Journal of Fluorine Chemistry 245 (2021) 109778

Contents lists available at ScienceDirect

Journal of Fluorine Chemistry

journal homepage: www.elsevier.com/locate/fluor

Short Communication

t-BuOK-promoted methylthiolation of aryl fluorides with dimethyldisulfide under transition-metal-free and mild conditions

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ARTICLE INFO

Keywords:
Methylthiolation
Transition-metal-free
Dimethyldisulfide
Aryl methyl sulfide
Aryl fluoride

ABSTRACT

In the presence of potassium *tert*-butoxide (*t*-BuOK), the cross-coupling reaction between aryl fluorides and dimethyldisulfide was developed. A series of aryl methyl sulfides were obtained in moderate to good yields under transition-metal-free and mild conditions.

1. Introduction

Aryl methyl sulfides are highly important structural units existing in biological and pharmaceutical active compounds as well as functional materials [1]. In addition, they are extensively used as effective reagents and building blocks in synthetic organic chemistry. Specifically, these compounds can be expediently transferred into sulfoxides or sulfones [2], thiols or arenes [3] and trifluoromethylthioarenes [4]. Meanwhile, they also play as promising precursor of arylating agent for the construction of C—C [5], C—N [6], C—B [7] or C—P [8] bond and the carbothiolation of terminal alkynes [9] via C(sp^2)-S bond cleavage. Consequently, numerous synthetic strategies for methylthiolation from various organic species have been investigated so far. Indubitably, the cross-coupling of available thiols with aryl halides or pseudohalides catalyzed by palladium [10] or copper [11] complexes dominated approaches to various aryl sulfides under relatively mild conditions, however, methylthiolation in this fashion was constrained presumably due to low-boiling point of methanethiol. In general, prevalent routes to aryl methyl sulfides were mainly achieved by the reduction of avaliable sulfoxides [12], the coupling of aryl thiols with iodomethane [13] or dimethylcarbonate [14], and several other sulfur transferring agents like sodium methylthiolate or sulfinate [15], sulfur powder [16], *S*-methylisothiourea sulfate [17] as well as the alliance of masked inorganic sulfur and dimethylcarbonate [18] with aryl halides. In addition, dimethyl sulfoxide has also been proven to be a cheap and direct methylthiolation surrogate to react with aryl halides [19], aryl carboxylic acids [20], and arenes or heteroarenes [21] recently.

Apart from the aforementioned thiomethyl sources, dimethyldisulfide, a commercial food additive and sulfidation agent, has also unquestionably received considerable attention because of high reactivity and relative stability. Earlier, thiolation of aryl iodides with dimethyldisulfide under neutral conditions by the assistance of nickel catalyst with zinc was reported by Taniguchi [22] and a similar procedure was developed by Morales-Morales et al. [23]. From a viewpoint of atom economy, directed ortho-lithiation of aryl C—H bond of phenylcarbamic acid 1,1-dimethylethyl ester and 2-chloropyridyl group complexation followed by electrophilic substitution with dimethyldisulfide were successfully achieved by Stanetty's [24] and Fort's group [25] separately. Dialkylhydrazides for directed orthometalations was also disclosed by Wuts's group [26]. Subsequently, a $Cu(OAc)_2$-mediated thioetherification of the C—H bond of 2-phenylpyridine with dimethyldisulfide was established by Yu et al [27]. Another protocol for direct thiolation of aryl C—H bonds with disulfides employing palladium and copper as co-catalyst was reported by Nishihara's group [28]. Moreover, several other elegant protocols were exploited under the popular conditions which exactly in accordance with green and sustainable chemistry recently. For instance, methylthiolation from arenediazonium salts and dimethyldisulfide using organic dyes eosin Y under irradiation of visible-light or only adding a weak and eco-friendly base was feasible [29]. And Polyzos et al. [30] established a unified method for the thiolation of aryl and vinyl iodides with dimethyldisulfide using visible light photocatalyst *fac*-$Ir(ppy)_3$ under mild condition. Besides, an efficient di-*tert*-butyl peroxide (DTBP)-mediated C(aryl)-S bond formation through arylboronic acids and dimethyldisulfide under

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https://doi.org/10.1016/j.jfluchem.2021.109778
Received 13 January 2021; Received in revised form 12 March 2021; Accepted 14 March 2021
Available online 16 March 2021

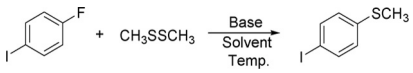
transition-metal-free and neutral condition, and very recently a copper-catalyzed methylthiolation of aryl iodides and bromides with dimethyldisulfide in neat water were reported by our group [31].

Despite of substantial progress over the years, the existing cases still suffer from one or more disadvantages, such as the employment of transition metal catalyst, participation of an appropriate additive or specially designed ligand, prefunctionalization or limitation of substrates, organic peroxide, inert atmosphere, as well as requirement of high temperature. Concerning on environmental issues and the avoidance of trace-metal impurity in the target molecules, especially in the pharmaceutical industry, the improvement of a transition-metal-free and mild process for aryl methyl sulfides was desirable yet. On the other hand, owing to the contradiction between the abundance of low-cost aryl fluorides and the formidable challenge of the activation of relatively inert C—F bond which was generally assisted by transition metal catalysts [32] or recently by some main group based molecules [33], an efficient route to realize C(sp^2)-S bond *via* C(sp^2)-F bond cleavage remains attractive and expectative. Although several protocols for syntheses of fluorinated thioether derivatives possessing specific physiological activities have been explored by some groups to date [34], regrettably, these procedures were generally catalyzed by expensive transition metal catalysts and limited to the coupling of highly reactive polyfluoroarenes such as hexafluorobenzene, pentafluorobenzene as well as pentafluorobenzoic acid with disulfides or aryl thiols. Hence, herein, we would like to report an efficient methylthiolation from simple fluoroarenes with dimethyldisulfide under transition-metal-free and mild conditions.

2. Results and discussion

In the course of our continuing research toward the C(sp^2)-S bond construction under transition-metal-free conditions, we observed that an advance in radical chemistry that aryl halides could be smoothly transformed to aryl radicals via a single electron transfer and C-halogen bond cleavage sequence in the case of an organic additive and a base [35]. In addition, it was acquired that only C(sp^2)-F bond was reserved during the work that transition-metal-free arylation of enolizable aryl ketones with aryl halides using *t*-BuOK in *N,N*-dimethylformamide (DMF) via a radical chain reaction [36]. So, we envisioned that the in situ generated aryl radical from aryl halides would also probably couple with dimethyldisulfide in one reaction system through a radical mechanism with no need for metal catalyst. Consequently, when treating 4-fluoroiodobenzene with dimethyldisulfide in the presence of *t*-BuOK and DMF in air at 70 °C, without any transition-metal catalyst or additive, 4-fluorothioanisole might be the target mole. Surprisingly, 4-iodothioanisole was obtained as a major product in 73 % yield after 6 h, namely, C—F bond was broken and C—I bond remained intact (Table1, entry 1), which was obviously not consistent with the radical mechanism disclosed in the above-mentioned reports. However, these results exactly led us to pay our attention to introduce methylthio group into aromatic compounds through C(sp^2)-F bond cleavage. Further closer investigation illustrated only negligible amount of 1,4-bis(methylthio) benzene derived from the cleavage of both C—F and C—I bond was determined by HNMR analysis. Encouraged by this intriguing consequence, we further optimized the reaction conditions and the results were summarized in Table 1. First, several other solvents were tested and CH_3CN provided the highest yield (entry 2). Transformation proceeded in dimethylacetamide (DMA) and dimethyl sulfoxide (DMSO) afforded the target product in similar yields, approximate to 30 %, whereas the employment of 1,2-dichloroethane (DCE), tetrahydrofuran (THF) and CH_3OH as solvents resulted in lower yields or invalid reaction (entries 3–7). Subsequently, the screening of various bases revealed that *t*-BuONa and KOH gave relatively low conversion and the other bases such as Cs_2CO_3, K_3PO_4 and K_2CO_3 were nearly ineffective in the reaction (entries 8–12). Meanwhile, a control experiment displayed that methylthiolation reaction did not occur in the absence of any base (entry 13).

Table 1
Optimization of reaction conditions[a].



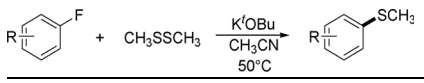
Entry	Base	Solvent	T(°C)	Yield(%)[b]
1	*t*-BuOK	DMF	70	73
2	*t*-BuOK	CH_3CN	70	76
3	*t*-BuOK	DMSO	70	28
4	*t*-BuOK	DMA	70	35
5	*t*-BuOK	DCE	70	10
6	*t*-BuOK	THF	70	< 5
7	*t*-BuOK	CH_3OH	70	< 5
8	*t*-BuONa	CH_3CN	70	55
9	KOH	CH_3CN	70	60
10	Cs_2CO_3	CH_3CN	70	12
11	K_3PO_4	CH_3CN	70	< 5
12	K_2CO_3	CH_3CN	70	< 5
13	/	CH_3CN	70	N. D
14	*t*-BuOK (1.0eq.)	CH_3CN	70	45
15	*t*-BuOK (3.0eq.)	CH_3CN	70	82
16	*t*-BuOK (4.0eq.)	CH_3CN	70	78
17	*t*-BuOK (3.0eq.)	CH_3CN	80	72
18	*t*-BuOK (3.0eq.)	CH_3CN	60	83
19	*t*-BuOK (3.0eq.)	CH_3CN	50	90
20	*t*-BuOK (3.0eq.)	CH_3CN	40	68
21	*t*-BuOK (3.0eq.)	CH_3CN	r.t.	40
22[c]	*t*-BuOK (3.0eq.)	CH_3CN	50	42
23[d]	*t*-BuOK (3.0eq.)	CH_3CN	50	93
24[e]	*t*-BuOK (3.0eq.)	CH_3CN	50	89
25[f]	*t*-BuOK (3.0eq.)	CH_3CN	50	89
26[g]	*t*-BuOK (3.0eq.)	CH_3CN	50	90

[a] Reaction conditions: 4-fluoroiodobenzene (1.0 mmol), CH_3SSCH_3 (1.0 mmol), base (2.0 mmol), solvent (2.0 mL), 6 h, sealed tube, in air.
[b] Isolated yield.
[c] CH_3SSCH_3 (0.5 eq.).
[d] CH_3SSCH_3 (1.2 eq.).
[e] CH_3SSCH_3 (1.5 eq.).
[f] 12 h.
[g] N_2.

Further, it was found that reducing the amount of *t*-BuOK led to a low efficiency and the reaction could be conducted well when 3.0 equiv. amount of *t*-BuOK based on 4-fluoroiodobenzene was devoted (entries 14–16). In addition, the effect of reaction temperature was tested. A diminution to 40 °C or at room temperature resulted in lower yields and an elevated temperature to 80 °C did not afford a better yield (entries 17–21). So the optimal reaction temperature was at 50 °C or so. A lower loading of dimethyldisulfide led to diminished yield and no apparent improvement was observed when increasing the amount over 1.2 equiv. (entries 22–24). Extending the reaction time to 12 h did not provide a better result under otherwise identical conditions and the argon atmosphere was unnecessary (entries 2526). So, it was concluded that the optimal condition for the methylthiolation of 4-fluoroiodobenzene with dimethyldisulfide was the combination of *t*-BuOK and CH_3CN at 50 °C for 6 h in air.

With the optimized reaction conditions, the generality of methylthiolation of aryl fluorides with dimethyldisulfide was evaluated. As illustrated in Table 2, a variety of aryl fluorides were subjected to the reaction conditions, and the corresponding products were obtained in 40–93 % yields. Obviously, similar to 4-fluoroiodobenzene, in the case of dihaloarenes bearing fluorine group along with an additional halogen substituent, the major products formed through the selective C—F bond cleavage coupling were achieved while keeping iodo, bromo or chloro group intact, which demonstrating that a high degree of chemoselectivity is possible in this methylthiolation reaction and undoubtedly these reserved functional groups might be convenient for further functionalization through a cross-coupling reaction (Table 2, entries 1–9). Similarly, several disubstituted aryl fluorides containing additional two different halogen groups, the yield of target mole formed selectively via

Table 2
Methylthiolation of aryl fluorides with dimethyldisulfide [a].



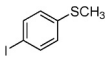
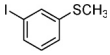
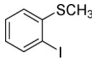
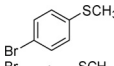
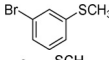
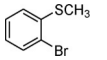
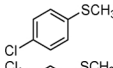
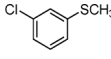
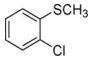
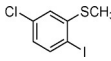
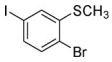
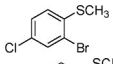
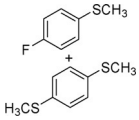
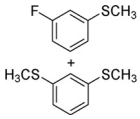
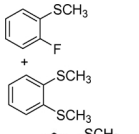
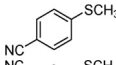
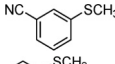
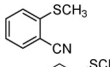
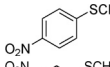
Entry	Substrate	Product	Yield(%)[b]
1			93
2			88
3			72
4			87
5			80
6			68
7			81
8			70
9			68
10			48
11			62
12			40
13			trace
14[c]			45(30)
15[c]			45(20)
16			80
17			75
18			63
19			76
20			60
21			71
22			63
23			trace
24			trace
25			trace
26			50

Table 2 (*continued*)

Entry	Substrate	Product	Yield(%)[b]
27			40
28			55
29			42
30			61
31			46

[a] Reaction conditions: aryl halides (1.0 mmol), CH_3SSCH_3 (1.2 mmol), *t*-BuOK (3.0 mmol), CH_3CN (2 mL), 50 °C, 6 h, sealed tube, in air.
[b] Isolated yield.
[c] Yields of double methylthiolation products are shown in parentheses.

C—F bond cleavage, that is to say, the Cl, Br or I group was untouched, was satisfactory (entries 10–12). Notably, as for difluorobenzene such as 1,3- and 1,2-difluorobenzene, the mono methylthiolation products were formed in reasonable yields, together with non-negligible amounts of the corresponding doubly substitutional products, however, 1,4-difluorobenzene only gave minimal conversion under the tested conditions (entries 13–15). Obviously, the observed phenomena were in accordance with previously reported studies about *N*-arylation of amines with unactivated fluorobenzenes by Diness [37]. Furthermore, using *para*-, *meta*- and *ortho*-fluorobenzonitrile as substrates could also afford the corresponding products in satisfactory yields (entries 16–18). Unexpectedly, despite 4-fluoronitrobenzene and 3-fluoronitrobenzene were appropriate to this reaction, the yields of target moles were moderate (entries 1920). Generally, compared with *para*- and *meta*-substituted fluoroarenes described above, a substrate with an *ortho*-substituent could also proceed smoothly, however, generating the desired product in a relatively lower yield, which is possibly due to the steric hindrance.

Considering that heteroaryl methyl sulfides were of much interest for biological activity, heteroaryl analogues were evaluated. For example, pyridinyl fluorides such as 2-fluoropyridine and 3-fluoropyridine appeared as suitable substrates in this methylthiolation process, affording the corresponding products in 71 % and 63 % of yields (entries 2122).

Regretfully, the optimal conditions was not suitable for fluorobenzene and the electron-donating group substituted fluoroarenen like 4-fluorotoluene or 3-fluorotoluene and only trace amount of target mole was detected (entries 23–25). Not so surprisingly, although the invalidation of unsubstituted halobenzene (including I, Br and Cl) similar to fluorobenzene, 4-bromoiodobenzene or 4-chlorobromobenzene used as substrates could obtain a mixture of two mono methylthiolation products with somewhat limited yields, which were detected by HNMR analysis but difficult to be separated. Gladly, 1,3- or 1,4-dihalobenzene possessing two same halogen groups (including I, Br and Cl) also proved to be compatible with the reaction conditions and gave reasonable yield of the corresponding mono-methylthiolation products even if the presence of 3 equiv. of dimethyldisulfide (entries 26–31).

In order to verify the absence of aryl radicals during the process, a radical-trap experiment was initially performed. When stoichiometric amount of the radical scavengers, 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO, 3 equiv. based on 4-fluoroiodobenzene) was added to the reaction mixture under exactly the same reaction conditions, the formation of expected product was almost unaffected, which might rule out radical intermediates in the C–F bond cleavage process. In addition, without the presence of regioisomers of the target mole in NMR spectra of the crude reaction mixture could eliminate aryne intermediate in the

process. Based on the aforementioned observation and the related publications [15a,38], the procedure was likely to follow the classical nucleophilic aromatic substitution reaction in which fluorine was normally displaced faster than chlorine, bromine and iodine. Indeed, the observed selectivity in fluorobenzene derivatives containing other halogen atom was consistent with the substituent effect. A plausible mechanism was illustrated in Scheme 1. The reactive sulfhydryl ion intermediates were generated from dimethyldisulfide in the presence of base, which then served as the nucleophilic partner in the addition steps to yield intermediate **B**. Then the following elimination steps led to aryl methyl sulfide product **C**.

3. Conclusions

In summary, methylthiolation of aryl fluorides with dimethyldisulfide under transition-metal-free and mild conditions has been established, which would effectively avoid trace metal impurity in the target products. We hope this protocol can provide an attractive complement to the existing strategies for the access to aryl methyl sulfides and C—F bond functionalization.

4. Experimental section

4.1. General remarks

All reagents were used directly without further purification. Hydrogen nuclear magnetic resonance spectra (^{1}H NMR) were obtained at 300 MHz in $CDCl_3$ solution. Carbon-13 nuclear magnetic resonance spectra (^{13}C NMR) were obtained at 75 MHz in $CDCl_3$ solution. Chemical shifts are reported in ppm relative to the TMS (^{1}H NMR) and to the solvent (^{13}C NMR). Gas chromatography mass spectra (GC/MS) were recorded on a Saturn 2000GC/MS instrument. Thin layer chromatography (TLC) was performed using Merck Silica Gel GF254, 0.25 mm thickness.

4.2. Synthetic procedures and spectral data

4.2.1. Synthetic procedures

Aryl fluorides (1.0 mmol), CH_3SSCH_3 (1.2 mmol), *t*-BuOK (3.0 mmol) and CH_3CN (2 mL) were taken in a 25-ml sealed tube. The reaction mixture was stirred at 50 °C for 6 h under air. After cooling to room temperature, the product was diluted with H_2O (5 mL) and extracted with EtOAc (4 × 10 mL). The extracts were combined and washed by brine (3 × 10 mL), dried over $MgSO_4$, filtered, and evaporated, and purified by chromatography on silica gel to obtain the desired products with ethyl acetate/hexane (v/v = 1:30 ~ 1:100). The products were characterized by their spectral and analytical data and compared with those of the known compounds (See supporting information).

4.2.2. Spectral data

4.2.2.1. 4-Iodothioanisole (Table 2, entry 1)[29a]. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.56 (d, *J* = 8.1 Hz, 2 H), 6.96 (d, *J* = 8.1 Hz, 2 H), 2.44 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 138.6, 137.6, 128.2, 89.3, 15.7. GC–MS (EI, *m/z*): 250 [M+].

4.2.2.2. 3-Iodothioanisole (Table 2, entry 2)[39]. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.54 (s, 1 H), 7.42 (d, *J* = 7.5 Hz, 1 H), 7.16 (d, *J* = 7.8 Hz, 1 H), 6.98–6.93 (m, 1 H), 2.43 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 141.0, 134.5, 133.9, 130.2, 125.6, 94.9, 15.7. GC–MS (EI, *m/z*): 250 [M+].

4.2.2.3. 2-Iodothioanisole (Table 2, entry 3)[40]. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.76 (d, *J* = 7.8 Hz, 1 H), 7.35–7.30 (m, 1 H), 7.06 (d, *J* = 7.8 Hz, 1 H), 6.85–6.80 (m, 1 H), 2.44 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 143.1, 139.3, 128.7, 126.0, 124.8, 97.3, 17.0. GC–MS (EI, *m/z*): 250 [M+].

4.2.2.4. 4-Bromothioanisole (Table 2, entry 4)[29a]. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.37 (d, *J* = 8.4 Hz, 2 H), 7.09 (d, *J* = 8.4 Hz, 2 H), 2.44 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 137.7, 131.8, 128.1, 118.6, 15.9. GC–MS (EI, *m/z*): 203 [M+].

4.2.2.5. 3-Bromothioanisole (Table 2, entry 5)[31a]. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.34 (s, 1 H), 7.24–7.22 (m, 1 H), 7.13–7.10 (m, 2 H), 2.45 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 141.0, 130.1, 128.6, 127.9, 124.9, 122.9, 15.6. GC–MS (EI, *m/z*): 203 [M+].

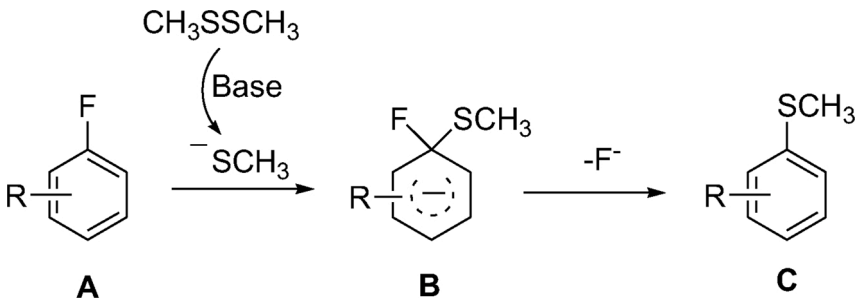
4.2.2.6. 2-Bromothioanisole (Table 2, entry 6)[30]. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.50 (d, *J* = 7.8 Hz, 1 H), 7.30–7.25 (m, 1 H), 7.09 (d, *J* = 7.8 Hz, 1 H), 7.00–6.95 (m, 1 H), 2.44 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 139.7, 132.6, 127.8, 125.7, 125.4, 121.7, 15.7. GC–MS (EI, *m/z*): 203 [M+].

4.2.2.7. 4-Chlorothioanisole (Table 2, entry 7)[29a]. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.23 (d, *J* = 8.7 Hz, 2 H), 7.14 (d, *J* = 8.7 Hz, 2 H), 2.45 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 137.0, 130.8, 128.9, 127.8, 16.0. GC–MS (EI, *m/z*): 159 [M+].

4.2.2.8. 3-Chlorothioanisole (Table 2, entry 8)[29a]. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.19–7.17 (m, 2 H), 7.10–7.09 (m, 2 H), 2.45 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 140.7, 134.7, 129.8, 125.8, 125.0, 124.4, 15.5. GC–MS (EI, *m/z*): 159 [M+].

4.2.2.9. 2-Chlorothioanisole (Table 2, entry 9) [41]. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.33–7.31 (m, 1 H), 7.25–7.20 (m, 1 H), 7.14–7.12 (m, 1 H), 7.08–7.03 (m, 1 H), 2.45 (s, 3 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 137.7, 131.7, 129.3, 127.2, 125.5, 15.2. GC–MS (EI, *m/z*): 159 [M+].

4.2.2.10. 5-Chloro-2-iodothioanisole (Table 2, entry 10)[40]. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.62 (d, *J* = 8.1 Hz, 1 H), 6.96 (s, 1 H), 6.79 (d, *J* = 8.4 Hz, 1 H), 2.44 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 145.4, 140.0, 135.1, 125.9, 124.2, 93.9, 17.0. GC–MS (EI, *m/z*): 284 [M+].



Scheme 1. Plausible reaction mechanism.

4.2.2.11. 2-Bromo-5-iodothioanisole (Table 2, entry 11)[42]. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.30−7.16 (m, 3 H), 2.44 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 142.3, 134.4, 134.0, 133.3, 121.1, 93.2, 15.9. GC–MS (EI, *m/z*): 329 [M+].

4.2.2.12. 2-Bromo-4-chlorothioanisole (Table 2, entry 12)[43]. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.52−7.51 (m, 1 H), 7.27−7.24 (m, 1 H), 7.03−7.00 (m, 1 H), 2.45 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 138.5, 132.2, 130.6, 128.0, 126.1, 121.8, 15.9. GC–MS (EI, *m/z*): 238 [M+].

4.2.2.13. 3-Fluorothioanisole (Table 2, entry 14)[44]. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.22−7.17 (m, 1 H), 7.00−6.91 (m, 2 H), 6.83−6.77 (m, 1 H), 2.45 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 163.0 (164.7, 161.4, d, J = 245.8 Hz), 141.1, 130.0, 121.8, 112.9 (113.1, 112.8, d, J = 23.6 Hz), 111.7 (111.9, 111.6, d, J = 21.2 Hz), 15.4. GC–MS (EI, *m/z*): 142 [M+].

4.2.2.14. 1,3-Bis(methylthio)benzene (Table 2, entry 14)[45]. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.27−7.13 (m, 2 H), 7.03−7.00 (m, 2 H), 2.48 (s, 6 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 139.3, 129.1, 124.0, 123.1, 15.7. GC–MS (EI, *m/z*): 170 [M+].

4.2.2.15. 2-Fluorothioanisole (Table 2, entry 15)[46]. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.27−7.22 (m, 1 H), 7.17−6.99 (m, 3 H), 2.45 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 160.3 (162.0, 158.7, d, J = 242.8 Hz), 128.6, 126.9, 125.5 (125.6, 125.4, d, J = 17.0 Hz), 124.5, 115.3 (115.4, 115.1, d, J = 21.6 Hz), 15.5. GC–MS (EI, *m/z*): 142 [M+].

4.2.2.16. 1,2-Bis(methylthio)benzene (Table 2, entry 15)[45]. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.21−7.12 (m, 4 H), 2.45 (s, 6 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 137.4, 126.6, 125.9, 16.3. GC–MS (EI, *m/z*): 170 [M+].

4.2.2.17. 4-Cyanothioanisole (Table 2, entry 16)[19b]. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.52 (d, J = 8.1 Hz, 2 H), 7.24 (d, J = 8.4 Hz, 2 H), 2.50 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 146.2, 132.1, 125.4, 119.0, 107.5, 14.7. GC–MS (EI, *m/z*): 149 [M+].

4.2.2.18. 3-Cyanothioanisole (Table 2, entry 17)[31a]. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.45−7.36 (m, 4 H), 2.50 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 140.9, 130.3, 129.3, 128.7, 128.2, 118.5, 113.0, 15.3. GC–MS (EI, *m/z*): 149 [M+].

4.2.2.19. 2-Cyanothioanisole (Table 2, entry 18)[6c]. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.60−7.51 (m, 2 H), 7.33−7.30 (m, 1 H), 7.24−7.19 (m, 1 H), 2.57 (s, 3 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 143.6, 133.5, 132.9, 126.1, 125.1, 117.0, 111.5, 15.7. GC–MS (EI, *m/z*): 149 [M+].

4.2.2.20. 4-Nitrothioanisole (Table 2, entry 19)[29a]. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.12 (d, J = 9.0 Hz, 2 H), 7.28 (d, J = 9. Hz, 2 H), 2.56 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 149.0, 144.6, 124.9, 123.8, 14.8. GC–MS (EI, *m/z*): 169 [M+].

4.2.2.21. 3-Nitrothioanisole (Table 2, entry 20)[29a]. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.01 (s, 1 H), 7.93 (d, J = 8.1 Hz, 1 H), 7.52 (d, J = 8.1 Hz, 1 H), 7.45−7.43 (m, 1 H), 2.55 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 148.6, 141.6, 131.8, 129.4, 120.1, 119.5, 15.3. GC–MS (EI, *m/z*): 169 [M+].

4.2.2.22. 2-Methylthiopyridine (Table 2, entry 21)[19b]. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.44−8.42 (m, 1 H), 7.49−7.44 (m, 1 H), 7.16 (d, J = 7.8 Hz, 1 H), 6.98−6.94 (m, 1 H), 2.56 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 159.9, 149.4, 135.8, 121.4, 119.0, 13.2. GC–MS (EI, *m/z*): 125 [M+].

4.2.2.23. 3-Methylthiopyridine (Table 2, entry 22)[47]. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.51 (s, 1 H), 8.38 (s, 1 H), 7.55 (d, J = 7.8 Hz, 1 H), 7.22−7.18 (m, 1 H), 2.50 (s, 3 H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 147.8, 146.1, 135.4, 134.1, 123.4, 15.7. GC–MS (EI, *m/z*): 125 [M+].

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests.

Acknowledgements

We are grateful for financial support from the Natural Science Foundation of Zhejiang Province (No.LQ18B020001).

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