GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 71.080.01
G 15

GB/T 7534-2004

Replacing GB/T 7534-1987

Volatile organic liquids for industrial use - Determination of boiling range

工业用挥发性有机液体 沸程的测定

(ISO 4626:1980, Volatile organic liquids - Determination of boiling range of organic solvents used as raw materials, MOD)

Issued on: March 15, 2004 **Implemented on: December 1, 2004**

Issued by: General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China;

Standardization Administration of the People's Republic of China.

Table of Contents

Foreword

This Standard uses ISO 4626:1980 *Volatile organic liquids - Determination of boiling range of organic solvents used as raw materials (EN)* after modification.

This Standard was redrafted in accordance with ISO 4626:1980. The contrast table for section numbers of this Standard and ISO 4626:1980 is listed in Annex A.

Taking China's national situation into consideration, this Standard has made some modifications when using ISO 4626:1980. The relevant technical differences have been compiled into the text, and have been identified by vertical single lines in the margins of the terms to which they relate. The list of the technical differences between this Standard and ISO 4626:1980 and corresponding reasons have been given in Annex B for reference.

This Standard replaces GB/T 7534-1987 *Volatile organic liquids for industrial use - Determination of boiling range*.

Compared with GB/T 7534-1987, the major changes in this Standard are as follows:

— MODIFY the boiling range applicable to this Standard from (50 ~ 200) °C to (30 ~ 300) °C (SEE Paragraph 1 of 1987 edition and Chapter 1 of this edition);

— ADD 200mL distillation flask (SEE Article 3.1 of 1987 edition and Article 5.1 of this edition);

— ADJUST the dimensions of the thermometer (SEE Article 3.2 of 1987 edition and Article 5.2 of this edition);

— ADD the distillation apparatus diagram for the electric heater in use (SEE Figure 3);

— ADD the safety and precautionary measures, factors causing superheating and operating precautions (SEE Chapters 6 and 9);

— ADJUST the requirements for the temperature of cooling water (SEE Chapter 4 of 1987 edition and Article 7.4 of this edition);

— ADD the requirements for the temperature of test pieces (SEE Article 7.4);

— MODIFY the total recovery volume from not less than 98mL to not less than 97% (volume fraction) for non-viscous liquid and not less than 95% (volume fraction) for viscous liquid (SEE Chapter 5 of 1987 edition and

Article 8.6 of this edition);

— ADD the compound category to the table for the corrected values of the boiling points with pressure (SEE Annex C);

— The precision of method is given (SEE Article 10.4).

The Annexes A, B and C in this Standard are informative annexes.

This Standard was proposed by China Petroleum and Chemical Industry Association (changed to “China Petroleum and Chemical Industry Federation” on May 10, 2010).

This Standard shall be under the jurisdiction of the Subcommittee of Organic Chemical Industry of the National Technical Committee for Standardization of Chemistry (CSBTS/TC 63/SC 2).

Drafting organization of this Standard: SINOPEC Beijing Research Institute of Chemical Industry.

Main drafters of this Standard: Guo Yanling and Dong Wei.

This Standard was first released in March 1987.

Volatile organic liquids for industrial use - Determination of boiling range

Warning - This Standard does not address all safety issues related to the use of this Standard. It is the user's responsibility to take appropriate safety and health measures and to ensure compliance with the relevant national regulations. The description of relevant safety and precautionary measures is provided in Chapter 6.

1 Scope

This Standard specifies the method for determining the boiling range of the volatile organic liquids.

This Standard **is applicable to** the organic liquids that boil between 30°C and 300°C at normal pressure, and that are chemically stable during the distillation (such as hydrocarbons, esters, alcohols, ketones, ethers and similar organic compounds).

2 Normative references

The provisions in the following documents become the provisions of this Standard through reference in this Standard. For 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 editions of these documents are applicable. For undated references, the latest editions apply to this Standard.

GB/T 3723 *Sampling of chemical products for industrial use - Safety in sampling (GB/T 3723-1999, idt ISO 3165:1976)*

3 Terms and definitions

The following terms and definitions are applicable to this Standard.

3.1 Initial boiling point

It refers to the temperature observed (corrected if necessary) at the moment when the first drop of condensate falls from the tip of the condenser tube during a distillation carried out under standardized conditions.

3.2 Dry point

It refers to the temperature observed (corrected if necessary) at the moment of vaporization of the last drop of liquid at the bottom of the distillation flask during a distillation carried out under standardized conditions, disregarding any liquid on the side of the distillation flask and on the thermometer.

3.3 Boiling range

It refers to the temperature interval between the initial boiling point and the dry point.

3.4 End point, final boiling point

It refers to the maximum temperature observed (corrected if necessary) during the final phase of a distillation carried out under standardized conditions.

4 Method summary

DISTILL 100mL of test piece under prescribed conditions. Systematically OBSERVE the thermometer readings and volumes of condensate. READ the initial boiling point and the dry point from the thermometer. OBTAIN the boiling range of the test piece being tested by the calculation of the observed data. CORRECT the results to standardized conditions.

5 Apparatus

5.1 Distillation flask

It is made of heat-resistant glass, with a capacity of 100mL or 200mL, conforming to the dimensions shown in Figure 1.

Note: Superheating of liquid in a new flask may be prevented by depositing a small amount of carbon at the bottom of the flask, which may be accomplished by heating and decomposing a pinch of tartaric acid at the bottom of the flask. The flask is then prepared for use by washing with water, rinsing with acetone, and drying (An exception is made for diacetone alcohol: in order to avoid an erratic value for the initial boiling point, the distillation flask shall be clean and free of any residual carbon decomposition product).

5.2 Thermometer

Mercury-in-glass type, nitrogen-filled, enamel-backed, and complying with the requirements in Table 1. If using a full-immersion thermometer, the division value shall be 0.1°C or 0.2°C, and an auxiliary thermometer shall be used to correct the mercury column of the part above the plug of the master thermometer during distillation. The auxiliary thermometer is generally of

6.2 Certain solvents and chemical intermediates, particularly ethers and unsaturated compounds, may form peroxides during storage. These peroxides may present an explosion hazard when these chemicals are distilled, especially as the dry point is approached. Peroxides shall be pretested, or appropriate precautions shall be taken, if necessary.

Note: Test for peroxides: ADD (0.5 ~ 1.0) mL of the test piece to be tested to an equal volume of glacial acid to which has been added about 100mg of sodium or potassium iodide crystals. A comparatively yellow color indicates a low and a brown color a high concentration of peroxide in the test piece.

6.3 Most organic solvents and chemical intermediates are flammable. A fire hazard might exist when a distillation is carried out, and safety precautions shall be taken. For instance, the flask shall be examined for cracks; the apparatus shall be examined for tightness and shield. Adequate ventilation shall be provided to maintain the solvent vapor concentration below the explosive limit in the immediate vicinity of the distillation apparatus, and below the threshold limit value in the general work area.

7 Apparatus assembly and test preparation

7.1 The apparatus shall be assembled as shown in Figure 2 or Figure 3. The parts and junctions shall be respectively examined for integrity and tightness. USE a piece of soft and lint-free cloth to clean and dry the condenser tube.

7.2 SELECT the thermometer or USE the thermometer required in the standard for the products to be tested by reference to Table C.1 in Annex C. The scales of the selected thermometer shall include the entire boiling range of this product. USE an appropriate rubber or wooden plug to fix the thermometer in the neck of the flask so that the upper end of the thermometer's contraction chamber is level with the lower side of the junction of the distillation flask's neck and the flask branch. If using a full-immersion thermometer, an auxiliary thermometer shall be attached to the master thermometer so that the mercury bulb is located at half the height of the mercury column at which the master thermometer is beyond the plug at the boiling point.

7.3 FIX the distillation flask in the position next to the hole ring of the heat-resistant separator. USE a plug to tightly connect its branch with the upper end of the condenser tube. ENSURE that the branch inserted into the condenser tube is about 25mm long and on the same centerline.

7.4 FILL the condenser with sufficient amount of water at appropriate temperature, so as to ensure that the temperature of cooling water complies with the requirements in Table 2 at the start of and during distillation.

temperatures at different distillate volumes. RECORD the temperature at the instant when the last drop of liquid at the bottom of the distillation flask gasifies (corrected to standardized conditions) as dry point. STOP heating immediately.

When a dry point cannot be obtained (when the test piece decomposes before reaching the dry point, that is to say, there is vapor or thick smoke escaping rapidly, or the maximum temperature has been observed on the thermometer while there is still liquid left on the bottom of the flask), RECORD this phenomenon.

8.4 When a dry point cannot be obtained, REPORT the maximum temperature observed on the thermometer as the end point. When active decomposition is encountered, the rapid evolution of vapor and heavy fumes is usually followed by a gradual decrease in the distillation temperature. RECORD the temperature and REPORT the decomposition point. If the expected drop in temperature does not occur, RECORD the maximum temperature observed 5min after the 95% (volume fraction) distillate point has been reached. REPORT as “end point, 5min”. This notation shows that a true end point cannot be reached within the given time limit. The end point shall not exceed 5min after the 95% (volume fraction) distillate point.

8.5 READ and RECORD the barometric pressure to the nearest 0.1kPa. In the meantime, RECORD the room temperature.

8.6 The total recovery rate of the distillate obtained from non-viscous liquid with a boiling range of less than 10°C shall not be less than 97% (volume fraction). For viscous liquid with a boiling range of greater than 10°C, a recovery rate of 95% (volume fraction) shall be satisfactory. If the recovery rate cannot comply with the above-mentioned requirements, the test shall be repeated.

8.7 If any residue is present, COOL the flask to room temperature. POUR the residue into a small measuring cylinder graduated in 0.1mL subdivisions. MEASURE the volume. RECORD it as residue. After the condenser tube has been drained, READ the total volume of the distillate as recovery record. RECORD the difference between 100 and the sum of the residue plus recovery as distillation loss.

9 Factors causing superheating and operating precautions

9.1 Introduction

In general, any condition whereby the temperature surrounding the vapor exceeds the temperature of the vapor in equilibrium with the liquid will cause

may be minimized, but not completely eliminated, by selecting an electric heater which, by its design, concentrates the heating elements to a minimum area, and which contains a minimum amount of ceramic material in its overall construction. The fulfilment of these requirements will reduce, but not completely eliminate, the amount of extraneous heat radiating around the perimeter of the heat-resistant board on which the distillation flask is placed.

10 Result calculation

10.1 APPLY the correction for the inner diameter of the thermometer and shrinkage of the mercury bulb according to a certain standard method or by relevant verification department.

10.2 APPLY the corrections for thermometer reading for deviation of the barometric pressure from normal according to Formula (1). TAKE the algebraic sum of the thermometer reading and the corrected value as the determination result. The corrected value (δ_t) is calculated as follows:

$$(\delta_t) = K\ (101.3 - P) \quad \text{..... (1)}$$

Where:

K - Rate of change of boiling point with pressure, in degrees Celsius per kilopascal (°C/kPa), as given in Table C.1 in Annex C;

P - Test barometric pressure corrected to 0°C via Table C.2, in kilopascal (kPa).

Note: For other pure compounds not listed in Table C.1, the value of K can be obtained from the literature. For narrow-boiling hydrocarbon materials, the value of K may be assumed as 0.000 12 times the normal boiling point (on the absolute temperature scale).

10.3 If the boiling range does not exceed 2°C, combined thermometer and barometric corrections may be made on the basis of the difference between the observed 50% (volume fraction) boiling point and the standard boiling point at 101.3kPa, as given in Table C.1.

10.4 The boiling range of the test piece shall be expressed as the temperature interval between the initial boiling point and the dry point, in degrees Celsius (°C).

11 Precision

The precision of the method obtained by statistical examination of inter-laboratory results is related to the purity and the boiling point of the test piece. In general, the precision increases with increasing purity and decreasing boiling

Diethylene glycol-monoethyl ether	104C	201.9	0.36
Diethylene glycol-monomethyl ether	104C	193.8	0.35
Dimethyl formamide	102C	153.0	0.33
Dipropylene glycol	106C	232.8	0.38
Di-iso-propyl ether	39C	68.3	0.31
Ethanol	39C	78.3	0.25
Ethyl acetate	39C	77.2	0.31
Ethyl benzene	41C	136.2	0.37
Ethylene glycol	104C	197.6	0.32
Ethylene glycol-mono-n-butyl ether (2-butoxyethanol)	103C	171.2	0.35
Ethylene glycol-monoethyl ether (2-ethoxyethanol)	102C	135.1	0.33
Ethylene glycol-monoethyl ether acetate (2-ethoxy ethylacetate)	102C	156.3	0.35
Ethylene glycol-monomethyl ether	41C	124.5	0.31
Ethylene glycol-mono-iso-propyl ether	102C	142.8	0.33
2-Ethyl hexanol	104C	184.8	0.34
Ethyl-iso-amyl ketone	103C	158.2	0.37
n-Hexyl acetate	103C	171.6	0.38
Hexylene glycol	104C	197.1	0.34
Isophorone (3,5,5-trimethyl-cyclohexene-2-one-1)	105C	215.3	0.43
Mesityl oxide (4-methyl-3-pentene-2-one)	41C	129.8	0.35
4-Methoxy-4-methyl-pentene-2-one	103C	160.6	0.37
Methanol	39C	64.6	0.25
Methyl ethyl ketone	39C	79.6	0.32
Methyl-iso-amyl acetate	102C	146.2	0.36
Methyl-iso-amyl ketone	102C	144.9	0.36
Methyl-iso-butyl carbinol	41C	131.8	0.31
Methyl-iso-butyl-ketone	41C	116.2	0.35
Ethanol amine	103C	170.7	0.30
Perchloroethylene	41C	121.2	0.36
n-Propyl acetate	40C	101.6	0.32
iso-Propyl acetate	40C	88.5	0.31
n-Propyl alcohol	40C	97.2	0.26
iso-Propyl alcohol	40C	82.3	0.25
Propylene glycol	104C	187.6	0.32
Propylene oxide (1,2-epoxy propane)	38C	34.3	0.27
Pyridine	41C	115.4	0.35
Toluene	41C	110.6	0.35
Triethylene glycol	107C	287.6	0.38
Triethylene glycol-monoethyl ether	106C	255.4	0.38
Trichlorethylene	40C	87.1	0.32
Vinyl acetate	39C	72.7	0.30
White spirit	103C	-	0.41
Xylene (isomer mixture)	41C	-	0.37
o-Xylene	41C	144.4	0.37

This is an excerpt of the PDF (Some pages are marked off intentionally)

------ The End ------