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 AlCl₃ 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 AlCl₃ 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 °C to 6 °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.
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 Polycone cap
(2) 3-mL or 4-mL Poly dropper containing 38% aluminum chloride (AlCl₃) 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) AlCl₃ (38% solution): Completely dissolve 500-g bottle of AlCl₃ • 6H₂O 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 Zn(C₂H₃O₂)₂ • 2H₂O in 174 mL of PDI. This will make 200 mL of solution. Store in a 250 mL glass bottle.