

JIS

JAPANESE INDUSTRIAL STANDARD

**Testing methods
for polyvinyl alcohol**

JIS K 6726—1994

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**In the event of any doubt arising,
the original Standard in Japanese is to be final authority.**

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J I S

Testing methods
for polyvinyl alcohol

K 6726-1994

1. Scope This Japanese Industrial Standard specifies the testing methods for polyvinyl alcohol of which saponification degree is about 70 (mol %) or more.

- Remarks 1. The standards cited in this Standard are shown in Attached Table 1.
2. The units and numerical values given in { } in this Standard are based on the traditional units, and are appended for informative reference.

2. Sampling method

- (1) Summary Sample shall be taken from the one lot, whose quality is thought to be uniform according to the following method. From plural containers (such as paper bag or flexible container), take respectively the same amount of sample, transfer them into a suitable sample container, mix them well, and take its 500 g or more as test sample.
- (2) Implement and apparatus Implement and apparatus shall be as follows:
- (a) Sampler Made of stainless steel and capable of taking samples of required quantity. Fig. 1 and Fig. 2 show its example.
- (b) Sample reducer Capable of reducing sample. Fig. 3 shows its example.
- (3) Operation Operation shall be as follows:
- (a) Number of containers to be sampled When plural containers are to be sampled from one lot, take at random specified number of containers shown in Table 1.

If the quality of sample is confirmed to be stable, the number of containers to be sampled may be varied.

Table 1. Number of containers to be sampled

Number of containers	Containers to be sampled	Number of containers	Containers to be sampled
1 to 4	Whole containers	76 to 100	10
5 to 10	5	101 to 125	11
11 to 20	6	126 to 150	12
21 to 30	7	151 to 200	13
31 to 50	8	201 to 250	14
51 to 75	9	251 or more	14 + $\alpha^{(1)}$

Note ⁽¹⁾ " α " shall be calculated according to the following formula and smaller part than decimal point is raised up to make a whole number.

$$\alpha = \frac{n - 250}{50}$$

where, n : number of containers

- (b) Sampling method of sample Open the containers that were sampled, pick up respectively the same quantity of sample from spots as many as possible using a previously cleaned and dried sampler, and then mix them sufficiently. Next, reduce it until specified amount (500 g or more) using a sample reducer, and make it a test sample ⁽²⁾.

Note ⁽²⁾ When test is not immediately performed, store it in a closely sealed container.

Fig. 1. Example of sampler (A)

Fig 2. Example of sampler (B)

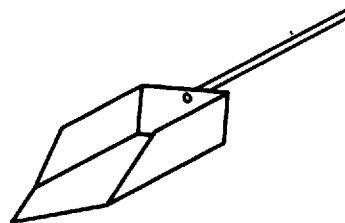
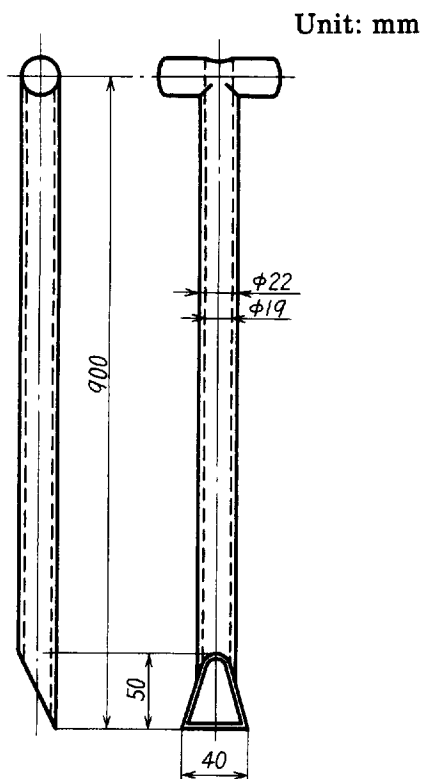
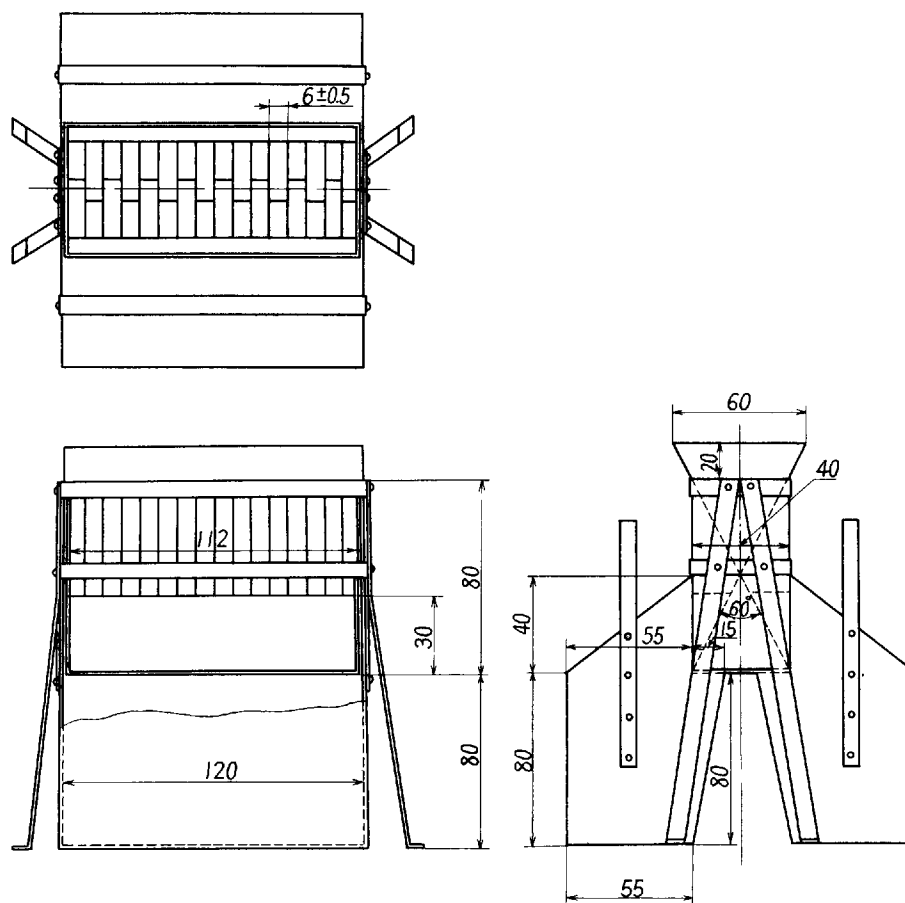


Fig. 3. Example of sample reducer

Unit: mm



3. Testing methods

3.1 General matters General matters applicable in common to tests shall follow JIS K 0050.

3.2 Expression of numeral test results Round off test result according to JIS Z 8401.

3.3 Test items

- (1) Volatile component
- (2) Saponification degree
- (3) Sodium acetate
- (4) Average degree of polymerization
- (5) Ash content
- (6) Purity
- (7) Grain size
- (8) Viscosity
- (9) Transparency
- (10) pH

3.4 Volatile component

- (1) Summary Find the loss when sample is heated at $105 \pm 2^\circ\text{C}$.
- (2) Implements and apparatus Implements and apparatus shall be as follows:
- (a) Weighing bottle Flat-type weighing bottle, measuring 60×30 mm, specified in JIS R 3503.
 - (b) Desiccator Specified in JIS R 3503 and using desiccant of silica gel type A class 1 specified in JIS C 0701.
 - (c) Thermostatic drier Capable of keeping temperature of $105 \pm 2^\circ\text{C}$.
 - (d) Chemical balance or electrobalance

Remarks: When measuring volatile component, "moisture meter" or "electronic moisture meter", which are found on the market, may be used. In this case, in order that there is no difference between the result by these meters and the result obtained by the drier at $105 \pm 2^\circ\text{C}$, measuring condition shall be established after measuring and comparing the volatile component in the same sample.

- (3) Operation Operation shall be as follows:
- (a) Dry a previously washed weighing bottle in a thermostatic drier at $105 \pm 2^\circ\text{C}$ for one hour or longer, allow it to cool in a desiccator to room temperature and weigh its mass to the nearest 1 mg.
 - (b) Then, weigh accurately about 5 g of sample into the weighing bottle to the nearest 1 mg and dry it at $105 \pm 2^\circ\text{C}$ for 3 h.
 - (c) Take out the weighing bottle from the drier, allow it to cool to room temperature in a desiccator and weigh accurately the mass of the weighing bottle to the nearest 1 mg.
- (4) Calculation Calculate according to the following formula. Round off the result to get the second decimal place.

$$R = \frac{W_1 - W_2}{W_1 - W_3} \times 100$$

where, R : volatile component (%)

W_1 : mass of sample and weighing bottle before drying (g)

W_2 : mass of sample and weighing bottle after drying (g)

W_3 : mass of weighing bottle (g)

3.5 Saponification degree

- (1) Summary Determine residual acetate acid radical (mol %) in sample using sodium hydroxide, and subtract it from 100 to find saponification degree (mol %).

- (2) Implements and apparatus Implements and apparatus shall be as follows:
- (a) Interchangeable ground joint Erlenmeyer flask 300 ml, specified in JIS R 3503.
 - (b) Buret 50 ml, specified in JIS R 3505.
 - (c) Measuring cylinder 100 ml, specified in JIS R 3505.
 - (d) Dissolving apparatus with a heater and a stirrer Capable of heating while stirring.
- (3) Reagents Reagents shall be as follows:
- (a) 0.1 mol/l sodium hydroxide solution Specified in 4.5 (19.4) of JIS K 8001.
 - (b) 0.5 mol/l sodium hydroxide solution Specified in 4.5 (19.2) of JIS K 8001.
 - (c) 0.1 mol/l sulfuric acid Specified in 4.5 (26.3) of JIS K 8001.
 - (d) 0.5 mol/l sulfuric acid Specified in 4.5 (26.1) of JIS K 8001.
 - (e) 0.1 mol/l hydrochloric acid Specified in 4.5 (5.5) of JIS K 8001.
 - (f) 0.5 mol/l hydrochloric acid Specified in 4.5 (5.3) of JIS K 8001.
 - (g) Phenolphthalein solution (10 g/l) Prepare according to 4.4 of JIS K 8001.
- (4) Operation Operation shall be as follows:
- (a) Weigh sample corresponding to estimated saponification degree, according to the amount of sample specified in Table 2, into an interchangeable ground joint Erlenmeyer flask to the nearest 1 mg.
 - (b) Add 100 ml of water ⁽³⁾ and 3 drops of phenolphthalein solution ⁽⁴⁾, and dissolve the sample completely in a dissolving apparatus with a heater and a stirrer at 90°C or higher while heating and stirring.
 - (c) After cooling it to room temperature, as shown in Table 2, add 25.00 ml of 0.1 mol/l or 0.5 mol/l sodium hydroxide solution by a buret, and after stoppering and shaking enough, keep it at room temperature for at least 2 h.
 - (d) Add 25.00 ml of sulfuric acid or hydrochloric acid, which has the same concentration as that of the sodium hydroxide solution, into the interchangeable ground joint Erlenmeyer flask by a buret, stopper it, and shake it sufficiently.
 - (e) Titrate it with 0.1 mol/l or 0.5 mol/l sodium hydroxide solution until it turns faint red according to Table 2.
 - (f) Carry out a blank test according to the procedures from (b) to (e) without adding sample.

Notes ⁽³⁾ The sample with low saponification degree may give white turbidity. In this case, it is preferable to gradually cool while stirring gently.

Alternatively, the solution mixed of water and methanol specified in JIS K 8891 at the ratio of 3 : 1 (volume ratio) may be used.

(4) If phenolphthalein gives red coloring, separately titrate the same sample with 0.1 mol/l or 0.5 mol/l sulfuric acid or hydrochloric acid until the faint red disappears, and then correct the saponification degree making use of its consumed volume.

If it gives no faint red coloring, titrate it with 0.1 mol/l or 0.5 mol/l sodium hydroxide solution until it turns faint red, and then correct the saponification degree making use of its consumed volume, as occasion calls.

Table 2. Estimated saponification degree, sampled amount and normal solution used

Estimated saponification degree mol %	Sampled amount g	Normal solution used	
		Concentration mol/l	Volume used ml
97 or more	3	0.1	25.00
90 or over to and excl. 97	3	0.5	25.00
80 or over to and excl. 90	2	0.5	25.00
70 or over to and excl. 80	1	0.5	25.00

Remarks: As a convenient method, after the procedures from (a) to (c) has finished, it may be directly titrated with 0.1 mol/l or 0.5 mol/l sulfuric acid or hydrochloric acid ⁽⁵⁾.

(5) Calculation Calculate according to the following formulae. Round off the value to get the second decimal place.

$$X_1 = \frac{(a - b) \times f \times D \times 0.06005}{S \times \frac{P}{100}} \times 100$$

$$X_2 = \frac{44.05 \times X_1}{60.05 - 0.42 \times X_1}$$

$$H = 100 - X_2$$

- where, X_1 : amount of acetic acid equivalent to residual acetate acid radical (%)
- X_2 : residual acetate acid radical (mol %)
- H : saponification degree (mol %)
- a : consumed volume of 0.1 mol/l or 0.5 mol/l sodium hydroxide solution (ml)
- b : consumed volume of 0.1 mol/l or 0.5 mol/l sodium hydroxide solution at blank test (ml)