Electronic Supplementary Material (ESI) for Analyst.

Supporting Information

Highly Resistant and Sensitive Colorimetric Immunochromatographic Assay for Sibutramine Illegally Adulterated into Diet Food based on PDA/AuNPs Labelling

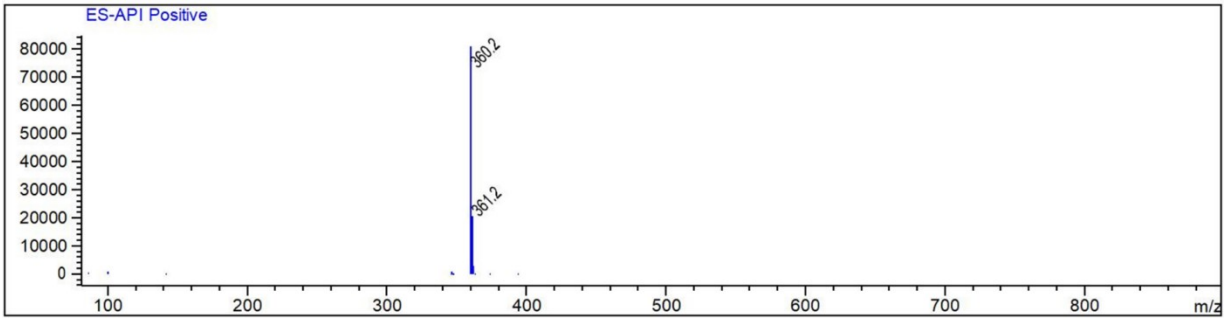


Figure. S1. Positive ion electrospray mass spectra of hapten.

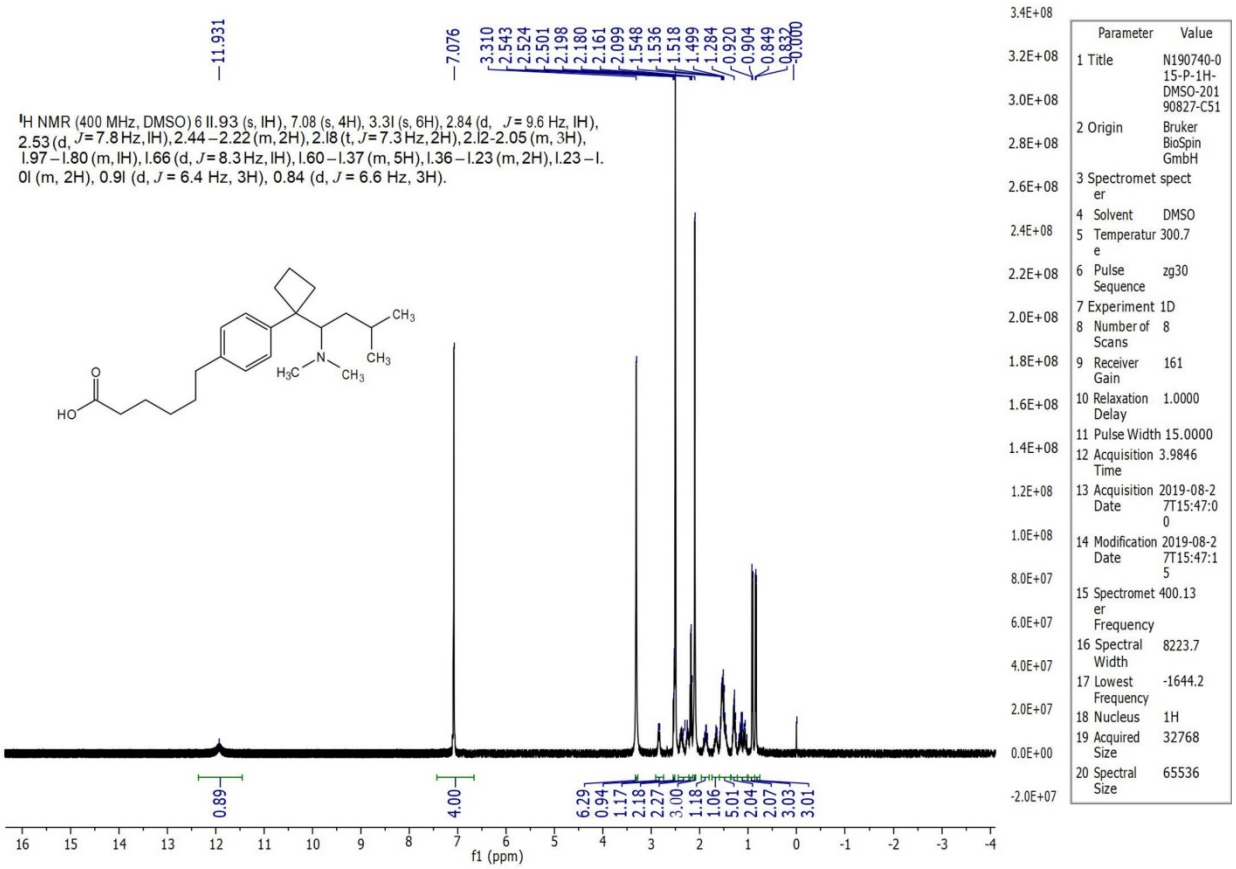


Figure. S2. [1]H-NMR spectra of hapten measured in DMSO at 400 MHz.

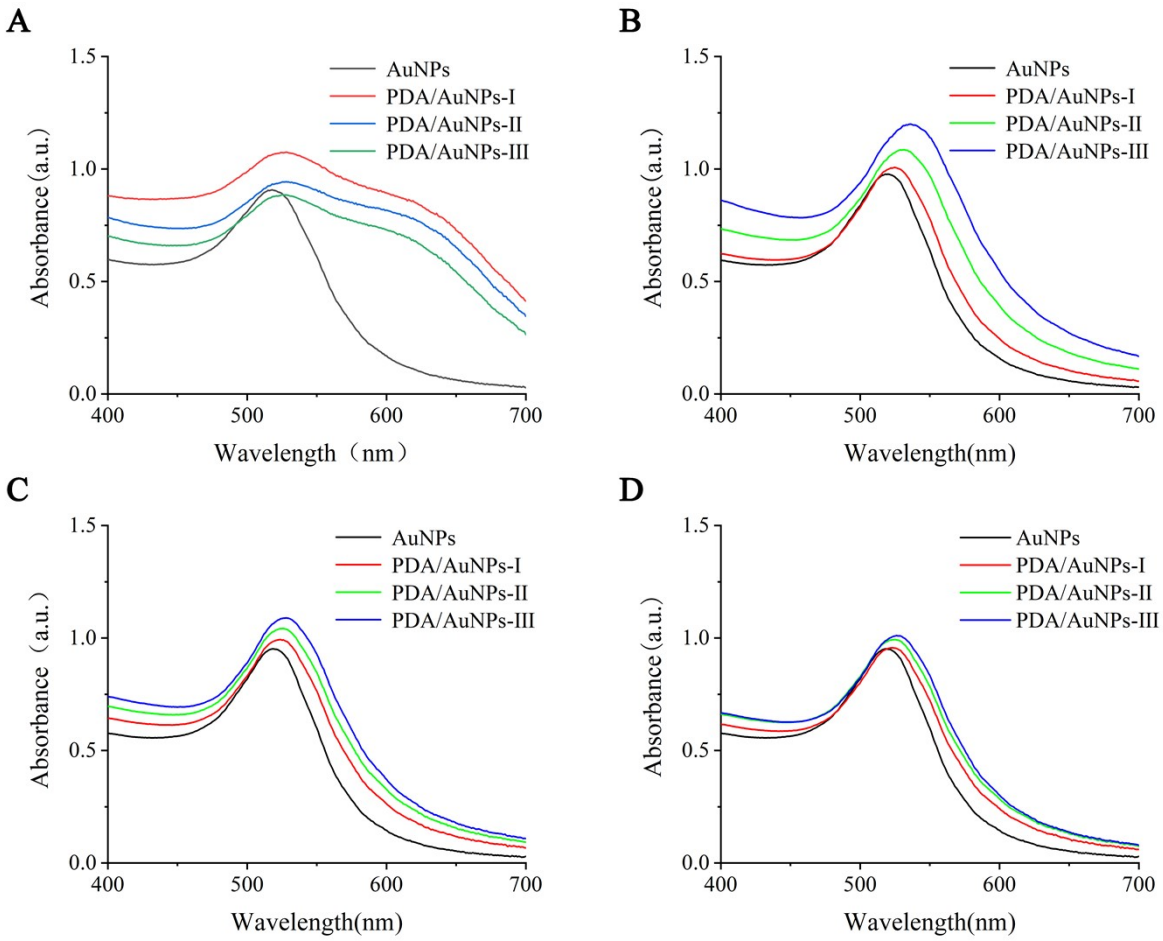


Figure. S3. UV–vis absorption spectra of PDA/AuNPs-I (5 μg DA·HCl addition), PDA/AuNPs-II (10 μg DA·HCl addition), and PDA/AuNPs-III (15 μg DA·HCl addition) in alkaline solution (pH 8.5) with (A) 0, (B) 4, (C) 6, and (D) 8 μL of 3% H_2O_2.

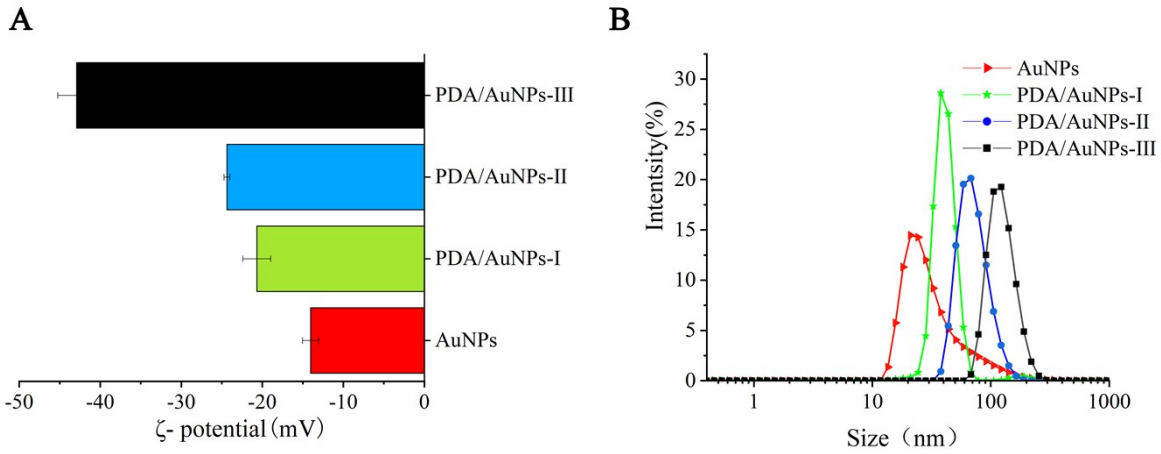


Figure. S4. (A) ζ-Potential and (B) Size distribution of hydrodynamic diameter of AuNPs, PDA/AuNPs-I, PDA/AuNPs-II, and PDA/AuNPs-III.

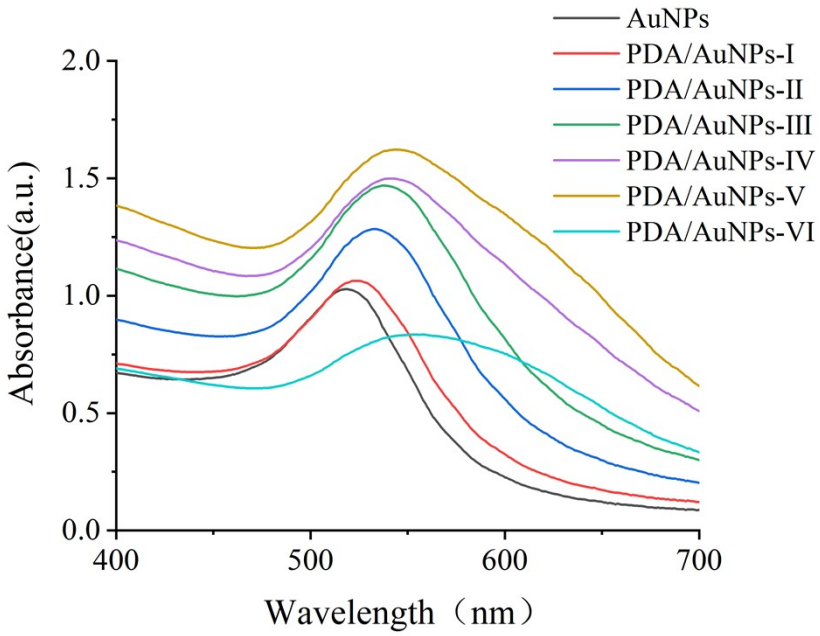


Figure. S5 UV−vis absorption spectra of PDA/AuNPs with excessive addition of DA·HCl.

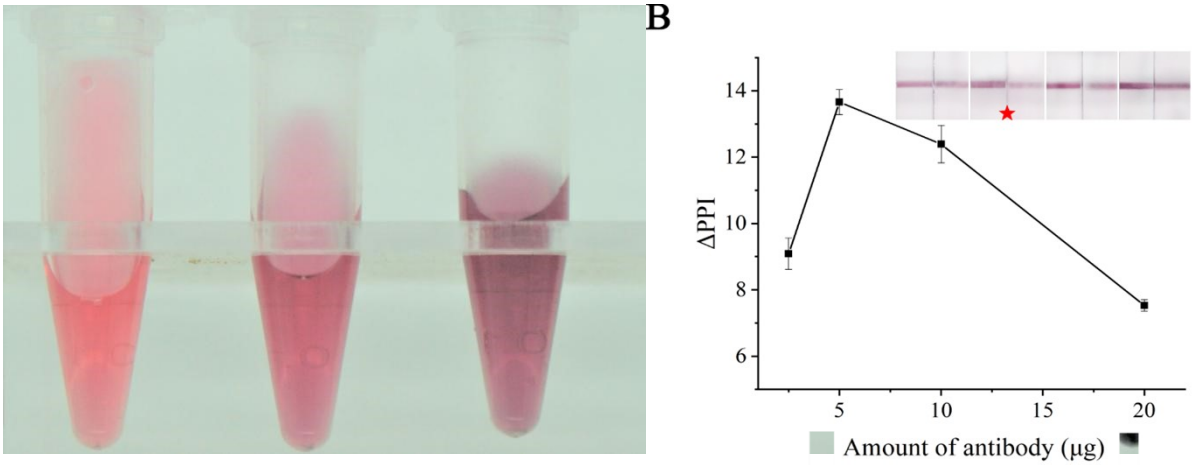


Figure. S6. Aggregation of particles with excessive addition of DA·HCl overnight.

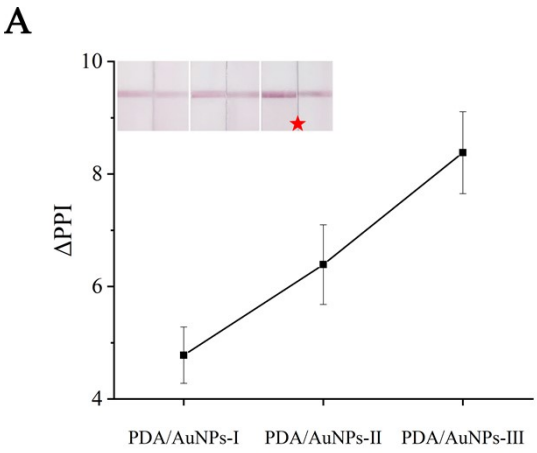


Figure. S7. Optimization results of (A) thickness of PDA/AuNPs and (B) amount of antibody for PDA/AuNPs-III conjugation. Red stars represent the optimal parameters.

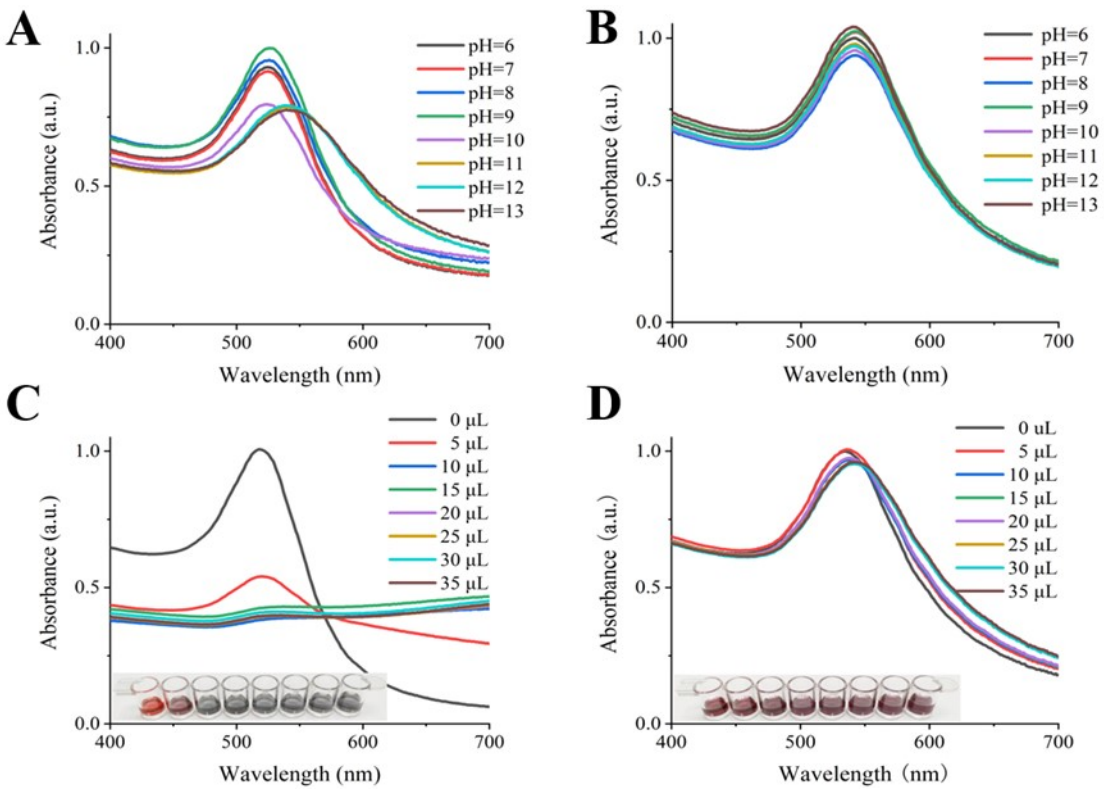
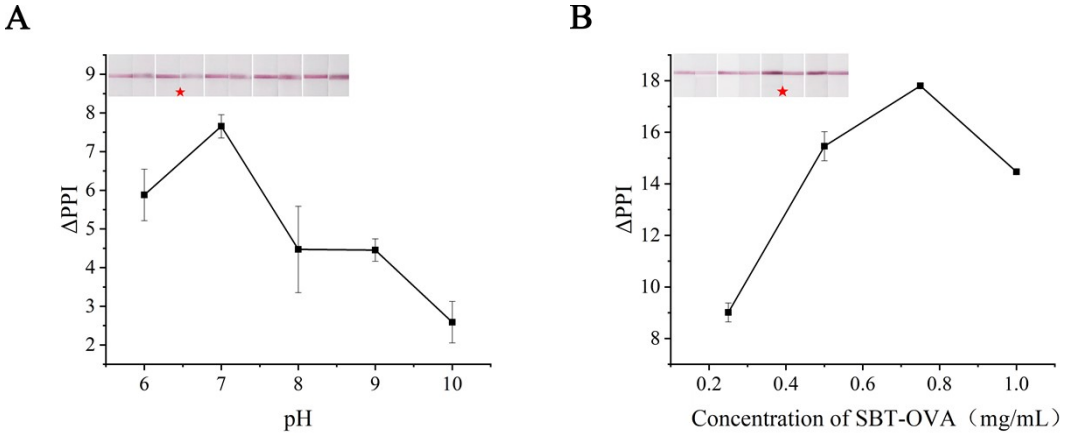


Figure. S8. Uv–vis absorption spectra and images of AuNPs-mAb and PDA/AuNPs-III-mAb at different pH values and salt stress (by adding different volumes of 10% NaCl [μL]): (A) tolerance to pH of AuNPs-mAb and (B) PDA/AuNPs-mAb, and tolerance to (C) NaCl AuNPs-mAb and (D) PDA/AuNPs-mAb.



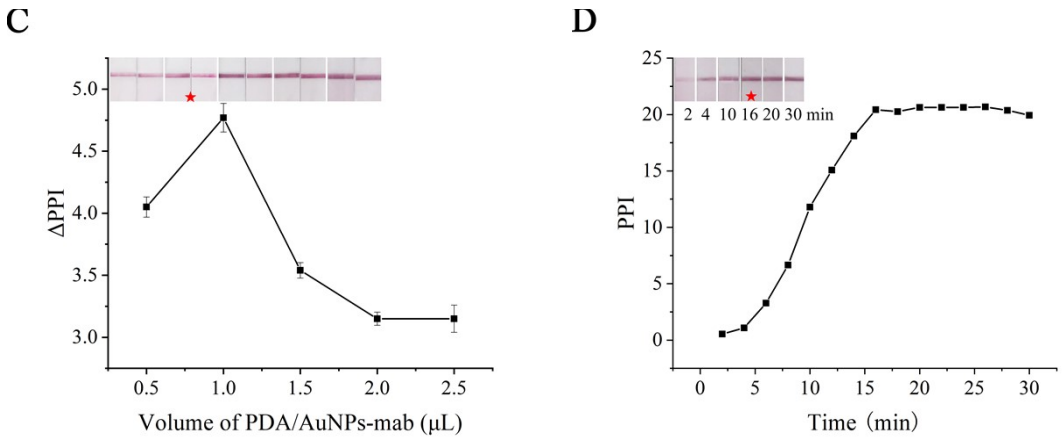


Figure. S9. Optimization of (A) pH, (B) concentration of SBT–OVA, (C) volume of PDA/AuNPs-mAb, and (D) immunochromatographic time. Red stars represent the optimal parameters.

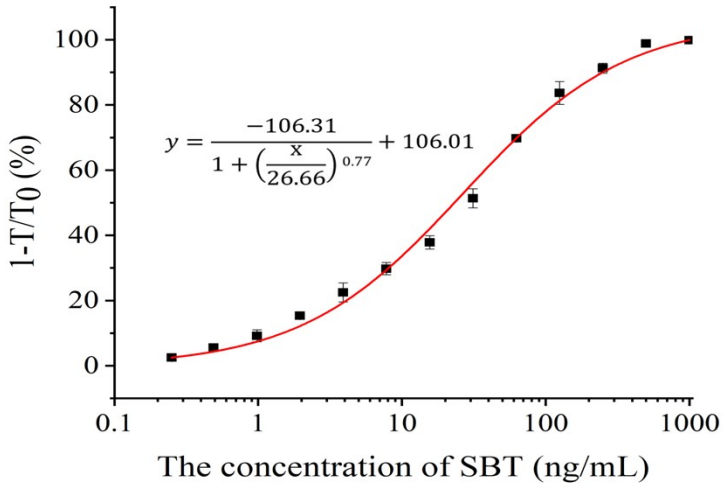


Figure S10. Standard curve of PDA/AuNPs-based LFIA for 0, 0.25, 0.49, 0.98, 1.95, 3.9, 7.81, 15.63, 31.25, 62.5, 125, 250, 500 ng/mL M_1 in buffer. T was the value with M_1 in PBS and T_0 represented the value without any drug.

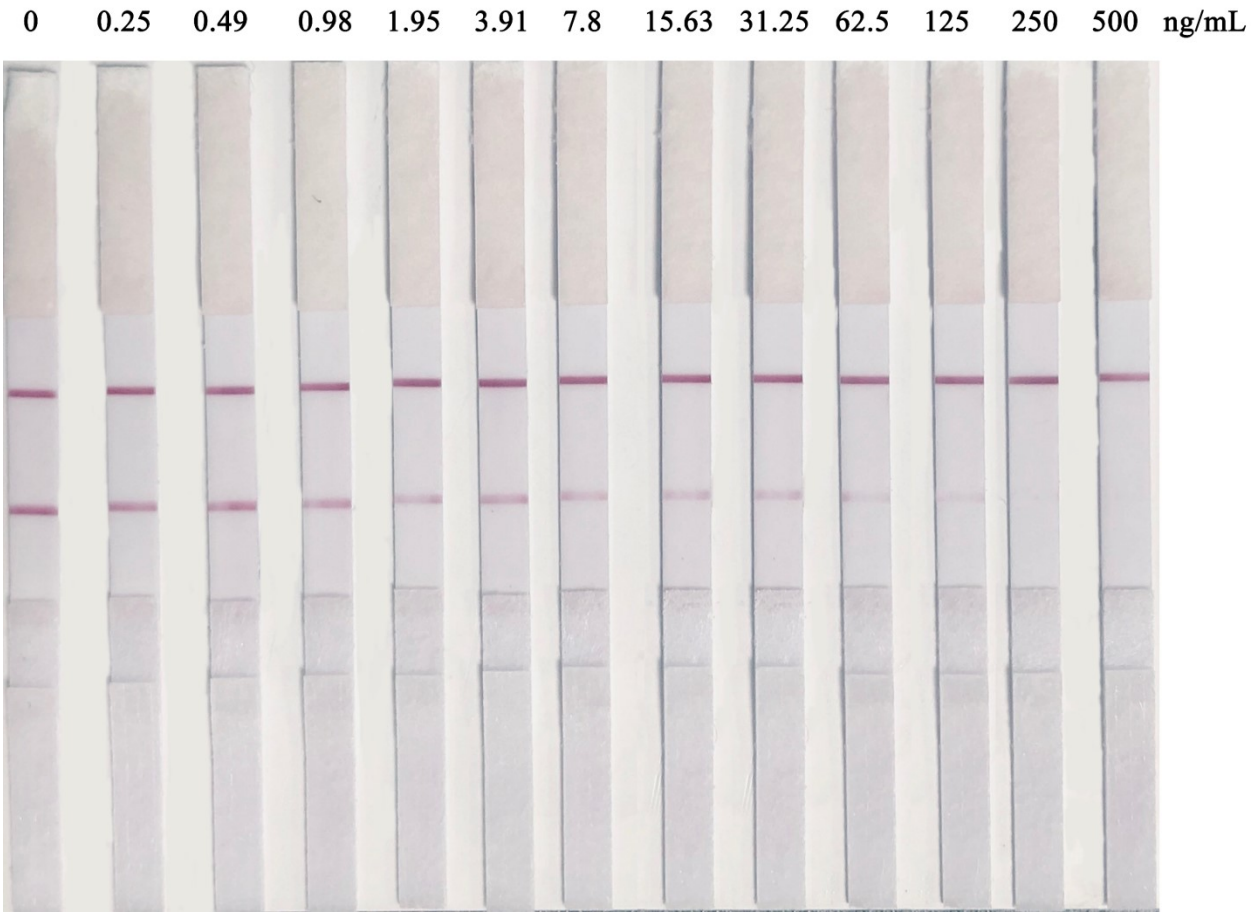


Figure S11. photographs of PDA/AuNPs-based LFIA for 0, 0.25, 0.49, 0.98, 1.95, 3.9, 7.81, 15.63, 31.25, 62.5, 125, 250, 500 ng/mL M_1 in buffer.

Table S1. Cross-reaction Study of mAb Against SBT and its Analogues (ELISA).

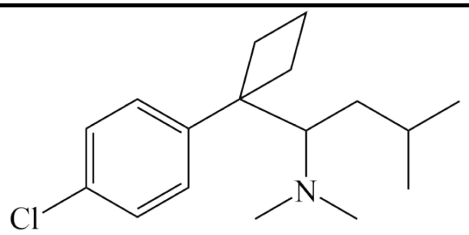
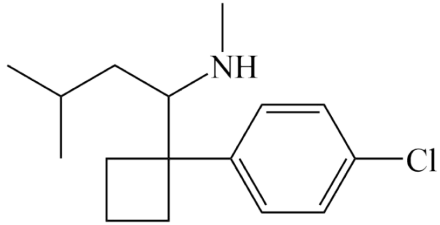
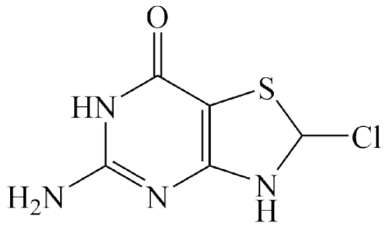
Analogues	Structure	IC_{50} (ng/mL)	CR (%)
SBT		0.69	100
M_1		1.23	56.10
M_2		＞5000	＜0.01

Table S2. Comparison of different methods for the determination of SBT

Method	Limit of quantitation	Linear range	Consumption of time	Reference
Electrochemical immunosensor	5 ng/mL	0.015 μg/mL~10 μg/mL	8 min	[1]

SERS sensor	0.28 ng/mL	Not mentioned	＞ 60 min	[2]
Capillary Electrophoresis	30 ng/mL	0.1 μg/mL~50 μg/mL	＞ 60 min	[3]
High-Performance Liquid Chromatography	5×10^2 ng/mL	5×10^2 ng/mL~2×10^4ng/mL	＞ 60 min	[4]
Liquid Chromatography-Electrospray Ionisation-Mass Spectrometry	4 ng/mL	5 ng/mL~30 ng/mL	＞ 60 min	[5]
Au-ICA	11.58 ng/mL	23.07 ng/mL~194.84 ng/mL	16 min	This work
PDA/AuNPs-ICA	0.98 ng/mL	3.39 ng/mL~69.60 ng/mL	16 min	This work

Table S3. Determination of SBT in samples (*n*=3)

Sample	Spiked (ng mL^{-1})	LFIA detected (ng mL^{-1})	Recovery (%)	RSD (%)	LC-MS/MS detected (ng mL^{-1})	Recovery (%)	RSD (%)
Dietary Fiber in Capsules	5	4.87	97.41	2.74	5.27	105.40	4.65
	10	9.86	98.63	4.92	10.46	104.60	6.12
	20	19.61	98.05	4.38	21.33	106.65	5.78

Table S4. Determination of M1 in samples (*n*=3)

Sample	Spiked (ng mL^{-1})	LFIA detected (ng mL^{-1})	Recovery (%)	RSD (%)	LC-MS/MS detected (ng mL^{-1})	Recovery (%)	RSD (%)
Human Serum	5	5.43	108.60	3.85	4.76	95.20	5.04
	10	10.67	106.70	4.17	9.81	98.10	4.83
	20	19.45	97.25	6.01	19.97	99.85	6.27

References

(1) Saichanapan, J.; Promsuwan, K.; Limbut, W. Adsorption and determination of sibutramine in illegal slimming product using porous graphene ink-modified electrode. *Talanta* **2020**, *212*, 120788.

(2) Ouyang, L.; Jiang, Z.; Wang, N.; Zhu, L.; Tang, H. Rapid Surface Enhanced Raman Scattering (SERS) Detection of Sibutramine Hydrochloride in Pharmaceutical Capsules with a beta-Cyclodextrin- Ag/Polyvivnyl Alcohol Hydrogel Substrate. *Sensors (Basel)* **2017**, *17*.

(3) Wang, D.; Man, R.; Shu, M.; Liu, H.; Gao, Y.; Luan, F. Detection of sibutramine and phenolphthalein in functional foods using capillary electrophoresis. *Analytical Methods* **2016**, *8*, 621-626.

(4) Ariburnu, E.; Uludag, M. F.; Yalcinkaya, H.; Yesilada, E. Comparative determination of sibutramine as an adulterant in natural slimming products by HPLC and HPTLC densitometry. *J Pharm Biomed Anal* **2012**, *64-65*, 77-81.

(5) Yano, H. M.; Arias, F. F.; Bianco, M.; Trujillo, L. M. Determination of the sibutramine content of dietary supplements using LC-ESI-MS/MS. *LATIN AMERICAN JOURNAL OF PHARMACY* **2013**, *2013*, 1164-1169.