

# Estimation of Sulphur by Messenger Method Index.

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## ABSTRACT

The quantitative estimation of sulphur in organic compounds is an important aspect of elemental analysis in organic chemistry. The Messenger method is a classical and reliable analytical technique used for the determination of sulphur content. In this method, the organic compound is heated with fuming nitric acid in the presence of potassium chlorate, leading to the oxidation of sulphur into sulphate ions. The formed sulphate is then precipitated as barium sulphate using barium chloride solution, which is subsequently filtered, dried, and weighed. The amount of sulphur present in the sample is calculated gravimetrically from the mass of barium sulphate obtained.

This study focuses on the principles, procedure, and accuracy of the Messenger method in sulphur estimation. The method is advantageous due to its simplicity, reproducibility, and applicability to a wide range of organic compounds. However, careful control of experimental conditions is necessary to avoid errors caused by incomplete oxidation or loss of precipitate. The results demonstrate that the Messenger method provides reliable and precise estimation of sulphur when performed under controlled laboratory conditions.

**Keywords:** Sulphur Estimation, Messenger Method, Quantitative Analysis, Sulphur Determination, Analytical Chemistry

## INTRODUCTION

Sulphur is an essential non-metallic element widely present in organic and inorganic compounds, playing a significant role in various chemical, biological, and industrial processes. The accurate determination of sulphur content is crucial in fields such as pharmaceuticals, environmental analysis, agriculture, and petrochemical industries, where its concentration directly influences the quality and functionality of materials.

In analytical chemistry, several methods have been developed for the quantitative estimation of sulphur. Among these, the Messenger method is considered a reliable and classical technique for determining sulphur in organic compounds. This method is based on the principle of oxidizing sulphur present in a compound into sulphate form, which is then precipitated as barium sulphate using barium chloride. The precipitate is filtered, dried, and weighed to calculate the sulphur content gravimetrically.

The Messenger method is particularly valued for its simplicity, accuracy, and cost-effectiveness. It involves controlled combustion or oxidation of the sample in the presence of suitable reagents, often using an air condenser to ensure complete conversion and prevent loss of sulphur-containing gases. The formation of a stable, insoluble precipitate (barium sulphate) allows precise measurement, making the method suitable for routine laboratory analysis.

Despite the availability of modern instrumental techniques, the Messenger method continues to be widely used in academic and research laboratories due to its fundamental importance in understanding classical analytical procedures. It also provides a strong foundation for students and researchers in mastering gravimetric analysis and precipitation reactions.

This research focuses on the principles, procedure, and accuracy of sulphur estimation using the Messenger method, highlighting its applications, advantages, and limitations in comparison with other analytical techniques.

## LITERATURE REVIEW

The determination of sulphur has been an important subject in analytical chemistry for over a century, with various classical and modern methods developed to improve accuracy, sensitivity, and efficiency. Early analytical techniques were primarily based on gravimetric methods, where sulphur was converted into a stable compound such as barium sulphate and measured by weight. One of the earliest studies on sulphur determination in materials like iron and steel highlighted the reliability of gravimetric estimation, although it required careful handling and time-consuming procedures.

Over time, researchers introduced volumetric and titrimetric methods to simplify sulphur estimation. For instance, methods developed in the early and mid-20<sup>th</sup> century involved oxidation of sulphur to sulphate followed by titration, improving speed while maintaining acceptable accuracy. These techniques reduced experimental complexity but still required controlled conditions to avoid interference from other substances.

The Messenger method, a classical gravimetric approach, evolved from these foundational techniques. It relies on the oxidation of sulphur present in organic compounds into sulphate, which is then precipitated as barium sulphate. This approach is consistent with one of the oldest and most widely accepted principles of sulphur determination—precipitation of sulphate for gravimetric analysis. The method gained popularity due to its simplicity, cost-effectiveness, and suitability for routine laboratory analysis.

Further advancements in sulphur estimation introduced alternative gravimetric procedures, such as conversion of sulphur into copper sulphide for measurement. Studies have shown that such methods can detect sulphur in both micro and macro quantities with high sensitivity, demonstrating the versatility of gravimetric techniques. However, these methods may involve additional steps and reagents compared to the Messenger method.

In recent decades, modern instrumental techniques such as atomic absorption spectrometry (AAS), inductively coupled plasma mass spectrometry (ICP-MS), X-ray fluorescence (XRF), and ion chromatography have been developed to enhance sensitivity and reduce analysis time. These methods provide lower detection limits and higher precision but require expensive instrumentation and skilled operation. Consequently, classical methods like the Messenger method remain relevant in academic laboratories and for basic analytical studies.

Literature also highlights that the choice of sulphur estimation method depends on factors such as sample type, required accuracy, and available resources. While instrumental methods are preferred for advanced research and industrial applications, gravimetric methods continue to be widely used for educational purposes and routine analysis due to their reliability and fundamental importance.

In conclusion, the Messenger method represents a significant development in the evolution of sulphur estimation techniques. It combines the reliability of classical gravimetric analysis with relatively simple experimental procedures, making it a valuable method in both teaching and research laboratories.

### Principle of Messenger Method

The Messenger method is a classical gravimetric technique used for the quantitative estimation of sulphur present in organic compounds. The principle of this method is based on the complete oxidation of sulphur into sulphate form, followed by its precipitation and measurement.

In this method, the organic compound containing sulphur is heated with strong oxidizing agents (commonly fuming nitric acid) under controlled conditions. During this process, all the sulphur present in the compound is converted into sulphuric acid ( $H_2SO_4$ ). To ensure complete oxidation and to prevent the loss of volatile sulphur compounds, an air condenser is used.

After oxidation, the solution is treated with barium chloride ( $\text{BaCl}_2$ ). This leads to the formation of a white, insoluble precipitate of barium sulphate ( $\text{BaSO}_4$ ) according to the reaction:

**The precipitated barium sulphate is then:**

Filtered

Washed to remove impurities

Dried and ignited to a constant weight

Since barium sulphate is highly stable and insoluble, its mass can be accurately measured. The amount of sulphur in the original sample is then calculated from the weight of barium sulphate using stoichiometric relationships.

**Key Points of the Principle**

Sulphur is oxidized to sulphate form

Sulphate is precipitated as  $\text{BaSO}_4$

The method follows gravimetric analysis

Measurement is based on mass of precipitate

Ensures high accuracy and reliability

Thus, the Messenger method works on the fundamental concept of converting sulphur into a measurable, stable compound, allowing precise quantitative estimation.

**Apparatus Required for Messenger Method**

**The following apparatus are required for the estimation of sulphur by the Messenger method:**

Basic Laboratory Apparatus

Beakers (various sizes)

Conical (Erlenmeyer) flasks

Measuring cylinder

Glass rod (for stirring)

Funnel

Heating & Combustion Setup

Bunsen burner or heating mantle

Tripod stand

Wire gauze

Condensation System

Air condenser (essential to prevent loss of volatile sulphur compounds)

Filter paper (ashless preferred)

Filtration funnel

Vacuum filtration setup (optional for faster filtration)

Weighing Equipment

Analytical balance (high precision)

Weighing bottle or watch glass

Precipitation & Drying

Crucible (porcelain or silica)

Crucible tongs

Desiccator (to cool the precipitate and avoid moisture absorption)

Additional Equipment

Pipettes and burettes (for accurate measurement of reagents)

Wash bottle (distilled water)

Drying oven or muffle furnace (for drying/ignition of precipitate)

### **Chemicals Required for Messenger Method**

The following chemicals are required for the quantitative estimation of sulphur by the Messenger method:

#### **Primary Reagents**

Organic compound sample (containing sulphur)

Fuming nitric acid ( $\text{HNO}_3$ ) – used as a strong oxidizing agent to convert sulphur into sulphate

#### **Precipitating Agent**

Barium chloride solution ( $\text{BaCl}_2$ ) – used to precipitate sulphate ions as barium sulphate ( $\text{BaSO}_4$ )

#### **Supporting Chemicals**

Distilled water – for dilution, washing, and preparation of solutions

Hydrochloric acid ( $\text{HCl}$ ) – to maintain acidic conditions and prevent interference during precipitation

#### **Washing & Purification**

Hot distilled water – for washing the precipitate to remove impurities

Dilute hydrochloric acid – sometimes used to wash and purify the precipitate

Optional / Auxiliary Reagents

Silver nitrate ( $\text{AgNO}_3$ ) – to test completeness of washing (checks presence of chloride ions) Indicators (if required) – for monitoring reaction conditions

### **Experimental Procedure for Messenger Method**

The following step-by-step procedure is used for the quantitative estimation of sulphur by the Messenger method:

#### **Preparation of Sample**

Weigh accurately a known quantity of the organic compound containing sulphur using an analytical balance.

Transfer the sample into a clean, dry flask.

#### **Oxidation of Sulphur**

Add fuming nitric acid ( $\text{HNO}_3$ ) to the flask containing the sample.

Attach an air condenser to the flask to prevent loss of volatile sulphur compounds.

Heat the mixture gently on a burner or heating mantle.

Continue heating until the organic matter is completely oxidized and a clear solution is obtained.

During this process, sulphur present in the compound is converted into sulphuric acid ( $\text{H}_2\text{SO}_4$ ).

#### **Dilution**

Allow the solution to cool.

Dilute it with distilled water to reduce acidity and prepare it for precipitation.

#### **Precipitation of Sulphate**

Add a solution of barium chloride ( $\text{BaCl}_2$ ) slowly to the diluted solution with constant stirring.

A white precipitate of barium sulphate ( $\text{BaSO}_4$ ) is formed.

Heat the mixture gently to ensure complete precipitation.

Allow the precipitate to settle.

#### **Filtration**

Filter the precipitate using ashless filter paper.

Wash the precipitate several times with hot distilled water to remove impurities (like excess chloride ions).

#### **Drying and Ignition**

Transfer the filter paper along with precipitate into a weighed crucible.

Dry it in an oven and then ignite it at high temperature until a constant weight is obtained.

Cool the crucible in a desiccator to avoid moisture absorption.

### Weighing

Weigh the crucible containing barium sulphate (BaSO<sub>4</sub>) accurately.

Record the final constant weight.

### Calculation

Calculate the amount of sulphur present using the weight of BaSO<sub>4</sub> obtained.

Use the relation:

233 g of BaSO<sub>4</sub> ≡ 32 g of Sulphur

### 9. Observation and Calculation

### Observations

Table 1: Experimental Observations

Trial	W <sub>1</sub> : Empty Crucible (g)	W <sub>2</sub> : Crucible + BaSO <sub>4</sub> (g)	BaSO <sub>4</sub> (W <sub>2</sub> -W <sub>1</sub> ) (g)	Sample Weight (W) (g)
1	25.632	26.145	0.513	0.250
2	25.610	26.120	0.510	0.250
3	25.598	26.105	0.507	0.250

### Calculations

#### Molecular Weight Relation

BaSO<sub>4</sub> = 233 g/mol

Sulphur (S) = 32 g/mol

**233 g BaSO<sub>4</sub> = 32 g S**

#### Formula Used

$$\% \text{ Sulphur} = [(W_2 - W_1) \times 32 \times 100] / (233 \times W) \text{ Where}$$

- W<sub>1</sub> = weight of empty crucible
- W<sub>2</sub> = weight of crucible + BaSO<sub>4</sub>
- W = weight of organic sample
- 32 = atomic mass of sulphur
- 233 = molecular weight of BaSO<sub>4</sub>

Table 2: Calculation of Sulphur Content

Trial	BaSO <sub>4</sub> (g)	Sulphur (g) = (BaSO <sub>4</sub> × 32/233)	% Sulphur
1	0.513	0.0705	28.17

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2	0.510	0.0700	28.00
3	0.507	0.0696	27.83

#### Statistical Result

Mean % Sulphur = 28.00 %

Standard Deviation =  $\pm 0.17$  %

#### Final Result

### RESULT

The percentage of sulphur present in the given organic compound was determined using the Messenger method. The sulphur in the sample was oxidized to sulphate and precipitated as barium sulphate ( $\text{BaSO}_4$ ), which was then filtered, dried, ignited, and weighed accurately.

Three experimental trials were performed to ensure precision and reproducibility of the results. The calculated sulphur content showed close agreement among the trials with low standard deviation.

The percentage of sulphur in the given sample was found to be:

$28.00 \pm 0.17$  %

This indicates that the method provides reliable and consistent results when carried out under controlled laboratory conditions.

#### Advantages of Messenger Method

Provides accurate and reliable results

Based on gravimetric analysis (high precision)

Simple and easy to perform

Cost-effective (no expensive instruments required)

Uses easily available chemicals

Forms stable  $\text{BaSO}_4$  precipitate for accurate measurement

Suitable for basic laboratory setups

Helps in understanding fundamental concepts

Gives reproducible results when performed carefully

#### Limitations of Messenger Method

Time-consuming due to multiple steps

Requires complete oxidation, otherwise gives errors

Possible loss of sulphur vapours during heating

Interference from impurities may affect results

Needs careful filtration and washing

Chances of human errors in handling and weighing

Not suitable for trace (very low) sulphur estimation

Less efficient compared to modern instrumental methods

## CONCLUSION

The Messenger method is a classical and well-established technique for the quantitative estimation of sulphur in organic compounds. It is based on the fundamental principle of converting sulphur into sulphate through oxidation, followed by precipitation as barium sulphate, which is then measured gravimetrically. This method is widely appreciated for its accuracy, reliability, and simplicity, making it suitable for routine laboratory analysis and educational purposes.

Despite the availability of advanced instrumental techniques such as spectroscopic and chromatographic methods, the Messenger method continues to hold importance due to its cost-effectiveness and minimal requirement for sophisticated equipment. It also helps in developing a strong understanding of basic analytical concepts such as oxidation, precipitation, filtration, and gravimetric calculations.

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