

监测分析

分光光度法测定三激励值表示水和废水的色度

张 莘 民*

(泰州市环境保护监测站)

色度是表示水质指标的重要内容,日、美等国采用分光光度法、三激励滤光法作为测定水和废水色度的标准方法^[1],均用测定三激励值的方法以主波长、色调、明度和纯度来表示水样的色度。铂钴法适于较清洁的水样(如地面水)测定,稀释倍数法则用于废水的色度测定^[2]。由于比视感度的差异使稀释倍数法存在较大的主观误差,而本法重现性好,适用范围广,能更精确地表达水和废水的色度。

一、实 验

(一) 原理

过滤水样的色度可用分光光度法测定水样的三激励值以 x, y, z 来表示,色调(hue)是以“主要波长”(dominant wavelength)表示,亮度(brightness)以明度(luminance)表示,饱和度(saturation)以纯度(Purity)表示。

(二) 仪器

1. 721 分光光度计 (上海第三分析仪器厂);
2. 过滤装置(见图 1);
3. pH S-2 型酸度计(上海第二分析仪器厂);
4. 离心机(3000 转/分)江阴市机械厂。

(三) 试剂

1. 0.05 mol 氢氧化钠溶液;
2. 0.05 mol 硫酸溶液;
3. 25mg/L K-RN 活性黄染料溶液;
4. 25 mg/L 3 × B 活性红染料溶液;
5. 25 mg/L 活性蓝染料溶液;
6. 硅藻土化学纯(助滤剂)。

(四) 操作方法

1. 水样预处理

取二份水样各 50ml,一份用原始的 pH 值,另一份用 H_2SO_4 或 NaOH 溶液调节 pH 至 7.60,离心除去悬浮物,加入 0.2g 助滤剂混合均匀后减压过滤,收集澄清的滤液。

2. 测定

按表 1 所列各个波长测定水样的透光率,并用去离子水作空白。

3. 计算

按表 1 将 X, Y, Z 行波长相应的透光率相加得到的总值,乘上适当的系数(10 个或 30 个座标)就得到 x, y, z 的三激励值(tristimulus)。 y 是明度百分比。

用下列公式计算三系数

$$x = \frac{X}{X + Y + Z},$$

* 泰州市职工大学环保专业 1987 届毕业生徐雨霖、尹必龙、陈玉琴参加了本实验工作,蔡彭骥老师也参加了部分工作。

表 1 用分光光度计测定色度时选择的纵座标

座标数	波 长 (mμ)		
	x X	y Y	z Z
1	424.4	465.9	414.1
2*	435.5*	489.5*	422.2*
3	443.9	500.4	426.3
4	452.1	508.7	429.4
5*	461.2*	515.2*	432.0*
6	474.0	520.6	434.3
7	531.2	525.4	436.5
8*	544.3*	529.8*	438.6*
9	552.9	533.9	440.6
10	558.7	537.7	442.5
11*	564.1*	541.4*	444.4*
12	568.9	544.9	446.3
13	573.2	548.4	448.2
14*	577.4*	551.8*	450.1*
15	581.3	555.1	452.1
16	585.0	558.5	454.0
17*	588.7*	561.9*	455.9*
18	592.9	565.3	457.9
19	596.0	568.9	459.9
20*	599.6*	572.5*	462.0*
21	603.3	576.4	464.1
22	607.0	580.4	466.3
23*	610.9*	584.8*	468.7*
24	615.0	589.6	471.4
25	619.4	594.8	474.3
26*	624.2*	600.8*	477.7*
27	629.8	607.7	481.8
28	636.6	616.1	487.2
29*	645.9*	627.3*	495.2*
30	663.0	647.4	511.2

用 30 个纵座标时的因数 0.03269 0.03333 0.03938

用 10 个纵座标时的因数 0.09806 0.10000 0.11814

* 往每一直行内插入相当于所列的波长的透光率(%)。如不需要特别准确,仅用带*号的10个纵座标。

表 2 各种主波长范围的色调

波长范围 (mμ)	色 调
400—465	紫
465—482	蓝
482—497	蓝绿
497—530	绿
530—575	绿黄
575—580	黄
580~587	黄橙
587~598	橙
598~620	橙红
620~700	红
400~530	蓝紫
530~700	红紫

$$y = \frac{Y}{X + Y + Z}$$

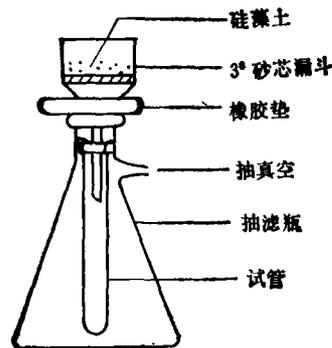


图 1 过滤系统

在图 2 中的一个色度图解上设置点(X、Y),并从图解上测定主波长和纯度(见图 2),根据表 2 所列范围,由主波长测定色调。

4. 结果表示

表 3 两种方法测定地面水、自来水色度的结果*

水 样	pH 值	分 光 光 度 法				铂钴比色法(度)
		主波长(mμ)	色 调	明度(%)	纯度(%)	
地面水	7.60	579	黄 —	98.9	0.6	15 0.00
		CV(%)		0.6	58.3	
自来水	7.60	579	黄 —	97.5	1.17	9.7 6.0
		CV(%)		0.6	5.0	

* 为三次重复测定结果的平均值。

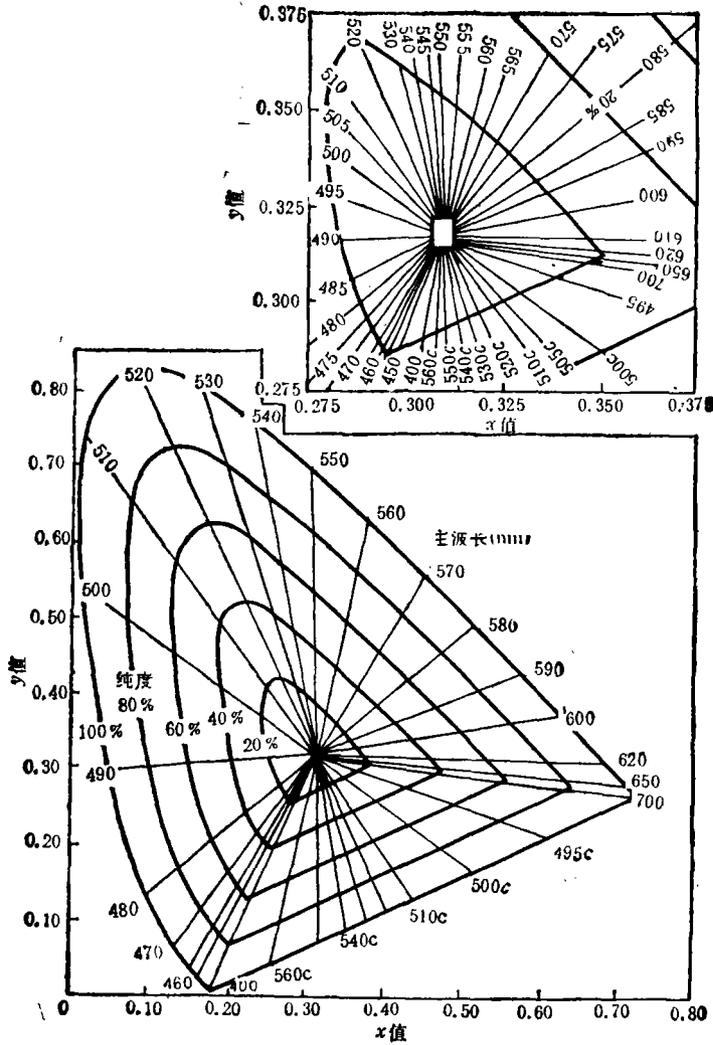


图2 色度图解(图中c为紫色轨迹)

表4 两种方法测定纯染料和染色废水色度的结果*(泰州市针织厂染色废水)

水 样	pH	分光光度法				稀释倍数法	
		主波长(mμ)	色 调	明度(%)	纯度(%)	倍 数	文字描述
染 色 废 水	7.60	483	蓝	52.2	40.2	1167	深蓝
		CV (%)		0.11	0.72	12.4	
活 性 黄	7.60	572	绿黄	90.4	27.4	389	深黄
		CV (%)		0.32	4.4	25	

* 同表3

表 5 两种方法测定不同浓度活性黄染料色度的结果

pH 值	浓度 mg/L	分光光度法				稀释倍数法	
		主波长 (mμ)	色调	明度(%)	纯度(%)	倍数	文字描述
7.60	25.0	572	绿黄	90.6	26.0	500	绿黄
	31.2	570	绿黄	90.4	35.0	500	绿黄

表 6 用 10 个和 30 个纵座标数测定的结果*(泰州市针织厂染色废水)

水 样	pH	纵座标数	主波长(mμ)	色 调	明度(%)	纯度(%)
染色废水	7.60	30	482	蓝 蓝	52.2	40.0
		10	482		51.8	40.0
		均值 相对偏差			52.0 0.28	40.0 0.00
1.95mg/L 活性黄染 料溶液	7.60	30	580	黄 黄	98.7	1.2
		10	580		98.8	1.2
		均值 相对偏差			98.8 0.00	1.2 0.00

* 同表 3

色度的特征以主波长(mμ),色调(如蓝、绿等),明度(百分比)和纯度(百分比)来表述。

(五) 不同方法测定水、废水和纯染料色度及 pH、浓度对色度的影响(见表 3、4、5 和图 3、4、5)。

二、讨 论

(一) 本法、铂钴法具有相同的精密度,因而本法也适用于测定地面水和饮用水的色度。

(二) 与稀释倍数法相比较,本法测定纯

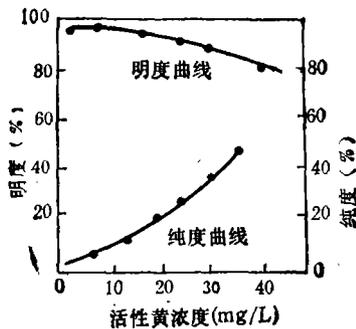


图 3 活性黄染料不同浓度的明度、纯度变化曲线

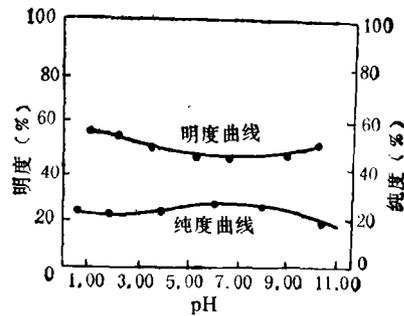


图 4 pH 值对活性黄染料色度的影响

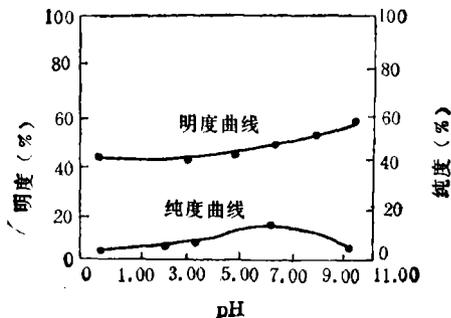


图 5 pH 对染色废水色度的影响

染料和染色废水的色度,具有较好的重现性。

(三) 随着 pH 值上升,色度呈现增加的趋势。

(四) 当活性黄染料浓度增加时,色度也随着增加。

(五) 通常用 10 个或 30 个波长测定结果相近。在要求不十分精确时,可以用 10 个波长测定。

三、结 论

本法测定三激励值来表示水和废水的色度,比铂钴法、稀释倍数法具有更多的优点,本法不仅精确、重现性好,而且适用范围广。

如何简化操作、缩短分析时间还有待于今后进一步探讨。

参 考 文 献

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 【2】 污染源统一监测分析方法编写组,污染源统一监测分析方法(废水部分),25 页,技术标准出版社,北京,1982 年。

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四氢呋喃比浊法测定土壤中的微量油

张家祥 张书香 周胜 荆云鹏

(山东建筑材料工业学院)

近代石油工业对环境的影响引起人们的重视。土壤被油污染导致农业减产亦已引起人们的关注^[1]。然而,迄今土壤中微量油的测定尚无令人满意的方法^[2]。已有的方法大多通过氯仿等有机溶剂抽提,而后用重量法或红外法等测定,我们研究用四氢呋喃抽提,然后用比浊法测定。由于四氢呋喃溶油能力强,可缩短抽提时间。又四氢呋喃与水可互溶,便于制成乳浊液^[3]。据此,我们拟定了一个土壤中微量油的测定方法,手续简便,重现性与灵敏度均有提高。方法适用下限为 10g 样品含油 0.025mg 以上。方法试用于小区域测定,结果令人满意。

实 验 方 法

(一) 仪器与试剂

721 分光光度计 上海第三分析仪器厂。

四氢呋喃 分析纯,西安化学试剂厂。

油标准溶液 用胜利油田原油配制成 1mg/ml 的四氢呋喃溶液。

标准含油土样 称取在 500℃ 灼烧 2 小时去油的土样 10.00g 置于 50ml 烧杯,加入一定量的油标准溶液,再加少量四氢呋喃润湿土壤,搅拌均匀,置阴凉处风干即成。

其他试剂均为分析纯。

(二) 标准曲线绘制

取干净的 25ml 比色管 7 只,依次加入油标准溶液 0.00, 0.08, 0.16, 0.24, 0.32, 0.40, 0.48mg 油,分别加不同量四氢呋喃,使各管中体积均为 3.0ml (标准油体积与所加四氢呋喃体积之和)。用 2mol/L 盐酸稀释至刻

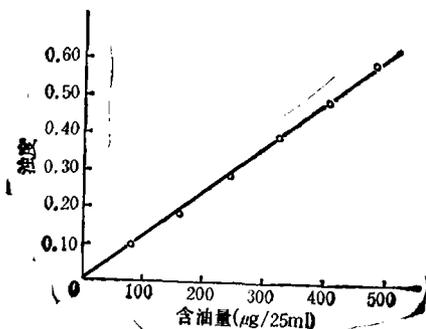


图 1 标准曲线

maximum distance and height of hot smoke cloud is involved. This model has been verified with photograph measurement. (See pp. 26—30)

Toxicological Assessment of the Pesticide Fenvalerate by Bioassay System of *Daphnia magna* Straus

Xiu Ruiqin, Gao Shirong and Xu Yongxiang (Institute of Environmental Health and Engineering, Chinese Academy of Preventive Medicine, Beijing)

In this paper, the experimental result shows that 24h EC₅₀ (median effective concentration) value is 2.5ppb (range 1.8—3.2ppb), 48h EC₅₀ 0.049ppb (range 0.032—0.1ppb) and 96h EC₅₀ 0.0128ppb (range 0.01—0.016 ppb). The LC₅₀ (lethal concentration fifty) values are 25.88ppm (18—32ppm) at 24h, 0.133ppb (0.1—0.18ppb) at 48h and 0.017ppb (0.01—0.022ppb) at 96 h for *Fenvalerate* by using *Daphnia magna*. The result of nonlethal concentration is 0.0001ppb at 96h and 100% lethal concentration 0.0032ppb at 96h. That means *Fenvalerate* is highly toxic to aquatic organism.

The bioassay system of *Daphnia magna* is a method of rapid, sensitive and inexpensive screening procedures for new pesticides, according to the authors. (See pp. 31—33)

Imitation of Nitrification of Sediments Sampled from the Chuanfung River, Kunming, (Yunnan Province)

Kon Yunhong (Kunming Industrial College) and *Sheng Lingling* (Yunnan Institute of Microbiology, Kunming)

In this paper, the effects of aeration, temperature, pH and ammonium ions on nitrification of the sediment sampled from the Chuanfung River have been studied in the laboratory. Orthogonal design was applied to the experiment. The results show that each of the four factors has significant influence on nitrification, and the best conditions for the nitrification of sediment samples are aeration day and night, temperature 34°C, NH₄-N 35 mg/L and pH 8.5. (See pp. 33—37)

Influence of Technological Conditions on the Content of Halogenated-hydrocarbons in Potable Water

Wang Xinmin, Ding Zaiju and Yao Shouren (Anhui Provincial Institute of Environmental Protection)

The paper deals with the relationship between contents of the volatile halogenated hydrocarbons in potable water and technological conditions under which flocculation and quantity of chlorine are different, and some of the methods to reduce halogenated hydrocarbons in potable water are suggested. The results show that halogenated hydrocarbons in liquid chlorine are one of the main sources besides the halogenated hydrocarbons which have been originated from chlorine and organic compounds in water source. (See pp. 37—41)

A Study on the Persistence of Zineb in Eggplants, Tomatoes and Soil

Mo Hanhong, An Fengchun and Zhang Lianzhong (Research Center for Eco-Environmental Sciences, Academia Sinica)

This paper deals with residues of the pesticide *Zineb*. Eggplant and tomato plants grown in plastic buckets were treated with ethylenethiourea-free *Zineb* for 1—3 times at a rate of 0.1g per plant. Plant leaves, fruits and soil samples were collected periodically. Colorimetric method was used for quantitative determination. The results show that half-life of *Zineb* on both eggplant and tomato leaves was determined to be about 14 days. Higher amounts of *Zineb* residues in the plants were observed when multiply applied. *Zineb* residues in the test soils disappeared much more rapidly with half-life of 3.9—4.6 days. Lower amounts of *Zineb* were found to be physically absorbed on eggplant and tomato fruit skin. (See pp. 41—45)

Treatment of Wastewater Discharged from the Factory Producing Phthalic Acid

Wang Jusi, Xu Kun and Xu Liangcai (Research Center for Eco-Environmental Sciences, Academia Sinica)

A process of "flocculation—clarification—filtration—neutralization by lime" was developed for treatment of high concentration wastewater discharged from a phthalic acid production factory. The pollutants can be removed from wastewater very effectively, i.e. phthalic acid can be removed from the original 2000—3000 mg/L to less than 50 mg/L, COD from thousands mg/L to the level about 500 mg/L. Furthermore, pH values of wastewater after treatment can be controlled in the range of 6—7 constantly no matter how high (higher than 13) or how low (lower than 4) in the raw wastewater. After treatment, the water will become degradable by biological process. (See pp. 46—52)

Determination of a Small Amount of Crude Oil in Soli Using Tetrahydrofuran-Turbidimetric Method

Zhang Jiexiang, Zhang Shuxiang, Zhou Sheng and Jing Yunpeng (Shandong Institute of Building Materials)

In this report, the authors intended to find out a method for determining a small amount of crude oil in soil. First oil is extracted from soil by tetrahydrofuran, then subjected to centrifugal settling. And the supernatant is pipetted for determining the content of crude oil by turbidimetry.

The method is limited to an amount of oil 0.025 mg in a 10 g sample. It gives extraction yield about 98%. Compared with the gravimetric analysis, the method is simple and speedy with high yield. The determination of a sample shows that standard deviation of the method is 0.036 and coefficient variation 0.035. Therefore it can be satisfactory to determine a small amount of crude oil containing in soil. (See pp. 57—58)