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TDS of N-Methyl-2-pyrrolidone（NMP）

CAS.NO.：872-50-4 UN.NO.:1760

1. Scope

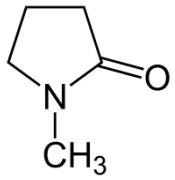
This standard specifies the requirements, test methods, inspection rules and markings, packaging, and transportation of N-Methyl-2-pyrrolidone (abbreviated code NMP)

transmission and storage.

This standard applies to N-Methyl-2-pyrrolidone (hereinafter referred to as the product).

Molecular formula: C_5H_9NO

Molecular structural formula:



2. Normative references

The following documents are essential for the application of this document. For dated references, only the dated version applies to this document. For undated references, the latest edition (including all amendments) applies to this document.

GB/T 325.1 Packaging containers - Steel drums - Part 1: General technical requirements

GB/T 3143 Liquid chemical product color determination method (Hazen unit - platinum-cobalt color number)

GB/T 4472 Density and relative density determination of chemical products

GB/T 6283 Determination of moisture content in chemical products Karl Fischer method (Coulomb method)

GB/T 6680 General rules for sampling of liquid chemical products

GB/T 6488 Liquid chemical products - Determination of refractive index

GB/T8170 Numerical Rounding Rule and Representation and Judgment of Limit

Values GB/T 9722 Chemical Reagents General Principles of Gas Chromatography

GB/T 601 Preparation of standard titration solutions of chemical reagents

GB/T9724-2007 General rules for the determination of pH value of chemical reagents (ISO6353-1:1982, NEQ)

GB/T9725-2007 Chemical reagents - General rules for potentiometric titration (ISO6353-1:1982, NEQ)

3. Electronic grade NMP product specifications:

Item	Spec.
Purity / %	≥99.8
Moisture / ppm	≤500
Hazen	≤20
pH【（1ml/10ml）aqueous】	6-9
Total Amine/ ppm	≤50
Density（20℃）/（g/cm^3）	1.026-1.033
Refractive index	1.467-1.471

4. Experiment method

4.1 Appearance

Take an appropriate amount of the sample in a clean glass container and observe it visually under natural light.

4.2 Purity

4.2.1 Method Summary

Using capillary column gas chromatography, under the selected chromatographic operating conditions, the sample is vaporized and separated by a chromatographic column.

Detector (FID) detection, using the area normalization method to quantify, andremove the moisture to obtain the content of N-Methyl-2-pyrrolidone.

4.2.2 Instrument

4.2.2.1 Gas chromatograph: equipped with a hydrogen flame ion detector (FID), the sensitivity and stability of the whole machine meet the requirements of GB/T9722, and the linear range meets the analysis requirements.

4.2.2.2 Data processing system: N2000 workstation or other chromatographic workstations that meet the requirements.

4.2.2.3 Injector: 10 μL microinjector.

4.2.2.4 Chromatographic analysis conditions

The chromatographic column and chromatographic operating conditions of this standard are shown in the table below. Other chromatographic columns and chromatographic conditions that can achieve the same degree of separation can also be used.

Column and chromatographic operating conditions

Capillary column	30m×0.53mm×2.65μm（Column length×column inner diameter×liquid film thickness）
Stationary Phase	(5% phenyl)-methyl polysiloxane
Pillar temperature/℃	178
Gasification chamber temperature/℃	240
Detector temperature/℃	260
Carrier gas（N_2）flow/（mL/min）	1.0
Hydrogen flow/（mL/min）	30
Air flow/（mL/min）	300
Split ratio	25：1
Injection volume/μL	0.4

4.2.3 Analysis results

Purity is calculated according to formula (1):

$$C_{sample}=C_{test}\times(100-C_{moisture})/100 \quad \text{.....(1)}$$

C_{sample}—Final true data (%) of N-Methyl-2-pyrrolidone content in the tested sample;

C_{test}—The content data of N-Methyl-2-pyrrolidone in the tested sample displayed by the chromatography workstation (%);

$C_{moisture}$—The percentage (%) of moisture content in the tested sample.

4.3 Moisture

According to the provisions of GB/T 6283.

The arithmetic mean of the results of the two parallel determinations is taken as the report result, and the absolute difference between the two parallel determination results is not more than 0.002%.

4.4 Hazen

According to the provisions of GB/T 3143.

4.5 Density

According to the provisions of GB/T 4472.

4.6 pH value

Use a pipette to transfer 10.0ml of the sample into a 100ml volumetric flask, add carbon dioxide-free water to dilute to the mark, shake well and transfer it into a beaker,

The following steps are determined in accordance with the provisions of Chapter 6 in GB/T9724-2007.

4.7 Total amines

4.7.1 Method Summary

The sample is dissolved in isopropanol, and the alkaline substance in the sample is titrated with standard hydrochloric acid, and the titration endpoint is indicated by a potentiometer or acidity meter.

The amount is based on monomethyl amine, and the total amine content is calculated.

4.7.2 Reagent

4.7.2.1 Isopropyl alcohol

4.7.2.2 Standard titration solution of hydrochloric acid: c（HCL）=0.02mol/L.

4.7.3 Instrument

4.7.3.1 Potentiometer: The accuracy of the potentiometer is ±0.01mV.

4.7.3.2 Acidity meter: The accuracy of the acidity meter is PH±0.01, and it has the function of temperature compensation.

4.7.4 Analysis steps

Weigh 65g of laboratory sample, accurate to 0.01g, put it in a 250ml beaker, add 100ml of isopropanol to dissolve, and shake well. The following steps are determined in accordance with the provisions in Chapter 6 of GB/T9725-2007.

4.7.5 result calculation

The total amine content is the mass fraction W2 of monomethyl amine (CH3NH2), the value is expressed in %, and it is calculated according to formula (3):

$$W2 = \frac{VcM}{m} x100 \quad \text{.....(3)}$$

V—the value of the volume of the standard hydrochloric acid titration solution (4.7.2.2) consumed by the sample, in milliliters (ml);

c—the exact value of the concentration of the standard titration solution of hydrochloric acid, in moles per liter (mol/L);

m—the value of the mass of the sample, in grams (g)

M—the value of the molar mass of monomethyl amine (CH3NH2), in grams per mole (g/mol) (M=31.06);

Take the arithmetic mean of the results of the two parallel determinations as the report result, and the absolute difference between the two parallel determination results is not more than 0.001%.

4.8 Free Amine

4.8.1 Experiment equipment

——Electronic balance, 0.0001g;

——Erlenmeyer flask, 250mL;

——Graduated cylinder, 100mL.

4.8.2 Reagent

——Hydrochloric acid standard solution:0.01mol/L

According to the provisions of GB/T 601.

——Anhydrous ethanol: content ≥ 99.7%.

——Bromocresol Green-Methyl Red Indicator:

Solution I: Accurately weigh 0.1 g of bromocresol green, dissolve in absolute ethanol, and dilute to 100 mL with absolute ethanol.

Solution II: Accurately weigh 0.2 g of methyl red, dissolve it in absolute ethanol, and dilute it to 100 mL with absolute ethanol.

Take 30 mL of solution I and 10 mL of solution II and mix them evenly.

4.8.3 Experimental procedure

4.8.3.1 Put the conical flask on the electronic balance, peel it, measure 50 mL of NMP with a graduated cylinder and pour it into the conical flask, accurately weigh the mass of NMP, and record it as m;

4.8.3.2 Add 50mL of absolute ethanol (or isopropanol) and 3-4 drops of bromocresol green-methyl red indicator to the conical flask in turn, shake it smoothly to make it uniform;

4.8.3.3 Titrate with 0.01mol/L hydrochloric acid standard solution from green to slightly yellow, and record the consumption volume V of hydrochloric acid standard solution;

4.8.3.4 At the same time, a blank test was performed, and the consumption volume V0 of the standard solution of hydrochloric acid was recorded.

4.8.4 Calculation

The free amine content is calculated according to formula (2):

$$\text{Free amine}\ (\text{ppm}) = \frac{C \times (V - V0) \times 1000 \times M}{m} \quad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\ (2)$$

C—concentration of hydrochloric acid standard solution, mol/L (the concentration of hydrochloric acid required to be prepared is 0.01mol/L);

V—the volume of the standard solution of hydrochloric acid consumed by the titration, mL;

V0—the volume of the standard solution of hydrochloric acid consumed by the blank experiment, mL;

The molar mass of M—monomethyl amine, 31.06g/mol;

m—the weight of the NMP sample weighed, g

4.9 Refractive index

Measure according to the method specified in GB/T6488, and the test temperature is 20℃.

5 Testing regulations

5.1 Batching and sampling

5.1.1 Batching：Take the product from the same finished storage tank every day as a batch.

5.1.2 Sampling: according to the provisions of GB/T 6680.

5.2 Factory inspection

5.2.1 Purity, moisture, chromaticity, density, free amine, PH, and refractive index are factory inspection items.

5.2.2 The product shall be inspected and qualified by the inspection department of our factory, and the quality inspection sheet shall be attached before leaving the factory.

5.3 Type test

5.3.1 Type inspection should be carried out in the following cases:

——When a new product is identified;

——When there is a major change in the formula process of the original product;

——When production is resumed after long-term shutdown (more than 1 year);

——When the national quality supervision and inspection agency proposes type inspection requirements.

5.3.2 Type inspection items are all items specified in this standard.

5.4 Judge rule

After inspection, if one item does not meet the standard requirements, it should be re-sampled for re-inspection. After the re-inspection, the item still does not meet the standard requirements, the batch is judged to be unqualified products, and unqualified products are not allowed to leave the factory.

6. Marking, packaging, transportation, storage

6.1 Marking

The product should have a paper label on the side of the packaging barrel, and the label should indicate the following: factory name, factory address, product name, net weight, batch number, date of manufacture, storage period, and standard number.

6.2 Packing

Industrial NMP should be packed in steel drums, PE drums, IBC drums or special tank trucks that meet the requirements of GB/T325.

6.3 Transportation

During the transportation of NMP, it should be ensured that the container does not leak, collapse, fall, or be damaged. When handling, it should be lightly loaded and unloaded to prevent the packaging container is damaged. During transportation, avoid sun and rain, and keep away from fire, heat sources and, high-temperature areas.

6.4 Storage

NMP storage location should be dry, ventilated, away from fire and other dangerous goods, and avoid direct sunlight. Under the specified storage conditions, the quality guarantee period is 1 year.