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原子吸收法直接测定土壤中的铅

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摘 要: 用 HNO₃- HF- HClO₄ 消解土壤样品, 直接用原子吸收分光光度计测定其中的铅. 实验结果表明, 此方法有较好的准确度, 精密度和回收率. 测定标准土样, 结果与推荐值相吻合.

关 键 词: 土壤铅; 原子吸收法; 测定
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测定土壤中的铅, 由于其含量低, 基体干扰大, 一般采用萃取分离基体元素后, 用石墨炉原子吸收法测定. 但萃取分离过程复杂、烦琐. 石墨炉原子吸收法, 对样品的加入要求有较高的准确性和稳定性, 其最佳工作条件即干燥、灰化、原子化、清洗的温度和时间难以摸索. 铅属低温原子化元素, 其实无须用石墨炉原子吸收法. 本方法适当加大样品量, 采用硝酸- 氢氟酸- 高氯酸消解土壤样品, 直接用火焰原子吸收法测定铅, 步骤简便、省时、省试剂, 结果令人满意.

1 实 验

1.1 仪器及工作条件

1.1.1 仪器 澳大利亚 GBC- 932 型原子吸收分光光度计, 铅空心阴极灯.
1.1.2 工作条件 波长: 217. 0nm; 狭缝: 1. 0nm; 灯电流: 5. 0mA; 燃烧器高度: 12. 5mm; 空气流量: 10. 0l/min; 乙炔流量: 2. 00l/min.

1.2 试剂

硝酸(优级纯); 氢氟酸(优级纯); 高氯酸(优级纯); 盐酸(优级纯).
铅标准溶液 称取光谱纯金属铅 0. 1000g 于烧杯中, 加 5ml 浓硝酸, 加热溶解, 冷却后转入 100ml 容量瓶中定容, 即浓度为 100μg/ ml 的铅标准储备液.

1.3 操作步骤

准确称取土样 1. 000g 于聚四氟乙烯坩埚中, 加硝酸 5ml、高氯酸 2ml、氢氟酸 5ml, 在电热板上加热溶解, 成黄白色糊状, 冷却后, 再加高氯酸 2ml 以赶尽氢氟酸, 冷却, 再加 2mol/ L 盐酸 5ml, 微热溶解残渣, 移至 50ml 容量瓶定容, 同时做空白, 待测.

2 结 果

2.1 方法的准确度、精密度和检出限

按以上方法测定国家地质矿产部化探分析质量监控站提供的地球化学标准样, 结果见表 1. 检出限按 D. L. = 3S_{wb}计算.

样品	Pb			
	推荐值 (μg/ g)	测定值 (μg/ g)	RSD (%)	检出限 (μg/ g)
GSS- 3	26±1. 3	27. 55	2. 52	0. 06
GSS- 4	58. 5±2. 1	59. 96	1. 08	0. 06
GSS- 5	552±14	568. 50	0. 54	0. 06
GSS- 7	13. 6±1. 2	15. 68	4. 18	0. 06

从表 1 中可看出, 标准样品中铅的含量, 无论高或低, 测定值与推荐值基本吻合, 低含量的测定值稍偏高. 5 次测定的平均值与推荐值的相对标准偏差 < 5%, 这表明本方法的准确度和精密度都很好.

2.2 回收率试验结果

在土样消化液中加入不等量的铅标准液, 测定其中铅的含量, 计算其回收率, 结果如表 2.

样 品	标准土样中铅的回收率			
	Pb 测定值 (μg/ g)	Pb 加入值 (μg/ g)	Pb 总量测定值 (μg/ g)	加标回收率 (%)
GSS- 3	27. 55	10	38. 12	105. 7
GSS- 4	59. 96	10	70. 12	101. 6
GSS- 5	568. 50	5	573. 27	95. 4
GSS- 7	15. 68	10	26. 05	103. 7

从表 2 中可看出, 加标回收率在 95% ~ 105% 之间, 说明本方法有较好的回收率.

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3 讨论

1. 此方法由于使用了氢氟酸,使土壤中的大量硅挥发除去,减少了基体元素的干扰,但在消解过程中一定要除尽氢氟酸,否则将腐蚀玻璃器皿,而造成结果偏高。

2. 总的测定结果尽管都在误差允许的范围之内,但都有稍稍偏高的趋势,这可能是消化过程中引起的污染。

3. 消化过程中,应将样品消化为白色或略带黄色,呈水银状,否则将因消化不完全而造成结果偏低。

总之,通过试验,我们可以直接用原子吸收火焰法测定土壤中的铅。比萃取火焰法或萃取石墨炉法操作简便,节省人力、物力,同样能获得满意的结果。

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Contents & Abstracts

Comparison of taxonomic classification between Udic Luvisol and Udic Camisol (1)

WU Ke-ning, QU Chen-xiao, LU Qiao-ling (Henan Agricultural University, Henan Zhengzhou 450002, China)

Abstract: We selected some representative soil profiles in Hanzhong, Nanyang, and Nanjing. The soil properties from the profiles were analyzed and diagnostic horizons and features were identified. Thereafter, we classified the soils and did a comparison by using Chinese, US and international systems respectively.

Keywords: Luvisol; Camisol; Soil taxonomy; Comparison

Properties and genetic mechanisms of the secondary salinized soils in Jingdian irrigation area of Gansu Province (4)

LI Xiao-gang, CUI Zhi-jun, WANG Lin-ying (Dept. of Resources and Environmental Sciences, Gansu Agricultural University, Gansu Lanzhou 730070, China)

Abstract: The secondary salinized soils in Jingdian irrigation area were investigated. The salinized soils were classified as the types of chloride-sulfate and sulfate-chloride and water soluble cations were mainly sodium in the soils. Ca carbonate contents in the whole profiles were high and a low amount of gypsum was found. The saturation of Na was also high, thus the soils were solonized by sodium and the solonization was accompanied by salinization.

Keywords: Jingdian irrigation area; Secondary salinization; Genetic mechanisms; Protection measures

Effects of water and Boron in subsoils on oilseed rape growing under different soil moistures (8)

FANG Yi-hua (Zhejiang University, Zhejiang Hangzhou 310029, China)

Abstract: In order to investigate the effect of water and boron in subsoils on oilseed rape planted under different soil moistures, a pot experiment was conducted in greenhouses. The soil in pots was constructed with 3 layers of 15, 2, 20 cm respectively from top to bottom. The oilseed rape was treated for 30 days at the stem elongation stage. The results showed that the plant growth was decreased greatly by low soil moisture. However, the plant used boron more efficiently when soil moisture was high in subsoil and the availability of boron was also changed.

Keywords: Oilseed rape; Subsoil; Soil moisture; Boron

Separation and identification of biological active organic acids in chicken feces (12)

WANG Lin-quan, WANG Jun-ru, ZHOU Chun-jun, et al., (Northwestern Sci-Tech University of Agriculture and Forestry, Shaanxi Yangling 712100, China)

Abstract: Biological active organic acids were studied by using biological vitality tracing methods and a biological active substance was identified by infrared spectrum and mass spectrographic analysis as 3-O-glucosyl-1, 6-D-glucuronate-2(1), 20-20L-cholurate Na(C₃₆H₅₆O₁₈Na).

Keywords: Chicken feces; Organic acids; Infrared spectrum; Mass spectrography

Soil amendment by the addition of waste solution from mushroom culture (15)

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