Supporting Informations

tert-Butyl Nitrite Mediated Synthesis of 1,2,4-Oxadiazol-5(*4H*)-ones from Terminal Aryl Alkenes

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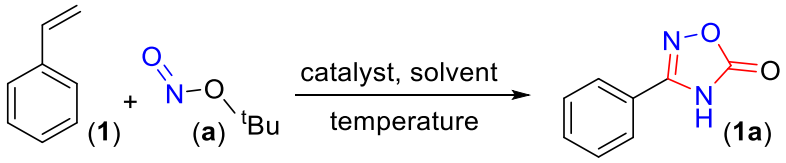
Contents:

General information:

All the reagents were commercial grade and purified according to the established procedures. Organic extracts were dried over anhydrous sodium sulphate. Solvents were removed in a rotary evaporator under reduced pressure. Silica gel (60-120 mesh size) was used for the column chromatography. Reactions were monitored by TLC on silica gel 60 F_{254} (0.25 mm). NMR spectra were recorded in DMSO-d_6 as the internal standard for ^{1}H NMR (400 and 600 MHz) and in for ^{13}C NMR (100 and 150 MHz) DMSO-d_6 as the internal standard. MS spectra were recorded using ESI mode. IR spectra were recorded in KBr or neat.

Table S1. Optimization of the Reaction Conditions[a]



entry	catalyst (mol %)	solvent	TBN (equiv)	yield %[b]
1	$Sc(OTf)_3$ (10)	DMSO	^{t}BuONO (3)	47
2	$Sc(OTf)_3$ (10)	DMF	^{t}BuONO (3)	31
3	$Sc(OTf)_3$ (10)	DMA	^{t}BuONO (3)	29
4	$Sc(OTf)_3$ (10)	CH_3CN	^{t}BuONO (3)	0
5	$Sc(OTf)_3$ (10)	DCE	^{t}BuONO (3)	0
6	$Cu(OTf)_3$ (10)	DMSO	^{t}BuONO (3)	41
7	-	DMSO	^{t}BuONO (3)	59
8	**-**	**DMSO**	**^{t}BuONO (4)**	**67**
9	-	DMSO	^{t}BuONO (5)	69
10	-	DMSO	^{t}BuONO (4)	51[c]
11	-	DMSO	^{t}BuONO (4)	57[d]

[a]Reaction conditions: Styrene (**1**) (0.25 mmol) and *tert*-butyl nitrite (**a**) (equiv) and solvent (2 mL) at 80 °C. [b]Yield after 6 h. [c]Temperature 100 °C. [d]Temperature 60 °C.

Crystallographic description:

Diffraction data were collected at 292 K with MoKα radiation (λ = 0.71073 Å) using a Bruker Nonius SMART APEX CCD diffractometer equipped with graphite monochromator and Apex CD camera. The SMART software was used for data collection and for indexing the reflections and determining the unit cell parameters. Data reduction and cell refinement were performed using SAINT[1,2] software and the space groups of these crystals were determined from systematic absences by XPREP and further justified by the refinement results. The structures were solved by direct methods and refined by full-matrix least-squares calculations using SHELXTL-97[3] software. All the non-H atoms were refined in the anisotropic approximation against F^2 of all reflections.

1. G. M. Sheldrick, SADABS, 1996, based on the method described in: R. H. Blessing, *Acta Crystallogr.* 1995, **A51**, 33–38.
2. SMART and SAINT, Siemens Analytical X-ray Instruments Inc., Madison, WI, 1996.
3. G. M. Sheldrick, *Acta Crystallogr.*, 2008, **A64**, 112–122.

Crystallographic description of 3-(*p*-tolyl)-1,2,4-oxadiazol-5(4H)-one (3a):

$C_{17}H_{11}ClN_2$, crystal dimensions 0.26 x 0.22 x 0.14 mm, M_r = 176.17, Orthorhombic, space group P 21/c, a = 9.8201(13), b = 11.2635(14), c = 7.6733(10) Å, α = 90 °, β = 103.464 °(8), γ = 90 °, V = 825.41(19) Å^3, Z = 4, ρ_{calcd} = 1.418 mg/m^3, μ = 0.103 mm^{-1}, F(000) = 368.0, refinement method = full-matrix least-squares on F^2, final R indices [$I > 2\sigma(I)$]: R_1 = 0.0362, wR_2 = 0.0735(1463), goodness of fit = 1.000. CCDC 1911236 for 3-(*p*-tolyl)-1,2,4-oxadiazol-5(4H)-one (**3a**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

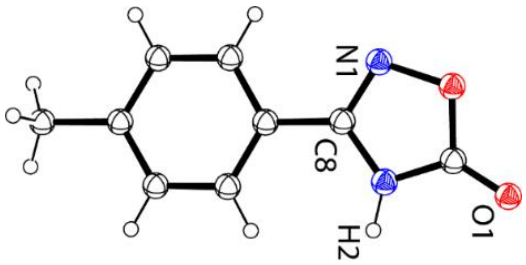


Figure S1. ORTEP Diagram of Compound (**3a**)

General Procedure for the Synthesis of 3-Phenyl-1,2,4-oxadiazol-5(4H)-one (1a) from Styrene (1) and *tert*-Butyl nitrite (a):

To an oven-dried 10 mL round bottom flask fitted with a reflux condenser was added styrene (**1**) (26 mg, 0.25 mmol), *tert*-butyl nitrite (**a**) (103 mg, 1 mmol), and DMSO (1.5 mL). The reaction mixture was heated in an oil bath preheated to 80 °C for 6 h. The reaction mixture was cooled to room temperature, admixed with ethyl acetate (25 mL) and the organic layer was washed with cold water (1 x 10 mL), saturated sodium bicarbonate solution (1 x 5 mL). The organic layer was dried over anhydrous sodium sulfate (Na_2SO_4), and solvent was evaporated under reduced pressure. The crude product so obtained was purified over a column of silica gel (hexane / ethyl acetate, 9:1) to give pure 3-phenyl-1,2,4-oxadiazol-

5(4H)-one (**1a**) (27 mg, yield 67%). The identity and purity of the product were confirmed by spectroscopic analysis.

H_2O^{18} Labelling Experiment for the Formation of 3-Phenyl-1,2,4-oxadiazol-5(4H)-one (1a) from Styrene:

To an oven-dried 10 mL round bottom flask fitted with a reflux condenser was added sequentially styrene (**1**) (26 mg, 0.25 mmol), *tert*-butyl nitrite (**a**) (103 mg, 1 mmol), H_2O^{18} (9 mg, 0.5 mmol), and DMSO (1.5 mL). The reaction mixture was heated in an oil bath preheated to 80 ^oC. After completion of the reaction (6 h) the crude product was admixed with ethyl acetate (25 mL) and the organic layer was washed with cold water (1 x 10 mL) saturated sodium bicarbonate solution (1 x 5 mL), dried over anhydrous sodium sulfate (Na_2SO_4), and evaporated under reduced pressure. The identity of the ^{18}O labeled product was confirmed by HRMS (Figure S4) and $^{13}C\{^1H\}$ NMR (Figure S5).

Mechanistic Investigation:

ESI-MS study for the detection of reaction intermediates during the synthesis of 3-phenyl-1,2,4-oxadiazol-5(*4H*)-one (1a) from styrene (1) and *tert*-butyl nitrite (a) at different time interval:

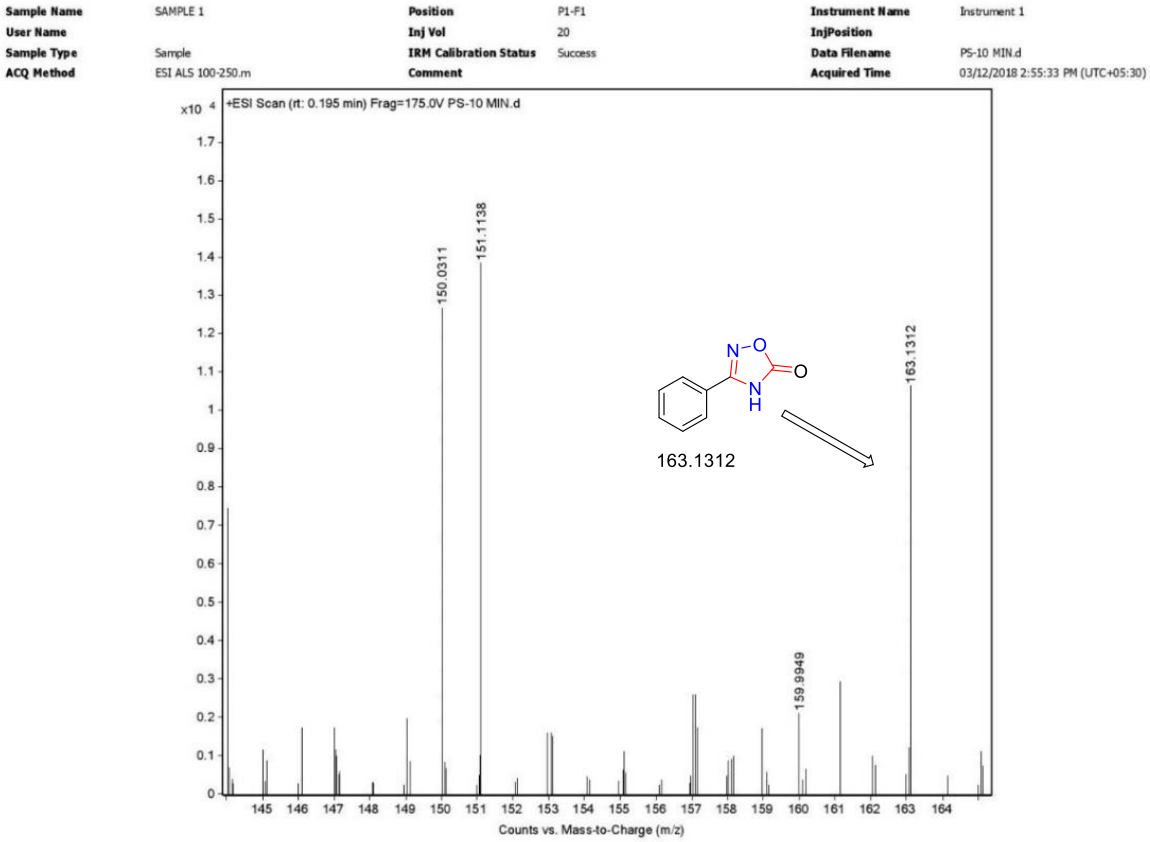
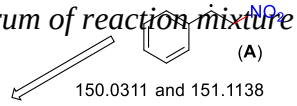


Figure S1. *HRMS spectrum of reaction mixture after 10 minute*



Sample Name	SAMPLE 1	**Position**	P1-F1	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	20	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	PS-10 MIN.d
ACQ Method	ESI ALS 100-250.m	**Comment**		**Acquired Time**	03/12/2018 2:55:33 PM (UTC+05:30)

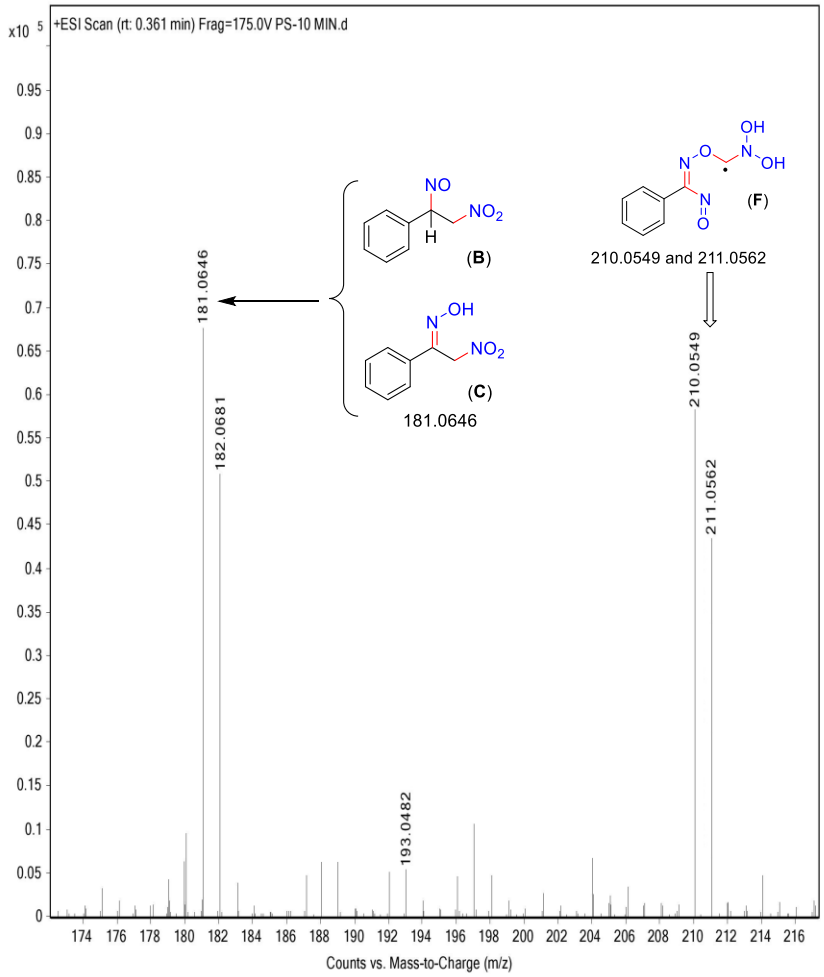


Figure S2. *HRMS spectrum of the reaction mixture after 10 minute*

ESI-MS and $^{13}C\{^{1}H\}$ NMR for the H_2O^{18} labelled experiment for the formation of 3-phenyl-1,2,4-oxadiazol-5(4H)-one (1a) from styrene

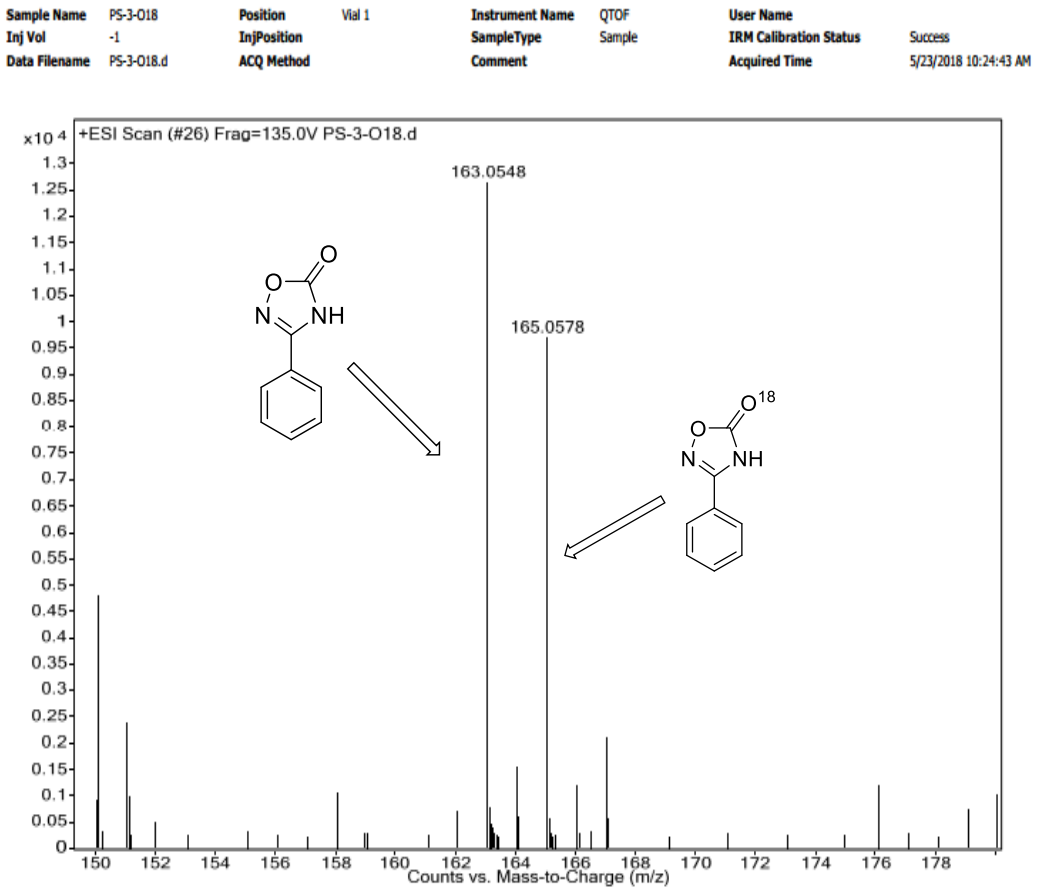


Figure S3. *HRMS spectrum of ^{18}O labelled (**1a**)*

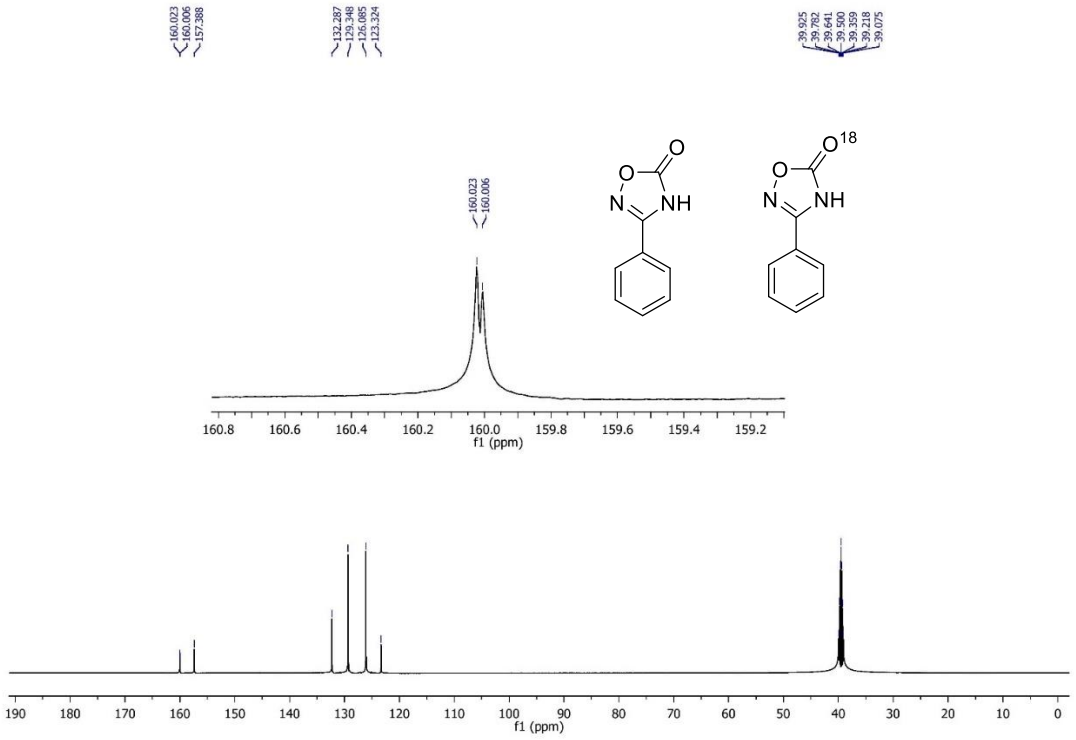
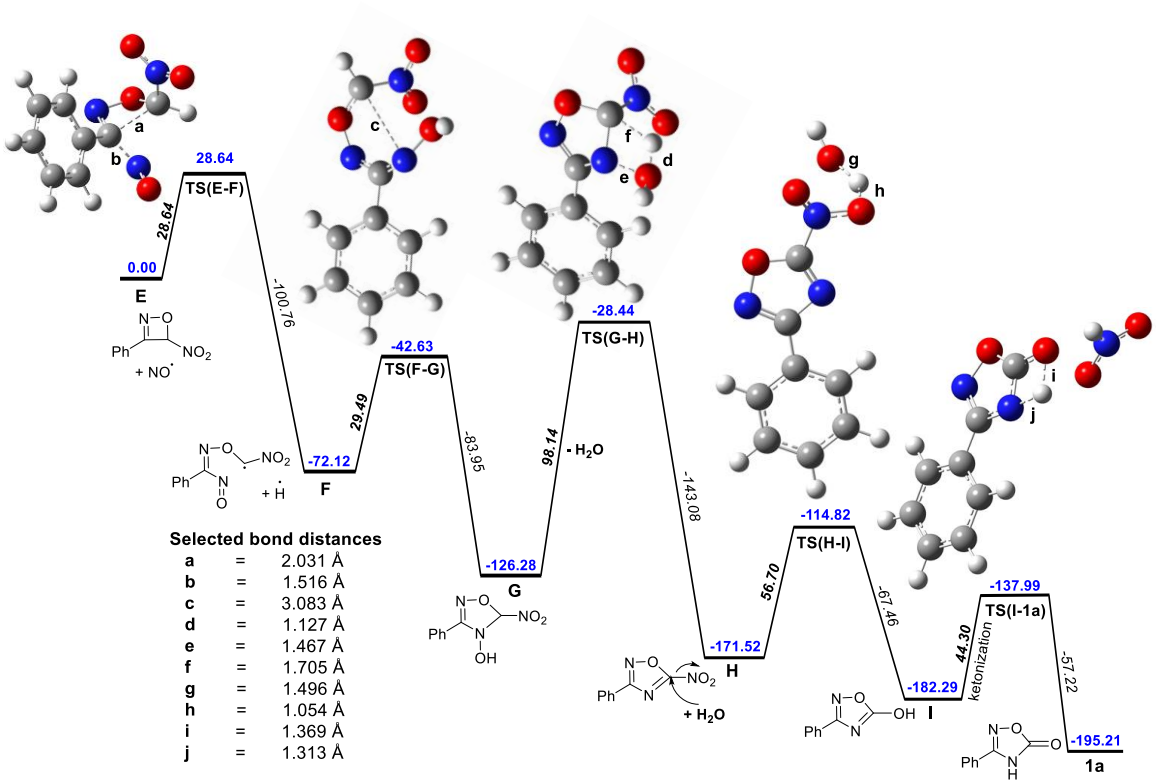


Figure S4. *$^{13}C\{^{1}H\}$ NMR spectrum of ^{18}O labelled (**1a**)*

DFT Calculations:



***Scheme* S1.** Energy Profile Diagram for the Formation of (**1a**) from Intermediate **E** (see Scheme 5 and 6 for further details). Relative energy (blue colour), activation barrier (italic bold) and stabilization gained (italic normal) are given in kcal/mol and calculated at M06/6-31+G(d,p) level of DFT. Selected interatomic distances of the transition states are given in Ångstrom unit (Å).

Gaussian-09 (revision D.01) program package was used for all the calculations [1]. Geometries for all the considered reactants, intermediates and transition sates were optimized at M06/6-31+G(d,p) level of theory. Frequency calculations characterize the obtained stationary points as minima structures or transition states on the potential energy surface. Intrinsic reaction coordinate (IRC) calculations were also performed to connect the obtained transition states with their reactants and products.

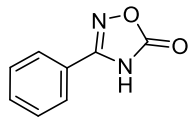
1. References

Gaussian 09, Revision D.01, Frisch, M. J.; Trucks, G. W.; Schlegel, H. B.; Scuseria, G. E.; Robb, M. A.; Cheeseman, J. R.; Scalmani, G.; Barone, V.; Mennucci, B.; Petersson, G. A.; Nakatsuji, H.; Caricato, M.; Li, X.; Hratchian, H. P.; Izmaylov, A. F.; Bloino, J.; Zheng, G.; Sonnenberg, J. L.; Hada, M.; Ehara, M.; Toyota, K.; Fukuda, R.; Hasegawa, J.; Ishida, M.; Nakajima, T.; Honda, Y.; Kitao, O.; Nakai, H.; Vreven, T.; Montgomery, J. A.; Peralta, Jr., J.

E.; Ogliaro, F.; Bearpark, M.; Heyd, J. J.; Brothers, E.; Kudin, K. N.; Staroverov, V. N.; Keith, T.; Kobayashi, R.; Normand, J.; Raghavachari, K.; Rendell, A.; Burant, J. C.; Iyengar, S. S.; Tomasi, J.; Cossi, M.; Rega, N.; Millam, J. M.; Klene, M.; Knox, J. E.; Cross, J. B.; Bakken, V.; Adamo, C.; Jaramillo, J.; Gomperts, R. ; Stratmann, R. E. ; Yazyev, O.; Austin, A. J.; Cammi, R.; Pomelli, C.; Ochterski, J. W.; Martin, R. L.; Morokuma, K.; Zakrzewski, V. G.; Voth, G. A.; Salvador, P.; Dannenberg, J. J.; Dapprich, S.; Daniels, A. D.; Farkas, O.; Foresman, J. B.; Ortiz, J. V.; Cioslowski, J.; J. Fox, D. Gaussian, Inc., Wallingford CT, **2013**.

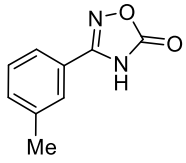
Spectral Data

*3-Phenyl-1,2,4-oxadiazol-5(4H)-one (**1a**):*



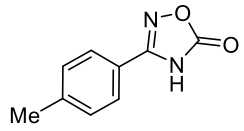
Yield: 67% (27 mg) as a white solid; ^{1}H NMR (DMSO-d_6, 600 MHz): δ 13.01 (s, 1H), 7.81 (d, 2H, J = 7.2 Hz), 7.63 (t, 1H, J = 7.2 Hz), 7.58 (t, 2H, J = 7.5 Hz); ^{13}C NMR (DMSO-d_6, 150 MHz): δ 159.9, 157.4, 132.3, 129.3, 126.1, 123.3; IR (KBr, cm^{-1}): 3128, 3049, 1762, 1741, 1608, 1551, 1462, 1257; HRMS (ESI/Q-TOF) (m/z): calcd for $C_8H_7N_2O_2$, $[M+H]^+$: 163.0502, found 163.0506.

*3-(m-Tolyl)-1,2,4-oxadiazol-5(4H)-one (**2a**):*



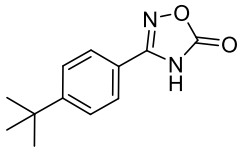
Yield: 64% (28 mg) as a white solid; ^{1}H NMR (DMSO-d_6, 600 MHz): δ 12.91 (s, 1H), 7.64 (s, 1H), 7.59 (d, 1H, J = 7.2 Hz), 7.47–7.43 (m, 2H), 2.37 (s, 3H); ^{13}C NMR (DMSO-d_6, 150 MHz): δ 159.9, 157.4, 138.8, 132.8, 129.2, 126.4, 123.3, 123.2, 20.9; IR (KBr, cm^{-1}): 3120, 3041, 1760, 1741, 1609, 1550, 1461, 1257; HRMS (ESI/Q-TOF) (m/z): calcd for $C_9H_9N_2O_2$, $[M+H]^+$: 177.0659, found 177.0654.

*3-(p-Tolyl)-1,2,4-oxadiazol-5(4H)-one (**3a**):*



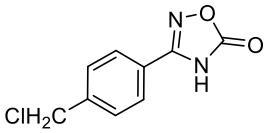
Yield: 66% (29 mg) as a white solid; ^{1}H NMR (DMSO-d_6, 600 MHz): δ 12.89 (s, 1H), 7.69 (d, 2H, J = 7.8 Hz), 7.39 (d, 2H, J = 8.4 Hz), 2.38 (s, 3H); ^{13}C NMR (DMSO-d_6, 150 MHz): δ 159.9, 157.3, 142.4, 129.8, 125.9, 120.5, 21.1; IR (KBr, cm^{-1}): 3127, 3049, 1761, 1745, 1609, 1552, 1461, 1254; HRMS (ESI/Q-TOF) (m/z): calcd for $C_9H_9N_2O_2$, $[M+H]^+$: 177.0659, found 177.0649.

*3-(4-(tert-Butyl)phenyl)-1,2,4-oxadiazol-5(4H)-one (**4a**):*



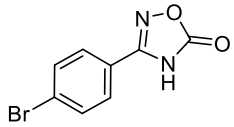
Yield: 69% (38 mg) as a white solid; ^{1}H NMR (DMSO-d_6, 600 MHz): δ 12.95 (s, 1H), 7.74 (d, 2H, J = 8.4 Hz), 7.59 (d, 2H, J = 8.4 Hz), 1.29 (s, 9H); ^{13}C NMR (DMSO-d_6, 150 MHz): δ 160.1, 157.3, 155.3, 126.2, 125.9,120.5, 34.9, 30.8; IR (KBr, cm^{-1}): 3126, 3041, 1761, 1745, 1607, 1550, 1461, 1255; HRMS (ESI/Q-TOF) (m/z): calcd for $C_{12}H_{15}N_2O_2$, $[M+H]^+$: 219.1128, found 219.1120.

*3-(4-(chloromethyl)phenyl)-1,2,4-oxadiazol-5(4H)-one (**5a**):*



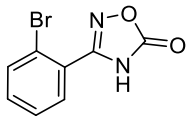
Yield: 68% (35 mg) as a white solid; ^{1}H NMR (DMSO-d_6, 400 MHz): δ 7.81 (d, 2H, J = 8.4 Hz), 7.63 (d, 2H, J = 8.0 Hz), 4.83 (s, 2H); ^{13}C NMR (DMSO-d_6, 100 MHz): δ 159.9, 157.1, 141.9, 129.7, 126.5, 123.2, 45.3; IR (KBr, cm^{-1}): 3124, 3045, 1760, 1741, 1610, 1555, 1475, 1251; HRMS (ESI/Q-TOF) (m/z): calcd for $C_9H_8ClN_2O_2$, $[M+H]^+$: 211.0269, found 211.0271.

*3-(4-Bromophenyl)-1,2,4-oxadiazol-5(4H)-one (**6a**):*



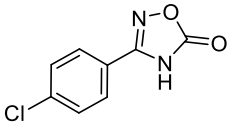
Yield: 71% (42 mg) as a white solid; ^{1}H NMR (DMSO-d_6, 400 MHz): δ 7.81 (d, 2H, J = 8.8 Hz), 7.74 (d, 2H, J = 8.4 Hz); ^{13}C NMR (DMSO-d_6, 100 MHz): δ 159.9, 156.8, 132.4, 127.9, 125.9, 122.6; IR (KBr, cm^{-1}): 3127, 3045, 1761, 1745, 1607, 1555, 1461, 1261; HRMS (ESI/Q-TOF) (m/z): calcd for $C_8H_6BrN_2O_2$, $[M+H]^+$: 240.9607, found 240.9611.

*3-(2-Bromophenyl)-1,2,4-oxadiazol-5(4H)-one (**7a**):*



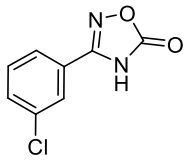
Yield: 65% (38 mg) as a white solid; ^{1}H NMR (DMSO-d_6, 400 MHz): δ 12.92 (s, 1H), 7.87–7.85 (m, 1H), 7.70–7.68 (m, 1H), 7.59–7.57 (m, 2H); ^{13}C NMR (DMSO-d_6, 100 MHz): δ 159.5, 157.5, 133.61, 133.57, 131.8, 128.2, 125.2, 121.5; IR (KBr, cm^{-1}): 3130, 3050, 1760, 1745, 1609, 1553, 1460, 1257; HRMS (ESI/Q-TOF) (m/z): calcd for $C_8H_6BrN_2O_2$, $[M+H]^+$: 240.9607, found 240.9601.

*3-(4-Chlorophenyl)-1,2,4-oxadiazol-5(4H)-one (**8a**):*



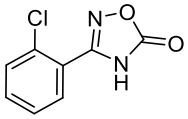
Yield: 74% (36 mg) as a white solid; ^{1}H NMR (DMSO-d_6, 400 MHz): δ 13.06 (s, 1H), 7.82 (d, 2H, J = 8.4 Hz), 7.67 (d, 2H, J = 8.8 Hz); ^{13}C NMR (DMSO-d_6, 100 MHz): δ 159.8, 156.6, 136.9, 129.5, 127.8, 122.2; IR (KBr, cm^{-1}): 3129, 3049, 1761, 1741, 1608, 1551, 1462, 1259; HRMS (ESI/Q-TOF) (m/z): calcd for $C_8H_6ClN_2O_2$, $[M+H]^+$: 197.0112, found 197.0110.

*3-(3-Chlorophenyl)-1,2,4-oxadiazol-5(4H)-one (**9a**):*



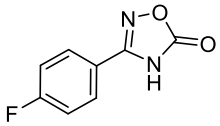
Yield: 69% (34 mg) as a white solid; ^{1}H NMR (DMSO-d_6, 400 MHz): δ 7.85–7.84 (m, 1H), 7.79–7.76 (m, 1H), 7.72–7.69 (m, 1H), 7.63–7.59 (m, 1H); ^{13}C NMR (DMSO-d_6, 100 MHz): δ 159.8, 156.4, 133.9, 132.0, 131.3, 125.9, 125.3, 124.7; IR (KBr, cm^{-1}): 3128, 3049, 1762, 1741, 1608, 1551, 1462, 1257; HRMS (ESI/Q-TOF) (m/z): calcd for $C_8H_6ClN_2O_2$, $[M+H]^+$: 197.0112, found 197.0119.

*3-(2-Chlorophenyl)-1,2,4-oxadiazol-5(4H)-one (**10a**):*



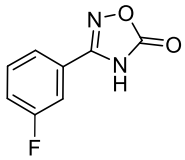
Yield: 67% (32 mg) as a white solid; ^{1}H NMR (DMSO-d_6, 400 MHz): δ 12.85 (s, 1H), 7.73–7.69 (m, 2H), 7.68–7.64 (m, 1H), 7.57–7.53 (m, 1H); ^{13}C NMR (DMSO-d_6, 100 MHz): δ 159.6, 156.4, 133.5, 132.0, 131.4, 130.5, 127.8, 122.9; IR (KBr, cm^{-1}): 3130, 3046, 1762, 1745, 1608, 1552, 1462, 1259; HRMS (ESI/Q-TOF) (m/z): calcd for $C_8H_6ClN_2O_2$, $[M+H]^+$: 197.0112, found 197.0122.

*3-(4-Fluorophenyl)-1,2,4-oxadiazol-5(4H)-one (**11a**):*



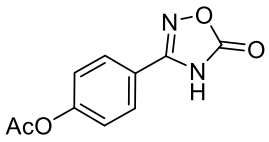
Yield: 75% (34 mg) as a white solid; ^{1}H NMR (DMSO-d_6, 400 MHz): δ 7.89–7.85 (m, 2H), 7.44 (t, 2H, J = 8.8 Hz); ^{13}C NMR (DMSO-d_6, 100 MHz): δ 165.4, 162.9, 159.9, 156.6, 128.8 (d, J = 9.1 Hz), 119.9 (d, J = 3.2 Hz), 116.6 (d, J = 22.2 Hz); ^{19}F NMR (DMSO-d_6 + Hexafluorobenzene): δ -109.4 (s); IR (KBr, cm^{-1}): 3128, 3049, 1761, 1741, 1611, 1551, 1462, 1255; HRMS (ESI/Q-TOF) (m/z): calcd for $C_8H_6FN_2O_2$, $[M+H]^+$: 181.0408, found 181.0405.

*3-(3-Fluorophenyl)-1,2,4-oxadiazol-5(4H)-one (**12a**):*



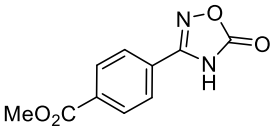
Yield: 74% (33 mg) as a white solid; ^{1}H NMR (DMSO-d_6, 400 MHz): δ 7.68-7.59 (m, 3H), 7.52-7.46 (m, 1H); ^{13}C NMR (DMSO-d_6, 100 MHz): δ 163.4, 160.9, 159.9, 156.6 (d, J = 2.9 Hz), 131.8 (d, J = 8.3 Hz), 125.4 (d, J = 8.6 Hz), 122.4 (d, J = 3.1 Hz), 119.2 (d, J = 20.9 Hz), 113.1 (d, J = 24.2 Hz); ^{19}F NMR (DMSO-d_6 + Hexafluorobenzene): δ -113.6 (s); IR (KBr, cm^{-1}): 3126, 3039, 1758, 1745, 1610, 1555, 1470, 1255; HRMS (ESI/Q-TOF) (m/z): calcd for $C_8H_6FN_2O_2$, $[M+H]^+$: 181.0408, found 181.0410.

*4-(5-Oxo-4,5-dihydro-1,2,4-oxadiazol-3-yl)phenyl acetate (**13a**):*



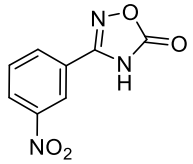
Yield: 79% (43 mg) as a white solid; ^{1}H NMR (DMSO-d_6, 400 MHz): δ 12.99 (s, 1H), 7.85 (d, 2H, J = 8.4 Hz), 7.37 (d, 2H, J = 8.8 Hz), 2.30 (s, 3H); ^{13}C NMR (DMSO-d_6, 100 MHz): δ 168.9, 159.9, 156.9, 153.2, 127.6, 122.9, 120.9, 20.9; IR (KBr, cm^{-1}): 3128, 3048, 1762, 1741, 1609, 1551, 1461, 1257; HRMS (ESI/Q-TOF) (m/z): calcd for $C_{10}H_9N_2O_4$, $[M+H]^+$: 221.0557, found 221.0553.

*Methyl 4-(5-oxo-4,5-dihydro-1,2,4-oxadiazol-3-yl)benzoate (**14a**):*



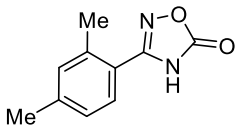
Yield: 77% (42 mg) as a white solid; ^{1}H NMR (DMSO-d_6, 400 MHz): δ 13.15 (s, 1H), 8.13 (d, 2H, J = 8.0 Hz), 7.95 (d, 2H, J = 8.0 Hz), 3.89 (s, 3H); ^{13}C NMR (DMSO-d_6, 100 MHz): δ 165.4, 159.9, 156.8, 132.6, 129.9, 127.5, 126.5, 52.5; IR (KBr, cm^{-1}): 3125, 3049, 1765, 1741, 1611, 1551, 1462, 1255; HRMS (ESI/Q-TOF) (m/z): calcd for $C_{10}H_9N_2O_4$, $[M+H]^+$: 221.0557, found 221.0550.

*3-(3-Nitrophenyl)-1,2,4-oxadiazol-5(4H)-one (**15a**):*



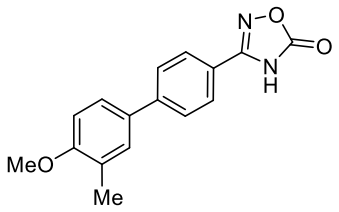
Yield: 75% (39 mg) as a white solid; ^{1}H NMR (DMSO-d_6, 600 MHz): δ 8.62 (s, 1H), 8.46–8.44 (m, 1H), 8.23–8.22 (m, 1H), 7.87 (t, 1H, J = 8.1 Hz); ^{13}C NMR (DMSO-d_6, 100 MHz): δ 159.9, 156.3, 148.2, 132.2, 131.2, 126.7, 125.0, 121.1; IR (KBr, cm^{-1}): 3128, 3049, 1766, 1748, 1608, 1551, 1468, 1257; HRMS (ESI/Q-TOF) (m/z): calcd for $C_8H_6N_3O_4$, $[M+H]^+$: 208.0353, found 208.0357.

*3-(2,4-Dimethylphenyl)-1,2,4-oxadiazol-5(4H)-one (**16a**):*



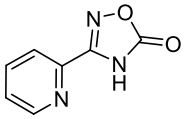
Yield: 62% (29 mg) as a white solid; ^{1}H NMR (DMSO-d_6, 400 MHz): δ 12.70 (s, 1H), 7.46 (d, 1H, J = 7.6 Hz), 7.23 (s, 1H), 7.19 (d, 1H, J = 7.6 Hz), 2.42 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (DMSO-d_6, 100 MHz): δ 159.7, 157.9, 141.4, 137.1, 132.1, 128.8, 126.8, 119.9, 20.8, 20.6; IR (KBr, cm^{-1}): 3131, 3051, 1768, 1745, 1609, 1551, 1467, 1257; HRMS (ESI/Q-TOF) (m/z): calcd for $C_{10}H_{11}N_2O_2$, $[M+H]^+$: 191.0815, found 191.0810.

*3-(4'-Methoxy-3'-methyl-[1,1'-biphenyl]-4-yl)-1,2,4-oxadiazol-5(4H)-one (**17a**):*



Yield: 81% (57 mg) as a white solid; ^{1}H NMR (DMSO-d_6, 400 MHz): δ 12.92 (s, 1H), 7.97 (d, 2H, J = 8.4 Hz), 7.74 (d, 2H, J = 8.4 Hz), 7.57–7.55 (m, 2H), 7.04 (d, 1H, J = 9.2 Hz), 3.83 (s, 3H), 2.22 (s, 3H); 13C NMR (DMSO-d_6, 150 MHz): δ 167.3, 157.7, 144.2, 130.8, 129.9, 128.9, 128.7, 126.3, 126.1, 125.6, 110.8, 55.4, 16.2; IR (KBr, cm^{-1}): 3128, 3051, 1762, 1748, 1633, 1608, 1551, 1526, 1462, 1257; HRMS (ESI/Q-TOF) (m/z): calcd for $C_{16}H_{15}N_2O_3$, $[M+H]^+$: 283.1077, found 283.1075.

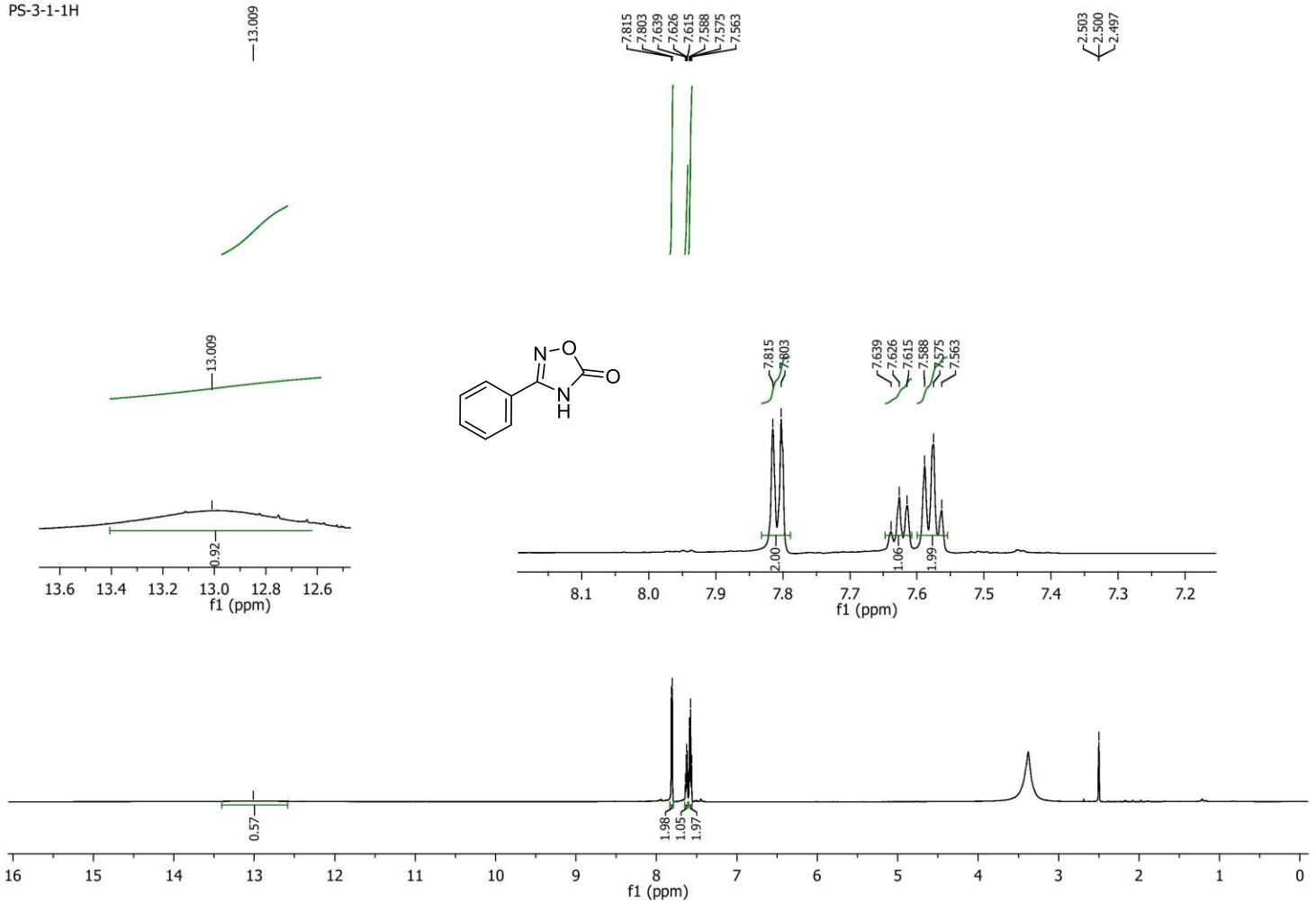
*3-(Pyridin-2-yl)-1,2,4-oxadiazol-5(4H)-one (**18a**):*



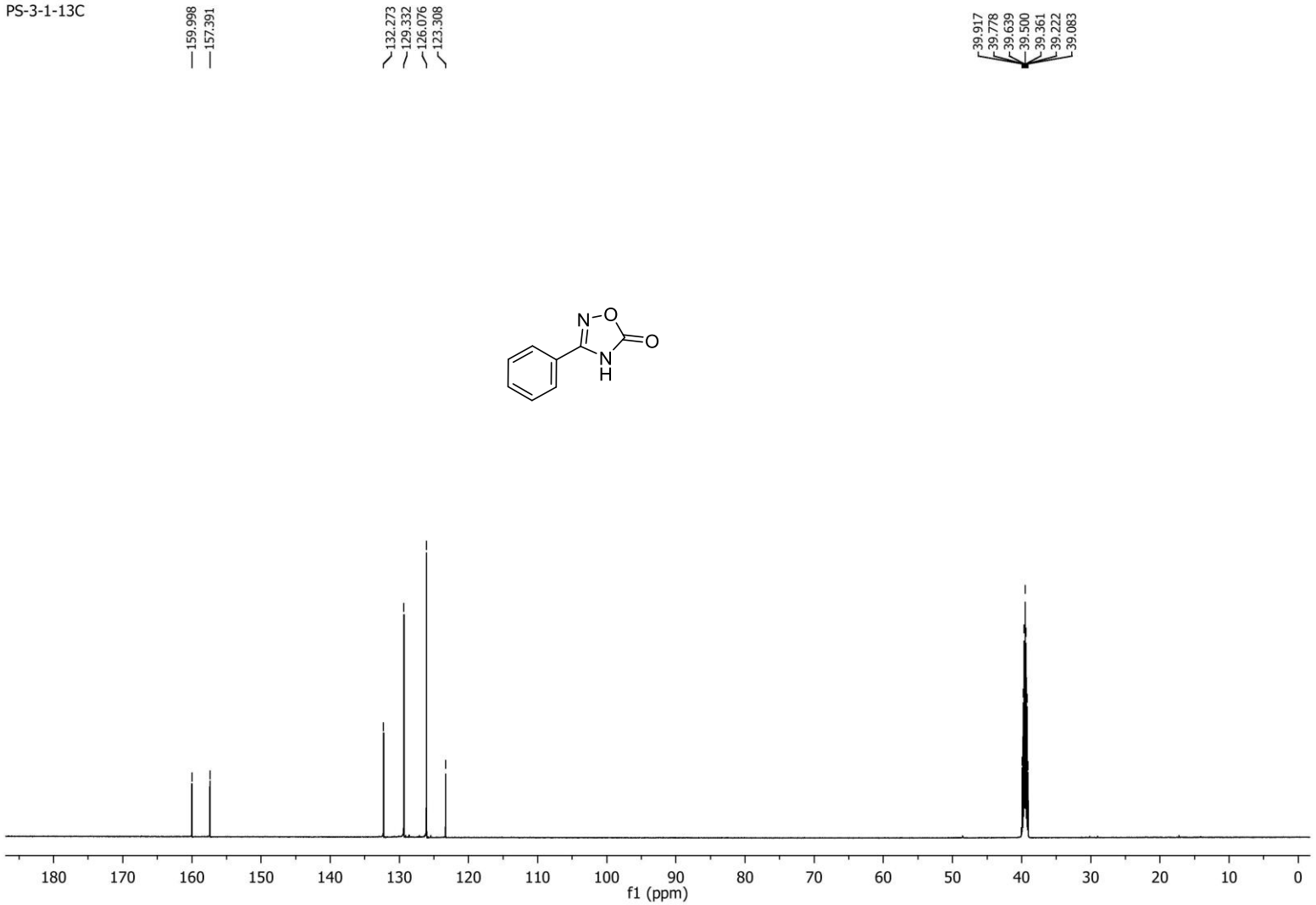
Yield: 73% (30 mg) as a white solid; ^{1}H NMR (DMSO-d_6, 400 MHz): δ 13.17 (s, 1H), 8.77–8.76 (m, 1H), 8.07–7.98 (m, 2H), 7.67–7.64 (m, 1H); ^{13}C NMR (DMSO-d_6, 100 MHz): δ 159.7, 157.7, 149.9, 142.7, 137.9, 126.8, 121.5; IR (KBr, cm^{-1}): 3128, 3051, 1768, 1741, 1611, 1551, 1462, 1257; HRMS (ESI/Q-TOF) (m/z): calcd for $C_7H_6N_3O_2$, $[M+H]^+$: 164.0455, found 164.0455.

Spectra

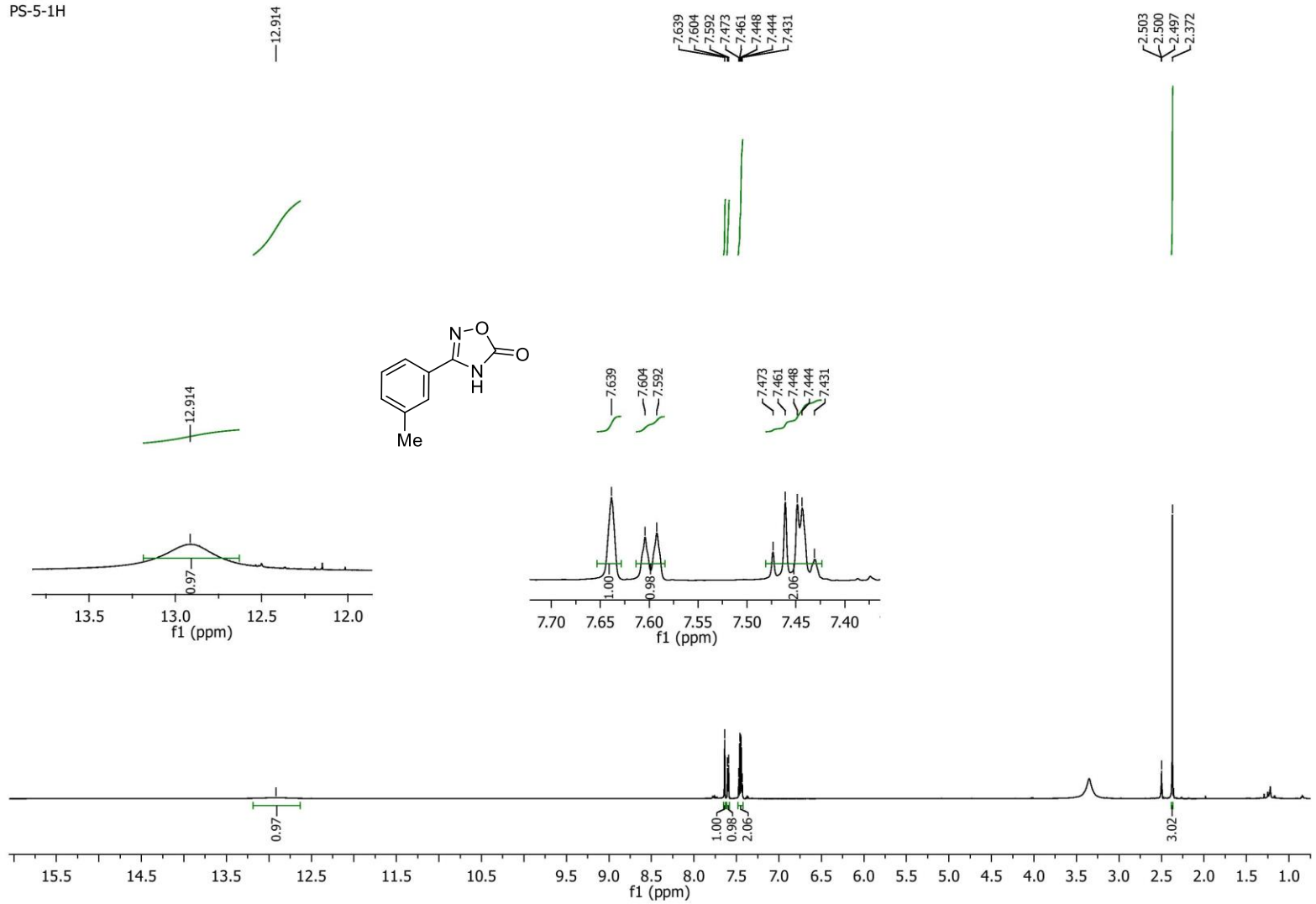
3-Phenyl-1,2,4-oxadiazol-5(4*H*)-one (1a): ^{1}H NMR (DMSO-d_6, 600 MHz)



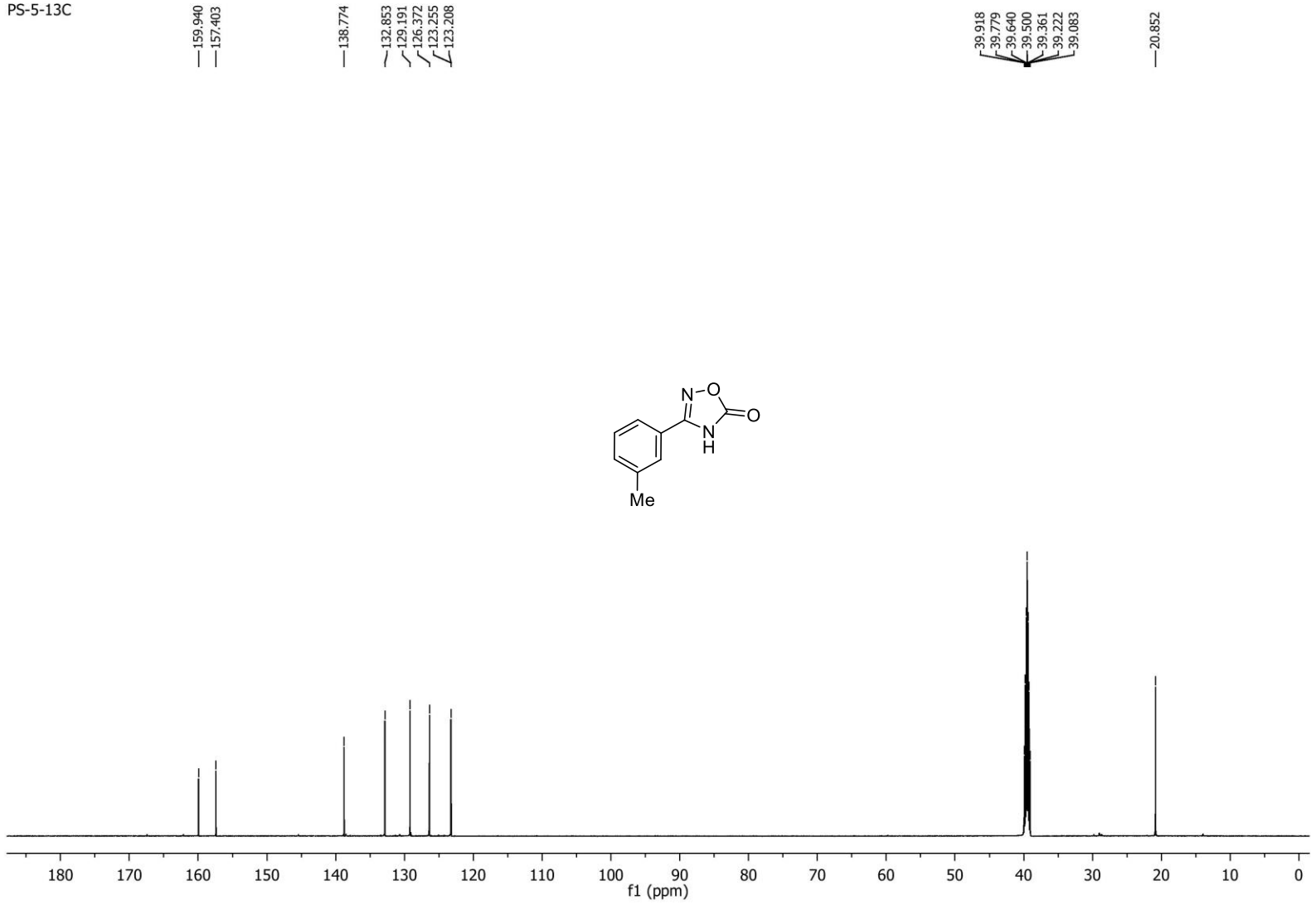
3-Phenyl-1,2,4-oxadiazol-5(4*H*)-one (1a): ^{13}C NMR (DMSO-d_6, 150 MHz)



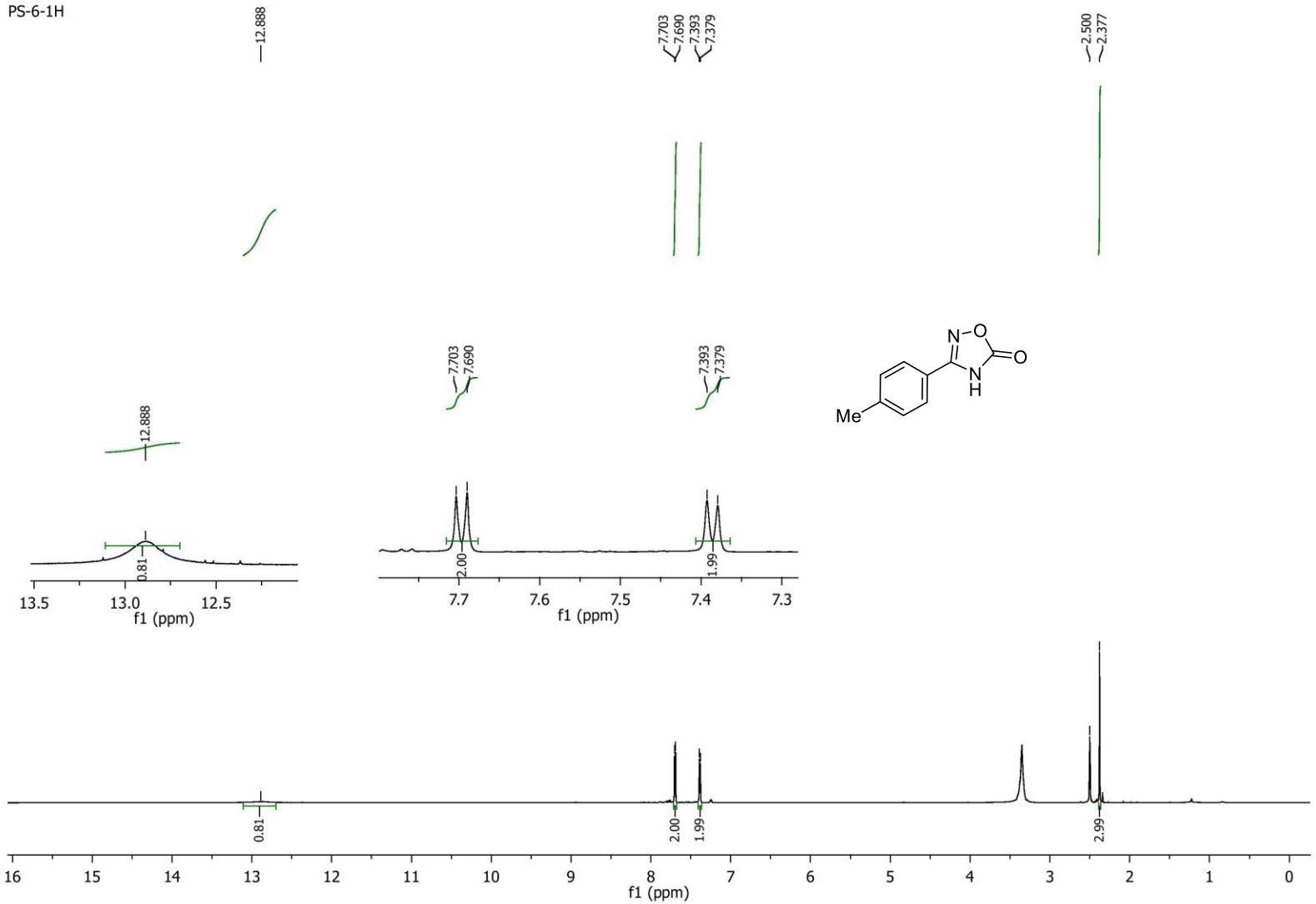
3-(*m*-Tolyl)-1,2,4-oxadiazol-5(4*H*)-one (2a): ^{1}H NMR (DMSO-d_6, 600 MHz)



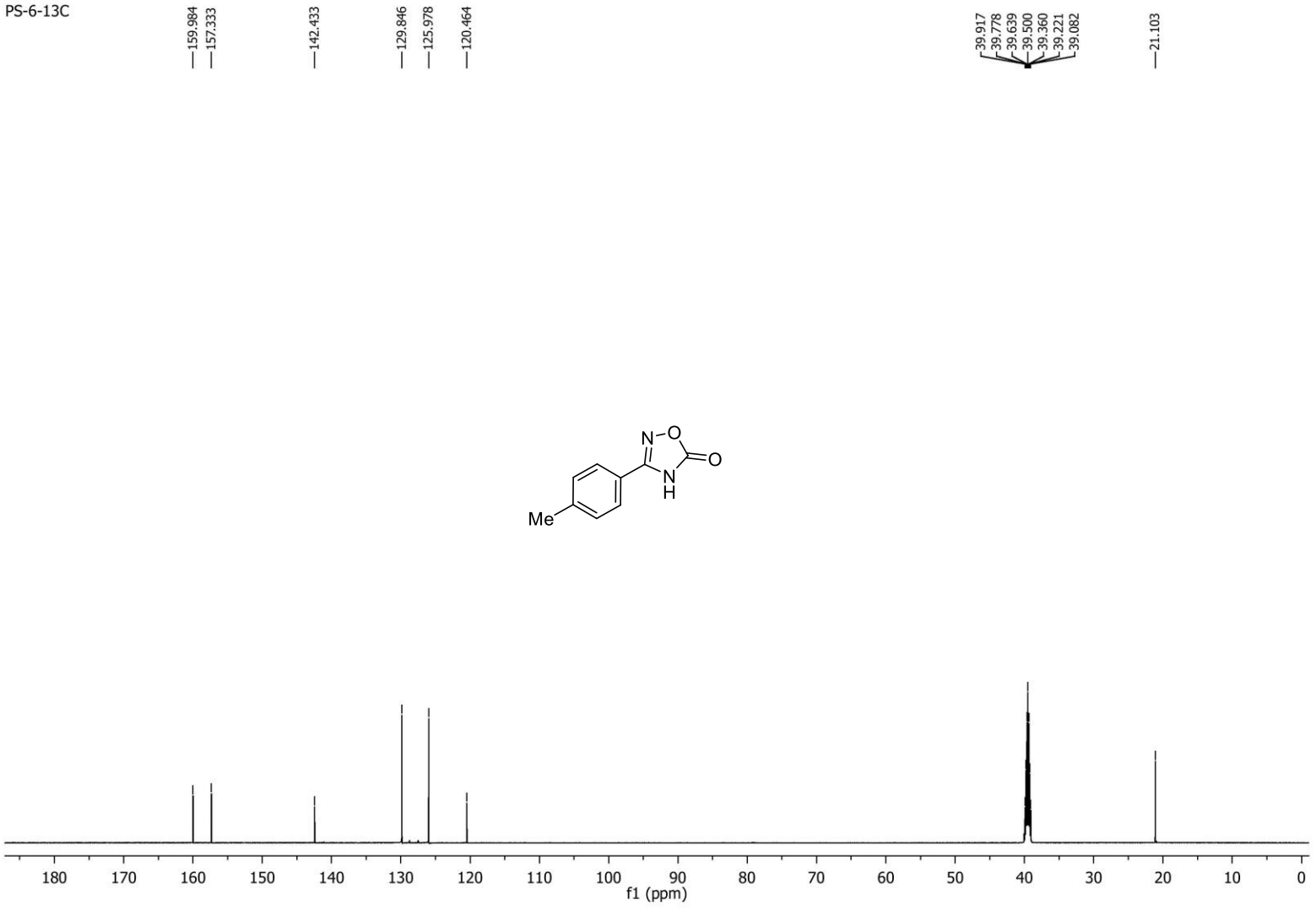
3-(*m*-Tolyl)-1,2,4-oxadiazol-5(4*H*)-one (2a): ^{13}C NMR (DMSO-*d*$_6$, 150 MHz)



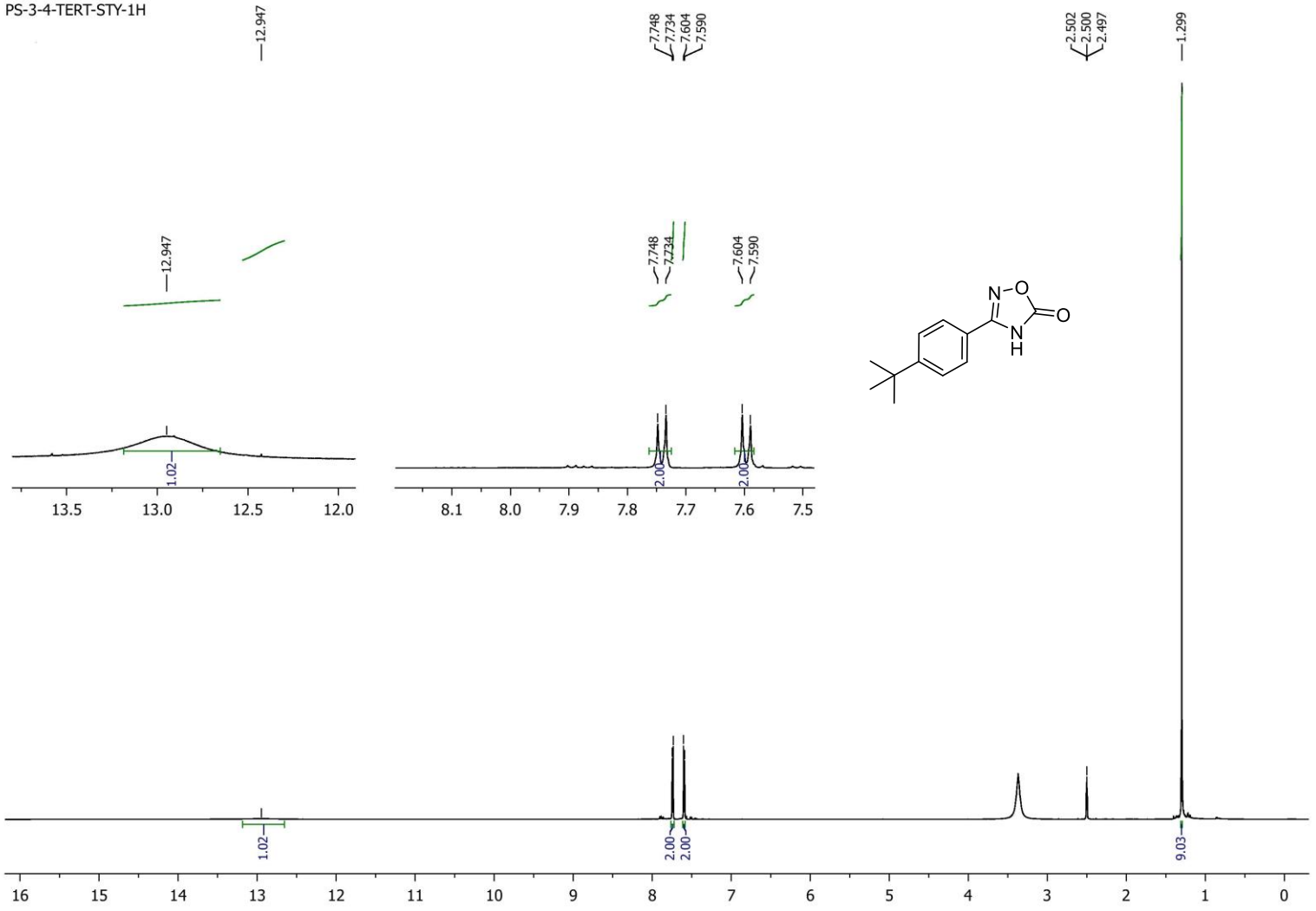
3-(*p*-Tolyl)-1,2,4-oxadiazol-5(4*H*)-one (3a): ^{1}H NMR (DMSO-d_6, 600 MHz)



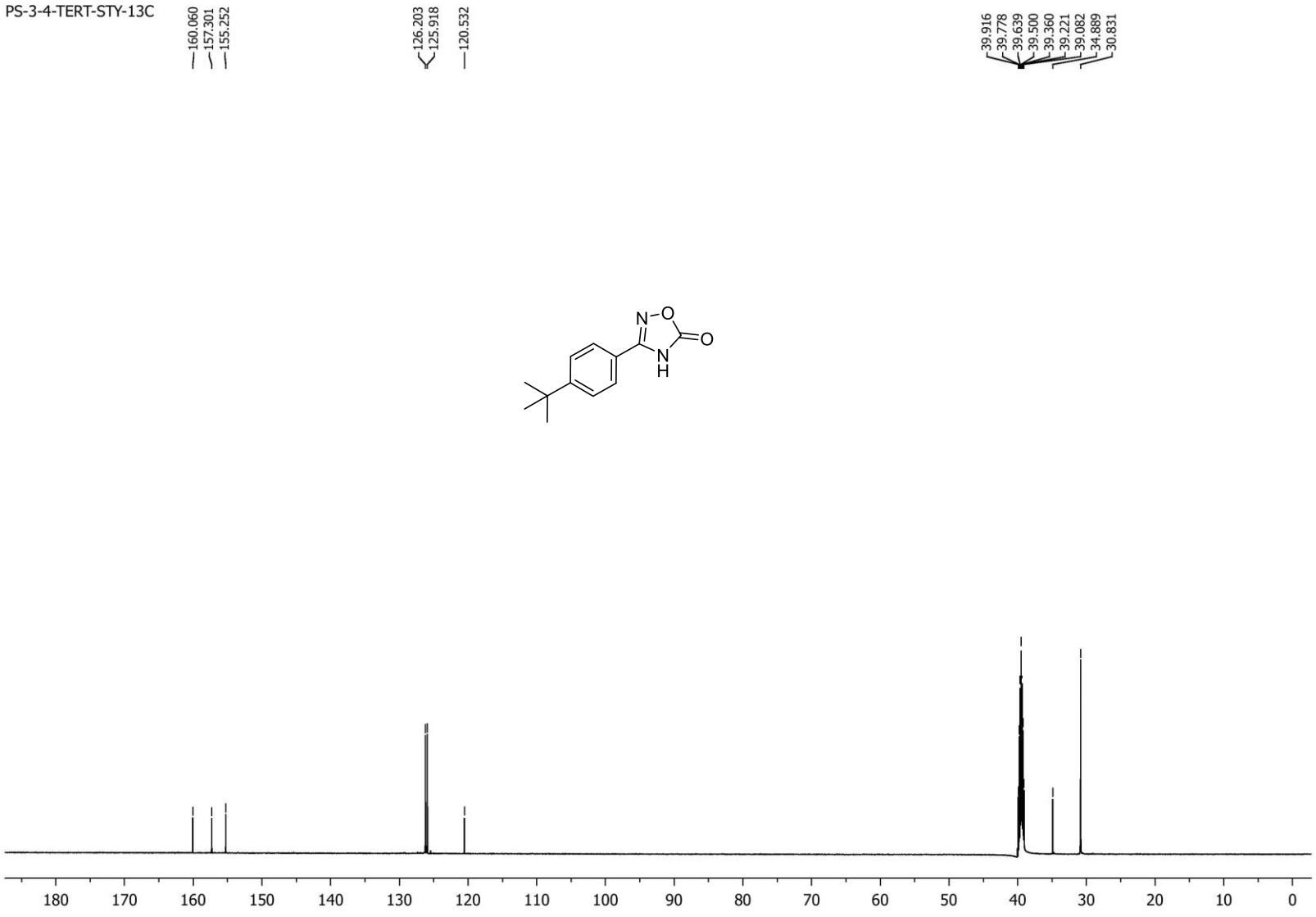
3-(*p*-Tolyl)-1,2,4-oxadiazol-5(4*H*)-one (3a): ^{13}C NMR (DMSO-d_6, 150 MHz)



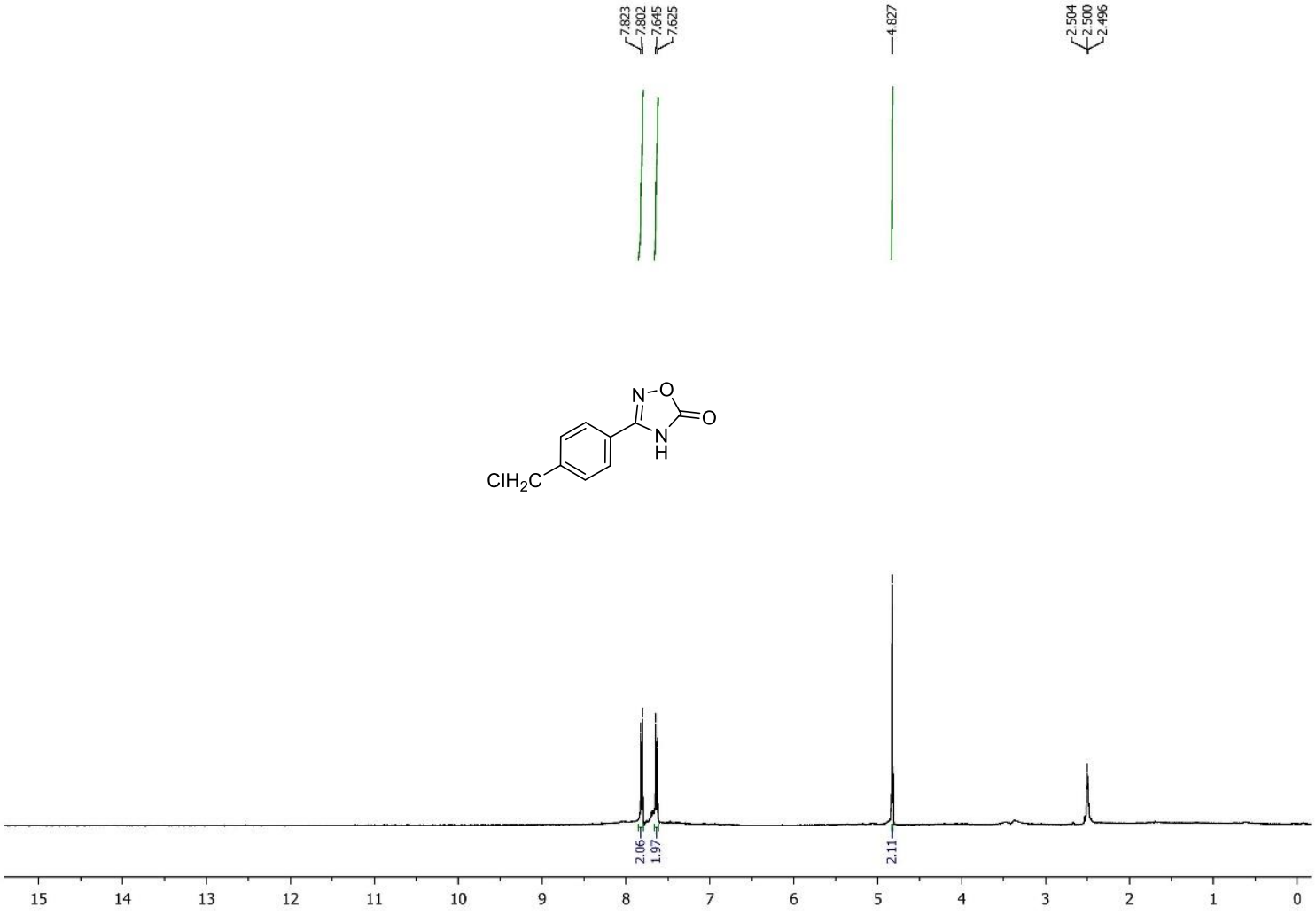
3-(4-(*tert*-Butyl)phenyl)-1,2,4-oxadiazol-5(4*H*)-one (4a): ^{1}H NMR (DMSO-d_6, 600 MHz)



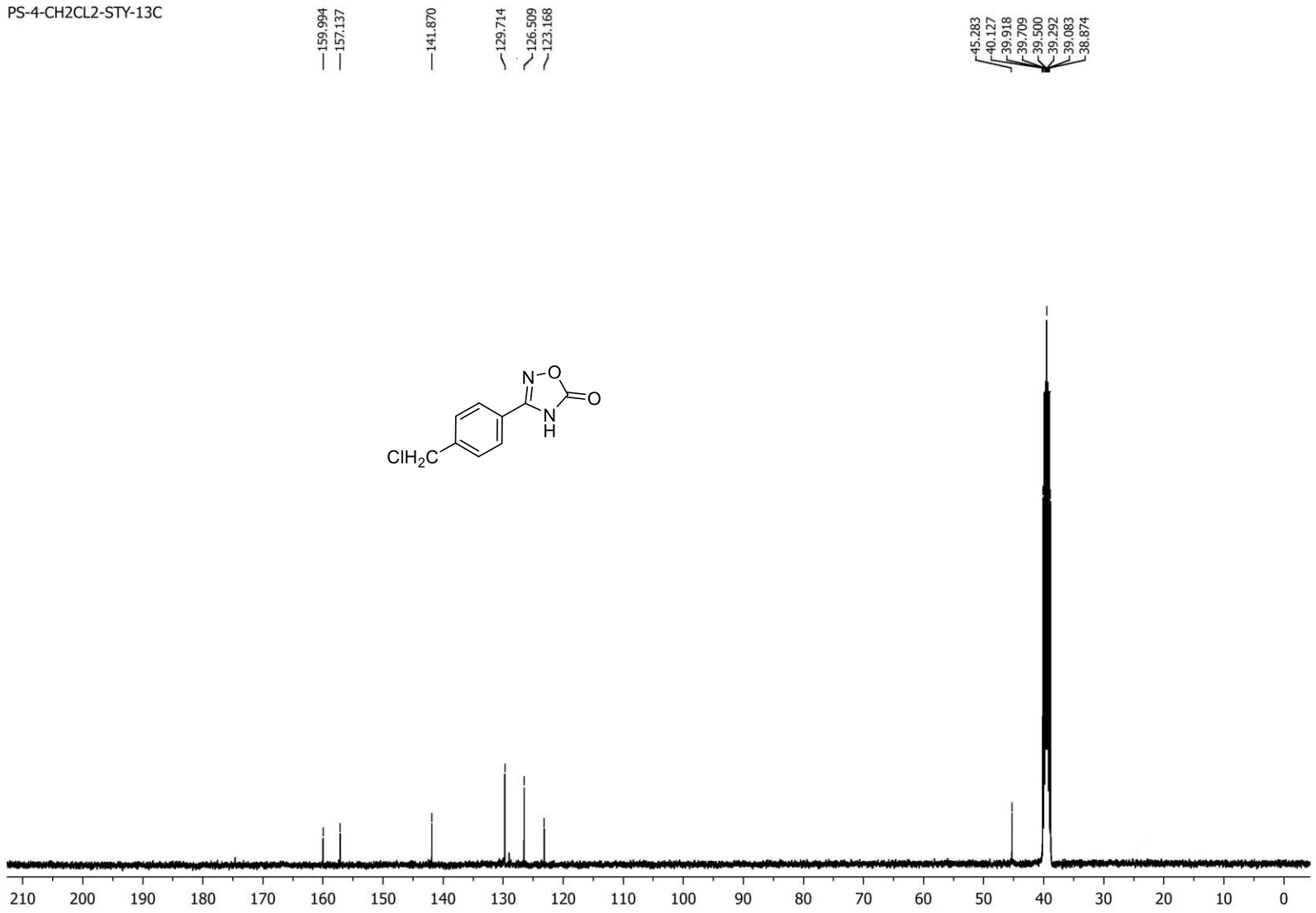
3-(4-(*tert*-Butyl)phenyl)-1,2,4-oxadiazol-5(4*H*)-one (4a): ^{13}C NMR (DMSO-d_6, 150 MHz)



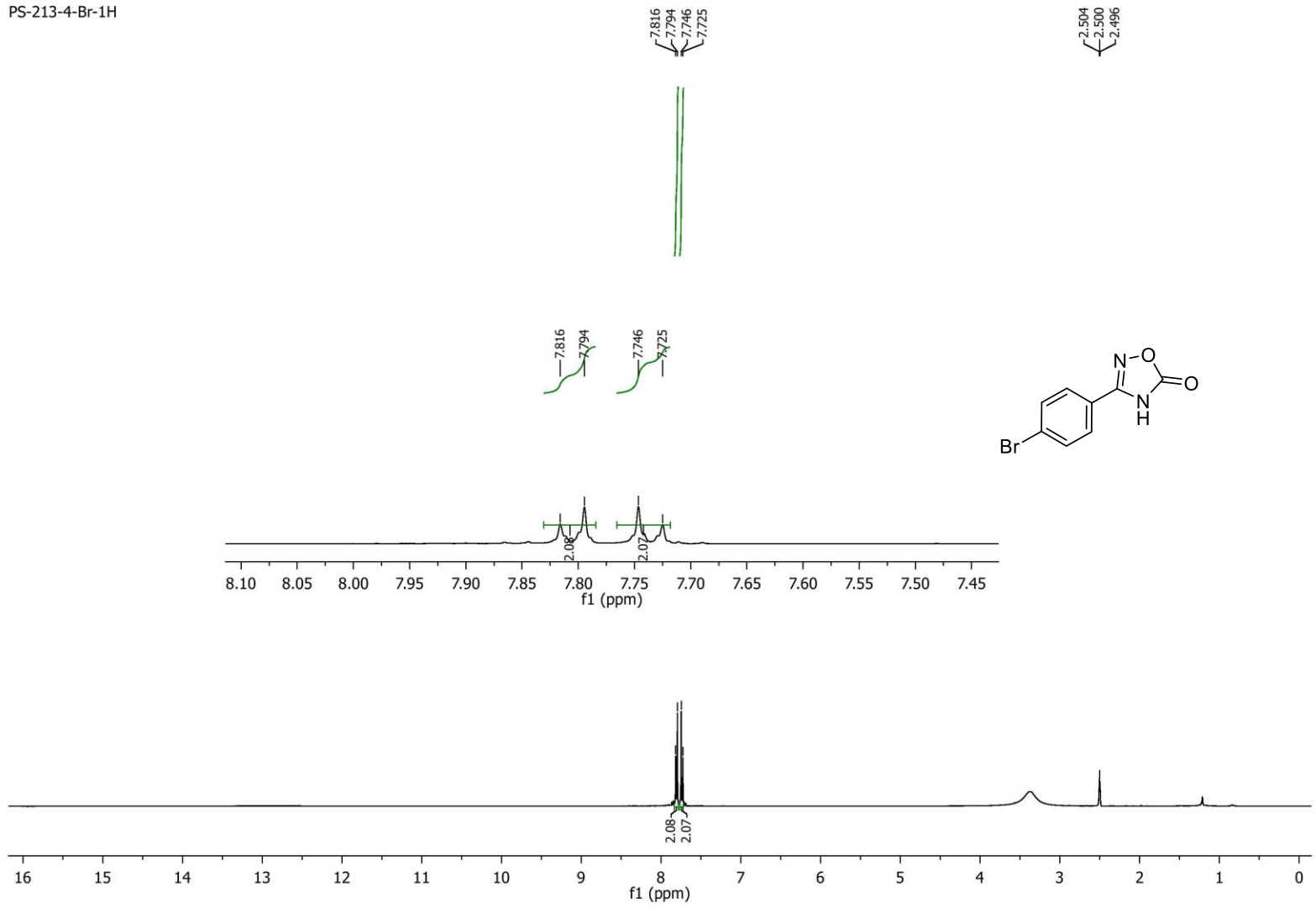
3-(4-(Chloromethyl)phenyl)-1,2,4-oxadiazol-5(4*H*)-one (5a*)*: ^{1}H NMR (DMSO-d_6, 400 MHz)



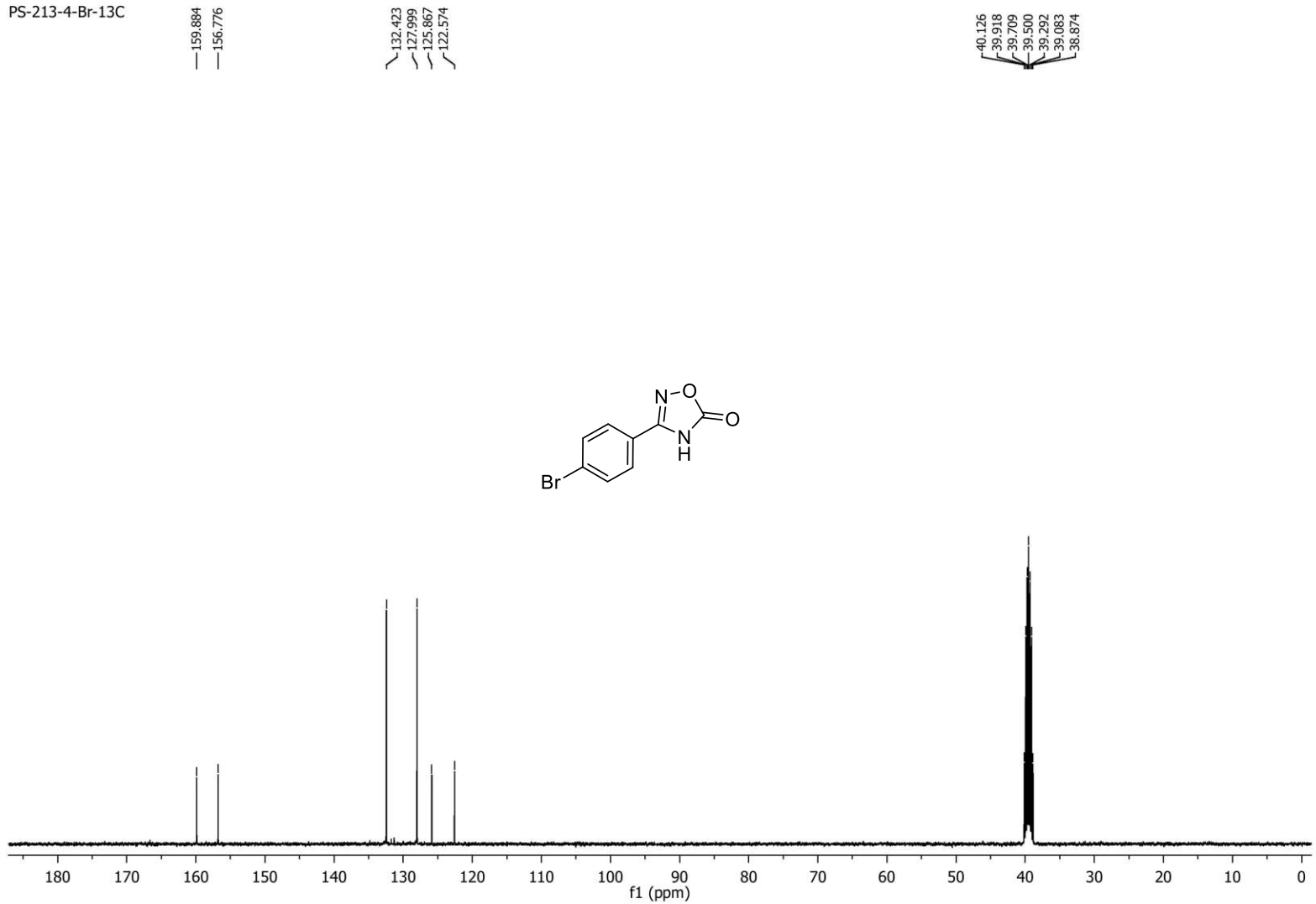
3-(4-(Chloromethyl)phenyl)-1,2,4-oxadiazol-5(4*H*)-one (5a): ^{13}C NMR (DMSO-*d*$_6$, 100 MHz)



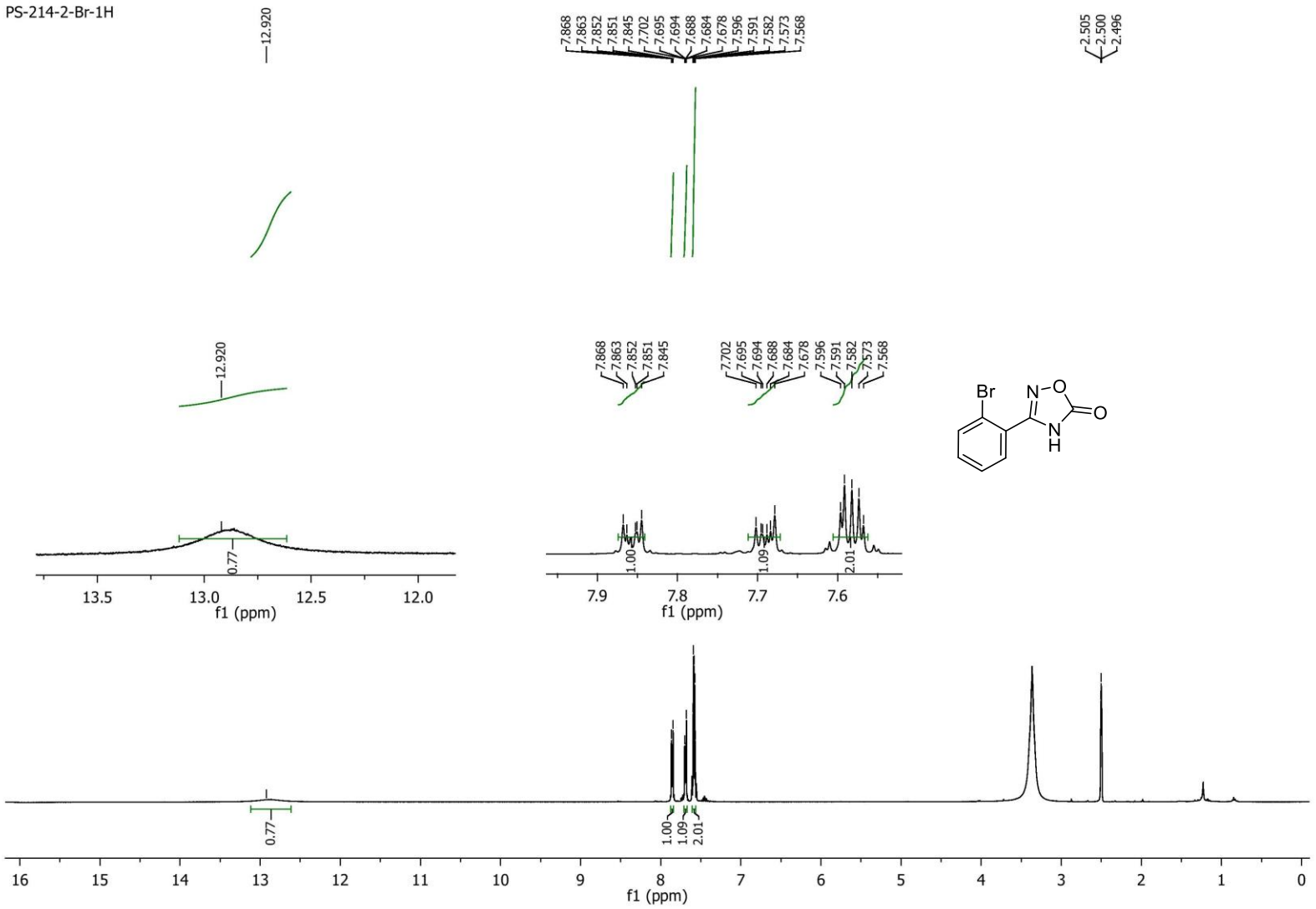
3-(4-Bromophenyl)-1,2,4-oxadiazol-5(4*H*)-one (6a): ^{1}H NMR (DMSO-*d*$_6$, 400 MHz)



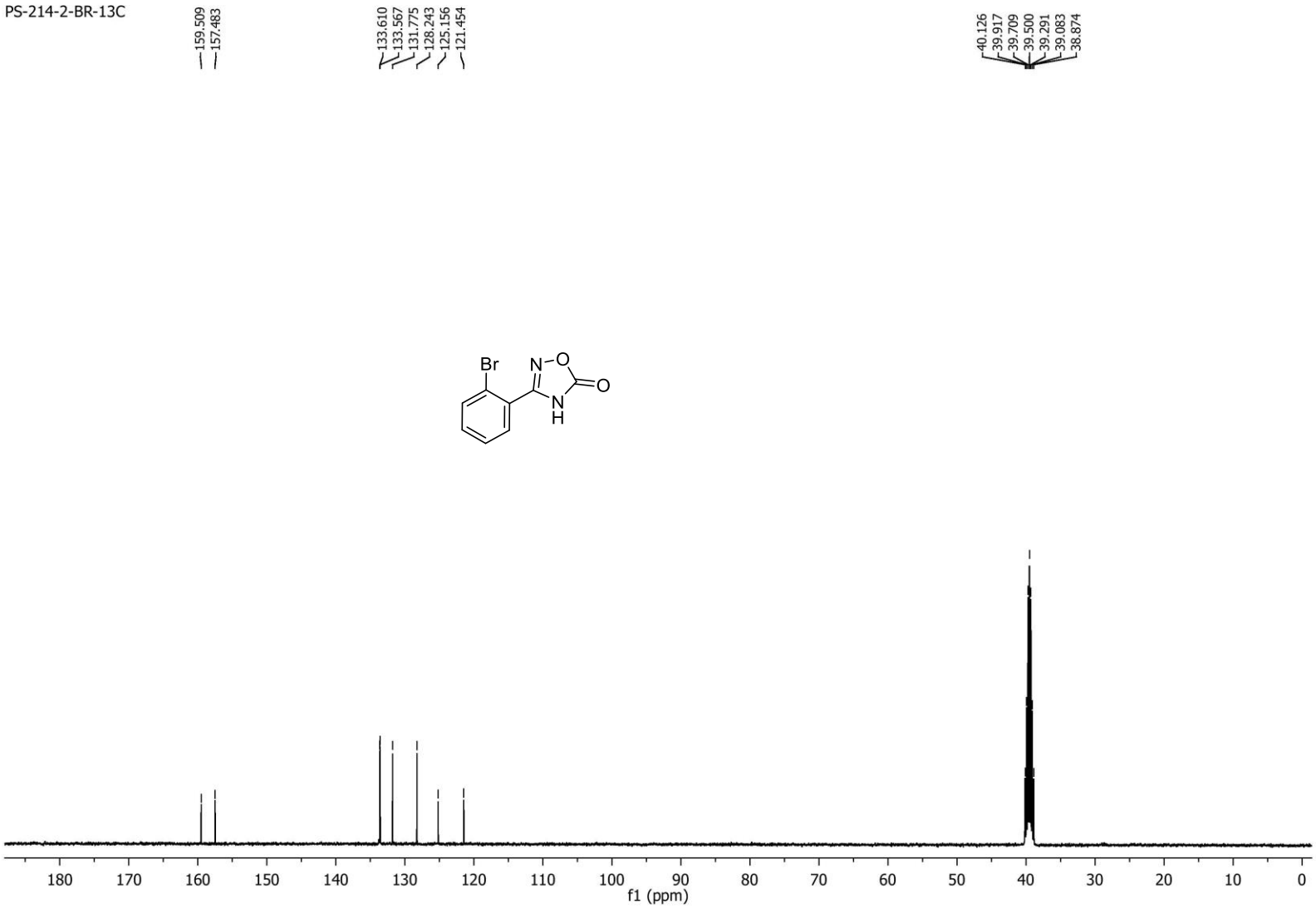
3-(4-Bromophenyl)-1,2,4-oxadiazol-5(4*H*)-one (6a): ^{13}C NMR (DMSO-d_6, 100 MHz)



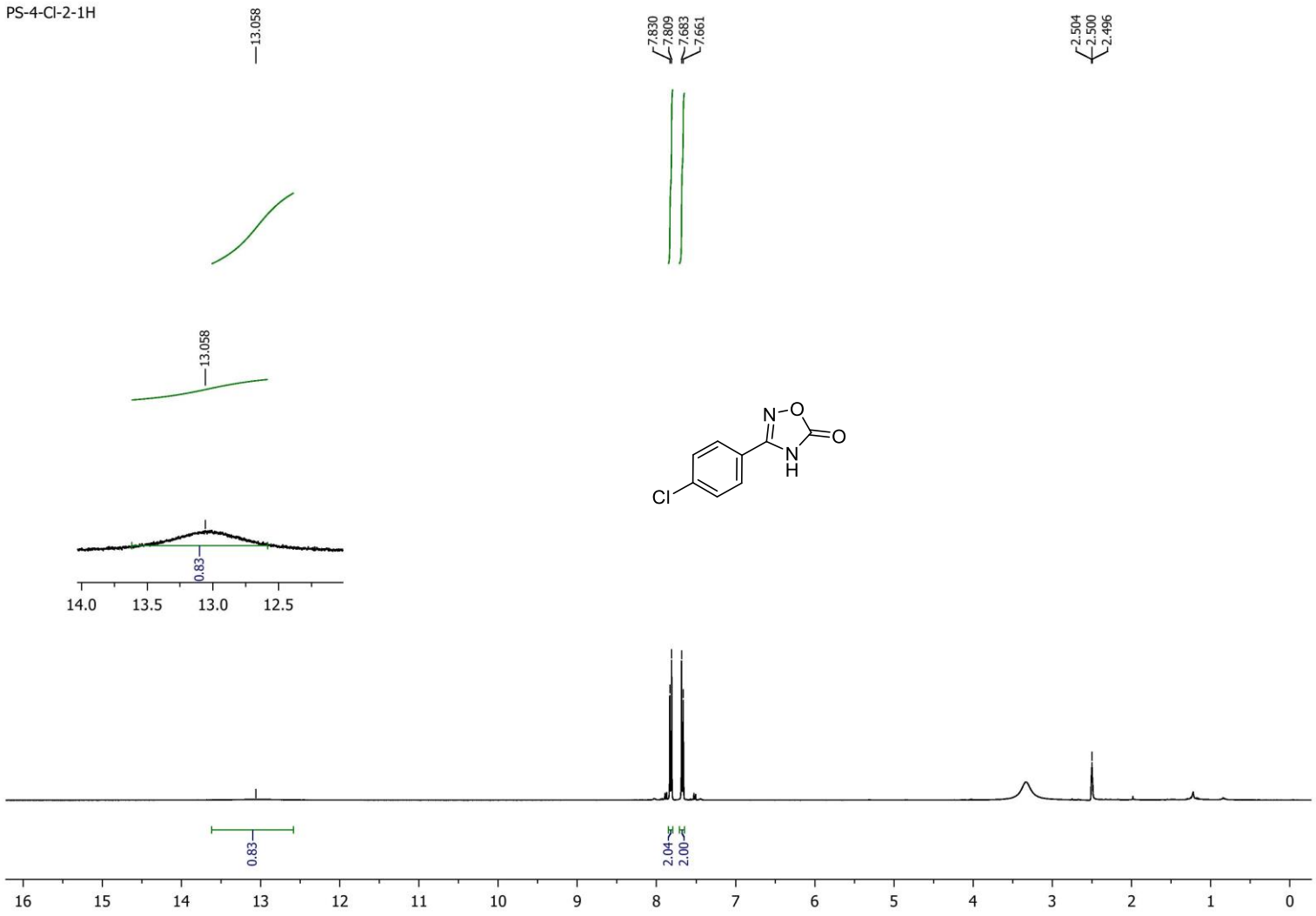
3-(2-Bromophenyl)-1,2,4-oxadiazol-5(4*H*)-one (7a): ^{1}H NMR (DMSO-*d*$_6$, 400 MHz)



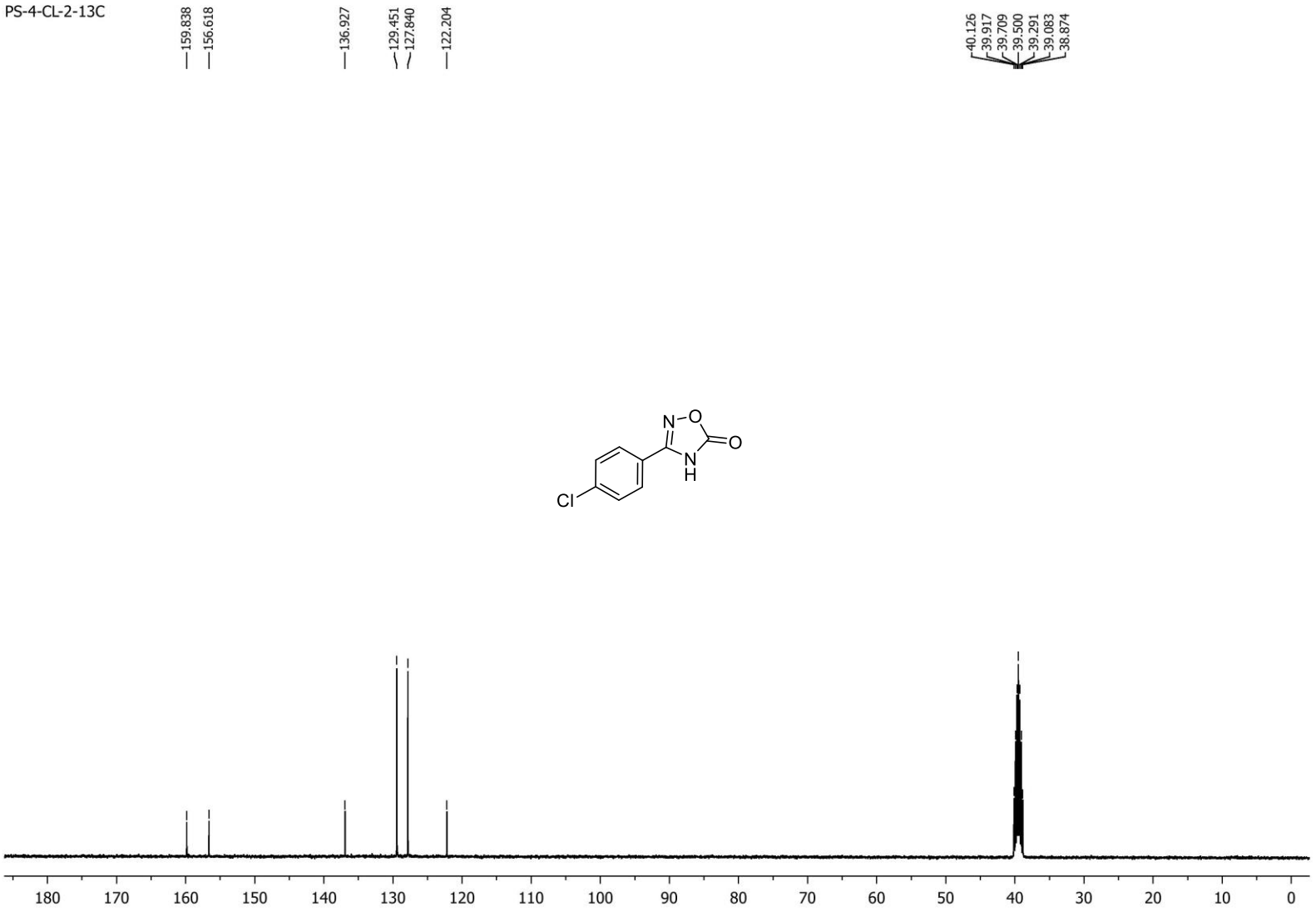
3-(2-Bromophenyl)-1,2,4-oxadiazol-5(4*H*)-one (7a): ^{13}C NMR (DMSO-d_6, 100 MHz)



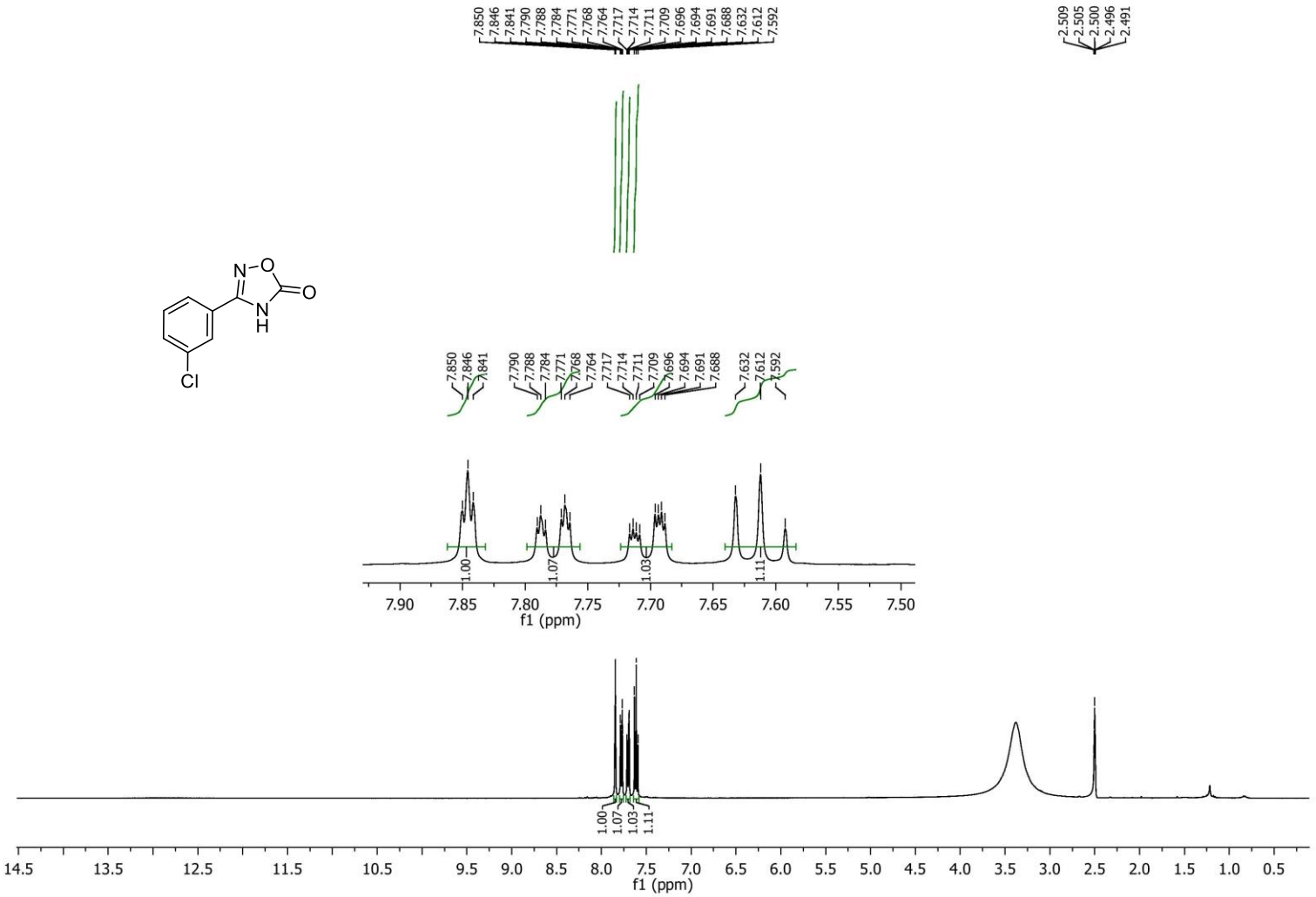
3-(4-Chlorophenyl)-1,2,4-oxadiazol-5(4*H*)-one (8a): ^{1}H NMR (DMSO-d_6, 400 MHz)



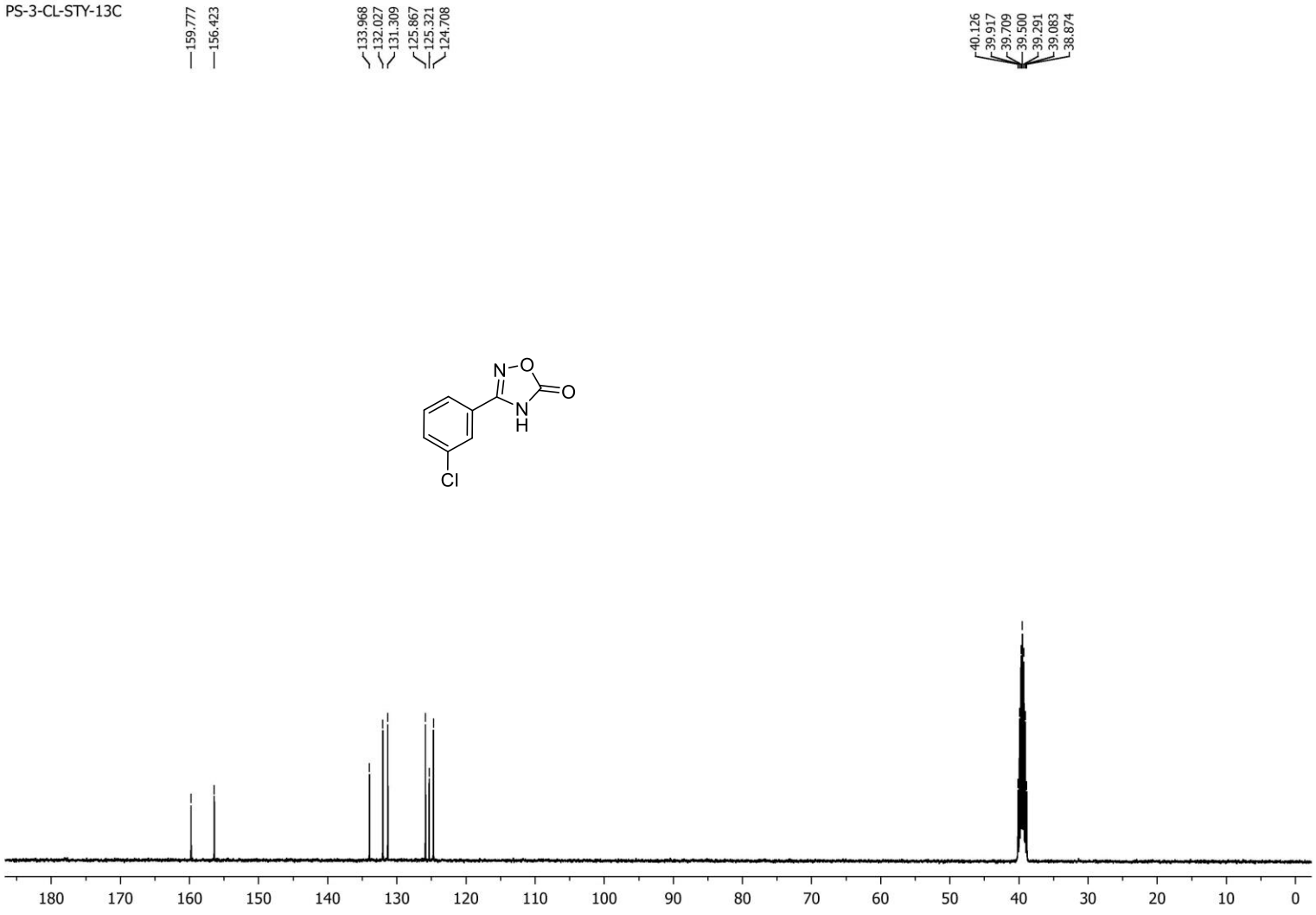
3-(4-Chlorophenyl)-1,2,4-oxadiazol-5(4*H*)-one (8a): ^{13}C NMR (DMSO-d_6, 100 MHz)



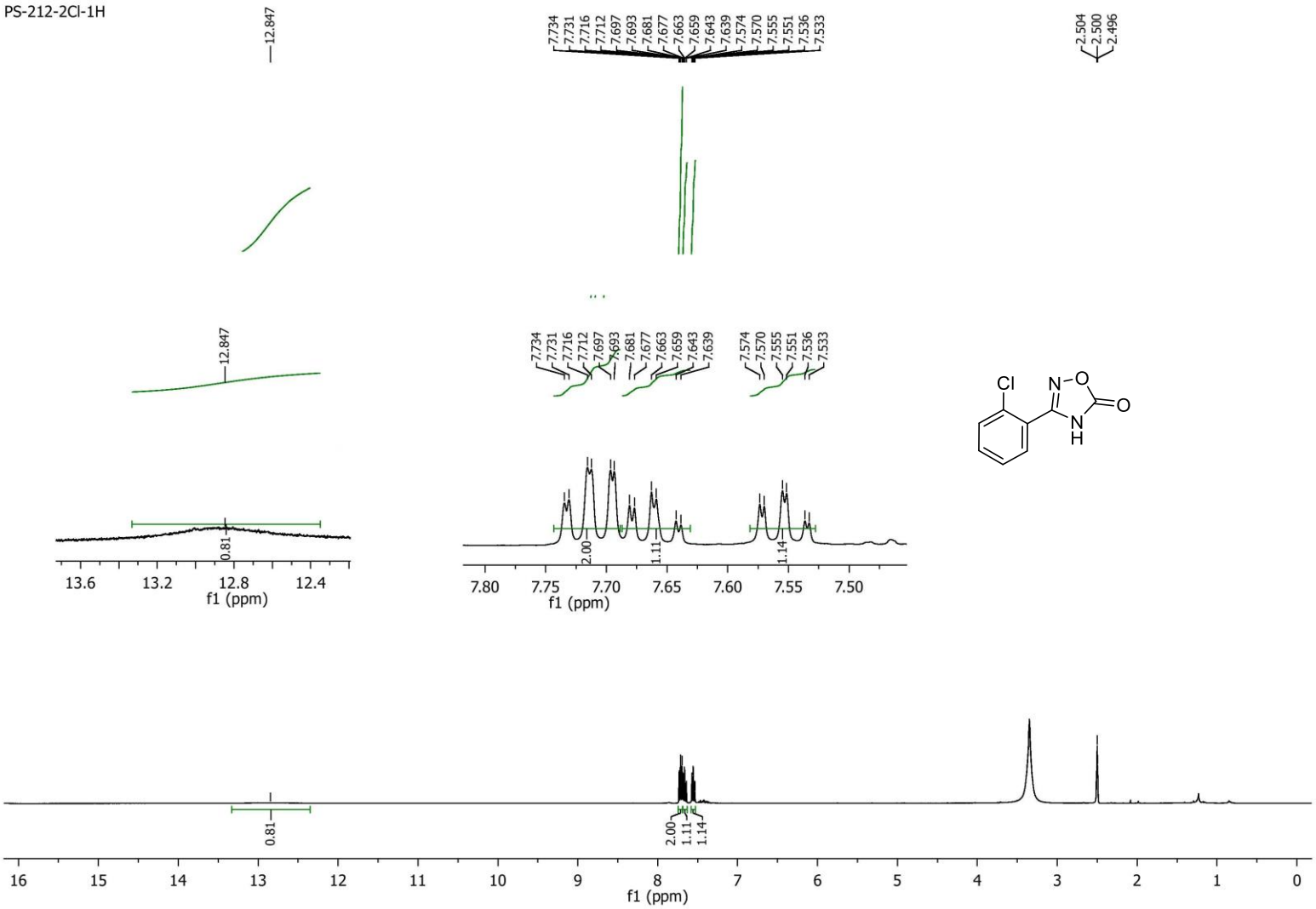
3-(3-Chlorophenyl)-1,2,4-oxadiazol-5(4*H*)-one (9a): ^{1}H NMR (DMSO-d_6, 400 MHz)



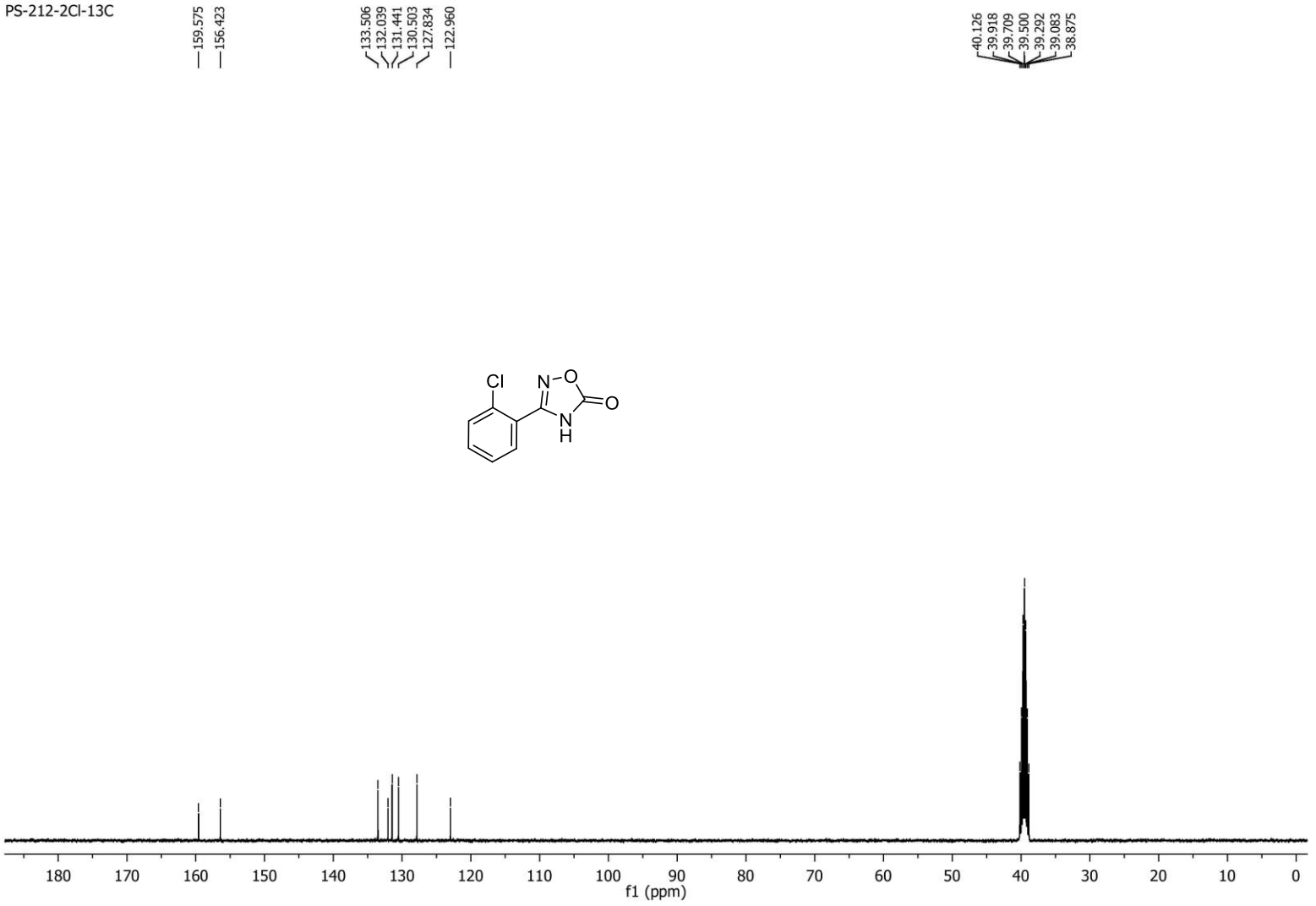
3-(3-Chlorophenyl)-1,2,4-oxadiazol-5(4*H*)-one (9a): ^{13}C NMR (DMSO-d_6, 100 MHz)



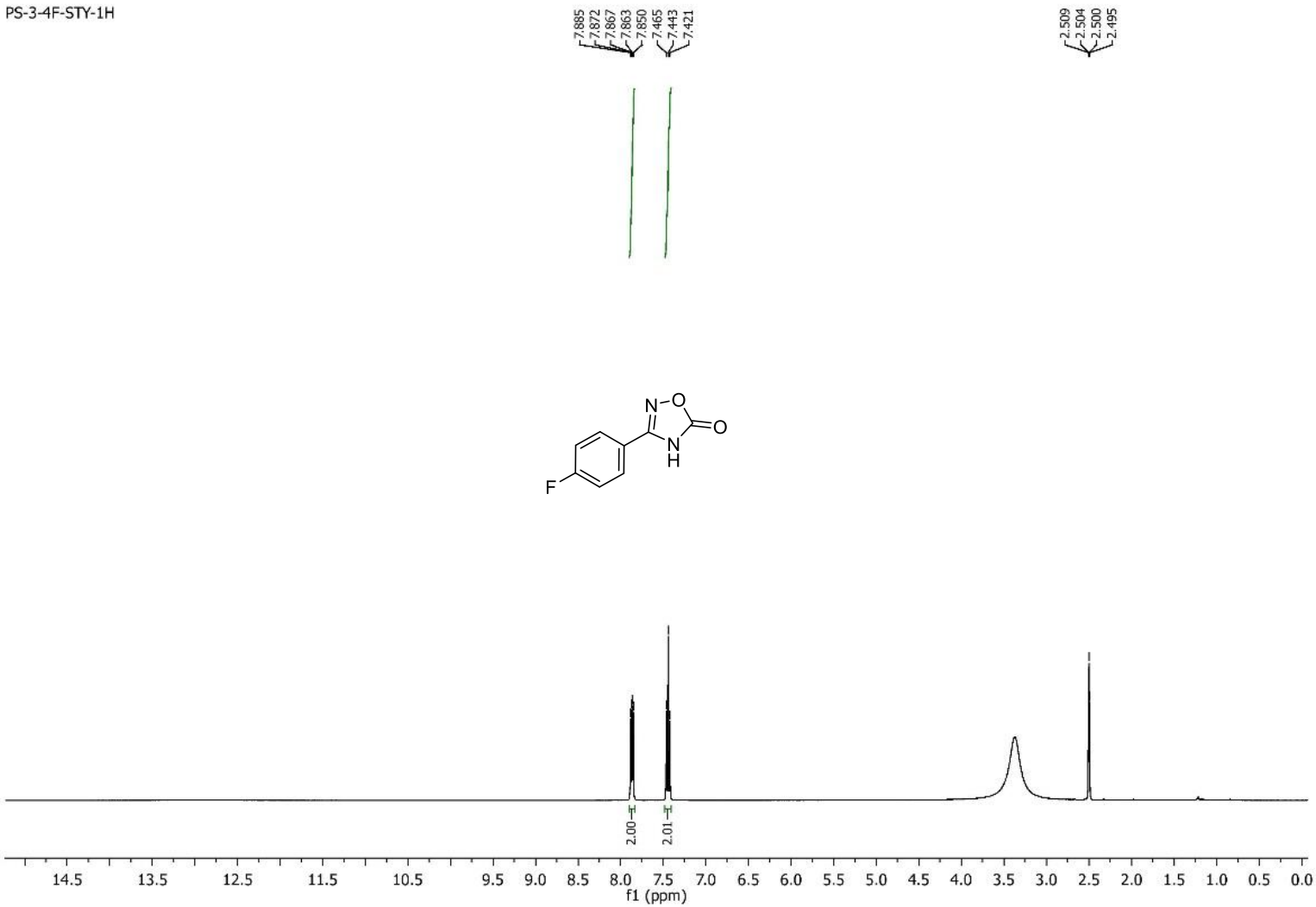
3-(2-Chlorophenyl)-1,2,4-oxadiazol-5(4*H*)-one (10a): ^{1}H NMR (DMSO-d_6, 400 MHz)



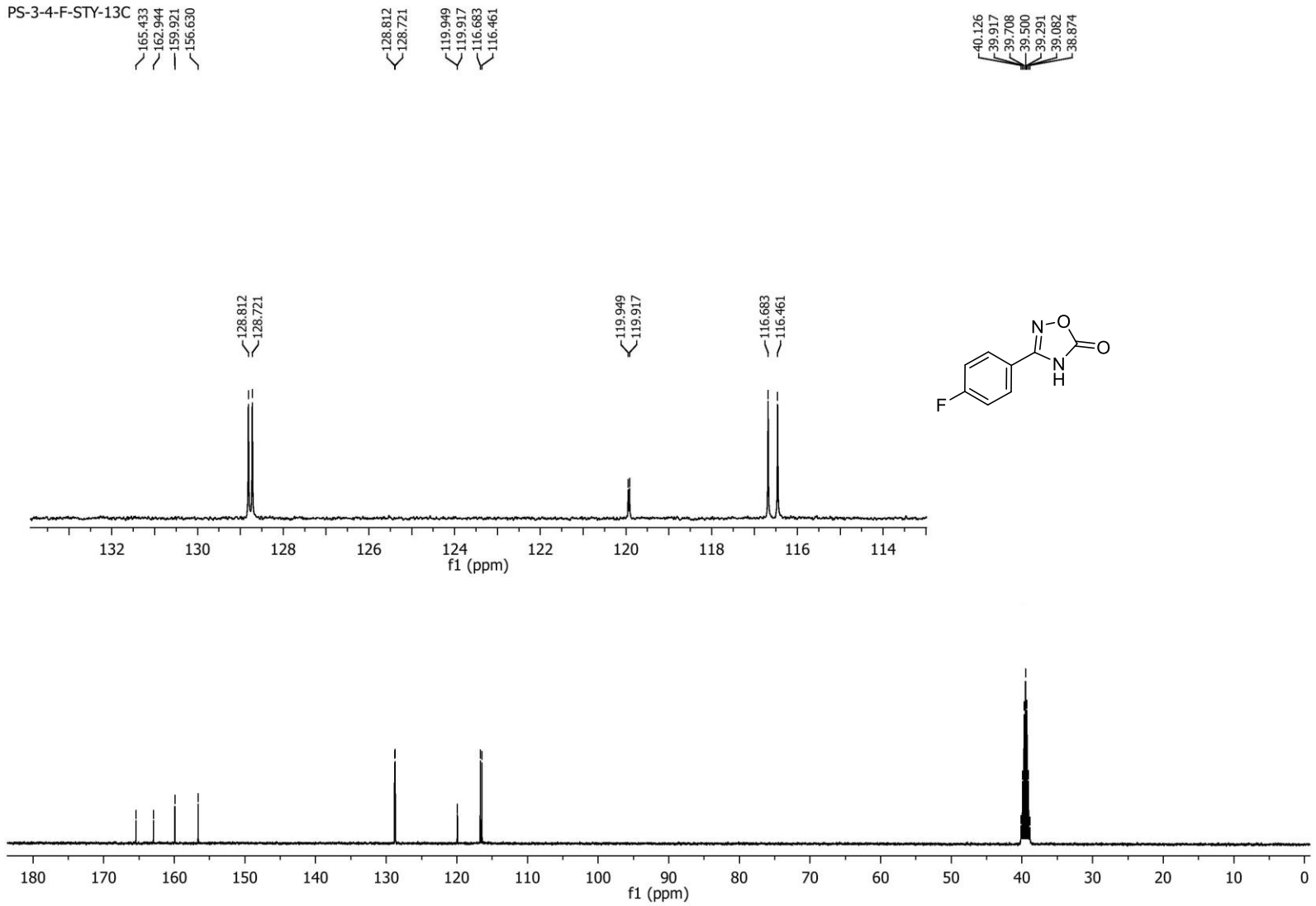
3-(2-Chlorophenyl)-1,2,4-oxadiazol-5(4*H*)-one (10a): ^{13}C NMR (DMSO-*d*$_6$, 100 MHz)



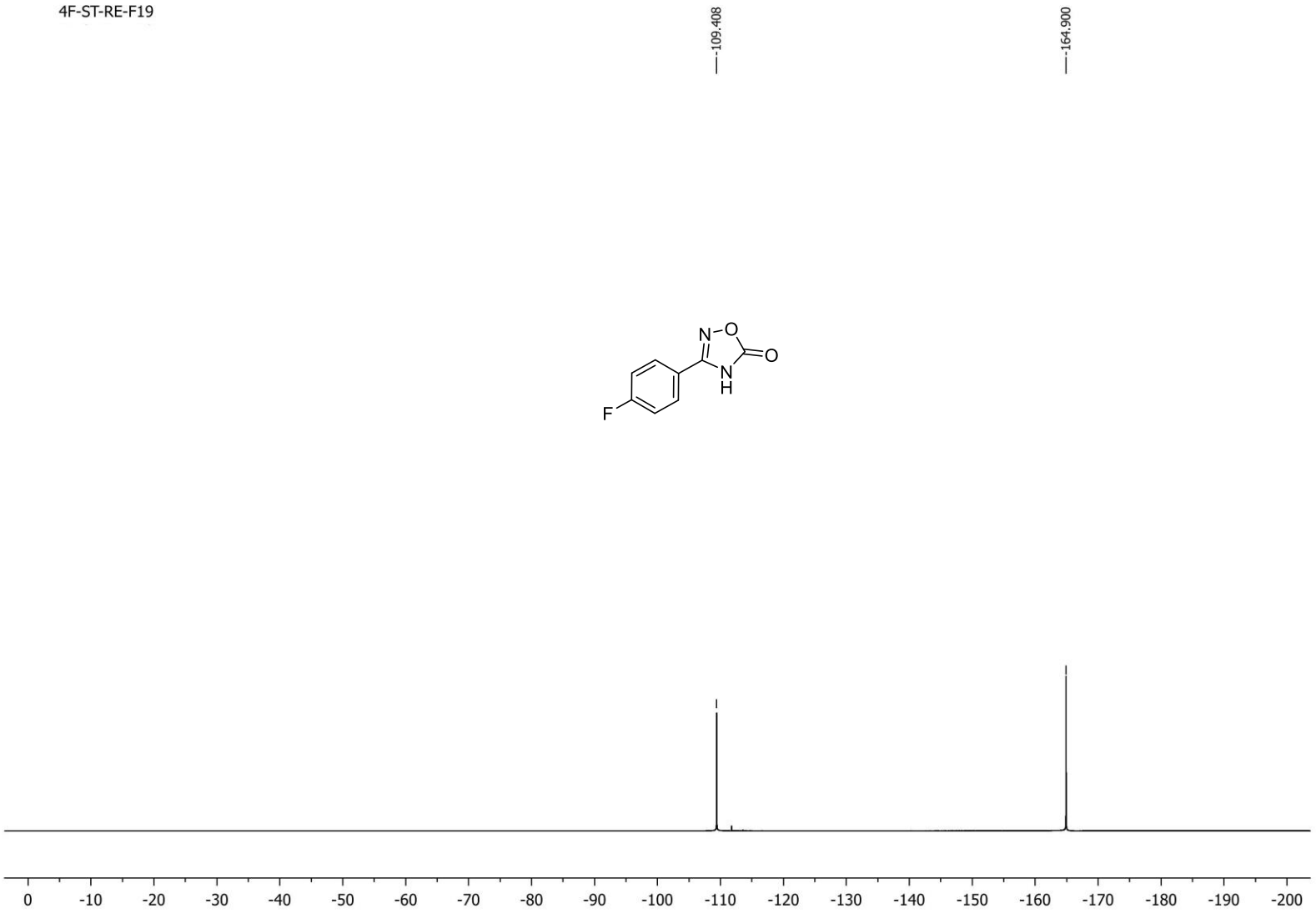
3-(4-Fluorophenyl)-1,2,4-oxadiazol-5(4*H*)-one (11a): ^{1}H NMR (DMSO-*d*$_6$, 400 MHz)



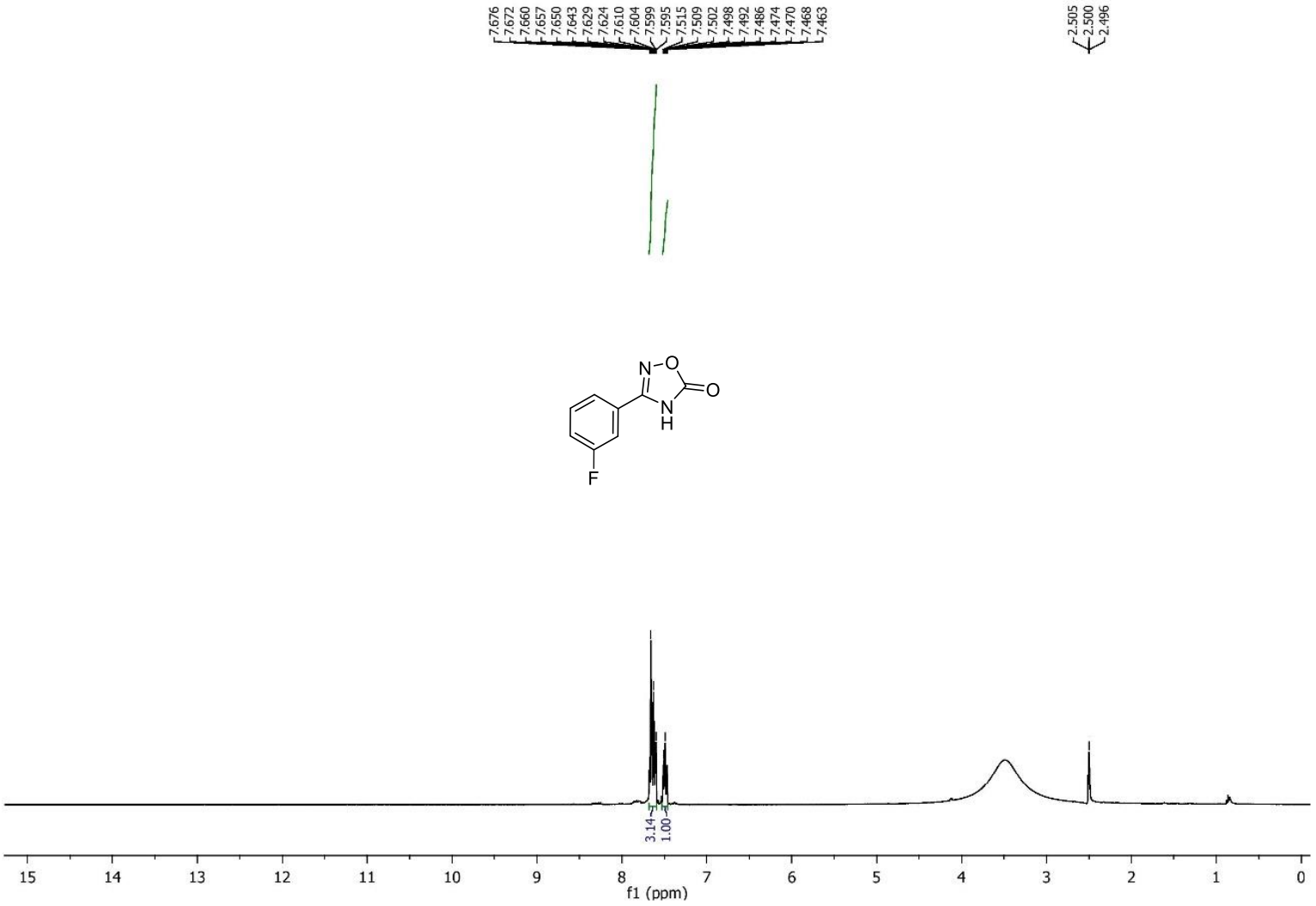
3-(4-Fluorophenyl)-1,2,4-oxadiazol-5(4*H*)-one (11a): ^{13}C NMR (DMSO-d_6, 100 MHz)



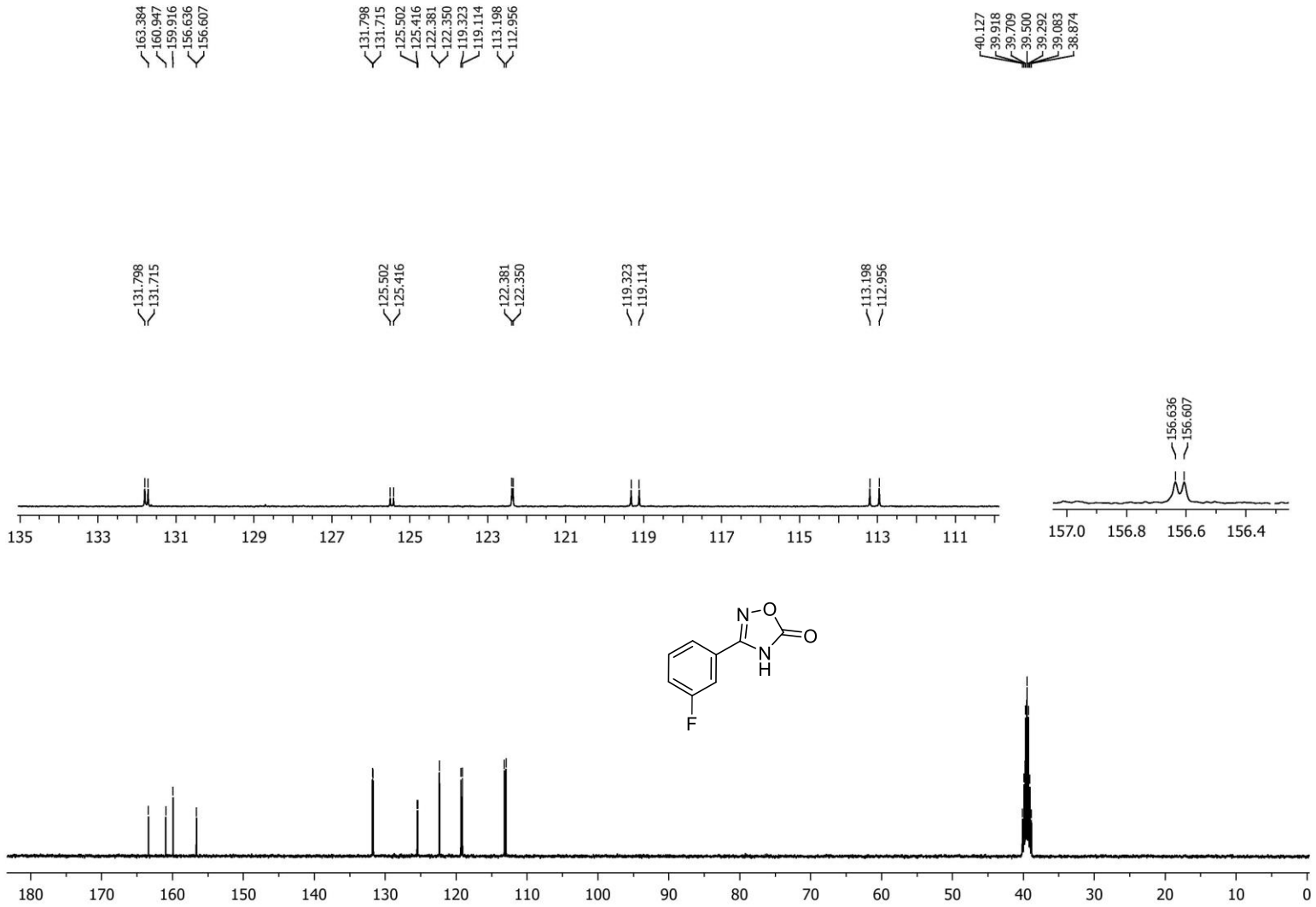
3-(4-Fluorophenyl)-1,2,4-oxadiazol-5(4*H*)-one (11a): ^{19}F NMR (DMSO-d_6 + C_6F_6)



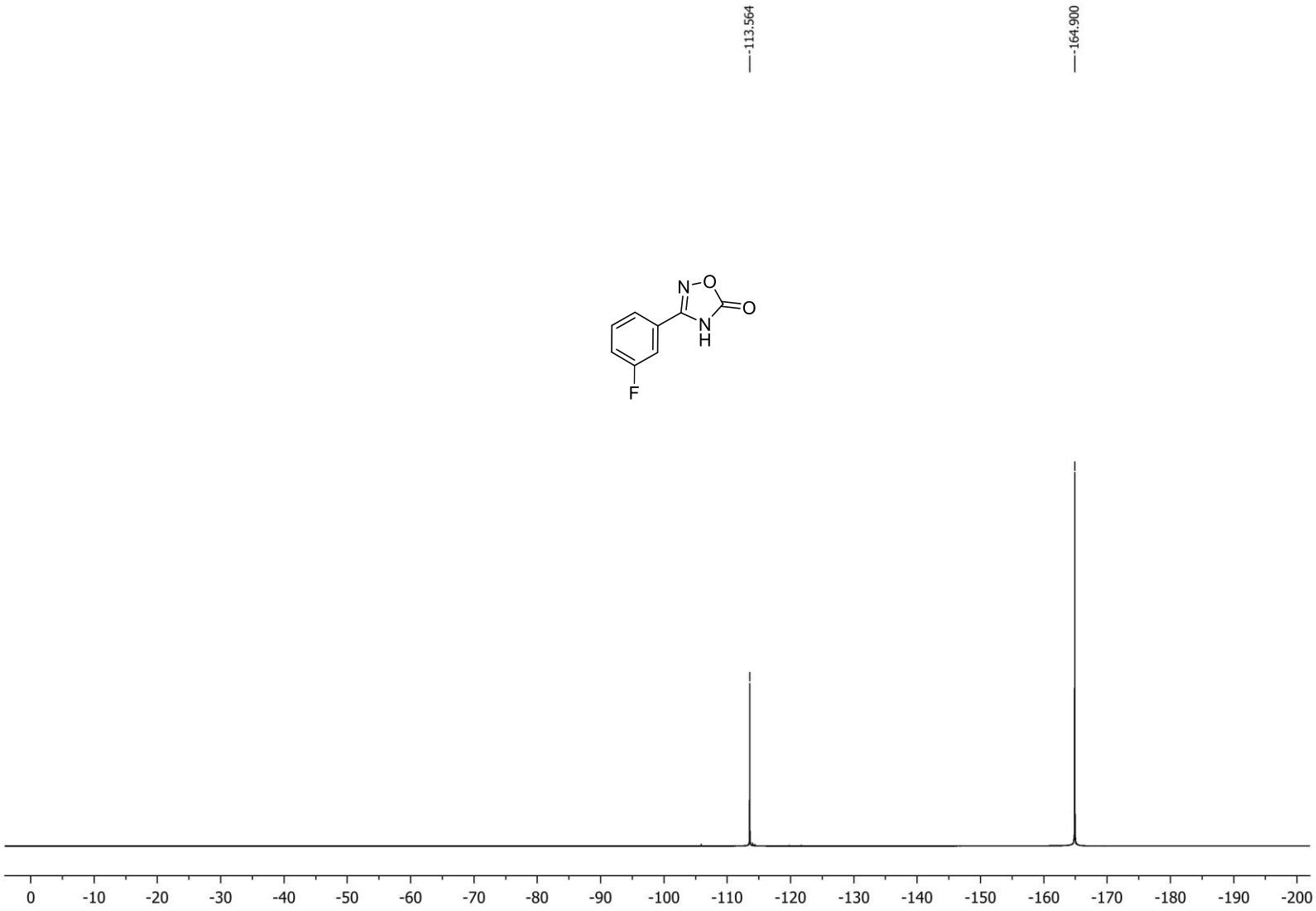
3-(3-Fluorophenyl)-1,2,4-oxadiazol-5(4*H*)-one (12a): ^{1}H NMR (DMSO-*d*$_6$, 400 MHz)



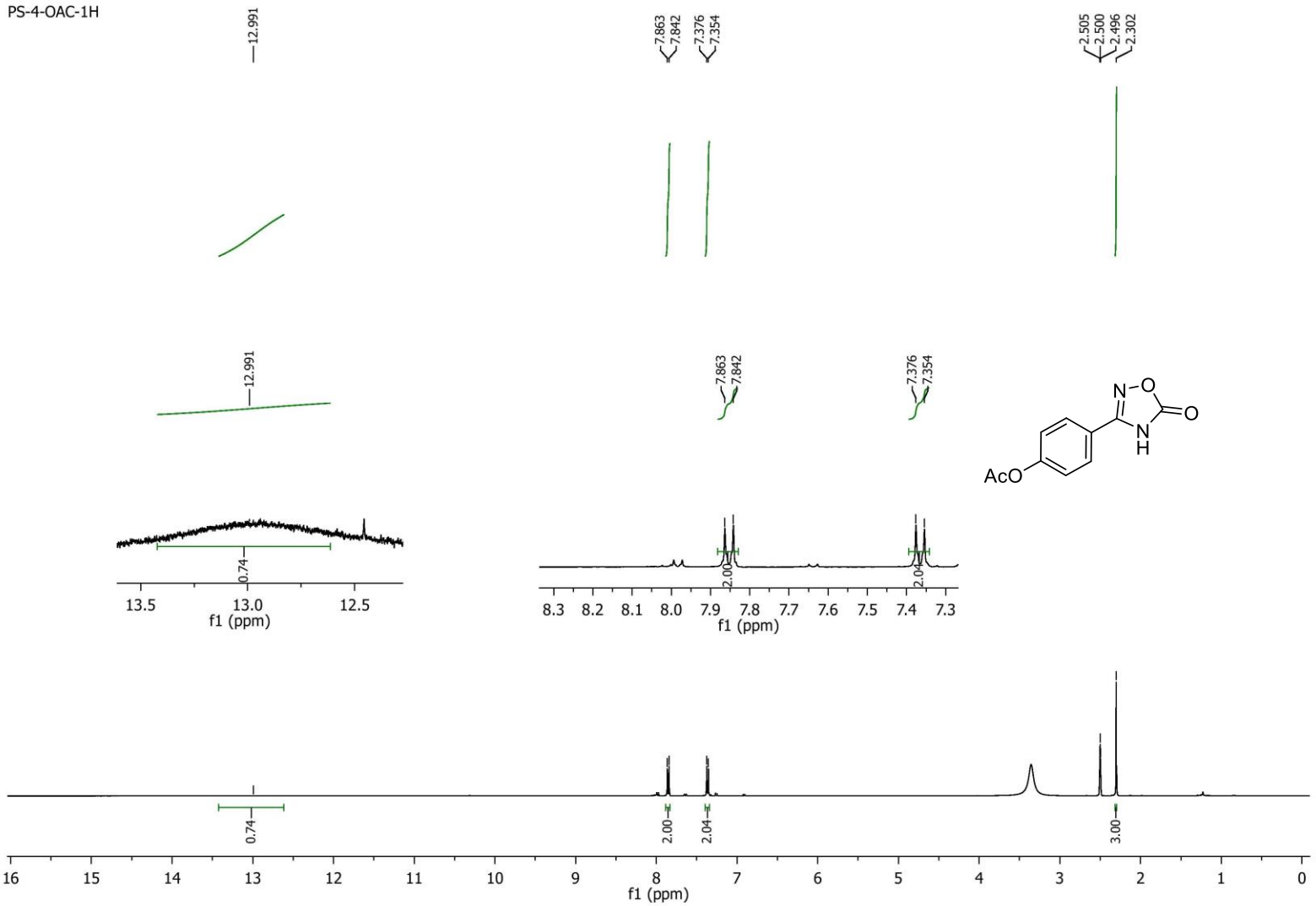
3-(3-Fluorophenyl)-1,2,4-oxadiazol-5(4*H*)-one (12a): ^{13}C NMR (DMSO-d_6, 100 MHz)



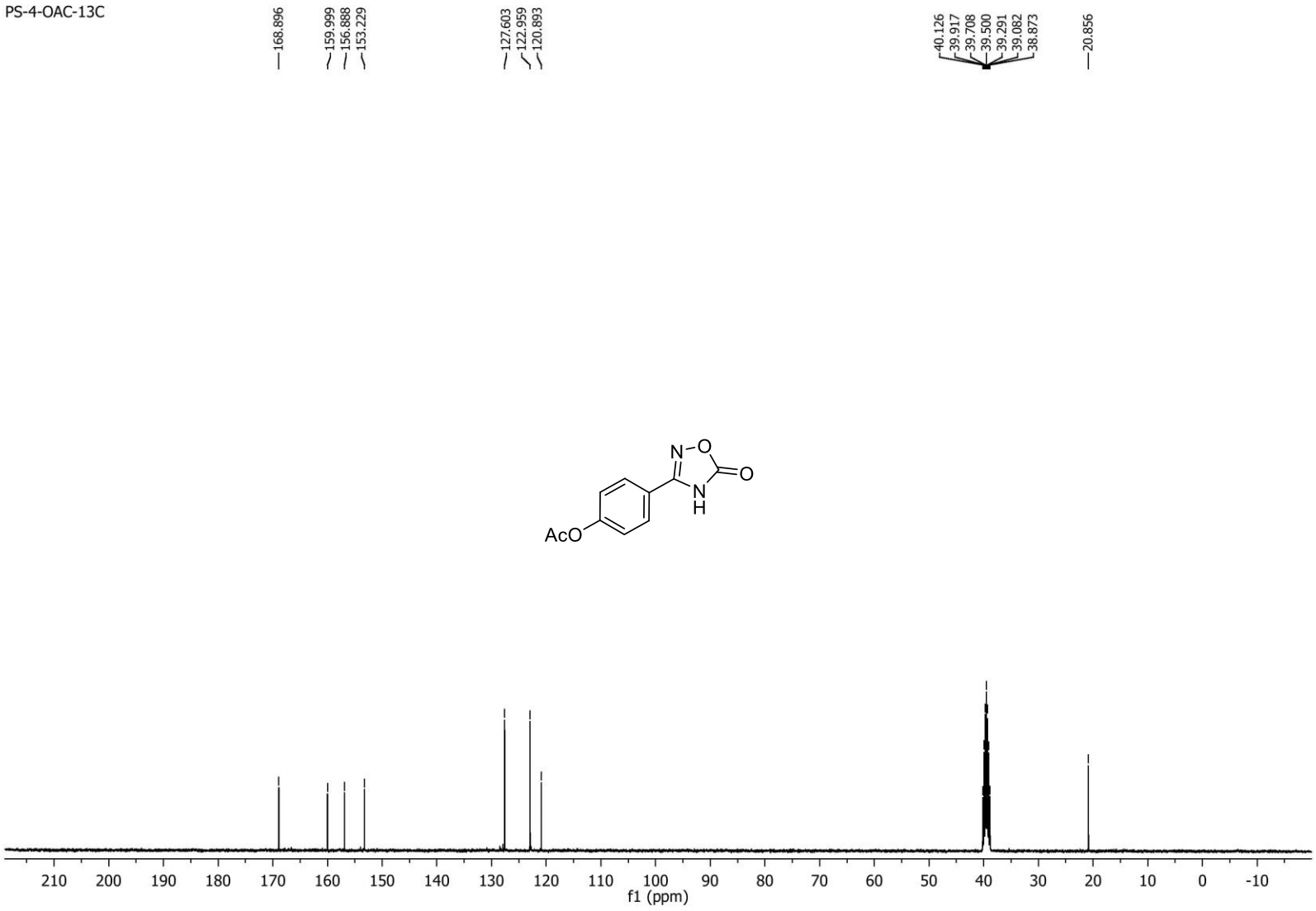
3-(3-Fluorophenyl)-1,2,4-oxadiazol-5(4*H*)-one (12a): ^{19}F NMR (DMSO-d_6 + C_6F_6)



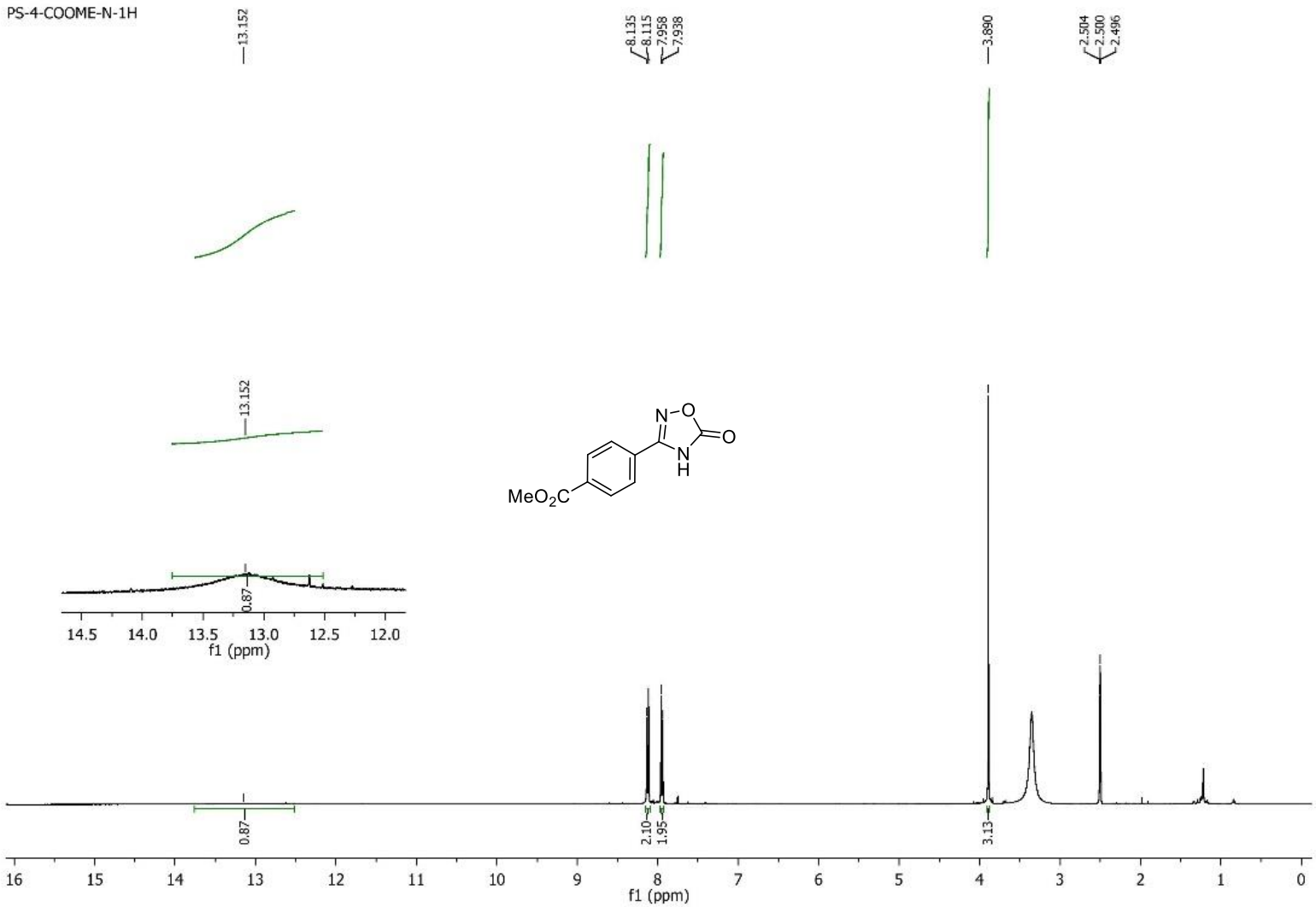
4-(5-Oxo-4,5-dihydro-1,2,4-oxadiazol-3-yl)phenyl acetate (13a): ^{1}H NMR (DMSO-*d*$_6$, 400 MHz)



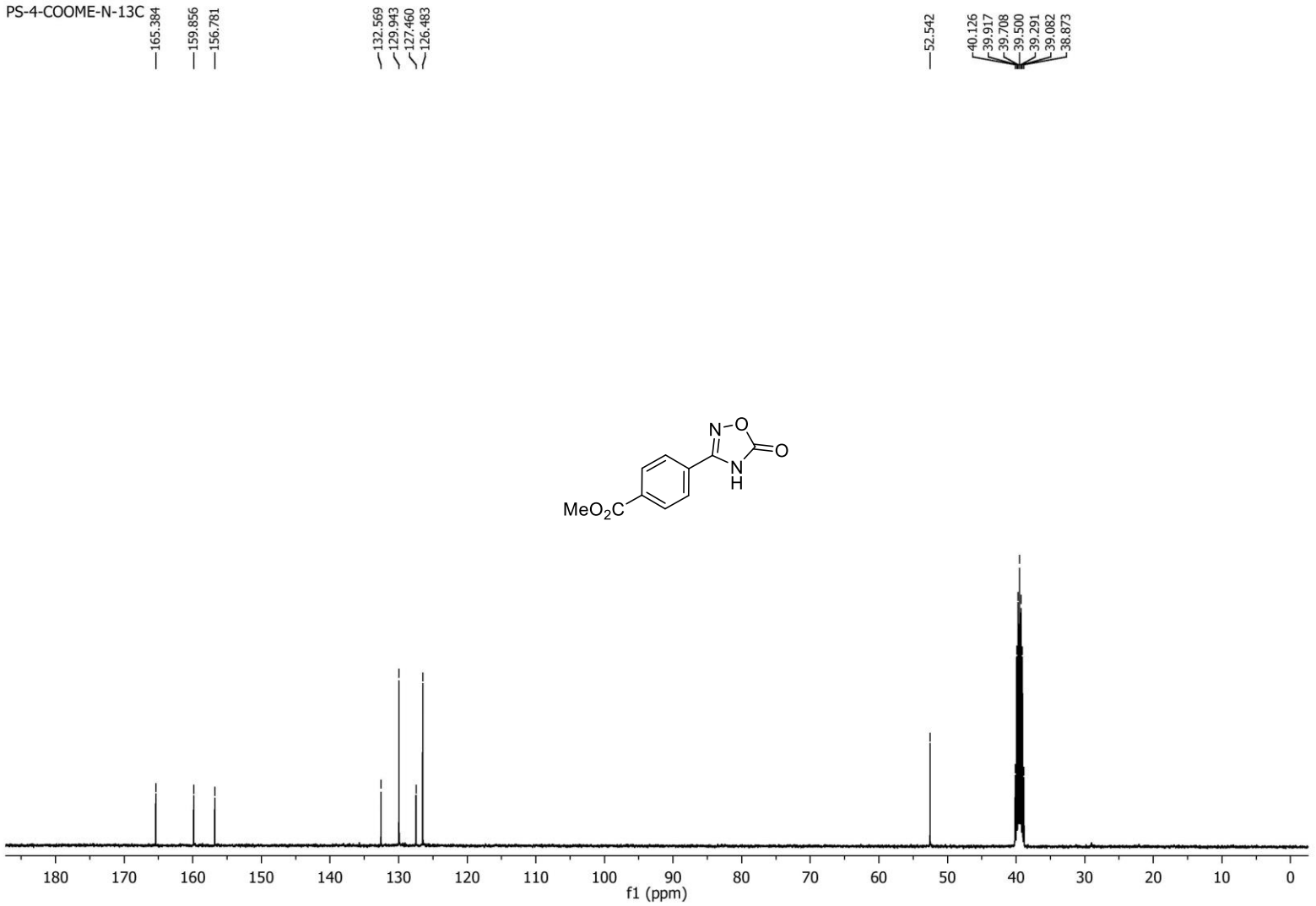
4-(5-Oxo-4,5-dihydro-1,2,4-oxadiazol-3-yl)phenyl acetate (13a): ^{13}C NMR (DMSO-d_6, 100 MHz)



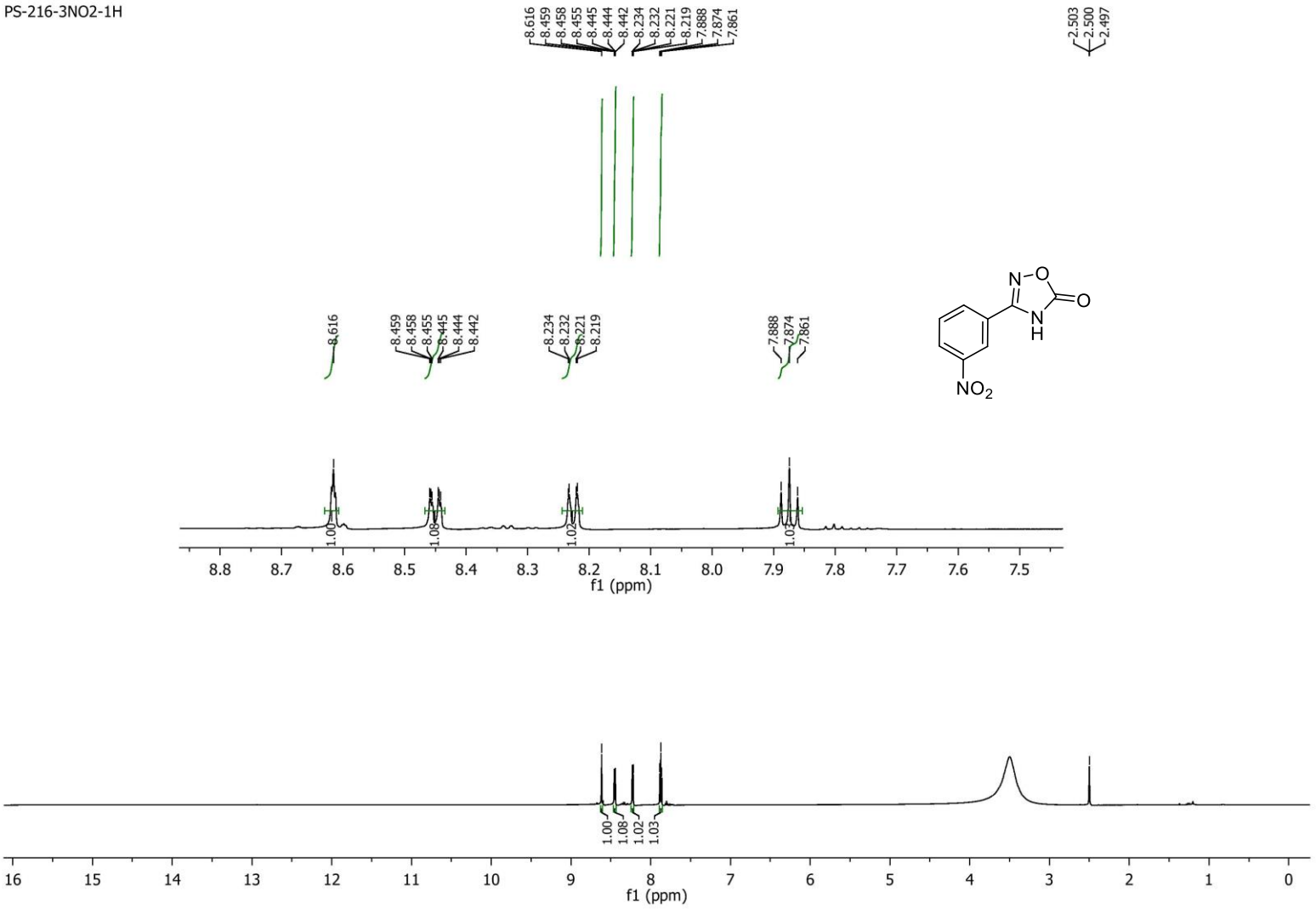
Methyl 4-(5-oxo-4,5-dihydro-1,2,4-oxadiazol-3-yl)benzoate (14a): ^{1}H NMR (DMSO-d_6, 400 MHz)



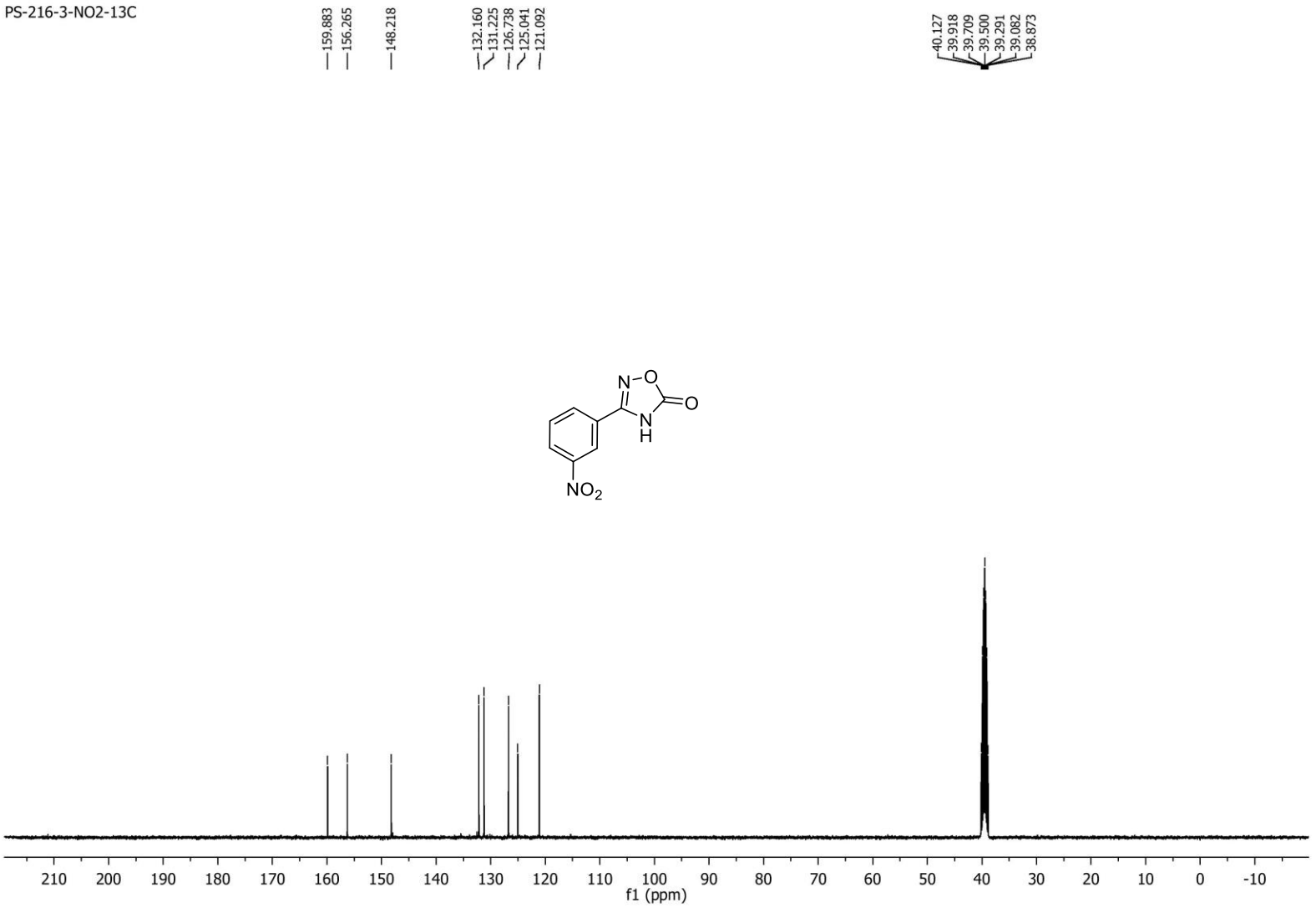
Methyl 4-(5-oxo-4,5-dihydro-1,2,4-oxadiazol-3-yl)benzoate (14a): ^{13}C NMR (DMSO-*d*$_6$, 100 MHz)



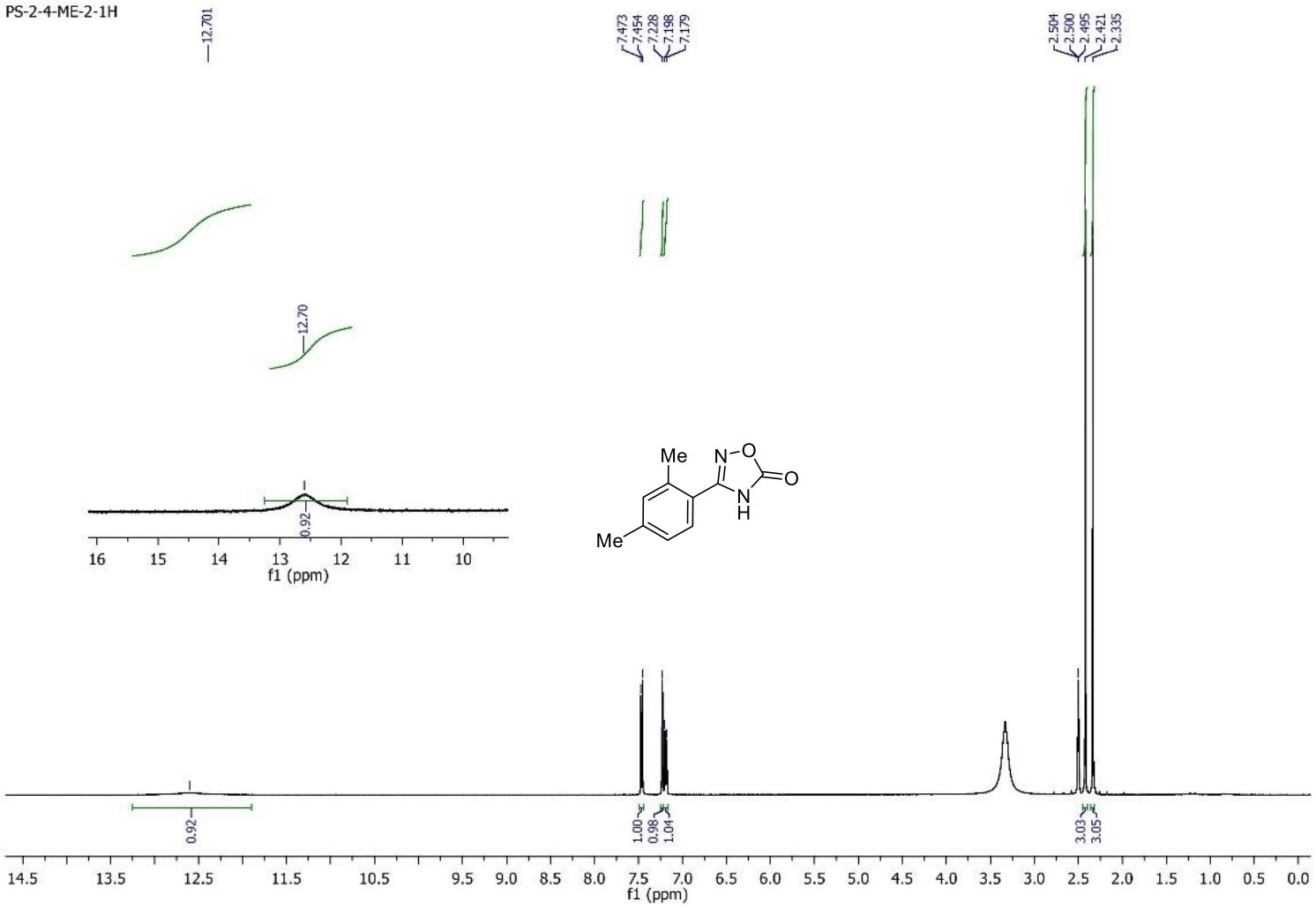
3-(3-Nitrophenyl)-1,2,4-oxadiazol-5(4H)-one (15a): ^{1}H NMR (DMSO-d_6, 600 MHz)



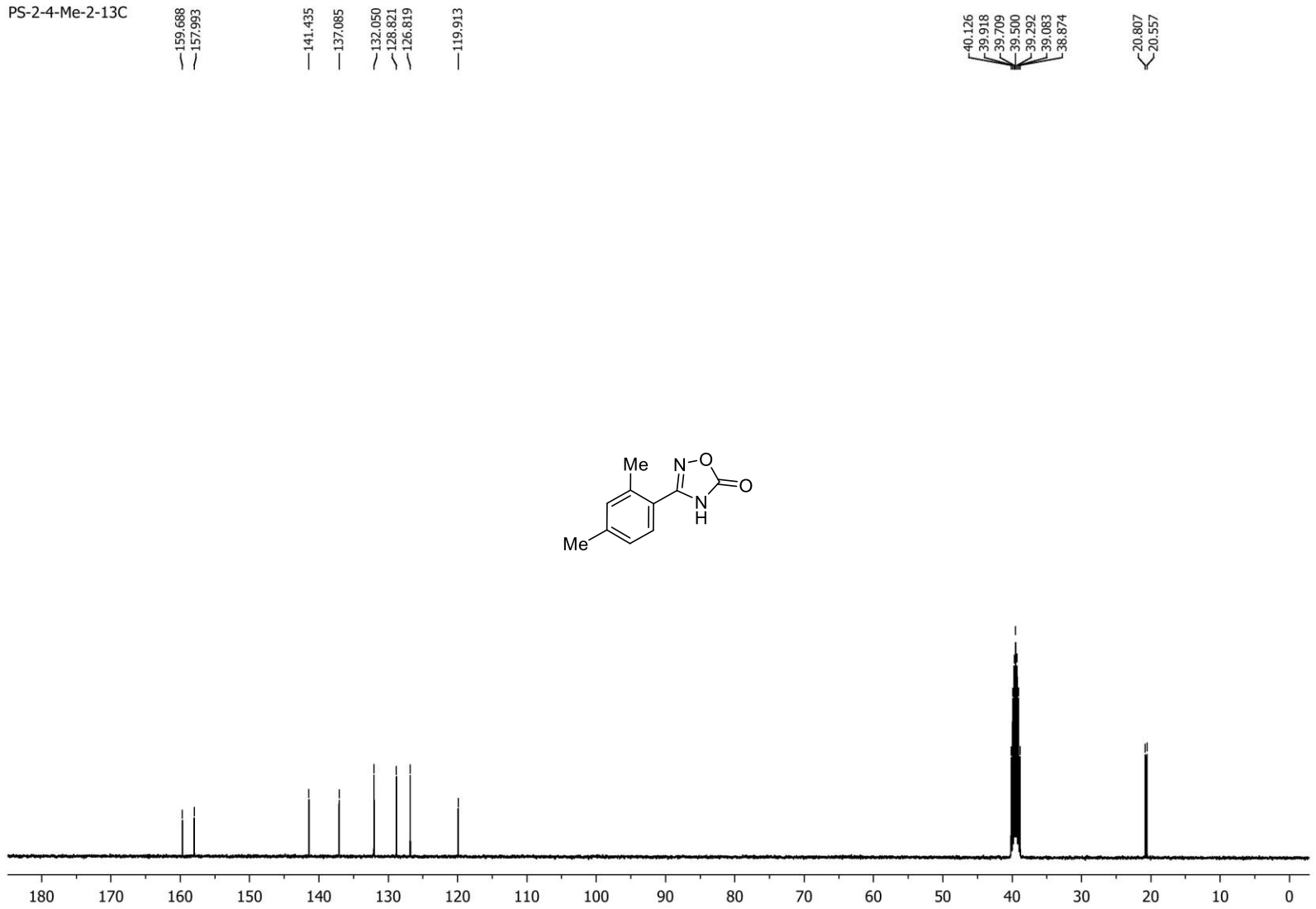
3-(3-Nitrophenyl)-1,2,4-oxadiazol-5(4*H*)-one (15a): ^{13}C NMR (DMSO-d_6, 100 MHz)



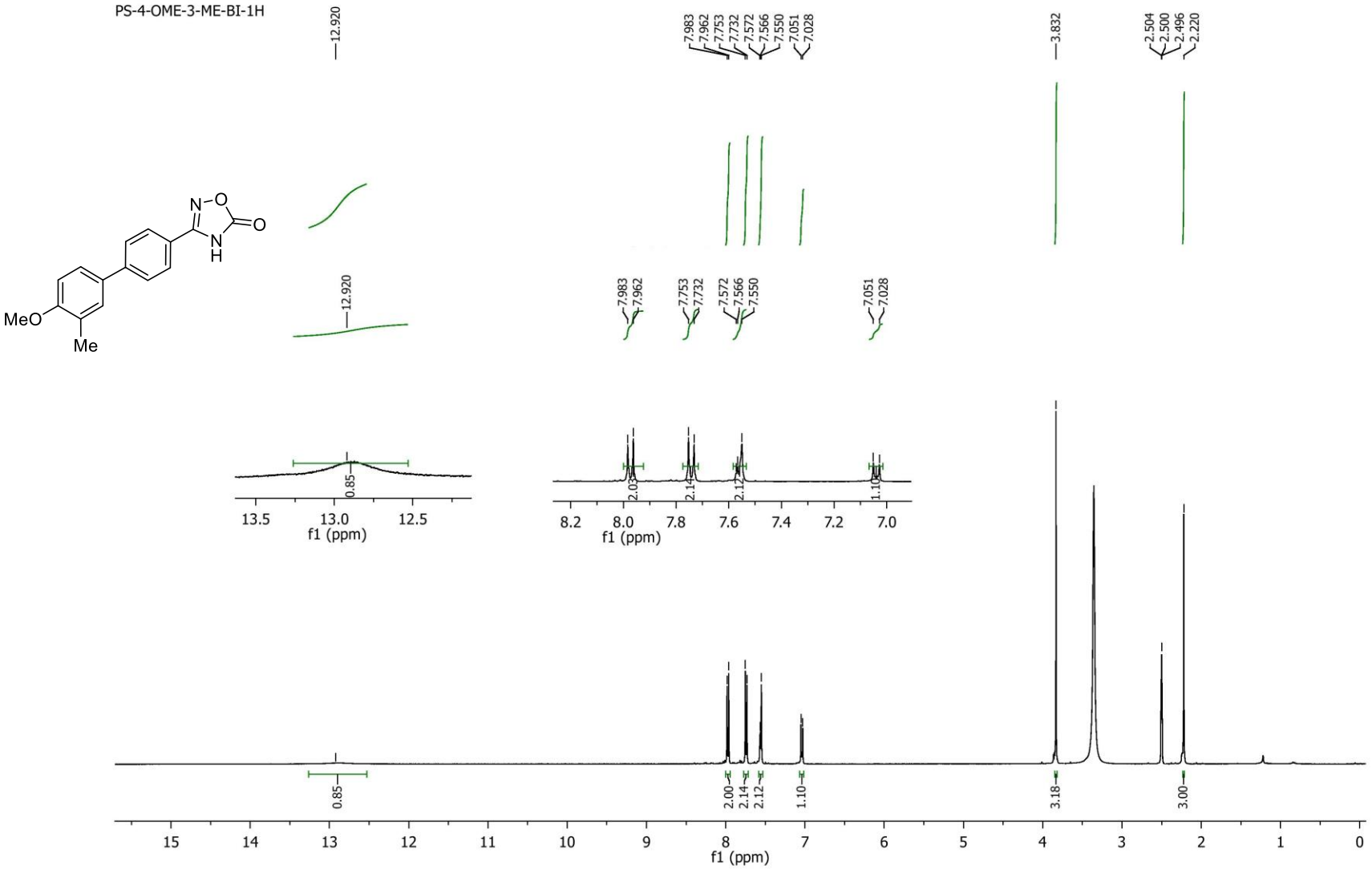
3-(2,4-Dimethylphenyl)-1,2,4-oxadiazol-5(4*H*)-one (16a): ^{1}H NMR (DMSO-d_6, 400 MHz)



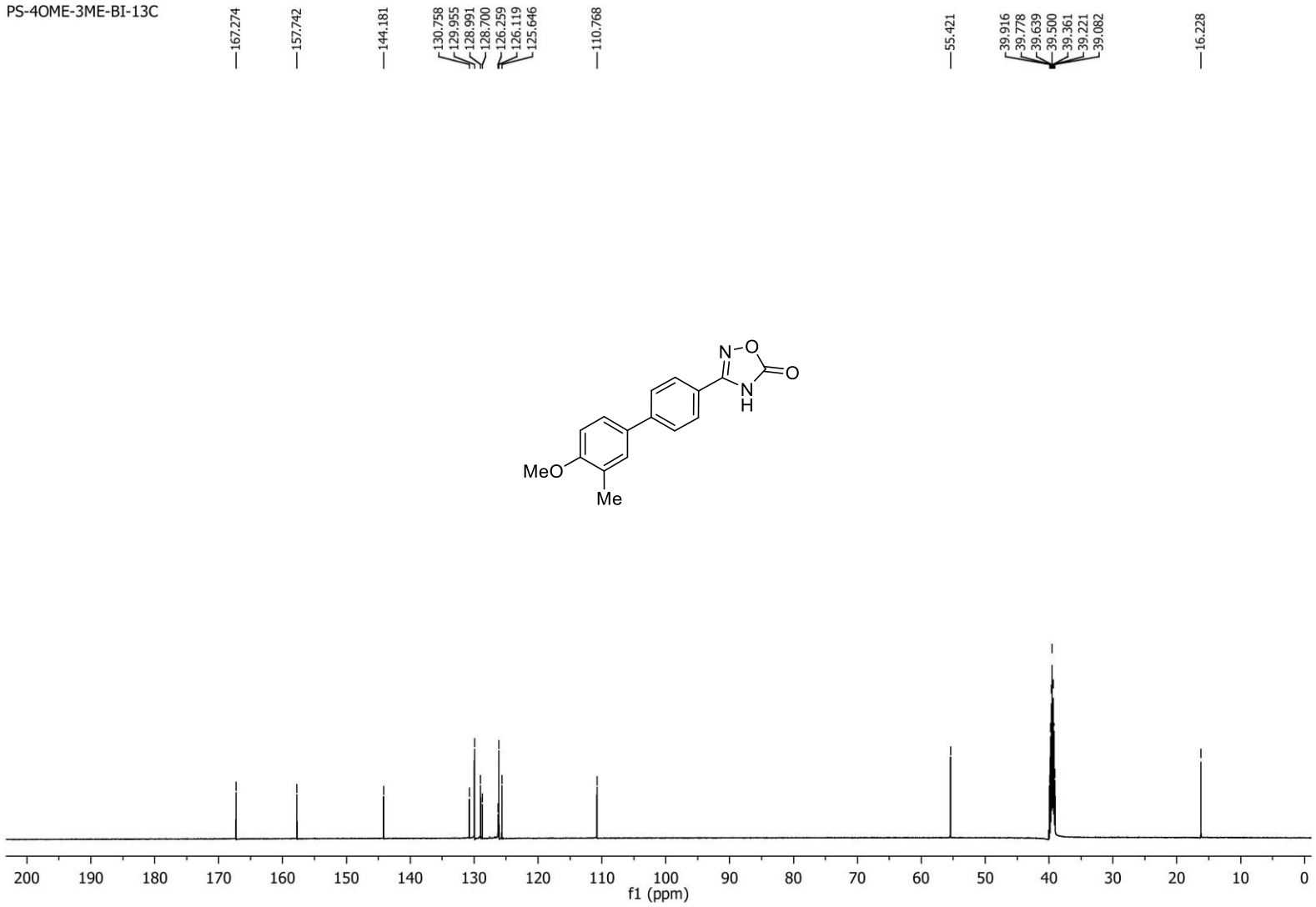
3-(2,4-Dimethylphenyl)-1,2,4-oxadiazol-5(4*H*)-one (16a): ^{13}C NMR (DMSO-d_6, 100 MHz)



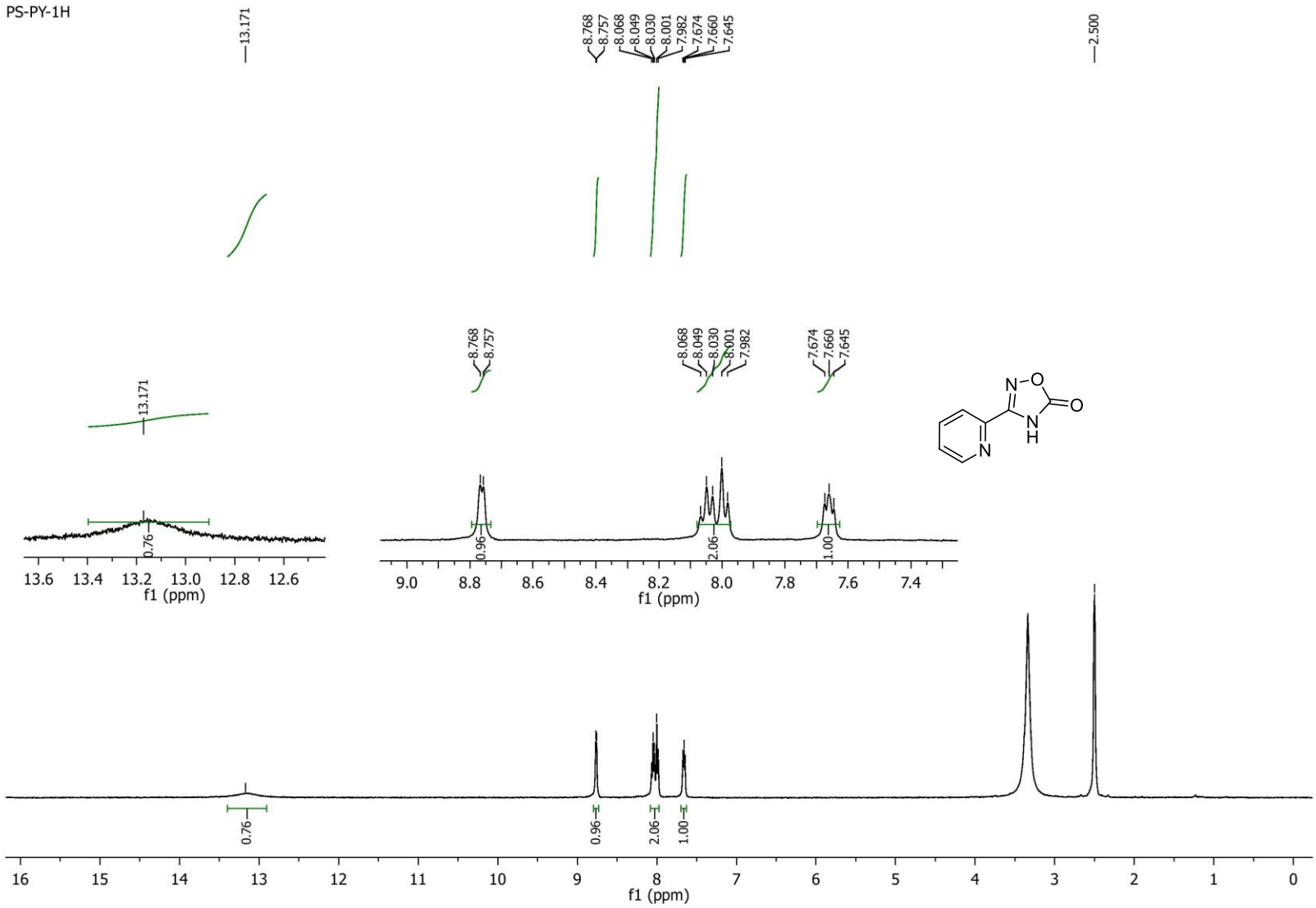
3-(4'-Methoxy-3'-methyl-[1,1'-biphenyl]-4-yl)-1,2,4-oxadiazol-5(4*H*)-one (17a): ^{1}H NMR (DMSO-d_6, 400 MHz)



3-(4'-Methoxy-3'-methyl-[1,1'-biphenyl]-4-yl)-1,2,4-oxadiazol-5(4*H*)-one (17a): ^{13}C NMR (DMSO-*d*$_6$, 150 MHz)



3-(Pyridin-2-yl)-1,2,4-oxadiazol-5(4*H*)-one (18a): ^{1}H NMR (DMSO-d_6, 400 MHz)



3-(Pyridin-2-yl)-1,2,4-oxadiazol-5(4*H*)-one (18a): ^{13}C NMR (DMSO-d_6, 100 MHz)