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JAPANESE INDUSTRIAL STANDARD

Glycerines for Industrial Use

Ⓔ JIS K 3351 —1984



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JAPANESE INDUSTRIAL STANDARD

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Glycerines for Industrial Use

K 3351-1984

1. Scope

This Japanese Industrial Standard specifies the glycerines for industrial use.

2. Classification

The classification shall be as follows:

(1) Purified glycerine

- (a) No. 1 (purified glycerine)
- (b) No. 2 (glycerine for dynamite)

(2) Crude glycerine

- (a) No. 1 (cracked glycerine)
- (b) No. 2 (by-product glycerine)

3. Quality

The quality of glycerines for industrial use shall be tested in accordance with 5. Then, purified glycerine shall conform to the requirements specified in Table 1 and crude glycerine shall conform to the requirements specified in Table 2.

Applicable Standards and

Corresponding International Standards: See pages 25 and 26.

**Documents**

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Table 1. Purified Glycerine

Test items	No. 1 (purified glycerine)	No. 2 (glycerine for dynamite)
Property	It is colourless transparent, and almost odorless viscous liquid	It is colourless transparent, and viscous liquid
Colour number Hazen unit	20 max.	30 max.
Acidity or alkalinity meq/100 g	0.3 max.	0.3 max.
Density (20°C)	1.257 min.	1.257 min.
Glycerine content %	98.5 min.	98.5 min.
Ignition residue (sulfate) %	0.05 max.	0.05 max.
Saponification equivalent meq/100 g	3.0 max.	3.0 max.
Reducing matter test	Accepted	Accepted
Chloride test	—	Accepted
Nitrification and separation test ⁽¹⁾ (Reference)	—	Accepted

Note ⁽¹⁾ The nitrification and separation test shall be carried out as agreed between the parties concerned.

Table 2. Crude Glycerine

Test items	No. 1 (cracked glycerine)	No. 2 (by-product glycerine)
Property	It is yellow or thick brown viscous liquid	It is yellow or thick brown viscous liquid
Liquid properties	It is neutral or slightly alkaline	It is neutral or slightly alkaline
Glycerine content %	88.0 min.	80.0 min.
Ash content %	1.0 max.	10.0 max.
Organic impurity content %	3.0 max.	6.0 max.
Arsenic test	Accepted	Accepted

4. Sampling Method

4.1 Sampling The sampling shall be carried out concerning each one lot of products in accordance with 4.2 and 4.4 according to the type of vessel specified as follows:

- (1) Small Size Vessel (18 l can, drum, etc.)
- (2) Large Size Vessel (tank, tank lorry, tanker, etc.)

4.2 Lot The one lot means the following products considered, as a rule, as the same quality: The products produced from the same batch, the products in the same tank, or the products filled up from the same tank and stored under the same conditions.

4.3 Identification of Lot The sample vessel shall be recorded on each vessel with sampling date and lot number or tank number so as to identify the lot.

4.4 Sampling Method The sample shall be taken in accordance with the following method according to either of small size vessel or large size vessel.

- (1) Small Size Vessel Extract the number of pieces given in Table 3 at random in accordance with random number die, table of random numbers or other suitable method, sample an all layer sample from each vessel and mix them to be the representative sample.

Table 3. Sampling Number of Pieces in Small Size Vessel

Purified glycerine		Crude glycerine	
Number of vessels	Sampling number of pieces	Number of vessels	Sampling number of pieces
1 to 10	1	1 to 10	1 to 3
11 to 100	2	11 to 25	2 to 4
101 to 300	3	26 to 50	3 to 6
301 to 500	4	51 to 75	6 to 8
501 min.	5	76 to 100	8 to 10
		101 min.	10 % (2)

Note (2) The decimal place shall be rounded up to a integer.

- (2) Large Size Vessel For the large size vessel, the sampling shall be as follows:

- (a) For Sampling from Vessel For purified glycerine, sample of one piece at least from either one of the upper, middle and lower layers of vessel (about 15 %, 50 % and 85 % in depth from the solution surface).

For two pieces min., change the position of sampling. For crude glycerine, sample total 3 pieces from abovementioned respective layers. When the taken sample is of one piece, allow it as it is to be the representative sample and when the taken sample is of two pieces min., mix them to be the representative sample.

- (b) For Sampling from Pipeline For purified glycerine, sample one piece at least from either one of the initial, middle and final periods thereof during transportation. When the taken sample is of two pieces min., change the sampling period.

For crude glycerine, respectively by sample one piece from the initial, middle and final periods thereof during transportation and mix them to be the representative sample.

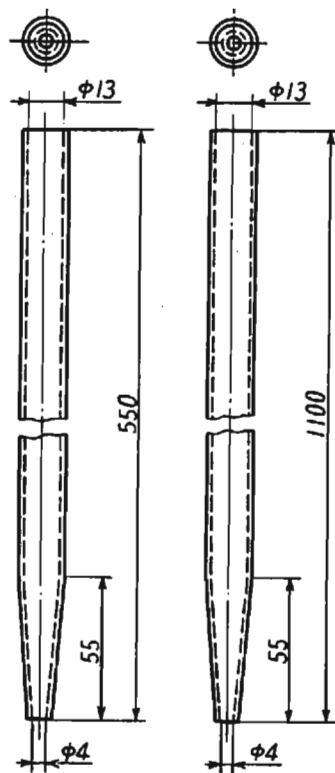
- (3) Sampling Amount of Representative Sample The sampling amount of representative sample shall be 1 to 1.5 l, as required.
- (4) Analysis Sample The representative sample is divided into 2 to 3 pieces, as required, put into analysis sample vessels and sealed to be the analysis sample. The amount of analysis sample shall be 500 ml.

4.5 Devices and Sample Vessel The device and the sample vessel shall be as follows:

- (1) Device The sampling device of sample is of the shape as given in Figs. 1 and 2 and of the quality stable to the sample such as those of glass, stainless steel, etc.

Fig. 1. An Example of Small Size Liquid Sampler (Glass Tube)

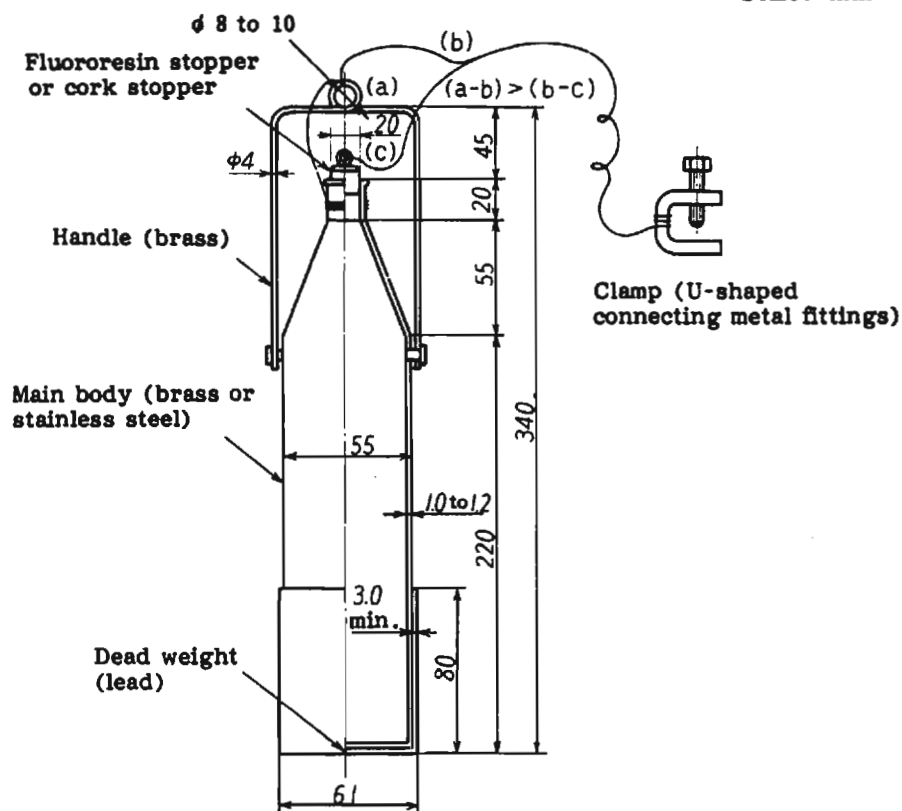
Unit: mm



(Capacity about 65 ml) (Capacity about 130 ml)

Fig. 2. An Example of Large Size Liquid Sampler
(Metallic Sampler with Dead Weight)

Unit: mm



- (2) Sample Vessel The sample vessel is the one of chemical resistant material such as hard glass or the like, with a cover capable of being sealed.

4.6 Cautions on Sampling Cautions on sampling shall be as follows:

- (1) Because glycerine is considerably easy to absorb moisture, it shall be always necessarily isolated from the wet atmosphere. Therefore, the sample vessel shall be sealed excepting the pouring time of sample and the taking out time thereof.
Further, the sampling shall be carried out under a roof as far as possible and the vessel shall be especially free from rain and other accidental contamination.
- (2) For the sample containing precipitates or suspension matters, the sample shall be put therein to a height of two thirds the vessel to make uniform easily the sample. For other cases, the sample shall be filled to the top of vessel.
- (3) Because the congeal point of pure glycerine is of about 18°C, the sample shall be preferably taken during filling or otherwise just after filling. When the analysis sample has been frozen, after making uniform all the glycerine by thawing the sample by heating it at 60°C max., it is used.
- (4) The clean, dry device and vessel shall be always used in service.

5. Test Methods

5.1 General Matters General matters common to the test shall be in accordance with JIS K 0050.

5.2 Property The sample shall be examined by taking the sample into a clean dry glass vessel.

5.3 Colour Number The measurement of colour number shall be as follows:

- (1) **Summary** Take a sample into a colour comparison tube, compare its colour with Hazen standard solution and express the colour number by Hazen unit.
- (2) **Devices** The devices shall be as follows:
 - (a) **Colour Comparison Tube** It is the flat-bottom glass tubes with ground stopper of 25 mm in inside diameter, respectively having the same quality, the same shape and the same thickness. Further, the tubes are notched with marked lines at the same height from the bottom so that the amount of solution becomes about 100 ml.
 - (b) **White Plate** Filter paper (qualitative analysis filter paper Class 2 or equivalent one)
- (3) **Hazen Standard Solution** Either of 2.65 g of hydrogen hexachloroplatinate (IV) hexahydrate specified in JIS K 8153⁽³⁾ or 2.49 g of potassium hexachloroplatinate specified in JIS K 8163⁽³⁾ and 2.00 g of cobalt chloride specified in JIS K 8129 are respectively weighed out, joined by 200 ml hydrochloric acid and sufficiently dissolved. Thereafter, it is diluted to 2 l by adding water to be the standard solution of Hazen 500. The standard solution of under 500 in Hazen is prepared by mixing the standard solution of 500 in Hazen and water in accordance with Table 4, sealed and stored in a cold dark place. In that case, the standard solution of 500 in Hazen for which one year min. has elapsed after preparation and otherwise the standard solution of under 500 in Hazen for which one month min. has elapsed, shall not, as a rule, be used.

Note (3) It is the amount containing 1.00 g platinum.

Table 4. Hazen Standard Solution

Number of Hazen standard solution	Standard solution of 500 in Hazen ml	Water ml	Number of Hazen standard solution	Standard solution of 500 in Hazen ml	Water ml
0	0	500	25	25	475
5	5	495	30	30	470
10	10	490	35	35	465
15	15	485	40	40	460
20	20	480	50	50	450

- (4) Operation The sample and the Hazen standard solution are respectively put into the colour comparison tube up to its marked line, placed on a white plate and the colours are compared by looking through them from the upside to the downside of colour comparison tube under scattering daylight.
- (5) Judgement The Hazen standard solution most closely resembling the sample is determined and the colour number of sample is expressed by that number of Hazen standard solution.

5.4 Liquid Property The test of liquid property shall be as follows:

- (1) Summary A sample solution is tested with a litmus test paper.
- (2) Operation The sample is diluted to twice the volume with water and its liquid property is judged with a litmus test paper.
- (3) Judgement When the litmus test paper does not turn red or otherwise it slightly turns blue, the sample shall be accepted.

5.5 Acidity or Alkalinity The test of acidity or alkalinity shall be as follows:

- (1) Summary Add phenolphthalein solution to a sample solution, titrate it with hydrochloric acid or sodium hydroxide standard solution and obtain the acidity or alkalinity as milli equivalent per 100 g (meq/100 g).
- (2) Reagents The reagents shall be as follows:
 - (a) 1/10 mol/l (N/10) Hydrochloric Acid Standard Solution It is the solution specified in JIS K 8006.
 - (b) 1/10 mol/l (N/10) Sodium Hydroxide Standard Solution It is the solution specified in JIS K 8006.
 - (c) Water Containing no Carbonic Acid It is the water specified in 13. of JIS K 8001.
 - (d) Phenolphthalein Solution (1 %/v %) It is the solution prepared by dissolving 1.0 g phenolphthalein specified in JIS K 8799 with 100 ml ethanol specified in JIS K 8102.
- (3) Device The device shall be as follows:

Microburet It is the one with the scale of 2 ml or 5 ml and 0.05 ml scale mark.
- (4) Operation The operation shall be carried out as follows:
 - (a) Weigh out about 100 g of a sample into an Erlenmeyer flask of 500 ml to the nearest 0.1 g.
 - (b) Add about 200 ml water containing no carbonic acid and sufficiently mix them by shaking.