

桑色素荧光法测定铍的研究*

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摘要 报告铍与桑色素试剂的荧光光谱特性, 给出其激发光谱, 发射光谱和同步荧光光谱图, $\Delta\lambda$ 选用 100nm 的同步荧光光谱效果最佳。当 Be^{2+} 的浓度为 $0.005\mu\text{g}/\text{ml}$ 时仍可获得良好的同步荧光光谱, 对大气样品和水中痕量铍的测定结果与常规方法的结果有可比性。

关键词 铍, 桑色素, 荧光光度法。

环境中铍的污染, 会造成对人体健康的严重危害, 故对铍的灵敏测定方法的研究一直受到环境分析化学工作者的重视。铍能与许多有机试剂形成会发出荧光的络合物, 应用最为普遍也是应用最早的分析试剂是桑色素(2', 3, 4', 5, 7-五羟基黄酮)^[1]。60 年代初, C. W. Sill 等曾对用桑色素荧光法测定铍进行过较详细的研究^[2, 3]以后荧光光度仪器有了很大的进展, 但却一直未见有用桑色素荧光法测定铍的荧光特性报告。国内李民等报告了用此法测定水中微量铍^[4], 对分离方法做了仔细的研究, 但所用仪器为国产老式四用荧光计。笔者用现代荧光分光光度计对铍与桑色素试剂形成络合物的荧光光谱进行较系统的分析, 研究了该络合物的荧光光谱和同步荧光光谱, 并建立了测定环境介质中痕量铍的分析方法。

1 材料与方法

1.1 仪器与试剂

日立 MPF-4 型荧光分光光度计, 脉冲氙灯光源, 可做恒波长间隔的同步扫描, 能进行“能量型”和“比例型”的荧光光谱测定。

铍标准液: 用氧化铍配制 (BeO , 分析纯, 进口分装), 贮存液浓度为 $100\text{mg}/\text{L}$, 用时稀释, 溶液 pH 不得小于 3。

桑色素溶液: 进口分装试剂, 重结晶后使用, 0.02% 乙醇溶液, 贮存于棕色瓶中。

1% 碱性氯化亚锡溶液: 北京化工厂分析纯

试剂, 用前用 1mol NaOH 溶液临时配制, 10% EDTA 水溶液。

1.2 实验方法

同步荧光光谱的测定: 于 25ml 的容量瓶中加入待测处理好的溶液, 再加入 $1\text{ml } 10\%$ EDTA 溶液, $2.0\text{ml } 1\text{mol NaOH}$ 溶液, $0.1\text{ml } 1\%$ 氯化亚锡碱性溶液, 摇匀, 加入 0.3ml 的 0.02% 桑色素溶液, 摇匀, 用水移至刻度再摇匀取部分溶液至 1cm 的石英比色池中, 测定同步荧光光谱, 根据发射波长 520nm 处的峰高计算含量。

仪器测定条件: 激发和发射单色器波长分别固定在 350nm 和 450nm 处 ($\Delta\lambda = 100\text{nm}$), 在光电倍增管电压 780V 条件下, 选择适当的灵敏度, 狭缝宽度和扫描速度, 从发射波长 450nm 至 600nm 进行同步扫描, 记录光谱。

铍的原子吸收分光光度法测定: 滤膜用硫酸-灰化法处理^[5], 无火焰原子吸收分光光度法测定。美国 EP 公司 P-E503 型原子吸收分光光度计。波长 234.9nm , 灯电流 30mA , 狭缝 0.7mm , 断气清洗, 扣除氘灯本底, 干燥温度与时间为 $110^\circ\text{C}/30\text{s}$, 灰化温度与时间为 $900^\circ\text{C}/7\text{s}$ 原子化温度与时间为 $2600^\circ\text{C}/7\text{s}$ 。

天然水样的预处理: 按文献^[4]方法用硅胶柱富集和解析铍, 水样量一般需 $2500\text{—}500\text{ml}$ 。使用 waters 公司生产的 SEP-PAK 硅胶小柱, 亦

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可获得较好的效果。

2 结果与讨论

2.1 光谱特性

图 1 给出铍与桑色素试剂在碱性溶液中的激发光谱与发射光谱。其激发光谱的峰值在 430nm, 发射光谱的峰值在 530nm, 2 种光谱均为重叠单一的峰。

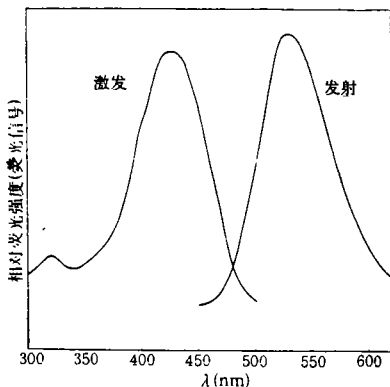


图 1 铍与桑色素试剂复合物的荧光光谱图(碱性溶液中)
铍浓度 0.008μg/ml, 激发波长由 λ_{ex} 300nm 起始, 狭缝带宽 6nm, λ_{em} 530nm, 狭缝带宽 10nm, 发射光谱由 λ_{em} 450nm 起始, 狭缝带宽 6nm, λ_{ex} 420nm, 狭缝带宽 10nm, 灵敏档 10×8

图 2 为不同浓度的铍与桑色素试剂的同步荧光光谱。同步荧光的信号强度 $I_{SL}(\lambda_{ex}, \lambda_{em})$, 可表示为:

$$I_{SL}(\lambda_{ex}, \lambda_{em}) = KcLE_x(\lambda_{ex})E_m(\lambda_{em}) \quad (1)$$

c 为待测物质的浓度, L 为试样溶液的厚度, $E_x(\lambda_{ex})$ 表示激发光谱, $E_m(\lambda_{em})$ 表示发射光谱, k 为实验的条件常数。由上式可见, 对于某种待测物质, 在实验条件相同的情况下, 同步荧光的信号强度与待测物质的浓度成正比, 故可用于定量测定。由图 2 可以看出铍的浓度与同步荧光强度有很好的线性关系。在图 2 的测定条件下(仪器灵敏档 10×1)铍的浓度应在 0.05μg 以下。在实际的测定中, 可根据所测样品的浓度范围选择适当的仪器灵敏档。在同步荧光中由于同时利用了复合物的吸收特性和发射特性, 提高了光谱的选择性, 使谱带宽度变小, 这可由图 1 和图 2 的对比中看出。

2.2 $\Delta\lambda$ 的选择

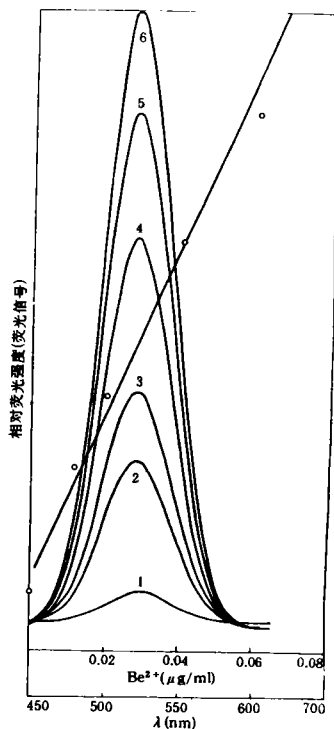


图 2 不同浓度的铍与桑色素试剂的同步荧光光谱
 $\Delta\lambda = 100\text{nm}$, 曲线 1—6 的铍浓度依次为 0, 0.012, 0.020, 0.040, 0.060 和 0.080(μg/ml)灵敏档 10×1

对于同步荧光光谱, 选择适当的 $\Delta\lambda$ 值十分重要。 $\Delta\lambda = \lambda_{em} - \lambda_{ex}$, 在同步扫描过程中它保持不变, 可表示为 λ_{em} 或 λ_{ex} 的函数, 代入式(1)中可表示为:

$$I_{SL} = KcLE_x(\lambda_{em} - \Delta\lambda)E_m(\lambda_{em}) \quad (2)$$

$$I_{SL} = KcLE_x(\lambda_{ex})E_m(\lambda_{ex} + \Delta\lambda) \quad (3)$$

因此 $\Delta\lambda$ 的值直接影响到同步荧光光谱的形状、带宽和信号强度。而 $\Delta\lambda$ 值主要通过实验进行选择。在 25ml 容量瓶中加入 0.2 μg 的铍, 0.3 ml 0.02% 桑色素和 2.5ml 的 1mol/L NaOH, 用蒸馏水加至刻度后, 分别用不同的 $\Delta\lambda$ 进行扫描, 结果如图 3 表示。由图中可以看出, 当 $\Delta\lambda = 100\text{nm}$ 时, 所得同步光谱的形状和强度最为理想, 故在我们的方法中选用的 $\Delta\lambda$ 为 100nm。

2.3 铍浓度对光谱特性的影响

对不同量的铍与 1mol 桑色素形成的复合物进行了光谱测定, 结果发现等 mol 的铍与桑色素形成复合物的荧光光谱同过量铍与桑色素形成

的光谱不同,两者的荧光强度并无显著差别,但

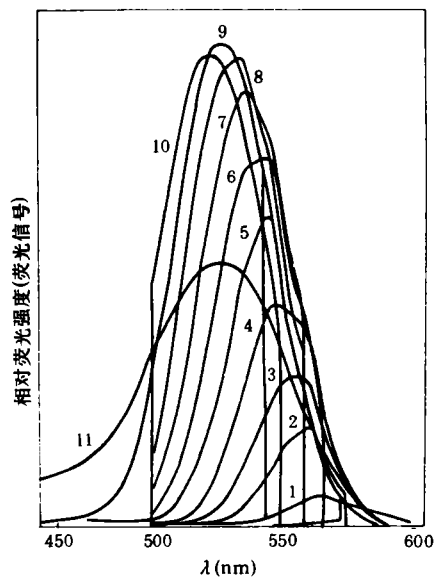


图 3 铍与桑色素试剂在碱性溶液中不同 $\Delta\lambda$ 的同步荧光光谱
曲线 1—11 的 $\Delta\lambda$ 依次为 20、30、40、50、60、70、80、90、100、110 和 150nm。铍 0.2 μ g/25ml,2.5ml,1mol/L NaOH,0.3ml
0.02%桑色素,起始 λ_{ex} 350nm, λ_{em} 450nm

过量铍形成复合物的光谱峰值紫移 15nm(图 4)。不论是对于荧光发射光谱还是同步荧光光谱均观察到同样的现象,其机理有待进一步讨论,在实际的测定中,在本法的测定条件下(25ml 体积,0.3ml 0.02%桑色素溶液)铍的量不得高于 1.8 μ g。图 4 为不同 mol 的铍与 1mol 桑色素形成

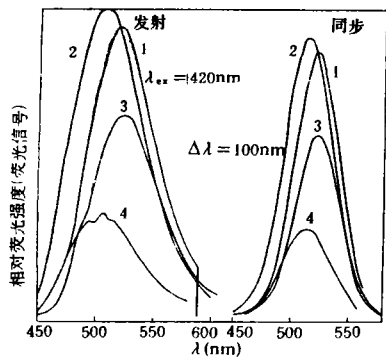


图 4 不同 mol 铍与 1mol 桑色素在碱性溶液中
形成复合物的荧光光谱图

左图为发射光谱,右图为同步光谱($\Delta\lambda=100\text{nm}$),
桑色素浓度均为 1mol,曲线 1、2、3、4 的铍浓度分别依次为
1、5、0.1 和 0 mol/L。

复合物的光谱图。可以看出当铍与桑色素的 mol 比大于 1 时,其光谱图与桑色素自身的光谱形状类似。而铍与桑色素的 mol 比值小于 1 时所获得的光谱图是本法定量测定的依据,说明在上述两种情况下形成复合物的性质不同。因为单纯的浓度增高所引起的内滤光效应难于解释光谱的紫移现象。

2.4 氢氧化钠和桑色素用量对测定的影响
在含有 0.2 μ g 铍的 25ml 测定溶液中,分别考察了不同量的 NaOH 溶液和桑色素试剂对同步荧光光谱的影响,增加 NaOH 的量反会使荧光强度降低,如表 1 所示。故在测定中选择 1mol/L NaOH 的用量为 2.0ml。

表 1 NaOH 用量对同步荧光强度影响

1mol/L NaOH(ml)	荧光强度 420nm(mm)
1.0	38
2.0	40
2.5	40
3.0	35
4.0	30
5.0	28

在测定过程中随着桑色素用量的加大,荧光强度逐渐增高,选择 0.3ml 0.02%的桑色素溶液用量,可完全满足测定浓度范围,而且空白的荧光强度也相当低。

通常对桑色素测铍时产生干扰的一些元素,用同步荧光光谱法测定时可完全消除。1000 倍过量的 Li^+ 、 La^{3+} 、 Th^{4+} 、 Y^{3+} 、 UO_2^{2+} 、 ZrO^{2+} 、 Cu^{2+} 、 Fe^{3+} 、 Cd^{2+} 、 Zn^{2+} 、对 0.10 μ g Be^{2+} 的测定均无影响。

2.5 大气中铍的测定

采好样的过氯乙烯样品滤膜用硫酸-灰化法处理^[5],样品滤膜在少量硫酸存在下,于 400℃ 加热使过氯乙烯滤膜灰化,加入少量 1mol 盐酸使残渣溶解,再在电热板上蒸至近干,然后在水浴上蒸干,用少许溶解并滤入 25ml 容量瓶中,按本文前述的实验方法进行铍的同步荧光测定,本法加标回收率为 85 $\pm$ 10%($n=8$)。对几个空气颗粒物样品用本法与无火焰原子吸收分光光度法进行了对照测定,结果如表 2 所示。所测 7 个

样品中有 5 个为焦炉顶样品, 2 个为环境空气样品, 两种方法测定结果偏差最大的为 25%。

表 2 2 种方法对空气颗粒物中铍的测定结果

样品编号	同步荧光法 ($\mu\text{g/g}$)	原子吸收法 ($\mu\text{g/g}$)	偏差 (%)
焦 IP-A1	1.3	1.1	+18.2
焦 IP-S1	3.0	3.4	-11.7
焦 IP-S2	3.4	3.3	+3.0
焦 TSP-1	5.0	4.6	+8.7
焦 F-3	1.0	0.80	+25
环 TSP-7	未检出	未检出	0
环 TSP-3	0.42	0.50	-5

2.6 天然水中铍的测定

使用李民等所建立的用硅胶柱富集铍与分离杂质^[4]后的样品, 用本法测定可获良好效果、灵敏度可提高 1 个数量级。对 n 个天然水样的测

定结果如表 3 所示。

表 3 2 种方法对天然水样的测定结果(Be^{2+} $\mu\text{g/L}$)

样品编号	水中 Be^{2+} 的含量($\mu\text{g/L}$)	
	同步荧光法	原子吸收法
T-1	未检出	未检出
S-3	0.08	0.05
S-5	0.20	0.16
S-6	0.02	未检出

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2.2.4 CO_2 浓度对冬小麦籽粒赖氨酸含量影响

700×10^{-6} 处理冬小麦籽粒赖氨酸含量比对照增加 11% 左右, 但当 CO_2 浓度由本底升高至 500×10^{-6} 时赖氨酸不仅没有增加反而降低 17%; $24\text{h} 700 \times 10^{-6}$ 处理比对照也呈减少趋势 (-4%)。3 种只有 1 种是呈增加趋势。7h 700×10^{-6} 处理之所以呈增加趋势, 可能是由于短时间高浓度的 CO_2 起了激化作用, 加快了冬小麦的生理生化过程, 有利于初生物质的积累。

上述分析中提到的 700×10^{-6} (7h) 处理蛋白质的增加可能也是这种原因。长时间高浓度冬小麦反而下降, 可能是因为冬小麦适应了这种环境激化作用, 不再起作用了。总的看来, CO_2 浓度升高对冬小麦赖氨酸含量的影响是复杂的, 还有些机理问题有待于今后进一步研究和探讨。

3 结论

(1) 在 7h 通气情况下, CO_2 浓度升高对大豆粗蛋白、粗脂肪含量的影响均为正效应。

(2) 在 7h 通气情况下, 大豆籽粒不饱和酸含

量随 CO_2 浓度升高而增加; 饱和酸呈相反的趨勢。

(3) CO_2 浓度升高对冬小麦籽粒含水量的影响不大。

(4) CO_2 浓度变化对冬小麦籽粒粗蛋白、赖氨酸的影响比较复杂, 短时间高 CO_2 浓度 (700×10^{-6}) 可能有一种生理生化的激化作用, 促进粗蛋白和赖氨酸的形成; 长时间处理激化作用消失, 对粗蛋白、赖氨酸形成不利。

(5) 从蛋白质、赖氨酸 2 项看, 当大气中 CO_2 含量增加 1 倍时, 对目前冬小麦品种的籽粒品质的影响是负效应。

以上的观点只是笔者根据近 2 年来试验结果分析所得, 关于 CO_2 浓度对作物生理生化作用的影响是很复杂的问题, 有很多问题还有待于进一步研究。

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waste gas, energy recovery could be made while the goal of pollution control being achieved.

Key words: catalyst, noble metal, organic waste gases.

Comparison in Vehicular Exhaust Emissions between Hong Kong and Guangzhou Cities. L. Y. Chen (Ph. D) and W. T. Hung (Civil and Structural Eng. Dept. Hong Kong Polytechnic, Hunghom, Hong Kong), Y. Qin (Institute of Environ. Sci., Zhongshan University, Guangzhou 510275); *Chin. J. Environ. Sci.*, **15**(5), 1994, pp. 56—60

The monitoring results of vehicular exhaust emissions in the urban areas of Hong Kong and Guangzhou were discussed. The monitoring exercise was carried out by using automatic gaseous analyser mounted on road side in Hong Kong for four years and for nine days in Guangzhou. The results show that the pollution level at Castle Peak Road of Hong Kong was comparatively lower than that in Guangzhou. The pollution level in Hong Kong satisfied the national air quality standard (Class 2) of China in both years 1988 and 1989. The pollutant concentration along Jiefang Middle Road was very high and exceeded the national air quality standard (Class 3). The traffic flow speed and composition were discovered to be the major reasons for the difference in pollutant concentrations in both cities.

Key words: vehicular exhaust emission, combined emission factor, traffic flow speed and composition.

SDS-PAGE Separation and HPLC-FID Identification of Selenoproteins in Soybean from Enshi Area Having a Higher Level of Selenium in Soil. Xie Shenmeng, Wang Zijian and Peng An (Research Center for Eco- Environmental Sciences, Chinese Academy of Sciences, Beijing 100085); *Chin. J. Environ. Sci.*, **15**(5), 1994, pp. 61—62

Selenoproteins in soybean from Enshi area where selenium in soil was at a higher level were separated by using sodium dodecyl sulfate-polyacrylamide gel electrophoresis (SDS-PAGE) and then identified by using high performance liquid chromatography with a fluorescent indication detector (HPLC-FID). Of 27 protein or protein subunit bands identified, 13 were found to be selenium species. According to a standard protein kit, their molecular weights were estimated at 58.3—60.3, 52.5—53.7, 46.8—50.1, 29.5—30.9, 28.8, 25.1—25.7, 24.3, 19.7—20.9, 18.4—18.6, 16.8—17.9, 16.1—16.2, 15.2—15.8 and 14.3—14.8 KDa, respectively.

Key words: SDS-PAGE, HPLC-FID, selenoprotein, speciation.

Acute Toxicity of Organotin Compounds to Benthos. Chen Tian'yi et al. (Dept. of Environ. Sci., Nankai University, Tianjin 300071); *Chin. J. Environ. Sci.*, **15**(5), 1994, pp. 63—64

The acute toxicities of three organotin compounds, i. e., monobutyltin (MBT), dibutyltin (DBT) and

tributyltin (TBT), to benthos (Chironomid larvae and tubificids) were reported. The results show that (1) the toxic effects of these compounds in a decreasing order were TBT > DBT > MBT; (2) the median lethal concentration (24h LC₅₀) of TBT to 4 species of benthos was 26.85 ppb for chironomid larvae (*Chironomus plumosus*), 241.55 ppb for chironomid larvae (*Chaetocladius sexpilosus*), 145.55 ppb for tubificids (*Branchiura sowerbyi*), and 355.63 ppb for tubificids (*Limnodrilus hoffmesteri*); (3) there was a strong negative correlation between the concentration of TBT and the median lethal time (LT₅₀) for *B. sowerbyi*.

Key words: organotin compounds, benthos, acute toxicity.

Effects of CO₂ on the Grain Compositions of Winter Wheat and Soybean. Gao Suhua and Wang Chun'yi (Chinese Academy of Meteorological Sciences, Beijing 100081); *Chin. J. Environ. Sci.*, **15**(5), 1994, pp. 65—66

Winter wheat and soybean crops were treated with different CO₂ concentrations in top-open chambers. The matured grains harvested from the crops were analysed for their compositions by using visible ultraviolet spectrometer, protein analyzer, gas chromatograph, YG-2 fat extractor and automanual nitrometer. The results show that an increased CO₂ concentration can have a positive effect on the contents of both rough protein and rough fat in soybean grain; and as the CO₂ concentration increases, the soybean grain would have an increased level of unsaturated acids and a decreased level of saturated acids. The change in CO₂ concentration had a more complicated effect on the levels of rough protein and lysine in winter wheat grain. In terms of both indicators of rough protein and lysine, a doubled concentration of atmospheric CO₂ had a negative effect on the quality of grains of the present varieties of winter wheat.

Key words: top-open chamber, CO₂ concentration, grain composition, winter wheat, soybean.

Study on the Fluorometric Determination of Beryllium Using Morin. Zhao Zhenhua et al. (Beijing Municipal Research Institute of Environmental Protection, Beijing 100037); *Chin. J. Environ. Sci.*, **15**(5), 1994, pp. 67—70

The fluorometric spectra of beryllium-morin complex were characterized and the graphs of excitation spectrum, emission spectrum and synchronous fluorescence spectrum were given. The synchronous spectrum of beryllium-morin in an alkaline solution had an optimum specific λ of 100nm. A synchronous scanning spectrofluorometry was used to determine beryllium at a level of submicrogram, with a detectable limit of 5 ng/ml Be²⁺. The results obtained with this method were comparable with those obtained with an atomic absorption spectrophotometric method for air samples and water samples.

Key words: beryllium, morin, fluorescence.

environmental quality classification.

Study on the Determination of Arsenic in Wastewater by Using Single Valve FIA combined with Hydride Generation Spectrophotometry. Liu Guoquan and Wang Chunxu (Dept. of Environ. Eng., Hebei Institute of Chemical Technology and Light Industry, Shijiazhuang 050018), He Yuaping (Shijiazhuang Chemical Fertilizer Factory, Shijiazhuang 050018); *Chin. J. Environ. Sci.*, **15** (5), 1994, pp. 71—72

A Single Valve FIA (Flow Injection Analysis) system with hydride generation spectrophotometric detection was designed for the determination of the trace arsenic in wastewater. This FIA system had no need for any carrying gas. The new hydride generator and the absorbing device made by the authors were used in the FIA system. The KBH_4 solution and the mixed silver nitrate-polyvinyl alcohol-ethanol solution were used for the generation and absorption, respectively, of arsenic hydride. The experimental results show that this method was very good for the determination of trace arsenic in wastewater. The FIA system had main advantages, including simple equipment, easy operation, high sensitivity, rapid determination (30 samples/h), good reproducibility ($R. S. D = 3.04\%$) and low detection limit ($1.82 \times 10^{-9} \text{ g/ml}$).

Key words: flow injection analysis, hydride generation, spectrophotometry, arsenic, wastewater.

Reversed-phase High Performance Liquid Chromatographic Determination of Methomyl in Soils. Chen Yanjun et al. (Jining Medical College, Jining 272113); *Chin. J. Environ. Sci.*, **15** (5), 1994, pp. 73—74

A reversed-phase HPLC method was described for determination of methomyl in soils. The detection limit of methomyl in soils was 0.1 μg , and the linear range was 1.0—20 $\mu\text{g/ml}$. The average recovery of methomyl as standard added to soils was in the range of 96.1%—100.2%. The relative standard deviations were below 5%. This method was simple with a higher sensitivity and a better accuracy.

Key words: methomyl, reversed-phase, HPLC, soil.

Study on Environmental Quality Classification Based on B-P Neural Network. Li Zuoyong (Chengdu Institute of Meteorology, Chengdu 610041); *Chin. J. Environ. Sci.*, **15** (5), 1994, pp. 75—77

Based on the back-propagation (B-P) algorithm of neural network, the classification models of water quality with 3 and 4 parameters were developed by taking the data on water quality indexes of 25 lakes as training samples. The verification of models for water quality classification of 6 lakes indicated that B-P neural network possessed not only good practicability, but also superiority over other classification methods of environmental quality because of its self-learning and self-adaptability.

Key words: neural network, B-P algorithm,

Qualitative Analysis on the Urban Vehicular Emission Pollution Model. Zhou Hongchang and Yang Peikun et al. (Dept. of Road & Traffic Eng., Tongji University, Shanghai 200092); *Chin. J. Environ. Sci.*, **15** (5), 1994, pp. 78—82

Based on the physical process of the vehicular emission dispersion in the urban street canyon, the monitored data of CO concentration near the urban traffic roads were analysed. The microscale near field dispersion model was particularly discussed, including the initial conditions, boundary conditions and the scope of the model as well as the problems that the model might deal with.

Key words: urban transportation, air pollution model, dispersion.

Current Status of Hazardous Waste Pollution and the Strategy for Its Control in China. Chen Liqui (Dept. of Science and Technology, National Environmental Protection Agency, Beijing 100035); *Chin. J. Environ. Sci.*, **15** (5), 1994, pp. 83—87

Hazardous waste pollutions in soils, waters, air and organisms are very serious in China, have caused negative effects and threats to human health in some parts of China, and have become a major environmental problem which urgently need to be solved at present. This paper deals with the current status of hazardous waste pollution in China, a comprehensive analysis and comparison on the pollution control technologies and management competencies for hazardous wastes in home and abroad, and the response strategies and recommendations on pollution control of hazardous wastes which are proposed according to the local conditions in China.

Key words: hazardous waste, pollution control, response strategy.

Development of Catalytic Oxidation Technology for the Treatment of Highly Concentrated and Refractory Organic Wastewater. Wen Donghui, Zhu Wanpeng (Dept. of Environ. Eng., Tsinghua Univ., Beijing 100084); *Chin. J. Environ. Sci.*, **15** (5), 1994, pp. 88—91

Four branches of the catalytic oxidation process have been formed, i. e., wet catalytic oxidation; photocatalytic oxidation; homogeneous catalytic oxidation and heterogeneous catalytic oxidation. Substantially, they all catalyze the decomposition of oxidant so as to speed up the chemical reactions between organic matters and oxidant in wastewater. Some powerful oxidants can produce much stronger radicals by catalysis to oxidate and decompose some highly concentrated and refractory organic matters. Therefore, catalytic oxidation becomes important as a new technology for the treatment of highly concentrated and refractory organic wastewater.

Key words: catalytic oxidation, organic wastewater, oxidants.