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

GB/T 13255.4-2009

Replacing GB/T 13255.4-1991

**Test methods of caprolactam for industrial use -- Part 4:
Determination of volatile bases content -- Titrimetric method
after distillation**

工业用己内酰胺试验方法 第4部分：挥发性碱含量的测定 蒸馏后滴定法
(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 Part of 413,255.

The partial modification of international standards ISO 8661. 1988 "Caprolactam for industrial use - Determination of volatile bases content - distillation drops Titration. " this section according to ISO 8661. 1988 redrafted. This section lists the reg number in Appendix A and ISO 8661. 1988 reg Control list numbered.

Taking into account China's national conditions, this part adopting ISO 8661. 1988 time made some editorial changes, this portion of the ISO 8661.

The main technical differences in 1988 is as follows.

- Analysis of water level defined by the "distilled water or equivalent purity" was changed to "GB/T 6682-2008 stipulated three water" (ISO 8661. 1988 version of Chapter 3; Chapter 4 of this section);
- Necked flask Nitrogen (Kelvin) instead of a ground stopper flask (ISO 8661. 1988 version

of Chapter 4; Chapter 5 of this section);

--- Analytical instruments increased the injector separating funnel (ISO 8661. 1988 version of Chapter 4; Chapter 5 of this section).

This Part replaces GB/T 13255.4-1991 "After distillation industry caprolactam volatile alkali content of the titration." this section compared with the GB/T 13255.4-1991 The main changes are as follows.

--- Will analyze the water level is defined by "less than 1.0 μ S/distilled water or equivalent purity cm of water" to "conform GB/T 6682-1992 stipulated three water "(Chapter 4 of this edition of the 1991 version of Chapter 4);

--- 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. Shijiazhuang Chemical Fiber Co., Ltd.

1 Scope

GB/T 13255 provisions of this section was determined by titration after distillation of volatile alkali content of caprolactam for industrial use.

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.

Preparation of GB/T 601-2002 Chemical Reagent Titration analysis standard solution GB/T 603-2002 Chemical Reagent Test Methods for preparing reagents and products used (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 \u200b\u200band judgment

3 PRINCIPLE OF THE METHOD

In an alkaline medium, the distillation of the volatile base, absorb with an excess of standard solution of hydrochloric acid, methyl red - methylene blue ethanol indicative Agents, with sodium hydroxide standard titration solution back titration.

4 Reagents

Unless otherwise stated, use only in the analysis confirmed analytically pure reagents and third grade water GB/T 6682-2008 requirements.

Analysis in the standard titration solution, preparations and products, did not indicate when the other requirements, according to GB/T 601-2002, GB/T 603-2002 Preparation of the provisions.

4.1

Sodium hydroxide solution. 160g/L.

4.2

sodium hydroxide standard titration solution. $c(\text{NaOH}) = 0.01\text{mol/L}$.

4.3

hydrochloric acid standard titration solution. $c(\text{HCl}) = 0.01\text{mol/L}$.

4.4

methyl red - methylene blue mixed indicator.

5 Instruments

5.1

Distillation Apparatus.

Distillation of the components of the instrument with rubber and rubber tubing connections, the instrument shown in FIG. 1, includes the following components.

5.1.1

Nitrogen-necked flask (Kelvin). 500mL.

5.1.2

separatory funnel. 100mL.

5.1.3

Straight condenser. outer tube length 400mm.

5.1.4

Nitrogen ball. diameter (70 ± 5) mm.

5.1.5

receiver. 250mL volumetric flasks.

5.2

EAF. with regulating transformer.

5.3

zeolite.