

QuEChERS-超高效液相色谱-串联质谱法在食品 真菌毒素检测中应用的研究进展

马帅, 王蒙, 韩平, 王纪华, 冯晓元*

(北京农业质量标准与检测技术研究中心, 农业部农产品质量安全风险评估实验室(北京),
农产品产地环境监测北京市重点实验室, 北京 100097)

摘要: 真菌毒素是一类由产毒丝状真菌产生的有毒次生代谢产物, 能诱发人畜各种生理损害, 是世界各地食品和农产品的主要污染物之一。建立高效的真菌毒素分析方法将有效降低真菌毒素的暴露风险, 复杂基质中多种真菌毒素的联合提取净化和检测是建立这类分析技术的前提保证。QuEChERS(quick, easy, cheap, effective, rugged and safe)方法具有简单、快速、回收率高、试剂用量少、安全等优点, 适用于大批量样品的检测。超高效液相色谱-串联质谱法(ultra performance liquid chromatography-tandem mass spectrometry, UPLC-MS/MS)可同时用于定性和定量分析, 具有检出限低、灵敏度高优点。本文根据 QuEChERS 前处理技术的特点, 结合样品真菌毒素的理化性质和污染特点, 对 QuEChERS-UPLC-MS/MS 技术在真菌毒素分析中的应用做了简要综述。

关键词: QuEChERS; 超高效液相色谱-串联质谱法; 真菌毒素; 食品

Research advances on application of QuEChERS-ultra performance liquid chromatography-tandem mass spectrometry in mycotoxin analysis in foods

MA Shuai, WANG Meng, HAN Ping, WANG Ji-Hua, FENG Xiao-Yuan*

(Beijing Research Center for Agricultural Standards and Testing, Risk Assessment Lab for Agro-products (Beijing),
Ministry of Agriculture, Beijing Municipal Key Laboratory of Agriculture Environment Monitoring,
Beijing 100097, China)

ABSTRACT: Mycotoxins are the secondary metabolites produced by certain filamentous fungi, which can induce physiological damage on animals and humans, and they are regarded as one of the main pollution source in foods and agriculture products all over the world. Simultaneous extraction of mycotoxins from complex sample matrices is a prerequisite to establish a high-throughput analytical method, which in turn will effectively reduce the mycotoxins exposure risk. QuEChERS is a sample preparation technique with the advantages of being simple and rapid, high recovery rate, low solvent consumption and safety, which is suitable for the detection of large beach samples. Ultra performance liquid chromatography-tandem mass spectrometry (UPLC-MS/MS) can be used for both qualitative and quantitative analysis with the advantages of low detection limit and high sensitivity. This paper summarized the application of QuEChERS-UPLC-MS/MS techniques in the analysis of mycotoxins, which combined with the

基金项目: 公益性行业(农业)科研专项(201303075)、农业部农产品质量安全风险评估实验室(北京)开放课题项目(ZBZXKFKT 201503)

Fund: Supported by the Special Fund for Agro-scientific Research in the Public Interest (201303075) and the Open Project of the Risk Assessment Lab for Agro-products (Beijing) Ministry of Agriculture (ZBZXKFKT201503)

*通讯作者: 冯晓元, 研究员, 博士, 主要研究方向为果品质量与安全。E-mail: fengxiaoyuan2014@126.com

*Corresponding author: FENG Xiao-Yuan, Researcher, Ph.D, Beijing Research Center for Agricultural Standards and Testing, Risk Assessment Lab for Agro-products (Beijing), Ministry of Agriculture, Beijing Municipal Key Laboratory of Agriculture Environment Monitoring, Beijing 100097, China., E-mail: fengxiaoyuan2014@126.com

characteristics of QuEChERS method technology and the physicochemical properties and pollution characteristics of mycotoxins.

KEY WORDS: QuEChERS; ultra performance liquid chromatography-tandem mass spectrometry; mycotoxins; food

1 引言

真菌毒素(mycotoxins)是一类由产毒丝状真菌产生的有毒次生代谢产物,能诱发人畜各种生理损害,是世界各地农产品和食品的主要污染物之一。目前,已发现了300~400种真菌毒素,主要来源于曲霉属(*Aspergillus*)、镰刀菌属(*Fusarium*)和青霉属(*Penicillium*)的200多种产毒丝状真菌^[1,2]。适宜条件下,产毒丝状真菌在生长中分泌的真菌毒素会侵染农作物,造成真菌毒素残留。这些真菌毒素不仅具有致癌、致畸和致突变等作用,还具有肝细胞毒性、中毒性肾损害、生殖紊乱以及免疫抑制等作用,对人畜健康造成极大威胁^[3]。根据联合国粮食农业组织统计,全世界每年约有25%的谷物被真菌毒素污染,2%的农产品因污染严重而失去营养和经济价值,造成数百亿美元的经济损失^[4]。因此,要防止食品受污染,需加强对真菌毒素的监测和检测力度。

国内外学者运用不同的检测技术对食品中真菌毒素的检测做了大量的研究,推动了真菌毒素检测领域的发展。目前,真菌毒素分析的前处理方法主要包括固相萃取(solid-phase extraction)^[5]、加速溶剂萃取(accelerated solvent extraction)^[6]、基质固相分散萃取(matrix solid-phase dispersion)^[7]和液液微萃取(liquid-liquid microextraction)^[8]等。近年来,QuEChERS前处理技术广泛应用于农药残留的分析检测中,其高效、简便和绿色的技术理念正逐渐在类固醇激素等环境污染物^[9]、食品添加剂^[10]和生物毒素^[11]等分析检测领域得到应用。真菌毒素的检测方法主要包括薄层色谱法^[12]、酶联免疫法^[13-15]、气相色谱-串联质谱联用法(gas chromatography-tandem mass spectrometry, GC-MS/MS)^[16,17]、液相色谱-串联质谱联用法(liquid chromatography-tandem mass spectrometry, LC-MS/MS)^[18-20]。薄层色谱法简捷、便利,但重现性差、精密率低;酶联免疫法特异性强、前处理简单,但假阳性率高,不能作为确证方法;气质联用法需要衍生化前处理、操作费时费力。液相色谱-串联质谱联用技术具有更高的选择性和灵敏度,成为同时检测多种真菌毒素的主要手段。本文根据QuEChERS技术的特点,结合样品基质特征和真菌毒素的理化性质对QuEChERS-超高效液相色谱-串联质谱法(QuEChERS-ultra performance liquid chromatography-tandem mass spectrometry, QuEChERS-UPLC-MS/MS)技术在真菌毒素分析中的应用进行了简要综述。

2 食品中真菌毒素的污染特点及危害

2.1 常见真菌毒素的污染特点

自然界中真菌毒素多以固体形式存在,大多数真菌毒素化学性质稳定、不易分解,易溶于有机试剂且熔点高。常见的真菌毒素种类有黄曲霉毒素(afatoxins, AFs)、赭曲霉毒素A(ochratoxin, OTA)、玉米赤霉烯酮(zearalenone, ZEN)、伏马菌素(fumonisin, FBs)、脱氧雪腐镰刀菌烯醇(deoxynivalenol, DON)和展青霉素(patulin, PAT)等。真菌毒素多具有极强的生物毒性,其中AFTB1是目前发现毒性最强的毒素^[21]。AFs主要侵染谷物和坚果等,其中以花生、玉米污染最为严重,同时毒素可通过食物链间接污染肉类、乳及乳制品^[22]。OTA在自然界分布最广泛,在农作物中污染水平最高,毒性最强,对人类和动植物影响也最大^[23]。ZEN多存在于大麦和燕麦等谷物,FBs主要污染玉米及其制品^[24]。DON主要污染小麦、大麦、玉米等谷类作物,也会污染粮食制品。PAT主要存在于苹果、山楂等水果以及浆果、蔬菜、面包和肉类制品中^[25]。

2.2 常见真菌毒素的危害及致病机制

真菌毒素可直接污染植物体及其产品,也可通过被污染的饲料进入动物组织、牛奶、鸡蛋等食品中,从而通过食物链的逐级传递对人类和动物健康产生潜在威胁。食品中几种常见真菌毒素的致病机制大致如下:(1)AFs:可抑制DNA、RNA的合成,破坏凝血机制及某些酶类,具有强致癌性和强免疫抑制性^[26];(2)OTA:可抑制ATP酶、琥珀酸脱氢酶以及细胞色素C氧化酶,从而对羟化过程产生影响,除具有特异性肾毒性作用以外,OTA还对免疫系统有毒性,并有三致作用^[27,28];(3)ZEN:具有显著的雌性激素作用,对动物和人的生长发育及生殖系统有很强影响和破坏作用^[29];(4)FBs:伏马菌素是一类细胞毒素,也是致癌物质,其中FB1毒性最强,是一种较强的肝脏、肾脏毒^[30];(5)DON:主要是抑制蛋白质、RNA、DNA等大分子物质的合成,破坏细胞膜和酶类的功能,对造血系统和免疫系统有较强的毒作用^[31];(6)PAT:展青霉素是一种有毒内酯,具有强烈的抗菌活性,对动物的细胞和组织具有很强的毒性,并具有潜在的致癌性和诱变性。

3 QuEChERS-UPLC-MS/MS 方法的优势及应用

3.1 QuEChERS 技术在真菌毒素分析中的优势

QuEChERS技术是由化学家Lehotay和Anastassiadas

于2003年提出^[32],目前,根据真菌毒素和样品基质的理化特性,开发多真菌毒素的QuEChERS技术正逐渐成为真菌毒素研究领域的热点。QuEChERS技术分为提取、盐析和净化3个步骤。提取是QuEChERS技术重要的组成部分,提取液的提取效率决定着最终分析结果的准确性。综合QuEChERS的提取效果和提取试剂的理化性质,乙腈的提取率高,适用范围广。对于极性范围敏感的目标毒素提取时,需在提取液中添加辅助试剂(乙酸、甲酸和甲醇等)以增强联合提取的效果^[33]。样品经过提取后,提取液中仍然存在大量的共萃物,通过盐析步骤,可以初步去除部分水溶性杂质。在QuEChERS技术中,3种典型盐析剂为:4 g MgSO₄+1 g NaCl(萃取体系中性)、6 g MgSO₄+1.5 g NaOAc(萃取体系pH 4.8)和4 g MgSO₄+1 g NaCl+1 g Na₃Cit·2H₂O+0.5 g Na₂Cit·1.5H₂O(萃取体系pH 5~5.5)。根据目标真菌毒素的性质,选择不同的缓冲盐体系以获得理想的盐析效果。盐析分层后,少量的色素、糖以及蛋白质等组分会不可避免的与真菌毒素共萃取出来,这些组分的残留会干扰样品分析结果。净化过程中通过选择不同净化剂除去干扰组分:PSA(primary secondary amine)结构上有两个氨基,可与分子结构上含有羟基的极性物质发生氢键相互作用,吸附提取液中的碳水化合物、色素和有机酸;C₁₈通过非极性相互作用吸附非极性物质,吸附提取液中的淀粉、脂类和固醇类物质;GCB(graphite carbon black)具有阴离子交换作用,通过疏水相互作用和氢键相互作用吸附杂质,可吸附类胡萝卜素和叶绿素。

传统的真菌毒素提取净化方法,通常需要经过多次溶剂提取净化,存在较高的真菌毒素暴露风险。较之传统的真菌毒素提取净化方法,QuEChERS技术的整个分析体系具有以下显著优势:(1)仪器设备简单。实验处理仅需要离心管、振荡器、离心机即可完成。(2)试剂使用量少,对环境污染小。(3)前处理步骤少,操作技术难度低,适宜推广。(4)处理时间短。全部提取净化步骤可在30 min内完成,适合批量分析。(5)实现多组分提取净化。通过设计提取液配方,选择适宜盐析剂和净化剂,可实现多真菌毒素的联

合提取,提高分析效率。(6)操作人员暴露风险低。

3.2 UPLC-MS/MS分析在真菌毒素分析中的优势

针对食品中真菌毒素的痕量分析,MS是使用最广泛且最高效的检测器,UPLC-MS/MS技术提高分析的灵敏度和可靠性。电喷雾电离(electrospray ionization, ESI)是一种软电离方式,即便是稳定性差的化合物,也不会电离过程中发生分解。其工作原理为:样品先带电再喷雾,带电液滴在去溶剂化过程中形成样品离子,从而被检测,大部分极性较大的真菌毒素和代谢物在该电离模式下都能实现较好电离。对于极性小的某些真菌毒素,用ESI不能产生足够强的离子,可以采用大气压化学电离(atmospheric pressure chemical ionization, APCI)方式增加离子产率。鉴于UPLC-MS/MS具有多种离子源(ESI, APCI)、多种质量分析器(四级杆分析器、飞行时间质量分析器和离子阱)和各种数据分析软件可供选择,其在多组分真菌毒素的检测中具有不可比拟的优势,同时可实现各组分快速、高效的定性和定量分析,由此食品中真菌毒素和代谢物的多组分检测技术得以快速发展。

3.3 QuEChERS-UPLC-MS/MS方法的应用

目前,现已报道的QuEChERS技术已应用于毒素提取净化,主要包括黄曲霉毒素(AFB₁、AFB₂、AFG₁、AFG₂)、玉米赤霉烯酮(ZON)、脱氧雪腐镰刀菌烯醇(DON)、赭曲霉毒素A(ochratoxin A, OTA)、伏马菌素(fumonisin, FUN)、展青霉毒素(PAT)、链格孢酚和橘青霉素橘霉素(citrinin, CIT)等^[34-40]。陈慧菲等^[34]建立了谷物中8种真菌毒素(AFB₁、AFB₂、AFG₁、AFG₂、ZON、DON、3-乙酰基DON和15-乙酰基DON)的测定方法,样品采用改良的QuEChERS-UPLC-MS/MS进行测定,8种毒素检出限为0.3~1.0 μg/kg,加标回收率为76.5%~113.4%。Frenich等^[39]采用QuEChERS-UPLC-MS/MS法测定蛋品中的10种毒素(白僵菌素、环肽A、环肽A₁、环肽B₁、桔霉素、AFB₂、AFG₁、AFG₂、G₂和OTA),10种毒素的加标回收率为70%~110%,检出限为1.0~5.0 μg/kg,具体见表1。

表1 QuEChERS-UPLC-MS/MS方法在真菌毒素检测中的应用
Table 1 Application of QuEChERS-UPLC-MS/MS in the analysis of mycotoxins

样品	毒素种类	回收率/%	检出限/(μg/kg)	发表年份
玉米和小麦粉	AFB ₁ 、AFB ₂ 、AFG ₁ 、AFG ₂ 、DON等8种毒素	76.5~113.4	0.3~1.0	2016 ^[34]
豆制品和奶粉	AFM ₁ 、AFG ₂ 、OTA、ZON等14种毒素	65.3~130.1	0.5~1.0	2014 ^[35]
柑橘	赭曲霉毒素、链格孢酚和橘青霉素	78.0~103.3	0.2~0.7	2014 ^[36]
婴儿谷辅食	ZEN、DON、FB ₁ 、OTA等9种真菌毒素	77.6~109.6	0.1~15.8	2014 ^[37]
面粉	PAT、AFs、CIT、OTA等17种真菌毒素	78.6~103.4	0.5~5.0	2014 ^[38]
蛋品	AFT、CIT、OTA等10种毒素	65.0~114.0	1.0~5.0	2011 ^[39]
玉米大麦	FUN、ZEN、NIV、DON等11种镰刀菌毒素	94.0~108.0	5.0~50.0	2010 ^[40]

4 结语与展望

真菌毒素和代谢物污染问题, 是食品和农产品重大的安全问题。如何监控食品中真菌毒素和代谢物的污染情况, 尤其是监控其中具有致癌、致畸、致细胞突变的高毒性化合物, 是各国食品安全部门的一个重点工作。QuEChERS与UPLC-MS/MS技术的结合, 为痕量多种真菌毒素和代谢物的同时检测提供了一个极具发展潜力的方向。如何建立灵敏度高、提取范围更广、提取效率更高、操作更简单、更环保的 QuEChERS-UPLC-MS/MS 分析方法是后续相关研究的重点。

参考文献

- [1] Berthiller F, Sulyok M, Krska R, *et al.* Chromatographic methods for the simultaneous determination of mycotoxins and their conjugates in cereals [J]. *Int J Food Microbiol*, 2007, 119(1/2): 33–37.
- [2] Zollner P, Mayer-helm B. Trace mycotoxin analysis in complex biological and food matrices by liquid chromatography atmospheric pressure ionisation mass spectrometry [J]. *J Chromatogr A*, 2006, 1136(2): 123–169.
- [3] 陈丽星. 真菌毒素研究进展 [J]. *河北工业科技*, 2006, 23(2): 124–126. Chen LX. Mycotoxins and their research progress [J]. *Hebei Ind Sci Technol*, 2006, 23 (2): 124–126.
- [4] Schatzmayr G, Zehner F, Taubel M, *et al.* Microbiologicals for deactivating mycotoxins [J]. *Mol Nutr Food Res*, 2006, 50(6): 543–551.
- [5] Ediage EN, Mavungu JD, Monbaliu S, *et al.* A validated multianalyte LC-MS/MS method for quantification of 25 mycotoxins in cassava flour, peanut cake and maize samples [J]. *J Agric Food Chem*, 2011, 59: 5173–5180.
- [6] Mccooney M, Kolakowski B, Boison J, *et al.* Evaluation of high-field asymmetric waveform ion mobility spectrometry mass spectrometry for the analysis of the mycotoxin zearalenone [J]. *Anal Chim Acta*, 2008, 627: 112–116.
- [7] Rubert J, Soler C, Mānes J. Evaluation of matrix solid-phase dispersion (MSPD) extraction for multi-mycotoxin determination in different flours using LC-MS/MS [J]. *Talanta*, 2011, 85(1): 206–215.
- [8] Campone L, Piccinelli AL, Celano R, *et al.* Application of dispersive liquid-liquid micro extraction for the determination of aflatoxins B₁, B₂, G₁ and G₂ in cereal products [J]. *J Chromatogr A*, 2011, 1218: 7648–7654.
- [9] 马帅, 冯晓元, 韩平, 等. 分散固相萃取-超高效液相色谱串联质谱法测定土壤中雌酮、雌二醇和乙炔基雌二醇 [J]. *食品安全质量检测学报*, 2016, 7(2): 505–510. Ma S, Feng XY, Han P, *et al.* Determination of estrone, 17 β -estradiol and 17 α -ethinylestradiol in soil by ultra-high performance liquid chromatography-tandem mass spectrometry with dispersive solid phase extraction [J]. *J Food Saf Qual*, 2016, 7(2): 505–510.
- [10] Mastovska K, Lehotay SJ. Rapid sample preparation method for LC-MS/MS or GC-MS analysis of acrylamide in various food matrices [J]. *J Agric Food Chem*, 2006, 54(19): 7001–7008.
- [11] Franz B, Buttinger G. Rapid simultaneous determination of major type A- and B-trichothecenes as well as zearalenone in maize by HPLC-MS [J]. *J Chromatogr A*, 2005, 1: 209–216.
- [12] Rizzo I, Vedoya G, Maurutto S, *et al.* Assessment of toxigenic fungi on Argentinean medicinal herbs [J]. *Microbiol Res*, 2004, 159(2): 113.
- [13] Li Y, Luo X, Yang S, *et al.* High Specific Monoclonal Antibody Production and Development of an ELISA Method for Monitoring T-2 Toxin in Rice [J]. *J Agric Food Chem*, 2014, 62(7): 1492–1497.
- [14] Li Y, Shi W, Shen J, *et al.* Development of a rapid competitive indirect ELISA procedure for the determination of deoxynivalenol in cereals [J]. *Food Agric Immunol*, 2011, 23(1): 41–49.
- [15] Ling S, Pang J, Yu J, *et al.* Preparation and identification of monoclonal antibody against fumonisin B₁ and development of detection by Ic-ELISA [J]. *Toxicol*, 2014, 80: 64–72.
- [16] Ferreira I, Fernandes JO, Cunha SC. Optimization and validation of a method based in a QuEChERS procedure and gas chromatography–mass spectrometry for the determination of multi-mycotoxins in popcorn [J]. *Food Control*, 2012, 27(1): 188–193.
- [17] Taba ta S, Iida K, Suzuki J, *et al.* A quantification and confirmation method of patulin in apple juice by GC/MS [J]. *J Food Hyg Soc Japan*, 2004, 45(5): 245–249.
- [18] Liliana S, Mónica FF, Guillermina F, *et al.* Analysis of fumonisins in corn-based food by liquid chromatography with fluorescence and mass spectrometry detectors [J]. *Food Chem*, 2009, 112(4): 1031–1037.
- [19] Sulyok M, Berthiller F, Krska R, *et al.* Development and validation of a liquid chromatography/tandem mass spectrometric method for the determination of 39 mycotoxins in wheat and maize [J]. *Rapid Commun Mass Spectrom*, 2006, 20(18): 2649–2659.
- [20] Rasmussen RR, Storm IM, Rasmussen PH, *et al.* Multi-mycotoxin analysis of maize silage by LC-MS/MS [J]. *Anal Bioanal Chem*, 2010, 397(2): 765–776.
- [21] 王红星, 陶正国. 饲料中的真菌毒素的危害性及其防治措施 [J]. *兽药与饲料添加剂*, 2000, 5(3): 19–20. Wang HX, Tao ZG. The harm and prevention measures of mycotoxins in feed [J]. *Vet Drug Feed Addit*, 2000, 5(3): 19–20.
- [22] Nonaka Y, Saito K, Hanioka N, *et al.* Determination of aflatoxins in food samples by automated online in tube solid phase micro extraction coupled with liquid chromatography–mass spectrometry [J]. *J Chromatogr A*, 2009, 1216(20): 4416–4422.
- [23] 刘小杰, 何国庆, 陈启和, 等. 赭曲毒素 A 的研究进展 [J]. *粮油加工与食品机械*, 2002, (2): 38–39. Liu XJ, He GQ, Chen QH, *et al.* Research progress of ochratoxin A [J]. *Cereals Oils Process*, 2002, (2): 38–39.
- [24] Rheeder JP, Marasas WF, Vismer HF. Production of fumonisin analogs by *Fusarium* Species [J]. *Appl Environ Microbiol*, 2002, 68(5): 2101–2105.
- [25] Mahfoud R, Maresca M, Garmy N, *et al.* The mycotoxin patulin alters the barrier function of the intestinal epithelium: mechanism of action of the toxin and protective effects of glutathione [J]. *Toxicol Appl Pharm*, 2002, 181(3): 209–218.
- [26] Santi DU, Ha GS, Chan HL. Comparative study on the aflatoxin B₁ degradation ability of rumen fluid from Holstein steers and Korean native goats [J]. *Vet Sci*, 2009, 10(1): 29–34.
- [27] 高翔, 李梅. 赭曲霉毒素 A 的毒性研究进展 [J]. *国外医学卫生学分册*, 2005, 32(1): 51–55.

- Gao X, Gao M. Research progress of ochratoxin A [J]. Foreign Med Sci (Section Hyg), 2005, 32(1): 51-55.
- [28] Grollman PA, Jelakovic B. Role of environmental toxins in endemic (Balkan) nephropathy [J]. J Am Soc Nephrol, 2007, 18: 2817-2823.
- [29] VinodK, BasuaM S, RajendranTP. Mycotoxin research and mycoflora in some commercially important agricultural commodities [J]. Crop Protect, 2008, 27: 891-905.
- [30] 赵丹霞, 丁晓雯. 伏马菌素对食品的污染及毒性[J]. 现代食品科技, 2005, 21(2): 206-209.
Zhao DX, Ding XW. The toxicity of fumonisins and its contamination in food [J]. Mod Food Sci Technol, 2005, 21(2): 206-209.
- [31] 霍星华, 赵宝玉, 万学攀, 等. 脱氧雪腐镰刀菌烯醇的毒性研究进展[J]. 毒理学杂志, 2008, 22(2): 151-154.
Huo XH, Zhao BY, Wan XP, *et al.* Research progress on the toxicity of the DON [J]. J Toxicol, 2008, 22(2): 151-154.
- [32] Anastassiades M, Lehotay SJ, Stajnbaher D, *et al.* Fast and easy multiresidue method employing acetonitrile extraction/partitioning and "dispersive solid-phase extraction" for the determination of pesticide residues in produce [J]. J AOAC Int, 2003, 86(2): 412-431.
- [33] Sospedra I, Blesa J, Soriano JM, *et al.* Use of the modified quick easy cheap effective rugged and safe sample preparation approach for the simultaneous analysis of type A- and B-trichothecenes in wheat flour [J]. J Chromatogr A, 2010, 1217(9): 1437-1440.
- [34] 陈慧菲, 朱天仪, 陈凤香, 等. QuEChERS-超高效液相色谱串联质谱法测定谷物中的8种真菌毒素[J]. 粮食与油脂, 2016, 29(5): 67-70.
Chen HF, Zhu TY, Chen FX, *et al.* Simultaneous determination of 8 mycotoxins in cereal with QuEChERS- based extraction and ultra performance liquid chromatography-tandem mass spectrometry [J]. Cereals Oils, 2016, 29(5): 67-70.
- [35] 史娜, 侯彩云, 路勇, 等. QuEChERS-高效液相色谱-质谱法检测食品中14种真菌毒素[J]. 食品科学, 2014, 35(16): 190-196.
Shi N, Hou CY, Lu Y, *et al.* Simultaneous screening for 14 mycotoxin contaminants in foods by QuEChERS-LC-MS-MS [J]. Food Sci, 2014, 35(16): 190-196.
- [36] 史文景, 赵其阳, 焦必宁. UPLC-ESI-MS-MS 结合 QuEChERS 同时测定柑橘中的4种真菌毒素[J]. 食品科学, 2014, 35(20): 170-174.
Shi WJ, Zhao QY, Jiao BN. Simultaneous determination of four mycotoxins in citrus fruits by ultra performance liquid chromatography-electrospray ionization tandem mass spectrometry combined with modified QuEChERS [J]. Food Sci, 2014, 35(20): 170-174.
- [37] 胡文彦, 许磊, 杨军, 等. 基于 QuEChERS 提取的快速液相色谱-串联质谱法测定婴幼儿谷基辅助食品中的9种真菌毒素[J]. 色谱, 2014, 32(2): 133-138.
Hu WY, Xu L, Yang J, *et al.* QuEChERS-based extraction procedure and rapid resolution liquid chromatography coupled to triple quadrupole mass spectrometry for the determination of nine mycotoxins in cereal-based complementary foods for infants and young children [J]. Chin J Chromatogr, 2014, 32(2): 133-138.
- [38] 王峰, 仓以鹏, 蔡晶, 等. 高效液相色谱-三重四级杆质谱法测定面粉中17种真菌毒素[J]. 食品科技, 2014, 39(11): 331-334.
Wang F, Cang YP, Cai J, *et al.* Determination of 17 mycotoxins in flour by high performance liquid chromatography-tandem mass spectrometry [J]. Food Sci Technol, 2014, 39(11): 331-334.
- [39] Frenich AG, Romero-gonzález R, Gómez-pérez ML, *et al.* Multi-mycotoxin analysis in eggs using a QuEChERS-based extraction procedure and ultra-high-pressure liquid chromatography coupled to triple quadrupole mass spectrometry [J]. J Chromatogr A, 2011, 1218(28): 4349-4356.
- [40] Zachariasova M, Lacina O, Malachova A, *et al.* Novel approaches in analysis of *Fusarium* mycotoxins in cereals employing ultra performance liquid chromatography coupled with high resolution mass spectrometry [J]. Anal Chim Acta, 2010, 662(1): 51-61.

(责任编辑: 姚菲)

作者简介



马 帅, 硕士, 主要研究方向为农田环境与农产品质量安全。

E-mail: mashuai19890926@126.com



冯晓元, 博士, 研究员, 主要研究方向为果品质量与安全。

E-mail: fengxiaoyuan2014@126.com