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Electronic Supplementary Information

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1. General information

All reactions were carried out in sealed air using oven dried glassware. Acetone, sodium chloride, graphite felt, platinum electrodes are all available from commercial sources. Deionized water is obtained by ultra pure water machine. The electrochemical instrument is HONGSHENGFENG DPS-305BM. Column chromatography was performed on silica gel (200-300 mesh). NMR spectra were recorded in CDCl₃ on 500 MHz spectrometers. ¹H NMR chemical shifts (δ) are reported in parts per million relative to tetramethylsilane (0 ppm). The following abbreviations are used for multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, and dt = doublet of triplets, m = multiplet. HRMS were obtained on an Ultima Global spetrometer with an ESI source. Melting points are uncorrected.

2. Synthesis of sulfides



The starting materials sulfides S11-S12¹, S14², S16³, S20⁴, S21⁵, S22⁶, S30⁷, S31⁸, S32⁹, S33¹⁰, S38¹¹, S39¹² were prepared according to literature procedure. Others were all obtained from commercial sources.

3. General procedure



Figure S1. Components required for the reaction



Figure S2. Typical reaction set up

To the mixed solution of acetone (4.5 mL) and water (0.5 mL), sulfide (0.2 mmol, 1 equivalent) and sodium chloride (0.8 mmol, 4 equivalent) were added. Equipped with graphite felt ($10 \times 10 \times 2$ mm) as anode and graphite felt ($10 \times 10 \times 2$ mm) as cathode, non-separating electrolytic cell device. The reaction mixture was stirred at constant current of 5 mA at 20 °C in air for 2-4 hours. After the reaction (monitored by TLC), the reaction system was dried with anhydrous MgSO₄, filtered and concentrated under reduced pressure. The residue was purified by silica gel column chromatography and eluted with petroleum ether and ethyl acetate.

4. Gram-scale reaction



Figure S3. Components required for gram-scale reaction



At the beginning of the reaction

At the end of the reaction

Figure S4. Typical reaction set up for gram-scale reaction



To the mixed solution of acetone (90 mL) and water (10 mL), thioanisole (10 mmol, 1 equivalent) and sodium chloride (20 mmol, 2 equivalent) were added. Equipped with graphite felt ($20 \times 10 \times 2$ mm) as anode and graphite felt ($20 \times 10 \times 2$ mm) as cathode, non-separation cell device. The reaction mixture was stirred in air at room temperature for 13 hours at a constant current of 50 mA. After the reaction (monitored by TLC), acetone was separated by rotary evaporation, and the residual liquid was extracted by

dichloromethane. Anhydrous $MgSO_4$ drying organic phase, filtered and concentrated under reduced pressure. The product was purified by silica gel column chromatography, eluted with petroleum ether and ethyl acetate, and the light yellow oil was obtained in 96% yield (1.35 g).

5. Decagram-scale reaction



Figure S5. Components required for the decagram-scale reaction



At the beginning of the reaction



At the end of the reaction

Figure S6. Typical reaction set up for the decagram-scale reaction



To the mixed solution of acetone (450 mL) and water (50 mL), thioanisole (100 mmol,

1 equivalent) and sodium chloride (100 mmol, 1 equivalent) were added. Equipped with graphite felt ($40 \times 15 \times 2$ mm) as anode and graphite felt ($40 \times 15 \times 2$ mm) as cathode, non-separation cell device. The reaction mixture was stirred in air at room temperature for 72 hours at a constant current of 100 mA. After the reaction (monitored by TLC), acetone was separated by rotary evaporation, and the residual liquid was extracted by dichloromethane. Anhydrous MgSO₄ drying organic phase, filtered and concentrated under reduced pressure. The product was purified by silica gel column chromatography, eluted with petroleum ether and ethyl acetate, and the light yellow oil was obtained in 85% yield (11.92 g).



Figure S7. Pure product

6. Detection of product by GC anlysis and NMR



Figure S8 GC of methyl phenyl sulfone

Methyl phenyl sulfone (0.5 mmol) standard was dissolved in acetone (1 mL), and the picture above was obtained by GC



Figure S9 GC of methyl phenyl sulfoxide

Methyl phenyl sulfoxide (0.5 mmol) standard was dissolved in acetone (1 mL), and the picture above was obtained by GC



Figure S10 GC of reaction system

After the reaction, the system was dried with anhydrous magnesium sulfate, the solid was separated by suction filtration, the solvent was separated by rotary evaporation, acetone (1 mL) was added, and the picture above was obtained by GC



Figure S11 In-situ ¹H NMR of the reaction system

After the reaction, the system was dried over anhydrous MgSO₄, the solid was separated by suction filtration, the solvent was separated by rotary evaporation, $CH_2Br_2(14 \ \mu L)$ and $CDCl_3$ were added, and the ¹H NMR was detected to obtain the above figure.

7. SEM image of graphite



Figure S12 SEM image of graphite

Place the anode graphite felt with different cycles in an oven at 100 °C, dry for 4 hours, and observe under SEM.

8. Cyclic voltammetry study



Figure S13. Cyclic voltammogram of S1

General procedure for cyclic voltammetry (CV): Cyclic voltammograms of S1 (0.2 mmol) were performed in a three-electrode cell at room temperature. The working electrode was a steady glassy carbon, the counter electrode was a platinum wire, and the reference was an Ag/AgCl electrode submerged in saturated aqueous KCl solution. Mixed solvent (acetone = 4.5 mL, H₂O = 0.5 mL) containing LiClO₄ (0.2 mmol) were poured into the electrochemical cell in cyclic voltammetry experiments. The scan rate was 100 mV/s, ranging from 0.0 V to 3.0 V.



Figure S14. Cyclic voltammogram of NaCl

General procedure for cyclic voltammetry (CV): Cyclic voltammograms of NaCl (0.8 mmol) were performed in a three-electrode cell at room temperature. The working electrode was a steady glassy carbon, the counter electrode was a platinum wire, and the reference was an Ag/AgCl electrode submerged in saturated aqueous KCl solution.

Mixed solvent (acetone = 4.5 mL, $H_2O = 0.5 \text{ mL}$) containing LiClO₄ (0.2 mmol) were poured into the electrochemical cell in cyclic voltammetry experiments. The scan rate was 100 mV/s, ranging from 0.0 V to 3.0 V.



Figure S15. Cyclic voltammogram of protocol

General procedure for cyclic voltammetry (CV): Cyclic voltammograms of S1 (0.2 mmol) and NaCl (0.8 mmol) were performed in a three-electrode cell at room temperature. The working electrode was a steady glassy carbon, the counter electrode was a platinum wire, and the reference was an Ag/AgCl electrode submerged in saturated aqueous KCl solution. Mixed solvent (acetone = 4.5 mL, H₂O = 0.5 mL) containing LiClO₄ (0.2 mmol) were poured into the electrochemical cell in cyclic voltammetry experiments. The scan rate was 100 mV/s, ranging from 0.0 V to 3.0 V.



Figure S16. Cyclic voltammogram of TEMPO

General procedure for cyclic voltammetry (CV): Cyclic voltammograms of TEMPO (0.8 mmol) were performed in a three-electrode cell at room temperature. The working

electrode was a steady glassy carbon, the counter electrode was a platinum wire, and the reference was an Ag/AgCl electrode submerged in saturated aqueous KCl solution. Mixed solvent (acetone = 4.5 mL, $H_2O = 0.5 \text{ mL}$) containing LiClO₄ (0.2 mmol) were poured into the electrochemical cell in cyclic voltammetry experiments. The scan rate was 100 mV/s, ranging from 0.0 V to 3.0 V.



Figure S17. Cyclic voltammogram of BHT

General procedure for cyclic voltammetry (CV): Cyclic voltammograms of BHT (0.8 mmol) were performed in a three-electrode cell at room temperature. The working electrode was a steady glassy carbon, the counter electrode was a platinum wire, and the reference was an Ag/AgCl electrode submerged in saturated aqueous KCl solution. Mixed solvent (acetone = 4.5 mL, H₂O = 0.5 mL) containing LiClO₄ (0.2 mmol) were poured into the electrochemical cell in cyclic voltammetry experiments. The scan rate was 100 mV/s, ranging from 0.0 V to 3.0 V.



9. Control experiments



a) To the mixed solution of MeCN (4.5 mL) and H_2O (0.5 mL), sulfide S1(0.2 mmol, 1 equivalent) and sodium chloride (0.8 mmol, 4 equivalent) were added. Equipped with graphite felt ($10 \times 10 \times 2$ mm) as anode and graphite felt ($10 \times 10 \times 2$ mm) as cathode, non-separating electrolytic cell device. The reaction mixture was stirred at constant current of 5 mA at 20 °C in N₂ for 2 hours. After the reaction (monitored by TLC), the reaction system was dried with anhydrous MgSO₄, filtered and concentrated under reduced pressure. The residue was purified by silica gel column chromatography and eluted with petroleum ether and ethyl acetate and the light yellow oil was obtained in

72% yield (0.0201 g).

b) To the mixed solution of acetone (4.5 mL) and H_2O^{18} (0.5 mL), sulfide S1(0.2 mmol, 1 equivalent) and sodium chloride (0.8 mmol, 4 equivalent) were added. Equipped with graphite felt (10 × 10 × 2 mm) as anode and graphite felt (10 × 10 × 2 mm) as cathode, non-separating electrolytic cell device. The reaction mixture was stirred at constant current of 5 mA at 20 °C in N₂ for 2 hours. After the reaction (monitored by TLC), the reaction system was dried with anhydrous MgSO₄, filtered and concentrated under reduced pressure. The residue was purified by silica gel column chromatography and eluted with petroleum ether and ethyl acetate and the light yellow oil was obtained in 96% yield (0.0273g). **HRMS** (ESI-TOF, [M + H⁺]): calcd for C₇H₈¹⁸OS, 143.0411, found 143.0414. C₇H₈¹⁶OS: C₇H₈¹⁸OS = 1: 10



Figure S19 HRMS (ESI-TOF, $[M + H^+]$): calcd for $C_7H_8^{18}OS$

c) To the mixed solution of acetone (4.5 mL) and H₂O (0.5 mL), sulfide **S1**(0.2 mmol, 1 equivalent), TEMPO (0.8 mmol, 4.0 equivalent) and sodium chloride (0.8 mmol, 4 equivalent) were added. Equipped with graphite felt ($10 \times 10 \times 2$ mm) as anode and graphite felt ($10 \times 10 \times 2$ mm) as cathode, non-separating electrolytic cell device. The reaction mixture was stirred at constant current of 5 mA at 20 °C in air for 2 hours.

Finally, it was found that the reaction did not occur.

d) To the mixed solution of acetone (4.5 mL) and H_2O (0.5 mL), sulfide S1(0.2 mmol, 1 equivalent), BHT (0.8 mmol, 4.0 equivalent) and sodium chloride (0.8 mmol, 4 equivalent) were added. Equipped with graphite felt ($10 \times 10 \times 2$ mm) as anode and graphite felt ($10 \times 10 \times 2$ mm) as cathode, non-separating electrolytic cell device. The reaction mixture was stirred at constant current of 5 mA at 20 °C in air for 2 hours. After the reaction (monitored by TLC), the reaction system was dried with anhydrous MgSO₄, filtered and concentrated under reduced pressure. The residue was purified by silica gel column chromatography and eluted with petroleum ether and ethyl acetate. The target product was not obtained, and **D** and **E** which were difficult to separate by silica gel column chromatography were obtained.



¹H NMR (500 MHz, CDCl₃)) δ 6.80 (s, 2H), 5.04 (s, 1H), 1.40 (s, 18H).
HRMS (ESI-TOF, [M + Na⁺]): calcd for C₁₅H₂₃Cl₂O, 311.0940, found 311.0946



¹**H NMR** (500 MHz, CDCl₃)) δ6.51 (s, 2H), 2.74 (s, 2H), 1.18 (s, 18H). **HRMS** (ESI-TOF, [M + H⁺]): calcd for C₁₅H₂₃ClO, 255.1510, found 255.1503.



S18



Figure S20 Gas chromatography by Trace1300E

Integral result						
Serial number		Retention	Peak	Peak	Relative	Relative peak
	Peak name	time (mi	area (pA*mi	height (pA	peak	height(%)
		n)	n))	area(%)	
1	Solvent	1.020	1664.574	55007.570	97.10	94.53
2	D	1.810	27.739	1306.723	1.27	2.25
3	Е	1.857	27.902	1878.692	1.63	3.23

BHT was used to participate in the system reaction to obtain two products **D** and **E** which were difficult to be separated by column chromatography. After gas chromatography, the ratio was 1.27% : 1.63% (44:56).

e) To the mixed solution of acetone (4.5 mL) and H₂O (0.5 mL), sulfide S1(0.2 mmol, 1 equivalent), vinyl cyclopropane derivative F (0.2 mmol, 1 equivalent) and sodium chloride (0.8 mmol, 4 equivalent) were added. Equipped with graphite felt ($10 \times 10 \times 2$ mm) as anode and graphite felt ($10 \times 10 \times 2$ mm) as cathode, non-separating electrolytic cell device. The reaction mixture was stirred at constant current of 5 mA at 20 °C in air for 2 hours. Detected G by HPLC-MS. HRMS (ESI-TOF, [M + Na⁺]): calcd for C₂₁H₂₀Cl₂O₄ 429.0631, found 429.0629.

f) To the mixed solution of acetone (4.5 mL) and H₂O (0.5 mL), sulfide **S1**(0.2 mmol, 1 equivalent), 1,1-Diphenylethylene **H** (0.2 mmol, 1 equivalent) and sodium chloride (0.8 mmol, 4 equivalent) were added. Equipped with graphite felt ($10 \times 10 \times 2$ mm) as anode and graphite felt ($10 \times 10 \times 2$ mm) as cathode, non-separating electrolytic cell device. The reaction mixture was stirred at constant current of 5 mA at 20 °C in air for

2 hours. Detected I by HPLC-MS. HRMS (ESI-TOF, $[M + H^+]$): calcd for C₁₄H₁₁Cl 215.0622, found 215.0630.

j) To the mixed solution of acetone (4.5 mL) and H_2O (0.5 mL), **S1** (0.2 mmol, 1 equivalent), NaClO (0.8 mmol, 4.0 equivalent). The reaction mixture was stirred at 20 °C in air for 2 hours. Finally, it was found that the reaction did not occur.

h) To the mixed solution of acetone (4.5 mL) and HCl (2 M, 0.5 mL), **S1** (0.2 mmol, 1 equivalent), NaClO (0.8 mmol, 4.0 equivalent). The reaction mixture was stirred at 20 °C in air for 2 hours. Finally, it was found that the reaction did not occur.

Table 1 Table the effect of Cl ion

	51 1	
Entry	Deviation from standard conditions ^a	Yield ^b (%)
1	KCl as the electrolyte	93
2	MgCl ₂ as the electrolyte	93
3	NH_4Cl as the electrolyte	86
4	LiCl as the electrolyte	87
5	$ZnCl_2$ as the electrolyte	80
6	^{<i>n</i>} Bu ₄ NCl as the electrolyte	88
7	Me_4NCl as the electrolyte	87
8	NaBr as the electrolyte	40
9	NaTs as the electrolyte	77
10	$NaNO_3$ as the electrolyte	45
11	NaNO ₃ as the electrolyte, catalytic amount of NaCl	93 ^c
12	$^{n}\mathrm{Bu}_{4}\mathrm{NPF}_{6}$ as the electrolyte	54
13	$^{n}\mathrm{Bu}_{4}\mathrm{NPF}_{6}$ as the electrolyte, catalytic amount of NaCl	88 ^c

 $\bigcup_{S1}^{S_{n}} \xrightarrow{CF}_{CF}^{I_{n}} \bigoplus_{S_{n}}^{O}$

^{*a*}Standard conditions: CF anode, CF cathode, undivided cell, constant current = 5 mA, **S1** (0.2 mmol), electrolyte (4.0 eq.), acetone = 4.5 mL, $H_2O = 0.5$ mL, 20 °C, air, 2 h (1.86 F mol⁻¹). ^{*b*}Isolated yields. ^{*c*}NaCl (0.2 eq.)

Table 2 Carbon-based electrode control experiments



Entry	Deviation from standard conditions ^a	$\mathbf{Yield}^{b}(\mathbf{\%})$
1	Carbon stick as anode	68
2	RVC^{c} as anode	58
3	GCE^d as anode	70

^{*a*}Standard conditions: CF cathode, undivided cell, constant current = 5 mA, **S1** (0.2 mmol), NaCl (4.0 eq.), acetone = 4.5 mL, H₂O = 0.5 mL, 20 °C, air, 2 h (1.86 F mol⁻¹). ^{*b*}Isolated yields. ^{*c*}Reticulated vitreous carbon. ^{*d*}Glassy carbon electrode.

Table 3 Solvent water	content control	experiments
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S1	1	
iation from standard conditions ^a		

Entry	Deviation from standard conditions ^a	Yield ^b (%)
1	Acetone: $H_2O = 0.0 \text{ mL}$: 5.0 mL	0
2	Acetone: $H_2O = 2.5 \text{ mL}: 2.5 \text{ mL}$	59
3	Acetone: $H_2O = 3.0 \text{ mL}$: 2.0 mL	63
4	Acetone: $H_2O = 4.0 \text{ mL}$: 1.0 mL	75
5	Acetone: $H_2O = 4.2 \text{ mL}: 0.8 \text{ mL}$	81
6	Acetone: $H_2O = 4.4 \text{ mL}: 0.6 \text{ mL}$	92
7	Acetone: $H_2O = 4.5 \text{ mL}: 0.5 \text{ mL}$	96
8	Acetone: $H_2O = 4.6 \text{ mL}: 0.4 \text{ mL}$	81
9	Acetone: $H_2O = 4.8 \text{ mL}: 0.2 \text{ mL}$	68

^{*a*}Standard conditions: CF anode, CF cathode, undivided cell, constant current = 5 mA, **S1** (0.2 mmol), NaCl (4.0 eq.), acetone = 4.5 mL, $H_2O = 0.5$ mL, 20 °C, air, 2 h (1.86 F mol⁻¹). ^{*b*}Isolated yields.



Figure S21 Solvent water content control experiments

10. NaClO probe experiments



Figure S22 Fluorescence analysis method to measure NaClO

Background: 1 mL of mixed solution (S1 = 0.2 mmol, NaCl = 0.8 mmol, acetone = 4.5 mL, $H_2O = 0.5$ mL) was added NaClO fluorescent indicator (1 μ L).

10⁻⁵mol/L ClO⁻: 1 mL of mixed solution (S1 = 0.2 mmol, NaCl = 0.8 mmol, acetone = 4.5 mL, $H_2O = 0.5$ mL) was added NaClO solution (1mmol/L, 10µL) and NaClO fluorescent indicator (1 µL).

Reaction system: The solution after the reaction under standard conditions.

Sulfoxide: 1 mL of mixed solution (1 = 0.2 mmol, NaCl = 0.8 mmol, acetone = 4.5 mL, $H_2O = 0.5$ mL) was added NaClO fluorescent indicator (1 μ L).

5X10⁻⁵mol/L ClO⁻: 1 mL of mixed solution (S1 = 0.2 mmol, NaCl = 0.8 mmol, acetone

= 4.5 mL, H_2O = 0.5 mL) was added NaClO solution (1mmol/L, 50µL) and NaClO fluorescent indicator (1 µL).



Figure S23 Experiment of pH test paper

11. Detection of H₂ by GC analysis



Figure S24 Collected gas from gram-scale experiment

12. Another possible mechanism.



Intermediate A was easily produced by single electron transfer from sulfide. Then H_2O reacted with intermediate A to form sulfoxide. Finally, hydrogen gas was released from the cathode.¹³

13. Characterization data of sulfoxide



26.9 mg, 96% yield. The spectra matched with the previous report.¹⁴

¹**H NMR** (500 MHz, CDCl₃) δ 7.64 (dt, J = 6.2, 1.6 Hz, 2H), 7.55 – 7.45 (m, 3H), 2.71

(s, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 145.67, 131.05, 129.36, 123.50, 43.93.



36.7 mg, 84% yield. The spectra matched with the previous report.¹⁴

¹**H NMR** (500 MHz, CDCl₃) δ 7.94 (dd, J = 7.8, 1.7 Hz, 1H), 7.66 – 7.51 (m, 2H), 7.37 (td, J = 7.6, 1.7 Hz, 1H), 2.81 (s, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 145.35, 132.91, 132.26, 128.73, 125.67, 118.40, 41.89.



35.4 mg, 81% yield. The spectra matched with the previous report.¹⁴

¹**H NMR** (500 MHz, CDCl₃) δ 7.65 (d, J = 8.2 Hz, 2H), 7.51 (d, J = 8.2 Hz, 2H), 2.70 (s, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 144.86, 132.58, 125.46, 125.15, 44.00.



30.7 mg, 88% yield. The spectra matched with the previous report.¹⁴

¹**H NMR** (500 MHz, CDCl₃) δ 7.58 (d, J = 8.3 Hz, 2H), 7.50 (d, J = 8.4 Hz, 2H), 2.70 (s, 3H).



32.6 mg, 96% yield. The spectra matched with the previous report.¹⁴

¹**H NMR** (500 MHz, CDCl₃) δ 7.56 (dd, J = 8.8, 2.4 Hz, 2H), 7.02 – 6.97 (m, 2H), 3.82

(d, J = 2.3 Hz, 3H), 2.67 (d, J = 2.3 Hz, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 161.97, 136.60, 125.47, 114.85, 55.54, 44.03.



28.8 mg, 78% yield. The spectra matched with the previous report.¹⁴

¹**H NMR** (500 MHz, CDCl₃) δ 8.38 (d, J = 8.6 Hz, 2H), 7.84 (d, J = 8.8 Hz, 2H), 2.79 (s, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 153.27, 149.51, 124.68, 124.48, 43.86.



34.6 mg, 94% yield. The spectra matched with the previous report.¹⁶

¹**H NMR** (500 MHz, DMSO) δ 8.09 (d, J = 8.5 Hz, 2H), 7.79 (d, J = 8.3 Hz, 2H), 2.77 (s, 3H).

¹³C NMR (125 MHz, DMSO) δ 171.81, 156.49, 137.98, 135.20, 129.06, 129.01, 48.23.



28.0 mg, 77% yield. The spectra matched with the previous report.¹⁵

¹**H NMR** (500 MHz, CDCl₃) δ 8.09 (d, J = 8.2 Hz, 2H), 7.73 (d, J = 8.2 Hz, 2H), 2.75 (s, 3H), 2.64 (s, 3H).



30.9 mg, 83% yield. The spectra matched with the previous report.¹⁷

¹**H NMR** (500 MHz, CDCl₃) δ 7.54 (d, J = 8.3 Hz, 2H), 7.34 (d, J = 8.3 Hz, 2H), 2.70

(s, 3H), 2.51 (s, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 143.17, 141.65, 126.38, 124.01, 44.00, 15.21.



33.9 mg, 84% yield.

¹H NMR (500 MHz, CDCl₃) δ 7.80 (s,4H), 2.75 (s, 6H).

¹³C NMR (125 MHz, CDCl3) δ 149.27, 124.55, 44.02.

HRMS (ESI-TOF, $[M + H^+]$): calcd for $C_8H_{10}O_2S_2$, 203.0195, found 308.0719



36.7 mg, 72% yield. The spectra matched with the previous report.¹⁸

¹**H NMR** (500 MHz, CDCl₃) δ 7.55 (d, J = 20.2 Hz, 4H), 2.68 (s, 3H), 1.49 (s, 9H).

¹³C NMR (125 MHz, CDCl₃) δ 152.51, 141.49, 138.67, 124.71, 118.88, 81.12, 43.97, 28.28.



43.1 mg, 80% yield.

¹**H NMR** (500 MHz, CDCl₃) δ 7.58 (d, J = 8.3 Hz, 2H), 7.41 (d, J = 8.2 Hz, 2H), 3.27 (s, 3H), 2.69 (s, 3H), 1.45 (s, 9H).

¹³**C NMR** (125 MHz, CDCl₃) δ 154.27, 146.38, 141.55, 125.77, 123.94, 81.08, 43.94, 37.04, 28.27.

HRMS (ESI-TOF, [M + Na⁺]): calcd for C₁₃H₁₉NO₃S, 292.0983, found 292.0987



28.2 mg, 81% yield. The spectra matched with the previous report.¹⁹

¹H NMR (500 MHz, CDCl₃) δ 7.65 (s, 1H), 7.53 – 7.42 (m, 3H), 2.73 (s, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 147.80, 135.71, 131.21, 130.61, 123.64, 121.61, 44.02.



32.7 mg, 86% yield. The spectra matched with the previous report.¹⁴

¹**H NMR** (500 MHz, CDCl₃) δ 8.15 (d, J = 1.8 Hz, 1H), 7.92 (d, J = 8.5 Hz, 1H), 7.87 (dt, J = 8.5, 2.8 Hz, 1H), 7.86 – 7.81 (m, 1H), 7.53 (dt, J = 9.5, 3.2 Hz, 3H), 2.72 (s, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 142.77, 134.44, 132.93, 129.61, 128.52, 128.07, 127.79, 127.36, 124.05, 119.45, 43.80.



23.7 mg, 84% yield. The spectra matched with the previous report.¹⁵ ¹H NMR (500 MHz, CDCl₃) δ 8.54 (d, J = 4.3 Hz, 1H), 7.94 (d, J = 7.8 Hz, 1H), 7.87 (td, J = 7.6, 1.7 Hz, 1H), 7.30 (ddd, J = 7.7, 4.7, 1.2 Hz, 1H), 2.77 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 166.00, 149.55, 138.11, 124.58, 119.25, 41.29.



33.0 mg, 67% yield. The spectra matched with the previous report.¹⁸
¹H NMR (500 MHz, CDCl₃) δ 7.59 (d, J = 8.5 Hz, 2H), 7.46 – 7.31 (m, 5H), 7.10 (d, J = 8.6 Hz, 2H), 5.11 (s, 2H), 2.70 (s, 3H).
¹³C NMR (125 MHz, CDCl₃) δ 161.12, 136.97, 136.20, 128.71, 128.26, 127.47, 125.49, 115.76, 70.28, 44.01.



20.0 mg, 60% yield. The spectra matched with the previous report.²⁰ ¹H NMR (500 MHz, CDCl₃) δ 7.67 – 7.62 (m, 2H), 7.57 – 7.47 (m, 3H), 4.17 (ddd, J = 12.2, 8.9, 3.3 Hz, 1H), 3.99 (dt, J = 12.2, 4.7 Hz, 1H), 3.78 (s, 1H), 3.15 (ddd, J = 13.2, 8.9, 4.2 Hz, 1H), 2.90 (ddd, J = 13.5, 5.3, 3.2 Hz, 1H). ¹³C NMR (125 MHz, CDCl₃) δ 143.07, 131.22, 129.43, 123.97, 58.67, 56.84.



27.6 mg, 83% yield. The spectra matched with the previous report.¹⁴

¹**H NMR** (500 MHz, CDCl₃)) δ 7.62 – 7.55 (m, 2H), 7.54 – 7.45 (m, 3H), 5.63 (ddt, J = 17.5, 10.3, 7.5 Hz, 1H), 5.32 (d, J = 10.1 Hz, 1H), 5.18 (dd, J = 16.9, 1.4 Hz, 1H), 3.59 – 3.46 (m, 2H).

¹³C NMR (125 MHz, CDCl₃) δ 142.91, 131.11, 129.04, 125.24, 124.34, 123.86, 60.85.



50.2 mg, 88% yield.

¹**H NMR** (500 MHz, CDCl₃) δ 7.95 (d, J = 7.3 Hz, 1H), 7.77 – 7.71 (m, 1H), 7.72 – 7.61 (m, 2H), 7.30 (t, J = 7.5 Hz, 2H), 7.24 – 7.17 (m, 3H), 4.70 – 4.56 (m, 2H), 2.82 (t, J = 7.6 Hz, 2H), 2.29 – 2.17 (m, 2H).

¹³C NMR (125 MHz, CDCl₃) δ 170.89, 154.72, 140.73, 132.41, 132.15, 129.90, 128.57, 128.42, 126.20, 124.88, 124.04, 70.14, 32.10, 30.01.

HRMS (ESI-TOF, [M + Na⁺]): calcd for C₁₆H₁₅NO₂S, 308.0721, found 308.0719



23.8 mg, 60% yield. The spectra matched with the previous report.²⁰

¹**H NMR** (500 MHz, CDCl₃) δ 7.68 (dd, J = 6.5, 2.8 Hz, 2H), 7.57 – 7.51 (m, 3H), 3.84 (d, J = 13.7 Hz, 1H), 3.70 (s, 3H), 3.67 (d, J = 13.7 Hz, 1H).

¹³C NMR (125 MHz, CDCl₃) δ 165.20, 143.03, 131.84, 129.45, 124.10, 61.57, 52.78.



38.7 mg, 93% yield. The spectra matched with the previous report.²¹

¹**H NMR** (500 MHz, CDCl₃) δ 7.57 (dd, J = 7.6, 2.0 Hz, 2H), 7.54 – 7.40 (m, 3H), 2.54 (tt, J = 11.7, 3.4 Hz, 1H), 1.82 (tt, J = 12.8, 3.3 Hz, 4H), 1.63 (dd, J = 11.4, 4.0 Hz, 1H), 1.40 (dtd, J = 27.8, 12.1, 8.6 Hz, 2H), 1.30 – 1.09 (m, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 141.92, 130.90, 128.88, 125.00, 63.16, 26.28, 25.60, 25.45, 25.31, 24.00.



21.6 mg, 54% yield. The spectra matched with the previous report.²² ¹H NMR (500 MHz, CDCl₃) δ 7.60 (d, J = 8.4 Hz, 2H), 7.49 (d, J = 8.4 Hz, 2H), 2.23 (tt, J = 8.0, 4.8 Hz, 1H), 1.27 – 1.18 (m, 1H), 1.07 – 0.89 (m, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 143.48, 137.13, 129.46, 125.43, 33.87, 3.48, 2.75.



35.7 mg, 72% yield. The spectra matched with the previous report.²³ ¹**H NMR** (500 MHz, CDCl₃) δ 7.70 (dd, J = 6.8, 3.0 Hz, 2H), 7.49 (dd, J = 5.0, 1.9 Hz, 3H), 7.47 – 7.41 (m, 2H), 7.33 – 7.24 (m, 3H), 4.20 (d, J = 13.5 Hz, 1H), 4.08 (d, J = 13.5 Hz, 1H).

¹³C NMR (125 MHz, CDCl₃) δ 133.59, 131.68, 131.01, 129.31, 129.15, 127.78, 124.91, 60.97.



29.6 mg, 85% yield. The spectra matched with the previous report.¹⁵

¹**H NMR** (500 MHz, CDCl₃) δ 7.73 – 7.67 (m, 2H), 7.59 – 7.53 (m, 3H), 4.38 (dd, J = 5.1, 1.2 Hz, 2H).

¹³C NMR (125 MHz, CDCl₃) δ 140.97, 132.18, 129.36, 124.83, 61.29.



34.6 mg, 80% yield. The spectra matched with the previous report.¹⁴ ¹H NMR (500 MHz, CDCl₃) δ 7.52 – 7.34 (m, 5H), 7.32 – 7.21 (m, 3H), 7.01 – 6.95

(m, 2H), 4.09 (d, J = 12.6 Hz, 1H), 4.00 (d, J = 12.6 Hz, 1H).

¹³C NMR (125 MHz, CDCl₃) δ 142.79, 131.16, 130.36, 129.15, 128.85, 128.45, 128.25, 124.44, 63.59.



36.8 mg, 80% yield. The spectra matched with the previous report.²⁰ ¹**H NMR** (500 MHz, CDCl₃) δ 7.42 – 7.33 (m, 6H), 7.32 – 7.26 (m, 4H), 3.90 (q, J = 13.0 Hz, 4H).

¹³C NMR (125 MHz, CDCl₃) δ 130.16, 130.13, 128.96, 128.36, 57.32.



16.0 mg, 77% yield. The spectra matched with the previous report.²⁴ ¹H NMR (500 MHz, CDCl₃) δ 3.05 – 2.73 (m, 4H), 2.46 (tt, J = 9.3, 5.8 Hz, 2H), 2.04 (q, J = 8.0 Hz, 2H).

¹³C NMR (125 MHz, CDCl₃) δ 54.53, 25.53.



39.6 mg, 98% yield. The spectra matched with the previous report.¹⁹ ¹H NMR (500 MHz, CDCl₃) δ 7.67 – 7.62 (m, 4H), 7.47 – 7.40 (m, 6H). ¹³C NMR (125 MHz, CDCl₃) δ 145.60, 131.07, 129.34, 124.78.



54.7 mg, 76% yield. The spectra matched with the previous report.²⁵ ¹H NMR (500 MHz, CDCl₃) δ 7.59 (dd, J = 8.5, 1.8 Hz, 4H), 7.51 – 7.46 (m, 4H). ¹³C NMR (125 MHz, CDCl₃) δ 144.36, 132.71, 126.17, 125.94.



52.8 mg, 78% yield. The spectra matched with the previous report.²⁶ ¹H NMR (500 MHz, CDCl₃) δ 7.80 (d, J = 8.2 Hz, 4H), 7.74 (d, J = 8.2 Hz, 4H). ¹³C NMR (125 MHz, CDCl₃) δ 149.18, 133.48 (d, J_{C-F} = 33.0 Hz), 126.64 (d, J_{C-F} = 3.7 Hz), 124.90, 123.31 (q, J_{C-F} = 272.7 Hz).



39.3 mg, 78% yield. The spectra matched with the previous report.²⁷ ¹H NMR (500 MHz, CDCl₃) δ 8.31 (s, 1H), 7.97 – 7.92 (m, 1H), 7.89 – 7.81 (m, 2H), 7.72 – 7.66 (m, 2H), 7.60 – 7.53 (m, 2H), 7.50 (dd, J = 8.6, 1.8 Hz, 1H), 7.48 – 7.39 (m, 3H).

¹³**C NMR** (125 MHz, CDCl₃) δ 145.42, 142.55, 134.39, 132.81, 131.12, 129.71, 129.36, 128.65, 128.03, 127.91, 127.29, 125.34, 125.01, 120.69.



32

46.1 mg, 89% yield.

¹**H NMR** (500 MHz, CDCl₃) δ 8.03 (dd, J = 8.3, 2.9 Hz, 1H), 7.90 (dd, J = 10.1, 7.1 Hz, 3H), 7.51 (tq, J = 8.6, 4.6, 3.4 Hz, 4H), 7.43 (td, J = 7.6, 2.9 Hz, 1H).

¹³C NMR (125 MHz, CDCl₃) δ 178.26, 153.72, 143.04, 135.85, 132.01, 129.60, 126.83, 126.36, 124.64, 124.19, 122.26.

HRMS (ESI-TOF, [M + Na⁺]): calcd for C₁₃H₉NOS₂, 282.0023, found 282.0021



40.8 mg, 91% yield.

¹**H NMR** (500 MHz, CDCl₃) δ 7.79 (td, J = 5.9, 5.1, 3.3 Hz, 2H), 7.56 – 7.49 (m, 3H), 2.76 (s, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 179.32, 169.13, 142.85, 132.17, 129.75, 124.17, 16.11. HRMS (ESI-TOF, [M + H⁺]): calcd for C₉H₈N₂OS₂, 225.0156, found 225.0157



34.0 mg, 85% yield. The spectra matched with the previous report.²⁸

¹**H NMR** (500 MHz, CDCl₃) δ 7.98 (d, J = 7.6 Hz, 2H), 7.80 (d, J = 7.7 Hz, 2H), 7.59 (t, J = 7.5 Hz, 2H), 7.49 (t, J = 7.5 Hz, 2H).

¹³C NMR (125 MHz, CDCl₃) δ 145.13, 137.08, 132.56, 129.54, 127.51, 121.93.



50.8 mg, 91% yield. The spectra matched with the previous report.²⁹

¹**H NMR** (500 MHz, CDCl₃) δ 7.98 (d, J = 7.6 Hz, 1H), 7.92 (d, J = 1.8 Hz, 1H), 7.82 (d, J = 8.2 Hz, 1H), 7.75 (d, J = 7.6 Hz, 1H), 7.60 (td, J = 7.3, 6.7, 1.4 Hz, 3H), 7.56 – 7.49 (m, 1H).

¹³C NMR (125 MHz, CDCl₃) δ 145.66, 143.90, 139.00, 135.83, 132.71, 132.38, 130.24, 128.79, 127.65, 127.41, 125.23, 122.16.





37.6 mg, 87% yield. The spectra matched with the previous report.³⁰

¹**H NMR** (500 MHz, CDCl₃) δ 7.93 (dd, J = 7.8, 1.7 Hz, 2H), 7.62 (td, J = 7.9, 7.2, 1.7

Hz, 2H), 7.46 – 7.33 (m, 4H).

¹³C NMR (125 MHz, CDCl₃) δ 149.52, 133.77, 131.06, 124.86, 123.77, 118.82.



24.6 mg, 54% yield. The spectra matched with the previous report.³¹

¹**H NMR** (500 MHz, CDCl₃) δ 8.37 (d, J = 7.7 Hz, 2H), 8.17 (d, J = 7.9 Hz, 2H), 7.86 (t, J = 7.7 Hz, 2H), 7.73 (t, J = 7.6 Hz, 2H).

¹³C NMR (125 MHz, CDCl₃) δ 179.83, 144.93, 133.77, 131.42, 129.40, 128.61, 127.04.



40.1 mg, 78% yield. The spectra matched with the previous report.³²

¹**H NMR** (500 MHz, CDCl₃) δ 7.87 (dd, J = 7.6, 1.6 Hz, 1H), 7.66 (dd, J = 7.7, 1.4 Hz, 1H), 7.50 (dtd, J = 21.5, 7.5, 1.5 Hz, 1H), 2.33 (s, 1H).

¹³C NMR (125 MHz, CDCl₃) δ 168.25, 140.19, 133.71, 130.48, 127.57, 126.51, 124.49, 23.39.



45.6 mg, 84% yield. The spectra matched with the previous report.³³

¹**H NMR** (500 MHz, CDCl₃) δ 7.94 (dt, J = 7.6, 1.5 Hz, 2H), 7.62 (td, J = 7.9, 7.2, 1.6 Hz, 2H), 7.42 (d, J = 8.5 Hz, 2H), 7.24 (t, J = 7.4 Hz, 2H), 4.22 (t, 1H), 1.95 (h, J = 6.9, 6.1 Hz, 2H), 1.57 (h, J = 7.4 Hz, 2H), 1.07 (t, J = 7.4 Hz, 3H).

¹³C NMR (125 MHz, CDCl₃) δ 138.36, 132.86, 131.73, 124.08, 121.68, 115.71, 47.90, 28.28, 20.09, 13.82.



38.5 mg, 83% yield.

¹**H NMR** (500 MHz, CDCl₃) δ 7.92 (dd, *J* = 7.8, 1.5 Hz, 2H), 7.62 (dd, *J* = 7.7, 1.3 Hz, 2H), 7.54 (ddd, *J* = 9.1, 6.9, 1.3 Hz, 2H), 7.46 – 7.36 (m, 2H).

¹³C NMR (125 MHz, CDCl₃) δ 141.40, 129.84, 129.01, 128.42, 124.47.

HRMS (ESI-TOF, $[M + H^+]$): calcd for $C_{12}H_8OS_2$, 233.0089, found 233.0096



42.1 mg, 85% yield.

¹**H NMR** (500 MHz, CDCl₃) δ 8.07 – 8.02 (m, 4H), 7.70 (t, J = 4.5 Hz, 4H).

¹³C NMR (125 MHz, CDCl₃) δ 138.35, 130.79, 123.70.

HRMS (ESI-TOF, $[M + H^+]$): calcd for $C_{12}H_8O_2S_2$ 249.0038, found 249.0048



40.1 mg, 58% yield. The spectra matched with the previous report.¹⁶

¹H NMR (500 MHz, CDCl₃) δ 8.25 (s, 1H), 7.40 (d, J = 8.6 Hz, 1H), 7.03 (d, J = 2.6 Hz, 1H), 6.81 (dt, J = 8.8, 2.5 Hz, 1H), 4.36 (d, J = 2.4 Hz, 2H), 3.83 (d, J = 2.3 Hz, 3H), 3.77 (d, J = 2.4 Hz, 3H), 2.30 (d, J = 2.4 Hz, 3H), 2.26 (d, J = 2.3 Hz, 3H).
¹³C NMR (125 MHz, CDCl₃) δ 165.08, 155.94, 155.89, 150.62, 148.44, 126.38, 125.49, 111.02, 60.09, 55.84, 35.10, 29.28, 13.41, 11.29.


34.8 mg, 62% yield. The spectra matched with the previous report.¹⁶ ¹**H NMR** (400 MHz, DMSO) δ 11.92 (s, 2H), 7.73 (d, J = 1.6 Hz, 1H), 7.58 (d, J = 8.3 Hz, 1H), 7.34 (dd, J = 8.3, 1.7 Hz, 1H), 3.80 (s, 3H), 2.94 – 2.68 (m, 2H), 1.68 – 1.37 (m, 2H), 0.96 (t, J = 7.4 Hz, 3H).

¹³C NMR (100MHz, DMSO) δ 154.87, 149.09, 136.61, 117.25, 114.87, 110.23, 58.67, 53.09, 15.89, 13.42.

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15. NMR Spectra



100 f1 (ppm)









210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 f1 (ppm)















































































S52



















- 2.70









√ 58.67 √ 56.84



7.00 <t

















210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 f1 (ppm)









 $\begin{array}{c} 7.61\\ 7.559\\ 7.5$







7.7 7.7 7.7 7.7 7.7 7.5 7.7 7.5 7.7 7.5 7.7 7.5 7.7 7.5 7.67 7.5 7.7 7.5





















7.146 7.146 7.146 7.145 <tr/tr> <tr/tr>







S65















-57.32











7.65 7.65 7.63 7.63 7.63 7.47 7.45 7.45 7.45 7.45 7.45 7.42 7.42 7.41









7.60 7.59 7.58 7.58 7.58 7.49 7.48






























33





210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 f1 (ppm)

7.99 7.797 7.797 7.79 7.79 7.50 7.57 7.57 7.49















7.94 7.92 7.92 7.92 7.92 7.92 7.92 7.92 7.92 7.92 7.92 7.92 7.92 7.92 7.92 7.92 7.93 7.16 7.16 7.16 7.16 7.16 7.16 7.16 7.16 7.16 7.16 7.16 7.16 7.16 7.16 7.16 7.16 7.16 7.17 7.16 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 <t









210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 f1 (ppm)













- 2.33



S78









110 100 f1 (ppm)

7.93 7.92 7.92 7.92 7.63 7.61 7.61 7.61 7.61 7.61 7.61 7.61 7.54 7.54 7.53 7.42 7.42 7.42 7.42









8.05 8.05 8.04 7.71 7.70 7.69



















210 200 190 180 170 180 150 140 150 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 f1 (ppm)

