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Neutral, acetic acid and copper-accelerated
acetic acid salt spray
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Neutral, acetic acid and copper-accelerated acetic acid salt spray

Introduction

This Korea Industrial Standards was established first in 1964, amended in 1992, 2002, 2005, 2007 and amended for the fifth time in 2009. In the amendment in 2002, the technical items of ISO 9227, Corrosion tests in artificial atmospheres — Salt spray tests which was the corresponding international standard were added to the previous standard in accordance with the international standard. In this amendment, the application range of three salt spray tests was clarified and the normative references were updated to the latest version.

1 Scope

This standard provides necessary equipment, reagents, technique and judgment method when the corrosion resistance test of metal material or plated metal material or coated with inorganic or organic film is carried out according to the neutral salt spray test (NSS), acetic acid salt spray test (AASS) or copper-accelerated acetic acid salt spray test (CASS).

The NSS test applies to metal and alloy, specific metal coating (anode and cathode), specific conversion treatment coating, specific anodic oxidation coating and organic coating applied on the metallic material.

The AASS test is especially useful for testing copper + nickel + chrome or nickel + chrome which are decoration coatings. It is also known to be appropriate for testing anodic oxidation coating on aluminum.

The CASS test is useful for decoration costing of testing copper + nickel + chrome or nickel + chrome. It is also known to be appropriate for testing anodic oxidation coating on aluminum.

2 Normative References

The following list of the normative references is indispensable for the application of this standard. For the standards with the year of publication, only the referenced edition must be applied. For those without the publication year, the latest edition (including all the addenda) shall be applied.

- KS D 3512, Cold-reduced carbon steel sheets and strip
- KS M 0011, Methods for determination of pH of aqueous solutions
- KS M 8165, Antimony trioxide
- KS M 8299, Barium acetate
- KS M 8348, Sodium cyanide
- KS M 8389, Sodium dichromate dihydrate
- KS M 8432, Hexamethylenetetramine
- KS M 8526, Disodium hydrogenphosphate
- KS M 8544, Copper(II) chloride dihydrate

KS M ISO 6353 – 2, Reagents for chemical analysis — Part 2: Specifications — First series
KS M ISO 6353 – 3, Reagents for chemical analysis — Part 3: Specifications — Second series
JIS K 0557, Water used for industrial water and wastewater analysis
JIS K 8013, Zinc powder
JIS K 8252, Ammonium peroxodisulfate (Reagent)
JIS K 8284, Diammonium hydrogen citrate (Reagent)
JIS K 8550, Silver nitrate
JIS K 8625, Sodium carbonate
JIS K 8917, Hydroiodic acid
JIS K 9012, Trisodium phosphate 12-water (Reagent)

3 Equipment

Equipment required for salt spray test shall include spray device, storage tank of salt solution for test, test specimen support, spray collection container, spray chamber equipped with a thermostat, salt solution supply tank, compressed air supply system, air saturator and exhaust gas system and the following conditions shall be satisfied.

- a) The spray device shall have the performance to spray water on the test specimen evenly from the top (See Annex 2).
- b) The volume of spray chamber shall be 0.2 m^3 and more. However, the shape and measurements of spray chamber may be selected discretionally.
- c) The ceiling or lid of spray chamber shall have a shape that a drop of solution from the inside of the spray chamber does not fall down on top of the test specimen.
- d) Corrosive material shall not be used for device material.
- e) Equipment shall have a structure that the temperature inside the spray chamber and spraying are not affected by outside air and solution dropped on the test specimen is not used again for the test.
- f) The test specimen support shall be able to maintain the test specimen at a fixed angle¹⁾.
- g) The spray collection container shall be a clean container with the diameter of collection surface at 100 mm and the horizontal collection area of approximately 80 cm^2 and it shall be placed on two or more places to check uniform spraying. For example, one container shall be placed close the spray device and the other container shall be placed far from the spray device around the test specimen.
- h) The exhaust gas system shall not be affected by the wind pressure of outside air.
- i) The maintenance of equipment shall be carried out correctly to satisfy the specified conditions always.

4 Test Specimen

The measurements and shape of test specimen may be a plate in measurements of $70 \text{ mm} \times 150 \text{ mm} \times 1.0 \text{ mm}$ or $60 \text{ mm} \times 80 \text{ mm} \times 1.0 \text{ mm}$. Also, other measurements and shape of test specimen may be used according to the agreement between parties to the transaction.

NOTE Two different kinds of metallic test specimens which could affect to each other shall not be tested at the same time.

5 Production of Test Specimen

- 1) The material of test specimen support shall be glass, rubber, plastic or wood coated with a proper method and the test specimen shall be supported at the bottom of back side or the sides. If the test specimen cannot be maintained at a fixed position, it may be hung with a glass hanger or vinyl string, and in such case, the bottom of the test specimen shall be supported if necessary.

Test specimen shall not have contamination or crack. The followings shall apply for the production of test specimen.

- a) The cross section of test specimen shall be protected with coating material²⁾ which is safe under the test condition as necessary.
- b) The test specimen of metal and metallic film shall be cleaned in advance with proper methods. Clean the test specimen with a proper method which could deal with the surface property and contamination. Abrasives except for precipitated calcium carbonate in the form of paste, aluminum oxide and magnesium oxide and cleaning agents which create corrosive or protection film shall not be used. Also, after treating the test specimen, it shall be prevented from being contaminated again.
- c) The test specimen coated with paint and non-metallic film shall not be cleaned or applied with other treatment before test. However, attachments which are not appropriate for the test may be removed. Also, in case the measurement of corrosion at the damaged part is required, scratch the film to expose metal before carrying out the test. In such case, the scratching method shall be determined according to the agreement between parties to the transaction.
For instance, the following method may be used.

EXAMPLE Protect 5 mm of the test specimen with coating material and scratch an "×" at the place of 1/2 ~ 1/3 below in the longitudinal direction of test specimen. Scratches shall have a depth equivalent to the basic metal surface and the constant sectional dimension. A cutter knife and razor may be used as equipment to create a scratch.

6 Angle and Position of Test Specimen during the Test

The angle and position of test specimen in the spray chamber during the test shall satisfy the following conditions (See Annex 3).

- a) The angle of test specimen to the vertical line shall be $20^\circ \pm 5^\circ$. However, in case of subsidiary material, the angle of its effective surface to the vertical line shall be $20^\circ \pm 5^\circ$.
Also, other angles may be used according to the agreement between parties to the transaction.
The surface of test specimen shall come into contact with the movement of spray solution freely and the test specimen shall be placed in the spray chamber not to be orthogonal to the flowing direction of spray solution from the spray nozzle.
- b) The test specimen shall not come into contact with any object except for the support.
- c) The position and gap between test specimens shall not interrupt free fall of spray.
- d) A drop of salt solution from one test specimen shall not fall down on top of other test specimens.

7 Salt Solution for Test

7.1 Production method of salt solution for test

Salt solution shall be produced as follows.

- a) **Salt** Special grade sodium chloride provided in KS M ISO 6353-2(R32) or equivalent shall be used³⁾.
- b) **Water** Deionized water or distilled water with the conductivity of 20 $\mu\text{S}/\text{cm}$ or less at $(25 \pm 2)^\circ\text{C}$ shall be used.
Also, the conductivity shall be 1 $\mu\text{S}/\text{cm}$ or less.

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- 2) Coating material shall include tape, paint and paraffin.
 - 3) "Or equivalent" means that when sodium chloride is measured with atomic absorption spectrometry or similar analysis method, copper contents shall be below 0.01 g/kg and nickel contents shall be below 0.01 g/kg. Also, sodium iodide shall not exceed 1.0 g/kg and the total amount of impurities shall not exceed 5.0 g/kg when it is converted into dried salt.

- c) **Production method** Dissolve salt specified in a) in water specified in b) to make the salt concentration of (50 ± 5) g/L. Measure the density of product using a hydrometer to see if the density falls in the range between 1.029 and 1.036 at 25 °C.

If the density is outside the range, produce the salt solution again.

7.2 Regulating pH

7.2.1 Neutral salt spray test

The salt solution for test shall have no suspended material before spraying ⁴⁾ and pH of spray solution collected when it is sprayed shall fall between pH 6.5 and pH 7.2 ⁵⁾.

When adjusting pH, 0.1 mol/L solution of sodium hydroxide provided in KS M ISO 6353–2(R34) or hydrochloric acid provided in KS M ISO 6353–2(R13) may be used as necessary.

The pH measurement shall be carried out at (25 ± 2) °C according to KS M 0011.

Also, pH may be adjusted using pH test paper which can be read up to 0.3 in routine check.

7.2.2 Acetic acid salt spray test

The salt solution for test shall have no suspended material before spraying ⁴⁾ and acetic acid provided in KS M ISO 6353–2(R1) shall be applied properly so that the spray water collected when spraying shall be pH 3.1~3.3. In order to make pH of collected solution fall in the regulated range, pH of salt solution may be adjusted between 3.0 and 3.1.

The pH measurement shall be carried out at (25 ± 2) °C according to KS M 0011. Acetic acid provided in KS M ISO 6353–2(R1) or sodium hydroxide provided in KS M ISO 6353–2(R34) shall be added properly as necessary.

Also, pH may be checked using pH test paper which can be read up to 0.1 in routine check.

NOTE It is proper to use 0.1 mol/L solution of acetic acid and sodium hydroxide.

7.2.3 CASS test

Add $(0.205 + 0.015)$ g of copper chloride (II) $[(0.26 \pm 0.02)$ g in case of copper chloride (II) dihydrate provided in KS M 8544] to 1 L of salt solution for test. Next, adjust pH according to the method provided in clause 7.2.2.

4) If the suspended material cannot be disappeared well after mixing the solution properly, use the filtered solution with a filter paper.

5) The following methods may be used to make pH of collected solution when spraying salt solution for test at 35 °C fall between 6.5 and 7.2.

- In case pH of salt solution is adjusted at room temperature and sprayed at 35 °C, pH of the collected solution becomes higher than the previous pH of the solution due to the volatilization of dissolved carbon dioxide in the solution. Therefore, in case of adjusting pH of salt solution to (25 ± 2) °C, maintain pH at approximately 6.5.
- Boil salt solution for approximately 30 seconds, cool down to 25 °C or maintain at 35 °C for 48 hours and adjust pH.
- Adjust salt solution and pH using water which is boiled at a temperature of 35 °C and more not to contain carbon dioxide (NOTE 4 in clause 4 of JIS K 0557).

8 Supply Air

In order to spray salt solution, the compressed air which is sent to the spray nozzle shall not include oil and dust, and the pressure of the compressed air shall be maintained between 0.07 MPa and 0.17 MPa. It shall be maintained at (0.098 ± 0.010) MPa. To prevent change in the salt concentration of salt spray, the relative humidity of supply air shall be maintained 95 ~ 98 % when spraying. For this, if the pressure of supply air is 0.098 MPa in neutral salt spray test, the temperature of supply air saturator shall be maintained at (47 ± 2) °C. Also, water in air saturator shall be replaced at fixed periods to remove impurities in air.

9 Conditions of Spray Chamber

The followings shall apply for the conditions of test specimen.

- a) **Temperature** The temperature near the test specimen support inside the spray chamber shall be maintained at (35 ± 2) °C in neutral and acetic acid salt spray test and (50 ± 2) °C in CASS test. The temperature shall be measured at a position which is 100 mm and more away from the wall.
- b) **Temperature and water level of storage tank of salt solution for test** The temperature of storage tank of salt solution for test shall be maintained at (35 ± 2) °C in neutral and acetic acid salt spray test and (50 ± 2) °C in CASS test. Also, maintain the constant water level for the container of salt solution for test.
- c) **Spray** Spray solution shall free fall and the spray nozzle shall face in the direction that spray solution does not apply directly to the test specimen so that direct spraying is blocked.
- d) **Collected spray solution** Operate for 24 hours with the test specimens of defined measurements and shape while the conditions of spray chamber are satisfied. At this time, the collected amount of spray solution shall be (1.5 ± 0.5) mL per hour on average at each horizontal collection surface of 80 cm². In such case, the salt concentration of collected spray shall be (50 ± 5) g/L. Also, pH of collected salt solution shall fall between 6.5 and 7.2 in the neutral salt spray test and between 3.1 and 3.3 in the acetic acid salt spray test and CASS test. pH shall be measured according to the method provided in clause 7.2.

NOTE In case of measuring with a hydrometer, if the density is in between 1.029 and 1.036 at 25 °C in case of neutral salt spray test, the salt concentration of spray shall meet the provision.

10 Reuse of Equipment

If the equipment has been used for spray test using other salt solution for test or other purposes, clean the equipment before using again.

When resuming the test, the equipment shall be operated at least 24 hours before installing the test specimen in the water tank and it shall be checked that pH of the collected salt solution is same with the defined value throughout the whole spraying period.

11 Reproducibility Evaluation Method of Test Equipment

In order to confirm the reproducibility of test result in one test equipment or the reproducibility of test result in the same test equipment in a different testing laboratory, it shall be necessary to check the operation of equipment periodically according to clauses 11.1 ~ 11.3.

11.1 Neutral salt spray test

11.1.1 Comparison test specimen

In order to confirm the operation of equipment, 4 comparison test specimens of SPCE steel sheet which have the surface of 70 mm×150 mm in measurements and 1 mm±0.2 mm in thickness with no fault according to KS D 3512 shall be used [illumination (geometric mean), deviation of $R_a = (0.8 \pm 0.3) \mu\text{m}$. Except, value of direction according to the rolling direction]. These test specimens shall be cut from the cold rolled steel sheet.

Before carrying out the test, clean the comparison test specimen carefully. While cleaning, contamination, oil and other foreign substances that could influence the test result shall be removed. In such case, use the following method.

- a) Clean the comparison test specimen properly with appropriate organic solvent (boiling point range of hydrocarbon, 60~120 °C) using clean and soft brush and ultrasonic cleansing device. Carry out cleaning inside the container filled with solvent at room temperature. After washing, rinse the comparison test specimen with new solvent and dry.
- b) A different method may be used according to the agreement between parties to the transaction if the same result can be obtained. Weight 1 mg of comparison test specimen. Protect the one side of comparison test specimen with coating material such as adhesive tape.

11.1.2 Arrangement of comparison test specimen

Place 4 comparison test specimens at four corners inside the chamber at an angle of $20^\circ \pm 5^\circ$ with the side with no coating material facing upward. The support for comparison test specimen shall be made of inactive material such as plastic or coated. The bottom of the comparison test specimen shall be placed almost same as the top of the spray collection container to expose same as the test specimen of plate shape. The test time shall be 96 hours.

11.1.3 Measurement of weight loss

After finishing the test, remove the coating material and rinse the comparison test specimen with cold water to remove salt deposit. Place the comparison test specimen on the cleaning solution (provided by adding 1 volume of hydrochloric acid provided in KS M ISO 6353-2(R13) with 1 volume of water provided in clause 7.1 b) [50 % (v/v)] added with 3.5 g of hexamethylphosphoric triamide provided in KS M 8432 as corrosion-inhibitor for every 1 L and repeat washing until there is no change in weight loss to remove corrosive material properly. The temperature of cleaning solution at $(25 \pm 2)^\circ\text{C}$ shall be proper. After removing corrosive substance, wash the comparison test specimen with water at room temperature properly and dry.

NOTE For washing with water properly, it shall be proper to wash the floating material on the surface away with tap water.

Weight 1 mg of comparison test specimen and calculate the mass loss in g/m^2 .

11.1.4 Inspection of equipment operation status

If the mass loss in any of four test specimens is $(140 \pm 30) \text{g/m}^2$, it indicates that the equipment is being operated satisfactorily.

NOTE A zinc sheet may be used instead of a steel sheet. In such case, the measurement, washing and weight loss measurement method of test specimen shall be carried out according to clause 11.2, and if the mass loss is $(50 \pm 15) \text{g/m}^2$, it shall be regarded that the equipment is being operated satisfactorily.

11.2 Acetic acid salt spray test

11.2.1 Comparison test specimen

In order to confirm the operation of the equipment, 4 comparison test specimens of zinc sheet which have the surface of 70 mm×150 mm in measurements and 1 mm±0.2 mm in thickness with no fault satisfying the chemical ingredients in Table 1 shall be used [Illumination (geometric mean) deviation of $R_a = (0.05 \pm 0.02) \mu\text{m}$. Except, value of direction according to the rolling direction]. These test specimens shall be cut from the sheet. Cut 4 test specimens from the sheet in the rolling direction.

Table 1 — Chemical components of comparison test specimen (zinc sheet)

Unit: %

Zn	Pb	Fe	Cd	Cu
98.5 and more	1.3 or less	0.09 or less	0.40 or less	0.01 or less

Before carrying out the test, clean the comparison test specimen carefully. While washing, contamination, oil and other foreign substances that could influence the test result shall be removed. In such case, use the following method.

- Clean the comparison test specimen properly with appropriate organic solvent (boiling point range of hydrocarbon, 60~120 °C) using clean and soft brush and ultrasonic cleansing device. Carry out cleaning inside the container filled with solvent at room temperature. After washing, rinse the comparison test specimen with new solvent and dry.
- A different method may be used according to the agreement between parties to the transaction if the same result can be obtained.

Weight 1 mg of comparison test specimen. Protect the one side of comparison test specimen with coating material such as adhesive tape.

11.2.2 Arrangement of comparison test specimen

Place 4 comparison test specimens at four corners inside the chamber at an angle of $20^\circ \pm 5^\circ$ with the side with no coating material facing upward. The support for comparison test specimen shall be made of inactive material such as plastic or coated. The bottom of the comparison test specimen shall be placed almost same as the top of the spray collection container to expose same as the test specimen of plate shape. The test time shall be 24 hours.

11.2.3 Measurement of weight loss

After finishing the test, remove the coating material and rinse the comparison test specimen with cold water to remove salt deposit. Place the test specimen on chromic acid solution ⁶⁾ which satisfies the chemical ingredients in Table 2 or chromic acid solution (300 g/L) diluted with water specified in 7.1 b) at $(25 \pm 2)^\circ\text{C}$ for 3 minutes or chromic acid solution (200 g/L) at 80°C for 1 minutes and repeat washing until there is no change in weight loss to remove corrosive material properly. The temperature of cleaning solution at $(25 \pm 2)^\circ\text{C}$ shall be proper. After removing corrosive material, wash the comparison test specimen properly with warm water at the temperature of $(40 \pm 5)^\circ\text{C}$, dry in a dryer at the temperature of 105°C , and cool down at room temperature.

- Chromic oxide and chromic acid solutions are toxic, so special caution shall be taken for handling to prevent skin and membrane from coming into contact.

Weight 1 mg of comparison test specimen and calculate the mass loss in g/m^2 .

Table 2 — Chemical components of chromic oxide (VI)(chromic acid anhydride)

Unit: %

Solution status	Chloride Cl	Sulphate SO ₄	Alkaline earths and alkali (sulphate)	Sediment from ammonia solution	Content
Within limits	0.005 or less	0.005 or less	0.1 or less	0.03 or less	98.0 and more

11.2.4 Inspection of equipment operation status

If the mass loss in any of four test specimens is $(40 \pm 12) \text{ g/m}^2$, it indicates that the equipment is being operated satisfactorily.

11.3 CASS test

11.3.1 Comparison test specimen

Clause 11.2.1 shall apply.

11.3.2 Arrangement of comparison test specimen

Clause 11.2.2 shall apply.

11.3.3 Measurement of weight loss

Clause 11.2.3 shall apply.

11.3.4 Inspection of equipment operation status

If the mass loss in any of four test specimens is $(95 \pm 25) \text{ g/m}^2$, it indicates that the equipment is being operated satisfactorily.

12 Beginning of Test

9. Check the conditions of spray chamber, stop spraying temporarily, place the test specimens in the spray chamber and begin the test.

13 Continuation of Test

The test shall be carried out continuously during the test period in principle. However, it may be carried out intermittently according to the agreement between parties to the transaction.

In case of suspending the test for inspecting the test specimen and entering, only spraying shall be suspended to minimize the suspension time.

14 Test Time

The test time provided for material and product standard shall be used. If it is not provided, it shall be determined by agreement between parties to the transaction.

Also, the recommended exposure time shall be 2 h, 6 h, 24 h, 48 h, 96 h, 168 h, 240 h, 480 h, 720 h or 1 000 h.

NOTE If corrosion is discovered, the test may be carried out until corrosion occurs.

15 Treatment of Test Specimen after Test

After test, the test specimen shall be treated as follows.

- a) Remove the test specimen from the test water tank carefully and dry the test specimen immediately for 0.5~1.0 hour.
- b) To remove sodium chloride attached on the surface of test specimen, wash the test specimen with water whose temperature ranges between 15~40 °C and dry immediately.
NOTE Drying may be carried out with the air compressed at the pressure not exceeding 200 kPa at a position appropriately 300 mm away from the test specimen.
- c) In case of removing corrosion products, it shall be done by the mechanical method including brushing, ultrasonic irrigation, fine particle spraying or water spraying, chemical method, method by electrolysis (See Reference Table 1 or Reference Table 2) or a combination of these methods.

16 Judgment Method

The judgment of test result shall be made according to the following items. However, other different methods⁷⁾ may be used according to the agreement between parties to the transaction.

- a) Rating number
- b) Appearance after test
- c) Appearance after most corrosion products are removed
- d) Corrosion defect, in other words, number and distribution of holes, cracks and bloating ; This may be evaluated by the method mentioned in ISO 10289, Methods for corrosion testing of metallic and other inorganic coatings on metallic substrates — Rating of test specimens and manufactured articles subjected to corrosion tests.
- e) Time taken until initial sign of corrosion appears
- f) Mass change
- g) Changes discovered through a microscopic examination
- h) Change in mechanical property

NOTE When judging the corrosion result according to the area method, a plate of 70 mm×150 mm×1 mm shall be used.

17 Recording

The following items shall be recorded.

- a) Type of test
- b) Name, form of test equipment and method of spray device
- c) Shape and measurement of test specimen and subsidiary material or number or type of parts
- d) Washing method of test specimen before and after test
- e) Number of test specimens provided in the test
- f) Production method of test specimen

- 7) Other different methods include appearance, recording of change by photomicrograph and method according to the time elapsed until the first sign of corrosion occurs.