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Determination Study of Measurement Uncertainty On Crude Palm Oil (CPO) Using Several Chemical Analysis Methods

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Abstract. The measurement uncertainty on crude palm oil (CPO) for the determination of free fatty acid using the neutralization titration method base AOCS Ca5a – 40, and the determination of the Fe^{2+} using the spectrophotometric method have been studied. The process of uncertainty estimation is based on specifying measurand, identifying uncertainty sources, quantifying uncertainty using type A (by the statistical analysis of series of observations) and type B (by the other statistical analysis of series observations) evaluation of uncertainty, then converting them to deviation standards, and the last step was to calculate and combine the uncertainty standard and uncertainty expanded. Spreadsheet software MS Exel 2007 based on Kragten spreadsheet uncertainty calculation method was used to simplify the calculation of the combined standard uncertainty. The combined standard uncertainty was multiplied with a coverage factor of 2, and the expanded uncertainty for analysis determination of free fatty acid using the neutralization titration method based on AOCS Ca5a–40 with a concentration of 0.1612 mol/L in the sample solution was ±0.0002 mol/L. Whereas the expanded uncertainty for the determination of Fe^{2+} using a spectrophotometric method with a concentration of 0.131 mg/L in the sample solution was ±0.083 mg/L and calculated using a coverage factor of 2.14.

Keywords: Measurement Uncertainty, Measurand, Kragten Spreadsheet, Coverage

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1 Introduction

Quantitative analysis is the determination of how many certain substances are in a sample. The substance specified, often indicated as the substance of interest or analyte, may comprise a small or large portion of the sample being analyzed. In quantitative chemical analysis, a lot of analysis is carried out using chemical analysis methods. (Underwood, AL 1980). The analysis results obtained are expected to be used for certain purposes, such as checking the quality of drinking water, forensic analysis of body parts in several criminal cases, and determining the quality of an industrial product to be exported abroad.

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Therefore, it is important to know the quality of the measurement results by prioritizing quality assurance on the measurements made by the laboratory concerned. This is intended as a rule that a laboratory is capable and has data of the required quality. The chemical analysis places great emphasis on the precision and accuracy of the analytical results obtained using standard methods. For this reason, it is important to state the quality of the measurement results obtained so that their suitability can be seen by including the level of confidence in the measurement. One of the useful measurements is measurement uncertainty.

Measurement uncertainty began to be published in 1993 by ISO (International Organization for Standardization) in collaboration with BIPM (International Bureau of Weights and Measures), IEC (International Electrotechnical Commission), IFCC (International Federation of Clinical Chemistry), IUPAC (International Union of Pure and Applied Physics) and OIML (International Organization on Legal Metrology) in "Guide to the Expression of Uncertainty in Measurement" (GUM) who wrote about general rules for calculating and expressing measurement uncertainty in analytical reports will become commonplace in the future. which will come. (Ellison et al. 1999)

In this study, the authors are interested to calculate the measurement uncertainty of crude palm oil (CPO) by using analytical methods with neutralization titration and UV-Vis spectrophotometry.

2 Materials and Methods

2.1 Equipments

In this study, the equipments used were Agilent Carry 40 Spectrophotometer, glassware, analytical balance, furnace, and hot plate.

2.2 Materials

The main materials used were crude palm oil (CPO). glacial acetic acid (CH_3COOH), hydrochloric acid (HCl), hydroxylamine,1,10-phenanthroline, sodium acetate (CH_3COONa), $Fe(NH_4OH)_2S0_4$, and zinc oxide (ZnO), phenolphthalein indicator and distilled water.

2.3 Determination of Fe^{2+} Concentration in Standard Solution

As much as 50 mL of the standard series solution of 0.2 mg/L Fe^{2+} ion was put into a glass beaker, added 1 mL of HCl, then added 3 mL of hydroxylamine-HCl 5%. After that, evaporated to half the initial volume, and cooled. After being cooled, CH_3COONa was added while stirring until the pH was 3. As much as 10 mL acetate buffer was added and added 2 mL of 1.10-phenanthroline 0.1% solution, then transferred to a 50 mL volumetric flask, diluted with distilled water to the marked line, homogenized, and allowed to stand for 15 minutes. Finally, the absorbance was measured at λ =510 nm. The same treatment was carried out for the standard solution of 0.4; 0.6; 0.8; and 1.0 mg/L and blank solution.

2.4 Determination of Fe^{2+} Concentration in Sample

As much as 50 mL CPO was put in a porcelain cup, then added 4.0 g of ZnO, then burned until the fire was extinguished. After being burned, it was ashed in a furnace at 600ºC for 2 hours. After cooling, the sample was put into a 50 mL volumetric flask, added 1 mL of HCl, 3 mL of hydroxylamine-HCl 5%, 10 mL of acetate buffer, and 2 mL of 1,10-phenanthroline 0.1% solution, then diluted with distilled water to the marked line, homogenized and allowed to stand for 15 minutes, and the absorbance was measured at λ=510 nm.

The concentration of $FeSO_4$ solution was obtained by using the formula below:

$$C_{Fe^{2+}} = \frac{A_{sampel} - A}{B} x\ L$$

Where:

A = Intercept of calibration curve [AU]

A_{sample} = Fe^{2+} absorbance in sample solution [AU]

B = slope of calibration curve [AU $L.g^{-1}$]

$C_{Fe}{}^{2+}$ = Fe^{2+} ion concentration in sample solution [$mg.L^{-1}$]

L = diluent factor

2.5 Free Fatty Acid Determination by Neutralization Titration Method.

A total of 7.05 g CPO was put into an Erlenmeyer and added 50 mL of neutral alcohol, and added 3 drops of phenolphthalein indicator, then titrated with NaOH 0.1N until a purple-red color was formed.

The NaOH concentration was obtained by using the formula below:

$$C_{NaOH} = \frac{1000.M_{KHP}.P_{KHP}}{F_{KHP}.V_T} \qquad [mol.L^{-1}]$$

Where :

C_{NaOH} = NaOH solution concentration ($mol.L^{-1}$)

F_{KHP} = Molecular weight of KHP ($g.mol^{-1}$)

M_{KHP} = KHP weight (g)

P_{KHP} = KHP Purity

V_T = Average volume of NaOH solution (mL)

2.6 Uncertainty Determination Procedure

2.6.1 Masurand Specification

The measurand specification is an explanation of the measurement procedure which consists of the measurement steps and the mathematical equations used to determine the measurand. In this case, the measurand is Fe^{2+} concentration.

2.6.2 Identification of Uncertainty Sources

The sources of uncertainty can be identified from the parameters contained in the mathematical equations used to determine the measure, where all these parameters also have their sources of uncertainty.

2.6.3 Uncertainty Calculation

Because only a few method treatment data are available, the uncertainty calculation was carried out by calculating the components of the uncertainty separately. The following are the methods used to derive the individual components of uncertainty:

a. Calculation of type A uncertainty: obtained from the repeatability of the experiment and calculated as the standard deviation of the measured value.

b. Calculation of type B uncertainty, which consists of i). calculation based on results or other data, such as glassware tolerances, substance purity, drift and rounding of analytical balance numbers, drift and rounding of absorbance numbers on a spectrophotometer, and ii).through theoretical principles, such as the effect of temperature on the volume that can affect the results.

2.6.4 Combined Uncertainty Calculation

Standard Uncertainty

All uncertainty contributions are converted to standard uncertainties, i.e. as standard deviations. The following is how to convert the contribution of uncertainty into standard uncertainty, namely: i). If the uncertainty is derived from a type A uncertainty calculation, the standard uncertainty is the standard deviation of the used, ii). If the uncertainty comes from a type B uncertainty calculation, then three probability distributions are used to convert it to a standard uncertainty, with the following criteria below:

a. If the limits of ± *a* are given with the confidence level, then to calculate the standard deviation, the value of *a* is divided by the percentage point of the normal distribution that is appropriate for that confidence level.

b. If the limits of ± *a* are given without a confidence level and it is reasonable to consider the margins to be preferable, uniform distribution with a standard deviation is used.is $a/\sqrt{3}$

c. If the boundaries of ± *a* are given without a degree of confidence, but it is reasonable to assume that the margins are not favored, but rather are the midpoints, a triangular distribution, with a standard deviation was used.

Combined Standard Uncertainty

In order to calculate the combined standard uncertainty, it was calculated using the Kragten spreadsheet uncertainty calculation method using the MS Excel 2007 software program (Microsoft Inc.).

Expanded Uncertainty

In order to calculate the expanded uncertainty, it was calculated based on the equation U = k.x.u_c (y) where the coverage factor was obtained using a t-distribution table at a 95% of confidence level.

$$V_{eff} = \frac{u_c^4(y)}{\sum_{I=1}^{N} \frac{c_i^4 u^4(x_i)}{v_i}}$$

3 RESULT AND DISCUSSION

Tabel 1. Uncertainty calculation results with neutralization titration and spectrophotometric methods.

No	Analysis Method	Analyte Concentration (y) (mol.L^{-1})	Expanded Uncertainty (U_p(y)) (mol.L^{-1})	Coverage Factor (k_p)
1	Neutralization Titration	0.1612	±0.0002	2
2	Spectrophotometry	0.1310	±0.0083	2.14

The determination analysis results on FFA by the neutralization titration method and determination analysis results by using the spectrophotometric method are presented in Table 1.

3.1 Uncertainty Determination on FFA by Neutralization Titration Method

Uncertainty Sources

The analysis of uncertainty sources will be easier to understand by drawing an Ishikawa diagram, as shown in figures 1 to 3. The repeatability uncertainty components are contained in the mass parameters of KHP standard titrimetry and the titration volume of NaOH.

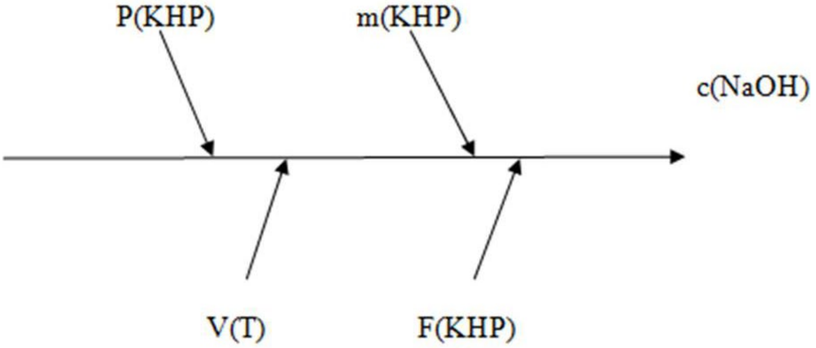

Figure 1.The simple Ishikawa diagram to determine the FFA

Standard Uncertainty

Type A Uncertainty for Repeatability of Measurement Result (rep)

Based on the results of data processing obtained a relative standard deviation (RSD) of 0.1%. This repeatability value includes repeatability values for all sources of uncertainty, namely KHP mass, KHP mass purity standards, molecular weight standards, and NaOH volume standards used in the titration. Therefore, the repeatability uncertainty component for each of these sources of uncertainty is not recalculated.

Type B Uncertainty for Repeatability of KHP Mass Uncertainty

The logs of the quality control showed that standard uncertainties from 0.05 mg at the same weight as checked to 100 g. This value for repeatability was determined by ten measurements of tare and gross weight followed by different calculations of each pair of measurements and an evaluation of the different standard deviations. The contribution of this repeatability was only added once due to the standard deviation different directly from the given experiments.

Calibration/Linearity

The calibration description of the quota balance was approximately ±0.15 mg for the linearity of this value providing the maximum difference between the actual weight of the data and the scale reading. The equilibrator recommended the indeterminate evaluation use a quadrilateral distribution to convert it into a linear contribution to an indeterminate standard. Linear contribution balance according to figure 2. This contribution has been calculated twice and the first was for tare and the other for gross weight.

The combination of these two contributions gave an uncertain standard u(m_{KHP}) of the m_{KHP} mass.

F(KHP)

From the International Union of Pure and Applied Chemistry (IUPAC) table, the last atomic weight and uncertain data for the elements of KHP($C_8H_5O_4K$) displayed in Table 2.

Tabel 2. Uncertainty data for KHP($C_8H_5O_4K$) elements

Unsur	Atomic Weight	Uncertainity	Standard Uncertainity
C	12.0107	± 0.0008	0.00046
H	1.00794	± 0.00007	0.000040
O	15.9994	± 0.0003	0.00017
K	39.0983	± 0.0001	0.000058

(Ellison et al. 1999)

For each element, the uncertainty standard was found by processing the uncertain IUPAC quota as a bound form of a rectangular distribution. The uncertain standard adjustment was obtained by dividing the value by the root.

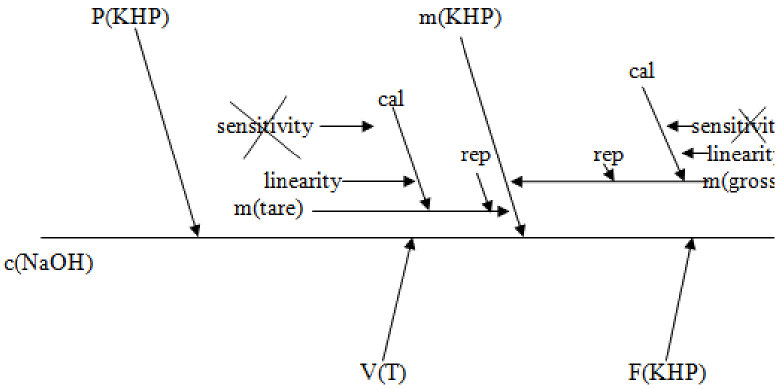

Figure 2. Ishikawa diagram to determine the FFA

P(KHP)

It provides the information that did not further relate to the uncertainties in the list which was these uncertainties have a quadrilateral distribution.

The contribution of the separate elements gives the weight formula which, together with the uncertain contribution for each data, is:

Table 3. Uncertainty data for KHP($C_8H_5O_4K$)

Unsure	**Calculation**	**Result**	**Standard Uncertainty**
C_8	8 x 12.0107	96.0856	0.0037
H_5	5 x 1.00794	5.0397	0.00020
O_4	4 x 15.9994	63.9976	0.00068
K	1 x 39.0983	39.0983	0.000058

(Ellison et al., 1999).

The uncertainty of each of these values was calculated by multiplying the standard uncertainty from the previous table of the number of atoms. It provided the formula weight for KHP. Since this statement was the sum of the independent values, the standard uncertainty $u(F_{KHP})$ was the square root of the sum of the squares of each contribution.

V(T)

1. Repeatability of The Volume Used

The burettes used were arranged using a limited volume which must be examined and recorded; for example 0 to 10, 10 to 20, 5 to 15, and so on. The equation should be investigated in terms of repeatability of the different volumes used, such as 5, 10, 15 mL, and so on. For example, repeatability of the previous data used 19 mL stored and given a deviation standard sample of 0.004 mL, using the direct measurement as an uncertain standard.

2. Calibration

The limited accuracy of the used volume is shown in figure 3. For a 20 mL burette, this amount was typically about 0.03 mL. It was assumed that the triangular distribution provides an uncertain standard of $\frac{0.03}{\sqrt{6}}$ was 0.012 mL.

3. Temperature

The uncertainty due to lack of temperature control was calculated in the same way as in the previous example, but at this point, we will take the possible temperature variations of 3ºC (with the accuracy of 95%). Then use the coefficient of volume expansion for water which was 0.00021/ºC.

a. Repeatability of the acquired point

The repeatability of the endpoints obtained was carefully examined during the actual method. Then gave an uncertain standard condition of 0.004 mL which was suitable data.

b. Deviation of the obtained endpoint

There was no other indication where the endpoint was determined from the shape of the pH curve that did not match the equivalence point because a strong acid was titrated with a strong base. Therefore, it was estimated that deviations from the obtained endpoints and uncertain data can be ignored. The V_T was found to be 18.64 mL and was used to mix the remaining 4 of the uncertain contribution $u(V_T)$ of the V_T volume.

3.2 Uncertainty Determination on Fe^{2+} by Spectrophotometric Method

Uncertainty Sources

The sources of uncertainty contained in the Fe^{2+} analysis using the spectrophotometry method were: a). random variation in the measurement of y, which affects the response of the calibration standard solution y_i and the response of the measured solution y_0, b). random variation derived from the errors to obtain the value of the calibration standard solution x_i, c). the linear relationship between x_i and y_i, d). The xi and yi values contained the constant uncertainty, which rise because the x values were generated from a series of stock solution dilutions, and also due to the y values were obtained from absorbance measurements using a spectrophotometer. The four sources of uncertainty above illustrated in figure 4 to 5.

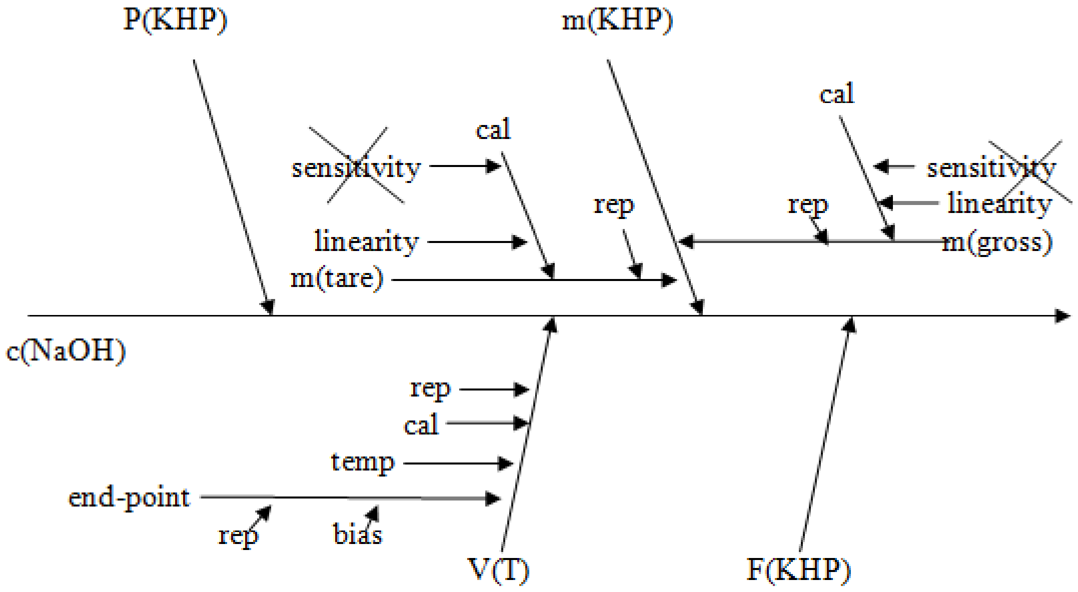

Figure 3. Ishikawa calibration diagram to determine the FFA

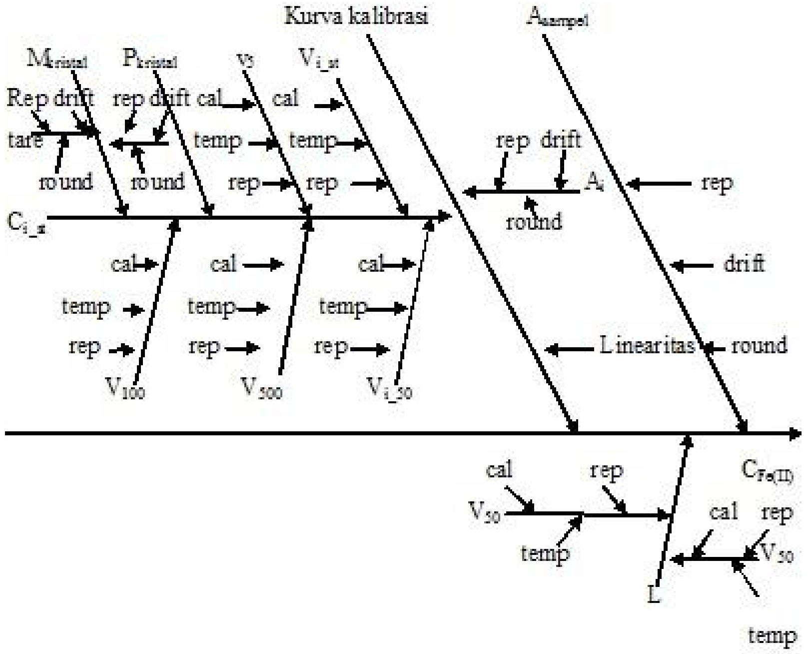

Figure 4. Ishikawa diagram to determine the Fe^{2+} ion

Standard Uncertainty

Type A Uncertainty for Repeatability of Measurement Result (rep)

From the results of data processing obtained S_{x0} was 0.0039 mg/L. This value was the uncertainty value in the measurement of y, which affects the response of the calibration standard solution y_i and the response of the measured solution y_0 which was denoted by $u(x_0, y_i)$.

In addition, the results data in Table 3 showed that F_{count}, was smaller than F_{table}, so the null hypothesis was accepted and there was a relationship between y and x. It meant that the uncertainty due to the linear relationship between x and y was not large enough to affect the combined standard uncertainty, so it was ignored.

Therefore, the uncertainty component of $u(x_0,x_i)$and the linearity relationship between x and y were neglected, then only the uncertainty contribution S_{x0} was used in the calculation of the combined standard uncertainty, and this value included the repeatability uncertainty component value for all sources of uncertainty in the analysis using spectrophotometric methods.

Calculation of type B Uncertainty

The x and y values contained constant uncertainties because x_i was generated from a series of stock solution dilutions and also because y_i was obtained from absorbance measurements using a spectrophotometer.

Uncertainty of purity standard of $Fe(NH_4)_3(S0_4)_2.6H_2O$ *crystal* $[u(P_{crystal})]$

From the data processing results, the value of u($P_{crystal}$) obtained was 0.0058 g. This value was obtained from the crystal purity value given by the supplier, which was 100% ± 1%.

Uncertainty of crystal mass standard [u($m_{crystal}$)]

From the data processing results, the value of u($m_{crystal}$) obtained was 0.00017 g. Due to the weighing was done by subtracting the mass of crystals and the container (gross) with the remaining mass of a little crystal and the container (tare), both of which have the same components of uncertainty, namely drift and rounding on the balance. Because no special research was conducted to determine the value of drift uncertainty, this value was obtained.

The standard uncertainty of 100 mL volumetric flask [u(V_{100})]

From the data processing results, the value of u(V_{100}) obtained was 0.13 mL. This value was obtained by combining the two components of the uncertainty of the 100 mL volumetric flask, namely the calibration uncertainty and the difference between the calibration temperature and the laboratory temperature. The calibration uncertainty value was obtained from the value indicated on the 100 mL graduated cylinder, namely (100 ± 0.08) mL. This uncertainty was assumed to be a triangular distribution. Meanwhile, the temperature uncertainty component was obtained by multiplying the difference in calibration temperature with the laboratory temperature, which varies ±10ºC (calibration temperature was 20ºC), with the coefficient of expansion of the water volume. Since this variation was not specifically observed, it was assumed to be a uniform distribution.

The standard uncertainty of 500 mL volumetric flask [u(V_{500})]

From the data processing results, the value of u(V_{500}) obtained was 0.61 mL. This value was obtained by combining the calibration uncertainty and the difference between the calibration temperature and the laboratory temperature. The calibration uncertainty value was obtained from the tolerance value indicated on the 500 mL graduated cylinder, namely (500 ± 0.15), calibrated at 20ºC, which was assumed to be a triangular distribution. Meanwhile, the temperature uncertainty component was obtained by switching the difference between the calibration temperature and the laboratory temperature with the coefficient of water volume expansion.

The standard uncertainty of volume pipette 5M [u(V_5)]

From the data processing results, the value of u(V_5) obtained was 0.0073 mL. This value was obtained by combining the calibration uncertainty and the difference between the calibration temperature and the laboratory temperature. The calibration uncertainty value was obtained from the tolerance value indicated on the 5 mL volumetric pipette apparatus, namely (5 ± 0.001) mL, which was calibrated at 20ºC. In contrast, the temperature uncertainty component was obtained by multiplying the difference in calibration temperature with the laboratory temperature by the coefficient of water volume expansion.

The standard uncertainty of 50 mL volumetric flask used to prepare calibration standard solution [u(V_{i_50})]

From the data processing results, the value of u(V_{i_50}) obtained was 0.064 mL. This value was obtained by combining the calibration uncertainty and the difference between the calibration temperature and the laboratory temperature. The calibration uncertainty value was obtained from the tolerance value indicated on the 50 mL measuring flask, namely (50 ± 0.005) mL, which was calibrated at 20ºC. Meanwhile, the temperature uncertainty component was obtained by multiplying the difference in calibration temperature with the laboratory temperature by the coefficient of water volume expansion.

The standard uncertainty of the volume taken to prepare the first calibration standard solution [u(V_{1_st})]

From the data processing results, the value of u(V_{1_st}) obtained was 0.0043 mL. This value was obtained by combining the calibration uncertainty and the difference between the calibration temperature and the laboratory temperature. The value of calibration uncertainty was obtained from the tolerance value stated, namely (1 ± 0.01) mL on a 1 mL volume pipette, which was calibrated at 20ºC. While the temperature uncertainty component was obtained by multiplying the difference in calibration temperature with the laboratory temperature by the coefficient of water volume expansion.

The standard uncertainty of the volume taken to prepare the second calibration standard solution [u(V_{2_nd})]

From the data processing results, the value of u(V_{2_nd}) obtained was 0.0085 mL. This value was obtained by combining the calibration uncertainty and the difference between the calibration temperature and the laboratory temperature. The calibration uncertainty value was obtained from the tolerance value indicated on the 2 mL volumetric pipette apparatus, namely (2 ± 0.02) mL, which was calibrated at 20ºC. In contrast, the temperature uncertainty component was obtained by multiplying the difference in calibration temperature with the laboratory temperature by the coefficient of water volume expansion.

Standard uncertainty of the volumes taken to prepare the third [u(V_{3_th})], fourth [u(V_{4_th})], and fifth [u(V_{5_th})] calibration standard solutions.

In order to make the third to fifth calibration standard solutions, a 5 mL of volume pipette was used so that the uncertainty of the three calibration standards was the same. From the results of data processing, obtained u(V_{3_th}), u(V_{4_th}), and u(V_{5_th}) were 0.0085 mL. This value was obtained by combining the calibration uncertainty and the difference between the calibration temperature and the laboratory temperature. The calibration uncertainty value was obtained from the tolerance value indicated on the 5 mL volumetric pipette apparatus, namely (5 ± 0.05)mL, which was calibrated at 20ºC. While the temperature uncertainty component was obtained by multiplying the difference in calibration temperature with the laboratory temperature by the coefficient of water volume expansion.

The standard uncertainty of 50 mL volumetric flask [u(V_{50})]

The standard uncertainty value u(V50) was the same as the standard uncertainty value u(V_{i_50}), which was 0.064 mL.

The standard uncertainty of the volume of sample solution taken for spectrophotometric analysis [u(V_{50})]

For this volume measurement, a 100 mL of graduated cylinder with a tolerance of (100 ± 1) mL was used, which was calibrated at 20ºC. From the data processing results, the value of u(V_{40}) obtained was 0.41 mL. This value was obtained by combining the calibration uncertainty and the difference between the calibration temperature and the laboratory temperature. The calibration uncertainty value was obtained from the tolerance value listed on the 100 mL measuring cylinder. While the temperature uncertainty component was obtained by multiplying the difference in laboratory temperature by the coefficient of water volume expansion.

Uncertainty of absorbance measurement standards using a spectrophotometer

All absorbance measurement uncertainties are influenced by three main sources of uncertainty, namely drift, repeatability, and rounding of the spectrophotometer absorbance value. However, since repeatability uncertainty has been used in calculating the yield standard deviation, only drift uncertainty and rounding of spectrophotometer absorbance values are calculated. Because the spectrophotometer is the same for measuring all absorbance solutions, the uncertainty of rounding the spectrophotometer absorbance values for all absorbance values is the same.

Rounding uncertainty [u(A_{round})]

This uncertainty value was half of the last digit of the absorbance value read on the spectrophotometer, which was 0.0005 AU. By using a uniform distribution, the standard uncertainty u(A_{round}), was 0.00029 AU.

Drift uncertainty (A_{drift})

The spectrophotometer drift was listed in the supplier's catalog as ± 0.002 AU per hour at an absorbance level close to 0.0 AU, and ± 0.005 AU per hour at an absorbance level close to 1.0 AU.

Calculation of The Combined Standard Uncertainty

Calculation of standard uncertainty was calculated using the Kragten spreadsheet uncertainty calculation method.

The expanded uncertainty value of $U(C_{Fe}{}^{2+})$ was 0.0083 $mg.L^{-1}$ which was obtained by multiplying $U_c(C_{Fe}{}^{2+})$ with a coverage factor of 2.14, which was obtained by using a student-t distribution table with effective degrees of freedom V*eff* 14 at a 95% of confidence level. This value means that the true value was between (0.1310±0.0083) $mg.L^{-1}$ and (0.1310±0.0083) mg/L with 95% probability.

Comparison of the contribution of each uncertainty component to the combined standard uncertainty value $U_c(C_{Fe}{}^{2+})$. The contribution diagram of u(L) illustrated in figure 5 showed that the contribution of u(L) and u(A_{sample}) uncertainties are the two main sources that affect the combined uncertainty value.

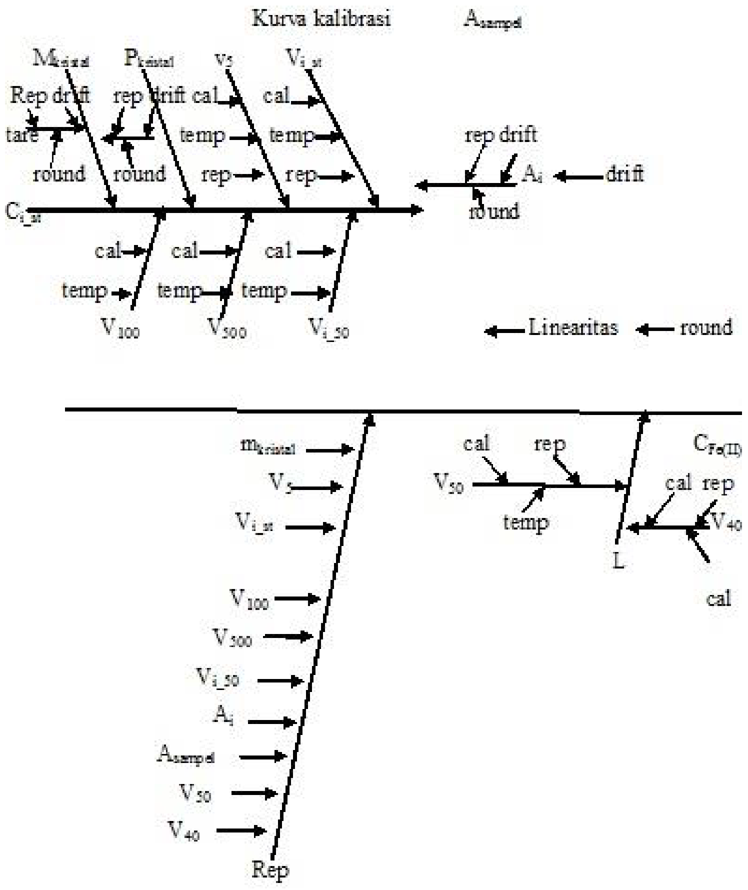

Figure 5. Ishikawa diagram to determine Fe^{2+} solution

4 Conclusion

In conclusion, the determination of free fatty acids by the neutralization titration method based on AOCS Ca5a-40 was (0.1612 ± 0.0002 mol.L^{-1}). Where the uncertainty was calculated using a coverage factor of 2 at a 95% of confidence level. This study also found that determination analysis results of Fe^{2+} using the spectrophotometric method was obtained the concentration of Fe^{2+} in the sample solution was (0.1310 ± 0.0083 mg.L^{-1}), which the uncertainty obtained by using a coverage factor was 2.14 at a 95% of confidence levels.

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