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# Visible-Light Photoredox-Catalyzed Thioacetalization of Aldehydes Under Metal-Free and Solvent-Free Conditions

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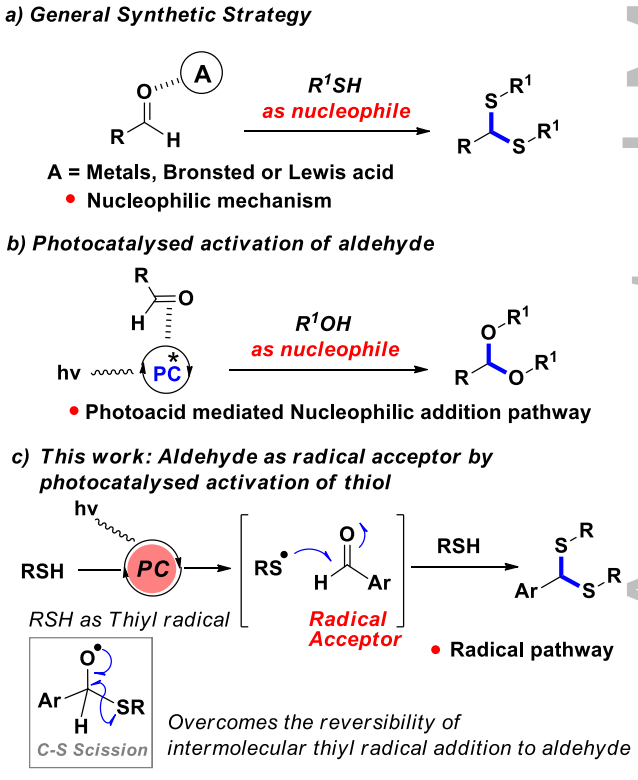
**Abstract.** A first visible-light photoredox-catalyzed thioacetalization of aldehydes under metal-free and solvent-free conditions is described. Under blue LED irradiation, a reactive thiyl radical was initially generated through single-electron oxidation of thiol, which subsequently reacted with aldehydes to afford dithioacetals following a radical mechanism. The reaction proceeded under ambient conditions, and a wide range of acyclic and cyclic dithioacetal derivatives were afforded in good to excellent yields.

**Keywords:** Thioacetalization; Radical reaction; Photocatalyst; Aldehyde; Thiol

## Introduction

Organosulfur compounds are important molecules used for major structural diversification in drug discovery,[1] pharmaceuticals,[2] and material science.[3] These sulfur-containing precursors are used as key intermediates in total synthesis of natural products[4] and diverse synthetic modifications.[5] In particular, dithioacetals act as the prominent protecting group for carbonyl moieties of aldehydes or ketones; they also function as equivalent acyl anions for specific chemical transformations.[6] Dithioacetals have been used as precursors in fluorination,[7a] hydrogenolysis,[7b] alkylation,[7c,d] and olefination[7e]. Recently, dithioacetals were also utilized in C–H or C–S bond activation,[8a,b] hydrodesulfurization,[8c] autoxidation,[9] and dynamic library access of dithioacetals.[10] Various synthetic approaches, including metal-catalyzed reactions,[11] Brønsted or Lewis acid-mediated reactions,[12] solid-supported-linker[13a] or resin-bounded–solid-phase reactions,[13b] and HBA-UV irradiation reactions,[14] can be used for thioacetalization of aldehydes (Scheme 1a); however, these methods can often be toxic and hazardous, have limited substrate scope and side-product–related issues, and require harsh reaction conditions. Therefore, establishing a facile methodology for thioacetalization of aldehydes remains challenging and requires further development. Moreover, visible-light–promoted photoredox catalysis has recently received considerable attention




**Scheme 1.** Reaction design, activation, and pathways

because of its mildness and high functional group tolerance in the development of organic methodologies.[15] It acts as a single-electron transfer (SET) system that prevents undesired reaction

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pathways, to accomplish simple and clean organic transformation processes,[16] and its homogenous nature increases the catalyst efficiency with the lowest catalyst loading in the reaction system.

Visible-light–driven reactions have emerged as versatile methods for C–S bond formation.[17] Lei et al.[18] developed visible-light-promoted acetalization of aldehydes with alcohols through the photoacid-mediated nucleophilic addition pathway (Scheme 1b). We envisioned that photoredox catalysis with uniform blue light penetration may activate thiol through SET to enable radical addition on aldehyde[19] to access dithioacetals. As part of our ongoing progress in the field of C–S bond coupling reaction,[20] herein we report an environment friendly thioacetalization of aldehydes with thiols through visible-light–driven eosin Y-catalyzed C–S bond formation at ambient temperature under metal-free and solvent-free conditions (Scheme 1c).

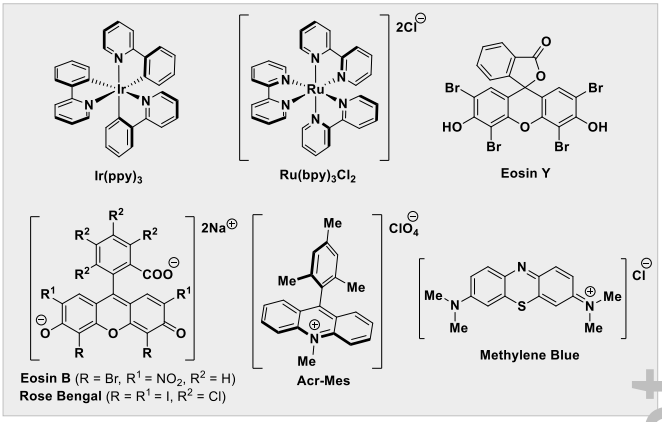
## Results and Discussion

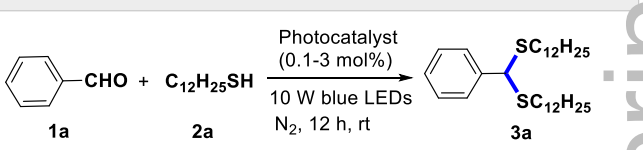
For optimization, benzaldehyde (**1a**) and 1-dodecanethiol (**2a**) were selected as model substrates under solvent-free conditions, and the reaction was initially carried out using 3.0 mol% photoredox catalysts $Ir(ppy)_3$ and $Ru(bpy)_3Cl_2$ with 10-W blue LEDs, which provided the desired product dithioacetal, **3a**, in 58% and 73% yields, respectively (Table 1, entries 1 and 2). Notably, when 0.1 mol% eosin Y was employed as an organic photocatalyst, the product was obtained in 99% yield (Table 1, entry 3). Other organo photocatalysts, such as eosin B, rose bengal, Acr-$Mes^+ClO_4^-$, and methylene blue, were also screened (Table 1, entries 4–7); nevertheless, eosin Y is the best among those organo photocatalysts for thioacetalization of benzaldehyde (Table 1, entry 3).

To study the substrate scope for this notable metal-free, visible-light photoredox-catalyzed thioacetalization, various aldehydes **1a–u** were employed for the reaction with thiol **2a** under optimized reaction conditions (Table 2). 2-Bromobenzaldehyde or 4-bromobenzaldehyde afforded dithioacetals **3b** and **3c** in 85% yield. Similarly, 2-chlorobenzaldehyde or 3-chlorobenzaldehyde afforded **3d** and **3e** in 81% yield. Electron-rich aldehydes, such as 2-methoxybenzaldehyde or 4-methoxybenzaldehyde, were superior to other aldehydes and afforded dithioacetals **3f** and **3g** in 96% yield. Different tolualdehydes reacted well under the optimized reaction conditions to provide the resulting dithioacetals **3h–j** in 76%–99% yields. Electron-deficient aldehydes, namely 2-nitrobenzaldehyde, 3-nitrobenzaldehyde, and 4-nitrobenzaldehyde, also reacted well with thiol **2a** to provide the products **3k–m** in relatively good yields (52%–78%). 3-Formylbenzonitrile also tolerated the reaction conditions well and afforded the product **3n** in 73% yield. Salicylaldehyde reacted well with **2a** to provide **3o** in 99% yield. 3,5-Disubstituted and 2,3,5-trisubstituted benzaldehydes reacted smoothly with **2a** and the corresponding products **3p** and **3q** were obtained in 95% and 74% yields, respectively. Heteroaldehydes were also found to be compatible with the optimized reaction conditions, delivering the products **3r** and **3s** in 77% and 98% yields, respectively. Other aldehydes, such as 1-naphthaldehyde and cinnamaldehyde, reacted with **2a** and provided the products **3t** and **3u** in 99% and 71% yields, respectively.

Next, we screened different thiols **2b–d** for thioacetalization with different aldehydes under optimized reaction conditions (Table 3). We began our studies with the reaction of cyclohexanethiol (**2b**) with benzaldehyde, 2-tolualdehyde, and 4-methoxybenzaldehyde under identical reaction conditions, which provided the corresponding dithioacetals **4a–c** in quantitative yields. 2-Butanethiol (**2c**) also reacted well with benzaldehyde to afford the desired product **4d** in 99% yield. However, thiophenol (**2d**) on reaction with 4-methoxybenzaldehyde could only provide the desired product **4e** in 30% yield.

**Table 1.** Optimization of reaction conditions[a]




| Entry | Photocatalyst | Yield of **3a**[b] |
|---|---|---|
| 1[c] | 3 mol% $Ir(ppy)_3$ | 58 |
| 2[c] | 3 mol% $Ru(bpy)_3Cl_2$ | 73 |
| 3 | ***0.1 mol% Eosin Y*** | ***99*** |
| 4 | 0.1 mol% Eosin B | 95 |
| 5 | 0.1 mol% Rose Bengal | 48 |
| 6 | 0.1 mol% Acr-$Mes^+ClO_4^-$ | 65 |
| 7 | 0.1 mol% Methylene blue | 75 |

[a]Reaction conditions: **1a** (1.0 mmol), **2a** (2.2 mmol), photocatalyst (3 mol% for entries 1 and 2 and 0.1 mol% for entries 3–7), 10-W blue LEDs. [b]Isolated yields. [c]Reaction carried out with **1a** (0.5 mmol) and **2a** (1.1 mmol).

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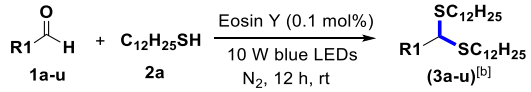
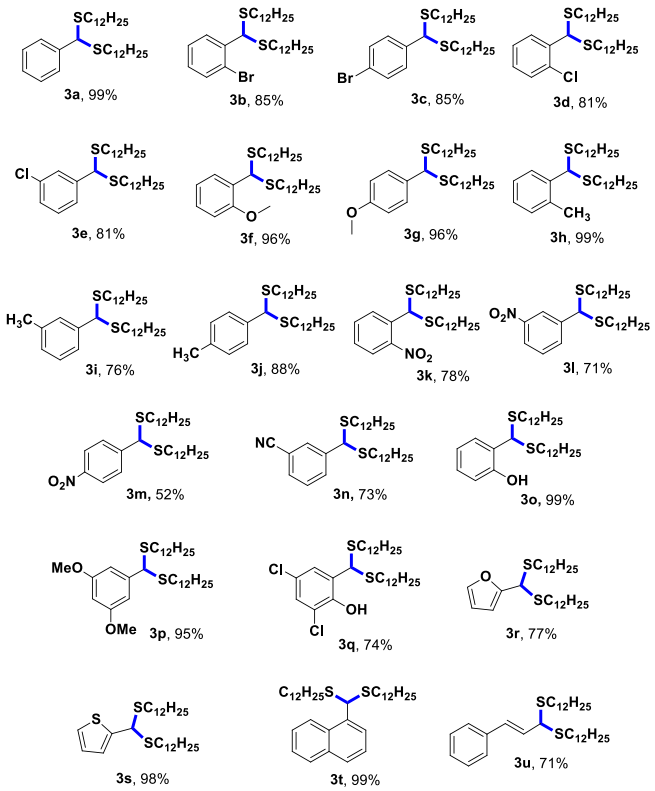
**Table 2.** Scope of aldehydes with **2a**[a]



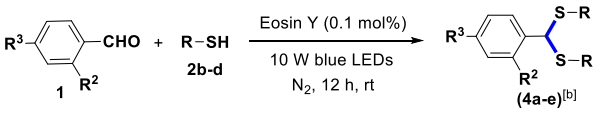



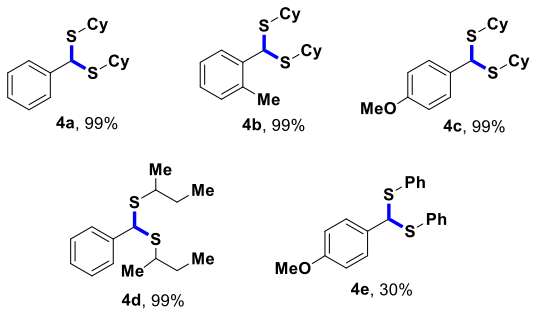

[a]Reaction conditions: **1** (1.0 mmol), **2** (2.2 mmol), eosin Y (0.1 mol%), 10-W blue LEDs. [b]Isolated yields.

Next, dithiols **2e** and **2f** were tested for thioacetalization with different aldehydes under optimized reaction conditions. They provided the cyclic dithioacetal products **5a–5d** in 35%–81% yields (Table 4). However, alkyl aldehydes do not work well in the present system; for example, when butyraldehyde and thiophenol were exposed to the standard reaction conditions, no product was detected. On the other hand, only a trace amount of product was observed when the reaction was performed by using butyraldehyde and cyclohexanethiol as the starting materials.

We also investigated the reaction efficiency at a larger scale by conducting a 10-g scale reaction between **1a** and **2a** in the presence of 0.1 mol% eosin Y under standard reaction conditions. The desired product **3a** was obtained in excellent yield (Scheme 2).

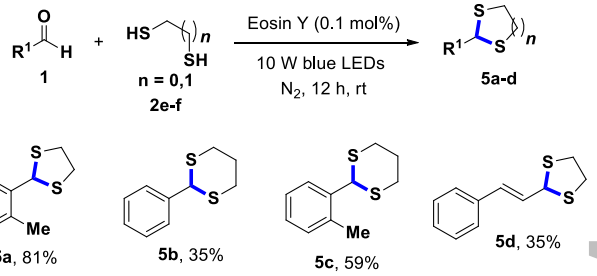
**Table 3.** Scope of various thiols with aromatic aldehydes[a]



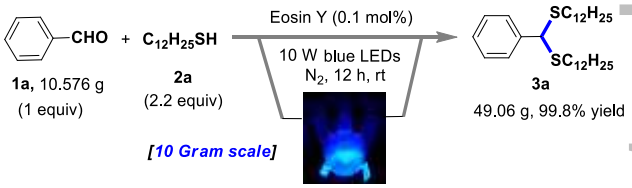




[a]Reaction conditions: **1** (1.0 mmol), **2** (2.2 mmol), eosin Y (0.1 mol%), 10-W blue LEDs. [b]Isolated yields.

**Table 4.** Synthesis of cyclic dithioacetals **5**[a],[b]



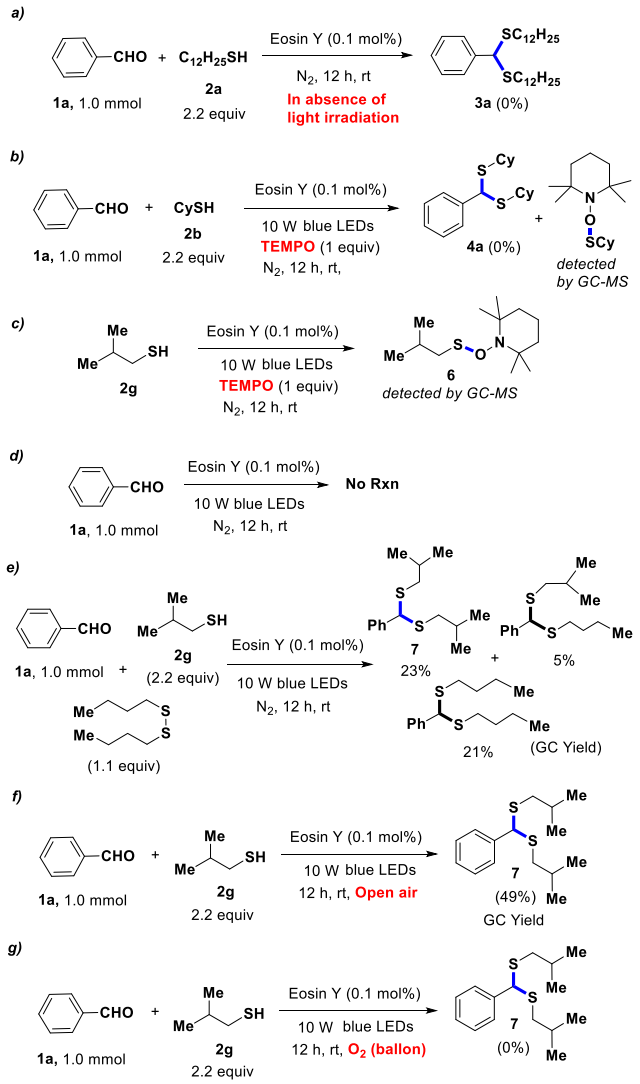

[a]Reaction conditions: **1** (1.0 mmol), **2** (1.1 mmol), eosin Y (0.1 mol%), 10-W blue LEDs. [b]Isolated yields.




**Scheme 2.** Ten-gram scale synthesis of dithioacetal

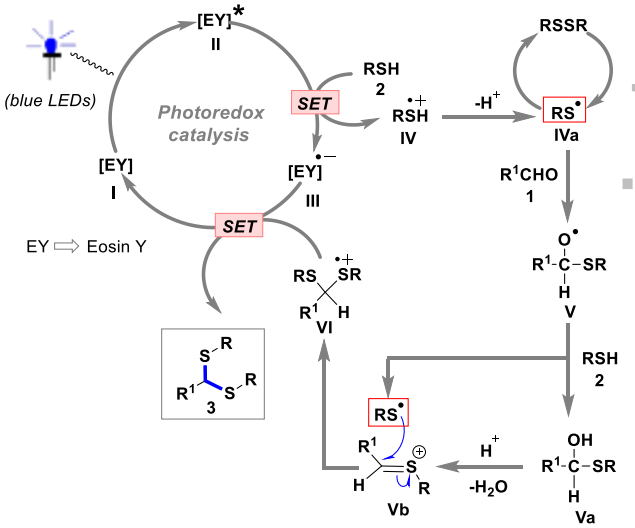
To propose a plausible reaction mechanism of this visible-light photoredox-catalyzed thioacetalization, various control experiments were performed (Scheme 3). In the absence of blue-light irradiation, **3a** was not formed. Therefore, light radiation is necessary to carry out this transformation (Scheme 3a). In the presence of one equivalent of the radical inhibitor TEMPO, the reaction failed to deliver the product **4a,** indicating that the reaction proceeds through a radical pathway (Scheme 3b). This experiment also ruled out the hypothesis of a photoacid-mediated pathway and acid-catalyzed pathway (released from the oxidation of thiol to a thiyl radical). In addition, on reaction with thiol **2g** under standard reaction conditions in the presence of TEMPO, the thiyl radical adduct, 1-((isobutylthio)oxy)-2,2,6,6-tetramethyl piperidine **6**,

was formed, which was detected through GC–MS (Scheme 3c). Moreover, **1a** remained intact, without any reaction in the absence of thiol, thus indicating that only a thiyl radical from thiol triggered the aldehyde (Scheme 3d). To understand disulfide participation, a crossover experiment between **1a** and **2g** was conducted in the presence of *n*-butyl disulfide under standard reaction conditions. Subsequently, crossover products were carried out. This result demonstrates that disulfide may also participate in this transformation (Scheme 3e). In an open-air atmosphere, **7** was obtained in 49% yield, whereas in an $O_2$ atmosphere, **7** did not form. These results implied that a nitrogen atmosphere is essential for this transformation (Schemes 3f and 3g). These reaction mechanisms were studied further through EPR experiments (see SI).




**Scheme 3.** Control experiments

On the basis of recent literature[16b,21] and the results of the aforementioned control experiments, we propose a plausible mechanism for this transformation as follows: Thiol **2** initially interacts with the excited organophotocatalyst **II** and undergoes a single-electron oxidation process to form species **IV**. The generated thiyl radical **IVa** reacts with **1** to form an intermediate **V**. Thiol **2** further reacts with **V**, which delivers a thiyl radical and **Va**. **Va** subsequently affords **Vb** after the elimination of its water molecule. The thiyl radical then reacts with **Vb** to afford **VI**. **VI** then favors a single-electron reduction process from the reduced organophotocatalyst **III**, leading to the formation of the dithioacetal product **3** (Scheme 4).




**Scheme 4.** Plausible mechanism

## Conclusion

In summary, a mild and efficient protocol for thioacetalization of aldehydes using a visible-light–promoted metal-free photoredox catalyst under solvent-free conditions has been developed. A wide range of aldehydes containing aromatic, heteroaromatic, and α,β-unsaturated aromatic rings are all well tolerated under the reaction conditions employed. The protocol provides the expedient synthesis of cyclic dithioacetal derivatives with five- or six-membered rings. The reaction is amenable to a 10-g scale synthesis. The control experimental studies validated the insight of the reaction mechanism. The eminent features of the present protocol include the simple and convenient operation, ecofriendliness, low catalyst loading, high functional group tolerance, ease of scalability, and practicality.

## Experimental Section

**General information:**

Merck silica gel 60 (230–400 mesh) were used to purify the compounds by column chromatography. All commercial chemicals were used as such. GC-MS analyses were undertaken on Agilent Technologies 5977A GC equipped with Agilent 7890B MS. Varian Unity Inova-600 or a Varian Mercury-400 NMR instrument were used to

record NMR by using $CDCl_3$ solvent, and multiplicity were determined as singlet s, doublet d, triplet t, double of doublet dd, multiplet m and broad brs. Chemical shifts and coupling constant values are determined in parts per million (ppm) and in hertz (Hz). High-resolution mass spectra (HRMS) were analyzed on a Jeol JMS-HX 110 spectrometer by the services provided at the National Chung Hsing University.

**General procedure for the synthesis of dithioacetals:**

Aldehyde **1**, thiol **2** and eosin Y (0.1 mol%) were added to the Schlenk tube containing a Teflon-coated magnetic stir bar and was purged by evacuating the tube and back filled with nitrogen. The reaction mixture was kept for stirring under 10 W blue LEDs irradiation at room temperature for 12 hours. After completion of the reaction, the crude reaction mixture was purified by silicagel column chromatography to afford the pure dithioacetal product.

**Benzaldehyde didodecyldithioacetal (3a):**[9] Following the general procedure, using benzaldehyde (0.106g, 1.0 mmol), 1-dodecanethiol (0.445g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **3a** as the yellow oil (0.485 g, 99% yield). $^1$H NMR (400 MHz, $CDCl_3$): δ 7.43 (d, *J* = 8.0 Hz, 2H), 7.31 (t, *J* = 8.0 Hz, 2H), 7.24 (t, *J* = 8.0 Hz, 1H), 4.87 (s, 1H), 2.60 – 2.46 (m, 4H), 1.57 – 1.50 (m, 4H), 1.24 (brs, 36H), 0.88 (t, *J* = 8.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ 140.55, 128.37, 127.63, 127.59, 53.07, 32.16, 31.88, 29.63, 29.61, 29.56, 29.46, 29.33, 29.15, 29.08, 28.83, 22.66, 14.10. The above similar reaction procedure was followed for 10-gram scale reaction, providing 99.8% yield.

**2-Bromobenzaldehyde didodecyldithioacetal (3b):** Following the general procedure, using 2-bromobenzaldehyde (0.185g, 1.0 mmol), 1-dodecanethiol (0.445g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **3b** as the colorless oil (0.485 g, 85% yield). $^1$H NMR (400 MHz, $CDCl_3$): δ 7.76 (d, *J* = 8.0 Hz, 1H), 7.48 (d, *J* = 8.0 Hz, 1H), 7.28 (t, *J* = 8.0 Hz, 1H), 7.06 (t, *J* = 8.0 Hz, 1H), 5.41 (s, 1H), 2.64 – 2.46 (m, 4H), 1.61 – 1.53 (m, 4H), 1.25 (brs, 36H), 0.88 (t, *J* = 8.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 139.87, 132.36, 129.74, 128.79, 127.67, 123.20, 51.26, 32.28, 31.85, 29.61, 29.59, 29.54, 29.43, 29.31, 29.13, 29.10, 28.70, 22.62, 14.05. HRMS (EI) calcd for $C_{31}H_{55}BrS_2$ $[M]^+$ 570.2929, found 570.2934.

**4-Bromobenzaldehyde didodecyldithioacetal (3c):** Following the general procedure, using 4-bromobenzaldehyde (0.185g, 1.0 mmol), 1-dodecanethiol (0.445g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **3c** yellow oil (0.484 g, 85% yield). $^1$H NMR (400 MHz, $CDCl_3$): δ 7.42 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 4.82 (s, 1H), 2.59 – 2.44 (m, 4H), 1.56 – 1.49 (m, 4H), 1.25 (brs, 36H), 0.88 (t, *J* = 8.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ 139.67, 131.39, 129.26, 121.31, 52.39, 32.07, 31.85, 29.61, 29.59, 29.54, 29.43, 29.31, 29.11, 28.99, 28.78, 22.63, 14.06. HRMS (EI) calcd for $C_{31}H_{55}BrS_2$ $[M]^+$ 570.2929, found 570.2938.

**2-Chlorobenzaldehyde didodecyldithioacetal (3d):** Following the general procedure, using 2-chlorobenzaldehyde (0.140. g, 1.0 mmol), 1-dodecanethiol (0.445g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **3d** as the colorless oil (0.426 g, 81% yield). $^1$H NMR (400 MHz, $CDCl_3$): *δ* 7.75 (d, *J* = 8.0 Hz, 1H), 7.31 (d, *J* = 8.0 Hz, 1H), 7.25 (t, *J* = 8.0 Hz, 1H), 7.15 (t, *J* = 8.0 Hz, 1H), 5.43 (s, 1H), 2.64 – 2.47 (m, 4H), 1.60 – 1.53 (m, 4H), 1.25 (brs, 36H), 0.88 (t, *J* = 8.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): *δ* 138.27, 132.44, 129.52, 129.15, 128.53, 127.10, 48.42, 32.29, 31.87, 29.62, 29.60, 29.55, 29.44, 29.32, 29.11, 28.74, 22.64, 14.07. HRMS (EI) calcd for $C_{31}H_{55}ClS_2$ $[M]^+$ 526.3434, found 526.3440.

**3-Chlorobenzaldehyde didodecyldithioacetal (3e):** Following the general procedure, using 3-chlorobenzaldehyde (0.140 g, 1.0 mmol), 1-dodecanethiol (0.445g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **3e** white oil (0.427 g, 81% yield). $^1$H NMR (400 MHz, $CDCl_3$): *δ* 7.44 (s, 1H), 7.32 (d, *J* = 8.0 Hz, 1H), 7.24 – 7.21 (m, 2H), 4.81 (s, 1H), 2.60 – 2.46 (m, 4H), 1.55 – 1.52 (m, 4H), 1.25 (brs, 36H), 0.88 (t, *J* = 8.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): *δ* 142.77, 134.18, 129.52, 127.74, 125.79, 52.53, 32.15, 31.87, 29.60, 29.55, 29.45, 29.32, 29.13, 29.01, 28.78, 22.64, 14.06. HRMS (EI) calcd for $C_{31}H_{55}ClS_2$ $[M]^+$ 526.3434, found 526.3426.

**2-Methoxybenzaldehyde didodecyldithioacetal (3f):** Following the general procedure, using *o*-anisaldehyde (0.136 g, 1.0 mmol), 1-dodecanethiol (0.445g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **3f** as the colorless oil (0.502 g, 96% yield). $^1$H NMR (400 MHz, $CDCl_3$): *δ* 7.60 (dd, *J* = 8.0, 4.0 Hz, 1H), 7.18 (t, *J* = 8.0, 1H), 6.93 (t, *J* = 8.0 Hz, 1H), 6.82 (d, *J* = 8.0 Hz, 1H), 5.46 (s, 1H), 3.81 (s, 3H), 2.62 – 2.45 (m, 4H), 1.58 – 1.51 (m, 4H), 1.25 (brs, 36H), 0.88 (t, *J* = 8.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): *δ* 155.47, 128.82, 128.44, 128.28, 120.58, 110.21, 55.34, 44.87, 31.95, 31.80, 29.56, 29.54, 29.50, 29.40, 29.25, 29.10, 29.05, 28.75, 22.56, 13.98. HRMS (EI) calcd for $C_{32}H_{58}OS_2$ $[M]^+$ 522.3929, found 522.3923.

**4-Methoxybenzaldehyde didodecyldithioacetal (3g):** Following the general procedure, using *p*-anisaldehyde (0.136 g, 1.0 mmol), 1-dodecanethiol (0.445g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **3g** white solid (0.502 g, 96% yield). m.p. 39-41°C; $^1$H NMR (400 MHz, $CDCl_3$): *δ* 7.35 (d, *J* = 12.0 Hz, 2H), 6.83 (d, *J* = 8.0 Hz, 2H), 4.85 (s, 1H), 3.76 (s, 3H), 2.59 – 2.44 (m, 4H), 1.57 – 1.50 (m, 4H), 1.25 (brs, 36H), 0.88 (t, *J* = 8.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): *δ* 158.88, 132.46, 128.67, 113.57, 54.99, 52.45, 32.07, 31.83, 29.58, 29.57, 29.52, 29.42, 29.28, 29.11, 29.06, 28.79, 22.60,

14.02. HRMS (EI) calcd for $C_{32}H_{58}OS_2$ $[M]^+$ 522.3929, found 522.3931.

**2-Methylbenzaldehyde didodecyldithioacetal (3h):** Following the general procedure, using *o*-tolualdehyde (0.120 g, 1.0 mmol), 1-dodecanethiol (0.445g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **3h** as the colorless oil (0.502 g, 99% yield). $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 7.65 (d, $J$ = 8.0 Hz, 1H), 7.17 – 7.09 (m, 3H), 5.11 (s, 1H), 2.63 – 2.43 (m, 4H), 2.39 (s, 3H), 1.57 –1.49 (m, 4H), 1.25 (brs, 36H), 0.88 (t, $J$ = 8.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 137.82, 134.86, 130.25, 127.59, 127.25, 126.02, 49.54, 31.86, 29.61, 29.60, 29.55, 29.45, 29.32, 29.13, 29.10, 28.81, 22.62, 19.11, 14.03. HRMS (EI) calcd for $C_{32}H_{58}S_2$ $[M]^+$ 506.3980, found 506.3975.

**3-Methylbenzaldehyde didodecyldithioacetal (3i):** Following the general procedure, using *m*-tolualdehyde (0.120 g, 1.0 mmol), 1-dodecanethiol (0.445g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **3i** as the colorless oil (0.385 g, 76% yield). $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 7.24 – 7.15 (m, 3H), 7.04 (d, $J$ = 8.0 Hz, 1H), 4.83 (s, 1H), 2.60 – 2.46 (m, 4H), 2.33 (s, 3H), 1.57 – 1.50 (m, 4H), 1.25 (brs, 36H), 0.88 (t, $J$ = 8.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 140.42, 137.92, 128.39, 128.12, 124.63, 53.08, 32.13, 31.86, 29.60, 29.59, 29.54, 29.44, 29.31, 29.13, 29.06, 28.80, 22.62, 21.30, 14.04. HRMS (EI) calcd for $C_{32}H_{58}S_2$ $[M]^+$ 506.3980, found 506.3987.

**4-Methylbenzaldehyde didodecyldithioacetal (3j):** Following the general procedure, using *p*-tolualdehyde (0.120 g, 1.0 mmol), 1-dodecanethiol (0.445g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **3j** colorless oil (0.446 g, 88% yield). $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 7.31 (d, $J$ = 8.0 Hz, 2H), 7.09 (d, $J$ = 8.0 Hz, 2H), 4.84 (s, 1H), 2.59 – 2.45 (m, 4H), 2.31 (s, 3H), 1.57 – 1.50 (m, 4H), 1.25 (brs, 36H), 0.88 (t, $J$ = 8.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 137.48, 137.13, 128.94, 127.42, 52.79, 32.05, 31.85, 29.60, 29.59, 29.54, 29.43, 29.30, 29.12, 29.05, 28.79, 22.62, 20.99, 14.03. HRMS (EI) calcd for $C_{32}H_{58}S_2$ $[M]^+$ 506.3980, found 506.3982.

**2-Nitrobenzaldehyde didodecyldithioacetal (3k):** Following the general procedure, using 2-nitrobenzaldehyde (0.151 g, 1.0 mmol), 1-dodecanethiol (0.445g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **3k** as the yellow oil (0.419 g, 78% yield). $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 7.99 (d, $J$ = 8.0 Hz, 1H), 7.81 (d, $J$ = 8.0 Hz, 1H), 7.59 (t, $J$ = 8.0 Hz, 1H), 7.37 (t, $J$ = 8.0 Hz, 1H), 5.72 (s, 1H), 2.66 – 2.51 (m, 4H), 1.54 (d, $J$ = 8.0 Hz, 4H), 1.25 (brs, 36H), 0.88 (t, $J$ = 8.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 147.90, 135.99, 132.92, 130.42, 128.10, 124.06, 46.67, 32.67, 31.83, 29.56, 29.50, 29.39, 29.28, 29.04, 28.97, 28.68, 22.60, 14.02. HRMS (EI) calcd for $C_{31}H_{55}NO_2S_2$ $[M]^+$ 537.3674, found 537.3676.

**3-Nitrobenzaldehyde didodecyldithioacetal (3l):** Following the general procedure, using 3-nitrobenzaldehyde (0.151 g, 1.0 mmol), 1-dodecanethiol (0.445g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **3l** yellow oil (0.381 g, 71% yield). $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 8.32 (s, 1H), 8.12 (d, $J$ = 8.0 Hz, 1H), 7.83 (d, $J$ = 8.0 Hz, 1H), 7.51 (t, $J$ = 8.0 Hz, 1H), 4.95 (s, 1H), 2.64 – 2.48 (m, 4H), 1.58 – 1.52 (m, 4H), 1.25 (brs, 36H), 0.88 (t, $J$ = 8.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 148.04, 143.15, 133.72, 129.32, 122.56, 52.32, 32.20, 31.82, 29.56, 29.55, 29.49, 29.39, 29.27, 29.06, 28.94, 28.72, 22.59, 14.01. HRMS (EI) calcd for $C_{31}H_{55}NO_2S_2$ $[M]^+$ 537.3674, found 537.3666.

**4-Nitrobenzaldehyde didodecyldithioacetal (3m).** Following the general procedure, using 4-nitrobenzaldehyde (0.151 g, 1.0 mmol), 1-dodecanethiol (0.445g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **3m** white solid (0.279 g, 52% yield). m.p. 44-46°C; $^1$H NMR (400 MHz, $CDCl_3$): $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 8.19 (d, $J$ = 8.0 Hz, 2H), 7.62 (d, $J$ = 12.0 Hz, 2H), 4.91 (s, 1H), 2.63 – 2.47 (m, 4H), 1.59 – 1.51 (m, 4H), 1.25 (brs, 36H), 0.88 (t, $J$ = 8.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 148.23, 147.04, 128.46, 123.62, 52.34, 32.18, 31.82, 29.55, 29.49, 29.39, 29.26, 29.06, 28.94, 28.73, 22.59, 14.01. HRMS (EI) calcd for $C_{31}H_{55}NO_2S_2$ $[M]^+$ 537.3674, found 537.3677.

**3-Cyanobenzaldehyde didodecyldithioacetal (3n).** Following the general procedure, using 3-formylbenzonitrile (0.131 g, 1.0 mmol), 1-dodecanethiol (0.445g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **3n** colorless oil (0.378g, 73% yield). $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 7.75 (s, 1H), 7.71 (d, $J$ = 8.0 Hz, 1H), 7.55 (d, $J$ = 4.0 Hz, 1H), 7.43 (t, $J$ = 8.0 Hz, 1H), 4.87 (s, 1H), 2.62 – 2.46 (m, 4H), 1.58 – 1.51 (m, 4H), 1.25 (brs, 36H), 0.88 (t, $J$ = 8.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 142.43, 132.05, 131.12, 129.15, 118.31, 112.39, 52.18, 32.11, 31.77, 29.52, 29.50, 29.45, 29.34, 29.22, 29.02, 28.88, 28.68, 22.55, 13.99. HRMS (EI) calcd for $C_{32}H_{55}NS_2$ $[M]^+$ 517.3776, found 517.3783.

**2-Hydroxybenzaldehyde didodecyldithioacetal (3o):** Following the general procedure, using salicylaldehyde (0.122 g, 1.0 mmol), 1-dodecanethiol (0.445g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **3o** as the colorless oil (0.504g, 99% yield). $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 7.20 – 7.17 (m, 2H), 7.04 (s, 1H), 6.89 – 6.83 (m, 2H), 5.08 (s, 1H), 2.60 – 2.48 (m, 4H), 1.59 – 1.51 (m, 4H), 1.24 (brs, 36H), 0.88 (t, $J$ = 8.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 154.62, 129.32, 129.21, 123.65, 120.07, 117.33, 109.81, 49.58, 32.22, 31.81, 29.55, 29.49, 29.37, 29.26, 29.02, 28.92, 28.66,

22.58, 13.99. HRMS (EI) calcd for $C_{31}H_{56}OS_2$ $[M]^+$ 508.3773, found 508.3769.

**3,5-Dimethoxybenzaldehyde didodecyldithioacetal (3p):** Following the general procedure, using 3,5-dimethoxybenzaldehyde (0.166 g, 1.0 mmol), 1-dodecanethiol (0.445g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **3p** colorless oil (0.525g, 95% yield). $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 6.61 (d, $J$ = 4.0 Hz, 2H), 6.34 (s, 1H), 4.78 (s, 1H), 3.76 (s, 6H), 2.61 – 2.48 (m, 4H), 1.58 – 1.51 (m, 4H), 1.25 (brs, 36H), 0.88 (t, $J$ = 8.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 160.55, 142.83, 105.46, 99.63, 55.07, 53.26, 32.22, 31.81, 29.56, 29.54, 29.50, 29.41, 29.26, 29.10, 29.02, 28.76, 22.57, 13.99. HRMS (EI) calcd for $C_{33}H_{60}O_2S_2$ $[M]^+$ 552.4035, found 552.4033.

**3,5-Dichloro-2-hydroxybenzaldehyde didodecyldithioacetal (3q):** Following the general procedure, using 3,5-Dichlorobenzaldehyde (0.175 g, 1.0 mmol), 1-dodecanethiol (0.445g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **3q** as the colorless oil (0.428g, 74% yield). $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 7.40 (d, $J$ = 4.0 Hz, 1H), 7.26 (d, $J$ = 4.0 Hz, 1H), 6.36 (s, 1H), 5.21 (s, 1H), 2.64 – 2.49 (m, 4H), 1.61 – 1.53 (m, 4H), 1.25 (brs, 36H), 0.88 (t, $J$ = 8.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 147.50, 128.84, 127.93, 127.60, 125.30, 121.08, 46.71, 39.03, 31.85, 29.58, 29.53, 29.42, 29.30, 22.61, 14.03. HRMS (EI) calcd for $C_{31}H_{54}Cl_2OS_2$ $[M]^+$ 576.2993, found 576.2995.

**2-Furaldehyde didodecyldithioacetal (3r):** Following the general procedure, using furfural (0.096 g, 1.0 mmol), 1-dodecanethiol (0.445g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **3r** as the colorless oil (0.371g, 77% yield). $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 7.34 (s, 1H), 6.32 – 6.28 (m, 2H), 4.98 (s, 1H), 2.68 – 2.51 (m, 4H), 1.56 – 1.50 (m, 4H), 1.26 (brs, 36H), 0.88 (t, $J$ = 8.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 152.27, 141.82, 110.14, 107.37, 45.80, 31.81, 31.24, 29.56, 29.54, 29.50, 29.40, 29.26, 29.09, 29.01, 28.80, 22.58, 13.99. HRMS (EI) calcd for $C_{29}H_{54}OS_2$ $[M]^+$ 482.3616, found 482.3617.

**2-Thiophenecarboxaldehyde didodecyldithioacetal (3s):** Following the general procedure, using 2-Thiophenecarboxaldehyde (0.112 g, 1.0 mmol), 1-dodecanethiol (0.445g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **3s** as the colorless oil (0.489g, 98% yield). $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 7.18 (d, $J$ = 4.0, 1H), 7.06 (s, 1H), 6.88 – 6.86 (m, 1H), 5.17 (s, 1H), 2.69 – 2.52 (m, 4H), 1.60 – 1.53 (m, 4H), 1.25 (brs, 36H), 0.88 (t, $J$ = 8.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 145.12, 126.10, 125.43, 125.05, 48.30, 31.83, 29.58, 29.56, 29.52, 29.42, 29.28, 29.11, 28.94, 28.83, 22.60, 14.02. HRMS (EI) calcd for $C_{29}H_{54}S_3$ $[M]^+$ 498.3388, found 498.3379.

**1-Naphthaldehyde didodecyldithioacetal (3t):** Following the general procedure, using 1-Naphthaldehyde (0.156 g, 1.0 mmol), 1-dodecanethiol (0.445g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **3t** as the colorless oil (0.537g, 99% yield). $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 8.24 (bs, 1H), 7.80 – 7.69 (m, 3H), 7.49 (t, $J$ = 8.0 Hz, 1H), 7.43 – 7.36 (m, 2H), 5.67 (bs, 1H), 2.66 – 2.47 (m, 4H), 1.57 – 1.49 (m, 4H), 1.25 (brs, 36H), 0.87 (t, $J$ = 8.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 135.15, 133.93, 130.39, 128.78, 128.33, 125.87, 125.69, 125.49, 124.90, 123.16, 32.34, 31.86, 29.60, 29.59, 29.53, 29.42, 29.31, 29.11, 29.02, 28.80, 22.63, 14.05. HRMS (EI) calcd for $C_{35}H_{58}S_2$ $[M]^+$ 542.3980, found 542.3981.

**Cinnamaldehyde didodecyldithioacetal (3u):**[22a] Following the general procedure, using cinnamaldehyde (0.132g, 1.0 mmol), 1-dodecanethiol (0.445g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **3u** as the yellow oil (0.367g, 71% yield). $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 7.37 (d, $J$ = 8.0 Hz, 2H), 7.29 (t, $J$ = 8.0 Hz, 2H), 7.21 (t, $J$ = 8.0 Hz, 1H), 6.51 (d, $J$ = 16.0 Hz, 1H), 6.19 – 6.06 (m, 1H), 4.47 (d, $J$ = 8.0 Hz, 1H), 2.67 – 2.56 (m, 4H), 1.63 – 1.56 (m, 4H), 1.25 (brs, 36H), 0.88 (t, $J$ = 8.0 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 136.03, 130.55, 128.41, 128.04, 127.65, 127.56, 126.39, 51.25, 31.84, 31.10, 29.57, 29.53, 29.44, 29.33, 29.29, 29.15, 28.84, 22.61, 14.04. HRMS (EI) calcd for $C_{33}H_{58}S_2$ $[M]^+$ 518.3980, found 518.3989.

**Benzaldehyde dicyclohexyldithioacetal (4a):**[22b] Following the general procedure, using benzaldehyde (0.106g, 1.0 mmol), cyclohexanethiol (0.256 g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **4a** as the yellow oil (0.318 g, 99% yield). $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 7.45 (d, $J$ = 8.0 Hz, 2H), 7.28 (t, $J$ = 8.0 Hz, 2H), 7.20 (t, $J$ = 8.0 Hz, 1H), 5.00 (s, 1H), 2.76 – 2.69 (m, 2H), 1.93 – 1.86 (m, 4H), 1.73 – 1.69 (m, 4H), 1.55 (s, 2H), 1.36 – 1.24 (m, 10H). $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 141.06, 128.14, 127.41, 127.30, 126.81, 49.52, 43.88, 33.06, 25.56. HRMS (EI) calcd for $C_{19}H_{28}S_2$ $[M]^+$ 320.1632, found 320.1629.

**2-Methylbenzaldehyde dicyclohexyldithioacetal (4b):** Following the general procedure, using *o*-tolualdehyde (0.120 g, 1.0 mmol), cyclohexanethiol (0.256 g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **4b** as the colorless oil (0.331 g, 99% yield). $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 7.76 (d, $J$ = 8.0 Hz, 1H), 7.19 – 7.08 (m, 3H), 5.24 (s, 1H), 2.77 (t, $J$ = 8.0 Hz, 2H), 2.41 (s, 3H), 1.94 – 1.84 (m, 4H), 1.70 (s, 4H), 1.55 (s, 2H), 1.36 – 1.24 (m, 10H). $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 138.37, 134.51, 130.09, 127.93, 127.16, 126.13, 46.11, 43.93, 33.26, 33.17, 25.64, 19.03. HRMS (EI) calcd for $C_{20}H_{30}S_2$ $[M]^+$ 334.1789, found 334.1791.

**4-Methoxylbenzaldehyde dicyclohexyldithioacetal (4c):** Following the general procedure, using *p*-anisaldehyde

(0.136 g, 1.0 mmol), cyclohexanethiol (0.256 g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **4c** as the colorless oil (0.347 g, 99% yield). $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 7.38 (d, $J$ = 8.0 Hz, 2H), 6.82 (d, $J$ = 8.0 Hz, 2H), 4.99 (s, 1H), 3.76 (s, 3H), 2.71 (s, 2H), 1.89 (s, 4H), 1.70 (s, 4H), 1.54 (s, 2H), 1.41 – 1.23 (m, 10H). $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 158.67, 132.99, 128.55, 113.50, 54.95, 48.89, 43.84, 33.08, 25.60. HRMS (EI) calcd for $C_{20}H_{30}OS_2$ $[M]^+$ 350.1738, found 350.1740.

**(Phenylmethylene)bis(sec-butylsulfane) (4d):**[9] Following the general procedure, using benzaldehyde (0.106g, 1.0 mmol), 2-butanethiol (0.198 g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **4d** as the colorless oil (0.266 g, 99% yield). $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 7.47 (d, $J$ = 4.0 Hz, 2H), 7.29 (t, $J$ = 8.0 Hz, 2H), 7.22 (t, $J$ = 8.0 Hz, 1H), 4.94 (s, 1H), 2.86 – 2.64 (m, 2H), 1.58 – 1.46 (m, 4H), 1.21 (t, $J$ = 8.0 Hz, 6H), 0.95 – 0.88 (m, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 140.98, 128.27, 127.63, 127.48, 50.50, 42.30, 29.37, 20.51, 11.02. HRMS (EI) calcd for $C_{15}H_{24}S_2$ $[M]^+$ 268.1319, found 268.1323.

**4-Anisaldehyde diphenyldithioacetal (4e):**[14] Following the general procedure, using *p*-anisaldehyde (0.136 g, 1.0 mmol), thiophenol (0.242 g, 2.2 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **4e** as the colorless oil (0.101 g, 30% yield). $^1$H NMR (400 MHz, $CDCl_3$): δ 7.35 – 7.21 (m, 12H), 6.79 (d, $J$ = 8.0 Hz, 2H), 5.42 (s, 1H), 3.76 (s, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ 159.21, 134.68, 132.31, 131.59, 129.04, 128.75, 127.61, 113.77, 59.68, 55.22.

**2-(*o*-Tolyl)-1,3-dithiolane (5a):**[22c] Following the general procedure, using *o*-tolualdehyde (0.120g, 1.0 mmol), 1,2-ethanedithiol (0.103g, 1.1 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **5a** as the yellow oil ( 0.159g, 81% yield). $^1$H NMR (400 MHz, $CDCl_3$): δ 7.79 (d, $J$ = 8.0 Hz, 1H), 7.26 – 7.12 (m, 3H), 5.88 (s, 1H), 3.54 – 3.32 (m, 4H), 2.43 (s, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ 137.83, 135.55, 130.23, 127.58, 127.50, 126.26, 52.75, 39.64, 19.56.

**2-Phenyl-1,3-dithiane (5b):**[9] Following the general procedure, using benzaldehyde (0.106g, 1.0 mmol), 1,3-propanedithiol (0.119g, 1.1 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **5b** white solid (0.069 g, 35% yield). m.p. 60-62°C; $^1$H NMR (400 MHz, $CDCl_3$): δ 7.48 – 7.46 (m, 2H), 7.35 – 7.25 (m, 3H), 5.17 (s, 1H), 3.10 – 3.03 (m, 2H), 2.93 – 2.88 (m, 2H), 2.20 – 2.14 (m, 1H), 1.99 – 1.88 (m, 1H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ 139.00, 128.58, 128.28, 127.61, 51.33, 31.95, 24.97.

**2-(*o*-Tolyl)-1,3-dithiane (5c):**[9] Following the general procedure, using *o*-tolualdehyde (0.120g, 1.0 mmol), 1,3-propanedithiol (0.119g, 1.1 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **5c** white solid (0.124 g, 59% yield). m.p. 80-82 °C; $^1$H NMR (400 MHz, $CDCl_3$): δ 7.59 (d, $J$ = 8.0 Hz, 1H), 7.23 – 7.14 (m, 3H), 5.32 (s, 1H), 3.13 – 3.06 (m, 2H), 2.95 – 2.90 (m, 2H), 2.45 (s, 3H), 2.22 – 2.15 (m, 1H), 2.00 – 1.89 (m, 1H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ 137.06, 134.92, 130.33, 128.06, 127.73, 126.51, 48.16, 32.29, 25.16, 18.99.

**(*E*)-2-Styryl-1,3-dithiolane (5d):**[22d] Following the general procedure, using cinnamaldehyde (0.132g, 1.0 mmol), 1,2-ethanedithiol (0.103g, 1.1 mmol), eosin Y (0.6 mg, 0.1 mol%), then purified by column chromatography ($SiO_2$, 10% ethyl acetate in hexanes) to provide **5d** as the yellow oil (0.073g, 35% yield). $^1$H NMR (400 MHz, $CDCl_3$): δ 7.38 – 7.36 (m, 2H), 7.32 – 7.28 (m, 2H), 7.26 – 7.21 (m, 1H), 6.51 (d, $J$ = 16.0 Hz, 1H), 6.21 (dd, $J$ = 16.0, 8.0 Hz, 1H), 5.23 (d, $J$ = 8.0 Hz, 1H), 3.41 – 3.25 (m, 4H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ 135.98, 130.05, 128.96, 128.44, 127.72, 126.52, 54.40, 39.52.

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FULL PAPER

# Visible-Light Photoredox-Catalyzed Thioacetalization of Aldehydes Under Metal-Free and Solvent-Free Conditions

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