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# Spectrofluorimetric determination of trace nitrite in food products with a new fluorescent probe 1,3,5,7-tetramethyl-2,6-dicarbethoxy-8-(3′,4′-diaminophenyl)-difluoroboradiaza-*s*-indacene

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**Abstract**

A new fluorescent probe 1,3,5,7-tetramethyl-2,6-dicarbethoxy-8-(3′,4′-diaminophenyl)-difluoroboradiaza-*s*-indacene (TMDCDABODIPY) has been developed to detect nitrite in meat products and vegetables. The fluorescence of TMDCDABODIPY is very weak, but when it reacts with nitrite, a strong fluorescent triazole forms in aqueous medium at room temperature, which offers the advantage of specificity and sensitivity for the determination of nitrite. The fluorescence intensity was linear over a nitrite concentration of 9–300 nmol $l^{-1}$ with a detection limit of 0.21 nmol $l^{-1}$ (S/N = 3). The proposed method has been used for the determination of trace nitrite in food products with the recoveries of 94.62–105.48%.


*Keywords:* 1,3,5,7-Tetramethyl-2,6-dicarbethoxy-8-(3′,4′-diaminophenyl)-difluoroboradiaza-*s*-indacence (TMDCDABODIPY); Fluorescent probe; Spectrofluorimetry; Nitrite

## 1. Introduction

Leafy vegetables are an excellent source of vitamins, minerals and biologically active compounds [1,2]. The incidence of coronary heart disease, atherosclerosis and stroke can be reduced by increasing vegetable consumption, as can that of the major cancers such as cancer of the stomach, lung, mouth, esophagus, colon and rectum.

However, leafy vegetable is one of the main sources of nitrite in our bodies. Commonly nitrates are abundant in food primarily because plants take up nitrogen from the soil in this ionic form. The nitrates in foods then can be reduced to nitrite because of some bacteria's action [3]. In another way, nitrite is widely used as preservatives in meat products due to their ability to inhibit the growth of spores of *Clostridium botulinum*. It is added (particularly to ground meat products) in the meat curing process to speed curing and the formation of the required colours and flavours.

It is well known that nitrite is a reactive chemical and must be used with caution. It is lethal to humans in a dose of approximately 1 g [4]. Nitrite can interfere with the oxygen transport system in the body and may result in the condition known as methaemoglobinaemia, in which the ability of haemoglobin to exchange oxygen is seriously reduced [5]. Infants under 3 months are thought to be more susceptible than adults [3]. Nitrite also acts as a nitrosating agent and under appropriate conditions produces nitroso compounds, some of which are specific and potent carcinogens. Furthermore, nitrite can be converted to nitric oxide, an active nitrosating agent, which can react with secondary amines and tertiary amines to form carcinogenic nitrosamines.

Due to these toxic effects, it is important to develop new analysis methods for determination of nitrite in food products. Many analytical methods for the determination of nitrite have been developed. But not all are suitable for routine ultra-trace determinations. The commonly employed methods for nitrite determination have included spectrophotometry [6–9], chemiluminescence [10,11], electrometric methods [12–16], chromatography [17,18] and spectrofluorimetry [19–21], etc. However, the spectrophotometric methods [22,23] suffer from poor sensitivity and interference from some anions. The chemiluminescence meth-

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ods are also widely applied, which are based either on the catalytic or photolytic reduction of $NO_2$ to NO and subsequent gas-phase reaction with ozone [24], or on the chemiluminescence reaction of $NO_2$ with an alkaline solution of luminol [25]. However, some of the methods encounter the interference from species such as $SO_2$, $H_2S$, $CO_2$, and $O_3$. In addition, complex and costly instrumentation is required. Various electrochemical techniques have also been proposed for the determination of nitrite, such as polarography. These methods are subject to severe interference from nitrate. Chromatography is also used [26,27], but suffers from more or less time-consuming procedures and complicated instrumentation. More recently, the capillary electrophoretic methods have been used in the fast separation of inorganic ions [28–30], Jimidar et al. [31] have determined the nitrate amounts in vegetable samples using capillary electrophoresis with the indirect detection method. Marshall and Trenerry [32] have proposed a direct detection method for the simultaneous determination of nitrites and nitrates in a variety of food tuffs. But the high-cost could prevent it from current monitoring use.

Spectrofluorimetry for nitrite determination have been developed [33–35], and both sensitivity and selectivity have been improved. It is a sensitive determination method of nitrite. In spectrofluorimetric method, nitrite ion is either chemically reactive or a catalyst for several types of chemical reactions on products, with a subsequent effect on the fluorescence properties (development, inhibition, or enhancement) in either a direct or an indirect way. Difluoroboradiaza-*s*-indacenes (BODIPY) are a class of highly rigidized, and polymethine-like fluorescent dyes that have been found numerous applications in biochemistry and molecular biology because of their high extinction coefficients, high fluorescence quantum efficiency [36] and stability to light. 1,3,5,7-Tetramethyl-2,6-dicarbethoxy-8-(3′,4′-diaminophenyl)-difluoroboradiaza-*s*-indacence (TMDCDABODIPY) is a novel fluorescence increasing probe. When it reacts with nitrite to produce corresponding triazole, the fluorescence of the fluorophore is strongly increased. This gives it very good sensitivity and selectivity in determination of nitrite. In a HCl medium and at room temperature, TMDCDABODIPY reacted with nitrite and yielded a strong fluorescent triazole. The determination of nitrite was based on the alteration of relative fluorescence intensity. The florescence intensity was linear over a nitrite concentration of 9–300 nmol $l^{-1}$ with a detection limit of 0.21 nmol $l^{-1}$ (S/N = 3). We firstly used it in the determination of trace nitrite in food products. The method offered several distinct advantages over other fluoremetric methods for detecting nitrite: high sensitivity and excellent specificity and photostability and ease of operation, which has been proved to be appropriate for determination of nitrite in food products.

## 2. Materials and methods

### 2.1. Apparatus

A RF-5000 spectrofluorimeter (Shimadzu, Japan) equipped with a 1 cm × 1 cm quartz cell was employed for the fluorescence intensity measurements. The shift widths in terms of wavelength were both 3 nm for excitation and emission. Absorption spectra were recorded with a Shimadzu (Kyoto, Japan) UV-1601 spectrophotometer. A DF-801 pH meter (Zhongshan University, China) was used.

### 2.2. Reagents

All chemicals used were of analytical reagent grade that obtained from Shanghai Chemical Reagent Co. (Shanghai, China). Meat products and fresh vegetables were purchased from local markets. All solutions were prepared with double-distilled water. TMDCDABODIPY (synthesized according to Refs. [37–39]) solution ($5 \times 10^{-4}$ mol $l^{-1}$) was prepared in ethanol.

A standard nitrite solution ($1 \times 10^{-5}$ mol $l^{-1}$) was prepared by drying sodium nitrite at 110 °C for 4 h and dissolving it in water. Twenty drops of chloroform and a pellet of sodium hydroxide were added in order to prevent liberation of nitrous acid and to inhibit bacterial growth and thus make the nitrite solution stable [40]. This standard solution was prepared weekly and kept in a refrigerator, and further dilution was made daily as required.

### 2.3. Methods

#### 2.3.1. Determination of nitrite

Transfer 1.2 ml of TMDCDABODIPY solution ($5 \times 10^{-6}$ mol $l^{-1}$), 0.5 ml of an assay solution of nitrite ($4 \times 10^{-6}$ mol $l^{-1}$) and 1.0 ml of 1.0 mol $l^{-1}$ HCl solution into a 10 ml volumetric flask. Dilute the mixture to the volume of 5 ml with water and leave to stand for 15 min at 30 °C. Render alkaline with 0.51 ml of 2.0 mol $l^{-1}$ sodium hydroxide. Dilute the whole solution to the volume of 10 ml with water, mixed well and measure the fluorescence intensity at $\lambda_{ex}/\lambda_{em}$ = 500/510 nm using a blank prepared in the same way, by omitting nitrite solution.

#### 2.3.2. Preparation of samples

The preparation procedure for samples was performed as described in Ref. [41]. Fresh vegetables (including cabbage) were cleaned and dried with a blower in room temperature for 20 min, then 10 g of which were weighed in a beaker. About 100 ml double-distilled water was added and incubated for 30 min in a warm water bath at 40 °C. After homogenizing in blender for 2 min, the volume was diluted to 250 ml. After being filtered through a Whatman filter paper, 1 ml of the solution was transferred into a 10 ml volumetric flask and diluted with water, and was determined with the method described above.

About 10 g cabbage was cleaned and dried with a blower in room temperature for 20 min, then immerged in 100 °C 100 ml water for 10 min. Then it was homogenized in blender for 2 min and was diluted to 250 ml with double-distilled water. After the same filtration process, 1 ml of the solution was transferred into a 10 ml volumetric flask and diluted with water, and was determined with the method described above.

Meat products (10 g) were weighed in a beaker. Double-distilled water (100 ml) was added and blended for 5 min in a

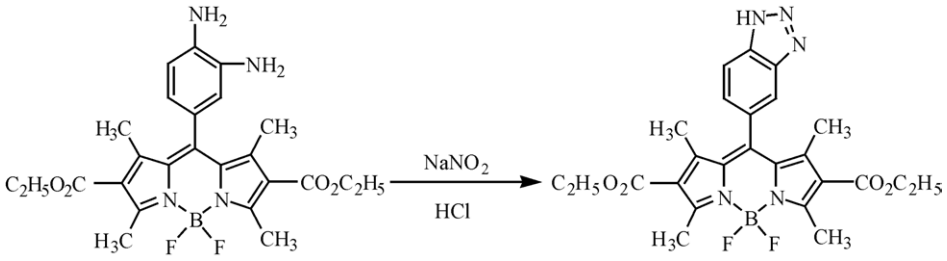


Fig. 1. Reaction of TMDCDBODIPY with nitrite.

laboratory blender. The suspension was incubated for 15 min in a warm water bath at 50 °C. After cooling, the volume was diluted to 250 ml with double-distilled water. After filtered through a Whatman filter paper, 1 ml of the solution was transferred into a 10 ml volumetric flask and diluted with water, and was determined with the proposed method.

For recovery experiments, the known amount of nitrite solutions was added to the second portions of earlier solutions of every sample. After the same filtration process, 1 ml of the solution was transferred into a 10 ml volumetric flask and diluted with water, and was determined with the proposed method.

## 3. Results and discussion

### *3.1. Nitrite reacted with TMDCDABODIPY and yielded an intensely fluorescent triazole derivative, TMDCDABODIPY-T*

Fig. 1 shows the conversion of TMDCDABODIPY to TMDCDABODIPY-T. The fluorescence spectra of TMDCDABODIPY and its product, TMDCDABODIPY-T, the formed triazole with NO, were shown in Fig. 2. The maximum excitation wavelength of TMDCDABODIPY was at 495 nm and its emission wavelength was at 505 nm. The maximum excitation wavelength of the triazole was at 500 nm following the emission wavelength at 510 nm.

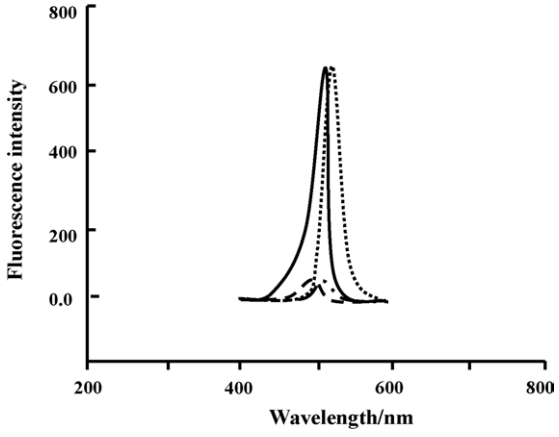


Fig. 2. Fluorescence spectra of TMDCDABODIPY and the triazole: $C_{TMDCDABODIPY} = 5 \times 10^{-6}$ mol l$^{-1}$, $C_{NO_2^-} = 2 \times 10^{-5}$ mol l$^{-1}$; —, excitation spectrum of triazole emitted at 510 nm; ···, emission spectrum of triazole excited at 500 nm; - - -, excitation spectrum of the TMDCDABODIPY emitted at 505 nm; ···, emission spectrum of the TMDCDABODIPY excited at 495 nm. The slit of excitation and emission are both 3 nm.

The fluorescence quantum yields of TMDCDABODIPY and its derivatives were also studied according to Ref. [42]. The fluorescence emission spectra of the sample solution (TMDCDABODIPY) and its derivative in water and the standard solution (fluorescein in 0.1 mol l$^{-1}$ NaOH, $\Phi = 0.92$) were recorded at an excitation wavelength of 488 nm. The fluorescence quantum yield was determined using the expression: $\Phi_u = \Phi_s(D_u A_s)/(D_s A_u)$, where $\Phi_u$, $\Phi_s$ were the fluorescence quantum yield of samples and the standard solution, respectively; $D_u$ and $D_s$ were the areas under the emission curves of the samples and the standard, respectively; and $A_u$, $A_s$ were the absorbance of the samples and the standard, respectively. For fluorescence efficiency measurements, the concentrations of the solutions were adjusted so that the absorbance was less than 0.1, to minimize error arising from inner filter effects. The fluorescence of TMDCDABODIPY was very weak ($\Phi_{fl} = 0.0020$). When it reacted with nitrite, the fluorescence increased greatly because of the formation of the triazole ($\Phi_{fl} = 0.58$).

In this work, the stability of TMDCDABODIPY and its derivatives at room temperature was also investigated according to the literature [43]. It was performed by irradiating a $4.5 \times 10^{-7}$ mol l$^{-1}$ sample using a lamp with a 200 W soft white bulb (General Electric, China) positioned 10 cm from the sample flask. The flask was cooled with flowing room temperature water. Fluorescence spectra were recorded on aliquots taken at different exposure times. After exposed for about 60 h, the fluorescent of TMDCDABODIPY and its derivatives exhibited slightly change.

### *3.2. Optimization of reaction condition of TMDCDABODIPY with nitrite*

The reaction of TMDCDABODIPY with nitrite was mainly affected by reagent concentration, acidity, time and temperature.

It was observed that the relative fluorescent intensity was stationary at a concentration which ranged from $5 \times 10^{-7}$ to $7 \times 10^{-7}$ mol l$^{-1}$ in Fig. 3a. A $6 \times 10^{-7}$ mol l$^{-1}$ of TMDCDABODIPY solution was chosen as optimal for at which the highest sensitivity was obtained.

As the acid was an important factor for the reaction of nitrite with TMDCDABODIPY, their effects on the relative fluorescence intensity had been investigated. The effect of concentration of HCl was studied in the range of 0.020–0.16 mol l$^{-1}$ (Fig. 3b). It was found that the relative fluorescent intensity was

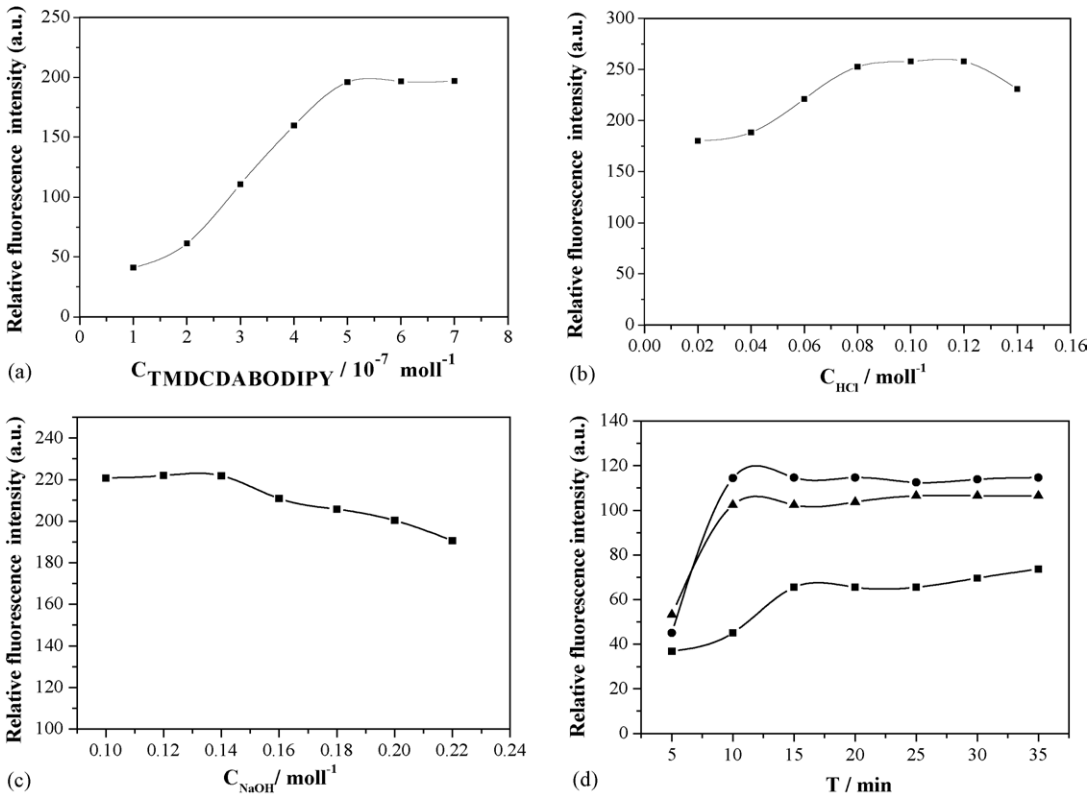


Fig. 3. Effect of some factors on the reaction and the relative fluorescence intensity. (a) Effect of TMDCDABODIPY concentration, $C_{NO_2^-} = 2 \times 10^{-7}$ mol l$^{-1}$; $C_{HCl} = 0.1$ mol l$^{-1}$; $C_{NaOH} = 0.12$ mol l$^{-1}$; reaction time, 15 min; reaction temperature, 30 °C. (b) Effect of HCl concentration, $C_{TMDCDABODIPY} = 6 \times 10^{-6}$ mol l$^{-1}$; $C_{NO_2^-} = 2 \times 10^{-7}$ mol l$^{-1}$; $C_{NaOH} = 0.12$ mol l$^{-1}$; reaction time, 15 min; reaction temperature, 30 °C. (c) Effect of NaOH concentration, $C_{TMDCDABODIPY} = 6 \times 10^{-6}$ mol l$^{-1}$; $C_{NO_2^-} = 2 \times 10^{-7}$ mol l$^{-1}$; $C_{HCl} = 0.1$ mol l$^{-1}$; reaction time, 15 min; reaction temperature, 30 °C. (d) Effect of reaction time and temperature, $C_{TMDCDABODIPY} = 6 \times 10^{-6}$ mol l$^{-1}$; $C_{NO_2^-} = 2 \times 10^{-7}$ mol l$^{-1}$; $C_{HCl} = 0.1$ mol l$^{-1}$; $C_{NaOH} = 0.12$ mol l$^{-1}$, ■, 20 °C; ●, 30 °C; ▲,40 °C.

constant at a concentration ranged from 0.080 to 0.12 mol l$^{-1}$. Finally, 0.10 mol l$^{-1}$ HCl was used.

We found that two amino of TMDCDABODIPY could react with HCl and this could lead to fluorescence increasing, and then induced relatively great interference. We added appropriate capacity alkali to eliminate the interference. When the effect of sodium hydroxide concentration was evaluated from 0.10 to 0.22 mol l$^{-1}$, it could be seen from Fig. 3c that the values of the relative fluorescent intensity had no obvious change in the range of 0.10–0.14 mol l$^{-1}$. Then 0.12 mol l$^{-1}$ of sodium hydroxide was chosen for further investigation.

Time and temperature were both critical factors in the reaction of TMDCDABODIPY and nitrite. The effect of time and temperature was indicated in Fig. 3d. When the reaction was carried out at 30 °C, the relative fluorescent intensity was maximum. The reaction temperature at 30 °C was used. The relative fluorescent intensity showed relatively stable after 15 min. The reaction time of 15 min was used.

### 3.3. Linearity, sensitivity and precision

According to the proposed method, a calibration curve was constructed. The relative fluorescent intensity was proportional to nitrite concentration in the range of $0.090 \times 10^{-7}$ to $3.00 \times 10^{-7}$ mol l$^{-1}$ ($\gamma = 0.9996$). The concentration of nitrite was calculated from the linear regression equation: $Y = 521.57x + 114.87$. $Y$ was the relative fluorescent intensity and $x$ was the concentration of nitrite ($1 \times 10^{-6}$ mol l$^{-1}$). The relative standard deviation ($n = 10$) was 2.40% at a nitrite concentration of $1.0 \times 10^{-7}$ mol l$^{-1}$. The limit of detection of 0.21 nmol l$^{-1}$ nitrite was calculated with the signal to noise ratio (S/N) of 3.

### 3.4. Effect of foreign ions

The influence of common ions normally found in food samples was examined under the selected conditions. It was found that 14 cations, anions and complexing agents had negligible interference in the determination of $2 \times 10^{-7}$ mol l$^{-1}$ nitrite, when present in relatively large to moderately large mass excess. The tolerance limit of an ion was taken as the maximum amount causing an error of ≤5% in the fluorescence of sample. The study revealed that, 10 000-fold excesses of $Mg^{2+}$, $Ca^{2+}$ and nitrate, 1000-fold excesses of EDTA, citrate, carbonate, sulphate, phosphate, $Cu^{2+}$, $Zn^{2+}$, $Br^-$, $I^-$, 100-fold excesses of $Pb^{2+}$ had negligible interference. After being masked up with 0.10 ml of 1% EDTA, 1000-fold excesses of $Fe^{3+}$ showed negligible interference to the determination.

Table 1
Analytical results of nitrite in cabbage with TMDCDABODIPY

| Sample | Added ($mg\,kg^{-1}$) | Found ($mg\,kg^{-1}$) | R.S.D (%) ($n=6$) | Recovery (%) |
|---|---|---|---|---|
| Vegetable (flesh cabbage kept in refrigerator) | | | | |
| After 0.0 h | 0 | 0.87 | 1.72 | 104.31 |
| | 1 | 1.9118 | 2.70 | |
| After 24 h | 0 | 2.39 | 2.85 | 94.62 |
| | 3 | 5.22 | 1.46 | |
| After 36 h | 0 | 5.57 | 3.40 | 102.70 |
| | 5 | 10.70 | 2.64 | |
| After 48 h | 0 | 9.23 | 1.62 | 95.82 |
| | 10 | 18.82 | 3.95 | |
| Vegetable (hard-boiled cabbage kept in refrigerator) | | | | |
| After 0 h | 0 | 0.89 | 2.45 | 97.74 |
| | 1 | 1.86 | 3.12 | |
| After 24 h | 0 | 3.18 | 1.42 | 98.82 |
| | 3 | 6.15 | 4.02 | |
| After 36 h | 0 | 7.11 | 3.60 | 104.64 |
| | 7 | 14.43 | 2.84 | |
| After 48 h | 0 | 11.51 | 2.74 | 103.40 |
| | 12 | 23.92 | 1.15 | |

### 3.5. Sample analysis

The proposed method had been applied to the determination of trace nitrite in the food products including fresh vegetables and meats. All samples were analyzed for six times in the same recommended procedure and conditions. The results were shown in Table 1 and Table 2. From Table 1 we could see that with the time vegetables were kept in refrigerator increasing, the content of nitrite obviously increased. Kept in refrigerator 48 h later, both nitrite contents of fresh vegetable (cabbage) and hard-boiled vegetable (cabbage) increased above 10 times. Especially the hard-boiled vegetable, its increasing extent was more than that of the fresh vegetable. This reminded us cooked food could not be kept too long even in the refrigerator. From Table 2, we can see that the nitrite in the other vegetable was comparatively high. The reason may be that the vegetable had been added some additives during the preservation or the time that spent on the transportation was too long.

Table 2
Analytical results of nitrite in food products with TMDCDABODIPY

| Sample | Added ($mg\,kg^{-1}$) | Found ($mg\,kg^{-1}$) | R.S.D (%) ($n=6$) | Recovery (%) |
|---|---|---|---|---|
| Cucumber | 0 | 4.19 | 2.68 | 102.35 |
| | 5 | 9.31 | 2.42 | |
| Potato | 0 | 4.30 | 1.92 | 96.48 |
| | 5 | 9.12 | 2.36 | |
| Tomato | 0 | 3.96 | 2.32 | 99.10 |
| | 5 | 8.92 | 1.45 | |
| Garlic | 0 | 4.22 | 2.65 | 98.56 |
| | 5 | 9.15 | 2.45 | |
| Onion | 0 | 4.68 | 1.89 | 97.52 |
| | 5 | 9.56 | 3.72 | |
| Beef | 0 | 4.18 | 3.24 | 105.48 |
| | 5 | 9.46 | 2.19 | |
| Eggplant | 0 | 9.34 | 2.68 | 94.78 |
| | 10 | 18.82 | 3.64 | |
| Cheese | 0 | 16.34 | 1.76 | 96.71 |
| | 10 | 26.01 | 1.38 | |
| Sausage | 0 | 29.83 | 2.54 | 103.45 |
| | 10 | 40.17 | 3.14 | |
| Ham | 0 | 30.79 | 3.12 | 98.62 |
| | 10 | 40.65 | 4.25 | |

Table 3
Comparison of detection limits for spectrofluorimetric determinations of nitrite with different reagents

| Reagents | Detection limit ($ng\,ml^{-1}$) | References |
|---|---|---|
| 2,3-Diaminonaphthalene | <0.3 | [33] |
| 2,6-Diaminopyridine | 2 | [45] |
| Resorcinol | 33 | [46] |
| 5-Aminofluorescein | 0.5 | [47] |
| 4-Hydroxycoumarin | 3 | [34] |
| Tryptophan | 1 | [48] |
| 5,6-Diamino-1,3-naphthalene disulphonic acid | 0.09 | [44] |
| TMABODIPY | 0.03 | [49] |
| TMDABODIPY | 0.014 | [50] |
| TMDCDABODIPY | 0.0097 | This paper |

## 4. Conclusion

A new reagent, 1,3,5,7-tetramethyl-2,6-dicarbethoxy-8-(3′,4′-diaminophenyl)-difluoroboradiaza-*s*-indacene (TMDCDABODIPY), had been used in the spectrofluorimetric determination of trace nitrite in the food products. The advantage of the proposed method was that the reaction of TMDCDABODIPY with nitrite was carried out in a short time in aqueous medium without an extraction procedure, and it offered the advantages of specificity, sensitivity and simplicity. The sensitivity of the method was higher than other spectrofluoremetric methods (see Table 3).

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