Electronic Supplementary Material (ESI)

Synthesis of 2-carboxyaniline-substituted maleimides from 2'-nitrochalcones
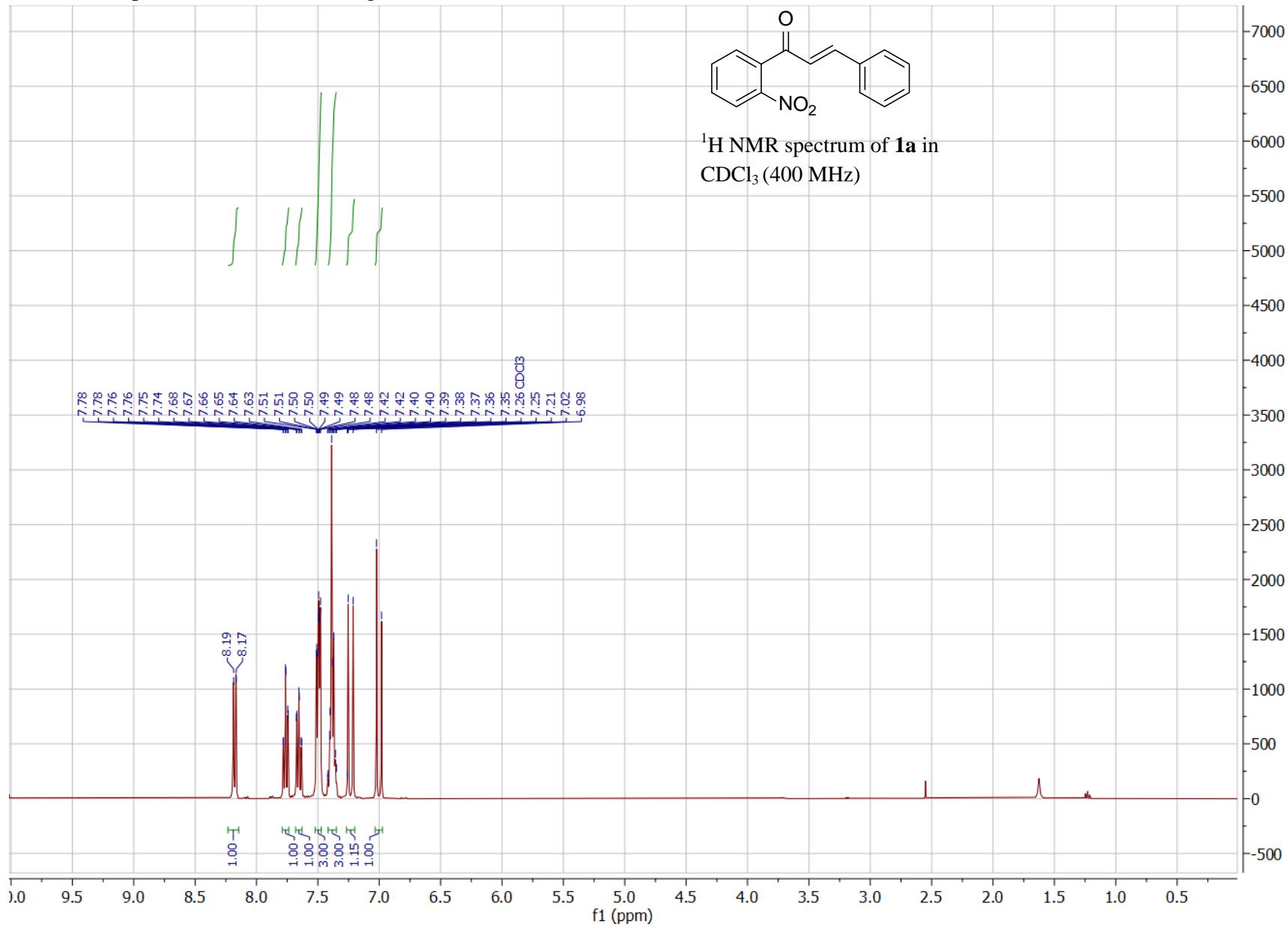
Nicolai A. Aksenov,* Dmitrii A. Aksenov, Daniil D. Ganusenko, Igor A. Kurenkov, Alexander V. Leontiev and Alexander V. Aksenov

Aksenov
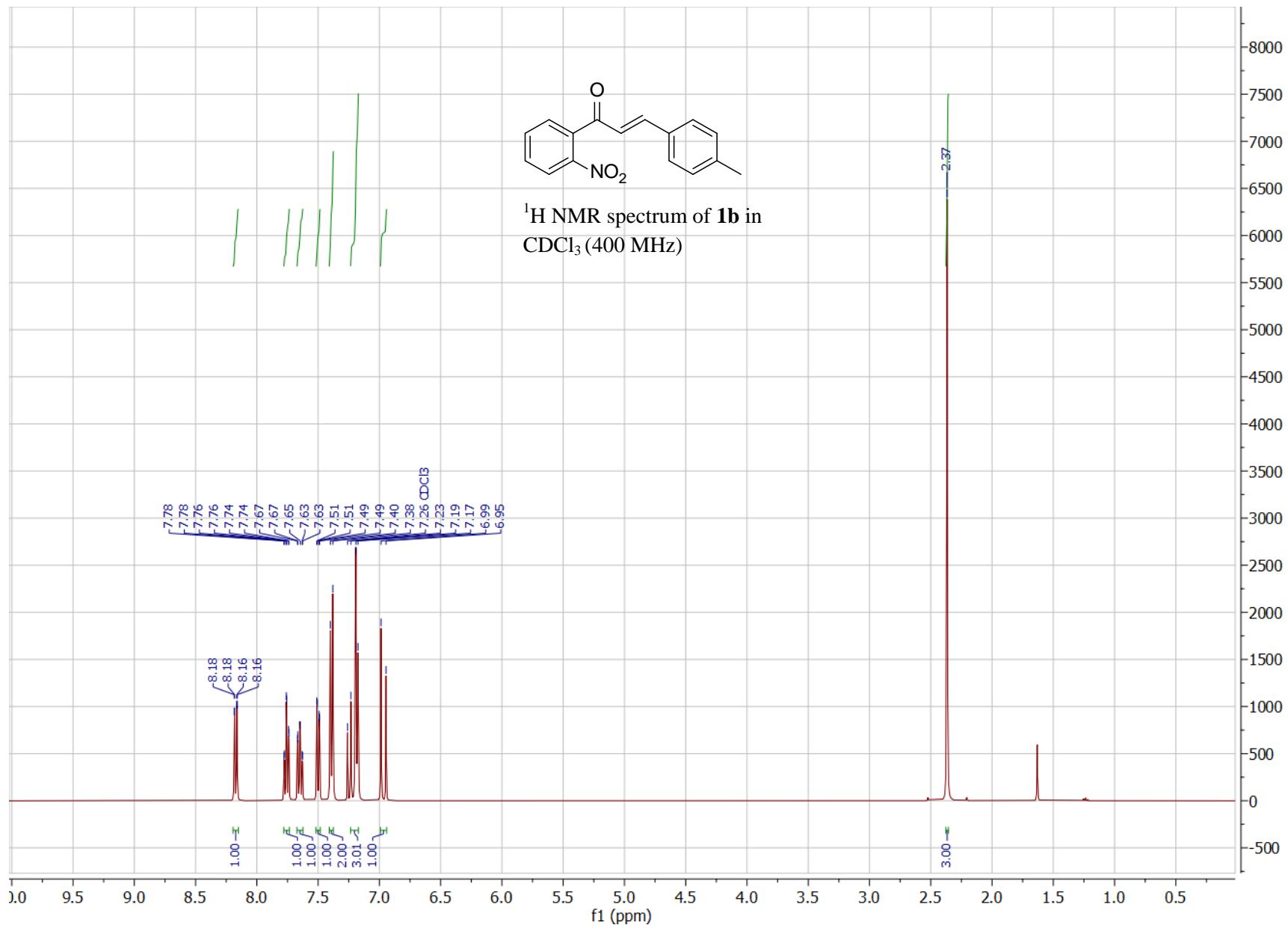
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Supporting Information

^{1}H and ^{13}C Spectral Charts for starting chalcones (1)

^{1}H NMR spectrum of **1a** in CDCl$_3$ (400 MHz)

7.78, 7.78, 7.76, 7.76, 7.75, 7.74, 7.68, 7.67, 7.66, 7.65, 7.64, 7.63, 7.51, 7.51, 7.50, 7.50, 7.49, 7.49, 7.48, 7.48, 7.42, 7.42, 7.40, 7.40, 7.39, 7.38, 7.37, 7.36, 7.35, 7.26 CDCl3, 7.25, 7.21, 7.02, 6.98

8.19, 8.17

1.00, 1.00, 1.00, 3.00, 3.00, 1.15, 1.00

f1 (ppm)

^{1}H NMR spectrum of **1b** in CDCl$_3$ (400 MHz)

^{1}H NMR spectrum of **1c** in CDCl$_3$ (400 MHz)

8.17, 8.15

7.79, 7.77, 7.75, 7.68, 7.66, 7.64, 7.60, 7.59, 7.57, 7.56, 7.54, 7.53, 7.31, 7.29, 7.28, 7.26 CDCl3, 7.23, 7.22, 7.19, 7.17, 6.93, 6.89

2.29

^{1}H NMR spectrum of **1d** in CDCl$_3$ (400 MHz)

8.18, 8.18, 8.16, 8.16

7.78, 7.77, 7.76, 7.76, 7.74, 7.74, 7.67, 7.66, 7.65, 7.64, 7.63, 7.62, 7.51, 7.51, 7.49, 7.49, 7.43, 7.41, 7.26 CDCl3, 7.24, 7.22, 7.20, 6.99, 6.95

2.69, 2.67, 2.65, 2.63

1.25, 1.23, 1.21

1.00

1.01, 1.01, 1.00, 2.00, 3.00

2.01

3.01

^{1}H NMR spectrum of **1e** in CDCl$_3$ (400 MHz)

^{1}H NMR spectrum of **1f** in CDCl$_3$ (400 MHz)

^{1}H NMR spectrum of **1g** in CDCl$_3$ (400 MHz)

8.17, 8.15

7.77, 7.75, 7.73, 7.66, 7.64, 7.62, 7.60, 7.55, 7.52, 7.50, 7.39, 7.37, 7.35, 7.26 CDCl3, 7.12, 7.08, 6.98, 6.96, 6.94, 6.90, 6.88

3.82

1.00, 1.00, 1.00, 1.00, 2.00, 1.00, 1.00, 1.01, 1.01, 3.00

f1 (ppm)

^{1}H NMR spectrum of **1h** in CDCl$_3$ (400 MHz)

^{1}H NMR spectrum of **1i** in CDCl$_3$ (400 MHz)

^{1}H NMR spectrum of **1j** in CDCl$_3$ (400 MHz)

^{1}H NMR spectrum of **1k** in CDCl$_3$ (400 MHz)

^{1}H NMR spectrum of **1l** in CDCl$_3$ (400 MHz)

7.79, 7.77, 7.75, 7.68, 7.66, 7.64, 7.50, 7.48, 7.45, 7.38, 7.36, 7.34, 7.34, 7.32, 7.30, 7.28, 7.26 CDCl3, 7.19, 7.15, 6.99, 6.95

8.18, 8.16

1.00

0.99, 1.02, 0.99, 1.00, 3.02, 1.01, 1.02

^{1}H NMR spectrum of **1m**
in CDCl$_3$ (400 MHz)

7.80
7.79
7.77
7.69
7.67
7.65
7.61
7.54
7.52
7.40
7.37
7.34
7.33
7.31
7.30
7.26 CDCl3

8.21
8.19

6.99
6.95

1.00

1.00
3.00
1.00
1.00
2.00

1.00

^{1}H NMR spectrum of **1n** in CDCl$_3$ (400 MHz)

^{13}C NMR spectrum of **1n** in CDCl$_3$ (100 MHz)

^{1}H NMR spectrum of **1o** in CDCl$_3$ (400 MHz)

^{1}H NMR spectrum of **1p** in CDCl$_3$ (400 MHz)

^{13}C NMR spectrum of **1p** in CDCl$_3$ (100 MHz)

^{1}H NMR spectrum of **1q** in CDCl$_3$ (400 MHz)

^{1}H NMR spectrum of **1r** in CDCl$_3$ (400 MHz)

8.21
8.19
8.18
8.17

7.79
7.77
7.75
7.68
7.66
7.64
7.52
7.50
7.47
7.46
7.43
7.39
7.27
7.26 CDCl3
7.08
7.07
7.06
6.83
6.79

1.03
1.00
1.00
1.00
1.00
1.00
1.00
1.00

^{1}H NMR spectrum of **1s** in CDCl$_3$ (400 MHz)

^{13}C NMR spectrum of **1s** in CDCl$_3$ (100 MHz)

136.05
134.29
134.06
133.54
132.01
130.90
129.01
128.96
128.08
128.01
125.83
124.69

146.80
144.74

77.16 CDCl3

193.04

^{1}H NMR spectrum of **1t** in CDCl$_3$ (400 MHz)

^{1}H and ^{13}C Spectral Charts for 2-((2,5-dioxo-4-aryl-2,5-dihydro-1H-pyrrol-3-yl)amino)benzoic acids (4)

^{1}H NMR spectrum of **4a** in DMSO-d_6 (400 MHz)

Peak labels: 7.24, 7.24, 7.22, 7.22, 7.21, 7.19, 7.18, 7.17, 7.07, 7.07, 7.06, 7.06, 7.05, 7.04, 6.96, 6.95, 6.94, 6.93, 6.92, 6.92, 6.91, 6.90

13.62, 11.01, 10.38, 7.91, 7.90, 7.89, 7.89, 7.88, 6.17, 6.17, 6.16, 6.15, 2.50 DMSO-d_6

Integrations: 0.35, 0.98, 0.98, 1.00, 3.00, 2.00, 2.00, 1.00

f1 (мд)

^{13}C NMR spectrum of **4a** in DMSO-d_6 (100 MHz)

138.87
135.20
132.07
130.96
129.87
129.62
127.62
127.36
121.97
120.42
117.72
172.65
169.30
106.47
39.52 DMSO-d6

f1 (ppm)

^{1}H NMR spectrum of **4b** in DMSO-d_6 (400 MHz)

^{13}C NMR spectrum of **4b** in DMSO-d_6 (100 MHz)

172.72
169.42
169.40

139.07
137.11
134.53
132.24
130.98
129.53
127.98
126.93
121.75
120.19
117.27

107.14

39.52 DMSO-d6

20.98

f1 (ppm)

^{1}H NMR spectrum of **4c** in DMSO-d_6 (400 MHz)

^{13}C NMR spectrum of **4c** in DMSO-d_6 (100 MHz)

^{1}H NMR spectrum of **4d** in DMSO-d_6 (400 MHz)

^{13}C NMR spectrum of **4d** in DMSO-d_6 (100 MHz)

^{1}H NMR spectrum of **4e** in DMSO-d_6 (400 MHz)

^{13}C NMR spectrum of **4e** in DMSO-d_6 (100 MHz)

^{1}H NMR spectrum of **4f** in DMSO-d_6 (400 MHz)

^{13}C NMR spectrum of **4f** in DMSO-d_6 (100 MHz)

Peaks: 172.83, 169.52, 169.47, 158.73, 139.18, 133.85, 132.24, 130.98, 122.08, 121.52, 119.97, 117.24, 112.94, 107.48, 55.17, 39.52 DMSO-d6

^{1}H NMR spectrum of **4g** in DMSO-d_6 (400 MHz)

^{13}C NMR spectrum of **4g** in DMSO-d_6 (100 MHz)

^{1}H NMR spectrum of **4h** in DMSO-d_6 (400 MHz)

138.73
135.26
132.10
131.73
131.65
131.02
126.31
126.28
122.16
120.54
118.01
114.44
114.22

172.64
169.27
169.23

162.52
160.07

105.32

^{13}C NMR spectrum of **4h** in DMSO-d_6 (100 MHz)

f1 (ppm)

S40

^{1}H NMR spectrum of **4i** in DMSO-d_6 (400 MHz)

^{13}C NMR spectrum of **4i** in DMSO-d_6 (100 MHz)

^{1}H NMR spectrum of **4j** in DMSO-d_6 (400 MHz)

^{13}C NMR spectrum of **4j** in DMSO-d_6 (100 MHz)

^{1}H NMR spectrum of **4k** in DMSO-d_6 (400 MHz)

^{13}C NMR spectrum of **4k** in DMSO-d_6 (100 MHz)

^{1}H NMR spectrum of **4l** in DMSO-d_6 (400 MHz)

^{13}C NMR spectrum of **4l** in DMSO-d_6 (100 MHz)

^{1}H NMR spectrum of **4m**
in DMSO-d_6 (400 MHz)

^{13}C NMR spectrum of **4m**
in DMSO-d_6 (100 MHz)

^{1}H NMR spectrum of **4n** in DMSO-d_6 (400 MHz)

^{13}C NMR spectrum of **4n** in DMSO-d_6 (100 MHz)

^{1}H NMR spectrum of **4o** in DMSO-d_6 (400 MHz)

^{13}C NMR spectrum of **4o** in DMSO-d_6 (100 MHz)

^{1}H NMR spectrum of **4p** in DMSO-d_6 (400 MHz)

^{13}C NMR spectrum of **4p** in DMSO-d_6 (100 MHz)

^{1}H NMR spectrum of **4q** in DMSO-d_6 (400 MHz)

^{13}C NMR spectrum of **4q** in DMSO-d_6 (100 MHz)

^{1}H NMR spectrum of **4r** in DMSO-d_6 (400 MHz)

^{13}C NMR spectrum of **4r** in DMSO-d_6 (100 MHz)

132.66
132.53
131.06
130.18
129.53
128.65
126.47
121.80
120.30
117.18

171.82
169.51
169.06

139.12

105.32

39.52 DMSO-d6

f1 (ppm)

^{1}H NMR spectrum of **4s** in DMSO-d_6 (400 MHz)

S61

^{13}C NMR spectrum of **4s** in DMSO-d_6 (100 MHz)

Peaks: 171.89, 169.07, 168.84, 139.05, 137.15, 132.22, 131.92, 131.82, 131.76, 130.94, 130.05, 127.18, 125.20, 122.75, 119.88, 118.61, 104.23, 39.52 DMSO-d6

^{1}H and ^{13}C Spectral Charts for (Z)-2-((3-amino-1-cyano-3-oxo-2-(*p*-tolyl)prop-1-en-1-yl)amino)benzoic acid (**10b**)

^{1}H NMR spectrum of **10b**
in DMSO-d_6 (400 MHz)

^{13}C NMR spectrum of **10b**
in DMSO-d_6 (100 MHz)
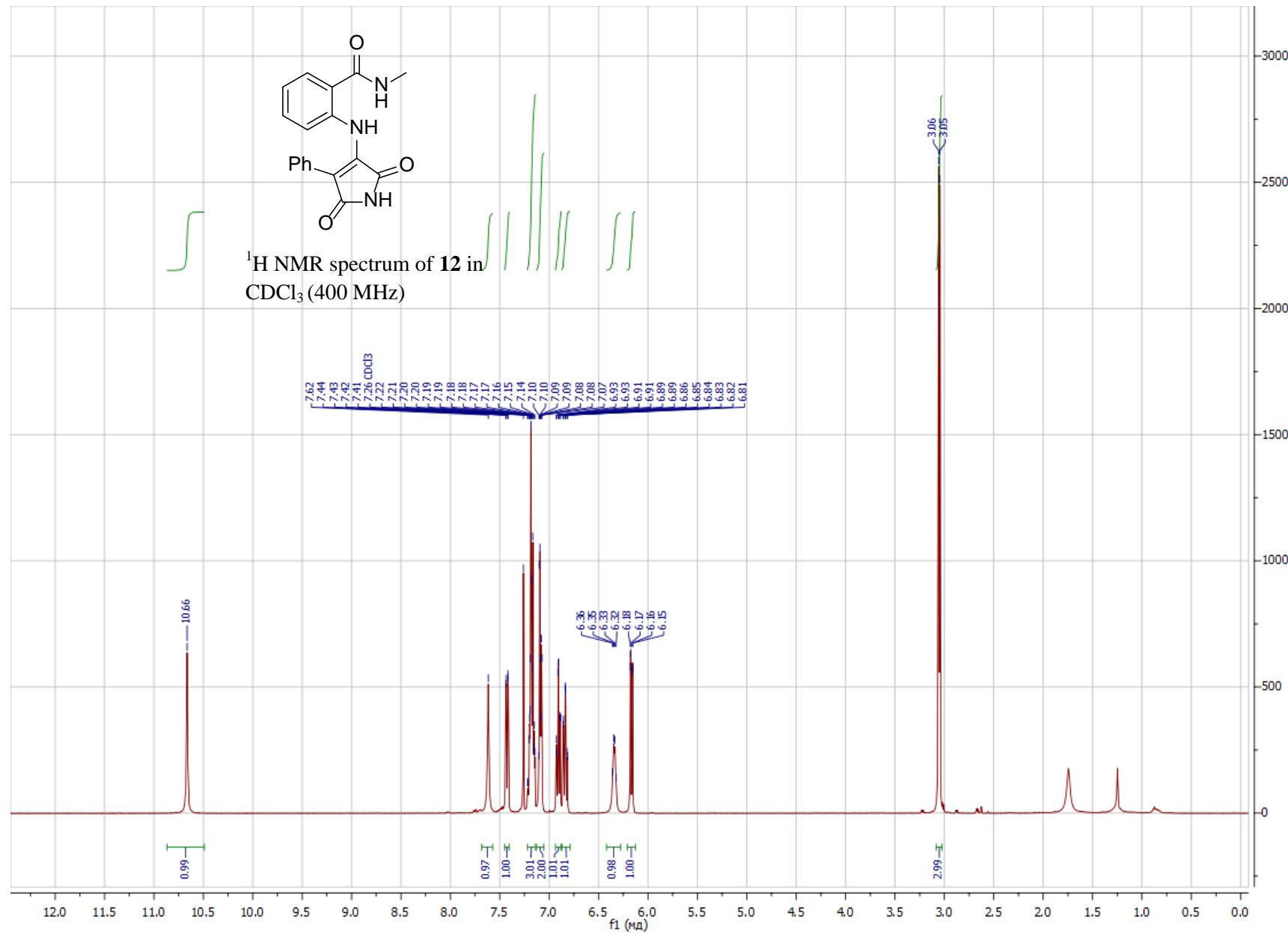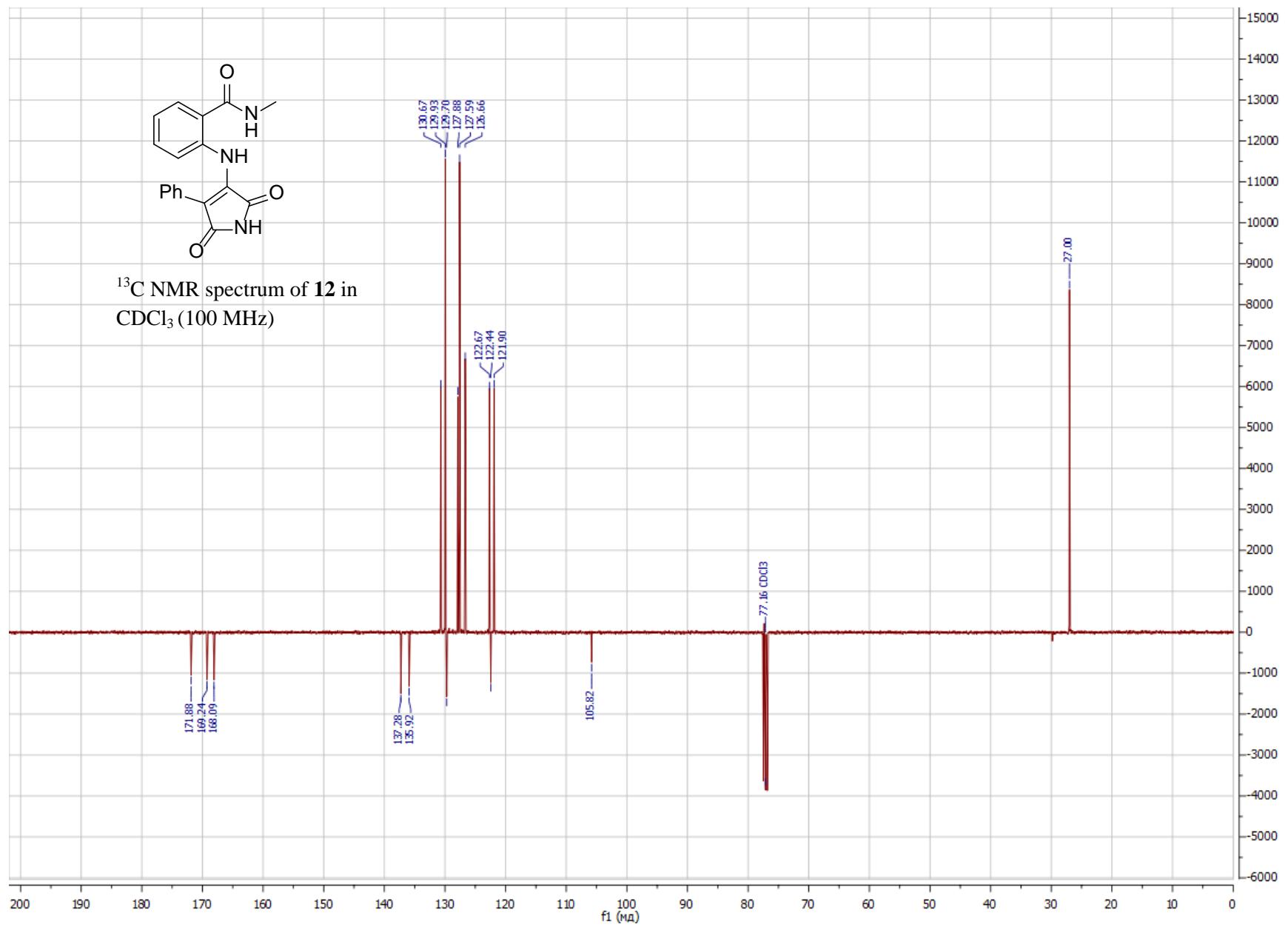
^{1}H and ^{13}C Spectral Charts for acetic 2-(6-cyano-2-methyl-4-oxo-5-(*p*-tolyl)pyrimidin-1(4*H*)-yl)benzoic anhydride (**11b**)

^{1}H NMR spectrum of **11b** in DMSO-d_6 (400 MHz)

^{13}C NMR spectrum of **11b**
in DMSO-d_6 (100 MHz)
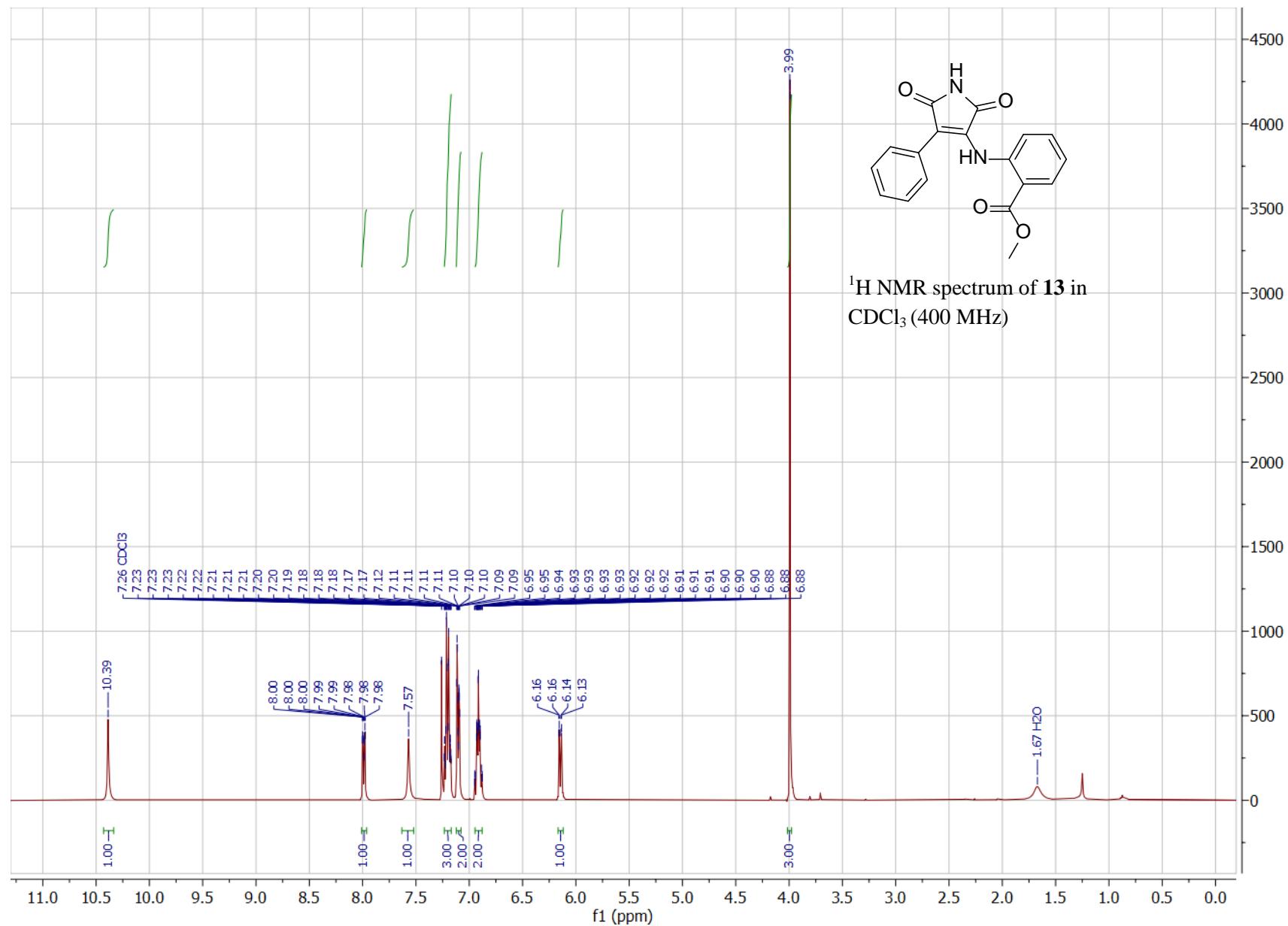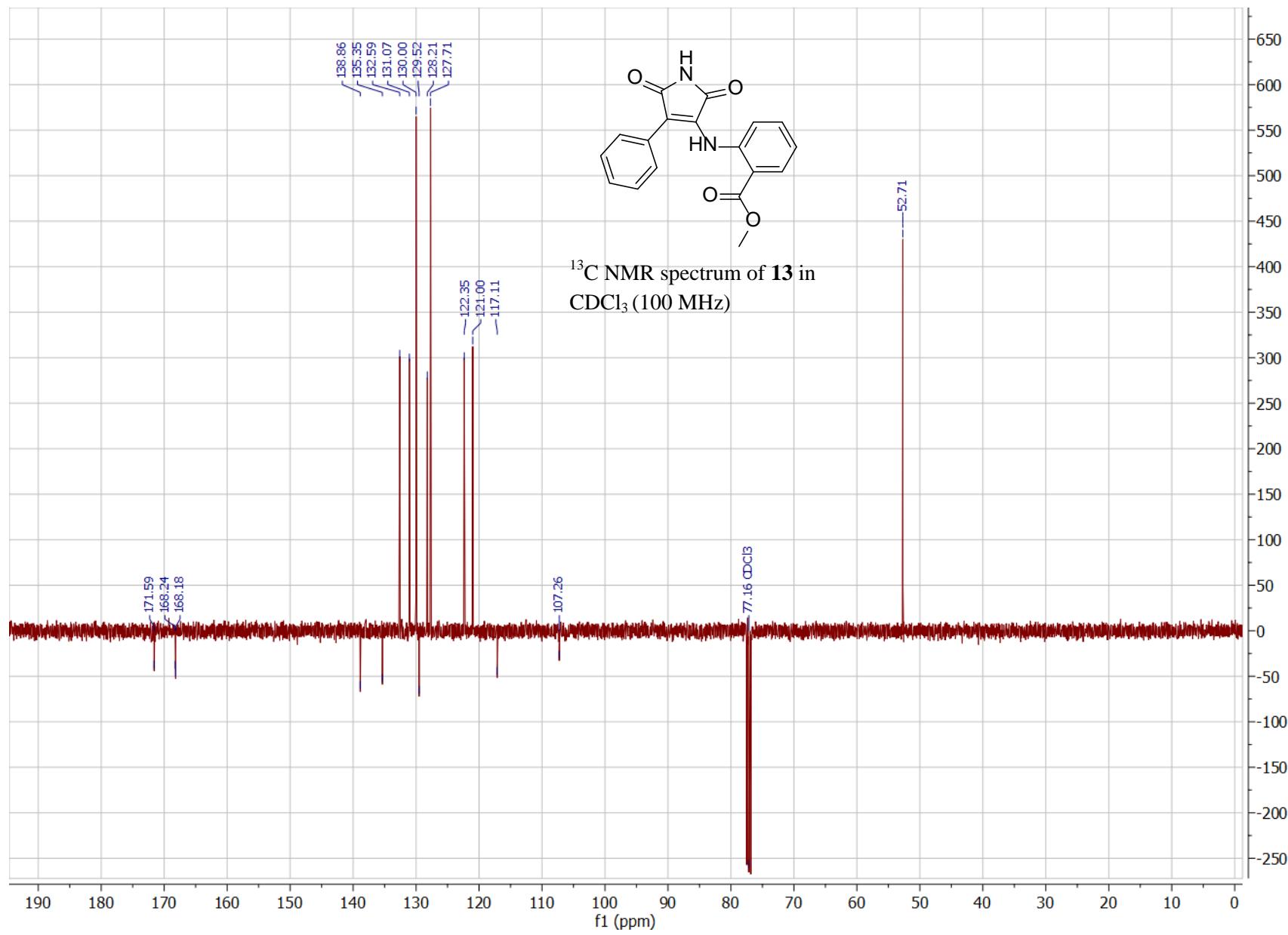
^{1}H and ^{13}C Spectral Charts for 2-((2,5-dioxo-4-phenyl-2,5-dihydro-1*H*-pyrrol-3-yl)amino)-*N*-methylbenzamide (**12**)

^{1}H NMR spectrum of **12** in CDCl$_3$ (400 MHz)

^{13}C NMR spectrum of **12** in CDCl$_3$ (100 MHz)
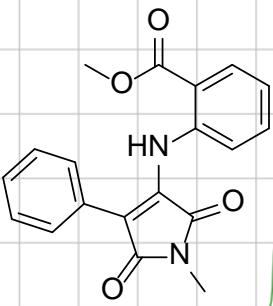
^{1}H and ^{13}C Spectral Charts for methyl 2-((2,5-dioxo-4-phenyl-2,5-dihydro-1*H*-pyrrol-3-yl)amino)benzoate (**13**)

^{1}H NMR spectrum of **13** in CDCl$_3$ (400 MHz)

^{13}C NMR spectrum of **13** in CDCl$_3$ (100 MHz)
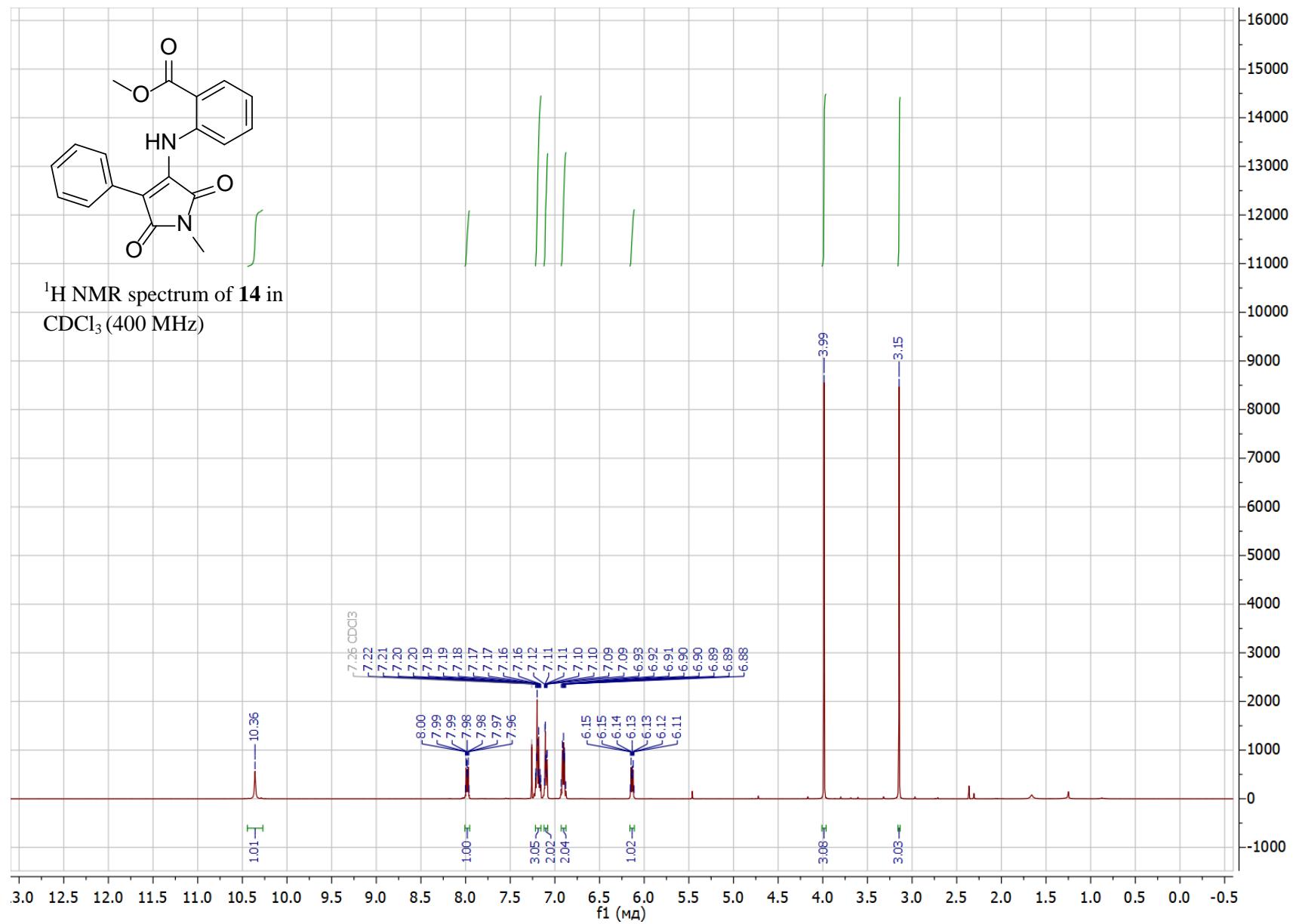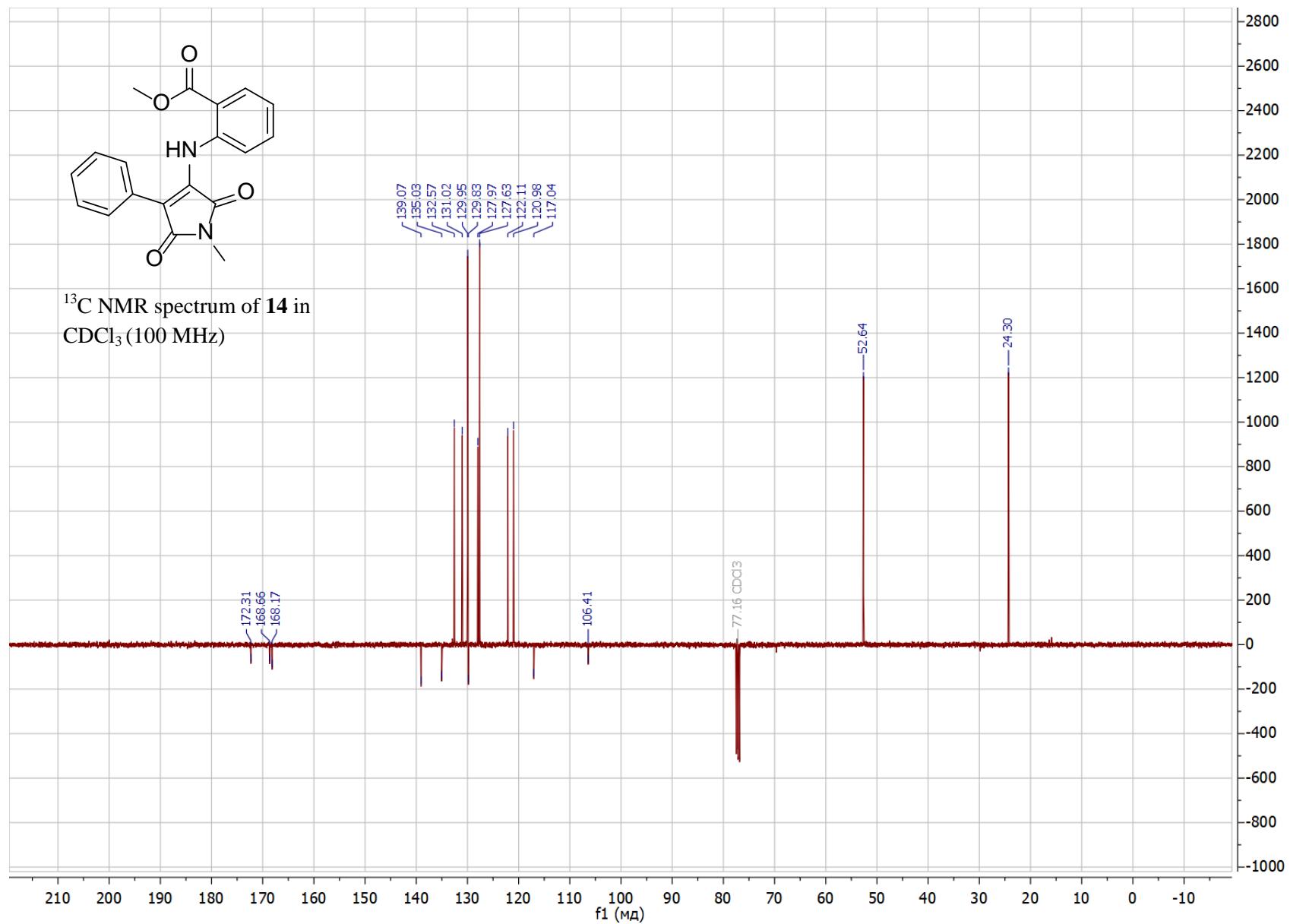
^{1}H and ^{13}C Spectral Charts for methyl 2-((1-methyl-2,5-dioxo-4-phenyl-2,5-dihydro-1H-pyrrol-3-yl)amino)benzoate (**14**)
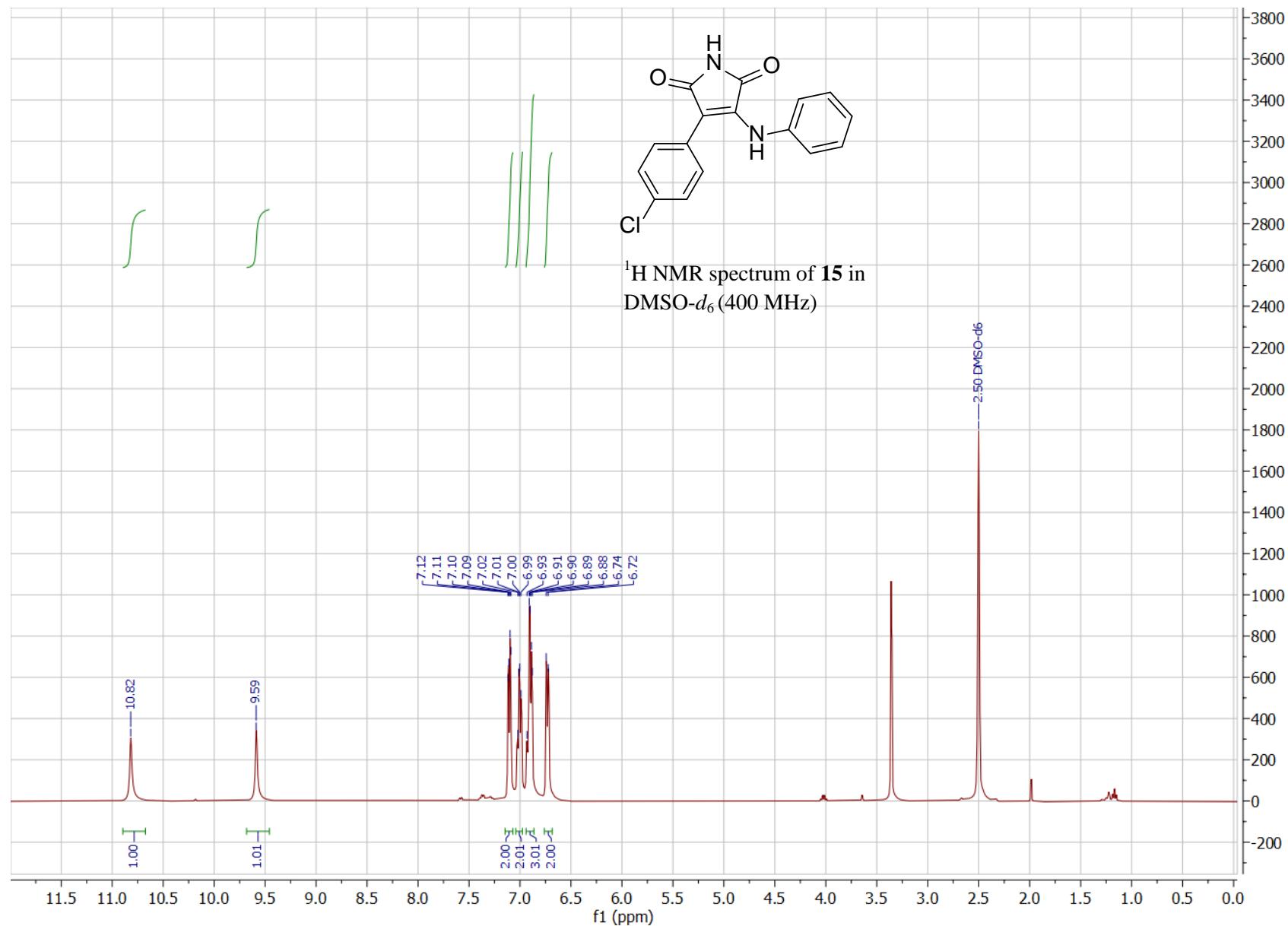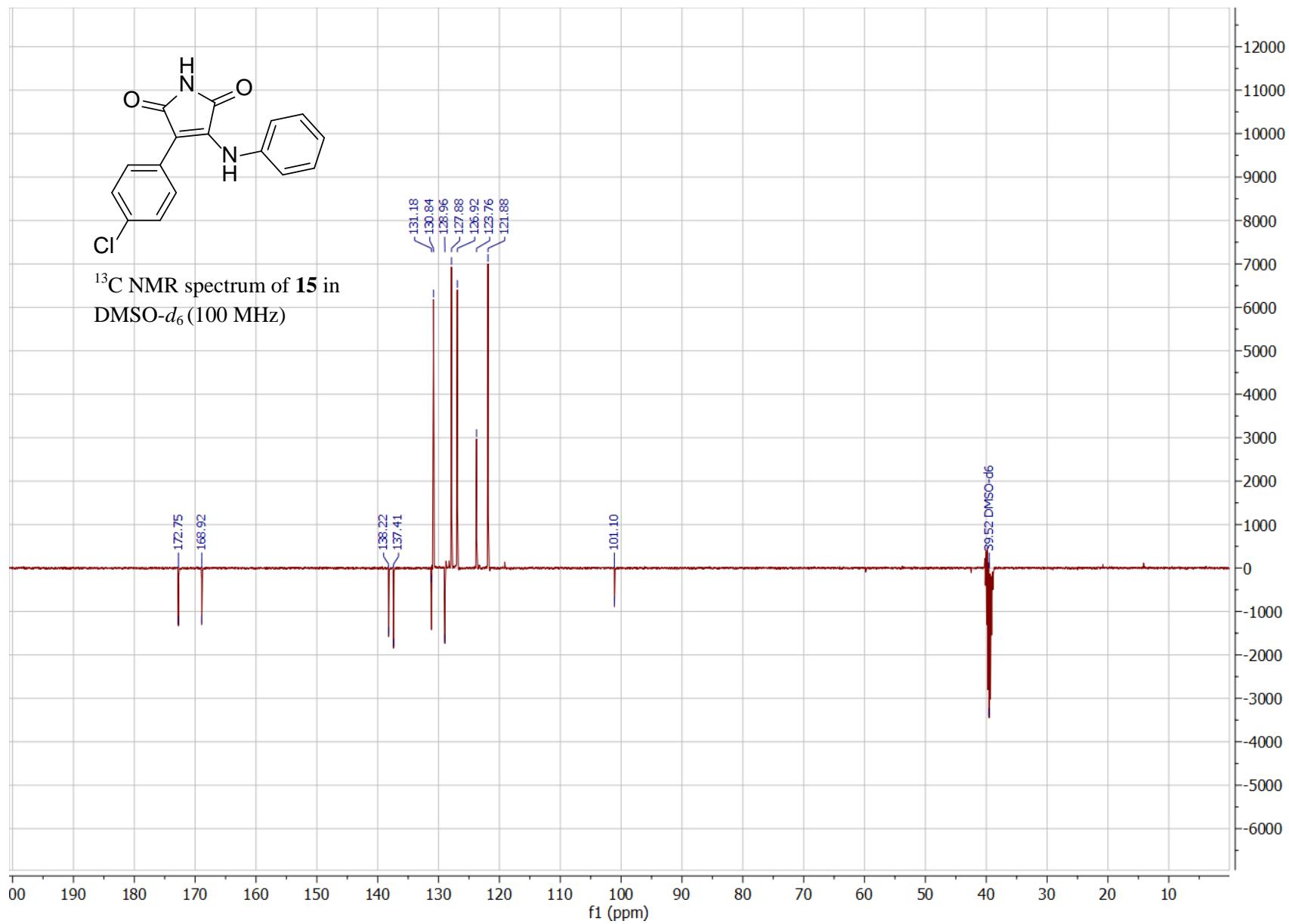
^{1}H NMR spectrum of **14** in CDCl$_3$ (400 MHz)

^{13}C NMR spectrum of **14** in CDCl$_3$ (100 MHz)

172.31
168.66
168.17
139.07
135.03
132.57
131.02
129.95
129.83
127.97
127.63
122.11
120.98
117.04
106.41
77.16 CDCl3
52.64
24.30
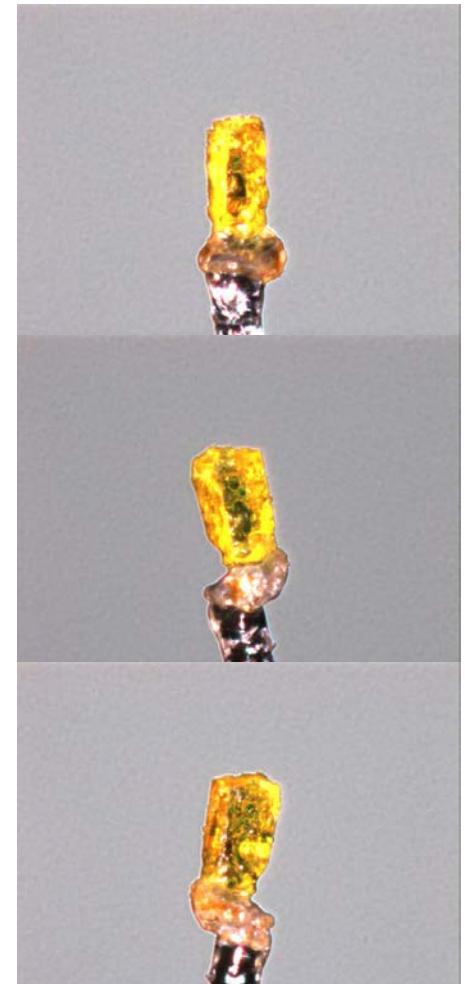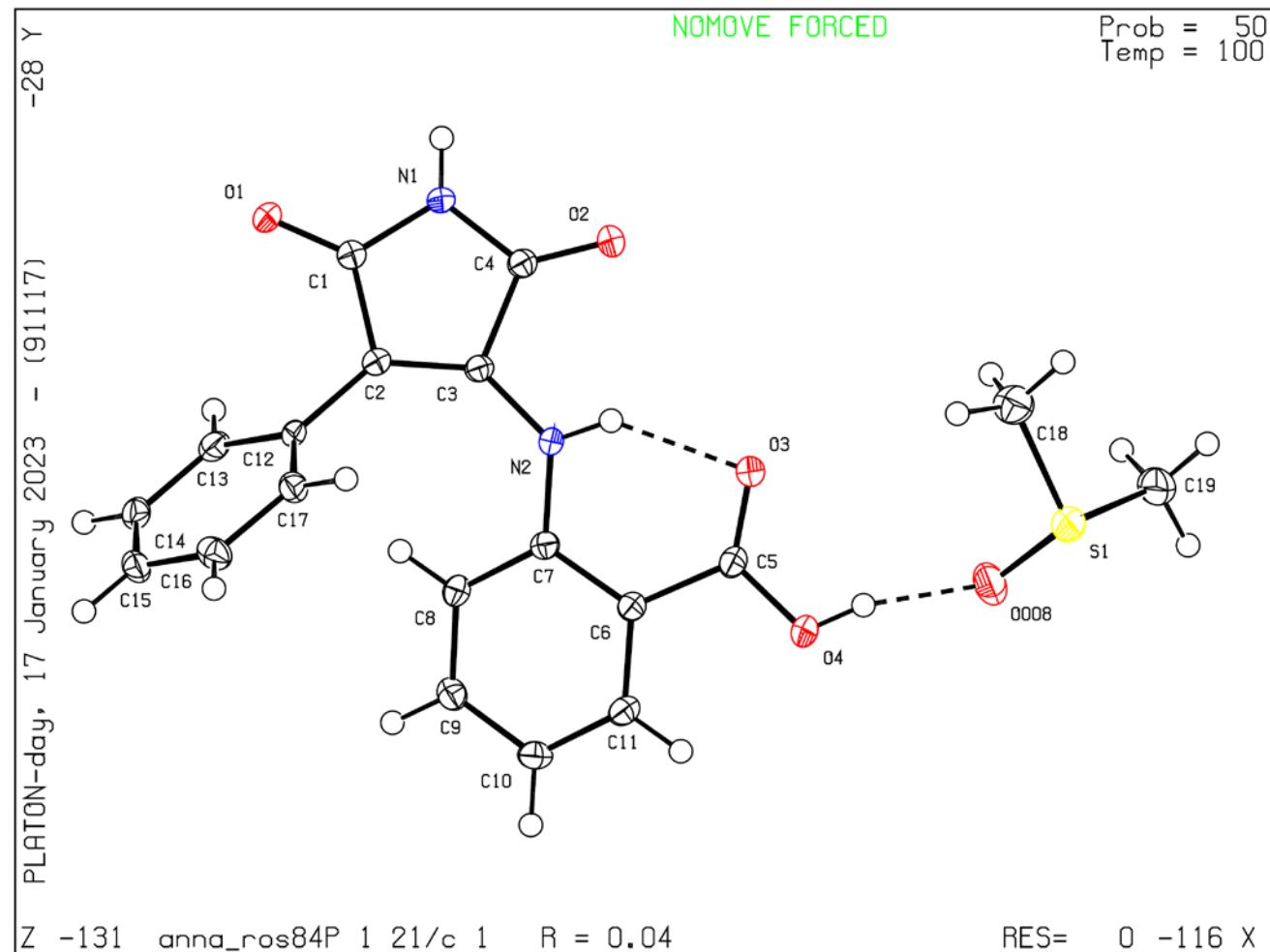
^{1}H and ^{13}C Spectral Charts for 3-(4-chlorophenyl)-4-(phenylamino)-1H-pyrrole-2,5-dione (15)

^{1}H NMR spectrum of **15** in DMSO-d_6 (400 MHz)

2.50 DMSO-d6

7.12
7.11
7.10
7.09
7.02
7.01
7.00
6.99
6.93
6.91
6.90
6.89
6.88
6.74
6.72

10.82
9.59

1.00
1.01
2.00
2.01
3.01
2.00

f1 (ppm)

^{13}C NMR spectrum of **15** in DMSO-d_6 (100 MHz)

X-Ray Crystallography data for 2-((2,5-dioxo-4-aryl-2,5-dihydro-1H-pyrrol-3-yl)amino)benzoic acid **(4a)**

Figure S1. ORTEP drawing of the crystal structure showing 50% probability thermal ellipsoids (left, CCDC 2236719) and microphotography of the single crystal (right) of compound **4a** used for X-Ray diffraction analysis at the bottom.

Table S1 Crystal data and structure refinement for 4a.

Identification code	ANNA_ROS845_YELLOW_2
Empirical formula	$C_{19}H_{18}N_2O_5S$
Formula weight	386.41
Temperature/K	100.01(10)
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	9.71100(10)
b/Å	8.91200(10)
c/Å	20.7791(2)
α/°	90
β/°	99.2150(10)
γ/°	90
Volume/Å^3	1775.11(3)
Z	4
ρ_{calc}g/cm^3	1.446
μ/mm^{-1}	1.928
F(000)	808.0
Crystal size/mm^3	$0.339 \times 0.178 \times 0.15$
Radiation	synchrotron (λ = 1.54184)
2Θ range for data collection/°	8.622 to 152.264
Index ranges	$-12 \leq h \leq 12, -11 \leq k \leq 7, -26 \leq l \leq 25$
Reflections collected	19149
Independent reflections	3715 [R_{int} = 0.0225, R_{sigma} = 0.0149]
Data/restraints/parameters	3715/0/258
Goodness-of-fit on F^2	1.039
Final R indexes [I>=2σ (I)]	R_1 = 0.0393, wR_2 = 0.1051
Final R indexes [all data]	R_1 = 0.0403, wR_2 = 0.1059
Largest diff. peak/hole / e Å^{-3}	0.36/-0.64

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

Atom	x	y	z	U(eq)
S1	537.6(4)	8138.9(5)	7348.9(2)	27.45(13)
O2	6769.5(11)	9343.0(12)	5950.3(5)	18.8(2)
O1	9174.7(11)	9161.8(14)	4238.5(5)	22.1(3)
O3	3249.0(12)	7963.7(13)	5912.4(5)	21.9(3)
O4	1241.4(13)	6686.9(16)	5722.9(6)	27.9(3)
N2	4919.3(13)	7821.8(14)	5005.1(6)	14.8(3)
N1	8283.1(14)	9468.4(16)	5196.1(6)	19.2(3)
O008	832.2(17)	6864.7(16)	6912.3(7)	38.4(4)
C3	6159.7(15)	8309.3(16)	4855.7(7)	14.5(3)
C7	4028.6(15)	6691.5(16)	4720.5(7)	14.3(3)
C12	6478.3(15)	7833.6(16)	3647.2(7)	14.8(3)
C4	7066.9(15)	9102.4(17)	5416.0(7)	15.9(3)
C8	4373.9(15)	5785.9(16)	4220.4(7)	15.8(3)
C6	2782.2(15)	6381.8(17)	4969.5(7)	15.9(3)
C17	5284.1(16)	8414.8(17)	3267.8(7)	17.3(3)
C9	3484.9(16)	4661.2(17)	3947.5(7)	18.0(3)
C5	2461.0(16)	7107.2(18)	5573.4(7)	18.0(3)
C10	2221.7(16)	4403.8(18)	4163.5(8)	19.9(3)
C2	6853.9(15)	8284.4(17)	4336.4(7)	15.4(3)
C11	1899.1(16)	5243.9(18)	4677.6(8)	19.2(3)
C1	8225.9(16)	8991.2(18)	4555.3(7)	17.6(3)
C16	4943.4(18)	8014.5(17)	2613.2(8)	20.8(3)
C15	5784.0(18)	7022.0(18)	2338.1(8)	21.3(3)
C14	6974.3(17)	6438.9(18)	2713.0(8)	21.7(3)
C13	7330.0(16)	6845.4(17)	3364.8(8)	19.2(3)
C18	1875.2(19)	9489(2)	7327.9(9)	28.3(4)
C19	1071(2)	7428(3)	8154.0(9)	39.4(5)

Table S3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for 4a. The Anisotropic displacement factor exponent takes the form: -$2\pi^2[h^2a*^2U_{11}+2hka*b*U_{12}+...]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S1	19.6(2)	39.3(3)	24.1(2)	−5.87(17)	5.42(16)	−3.97(16)
O2	19.5(5)	23.0(5)	14.6(5)	−2.8(4)	4.9(4)	−2.1(4)
O1	16.0(5)	32.5(6)	19.1(5)	−4.4(5)	6.3(4)	−7.6(5)
O3	20.8(6)	27.3(6)	19.1(5)	−6.9(4)	7.5(4)	−6.8(5)
O4	20.2(6)	43.8(7)	22.2(6)	−11.4(5)	11.0(5)	−11.8(5)
N2	14.4(6)	17.6(6)	13.2(6)	−1.8(5)	4.7(4)	−2.8(5)
N1	14.4(6)	27.9(7)	15.3(6)	−4.9(5)	3.0(5)	−7.6(5)
O008	56.8(9)	37.6(8)	24.8(7)	−10.0(6)	18.5(6)	−17.2(7)
C3	13.3(7)	14.9(6)	15.3(7)	−0.2(5)	2.0(5)	−1.2(5)
C7	14.6(7)	14.2(6)	13.6(6)	2.2(5)	1.1(5)	−0.8(5)
C12	16.3(7)	15.3(7)	13.9(7)	−1.2(5)	6.2(5)	−4.9(5)
C4	15.0(7)	16.6(7)	15.9(7)	0.2(5)	2.4(5)	−0.9(5)
C8	16.4(7)	15.8(7)	15.8(7)	2.3(5)	4.8(5)	−0.2(5)
C6	14.6(7)	18.3(7)	15.0(7)	0.0(5)	2.8(5)	−1.4(5)
C17	20.7(7)	15.9(7)	16.0(7)	−1.4(5)	4.9(6)	−1.5(6)
C9	21.9(7)	16.6(7)	15.9(7)	−1.0(6)	4.1(6)	0.3(6)
C5	15.4(7)	22.4(7)	16.9(7)	0.3(6)	5.1(6)	−1.9(6)
C10	19.7(7)	19.4(7)	20.1(7)	−2.6(6)	1.1(6)	−4.9(6)
C2	13.4(7)	17.2(7)	15.8(7)	−0.6(5)	2.7(5)	−2.3(5)
C11	15.1(7)	22.9(8)	19.8(7)	0.3(6)	3.7(6)	−4.1(6)
C1	16.0(7)	21.2(7)	15.7(7)	−1.7(6)	3.1(5)	−2.9(6)
C16	27.7(8)	18.2(7)	15.6(7)	1.5(6)	0.5(6)	−2.2(6)
C15	32.7(9)	18.3(7)	14.5(7)	−2.9(6)	8.3(6)	−8.6(6)
C14	23.9(8)	19.6(7)	24.7(8)	−7.4(6)	13.1(6)	−6.1(6)
C13	15.5(7)	19.7(7)	23.5(8)	−2.8(6)	6.7(6)	−2.9(6)
C18	32.1(9)	25.2(8)	27.1(9)	3.0(7)	3.6(7)	−2.8(7)
C19	51.1(12)	47.4(12)	21.0(9)	−3.3(8)	9.4(8)	−25.0(10)

Table S4 Bond Lengths for 4a.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
S1	O008	1.5092(14)	C7	C6	1.418(2)
S1	C18	1.7765(19)	C12	C17	1.394(2)
S1	C19	1.786(2)	C12	C2	1.476(2)
O2	C4	1.2101(19)	C12	C13	1.400(2)
O1	C1	1.2246(19)	C8	C9	1.384(2)
O3	C5	1.2214(19)	C6	C5	1.488(2)
O4	C5	1.3256(19)	C6	C11	1.402(2)
N2	C3	1.3624(19)	C17	C16	1.394(2)
N2	C7	1.3960(19)	C9	C10	1.391(2)
N1	C4	1.3722(19)	C10	C11	1.381(2)
N1	C1	1.3906(19)	C2	C1	1.478(2)
C3	C4	1.517(2)	C16	C15	1.387(2)
C3	C2	1.361(2)	C15	C14	1.387(2)
C7	C8	1.398(2)	C14	C13	1.391(2)

Table S5 Bond Angles for 4a.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O008	S1	C18	106.57(9)	C11	C6	C7	118.81(14)
O008	S1	C19	103.99(10)	C11	C6	C5	118.86(13)
C18	S1	C19	99.20(9)	C12	C17	C16	120.20(14)
C3	N2	C7	130.10(13)	C8	C9	C10	120.78(14)
C4	N1	C1	109.98(12)	O3	C5	O4	122.84(14)
N2	C3	C4	113.55(13)	O3	C5	C6	124.13(14)
C2	C3	N2	138.06(14)	O4	C5	C6	112.99(13)
C2	C3	C4	108.39(12)	C11	C10	C9	118.76(14)
N2	C7	C8	121.68(13)	C3	C2	C12	134.18(14)
N2	C7	C6	119.55(13)	C3	C2	C1	106.60(13)
C8	C7	C6	118.64(13)	C12	C2	C1	119.02(13)

Table S5 Bond Angles for 4a.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C17	C12	C2	120.12(13)	C10	C11	C6	121.87(14)
C17	C12	C13	119.38(14)	O1	C1	N1	123.96(14)
C13	C12	C2	120.48(14)	O1	C1	C2	127.32(14)
O2	C4	N1	127.44(14)	N1	C1	C2	108.71(12)
O2	C4	C3	126.29(14)	C15	C16	C17	120.13(15)
N1	C4	C3	106.27(12)	C14	C15	C16	119.98(14)
C9	C8	C7	120.97(14)	C15	C14	C13	120.25(15)
C7	C6	C5	121.98(13)	C14	C13	C12	120.05(15)

Table S6 Torsion Angles for 4a.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
N2	C3	C4	O2	-1.5(2)	C8	C7	C6	C5	169.08(14)
N2	C3	C4	N1	177.83(13)	C8	C7	C6	C11	-4.1(2)
N2	C3	C2	C12	8.4(3)	C8	C9	C10	C11	-3.1(2)
N2	C3	C2	C1	-177.02(17)	C6	C7	C8	C9	3.5(2)
N2	C7	C8	C9	179.41(13)	C17	C12	C2	C3	52.2(2)
N2	C7	C6	C5	-6.9(2)	C17	C12	C2	C1	-121.90(16)
N2	C7	C6	C11	179.95(13)	C17	C12	C13	C14	-0.7(2)
C3	N2	C7	C8	3.7(2)	C17	C16	C15	C14	-0.7(2)
C3	N2	C7	C6	179.54(14)	C9	C10	C11	C6	2.5(2)
C3	C2	C1	O1	178.41(16)	C5	C6	C11	C10	-172.24(15)
C3	C2	C1	N1	-2.22(18)	C2	C3	C4	O2	178.98(15)
C7	N2	C3	C4	-157.52(14)	C2	C3	C4	N1	-1.70(17)
C7	N2	C3	C2	21.8(3)	C2	C12	C17	C16	178.66(14)
C7	C8	C9	C10	0.1(2)	C2	C12	C13	C14	-

Table S6 Torsion Angles for 4a.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
									179.36(14)
C7	C6	C5	O3	-3.0(2)	C11	C6	C5	O3	170.11(15)
C7	C6	C5	O4	179.27(14)	C11	C6	C5	O4	-7.6(2)
C7	C6	C11	C10	1.1(2)	C1	N1	C4	O2	179.57(15)
C12	C17	C16	C15	0.7(2)	C1	N1	C4	C3	0.26(17)
C12	C2	C1	O1	-6.0(2)	C16	C15	C14	C13	0.0(2)
C12	C2	C1	N1	173.37(13)	C15	C14	C13	C12	0.7(2)
C4	N1	C1	O1	-179.45(15)	C13	C12	C17	C16	0.0(2)
C4	N1	C1	C2	1.15(18)	C13	C12	C2	C3	-129.14(18)
C4	C3	C2	C12	-172.30(16)	C13	C12	C2	C1	56.8(2)
C4	C3	C2	C1	2.32(16)					

Table S7 Hydrogen Atom Coordinates ($\text{Å}\times10^4$) and Isotropic Displacement Parameters ($\text{Å}^2\times10^3$) for 4a.

Atom	x	y	z	U(eq)
H8	5213.41	5941.77	4069.08	19
H17	4712.75	9071.79	3451.89	21
H9	3734.87	4070.5	3615.73	22
H10	1605.9	3679.39	3965.43	24
H11	1072.51	5049.15	4834.72	23
H16	4151.11	8413.49	2360.39	25
H15	5549.51	6747.65	1902.47	26
H14	7536.51	5773.62	2527.82	26
H13	8134.35	6460.41	3613.07	23
H18A	2769.5	9020.31	7447.35	42
H18B	1776.11	10283.56	7629.21	42
H18C	1802.75	9894.13	6895.58	42

Table S7 Hydrogen Atom Coordinates ($\text{Å}\times10^4$) and Isotropic Displacement Parameters ($\text{Å}^2\times10^3$) for 4a.

Atom	x	y	z	U(eq)
H19A	495.38	6588.39	8225.89	59
H19B	977.14	8200.69	8465.97	59
H19C	2027.56	7113.07	8202.85	59
H2	4720(20)	8130(20)	5362(12)	30(6)
H1	9000(20)	9870(30)	5407(11)	31(6)
H4	1150(30)	6960(30)	6107(15)	61(9)

Experimental

Single crystals of $C_{19}H_{18}N_2O_5S$ 4a were obtained by slow evaporation of saturated solution in EtOAc. A suitable crystal was selected and mounted on the glass stick by acrylic glue on a SuperNova, Dual, Cu at home/near, AtlasS2 diffractometer. The crystal was kept at 100.01(10) K during data collection. Using Olex2 [S1], the structure was solved with the SHELXT [S2] structure solution program using Intrinsic Phasing and refined with the SHELXL [S3] 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 4a

Crystal Data for $C_{19}H_{18}N_2O_5S$ (M =386.41 g/mol): monoclinic, space group P2$_1$/c (no. 14), a = 9.71100(10) Å, b = 8.91200(10) Å, c = 20.7791(2) Å, β = 99.2150(10)°, V = 1775.11(3) Å^3, Z = 4, T = 100.01(10) K, μ(synchrotron) = 1.928 mm^{-1}, *Dcalc* = 1.446 g/cm^3, 19149 reflections measured (8.622° ≤ 2Θ ≤ 152.264°), 3715 unique (R_{int} = 0.0225, R$_{sigma}$ = 0.0149) which were used in all calculations. The final R_1 was 0.0393 (I > 2σ(I)) and wR_2 was 0.1059 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.

Details:
```
1. Fixed Uiso
 At 1.2 times of:
  All C(H) groups
 At 1.5 times of:
  All C(H,H,H) groups
2.a Aromatic/amide H refined with riding coordinates:
 C8(H8), C17(H17), C9(H9), C10(H10), C11(H11), C16(H16), C15(H15), C14(H14),
 C13(H13)
2.b Idealised Me refined as rotating group:
 C18(H18A,H18B,H18C), C19(H19A,H19B,H19C)
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References

(S1) Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. OLEX2: a Complete Structure Solution, Refinement and Analysis Program. *J. Appl. Cryst*. **2009**, *42*, 339-341.

(S2) Sheldrick, G.M. SHELXT - Integrated Space-Group and Crystal-Structure Determination. *Acta Cryst*. **2015**, *A71*, 3-8.

(S3) Sheldrick, G.M. Crystal Structure Refinement with SHELXL. *Acta Cryst*. **2015**, *C71*, 3-8.