REVISIONS

METHOD 9030A

ACID-SOLUBLE AND ACID-INSOLUBLE SULFIDES

1.0 SCOPE AND APPLICATION

1.1 The distillation procedure described in this method is designed for the determination of sulfides in aqueous, solid waste materials, or effluents.

1.2 This method provides only a semi-quantitative determination of sulfide compounds considered "acid-insoluble" (e.g., CuS and SnS_2) in solid samples. Recovery has been shown to be 20 to 40% for CuS, one of the most stable and insoluble compounds, and 40 to 60% for SnS_2 which is slightly more soluble.

1.3 This method is not applicable to oil or multiphasic samples or samples not amenable to the distillation procedure which can be analyzed by Method 9031.

1.4 Method 9030 is suitable for measuring sulfide concentrations in samples which contain between 0.2 and 50 mg/kg of sulfide.

1.5 This method is not applicable for distilling reactive sulfide, however, Method 9030 is used to quantify the concentration of sulfide from the reactivity test. Refer to Chapter Seven, Step 7.3.4.1 for the determination of reactive sulfide.

1.6 This method measures total sulfide which is usually defined as the acid-soluble fraction of a waste. If, however, one has previous knowledge of the waste and is concerned about both soluble sulfides such as H_2S, and metal sulfides, such as CuS and SnS_2, then total sulfide is defined as the combination of both acid-soluble and acid-insoluble fractions. For wastes where only metal sulfides are suspected to be present, only the acid-insoluble fraction needs to be performed.

2.0 SUMMARY OF METHOD

2.1 For acid-soluble sulfide samples, separation of sulfide from the sample matrix is accomplished by the addition of sulfuric acid to the sample. The sample is heated to 70°C and the hydrogen sulfide (H_2S) which is formed is distilled under acidic conditions and carried by a nitrogen stream into zinc acetate gas scrubbing bottles where it is precipitated as zinc sulfide.

2.2 For acid-insoluble sulfide samples, separation of sulfide from the sample matrix is accomplished by suspending the sample in concentrated hydrochloric acid by vigorous agitation. Tin(II) chloride is present to prevent oxidation of sulfide to sulfur by the metal ion (as in copper(II)), by the matrix, or by dissolved oxygen in the reagents. The prepared sample is distilled under acidic conditions at 100°C under a stream of nitrogen. Hydrogen sulfide gas is released from the sample and collected in gas scrubbing bottles containing zinc(II) and a strong acetate buffer. Zinc sulfide precipitates.

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2.3 The sulfide in the zinc sulfide precipitate is oxidized to sulfur with a known excess amount of iodine. Then the excess iodine is determined by titration with a standard solution of phenyl arsine oxide (PAO) or sodium thiosulfate until the blue iodine starch complex disappears. As the use of standard sulfide solutions is not possible because of oxidative degradation, quantitation is based on the PAO or sodium thiosulfate.

3.0 INTERFERENCES

3.1 Aqueous samples must be taken with a minimum of aeration to avoid volatilization of sulfide or reaction with oxygen, which oxidizes sulfide to sulfur compounds that are not detected.

3.2 Reduced sulfur compounds, such as sulfite and hydrosulfite, decompose in acid, and may form sulfur dioxide. This gas may be carried over to the zinc acetate gas scrubbing bottles and subsequently react with the iodine solution yielding false high values. The addition of formaldehyde into the zinc acetate gas scrubbing bottles removes this interference. Any sulfur dioxide entering the scrubber will form an addition compound with the formaldehyde which is unreactive towards the iodine in the acidified mixture. This method shows no sensitivity to sulfite or hydrosulfite at concentrations up to 10 mg/kg of the interferant.

3.3 Interferences for acid-insoluble sulfides have not been fully investigated. However, sodium sulfite and sodium thiosulfate are known to interfere in the procedure for soluble sulfides. Sulfur also interferes because it may be reduced to sulfide by tin(II) chloride in this procedure.

3.4 The iodometric method suffers interference from reducing substances that react with iodine, including thiosulfate, sulfite, and various organic compounds.

3.5 The insoluble method should not be used for the determination of soluble sulfides because it can reduce sulfur to sulfide, thus creating a positive interference.

4.0 APPARATUS AND MATERIALS

4.1 Gas evolution apparatus as shown in Figure 1

4.1.1 Three neck flask - 500-mL, 24/40 standard taper joints.

4.1.2 Dropping funnel - 100-mL, 24/40 outlet joint.

4.1.3 Purge gas inlet tube - 24/40 joint, with coarse frit.

4.1.4 Purge gas outlet - 24/40 joint reduced to 1/4 in. tube.

4.1.5 Gas scrubbing bottles - 125-mL, with 1/4 in. o.d. inlet and outlet tubes. Impinger tube must be fritted.

4.1.6 Tubing - 1/4 in. o.d. Teflon or polypropylene. Do not use rubber.

NOTE: When analyzing for acid-insoluble sulfides, the distillation apparatus is identical to that used in the distillation procedure for acid-soluble sulfides except that the tubing and unions downstream of the distillation flask must be all Teflon, polypropylene or other material resistant to gaseous HCl. The ground glass joints should be fitted with Teflon sleeves to prevent seizing and to prevent gas leaks. Pinch clamps should also be used on the joints to prevent leaks.

4.2 Hot plate stirrer.

4.3 pH meter.

4.4 Nitrogen regulator.

4.5 Flowmeter.

4.6 Top-loading balance - capable of weighing 0.1 g.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Reagent water. All references to water in this method refer to reagent water, as defined in Chapter One.

5.3 Zinc acetate solution for sample preservation (2N), $Zn(CH_3COO)_2 \cdot 2H_2O$. Dissolve 220 g of zinc acetate dihydrate in 500 mL of reagent water.

5.4 Sodium hydroxide (1N), NaOH. Dissolve 40 g of NaOH in reagent water and dilute to 1 liter.

5.5 Formaldehyde (37% solution), CH_2O. This solution is commercially available.

5.6 Zinc acetate for the scrubber

5.6.1 For acid-soluble sulfides: Zinc acetate solution (approximately 0.5M). Dissolve about 110 g zinc acetate dihydrate in 200 mL of reagent water. Add 1 mL hydrochloric acid (concentrated), HCl, to prevent precipitation of zinc hydroxide. Dilute to 1 liter.

5.6.2 For acid-insoluble sulfides: Zinc acetate/sodium acetate buffer. Dissolve 100 g sodium acetate, $NaC_2H_3O_2$, and 11 g zinc acetate dihydrate in 800 mL of reagent water. Add 1 mL concentrated hydrochloric acid and dilute to 1 liter. The resulting pH should be 6.8.

5.7 Acid to acidify the sample

5.7.1 For acid-soluble sulfides: Sulfuric acid (concentrated), H_2SO_4.

5.7.2 For acid-insoluble sulfides: Hydrochloric acid (9.8N), HCl. Place 200 mL of reagent water in a 1-liter beaker. Slowly add concentrated HCl to bring the total volume to 1 liter.

5.8 Starch solution - Use either an aqueous solution or soluble starch powder mixtures. Prepare an aqueous solution as follows. Dissolve 2 g soluble starch and 2 g salicylic acid, $C_7H_6O_3$, as a preservative, in 100 mL hot reagent water.

5.9 Nitrogen.

5.10 Iodine solution (approximately 0.025N)

5.10.1 Dissolve 25 g potassium iodide, KI, in 700 mL of reagent water in a 1-liter volumetric flask. Add 3.2 g iodine, I_2. Allow to dissolve. Add the type and amount of acid specified in Step 7.3.2. Dilute to 1 liter and standardize as follows.

5.10.2 Dissolve approximately 2 g KI in 150 mL of reagent water. Add exactly 20 mL of the iodine solution (Step 5.10.1) to be titrated and dilute to 300 mL with reagent water.

5.10.3 Titrate with 0.025N standardized phenylarsine oxide or 0.025N sodium thiosulfate until the amber color fades to yellow. Add starch indicator solution. Continue titration drop by drop until the blue color disappears.

5.10.4 Run in replicate.

5.10.5 Calculate the normality as follows.

$$\text{Normality } (I_2) = \frac{\text{mL of titrant x normality of titrant}}{\text{sample size in mL}}$$

5.11 Sodium sulfide nonanhydrate, $Na_2S \cdot 9H_2O$. For the preparation of standard solutions to be used for calibration curves. Standards must be prepared at pH > 9 and < 11. Protect standard from exposure to oxygen by preparing it without headspace. These standards are unstable and should be prepared daily.

5.12 Tin(II) chloride, $SnCl_2$, granular.

5.13 Titrant.

5.13.1 Standard phenylarsine oxide solution (PAO) (0.025N), C_6H_5AsO. This solution is commercially available.

CAUTION: PAO is toxic.

5.13.2 Standard sodium thiosulfate solution (0.025N), $Na_2S_2O_3 \cdot 5H_2O$. Dissolve 6.205 ± 0.005 g $Na_2S_2O_3 \cdot 5H_2O$ in 500 mL reagent water. Add 9 mL 1N NaOH and dilute to 1 liter.

5.14 Sodium hydroxide (6N), NaOH. Dissolve 240 g of sodium hydroxide in 1 liter of reagent water.

5.15 Hydrochloric acid (6N), HCl. Place 51 mL of reagent water in a 100 mL Class A volumetric flask. Slowly add concentrated HCl to bring the total volume to 100 mL.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

6.2 All aqueous samples and effluents must be preserved with zinc acetate and sodium hydroxide. Use four drops of 2N zinc acetate solution per 100 mL of sample. Adjust the pH to greater than 9 with 6N sodium hydroxide solution. Fill the sample bottle completely and stopper with a minimum of aeration. The treated sample is relatively stable and can be held for up to seven days. If high concentrations of sulfide are expected to be in the sample, continue adding zinc acetate until all the sulfide has precipitated. For solid samples, fill the surface of the solid with 2N zinc acetate until moistened. Samples must be cooled to 4°C and stored headspace free.

6.3 Sample Preparation

6.3.1 For an efficient distillation, the mixture in the distillation flask must be of such a consistency that the motion of the stirring bar is sufficient to keep the solids from settling. The mixture must be free of solid objects that could disrupt the stirring bar. Prepare the sample using one of the procedures in this section then proceed with the distillation step (Section 7.0).

6.3.2 If the sample is aqueous, shake the sample container to suspend any solids, then quickly decant the appropriate volume (up to 250 mL) of the sample to a graduated cylinder, weigh the cylinder, transfer to the distillation flask and reweigh the cylinder to the nearest milligram. Be sure that a representative aliquot is used, or use the entire sample.

6.3.3 If the sample is aqueous but contains soft clumps of solid, it may be possible to break the clumps and homogenize the sample by placing the sample container on a jar mill and tumble or roll the sample for a few hours. The slurry may then be aliquotted and weighed as above to the nearest milligram then diluted with reagent water up to a total volume of 250 mL to produce a mixture that is completely suspended by the stirring bar.

6.3.4 If the sample is primarily aqueous, but contains a large proportion of solid, the sample may be roughly separated by phase and the amount of each phase measured and weighed to the nearest milligram into

the distillation flask in proportion to their abundance in the sample. Reagent water may be added up to a total volume of 250 mL. As a guideline, no more than 25 g dry weight or 50 g of sludge can be adequately suspended in the apparatus.

6.3.5 If the sample contains solids which absorb water and swell, limit the sample size to 25 g dry weight. Otherwise, the solids will restrict the fluid motion and lower the recovery.

6.3.6 If the sample contains solid objects that cannot be reduced in size by tumbling, the solids must be broken manually. Clay-like solids should be cut with a spatula or scalpel in a crystallizing dish. If the solids can be reduced to a size that they can be suspended by the stirring bar, the solid and liquid can be proportionately weighed.

6.3.7 Non-porous harder objects, for example stones or pieces of metal, may be weighed and discarded. The percent weight of non-porous objects should be reported and should be used in the calculation of sulfide concentration if it has a significant effect on the reported result.

7.0 PROCEDURE

For acid-soluble sulfide samples, go to 7.1
For acid-insoluble sulfide samples, go to 7.2

7.1 Acid-Soluble Sulfide

7.1.1 In a preliminary experiment, determine the approximate amount of sulfuric acid required to adjust a measured amount of the sample to pH less than or equal to 1. The sample size should be chosen so that it contains between 0.2 and 50 mg of sulfide. Place a known amount of sample or sample slurry in a beaker. Add reagent water until the total volume is 200 mL. Stir the mixture and determine the pH. Slowly add sulfuric acid until the pH is less than or equal to 1. Discard this preliminary sample.

CAUTION: Toxic hydrogen sulfide may be generated from the acidified sample. This operation must be performed in the hood and the sample left in the hood until the sample has been made alkaline or the sulfide has been destroyed. From the amount of sulfuric acid required to acidify the sample and the mass or volume of the sample acidified, calculate the amount of acid required to acidify the sample to be placed in the distillation flask.

7.1.2 Prepare the gas evolution apparatus as shown in Figure 1 in a fume hood.

7.1.2.1 Prepare a hot water bath at 70°C by filling a crystallizing dish or other suitable container with water and place it on a hot plate stirrer. Place a thermometer in the bath and monitor the temperature to maintain the bath at 70°C.

7.1.2.2 Assemble the three neck 500-mL flask, fritted gas inlet tube, and exhaust tube. Use Teflon sleeves to seal the ground glass joints. Place a Teflon coated stirring bar into the flask.

7.1.2.3 Place into each gas scrubbing bottle 10 ± 0.5 mL of the 0.5M zinc acetate solution, 5.0 ± 0.1 mL of 37% formaldehyde and 100 ± 5.0 mL reagent water.

7.1.2.4 Connect the gas evolution flask and gas scrubbing bottles as shown in Figure 1. Secure all fittings and joints.

7.1.3 Carefully place an accurately weighed sample which contains 0.2 to 50 mg of sulfide into the flask. If necessary, dilute to approximately 200 mL with reagent water.

7.1.4 Place the dropping funnel onto the flask making sure its stopcock is closed. Add the volume of sulfuric acid calculated in Step 7.1.1 plus an additional 50 mL into the dropping funnel. The bottom stopcock must be closed.

7.1.5 Attach the nitrogen inlet to the top of the dropping funnel gas shut-off valve. Turn on the nitrogen purge gas and adjust the flow through the sample flask to 25 mL/min. The nitrogen in the gas scrubbing bottles should bubble at about five bubbles per second. Nitrogen pressure should be limited to approximately 10 psi to prevent excess stress on the glass system and fittings. Verify that there are no leaks in the system. Open the nitrogen shut-off valve leading to the dropping funnel. Observe that the gas flow into the sample vessel will stop for a short period while the pressure throughout the system equalizes. If the gas flow through the sample flask does not return within a minute, check for leaks around the dropping funnel. Once flow has stabilized, turn on magnetic stirrer. Purge system for 15 minutes with nitrogen to remove oxygen.

7.1.6 Heat sample to 70°C. Open dropping funnel to a position that will allow a flow of sulfuric acid of approximately 5 mL/min. Monitor the system until most of the sulfuric acid within the dropping funnel has entered the sample flask. Solids which absorb water and swell will restrict fluid motion and, therefore, lower recovery will be obtained. Such samples should be limited to 25 g dry weight.

7.1.7 Purge, stir, and maintain a temperature of 70°C for a total of 90 minutes from start to finish. Shut off nitrogen supply. Turn off heat.

7.1.8 Proceed to Step 7.3 for the analysis of the zinc sulfide by titration.

7.2 Acid-Insoluble Sulfide

7.2.1 As the concentration of HCl during distillation must be within a narrow range for successful distillation of H_2S, the water content must be controlled. It is imperative that the final concentration of HCl in the distillation flask be about 6.5N and that the sample is mostly suspended in the fluid by the action of the stirring bar. This is achieved by adding 50 mL of reagent water, including water in the sample, 100 mL of 9.8N HCl, and the sample to the distillation flask. Solids which absorb water and swell will restrict fluid motion and, therefore, lower recovery will be obtained. Such samples should be limited to 25 g dry weight. Other samples can range from 25 to 50 g.

7.2.2 If the matrix is a dry solid, weigh a portion of the sample such that it contains 0.2 to 50 mg of sulfide. The solid should be crushed to reduce particle size to 1 mm or less. Add 50 mL of reagent water.

7.2.3 If the matrix is aqueous, then a maximum of 50 g of the sample may be used. No additional water may be added. As none of the target compounds are volatile, drying the sample may be preferable to enhance the sensitivity by concentrating the sample. If less than 50 g of the sample is required to achieve the 0.2 to 50 mg of sulfide range for the test, then add reagent water to a total volume of 50 mL.

7.2.4 If the matrix is a moist solid, the water content of the sample must be determined (Karl Fischer titration, loss on drying, or other suitable means) and the water in the sample included in the total 50 mL of water needed for the correct HCl concentration. For example, if a 20 g sample weight is needed to achieve the desired sulfide level of 0.2 to 50 mg and the sample is 50% water then 40 mL rather than 50 mL of reagent water is added along with the sample and 100 mL of 9.8N HCl to the distillation flask.

7.2.5 Weigh the sample and 5 g $SnCl_2$ into the distillation flask. Use up to 50 mL of reagent water, as calculated above, to rinse any glassware.

7.2.6 Assemble the distillation apparatus as in Figure 1. Place 100 $\pm$ 2.0 mL of zinc acetate/sodium acetate buffer solution and 5.0 $\pm$ 0.1 mL of 37% formaldehyde in each gas scrubbing bottle. Tighten the pinch clamps on the distillation flask joints.

7.2.7 Make sure the stopcock is closed and then add 100 $\pm$ 1.0 mL of 9.8N HCl to the dropping funnel. Connect the nitrogen line to the top of the funnel and turn the nitrogen on to pressurize the dropping funnel headspace.

7.2.8 Set the nitrogen flow at 25 mL/min. The nitrogen in the gas scrubbing bottles should bubble at about five bubbles per second. Purge the oxygen from the system for about 15 minutes.

7.2.9 Turn on the magnetic stirrer. Set the stirring bar to spin as fast as possible. The fluid should form a vortex. If not, the distillation will exhibit poor recovery. Add all of the HCL from the dropping funnel to the flask.

7.2.10 Heat the water bath to the boiling point (100°C). The sample may or may not be boiling. Allow the purged distillation to proceed for 90 minutes at 100°C. Shut off nitrogen supply. Turn off heat.

7.2.11 Proceed to Step 7.3 for the analysis of the zinc sulfide by titration.

7.3 Titration of Distillate

7.3.1 Pipet a known amount of standardized 0.025N iodine solution (See Step 5.10.5) in a 500-mL flask, adding an amount in excess of that needed to oxidize the sulfide. Add enough reagent water to bring the volume to 100 mL. The volume of standardized iodine solution should be about 65 mL for samples with 50 mg of sulfide.

7.3.2 If the distillation for acid-soluble sulfide is being used, add 2 mL of 6N HCl. If the distillation for acid-insoluble sulfides is performed, 10 mL of 6N HCl should be added to the iodine.

7.3.3 Pipet both of the gas scrubbing bottle solutions to the flask, keeping the end of the pipet below the surface of the iodine solution. If at any point in transferring the zinc acetate solution or rinsing the bottles, the amber color of the iodine disappears or fades to yellow, more 0.025N iodine must be added. This additional amount must be added to the amount from Step 7.3.1 for calculations. Record the total volume of standardized 0.025N iodine solution used.

7.3.4 Prepare a rinse solution of a known amount of standardized 0.025N iodine solution, 1 mL of 6N HCl, and reagent water to rinse the remaining white precipitate (zinc sulfide) from the gas scrubbing bottles into the flask. There should be no visible traces of precipitate after rinsing.

7.3.5 Rinse any remaining traces of iodine from the gas scrubbing bottles with reagent water, and transfer the rinsate to the flask.

7.3.6 Titrate the solution in the flask with standard 0.025N phenylarsine oxide or 0.025N sodium thiosulfate solution until the amber color fades to yellow. Add enough starch indicator for the solution to turn dark blue and titrate until the blue disappears. Record the volume of titrant used.

7.3.7 Calculate the concentration of sulfide using the following equation:

$$\frac{(mL\ I_2 \times N\ I_2) - (mL\ titrant \times N\ titrant) \times \left(\frac{32.06\ g}{2\ eq.}\right)}{sample\ weight\ (kg)\ or\ sample\ volume\ (L)} = sulfide\ (mg/kg)\ or\ (mg/L)$$

8.0 QUALITY CONTROL

8.1 All quality control data must be maintained and available for reference or inspection for a period of three years. This method is restricted to use by or under supervision of experienced analysts. Refer to the appropriate section of Chapter One for additional quality control guidelines.

8.2 A reagent blank should be run once in twenty analyses or per analytical batch, whichever is more frequent.

8.3 Check standards are prepared from water and a known amount of sodium sulfide. A check standard should be run with each analytical batch of samples, or once in twenty samples. Acceptable recovery will depend on the level and matrix.

8.4 A matrix spiked sample should be run for each analytical batch or twenty samples, whichever is more frequent, to determine matrix effects. If recovery is low, acid-insoluble sulfides are indicated. A matrix spiked sample is a sample brought through the whole sample preparation and analytical process.

9.0 METHOD PERFORMANCE

9.1 Accuracy - Accuracy for this method was determined by three independent laboratories by measuring percent recoveries of spikes for both clean matrices (water) and actual waste samples. The results are summarized below.

<u>For Acid-Soluble Sulfide</u>

Accuracy of titration step only
  Lab A 84-100% recovery
  Lab B 110-122% recovery
Accuracy for entire method for clean matrices (H_2O)
  Lab C 94-106% recovery
Accuracy of entire method for actual waste samples
  Lab C 77-92% recovery

Spiking levels ranged from 0.4 to 8 mg/L

<u>For Acid-Insoluble Sulfide</u>

The percent recovery was not as thoroughly studied for acid-insoluble sulfide as it was for acid-soluble sulfide.

Accuracy of entire method for synthetic waste samples
Lab C 21-81% recovery

Spiking levels ranged from 2.2 to 22 mg/kg

9.2 Precision

For Acid-Soluble Sulfide

Precision of titration step only
Lab A CV% 2.0 to 37
Lab B CV% 1.1 to 3.8
Precision of entire method for clean matrices (H_2O)
Lab C CV% 3.0 to 12
Precision of entire method for actual waste samples
Lab C CV% 0.86 to 45

For Acid-Insoluble Sulfide

Precision of entire method with synthetic wastes
Lab C CV 1.2 to 42

9.3 Detection Limit - The detection limit was determined by analyzing seven replicates at 0.45 and 4.5 mg/L. The detection limit was calculated as the standard deviation times the student's t-value for a one-tailed test with n-1 degrees of freedom at 99% confidence level. The detection limit for a clean matrix (H_2O) was found to be between 0.2 and 0.4 mg/L.

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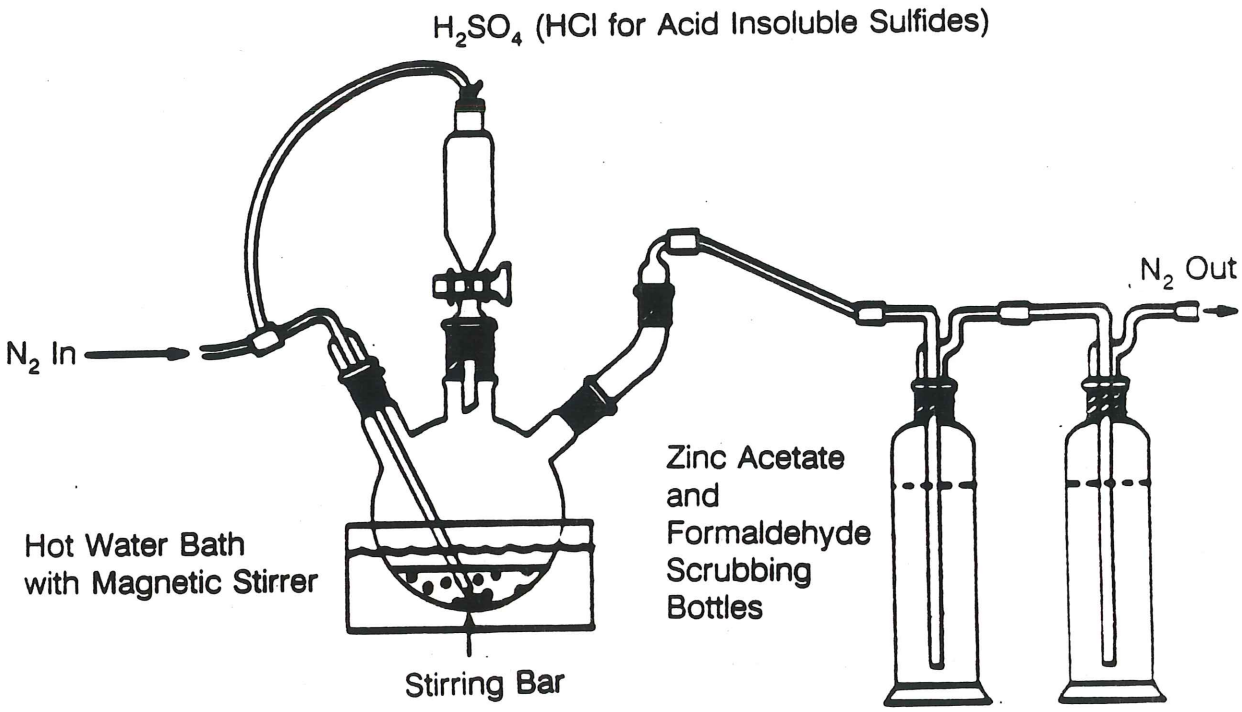
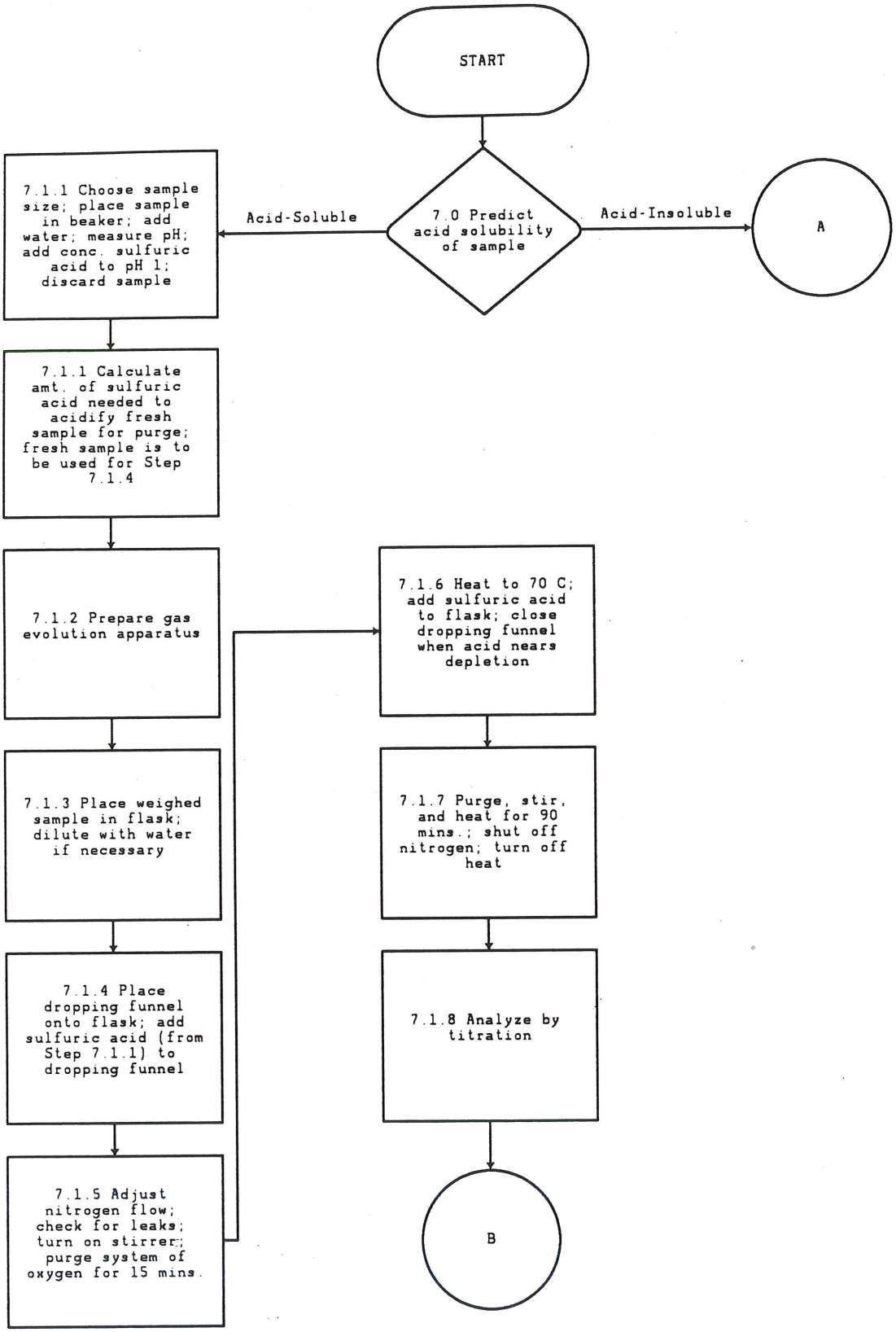
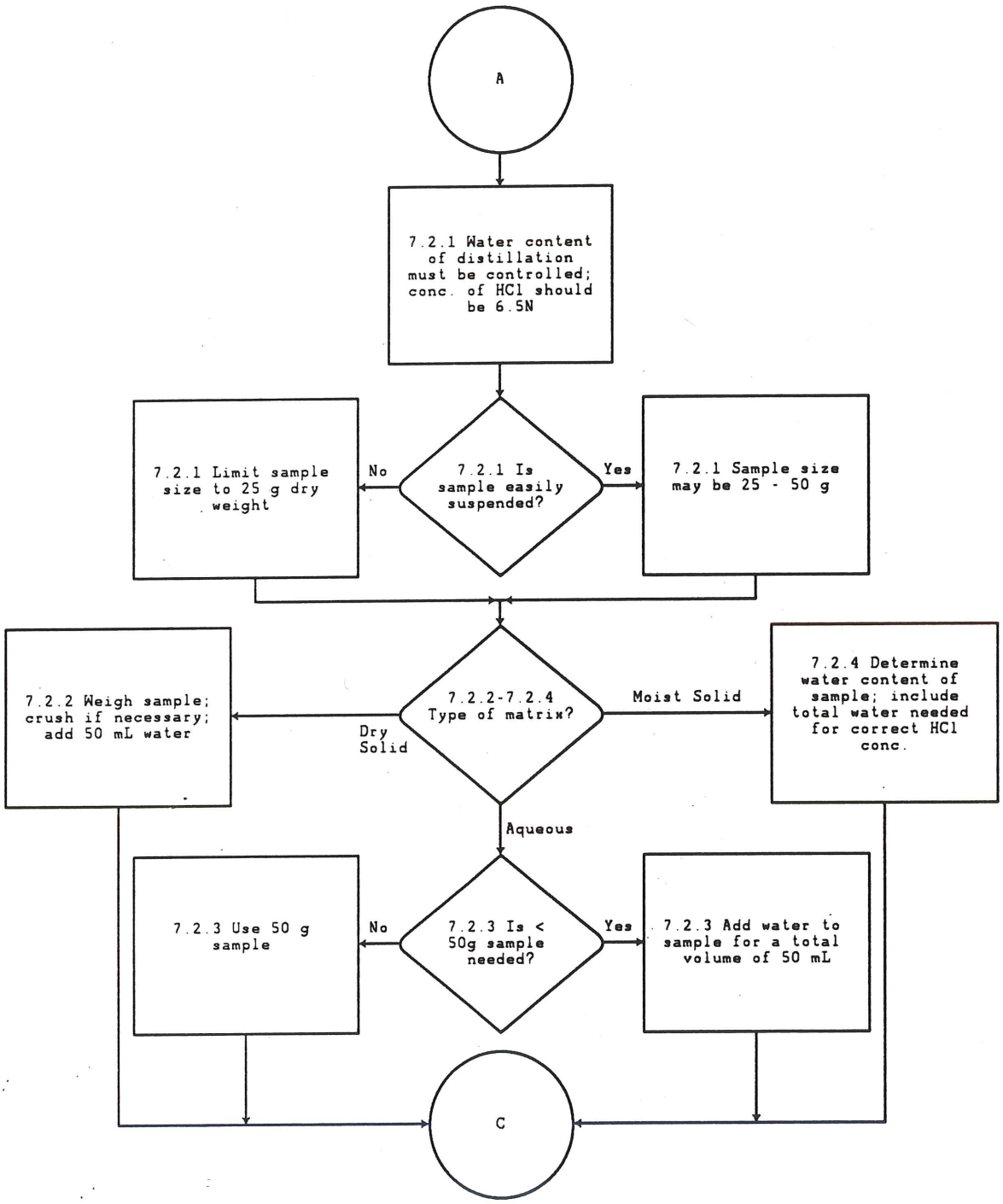
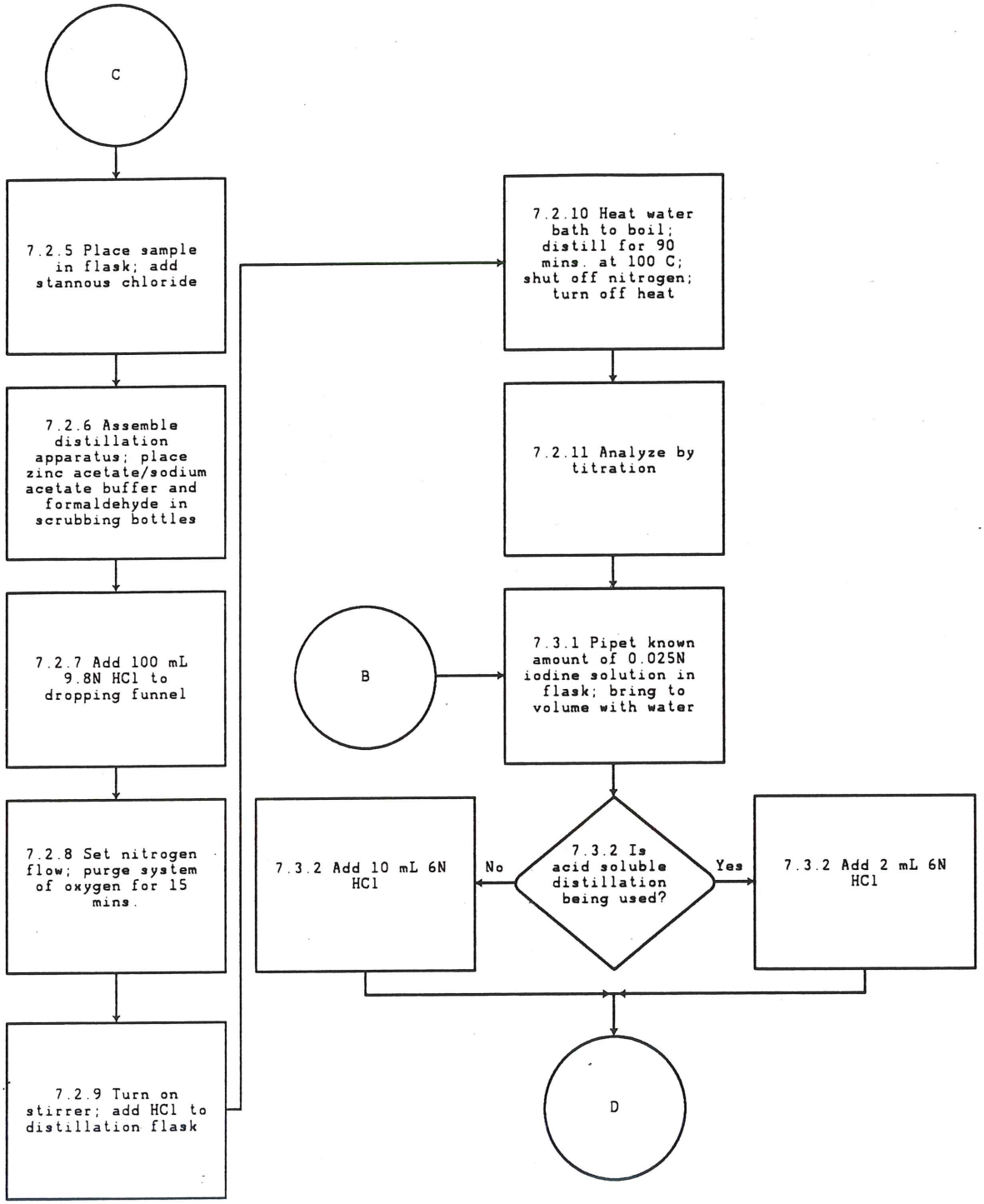
FIGURE 1.
GAS EVOLUTION APPARATUS

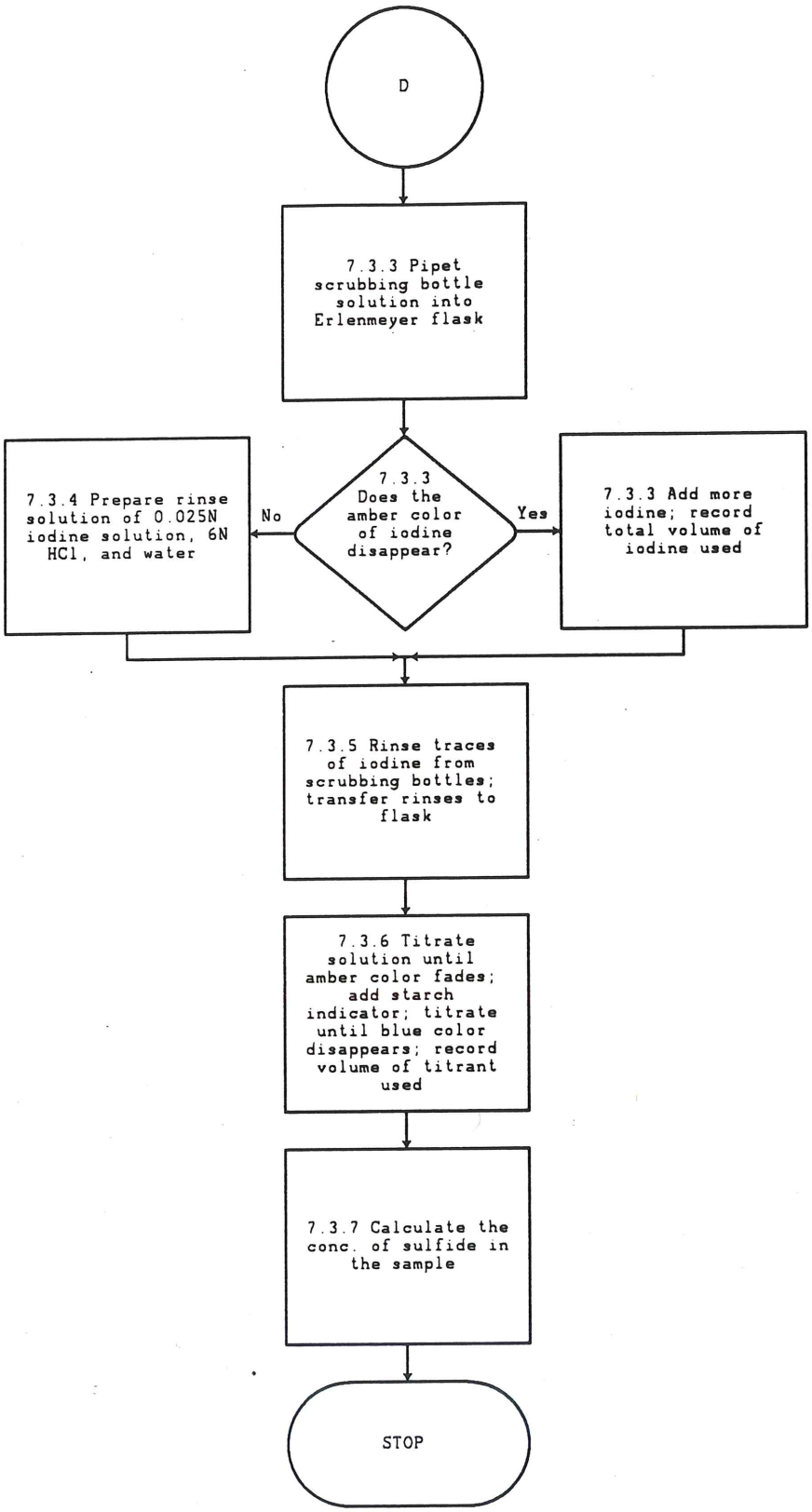


METHOD 9030A
ACID-SOLUBLE AND ACID-INSOLUBLE SULFIDES




A
7.2.1 Water content of distillation must be controlled; conc. of HCl should be 6.5N
7.2.1 Is sample easily suspended?
No
7.2.1 Limit sample size to 25 g dry weight
Yes
7.2.1 Sample size may be 25 - 50 g
7.2.2-7.2.4 Type of matrix?
Dry Solid
7.2.2 Weigh sample; crush if necessary; add 50 mL water
Moist Solid
7.2.4 Determine water content of sample; include total water needed for correct HCl conc.
Aqueous
7.2.3 Is < 50g sample needed?
No
7.2.3 Use 50 g sample
Yes
7.2.3 Add water to sample for a total volume of 50 mL
C


C
7.2.5 Place sample in flask; add stannous chloride
7.2.6 Assemble distillation apparatus; place zinc acetate/sodium acetate buffer and formaldehyde in scrubbing bottles
7.2.7 Add 100 mL 9.8N HCl to dropping funnel
7.2.8 Set nitrogen flow; purge system of oxygen for 15 mins.
7.2.9 Turn on stirrer; add HCl to distillation flask
7.2.10 Heat water bath to boil; distill for 90 mins. at 100 C; shut off nitrogen; turn off heat
7.2.11 Analyze by titration
B
7.3.1 Pipet known amount of 0.025N iodine solution in flask; bring to volume with water
7.3.2 Is acid soluble distillation being used?
No
Yes
7.3.2 Add 10 mL 6N HCl
7.3.2 Add 2 mL 6N HCl
D


D
7.3.3 Pipet scrubbing bottle solution into Erlenmeyer flask
7.3.3 Does the amber color of iodine disappear?
No
Yes
7.3.4 Prepare rinse solution of 0.025N iodine solution, 6N HCl, and water
7.3.3 Add more iodine; record total volume of iodine used
7.3.5 Rinse traces of iodine from scrubbing bottles; transfer rinses to flask
7.3.6 Titrate solution until amber color fades; add starch indicator; titrate until blue color disappears; record volume of titrant used
7.3.7 Calculate the conc. of sulfide in the sample
STOP