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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 13255.7-2009

Replacing GB/T 13255.7-1991

**Caprolactam for industrial use test methods -- Part 7:
Determination of iron content
(English Translation)**

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Foreword

GB/T 13255 "Industrial caprolactam test method" is divided into eight sections.

- Part 1. Determination of a 50% aqueous solution of chromaticity spectrophotometry;
- Part 2. Determination of crystallizing point;
- Part 3. Determination of permanganate absorption spectrophotometry value;
- Part 4. After distillation of volatile alkali content of titration;
- Part 5. 290nm wavelength measured absorbance;
- Part 6. Determination of acidity or alkalinity;
- Part 7. Determination of iron content;
- Part 8. Determination of cyclohexanone oxime content.

This section GB/T 13255 Part 7.

The partial modification of a Russian national standards GOCT26743.4-1985 "Determination of caprolactam iron content." According to this section GOCT26743.4-1985 redrafted. The standard lists the reg number in Appendix A and GOCT26743.4-1985 reg control list numbered.

Taking into account China's national conditions, in this section when using GOCT26743.4-1985 made some editorial changes, this portion the main technical difference GOCT26743.4-1985 follows.

--- Different color than the time requirements, GOCT26743.4-1985 color is added immediately after the colorimetric agent, was added to this section is Within 15min after

toner colorimetric;

--- Results of different expressions, GOCT26743.4-1985 percentage by mass expressed by this section in mg/kg.

This Part replaces GB/T 13255.7-1991 "Determination of caprolactam industry iron content." this section compared with the GB/T 13255.7-1991 The main changes are as follows.

--- Increasing the level of balance requirements (see 5.3);

--- Will analyze the water level defined as "third grade water GB/T 6682-2008 specified in" (1991 edition, Chapter 4, edition Chapters 4);

--- The microburette to different specifications of the Class A Graduated pipettes (1991 Edition Chapter 5, Chapter 5 of this edition);

--- The results of iron content representation from "the mass fraction (10-6)" to "mg/kg" (1991 edition, Chapter 7, the first edition Chapter 7);

--- The "poor results of two parallel determination of not more than 0.1×10^{-6} " to "difference between two parallel determination results is not more than 0.1mg/kg" (1991 edition, Chapter 7, Chapter 7 of this edition);

--- Added "Report" (see Chapter 8).

Appendix A of this section is an informative annex.

This part is proposed by the China Petroleum and Chemical Industry Association.

This part of the National Standardization Technical Committee on Organic Chemistry Club (SAC/TC63/SC2) centralized.

This section is drafted. DSM Nanjing Chemical Co., Ltd.

1 Scope

GB/T 13255 provisions of this part of the caprolactam for industrial use Determination of iron content in Thiocyanate spectrophotometric.

2 Normative references

The following documents contain provisions which, through reference GB/T 13255 in this section constitute provisions of this section. For dated reference documents Member, all subsequent amendments (not including errata content) or revisions do not apply to this section, however, encouraged to reach under this section Parties to research agreement to use the latest versions of these documents. For undated reference documents, the latest versions apply to this section.

GB/T 602-2002 Chemical Reagent preparation impurity standard solution was determined by (ISO 6353-1. 1982, NEQ) GB/T 6682-2008 analytical laboratory use specifications and test methods (ISO 3696. 1987, MOD) GB/T 8170 repair numerical representation about rules and limit values and judgment

3 PRINCIPLE OF THE METHOD

Hydrogen peroxide in the sample solution divalent iron ions are oxidized to trivalent. In acidic solution, ferric ion and thiocyanate halophytes into the orange-red complexes, the maximum absorption wavelength at 500nm, absorbance was measured using a spectrophotometer.

4 Reagents

Unless otherwise stated, use only in the analysis confirmed analytically pure reagents and third grade water GB/T 6682-1992 requirements.

4.1

hydrochloric acid.

Hydrogen peroxide solution is 4.2. 1 + 9.

4.3

ammonium thiocyanate solution. 100g/L.

4.4

iron (Fe) standard solution. 100mg/L, prepared according to the provisions of GB/T 602-2002 in.

4.5

iron (Fe) standard solution. 10mg/L, the iron standard solution (4.4) accurately diluted 10-fold. Preparation used.

5 Instruments

5.1

Spectrophotometer. Accuracy $\pm 0.004A$. 5cm with absorption cell 2.

5.2

Graduated pipettes. A level, different specifications.

5.3

Balance. sensitivity, respectively 0.1g and 0.1mg.

6 Step, Analysis

Draw 6.1 Working curve with graduated pipettes are accurate pipetting 0.0mL, 1.0mL, 2.0mL, 3.0mL, 4.0mL, 5.0mL than six iron standard solution A 100mL flask, followed by adding 2mL of hydrochloric acid, ammonium thiocyanate solution 5mL diluted with water to the mark, mix, 15min intrinsic 500nm wavelength with 5cm absorption cell, with water as a reference, the absorbance is measured on a spectrophotometer.

With an iron content of abscissa and the corresponding absorbance for the vertical axis, drawing working curve.

6.2 Determination

Weigh about 50g laboratory samples, accurate to 0.1g, placed in 250mL beaker, was added 20mL of water to dissolve, then add