

Supplementary Materials

Table S1. Gradient mobile phase conditions of HSS T3 column.

Time (min)	Flow (mL/min)	A: water containing 1 mM ammonium fluoride (%)	B: Methanol (%)
0	0.3	100	0
0.5	0.3	100	0
3.0	0.3	35	65
5.0	0.3	35	65
5.1	0.3	5	95
6.5	0.3	5	95
6.6	0.3	100	0
8.0	0.3	100	0

Table S2. Gradient mobile phase conditions of CSH PFP column.

Time (min)	Flow (mL/min)	A: water containing 0.1% formic acid (%)	B: acetonitrile (%)
0	0.3	100	0
1.0	0.3	100	0
5.5	0.3	5	95
6.5	0.3	5	95
6.6	0.3	100	0
8.0	0.3	100	0

Table S3. Gradient mobile phase conditions of Trinity P1 column.

Time (min)	Flow (mL/min)	A: water containing 5 mM ammonium formic (%)	B: acetonitrile containing 20% water and 5 mM ammonium formic (%)
0	0.5	98	2
1.0	0.5	98	2
6.0	0.5	2	98
8.0	0.5	2	98
8.1	0.5	98	2
10.0	0.5	98	2

Table S4. Gradient mobile phase conditions of ACE C18-Amide column.

Time (min)	Flow (mL/min)	A: water containing 1 mM ammonium fluoride (%)	B: Methanol (%)
0	0.3	100	0
1.0	0.3	100	0
6.0	0.3	5	95
6.5	0.3	5	95
6.6	0.3	100	0
8.0	0.3	100	0

Table S5. Gradient mobile phase conditions of BEH Amide column.

Time (min)	Flow	A: water containing 0.5% formic acid	B: acetonitrile
	(mL/min)	(%)	(%)
0	0.35	5	95
0.5	0.35	5	95
1.5	0.35	40	60
4.0	0.35	50	50
8.0	0.35	50	50
8.1	0.35	5	95
10.0	0.35	5	95

Table S6. Literature summary of mushroom toxins detection using LC-MS/MS.

Country	Matrix	Compound	MDL	Column	Pretreatment
Japan[19]	Mushroom	Muscimol, ibotenic acid,	10 µg/g	TSK-GEL Amide-80 column (150 × 2.0 mm, 3 µm)	Extracted with 50 % methanol aqueous solution and purified using an Oasis MAX SPE cartridge.
China[14]	Mushroom	α-Amanitin, β-amanitin, and phalloidin	0.02–0.05 mg/kg	BEH shield RP18 column (150 × 2.1 mm, 1.7 µm)	Extracted with acidic methanol aqueous solution
Germany[15]	Urine	α-Amanitin, β-amanitin, psilocin, bufotenine, muscarine, muscimol, ibotenic acid, and ricinine	1~2000 ng/mL	Nucleodur HILIC column (100 × 2 mm, 1.8 µm)	Hydrophilic-phase liquid-liquid extraction using dichloromethane and subsequent solid-phase extraction and precipitation, performed in parallel.
Germany[35]	Urine	α-Amanitin, β-amanitin, γ-amanitin, muscarine, muscimol, and ibotenic acid	1~750 ng/mL	Nucleodur HILIC column (100 × 2 mm, 1.8 µm), running in two methods	Washed with dichloromethane and purified using a Strata X-Drug B cartridge Diluted with acetonitrile and centrifuged
China[16]	Mushroom	Ibotenic acid, muscimol, psilocybin, psilocin, buiotenine, α-amanitin, β-amanitin, γ-amanitin, phalloidin, phallacidin, phallisacin	0.1~1.0 mg/kg	Atlantis T3 column (2.1 × 100 mm, 3.0 µm)	Extracted with methanol aqueous solution and purified using a QuCHERS-PP column
Portugal[36]	Plasma	α-Amanitin and β-amanitin	24–64 µg/mL	Spherisorb® ODS-2 C18 column (150 × 2.1 mm, 3 µm)	Extracted with acidic methanol aqueous solution and purified using an Oasis PRiME HLB SPE cartridge
Japan[33]	Mushroom	Illudin S	0.5 µg/g	TSK-GEL ODS-100Z column (2.0 × 150 mm, 3.0 µm)	Extracted with methanol
Japan[37]	Mushroom	Ibotenic acid, propargylglycine, choline, muscimol, muscarine, α-amanitin, β-amanitin, phalloidin, phallacidin	0.0098–4.9 µg/g	Ascentis Express F5 column (100 × 2.1 mm, 2.7 µm)	Extracted with acidic methanol aqueous solution and purified using an Oasis HLB SPE cartridge
This Study /China	Mushroom, serum, urine and simulated gastric fluid	Ibotenic acid, muscimol, muscarine, β-amanitin, α-amanitin, desoxoviroidin, γ-amanitin, phallisacin, illudin S, phallacidin, phalloidin and illudin M	Mushrooms: 0.01 ~ 0.2 mg/kg; biological samples: 0.15 ~ 2.0 µg/L	ACQUITY UPLC HSS T3 column (100mm×2.1mm, 1.7µm)	Extracted with water or acetonitrile solution and the serum sample was further purified by PSA sorbent

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