岩矿测试
ROCK AND MINERAL ANALYSIS
中文核心期刊

文章摘要

林立, 王琳琳, 孙海波, 孙继红. 离子色谱-电感耦合等离子体质谱法测定乳粉的汞形态[J]. 岩矿测试, 2014, 33(3):390~396

离子色谱-电感耦合等离子体质谱法测定乳粉的汞形态

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Speciation Analysis of Mercury in Milk Powder using Ion Chromatography-Inductively Coupled Plasma Mass Spectrometry Technique

投稿时间： 2013-11-02

DOI：

中文关键词： 乳粉 汞形态 解离 超声提取 离子色谱法 电感耦合等离子体质谱法

英文关键词: milk powder mercury speciation dissociation ultrasonic extraction Ion Chromatography Inductively Coupled Plasma-Mass Spectrometry

基金项目:国家质检总局科技计划项目（2012QK013）；质检公益性行业科研专项（201210116）

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中文摘要:

对于乳粉的汞形态分析，由于基质的复杂性，有机汞非常容易与样品中蛋白质上的巯基结合，形成稳定的络合物，在前处理过程中须保证各形态提取完全且各形态之间不会发生相互转化，因此样品前处理是汞形态分析的难点；同时乳粉中汞含量极低，对方法检出限提出了更高的要求。本文通过优化样品前处理过程，建立了离子色谱-电感耦合等离子体质谱测定乳粉中三种汞形态（二价汞、甲基汞、乙基汞）的方法。实验采用多种复合酶（蛋白酶、脂肪酶、淀粉酶）对乳粉基质中的蛋白、脂肪、淀粉进行解离，采用L-半胱氨酸-盐酸-甲醇的混合溶液作为提取剂进行超声提取，样品过RP固相萃取小柱去除杂质后用C18色谱柱（5 μm，4.6 mm×150 mm）进行分离，流动相采用10 mmol/L乙酸铵-0.12% L-半胱氨酸-5%甲醇混合溶液进行淋洗，5 min内即可实现三种汞形态的基线分离。二价汞、甲基汞和乙基汞的加标回收率在79.9%~111.2%之间，检出限分别为0.5 μg/kg、0.6 μg/kg、0.9 μg/kg。实际样品分析表明，汞总量很低的乳粉，汞各形态的提取率也能达到70%以上，能够满足检测要求。本方法在样品前处理过程中采用酶解的方式解离复杂基体中的汞形态，提高了提取率至80%以上；仪器分析方面采用甲醇作为增敏剂，提高了检测灵敏度，适用于乳粉样品中痕量汞形态的检测。

英文摘要:

Sample pre-treatment difficulties arise during the analysis for mercury speciation in milk powder due to the complexity of the sample matrix. The organic mercury combines easily with sulfydryl of proteins in the matrix sample, to form a stable complex. Therefore it is very important to

ensure that all forms are extracted completely and do not transform during the pre-treatment process. The method with a lower detection limit was necessary because the concentration of mercury was too low. For this purpose, the method for determination of mercury speciation (inorganic mercury, methylmercury, ethylmercury) in milk was established by ion chromatography inductively coupled plasma mass spectrometry with optimized pre-treatment conditions. Protein, fat, and starch in milk powder were dissociated using a variety of composite enzymes (protease, lipase and amylase). The sample was ultrasonically extracted using mixed solution of L-cysteine, hydrochloric acid with methanol and was further purified by RP solid phase column. Mercury speciation was separated by Agilent Eclipse XDB-C18 column (5 μm, 4.6 mm×150 mm).The mobile phase contained 10 mmol/L ammonium acetate, 0.12% L-cysteine, and 5% (*m*:*m*) methanol solution. Three kinds of? mercury speciation were baseline separated within 5 min. The spiked recoveries of inorganic mercury, methylmercury and ethylmercury were obtained in the range of 79.9%-111.2%. The instrument detection limits were 0.5 μg/kg, 0.6 μg/kg and 0.9 μg/kg, respectively. The results obtained from actual? sample testing show that the extraction rate of total mercury can reach more than 70% in low concentration mercury milk powder, which meets the test requirements. The extraction rate can reach more than 80% because the composite enzyme was used in this method to dissociate mercury speciation from the complex matrix during sample pre-treatment processing. Methanol was applied as a sensitizer to improve detection sensitivity. A simple, rapid and reliable method was developed for the determination of mercury speciation in milk powder.

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