

Supporting Information

Direct Access to Hydrazides and Amides from Carboxylic Acids *via* Acyloxyphosphonium ion

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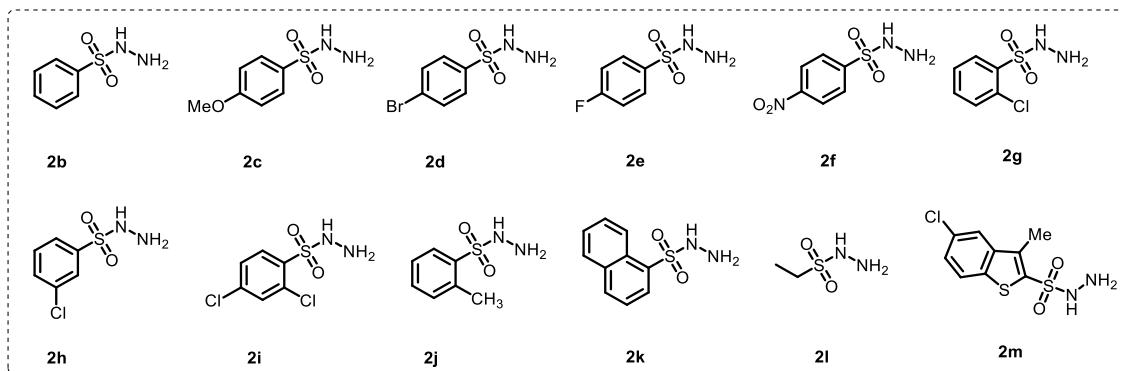
1. General information

All reagents and solvents were of pure analytical grade. All experiments were carried out in a round-bottom flask equipped with a stirring bar. Chemicals were purchased from Sigma-Aldrich, TCI, Alfa-Aesar, and Sisco Research Laboratories (SRL) and used without further purification. Analytical thin-layer chromatography (TLC) was carried out using 0.2 mm commercially available silica gel plates (silica gel 60, F254, EMD Chemical). Visualization of the developed TLC plate was performed by irradiation with UV light. High-resolution mass spectra (HRMS) were recorded on a mass spectrometer using electrospray ionization-time-of-flight (ESI-TOF) reflectron experiments. ^1H -NMR and ^{13}C -NMR were recorded on 500 MHz and 400 MHz spectrometers, using CDCl_3 and $\text{DMSO}-d_6$ as a solvent; the chemical shifts are reported as parts per million (ppm) referenced to residual protium or carbon of the solvents; $\text{CDCl}_3 \delta \text{ H}$ (7.26 ppm) and $\text{DMSO}-d_6 \delta \text{ H}$ (2.50 ppm). Coupling constants are reported in Hertz (Hz). Data for ^1H NMR spectra are reported as follows: chemical shift (ppm, referenced to protium; s = singlet, d = doublet, t = triplet, q = quartet, quin = quintet, sext = sextet, dd = doublet of doublets, td = triplet of doublets, ddd = doublet of doublet of doublets, m = multiplet, coupling constant (Hz), and integration).

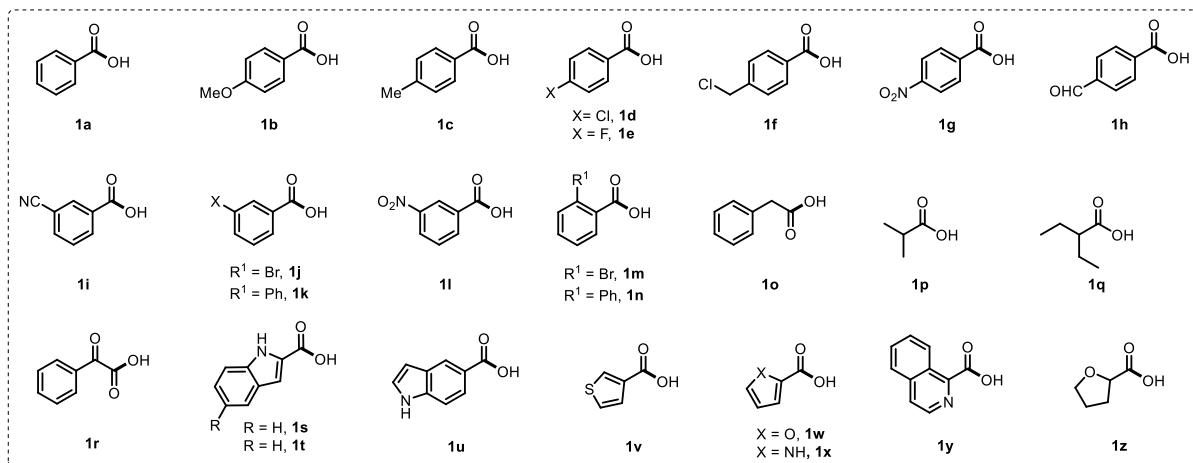
2. Experimental Section

2.1 Preparation of Sulphonyl Hydrazides

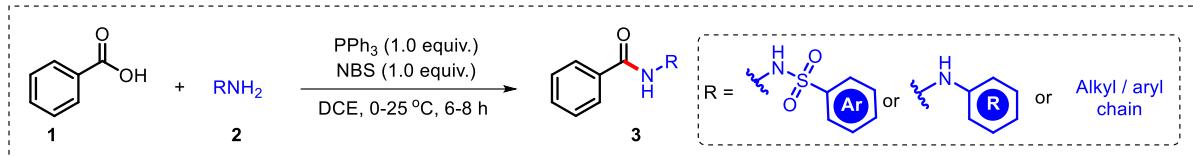
The sulphonyl hydrazides **2b-2m** were prepared according to previously reported procedures.^[1]



2.2 Acid derivatives



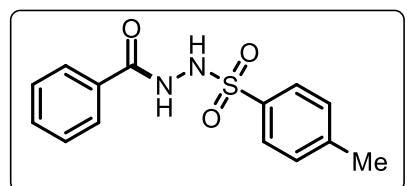
3. General Procedure (GP1) for Hydrazides and Amides (3a-3x, 3aa-3at, 4a, 4b, 5, 6, 7):



In a reaction vial (5.0 mL), triphenylphosphine (0.2 mmol, 1.0 equiv.), *N*-Bromosuccinamide (0.2 mmol, 1.0 equiv.) was taken, followed by adding 1.0 mL DCE, and stirred for 5 min at 0 °C. After that, Benzoic acid (**1**, 0.2 mmol, 1.0 equiv.) was added to it and kept for stirring for 15 min. finally, hydrazide derivative (**2**, 0.24 mmol, 1.2 equiv.) was added. The reaction was stirred at 0 °C to rt for 6-8 hours. A TLC plate monitored the completion of the reaction in 30% EtOAc in hexane. The crude was purified by column chromatography and eluted with hexane/EtOAc to afford the desired products. The product was characterized and identified by analyzing spectral data (¹H and ¹³C NMR and HRMS).

4. Characterization Data of Products

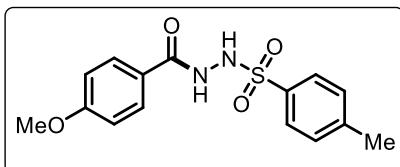
***N'*-Benzoyl-4-methylbenzenesulfonohydrazide (3a):** The compound **3a** was synthesized using



the **GP1**, **1a** (1.0 equiv., 0.2 mmol, 24.4 mg), *p*-toluene benzene sulfonyl hydrazide **2a** (1.2 equiv., 0.24 mmol, 44.7 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography

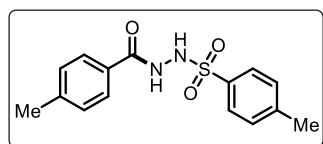
(hexane/ethyl acetate = 70:30); white solid (52 mg, 90% yield). **¹H NMR** (500 MHz, CDCl₃) δ 8.68 (s, 1H), 7.90 (d, *J* = 8.2 Hz, 2H), 7.74 (d, *J* = 7.6 Hz, 3H), 7.61 (t, *J* = 7.5 Hz, 1H), 7.49 (t, *J* = 7.8 Hz, 2H), 7.32 (d, *J* = 8.2 Hz, 2H), 2.44 (s, 3H). **¹³C{¹H} NMR** (126 MHz, CDCl₃) δ 166.3, 145.1, 133.2, 132.9, 130.7, 129.8, 128.9, 128.7, 127.4, 21.8. HRMS (ESI-TOF) calculated for C₁₄H₁₄N₂NaO₃S [M + Na]⁺: 313.0623, found: 313.0625.

N'-(4-Methoxybenzoyl)-4-methylbenzenesulfonohydrazide (3b): The compound **3b** was



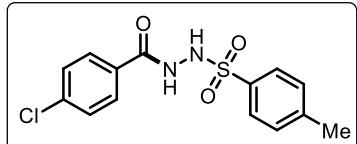
synthesized using the **GP1**, 4-methoxy benzoic acid (**1b**, 1.0 equiv., 0.2 mmol, 30.4 mg), **2a** (1.2 equiv., 0.24 mmol, 44.7 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (58.9 mg, 92% yield). **¹H NMR** (500 MHz, DMSO-*d*₆) δ 10.51 (s, 1H), 9.81 (s, 1H), 7.70 (dd, *J* = 13.6, 8.5 Hz, 4H), 7.31 (d, *J* = 8.2 Hz, 2H), 6.96 (d, *J* = 8.9 Hz, 2H), 3.78 (s, 3H), 2.34 (s, 3H). **¹³C{¹H} NMR** (126 MHz, DMSO-*d*₆) δ 162.1, 143.2, 136.3, 129.4, 129.3, 127.74, 127.69, 124.1, 113.7, 55.4, 21.0.

4-Methyl-N'-(4-methyl benzoyl)benzenesulfonohydrazide (3c): The compound **3c** was



synthesized using the **GP1**, 4-methyl benzoic acid (**1c**, 1.0 equiv., 0.2 mmol, 27.23 mg), **2a** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (54.8 mg, 90% yield). **¹H NMR** (500 MHz, DMSO-*d*₆) δ 10.59 (s, 1H), 9.87 (s, 1H), 7.72 (d, *J* = 8.2 Hz, 2H), 7.59 (d, *J* = 8.1 Hz, 2H), 7.31 (d, *J* = 8.2 Hz, 2H), 7.23 (d, *J* = 8.1 Hz, 2H), 2.34 (s, 3H), 2.31 (s, 3H). **¹³C{¹H} NMR** (126 MHz, DMSO-*d*₆) δ 165.4, 143.3, 142.1, 136.2, 129.4, 129.3, 128.9, 127.8, 127.5, 21.1, 21.0.

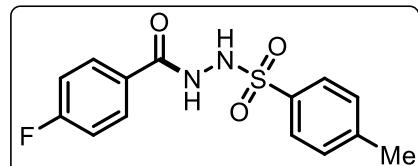
N'-(4-Chlorobenzoyl)-4-methylbenzenesulfonohydrazide (3d): The compound **3d** was



synthesized using the **GP1**, 4-chlorobenzoic acid (**1d**, 1.0 equiv., 0.2 mmol, 31.3 mg), **2a** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg)

in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (54.8 mg, 90% yield). **¹H NMR** (500 MHz, DMSO-*d*₆) δ 10.74 (s, 1H), 9.95 (s, 1H), 7.70 (t, *J* = 8.3 Hz, 4H), 7.51 (d, *J* = 8.5 Hz, 2H), 7.32 (d, *J* = 8.4 Hz, 2H), 2.34 (s, 3H). **¹³C{¹H} NMR** (126 MHz, DMSO-*d*₆) δ 164.6, 143.3, 136.8, 136.1, 130.8, 129.4, 129.3, 128.6, 127.7, 21.1. HRMS (ESI-TOF) calculated for C₁₄H₁₄N₂NaClO₃S [M + Na]⁺: 325.0414, found: 325.0413.

N'-(4-Fluorobenzoyl)-4-methylbenzenesulfonohydrazide (3e): The compound **3e** was

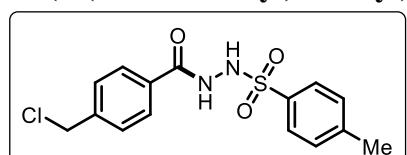


synthesized using the **GP1**, 4-fluoro benzoic acid (**1e**, 1.0 equiv., 0.2 mmol, 28 mg), **2a** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column

chromatography (hexane/ethyl acetate = 70:30); white solid (54.2 mg, 88% yield). **¹H NMR** (500 MHz, DMSO-*d*₆) δ 10.68 (s, 1H), 9.91 (s, 1H), 7.77 (t, *J* = 7.0 Hz, 1H), 7.72 (d, *J* = 8.1 Hz, 2H), 7.38 (t, *J* = 7.2 Hz, 1H), 7.32 (d, *J* = 7.9 Hz, 2H), 7.27 (t, *J* = 8.9 Hz, 2H), 2.35 (s, 3H). **¹³C{¹H} NMR** (126 MHz, DMSO-*d*₆) 165.39, 164.8 (d, *J* = 250.1), 144.2, 139.1, 136.2, δ 130.7 (d, *J* = 9.2 Hz), 129.9, 128.9, 128.2, 126.1

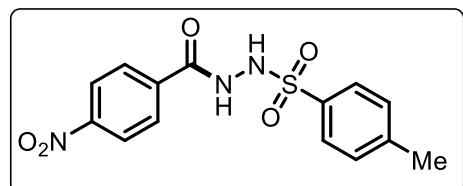
(d, $J = 14.1$ Hz), 116.1 (d, $J = 22.0$ Hz), 21.5. HRMS (ESI-TOF) calculated for $C_{14}H_{14}N_2NaO_3SF$ [M + H]⁺: 309.0709, found: 309.0708.

***N'*-(4-(Chloromethyl)benzoyl)-4-methylbenzenesulfonohydrazide (3f):** The compound **3f**



was synthesized using the **GP1**, 4-chloromethyl benzoic acid (**1e**, 1.0 equiv., 0.2 mmol, 34.1 mg), **2a** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (54.2 mg, 80% yield); ¹H NMR (500 MHz, DMSO-d₆) δ 10.71 (s, 1H), 9.94 (s, 1H), 7.76 – 7.71 (m, 3H), 7.67 (d, $J = 7.6$ Hz, 1H), 7.60 (d, $J = 7.5$ Hz, 1H), 7.45 (t, $J = 8.5$ Hz, 1H), 7.32 (d, $J = 7.5$ Hz, 2H), 4.78 (s, 2H), 2.34 (s, 3H). ¹³C{¹H} NMR (126 MHz, DMSO-d₆) δ 165.2, 143.3, 138.1, 136.2, 129.3, 128.9, 128.0, 127.7, 127.3, 45.6, 21.1. HRMS (ESI-TOF) calculated for $C_{15}H_{15}ClN_2NaO_3S$ [M + Na]⁺: 361.0390, found: 361.0391.

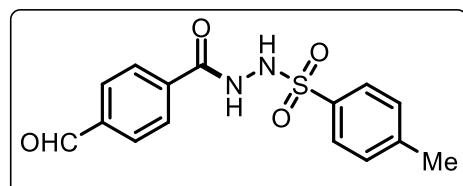
4-Methyl-*N'*-(4-nitrobenzoyl)benzenesulfonohydrazide (3g): The compound **3g** was



synthesized using the **GP1**, 4-nitrobenzoic acid (**1g**, 1.0 equiv., 0.2 mmol, 33.4 mg), **2a** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified

by column chromatography (hexane/ethyl acetate = 70:30); white solid (57.6 mg, 86% yield); ¹H NMR (500 MHz, DMSO-d₆) δ 10.99 (s, 1H), 10.05 (s, 1H), 8.28 (d, $J = 8.9$ Hz, 2H), 7.92 (d, $J = 8.9$ Hz, 2H), 7.75 (d, $J = 8.4$ Hz, 2H), 7.34 (d, $J = 8.2$ Hz, 2H), 2.35 (s, 3H). ¹³C{¹H} NMR (126 MHz, DMSO-d₆) δ 164.0, 149.4, 143.4, 137.7, 136.0, 129.3, 128.9, 127.7, 127.6, 123.6, 21.0. HRMS (ESI-TOF) calculated for $C_{14}H_{13}N_3NaO_5S$ [M + Na]⁺: 358.0474, found: 358.0473.

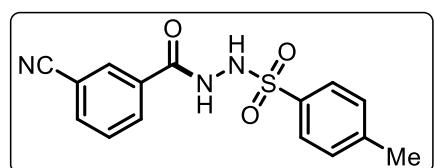
***N'*-(4-Formylbenzoyl)-4-methylbenzenesulfonohydrazide (3h):** The compound **3h** was



synthesized using the **GP1**, 4-formyl benzoic acid (**1h**, 1.0 equiv., 0.2 mmol, 30 mg), **2a** (1.2 equiv., 0.24 mmol, 44.7 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was

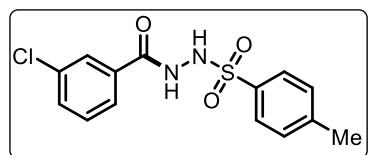
purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (54 mg, 85% yield). ¹H NMR (500 MHz, DMSO-d₆) δ 11.62 (s, 1H), 10.69 (s, 1H), 9.90 (s, 1H), 7.92 (s, 1H), 7.76 (d, $J = 8.2$ Hz, 1H), 7.73 – 7.64 (m, 2H), 7.60 (d, $J = 8.4$ Hz, 2H), 7.40 (d, $J = 8.1$ Hz, 1H), 7.32 (d, $J = 7.9$ Hz, 1H), 2.35 (s, 3H). ¹³C{¹H} NMR (126 MHz, DMSO-d₆) δ 179.5, 164.9, 145.7, 143.6, 143.3, 129.8, 129.3, 127.9, 127.7, 127.2, 126.7, 21.1. HRMS (ESI-TOF) calculated for $C_{15}H_{13}N_2NaO_4S$ [M + Na]⁺: 341.0572, found: 341.0571.

N'-(3-Cyanobenzoyl)-4-methylbenzenesulfonohydrazide (3i): The compound **3i** was



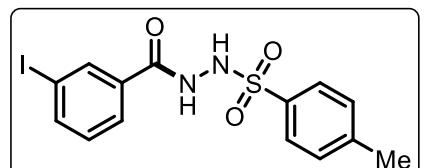
synthesized using the **GP1**, 3-cyano benzoic acid (**1i**, 1.0 equiv., 0.2 mmol, 27.2 mg), **2a** (1.2 equiv., 0.24 mmol, 44.7 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (55.5 mg, 87% yield). **¹H NMR** (500 MHz, DMSO-*d*₆) δ 10.85 (d, *J* = 3.4 Hz, 1H), 10.03 (d, *J* = 3.4 Hz, 1H), 8.10 (s, 1H), 8.00 (dd, *J* = 15.6, 7.9 Hz, 2H), 7.74 (d, *J* = 8.2 Hz, 2H), 7.67 (t, *J* = 7.8 Hz, 1H), 7.34 (d, *J* = 8.2 Hz, 2H), 2.35 (s, 3H). **¹³C{¹H} NMR** (126 MHz, DMSO-*d*₆) δ 163.8, 143.4, 136.1, 135.4, 133.0, 132.2, 131.0, 129.9, 129.3, 127.7, 111.7, 21.0. HRMS (ESI-TOF) calculated for C₁₅H₁₃N₃NaO₃S [M + Na]⁺: 338.0576, found: 338.0575.

N'-(3-Chlorobenzoyl)-4-methylbenzenesulfonohydrazide (3j): The compound **3j** was



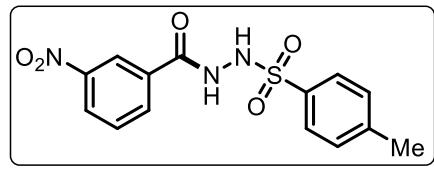
synthesized using the **GP1**, 3-chlorobenzoic acid (**1j**, 1.0 equiv., 0.2 mmol, 31.3 mg), **2a** (1.2 equiv., 0.24 mmol, 44.7 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (53.5 mg, 88% yield). **¹H NMR** (500 MHz, DMSO-*d*₆) δ 10.76 (s, 1H), 9.96 (d, *J* = 3.4 Hz, 1H), 7.84 (s, 1H), 7.72 (td, *J* = 15.6, 8.4 Hz, 4H), 7.42 (t, *J* = 7.9 Hz, 1H), 7.34 (d, *J* = 8.2 Hz, 2H), 2.36 (s, 3H). **¹³C{¹H} NMR** (126 MHz, DMSO-*d*₆) δ 164.0, 143.3, 136.1, 134.7, 134.1, 130.6, 130.0, 129.3, 127.7, 126.6, 121.6, 21.0. HRMS (ESI-TOF) calculated for C₁₄H₁₃ClN₂NaO₃S [M + Na]⁺: 347.0233, found: 347.0231.

N'-(3-Iodobenzoyl)-4-methylbenzenesulfonohydrazide (3k): The compound **3k** was



synthesized using the **GP1**, 3-iodobenzoic acid **1k** (1.0 equiv., 0.2 mmol, 50 mg), **2a** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (74.8 mg, 90% yield). **¹H NMR** (500 MHz, DMSO-*d*₆) δ 10.75 (d, *J* = 3.5 Hz, 1H), 9.96 (d, *J* = 3.5 Hz, 1H), 8.03 (s, 1H), 7.89 (d, *J* = 8.1 Hz, 1H), 7.72 (dd, *J* = 12.6, 8.0 Hz, 3H), 7.32 (d, *J* = 8.4 Hz, 2H), 7.25 (t, *J* = 7.9 Hz, 1H), 2.34 (s, 3H). **¹³C{¹H} NMR** (126 MHz, DMSO-*d*₆) δ 164.0, 143.3, 140.5, 136.2, 135.8, 133.9, 130.6, 129.3, 127.7, 126.9, 94.6, 21.0. HRMS (ESI-TOF) calculated for C₁₄H₁₃IN₂NaO₃S [M + Na]⁺: 438.9589, found: 438.9584.

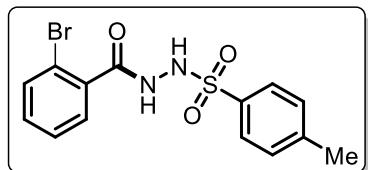
4-Methyl-N'-(3-nitrobenzoyl) benzenesulfonohydrazide (3l): The compound **3l** was



synthesized using the **GP1**, 3-nitrobenzoic acid **1l** (1.0 equiv., 0.2 mmol, 33.4 mg), **2a** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (59.6 mg, 89% yield). **¹H NMR** (500 MHz,

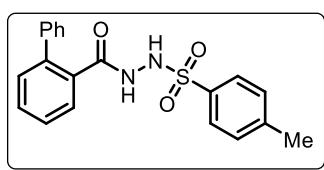
DMSO-*d*₆) δ 11.04 (s, 1H), 10.09 (s, 1H), 8.51 (s, 1H), 8.39 (d, *J* = 8.2 Hz, 1H), 8.13 (d, *J* = 7.8 Hz, 1H), 7.74 (d, *J* = 8.2 Hz, 3H), 7.35 (d, *J* = 8.4 Hz, 2H), 2.36 (s, 3H). ¹³C{¹H} NMR (126 MHz, DMSO-*d*₆) δ 163.5, 147.7, 143.4, 136.1, 133.8, 133.4, 130.4, 129.4, 127.7, 126.6, 122.2, 21.0. HRMS (ESI-TOF) calculated for C₁₄H₁₃N₃NaO₅S [M + Na]⁺: 358.0474, found: 358.0473.

***N'*-(2-Bromobenzoyl)-4-methylbenzenesulfonohydrazide (3m):** The compound **3m** was



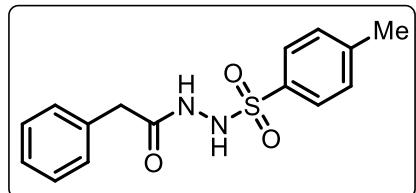
synthesized using the **GP1**, 4-chlorobenzoic acid **1m** (1.0 equiv., 0.2 mmol, 31.3 mg), **2a** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (65 mg, 88% yield). ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.57 (s, 1H), 10.12 (s, 1H), 7.81 (d, *J* = 8.2 Hz, 2H), 7.62 (d, *J* = 7.9 Hz, 1H), 7.46 – 7.40 (m, 1H), 7.37 (d, *J* = 7.8 Hz, 3H), 7.26 (dd, *J* = 7.6, 1.8 Hz, 1H), 2.36 (s, 3H). ¹³C{¹H} NMR (126 MHz, DMSO-*d*₆) δ 165.7, 143.4, 136.2, 136.1, 132.9, 131.7, 129.3, 129.2, 128.0, 127.6, 119.3, 21.1. HRMS (ESI-TOF) calculated for C₁₄H₁₃BrN₂O₃S [M + H]⁺: 390.9728, found: 390.9732.

***N'*-([1,1'-Biphenyl]-2-carbonyl)-4-methylbenzenesulfonohydrazide (3n):** The compound **3n**



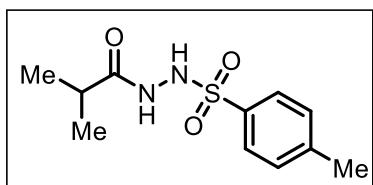
was synthesized using the **GP1**, biphenyl 2-carboxylic acid (**1n**, 1.0 equiv., 0.2 mmol, 39.6 mg), **2a** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (60 mg, 82% yield). ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.43 (s, 1H), 9.88 (s, 1H), 7.68 (d, *J* = 8.4 Hz, 2H), 7.51 (t, *J* = 8.3 Hz, 1H), 7.42 (t, *J* = 7.5 Hz, 2H), 7.38 – 7.28 (m, 8H), 2.35 (s, 3H). ¹³C{¹H} NMR (126 MHz, DMSO-*d*₆) δ 167.9, 143.2, 139.8, 139.7, 136.4, 134.0, 130.1, 129.2, 128.4, 128.3, 128.2, 127.7, 127.3, 127.2, 127.0, 125.5, 21.0. HRMS (ESI-TOF) calculated for C₂₀H₁₉N₂O₃S [M + H]⁺: 367.1116, found: 367.1118.

4-Methyl-N'-(2-phenylacetyl)benzenesulfonohydrazide (3o): The compound **3o** was

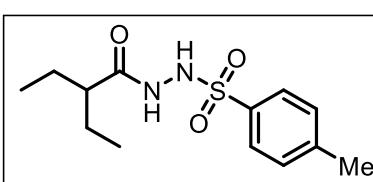


synthesized using the **GP1**, 2-phenylacetic acid (**1o**, 1.0 equiv., 0.2 mmol, 39.6 mg), **2a** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (46 mg, 76% yield). ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.26 (s, 1H), 9.76 (s, 1H), 7.58 (d, *J* = 8.2 Hz, 2H), 7.25 (p, *J* = 7.1 Hz, 5H), 7.10 (d, *J* = 7.9 Hz, 2H), 3.30 (s, 2H), 2.34 (s, 3H). ¹³C{¹H} NMR 126 MHz, DMSO-*d*₆) ¹³C NMR (126 MHz, DMSO) δ 168.71, 143.17, 135.78, 135.32, 129.20, 128.94, 128.15, 127.68, 126.52, 110.95, 40.04, 21.06. HRMS (ESI-TOF) calculated for C₁₅H₁₆N₂NaO₃S [M + Na]⁺: 327.0779, found: 327.0781.

N'-isobutyryl-4-methylbenzenesulfonohydrazide (3p): The compound **3p** was synthesized using the **GP1**, 2-phenylacetic acid (**1p**, 1.0 equiv., 0.2 mmol, 17.6 mg), **2a** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (44 mg, 87% yield). **¹H NMR** (400 MHz, CDCl₃) δ 8.34 (s, 1H), 7.78 (d, *J* = 6.5 Hz, 2H), 7.47 (s, 1H), 7.28 (d, *J* = 6.4 Hz, 2H), 2.40 (s, 3H), 2.33 – 2.25 (m, 1H), 0.95 – 0.91 (m, 6H). **¹³C{¹H} NMR** (101 MHz, CDCl₃) δ 175.64, 145.08, 132.98, 129.61, 128.89, 33.31, 21.80, 19.09.



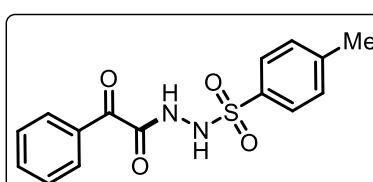
N'-(2-Ethylbutanoyl)-4-methylbenzenesulfonohydrazide (3q): The compound **3q** was



synthesized using the **GP1**, 2-phenylacetic acid (**1q**, 1.0 equiv., 0.2 mmol, 23.2 mg), **2a** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography

(hexane/ethyl acetate = 70:30); white solid (50 mg, 89% yield). **¹H NMR** (400 MHz, CDCl₃) δ 8.39 (s, 1H), 7.76 (d, *J* = 8.4 Hz, 2H), 7.63 (s, 1H), 7.23 (d, *J* = 8.7 Hz, 2H), 2.34 (s, 3H), 1.82 (qd, *J* = 5.4, 2.9 Hz, 1H), 1.37 – 1.23 (m, 4H), 0.58 (t, *J* = 7.4 Hz, 6H). **¹³C{¹H} NMR** (101 MHz, CDCl₃) δ 174.6, 129.6, 128.9, 128.1, 128.0, 48.0, 25.1, 21.7, 11.6.

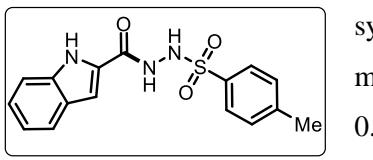
4-Methyl-N'-(2-oxo-2-phenylacetyl) benzenesulfonohydrazide (3r): The compound **3r** was



synthesized using the **GP1**, 4-chlorobenzoic acid (**1p**, 1.0 equiv., 0.2 mmol, 31.3 mg), **2a** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column

chromatography (hexane/ethyl acetate = 70:30); white solid (54.8 mg, 87% yield). **¹H NMR** (500 MHz, DMSO-d₆) δ 11.05 (d, *J* = 3.2 Hz, 1H), 10.27 (d, *J* = 3.2 Hz, 1H), 7.78 (d, *J* = 8.2 Hz, 2H), 7.74 (t, *J* = 6.6 Hz, 1H), 7.65 (d, *J* = 8.4 Hz, 2H), 7.56 (t, *J* = 7.9 Hz, 2H), 7.44 (d, *J* = 8.2 Hz, 2H), 2.42 (s, 3H). **¹³C{¹H} NMR** 126 MHz, DMSO-d₆) δ 189.4, 164.2, 143.8, 135.8, 135.1, 132.2, 129.5, 129.4, 129.1, 127.9, 21.1. HRMS (ESI-TOF) calculated for C₁₅H₁₄N₂NaO₄S [M + Na]⁺: 341.0572, found: 341.0575.

N'-(1*H*-Indole-2-carbonyl)-4-methylbenzenesulfonohydrazide (3s): The compound **3s** was

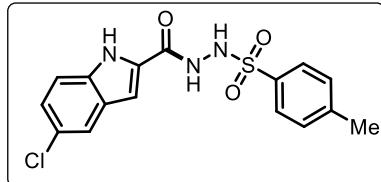


synthesized using the **GP1**, indole 2-carboxylic acid (**1q**, 1.0 equiv., 0.2 mmol, 32.2 mg), **2a** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl

acetate = 70:30); white solid (54 mg, 82% yield). **¹H NMR** (500 MHz, DMSO-d₆) δ 11.58 (s, 1H), 10.66 (s, 1H), 9.98 (s, 1H), 7.74 (d, *J* = 8.2 Hz, 2H), 7.61 (d, *J* = 7.9 Hz, 1H), 7.37 (d, *J* = 8.1 Hz, 1H), 7.33 (d, *J* = 8.4 Hz, 2H), 7.19 (dd, *J* = 13.8, 6.6 Hz, 2H), 7.03 (t, *J* = 7.5 Hz, 1H), 2.36 (s, 3H). **¹³C{¹H} NMR**

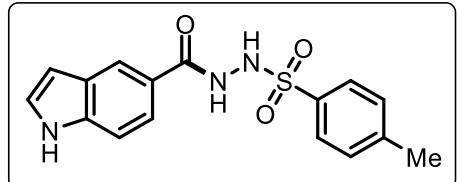
NMR (126 MHz, DMSO-*d*₆) ¹³C NMR (126 MHz, DMSO) δ 160.1, 143.1, 136.6, 136.5, 129.3, 128.0, 127.7, 126.8, 123.8, 121.7, 119.9, 112.3, 103.9, 21.0. HRMS (ESI-TOF) calculated for C₁₆H₁₅N₃NaO₃S [M + Na]⁺: 352.0732, found: 352.0730.

***N'*-(5-Chloro-1*H*-indole-2-carbonyl)-4-methylbenzenesulfonohydrazide (3t):** The



compound **3t** was synthesized using the **GP1**, 5-chloroindole-2 carboxylic acid (**1r**, 1.0 equiv., 0.2 mmol, 39.1 mg), **2a** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (60.4 mg, 83% yield). ¹H NMR (500 MHz, DMSO-*d*₆) δ 11.81 (s, 1H), 10.75 (s, 1H), 10.04 (s, 1H), 7.70 (d, *J* = 3.8 Hz, 2H), 7.35 (dd, *J* = 16.0, 8.5 Hz, 3H), 7.18 (d, *J* = 6.7 Hz, 2H), 7.11 (d, *J* = 7.9 Hz, 1H), 2.35 (s, 3H). ¹³C{¹H} NMR (126 MHz, DMSO-*d*₆) δ 159.8, 143.2, 136.5, 135.0, 130.2, 129.3, 127.7, 127.5, 124.4, 123.9, 120.8, 113.9, 103.40, 21.0. HRMS (ESI-TOF) calculated for C₁₆H₁₄N₃NaO₃SCl [M + Na]⁺: 386.0342, found: 386.0340.

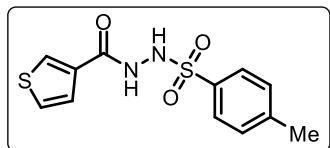
***N'*-(1*H*-Indole-5-carbonyl)-4-methylbenzenesulfonohydrazide (3u):** The compound **3u** was



synthesized using the **GP1**, indole 5-carboxylic acid (**1s**, 1.0 equiv., 0.2 mmol, 32.2 mg), **2a** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified

by column chromatography (hexane/ethyl acetate = 70:30); white solid (56.5 mg, 86% yield). ¹H NMR (500 MHz, DMSO-*d*₆) δ 11.75 (s, 1H), 10.64 (s, 1H), 9.84 (s, 1H), 7.89 (s, 1H), 7.73 (d, *J* = 8.2 Hz, 3H), 7.66 (d, *J* = 2.6 Hz, 1H), 7.53 (d, *J* = 8.5 Hz, 1H), 7.44 (d, *J* = 8.5 Hz, 1H), 7.32 (d, *J* = 8.2 Hz, 2H), 2.34 (s, 3H). ¹³C{¹H} NMR (126 MHz, DMSO-*d*₆) δ 166.7, 143.1, 142.9, 136.4, 129.5, 129.2, 127.8, 127.7, 126.9, 120.6, 120.5, 111.1, 102.3, 21.0. HRMS (ESI-TOF) calculated for C₁₆H₁₅N₃NaO₃S [M + Na]⁺: 352.0732, found: 352.0729.

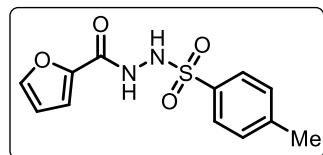
4-Methyl-N'-(thiophene-3-carbonyl)benzenesulfonohydrazide (3v): The compound **3v** was



synthesized using the **GP1**, thiophene 3-carboxylic acid (**1t**, 1.0 equiv., 0.2 mmol, 25.6 mg), **2a** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in

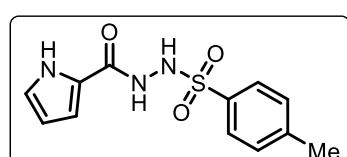
DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (50.3 mg, 85% yield). ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.49 (s, 1H), 9.88 (s, 1H), 8.14 (s, 1H), 7.71 (d, *J* = 8.4 Hz, 2H), 7.58 – 7.52 (m, 1H), 7.41 – 7.38 (m, 1H), 7.33 (d, *J* = 8.2 Hz, 2H), 2.35 (s, 3H). ¹³C{¹H} NMR (126 MHz, DMSO-*d*₆) ¹³C NMR (126 MHz, DMSO) δ 160.9, 143.2, 136.4, 134.6, 130.0, 129.3, 127.7, 127.04, 126.7, 21.1. HRMS (ESI-TOF) calculated for C₁₂H₁₂N₂NaO₃S₂ [M + Na]⁺: 319.0187, found: 319.0185.

N'-(Furan-2-carbonyl)-4-methylbenzenesulfonohydrazide (3w): The compound **3w** was



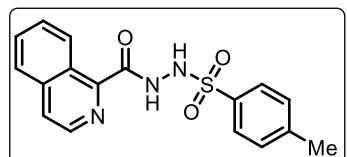
synthesized using the **GP1**, 2-furoic acid (**1u**, 1.0 equiv., 0.2 mmol, 22.4 mg), **2a** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (46.4 mg, 83% yield). **¹H NMR** (500 MHz, DMSO-*d*₆) δ 10.57 (s, 1H), 9.97 (s, 1H), 7.83 (s, 1H), 7.70 (d, *J* = 8.1 Hz, 2H), 7.37 – 7.31 (m, 2H), 7.18 (s, 1H), 6.60 (s, 1H), 2.36 (s, 3H). **¹³C{¹H} NMR** (126 MHz, DMSO-*d*₆) δ 156.9, 146.1, 145.5, 143.3, 136.4, 129.4, 127.7, 115.0, 111.9, 21.1. HRMS (ESI-TOF) calculated for C₁₂H₁₃N₂O₄S [M + H]⁺: 281.0596, found: 281.0591.

4-Methyl-N'-(1*H*-pyrrole-2-carbonyl)benzenesulfonohydrazide (3x): The compound **3x** was



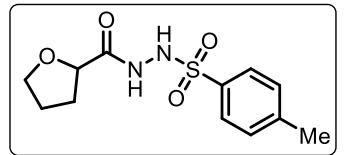
synthesized using the **GP1**, pyrrole-2-carboxylic acid (**1v**, 1.0 equiv., 0.2 mmol, 22.2 mg), **2a** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (47 mg, 86% yield). **¹H NMR** (500 MHz, DMSO-*d*₆) δ 11.50 (s, 1H), 10.16 (s, 1H), 9.79 (s, 1H), 7.70 (d, *J* = 7.9 Hz, 2H), 7.31 (d, *J* = 7.8 Hz, 2H), 6.85 (s, 2H), 6.07 (s, 1H), 2.36 (s, 3H). **¹³C{¹H} NMR** (126 MHz, DMSO-*d*₆) δ 159.8, 143.4, 136.7, 129.5, 127.9, 124.7, 123.5, 122.9, 109.2, 21.3. HRMS (ESI-TOF) calculated for C₁₂H₁₄N₃O₃S [M + H]⁺: 280.0756, found: 280.0750.

N'-(Isoquinoline-1-carbonyl)-4-methylbenzenesulfonohydrazide (3y): The compound **3y**



was synthesized using the **GP1**, 4-chlorobenzoic acid (**1w**, 1.0 equiv., 0.2 mmol, 34.6 mg), **2a** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (54.5 mg, 80% yield). **¹H NMR** (500 MHz, DMSO-*d*₆) δ 10.80 (s, 1H), 10.30 (s, 1H), 8.52 (d, *J* = 5.5 Hz, 1H), 8.07 – 8.00 (m, 2H), 7.98 (d, *J* = 5.5 Hz, 1H), 7.84 – 7.78 (m, 3H), 7.68 – 7.63 (m, 1H), 7.39 (d, *J* = 8.4 Hz, 2H), 2.39 (s, 3H). **¹³C{¹H} NMR** (126 MHz, DMSO-*d*₆) δ 165.1, 151.0, 143.5, 141.1, 136.2, 136.1, 130.9, 129.4, 128.3, 128.0, 127.2, 125.6, 125.1, 123.2, 21.1. HRMS (ESI-TOF) calculated for C₁₇H₁₆N₃O₃S [M + H]⁺: 342.0912, found: 342.0914.

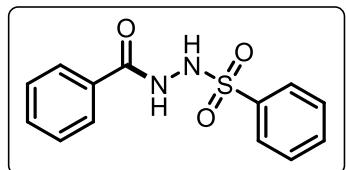
4-Methyl-N'-(tetrahydrofuran-2-carbonyl)benzenesulfonohydrazide (3z): The compound



3z was synthesized using the **GP1**, tetrahydrofuran 2-carboxylic acid (**1x**, 1.0 equiv., 0.2 mmol, 23.4 mg), **2a** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (48.4 mg, 86% yield). **¹H NMR** (500 MHz, DMSO-*d*₆) δ 9.97 (s, 1H), 9.68 (s, 1H),

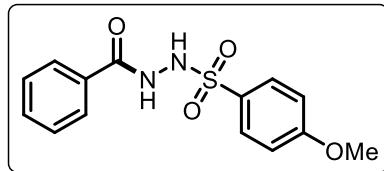
7.67 (d, $J = 8.2$ Hz, 2H), 7.34 (d, $J = 8.2$ Hz, 2H), 4.11 (s, 1H), 3.85 – 3.56 (m, 3H), 2.37 (s, 3H), 2.01 (s, 1H), 1.79 – 1.66 (m, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, DMSO- d_6) δ 171.4, 143.3, 136.0, 129.3, 127.8, 76.4, 68.7, 29.6, 24.9, 21.1. HRMS (ESI-TOF) calculated for $\text{C}_{12}\text{H}_{17}\text{N}_2\text{O}_4\text{S} [\text{M} + \text{H}]^+$: 285.0909, found: 285.0905.

***N'*-Benzoylbenzenesulfonohydrazide (3aa):** The compound **3aa** was synthesized using the **GP1**,



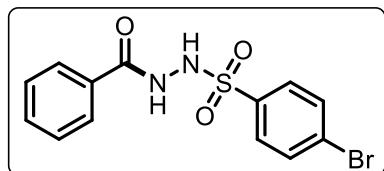
benzoic acid (**1a**, 1.0 equiv., 0.2 mmol, 24.4 mg), **2b** (1.2 equiv., 0.24 mmol, 40.8 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh_3 (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (49.6 mg, 90% yield). ^1H NMR (500 MHz, DMSO- d_6) δ 10.34 (s, 2H), 7.85 (d, $J = 7.6$ Hz, 2H), 7.68 (d, $J = 7.3$ Hz, 2H), 7.61 (d, $J = 7.5$ Hz, 1H), 7.53 (t, $J = 6.8$ Hz, 3H), 7.43 (t, $J = 7.7$ Hz, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, DMSO- d_6) δ 165.6, 139.1, 133.0, 132.1, 132.0, 128.8, 128.5, 127.7, 127.5.

***N'*-Benzoyl-4-methoxybenzenesulfonohydrazide (3ab):** The compound **3ab** was synthesized



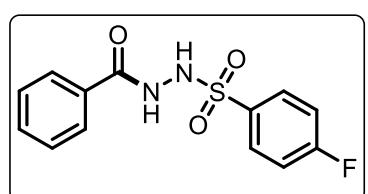
using the **GP1**, **1a** (1.0 equiv., 0.2 mmol, 24.4 mg), 4-methoxy benzenesulfonyl hydrazide **2c** (1.2 equiv., 0.24 mmol, 48.4 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh_3 (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (55.6 mg, 91% yield). ^1H NMR (500 MHz, DMSO- d_6) δ 10.65 (d, $J = 3.7$ Hz, 1H), 9.81 (d, $J = 3.7$ Hz, 1H), 7.76 (d, $J = 9.0$ Hz, 2H), 7.68 (d, $J = 7.5$ Hz, 2H), 7.53 (t, $J = 7.4$ Hz, 1H), 7.43 (t, $J = 7.7$ Hz, 2H), 7.04 (d, $J = 8.9$ Hz, 2H), 3.79 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, DMSO- d_6) δ 165.6, 162.7, 132.1, 131.9, 130.5, 130.0, 128.5, 127.5, 114.1, 55.7.

***N'*-Benzoyl-4-bromobenzenesulfonohydrazide (3ac):** The compound **3ac** was synthesized using



the **GP1**, **1a** (1.0 equiv., 0.2 mmol, 24.4 mg), 4-bromo benzene sulfonyl hydrazide **2d** (1.2 equiv., 0.24 mmol, 60.2 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh_3 (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (62.4 mg, 88% yield). ^1H NMR (500 MHz, DMSO- d_6) δ 10.71 (s, 1H), 10.17 (s, 1H), 7.76–7.67 (m, 4H), 7.68 (d, $J = 7.8$ Hz, 2H), 7.53 (t, $J = 7.5$ Hz, 1H), 7.43 (t, $J = 7.7$ Hz, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, DMSO- d_6) δ 165.7, 138.6, 132.1, 132.0, 131.9, 129.7, 128.6, 127.5, 127.0, 79.2.

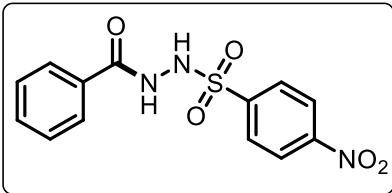
***N'*-Benzoyl-4-fluorobenzenesulfonohydrazide (3ad):** The compound **3ad** was synthesized using



the **GP1**, benzoic acid **1a** (1.0 equiv., 0.2 mmol, 24.4 mg), 4-fluoro benzene sulfonyl hydrazide **2e** (1.2 equiv., 0.24 mmol, 45.6 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh_3 (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography

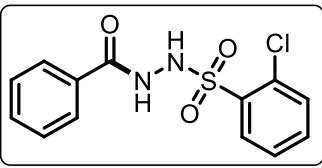
(hexane/ethyl acetate = 70:30); white solid (49.9 mg, 85% yield). **¹H NMR** (500 MHz, DMSO-*d*₆) δ 10.71 (d, *J* = 3.2 Hz, 1H), 10.09 (d, *J* = 3.4 Hz, 1H), 7.92 – 7.87 (m, 2H), 7.69 (d, *J* = 8.1 Hz, 2H), 7.53 (t, *J* = 7.5 Hz, 1H), 7.43 (t, *J* = 7.6 Hz, 2H), 7.37 (t, *J* = 8.9 Hz, 2H). **¹³C{¹H} NMR** (126 MHz, DMSO-*d*₆) δ 164.6 (d, *J* = 249.9), 135.5, 132.0 (d, *J* = 13.5), 130.8 (d, *J* = 9.9), 128.5, 127.5, 116.0 (d, *J* = 22.7). HRMS (ESI-TOF) calculated for C₁₃H₁₂N₂O₃SF [M + H]⁺: 295.0553, found: 295.0555.

***N'*-Benzoyl-4-nitrobenzenesulfonohydrazide (3ae):** The compound 3ae was synthesized using



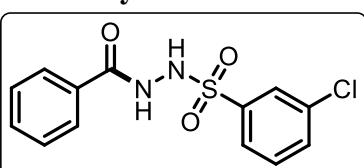
the **GP1**, **1a** (1.0 equiv., 0.2 mmol, 24.4 mg), 4-nitrobenzene sulfonyl hydrazide **2f** (1.2 equiv., 0.24 mmol, 52.8 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (52 mg, 81% yield). **¹H NMR** (500 MHz, DMSO-*d*₆) δ 10.82 (s, 1H), 10.51 (d, *J* = 2.9 Hz, 1H), 8.38 (d, *J* = 6.7 Hz, 2H), 8.11 (d, *J* = 8.9 Hz, 2H), 7.70 (d, *J* = 7.3 Hz, 2H), 7.55 (t, *J* = 7.5 Hz, 1H), 7.45 (t, *J* = 7.7 Hz, 2H). **¹³C{¹H} NMR** (126 MHz, DMSO-*d*₆) δ 165.9, 149.9, 145.2, 132.2, 131.7, 129.3, 128.5, 127.5, 124.2.

***N'*-Benzoyl-2-chlorobenzenesulfonohydrazide (3af):** The compound 3af was synthesized using



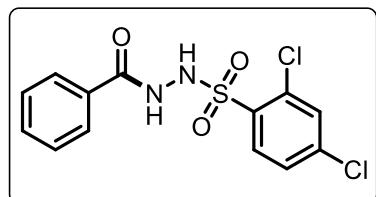
the **GP1**, **1a** (1.0 equiv., 0.2 mmol, 24.4 mg), 2-chloro benzene sulfonyl hydrazide **2g** (1.2 equiv., 0.24 mmol, 49.44 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (52.7 mg, 85% yield). **¹H NMR** (500 MHz, DMSO-*d*₆) δ 10.67 (s, 1H), 10.11 (s, 1H), 7.99 (d, *J* = 8.1 Hz, 1H), 7.69 (d, *J* = 7.8 Hz, 2H), 7.62 (dt, *J* = 15.3, 7.8 Hz, 2H), 7.53 (t, *J* = 7.4 Hz, 1H), 7.44 (q, *J* = 7.0 Hz, 3H). **¹³C{¹H} NMR** (126 MHz, DMSO-*d*₆) δ 165.8, 137.2, 134.3, 132.1, 132.0, 131.9, 131.7, 131.1, 128.4, 127.5, 127.1.

***N'*-Benzoyl-3-chlorobenzenesulfonohydrazide (3ag):** The compound 3ag was synthesized using



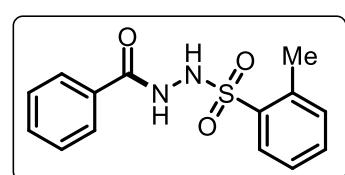
the **GP1**, **1a** (1.0 equiv., 0.2 mmol, 24.4 mg), 2-chloro benzene sulfonyl hydrazide **2h** (1.2 equiv., 0.24 mmol, 49.44 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (52.7 mg, 85% yield). **¹H NMR** (500 MHz, DMSO-*d*₆) δ 10.58 (s, 1H), 7.83 (s, 1H), 7.78 (d, *J* = 7.9 Hz, 1H), 7.70 (t, *J* = 8.6 Hz, 3H), 7.58 – 7.52 (m, 2H), 7.44 (t, *J* = 7.7 Hz, 2H). **¹³C{¹H} NMR** (126 MHz, DMSO-*d*₆) δ 165.7, 141.2, 133.5, 132.9, 132.1, 131.9, 130.9, 128.5, 127.5, 127.3, 126.4. HRMS (ESI-TOF) calculated for C₁₃H₁₂ClN₂O₃S [M + H]⁺: 311.0257, found: 311.0253.

N'-Benzoyl-2,4-dichlorobenzenesulfonohydrazide (3ah): The compound **3ah** was synthesized



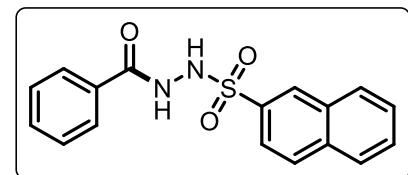
using the **GP1**, **1a** (1.0 equiv., 0.2 mmol, 24.4 mg), **2i** (1.2 equiv., 0.24 mmol, 57.8 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (58.6 mg, 85% yield). ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.62 (s, 2H), 7.96 (d, *J* = 8.5 Hz, 1H), 7.85 (s, 1H), 7.69 (d, *J* = 7.6 Hz, 2H), 7.57 – 7.51 (m, 2H), 7.44 (t, *J* = 7.7 Hz, 2H). ¹³C{¹H} NMR (126 MHz, DMSO-*d*₆) δ 165.9, 138.3, 136.4, 133.5, 132.5, 132.1, 131.8, 131.2, 128.5, 127.5, 127.3. HRMS (ESI-TOF) calculated for C₁₃H₁₀N₂NaO₃SCl₂ [M + Na]⁺: 366.9687, found: 366.9688.

N'-Benzoyl-2-methylbenzenesulfonohydrazide (3ai): The compound **3ai** was synthesized using



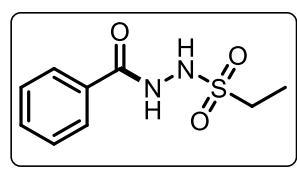
the **GP1**, **1a** (1.0 equiv., 0.2 mmol, 24.4 mg), 2-methyl benzene sulfonyl hydrazide **2j** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (54.6 mg, 89% yield). ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.68 (s, 1H), 9.96 (s, 1H), 7.73 – 7.62 (m, 5H), 7.54 (t, *J* = 7.4 Hz, 1H), 7.46 – 7.42 (m, 3H), 2.33 (s, 3H). ¹³C{¹H} NMR (126 MHz, DMSO-*d*₆) δ 165.6, 138.9, 138.4, 133.6, 132.1, 131.9, 128.7, 128.4, 127.9, 127.5, 124.8, 20.8. HRMS (ESI-TOF) calculated for C₁₄H₁₄N₂NaO₃S [M + Na]⁺: 313.0623, found: 313.0626.

N'-Benzoylnaphthalene-2-sulfonohydrazide (3aj): The compound **3aj** was synthesized using



the **GP1**, **1a** (1.0 equiv., 0.2 mmol, 24.4 mg), naphthalene 2-sulfonyl hydrazide **2k** (1.2 equiv., 0.24 mmol, 53.2 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (58.6 mg, 90% yield). ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.75 (s, 1H), 10.16 (s, 1H), 8.51 (s, 1H), 8.12 (d, *J* = 7.8 Hz, 1H), 8.08 (d, *J* = 8.9 Hz, 1H), 8.01 (d, *J* = 8.1 Hz, 1H), 7.91 (d, *J* = 8.7 Hz, 1H), 7.66 (d, *J* = 9.3 Hz, 3H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.51 (t, *J* = 7.4 Hz, 1H), 7.41 (t, *J* = 7.6 Hz, 2H). ¹³C{¹H} NMR (126 MHz, DMSO-*d*₆) δ 165.7, 136.4, 134.5, 132.0, 131.9, 131.7, 129.3, 128.9, 128.8, 128.7, 128.5, 127.8, 127.5, 127.4, 123.4. HRMS (ESI-TOF) calculated for C₁₇H₁₄N₂NaO₃S [M + Na]⁺: 349.0623, found: 349.0625.

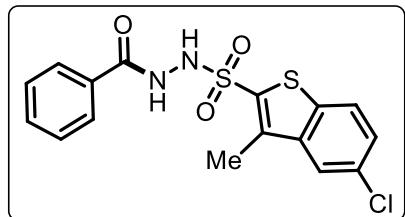
N'-Benzoylethanesulfonohydrazide (3ak): The compound **3ak** was synthesized using the **GP1**,



1a (1.0 equiv., 0.2 mmol, 24.4 mg), ethyl sulfonyl hydrazide **2l** (1.2 equiv., 0.24 mmol, 29.7 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (41.4 mg, 90% yield). ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.65 (s, 1H), 9.55 (s, 1H), 7.87 (d, *J* = 7.3 Hz, 2H), 7.59 (t,

$J = 7.4$ Hz, 1H), 7.50 (t, $J = 7.7$ Hz, 2H), 3.10 (q, $J = 7.3$ Hz, 2H), 1.32 (t, $J = 7.3$ Hz, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, DMSO- d_6) δ 166.2, 132.1, 131.9, 128.5, 127.6, 46.4, 7.9. HRMS (ESI-TOF) calculated for $\text{C}_9\text{H}_{13}\text{N}_2\text{O}_3\text{S}$ [M + H] $^+$: 229.0647, found: 229.0649.

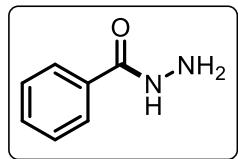
***N'*-Benzoyl-5-chloro-3-methylbenzo[*b*]thiophene-2-sulfonohydrazide (3al):** The compound



3al was synthesized using the **GP1**, benzoic acid (**1a**, 1.0 equiv., 0.2 mmol, 24.4 mg), **2m** (1.2 equiv., 0.24 mmol, 44.70 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 40:60); white solid (60.8

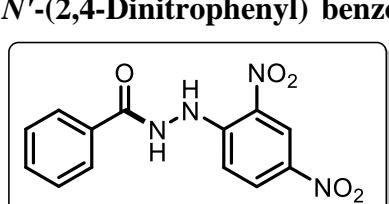
mg, 80% yield). ^1H NMR (500 MHz, DMSO- d_6) δ 10.79 (s, 1H), 10.50 (s, 1H), 8.05 (d, $J = 8.7$ Hz, 2H), 7.68 (d, $J = 7.2$ Hz, 2H), 7.53 (t, $J = 8.5$ Hz, 2H), 7.43 (t, $J = 7.7$ Hz, 2H), 2.66 (s, 3H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, DMSO- d_6) δ 165.8, 140.7, 137.8, 137.6, 136.1, 132.1, 131.8, 130.3, 128.5, 127.44, 127.41, 124.6, 123.5, 12.4. HRMS (ESI-TOF) calculated for $\text{C}_{16}\text{H}_{14}\text{N}_2\text{O}_3\text{S}_2\text{Cl}$ [M + H] $^+$: 381.0134, found: 381.0132.

Benzohydrazide (3am): The compound **3am** was synthesized using the **GP1**, benzoic acid (**1a**, 1.0



equiv., 0.2 mmol, 24.4 mg), **2n** (1.2 equiv., 0.24 mmol, 39 μL), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 40:60); white solid (25.8 mg, 95% yield). ^1H NMR (500 MHz, DMSO- d_6) δ 10.52 (s, 1H), 7.94 (d, $J = 8.5$ Hz, 2H), 7.63 – 7.59 (m, 1H), 7.54 (t, $J = 7.6$ Hz, 2H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, DMSO- d_6) δ 165.9, 132.6, 131.9, 128.6, 127.5. HRMS (ESI-TOF) calculated for $\text{C}_7\text{H}_8\text{N}_2\text{NaO}$ [M + Na] $^+$: 159.0534, found: 159.0531.

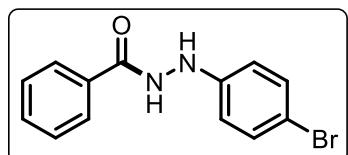
***N'*-(2,4-Dinitrophenyl) benzohydrazide (3an):** The compound **3an** was synthesized using the



GP1, benzoic acid **1a** (1.0 equiv., 0.2 mmol, 24.4 mg), **2o** (1.2 equiv., 0.24 mmol, 47.5 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 40:60);

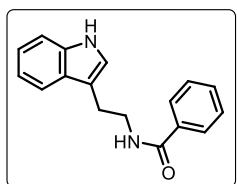
white solid (60.4 mg, 83% yield). ^1H NMR (500 MHz, DMSO- d_6) δ 11.08 (s, 1H), 10.28 (s, 1H), 8.89 (d, $J = 2.6$ Hz, 1H), 8.32 (dd, $J = 9.6, 2.7$ Hz, 1H), 7.98 (d, $J = 7.2$ Hz, 2H), 7.67 – 7.62 (m, 1H), 7.56 (t, $J = 7.6$ Hz, 2H), 7.33 (d, $J = 9.5$ Hz, 1H). $^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, DMSO- d_6) δ 166.1, 148.7, 136.8, 132.4, 131.9, 130.2, 129.8, 128.7, 127.7, 123.2, 115.6. HRMS (ESI-TOF) calculated for $\text{C}_{13}\text{H}_{10}\text{N}_4\text{NaO}_5$ [M + Na] $^+$: 325.0549, found: 325.0542.

N'-(4-Bromophenyl) benzohydrazide (3ao): The compound **3ao** was synthesized using the **GP1**,



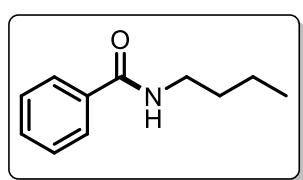
benzoic acid (**1a**, 1.0 equiv., 0.2 mmol, 24.4 mg), **2p** (1.2 equiv., 0.24 mmol, 53.5 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 40:60); white solid (50 mg, 86% yield). ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.39 (s, 1H), 8.11 (s, 1H), 7.91 (d, *J* = 7.8 Hz, 1H), 7.66 (s, 1H), 7.59 (d, *J* = 7.5 Hz, 2H), 7.51 (d, *J* = 7.5 Hz, 1H), 7.39 (d, *J* = 5.8 Hz, 2H), 7.30 (d, *J* = 6.9 Hz, 1H), 6.74 (d, *J* = 7.2 Hz, 1H). ¹³C{¹H} NMR (126 MHz, DMSO-*d*₆) ¹³C NMR (126 MHz, DMSO) δ 166.4, 148.9, 131.7, 131.4, 129.3, 128.7, 128.5, 127.3, 114.3. HRMS (ESI-TOF) calculated for C₁₃H₁₁N₂NaOSBr [M + Na]⁺: 312.9952, found: 312.9955.

N-(2-(1*H*-Indol-3-yl) ethyl)benzamide (3ap): The compound **3ap** was synthesized using the



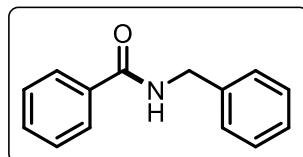
GP1, benzoic acid (**1a**, 1.0 equiv., 0.2 mmol, 24.4 mg), tryptamine (1.2 equiv., 0.24 mmol, 38.4 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 40:60); white solid (45.4 mg, 86% yield). ¹H NMR (500 MHz, CDCl₃) δ 8.38 (s, 1H), 7.71 – 7.60 (m, 3H), 7.46 (t, *J* = 7.4 Hz, 1H), 7.41 – 7.34 (m, 3H), 7.21 (t, *J* = 7.6 Hz, 1H), 7.13 (t, *J* = 7.5 Hz, 1H), 7.03 (s, 1H), 6.33 (s, 1H), 3.80 (q, *J* = 6.3 Hz, 2H), 3.09 (t, *J* = 6.7 Hz, 2H). ¹³C{¹H} NMR (126 MHz, CDCl₃) δ 167.7, 136.6, 134.8, 131.5, 128.6, 127.4, 126.9, 122.3, 119.6, 118.8, 112.9, 111.5, 40.4, 25.4.

N-Butyl benzamide (3aq): The compound **3aq** was synthesized using the **GP1**, benzoic acid (**1a**,



1.0 equiv., 0.2 mmol, 24.4 mg), *N*-butyl amine (1.2 equiv., 0.24 mmol, 38.4 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 60:40); white solid (21 mg, 58% yield). ¹H NMR (500 MHz, CDCl₃) δ 7.78 (d, *J* = 7.0 Hz, 2H), 7.51 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.5 Hz, 2H), 6.16 (s, 1H), 3.48 (q, *J* = 7.2 Hz, 2H), 1.63 (p, *J* = 7.6 Hz, 2H), 1.45 (p, *J* = 7.2 Hz, 2H), 0.98 (t, *J* = 7.3 Hz, 3H). ¹³C{¹H} NMR (126 MHz, CDCl₃) δ 167.67, 135.00, 131.41, 128.65, 126.92, 39.92, 31.86, 20.27, 13.88.

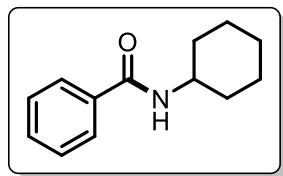
N-Benzyl benzamide (3ar): The compound **3ar** was synthesized using the **GP1**, benzoic acid (**1a**,



1.0 equiv., 0.2 mmol, 24.4 mg), *N*-benzyl amine (1.2 equiv., 0.24 mmol, 38.4 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 60:40); white solid (31 mg, 71% yield). ¹H NMR (500 MHz, CDCl₃) δ 7.79 (d, *J* = 8.5 Hz, 2H), 7.50 (t, *J* = 7.4 Hz, 1H), 7.42 (t, *J* = 7.5 Hz, 2H), 7.35 (d, *J* = 4.4 Hz, 4H), 7.30 (q, *J* = 4.7 Hz, 1H), 6.50 (s, 1H), 4.64 (d, *J* = 5.8 Hz, 2H).

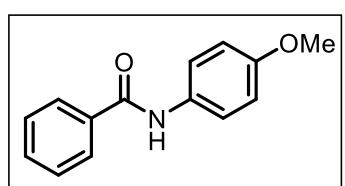
¹³C{¹H} NMR (126 MHz, CDCl₃) δ 167.51, 138.31, 134.51, 131.67, 128.91, 128.72, 128.04, 127.75, 127.09, 44.27.

N-Cyclohexyl benzamide (3as): The compound **3as** was synthesized using the **GP1**, benzoic acid



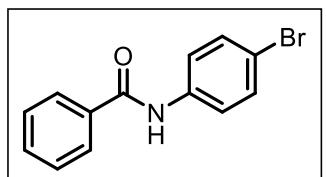
(**1a**, 1.0 equiv., 0.2 mmol, 24.4 mg), *N*-cyclohexylamine (1.2 equiv., 0.24 mmol, 38.4 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 60:40); white solid (28 mg, 68% yield). ¹H NMR (500 MHz, CDCl₃) δ 7.77 (d, *J* = 8.7 Hz, 2H), 7.52 – 7.47 (m, 1H), 7.43 (t, *J* = 7.6 Hz, 2H), 6.06 (s, 1H), 4.00 (d, *J* = 15.0 Hz, 1H), 2.05 (d, *J* = 13.0 Hz, 2H), 1.77 (d, *J* = 17.5 Hz, 2H), 1.67 (d, *J* = 17.2 Hz, 1H), 1.44 (q, *J* = 13.8 Hz, 2H), 1.31 – 1.25 (m, 3H). ¹³C{¹H} NMR (126 MHz, CDCl₃) δ 166.78, 135.24, 131.35, 128.62, 126.95, 48.81, 33.35, 25.70, 25.04.

N-(4-Methoxyphenyl) benzamide (3au): The compound **3au** was synthesized using the **GP1**,



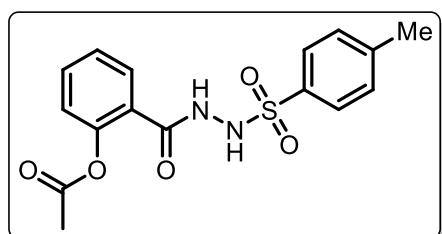
benzoic acid (**1a**, 1.0 equiv., 0.2 mmol, 24.4 mg), *p*-anisidine (1.2 equiv., 0.24 mmol, 29.5 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 60:40); white solid (36 mg, 80% yield). ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.10 (s, 1H), 7.90 (d, *J* = 8.3 Hz, 2H), 7.64 (d, *J* = 9.2 Hz, 2H), 7.57 – 7.44 (m, 3H), 6.89 (d, *J* = 9.2 Hz, 2H), 3.70 (s, 3H). ¹³C{¹H} NMR (101 MHz, DMSO-*d*₆) δ 165.14, 155.57, 135.08, 132.26, 131.43, 128.40, 127.58, 122.00, 113.76, 55.19.

N-(4-Bromophenyl) benzamide (3av): The compound **3av** was synthesized using the **GP1**,



benzoic acid (**1a**, 1.0 equiv., 0.2 mmol, 24.4 mg), *p*-bromoaniline (1.2 equiv., 0.24 mmol, 41.2 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 60:40); white solid (46 mg, 82% yield). ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.39 (s, 1H), 7.98 – 7.93 (m, 2H), 7.81 – 7.76 (m, 2H), 7.62 – 7.57 (m, 1H), 7.55 – 7.52 (m, 3H). ¹³C{¹H} NMR (101 MHz, DMSO-*d*₆) δ 165.72, 138.63, 134.75, 131.75, 131.47, 128.47, 128.46, 128.44, 127.74, 122.24, 115.38.

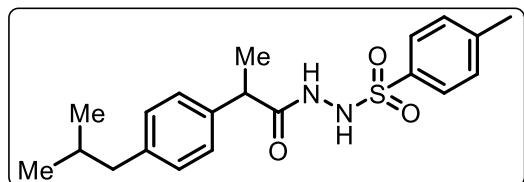
2-(2-Tosylhydrazine-1-carbonyl) phenyl acetate (4a): The compound **4a** was synthesized using



the **GP1**, Aspirin (1.0 equiv., 0.2 mmol, 36.03 mg), **2a** (1.2 equiv., 0.24 mmol, 44.6 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 50:50); white solid (42.2 mg, 85% yield). ¹H NMR

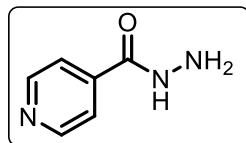
(500 MHz, DMSO-*d*₆) δ 11.33 (s, 1H), 11.20 (s, 1H), 7.96 (s, 2H), 7.84 (d, *J* = 9.6 Hz, 1H), 7.46 (dd, *J* = 16.8, 6.9 Hz, 3H), 7.06 – 6.95 (m, 2H), 2.41 (s, 3H), 2.05 (s, 3H). ¹³C{¹H} NMR (126 MHz, DMSO-*d*₆) δ 170.7, 167.1, 157.9, 144.9, 135.4, 134.4, 129.7, 129.2, 129.2, 127.7, 119.4, 117.1, 115.9, 22.2, 21.2. HRMS (ESI-TOF) calculated for C₁₆H₁₆N₂NaO₅S [M + Na]⁺: 371.0678, found: 371.0672.

***N'*-(2-(4-Isobutylphenyl)propanoyl)-4-methylbenzenesulfonohydrazide (4b):** The



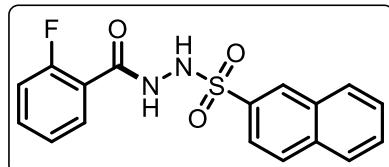
compound **4b** was synthesized using the **GP1**, ibuprofen (1.0 equiv., 0.2 mmol, 41.2 mg), **2a** (1.2 equiv., 0.24 mmol, 44.6 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 50:50); white solid (65.8 mg, 88% yield). ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.22 (s, 1H), 9.69 (s, 1H), 7.51 (d, *J* = 7.9 Hz, 2H), 7.20 (d, *J* = 7.9 Hz, 2H), 7.05 (s, 4H), 3.51 (q, *J* = 7.0 Hz, 1H), 2.42 (d, *J* = 7.0 Hz, 2H), 2.35 (s, 3H), 1.82 (dt, *J* = 14.0, 7.0 Hz, 1H), 1.35 (d, *J* = 7.1 Hz, 1H), 1.16 (d, *J* = 7.0 Hz, 3H), 0.87 (d, *J* = 6.4 Hz, 6H). ¹³C{¹H} NMR (126 MHz, DMSO-*d*₆) δ 171.9, 142.9, 139.4, 138.3, 135.7, 129.0, 128.9, 128.6, 127.7, 127.1, 127.0, 44.3, 42.3, 29.6, 29.6, 22.2, 22.1, 21.0, 17.9. HRMS (ESI-TOF) calculated for C₂₀H₂₆N₂NaO₃S [M + Na]⁺: 397.1562, found: 397.1566.

Isoniazide (5): The compound **5** was synthesized using the **GP1**, isonicotinic acid (1.0 equiv., 0.2



mmol, 24.6 mg), NH₂NH₂.H₂O (1.2 equiv., 0.24 mmol, 39 μL), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 40:60); white solid (21.8 mg, 80% yield). ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.09 (s, 1H), 8.69 (s, 2H), 7.72 (s, 2H), 4.64 (s, 1H). ¹³C{¹H} NMR (126 MHz, DMSO-*d*₆) δ 164.0, 150.2, 140.3, 121.1 HRMS (ESI-TOF) calculated for C₈H₈N₃O [M + H]⁺: 138.0667, found: 138.0673.

***N'*-(2-fluorobenzoyl)naphthalene-2-sulfonohydrazide (6):** The compound **6** was synthesized



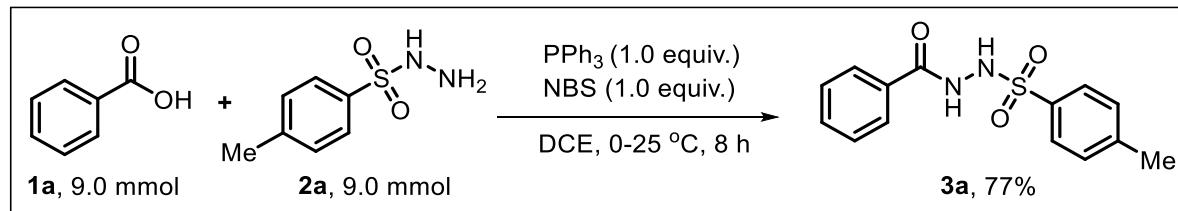
using the **GP1**, 2-fluoro benzoic acid (1.0 equiv., 0.2 mmol, 28 mg), **2k** (1.2 equiv., 0.24 mmol, 53.3 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (55 mg, 80% yield). ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.64 (s, 1H), 10.26 (s, 1H), 8.55 (s, 1H), 8.15 (d, *J* = 8.2 Hz, 1H), 8.10 (d, *J* = 8.5 Hz, 1H), 8.02 (d, *J* = 8.2 Hz, 1H), 7.94 (d, *J* = 8.9 Hz, 1H), 7.66 (dd, *J* = 13.9, 7.6 Hz, 2H), 7.53 – 7.46 (m, 1H), 7.40 (t, *J* = 7.5 Hz, 1H), 7.23 (t, *J* = 8.8 Hz, 2H). ¹³C{¹H} NMR (126 MHz, DMSO-*d*₆) δ 163.1, δ 159.05 (d, *J* = 250.5 Hz), 136.1, 134.6, 133.00 (d, *J* = 8.4 Hz), 131.7, 129.81 (d, *J* = 2.7 Hz), 129.2, 128.89 (d, *J* = 5.6 Hz), 127.8, 127.4, 124.46 (d, *J* = 3.6 Hz), 123.7, 123.3, 116.15 (d, *J* = 21.6 Hz). HRMS (ESI-TOF) calculated for C₁₇H₁₃N₂KFO₃S [M + K]⁺: 383.0268, found: 313.0261.

N'-Isopropylbenzohydrazide (7): The compound **7** was synthesized using the **GP1**, 2-fluoro benzoic acid (1.0 equiv., 0.2 mmol, 28 mg), isopropylhydrazine (1.2 equiv., 0.24 mmol, 53.3 mg), NBS (1.0 equiv., 0.2 mmol, 35.5 mg) and PPh₃ (1.0 equiv., 0.2 mmol, 52.4 mg) in DCE (1.0 mL) and was purified by column chromatography (hexane/ethyl acetate = 70:30); white solid (27 mg, 78% yield). ¹H NMR (500 MHz, DMSO-d₆) δ 9.98 (s, 1H), 7.85 (d, *J* = 7.5 Hz, 1H), 7.52 (d, *J* = 7.6 Hz, 2H), 7.46 (t, *J* = 7.6 Hz, 2H), 3.07 (h, *J* = 6.3 Hz, 1H), 1.03 (d, *J* = 6.4 Hz, 6H). ¹³C{¹H} NMR (126 MHz, DMSO) δ 165.62, 133.22, 131.21, 128.29, 127.09, 50.40, 20.92.

5. Synthetic utility and Mechanistic studies

5.1 Gram-Scale Synthesis:

In a reaction vial (10.0 mL), triphenylphosphine (9.0 mmol, 1.0 equiv.), *N*-Bromosuccinamide (9.0 mmol, 1.0 equiv.) was taken, followed by adding 1.0 mL DCE, and stirred for 5 min at 0 °C. After that, Benzoic acid (**1a**, 9.0 mmol, 1.0 equiv.) was added to it and again kept for stirring for 15 min., and finally, *p*-toluene hydrazide derivative (**2a**, 10.8 mmol, 1.2 equiv.) was also added. The reaction was stirred at 0 °C to rt for 8 hours. A TLC plate monitored the completion of the reaction in 30 % EtOAc in hexane. The crude was purified by column chromatography and eluted with hexane/EtOAc to afford the desired products. The product was obtained with a 77% yield.



5.2 Control Experiments:

Identification of reaction intermediates by NMR spectroscopic studies

The following control experiments were conducted to understand the current transformation process.

Control experiment 1

On a benchtop (0.4 mmol, 104.8 mg), PPh₃ was added to an oven-dried crimp-top vial with a magnetic stir bar. This vial was evacuated, sealed with a septum, and backfilled three times with nitrogen before adding 1.5 mL of anhydrous toluene while a nitrogen stream ran. The mixture was then cooled to 0 °C in an ice bath. Using a syringe, *N*-bromosuccinimide (NBS), which was dissolved in anhydrous toluene (1.5 mL), was added at 0 °C in an amount of 2.1 equivalents (0.42 mmol, 75 mg). The solution changed

color rapidly after two minutes of swirling. A one-millilitre sample was taken for NMR analysis. The ^{31}P NMR spectrum showed two new signals at δ 31.1, assigned to bromophosphonium ion **I**, and δ -5.38, assigned to unreacted triphenylphosphine (Figure S1).

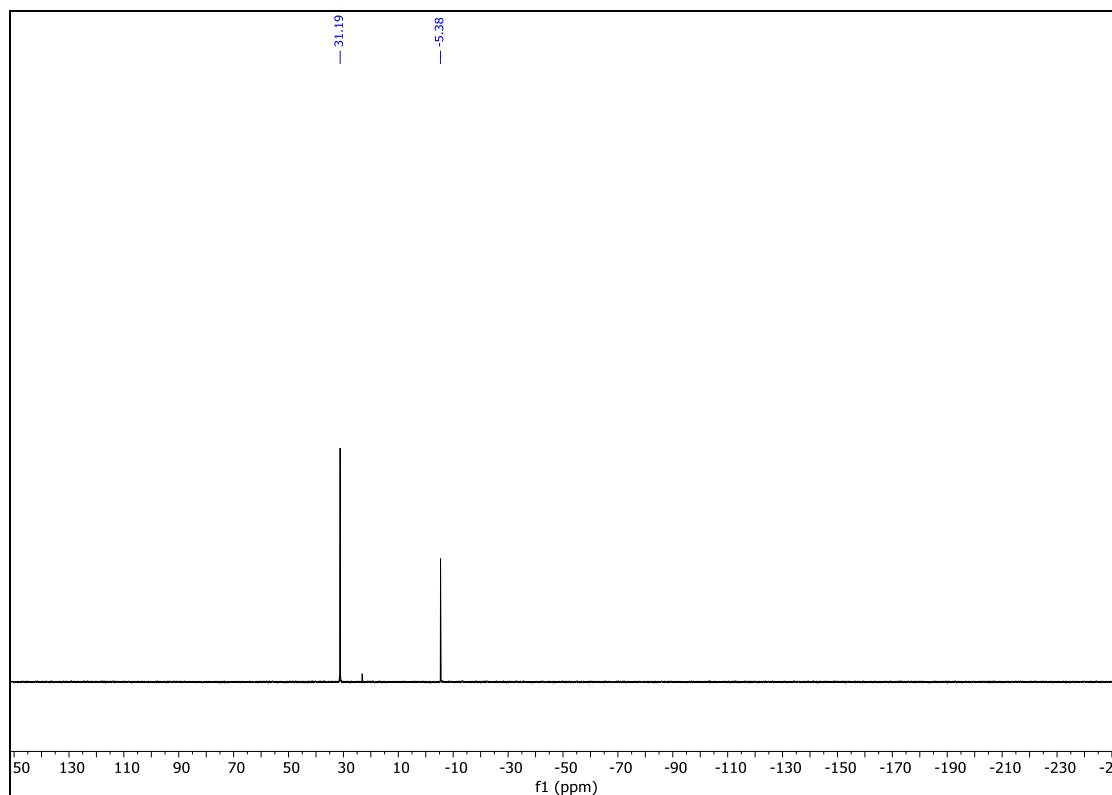


Figure S1: ^{31}P NMR of bromophosphonium ion **I**

Control Experiment 2: In a different experiment, a crimp-top vial fitted with a magnetic stir bar was charged with bromophosphonium ion **I** along with benzoic acid (**1a**, 24.4 mg, 0.2 mmol) and followed by the addition of DCE (1.5 mL). The resulting mixture was cooled to 0 °C using an ice bath. After 15 min., tosyl sulfonyl hydrazide (0.24 mmol) was added, and the reaction was kept at 25 °C for 6 h. A TLC plate monitored the completion of the reaction in 30 % EtOAc in hexane. The crude was purified by column chromatography and eluted with hexane/EtOAc to afford the desired products. Product 3a was obtained with a 78% yield, confirmed by ^1H and ^{13}C NMR.

Control Experiment 3: In a different experiment, a crimp-top vial fitted with a magnetic stir bar was charged with benzoic acid (**1a**, 24.4 mg, 0.2 mmol) and triphenylphosphine, PPh_3 (104.8 mg, 0.4 mmol). Toluene (1.5 mL) was added. The resulting mixture was cooled to 0 °C using an ice bath. *N*-bromosuccinimide (74 mg, 0.42 mmol, 2.1 equiv), dissolved in toluene (1.5 mL), was added via syringe at 0 °C. After 2 minutes of stirring, an immediate color change was observed, and an aliquot (1 mL) was taken for NMR analysis. The ^{31}P NMR spectrum displayed 45.9 ppm assigned to acyloxyphosphonium ion **II** (Figure S2).

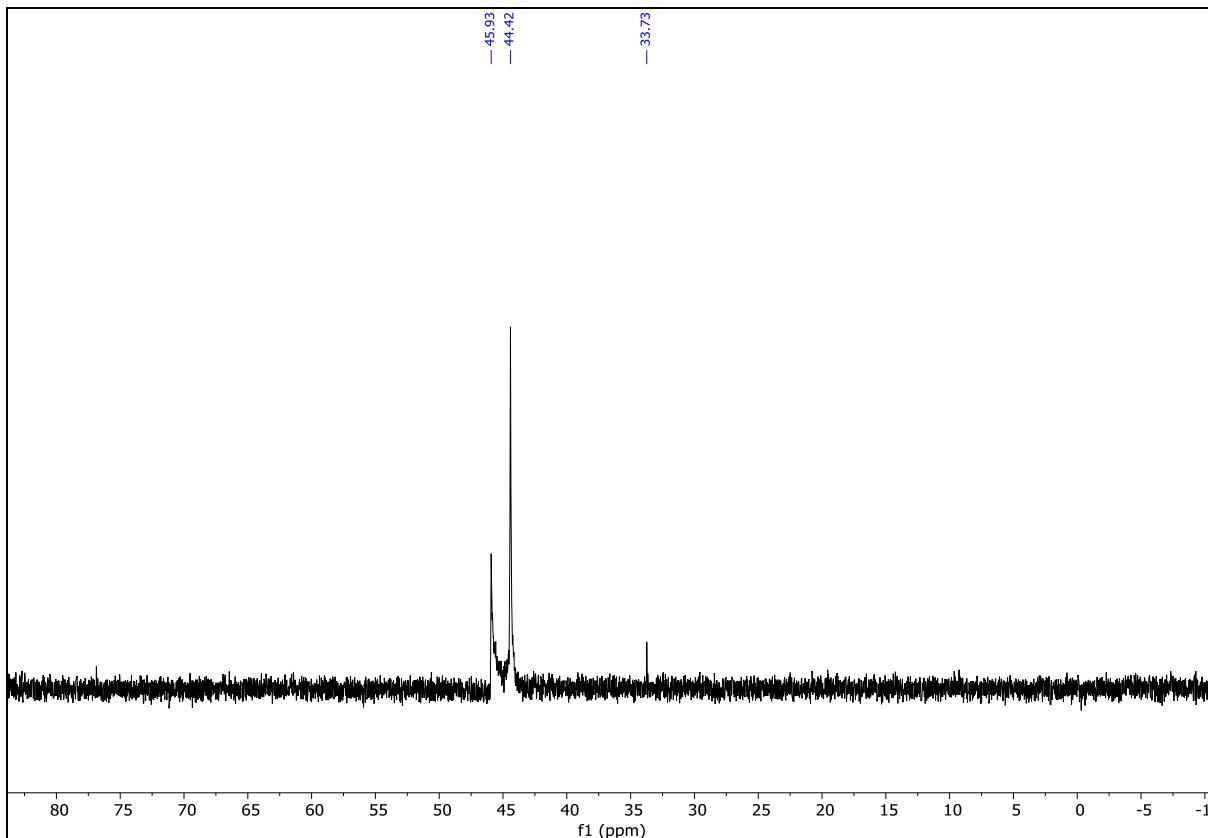


Figure S2: ^{31}P NMR of acyloxyphosphonium ion **II**

6. X-ray Data of Compound 3ag

Table S1: Crystal data and structure refinement for APARNA_06062023_0m.

Identification code	APARNA_06062023_0m
Empirical formula	C ₁₄ H ₁₃ ClN ₂ O _{3.5} S _{1.5}
Formula wESI-TOFght	348.80
Temperature/K	305.00
Crystal system	triclinic
Space group	P-1
a/Å	8.3039(6)
b/Å	10.6015(7)
c/Å	11.5759(8)
α/°	74.614(2)
β/°	70.161(2)
γ/°	87.736(2)
Volume/Å ³	922.80(11)
Z	2
ρcalcg/cm ³	1.255
μ/mm ⁻¹	0.390
F(000)	360.0
Radiation	MoKα ($\lambda = 0.71073$)
2Θ range for data collection/°	3.99 to 56.578
Index ranges	-11 ≤ h ≤ 11, -14 ≤ k ≤ 14, -15 ≤ l ≤ 15
Reflections collected	35334
Independent reflections	4527 [Rint = 0.0474, Rsigma = 0.0280]
Data/restraints/parameters	4527/0/182
Goodness-of-fit on F2	1.054
Final R indexes [I>=2σ (I)]	R1 = 0.0896, wR2 = 0.2702
Final R indexes [all data]	R1 = 0.1056, wR2 = 0.2880
Largest diff. peak/hole / e Å ⁻³	1.38/-0.76

Table S2: Bond Lengths for APARNA_06062023_0m.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C11	C2	1.744(5)	C3	C4	1.386(6)
S1	O1	1.438(3)	C4	C12	1.382(6)
S1	O2	1.439(3)	C5	C6	1.512(5)
S1	N1	1.664(3)	C6	C7	1.377(6)
S1	C4	1.771(4)	C6	C11	1.397(6)
O4	C5	1.220(4)	C7	C9	1.396(7)
N1	N2	1.384(5)	C8	C9	1.353(9)
N2	C5	1.344(5)	C8	C10	1.354(8)
C1	C2	1.376(7)	C10	C11	1.388(7)
C1	C13	1.400(8)	C12	C13	1.372(7)
S1	O1	1.438(3)	C4	C12	1.382(6)
C2	C3	1.377(6)			

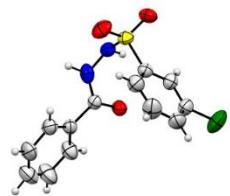
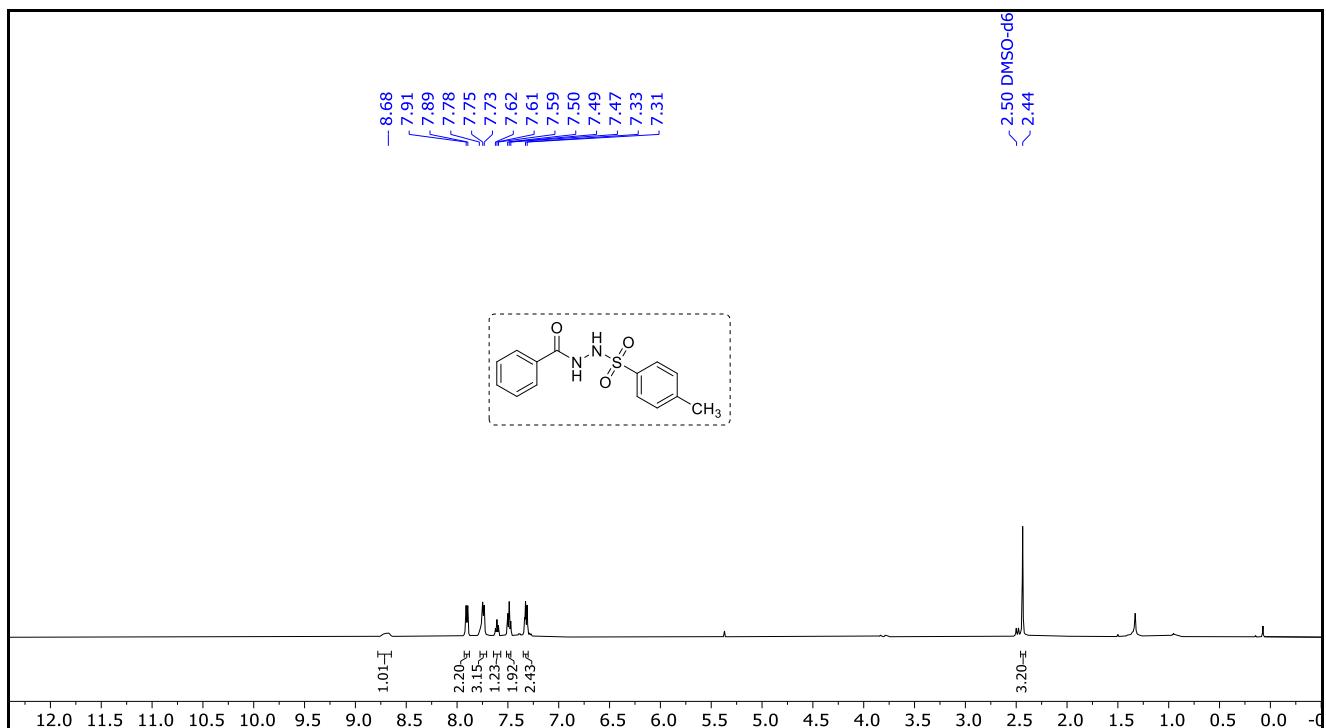


Figure S3: A perspective view of compound **3ag**

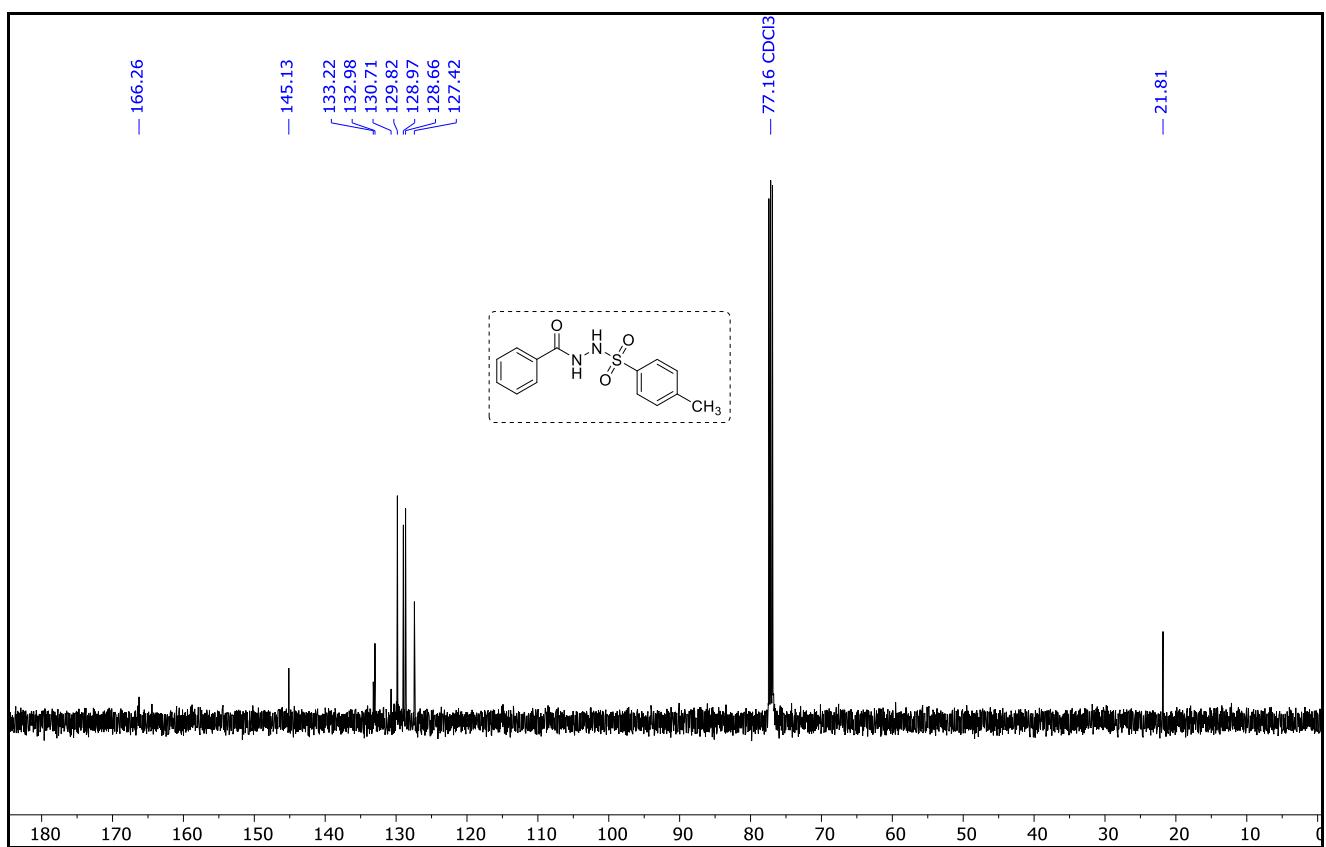
7. Copies of ^1H NMR and ^{13}C NMR Spectra of Products

N'-Benzoyl-4-methylbenzenesulfonohydrazide (3a):

^1H NMR (500 MHz, CDCl_3)

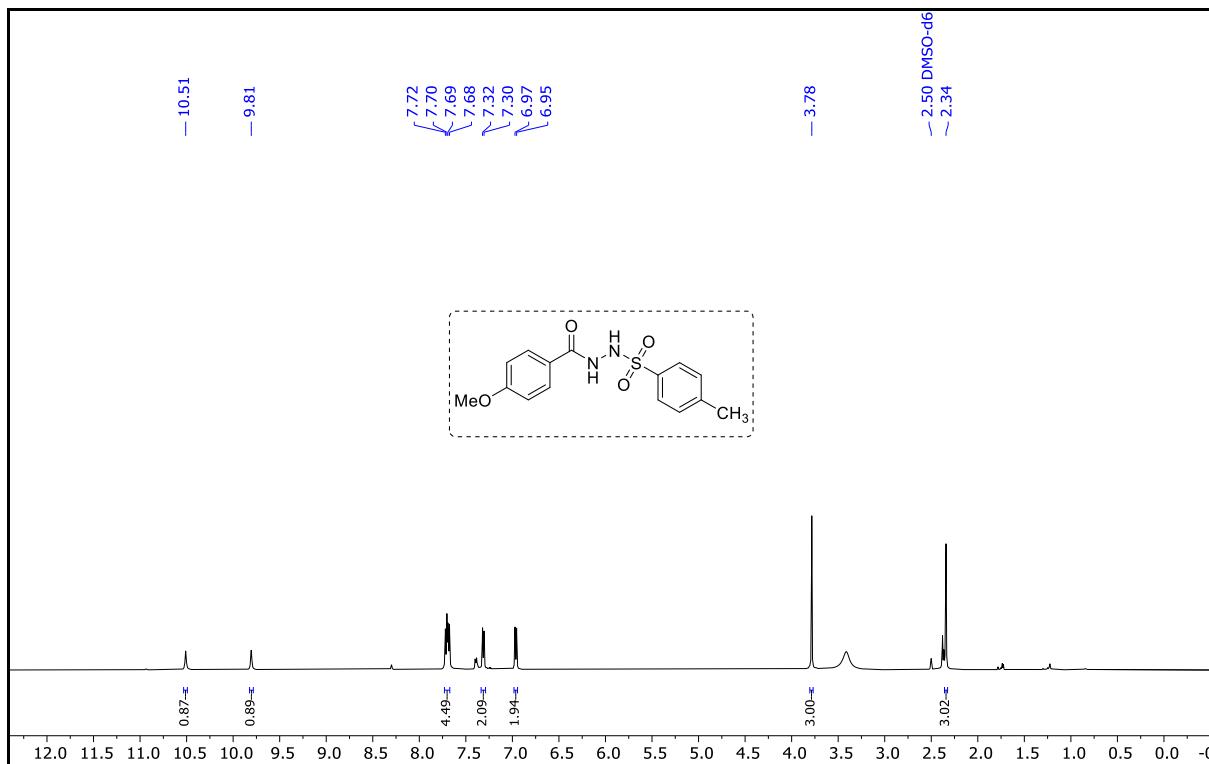


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CDCl_3)

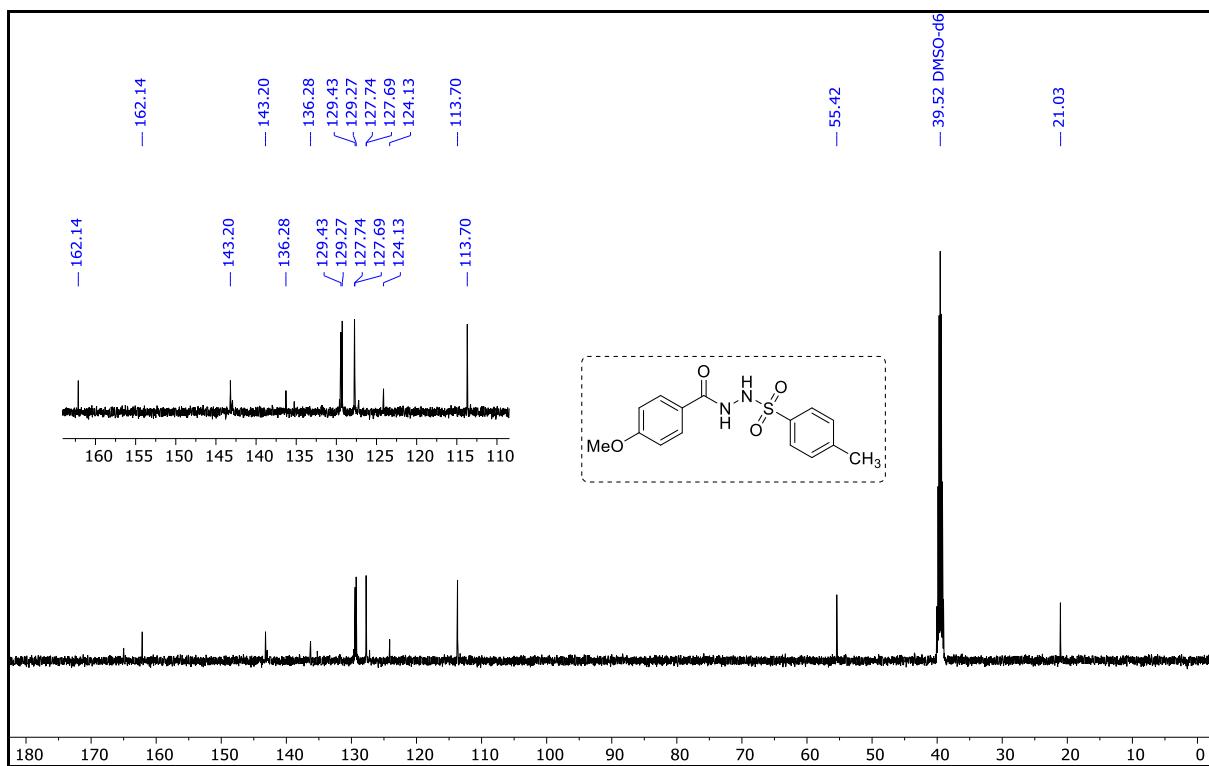


***N'*-(4-Methoxybenzoyl)-4-methylbenzenesulfonohydrazide (3b):**

^1H NMR (500 MHz, DMSO- d_6)

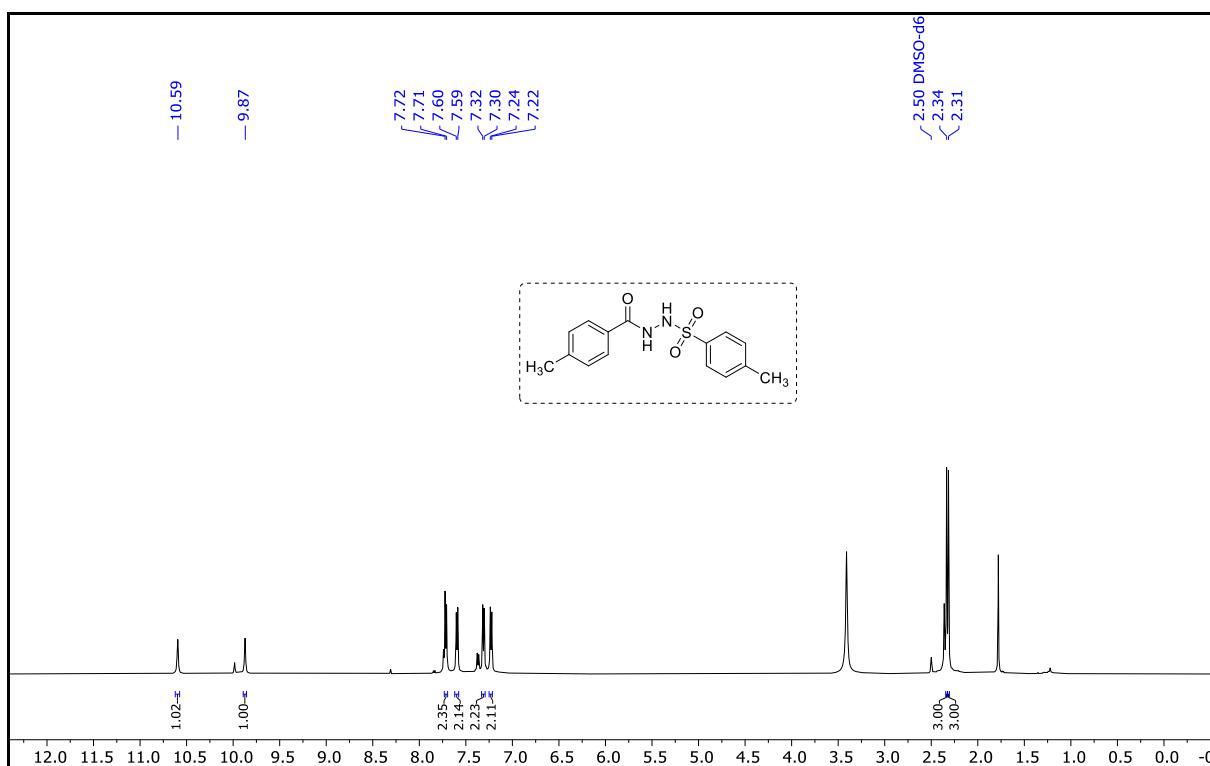


$^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, DMSO- d_6)

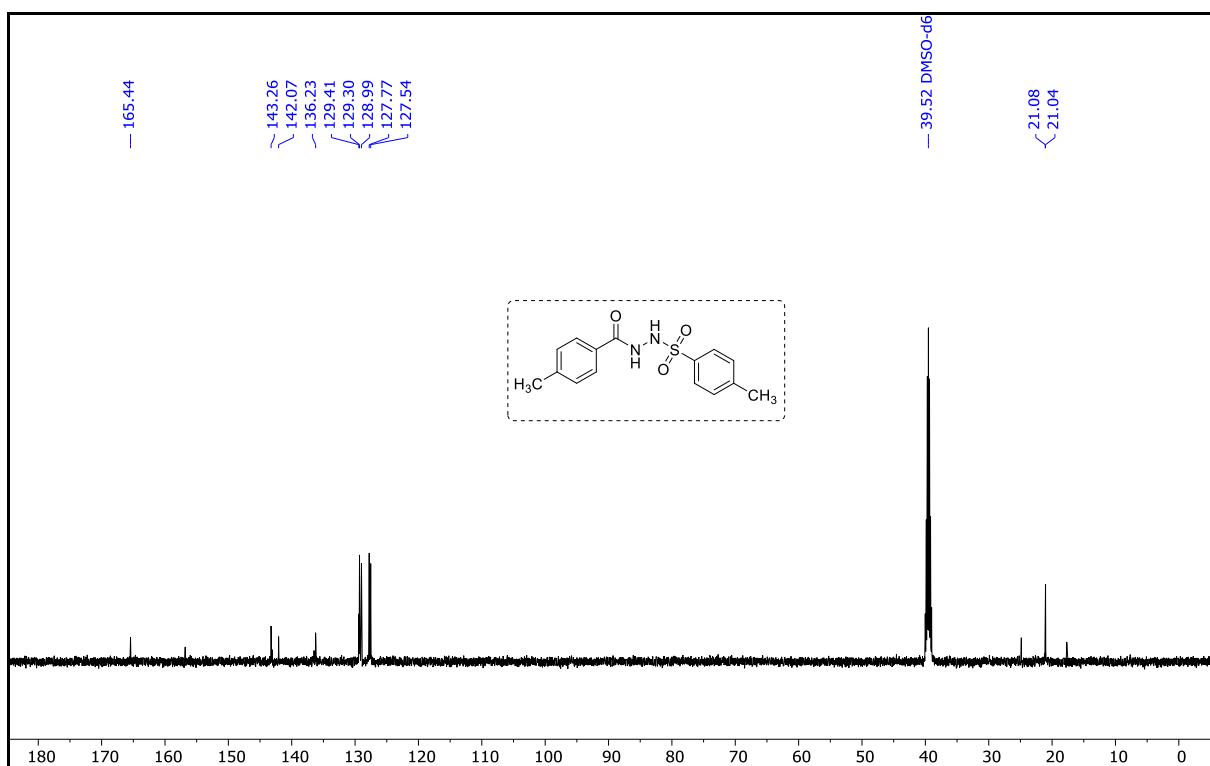


4-Methyl-N'-(4-methylbenzoyl) benzenesulfonohydrazide (3c)

^1H NMR (500 MHz, DMSO- d_6)

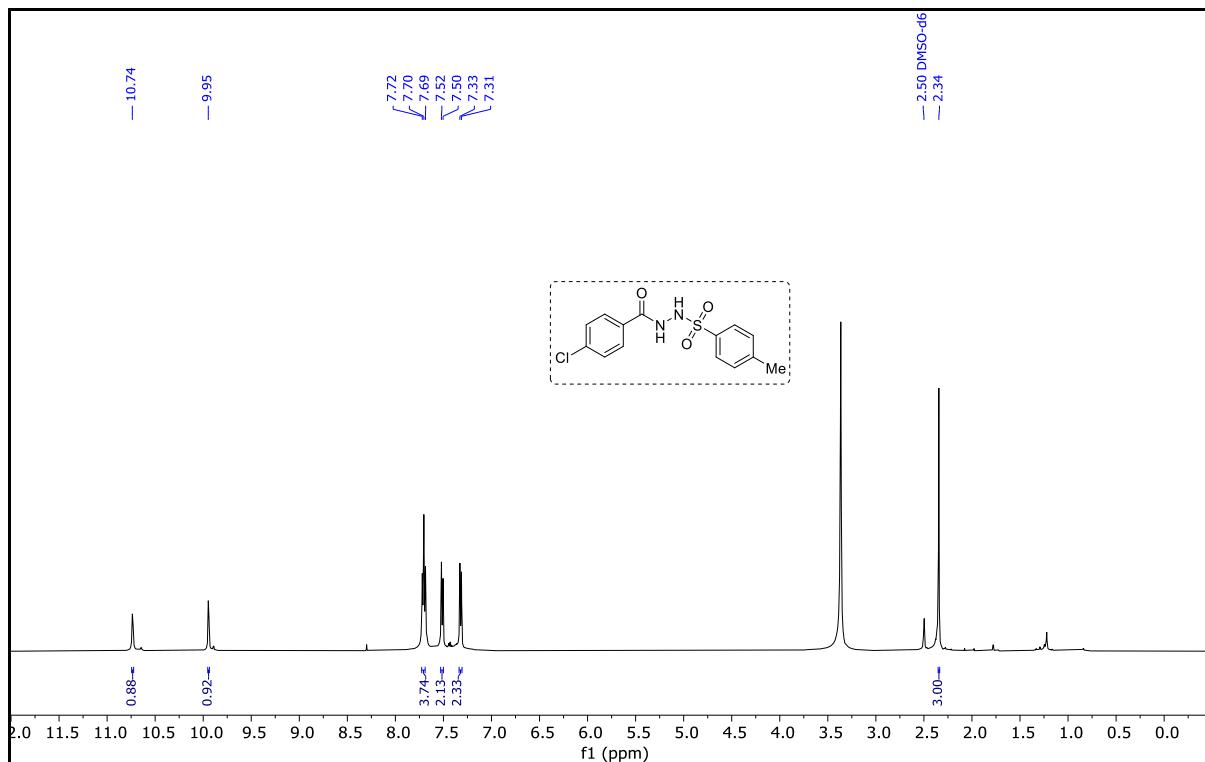


$^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, DMSO- d_6)

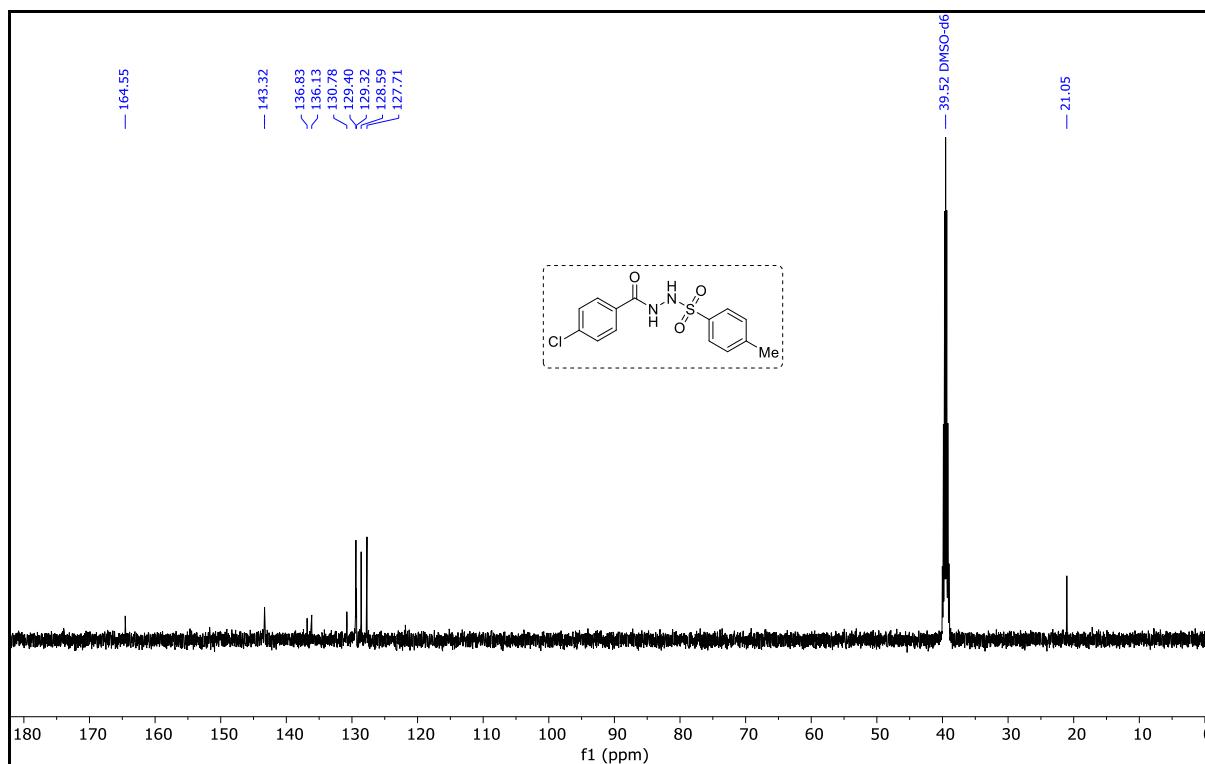


***N'*-(4-Chlorobenzoyl)-4-methylbenzenesulfonohydrazide (3d):**

^1H NMR (500 MHz, DMSO- d_6)

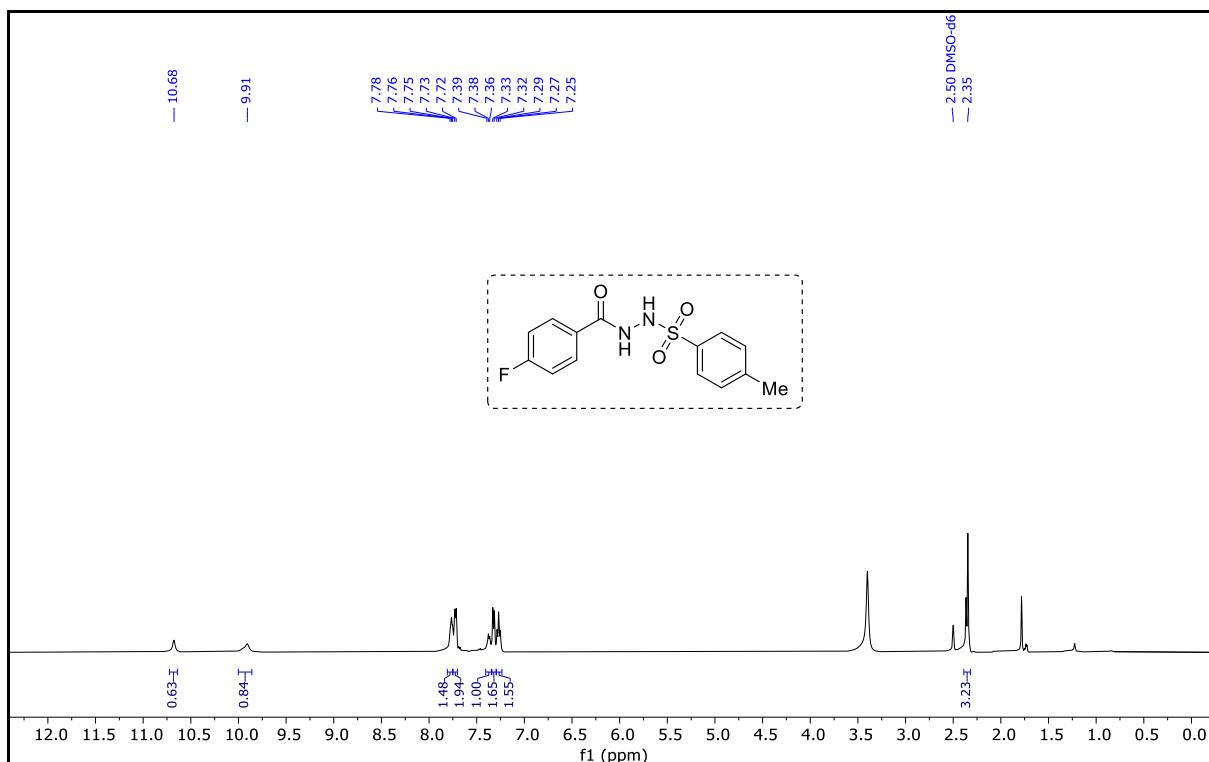


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

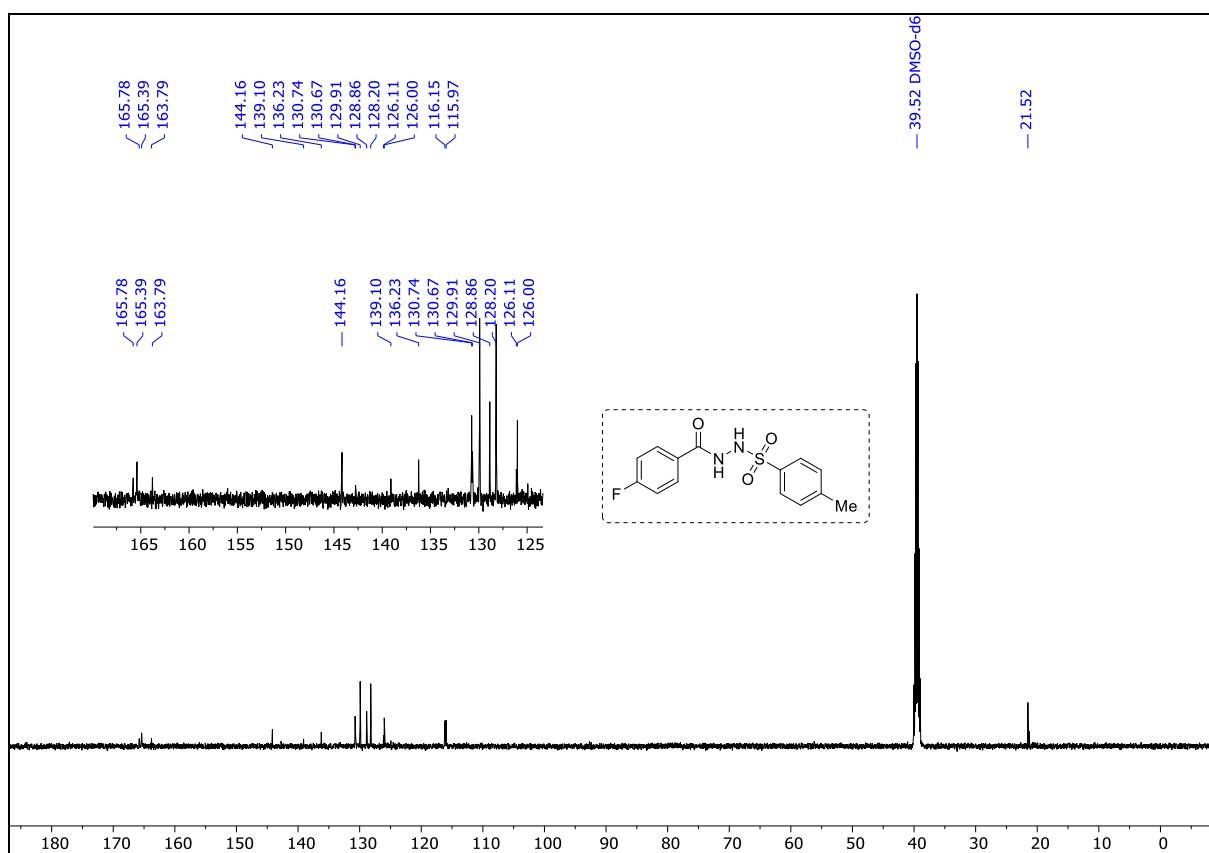


***N'*-(4-Fluorobenzoyl)-4-methylbenzenesulfonohydrazide (3e):**

^1H NMR (500 MHz, DMSO- d_6)

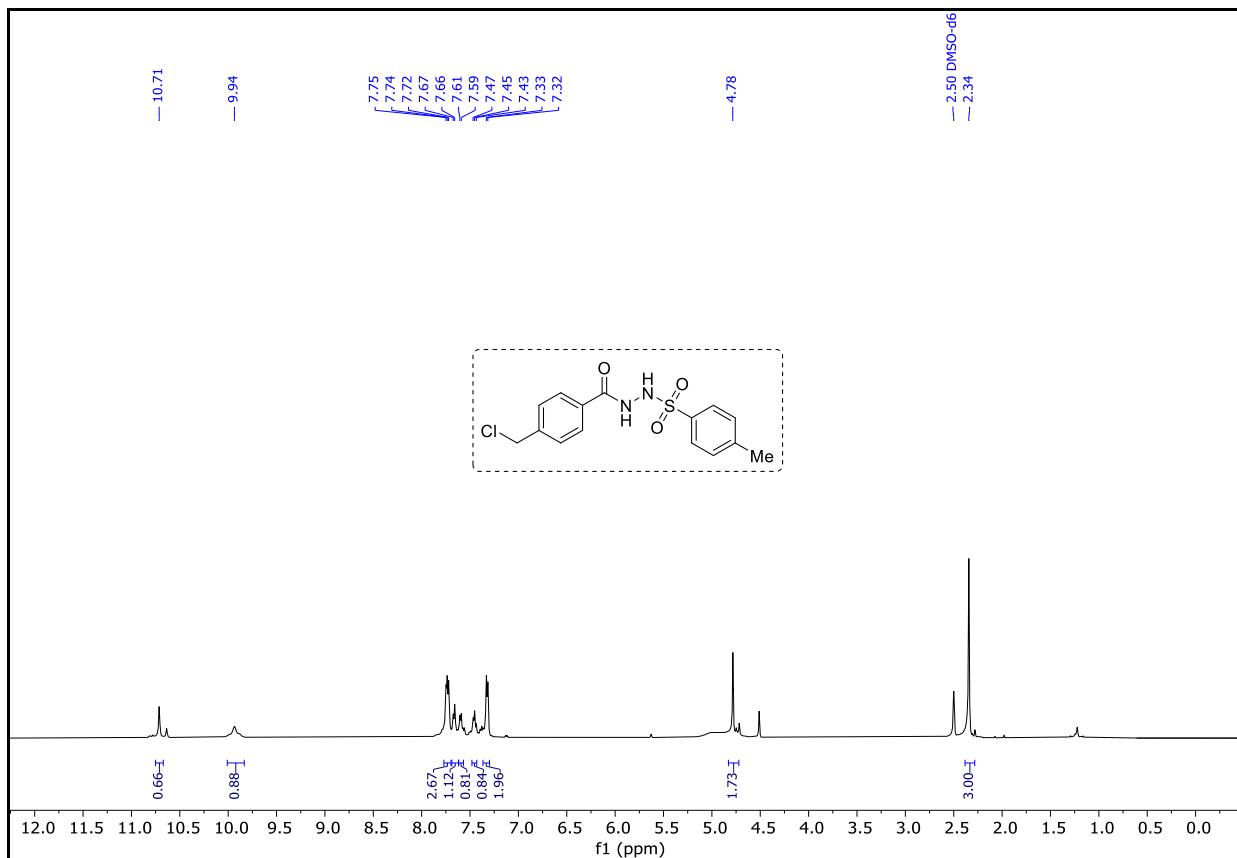


$^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, DMSO- d_6)

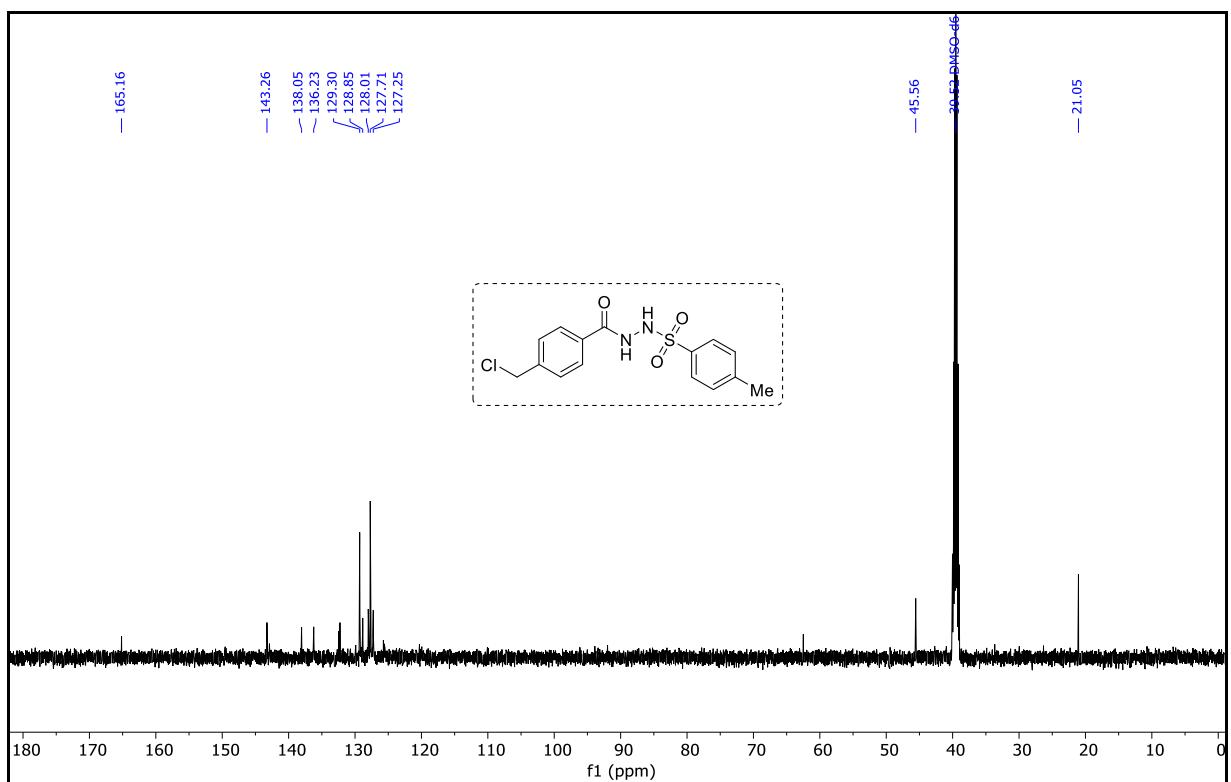


N'-(4-(Chloromethyl) benzoyl)-4-methylbenzenesulfonohydrazide (3f):

^1H NMR (500 MHz, DMSO- d_6)

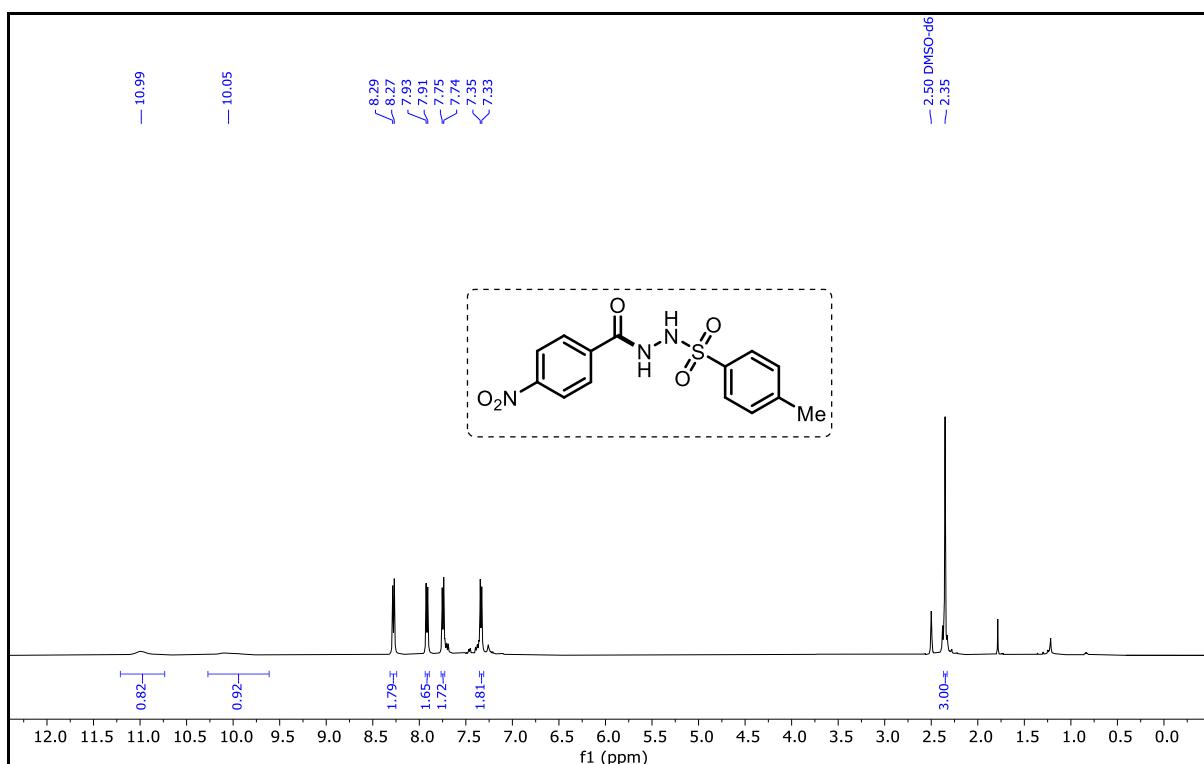


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

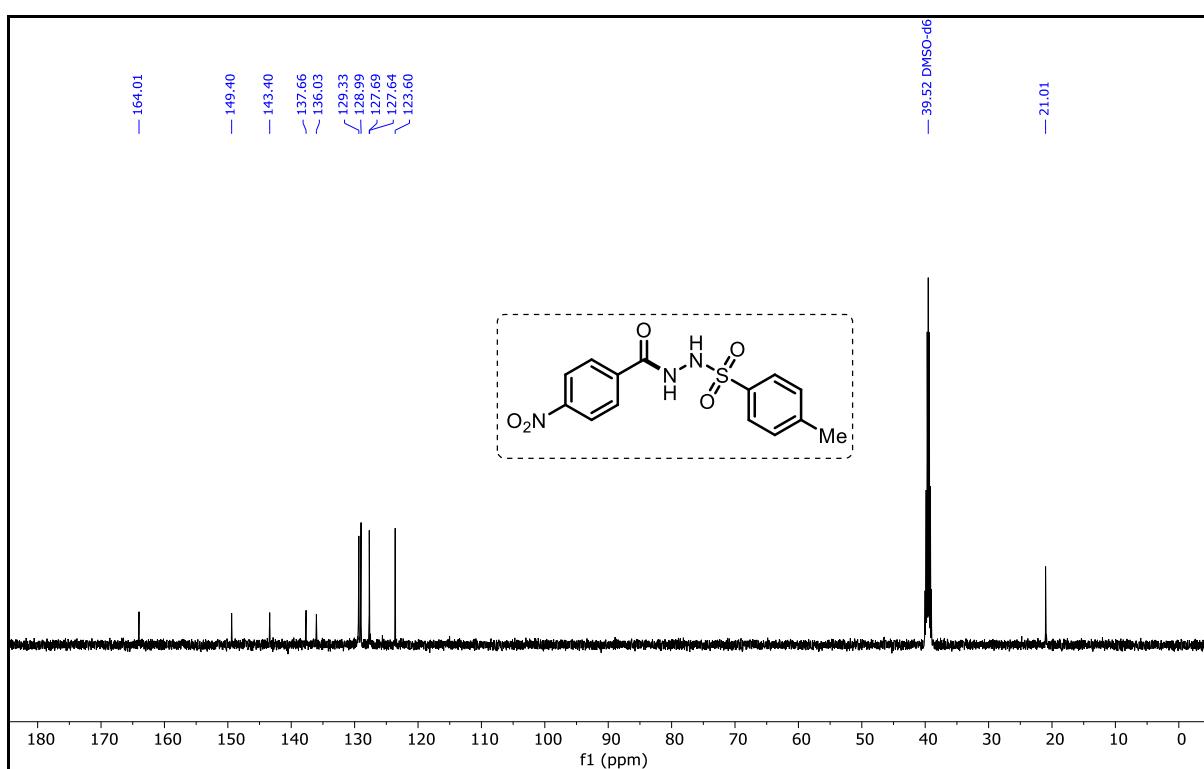


4-Methyl-N'-(4-nitrobenzoyl) benzenesulfonohydrazide (3g):

^1H NMR (500 MHz, DMSO- d_6)

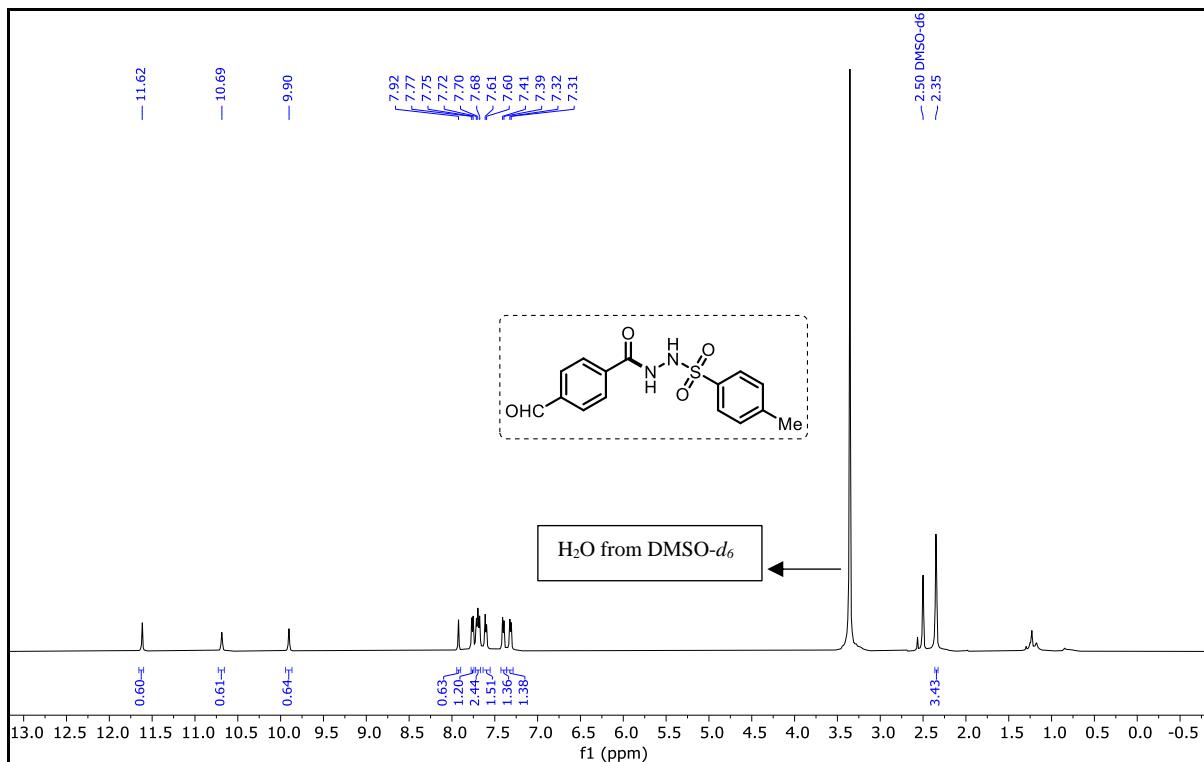


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

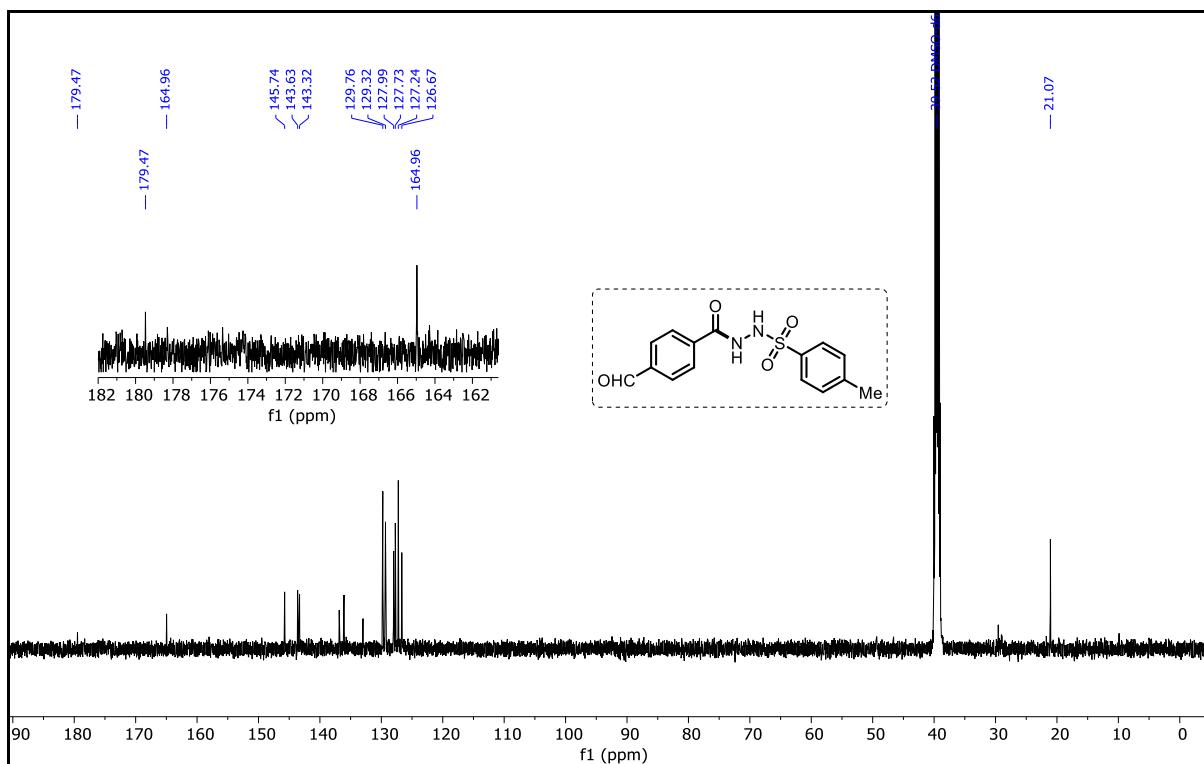


***N'*-(4-Formylbenzoyl)-4-methylbenzenesulfonohydrazide (3h):**

¹H NMR (500 MHz, DMSO-*d*₆)

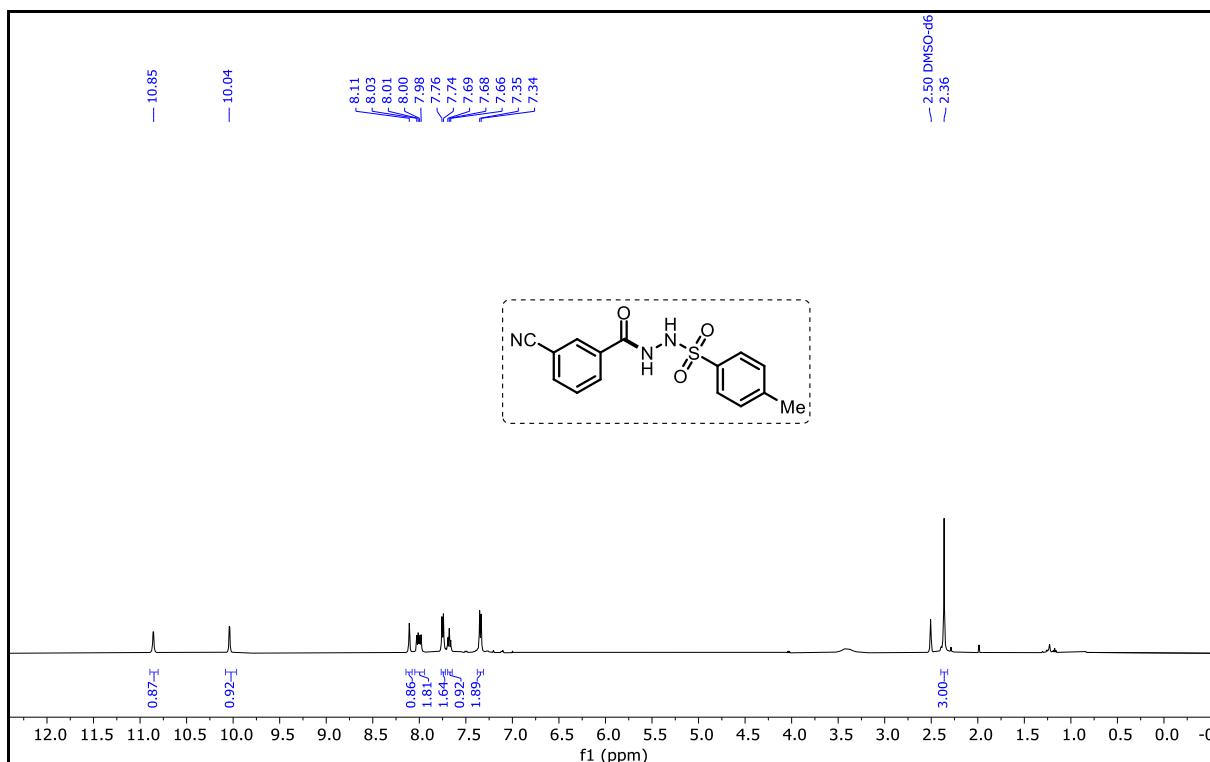


¹³C{¹H} NMR (126 MHz, DMSO-*d*₆)

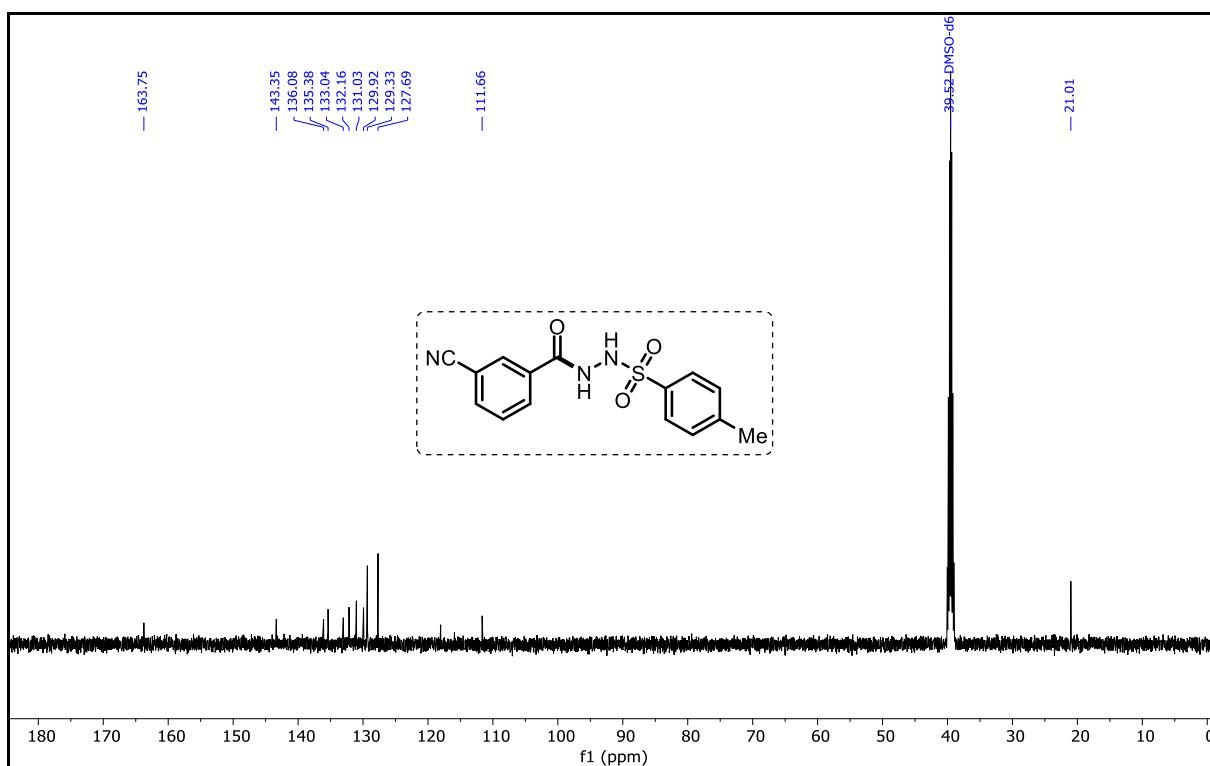


***N'*-(3-Cyanobenzoyl)-4-methylbenzenesulfonohydrazide (3i):**

^1H NMR (500 MHz, DMSO- d_6)

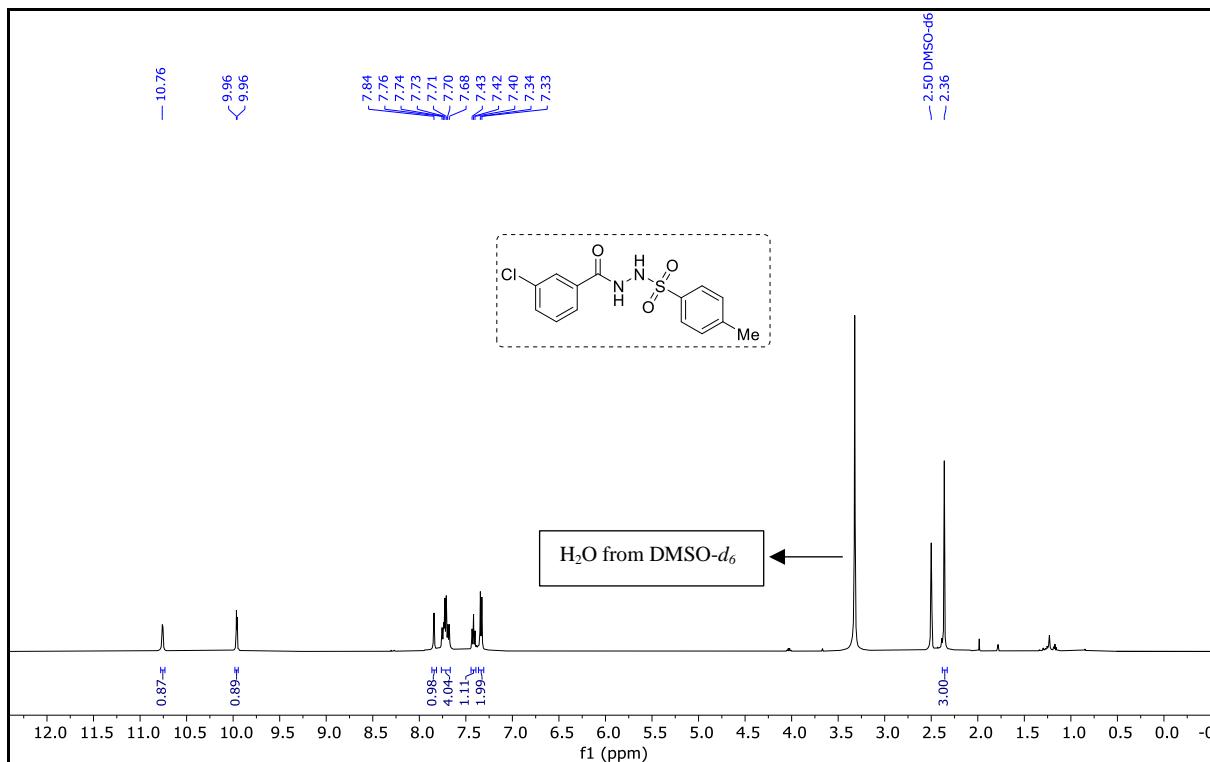


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

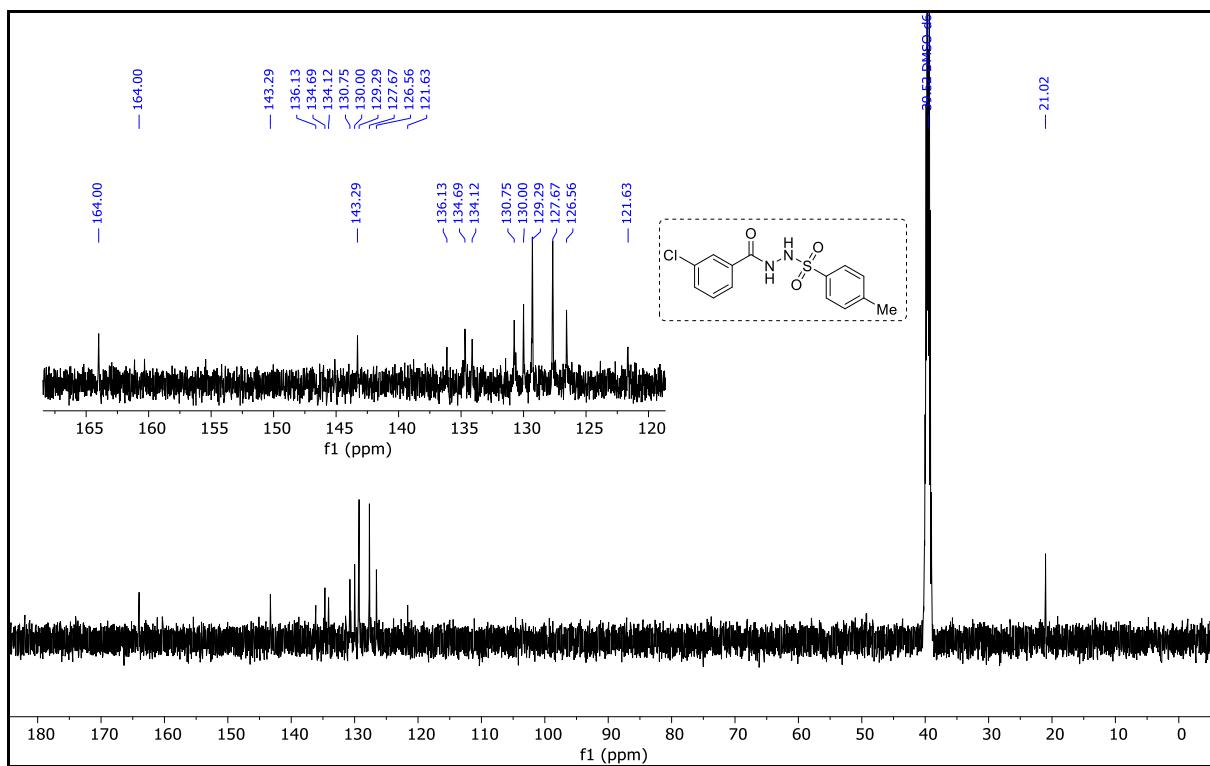


***N'*-(3-Chlorobenzoyl)-4-methylbenzenesulfonohydrazide (3j):**

^1H NMR (500 MHz, DMSO- d_6)

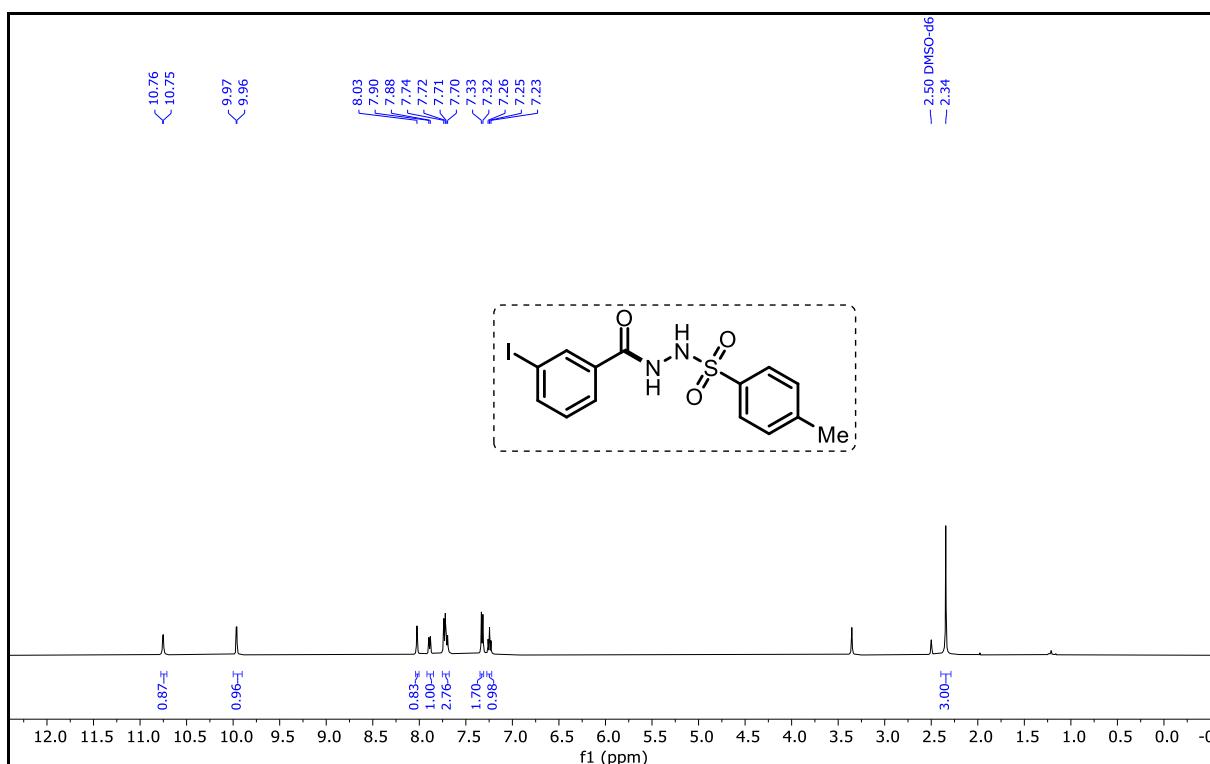


$^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, DMSO- d_6)

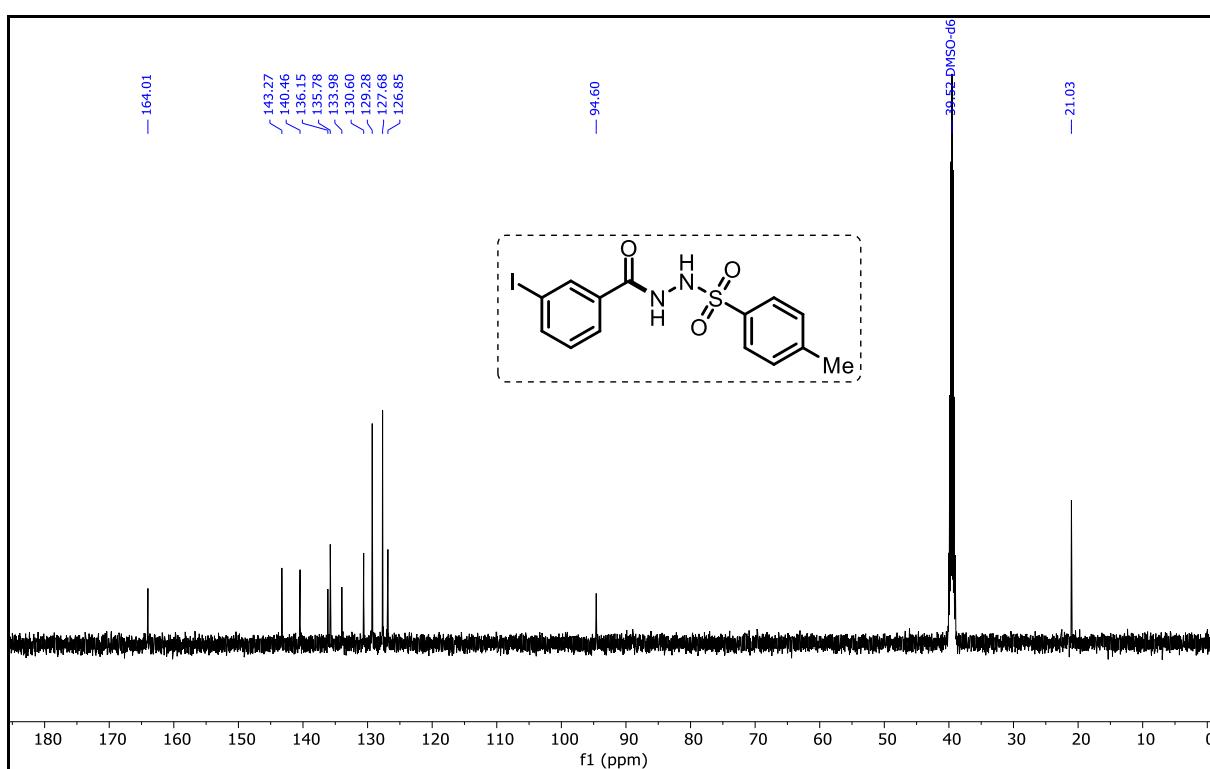


***N'*-(3-Iodobenzoyl)-4-methylbenzenesulfonohydrazide (3k):**

^1H NMR (500 MHz, DMSO- d_6)

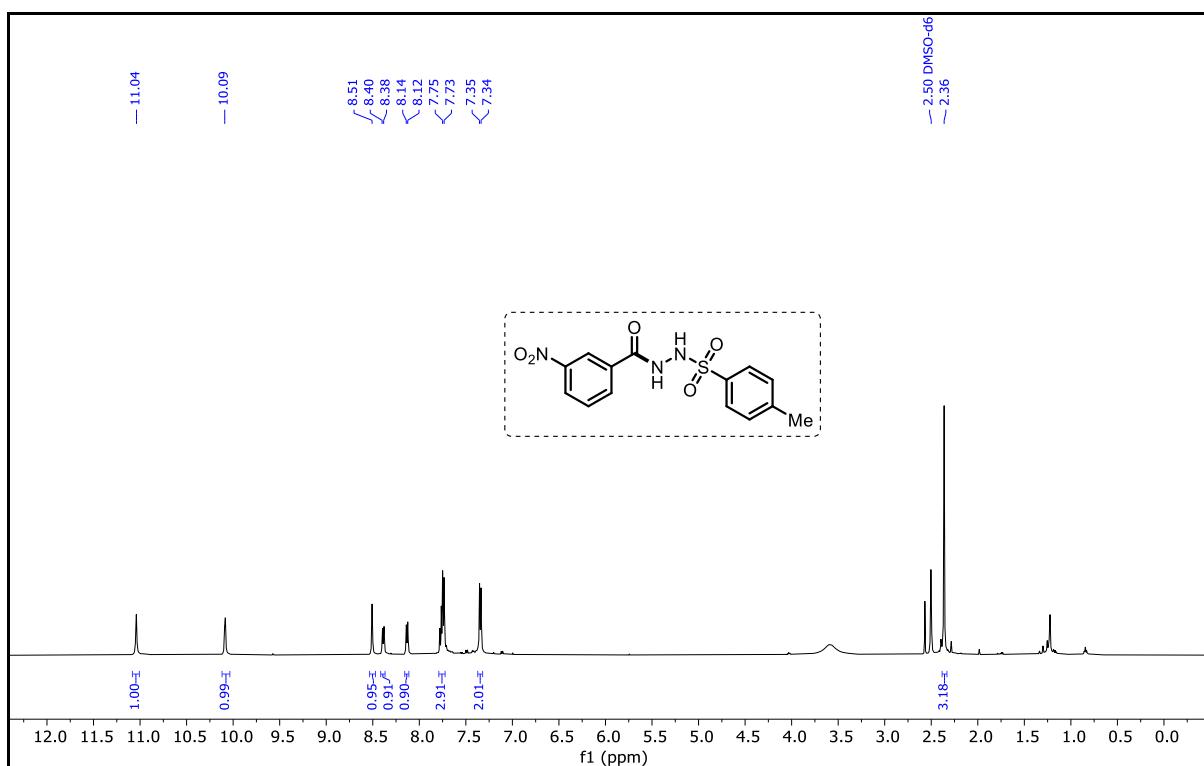


$^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, DMSO- d_6)

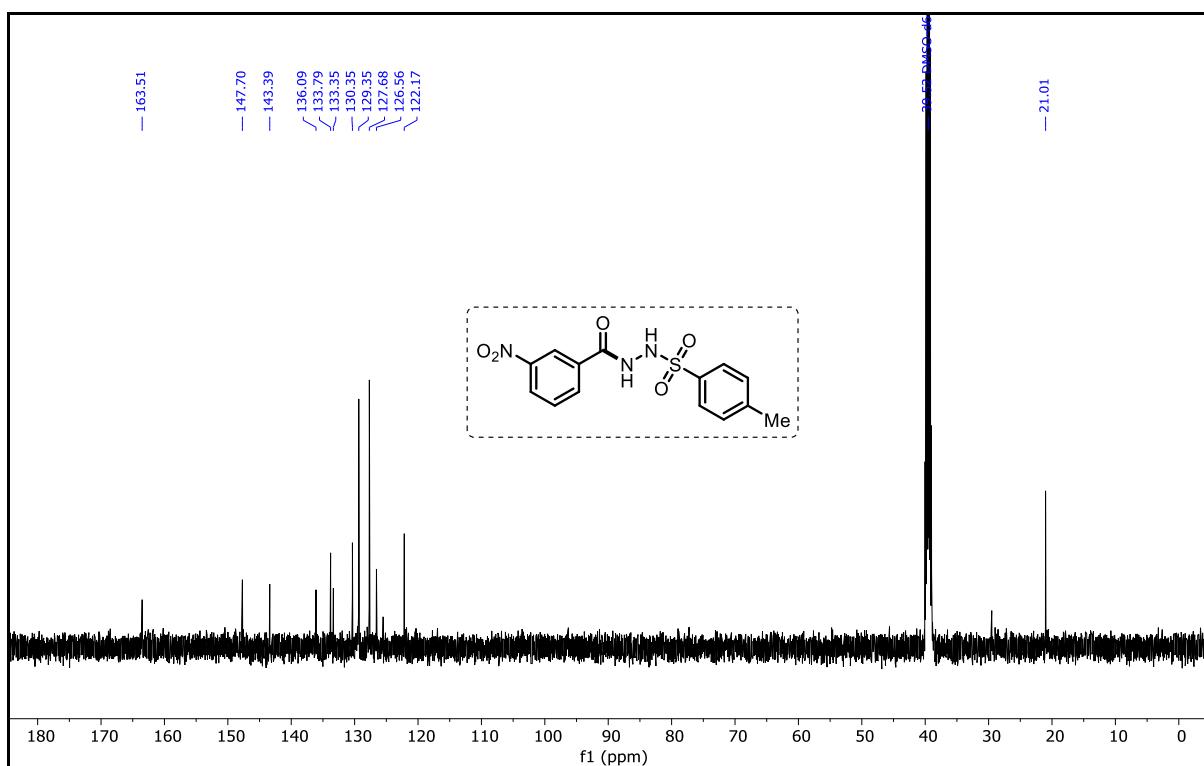


4-Methyl-N'-(3-nitrobenzoyl) benzenesulfonohydrazide (3l):

^1H NMR (500 MHz, DMSO- d_6)

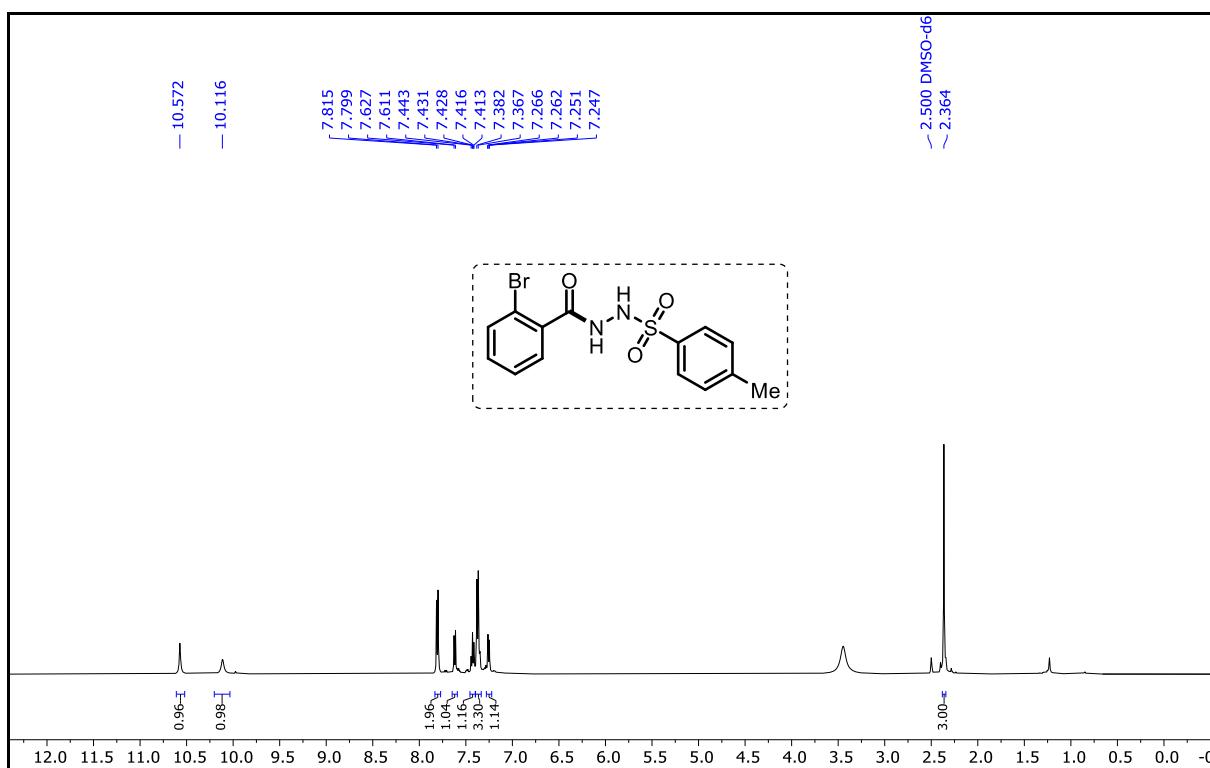


$^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, DMSO- d_6)

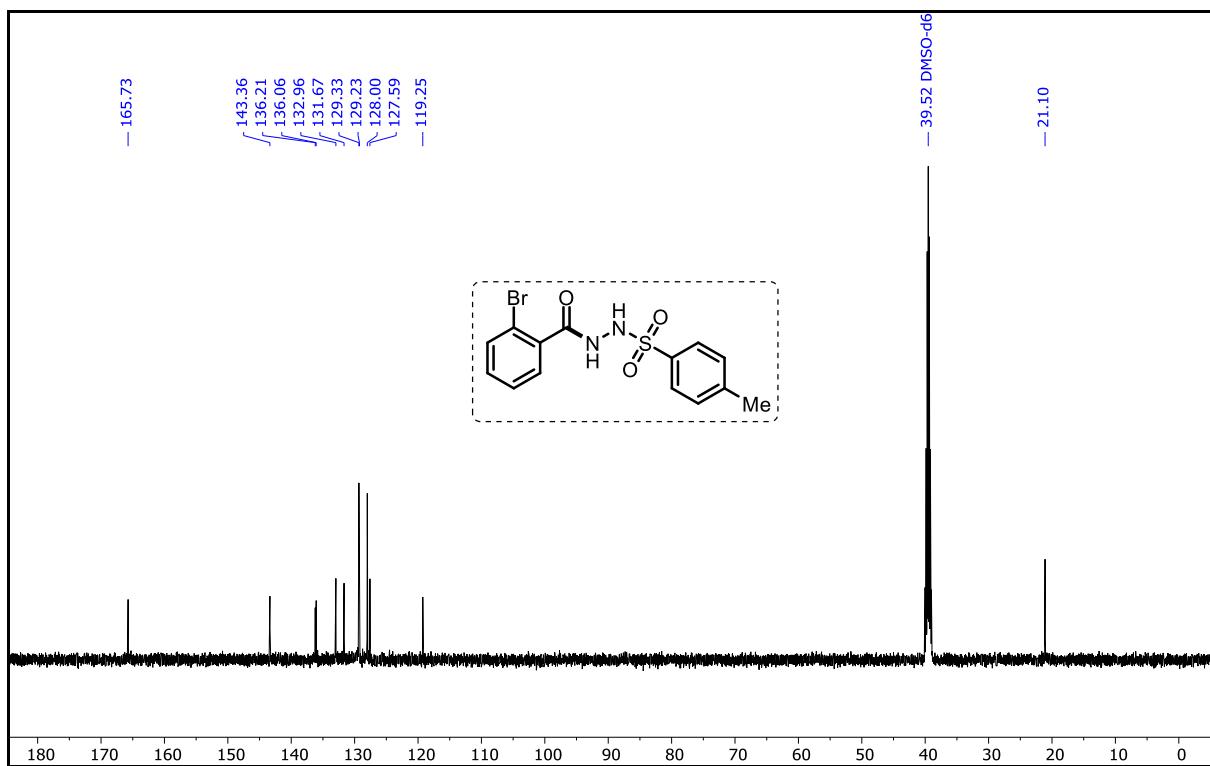


***N'*-(2-Bromobenzoyl)-4-methylbenzenesulfonohydrazide (3m):**

^1H NMR (500 MHz, DMSO- d_6)

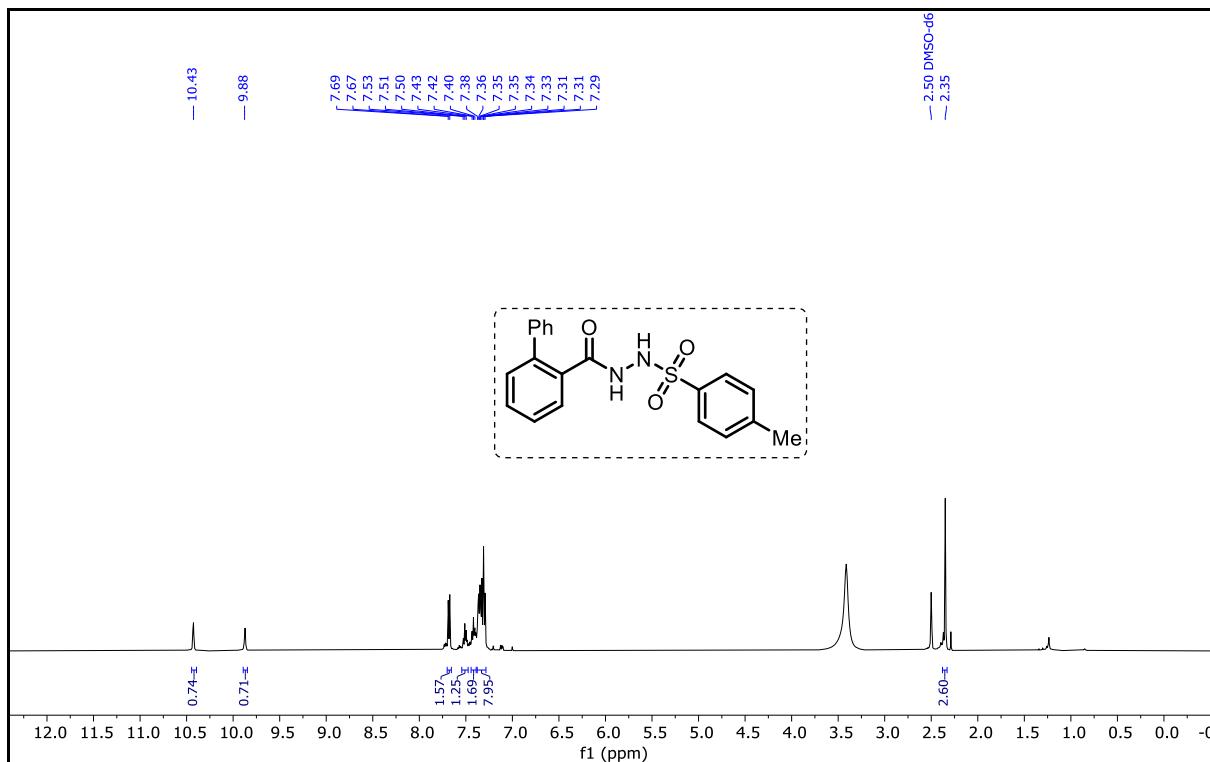


$^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, DMSO- d_6)

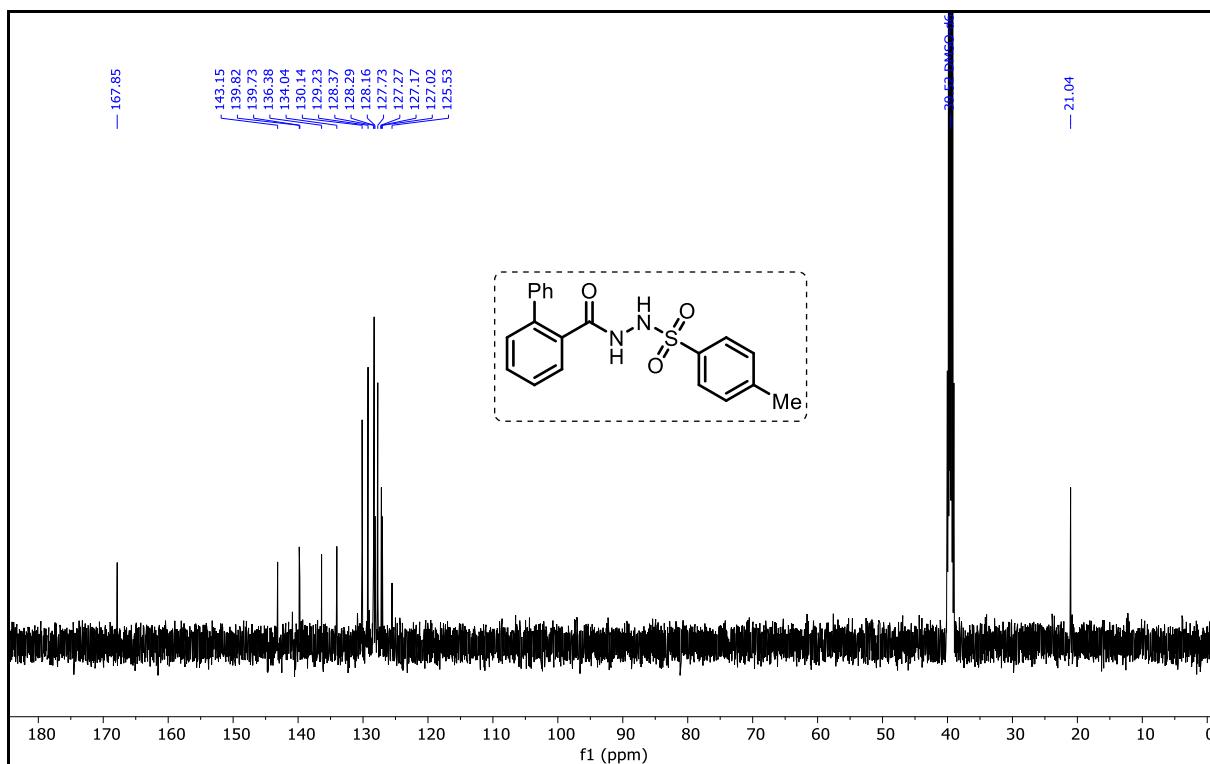


***N'*-([1,1'-Biphenyl]-2-carbonyl)-4-methylbenzenesulfonohydrazide (3n):**

^1H NMR (500 MHz, DMSO- d_6)

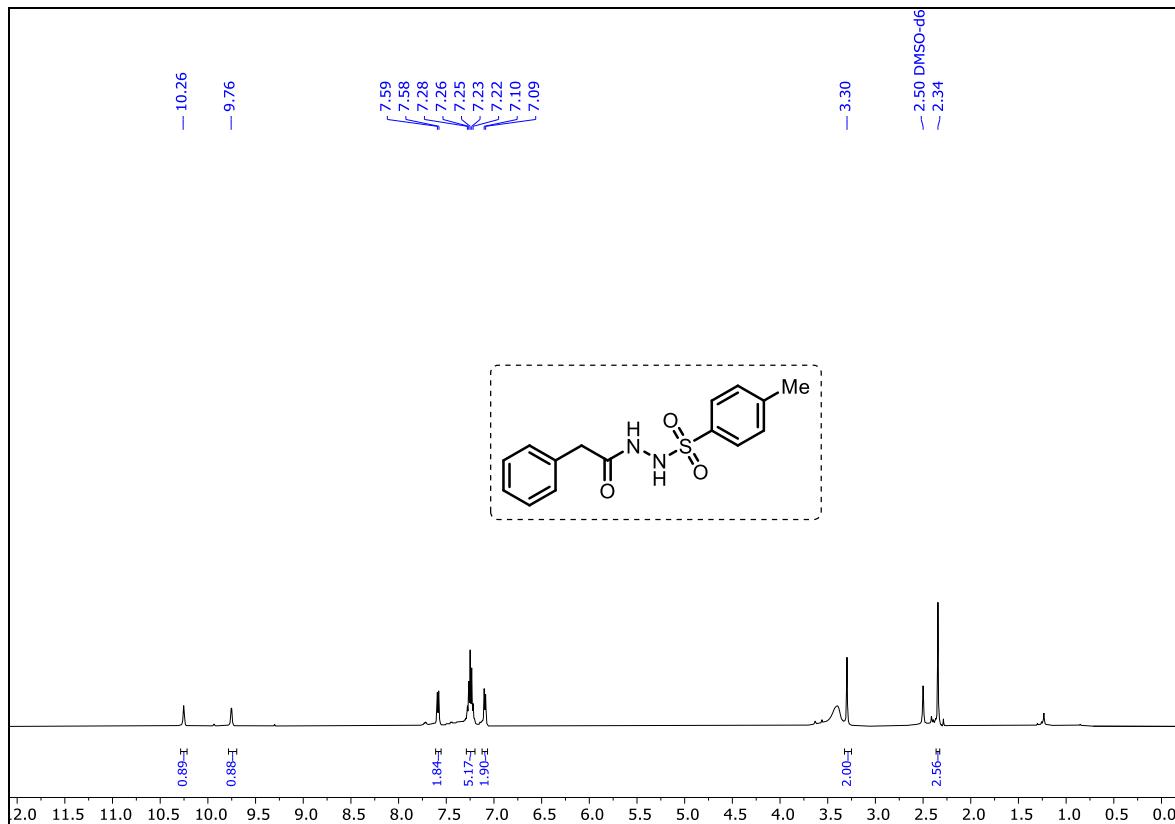


$^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, DMSO- d_6)

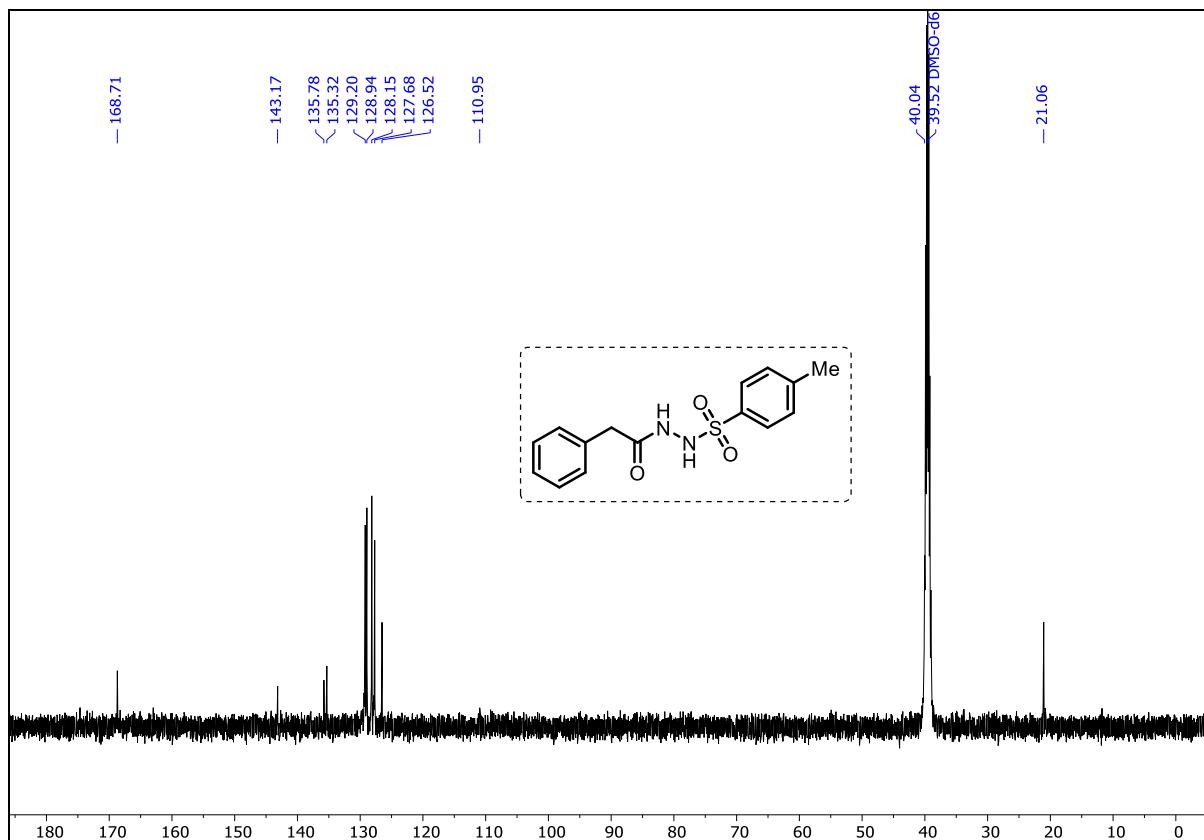


4-Methyl-N'-(2-phenyl acetyl) benzenesulfonohydrazide (3o):

^1H NMR (500 MHz, DMSO- d_6)

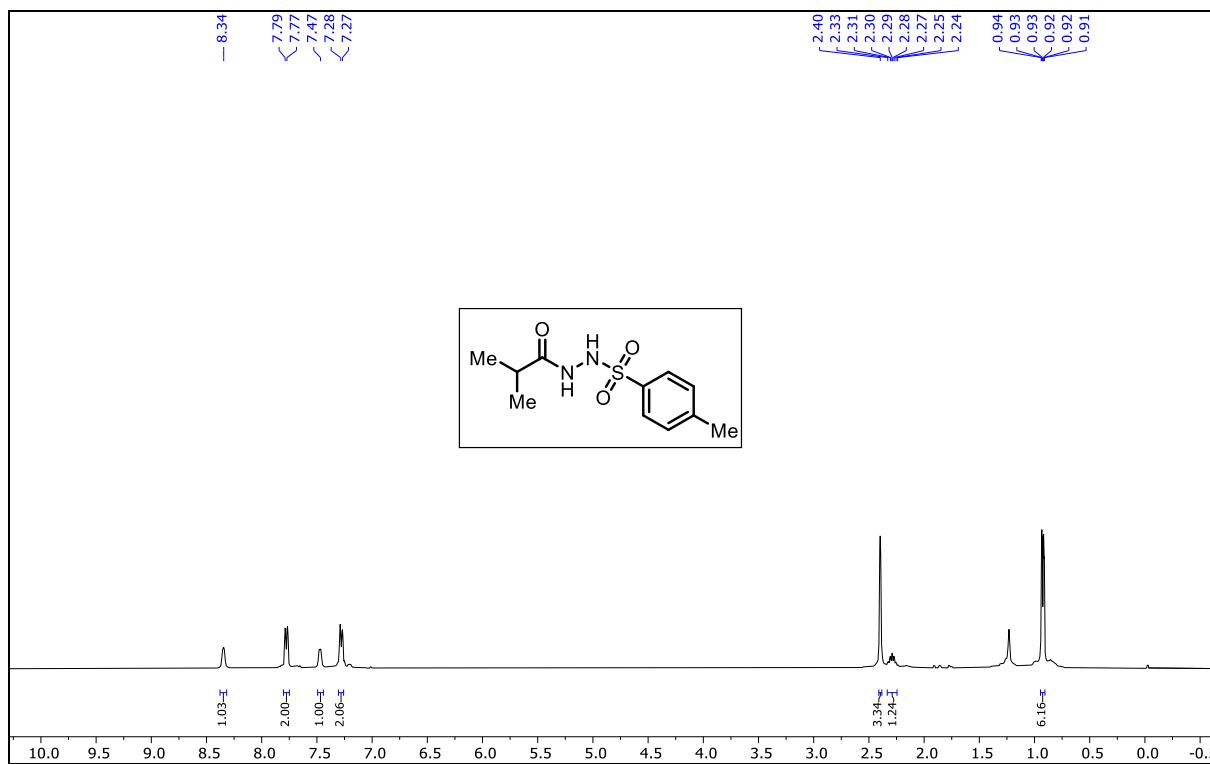


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

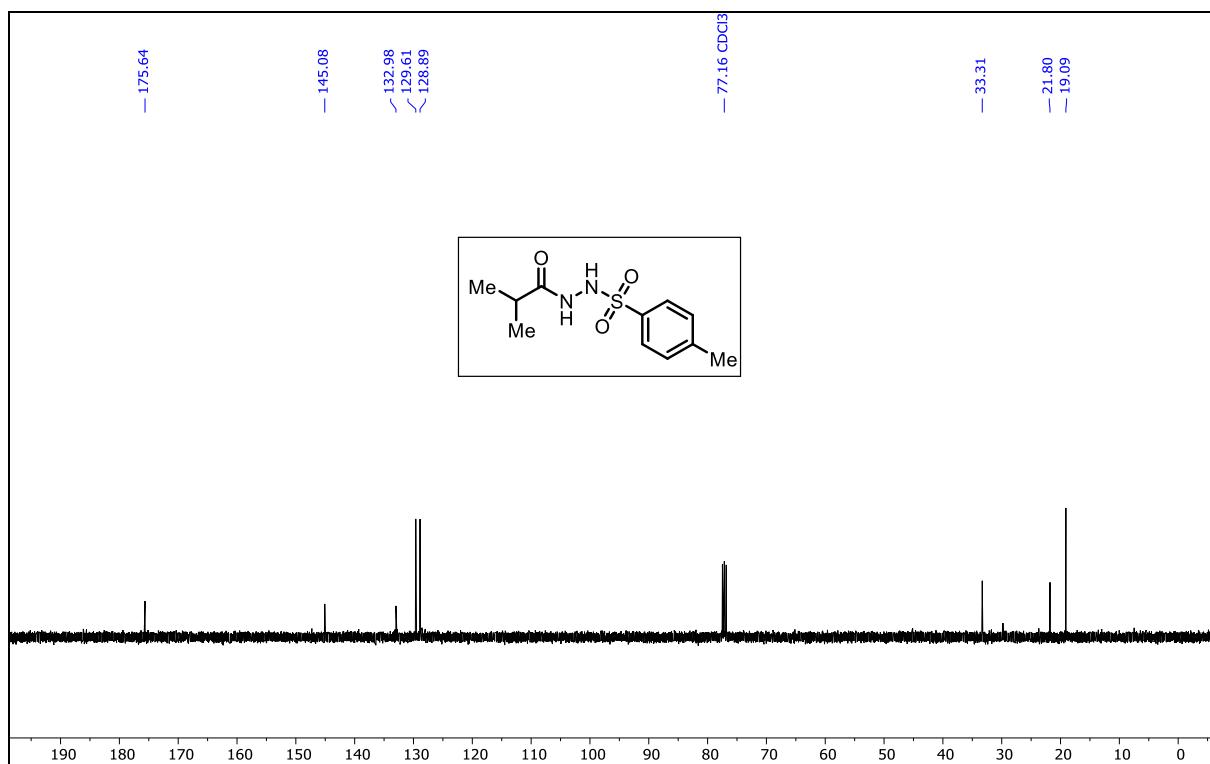


***N'*-Isobutyryl-4-methylbenzenesulfonohydrazide (3p):**

^1H NMR (400 MHz, CDCl_3)

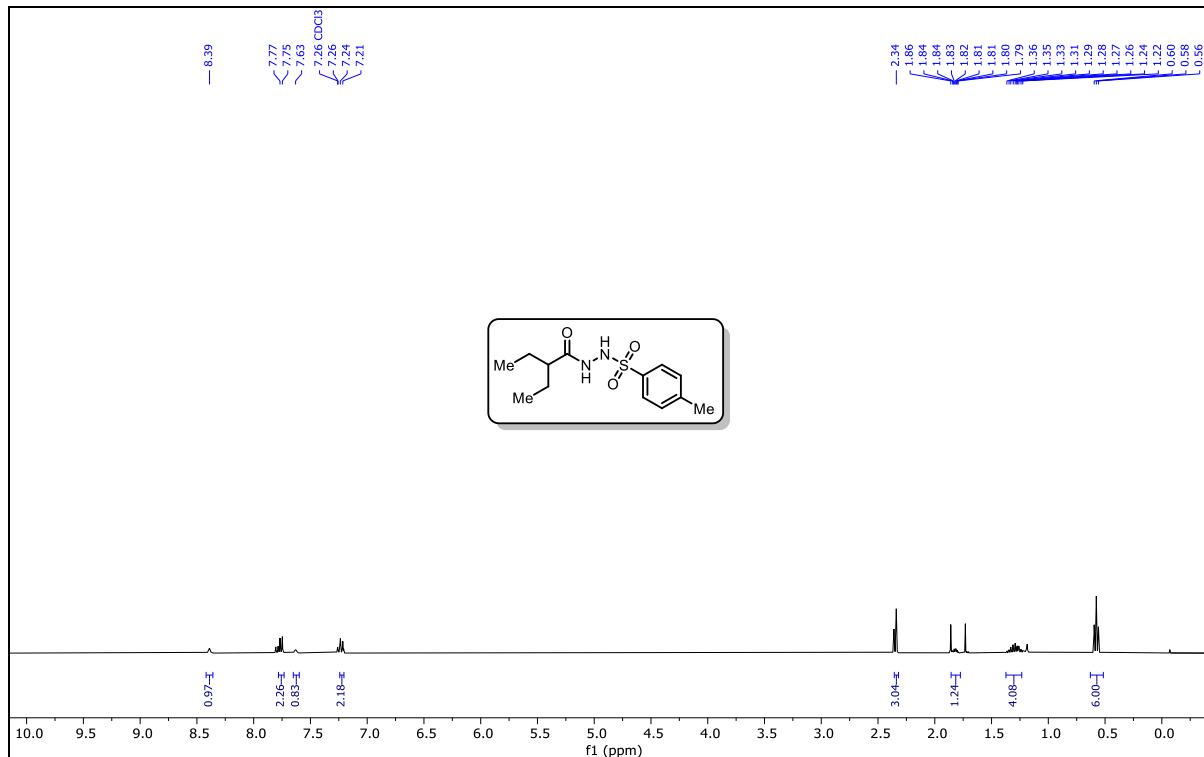


$^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3)

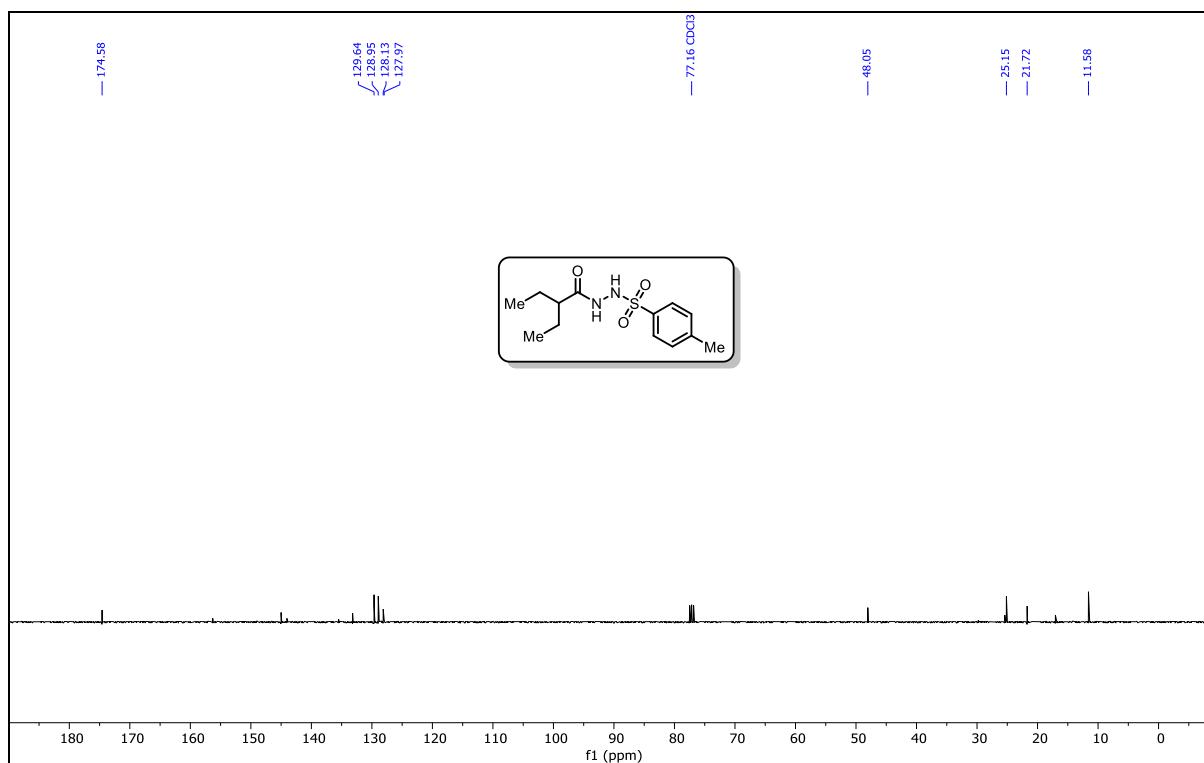


N'-(2-Ethylbutanoyl)-4-methylbenzenesulfonohydrazide (3q):

^1H NMR (400 MHz, CDCl_3)

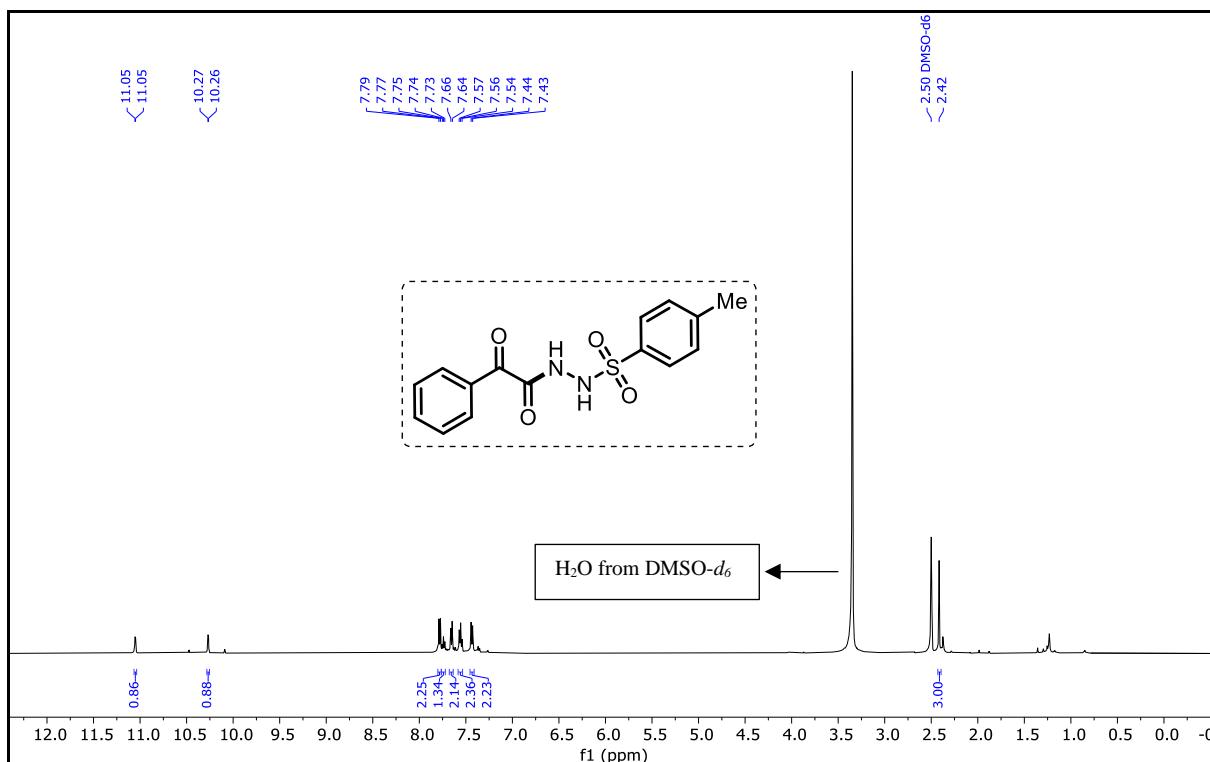


¹³C{¹H} NMR (101 MHz, CDCl₃)

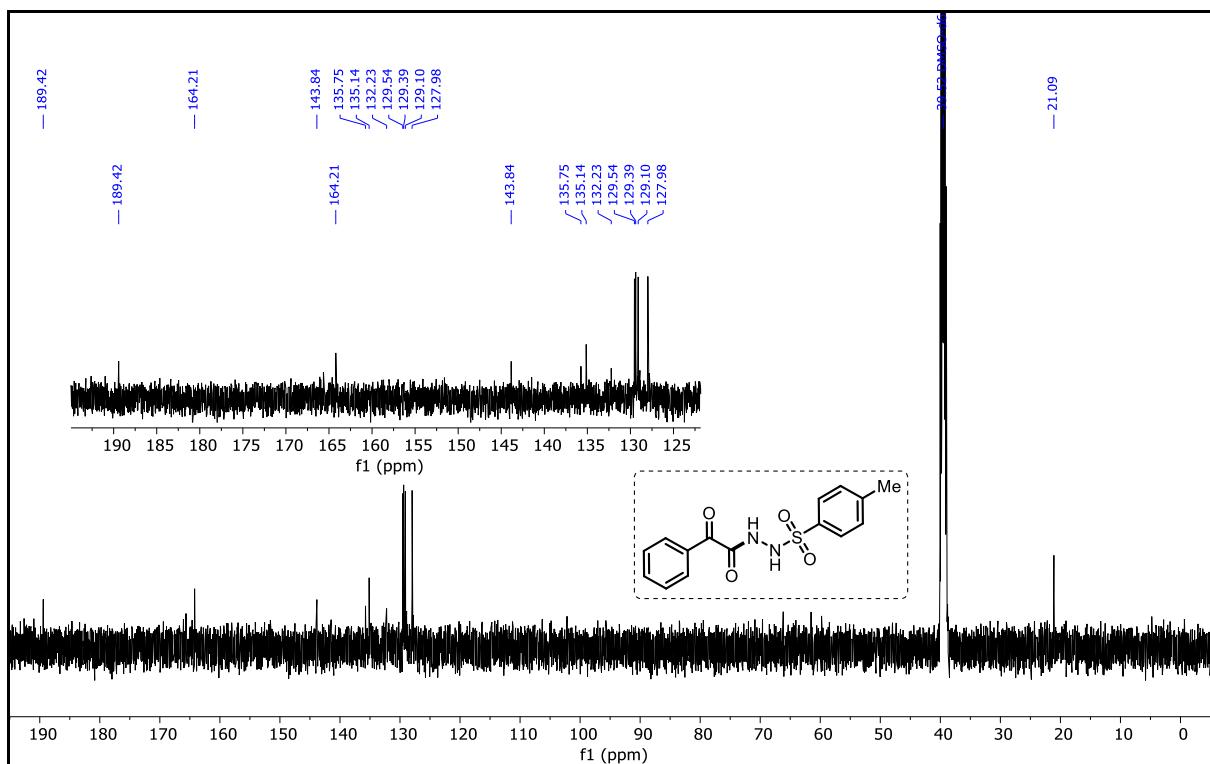


4-Methyl-N'-(2-oxo-2-phenyl acetyl) benzenesulfonohydrazide (3r):

^1H NMR (500 MHz, DMSO- d_6)

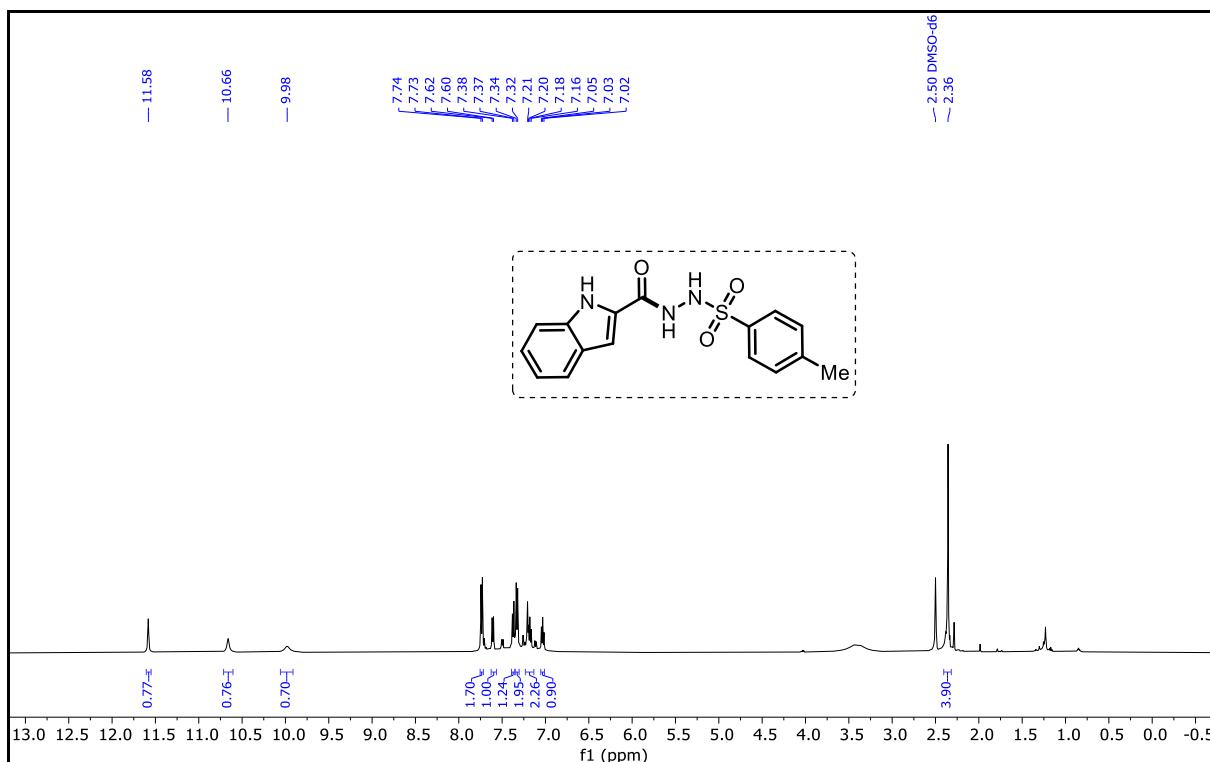


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

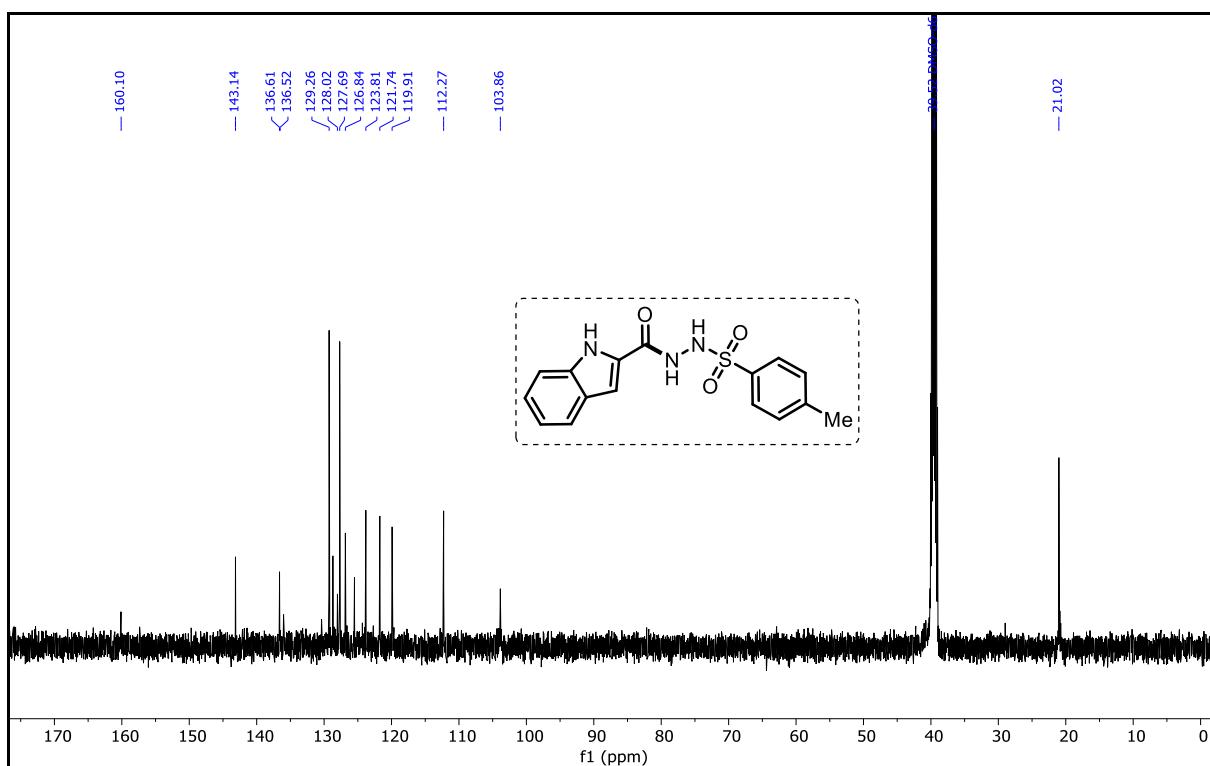


***N'*-(1*H*-Indole-2-carbonyl)-4-methylbenzenesulfonohydrazide (3s):**

^1H NMR (500 MHz, DMSO- d_6)

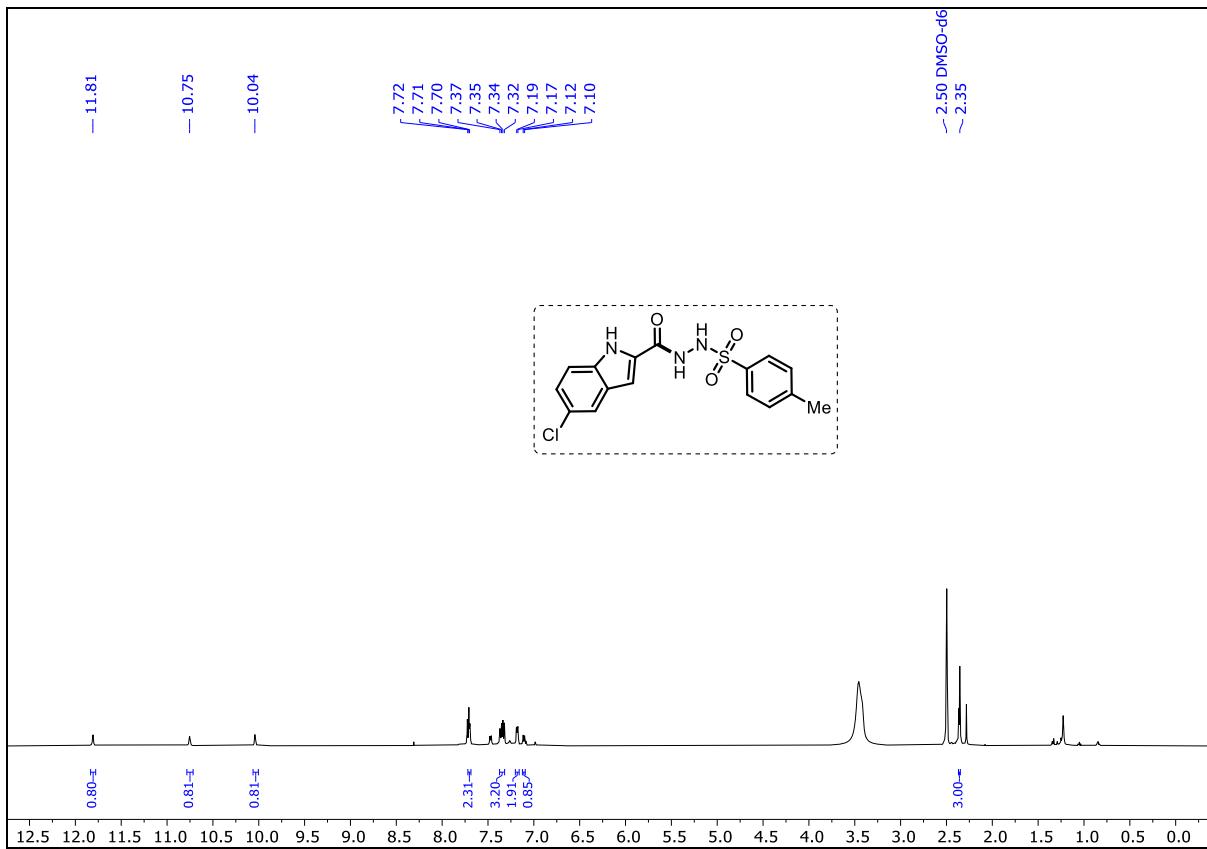


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

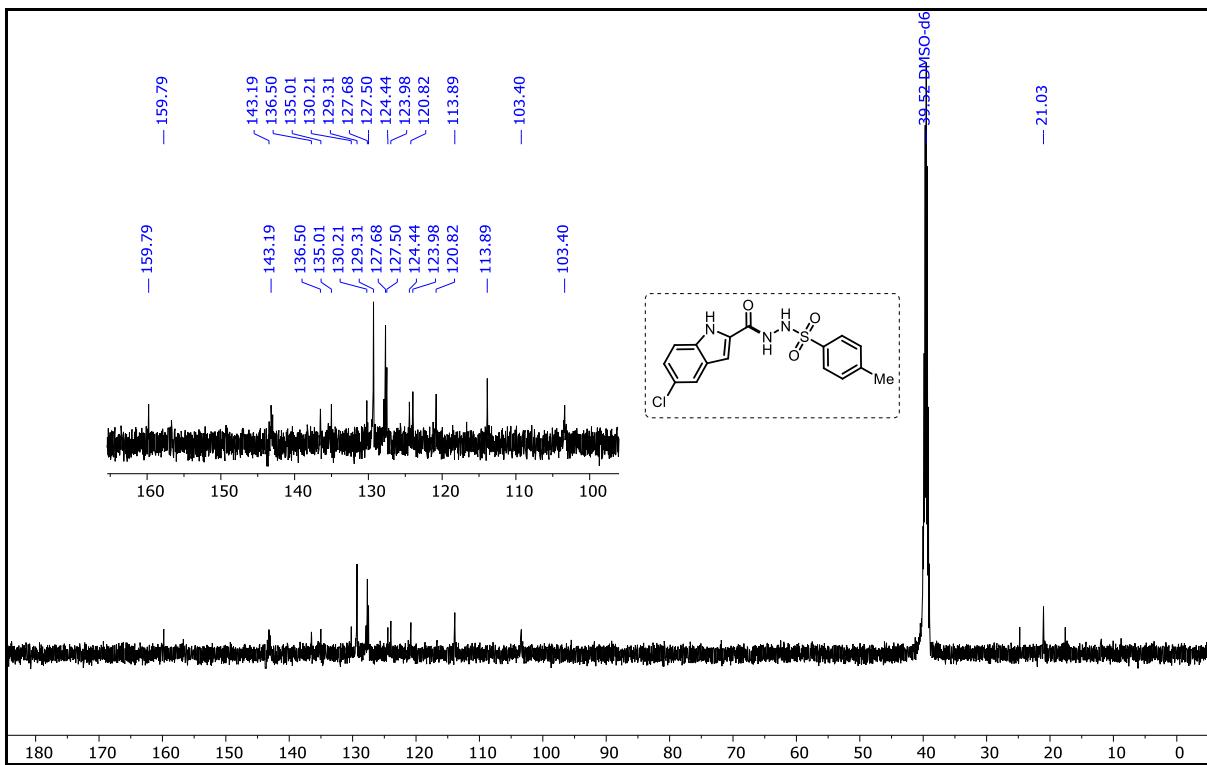


N'-(5-Chloro-1*H*-indole-2-carbonyl)-4-methylbenzenesulfonohydrazide (3t):

¹H NMR (500 MHz, DMSO-d₆)

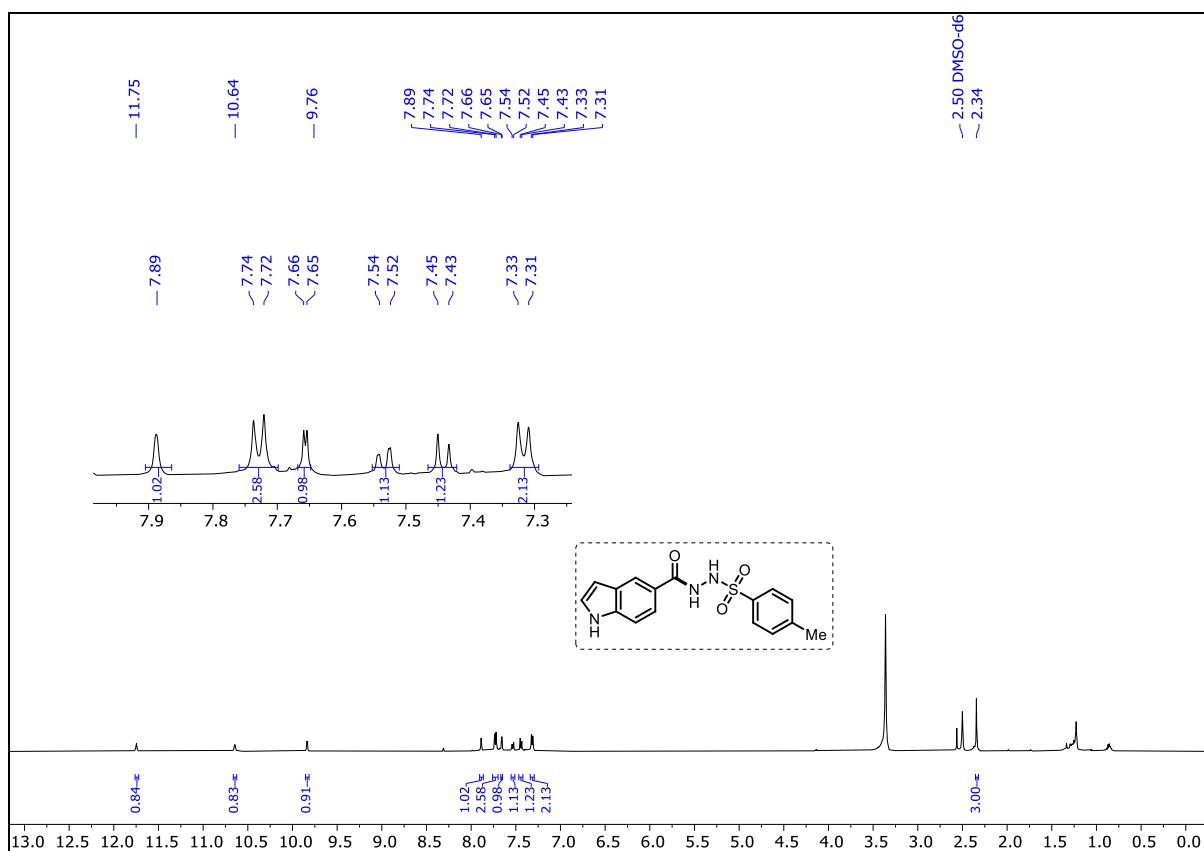


¹³C{¹H} NMR (126 MHz, DMSO-*d*₆)

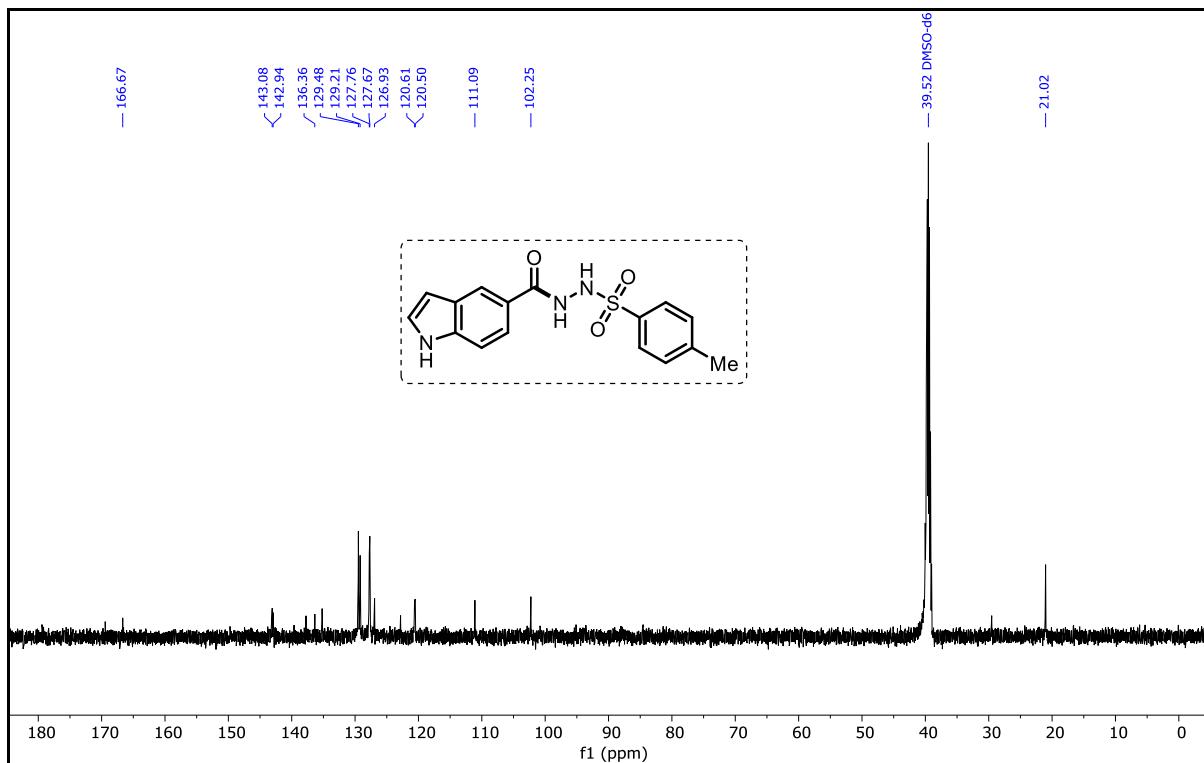


N'-(1*H*-Indole-5-carbonyl)-4-methylbenzenesulfonohydrazide (3u):

¹H NMR (500 MHz, DMSO-*d*₆)

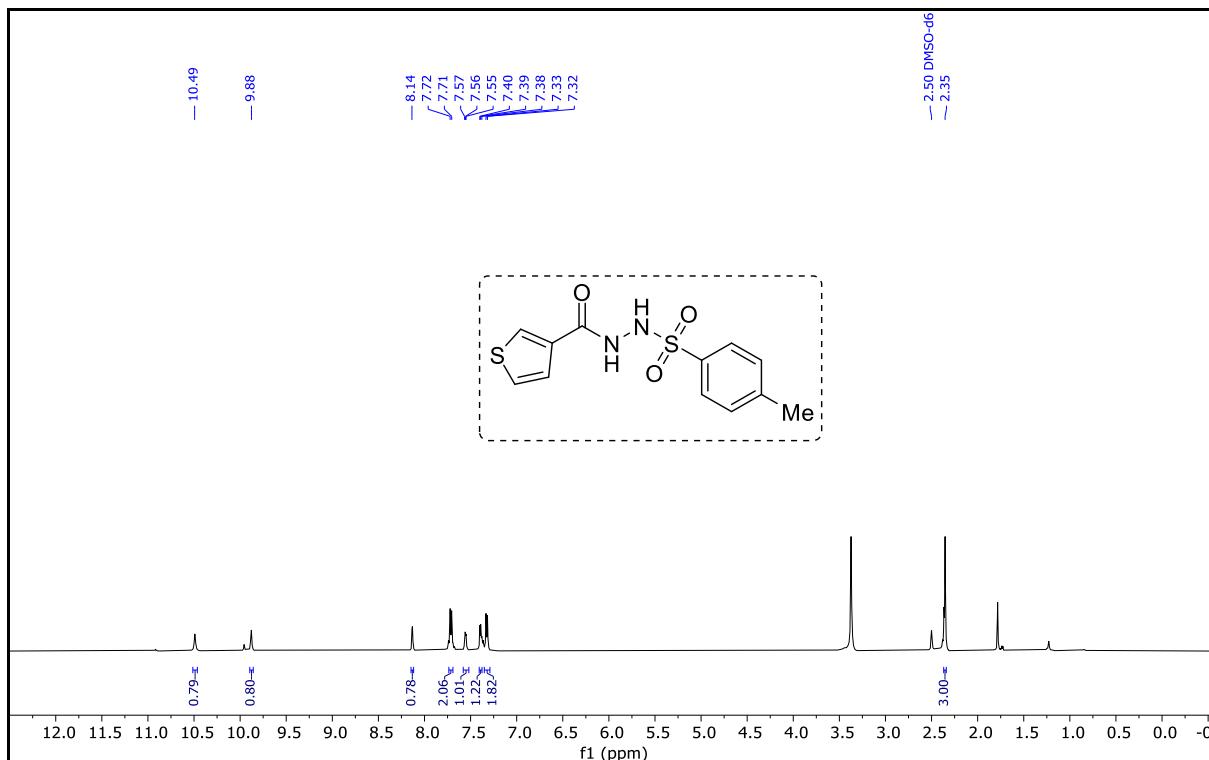


¹³C{¹H} NMR (126 MHz, DMSO-*d*₆)

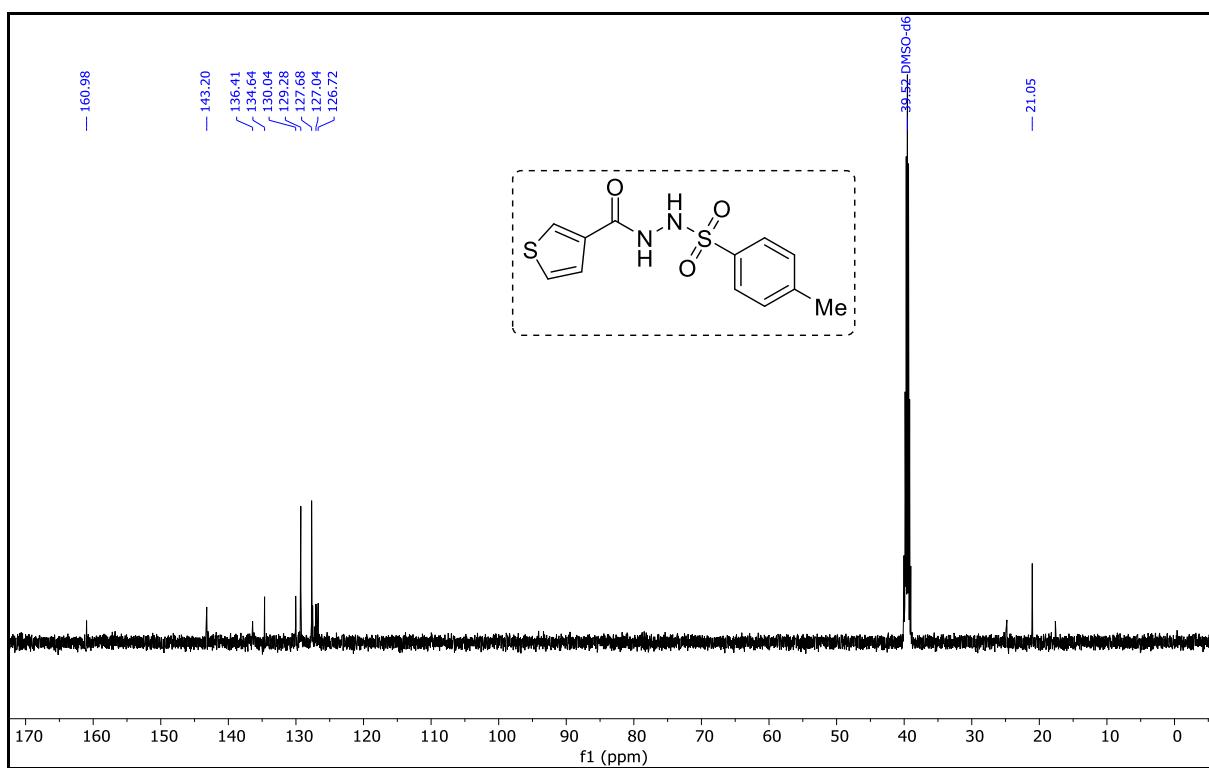


4-Methyl-N'-(thiophene-3-carbonyl)benzenesulfonohydrazide (3v):

^1H NMR (500 MHz, DMSO- d_6)

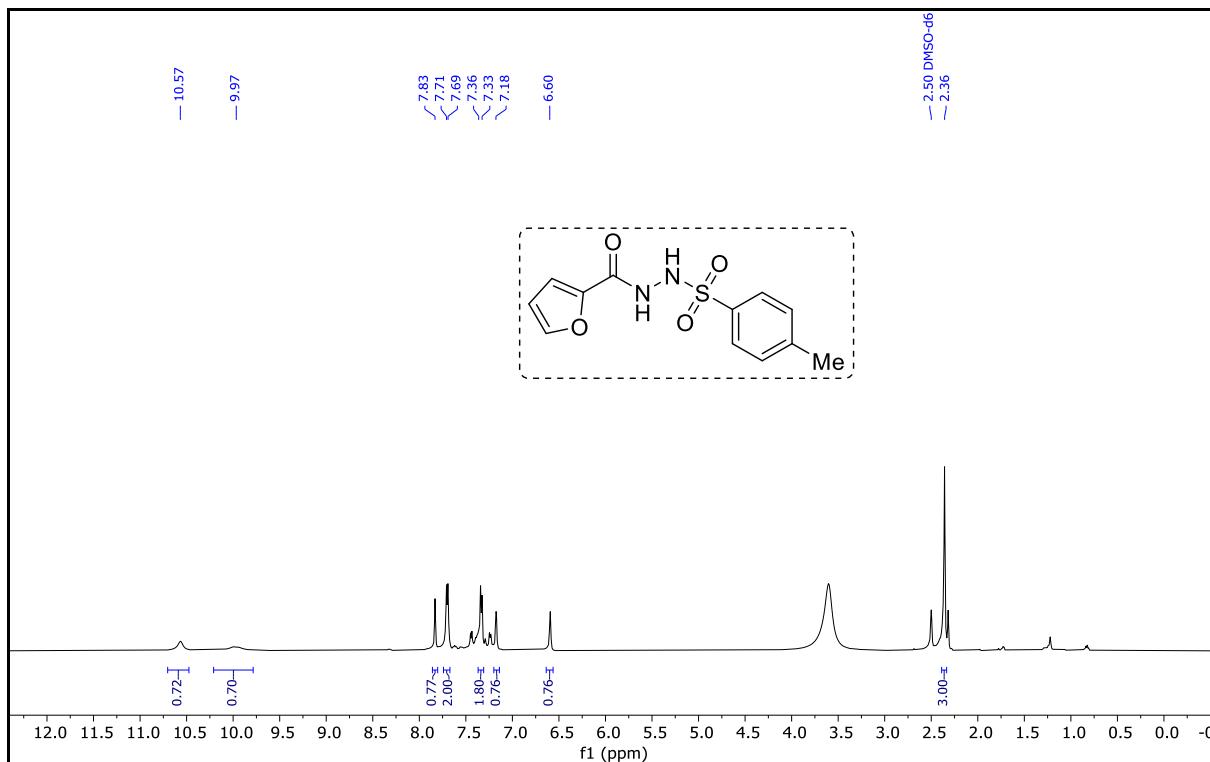


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

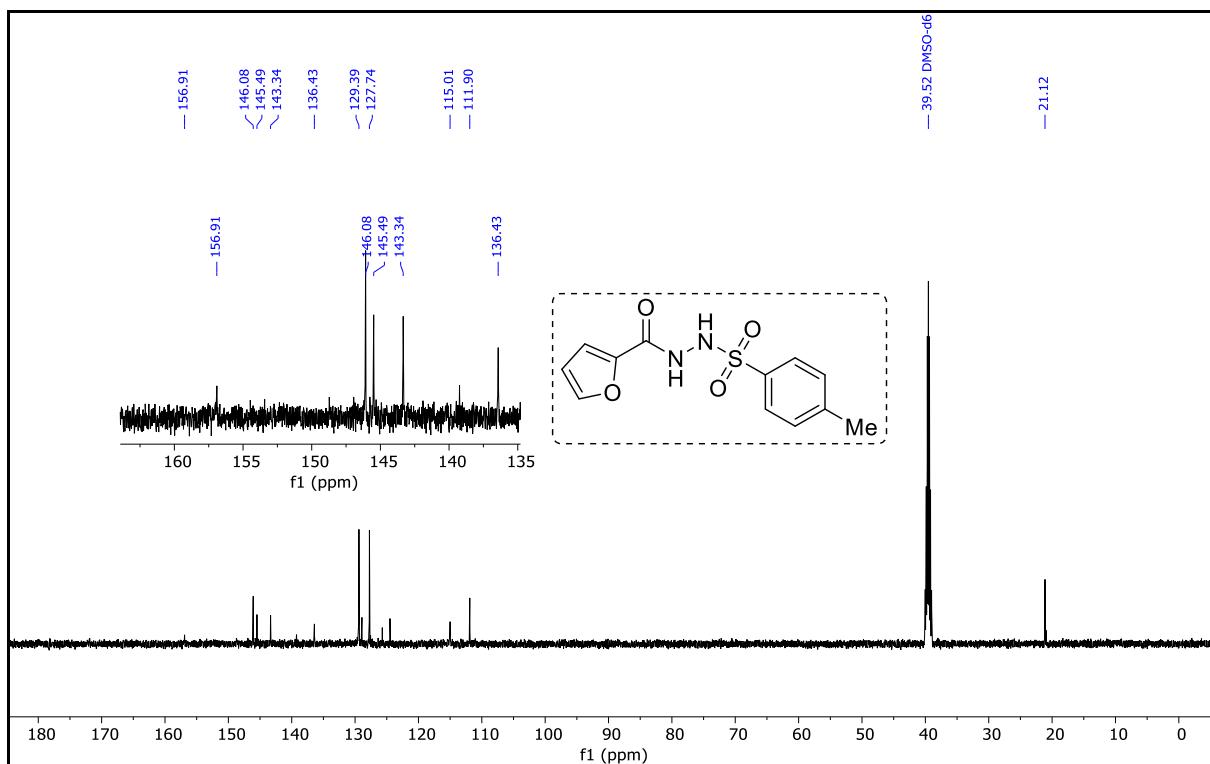


***N'*-(Furan-2-carbonyl)-4-methylbenzenesulfonohydrazide (3w):**

^1H NMR (500 MHz, DMSO- d_6)

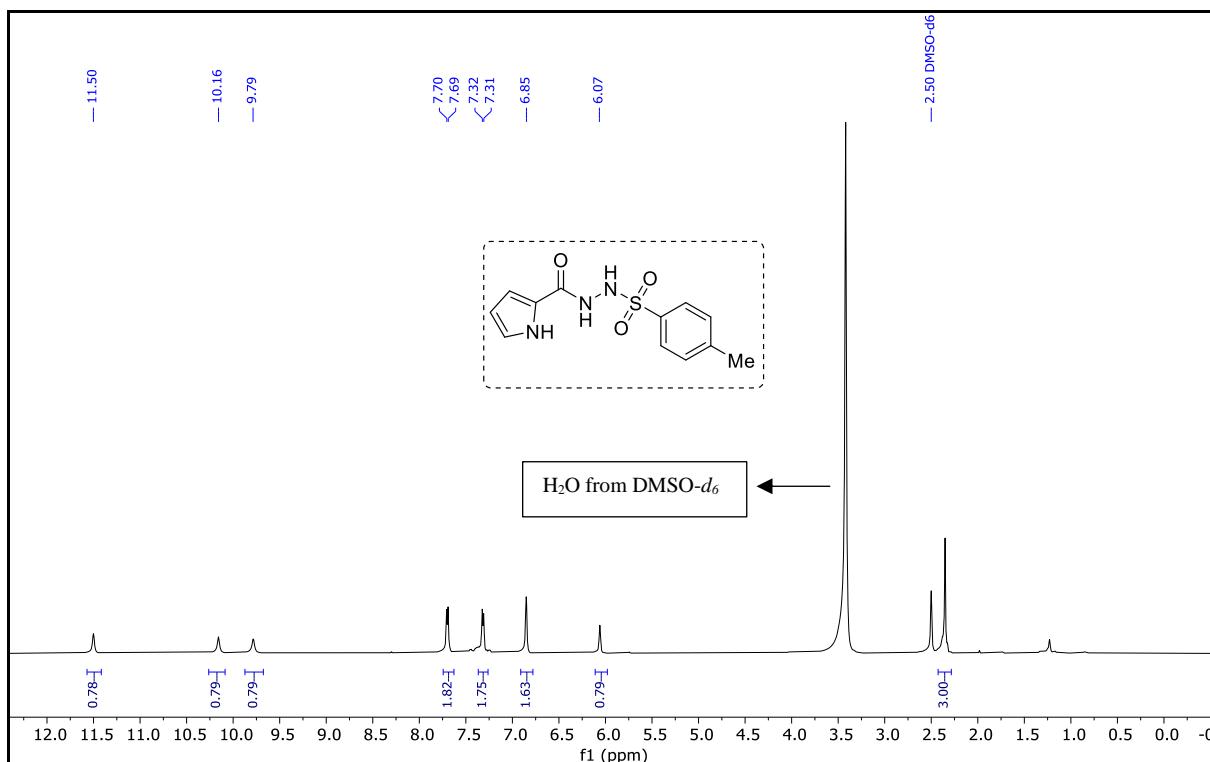


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

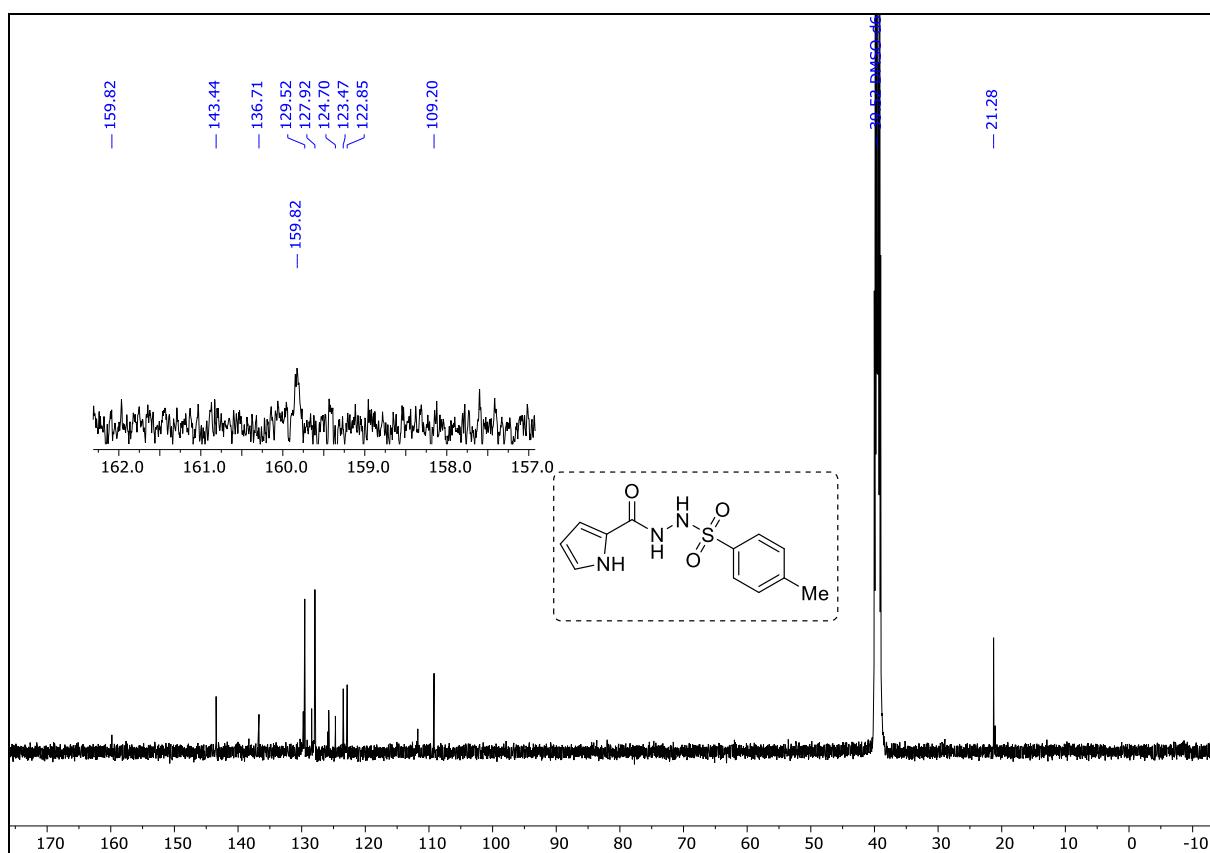


4-Methyl-N'-(1*H*-pyrrole-2-carbonyl) benzenesulfonohydrazide (3x):

^1H NMR (500 MHz, DMSO- d_6)

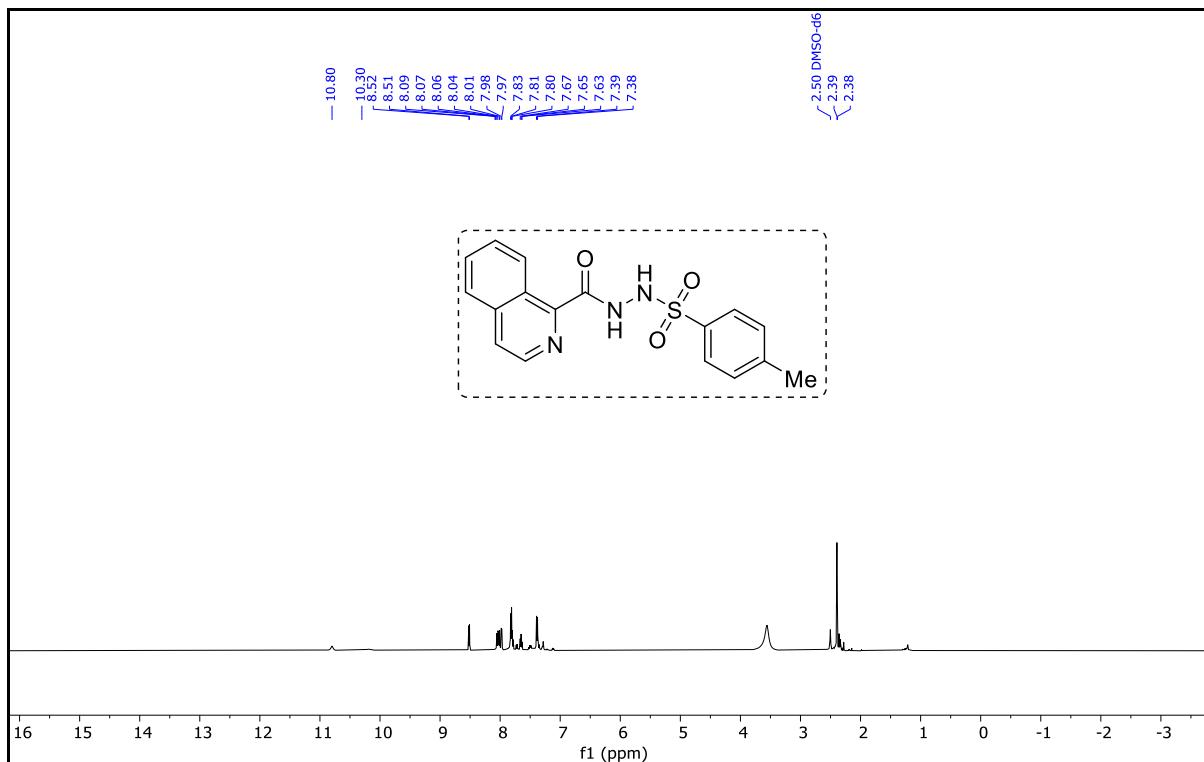


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

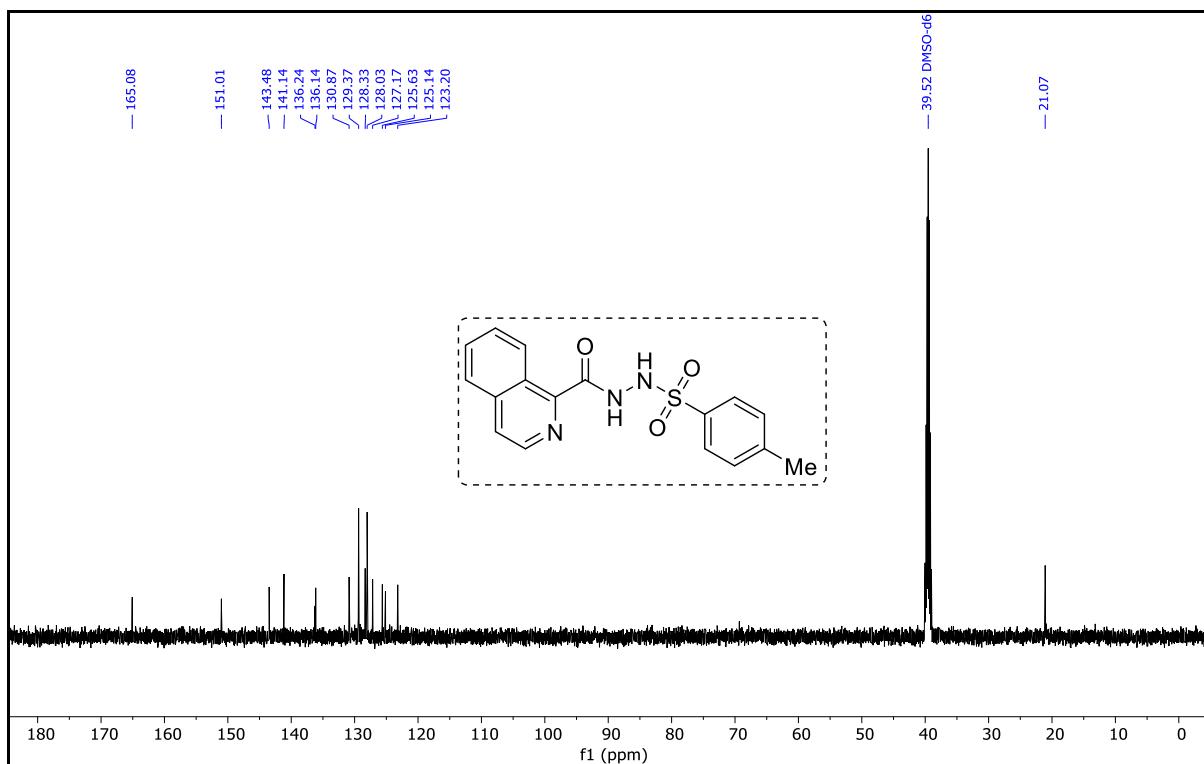


***N'*-(Isoquinoline-1-carbonyl)-4-methylbenzenesulfonohydrazide (3y):**

^1H NMR (500 MHz, DMSO- d_6)

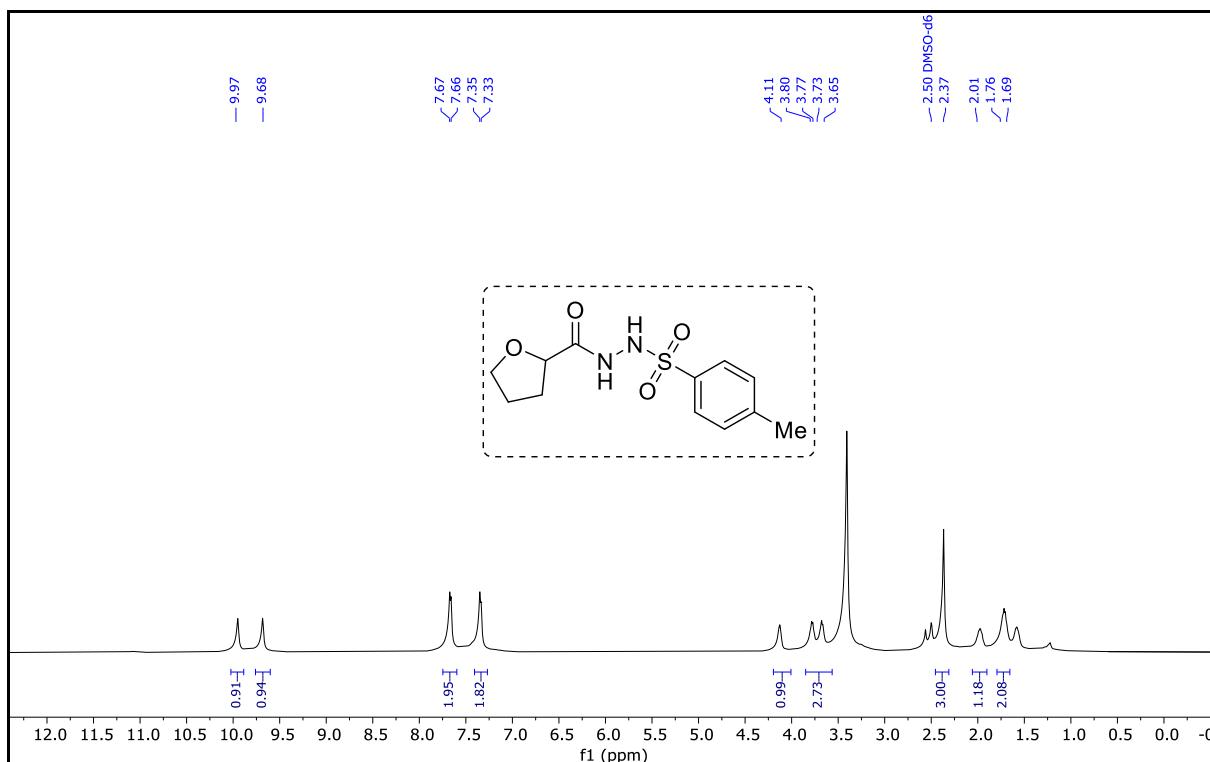


$^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, DMSO- d_6)

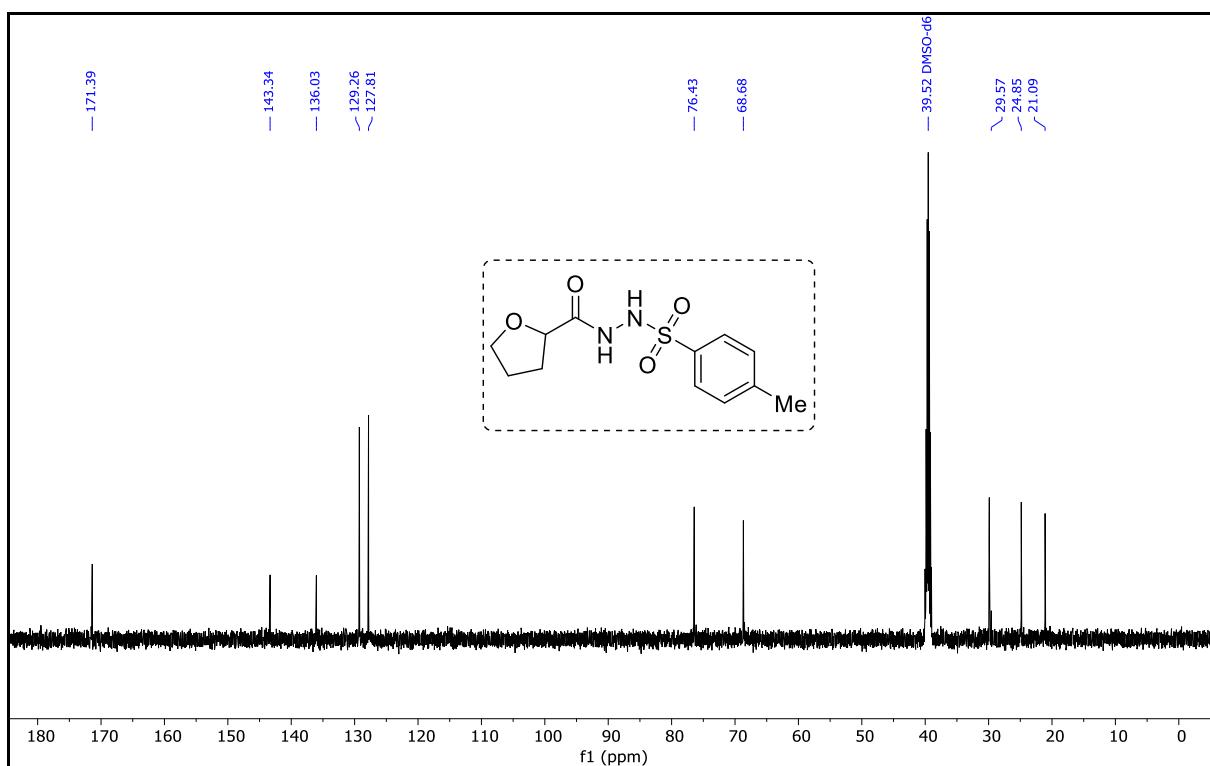


4-Methyl-N'-(tetrahydrofuran-2-carbonyl) benzenesulfonohydrazide (3z):

^1H NMR (500 MHz, DMSO- d_6)

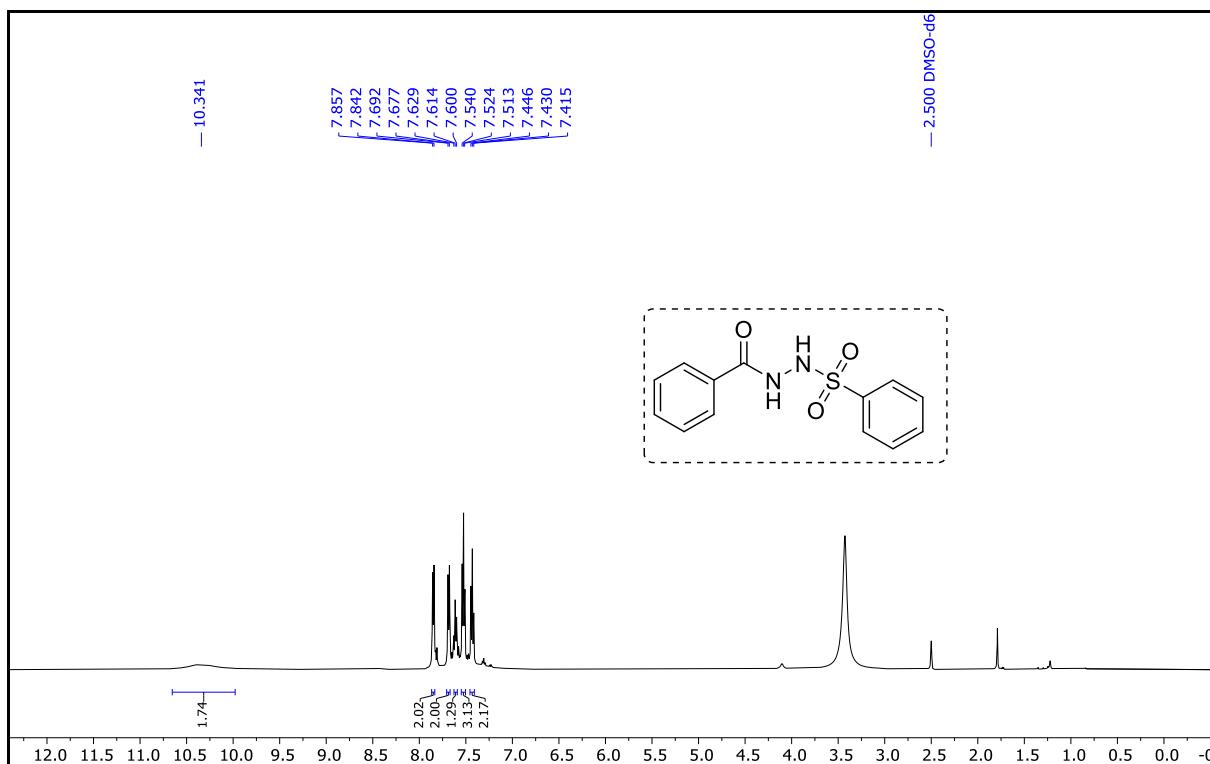


$^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, DMSO- d_6)

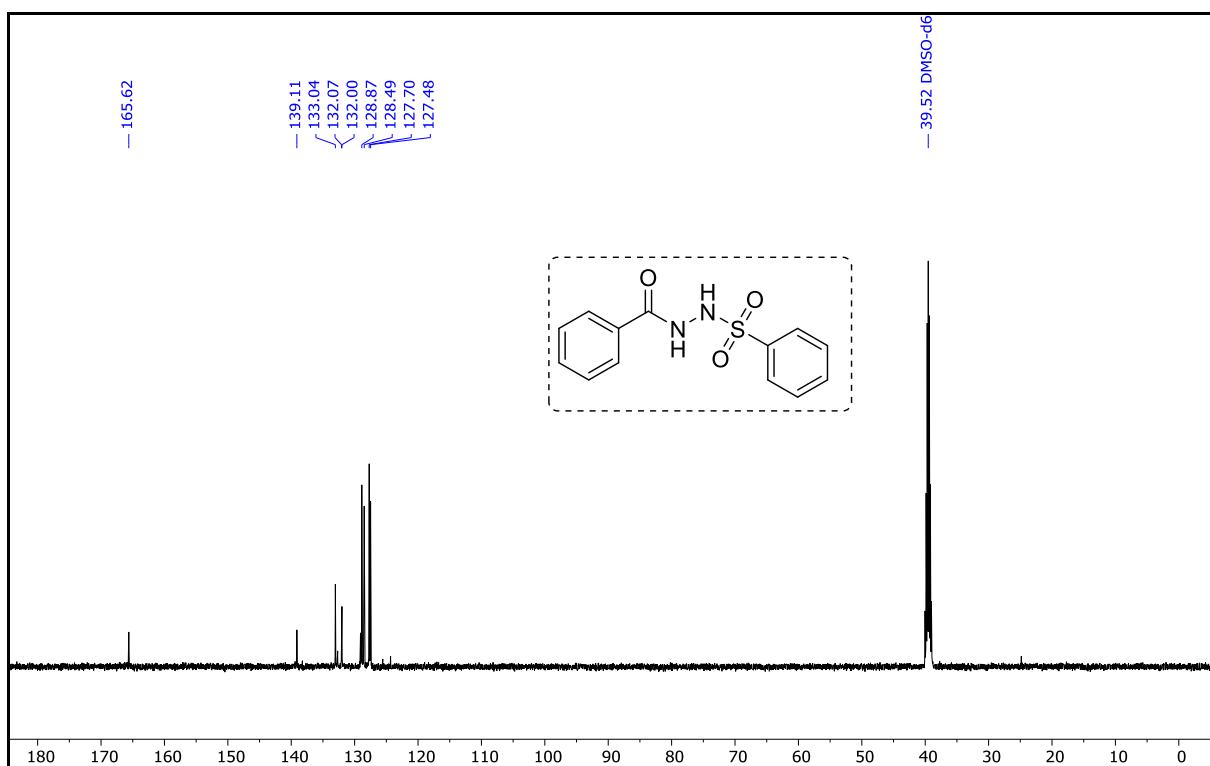


***N'*-Benzoylbenzenesulfonohydrazide (3aa):**

^1H NMR (500 MHz, DMSO- d_6)

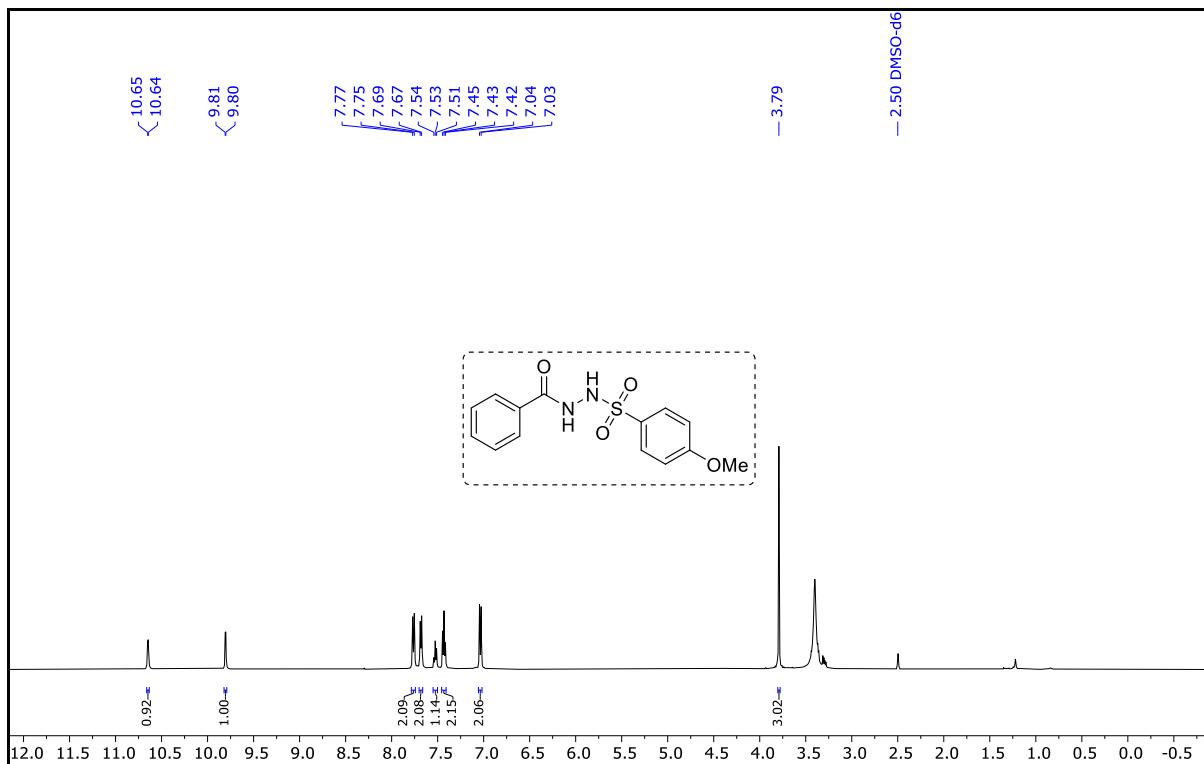


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

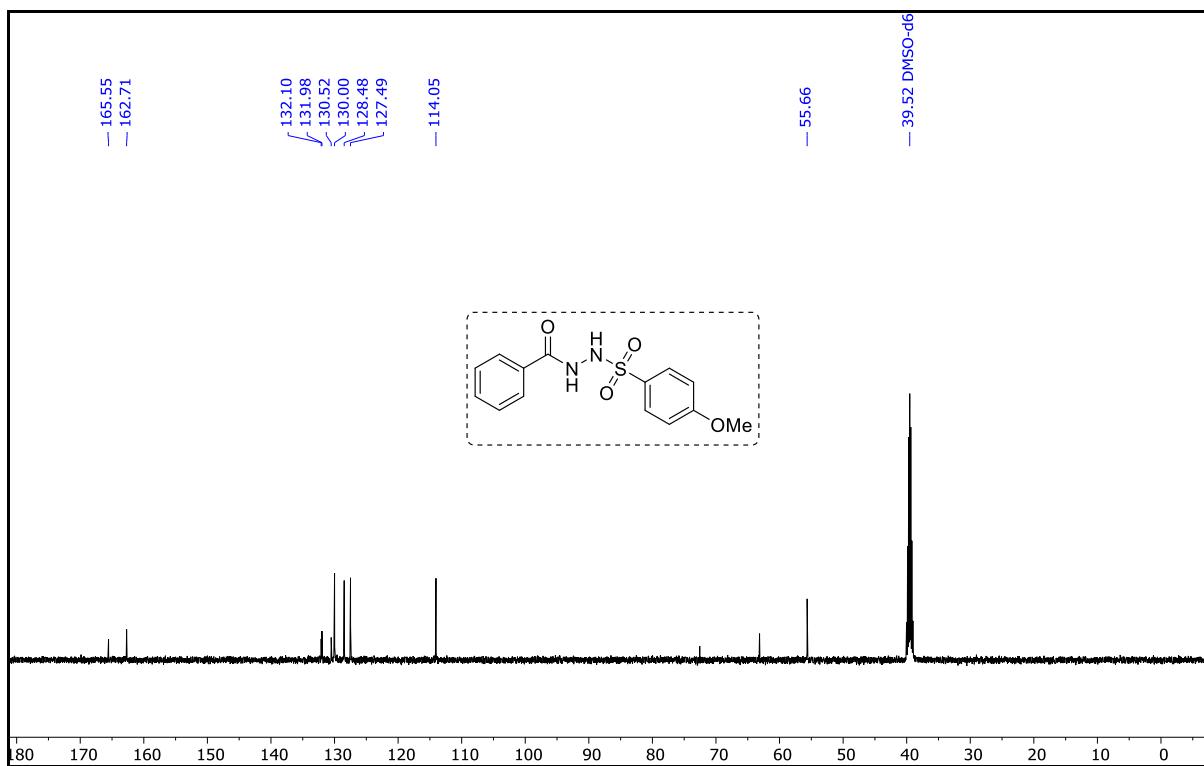


***N'*-Benzoyl-4-methoxybenzenesulfonohydrazide (3ab):**

^1H NMR (500 MHz, DMSO- d_6)

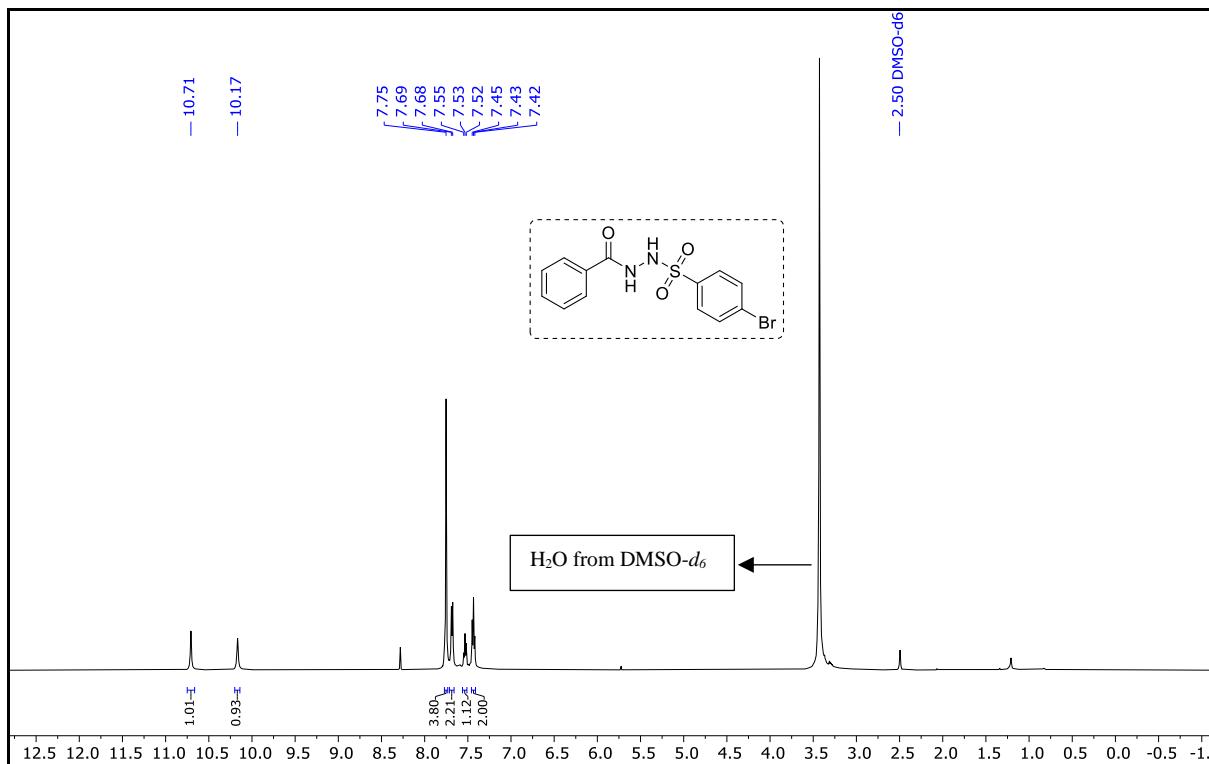


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

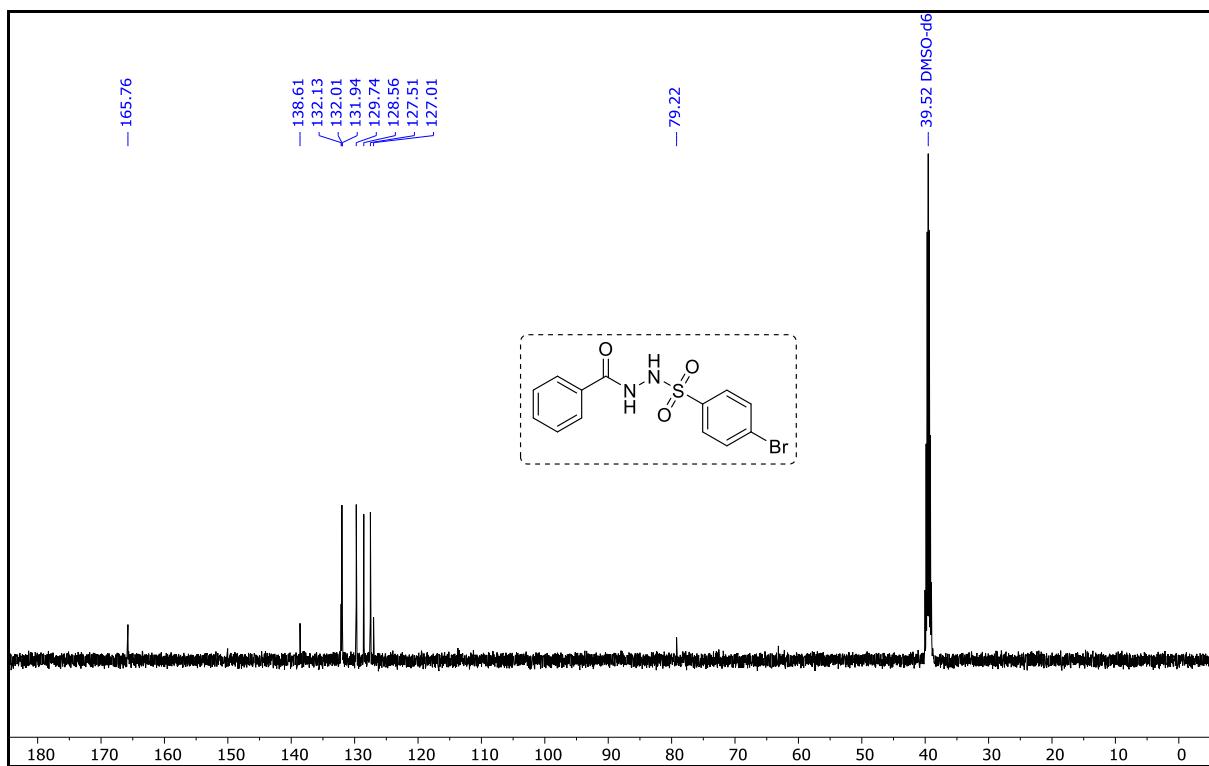


***N'*-Benzoyl-4-bromobenzenesulfonohydrazide (3ac):**

^1H NMR (500 MHz, DMSO- d_6)

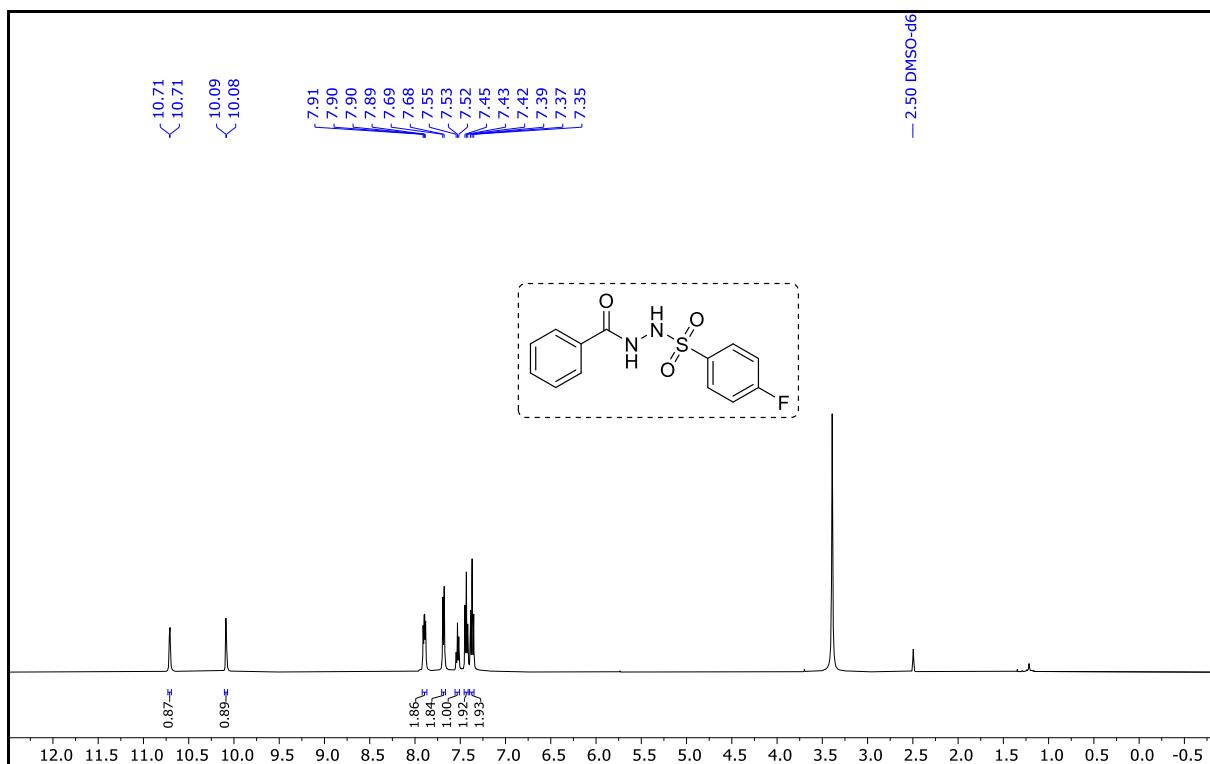


$^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, DMSO- d_6)

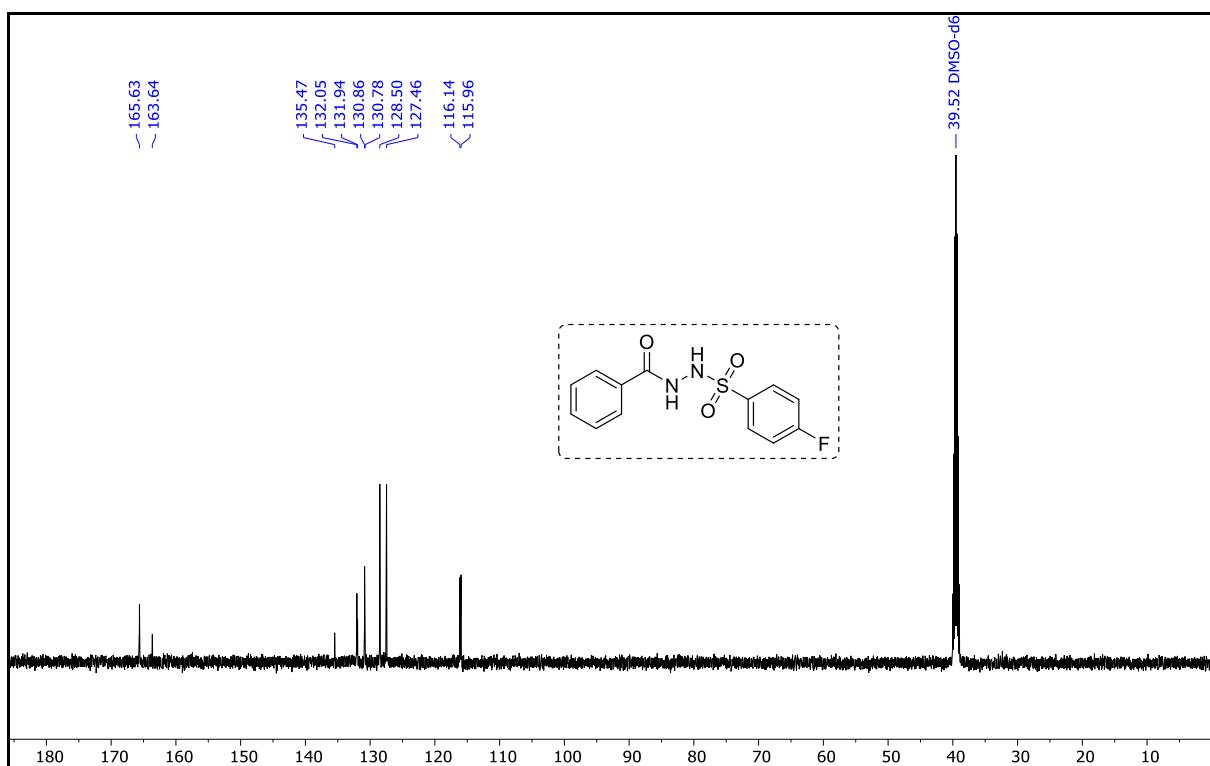


***N'*-Benzoyl-4-fluorobenzenesulfonohydrazide (3ad):**

^1H NMR (500 MHz, DMSO- d_6)

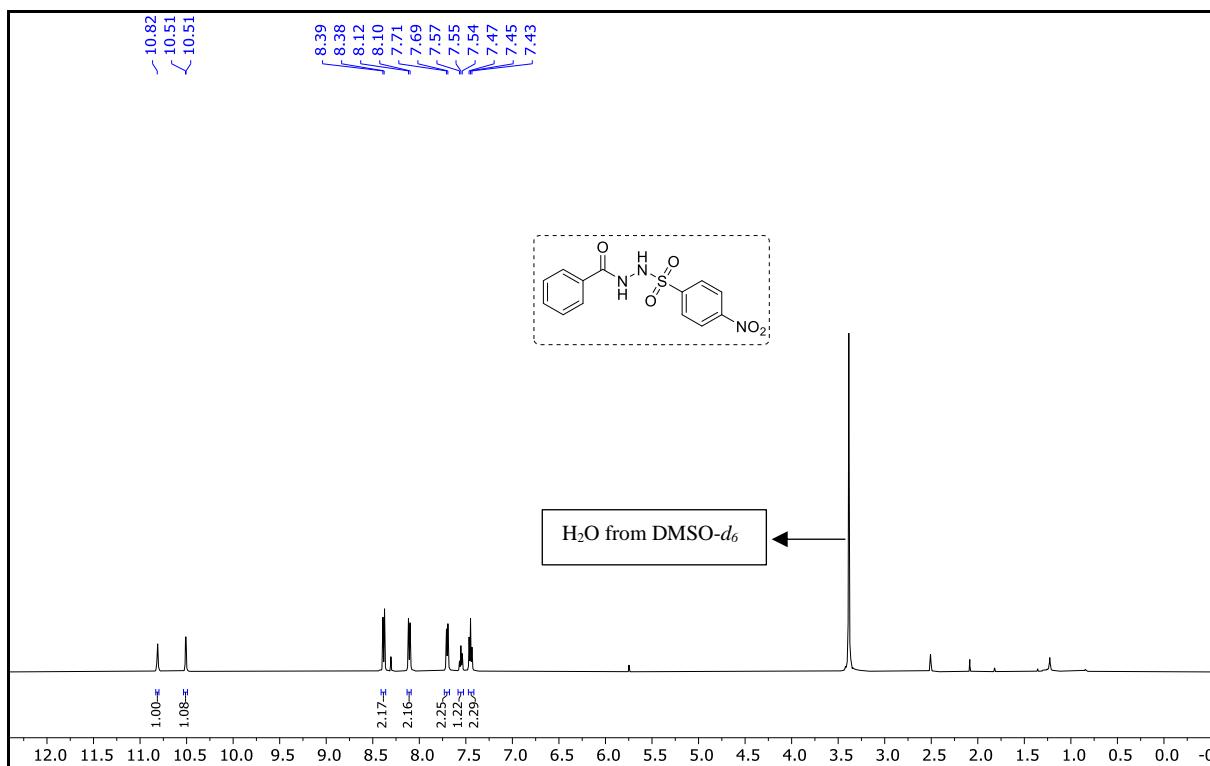


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

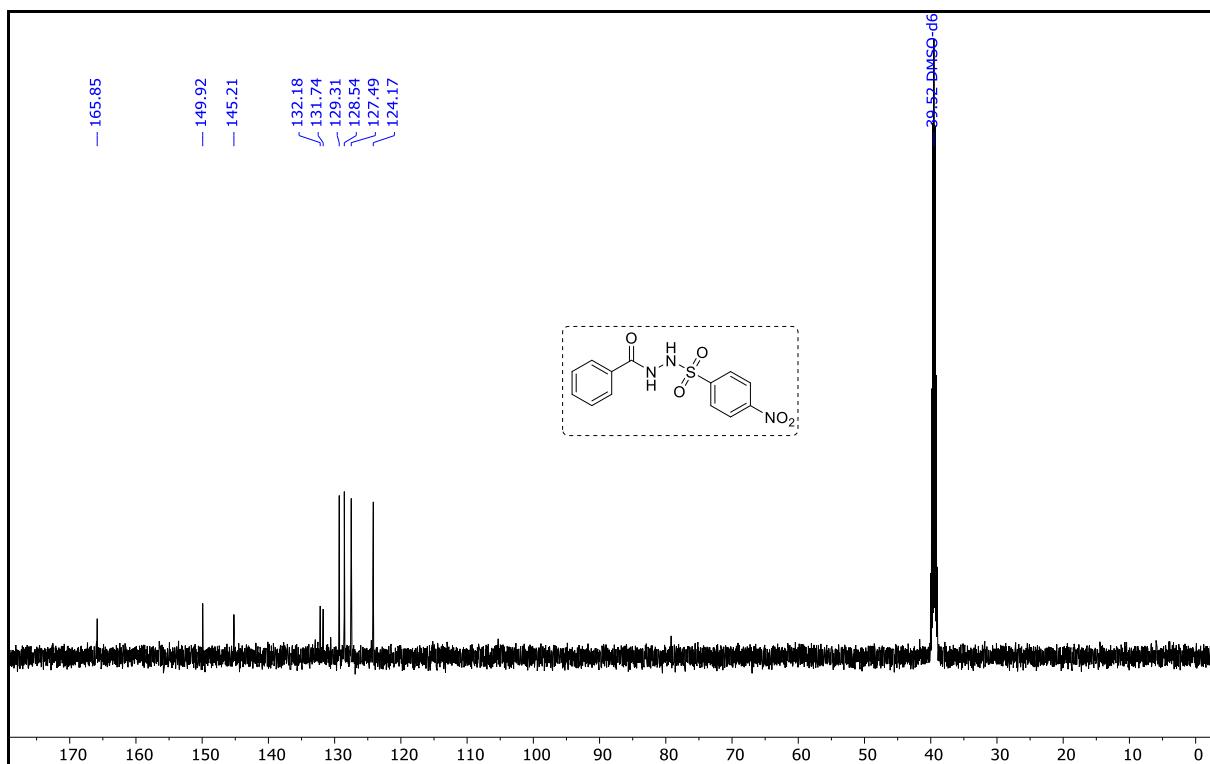


***N'*-Benzoyl-4-nitrobenzenesulfonohydrazide (3ae):**

^1H NMR (500 MHz, DMSO- d_6)

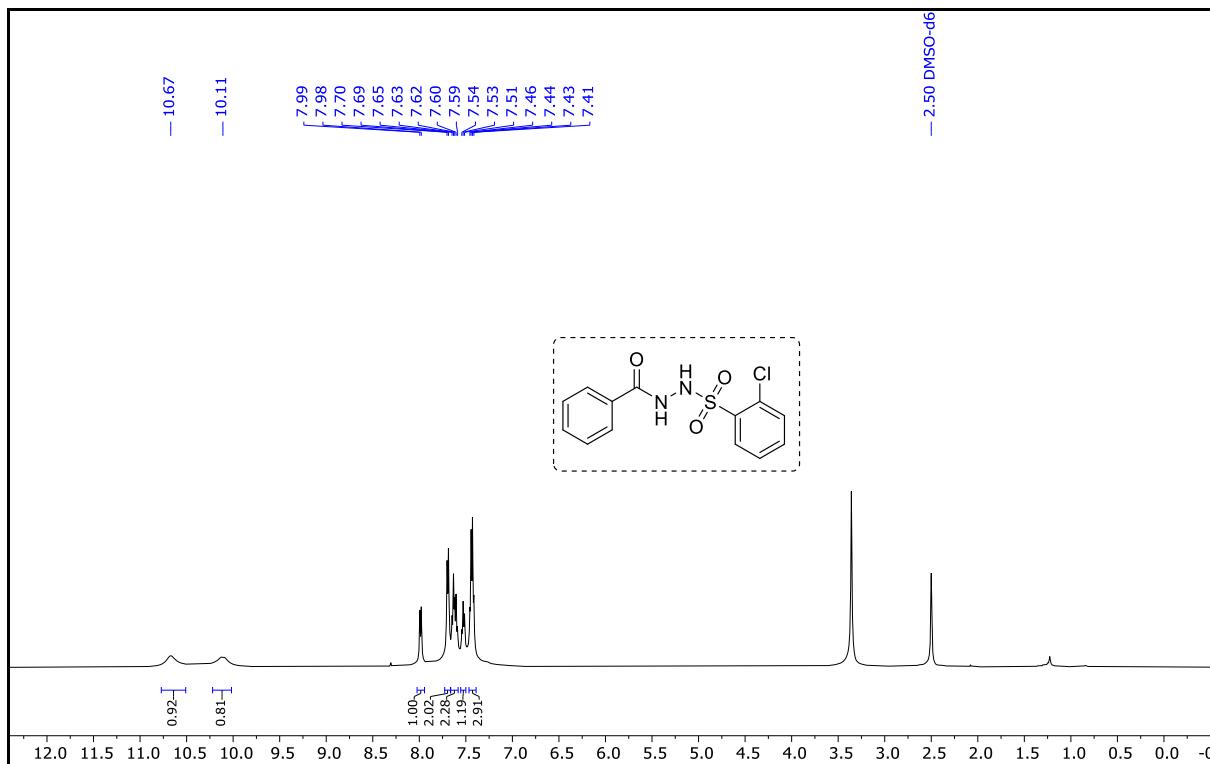


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

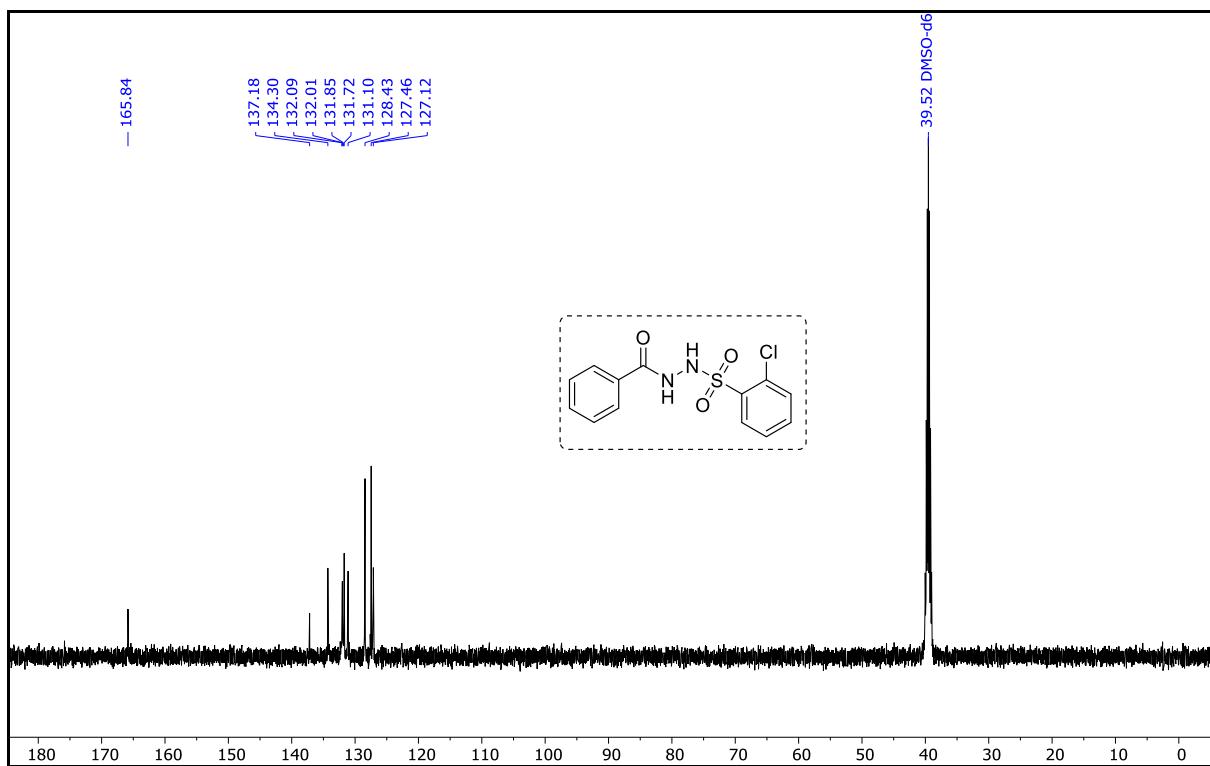


***N'*-Benzoyl-2-chlorobenzenesulfonohydrazide (3af):**

^1H NMR (500 MHz, DMSO- d_6)

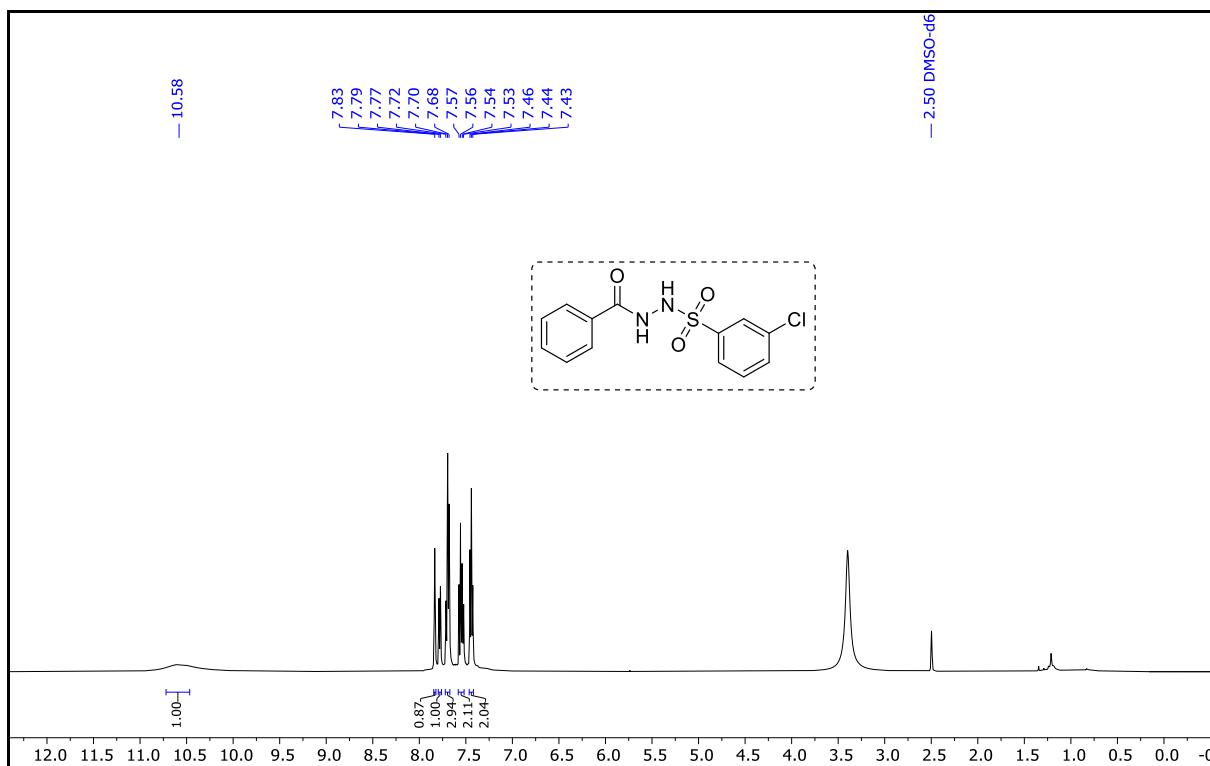


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

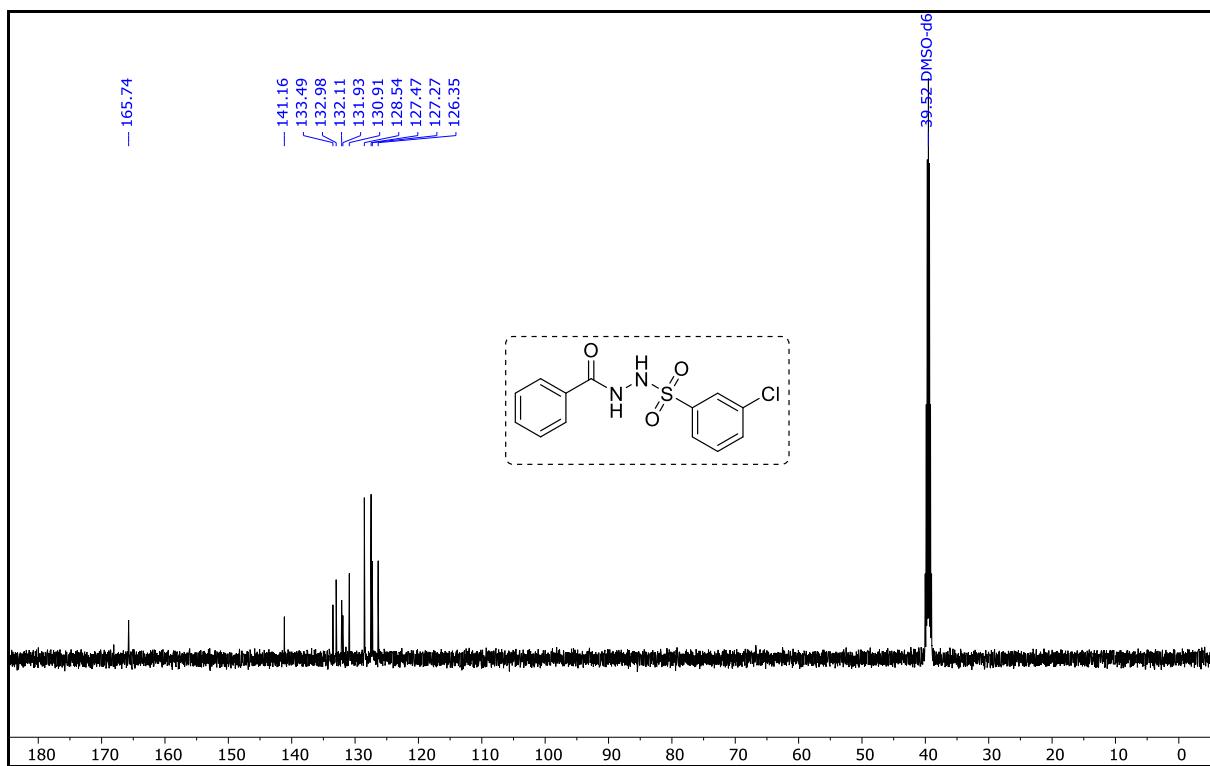


***N'*-Benzoyl-3-chlorobenzenesulfonohydrazide (3ag):**

^1H NMR (500 MHz, DMSO- d_6)

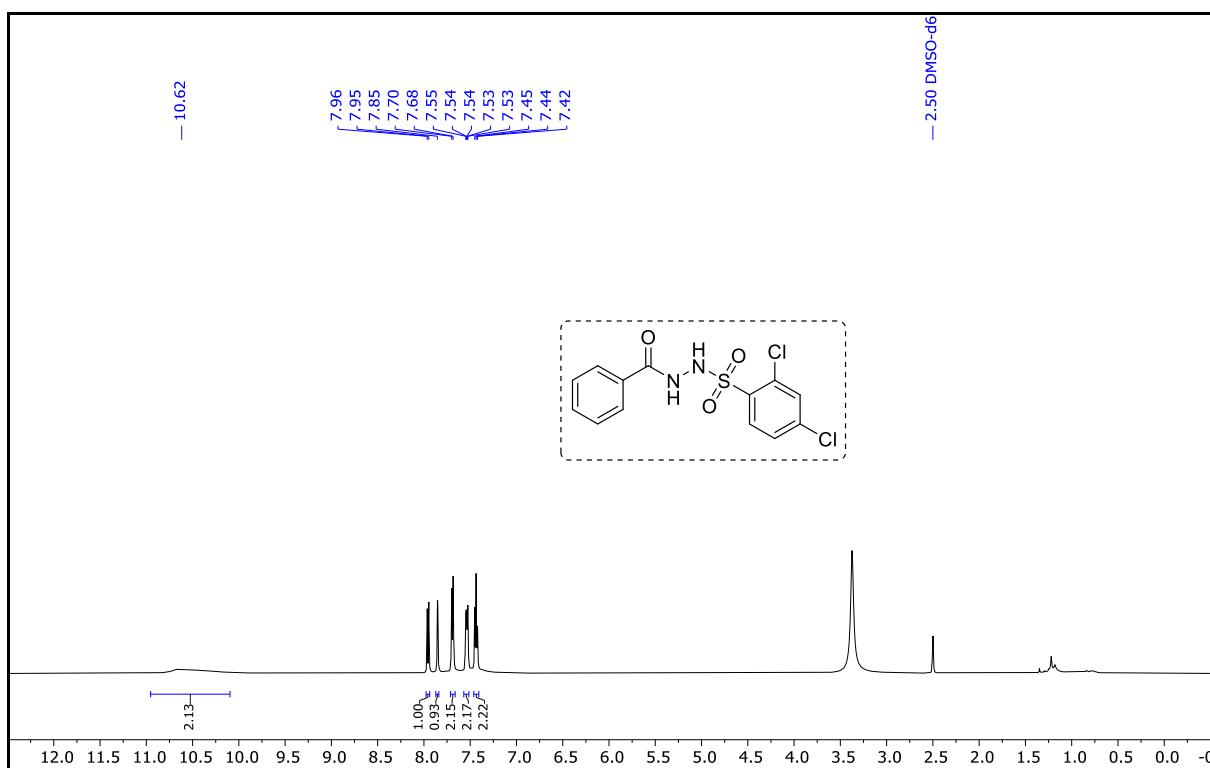


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

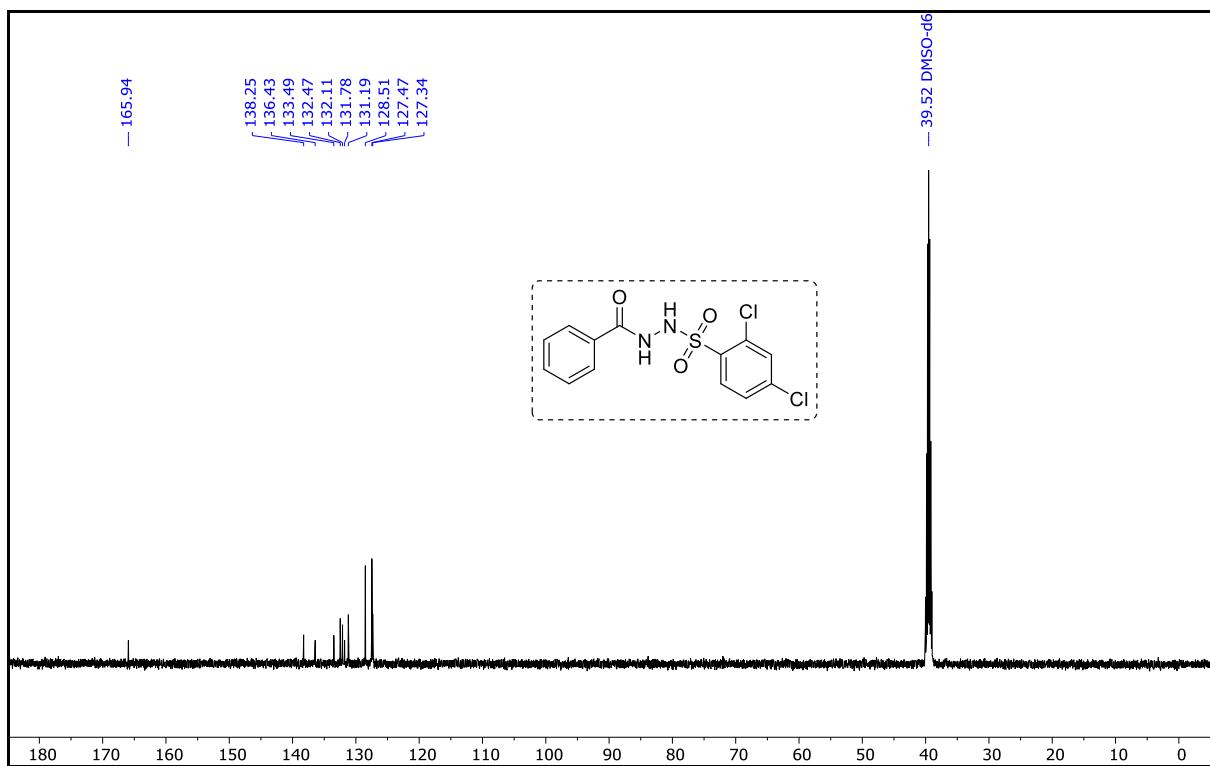


***N'*-Benzoyl-2,4-dichlorobenzenesulfonohydrazide (3ah):**

^1H NMR (500 MHz, DMSO- d_6)

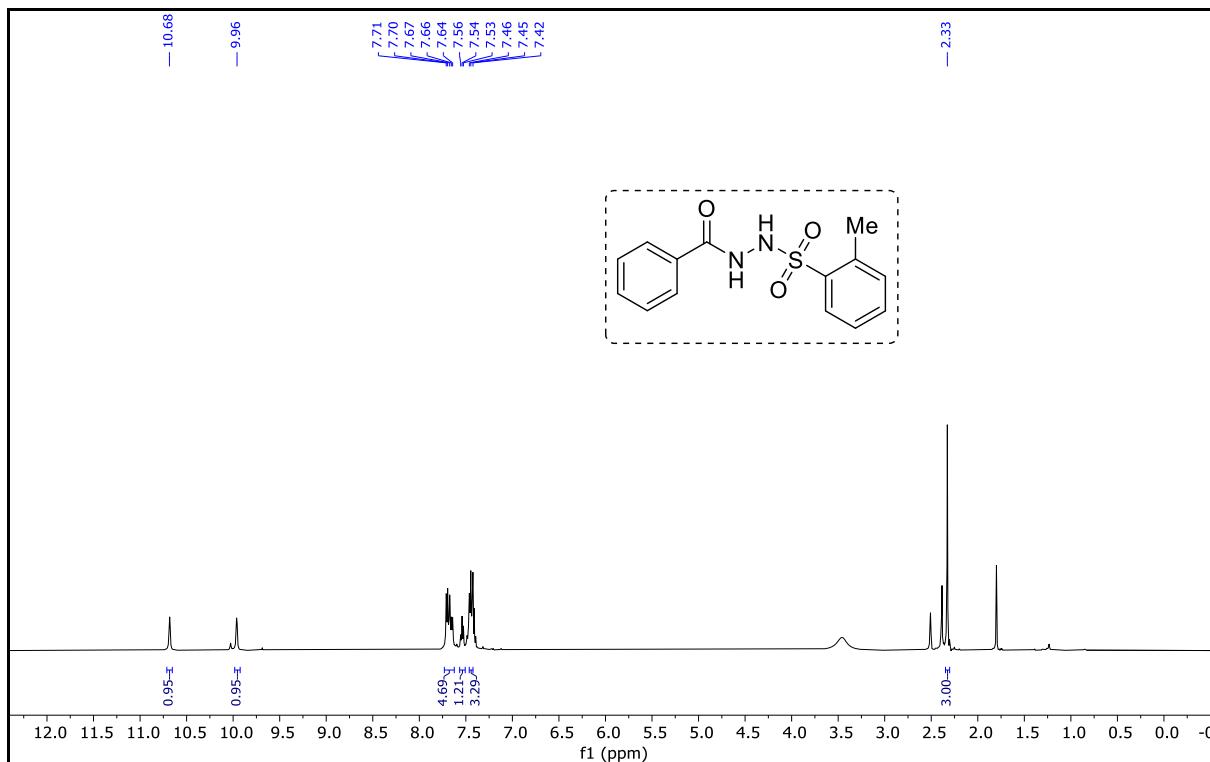


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

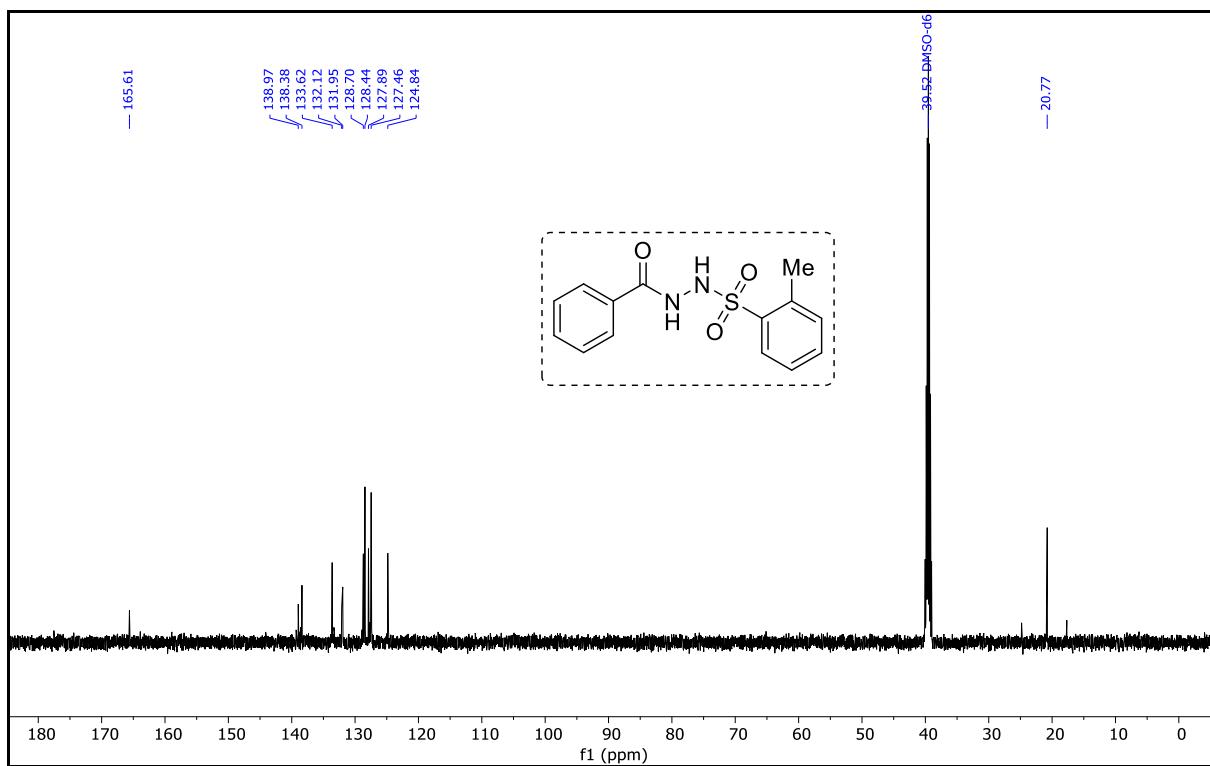


***N'*-Benzoyl-2-methylbenzenesulfonohydrazide (3ai):**

^1H NMR (500 MHz, DMSO- d_6)

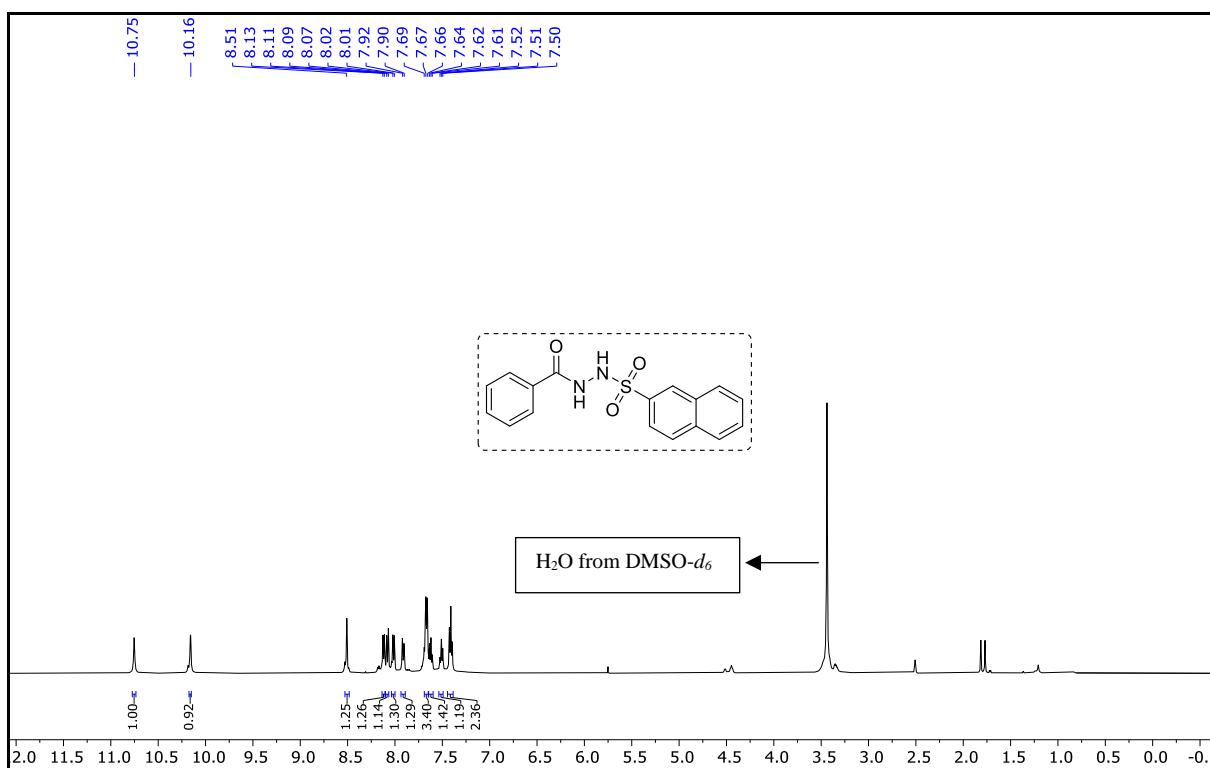


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

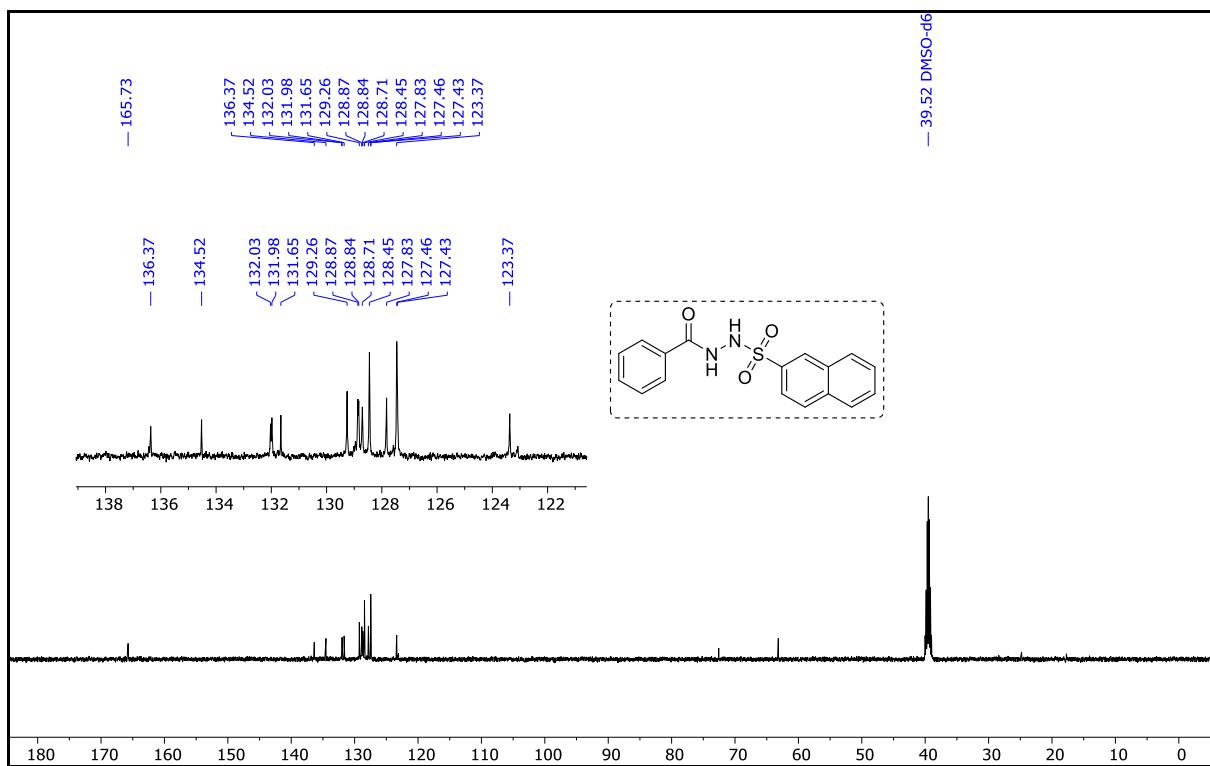


***N'*-Benzoylnaphthalene-2-sulfonohydrazide (3aj):**

¹H NMR (500 MHz, DMSO-*d*₆)

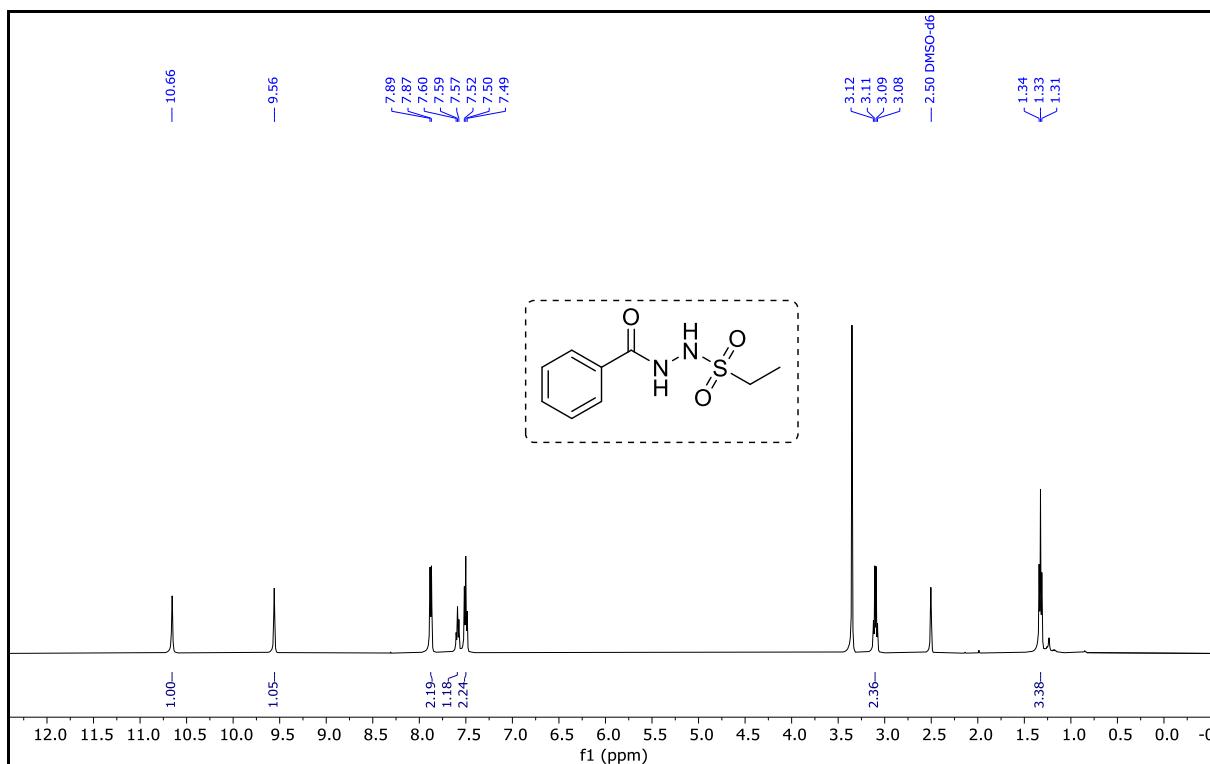


¹³C{¹H} NMR (126 MHz, DMSO-*d*₆)

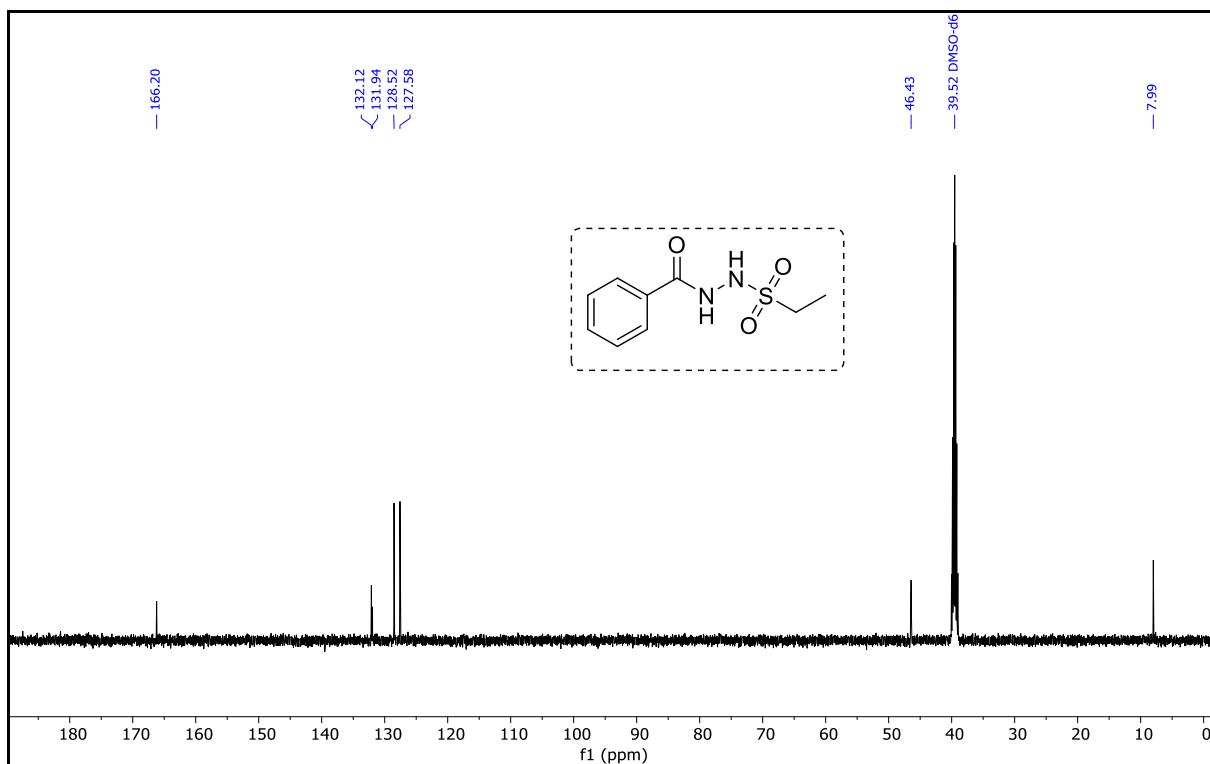


***N'*-Benzoylethanethanesulfonohydrazide (3ak):**

^1H NMR (500 MHz, DMSO- d_6)

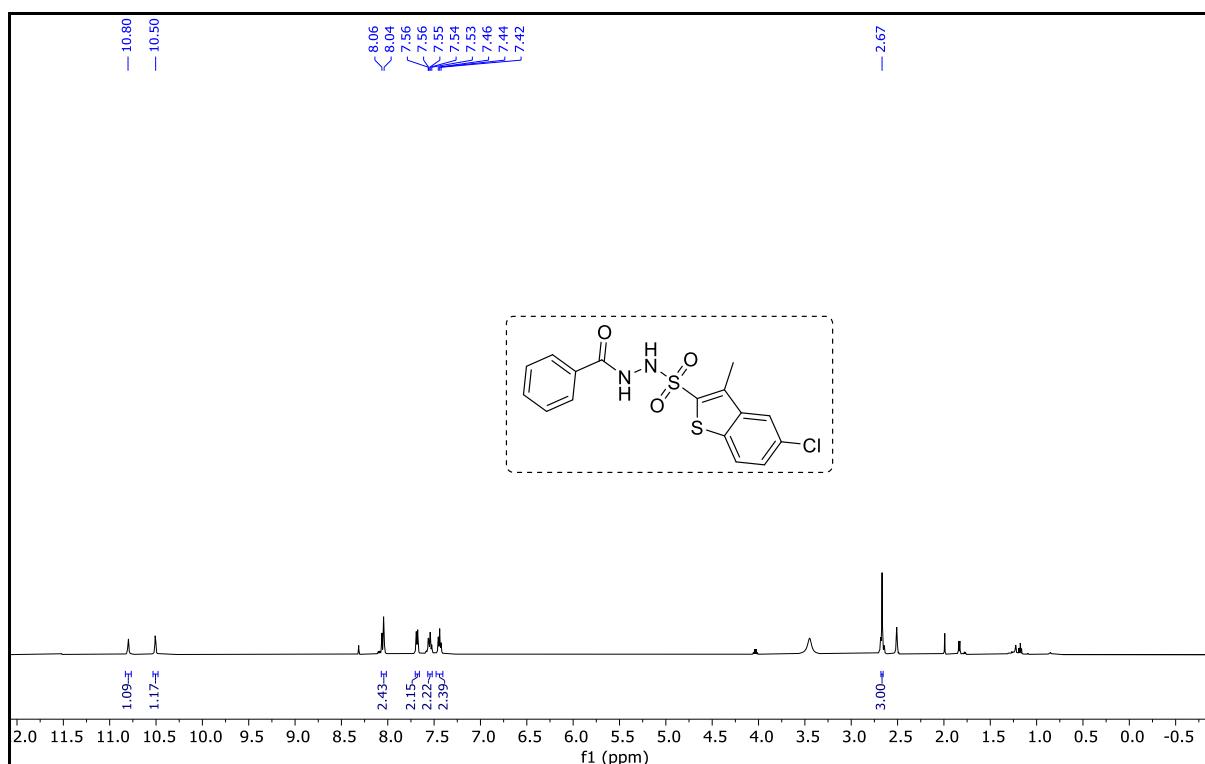


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

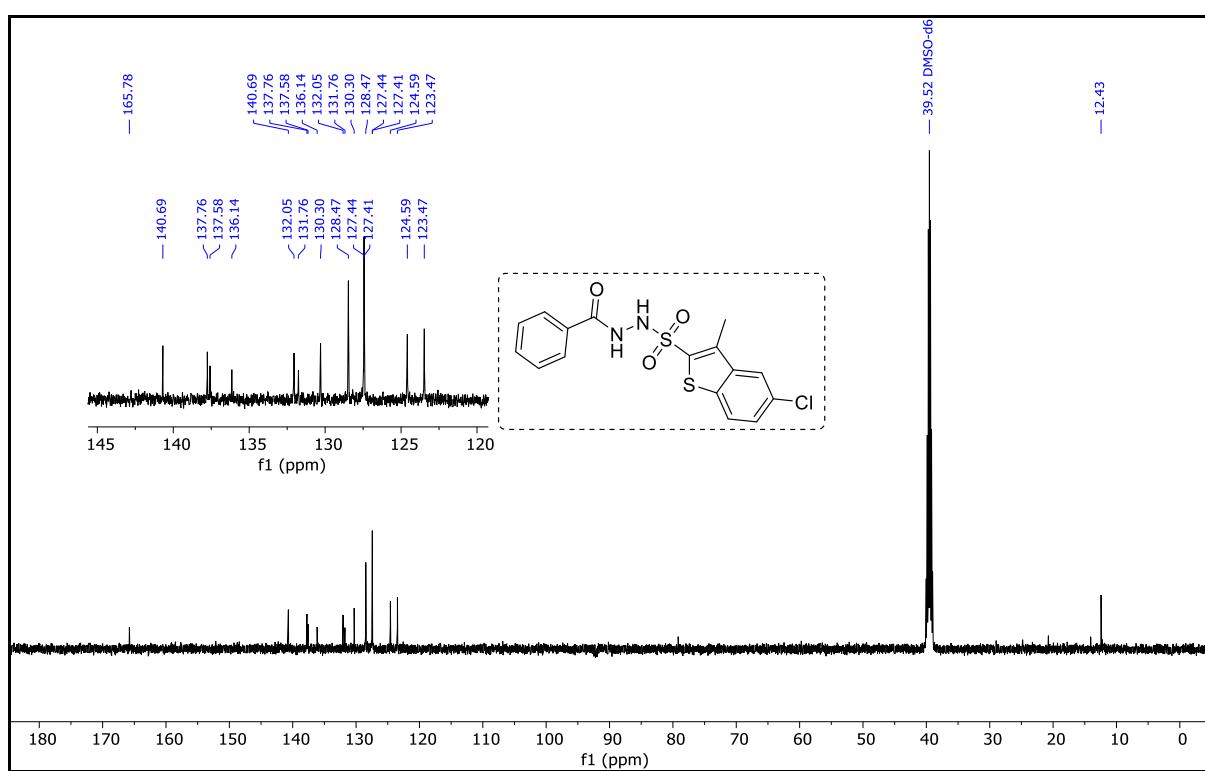


***N'*-Benzoyl-5-chloro-3-methylbenzo[b]thiophene-2-sulfonohydrazide (3al):**

^1H NMR (500 MHz, DMSO- d_6)

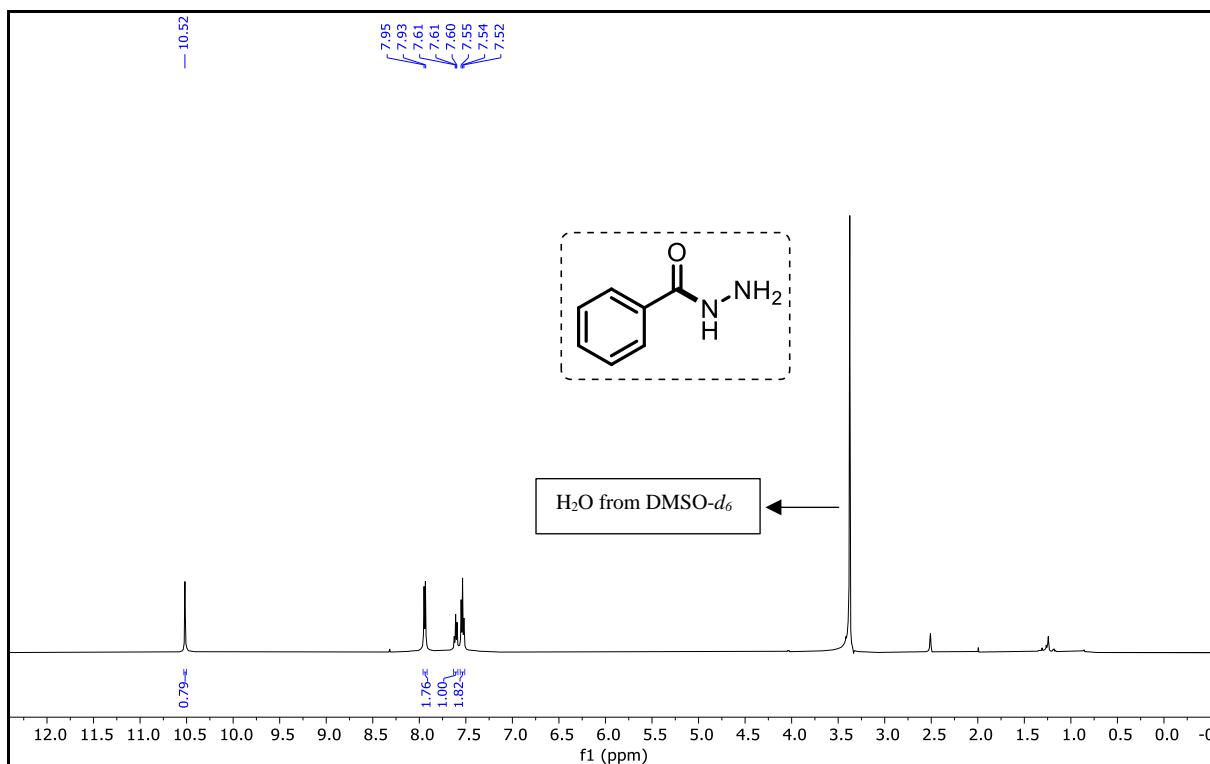


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

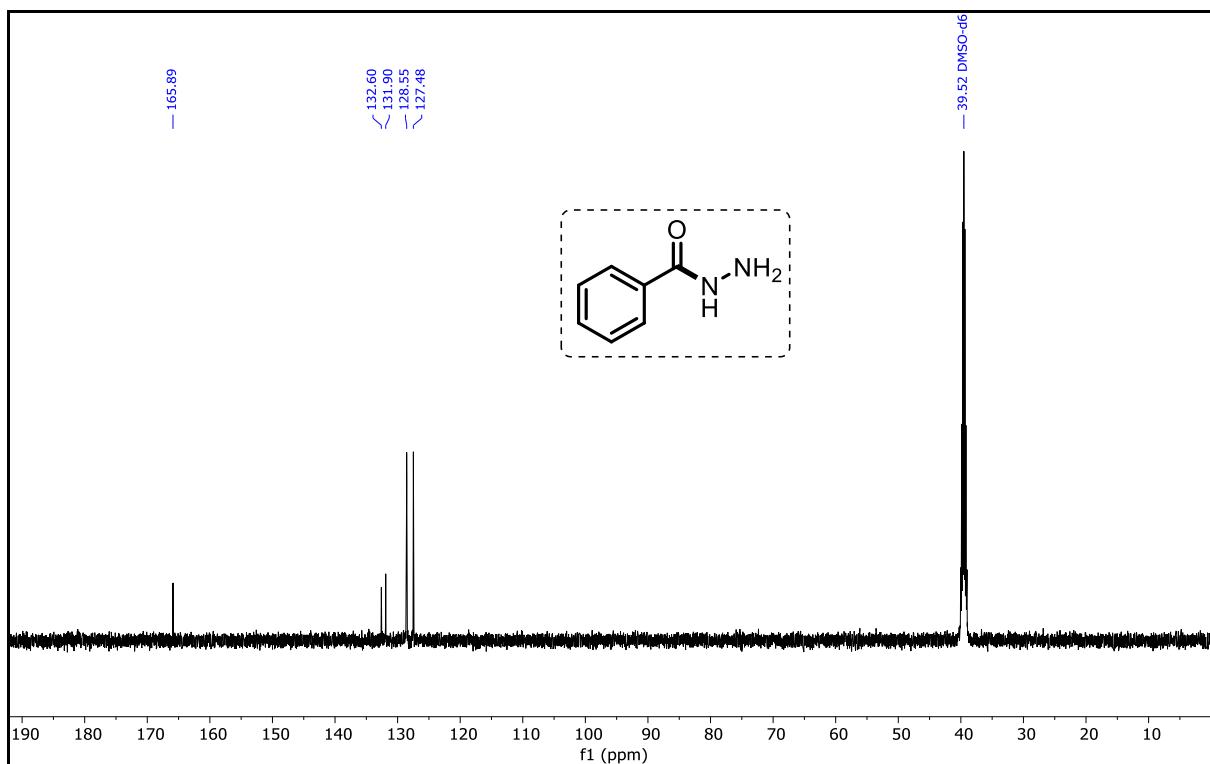


Benzohydrazide (3am):

^1H NMR (500 MHz, $\text{DMSO}-d_6$)

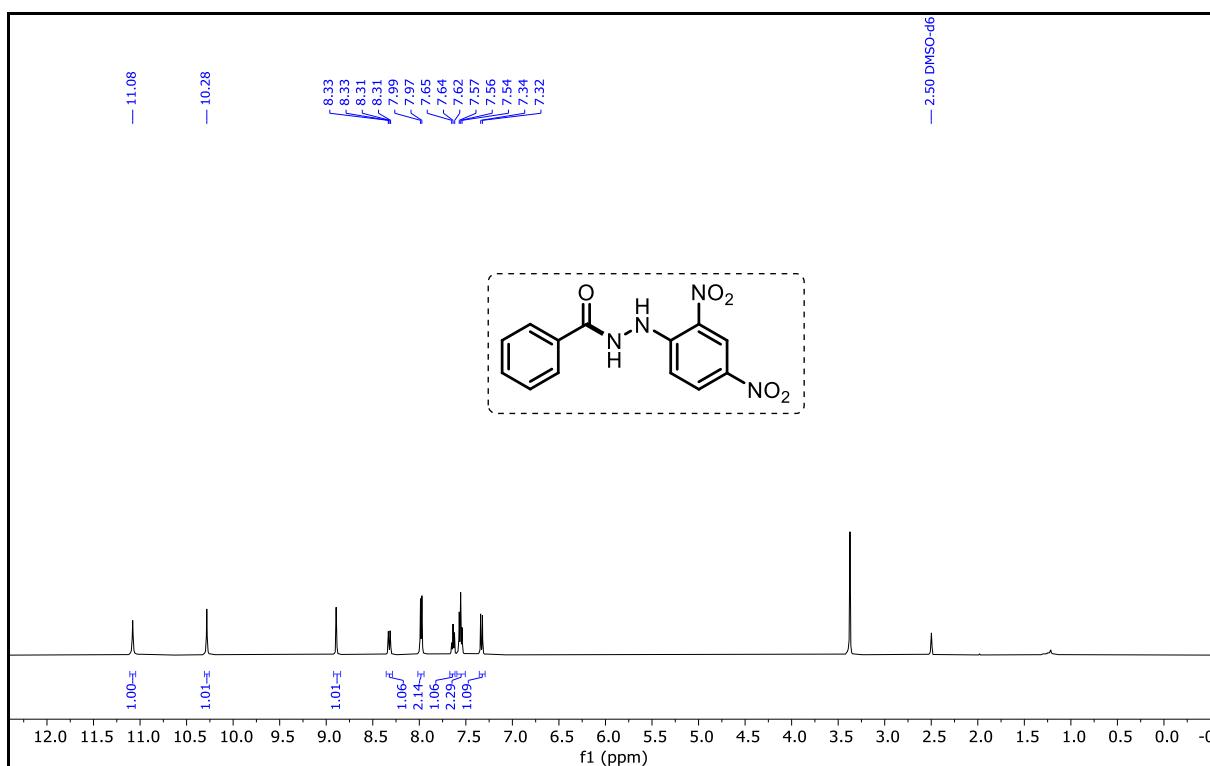


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, $\text{DMSO}-d_6$)

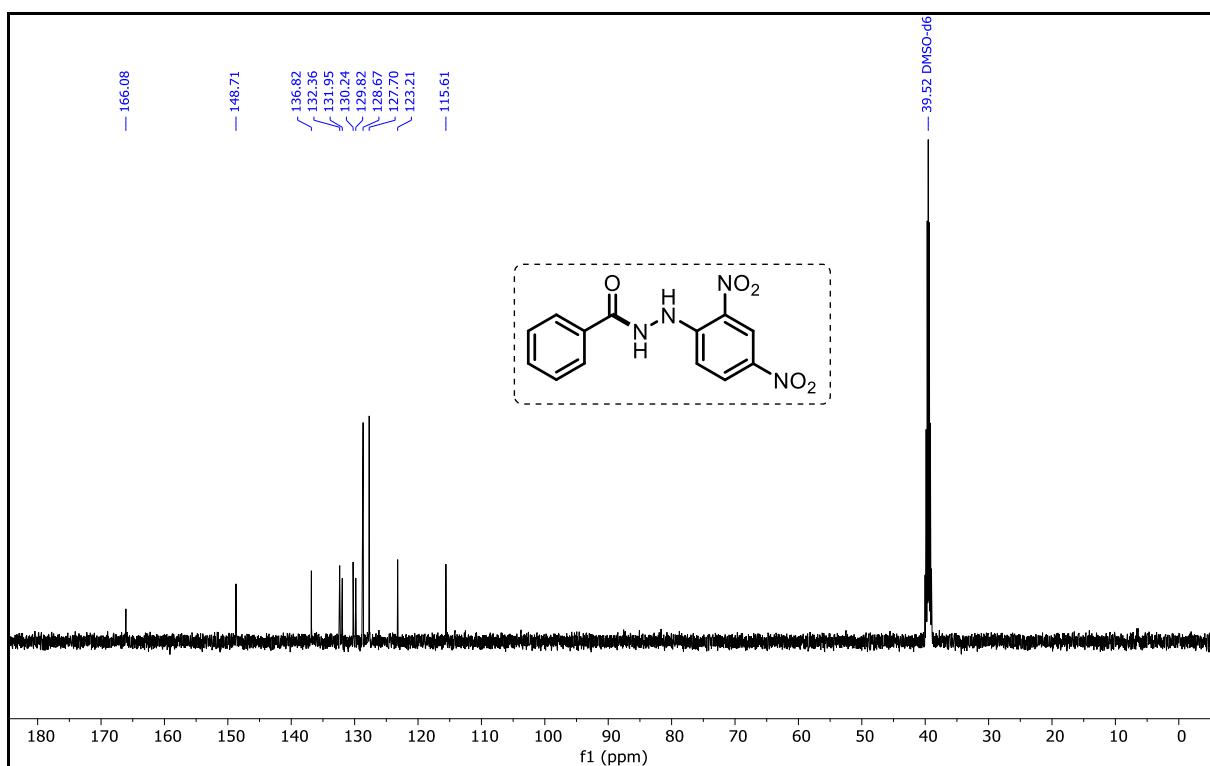


***N'*-(2,4-Dinitrophenyl) benzo hydrazide (3an):**

^1H NMR (500 MHz, DMSO- d_6)

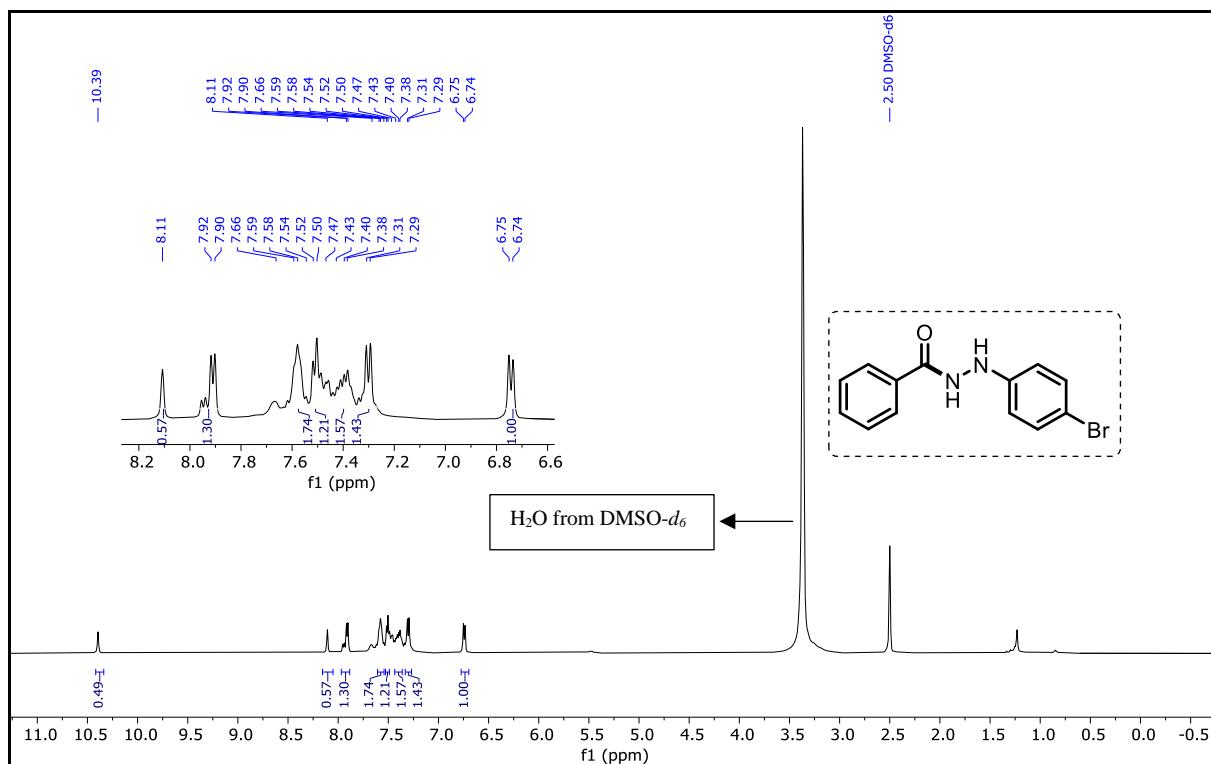


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

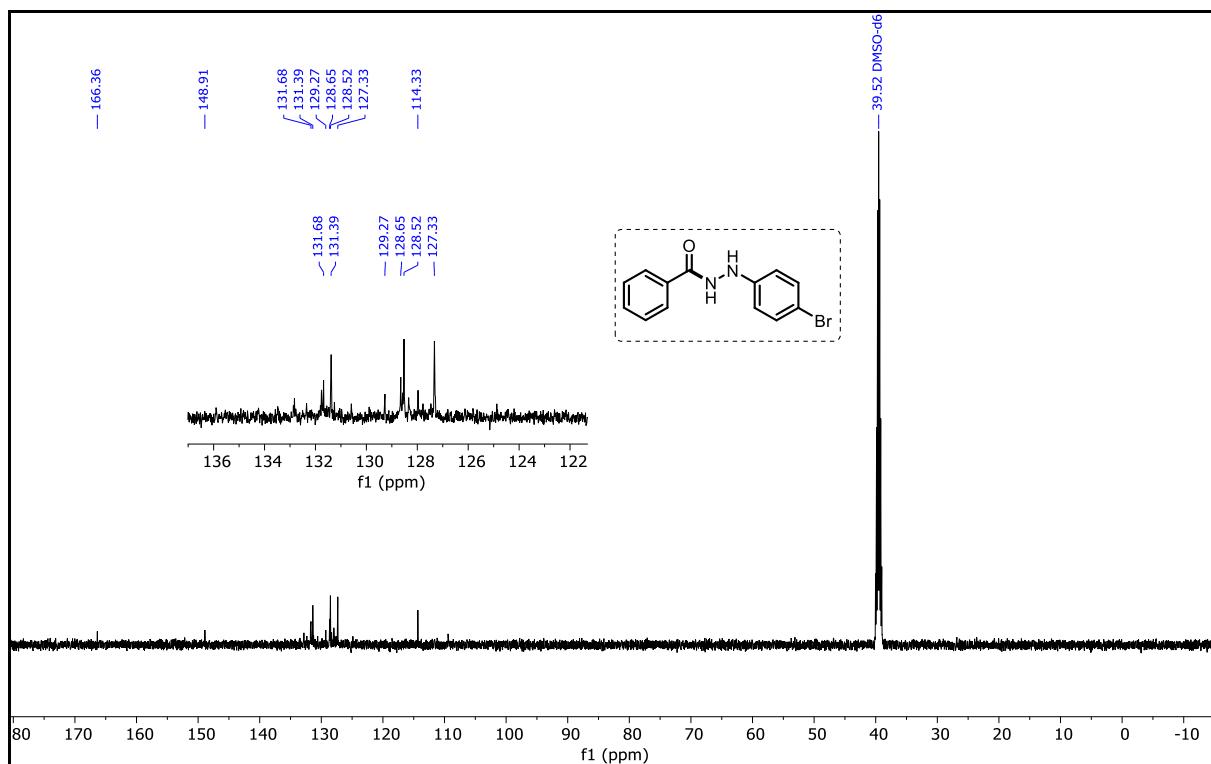


***N'*-(4-Bromophenyl) benzo hydrazide (3ao):**

^1H NMR (500 MHz, DMSO- d_6)

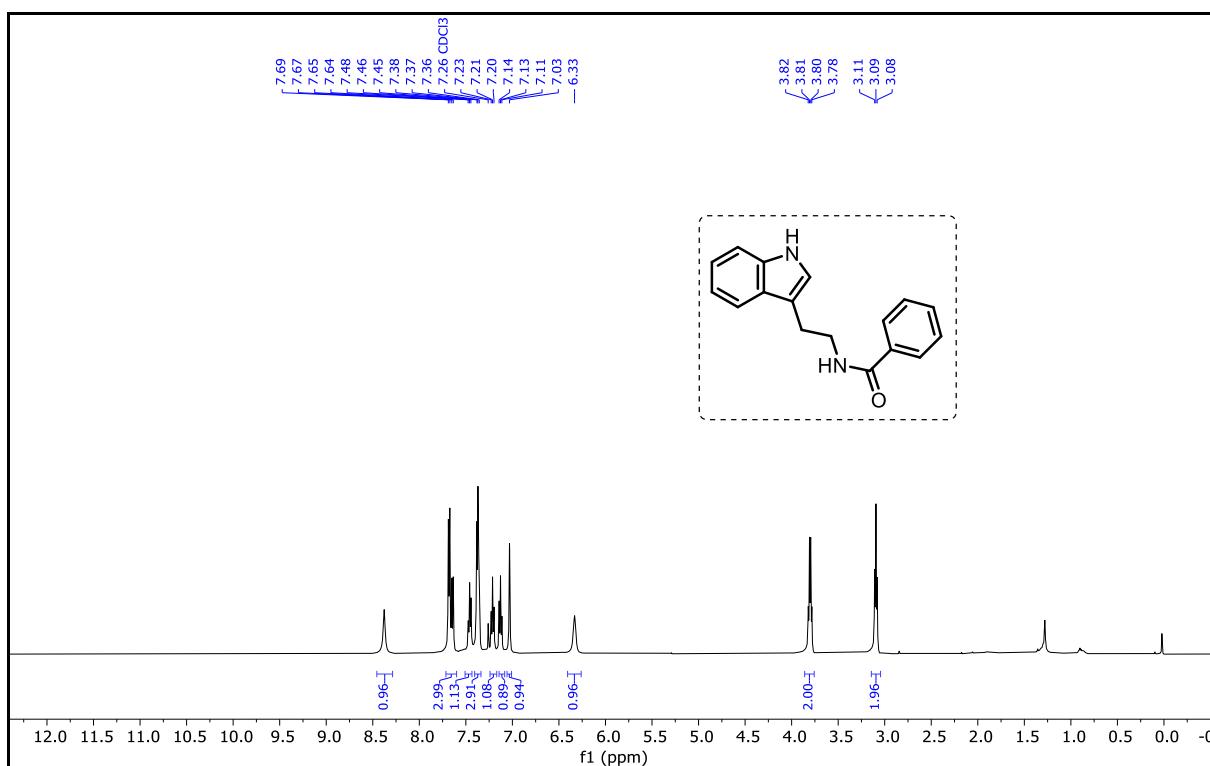


$^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, DMSO- d_6)

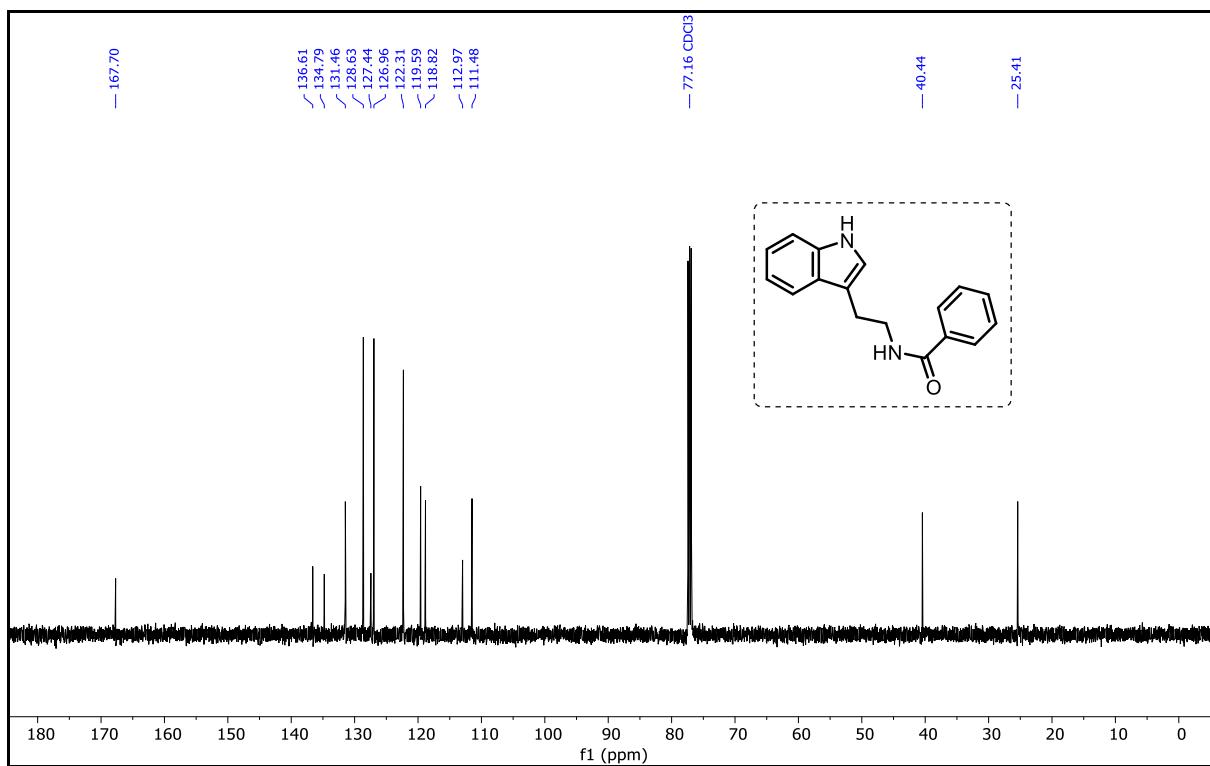


***N*-(2-(1*H*-Indol-3-yl) ethyl) benzamide (**3ap**):**

^1H NMR (500 MHz, CDCl_3)

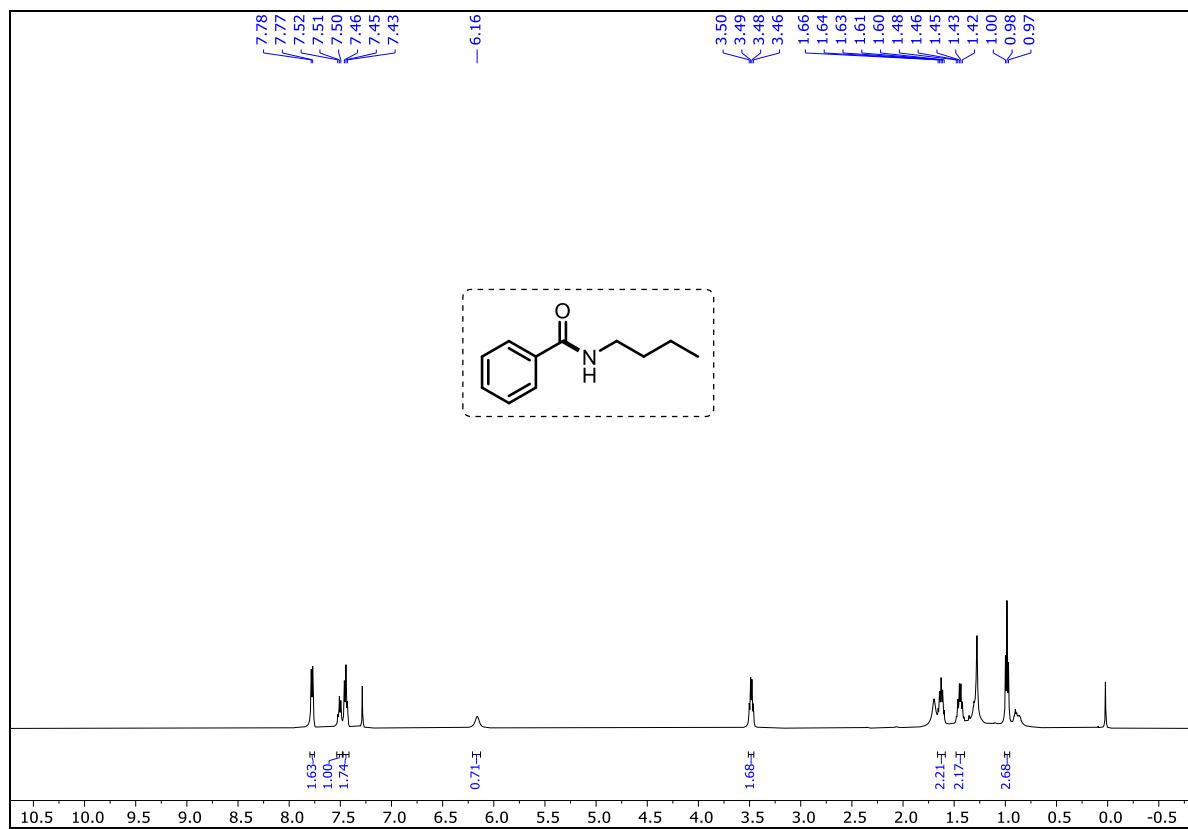


$^{13}\text{C}\{\text{H}\}$ NMR (126 MHz, CDCl_3)

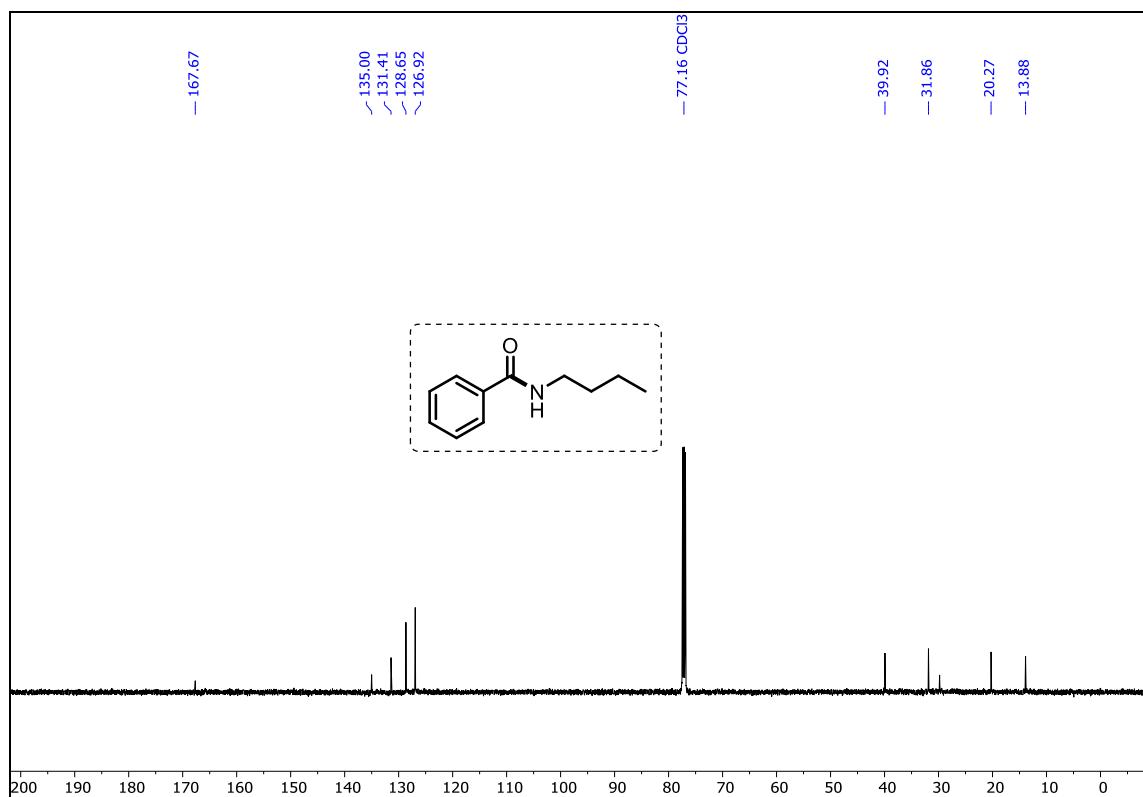


N-Butyl benzamide (3aq):

^1H NMR (500 MHz, CDCl_3)

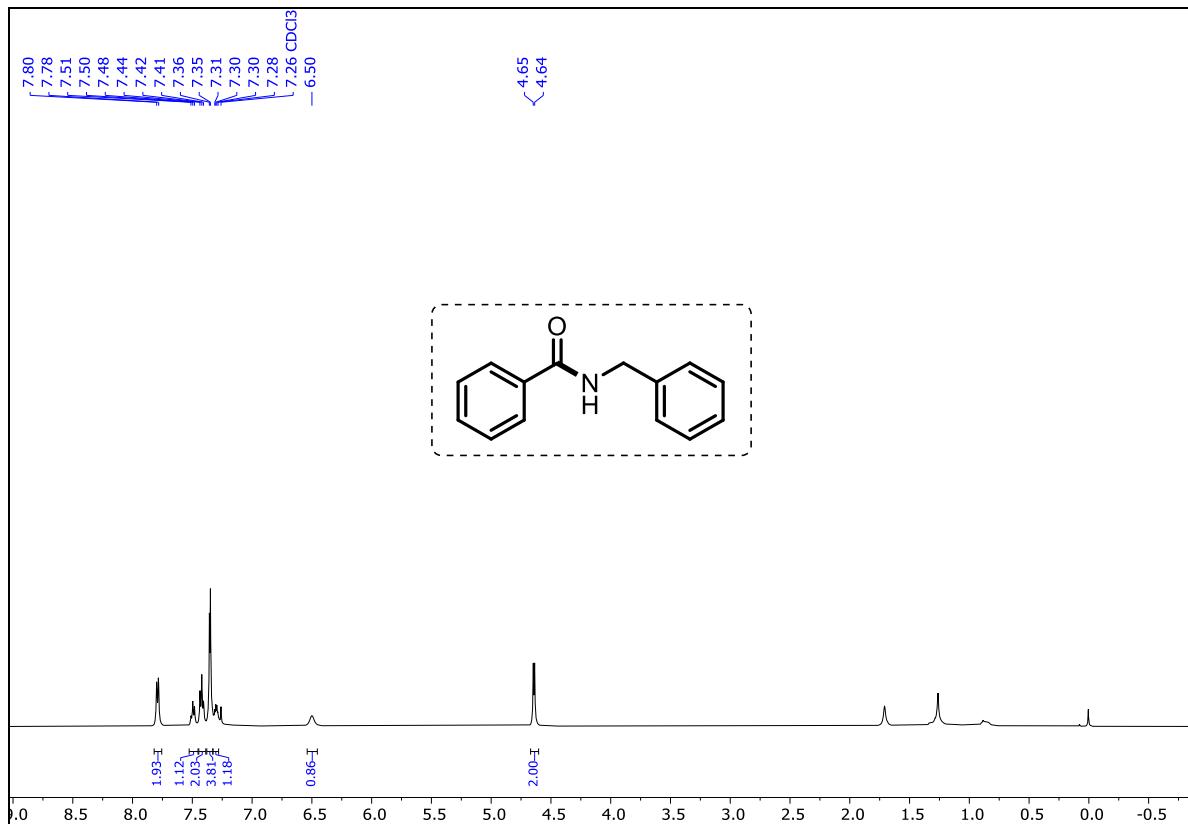


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CDCl_3)

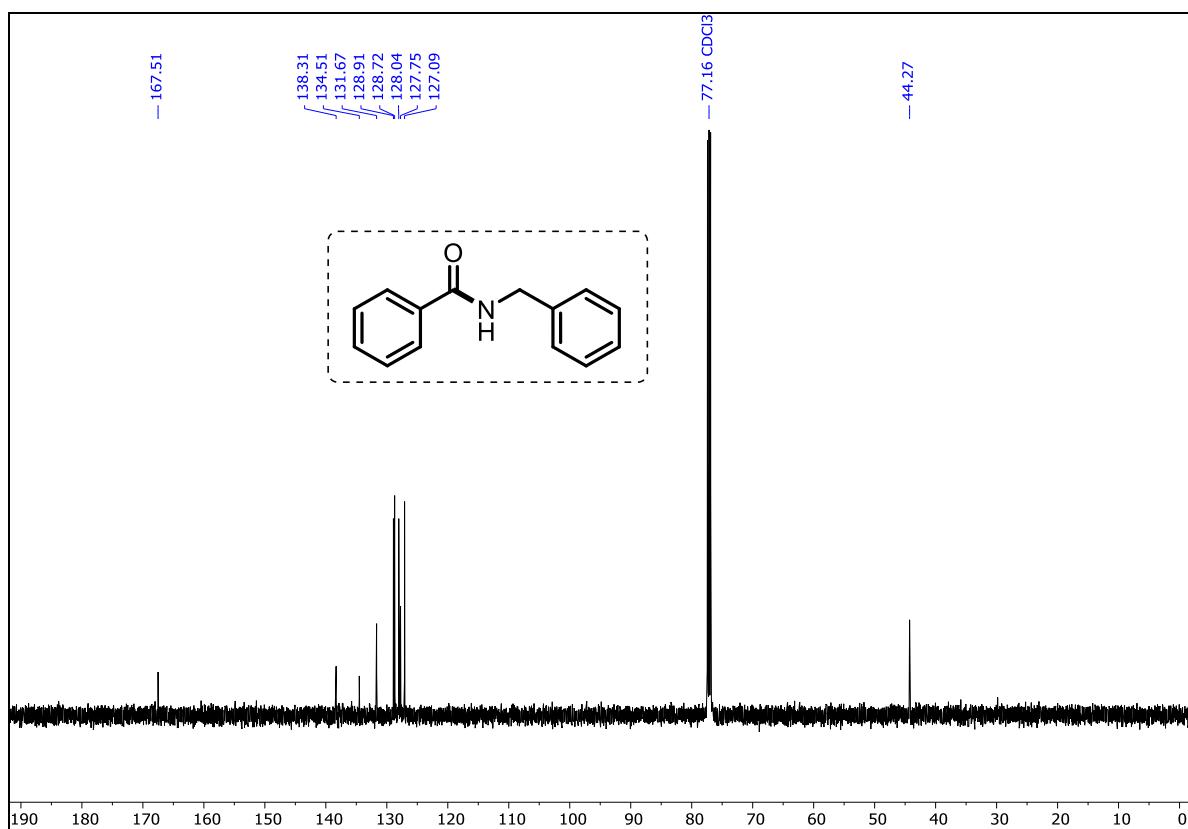


N-Benzyl benzamide (3ar):

^1H NMR (500 MHz, CDCl_3)

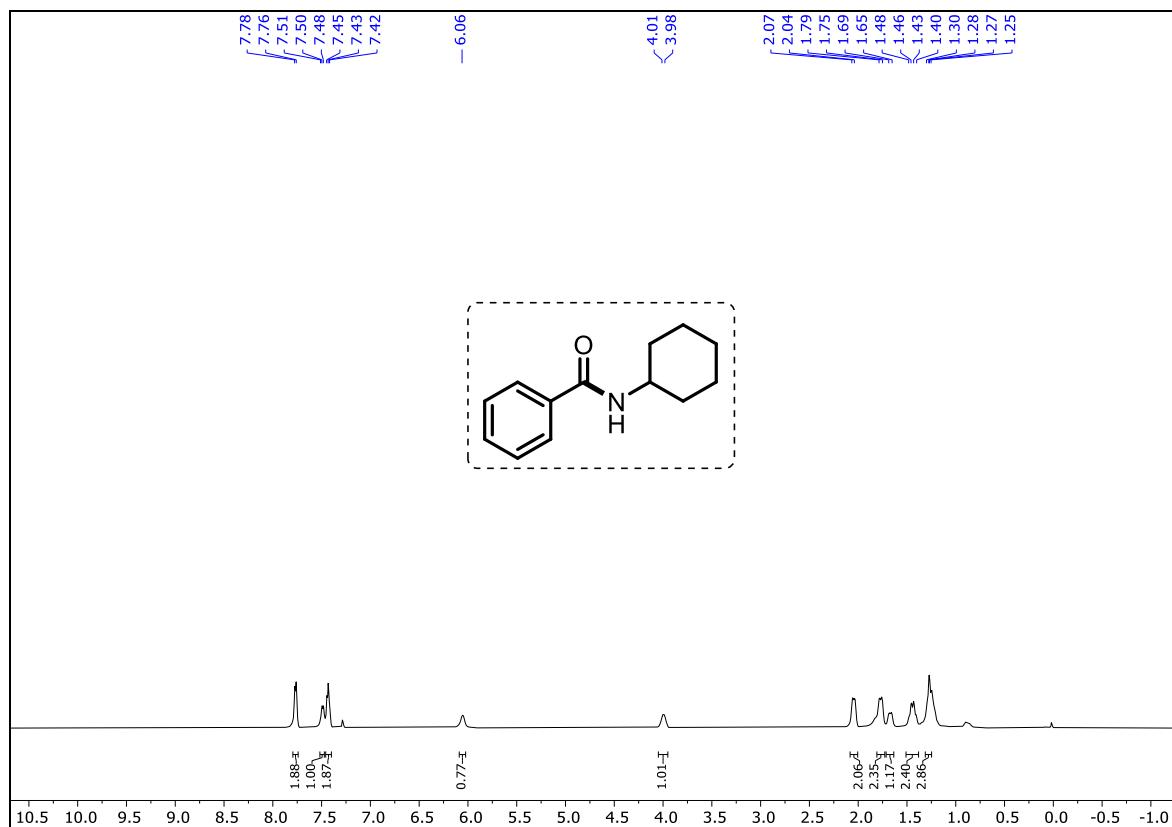


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CDCl_3)

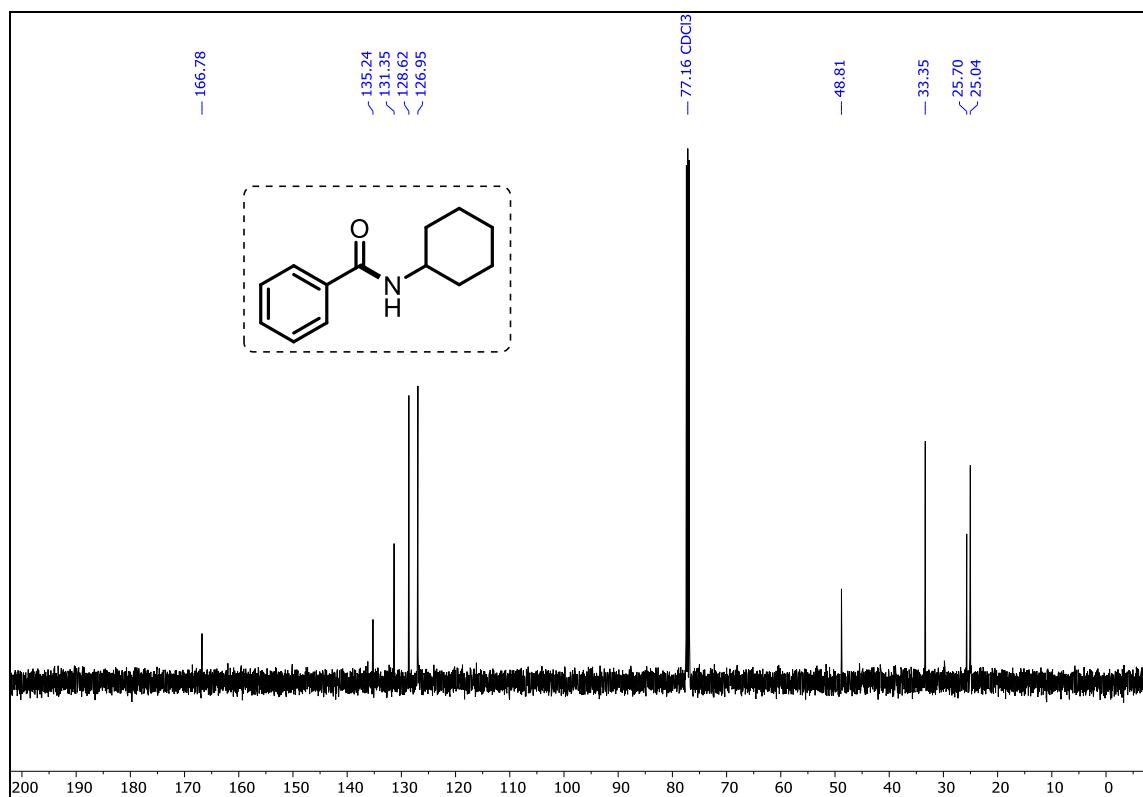


N-Cyclohexyl benzamide (3as):

^1H NMR (500 MHz, CDCl_3)

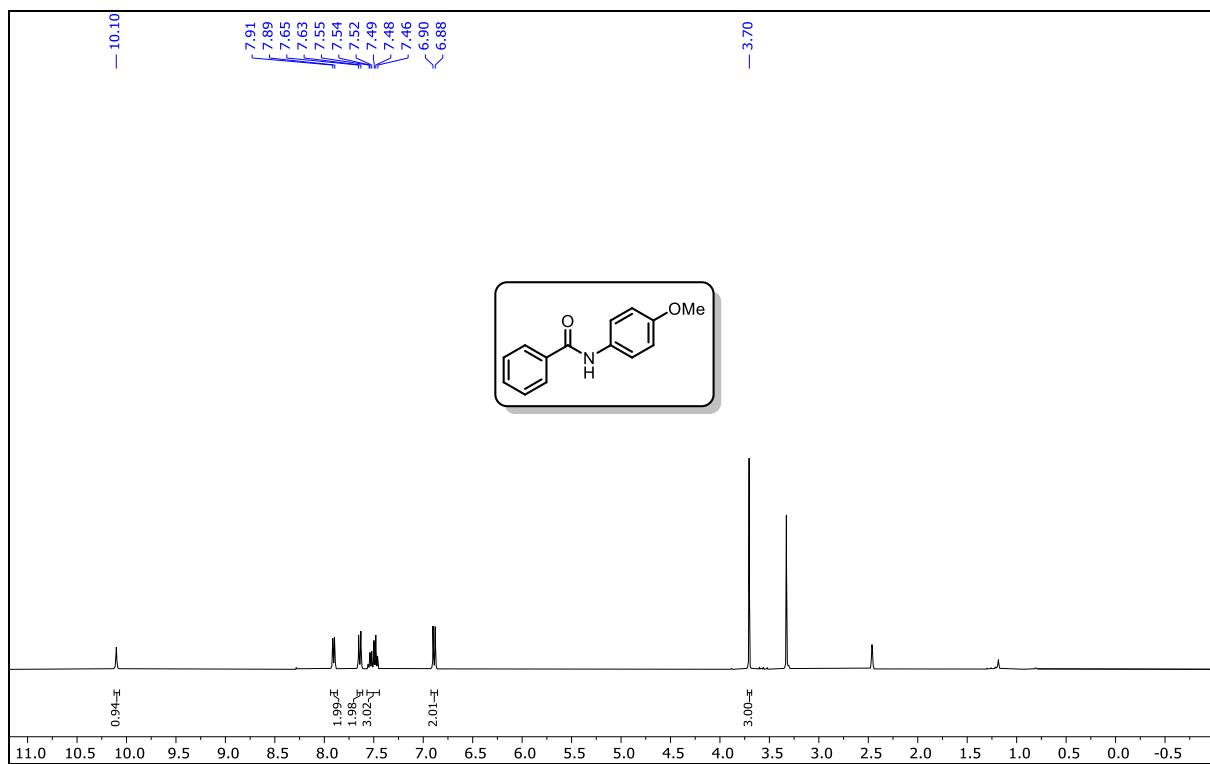


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CDCl_3)

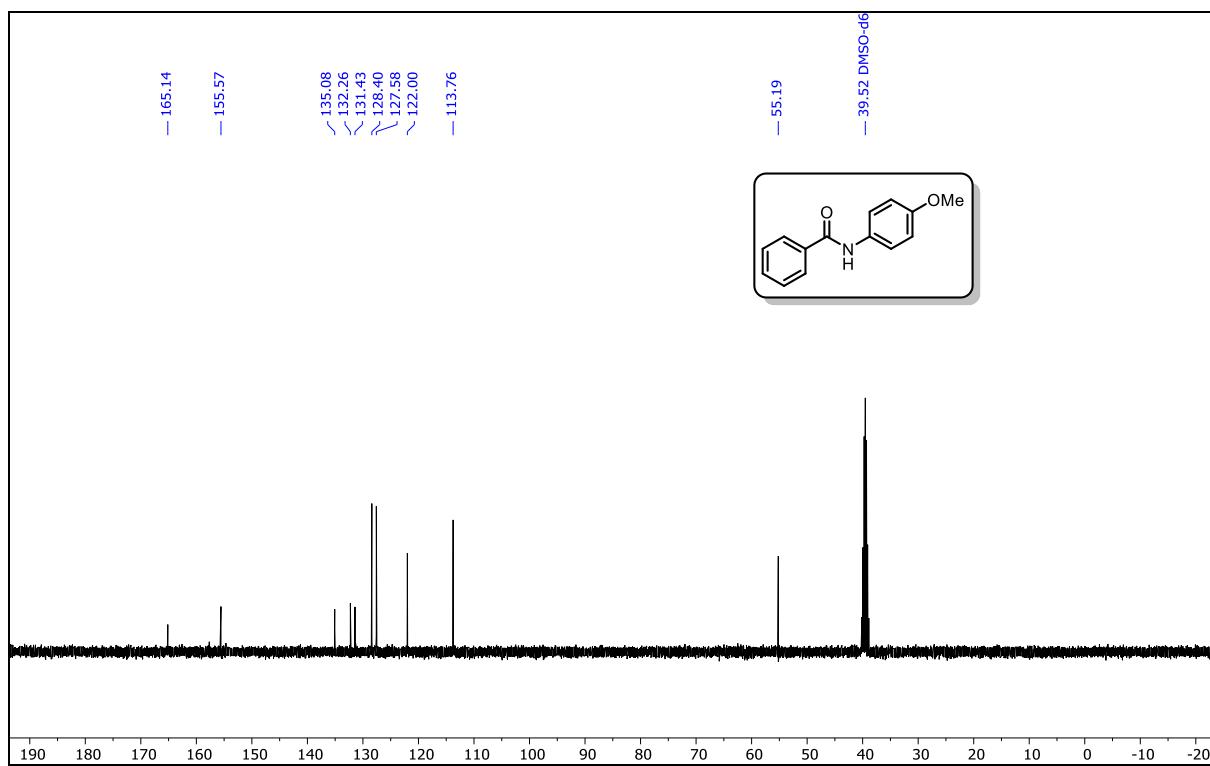


N-(4-Methoxyphenyl) benzamide (3au):

^1H NMR (400 MHz, DMSO- d_6)

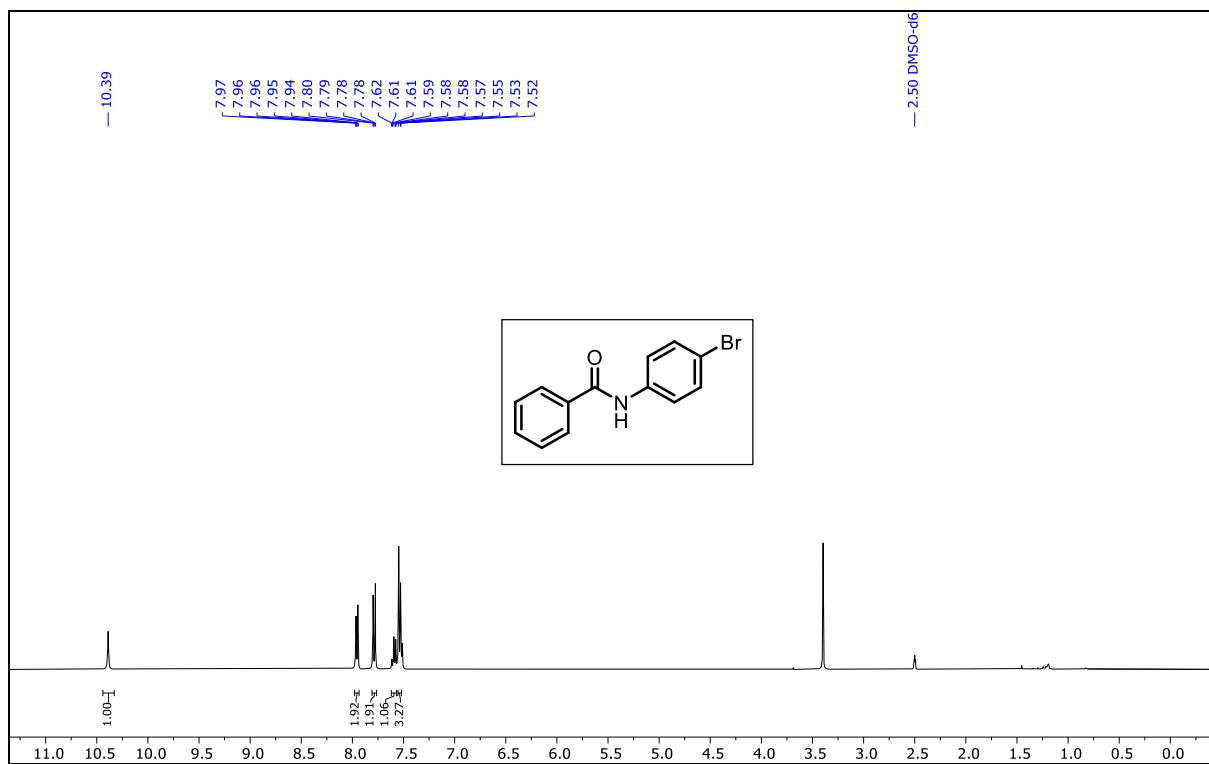


$^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, DMSO- d_6)

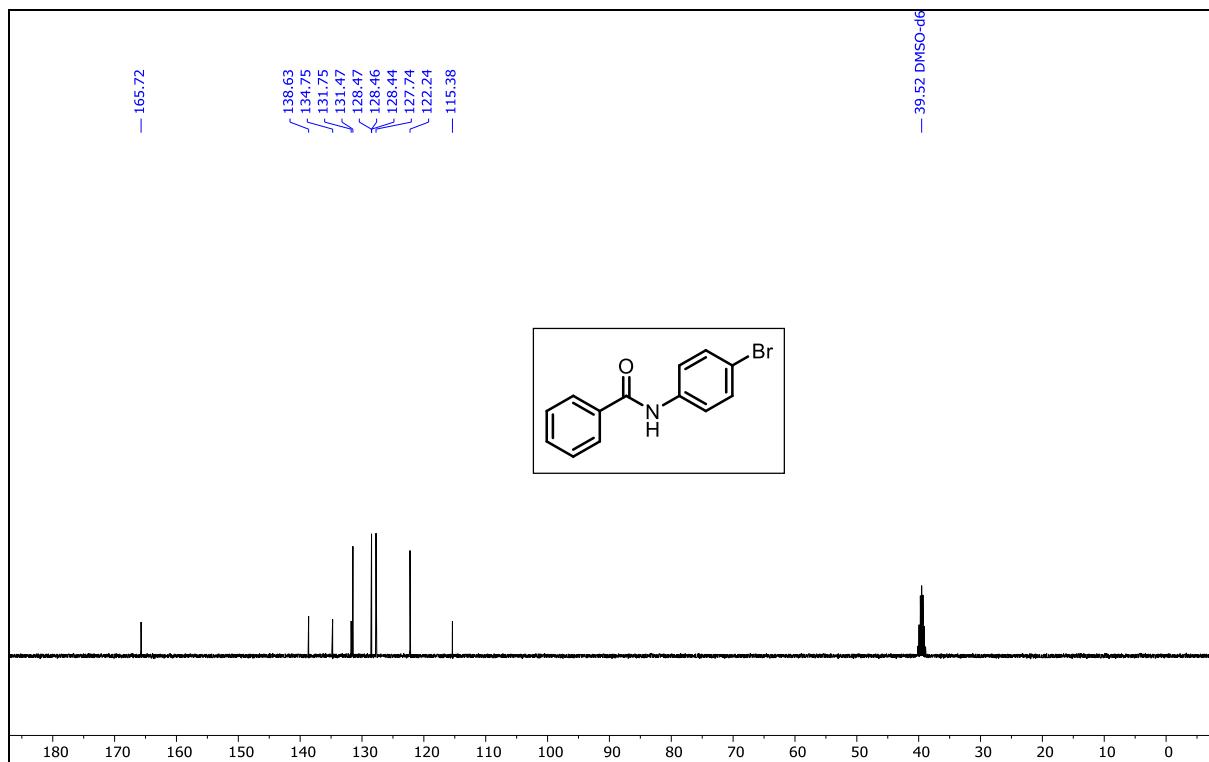


N-(4-Bromophenyl) benzamide (3av):

¹H NMR (400 MHz, DMSO-*d*₆)

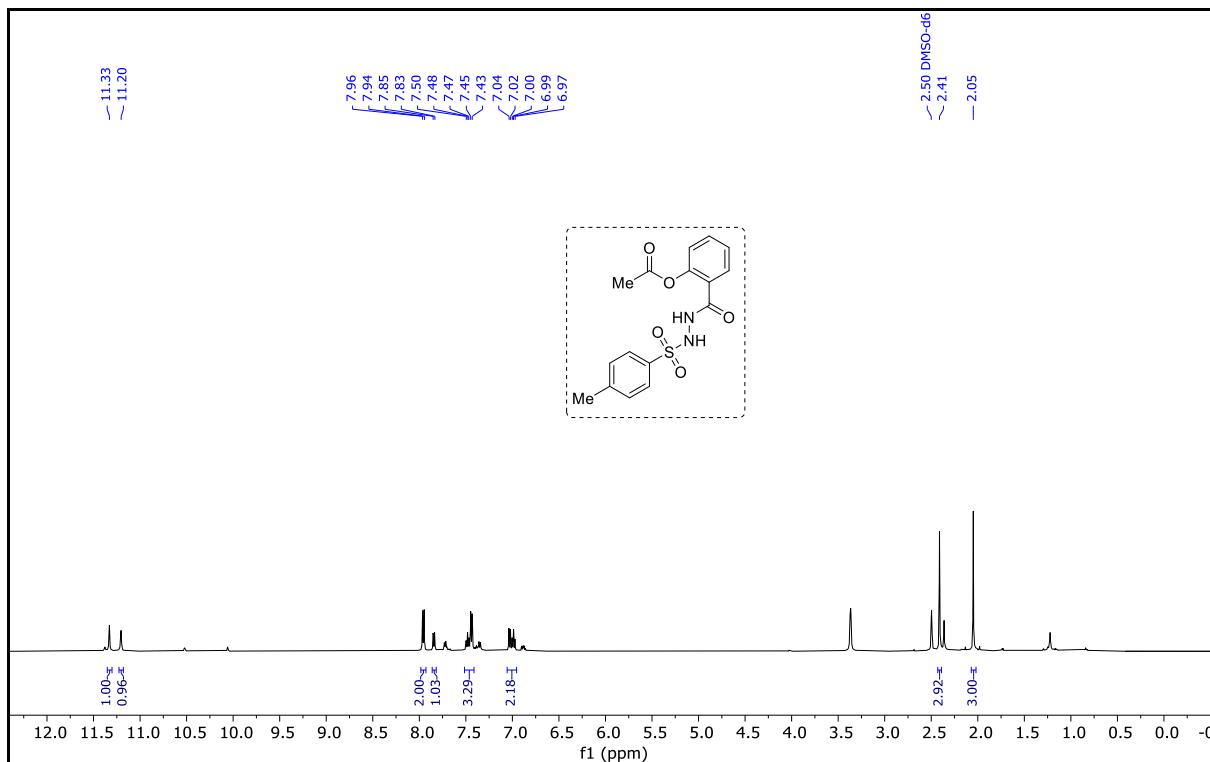


¹³C{¹H} NMR (101 MHz, DMSO-*d*₆)

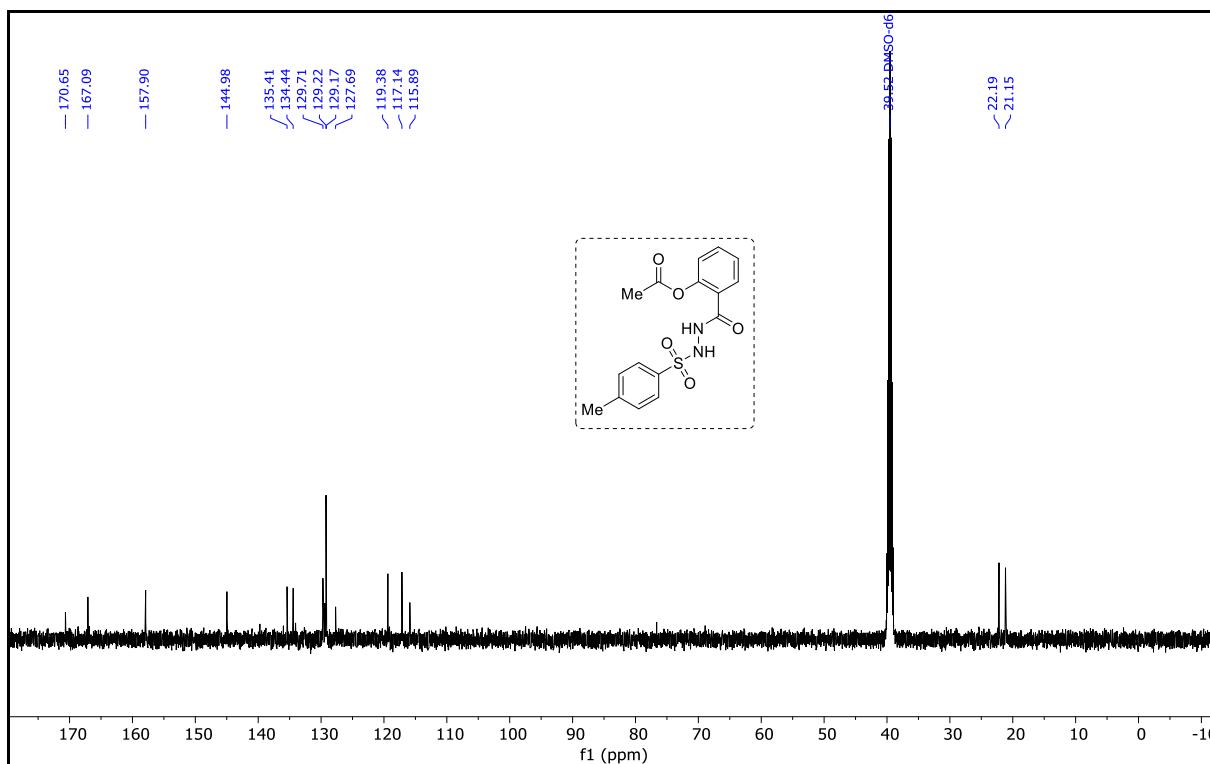


2-(2-Tosylhydrazine-1-carbonyl) phenyl acetate (4a**):**

^1H NMR (500 MHz, DMSO- d_6)

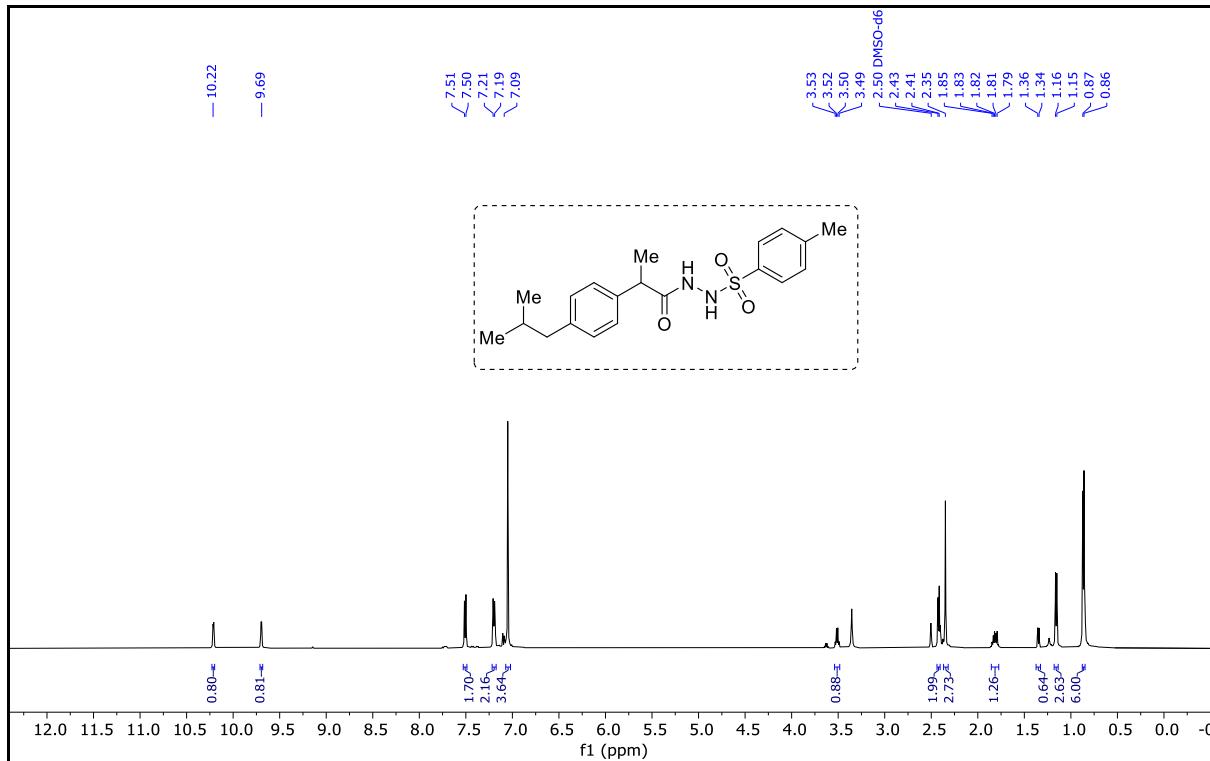


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

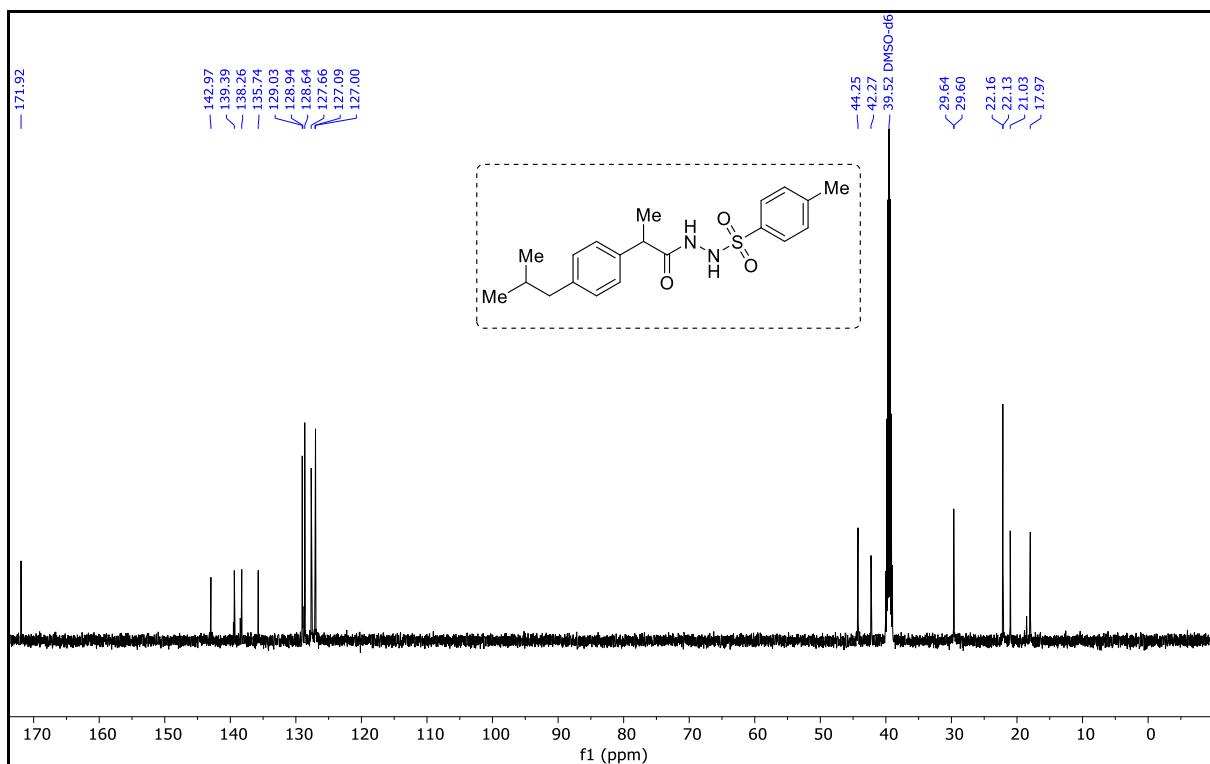


N'-(2-(4-Isobutylphenyl) propanoyl)-4-methylbenzenesulfonohydrazide (4b):

¹H NMR (500 MHz, DMSO-d₆)

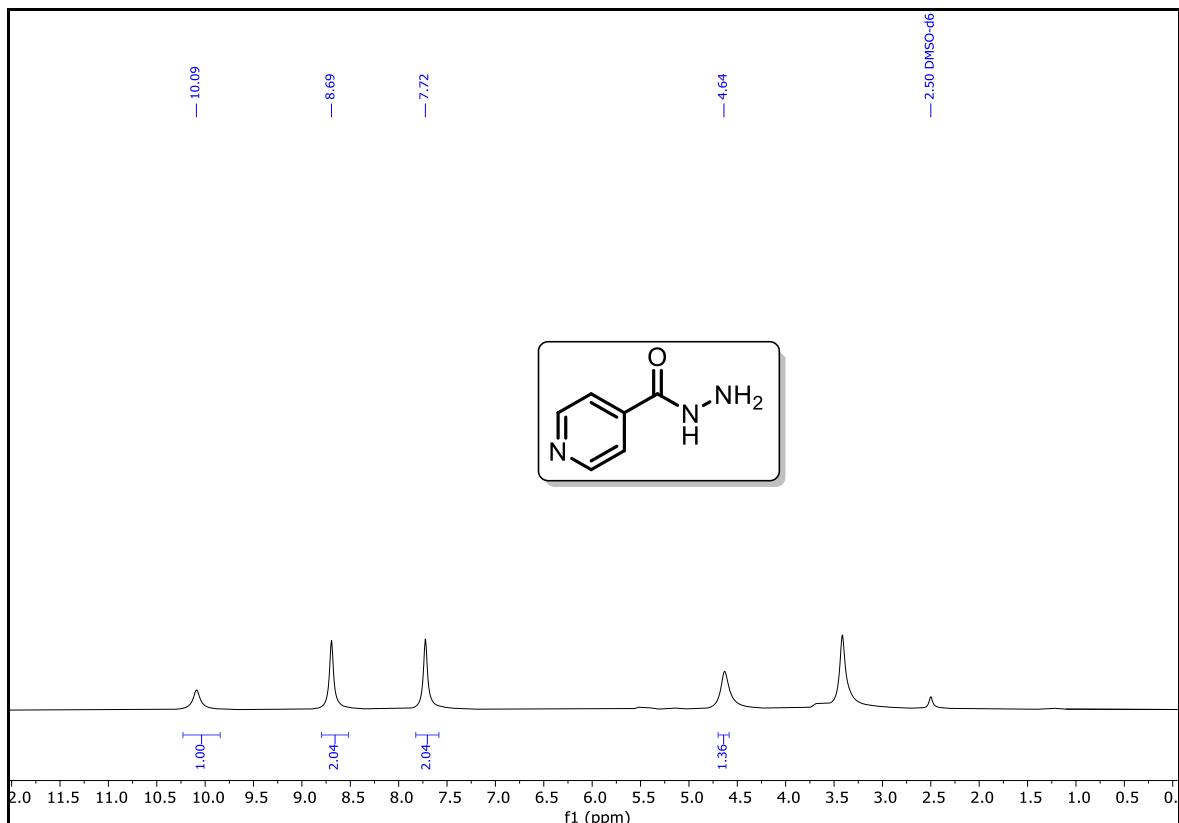


¹³C{¹H} NMR (126 MHz, DMSO-*d*₆)

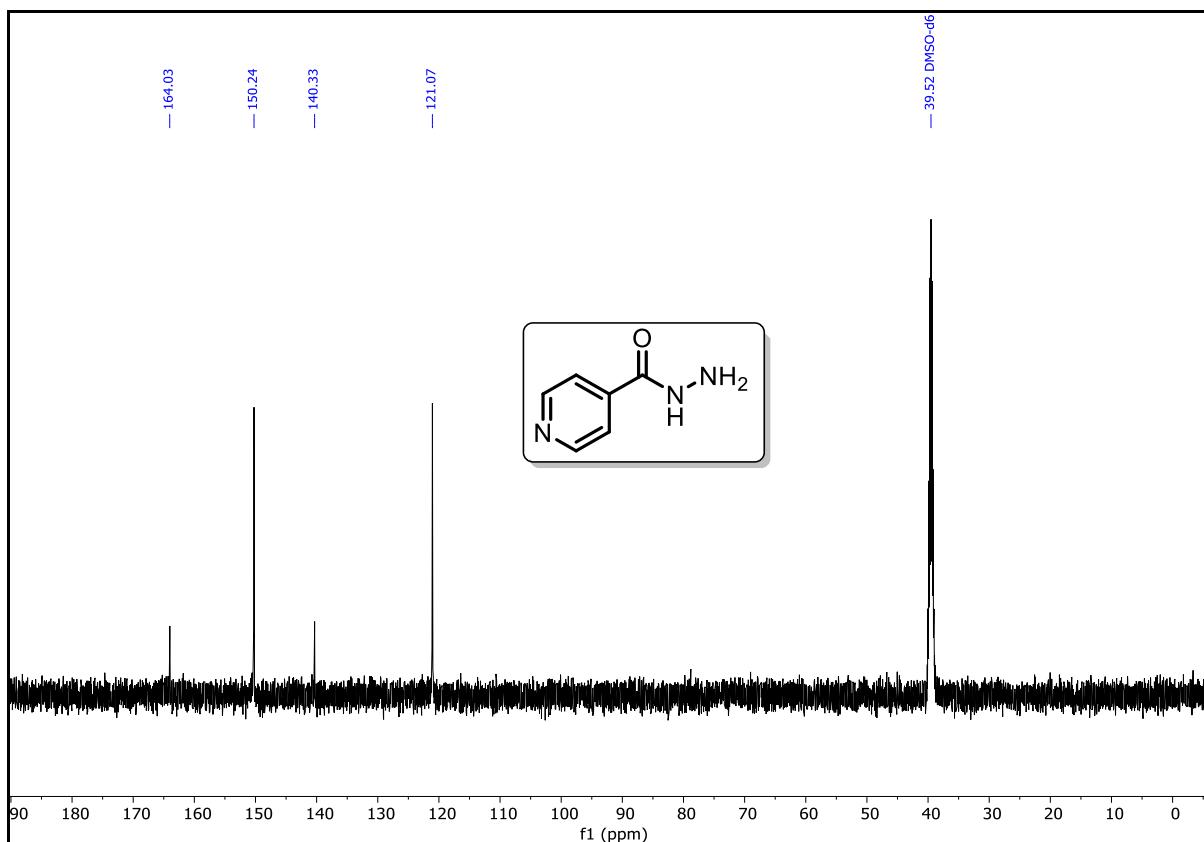


Isoniazide (5):

^1H NMR (500 MHz, $\text{DMSO}-d_6$)

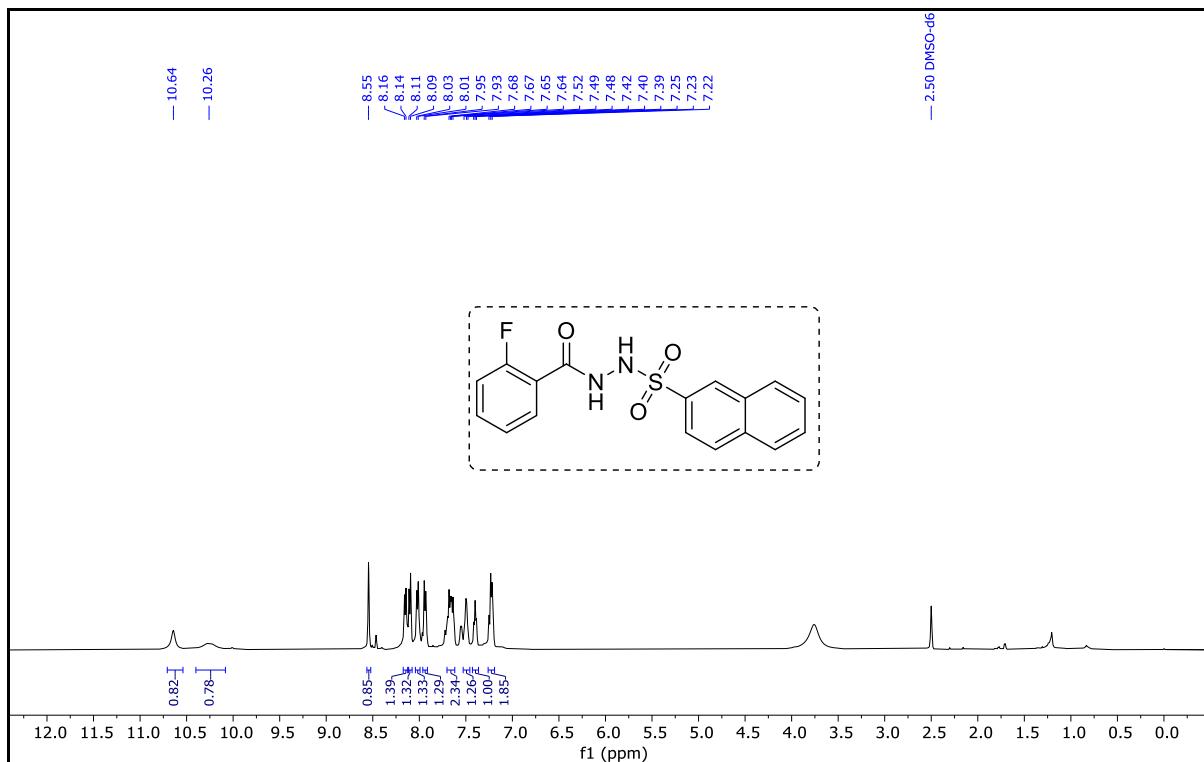


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, $\text{DMSO}-d_6$)

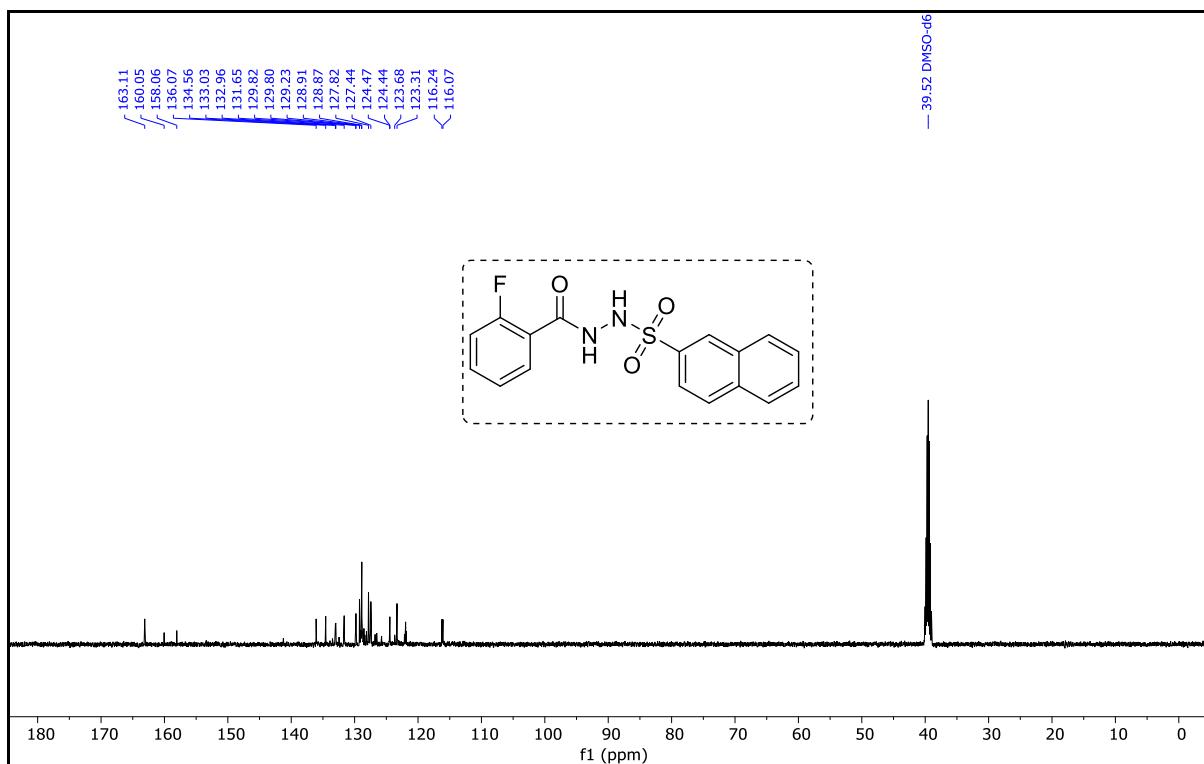


***N'*-(2-Fluorobenzoyl) naphthalene-2-sulfonohydrazide (6):**

^1H NMR (500 MHz, DMSO- d_6)

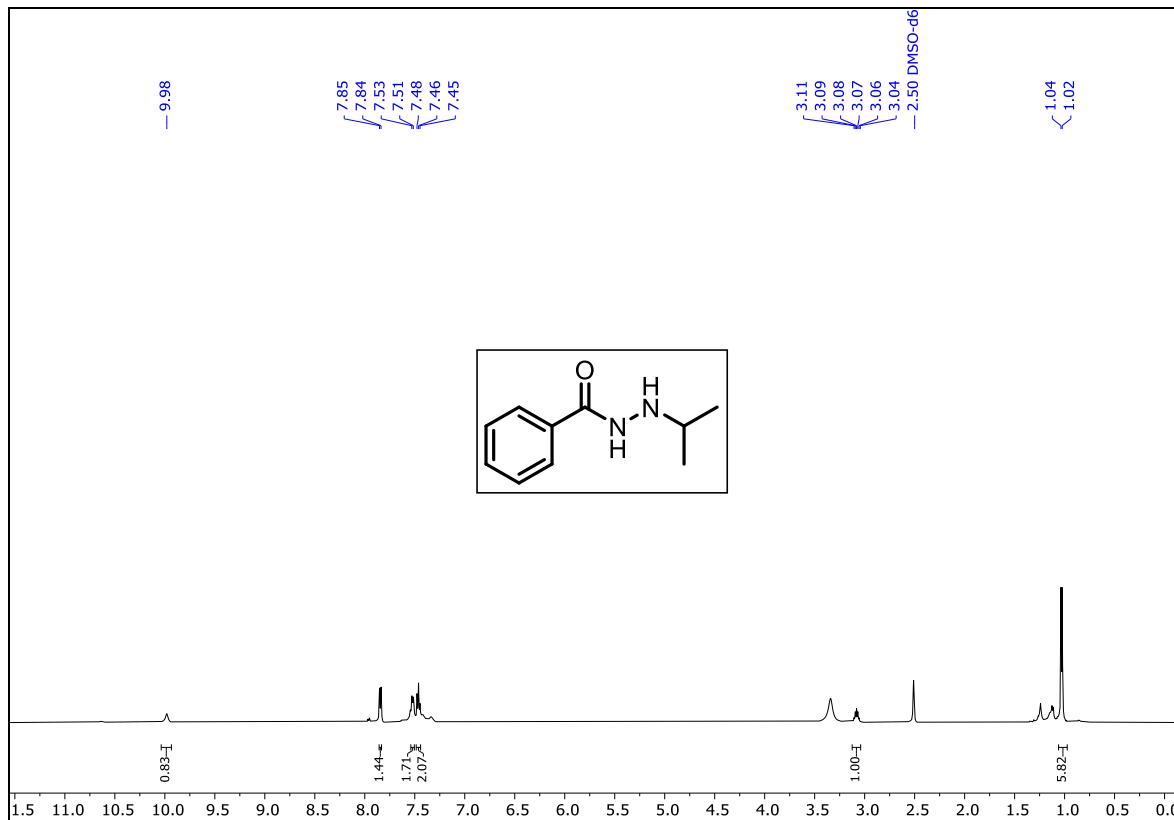


$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)

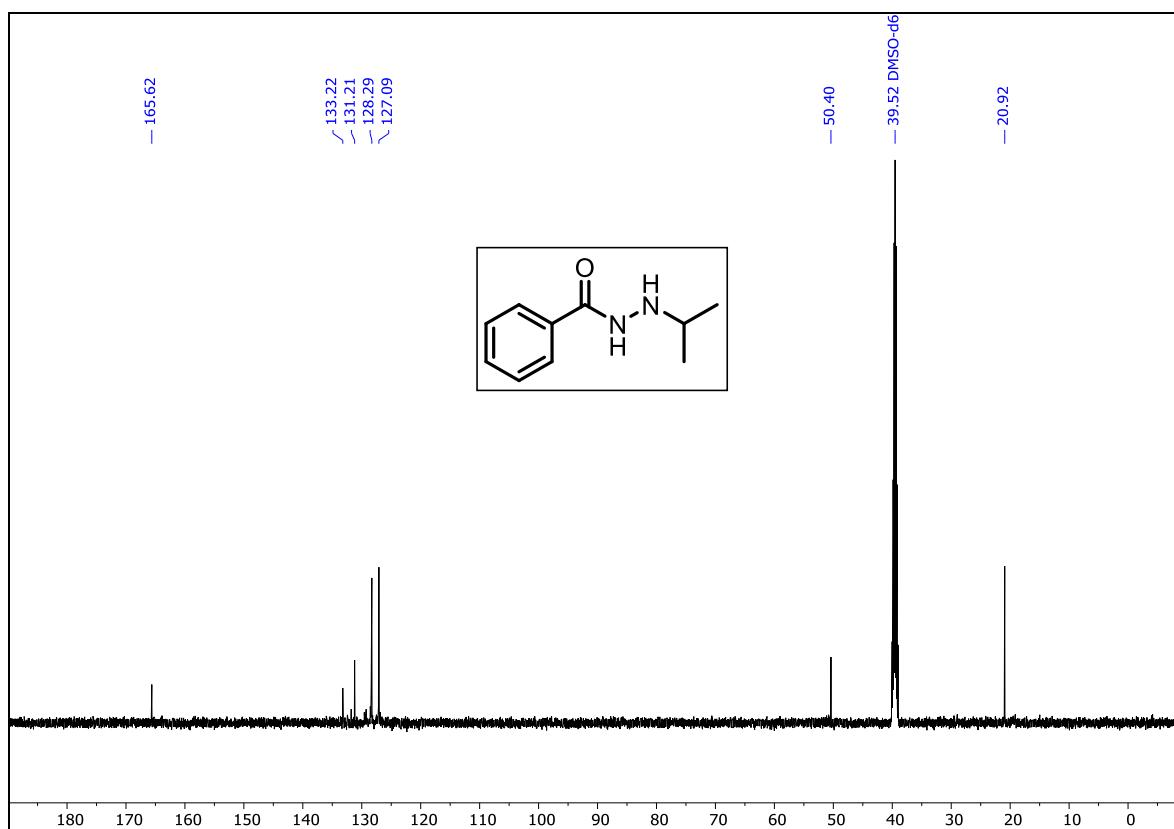


***N'*-Isopropylbenzohydrazide (7):**

^1H NMR (500 MHz, DMSO- d_6)



$^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, DMSO- d_6)



8. References

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