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Dual-Wavelengths Spectrophotometry for Improving the Determination of Acetone in Waste Water

HONG-WEN GAO
School of Chemistry and Chemical Engineering
Anhui University, Hefei-230 039, P.R. China

The conventional reaction of acetone with furfural in the presence of sulfuric acid to form a red substance which has been applied for the improvement of the determination of trace amounts of acetone in water and waste water by the updated method named primary-secondary wavelengths spectrophotometry (PSWS). The results showed that the analytical precision and accuracy were increased and gave the higher sensitivity than the ordinary spectrophotometry. The relative standard deviations were less than 2% and the recovery rate of acetone between 92.7 and 101%.

INTRODUCTION

Acetone usually exists in water polluted by chemical, oil and pesticide industries. At present acetone is determined by colourimetric methods[1, 2]. The reaction of acetone with furfural in sulfuric acid solution can give the high sensitivity and selectivity, and accurate result, so it remains to be applied widely for the analysis of trace amounts of acetone in environment water. Here the above reaction was first to be used for improvement of determination of trace amounts of acetone by the updated method named primary-secondary wavelength spectrophotometry (PSWS). Because PSWS can give the higher precision and sensitivity than the conventional spectrophotometry and give the stable calibration model. The recovery rate of acetone was between 92.7 and 101%, the relative standard deviations less than 2% and the detection limit only 0.02 mg/L.

From the theory of spectrophotometric absorption of a colloid solution or a suspension liquid, the following expression[3] is established for determination of trace amounts of pollutants in a formed colour solution.

$$\left(\frac{Ap+1}{As+1}\right)=\left(\frac{\lambda p}{\lambda s}\right)^{-y} \tag{1}$$

where λp named as primary wavelength which is selected at the maximum absorption point and λs as secondary one which is often chosen at half-peak absorption point. Ap and As are the corresponding absorbance at λp and λs, respectively. The factor y is exponent to the analytical component concentration (X mg/L or μg), as follows.

$$y=\alpha X^{\beta} \tag{2}$$

where both α and β are constants.

From the above expression, the value of y may be obtained, which in turn gives X value from this expression. Both A_p and A_s happen change with the variation of the operation conditions for example room temperature. It is found that $(A_p + 1)/(A_s + 1)$ value changes little. Therefore, both α and β are almost constant, which will happen little change with the variation of the operation conditions.

From the above principle, PSWS is easy to understand , which is different from the other dual-wavelengths methods[4, 5].

EXPERIMENTAL

Visible spectra were recorded with a Model 721 spectrophotometer (Shanghai, China), in a 50 mm glass cell.

Standard acetone solution: weighing a 25 mL volumetric flask with 10 mL of water and adding 2–4 drops of acetone (Shanghai Chemical) distilled weighing it. Diluted to volume and calculated acetone concentration.

Standard acetone solution, 10.0 mg/L: preparing with the above standard acetone solution

furfural solution, 1%: dissolving 1.0 g of furfural (Shanghai Chemical) in 100 mL of anhydrous ethanol (A.R., Shanghai Organic Chemical)

sodium hydroxide solution, 10%

concentrated sulfuric acid (A.R., Shanghai Chemical)

Recommended Procedures

A known volume (less than 5 mL) of a water sample containing less than 50 μg of acetone was taken in a 10 mL volumetric flask. Added 2 mL of 10% sodium hydroxide and 1.0 mL of 1% furfural. Mixed well and heated it in 60°C water bath for 6 min. After cooling, added 2 mL of concentrated sulfuric acid and diluted to volume with ion exchange water and mixed well. After 10 min, measure the absorbance at 520 and 570 nm, respectively, against a reagent blank.

RESULT AND DISCUSSION

Absorption Spectra: Fig. 1 gave the absorption spectra of the colored solution

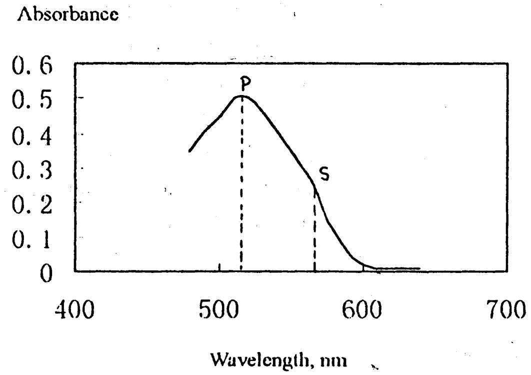

Fig. 1. Absorption spectrum of acetone coloured solution with furfural against reagent blank: P point-520 nm and S point-570 nm

of acetone with furfural. This solutions's absorption reached maximum at 520 nm. According to the above principle the primary wavelength should be selected at 520 nm. The secondary wavelength was arranged at 570 nm here. That is $\lambda p = 520$ nm and $\lambda s = 570$ nm.

Effect of Ligand Reagent: Figure 2 shows the effect of the addition of furfural solution on factor y calculate from expression 2. We found that the addition of 1% furfural was more than 1.5 mL, y remained almost constant and reached maximum. In this work, 2 mL of ligand reagent solution was selected.

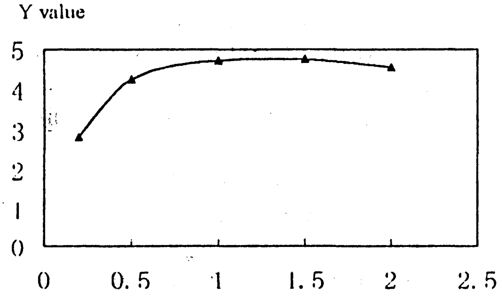

Fig. 2. Effect of ligand solution, 1% furfural on factor y

Calibration Graph: A series of standard acetone solutions were prepared and the absorbance of each was measured and plotted. From the measurement results, Curve log y against the logarithm value of acetone amounts (x μg), log x was shown in Fig. 3.

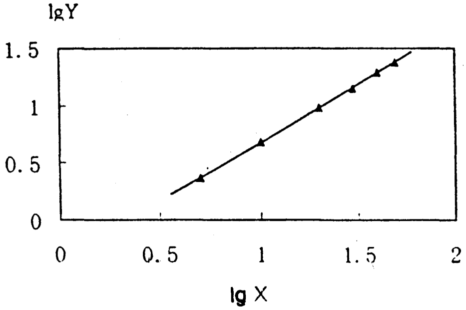

Fig. 3. Standard curve for the determination of acetone

We found that the curve linearity was obvious. The cut distance log α was calculated to be −0.355 and the oblique rate β to be 1.03 from the straigth line in Fig. 3. The relation of y with x was therefore obtained by the following expression.

$$y = 0.442x^{1.03} \quad (3)$$

Effect of Foreign Ions: None of the following ions will affect the direct determination of 20 μg of acetone (<10% error): 100 mg of Cl^-, SO_4^{2-}, PO_4^{3-}, CO_3^{2-}, NO_3^-, Ca(II), Mg(II); 10 mg of Zn(II), Fe(II), Ti(IV), Br^-, Al(III), I^-, F^-, NH_4^+, Cu(II), Co(II), Ni(II), Cd(II), 1 mg of Cr(III), Sn(II).

Precision and Detection Limit: Six replicate determinations of standard acetone solution containing 10 mg/L was carried out, the relative standard deviatin (RSD) being 0.7%. However, the RSD with the single wavelength method was 2.1%. The precision for PSWS was therefore better than that for the ordinary spectrophotometric method.

We used $L_{min} = kS_b/S$ to calculate the detection limit of acetone and tannin by PSWS, where k = 3, Sb named as standard deviation and S named as sensitivity. Replicate determination of twenty reagent blanks gave S_b of y value computed from (Ap + 1)/(As + 1) was equal to 0.03. The analytical sensitivity S was equal to the above α value, 0.442. Therefore the detection limit of acetone was L_{min} = 0.02 mg/L.

Samples Analyzed: As a test of the method acetone was determined in, for example waste water and surface water. The results were listed in Table 1. We found the results by the recommended method agreed with the conventional method. The RSDs were less than 2% and the recovery rate of acetone between 92.7 and 101%.

TABLE- 1
DETERMINATION OF ACETONE IN WATER SAMPLES

Sample	By conventional method	By PSWS		
		Added	Found	Recovery, %
Waste water	0.423	0	0.432 0.451 0438	
			0.429 0.449 0.439	
		0.500	0.947 0.938 0.956	101
Sewage	0.170	0	0.175 0.183 0.176	
		0.100	0.231 0.269 0.279	98.3
River water	< 0.1	0	0.070 0.060 0.70	
		0.100	0.161 0.159 0.169	96.3
Chemical	1.18	0	1.39 1.19 1.32	
Waste water		1.00	2.07 2.28 2.33	92.7

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