The Differences of the Result of Copper Test Using UV-Vis Spectrophotometry with Neocuproine Complexing Agent and AAS

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Keywords: Copper, UV-Vis Spectrophotometry, Atomic Absorption Spectrophotometry

Abstract: Copper is a hazardous heavy metal and often pollutes the environment that may degrade the water quality. This study is a comparative study, conducted in Baristand Industry Laboratory of Palembang. The samples are SRM (Standard Reference Material) based on the standard working ranges that are made from pure copper products from NIST (National Institute of Standards and Technology). The samples are assigned into three points 0.5 ppm; 1 ppm; and 2 ppm from the standard working range. Data of the study differences of copper examination results using UV-Vis Spectrophotometry with neocuproine complexing agent and Atomic Absorption Spectrophotometry were analyzed by independent t-test. Levels of the three samples of UV-Vis Spectrophotometry method were 0.5021 ppm; 1.0298 ppm; and 2.0109 ppm, respectively while level of the three samples of Atomic Absorption Spectrophotometry method were 0.4803 ppm; 0.9957 ppm; and 2.0024 ppm, respectively. Result of independent t-test exhibited the sig (2-tailed) value of $p = 0.974$ with the average difference of 0.0214667. Obtained p value was $p > 0.05$. Based on this study, it can be concluded that there are no difference of copper examination results using UV-Vis Spectrophotometry with neocuproine complexing agent and Atomic Absorption Spectrophotometry.

1. INTRODUCTION

Health laboratory is a health facility that performs measurement, determination and testing of human-derived materials or non-human derived materials for the determination of diseases, health conditions or factors that may affect the health individual and society according to KEPMENKES RI No: 364/ MENKES/SK/III/2003.

Laboratory services in Indonesia are currently being held in various types and levels of services, such as in Puskesmas (Government Primary Health Care Service) Laboratories, Regency/Municipal Health Laboratories, Regency/Municipal Hospitals, Public and Private Hospitals, Private Clinical Laboratories, Central Laboratory of Health (BBLK) and Health Laboratory Center (BLK) according to KEPMENKES RI No: 1792/MENKES/SK/XII/ 2010.

The types of health laboratories based on services consists of clinical and public health laboratories. Public health laboratory is a laboratory which conducts examination services in the field of microbiology, physics, chemistry and or other fields related to public health interest and environmental health, especially to support prevention of disease and improvement of public health in accordance to KEPMENKES RI No: 364/MENKES/SK/III/2003.

The chemical field consists of Aluminium (Al), Iron (Fe), Hardness, Chloride (Cl), Manganese (Mn), pH, Zinc (Zn), Sulphate (SO4), Ammonia (NH3) and Copper (Cu) level assessment. Cu plays an important role in the formation of red blood cells, release of iron from the tissues, formation of bone and central nervous system and other connective tissues. Cu is also a component of certain enzymes. United States of America assigned a safe ingested level of Copper as 1.5 - 3.0 mg a day (Almatsier, 2009). Excess amount of copper will cause gastric irritation, capillary blood vessel damage, damage of liver, kidney and nerve tissues resulting in depression (Windri, 2011).

Techniques used for metal examinations include Atomic Absorption Spectrophotometry (AAS) and UV-Vis Spectrophotometry. AAS is an expensive sophisticated tool and not all laboratories possess it while UV-Vis Spectrophotometry is the most convenient technique due to its availability of

Sari, I., Merdaliani, R., Mariadi, P. and Sebayang, R.
The Differences of the Result of Copper Test Using UV-Vis Spectrophotometry with Neocuproine Complexing Agent and AAS.
DOI: 10.5220/0007542703510355
In *Proceedings of the 2nd International Conference Postgraduate School (ICPS 2018)*, pages 351-355
ISBN: 978-989-758-348-3

instrumentation, simplicity, speed, precision, accuracy, and low cost (Tehrani et al., 2014).

Copper determination can be conducted by atomic absorption spectrophotometry (AAS). Several studies had determined Cu levels in samples such as Windri (2011), Listiowati et al., (2011) and Sa'adah and Winata (2010) that examinied Cu levels in samples using AAS.

Another method in addition to AAS is UV-Vis Spectrophotometry which use a variety of complexing agents. Applicable complexing agents are 6- (2-Methoxynaphthyl) -2,3-dihydro-1,2,4-triazine-3-thione that was carried out by Tehrani et al. (2014), Ligan (2- [6-Nitro -2-benzothiazolylazo] -4-hydroxy benzoic acid) that was carried out by Jreo (2015) and neocuproine that was carried out by Itnawita and Bali (2012).

Copper (Cu) examination using UV-Vis Spectrophotometry with neocuproine complexing agent (American Public Health Association) and Atomic Absorption Spectrophotometry (SNI 06-6989.6-2004) are well established and standardized methods. Validation is usually performed for newly manufactured and developed analytical methods, whereas for available and standardized methods (e.g. from AOAC, ASTM, and others) in exception for the first time use in certain laboratories, validation is usually unnecessary, only verification is required.

Verification of analysis method is a method validation measure, an assessment of certain parameters aimed to prove that the parameters meet the requirements for its application. Parameter verification methods include: linearity, detection and quantitative limit, accuracy, and precision (Anggraini, 2016). Based on description above, the authors aim to find the difference of copper (Cu) examination results using UV-Vis Spectrophotometry with neocuproine complexing agents and Atomic Absorption Spectrophotometry (AAS) in which method verification will be performed.

2. SUBJECT AND METHODS

This study was conducted in Balai Riset and Standarisasi Industri (BARISTAND) from April 09 to May 03, 2017. The samples in this study were made using SRM based on the standard working range that were made from pure Copper (Cu) products from NIST (National Institute of Standards and Technology). The standard working range of UV-Vis Spectrophotometry were 0.0; 0.4; 1,2; 2.0; 2.8; and 3.6 ppm (Standard Method, 2005) while Atomic Absorption Spectrophotometry were 0.0 ppm; 0.2 ppm; 0.5 ppm; 1 ppm; 2 ppm; 3 ppm and 4 ppm (SNI 06-6989.6-2004). Water samples were taken at three points 0.5 ppm; 1.0 ppm; and 2.0 ppm.

This study is a comparative study. The examination methods used in this study is UV-Vis Spectrophotometry with neocuproine complexing agent and Atomic Absorption Spectrophotometry (AAS).

3. RESULTS AND DISCUSSION

3.1 Verification of UV-Vis Spectrophotometry and AAS Methods

Method verification is a reconfirmation measure by testing a method by completing objective evidences, whether the methods meet the established requirements and fit the objectives.

3.1.1 Determination of Wavelength on UV-Vis Spectrophotometry

Determination of wavelength was conducted by measuring the absorbance of copper standard solution of 2 ppm concentration in the wavelength range of 400 – 550 nm. The results of maximum wavelength measurements were presented in Figure 1.

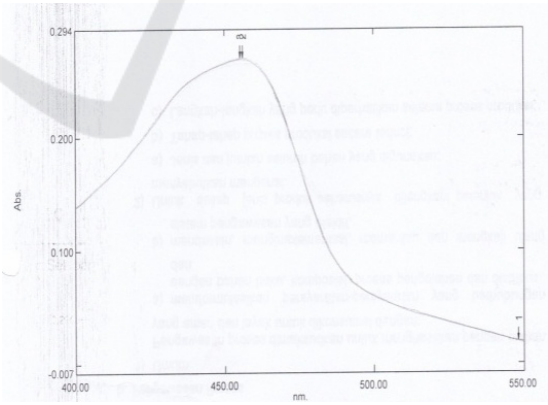

Figure 1: UV-Vis Spectrophotometry Wavelength Curve

Based on above figure of wavelength spectrum, a wavelength of 456 nm was obtained. Wavelength range for copper examination using UV-Vis spectrophotometry with a neocuproine complexing agent is 450 – 460 nm (American Public Health Association).

3.1.2 Linearity

Method linearity is used to determine the standard capability, so it can prove a linear relationship between the analytical concentration and the detector response (Wardani, 2012).

Linearity test is obtained by making standard curve of copper examination conducted by making a series of copper standard solution with various concentrations of UV-Vis Spectrophotometry at 0; 0.4; 1,2; 2; 2.8; and 3.6 ppm whereas of AAS at 0; 0.2; 0.5; 1; 2; 3; and 4 ppm, made from a 1000 ppm solution. Solutions were diluted to 20 ppm for UV-Vis Spectrophotometry and 10 ppm for AAS then each standard solution was read on UV-Vis Spectrophotometer and AAS devices. The results of the copper linearity curve using UV-Vis Spectrophotometry can be seen in Figure 2 below.

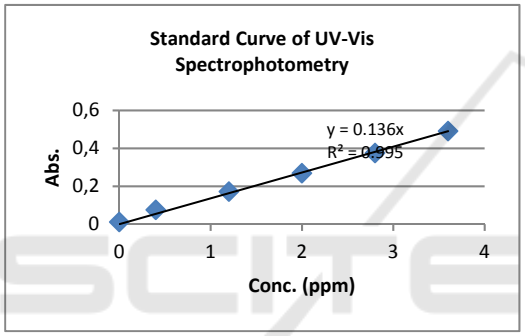

Figure 2: Standard Curve of UV-Vis Spectrophotometry

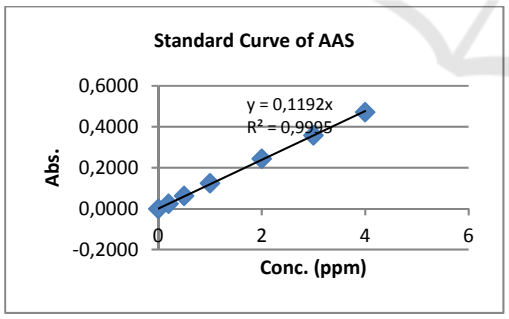

Figure 3: Standard Curve of AAS

The absorbance value seen on the standard curve of copper examination using UV-Vis Spectrophotometry was y= 0,136x with linear regression of r= 0.995. The absorbance value seen on the standard curve of copper examination using AAS was y= 0.119x with linear regression of r= 0.999. Both linear regression values (r) have met the established requirements of r > 0.995 (Wardani, 2012).

3.1.3 Accuracy

Meticulousness is expressed as a percent return of added analytics and the value of precision can be expressed by percent recovery (Wardani, 2012). The accuracy test was conducted by adding 1 ppm concentration of copper standard solution into 1 ppm copper sample, subsequently read using UV-Vis Spectrophotometer and AAS % recovery then calculated.

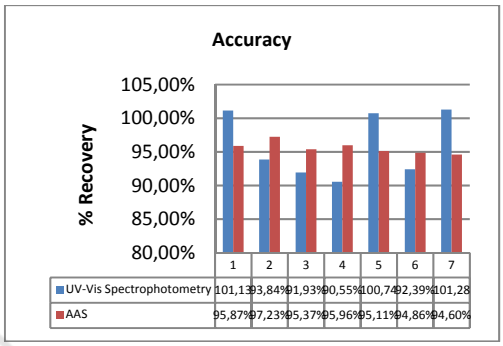

Figure 4: Copper Accuracy Examination

The average % recovery with 7 times measurements is 95.98% in UV-Vis Spectrophotometry and 95.57% in AAS. The result of this retrieval test has met the precision of the predetermined requirements. The recovery results for the analytic in1 ppm matrix (%) sample, the accepted recovery (%) was in the range of 80 - 110% (Wardani, 2012). Thus, the results of this retrieval tests on both methods have met the prescribed conditions based on the acceptable recovery range.

3.1.4 Precision

Precision is a measure indicating the degree of conformity among individual test results, measured by distribution of individual results from the mean if the procedure is applied repeatedly to the samples taken from a homogeneous mixture. Precision is measured based on standard deviation or relative standard deviation (Riyanto, 2014). Precision test was conducted by measuring standard copper solution of 2 ppm concentration 7 times. The precision test was obtained by calculating the value of % relative standard deviation (% RSD).

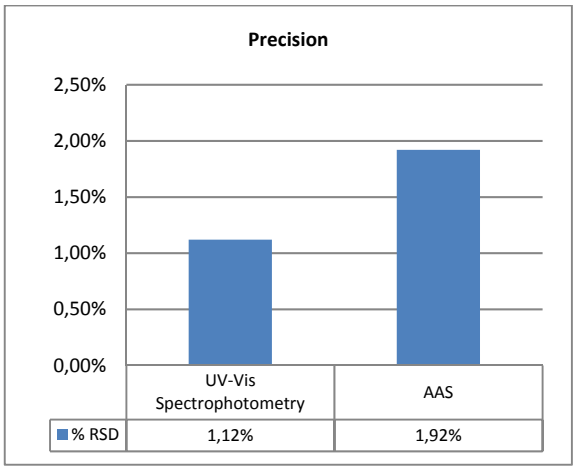

Figure 5: Precision of copper examination

Obtained %RSD was 1.12% in UV-Vis Spectrophotometry and 1.92% in AAS. The result of this precision tests indicated that both methods have met the precision criteria. Precision criteria are given if the method provide a relative standard deviation (RSD) or a coefficient of variation (CV) of 2% or less (Riyanto, 2014). The results of the second precision test of this method showed that the obtained accuracy is precise i.e. 1% < RSD ≤2% (Wardani, 2012).

3.1.5 LOD and LOQ

LOD is the smallest amount of detectable analytic in the sample and still provide a significant response compared to the blank, while LOQ is the smallest amount of analytic in the samples that still meet the accuracy and precision criteria and quantifiable with good accuracy and precision (Wardani, 2012).

LOD and LOQ tests were performed by measuring the absorbances of the smallest copper solution of 0.4 ppm for UV-Vis Spectrophotometry and 0.2 for AAS. LOD and LOQ values were calculated from regression equation of the obtained copper standard calibration curve.

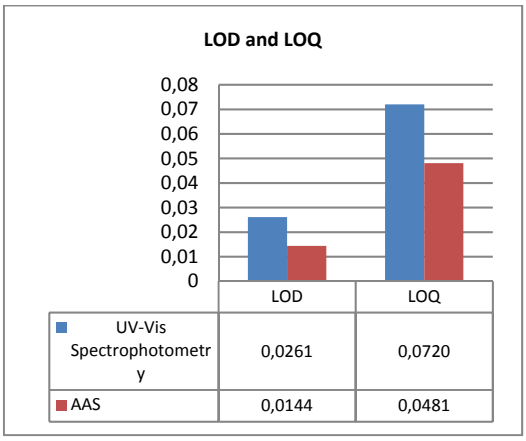

Figure 6: LOD and LOQ of copper examination

Obtained LOD and LOQ values in UV-Vis Spectrophotometry were 0.0216 and 0.0720, respectively while in AAS were 0.0144 and 0.0481. Level below 0.0216 was no longer detectable by UV-Vis Spectrophotometry and level below 0.0144 was no longer detectable by AAS, it is known that AAS has smaller LOD than UV-Vis Spectrophotometry so that AAS remains capable of detecting very small samples. The LOD and LOQ values met the specified requirements as no sample levels were below the LOD and LOQ values.

3.2 Copper Examination on Samples

Examination of copper in this study was conducted using two methods: UV-Vis Spectrophotometry with neocuproine complexing agent and AAS using aquadest solvent. Copper examination was conducted simultaneously on the same day and time for each sample, using a double beem UV-Vis Spectrophotometry and AAS, as well as glass apparatus including a separating funnel used for extraction in UV-Vis Spectrophotometry.

Samples made from SRM were heated by adding concentrated nitric acid performed in a fume hood. After heating in the Spectrophotometry UV-Vis method, neocuproine was added then extracted using chloroform and the extract was subsequently diluted using methanol, whereas in the AAS method, the sample filtered using filter paper and diluted using aquadest. Sample examination results of UV-Vis Spectrophotometry and AAS were shown in figure 7

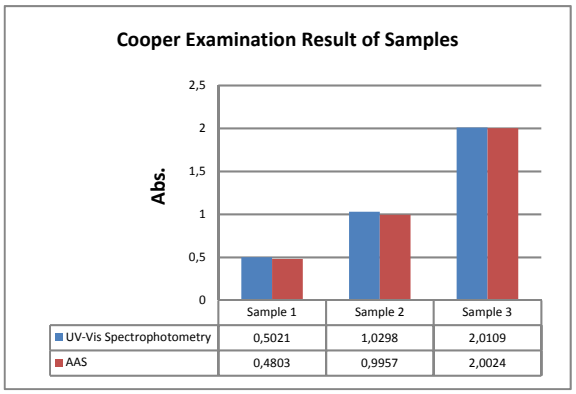

Figure 7: Copper examination result of samples

Above figure showed that the results of copper examination on the three samples using UV-Vis Spectrophotometry and AAS methods exhibited relatively no different results.

4. CONCLUSIONS

There are no difference in copper (Cu) examination results using UV-Vis Spectrophotometry with neocuproine complexing agent and AAS.

5. SUGGESTION

A one-time extraction and two-time extraction differences are required in copper examination using UV-Vis Spectrophotometry with neocuproine complexing agent. Copper examinations using samples taken from nature, for example: river water, well water and wastewater are necessary.

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