



THE DEVELOPMENT MODEL ANALYSIS OF COPPER USING VAPOR HEAT BY FORMATION COPPER HYDRIDE COMPOUNDS FOR HV-AAS METHOD

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ABSTRACT

The copper hydride is an intermediate hydride which decomposed to copper atom and hydrogen at temperature 110°C. In the research, resulted copper atom is measured on heated open absorption cell. The copper hydride was formed by reduction of Copper (II) with hypophosphorus acid 4% at temperature 80 °C. Sodium lauryl sulphate 0.01 M was used as a misel production to carry out copper hydride to absorption cell of hot vapour atomic absorption spectrometry (HV-AAS). The optimum condition of experiment were; 0.5 ml of H₃PO₂ 4%, 80±5°C of reaction temperature, 0.5 mL of sodium lauryl sulfat 0, 01 M, 60±5 second of formation time, and 15.35 cm long of glass a pipe. Argon or air for blowing was conducted at the base of flask reaction with 324.7 nm wave length measurement. The duration of measurement was ± 35 seconds. Regression equation of standard copper absorbance 5 to 30 mg/L was $Y = 0,026 + 0,0091x$. By using the equation, the characteristics of analytical procedure were found: 3.65 mg/L detection limit, 0.18 mg/L sensitivity. The accuracy of the procedure is 1.41%, its precision 10.59%, repeatability is 3.79 at 10 mg/L concentration of solution and average value of confidence interval 95% was 9.86 ± 0.8 mg/L or at range 9.1 to 10.7 mg/L. Linearity range standard of concentration is 5 to 25 mg/L (absorbance 0.088 to 0.258). The analysis procedure characteristics obtained with HV-AAS is less effective than flame atomic absorption spectrometry (FAAS) as its detection limit is 0.004 mg/L and its sensitivity is 0.03 mg/L.

Keyword: flameless atomic absorption spectrometry, copper, copper hydride.

INTRODUCTION

Copper is one of the hydride-forming elements include, in addition to seven hydride-forming elements among others are: magnesium, beryllium, zinc, cadmium, mercury, indium and thallium. According to Lee (1994), hydride compounds can be prepared by reducing salt compound with a strong reducing agent such as sodium borohydride, lithium aluminum hydride and hypophosphite acid.

Compounds belonging to the hydride-between in general have the nature unstable, volatile, can be decomposed at low temperatures, such as cadmium and mercury hydride hydride which can be decomposed at room temperature produce hydrogen gas and atomic cadmium and mercury, while copper hydride stable under 50°C and decomposes rapidly above 100 ° C, while zinc and beryllium metal hydride can be decomposed at temperatures above 120°C (Henslop, 1962).

Based on the properties of metastable-hydride compounds among these, some elements have been determined by atomic absorption spectrometry without flame. Medel in 1995 has successfully developed the technique of cold vapor atomic absorption spectrometry for the determination of cadmium metal through hydride formation, with the help of the surfactant as a carrier of the absorption cell to the reaction flask. Meanwhile, the hydride-forming elements other until now has never developed its determination by atomic absorption spectrometry technique without flame.

Copper hydride was first made by Duran (1962), by reducing the copper (II) of copper sulphate solution by using a

reducing agent hypophosphite acid, this compound shaped reddish brown precipitate. Copper hydride is stable below 50 ° C and can be decomposed rapidly at temperatures above 110°C.

Glancing properties of copper hydride instability of this, it is possible absorbannya measurements done by HV-AAS with quartz absorption cell lines T hot models, with the hope pengatoman can occur in cells that will be passed by the absorption of light cathode of atomic absorption spectrometry instruments.

METHODOLOGY

This research was carried out by flame atomic absorption spectrometry without absorption cell is open, the metal to be determined first made in the form of hydride in a three-neck flask were heated with oil bath lubricant to a temperature 80°C. Copper hydride formation using a solution of copper sulfate pentahydrate 1000 mg / L (stock solution) was added a few drops of nitric acid, and then reacted with sodium borohidrat 4% added a little hydrochloric acid, and hypophosphite acid 50% (stock solution).

The process of copper metal pengatoman hidridanya compound made in the heat absorption cell (ridden by element 450 watts) equipped with a temperature control. To bring the atoms / copper hydride is formed is used surfactant sodium lauryl sulfate. Between the reaction flask with absorption cells connected by a small U-shaped pipe that contains silikagel (water



absorbing). Absorbance measurements carried out at a wavelength of 324.7 nm

Atomization of copper with the help of sodium lauryl sulfate (NLS)

Standard solution of copper (II), 0.4 M hydrochloric acid and a solution of 0.01 M NLS put in a three-necked round flask were placed on a water bath. A solution of sodium borohydride and hypophosphite acid is injected through the neck of another. Volatile species formed as copper hydride compound decomposes and particles dispersed in the liquid form of copper hydride compounds, with the help of the NLS to the absorption cell which has been heated to a temperature of 120°C is placed on the beam atomic absorption spectrometry.

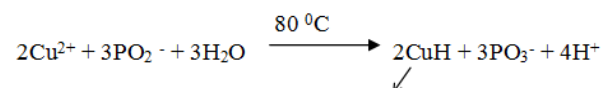
The optimum conditions to be determined are: reaction temperature, time copper hydride formation, the volume of surfactants, surfactant deployment time, temperature absorption cell and measurement time. Hypophosphite acid used is 5% H_3PO_2 (Merck). To determine the optimum conditions were used concentrations of copper (II) 10 mg / L were taken from the mother liquor 1000 mg / L. Standard calibration curve of copper (II) was prepared by diluting the stock standard solution of 1000 mg / L, diencekan be varied concentrations namely: 1.00; 5.00., 10.00; 15.00; 20.00; 25,00 and 30 mg / L. Then Measurements were made using optimum conditions HV-AAS predetermined.

RESULTS AND DISCUSSIONS

Qualitative analysis. Preliminary test of were conducted in the early stages of research, aiming to prove the formation of copper hydride (CuH) by reducing cupric sulphate Cu (II) with a reducing agent hypophosphite acid (H_3PO_2). The concentration of copper (II) used was 200 mg / L were diluted from the stock solution 1000 mg / L, and 50% hypophosphite acid. Reaction results showed that after heating to a temperature of 80°C, and allowed to stand a few minutes, when the temperature approached 80°C begins to form copper hydride, which is characterized by reddish-brown coloration. This color will began to lost if the temperature is raised above 100°C. The reaction occurs in the reaction flask is:



In the above reaction Cu^{2+} ions are reduced to Cu^+ , while phosphorus is oxidized from P^+ to P_3^+ , by releasing two elektron. Redox reactions that occur are;

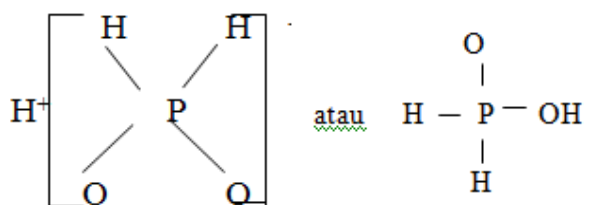


Reddish brown

From the macro-scale test showed that the manufacture of copper hydride can be done by reducing

the copper (II) by using a reducing agent hypophosphite acid, as ever undertaken by Baruer, (1954), Heslop, (1961) and Moeller, (1963). Hypophosphite acid is a strong reducing agent, these compounds can be used in reducing sediment aromatic diazonium, and more importantly used for the manufacture of copper hydride salt solution of copper (II). Hypophosphite acid is a tribasic acid, salt and acid form, easily soluble in water, the solution of alkali metal and in alcohol.

Acid-base properties of the hypophosphite be an indication that a hydrogen atom in the molecule is different from the other two hydrogen atoms, the structure of this compound tetrahedral shape, (Parkers, 1961).



the presence of two PH bond is what makes the compound hypophosphite acid H_2PO_2 is a strong reducing agent.

The condition optimum form measurements Copper using HV-AAS can be seen in Table-1 below:

Table-1. Conditions for determination of copper by atomic absorption spectrometry hot vapor.

Parameters	Condition unit
volume H_3PO_2 5 %	4 mL
reaction temperature	80±5°C
time Formation CuH	60 ± 5 sec
volume NLS	0,5 mL
time deployment NLS	30 ± 5 sec
blowing gas Argon	On basis reaction flask
temperature sel absorption	110 ± 10°C
length of pipe (U)	15,3 cm
silikagel	10,50 g
wavelength (λ)	324.7 nm
time measurement	30±5 sec

Determination Absorbance For Calibration curve Standard.

Standard curve was constructed based on the absorbance obtained from the measurement of several concentrations of standard solutions.

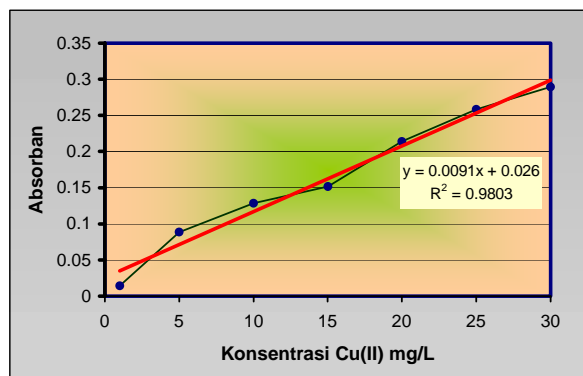


Figure-1. The standard curve of copper (II) for the HV-AAS.

Line obtained in the calibration curve, do not show good linearity, but statistically they can be said curve was linear, with the price of $R^2 = 0.9803$. Equation obtained from the standard curve of copper (II) is $Y = 0.0091x + 0.0026$. Major deviations occur is on measuring the absorbance with concentration of 15 mg / L, should the measured absorbance is greater than this value, it is due to the difficulty of controlling the variables that affect the measurement

Determination of performance characteristics analysis procedures

Analytical parameters determined to see the characteristics of the analysis procedure AAS-VHare among others: the limit of detection, sensitivity precision, accuracy and repeatability. To determine the analytical parameters absorbance measurements carried out on the concentration of copper (II) 10 mg / L as much as 10 repetitions. From the results of the determination of the parameters in the above analysis, it is known that the standard deviation in this study enough large, which resulted in the value of accuracy, precision ($9, 86 \pm 0, 80$ mg/L) and high repeatability. From the above table, it is known that the precision of the assay procedure copper absorption cell HV-AAS is 80,61 % with accuracy, 98, 6 % (to 10 times the standard concentration measurements at 10 mg / L), it indicates that the research process is still a professional errors or mistakes, especially in the determination of optimal conditions.

Detection limit and sensitivity obtained in the determination of copper content in HV-AAS is 3.65 mg / L and 0.18 mg / L, indicating that the analytical procedures used for the determination of copper content in HV-AAS has been able to measure up in the concentration range of 5 to 25 mg / L with a detection limit of 3.65 mg / L and sensitivity 0.01 mg / L, the value is not as good as compared to the flame AAS system, with a detection limit of 0.04 mg / L with a sensitivity of 0.03 mg / L, (Hanswell, 1991).

In the process of measuring the absorbance of copper (II) without flame atomic absorption spectrometry, an issue that always comes up is the increase in signal

from deuterium lamp / background is continuous, although already in conditioning your absorption cell in such a way and heated to temperatures higher than 150°C.

The increase in signal from background correction spectra showed that disruption caused by the absorption band width or spread of light (Dobson, 1998). Spreading light or wide band dispersion is caused by water vapor, or micelles are broken at the time of occurrence of copper hydride decomposition in heat absorption cell. Micelles derived from the process of transporting the copper hydride to the absorption cell, micelles consisting of sodium lauryl sulfate surfactant molecules and water molecules. At the time of micelles into the absorption cell rapid decomposition of copper hydride vapor releasing hydrogen and copper atoms, in addition to releasing water vapor and molecular micelles themselves, then there was the spread of the light of the deuterium lamp.

Due to the spread of the light signal deuterium lamp always rise with the increase in absorbance of copper, so that obtain the optimum measurement conditions is quite difficult and time consuming.

The advantage of a logama assay is technically flame atomic absorption spectrometry without hot vapor through the formation of this coating is quite easy and simple job, the number of samples a little, save the use of chemicals, do not use gas burners (more economical), and the possibility of chemical interference, matrix interference and interference small ionization.

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CONCLUSIONS AND RECOMMENDATIONS

Based on the research that has been done that the copper hydride can be prepared by the reduction of copper (II) with hypophosphite acid at a temperature of $80 \pm 5^\circ\text{C}$ and Techniques flame atomic absorption spectrometry without hot vapor (HV-AAS) can be used for the determination of copper content through hydride compound at a range of concentrations 5 to 25 mg / L. Absorbance measurements of copper through the formation of compounds in HV-AAS hydride influenced by; measurement time, reaction temperature, the volume of surfactant, the old copper hydride formation, the time spread of surfactant (sodium lauryl sulfate) and temperature absorption cell. Standard curve regression equation of copper content measurement in HV-AAS is $Y = 0.0091x + 0.0026$ and performance characteristics of the analysis procedure is; detection limit of 3.65 mg / L, the sensitivity of 0.18 mg / l, the precision of 80,61% with accuracy, 98, 6% and the repeatability of 3.79. This research is the development of methods of analysis of heavy metals, especially metal hydride forming between and still need further study to get good accuracy and validation, so that the model development this analysis method can be applied to the field in the measurement of heavy metals.

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