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电化学氧化三氟甲基亚磺酸钠与 α-羰基二硫缩烯酮的三氟甲基化反应

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摘要 报道了一种无过渡金属催化剂、外加化学氧化剂的条件下, 电化学氧化三氟甲基亚磺酸钠与 α-羰基二硫缩烯酮的高效三氟甲基化反应. 该方法具有反应条件温和与较好的官能团兼容性的优点. 相关的机理实验表明该反应是通过自由基机理进行的.

关键词 电化学; 三氟甲基化; α-羰基二硫缩烯酮; 自由基; 绿色化学

Electrochemical Oxidative Trifluoromethylation of α-Oxoketene Ketene Dithioacetals with CF_3SO_2Na

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Abstract An efficient electrochemical oxidative trifluoromethylation of α-oxoketene dithioacetals with CF_3SO_2Na is developed without the usage of transition-metal catalysts and external oxidants. This protocol features mild reaction conditions and wide functionality tolerance. Mechanistic insights indicate that the reaction proceeded via radical mechanism.

Keywords electrochemical; trifluoromethylation; α-oxoketene dithioacetals; radical; green chemistry

1 Introduction

The oxidative functionalization of internal alkenes has been acknowledged as one of the most efficient approaches to introduce functional groups in molecular structures.[1] Therefore, great advances have been made in this research field of organic chemistry in the past decade.[2] Among these, the functionalization of α-oxoketene dithioacetals via different approaches has been extensively studied due to its wide application as versatile intermediates in the construction of complex structures.[3] Meanwhile, the incorporation of trifluoromethyl group into alkenes is of great interest due to its electro-withdrawing nature and significant effect on properties like lipophilicity, permeability and metabolic stability, which have been proven relevant in medicinal and agrochemistry.[4-5] As a result, several useful and practical strategies have been developed to enable direct trifluoromethylation of α-oxoketene dithioacetals. In 2013, Wang and Liu[6] innovatively reported a metal-free oxidative α-trifluoromethylation of α-oxoketene dithioacetals via *in-situ* generated hypervalent iodide trifluoromethylating species $[PhICF_3]^+$ derived from PhI-$(OAc)_2$, $TMSCF_3$ and KF, mechanistic details suggested that the reaction proceeded through electrophilic pathway rather than single-electron-transfer mechanism (Scheme 1a). Later, Yu and coworkers[7] developed a Cu^{II}-catalyzed trifluoromethylation of α-oxoketene dithioacetals with stoichiometric amount of Ag salts as oxidant and $TMSCF_3$ as trifluoromethylating reagent, to afford diverse trifluoromethylated α-oxoketene dithioacetals in good to excellent yields (Scheme 1b). Recently, photoredox-mediated radical functionalization reaction has emerged as an ideal strategy for transformations and has drawn considerable attention. In this regard, Xia and coworkers[8] revealed a visible-light irradiated direct trifluoromethylation of α-oxoketene dithioacetals using $Ru(bpy)_3Cl_2$ as photocatalyst and Togni's reagent as trifluoromethylating agent (Scheme 1c). Although the success of $C(sp^2)$—CF_3 bond formation in the presence of metal catalyst and/or stoichiometric amount of strong oxidants definitely represent attractive strategies to trifluoromethylated α-oxoketene dithioacetals, further exploration of an alternative strategy which allows for the incorporation of trifluoromethyl group into the core struc-

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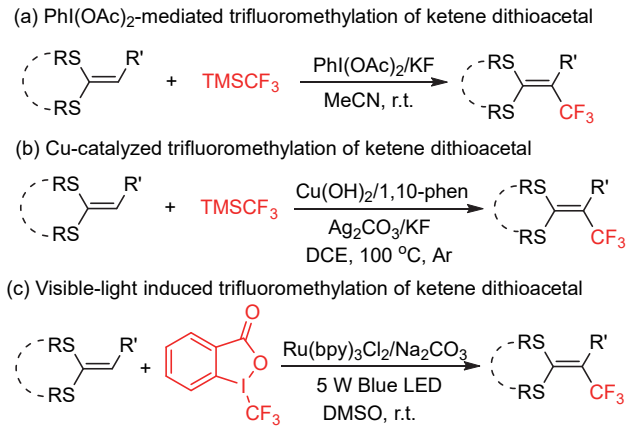
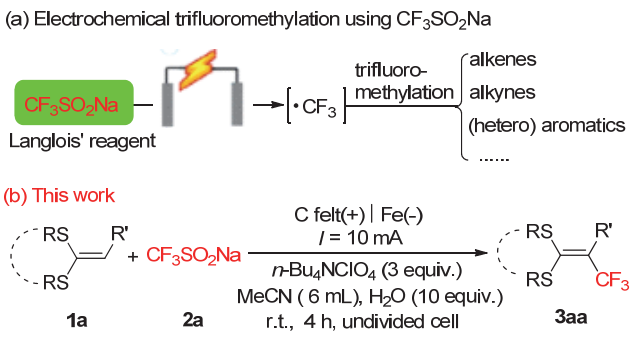
ture with cheap and bench-stable CF_3SO_2Na (Langlois' reagent) under benign conditions is still highly desirable.



Scheme 1 Trifluoromethylation of α-ketene dithioacetal

On the other hand, electrochemistry, which is featured as greenness and sustainability, has evolved to be a powerful strategy for the assembly of functional molecules by utilization of electric current for radical generation, while obviating the need for stoichiometric oxidizing or reducing reagents.[9] In this context, the application of electrochemical anodic oxidation in the construction of C(sp^2)—CF_3 bond has received increasing attention among synthetic community (Scheme 2a).[10-11] For example, Lei and coworkers[12] have documented an electrochemical oxidative trifluoromethylation of allyl alcohols for the synthesis of β-trifluoromethylated ketones via 1,2-migration. Wang's group[13] disclosed an elegant electrochemically-enabled fluoroalkylation/migration reaction of unactivated olefins, affording fluorinated aryl ketones with good yields. Ackermann and coworkers[14] reported an electro-oxidative dearomatization of biaryls leading to tri- and difluoromethylated spiro[5.5]trienones. Xu and coworkers [15] innovatively developed the first example of electrochemical difluoromethylation and trifluoromethylation triggered lactonization of alkenes, affording a wide array of CF_2H and CF_3-containing lactones. Inspired by the recent developments in trifluoromethylation reactions and our ongoing interests in electrochemistry and fluorine chemistry,[16] we report herein an electrochemical oxidative trifluoromethylation of α-oxoketene dithioacetals with CF_3SO_2Na as inexpensive and stable trifluoromethyl source. This protocol features mild reaction conditions without an oxidative reagent or metal catalyst and broad functionality tolerance.

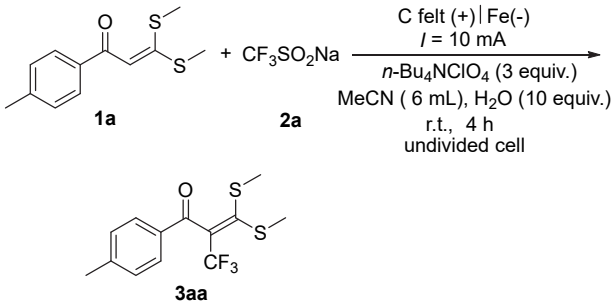


Scheme 2 Electrochemical trifluoromethylation reaction

2 Results and discussion

A brief survey of α-oxoketene dithioacetal (**1a**), CF_3SO_2Na (**2a**) as the mode reaction in different solvents and electrolytes under a constant current of 10 mA was conducted (Table 1). Performing the reaction in an undivided cell with a carbon felt as the anode, an iron plate (Fe) as the cathode and *n*-Bu_4NClO_4 (3 equiv.) as the electrolyte with an additional of H_2O (10 equiv.) in MeCN at ambient temperature for 4 h gave the desired trifluoromethylated α-oxoketene dithioacetal (**3aa**) in 77% isolated yield (Entry 1). Initially, the effects of electrode materials on the reaction were evaluated. The replacement of carbon felt anode/iron plate cathode with other electrode like carbon felt or nickel plate led to decreased yields (Entries 2～3). Other electrolytes were also suitable for the reaction to

Table 1 Optimization of reaction conditions[a]



Entry	Variation from the standard conditions	Yield[b]/%
1	None	77
2	C felt (+) | C felt(−) Instead of C felt (+) | Fe (−)	Trace
3	C felt (+) | Ni felt(−) Instead of C felt (+) | Fe (−)	62
4	*n*-Bu_4NPF_6 instead of *n*-Bu_4NClO_4	74
5	*n*-Bu_4NBF_4 instead of *n*-Bu_4NClO_4	71
6	Without electrolyte	61
7	DCE instead of MeCN	Trace
8	DMF instead of MeCN	Trace
9	AcOH instead of MeCN	52
10	Without H_2O	60
11	H_2O (20 equiv.)	75
12	50 ℃ instead of r.t.	45
13	**2a** (2 equiv.)	35
14	5 mA instead of 10 mA	57
15	$TMSCF_3$ instead of CF_3SO_2Na	Trace
16	No electric current	N.R.
17	*n*-Bu_4NClO_4 (2 equiv.)	73

[a] Standard conditions: **1a** (0.4 mmol), **2a** (3.0 equiv.), H_2O (10.0 equiv.), *n*-Bu_4NClO_4 (3.0 equiv.), MeCN (6 mL), carbon felt anode, iron plate cathode, I=10 mA, undivided cell, 25 ℃, 4 h. [b] Isolated yields. [c] Reaction time: 6 h.

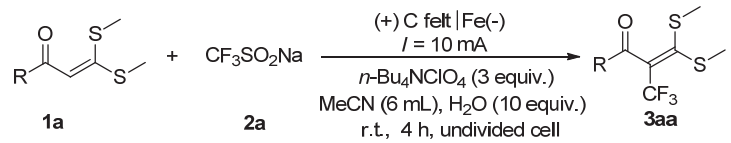
deliver **3aa** with albeit lower yields (Entries 4～5). Interestingly, **3aa** could also be obtained in 61% yield in the absence of electrolytes (Entry 6). Subsequently, various solvents were examined and MeCN was shown to be a better solvent (Entries 7～9). Finally, control experiments revealed that H_2O was crucial for this transformation, probably due to the increased conductivity of the mixture with addition of H_2O, while similar yields was obtained with increased amount of H_2O (Entries 10～11). The yield decreased significantly by elevating the temperature to 50 ℃ or reducing the amount of **2a** (Entries 12～13). Reducing the current to 5 mA resulted in an inferior yield with 6 h (Entry 14). Furthermore, the replacement of trifluoromethyl source from CF_3SO_2Na to $TMSCF_3$ completely shut down the reaction. As anticipated, no desired product was detected without electric current under otherwise identical conditions even with prolonged reaction time (Entry 16). Reducing the loading of electrolyte *n*-Bu_4NClO_4 to 2 equiv. led to a decrease to 73% yield after extending the reaction time to 6 h (Entry 17).

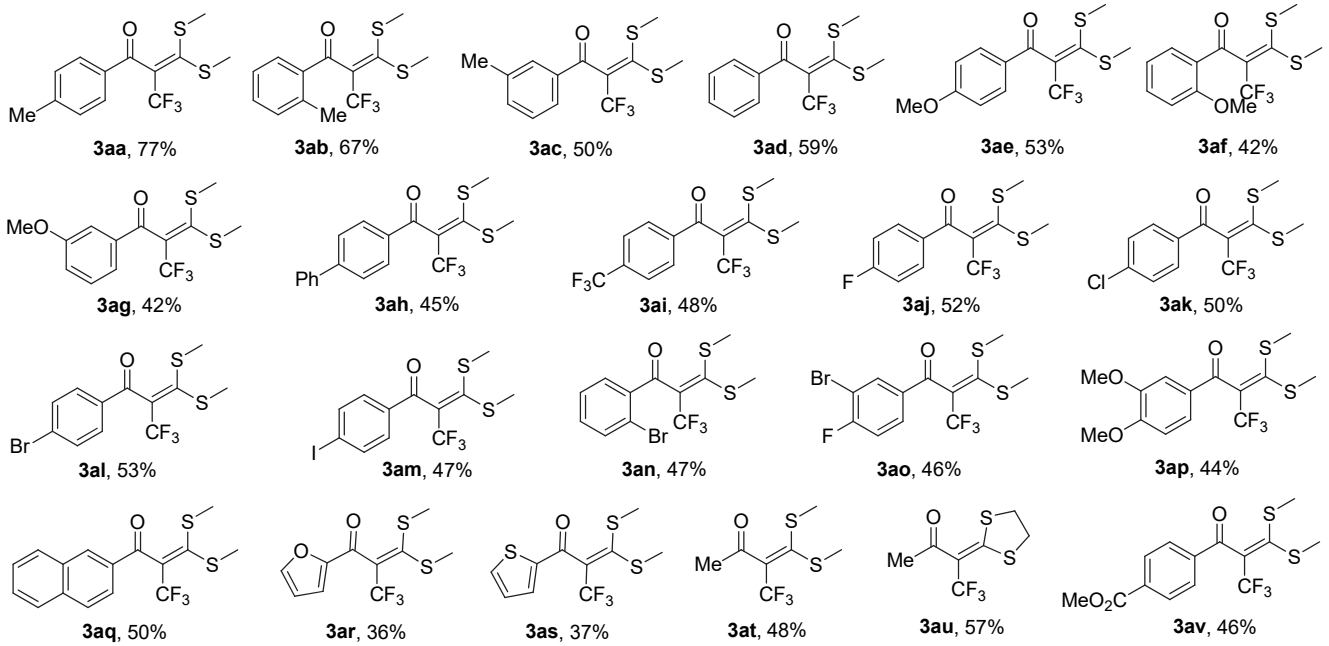
Having established the optimal reaction conditions, the scope of *α*-oxoketene dithioacetals was examined. As displayed in Table 2, *α*-oxoketene dithioacetals with methyl groups at the *para*-, *ortho*- and *meta*-position of the aromatic ring were compatible in the transformation to afford **3aa**～**3ac** with comparable yields, to indicate that the position of the substituents on the phenyl ring had a minor effect on the reaction efficiency. Additionallly, *α*-oxoketene dithioacetals with both electron-donating groups (Me, OMe, Ph) and electron-withdrawing groups (CF_3) could endow the *α*-trifluoromethylated products with moderate to good yields (**3ae**～**3ai**). Notably, the introductions of halide groups at the *para*- or *ortho*-position of the phenyl ring were also suitable substrates with this protocol to access **3aj**～**3an** in moderate yields, which were amenable for further elaboration.

In addition, di-substituted *α*-oxoketene dithioacetals could also react under the standard reaction conditions to give **3ao** and **3ap** in 46% and 44% yields. It is worth mentioning that *α*-oxoketene dithioacetals bearing naphthyl, thienyl and furyl groups were all converted to the desired products in relatively lower yields (**3aq**～**3as**). Subsequently, by changing the aromatic ring connecting to carbonyl group to alkyl group, the corresponding product **3at** was obtained in 48% yield. To our delight, the cyclic dimethylthio analogue of **1u** was also viable for the reaction to furnish **3au** in 57% yield. Furthermore, **1av** with ester as reactive group was also tested to give **3av** in 46% yield. Notably, the starting materials in the reaction were completely consumed, and the relatively lower efficiency of this protocol may be attributed to the generation of by-products which remained to be characterized.

To demonstrate the potential practicability of this protocol, gram-scale reaction was conducted between **1ad** and

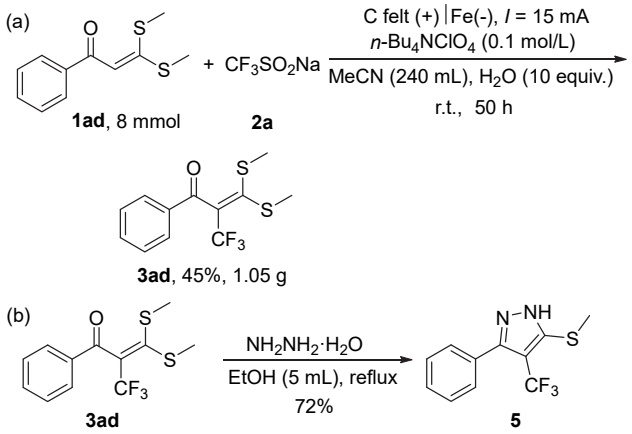
Table 2 Substrate scope[a,b]





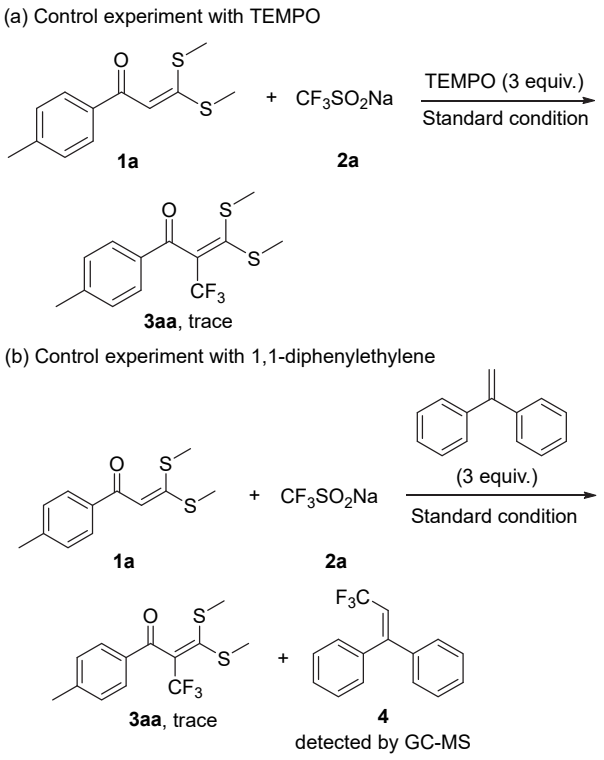
[a]Conditions: **1a**～**1u** (0.4 mmol), **2a** (3.0 equiv.), H_2O (10.0 equiv.), *n*-Bu_4NClO_4 (3.0 equiv.), MeCN (6 mL), carbon felt anode, iron plate cathode, I=10 mA, undivided cell, 25 ℃, 4 h. [b] Isolated yields.

CF_3SO_2Na (**2a**) to afford **3ad** in 45% yield (Scheme 3a). Furthermore, CF_3-containing 1*H*-pyrazoles (**5**) could be obtained by condensation of **3ad** with hydrazine.[17]



Scheme 3 Gram-scale and derivatization reaction

Afterwards, several control experiments were conducted to gain insight into this electrochemical trifluoromethylation reaction. As shown in Scheme 4, no desired product **3aa** was detected in the presence of radical trapping reagent 2,2,6,6-tetramethylpiperidinooxy (TEMPO). By adding 3 equiv. of 1,1-diphenylethylene into the model reaction, the CF_3-adduct was detected by GC-MS, to further indicate that the reaction may proceed through radical mechanism.



Scheme 4 Control experiment

To gain insights into this electrochemical reaction, the cyclic voltammetry experiments were carried out. As displayed in Figure 1, an oxidative peak of CF_3SO_2Na (**2a**) was observed at 1.44 V, whereas a higher potential oxidative peak of *α*-oxoketene dithioacetals (**1a**) was detected at 1.79 V, the CV measurement of the reaction mixture indicated that the two oxidation waves corresponds to the respective reactants **1a** and **2a**. These results suggested that CF_3SO_2Na (**2a**) could possibly firstly undergo anodic oxidation in this reaction.

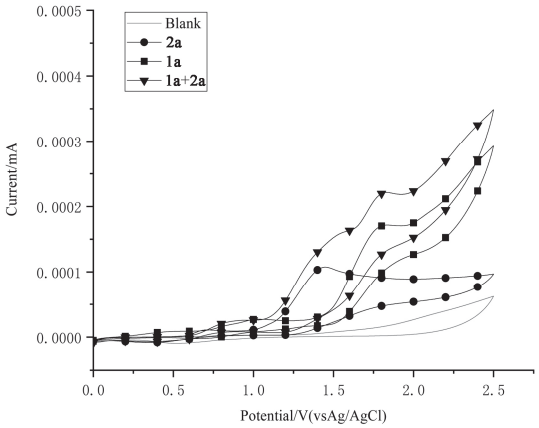
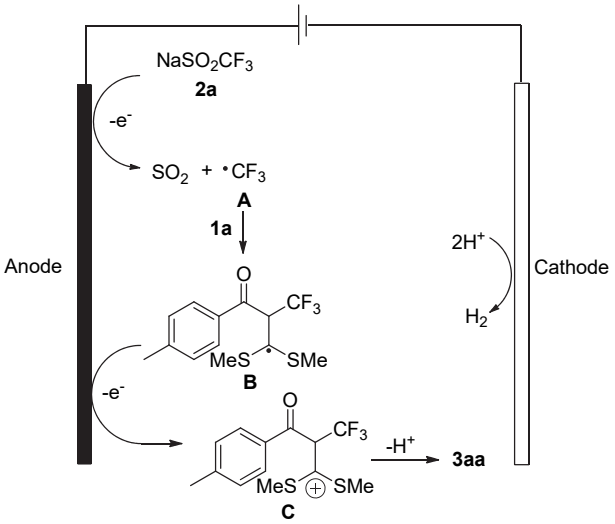


Figure 1 Cyclic voltammetry

Based on the documented previous literatures[10-14,3h] and above experimental results, a plausible mechanism is depicted in Scheme 5. Initially, anodic oxidation of CF_3SO_2Na (**2a**) would generate CF_3 radical **A**, radical addition of **A** to *α*-oxoketene dithioacetals (**1a**) affords intermediate **B**, which undergoes anodic oxidation to deliver carbocation **C**. Subsequently, deprotonation of intermediate **C** affords the desired product **3aa**. Simultaneously, the catalytic cycle was completed by hydrogen evolution reaction at the cathode.



Scheme 5 Plausible mechanism

3 Conclusions

In summary, an electrochemical trifluoromethylation of

α-oxoketene dithioacetals using CF_3SO_2Na as cheap and stable trifluoromethyl source has been developed, synthesizing various trifluoromethylated α-oxoketene dithioacetals in moderate to good yields by the usage of electric current as a green and benign oxidation tool. Further studies on the application of this strategy are currently underway in our laboratory.

4 Experimental section

4.1 General information

All ^1H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded on a Bruker Avance-III HD spectrometer in $CDCl_3$. TMS was used as an internal reference. HRMS were obtained on a Bruker micrOTOF-Q II spectrometer. PE is petroleum ether (60～90 ℃). All α-oxoketene dithioacetals are known compounds.[3] They were prepared according to the reported procedures. Unless otherwise noted, materials obtained from commercial suppliers were used without further purification.

4.2 General procedure for the synthesis of trifluoromethylated α-oxoketene dithioacetals (3)

In 10 mL flask with carbon felt anode (10 mm×10 mm×0.3 mm), iron plate cathode (10 mm×10 mm×0.3 mm), a mixture of α-oxoketene dithioacetals (**1a**～**1u**, 0.4 mmol), sodium trifluoromethanesulfinate (**2a**, 1.2 mmol, 3.0 equiv.), *n*-Bu_4NClO_4 (3.0 equiv.), and H_2O (10.0 equiv.) in CH_3CN (6 mL) was stirred with constant current of 10 mA at room temperature for 4 h [monitored by thin-layer chromatography (TLC)]. After the starting material was consumed, the mixture was poured into water (15 mL) and extracted with EtOAc (15 mL×3). The combined organic layers were washed with brine (15 mL×2) and dried over $MgSO_4$. The solvent was removed by vacuum and the residue was purified by column chromatography (PE) to give the corresponding products **3aa**～**3au**.

4.3 Cyclic voltammetry

CV measurements were performed on a CHI 610E potentiostat, and the conditions are as follows: 2 mm diameter Pt disc as working electrode, Pt wire as counter electrode, Ag/AgCl electrode submerged in saturated aqueous KCl solution as reference electrode, scan rate=0.10 V/s (0～2.5 V), and 0.3 mol/L of *n*-Bu_4NClO_4 as supporting electrolyte. The measurements were carried out as follows: 0.3 mol/L *n*-Bu_4NClO_4 as supporting electrolyte in CH_3CN was added to the measuring cell. The reactants **1a** (0.3 mol/L) and **2a** (0.3 mol/L) were added, respectively. The cyclic voltammogram was recorded with one to three scans.

4.4 Gram-scale experiment

In an oven-dried undivided beaker (500 mL) equipped with a magnetic stirring bar, compound **1ad** (8 mmol), CF_3SO_2Na (**2a**, 24 mmol) and *n*-Bu_4NClO_4 (24 mmol) were added into H_2O (10 equiv.) and CH_3CN (240 mL). The beaker was equipped with carbon felt (1.5 cm×1.5 cm×1 mm) as the anode and iron plate (1.5 cm×1.5 cm×0.3 mm) as the cathode. The electrolysis was carried out at a constant current of 15 mA for 50 h at room temperature. The reaction was monitored by TLC until complete consumption of the substrate, the reaction mixture was washed with water and extracted with EtOAc (100 mL×3). The organic layers were combined, dried over Na_2SO_4, and concentrated. The pure product **3ad** was obtained in 45% yield by flash column chromatography on silica gel (petroleum).

4.5 A typical procedure for the preparation of 5 by condensation of 3ad with hydrazine

To a stirred solution of **3ad** (292.3 mg, 0.5 mmol) in ethanol (5 mL), hydrazine hydrate (85%, 88.3 mg, 0.75 mmol) was added, and the resultant mixture was refluxed for 12 h. The reaction was monitored by TLC until complete consumption of the substrate, the solvent was removed under reduced pressure. The resulting residue was dissolved in EtOAc (20 mL), washed with water (2×20 mL) and brine (20 mL), and dried over anhydrous Na_2SO_4. The filtrate was evaporated under reduced pressure. The resultant residue was purified by silica gel column chromatography [eluent: *V*(petroleum ether)∶*V*(ethyl acetate)=5∶1] to afford **5** as a white solid (185.8 mg, 72%).

3,3-Bis(methylthio)-1-(*p*-tolyl)-2-(trifluoromethyl)prop-2-en-1-one (**3aa**): White solid, m.p. 50～52 ℃ (lit.[17] 53～55 ℃); ^1H NMR (400 MHz, $CDCl_3$) δ: 7.82 (d, *J*=8.3 Hz, 2H), 7.29 (d, *J*=7.7 Hz, 2H), 2.45 (s, 3H), 2.43 (s, 3H), 2.19 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 189.0, 149.9 (q, *J*=4.0 Hz), 145.1, 133.6, 132.1 (q, *J*=30.9 Hz), 129.6, 129.3, 121.0 (q, *J*=273.8 Hz), 21.7, 17.2, 16.9; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −56.52 (s).

3,3-Bis(methylthio)-1-(*o*-tolyl)-2-(trifluoromethyl)prop-2-en-1-one (**3ab**): Yellow oil. ^1H NMR (400 MHz, $CDCl_3$) δ: 7.60 (dd, *J*=7.7, 1.4 Hz, 1H), 7.44～7.40 (m, 1H), 7.30～7.26 (m, 2H), 2.63 (s, 3H), 2.43 (s, 3H), 2.06 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 190.9, 150.6 (q, *J*=3.5 Hz), 140.9, 136.0, 133.7 (q, *J*=30.5 Hz), 132.5, 132.2, 130.4, 125.5, 121.2 (q, *J*=274.0 Hz), 21.6, 16.9, 16.8; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −56.53 (s); HRMS (ESI) calcd for $C_{13}H_{14}F_3OS_2$ $[M+H]^+$ 307.0433, found 307.0437.

3,3-Bis(methylthio)-1-(*m*-tolyl)-2-(trifluoromethyl)prop-2-en-1-one (**3ac**): Yellow solid, m.p. 56～58 ℃ (lit.[17] 58～60 ℃); ^1H NMR (400 MHz, $CDCl_3$) δ: 7.77～7.75 (m, 1H), 7.69～7.66 (m, 1H), 7.43～7.36 (m, 2H), 2.46 (s, 3H), 2.43 (s, 3H), 2.18 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 189.5, 150.2 (q, *J*=3.6 Hz), 138.8, 136.1, 134.8, 132.1 (q, *J*=30.9 Hz), 129.4, 128.7, 126.6, 121.0 (q, *J*=273.8 Hz), 21.3, 17.2, 16.9; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −56.54 (s).

3,3-Bis(methylthio)-1-phenyl-2-(trifluoromethyl)prop-2-en-1-one (**3ad**): White solid, m.p. 84～86 ℃ (lit.[17] 88～90 ℃); ^1H NMR (400MHz, $CDCl_3$) δ: 7.93～7.91 (m, 2H), 7.63～7.59 (m, 1H), 7.52～7.48 (m, 2H), 2.46 (s, 3H), 2.18 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 189.3, 150.4 (q, *J*=3.62 Hz), 136.2, 133.9, 131.9 (q, *J*=30.99

Hz), 129.1, 128.8, 121.0 (q, J=273.58 Hz), 17.2, 16.9; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −56.51 (s).

1-(4-Methoxyphenyl)-3,3-bis(methylthio)-2-(trifluoromethyl)prop-2-en-1-one (**3ae**): Yellow solid, m.p. 74～76 ℃ (lit.[17] 74～76 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.89 (d, J=8.9 Hz, 2H), 6.98 (d, J=8.9 Hz, 2H), 3.88 (s, 3H), 2.45 (s, 3H), 2.22 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 188.0, 164.3, 149.6 (q, J=3.6 Hz), 132.2 (q, J=30.8 Hz), 131.6, 129.1, 121.1 (q, J=273.8 Hz), 114.2, 55.5, 17.2, 16.9; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −56.52 (s).

1-(2-Methoxyphenyl)-3,3-bis(methylthio)-2-(trifluoromethyl)prop-2-en-1-one (**3af**): Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.89 (dd, J=7.8, 1.8 Hz, 1H), 7.54～7.50 (m, 1H), 7.07～7.03 (m, 1H), 6.98～6.95 (m, 1H), 3.86 (s, 3H), 2.39 (s, 3H), 2.07 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 188.1, 159.4, 145.0 (q, J=3.7 Hz), 136.4 (q, J=30.2 Hz), 134.9, 131.7, 126.5, 121.2 (q, J=273.7 Hz), 120.8, 111.9, 55.5, 16.8, 16.7; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −56.85 (s). HRMS (ESI) calcd for $C_{13}H_{14}F_3O_2S_2$ $[M+H]^+$ 323.0382, found 323.0386.

1-(3-Methoxyphenyl)-3,3-bis(methylthio)-2-(trifluoromethyl)prop-2-en-1-one (**3ag**): White solid, m.p. 74～76 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.50～7.49 (m, 1H), 7.45～7.43 (m, 1H), 7.40 (t, J=7.8 Hz, 1H), 7.17～7.14 (m, 1H), 3.87 (s, 3H), 2.46 (s, 3H), 2.19 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 189.1, 160.0, 150.5 (q, J=3.6 Hz), 137.5, 131.8 (q, J=31.0 Hz), 129.8, 122.0, 121.0 (q, J=273.8 Hz), 120.7, 112.7, 55.4, 17.2, 16.9; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −56.55 (s); HRMS (ESI) calcd for $C_{13}H_{14}F_3O_2S_2$ $[M+H]^+$ 323.0382, found 323.0383.

1-([1,1'-Biphenyl]-4-yl)-3,3-bis(methylthio)-2-(trifluoromethyl)prop-2-en-1-one (**3ah**): Yellow solid, m.p. 88～90 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.99 (d, J=8.4 Hz, 2H), 7.72 (d, J=8.6 Hz, 2H), 7.64～7.62 (m, 2H), 7.49～7.45 (m, 2H), 7.43～7.39 (m, 1H), 2.47 (s, 3H), 2.21 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 188.9, 150.4 (q, J=3.6 Hz), 146.6, 139.6, 134.8, 131.9 (q, J=31.0 Hz), 129.8, 129.0, 128.4, 127.5, 127.2, 121.0 (q, J=273.9 Hz), 17.2, 17.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −56.41 (s); HRMS (ESI) calcd for $C_{18}H_{16}F_3OS_2$ $[M+H]^+$ 369.0589, found 369.0585.

3,3-Bis(methylthio)-2-(trifluoromethyl)-1-(4-(trifluoromethyl)phenyl)prop-2-en-1-one (**3ai**):[18] Yellow solid, m.p. 70～72 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.03 (d, J=8.1 Hz, 2H), 7.77 (d, J=8.2 Hz, 2H), 2.48 (s, 3H), 2.19 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 188.1, 152.1 (q, J=3.5 Hz), 139.1, 135.0 (q, J=32.6 Hz), 130.8 (q, J=31.3 Hz), 129.3, 125.9 (q, J=3.73 Hz), 123.5 (q, J=271.2 Hz), 120.9 (q, J=273.9 Hz), 17.3, 17.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −56.47 (s), −63.18 (s).

1-(4-Fluorophenyl)-3,3-bis(methylthio)-2-(trifluoromethyl)prop-2-en-1-one (**3aj**):[18] Yellow viscous material. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.95 (dd, J=8.7, 5.4 Hz, 2H), 7.18 (t, J=8.6 Hz, 2H), 2.47 (s, 3H), 2.21 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 187.8, 166.2 (d, J=255.2 Hz), 150.8 (q, J=3.6 Hz), 132.7 (d, J=2.8 Hz), 131.8 (d, J=9.6 Hz), 131.6 (q, J=31.1 Hz), 121.0 (q, J=274.0 Hz), 116.1 (d, J=22.0 Hz), 17.2, 17.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −56.49 (q), −103.27 (t).

1-(4-Chlorophenyl)-3,3-bis(methylthio)-2-(trifluoromethyl)prop-2-en-1-one (**3ak**): Yellow solid, m.p. 48～50 ℃ (lit.[18] 50～52 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.85 (d, J=8.6 Hz, 2H), 7.48 (d, J=8.5 Hz, 2H), 2.47 (s, 3H), 2.20 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 188.1, 151.1 (q, J=3.5 Hz), 140.5, 134.6, 131.4 (q, J=31.2 Hz), 130.5, 129.2, 120.9 (q, J=273.8 Hz), 17.3, 17.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −56.50 (s).

1-(4-Bromophenyl)-3,3-bis(methylthio)-2-(trifluoromethyl)prop-2-en-1-one (**3al**):[18] Yellow viscous material. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.78 (d, J=8.5 Hz, 2H), 7.64 (d, J=8.5 Hz, 2H), 2.47 (s, 3H), 2.20 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 188.3, 151.2 (q, J=3.5 Hz), 135.0, 132.2, 131.3 (q, J=31.4 Hz), 130.5, 129.3, 120.9 (q, J=273.9 Hz), 17.3, 17.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −56.49 (s).

1-(4-Iodophenyl)-3,3-bis(methylthio)-2-(trifluoromethyl)prop-2-en-1-one (**3am**): Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.87 (d, J=8.5 Hz, 2H), 7.62 (d, J=8.5 Hz, 2H), 2.46 (s, 3H), 2.20 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 188.6, 151.2 (q, J=3.5 Hz), 138.2, 135.6, 131.2 (q, J=30.5 Hz), 130.3, 120.9 (q, J=273.9 Hz), 102.2, 17.3, 17.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −56.46 (s); HRMS (ESI) calcd for $C_{12}H_{11}F_3IOS_2$ $[M+H]^+$ 418.9243, found 418.9247.

1-(2-Bromophenyl)-3,3-bis(methylthio)-2-(trifluoromethyl)prop-2-en-1-one (**3an**): Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.61～7.58 (m, 2H), 7.34～7.30 (m, 1H), 7.28～7.24 (m, 1H), 2.38 (s, 3H), 1.97 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 188.3, 153.8 (q, J=3.1 Hz), 138.3, 134.7, 132.8, 132.1 (q, J=30.1 Hz), 131.3, 127.1, 121.8, 121.3 (q, J=274.4 Hz), 17.18, 17.02; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −56.01 (s); HRMS (ESI) calcd for $C_{12}H_{11}BrF_3OS_2$ $[M+H]^+$ 370.9381, found 370.9383.

1-(3-Bromo-4-fluorophenyl)-3,3-bis(methylthio)-2-(trifluoromethyl)prop-2-en-1-one (**3ao**): White solid, m.p. 58～60 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.16 (dd, J=6.5, 2.2 Hz, 1H), 7.83 (ddd, J=8.6, 4.6, 2.2 Hz, 1H), 7.24 (t, J=8.2 Hz, 1H), 2.48 (s, 3H), 2.22 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 186.6, 162.4 (d, J=255.5 Hz), 152.0 (q, J=3.4 Hz), 134.7 (d, J=1.8 Hz), 133.8 (d, J=3.4 Hz), 130.6 (q, J=31.3 Hz), 130.3 (d, J=8.7 Hz), 120.8 (q, J=273.9 Hz), 116.9 (d, J=23.1 Hz), 110.2 (d, J=21.8 Hz), 17.3, 17.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −56.42 (s), −97.71 (s); HRMS (ESI) calcd for $C_{12}H_{10}BrF_4OS_2$ $[M+H]^+$ 388.9287, found 388.9287.

1-(3,4-Dimethoxyphenyl)-3,3-bis(methylthio)-2-(trifluoromethyl)prop-2-en-1-one (**3ap**): Yellow solid, m.p. 84～86 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.56 (d, J=2.0 Hz, 1H), 7.45 (dd, J=8.4, 2.0 Hz, 1H), 6.93 (d, J=8.4 Hz, 1H), 3.96 (d, J=8.0 Hz, 6H), 2.46 (s, 3H), 2.23 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 188.1, 154.2, 149.7 (q, J=3.6 Hz), 149.4, 132.0 (q, J=31 Hz), 129.2, 124.8, 121.0 (q, J=273.7), 113.5, 110.1 (d, J=9.4 Hz), 56.0, 55.9,

17.2, 16.9; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −56.56 (s); HRMS (ESI) calcd for $C_{14}H_{16}F_3O_3S_2$ $[M+H]^+$ 353.0487, found 353.0489.

3,3-Bis(methylthio)-1-(naphthalen-2-yl)-2-(trifluoromethyl)prop-2-en-1-one (**3aq**): Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.38～8.38 (m, 1H), 8.03～7.97 (m, 2H), 7.94～7.88 (m, 2H), 7.64～7.60 (m, 1H), 7.58～7.54 (m, 1H), 2.48 (s, 3H), 2.16 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 189.3, 150.6 (q, J=3.6 Hz), 136.0, 133.6, 132.5, 132.0 (q, J=31.0 Hz), 131.4, 129.7, 129.0, 128.9, 127.9, 127.0, 124.1,121.1 (q, J=273.9 Hz), 17.2, 17.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −56.41 (s); HRMS (ESI) calcd for $C_{16}H_{14}F_3OS_2$ $[M+H]^+$ 343.0433, found 343.0436.

1-(Furan-2-yl)-3,3-bis(methylthio)-2-(trifluoromethyl)-prop-2-en-1-one (**3ar**):[18] Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.68 (dd, J=1.6, 0.8 Hz, 1H), 7.19～7.18 (m, 1H), 6.59 (dd, J=3.6, 1.7 Hz, 1H), 2.47 (s, 3H), 2.27 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 176.6, 152.6(q, J=3.5 Hz), 151.9, 148.0, 130.7 (q, J=31.4 Hz), 121.0 (q, J=273.7 Hz), 119.9, 112.7, 17.6, 17.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −56.40 (s).

3,3-Bis(methylthio)-1-(thiophen-2-yl)-2-(trifluoromethyl)prop-2-en-1-one (**3as**):[18] Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.75 (dd, J=4.9, 1.2 Hz, 1H), 7.64 (dd, J=3.8, 1.2 Hz, 1H), 7.16 (dd, J=4.9, 3.8 Hz, 1H), 2.46 (s, 3H), 2.26 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 181.5, 151.6 (q, J=3.5 Hz), 143.5, 135.6, 134.2, 131.6 (q, J=31.3 Hz), 128.4, 120.9 (q, J=273.8 Hz), 17.5, 17.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −56.31 (s).

4,4-Bis(methylthio)-3-(trifluoromethyl)but-3-en-2-one (**3at**):[17] Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 2.43 (s, 6H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 196.0, 150.4 (q, J=3.6 Hz), 134.0 (q, J=30.5 Hz), 121.0 (q, J=273.7 Hz), 30.7 (2C), 17.28; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −56.24 (s).

3-(1,3-Dithiolan-2-ylidene)-4,4,4-trifluorobutan-2-one (**3au**):[17] Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 3.41 (dd, J=7.4, 4.9 Hz, 2H), 3.32 (dd, J=7.7, 5.4 Hz, 2H), 2.38 (q, J=2.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 191.0, 174.9, 124.8 (q, J=271.5 Hz), 115.7 (q, J=31.5 Hz), 37.7, 36.7 (q, J=3.1 Hz), 28.6 (q, J=4.2 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ: −54.02 (s).

Methyl 4-(3,3-bis(methylthio)-2-(trifluoromethyl)-acryloyl)benzoate (**3av**): White solid, m.p. 74～76 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.17～8.15 (m, 2H), 7.98～7.95 (m, 2H), 3.96 (s, 3H), 2.47 (s, 3H), 2.16 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 188.6, 166.0, 151.7 (q, J=4 Hz), 139.6, 134.4, 131.2 (q, J=31 Hz), 130.0, 128.9, 120.9 (q, J=274 Hz), 52.5, 17.2, 17.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −56.49 (s); HRMS (ESI) calcd for $C_{14}H_{14}F_3O_3S_2$ $[M+H]^+$ 351.0331, found 351.0337.

5-(Methylthio)-3-phenyl-4-(trifluoromethyl)-1*H*-pyrazole (**5**): White solid, m.p. 94～96 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 11.51 (brs, 1H), 7.44～7.41 (m, 5H), 2.46 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 146.3, 130.0, 128.8 (2C), 128.5, 122.9 (q, J=266 Hz), 108.7, 17.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −54.01 (s); HRMS (ESI) calcd for $C_{11}H_{10}F_3N_2S$ $[M+H]^+$ 259.0511, found 259.0517.

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