ICS 83.080.20
CCS G31

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 7139-2023

Plastics - Vinyl chloride homopolymers and copolymers - Determination of chlorine content

塑料 氯乙烯均聚物和共聚物 氯含量的测定

Issued on: August 6, 2023 **Implemented on: March 1, 2024**

Issued by: State Administration for Market Regulation;
Standardization Administration of the PRC

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Foreword

This document was issued on 6 August 2023 by the State Administration for Market Regulation; Standardization Administration of the PRC and takes effect on 1 March 2024.

It is a GB/T standard: recommended rather than compulsory, but it is the text a Chinese reviewer applies when assessing a submission.

1 Scope

GB/T 7139-2023 measures how much chlorine a vinyl chloride homopolymer or copolymer holds, which is the routine way of establishing what a PVC resin actually is: the chlorine figure separates homopolymer from copolymer, shows how much comonomer went into the chain. Two procedures are given. The first burns a weighed specimen in a combustion bomb charged with sodium peroxide together with starch or sucrose, so that organically bound chlorine ends up as chloride in solution; the second oxidises the specimen with oxygen in a closed vessel and titrates the resulting chloride potentiometrically. The document also fixes what the test report has to state, while informative annexes map its structure against ISO 1158:1998, which it modifies, and describe how the bomb and its charge are filled. A warning at the head of the method notes that the operator needs practical laboratory experience, and it is warranted: both procedures ignite a halogenated polymer inside a sealed vessel with an oxidiser. Chlorine content governs processing behaviour, thermal stability and the additive package a compounder has to build around the resin, so a resin accepted on a supplier's data sheet alone can be wrong in a way that first appears on the extruder. Resin producers, compounders, converters and testing

laboratories use it.

This document describes methods for the determination of the chlorine content of homopolymers and copolymers of vinyl chloride.

This document is applicable to the determination of chlorine content in vinyl chloride homopolymers and copolymers without plasticizers and additives. It can be used as a reference for the determination of chlorine content in other chlorine-containing polymers without plasticizers and additives.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 601, Chemical reagent -- Preparations of reference titration solutions

GB/T 603, Chemical reagent -- Preparations of reagent solutions for use in test methods

GB/T 6682, Water for analytical laboratory use -- Specification and test methods

GB/T 9725-2007, Chemical reagent -- General rule for potentiometric titration

3 Terms and definitions

There are no terms or definitions that require definition in this document.

of the bomb body. Then remove the empty incendiary bomb. Put in the specimen

incendiary bomb. Heat the specimen incendiary bomb to 300 degrees C~400 degrees C for about 10

min. Combustion usually starts at 50 degrees C~60 degrees C, with a crackling sound, and the bottom

of the bomb body begins to emit a glowing light.

If you use an electric ignition incendiary bomb, operate according to the equipment instruction manual.

4.4.4 Cool the bomb. If the bomb is cooled in water, be careful not to allow water to come into contact with the joint between the plug and the bomb. When there is doubt

whether a reaction has occurred, the bomb material shall not be dissolved in water as this may reason an explosion. It is advisable to spread the bomb contents flat on dry sand and spray with water from a safe distance and then rinse with plenty of water.

After the bomb has cooled, if a gas-ignited bomb is used, open the bomb. Remove the nickel crucible and carefully place it in a beaker containing 100 mL of distilled water. Immediately cover the beaker with a watch glass. When the reaction stops, rinse the inside of the bomb and the stopper. Place the wash in the beaker.

After the bomb body cools down, if an electric ignition bomb is used, remove it after cooling. Open the head and pour the material into a beaker filled with 100 mL of distilled water. Place the melting cup and the head horizontally in the same beaker and immediately cover with a watch glass.

4.4.5 Heat the beaker until the contents are boiling. Cool. Remove the nickel crucible and lid or the crucible and head. Rinse with water and collect the washings in the beaker.

4.4.6 Slowly add 15 mL of concentrated nitric acid (4.2.2). Then, add nitric acid solution (4.2.3) dropwise while stirring until the mixture is neutral. Then, add 2 mL of nitric acid solution (4.2.3) in excess. Dilute the contents of the beaker with water to about 200 mL.

NOTE: Methyl orange is a suitable indicator for neutralization.

4.4.7 Without adding the specimen, a blank combustion test is carried out using exactly the same test steps, reagents and amounts as those for the specimen.

4.4.8 The chlorine content is determined by potentiometric titration. The titration endpoint is automatically given by the instrument or determined according to the graphical method in 6.2 of GB/T 9725-2007 or the second-order derivative method.

4.5 Test data processing

The chlorine (Cl) content in the sample is expressed as mass fraction w1 and is calculated according to formula (1):

Where,

c1 - The exact value of the concentration of the standard silver nitrate titration solution, in moles per liter (mol/L);

M - The molar mass of chlorine, in grams per mole (g/mol) (M = 35.453);

V1 - The volume of the standard silver nitrate titration solution used to measure the specimen, in milliliters (mL);

V2 - The volume of the standard silver nitrate titration solution used to measure the blank combustion test, in milliliters (mL);

m1 - The mass of the specimen, in grams (g).

The calculation result is rounded to one decimal place.

4.6 Allowable difference

The absolute value of the difference between two measured values shall not exceed 0.2%. The arithmetic mean of the two parallel measurement results shall be reported as the result.

5 Method B - Molotov cocktail method

5.1 Principle

The specimen is oxidized with oxygen. The resulting chloride is then titrated potentiometrically.

5.2 Reagents or materials

Unless otherwise specified, analytically pure reagents and grade 3 water in GB/T 6682 or water of equivalent purity shall be used in the analysis. The required standard titration solutions, preparations and products shall be prepared in accordance with the provisions of GB/T 601 and GB/T 603.

5.2.1 Sodium nitrate.

5.2.2 Hydrogen peroxide solution: 300 g/L.

5.2.3 Potassium hydroxide solution: 100 g/L. Weigh 100 g potassium hydroxide and add it to a beaker containing 300 mL of water in 3~5 times. Cool to room temperature under cold water while stirring. Transfer to a 1000 mL volumetric flask. Dilute to the mark.

5.2.4 Nitric acid solution: 125 g/L. Take 135 mL of concentrated nitric acid and dilute

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