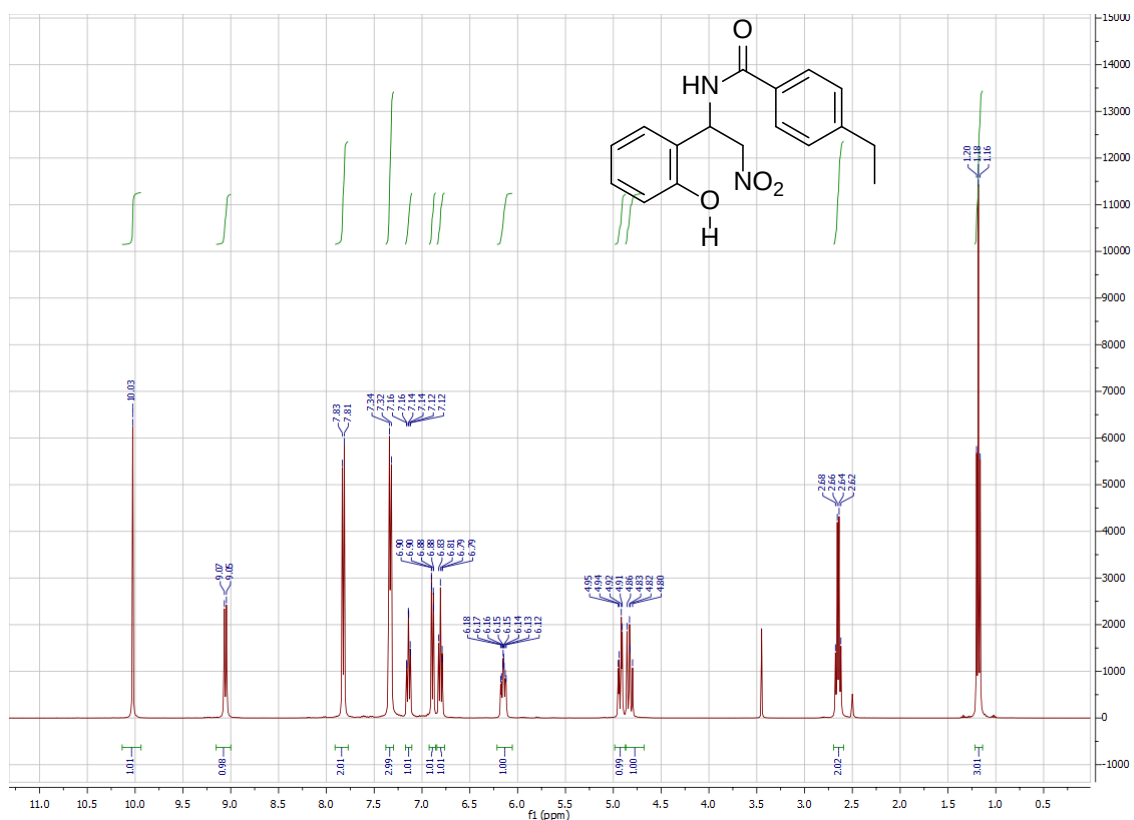
¹³C NMR (100 MHz, DMSO-*d*₆) for *N*-(1-(2-hydroxyphenyl)-2-nitroethyl)benzamide (3a):



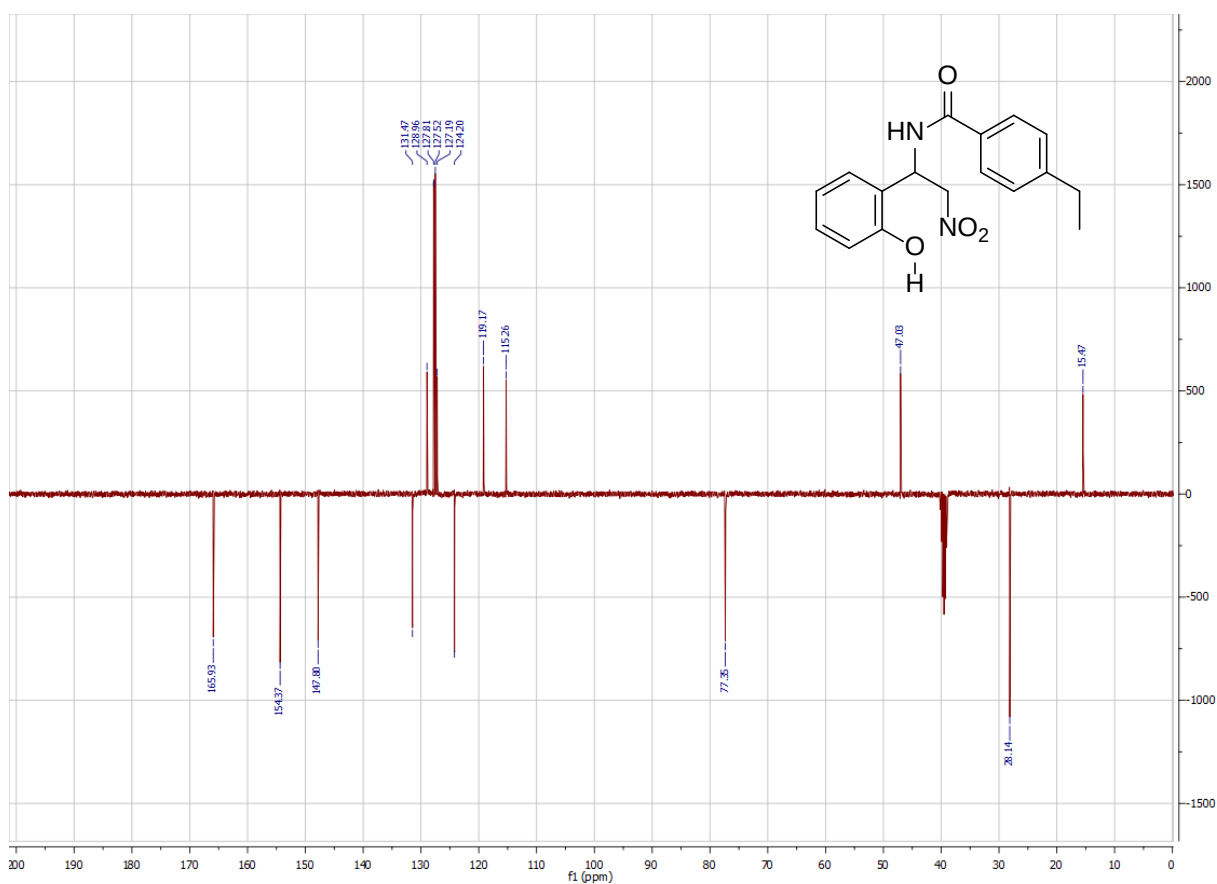
¹H NMR (400 MHz, DMSO-*d*₆) for *N*-(1-(2-hydroxyphenyl)-2-nitroethyl)-4-methylbenzamide (3b)

¹³C NMR (100 MHz, DMSO-*d*₆) for *N*-(1-(2-hydroxyphenyl)-2-nitroethyl)-4-methylbenzamide (3b)

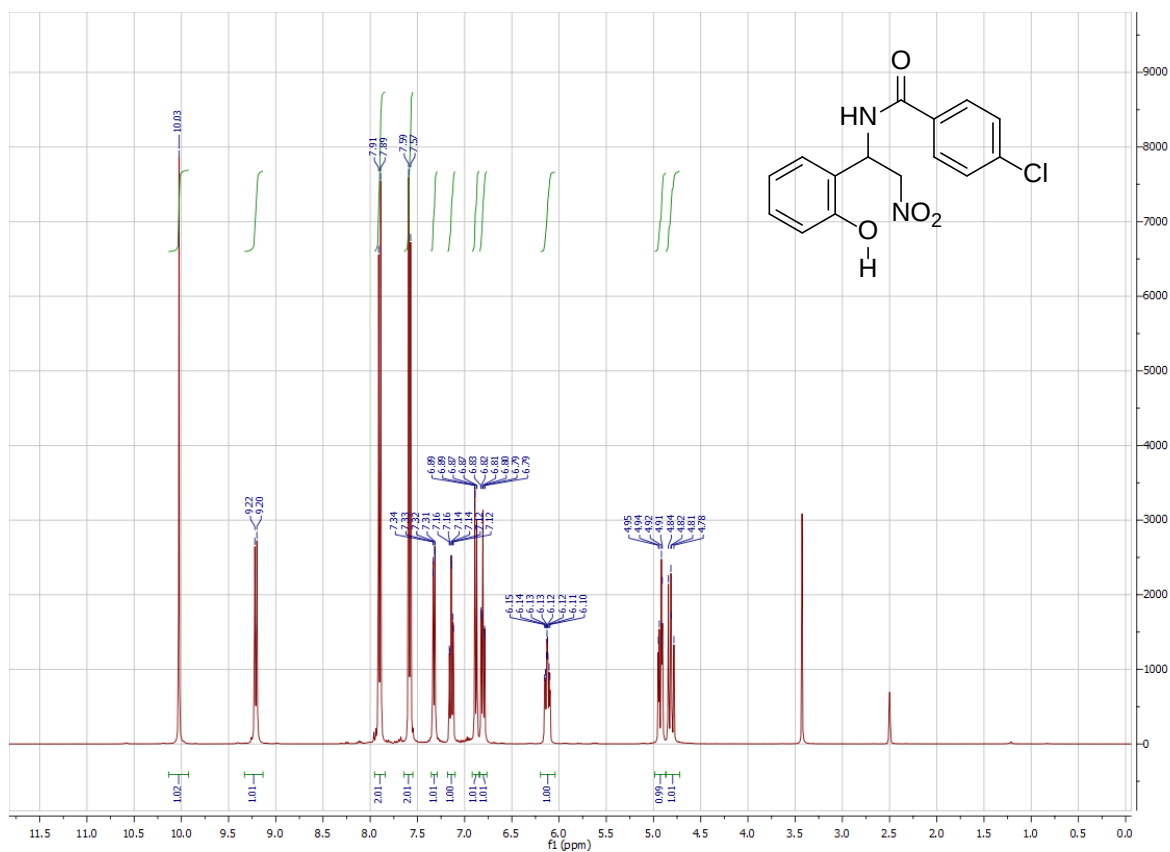
¹H NMR (400 MHz, DMSO-*d*₆) for *N*-(1-(2-hydroxyphenyl)-2-nitroethyl)-4-ethylbenzamide (3c):



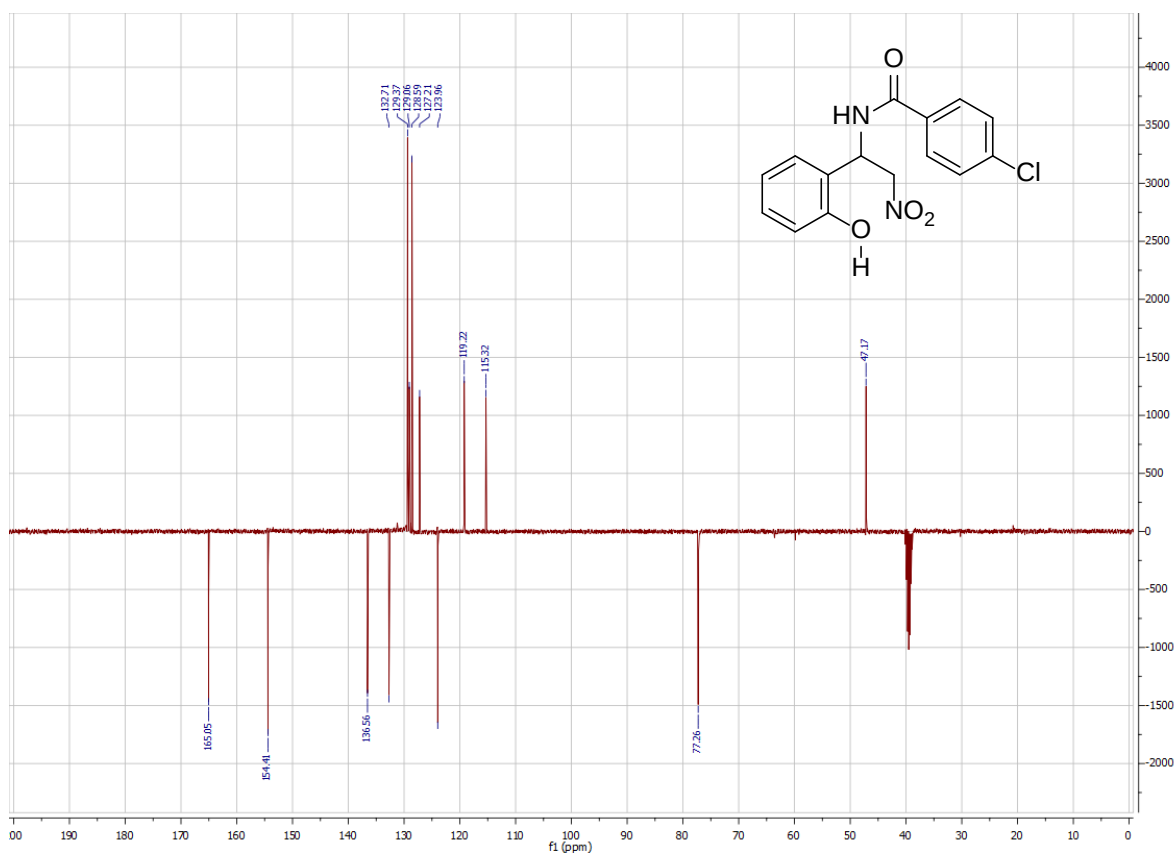
¹³C NMR (100 MHz, DMSO-*d*₆) for *N*-(1-(2-hydroxyphenyl)-2-nitroethyl)-4-ethylbenzamide (3c):



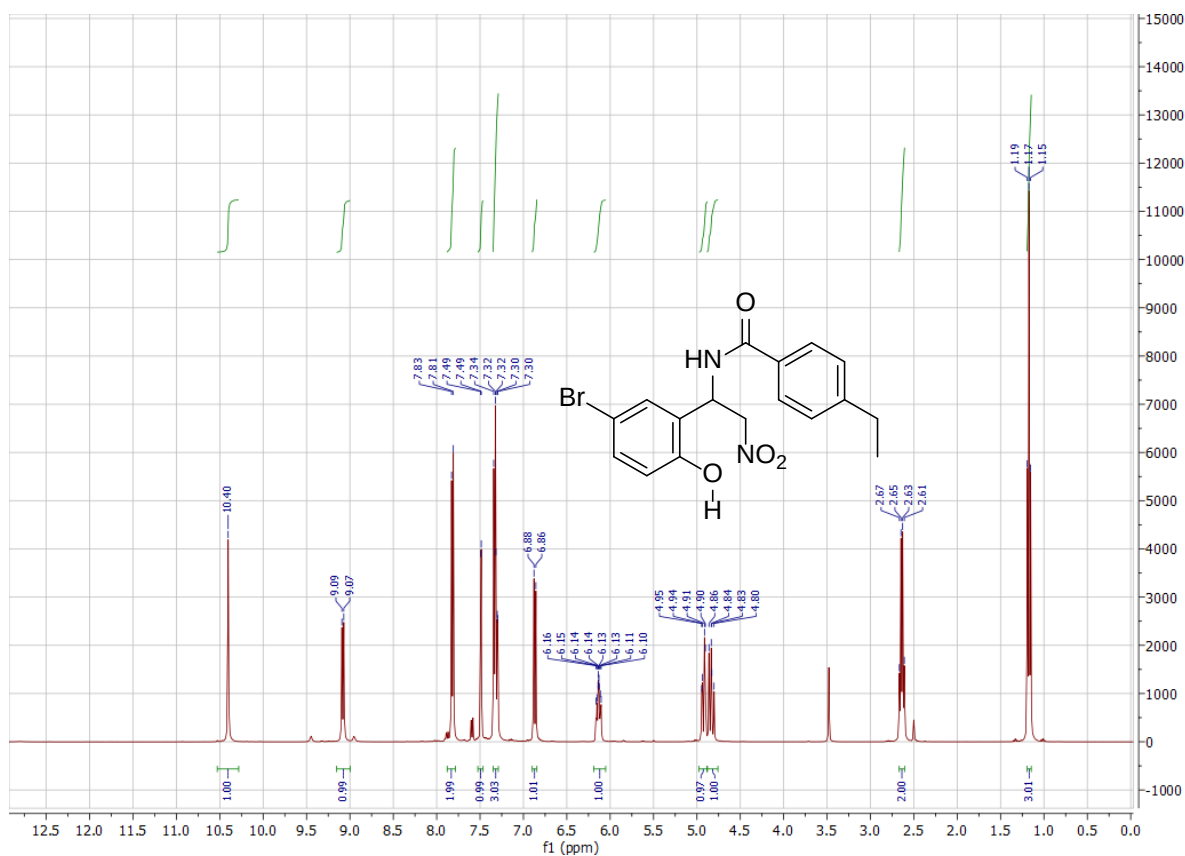
¹H NMR (400 MHz, DMSO-*d*₆) for *N*-(1-(2-hydroxyphenyl)-2-nitroethyl)-4-chlorobenzamide (3d):



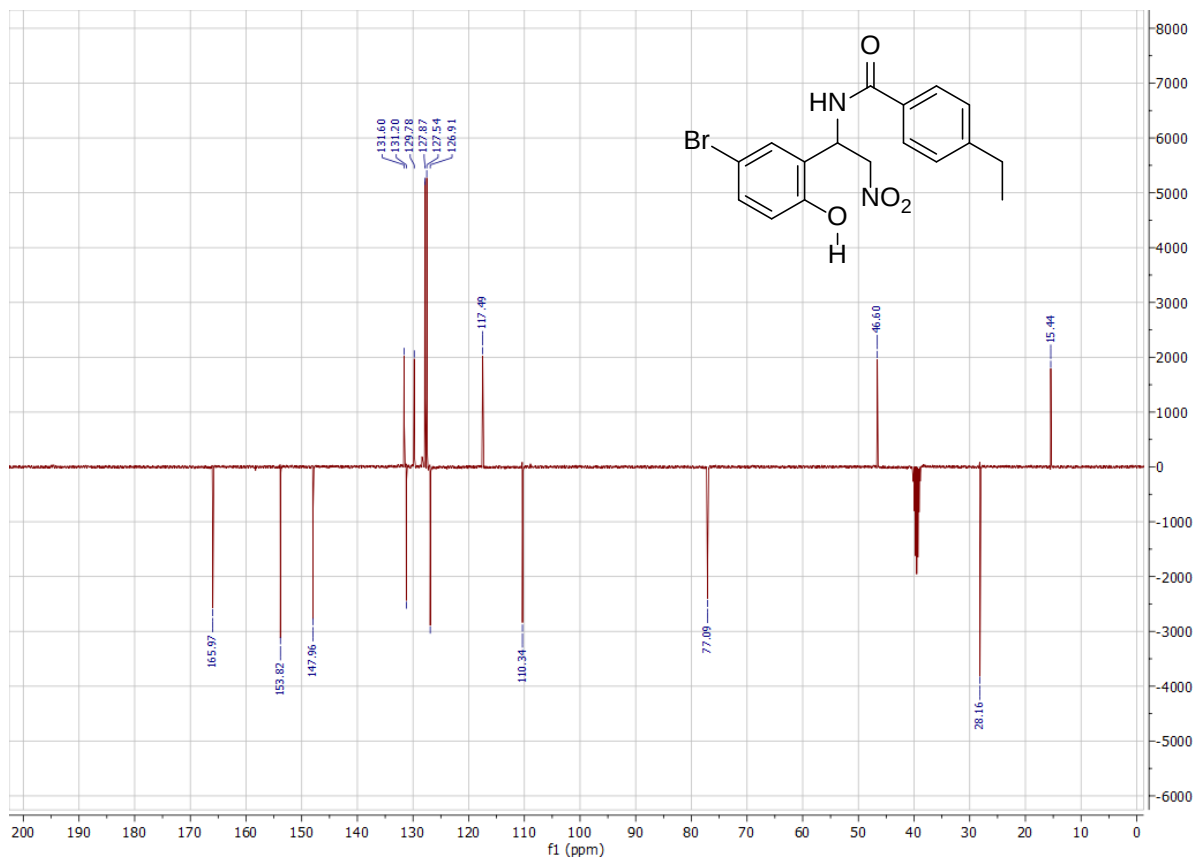
¹³C NMR (100 MHz, DMSO-*d*₆) for *N*-(1-(2-hydroxyphenyl)-2-nitroethyl)-4-chlorobenzamide (3d):



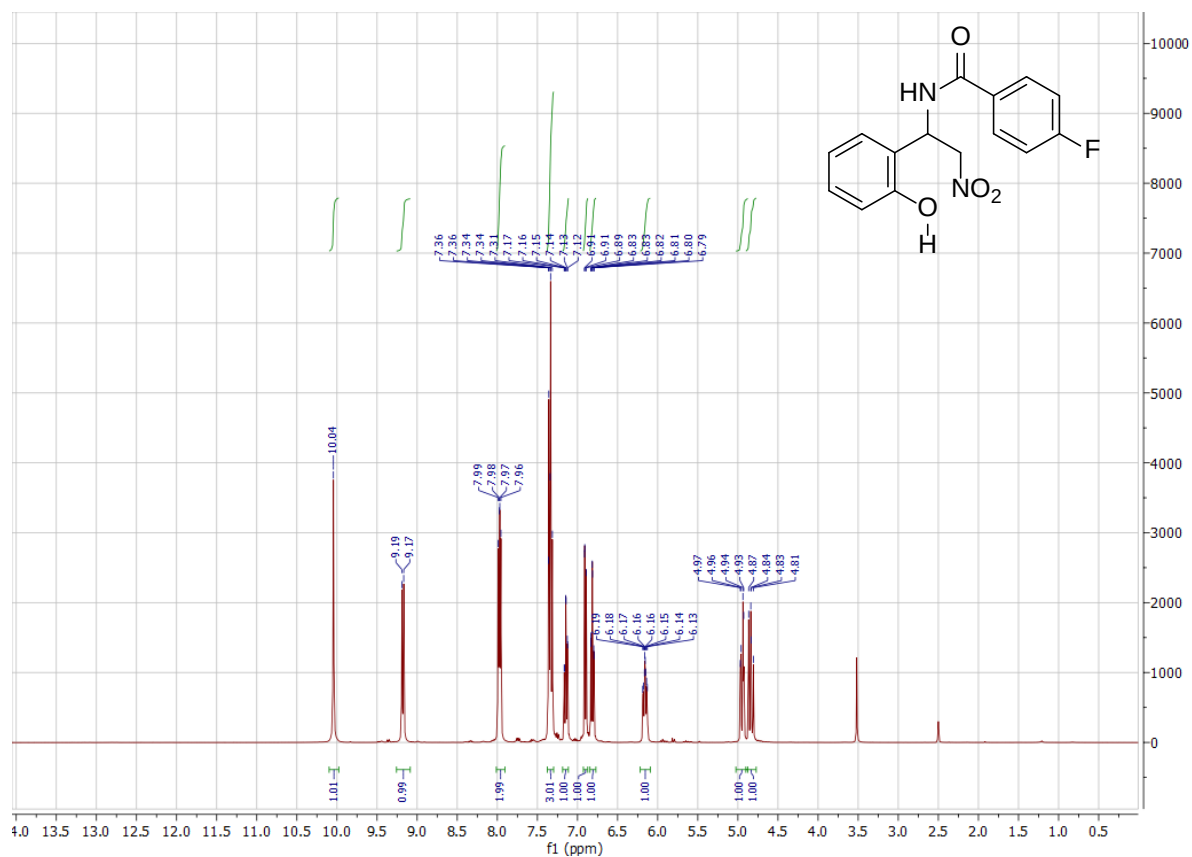
¹H NMR (400 MHz, DMSO-*d*₆) for *N*-(1-(5-bromo-2-hydroxyphenyl)-2-nitroethyl)-4-ethylbenzamide (3e):



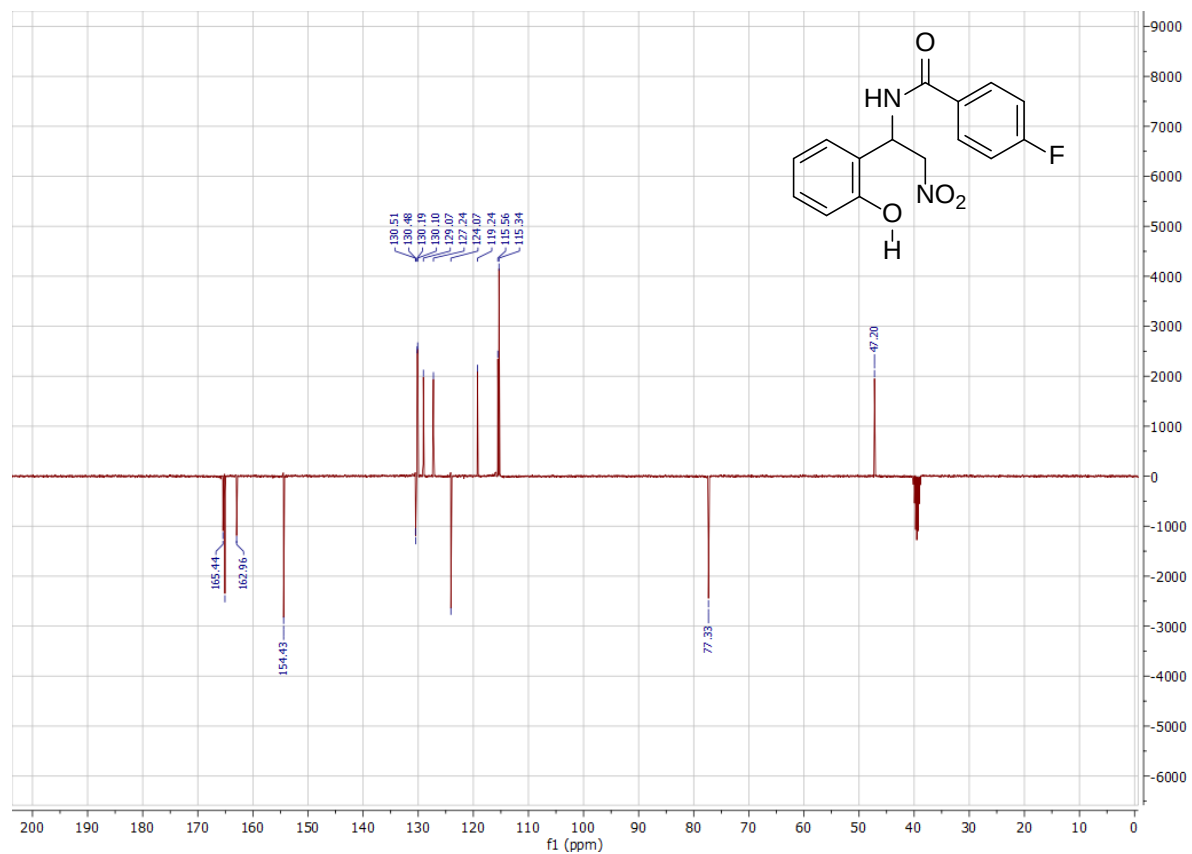
¹³C NMR (100 MHz, DMSO-*d*₆) for *N*-(1-(5-bromo-2-hydroxyphenyl)-2-nitroethyl)-4-ethylbenzamide (3e):



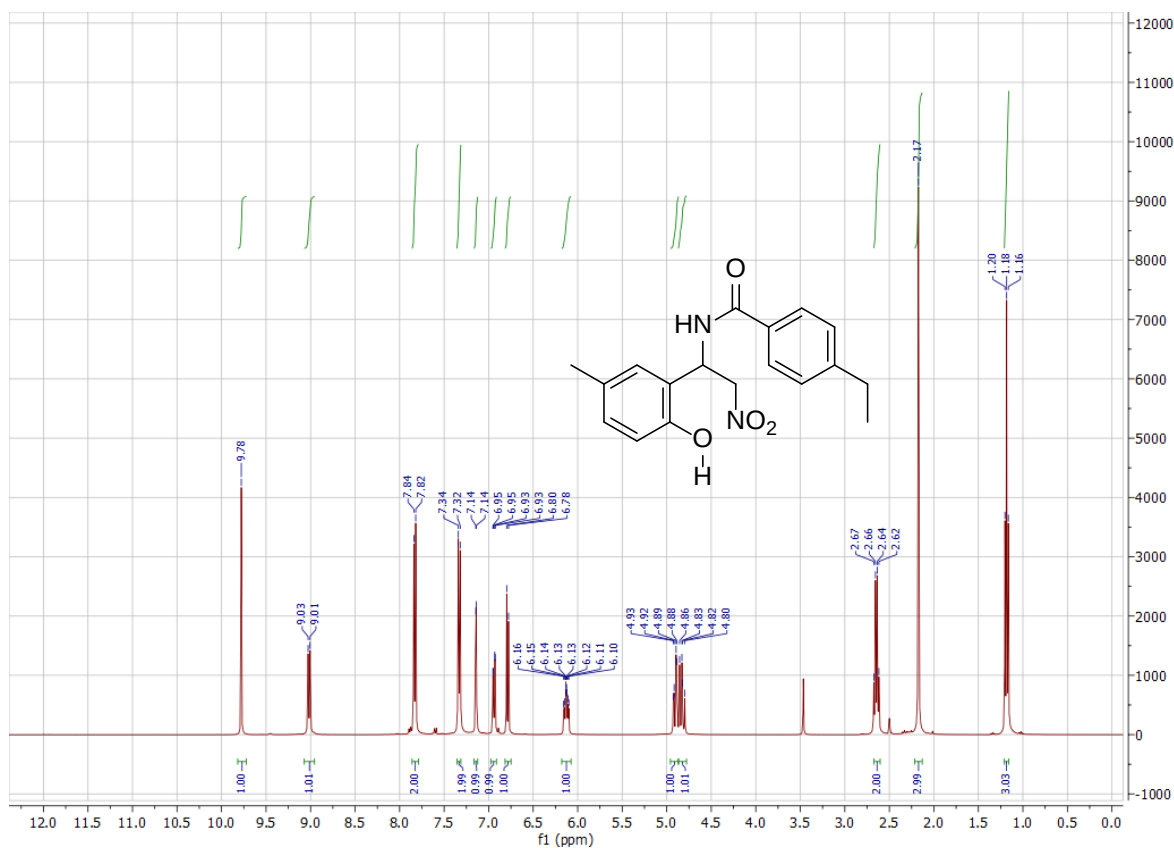
^1H NMR (400 MHz, DMSO- d_6) for *N*-(1-(2-hydroxyphenyl)-2-nitroethyl)-4-fluorobenzamide (3f):



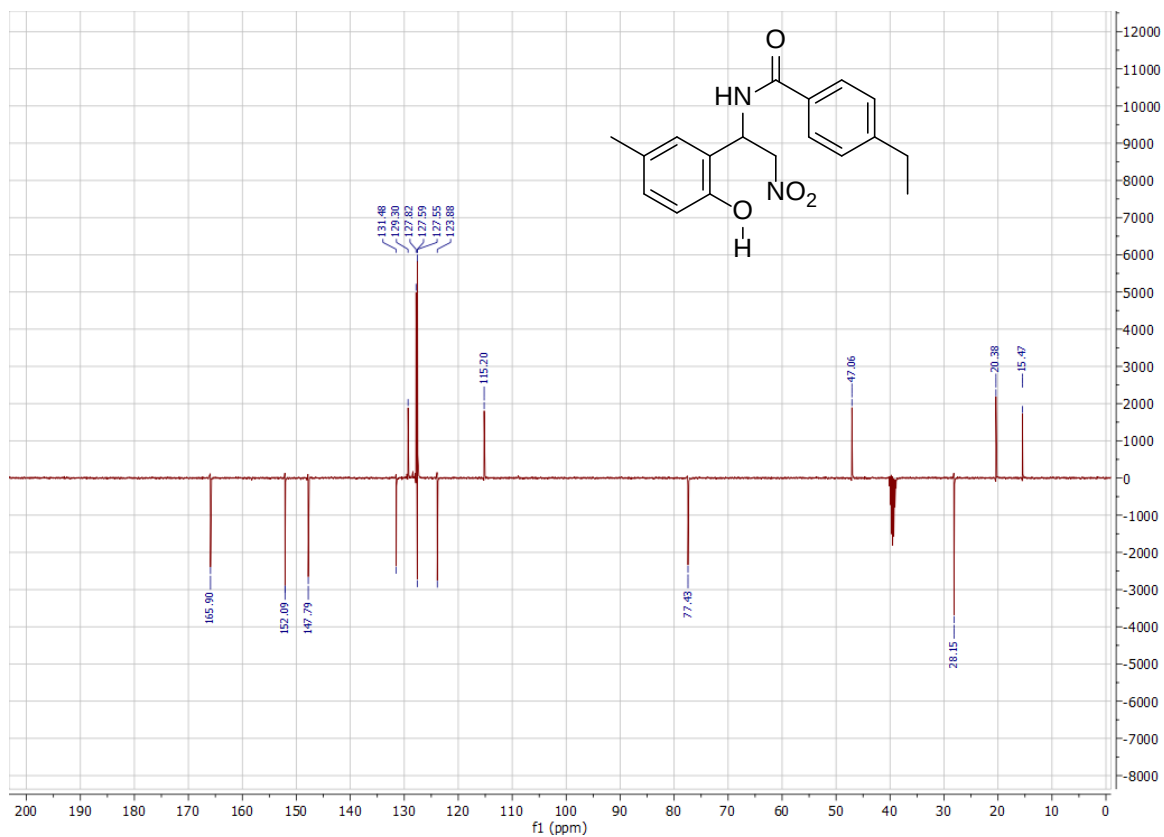
^{13}C NMR (100 MHz, DMSO- d_6) for *N*-(1-(2-hydroxyphenyl)-2-nitroethyl)-4-fluorobenzamide (3f):



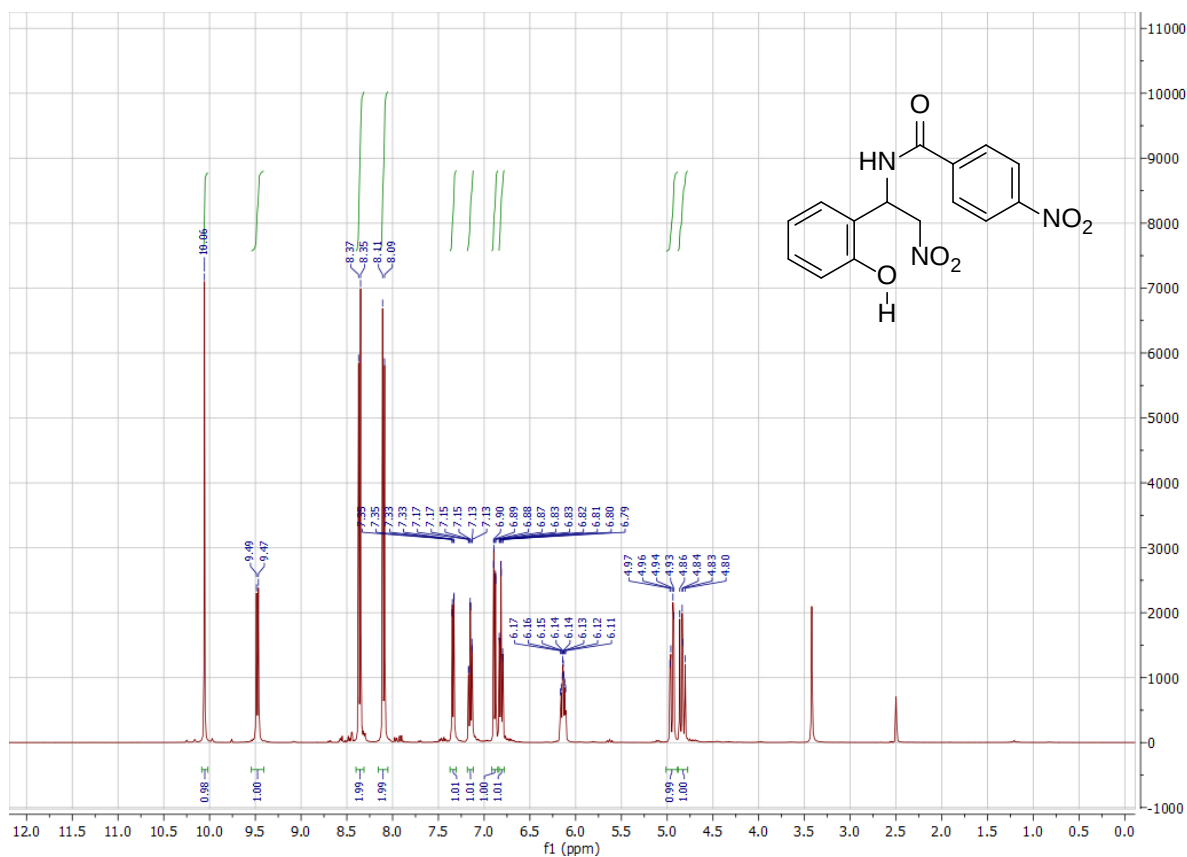
¹H NMR (400 MHz, DMSO-*d*₆) for *N*-(1-(2-hydroxy-5-methylphenyl)-2-nitroethyl)-4-ethylbenzamide (3g):



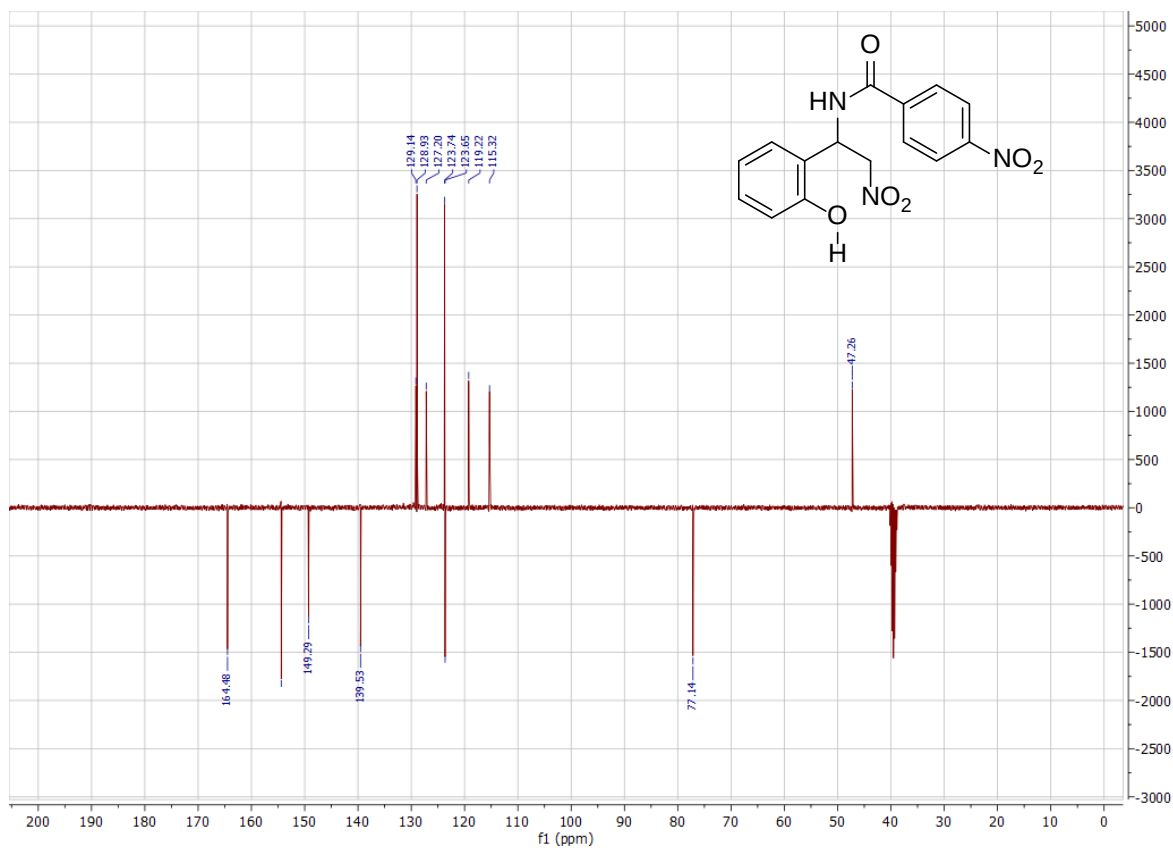
¹³C NMR (100 MHz, DMSO-*d*₆) for *N*-(1-(2-hydroxy-5-methylphenyl)-2-nitroethyl)-4-ethylbenzamide (3g):



¹H NMR (400 MHz, DMSO-*d*₆) for *N*-(1-(2-hydroxyphenyl)-2-nitroethyl)-4-nitrobenzamide (3h):



¹³C NMR (100 MHz, DMSO-*d*₆) for *N*-(1-(2-hydroxyphenyl)-2-nitroethyl)-4-nitrobenzamide (3h):



X-Ray Crystallography data for *N*-(1-(2-hydroxyphenyl)-2-nitroethyl)benzamide (3a)

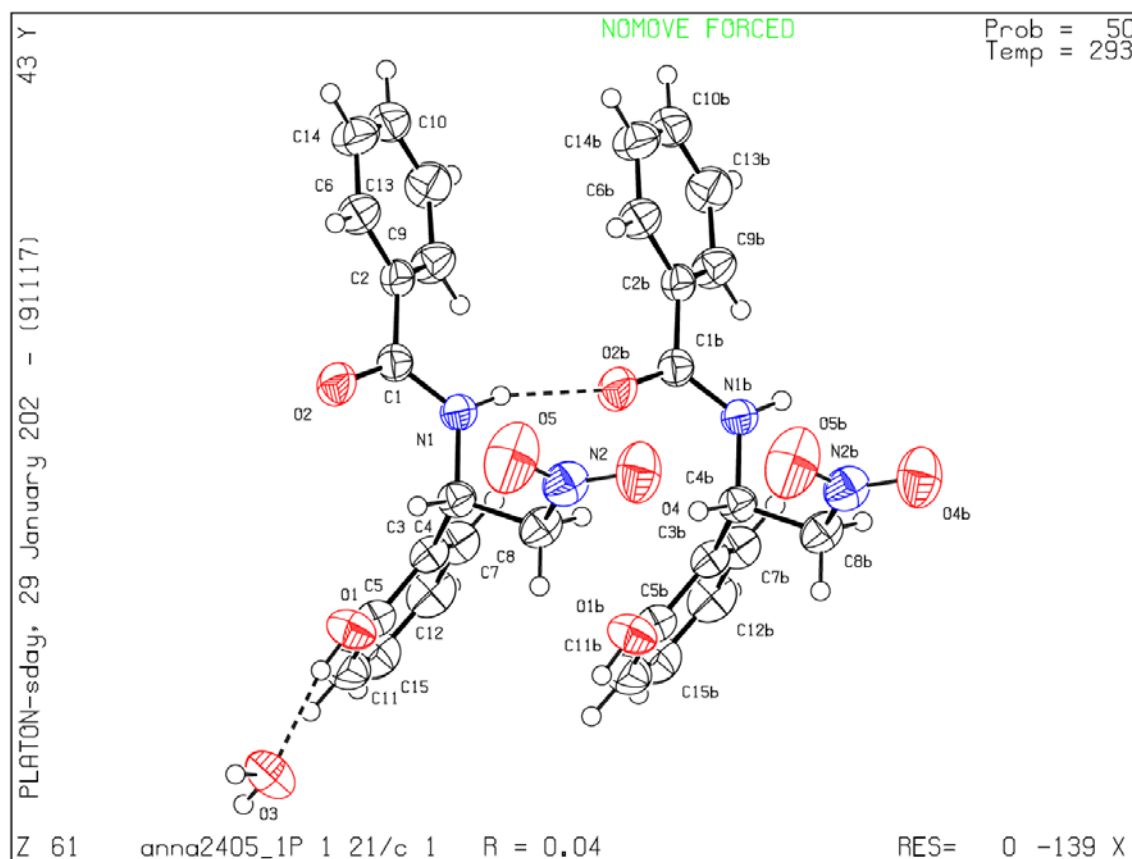
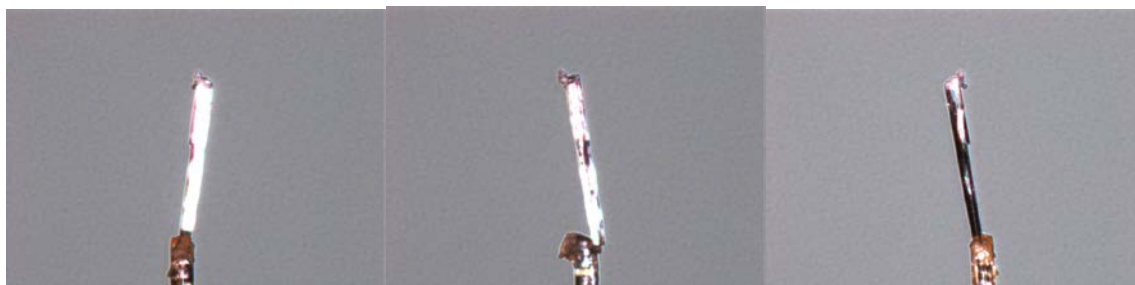


Table S1 Crystal data and structure refinement for 3a.

Identification code	ANNA2405
Empirical formula	C ₁₅ H ₁₆ N ₂ O ₅
Formula weight	304.30
Temperature/K	293(2)
Crystal system	monoclinic
Space group	P2 ₁ /c
a/Å	15.7555(3)
b/Å	5.18360(10)
c/Å	17.7967(3)
α/°	90
β/°	91.081(2)
γ/°	90
Volume/Å ³	1453.20(5)
Z	4

$\rho_{\text{calc}}/\text{g}/\text{cm}^3$	1.391
μ/mm^{-1}	0.888
F(000)	640.0
Crystal size/ mm^3	$0.53 \times 0.04 \times 0.03$
Radiation	Cu K α ($\lambda = 1.54184$)
2 Θ range for data collection/ $^\circ$	5.61 to 152.346
Index ranges	$-19 \leq h \leq 19, -6 \leq k \leq 4, -22 \leq l \leq 22$
Reflections collected	12791
Independent reflections	3040 [$R_{\text{int}} = 0.0301, R_{\text{sigma}} = 0.0199$]
Data/restraints/parameters	3040/2/234
Goodness-of-fit on F^2	1.035
Final R indexes [$I \geq 2\sigma(I)$]	$R_1 = 0.0441, wR_2 = 0.1221$
Final R indexes [all data]	$R_1 = 0.0501, wR_2 = 0.1293$
Largest diff. peak/hole / $\text{e } \text{\AA}^{-3}$	0.22/-0.15

Table S2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for 3a. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij} tensor.

Atom	x	y	z	U(eq)
O1	8736.7(7)	7188(2)	6501.6(6)	52.5(3)
O2	7126.4(7)	7904.4(19)	4754.6(7)	53.0(3)
N1	7338.1(7)	3625(2)	4910.3(6)	39.0(3)
O3	9591.5(8)	9733(3)	7626.9(8)	64.2(3)
N2	9111.6(8)	2593(3)	4445.3(8)	53.1(3)
C1	6896.3(8)	5665(2)	4653.1(7)	37.5(3)
C2	6082.3(8)	5083(2)	4235.2(7)	36.7(3)
O4	9395(6)	793(9)	4089(3)	74.3(16)
C3	7851.8(8)	3646(3)	6235.1(7)	39.0(3)
C4	8072.0(8)	3967(2)	5413.4(7)	37.9(3)
C5	8199.9(8)	5348(3)	6761.8(7)	42.1(3)
O5	9055(13)	4768(14)	4190(6)	95(3)
C6	5797.8(9)	6792(3)	3686.8(8)	46.8(3)
C7	7320.4(9)	1683(3)	6478.6(9)	48.7(3)
C8	8774.3(9)	2097(3)	5216.0(8)	46.1(3)
C9	5579.4(10)	2981(3)	4414.3(9)	49.6(3)
C10	4518.7(10)	4333(3)	3517.6(9)	52.8(4)
C11	7996.6(11)	5134(3)	7516.4(8)	54.7(4)
C12	7127.7(10)	1442(3)	7230.2(9)	56.2(4)
C13	4797.8(10)	2631(3)	4055.8(10)	56.4(4)
C14	5020.1(11)	6397(3)	3327.5(9)	56.1(4)
C15	7459.3(11)	3175(4)	7742.9(9)	58.6(4)
O6	9313(13)	4760(20)	4280(11)	75(3)
O7	9200(20)	770(30)	4062(7)	108(5)

Table S3 Anisotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for 3a. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\dots]$.

Atom	U ₁₁	U ₂₂	U ₃₃	U ₂₃	U ₁₃	U ₁₂
O1	57.4(6)	56.2(6)	43.7(5)	0.0(5)	-5.6(4)	-15.9(5)
O2	53.0(6)	33.3(5)	72.4(7)	-1.2(4)	-11.6(5)	-0.1(4)
N1	41.0(6)	32.7(5)	42.9(6)	0.1(4)	-6.0(4)	-0.6(4)
O3	63.6(7)	74.0(8)	55.1(7)	-19.2(6)	4.8(5)	-13.5(6)
N2	44.3(7)	63.5(8)	51.9(7)	4.6(6)	7.9(5)	3.8(6)
C1	39.6(6)	36.0(6)	36.9(6)	-0.8(5)	2.0(5)	1.7(5)
C2	36.9(6)	36.9(6)	36.5(5)	-2.7(5)	3.1(4)	4.0(4)
O4	75(3)	72(3)	77(2)	-4(2)	28.9(17)	15(2)
C3	33.6(6)	41.7(6)	41.6(6)	4.7(5)	0.3(5)	3.2(5)
C4	36.6(6)	36.9(6)	40.1(6)	2.5(5)	-1.8(5)	-2.3(5)
C5	38.9(6)	45.3(7)	42.0(6)	4.4(5)	-1.9(5)	2.5(5)
O5	121(7)	90(3)	77(3)	33(2)	51(4)	28(3)
C6	48.0(8)	46.1(7)	46.3(7)	6.7(6)	0.1(6)	0.7(6)
C7	44.2(7)	49.0(8)	52.9(8)	5.9(6)	2.1(6)	-3.6(6)
C8	40.3(7)	51.0(8)	47.3(7)	8.7(6)	4.5(5)	3.8(5)
C9	45.4(8)	46.2(7)	57.1(8)	10.5(6)	-4.2(6)	-1.4(6)
C10	41.9(7)	60.1(9)	56.1(8)	-11.3(7)	-6.3(6)	5.8(6)
C11	60.3(9)	63.3(9)	40.5(7)	0.1(6)	-1.2(6)	4.5(7)
C12	49.7(8)	60.1(9)	59.2(9)	17.3(7)	10.8(7)	-1.0(7)
C13	44.0(8)	52.6(8)	72.6(10)	1.4(7)	-1.9(7)	-6.8(6)
C14	57.6(9)	62.3(9)	48.1(8)	7.7(7)	-9.9(6)	8.8(7)
C15	59.8(9)	72.7(10)	43.8(7)	14.0(7)	12.0(6)	11.0(8)
O6	80(6)	54(4)	94(6)	17(3)	41(4)	-2(3)
O7	108(10)	141(10)	74(4)	-60(7)	22(4)	-29(6)

Table S4 Bond Lengths for 3a.

Atom	Atom	Length/ $\text{\AA}$	Atom	Atom	Length/ $\text{\AA}$
O1	C5	1.3617(17)	C3	C4	1.5185(17)
O2	C1	1.2286(16)	C3	C5	1.3922(19)
N1	C1	1.3414(17)	C3	C7	1.3921(19)
N1	C4	1.4598(16)	C4	C8	1.5173(18)
N2	O4	1.217(5)	C5	C11	1.391(2)
N2	O5	1.218(7)	C6	C14	1.387(2)
N2	C8	1.5024(19)	C7	C12	1.383(2)
N2	O6	1.206(10)	C9	C13	1.388(2)
N2	O7	1.176(10)	C10	C13	1.369(2)
C1	C2	1.5009(18)	C10	C14	1.376(2)
C2	C6	1.3862(19)	C11	C15	1.387(2)
C2	C9	1.3881(19)	C12	C15	1.376(3)

Table 5 Bond Angles for 3a.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C1	N1	C4	120.91(11)	N1	C4	C3	112.62(10)
O4	N2	O5	122.7(5)	N1	C4	C8	110.68(11)
O4	N2	C8	118.9(3)	C8	C4	C3	109.51(10)
O5	N2	C8	118.3(4)	O1	C5	C3	117.01(12)
O6	N2	C8	118.8(8)	O1	C5	C11	122.70(14)
O7	N2	C8	115.9(8)	C11	C5	C3	120.29(13)
O7	N2	O6	125.2(12)	C2	C6	C14	120.10(14)
O2	C1	N1	123.00(12)	C12	C7	C3	120.75(15)
O2	C1	C2	120.63(12)	N2	C8	C4	111.97(11)
N1	C1	C2	116.36(11)	C13	C9	C2	120.14(14)
C6	C2	C1	118.86(12)	C13	C10	C14	119.65(14)
C6	C2	C9	119.01(13)	C15	C11	C5	119.49(15)
C9	C2	C1	122.00(12)	C15	C12	C7	119.66(15)
C5	C3	C4	118.99(11)	C10	C13	C9	120.55(15)
C7	C3	C4	121.97(12)	C10	C14	C6	120.53(14)
C7	C3	C5	119.03(12)	C12	C15	C11	120.75(14)

Table S6 Hydrogen Bonds for 3a.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
O1	H1	O3	0.83(2)	1.92(3)	2.7320(16)	167(2)

Table S7 Torsion Angles for 3a.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
O1	C5	C11	C15	178.60(14)	C4	C3	C5	C11	178.72(13)
O2	C1	C2	C6	29.56(18)	C4	C3	C7	C12	179.74(13)
O2	C1	C2	C9	146.21(14)	C5	C3	C4	N1	138.09(12)
N1	C1	C2	C6	151.28(12)	C5	C3	C4	C8	98.30(14)
N1	C1	C2	C9	32.95(18)	C5	C3	C7	C12	1.0(2)
N1	C4	C8	N2	63.91(15)	C5	C11	C15	C12	0.1(3)
C1	N1	C4	C3	96.59(14)	O5	N2	C8	C4	26.5(12)
C1	N1	C4	C8	140.46(12)	C6	C2	C9	C13	-1.1(2)
C1	C2	C6	C14	175.63(13)	C7	C3	C4	N1	42.69(17)
C1	C2	C9	C13	174.69(14)	C7	C3	C4	C8	-80.91(16)
C2	C6	C14	C10	0.9(2)	C7	C3	C5	O1	178.02(12)
C2	C9	C13	C10	0.7(3)	C7	C3	C5	C11	-2.0(2)
O4	N2	C8	C4	-150.3(6)	C7	C12	C15	C11	-1.1(3)
C3	C4	C8	N2	-	C9	C2	C6	C14	0.3(2)

Table S7 Torsion Angles for 3a.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
				171.35(12)					
C3	C5	C11	C15	1.5(2)	C13	C10	C14	C6	-1.2(2)
C3	C7	C12	C15	0.5(2)	C14	C10	C13	C9	0.4(3)
C4	N1	C1	O2	7.9(2)	O6	N2	C8	C4	49.9(12)
C4	N1	C1	C2	-	O7	N2	C8	C4	-133.7(17)
C4	C3	C5	O1	171.21(10)					-1.22(18)

Table S8 Hydrogen Atom Coordinates ($\text{\AA} \times 10^4$) and Isotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for 3a.

Atom	x	y	z	U(eq)
H4	8287.63	5722.85	5344.48	45
H6	6129.09	8205.31	3559.54	56
H7	7092.41	520.15	6131.83	58
H8A	9233.88	2251.97	5583.59	55
H8B	8557.95	347.24	5238.97	55
H9	5766.65	1803.11	4775.16	60
H10	3992.81	4095.55	3281.54	63
H11	8219.27	6294.92	7866.23	66
H12	6775.87	116.53	7388.41	67
H13	4460.97	1229.69	4182.47	68
H14	4835.94	7535.93	2955.08	67
H15	7321.51	3030.62	8246.98	70
H1A	7176(10)	2040(30)	4821(9)	40(4)
H1	8920(15)	8050(40)	6863(14)	74(6)
H3A	9790(20)	11390(60)	7485(17)	111(9)
H3B	9480(20)	9990(60)	8070(20)	113(10)

Table S9 Atomic Occupancy for 3a.

Atom	Occupancy	Atom	Occupancy	Atom	Occupancy
O4	0.66(4)	O5	0.66(4)	O6	0.34(4)
O7	0.34(4)				

Experimental

Single crystals of $\text{C}_{15}\text{H}_{16}\text{N}_2\text{O}_5$ **3a** were crystallized from solution in CHCl_3 , Hexane, EtOAc, EtOH, i PrOH. A suitable crystal was selected and mounted on acrylic glue on the glass stick on a SuperNova, Dual, Cu at home/near, AtlasS2 diffractometer. The crystal was kept at 293(2) K during data collection. Using Olex2,¹ the structure was solved with the SHELXT² structure solution program using Intrinsic Phasing and refined with the SHELXL³ refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J., Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Sheldrick, G.M. (2015). Acta Cryst. A71, 3-8.
3. Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

Crystal structure determination of 3a

Crystal Data for $C_{15}H_{16}N_2O_5$ ($M = 304.30$ g/mol): monoclinic, space group $P2_1/c$ (no. 14), $a = 15.7555(3)$ Å, $b = 5.18360(10)$ Å, $c = 17.7967(3)$ Å, $\beta = 91.081(2)^\circ$, $V = 1453.20(5)$ Å³, $Z = 4$, $T = 293(2)$ K, $\mu(\text{Cu K}\alpha) = 0.888$ mm⁻¹, $D_{\text{calc}} = 1.391$ g/cm³, 12791 reflections measured ($5.61^\circ \leq 2\theta \leq 152.346^\circ$), 3040 unique ($R_{\text{int}} = 0.0301$, $R_{\text{sigma}} = 0.0199$) which were used in all calculations. The final R_1 was 0.0441 ($I > 2\sigma(I)$) and wR_2 was 0.1293 (all data).

Refinement model description

Number of restraints - 2, number of constraints - unknown.

Details:

1. Fixed Uiso

At 1.2 times of:

All C(H) groups, All C(H,H) groups

2. Restrained distances

N2-O4 $\approx$ N2-O7

with sigma of 0.02

N2-O5 $\approx$ N2-O6

with sigma of 0.02

3. Others

Sof(O6)=Sof(O7)=1-FVAR(1)

Sof(O4)=Sof(O5)=FVAR(1)

4.a Ternary CH refined with riding coordinates:

C4(H4)

4.b Secondary CH2 refined with riding coordinates:

C8(H8A,H8B)

4.c Aromatic/amide H refined with riding coordinates:

C6(H6), C7(H7), C9(H9), C10(H10), C11(H11), C12(H12), C13(H13), C14(H14),
C15(H15)

This report has been created with Olex2, compiled on 2025.03.04 svn.red2d20d0 for OlexSys. Please [let us know](#) if there are any errors or if you would like to have additional features.