|  |
| --- |
| Table 1. LC-MS methods for FBs without clean up |
| Ref | **FBs** | **Sample (g)** | **Sample treatment** | **LC conditions** | **MS conditions, Limits** |
| **Matrix** |  | **Extraction procedure** | **Column / Injection volume / Mobile Phase** **Flow / Analysis Time** | **Mass Conditions / Limits** |
| Maize and corn-based products |
| (Zitomer et al. 2008) | B1, B2, B3 | 0.01 | 1.-Add 2 mL ACN/H2O 1:1 (5% FA); 2.- Gently shaken for 3 h; 3.- Centrifugate to 15000 g; 4.- Filter; 5.- Dilute 1:10 | Metachem Inertsil ODS-3, 150 x3 mm, 5 µmInj vol 20 µL, A) H2O/ACN/FA 97:2:1, B) H2O/ACN/FA 2:97:1. 70-50% B in 9 min, 50-100% B in 2 min, keep 10 min; initial conditions for 10 min | **QTrap**CaT : 210°C |
| Maize leaf |
| Flow: 0.20 mL/min | Time: *t*an=21 min, *t*Tot=31 min | LOD: 0.01 µg/kg all FBs |
| (De Girolamo et al. 2014) | B1, B2, PHF (B1, B2), HF (B1, B2) | 20 | 1.- 100 mL MeOH/ACN/citrate-phosphate buffer 25:25:50; 2.- Shake 1h; 3.- Dilute 1:10 with MeOH/H2O 80:20 with 0.5% AcOH; 4.- Filter | Gemini C18, 150 x 2.0 mm, 5 µm at 40 °CInj vol 20 µL, A) H2O, B) MeOH, both with 0.5% AcOH40-60% B in 30 min, 60 to 40% B in 1 min; initial conditions for 9 min | **Orbitrap**CaV 45 V; SV 4 kV; RF Lens 75 V; ST 300 °C; CaT: 300 °C; SG 30 U; GF 10 skimmer V 18 V |
| Maize based products |
| Flow: 0.2 mL/min | Time: *t*an=30 min, *t*Tot=40 min | LOD: 5 µg/kg,LOQ: 10 µg/kg all FBs |
| (Beltrán et al. 2009) | B1, B2 | 2.5 | 1.- Add ACN/H2O 80:20 + 0.1% AcOH, 2.-shake 90 min, 3.-centrifuge to 4000 rpm, 10 min; 4.-dilute 1:2 with H2O, 5.-filter (0.22 mm nylon filter) | Acquity UPLC BEH C18, 50 x 2.1 mm, 1.7 µm at 40°CInj vol 20 µL, A) H2O, B) MeOH, both with 0.5 mM AmAc and 0.1% AcOH10-90 % B in 4 min, initial conditions for 3 min | **QQQ**CaV 3.5 kV; DGT 500°C; ST 120 °C; T 40 °C; DGF 1200 L/h, CoG 4 x 10-3 mbar |
| Maize, kernel, dry pasta, baby food |
| Flow: 0.3 mL/min | Time: *t*an=4 min, *t*Tot=7 min | LOD: 1 µg/kg,LOQ: 3.5 µg/kg |
| (C. Dall’Asta et al. 2008) | B1, B2, B3 | 25 | LLE1.- Add 100 mL H2O/ACN/MeOH 50:25:25, 2.- blend (6000 rpm/5 min); 3.- take 4 mL; 4.- filter; 5.- dry N2; 6.- reconstitute 1mL in H2O/ACN 1:1; 7.- filter | XTerra C18, 250 × 2.1 mm, 5 µm at 30°CInj vol 10 µL, A) H2O, B) MeOH, both with 0.1% FA0% B for 3 min, 0-45% B in 2 min, keep 5 min, 45-85% B in 15 min, keep for 10 min, initial conditions for 10 min | **QQQ**CaV 3.2 kV; CV 30 V; EV 3 V; ST 120 °C; DGT 160 °C; CGF 70 L/h; DGF 650 L/h (N2 for both) |
| Maize, maize-based products |
| Flow: 0.2 mL/min | Time: *t*an=35 min, *t*Tot=45 min | LOD: B1, B2 1 µg/kg, FB3 8 µg/kgLOQ: B1, B2 5 µg/kg, FB3 12 µg/kg |
| (Arroyo-Manzanares et al. 2018) | B1, B2 and other toxins | 2 | QuEChERS1.-Add 8 mL of H2O; 2.-shake 10 s; 3.-add 10 mL 5% FA in ACN; 4.-shake 2 min; 5.-add 4 g MgSO4 + 1 g NaCl; 6.-shake 1 min: 7.-vortex 2 min; 8.-centrifuge to 4500 rpm, 5 min, 4 °C; 9.-take 5 mL; 10-dry under N2 at 40 °C; 11.-reconstitute (0.2 mL MeOH/H2O 1:1); 12.-centrifuge to 14000 g, 5 min, 4 °C | ACQUITY HSS UPLC T3, 150 x 2.1 mm, 1.8 µm at 30 °CInj vol 10 µL, A) H2O, B) MeOH, both with 0.3% FA and 5 mM AmF5% B, keep 0.5 min, 5-94% B in 19.5 min, keep 1 min, 94-5% B in 3 min; initial conditions for 4 min | **QQQ**ST 150 °C; DGT 400 °C; NG 7 bar (N2); CGF 150 L/h; DGF 1000 L/h |
| Wheat, maize |
| Flow: 0.4 mL/min | Time: *t*an=21 min, *t*Tot=28 min | LOD; 1.28 B1, 0.25 FB2, 0.27 B3 µg/kg LOQ: 4.24 B1, 0.82 FB2, 0.89 B3 µg/kg |
| (Chiara Dall’Asta, Galaverna, et al. 2009) | B1, B2, B3 | 5 | 1.- Add 50 mL H2O/MeOH 30:70; 2.- Blend to 6000 rpm, 10 min; 3.- Stir for 60 min; 4.- re-extract the solid (same way); 5.- Filter; 6.- Dry 4 mL; 7.- Dissolve in 2 mL MeOH | Xterra C18, 250 x 2.1 mm, 5 µm, at 30 °CInj vol 5 µL, A) H2O, B) MeOH, both with 0.2% FA30% B for 2 min, 30-45% B in 3 min, 45-90% B in 20 min, keep for 10 min, 30% B in 1 min; initial conditions for 20 min | **QQQ**CaV 3.2 kV; EV 3 V; ST 120 °C; DGT 160 °C; CGF 70 L/h; DGF 650 L/h (N2, both) |
| Corn-based products |
| Flow: 0.2 mL/min | Time: *t*an=35 min, *t*Tot=56 min | LOD: FB1 4 µg/kg, B2, FB3 8 µg/LOQ: B1 B2 5, B3 12 µg/kg |
| (Chiara Dall’Asta, Mangia, et al. 2009) | B1, B2, B3 | 5 | 1.- Add 50 mL H2O/MeOH 30:70; 2.-Blend to 6000 rpm, 10 min; 3.- Stir for 50 min; 4.- Centrifuge to 3500 g, 15 min; 5.- Filter (2 mL) | Xterra C18, 250 x 2.1 mm, 5 µm at 30°CInj vol 10 µL, A) H2O, B) MeOH, both with 0.1% FA30 % B for 2 min, 30-45% B in 3 min, 45-90% B in 20 min, keep for 10 min; initial conditions for 15 min | **QQQ**CaV 4 kV; EV 2 V; ST 120°C; DGT 350 °C; CGF 50 L/h; DGF 600 L/h |
| Ground corn |
| Flow: 0.2 mL/min | Time: *t*an=35 min, *t*Tot=50 min | LOD: 5 µg/Kg |
| (Chiara Dall’Asta, Mangia, et al. 2009) | B1, B2, B3 | 5 | 1.- Add 2 ml H2O/ACN/AcOH 20:79:1; 2.- Extract 90 min in rotatory shaker; 3.- Centrifuge 3000 rpm, 3 min; 4.- Take aliquot 350 µL and dilute 1:1 with extraction solvents | Gemini C18, 150 x 4.6 mm, 5μm at 25 °CInj vol 5 µL, A) H2O/ACN/AcOH 89:10:1, B) H2O/ACN/AcOH 2:97:1, both with 5 mM AmAc0% B for 2 min, 0-100% B in 12 min, keep for 3 min; initial conditions for 4 min | **QQQ**CaV 4.0 kV; EV 3 V; ST 550 °C; CUR 10 psi |
| Corn-based products |
| Flow: 1.0 mL/min | Time *t*an=17 min, *t*total=21 min | LOD: 8 µg/kg |
| (G. B. de Oliveira et al. 2017) | B1, B2 | 1 | 1.- Add 1 g Silica gel as dispersant; 2.- Mix in polypropylene cartridges, MSPD; 3.- Elute with 16 mL of 20 mM AmFo buffer:MeOH 9:1 (pH 7); 4.- Collect 2 mL fractions; 5.- Centrifuge to 4000 rpm, 10 min; 6.- Filter | Poroshell, C18, 100 x 3 mm, 2.7 µm, 40 °CInj vol 10 µL, A) Ultrapure H2O, B) ACN, both with 0.1% FA20-90% B in 3 min, keep 0.4 min, 90-20 % B in 0.1 min; initial conditions for 6 min | **QQQ**CaV 4.5 kV; EP 10 V; DGT 650 °C; NG 40 CUR 18 a.u, |
| Maize |
| Flow: 0.5 mL/min | Time: *t*an=3.4 min, *t*Tot=9.5 min | LOD: B1 514, B2 176 µg/kgLOQ: B1 594, B2 210 µg/kg |
| (D’Arco et al. 2008) | B1, B2, B3 | 3 | 1.- Add 100 µL of a 5 µg/mL Fbs solution (0.5 µg) and keep 15 min at RT; 2.-pack into 11 mL PLE pressure resistant stainless steel extraction cell; 3.-elute with 22 mL of MeOH 60% at 40°C and 34 atm, 2 min of preheating, 5 min of static time, 60 s of purge time; 4.-concentrate to 5 mL (40 °C and 80 mbar); 5.-transfer to a 15 mL conical tube; 6.-evaporate to dryness at 55°C with N2 ; 7.reconstitute 1 mL MeOH/H2O 50:50; 8.-filter | Luna C18, 150x4.6 mm, 5 µm (Temp NR)Inj vol NR, A) H2O, B) MeOH, both with 0.5% FA65% B for 3 min, 65-95% B in 4 min, keep 3 min, initial conditions in 10 min | **QQQ**CaV 3.20 kV; CoV 50 V; EV 3 V; RF lens 0.2 V; ST 125 °C; DGT 300 °C; DGF 500 L/h; CGF gas 50 L/h |
| Corn-based baby food |
| Flow: 0.30 mL/min | Time: *t*an=10 min, *t*Tot=20 min | LOD: 0.7 B1 and B2, 1.5 µg/kg B3 LOQ: 2 B1 and B2, 5 µg/kg B3 |
| (Chiara Dall’Asta, Mangia, et al. 2009) | B1, B2, B3 | 100 | 1.- Add 50 mL KOH 2M; 2.-Centrifuge to 6000 rpm, 10 min; 3.- Stir (50 min); 4.- Add 50 mL ACN; 5.- Stir 10 min; 6.- Separate 20 mL and dry under N2; 7.- Redissolve in 50 mL KOH 2M; 8.- Centrifuge to 3500 rpm, 15 min; 9.- Dry under N2; 10.- Redissolve in H2O/MeOH 30:70 | Hypersil C18, 150 x 2.1 mm, 5 μm at 25°CInj vol 10 µL, A) H2O, B) MeOH, both with 0.2% FA20% B for 1 min, 20-100% B inwalnut 5 min, keep 3 min, initial conditions for 4 min | **QQQ QTrap**CaV 4 kV; CoV 50 V; ST 425°C; DGT 350°C; CGF 50 L/h; DGF 600 L/h (N2, both) |
| Raw corn |
| Flow: 0.6 mL/min | Time: *t*an=8 min, *t*Tot=13 min | LOD: <15 µg/kg |
| (Hu et al. 2019) | B1, B2 | 1 | 1.- 10 mL ACN/H2O/AcOH 70:29:1; 2.- Shake 30 min; 3.- Centrifuge to 4500 rpm, 10 min; 4.- Filter supernatant; 5.- Take 1 mL; 6.- Add 10 µL, 1 µg/mL 13C-34 FB1 and 13C-34 FB2 | Luna C18, 150 x 2 mm, 3 µm at 40°CInj vol 5 µL, A) H2O, B) MeOH, both with 2 mM AmAc40-90% B in 6 min, keep 1 min, 90-100% B in 1 min, keep 1 min, 100-40% B in 2 min; initial conditions for 4 min | **Qtrap**CaV 5.5 kV; EP 10 V; ST 600°C; CUR 40 psi; CoV 10 V; dwell time 100 ms |
| Raw maize |
| Flow: 0.2 mL/min | Time: *t*an=9 min, *t*Tot=15 min | LOD: 7 B1; 6 B2 µg/kgLOQ: 28 B1; 27 B2 µg/kg |
| (Bergmann, Hübner, and Humpf 2013) | B1 | 10 | 1.- Add 20 mL ACN/H2O 70:30 with 1% FA; 2.-Vortex 30 s; 3.- Sonicate 10 min; 4.- Shake 15 min; 5.- Centrifugate to 8000 g, 15 min, 25 °C; 6.- Dilute 1:1 1% FA; 7.- Filter if necessary | Hyperclone C8 BDS, 150 x 2.0 mm, 3 µm at 40° CInj vol 20 µL, A) H2O, B) ACN, both with 1% FA65% B for 4 min, 37.5% B for 0.5 min, 5% B for 2 min, keep for 0.5 min, initial conditions for 4 min | **QTrap**CaV 5.5 kV; DG 350 °C; NG 35 psi; DG 45 psi; CUR (N2) 30 psi; CoG 5 x 10−5 Torr; QTrap CUR 20 psi |
| Maize |
| Flow: 0.30 mL/min | Time: *t*an=7 min, *t*Tot=11 min | LOD: 53 µg/kg, LOQ: 188 µg/kg |
| (de Matos et al. 2021) | B1, B2, HB1, HB2 | 5 | 1.- Add ACN:H2O:FA 75.24:1; 2.- shake for 2 min; 3.- sonicate for 10 min; 4.- centrifuge at 3000 rpm for 7 min; 5.-take 0.05 mL of extract; 6.- dilute with 0.95 mL 0.05% of AF in MeOH:H2O 1.1; 7.- filter | ACQUITY BEH C18 100 x 2.1 mm, 1.7 μm at 35°C5 μL of sampleA) H2O (0.1% FA), B) MeOH65-80% B in 3 min, hold for 1 min, 100% B in 1 min, initial condition for 2 min | **QQQ**CaV: 3kV; DGT: 400 °C; ST: 150 °C; CGF: 15 L/h; DGF:750 L/h |
| Corn products |
| Flow 0.3 mL/min | Time: tan= 5 min, tTot= 7 min | LOD: (B1: 0.43-1.98, FB2 0.19-1.37, HB1 0.72-1.39, HB2 0.36-0.70) μg/KgLOQ: (B1:1.43-6.59, FB2 0.60-4.60, HB12.40-4.60, HB2 1.20-2.30) μg/Kg |
| (Lin et al. 2011) | B1, B2 | 5 | 1.- Add 25 mL MeOH/H2O 3:1; 2.-Ultrasonic bath for 10 min at RT, output powder 120 W; 3.- Centrifugate to 5000 g, 5 min; 4.- Filter (0.22 mm nylon filter) | Zorbax Eclipse XDB-C18, 150 x 2.1 mm, 3.5 µm at 30°CInj vol 10 µL, MeOH/H2O/FA 75:25:0.2 | **Q**CaV 3.5 kV; CoV 50 V; ST 120 °C; DGT 350°C; DGF 600 L/h |
| Corn |
| Flow: 0.20 mL/min | Time *t*an=total= 4 min | LOD: 3.5 B1, 2.5 µg/kg B2LOQ: 11.7 B1, 8.3 µg/kg B2 |
|  |  |  |  |
| (A. S. Silva et al. 2019) | B1, B2 | 2 | 1.- Add 10 mL ACN 80%; 2.- Shake at 110 rpm, 1h; 3.- Centrifuge to 3000 rpm, 10 min; 4.- Remove supernatant; 5.- Re-extract the solid, same way; 6.- Centrifuge to 3000 rpm, 10 min; 7.- Dilute 1:1 with H2O; 8.- Filter | Zorbax Eclipse Plus C18, 2.1 x 50 mm, 1.8 µm at 30 °CInj vol 20 µL, A) 0.1% FA, B) ACN10-70% B in 12 min, 70-90% B in 1 min, keep 1 min, 90-10% B in 1 min, initial conditions for 2 min | **TOF**CaV 5.5 KV; ST 575 °C; CUR 30 psi; Gas 1 and Gas 2, 55 psi both; DP 100 V; Full scan 100-750 Da |
| Maize flour |
| Flow: 0.5 mL/min | Time: *t*an=14 min, *t*Tot=17 min | LOD: 62.5 µg/kg,LOQ: 125 µg/kg all FBs |
| Other cereal and seeds |
| (Bartók et al. 2006) | B1, B2, B3, its analogs | 3 | 1.- Add 25 mL of ACN/H2O 75:25; 2.- Centrifuge to 13,500 rpm, 1 min; 3.- Shake 1 h; 4.- Centrifuge to 10,000 g, 10 min; 5.- Filter | Supelcosil ABZ Plus, 250 x 2.1 mm, 5 µm at 40 °CInj vol 1 µL, A) H2O, B) ACN, both with 0.1% FA25-40 % B in 22 min, 40-100% B in 5 min, keep for 3 min. | **QTrap**CaV 3.5 kV; EV 200 V; HED Voltage 7 kV; NG 40 psi; DGF 9 L/min; DGT 350 °C; trap drive 53.9; max accumulation time 300 ms; full scan 50-1100 *m/z* |
| Rice |
| Flow: 0.3 mL/min | Time: *t*an=27 min, *t*Tot=30 min | LOD / LOQ: NR |
| (Soleimany, Jinap, and Abas 2012) | B1, B2 | 10 | 1.- Add 40 mL H2O/ACN/AcOH 20:79:1; 2.- Shake 60 min; 3.- Centrifuge the supernatant at 3000 rpm, 10 min; 4.- Dilute 1:1 in H2O/ACN/AcOH 79:20:1; 5.- Filter | Thermo Scientific C18, 150 x 4.6 mm, 3 µm at 30°CInj vol 20 µL; A) H2O, B) MeOH both with 0.1% AcOH5% B for 8 min, 5-90% B in 14 min; 90-5% B in 3 min | **QQQ**CaV 3 kV; ST 120°C; DGT 400 °C; spray gas N2 |
| Cereals |
| Flow: 0.25 mL/min | Time: *t*an=22 min, *t*Tot=25 min | LOD: 20 ng/g, LOQ: 40 ng/g |
| (Rausch, Brockmeyer, and Schwerdtle 2020) | B1, B2, B3 and other toxins | 1 | QuEChERS1.- Add 2 mL H2O, 2.-mix 1 min, RT, 10 min; 3.- extract with 8 mL ACN/FA 75:5; 4.- Shake 15 min; 5.- add 4 g anhydrous MgSO4, 1 g NaCl, 1 g Na2HCit 1.5 H2O, Na3Cit 2 H2O, 6.- Mix 1 min; 7.- Shake 15 min; 7.- Centrifuge to 2140 g, 2 min; 8.- Filter; 9.- Take 500 µL, dry; 10.- Redissolved in 250 µL MeOH/H2O 20:80 | Raptor Fluoro Phenyl 50 x 2.1 mm, 2.7 µm in series withRaptor Biphenyl 50 x 2.1 mm, 2.7μm at 30 °CInj vol 10 µL, H2O, 0.3% FA, B) MeOH, both with 5 mM AmFo20% B for 0.6 min, 20-40 % B in 0.4 min, 40-90% in 8 min, keep 1 min, initial conditions for 3.5 min | **QQQ**CaV 4.5 kV; ST 500 °C; CUR 40 psi; ISG 1 60 psi; ISG 2 65 psi |
| Cereals |
| Flow: 0.4 mL/min | Time: *t*an=10 min, *t*Tot=13.5 min | LOQ: depending on the matrix, FBs 4-15 µg/kg |
| (Aurelien Desmarchelier et al. 2010) | B1, B2 and other mycotoxins | 5 | QuEChERS1.- Add 10 mL H2O + 10 mL 0.5% AcOH in ACN; 2.- Shake at 300 rpm, 5 min; 3.- Add 5 g MgSO4/NaCl 4:1, 4.- Shake; 5.- Centrifuge to 4000 g, 15 min, RT; 6.- Take 5 mL; 7.- Shake at 200 rpm, 5 min; 8.- Centrifuge to 4000 g, 1 min; 9.- Dry 1 mL at 40 °C (N2); 10.- Add 75 µL MeOH; 11.- Sonicate; 12.- Add 75 µL H2O, mix; 13.- Centrifuge to 8500 g, 10 min, RT; 14.- Dilute 60 µL with 140 µL H2O; 15.- Centrifugate to 8500 g, 10 min, RT | Zorbax Bonus-RP, 150 x 2.1 mm, 3.5 µmA) H2O 0.15% FA, 10 mM AmFo, B) MeOH 0.05% FA15% B 0.5 min, 15-100% B 8.5 min, keep for 6 min, 15% B in 1 min, initial conditions for 9.5 min | **QTrap SRM**ST 550 °C; NG 50 psi; CUR 40 psi; TG 30 psi; CoG 1.2 x 10-4 psi |
| Cereals |
| Flow: 0.25 mL/min | Time: *t*an=15 min, *t*Tot=25.5 min | LOQ: 50 µg/kg all FBs |
| (Liao et al. 2013) | B1, B2 and other toxins | 1 | 1.-Add 5 mL H2O/ACN 15:85; 2.-shake to 1550 rpm, 30 min; 3.-centrifugate to 4500 rpm, 5 min; 4.-take 500 µL; 5.-add 20 µL of 13C-34 FB1 (25 µg/mL) + 480 µL 20 mM FA; 6.-vortex 15 s; 7.-filter | Ultra-Aqueous C18, 100 x 2.1 mm, 3 μm, at 40 °CInj vol 10 µLA) H2O, B) MeOH, both with 0.1% FA+ 10 mM AmFo10 % B for 1 min, 10-100% B in 6 min, keep for 3 min, initial conditions for 5 min | **QTrap**Conditions NR |
| Finished grain, nut products |
| Flow: 0.5 mL/min | Time: *t*an=10 min, *t*Tot=15 min | LOD: FBs 2.2-2.9 µg/kg, LOQ: FBs 7.3-9.6 µg/kg, depending on the matrix |
| (Bartók et al. 2010) | Isomers of B1 | 1 | 1.-8 mL MeOH/H2O 75:25; 2.-homogenize 9,500 rpm, 4 min: 3.-centrifuge to 10,000 rpm, 10 min, 4.-filter | YMC-Pack J’sphere ODS H80, 250 x 2.1 mm, 4 µm, 40 °CInj vol 1 µLA) H2O, B) ACN, both with 0.1% FA24-40% B for 79 min, 40-100 % B for 15 min, keep for 10 min | **TOF, full scan MS**CaV 3.5 kV; Fragmentor 170 V; skimmer 70 V; DGT 350 °C; DGF 10 mL/min; NG 20 psi; full scan 100-1700; acquisition rate 250 ms/spectrum |
| Rice |
| Flow: 0.20 mL/min | Time: *t*an=79 min, *t*Tot=104 min | LOD/LOQ: NR |
| (Oueslati et al. 2012) | B1, B2 | 5 | 1.- Add 10 mL ACN/H2O 80:20; 2.-vortex 2 min, shake 60 rpm x 10 min; 3.-centrifuge to 5000 rpm, 5 min; 4.-filter 2 mL (0.20 μm, Millipore) | Acquity UPLC BEH C18, 100x2.1 mm, 1.7 µm at 30°CInj vol 5 µL, A) H2O with 5 mM AmFo, B) MeOH25-75% B in 3 min, 75-100% B in 2 min, keep for 1.5 min, 100-25% B in 1 min; initial conditions for 1 min | **QQQ**CaV 3.5 kV; CoV FB1 45 V, FB2 55 V; EV 3 V; ST 120 °C; DGT 350 °C; CGF 50 L/h; DGF 650 L/h |
| Cereals, derived products |
| Flow: 0.35 mL/min | Time: *t*an=6.5 min, *t*Tot=8.5 min | LOD: B1 and B2 1 µg/kgLOQ: B1 and B2 5 µg/kg |
| (Rausch, Brockmeyer, and Schwerdtle 2021) | B1, B2, B3, HB1, HB2, HB3 | 2.5 | 1.- Add ACN:H2O:FA 79.20:1; shake for 15 min at RT; 3.- Add 20 µL of Deuterated internal standard; 4.- rotary agitation for 30 min; 5.- centrifuge at 1902 g, 6.- take an aliquot of supernatant, 7.- filter | First dimension: YMC-Pack Diol-NP C18 100 × 2.1 mm, 5 μm at 40 °C.Vol. inj: 10 μL of sampleA) H2O, B) ACN:H2O 90:10 Both (0.1% FA, 10 mM AmFo)100% B in 2.5 min, 100-90% B in 0.5 min, 90-20 % B in 0.8 min, hold for 3.8 min, 20-100% B in 0.20 min. initial condition for 17.20 min.Second dimension: 2 columns connected in series RaptorFluoroPhenyl, 50 × 2.1 mm, 2.7 μm and Raptor Biphenyl50 × 2.1 mm, 2.7 μm,5% B for 1.2 min, 5-0% B in 0.10 min, hold for 7.15 min, 0-5% B in 0.05 min, 5-50% B in 1.1 min, 50-70% B in 4.4 min, 70-85% B in 2.5 min, 85-100% B in 3 min, hold for 2 min, 100-5% B in 0.10 min, initial condition for 4 min | **QQQ**CaV: 4.5 kV; CUR: 40 psi; ST: 500 °C; |
| Cereals |
| Flow 0.2 mL/ min, 0.3 ml/min | Time: tan= 7.6 min tTol= 25 minTime: tan= 15.50 min tTol= 25 min | LOQ: (B1-3: 10, HB1-3: 100) μg/Kg |
| Other samples |
| (Škrbić, Živančev, and Godula 2014) | B1, B2 and other toxins | 10 | 1.- Add 40 mL ACN/H2O/AcOH 79:20:1; 2.- Shake 1h; 3.- Filter; 4.- Take 20 mL; 5.- Add 20 mL hexane; 6.- Mix 2 min; 7.- Centrifuge to 5000 rpm, 5 min; 8.- Eliminate hexane phase. 9.- Filter aqueous phase | Hypersil GOLD C18, 50 x 2.1 mm, 1.9 µm at 25 °CInj vol 10 µL, A) H2O, B) MeOH, both with 1% AcOH and 5 mM AmAc5 % B for 0.5 min, 5-95 % B in 2.5 min, keep 2 min, 95-5% B in 1.2 min, initial conditions for 1.8 min | **QQQ**CaV 3.4 kV; ST 350 °C; SG 40 arbitrary units; aux gas 10 arbitrary units; CaT 270 °C |
| Crude extracts of nuts |
| Flow: 0.50 mL/min | *t*an=6 min, *t*Tot=8 min | LOD: 0.24 B1, 0.05 B2 µg/kgLOQ: 0.8 B1, 0.17 B2 µg/kg |
| (Yibadatihan, Jinap, and Mahyudin 2014) | B1, B2 and other toxins | 5 | 1.- Add 20 ml H2O/ACN/FA 20:79:1; 2.- Shake 60 min; 3.- Centrifuge supernatant to 3000 rpm, 10 min; 4.-Dilute 1:4 with water; 5.-Filter | Symmetry C18, 150 x 2.0 mm, 3μm, 30 °CInj vol. 25 µL, A) H2O, 0.2% FA, B) MeOH10% B for 8 min, 10-90 % B in 2 min, keep 7 min, from 90-10% B in 3 min, initial conditions for 5 min | **QQQ**CaV 3 kV; ST 120 °C; DGT 350 °C |
| Palm kernel cake |
| Flow: 0.20 mL/min | Time: *t*an=17 min, *t*Tot= 25 min | LOD both: Std 5.6 µg/kg LOQ both: Std 18 µg/kgLOD both: Samples 17.5 µg/kg LOQ both: samples 58 µg/kg |
| (Qian et al. 2018) | B1, B2 and other toxins | 2 | QuEChERS1.- Add 1.5 g NaCl + 10 mL 3% AcOH in ACN/H2O 80:20; 2.-Vortex 1 min, 3.-Ultrasound 20 min; 4.-Add 2 g anh MgSO4; 5.-Vortex 1 min; 6.-Centrifuge to 8000 rpm, 5 min; 7.-Dry (N2, 40 °C); 8.-Dissolve in MeOH:H2O 1:1; 9.-Filter | ACQUITY UPLC HSS T3, 100 x 2.1 mm, 1.8 µm at 40°CInj vol. 5 µLA) H2O, 0.1% FA, 1 mM AmAc; B) MeOH0-10% B in 1 min, 10-20% B in 2 min, 20-99% B in 8 min, keep 2.5 min; 99-10% B in 0.1 min; initial conditions for 5 min | QQQCaV 5.5 kV; ST 550°C; Auxiliary gas 40 psi |
| Feed |
| Flow: 0.3 mL/min | Time: *t*an=13.5 min, *t*Tot= 18.5 min | LOQ: 0.4 µg/kg for both B1 y B2 |
| (Spanjer, Rensen, and Scholten 2008) | B1, B2, B3 and other toxins | 25 | 1.- Add 100 mL ACN/H2O 80:20, 2.- Shake 2h; 3.-Dilute 1:4 with H2O; 4.- Filter if necessary(For raisins and figs use MeOH) | Alltima C18, 150 x 3.2 mm, 5 µm at 30 °CInj vol 20 µL, A) H2O, B) ACN, both with 0.1% FA10-70% B in 12 min (curve 1), keep 4 min, 70-90 % B in 1.5 min (curve 6), keep 2.5 min, 90-10 % B in 1 min (curve 1), initial conditions for 5 min | **QQQ**CaV 2.5 kV; CoV 75 V; DGT 450°C; CGF 100 L/h (N2); DGF 600 L/h |
| Peanut, pistachio, wheat, maize, cornflakes, raisins, figs |
| Flow: 0.3 mL/min | Time: *t*an=20 min, *t*Tot=25 min | LOQ: depending on the matrix, B1 5-100 µg/kg, B2 1-100 µg/kg |
| (Aurélien Desmarchelier et al. 2014) | B1, B2 and other toxins | 25 | 1.- Add 50 mL H2O, 2.- Homogenize 1 min 10000 rpm, 3.-Take 5 g of sample (peanut, green cofee, cocoa, paprika) or 2 g (infant formula, sunflower oil), 4.-Add 100 µL of 13C-FB standard (FB1 and FB2 each 10 µg/mL), 5.-Add 10 mL H2O and 10 mL ACN, 0.5% AcOH, 6.- Add 5 g MgSO4:NaCl 4:1 Centrifuge 4000g, 15 min, 7.-Defat 5 mL ACN phase with 5 ml hexane. 8.- Take 1 mL of ACN phase, dry, 9.-Reconstitute in 150 µL H2O/MeOH 1:1, 10.-Centrifuge 8500 g, 10 min, 11.-Take 60 µL, add 140 µL H2O, 12.-Centrifuge 8500 g, 10 min | Zorbax Bonus-RP C18, 150 x 2.1 mm, 3.5 µm at 50 °CInj vol 20 µLA) H2O, 0.15% FA, 10 mM AmFo, B) MeOH, 0.05% FA15% B for 0.5 min, 15-100 % B in 6 min, keep for 4.5 min, 100-15% B in 0.5 min, initial conditions for 7.5 min | **QTrap, QQQ**ST 550 °C; CUR 40 psi, Nebulizer 50 psi; Turbo gas 30 psi |
| Cereals, cocoa, oil, spices, infant formula, coffee, nuts |
| Flow: 0.35 mL/min | Time: *t*an=11 min, *t*Tot= 19 min | LOD/LOQ: NR |
| (Shar et al. 2020) | B1, B2 and other toxins | 5 | 1.- Add ACN/H2O/FA 79:20:1; 2.- Shake for 90 min to 180 rotations/s; 3.-Centrifuge to 4000 rpm, 2 min, 4.-Filter | Acquity C18, 100 x 2.1 mm, 1.8 µm, 40 °CInj vol 20 µLA) H2O, 1% FA, B) MeOH/H2O/FA, 97:2:1, both with 10 mM AmFo.0% B for 2 min, 0-50% B in 0.5 min, 50-100% B in 3.5 min, keep 1 min, initial conditions in 1 min, seal wash for 5 min | **sQ**CaV 2.79 kV; ST 150 °C; DGT 350 °C; CGF 50 L/h; CGF 600 L7h |
| Feed, its ingredients |
| Flow: 0.5 mL/min | Time: *t*an=7 min, *t*Tot= 8 min | LOD B1: 0.07 µg/kg,LOQ B1: 0.22 µg/kgLOD B2: 0.03 µg/kg,LOQ B2: 0.08 µg/kg |
| (Frenich et al. 2009) | B1, B2 | 5 | 1.- Add 10 mL ACN/H2O 80:20 (for biscuit add 20 mL); 2.- Vortex 2 min; 3.- Shake to 60 rpm, 10 min; 4.- Centrifuge to 4500g, 5 min; 5.- Take and filter 2 mL | Acquity C18, 100 x 2.1 mm, 1.7 μm at 30°CInj vol 5 µL, A) H2O with AmFo 5 mM, B) MeOH25-75% B in 3 min, 75-100% B in 2 min, keep for 1.5 min, 100- 25% B in 1 min; initial conditions for 1 min | **sQ**CaV 3.5 kV; EV 3 V; ST 120°C; DGT 350°C; CGF 50 L/h; DGF 650 L/h (N2 for both) |
| Maize, walnut, breakfast cereal, biscuit |
| Flow: 0.35 mL/min | Time: *t*an=6.5 min, *t*Tot=8.5 min | LOD maize: B1 0.1 µg/kg, B2 0.2 µg/kg,LOQ maize: B1 0.5 µg/kg, B2 0.6 µg/kg; LOD breakfast cereal: B1 2.1 µg/kg, B2 0.7 µg/kg LOQ breakfast cereal: B1 6.2 µg/kg, B2 2.5 µg/kg |
| Beverages |
| (Rubert et al. 2011) | B1, B2, B3 and other toxins | 10 mL | 1.- Sonicate 25 min, 2.-Condition SPE Oasis HLB cartridges with 5 mL ACN/MeOH 1:1; 3.- 5 mL H2O; 4.- 10 mL sample into cartridge; 5.-Wash with 5 mL H2O; 6.- Dry 30 min; 7.- Eluate with 4mL ACN:MeOH 1:1; 8.- Dry (N2, 35 °C), 9.- Reconstitute in 1 mL (ACN/MeOH 1:1); 10.-Filter | Gemini C18, 150 x 2.0 mm, 5 µm, at 35 °CInj vol 10 µLA) H2O, 0.1% FA, B) MeOH, both with 5 mM AmFo5-95% B in 10 min, 95-80% B in 5 min, initial conditions 5 min | QQQ Orbitrap XLCaV 30 V; SV 4 kV; Source Temp 275 °C; Capillary gas sheat 35 units; auxiliary gas 30 arbitrary units |
| Beer |
| Flow: 0.2 mL/min | Time: *t*an=10 min, *t*Tot= 20 min | LOD: 30-35 µg/L, LOQ: 90-105 µg/L all Fbs depending of the beer type |
| (Huang et al. 2018) | B1, B2 and other toxins | 2 | QuEChERS1.-Add 100 µL of D-atrazine (60 µg/L), 15 mL acetate buffer pH 3.0, 10 mL 5% FA in ACN; 2.- Shake; 3.- Extract with ultrasonic (53 KHz, 5 min, 20°C); 4.- Add 4 g MgSO4 + 1 g NaCl + 0.5 g Na2HCit·1.5H2O, 1 g Na3Cit·2H2O; 5.- Shake to 1500 strokes/min, 5 min; 6.- Ice bath 10 min, 7.-Centrifuge to 18514 g, 10 min; 8.- Take 6.0 mL; 9.- Transfer supernatant into 15 mL centrifugation tube containing 900 mg MgSO4, 600 mg C18, 150 mg PSA, 150 mg Si; 10.- Shake 5 min, 11.- Centrifuge 10 min; 12.- Take 2 mL, reduce volume <0.5 mL with N2; 13.- Complete to 1 mL with H2O/MeOH 80:20; 14.-filter | Poroshell EC-C18, 150 x 3 mm, 2.7 µm at 20°CA) H2O, B) MeOH, 0.2% FA and 2 mM AmFInj vol 5 µL20% B for 2 min, 20-50% B in 2 min, 50-100% B in 7 min, keep 1 min, 100-20% B in 1 min, initial conditions for 2 min | **QQQ**CaV 5.5 kV; DP 150 eV; EP 10 eV; CUR 30 psi; GS1: 50 psi, turbo gas (gas 2) 50 psi, GT: 450°C |
| Liquorice |
| Flow: 0.45 mL/min | Time: *t*an=12 min, *t*Tot= 15 min | LOD: B1, B2, 0.05 µg/kgLOQ: B1. B2, 0.125 µg/kg |
| (Tamura et al. 2012) | B1, B2, B3 and other toxins | 5 mL | 1.-Add 25 mL AmAc 10 mM, mix, 2.-wash in Oasis HLB SPE Cartridge conditioned with 5 mL AmAc 10 mM, 3.-elute with 5 mL AmAc 10 mM/ACN 1:1, 4.-elute 5 mL ACN, mix, dry N2 40°C, 5.-dissolve in 1mL H2O, 6.-60 µL FA + 5 mL ACN, mix, 7.-apply to multistep #229 Ochra cartridge. 8.-Dry 4 mL of eluate with N2 40°C, 9.-dissolve in 500 µL AmAc 10 mM/ACN 85:15, 10.-filter | Acquity UPLC BEH C18, 100 x 2.1 mm, 1.7 µm at 40°C1. H2O; B) MeOH, with 2% AcOH, 0.1 mM AmAc

Inj vol 5 µL55-80% B in 5 min, initial conditions for 2 min | **QQQ**CaV 3 kV; ST 120°C; DGT 450 °C; CGF 50 L/h; DGF 800 L/h |
| Wine |
| Flow: 0.3 mL/min | Time: *t*an=5 min, *t*Tot= 7 min | LOD: 0.30 µg/L, LOQ: 1 µg/L all Fbs |
| (Miró-Abella et al. 2017) | B1, B2 and other toxins | 10 mL | 1.- Add 10 mL 1% FA in ACN in a 50 mL centrifuge tube, 2.- Shake 3 min; 3.- Add 4 g MgSO4 + 1 g NaCl; 4.- Shake vigorously 3 min; 5.- Centrifuge to 10000 rpm, 5 min, 20ºC, 6.-dilute 1:1 with phase A 7.-filter | Cortecs UHPLC C18, 100 x 2.1 mm, 1.6 μm at 40°CInj vol 5 µLA) H2O, B) MeOH, both with 0.1% AcOH, 5 mM AmAc10-50% B in 4.5 min, 50-95% in 7.5 min, keep 2.5 min | **QQQ**CaV 4 kV; DGF 18 L/min; DGT 160ºC; nebulizer 35 psi; nozzle voltage 0.5 kV; Frag Vol 380 V |
| Plant-based beverages |
| Flow: 0.45 mL/min | Time: *t*an= 14.5 min, *t*Tot= NR | LOD: 0.80; LOQ: 2.68 µg/kg all Fbs |
| (B. Zhang et al. 2018) | B1 and other toxins | 5 | 1.- Add 5 mL distilled H2O, 10 mL 1% AcOH in ACN; 2.- Shake to 3000 rpm; 3.- Add 1 g NaCl + 4 g MgSO4, 4.- Centrifuge to 13000 rpm, 5 min, 10 °C; 5.- Transfer into 10 mL polypropylene tube containing 450 mg MgSO4; 6.- Shake 30 s; 7.-Centrifuge to 5000 rpm, 5 min, 10 °C | ZORBAX RRHD Eclipse Plus C18, 50 x 2.1 mm, 1.8 µm at 30°C, Inj vol 2 µLA) H2O, B) ACN, both with 0.1% FA10-42% B in 2.4 min, 42-51% B in 3.6 min, 51-95% B in 0.2 min, 95-10% B for 0.8 min, initial conditions for 5 min | **QQQ**CaV 4 kV; DG temperature 350 °C; DG flow 10 L/min; Nebulizer 40 psi |
| Grapes, wines |
| Flow: 0.3 mL/min | Time: *t*an=6.2 min, *t*Tot= 12 min | LOD: 1 µg/L, LOQ: 3 µg/L |
| (Pizzutti et al. 2014) | B1, B2, B3 and other toxins | 5 | 1.- Add 5 mL H2O, 10 mL 1% AcOH in ACN, 25 µg/mL of: FB1 (ACN/H2O 1:2), FB2 (CAN/H2O 1:3), and FB3 (ACN); 2.-Mix to 300 rpm, 1 min; 3.- Add 3 g anh. MgSO4; 4.- Shake 1 min; 5.- Centrifuge 13000 rpm, 5 min, 6.-Take 3 mL of superior phase; 6.- Mix with 450 mg anh. MgSO4; 7.- Mix 10 s, centrifuge 4000 rpm, 4 min, 10 °C; 8.- Filter and dilute1:1 with MeOH | Acquity UPLC BEH C18, 100 x 2.1 mm, 1.7 µm, 50 °CInj vol 5 µLA) H2O, B) ACN, both with 0.1% FA10-70% B in 10 min, 90 % B for 2 min, initial conditions for 1 min | **QQQ**CaV 2 kV; ST 120 °C; DGT 400 °C; DGF 100 L/h; CGF 700 L/h |
| Wines |
| Flow: 0.4 mL/min | Time: *t*an=12 min, *t*Tot= 13 min | LOQ: 50 µg/kg all Fbs |
| (Pérez-Ortega et al. 2012) | B1 and other toxins | 4 mL | Oasis HLB, Bond Elut Plexa1.- SPE cartridges preconditioned with 4 mL MeOH, 2.- 4 mL H2O at 2 mL/min; 3.- Add sample into cartridge; 4.- Elute with MeOH/H2O 5:95; 5.- Dry in vacuum 1 min; 6.- Elute twice/4 mL MeOH, 1 mL/min; 6.- Evaporate (N2, 37°C); 7.-Reconstitute (1 mL MeOH:H2O 2:8); 8.- Filter | Zorbax Eclipse XDB-C18, 50 x 4.6 mm, 1.8 µm, temp NRInj vol 20 µLA) H2O, 0.1% FA; B) ACN10 % B for 2 min, 10-50% B in 3 min, 50-100% B in 10 min, keep 3 min | **TOF**CaV 4kV; NGP 40 psi; DGF 9 L/min; DGT 325 °C; Frag Vol 190 V; range 50 -1000 |
| Wine |
| Flow: 0.5 mL/min | Time: *t*an=18 min, *t*Tot= NR | LOD: 0.8 µg/L, LOQ: 2.68 µg/L |
| Samples of animal origin |
| (Cao et al. 2018) | B1, B2 | 200 µL urine200 µLplasma | 1.- Add 50 µL β-glucuronidase + 20 µL SI (13C34-FB1 1 mg/mL); 2.-incubate 37 °C overnight; 3.-centrifuge to 10000 rpm, 5 min; 4.-take supernatant, add 730 µL H2O/ACN 90:10; 5.-filter1.- Add 50 µL β-glucuronidase + 20 µL SI (13C34-FB1 1 mg/mL); 2.-incubate 27°C overnight; 3.-add 1mL ACN:AcOH 99:1; 4.-vortex 30 s; 5.-centrifuge to 5000 rpm, 10 min; 6.-dry at 45°C; 7.-reconstitute in 200 µL of H2O:ACN 9:1; 8.-mix 30 s; 9.-filter | Kinetex C18, 100 x 2.1 mm, 2.6 µm, 40°CInj vol 10 µLA) H2O, 0.2 mmol/L AcOH; B) MeOH25% B for 1 min, 25-70% B in 2 min, 70-25% B in 0.5 min, initial conditions for 1.5 min | **QQQ, TISP**CUR 20 psi; CoG (CAD) 8 psi; GS1 20 psi; GS2 15 psi; GT 600℃; EP 10.0; CP 12.0 |
| Urine, plasma |
| Flow: 0.2 mL/min | Time: *t*an=3 min, *t*Tot= 5 min | LOD B1:urine 0.12 µg/L, LOQ B1: urine 0.45 µg/LLOD B1: plasma 0.19 µg/L, LOQ B1: plasma 0.39 µg/L |
| (Devreese et al. 2012) | B1 and other toxins | 250 µL | 1.- Add 12.5 µL 13C-34 FB1 (25 µg/mL in ACN) + 750 µL ACN (deproteinization); 2.-vortex 15 s; 3.-centrifuge to 8517 g, 10 min, 4°C; 4.-evaporate supernatant (N2, 45 °C); 5.-reconstitute with 200 µL H2O/MeOH 85:15; 6.-vortex 15 s, 7.-filter | Hypersil Gold C18, 50 x 2.1 mm, 1.9 μm at 45 °CInj vol 2.5-10 µL, A) H2O with 0.1% AcOH, B) MeOH35 % B for 1.5 min, 90 % B in 0.5 min, keep 1.5 min, 90-35 % B in 0.2 min, initial conditions 2.3 min | **QQQ**CaV 4 kV, ST 300 °C; Aux gas 18 au; ISGP 4 au; SGP 23 au; VT 300 °C; |
| Pig plasma |
| Flow:0.30 mL/min | Time: *t*an=3.5 min, *t*Tot= 6 min | LOD: 0.8 µg/L, LOQ: 1 µg/L |
| (Arroyo-Manzanares, García-Campaña, and Gámiz-Gracia 2013) | B1, B2 | 2 | QuEChERS1.- Add 8 mL of 30 mM NaH2PO4 (pH 7.1); 2.-vortex 10 s; 3.-add 5 mL ACN with 5% FA; 4.- shake 2 min; 5.-sdd 4 g MgSO4 + 1 g NaCl + 1 g NaCit + 0.5 g Na2HCit 1.5 H2O; 6.- shake 1 min; 7.-centrifuge to 4500 rpm, 5min); 8.-take 1 mL; 9.- dry; 10.-reconstitute with 1 mL MeOH/H2O 1:1; 11.-filter | Zorbax Eclipse C18, 50 x 2.1 mm, 1.8 μm at 35 °CInj vol 5 µL, A) H2O, B) MeOH, both with 0.3% FA, 5 mM AmFo5-50% B in 1 min, 50-72 % B for 2 min, 72-80 % B for 2 min, 80-90 %B for 2 min, 90-5% B in 0.2 min | **QQQ**ST 500 °C; CUR 30 psi; ISV 5 kV; gas 1 and gas 2 50 psi |
| Milk thistle *Silybum marianum* |
| Flow: 0.4 mL/min | Time: *t*an=7.2 min | LOD: B1 3.9 µg/kg,13.7 µg/kgLOQ: B1 13.5 µg/kg, B2 45.7 µg/kg |
| (S. Zhang et al. 2022) | B1, B2, B3 | 5 | 1.- Add 20 mL of ACN:H2O; 2.- shake for 30 min; 3.- ultrasonic for 30 min; 4.- take 50 μL; 5.- centrifuge at 8000 rpm for 15 min; 6.- add 950 μL of H2O and vortex; 7.- take 50 μL; 8.- add 10 μL of IS 13C-FBs; 9.- dilute with 850 μL of in MeOH:H2O 1:9 (0.2 % -FA) | CORTEX C18 10 x 4.6 mm, 5 μm at 40 °CVol. Inj NRA) H2O B) MeOH both with 0.2 % -FA10-90%B in 6 min; hold for 2 min; initial condition for 2 min | **QQQ**CaV 2.5 kV; CoG: 0.15 mL/ min, DGT 500 °C; DGF: 800 L/h; |
| Broiler Chicken Feed and Excreta |
| Flow 0.4 mL/min | Time: tan= 8 min tTot= 10 min | LOD: 50 μg/Kg all Fbs LOQ 160 μg/Kg all Fbs |
| (Weiying et al. 2022) | B1, B2 | 1 | 1.- Add IS (13C34-FB1117 (13C34-FB1), 13C34-fumonisin B2 (13C34-FB2) mixed internal standard (25 μg/mL); 2.- add 5 mL of ACN:H2O (2% FA); 3.- vortex for 10 min; 3.- Centrifuge at 3900 rpm for 3 min; 4.- evaporate to dryness at 40 °C under N2; 5.- redissolved in 5 mL of H2O; 6.- Add 6 mg of DSPME MIL-101 (Cr); 7.-ultrasonic for 10 min; 8.- centrifuge at 1200 rpm for 5 min; 9.- filter | Shimadzu C18 100 × 2.1mm, 1.8 μ m at 40 °CVol. Inj. 3 μL of sampleA) H2O (1% FA), B) ACN5% B for 1 min; 5 -90 %B in 3.5 min; hold for 2.5 min; initial condition in 0.1 min; hold for 1.9 min | **Qtrap**CaV: 5.5 kV; CoG: 35 psi; CUR: 35 psi; GS2: 45 psi |
| Milk |
| Flow 0.4 mL/min | Time: tan= 8 min tTot= 10 min | LOD: 1.5 μg/Kg all Fbs LOQ 5 μg/Kg all Fbs |
| (Flores-Flores and González-Peñas 2018) | B1 B2, B3 | 1 mL | LLE1.- Add 4 mL 2 % FA in ACN, 2.-shake 15 min; 3.-centrifuge 5000 rpm, 10 min, 4.-take 4 mL supernatant, 5.-add 60 mg NaOAc, 6.-shake 15 min, 7.- centrifuge 5000 rpm, 5 min, 8.-take 3.5 mL of ACN phase, dry at 65°C, 9.-reconstitute in 200 μL of mobile phase, 10.-filter | Ascentis Express C18, 150 x 2.1 mm, 2.7 μm, 45°CInj vol 20 µL, A) H2O, B) MeOH/H2O 95:5, both with 0.1% FA and 5 mM AmFo5-28% B in 5 min, 28-45 in 5.5 min, 45-60% B in 0.5 min, 60-90% B in 5 min, keep for 1 min, initial conditions for 13 min | **QQQ**CaV 4 kV; DGT (high-purity N2) 350°C; DGF 9 L/min; 275.8 Pa, dry gas 40 psi |
| Milk |
| Flow: 0.4 mL/min | Time: 16 min | LOD/LOQ: FB1 10 µg/L, FB2 2.5 µg/L, FB3 0.625 µg/L |
| (Song et al. 2013) | B1 and other toxins | 5 mL | 1.- Add 10 mL MgSO4 (2 M) with EtOAc/FA 99:1, shake 15 min; 2.-centrifuge to 4000 g, 15 min; 3.-take aqueous phase, add 5 mL ACN/FA 99:1; 4.-repeat extraction; 5.-dry (N2, 60°C); 6.-reconstitute with 500 µL 1:1 A:B; 7.-filter; 8.-centrifuge to 10000 g, 5 min | Symmetry C18, 150 x 2.1 mm, 5 μm at RTInj vol 20 µLA) H2O, B) MeOH, both with 0.3% FA, 5 mM AmFo5% B for 1 min, 5-25% B in 4 min, 25-60%B in 2 min, 60-80% B in 8 min, 80-100 B in 1 min, keep 6 min, 100-5 % B in 3 min | **QQQ**CaV 3.2 kV; DGF 800 L/h; CGF 20 L/h; DGT 350 °C; ST 120 °C |
| Pig, human urine |
| Flow: 0.25 mL/min | Time: *t*an=22 min, *t*Tot= 25 min | LOD: 0.05 ng/mL, LOQ: 0.17 ng/mL |
| (K. Zhang et al. 2013) | B1, B2, B3 and other toxins | 0.5 | 1.- Add 25 µL IS (13C34 FB1, 13C34 FB2, 13C34 FB3 500 ng/mL); 2.- Vortex 30 s; 3.- Add 5 mL ACN/H2O 1:1; 4.- Shake 10 min at 30-35 pulsations/min; 5.- Take an aliquot of 2 mL; 6.- Filter 2 mL; 7.-Centrifuge to 4500 rpm, 30 min | Phenomenex Kinetex XB-C18, 100 x 2.1 mm, 2.6 μm, 40°CInj vol 5 µLA) H2O, B) MeOH, both with 0.1% FA, 10 mM AmFo5-40% B lineal in 2 min, 40-100% exponential B in 7 min, keep 2.5 min, 100-5% B in 0.5 min, initial conditions for 3 min | **QQQ-IT**CaV 5.5 kV; CUR 30 psi; ST 450 °C; gas 1 and gas 2 60 psi |
| Milk based infant foods |
| Flow: 0.3 mL/min | Time: *t*an=11.5 min, *t*Tot= 15 min | LOQ B1: 2 µg/kg all fbs |
| (Abia et al. 2013) | B1, B2 and other toxins | 1 mL | 1.- Centrifuge to 5600 g, 3 min; 2.-take 100 µL 3.-add 900 µL H2O/ACN 9:1 | Gemini 150 x 4.6 mm, 5 µmInj vol 5 µL, A) H2O, B) ACN, both with 0.1% AcOH5 % B for 2 min, 5-30 % B in 8 min, 30-96 % B in 4 min, keep 1 min, initial conditions for 2.25 min | **QTrap**ST 650 °C, CUR 30 psi, SG 80 psi, DG 80 psi |
| Urine |
| Flow: 0.6 mL/min | Time: *t*an=15 min, *t*Tot= 17.25 min | LOD: B1 and B2 0.5 µg/L, LOQ: B1 and B2 1.7 µg/L |
| (Nualkaw et al. 2020) | B1, B2 and other toxins | 1 | QuEChERS1.-Add 10 mL H2O 1% FA, 2.-soak 30min; 4.-add 10 mL ACN: 5.-shake to 240 rpm, 30 min; 6.-add 1 g NaCl + 4 g MgSO4; 7.-shake 30 s: 8.-centrifuge to 10000 rpm, 5 min; 9.-take 2 mL; 10.-add 0.1 g silica C18 + 0.3 g MgSO4; 11.-mix; 12.-centrifugate 1 min; 13.-dry at 40 °C, 14.-reconstitute in 960 µL MeOH 20% + 40 µL (250 ng/mL 13C-34 FB1+50 ng/mL 13C-34 FB2); 15.-filter | Accucore C18, 100 x 2.1 mm, 2.6 µm, 25 °CInj vol 3 µLA) deionized H2O, 0.1% FA, 5mM AmF; B) MeOH0-20% B in 4 min, 20-40% B in 5.5 min; 40-100% B in 10.5 min, keep 2.5 min; initial conditions for 3 min | **Qtrap**Needle voltage 4.5 kV; CUR 30 psi; nebulizer (Gas1), turbo gas (Gas2) 55 psi; turbo gas temperature 500 °C |
| Swine, Poultry, Dairy Feeds |
| Flow: 0.4 mL/min | Time: *t*an=22.5 min, *t*Tot= 25.5 min | LOD: B1 15 µg/kg, B2 4.5 µg/kg; LOQ: B1 30 µg/kg, B2 9 ng/kg |
| (Osteresch et al. 2017) | B1 and other toxins | 100 µL | LLE1.-Spott 4 times on filter paper; 2.-dry overnight at RT, 3.-Extract with 1 mL H2O/acetone/ACN 30:35:35 in 2 mL safe-lock tubes; 4.-Sonicate 30 min; 5.-Take 800 μL; 7.-Dry at 50°C under reduced pressure; 8.-Reconstitute with H2O/ACN/AcOH 95:5:0.1; 9.-Centrifuge to 22000 g, 10 min | Gravity SB C18, 100 x 2.0 mm, 3 µm at 45°CInj vol 30 μLA) H2O, 0.1% AcOH, B) ACN, 2% AcOH3-15% B in 3 min, 15-55% B in 1.5 min, keep for 1.5 min, 55-100% B in 2 min, keep 10 min, initial conditions 1.5 min | **QTrap**CaV 5.5 kV; ST 500 °C; DP 125 V; CUR 40 psi; GS1 45 psi; GS2 50 psi |
| Blood or serum |
| Flow: 0.75 (0-6), 0.85 (6.1-10), 0.75 (10.1-11.5) mL/min | Time: *t*an=10 min, *t*Tot= 11.5 min | LOD: 0.521 ng/L LOQ: 2.5 ng/mL |
| (ACN) Acetonitrile, (AcOH) Acetic acid, (AE) Appearance energy, (AmAc): ammonium acetate, (AmFo) Ammonium formate, (CaV) Capillary voltage, (CaT) Capillary temperature, (CGF) Cone gas flow, (CoG) Collision gas, (CUR) Curtain gas, (DG) Drying gas, (DGF) Desolvation gas flow, (DGT) Desolvation gas temperature, (EV) Extractor voltage, (FA) Formic acid, (Frag Vol) Fragmentor Voltage, (GF) Gas flow, (GT) Gas Temp, (LIT) linear ion tramp, (MeOH) Methanol, (MSPD) Matrix Solid Phase Dispertion, (NG) Nebulizer gas, (NR) Not reported, (PLE) Pressurize Liquid Extraction, (RT) Room temperature, (ST) Source temperature, (SV) Source voltage, (*tan*) analysis time, *(tTot*) total time including column conditioning. |