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Styrene — Part 2: Test method

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Foreword

This translation has been made based on the original Japanese Industrial Standard established by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee according to the proposal for establishment of Japanese Industrial Standard submitted by Japan Styrene Industry Association (JSIA)/Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law.

Consequently **JIS K 6727:2006** has been withdrawn and partially replaced with this Standard.

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JIS K 6727 series consists of the following two parts under the general title "*Styrene*":

Part 1 : Quality and marking

Part 2 : Test method

Styrene — Part 2 : Test method

Introduction

JIS K 6727 was established in 1963, and no technical revision has been made since then. This time, the specifications of test method have been revised to suit the state-of-the-art technology, and the part of quality standard and that of test method have been divided into independent standards.

No corresponding International Standard has been established at this point.

1 Scope

This Standard specifies the test method of styrene used as chemicals for resin use, synthetic rubber use, and coating use, etc.

WARNING : Persons using this Standard should be familiar with normal laboratory practice. This Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

JIS C 7601 *Fluorescent lamps for general lighting service*

JIS K 0050 *General rules for chemical analysis*

JIS K 0062 *Test methods for refractive index of chemical products*

JIS K 0071-1 *Testing methods for colour of chemical products — Part 1 : Estimation of colour in Hazen units (platinum-cobalt scale)*

JIS K 0114 *General rules for gas chromatography*

JIS K 0115 *General rules for molecular absorptiometric analysis*

JIS K 0557 *Water used for industrial water and wastewater analysis*

JIS K 1101 *Oxygen*

JIS K 1107 *Nitrogen*

JIS K 2249-1 *Crude petroleum and petroleum products — Determination of density — Part 1 : Oscillating U-tube method*

JIS K 2249-3 *Crude petroleum and petroleum products — Determination of density — Part 3 : Capillary pyknometer method*

JIS K 2276 *Petroleum products — Testing methods for aviation fuels*

- JIS K 2541-2 *Crude oil and petroleum products — Determination of sulfur content Part 2 : Oxidative microcoulometry*
- JIS K 2541-6 *Crude petroleum and petroleum products — Determination of sulfur content Part 6 : Ultraviolet fluorescence method*
- JIS K 6727-1 *Styrene — Part 1 : Quality and marking*
- JIS K 8073 *Benzoic acid (Reagent)*
- JIS K 8121 *Potassium chloride (Reagent)*
- JIS K 8150 *Sodium chloride (Reagent)*
- JIS K 8180 *Hydrochloric acid (Reagent)*
- JIS K 8201 *Hydroxylammonium chloride (Reagent)*
- JIS K 8247 *Potassium permanganate (Reagent)*
- JIS K 8271 *Xylene (Reagent)*
- JIS K 8355 *Acetic acid (Reagent)*
- JIS K 8372 *Sodium acetate (Reagent)*
- JIS K 8401 *Titanium (III) chloride solution (Reagent)*
- JIS K 8541 *Nitric acid (Reagent)*
- JIS K 8548 *Potassium nitrate (Reagent)*
- JIS K 8550 *Silver nitrate (Reagent)*
- JIS K 8574 *Potassium hydroxide (Reagent)*
- JIS K 8576 *Sodium hydroxide (Reagent)*
- JIS K 8643 *Thymol blue (Reagent)*
- JIS K 8680 *Toluene (Reagent)*
- JIS K 8780 *Pyrogallol (Reagent)*
- JIS K 8891 *Methanol (Reagent)*
- JIS K 8951 *Sulfuric acid (Reagent)*
- JIS K 8979 *Ammonium iron (II) sulfate hexahydrate (Reagent)*
- JIS K 8987 *Sodium sulfate (Reagent)*
- JIS K 9001 *Potassium thiocyanate (Reagent)*
- JIS P 3801 *Filter paper (for chemical analysis)*
- JIS R 3503 *Glass apparatus for chemical analysis*
- JIS Z 8401 *Guide to the rounding of numbers*
- JIS Z 8710 *Temperature measurement — General requirement*

3 Test method

3.1 General

The general rules for chemical analysis shall be in accordance with **JIS K 0050**, the gas chromatographic analysis in accordance with **JIS K 0114**, and the rounding of numerical values in accordance with **JIS Z 8401**. The rest shall be as follows.

- a) When reading a scale at liquid surface, use a measuring cylinder. In other cases, use the lower edge.
- b) The thermometer calibrated according to **JIS Z 8710** shall be used.

3.2 Preparation of test samples

3.2.1 General

The general matters related to the preparation of test samples shall be as follows.

- a) Sampling shall be carried out for each lot of the products. Lot means the products which can be regarded as having the same quality, and also means the following.
 - the products manufactured by the same batch
 - the stocks in the same tank
 - those delivered from the same tank to different tanks, and stored under the same condition.
- b) Sampling shall be basically carried out at the timing of factory shipment, but it may be determined between the parties concerned.
- c) In case a lot consists of at least two containers, some containers shall be taken out of the lot first, and samples shall be taken. By means of the random number die, random number table or other suitable methods, at least the number of samples given in table 1 shall be extracted.
- d) The mixed sample is prepared from the extracted sample so as to represent the average quality and it shall be made the test sample.

Table 1 Number of samples

Number of containers	Number of samples	Number of containers	Number of samples
1	1	126 to 216	6
2 to 8	2	217 to 343	7
9 to 27	3	344 to 512	8
28 to 64	4	513 to 729	9
65 to 125	5	730 to 1 000	10

3.2.2 Sampler and operation

The sampler, which is in accordance with the groups of container used for storage and transport of styrene, shall be used. The groups of container are divided into a

small-sized container (18-litre can, drum can, etc.) and a large-sized container (tank, petrol tanker, tanker, etc.). Each shall be as follows.

- a) **In the case of small-sized container** Use a small sampler. An example of its shape and dimension is shown in figure 1. Place the sampler vertically into the container with its opening left unclosed. After the sample reaches the bottom of the container, close the opening, take out the sampler and transfer it to an adequate container. Mix equal amount of sample taken from each container and use them for test.
- b) **In the case of large-sized container** Use a large sampler. Examples of shape and dimension of the large-sized samplers (A) and (B) are shown in figure 2 and figure 3 respectively. Open the lid at the upper part of large-container, put the sampler vertically in the large-sized container, with its cork stopper closed. After the opening of the sampler reaches the prescribed sampling location, fill the container with the sample by removing the stopper and take out the sampler as it is. Transfer the sample to an adequate container and mix it. For sampling locations and proportions of sampling amount, follow table 2 in the case of a horizontal and cylindrical tank. In other cases, divide the contents almost equally into three layers and take the same amount of the sample from central part of each layer.

Unit : mm

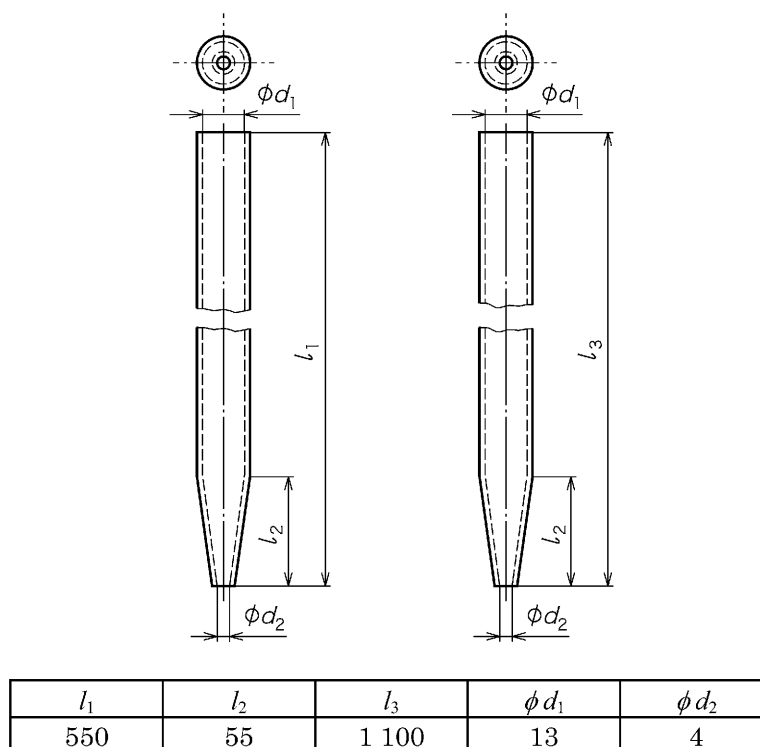
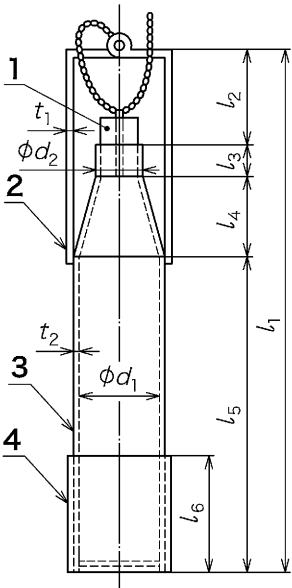


Figure 1 Example of small sampler (glass tube)

Unit : mm

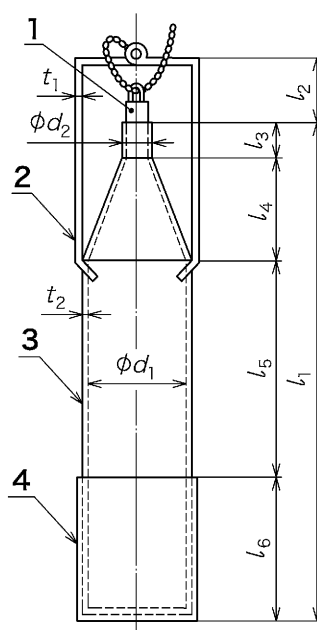


- 1 Cork stopper
- 2 Handle (made of stainless steel SUS304)
- 3 Stainless steel SUS304
- 4 Stainless steel SUS304 (at least 3 mm thick)

l_1	l_2	l_3	l_4	l_5	l_6	t_1	t_2	ϕd_1	ϕd_2
255	50	15	40	150	60	3	0.6	45	22

Figure 2 Example of large sampler (type A)

Unit: mm



- 1 Cork stopper
- 2 Handle (made of stainless steel SUS304)
- 3 Stainless steel SUS304
- 4 Stainless steel SUS304 (at least 5 mm thick)

l_1	l_2	l_3	l_4	l_5	l_6	t_1	t_2	ϕd_1	ϕd_2
345	45	25	70	150	100	3	0.6	76	22

Figure 3 Example of large sampler (type B)

Table 2 Sampling locations and proportions of sampling amount

Depth of content (% to diameter of tank)	Sampling locations (height from bottom) (% to diameter)			Proportion of sampling amount		
	Top	Middle	Bottom	Top	Middle	Bottom
100	80	50	20	3	4	3
90	75	50	20	3	4	3
80	70	50	20	2	5	3
70	—	50	20	—	6	4
60	—	50	20	—	5	5
50	—	40	20	—	4	6
40	—	—	20	—	—	10
30	—	—	15	—	—	10
20	—	—	10	—	—	10
10	—	—	5	—	—	10

3.3 Appearance

Take a part of the sample in a cylinder or wide-mouthed bottle, and observe and confirm the transparency and colourlessness without turbidity and the absence of mingling foreign matters such as floating and water drop.

3.4 Colour tone

Colour tone shall be measured by a colorimeter.

- a) **Apparatus and implement** The main apparatus and implement used for the measurement of colour tone shall be as follows.
- 1) **Colorimeter** Use the colorimeter specified in **JIS K 0071-1**.
 - 2) **Fluorescence discharge tube** Use the fluorescence discharge tube of day light and 6 500 K in colour temperature specified in **JIS C 7601**.
 - 3) **Colour comparison tube** Use the colour comparison tube specified in **JIS K 0071-1**.
 - 4) **Spectrophotometer** Use the spectrophotometer specified in **JIS K 0115**.
- b) **Standard solution** Standard solution shall be prepared as follows.
- 1) **Standard colorimetric solution stock solution** Prepare according to the method specified in **JIS K 0071-1**.
 - 2) **Standard colorimetric solution** Prepare the standard colorimetric solution corresponding to Hazen unit colour number 20 by the method specified in **JIS K 0071-1**.
- c) **Operation** Place the test sample in colour comparison tube up to the scale of 100 ml. Pour the standard colorimetric solution in a different colour comparison tube up to the scale of 100 ml, and put both tubes in the colorimeter and compare colours. A spectrophotometer may be used to compare colours, and if used, a wavelength of 375 nm shall be used.

3.5 Density and specific gravity

3.5.1 Density

Density shall be measured at 25 °C, in accordance with **JIS K 2249-1** or **JIS K 2249-3**.

3.5.2 Specific gravity

The specific gravity d shall be calculated from the formula.

$$d = \frac{D_s}{D_0}$$

where, D_s : density of test sample at 25 °C
 D_0 : density of water at 25 °C (0.997 05)

3.6 Refractive index

Refractive index shall be measured at 25 °C, in accordance with **JIS K 0062**.

3.7 Purity

Separate the test sample by using a capillary column according to gas chromatography, and obtain the purity from the peak area of chromatogram by the correction area

percentage method.

- a) **Apparatus and implement** The apparatus and implement shall be in accordance with JIS K 0114. Examples of main apparatus and implement are shown as follows.
- 1) **Apparatus** Gas chromatograph
 - 2) **Detector** Flame ionization detector (FID)
 - 3) **Separation column** Stainless steel tube, hard glass tube, or fused silica tube coated inside or chemically bonded with strong polar stationary liquid such as polyethylene glycol, of 0.2 mm to 0.6 mm in inside diameter, 25 m to 90 m in length.
 - 4) **Sample introduction part**, being equipped with an injection opening of liquid sample and a vaporizer.
 - 5) **Balance**, being capable of weighing to the nearest 1 mg.
 - 6) **Microsyringe**, being 1 μ L to 10 μ L in full scale.
 - 7) **Data processing apparatus**
- b) **Reagents and carrier gas** The reagents and carrier gas shall be as follows.
- 1) **Styrene**, whose purity is at least 99.7 % (mass fraction) and known.
 - 2) **Ethylbenzene**, whose purity is at least 98 % (mass fraction).
 - 3) ***o*-, *m*-, *p*-Xylene**, being specified in JIS K 8271.
 - 4) ***n*-Propylbenzene**, whose purity is at least 98 % (mass fraction).
 - 5) **4-Ethyltoluene**, whose purity is at least 98 % (mass fraction).
 - 6) **α -Methylstyrene**, whose purity at least 98 % (mass fraction).
 - 7) **Toluene**, being specified in JIS K 8680.
 - 8) **Phenylacetylene**, whose purity is at least 98 % (mass fraction).
 - 9) **Cumene**, whose purity is at least 98 % (mass fraction).
 - 10) **Nonane**, whose purity is at least 98 % (mass fraction).
 - 11) **Carrier gas**, being helium of at least 99.99 % (volume fraction) in purity or nitrogen specified in JIS K 1107.
- c) **Preparation of apparatus** Apparatus shall be prepared as follows.
- 1) **Pretreatment of separation column** Pass through carrier gas at service temperature for several hours and remove volatile matter sufficiently. In this case, the carrier gas exhausted shall not pass through the detector, since the volatile matter may contaminate the detector.
 - 2) **Confirmation of precision of apparatus** Operate the whole apparatus and confirm in chromatogram that the each peak is at least twice as high as noise level when 0.1 μ L to 2 μ L of the sample containing 0.01 % (mass fraction) of cumene and