

7.0 Procedure

- 7.1 Pipet 50 mL of the sample into a 250 mL beaker.
- 7.2 Measure the pH of the sample. If the pH is above 4.0, add standard sulfuric acid (6.3) in 5.0 mL increments to lower the pH to 4.0 or less. If the initial pH of the sample is less than 4.0, the incremental addition of sulfuric acid is not required.
- 7.3 Add 5 drops of hydrogen peroxide (6.1).
- 7.4 Heat the sample to boiling and continue boiling for 2 to 4 minutes. In some instances, the concentration of ferrous iron in a sample is such that an additional amount of hydrogen peroxide and a slightly longer boiling time may be required.
- 7.5 Cool the sample to room temperature and titrate electrometrically with standard sodium hydroxide (6.2) to pH 8.2.

8.0 Calculations

$$8.1 \quad \text{Acidity, as mg/ L CaCO}_3 = \frac{[(A \times B) - (C \times D)] \times 50,000}{\text{mL of sample}}$$

where:

- A = vol. of standard sodium hydroxide used in titration
- B = normality of standard sodium hydroxide
- C = volume of standard sulfuric acid used to reduce pH to 4 or less
- D = normality of standard sulfuric acid

- 8.2 If it is desired to report acidity in milliequivalents per liter, the reported values as CaCO₃ are divided by 50, as follows:

$$\text{Acidity, as meq/L} = \frac{\text{mg/L CaCO}_3}{50}$$

9.0 Precision

- 9.1 On a round robin conducted by ASTM on 4 acid mine waters, including concentrations up to 2000 mg/ L , the precision was found to be ± 10 mg/L.

Bibliography

1. Annual Book of ASTM Standards, Part 31, "Water", p 116, D 1067, Method E (1976).
2. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 277, Method 402(4d) (1975).