

Field Flocculation for Dissolved Sulphide

Technical Guidance for Sampling and Preservation

Dissolved sulphide is highly unstable in environmental samples and can be rapidly lost through oxidation or volatilization as hydrogen sulfide (H_2S). Accurate measurement therefore requires immediate field treatment at the time of collection to preserve sample integrity.

Dissolved vs Total Sulphide

Sulphide in water can be present in both dissolved and particulate forms.

- **Total sulphide** includes dissolved sulphide as well as sulphide associated with suspended solids
- **Dissolved sulphide** includes only the sulphide present in solution

Dissolved sulphide is defined as the fraction remaining after flocculation and settling, which separates particulate sulphides from the sample matrix. Field flocculation isolates this fraction at the time of sampling.

Results for dissolved sulphide and total sulphide may differ significantly depending on sample composition.

Field Flocculation Process Overview



1 Sample Collection



2 Add Reagents



3 Mix



4 Floc Formation



5 Settle



6 Decant & Preserve





Field Flocculation Procedure

Flocculation must be completed prior to preservation of the dissolved fraction.

1. Reagent Addition

Add the following to the 250 mL glass bottle containing the sample:

- 0.5 mL sodium hydroxide (6N)
- 0.5 mL aluminum chloride solution

Reagent volumes are based on AGAT kit design and may be adjusted if required to achieve proper flocculation and pH control.

2. Mixing

Cap the bottle and minimize headspace. Mix by rotating for approximately 1 minute to promote floc formation.

3. pH Control

Confirm that the sample pH is within the range of 6 to 9 following reagent addition. If necessary, pH should be adjusted to within range using appropriate dilute acid or base.

Adjustment reagents are not included in the kit. Two pH strips are provided, one for verification following flocculation and one for confirmation of preserved sample pH. Additional pH verification materials may be required depending on sampling conditions.

4. Settling

Allow the sample to settle until a clear supernatant is obtained, typically within 5 to 15 minutes. Avoid prolonged settling.

5. Decanting and Preservation

Decant approximately 60 mL of clear supernatant without disturbing the settled solids into the provided preserved container (zinc acetate and sodium hydroxide).

Verify that the preserved sample pH is ≥ 9 using the second provided pH strip. If the target pH is not met, contact AGAT for guidance.

Sampling and Handling Considerations

- Perform flocculation within 15 minutes of sample collection
- Minimize exposure to atmospheric oxygen during handling
- Avoid agitation or aeration
- Do not use filtration

Preservation with zinc acetate and sodium hydroxide stabilizes sulphide by forming insoluble zinc sulfide and maintaining alkaline conditions that prevent volatilization.

Store samples at $\leq 10^{\circ}\text{C}$ during transport.

Data Quality Considerations

Improper handling may result in low-biased sulphide concentrations due to oxidation, volatilization of H_2S , or disturbance of settled solids. Minimizing oxygen exposure during all steps is essential to maintaining data quality.

AGAT Flocculation Kit

Test Code 58540

Sulphide, Dissolved Flocculation Kit (10 Samples) — \$35.00

Available as:

Flocculation kit only

- 250 mL clear glass bottle
- Aluminum chloride (AlCl_3) solution
- Sodium hydroxide (NaOH, 6N) solution
- Dropper
- 2 X pH strips

Complete sampling kit

- All components above
- 60 mL plastic container pre-charged with zinc acetate and sodium hydroxide preservative

Summary

Field flocculation is a critical step in dissolved sulphide analysis. When performed in accordance with AGAT procedures, it ensures accurate separation of dissolved sulphide and supports reliable, defensible results for environmental assessment and regulatory compliance.