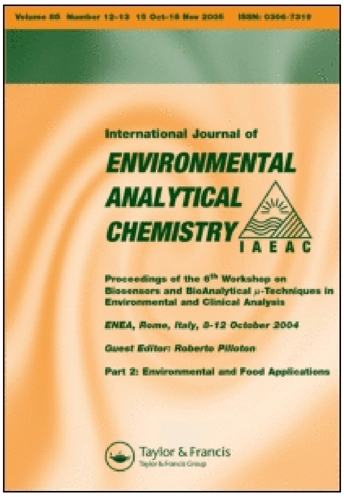

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Catalytic solid substrate-room temperature phosphorimetry for the determination of trace As(V) based on oxidising reaction between hydrogen peroxide and fullerenol using tween-80 as sensitizer

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A new catalytic solid substrate-room temperature phosphorimetry (SS-RTP) for the determination of trace arsenic(V) has been established. It is based on the fact that fullerenol (F-ol) emitted strong and stable room temperature phosphorescence (RTP) on nitric acid cellulose membrane (NCM) substrate. H_2O_2 could oxidise F-ol to cause the quenching of RTP. As(V) could catalyse H_2O_2 to oxidise F-ol and decrease the RTP signal of F-ol sharply. After adding tween-80 in the system, its ΔI_p enhanced 7.7 times compared with the without-tween-80 levels. Under the optimum conditions, the linear dynamic range of this method was 0.016−11.2 ag spot^{-1} with a detection limit (LD) of 9.3 zg spot^{-1} (corresponding concentration: $2.3 \times 10^{-17}\,\mathrm{g\,mL^{-1}}$). This sensitive, simple and selective method has been successfully applied to the determination of trace As(V) in human hair and tea samples. The reaction mechanism for SS-RTP is also discussed.

Keywords: arsenic(V); fullerenol; tween-80; catalytic solid substrate-room temperature phosphorimetry

1. Introduction

In recent years, many methods have been developed for determination of As(V), such as hydride generation atom fluorescence spectrometry (LD: 8.9×10^{-11} g As(V) mL^{-1}) [1], ion chromatography–hydride generation-atomic fluorescence spectrometry (LD: 8.3×10^{-9} g As(III) mL^{-1}) [2], fluorimetric (LD: 6.0×10^{-10}g As mL^{-1}) [3], inhibitory kinetics spectrophotometry (LD: 4.0×10^{-10} g As(V) mL^{-1}) [4], hydride generation-axial view inductively coupled plasma atomic emission spectrometry(ICP-AES) (LD: 1.0×10^{-10} g As(V) mL^{-1}) [5], hydride generation atomic absorption spectrometry (LD: 1.0×10^{-10} g As(V) mL^{-1} [6], 2.5×10^{-10} g As mL^{-1} [7], atomic absorption spectrometry (LD: 2.4×10^{-11}g As(III) mL^{-1}) [8], electrochemical hydride generation

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atomic absorption spectrometry (LD: 2.0×10^{-10} g As(III) mL^{-1}, 5.0×10^{-10} g As (V) mL^{-1}) [9], high performance liquid chromatography–hydride generation–(fast sequential) atomic absorption spectrometry (LD: 7.8×10^{-9} g As(III) mL^{-1}, 1.2×10^{-8} g As(V) mL^{-1}) [10], HG-AAS and ICP-SF-MS (LD: 2.3×10^{-8} g As g^{-1}, 1.4×10^{-9} g As g^{-1}) [11], HPLC–ICP-MS (LD: 2.0×10^{-8} g As(III) mL^{-1}) [12], miniaturized isotachophoresis (LD: 8.5×10^{-7} g As mL^{-1}) [13], tubular potentiometric detector (LD: 4×10^{-6} mol As(V) L^{-1}) [14] and so on. Kinetics spectrophotometry and electrophoresis are of low sensitivity; the operation of atomic fluorescence spectrometry is complicated; ICP-AES, ICP-SF-MS and HPLC–ICP-MS can't be widely used because of their use of expensive instruments. All of these methods are not suitable for the determination of trace As(V) in life samples and air environment. Therefore, searching for a more sensitive, more direct and more accurate analytical method has been a very important focus for domestic and international scholars.

There have been many reports on the syntheses of C_{60} fullerene-cyclodextrin complex, C_{60} fullerene-calixarenes complex, fullerenol (F-ol), water-soluble C_{60} dendrimer [15–17], and fluorescent property of water-soluble F-ol [18]. However, the analytical application of F-ol has been rarely reported.

In our research, we found that F-ol emitted room temperature phosphorescence (RTP) on nitric acid cellulose membrane (NCM) using Li^+ as a perturber, but the signal was so weak that it had no analytical value. Tween-80 could sharply enhance the RTP signal of F-ol. It provided the possibility of the application of F-ol in solid substrate-room temperature phosphorimetry (SS-RTP). In the presence of H_2O_2, F-ol could be oxidised to cause the quenching of RTP. Moreover, As(V) could make the RTP signal of F-ol quench sharply ($\Delta I_p = 110.5$), and the content of As(V) was directly proportional to the value of ΔI_p. Based on these facts, a new catalytic SS-RTP for determination of trace As(V) has been established. The regression equation of working curve could be expressed as $\Delta I_p = 0.7648 + 9.679\,m$ As(V) (ag $spot^{-1}$), with a detection limit of 9.3 zg $spot^{-1}$ As(V) (0.40 µL sample solution per spot, corresponding concentration: 2.3×10^{-17} g mL^{-1}As(V)), indicating the sensitivity was high. For 0.016 and 11.2 ag $spot^{-1}$ As(V), RSD were 4.8% and 3.6%, respectively ($n = 6$). This simple, accurate, selective and sensitive method with good repeatability has been applied to the determination of trace As(V) in human hair and tea samples with satisfactory results. According to the reaction mechanism for determination of trace As(V) by SS-RTP, when As(V) was deoxidised to As(III), the RTP signal of F-ol changed. Based on that, the trace As(III) could be also determined by SS-RTP.

2. Experimental

2.1 *Apparatus and reagents*

Phosphorescent measurements were carried out on Perkin–Elmer LS-55 luminescence spectrophotometer with a front-surface attachment and a solid sample shelf (Perkin-Elmer). The instrument's main parameters are as follows: Ex. Slit: 15.0 nm; Em. Slit: 8.0 nm; scan speed: 1500 nm min^{-1}. The pHS-3B precision acidometer (Shanghai Medical Laser Instrument Plant); 85-1 constant temperature magnetic stirrer (Beijing Taike Instruments Company); AE240 electronic analytical balance (Mettler-Toledo

Instruments Shanghai Company); a 0.50 µL flat head micro-injector (Shanghai Medical Laser Instrument Plant) was used to introduce solution.

Preparation of As(V) working solution (GSBG 62028-90 3302): 1.00 mg mL^{-1} As(V) primary standard solution was diluted to 10.0 or 100.00 fg mL^{-1} as working solution; 1.0×10^{-5} mol mL^{-1} F-ol; 5.0%(W/V) tween-80; 0.30%(V/V) H_2O_2. All reagents are of AR grade, except for As(V). The water used was of thrice sub-boiling distillation.

Filter paper used was purchased from Xinhua paper corporation (Hangzhou, China). Polyamide membrane (PAM), acetylcellulose membrane (ACM) and NCM were all precut into small wafers ($\Phi = 15$ mm) for preparation (Luqiaosijia biochemical plastic plant, Hangzhou, 310000, China).

2.2 *Experimental method*

To a 25-mL colorimetric tube, a certain amount of As(V) working solution, 2.00 mL F-ol, 3.00 mL tween-80 and 1.20 mL H_2O_2 were added, diluted to 25 mL with water, and mixed homogeneously. The tube was kept at 70°C for 20 min, cooled by flowing water for 5 min and placed for determination. The NCM precut wafers were immersed in 1.50 mol L^{-1} LiCl solution for 10 s, then dried at 90 ± 1°C for 2 min. A 0.40 µL drop of test solution was suspended onto the indentation centre of membrane wafers by a 0.50 µL flat head micrometer syringe, and the NCM was dried at 90 ± 1°C for 2 min. A blank test was conducted simultaneously. The phosphorescence intensity was measured at $\lambda_{ex}^{max}/\lambda_{em}^{max} = 469/635$ nm. The signal of system (containing 2.00 mL F-ol, 3.00 mL tween-80 and 1.20 mL H_2O_2) was defined as the reagent blank intensity (I_{p1}), and the signal of test solution (containing a certain amount of As(V), 2.00 mL F-ol, 3.00 mL tween-80 and 1.20 mL H_2O_2) was defined as sample intensity for test solution (I_{p2}). Then ΔI_p ($= I_{p1} - I_{p2}$) was calculated.

3. Results and discussion

3.1 *Phosphorescence spectra*

The phosphorescence spectra of tween-80-F-ol-H_2O_2-As(V) were scanned by the experimental method. Results showed that F-ol could emit weak RTP on NCM at $\lambda_{ex}^{max}/\lambda_{em}^{max} = 479.5/645.9$ nm ($I_p = 55.6$, Figure 1, curve 4.4′). Tween-80 could enhance the RTP signal of F-ol at $\lambda_{ex}^{max}/\lambda_{em}^{max} = 469.2/636.7$ nm ($I_{p\ reagent}/I_{p\ sample} = 55.6/183.4$) with a blue shift of λ_{em}^{max} for 9.3 nm (Figure 1, curve 5.5′). The reason might be that tween-80 could react with F-ol to form micelle complex, making F-ol molecules in the micelle orderly. In the presence of H_2O_2, the RTP signal of F-ol was quenched sharply ($\lambda_{ex}^{max}/\lambda_{em}^{max} = 469.6/637.0$ nm, $I_{p\,reagent}/I_{p\,sample} = 183.4/171.1$, Figure 2, curve 4.4′) and $\lambda_{ex}^{max}/\lambda_{em}^{max}$ remained almost unchanged, which might be explained by the fact that H_2O_2 oxidised F-ol to form none phosphorescence compound. After As(V) being added, the RTP of F-ol was quenched more sharply ($\lambda_{ex}^{max}/\lambda_{em}^{max} = 469.0/635.2$ nm, $I_{p\,reagent}/I_{p\,sample} = 171.1/60.6$, Figure 2, curve 2.2′). Results showed that As(V) catalysed H_2O_2 to oxidise F-ol and the $\lambda_{ex}^{max}/\lambda_{em}^{max}$ stayed almost unchanged, so 470/637 nm was chosen as the working wavelengths to determine trace As(V).

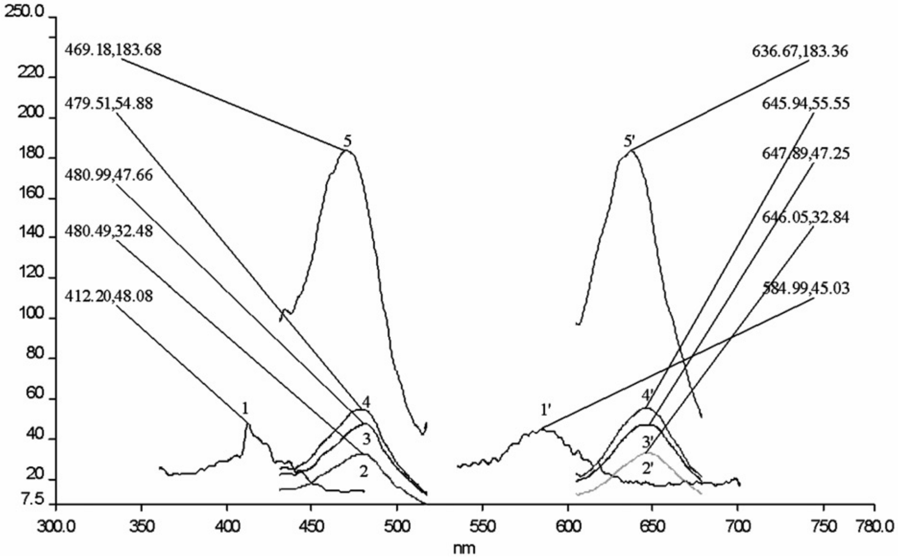

Figure 1. The phosphorescence spectra of F-ol-tween-80, F-ol-H_2O_2 and F-ol-H_2O_2-As(V) system. Notes: 1.1′ : NCM; 4.4′ : 2.00 mL F-ol; 5.5′ : 4.4′ + 3.00 mL tween-80; 3.3′ : 4.4′ + 1.20 mL H_2O_2; 2.2′ : 3.3′ + 700.0 fg As(V).

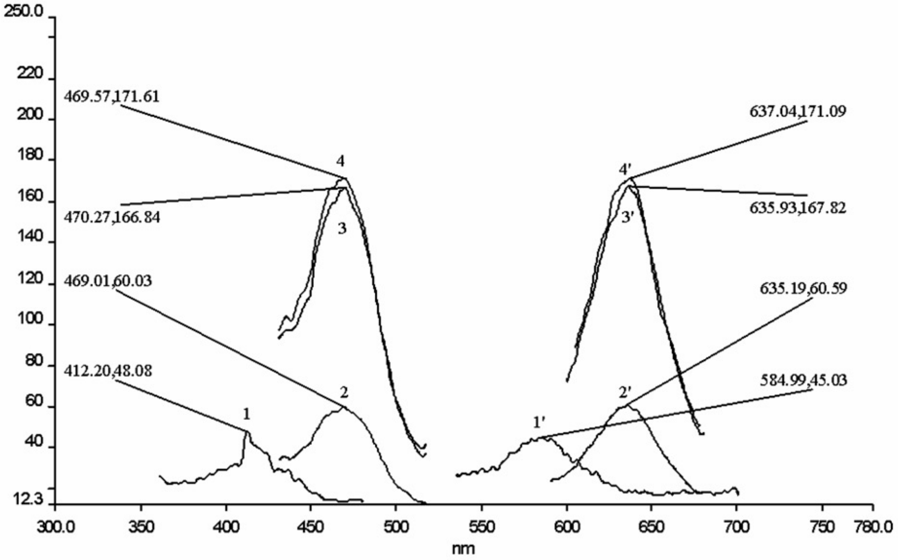

Figure 2. The phosphorescence spectra of F-ol-tween-80-H_2O_2-As(V) system. Notes: 1.1′ : NCM; 4.4′ : 2.00 mL F-ol + 3.00 mL tween-80 + 1.20 mL H_2O_2; 3.3′ : 4.4′ + 1.00 fg As(V); 2.2′ : 4.4′ + 700.0 fg As(V).

Table 1. Optimisation of the volume and the concentration of reagents ($n = 6$).

Reagents	Concentrations and volumes	ΔI_p	Best conditions
F-ol ($\times 10^{-4}$ mol L^{-1})	1.0, 0.10, 0.010	19.3, 28.3, 13.7	1.0×10^{-5} mol L^{-1} F-ol
RSD (%)		1.3, 2.6, 4.8	
(mL)	0.50, 1.00, 1.50, 2.00, 2.50	5.4, 7.8, 14.5, 29.5, 11.2	2.00 mL F-ol
RSD (%)		4.9, 3.7, 3.6, 2.0, 5.0	
H_2O_2 (%,V/V)	0.1, 0.3, 0.6, 0.9, 1.2	10.3, 29.1, 17.4, 9.8, 4.4	0.3 0% H_2O_2
RSD (%)		3.7, 2.7, 1.5, 3.9, 4.7	
(mL)	0.10, 0.60, 1.20, 1.80, 2.40	2.4, 9.5, 30.2, 9.6,1.8	1.20 mL H_2O_2
RSD (%)		4.8, 2.6, 2.2, 1.6, 4.6	
Tween-80 (%)	3.0, 3.5, 4.0, 5.0, 6.0	3.2, 5.4, 7.8, 29.6,10.5	5% tween-80
RSD (%)		3.6, 4.4, 4.8, 1.8, 2.0	
(mL)	1.00, 2.00, 2.50, 3.00, 3.50	4.8, 9.1, 11.5, 29.8, 12.3	3.00 mL tween-80
RSD (%)		3.1, 4.8, 3.5, 4.4, 4.1	

3.2 *Optimum measurement condition*

3.2.1 *Concentration and volume of reagents*

For the system containing 3.2 ag As(V) spot^{-1}, the volumes or concentrations of reagents were changed, respectively. The results showed that when the volumes and concentrations were 2.00 mL of 1.0×10^{-5} mol L^{-1} F-ol, 3.00 mL of 5% tween-80 and 1.2 mL of 0.30%H_2O_2, the ΔI_p of the system reached the maximum and remained stable (Table 1). At this time, the pH value of reaction system was 7.14.

3.2.2 *Sensitizer*

For the system containing 3.2 ag As(V) spot^{-1}, the effects of different sensitizers on ΔI_p of the system, such as 3% of sodium lauryl sulfonate (A), tween-80 (B), cetyltrimethyammonium bromide (C), triton X-100 (D), sodium dodecyl benzene sulfonate (E), polyvinyl alcohol (F) and cetylpyridinium bromide (G) were examined, respectively. The results showed that the ΔI_p of the system reached the maximum (Figure 3) when tween-80 was used as sensitizer.

3.2.3 *Ion perturber*

For the system containing 3.2 ag As(V) spot^{-1}, the effects of different ions such as 1.00 mol L^{-1} $LiNO_3$, NaI, $Cu(NO_3)_2$, $Pb(Ac)_2$ and $AgNO_3$ ΔI_p of the system were examined, respectively. The results showed that the ΔI_p reached the maximum (Figure 4) when Li^+ was used as the ion perturber. Meanwhile, the effects of Li^+ with different concentrations on ΔI_p were examined. Results showed that the ΔI_p of the system reached the maximum when 1.50 mol L^{-1} Li^+ used (Figure 5). For F-ol, the heavy atom effects of Li^+, I^-, Ag^+ and Cu^{2+} were obviously higher than those of perturbers commonly used such as $Pb(Ac)_2$. Moreover, the heavy atom effect of Li^+ was the highest and the phosphorescence intensity reached the maximum. This showed light element ions had the heavy atom effect [19] would not cause pollution.

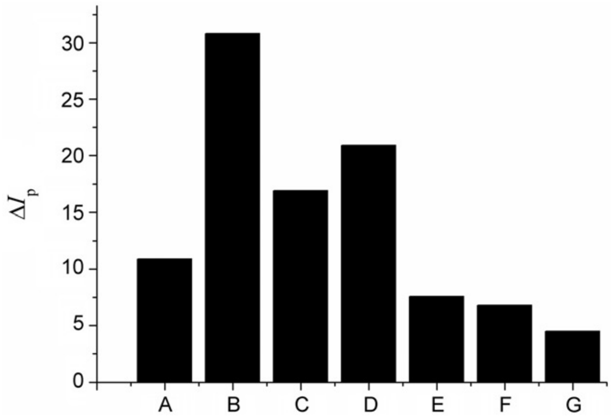

Figure 3. Effect of sensitizer on ΔI_p of reaction system.
Notes: A: 3% sodium lauryl sulfonate; B: 3% tween-80; C: 3% cetyltrimethyammonium bromide; D: 3% Triton X-100; E: 3% sodium dodecyl benzene sulfonate; F: 3% polyvinyl alcohol; G: 3% cetylpyridinium bromide.

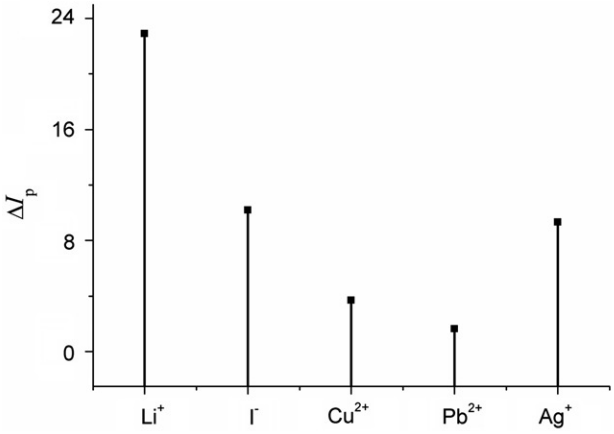

Figure 4. Effect of ion perturbation on ΔI_p of reaction system.

3.2.4 *Solid substrate*

For the system containing 3.2 ag As(V) spot^{-1}, the effects of different substrates, such as ACM, PAM, NCM and paper on ΔI_p were examined, respectively. The results showed that the ΔI_p of the system reached the maximum (Figure 6) when NCM was used as the solid substrate.

3.2.5 *Reaction acidity*

For the system containing 3.2 ag As(V) spot^{-1}, the effects of the values of pH on ΔI_p of the system were examined. The result showed that the ΔI_p of the system remained stable and reached the maximum (Figure 7) when the values of pH were within 6.81 to 9.68.

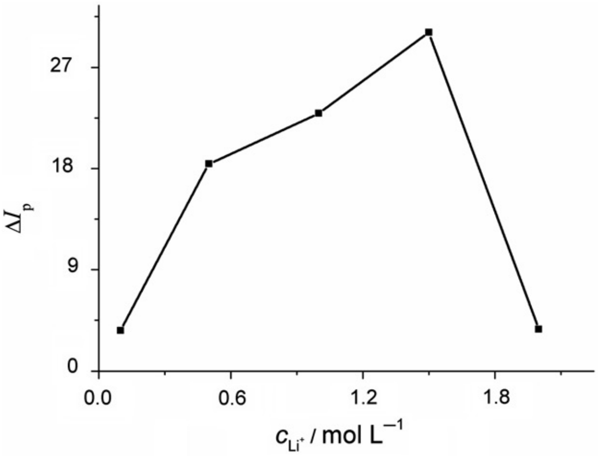

Figure 5. Effect of the different concentration of Li^+ on ΔI_p of reaction system.

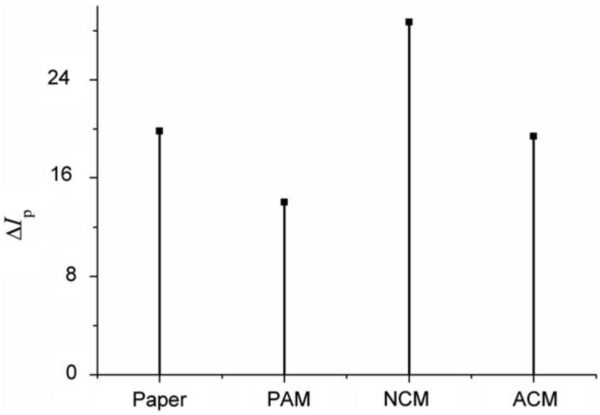

Figure 6. Effect of solid substrate on ΔI_p of reaction system.

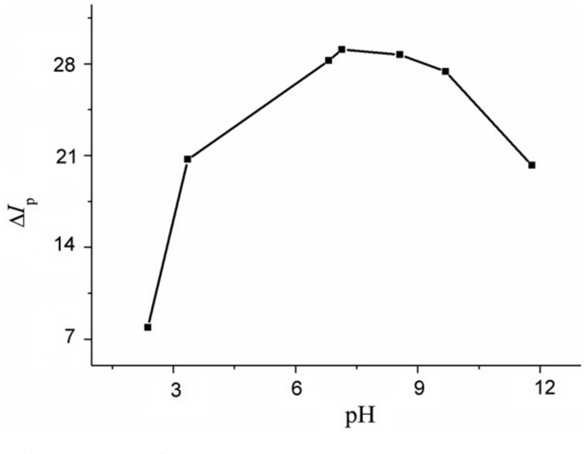

Figure 7. Effect of acidity on ΔI_p of reaction system.

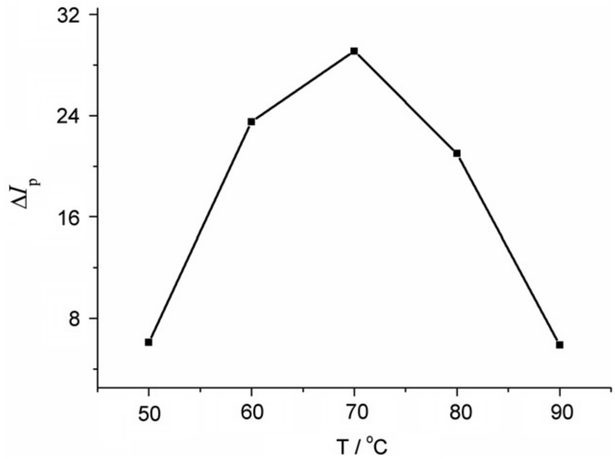

Figure 8. Effect of temperature on ΔI_p of reaction system.

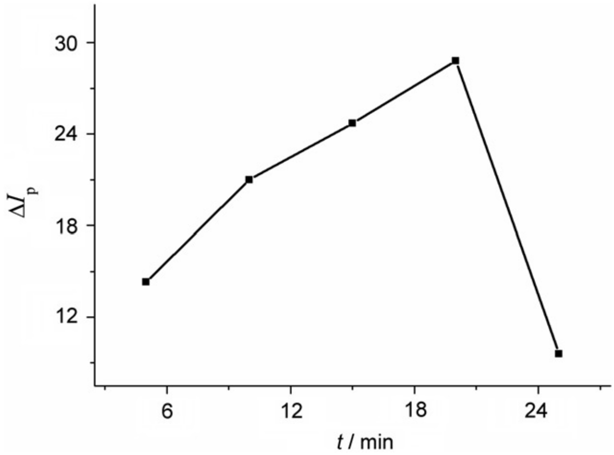

Figure 9. Effect of reaction time on ΔI_p of reaction system.

3.2.6 *Temperature and time for reaction*

For the system containing 3.2 ag As(V) spot^{-1}, the effects of reaction temperature and time on ΔI_p of the system were examined, respectively. Results showed that the ΔI_p of the system reached maximum and remained stable (Figures 8 and 9) when the reaction temperature and time were 70°C and 20 min, respectively.

3.2.7. *Oxygen and humidity*

Due to the remarkable quenching effects of oxygen and water on RTP, the effects of oxygen and humidity on ΔI_p of the system must be examined in SS-RTP. For the sample containing 3.2 ag As(V) spot^{-1}, when desiccated O_2 was passed for 5, 10, 15, 20, 25 and 30 min, respectively, the ΔI_p of the system were 28.6, 28.5, 28.6, 28.6, 28.4 and 27.5, respectively; when humid O_2 was passed for 5, 10, 15, 20, 25 and 30 min, respectively, the

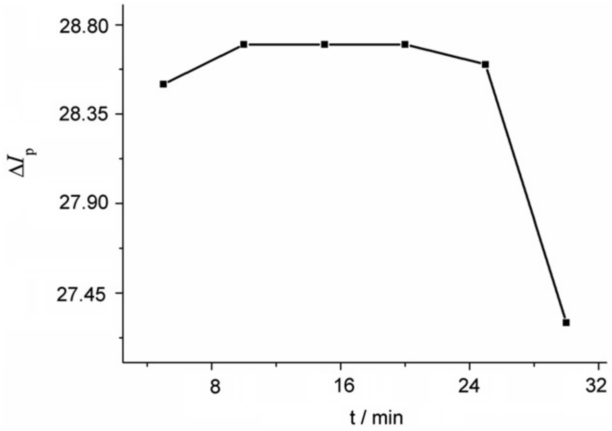

Figure 10. Effect of stability time on ΔI_p of reaction system.

ΔI_p of the system were 28.5, 28.4, 28.5, 28.5, 28.3 and 27.4, respectively. Results showed that the ΔI_p of system was stable whether desiccated or humid O_2 was passed. While desiccated N_2 was passed for 5, 10, 15, 20, 25 and 30 min, respectively; the ΔI_p of the system were 28.7, 28.6, 28.6, 28.7, 28.5 and 27.6, respectively. When there was no desiccated N_2 passed, the ΔI_p of the system were 28.8, 28.7, 28.4, 28.6, 28.7 and 27.7, respectively. Results showed that the system was stable whether desiccated N_2 was passed or not. Therefore, we would not pass desiccated N_2 to simplify the experiment.

3.2.8 *Stability of system*

For the system containing 3.2 ag As(V) spot^{-1}, ΔI_p for the reagent blank and test solution on the NCM remained almost unchanged in the following 5–25 min after cooling by flowing water for 5 min, indicating that the system had good stability (Figure 10).

3.3 *Working curve, linear range and detection limit*

The linear range, the regression equation of working curve, the correlation coefficient (*r*), RSD% (6 fold replicate measurements for 0.016 and 11.2 ag spot^{-1} As(V)) and the detection limit (11 fold replicate measurements, calculated by 3Sb/k) of this method were compared with reference 4. Results were listed in Table 2.

Results showed that the sensitivity of this method was higher and the linear range was wider than reference 4.

3.4 *Interference experiment*

For the sample containing 3.2 ag As(V) spot^{-1}, the allowed concentration (multiple) of coexistent ions or coexistent materials (Er $\leq\pm$ 5%) are listed in Table 3, which indicated high selectivity of this method.

Table 2. Analysis parameter ($n = 6$).

Method	Linear range	Regression equation	Correlation coefficient	RSD (%)	Detection limit
Present method	0.016–11.2 (ag spot^{-1})	$\Delta I_p = 0.7648 + 9.679$ $m_{As(v)}$ (ag spot^{-1})	0.9990	4.8–3.6	9.3 (zg spot^{-1})
	0.040–28 (fg mL^{-1})				0.023 (fg mL^{-1})
Ref. [4]	0.0–16 (ng mL^{-1})	$\lg(A_t/A_0) = K_2C_{As(v)}$	0.9996	1.1	0.4 (ng mL^{-1})

Table 3. Effects of coexistent ions.

Present method			Ref. [4]	
Coexistent ions	Multiple	Relative error (%)	Coexistent ions	Multiple
$Co(NO_3)_2$	400	−3.0	Co^{2+}	200
$SnCl_4$	100	−2.8	Sn^{4+}	–
$Ni(NO_3)_2$	400	−1.1	Ni^{2+}	200
$NaNO_3$	800	+2.9	Na^+	500
KNO_3	1000	−1.0	K^+	500
$Ca(NO_3)_2$	5.0×10^5	−2.4	Ca^{2+}	500
NaAc	5.0×10^5	−3.0		
Na_3PO_4	5.0×10^5	−4.4		
$Mg(NO_3)_2$	1000	−3.7	Mg^{2+}	500
$Al(NO_3)_3$	1.0×10^5	−3.4	Al^{3+}	200
Na_2MoO_4	1.0×10^5	+2.9		
$Sr(NO_3)_2$	1.0×10^5	+4.2		
$Ba(NO_3)_2$	250	−1.7	Ba^{2+}	–
$Zn(NO_3)_2$	500	−0.10	Zn^{2+}	200
Na_2HPO_4	5.0×10^5	+2.6	HPO_4^{2-}	–
NaBr	100	−2.0	Br^-	–
Na_2SO_4	5.0×10^5	−0.10	SO_4^{2-}	–
$NaNO_3$	5.0×10^5	−2.3	NO_3^-	500
$NaNO_2$	1000	+4.3		
Na_2SO_3	1000	−1.5		
$Na_2S_2O_3$	1000	+4.1		
$Na_2C_2O_4$	1000	−4.5		
$Mn(NO_3)_2$	1.0×10^4	−2.7		
NH_4NO_3	1.0×10^4	+3.3		
$CrCl_3$	1.0×10^4	−3.9		
K_2CrO_4	1000	−1.8		
NaSCN	5.0×10^3	−4.2		
$Fe(NO_3)_3$	800	+1.8		
$Bi(NO_3)_3$	400	+3.5		
$Fe(NO_3)_2$	500	−3.7		
$Sb(NO_3)_3$	250	−4.6		

Table 4. The analytical results of arsenic in hair and tea samples.

Sample	Average found ($\mu g\,g^{-1}$, $n=7$)	RSD (%, $n=7$)	AAS ($\mu g\,g^{-1}$, $n=7$)
Hair	3.4 (3.3, 3.3, 3.4, 3.4, 3.4, 3.5, 3.5)	2.4	0.33
Tea	0.25 (0.24, 0.25, 0.25, 0.25, 0.25, 0.25, 0.27)	3.6	0.26

3.5 *Sample analysis*

0.4 g (±0.1 mg) of tea stoved in dry oven at 105°C for 2 h was weighed accurately, stirred to pieces and digested by 20.0 mL of a mixture solution of $HClO_4$ and HNO_3(1 : 3, V/V) to colourless. The solution was heated to nearly dryness, diluted to 1000 mL with water, then 1.00 mL of the diluent was took and diluted to 10^5 multiple with water before used. Human hair sample was washed three times with detergent, and washed with water until no foam was left, then dried and immersed in acetone for 15 min, at last dried naturally. Stirred to pieces, 0.4 g (±0.1 mg) of sample was weighed accurately, and treated in the same way as the tea sample. 1.00 mL of sample solution was taken and the content of As(V) in the hair or tea solution was determined according to the experimental method described above. Table 4 compares the results of this method with the AAS method.

3.6 *Mechanism of reaction*

F-ol could emit stable RTP on NCM, but the I_p was weak (Figure 1, curve 4.4′). Tween-80 could make the RTP signal of F-ol enhanced with a blue shift of λ_{em}^{max} for 9.3 nm (Figure 1, curve 5.5′). The possible reason was that tween-80 interacted with F-ol to form glue compound and made the F-ol molecules on the glue orderly. With the effect of H_2O_2, RTP signal was quenched sharply (Figure 2, curve 4.4′). The reaction could be expressed as follows [20]:

$$\text{F-ol} + H_2O_2 + H^+ \rightarrow \text{F-ol}' + H_2O$$

In presence of As(V), As(V) could oxidise F-ol to form none phosphorescence compound and As(V) was deoxidised to As(III):

$$\text{As(V)} + \text{F-ol} \rightarrow \text{As(III)} + \text{F-ol}'$$

As(III) could react with H_2O_2, then As(III) was oxidised to As(V):

$$\text{As(III)} + H_2O_2 + H^+ \rightarrow \text{As(V)} + H_2O$$

Therefore, As(V) could catalyse H_2O_2 to oxidise F-ol, then caused RTP signal of F-ol to quench acutely ($\Delta I_p = 171.1–60.6 = 110.5$). ΔI_p was 7.7 times larger than the reaction without tween-80 ($\Delta I_p = 47.2–32.8 = 14.4$). It showed that tween-80 could spike the reaction that As(V) catalysing H_2O_2 to oxidise F-ol and the ΔI_p was directly proportional to the content of As(V). Thus, trace arsenic could be determined by the catalytic SS-RTP.

4. Conclusion

A new sensitive and quick catalytic SS-RTP method for the determination of trace As(V) has been established based on oxidising reaction between hydrogen peroxide and fullerol using tween-80 as a sensitizer. Fullerol has been exploited in the field of SS-RTP and for the development of detection technology for trace ions.

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