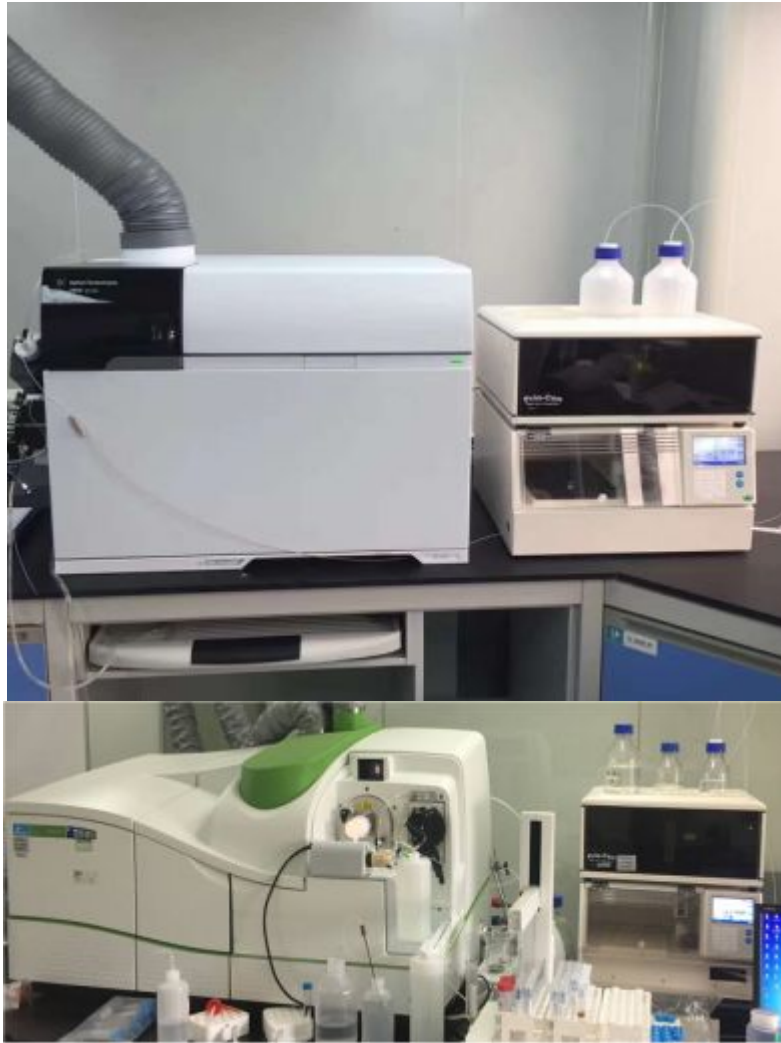


Prin-Cen 高效快速测定食品中的无机砷解决方案(LC-ICPMS)



说明：此方法适用GB5009.11-2014中三类样品的分析，也适用GB2762-2017中有无机砷限值要求的样品

内容

- 一、试剂准备
- 二、仪器条件
- 三、标准溶液的配制
- 四、样品前处理
- 五、分析说明

一、试剂准备

1、标准溶液：国家标物中心

AsB: 0.518 μmol/g

DMA: 0.706 μmol/g

As³⁺: 1.011 μmol/g

MMA: 0.335 μmol/g

As⁵⁺: 0.233 μmol/g



2、试剂：优级纯HNO₃，甲醇，正己烷，prin-cen ONLY WATER KIT

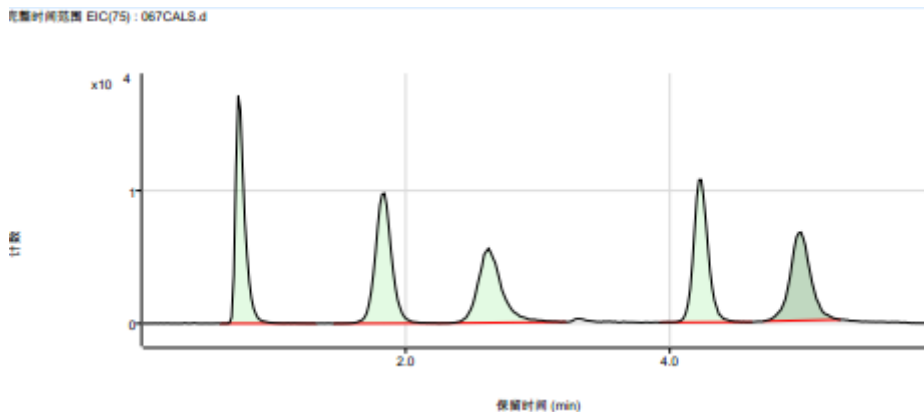
3、耗材：50ml离心管，15ml离心管，C18小柱、0.45um滤膜，5ml注射器

二、仪器条件

液相参数		ICPMS参数	
色谱柱	As Spec Guard column + As Spec Fast Column	监测离子	⁷⁵ As
流动相	ONLY WATER KIT for ICPMS 10X Mobile Phase,用超纯水稀释10倍	模式	标准模式 (No Gas)
流速	1mL/min	雾化气流量	1.0L/min
进样量	10 μL	积分时间	0.5秒
运行时间	6.0 min	采样时间	6.0 min

三、标准溶液的配制

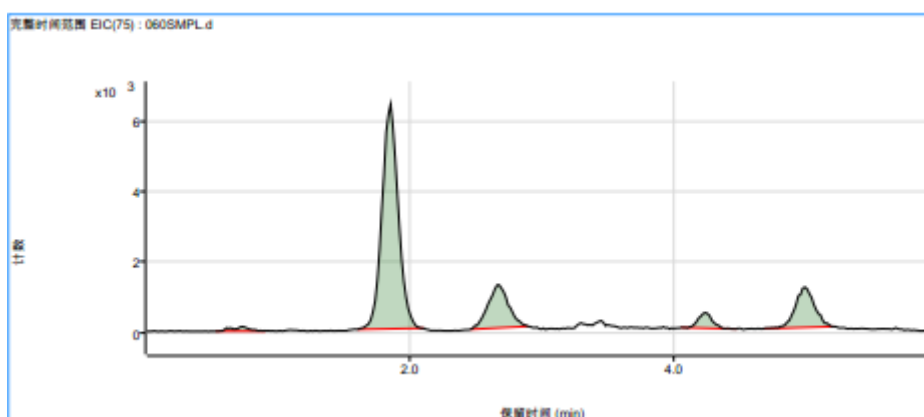
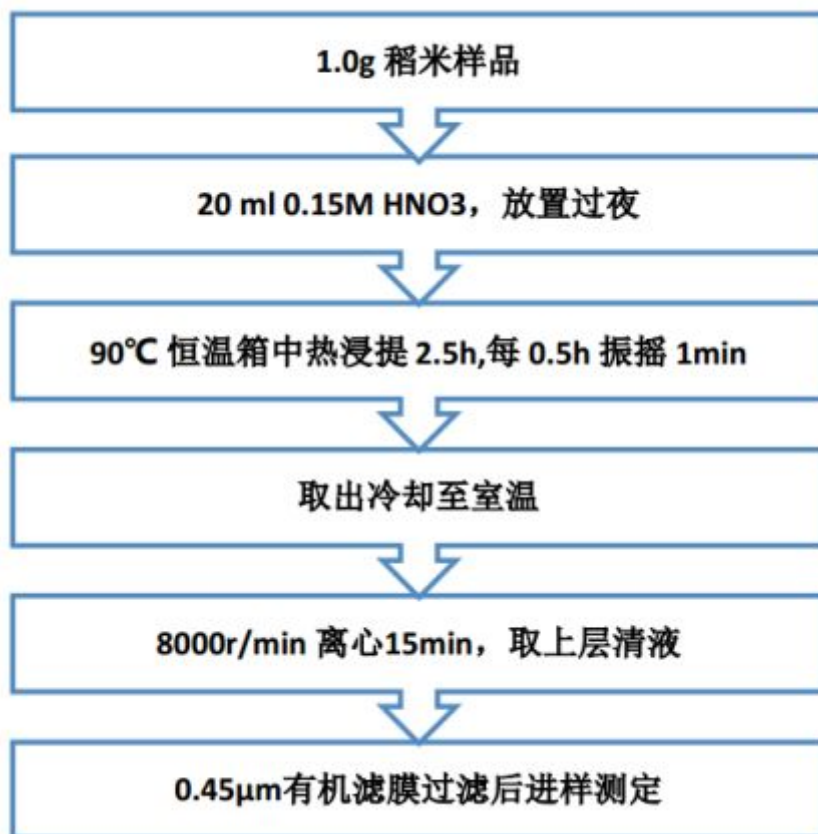
用纯水把五种砷形态配成1ppm的储备液，把1ppm储备液稀释到200ppb的中间液，将200ppb的中间液用纯水逐级稀释到0ppb、1ppb、5ppb、10ppb、20ppb、50ppb，然后直接进样。



10ppb 5种砷形态标准溶液色谱图

四、样品前处理

1、稻米样品



NIST有证标物1568b色谱图

Table 2. Certified Mass Fraction Values (Dry-Mass Basis) for Selected Arsenic Species in SRM 1568b

Arsenic Species	Mass Fraction (mg/kg, as As)	Coverage Factor, <i>k</i>
Dimethylarsinic acid (DMA) ^(a,b,c)	0.180 ± 0.012	2.0
Monomethylarsonic acid (MMA) ^(a,c)	0.0116 ± 0.0035	2.0
Inorganic arsenic (iAs) ^(a,c,d)	0.092 ± 0.010	2.0

^(a) NIST IC/ICP-MS

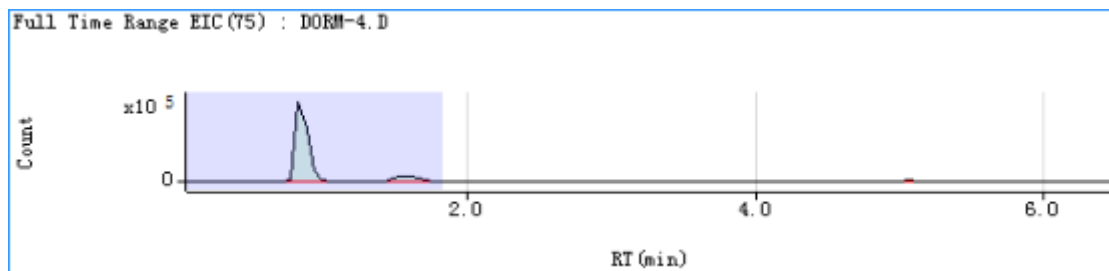
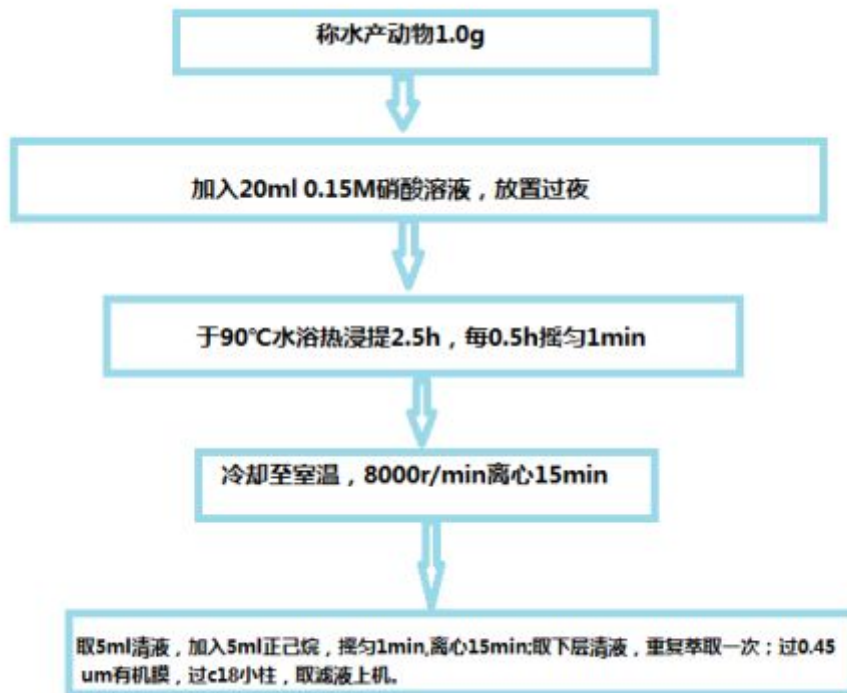
^(b) NIST LC/MS/MS

^(c) NMJ IC/ICP-MS

^(d) As (III) and As (V), reported as total inorganic arsenic (iAs)

1568b证书无机砷标称值

2、水产动物样品（含高脂肪样品）



DORM-4有证标物色谱图

DORM-4: Fish protein certified reference material for trace metals

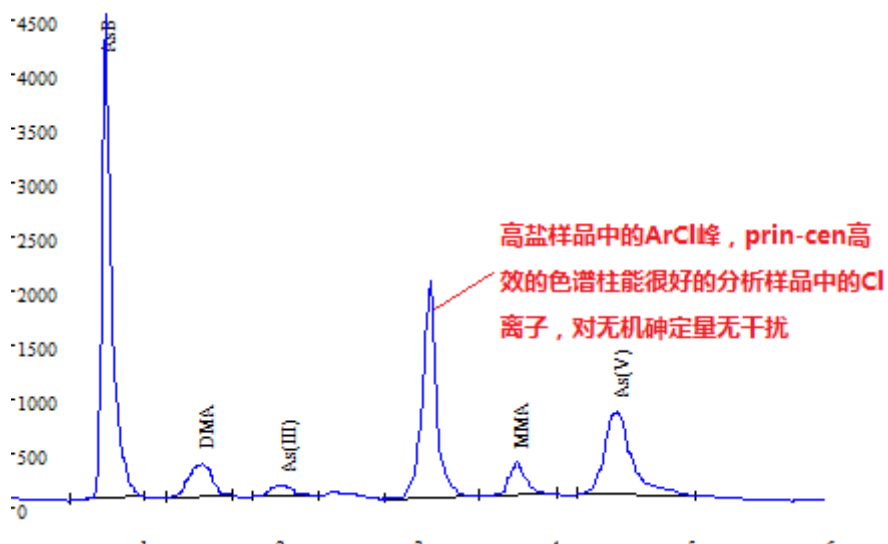
The following table shows those elements for which certified values have been established for this reference material. Certified values are based on results from data generated at NRC. The expanded uncertainty (U_{CRM}) in the certified value is equal to $U = k u_c$ where u_c is the combined standard uncertainty calculated according to the JCGM Guide and k is the coverage factor. It is intended that U_{CRM} encompasses every aspect that reasonably contributes to the uncertainty of the measurement. A coverage factor of 2 was applied for all below lists certified values for DORM-4 expressed on a dry mass basis.

Product documentati

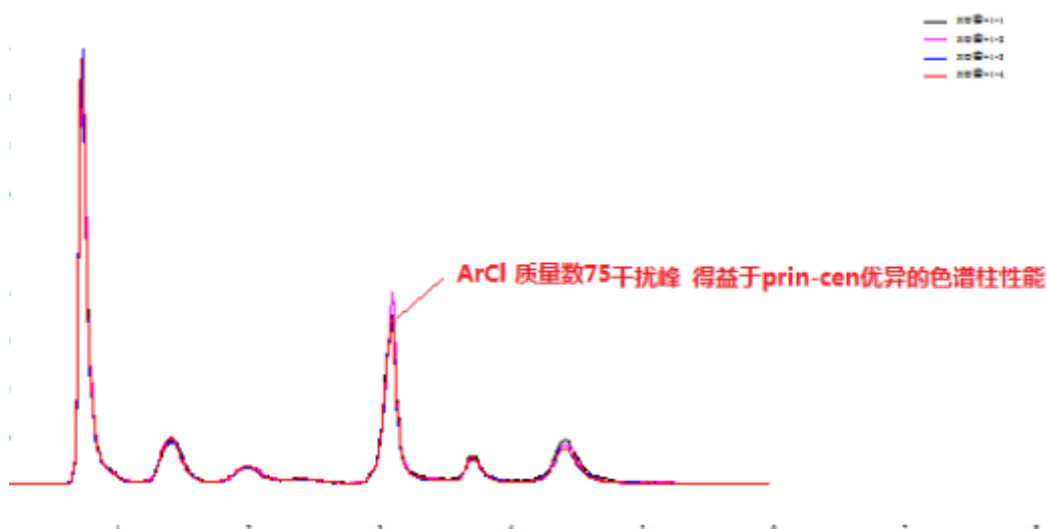
[DORM-4 \(PDF, 17](#)

Substance	Mass fraction, (mg/kg)
Arsenobetaine (as As) (h,i)	3.95 ± 0.36
Methylmercury (as Hg) (j,k)	0.355 ± 0.028

DORM-4有证标物标称值



XO酱高盐样品色谱图（标准模式）



4针XO酱 spike1ppb 5种砷形态标液

五、分析说明

1、由于砷形态存在不同形态的转化，特别在低浓度时，As³⁺和As⁵⁺很容易发生相互转化，建议配置好的标液要尽快上机测试。

2、建议试用Prin-Cen的ONLY WATER KIT只加水分析包，对ICPMS仪器友好；高盐、高磷、高碳的流动相会对锥造成堵塞的风险，从而降低仪器灵敏度。

3、Prin-Cen的方案对于一些氯含量的样品既可以使用标准模式(Prin-Cen优异的色谱柱性能，可以把样品的Cl离子分离开，避免ArCl对无机砷的干扰)，也可以使用碰撞气模式,把ArCl干扰降到最低。