经验交流

消泡剂硅油 I 在苯胺测定中的应用

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摘要 用预蒸馏—重氮偶合比色法测定印染废水中的苯胺时，蒸馏过程中大量的泡沫出现，是影响测定结果准确度的重要因素。本研究对色泽很深，含酚量较高的废水加入消泡剂硅油 I 以抑制蒸馏产生的泡沫。结果表明，在 100ml 水样中加硅油 I0.05—0.5ml 即可收到理想效果。硅油 I 性质稳定，不随水蒸汽蒸出。对测定无干扰。标准加入回收率为 91.6%—97.3%，蒸馏得率平均为 96%。

关键词 消泡剂，硅油 I，蒸馏，苯胺。

环境中的苯胺污染多来自印染废水。目前通常采用预蒸馏的方法以消除色泽或含酚量的干扰。由于印染废水中有一定量的表面活性剂，预蒸馏时常有大量泡沫冲出而使蒸馏无法进行。本研究选用硅油 I 作为消泡剂，效果显著。

1 实验部分

1.1 仪器与试剂

(1)仪器 721 型分光光度计、10mm 比色皿 。

(2)试剂 5%的亚硝酸钠溶液，2.5%的氨基磺酸铵溶液，2%盐酸萘乙二胺溶液，硅油 I(又称"甲基苯基硅油")，苯胺标准溶液用分析纯苯胺配制[1]。

1.2 实验方法

量取水样 100ml 数份于蒸馏瓶中，各加入不同量的硅油 I(一般 0.05—0.5ml 之间)，加热蒸馏，用 100ml 容量瓶收集馏出液。待蒸出大约 80ml 时，停止加热，稍冷后向蒸馏瓶中再加入 20ml 蒸馏水，继续蒸馏直至馏出液为 100ml 止。摇匀，吸取 10ml 于 25ml 比色管中，用无水碳酸钠调节 pH1.5—2.0，加入 1 滴 5%$NaNO_2$ 溶液，摇匀，放置 3min。加 2.5%氨基磺酸铵溶液 0.5ml，充分振荡，待气泡消失后，加 2%盐酸萘乙二胺溶液，摇匀，放置 40min 后，用波长 545nm，10mm 比色皿，测定吸光度。测得各水样中苯胺的含量。

取水样 100ml 数份分别加入不同量的硅油 I，各加入苯胺标准液 8.0ml，按上述步骤收集馏出液 100ml，测定吸光度，根据测定结果，计算回收率。

取蒸馏水 100ml 数份各加入硅油 I0.2ml、苯胺标准液 8.0ml(加标浓度 0.74mg/L)，按上述步骤收集馏出液 100ml，测定吸光度，根据测定结果，计算蒸馏得率。

2 结果与讨论

2.1 加入不同量的硅油 I 对测定结果的影响

用同一水样取 4 份分别加入 0、0.05、0.20、0.50ml 硅油 I 进行测定，其结果经 t 检验，加入硅油 I 与不加入时的测定结果无显著性差异。

2.2 回收率实验结果

在蒸馏水中加入苯胺标准溶液及硅油 I，进行蒸馏得率试验，试验结果表明，加入适量的硅油 I，平均蒸馏得率为 95.8%，对蒸馏效果无影响。

3 小结

硅油 I 作为苯胺测定预蒸馏中的泡沫抑制剂效果显著，其加入量随着泡沫的高低一般控制在每 100ml 水样加 0.05—0.50ml。硅油 I 性质稳定，不随水蒸汽蒸出，对苯胺测定无干扰。该方法最低检出浓度约为 0.03mol/L。

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参 考 文 献

1 国家环保局。水和废水监测分析方法。第三版，北京：中国环境科学出版社，1989：422

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ng/ml. The characteristic mass and detection limit of copper were 22.0ng and 6.6ng/ml, respectively. The results of analysis of the National Bureau of Standard (NBS), Standard Reference Material (SRM) No. 1648, urban particulate matter, gave recovery of 98% and a precision of 3.3% RSD.

Key words: trace copper, atmospheric particulate matter, graphite probe, atomic absorption spectrometry.

Distribution Pattern of Polycyclic Aromatic Hydrocarbons in the Atmosphere in Huhhot City. Gao Chunmei et al. (Inner Mongolian Scientific Research Institute of Environmental Protection, Huhhot 010010): *Chin. J. Environ. Sci.*, **14**(5), 1993, pp. 79—81

The PAHs in the vapor phase and on the particulates have been collected simultaneously through PUF and fiberglass filter film. The batch collecting samples have been carried out respectively in both inhabited areas and clean meadow areas in the two seasons of winter and summer. The contents of PAHs, such as anthracene, pyrene, chrysene, perylene, bezo(a) pyrene, dibenz(a, h) anthracene and benzo[ghi]-penylene, have been determined by using HPLC. The results show that over half of the PAHs of below four rings were found in the vapor phase and most of the PAHs of over four rings were found in particulates; the percentage of each of PAHs in the vapor phase in summer was higher than that in winter; the percentages of the PAHs in the vapor phase and on particulates in clean meadow areas were different from those in inhabited areas; and in winter the time at which the pollution in inhabited areas was most serious was 10：00-11：30 and 17：30-21：30 but in summer the time was 6：00-10：00 and 18：00-22：00.

Key words: polyurethane foam (PUF), polycyclic aromatic hydrocarbons, vapor phase and particulates, high performance liquid chromatography (HPLC).

Determination for Valence Distribution of Iron with a Stopped Flow-Kinetic Spectrophotometric Method. Wang Jianhua et al. (Department of Chemistry, Yantai Teacher's College, Yantai 640000): *Chin. J. Environ. Sci.*, **14**(5), 1993, pp. 82—84

A stopped flow-kinetic spectrophotometric method for the determination of Fe(Ⅰ) was proposed based on the inductive effect of Fe(Ⅰ)-Cr(Ⅵ) reaction on the Cr(Ⅵ)-iodide redox reaction systems under the optimal conditions of: $[Cr(Ⅵ)]=1.8\times10^{-4}$mol·L^{-1}, $[I^-]=2.0\times10^{-2}$mol·L^{-1}, pH=1.9 590nm. The calibration graph is linear for 0-2.2μg·ml^{-1}Fe(Ⅰ) and the detection limit is 0.009 μg·ml^{-1}Fe(Ⅰ). 25 times higher concentrations of Fe(Ⅱ) do not interfere with the determination of Fe(Ⅰ). Fe(Ⅱ) was reduced to Fe(Ⅰ) and total iron content was measured after the determination of Fe(Ⅰ) content and the recovery of Fe(Ⅰ) was greater than 98%. Valence distributions of iron of several samples were analysed with the proposed method and the results are satisfactory.

Key words: iron, valence distribution, kinetic spectrophotometry.

A Review on the Methods for Determining Partition Coefficient (K_d). Ye Yanmei et al. (Chengdu College of Geology, chengdu 616059): *Chin. J. Environ. Sci.*, **14**(5), 1993, pp. 85—89

The factors affecting partition coefficient (K_d) were discussed, based on experimental data, It is considered that the key to precisely determining a partition coeffieient is to have an experimental model which is sufficiently imitable to the natural system. An experimental method for determing K_d by dynamical simulation is suggested. The K_d values calculated with this method are close to the real values in many cases and the method is found to be simple, convenient and practical. In some cases such as quick reaction, the K_d values may be determined by using a static simulation experiment which is more simple and a special case of the dynamical simulation experiment, and the results are also close to the real situation.

Key words: partition coefficient, dynamical simulation experiment, affecting factors.

Use of Dimethyl Silicone Oil for Defoaming in the Measurement of Aniline. Zhang Qun et al. (Yancheng Environmental Monitoring station, Jiangsu 224002): *Chin, J. Environ. Sci.*, **14**(5), 1993, pp. 90

This paper deals with the use of a dimethyl silicone oil (Silicone Oil Ⅰ) as a defoaming agent in the measurement of aniline present in a textile dyeing and printing wastewater which has a quite dark colour and a higher content of phenols. In the pre-distillation-diazotization-colorimetric determination of the above aniline, a large volume of foams formed during the distillation is an important factor affecting the precision of results and can be defoamed by adding the above silicone oil. It has been

found that the addition of 0.05-0.5ml of silicone Oil I to 100ml of water sample can give a desirable result. Silicone Oil I has a stable property, will not be vaporized out with steam and will not interfere with the measurement. The recovery of standard reference added is 91.1-97.3% and the yield by distillation is 96% on average.

Key words: defoaming agent, silicone Oil I, aniline, distillation.

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