



# A simple and highly sensitive assay of perfluorooctanoic acid based on resonance light scattering technique



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## ABSTRACT

A simple, highly sensitive resonance light scattering (RLS) method for the detection of perfluorooctanoic acid (PFOA) has been developed based on the interaction with crystal violet (CV). It was found that PFOA can form complexes with CV in acid medium resulting in remarkable enhancement of the RLS intensity of the system. And the enhanced RLS intensities are in proportion to the concentration of PFOA in the range of 0.1–25.0  $\mu\text{mol/L}$  ( $R^2 = 0.9998$ ), with a detection limit of 11.0 nmol/L ( $S/N = 3$ ). In this work, the optimum reaction conditions and the interferences of foreign substances were investigated. The reaction mechanism between CV and PFOA was also studied by the absorption spectrum and scanning electron microscope (SEM). This method is successfully applied to the determination of PFOA in tap water and Jialing river water samples with  $RSD \leq 4.04\%$ .

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## 1. Introduction

Perfluorinated compounds (PFCs) have been widely used in industrial and commercial areas for more than 50 years as surfactants, polymer additives, fire retardants, and various household products [1,2]. Perfluorooctanoic acid (PFOA) is a typical perfluorochemical, which can be accumulated in biotic environment and show potential toxic effects on organisms [3–5]. Since it is found to be globally distributed, environmentally persistent, bioaccumulative and potentially toxic, PFOA has received increasing global concerns in recent years and also has been proposed as a candidate of persistent organic pollutants (POPs) [6,7]. During the period of 1951 ~ 2004, the total amount of produced PFOA is about 3600 ~ 5700 t worldwide, and about 400 ~ 700 t was discharged into the environment [8]. Moreover, PFOA has higher water solubility than other typical POPs and can transport easily in water environment. Up to now, PFOA has been detected in waste water, surface water, groundwater and even tap water throughout the world [9]. It is very significant for the environmental monitoring and assessment to develop the assay method of PFOA.

The detection methods for PFOA in various environmental matrices have been developed since 1960s [10]. By now, the main analysis techniques of PFOA are GC–MS [11,12], HPLC–MS [13], LC–MS [14], LC–MS–MS [15,16], and so on. Although some of them have high sensitivity, they suffered expensive instruments, complicated operations, time consuming, complicated sample preparation and professional technical

operations staff. Therefore, it is important to develop a simple, sensitive, and low-cost assay method to detect PFOA.

Resonance Light Scattering (RLS) technique is simple, rapid and high sensitivity. It has been widely applied in quantitative analysis of drugs [17,18], nucleic acids [19,20], proteins [21], heavy metal ions [22,23] and PFOS [24,25], etc. However, few papers can be found about PFOA determination based on RLS technique by now. In this work, a novel RLS assay method of

PFOA was developed based on the interaction between PFOA and Crystal Violet (CV), resulting in remarkable enhanced RLS signals. The detection mechanism of PFOA is outlined in Scheme 1. Furthermore, the optimal conditions, hydrodynamic size, absorption spectrum and scanning electron microscope (SEM) were also investigated.

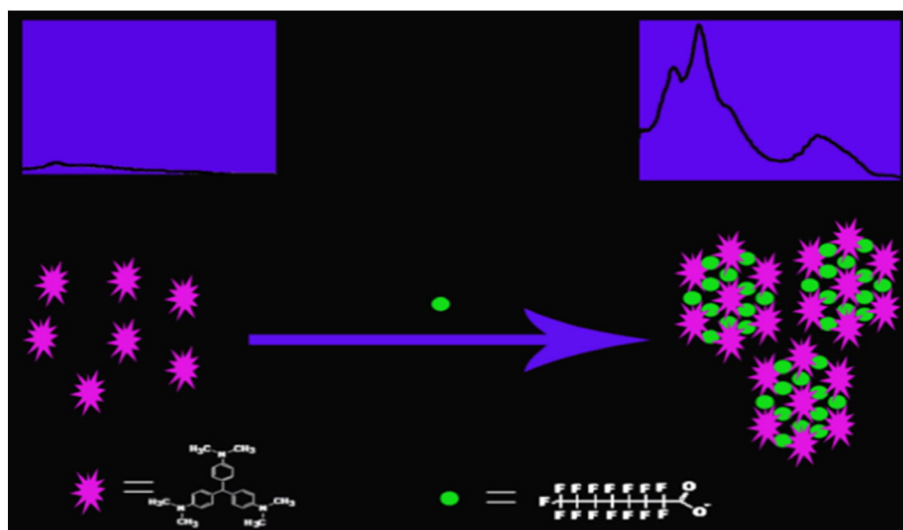
This experiment provides a new possibility for sensitively detecting PFOA in tap water, industrial waste water and other environment water. This method is simple, practical, and cost-effective.

## 2. Experimental

### 2.1. Apparatus

The RLS signal was measured by synchronous scanning at  $\lambda_{\text{ex}} = \lambda_{\text{em}}$  using a Hitachi F-7000 fluorescence spectrophotometer (Tokyo, Japan) with a 150 W xenon lamp and a 1 cm quartz cell. The absorption spectra were recorded by using a Shimadzu UV-2600 spectrophotometer (Tokyo, Japan). The SEM images of system were performed with a Hitachi S-4800 scanning electron microscope (Tokyo, Japan). The hydrodynamic size was measured by using a Zetasizer Nano (Malvern, England). A FE20/EL20 pH meter (Shanghai, China) was used to

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**Scheme 1.** Schematic illustration of the detection of PFOA in the system of RLS.

measure the pH values of the aqueous solution and a vortex mixer QL-901 (Haimen, China) was used to blend the solution.

## 2.2. Reagents

PFOA standard substance (purity greater than 98.0%) was purchased from TCI Development Co., Ltd. (Shanghai, China). CV standard substance (purity greater than 90.0%) was obtained from Aladdin Reagent Co., Ltd. (Shanghai, China). Britton–Robinson (BR) buffer solution was used to control the acidity of the system. All other routine reagents are of analytical grade without further purification. Ultrapure water (18.2  $\Omega$ M) was used throughout.

## 2.3. Procedures

40  $\mu$ L 50  $\mu$ mol/L CV and 200  $\mu$ L BR buffer (pH 3.23) were first pipetted into a 2.0 mL vial. Subsequently, an appropriate volume of PFOA working solution was added, diluted to 2.0 mL with ultrapure water and vortex-mixed thoroughly. The mixture was placed for 10 min and then transferred for RLS measurement and absorption measurement.

## 3. Results and discussion

### 3.1. Spectral characteristics

The RLS spectra of CV-PFOA system were investigated. As shown in Fig. 1, the system exhibits weak RLS peaks in the absence of PFOA. However, the remarkable enhanced RLS signals characterized at 277.0 nm, 320.0 nm and 507.0 nm are observed in the presence of PFOA. All of the RLS signals increase with the increasing concentration of PFOA in this system. The enhanced RLS intensity is proportional to the concentration of PFOA with a range of 1.0 ~ 20  $\mu$ mol/L (peaked at 277.0 nm), 0.10 ~ 25.0  $\mu$ mol/L (peaked at 320.0 nm) and 1.0 ~ 20  $\mu$ mol/L (peaked at 507.0 nm), respectively. The enhancement of RLS signals can be due to the formation of ion-association particles based on the interaction between PFOA and CV by electrostatic attraction. From the insets of Fig. 1, it is easy to see that the RLS intensity peaked at 320.0 nm is the most sensitive with the increasing of PFOA concentration among that at 277.0 nm and 507.0 nm. All RLS signals were measured at 320.0 nm in this experiment.

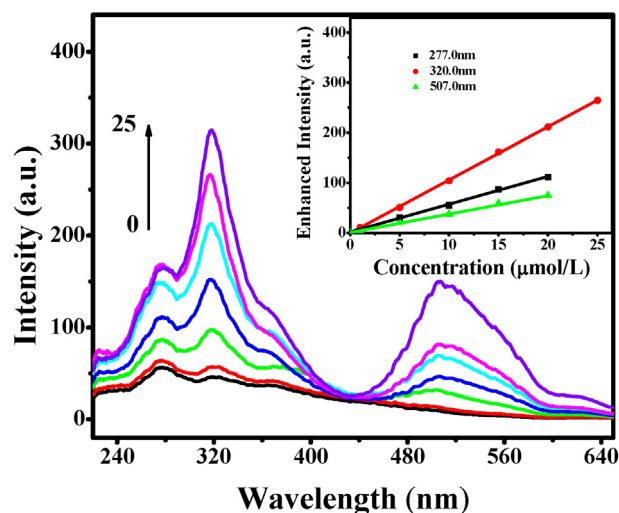
### 3.2. Mechanism

Known from the resonance Rayleigh scattering formula, [26]

$$I = \frac{24\pi^3 N v^2}{\lambda^4} \left( \frac{n_1^2 - n_0^2}{n_1^2 + 2n_0^2} \right)^2 I_0 \quad (1)$$

the scattering light intensity ( $I$ ) is proportional to the number of ions ( $N$ ) per unit volume, which is the basis of quantitative analysis. Besides, it is also proportional to the square of particle volume ( $v^2$ ), which further demonstrates the importance of particle size on light scattering [26].

The SEM images of CV and CV-PFOA were shown in Fig. 2. Compared with CV (Fig. 2a), the CV-PFOA complex (Fig. 2b) generated a large number of irregularly shaped particles. This may be due to the interaction between CV and PFOA by electrostatic attraction. The increase of the molecular size is a significant factor for the increase of RLS intensity



**Fig. 1.** RLS spectra of CV in the absence and presence of different concentrations of PFOA and the linear graph of PFOA (inset). The inset shows that the scattering intensity varies with the increasing concentration of PFOA at 277.0 nm, 320.0 nm and 507.0 nm, respectively. Concentration: CV, 1.0  $\mu$ mol/L; PFOA, 0, 1.0, 5.0, 10.0, 15.0, 20.0, 25.0  $\mu$ mol/L; pH 3.23.

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