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नेपाल गुणस्तर
NEPAL STANDARD

METHODS OF TEST FOR CABLES

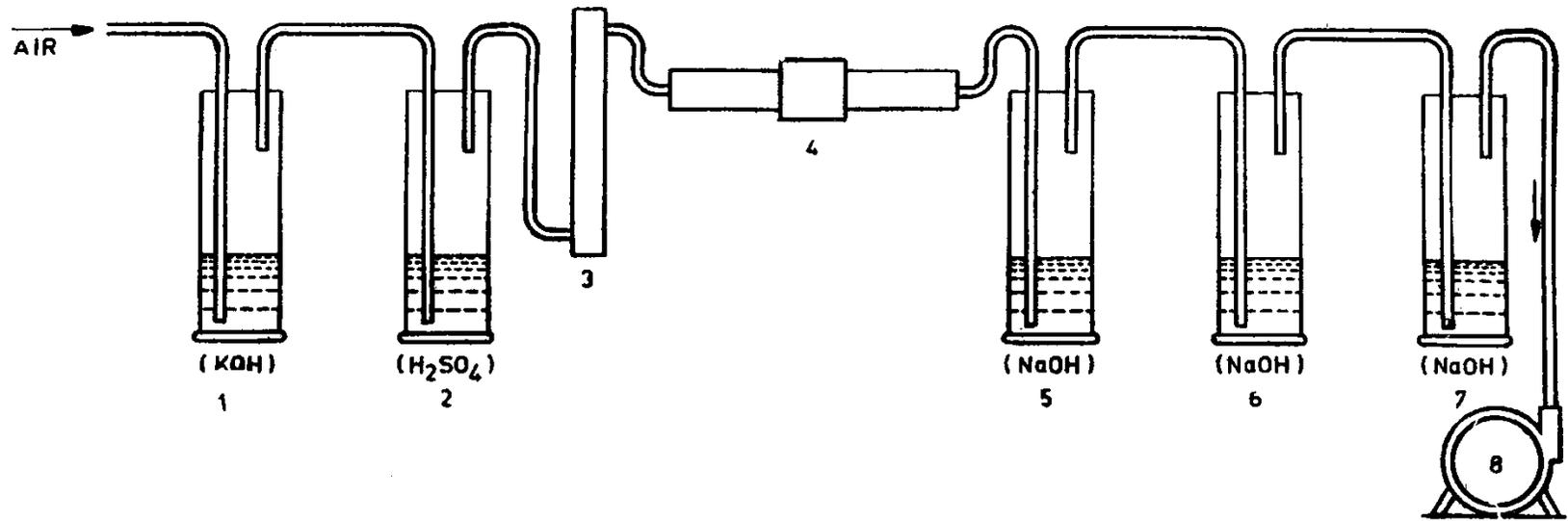
**PART 8 DETERMINATION OF THE AMOUNT OF
HALOGEN ACID GAS EVOLVED DURING COMBUSTION
OF POLYMERIC MATERIALS TAKEN FROM CABLES**

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METHOD OF TEST FOR CABLES

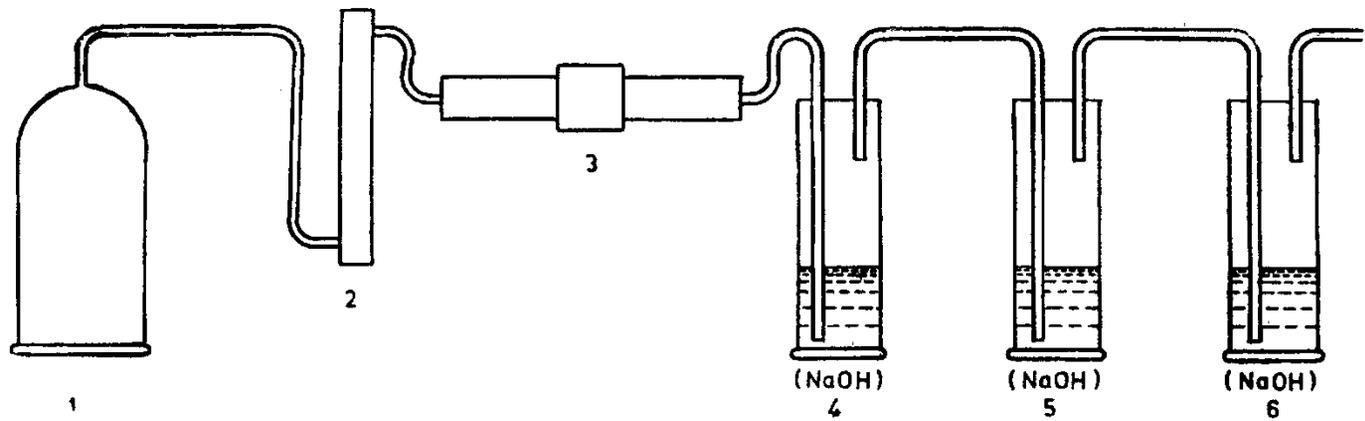
PART 8 DETERMINATION OF THE AMOUNT OF HALOGEN ACID GAS EVOLVED DURING COMBUSTION OF POLYMERIC MATERIALS TAKEN FROM CABLES

- 1. Scope:** This standard describes method for determination of the amount of halogen acid gas other than hydrofluoric acid evolved during the combustion of compounds based on halogenated polymers and compounds containing halogenated additives taken from cable insulation and outer sheath.
- 2. Significance:** For certain locations, the amount of halogen acid gas evolved during burning of cables is very critical since this signifies the extent of corrosion that a compound is capable of causing in an environment.
This test is, therefore, carried out on polymeric material components used in cable construction, to determine the amount of halogen acid gas evolved during combustion.
This test method is not recommended for use where the amount of halogen acid evolved is less than 5mg/g of specimen taken.
- 3. Principle of method:** Cable compound is heated up to maximum of 800° C and is decomposed completely. Halogen acid gas evolved during this is suitably collected and calculated in milligrams of hydrochloric acid per gram of sample taken.
- 4. Apparatus:**
 - 4.1.** Tube Furnace: With thermoplastic temperature control up to 1000° C. The length of the tube furnace shall be at least 100 mm.
 - 4.2.** Quartz (or Other Suitable) Combustion Tube- Approximately 19 mm inside diameter, 25mm outside diameter and 700 mm long,
 - 4.3.** Porcelain (or Other Suitable) Combustion Boat- Approximately 76 X 10 X 9 mm.
 - 4.4.** Three Wash Bottles- Diameter 55 ±5 mm.
 - 4.5.** Glass Tubing and Silicone Rubber Stoppers- Used to connect the wash bottles to the combustion tube. Connection between the wash bottles shall be made using silicone rubber with the glass tubing butting together in the connection or by using ground glass joints.
 - 4.6.** Physical Balance- Accuracy 0.1 mg
 - 4.7.** Air Flow Meter- 0 to 200 ml/min. For set of apparatus (see Fig.1 or 2).
- 5. Materials**
 - 5.1.** Sodium Hydroxide Solution- N/10
 - 5.2.** Nitric Acid Solution- 6N
 - 5.3.** Silver Nitrate Solution- N/10
 - 5.4.** Ammonium Thiocyanate Solution- N/10



- 1,2. Wash Bottles
 3. Air Flow Meter
 4. Combustion Furnace
 5,6,7. Wash Bottles with Sintered Glass Filters
 8. Vacuum Pump

FIG. 1 TEST SET UP



1. Dry Air
 2. Air Flow Meter
 3. Combustion Furnace
 4,5,6. Wash Bottles with Sintered Glass Filters

FIG. 2 ALTERNATE ARRANGEMENT FOR TEST SET UP

5.5. Ferric Ammonium Sulphate Solution-40 percent

5.6. Dry Air

5.7. Potassium hydroxide- 0.1 N

5.8. Sulphuric Acid – 0.1 N

6. Test Specimen- Piece of sheath of cable cut into suitable size or powdered, 0.5 to 1 g of test specimen shall be taken.

7. Conditioning- No preconditioning is required for this test.

8. Procedure

8.1. Test specimen shall be weighted to an accuracy of ± 0.1 mg in the combustion boat, which shall then be inserted into the combustion tube in the tube furnace, In order to reduce condensation in the tube, the exit end of the combustion tube shall not project more than 60 mm from the end of the furnace.

8.2. The combustion tube shall be connected to the three wash bottles each containing 100 ml of N/10 (0.1 N) sodium hydroxide. The second and third bottle shall be fitted with sintered glass diffuser and dry air shall be passed through the apparatus at a rate of 100 ± 5 ml/min.

8.3. The temperature of the tube furnace shall then be raised to $800 \pm 10^\circ \text{C}$ at a rate of approximately $20^\circ \text{C}/\text{min}$ and maintained at $800 \pm 10^\circ \text{C}$ for 20 minutes.

8.4. The three wash bottles shall then be disconnected and when cool, the combustion tube and connecting tubes shall be washed with distilled water and the total volume shall be made up to 500 ml with distilled water. 100 ml of the diluted solution shall then be measured into flask and 2ml of concentrated nitric acid, 20 ml of 0.1 N silver nitrate and 3 ml of 40 percent aqueous solution of ferric ammonium sulphate containing a few drops of 6 N nitric acid added and mixed together.

8.4.1. After filtration through on sintered glass crucible to remove precipitated silver chloride, the solution shall then be titrated with 0.1 N (N/10) ammonium thiocyanate solution.

Note1- Using this method, halogen acids evolved will be expressed as hydrochloric acid.

Note2- For a rapid control test, the tube may be preheated to 800°C , the air flow rate adjusted and the combustion boat containing the sample advanced slowly into the combustion zone to guard against any detrimental reaction. The results of such a test are for guidance purpose only and do not constitute compliance with this standard.

9. Method to Determine Factor

9.1. Preparation of Ammonium Thiocyanate Solution (0.1 N) - Weigh about 9 g of AR ammonium thiocyanate and dissolve it in 1 litre of distilled water in volumetric flask. Shake well. Standardization of this solution is carried out with 0.1 N AgNO_3 .

9.2. Preparation of AR Silver Nitrate (0.1 N)- Dry some finely powdered AR AgNO_3 at 150°C for 2 hours and allow it to cool in a desiccators. Weigh out accurately 8.496g. Dissolve it in water and make up the volume to 500 ml in a volumetric flask. This gives 0.1000 N AgNO_3 solutions. The weight of AgNO_3 will vary depending upon the purity of AgNO_3 used for

example, if this is 99.9 percent pure, then the weight should be multiplied by the purity factor of AR AgNO₃ in this concentration:

$$8.496 \times \frac{1}{0.999} = 8.5045 \text{ g}$$

9.3. Procedure- Take the above solution of NH₄CNS in a burette (50ml). Pipette out 25 ml of 0.1 N AgNO₃ in a conical flask. Add 5 ml of 6 N HNO₃ and 2ml of the ferric ammonium sulphate (ferric alum indicator) is prepared by making 40 percent aqueous solution containing few drops of 6N nitric acid. Run in NH₄CNS solution from the burette. At first a while precipitate is produced rendering the liquid milky appearance, as each drop of thiocyanate falls in it, it produces a reddish brown cloud which quickly disappears on shaking. As the end point approaches, the precipitate becomes flocculent and settles easily. Finally one drop of the thiocyanate solution produces a faint brown colour, which no longer disappears upon shaking. This is the end point. Note it as X ml.

Now, calculate the normality of ammonium thiocyanate as follows:

$$\begin{aligned} \text{NH}_4\text{CNS} &= \text{AgNO}_3 \\ N_1 V_1 &= N_2 V_2 \\ N_1 \times (X) &= 0.1 \times 25 \\ N_1 &= \frac{0.1 \times 25}{X} \end{aligned}$$

Where,

N₁= normality of NH₄CNS

V₁= Volume of NH₃CNS used for this titration= X

N₂= normality of AgNO₃

V₂= volume of AgNO₃ (25ml) taken for standardization.

This (N₁) is to be treated as factor for NH₄CNS

$$N_1 = \text{Factor} = F$$

10. Tabulation of Observations

Mass of Specimen	Volume of Ammonium Thiocyanate Solution A	Volume of Ammonium Thiocyanate Solution Used in Bank Test B	Factor for Ammonium Thiocyanate Solution F

11. Calculation – the amount of halogen acid expressed as milligrams of hydrochloric acid per gram of sample taken is calculated using the following formula:

$$\frac{36.5 \times (B - A) \times F \times 500}{100 \times m}$$

Where,

B= Volume of 0.1N ammonium thiocyanate solution used in the blank test,

A= Volume of 0.1 N ammonium thiocyanate solution used in the determination.

F= factor for 0.1 N ammonium thiocyanate solution, and

M= mass of the sample taken in grams.

12. Report

12.1. Reference Specification

12.2. Results

Specimen Number	Amount of Hydrochloric Acid, mg/g	
	Observed	Specified

12.3. Conclusion – Specimen meets/ does not meet the requirements of the specification.

Reference:

This Standards has been formulated based on **IS 10810 (Part 59) :1988 (Reaffirmed 2020) Methods of Test for Cables part 59 Determination Of The Amount Of Halogen Acid Gas Evolved During Combustion Of Polymeric Materials Taken From Cables.**