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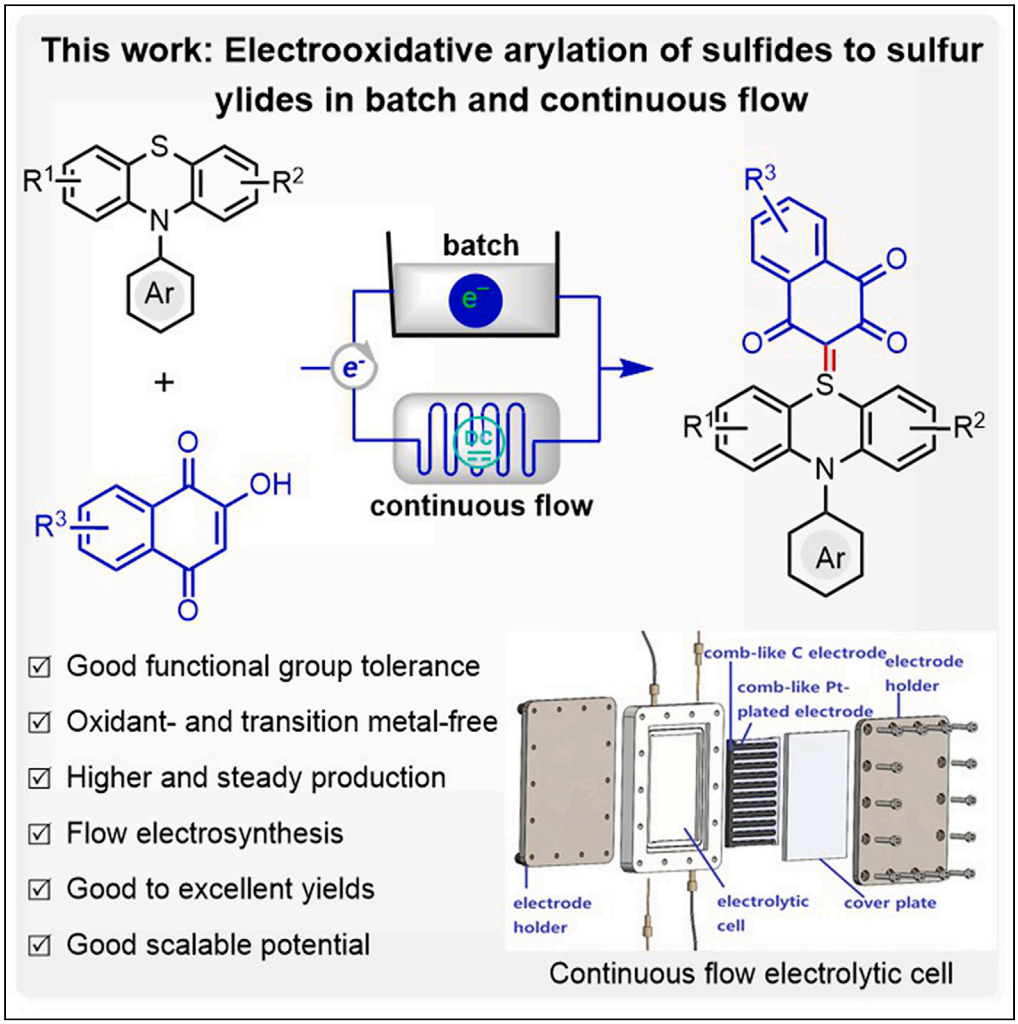

Xiangxing Huang, Yifei Yao, Xing Yin, ..., Hong Qin, Chengkou Liu, Kai Guo

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Highlights

The preparation of sulfur ylides through electro-oxidative quinylation of sulfides

Exogenous oxidant and transition metal-free electrolysis conditions

Excellent functional group compatibility and good concentration tolerance

Good scalable potential, higher and steady production using micro-flow cells

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Electro-oxidative quinylation of sulfides to sulfur ylides in batch and continuous flow

Xiangxing Huang,[1] Yifei Yao,[1] Xing Yin,[2] Wenjing Guan,[1] Chengcheng Yuan,[1] Zheng Fang,[1] Hong Qin,[1] Chengkou Liu,[1,4,*] and Kai Guo[1,3]

SUMMARY

An unprecedented strategy for preparing a series of sulfur ylides through electro-oxidative quinylation of sulfides in batch and continuous flow has been developed. Good to excellent yields were obtained with excellent functional group compatibility and good concentration tolerance under exogenous oxidant- and transition metal-free conditions. Advantageously, this electrosynthesis methodology was scalable with higher daily production and steady production was achieved attributing to the use of micro-flow cells.

INTRODUCTION

Sulfur ylides have occupied a prominent role in synthetic chemistry because of their abundant and unique reactivity.[1–3] Indeed, a variety of name reactions were recorded which reported highly useful and appealing transformations based on sulfur ylides, including but not limited to Swern oxidation,[4–6] Kornblum oxidation,[7–9] Pummerer rearrangement,[10] Johnson-Corey-Chaykovsky epoxidation, aziridination and cyclopropanation,[11,12] Stevens rearrangement,[13] and Gassman indole synthesis.[14] Moreover, metal carbenes had been widely produced from sulfur ylides and were transformed into a series of cross coupling[15–17] and heterocycles products.[18–23] In addition, the sulfur (IV)-containing molecules are prevalent in natural products, potential medicines, functional materials and biosynthetic machinery.[24–29] In general, deprotonation of sulfonium salts in the presence of base gave the corresponding sulfur ylides.[30,31] Most recently, Martin's sulfurane (bis[α,α-bis(trifluoromethyl)benzyloxy] diphenylsulfur) was proved to be the efficient reagent to give the sulfur ylides.[32,33] Moreover, sulfur ylides could also be prepared from metal carbenes which could be generated from diazo compounds[34–37] or iodonium ylides[38] in the presence of metal catalyst. On this basis, Koenigs and co-workers described a transition metal-free photochemical generation of carbenes from diazo compounds, which were transformed into the corresponding sulfur ylides smoothly from the coupling between highly active carbene species and sulfides (Scheme 1A).[39,40] Then, a series of photochemical Doyle-Kirmse reactions to organosulfur compounds via the [2,3]-sigmatropic rearrangement of sulfur ylide intermediates were achieved[41–45] (Scheme 1A). However, the sulfur ylide preparations that had been reported so far required tedious preparation procedures and/or the use of metal salts and costly ylide transfer reagents which compromised the atom-economy and step-economy and gave stoichiometric amounts of undesired by-products. Therefore, it is desirable to develop a more straightforward, economical and sustainable strategy to produce sulfur ylides.

Organic electrochemistry has been recognized as an inherently green and versatile synthetic technology because it employs traceless "electron" to promote redox reactions and obviates the use of oxidizing or reducing "reagents."[46–52] Moreover, it is controllable because of tunable electrode potentials and can be switched off anytime.[53,54] With our continued interest in electro-redox processes, many electro-initiation of carbon,[55,56] nitrogen,[57–59] or sulfur-centered radical or radical-cation intermediates[60,61] processes were developed by us recently. Inspired by the electrochemical generating of sulfur-centered radical or radical-cation intermediates, we speculated that the corresponding sulfur (IV) motif including sulfur ylide and sulfonium salt might be produced via the anode oxidative coupling of anion or radical with *in-situ* generated S-centered radical-cation intermediate (Scheme 1C-1). In this context, a straightforward electro-oxidative coupling of sulfides with activated methylene compounds to the corresponding sulfur ylides in continuous flow via anion cation cross coupling was developed in 2021 by us (Scheme 1C-2).[61] Attributing to the use of continuous flow reactor, good scalable potential was observed. At around the same time, Wickens and co-workers reported electrochemically transformed un-activated alkenes into metastable, dicationic intermediates, which could further undergo aziridination with primary amines (Scheme 1B).[62] These aforementioned results promoted us to construct more electrosynthesis of unavailable sulfur ylides by traditional methodologies and carry out the further application researches. Herein, the straightforward and unique preparation of sulfur ylides from the anode oxidative quinylation of sulfides with naphoquinones via radical-radical coupling under exogenous oxidant-, and transition metal-free conditions was developed (Scheme 1C-3). In addition, higher yields were

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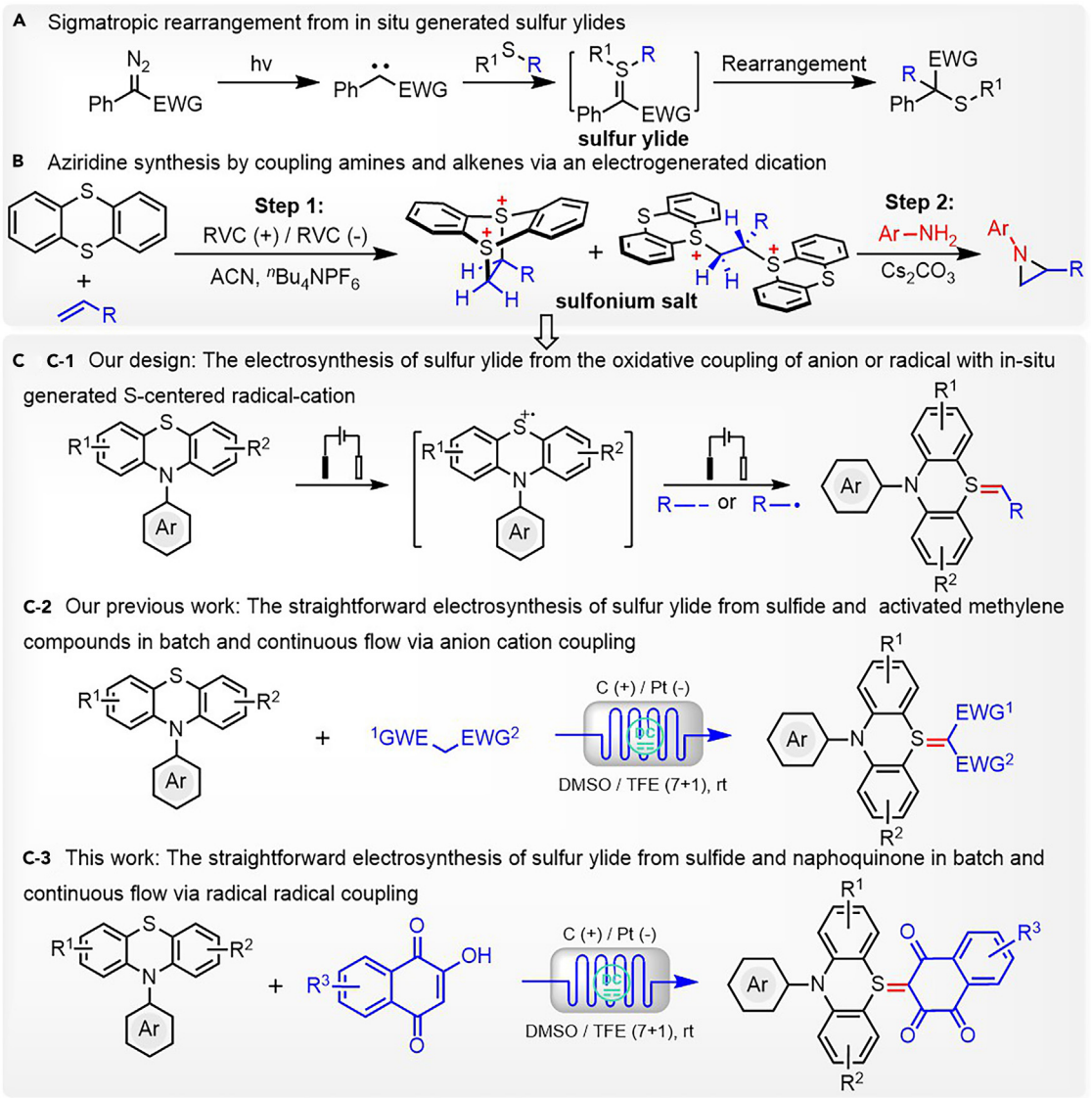


Scheme 1. Synthesis and transformation of S(IV) motifs

(A) Sigmatropic rearrangement from in situ generated sulfur ylides.

(B) Aziridine synthesis by coupling amines and alkenes via an electrogenerated dication.

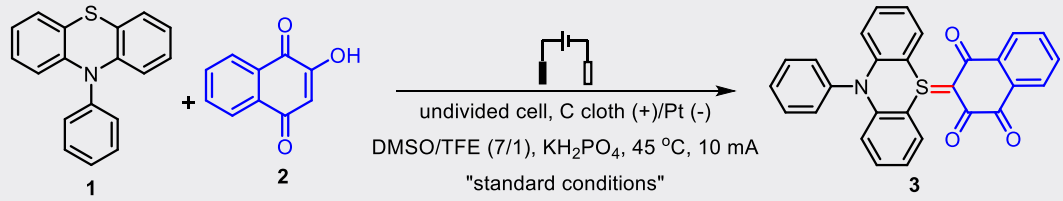
(C) C-1: Our design: The electrosynthesis of sulfur ylide from the oxidative coupling of anion or radical with in-situ generated S-centered radical-cation; C-2: Our previous work: The straightforward electrosynthesis of sulfur ylide from sulfide and activated methylene compounds in batch and continuous flow via anion cation coupling; C-3: This work: The straightforward electrosynthesis of sulfur ylide from sulfide and naphoquinone in batch and continuous flow via radical radical coupling.

obtained in the absence of quaternary ammonium salt under higher concentration of substrates, which contributed to simplifying purification process and solvent economy. Good scalable potential was observed using the continuous-flow reactor.

RESULTS AND DISCUSSION

The straightforwardly electrochemical oxidative coupling of easily or commercially available 10-phenyl-10*H*-phenothiazine **1** and 2-hydroxynaphthalene −1,4-dione **2** to the corresponding sulfur ylides was investigated (Table 1). Firstly, the reaction was carried out under the conditions that we developed for the electro-oxidative coupling of sulfides with activated methylene compounds in an undivided cell at a constant current of 10 mA with a carbon cloth anode, a platinum plate cathode using KH_2PO_4 and $^nBu_4NOAc$ as base and electrolyte, respectively (entry 2). The desired product was formed in 65% yield. To our delight, it was found that removal of electrolyte significantly improved the yield to 89% (entry 1).

Table 1. Optimization of the electrosynthesis of sulfur ylides



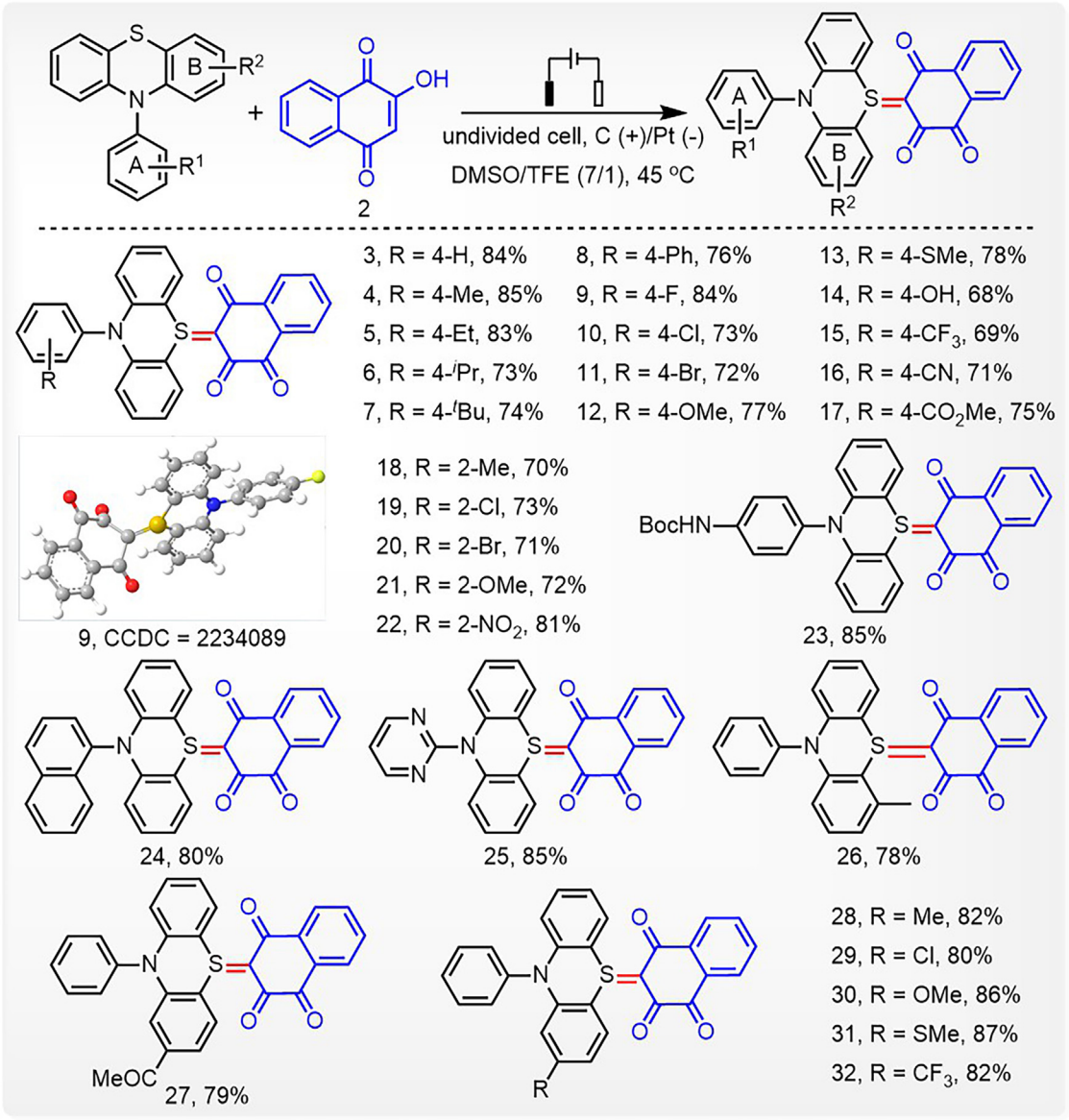
Entry	Variation from "standard conditions"	Yield (%)
1	none	89
2	1 equiv. $^{n}Bu_4NOAc$ was added	65
3	K_2HPO_4, K_3PO_4, K_2CO_3, NaH_2PO_4 or $NaHCO_3$ instead of KH_2PO_4	37, 41, 41, 69, 53
4	TEA, DBU or DIPEA instead of KH_2PO_4	0, 80, 76
5	DMF, ACN or DCE instead of DMSO	57, 41, 0
6	MeOH, EtOH or HFIP instead of TFE	73, 73, 61
7	DMSO, DMF or ACN as solvent	69, 77, 0
8	C rod, RVC or Pt anode	49, 69, 49
9	Ni or Fe cathode	0, 20
10	8 or 12 mA	85, 77
11	25°C	69
12	no current	0

Reaction conditions: undivided cell, C cloth anode (35 × 15 × 0.1 mm), Pt plate cathode (10 × 10 × 0.1 mm), **1** (0.6 mmol), **2** (0.6 mmol), KH_2PO_4 (0.4 mmol), DMSO/TFE (7 + 1) mL, 10 mA, 45°C, 5 h. Yields were determined by ^{1}H NMR using *p*-dinitrobenzene as the internal standard. NMR: nuclear magnetic resonance; TEA: triethylamine; DBU: 1,8-diazabicyclo[5,4,0]undec-7-ene; DIPEA: N,N-diisopropylethylamine; DMF: N,N-dimethylformamide; ACN: acetonitrile; DCE: 1,2-dichloroethane; DMSO: dimethyl sulfoxide; TFE: 2,2,2-trifluoroethanol; HFIP: 1,1,1,3,3,3-hexafluoro-2-propanol.

The inclusion of KH_2PO_4 was critical in the product formation, as replacing it with other base such as K_2HPO_4, K_3PO_4, K_2CO_3 or NaH_2PO_4 resulted in lower yields (entry 3). Some organic bases including TEA, DBU, and DIPEA were investigated (entry 4). It was found that use TEA completely abolished the formation of the desired product. However, comparable yield was obtained when DBU or DIPEA was involved. Using other solvent instead of the mixed solvent of DMSO/TFE (7/1) also led to the dramatic decrease of the yield (entries 5–7). It was noteworthy that desired product formation was completely abolished when DMSO was replaced by DCE or ACN was used solely (entries 5 and 7). Dramatic yield reduction was also observed when C rod, reticulated vitreous carbon (RVC) or Pt plate was used as anode or Fe was used as cathode (entries 8 and 9). No target product was detected with Ni as cathode, which might attribute to the following two reasons (entry 9). As the catalyst and the reactive site of electrosynthesis, electrode material plays a crucial role in the electrochemical reaction efficiency and selectivity. Furthermore, the reduction of TFE on cathode leads to the formation of H_2 and the base, which contributes to the balance of electron and prevents the decomposition of the target products. Moreover, the *in situ* generated base is profit for the deprotonation. However, Ni exhibits higher reduction potential of H^+ compared with Fe or Pt, which does detriment of the balance of electron and the deprotonation process. Decreasing the current to 8 mA gave a comparable yield (entry 10). However, lower yield was obtained under higher current density or lower temperature (entries 10 and 11). Importantly, the electric current was indispensable in this sulfur ylide electrosynthesis (entry 12).

With the optimal conditions defined, the reaction scopes in batch with substituents at sulfide and naphoquinone moieties were investigated (Schemes 2 and 3). To our delight, the phenyl ring **A** and **B** could both be substituted with electron donating or electron withdrawing groups with good to excellent yields obtained (**3**–**32**). Even though highly electron donating group OMe (**12**, **21**, **30**), SMe (**13**, **31**), or OH (**14**) or highly electron withdrawing group CN (**16**), CO_2Me (**17**), COMe (**27**), or NO_2 (**22**) was involved, this electrosynthesis of sulfur ylide proceed with good yield. In addition, excellent functional group compatibility was observed including H (**3**), alkyl groups (**4**–**7**, **18**, **26**, **28**), phenyl group (**8**), halogens (**9**–**11**, **19**, **20**, **29**), OMe (**12**, **21**, **30**), SMe (**13**, **31**), OH (**14**), CF_3 (**15**, **32**), CN (**16**) and CO_2Me (**17**), NO_2 (**22**), and COMe (**27**). No further oxidation of SMe was observed. Sulfide with substituent at the different position was converted into the sulfur ylide in good yield. Importantly, this electrosynthesis was compatible with protecting group NHBoc (**23**). No de-halogenated by-product was detected (**9**–**11**, **19**, **20**, **29**). Fused ring and heterocyclic ring could also be transformed into the desired product (**24**, **25**).

The scope investigation with respect to substituents at naphoquinone moiety revealed that naphoquinone moiety could also bear a series functional groups including alkyl group (**33**), halogens (**34**–**36**), and OMe (**37**, **38**). Besides, replacing phenyl ring of naphoquinone with naphthyl ring led to the formation of the corresponding product in good yield (**39**).

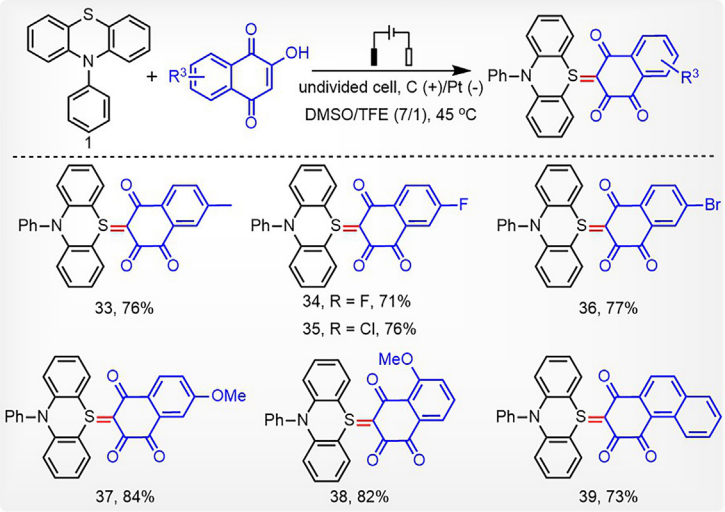


Scheme 2. Substrate scope of the synthesis of sulfur ylide with substituent at sulfide moiety

Reaction conditions, see Table 1, entry 1, isolated yield.

However, use commercially available raw materials such as phenothiazine, N-methyl-phenothiazine, menadione, and thianthrene completely abolished the formation of the desired products (Table S4). The corresponding sulfoxide by-product was obtained with 19% isolated yield when thianthrene was used (Table S4; Equation 1).

Intrigued by this attractive electrosynthesis of sulfur ylide from the coupling of sulfides with naphoquinones, the scale up investigations were carried out (Scheme 4). The concentration was investigated firstly (Scheme 4A). It was found that the comparable yield was obtained until the concentration based on **1** was increased to 0.1875 M, which revealed good scalable potential (Scheme 4A). However, dramatic yield reduction was observed under higher current density when five times substrates were involved (Scheme 4B). In addition, higher charge and longer electrolysis time were consumed to ensure the full conversion. It was noteworthy that further increasing the current density was unreachable because of the low conductivity in the absence of electrolyte, which also made this reaction unsuitable for scale up production. However, the inclusion of quaternary ammonium salt led to the dramatic decrease of the yield. In contrast, attributing to small electrode distance, large ratio of electrode surface-to-reactor volume and excellent mass and heat transfers of micro-flow cells,[63–65] the steady and scale up production might be realized easily under supporting electrolyte-free conditions or using a small amounts of quaternary ammonium salt. Therefore, the further scale up investigations in continuous flow were carried out using a sandwich structure with two aluminum holders, an electrolytic cell, two comb-like electrodes and a cover plate (Scheme 4C). A comb-like C electrode and a comb-like Pt-plated electrode



Scheme 3. Substrate scope of the synthesis of sulfur ylide with substituent at naphoquinone moiety
Reaction conditions, see Table 1, entry 1, isolated yield.

interlaced together in the electrolytic cell to form the reaction channel with 4 mm cell path height, 1 mm electrode distance, 1,260 mm cell path length, and 5.5 mL residence volume. To our delight, 98% yield was obtained using the micro-flow cell above at room temperature (Scheme 4D, entries 1 and 2). Moreover, the daily production was increased from 0.75 g/d to 21.29 g/d with lower charge consumed (Scheme 4D, entries 3 and 4). These results revealed that scale up production using this micro-flow cell could be achieved easily through extending the electrolysis time and paralleling operation at the same reaction conditions.

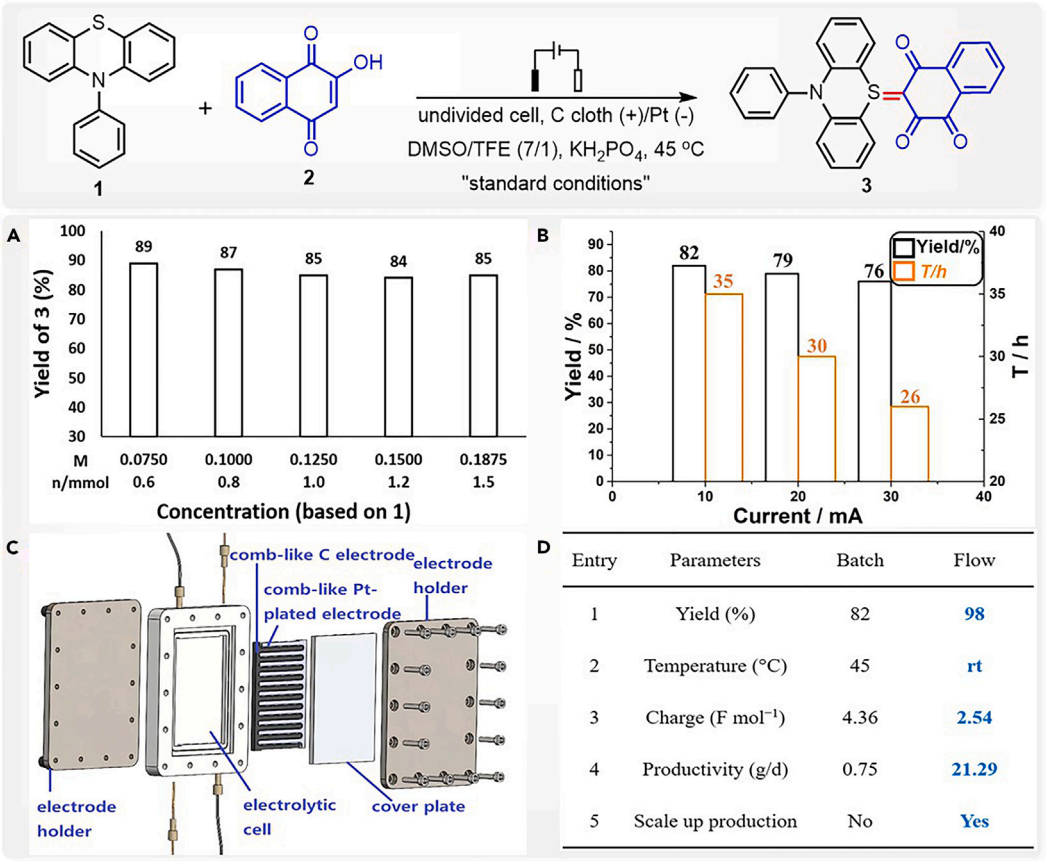
More substrates were investigated using micro-flow cells (Scheme 5). To our delight, comparable or higher yield was obtained when the reaction was carried out using micro-flow cells. The synthetic applications of the prepared sulfur ylides were carried out. The cross-coupling of the prepared sulfur ylides and boronic acids or fumarates to the corresponding C-C coupling products was investigated (Scheme S1). However, no desired product was detected which might attribute to the excellent stability of the prepared sulfur ylides. And, further application studies are ongoing in our laboratory.

Intrigued by this straightforward electrosynthesis of sulfur ylides from the oxidative coupling of sulfides with naphoquinones in batch and continuous flow, a series of mechanistic studies including radical-trapping and cyclic voltammetry experiments were carried out (Scheme 6; Figure 1). Adding radical scavenger including 1,1-diphenylethylene or BHT resulted in dramatic decrease of the yields (Scheme 6-1 a1, 6-2). The formation of the desired product was completely abolished when TEMPO was involved (Scheme 6-1 a2). Moreover, the corresponding radical trapping product was generated in 45% isolated yield in the presence of BHT, which lent support to the hypothesis that the radical-radical coupling gave the target product (Scheme 6-2). Furthermore, an obvious oxidation peak of **1** was observed at 1.30 V, which might result from the oxidation of sulfur (Figure 1A, black line). The oxidation potential of **1** shifted to 1.65 V in the presence of base (Figure 1A, red line). The solution of substrate **2** showed a slightly higher oxidation peak at about 1.88 V (Figure 1B, black and red lines). It was noteworthy that the mixtures of **1** and **2** only showed one obvious oxidation peak at about 1.62 V, which was consistent with the oxidation potential of **1** (Figure 1A, blue line). These results of cyclic voltammetry experiments revealed that **1** was likely to be oxidized at anode firstly to give the stable sulfur-centered radical-cation intermediate, which might oxidize **2** to the corresponding intermediate via single electron transfer (SET) process.

On the basis of the aforementioned mechanistic findings and previous reports,[66–69] a plausible reaction process of this electrochemically oxidative coupling of sulfides with naphoquinones to the corresponding sulfur ylides was put forward (Scheme 7). The stable sulfur radical cation intermediate **1-1** was generated from the anode oxidation firstly. The corresponding radical cation intermediate **2-1** was formed through a SET from **2** to **1-1** with the regeneration of **1**. The radical-radical coupling between **1-1** and **2-1** and deprotonation led to the formation of the cation intermediate **3-1**, which was transformed into the desired product from the further deprotonation in the presence of base.

Conclusion

In summary, a straightforward electrosynthesis of sulfur ylides from the oxidative quinylation of sulfides with naphoquinones via radical-radical coupling under exogenous oxidant and transition metal-free conditions in batch and continuous flow was developed. Good concentration tolerance and quaternary ammonium salt free conditions contributed to solvent economy and simplifying purification process. In addition, Good scalable potential, higher daily production and steady production were achieved attributing to the use of micro-flow cells.



Entry	Parameters	Batch	Flow
1	Yield (%)	82	**98**
2	Temperature (°C)	45	**rt**
3	Charge (F mol^{-1})	4.36	**2.54**
4	Productivity (g/d)	0.75	**21.29**
5	Scale up production	No	**Yes**

Scheme 4. Scale up investigations in batch and continuous flow

(A) the investigation into concentration in batch.

(B) the investigation into current density in batch, **1** (3 mmol).

(C) schematic diagram of the continuous-flow electrochemical reactor.

(D) comparison of optimized conditions of batch and continuous flow, batch: **1** (3 mmol).

Limitations of the study

This work reports a straightforward electrosynthesis of sulfur ylides from the oxidative quinylation of sulfides with naphoquinones. Although excellent functional group compatibility was demonstrated, the phenothiazine rings and aromatic rings on the linking nitrogen of the corresponding sulfide substrates are indispensable.

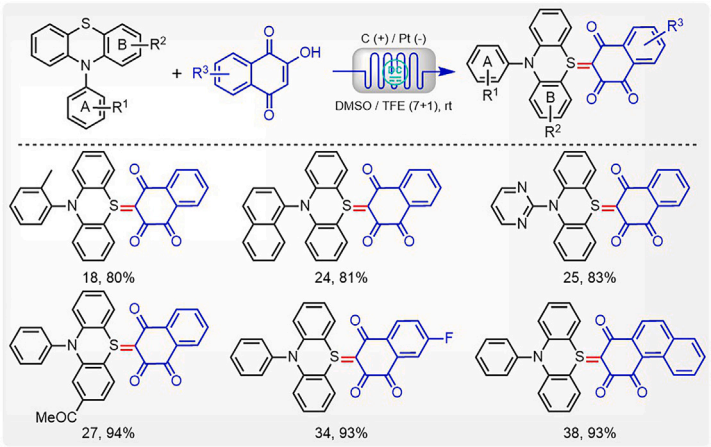
STAR★METHODS

Detailed methods are provided in the online version of this paper and include the following:

- KEY RESOURCES TABLE
- RESOURCE AVAILABILITY
  - Lead contact
  - Materials availability
  - Data and code availability
- METHOD DETAILS
  - Preparation of substrates
  - Preparation of products

SUPPLEMENTAL INFORMATION

Supplemental information can be found online at https://doi.org/10.1016/j.isci.2023.108605.



Scheme 5. Substrate scope of the synthesis of sulfur ylide using micro-flow cells
Reaction conditions, see Table S3, entry 3, isolated yield.

ACKNOWLEDGMENTS

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AUTHOR CONTRIBUTIONS

C.-K.L. and K.G. designed the experiments; C.-K.L. and X.-X.H. wrote this paper; X.-X.H., Y.-F.Y., and C.-C.Y. conducted the experiments; C.-K.L., X.-X.H., W.-J.G., and H.Q. analyzed the data; C.-K.L., X.Y., and Z.F. designed and produced the continuous flow electrolysis cells; K.G. guided the research.

DECLARATION OF INTERESTS

The authors declare no competing interests.

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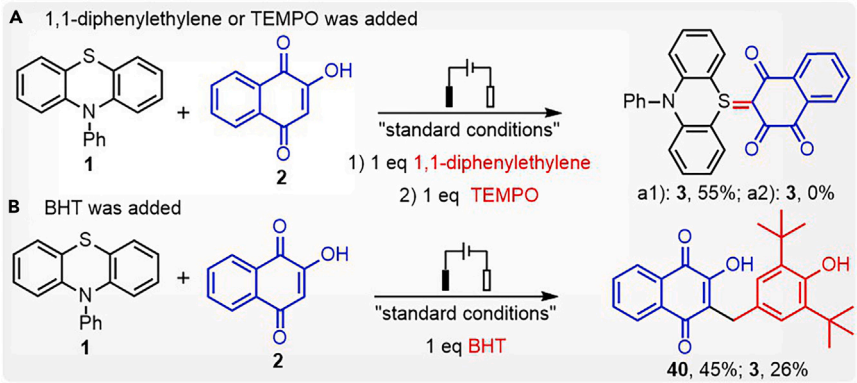

Scheme 6. Radical-trapping experiments
(A) 1,1-diphenylethylene or TEMPO was added. TEMPO: 2,2,6,6-Tetramethylpiperidinooxy.
(B) BHT was added. BHT: butylated hydroxytoluene.

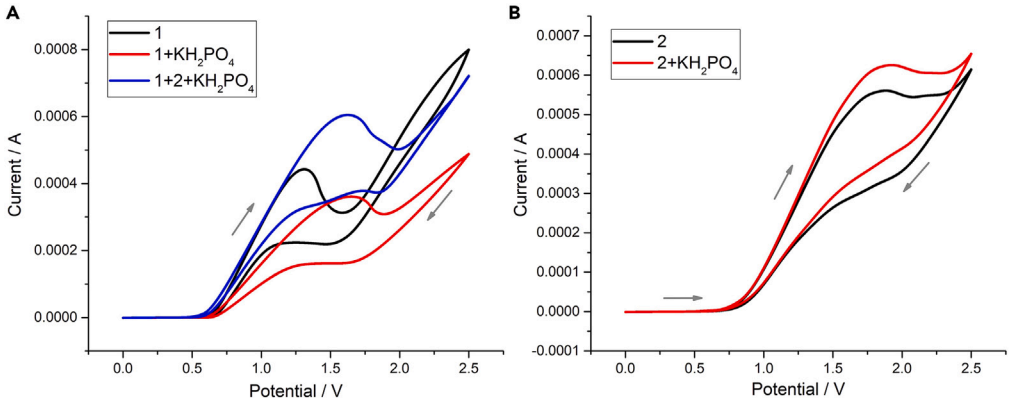

Figure 1. Cyclic voltammetry experiments

IUPAC Convention, rt.

(A) Cyclic voltammetry experiments of substrate **1** and reaction mixture: (black) **1**; (red) **1** and KH_2PO_4; (blue) **1**, **2** and KH_2PO_4.

(B) Cyclic voltammetry experiments of substrate **2**: (black) **2**; (red) **2** and KH_2PO_4.

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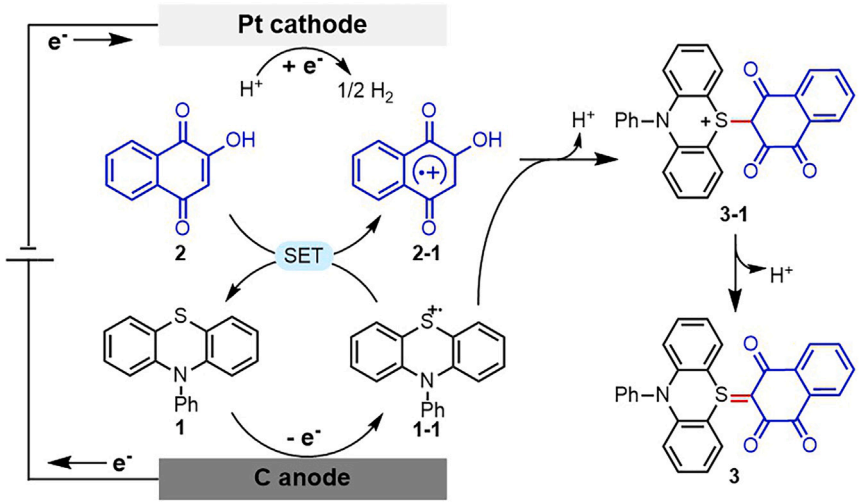

Scheme 7. Plausible mechanism for electrosynthesis of sulfur ylides

sulfide complexes in the synthesis of indoles, oxindoles, and alkylated aromatic amines. J. Am. Chem. Soc. *95*, 6508–6509.
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STAR★METHODS

KEY RESOURCES TABLE

REAGENT or RESOURCE	SOURCE	IDENTIFIER
Phenothiazine	Energy chemical	Cas: 92-84-2
Bromobenzene	Energy chemical	Cas: 108-86-1
Bis(dibenzylideneacetone)palladium	Energy chemical	Cas: 32005-36-0
Tri-*tert*-butylphosphine tetrafluoroborate	Energy chemical	Cas: 131274-22-1
Sodium tert-butoxide	Energy chemical	Cas: 865-48-5
Ethyeneglycol	Energy chemical	Cas: 123-91-1
Potassium phosphate monobasic	Energy chemical	Cas: 7778-77-0
Tetrabutylammonium acetate	Energy chemical	Cas: 10534-59-5
Dimethyl sulfoxide	Energy chemical	Cas: 67-68-5
2,2,2-Trifluoroethanol	Energy chemical	Cas: 75-89-8
2-Hydroxy-1,4-naphoquinone	Energy chemical	Cas: 83-72-7
4-Bromotoluene	Energy chemical	Cas: 106-38-7
4-Bromoethylbenzene	Energy chemical	Cas: 1585-07-5
4-Bromocumene	Energy chemical	Cas: 586-61-8
1-Bromo-4-*tert*-butylbenzene	Energy chemical	Cas: 3972-65-4
4-Bromobiphenyl	Energy chemical	Cas: 92-66-0
4-Bromofluorobenzene	Energy chemical	Cas: 460-00-4
4-Bromochlorobenzene	Energy chemical	Cas: 106-39-8
1,4-Dibromobenzene	Energy chemical	Cas: 106-37-6
4-Bromoanisol	Energy chemical	Cas: 104-92-7
4-Bromothioanisole	Energy chemical	Cas: 104-95-0
4-Bromophenol	Energy chemical	Cas: 106-41-2
4-Bromobenzotrifluoride	Energy chemical	Cas: 402-43-7
4-Bromobenzonitrile	Energy chemical	Cas: 623-00-7
Methyl 4-bromobenzoate	Energy chemical	Cas: 619-42-1
2-Bromotoluene	Energy chemical	Cas: 95-46-5
2-Bromochlorobenzene	Energy chemical	Cas: 694-80-4
1,2-Dibromobenzene	Energy chemical	Cas: 583-53-9
2-Bromoanisole	Energy chemical	Cas: 578-57-4
4-Bromobenzoic acid *tert*-butyl ester	Energy chemical	Cas: 59247-47-1
1-Bromonaphthalene	Energy chemical	Cas: 90-11-9
2-Bromopyrimidine	Energy chemical	Cas: 4595-60-2
7-Methyl-1-tetralone	Energy chemical	Cas: 22009-37-6
7-Fluoro-1-tetealone	Energy chemical	Cas: 2840-44-0
7-Chloro-1-tetralone	Energy chemical	Cas: 26673-32-5
7-Bromo-1-tetralone	Energy chemical	Cas: 32281-97-3
7-Methoxy-1-tetralone	Energy chemical	Cas: 6836-19-7
1,2,3,4-Tetrahydrophenanthrene-4-one	Energy chemical	Cas: 778-48-3
Carbon cloth	Shanghai Jing Chong Electronic Technology Development Co., Ltd	
Graphite rod (ø 6 mm)	Shanghai Jing Chong Electronic Technology Development Co., Ltd	

(*Continued on next page*)

Continued

REAGENT or RESOURCE	SOURCE	IDENTIFIER
Platinum plate	Shanghai Jing Chong Electronic Technology Development Co., Ltd	
Ni plate	Shanghai Jing Chong Electronic Technology Development Co., Ltd	
Fe plate	Shanghai Jing Chong Electronic Technology Development Co., Ltd	
Reticulated vitreous carbon (RVC)	Gaoss Union (Tianjin) Photoelectric Technology Co., Ltd	
Deposited data		
CIF of **9**	CCDC 2234089	https://www.ccdc.cam.ac.uk/structures/
Other		
Silica gel (200–300 mesh)	Shanghai Titan Scientific Co., Ltd	
Thin-layer chromatography using TLC silica gel plates	Yantai Xinnuo New Material Technology Co., Ltd	
AVIII 400 MHz	Bruker	https://bruker.com
X-ray diffraction	Rigaku	https://www.rigaku.com/zh-hans
HRMS	Agilent	https://www.agilent.com.cn/
Software and algorithms		
ChemDraw Ultra 12.0	https://www.perkinelmer.com/category/chemdraw	

RESOURCE AVAILABILITY

Lead contact

Further information and requests should be directed to the lead contact, Chengkou Liu (liuchengkou@njtech.edu.cn).

Materials availability

All commercially available reagents and solvents were used without any further purification.

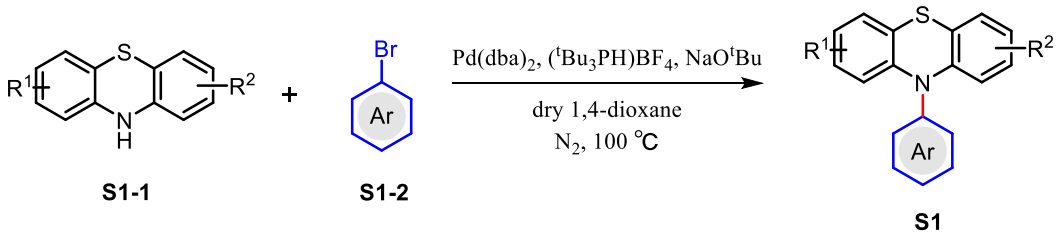
Data and code availability

Crystallographic data for the structure reported in this article had been deposited at the Cambridge Crystallographic Data Center (CCDC) under accession number CCDC 2234089 (**9**). Copies of the data can be obtained free of charge from https://www.ccdc.cam.ac.uk/structures/. All other data are available from the lead contact upon reasonable request.

METHOD DETAILS

Preparation of substrates

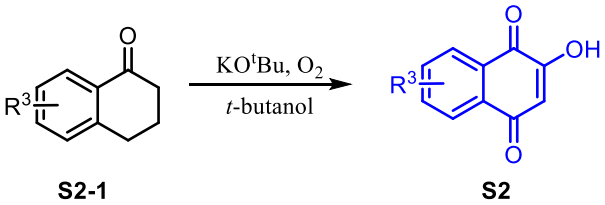
General procedure for the synthesis of substrates (S1)



An oven-dried round bottom flask equipped with a magnetic stirring bar was charged with the phenothiazine **S1-1** (1.1 eq, 11.0 mmol), $Pd(dba)_2$ (0.06 eq, 0.6 mmol, 340.2 mg), tri-*tert*-butylphosphane tetrafluoroborate (0.06 eq, 0.6 mmol, 174.1 mg) and sodium *tert*-butoxide (1.15 eq, 11.5 mmol, 1105.2 mg) under nitrogen atmosphere[70]. Then, dry 1,4-dioxane (50 mL) was added into the mixture followed by the addition of substituted bromobenzene **S1-2** (1 eq, 10 mmol). The reaction mixture was stirred at 100°C for 14 h. The reaction solution was

diluted with ethyl acetate (100 mL) and washed with H_2O (100 mL). The separated organic layer was dried over anhydrous Na_2SO_4 and filtered. The filtrate was concentrated under reduced pressure to give the crude product, which was purified by column chromatographic separation (petroleum ether/ethyl acetate 100:1) to obtain the desired product **S1**.

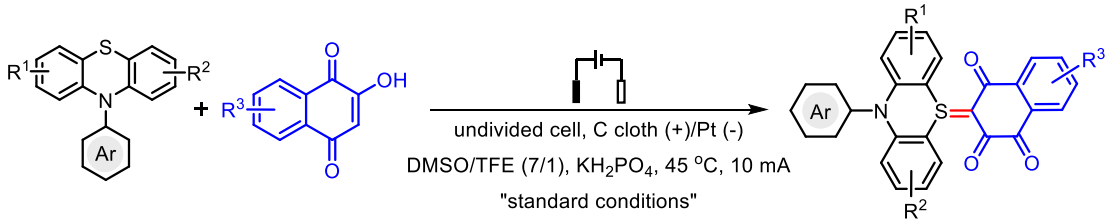
General procedure for the synthesis of substrates (S2)



The reaction flask was first degassed and then sealed with a O_2 balloon[71]. All the reactants and solvent were added under a O_2 atmosphere. The solution of atetralone **S2-1** (10 mmol) in *t*-butanol (30 mL) was added into the solution of potassium *t*-butoxide in *t*-butanol (1 M, 50 mL). Then, the mixture was stirred under an oxygen atmosphere. After 2 h, the reaction mixture was acidified with HCl (1 M, 20 mL) and diluted extracted with CH_2Cl_2 (200 mL). The combined organic solution was then washed with a saturated of $NaHCO_3$ aqueous solution (20 mL) and HCl aqueous solution (1 M, 40 mL). The separated organic layer was dried with anhydrous Na_2SO_4 and filtered. The filtrate was concentrated under reduced pressure to give the crude product, which was purified by column chromatographic separation (petroleum ether/ethyl acetate: 1:3) to obtain the desired product **S2**.

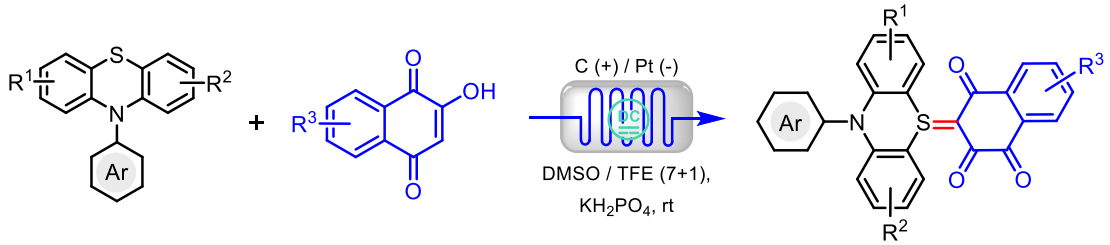
Preparation of products

General procedure for the electrosynthesis of sulfur ylides in batch



In an undivided cell equipped with a carbon cloth anode (35 mm × 15 mm) and a Pt plate cathode (10 mm × 10 mm x 0.1 mm), sulfide (0.6 mmol), naphoquinone (0.6 mmol) and KH_2PO_4 (0.4 mmol, 54.4 mg) were dissolved in a mixed solvent of DMSO/TFE (7 mL/1 mL). At 45°C, the mixture above was stirred and electrolyzed at a constant current of 10 mA for 5 h under the open air. The reaction solution was diluted with ethyl acetate (100 mL) and washed with H_2O (100 mL). The separated organic layer was dried over anhydrous Na_2SO_4 and filtered. The filtrate was concentrated under reduced pressure to give the crude product, which was purified by column chromatographic separation (petroleum ether/dichloromethane/ethyl acetate 30:20:5) to obtain the desired sulfur ylides.

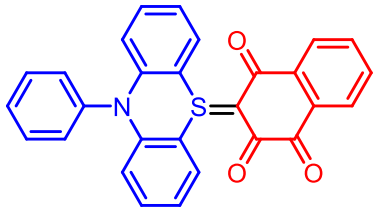
General procedure for the electrosynthesis of sulfur ylides in continuous flow



Sulfide (0.6 mmol), naphoquinone (0.6 mmol), KH_2PO_4 (0.4 mmol, 54.4 mg) and ^{n}Bu$_4$NOAc (0.2 mmol, 60.3 mg) were dissolved in a mixed solvent of DMSO/TFE (7 mL/1 mL). At ambient temperature, the reaction mixtures were introduced into the reactor at 0.45 mL/min at a constant current of 200 mA. The reaction solution was diluted with ethyl acetate (100 mL) and washed with H_2O (100 mL). The separated organic

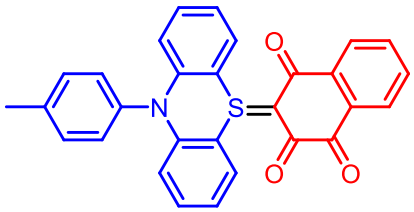
layer was dried over anhydrous Na_2SO_4 and filtered. The filtrate was concentrated under reduced pressure to give the crude product, which was purified by column chromatographic separation (petroleum ether/dichloromethane/ethyl acetate 30:20:5) to obtain the desired product.

Characterization of products 3–39



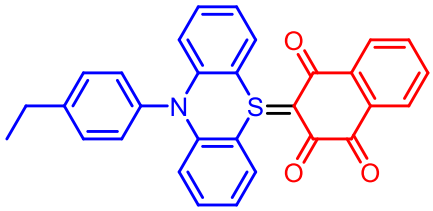
3

3-(10-phenyl-10H-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione (**3**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 225.3 mg, 84%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.21 (dd, *J* = 7.9, 1.3 Hz, 1H), 8.06 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.80–7.68 (m, 5H), 7.66–7.54 (m, 4H), 7.25–7.18 (m, 2H), 7.04–6.97 (m, 2H), 6.46 (dd, *J* = 8.8, 1.0 Hz, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.10, 178.07, 172.06, 143.31, 139.18, 134.64, 134.36, 132.75, 132.04, 131.43, 131.10, 130.46, 129.63, 127.40, 126.93, 122.52, 116.99, 107.66, 103.78; HRMS (ESI-TOF) Calcd for $C_{28}H_{18}NO_3S$ $[M + H]^+$: 448.1002; found: 448.1006.



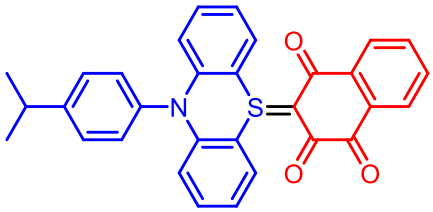
4

3-(10-(p-tolyl)-10H-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione (**4**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 235.2 mg, 85%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.21 (dd, *J* = 7.8, 1.2 Hz, 1H), 8.05 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.70 (td, *J* = 7.6, 1.4 Hz, 1H), 7.61 (t, *J* = 7.0 Hz, 2H), 7.59–7.48 (m, 5H), 7.24–7.18 (m, 2H), 7.03–6.95 (m, 2H), 6.49 (d, *J* = 8.7 Hz, 2H), 2.53 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.13, 178.06, 172.04, 143.42, 139.67, 136.45, 134.65, 134.34, 132.69, 132.02, 130.70, 130.40, 127.39, 126.93, 122.41, 117.05, 107.71, 103.73, 21.44; HRMS (ESI-TOF) Calcd for $C_{29}H_{20}NO_3S$ $[M + H]^+$: 462.1158; found: 462.1165.



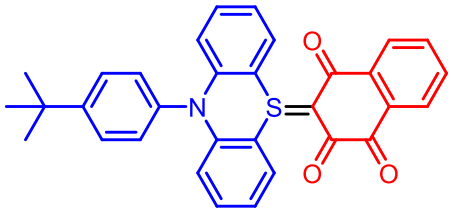
5

3-(10-(4-ethylphenyl)-10H-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione (**5**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 236.6 mg, 83%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.21 (dd, *J* = 7.9, 1.3 Hz, 1H), 8.05 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.70 (td, *J* = 7.5, 1.4 Hz, 1H), 7.64 (d, *J* = 7.8 Hz, 2H), 7.60–7.51 (m, 5H), 7.25–7.19 (m, 2H), 6.99 (td, *J* = 7.6, 1.2 Hz, 2H), 6.49 (dd, *J* = 8.7, 1.1 Hz, 2H), 2.83 (q, *J* = 7.6 Hz, 2H), 1.38 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.13, 178.06, 172.05, 145.87, 143.44, 136.60, 134.65, 134.34, 132.70, 132.02, 130.78, 130.72, 130.40, 127.39, 126.92, 122.40, 117.07, 107.73, 103.71, 28.71, 15.44; HRMS (ESI-TOF) Calcd for $C_{30}H_{22}NO_3S$ $[M + H]^+$: 476.1315; found: 476.1321.



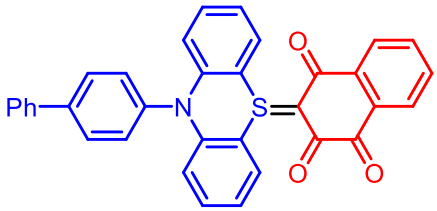
6

3-(10-(4-isopropylphenyl)-10H-5λ⁴-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione (**6**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 214.2 mg, 73%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.21 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.06 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.70 (td, *J* = 7.6, 1.4 Hz, 1H), 7.64 (d, *J* = 7.9 Hz, 2H), 7.59–7.53 (m, 5H), 7.25–7.19 (m, 2H), 7.03–6.96 (m, 2H), 6.49 (dd, *J* = 8.7, 1.2 Hz, 2H), 3.09 (p, *J* = 6.9 Hz, 1H), 1.38 (d, *J* = 7.0 Hz, 6H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.13, 178.06, 172.05, 150.48, 143.46, 136.64, 134.68, 134.33, 132.69, 132.05, 132.01, 130.68, 130.39, 129.33, 127.39, 126.91, 122.40, 117.09, 107.71, 103.75, 34.03, 24.04; HRMS (ESI-TOF) Calcd for $C_{31}H_{24}NO_3S$ [M + H]$^{+}$: 490.1471; found: 490.1476.



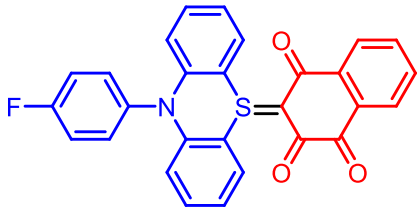
7

*3-(10-(4-(tert-*butyl)phenyl)-10*H*-5λ⁴-phenothiazin-5-ylidene)naphthalene-1,2,4(3*H*)-trione (**7**):. Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 223.4 mg, 74%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.20 (d, *J* = 7.7 Hz, 1H), 8.05 (d, *J* = 7.6 Hz, 1H), 7.75–7.62 (m, 5H), 7.55 (d, *J* = 8.4 Hz, 3H), 7.23 (t, *J* = 8.0 Hz, 2H), 6.99 (t, *J* = 7.5 Hz, 2H), 6.49 (d, *J* = 8.5 Hz, 2H), 1.45 (s, 9H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.10, 178.03, 172.06, 152.80, 143.46, 136.38, 134.70, 134.31, 132.68, 132.00, 130.39, 130.33, 128.25, 127.38, 126.90, 122.40, 117.10, 107.65, 103.80, 35.00, 31.46; HRMS (ESI-TOF) Calcd for $C_{32}H_{26}NO_3S$ [M + H]$^{+}$: 504.1628; found: 504.1628.



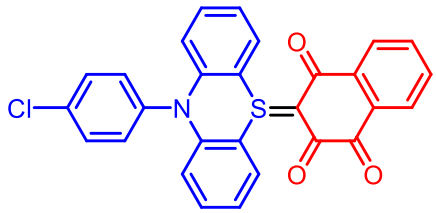
8

3-(10-([1,1′-biphenyl]-4-yl)-10H-5λ⁴-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione (**8**):. Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 238.5 mg, 76%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.22 (dd, *J* = 7.9, 1.3 Hz, 1H), 8.06 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.94 (d, *J* = 8.5 Hz, 2H), 7.83 (d, *J* = 7.9 Hz, 2H), 7.75–7.68 (m, 3H), 7.61–7.51 (m, 5H), 7.47–7.42 (m, 1H), 7.28–7.22 (m, 2H), 7.05–6.99 (m, 2H), 6.57 (dd, *J* = 8.7, 1.1 Hz, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.12, 178.10, 172.08, 143.34, 142.54, 139.87, 138.24, 134.65, 134.37, 132.80, 132.06, 131.43, 130.50, 130.02, 129.08, 128.09, 127.43, 127.32, 126.95, 122.58, 117.07, 107.65, 103.87; HRMS (ESI-TOF) Calcd for $C_{34}H_{22}NO_3S$ [M + H]$^{+}$: 524.1315; found: 524.1321.



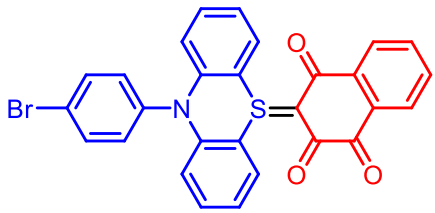
9

3-(10-(4-fluorophenyl)-10H-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione (**9**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 234.4 mg, 84%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.20 (d, *J* = 7.7 Hz, 1H), 8.04 (d, *J* = 7.6 Hz, 1H), 7.79 (dd, *J* = 8.2, 4.7 Hz, 2H), 7.70 (t, *J* = 7.5 Hz, 1H), 7.60–7.54 (m, 3H), 7.42 (t, *J* = 8.3 Hz, 2H), 7.28–7.22 (m, 2H), 7.02 (t, *J* = 7.5 Hz, 2H), 6.47 (d, *J* = 8.6 Hz, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.05, 178.07, 172.02, 164.15, 161.67, 143.35, 135.04 (d, *J* = 3.5 Hz), 134.58, 134.39, 133.12 (d, *J* = 8.7 Hz), 132.85, 132.08, 132.01, 130.57, 127.41, 126.93, 122.72, 118.59, 118.36, 116.85, 107.52, 103.98; ^{19}F NMR (376 MHz, Chloroform-*d*) δ −110.48; HRMS (ESI-TOF) Calcd for $C_{28}H_{17}FNO_3S$ [M + H]$^+$: 466.0908; found: 466.0905.



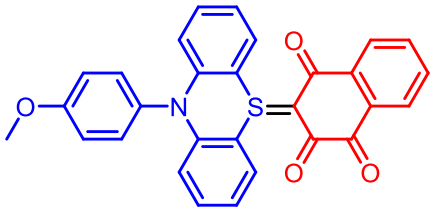
10

3-(10-(4-chlorophenyl)-10H-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione (**10**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 210.7 mg, 73%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.21 (dd, *J* = 7.7, 1.3 Hz, 1H), 8.05 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.77–7.69 (m, 5H), 7.61–7.55 (m, 3H), 7.28–7.22 (m, 2H), 7.06–7.00 (m, 2H), 6.47 (dd, *J* = 8.7, 1.1 Hz, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.06, 178.11, 172.03, 143.13, 137.66, 135.70, 134.57, 134.41, 132.87, 132.71, 132.11, 132.02, 131.77, 130.60, 127.44, 126.95, 122.79, 116.81, 107.49, 104.01; HRMS (ESI-TOF) Calcd for $C_{28}H_{17}ClNO_3S$ [M + H]$^+$: 482.0612; found: 482.0611.



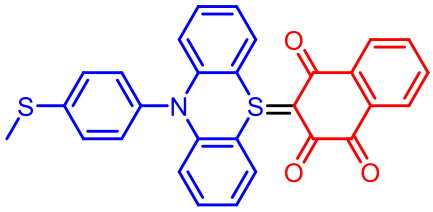
11

3-(10-(4-bromophenyl)-10H-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione (**11**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 226.8 mg, 72%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.21 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.06 (dd, *J* = 7.6, 1.3 Hz, 1H), 7.87 (d, *J* = 8.2 Hz, 2H), 7.74–7.66 (m, 3H), 7.61–7.54 (m, 3H), 7.28–7.22 (m, 2H), 7.06–7.00 (m, 2H), 6.46 (dd, *J* = 8.6, 1.1 Hz, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.07, 178.13, 172.04, 143.05, 138.20, 134.80, 134.57, 134.42, 133.05, 132.87, 132.12, 132.02, 130.61, 127.45, 126.95, 123.83, 122.81, 116.81, 107.47, 104.01; HRMS (ESI-TOF) Calcd for $C_{28}H_{17}BrNO_3S$ [M + H]$^+$: 526.0107; found: 526.0108.



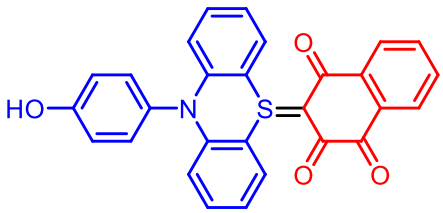
12

3-(10-(4-methoxyphenyl)-10H-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione (**12**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 220.4 mg, 77%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.21 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.05 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.72–7.63 (m, 3H), 7.59–7.52 (m, 3H), 7.26–7.19 (m, 4H), 7.02–6.97 (m, 2H), 6.52 (dd, *J* = 8.7, 1.1 Hz, 2H), 3.94 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.11, 178.04, 172.03, 160.15, 143.67, 134.66, 134.32, 132.71, 132.07, 132.00, 131.63, 130.41, 127.37, 126.92, 122.43, 117.06, 116.45, 107.69, 103.81, 55.66; HRMS (ESI-TOF) Calcd for $C_{29}H_{20}NO_4S$ [M + H]$^+$: 478.1108; found: 478.1110.



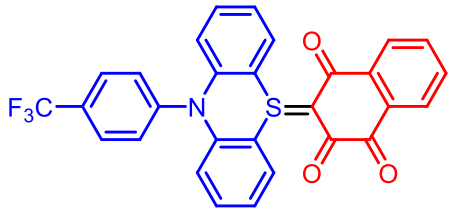
13

3-(10-(4-(methylthio)phenyl)-10H-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione (**13**):. Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 230.8 mg, 78%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.21 (d, *J* = 7.7 Hz, 1H), 8.05 ((dd, *J* = 7.8, 1.2 Hz, 1H), 7.73–7.63 (m, 3H), 7.61–7.52 (m, 5H), 7.26–7.21 (m, 2H), 7.01 (t, *J* = 7.5 Hz, 2H), 6.51 (d, *J* = 8.6 Hz, 2H), 2.61 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.10, 178.08, 172.03, 143.35, 140.92, 135.70, 134.62, 134.37, 132.78, 132.05, 131.40, 130.48, 128.38, 127.41, 126.93, 122.58, 116.99, 107.61, 103.85, 15.43; HRMS (ESI-TOF) Calcd for $C_{29}H_{20}NO_3S_2$ [M + H]$^+$: 494.0879; found: 494.0881.



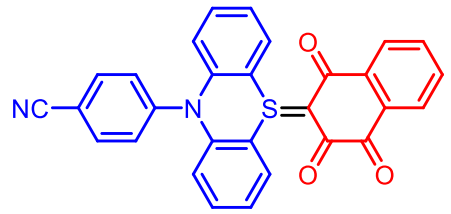
14

3-(10-(4-hydroxyphenyl)-10H-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione (**14**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 188.9 mg, 68%; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.09 (s, 1H), 8.05 (d, *J* = 7.7 Hz, 1H), 7.91 (d, *J* = 7.6 Hz, 1H), 7.81 (t, *J* = 7.5 Hz, 1H), 7.69 (t, *J* = 7.5 Hz, 1H), 7.60–7.49 (m, 4H), 7.41–7.33 (m, 2H), 7.16 (d, *J* = 8.3 Hz, 2H), 7.08 (t, *J* = 7.5 Hz, 2H), 6.50 (d, *J* = 8.6 Hz, 2H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 182.75, 177.04, 171.69, 158.61, 143.61, 135.12, 134.52, 133.36, 132.87, 132.06, 131.94, 130.74, 130.34, 127.22, 126.79, 122.95, 118.39, 117.04, 107.87, 104.56; HRMS (ESI-TOF) Calcd for $C_{28}H_{18}NO_4S$ [M + H]$^+$: 464.0951; found: 464.0961.



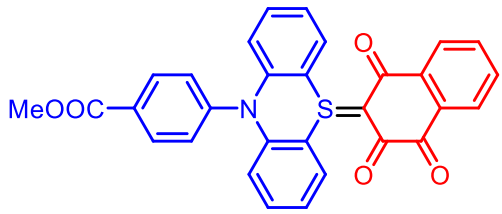
15

3-(10-(4-(trifluoromethyl)phenyl)-10H-5λ⁴-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione (**15**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 213.2 mg, 69%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.21 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.06–7.96 (m, 5H), 7.71 (td, *J* = 7.6, 1.3 Hz, 1H), 7.61–7.55 (m, 3H), 7.29–7.23 (m, 2H), 7.07–7.02 (m, 2H), 6.40 (dd, *J* = 8.7, 1.1 Hz, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.01, 178.10, 172.04, 142.88, 142.51 (q, *J* = 7.0 Hz), 134.55, 134.42, 132.94, 132.41, 132.13, 132.08, 132.01, 131.75, 131.42, 130.69, 128.63 (q, *J* = 3.6 Hz), 127.77, 127.44, 126.93, 125.06, 122.98, 122.35, 116.69, 107.32, 104.17; ^{19}F NMR (376 MHz, Chloroform-*d*) δ −62.51; HRMS (ESI-TOF) Calcd for $C_{29}H_{17}F_3NO_3S$ $[M + H]^+$: 516.0876; found: 516.0882.



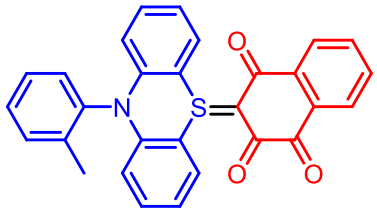
16

4-(5-(1,3,4-trioxo-3,4-dihydronaphthalen-2(1H)-ylidene)-5λ⁴-phenothiazin-10(5H)-yl)benzonitrile (**16**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 201.1 mg, 71%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.21 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.09–7.98 (m, 5H), 7.71 (td, *J* = 7.5, 1.3 Hz, 1H), 7.63–7.55 (m, 3H), 7.29–7.24 (m, 2H), 7.10–7.04 (m, 2H), 6.37 (dd, *J* = 8.7, 1.0 Hz, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 182.97, 178.14, 172.05, 143.41, 142.68, 135.39, 134.48, 133.02, 132.80, 132.20, 131.99, 130.81, 127.48, 126.95, 123.20, 117.86, 116.57, 113.97, 107.19, 104.32; HRMS (ESI-TOF) Calcd for $C_{29}H_{17}N_2O_3S$ $[M + H]^+$: 473.0954; found: 473.0965.



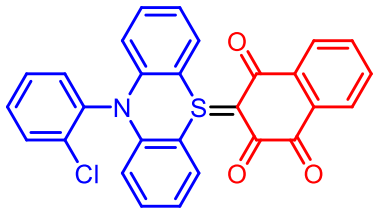
17

methyl 4-(5-(1,3,4-trioxo-3,4-dihydronaphthalen-2(1H)-ylidene)-5λ⁴-phenothiazin-10 (5H)-yl)benzoate (**17**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 227.3 mg, 75%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.41 (d, *J* = 8.1 Hz, 2H), 8.22 (dd, *J* = 7.8, 1.2 Hz, 1H), 8.07 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.89 (d, *J* = 8.1 Hz, 2H), 7.71 (td, *J* = 7.6, 1.2 Hz, 1H), 7.62–7.54 (m, 3H), 7.26–7.20 (m, 2H), 7.05 (t, *J* = 7.4 Hz, 2H), 6.41 (dd, *J* = 8.6, 1.1 Hz, 2H), 4.02 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.04, 178.14, 172.08, 166.19, 143.30, 142.88, 134.60, 134.41, 132.88, 132.80, 132.13, 131.51, 130.63, 127.46, 126.96, 122.85, 116.75, 107.57, 104.08, 52.60; HRMS (ESI-TOF) Calcd for $C_{30}H_{20}NO_5S$ $[M + H]^+$: 506.1057; found: 506.1051.



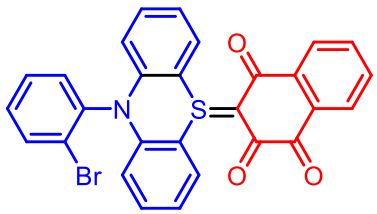
18

3-(10-(*o*-tolyl)-10*H*-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3*H*)-trione (18): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 193.7 mg, 70%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.14 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.10–8.05 (m, 1H), 8.02–7.97 (m, 1H), 7.66–7.59 (m, 1H), 7.54–7.34 (m, 6H), 7.19–7.09 (m, 2H), 7.02–6.87 (m, 2H), 6.26 (dd, *J* = 8.7, 1.1 Hz, 1.67H), 6.20 (dd, *J* = 8.8, 1.1 Hz, 0.33H), 2.57 (s, 0.5H), 1.97 (s, 2.5H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.12, 178.05, 172.05, 142.20, 141.57, 139.96, 137.83, 137.79, 137.61, 134.67, 134.35, 134.32, 133.12, 132.85, 132.07, 132.03, 131.50, 131.45, 130.65, 130.17, 129.94, 129.91, 129.76, 128.85, 127.41, 126.93, 126.77, 122.53, 122.47, 116.24, 107.83, 104.82, 103.79, 17.51, 17.30; HRMS (ESI-TOF) Calcd for $C_{29}H_{20}NO_3S$ [M + H]$^+$: 462.1158; found: 462.1177.



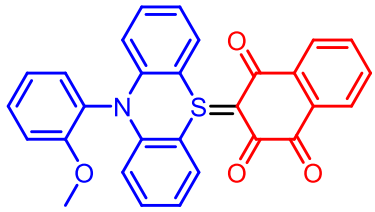
19

3-(10-(2-chlorophenyl)-10*H*-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3*H*)-trione (19): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 210.7 mg, 73%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.37 (dd, *J* = 7.8, 1.7 Hz, 1H), 8.22 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.06 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.75–7.64 (m, 3H), 7.63–7.55 (m, 4H), 7.31–7.26 (m, 2H), 7.09–7.02 (m, 2H), 6.35 (dd, *J* = 8.6, 1.1 Hz, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.10, 178.12, 172.03, 142.10, 136.60, 135.28, 134.63, 134.39, 133.61, 133.10, 132.08, 132.04, 131.24, 130.77, 130.63, 127.42, 126.95, 122.94, 116.39, 107.48, 104.20; HRMS (ESI-TOF) Calcd for $C_{28}H_{17}ClNO_3S$ [M + H]$^+$: 482.0612; found: 486.0612.



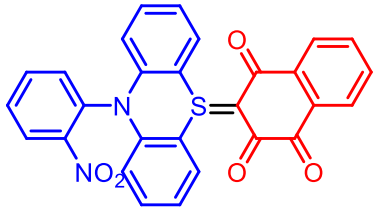
20

3-(10-(2-bromophenyl)-10*H*-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3*H*)-trione (20): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 223.7 mg, 71%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.38 (dd, *J* = 7.9, 1.7 Hz, 1H), 8.22 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.06 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.89 (dd, *J* = 8.1, 1.4 Hz, 1H), 7.72 (tt, *J* = 7.6, 1.7 Hz, 2H), 7.63–7.56 (m, 3H), 7.51 (td, *J* = 7.7, 1.5 Hz, 1H), 7.31–7.26 (m, 2H), 7.09–7.00 (m, 2H), 6.38–6.30 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.11, 178.13, 172.02, 141.88, 138.24, 134.62, 134.40, 134.05, 133.79, 133.06, 133.01, 132.08, 131.35, 130.63, 127.43, 126.96, 125.64, 122.94, 116.53, 107.55, 104.13; HRMS (ESI-TOF) Calcd for $C_{28}H_{17}BrNO_3S$ [M + H]$^+$: 526.0107; found: 526.0109.



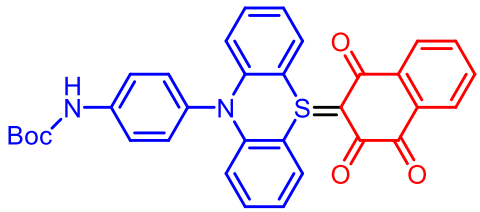
21

3-(10-(2-methoxyphenyl)-10H-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione (21). Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 206.1 mg, 72%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.21 (d, *J* = 7.7 Hz, 1H), 8.15 (d, *J* = 7.7 Hz, 1H), 8.05 (d, *J* = 7.6 Hz, 1H), 7.69 (t, *J* = 7.5 Hz, 1H), 7.62–7.53 (m, 4H), 7.32–7.17 (m, 4H), 6.99 (t, *J* = 7.5 Hz, 2H), 6.41 (d, *J* = 8.6 Hz, 2H), 3.77 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.15, 178.06, 172.03, 156.76, 142.99, 134.71, 134.33, 132.86, 132.73, 131.99, 131.27, 130.38, 127.35, 127.19, 126.90, 123.38, 122.40, 116.77, 112.09, 107.72, 104.03, 55.75; HRMS (ESI-TOF) Calcd for $C_{29}H_{20}NO_4S$ [M + H]$^+$: 478.1108; found: 478.1109.



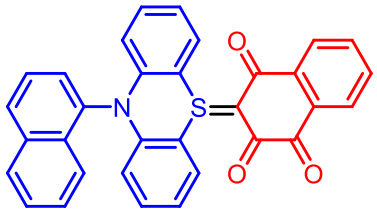
22

3-(10-(2-nitrophenyl)-10H-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione (22): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 239.2 mg, 81%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.56 (d, *J* = 7.9 Hz, 1H), 8.31 (dd, *J* = 8.1, 1.5 Hz, 1H), 8.23 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.09–8.02 (m, 2H), 7.85 (td, *J* = 8.1, 1.6 Hz, 1H), 7.72 (td, *J* = 7.6, 1.4 Hz, 1H), 7.62–7.56 (m, 3H), 7.29–7.24 (m, 2H), 7.10–7.03 (m, 2H), 6.31 (dd, *J* = 8.6, 1.1 Hz, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.12, 178.14, 171.99, 148.53, 142.26, 137.36, 134.88, 134.53, 134.49, 133.13, 132.65, 132.16, 132.02, 131.27, 130.75, 127.43, 127.00, 125.89, 123.25, 116.28, 107.40, 104.55; HRMS (ESI-TOF) Calcd for $C_{28}H_{17}N_2O_5S$ [M + H]$^+$: 493.0853; found: 493.0852.



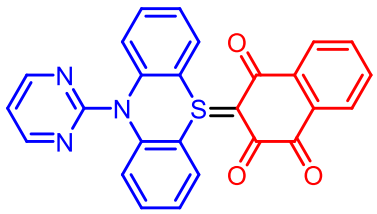
23

tert-butyl (4-(5-(1,3,4-trioxo-3,4-dihydronaphthalen-2(1H)-ylidene)-5λ^4-phenothiazin-10(5H)-yl)phenyl)carbamate (23): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 286.7 mg, 85%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.21 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.05 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.77 (d, *J* = 8.4 Hz, 2H), 7.73–7.52 (m, 6H), 7.24–7.15 (m, 3H), 7.03–6.96 (m, 2H), 6.51 (dd, *J* = 8.7, 1.1 Hz, 2H), 1.50 (s, 9H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.10, 178.11, 172.08, 152.79, 143.50, 139.82, 134.63, 134.38, 133.26, 132.78, 132.05, 132.03, 131.52, 130.39, 127.36, 126.94, 122.49, 120.86, 117.07, 107.77, 103.73, 80.90, 28.32; HRMS (ESI-TOF) Calcd for $C_{33}H_{27}N_2O_5S$ [M + H]$^+$: 563.1635; found: 563.1637.



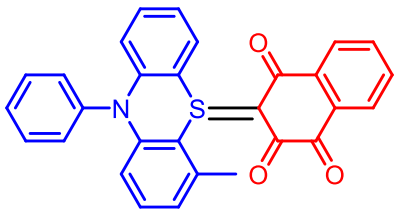
24

3-(10-(naphthalen-1-yl)-10H-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione (**24**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 238.6 mg, 80%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.20 (d, *J* = 8.4 Hz, 0.25H), 8.52 (dd, *J* = 7.3, 1.2 Hz, 0.75H), 8.27–8.19 (m, 1H), 8.15–8.03 (m, 3H), 7.87–7.78 (m, 1H), 7.77–7.48 (m, 6H), 7.48–7.36 (m, 1H), 7.14–7.05 (m, 2H), 7.03–6.92 (m, 2H), 6.26–6.15 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.13, 178.15, 172.13, 143.27, 142.32, 136.03, 135.88, 135.41, 134.82, 134.69, 134.38, 134.34, 132.95, 132.79, 132.15, 132.07, 131.54, 131.00, 130.47, 130.20, 130.05, 130.01, 129.83, 129.17, 128.85, 128.37, 128.01, 127.95, 127.90, 127.80, 127.48, 127.44, 127.03, 126.97, 126.78, 126.59, 124.08, 122.61, 122.55, 122.48, 117.35, 116.95, 107.69, 104.88, 103.96; HRMS (ESI-TOF) Calcd for $C_{32}H_{20}NO_3S$ [M + H]$^+$: 498.1158; found: 498.1162.



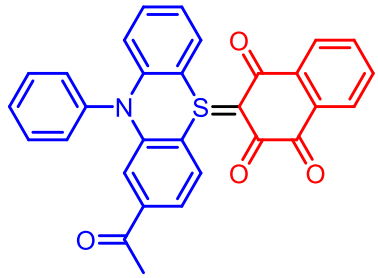
25

3-(10-(pyrimidin-2-yl)-10H-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione (**25**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 229.0 mg, 85%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.50 (d, *J* = 4.8 Hz, 2H), 8.25 (dd, *J* = 7.6, 1.5 Hz, 1H), 8.16 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.99 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.79–7.69 (m, 2H), 7.62–7.56 (m, 2H), 7.42 (dd, *J* = 8.1, 1.5 Hz, 2H), 7.36–7.30 (m, 2H), 6.98 (t, *J* = 4.8 Hz, 1H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 181.46, 178.25, 173.78, 159.43, 158.15, 137.49, 135.00, 134.58, 132.67, 132.20, 130.66, 128.84, 127.98, 127.05, 126.86, 125.23, 123.37, 115.80; HRMS (ESI-TOF) Calcd for $C_{26}H_{16}N_3O_3S$ [M + H]$^+$: 450.0907; found: 450.0907.



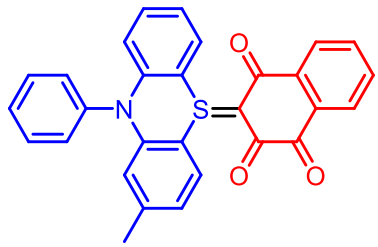
26

(Z)-3-(4-methyl-10-phenyl-10H-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione (**26**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 215.8 mg, 78%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.20 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.04 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.90–7.51 (m, 8H), 7.22–7.16 (m, 1H), 7.13–7.05 (m, 1H), 7.01–6.93 (m, 1H), 6.85 (d, *J* = 7.3 Hz, 1H), 6.41 (dd, *J* = 8.7, 1.1 Hz, 1H), 6.32 (d, *J* = 8.6 Hz, 1H), 2.60 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.00, 177.67, 172.15, 144.24, 143.07, 139.65, 139.45, 134.67, 134.29, 132.72, 132.02, 131.95, 131.92, 131.35, 131.16, 130.51, 129.50, 127.32, 126.95, 124.07, 122.33, 116.78, 115.07, 105.87, 103.65, 102.49, 20.05; HRMS (ESI-TOF) Calcd for $C_{29}H_{20}NO_3S$ [M + H]$^+$: 462.1158; found: 462.1163.



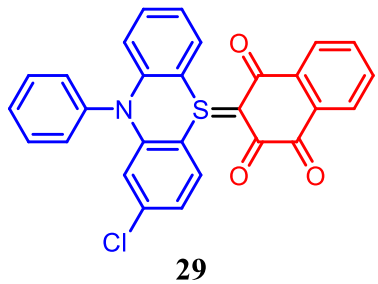
27

(*Z*)-3-(2-acetyl-10-phenyl-10*H*-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3*H*)-trione (27): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 231.8 mg, 79%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.21 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.06 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.80–7.74 (m, 4H), 7.71 (td, *J* = 7.6, 1.4 Hz, 1H), 7.69–7.64 (m, 2H), 7.62–7.56 (m, 2H), 7.51 (dd, *J* = 8.2, 1.7 Hz, 1H), 7.28–7.23 (m, 1H), 7.08–7.03 (m, 1H), 7.02 (d, *J* = 1.6 Hz, 1H), 6.49 (dd, *J* = 8.7, 1.1 Hz, 1H), 2.37 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 196.53, 182.82, 178.07, 172.17, 143.53, 143.01, 140.32, 138.70, 134.51, 134.46, 133.03, 132.22, 132.02, 131.68, 130.84, 130.48, 130.05, 127.52, 126.96, 123.08, 121.40, 117.30, 116.40, 111.52, 107.88, 107.08, 103.79, 26.55; HRMS (ESI-TOF) Calcd for $C_{30}H_{20}NO_4S$ [M + H]$^+$: 490.1108; found: 490.1115.



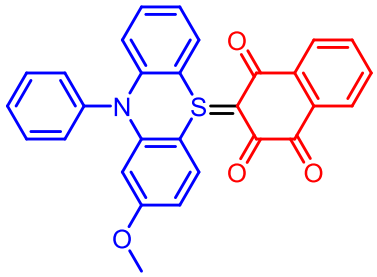
28

(*Z*)-3-(2-methyl-10-phenyl-10*H*-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3*H*)-trione (28):. Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 226.9 mg, 82%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.20 (dd, *J* = 7.7, 1.3 Hz, 1H), 8.04 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.79–7.66 (m, 5H), 7.65–7.60 (m, 1H), 7.59–7.53 (m, 2H), 7.44 (d, *J* = 8.0 Hz, 1H), 7.24–7.16 (m, 1H), 7.02–6.95 (m, 1H), 6.83 (dd, *J* = 8.1, 1.6 Hz, 1H), 6.43 (dd, *J* = 8.7, 1.1 Hz, 1H), 6.24 (d, *J* = 1.6 Hz, 1H), 2.15 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.14, 178.03, 172.02, 143.74, 143.36, 143.22, 139.25, 134.69, 134.29, 132.63, 132.04, 131.96, 131.37, 131.13, 130.46, 130.26, 129.58, 127.33, 126.89, 123.77, 122.35, 117.26, 116.99, 107.96, 103.96, 100.63, 21.97; HRMS (ESI-TOF) Calcd for $C_{29}H_{20}NO_3S$ [M + H]$^+$: 462.1158; found: 462.1171.



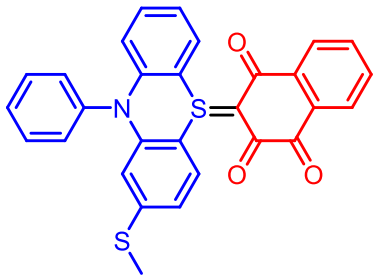
29

(*Z*)-3-(2-chloro-10-phenyl-10*H*-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3*H*)-trione (29):. Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 230.9 mg, 80%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.21 (dd, *J* = 7.9, 1.3 Hz, 1H), 8.07 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.76 (d, *J* = 4.7 Hz, 4H), 7.72 (td, *J* = 7.8, 1.5 Hz, 1H), 7.68–7.63 (m, 1H), 7.62–7.54 (m, 2H), 7.50 (d, *J* = 8.4 Hz, 1H), 7.26–7.22 (m, 1H), 7.07–7.02 (m, 1H), 6.98 (dd, *J* = 8.4, 2.0 Hz, 1H), 6.47–6.42 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 182.90, 178.10, 172.14, 144.17, 142.81, 139.17, 138.63, 134.55, 134.42, 132.94, 132.18, 132.05, 131.66, 131.51, 130.83, 130.49, 130.03, 127.50, 126.93, 123.12, 122.76, 117.27, 116.75, 107.35, 104.09, 102.14; HRMS (ESI-TOF) Calcd for $C_{28}H_{17}ClNO_3S$ [M + H]$^+$: 482.0612; found: 482.0615.



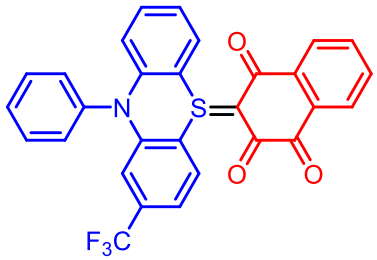
30

(*Z*)-3-(2-methoxy-10-phenyl-10*H*-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3*H*)-trione (**30**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 246.2 mg, 86%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.21 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.05 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.79–7.67 (m, 5H), 7.64–7.54 (m, 3H), 7.49 (d, *J* = 8.8 Hz, 1H), 7.24–7.18 (m, 1H), 7.03–6.97 (m, 1H), 6.59 (dd, *J* = 8.8, 2.5 Hz, 1H), 6.45 (dd, *J* = 8.7, 1.1 Hz, 1H), 5.94 (d, *J* = 2.5 Hz, 1H), 3.60 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.21, 178.06, 171.98, 162.98, 144.92, 143.13, 139.21, 134.68, 134.30, 132.62, 132.03, 131.97, 131.94, 131.38, 131.03, 130.48, 129.66, 127.34, 126.88, 122.46, 117.01, 109.04, 108.27, 104.34, 102.56, 95.22, 55.33; HRMS (ESI-TOF) Calcd for $C_{29}H_{20}NO_4S$ [M + H]$^+$: 478.1108; found: 478.1111.



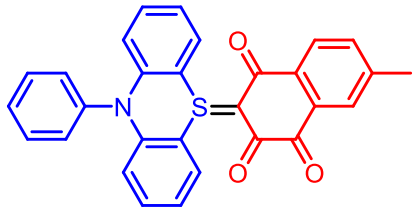
31

(*Z*)-3-(2-(methylthio)-10-phenyl-10*H*-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3*H*)-trione (**31**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 257.4 mg, 87%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.21 (d, *J* = 7.6 Hz, 1H), 8.06 (d, *J* = 7.6 Hz, 1H), 7.79–7.68 (m, 5H), 7.66–7.61 (m, 1H), 7.60–7.54 (m, 2H), 7.44 (d, *J* = 8.3 Hz, 1H), 7.26–7.20 (m, 1H), 7.01 (t, *J* = 7.5 Hz, 1H), 6.84 (dd, *J* = 8.5, 1.9 Hz, 1H), 6.46 (d, *J* = 8.6 Hz, 1H), 6.22 (d, *J* = 1.8 Hz, 1H), 2.21 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.11, 178.08, 172.05, 145.80, 143.42, 143.16, 138.99, 134.66, 134.34, 132.72, 132.03, 131.42, 131.05, 130.48, 130.41, 129.74, 127.40, 126.90, 122.62, 119.60, 117.11, 112.93, 107.87, 104.20, 99.30, 14.64; HRMS (ESI-TOF) Calcd for $C_{29}H_{20}NO_3S_2$ [M + H]$^+$: 494.0879; found: 494.0881.



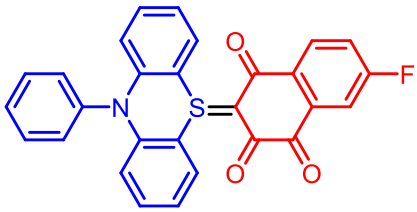
32

(*Z*)-3-(10-phenyl-2-(trifluoromethyl)-10*H*-5λ^4-phenothiazin-5-ylidene)naphthalene-1,2,4(3*H*)-trione (**32**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 253.4 mg, 82%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.22 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.08 (dd, *J* = 7.6, 1.3 Hz, 1H), 7.79–7.73 (m, 4H), 7.73–7.65 (m, 3H), 7.63–7.56 (m, 2H), 7.30–7.20 (m, 2H), 7.10–7.04 (m, 1H), 6.68 (d, *J* = 1.8 Hz, 1H), 6.49 (d, *J* = 8.6 Hz, 1H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 182.78, 178.10, 172.19, 143.58, 142.86, 138.40, 135.08, 134.75, 134.47 (d, *J* = 2.9 Hz), 134.09, 133.12, 132.78, 132.27, 132.04, 131.77, 131.59, 131.32, 130.74, 130.48, 130.21, 130.15, 127.57, 126.98, 126.92, 124.21, 123.40, 121.49, 118.57 (q, *J* = 3.4 Hz), 117.38, 113.63 (q, *J* = 4.2 Hz), 107.22, 106.89, 103.89; ^{19}F NMR (376 MHz, Chloroform-*d*) δ −63.70; HRMS (ESI-TOF) Calcd for $C_{29}H_{17}F_3NO_3S$ [M + H]$^+$: 516.0876; found: 516.0884.



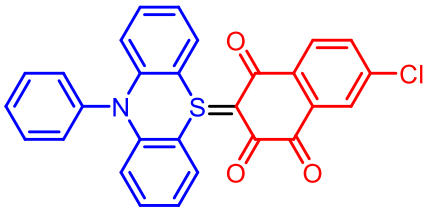
33

7-methyl-3-(10-phenyl-10H-5λ^4*-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione* (**33**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 210.3 mg, 76%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.01 (d, *J* = 7.9 Hz, 1H), 7.77 (d, *J* = 1.7 Hz, 1H), 7.71–7.60 (m, 4H), 7.53 (tt, *J* = 7.1, 1.6 Hz, 1H), 7.47 (dd, *J* = 8.0, 1.6 Hz, 2H), 7.41 (dd, *J* = 8.0, 1.8 Hz, 1H), 7.16–7.09 (m, 2H), 6.95–6.88 (m, 2H), 6.37 (dd, *J* = 8.7, 1.1 Hz, 2H), 2.33 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.37, 178.21, 172.18, 143.29, 142.78, 139.20, 135.13, 132.68, 132.28, 131.91, 131.41, 131.12, 130.45, 129.61, 127.73, 127.03, 122.48, 116.97, 107.26, 103.93, 21.44; HRMS (ESI-TOF) Calcd for $C_{29}H_{20}NO_3S$ [M + H]$^+$: 462.1158; found: 462.1204.



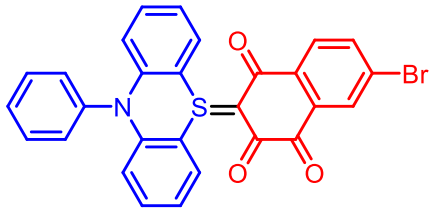
34

7-fluoro-3-(10-phenyl-10H-5λ^4*-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione* (**34**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 198.1 mg, 71%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.23 (dd, *J* = 8.6, 5.3 Hz, 1H), 7.78–7.68 (m, 5H), 7.66–7.60 (m, 1H), 7.56 (dd, *J* = 7.9, 1.5 Hz, 2H), 7.37 (td, *J* = 8.2, 2.7 Hz, 1H), 7.26–7.19 (m, 2H), 7.01 (t, *J* = 7.5 Hz, 2H), 6.47 (d, *J* = 8.6 Hz, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 182.19, 177.05, 171.69, 166.26, 163.73, 143.33, 139.13, 134.24 (d, *J* = 7.2 Hz), 132.83, 131.45, 131.04, 130.99, 130.45, 129.83 (d, *J* = 8.3 Hz), 129.67, 122.57, 121.51, 121.29, 117.02, 113.81, 113.58, 107.51, 103.57; ^{19}F NMR (376 MHz, Chloroform-*d*) δ −105.90; HRMS (ESI-TOF) Calcd for $C_{28}H_{17}FNO_3S$ [M + H]$^+$: 466.0908; found: 466.0908.



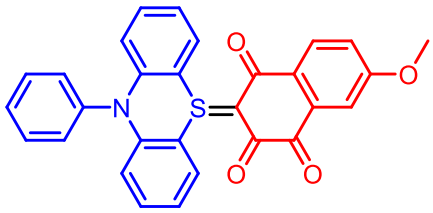
35

7-chloro-3-(10-phenyl-10H-5λ^4*-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione* (**35**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 219.4 mg, 76%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.16 (d, *J* = 8.1 Hz, 1H), 8.00 (d, *J* = 2.2 Hz, 1H), 7.78–7.70 (m, 4H), 7.66–7.60 (m, 2H), 7.56 (dd, *J* = 7.9, 1.6 Hz, 2H), 7.26–7.21 (m, 2H), 7.04–6.99 (m, 2H), 6.47 (dd, *J* = 8.7, 1.2 Hz, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 182.16, 177.03, 171.52, 143.33, 139.11, 138.75, 134.25, 133.06, 132.89, 132.87, 131.46, 131.04, 130.46, 129.68, 128.67, 127.09, 122.59, 117.04, 107.80, 103.48; HRMS (ESI-TOF) Calcd for $C_{28}H_{17}ClNO_3S$ [M + H]$^+$: 482.0612; found: 482.0617.



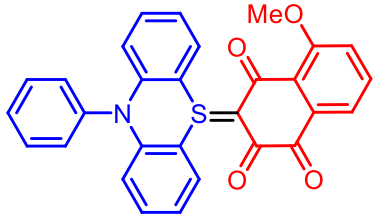
36

7-bromo-3-(10-phenyl-10H-5λ⁴-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione (**36**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 242.6 mg, 77%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.08 (d, J = 2.0 Hz, 1H), 7.99 (d, J = 8.3 Hz, 1H), 7.72 (dd, J = 8.3, 2.1 Hz, 1H), 7.70–7.60 (m, 4H), 7.55 (tt, J = 6.3, 2.1 Hz, 1H), 7.47 (dd, J = 7.9, 1.6 Hz, 2H), 7.18–7.12 (m, 2H), 6.98–6.89 (m, 2H), 6.38 (dd, J = 8.7, 1.1 Hz, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 182.11, 177.13, 171.42, 143.32, 139.09, 137.23, 133.27, 133.04, 132.88, 131.47, 131.04, 130.46, 130.09, 129.69, 128.75, 127.13, 122.60, 117.04, 107.90, 103.43; HRMS (ESI-TOF) Calcd for $C_{28}H_{17}BrNO_3S$ $[M + H]^+$: 526.0107; found: 526.0159.



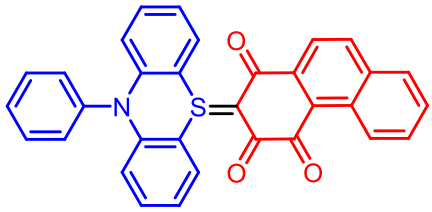
37

7-Methoxy-3-(10-phenyl-10H-5λ⁴-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione (**37**). Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 240.5 mg, 84%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.13 (d, *J* = 8.6 Hz, 1H), 7.79–7.68 (m, 4H), 7.65–7.59 (m, 1H), 7.56 (dd, *J* = 7.9, 1.6 Hz, 2H), 7.50 (d, *J* = 2.7 Hz, 1H), 7.24–7.17 (m, 3H), 7.03–6.98 (m, 2H), 6.45 (dd, *J* = 8.8, 1.1 Hz, 2H), 3.88 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 183.17, 178.02, 172.14, 162.71, 143.29, 139.21, 132.71, 132.67, 131.42, 131.11, 130.47, 129.60, 128.95, 127.91, 122.48, 121.42, 116.95, 110.13, 106.75, 104.00, 55.87; HRMS (ESI-TOF) Calcd for $C_{29}H_{20}NO_4S$ $[M + H]^+$: 478.1108; found: 478.1115.



38

5-Methoxy-3-(10-phenyl-10H-5λ⁴-phenothiazin-5-ylidene)naphthalene-1,2,4(3H)-trione (**38**). Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 234.6 mg, 82%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.78–7.65 (m, 5H), 7.63–7.54 (m, 3H), 7.53–7.46 (m, 1H), 7.29–7.25 (m, 1H), 7.18 (ddd, *J* = 8.8, 7.2, 1.6 Hz, 2H), 6.99–6.93 (m, 2H), 6.41 (dd, *J* = 8.6, 1.1 Hz, 2H), 4.02 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 179.13, 174.57, 165.84, 155.02, 138.61, 134.47, 129.63, 128.13, 127.81, 126.58, 126.44, 125.73, 124.77, 117.63, 116.59, 115.82, 114.12, 112.17, 104.88, 99.12, 51.91; HRMS (ESI-TOF) Calcd for $C_{29}H_{20}NO_4S$ $[M + H]^+$: 478.1108; found: 478.1107.



39

2-(10-phenyl-10H-5λ^4*-phenothiazin-5-ylidene)phenanthrene-1,3,4(2H)-trione* (**39**): Yellow solid; Eluent: petroleum ether/dichloromethane/ethyl acetate 30:20:5; 217.7 mg, 73%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.37 (dd, *J* = 8.8, 1.2 Hz, 1H), 8.29 (d, *J* = 8.6 Hz, 1H), 8.06 (d, *J* = 8.6 Hz, 1H), 7.77 (dd, *J* = 8.2, 1.4 Hz, 1H), 7.72 (d, *J* = 7.2 Hz, 2H), 7.69–7.64 (m, 2H), 7.61–7.55 (m, 2H), 7.54–7.46 (m, 3H), 7.18–7.12 (m, 2H), 6.94 (td, *J* = 7.6, 1.2 Hz, 2H), 6.40 (dd, *J* = 8.7, 1.1 Hz, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 185.55, 178.24, 172.62, 143.31, 139.21, 135.81, 135.48, 135.37, 132.75, 131.44, 131.13, 130.53, 130.45, 129.89, 129.64, 128.58, 127.97, 127.81, 127.72, 122.99, 122.57, 117.04, 104.68, 103.89; HRMS (ESI-TOF) Calcd for $C_{32}H_{20}NO_3S$ $[M + H]^+$: 498.1158; found: 498.1184.