Electronic Supplementary Information for Iodine-mediated Etching of Gold Nanorods for Plasmonic Sensing of Dissolved Oxygen and Salt Iodine

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1. Experimental Section

Preparation of Gold Nanorods. The AuNRs were synthesized using a modified method by changing the amount of AgNO₃.¹ Seed preparation: To 7.5 mL of CTAB (0.10 M) solution, 0.25 mL of HAuCl₄·3H₂O (0.01 M) and 0.60 mL of ice-cold NaBH₄ (0.01 M) were added in sequence. The mixed solution was kept in a 26 °C water bath for 2 h. (2) *AuNRs Growth*: 1.2 mL of 0.05 M HAuCl₄·3H₂O, 0.3 mL AgNO₃ (0.01 M) and 0.96 mL ascorbic acid (0.1 M) was added to 100 mL of CTAB (0.10 M) in sequence with stirring,. Finally, 0.2 mL seed solution prepared in step (1) was added at room temperature. The color of the solution gradually changed to purple within 20 min. The solution was further left for 2 days without stirring.

Procedure for the Mass Spectra of Etching products. 1.5 mL AuNRs solution were centrifuged twice at 7500 rpm for 15 min to remove excess CTAB. Then the soft soft sediment was re-suspended in 100 μ L deionized water containing 0.1 M CTAB. Then, 900 μ L glycine/HCl buffer solution (50 mM, pH 2.2) was added in to the AuNRs solution. Afterward, 10 μ L KI (0.1 M) and 25 μ L KIO₃ (0.1 M) were added into the above solution. The mixture solution was incubated at 50 °C for 15 min and the final solution was subjected to measure mass spectra.

2. Figures



Figure S1. Absorption spectra in glycine/HCl buffer (pH 2.2) in the presence of 1.0 mM KI (a), mixture of 1.0 mM KI and 1.0 mM IO_3^- (b), mixture of 1.0 mM KI, 1.0 mM IO_3^- and 0.1% starch (c).



Figure S2 Extinction spectra and colors of AuNRs in glycine/HCl buffer solution in the presence of 1.0 mM KI after incubation with 20 (a), 30 (b), 50 μ M KIO₃ (c) at 50 °C for 15 min.



Figure S3 Optimization of CTAB concentration for the detection of IO₃⁻.



Figure S4 Optimization of pH for the detection of IO₃⁻.



Figure S5 Optimization of KI concentration for the detection of IO₃⁻.



Figure S6 Optimization of incubation temperature for the detection of IO₃⁻.



Figure S7 Optimization of incubation time for the detection of IO₃⁻.



Figure S8 Absorption spectra in glycine/HCl buffer (pH 2.2) in the presence of 1.0 mM KI (a), mixture of 1.0 mM KI and mixed ions (b), mixture of 1.0 mM KI, mixed ions and 1.0 uM $IO_3^{-}(c)$, mixture of 1.0 mM KI, mixed ions and 10 uM $IO_3^{-}(c)$. Mixed ions : 10 μ M for Li⁺, K⁺, Ca²⁺, Mg²⁺, Al³⁺, Zn²⁺, Fe³⁺, Co²⁺, Ni³⁺, Cu²⁺, Mn²⁺, Ac⁻, F⁻, CO₃²⁻, NO₃⁻, SO₄²⁻, PO₄³⁻, ClO₄⁻; 1.0 μ M for H₂O₂, NO₂⁻, Cr(VI).



Figure S9 X-ray diffraction spectrum of gold nanorods.

Determination of iodate in table salt. Deficiency of iodine may lead to serious health problems, such as endemic goiter and cretinism.² The adding of iodate into table salt is one of the most effective ways against iodine-deficiency. However, excess intake of iodine may also lead to some diseases, hyperthyroidism.³ Various methods have been developed for detection of iodate, such as fluorescence spectroscopy,^{4, 5} ion chromatography^{6, 7} and electrochemistry^{8, 9}. However, not much attention has been paid to develop the plasmonic assay for iodate.

It is also reasonable for detection salt iodine using the proposed method due to its simple components. Since table salt is a very "clean" sample and these interferences species, such as H_2O_2 , NO_2^- and Cr(VI), will never coexist. So, after dissolving the table salt samples in water, the concentration of iodate in these samples can be determinated by using the linear relationships between peak-shift and the concentration of IO_3^- (Figure 3B). Table S1 shows the detection results using the proposed method. The detection results were consistent with the certified

concentrations with the recovery from 92.7% to 105.4%. It indicates that this method could be used for the rapid determination of iodate in table salt.

Table S1. The recovery of certified iodate in table salt; The standard deviation of each sample was obtained by three measurements.

Sample	Certified	Detected	RSD	Recovery
	(mg/kg)	(mg/kg)	(%)	(%)
1	18.0	17.3	16.0	95.6
2	25.0	26.3	13.4	105.4
3	36.0	33.4	8.3	92.7

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