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ORIGINAL PAPER

Multiple BODIPY derivatives with 1,3,5-triazine as core: balance between fluorescence and numbers of BODIPY units

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Abstract

The multiple BODIPY derivatives could exhibit higher fluorescence than mono-BODIPY derivatives but also might produce the stronger aggregation-induced quenching effect. It is important to investigate the relationship between fluorescence and numbers of BODIPY units. In this work, series of multiple BODIPY derivatives **2–5** with 1,3,5-triazine as core were designed and synthesized in high yields. A subtle balance between the fluorescence enhancement of multiple BODIPY units and fluorescence quenching based on *H*-aggregation of multiple BODIPY units was observed for these multiple BODIPY derivatives. Compound **3** bearing two BODIPY units with triazine as core presented the strongest fluorescence emission and highest fluorescence quantum yield. Compound **3** was successfully applied in viscosity measurement, exhibiting the linear relationship between fluorescence intensity (or steady-state emission anisotropy) and viscosity.

Keywords Triazine · BODIPY · Synthesis · Fluorescence · Multiple

Introduction

In the past decades, 4,4-difluoro-4-borata-3a,4a-diaza-s-indacene (abbreviated as BODIPY), as a class of organic laser dye, is of wide importance and attracts increasing attention based on the unique fluorescence [1–4]. Various BODIPY derivatives had been prepared and exhibited extensive applications in the field of biological sensing, electrogenerated chemiluminescence, viscosity detection, ion recognition and fluorescent liquid crystals, etc. [5–18]. In general, when the BODIPY derivatives were used as the fluorescent probes or fluorescent materials, the brightness of the fluorophore played the crucial role for the intensity of the detected signal and the sensitivity of the overall protocol [19, 20]. To improve the brightness of fluorescence, one way is to increase the concentration of dye, but resulting in the aggregation-induced quenching (ACQ) effect usually [21, 22]. Another way is to enhance the intrinsic luminous abilities of dyes by the suitable structural modification. Thus, some research focused on the preparation of multiple BODIPY derivatives or BODIPY oligomers with strong fluorescence, which were applied successfully in biological sensing and viscosity detection [7, 23–26]. Survey of these literatures suggested that the multiple BODIPY derivatives normally showed stronger fluorescence than mono-BODIPY derivatives and the fluorescence changed dramatically with the variation of structures and numbers of BODIPY units. But no research concerned on the investigation of the relationship between fluorescence and numbers of BODIPY units, which is a very important characteristic for multiple BODIPY derivatives due to the ACQ effect observed usually for interaction of multiple BODIPY unit. Thus, the optimization of the numbers of BODIPY units is expected to obtain the high fluorescence for multiple BODIPY derivatives.

On the other hand, cyanuric chloride, possessing three Cl groups with different reaction activity [27, 28], is a good platform to construct multiple BODIPY derivatives. Up to now, several triazine derivatives with one, two or three BODIPY derivatives bridged by different spacers were reported and presented interesting properties such as Cu(II) sensor [8, 29–32]. The preparation of these triazine-BODIPY

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derivatives usually involved the step-by-step synthesis of BODIPY unit on triazine core, leading to the low yield and complicated separation procedure. Moreover, the relationship between fluorescence and numbers of BODIPY units on triazine core was also not studied. In this paper, we wish to report a simple synthesis of multiple BODIPY derivatives using 1,3,5-triazine as core with one, two and three BODIPY units by straightforward nucleophilic substitution in high yields. More importantly, the relationship between fluorescence and numbers of BODIPY units was explored for the first time, suggesting that the triazine derivative with two BODIPY units showed the strongest fluorescence among the series of multiple BODIPY derivatives. Furthermore, the triazine derivative with two BODIPY units was used for the viscosity measurement. The measurement results exhibited the linear relationship between fluorescence intensity and viscosity, and were in accordance with Förster–Hoffman theory, implying the potential application prospective for viscosity detection.

Experimental

Apparatus and reagents

The chemical reagents were purchased from commercial suppliers. All organic solvents and inorganic reagents were used directly without purification. TLC analysis was carried out on pre-coated glass plates. Column chromatography was done with silica gel (200–300 mesh). NMR spectra were recorded on Bruker-ARX 600 instrument at 26 °C. MS spectra were measured on Bruker mass spectrometer. Compound **1** was prepared by the published method [33]. UV–Vis was performed on Varian UV–Vis spectrometer. Fluorescence spectra were obtained on a Hitachi F-4500 spectrometer with a conventional quartz cell ($10 \times 10 \times 45$ mm) at 25 °C. The excitation and emission slits were 5 nm wide. The fluorescence absolute Φ_F values were measured using an Edinburgh Instruments FLS920 Fluorescence Spectrometer with a 6-inch integrating sphere.

Synthesis of mono-BODIPY derivative 2

Under N_2 atmosphere, a mixture of cyanuric chloride (0.12 g, 0.65 mmol), compound **1** (0.18 g, 0.53 mmol) and $NaHCO_3$ (0.065 g, 0.77 mmol) was stirred in 20 mL of dry acetone at 0–5 °C for 1.5 h. The TLC detection indicated the disappearance of compound **1**. Then, the reaction mixture was poured into 100 mL of ice water. The obtained mixture was stirred and the red precipitate emerged gradually. The red precipitate was filtered, washed by ice water and dried under vacuum. Compound **2** was obtained as the red solid in yield of 88%. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 1.46 (s, 6H, CH_3), 2.56 (s, 6H, CH_3), 6.01 (s, 2H, ArH), 7.31 (d, $J = 8.0$ Hz, 2H, ArH), 7.40 (d, $J = 8.0$ Hz, 2H, ArH). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 14.53, 29.72, 121.55, 122.28, 129.90, 131.33, 133.95, 139.90, 143.03, 151.61, 155.98, 171.25, 173.17; MS(MALDI-TOF)(*m*/*z*) : 489.338$(M + H)^+$. HR-MS(ESI)(*m*/*z*): calcd for $C_{22}H_{18}BCl_2F_2N_5O$ 488.1026 $(M + H)^+$, found 488.1028.

Synthesis of bis-BODIPY derivative 3

Under N_2 atmosphere, a mixture of compound **1** (0.18 g, 0.53 mmol), compound **2** (0.26 g, 0.53 mmol), and Na_2CO_3 (0.074 g, 0.7 mmol) was stirred in 30 mL of dry acetone at room temperature for 2 h. The TLC detection indicated the disappearance of strarting materials. Then, the reaction mixture was poured into 100 mL of ice water. The obtained mixture was stirred severely and the red precipitate appeared gradually. The red precipitate was filtered, washed by ice water and dried under vacuum. After purified by column chromatography (eluent: CH_2Cl_2:petroleum ether = 2:1), compound **3** was collected as the red solid in yield of 85%. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 1.47 (s, 12H, CH_3), 2.56 (s, 12H, CH_3), 6.01 (s, 4H, ArH), 7.32 (d, $J = 8.0$ Hz, 4H, ArH), 7.39 (d, $J = 8.0$ Hz, 4H, ArH). ^{13}C NMR (100 MHz, $CDCl_3$) δppm: 14.42, 29.39, 121.20, 122.26, 129.51, 131.38, 133.46, 140.11, 143.06, 151.88, 155.82, 171.34, 173.62; MS(MALDI-TOF) (*m*/*z*): 792.280$(M + H)^+$. HR-MS(ESI)(*m*/*z*): calcd for $C_{41}H_{36}B_2ClF_4N_7O_2$ 814.2647 $(M + Na)^+$, found 814.2632.

Synthesis of tri-BODIPY derivative 4

Under N_2 atmosphere, a mixture of compound **1** (0.09 g, 0.27 mmol), compound **3** (0.21 g, 0.27 mmol), and Na_2CO_3 (0.1 g, 0.9 mmol) was stirred and refluxed in 30 mL of dry THF for 3 h. The TLC detection indicated the disappearance of strarting materials. Then, the reaction mixture was poured into 100 mL of ice water. The obtained mixture was extracted by 30 mL of $CHCl_3$. The organic layer was separated and concentrated. The residue was purified by chromatographic column (eluent: CH_2Cl_2). Compound **4** was collected as dark red solid in yield of 72%. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 1.47 (s, 18H, CH_3), 2.56 (s, 18H, CH_3), 5.98 (s, 6H, ArH), 7.34 (d, $J = 8.0$ Hz, 6H, ArH), 7.38 (d, $J = 8.0$ Hz, 6H, ArH). ^{13}C NMR (100 MHz, $CDCl_3$) δppm: 14.45, 29.36, 121.49, 122.51, 129.48, 131.41, 133.08, 140.29, 142.92, 152.21, 155.91, 173.48; MS(MALDI-TOF)(*m*/*z*) : 1096.385$(MH)^+$. HR-MS(ESI) (*m*/*z*): calcd for $C_{60}H_{54}B_3F_6N_9O_3$ 1057.6985$(M\text{-}2F)^+$, found 1057.6970.

Synthesis of bis-BODIPY derivative 5

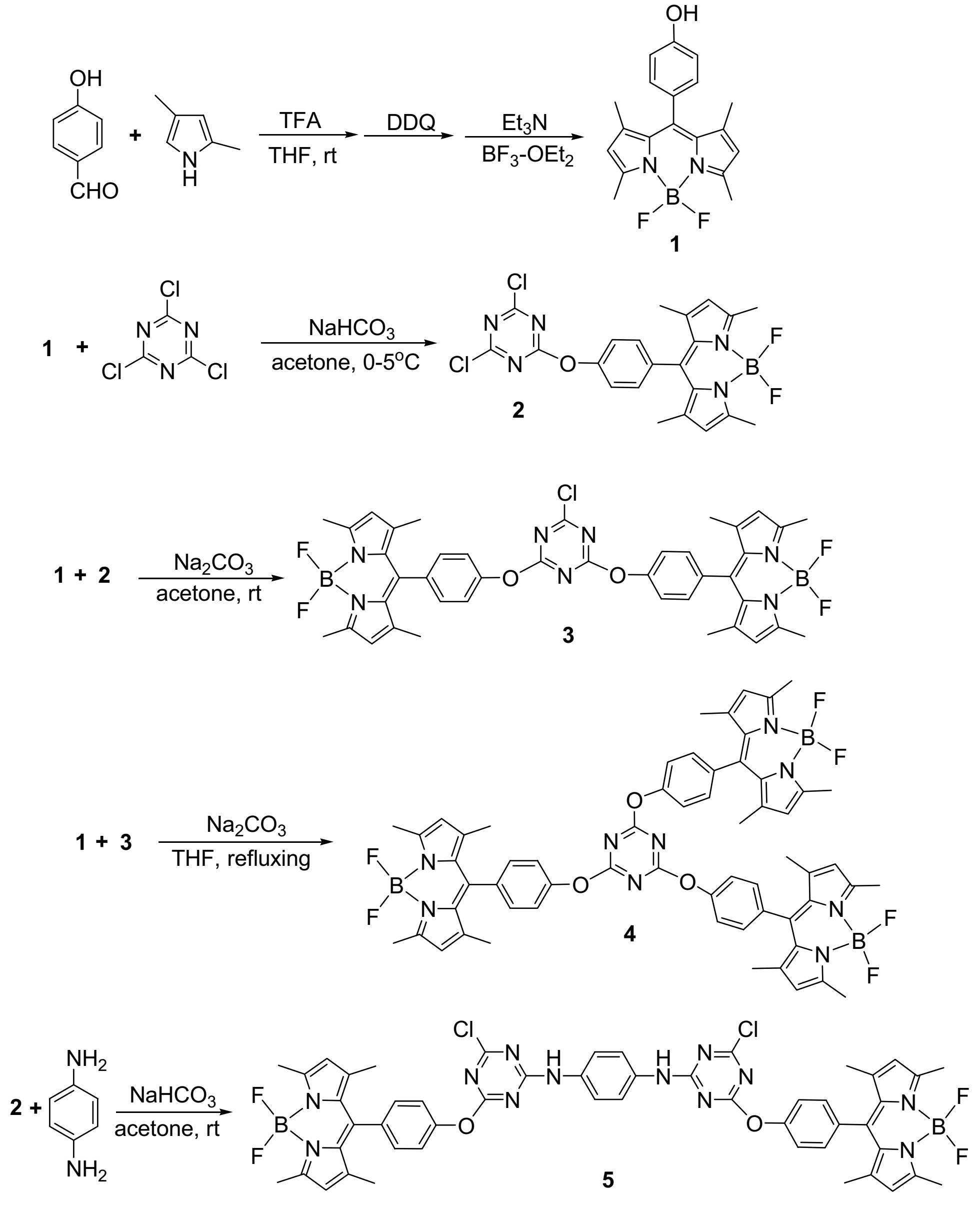
Under N_2 atmosphere, a mixture of compound **2** (0.25 g, 0.32 mmol), benzene-1,4-diamine (0.017 g, 0.16 mmol), and $NaHCO_3$ (0.059 g, 0.7 mmol) was stirred and refluxed in 30 mL of dry acetone at room temperature for 6 h. The TLC detection indicated the disappearance of strarting materials. Then, the reaction mixture was poured into 100 mL of ice water. The obtained mixture was extracted by 30 mL of $CHCl_3$. The organic layer was separated and concentrated. The residue was purified by chromatographic column (eluent:CH_2Cl_2:$CH_3COOEt = 3:2$). Compound **5** was collected as red solid in yield of 67%. ^1H NMR (400 MHz, $CDCl_3$) δ (ppm): 1.48 (s, 12H, CH_3), 2.56 (s, 12H, CH_3), 5.34 (bs, 2H, NH), 6.00 (s, 4H, ArH), 6.58 (s, 4H, ArH), 7.31 (d, $J = 8.0$ Hz, 4H, ArH), 7.35 (d, $J = 8.0$ Hz, 4H, ArH). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 14.54, 29.84, 120.78, 121.19, 122.71, 123.87, 128.83, 131.86, 1337.61, 140.80, 142.98, 152.60, 155.60, 164.84, 166.97, 174.85; MS(MALDI-TOF)(*m/z*): 1049.881$(M+K)^+$. MS(ESI)(*m/z*): calcd for $C_{50}H_{42}B_2Cl_2F_4N_{12}O_2$ 1049.2701$(M+K)^+$, found 1049.2741.

Results and discussion

Syntheses and characterization

The synthetic routes were exhibited in Scheme 1. To avoid the step-by-step synthesis of BODIPY unit on triazine core,

Scheme 1 Synthesis of multiple Bodipy derivatives **2–5**



the hydroxyl BODIPY **1** was synthesized beforehand in moderate yield of 40% according to the published procedure [33]. Then, based on the different reaction activities of three Cl groups on cyanuric chloride, the BODIPY derivatives using 1,3,5-triazine as core with one, two and three BODIPY units were obtained selectively in high yields by controlling the reaction condition with different bases and reaction temperature. By the nucleophilic substitution of cyanuric chloride with compound **1** in $NaHCO_3$/acetone system at 0–5 °C, triazine derivative with one BODIPY unit **2** was prepared in yield of 88% by simple precipitation procedure. Further treating compound **2** with compound **1** in Na_2CO_3/acetone at room temperature, triazine derivative with two BODIPY units **3** was conveniently collected in yield of 86% after column chromatography. The triazine derivative with three BODIPY units **4** was synthesized by refluxing compound **1** with compound **3** in Na_2CO_3/THF system in yield of 72%. Moreover, for the purpose of fluorescent comparison, another triazine derivative with two BODIPY units **5** bridged by *p*-phenylenediamine were prepared by reacting compound **2** with *p*-phenylenediamine in $NaHCO_3$/acetone at room temperature in yield of 67%. One can see that, by comparing with the reported triazine-BODIPY derivatives [8, 29–32], all these new multiple BODIPY derivatives with 1,3,5-triazine core **2–5** were prepared in high yields and simple procedure.

New compounds **2–5** were characterized by ^{1}H NMR, ^{13}C NMR, MALDI-TOF-MS and HR-ESI-MS spectra. In their ^{1}H NMR spetra, a pair of doublet and one singlet for ArH, and two singlets for CH_3 were clearly observed, suggesting the structure of BODIPY unit. In the HR-ESI-MS spectra, the molecular-ion peaks for compounds **2, 3, 4** and **5** appeared at 488.1026, 814.2647, 1057.6985 and 1049.2701, which were exactly in accordance with their molecular weights (the deviation values < 5 ppm). The ^{13}C NMR spectra also supported their structures. All these characterizations confirmed the structures of compounds **2, 3, 4** and **5** as shown in Scheme 1.

Photophysical properties

The photophysical properties of compounds **2–5** were investigated by UV–Vis spectra and fluorescence spectra. The UV–Vis spectra in different solutions with various polarities were illustrated in Figs. S17–S20. All of them exhibited the maximum absorption at about 500 nm, which were similar to that of compound **1** [33]. The triazine core and the numbers of BODIPY units influenced little on absorption wavelength. These results indicated that the weak electron conjugation effect existed among the triazine core and the BODIPY units in compounds **2–5**. The similar phenomena were also observed for fluorescence spectra as shown in Figs. 1, 2, 3 and 4 and Table 1. Their maximum emissions appeared at 515 nm approximately, which also were influenced little by the number of BODIPY units. These results could be attributed to the little intramolecular electron interaction among these BODIPY and triazine units, resulting in the separated emission for each BODIPY unit. In theory, the separated emission for each BODIPY unit implied that the more BODIPY units in compounds **2–5** should lead to the stronger fluorescence. However, one can see that compound **3** with two BODIPY units exhibited the highest fluorescence intensities among the compounds **2, 3** and **4** with one, two or three BODIPY units. For example, the fluorescence intensity of compound **3** in MeOH was 5836 a.u., which is higher than that of compounds **2** and **4** (2350 and 2845 a.u., respectively). These results could be explained by that, although little intramolecular electron interaction for BODIPY unit existed, the intermolecular π–π stacking action (resulting *H*-aggregation-induced quenching) for BODIPY unit played an important role for fluorescence emission, that is to say, a subtle balance existed between fluorescence enhancement based on multiple BODIPY units and fluorescence quenching based on stronger *H*-aggregation of multiple BODIPY units. Compound **3** with two BODIPY units possessed strongest fluorescence, because of not only the superposed strong emission of two BODIPY units but also the weak

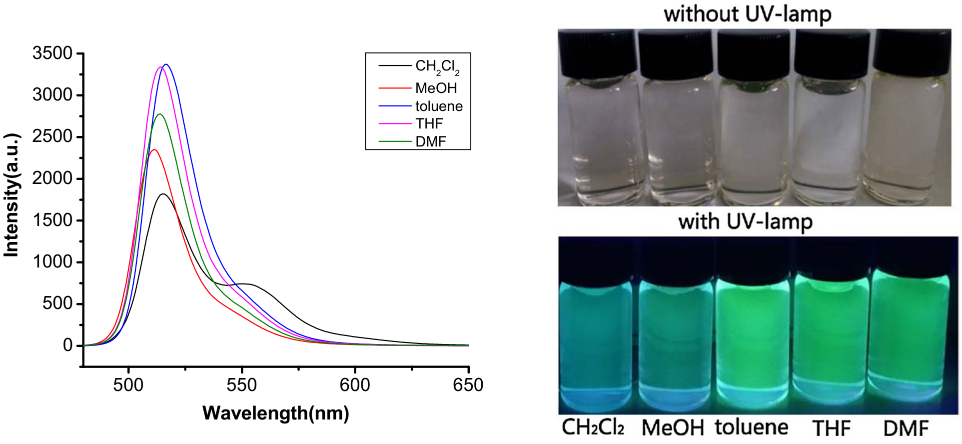


Fig. 1 The fluorescence spectra and photos of compound **2** in different solvents (2×10^{-6} M, $\lambda_{ex} = 480$ nm)

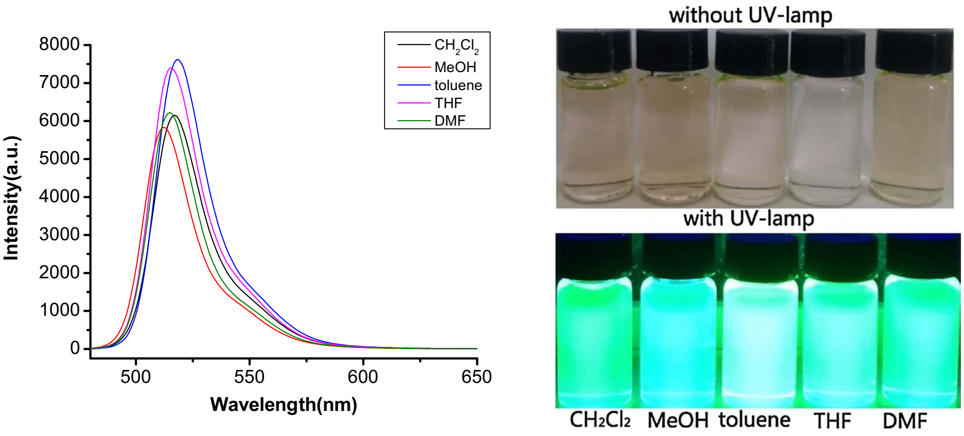


Fig. 2 The fluorescence spectra and photos of compound **3** in different solvents (2×10^{-6} M, $\lambda_{ex} = 480$ nm)

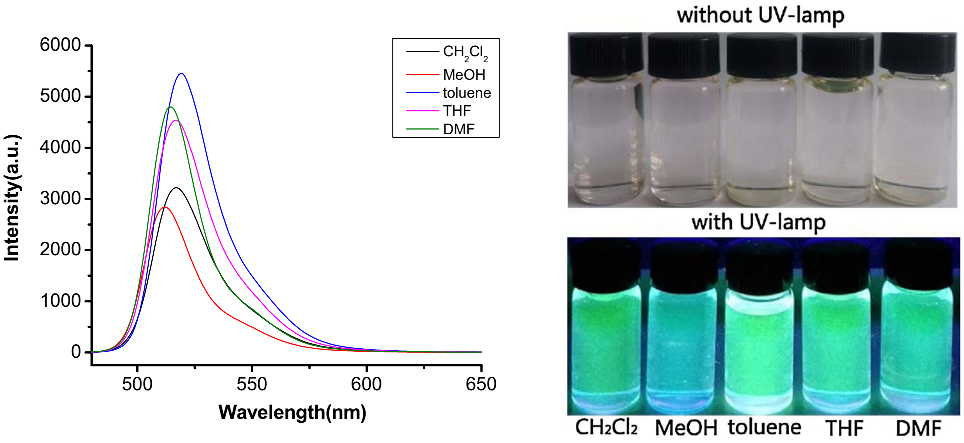


Fig. 3 The fluorescence spectra and photos of compound **4** in different solvents (2×10^{-6} M, $\lambda_{ex} = 480$ nm)

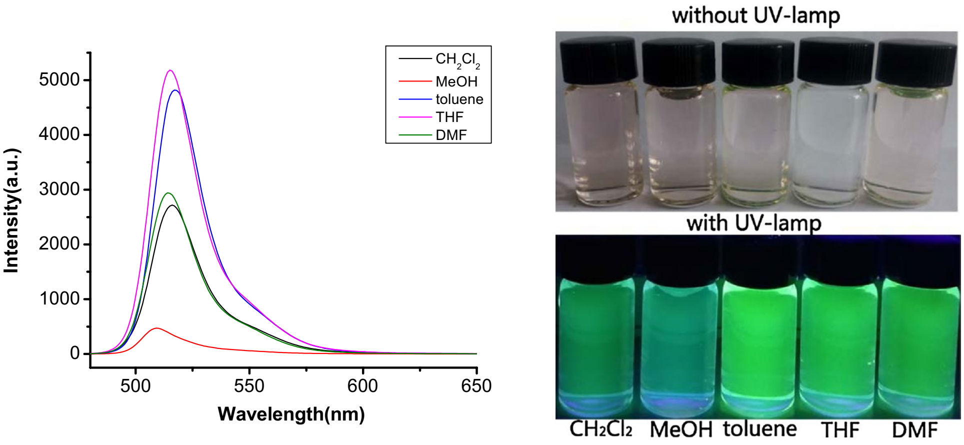


Fig. 4 The fluorescence spectra and photos of compound **5** in different solvents (2×10^{-6} M, $\lambda_{ex} = 480$ nm)

H-aggregation due to poor molecular symmetry. Although compound **4** had three BODIPY units, its higher molecular symmetry produced strong π–π stacking action and *H*-aggregation quenching, resulting in the weaker fluorescence than that of compound **3** with two BODIPY units. To confirm this speculation further, compound **5** with two BODIPY units bridging by large rigid structure was prepared for the purpose of comparison. Its fluorescence intensity was only 469 in MeOH solution, indicating the strong *H*-aggregation quenching due to the intense π–π stacking action based on the higher molecular symmetry of large rigid bridging structure than that of compound **3**. The speculated *H*-aggregation effect was confirmed by the blue shift of spectra in toluene (low polarity) and MeOH (high polarity). It can be seen that the obvious blue shifts of UV–Vis spectra for compounds **2, 3, 4** and **5** were observed as 6, 5, 6 and 6 nm from toluene

Table 1 The photophysical properties of compounds **1, 2, 3, 4** and **5** in different solvents

Dyes	Solvents	λ_{abs} (nm)	λ_{em} (nm)	Stokes shift (nm)	Fluorescence intensity	Φ_f
2	CH_2Cl_2	503	515	12	1821	0.13
	MeOH	499	511	12	2350	0.14
	Toluene	505	517	12	3371	0.17
	THF	502	514	12	3341	0.15
	DMF	502	514	12	2777	0.28
3	CH_2Cl_2	503	517	14	6152	0.12
	MeOH	499	512	13	5836	0.15
	Toluene	504	518	14	7617	0.18
	THF	502	515	13	7395	0.14
	DMF	501	515	14	6223	0.29
4	CH_2Cl_2	503	517	14	3223	0.07
	MeOH	499	512	13	2845	0.09
	Toluene	505	519	14	5459	0.14
	THF	501	517	16	4542	0.09
	DMF	501	514	13	4801	0.23
5	CH_2Cl_2	503	516	13	2717	0.09
	MeOH	499	509	10	469	0.07
	Toluene	505	517	12	4818	0.15
	THF	502	515	13	5185	0.13
	DMF	502	514	12	2940	0.17
1[a]	Toluene	503	514	11	–	0.94

[a] These data of compound **1** (1×10^{-6} M) in toluene were cited in Ref. [33]

to MeOH, respectively. In general, the blue shift indicates the *H*-aggregation, resulting in the fluorescence quenching [34–37]. Compound **3** showed the smallest blue shift, suggesting the weakest *H*-aggregation. On the other hand, their fluorescence quantum yields (Φ_f) were measured in different solvents. Compound **3** also presented the highest fluorescence quantum yield, further supporting that compound **3** with two BODIPY units was the best fluorescent dye among these multiple BODIPY derivatives. As compound **3** had strong fluorescence intensity and its Φ_f values in high polar solvent of MeOH and DMF attained 0.15 and 0.29, respectively, it exhibited the potential application prospective for viscosity detection of high polar solvents, such as the mixtures of ethanol and glycerol.

Viscosity measure

It is well known that the molecular viscosity could be measured based on suppressing the non-radiative relaxation in a viscosity-dependent manner and enhancing the fluorescence radiation [38]. The fluorescence characteristics of the probe could be tuned by the freedom of conformational change. As the rotation of aromatic rings in BODIPY depended on the viscosity of the local environment, the linear dependence of the fluorescence intensity and steady-state emission anisotropy as a function of medium's viscosity could be observed [7, 39, 40]. The relationship was usually examined by Förster–Hoffmann equation as follows [7, 39, 40]:

$$\text{Log}\, I = C + y \log \eta, \tag{1}$$

where η and I are the viscosity and the emission intensity, respectively. C means a constant and y is the sensitivity of the probe to viscosity. As compounds **3** had good emission and weak *H*-aggregation in polar solvents, its fluorescence and emission anisotropy in the mixtures of ethanol:glycerol with different polarities were investigated. The results were shown in Figs. 5 and 6, respectively.

In Fig. 5, one can see that the fluorescence intensity of compound **3** enhanced from 5973 to 21,026 a.u. as viscosity increased from 1.2 to 664 cP. After calculated by the formula (1), the good linear manner was obtained for the relationship between log I and log η with slope of 0.21 and R^2 of 0.99 (Fig. 5, insert), indicating that the medium's viscosity could be determined by the fluorescence change of compound **3**. Moreover, steady-state emission anisotropy could be applied as a function of medium viscosity for compound **3**. As shown in Fig. 6, emission anisotropy of compound **3** at viscosity of 1.2 cP was 0.074, which increased to 0.198 at viscosity of 664. The linear dependence of emission anisotropy as a function of medium's viscosity was also observed, suggesting that the viscosity could be measured by the detection of steady-state emission anisotropy.

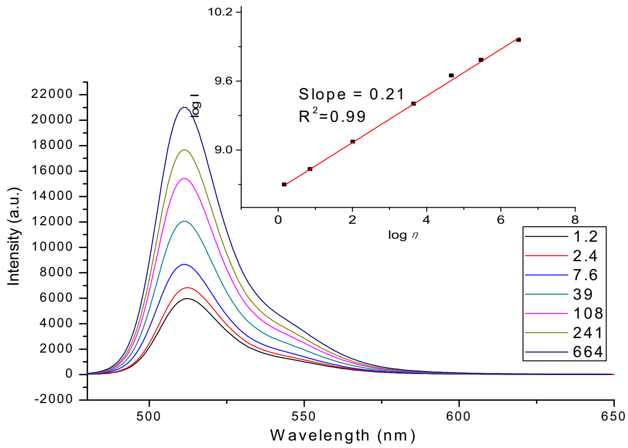


Fig. 5 Changes of fluorescence spectra of compound **3** (2×10^{-6}M, $\lambda_{ex}=480$ nm) with the variation of solution viscosity (mixtures of ethanol:glycerol); insert: linear relationship of log *I* and log *η*

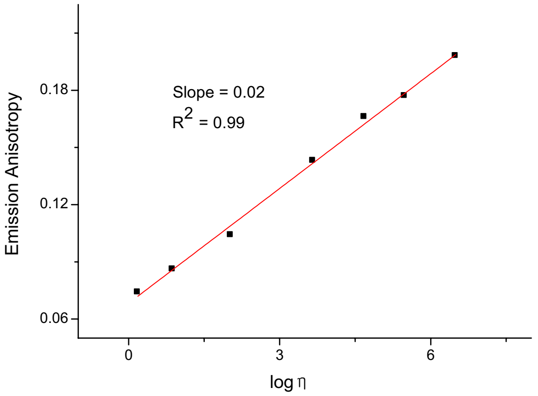


Fig. 6 Steady-state emission anisotropy of compound **3** as a function of viscosity (2×10^{-6} M, $\lambda_{ex}=480$ nm)

Combing the above analysis of Figs. 5 and 6, it could be summarized that compound **3** exhibited good application prospective for viscosity measurement.

Conclusions

In conclusion, series of multiple BODIPY derivatives **2–5** with 1,3,5-triazine core were prepared in high yields and simple procedure. The investigation of fluorescence suggested that a subtle balance between fluorescence and numbers of BODIPY units due to the existence of fluorescence enhancement based on BODIPY units and fluorescence quenching based on *H*-aggregation of BODIPY units. Compound **3** bearing two BODIPY units with triazine as core exhibited the strongest fluorescence emission and highest fluorescence quantum yield. The viscosity measurement based on the fluorescence change of compound **3** indicated the linear relationship between fluorescence intensity or steady-state emission anisotropy and viscosity according to Förster–Hoffmann theory, indicating the potential application prospective of compound **3** for viscosity detection.

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