

Aggregation-Induced Emission

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1,1-Diphenylvinylsulfide as a Functional AIEgen Derived from the Aggregation-Caused-Quenching Molecule 1,1-Diphenylethene through Simple Thioetherification

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Abstract: An efficient and readily scalable thioetherification between 1,1-diphenylethene (DPE) and sodium arylsulfinate was developed for the synthesis of 1,1-diphenylvinylsulfide (DPVS) with the yield up to 99%. The photophysical properties of DPVS show that the introduction of arylsulfenyl groups onto the parent molecule DPE makes DPVS a novel type of aggregation-induced emission (AIE) luminogen (AIEgen) with large Stoke's shift (up to 188 nm). These DPVS possess AIE properties due to restriction of intramolecular motions (RIM), as demonstrated by crystal structure analysis. Importantly, the AIE performance of DPVS can be applied to sense the nitroaromatic explosive picric acid in aqueous systems through a "turn-off" response.

Aggregation-induced emission (AIE) or aggregation-induced enhanced emission (AIEE), which were discovered by the Tang^[1] and Park^[2] groups, respectively, can overcome the aggregation-caused quenching (ACQ) effects of traditional luminogens, and have attracted extensive attention recently.^[3] As described by the Tang group,^[4] the AIE mechanism can be ascribed to restriction of intramolecular motions (RIM). In particular, both restriction of intramolecular rotations (RIR) and restriction of intramolecular vibrations may encourage AIE luminogens (AIEgens) to strongly emit in the solid or aggregated state. At present, many typical AIEgens, such as triphenylamine,^[4,5] tetraphenylethene,^[6] and silole derivatives^[7] have been developed, and they are promising for OLEDs,^[8] bioimaging,^[9] chemosensors^[10] and theranostics.^[11]

By contrast, the design and synthesis of trisubstituted-ethene-type AIEgens based on the RIR mechanism has

always been challenging (Figure 1). Since *cis,cis*-1,2,3,4-tetraphenylbutadiene (TPBD) with AIE properties due to RIR of the phenyl groups was first reported in 2004 by the Cao group,^[12] 1,4-di[(E)-2-phenyl-1-propenyl]benzene (PPB) and

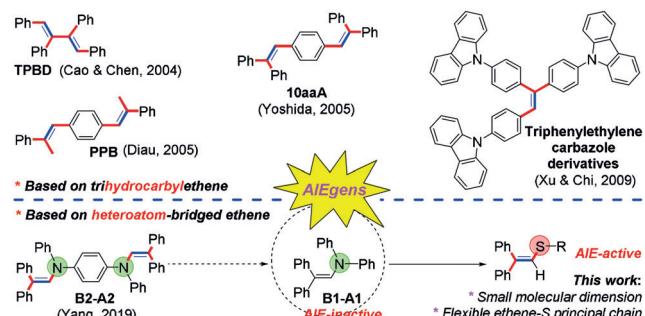


Figure 1. Reported examples of trisubstituted-ethene-type AIEgens and an AIE-inactive luminogen.

a series of triarylethene derivatives have been discovered by the Diau^[13] and Yoshida groups,^[14] respectively. Moreover, triphenylethylene carbazole derivatives have been explored by the Xu group.^[15] Due to their important applications,^[16] novel and excellent trisubstituted-ethene-type AIEgens have been well studied.^[17] However, trisubstituted ethene type AIEgens with a p–π conjugation structure directly connecting to a heteroatom are rarely reported. Recently, Yang et al.^[18] designed a class of oligoaniline-based derivatives, and found that B2-A2 is an AIEgen but B1-A1 is AIE-inactive due to its long ethylene-N principal chain and small size. Thus, it is a challenging task to design novel trisubstituted-ethene-type AIEgens with simple heteroatom structural skeleton.

At the same time, in the last few years, sulfides have been developed as unique functional materials, especially in metal-organic frameworks (MOFs), covalent organic frameworks (COFs), and sensors.^[19] Meanwhile, sodium sulfinate is an important reagent for construction of the C–S bond in sulfides.^[20–24] On the basis of the previous research on the utilization of sodium sulfinate reagent in our laboratory,^[25] we were surprised at the unexpected discovery that a thioetherification reaction between radical scavenger 1,1-diphenylethene (DPE) and sulfinate can give products with high yields. More importantly, the facile synthesis of these novel trisubstituted ethenes with p–π conjugation between the p orbital of the S atom and the ethene backbone converts the parent compound DPE, which is a traditional aggregation-caused

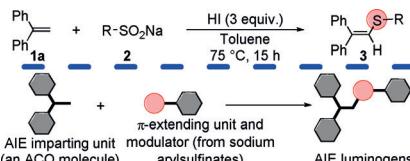
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quenching (ACQ) molecule with poor emission, into AIEgens for the introduction of arylsulfenyl groups to extend the conjugation over the whole molecule and weaken $\pi-\pi$ stacking interaction between molecules (Scheme 1). Thus, it is desirable to develop novel trisubstituted-ethene-type AIEgens with simple *S*-bridged structure that function through a RIM mechanism.



Scheme 1. The design of AIE luminogens.

Initially, we optimized the reaction conditions using DPE (**1a**) and sodium *p*-toluenesulfinate (**2a**) as model substrates. The optimal conditions were identified as reductant HI (45% aqueous, 3 equiv) and **1a/2a** = 1:1.5 in toluene as solvent at 75 °C for 15 hours (Table S1 in Supporting Information). With the optimized conditions in hand, the substrate scope was further investigated (Table 1). The products 1,1-diphenylvinylsulfide (DPVS) **3** and **4** (the structures of all obtained products **3** and **4** were confirmed, including by X-ray analysis for **3a**, **3c**, **3g**, and **3i**;^[26] see the Supporting Information for more details) were smoothly obtained. In most cases, the yield is over 80%. Even for 1,1-diarylethene derivatives with a different substituent on phenyl, the reaction is usually successful (see the Supporting Information for further discussion of the effect of different groups on the yield of **3** and **4**). Importantly, when DPVSs **3** (e.g. **3a–3d**, **3g**, **3i** and **3u**) are prepared on a large scale under standard conditions,

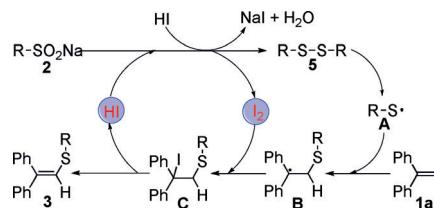
Table 1: Substrate scope and their application of gram-scale preparation for DPVS **3**.^[a,b]

1	2	HI (3 equiv.)	Toluene	3/4
		75 °C, 15 h		
	3a , R' = CH ₃ CCDC 1921763	97% (91%) ^[c]		
	3b , R' = H CCDC 1921764	85% (83%) ^[c]		
	3c , R' = t-Bu CCDC 1921764	87% (87%) ^[c]		
	3i	95%		
	3p , 75%			
	3q , 92%			
	3t , 40%			
	3u , 99% (88%) ^[c]			
	4a , Ar = 4-MeC ₆ H ₄ , R' = C ₆ H ₅ , 72%			
	4b , Ar = 4-MeC ₆ H ₄ , R' = 4-CIC ₆ H ₄ , 72%			
	4c , Ar = 4-(CH ₃) ₂ NHC ₆ H ₄ , R' = 4-CH ₃ OCH ₂ H ₄ , 22%			
	4d , Ar = 4-CIC ₆ H ₄ , R' = 2-CH ₃ C ₆ H ₄ , 72%			
	4f , Ar = 4-CIC ₆ H ₄ , R' = CH ₃ , 83%			
	4g , Ar = 4-CH ₃ OCH ₂ H ₄ , R' = 4-CH ₃ C ₆ H ₄ , 0%			

[a] Reaction conditions: **1** (0.3 mmol), **2** (0.45 mmol), HI (45% aqueous, 3 equiv), toluene (2 mL), 75 °C, 15 h, unless otherwise noted. [b] Yields of isolated product. [c] Yields of isolated product for large-scale synthesis of DPVS **3** are given in parenthesis (see more details in Table S3).

the yields are good to excellent (Table 1, yields shown in parenthesis).

To obtain more information about this thioetherification, several control experiments were carried out (Scheme S1 in the Supporting Information) and the results indicated that it is a radical process. Furthermore, the existence of both I₂ and disulfide **5** as important intermediates can be confirmed (see the Supporting Information). Based on these results and previous reports,^[20,22,23] a plausible mechanism is suggested (Scheme 2). First, sodium arylsulfinate **2** is reduced by HI,



Scheme 2. Proposed mechanism for sulfonylation of DPE.

producing intermediate disulfide **5** and oxidation product I₂. Subsequently, homolytic cleavage of **5** generates arylsulfenyl radical **A**, which react with **1a** through radical addition to afford intermediate **B**. Finally, the radical cross-coupling of **B** with an iodine radical from I₂ forms intermediate **C**, which undergoes elimination to generate highly conjugated product **3** and HI.

In the above mechanism, partial HI can take part in the cycle, because the oxidation product iodine may act as an “internal oxidant” to react with radical **B** in this transformation. Thus, for synthesis of DPVS, compared with the traditional method using expensive alkynoates and poisonous thiols as raw materials under a reaction temperature of 120 °C with lower yields (only up to 76%),^[27] our method has many advantages, such as readily available and non-toxic materials, lower reaction temperature, higher yields, and notably no need for extra oxidants. Notably, even though there are many reports on the synthesis of sulfides from sodium sulfites,^[20–22] our method also has the advantage of avoiding metal catalysts or additional oxidants (e.g., I₂). In summary, we have successfully developed a systematic and accessible method for the synthesis of sulfides, especially DPVS.

On the other hand, we further investigated the AIE properties of DPVS and its application. Selecting **3a**, **3c**, **3g**, and **3i** as representative DPVS compounds, their photophysical properties are summarized in Table 2 (some corresponding figures can be seen in the Supporting Information) and Figure 2; the photophysical features of other synthesized compounds are given in Table S8 in Supporting Information. For compounds **3a** and **3c**, the similar emission peak in the solid state can be found in Figure S6. For **3g**, there is a slight blueshift. In contrast, the nitro group as a typical electron-withdrawing group in compound **3i** gives rise to the obvious redshift (Figure 2d) and large Stoke’s shift (Table 2). Thus, our thioetherification by introducing different arylsulfenyl groups onto DPE can modulate the color of the target product (Figure S7). In addition, with compound **3i**, solvation effects for DPVS were also observed (Figure S38). There is an

Table 2: Photophysical data of representative DPVs.^[a]

Title compound	Solution [nm] λ_{abs}	Solution [nm] λ_{em}	Stoke's shift	Solid λ_{em} [nm]	τ_F [ns]	Φ_F [%]	α_{AIE}	Photo images ^[b] Powder	Photo images ^[b] Crystal
1a	231	442 ^[c]	211 ^[c]	458 ^[d]	1.11 ^[d]	ND ^[d,e]	0.35	—	—
3a	312	456	144	445	2.07	17.8	2.65		
3c	311	444	133	445	1.78	3.7	2.60		
3g	313	444	131	440	0.63	0.3	5.98		
3i	368	556	188	540	0.60	0.5	2.56		
3u	300	443	143	450 ^[d]	0.28 ^[d]	3.3 ^[d]	0.92	—	—

[a] λ_{abs} and λ_{em} refer to optimal absorption wavelength and photoluminescence (PL) peak, respectively. τ_F is fluorescence lifetime. Φ_F is absolute fluorescence quantum yield. α_{AIE} is defined as PL intensity in aggregation state/PL intensity in solution state. [b] Photo images of the powder and crystal under UV light ($\lambda_{\text{ex}}=365$ nm). [c] DPE is ACQ molecule due to the PL peak with ACQ characteristics. [d] Measured in liquid state. [e] Not detected.

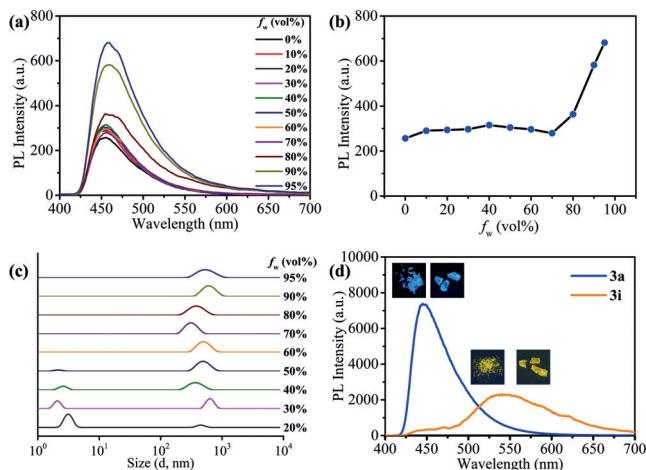


Figure 2. a) Emission spectra and b) relative emission intensity of compound **3a** in THF/H₂O mixture with increasing water fractions (f_w) to 95% ($c=150 \mu\text{M}$, $\lambda_{\text{ex}}=275$ nm). c) DLS curves of **3a** in THF/H₂O mixture with increasing f_w to 95% ($c=150 \mu\text{M}$). d) Solid emission spectra of compounds **3a** ($\lambda_{\text{ex}}=380$ nm) and **3i** ($\lambda_{\text{ex}}=275$ nm). Inset: photographs of powder and crystals compounds **3a** and **3i** under UV light ($\lambda_{\text{ex}}=365$ nm).

unusual but interesting phenomenon for **3i** (see the Supporting Information), which is consistent with previous reports.^[28]

The absorption spectra of DPVs in a THF/H₂O mixture show a similar tendency (Figures S41–69): when the water fraction (f_w) increases, broadened absorption bands and level-off tails can be observed. These phenomena are attributed to J-aggregation.^[29] The AIE behavior of these selected DPVs were investigated by measuring the photoluminescence (PL) spectra with varying f_w (Figure 2 and Figures S70–97). As

expected, PL spectra of **3a**, **3c** and **3g** exhibit similar changes. Using **3a** as an example, with an increase of f_w from 40 to 70%, the fluorescence intensity of **3a** slightly decreases (Figure 2a). This is probably due to the combined effect of AIE and the twisted intramolecular charge transfer (TICT).^[28,30] For DPVs **3i**, with increasing f_w (0–70%), a decrease in fluorescence emission can be observed due to stronger TICT effect (Figure S77). As the f_w is further gradually increased from 70 to 95%, there is a drastic increase for the fluorescence intensity of all selected samples solution, showing typical AIE features due to the formation of nanoaggregates. These results are also confirmed by dynamic light scattering (DLS) analysis, as shown in Figure 2c and Figures S98–101. For example, when the f_w increases, the average size of nanoaggregates of **3a** is altered (Figure 2c), and the

bigger nanoaggregates also can be observed and measured by scanning electron microscopy (SEM; Figure S102). Moreover, the α_{AIE} value of selected DPVs have been calculated (Table 2), thus indicating emission enhancement.^[31,32] Even though DPVs **3a**, **3c**, **3g**, and **3i** contain a heavy sulfur atom, which causes a reasonably low α_{AIE} value,^[31] they still are AIEgens. Thus, it is the arylsulfenyl group that imparts AIE feature to DPE framework (more discussions on the comparison with compound **3u**, synthesized from a kind of sodium alkyl sulfinate, can be found in the Supporting Information).

Determination and analysis of the single-crystal structure are useful for revealing AIE mechanism. Compounds **3a**, **3c**, **3g** and **3i** are arranged in a herringbone-type fashion^[17] in the crystalline state (Table S9, see the Supporting Information). The dihedral angles between the phenyl ring and the central double bond of the selected compounds range from 30.26° to 65.87°, thus suggesting that DPVs adopt a highly twisted conformation when packing (Figure 3a). There is little π – π stacking (Figure 3b) because of the steric hindrance created by aryl rings.^[3g,33] Furthermore, the intermolecular weak C–H··· π interaction^[17,31] may rigidify the conformation^[4] and restrict intramolecular motion (Figure 3c, Figures S103–109). In short, the twisted geometry is responsible for emission in the aggregated or solid state.^[1d] Furthermore, it can be seen from the results of DFT theoretical calculation that, for **3a**, **3c**, and **3g**, there are similar highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) energy levels (Figure S110) and their electron cloud is distributed on the whole molecule (see the Supporting Information for further discussion). These results indicate that the above three compounds have good molecular conjugation.^[34] For **3i**, both the HOMO and LUMO energy

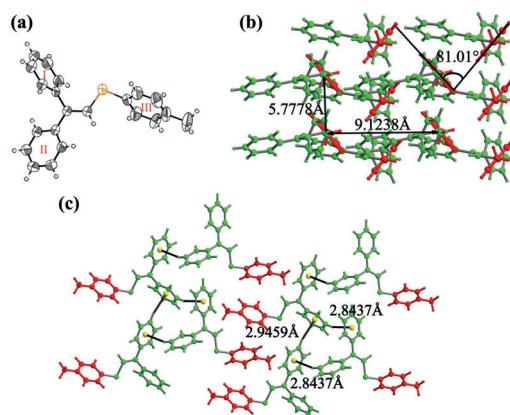


Figure 3. a) Molecular conformation of **3a**. b) Side view and c) top view of packing structure of **3a** crystal.

levels are lower, and this theoretical result is consistent with its fluorescent behaviour.^[35]

Achieving the detection of nitroaromatic explosives (NAEs), for example, picric acid (PA), is important for environmental protection and public security.^[36,37] In particular, the water solubility of PA poses hazards to water sources.^[36] Thus, the charge-transfer emission-quenching effect between PA, which is an electron-deficient compound, and electron-rich AIEgens is an approach for detecting PA in aqueous systems.^[37,38] On the basis of research on small-molecule fluorescent probes in our laboratory,^[39] using **3a** as example, we studied the PL spectra of **3a** upon adding a PA solution in THF/H₂O mixture ($f_w = 95\%$). As shown in Figure 4, the PL intensity of **3a** is significantly decreased at 470 nm in the presence of PA, accompanied by a redshift for the maximum emission peak. Therefore, detecting PA can be achieved based on the fluorescence-quenching effect of **3a** (see the Supporting Information for more sensing results and corresponding discussions).

In summary, this work provides a facile and readily scalable synthetic route to DPVS through HI-promoted thioetherification of DPE. Notably, this method imparts AIE properties to DPVS by extending conjugation, increasing steric hindrance, and producing RIM effects. The photo-

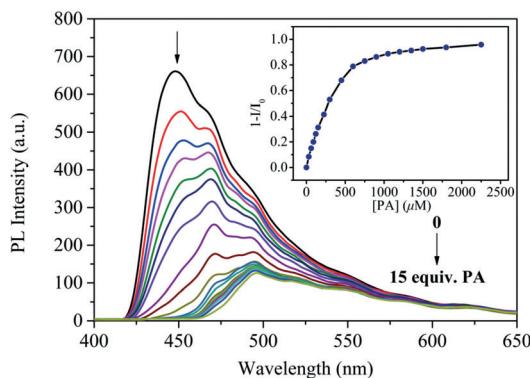


Figure 4. Fluorescence titration spectra of compound **3a** ($c = 150 \mu\text{M}$) with PA in THF/H₂O mixture ($f_w = 95\%$, $\lambda_{\text{ex}} = 275 \text{ nm}$) buffered with HEPES (pH 7.00).

physical properties of DPVSs are consistent with the results of corresponding theoretical calculation. In addition, DPVS can serve as a PA sensor with a “turn-off” response in aqueous systems. Therefore, this concise strategy of introducing arylsulfenyl group onto the ACQ molecule DPE to build novel trisubstituted-ethene-type AIEgens with S-bridged structures will be beneficial for the design and synthesis of more practical AIEgens with a simple heteroatom linker that operate through a RIM mechanism. Research along these lines is currently underway in our laboratories.

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Conflict of interest

The authors declare no conflict of interest.

Keywords: 1,1-diphenylethene · aggregation-induced emission · restriction of intramolecular motions · sodium sulfinate · thioetherification

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