A new resonance Rayleigh scattering method for trace Pb coupling the hydride generation reaction with nanogold reaction

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Experimental

Apparatus and Reagents

A model of F-7000 Hitachi fluorescence spectrometer (Hitachi company, Japan), a model of F95S fluorescence spectrometer (Shanghai Jinmin Instrumental Co. Ltd., China), a model of JSM-6380LV scanning electron microscope (Japan), a model of SK8200LH ultrasonic reactor with operating frequency of 59 KHz (Shanghai Family Guide Ultrasonic Instrument Co. Ltd., China), and a model of TU- 1901 double beam UV -Vis spectrophotometer (Beijing Purkinje General Instrument Co. Ltd., China) were used.

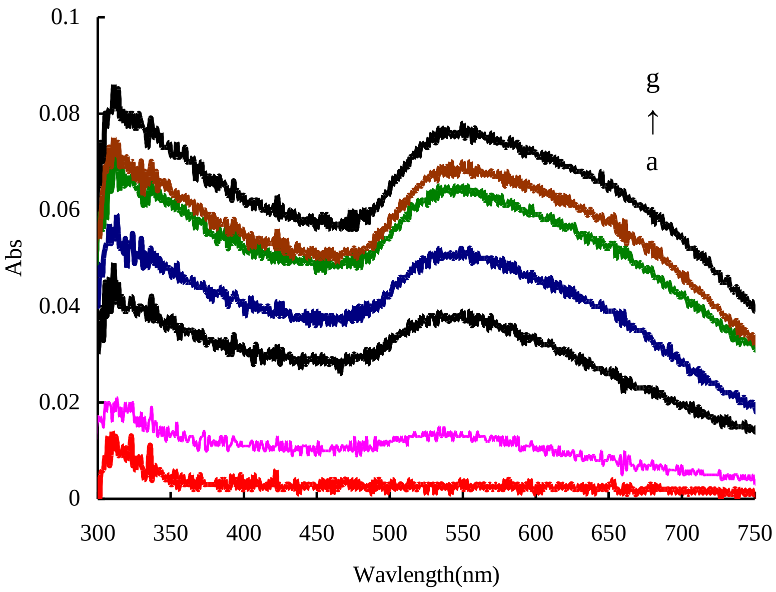
Taken 1.00g $HAuCl_4\cdot 3H_2O$ (National Pharmaceutical Group Chemical Reagents Company, China) to dissolve in 100 mL water to obtain a 3.33% Au^{3+} solution, and it was diluted with water to obtain a 1.0 % Au^{3+} solution for use. A 1.00×10^{-2} mol/L $Pb(NO_3)_2$ standard stock solution, 0.79 mol/L $NaBH_4$ solution, 0.3 mol/L $K_3[Fe(CN)_6]$ solution, 3.0×10^{-2} mol/L $K_2Cr_2O_7$, 1.47 mol/L H_2O_2, 0.2 mg/L arabic gum, 0.12 mol/L NH_4SCN, 7.5 mol/L H_3PO_4 were prepared. The absorption solution was mixed by 10 mL 7.5 mol/L H_3PO_4, 1.2 mL 1% $HAuCl_4$, 2 mL 0.12 mol/L NH_4SCN and 1 mL 0.2mg/mL arabic gum solution, and diluted to 100 mL with water. All reagents were of analytical grade and the water was doubly distilled.

Procedure

A certain amount of Pb^{2+} standard solution was added into the reaction bottle and diluted with water to 10 mL, then 1.0 mL 0.3 mol/L $K_3[Fe(CN)_6]$ solution, 1.0 mL 0.03 mol/L $K_2Cr_2O_7$ solution, 0.75 mL 1.47 mol/L H_2O_2 solution and 1.0 ml 4 mol/L HCl solution were added into the reaction bottle in turn, and mixed well. A 7 mL of the absorption solution was added into the absorb tube, and connected the experimental apparatus as in Figure 1. Under the ultrasonic condition, a 10 mL 0.79 mol/L $NaBH_4$ solution was added quickly by pear-shaped separatory funnel to produce PbH_4 gas that absorb with the absorption solution. Finally, the absorption solution was transferred to a 1 cm quartz cell, the RRS intensity(I) at 286 nm was recorded, meanwhile, a reagent blank solution (I_0) without Pb^{2+} was recorded, and a value of $\Delta I=I-I_0$ was calculated.

Absorption spectra

The absorption spectrum of nanoparticle sol also called as surface Plasmon resonance (SPR) that can be used to characterize the existence of nanoparticles. Under the selected conditions, in the absence of Pb^{2+}, there is no SPR absorption in visible region because there is no nanogold formation. In the presence of Pb^{2+}, the SPR absorption peak at 555 nm enhanced and showed that there are gold nanoparticles in the system. A SPR method can be also developed for determination of 2.1×10^{-6}-3.36×10^{-5} mol/L Pb, but the sensitivity is lower than the RRS method.



Absorption spectra

(a) 1.3×10^{-2} mol/L $K_3[Fe(CN)_6]$-1.3×10^{-3} mol/L $K_2Cr_2O_7$-4.6×10^{-2} mol/L H_2O_2-0.16 mol/L HCl-0.33 mol/L $NaBH_4$-0.75 mol/L H_3PO_4-3.84×10^{-4} mol/L $HAuCl_4$-2.4×10^{-3} mol/L NH_4SCN -2μg /mL rabic gum; (b) a-2.1×10^{-6} mol/L Pb^{2+}; (c) a-4.2×10^{-6} mol/L; (d) a-8.4×10^{-6} mol/L Pb^{2+}; (e) a-2.1×10^{-5} mol/L Pb^{2+}; (f) a-2.94×10^{-5} mol/L Pb^{2+}; (g) a-3.36×10^{-5} mol/L Pb^{2+}

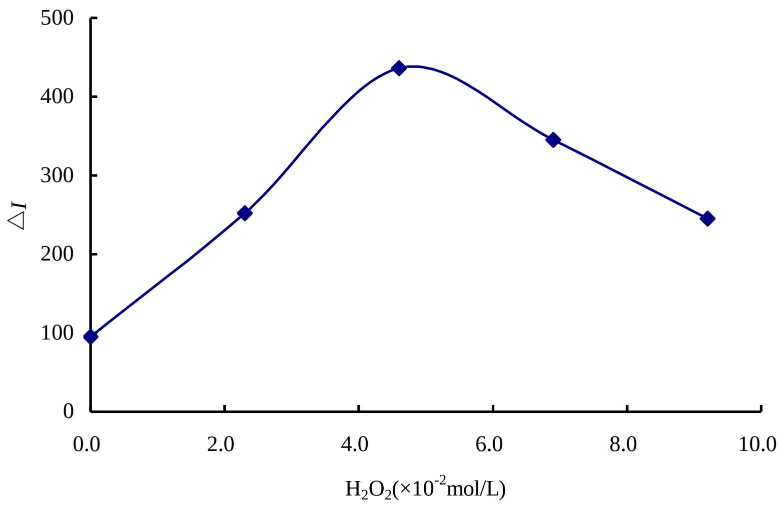


Fig. 1S Effect of the concentration of H_2O_2

1.68×10^{-5} mol/L Pb^{2+}-0.33 mol/L $NaBH_4$-1.3×10^{-2} mol/L $K_3[Fe(CN)_6]$-1.3×10^{-3} mol/L $K_2Cr_2O_7$ -0.16 mol/L HCl-0.75 mol/L H_3PO_4-3.84×10^{-4} mol/L $HauCl_4$-2.4×10^{-3} mol/L NH_4SCN-2μg /mL arabic gum.

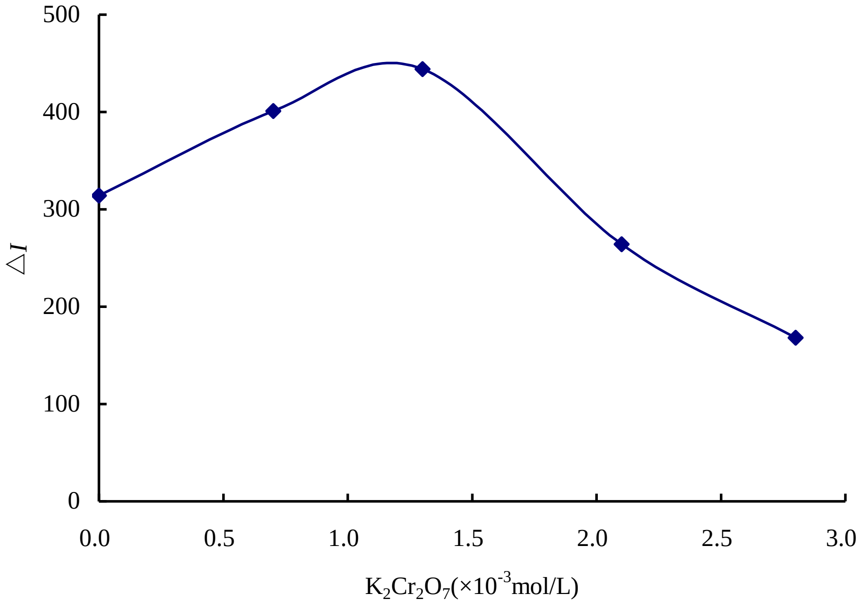


Fig. 2S Effect of the concentration of $K_2Cr_2O_7$

1.68×10^{-5} mol/L Pb^{2+}-0.33 mol/L $NaBH_4$-1.3×10^{-2} mol/L $K_3[Fe(CN)_6]$ -4.6×10^{-2} mol/L H_2O_2-0.16 mol/L HCl-0.75 mol/L H_3PO_4-3.84×10^{-4} mol/L $HauCl_4$-2.4×10^{-3} mol/L NH_4SCN -2μg /mL arabic gum.

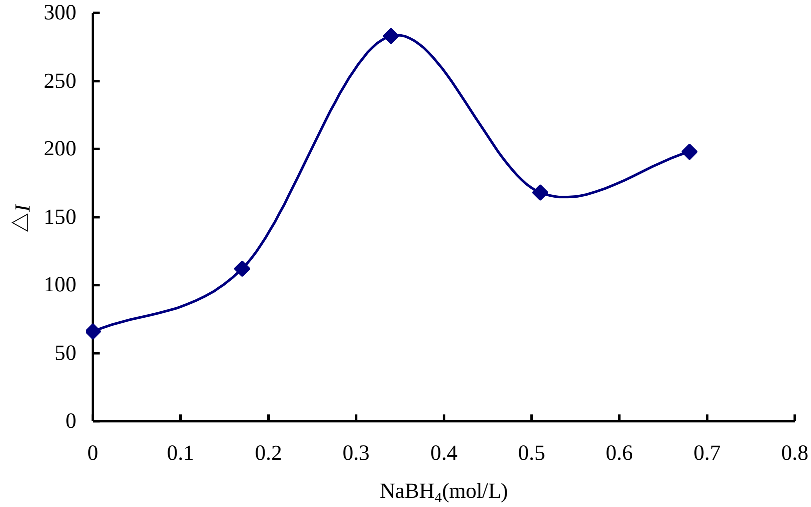


Fig. 3S Effect of the concentration of $NaBH_4$

1.68×10^{-5} mol/L Pb^{2+}-1.3×10^{-2} mol/L $K_3[Fe(CN)_6]$-1.3×10^{-3} mol/L $K_2Cr_2O_7$-4.6×10^{-2} mol/L H_2O_2-0.16 mol/L HCl-0.75 mol/L H_3PO_4-3.84×10^{-4} mol/L $HAuCl_4$-2.4×10^{-3} mol/L NH_4SCN-2μg /mL arabic gum.

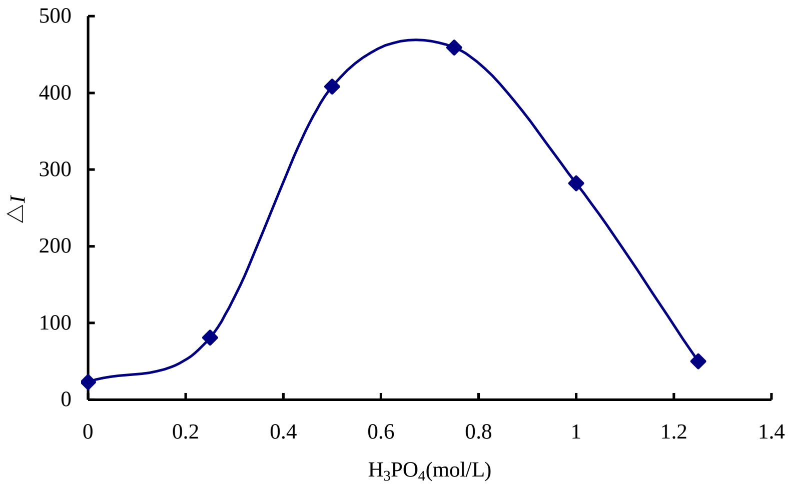


Fig. 4S Effect of the concentration of H_3PO_4

1.68×10^{-5} mol/L Pb^{2+}-0.33 mol/L $NaBH_4$-1.3×10^{-2} mol/L $K_3[Fe(CN)_6]$-1.3×10^{-3} mol/L $K_2Cr_2O_7$-4.6×10^{-2} mol/L H_2O_2-0.16 mol/L HCl-3.84×10^{-4} mol/L $HAuCl_4$-2.4×10^{-3} mol/L NH_4SCN-2μg /mL arabic gum.

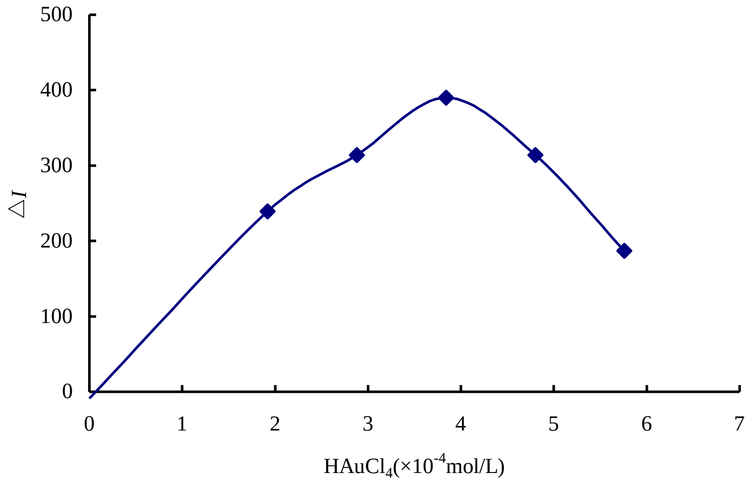


Fig. 5S Effect of the concentration of $HauCl_4$

1.68×10^{-5} mol/L Pb^{2+}-0.33 mol/L $NaBH_4$-1.3×10^{-2} mol/L $K_3[Fe(CN)_6]$-1.3×10^{-3} mol/L $K_2Cr_2O_7$-4.6×10^{-2} mol/L H_2O_2-0.16 mol/L HCl-0.75 mol/L H_3PO_4 -2.4×10^{-3} mol/L NH_4SCN-2 μg/mL arabic gum.