

Supporting Information

Hydrazone-based covalent organic frameworks as efficient photocatalytic redox reactions

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Section S1. Reagents and solvents

All the chemicals are commercially available and used without further purification. Benzaldehyde and its derivatives were purchased from Macklin Co., Ltd. 2-Aminophenylthiol, 2-amino-4-chlorobenzenethiol, hexamethylenetetramine (HMTA), 2,5-dihydroxyterephthalic acid, resorcinol, deuterated solvents for NMR measurement were purchased from Aladdin Industries Inc. 2,2,6,6-Tetramethylpiperidine oxide (TEMPO, 98%), 5,5-dimethyl-1-pyrroline N-oxide (DMPO, 98%), benzoquinone (AR), sodium azide (NaN_3 , AR), potassium iodide (KI, 99%), copper sulfate (CuSO_4) were purchased from Macklin Inc. Ethanol (EtOH), methanol (MeOH), tetrahydrofuran (THF), acetonitrile (CH_3CN), mesitylene and 1,4-dioxane were purchased from Beijing Chemical Plant Co., Ltd. Trifluoroacetic acid (TFA), phenol, hydrazine hydrate were purchased from Tianjin Fuyu Co., Ltd. The monomers were synthesized according to the reported methods.¹⁻⁴

Section S2. The characterization

2.1 The Fourier Transform Infrared (FT-IR) spectroscopy

The Fourier Transform Infrared (FT-IR) spectroscopy was recorded on a Nicolet is5 spectrometer using KBr pellets within the wavenumber range 400 to 4000 cm^{-1} .

2.2 Nuclear magnetic resonance (NMR) spectroscopy

^1H NMR and ^{13}C NMR spectra were carried out on a Bruker-Avance III 400 MHz spectrometer. The solid-state ^{13}C CP/MAS NMR measurement was recorded using a Bruker AVANCE III 400 WB spectrometer.

2.3 Powder X-ray diffraction (PXRD)

PXRD data was collected on a PANalytical BV Empyrean diffractometer with a 2θ range of 1° to 30° at 298 K. The Pawley refinement of the experimental PXRD was conducted by the Reflux module in the Material Studio 2019.

2.4 Thermogravimetric analysis (TGA)

Thermogravimetric analyses (TGA) were recorded from room temperature to 800 $^\circ\text{C}$ with a heating rate of 10 $^\circ\text{C}/\text{min}$ on a NETZSCH STA449 F3 thermal analyzer under a N_2 atmosphere.

2.5 Nitrogen adsorption-desorption isotherm measurements (BET)

The N_2 adsorption and desorption isotherms were performed at 77 K with a Micromeritics ASAP 2020M system. The Brunauer-Emmett-Teller (BET) method was used to calculate surface area and pore volume. Nonlocal density functional theory (NLDFT) was utilized to analyze the pore size distributions.

2.6 X-ray photoelectron spectroscopy (XPS)

X-ray photoelectron elemental distribution binding energy and valence band (VB) energy were obtained by Thermo ESCALAB 250 X-ray photoelectronic spectrometer.

2.7 UV-visible absorption spectra (Solid) and Kubelka-Munk function

The solid-state UV-Vis diffuse reflectance spectra were recorded from 200 to 800 nm on a Shimadzu UV-4100-NIR spectrophotometer. The variation relationship between the diffuse reflectance of a solid sample and wavelength was measured using spectrophotometer. It is worth noting that R_∞ was the diffuse reflectance of the sample. The Kubelka-Munk function is defined according to the formula:

$$F(R_{\infty}) = \frac{(1 - R_{\infty})^2}{2R_{\infty}}$$

Typically, the wavelength can be converted into photon energy ($h\nu$) using the conversion formula:

$$h\nu = \frac{hc}{\lambda}$$

where h is Planck's constant, c is the speed of light, and λ is the wavelength. The band gap E_g is calculated from the Tauc plot graph, where $(F(R_{\infty})h\nu)^2$ is plotted against the photon's energy $h\nu$.

Subsequently, we constructed a plot with $(F(R_{\infty})h\nu)^2$ as the vertical axis and $h\nu$ as the horizontal axis. By extending the linear portion of the curve to its intersection with the horizontal axis, the value at this intersection corresponds to the band gap energy E_g of the material.

2.8 Scanning electron microscopy (SEM) and high-resolution transmission electron microscopy (HR-TEM)

Scanning electron microscopy (SEM) and high-resolution transmission electron microscopy (HR-TEM) images were obtained by a Hitachi SU8020 microscope and a Tecnai G2 S-Twin F20 field-emission transmission electron microscope, respectively.

2.9 The water contact angle experiment

The water contact angle experiment was tested using a DSA-25-KRÜSS GMBH goniometer.

2.10 Photoluminescence (PL) spectroscopy

The Solid-state fluorescence and fluorescence lifetime were performed on an Edinburgh Instrument FLS920 spectrometer.

2.11 Electron Paramagnetic Resonance (EPR) test

The electron paramagnetic resonance (EPR) spectra were acquired using a Bruker EMXnano ESR spectrometer. 1 mg of COF sample was suspended in 1 mL of methanol, followed by the addition of 5 μ L of DMPO. The mixture was subsequently drawn into a capillary tube, which was then placed in EPR specific tube for further radical production and detection. EPR spectra was acquired with scanning width of 100 G, scanning power of 1 mW, central field of 3350 G, sweep time of 40s and temperature of 25°C.

Section S3. Electrochemical measurements

Photoelectrochemical measurements were taken at room temperature on the instrument workstation (CHI760E, China) using a standard three-electrode system. The system included a platinum wire electrode as an assistant electrode, an Ag/AgCl reference electrode, and a glassy carbon electrode as a working electrode.

3.1 EIS measurement

Sample preparation: 5 mg of the material was added to a centrifuge tube containing anhydrous ethanol (225 μ L), deionized water (225 μ L), and 50 μ L *N*-methylpyrrolidone (NMP) solution of 1.5 wt% polyvinylidene fluoride (PVDF). The impedance tests were carried out in a 0.1 mol L⁻¹ KCl solution containing 5 mmol L⁻¹ [Fe(CN)₆]^{3-/4-}.

3.2 Mott-Schottky and photocurrent measurements

The 0.1 mol L⁻¹ Na₂SO₄ solution as electrolyte was employed to carry out Mott-Schottky and photocurrent measurements. 5 mg of COF was dispersed with 1 mL EtOH containing 10 µL of 5% Nafion. The suspension was meticulously placed onto the glassy carbon electrode and subsequently subjected to a drying process at 70 °C for 20 minutes. Mott-Schottky measurement was conducted in 0.1 mol L⁻¹ Na₂SO₄ solution at 1000, 1500, and 2000 Hz frequencies with 5 mV amplitude. The photocurrent measurements were performed in 0.1 mol L⁻¹ Na₂SO₄ aqueous solution under a 300 W Xe lamp irradiation.

Section S4. Synthesis of MOH-Tf1₁Tf2₂

Place **Tf1** (8.9 mg, 0.05 mmol), **Tf2** (19.4 mg, 0.1 mmol), **MOH** (57.2 mg, 0.225 mmol), and a mixed solvent of 1,4-dioxane/mesitylene (1:3, v/v, 2.4 mL) in an ampoule. The mixture was then ultrasonicated for 15 minutes to form a uniform suspension. Next, 0.3 mL of acetic acid solution (6 M) was added with continuing sonication for 10 minutes. The ampoule was flame-sealed after three freeze-pump-thaw cycles and heated at 120 °C for 72 h. The obtained precipitate was filtered and washed alternately with THF (30 mL) and acetone (30 mL) to remove organic residues. The solid was then dried at 80 °C for 24 hours to obtain a yellow powder in an 85% yield.

Section S5. Results and discussion

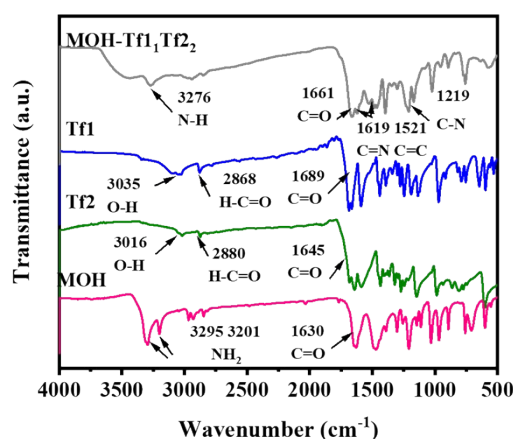


Fig. S1 FT-IR spectra of **MOH-Tf1₁Tf2₂** and corresponding monomers.

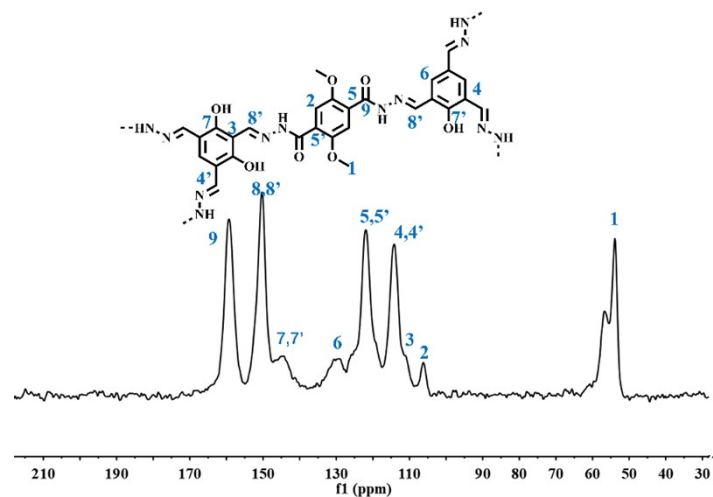


Fig. S2 ^{13}C CP-MAS solid-state NMR spectrum of **MOH-Tf₁₁Tf₂₁**.

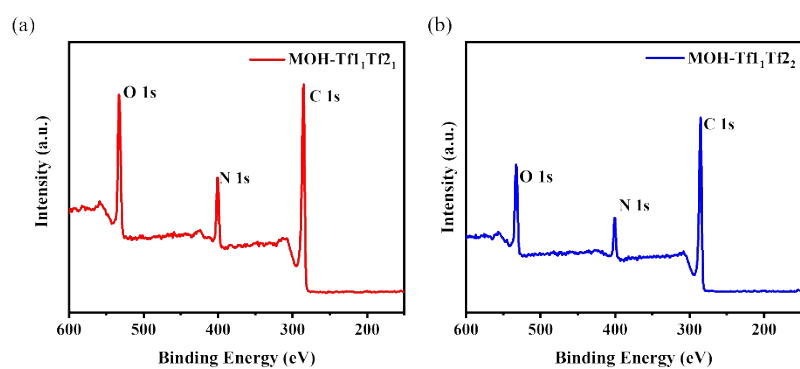


Fig. S3 (a) XPS full-scale survey spectrum of **MOH-Tf₁₁Tf₂₁**. (b) XPS full-scale survey spectrum of **MOH-Tf₁₁Tf₂₂**.

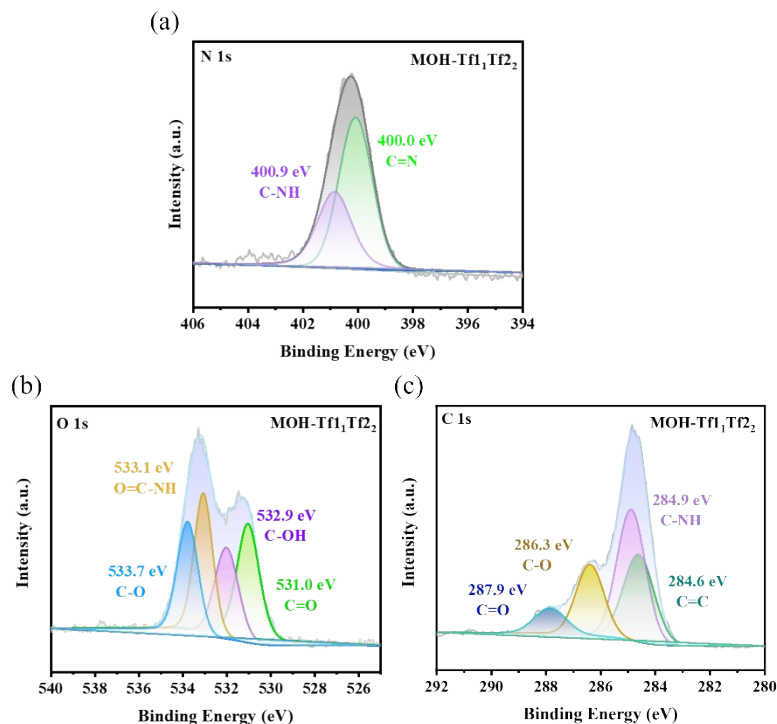


Fig. S4 (a) XPS spectra in the N 1s regions of **MOH-Tf₁Tf₂**. (b) XPS spectra in the O 1s regions of **MOH-Tf₁Tf₂**. (c) XPS spectra in the C 1s regions of **MOH-Tf₁Tf₂**.

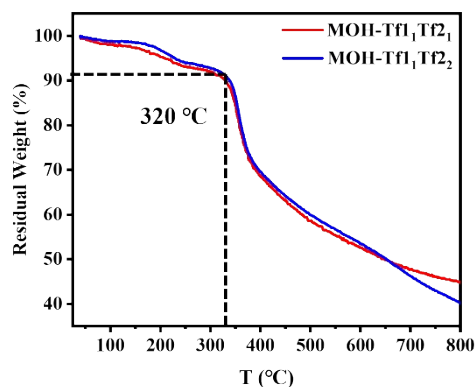


Fig. S5 The TGA curves of **MOH-Tf₁Tf₂₁** and **MOH-Tf₁Tf₂**.

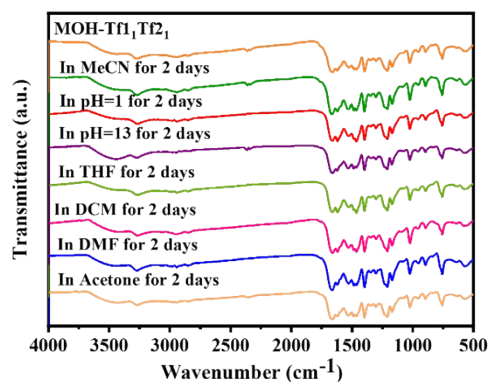


Fig. S6 FT-IR spectra of **MOH-Tf₁Tf₂₁** before and after treatment in different media for 2 days.

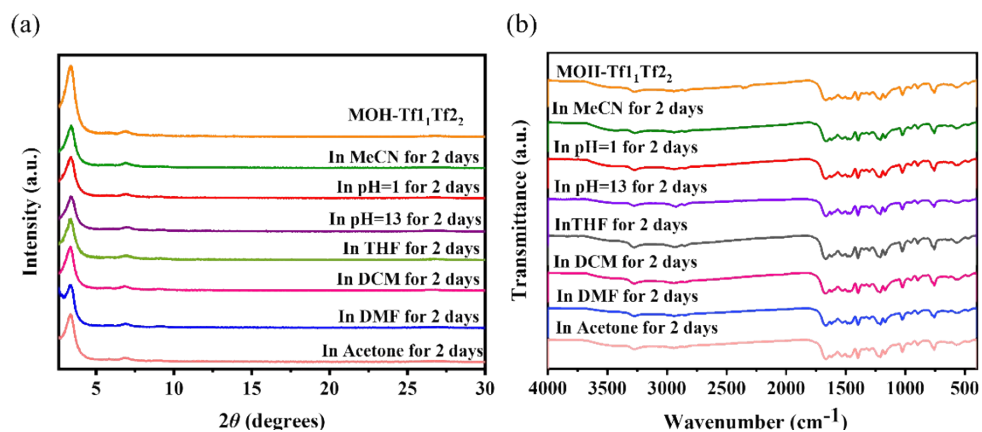


Fig. S7 (a) The PXRD profiles of **MOH-Tf₁Tf₂** before and after treatment in different media for 2 days. (b) FT-IR spectra of **MOH-Tf₁Tf₂** before and after treatment in different media for 2 days.

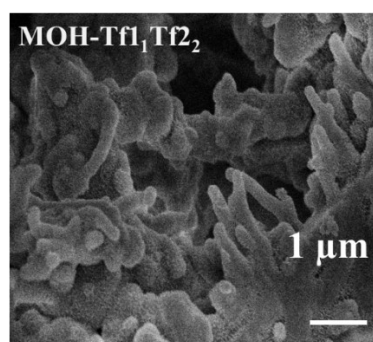


Fig. S8 The SEM of **MOH-Tf₁Tf₂**.

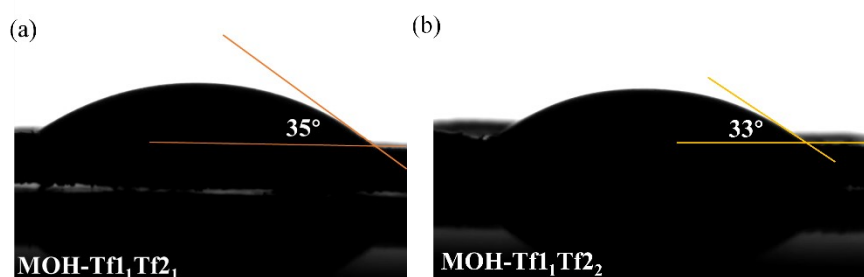


Fig. S9 (a) The water contact angle of **MOH-Tf₁Tf₁**. (b) The water contact angle of **MOH-Tf₁Tf₂**.

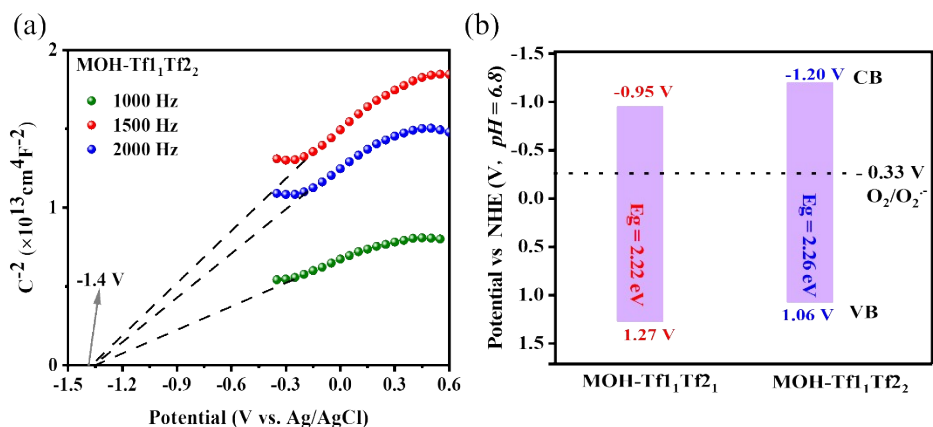


Fig. S10 (a) Mott-Schottky plots of **MOH-Tf1₁Tf2₂**. (b) Band-structure diagrams of **MOH-Tf1₁Tf2₁** and **MOH-Tf1₁Tf2₂**.

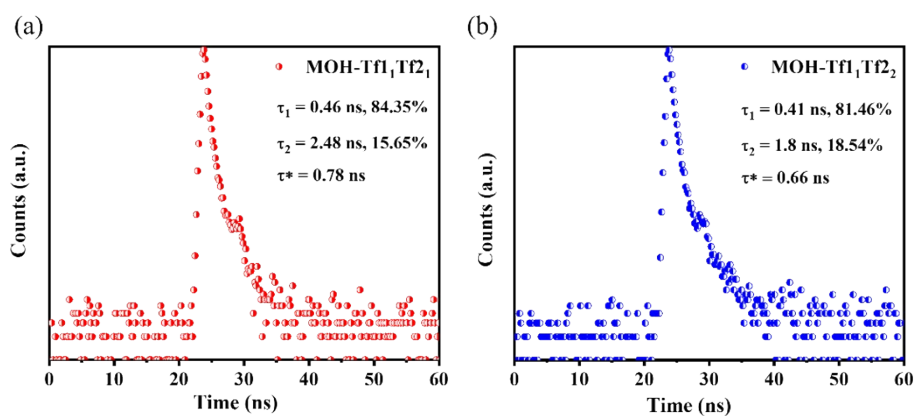


Fig. S11. (a) Fluorescence lifetime spectrum of **MOH-Tf1₁Tf2₁** upon excitation at 350 nm. (b) Fluorescence lifetime spectrum of **MOH-Tf1₁Tf2₂** upon excitation at 350 nm.

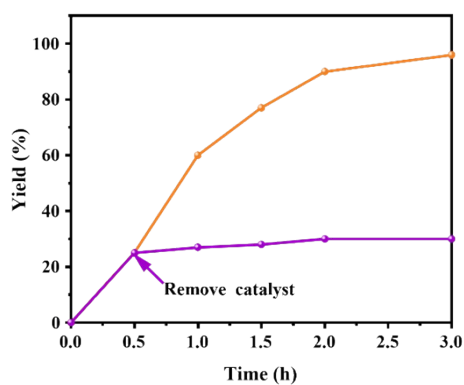


Fig. S12 Kinetic curve and hot filtration experiment of forming 2-phenylbenzothiazole using the **MOH-Tf1₁Tf2₁** as a photocatalyst under visible-light irradiation (The yellow line represents the kinetic curve; and the purple line represents the heat filtration curve).

Table S1. Optimization of the conditions for synthesizing 2-phenylbenzothiazole using **MOH-Tf1₁Tf2₁** photocatalyst under visible-light irradiation.^a

Reaction scheme: 2-aminobenzenethiol (1a) + benzaldehyde (2a) $\xrightarrow[\text{Solvent, O}_2, \text{rt. 30 W blue light}]{\text{Photocatalyst}}$ 2-phenylbenzothiazole (3a)

Entry	Catalyst	Solvents	Amount of catalyst (mg)	Time (h)	Yield (%) ^b
1	MOH-Tf1₁Tf2₁	THF	3	3	65
2	MOH-Tf1₁Tf2₁	MeCN	3	3	45
3	MOH-Tf1₁Tf2₁	DMF	3	3	55
4	MOH-Tf1₁Tf2₁	EtOH	3	3	82
5	MOH-Tf1₁Tf2₁	MeOH	3	3	85
6	MOH-Tf1₁Tf2₁	H ₂ O	3	3	60
7	MOH-Tf1₁Tf2₁	MeOH/H ₂ O=1:1	3	3	96
8	MOH-Tf1₁Tf2₁	MeOH/H ₂ O=1:3	3	3	80
9	MOH-Tf1₁Tf2₁	MeOH/H ₂ O=3:1	3	3	89
10	MOH-Tf1₁Tf2₁	MeOH/H ₂ O=1:1	1	3	60
11	MOH-Tf1₁Tf2₁	MeOH/H ₂ O=1:1	2	3	82
12 ^c	MOH-Tf1₁Tf2₁	MeOH/H ₂ O=1:1	3	3	trace
13 ^d	MOH-Tf1₁Tf2₁	MeOH/H ₂ O=1:1	3	3	trace
14 ^e	MOH-Tf1₁Tf2₁	MeOH/H ₂ O=1:1	3	3	trace
15 ^f	MOH-Tf1₁Tf2₂	MeOH/H ₂ O=1:1	3	3	90

^aReaction condition: 2-aminobenzenethiol (0.22 mmol), benzaldehyde (0.2 mmol), photocatalyst (3 mg), and MeOH/H₂O (1.6 mL, v/v = 1:1,) was placed in a 5 mL flask and irradiated with a 30 W blue LED under an O₂ atmosphere at room temperature for 3 h.

^bYield of isolated product.

^cReaction was carried out under Ar.

^dReaction was carried out in the dark.

^eReaction was carried out without a photocatalyst.

^fReaction was carried out with **MOH-Tf1₁Tf2₂** as a photocatalyst.

Table S2. Comparison of other catalysts for photocatalytic synthesis of benzothiazole with the catalysts in this paper.

Catalyst	Solvents	Amount of catalyst	Temperature (°C)	Time (h)	Yield (%)	Reference
CMP-Th-Ph-T	MeOH	8 mg	25 °C	4	99	S5
fluorescein	MeOH	10 mol%	25 °C	3	92	S6
TPA-MP-1	EtOH	5 mg	25 °C	3	86	S7
CdNS	MeOH	5 mg	25 °C	0.5	96	S8
TAPT-TP-COF	EtOH : H ₂ O=1:1	5 wt%	25 °C	10	98	S9
[Bmim]PF₆	/		110 °C	2	89	S10
VHb	DMSO	0.05 mol%	25 °C	12	16	S11
MOH-Tf1₁Tf2₁	MeOH : H ₂ O=1:1	3 mg	25 °C	3	96	<i>This work</i>

MOH-Tf1₁Tf2₂	MeOH : H ₂ O=1:1	3 mg	25 °C	3	90	<i>This work</i>
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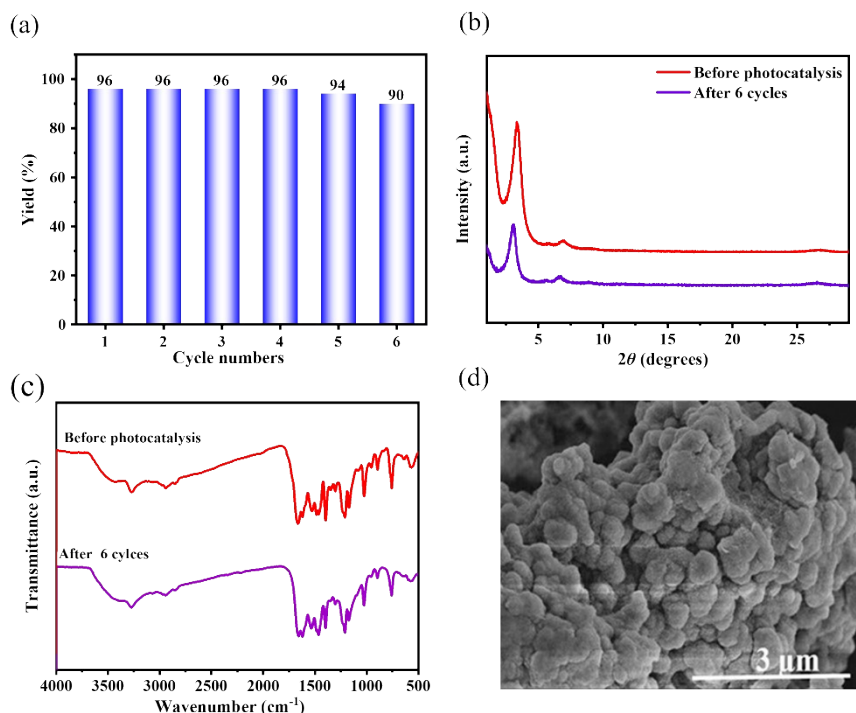
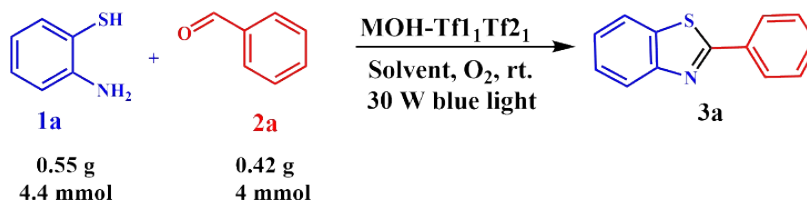


Fig. S13 (a) Catalytic recycle experiments of **MOH-Tf1₁Tf2₁** as the photocatalyst for the synthesis of 2-phenylbenzothiazole. (b) PXRD patterns of the **MOH-Tf1₁Tf2₁** before and after six catalytic cycles. (c) The FT-IR spectra of the **MOH-Tf1₁Tf2₁** before and after six catalytic cycles. (d) SEM image of the **MOH-Tf1₁Tf2₁** after six cycles.



Scheme S1. The large-scale photocatalytic synthesis of 2-phenylbenzothiazole by **MOH-Tf1₁Tf2₁** (30 mg) as a heterogeneous photocatalyst under optimal conditions.

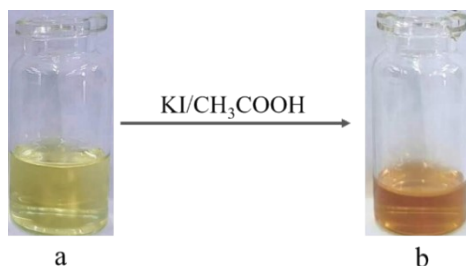
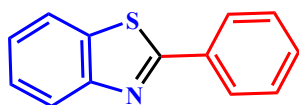


Fig. S14. Detection of H₂O₂ in the reaction mixture of photocatalytic synthesis of 2-phenyl benzothiazole with KI/CH₃COOH. 2-Aminothiophenol (0.22 mmol), benzaldehyde (0.2 mmol) and catalyst **MOH-Tf1₁Tf2₁** (3 mg) were added into MeOH: H₂O (v/v, 1/1, 1.6 mL). After being irradiated for 3 hours under an O₂ atmosphere, the catalyst was removed. Then, KI (0.1 M aqueous solution) and aqueous acetic acid (0.1 M) were added to the filtrate, resulting in a color change of the reaction

mixture from yellowish to brown.

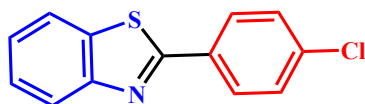
Section S6. NMR data of products



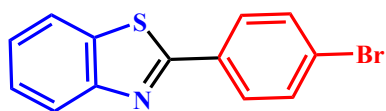
2-phenylbenzo[d]thiazole (3a). White solid (40.6 mg, 96%). ^1H NMR (400 MHz, CDCl_3): δ 8.08 (d, J = 8.1 Hz, 3H), 7.91 (d, J = 8.0 Hz, 1H), 7.50 (s, 4H), 7.39 (t, J = 7.6 Hz, 1H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 168.06, 154.19, 135.11, 133.67, 130.97, 129.03, 127.58, 126.33, 125.20, 123.27, 121.63 ppm.



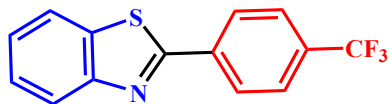
2-(4-fluorophenyl)benzo[d]thiazole (3b). White solid (39.1 mg, 85%). ^1H NMR (400 MHz, CDCl_3): δ 8.25–8.01 (m, 3H), 7.90 (d, J = 7.9 Hz, 1H), 7.50 (t, J = 7.3 Hz, 1H), 7.39 (t, J = 7.6 Hz, 1H), 7.18 (t, J = 8.6 Hz, 2H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 166.76, 165.71, 163.21, 154.10, 135.05, 129.96 (d, J = 3.2 Hz), 129.53 (d, J = 8.7 Hz), 126.43, 125.33, 123.20, 121.63, 116.29 (d, J = 8.7 Hz) ppm.



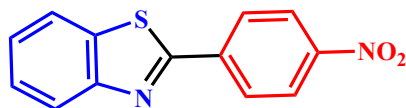
2-(4-chlorophenyl)benzo[d]thiazole (3c). White solid (42.6 mg, 87%). ^1H NMR (400 MHz, CDCl_3): δ 8.07 (d, J = 8.1 Hz, 1H), 8.02 (d, J = 8.6 Hz, 2H), 7.90 (d, J = 8.0 Hz, 1H), 7.54–7.48 (m, 1H), 7.47 (d, J = 8.6 Hz, 2H), 7.40 (t, J = 8.1 Hz, 1H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 166.63, 154.06, 137.04, 135.05, 132.11, 129.28, 128.71, 126.50, 125.43, 123.31, 121.67 ppm.



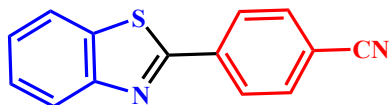
2-(4-bromophenyl)benzo[d]thiazole (3d). Yellow solid (52.0 mg, 90%). ^1H NMR (400 MHz, CDCl_3): δ 8.07 (d, J = 8.1 Hz, 1H), 7.96 (d, J = 8.4 Hz, 2H), 7.91 (d, J = 7.9 Hz, 1H), 7.63 (d, J = 8.3 Hz, 2H), 7.51 (t, J = 7.6 Hz, 1H), 7.41 (t, J = 7.5 Hz, 1H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 166.71, 154.07, 135.04, 132.55, 132.25, 128.92, 126.52, 125.47, 125.44, 123.32, 121.68 ppm.



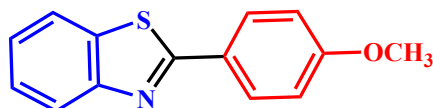
2-(4-(trifluoromethyl)phenyl)benzo[d]thiazole (3e). White solid (49.7 mg, 89%). ^1H NMR (400 MHz, CDCl_3): δ 8.20 (d, J = 8.1 Hz, 2H), 8.11 (d, J = 8.1 Hz, 1H), 7.93 (d, J = 7.9 Hz, 1H), 7.75 (d, J = 8.2 Hz, 2H), 7.53 (t, J = 8.2 Hz, 1H), 7.43 (t, J = 8.1 Hz, 1H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 166.05, 154.03, 136.75, 135.20, 132.60, 132.28, 127.77, 126.67, 126.01 (q, J = 3.8 Hz), 125.80, 123.63, 121.76 ppm.



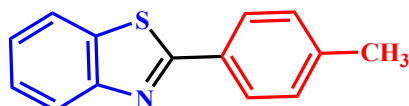
2-(4-nitrophenyl)benzo[d]thiazole (3f). Yellow solid (46 mg, 90%). ^1H NMR (400 MHz, CDCl_3): δ 8.36 (d, J = 8.8 Hz, 2H), 8.27 (d, J = 8.8 Hz, 2H), 8.14 (d, J = 8.2 Hz, 1H), 7.97 (d, J = 8.0 Hz, 1H), 7.56 (t, J = 8.1 Hz, 1H), 7.47 (t, J = 7.6 Hz, 1H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 164.85, 154.08, 149.00, 139.17, 135.5, 128.24, 126.94, 126.24, 124.34, 123.93, 121.86 ppm.



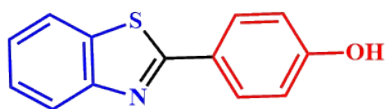
4-(benzo[d]thiazol-2-yl)benzonitrile (3g). White solid (44.9 mg, 95%). ^1H NMR (400 MHz, CDCl_3): δ 8.21 (d, J = 8.5 Hz, 2H), 8.12 (d, J = 8.1 Hz, 1H), 7.95 (d, J = 7.9 Hz, 1H), 7.79 (d, J = 8.5 Hz, 2H), 7.55 (t, J = 8.3 Hz, 1H), 7.46 (t, J = 8.1 Hz, 1H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 165.35, 154.03, 137.51, 135.32, 132.80, 127.95, 126.84, 126.09, 123.82, 121.82, 118.30, 114.14 ppm.



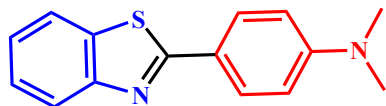
2-(4-methoxyphenyl)benzo[d]thiazole (3h). White solid (43.4 mg, 90%). ^1H NMR (400 MHz, CDCl_3): δ 8.11–7.99 (m, 3H), 7.88 (d, J = 7.9 Hz, 1H), 7.47 (t, J = 8.2 Hz, 1H), 7.35 (t, J = 8.1 Hz, 1H), 7.00 (d, J = 8.8 Hz, 2H), 3.88 (s, 3H, OCH_3) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 167.86, 161.92, 154.23, 134.87, 129.12, 126.45, 126.21, 124.79, 122.83, 121.51, 55.48 (OCH_3) ppm.



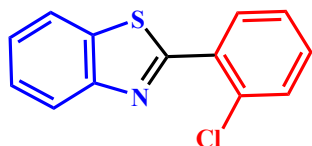
2-(p-tolyl)benzo[d]thiazole (3i). Yellow solid (41.4 mg, 92%). ^1H NMR (400 MHz, CDCl_3): δ 8.06 (d, J = 8.2 Hz, 1H), 7.99 (d, J = 8.1 Hz, 2H), 7.89 (d, J = 7.9 Hz, 1H), 7.48 (t, J = 8.2 Hz, 1H), 7.37 (t, J = 8.1 Hz, 1H), 7.30 (d, J = 8.0 Hz, 2H), 2.43 (s, 3H, CH_3) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 168.26, 154.16, 141.45, 134.94, 130.95, 129.73, 127.49, 126.26, 125.01, 123.05, 121.58, 21.56 (CH_3) ppm.



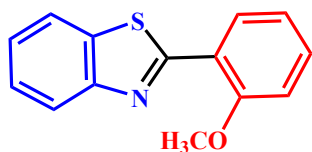
2-(4-hydroxyphenyl)benzo[d]thiazole (3j). Yellow solid (38.6 mg, 85%). ^1H NMR (400 MHz, $\text{DMSO}-d_6$): δ 10.25 (s, 1H), 8.09 (d, J = 7.9 Hz, 1H), 7.99 (d, J = 8.1 Hz, 1H), 7.94 (d, J = 8.6 Hz, 2H), 7.51 (t, J = 7.6 Hz, 1H), 7.39 (s, 1H), 6.96 (s, 2H) ppm. ^{13}C NMR (101 MHz, $\text{DMSO}-d_6$): δ 167.91, 160.99, 154.18, 134.56, 129.51, 126.89, 125.36, 124.49, 122.76, 122.58, 116.54 ppm.



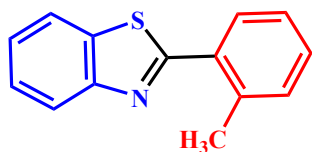
4-(benzo[d]thiazol-2-yl)-N,N-dimethylaniline (3k). Pale yellow solid (44.2 mg, 87%). ^1H NMR (400 MHz, CDCl_3): δ 7.98 (t, J = 8.0 Hz, 3H), 7.84 (d, J = 7.9 Hz, 1H), 7.44 (t, J = 7.7 Hz, 1H), 7.31 (t, J = 7.6 Hz, 1H), 6.75 (d, J = 8.9 Hz, 1H), 3.06 (s, 6H, $\text{N}(\text{CH}_3)_2$) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 168.84, 154.35, 152.18, 134.50, 128.88, 126.00, 124.20, 122.25, 121.36, 111.70, 40.20 ($\text{N}(\text{CH}_3)_2$) ppm.



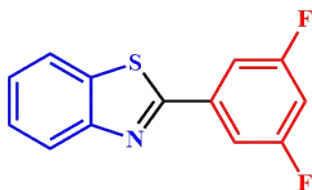
2-(2-chlorophenyl)benzo[d]thiazole (3l). White solid (39.2 mg, 79%). ^1H NMR (400 MHz, CDCl_3): δ 8.30–8.18 (m, 1H), 8.15 (d, J = 8.2 Hz, 1H), 7.95 (d, J = 8.0 Hz, 1H), 7.53 (t, J = 7.6 Hz, 2H), 7.47–7.35 (m, 3H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 164.17, 152.54, 136.14, 132.74, 132.31, 131.78, 131.14, 130.82, 127.11, 126.30, 125.45, 123.49, 121.40 ppm.



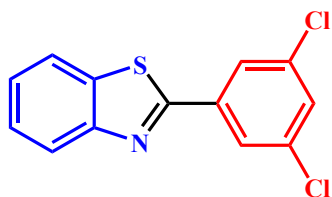
2-(2-methoxyphenyl)benzo[d]thiazole (3m). White solid (41.9 mg, 87%). ^1H NMR (400 MHz, CDCl_3): δ 8.53 (d, J = 9.5 Hz, 1H), 8.09 (d, J = 8.2 Hz, 1H), 7.92 (d, J = 7.9 Hz, 1H), 7.56–7.42 (m, 2H), 7.36 (t, J = 8.0 Hz, 1H), 7.13 (t, J = 7.6 Hz, 1H), 7.05 (d, J = 8.3 Hz, 1H), 4.04 (s, 3H, OCH_3) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 163.17, 157.24, 152.15, 136.12, 131.80, 129.55, 125.91, 124.60, 122.53, 122.29, 121.22, 121.18, 111.69, 55.72 (OCH_3) ppm.



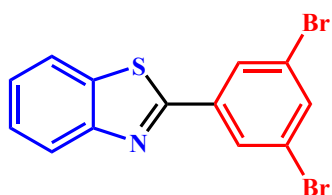
2-(o-tolyl)benzo[d]thiazole (3n). White solid (39.3 mg, 85%). ^1H NMR (400 MHz, CDCl_3): δ 8.11 (d, J = 8.2 Hz, 1H), 7.93 (d, J = 8.0 Hz, 1H), 7.76 (d, J = 7.6 Hz, 1H), 7.51 (t, J = 7.4 Hz, 1H), 7.41 (t, J = 7.6 Hz, 1H), 7.34 (dt, J = 14.8, 6.9 Hz, 3H), 2.66 (s, 3H, CH_3) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 168.01, 153.80, 137.26, 135.61, 133.10, 131.55, 130.55, 130.02, 126.14, 126.12, 125.10, 123.39, 121.38, 21.37 (CH_3) ppm.



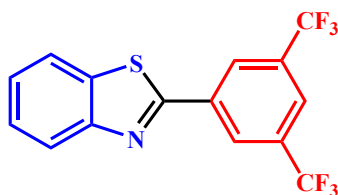
2-(3,5-difluorophenyl)benzo[d]thiazole (3o). White solid (44.5 mg, 90%). ^1H NMR (400 MHz, CDCl_3): δ 8.20–8.01 (m, 3H), 7.90 (d, J = 7.9 Hz, 1H), 7.50 (t, J = 7.6 Hz, 1H), 7.39 (t, J = 7.6 Hz, 1H), 7.18 (t, J = 8.3 Hz, 1H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 164.45, 153.83, 136.73, 136.05, 135.13, 129.05, 126.79, 125.95, 123.62 (d, J = 5.5 Hz), 121.79 ppm.



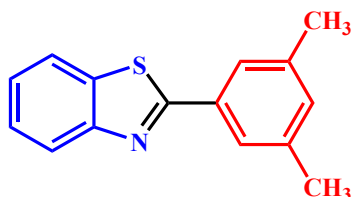
2-(3,5-dichlorophenyl)benzo[d]thiazole (3p). White solid (54.12 mg, 97%). ^1H NMR (400 MHz, CDCl_3): δ 8.09 (d, J = 8.2 Hz, 1H), 7.98 (d, J = 1.9 Hz, 2H), 7.93 (d, J = 8.0 Hz, 1H), 7.59–7.51 (m, 1H), 7.47 (t, J = 1.9 Hz, 1H), 7.45–7.41 (m, 1H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 164.69, 153.83, 136.27, 135.77, 135.11, 130.55, 126.76, 125.93, 125.76, 123.66, 121.78 ppm.



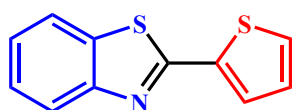
2-(3,5-dibromophenyl)benzo[d]thiazole (3q). White solid (68.9mg, 94%). ^1H NMR (400 MHz, CDCl_3): δ 8.18 (s, 2H), 8.09 (d, J = 8.1 Hz, 1H), 7.93 (d, J = 7.9 Hz, 1H), 7.77 (t, J = 1.7 Hz, 1H), 7.54 (t, J = 7.1 Hz, 1H), 7.44 (t, J = 7.1 Hz, 1H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 164.42, 153.80, 136.72, 136.05, 135.12, 129.04, 126.79, 125.95, 123.64, 123.59, 121.79 ppm.



2-(3,5-bis(trifluoromethyl)phenyl)benzo[d]thiazole (3r). White solid (66.6 mg, 96%). ^1H NMR (400 MHz, CDCl_3): δ 8.54 (s, 2H), 8.14 (d, J = 8.1 Hz, 1H), 8.01–7.92 (m, 2H), 7.57 (t, J = 7.5 Hz, 1H), 7.48 (t, J = 7.5 Hz, 1H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 164.12, 153.86, 135.63, 135.15, 133.11, 132.77, 132.44, 127.36 (d, J = 3.4 Hz), 127.00, 126.28, 124.35, 124.17–123.95 (m), 123.87, 121.89, 121.64 ppm.



2-(3,5-dimethylphenyl)benzo[d]thiazole (3s). White solid (43.1mg, 90%). ^1H NMR (400 MHz, CDCl_3): δ 8.07 (d, J = 8.1 Hz, 1H), 7.90 (d, J = 8.0 Hz, 1H), 7.71 (s, 2H), 7.49 (t, J = 7.6 Hz, 1H), 7.38 (t, J = 7.5 Hz, 1H), 7.13 (s, 1H), 2.41 (s, $2 \times 3\text{H}$, CH_3) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 168.59, 154.11, 138.74, 135.00, 133.44, 132.79, 126.27, 125.36, 125.07, 123.11, 121.60, 21.27 (CH_3) ppm.

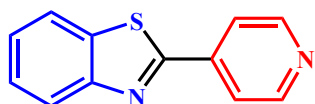


2-(thiophen-2-yl)benzo[d]thiazole (3t). White solid (41.7 mg, 96%). ^1H NMR (400 MHz, CDCl_3): δ 8.03

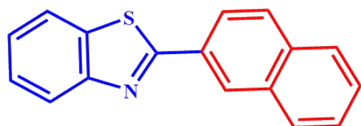
(d, $J = 8.2$ Hz, 1H), 7.86 (d, $J = 8.4$ Hz, 1H), 7.67 (d, $J = 3.7$ Hz, 1H), 7.49 (dd, $J = 17.9, 7.1$ Hz, 2H), 7.37 (t, $J = 7.6$ Hz, 1H), 7.17–7.12 (m, 1H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 161.39, 153.69, 137.34, 134.69, 129.30, 128.62, 128.05, 126.43, 125.23, 122.98, 121.46 ppm.



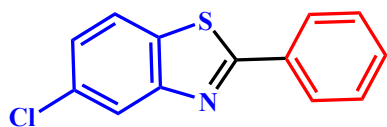
2-(furan-2-yl)benzo[d]thiazole (3u). White solid (36.2 mg, 90%). ^1H NMR (400 MHz, CDCl_3): δ 8.05 (d, $J = 8.1$ Hz, 1H), 7.90 (d, $J = 7.9$ Hz, 1H), 7.61 (s, 1H), 7.50 (t, $J = 8.2$ Hz, 1H), 7.39 (t, $J = 7.6$ Hz, 1H), 7.20 (d, $J = 3.5$ Hz, 1H), 6.61 (s, 1H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 157.57, 153.75, 148.74, 144.72, 134.27, 126.49, 125.21, 123.13, 121.59, 112.55, 111.45 ppm.



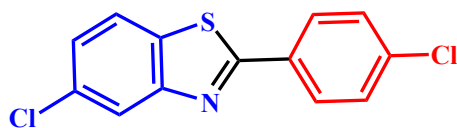
2-(pyridin-4-yl)benzo[d]thiazole (3v). White solid (36.0 mg, 85%). ^1H NMR (400 MHz, CDCl_3): δ 8.78 (d, $J = 4.9$ Hz, 2H), 8.13 (d, $J = 8.2$ Hz, 1H), 8.00–7.89 (m, 3H), 7.55 (t, $J = 7.6$ Hz, 1H), 7.46 (t, $J = 7.6$ Hz, 1H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 165.10, 153.95, 150.78, 140.44, 135.20, 126.83, 126.21, 123.91, 121.88, 121.19 ppm.



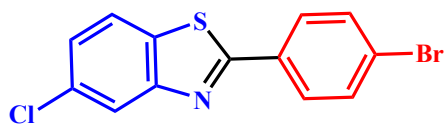
2-(naphthalen-2-yl)benzo[d]thiazole (3w). White solid (41.8 mg, 79%). ^1H NMR (400 MHz, CDCl_3): δ 8.55 (s, 1H), 8.21 (d, $J = 8.5$ Hz, 1H), 8.14 (d, $J = 7.8$ Hz, 1H), 8.04–7.83 (m, 4H), 7.66–7.48 (m, 3H), 7.39 (t, $J = 7.5$ Hz, 1H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 168.12, 154.25, 135.16, 134.62, 133.19, 130.98, 128.85, 128.83, 127.89, 127.60, 127.47, 126.89, 126.41, 125.26, 124.45, 123.26, 121.67 ppm.



5-chloro-2-phenylbenzo[d]thiazole (4a). White solid (45.6 mg, 95%). ^1H NMR (400 MHz, CDCl_3): δ 8.06 (dd, $J = 6.8, 3.3$ Hz, 3H), 7.79 (d, $J = 8.5$ Hz, 1H), 7.55–7.45 (m, 3H), 7.35 (dd, $J = 8.5, 1.9$ Hz, 1H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 169.93, 154.98, 133.31, 133.24, 132.32, 131.36, 129.11, 127.61, 125.66, 123.03, 122.32 ppm.

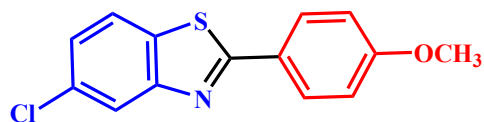


5-chloro-2-(4-chlorophenyl)benzo[d]thiazole (4b). White solid (47.4 mg, 85%). ^1H NMR (400 MHz, CDCl_3): δ 8.11–7.93 (m, 3H), 7.81 (d, $J = 8.5$ Hz, 1H), 7.47 (d, $J = 8.5$ Hz, 2H), 7.37 (d, $J = 8.5$ Hz, 1H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 168.42, 154.86, 137.44, 133.26, 132.49, 131.68, 129.34, 128.72, 125.86, 123.07, 122.32 ppm.

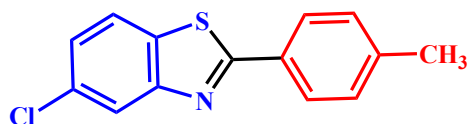


2-(4-bromophenyl)-5-chlorobenzo[d]thiazole (4c). White solid (52.3 mg, 81%). ^1H NMR (400 MHz, CDCl_3): δ 8.05 (s, 1H), 7.95 (d, $J = 8.4$ Hz, 2H), 7.82 (d, $J = 8.5$ Hz, 1H), 7.64 (d, $J = 8.3$ Hz, 2H), 7.38 (d, $J = 10.5$ Hz, 1H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 168.54, 154.90, 133.27, 132.53, 132.33, 132.16, 128.94,

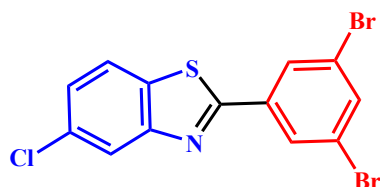
125.93, 125.88, 123.11, 122.36 ppm.



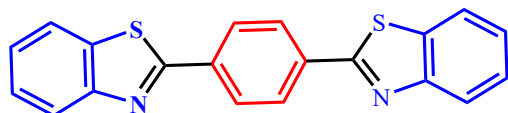
5-chloro-2-(4-methoxyphenyl)benzo[d]thiazole (4d). White solid (47.9 mg, 87%). ^1H NMR (400 MHz, CDCl_3): δ 8.00 (d, $J = 8.5$ Hz, 3H), 7.76 (d, $J = 8.5$ Hz, 1H), 7.32 (d, $J = 8.5$ Hz, 1H), 6.99 (d, $J = 8.7$ Hz, 2H), 3.88 (s, 3H, OCH_3) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 169.70, 162.19, 155.08, 133.10, 132.15, 129.19, 126.01, 125.17, 122.60, 122.17, 114.42, 55.49 (OCH_3) ppm.



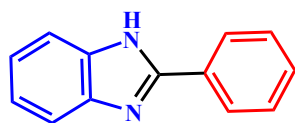
5-chloro-2-(p-tolyl)benzo[d]thiazole (4e). White solid (42.1 mg, 81%). ^1H NMR (400 MHz, CDCl_3): δ 8.08 (s, 1H), 7.82 (d, $J = 8.5$ Hz, 1H), 7.75 (d, $J = 7.6$ Hz, 1H), 7.45–7.27 (m, 4H), 2.65 (s, 3H, CH_3) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 169.95, 154.64, 137.38, 133.79, 132.64, 132.15, 131.69, 130.44 (d, $J = 20.7$ Hz), 130.33, 126.21, 125.58, 123.17, 122.08, 21.45 (CH_3) ppm.



5-chloro-2-(3,5-dibromophenyl)benzo[d]thiazole (4f). White solid (74.6 mg, 93%). ^1H NMR (400 MHz, CDCl_3): δ 8.12 (d, $J = 1.6$ Hz, 2H), 8.05 (d, $J = 1.9$ Hz, 1H), 7.82 (d, $J = 8.6$ Hz, 1H), 7.77 (s, 1H), 7.40 (d, $J = 8.6$ Hz, 1H) ppm.

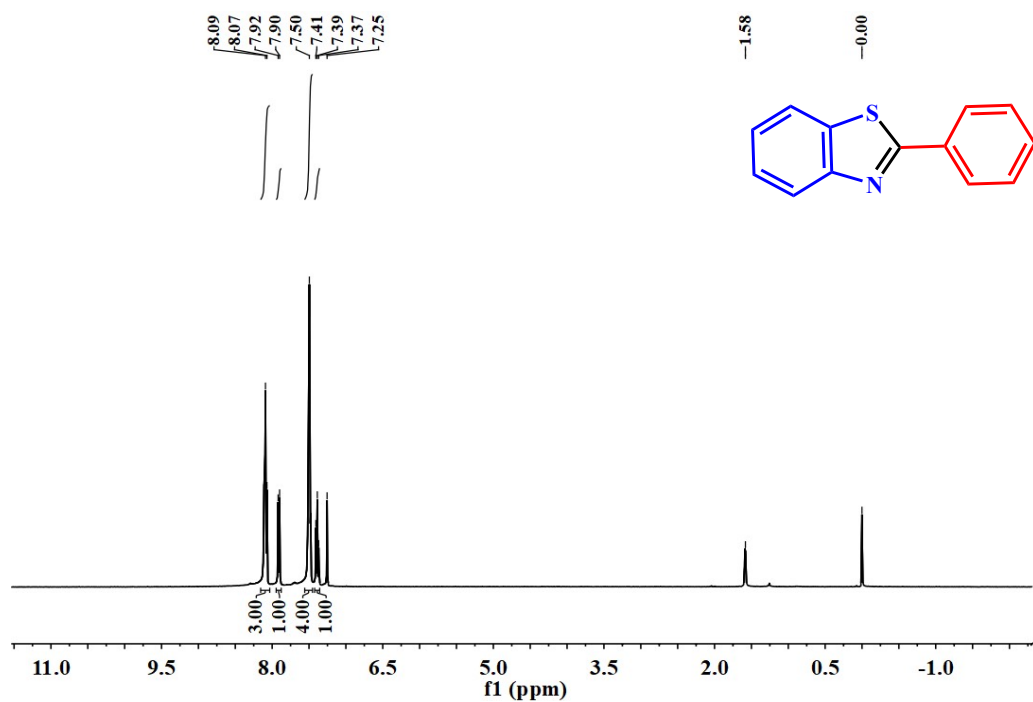


1,4-bis(benzo[d]thiazol-2-yl)benzene (4g). White solid (58.5 mg, 85%). ^1H NMR (400 MHz, CDCl_3): δ 8.24 (s, 4H), 8.12 (d, $J = 8.2$ Hz, 2H), 7.95 (d, $J = 8.0$ Hz, 2H), 7.53 (t, $J = 7.6$ Hz, 2H), 7.43 (t, $J = 7.5$ Hz, 2H) ppm.

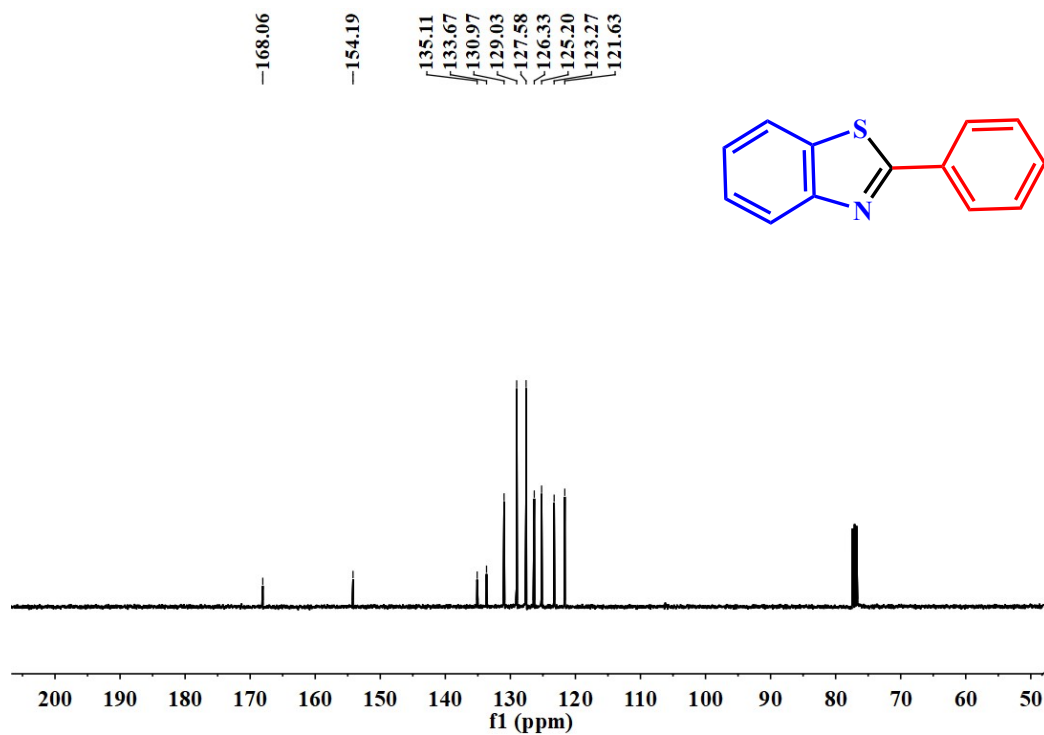


2-phenyl-1H-benzo[d]imidazole. White solid (38.8 mg, 85%). ^1H NMR (400 MHz, $\text{DMSO}-d_6$): δ 12.93 (s, 1H), 8.19 (d, $J = 7.2$ Hz, 2H), 7.68 (d, $J = 7.5$ Hz, 1H), 7.62–7.42 (m, 4H), 7.34–7.13 (m, 2H) ppm. ^{13}C NMR (101 MHz, $\text{DMSO}-d_6$): δ 151.68, 144.42, 135.65, 130.61, 130.31, 129.42, 126.89, 122.98, 122.16, 119.25, 111.78 ppm.

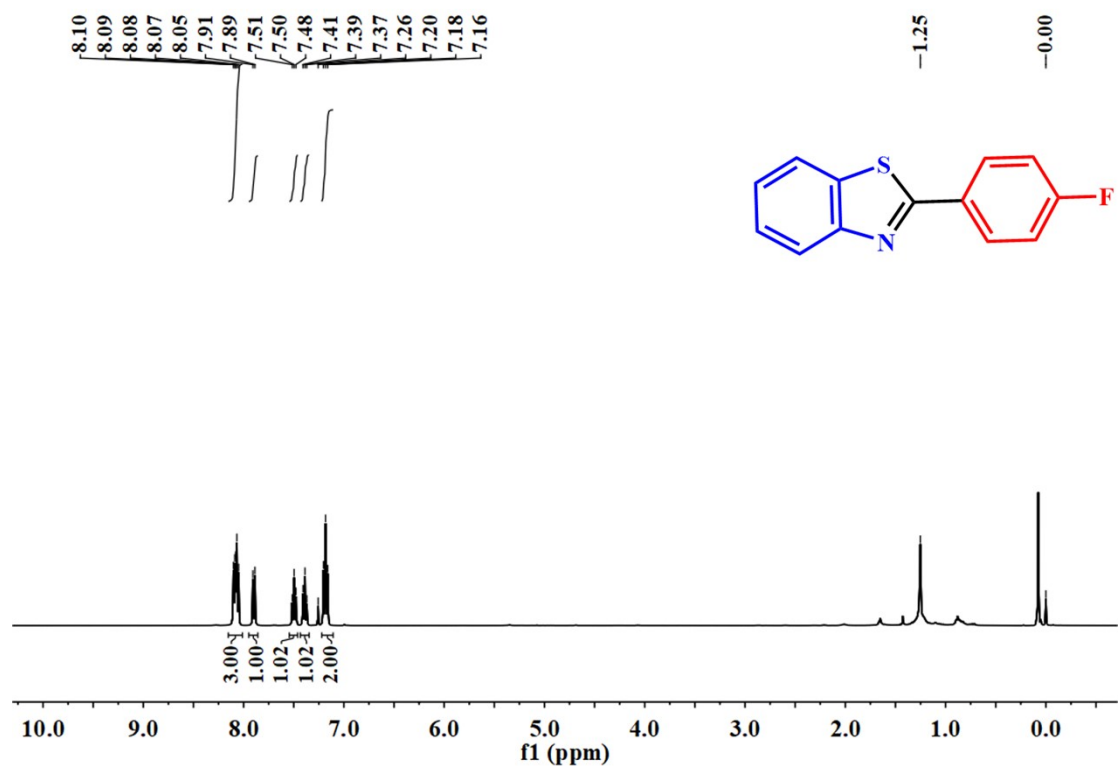
Section S7. ^1H and ^{13}C NMR spectra



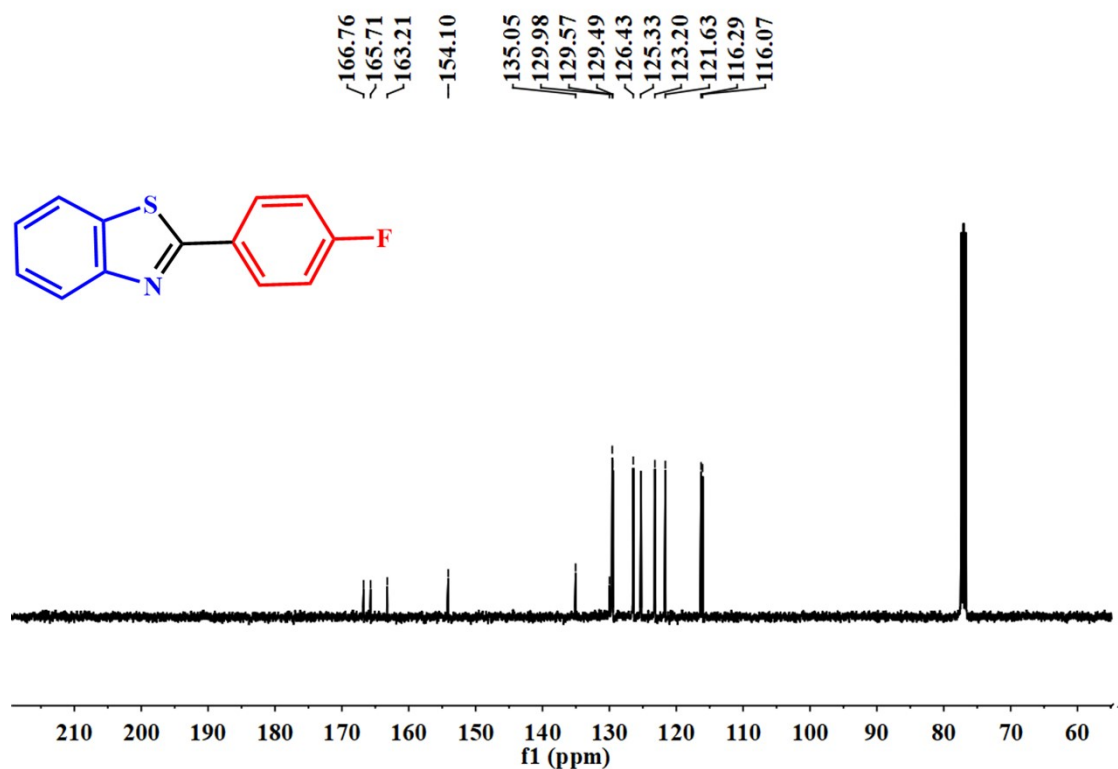
The ¹H-NMR spectrum of 2-phenylbenzo[d]thiazole (**3a**).



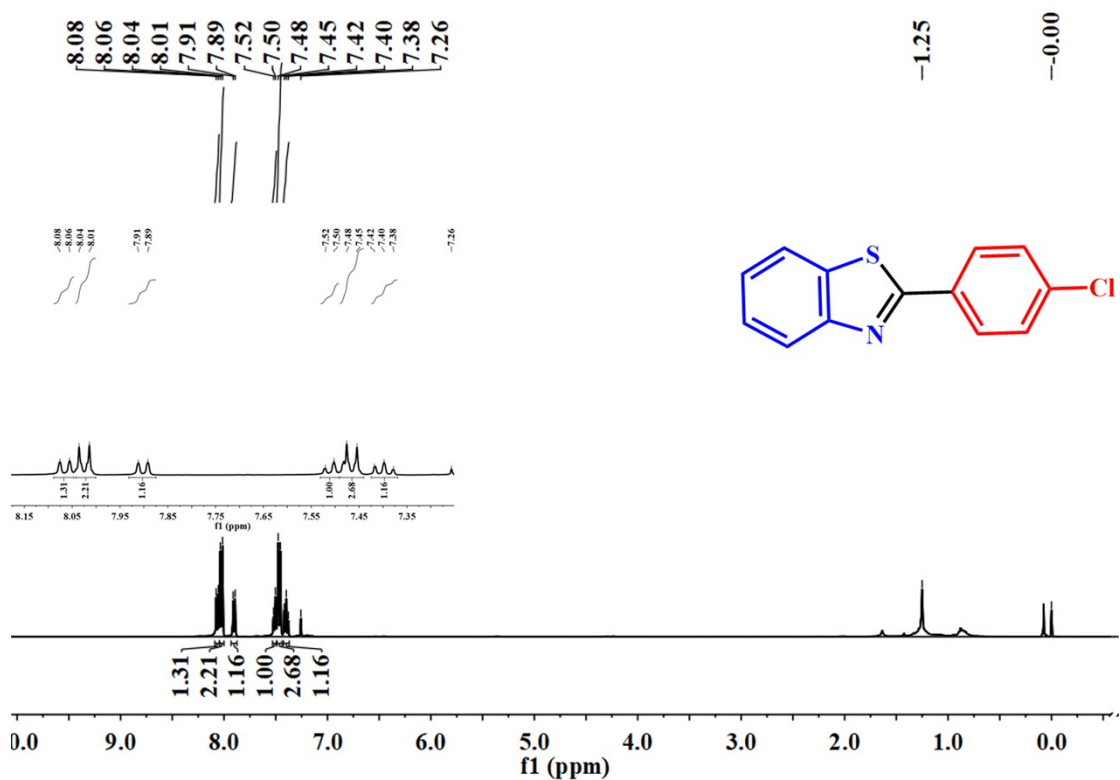
The ¹³C NMR spectrum of 2-phenylbenzo[d]thiazole (**3a**).



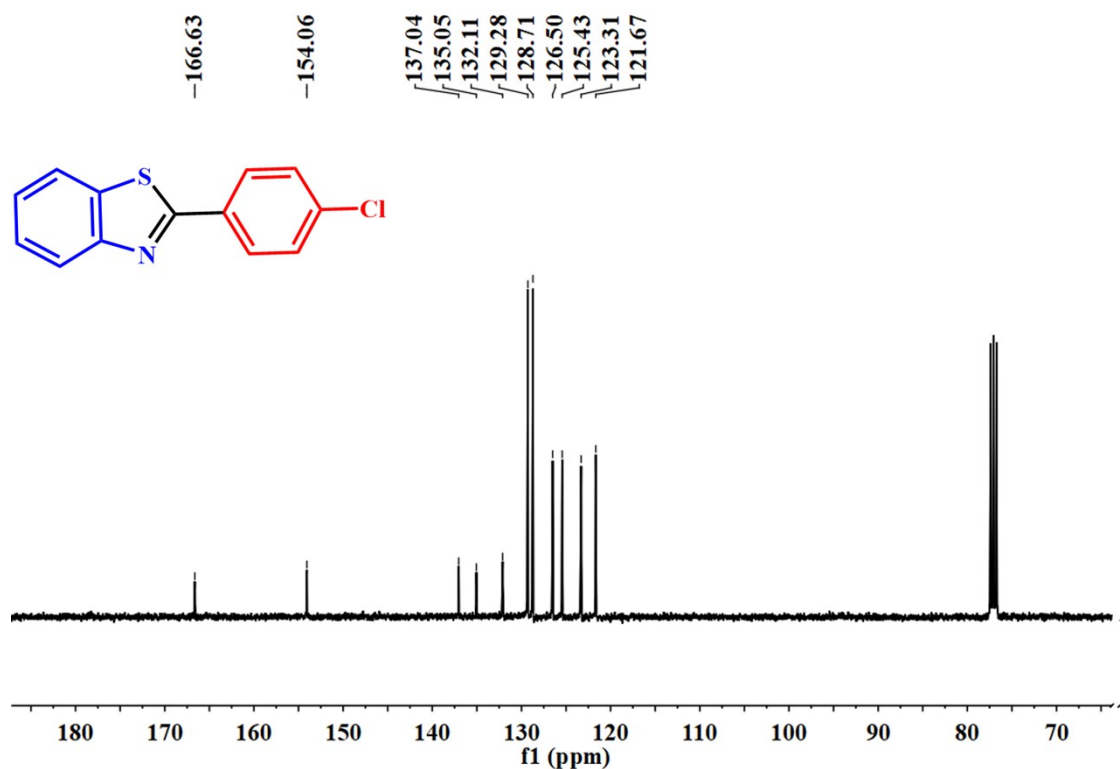
The ¹H-NMR spectrum of 2-(4-fluorophenyl)benzo[d]thiazole (**3b**).



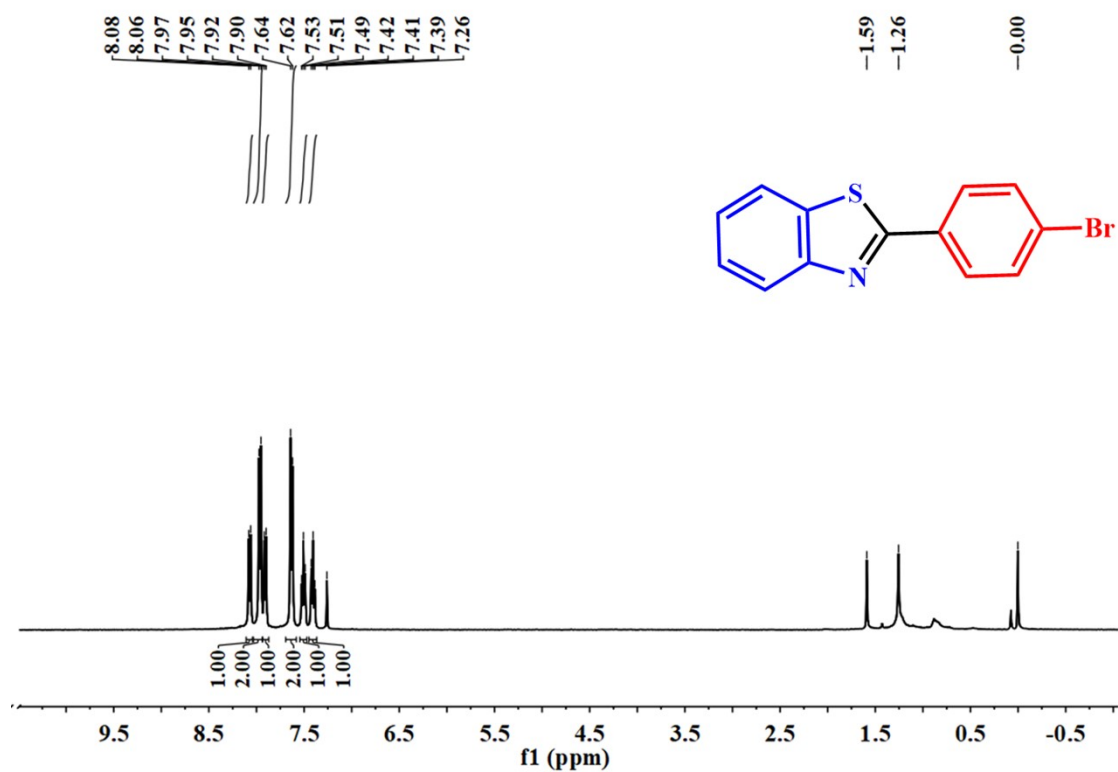
The ¹³C-NMR spectrum of 2-(4-fluorophenyl)benzo[d]thiazole (**3b**).



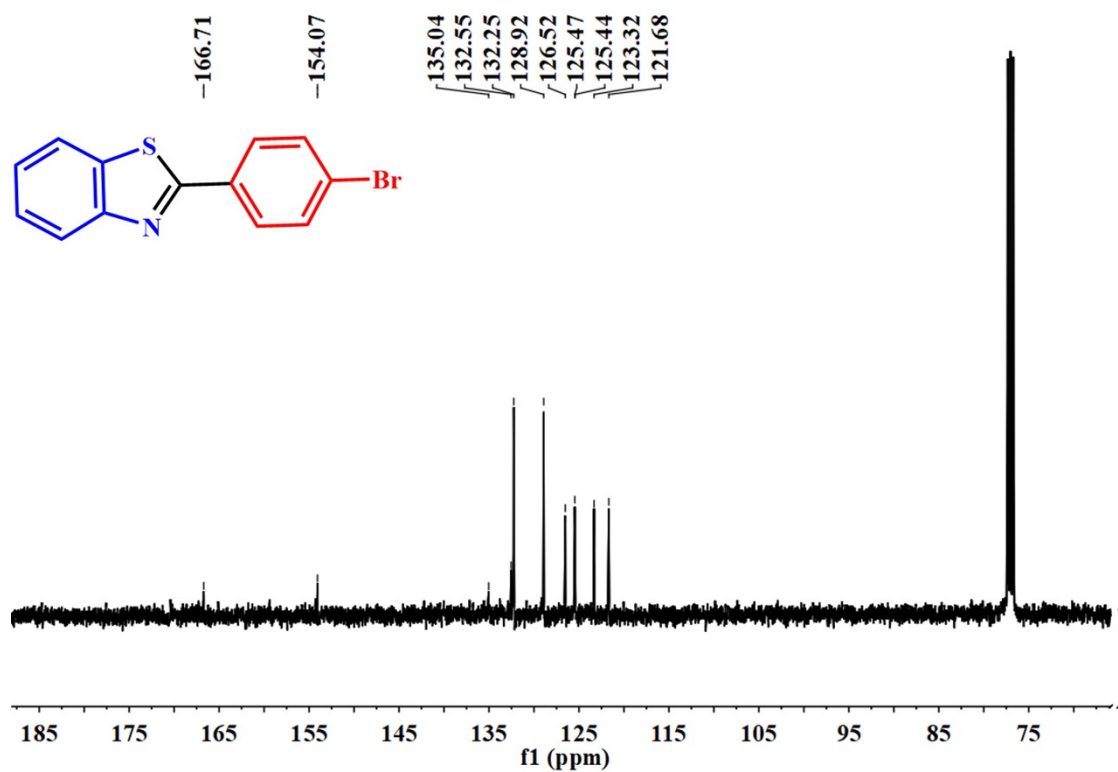
The ¹H-NMR spectrum of 2-(4-chlorophenyl)benzo[d]thiazole (3c).



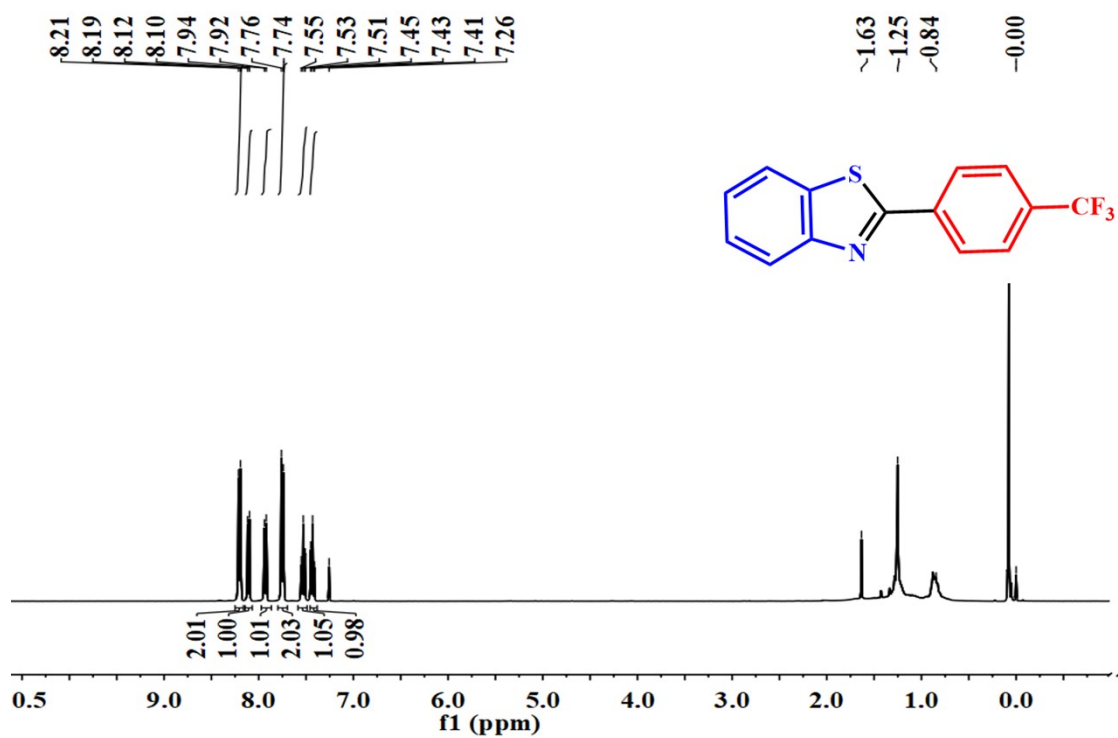
The ¹³C-NMR spectrum of 2-(4-chlorophenyl)benzo[d]thiazole (3c).



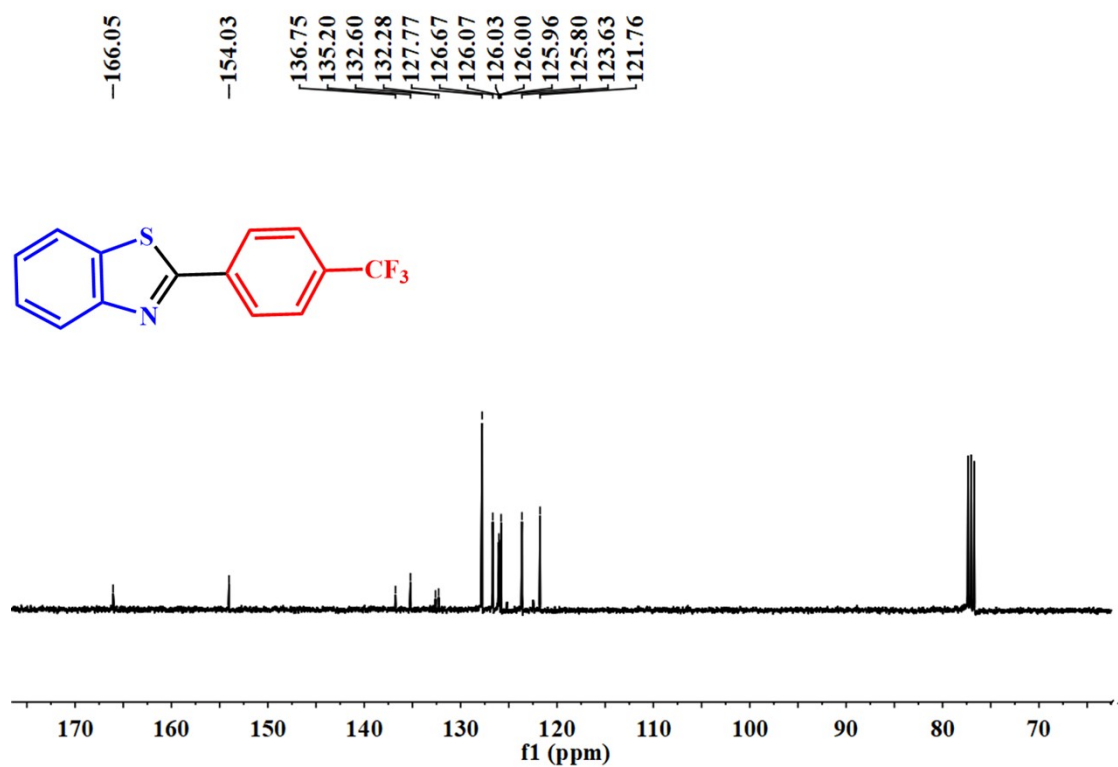
The ¹H-NMR spectrum of 2-(4-bromophenyl)benzo[d]thiazole (3d).



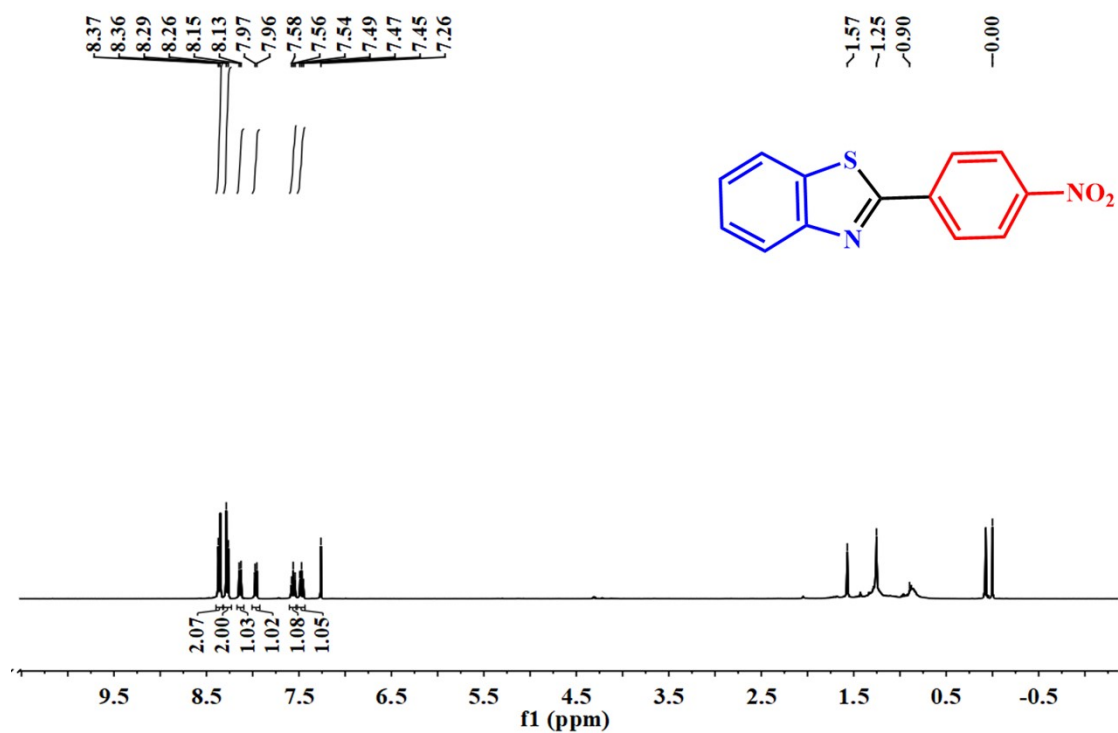
The ¹³C-NMR spectrum of 2-(4-bromophenyl)benzo[d]thiazole (3d).



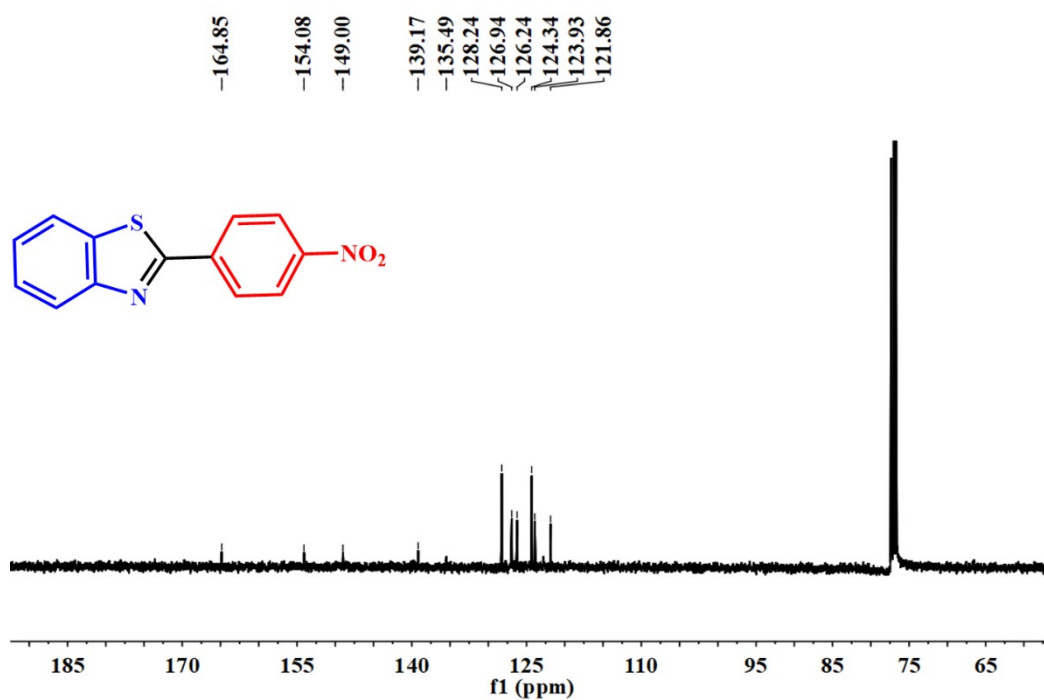
The ¹H-NMR spectrum of 2-(4-(trifluoromethyl)phenyl)benzo[d]thiazole (**3e**).



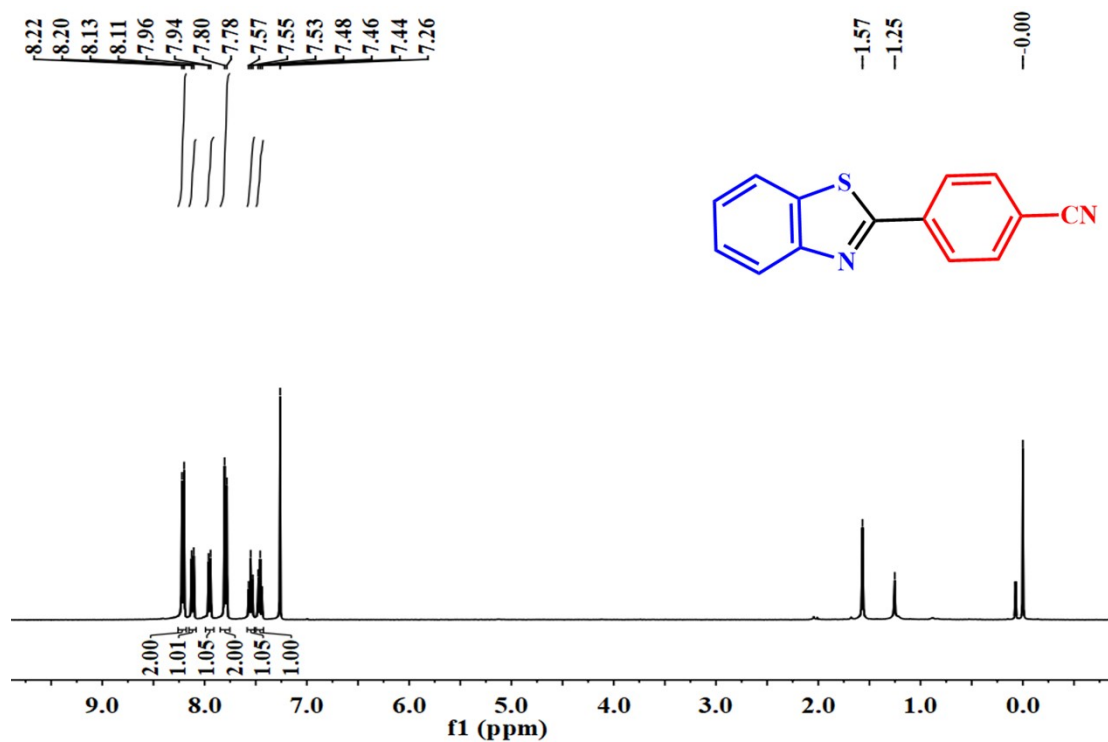
The ¹³C-NMR spectrum of 2-(4-(trifluoromethyl)phenyl)benzo[d]thiazole (**3e**).



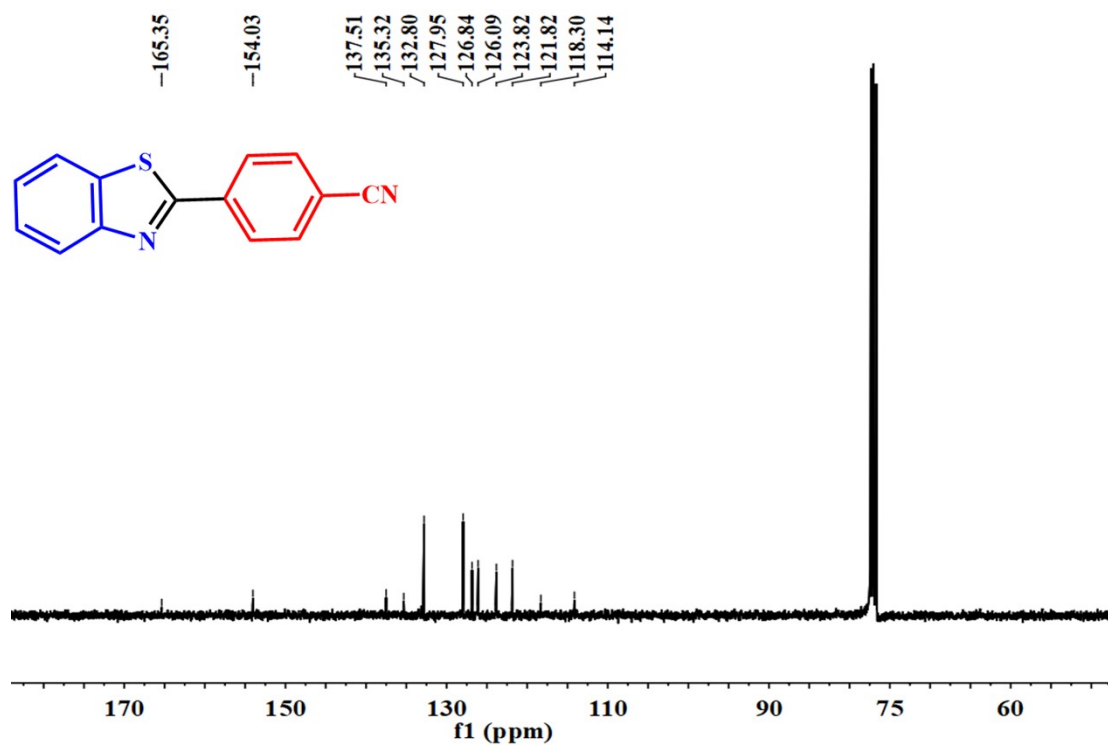
The ¹H-NMR spectrum of 2-(4-nitrophenyl)benzo[d]thiazole (3f).



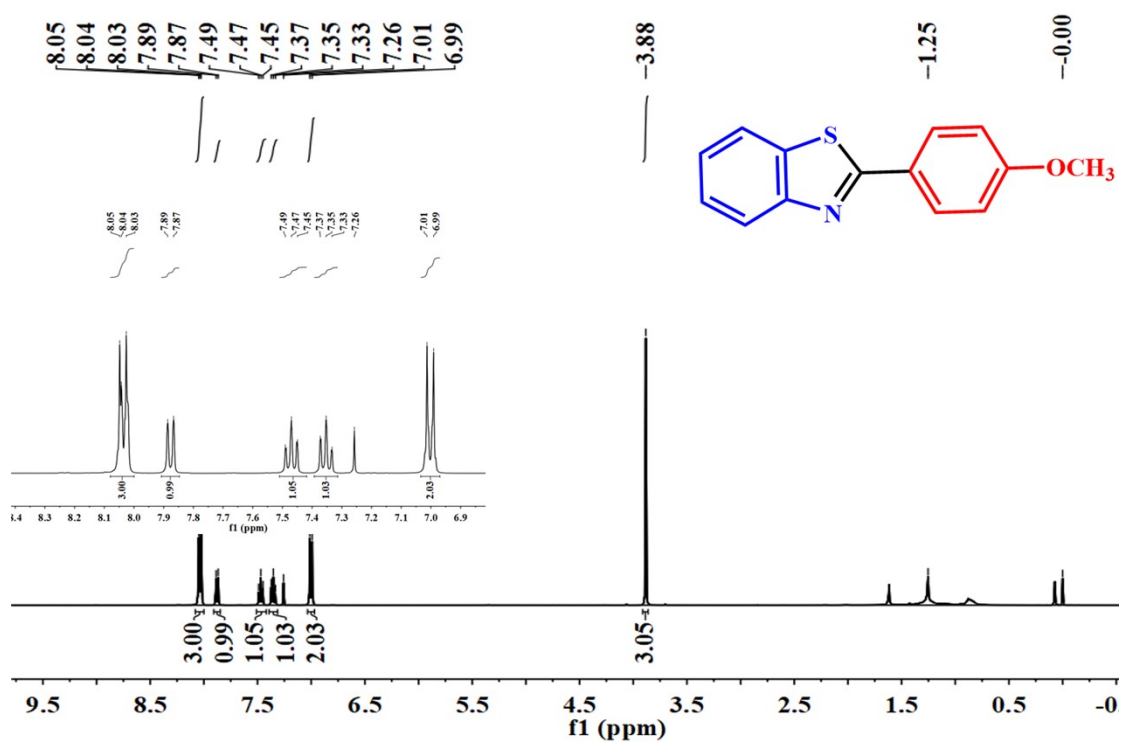
The ¹³C-NMR spectrum of 2-(4-nitrophenyl)benzo[d]thiazole (3f).



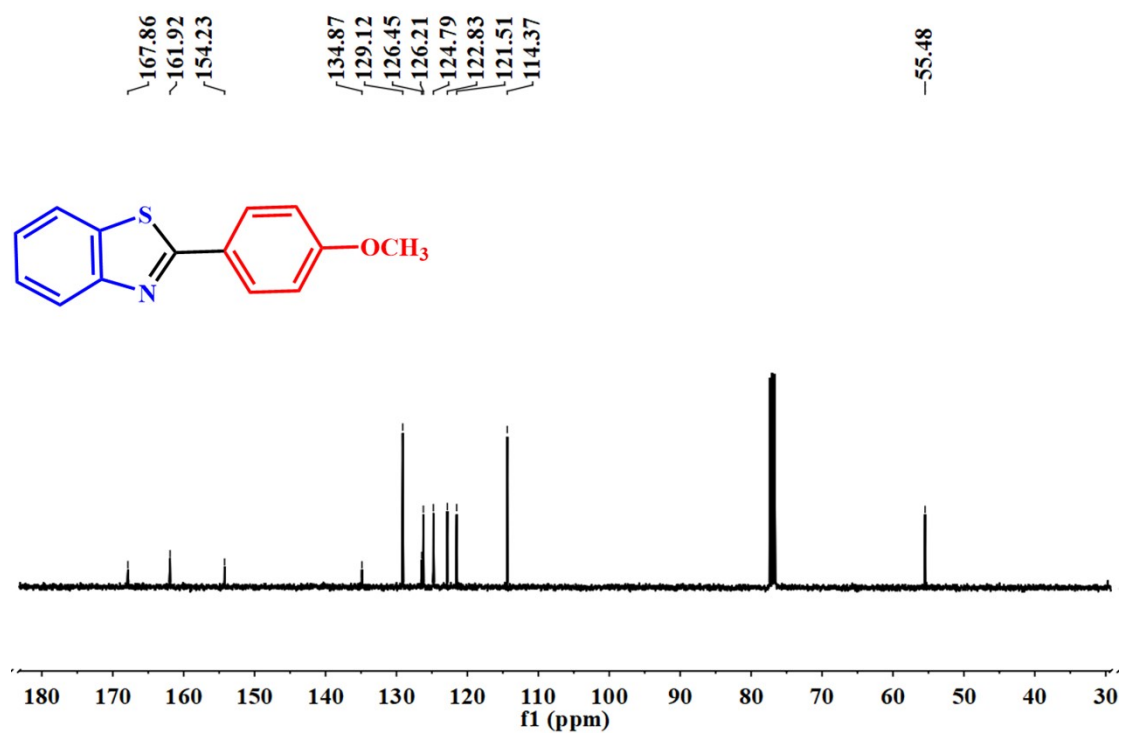
The ¹H-NMR spectrum of 4-(benzo[d]thiazol-2-yl)benzonitrile (**3g**).



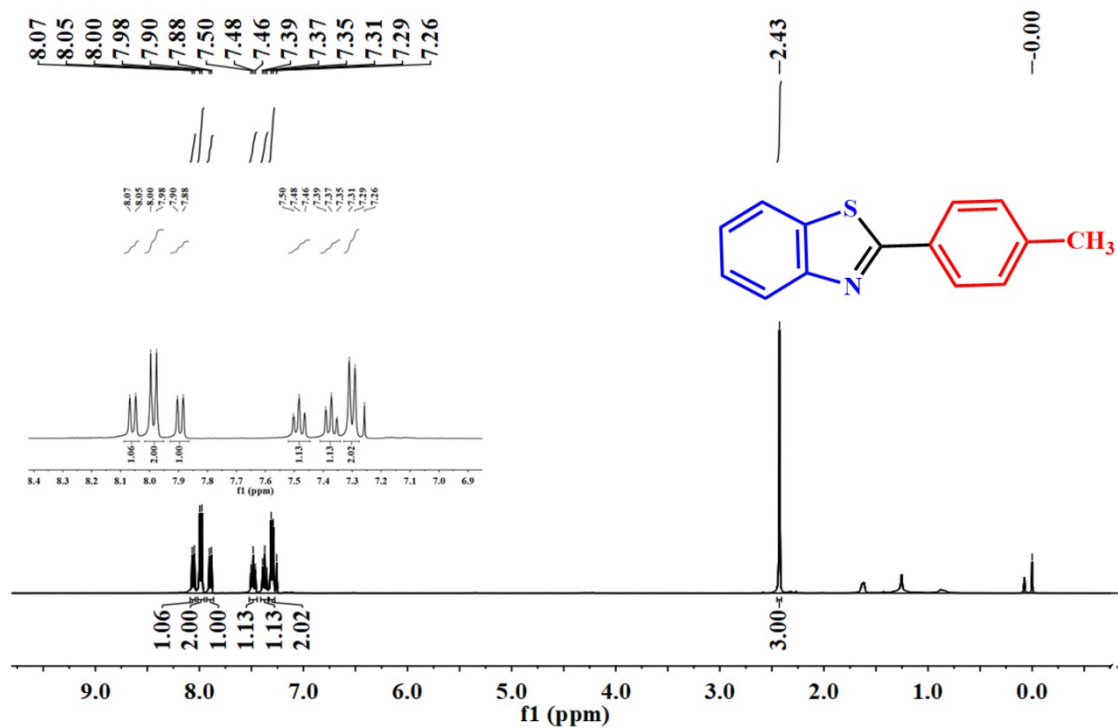
The ¹³C-NMR spectrum of 4-(benzo[d]thiazol-2-yl)benzonitrile (**3g**).



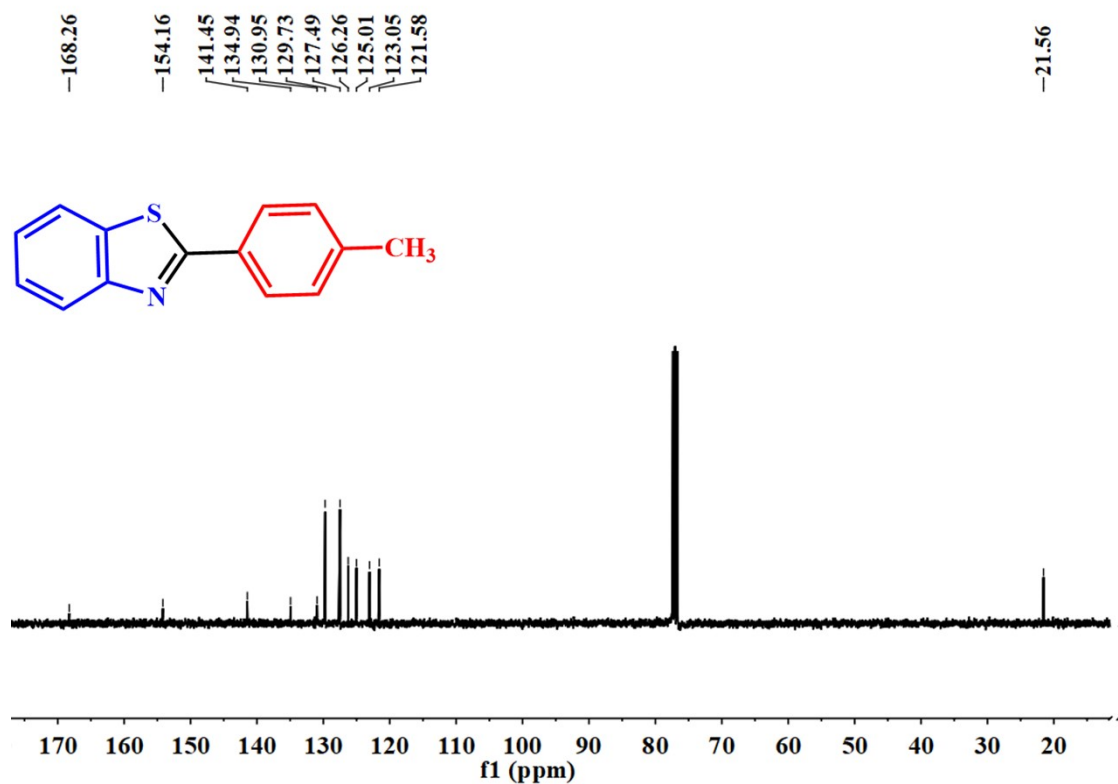
The ¹H-NMR spectrum of 2-(4-methoxyphenyl)benzo[d]thiazole (**3h**).



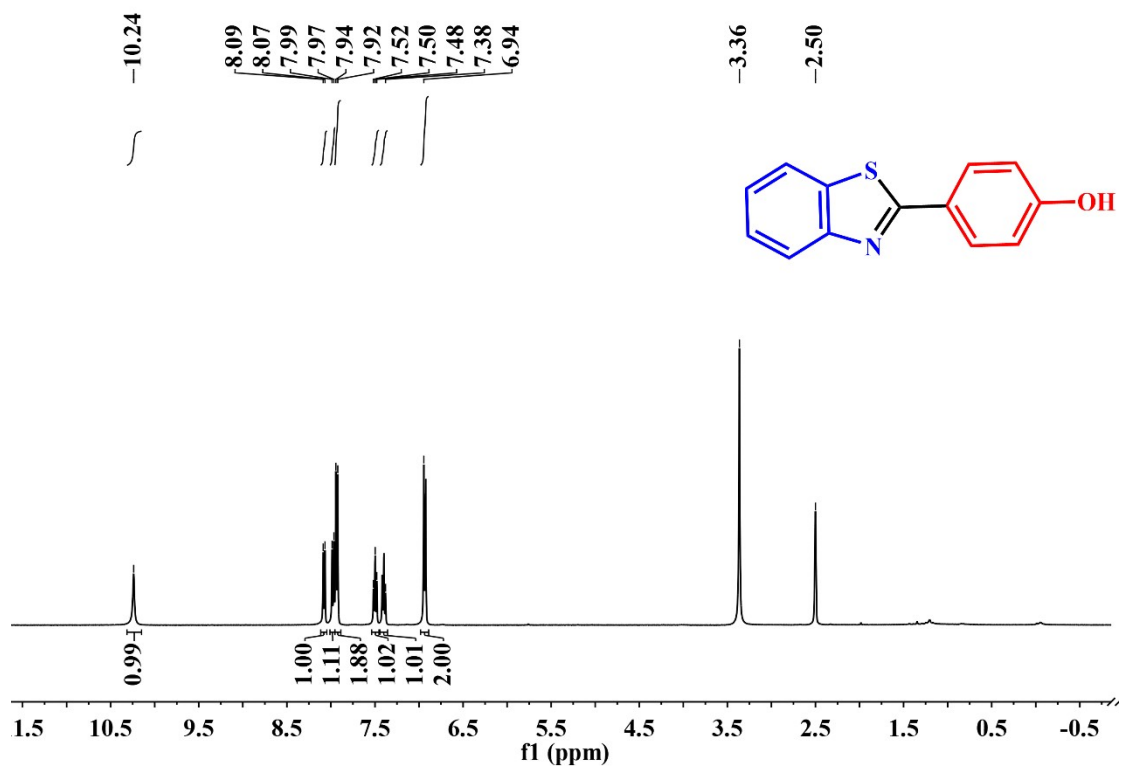
The ¹³C-NMR spectrum of 2-(4-methoxyphenyl)benzo[d]thiazole (**3h**).



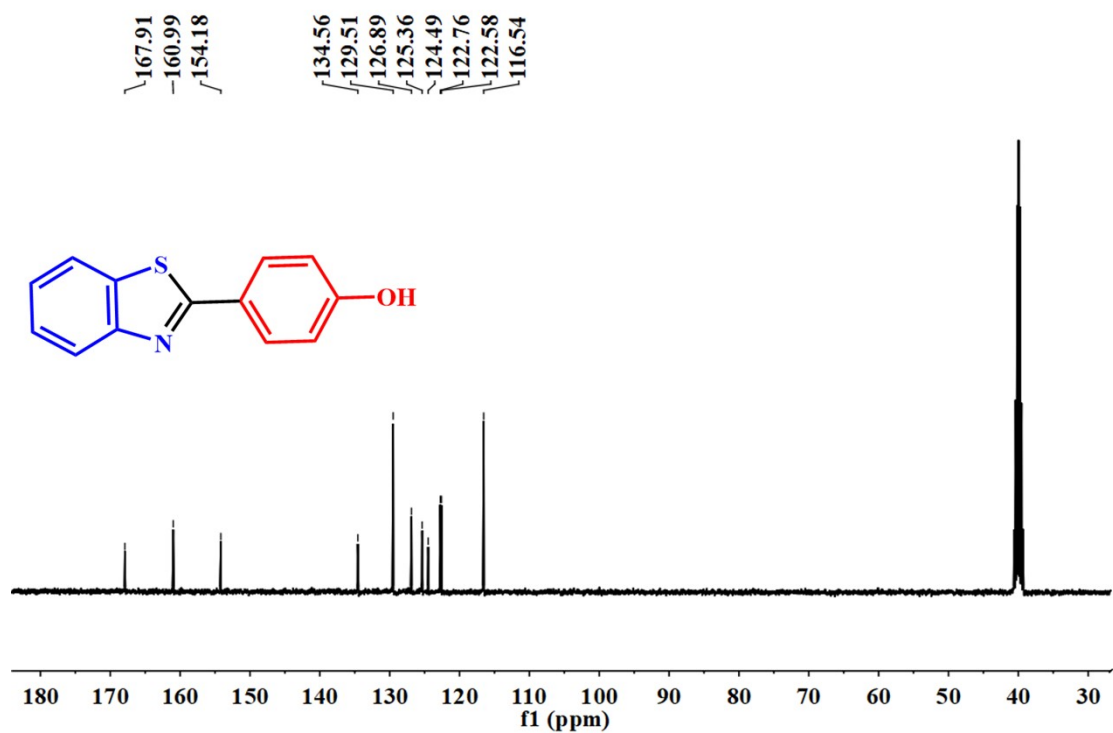
The ¹H-NMR spectrum of 2-(*p*-tolyl)benzo[d]thiazole (**3i**).



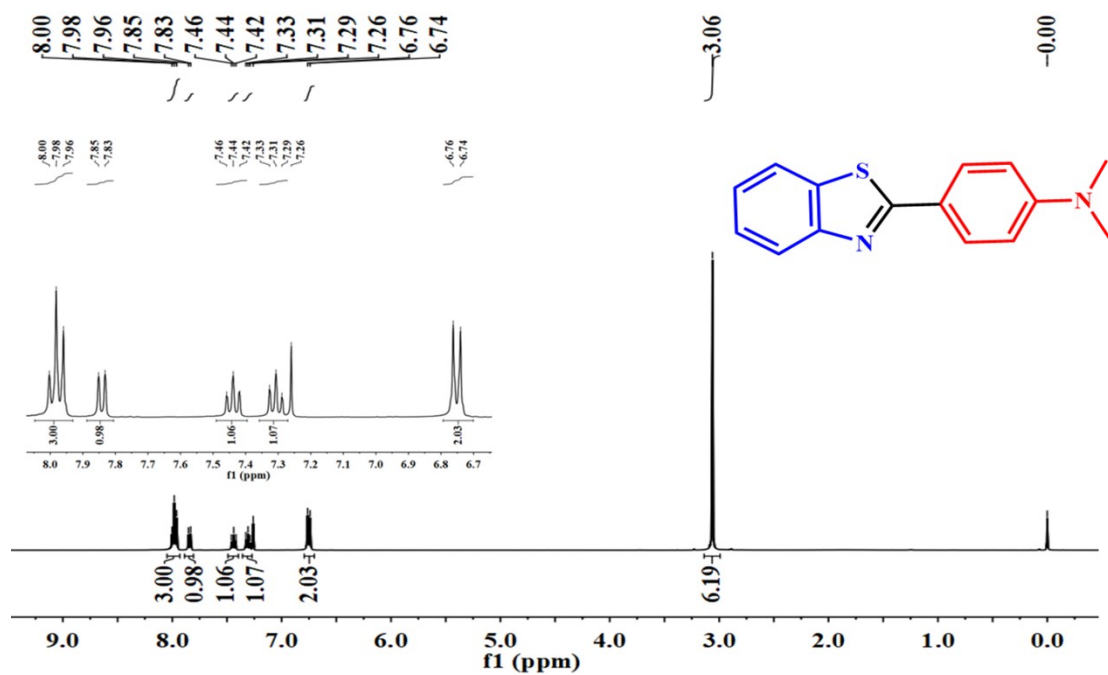
The ¹³C-NMR spectrum of 2-(*p*-tolyl)benzo[d]thiazole (**3i**).



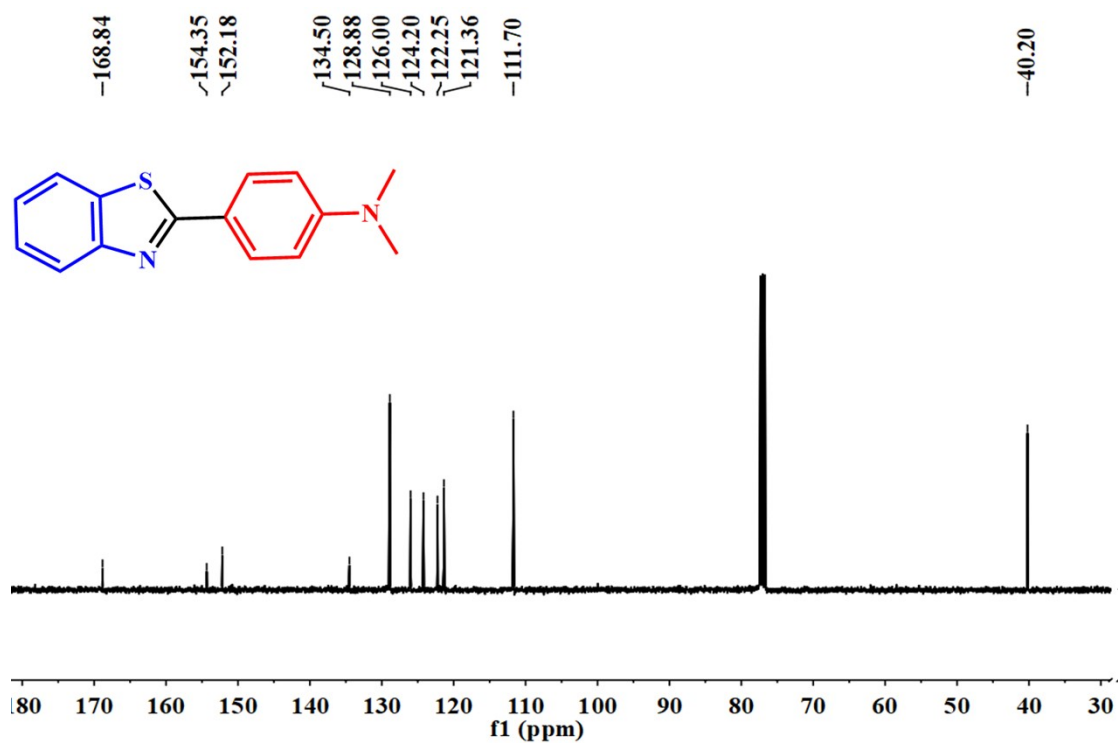
The ^1H -NMR spectrum of 2-(4-hydroxyphenyl)benzo[d]thiazol (**3j**).



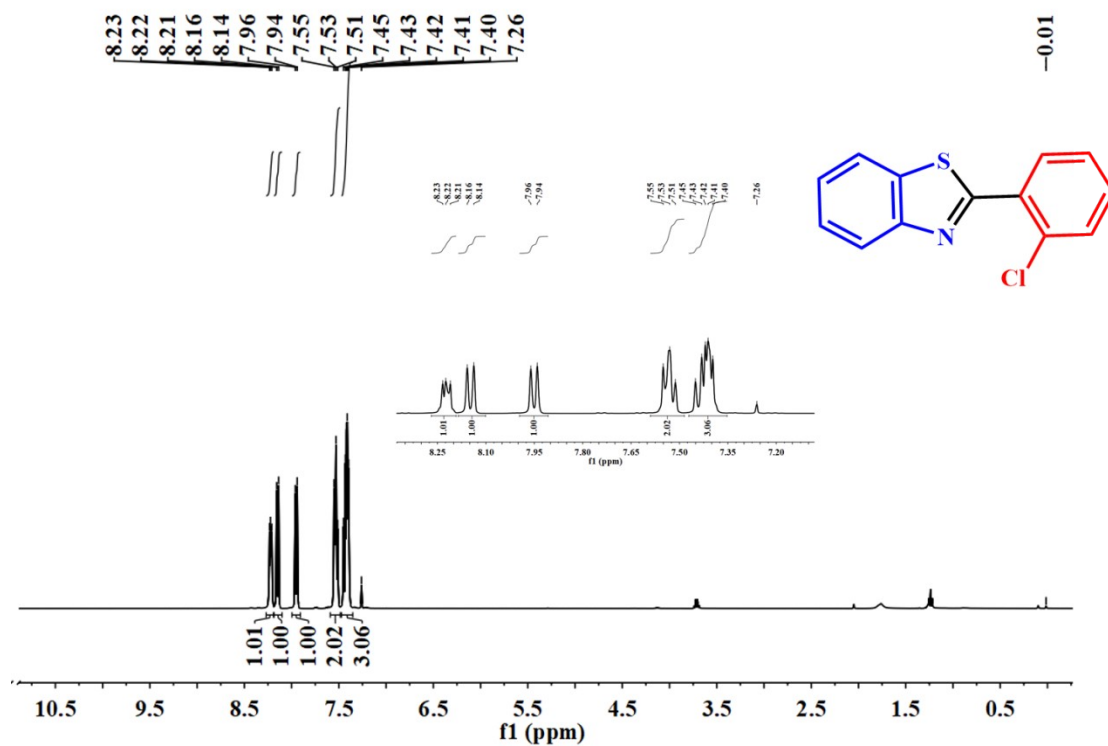
The ^{13}C -NMR spectrum of 2-(4-hydroxyphenyl)benzo[d]thiazol (**3j**).



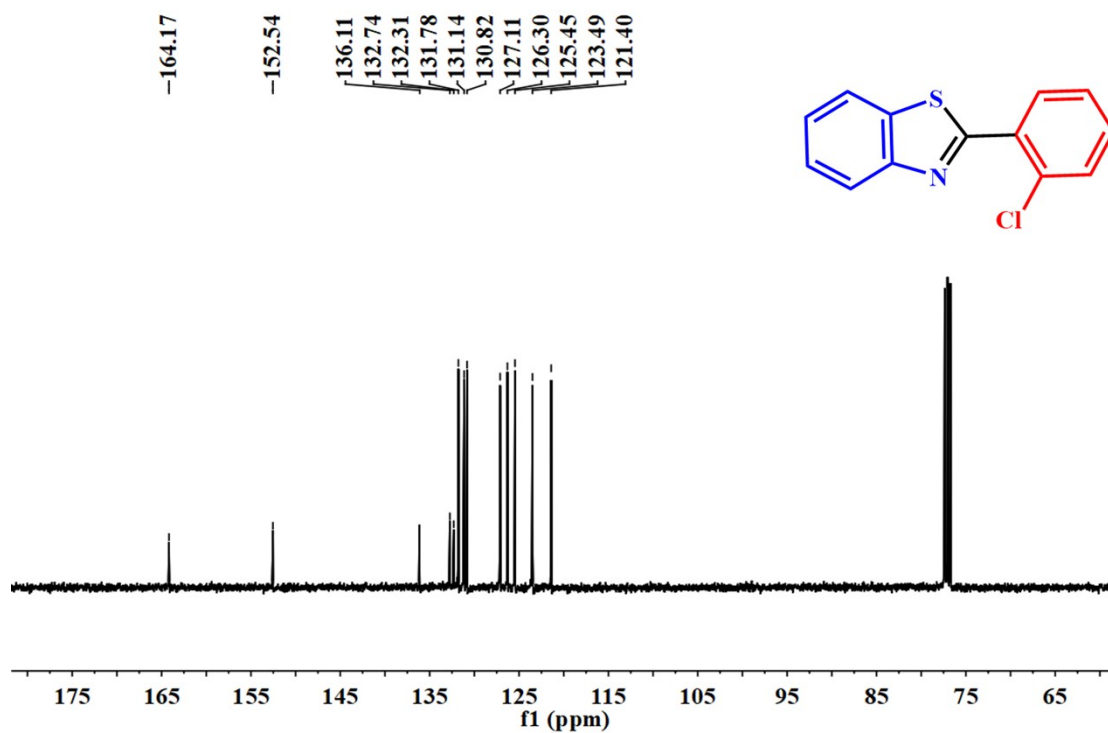
The ¹H-NMR spectrum of 4-(benzo[d]thiazol-2-yl)-*N,N*-dimethylaniline (**3k**).



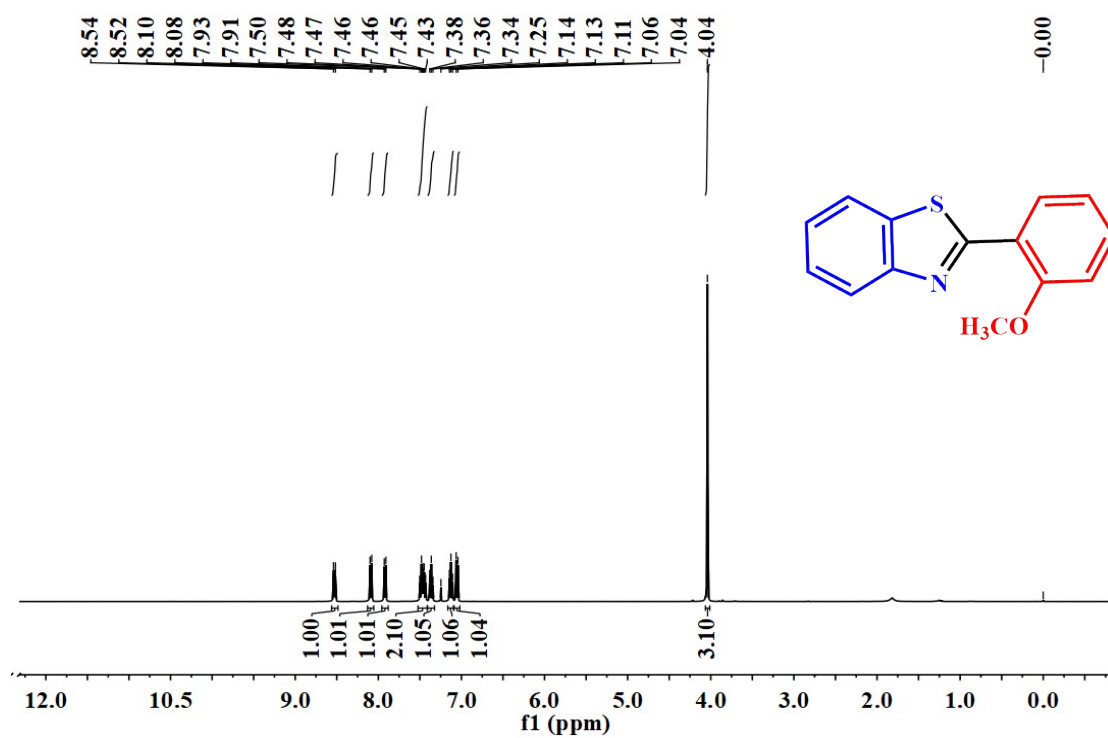
The ¹³C-NMR spectrum of 4-(benzo[d]thiazol-2-yl)-*N,N*-dimethylaniline (**3k**).



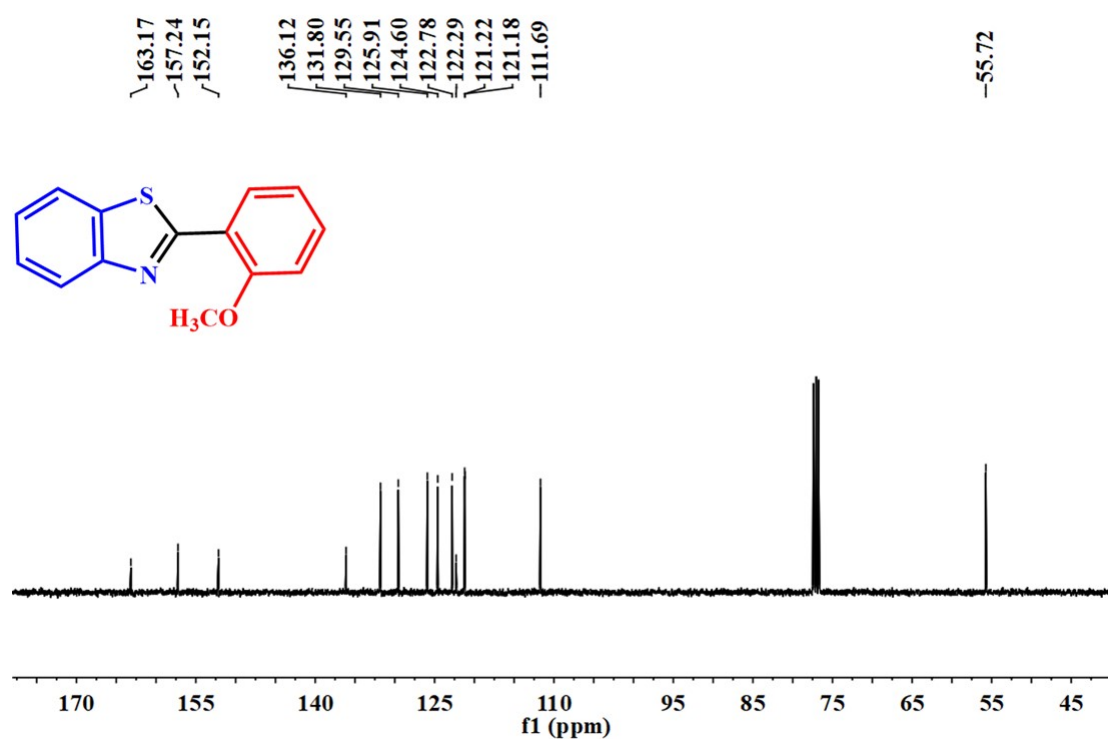
The ¹H-NMR spectrum of 2-(2-chlorophenyl)benzo[d]thiazole (3I).



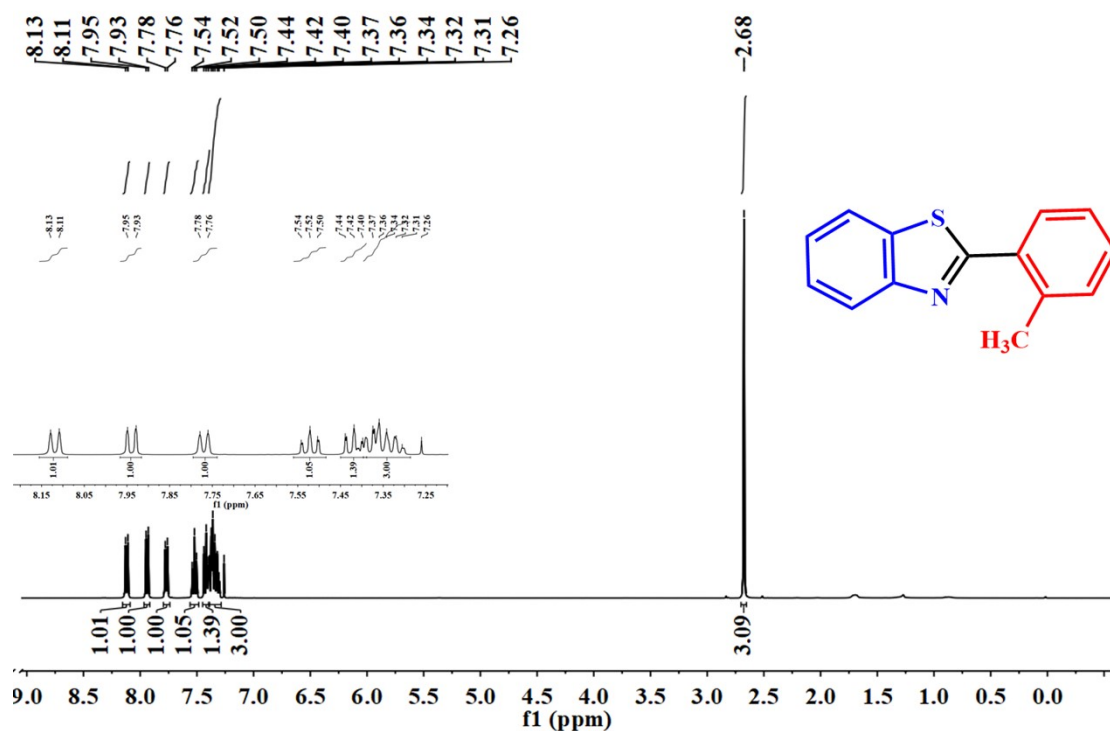
The ¹³C-NMR spectrum of 2-(2-chlorophenyl)benzo[d]thiazole (3I).



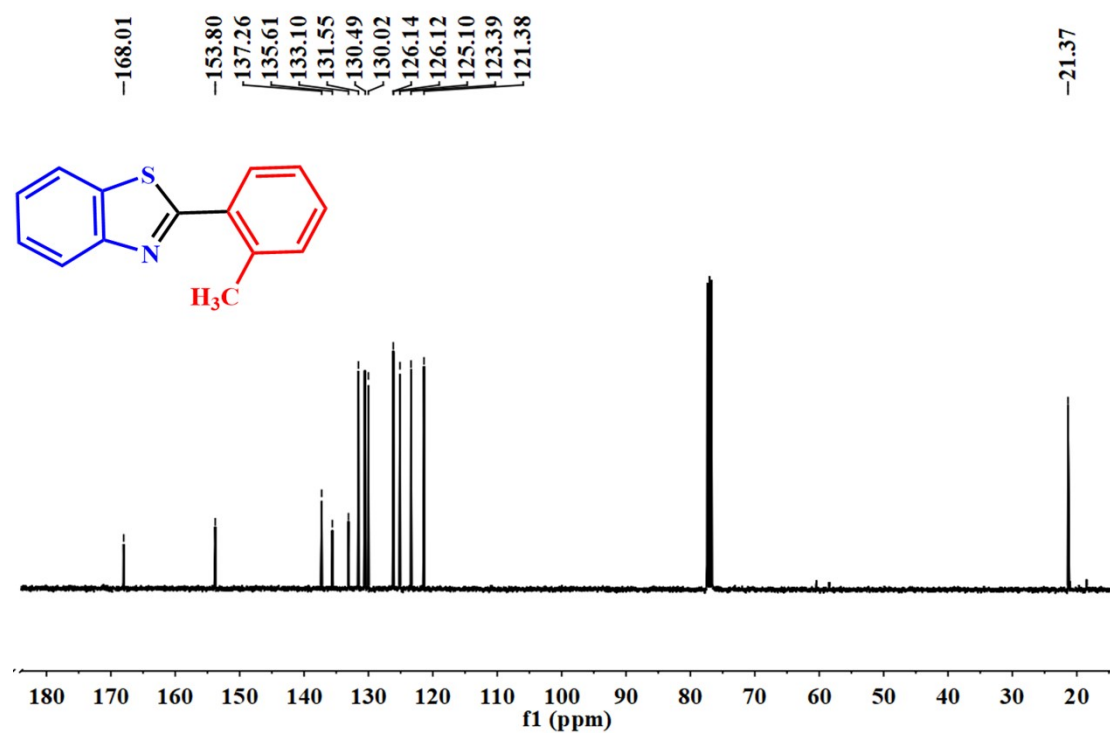
The ¹H-NMR spectrum of 2-(2-methoxyphenyl)benzo[d]thiazole (**3m**).



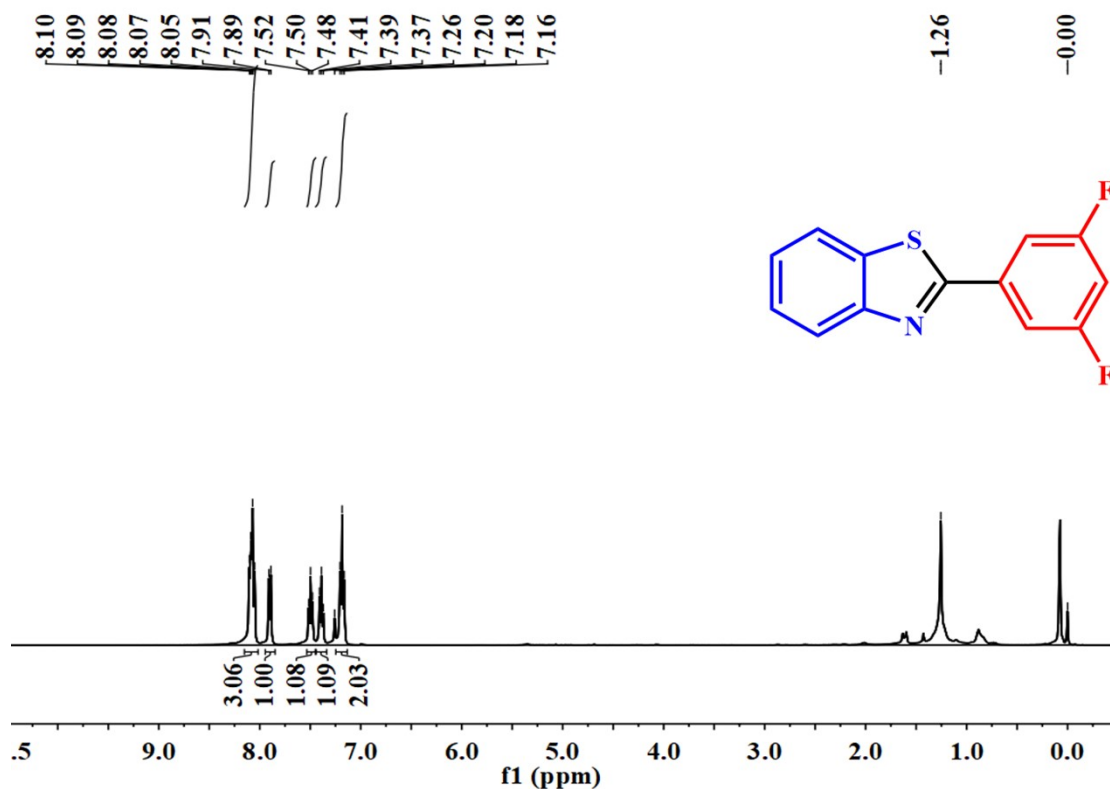
The ¹³C-NMR spectrum of 2-(2-methoxyphenyl)benzo[d]thiazole (**3m**).



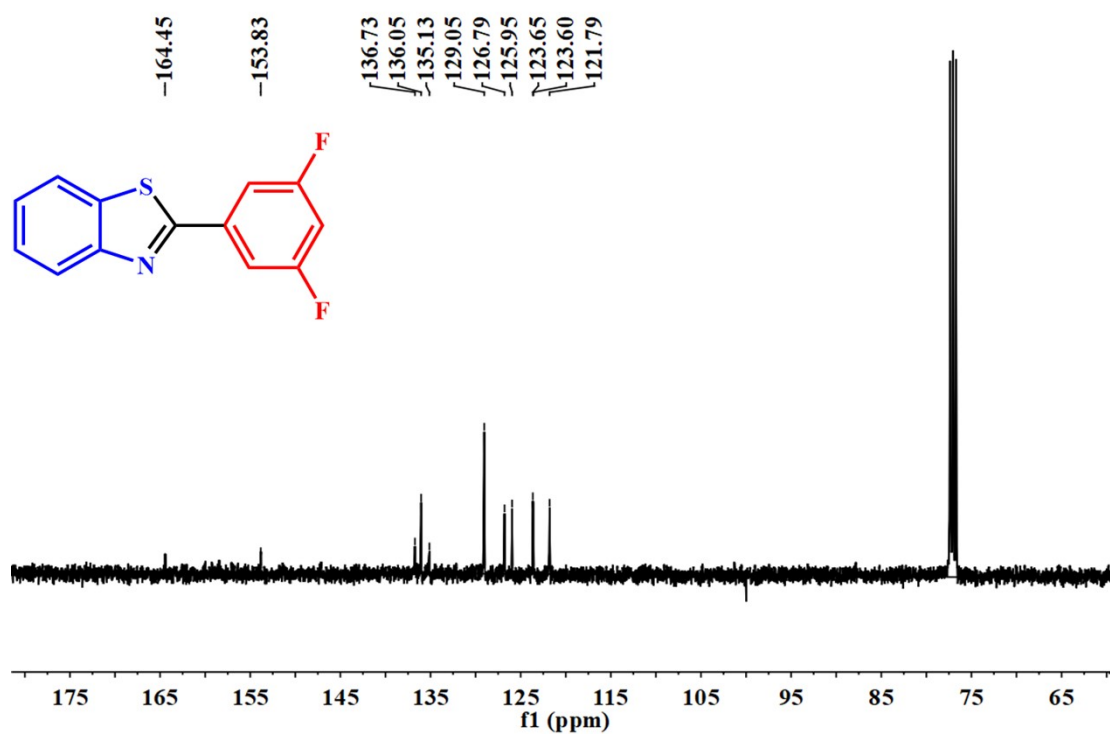
The ¹H-NMR spectrum of 2-(*o*-tolyl)benzo[d]thiazole (**3n**).



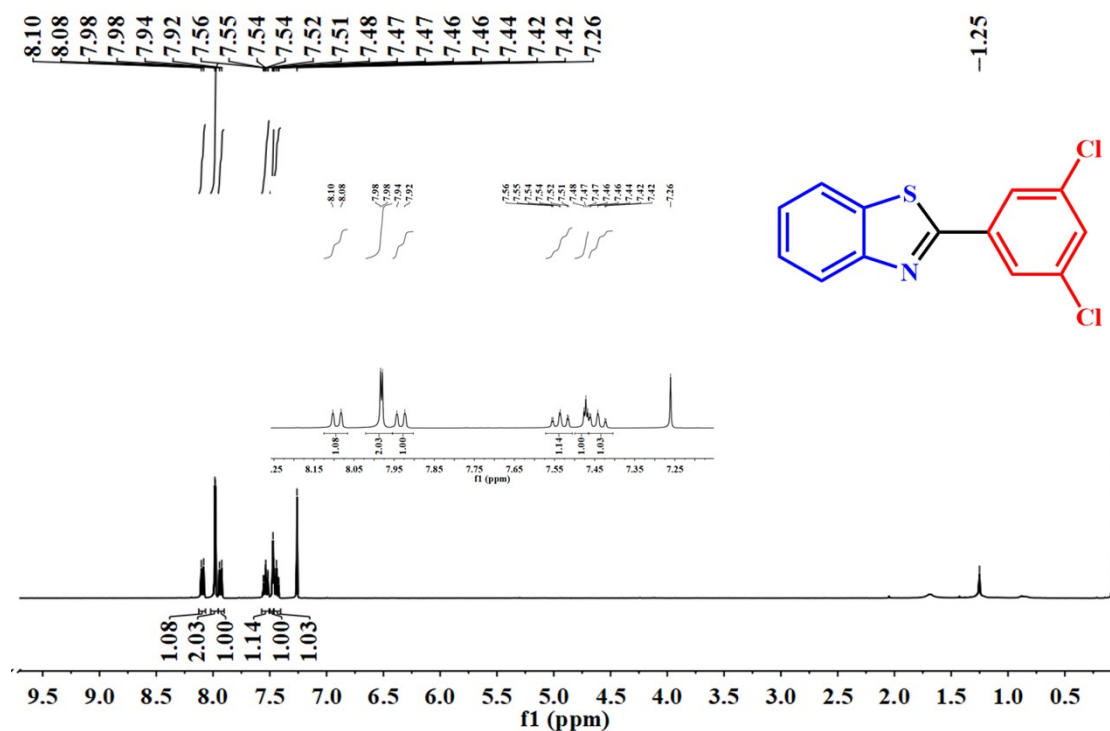
The ¹³C-NMR spectrum of 2-(*o*-tolyl)benzo[d]thiazole (**3n**).



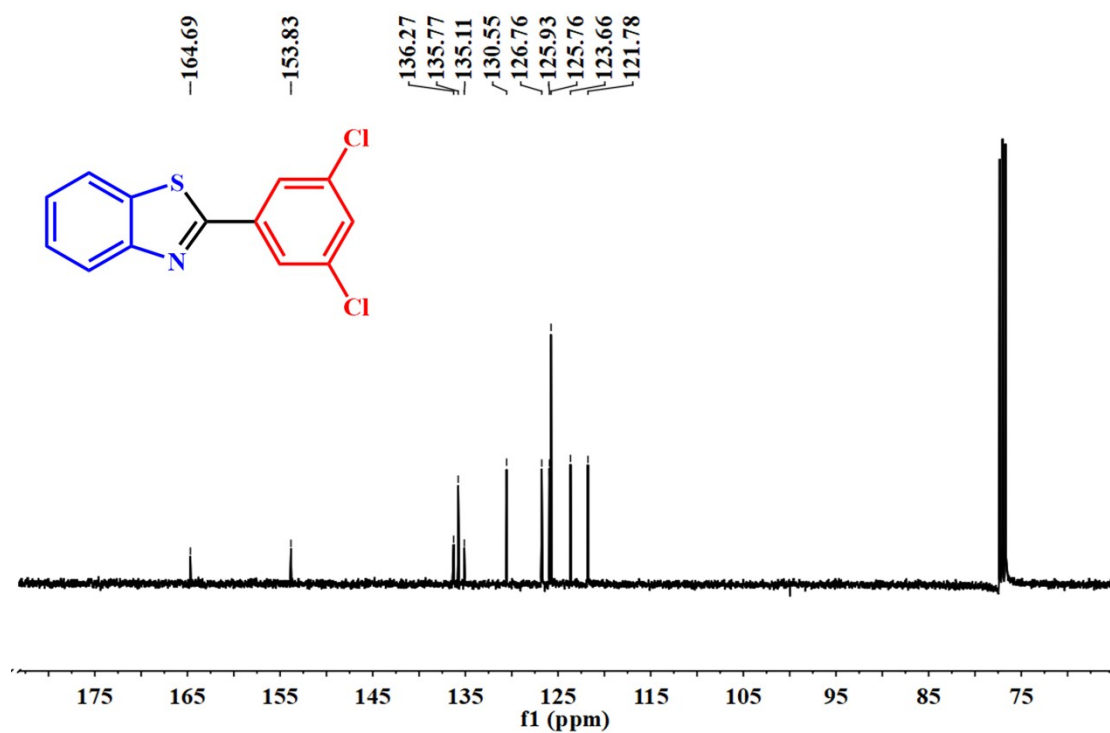
The ¹H-NMR spectrum of 2-(3,5-difluorophenyl)benzo[d]thiazole (3o).



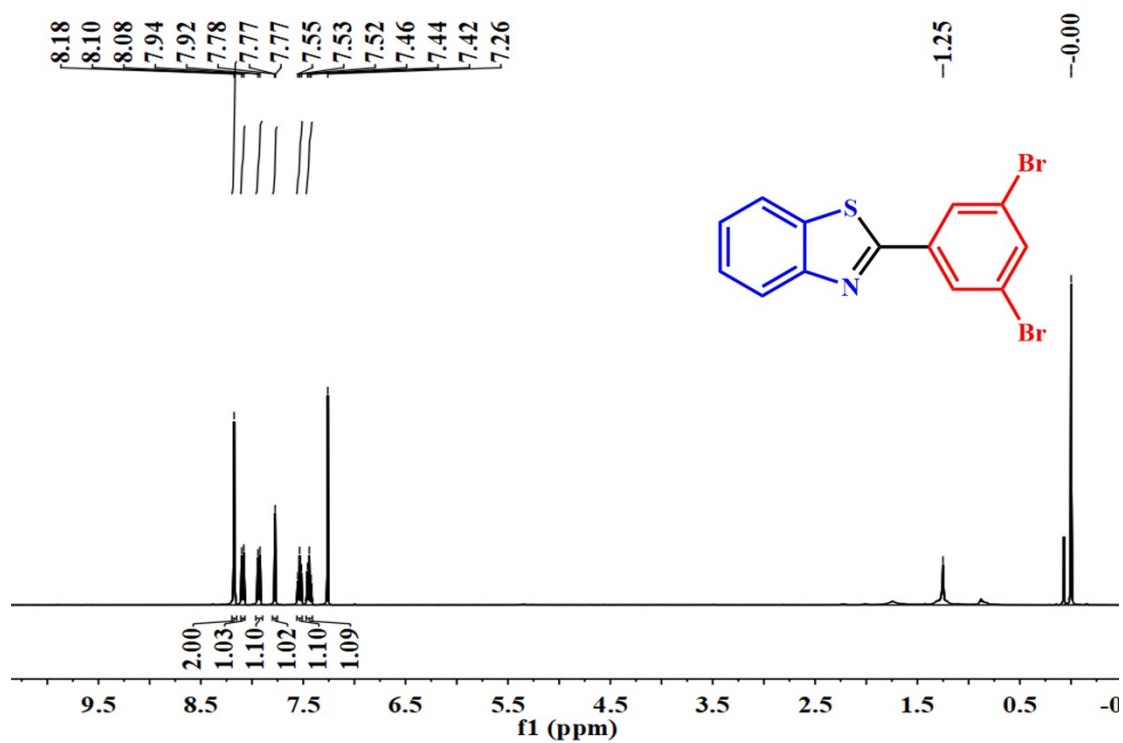
The ¹³C-NMR spectrum of 2-(3,5-difluorophenyl)benzo[d]thiazole (3o).



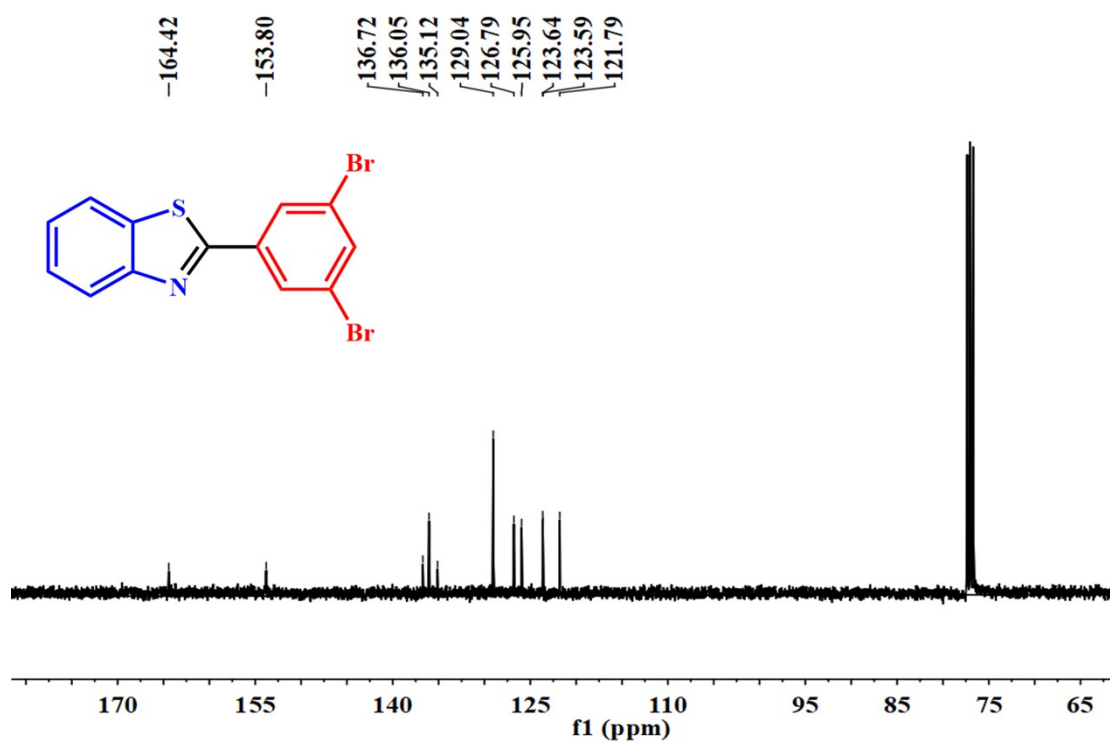
The ¹H-NMR spectrum of 2-(3,5-dichlorophenyl)benzo[d]thiazole (**3p**).



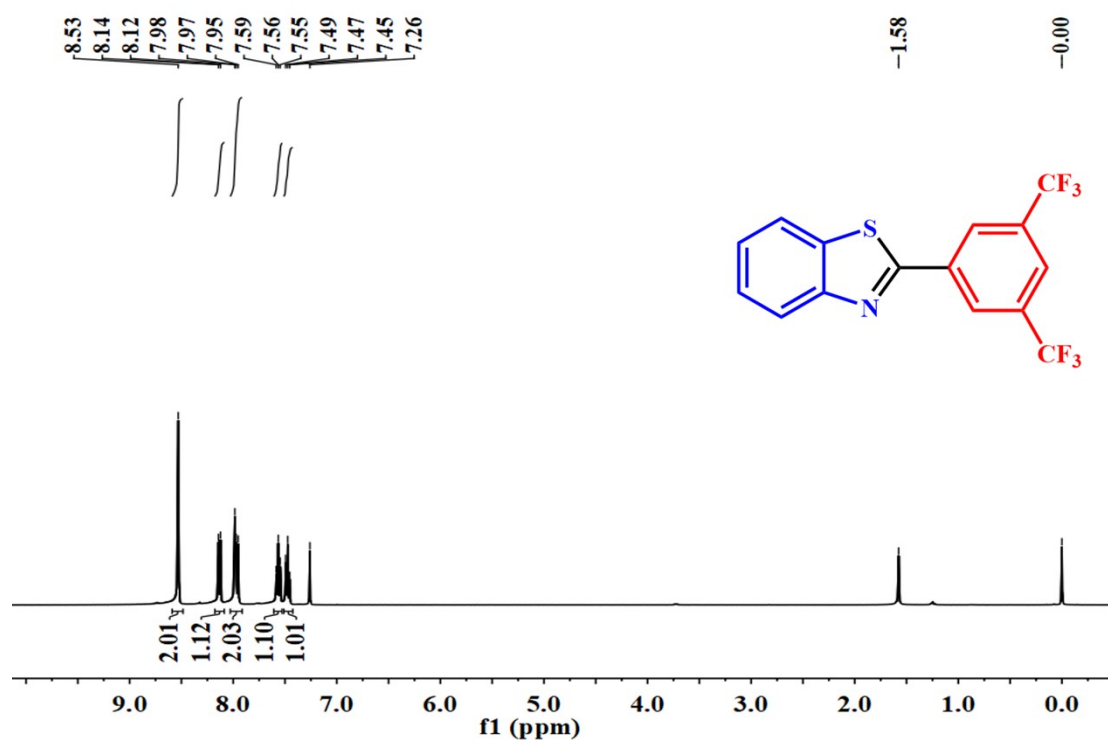
The ¹³C-NMR spectrum of 2-(3,5-dichlorophenyl)benzo[d]thiazole (**3p**).



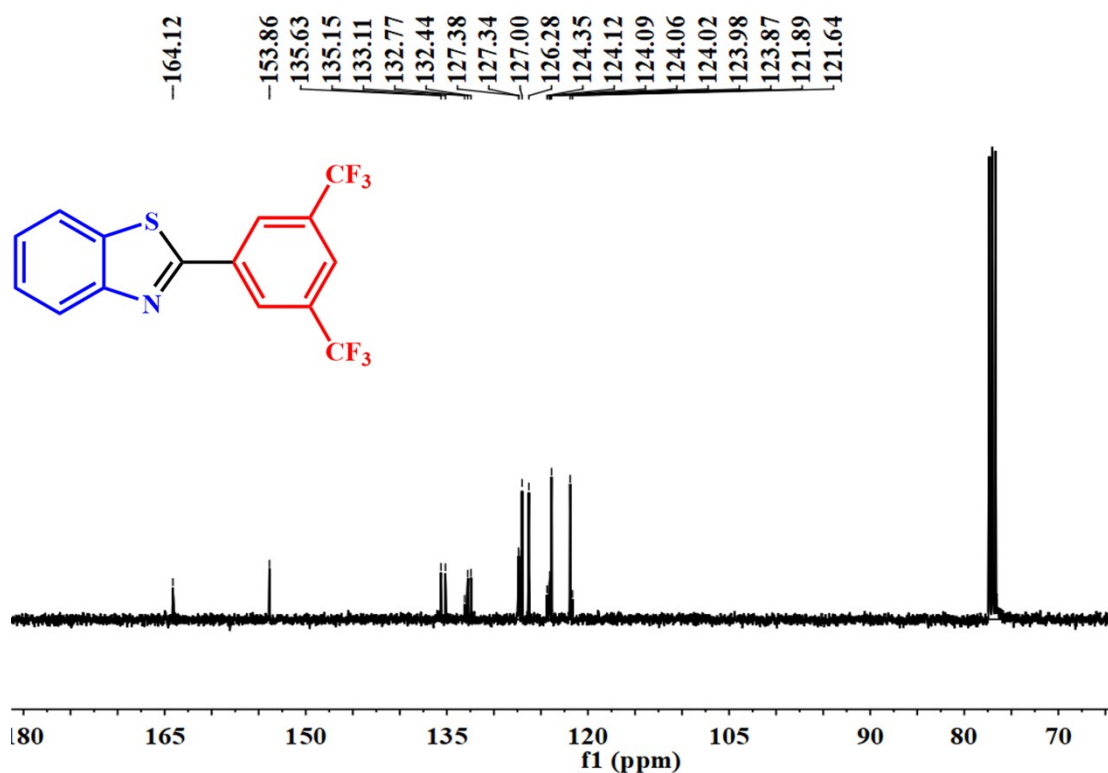
The ¹H-NMR spectrum of 2-(3,5-dibromophenyl)benzo[d]thiazole (**3q**).



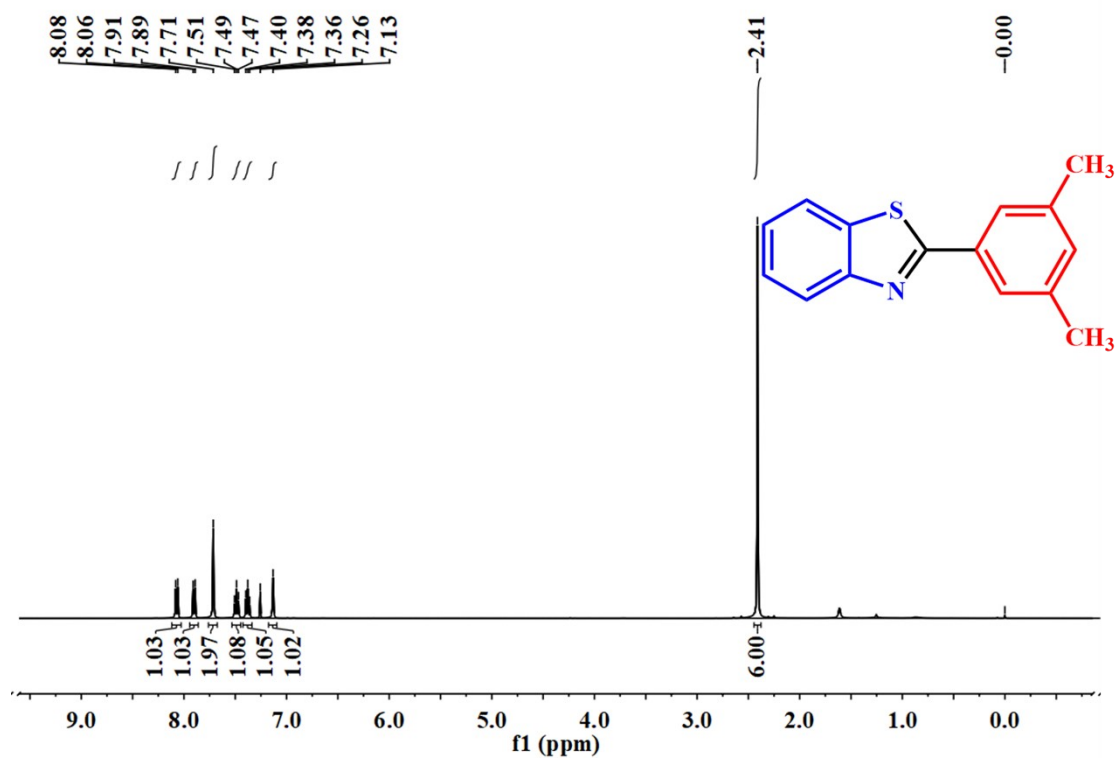
The ¹³C-NMR spectrum of 2-(3,5-dibromophenyl)benzo[d]thiazole (**3q**).



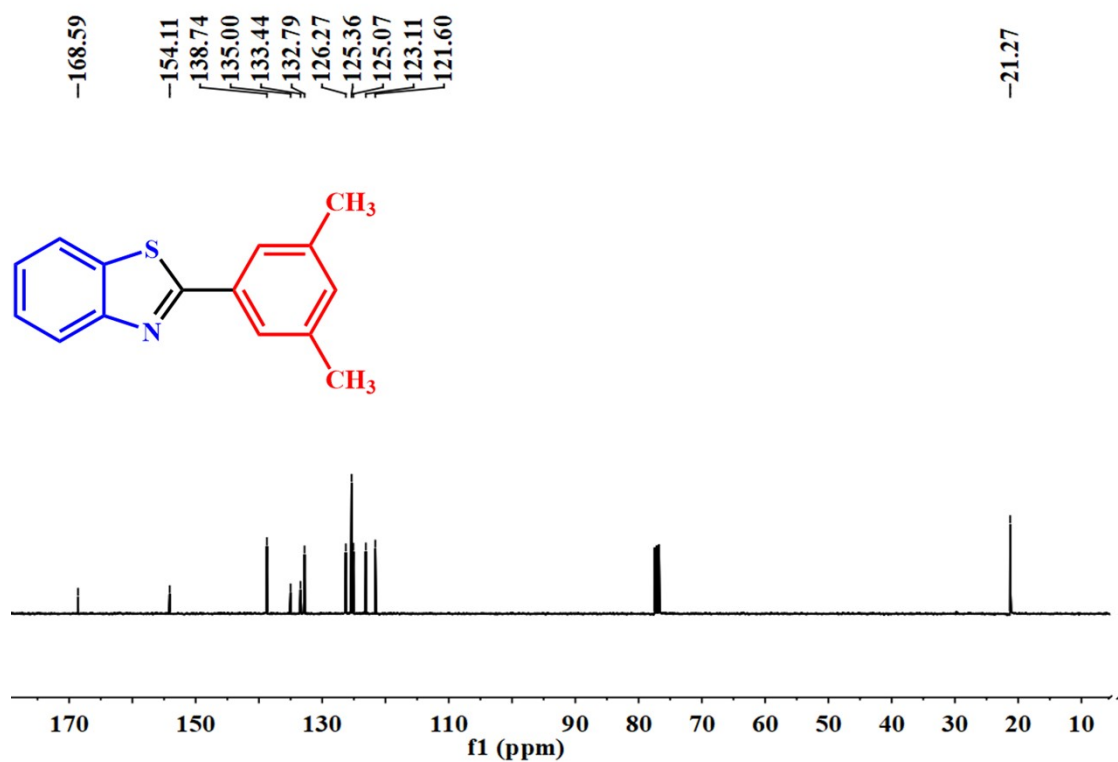
The ¹H-NMR spectrum of 2-(3,5-bis(trifluoromethyl)phenyl)benzo[d]thiazole (**3r**).



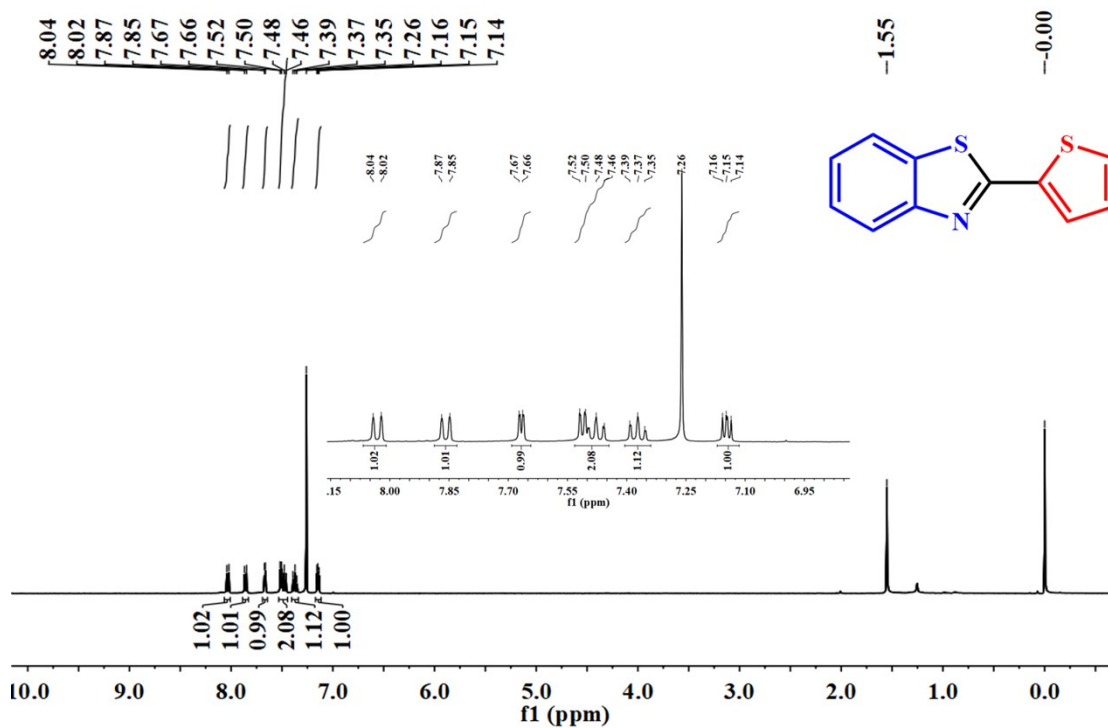
The ¹³C-NMR spectrum of 2-(3,5-bis(trifluoromethyl)phenyl)benzo[d]thiazole (**3r**).



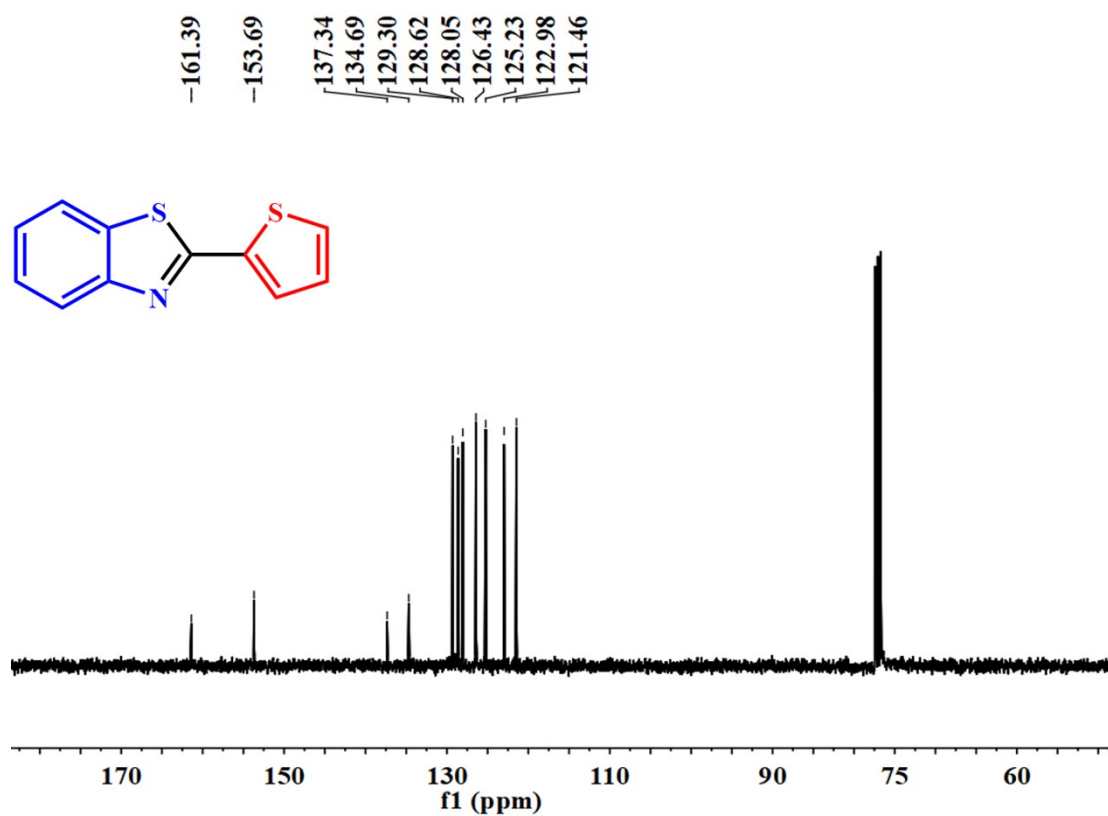
The ¹H-NMR spectrum of 2-(3,5-dimethylphenyl)benzo[d]thiazole (**3s**).



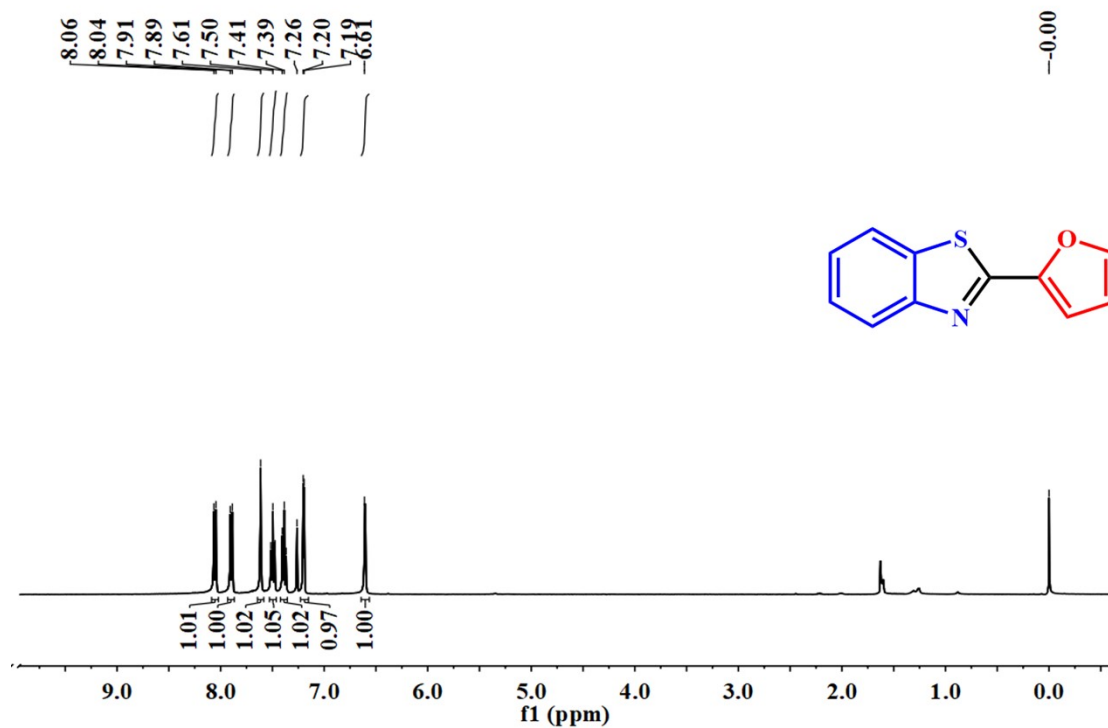
The ¹³C-NMR spectrum of 2-(3,5-dimethylphenyl)benzo[d]thiazole (**3s**).



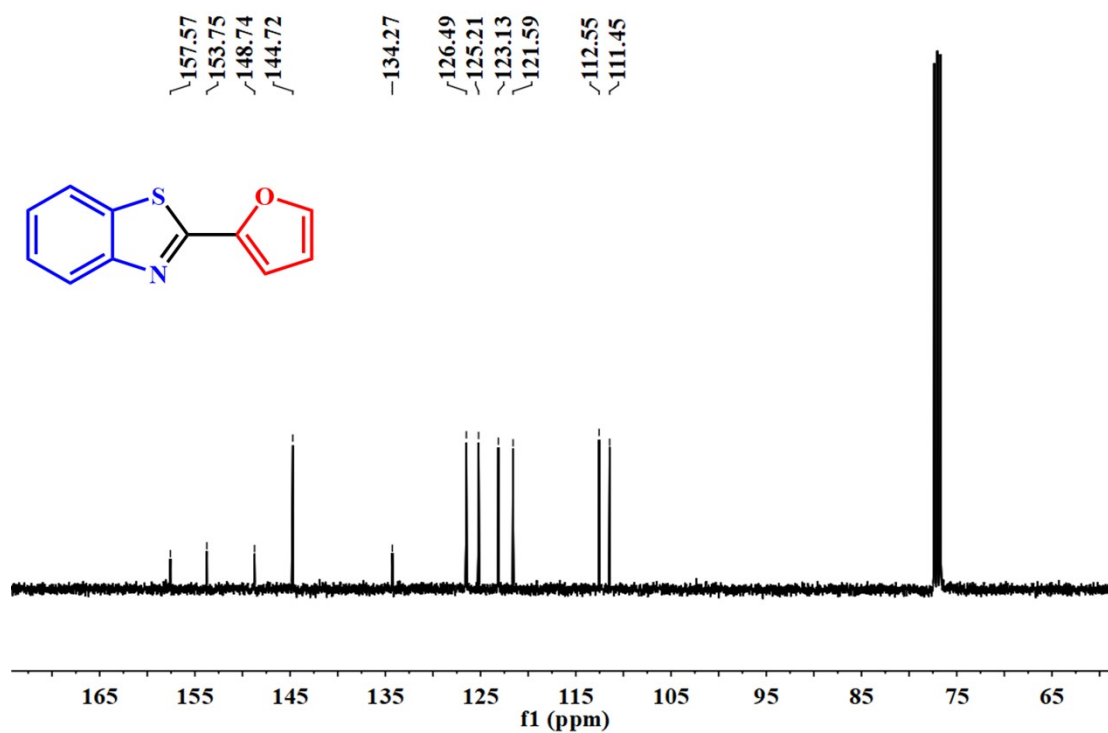
The ¹H-NMR spectrum of 2-(thiophen-2-yl)benzo[d]thiazole (**3t**).



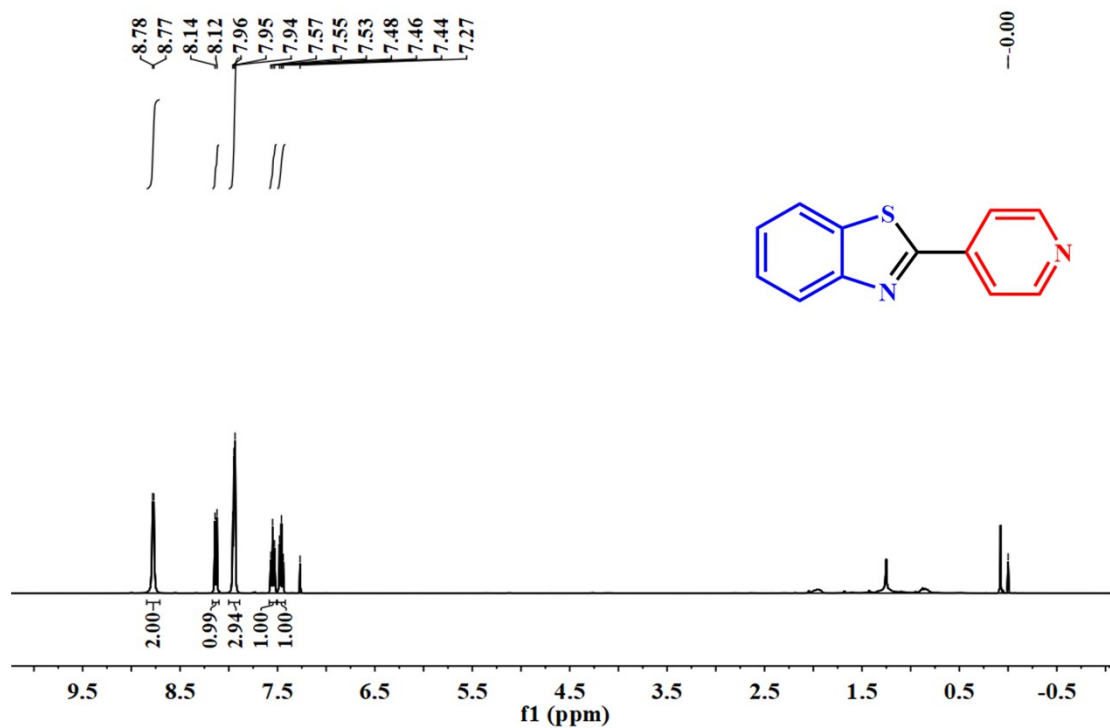
The ¹³C-NMR spectrum of 2-(thiophen-2-yl)benzo[d]thiazole (**3t**).



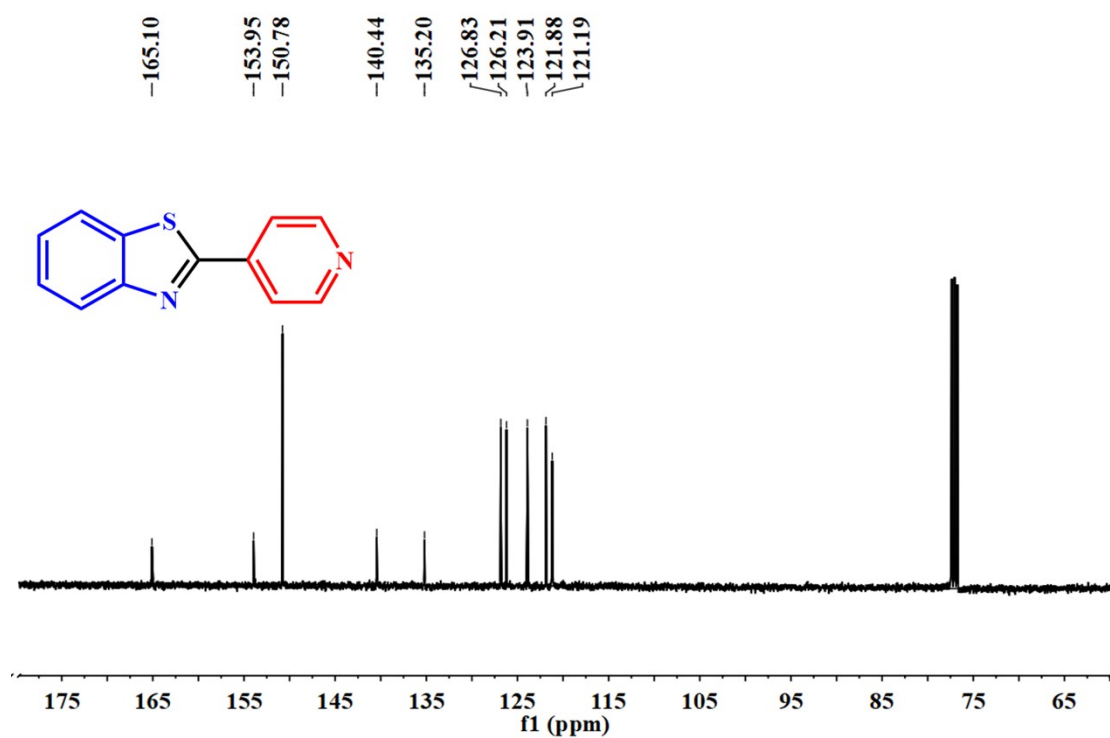
The ¹H-NMR spectrum of 2-(furan-2-yl)benzo[d]thiazole (**3u**).



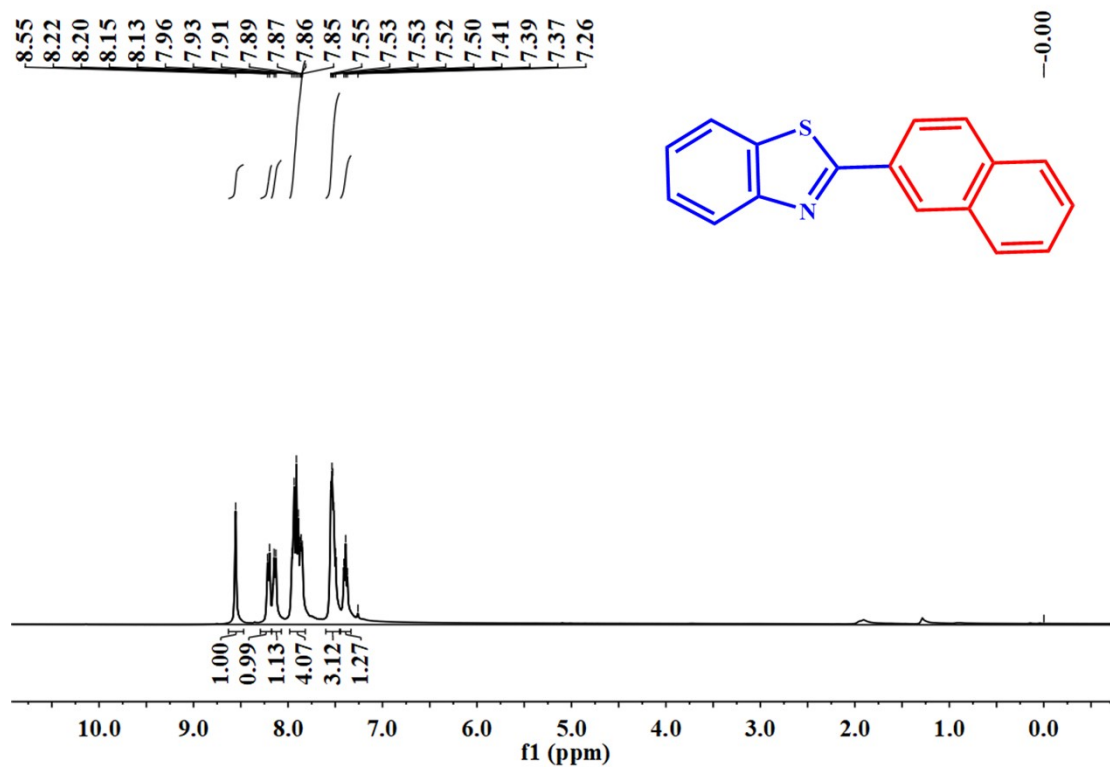
The ¹³C-NMR spectrum of 2-(furan-2-yl)benzo[d]thiazole (**3u**).



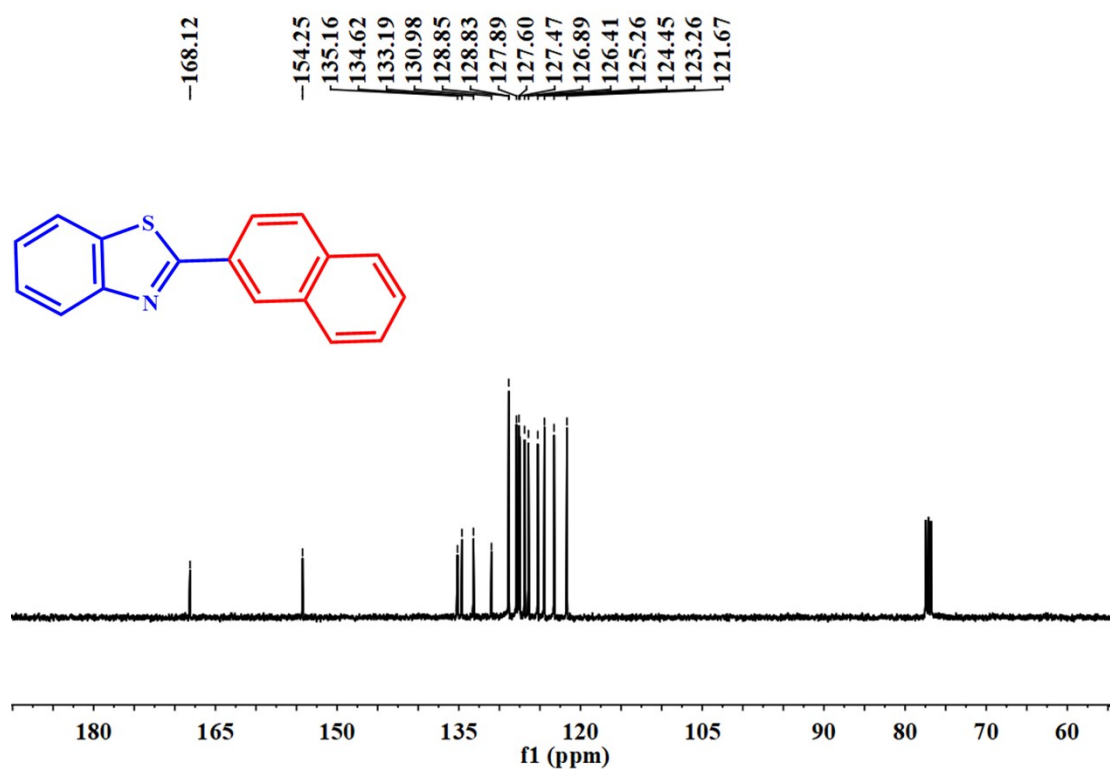
The ¹H-NMR spectrum of 2-(pyridin-4-yl)benzo[d]thiazole (**3v**).



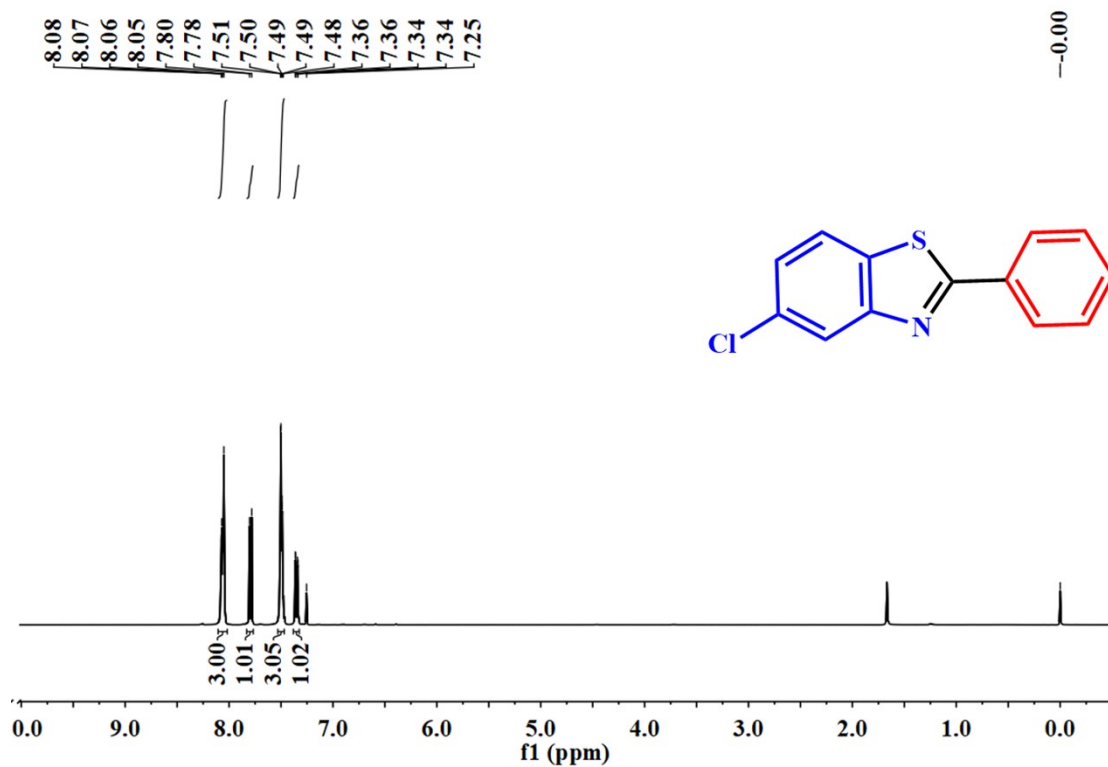
The ¹³C-NMR spectrum of 2-(pyridin-4-yl)benzo[d]thiazole (**3v**).



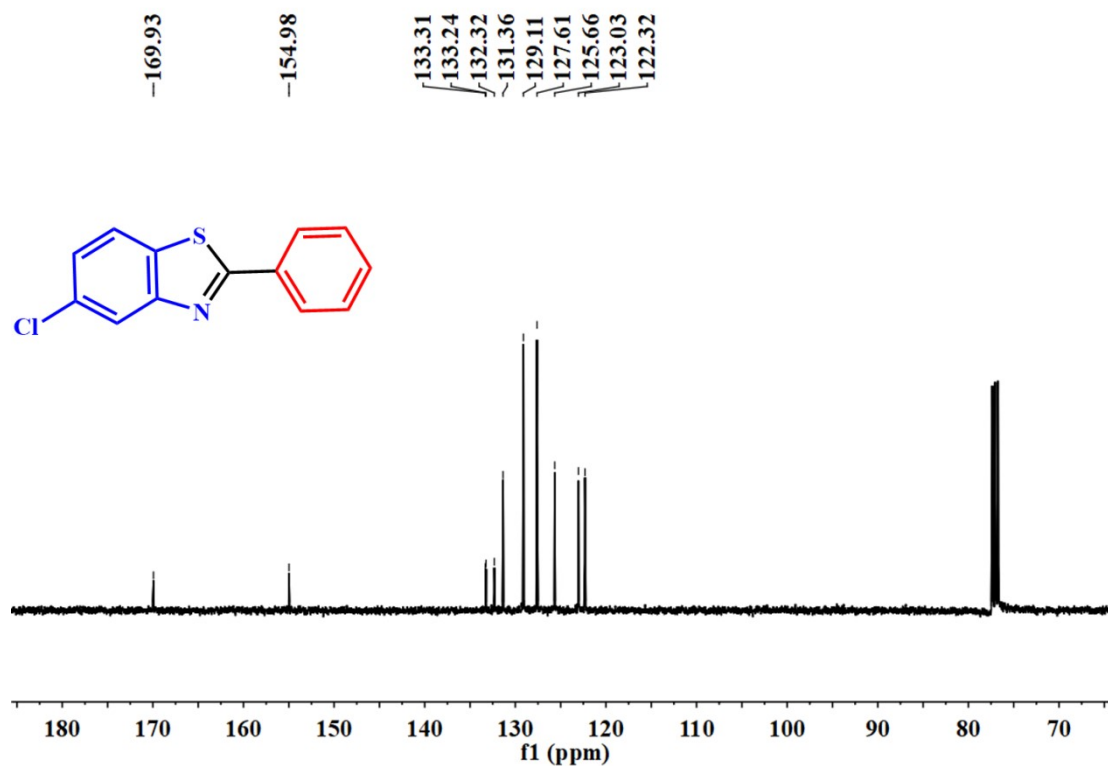
The ¹H-NMR spectrum of 2-(naphthalen-2-yl)benzo[d]thiazole (**3w**).



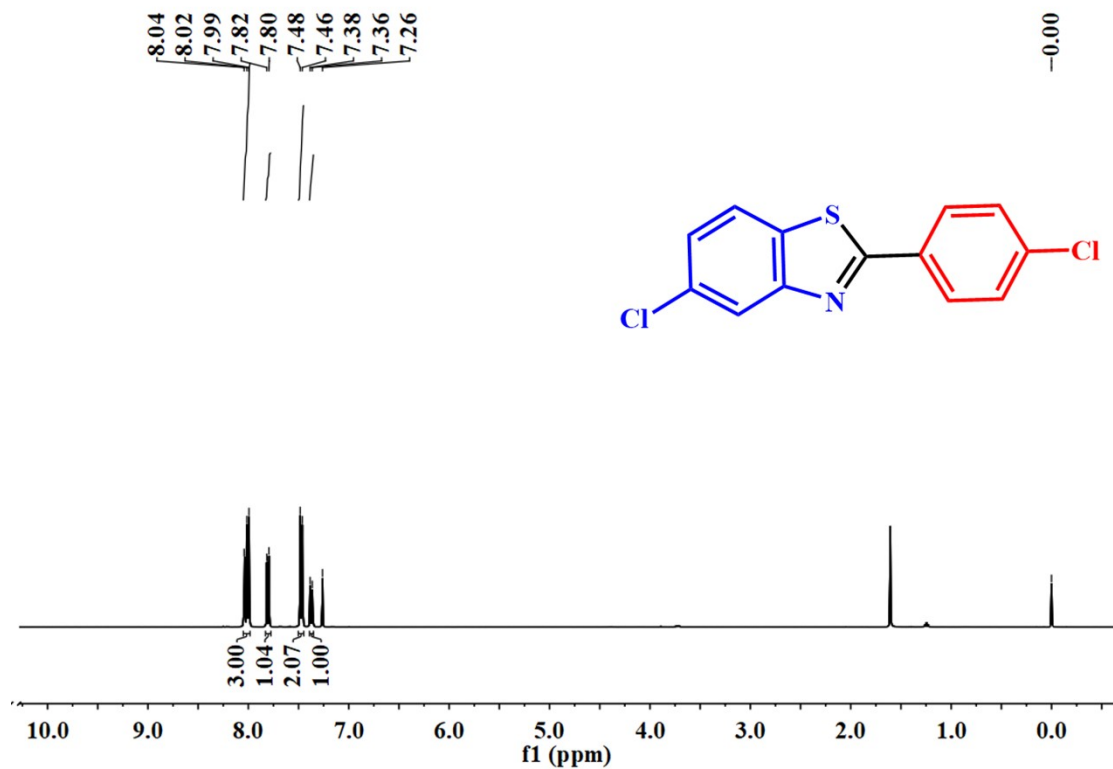
The ¹³C-NMR spectrum of 2-(naphthalen-2-yl)benzo[d]thiazole (**3w**).



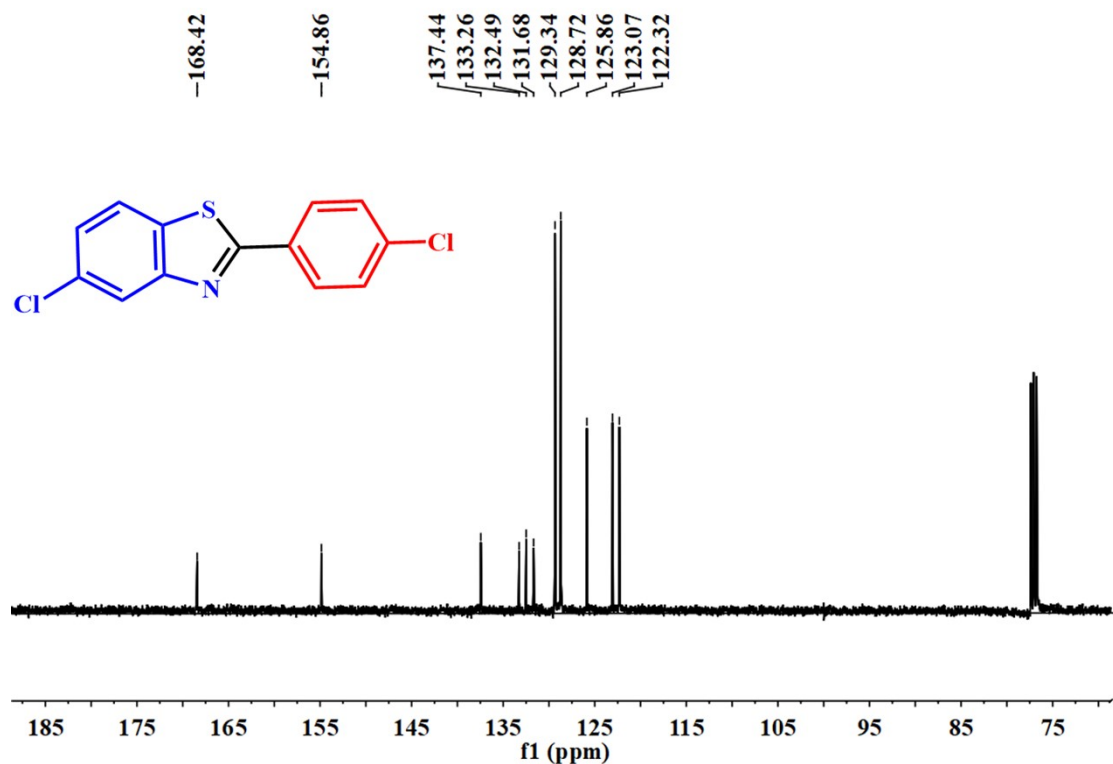
The ¹H-NMR spectrum of 5-chloro-2-phenylbenzo[d]thiazole (**4a**).



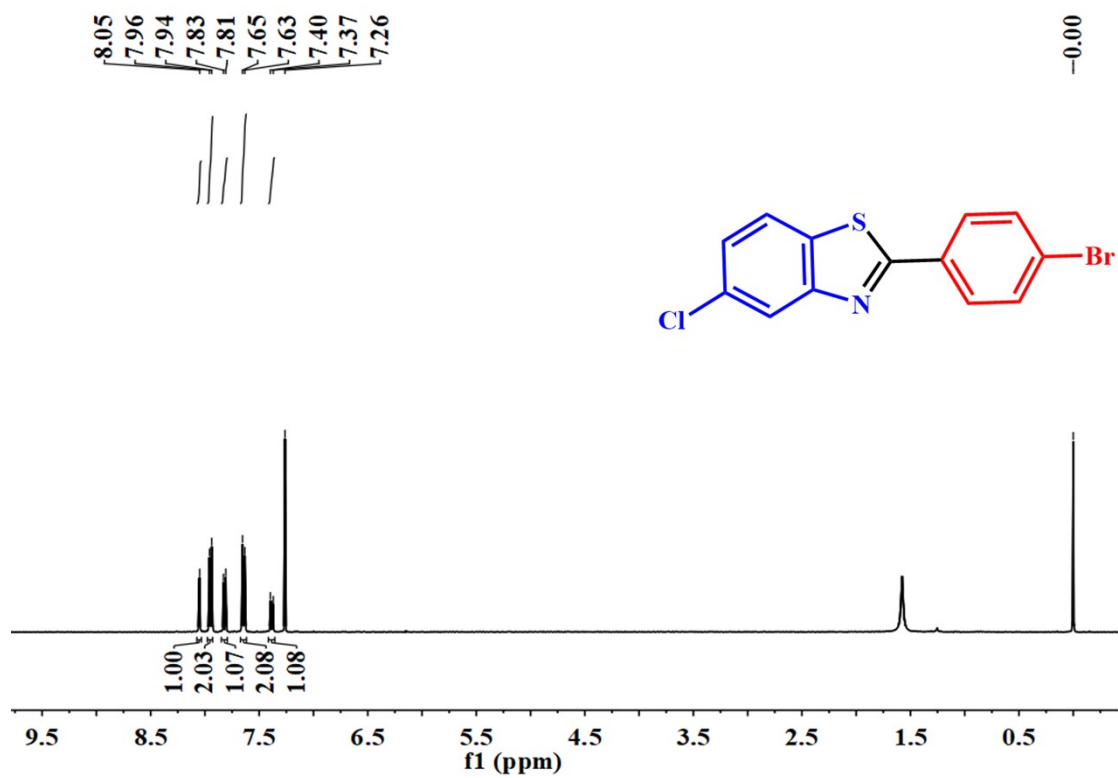
The ¹³C-NMR spectrum of 5-chloro-2-phenylbenzo[d]thiazole (**4a**).



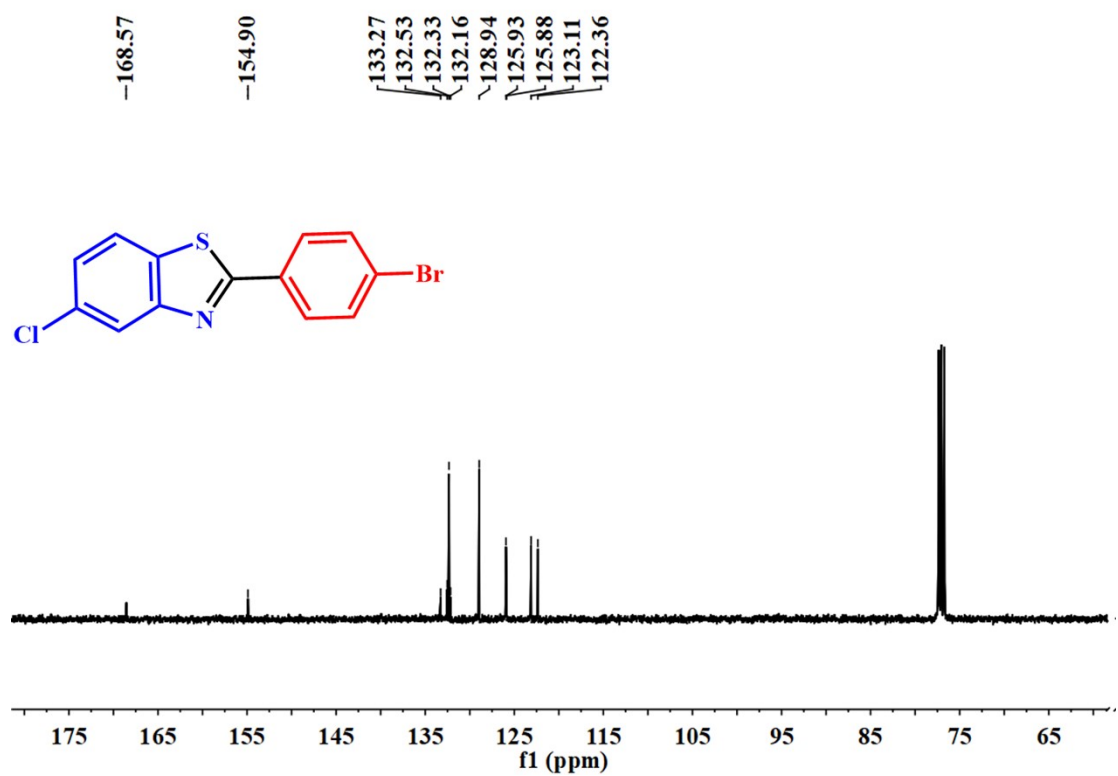
The ¹H-NMR spectrum of 5-chloro-2-(4-chlorophenyl)benzo[d]thiazole (**4b**).



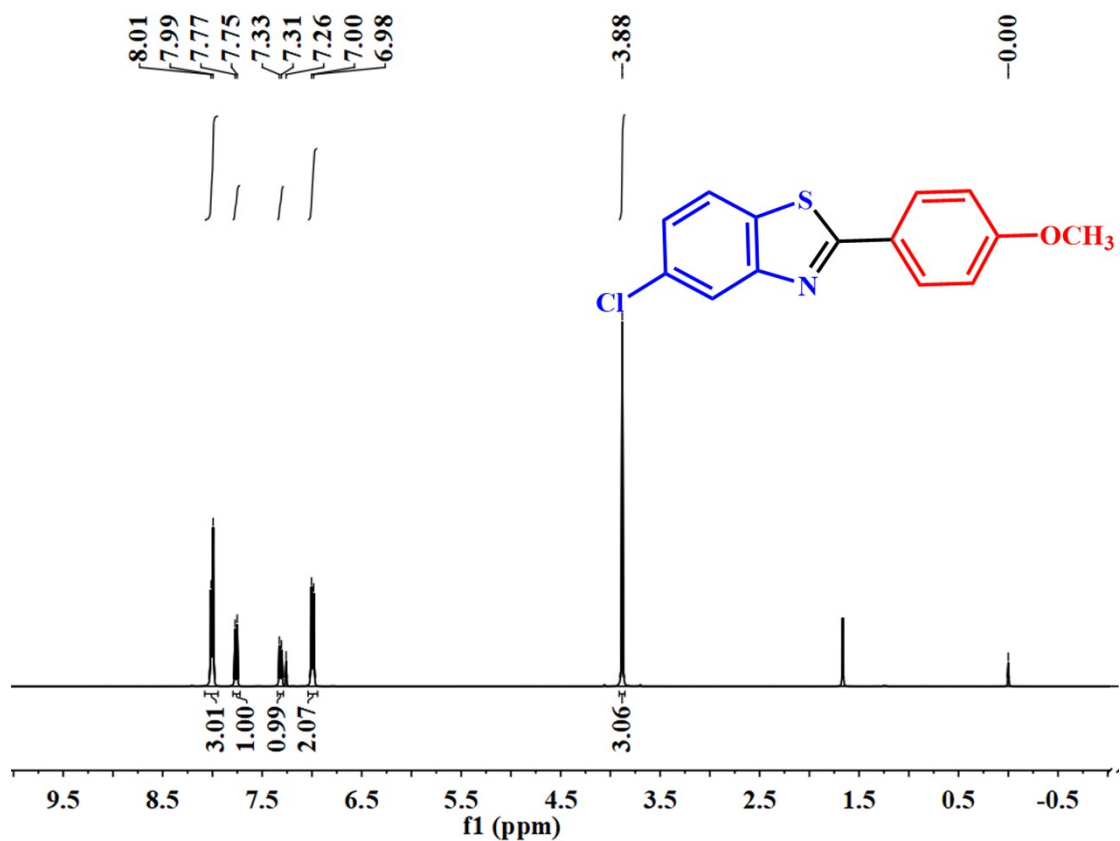
The ¹³C-NMR spectrum of 5-chloro-2-(4-chlorophenyl)benzo[d]thiazole (**4b**).



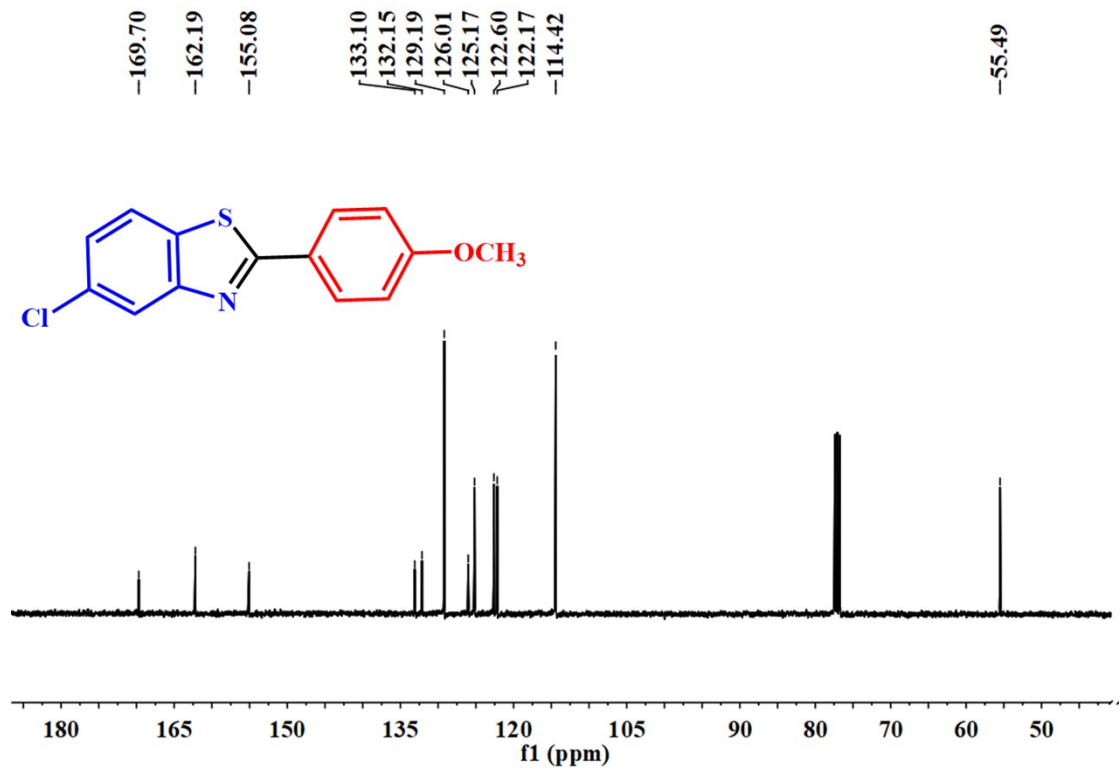
The ¹H-NMR spectrum of 2-(4-bromophenyl)-5-chlorobenzo[d]thiazole (4c).



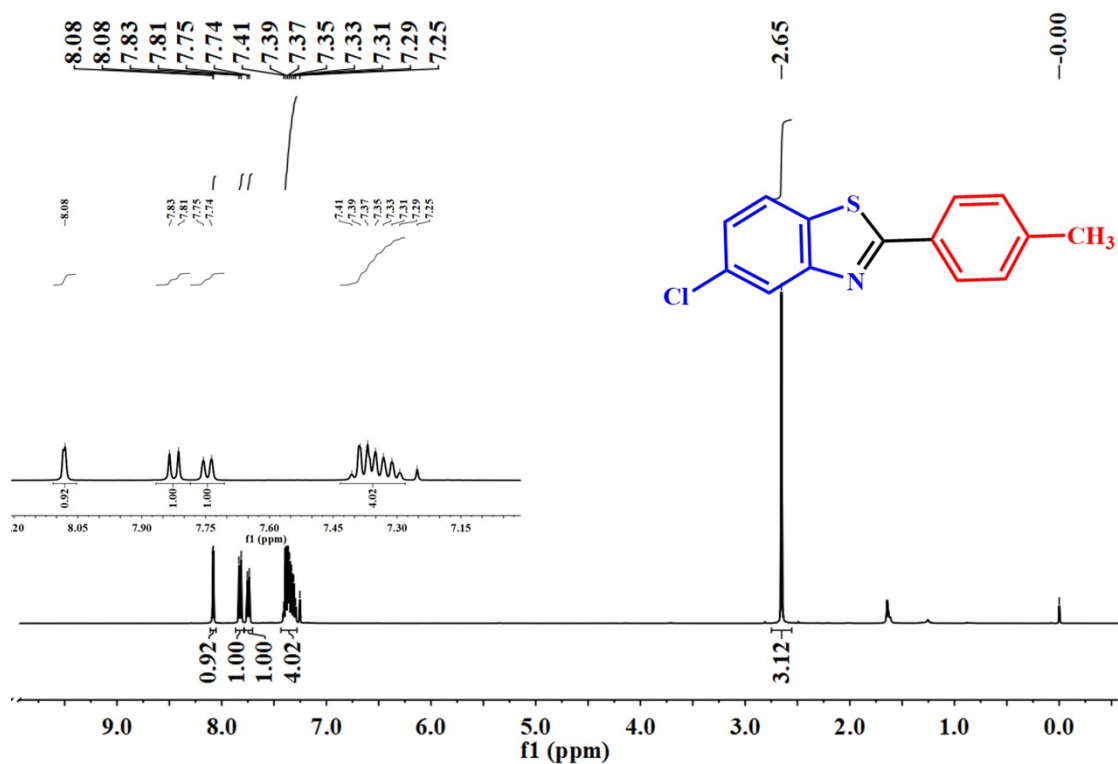
The ¹³C-NMR spectrum of 2-(4-bromophenyl)-5-chlorobenzo[d]thiazole (4c).



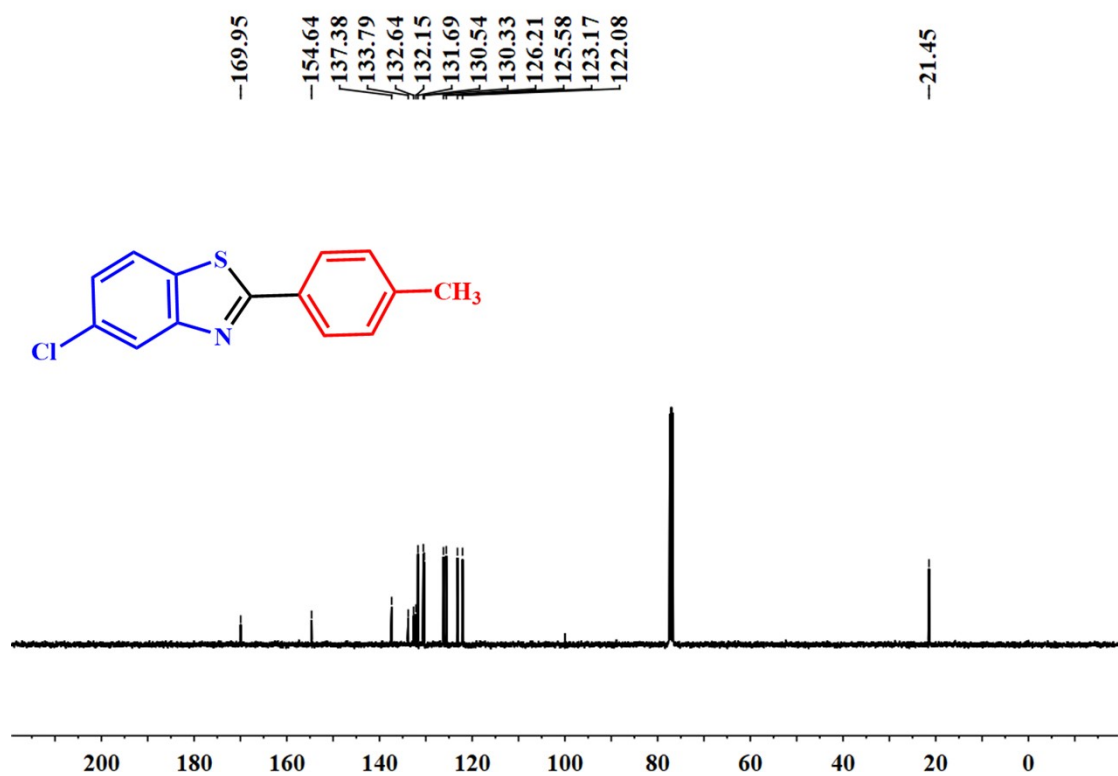
The ¹H-NMR spectrum of 5-chloro-2-(4-methoxyphenyl)benzo[d]thiazole (**4d**).



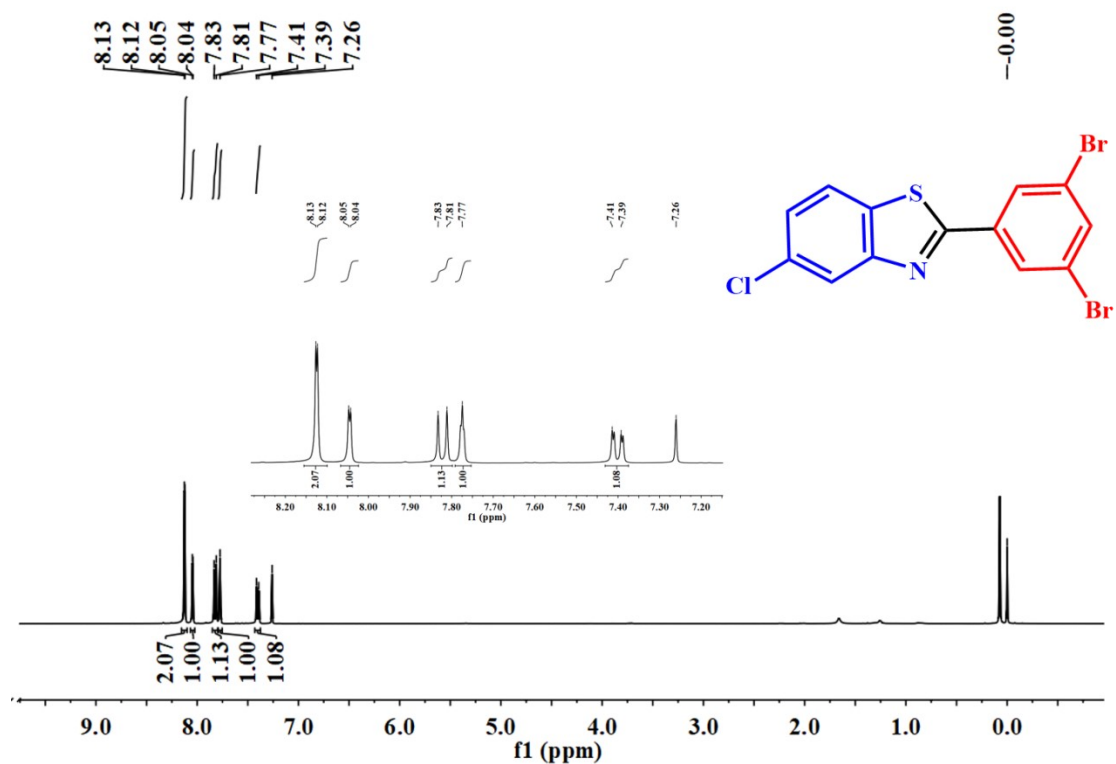
The ¹³C-NMR spectrum of 5-chloro-2-(4-methoxyphenyl)benzo[d]thiazole (**4d**).



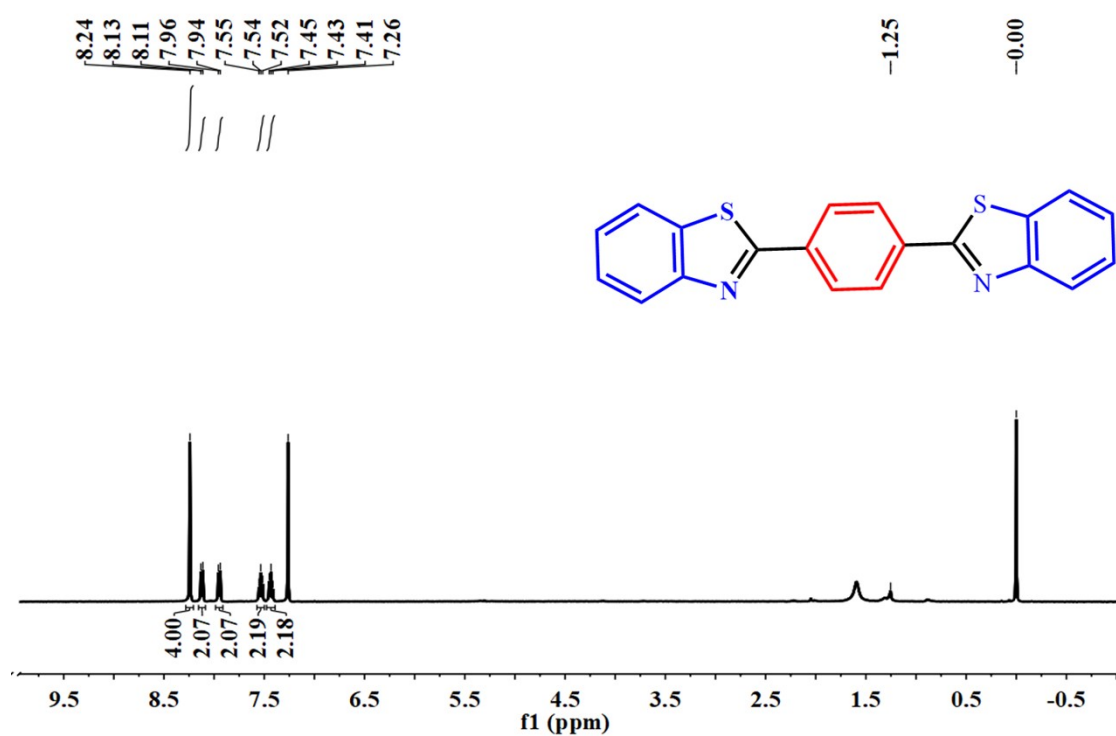
The ¹H-NMR spectrum of 5-chloro-2-(*p*-tolyl)benzo[d]thiazole (**4e**).



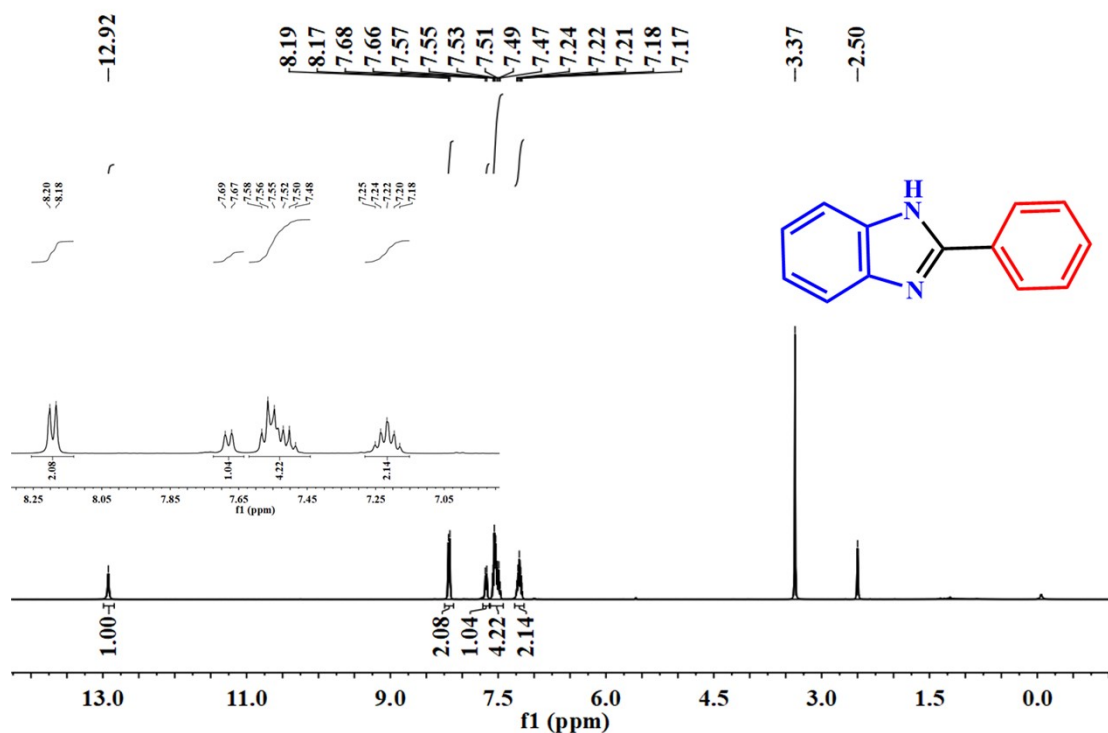
The ¹³C-NMR spectrum of 5-chloro-2-(*p*-tolyl)benzo[d]thiazole (**4e**).



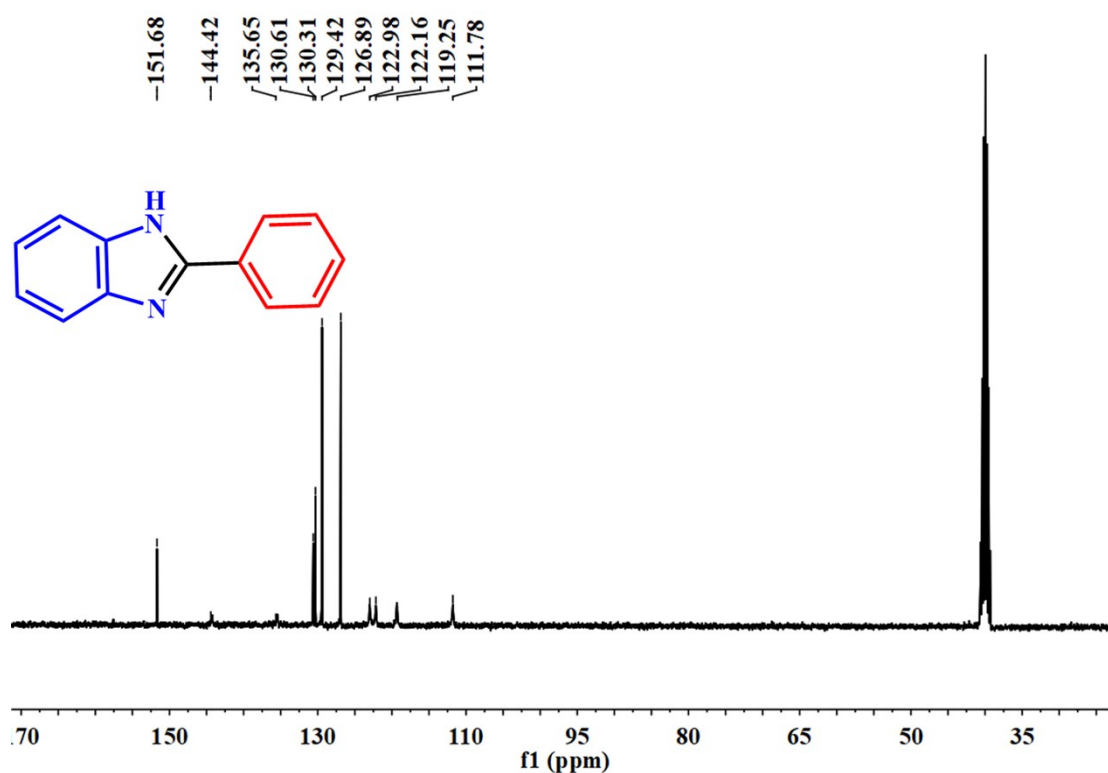
The ¹H-NMR spectrum of 5-chloro-2-(3,5-dibromophenyl)benzo[d]thiazole (**4f**).



The ¹H-NMR spectrum of 1,4-bis(benzo[d]thiazol-2-yl)benzene (**4g**).



The ¹H-NMR spectrum of 2-phenyl-1H-benzo[d]imidazole.



The ¹³C-NMR spectrum of 2-phenyl-1H-benzo[d]imidazole.

Section S8. Notes and references

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