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JAPANESE INDUSTRIAL STANDARD

Methods of Chemical Analysis for
Copper Nickel Resistance Material

JIS

H 1413-1976
(Reaffirmed: 1984)



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Translated

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Methods of Chemical Analysis for H 1413-1976
Copper Nickel Resistance Material (Reaffirmed: 1984)

1. Scope

This Japanese Industrial Standard specifies the methods of chemical analysis for chemical components (copper, nickel, cobalt, manganese) specified in JIS C 2521.

2. General Matters

General matters common to the chemical analysis methods shall be in accordance with JIS K 0050 and JIS K 0115.

3. Sampling and Treating of Sample for Analysis

3.1 The sampling shall be in accordance with 2.3 of JIS H 0321.

3.2 When a cast sample is sampled, not less than 2 samples per one fusion (one sample when the amount of one fusion is especially very little) shall be taken to obtain the sample representing its average material quality. Cares shall be taken so that the cast sample is especially free from segregation to obtain the same quality as that of product as perfectly as possible.

3.3 The chipping of sample shall be as follows:

- (1) When there are adhering substances on the surface of sample, the surface shall be cleaned by removing them with an emery paper or the like.
- (2) Drills and other tools shall be cleaned by using alcohol and the like.
- (3) When a cast sample is chipped out, the sample is pierced by perpendicularly drilling from one side of the part near the central part and both ends or the like, and otherwise it is drilled from both sides till reaching the central core part at least. Otherwise the sample is chipped out by other suitable methods.
- (4) For chipping out the sample from product samples of wire, band, plate, etc., they are chipped out in sizes suitable for analytical operation by using a drill or other suitable tools.

Applicable Standards: See page 20.

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- (5) Because the surfaces of chips are occasionally oxidized by generation of heat when drilled, drilling is carried out by giving the drill a pressure and a number of revolutions to the degree not to oxidize them. In that case, it is prohibited to use oils or other lubricants, or otherwise to pour water or the like for cooling.

Further, cares shall be taken so that chips are not mixed with wearing powders of drill.

- (6) The size of chips shall not be too thick and the length shall be about 5 mm max.

3.4 Treating of Sample

- (1) All the chipped out sample (generally 50 g min.) are collected and the mixed iron powders or the like are carefully removed by using a strong magnet. At last, they are sufficiently mixed to be the sample for analysis.
- (2) When the sampling of sample for analysis is difficult to conform to the above-mentioned specification, the sampling can be separately determined depending upon the agreements between the purchaser and the manufacturer.
- (3) The sample for analysis shall be put in a desiccator and let stand for 1 h or more. Thereafter, it shall be weighed out.

3.5 Weighing of Sample The weighing of sample shall be as follows:

- (1) On weighing out the sample, cares shall be so taken that the sample represents average chemical composition by sufficiently mixing the sample.
- (2) The sample shall, as a rule, be weighed out by using a chemical balance and an amount near as specified amount shall be weighed out to necessary places referring to the indicated number of places of analytical value.

4. Expression of Analytical Value and Caution on Operation

4.1 Expression of Analytical Value The analytical value shall be expressed by percentage and rounded off to places specified in JIS C 2521 in accordance with JIS Z 8401.

4.2 Caution on Analytical Operation The caution on analytical operation shall be as follows:

- (1) The same sample shall be analyzed not less than 2 times and the result shall be confirmed.
- (2) When analyzing, a blank test shall be carried out over all operations and the measured value shall be corrected.

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5. Method for Determination of Copper

5.1 Classification of Methods The method for determination of copper shall be in accordance with electrogravimetric analysis method.

5.2 Electrogravimetric Analysis Method

5.2.1 Summary After decomposing a sample with the mixed solution of nitric acid and sulfuric acid, it is electrolyzed by using a platinum electrode and copper is deposited on a cathode. Then, its mass is measured (the electrolytic residual liquid can be used for determination of nickel).

5.2.2 Reagents The reagents shall be as follows:

- (1) Mixed acid 20 ml of water is added with 5 ml of sulfuric acid while mixing them with stirring and cooled. Thereafter, it is added with 5 ml of nitric acid and sufficiently mixed.
- (2) Ethyl alcohol (95 v/v %)

5.2.3 Devices The following devices shall, as a rule, be used.

- (1) Electrolytic beaker (refer to Fig. 1)
- (2) Platinum electrode A (refer to Fig. 2)
- (3) Platinum electrode B (refer to Fig. 3)
- (4) Semicircular watch glass (refer to Fig. 4)

5.2.4 Weighing-Out Amount of Sample 1 g of the sample shall be weighed out.

5.2.5 Operation The determination operation shall be carried out according to the following procedures:

- (1) Weigh out a sample, transfer it into an electrolytic beaker, cover that with a watch glass, add 30 ml of mixed acid and quietly decompose it at as low temperature as possible. After completion of decomposition, allow it completely to be solution by carefully heating and expel nitrogen oxide. After washing the bottom surface of watch glass and the inside wall of beaker, dilute it to about 150 ml by adding water.
- (2) Allow the platinum electrode A the mass of which is preliminarily measured to be a cathode, utilize a platinum electrode B as an anode, cover them with two semicircular watch glasses, apply a current of 0.3 to 0.4 A at a liquid temperature of 20 to 30°C (when the temperature of liquid decrease to less than 20°C, the beaker is heated with a suitable heater) and electrolyze it for one night.

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- (3) Wash the bottom surface of watch glass, the inside wall of beaker and the part of the handle of electrode exposed above the liquid surface with a small amount of water, raise the surface of electrolyte by about 5 mm with that washing water and further continue to electrolyze it for about 30 minutes.
- (4) When copper is not deposited any more longer on the handle of cathode newly dipped in the electrolyte, gradually draw up both electrodes while washing them with water as current is applied, at last quickly dip them into new water and remove the cathode (the electrolytic residual solution may be used for determination of nickel).
- (5) Raise and lower the cathode several times and wash it with water, thereafter, sufficiently wash it by using ethyl alcohol, immediately quickly dry it in an air bath at about 80°C and allow it to stand to cool in a desiccator for about 30 minutes. Thereafter, measure its mass.

5.2.6 Calculation The content of copper in sample shall be calculated from the following formula:

$$\text{Copper (\%)} = \frac{w}{W} \times 100$$

where w : mass of copper deposited on cathode (g)

W : weighed-out amount of sample (g)

Fig. 1. Electrolytic Beaker

Unit: mm

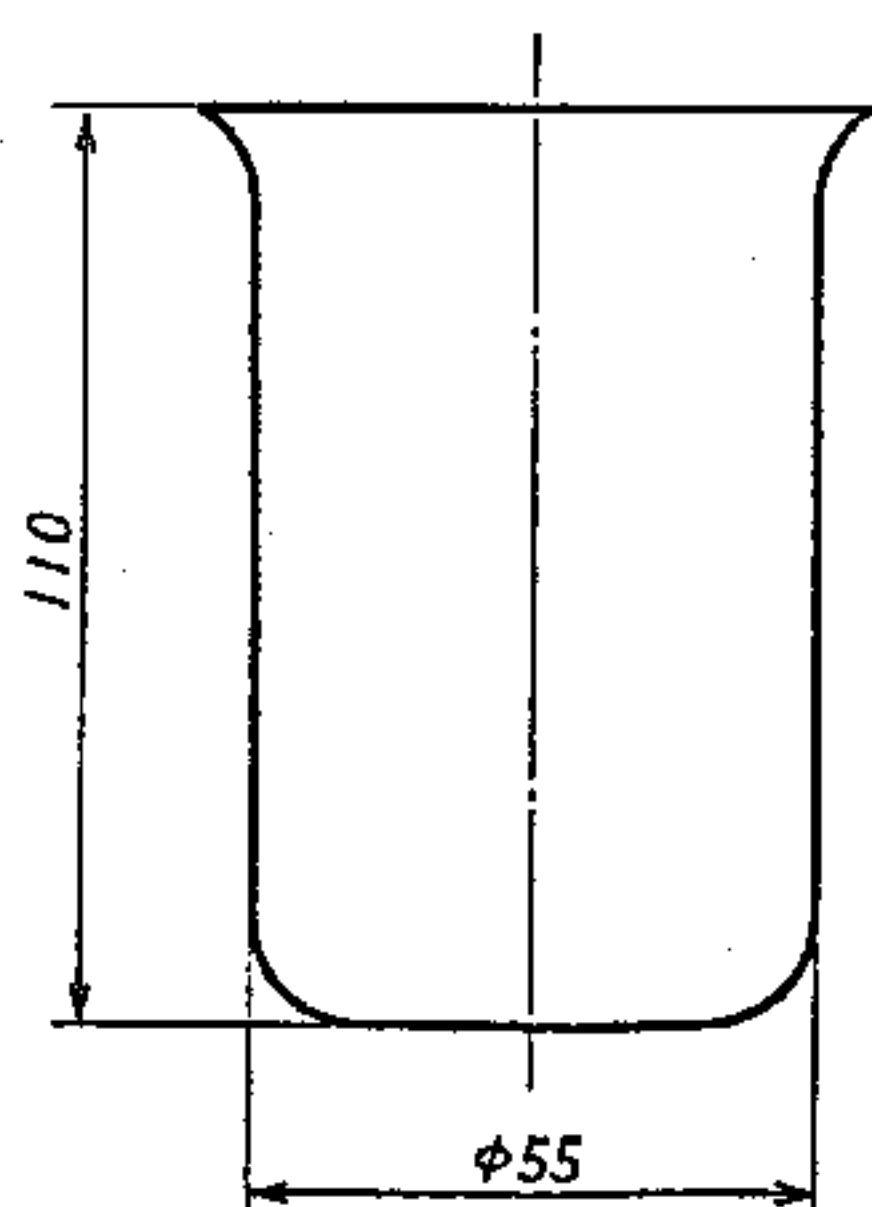
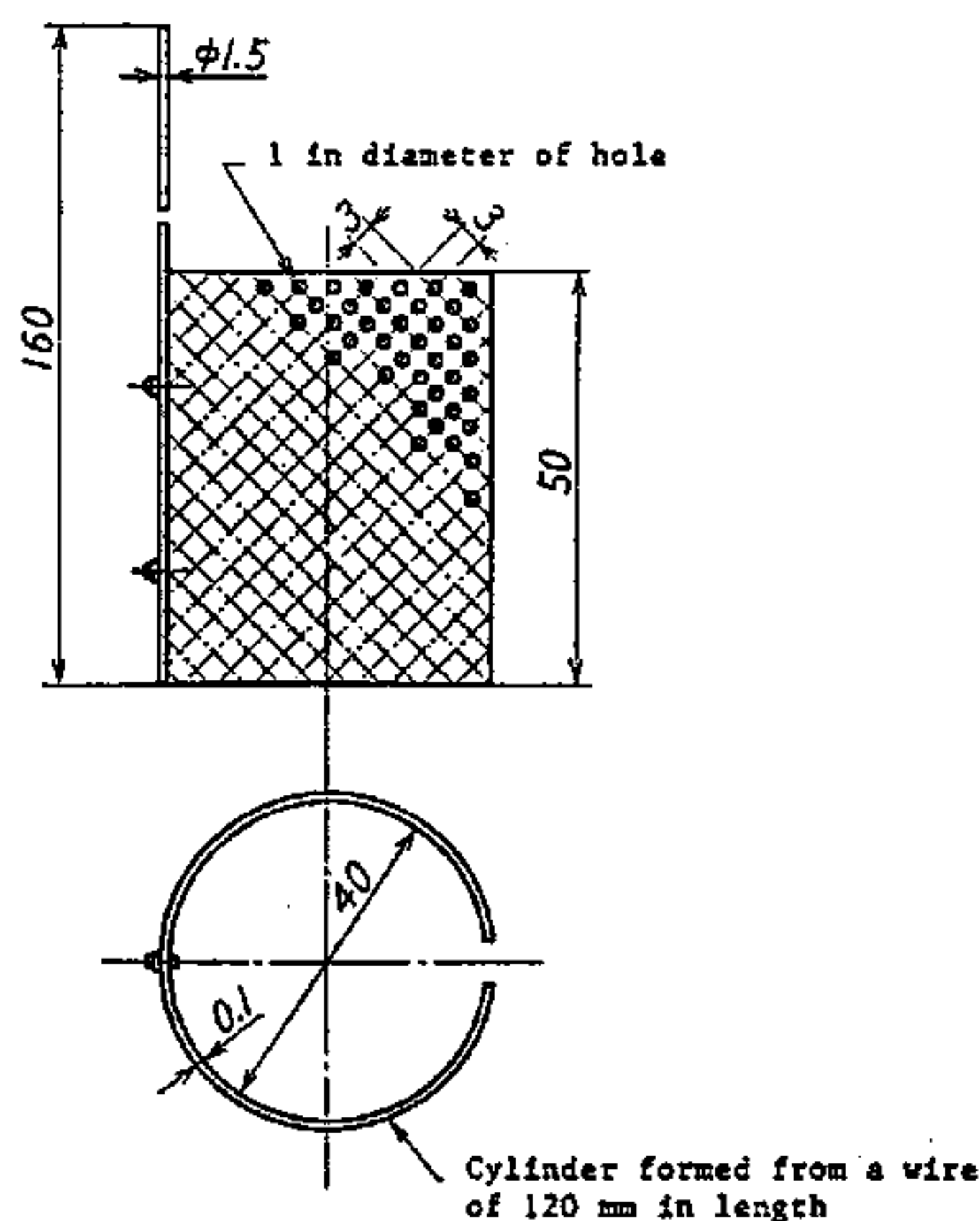


Fig. 2. Platinum Electrode A

Unit: mm



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Fig. 3. Platinum Electrode B

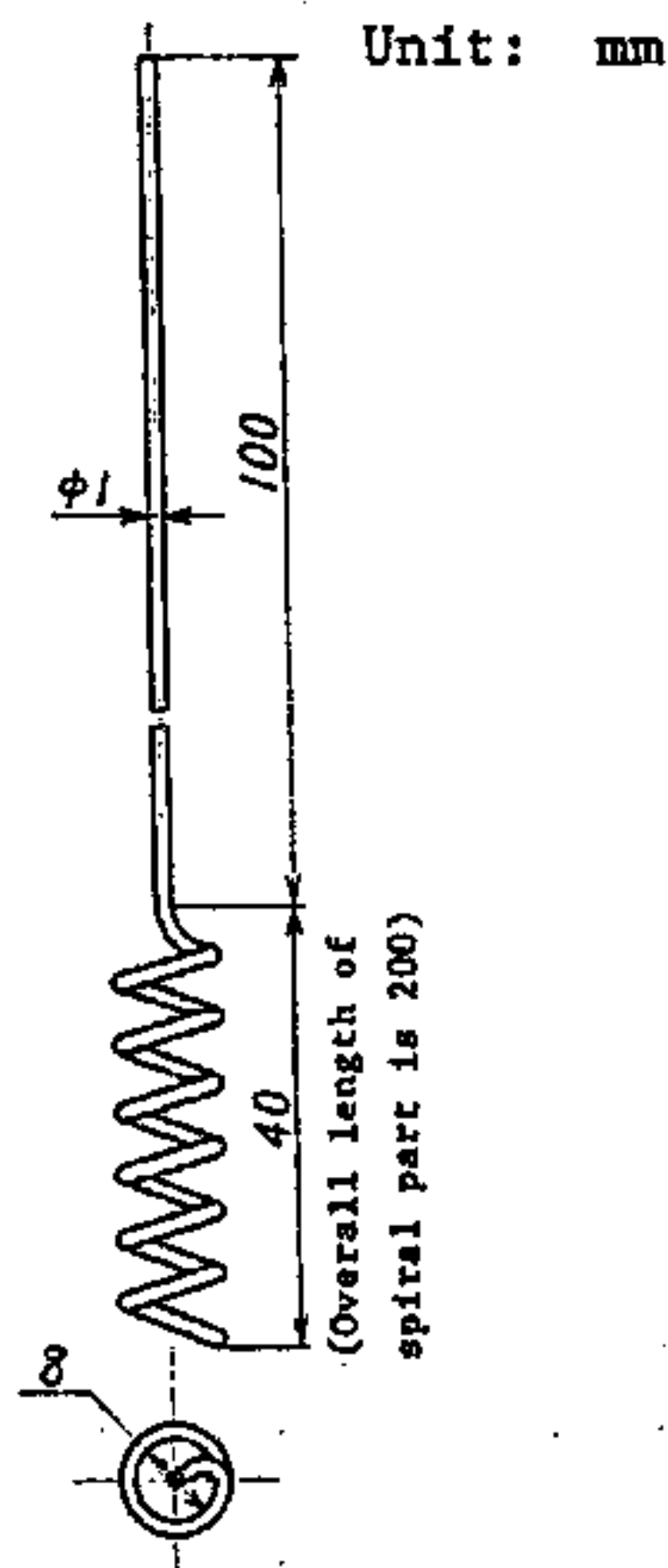
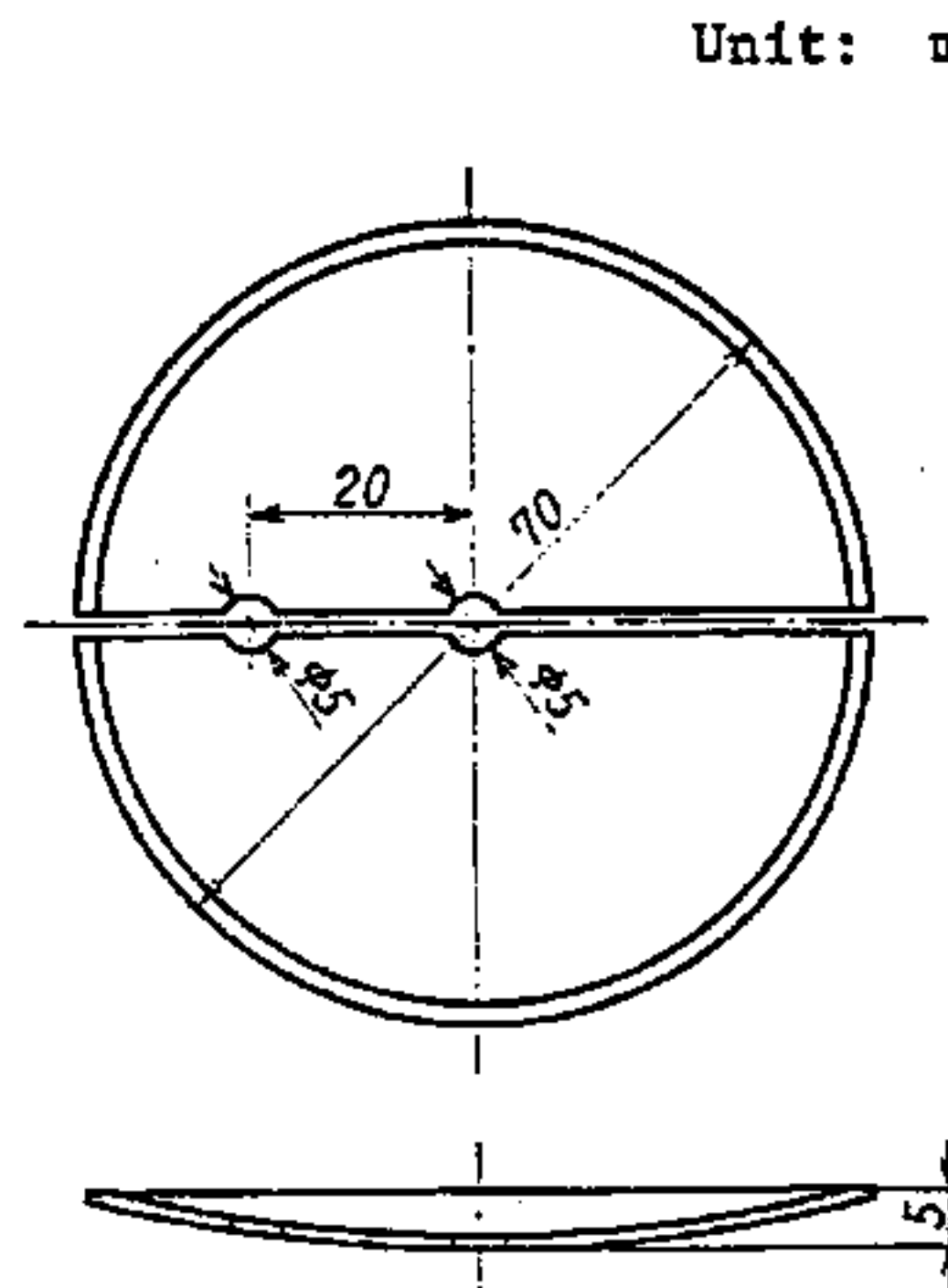


Fig. 4. Semicircular Watch Glass

6. Method for Determination of Nickel

6.1 Classification of Methods The method for determination of nickel shall be in accordance with either one of the following methods:

- (1) Titrimetric method
- (2) Gravimetric method

6.2 Titrimetric Method

6.2.1 Summary Decompose the sample with mixed acid, electrolyze it according to 4. and separate copper or the like. Add tartaric acid and ammonium chloride to the electrolytic residual liquid, make it alkaline with aqueous ammonia and deposit nickel by adding dimethylglyoxime. Thereafter, wash it with hydrochloric acid and dissolve the precipitate by heating. Add EDTA and EBT thereto and titrate it with zinc standard solution.

6.2.2 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid (1 + 1, 1 + 50)
- (2) Mixed acid It shall conform to 5.2.2 (1).
- (3) Aqueous ammonia
- (4) Aqueous ammonia (1 + 1)