

Sampling Water for Dissolved Sulfide (DS) using SM 4500-S D

SAMPLING PROCEDURE

BEFORE YOU BEGIN

- (1) When possible, ALL pretreatment and preservation steps must be completed WITHIN 15 MINUTES OF SAMPLE COLLECTION.
- (2) Unless otherwise specified, fill bottles to the bottom of the neck of the bottle. DO NOT RINSE, UNDERFILL, OR OVERFILL.
- (3) Put on latex or nitrile gloves, and other appropriate PPE, during sampling.

SAMPLING STEPS

- (1) Remove the cap from the **250-mL Clear Glass Bottle w/NaOH**, and slowly collect the water sample in the bottle.
- (2) Using the dropper bottle, place 10 drops of the AlCl_3 solution into the bottle so the drops travel down into the water sample. Add just enough sample to this bottle so it is completely filled to the top.
- (3) Screw the Polycone cap onto the bottle so there is zero headspace (no air) in the bottle.
- (4) Rotate the bottle back and forth about a transverse axis vigorously for about 1 minute or longer to flocculate the contents. Set the bottle down, leave totally undisturbed, and allow the flocculent to begin to settle.
- (5) Just before 15 minutes has passed, when at least 2/3 of the sample is clear and the flocculent appears to have gravity-settled close to the bottom of the bottle, start decanting the clear upper layer of the sample SLOWLY into the **120-mL Poly Bottle w/NaOH & ZnAc**, so as NOT to create bubbles that will disturb the lower layer of flocculent. Continue filling the **120-mL Poly Bottle w/NaOH & ZnAc** up to its neck, but BE CAREFUL NOT to transfer any of the layer in which the flocculent appears!
- (6) Cap the **120-mL Poly Bottle w/NaOH & ZnAc** and submit it to the lab for DS analysis. Cap the **250-mL Clear Glass Bottle w/NaOH** and return it to the lab with the AlCl_3 dropper bottle for disposal.

FINAL STEPS

- (1) Record the sampling date, time, site, and name of sampler on the bottle label and the Chain-of-Custody (CoC) Record (FORM-N0013A).
- (2) Begin to chill sample containers on natural ice, and maintain between $>0\text{ }^\circ\text{C}$ to $6\text{ }^\circ\text{C}$ until transferred to the laboratory.

ACKNOWLEDGEMENT

I hereby acknowledge that I ___ have or ___ have not (check one) collected all submitted samples for dissolved sulfide (DS) as summarized above. I understand that not collecting samples using the above procedure may jeopardize the validity of any results obtained.

(Signature)

(Date)

Submit this document with the completed Chain-of-Custody Record that accompanies samples.

Attachment 1
Reference Information for
Sampling Water for Dissolved Sulfide (DS) using SM 4500-S D

Bottles & Supplies in "DS Kit":

- (1) 250-mL (8-oz) Clear Glass Boston round containing NaOH, with Polycarbonate cap
- (2) 3-mL or 4-mL Poly dropper containing 38% aluminum chloride (AlCl_3) solution, with colored cap
- (3) 120-mL Poly containing NaOH and ZnAc (NOTE: Same bottle type is used for total sulfide.)

Preservatives and/or Preservation Solutions:

- (1) AlCl_3 (38% solution): Completely dissolve 500-g bottle of $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ in 720 mL of polished deionized water (PDI). Store in a labeled 1-L glass bottle. Transfer ~2 mL of this solution into labeled 3-mL dropper bottles as needed. Set "Expires:" date on label as 3 months from transfer date.
- (2) NaOH (5N solution): Carefully dissolve 200 g of NaOH pellets in 800 mL of PDI. Use CAUTION: The dissolving process is extremely exothermic. When cooled, bring the final volume to 1.00 L with PDI. Store in a labeled 1-L glass bottle. Transfer into the Dispensette bottle as needed.
- (3) ZnAc (2M solution): Dissolve 44 g of zinc acetate $\text{Zn}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$ in 174 mL of PDI. This will make 200 mL of solution. Store in a 250 mL glass bottle.