

我国幅员辽阔,各地自然情况相差很大,环境样品的种类亦不尽相同。因此,在环境放射性监测中,应用统一的  $^{226}\text{Ra}$  与  $^{238}\text{U}$  比值的可能性受到限制。然而,各地放射监测部门,只要对多年积累的数据资料进行必要的补充和完善,便可以得出本地区各种样品  $^{226}\text{Ra}$  与  $^{238}\text{U}$  的比值。在正常情况下,应用该比值,可以由铀分析的结果推算得到样品中  $^{226}\text{Ra}$  的含量。例如,辽宁省内一送检的井水样品,欲作  $^{226}\text{Ra}$ 、 $^{238}\text{U}$  的测定。则只作 U 的测定,便可以给出两个核素的结果。经测定,该水样 U 含量为  $2.1 \mu\text{g} \cdot \text{L}^{-1}$ , 则样品中  $^{226}\text{Ra}$  含量  $A(\text{Bq} \cdot \text{L}^{-1})$  可计算得到:

$$A = 2.1 \times 0.0122 \times 0.45 = 1.15 \times 10^{-2}$$

式中, 0.45 为辽宁井水中  $^{226}\text{Ra}$  与  $^{238}\text{U}$  的比值。

由此计算得到的该水样中  $^{226}\text{Ra}$  含量的最大相对误差为 82%, 其值范围在  $(0.2-2.1) \cdot 10^{-2} \text{Bq} \cdot \text{L}^{-1}$ , 低于饮水中  $^{226}\text{Ra}$  限量标准  $1.1 \text{Bq} \cdot \text{L}^{-1}$  近百倍。因此, 对于执行

相应的标准而言是可行的。

通过上面的论述可以确信, 样品中  $^{226}\text{Ra}$  与  $^{238}\text{U}$  比值在环境放射性监测工作中具有广阔的前景。但这一比值的确定必须在进行了较大量的调查研究之后。随意借用其它地区某种样品的比值来推算当地样品中  $^{226}\text{Ra}$  含量的做法是不足取的。

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## GC-ECD 检测水中有机氯和有机磷农残研究

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**摘要** 本文研究 GC-ECD 同时检测水体中十二个有机氯农药和有机磷农药组分的最佳测试条件。在选定的测试条件下, 方法检测下限达  $0.2 \text{ng/L}$  ( $\alpha$ -666) 和  $0.8 \text{ng/L}$  (甲基-1605), 线性范围均达  $10^3$  数量级, 水样加标回收率在 82.5—100% 之间。

**关键词:** GC-ECD; 水; 有机氯和有机磷农药。

环境样品中有机农药残留量测定已有许多报道<sup>[1-3]</sup>。有机氯农药和有机磷农药需分别用气相色谱法进行测定, 而且测试时间较长。本工作旨在建立一种高灵敏、高选择性、快速、简便的测试方法, 以适应水中有机农药残留量的检测。笔者详细研究了填充柱色谱分离, 电子捕获检测器 (ECD) 同时测定水

体中  $\alpha$ -666、 $\beta$ -666、 $\gamma$ -666、 $\delta$ -666、P、P'-DDD、P、P'-DDE、O、P'-DDT、P、P'-DDT、乐果、马拉硫磷、甲基-1605 和 1605 的测试条件, 探讨了这十二种农药组分的电子捕获机理, 表明所检测的农药组分电子捕获机理均属离解型化合物机理, 验证了使用单点外标法定量的可行性。测试方法的检测下限达

0.2 ng/L( $\alpha$ -666) 和 0.8 ng/L (甲基-1605), 线性范围均达  $10^3$  数量级, 水样加标回收率在 82.5—100% 之间。

## 一、实 验

### 1. 仪器和试剂

103 型气相色谱仪, 电子捕获检测器, 放射源为  $Ni^{63}$  (上海分析仪器厂)。

高纯氮气, 石油醚 (60—90°C AR), 其它试剂为分析纯, 有机溶剂均经提纯<sup>[1]</sup>。农药组分为农药标准品。

农药标准溶液: 以苯为溶剂, 分别配制 成 1 mg/ml 贮存液。最后工作用混合标准

溶液用石油醚稀释。

色谱柱为长 2m、内径 3mm 的玻璃柱, 填充后经 240°C 老化 24h。

### 2. 色谱柱的选择

为了能同时分离所检测的有机农药, 根据文献 [1] 和 [2], 试验了多种色谱柱, 结果表明: 能分离有机氯和有机磷农药组分的色谱柱为 OV-17 和 OV-210 混合柱。1.5% OV-17 + 2% OV-210/Chromosorb-WAW-DMCS-HP (80—100 目) 填充柱 (a 柱) 能满意地分离这十二个农药组分, 1% OV-17 + 3.4% OV-210/Chromosorb-WAW-DMCS-HP (80—100 目) 填充柱 (b 柱) 不能分离马

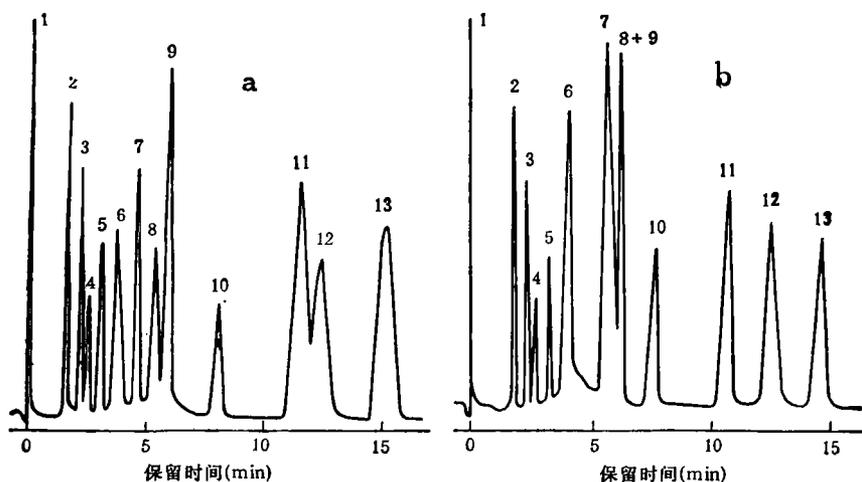


图 1 色谱流出曲线

a. 1.5% OV-17 + 2% OV-210, b. 1% OV-17 + 3.4% OV-210,  $T_{柱}$ : 200°C ECD. 1 溶剂; 2  $\alpha$ -666; 3  $\gamma$ -666; 4  $\beta$ -666; 5  $\delta$ -666; 6 乐果; 7 甲基-1605; 8 马拉硫磷; 9 1605; 10 P,P'-DDE; 11 O,P'-DDT; 12 P,P'-DDD; 13 P,P'-DDT.

拉硫磷和 1605, 这两支色谱柱的色谱流出曲线见图 1. 由图可见, a 柱对有机氯和有机磷农药组分的分离较好, 若是仅分离有机氯农药则 b 柱更佳。本实验选用 a 柱为有机氯农药和有机磷农药联合测定的色谱分离柱。

### 3. 分离条件的选择

分离条件试验表明, 随着柱温升高至 210°C 后,  $\gamma$ -666 和  $\beta$ -666 两峰部分重叠, 载气流量增至 100 ml/min 以后基流趋于稳

定, 达到饱和, 峰值急剧下降。ECD 脉冲间隔的选择, 以 150  $\mu$ s 的灵敏度最高, 基流最大或线性范围较宽的是 5  $\mu$ s。为了兼顾快速、灵敏和适宜的线性范围, 选择柱温为 200°C, 载气流量为 80 ml/min, 脉冲间隔为 50  $\mu$ s 的最佳测试条件。

### 4. 检测器温度对响应值的影响

ECD 的温度影响到基流和组分的电子吸收系数, 从而决定着检测器的灵敏度和线

表 1 方法测试性能参数

农药组分	$\alpha$ -666	$\beta$ -666	$\gamma$ -666	$\delta$ -666	乐果	甲基-1605
检测下限(ng/L)	0.2	0.6	0.2	0.3	15	0.8
线性范围	$3.2 \times 10^3$	$1.3 \times 10^3$	$2.6 \times 10^3$	$1.9 \times 10^3$	$2.6 \times 10^3$	$2.6 \times 10^3$
相关系数	0.9998	0.9999	0.9998	0.9997	0.9998	0.9998
农药组分	马拉硫磷	1605	P,P'-DDD	P,P'-DDE	O,P'-DDT	P,P'-DDT
检测下限 (ng/L)	11	1.1	1.1	1.1	1.5	1.8
线性范围	$1.9 \times 10^3$	$3.8 \times 10^3$	$1.3 \times 10^3$	$1.8 \times 10^3$	$2.6 \times 10^3$	$1.2 \times 10^3$
相关系数	0.9998	0.9994	0.9992	0.9999	0.9992	0.9999

表 2 加标回收试验

农药组分	样品含量 ( $\mu\text{g}$ )	加入量 ( $\mu\text{g}$ )	测定值 ( $\mu\text{g}$ )	回收率 (%)	加入量 ( $\mu\text{g}$ )	测定值 ( $\mu\text{g}$ )	回收率 (%)
$\alpha$ -666	0.0016	0.0010	0.00255	95.0	0.10	0.09830	96.7
$\beta$ -666	0.0010	0.0010	0.00200	100.0	0.10	0.09900	98.0
$\gamma$ -666	0.0012	0.0010	0.00216	96.0	0.10	0.09900	97.8
$\delta$ -666	0.0010	0.0010	0.00195	95.0	0.10	0.09870	97.7
乐果	0.020	0.020	0.0365	82.5	2.0	1.8620	92.1
甲基-1605	0.0015	0.0020	0.00341	95.5	0.20	0.19390	96.2
马拉硫磷	0.019	0.020	0.0379	94.5	2.0	1.9131	94.7
1605	0.0020	0.0020	0.00390	95.0	0.20	0.19400	96.0
P,P'-DDD	0.0015	0.0020	0.00335	92.5	0.20	0.19415	96.3
P,P'-DDE	0.0013	0.0020	0.00314	92.0	0.20	0.19312	95.9
O,P'-DDT	0.0023	0.0020	0.00423	96.5	0.20	0.19516	96.4
P,P'-DDT	0.0020	0.0020	0.00391	95.5	0.20	0.19632	97.2

性范围。试验结果见图 2, 随着 ECD 温度  $T_D$  升高, 电子与正离子复合的几率增大, 从而使基流  $I_b$  降低。

$T_D$  对组分响应值的影响与电子捕获的机理有关, 电子捕获的机理一般分为离解型和非离解型两类<sup>[4]</sup>。电子吸收系数与检测器温度和响应值间存在下列关系,

$$\ln K \cdot T_D^{3/2} = \ln \frac{k_{N_1} + k_{R_1}}{k_{N_2} + k_{R_2}} + \ln A + \frac{E_A}{k \cdot T_D} \quad (1)$$

$$K = \frac{C_2 \cdot F}{M \cdot Z} \cdot \frac{(I_b - I_e) \cdot \Delta x}{I_e} \quad (2)$$

式中,  $K$  为电子吸收系数;  $k_{N_1}$ 、 $k_{N_2}$ 、 $k_{R_1}$ 、 $k_{R_2}$  为电子捕获的反应速度常数,  $A$  为常数;  $E_A$  为电子亲合势;  $k$  为波尔兹曼常数;  $I_b$  为基

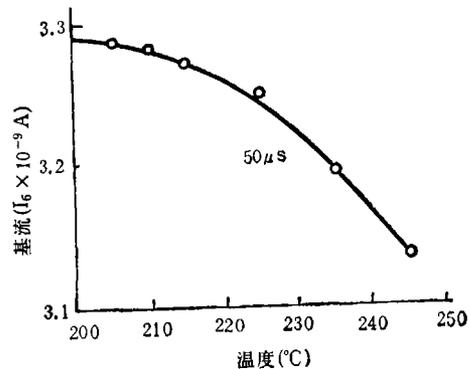


图 2 检测器温度对基流的影响

流;  $I_e$  为讯号流;  $C_2$  为纸速倒数;  $F$  为载气流量;  $M$  为试液的摩尔浓度;  $Z$  为进样量。

以  $\ln K T_D^{3/2} - \frac{1}{T_D}$  作图, 电子捕获机理不

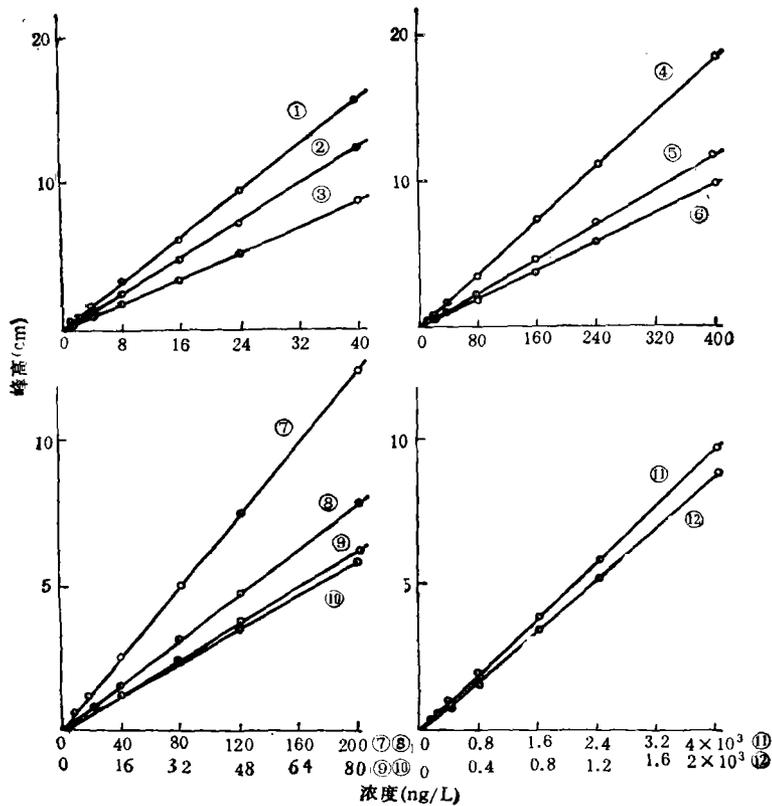


图 3 工作曲线

① $\alpha$ -666, ② $\gamma$ -666, ③ $\delta$ -666; ④1605 ⑤O, P'-DDT, ⑥P, P'-DDT ⑦甲基-1605, ⑧P'-DDD, ⑨ $\beta$ -666, ⑩P, P'-DDE, ⑪乐果, ⑫马拉硫磷。

同图形也不同,遵守离解型机理具有负斜率,具有正斜率则遵守非离解型机理。对所检测的有机农药进行了测定,按(2)式求得 $K$ 值,作出 $\ln KT_D^{1/2} - \frac{1}{T_D}$ 图。结果表明,这十二个农药

组分均呈负斜率变化,因此,各农药组分皆遵守离解型化合物机理捕获电子,它们的 $K$ 值随 $T_D$ 的升高而增大,即较高的检测器温度就能获得较高的灵敏度。但是,检测器温度升高造成基流下降,使测定的线性范围变窄,为了同时获得较高的灵敏度和较宽的线性范围,实验选择的检测器温为 $230^\circ\text{C}$ 。

#### 5. 提取条件的选择

分别使用石油醚、乙醚、正己烷、苯等溶剂进行提取试验。结果表明,石油醚和苯对

乐果提取率略低,其它溶剂无显著差异。考虑到溶剂的经济和毒性因素,选用石油醚作提取溶剂。提取时,500 ml 水样中加入 18 g 无水硫酸钠,50 ml 石油醚分两次,每次 3 min 萃取,污水样品提取后采用磺化法净化,提取液经无水硫酸钠干燥后,浓缩至 1 ml,供色谱分析用,进样量 $5 \mu\text{l}$ 。

#### 6. 测试性能参数

为了考察本方法的精密性、灵敏度和线性范围,分别进行了精密性试验和回收试验。精密性试验表明,8 次平行测定的变异系数均小于 $\pm 2.80\%$ 。最低检测下限达 ng/L 级,线性范围达 $10^3$ 数量级,其结果见表 1,工作曲线结果见图 3,图表中均测量峰高。图 3 中组分的工作曲线均过原点,在小时内考

了单点校正。试验表明,各单点峰高与工作曲线上各点的峰高的偏差小于 1.5 倍标准偏差。因此,在样品测试过程中,可以采用单点外标法进行定量分析,不必作工作曲线,只要在测定中,使用与样品含量接近的标准样品穿插进样,就能消除一些误差。

### 7. 回收试验

为了解水质基体对测定结果的影响和方法的准确度,将几个地下水、民用井水和地表水样品混合,进行了加标回收试验,8 次平行试验的结果见表 2。

## 二、小 结

通过实验,实现了 ECD 同时测定有机氯农药和有机磷农药,建立了简便、快速、灵敏、

准确的水体中 ng/L 级农药残留量测定方法。研究了电子捕获检测器检测农药的机理,提供了检测有机磷农药的一种高灵敏检测器——电子捕获检测器。

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## · 环境信息 ·

### 美国新建的环境物质致癌性资料数据库

环境物质致癌性资料数据库(The Carcinogenicity Information Database of Environmental Substances, CIDES) 对于约 1000 种有关环境或卫生的化学品从公开的文献中编辑出有数据的资料。它存储有 40000 条记录,精选自各种关于致癌性和诱变性研究的 5600 多种参考文献。该数据库也列出各种物质在环境中迁移和滞留的化学特性。CIDES

是纽约技术数据库服务处在国家环境卫生科学研究所的支持下开发出来的,用户可按下列地址和人员进行联系: Wendy Stern Fenwick, Technical Database Services, 10 Columbus Circle, Suite 2300, New York, NY 10019. U. S. A.

仲民摘译自 *ES&T*, 24 (6), 766 (1990)

### 无污染的清洗新方法

美国加州南埃尔蒙特的加尼福尼亚化学公司研究出一种新的化学清洗方法,可以清除在制药、生物技术、食品和饮料等工业生产中高纯度技术设备内的污染物。据该公司称,这种方法与现有的各种方法相比,更有利于环境安全。此方法称为 SCRCP,即特性螯合,还原和钝化(Specific chelating, reduction and passivation),可用于清洗输送水、空气、氮气、氧气和其它物质的设备中导管、管道、储罐、容器、阀门和设备配件。依据美国材料试验学会(ASTM)和美国机械工程师学会(ASME)制订的

现行清洗标准,对于清洗上述器件,指定要使用无机酸,但无机酸在清洗过程中会溶出有毒的重金属而污染高纯度设备,同时无机酸也很难清除净。SCRCP 方法使用的是无毒的有机酸(如柠檬酸)和螯合剂,它们不会溶出重金属,这样就可以在污水管道设备中清除掉溶液。目前加利福尼亚化学公司正力图修改 ASTM 和 ASME 制订的清洗标准,使之与他们的体系相融合,而成为使用无机酸的代用品。

申南竹摘译自 *Chemical & Engineering News*, 68 (7), 23 (1990).

Using the entrapped immobilized cells for treating wastewater is a new technique of biological treatment, but the entrapping agents are so expensive that application of them will be limited. The authors have developed polyvinyl alcohol (PVA) as an entrapping agent with PVA-boric acid method, and have prepared porous PVA-pellets for treating detergent wastewater. The results show that the PVA-pellets have high mechanical stability, good preservative ability, good recovery of activity, nearly no sludge produced, and viability of entrapped cells.

**Key Words:** entrapped immobilized microbial cells, polyvinyl alcohol.

#### Determination of Chromium in Wastewater with Nafion Exchange Preconcentration. Flameless Atomic Absorption Spectrometry.

Xu Tongming Shen Huakui Xu Xiaohong Xu Bo xing (Department of Chemistry East China Normal University): *Chin. J. Environ. Sci.*, **11**(5), 1990 pp. 45—48

Nafion is a new type cation exchange polymer. In this experiment coiled tungsten supporter was modified with a thin film of Nafion. Trace amount of Cr (III) in wastewater was exchangeable and could be preconcentrated by the modified supporter. Put it into a graphite cup to take atomic absorption spectrometric measurement. Cr (VI) was reduced to Cr (III) by  $\text{NH}_2\text{OH}\cdot\text{HCl}$ . A satisfactory result was obtained from the exchangeable preconcentration of chemically modified supporter with high selectivity of AAS. This paper introduces determination of Cr (III) and Cr(VI) in wastewater respectively. A buffer solution of 0.01 mol  $\text{NaAc}\cdot\text{HAc}$  (PH=4.5) was well suitable for preconcentration. The linear range for determination of Cr(III) was 5—100 ng/ml Cr(III) and recoveries 92—106%. Ten parallel tests of determining a sample solution containing 25 ng/ml Cr(III) gave a relative standard deviation of 5.9%. The characteristic concentration was 2.4 ng/ml Cr (III). More than 10 foreign ions did not interfere the determination.

**Key Words:** Nafion cation exchange polymer, Cr (III), flameless atomic absorption spectrometry, wastewater.

#### Volumetric Method for Silicon Determination in Certified Reference Silicates—Potassium Silicofluoride Volumetry.

Zhang Zhigang, Yang Shuzhen, Huang Yongfen(Shanghai Institute of Ceramics, Academia Sinica): *Chin. J. Environ. Sci.*, **11**(5), 1990, pp. 48—52

The optimal experimental conditions for determining silicon by using the volumetric method of potassium silicofluoride were presented in this paper. Different types of silicate materials such as clay, glass, refractories, silicate minerals, domestic certified samples and SRM of USNBS are also determined with this method, for which the authors participated the determination of the certified values of silicon in ERM (environmental reference material of soil and pond sediments. After comparing this method with others, it was found that the analytical results of the former one were accurate as those by the classical methods, and its RSD (relative standard deviation) was 0.09—0.17%. So the volumetric method for potassium silicofluoride can be recommended as an arbitrary method or one for determining the certified value of SRM.

**Key Words:** silicon determination, potassium silicofluoride volumetry

#### Determining the Ratio of $^{226}\text{Ra}$ to $^{238}\text{U}$ in Samples and Its Application in Environmental Monitoring.

Ma Junjie, Han Shouling (Liaoning Provincial Institute of Labour Hygiene, Shengyang): *Chin. J. Environ. Sci.*, **11**(5), 1990, pp. 53—56

This paper describes the ratio of  $^{226}\text{Ra}$  to  $^{238}\text{U}$  in 19 kinds of environmental samples in Liaoning Province. The determined results showed that the ratios of  $^{226}\text{Ra}$  to  $^{238}\text{U}$  in grain and vegetable samples were between 2.6—9.7, mean value was 4.8, those in drinking water between 0.36—0.45, mean value 0.40, and those in soil, ore and phosphate fertilizer were between 0.86—1.10, mean value 0.98. The authors presented a discussion on application of the ratios in environmental monitoring.

**Key Words** radium-226, uranium-238, environmental monitoring.

#### Simultaneous Detection of Chlorinated and Organophosphorus Pesticides in Water with GC-ECD.

Chen Qinghuo(Henan Provincial General Station of Environmental Hydrogeology, Zhengzhou): *Chin. J. Environ. Sci.*, **11**(5), 1990, pp.56—60

A highly sensitive analytical system of separation and simultaneous detection for chlorinated and organophosphorus pesticides in water with GC-ECD has been presented. The compounds,  $\alpha$ -BHC,  $\beta$ -BHC,  $\gamma$ -BHC,  $\sigma$ -BHC, dimethoate, methylparathion, malathion, parathion, p,p'-DDD, p, p'-DDE, o, p'-DDT, p, p'-DDT were well separated in 1.5% OV-17 + 2% OV-210/Chromosorb-WAW-DMCS-HP (80—100 mesh) column, and their retention time was found to be below 15 minutes. It was

demonstrated that these pesticide compounds belonged to the compounds of the dissociative model of capturing electron. The minimum detection limits of this system are 0.2ng/L ( $\alpha$ -BHC) and 0.8ng/L (methylparathion). All the linear ranges are at the level of  $10^3$  times. The standard recovery is 82.5—100%.

**Key Words:** simultaneous detection, chlorinated pesticide, organophosphorus pesticide, GC-ECD.

### Environmental Planning for A River Basin Aided with Computer Graphic Simulation.

Chen Xun (Institute of Systems Science, Academia Sinica, Beijing); Fu Guowei (Dept. of Environmental Engineering, Tsinghua University, Beijing). *Chin. J. Environ. Sci.*, 11(5), 1990, pp. 61—65

Made in the present article is a discussion on the availability and advantages of computer graphic simulation applied to a complicate environmental planning for a river basin. Some external trends and techniques abroad have been introduced, and the conditions to realize the interactive computer graphic simulation on a computer also discussed briefly.

**Key Words:** environmental planning river basin, computer graphic simulation.

### Application of Quantitative Structure Activity Relationship (QSAR) in Environmental Chemistry.

Bai Naibin (Research Center for Eco-Environmental Sciences, Academia Sinica Beijing): *Chin. J. Environ. Sci.*, 11(5), 1990, pp. 66—70

This paper reviews some approaches of QSAR in environmental chemistry in the literature abroad during past ten years, and introduces the author's work concerned in some aspects. QSAR is indeed a useful means for assessment of chemicals. Meanwhile, the limitations of its application and the probable prospects are discussed as well.

**Key Words:** quantitative, structure, activity, relationship, chemistry.

### Polonium-210 in Tobacco and Lung Cancers.

Kou Rongchun (Hebei Provincial Institute of Radiation Hygiene, Shijiazhuang): *Chin. J. Environ. Sci.*, 11(5), 1990, pp. 70—74

Besides the carcinogenic compounds of aromatic hydrocarbons and mutagens, tobacco also contains several radioactive nuclides, among which  $^{210}\text{Po}$  can be inhaled

into human respiratory system, and causes the risk of lung cancers. This article introduces radioactive levels of  $^{210}\text{Po}$  in tobacco in China and other countries, the results of animal tests in which the animals that inhaled smog containing  $^{210}\text{Po}$  led to come on of lung cancers, and estimation of carcinogenesis caused by lower dose  $\alpha$  particle exposure.

**Key Words:** polonium-210, tobacco, lung cancer.

### A Review on the Researches of Offensive Odor and Odor Measurement with the Sense of Smell.

Yao Renyu (Beijing Municipal Research Institute of Machinery and Electricity): *Chin. J. Environ. Sci.*, 11(5), 1990, pp.74—78

This article introduces some research results on the formation, interaction and intensity of offensive odor and the research methodology. An effective odor-measuring method with the sense of smell, namely the triangular odor bag method has been described. In the method, odor index, choosing and training a panel, sampling and measuring procedure, criterions for controlling the sense, and the data treatment are described.

**Key Words:** offensive odor, odor measurement, sense of smell.

### A Survey on the Cause of Nitric Radical- ( $\text{NO}_2^-$ ) Pollution in Matan Water-Supply, Source Site of Lanzhou City.

Zhang Mingquan, Gao Hongxuan, Wu Kejian (Research division of Water Resources & the Environment, Lanzhou University): *Chin. J. Environ. Sci.*, 11(5), 1990, pp. 79—82

The data of ground water quality in Mutan, Lanzhou City, show that  $\text{NO}_2^-$  pollution is getting worse. It is found that a close relation exists between the pollution tendency and environmental deterioration in the water-supply-source site. By analysis, the essential cause of  $\text{NO}_2^-$  pollution in ground water is derived from fertilizing the farmland around with a huge amount of nitrogenous manure.

**Key Words:** nitric radical pollution, ground water, nitrogenous manure.