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Determination of Nitrite in the Kapuas Besar River by Spectrophotometric Method

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Abstract: Pontianak City is located at the junction of the Kapuas Besar River, Kapuas Kecil River, and Landak River with a width of 400 meters, a depth of 12 to 16 meters, and its branches are 250 meters wide. The samples in this study were taken from 9 points on the Kapuas Besar River, dari hulu ke hilir sungai. Based on Government Regulation Number 22 of 2021 concerning the Implementation of Environmental Protection and Management, the nitrite quality standard for surface water is 0.06 mg/L. The method used to determine nitrite levels is spectrophotometry referring to SNI 06-9689.9-2004. The principle of this measurement is the formation of purplish red azo compounds from benzenediazonium ions reacted with (1-naphthyl)-ethylenediamine (NED) reagent so that they can be determine with a UV-Vis spectrophotometer. The nitrite standard calibration curve obtained has good linearity marked by the value of r = 0.999985 of 0.99 required value so that it can be used as a reference for measuring nitrite levels in surface water samples. The results of the accuracy test are seen from the % Trueness value, where the resulting value is 97.7383% of the minimum set value, which is 90-110%. The precision test results are seen from the %RPD value, where the resulting value is 0% of the maximum set value, which is 5%. The nitrite levels obtained were all below the maximum set value. The highest nitrite level was 0.028 mg/L with the sample code 119, and it below than 0,06 mg/L.

Keywords: Kapuas Besar River, nitrite, spectrophotometry.n, Information and Communication Technology (ICT), literacy.

Introduction

Humans have immeasurable needs, ranging from the need for clothing, boards, food, until water which plays important role in supporting life. For the people of West Kalimantan, especially Pontianak City, the Kapuas River is the largest source of water that can be found. Pontianak is located at the junction of the Kapuas Besar River, Kapuas Kecil River, and Landak River with a width of 400 meters, a depth of 12 to 16 meters, and its branches are 250 meters wide (Regional Development Planning Agency, 2020). Since long time ago, the Kapuas River has been the center of Pontianak community activities, although now this is slowly changing and giving several impacts on environment. Currently, the Kapuas River is a place where many settlements stand. This resulted in many unstructured buildings, irregular road patterns, and difficulties in accessing clean water.

The conversion of the function of Kapuas River in Pontianak indirectly urges the government and related agencies to monitor its condition based on several parameters. This is important because the water source for the Regional Drinking Water Company still comes from the Kapuas River. Parameters that serve as a reference for Kapuas River water quality include pH, turbidinity, dissolved oxygen (DO), temperature, total suspended solids (TSS), total dissolved solids (TDS), chemical oxygen demand (COD), biological oxygen demand (BOD), nitrate (NO_3), nitrite (NO_2^-), cyanide, sulfate, and dissolved metals (Ariadi, et al., 2021). If some of these parameters have levels outside the specified threshold permitted, it can cause disruption of the ecosystem in the river and the environment around it.

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One of parameters that should to be concerned is nitrite. High nitrite concentrations can occur due to several things, namely, water pollution by agriculture from the use of certain fertilizers and chemicals, water pollution by animal husbandry and fisheries, water pollution by industry, and water pollution by urban activities (Ardhaneswari and Wispriyono, 2022; Prabowo, 2017). Based on Government Regulation Number 22 of 2021 concerning the Implementation of Environmental Protection and Management, the nitrite quality standard for surface water is 0.06 mg/L. Nitrite levels that exceed the limit can cause the toxic effects of methemoglobinemia (more than 10% of hemoglobin is converted to methemoglobin). This can cause gastro intestinal, diarrhea, general depression, headaches, coma, and even death (Abdurrival and Syamsinar, 2017). In addition, nitrites can be carcinogenic if they react with aminos or amides, so they are very dangerous if consumed by humans (Hersa and Pratiwi, 2018). Therefore, it is very important to determine nitrite levels in the Kapuas Besar River in Pontianak City to monitor whether the water used by the community is safe or not.

The method used to determine nitrite levels is spectrophotometry, which refers to SNI 06-9689.9-2004. The purpose of determining this level is to determine the quality of the water being tested so that early treatment can be carried out if it is detected exceeding the set limit. The determination of nitrite levels carried out in practical work is expected to provide an overview and information regarding the determination of nitrite levels, water quality in the Kapuas Besar River, as well as complement existing sources. Therefore, by this research, it is hoped that the nitrite concentration in the Kapuas Besar River can be known so as to be able to create awareness of the importance of keeping the environment cleanness.

Materials and Methods

Study area

This research study is in the field of chemistry and environment, in which research examines the content or concentration of a chemical substance contained in the environment where samples were taken.

Procedures

Preparation of nitrite mother liquor (NO2-N)

Nitrite mother liquor was prepared by dissolving 1.232 grams of NaNO2 in distilled water and making up to 1000 mL. Nitrite mother liquor has a level of 250 mg/L NO2N.

Preparation of intermediate solutions

The intermediate solution was prepared using 10 mL of nitrite mother liquor which was diluted in 50 mL of distilled water. This solution has a concentration of 50 mg/L.

Preparation of standard solutions

The standard solution was prepared from 1 mL of intermediate solution which was diluted in 100 mL of distilled water. This solution has a concentration of 0.50 mg/L.

Preparation of working solutions

The working solution was prepared using a standard solution adjusted with distilled water in a 50 mL flask to create a calibration curve. The series has a concentration of 0 mg/L; 0.01 mg/L (1 mL standard solution); 0.02 mg/L (2 mL standard solution); 0.05 mg/L (5 mL standard solution); 0.1 mg/L (10 mL standard solution); 0.15 mg/L (15 mL standard solution); 0.20 mg/L (20 mL standard solution), and duplo of the clearest test sample.

Production of Certified Reference Material (CRM)

CRM was made by diluting CRM solution with a concentration of 0.619 mg/L 5 times, so that this solution has a concentration of 0.1238 mg/L.

Preparation of sulfanilamide reagent (H2NC6H4SO2NH2)

Sulfanilamide as much as 5 grams was dissolved in a mixture of 300 mL of distilled water and 50 mL of concentrated HCl. The solution was then diluted with distilled water to 500 mL.

Preparation of NED Dihydrochloride reagent

500 mg of N-(1-naphthyl)-ethylene diamine dihydrochloride (NED Dihydrochloride) dissolved

in 500 mL of distilled water. The solution is stored in a dark bottle in the refrigerator and replaced every month or when it turns brown.

Test samples, blanks, container blanks

The test sample, blank, and container blank are placed in a 50 mL flask. The test sample is a sample of water at nine points of the Kapuas Besar River. The blank and the container blank are distilled water, the difference between the two is that the blank is taken when it is to be used, while the container blank is made on the day the sample is taken.

Reagent addition

The addition of reagents was carried out in all solutions, namely working solution, test samples, blanks, container blanks, and CRM. First, the reagent added was 1 mL of each sulfanilamide. The solution was homogenized and allowed to stand for 8 minutes. Second, the reagent added was NED dihydrochloride, 1 mL each. The solution was homogenized and allowed to stand for 10 minutes. Measurement of the absorbance of the solution must be carried out immediately (no more than 2 hours).

Measurement of the calibration curve and absorbance of the test sample with a UV-Vis spectrophotometer

First, what is measured is the standard curve, in the following order: blank, container blank, Certified Reference Material (CRM), and working solution from a concentration of 0 mg/L to 0.2 mg/L. The absorbance is read at a wavelength of 543 nm and a calibration curve is created. Second, the test sample is measured by the same procedure as the calibration curve measurement. The linearity of the calibration curve (r) must be more than 0.99, and the difference in duplo results must not be more than 5%

Data analysis

There are two calculations in the nitrite test, namely %Trueness (%T) to determine accuracy and Relative Percent Difference (%RPD) to determine data precision. The equation can be seen below.

$$\%Trueness\ (\%T) = \frac{CRM\ Instrument}{\text{CRM Theory}} \times 100\%$$

$$\%\text{RPD} = \frac{\text{Value 1} - \text{Value 2}]}{\left[\frac{\text{Value 1+Value 2}}{2}\right]} \times 100$$

Results and Discussion

Results of Analysis of Nitrite Levels in Kapuas Besar River Water

Measurement of nitrite levels begins with determining the calibration curve of a standard nitrite solution. Calibration curves are made as a quality control measurement and are considered to have no interference. The linearity of the calibration curve (r) accepted according to SNI 06-6989.9-2004 is greater than or equal to 0.99. The linear calibration curve has an absorbance in the range of 0.2-0.8 according to the Lambert-Beer Law. The calibration curve method used in this study uses 7 standard solutions, namely. 0; 0.01; 0.02; 0.05; 0.1; 0.15; and 0.2 mg/L (Table 1).

Table 1. Nitrite Standard Series Measurement Results.

No	Concentration (mg/L)	Absorbance
1	0.000	0.001
2	0.001	0.036
3	0.002	0.075
4	0.050	0.182
5	0.100	0.360
6	0.150	0.541
7	0.200	0.716

The standard calibration curve of the measurements can be seen in Figure 1.

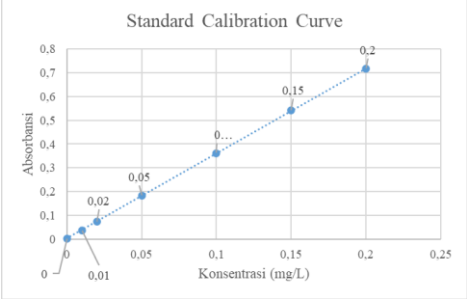

Figure 1. Standard Calibration Curve.

Measurement of nitrite levels in nine water samples in the Kapuas Besar River yielded concentrations and absorbances which can be seen in Table 2.

Table 2. Nitrite Standard Series Measurement Results.

Test Sample Code	absorbance	Concentration (mg/L)
BLK	0.005	0.001
Container BLK	0.007	0.001
CRM	0.438	0.122
117	0.059	0.016
118	0.061	0.017
119	0.031	0.008
120	0.101	0.028
121	0.049	0.013
122	0.056	0.015
123	0.051	0.014
123 (Duplo)	0.052	0.014
124	0.045	0.012
125	0.076	0.021

The sample test result curve can be seen in Figure 2.

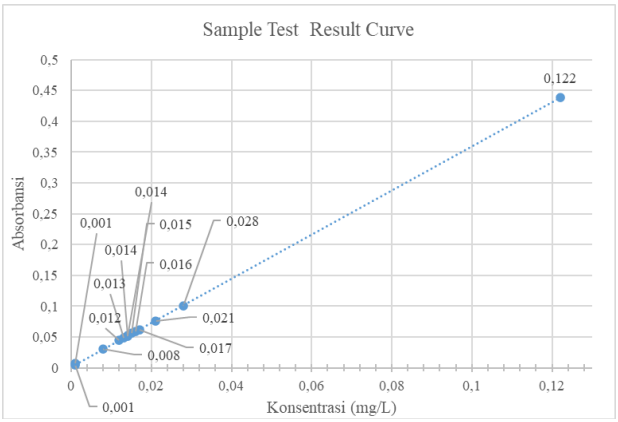

Figure 2. Sample Test Result Curve.

The result of calculating the level of accuracy using %Trueness is 97.74% while the result of calculating the level of precision of the data with %RPD is 0%, with the following calculation.

$$\%Trueness\ (\%T) = \frac{CRM\ Instrument}{\text{CRM Theory}} \times 100\% = \frac{0.121}{0.1238} \times 100\% = 97{,}74\%$$

$$\%\text{RPD} = \frac{\text{Value 1} - \text{Value 2]}}{\left[\frac{\text{Value 1+Value 2}}{2}\right]} \times 100\% = \frac{0{,}013 - 0{,}013}{\frac{(0{,}013+0{,}013)}{2}} \times 100\% = 0\%$$

Discussion

Nitrite is one of the water pollutant compounds formed due to human activities and microorganisms. The nitrite threshold stipulated in Government Regulation No. 22 of 2001 is 0.06 mg/L. Nitrite levels that exceed the threshold can cause negative impacts because they can trigger the formation of nitrosamine compounds which are teratogenic, mutagenic, and even carcinogenic. In addition, nitrites can also reduce oxygen transport in the bloodstream through the mechanism of oxidation of hemoglobin to methemoglobin, which is hemoglobin that cannot carry oxygen (Yugatama, et al., 2019).

The determination of nitrite levels was carried out on several surface water samples from the Kapuas Besar River using the spectrophotometric method. This is based on the confidence level obtained by 95% with a relative uncertainty of 5.88% of the 15% acceptance requirements set. The basic principle of this method is the absorption of electromagnetic radiation by the sample at a certain wavelength, so that the absorbance and concentration of the instrument can be obtained. Measurement of nitrite levels begins with determining the calibration curve of a standard nitrite solution. Calibration curves are made as a quality control measurement and are considered to have no interference. The linearity of the calibration curve accepted according to SNI 06-6989.9-2004 (r) is greater than or equal to 0.99. A linear calibration curve has an absorbance in the range 0.2-0,8 according to Lambert-Beer's Law.

Based on the calibration curve, the regression equation is obtained, namely, y = 3.57987x + 0.00185, with a coefficient of determination (r2) 0.99997, a correlation coefficient (r) 0.999985 and an intercept value of 0.00185. The intercept value serves to measure changes in the value of the dependent variable as a result of changes in the independent variables. The dependent variable on this graph is absorbance, while the independent variable is concentration. That is, the absorbance will be affected and changed by changes in time concentration as the independent variable (Makmur and Afrizal, 2019). The slope value obtained is 3.57987. Positive values indicate that the dependent variable and independent variable

have a directly proportional effect, where increasing concentration will cause the absorbance value to also increase (Yuliani, 2021). The intercept and slope values also indicate a systematic error in the experiment. A slope with a value of -3.57987 indicates a relative bias, while an intercept with a value of 0.00185 indicates a fixed bias (Hadi and Asiah, 2018). The coefficient of determination (r2) is 0.99997, close to 1 which indicates that there is a significant correlation between concentration and absorbance (Annisa, et al., 2019). The coefficient of determination of 0.99997 indicates that 99.997% of the data can be trusted while 0.003% is an error (Saefuddin, et al., 2009). In addition, the value of the correlation coefficient (r) obtained is 0.999985 from the value of 0.99 specified in SNI, so that the standard calibration curve value is acceptable. While an intercept with a value of 0.00185 indicates a fixed bias (Hadi and Asiah, 2018). The coefficient of determination (r2) is 0.99997, close to 1 which indicates that there is a significant correlation between concentration and absorbance (Annisa, et al., 2019). The coefficient of determination of 0.99997 indicates that 99.997% of the data can be trusted while 0.003% is an error (Saefuddin, et al., 2009). In addition, the value of the correlation coefficient (r) obtained is 0.999985 from the value of 0.99 specified in SNI, so that the standard calibration curve value is acceptable.

The calibration curve is used to see the linearity that will be obtained in a certain concentration range. In sample measurement, the calibration curve is used as a reference so that the results obtained have the appropriate results. The concentration of the sample measured should not be more or less than the standard concentration range because the results obtained are not known for their linearity. Nitrite mother liquor was prepared by dissolving 1.232 grams of NaNO2 in distilled water and making up to 1000 mL. Nitrite mother liquor has a level of 250 mg/L NO2-N. NaNO2 dissolved with distilled water will undergo oxidation to NO2-N which can form diazo compounds (Dianti, 2018).

Based on the results of measuring nitrite levels from 9 samples, the highest levels were contained in sample number 119. Nitrite levels tended to be higher in sample 119 due to the large number of community activities at the sampling location, where community activities can be a source of nitrite contaminants. Samples from other areas that have low nitrite content tend to be due to being far from settlements and population activities (Kusnoto and Pur Mintasari, 2018). The measurement results show that when the concentration of nitrite in the sample is greater, the absorbance is also greater. This is in accordance with the Lambert-Beer Law which states that concentration will be directly proportional to absorbance. The largest nitrite concentration obtained based on measurements was 0.028 with an absorbance of 0.101 in sample 120 . Then, the smallest nitrite concentration was 0.008 with an absorbance of 0.031 in sample 119.

The duplo sample used for measurement was sample number 123. The concentration results obtained after deducting the blank as a control were 0.013 in both samples, so the %RPD obtained was 0%. The condition for the acceptance of the %RPD value is no more than 5. The %RPD value indicates the precision of the results, so that the results obtained in the measurements are very precise. The CRM level obtained after deducting the blank as a control was 0.121 mg/L with an absorbance of 0.438. CRM is used to see accuracy, where accuracy is determined using the %Trueness value. The measurement results produce a %Trueness value of 97.738% from the range of 90-110% acceptance requirements, so the measurement results can be said to be accurate because they fall within the specified range. The results of measuring nitrite levels at 9 sample points of Kapuas Besar River water were below the set threshold, which was 0.06 mg/L.

Conclusions

Based on the research, it was known that the nitrite standard calibration curve has good linearity marked by the value of r = 0.999985 of 0.99 the standard. The results of the accuracy test are seen from the % Trueness value, where the resulting value is 97.7383% of the minimum standard, which is 90-110%. The precision test results are seen from the %RPD value, where the resulting value is 0% of

the maximum standard, which is 5%. Then, nitrite levels in Kapuas Besar River obtained were all below the quality standard. The highest nitrite level was 0.028 mg/L from the maximum value allowed, 0.06 mg/L, means that the water still has safe level of nitrite.

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