## 8-14 Test Method - Residual Chloride Content for Etched Foil & Cathode Formed Foil 1 of 2

## 1.Scope of Test

- 1) All Etched Foils
- 2) All Formed Foils for Cathode

#### 2.Principle

Combust test-specimen in Argon-Oxygen stream, and the produced Hydrogen Chloride is lead to titration cell, then titrate with electrolytically-generated AG-ION. Using quantity of electricity required for titration, level of chlorine is calculated based on 'Faraday's Law'.

### 3.Test Apparatus

Automated titrator which is composed of AUTO-BOARD-CONTROLLER; test specimen on specimen-board is lead to electric furnace and combusted, and TITORATOR; produced Hydrogen Chloride is titrated with electrolytically-generated AG-ION and the endpoint detected with set-up final electric potential.

Applicable Unit: Chlorine Analysis Devices such as TOX-2100H

#### 4.Reagents

1) 85% Acetic Acid Solution (Electrolyte Solution)

This solution is made by dissolving 0.8g of Gelatine (as specified in JIS K 6503 – first grade) in 150ml of deionized water, then, is warmed, stirred and stand cool.Secondly, put 1.35g (precise weighing) of Anhydrous Sodium Acetate (as specified in JIS K 8372-high grade reagent) and Acetic acid (as specified in JIS K 8355 – high grade reagent) into this solution and the total amount is adjusted to 1,000ml. After that, add 1ml of 0.01mol/L Hydrochloric Acid Solution.

(expire: 6months, keep in airtight container at cool and dark place)

2) 1mol/L Kalium Chloride Solution (inside-reference-extrode solution)

This solution is made by dissolving 7.46g (precise weighing) of Kalium Chloride (as specified in JIS K 8121 - high grade reagent) in 100ml of deionized water.

3) 1mol/L Potassium Nitrate Solution (outside-reference-electrode solution)

This solution is made by dissolving 10.1g (precise weighing) of Potassium Nitrate (as specified in JIS K8548 - high grade reagent) in 100ml of deionized water.

4) 10% Potassium Nitrate Solution (AG-generate electrode antipole solution)

This solution is made by dissolving 50g of Potassium Nitrate (as specified in JIS K 8548 - high grade reagent) in 500ml of deionized water.

5) Concentrated Sulfuric Acid (dewatering bath)

98% Sulfuric Acid (as specified in JIS K 8951 - high grade reagent) is used.

6) 0.01mol/L Hydrochloric Acid Solution (hydrochloric acid reference solution)

0.01 mol/L Hydrochloric Acid for volumetric analyses (as conform to JIS K 8001-5) is used.

# 5.Test Specimen

10cm (Projected Area) of Foil

# 6.Apparatus

- 1) Medicine Spoon
- 2) Analytical Balance
- 3) 25  $\mu$  l Microsyringe
- 4) Measuring Flask
- 5) Measuring Cylinder
- 6) AG-working electrode for chlorine, AG counter electrode for chlorine
- 7) AG-detection electrode for chlorine
- 8) Reference electrode

## 8-14 Test Method - Residual Chloride Content for Etched Foil & Cathode Formed Foil 2 of 2

# 7. Measurement

1) Combustion Temperature

in Argon stream : 850°C in Oxygen stream : 900°C

2) Test Titration

setup the end-electric potential of titration and titer coefficient.

end potential : 290-315mV

titer coefficient : within the range of 0.5-5.0 3) Coulometric Titration Yield (precision), exam 1

Put 10  $\mu$ I of Hydrochloric Acid reference solution in electrolysis cell directly using microsyringe, then titrate.

yield : examine within 95-105% 4) Coulometric Titration Yield (precision), exam 2

Put 10  $\mu$  I of Hydrochloric Acid reference solution on sample board using microsyringe, combust as same as real-

sample, generate chlorine gas, dehydrate and titrate.

yield : examine within 93-103% 5) Measurement

After exam whether the yield (precision) of 3)and 4) is within the allowance, start to measure.

#### 8.Calculation of Residential Chlorine Content

Chlorine analysis value of  $10cm^2$  reagent is analyses with  $\mu$  g index.

analytical value :  $\mu$  g/10cm2= mg/m2

effective digit : 2 digit

#### 9.Determination

Etched Foil, low voltage  $: \le 2.0 \text{mg/m}^2 : \text{acceptable}$ Etched Foil, middle to high voltage  $: \le 1.0 \text{mg/m}^2 : \text{acceptable}$ Etched Foil for cathode  $: \le 1.0 \text{mg/m}^2 : \text{acceptable}$ Formed Foil for cathode  $: \le 0.8 \text{mg/m}^2 : \text{acceptable}$