◈ Residual Chlorine Test

1. Test equipments

1) Centrifugal separator : Max. speed ≧ $50s^{-1}$ (3,000rpm).
2) Tube(s) for above※ : Capacity 50 ㎖.
3) Hot water bath : Capacity 2ℓ approx. Temp. 80±10℃ and 30±2℃.
4) Nephelometer : Dark box with fluorescent lamp.
5) Test tubes for above : Six. Same material and size.
   Capacity 50 ㎖, mark at 20 ㎖
6) Thermometer : JIS B 7411.
   Accuracy of every graduation ±1℃ below100℃.
7) Erlenmeyer flask※ : Capacity 100 ㎖.
8) Measuring flask※ : Capacity 100 ㎖.
9) Measuring pipette※ : Capacity 5 ㎖.
10) Cooling bath : Stainless steel beaker(3,000 ㎖).
11) Tweezers※, Scissors※ (※Must be clean)

2. Reagents for measurement

1) Aqueous solution of NaOH
   Dissolve 2.0g of sodium hydroxide(JIS K 8576 analytically pure) into 1,000 ㎖ of pure water. Store the solution in a polyethylene bottle.
   Blank test to confirm that it is chlorine free.
2) Aqueous solution of $AgNO_3$
   Dissolve 2.5g of silver nitrate (JIS K 8550 analytically pure) into 500 ㎖ of pure water. Store the solution in a brown bottle.
3) 60% HNO_3 solution
   JIS K 8541 analytically pure. Chlorine density≦0.5ppm.
4) Standard chlorine ion (1ppm)
   Let dry sodium chloride(JIS K 8150 solution, 1ppm analytically pure) at 120℃for more than 2 hours. Dissolve 1.65g of dried NaCl above into pure water to make the total volume 1,000 ㎖. Dilute 1 ㎖ of above solution to 1,000 ㎖.

3. Test piece

Projected area 125±5 ㎠. Cut it into small pieces(about 1 ㎠ each) with scissors.

4. Test procedure

1) Extraction
   Put all the cut pieces into the 100 ㎖ Erlenmeyer flask using a pair of tweezers. Add 60 ㎖ of sodium hydroxide solution. Stir a few times.
   Leave it at room temperature for about 7min.
2) Cleaning of extracted solution
   Put all the extracted solution, 1), and a solution obtained by rinsing

the foil surface twice with 10 ㎖ of pure water into the 100 ㎖ measuring flask.

Add 3 ㎖ of 60% HNO_3 solution.

Leave the flask in the hot water bath(at a controled temperature of 80±10℃) for about 20min. Take the flask out, and let it cool down temperature.

Add pure water up to the 100 ㎖ mark. Shake well.

This is the final sample solution to be tested.

3) Division

In the case of unformed foil, take 20 ㎖ of above extracted solution and put it into one of the test tubes of the nephelometer, and use it as the test sample.

In the case of formed foil, put 30 ~ 40 ㎖ of the extracted solution into the test tube of the centrifugal separator, and turn the machine at $50s^{-1}$ (3,000rpm) for about 15min. Take 20 ㎖ of the supernatant liquid, and put it into the test tube for measurement.

4) Nephelometer

Using measuring pipette, put 0, 1.0, 1.5, 2.0, 2.5 and 3.0 ㎖ of the 1ppm (1 ㎍/㎖ approx.) chlorine ion standard solution into five test tubes of the nephelometer. Add pure water to each of the test tubes to make the total volume 20 ㎖ each. In this way, standard solutions with chlorine densities of 0, 1.0/20, 1.5/20, 2.0/20, 2.5/20 and 3.0/20(㎍/㎖) are prepared.

Put about 1 ㎖ of the 60% nitric acid solution to each of the six test tubes (one test sample and five standards). Shake the tubes, and leave them in the hot water bath (controlled at 30±2℃) for 5 min.

Drop about 2 ㎖ of silver nitrate solution into each of those six test tubes (still in the hot water bath).

Shake the test tubes well to make the turbidity uniform.

Leave them in a dark place for 15min. Move the test tubes from the hot water bath to the dark box of the nephelometer, and compare the turbidities of the sample solution and the standard solutions to determine the chlorine density of the test sample solution (Y).

Additional standard solution (s) of different density may be used if necessary.

5) Calculation of the residual chlorine density of the foil

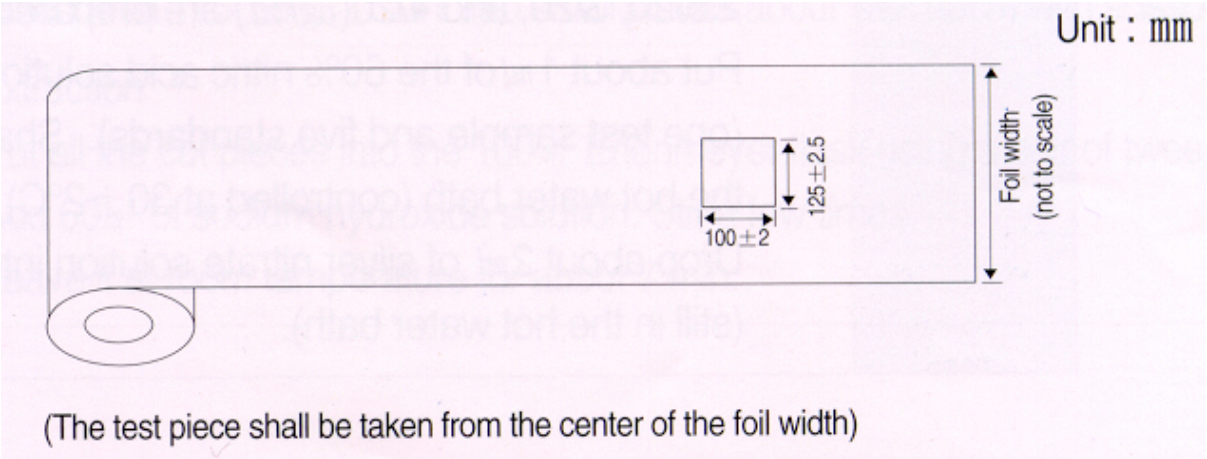
As the projected area of the test piece is 125 ㎠,and the volume of the extracted solution is 100 ㎖,

$$X = \frac{Y}{1{,}000} \times 100 \times \frac{10{,}000}{125} = Y \times 8$$

where X is the residual chlorine density per projected area (㎎/㎡) and Y is chlorine density (㎍/㎖) of the sample solution measured in (4).
Accordingly, the relation between Y and X is as shown below:

Y, chlorine density of the sample solution (㎍/㎖)	X, residual chlorine density of the foil (㎎/㎡)
0	0
1.0/20	0.4
1.5/20	0.6
2.0/20	0.8
2.5/20	1.0
3.0/20	1.2

5. Sampling



6. Criteria

Pass if the residual chlorine density is less than the specification value.

7. The others

basically, residual chlorine density of the foil is indicated by solution area 125 ㎠
but, the analysis of a low capacitance do not mind solution area 250 ㎠ however, chlorine concentration of a sample 5) set by 250 ㎠.