

8-12-① Test Method - Residual Chloride Content (Formed Foils) 1 of 1

1. Scope of Test

All Formed Foils

2. Principle

The chloride ion on foil is extracted in a sodium hydroxide solution. The extract is heated with nitric acid and clarified by centrifugation. A silver nitrate solution is added to the clarified extract to form a suspension of colloidal silver chloride. Residual chloride content of the sample solution is then compared with a standard solution by means of a turbidimetric test.

3. Reagents

(1) Sodium Hydroxide Solution

The solution is made by dissolving 2g of sodium hydroxide (as specified in JIS K 8576 – High Grade Reagent) in 1ℓ of deionized water and then stored in a polyethylene bottle.

(2) Silver Nitrate Solution

The solution is made by dissolving 2.5g of silver nitrate (as specified in JIS K 8550 – High Grade Reagent) in 500 ml of deionized water and then stored in a brown glass bottle.

(3) 60% Nitric Acid Solution

The solution is a high grade reagent with a chloride content of 0.5ppm or less as specified in JIS K 8541.

(4) Chloride 1ppm Reference Solution

The reference solution is made by dissolving 1.65g of sodium chloride (as specified in JIS K 8150 – High Grade Reagent) in 1ℓ of deionized water; then 1ml of this solution is diluted in deionized water, and the total amount is adjusted to 1ℓ.

4. Test Specimen

Using scissors cut foil from the Projected Area ($125 \pm 5 \text{ cm}^2$) into pieces that are approximately 1 cm^2 .

5. Apparatus

- (1) Centrifuge: 50 s^{-1} or higher.
- (2) Centrifuge Tube: 50ml
- (3) Turbidity Meter with Fluorescent Light: A dark box.
- (4) Turbidity Test Tubes: 6 tubes of the same material and shape with measuring increments and a volume of 50ml.
- (5) Warm Water Bath: Capable of maintaining approximately 2ℓ of water at $80 \pm 10^\circ\text{C}$ and $30 \pm 2^\circ\text{C}$.
- (6) Temperature Measurement Device: A thermometer as specified in JIS B 7411 with an accuracy of $\pm 1^\circ\text{C}$ at each gradation below 100°C .
- (7) Cooling Vessel: 3ℓ stainless steel beaker or similar device suitable for cooling.
- (8) Conical Flask: 100ml
- (9) Measuring Flask: 100ml
- (10) Measuring Pipette: 5ml
- (11) Tweezers
- (12) Scissors

6. Test Procedure

6.1 Extraction Using Sodium Hydroxide

- (1) Place test pieces in a 100ml conical flask with tweezers.
- (2) Pour 60ml of the Sodium Hydroxide Solution into the conical flask, stir once or twice and let it stand for approximately 7 minutes at room temperature.

6.2 Clarification of Extract

- (1) Rinse the test pieces two separate times in 10ml of deionized water. Pour this water and the extract obtained from step 6.1 (2) into the 100ml measuring flask and add 3ml of the 60% Nitric Acid Solution.
- (2) Heat the measuring flask with the solution obtained from step 6.2 (1) in a warm water bath ($80 \pm 10^\circ\text{C}$) for about 20 minutes.
- (3) Cool solution to room temperature, fill the measuring flask with deionized water to the 100ml mark and shake extract well before distribution.

6.3 Distribution of Extract

Transfer 30~40ml of extract into a centrifuge tube. After 15 minutes of centrifugation at 50 s^{-1} , transfer 20ml of the supernatant liquid to a turbidity test tube. This shall be the Test Solution.

6.4 Procedure for Turbidity Determination

- (1) Use the measuring pipette to transfer the Chloride 1ppm Reference Solution into two turbidity test tubes. Place 2.5ml of the solution into one tube for Reference Solution A and 4.0ml into the other tube for Reference Solution B. Add deionized water to each tube to adjust total amount to 20ml.
- (2) Add approximately 1ml of the 60% Nitric Acid Solution to the Reference Solutions A and B as well as the Test Solutions developed in 6.3 (1) and (2). Shake well and place the test tubes in a warm water bath ($30 \pm 2^\circ\text{C}$) for 5 minutes.
- (3) Keep the test tubes in the warm water bath and add drops of the Silver Nitrate Solution (approximately 2ml) into each test tube. In a dark room, shake well and let stand for 15 minutes.
- (4) Transfer the test tubes from the warm water bath to the dark box to compare the turbidity of the Reference Solutions with that of the Test Solutions.

7. Residual Chloride Content Measurement

- (1) If the turbidity of Test Solutions is less than or equal to the turbidity of Reference Solution A, the residual chloride ion content of foil is 1.0 mg/m^2 or less.
- (2) If the turbidity of Test Solutions is less than or equal to the turbidity of Reference Solution B, the residual chloride ion content of foil is 1.6 mg/m^2 or less.

8-12-② Test Method - Residual Chloride Content (Etched Foils)

1. Scope of Test

All Etched Foils

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-100, TOX-2100H

Manufacturers : Dia Instruments Co., Ltd. , etc

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

5. Test Specimen

10cm² (Projected Area) of Foil

6. Apparatus

- 1) Medicine Spoon
- 2) Analytical Balance
- 3) 25μl Microsyringe
- 4) Measuring Flask
- 5) Measuring Cylinder
- 6) AG-produce electrode for chlorine
- 7) AG-detection electrode for chlorine
- 8) Reference electrode

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 10μl 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 10μl 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 10cm² reagent is analyses with μg index.

analytical value : μg/10cm² = mg/m²

effective digit : 2 digit

9. Determination

Etched Foil, low voltage : ≤2.0mg/m²:acceptable
Etched Foil, middle to high voltage : ≤1.0mg/m²:acceptable
Etched Foil for cathode : ≤1.0mg/m²:acceptable