Kinetic Stability and Operational Optimization of Turbidimetric Sulfate Analysis Based on SNI 6989.20:2019 Methodology

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Abstract.
The measurement of sulfate (SO_4^{2-}) levels using the SNI 6989.20:2019 turbidimetric method is highly dependent on the stability of the barium sulfate ($BaSO_4$) suspension. This study aims to evaluate the effect of waiting time (standing time) variations ranging from 5 to 35 minutes and sample container handling protocols (single vs. multi-Erlenmeyer) on the accuracy of analytical results at concentration levels of 20 mg/L and 60 mg/L. Measurements were conducted using a UV–Vis spectrophotometer at a wavelength of 420 nm. The results indicated that, at low concentrations (20 mg/L), the suspension remained relatively stable, with recovery values within the acceptable limits (90–110%) up to 35 minutes. Conversely, at high concentrations (60 mg/L), a significant decrease in recovery was observed after the 10-minute threshold, reaching 77.83% at 35 minutes due to particle sedimentation. The multi-Erlenmeyer protocol provided more accurate kinetic data compared to the single-Erlenmeyer approach, which tended to produce positively biased results due to mechanical re-suspension effects during repeated sampling.It is concluded that strict adherence to a 5-minute waiting time window is essential to ensure the validity of water quality monitoring, particularly for samples with high pollutant loads.

Keywords: *Sulfate, Turbidimetry, SNI 6989.20:2019, Waiting Time and Kinetic Stability.*

I. INTRODUCTION

Water is an essential natural resource that plays a fundamental role in sustaining human life, supporting economic development, and maintaining environmental ecosystem balance. One of the important chemical parameters used in water quality assessment is sulfate ion concentration (SO_4^{2-}). Sulfate is a naturally occurring polyatomic anion found in surface water and groundwater originating from mineral weathering, sulfide oxidation processes, and atmospheric deposition through precipitation mechanisms (Hem, 1985). In addition to natural sources, anthropogenic activities such as mining operations, industrial discharges, land-use conversion, and domestic wastewater release significantly contribute to elevated sulfate concentrations in aquatic environments (World Health Organization [WHO], 2017).

Excessive sulfate levels may negatively influence both water quality and human health. Organoleptically, high sulfate concentrations can produce a bitter taste and increase corrosion potential within water distribution systems (WHO, 2017). From a public health perspective, consumption of water containing elevated sulfate levels, particularly magnesium sulfate or sodium sulfate, may cause laxative effects and gastrointestinal disturbances (United States Environmental Protection Agency [EPA], 2012). Moreover, atmospheric sulfur dioxide (SO_2) can undergo oxidation reactions forming sulfate aerosols that contribute to respiratory health risks and acid rain formation, thereby affecting aquatic ecosystems and agricultural productivity (Seinfeld & Pandis, 2016).

Considering these impacts, national and international regulations have established permissible sulfate limits in water quality standards. Drinking water guidelines recommend a maximum sulfate concentration of 250 mg/L to ensure safety and consumer acceptability (WHO, 2017). In Indonesia, water quality regulation is standardized through SNI 6989.20:2019, which provides analytical procedures for sulfate determination using the turbidimetric method. Accurate monitoring of sulfate concentration is therefore essential to ensure regulatory compliance and support evidence-based environmental management decisions (American Public Health Association [APHA], 2017).

One of the most widely applied analytical techniques for sulfate determination is the turbidimetric method. This method relies on the formation of barium sulfate ($BaSO_4$) precipitates after the addition of barium chloride reagent to sulfate-containing samples. The suspended $BaSO_4$ particles generate turbidity that can be quantified spectrophotometrically based on light scattering principles (Skoog et al., 2014). The method is favored due to its operational simplicity, relatively high sensitivity, and adoption within standardized analytical procedures such as SNI 6989.20:2019.

Despite its widespread use, the analytical reliability of the turbidimetric method is strongly influenced by operational parameters, particularly the waiting time prior to absorbance measurement. The formation of $BaSO_4$ particles follows nucleation and colloidal growth kinetics that evolve over time. Variations in waiting time may alter particle size distribution, suspension stability, and scattering intensity, ultimately affecting analytical accuracy and reproducibility (Harris, 2016). Standard protocols generally recommend controlled stirring followed by a stabilization period before measurement; however, deviations frequently occur during routine laboratory practice (European Medicines Agency, 2006).

Previous studies have primarily focused on validation parameters such as linearity, detection limits, and precision, while empirical investigations examining the influence of waiting time variation on turbidity stability remain limited. This condition reveals a research gap concerning the kinetic stability of $BaSO_4$ suspensions during turbidimetric sulfate analysis. Therefore, this study aims to evaluate the effect of waiting time variation on sulfate determination using the turbidimetric method based on SNI 6989.20:2019. The findings are expected to strengthen the scientific basis of standardized analytical procedures, improve laboratory quality control performance, and enhance the reliability of environmental water quality monitoring.Water is an essential natural resource that plays a fundamental role in sustaining human life, supporting economic activities, and maintaining environmental ecosystem balance. One of the chemical indicators commonly used in water quality assessment is the concentration of sulfate ions (SO_4^{2-}). Sulfate is a polyatomic anion naturally present in surface water and groundwater as a result of weathering of sulfur-containing minerals, sulfide oxidation, and atmospheric deposition through precipitation processes (Hem, 1985). In addition to natural sources, elevated sulfate concentrations in aquatic environments are also influenced by anthropogenic activities such as mining operations, land-use conversion, domestic wastewater discharge, and industrial effluents lacking adequate treatment (World Health Organization [WHO], 2017).

II. METHODS

Research Design

This study employed an experimental quantitative approach to evaluate the effect of waiting time variation on sulfate determination using the turbidimetric method based on SNI 6989.20:2019. The experiment focused on assessing analytical accuracy, precision, and kinetic stability of barium sulfate suspension under controlled laboratory conditions. Two sulfate concentration levels (20 mg/L and 60 mg/L) were selected to represent low and high concentration systems.

Materials and Instruments

The instruments used included a UV–Vis spectrophotometer equipped with a 10 mm cuvette path length, 250 mL Erlenmeyer flasks, volumetric pipettes, a magnetic stirrer, stopwatch, and analytical balance. Reagents consisted of a 1000 mg/L sulfate standard solution, buffer solution containing 36% HCl and NaCl in propanol–glycerol medium, $BaCl_2 \cdot 2H_2O$ reagent, and deionized water.

Experimental Procedure: Turbidimetric sulfate analysis flowchart

Waiting Time Variation

Absorbance measurements were conducted at different waiting times. The reference condition followed the standard protocol of 5 ± 1 minutes after mixing. Additional measurements were performed at total waiting times of 6, 8, 10, 15, and 35 minutes after $BaCl_2$ addition.

Two preparation schemes were applied:

Single-Erlenmeyer system, where measurements were performed sequentially using the same sample solution. Multi-Erlenmeyer system, where each waiting-time variation was prepared simultaneously using independent sample solutions.

Measurement and Data Analysis

After the designated waiting period, the suspension was transferred into a cuvette and measured at 420 nm against a reagent blank. Absorbance values were converted into sulfate concentrations using the calibration curve. Percent recovery (%Recovery) was calculated as the ratio between measured and nominal concentrations. Each measurement was repeated at least twice to evaluate analytical precision expressed as relative standard deviation (RSD).

III. RESULT AND DISCUSSION

Result

This study evaluated the effect of waiting time variation prior to absorbance measurement on sulfate determination using the turbidimetric method based on SNI 6989.20:2019 (Badan Standardisasi Nasional, 2019). The scientific findings demonstrate that waiting time represents a critical operational variable controlling analytical accuracy, particularly through its influence on the kinetic stability of the barium sulfate ($BaSO_4$) suspension formed during precipitation.

The fundamental principle of turbidimetric analysis explains this phenomenon. Unlike molecular spectrophotometric methods, the analytical signal in turbidimetry originates from light scattering by suspended particles rather than molecular absorption in a homogeneous solution. Consequently, particle stability, size distribution, and dispersion state during the standing period directly determine measured absorbance intensity (Skoog et al., 2014; Harris, 2016). Variations in waiting time modify particle growth dynamics, aggregation rate, and sedimentation behavior, ultimately influencing analytical response consistency.

The calibration curve obtained in this study exhibited excellent linearity within the concentration range of 0–100 mg/L (Figure 1). The regression equation was $y = 0.0108x - 0.0144$ with a correlation coefficient $r = 0.9986$, confirming proportional proportionality between sulfate concentration and instrumental response. High linearity indicates that instrumental performance and analytical procedures were properly controlled; therefore, recovery variations observed in this study primarily originate from kinetic processes associated with suspension stability rather than instrumental error (American Public Health Association [APHA], 2017).

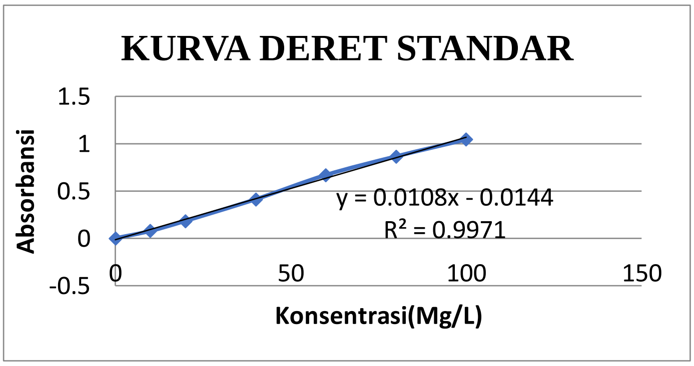

Fig. 1. Standard Calibration Curve for Sulfate Determination

Effect of Waiting Time on Low-Concentration Suspension (20 mg/L)

At a sulfate concentration of 20 mg/L, the $BaSO_4$ suspension exhibited relatively high kinetic stability. Recovery values remained within the acceptable analytical range of 90–110% even after extended observation up to 35 minutes. Lower particle density reduces interparticle collision frequency, minimizing aggregation and delaying sedimentation processes.

Minor fluctuations observed during measurement likely resulted from temporary nucleation events occurring during early precipitation stages. In precipitation-based analytical systems, secondary nucleation may temporarily increase turbidity before gravitational settling becomes dominant (Harris, 2016). Operationally, these findings indicate that low sulfate concentrations provide greater analytical flexibility, allowing moderate measurement delays without substantial accuracy loss. This behavior is relevant for environmental monitoring where sulfate levels are commonly low in natural waters (Hem, 1985).

Effect of Waiting Time on High-Concentration Suspension (60 mg/L)

In contrast, suspensions at 60 mg/L demonstrated significantly higher sensitivity to waiting time variation. Within the optimal measurement window (5–10 minutes), recovery values remained above 95%, indicating excellent analytical accuracy. However, recovery progressively decreased beyond this period, reaching 77.83% after 35 minutes.

This trend can be explained by precipitation kinetics. Higher sulfate concentrations increase supersaturation, accelerating nucleation and producing a larger number of particles during the initial reaction stage. Rapid nucleation initially enhances turbidity, but subsequent particle growth and agglomeration dominate system behavior. Larger aggregates possess greater mass, increasing sedimentation velocity and reducing suspension turbidity, which leads to decreased light scattering and lower measured absorbance values (Skoog et al., 2014).

The resulting effect produces a negative analytical bias, where measured sulfate concentrations become lower than actual values when measurement exceeds the optimal stabilization time. These findings confirm that turbidimetric systems at higher analyte concentrations are strongly governed by colloidal instability and sedimentation kinetics, emphasizing the importance of strict timing control in analytical procedures (APHA, 2017).

Influence of Sample Handling Protocol

Comparison between single-Erlenmeyer and multi-Erlenmeyer protocols revealed an additional important observation. Sequential measurements performed in a single reaction vessel tended to maintain higher recovery values than independent vessels. Repeated sampling and physical handling likely induced partial re-suspension of settled particles, artificially maintaining turbidity levels.

Conversely, the multi-Erlenmeyer approach provided a more realistic representation of suspension stability because each system evolved independently without mechanical disturbance. The observed recovery decline therefore reflects genuine physical degradation of the colloidal system rather than experimental artifacts. This finding highlights the necessity of minimizing physical disturbance during turbidimetric analysis to prevent pseudo-stability effects (Harris, 2016).

Comparison with Standard Method Requirements

The present results are consistent with standardized analytical recommendations emphasizing strict control of reaction timing and uniform analytical conditions between standards and samples (Badan Standardisasi Nasional, 2019; APHA, 2017). Analytical guidelines stress that identical reaction environments must be maintained to ensure comparability of scattering signals generated by $BaSO_4$ suspensions.

While previous studies primarily addressed validation parameters such as linearity and precision, this research contributes empirical evidence demonstrating that kinetic factors associated with waiting time constitute a major source of systematic analytical error. Therefore, this study expands the scientific basis of standardized turbidimetric sulfate determination.

Implications for Research Hypothesis

The research hypothesis proposed that variation in waiting time significantly influences the accuracy and precision of sulfate analysis using the turbidimetric method. Experimental findings clearly confirm this hypothesis. An optimal analytical window of approximately five minutes represents the period during which $BaSO_4$ suspension reaches maximum turbidity prior to sedimentation dominance.

Overall, the findings demonstrate that turbidimetric sulfate determination is governed not only by chemical reaction completion but also by time-dependent colloidal kinetics. Strict operational control is therefore essential to ensure valid environmental monitoring data and reliable laboratory quality assurance outcomes.

Discussion

Tab. 1. Percentage Recovery Of SO_4^{2-} For 20 Ppm And 60 Ppm Samples At Varying Holding Times

Waiting Time (minutes)	%Recovery (20 ppm, 1 Erlen.)	%Recovery (20 ppm, 6 Erlen.)	%Recovery (60 ppm, 1 Erlen.)	%Recovery (60 ppm, 6 Erlen.)
5 (reference)	109,8	107,3	102,4	102,6
6 (+1)	109,9	107,0	101,1	102,2
8 (+3)	101,4	111,5	98,8	99,2
10 (+5)	109,1	107,7	102,2	95,7
15 (+10)	106,7	107,6	96,6	89,1
35 (+30)	105,8	-	77,8	-

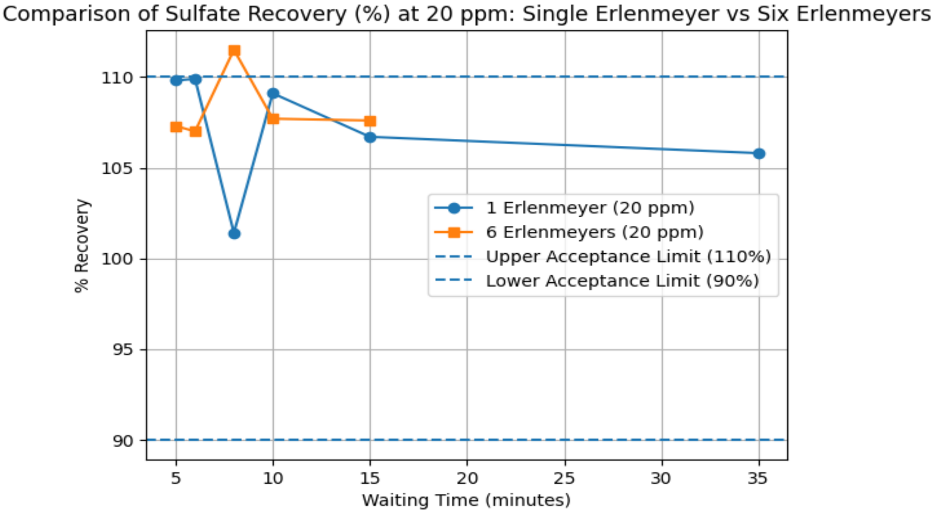

Fig 2. Comparison of % Recovery of Sulfate Test (20 ppm) 1 Erlenmeyer Vs 6 Erlenmeyer

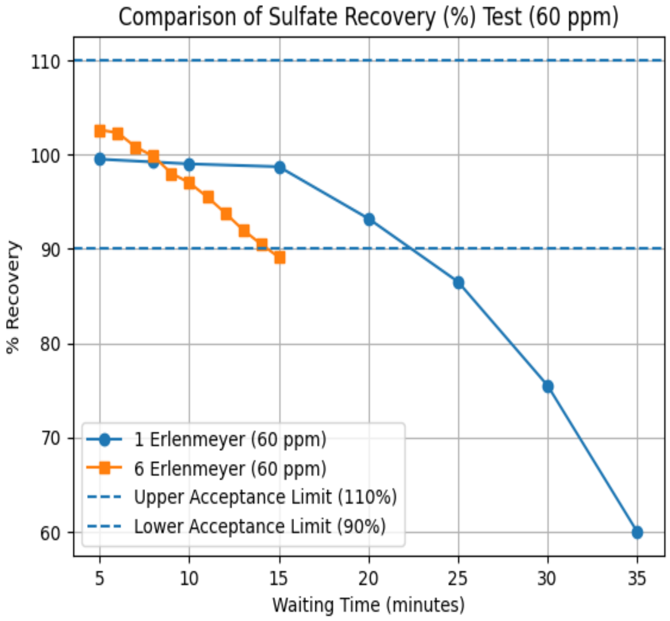

Fig. 3. Comparison of % Recovery of Sulfate Test (60 ppm) 1 Erlenmeyer Vs 6 Erlenmeyer

IV. CONCLUSION

Based on the conducted experiment and the analyzed data, the 60 ppm concentration using the six-Erlenmeyer method represents the most stable and acceptable condition for field application. This condition produced %Recovery values that generally fell within the acceptable standard range (90–110%), indicating good analytical accuracy and result stability. Although a slight decrease was observed at the 15th minute, the recovery value remained close to the lower acceptance limit and overall demonstrated consistent performance. In contrast, the single-Erlenmeyer method showed significant fluctuations and recovery values far below the acceptable range, likely caused by physical disturbances and inadequate mixing homogeneity. Therefore, the six-Erlenmeyer configuration at a concentration of 60 ppm is recommended for sulfate testing, as it provides more accurate, stable, and reliable results suitable for field implementation.The conclusion describes the answer to the hypothesis and/or the purpose of the study or scientific findings obtained. The conclusion does not contain a repetition of the results and discussion, but rather a summary of the findings as expected in the objectives or hypotheses

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