

## A new developed spectrophotometric method for the indirect determination of Aluminum in Al-Cu-Zn solder alloy

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### Abstract

Aluminum is used in numerous manufacturing products involving alloys because of its importance in developing physical and chemical properties. In this study, a sensitive, simple, economical, fast, and accurate indirect spectral analytical method was advanced using Eriochrome black T as ligand with abolition use of the separation and isolation to estimate the amount of Aluminum in the Al-Cu-Zn solder alloy by using UV-Visible spectrophotometer instrument. The method's accuracy was  $99.901 \pm 0.104$ , and the limit of detection (LOD) and qualification (LOQ) were 0.0961 and 0.2912  $\mu\text{g ml}^{-1}$ , respectively. The developed method can be employed for water, biological, environmental, and fuel samples.

**Keywords:** Solder alloy; Aluminum; Copper; Zinc; Eriochrome black T; Spectrophotometer

### 1. Introduction

The aluminum element is one of the most copious minerals in soil and stages third, with an estimated 8% of the land's crust, and its use, particularly in the automotive industry, is growing rapidly [1]. Aluminum and its alloys are considered one of the most important synthetics in the industrial field [2]. Certain the importance of welding in the manufacture of fridges, air conditioners, and heat exchangers, and due to the inimitable properties of aluminum, the demand for new methods of mixing aluminum with other metals in different proportions, such as copper and zinc, has increased. It has led to the fabrication of various kinds of aluminum alloys. The phenomena of occupying interstices were studied when the copper pieces were combined, aluminum together, and aluminum with copper using solder alloy [3]. Because of the lone qualities equipped by aluminum and its alloys, aluminum is second sole to steel used as a legitimate metal [4]. A painstaking analysis method has been developed to determine aluminum in numerous settings using one of the hydrazone derivatives[5]. A quick and observant method of mass molar analysis of aluminum mixture with elements was settled by using laser analysis spectroscopy [6]. By laser-induced breakdown spectroscopy, the amount of the aluminum element in the zinc composite was estimated [7]. By the EDTA addition, the problem of interfering zinc with aluminum at the determination of aluminum in different samples with the voltammetry method by a renewable electrode was resolved [8]. In a study, calibration-free laser spectroscopy was applied to analyze various samples of aluminum alloys and to estimate the amount of aluminum in them [9]. A solution based on several procedures was adopted to correct the signal changes resulting from the plasma reaction to decrease the effect of the medium and improve the accuracy of the quantitative estimate of the aluminum alloys and other metal alloys using laser spectroscopy[10]. Through in-electrode coulometric titration in a porous reticulated vitreous carbon electrode, a novel approach for the indirect detection of aluminum (III) in water has been established [11, 12]. Aluminum Determination in Food Products, Real Samples, and Standard Alloys was investigated using a liquid membrane sensor based on the interaction of aluminum ions with the reagent 2,9-dimethyl-4,11-diphenyl-1,5,8,12-tetraaza cyclote tradeca-1,4,8,11-tetraene [13]. Dual-cloud Point Extraction is used to preconcentrate aluminum, and Inductively Coupled Plasma-optical Emission Spectrometry is used to determine its concentration [14, 15].

In our study, a new indirect spectrophotometric method has been developed to measure the aluminum in solder alloy using Eriochrome Black T at a concentration greater than its quantification limit to eliminate signal interference with noise and to discover a decrease in the concentration of the reagent owing to its reaction with aluminum ions without difficulty by using UV-VIS spectrophotometer instrument.

### 2. Experimental

#### 2.1. Solutions of sample, working, standards, and reagent

All solutions were prepared with analytical grade reagents and freshly deionized distilled DI water. The solder alloy sample (95% (w/w) Cu, 4% (w/w) Zn, 1% (w/w) Al) was drilled, and about 0.5 grams of the fine drilling was accurately weighed and transferred to a 250 ml Erlenmeyer flask containing 9 ml of a 6-mol.  $\text{l}^{-1}$   $\text{HNO}_3$  solution and place the flask onto a hot plate with gentle boiling for 30 minutes or digest the sample with a microwave digester. After dissolution, the

solution was evaporated to a minor volume, chilled to room temperature, conveyed to a 100-ml volumetric flask, and marked up with DI water.

Stock solutions  $\mu\text{g}/\mu\text{l}$  of Al(III) and Zn(II), and  $10 \mu\text{g}/\mu\text{l}$  Cu(II) were prepared by dissolving (1.3905, 0.4548, and 3.8020 g) of aluminum nitrate nonahydrate, zinc nitrate hexahydrate and copper nitrate trihydrate respectively in DI water and made up to 100-ml volumetric flasks. Working solution (reference solution) was prepared by diluting 5, 20, and 47.5 ml of stock solutions  $\mu\text{g}/\mu\text{l}$  of the Al(III) and Zn(II) and  $10 \mu\text{g}/\mu\text{l}$  Cu(II) respectively, in a 100-ml volumetric flask. The reference solution has been used to create optimal conditions for maximum reliability and accuracy of the new method. Buffer solution pH 8.5 was prepared by mixing 0.2M ammonia and 0.2M  $\text{NH}_4\text{NO}_3$  solutions in suitable proportions, and the pH was adjusted using a pH meter. Stock standard solution  $\mu\text{g}/\mu\text{l}$  of Eriochrome BlackT (EBT) was prepared by transferring a (0.2 g) of (EBT) to a 200-ml volumetric flask using small portions of water. The total volume of water used was about 10 ml. Two milliliters of pH 8.5 buffer solution were added and diluted to 100 ml with absolute ethanol.

### 3. Procedure

To each flask in a set 50-ml volumetric flask, 5 ml of reference solution was added except the first flask, a variable volume of solution  $20 \mu\text{g}/\text{ml}$  Al(III) was added except the first flask and second flask, 2 ml of the solution (20% w/v) NaCN, 5 ml of the buffer solution pH 8.5 and 20 ml of the solution  $\mu\text{g}/\mu\text{l}$  (EBT) were added. Then all of the volumetric flasks were supplemented by deionized water to the marks. The blank solution was prepared using the same procedure without the additional solutions of EBT and aluminum. The absorbance of these solutions was measured at 590 nm. A straight line is obtained, which corresponds to the equation ( $A_{531} = 0.0112 C + 0.140$ . C is the amount of aluminum in  $\mu\text{g ml}^{-1}$ ). The developed method was applied to a live sample of solder alloy by using a sample solution in place of a reference solution to estimate the aluminum in the alloy.

### 4. Results and Discussion

The interference effect of the Cu and Zn matrix on the determination of Al was eliminated by using sodium cyanide as a masking agent. To ensure that there is no interference between the reagent EBT spectrum, the spectra of Cu(II) and Zn(II) cyanide complexes and Al(III)-EBT complex, the absorption spectrum was recorded for a solution containing a 5 ml of reference solution, 2 ml of solution (20% w/v) of NaCN, 5 ml of buffer solution pH 8.5, 20 ml of solution  $\mu\text{g}/\mu\text{l}$  of the (EBT) and diluted to 50 ml with deionized distilled water by using a UV-1600 Series by Shimadzu Scientific Instruments Inc. The blank solution was prepared using the same procedure without additional EBT and Aluminum solutions. The wavelengths of greatest absorption of EBT and Al(III)-EBT complex are 531 and 407 nm, respectively, as shown in Fig. 1 and Fig. 2 in this study. Moreover, the wavelengths of greatest absorption of complexes Cu(II) and Zn(II) cyanide are 341 and 300 nm, respectively [16].

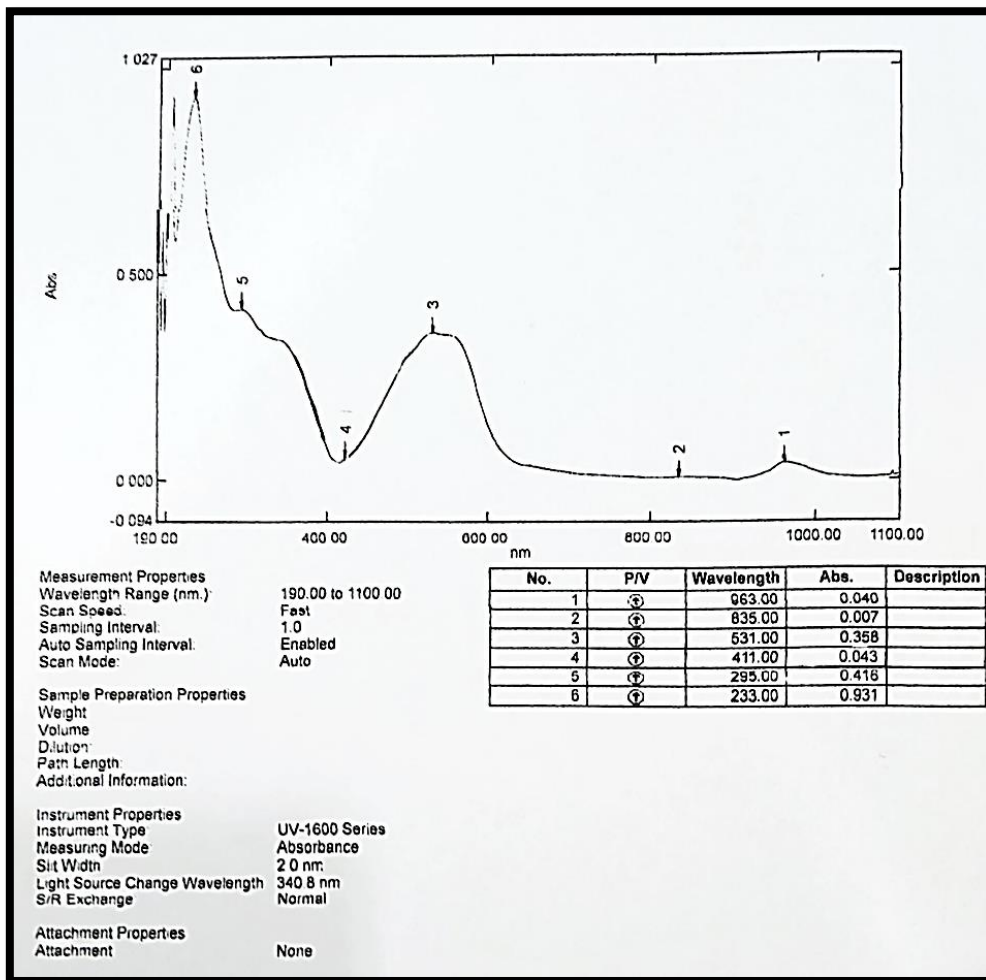


Figure 1. Spectroscopic scanning of EBT in UV-visible spectrophotometry

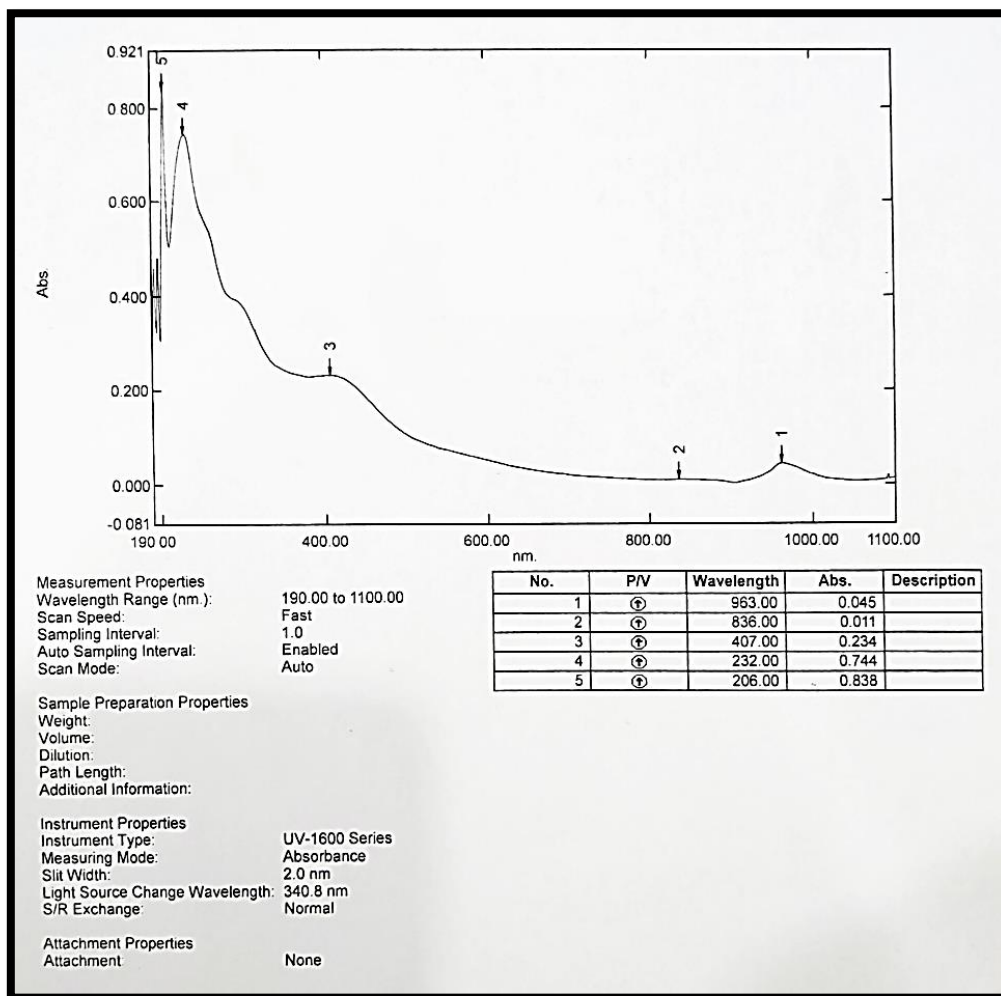


Figure 2. Spectroscopic scanning of Al(III)-EBT in UV-visible spectrophotometry

#### 4.1. Estimation of Aluminum quantity in solder alloy

Quantitative analysis of Aluminum in solder alloy is performed by measuring the decrease in the absorbance of the EBT using the new developed method based on the calculation of the difference between the absorbance of EBT at its initial concentration ( $A_{L_0}$ ) and its absorbance at residual concentration ( $A_{L_r}$ ) as a result of its reaction with Aluminum ions in the sample solution by using standard addition method and application of the following equation:

$$\text{Absorbance of Aluminum AML} = A_{L_0} - A_{L_r}$$

$$\text{Where } A_{L_0} = K C_{L_0}$$

$$A_{L_r} = K C_{L_r}$$

$C_{L_0}$  and  $C_{L_r}$  represent the initial and residual concentrations of EBT, respectively.

The absorbance of EBT at  $\lambda_{max}$  (531 nm) with a concentration (200 ppm) is equal (0.358), as shown in Fig.1. And the decrease in EBT concentration is illustrated in Fig. 3.

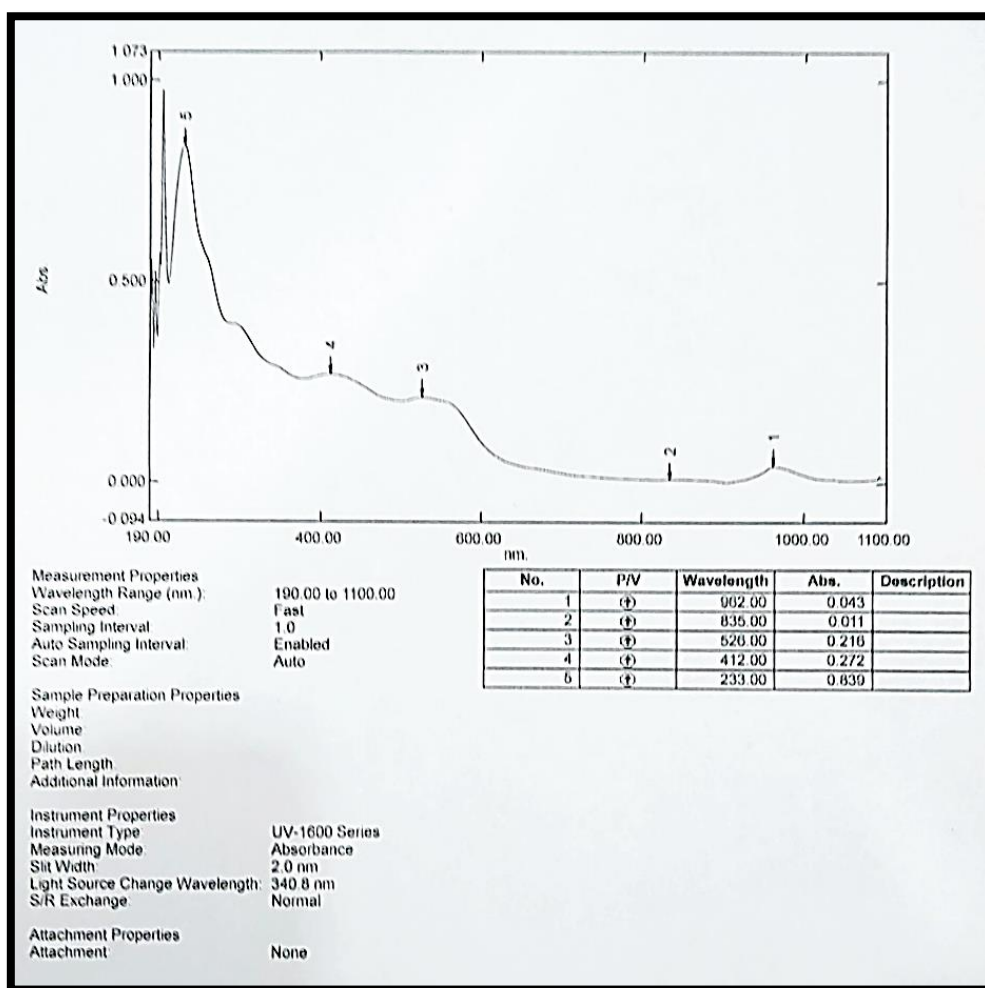


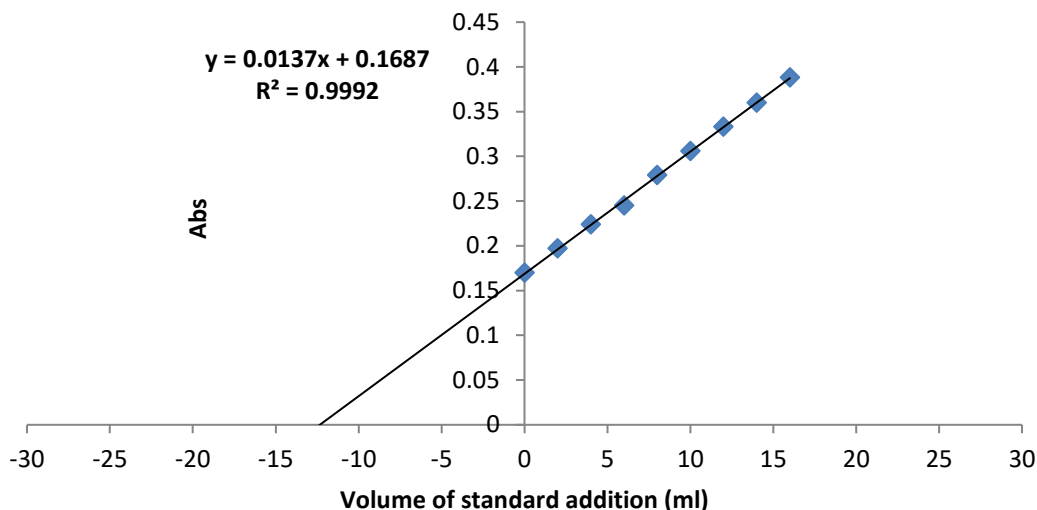
Figure 3. The spectrum of decreasing EBT absorbance and increasing of Al-EBT complex

One study indicated that [17] the molar ratio of Al:EBT is 1:1. This contributed to developing our new method for determining aluminum in the solder alloy, as shown in Table 1.

Table 1. Data were used to obtain absorbance of Al (III) as  $A_{ML} = A_{L_0} - A_{Lr}$

No.	Volume of sample ml	Volume of standard ml	Volume of EBT ml	$A_{L_0}$	$A_{Lr}$	$A_{ML}$
1	0	0	20	0.716	0.716	0
2	5	0	20		0.546	0.170
3	5	2	20		0.519	0.197
4	5	4	20		0.492	0.224
5	5	6	20		0.471	0.245

6	5	8	20		0.437	0.279
7	5	10	20		0.410	0.306
8	5	12	20		0.383	0.333
9	5	14	20		0.356	0.360
10	5	16	20		0.333	0.388



**Figure 4. Standard additions curve to determine the Aluminum in the preparative solder alloy solution using the newly developed spectral method**

In general (Serih, 2020):  $S = K \cdot C_a$

Where:  $C_a$  = concentration of analyte =  $(C_x \cdot V_x + C_s \cdot V_s) / V_t$

$K$  = constant

$S$  = instrument response (signal) =  $K \cdot (C_x \cdot V_x + C_s \cdot V_s) / V_t$

$V_x$  = volume of the sample aliquot

$C_x$  = concentration of the sample

$V_s$  = volume of standard

$C_s$  = concentration of the standard

$$S = K \cdot C_s \cdot V_s / V_t + K \cdot C_x \cdot V_x / V_t$$

From the linear regression:  $y = m \cdot x + b$

$$y = S$$

$$m = \text{slope} = K \cdot C_s / V_t$$

$$V_s = x$$

$$b = \text{intercept} = K \cdot C_x \cdot V_x / V_t$$

The concentration of analyte can be calculated by applying one of the two equation :

$$C_x = b \cdot C_s / m \cdot V_x \text{ or } C = (V_s)_0 \cdot C_s / V_x$$

Where  $(V_s)_0$  is the volume of standard when the device response is zero.

Then the percentage of analyte in the sample is calculated by the following equation :

$$Al \% = C_x \cdot V \cdot D.F \cdot 100 / W$$

Where  $C_x$  = Concentration of Aluminum ( $\mu\text{g/ml}$ ).

$V$  = Markup volume (ml)

D.F = dilution factor

$W$  = weight of sample

#### 4.2. Calculate the accuracy and precision of the developed method

To validate the developed analytical method, a series of standard solutions were prepared, and their absorption was measured according to the above procedure. The method's accuracy was estimated from the calculation of percentage recovery and its standard deviation. The standard error and deviation of intercept and the limits of detection (LOD) and quantification (LOQ) were also calculated from Fig. 5, as shown in Table 2.

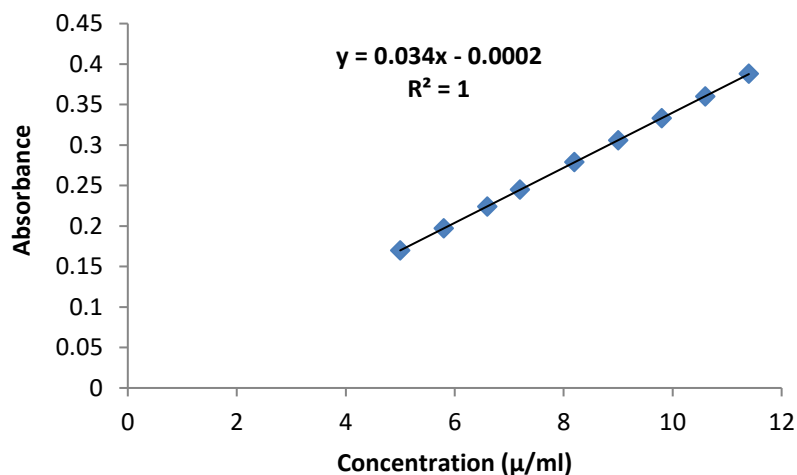


Figure 5. The calibration curve of the validation developed spectrophotometric analytical method

Table 2. Results of calibration curve analysis

Parameters	Value
Accuracy	99.901 ± 0.104
Slope	0.034
Intercept	2E-04
Linear range	0.1 – 40 µ ml <sup>-1</sup>
Correlation coefficient	1.00
SE of intercept	0.00033
SD of intercept	0.00099
LOD	0.09608
LOQ	0.29117

## 5. Conclusion

The modern method is a comprehensive analysis method for determining aluminum in many different samples, taking into account only the elements that interfere with the aluminum element during the estimation and quenching them using appropriate masking agents.

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