

# SN

## 中华人民共和国出入境检验检疫行业标准

SN/T 4063—2014

### 出口植物性中药材中汞含量的测定 直接进样-冷原子吸收光谱法

Determination of mercury in botanic Chinese medicinal material for export—  
Direct injection-cold atomic absorption spectroscopy method

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## 前 言

本标准按照 GB/T 1.1—2009 给出的规则起草。

请注意本文件的某些内容可能涉及专利。本文件的发布机构不承担识别这些专利的责任。

本标准由国家认证认可监督管理委员会提出并归口。

本标准起草单位：中华人民共和国重庆出入境检验检疫局。

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# 出口植物性中药材中汞含量的测定

## 直接进样-冷原子吸收光谱法

### 1 范围

本标准规定了植物性中药材中汞含量的直接进样-冷原子吸收光谱测定方法。

本标准适用于人参、甘草、大青叶、紫苏叶、菊花、红花、枸杞、山楂、麻黄、藿香、黄柏、肉桂等植物性中药材中汞含量的测定。

### 2 规范性引用文件

下列文件对于本文件的应用是必不可少的。凡是注日期的引用文件,仅注日期的版本适用于本文件。凡是不注日期的引用文件,其最新版本(包括所有的修改单)适用于本文件。

GB/T 602 化学试剂 杂质测定用标准溶液的制备

GB/T 5330 工业用金属丝编织方孔筛网

GB/T 6682 分析实验室用水规格和试验方法

### 3 方法提要

样品通过进样器导入测汞仪中,经干燥后热分解,分解后的汞被还原成汞原子,汞原子蒸气在齐化管中进行金汞齐化反应、高温解析后在波长 253.65 nm 处进行测定,按照标准曲线法定量。

### 4 试剂和材料

除另有规定外,所用试剂均为优级纯,水为 GB/T 6682 规定的一级水。

4.1 硝酸:优级纯。

4.2 10%硝酸溶液:量取 100 mL 硝酸(4.1),用水稀释成 1 000 mL。

4.3 重铬酸钾。

4.4 重铬酸钾溶液(1 g/L):称取 0.1 g 重铬酸钾(4.3)溶于 100 mL 水中。

4.5 汞标准储备溶液(1 000 mg/L):按 GB/T 602 方法配制,或者直接使用有证标准物质。

4.6 汞标准中间溶液(20 mg/L):准确移取 2 mL 汞标准溶液(4.5)于 100 mL 的容量瓶中,加入 1 mL 重铬酸钾溶液(4.4),用硝酸溶液(4.2)定容,混匀,于室温下存储。

4.7 汞标准中间溶液(2.0 mg/L):准确移取 10 mL 汞标准中间溶液(4.6)于 100 mL 的容量瓶中,加入 1 mL 重铬酸钾溶液(4.4),用水定容,混匀。此溶液含汞 2.0  $\mu\text{g/mL}$ (溶液用时现配)。

4.8 汞标准工作溶液:分别移取 0 mL、0.10 mL、0.25 mL、0.50 mL、1.50 mL、2.50 mL、5.00 mL、10.00 mL、15.00 mL 汞标准中间溶液(4.7)和 2.5 mL、5.0 mL、15.0 mL、25.0 mL 汞标准中间溶液(4.6)于两组 100 mL 的容量瓶中,用水稀释至刻度,混匀。此时标准工作溶液对应的汞浓度分别为 0  $\mu\text{g/L}$ 、2  $\mu\text{g/L}$ 、5  $\mu\text{g/L}$ 、10  $\mu\text{g/L}$ 、30  $\mu\text{g/L}$ 、50  $\mu\text{g/L}$ 、100  $\mu\text{g/L}$ 、200  $\mu\text{g/L}$ 、300  $\mu\text{g/L}$ (低含量系列)和 500  $\mu\text{g/L}$ 、1 000  $\mu\text{g/L}$ 、3 000  $\mu\text{g/L}$ 、5 000  $\mu\text{g/L}$ (高含量系列)。

4.9 载气:氧气,纯度 $\geq 99.99\%$ 。

## 5 仪器和设备

5.1 测汞仪:配 2 个吸收池。

5.2 样品舟:材质为镍或相当者。

5.3 分析天平:感量 0.1 mg。

5.4 移液器:量程 10  $\mu\text{L}$ ~200  $\mu\text{L}$ 。

## 6 试样的制备与保存

6.1 取有代表性样约 200 g,用分析研磨机将样品粉碎成细粉(95%以上粉末能通过符合 GB/T 5330 中 R40/3 系列孔径为 0.25 mm 的标准试验筛),装入洁净容器,存储于干燥器中。

6.2 制样操作过程中应防止样品受到污染或发生残留物含量的变化。

## 7 测定步骤

### 7.1 测定

称取 0.1 g(精确至 0.000 1 g)试样于样品舟中,参照附录 A 的仪器条件进行测定。

### 7.2 标准曲线

分别吸取 100  $\mu\text{L}$  标准工作溶液(4.8)于两组样品舟中,此时各样品舟中对应的汞的质量分别为 0 ng、0.2 ng、0.5 ng、1 ng、3 ng、5 ng、10 ng、20 ng、30 ng(低含量系列)和 50 ng、100 ng、300 ng、500 ng(高含量系列),按照含量由低到高的顺序,参照附录 A 条件在波长 253.65 nm 处测定其吸光度。以相应汞的质量(ng)为横坐标,吸光度为纵坐标,绘制两条标准工作曲线。

### 7.3 注意事项

7.3.1 在仪器的重要部件没有更换时,一般无需进行标准曲线的重新绘制。测试前应使用与样品具有相当水平的汞标样进行校正。

7.3.2 如果样品中汞含量超出工作曲线的范围,可减少称样量进行测定。

## 8 结果计算和表述

按式(1)计算试样中汞的含量:

$$X = \frac{m \times 1\,000}{m_0 \times 1\,000} \dots\dots\dots (1)$$

式中:

$X$  ——试样中汞含量,单位为微克每千克( $\mu\text{g}/\text{kg}$ );

$m$  ——从工作曲线上查得的汞的质量,单位为纳克(ng);

$m_0$  ——样品质量,单位为克(g)。

取两次平行测定结果的算术平均值为试样的最终结果。



9 定量限和回收率

9.1 定量限

本方法定量限为 10 μg/kg。

9.2 回收率

各种基质的添加水平及回收率范围见表 1。

表 1 各种基质的添加水平及回收率范围

基质	添加水平 μg/kg	回收率范围 %	RSD %	基质	添加水平 μg/kg	回收率范围 %	RSD %
人参	10	72.5~91.3	9.5	甘草	10	75.0~103.1	10.5
	200	80.9~99.7	6.9		200	81.0~105.5	8.9
	400	91.8~99.6	2.9		400	81.9~102.6	9.2
大青叶	10	85.0~106.3	7.3	紫苏叶	10	86.0~102.8	6.3
	200	83.3~107.1	8.2		200	84.3~100.4	6.6
	400	85.0~93.8	2.9		400	92.3~100.3	3.1
菊花	10	96.0~94.9	5.0	红花	10	72.5~92.4	9.8
	200	81.9~105.6	9.4		200	82.5~106.0	9.1
	400	89.8~97.6	3.0		400	82.5~106.5	9.0
枸杞	10	91.5~109.8	6.3	山楂	10	74.4~91.9	8.1
	200	84.4~100.3	6.0		200	86.3~102.5	6.6
	400	80.0~101.6	8.6		400	84.5~100.2	5.9
麻黄	10	75.8~89.3	5.7	藿香	10	80.5~98.8	7.2
	200	81.3~99.4	6.8		200	85.0~94.8	4.0
	400	82.9~104.1	7.6		400	84.1~98.6	5.4
黄柏	10	81.5~106.1	8.4	肉桂	10	72.5~91.3	10.4
	200	89.4~98.1	3.2		200	81.2~104.9	7.5
	400	84.7~98.2	5.8		400	81.4~101.8	9.6

附 录 A  
(资料性附录)  
仪器参考条件<sup>1)</sup>

由于测试结果取决于所使用仪器,因此不可能给出测汞仪的通用参数。设定的参数应保证样品中的汞含量能够得到有效的测定。

下列给出的参数证明是可行的:

- a) 干燥温度:300 ℃;
- b) 干燥时间:70 s;
- c) 分解温度:800 ℃;
- d) 分解时间:120 s;
- e) 催化温度:600 ℃;
- f) 等待时间:60 s;
- g) 金汞齐温度:600 ℃;
- h) 汞齐化时间:12 s;
- i) 记录测量信号时间:150 s;
- j) 载气输入压力:89.6 kPa~117.2 kPa;
- k) 载气输出压力 2.6 kPa~10.3 kPa。

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1) 非商业性声明:附录 A 所列参考条件是在 TELEDYNE Leeman Labs Hydra-C 型全自动测汞仪上完成的,此次列出试验用仪器型号仅为提供参考,并不涉及商业目的,鼓励标准使用者尝试不同厂家或型号的仪器。

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## Foreword

This standard was drafted according to GB/T 1.1—2009.

Please note that some of the elements of this document may be involved in patent, issued the document does not assume the responsibility identification of these patents.

This standard was proposed by and was under the charge of National Regulatory Commission for Certification and Accreditation of the People's Republic of China.

The standard was drafted by Chongqing Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China.

This standard was mainly drafted by Zheng Guocan, Li Xianliang, Zhu Meiwen, Wang Jing, Zhang Lei, Liu Yi, Chen Jiang, Xiang Qinghua.

SN/T 4063—2014

# Determination of mercury in botanic Chinese medicinal material for export—Direct injection-cold atomic absorption spectroscopy method

## 1 Scope

The standard specifies the method of determination of mercury in botanic Chinese medicinal material for export by direct injection-cold atomic absorption spectroscopy.

This standard is applicable to the determination of mercury in ginseng, licorice, folium isatidis, perilla leaf, chrysanthemum, safflower, Chinese wolfberry, hawthorn, ephedra, patchouli, cortex phellodendri, cinnamon.

## 2 Normative reference

The following documents are necessary for this standard. For dated references, only dated editions shall apply to this standard. For undated references, the latest edition of the normative document (including subsequent amendments) is referred to applies.

GB/T 602 Chemical reagent—Preparations of standard solutions for impurity

GB/T 5330 Industrial woven metal wire cloth (square opening series)

GB/T 6682 Water for analytical laboratory use—Specification and test method

## 3 Principle

The sample was guided into the the mercury analyzer through the injector, drying and thermal decomposition, mercury which decomposed was reduced to atomic mercury vapor, and was reacted in gold amalgam, and, quantitated at the wavelength of 253.65 nm after resolution under high temperature, according to the standard curve.

## 4 Reagents and materials

Unless specified, all reagents shall be of analytical grade; “Water” is the first water prescribed by GB/T 6682.



4.1 Nitric acid: GR.

4.2 Nitric acid solution(10%): dilute 100 mL nitric acid(4.1) to 1 000 mL with water.

4.3 Potassium dichromate.

4.4 Potassium dichromate solution (1 g/L): Dilute 0.1 g Potassium dichromate (4.3) to 100 mL with water.

4.5 Mercury standard solution (1 000 mg/L): Prepared by GB/T 602 method, or use certificated reference material.

4.6 Mercury standard middle solution(20 mg/L): Accurately measured 2 mL the standard solution (4.5) in an 100 mL volumetric flask and added 1 mL Potassium dichromate solution (4.4) and dissolved with nitric acid solution (4.2) to form a middle standard solution of 20 mg/L. The middle standard solution was stored at the room temperature.

4.7 Mercury standard middle solution (2.0 mg/L) : Accurately measured 10 mL the middle standard solution (4.6) in an 100 mL volumetric flask and added 1 mL Potassium dichromate solution (4.4) and dissolved with water to form a middle standard solution of 2.0 mg/L.

4.8 Mercury standard working solution: Respectively measured 0 mL, 0.10 mL, 0.25 mL, 0.50 mL, 1.50 mL, 2.50 mL, 5.00 mL, 10.00 mL, 15.00 mL mercury standard middle solution (4.7) and 2.5 mL, 5.0 mL, 15.0 mL, 25.0 mL mercury standard middle solution (4.6) in different volumetric flask and diluted to 100 mL with water, the concentrations were 0 µg/L, 2 µg/L, 5 µg/L, 10 µg/L, 30 µg/L, 50 µg/L, 100 µg/L, 200 µg/L, 300 µg/L (low content range) and 500 µg/L, 1 000 µg/L, 3 000 µg/L, 5 000 µg/L (high content range).

4.9 Carrier gas: Oxygen, purity  $\geq 99.99\%$ .

## 5 Apparatus and equipment

5.1 Mercury analyzer: With 2 absorption cell.

5.2 Sample boats: materials for nickel or equivalent.

5.3 Electronic balance (0.1 mg).

5.4 Pipettes: the range of 10 µL~200 µL.

## 6 Sample preparation and storage

6.1 Take a representative sample of about 200 g, crushed with grinding machine (which more than 95% powder can through the 0.25 mm standard test sieve of GB/T 5330 R40/3 series ), put into a clean container, stored in a desiccator.

6.2 Certain measures shall be taken to prevent contamination of the samples or decomposition of the residues during the sample preparation procedure.

## 7 Analytical procedure

### 7.1 Determination

Weigh 0.1 g (accurate to 0.000 1 g) sample in the sample boat, and determined refer to instrument conditions in appendix A.

### 7.2 Standard curve

Respectively absorb 100  $\mu\text{L}$  standard working solution (4.8) in sample boats, the contents of mercury were 0 ng, 0.2 ng, 0.5 ng, 1 ng, 3 ng, 5 ng, 10 ng, 20 ng, 30 ng (low content range ) and 50 ng, 100 ng, 300 ng, 500 ng (high content range), then determined refer to instrument conditions in appendix A from low to high, drawing two standard working curve, Corresponding to the mercury mass (ng) as abscissa, absorbance as ordinate.

### 7.3 Note

7.3.1 In general, the cuve don't need to draw, if the important component in the instrument without replacement, the mercury standard solution was used to calibrate before determination.

7.3.2 Sample weight can be reduced if mercury content beyond the work curve.

## 8 Calculation and expression of result

Calculating the content of mercury according to the formula (1)

$$X = \frac{m \times 1\,000}{m_0 \times 1\,000} \dots\dots\dots (1)$$

where:

$X$  —— the content of mercury in the test sample,  $\mu\text{g}/\text{kg}$ ;

$m$  ——the content of mercury checked from working curve, ng;

$m_0$  ——sample weight, g.

Two parallel determinations on the average as the final results of the sample.

## 9 Limit of quantification ( LOQ ) and recovery

### 9.1 LOQ

The Limit of quantification was 10  $\mu\text{g/kg}$ .

### 9.2 Recovery

The recoveries ranges of mercury in every matrix were listed in table 1.

Table1—The spiked levels and recoveries ranges of mercury in every matrix

Matrix	Spiked levels $\mu\text{g/kg}$	Recovery ranges %	RSD %	Matrix	Spiked levels $\mu\text{g/kg}$	Recovery ranges %	RSD %
ginseng	10	72.5~91.3	9.5	licorice	10	75.0~103.1	10.5
	200	80.9~99.7	6.9		200	81.0~105.5	8.9
	400	91.8~99.6	2.9		400	81.9~102.6	9.2
folium isatidis	10	85.0~106.3	7.3	perilla leaf	10	86.0~102.8	6.3
	200	83.3~107.1	8.2		200	84.3~100.4	6.6
	400	85.0~93.8	2.9		400	92.3~100.3	3.1
chrysanthemum	10	96.0~94.9	5.0	safflower carthamus	10	72.5~92.4	9.8
	200	81.9~105.6	9.4		200	82.5~106.0	9.1
	400	89.8~97.6	3.0		400	82.5~106.5	9.0
Chinese wolfberry	10	91.5~109.8	6.3	hawthorn	10	74.4~91.9	8.1
	200	84.4~100.3	6.0		200	86.3~102.5	6.6
	400	80.0~101.6	8.6		400	84.5~100.2	5.9
ephedra	10	75.8~89.3	5.7	patchouli	10	80.5~98.8	7.2
	200	81.3~99.4	6.8		200	85.0~94.8	4.0
	400	82.9~104.1	7.6		400	84.1~98.6	5.4
cortex phellodendri	10	81.5~106.1	8.4	cinnamon	10	72.5~91.3	10.4
	200	89.4~98.1	3.2		200	81.2~104.9	7.5
	400	84.7~98.2	5.8		400	81.4~101.8	9.6



**Annex A**  
**(Informative)**  
**Reference conditions of instrument<sup>1)</sup>**

Because the test results depends on the used instrument, it is not possible to give general parameters of mercury detector. Set parameters shall ensure that the mercury content in the samples can be effectively measured parameters are given.

The following is proved feasible:

- a) Drying temperature: 300 °C ;
- b) Drying time: 70 s ;
- c) Decomposition temperature: 800 °C ;
- d) Decomposition time: 120 s ;
- e) Catalytic temperature: 600 °C ;
- f) Wait time: 60 s ;
- g) Gold amalgam temperature: 600 °C ;
- h) Gold amalgam time: 12 s ;
- i) Time of record the measurement signal: 150 s ;
- j) Inlet pressure of carrier gas: 89.6 kPa~117.2 kPa ;
- k) Outlet pressure of carrier gas: 2.6 kPa~10.3 kPa.

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1) Non-commercial statement: the reference mass parameters in Annex A are accomplished by TELEDYNE Leeman Labs Hydra-C, the equipment and its type involved in the standard method is only for reference and not related to any commercial aim, and the analysts are encouraged to use equipments of different corporation or different type.

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