

线性范围的测定,所用浓度的上限为 2.14×10^{-7} g/mL,下限为 2.14×10^{-10} g/mL,分别求得不同浓度标样的两波长定域同步荧光光谱峰,再由其峰高对其浓度作图(取对数),可获得良好的线性关系,相关系数为0.9999(表3),测得该方法的线性范围至少可达三个数量级。

五、小 结

1. 建立的两波长定域同步荧光光谱法(SFDW),不经预分离而直接检测环境样品的提取物中BaP的含量,与目前常用的HPLC方法测定结果接近,相对误差小于 $\pm 20\%$,一个样品的一次测定由HPLC所需1小时左右时间缩短为几分钟,因此,适合现场环境监测使用。

2. 两波长定域同步荧光光谱法(SFDW)具有灵敏度高(检测限为 4.5×10^{-10} g),线性范围宽(至少三个数量级),回收率高(96.8%)等优点。

3. SFDW使用的仪器主要是同步荧光分光光度计,由于同步荧光分光光度计通常可由普通荧光光度计(目前我国标准方法中规定使用)改装而成,因此有可能以少量投资,即可推广应用。

4. 将超声提取法^[8]和SFDW联用,则可组成一个对环境样品作快速提取、直接测定的技术路线,为测定BaP的仪器化自动化提供了一种简便方案。

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地下水中²²²Rn的液体闪烁测量法

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摘要 本文介绍的测量地下水中²²²Rn的液体闪烁法,具有简便、快速、灵敏、可靠等优点。该方法仅需水样10ml,制样时间不超过1min,探测限可达0.82Bq/L,能实现样品测量的自动化。它不需要特殊的测量装置,普通的商品液体闪烁计数器即可满足要求。是一种进行大规模地下水样中²²²Rn浓度调查的,较为理想的方法。

关键词 液体闪烁,氡气,²²²Rn,水。

通常,水中的氡气都是使用闪烁室法^[1]测量的。即将溶解水中的氡气直接或先经过浓缩装置处理再转移至抽成真空的闪烁室内,然后把闪烁室坐在光电倍增管上,测量 α 放射性。这一方法的探测限可达十分之几Bq/L或更低。但花费时间多,制样麻烦,对设备的要求亦较严格。本文要介绍的液体闪烁法,比较简便、快速,灵敏度亦能够满足地下水样分析的要求。该法只需10ml水,样品制备只需1min,可使

用商品液体闪烁分析仪,能实现自动换样和数据处理,非常适于大规模水样分析。

一、方法原理

氡气能溶于水中,但溶解度并不很高,0℃时为0.51,20℃时为0.25,40℃时为0.16^[2]。然而,它却极易溶解于许多有机溶剂中。例如,氡

气在室温下,在经常用作液体闪烁测量溶剂的甲苯中的溶解度就相当高。氡气的这一特性给使用液体闪烁法测量水中的氡气浓度提供了理论基础。当将含氡水样与适宜的闪烁剂,在一个密闭系统中充分混合后,大部分氡气被萃取到闪烁液中,并很快与其短寿命子体达到放射平衡。例如,放置 3h,子体的放射性达到平衡值的 99%。在自 ^{222}Rn 至 ^{214}Po 的衰变链中包含有 5 个能量很高的放射性粒子——3 个 α 粒子(能量分别为 5.49、6.0 和 7.69 Mev)及 2 个 β 粒子(最大能量分别为 0.69 和 3.26 Mev)。因此,1Bq 与短寿命子体处于放射性平衡的 ^{222}Rn ,每分钟放射出 300 个粒子。由于液体闪烁测量法的固有优点,以及这些粒子的能量都很高,实际上对它们的探测效率接近 100%^[3]。

二、实验条件的选择

(一) 实验器材

测量装置为美国 PACKARD 公司生产的 TR1-CARB 2250 CA 型低水平液体闪烁分析仪。测量杯为同一公司生产的体积为 20ml 的玻璃质闪烁杯。闪烁剂为上海试剂一厂生产的 PPO (闪烁纯)和 POPOP (光谱纯)。溶剂为天津化学试剂二厂生产的分析纯甲苯。氡水是将活度为 10^{-3} 居里的液体镭源中积累的 Rn 气用鼓气法鼓入蒸馏水中制备。

(二) 闪烁液的选择

本世纪初就有人注意到氡气在许多有机溶剂中都有很高的溶解度^[4]。我们根据 Weigel 给出的公式^[5],计算了氡气在不同温度下,在几种有机溶剂中的溶解度并列于表 1 中。为进行

比较,在最后一行也列出了相应温度下,氡气在水中的溶解度。从表中可以看出,氡气在各种有机溶剂中的溶解度有很大差别,而且随温度变化很大。其中,二硫化碳、己烷、乙醚和甲苯都是氡气的良好溶剂。然而,从作为液体闪烁测量溶剂的角度来看,虽然氡气在二硫化碳中的溶解度最大,但它对闪烁过程有强烈的淬灭作用,因而不适宜作闪烁溶剂^[5];己醚的沸点太低,不易操作,亦不适用;氡气在甲苯中的溶解度虽低于己烷,但其光输出量却是己烷的 400%。对于实际使用更为重要的是,不仅氡气在甲苯和水中溶解度的比值很高,而且这一比值在室温下随温度变化不大。例如,这一比值在 20℃ 时为 50,在 30℃ 时为 52,在 35℃ 时为 53。因此,我们选取甲苯作为测量氡气的闪烁溶剂。我们选取的闪烁剂 PPO 和 POPOP 的浓度分别为 4g/L 和 50mg/L,这与 Prichard 等人^[2]使用的相同。

(三) 相比

这里所说的相比,是指在使用的标称体积为 20ml 的闪烁杯内水样和闪烁液的体积之比。为选择合适的相比,我们作了两个实验:①固定水样体积为 10ml,改变闪烁液用量;②固定闪烁液用量为 5ml,改变水样体积。这两个实验的结果分别绘于图 1 和图 2 中。从图 1 可见,闪烁液的体积对相对计数率有一定影响。开始,计数率随闪烁液体积增大而增加,闪烁液量超过 5ml 后,尽管体积增大,计数率亦基本保持不变。为节省试剂,选取 5ml 闪烁液进行样品分析。图 2 给出的是,取 5ml 闪烁液,10ml 水样的计数率为 1,对其它体积样品的计数率进

表 1 氡气在几种有机溶剂及水中的溶解度

溶 剂	温度(℃)	0	5	10	15	20	25	30	35
二硫化碳		33.13	29.49	26.40	23.78	21.55	19.65	18.05	16.68
己 烷		23.71	21.05	18.77	16.81	15.13	13.69	12.45	11.39
乙 醚		20.23	18.53	17.11	15.92	14.94	14.11	13.43	12.86
甲 苯		18.26	16.55	15.02	13.66	12.43	11.34	10.36	9.48
丙 酮		7.76	7.23	6.77	6.38	6.04	5.75	5.55	5.29
乙 醇		8.02	7.40	6.86	6.38	5.95	5.57	5.23	4.92
水		0.51	0.40	0.34	0.29	0.25	0.22	0.20	0.18

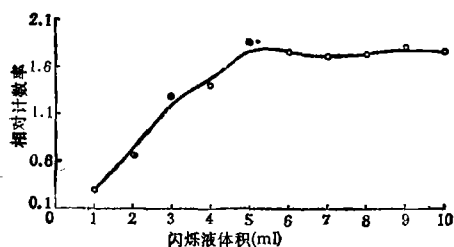


图1 闪烁液体积对测定结果的影响

行规一化处理后的结果。从图2可见,在总活度不变的情况下,水样体积对相对计数率几乎无影响。对于实际采集的样品,总活度和体积成正比,因此,为提高灵敏度,可使用稍大些体积的水样。我们考虑操作的方便,取10ml水进行分析。

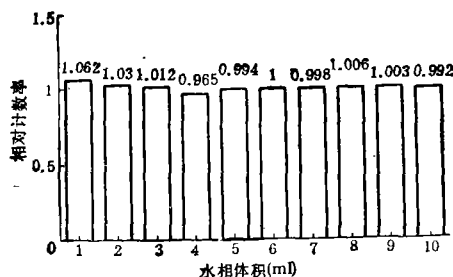


图2 水相体积对测定结果的影响

a. 1.062 b. 1.03 c. 1.012 d. 0.965 e. 1.0 f. 0.998
g. 1.006 h. 1.003 i. 0.992

(四) 闪烁杯不加盖时间的影响

取10个闪烁杯,各加入5ml闪烁液,然后向每个杯中加入10ml含 Rn 水样。除第一个杯加入水样后立即加盖密封外,其余各杯均放置不同时间后再加盖密封,观察闪烁杯不加盖时间长短对测定结果的影响。结果绘于图3中。从图中可见,加入水样后不加盖放置9min,

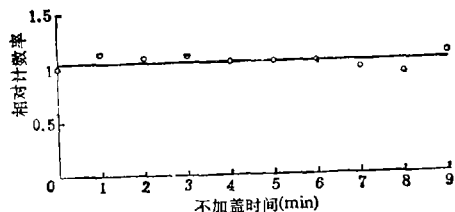


图3 不加盖时间对测定结果的影响

在此期间逸出的氡气不大于10%。在实际采

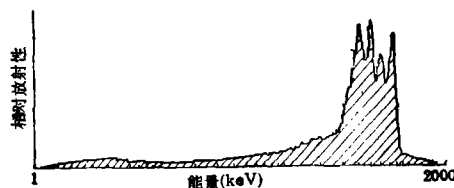
样过程中,水样加入闪烁杯内数秒钟后即可完成加盖密封操作,所以,不必考虑在加盖密封前氡气逸出对测定结果的影响。

(五) 闪烁杯的密封性

样品采集并密封在闪烁杯内后,至少要放置3h,等待氡气与其短寿命子体达到放射性平衡后才进行测量。有时,由于某些原因,样品测量可能要耽搁数小时乃至数日。因此,如果闪烁杯盖的密封性不良,就有可能导致氡气泄漏,使测定结果偏低。我们检查了三种厂牌的玻璃质闪烁杯,发现对于所有检查过的闪烁杯,氡气都有不同程度的泄漏,表明这类闪烁杯的盖不适于密封氡^[6]。经在盖内加入一层橡胶(自行车内胎,厚1mm)和一层0.01mm厚铝化聚脂膜,取代原来的内衫物或聚乙烯垫后,基本上解决了这一问题。笔者建议在开始使用一种新厂牌的闪烁杯测量氡气时,应检查其密封性;如密封性不良,就要采取补救措施,以保证分析结果的可靠性。

(六) 测量参数

TR1-CARB 2250CA 液体闪烁分析仪是先进的液闪谱测量装置。它内置有自我规模化、校准和性能评价系统,保证了长期工作的可靠性和稳定性^[7]。它有正常计数和低水平计数两种工作模式。为降低本底,提高测量灵敏度,我们使用低水平计数模式测量氡气。按上述选择的实验条件,获取并贮存氡气和本底样品的全谱(0—2000keV),然后用该仪器专用的Spectragraph谱分析软件包,对两个谱进行处理。按样品计数率 C 和本底计数率 B 的最大比值 $\left(\frac{C^2}{B}\right)$ 为标准,自动找出的最佳计数区为23 | 1350keV。在这一计数区,对氡的探测效率为55%左右,本底大约为6cpm。图4是以对数

图4 ^{222}Rn 液体闪烁测量能谱

坐标形式描划出来的典型氡能谱图。

三、分析程序

按上述选择的实验条件, 制定出如下分析程序: ①向经过洗液浸泡并冲洗干净的闪烁杯内加入 5ml 闪烁液, 加盖密封待用; 闪烁杯内壁一定要洁净, 否则在加样摇动后, 闪烁液滴容易粘敷在杯壁周围, 影响探测效率。②用干净的 10ml 注射器抽取直接由水源处流出或泵出的新鲜水样 10ml; 注意不要吸入气泡, 注射器内不留空腔。③立刻将注射器针头插入闪烁杯底部的闪烁液中, 把水样注入杯内, 加盖密封, 摇动约 1min。④密封至少 3 h 后, 在 TR1-CARB 2250CA 液体闪烁分析仪上按预选条件测量 1 h。⑤按下式计算出采样时水中的氡气浓度 A。

$$A(\text{Bq/L}) = \frac{C - B}{F \times V \times e^{-\lambda t}}$$

式中, C 为 23~1350keV 计数区内的样品总计数率 (cpm), B 为同一区内的本底计数率 (cpm), F 为仪器刻度系数 (cpm/Bq), $e^{-\lambda t}$ 为从采样至测量期间, ^{222}Rn 的衰变校正因子, V 为采样体积 (L)。

四、小 结

用液体闪烁法测量水中氡的浓度, 不仅具

有样品制备简单, 需要的样品量少, 以及能实现测量的自动化等优点, 而且在准确度、精密度和灵敏度方面也很令人满意。

笔者曾用经典的闪烁室法和液体闪烁法同时测定一含氡水样中的氡浓度。两种方法测定结果仅约相差 3%。亦检查了液体闪烁法测氡的重复性, 对同一样品测定 16 次的变异系数为 2.2%。本实验室测量装置对氡的刻度系数为 164.3cpm/Bq。本底测量每次 1 h, 16 次的平均值及标准差为 $6.24 \pm 0.41\text{cpm}$ 。取 $\alpha = \beta = 0.05$, 按文献 [8] 方法计算出的探测限为 0.82 Bq/L。调查资料^[9]表明, 绝大多数地下水中的氡浓度都高于此值。用此法测定了天津市地下水中的氡浓度, 取得满意的结果。

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Abstracts

Chinese Journal of Environmental Science

neous Desulfurization and Denitrification of Wet Flue Gas. Dixin Shen (Research Center for Eco-Environmental Sciences, Academia Sinica): *Chin. J. Environ. Sci.*, 13(2), 1992, pp. 47—56

A number of processes currently undergoing development for simultaneous removal of SO_2 and NO_x are based on either the oxidation of relatively insoluble NO to more soluble NO_2 or the employment of a water-soluble ferrous-chelating compound, which can react with NO to form complexes, as catalyst to aid in the absorption of the insoluble NO. Or, NO_x in solution can be reduced by the absorbed SO_2 to form molecular N_2 , N_2O , or reduced nitrogen compounds such as $\text{NOH}(\text{SO}_3^-)_2$, NH_4SO_3^- and NH_4^+ , while SO_2 is oxidized to sulfate. The kinetics and mechanisms of the reactions involved in these processes, the formation and disappearance of reaction intermediates in the processes are discussed.

Key Words desulfurization, denitrification, ferrous chelating reagents

The Application of Delphi Rule in the Study of Strategic Countermeasures in Provincial and District Environmental Programme and Analysis of Its Outcome. Cao Lei (Provincial Bureau of Environment Protection of Gansu): *Chin. J. Environ. Sci.* 13(2), 1992, pp.56—59

A draft programme for the provincial environmental protection of Gansu was formulated by using the Delphi Rule. Nearly one hundred experts and environment management officials were involved in the programme drafting process in which eleven categories of problems, including environmental problems in the province, the relative weights of the constituting factors of the macroscopic territorial environmental problems, the focal points of environmental protection strategy, the feasibility and weight of effectiveness of environmental protection control approaches, the relative importance of strategic countermeasures and steps of the environmental programme, and the proportion and direction of investment in environmental protection etc. in the next 5, 10 and 30 years, were integrately evaluated and predicted. More than 2000 fundamental data were obtained during the process. The data underwent statistical treatment and computer aided auxiliary judgement and a series of principal conclusions concerning the state of probability distribution were thus obtained. This provides the scientific basis for the strategic decision making process of Gansu provincial environmental programme.

Key Words: Delphi Rule, environmental programme, environmental protection.

Progress and Outlook on Removing Heavy Metals from Waste Water by Ferrite Process. Ding Ming, Zeng Huanxing (Department of Materials Science and Engineering, The University of Science and Technology of China): *Chin. J. Environ. Sci.*, 13(2), 1992, pp.59—67

Theoretical researches on the mechanism and condition of the formation of ferrite sediments, development and practical application of technological process and present situation of comprehensive utilization of ferrite sediments in recent years were reviewed. It was indicated that carrying out profound studies on formation mechanism of ferrites and composition, structure and property of ferrite sediments will promote the application of the process in treatment of heavy metal containing waste water.

Key Words: ferrite, waste water treatment, heavy metals.

Estimation of Losses Caused by Water Pollution in the Taihu Area of Jiangsu Province. Ge Jiqi (Nanjing Agricultural University): *Chin. J. Environ. Sci.* 13(2), 1992, pp.68—72

This paper reports the methods and results of the estimation of economical losses by water pollution in the Taihu area of Jiangsu province. The losses totaled 1008 million yuan during the period from 1985 to 1988 which accounted for 3.12 percent of average GNP.

Key Words: water pollution losses, Taihu area of Jiangsu.

Rapid Determination of Benzo (a) Pyrene with Synchronous Fluorimetric Scanning in Defined Range of Dual-Wavelengths. Wei Xi Yao, Tao Xiong, Wenxian Cui, Xiaobai Xu (Research Center for Eco-Environmental Sciences, Chinese Academy of Sciences, Beijing): *Chin. J. Environ. Sci.*, 13(2), 1992, pp.73—76

A rapid method for the determination of BaP using the synchronous fluorescence spectroscopic scanning in defined range of dualwavelengths (SFDW) was developed. No pre-separation for the samples under analysis is needed. The method proved to be simple, rapid and sensitive. The minimum detection limit of BaP is about 5×10^{-10} g and the linear range is over three orders of magnitude. The average relative deviation is less than $\pm 20\%$. The results obtained with this method are comparable with those obtained with high performance liquid chromatography (HPLC).

Key Words: synchronous fluorimetry, Benzo (a) pyrene, Carcinogens.

Measurement of ^{222}Rn in Groundwater by Liquid Scintillation Method. Jiang Huixia, Zhao Yongcheng, Liu Qingfen (Institute of Radiation Medicine, Chinese Academy of Medical Sciences): *Chin. J. Environ. Sci.*, 13(2), 1992, pp.76—79

A liquid scintillation counting method for determining ^{222}Rn in groundwater is described in this paper. the method proved to be simple, rapid, sensitive and reliable. Only 10 mL of water sample is needed for the analysis. Sample preparation can be finished in less than one minute. the detection limit is about 0.82 BqL^{-1} . Sample counting can be done automatically. A commercial liquid scintillation counting system is effective enough for the procedure. It proved to be an ideal method for conducting large-scale survey on ^{222}Rn le-

vels in groundwater.

Key Words: liquid scintillation, ^{222}Rn , groundwater.

Determination of Trace Amount of Vanadium in Environmental Samples with a Kinetic Spectrophotometric Method. Wang Jianhua, Zhou Quanfa, Ruan Wenjiu (Department of Chemistry, Yantai Normal College): *Chin. J. Environ. Sci.*, 13(2), 1992, pp 80—83

A novel kinetic spectrophotometric method for the determination of trace amount of vanadium was proposed based on the inductive effect of V(IV) on $\text{Cr}_2\text{O}_7^{2-}$ -I $^-$ -starch redox reaction. The optimal conditions for the determination were established as: $8.4 \times 10^{-4} \text{ mol L}^{-1}$ I $^-$, pH = 2.1 and the determination should be carried out at a wavelength of 585nm. The calibration graph was linear for vanadium (IV) concentration ranging from 0 to $6.6 \mu\text{g} \cdot \text{mL}^{-1}$ under optimal conditions mentioned above, and the detection limit was determined to be $0.02 \mu\text{gV(IV)} \cdot \text{mL}^{-1}$. The procedure is highly selective for V(IV) since most coexisted foreign ions have no interference on the determination except for Fe^{3+} , Fe^{2+} , Sn^{2+} and Ti^{3+} . Trace amount of vanadium waste in water and soil was determined and satisfactory were obtained. the recovery was observed to be 95.1—98.3%.

Key Words: vanadium, kinetic spectrophotometry.

Environmental Organisms Investigation and Environmental quality Assessment of Changjiang Water Intakes, the Second Water Source of Shanghai—Benthos, Fish and Residual Toxicity in Organisms. Zhao Lihua, Yao Gendi, Guo Luji, Sun Zhenzhong, Zheng Liexun, Jiang Xinpo, Zhang min (Shanghai Fisheries Research Institute), Yang Hequan (Shanghai Fisheries University): *Chin. J.*

Environ. Sci., 13(2), 1992, pp.84—88

The environmental quality of two water intakes in the Changjiang River, the second water source of Shanghai, was assessed by applying the phase-dividing method of saprobic system of benthos and fishes and measuring the remnant toxin contents of the system. Two sampling stations, Liuhe and Langgang, both located on the southern shore of Changjiang Estuary, were selected for study. Benthos investigations were conducted at the sections of the stations in low water (Feb. to March) and high water (July) periods of 1989. From April to July, species and quantity of fishes were sampled twice a month and study on index species was conducted. In addition, total concentrations of Hg, Cu, Zn, Cd, Pb, 666(BHC) and DDT in four benthos, i.e. *Cladophora* sp., *Spirogyra* sp., *Corbicula jiluminea* and *Sesarma deheni* were determined. Results of synthetic assessment showed that the two intakes under study could be classified as β -mesosaprobic Zone, within the area of clean zone. However, the environmental quality of the two water intakes indicated the complexity of the estuary environment.

Key Words: Changjiang water intake, environmental organism, assessment of water quality.

Formaldehyde Level of Indoor Air and Its Evaluation. Chen Yan, Huang Hongen, Sun Donghong (Henan Provincial Institute of Environmental Protection, Zhengzhou): *Chin. J. Environ. Sci.* 13(2), 1992, pp.89—91

During 1989, formaldehyde concentrations in indoor air were measured in apartments, offices, department stores, hotels and in wardrobes across the city of Zhengzhou. The results show that formaldehyde level of indoor air varied from 0.002 to 0.595 ppm with the mean values ranging from 0.012 to 0.147 ppm. Formaldehyde concentrations in wardrobes varied from 0.066 to 1.77 ppm changing with the age and type of wardrobes.

Key Words: indoor air pollution, formaldehyde.

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