

Supporting information

Visible-light active benzothiadiazole-based MOFs as efficient ROS generator for the synthesis of benzimidazoles and benzothiazoles

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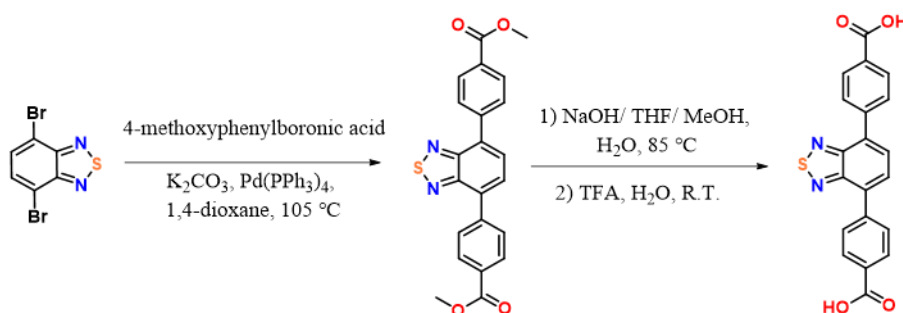
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1. Materials and Methods

Unless otherwise noted, all materials were used as received from commercial sources without further purification. The products phase purity was examined by powder XRD in the angular range of $2\theta = 5\text{-}50^\circ$, using a Bruker D8 ADVANCE X-ray powder diffractometer (Cu $K\alpha$, 1.5418 Å). Thermogravimetric analysis (TGA) curves were obtained on Mettler-Toledo thermal analyzer from 35°C to 800 °C with a heating rate of 10 °C min⁻¹ under a nitrogen gas atmosphere (20 mL·min⁻¹). UV-visible diffuse reflectance spectra were conducted with BaSO₄ pellets on a UV-3600PLUS spectrometer. Ultraviolet-visible spectra were recorded on a Hitachi-2910 spectrophotometer equipped with a xenon discharge lamp. The Fluorescent spectra were recorded on Hitach F-4600 fluorescence spectrometer equipped with a xenon discharge lamp. Nitrogen gas adsorption/desorption measurements were performed on Micromeritics ASAP 2020 Plus adsorption instrument. The electrochemical measurements were performed in a conventional three-electrode cell on a CHI-760E electrochemical workstation (Shanghai Chenhua Instrument Co., Ltd, China). Photocatalytic measurements were conducted on a WP-TEC-1020HSL of WATTCAS photoreactor instrument. Electron paramagnetic resonance (EPR) signals were recorded on a Bruker E500-9.5/12 at room temperature under visible-light irradiation using a blue LED (400-470 nm). ¹H NMR and ¹³C NMR experiments were performed with a JEOL ECZ400S spectrometer. Mass spectra were carried out on a Bruker microTOF-Q II Spectroscopy.

2. Synthesis and Characterization of Ligand and MOF

2.1 Synthesis of H₂BTDB ligand



The synthesis of Me₂BTDB: 4,7-dibromo-2,1,3-benzothiadiazole (1.0 g, 3.4 mmol), methyl 4-boronobenzoate (1.5 g, 8.5 mmol), potassium carbonate (2.6 g, 20.4 mmol) and Pd(PPh₃)₄ (78.6 mg, 0.068 mmol) were added into one 250 mL round-bottle flask containing 80 mL dioxane. The reaction solution was degassed four times. Then the mixture was heated to reflux at 110 °C for 72 h under argon atmosphere. After cooling down to room temperature, the mixture was extracted five

times with ethyl acetate, then the organic layers were combined and dried over anhydrous Na_2SO_4 and the solvent was removed in vacuo. The crude product was purified by silica gel column chromatography to give the target product (yield 85%). ^1H NMR (400 MHz, Chloroform-*d*) δ 8.22 (d, $J = 7.9$ Hz, 4H), 8.07 (d, $J = 7.7$ Hz, 4H), 7.87 (s, 2H), 3.98 (s, 6H).

The synthesis of H_2BTDB : 2.5 mmol compound Me_2BTDB was added to a solution containing 30 mL CH_3OH , 30 mL THF and 30 mL water with 1.0 g NaOH. The mixture was heated to reflux at 80 °C overnight. After cooling down to room temperature, the organic solvent was removed under reduced pressure and the resulted aqueous was filtered. Then the filtrate was neutralized using 2M HCl to obtain the precipitate, which was filtered to offer the final product H_2BTDB (yield 90%). ^1H NMR (400 MHz, DMSO-*d*₆) δ 13.11 (s, 2H), 8.17 (d, $J = 8.0$ Hz, 4H), 8.12 (d, $J = 8.3$ Hz, 4H), 8.09 (s, 2H).

2.2 Synthesis of UiO-68-BTDB

ZrCl_4 (11.2 mg, 0.048 mmol), H_2BTDB (0.07 mmol) and benzoic acid (222.6 mg, 1.82 mmol) were added into a 5 mL vial containing 3 mL DMF. After sonicated for 5 minutes, the vial was put into a preheated oven at 120 °C for 24 hours. After cooling down to room temperature, the precipitate were collected and solvent exchanged with DMF and acetone for several times to afford the as synthesized UiO-68-BTDB.

For ^1H NMR spectroscopy, the activated samples (around 20 mg) were digested by 10 M HCl aqueous solution and dried in a 100 °C oven. The solid was dissolved in about 0.5 mL DMSO-*d*₆. ^1H NMR spectra for UiO-68-BTDB is presented below.

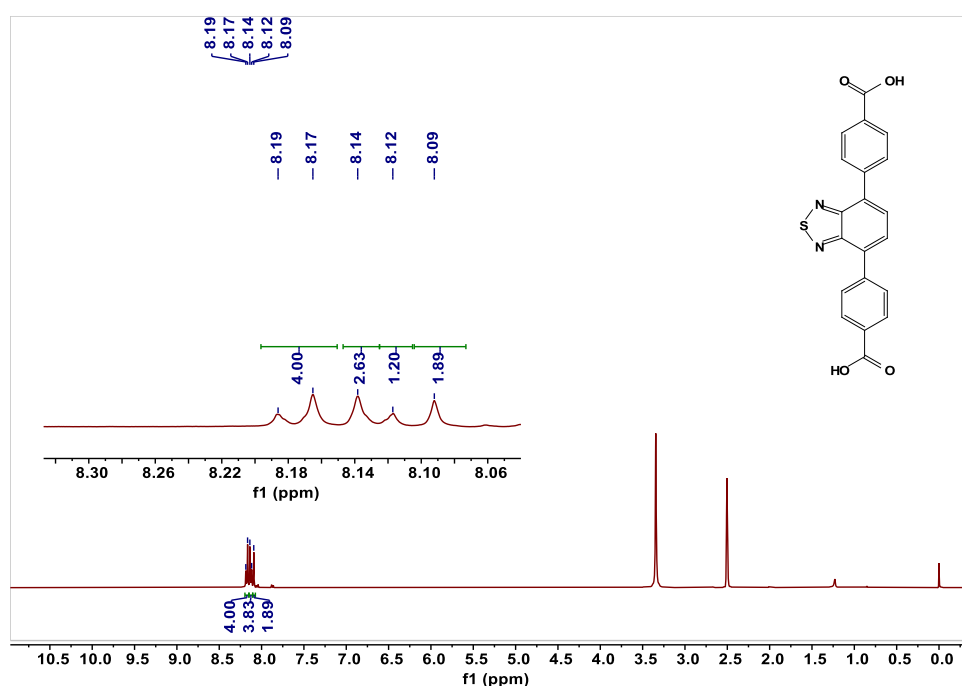


Figure S1. ^1H NMR spectroscopy of digested UiO-68-BTDB in DMSO-*d*₆.

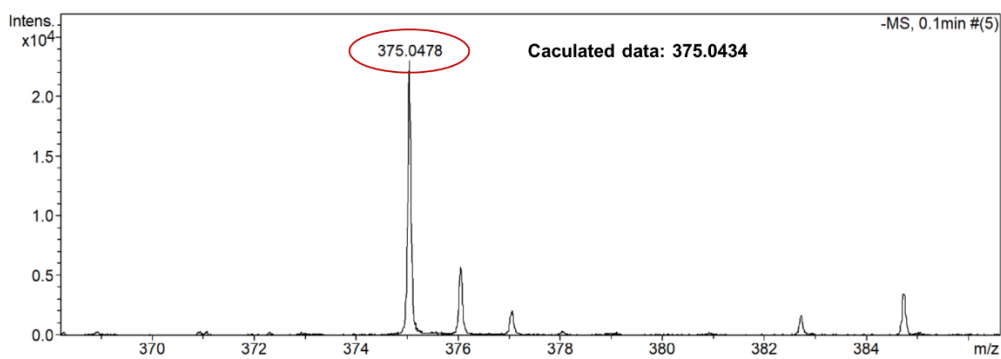


Figure S2. HRMS of digested UiO-68-BTDB.

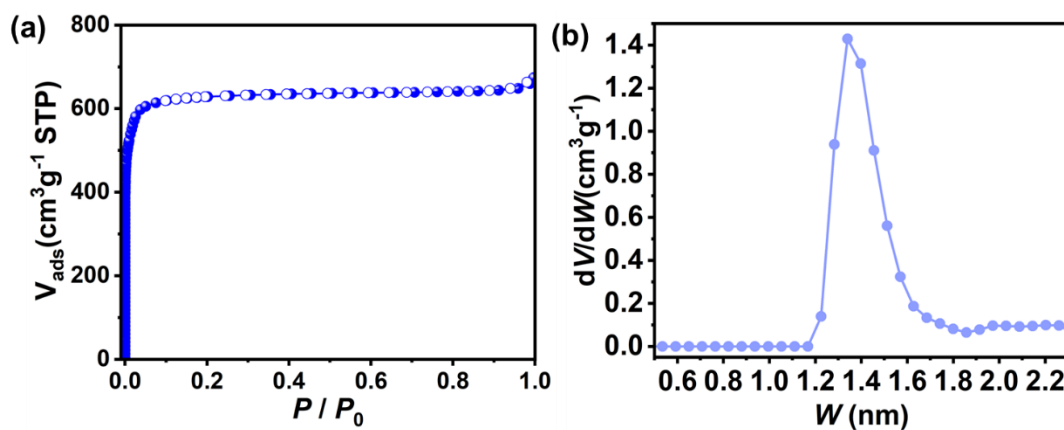


Figure S3. (a) N₂ adsorption/desorption isotherms at 77 K for UiO-68-BTDB. (b) Pore size distributions of UiO-68-BTDB.

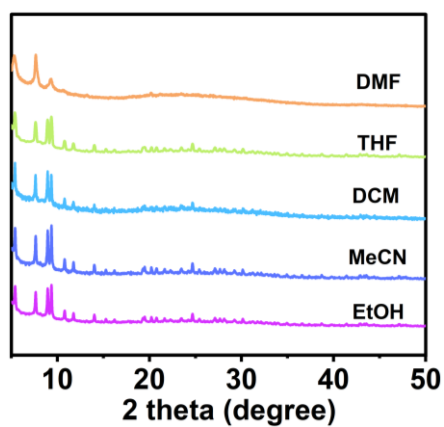


Figure S4. PXRD patterns of UiO-68-BTDB soaked in different organic solvents.

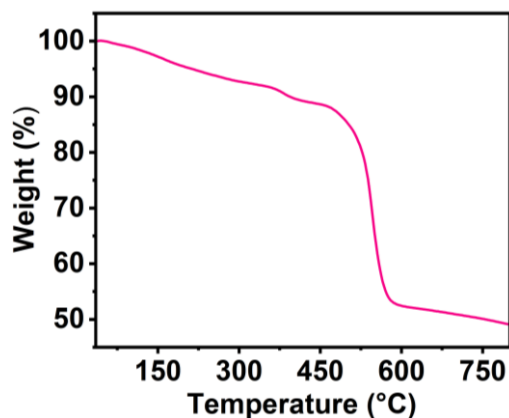


Figure S5. Thermogravimetric analysis curves of UiO-68-BTDB.

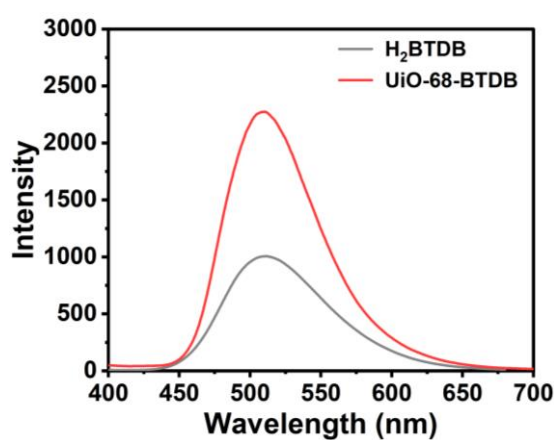


Figure S6. The emission spectra for solid UiO-68-BTDB.

2.4 Electrochemical Measurements

MOF powder was ground with poly(vinylidene fluoride) and then ultrasonically dispersed in acetone. The resultant slurry was then drop-casted onto indium tin oxide (ITO) glass with an area of $0.5 \times 0.5 \text{ cm}^2$. A Pt wire (counter electrode), a Ag/AgCl electrode (reference electrode), and a coated ITO conductive glass (working electrode) were assembled into a three-electrode system with 0.5 M Na_2SO_4 aqueous solution used as the electrolyte. The photocurrent measurements were conducted under the irradiation of a 300 W xenon lamp (100 mW cm^{-1}) with a 420 nm cut-off filter. The Mott-Schottky plots were collected at 500 Hz, 750 Hz, 1000 Hz. For electrochemical impedance spectroscopy (EIS) measurements, the amplitude of the sinusoidal wave was 5 mV with frequency ranging from 100 kHz to 0.05 Hz.

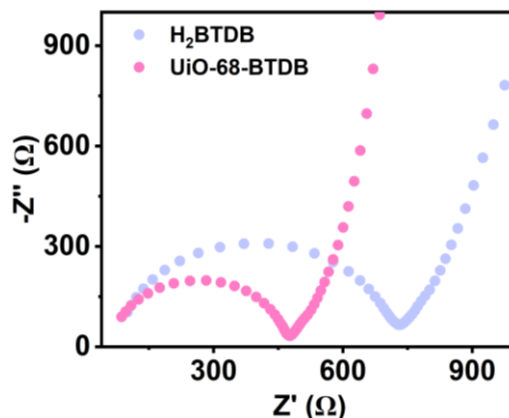


Figure S7. The EIS Nyquist plots of UiO-68-BTDB and H₂BTDB linker.

3. Photocatalytic reactions

3.1 General procedure for the synthesis of benzimidazoles. In a flat quartz glass jar with a magnetic stirrer, aromatic aldehyde (0.2 mmol), *o*-phenylenediamine (0.2 mmol), EtOH (3 mL) and UiO-68-BTDB (1 mol%) were added. The open-air reaction container was placed under a 10 W blue LED lamp (455-460 nm) and stirred for 3 h. After the reaction, the mixture was extracted three times with ethyl acetate, then the organic layers were combined and dried over anhydrous Na₂SO₄ and the solvent was removed in vacuo. The crude product was purified by silica gel column chromatography to give the target product.

3.2 General procedure for the synthesis of benzothiazoles. In a flat quartz glass jar with a magnetic stirrer, aromatic aldehyde (0.2 mmol), *o*-2-aminophenol (0.2 mmol), EtOH (3 mL) and UiO-68-BTDB (1 mol%) were added. The open-air reaction container was placed under a 10 W blue LED lamp (455-460 nm) and stirred for 1.5 h. After the reaction, the mixture was extracted three times with ethyl acetate, then the organic layers were combined and dried over anhydrous Na₂SO₄ and the solvent was removed in vacuo. The crude product was purified by silica gel column chromatography to give the target product.

3.3 Synthesis of UiO-68

A mixture of ZrCl₄ (50.0 mg, 0.21 mmol), 1,4-di(4-carboxyphenyl)benzene (H₂TPDC, 79 mg, 0.21 mmol), N,N-dimethylformamide (DMF, 3.0 mL) and TFA (0.49 mL, 6.40 mmol) was placed in a 20 mL vial and heated at 150 °C for 36 h. After it was cooled to room temperature, the precipitate were collected and solvent exchanged with DMF and acetone for several times to afford the as synthesized UiO-68.

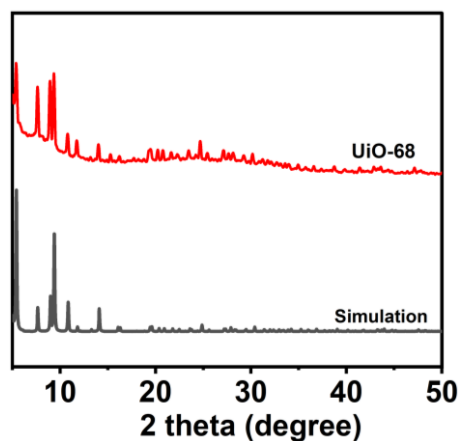


Figure S8. PXRD pattern of the as-synthesized UiO-68.

Table R1 Comparison of photocatalytic performance for the synthesis of benzimidazoles (BIs) / benzothiazoles (BTs) with other available materials

Catalyst	Product	Time(h)	Light Source	Yield	ROS	Nature ^[a]	Ref.
pytz	BIs, BTs	2	Visible light	90	O ₂	HMG	1
Fluorescein	BIs	2	10W LED	90	O ₂ ^{•-}	HMG	2
TZ-HCP	BIs	1	6W LED	98	¹ O ₂	HTG	3
MOF-1(Co)	BIs	1	10W LED	93	O ₂ ^{•-}	HTG	4
Ru(bpy) ₃ Cl ₂	BIs	24	45 W fluorescent light	72	O ₂ ^{•-}	HMG	5
RB	BIs	2	11W LED	90	O ₂ ^{•-}	HMG	6
BTT-TPA-COF	BIs	7	36W LED	85	O ₂ ^{•-}	HTG	7
Pt(0.2)/TiO ₂	BIs	4	2 KW Xe lamp	91	/	HTG	8
C-TiO ₂	BIs	0.5	300 W Xe lamp	85	O ₂	HTG	9
BTP-CMP	BIs	3	10W LED	93	O ₂ ^{•-}	HTG	10
UiO-68-BTDB	BIs, BTs	3	10W LED	94	¹O₂; O₂^{•-}	HTG	This work

[a] Nature: homogeneous (HMG), heterogenous (HMG)

3.3 Heterogeneous Catalysis Nature

Filtration experiment. In a flat quartz glass jar with a magnetic stirrer, benzaldehyde (0.2 mmol), o-phenylenediamine (0.2 mmol), EtOH (3 mL) and UiO-68-BTDB (1 mol%) were added. The open-air reaction container was placed under a 10 W blue LED

lamp and stirred for 3 h. After reacting for 1 h, the catalyst was removed through gravity filtration. The yields were then monitored at different periods.

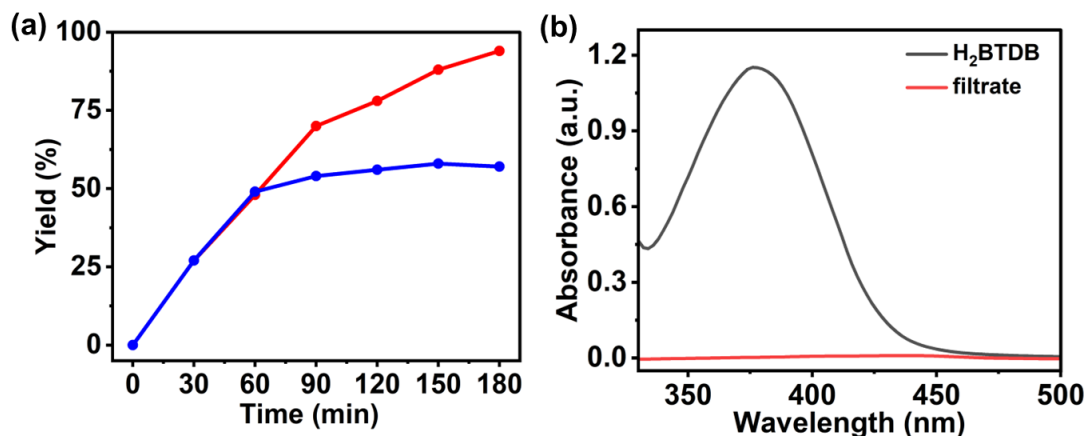


Figure S9. (a) Time-dependent curves of the synthesis for benzothiazoles. (b) UV-Vis absorption spectra were tested by taking H₂BTDB (5 μ M), and the reaction filtrate after 1 h of reaction.

Recycling experiments. The recycling experiments were carried out with *o*-phenylenediamine and benzaldehyde under the optimized reaction condition. After the reaction, UiO-68-BTDB was separated via filtration, then washed thoroughly with ethyl acetate. The recycled UiO-68-BTDB was dried and then subject to the next cycle.

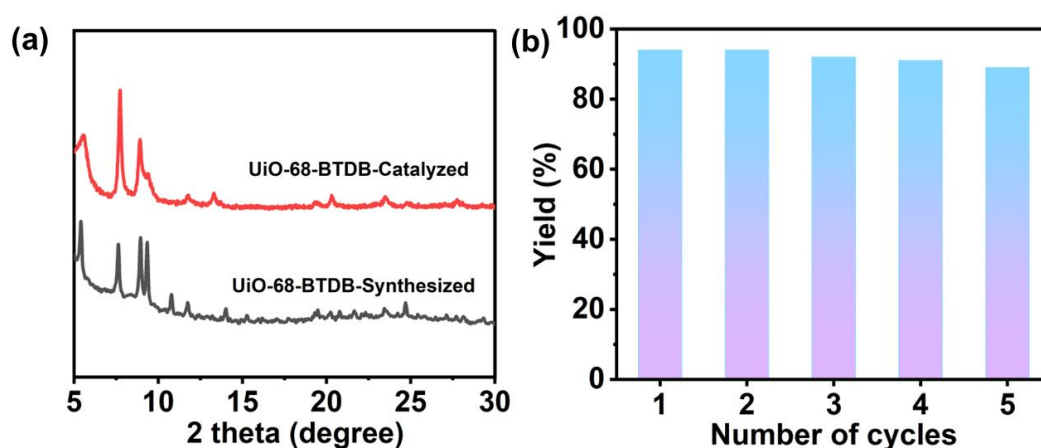
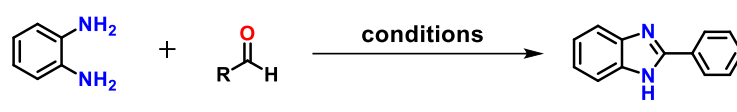


Figure S10. (a) PXRD patterns of the as-synthesized UiO-68-BTDB and catalysts after 5 cycles. (b) Recycling of UiO-68-BTDB for photocatalytic synthesis of 3a.

Table S2. Quenching experiments of reaction conditions ^a

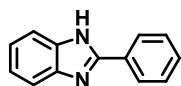


Entry	Deviation from standard condition	Yield ^b (%)
1	no variation	94
2	TEMPO	16
3	IPA	90
4	NaN ₃	26
5	BQ	23
6	AgNO ₃	25
7	C ₂ O ₄ (NH ₄) ₂	32

^a Standard conditions: *o*-phenylenediamine (0.2 mmol), benzaldehyde (0.2 mmol), UiO-68-BTDB, 3 mL EtOH, 10 W blue LED, room temperature, air, 3 h. ^b Isolated yields.

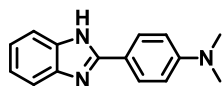
4. Characterization of Products

2-phenyl-1H-benzo[d]imidazole



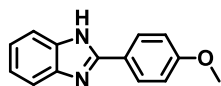
Isolated yield: 94%. White solid. ¹H NMR (400Hz, DMSO-*d*₆) δ 12.93 (s, 1H), 8.20 (d, *J* = 8.2 Hz, 2H), 7.68 – 7.52 (m, 4H), 7.49 (t, *J* = 7.3 Hz, 1H), 7.21 (dd, *J* = 6.0, 3.1 Hz, 2H). HRMS (ESI) calcd *m/z* for C₁₃H₁₀N₄⁺: [M+H]⁺ 195.0922, found 195.0925.

4-(1H-benzo[d]imidazol-2-yl)-N,N-dimethylaniline



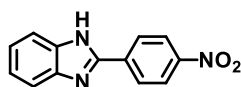
Isolated yield: 96%. White solid. ¹H NMR (400Hz, DMSO-*d*₆) δ 12.93 (s, 1H), 8.20 (d, *J* = 8.2 Hz, 2H), 7.68 – 7.52 (m, 4H), 7.49 (t, *J* = 7.3 Hz, 1H), 7.21 (dd, *J* = 6.0, 3.1 Hz, 2H). HRMS (ESI) calcd *m/z* for C₁₃H₁₀N₄⁺: [M+H]⁺ 195.0922, found 195.0925.

2-(4-methoxyphenyl)-1H-benzo[d]imidazole



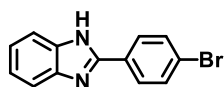
Isolated yield: 96%. ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.12 (d, *J* = 8.7 Hz, 2H), 7.62 (d, *J* = 7.4 Hz, 1H), 7.49 (d, *J* = 7.3 Hz, 1H), 7.18 – 7.14 (m, 2H), 7.11 (d, *J* = 8.7 Hz, 2H), 3.84 (s, 3H).

2-(4-nitrophenyl)-1H-benzo[d]imidazole

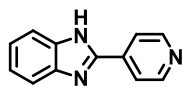


Isolated yield: 90%. Off white solid. ¹H NMR (400Hz, DMSO-*d*₆) δ = 13.31 (s, 1H), 8.43 (m, 4H), 7.74 (d, *J* = 7.6 Hz, 1H), 7.60 (d, *J* = 8.0 Hz, 1H), 7.32-7.24 (m, 2H). HRMS (ESI) calcd m/z for C₁₂H₉N₃O₂⁺: [M+H]⁺ 240.0773, found 240.0762.

2-(4-bromophenyl)-1H-benzo[d]imidazole

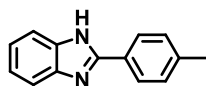


Isolated yield: 92%. White solid. ¹H NMR (400Hz, DMSO-*d*₆) δ = 12.95 (s, 1H), 8.08 (d, *J* = 8.4 Hz, 2H), 7.73(d, *J* = 8.8 Hz, 2H), 7.63(s, 1H), 7.51(s, 1H), 7.18(s, 1H). HRMS (ESI) calcd m/z for C₁₃H₉BrN₂⁺: [M+H]⁺ 273.0027, found 273.0027.



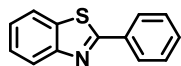
Isolated yield: 98%. White solid. ¹H NMR (400Hz, DMSO-*d*₆) δ = 13.28 (s, 1H), 8.78 (s, 2H), 8.11 (d, *J* = 6.0 Hz, 2H), 7.82-7.61 (m, 2H), 7.29 (s, 2H). HRMS (ESI) calcd m/z for C₁₂H₉N₃⁺: [M+H]⁺ 196.0875, found 196.0859.

2-(p-tolyl)-1H-benzo[d]imidazole



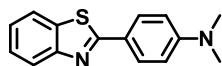
Isolated yield: 95%. White solid. ^1H NMR (400Hz, DMSO- d_6) δ = 12.77 (s, 1H), 8.03 (d, J = 8.4 Hz, 2H), 7.60 (d, J = 8.0 Hz, 1H), 7.47 (d, J = 8.4 Hz, 1H), 7.32 (d, J = 8.0 Hz, 2H), 7.19-7.12 (m, 2H), 2.35 (s, 3H). HRMS (ESI) calcd m/z for $\text{C}_{14}\text{H}_{12}\text{N}_2^+$: $[\text{M}+\text{H}]^+$ 209.1079, found 209.1073.

2-phenylbenzo[d]thiazole



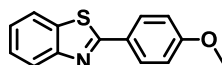
Isolated yield: 97%. ^1H NMR (400 MHz, Chloroform- d) δ 8.14 – 8.07 (m, 3H), 7.92 (d, J = 8.1 Hz, 1H), 7.54 – 7.48 (m, 5H), 7.40 (t, J = 7.6 Hz, 1H). HRMS (ESI) calcd m/z for $\text{C}_{13}\text{H}_9\text{NS}^+$: $[\text{M}+\text{H}]^+$ 212.0534, found 212.0500.

4-(benzo[d]thiazol-2-yl)-N,N-dimethylaniline



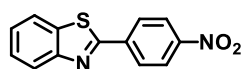
Isolated yield: 99%. ^1H NMR (400 MHz, Chloroform- d) δ 7.98 (t, J = 8.5 Hz, 3H), 7.84 (d, J = 7.9 Hz, 1H), 7.44 (t, J = 8.1 Hz, 1H), 7.31 (t, J = 7.3 Hz, 1H), 6.75 (d, J = 8.9 Hz, 2H), 3.06 (s, 6H). HRMS (ESI) calcd m/z for $\text{C}_{15}\text{H}_{14}\text{N}_2\text{S}^+$: $[\text{M}+\text{H}]^+$ 255.0956, found 255.0913.

2-(4-methoxyphenyl)benzo[d]thiazole



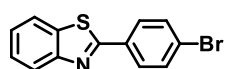
Isolated yield: 98%. ^1H NMR (400 MHz, Chloroform- d) δ 8.04 (d, J = 8.9 Hz, 3H), 7.87 (d, J = 7.9 Hz, 1H), 7.47 (t, J = 7.7 Hz, 1H), 7.35 (t, J = 7.5 Hz, 1H), 7.00 (d, J = 8.7 Hz, 2H). HRMS (ESI) calcd m/z for $\text{C}_{13}\text{H}_9\text{NS}^+$: $[\text{M}+\text{H}]^+$ 242.0640, found 242.0597.

2-(4-nitrophenyl)benzo[d]thiazole



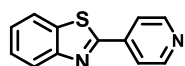
Isolated yield: 93%. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.36 (d, *J* = 8.8 Hz, 2H), 8.27 (d, *J* = 8.7 Hz, 2H), 8.14 (d, *J* = 9.4 Hz, 1H), 7.96 (d, *J* = 8.2 Hz, 1H), 7.56 (t, *J* = 7.6 Hz, 1H), 7.47 (t, *J* = 8.4 Hz, 1H). HRMS (ESI) calcd *m/z* for C₁₃H₈N₂O₂S⁺: [M+H]⁺ 257.0385, found 257.0346.

2-(4-bromophenyl)benzo[d]thiazole



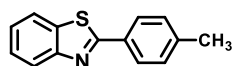
Isolated yield: 95%. ¹H NMR (400Hz, CDCl₃) δ = 8.07 (d, *J* = 9.6 Hz, 1H), 7.96 (d, *J* = 8.8 Hz, 2H), 7.90 (d, *J* = 7.6 Hz, 1H), 7.63 (d, *J* = 8.4 Hz, 2H), 7.52-7.48 (m, 1H), 7.42-7.38 (m, 1H). HRMS (ESI) calcd *m/z* for C₁₃H₈BrNS⁺: [M+H]⁺ 289.9639, found 289.9586.

2-(pyridin-4-yl)benzo[d]thiazole



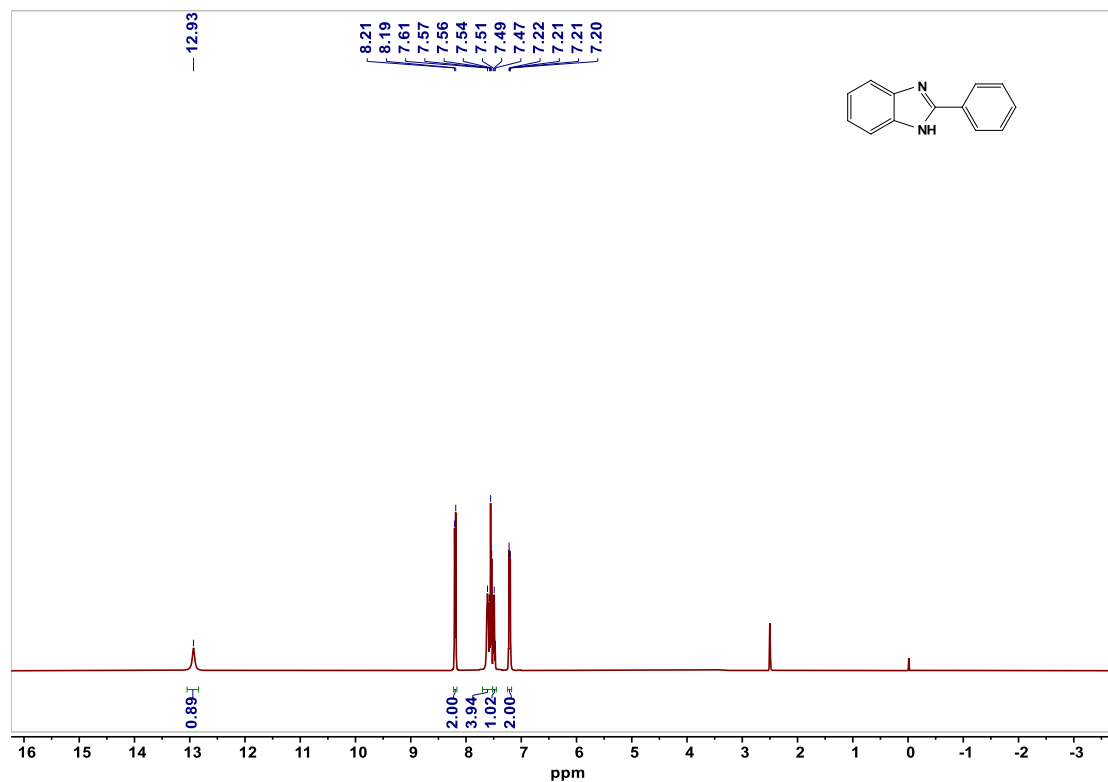
Isolated yield: 99%. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.78 (d, *J* = 5.3 Hz, 2H), 8.14 (d, *J* = 8.1 Hz, 1H), 7.98 – 7.92 (m, 3H), 7.55 (t, *J* = 7.6 Hz, 1H), 7.47 (t, *J* = 7.5 Hz, 1H). HRMS (ESI) calcd *m/z* for C₁₅H₁₁N₂OS⁺: [M+H]⁺ 213.0481, found 213.0446.

2-(p-tolyl)benzo[d]thiazole

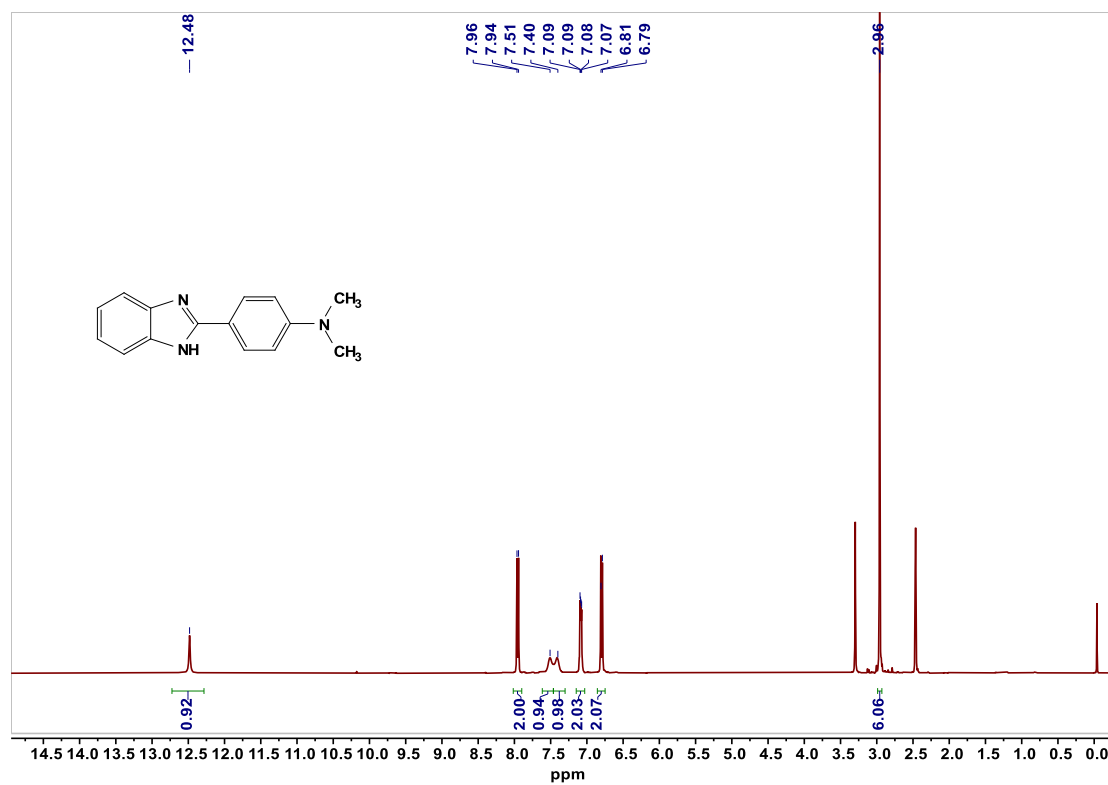


Isolated yield: 96%. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.06 (d, *J* = 8.0 Hz, 1H), 7.99 (d, *J* = 8.1 Hz, 2H), 7.90 (d, *J* = 8.0 Hz, 1H), 7.51 – 7.46 (m, 1H), 7.38 (t, *J* = 7.6 Hz, 1H), 7.30 (d, *J* = 8.0 Hz, 2H), 2.43 (s, 3H). HRMS (ESI) calcd *m/z* for C₁₄H₁₁NS⁺: [M+H]⁺ 226.0685, found 226.0649.

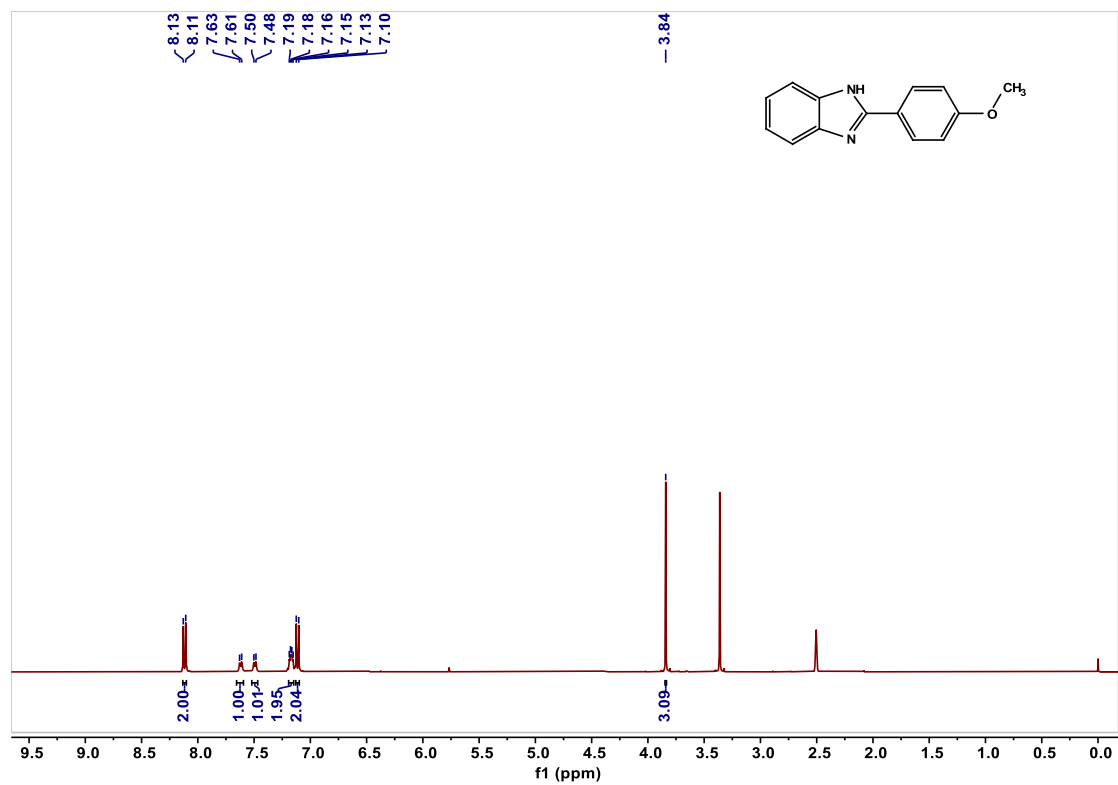
NMR spectra



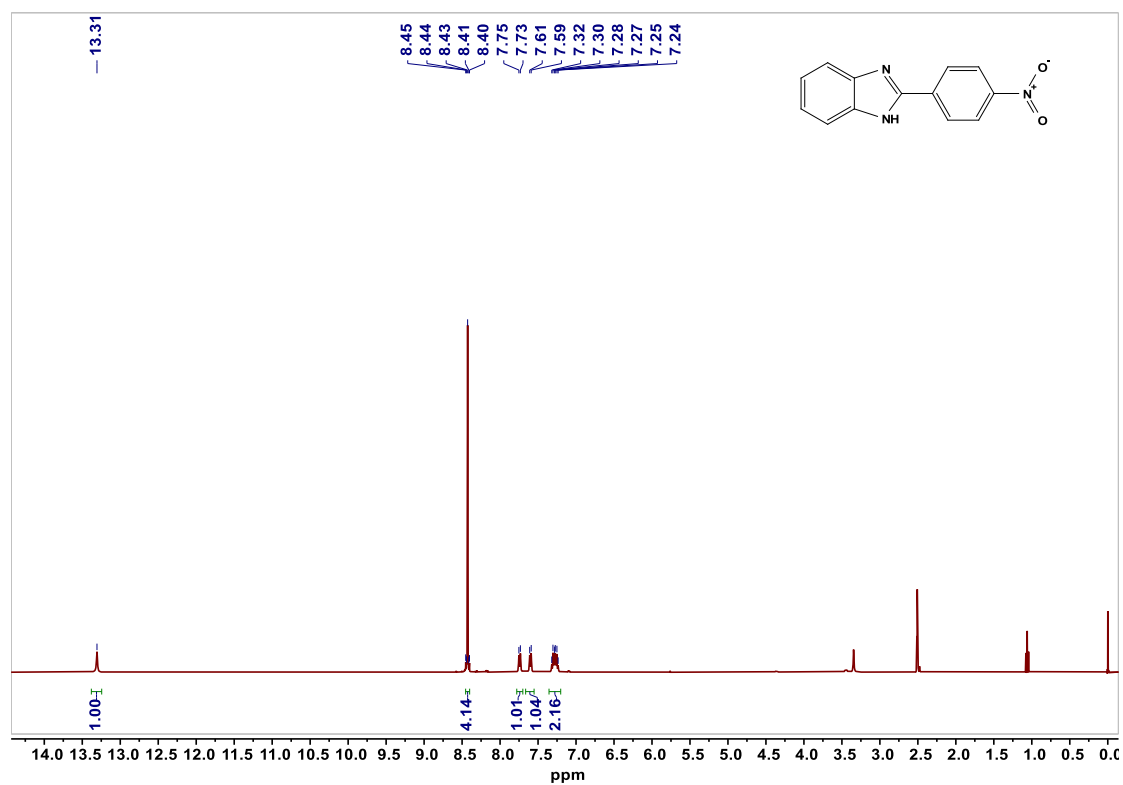
^1H NMR of **3a** in $\text{DMSO-}d_6$



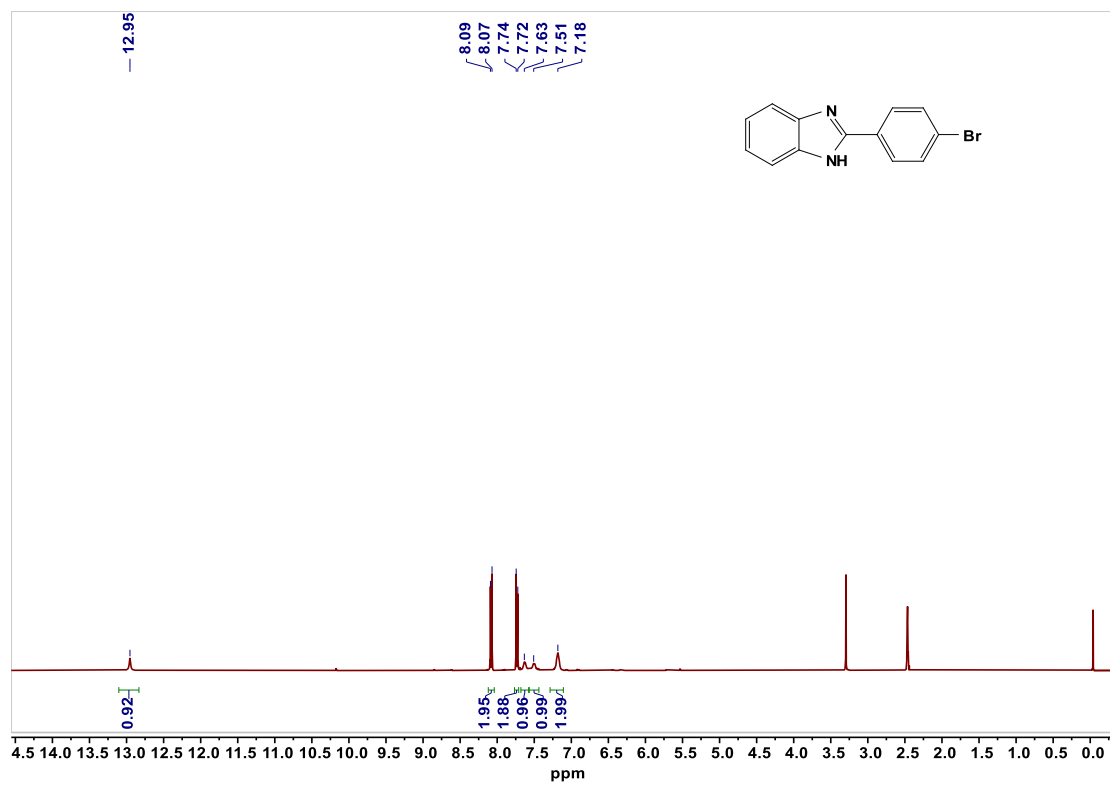
^1H NMR of **3b** in $\text{DMSO-}d_6$



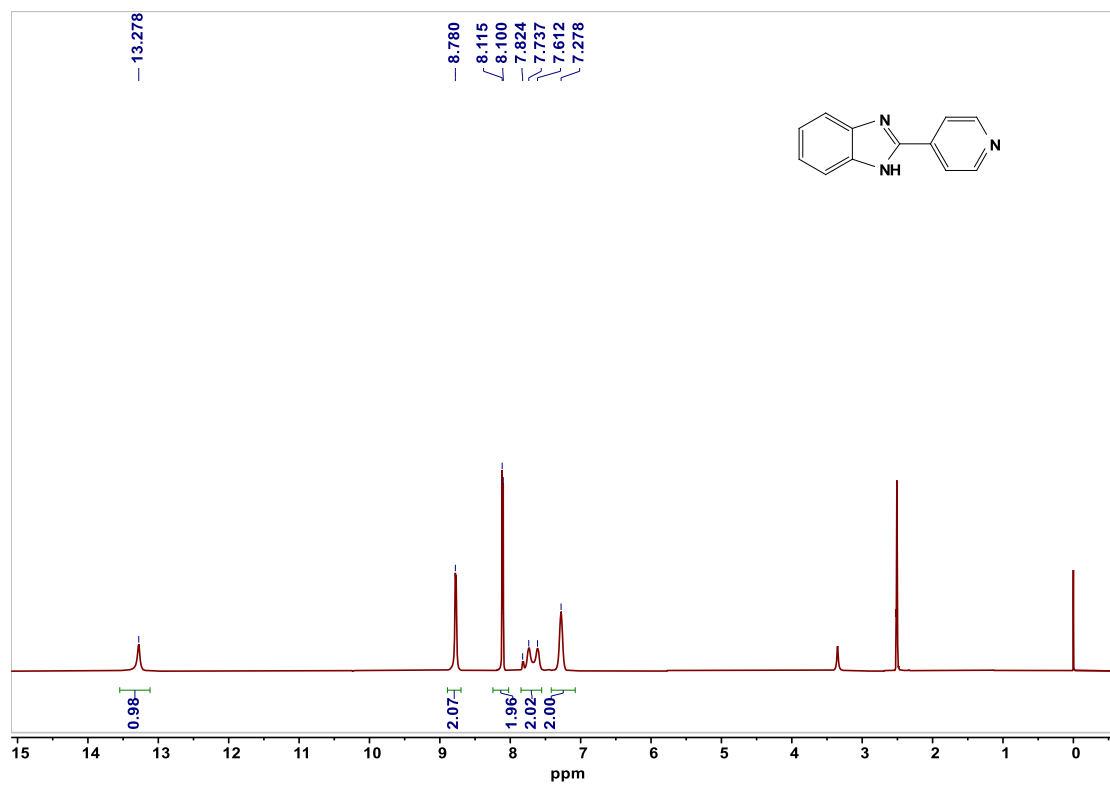
^1H NMR of **3c** in $\text{DMSO-}d_6$



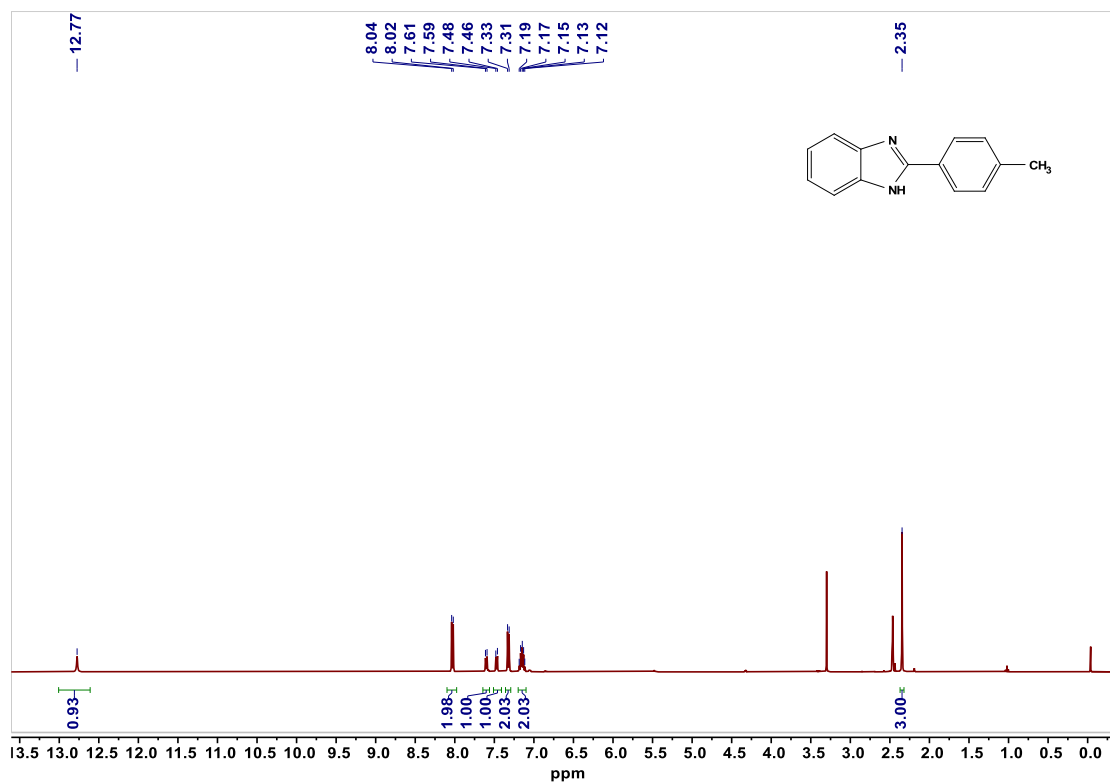
^1H NMR of **3d** in $\text{DMSO-}d_6$



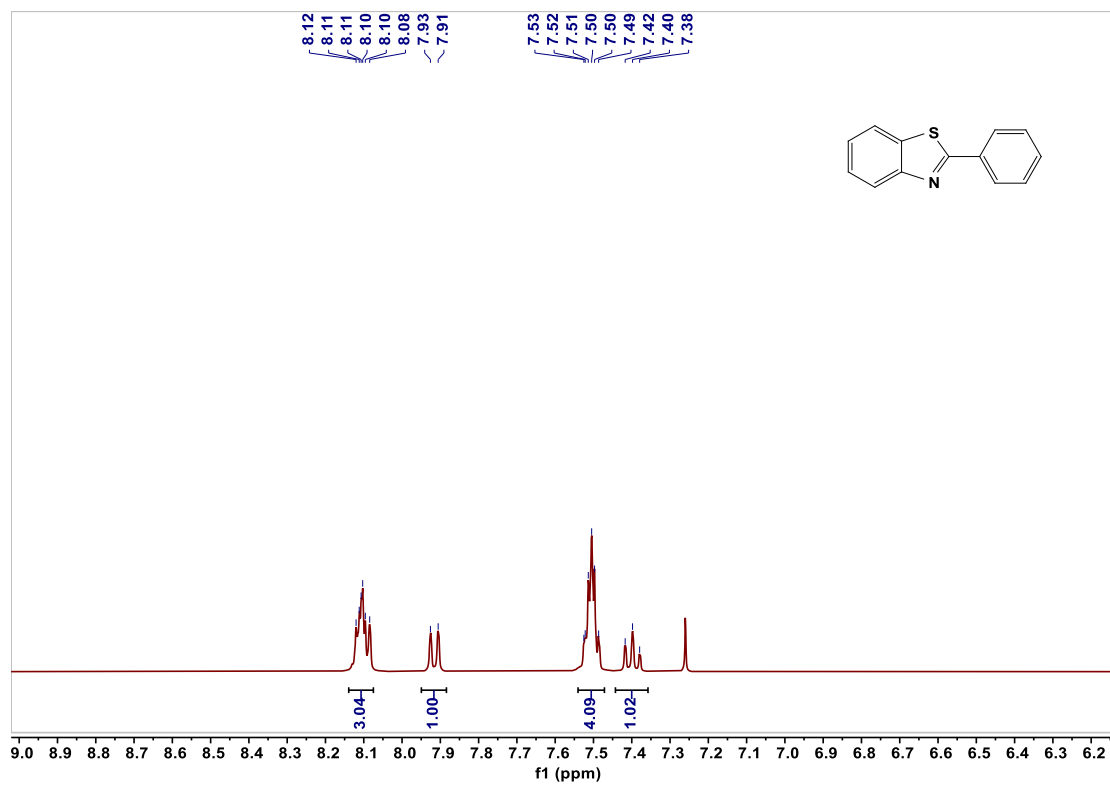
^1H NMR of **3e** in $\text{DMSO-}d_6$



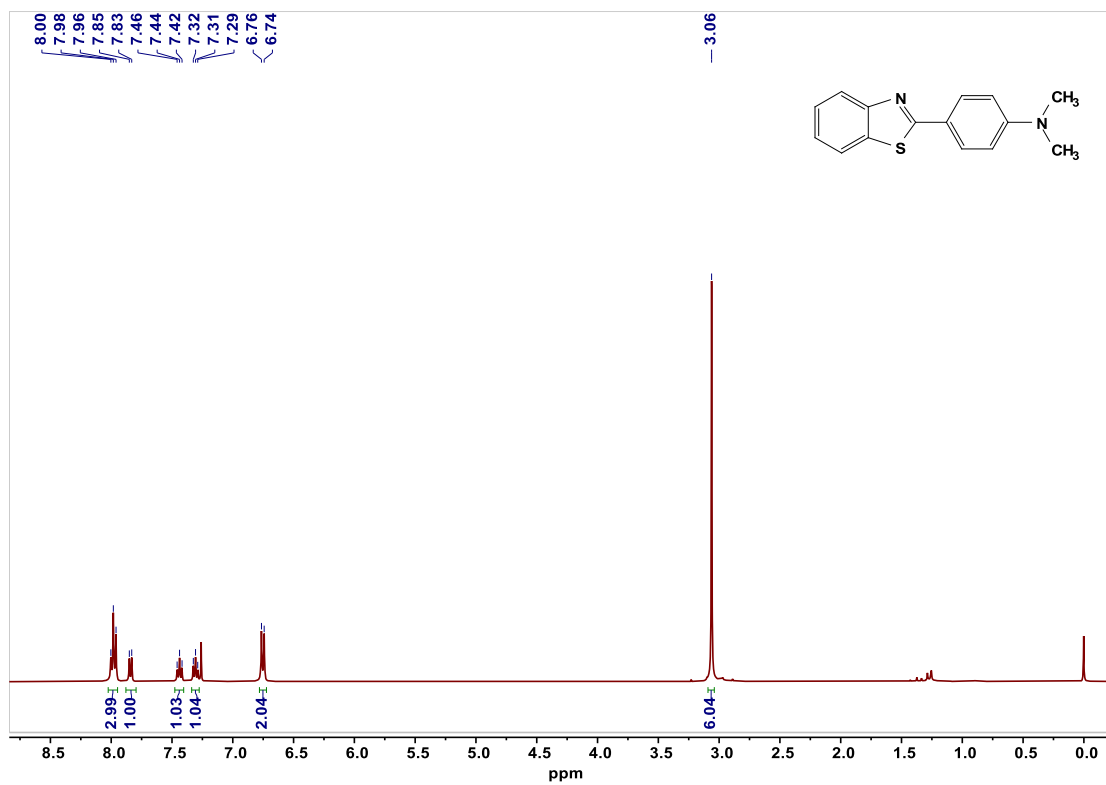
^1H NMR of **3f** in $\text{DMSO-}d_6$



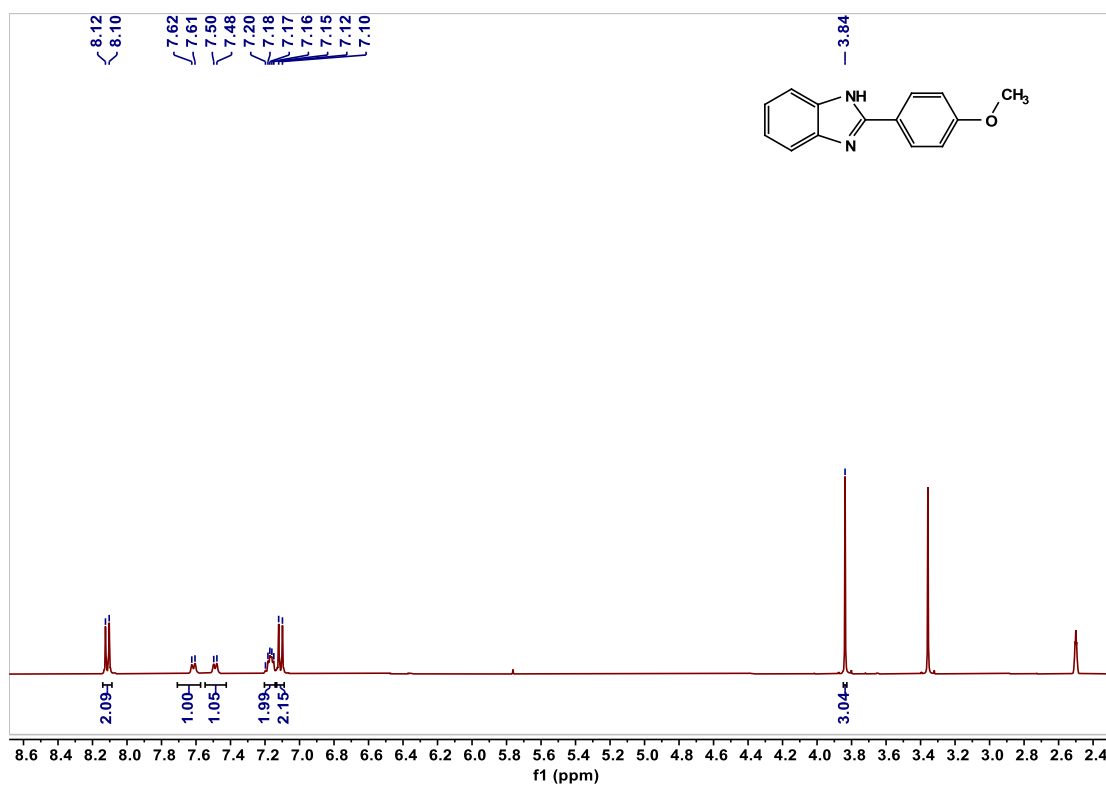
$^1\text{H NMR}$ of **3g** in $\text{DMSO-}d_6$



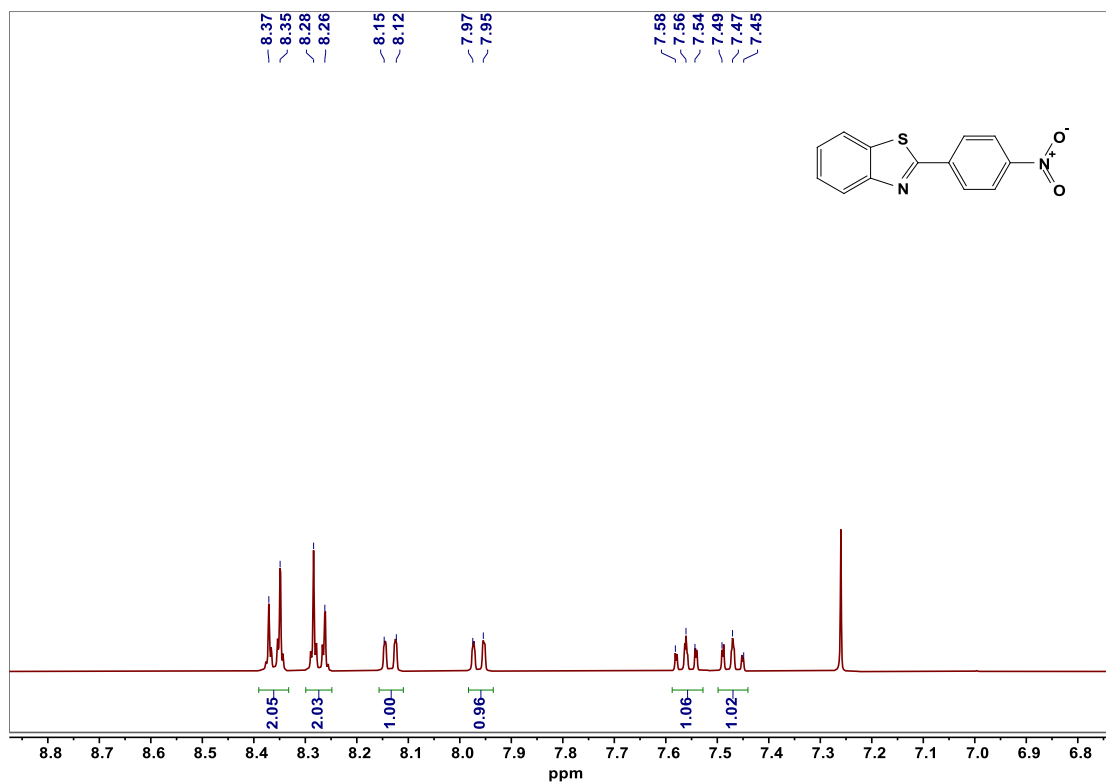
$^1\text{H NMR}$ of **6a** in CDCl_3



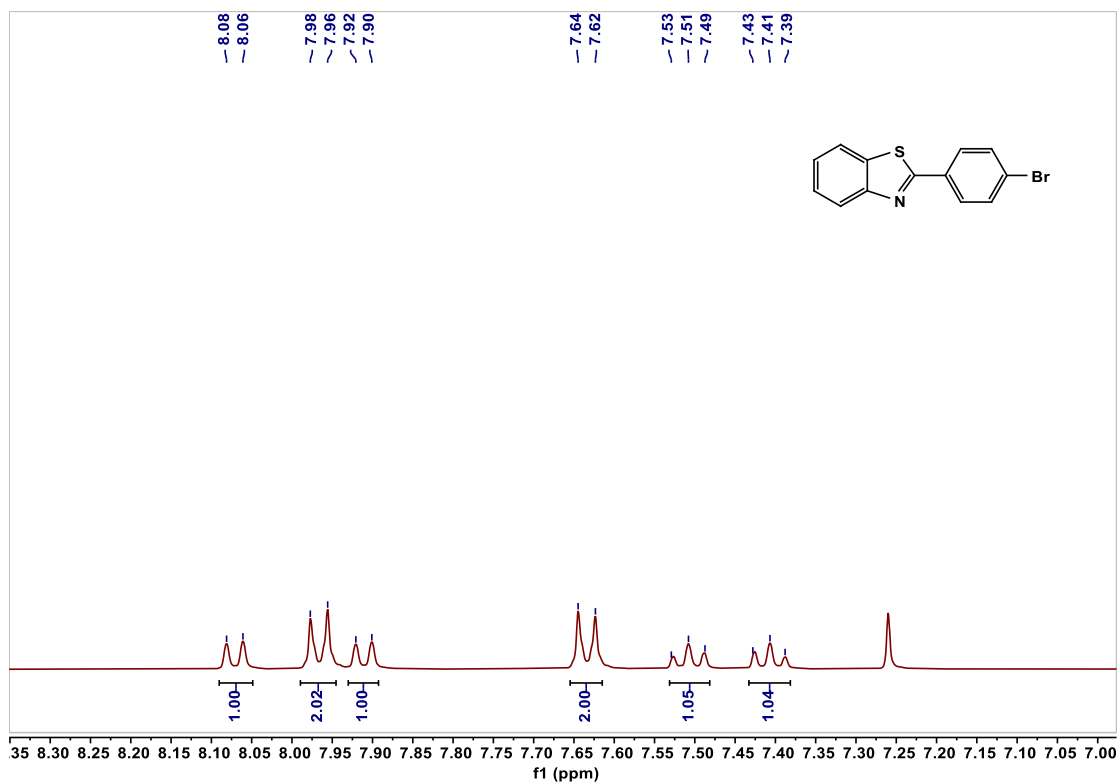
¹H NMR of **6b** in CDCl₃



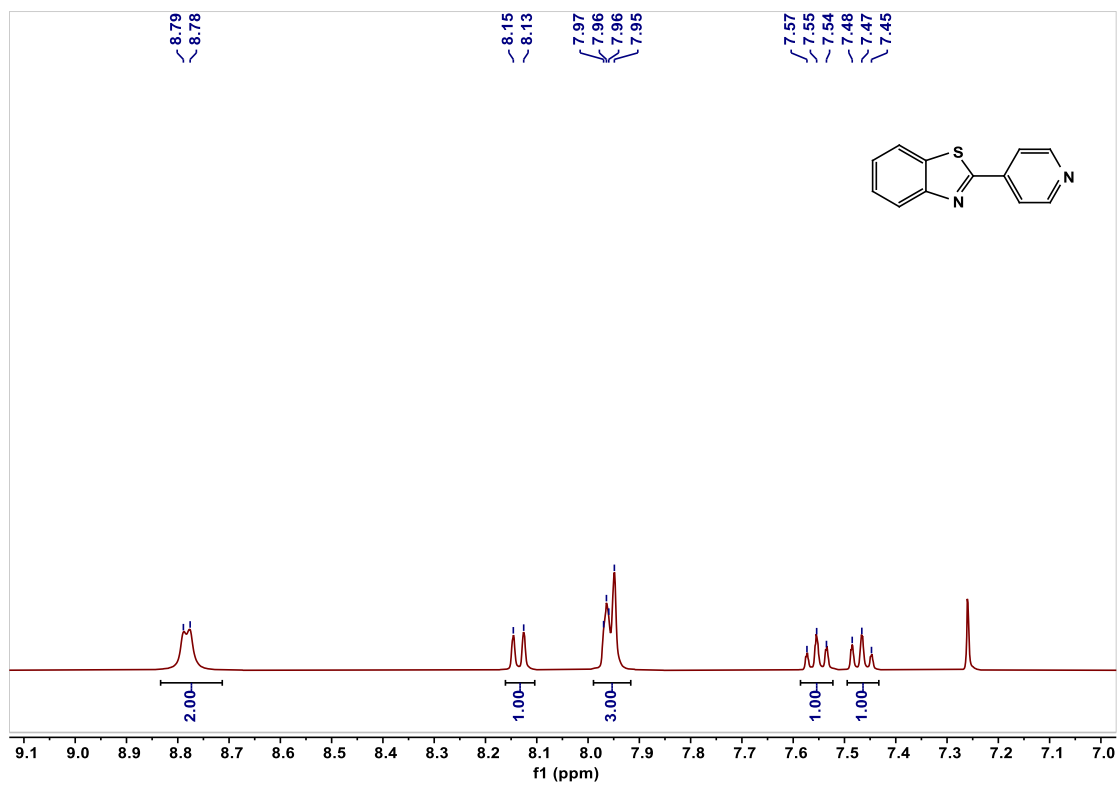
¹H NMR of **6c** in CDCl₃



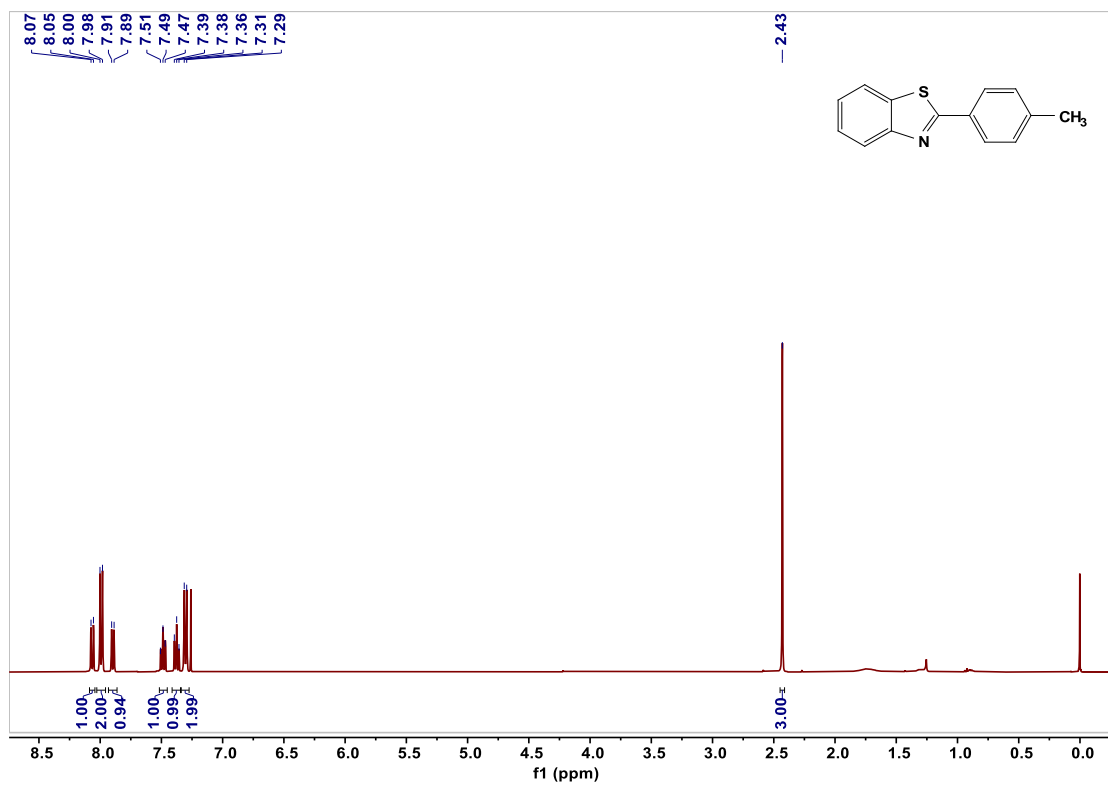
^1H NMR of **6d** in CDCl_3



^1H NMR of **6e** in CDCl_3



¹H NMR of **6f** in CDCl₃



¹H NMR of **6g** in CDCl₃

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