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Analysis of influencing factors for the determination of ammonia nitrogen in water by spectrophotometry

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Abstract

The content of ammonia nitrogen in water was determined by national standard method - nessler's reagent spectrophotometry. Through comparison, it was found that the method of reagent configuration, the amount of reagent, the detection wavelength, the color development time and the temperature had an impact on the analysis results in the ammonia nitrogen measurement in water by the reagent spectrophotometry. Based on the experimental results, the factors influencing the determination of ammonia nitrogen by spectrophotometry with nessler's reagent were discussed.

Keywords Nessler's reagent spectrophotometry, Ammonia nitrogen, Influencing factors, Blank value, ABS value, Correlation coefficient.

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1.Introduction

Ammonia nitrogen in aqueous solution is nitrogen in the form of free ammonia (or non-ionic ammonia, NH_3) or ionic ammonia (NH_4^+). The main sources of ammonia nitrogen in water are the decomposition products of nitrogen-containing organic matter in domestic sewage under microbial action, as well as the industrial wastewater from fertilizer production, nitric acid, coking, gas, nitrocellulose, rayon, synthetic rubber, calcium carbide, dye, varnish, electroplating, and petroleum exploitation and petroleum product processing. Water organisms, especially fish, are sensitive to non-ionic ammonia. To protect freshwater aquatic organisms, the concentration of non-ionic ammonia in water should be less than 0.02 mg L^{-1}. The inhalation of ammonia gas with a concentration of 0.1mg L^{-1} can cause irritation, while ammonia gas with a concentration higher than 1.75 mg L^{-1} can cause acute death.

At present, the commonly used method for the determination of ammonia nitrogen in environmental monitoring laboratories in China is nessler's reagent spectrophotometry, which has the characteristics of simplicity, rapidity, economy and high benefit. However, in actual operation, factors such as reagent allocation method, reagent dosage and temperature will affect the blank value, Abs value and correlation coefficient of ammonia nitrogen measurement. Therefore, in this paper, the blank value, Abs value and correlation coefficient of the reagent photometry were analyzed, and the optimal conditions were determined through comprehensive consideration, so as to achieve the optimal expected effect.

2. Principles and methods

2.1 Principle of ammonia nitrogen reaction

Ammonia nitrogen, in the form of free ammonia or ammonium ions, reacts with nessler's reagent to form a reddish brown complex, the complex of the absorbance is proportional to the ammonia nitrogen content, measuring absorbance wavelengths of 420 nm, due to the reaction with ammonia nitrogen nessler's reagent is more complex, so on different reaction substance content are respectively according to equation (1) ~ (5) .

$2[HgI_4]^{2-} + NH_3 + 3OH^-$ --- $OHgHgNH_2I\uparrow + 7I^- + 2H_2O$ (Brown orange) (1)

$2[HgI_4]^{2-} + NH_3 + 2OH^-$ ------ $OHO\text{-}HgI\text{-}HgNH_2I\uparrow + 6I^- + H_2O$ (Dark brown) (2)

$2[HgI_4]^{2-} + NH_3 + OH^-$ ------- $I\text{-}HgI\text{-}HgNH_2I\uparrow + 5I^- + H_2O$ (chocolate) (3)

$2[HgI_4]^{2-} + NH_4^+ + 4OH^-$ ------- $OHgHgNH_2I\uparrow + 7I^- + 3H_2O$ (Brown orange) (4)

$NH_4^+ + OH^- \rightarrow NH_3\cdot H_2O \rightarrow NH_3 + H_2O$ (5)

In general, nessler's reagent is mainly used for the determination of trace ammonia nitrogen. The reaction equations (5) and (8) and (9) indicate that NH_3 and NH_4^+ can be converted into each other in aqueous solution, which is mainly affected by the pH of the solution.

2.2 Methods

After the water sample was treated, 50mL was put into the 50mL colorimetric tube, 1.0ml potassium and sodium tartrate solution was added, and after mixing, 1.5ml sodium tartrate reagent was added, and the colorimetric determination was conducted at the wavelength of 420 nm.

3. Results and discussion

3.1 The influence of the amount of natrium's reagent on the absorption value

The method described in literature [1] was used to configure nessler's reagent, and the other steps were as 2.2. By adding different amounts of nessler's reagent solution, the blank value, Abs value and correlation coefficient were measured at different wavelengths, as shown in table 1-5.

Table 1 relative values (pH=13) were obtained by measuring the amount of nessler's reagent (1.0mL) at different wavelengths.

wavelength	the blank value	Abs value	correlation coefficient
410nm	0.070	0.756	0.9979
415nm	0.071	0.762	0.9932
420nm	0.036	0.683	0.999
425nm	0.029	0.643	0.9987

Table 2 relative values (pH=13) were obtained by measuring the amount of nessler's reagent (1.5mL) at different wavelengths.

wavelength	the blank value	Abs value	correlation coefficient
410nm	0.116	0.761	0.9993
415nm	0.075	0.73	0.9993
420nm	0.045	0.697	0.9995
425nm	0.036	0.657	0.9994

Table 3 relative values (pH=13) were obtained by measuring the amount of nessler's reagent (2.0 mL) at different wavelengths.

wavelength	the blank value	Abs value	correlation coefficient
410nm	0.005	0.761	0.9991
415nm	0.094	0.823	0.9997
420nm	0.055	0.7473	0.9999
425nm	0.036	0.689	0.9999

Table 4 relative values (pH=13) were obtained by measuring the amount of nessler's reagent (2.5 mL) at different wavelengths.

wavelength	the blank value	Abs value	correlation coefficient
410nm	0.049	0.706	0.9996
415nm	0.066	0.773	0.9991
420nm	0.119	0.850	0.9993
425nm	0.190	0.948	0.9986

Table 5 relative values (pH=13) were obtained by measuring the amount of nessler's reagent (3.0 mL) at different wavelengths.

wavelength	the blank value	Abs value	correlation coefficient
410nm	0.215	0.964	0.9502
415nm	0.129	0.740	0.9997
420nm	0.078	0.710	0.9998
425nm	0.051	0.673	0.9997

As can be seen from table 1-5, the blank value at each detection wavelength also increases with the increase of the dosage of nazarite reagent. When the dosage of nessler's reagent increases to 2.5ml and above, the blank value at the three wavelengths exceeds the limit value of 0.06, so it is excluded. The absorbance response value at each wavelength increased with the increase of the amount of nessler's reagent, and the absorbance increased from

1.0 mL to 2.0 mL. The dosage of nessler's reagent was 1.5-2.5ml, and good correlation was obtained at all wavelengths. To sum up, 2.0 mL was selected as the appropriate dosage for this method.

3.2 Influence of detection wavelength on absorption value

The methods described in literature[1-2] and 3.1 were used to configure the nessler's reagent. Other steps were as 2.2. The blank value, Abs value and correlation coefficient were measured at different wavelengths. As shown in table 6.

Table 6 Blank value, Abs value and correlation coefficient measured at different wavelengths

nessler's reagent	wavelength	the blank value	Abs value	correlation coefficient
2.0ml pH=13	410nm	0.140	0.761	0.9991
	415nm	0.094	0.823	0.9997
	420nm	0.055	0.7473	0.9999
	425nm	0.036	0.689	0.9999

It can be seen from table 6 that the blank value at the wavelength of 410nm is the highest, and the correlation coefficient of the Abs response value is low, so it is not adopted. Although the response value of Abs at 415nm is the highest and the correlation coefficient can also meet the requirements, the blank value is the highest, exceeding the limit value of 0.06, so it is not adopted. The correlation coefficients obtained at 420 and 425nm were good, and the blank value was lower than 0.06 limit (slightly higher at 420nm), but the response value of Abs at 420nm was higher than that at 420nm. To sum up: considering the blank value, the Abs response value and the linear correlation coefficient, the suitable wavelength of this method was selected as 420nm.

3.3 Influence of color rendering time on absorption value

The methods in literature[1] and 3.1 were used to configure nessler's reagent, and the other steps were as 2.2. The blank value, Abs value and correlation coefficient were determined at different color development times. The results are shown in table 7.

Table 7 Blank value, Abs value and correlation coefficient measured at different color rendering times

time	the blank value	Abs value	correlation coefficient
5min	0.048	0.727	0.9997
20min	0.048	0.732	0.9997
45min	0.050	0.738	0.9996
75min	0.051	0.739	0.9997

As can be seen from table 7, with the increase of color rendering time, both blank value and Abs value gradually increase, and good correlation is obtained at each time. To sum up, 10-30 is selected as the best color rendering time in this method.

3.4 influence of temperature on absorption value

The methods in literature[1-2] and 3.1 were used to configure nessler's reagent, and the other steps were as 2.2. The blank value, Abs value and correlation coefficient were measured at different temperatures, as shown in table 8.

Table 8 Blank value, Abs value and correlation coefficient measured at different temperatures

temperature	the blank value	Abs value	correlation coefficient
5℃	0.029	0.717	0.9990
15℃	0.040	0.761	0.9960
25℃	0.032	0.782	0.9983
30℃	0.036	0.808	0.9998

As can be seen from table 8, Abs increases with the increase of temperature, but the blank value of 35℃ has exceeded the limit value of 0.06, so it is removed, and the correlation at 30℃ is the best. To sum up, 35℃ is selected as the best temperature in this method.

3.5 Effects of different potassium sodium tartrate configurations on absorption value

The methods in literature [1-2] and 3.1 were used to configure nessler's reagent, and the other steps were as 2.2. The blank value, Abs value and correlation coefficient were measured respectively in boiling and non-boiling conditions when potassium sodium tartrate was configured, as shown in table 9.

Table 9 Blank value, Abs value and correlation coefficient measured by different potassium and sodium tartrate allocation methods

Potassium sodium tartrate configuration method	the blank value	Abs value	correlation coefficient
Not boil	0.028	0.693	0.9980
boil	0.029	0.717	0.9990

As can be seen from table 9, the boiled potassium sodium tartrate and the blank value without boiling were nearly the same, but the boiled potassium tartrate with high natron and Abs had a good correlation. To sum up, potassium sodium tartrate boiled was selected in this method.

3.6 Effects of different configurations of nessler's reagent on absorption value

The blank value, Abs value and correlation coefficient were determined by $HgCl_2$-KI-KOH and HgI_2-KI-NaOH, respectively, in the preparation of NaOH reagent in literature[1-2], as shown in table 10.

Table 10 Blank values, Abs values and correlation coefficients obtained by different methods of nessler's reagent configuration

Method of nessler's reagent configuration	the blank value	Abs value	correlation coefficient
$HgCl_2$-KI-KOH	0.025	0.767	0.9986
HgI_2-KI-NaOH	0.029	0.717	0.9990

As can be seen from table 10, although the blank value of hgcl2-ki-koh configuration method of NaOH reagent is low and the Abs value is high, the correlation is not as good as that of HgI_2-KI-NaOH configuration method. Moreover, the blank value of the two configuration methods is within the scope of national standards. HgI_2-KI-KOH method is difficult to configure and has low parallelicity, which is not conducive to preservation. In summary, the method of HgI_2-KI-NaOH configuration was selected to configure nessler's reagent.

4. Conclusion

The above results show that the method of reagent configuration, the amount of reagent, the detection wavelength, the color development time and the temperature have a certain influence on the analysis results in the ammonia nitrogen measurement of water by the reagent spectrophotometry. The dosage of 2ml NaOH prepared by HgI_2-KI-NaOH was selected after comprehensive consideration. Potassium sodium tartrate prepared under boiling condition was used to obtain the best results and the best effect at the wavelength of 420nm and 10-30min color reagent at 35℃.

References

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