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CHEMICAL INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HG/T 3937-2007

1,6-Hexanediamine for industrial use

工业用1, 6己二胺

(English Translation)

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1 Scope

This standard specifies the requirements, test methods, inspection rules, marking, packaging, transportation, storage and safety of 1,6-Hexanediamine for industrial use.

This standard applies to the production, inspection and sale of 1,6-

Hexanediamine for industrial use which is produced by catalytic hydrogenation of adiponitrile which is used as raw material.

Molecular formula: C₆ H₁₆ N₂

Relative molecular mass: 116.21 (according to 2005 international relative atomic mass)

Structural formula: H₂ N(CH₂)₆ NH₂

2 Normative references

The provisions in following documents become the provisions of this standard through reference in this standard. For the dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this standard;

however, parties who reach an agreement based on this standard are encouraged to study if the latest versions of these documents are applicable.

For undated references, the latest edition of the referenced document applies.

GB 190-1990 Labels for packages of dangerous goods

GB/T 601-2002 Chemical reagent - Preparations of standard volumetric solutions

GB/T 603-2002 Chemical reagent - Preparations of reagent solution for use in test methods (neq ISO 6353-1:1982)

GB/T 1250 Methods for representation and determination of limit values

GB/T 3143-1982 Determination of the color of liquid chemistry product (Hazen unit - platinum-cobalt chroma) (eqv ISO 2211:1973)

GB/T 3723 Sampling of chemical products for industrial use - Safety in sampling (idt ISO 3165:1976)

5.2 General provisions

Unless otherwise stated, only the validated analytically pure reagents and the grade III water as specified in GB/T 6682-1992 are used in the analysis.

The standard titration solution used in the test method, the standard solution for the determination of impurities, the preparation and the product shall, when no other requirements are specified, be prepared according to the provisions of

GB/T 601-2002 and GB/T 603-2002.

5.3 Appearance in molten state

Take appropriate amount of laboratory samples. Melt it at 60 °C. Pour it into the colorimetric tube. Place the colorimetric tube in a 42 °C ~ 45 °C water bath.

Make visual observation immediately, to check whether it is a colorless transparent liquid.

5.4.1 Summary of method

Using methyl red as an indicator, use the hydrochloric acid standard titration solution for titration.

5.4.2.1 Hydrochloric acid standard titration solution

$c(\text{HCl}) = 0.5 \text{ mol/L}$.

5.4.2.2

Methyl red indicator solution 1 g/L.

5.4.3 Analytical procedures

Weigh 1.0 g ~ 14 g of laboratory sample, accurate to 0.0002 g. Place it in a 250 mL conical flask. Use 50 mL of water to dissolve it. Add 2 ~ 3 drops of methyl red indicator solution. Use the hydrochloric acid standard titration solution to titrate the solution, until the color of solution changes from yellow to red, which is used as the end point.

5.4.4 Calculation of results

The mass fraction w_1 of 1,6-Hexanediamine ($C_6H_{16}N_2$), the value of which is expressed in %, is calculated according to formula (1):

Where:

V - The volume of the hydrochloric acid standard titration solution (5.4.2.1), in milliliters (mL);

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

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

M - The molar mass of the 1,6-Hexanediamine ($1/2C_6H_{16}N_2$), in grams per mole (g/mol) ($M = 58.1$).

Take the arithmetic mean of the results of two parallel determinations as the determination result. The absolute difference between the results of the two parallel determinations is not more than 0.1%.

5.5 Determination of chroma of aqueous solution

It is performed according to the provisions of GB/T 3143-1982. Weigh (70 ± 0.1) g of laboratory sample which is in molten state. Dissolve it in water. Dilute it to

100 mL.

5.6 Determination of moisture

It is performed according to the provisions of GB/T 6283-1986. Add 5 mL of glacial acetic acid into the titration cell. Place the titration cell in an ice water bath.

Take the arithmetic mean of the results of two parallel determinations as the determination result. The absolute difference between the results of the two parallel determinations is not more than 15% of the arithmetic mean of the two determined values.

5.7 Determination of crystallization points

It is performed according to the provisions of GB/T 7533-1993, wherein it allows the main thermometer to use the locally-immersed thermometer.

Take the arithmetic mean of the results of two parallel determinations as taken as the determination result. The absolute difference between the results of the two parallel determinations is not more than $0.05\text{ }^{\circ}\text{C}$.

5.8.1 Summary of method

Use the polarographic instrument to measure the substances which produce the polarographic wave near -1.5 V in the sample's impurities. Use the

5.8.4.4 Determination of polarographic wave

Take 15 mL of 0

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standard solution ($i = 0, 1, 2, 3$) in turn. Add it into the electrolytic tank. Lead in nitrogen for 10 min to remove oxygen. Measure the polarographic wave $h(i, j)$ of it under the 6 sensitivities in the polarograph ($j =$

1, 2 ... 6).

5.8.4.5 Calculation of values

The polarographic value $PI(t)$ corresponding to the 1

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standard solution, expressed as $[mmol(\text{isobutyraldehyde}) / t(1,6\text{-Hexanediamine})]$, is calculated according to formula (2).

Where m - The mass of isobutyraldehyde which is added to solution A, in grams (g);

T_1 - The mass fraction of isobutyraldehyde (5.8.2.1), in percentage (%),

V_i - The volume of the solution A which is added to the 1

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standard solution, in milliliters (mL);

M - The molar mass of isobutyraldehyde, in grams per mole (g/mol) ($M = 72.107$);

T_2 - The concentration of 1,6-Hexanediamine in solution B, in percentage (%);

500 - The volume of the volumetric flask, in milliliters (mL).

The height difference of polarographic wave $\Delta h(i, j)$ corresponding to 1

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