

## 直接进样石墨炉原子吸收法测定动物脏器中微量铅

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**摘要** 本文描述了一种直接进样原子吸收测定动物脏器中微量铅的方法。动物脏器被预先捣碎至一定微粒,然后配置成悬浊液直接被注入石墨原子化器进行分析。由于不经过样品消解、分离与富集等操作,铅损失的可能性大大减小。整个过程几乎不用其他化学试剂,因而可避免因使用化学试剂而带进的污染。实验中研究了颗粒度、石墨炉工作温度等因素对结果的影响。用该法对几种动物脏器的分析证明,方法的分析精度可达 3.5—8.6%,加入回收率在 90—108% 之间。本法可作为一种测定动物样品中痕量金属元素的快速、简便的分析方法。

动物脏器中有害微量元素的测定既是食品分析中的一个重要课题,又是检验环境污染的指标之一。通过分析,对其食用性及受污状况进行分析判断,已日益引起人们的重视和兴趣。

石墨炉原子吸收法具有灵敏、快速、选择性高等特点,但在测定生物样品,特别是动物样品的预处理时,常用湿法消解、干法灰化等方法<sup>[1-4]</sup>,往往需经过一系列繁琐的步骤,对某些元素如铅、镉等,由于预处理中的损失引起回收率偏低,另一方面,受所用化学试剂及处理器皿的污染,又会使结果偏高,在分析中引进各种误差。

本文为简化分析步骤,应用近年来发展起来的直接进样无焰原子吸收法<sup>[5]</sup>,将动物脏器捣碎后制成悬浊液试样,然后直接注入石墨炉中测定微量铅,研究了原子化阶段等实验条件,用标准加入法进行定量,取得满意的结果,是一种对生物样品快速微量元素分析的简便、可靠的实验方法。

### 实 验 部 分

#### 一、仪器与试剂

日立 180-80 型塞曼原子吸收分光光度计,日立热解石墨管原子化器, Eppendorf 微量进样器(10 微升)。

铅空心阴极灯(上海电光器件厂)。

DS-1 型高速组织捣碎机(上海标本模型厂)。

铅贮备液:准确称取高纯金属铅,溶于少量 6mol HNO<sub>3</sub> 后用重蒸水稀释配制成 1mg/ml 贮存液。标准溶液及工作液用时临时配制。

消泡剂:分析纯正丁醇。

保护气:99.99% 氩气(上钢三厂)。

#### 二、分析步骤

将新鲜动物脏器洗净后,用滤纸吸干表面水分后称重,置于高速组织捣碎机中,加入重蒸水约 150ml,在每分钟转速为 10000 转次高速捣碎 4min,加入 5ml 正丁醇消除泡沫,将制得的悬浊液定量转移至 250ml 容量瓶中,用重蒸水稀释至刻度。在反复振摇的情况下,根据样品中铅的不同含量,移取等体积四份 $\leq 8$ ml 试液于 10ml 容量瓶中,后三份分别加入铅标样,定容后加入铅浓度依次为 20、40、60ng/ml,最后在摇匀时用 Eppendorf 微量进样器,取样 10 $\mu$ l 进石墨炉测定之。

#### 三、石墨炉工作条件

干燥	50—120℃	30s
灰化(1)	400—550℃	30s
灰化(2)	550—550℃	10s
原子化	2000℃	7s
清洗	2400℃	3s

## 结 果 与 讨 论

### 一、悬浊液试样的准备

直接进样石墨炉原子吸收法要求试样粒度细而均<sup>[6]</sup>,否则会降低测定的灵敏度,影响精度。本文对猪肝试样的捣碎时间作过条件试验,结果列于表 1。

表 1 捣碎时间的影响\*

捣碎时间 (min)	1	2	3	4	5
吸光度 (A)	0.129	0.106	0.109	0.126	0.124
变异系数 (%)	12.1	10.8	6.2	5.9	9.7

\* 7 次测定平均值

由表 1 可见,在 1 至 2 min 时,样品的均匀性较差,变异系数大于 10%,3 至 4min 时,粒度分布较为均匀,精度改善,4min 时信号值也较高,而增加至 5min 时,由于匀浆过程中产生大量气泡,使精度再次下降,变异系数值升高,故采用捣碎时间为 4min 处理各种试样。此外,为保证试样的均匀性,在石墨炉测定进样前,必须充分振摇,不然会引进较大的进样误差。

### 二、石墨炉升温条件

根据动物脏器蛋白质和脂肪含量高的特点,本文研究了适宜的石墨炉程序升温条件。

干燥阶段为防止试样飞溅,选择在较低温度时斜坡升温,控制在 50—120℃ 持续 30 秒,以利于水份及正丁醇的蒸发。

灰化温度的选择对生物样品十分重要。图 1 显示了猪肝试样和标样(浓度为 60ng/ml)在灰化阶段最高灰化温度与吸光度信号的关系。试样(曲线 2)在 500—700℃ 范围内信号略有下降但总趋势仍较平稳,700℃ 以后明显下跌,而标样(曲线 1)系纯水溶液体系,仅在 500—600℃ 范围内保持恒定,高于 600℃ 后信号值下降,因而选择最后温度为 550℃,并采用两步灰化方式,先在 400—

550℃ 持续 30 秒,接着在 550℃ 保持 10 秒。在此条件下引起非原子吸收的背景值吸光度信号在 0.1—0.3 之间,而本文所用仪器扣除背景吸收的能力可达 1.5,背景值完全可以扣除。

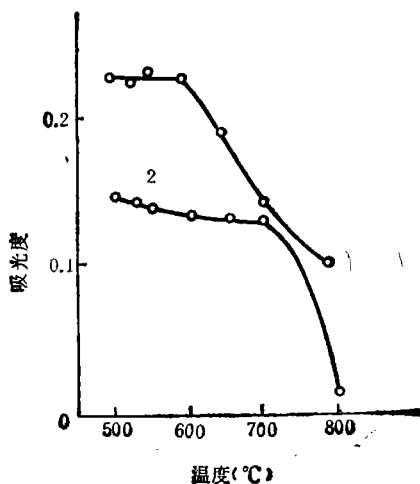


图 1 灰化温度曲线

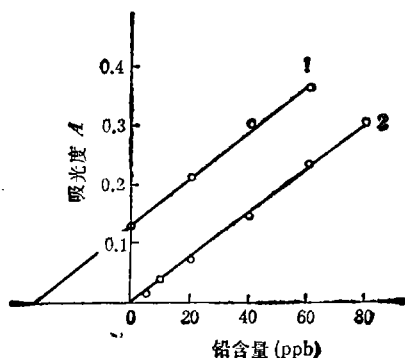


图 2 标准加入法用图

1. 猪肝试样 2. 标样

选择原子化温度的试验中,在 2000℃ 附近,随着原子化温度的升高,吸光度值也有所增加,考虑到石墨管的寿命,选用所用仪器推荐的原子化温度 2000℃、7 秒作为测试条件。

### 三、分析结果

分析方法应用标准加入法,以猪肝试样

表 2 回 收 率

试样	试液含铅量 (ng/ml)	铅加入量 (ng/ml)	铅测得量 (ng/ml)	回收率 (%)
猪肝	32	20	53	105
		40	75	108
		60	88	93
鸭肝	34	20	52	90
		40	76	105
		60	81	95
鸡肝	12	20	32	100
		40	50	95
		60	74	103
猪肾	46	20	68	110
		40	85	98
		60	103	95

为例作图, 见图 2。由图可见样线与标线斜率比较接近, 表明用本法测得的结果可靠性。

测定结果、回收率及变异系数列于表 2 及表 3。其中精度为未加标试液 10 次测定所得, RSD 在 3.5—8.6% 之间, 各种加入量的回收率在 90—110% 范围内。

本法快速、简便, 符合微量分析要求, 适

表 3 测定结果及变异系数

试 样	铅含量 (ng/g · 鲜重)	RSD (%)
猪肝	118	6.6
鸭肝	103	8.6
鸡肝	80	6.2
猪肾	329	3.5

用于生物样品中动物试样的分析研究。

## 参 考 文 献

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### Systems Analysis of Regional Environmental Management Information System

Cheng Shensong et al. (Department of Environmental Engineering, Tsinghua University, Beijing)

Systems analysis is in the first step of the structural design for Regional Environmental Management Information System (REMIS). The core of the step is to settle the problem "what has to do". The paper describes the general principles of systems analysis, the investigation of environmental management functions, the analysis of demands and the analysis of data construction and flow. An analytical method for REMIS has been suggested by stressing that systems analysis must face to the function demands of REMIS. (See pp. 52—58)

### Calculation of Energy Flow and Analysis of Ecological Benefit in the Agro-Ecosystem of

### Dongxu Village, Jiangsu Province

Lu Tienong et al. (Yancheng Municipal Environmental Protection Agency, Jiangsu Province)

The agro-ecosystem of Dongxu Village is divided into three subsystems: producer, consumer and decomposer. The authors set up a calculating method, by which energy flow in the process of biological production was calculated. It was showed that the ecological structure in the system was getting access to rationalization. The ratio of energy input-output was improved in recent years, that the system was changed consumptive type into productive type. By calculation of energy flow and benefit analysis, the results show that the system has coordinated economic development and eco-environmental construction simultaneously. (See pp. 71—76)

*Chen Zhongliang et al.* (Research Center for Eco-Environmental Sciences, Academia Sinica, Beijing)

Total suspended particulates (TSP) and their chemical composition in Beijing-Tianjin Area have been analyzed. There were 12 elements (Cd, Cu, K, Mn, Pb, Zn, Fe, Na, Ni, Cr, Sr, Ba), 5 kinds of ions (F, Cl, NO, SO, NH) and 26 kinds of organic pollutants in the regional distribution. The sources of TSP were estimated by means of organic odd/even carbon number ratios. Atmospheric pollution affected mutually among Beijing, Tianjin, Lanfang and Gixian County were studied with identification analysis of inorganic elements. (See pp. 24—27)

### **Economic Optimization in Noise Reduction by Sound-Absorbing Treatment**

*Kang Jian* (Architecture Department, Tsinghua University, Beijing)

In the design of noise reduction by sound-absorbing treatment, there may be several schemes that can attain the goal of anticipated noise reduction under approximate decorative level. However, the costs are very different. So it is necessary to choose an optional one. This paper presents a maths model to solve the optimization problem. The corresponding computer program and a case study are also given. (See pp. 30—33)

### **Removal of Cd<sup>2+</sup> and Cu<sup>2+</sup> Ions in Water Solution by the Modified CARIX Ion-Exchange Technology**

*Ma Shishen* (The Institute of Atomic Energy, Beijing);

*W. H. Hoell and S. H. Eberle* (Institute of Radiochemistry, Karlsruhe Nuclear Research Center, Federal Republic of Germany)

This paper reports a new technology of ion exchanges in removing Cd<sup>2+</sup> and Cu<sup>2+</sup> from the dilute solutions of cadmium sulfate and copper sulfate. Its advantages are derived from the use of carbon dioxide and magnesium compounds as regenerants. As carbon dioxide is a non-polluting chemical, it could diminish saline loads in water bodies. The experimental results show that a partial conversion of the resin to magnesium is achieved and the effect of desalination is satisfied. In addition, the concentration of magnesium ions in the regenerant is higher than that of carbonic acid and calcium ions during regeneration. It increases effective cation exchange capacity of the weakly acid cation exchange resins. (See pp. 36—40)

### **Petroleum-Sulfoxide Extract-Leach Resin: A New Resin for Treatment of Wastewater Containing Methylmercury**

*Jia Jinping* (Department of Applied Chemistry, Shanghai Jiao Tong University); *Zhou Yi* (Department of Applied Chemistry, China University of Sciences and

Technology, Hefei, Anhui Province); *Peng An* (Research Center for Eco-Environmental Sciences, Beijing)

This paper presents the study on possibilities of treating low-concentrated methylmercury in wastewater by petroleum sulfoxide (PSO) extract-leach resin, in which PSO as a cheap extractant, can be obtained from raw oil containing high sulfur content. The experimental results were as follows: (1) the resin was available to sorb more than 99% methylmercury (concentrations 10-20ppb) in pH range 5—8; (2) current velocity of effluent affected sorption capacity. As the velocity increased from 0.5 ml/min to 5.0 ml/min, the sorption capacity decreased from 99.66% to 98.90% in bed column (diameter 0.8 cm, length 10.3 cm, resin weight 3.0 g); (3) the resin could be regenerated easily by means of eluting it with the regenerant (4mol HCl); (4) Mg(II), Fe(II), Fe(III), Ag(I), Cu(II), Hg(II), FA(fulvic acid) and Cl<sup>-</sup> ions had no influence on sorption capacity of the resin except Hg(II) and Ag(I). (See pp. 40—44)

### **Determination of Trace Selenium Using Hydride Generation-Silver Selenide Sol Method**

*Niu Jianjun and Wang Bingwu* (Changchun Institute of Applied Chemistry, Academia Sinica)

A new method for determination of trace selenium has been presented. Se(IV) was converted into H<sub>2</sub>Se by reaction with KBH<sub>4</sub> pellet in 0.2 mol H<sub>2</sub>SO<sub>4</sub>-2% tartaric acid solution and the escaped hydride was absorbed and colored by silver nitrate solution in the presence of gum arabic. Beer's law was obeyed in the range of 0—3 μg Se(IV)/3ml, the colored solution gave an absorption maximum at 246 nm with detection limit 0.04ppm, most of the foreign ions did not interfere with the determination of selenium. The method has high sensitivity and selectivity and has been applied to analyse urine selenium yeast and other samples. (See pp. 45—48)

### **Determination of Trace Lead in Animal Organs Using Direct Sampling Flameless Atomic Absorption Spectrophotometry**

*Xu Tonming et al.* (Department of Chemistry, East China Normal University, Shanghai)

The animal organs were ground to be very fine particles, with which suspended solution was prepared and then it was analyzed in graphite atomizer by direct sampling. Because the whole procedure would be completed without sample dissolution, separation and concentration, and no reagents were used during sample preparation, the samples so were avoided from contamination. In the experiment, several parameters were investigated, for example, the effects of particle sizes of the samples and of temperature program of atomizer. The results showed that the precision of this method for analyzing different animal organs covered a range of 3.5—8.6% and recovery ratio a range of 90—108% with spiked 20—60 ng/ml of lead. (See pp. 49—51)

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