

Ag⁺-Cadion-T_x-100 胶束增溶光度法测定 废水中的氰化物*

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摘要 在 Triton X-100 存在下, 利用 Ag⁺与 Cadion 的灵敏显色反应, 建立了痕量氰化物的间接光度测定方法。该方法灵敏度高, 选择性好, 可用于废水中痕量氰化物的测定。测定范围为 0—10 μg/25 ml, 其相对标准偏差小于 9.0%。

关键词 氰化物, 镉试剂, 光度法。

镉试剂(Cadion)是一种灵敏的三氮烯类光度分析试剂, 曾用于 Ag⁺的萃取光度法^[1-2]和胶束增溶光度法^[3-4]测定。笔者利用银可与镉试剂络合显色, 又易与 CN⁻生成银氰络离子的特点, 建立了一个关于痕量氰的间接光度测定方法。该方法灵敏度高, 选择性好, 与文献方法^[5-7]比较, 其稳定性和重复性均令人满意。可用于废水中痕量氰化物的测定。

1 实验部分

1.1 试剂和仪器

银标准溶液 以优级纯的硝酸银配制含 Ag⁺ 1.00 mg/ml 的水溶液作为贮备液, 使用前用蒸馏水稀释 100 倍, 即 10.0 μg/ml。

Cadion 溶液 0.04% (W/V) 乙醇溶液。

氰标准溶液 以分析纯的氰化钾配制, 并用 AgNO₃ 标准溶液标定。稀释至 CN⁻ 为 2.00 μg/ml 作为工作标准。

Triton X-100 溶液 4% (V/V) 水溶液。

Sørensen 缓冲溶液 pH=9.2—9.4 的硼砂-氢氧化钠溶液。

753W 型分光光度计(上海分析仪器厂)。

1.2 试验方法

依次取 1 ml Triton X-100 溶液, 1.5 ml Cadion 溶液, 5 ml 缓冲溶液和 1.5 ml 10.0 μg

Ag⁺/ml 工作液于 25 ml 容量瓶中, 稳定片刻后加入不超过 10 μg 的 CN⁻ 标准, 用蒸馏水稀至刻度, 摇匀。15 min 后于波长 526 nm 处, 分别以不含 Ag⁺、CN⁻ 的空白液作参比用 1 cm 比色皿测其吸光度。

1.3 含氰废水的预蒸馏

预蒸馏采用环境标准方法推荐的酒石酸-硝酸锌法^[8]。取 250 ml 含氰废水于 250 ml 全玻蒸馏器中, 加入 2 g 酒石酸和 2 ml 10% 硝酸锌溶液, 迅速连接蒸馏装置并进行蒸馏。用盛有 5 ml 0.1 mol/L NaOH 溶液的 50 ml 容量瓶吸收馏分。并用蒸馏水定容, 移取适当体积的馏分于 25 ml 容量瓶中。

2 结果与讨论

2.1 吸收曲线

在 Triton X-100 存在及 pH=9.2—9.4 条件下, 试剂 Cadion 于波长 425 nm 处有最大吸收, Ag⁺-Cadion 络合物在 526 nm 波长处有最大的吸收峰, 其表观摩尔吸光系数为 9.8×10^4 L · mol⁻¹ · cm⁻¹ (见图 1)。若在 CN⁻ 存在下, 则 Ag⁺-Cadion 吸收峰逐渐减弱, 直至不吸收。

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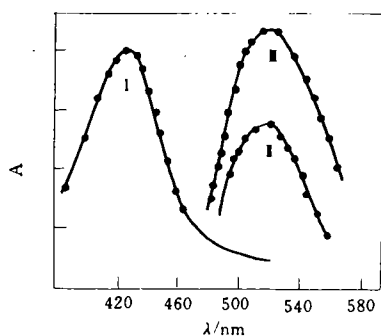


图 1 吸收曲线

I. Cation 溶液 II. Ag^+ -Cation 溶液 ($7 \mu\text{g Ag}^+$)III. Ag^+ -Cation 溶液 ($12 \mu\text{g Ag}^+$)

2.2 酸度的影响

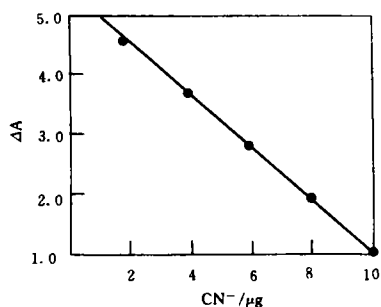
在 $\text{pH}=8.0-10.0$ 内考察了 $5 \mu\text{g CN}^-$ 存在下 Ag^+ -Cation 的显色情况。实验结果表明：在 $\text{pH}=9.0-9.5$ 内络合物具有稳定的吸光度。工作中选择 $\text{pH}=9.2-9.4$ 来测定 CN^- 含量。

2.3 Cation 用量的影响

研究表明，引入 CN^- 后的褪色程度（吸光度差值 ΔA ）与 Cation 用量有关，Cation 溶液用量为 $1.0-2.5 \text{ ml}$ ，则络合物颜色稳定且 ΔA 有最大值。工作中采用 1.5 ml Cation 溶液。

2.4 工作曲线

引入一系列 CN^- 标准于 Ag^+ -Cation 显色体系之中，分别测其吸光度。结果表明， $10 \mu\text{g}$ 以下的 CN^- 对显色反应的褪色具有稳定作用，其 ΔA 与 CN^- 量成线性关系（图 2）。

图 2 CN^- 测定的工作曲线

2.5 共存离子的影响

工作中分别考察了共存离子的影响：①

Zn^{2+} 、 Cd^{2+} 、 Hg^{2+} 、 Fe^{2+} 、 Fe^{3+} 对 Cation 有络合作用， $10 \mu\text{g}$ 内的 Zn^{2+} 、 Cd^{2+} 对 CN^- 褪色无影响， $10 \mu\text{g}$ 的 Fe^{2+} 、 Fe^{3+} 、 Hg^{2+} 对低于 $5 \mu\text{g}$ CN^- 的测定有严重影响， CN^- 测定率仅为 5% 左右。② $\text{Zn}(\text{CN})_4^{2-}$ 、 $\text{Cd}(\text{CN})_4^{2-}$ 使褪色加剧，并且添加 $\text{Zn}(\text{CN})_4^{2-}$ 、 $\text{Cd}(\text{CN})_4^{2-}$ 的量在一定范围内与 ΔA 值也具线性关系。③ $\text{Fe}(\text{CN})_6^{4-}$ 、 $\text{Fe}(\text{CN})_6^{3-}$ ， $\text{Hg}(\text{CN})_6^{4-}$ 对 CN^- 的测定无显著影响。④ Cl^- 、 Br^- 、 I^- 、 S^{2-} 、 SCN^- 等离子对 Ag^+ -Cation 显色也具褪色作用。

2.6 环境水样的测定

采集某电镀厂的废水进行测定，同时用加入回收法测定回收率。表 1 结果表明，测定值的相对平均偏差小于 9.0%，回收率达 91% 以上。

表 1 环境水样的测定结果

测定次数	平均值 (μg)	相对平均 偏差(%)	加入回收测定		
			加入量 (μg)	平均值 (μg)	回收率 (%)
10	4.82	3.7	2.00	6.77	97.5
10	6.17	4.1	2.00	8.09	91.0
10	7.00	8.4	2.00	8.93	91.5

3 结论

研究结果表明， Ag^+ -Cation-Tx-100 胶束增溶光度体系可用于氰化物的间接测定。对于含氰废水的样品，先经预蒸馏-碱液吸收法消除干扰，再用文中体系测定，其测定范围为 $0-10 \mu\text{g}/25 \text{ ml}$ ，测定值相对平均偏差小于 9.0%，结果令人满意。

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tor. Zhou Lu et al. (Dept. of Environ. Eng., Tsinghua Univ., Beijing 100084); *Chin. J. Environ. Sci.*, **17**(2), 1996, pp. 54–56

This paper indicated a new way to speed up granulation in UASB reactor by adding GAC. The results of experiment on UASB reactor with and without GAC treating effluence from petrochemical plant showed that the time of sludge granulation in the reactors with and without GAC were 39 days and 63 days respectively. The sludge pellet in the reactor with GAC had larger diameter than one in the reactor without GAC, and maximum COD removal rate was above 86%. Organic load of reactor with GAC was 2 times larger than the reactor without GAC. The reactor with GAC became more steady in process.

Key words: UASB reactor, granular active carbon, sludge pellet, granulation.

A Study on New Purification of High Concentration Sulphur Dyeing Sewage. Sun Jianhui et al. (Research Institute of Environ. Sci., Henan Normal Univ., Xinxiang 453002); *Chin. J. Environ. Sci.*, **17**(2), 1996, pp. 57–59

The combined technology of coagulative precipitation-iron chippings filtering-alkaline separation was used to purify the high concentration sulphur dyeing sewage. The results in the mode of production showed that the removal rates of sulphide, COD, BOD₅ and colority were 97.0%, 87.4%, 85.7% and 98.9% respectively. Every pollution index is up to the National Water Emission Standard. This technology system has the advantage in run stable, operation and management easy, engineering invest low, and treatment cost cheap.

Key words: coagulative precipitation, iron chippings filtering, alkaline separation, sulphur dyeing sewage.

Ecological Restoration of Coal Mining field in Loess Plain. Xue Ling et al. (Inner Mongolia Environmental Research Institute, Huhhot 010010); *Chin. J. Environ. Sci.*, **17**(2), 1996, pp. 60–63

This paper reports the results of an ecological restoration project in the Zhunger open-cut coal mining field in Inner Mongolia. The design, technique and effectiveness of the project are described. The results showed that the key points for ecological restoration in the Loess Plain condition include: selection of suitable plant species, proper soil treatments plus necessary construction work, adapt suitable ecological planting patterns based on site conditions. More than sixty plant species and eight ecological planting patterns were evaluated. Over 70% plant coverage and high yields of the crops (compared with the same crops in the nearby area) were obtained. A good ecological and economical return was achieved from the project.

Key words: ecological restoration, demonstrative project, Zhunger, open-cut coal mine in inner mongolia, land reclaim.

Determination of Atmospheric Methyl Bromide by Photoionization Detector. Zhong Jinxian and Liu Ye (Research Center for Eco-Environmental Sciences, Chinese Academy of Sciences, Beijing 100085); *Chin. J. Environ. Sci.*, **17**(2), 1996, pp. 64–65

Atmospheric CH₃Br was trapped by Tenax adsorbent and was determined by gas chromatography with photoionization detector. The CH₃Br concentrations in indoor and

outdoor are 24×10^{-12} (V/V) and 77.4×10^{-12} (V/V). The standard deviation and detection limit for the method are 0.0521, 10 pg respectively. The level of CH₃Br pollutions is 10^{-11} (V/V).

Key words: methyl bromide, GC, photoionization detector.

Indirect Spectrophotometric Determination of Trace Cyanide by Means of the Colour Reaction of Silver with Cation in Presence of Triton X-100. Gong Churu et al. (Dept. of Chemistry, Hubei Normal University, Huangshi 435002); *Chin. J. Environ. Sci.*, **17**(2), 1996, pp. 66–67

Silver gives a colour reaction with Cation in the presence of Triton X-100, and suppression of the colour competitive complex ion of the silver can be used for the indirect spectrophotometric determination of trace cyanide. Cyanide in waste water can be separated by distillation from other ions that also interfere, and then determined. The determination range of this method is 0–10 µg/25 ml. The recoveries of standard cyanide added to waste water samples are in the range of 91.0–100%, and its relative standard deviation is less than 9%.

Key words: cyanide, cation, indirect spectrophotometry.

The Liquid-Solid Extraction of Methomyl in Environmental Water and Gas Chromatographic Analysis. Chen Yanjun et al. (Dept. of Chemistry, Jining Medical College, 272113); *Chin. J. Environ. Sci.*, **17**(2), 1996, pp. 68–70

A solid-phase extraction procedure of methomyl from environmental water with active carbon cartridge for gas chromatographic analysis is presented. The detection limit and the minimum detectable concentration of methomyl in water were 0.2 ng and 0.2 µg/L, respectively. The average recoveries of methomyl as added to water were in the range of 95.8–100.7%. The relative standard deviations were lower than 5%.

Key words: methomyl, solid-phase extraction, active carbon, gas chromatography.

Determination of Trace Levels of Nitrophenols in Water by Polyvinylpyrrolidone Modified Carbon Paste Electrode. Wang Kaixiong et al. (Dept. of Environ. Protection, Zhejiang Agricultural University, Hangzhou 310029); *Chin. J. Environ. Sci.*, **17**(2), 1996, pp. 71–73

In this paper, a method to determine simultaneously p-nitrophenol and 2,4-dinitrophenol in water by differential pulse voltammetry (DPV) with a carbon paste electrode modified with 10% (W/W) polyvinylpyrrolidone (PVPr) was described. The electrode showed a strong response to the nitrophenols, and the sensitivity and selectivity were much higher than ordinary carbon paste electrode. The nitrophenols were preconcentrated in 0.1 mg/L KCl solution during open circuit period, then the preconcentrated nitrophenols at the electrode were determined in phosphate buffer solution (pH7.0). The oxidation peak potentials were +0.88 V and +1.23 V (VS Ag-AgCl) respectively and two current peaks were completely separated. The detection limits were 0.50 µg/L (p-nitrophenol) and 1.80 µg/L (2,4-dinitrophenol). This method had been applied to the determination of nitrophenols in pol-