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Methods of test for hot dip galvanized coatings

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of International Trade and Industry through deliberations at Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law. Consequently **JIS H 0401 : 1983** is replaced with **JIS H 0401 : 1999**.

In this revision, the conformity with normative references and the corresponding International Standard and the revision of the structure in accordance with **JIS Z 8301 : 1996** are mainly executed.

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Methods of test for hot dip galvanized coatings

Introduction This Japanese Industrial Standard has been prepared based on **ISO 1459** *Metallic coatings—Protection against corrosion by hot dip galvanizing—Guiding principles*, **ISO 1460** *Metallic coatings—Hot dip galvanized coatings on ferrous metals—Determination of the mass per unit area—Gravimetric method* and **ISO 1461** *Metallic coatings—Hot dip galvanized coatings on fabricated ferrous products—Requirements*, published in 1973 as the first edition, in such a manner that the parts (shape and dimension) in the corresponding International Standard are contained without modifying the technical contents and the items not specified in the corresponding International Standard are complementarily provided as a part of the Japanese Industrial Standard.

The portion with sidelines or dotted underlines in this Standard are the matters not specified in the corresponding International Standard.

1 Scope This Japanese Industrial Standard specifies the methods of test for hot dip galvanized coatings (hereafter referred to as “coating”) executed on iron and steel products. This Standard may be applicable to the electrolytic zinc-coating in place of the hot dip galvanized coating.

Remarks : The International Standards corresponding to this Standard are as follows.

ISO 1459-1973 *Metallic coatings—Protection against corrosion by hot dip galvanizing—Guiding principles*

ISO 1460-1992 *Metallic coatings—Hot dip galvanized coatings on ferrous materials—Gravimetric determination of the mass per unit area*

ISO 1461-1973 *Metallic coatings—Hot dip galvanized coatings on fabricated ferrous products—Requirements*

2 Normative references The following standards contain provisions which, through reference in this Standard, constitute provisions of this Standard. The most recent editions of the standards indicated below shall be applied.

JIS B 7729 *Erichsen cupping testers*

JIS G 3442 *Galvanized steel pipes for ordinary piping*

JIS K 1202 *Solid caustic soda*

JIS K 1310 *Hydrochloric acid*

JIS K 1431 *Potassium hydroxide*

JIS K 1433 *Copper sulfate for industrial use*

JIS K 8400 *Antimony (III) chloride*

JIS K 8407 *Antimony (III) oxide*

JIS K 8422 *Copper (II) oxide*

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JIS K 8847 *Hexamethylenetetramine*

JIS Z 2247 *Method of Erichsen cupping test*

3 General matters

3.1 Relation to product standard Classification of test methods applied to metallic coated iron and steel products, sampling method of test specimen, acceptability criteria of test and retest are specified in the respective product standards.

3.2 Classification of iron and steel products Iron and steel products shall be classified into the following seven types according to their shapes and dimensions for convenience to the test of coatings.

- a) **Pipes** Steel pipe for water supply, conduit, steel piping pipe, steel tubes for structural purposes, steel tubes for scaffolding and others
- b) **Steel sheets** Hot dip galvanized iron sheets and others
- c) **Wires** Iron wire, steel wire, wire rope, iron strand wire, steel strand wire, wire netting, barbed wire, wire cylinder, armored wire, core wire for steel core aluminum strand wire and others
- d) **Rolled steels** Rolled steels such as steel sheet, section, flat bar, steel bar and others
- e) **Prefabricated steel products** Prefabricated steel products such as iron tower, bridge, steel frame, metal fittings for shipbuilding, live hardware, tank and others
- f) **Bolts and nuts** Various kinds of bolts and nuts, cotters, washers and others
- g) **Castings and forgings** Iron casting, steel forging, steel casting, pipe joint (including that made of steel pipe), metal fittings for porcelain insulator and others

3.3 Classification of test methods The test methods shall be classified into four types as shown in Table 1 and application of each test method shall be in accordance with the respective product standards.

Table 1 Classification of Test Methods

Classification of iron and steel products	Test method								Properties test (alkali solubility test)
	Coating weight test		Copper sulphate test	Adhesion test					
	Direct method	Indirect method		Visual observation	Bending test	Erichsen test	Winding test	Hammer test	
Pipes	—	4.2	5	6.1	6.2	—	—	—	7
Steel sheets	4.1	4.2	—	—	6.2	6.3	—	—	—
Wires	—	4.2	5	—	6.2	—	6.4	—	—
Rolled steels	4.1	4.2	5	6.1	—	—	—	6.5	—
Prefabricated steel products	4.1	4.2	5	6.1	—	—	—	6.5	—
Bolts and nuts	4.1	4.2	5	6.1	—	—	—	6.5	—
Castings and forgings	4.1	4.2	5	6.1	—	—	—	6.5	—

Remarks : Numbers given in the table indicate test items corresponding thereto.

4 Method for coating mass test

4.1 Direct method

4.1.1 Test piece

a) **Sampling method of test piece** The crude material itself in manufacturing process shall be a test piece. Otherwise, the following process may be accepted according to agreement between the purchaser and the supplier:

- 1) When the crude material is too large or too heavy, or when it is inconvenient to be treated, cut off a suitable part from the original material and take it to be a test piece.
- 2) When the surface area of crude material is difficult to be determined, make an object as similar to the manufactured material as possible and take it to be a test piece.

4.1.2 Operation and calculation of coating mass After pickling, washing and drying of the test piece which the same operating method as those of other crude material represented thereby, weigh it and carry out coating. Thereafter weigh it again and the quotient of the increased amount divided by the surface area of the test piece (one side surface area for hot dip galvanized sheet) shall be a coating mass.

4.2 Indirect method

4.2.1 Test piece

a) **Sampling method of test piece** The test piece shall be taken by either of the following processes according to agreement between the purchaser and the supplier:

- 1) A product itself shall be a test piece.
- 2) A test piece shall be cut off from a coated product.
- 3) After cutting an original material representing a product in suitable size, coat it by the same method as that for the product and take it to be a test piece.

b) **Sampling position and size of test piece**

- 1) **For pipes** One piece of tubular test piece of about 60 mm in length shall be respectively taken from both ends in accordance with a) 1). When the test piece is too large, it may be cut in suitable size capable of being measured.
- 2) **For steel sheets** A test piece shall be taken by 57.2 mm × 57.2 mm or 64.5 mm in diameter in accordance with a) 2), by either method given in Fig. 1 or Fig. 2 upon the designation by agreement between the purchaser and the supplier.

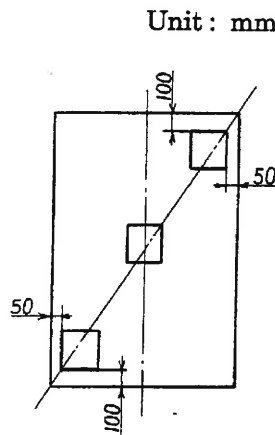


Fig. 1 Sampling position of test piece

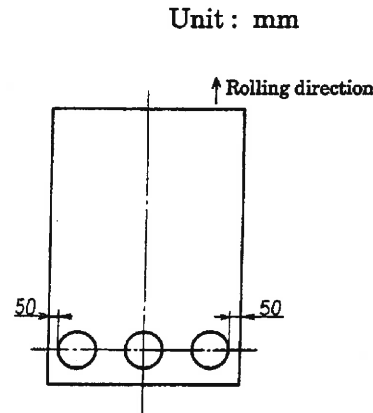


Fig. 2 Sampling position of test piece

- 2.1) **Three-point method** One sheet of the test piece from the center of the sheet, two sheets from positions at least 100 mm apart from the vertical end thereof and at least 50 mm apart from the lateral end thereof on opposite sides of diagonal line, total three sheets of the test piece shall be taken as given in Fig. 1. For those with continuous coating process, one sheet of the test piece from the center of the sheet and two sheets from positions at least 50 mm apart from both lateral ends of the sheet, total three sheets of the test piece shall be taken on one end of the sheet as given in Fig. 2.
- 2.2) **Least-point method** The test piece having the minimum coating mass among three sheets taken by the same method as the three-point method, shall be taken, or one sheet of the test piece shall be taken from an optional position at least 100 mm apart from vertical end of the sheet and at least 50 mm apart from the lateral end of the sheet.

- 3) **For wires** The test piece shall be taken by 300 to 600 mm in length in accordance with a) 2).
- 4) **For rolled steels and prefabricated steel products** The test piece shall be taken in accordance with 1), 2) or 3) of a). In 2) and 3), the test piece of shape as similar to square as possible having 100 cm² in coating area shall be a standard.
- 5) **For bolts, nuts, castings and forgings** The test piece shall be taken in accordance with 1), 2) or 3) of a). Its screw thread part may be omitted.

4.2.2 Test solution The test solution shall be used either a) or b) as follows.

- a) **Hexamethylenetetramine solution** Dissolve 3.5 g of hexamethylenetetramine specified in **JIS K 8847** in 500 ml of hydrochloric acid (synthetic) (not less than 1.18 g/cm³ in density) specified in **JIS K 1310**. Dilute the solution with pure water to 1 l.
- b) **Antimony chloride solution** Dissolve 32 g of antimony (III) chloride specified in **JIS K 8400**, or 20 g of antimony oxide specified in **JIS K 8407** in 1 l of hydrochloric acid (synthetic) (not less than 1.18 g/cm³ in density) specified in **JIS K 1310** to make a stock solution.

Add 5 ml of the stock solution to 100 ml of the above-described hydrochloric acid immediately before the test to make the test solution.

4.2.3 Cleaning of test piece The test piece shall be degreased with an organic solvent and dried if necessary. The organic solvent to be used shall be harmless to the coating.

4.2.4 Operation and calculation of coating mass The test piece shall be weighed before zinc dissolves.

The accuracy shall be 1 % or under of the presumed coating mass (the prospective coating mass). The quantity of the test solution shall be determined so as to be minimum 10 ml per 1 cm² of the surface area of the test piece. The test piece shall be dipped completely in the test solution at room temperature, and left until the coating film is completely dissolved. The progressive generation of hydrogen in the solution⁽¹⁾ ⁽²⁾ stops, which shows that the dissolution has completed. Then, rinse the test piece in the running water, wipe it well with cotton cloth, dry it sufficiently, or immerse the test piece in alcohol and dry it immediately, and weigh the mass again with the accuracy specified in this paragraph. After weighing, obtain the area *S* with the accuracy of measurement of 1 % or under of the actual surface area.

Notes (1) During the test, the temperature of the test solution shall not exceed 38 °C.

(2) The test solution may be repeatedly used as long as its coating layer can easily be removed.

- a) **For pipes, rolled steels, prefabricated steel products, castings and forgings** The coating mass shall be calculated from the following formula:

$$A = \frac{W_1 - W_2}{S} \times 10^6$$

where, *A* : coating mass (g/m²)

W_1 : mass of test piece before removing coating layer (g)

W_2 : mass of test piece after removing coating layer (g)

S : surface area of test piece (mm²)

b) **For steel sheets** In three-point method, the mean value of three sheets of the test piece shall be obtained by weighing them to 0.01 g both before and after operation, and the quotient of the mean value divided by the surface area on one side shall be taken as the coating mass. In least-point method, the test piece shall be weighed for every one sheet and the minimum coating mass shall be taken as the coating mass.

c) **For wires** The cleaned test piece shall be weighed to 0.01 g. When the test piece is too long by comparing it with its vessel, it shall be adjusted to be completely dipped in the test solution⁽¹⁾ ⁽²⁾ by suitably bending or winding the wire.

When the generation of hydrogen lessens and the coating layer is removed, the test piece shall be taken out, washed with water, and wiped sufficiently with a cotton cloth. Thereafter, dry it thoroughly. After weighing it to 0.01 g again, its diameter shall be measured to 0.01 mm in mutually rectangular directions at the same position and their mean value obtained.

The coating mass shall be calculated from the following formula:

$$A = \frac{W_1 - W_2}{W_2} \times d \times 1960$$

where, A : coating mass (g/m²)

W_1 : mass of test piece before removing coating layer (g)

W_2 : mass of test piece after removing coating layer (g)

d : diameter of test piece after removing coating layer (mm)

1960 : constant

d) **For bolts and nuts** The coating mass shall be calculated from the following formula:

$$A = \frac{W_1 - W_2}{S} \times 10^6$$

where, A : coating mass (g/m²)

W_1 : mass of test piece before removing coating layer (g)

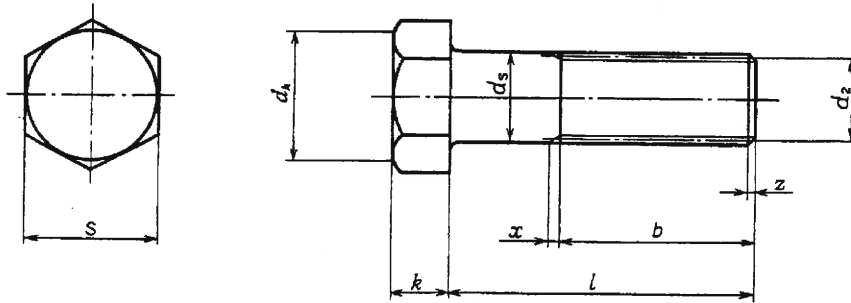
W_2 : mass of test piece after removing coating layer (g)

S : surface area of test piece (mm²)

Further, the surface area S (mm²) of test piece including screw thread part shall be calculated from the following formula. Furthermore, to respective numerical values used for calculation, nominal dimensions may be applied.

1) **Hexagon bolt (Metric screw thread)**

$$S = 1.95sd_k + 3.46sk - 0.187s^2 - 0.122d_k^2 + 3.14d_s[l - (b+x/2)] + 1.30d_s z - 0.130z^2 + (b-z+x/2)(5.27d_2 + 0.267p^2/d_2)$$

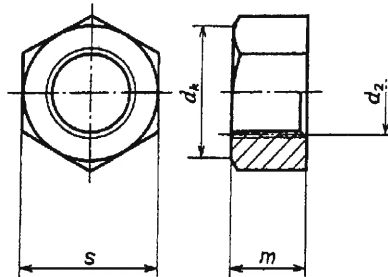


- where,
- S : surface area of test piece (mm²)
 - s : width across flats (mm)
 - k : height (mm)
 - d_k : circular diameter of head (mm)
 - d_s : shaft diameter (mm)
 - d_2 : pitch diameter (mm)
 - l : underhead length (nominal length) (mm)
 - b : thread length (mm)
 - x : length of incomplete thread (mm)
 - z : length of screw end (mm)
 - p : pitch

Fig. 3 Shape and dimensions of hexagon bolt (Metric screw thread)

2) **Hexagon nut (Metric screw thread)**

$$S = 1.95sd_k + 3.46sm - 0.187s^2 - 0.122d_k^2 - 1.57d_2^2 + m(5.19d_2 + 0.263p^2/d_2)$$



where, S : surface area of test piece (mm²)
 s : width across flats (mm)
 m : height (mm)
 d_2 : pitch diameter (mm)
 d_k : circular diameter of head (mm)
 p : pitch

Fig. 4 Shape and dimensions of hexagon nut (Metric screw thread)

5 **Method for copper sulphate test**5.1 **Test piece**

- a) **Sampling method of test piece** The test piece shall be taken in accordance with 4.2.1 a).
- b) **Sampling position and size of test piece**
 - 1) **For pipes** The test piece shall be taken in accordance with 4.2.1 b) 1).
 - 2) **For wires** The test piece shall be taken by 150 mm min. in accordance with 4.2.1 a) 1).
 - 3) **For rolled steels and prefabricated steel products** The test piece shall be taken by 100 mm⁽³⁾ in accordance with 1), 2) or 3) of 4.2.1 a). For sheets its size shall be 100 mm × 100 mm⁽³⁾.
 - 4) **For bolts and nuts** The test piece shall be taken in accordance with 1), 2) or 3) of 4.2.1 a). When the test piece exceeds 150 mm in length, it may be cut⁽³⁾ to an appropriate length suitable for the test, or may be partially immersed.
 - 5) **Castings and forgings** The test piece shall be taken in accordance with 1), 2) or 3) of 4.2.1 a). When the test piece is too large (the coating area exceeds 400 cm²), it may be cut⁽³⁾ to an appropriate size, or may be partially immersed.

5.2 Test solution The test solution shall be prepared by mixing 100 ml of water per 36 g of copper sulfate for industrial use specified in **JIS K 1433**, dissolved by heating, added with excessive powdered cupric hydroxide (II) $[\text{Cu}(\text{OH})_2]$ (pure for chemistry)⁽⁴⁾ ⁽⁵⁾ to neutralize free sulfuric acid, mixed and left for 24 h. Thereafter, it is filtered and conditioned at 18 °C to take the solution of 1.186 to 1.188 g/cm³ in density as the test solution.

5.3 Amount of test solution

- a) **For pipes, rolled steels, prefabricated steel products, bolts and nuts, and castings and forgings** Not less than 6 ml test solution per cm² of its surface area shall be used so that the test piece is completely dipped therein and the same solution may be used until the dipping number reaches 20 times.
- b) **For wires** A vessel not reacting with copper sulfate solution such as a plastic vessel or the like having 50 mm min. in inner diameter for a wire having under 2.6 mm in diameter, or having 75 mm min. in inner diameter for a wire having 2.6 mm min. in diameter shall be used and the depth of the solution shall be 100 mm min.

This amount can be used for 8 test pieces maximum. In the test for exceeding 8 test pieces, its test solution shall be exchanged.

5.4 Cleaning of test piece As in 4.2.3.

5.5 Operation A cleaned test piece shall be quietly dipped into the center of the test solution kept at 16 to 20 °C for 1 min. At that time, the solution shall not be mixed or the wall of the vessel shall not be touched.

The test piece taken-out shall be washed immediately in water and the copper adhered to the coating surface is wiped off with a brush or the like.

This operation shall be repeated.

5.6 Judgment of end point

5.6.1 Where becoming end point Where brilliant adherent metallic copper is deposited on the substrate of the coating.

5.6.2 Where not becoming end point

- a) Where not corresponding to 5.6.1.
- b) The following cases in 5.6.1 do not become end point:
 - 1) The case where the whole area deposited with brilliant adherent metallic copper does not reach 0.05 cm².
 - 2) The case where the brilliant adherent metallic copper can be scraped off with dull tool such as the back of the knife and coating layer appears under copper.
 - 3) The case where the brilliant adherent metallic copper is deposited on the corner of the test piece or within 10 mm from its end.

- 4) The case where the brilliant metallic copper is deposited on the cut and scratched parts generated after the coating and the part adjacent thereto.

Remarks : Supplementary test When there is any question as to the presence/absence of a coating layer under adherent metallic copper for various steel products, the adherent metallic copper is scraped off and one drop or several drops of dilute hydrochloric acid are dripped on this position. When there is some coating layer, the judgment can be made according to the active generation of hydrogen gas.

5.7 Acceptability judgment The case where the operation of 5.5 is carried out by specified times only and the end point is not reached, shall be acceptable.

Notes ⁽³⁾ When the concentration of the copper sulfate solution is remarkably reduced due to the excessively large dimension of the uncoated surface area, this part shall be coated with a suitable coating material.

⁽⁴⁾ The amount of cupric hydroxide is about 10 g per 10 l of the solution. It is determined by the precipitation at the bottom of the vessel that the excessive amount of the cupric hydroxides exists.

⁽⁵⁾ About 8 g of copper (II) oxide specified in **JIS K 8422** in place of cupric hydroxide per 10 l of the solution may be used. In this case, it shall be left for 48 h.

Otherwise, about 12 g of powder basic copper carbonate [$\text{CuCO}_3\text{-Cu(OH)}_2$] (pure for chemistry) per 10 l of the solution may be used. In this case, it shall be left for 24 h.

6 Methods for adhesion test

6.1 Method by visual observation The presence of cracks or exfoliation of the coating layer due to the ordinary handling shall be examined.

6.2 Bending test When bent by a specified angle with an inner side radius of specified times the diameter or thickness of the test piece, the surface condition of the coating layer such as the bent part or welded part shall be examined.

6.3 Erichsen test The Erichsen test shall be carried out by using an Erichsen tester specified in **JIS B 7729** according to the method for Erichsen test specified in **JIS Z 2247**, as appropriate, and the surface condition of the coating layer shall be examined.

6.4 Winding test When wound closely around a circular cylinder having a specified diameter by specified times, the surface condition of the coating layer shall be examined.

6.5 Hammer test When a blow is struck with a hammer, the surface condition of the coating layer shall be examined.

6.5.1 Apparatus for hammer test The apparatus for the hammer test is given in Fig. 5.

6.5.2 Setting of test piece The test piece shall be horizontally fixed so as not to be moved easily with a blow of a hammer, and the test surface shall be perpendicular to the hammer.

6.5.3 Test piece The test piece shall be 8 mm min. in thickness, and the surface to be tested shall be flat.

6.5.4 Operation The test surface shall be put horizontally and the hammer shall be naturally fallen from the position where its arm is vertical centering the support table. Blows shall be applied to 5 parallel positions at 4 mm intervals and the exfoliation and embossment between their traces shall be examined. The area within 10 mm from the corner or end shall not be tested. Further, each position shall be struck only once.

Unit: mm

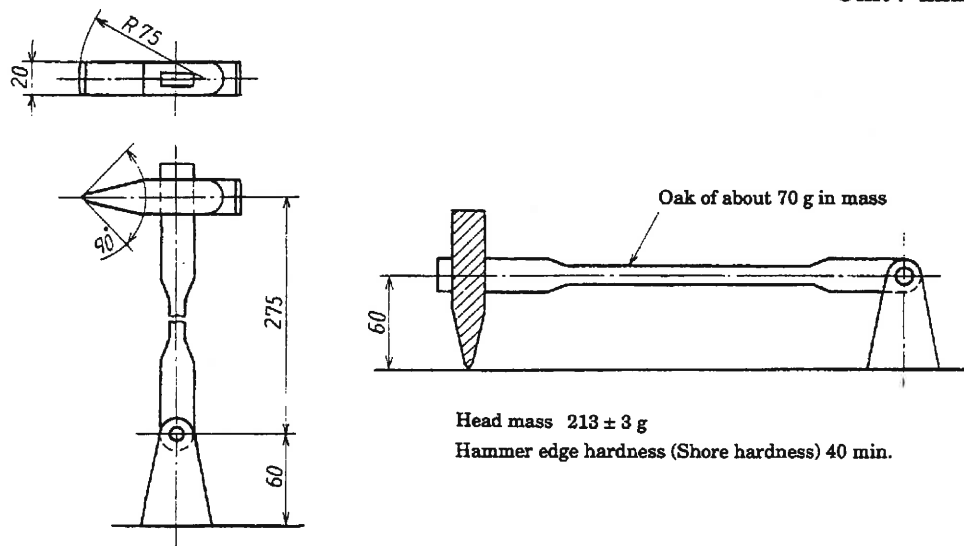


Fig. 5 Apparatus for hammer test

7 Method for properties test⁽⁶⁾ (alkali solubility test)

7.1 Test piece The test piece shall be taken in accordance with 4.2.1 a) 1) and its shape and dimensions shall conform to Table 2.

Table 2 Shape and dimensions of test piece

Designation of pipe	A	10	15	20	25	32	40
	B	$\frac{3}{8}$	$\frac{1}{2}$	$\frac{3}{4}$	1	$1\frac{1}{4}$	$1\frac{1}{2}$
Length of test piece (mm)	30						
Shape of section	Whole circle	Whole circle	Whole circle	$\frac{1}{2}$ circle	$\frac{1}{2}$ circle	$\frac{1}{2}$ circle	$\frac{1}{2}$ circle

Designation of pipe	A	50	65	80	90	100	125
	B	2	$2\frac{1}{2}$	3	$3\frac{1}{2}$	4	5
Length of test piece (mm)	30						
Shape of section	$\frac{1}{4}$ circle	$\frac{1}{4}$ circle	$\frac{1}{6}$ circle	$\frac{1}{6}$ circle	$\frac{1}{6}$ circle	$\frac{1}{6}$ circle	$\frac{1}{8}$ circle

Designation of pipe	A	150	175	200	225	250	300
	B	6	7	8	9	10	12
Length of test piece (mm)	30						
Shape of section	$\frac{1}{8}$ circle	$\frac{1}{8}$ circle	$\frac{1}{8}$ circle	$\frac{1}{8}$ circle	$\frac{1}{8}$ circle	$\frac{1}{8}$ circle	$\frac{1}{8}$ circle

7.2 Test solution The test solution shall be prepared by a method wherein 20 g solid caustic soda specified in JIS K 1202 or 28 g potassium hydroxide specified in JIS K 1431 is dissolved in 100 ml of water.

7.3 Cleaning of test piece As in 4.2.3.

7.4 Operation The amount of the test solution shall be 5 ml min. per cm² of coating area, which can completely dip the test piece, and a temperature of the test solution shall be kept at 75 to 80 °C.

When the test piece is thrown in, the coating layer dissolves as time passes. Though only a little amount of bubbles is generated while the zinc layer on surface dissolves, hydrogen gas is vigorously generated when the alloy layer is exposed and the inside of a glass vessel becomes to be filled with bubbles. Thereafter, when the iron surface is exposed, the reaction becomes slow and the generation of bubbles disappears. The time passed since the test piece has been thrown in shall be measured recognizing that time as the end point.

Further, the cross section of the test piece may be protected with a suitable coating material so that the iron substrate is not directly into contact with the test solution.

7.5 Acceptability judgment The test shall be preferably accepted if the time from throwing-in of the test piece to the end point is not less than 100 min.

Note (6) The properties test herein applies to JIS G 3442.

Informative reference: **Method for film thickness test**

- 1 **Test piece** The product itself shall be the test piece.
- 2 **Operation** The instrument for measuring the film thickness calibrated with a standard piece having a known thickness shall be used. The thickness at 5 positions min. per test piece shall be measured and their mean value shall be taken as the film thickness.

Further, to obtain a coating weight from a measured value of the film thickness, the following formula shall be used:

$$A = 7.2 \times t$$

where, A : coating weight of zinc (g/m²)
 7.2 : density of coating layer (g/cm³)
 t : film thickness (μm)

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