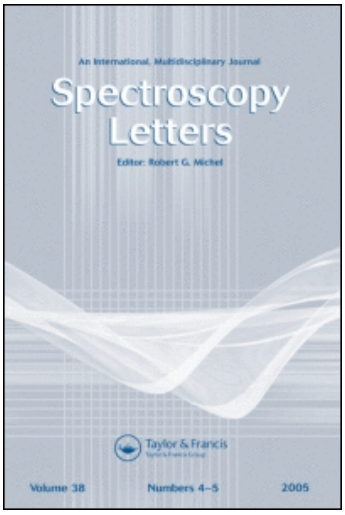


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# Determination of Trace Bismuth by a Solid Substrate Room Temperature Phosphorescence Quenching Method Based on Anionic Polymeric Acrylic Acid Lead Particles Containing Luminescent Eosine Molecules

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**Abstract:** Luminescent particles of Anionic Polymeric Acrylic Acid Lead ($Pb(PAA)_2$) which containing eosine($HFInBr_4$), Eosine nanomicroball of anionic polypropylene acid lead contained luminophores (short for E.N.PAA.L.C.L), were synthesized by a sol-gel method, using anionic polymeric acrylic acid sodium

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(PAANa) as the precursor and $Pb^{2+}$ as the precipitant. $NH_4Ac$-HAc can react with the $Pb^{2+}$ in E.N.PAA.L.C.L, causing it to decompose into aqueous soluble components. E.N.PAA.L.C.L- $NH_4Ac$-HAc can emit strong and stable solid substrate room temperature phosphorescence (SS-RTP) on filter paper, and bismuth, mercury or iodine can cause a decrease in phosphorescence intensity. Based on the facts above, a new method for the determination of trace bismuth by an SS-RTP quenching method was established. The linear range of this method was 0.01–0.20(pg $spot^{-1}$) ($4.0 \times 10^{-12}$ g $ml^{-1}$) of $Bi^{3+}$, with a detection limit (LD) of 0.0016 pg $spot^{-1}$, and the regression equation of the working curve was $\Delta Ip = 61.01 + 237.8\, m_{Bi3+}$ (pg $spot^{-1}$), $r = 0.9992$. This method was applied to the determination of trace bismuth in human hair and well water samples with satisfactory results. The mechanism of SS-RTP emission was also discussed.

**Keywords:** Eosine luminescent nanospheres, anionic polymeric acryclic acid lead, Bismuth solid substrate room temperature phosphorescence quenching



## INTRODUCTION

There are several methods for the determination of trace bismuth, such as $BiI_4^-$ together with Rhod.B, butyl-Rhod.B, Rhod.6G[$\varepsilon = 1.1 \sim 1.3$ ($\times 10^5$) $l \cdot mol^{-1} \cdot cm^{-1}$)[1] and alkaline azo-dye ($\varepsilon = 9.2 \times 10^4 \cdot mol^{-1} \cdot cm^{-1}$)[2] ionic associate photometry; 3-nitrophenylfluorone ($\varepsilon = 5.0 \times 10^4\, l \cdot mol^{-1} \cdot cm^{-1}$);[3] dithizone ($\varepsilon = 7.9 \times 10^4\, l \cdot mol^{-1} \cdot cm^{-1}$);[4] amplification of iodide starch($\varepsilon = 3 \times 10^5\, l \cdot mol^{-1} \cdot cm^{-1}$).[5] In recent years many methods for the determination of trace bismuth have been reported, such as potassium iodide-crystal violet inhibited photometry ($\varepsilon = 1.07 \times 10^5\, l \cdot\ mol^{-1} \cdot cm^{-1}$)[6] and cetyltrimethylammonium bromide-hydrogen peroxide-litchi Chinese red inhibited photometry ($\varepsilon = 9.3 \times 10^5\, l \cdot mol^{-1} \cdot cm^{-1}$);[7] hydrogen peroxide-Eosine Y(LD:$2.0 \times 10^{-11}$ g $ml^{-1}$);[8] adsorptive stripping voltammetry with a pyrogallol red modified carbon paste electrode (LD:$5.0 \times 10^{-10}$ g $ml^{-1}$).[9] But the $\varepsilon$ of these methods above only reached the $10^5$ level, and the lowest detection limit was only $10^{-11}$ g $ml^{-1}$, some are complex to use. There are some reports that refer to the use of eosine as the luminescent reagent, such as inclusion, and luminant properties, of eosin Y in mesoporous molecular sieve, MCM-41, film and powder[10] and the effects of different surfactants on the behavior of AgCl nano-particles in reversed micelles.[11] Using PAANa as precursor, $HFInBr_4$ as luminescence reagent and $Pb^{2+}$ as precipitant, E.N.PAA.L.C.L nano-particles were synthesized by a sol-gel method. $NH_4Ac$-HAc can react with the $Pb^{2+}$ in E.N.PAA.L.C.L, causing it to decompose into aqueous soluble components. E.N.PAA.L.C.L- $NH_4Ac$-HAc can emit strong and stable solid substrate room temperature phosphorescence (SS-RTP) on filter paper and bismuth, mercury or iodine can cause a decrease in phosphorescence intensity. Based on the facts above, a new method for the

determination of trace bismuth, mercury or iodine by SS-RTP quenching method was established. For $Bi^{3+}$, the linear range of this method was 0.01-0.20(pg $spot^{-1}$), with a detection limit (LD) of 0.0016 pg $spot^{-1}$ (corresponding concentration is $4.0 \times 10^{-12}$ g $ml^{-1}$), which is 20 times lower than the lowest detection limit[8] reported before. This sensitive, rapid and repeatable method has been applied to the determination of trace bismuth in human hair and well water samples successfully.

## EXPERIMENT

### Apparatus and Reagents

Perkin Elmer LS-55 luminescence spectrophotometer (Main parameters are: delay time: 0.1 ms, gate time: 2.0 ms, cycle time: 20 ms, flash count: 1; excitation slit: 10 nm, Emission slit: 10 nm, scan speed: 1500 nm $min^{-1}$); pHS-3B precision acidometer (Shanghai Medical Laser instrument plant); 85-1 constant temperature magnetic stirrer (ShenZhen Tian-nan-hai-bei company); AE240 electronic analytical balance (Mettler-Toledo instruments Shanghai company).

$Bi^{3+}$ stock reagent: 1.0 mg $ml^{-1}$ $Bi^{3+}$ primary standard reagent (GSBG 62069-90 8101); 1.0 μg $ml^{-1}$ $Bi^{3+}$ working solution (diluted from $Bi^{3+}$ primary standard reagent, and it is diluted to 1.0 ng $ml^{-1}$ before use); $1.0 \times 10^{-3}$ mol $l^{-1}$ $HFInBr_4$ stock reagent: 0.35 g $HFInBr_4$ powder was dissolved in 95%$C_2H_5OH$, then it was diluted to 500 ml with water; $NH_4Ac$-HAc (pH = 5.9). All the reagents are A.R grade except $Bi^{3+}$ was a primary standard. The water used was prepared using three passes through a sub-boiling still.

Filter paper (HangZhou Xin-Hua paper corporation) was precut into small wafers (Φ(diameter) = 15 mm), with a ring indentation (Φ = 4.0 mm) at the center of each wafer made by a standard pinhole plotter; Acetylcellulose membrane, nitrocellulose membrane and polyamide membrane (Lu-qiao-si-jia biochemical plastic plant).

### Synthesis of E.N.PAA.L.C.L Nano-Particle

To 100.0 ml of 3% PAANa, 100.0 ml of $HFInBr_4$ ($1.0 \times 10^{-3}$ mol $l^{-1}$) were added, and then 100.0 ml of 1.0 mol $l^{-1}$ $Pb^{2+}$ were added dropwise. Standing for 10 min to ensure precipitate completely, the mixture was centrifuged and the solid was washed by anhydrous alcohol. After dried and grinded, a rose powder of E.N.PAA.L.C.L was obtained. Under these conditions the yield of E.N.PAA.L.C.L nanomicroball synthesized was the highest and the phosphorescence signal was the strongest. The probable reaction of the synthesis

of the E.N.PAA.L.C.L nanomicroball is as following:

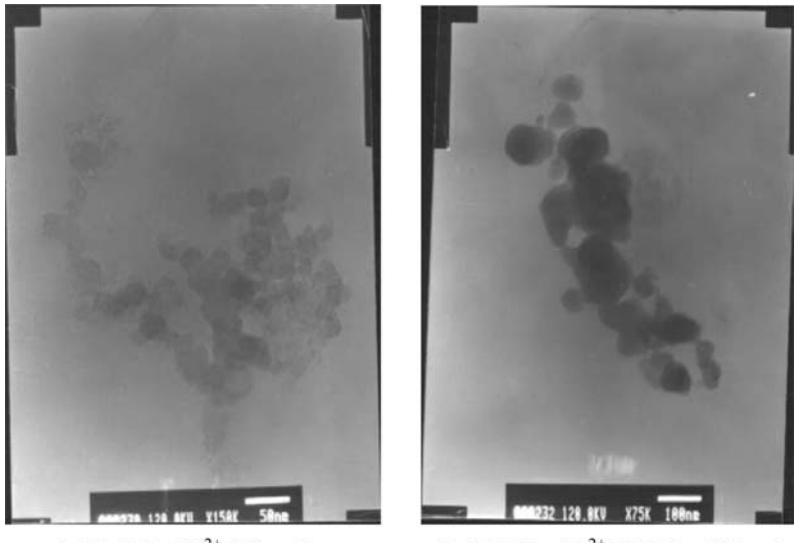
$$n\ PAANa + HFInBr_4 + Pb^{2+} \longrightarrow [(PAA)nPb]^{+} \cdot [FInBr_4]^{-} + n\ Na^{+} + H^{+}$$

## Measurement of Particle Size

The particle sizes of the synthesized nano-particles were measured by a JEM-2000EX transmission electron microscope (TEM, Made by Japan electric company). Results show that the particle size of PAANa-$Pb^{2+}$ and PAANa-$Pb^{2+}$-$HFInBr_4$ were about 50 nm and 100 nm, respectively (Fig. 1).

## Determination of SS-RTP

500($\pm$0.1) mg of E.N.PAA.L.C.L dry rose powder was dissolved by $NH_4Ac$-HAc solution, and then diluted to 10.0 ml. To a 10 ml cuvette, 1.00 ml of above solution and some of Hg, I, Bi were added respectively, diluted to the graduation and mixed homogeneously, then standing at 25°C for 10 min. Filter papers ($\Phi = 15$ mm) were immersed in $Pb(Ac)_2$ solution (1 mol $l^{-1}$) for 10 sec and then dried at 90 $\pm$ 1°C for 2 min. A 0.4 μl drop of test solution was suspended onto the center indentation of the filter paper by a 0.5 μl flat head micrometer syringe and then the paper sheet was dried



1. PAANa -$Pb^{2+}$ (50 nm) 2. PAANa- $Pb^{2+}$-$HFInBr_4$ (50 nm)

***Figure 1.*** TEM micrographs of PAANa-$Pb^{2+}$ and PAANa-$Pb^{2+}$-$HFInBr_4$ nanoparticle.

at 90 ± 1°C for 2 min, at the same time a reagent blank was obtained. The phosphorescence intensity was measured. The signal from the filter paper substrate was defined as the background intensity ($Ip_0$), the signal of E.N.PAA.L.C.L- $NH_4Ac$-HAc system (without $Bi^{3+}$) was defined as reagent blank intensity ($Ip_1$), the signal of the E.N.PAA.L.C.L- $NH_4Ac$-HAc- $Bi^{3+}$ system was defined as the sample intensity for the test solution ($Ip_2$). Then calculate $\Delta Ip$ ($=Ip_1-Ip_2$) and the sample/background ratio ($=Ip_2/Ip_0$).

## RESULTS AND DISCUSSION

### Phosphorescence and Fluorescence Spectra

The ordinary phosphorescence spectra of the blank solution and test solution are shown in Fig. 2, while the ordinary fluorescence spectra are shown in Fig. 3. Results showed that E.N.PAA.L.C.L- $NH_4Ac$-HAc can emit strong and stable solid substrate room temperature phosphorescence (SS-RTP) on filter paper at wavelengths $\lambda_{ex}/\lambda_{em} = 454/621$ nm. When $Bi^{3+}$ was added to the system, the phosphorescence intensity was decreased, but the $\lambda_{ex}/\lambda_{em}$ remained unchanged. The E.N.PAA.L.C.L- $NH_4Ac$-HAc system also can emit fluorescence, with its $\lambda ex/\lambda em$ at 516/543 nm (Fig. 3). There are obvious differences between the fluorescence and phosphorescence

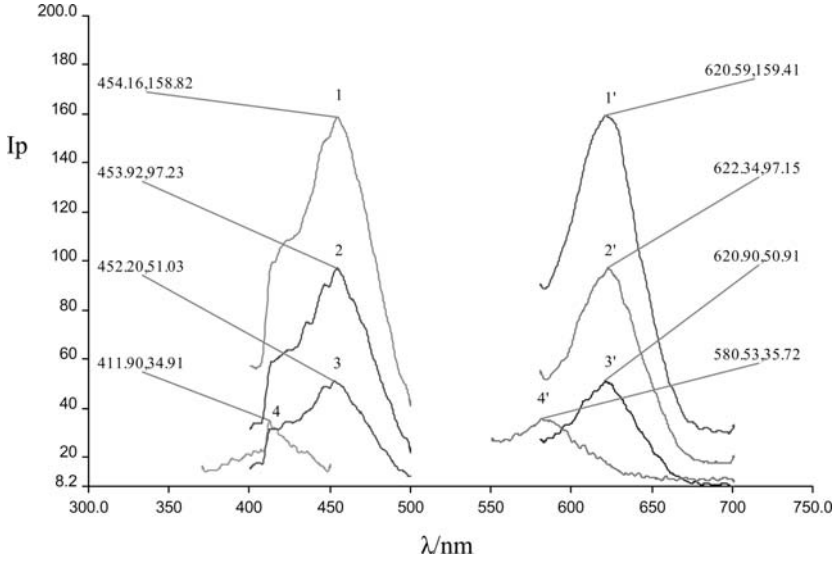



***Figure 2.*** Solid substrate room temperature phosphorescence (SS-RTP) spectra for E.N.PAA.L.C.L- $NH_4Ac$-HAc- $Bi^{3+}$ system. 1.1′ E.N.PAA.L.C.L- $NH_4Ac$-HAc, 2.2′ 1.1′ + 0.25 ng $Bi^{3+}$, 3.3′ 1.1′ + 5.00 ng $Bi^{3+}$, 4.4′ paper.

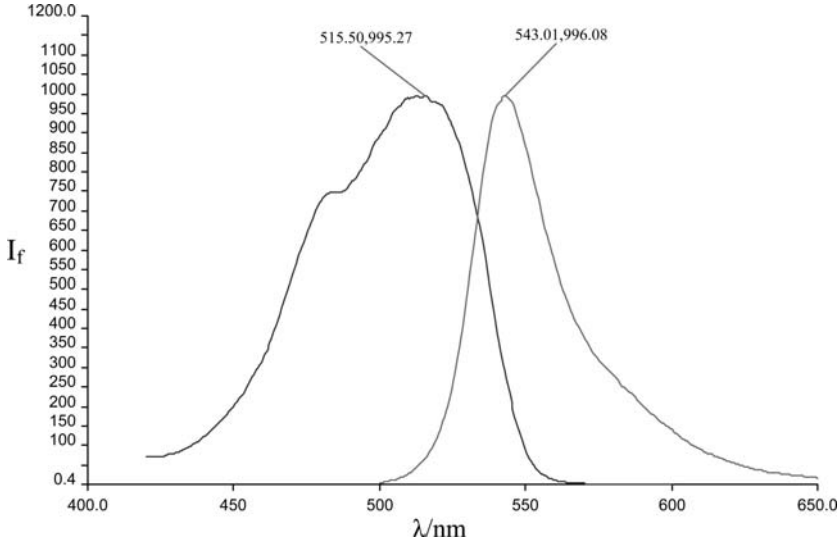



***Figure 3.*** Fluorescence spectra of E.N.PAA.L.C.L- $NH_4Ac$-HAc (Ex Slit 10.0 nm Em Slit 2.5 nm).



spectra in their $\lambda_{ex}/\lambda_{em}$. Therefore, 454/621 nm was chosen in this experiment as the working wavelength for the determination of bismuth. To 5.00 ng $Bi^{3+}$/mL, under $N_2$ Ip is 159.41, 159.42, 159.40, 159.41, 159.41, 159.40. The results showed that whether or not under nitrogen the system is stable.

## Optimum Measurement Condition

### Selecting Solid Substrate

For a system containing 0.10 ng $ml^{-1}$ of $Bi^{3+}$, the phosphorescence intensities of sample (Ip) and background ($Ip_0$) on four kinds of different solid substrates were measured at wavelengths $\lambda_{ex}/\lambda_{em} = 454/622$ nm. Results showed that for filter paper, acetylcellulose membrane and nitrocellulose membrane, the signal/background ratios ($Ip/Ip_0$) were 2.8, 2.5 and 1.9, respectively. So filter paper was chosen as the solid substrate in the following experiment for its highest $Ip/Ip_0$ ratio.

### The Concentration and Volume of Reagent

For the system containing 0.10 ng $ml^{-1}$ of $Bi^{3+}$, When the volume and concentration were 1.00 ml of $1.0 \times 10^{-3}$ mol/L $HFInBr_4$, 1.00 ml of 3%

PAANa solution and NH4Ac-HAc (pH = 5.9), the ΔIp of the system reached max. (The results showed that when the $HFInBr_4$ concentration respectively is 0.50, 0.75, 1.0, 1.25 and 1.50, the system ΔIp value is in turn 60.45, 69.96, 77.80, 76.88 and 75.37; When $1.0 \times 10^{-3}$mol/L $HFInBr_4$ volumes used respectively is 0.50, 1.00, 1.50, 2.00 (ml), the system ΔIp value is in turn 70.65, 77.78, 77.65 and 77.57; When the concentration of PAANa solution respectively is 1.00, 2.00, 3.00, 4.00 and 5.00 (%), the system ΔIp value is in turn 60.69, 71.74, 77.79, 77.25 and 76.83; When the PAANa volumes used 0.50, 0.75, 1.00, 1.25 and 1.50 (ml), the system ΔIp value is in turn 54.62, 70.01, 77.77, 75.07 and 69.12.).

## Acidity

For a system containing 0.10 ng $ml^{-1}$ of $Bi^{3+}$, the effects of acidity on the system was examined. Results showed that the signal/background ratios were 2.8, 2.8, 2.7, 2.6, 2.4 and 2.2 for pH values of 6.4, 5.8, 4.4, 3.8, 3.4 and 3.0, respectively. When the pH was 5.8, the effect of raising the signal/background ratio was the most significant (Fig. 4), so $NH_4$Ac-HAc (pH = 5.9) was choosen as the diluted solution.

## Heavy Atom Effect

For the system containing 0.10 ng $ml^{-1}$ of $Bi^{3+}$, the effects of heavy atoms such as $Pb^{2+}$, $Cu^{2+}$, $Ag^{+}$ on the signal/background ratio of the system were examined. Results showed that the signal/background ratios were 2.8, 2.5, and 2.3, respectively. Among these the ion $Pb^{2+}$ had the most significant perturbation effect (Fig. 5). The effects were examined of 0.25, 0.50, 0.75, 1.0 and 1.25 mol $l^{-1}$ $Pb^{2+}$ on the ΔIp of the system. The results indicated that when $C_{Pb}^{2+}$ was 1.0 mol $l^{-1}$ for the reaction system, the ΔIp of the system reached max.

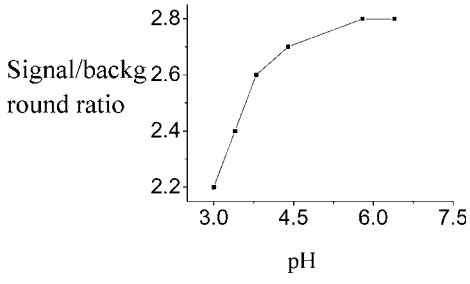



***Figure 4.*** Effect of pH on signal/background ratio for system.

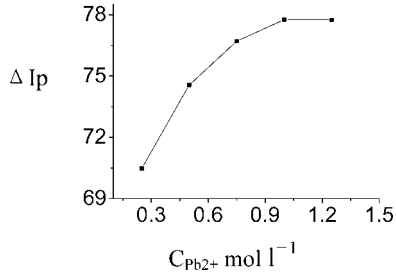



***Figure 5.*** Effect of $C_{Pb}^{2+}$ on ΔIp for system.

Time and Temperature for Reaction

For a system containing 0.10 ng $ml^{-1}$ of $Bi^{3+}$, the time and temperature for the reaction were changed respectively. Results showed that when the time was 10 minutes and temperature was 25°C, ΔIp reached a maximum. (Figs. 6 and 7).

Time and Temperature for Drying

The filter paper was immersed in $Pb(Ac)_2$ solution, after 10 sec, the paper sheet was dried at 90 ± 1°C for 2 min. Then a ring indentation was made and the sample solution was suspended onto the indentation of the paper sheet. The paper sheet was dried at 90 ± 1°C for 2 min. At the time the ΔIp (77.77,77.75) reached max.

## Analytical Parameters

When the amount of $Bi^{3+}$ is in the range of 0.010–0.20 pg $spot^{-1}$ (corresponding to a concentration of $Bi^{3+}$ of 0.025–0.50 pg $\mu l^{-1}$, with a sample volume of 0.4 μl), ΔIp obeyed Beer's law with a detection limit of 0.0016 pg $spot^{-1}$ (based on three times the standard deviation of the

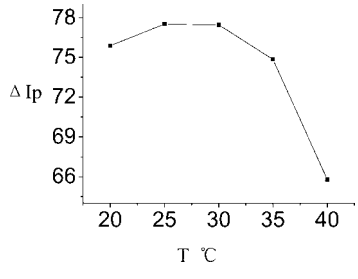



***Figure 6.*** Effect of temperature on ΔIp for reaction system.

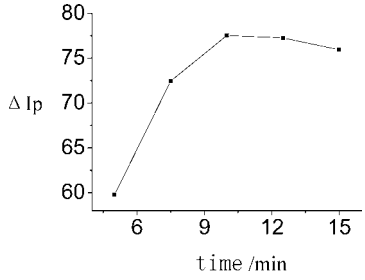



***Figure 7.*** Effect of time on ΔIp for reaction system.

background). The regression equation was $\Delta Ip = 61.01 + 237.8\ m\ B_i^{3+}$ (pg spot$^{-1}$), n = 8, r = 0.9992. For the sample containing 0.010 (pg spot$^{-1}$) $Bi^{3+}$ and 0.20 (pg spot$^{-1}$) $Bi^{3+}$, with a sample volume of 0.4 μl, the relative standard deviation (RSD) were 2.8% and 4.2% (n = 11), respectively.



## The Lifetime of Phosphorescence

The SS-RTP lifetime obtained from the RTP attenuation curve of a sample that contained 0.04 pg spot$^{-1}$ was 55.56 ms (Delay time: 0.1–2.0 ms, Gate time: 2.0 ms). According to the method in literature,[12] the regression equation of the attenuation curve can be expressed as ln Ip = 3.423–0.0180 t (ms) (r = −0.9966).

## Interference Experiment

For sample containing 0.10 ng ml$^{-1}$ of $Bi^{3+}$, the allowed concentration (multiple) of coexistent ions ($E_r$ ±5%) are as following: $NO_3^-$, $Cu^{2+}$(2000, +2.6%, +3.8%); $SO_4^{2-}$, $C_2O_4^{2-}$, $As^{3+}$, $As^{5+}$, $Al^{3+}$, $Cr^{3+}$, $Ca^{2+}$, $Hg^{2+}$(1600, +3.4%, +4.1%, +3.2%, −4.7%, −3.9%, −4.4%, +2.8%, +1.9%); $Ag^+$, $PO_4^{3-}$, $Fe^{3+}$, $NO_2^-$, $NH_4^+$, $ClO_4^-$, $BrO_3^-$, $Br^-$, $SCN^-$, $Ac^-$(1100, +4.6%, +3.7%, −2.2%, +3.8%, +4.5%, +4.4%, −2.7%, −3.6%, −4.9%, +1.4%); $Zn^{2+}$, $Na^+$, $Cr^{5+}$, $Cd^{2+}$, $Fe^{2+}$, $Co^{2+}$, $K^+$(700, +3.5%, −4.0%, −4.5%, −2.8%, +2.3%, +3.6%, −1.7%); $Ni^{2+}$, $Sn^{4+}$, $Hg^{2+}$ (600, +3.5%, +2.4%, + 3.4%); $Tl^+$(500); $CO_3^{2-}$, $SO_3^{2-}$, $S^{2-}$, $Sb^{3+}$(350, −3.7%, −4.2%, +3.9%, +4.2%); $I^-$, $Mn^{2+}$(300, +4.4%, −3.5%); $Mg^{2+}$(250, −2.8%).

## Analysis of Samples

0.10 g (±0.1 mg) of hair sample was washed and dried, then digested by a mixture of $HClO_4$ and $HNO_3$ to colorless, and the resulted solution was

*Table 1.* The analytical results of Bi in hair and well water

| Sample | Present method ($\mu g\ g^{-1}$, $\mu g\ l^{-1}$) | Added ($\mu g\ g^{-1}$, $\mu g\ l^{-1}$) | Obtained ($\mu g\ g^{-1}$, $\mu g\ l^{-1}$) | Recovery (%) | RSD (%) |
|---|---|---|---|---|---|
| Hair | 0.34 | 0.12 | 0.11 | 91.7 | 2.9 |
| Well | 0.023 | 0.43 | 0.42 | 97.7 | 3.1 |

heated to nearly dryness, then the residue was redissolved and diluted to 50.0 ml with water. The content of the $Bi^{3+}$ in samples was measured by the method described above. A standard addition recovery experiment was also conducted. The results are listed in Table 1.

2.00 ml well water was diluted to 50.0 ml with water, and the content of $Bi^{3+}$ determined according to the method above. Meanwhile, a standard addition recovery experiment was conducted. The results are listed in Table 1.

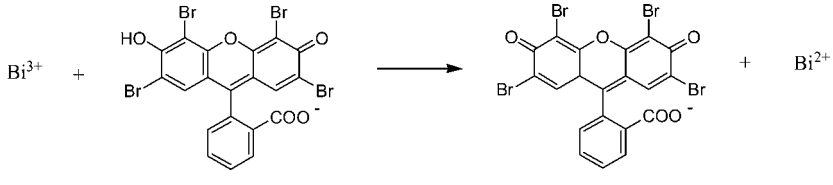


## The Mechanism for SS-RTP Emission

The reaction of E.N.PAA.L.C.L nano-particle handled by $NH_4Ac$-HAc is as following:

$$(PAA)nPb\text{-}\ HFInBr_4 + 2NH_4Ac \longrightarrow Pb(Ac)_2 + n\ PAA^- + HFInBr_4 + 2NH_4^+$$

The PAA- $HFInBr_4$- $NH_4Ac$-HAc system can emit room temperature phosphorescence on a solid substrate; its $\lambda_{ex}/\lambda_{em}$ is 454/621 nm. When the $Bi^{3+}$ is present, $Bi^{3+}$ will oxidize $HFInBr_4$ to a compound that can't emit room temperature phosphorescence signal. The oxidant reaction is probably the following:




It results in a decrease in the solid substrate room temperature phosphorimetry signal, and the $\lambda_{ex}/\lambda_{em}$ remained unchanged. According to the facts above, $Bi^{3+}$ ion can be determined by SS-RTP quenching method.

## ACKNOWLEDGEMENTS

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