Supporting Information

Triplet State Stabilization and Synergistic Energy Transfer in BOPHY-Functionalized NU-1000 for Enhanced Photocatalytic Aerobic Oxidation

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Materials and instrumentations

All chemicals were obtained from commercial sources and used without further purification.

The Powder X-ray diffraction (PXRD) patterns were measured on a Rigaku SmartLab diffractometer using Cu Kα radiation. Proton (^{1}H) and carbon (^{13}C) NMR spectra were recorded on a Bruker AVANCE III HD spectrometer. N_2 sorption isotherms were measured at 77 K using a BELSORP MAX (Micromeritics) gas adsorption instrument.

1. Synthesis of BOPHY Ligand

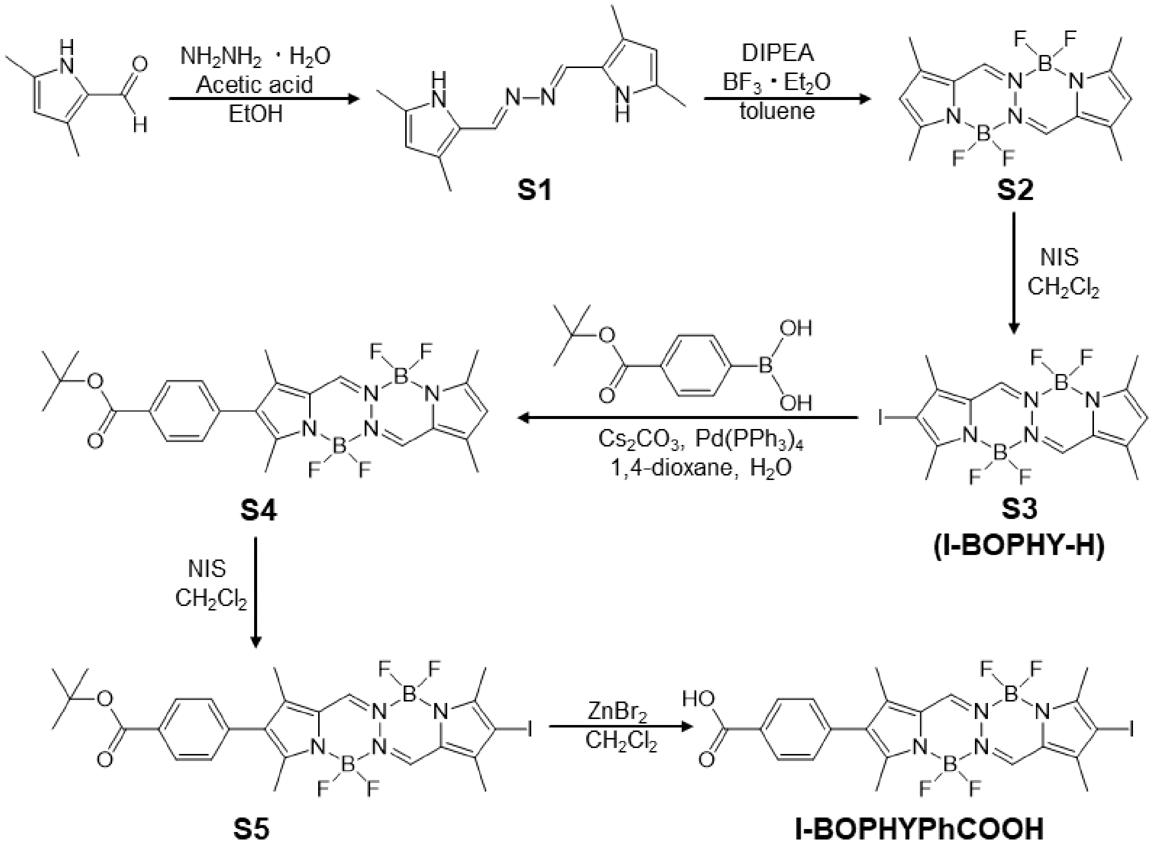


Fig S1 The schematic diagram of the synthesis of **I-BOPHYPhCOOH**

(1) Synthesis of S1- S3

S1, S2 and S3 were synthesized according to the previously reported methods.[1]

(2) Synthesis of S4

S3 (550 mg, 1.18 mmol), Cs_2CO_3 (1.2 g, 3.67 mmol, 3 eq) and 4 - (tert-butoxycarbonyl) phenylboronic acid (665 mg, 2.95 mmol, 2.5 eq) were added to a mixture of 1,4-dioxane (170 mL) and water (17 mL). The solution was bubbled with N_2 for around 30 min. Subsequently, $Pd(PPh_3)_4$ (150 mg, 0.13 mmol, 0.1 eq) was added and the reaction mixture was refluxed at 120°C for 2h or until the reaction is complete. Then the reaction solvent was completely removed using rotary evaporator. The reaction mixture was extracted with CH_2Cl_2 (200 mL) and water (200 mL). Then the crude product was purified by column chromatography (2CH_2Cl_2 : hexane) followed by (ethyl acetate : 4hexane).

Yellow powder; Yield = 62%.

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) = 8.08 (d, 2H, *J* = 8 Hz), 8.04 (s, 1H), 7.97 (s, 1H), 7.31 (d, 2H, *J* = 8 Hz), 2.51 (s, 3H), 2.47 (s, 3H), 2.35 (s, 3H), 2.29 (s, 3H), 1.62 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) = 165.48, 151.85, 148.78, 141.53, 137.46, 137.17, 135.06, 134.66, 131.01, 129.68, 129.62, 122.95, 118.77, 81.18, 28.23, 14.15, 12.90, 11.12, 10.02. ^{19}F NMR (376 MHz, $CDCl_3$): δ (ppm) = -141.91 to -142.41 (m).

(3) Synthesis of S5

S4 (200 mg, 0.39 mmol) and NIS (262.6 mg, 1.17 mmol) were dissolved in CH_2Cl_2 (100 mL) then stirred for 48h at room temperature. The crude mixture was purified by column chromatography (2CH_2Cl_2 : hexane).

Orange powder; Yield = 85%.

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) = 8.09 (d, 2H, *J* = 8 Hz), 8.03 (s, 1H), 8.02 (s, 1H), 7.31 (d, 2H, *J* = 8 Hz), 2.56 (s, 3H), 2.48 (s, 3H), 2.32 (s, 3H), 2.29 (s, 3H), 1.62 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) = 165.44, 151.60, 150.06, 142.98, 138.19, 137.14, 135.13, 131.12, 129.74, 129.60, 127.06, 123.29, 123.13, 81.26, 28.23, 15.13, 13.57, 13.03, 10.11. ^{19}F NMR (376 MHz, $CDCl_3$): δ (ppm) = -141.83 to -142.13 (m);

(4) Synthesis of I-BOPHYPhCOOH

I-BOPHYPhCOOH was synthesized according to the previously reported methods.[2] S5 (120 mg, 0.19 mmol) and $ZnBr_2$ (127 mg, 0.56 mmol) were dissolved in CH_2Cl_2 (10 mL) and stirred for 12h at room temperature. The reaction solvent was removed using rotary evaporator. The solid mixture was washed with water and the pure product was isolated by centrifugation.

Orange powder; Yield = 96%.

^{1}H NMR (400 MHz, DMSO-*d₆*): δ (ppm) = 8.57 (s, 1H), 8.52 (s, 1H), 8.07 (d, 2H, *J* = 8 Hz), 7.53 (d, 2H, *J* = 8 Hz), 2.51 (s, 3H), 2.47 (s, 3H), 2.38 (s, 3H), 2.37 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6): δ (ppm) = 167.58, 150.76, 149.40, 144.19, 139.57, 137.46, 137.28, 130.47, 130.29, 130.01, 123.41, 123.13, 82.38, 15.30, 13.75, 13.36, 10.41; ^{19}F NMR (376 MHz, $CDCl_3$): δ (ppm) = -141.16 to -141.40 (m).

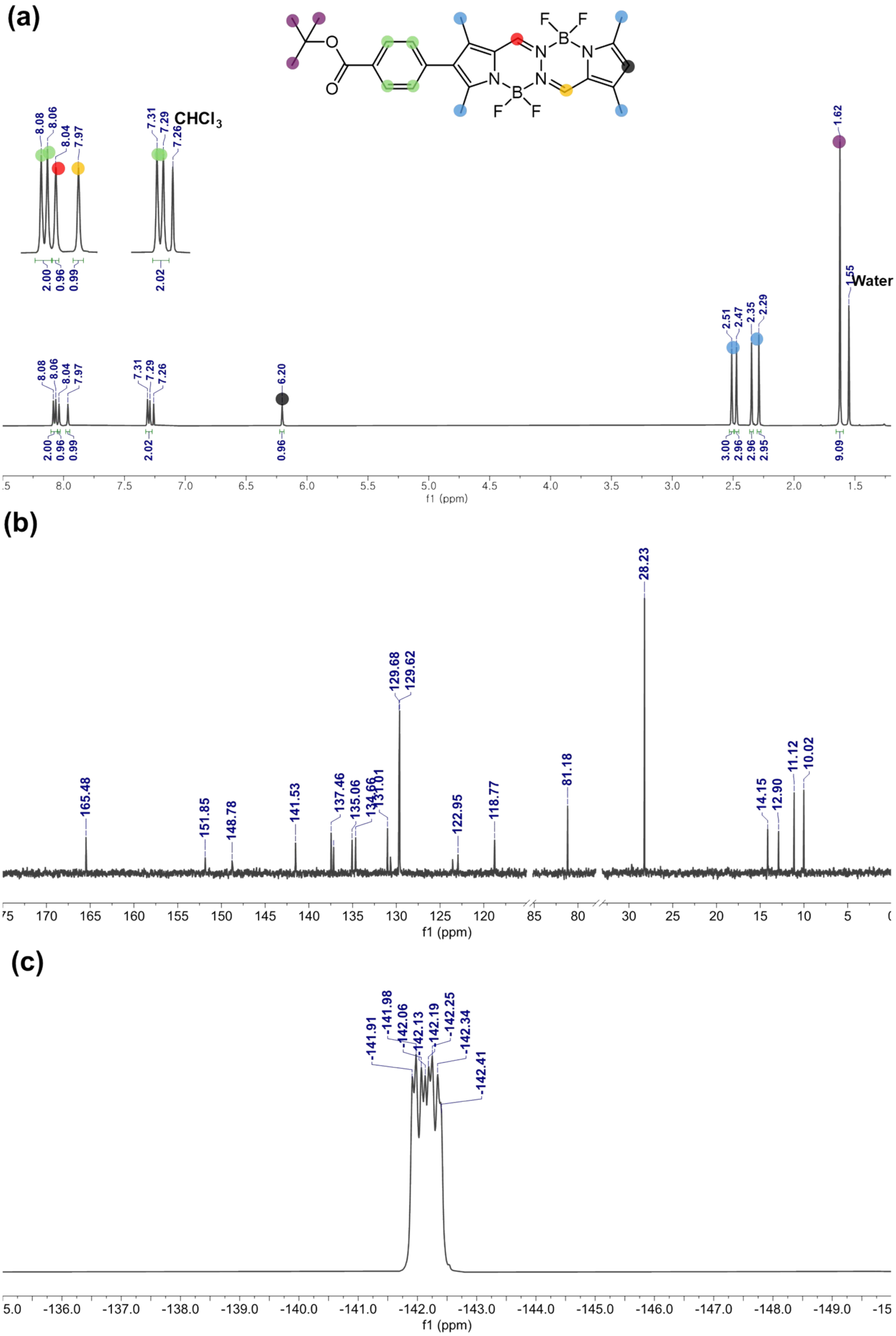


Fig S2 **(a)** ^{1}H NMR, **(b)** ^{13}C NMR, **(c)** ^{19}F NMR spectra of S4 in $CDCl_3$.

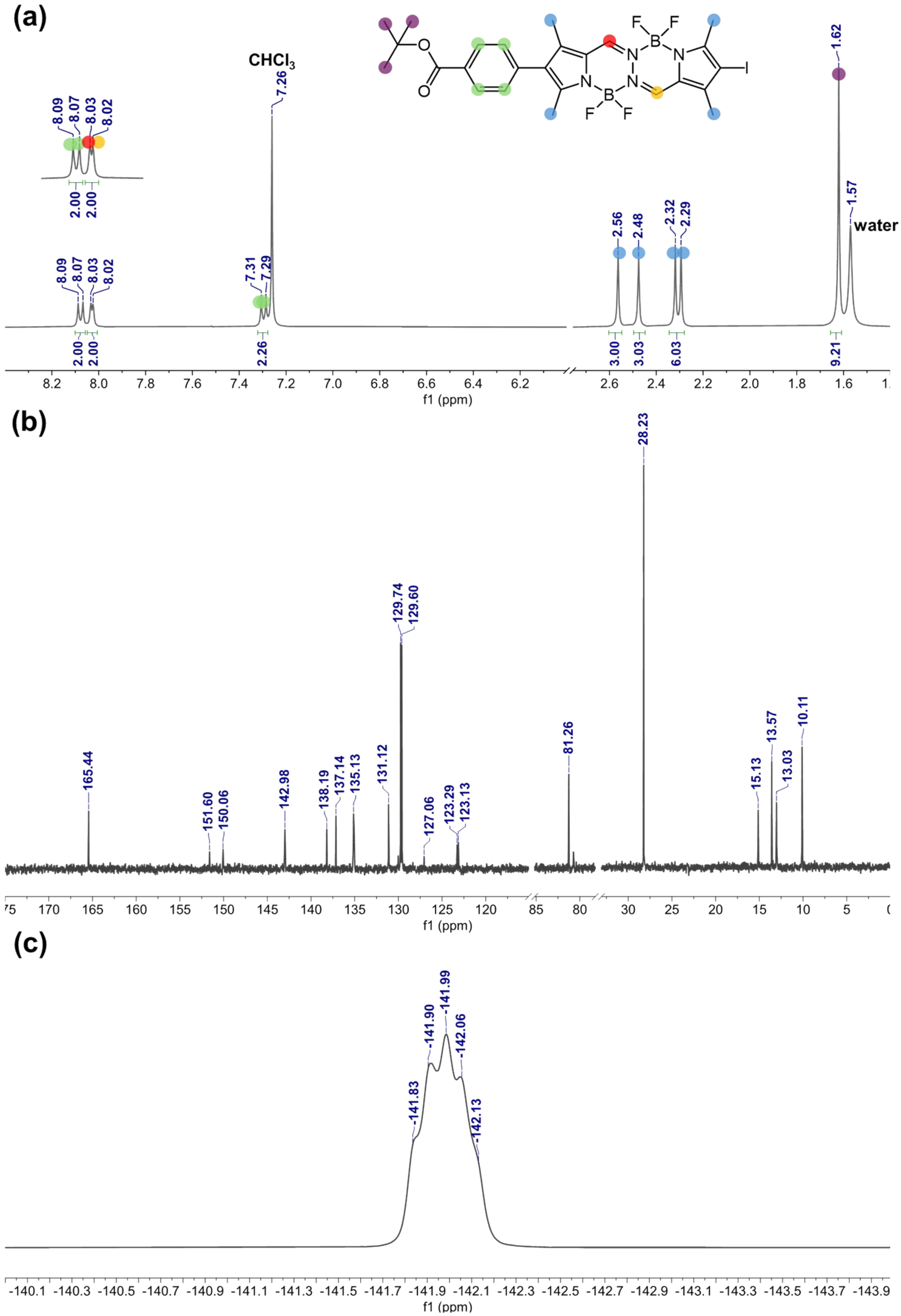


Fig S3 **(a)** ^{1}H NMR, **(b)** ^{13}C NMR, **(c)** ^{19}F NMR spectra of S5 in CDCl$_3$.

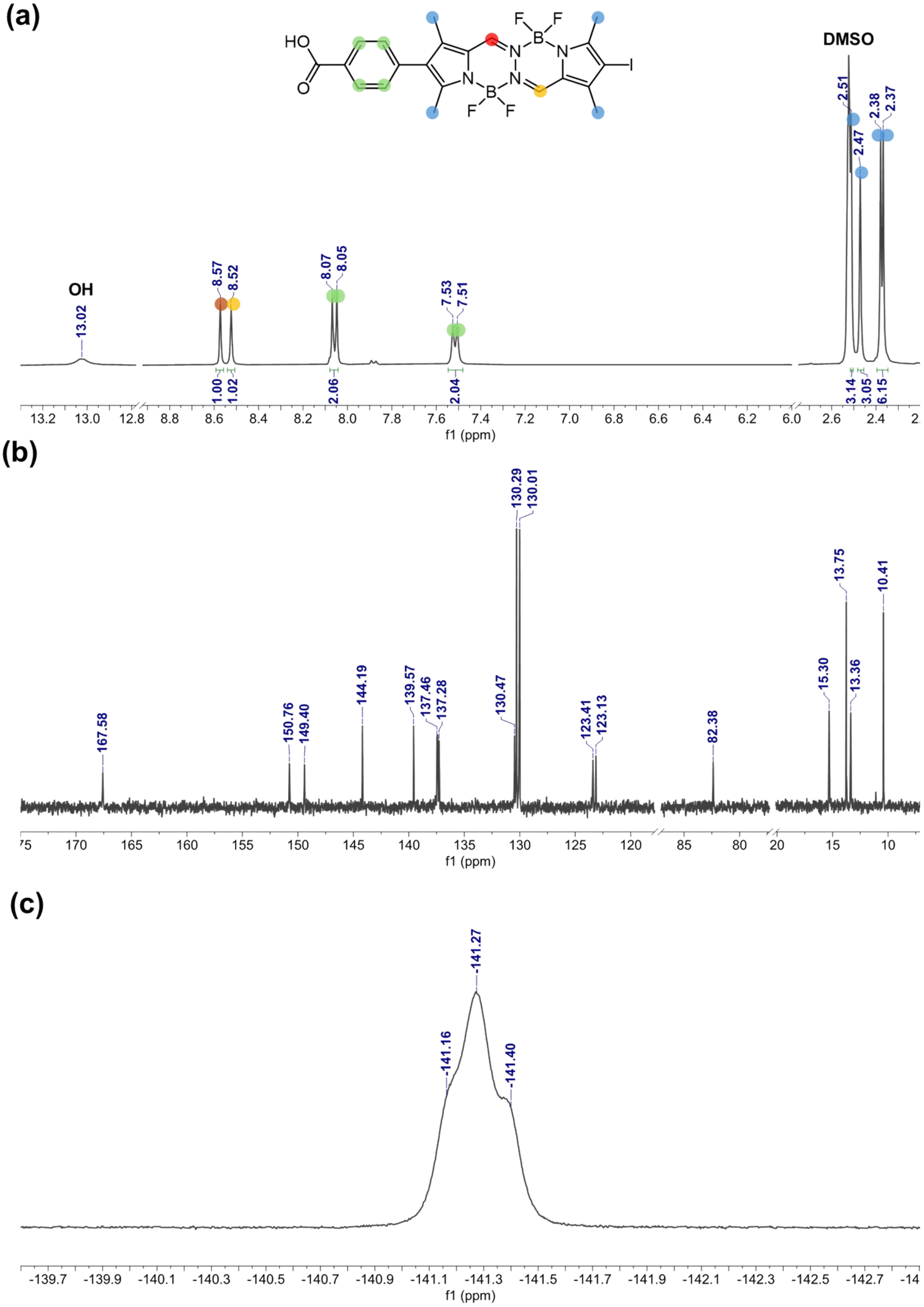


Fig S4 (a) ^{1}H NMR, (b) ^{13}C NMR, (c) ^{19}F NMR spectra of **I-BOPHYPhCOOH** in $CDCl_3$.

2. Synthesis of H_4TBAPy and NU-1000 1,3,6,8-Tetrakis-(*p*-benzoic acid)pyrene (H_4TBAPy) and NU-1000 were synthesized according to the reported methods.[3–5]

Activation of NU-1000. Approximately 25 mg of NU-1000 sample was dispersed in DMF (12 mL) in a 20 mL vial. Subsequently, HCl (0.5 mL, 8 M) was added, and the mixture was sonicated for approximately 10 min. The sample was heated in an oven at 100 ºC for 24 h. Upon completion, the resulting solid was collected by centrifugation, washed with DMF (25 mL, 3 times) and acetone (25 mL, 3 times), and dried in a vacuum oven for 12 h prior to use

3. Synthesis of I-BOPHY@NU-1000

I-BOPHY@NU-1000 were synthesized by SALI method. For the preparation of **I-BOPHY@NU-1000** (1eq), 90 mg of NU-1000 (0.021 mmol, activated with HCl) and an equimolar amount of **I-BOPHYPhCOOH** ligand were added to 6 mL of DMF. The mixture was capped and sonicated for 10 minutes to ensure complete dissolution of the ligand and uniform dispersion of the NU-1000 particles. Subsequently, the reaction mixture was heated at 60°C for 24 hr. After the reaction, synthesized **I-BOPHY@NU-1000** was washed with fresh DMF and acetone until the supernatant become colorless. The MOF particles were collected by centrifugation (10000 rpm, 5 min) and dried under a vacuum oven at 120 °C for 24 hr.

General photocatalytic experimental details

All Photocatalytic reactions were carried out using a Xe lamp (Abet Technologies, LS150, 150W/m^2) positioned at a distance of 15cm. The reaction was conducted under aerobic conditions at room temperature with stirring at 1100 rpm. After the reaction, MOF particles were separated by filtration through a compact cotton column. Then reaction solvent (MeOH) was removed using rotary evaporation. Without any purification, crude ^{1}H NMR spectra analysis was performed in $CDCl_3$. To compensate for the chemical shift, 150 µL of 1,1,2,2-tetrachloroethane was added as an internal standard which shows a singlet peak at 5.91 ppm. The TLC method (2 Ethyl acetate : Hexane) was used as an auxiliary means to observe the formed sulfide, sulfoxide, and sulfone.

The peak of A1 and its oxidation material A2 is marked in red and blue line in ^{1}H and ^{13}C NMR spectra respectively.

A1 ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) = 7.32-7.20 (m, 4H), 7.17-7.07(m, 1H), 2.47(s,3H); ^{13}C NMR(100 MHz, $CDCl_3$): δ (ppm) = 138.29, 128.72, 126.47, 124.91, 15.69;

A2 ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) = 7.62-7.60 (m, 2H), 7.51-7.44 (m, 3H), 2.68 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) = 145.58, 130.94, 129.25, 123.39, 43.78;

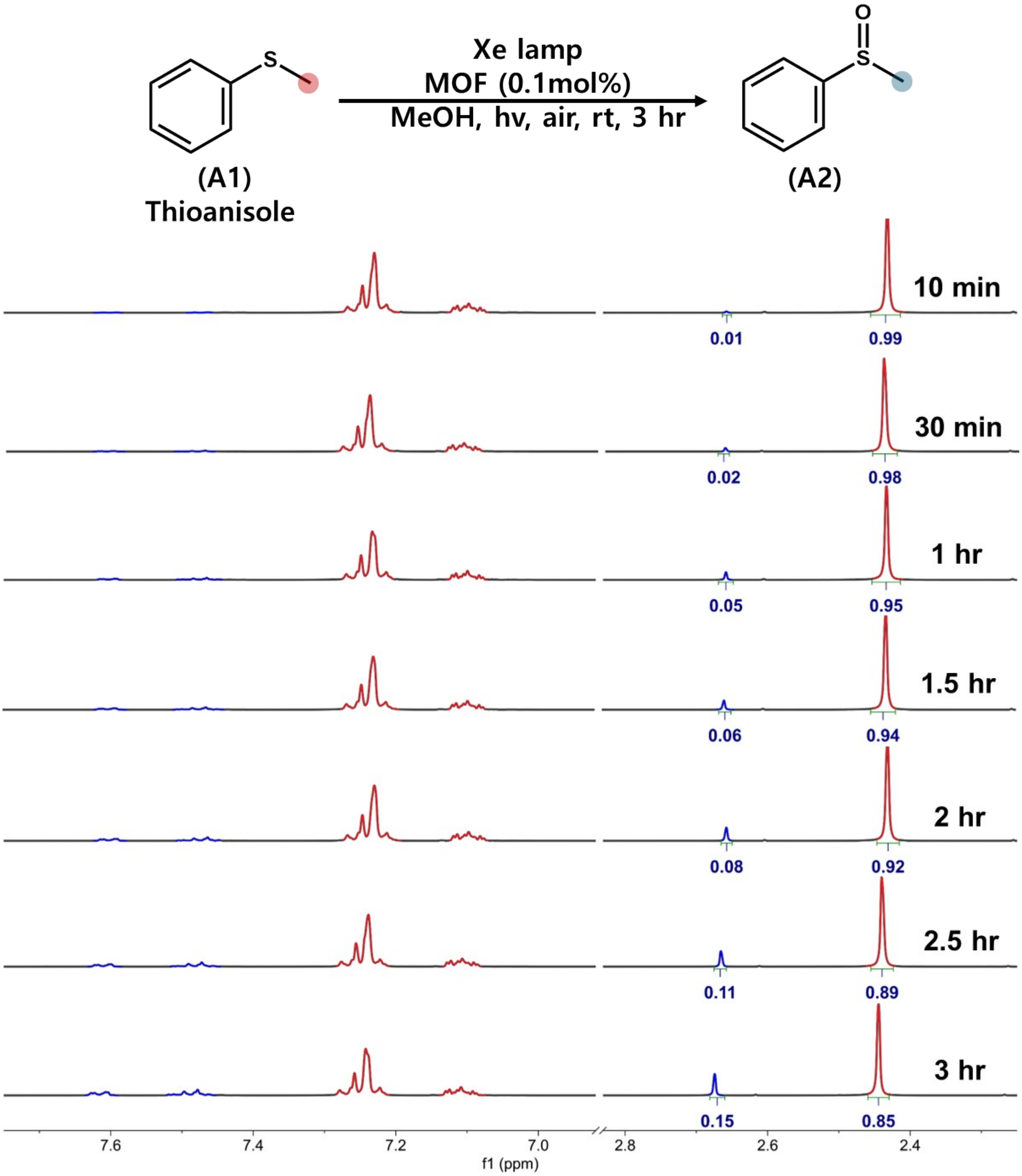


Fig S5 (a) Time-dependent ^{1}H NMR spectral changes during the photocatalytic reaction of NU-1000

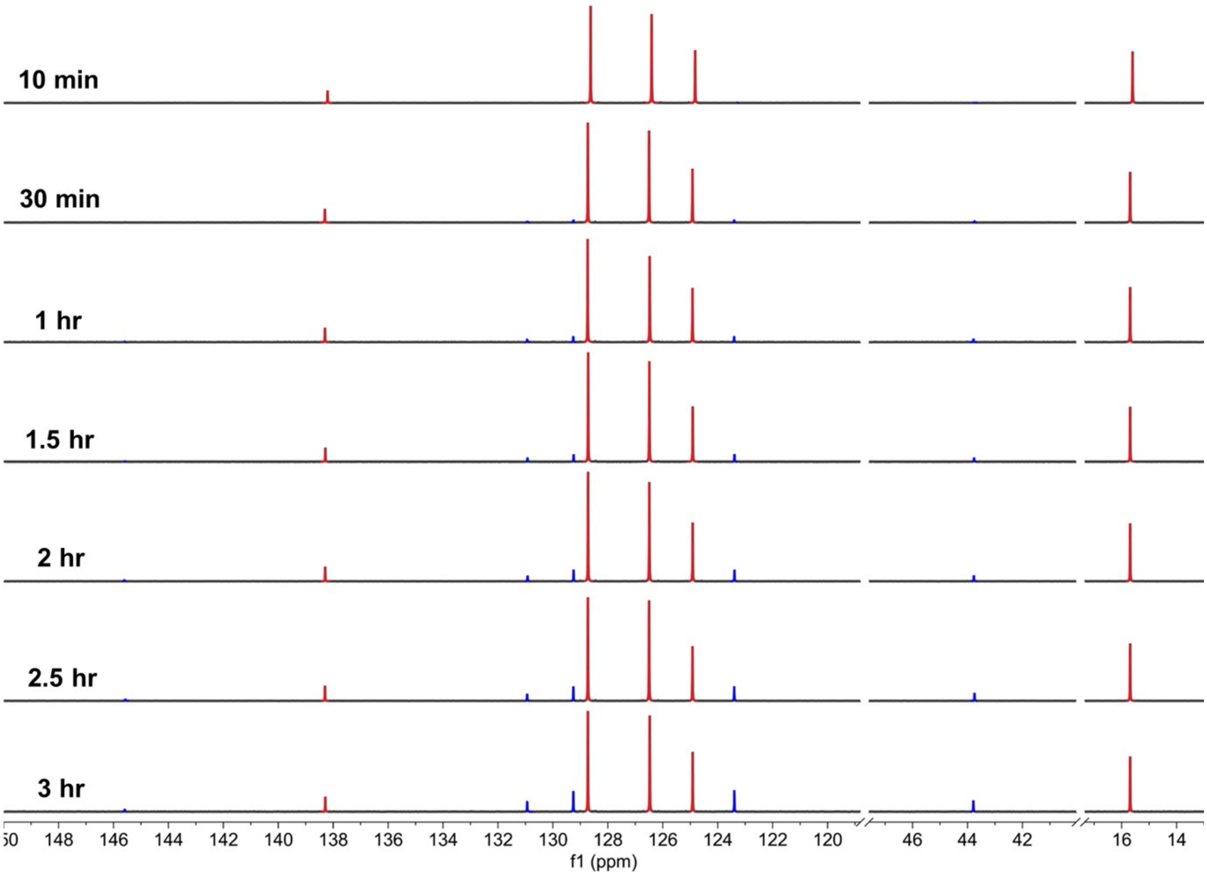


Fig S5 (b) Time-dependent ^{13}C NMR spectral changes during the photocatalytic reaction of NU-1000

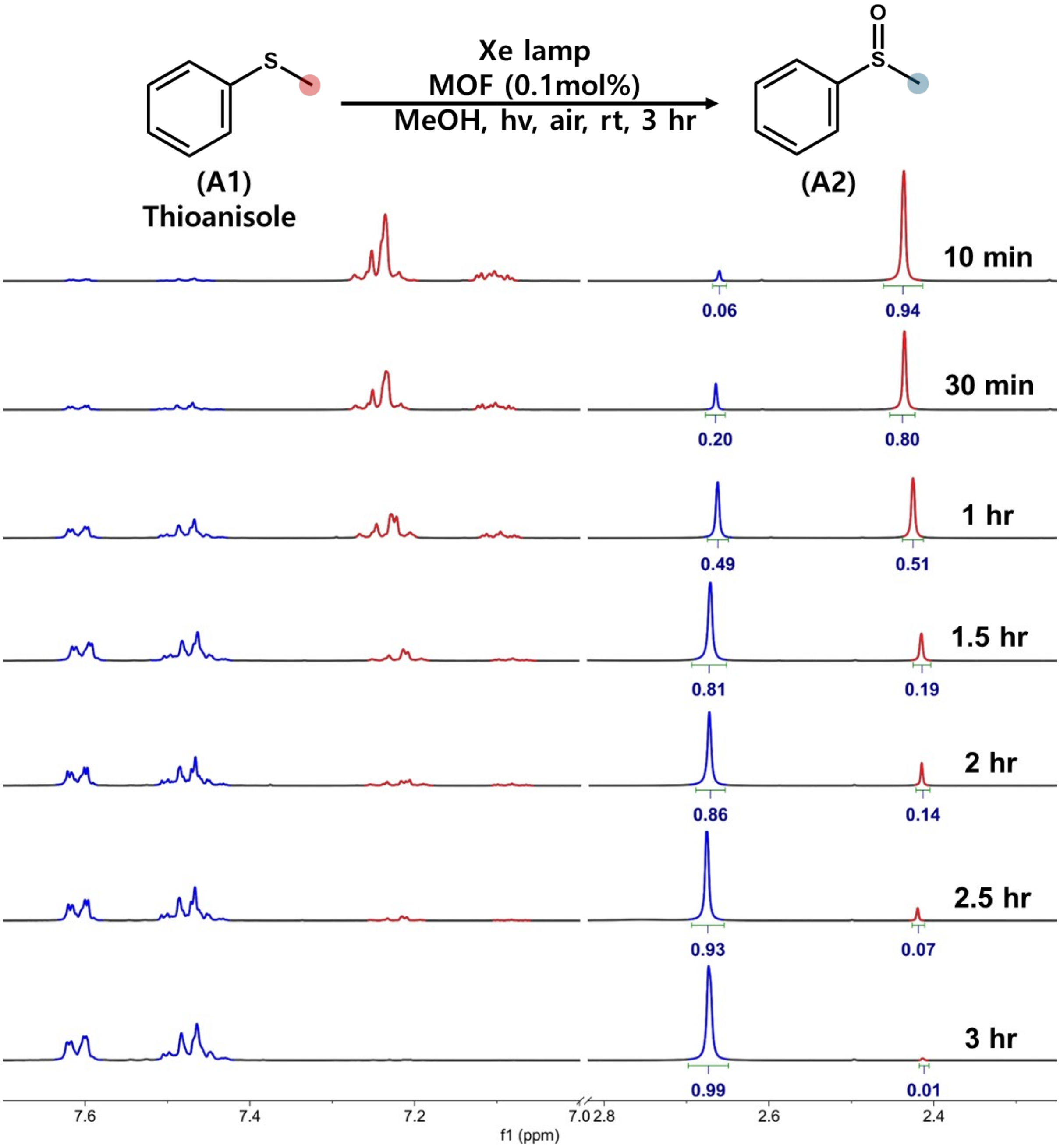


Fig S6 (a) Time-dependent ^{1}H NMR spectral changes during the photocatalytic reaction of **I-BOPHY@NU-1000** (1eq)

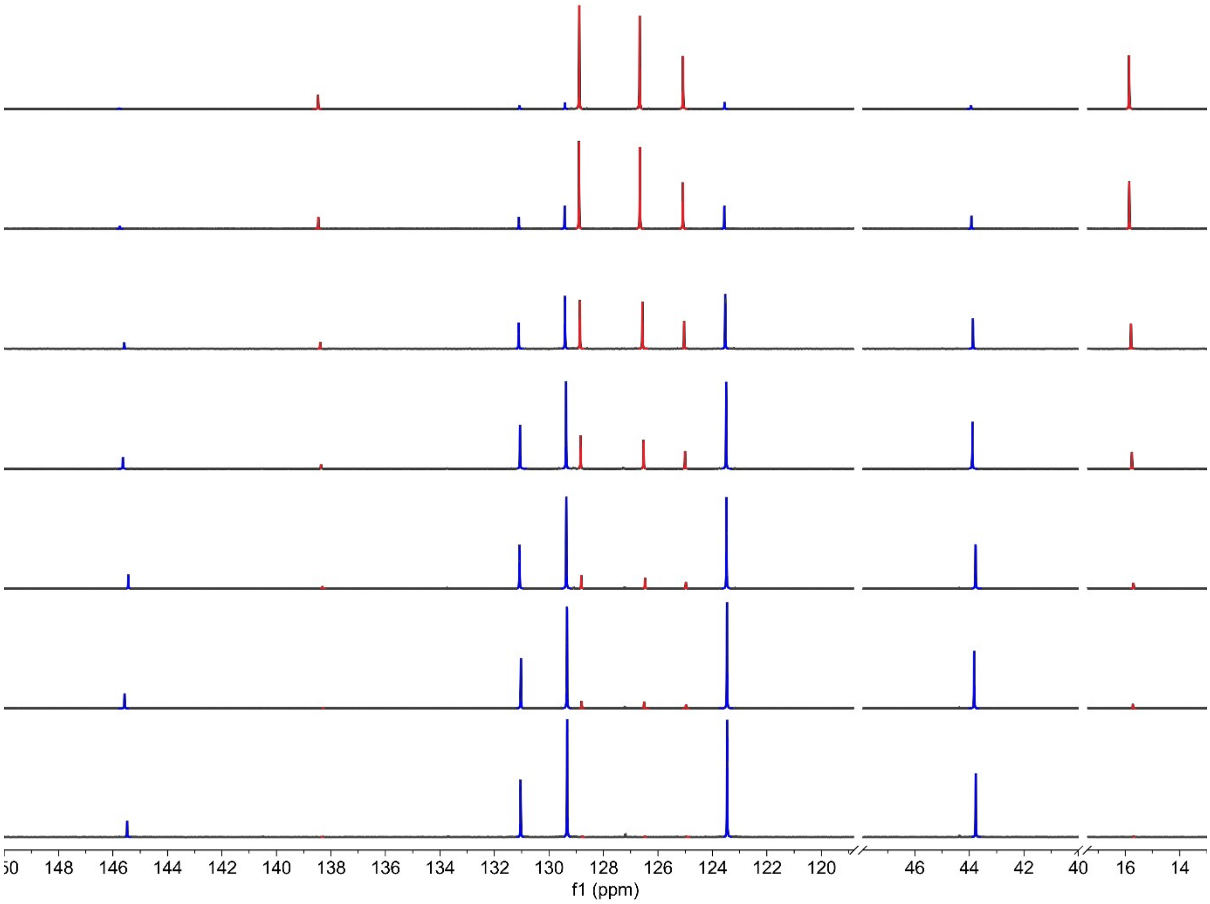


Fig S6 (b) Time-dependent ^{13}C NMR spectral changes during the photocatalytic reaction of **I-BOPHY@NU-1000** (1eq)

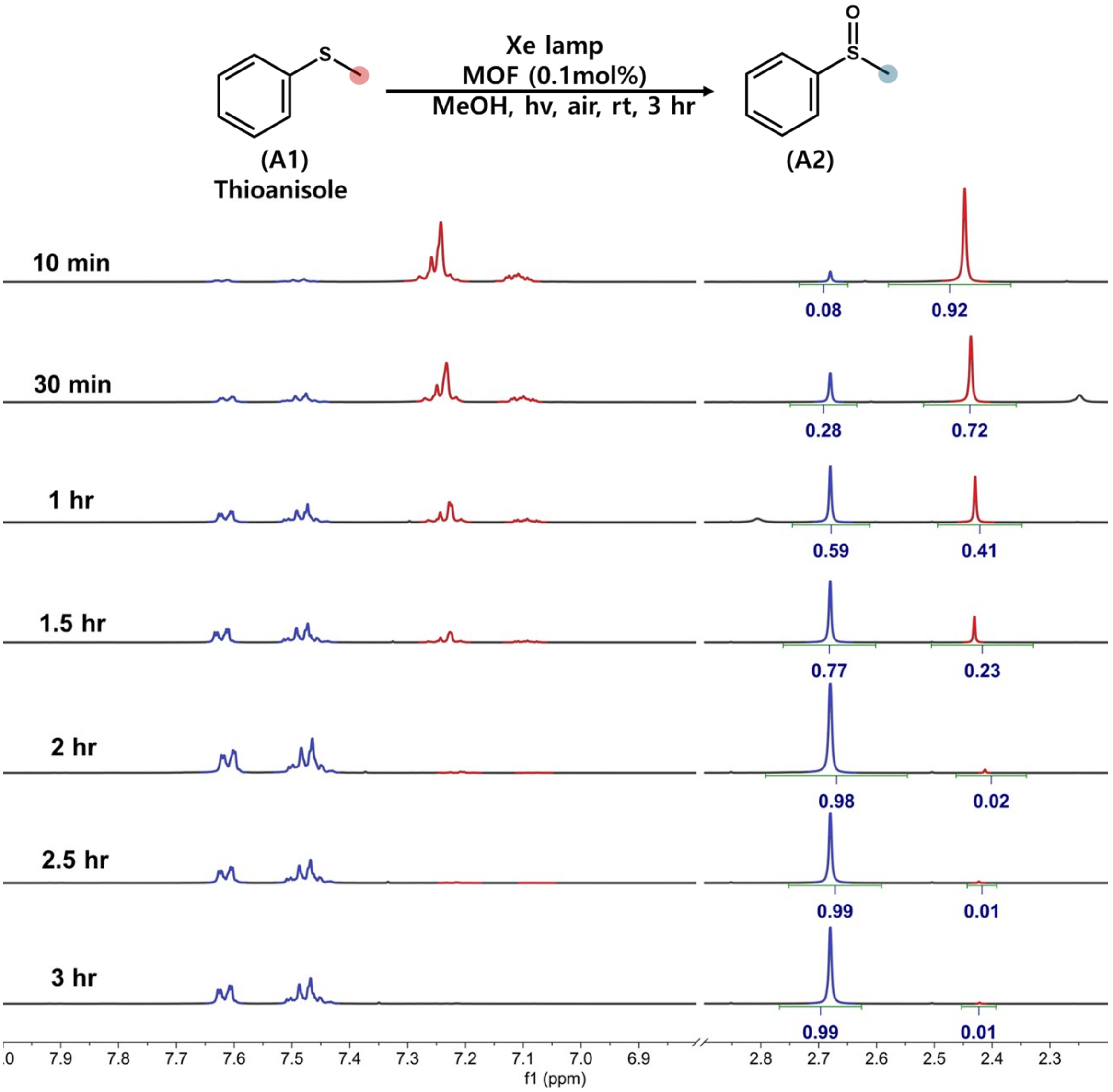


Fig S7 (a). Time-dependent ^{1}H NMR spectral changes during the photocatalytic reaction of I-BOPHYPhCOOH

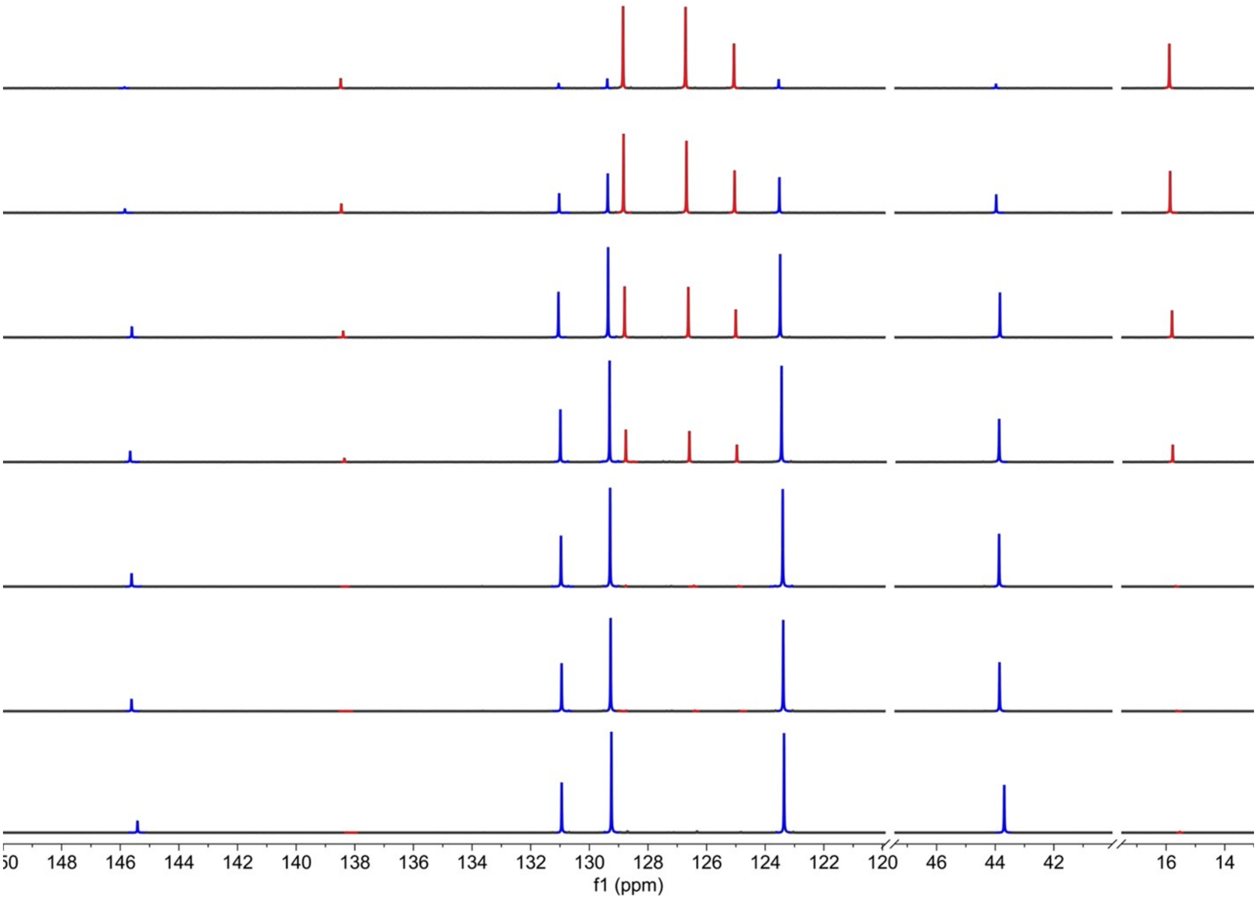


Fig S7b). Time-dependent ^{13}C NMR spectral changes during the photocatalytic reaction of **I-BOPHYPhCOOH**

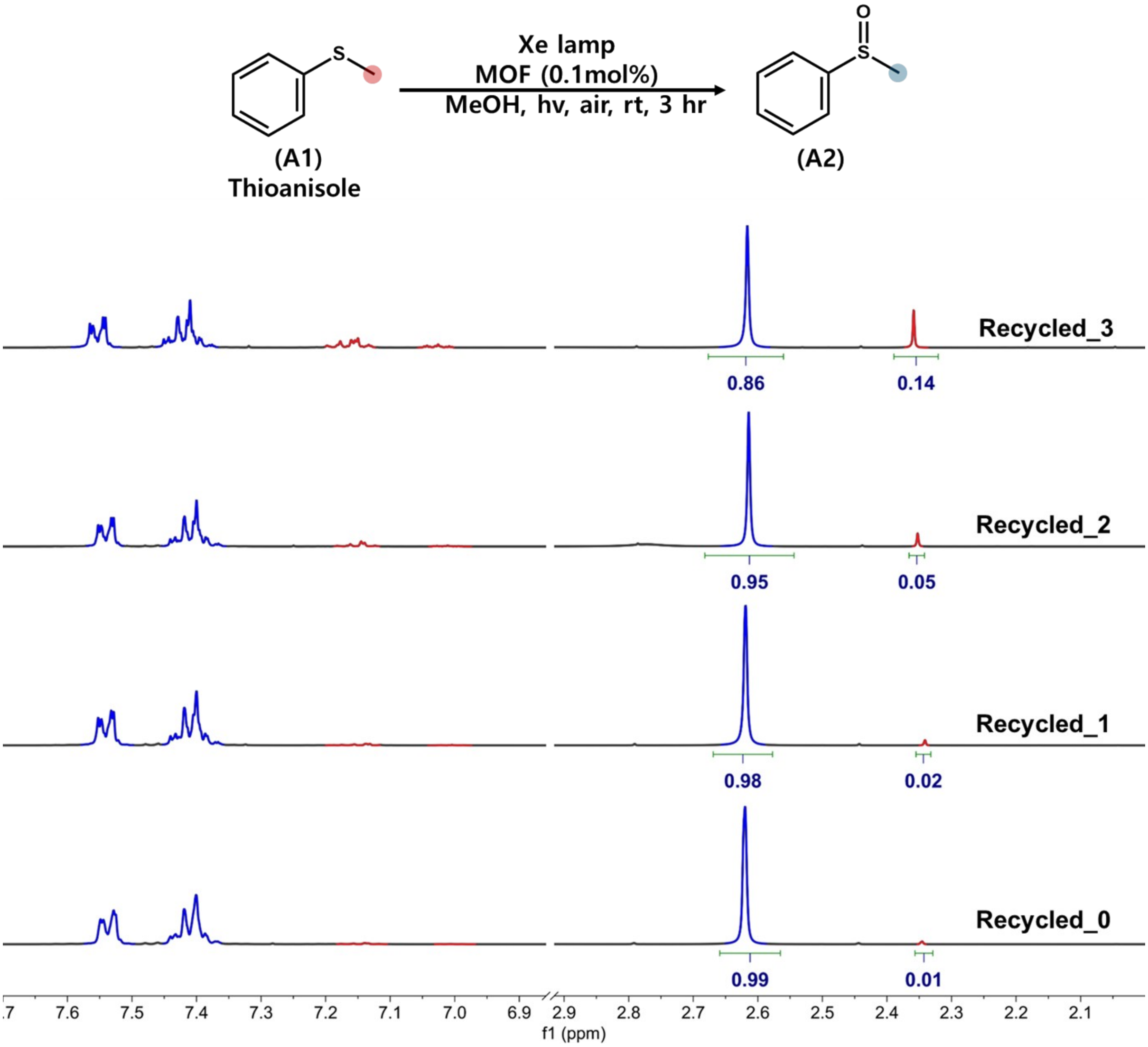


Fig S8 (a) ^{1}H NMR spectrums in each recycled photocatalytic reaction of **I-BOPHY@NU-1000** (1eq)

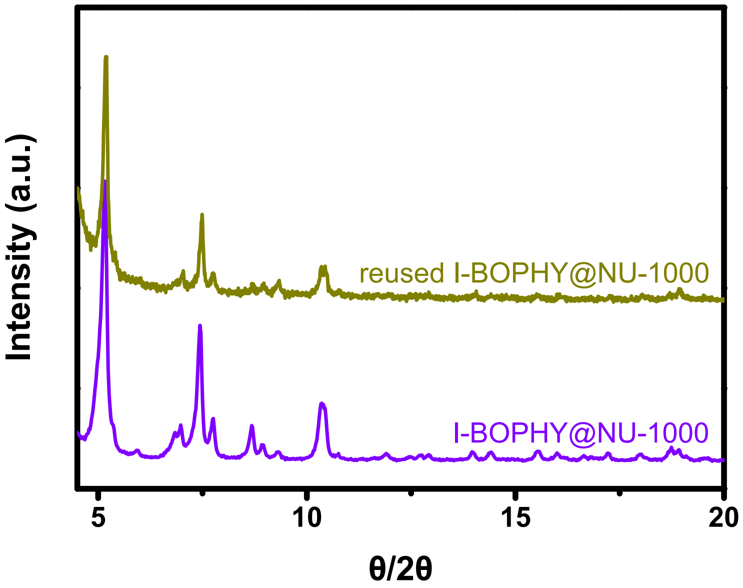


Fig S8 (b) PXRD patterns in each recycled photocatalytic reaction of **I-BOPHY@NU-1000**

Scavenger test

To prevent overlap in the NMR spectra between A1 and the scavengers, the final reaction mixture was processed as follows: The reaction solvent (methanol) was removed using a rotary evaporator, and 7 mL of dichloromethane was added to the residue. This solution was filtered through a compact cotton column to remove insoluble scavenger solids along with MOF particles. Despite these purification steps, slight chemical shifts were observed in compounds A1 and A2, likely due to interactions with residual scavenger materials that were not completely eliminated.

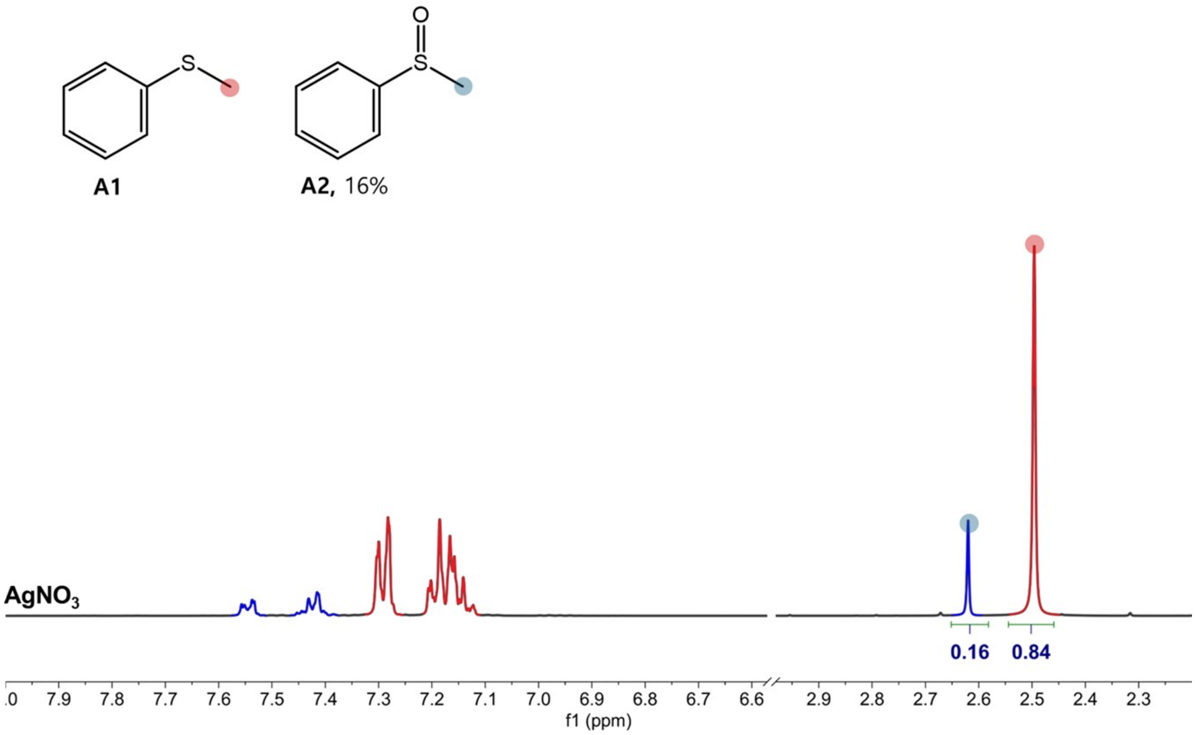


Fig S9 (a) ^{1}H NMR spectrum of trapping test with added AgNO$_3$

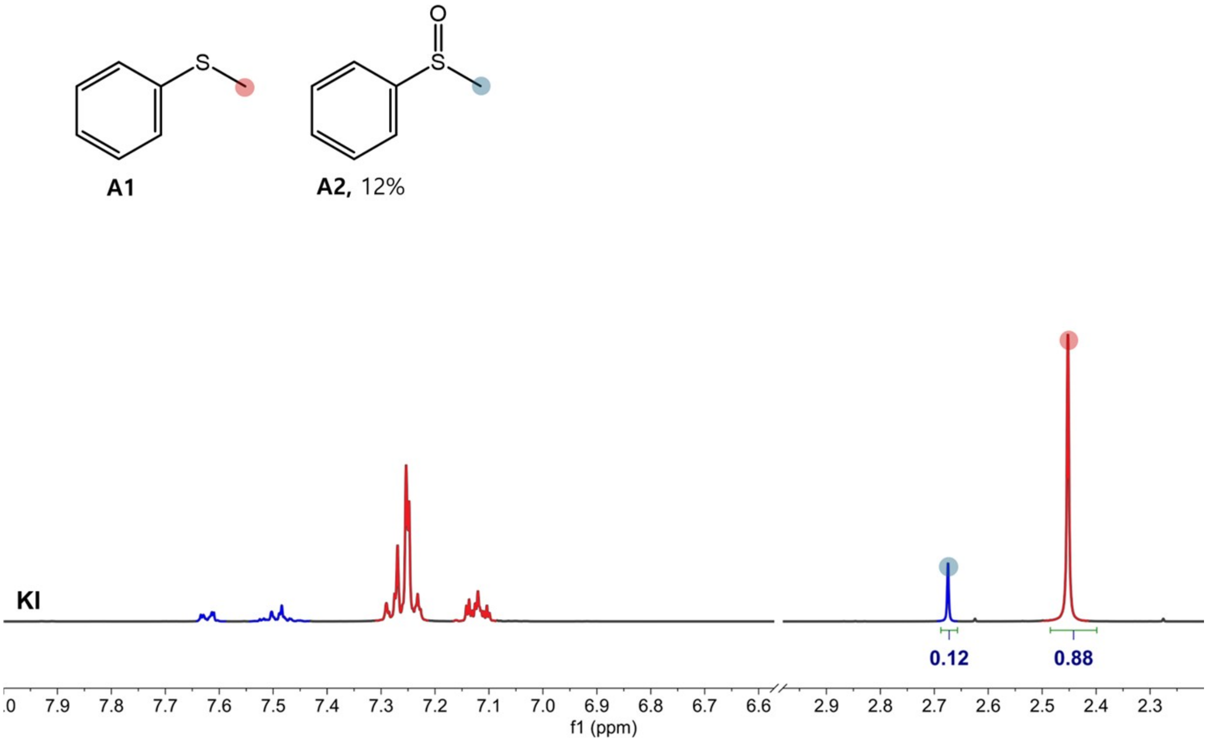


Fig S9 (b) ^{1}H NMR spectrum of trapping test with added KI

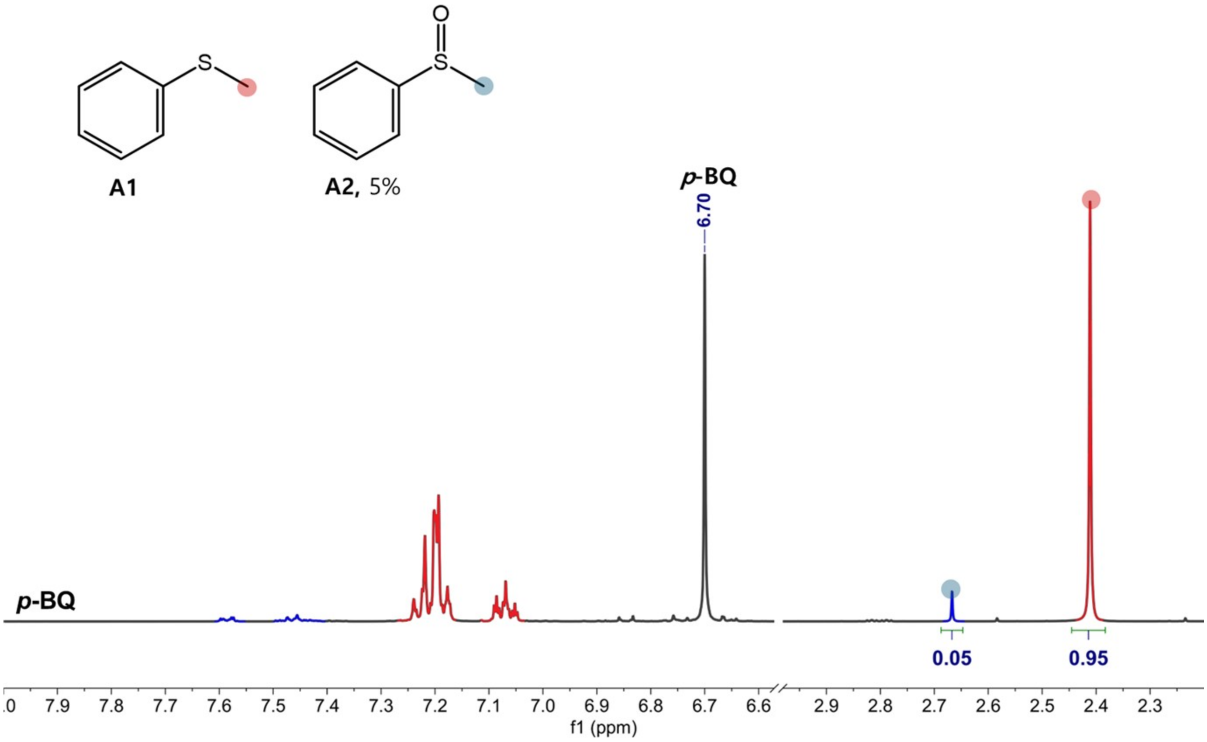


Fig S9 (c) ^{1}H NMR spectrum of trapping test with added *p*-BQ

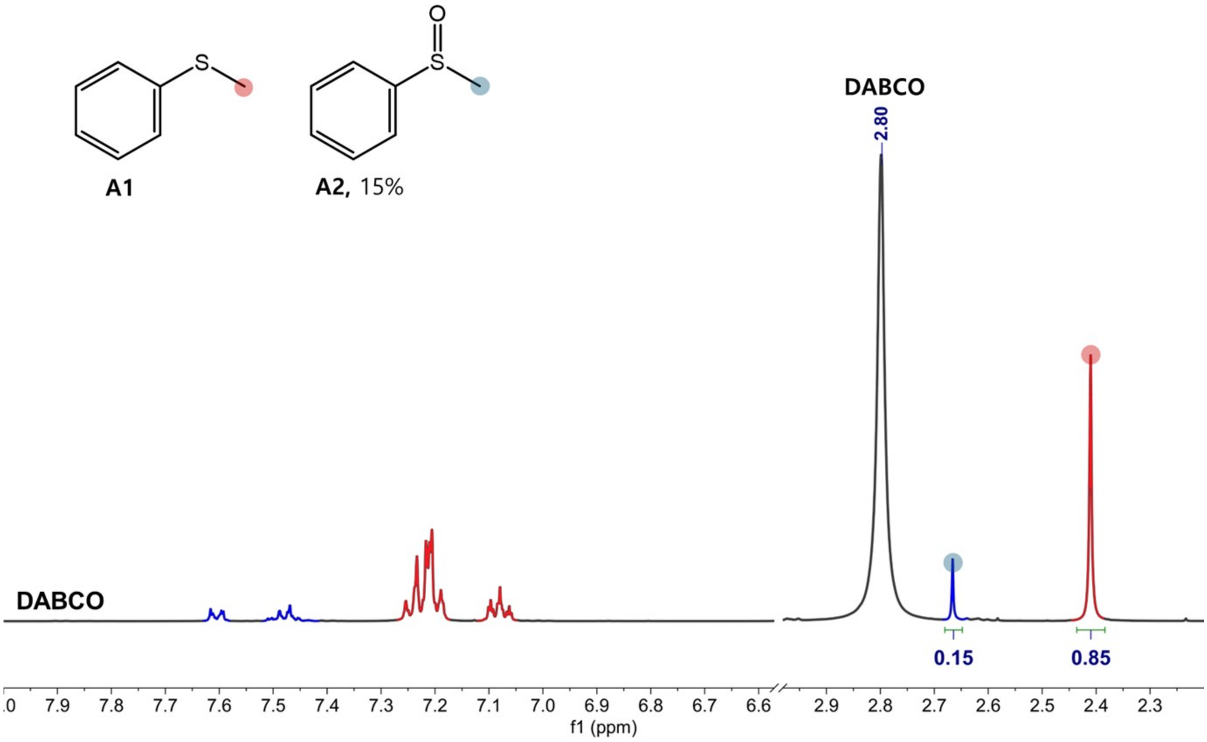


Fig S9 (d) ^{1}H NMR spectrum of trapping test with added DABCO

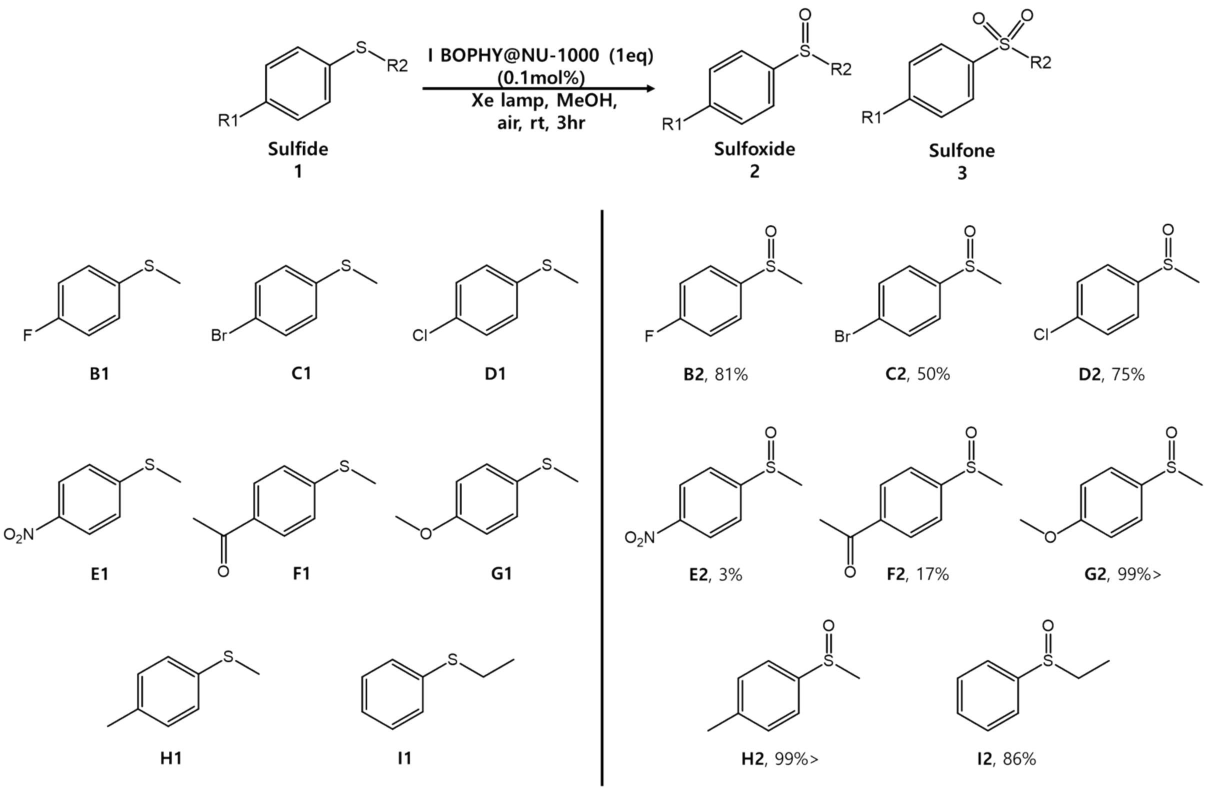
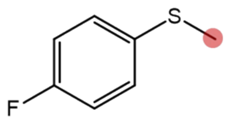
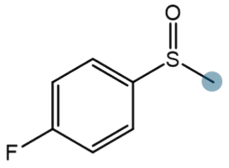


Fig S10 (a) The conversion rate was determined by ^{1}H NMR spectrums in the following figures.



B1



B2, 81%

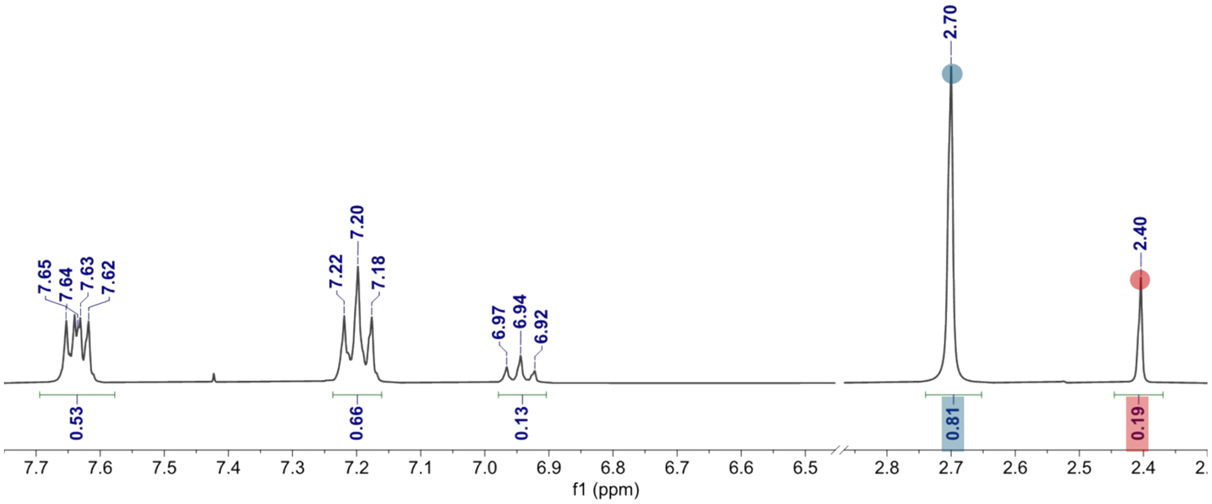
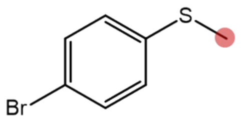
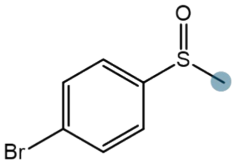


Fig S10 (b) ^{1}H NMR spectrum of B2 reaction

B2 ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) = 7.65-7.62 (m, 2H), 7.22-7.18 (m, 2H), 2.70 (S, 3H);



C1



C2, 50%

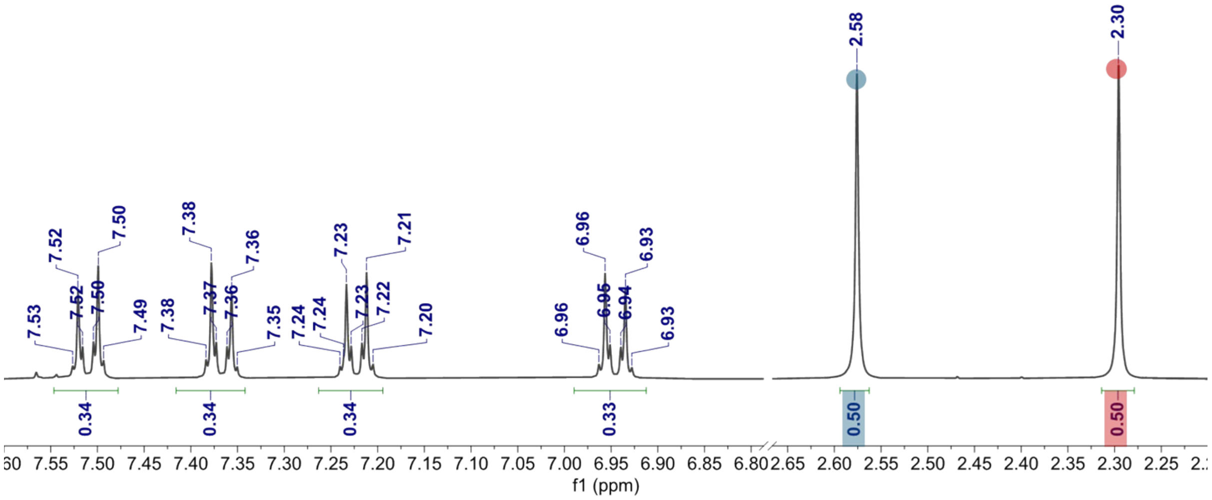
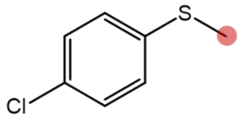
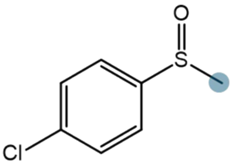


Fig S10 (c) ^{1}H NMR spectrum of C2 reaction

C2 ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) = 7.53-7.49 (m, 2H), 7.24-7.20 (m, 2H), 2.58 (S, 3H);



D1



D2, 75%

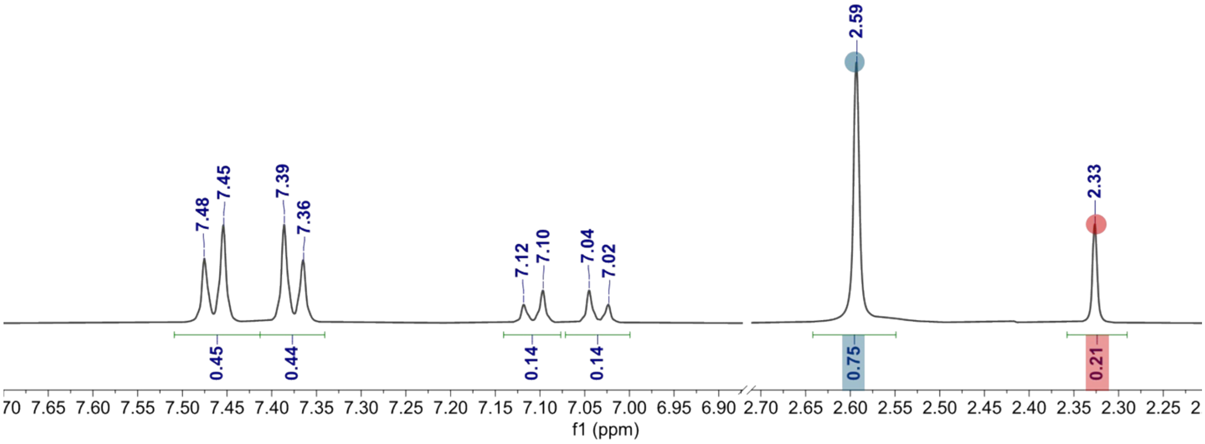


Fig S10 (d) ^{1}H NMR spectrum of D2 reaction

D2 ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) = 7.48 (d, 2H, *J*=12), 7.39 (d, 2H, *J*=12), 2.59 (s, 3H);

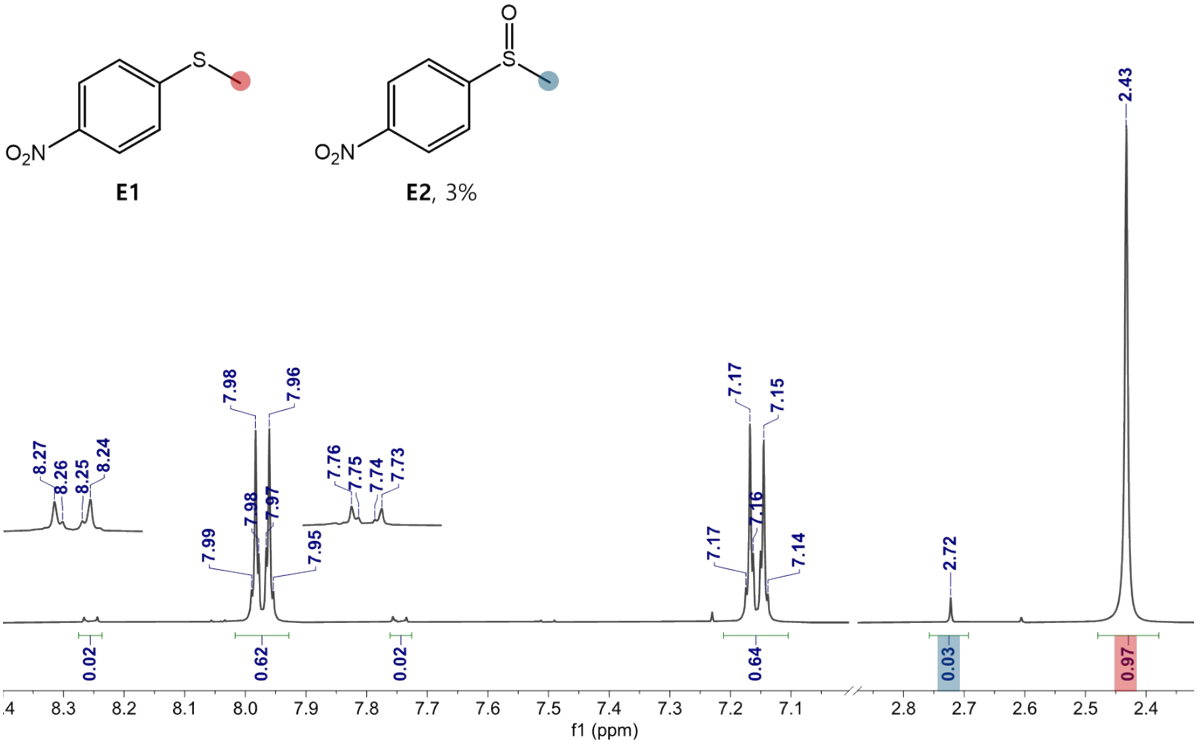


Fig S10 (e) ^{1}H NMR spectrum of E2 reaction

E2 ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) = 8.27-8.24 (m, 2H), 7.76-7.73 (m, 2H), 2.72 (s, 3H);

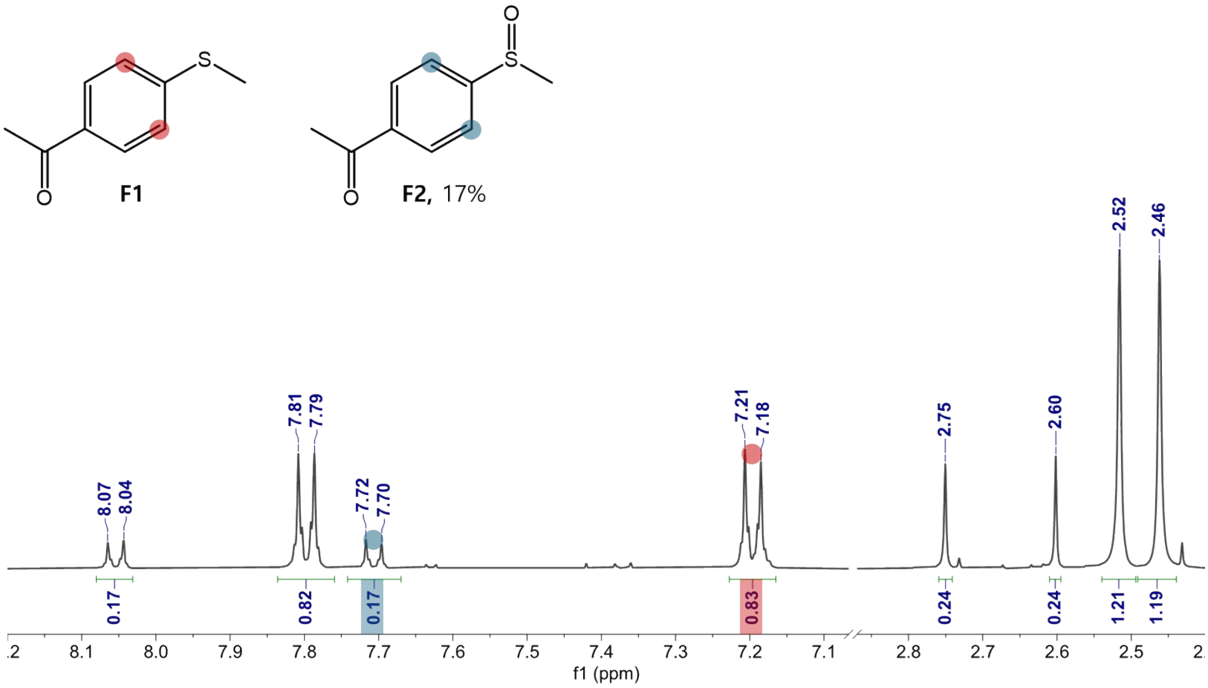


Fig S10 (f) ^{1}H NMR spectrum of F2 reaction

F2 ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) = 8.07-8.04 (m, 2H), 7.72-7.70 (m, 2H), 2.75(s, 3H), 2.60 (s, 3H);

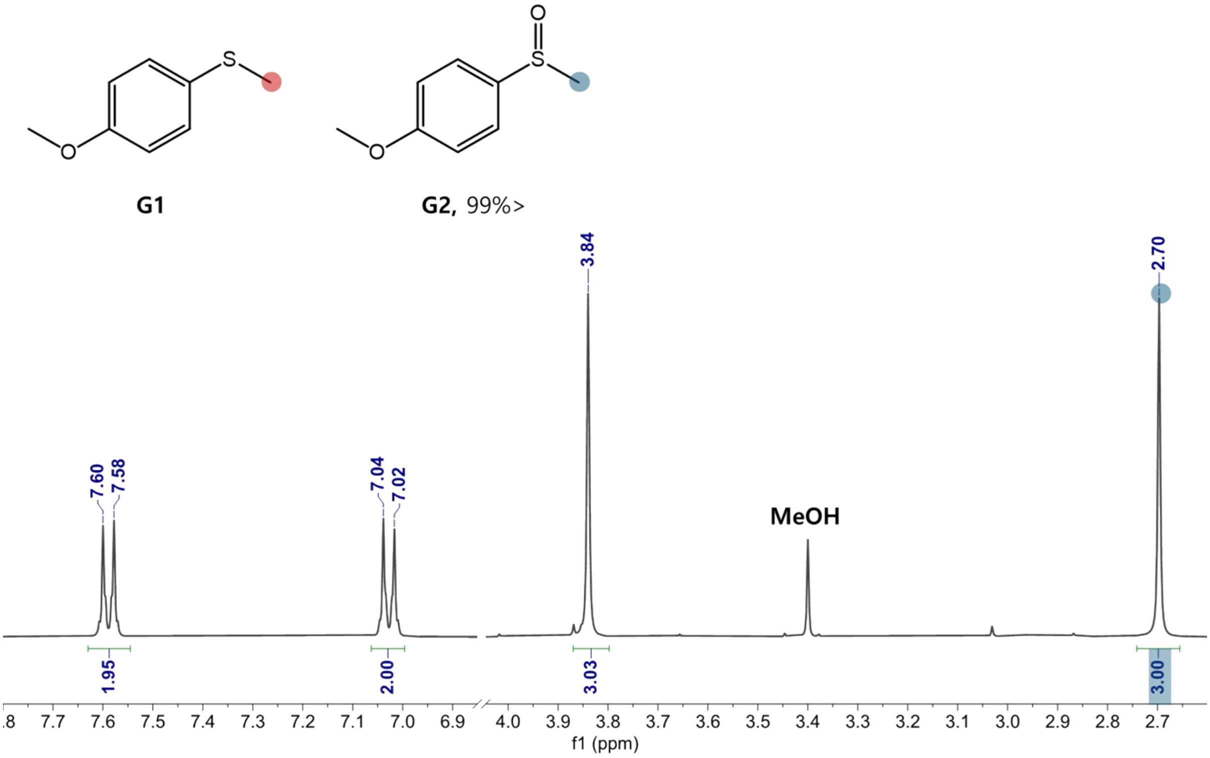


Fig S10 (g) ^{1}H NMR spectrum of G2 reaction

G2 ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) = 7.60-7.58 (m, 2H), 7.04-7.02 (m, 2H), 3.84 (s, 3H), 2.70 (s, 3H);

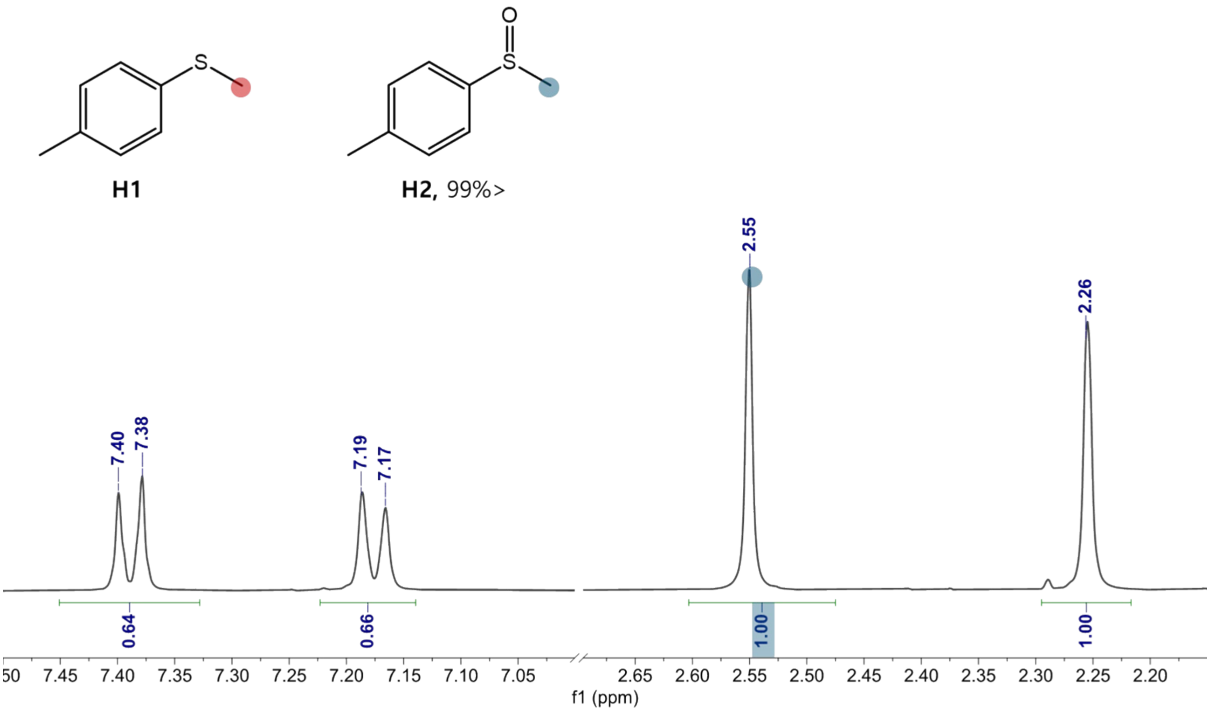


Fig S10 (h) ^{1}H NMR spectrum of H2 reaction

H2 ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) = 7.40 (d, 2H, J = 8), 7.19 (d, 2H, J = 8), 2.55 (s, 3H), 2.26 (s, 3H);

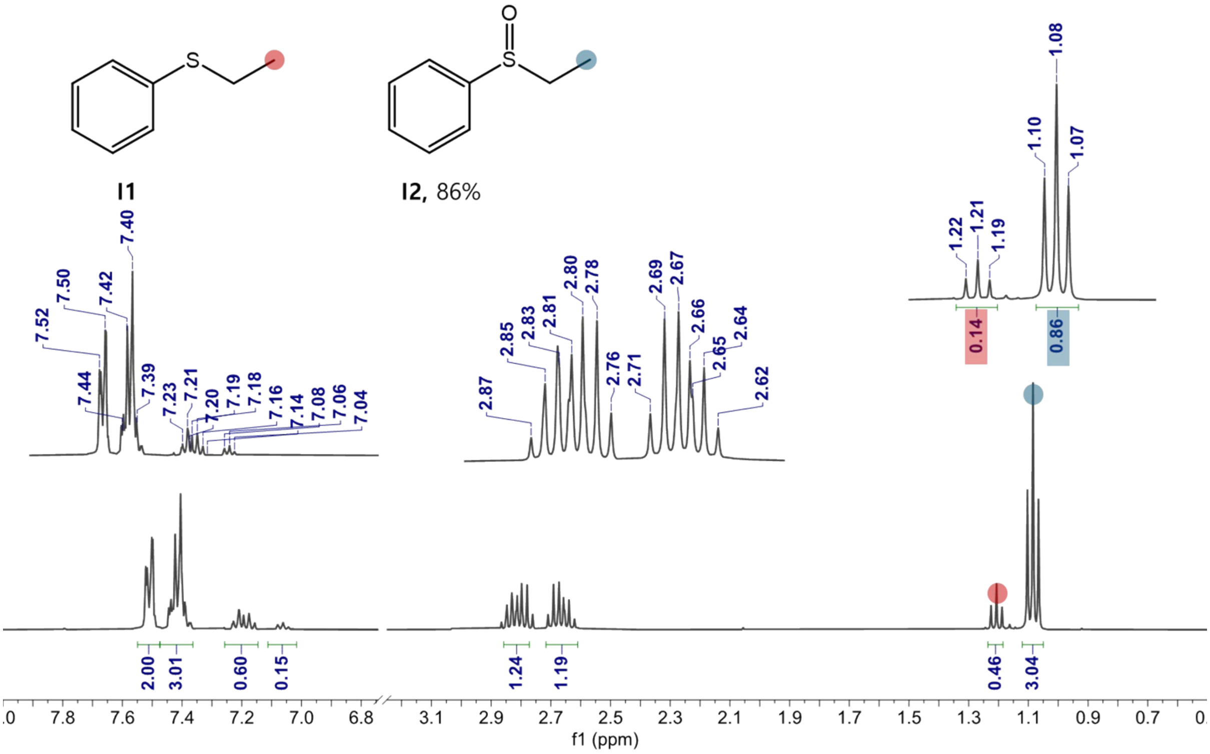


Fig S10 (i) ^{1}H NMR spectrum of I2 reaction

I2 ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) = 7.52-7.49 (m, 2H), 7.44-7.39 (m, 3H), 2.87-2.62 (m, 2H), 1.08 (t, 3H);

Steady-state and time-resolved optical spectroscopy

(1) *Absolute emission quantum yield measurements*

The absolute fluorescence quantum yield (Φ_F) of I-BOPHYPhCOOH in methanol was measured using an absolute PL quantum yield spectrometer equipped with an integrating sphere (C11347, Hamamatsu). To minimize self-absorption effect during the measurements, the concentration of I-BOPHYPhCOOH was adjusted to 1.0×10^{-5} M.

(2) *Time-resolved emission spectra measurements at 298 K*

The light source was a home-built cavity-dumped Ti:sapphire oscillator whose center wavelength was 800 nm, and the energy of the output pulses was 40 nJ at the repetition rate of 500 kHz. Pump pulses at 400 nm were generated by the second harmonic generation in a 100 μm thick BBO (β-barium borate) crystal. A commercial time-correlated single photon counting board (SPC-130-EMN, Becker & Hickl) was used to record time-resolved photoluminescence (TRPL) profiles directly with about 50 ps time resolution. A parabolic mirror was used to focus the excitation beam onto the sample, and the subsequent fluorescence was collected in a backscattering geometry using the same mirror. The emission was sent to a monochromator (SP-2155, Acton) and detected with an avalanche photodiode (id 100-50, id Quantique). Magic angle detection was used to avoid the effect of polarization. All the instruments were controlled in unison employing a home-built LabVIEW software, allowing us to record steady-state and TRPL spectra, directly. All samples were thoroughly degassed by purging with argon before the measurements.

(3) *Time-resolved emission spectra measurements at 77 K*

The light source was an optical parametric amplifier (Spectra-Physics, TOPAS prime) seeded by a regenerative amplifier system operating at 1 kHz (Spectra-Physics, Spitfire Ace, and MaiTai SP). The amplified output at 400 was delivered to a homemade sample holder placed inside a liquid-nitrogen reservoir using a bifurcated optical fiber. Photoluminescence was collected through the same fiber and directed to a polychromator (Princeton Instrument, SpectraPro SP-2300). The samples dissolved in 2-methyl tetrahydrofuran (m-THF) were frozen in liquid nitrogen to 77 K, and their TRPL spectra at 77 K were recorded using a streak scope system (Hamamatsu, C10627). A 450 nm long-pass filter was used to remove any stray light. Streak images were acquired in single-photon counting mode. The instrument response was about 450 ps in a 50 ns time window.

(4) *Nanosecond transient absorption measurements*

The long triple-state lifetimes of I-BOPHYPhCOOH solution (1.0×10^{-4} M) and I-BOPHY embedded in NU-1000 were determined by monitoring their ground state bleach (GSB) signals at 440 nm. Excitation source at 417 nm was made using the third harmonic of a Q-switched Nd:YAG laser (Continuum, Surelite II, pulse width of 4.5 ns) combined with a homemade Raman shifter. The temporal profiles at 440 nm were recorded using a photomultiplier (Zolix Instruments Co., CR 131) and a digital oscilloscope (Tektronix, TDS-784D) coupled to a monochromator (Dong-WooOptron, Monora 500i). The reported signals represent averages over 500 laser shots. The detection window was set to 50 μs, and the delay was fixed at 1 ms relative to the rigging edge of the laser.

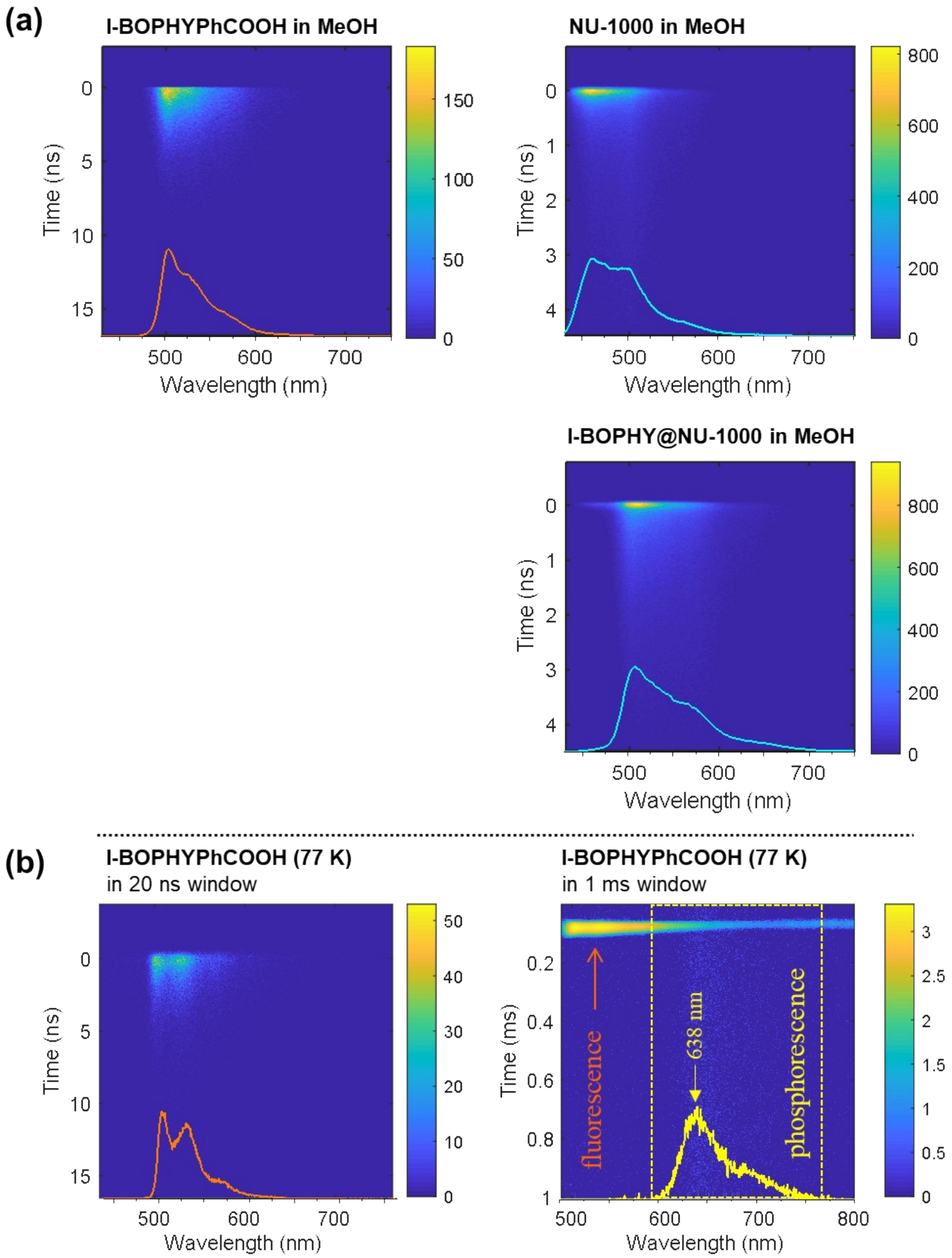


Fig S11. (a) Time-resolved photoluminescence (TRPL) spectra of I-BOPHYPhCOOH, NU-1000, and I-BOPHY@NU-1000 dispersed in methanol at 298 K. (b) TRPL spectra of I-BOPHYPhCOOH frozen in m-THF at 77 K. A long-lived triplet emission (τ_P>1 ms) is resolved in 1 ms time-window, which is limited by the 1 kHz repetition rate of the excitation source. The excitation wavelength was 400 nm. Time-integrated PL spectra are overlaid in each panel as solid lines. The concentration of the I-BOPHYPhCOOH solution was 1.0×10^{-5} M.

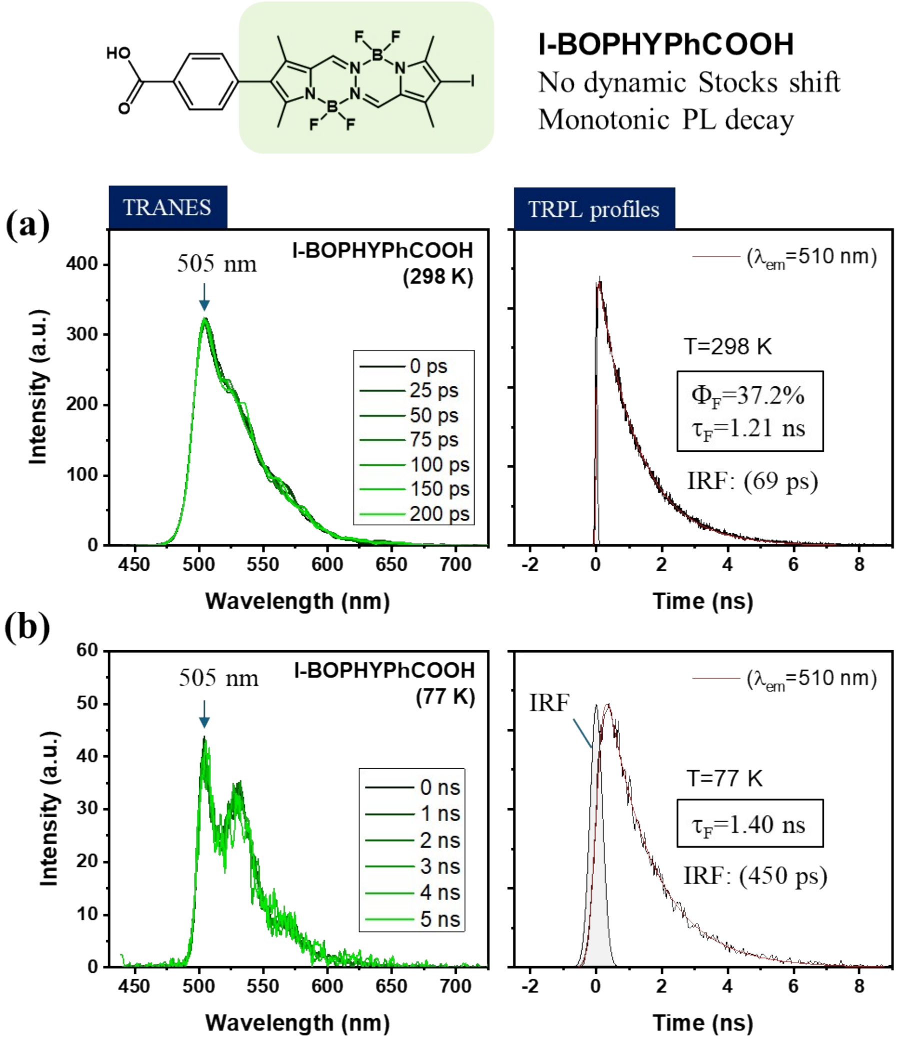


Fig S12. Time-resolved area-normalized emission spectra (TRANES) and single-color TRPL decay profiles of I-BOPHYPhCOOH at (a) 298 K and (b) 77 K. The iterative deconvolution fitting results using the instrument response function (IRF, FWHM: 69 ps at 298 K and 450 ps at 77 K) are overlaid on each decay profile.

Table S1. Photophysical properties of I-BOPHYPhCOOH

Solvent	T (K)	λ_A (nm)	λ_F (nm)	τ_F [a] (ns)	Φ_F [b] (%)	τ_R [c] (ns)	τ_{ISC} [d] (ns)	Φ_{ISC} [e] (%)	λ_P (nm)	τ_P (μs)
Methanol	298	475	505	1.21	37.2	3.25	2.45	52.0	–	1.98[f]
m-THF	77	–	505	1.40	–	–	–	–	638	>1 ms[g]

a) Fluorescence lifetime at 505 nm
b) Absolute fluorescence quantum yield
c) Radiative fluorescence lifetime, $\tau_R=\tau_F/\Phi_F$
d) Time constant for intersystem crossing (ISC): $\Phi_F=k_R/(k_R+k_{ISC}) \rightarrow \tau_{ISC}=(\Phi_F/(1-\Phi_F))\times\tau_R$
e) Reaction quantum yield for ISC process: $\Phi_{ISC}=k_{ISC}/(k_R+k_{ISC})$
f) Averaged triplet state lifetime by nanosecond transient absorption (Fig. S12).
g) Triplet state lifetime at 77 K by TRPL measurement.

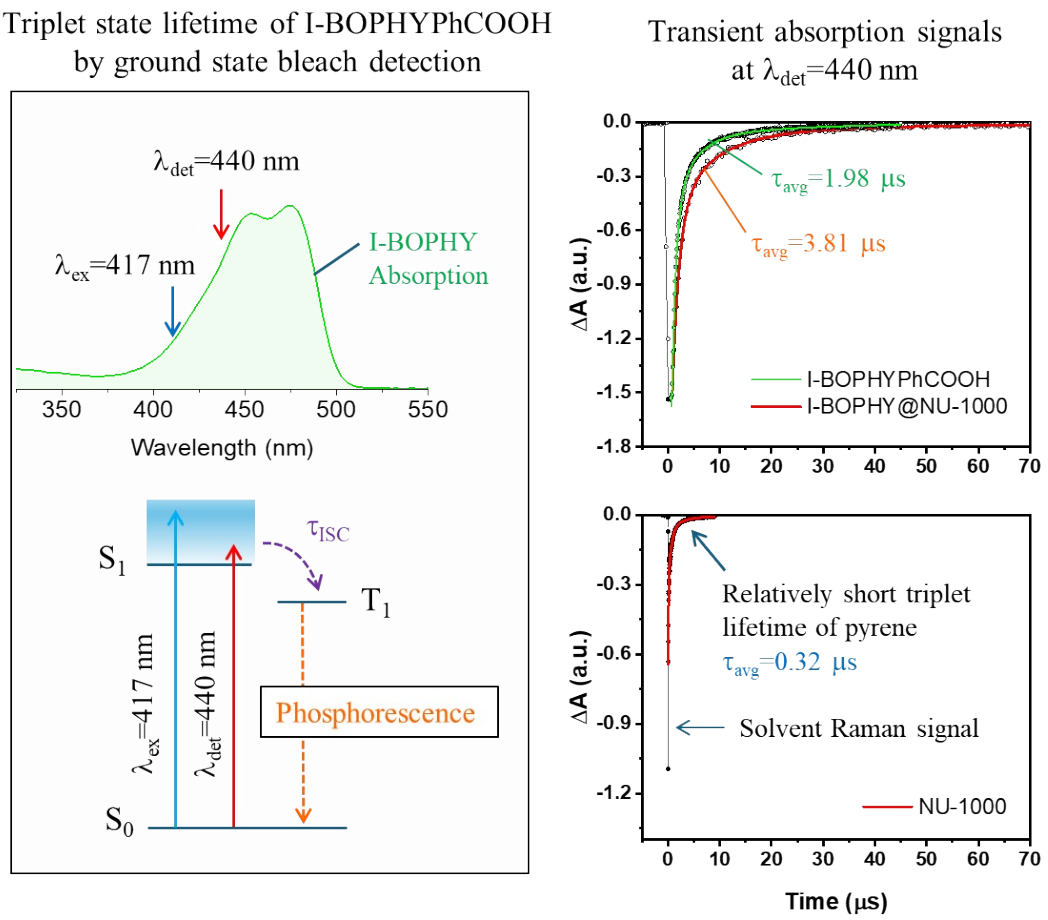


Fig S13. Nanosecond transient absorption (TA) measurements of I-BOPHYPhCOOH, NU-1000, and I-BOPHY@NU-1000 dispersed in methanol. The ground-state bleach (GSB) signal monitored at 440 nm was used to determine the triplet-state lifetimes. The concentration of the I-BOPHYPhCOOH solution was 1.0×10^{-4} M.

Table S2. Multi-exponential fitting results of nanosecond transient absorption signals monitored at 440 nm for I-BOPHYPhCOOH, NU-1000, and I-BOPHY@NU-1000 dispersed in methanol.

Samples	A_1 (%)	τ_1 (μs)	A_2 (%)	τ_2 (μs)	A_3 (%)	τ_3 (μs)	τ_{avg} (μs)[a)]
I-BOPHYPhCOOH	81.5	0.83	15.6	4.01	2.9	23.4	1.98
NU-1000	76.1	0.057	20.4	0.62	3.5	4.27	0.32
I-BOPHY@NU-1000	77.2	1.35	20.0	7.58	2.8	43.9	3.81

a) Averaged triplet-state lifetime, $\tau_{avg} = \sum_{1}^{3} A_i \times \tau_i$

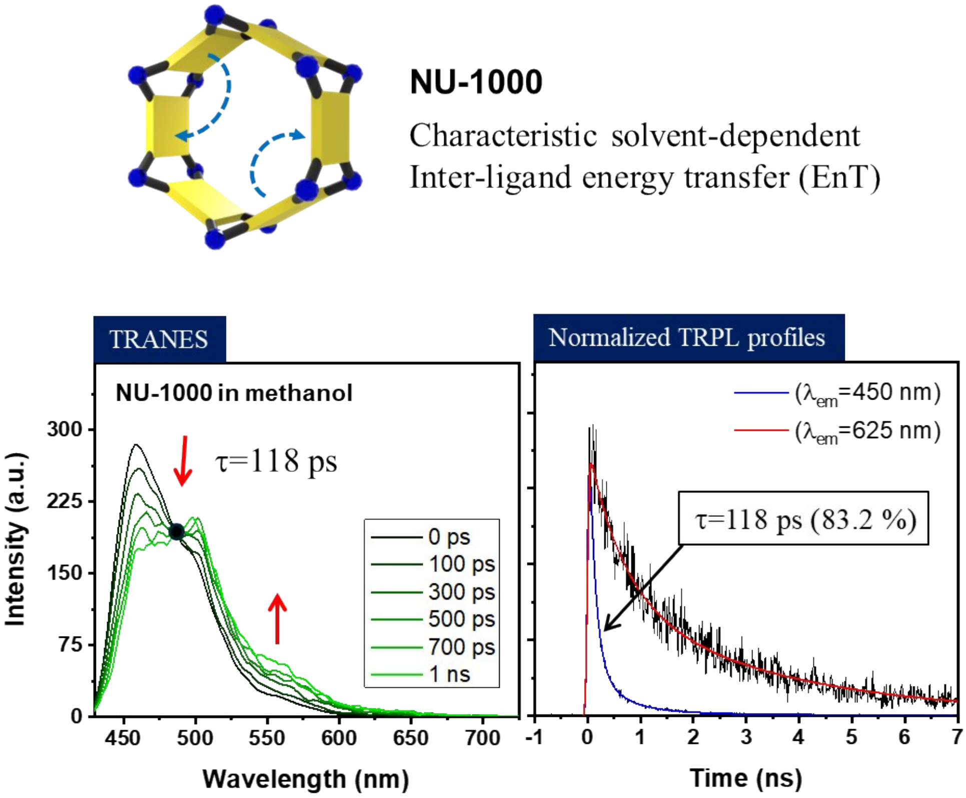


Fig S14. Time-resolved area normalized emission spectra (TRANES) of NU-1000 dispersed in methanol, and the corresponding TRPL decay profiles detected at 450 nm and 625 nm.

Table S3. Multi-exponential fitting results of picosecond TRPL profiles for NU-1000, and I-BOPHY@NU-1000 dispersed in methanol.

Samples	λ (nm)	A_1 (%)	τ_1 (ps)	A_2 (%)	τ_2 (ps)	A_3 (%)	τ_3 (ns)
NU-1000	450	83.2	118	15.3	498	1.5	2.96
	625			66.2	671	53.7	3.30
I-BOPHY@NU-1000	450	99.3	39.1	0.7	771		
	625	52.1	125	32.2	1034	15.7	4.39

Table S4. The calculated AQY for the sulfide oxidation of NU-1000 and I-BOPHY@NU-1000.

Samples	Conversion (%)	AQY (%)(a)
NU-1000	15	1.61
I-BOPHY@NU-1000	99	10.65

a) $AQY\ (\%) = \frac{n.N_{sulfoxide}}{N_{photons}} \times 100\%$, where n = 2 for sulfide oxidation to sulfoxide

Table S5. Comparison of the photocatalytic performance of I-BOPHY@NU-1000 with other catalysts reported in previous studies.

Entry	Catalyst	Condition	Time (h)	Conv. (%)	Ref.
1	I-BOPHY@NU-1000 (0.1 mol%)	Sulfides (1 mmol), MeOH (5 mL), 150W Xe–non lamp, air, R.T.	3	>99%	*This work*
2	PCBA@nMLM (0.1 mol %, 4.3mg)	Sulfides (1 mmol), MeOH (5 mL), 150W Xe–non lamp, air, R.T.	6	94%	[6]
3	C_3N_4/Zr-MOF (22wt%)	Sulfides (0.16 mmol), MeOH (1.25 mL), 300W Xenon lamp, O_2, R.T.	3.5	95%	[7]
4	I-BOPHY@UiO-67 (0.1 mol%)	Sulfides (1 mmol), MeOH (5 mL), 300W Xenon lamp, air, R.T.	3	>98%	[2]
5	In_2S_3/NU-1000-3 (20mg)	Sulfides (0.3 mmol), MeOD (2 mL), LED lamp irradiation, air, R.T.	3	94%	[8]
6	Zr_6-Irphen (4 mol%)	Sulfides (0.25 mmol), H_2O (4 mL), 100W Blue LED, O_2, R.T.	6	98%	[9]
7	Zr_{12}-NBC (2 mol%)	Sulfides (0.3 mmol), MeOH (4 mL), 24W Blue LED, air, R.T.	10	>99%	[10]
8	Zr-BTDB (2.5 mol%)	Sulfides (0.3 mmol), H_2O (1 mL), 3W Blue LED, air, R.T.	2	95%	[11]
9	Zr-MOF-OH (7 mg)	Sulfides (0.2 mmol), CF_3CH_2OH (3mL), White LED (5W), O_2, R.T.	8	>99%	[12]
10	PCBA@PCN–222 (0.2 mol%)	Sulfides (0.25 mmol), MeOH (5 mL), 150W Xe–non lamp, air, R.T.	5	>99%	[13]

Abbreviations

DMF: N,N-dimethylformamide

MC: dichloromethane

NIS: N-Iodosuccinimide

p-BQ: *p*-benzoquinone

DABCO: 1,4-diazabicyclo[2.2.2]octane

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