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Synthesis of aryl sulfides *via* visible light-induced solventylation in diarylazo sulfides

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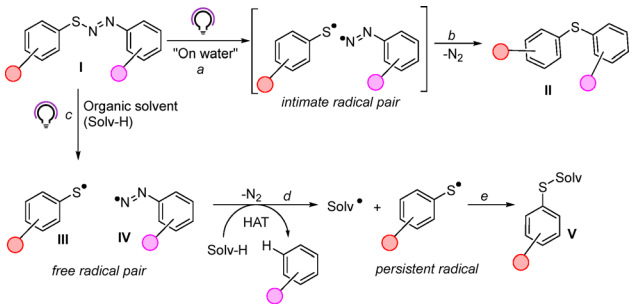
A metal-free C(sp^3)–H bond thiolation of common organic solvents to give valuable aryl sulfides was carried out under very mild conditions upon visible light irradiation of colored diarylazo sulfides.

Aryl sulfides are key scaffolds in several drugs or biologically active compounds that exhibit anti-tumour, anticonvulsant, anti-inflammatory and antioxidant properties.[1–3] Some examples are the azole antifungal medication Butoconazole,[4] the anorectic drug Tiflorex[5,6] and the coccidiostat medicine Toltrazuril.[7] These derivatives are mainly accessed starting from aryl electrophiles and thiols by metal-catalyzed C–S bond formation[8–12] or under photocatalytic conditions.[13] Metal-free, uncatalyzed preparation of aryl sulfides is obviously desirable.

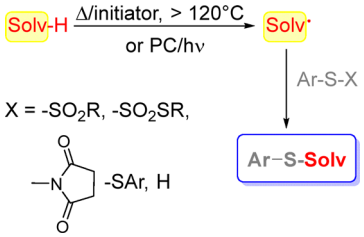
Recently, we discovered that diarylazo sulfides (**I**) when irradiated in the solid state in a water suspension underwent the cleavage of the S–N bond and the resulting intimate radical pair led to diaryl sulfides **II** upon nitrogen extrusion (Scheme 1, paths a and b).[14] We wondered whether by switching to homogeneous conditions we could divert the fate of the reaction, since the initial photohomolytic cleavage could release a free radical pair composed of a thiyl radical **III** and an aryldiazenyl radical **IV** (path c).

The latter intermediate easily loses nitrogen, and the resulting reactive aryl radical can trigger a hydrogen atom abstraction (HAT) reaction with the solvent (Solv-H, path d), ultimately producing valuable aryl sulfides **V** by radical coupling between the resulting Solv$^\bullet$ and the persistent ArS$^\bullet$ in what is considered a solventylation reaction (path e).

The preparation of compounds **V** under metal-free conditions is only sparsely reported and is resumed in Scheme 2. The strategy makes use of a solution of Ar-*S*-X derivatives in a chosen solvent



Scheme 1 Reaction design for the synthesis of solventylated derivatives **V**.



Scheme 2 Metal-free routes to Ar-*S*-Solv derivatives.

(Solv-H) by heating (>120 °C) in the presence of a radical initiator (*e.g. tert*-butylperoxide) or by irradiation in the presence of a photocatalyst *via* generation of a Solv$^\bullet$ intermediate.

To this aim, alkyl arylsulfinates,[15] sodium sulfinates,[16] thiosulfonates[17,18] and 1-(arylthio)pyrrolidine-2,5-diones[19] were used as sulfenylation agents. Alternatively, the thiolation of a C(sp^3)–H bond may be obtained by using diaryldisulfides[20–27] or, more rarely, a thiophenol[28] as sulfur sources. In this way, the thiolation of ethers, amides, cycloalkanes and so on was pursued.

In view of these premises, the aim of the present work is to achieve the hydrogen atom transfer-assisted C(sp^3)–H thioarylation of common organic solvents to form adducts **V** (Scheme 1).

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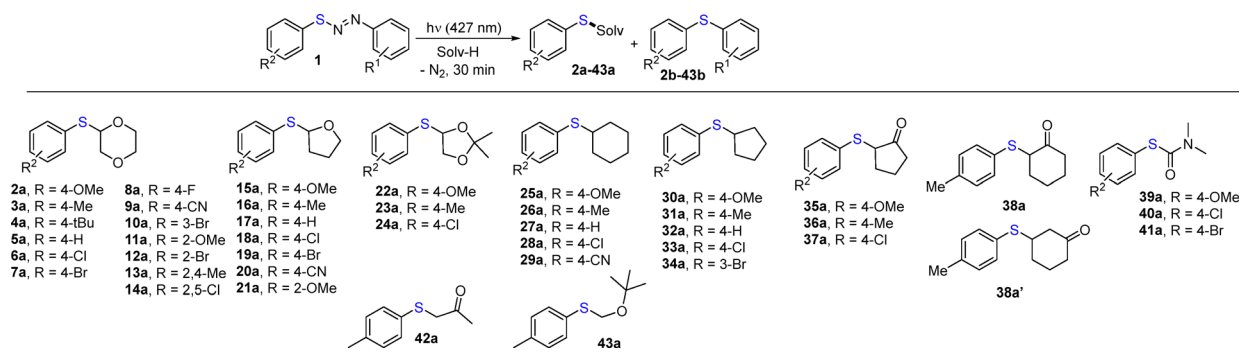
Diarylazo sulfides **1a–q** are bench-stable and coloured derivatives[14] and have been prepared starting from the corresponding anilines and aryl sulfides in up to 89% yield (Chart S1, SI). We initially tested the reaction of 1-(4-chlorophenyl)-2-(*p*-tolylthio)diazene **1b** upon visible light irradiation (λ = 427 nm), by using 1,4-dioxane as the coupling partner (Table S1).

Thus, a 0.05 M solution of **1b** in DMC in the presence of 1,4-dioxane (10 equiv.) was irradiated for 16 h and low amounts of the adduct **3a** (11% yield) were accompanied by the undesired photoextrusion product **3b** as the main product (entry 1). The same reaction was then performed in neat 1,4-dioxane again, leading to a mixture in which **3a** is now by far the major product (64%, entry 2). On shortening the irradiation time to 30 min, a total consumption of **1b** was likewise observed and the process (despite not clean) led to 82% of **3a** (entries 3 and 4). Increasing the concentration of **1b** up to 0.2 M and testing diarylazo sulfide **1p** in place of **1b** did not improve the yield (Table S1, entries 5–7). The reaction was completely suppressed when the vessel was covered by aluminium foil (entry 8).

With the reaction conditions in hand (Table S1, entry 4), we investigated the synthesis of various 2-(arylthio)-1,4-dioxanes by retaining the 4-chlorophenyl group as the hydrogen abstractor (Table 1). The reaction led to the desired compounds **2a–14a** in good to excellent yields, independent of the nature and the position of the substituents on the aromatic ring. Variable amounts of byproducts **2b–14b** derived from photoextrusion were likewise observed. In the preparation of compound **6a**, we tested different diarylazo sulfides as the aryl radical precursors (**1n**, **1o**) with lower efficiency compared to the same reaction carried out starting from **1e**. Substituents such as methyl and cyano in the aromatic ring (compounds **1m** and **1q**) likewise led to a satisfying yield (>70%) of **7a**. The reaction was extended to incorporate THF in the aryl sulfide structure. To this aim, sulfides **15a–21a** were isolated in yields ranging from 40 to 66%. By shifting to a different oxygen heterocycle (2,2-dimethyl-1,3-dioxolane), the yields increased up to 83% in the preparation of **22a–24a** again accompanied by diaryl sulfides **22b–24b**. We then moved to cycloalkane solutions and cyclohexyl

Table 1 Scope of the reaction[a]



Diarylazo sulfides 1	Medium (Solv-H)	% Yield	% Yield	Diarylazo sulfides 1	Medium (Solv-H)	% Yield	% Yield
1a, R^2 = 4-OMe, R^1 = 4-Cl	1,4-Dioxane	**2a**, 86%	**2b**, 12%	**1a**, R^2 = 4-OMe, R^1 = 4-Cl	2,2-Dimethyl-1,3-dioxolane	**22a**, 83%	**22b**, 11%
1b, R^2 = 4-Me, R^1 = 4-Cl	1,4-Dioxane	**3a**, 82%	**3b**, 8%	**1b**, R^2 = 4-Me, R^1 = 4-Cl	2,2-Dimethyl-1,3-dioxolane	**23a**, 75%	**23b**, 10%
1c, R^2 = 4-*t*Bu, R^1 = 4-Cl	1,4-Dioxane	**4a**, 77%	**4b**, 19%	**1e**, R^2 = 4-Cl, R^1 = 4-Cl	2,2-Dimethyl-1,3-dioxolane	**24a**, 73%	**24b**, 10%
1d, R^2 = 4-H, R^1 = 4-Cl	1,4-Dioxane	**5a**, 80%	**5b**, 11%	**1a**, R^2 = 4-OMe, R^1 = 4-Cl	Cyclohexane	**25a**, 64%	**25b**, 17%
1e, R^2 = 4-Cl, R^1 = 4-Cl	1,4-Dioxane	**6a**, 83%	**6b**, 12%	**1b**, R^2 = 4-Me, R^1 = 4-Cl	Cyclohexane	**26a**, 47%	**26b**, 9%
1n, R^2 = 4-Cl, R^1 = 4-CO_2Me	1,4-Dioxane	**6a**, 65%	**6b**, 13%	**1d**, R^2 = 4-H, R^1 = 4-Cl	Cyclohexane	**27a**, 68%	**27b**, 11%
1o, R^2 = 4-Cl, R^1 = 4-NO_2	1,4-Dioxane	**6a**, 27%	**6b**, 12%	**1n**, R^2 = 4-Cl, R^1 = 4-CO_2Me	Cyclohexane	**28a**, 63%	**28b**, 10%
1m, R^2 = 4-Br, R^1 = 4-Me	1,4-Dioxane	**7a**, 77%	**7b**, 13%	**1g**, R^2 = 4-CN, R^1 = 4-Cl	Cyclohexane	**29a**, 41%	**29b**, 12%
1q, R^2 = 4-Br, R^1 = 4-CN	1,4-Dioxane	**7a**, 70%	**7b**, 9%	**1a**, R^2 = 4-OMe, R^1 = 4-Cl	Cyclopentane	**30a**, 72%	**30b**, 12%
1f, R^2 = 4-F, R^1 = 4-Cl	1,4-Dioxane	**8a**, 78%	**8b**, 8%	**1b**, R^2 = 4-Me, R^1 = 4-Cl	Cyclopentane	**31a**, 61%	**31b**, 9%
1g, R^2 = 4-CN, R^1 = 4-Cl	1,4-Dioxane	**9a**, 61%	**9b**, 6%	**1d**, R^2 = 4-H, R^1 = 4-Cl	Cyclopentane	**32a**, 61%	**32b**, 7%
1h, R^2 = 3-Br, R^1 = 4-Cl	1,4-Dioxane	**10a**, 84%	**10b**, 13%	**1e**, R^2 = 4-Cl, R^1 = 4-Cl	Cyclopentane	**33a**, 65%	**33b**, 12%
1i, R^2 = 2-OMe, R^1 = 4-Cl	1,4-Dioxane	**11a**, 71%	**11b**, 5%	**1h**, R^2 = 3-Br, R^1 = 4-Cl	Cyclopentane	**34a**, 63%	**34b**, 10%
1j, R^2 = 2-Br, R^1 = 4-Cl	1,4-Dioxane	**12a**, 75%	**12b**, 13%	**1a**, R^2 = 4-OMe, R^1 = 4-Cl	Cyclopentanone	**35a**, 68%	**35b**, 13%
1k, R^2 = 2,4-Me, R^1 = 4-Cl	1,4-Dioxane	**13a**, 80%	**13b**, 14%	**1b**, R^2 = 4-Me, R^1 = 4-Cl	Cyclopentanone	**36a**, 67%	**36b**, 14%
1l, R^2 = 2,5-Cl, R^1 = 4-Cl	1,4-Dioxane	**14a**, 77%	**14b**, 13%	**1e**, R^2 = 4-Cl, R^1 = 4-Cl	Cyclopentanone	**37a**, 59%	**37b**, 9%
1a, R^2 = 4-OMe, R^1 = 4-Cl	THF	**15a**, 61%	**15b**, 11%	**1b**, R^2 = 4-Me, R^1 = 4-Cl	Cyclohexanone	**38a**, 34%; **38a'**, 23%	**38b**, 10%
1b, R^2 = 4-Me, R^1 = 4-Cl	THF	**16a**, 51%	**16b**, 9%	**1a**, R^2 = 4-OMe, R^1 = 4-Cl	DMF	**39a**, 35%	**39b**, 14%
1d, R^2 = 4-H, R^1 = 4-Cl	THF	**17a**, 59%	**17b**, 8%	**1e**, R^2 = 4-Cl, R^1 = 4-Cl	DMF	**40a**, 44%	**40b**, 17%
1e, R^2 = 4-Cl, R^1 = 4-Cl	THF	**18a**, 56%	**18b**, 9%	**1m**, R^2 = 4-Br, R^1 = 4-Me	DMF	**41a**, 31%	**41b**, 13%
1m, R^2 = 4-Br, R^1 = 4-Me	THF	**19a**, 52%	**19b**, 9%	**1b**, R^2 = 4-Me, R^1 = 4-Cl	Acetone	**42a**, 46%	**42b**, 43%
1g, R^2 = 4-CN, R^1 = 4-Cl	THF	**20a**, 40%	**20b**, 11%	**1b**, R^2 = 4-Me, R^1 = 4-Cl	*tert*-Butyl methyl ether	**43a**, 32%	**43b**, 36%
1i, R^2 = 2-OMe, R^1 = 4-Cl	THF	**21a**, 66%	**21b**, 8%				

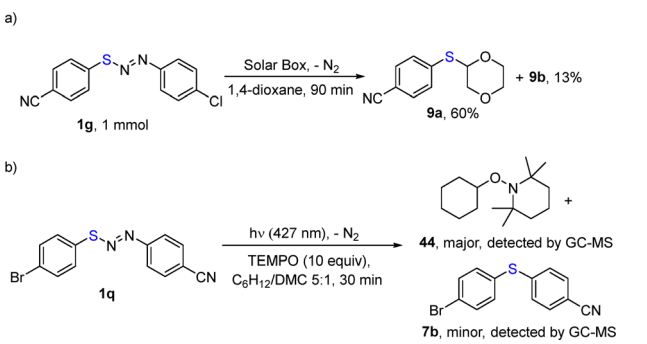
[a] Reaction conditions: An N_2 saturated solution of diarylazo sulfides **1a–1q** (0.05 M), irradiated for 30 min at 427 nm (a 32 W Kessil lamp) in the chosen medium.

(**25a–29a**) and cyclopentyl adducts (**30a–34a**) were formed in variable yields (mostly around 60%). Cycloalkanones were tested, and while cyclopentanone regioselectively afforded α-thioaryl adducts **35a–37a** with a yield in the range of 59–69%, cyclohexanone yielded a mixture of α- and β-adducts (compounds **38a** and **38a′** with the former slightly preferred). In the reaction with DMF, carbamothioates **39a–41a**, valuable building blocks for the synthesis of potentially bioactive derivatives,[29,30] were regioselectively formed but only in moderate yields (Table 1). In acetone and *tert*-butyl methyl ether the solventylation of the starting diarylazo sulfides gave the desired sulfides **42a** and **43a** in less than 43% yield, while diaryl sulfides **39b–43b** were formed in comparable amounts.

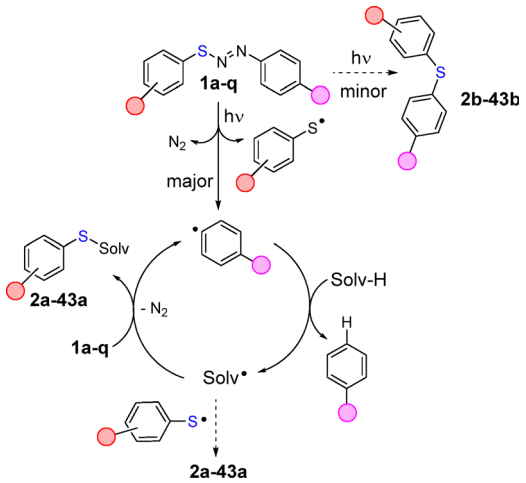
The preparation of **9a** was repeated on a larger scale (1 mmol) under simulated solar light conditions (Scheme 3a and SI, Section S1.4) without any appreciable decrease in yield (60%). To assess the radical nature of the process, we irradiated (427 nm, 30 min) a cyclohexane/DMC 5 : 1 solution of diarylazo sulfide **1q** (0.05 mmol) in the presence of (2,2,6,6-tetramethylpiperidin-1-yl)oxyl (TEMPO, 10 equiv.). GC-MS analysis of the crude mixture showed the presence of 1-(cyclohexyloxy)-2,2,6,6-tetramethylpiperidine **44** as the major product together with a minor amount of diaryl sulfide **7b** (Scheme 3b and SI). Finally, a value >1 of the consumption quantum yield (Φ_{-1} = 9.4) of **1b** in 1,4-dioxane was determined (Section S1.2, SI).

Based on these results, we proposed a mechanism illustrated in Scheme 4. Visible light photolysis of compounds **1a–q** induced photohomolysis of the N–S bond, releasing, upon nitrogen loss, an aryl radical and a thiyl radical. Radical coupling led to the formation of a small amount of adducts **2b–43b** resulting from nitrogen photoextrusion as previously observed in the solid state[14] but still present in solution. However, the photogenerated aryl radical may readily act as a hydrogen atom transfer (HAT) agent from the medium to form Solv•.[31–36]

The radical mechanism of the reaction was confirmed by the intermediacy of Solv• (see the isolation of TEMPO adduct **44** in the reaction with **1q**, Scheme 3b). Interestingly, a better electrophilic aryl radical is no guarantee of reaction success (see the case of **6a** where the strong 4-nitrophenyl radical[37] gave unsatisfactory performance).



Scheme 3 (a) Large-scale synthesis of **9a** under solar simulated conditions. (b) TEMPO trapping experiment.



Scheme 4 The proposed mechanism for the formation of compounds **2a–43a** and **2b–43b**.

Moreover, in the synthesis of **7a**, incorporation of a 4-methylphenyl or a 4-cyanophenyl residue in the starting diarylazo sulfides did not change the overall yield appreciably (Table 1). Quantum yield measurement pointed to a radical chain reaction.

The most likely pathway might be the addition of Solv• to the starting diarylazo sulfides with the concomitant formation of adducts **2a–43a** together with an aryl radical capable of restarting the HAT process. A possible competitive HAT reaction by a thiyl radical on the solvent may not be ruled out but this process is not so efficient when compared to the hydrogen abstracting capabilities of aryl radicals.[26,27,38,39] Minor amounts of aryl sulfides may arise from the radical coupling between Solv• and the thiyl radical.[40]

In the case of cycloalkanones and DMF regioselectivity in hydrogen abstraction (and the subsequent reaction onto **1**) is an issue. In cyclopentanone, only the more labile α-hydrogen[41] is removed (Table 1). This is in contrast with the C–H cleavage induced by other HAT agents where the β-hydrogens are selectively removed.[42,43] In the case of cyclohexanone radicals the α-isomer is more stable (resonantly stabilized) than the β- and γ-isomers.[44] In our case a mixture of α- and β-adducts was isolated in a comparable yield, in contrast to that observed in the hydrogen abstraction from a sulfate radical anion.[43] The selectivity in HAT reactions of aryl radicals is still an open issue.[34] The product distribution observed in the latter cases reflects the lability of C–H bonds in cycloalkanones indicating thermodynamic rather than kinetic control of the abstraction step usually observed in electrophilic hydrogen abstractors.[42] Moreover, the feasibility of the addition of Solv• to **1** may strongly affect the final product distribution.

In DMF, the abstraction of the H-atom of the –CHO group is claimed to be preferred over the methyl group H-atom.[45] This was effectively observed in the abstraction by an aryl radical[46] but not by the decatungstate anion photocatalyst where an exclusive formation of an α-amido radical resulted.[47]

In conclusion, a metal-free C(sp^3)–H bond thiolation of common organic solvents has been proposed herein. The process is based on the visible light activation in diarylazo sulfides, which causes the release of a thiyl radical (a persistent radical) and a reactive aryl radical, which abstracts a hydrogen atom from the reaction medium to form a solvent derived carbon radical prone to give the desired solventylation adduct by reaction with the starting diarylazo sulfide **1**. The reaction is particularly efficient with cyclic ethers (*e.g.* 1,4-dioxane) but modest results arose from open chain solvents (*e.g.* acetone, DMF or *t*-butyl methyl ether). The current protocol does not make use of additives (*e.g.*, TBHP and zinc salts) as in related photocatalyzed preparations.[25,26] The main disadvantage of the method is the poor atom economy (the aryl moiety is lost in the reaction). Nevertheless, this is a nice example where a visible light-induced metal-free forging of an ArS-C bond was devised under very mild conditions.

This work was conceptualized by M. F. and S. P., and experimentation was performed by H. I. M. A. The first draft of the manuscript was prepared by M. F., and the final version was edited and revised by S. P. and M. F.

Conflicts of interest

There are no conflicts to declare.

Data availability

The data supporting this article have been included as part of the supplementary information (SI). Supplementary information: experimental details and NMR spectra. See DOI: https://doi.org/10.1039/d5cc06764b.

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