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Water Analysis Kit

Part No. 144-95

Instruction Manual

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Ver. 1.1

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Table of Contents

Components

Labware:

#144-95-001	Auto Self-Zeroing Buret
#145-601	Hydrogen Sulfide Test Papers, Package of 100
#145-602	Hydrogen Sulfide Test Bottle with Cap
#145-603	Hydrogen Sulfide Color Chart with Instructions
#145-604	Alka Seltzer Tablets, Packet of 2
#147-50	pH Paper; Hydrion Dispenser; pH 2 - 10, 1 - 11
#153-12	Glass Graduated Cylinder; 100 mL × 1 mL
#153-15	Test Tube; 15 mm × 125 mm
#153-26	Polyethylene Titration Dish
#153-28	Polyethylene Stirring Rod; 4"
#153-34	Glass Pipette; 1 mL × 1/100 mL
#153-40	Glass Pipette; 10 mL × 1/10 mL
#153-75	3/16" Tygon Tubing; 4 Feet
#153-76	5/16" Tygon Tubing; 4 Feet
#153-83	#3 Rubber Stopper; One Hole

Reagents:

#145-551	Starch Indicator Solution; 2 oz.
#145-552	*Sulfide Buffer Solution; 2 oz.; UN #1789
#145-553	Iodine Titrating Solution, 8 oz.
#147-30	Buffer Solution; pH 7; 16 oz.
#200-10-1	3% Hydrogen Peroxide; 2 oz.
#205-02	Versenate Hardness Indicator Solution; 2 oz.
#205-04	*Versenate Hardness Buffer Solution; 2 oz.; UN #2672
#205-12	Versenate Hardness Titration Solution; 1 mL = 20 EPM
#205-14	*Versenate Calcium Buffer Solution; 2 oz.; UN #1842
#206-04	Deionized Water; 32 oz.
#210-00	Cal Ver II Indicator; 10 g
#215-00	Potassium Chromate Solution; 2 oz.
#220-00	Phenolphthalein Solution; 2 oz.
#230-04	*Sulfuric Acid; N/50; 16 oz.; UN #2796
#230-15	*Sulfuric Acid; 5 N; 2 oz.; UN #2796
#240-00	Methyl Orange Indicator Solution; 2 oz.
#250-00	Calcium Indicator Solution; 2 oz.
#255-00	*Sulfate Indicator Solution; 2 oz.; UN #1789
#265-08	Silver Nitrate Solution; .01 g; 0.282 N; 16 oz.
#275-00	*Hydrochloric Acid; 37%; Concentrated; 2 oz.; UN #1789
#285-37	Iron Indicator Solution; 2 oz.
#285-40	Iron Buffer Solution; 2 oz.

Case:

#144-96	Stainless Steel Case

Chloride Ion Content

Procedure:

1. Add at least 1 mL filtrate to a clean titration dish.

2. Add two or three drops of phenolphthalein solution. If the sample turns pink, add sulfuric acid (N/50), drop by drop, until it turns clear again.

3. If the filtrate is colored, add an additional 2 mL of sulfuric acid (N/50) and stir. Add 1 gram of calcium carbonate and stir.

4. Add 25 to 50 mL deionized water and 5 to 10 drops of potassium chromate solution.

5. While stirring continuously, add silver nitrate solution (0.282N) from a pipette until the color changes from yellow to deep red. The color must stay red for at least 30 seconds.

6. Record the volume (in mL) of silver nitrate required to reach the end point.

   ☑ **Tip**

   If over 10 mL of silver nitrate are used, repeat the test using a smaller initial volume of filtrate.

Calculation:

$$\text{Chloride (mg / L)} = \frac{\text{mL Silver Nitrate} \times 10{,}000}{\text{mL Filtrate}}$$

☑ **Tip**

If the chloride ion concentration is less than 10,000 ppm, use the silver nitrate concentration equivalent to 0.019 chloride ion/mL (0.0282N). The multiplication factor in the equation then becomes 1,000.

$$\text{Chloride (mg / L)} = \frac{\text{mL Silver Nitrate} \times 1{,}000}{\text{mL Filtrate}}$$

Alkalinity

Filtrate

1. Add at least 1 mL of filtrate to a titration dish.

2. Add two or three drops of phenolphthalein solution. If the sample turns pink, add sulfuric acid (N/50) from a pipette while stirring until it turns clear again.

   Tip

   If the sample is too dark to see the end point, use a pH meter instead. You will know the reaction has reached the end point when the pH drops to 8.3.

3. Record the volume of sulfuric acid (N/50) per milliliter of filtrate required to turn the sample clear again. This value is P_f.

4. Add two or three drops of methyl orange indicator solution to the same sample.

5. While stirring the sample, add sulfuric acid (N/50) from a pipette until the color changes from yellow to pink.

   Tip

   If the sample is too dark to see the end point, use a pH meter instead. You will know the reaction has reached the end point when the pH drops to 4.3.

6. Record the total volume of sulfuric acid (N/50) per milliliter of filtrate required to reach the final end point (including the amount required to reach P_f). This value is M_f.

Alkalinity

Fluid

1. Add 1 mL of fluid to a titration dish.

2. Dilute the sample to 25 - 50 mL with deionized water.

3. Add 4 or 5 drops of phenolphthalein indicator.

4. While stirring, titrate rapidly with sulfuric acid (N/50 or N/10) until the pink color disappears.

   Tip

   If the sample is too dark to see the end point, use a pH meter instead. You will know the reaction has reached the end point when the pH drops to 8.3.

5. Record the total volume of sulfuric acid required to reach the end point:

   P_m = mL of N/50 sulfuric acid

   or

   $P_m = 5 \times$ mL of N/10 sulfuric acid

Lime

Procedure:

1. Determine the phenolphthalein alkalinity of the filtrate and the fluid P_f and M_f.

2. Determine the volume fraction of water (F_w) in the fluid using a retort.

$$F_w = \frac{\%\ \text{water by volume}}{100}$$

Calculations:

$$\text{Lime } (lb_m / bbl) = 0.26\ (P_m - F_w \times P_f)$$

$$\text{Lime } (kg / m^3) = 0.742\ (P_m - F_w \times P_f)$$

Total Hardness as Calcium

Procedure:

1. Add at least 1 mL of filtrate to the titration dish.
2. If the filtrate is too dark to see the end point:
   a. Add 10 mL of bleach and stir.
   b. Add 1 mL of acetic acid and stir.
   c. Boil the sample for five minutes. Maintain the same volume by adding deionized water.
   d. Cool the sample and wash the sides of the beaker with deionized water.
3. Dilute the sample to 50 mL with deionized water.
4. Add approximately 3 mL hardness buffer solution. Mix thoroughly.
5. Add two to six drops of hardness indicator solution. Mix thoroughly.

   A deep red color will appear if calcium and/or magnesium are present.
6. While stirring, add titration solution from a pipette until the color changes from red to blue.
7. Record the volume of titration solution required to reach the end point.

Calculation:

$$\text{Total Hardness as Calcium (mg / L)} = 400 \times \frac{\text{Volume of Titrating Solution}}{\text{Volume of Filtrate}}$$

Calcium Content

Procedure:

1. With a pipette, add at least 1 mL filtrate to a titration dish. Record the filtrate volume.
2. Dilute the filtrate to 50 mL with deionized water.
3. Add 10 to 15 mL of calcium buffer solution.
4. Add 3 mL of sodium hydroxide (1N).
5. Add on scoop of calcium indicator. If calcium is present, the solution will turn pink.

   Important: Do not use total hardness indicator or buffer solution.
6. Add 1 mL of masking agent and stir.
7. While stirring, add hardness titrating solution from a pipette until the color changes from red to blue.

Calculation:

$$\text{Calcium Concentration (mg / L)} = 400 \times \frac{\text{Volume of Titrating Solution (mL)}}{\text{Volume of Filtrate (mL)}}$$

Magnesium Content

Procedure:

1. Determine the total hardness as calcium as described on page 6. Record the volume of titrating solution used as V_1.
2. Determine the calcium content as described above. Record the volume of titrating solution used as V_2.

Calculation:

Magnesium Concentration (mg / L) = $(V_1 - V_2) \times 243$

Calcium Sulfate

Procedure:

1. Add 5 mL of drilling fluid to 245 mL deionized water.

2. Stir for 15 minutes.

3. Filter the diluted drilling fluid through a standard filter press. Keep only the clear portion of the filtrate. Discard the cloudy part.

4. Titrate 10 mL of clear filtrate to the EDTA end point. Record the volume of EDTA as V_t.

5. Titrate 1 mL of the original drilling fluid filtrate to the EDTA end point. Record the volume of EDTA as V_f.

6. Determine the volume fraction of water (F_w) in the fluid using a retort.

   $$F_w = \frac{\text{\% water by volume}}{100}$$

Calculation:

Total Calcium Sulfate (kg / m³) = $6.79 \times V_t$

Total Calcium Sulfate (lb / bbl) = $2.38 \times V_t$

Undissolved Calcium Sulfate (kg / m³) = $(6.79 \times V_t) - 1.37 \times (V_f \times F_w)$

Undissolved Calcium Sulfate (lb / bbl) = $(2.38 \times V_t) - .48 \times (V_f \times F_w)$

Iron Count

Procedure:

1. Add 3 drops of concentrated hydrochloric acid (37%) solution to 100 mL of clear water (not filtrate). Check the pH with a pH meter or pH paper. The pH of the solution should be 1 to 2.

2. Add 0.5 mL of hydrogen peroxide solution. The color that develops (usually a pale yellow) will be the end point color.

3. Add 1.0 mL of iron indicator solution. A purple color will develop.

4. Add 0.5 mL of iron buffer solution. The pH should be between 2 and 3. Check the pH with a pH meter or pH paper and add additional buffer solution, if necessary.

5. Titrate with total hardness titrating (EDTA) solution (1 mL = 20 EPM Ca & Mg) back to the same color developed in step 2. If less than 1 mL of the 20 EPM titrating solution is used to reach the end point, repeat steps 1 through 4 and use the weak total hardness yitrating solution (1 mL = 2 EPM Ca & Mg) instead.

Calculations:

For 100 mL sample of water:

ppm Fe^{3+} = Total Hardness Titrating Solution, 20 EPM, mL × 5.6

ppm Fe^{3+} = Total Hardness Titrating Solution, 2 EPM, mL × 0.56

If it is necessary to convert to EPM Iron:

$$\text{EPM Iron } Fe^{3+} = \frac{\text{ppm Iron } Fe^{+++}}{18.6}$$

Sulfide Ion (S^{-2})

Procedure:

1. Add 1 mL of Iodine titrating solution to 50 mL of deionized water in a titrating dish.

2. Add 10 drops of Sulfide buffer solution and 20 drops of starch indicator solution.

3. With a pipette, add deionized water until the blue or red color completely disappears. Record the amount of water (in mL) required to reach the end point.

   $$\text{Sulfide Ion Content (mg / L)} = \frac{100}{\text{Water (mL)}}$$

4. If more than 100 mL of water are required to reach the end point, the concentration of sulfide ions is less than 1 mg / L.

   a. Add 100 mL deionized water, 10 drops of buffer solution, and 20 drops of starch indicator solution to a titration dish.

   b. With a pipette, add Iodine titrating solution until a permanent blue color develops.

   $$\text{Sulfide Ion Content (mg / L)} = \frac{100}{\text{Titrating Solution (mL)}}$$

5. If less than 1 mL of water was required to reach the end point in step 3, the concentration is more than 100 mg / L.

   a. Add 10 mL of iodine titration solution and 20 drops of starch indicator solution to 100 mL of deionized water in the titration dish.

   b. With a pipette, add deionized water until the blue or red color disappears completely.

   $$\text{Sulfide Ion Content (mg / L)} = \frac{1000}{\text{Water (mL)}}$$

Note

If one drop of iodine titrating solution produces a permanent blue color, the sulfide ion concentration is zero.