

Nonferrous
Metal

Free Sulfuric Acid in Electrolyte

Acid-base titration by
Automatic Potentiometric Titrator

Standard

1. Abstract

The concentration of free sulfuric acid in electrolytic solution is determined by potentiometry of the test sample. First add pure water and 10w/v% Potassium fluoride, and titrate with 1mol/L Sodium hydroxide up to the endpoint.

The endpoint is the maximum inflexion point on titration curve. The concentration of free sulfuric acid is obtained from titration volume of Sodium hydroxide.

When aluminum ion and iron (III) ions exist in the electrolyte solution, the hydroxide is generated and it leads to an excessive titration. These ions are masked by adding the fluoride to prevent an excessive titration.

2. Reference

- 1) Experiment and Calculation in Quantitative Analysis – Vol.2 – Seiji Takagi, Kyoritsu Publication

3. Cautions in measurement

- 1) Handle with care when you work on chemicals.

4. Post-measurement care

After a series of measurements is finished, wash away sample residues over the surface of each electrode under running water, and keep them in a beaker filled with water.

5. Test equipment

Main unit : Automatic potentiometric titrator (Standard preamplifier: STD-)

Electrode : Standard pH glass electrode

Standard Ceramic type reference electrode

6. Reagent

Titrant : 1mol/L—Potassium hydroxide (f=1.002)

Solvent : Pure water, 10w/v% Potassium fluoride

7. Measurement procedure

—Measurement—

- 1) Transfer 5mL sample to a 200mL beaker, and add 100mL of pure water.
- 2) Add 30mL of 10w/v% Potassium fluoride.
- 3) Titrate with 1mol/L—Potassium hydroxide to obtain concentration.

8. Formula

Free sulfuric acid (g/L) = (EP1 - BL1) × FA1 × C1 × K1 / SIZE

EP1 : Titration volume (mL)

BL1 : Blank level (0.00mL)

FA1 : Factor of titrant (1.002)

C1 : Concentration conversion coefficient (49mg/mL)

(1mol/L NaOH 1mL ≡ 49mg H₂SO₄)

K1 : Unit conversion coefficient (1)

SIZE : Sample size (mL)

9. Example of measurement

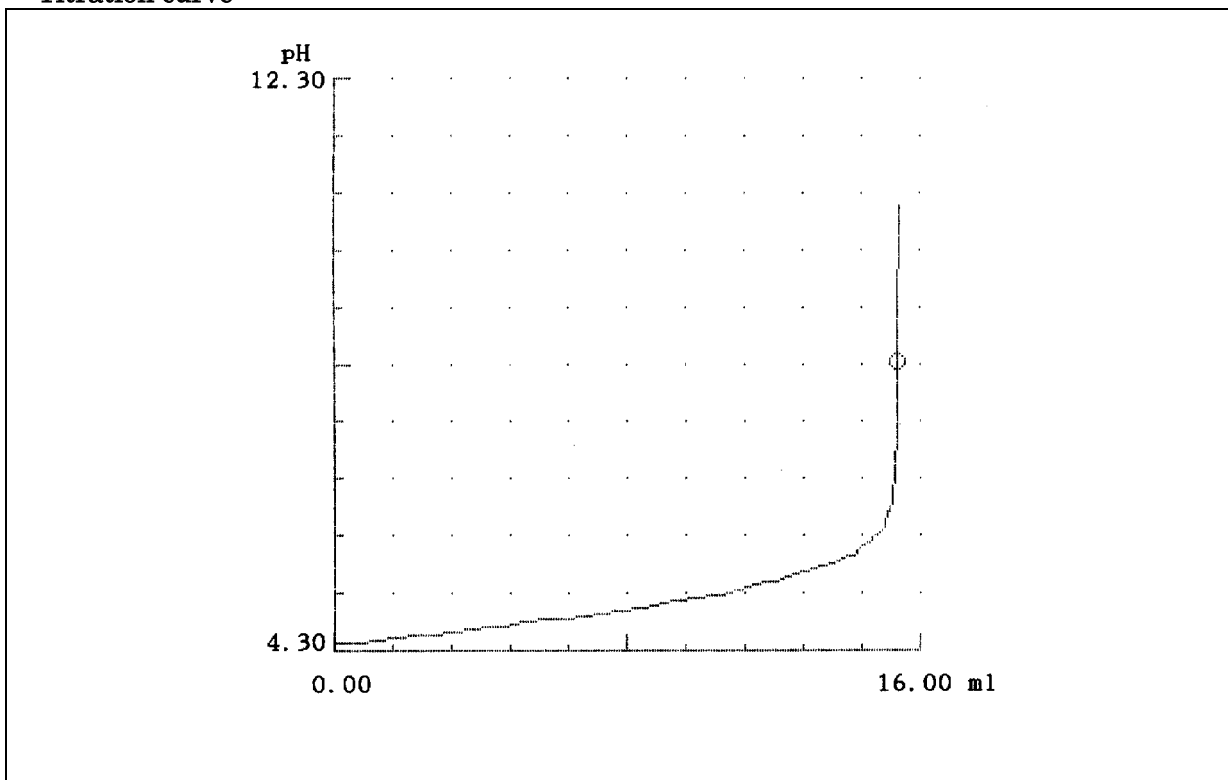
— Ambient condition —

Room temperature : 25 °C	Humidity : 50 %	Weather : Fair
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- Titration parameters -

[Titration parameter]			
Titration mode	: Auto	Preamplifier unit	: pH
Titration form	: EP Stop	Max. volume of titration	: 20.0mL
Titration burette	: 01	Auto stir control	: Off
Reagent	: NaOH	Wait time before titration	: 0s
Detector No.	: 1	Titration direction	: Auto
[Control parameter]			
Number of endpoints	: 1	Control speed	: 2.0
Auto simulation	: Off	Data sampling potential	: 4.0mV
EP sense (Potential)	: 50.0	Data sampling volume	: 0.5mL
EP sense (Differential)	: 100.0	Separate potential	: Off
Over-titration	: 0.0mL	Potential between inflexions	: 0.0pH
Gain	: 1	EP potential setup	: Off
		EP potential	: 0.0pH

-Titration curve-



(The above printout data were obtained from titration by AT-410 unit)

—Measurement results—

n	Sample (mL)	Free sulfuric acid (g/L)
1	5.0	151.48
2	5.0	151.24
3	5.0	151.21

Concentration of free sulfuric acid	
Mean	151.31 g/L
SD	0.15 g/L
RSD	0.10 %

* The above results were obtained by 3 tests of the same sample.

10. Summary

The Electrolyte Solution is the solution made electrically conductive by dissolving ionic substance in polar solvent like water. It is also called Ionic solution. In the narrow sense, it means the electrolyte solution for battery or electroplating bath.

The sample test shows a good repeatability of 0.1% relative standard deviation.

The automated potentiometry assures precise and reliable measurement.