भारतीय मानक Indian Standard

> मादक पेय के नमूना लेने के तरीके (दूसरा पुनरीक्षण)

Methods of Sampling for Alcoholic Drinks

(Second Revision)

ICS 67.160.10

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भारतीय मानक ब्यूरो BUREAU OF INDIAN STANDARDS मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002 MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG NEW DELHI - 110002 www.bis.gov.in www.standardsbis.in

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Price Group 6

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Alcoholic Drinks Sectional Committee had been approved by the Food and Agriculture Division Council.

The alcoholic industry is a well-organized and established industry in India. Substantial quantities of the products are also being exported. It is, therefore, imperative that there is strict quality control and due consideration is given to sampling procedures which will help in proper and objective evaluation of the various characteristics. This standard lays down the methods of sampling for alcoholic drinks. While evolving this standard, due weightage has been given to the trade practices.

The sampling procedures given in this standard include, besides lot inspection plan, recommended provisions for process control. Exercise of proper quality control during process would substantially reduce quality fluctuations and help the manufacturer in achieving in-built quality of the product. Lot inspection plan would enable the manufacturer or purchaser or other independent inspection and testing agencies to decide the conformity or otherwise of the product to the relevant specification.

This standard is intended to introduce uniform methods of sampling for alcoholic drinks. It does not deal with the specifications of the products, but prescribes only the sampling procedures for determining the conformity of the manufactured products to the specified quality, besides recommended process control provisions, and thus forms a necessary adjunct to the series of Indian Standard specifications for individual alcoholic drinks.

This standard was originally published in 1967 and first revised in 1984. This revision is being brought out to incorporate latest technological developments in alcoholic drinks industry and the major changes include:

- a) Use of sampling can as a sampling instrument has been removed as the same is no longer used;
- b) Design of sampling tube has been modified, as per latest industrial practice; and
- c) Scale of sampling has been enlarged to cover bigger lots.

This standard is subject to the restrictions imposed under the *Food Safety and Standards Act*, 2006 and the rules framed there under, and the *State Excise Duty Rules* which permit the withdrawal of duty free samples for testing. It is recommended that samples for testing by Bureau of Indian Standard or any other government recognized inspection and testing agency, whenever called for, may also be exempted from excise duty.

The composition of the Committee responsible for the formulation of this standard is given in <u>Annex C</u>.

For the purpose of deciding whether a particular requirement of the standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

METHODS OF SAMPLING FOR ALCOHOLIC DRINKS

(Second Revision)

1 SCOPE

This standard prescribes the methods of sampling and criteria for ascertaining the conformity of the alcoholic drinks to the relevant product specifications. It also includes the recommended provisions for process control for the guidance of manufacturers.

2 REFERENCES

The standard given below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the edition indicated was valid. This standard is subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of this standard.

IS No. Title

IS 490	5 : 201	5/	Random	sam	pling	and
ISO	24153	:	randomizat	tion	procee	lures
2009			(first revisi	ion)		

3 GENERAL REQUIREMENTS OF SAMPLING

In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed as far as possible:

- a) Samples shall be taken in a protected place not exposed to damp air, dust or soot;
- b) The sampling instrument shall be clean and dry;
- c) To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means;
- d) Precautions shall be taken to protect the samples, the product being sampled, the sampling instrument and the containers for samples from extraneous contamination;
- e) Samples shall be placed in suitable clean, dry and airtight glass containers;

- f) The sample containers shall be of such a size that sufficient head space to allow for expansion of the liquid, is left after pouring in the sample;
- g) Each sample container shall be sealed airtight with a suitable stopper after filling;
- Each sample container shall be marked with full details of sampling, the date of sampling and the year of manufacture of the product and other details necessary for traceability; and
- j) Samples shall be stored in a cool, dark and dry place.

4 SAMPLING INSTRUMENTS

4.1 The following types of sampling instruments may be used:

- a) Weighted sampling can for taking samples from various depths of large tanks; and
- b) Sampling tube.

4.2 Weighted Sampling Can

This can, shown in Fig. 1, shall be made of stainless steel. It shall be of suitable capacity, for example, 1 litre, and of such dimensions that it will pass freely through the tank dip-hatch. The can shall be of such a mass as to sink rapidly in the product to be sampled. The metal used to weigh the apparatus shall either be fitted externally or be contained in the space provided by fitting the can with a false bottom. The apparatus shall be provided with means to permit filling at any desired level and closure, and shall have a suitable long chain attached to it.

4.3 Sampling Tube

It is a tube made of metal with a protrusion so that it may be attached to dip tape (*see* Fig. 2) for easy insertion into the tank. For taking a sample, the tube is attached to the dip tape and lowered until the desired depth is reached. It is then allowed to admit the product and finally withdrawn.

The size of the sampling tube may vary as per requirement.

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FIG. 1 WEIGHTED SAMPLING CAN



FIG. 2 SAMPLING TUBE

5 PROCESS CONTROL

5.1 One sample from each consignment of product received in the factory shall be tested for important requirements of that product.

5.2 The manufacturer shall make appropriate checks and take representative samples at intermediate

stages of processing so as to control the quality fluctuations and hence ensure that the finished product is of desired quality.

5.3 The inspection levels given below are recommended for routine control over the manufacturing process.

5.3.1 Fermented Alcoholic Beverages (Beer and Wines)

5.3.1.1 Beer

The entire quantity of beer manufactured and stored at a time in each bottling vat just prior to bottling shall constitute a batch.

The recommended frequency of testing for various characteristics at different stages of manufacture is given in <u>Annex A</u>.

5.3.1.2 Wines

The provisions similar to that of beer given in 5.3.1.1 shall apply with the modification that the stage 'wort boiling' shall be deleted and the stage 'Meshing' shall be replaced by 'juice extraction'.

5.3.2 Distilled Alcoholic Beverages

5.3.2.1 Whisky, Rum, Gin and Brandy

The quantity of product manufactured at a time in the compounding/dilution/distillation tank shall constitute a batch.

5.3.2.2 The recommended frequency of testing for various characteristics at different stages of manufacture is given in <u>Annex B</u>.

6 LOT INSPECTION

6.1 If the manufacturer has maintained an adequate and satisfactory system of quality control in the manufacture of alcoholic drinks, the resulting data and information may be made available to the purchaser along with the product supplied to enable purchaser to judge the acceptability of the consignment. When it is not possible to provide this

information or if the purchaser so desires, the procedure laid down in the following clauses shall be followed for determining the conformity of product to the requirements of the specification.

6.2 Scale of Sampling

6.2.1 Lot

The quantity of packed alcoholic drink of the same type, belonging to the same batch of manufacture (*see* 5.3.1.1 and 5.3.2.1) and packed in a day, shall constitute a lot.

6.2.2 For ascertaining the conformity of the product to the requirements of the relevant specification, samples shall be tested from each lot separately.

6.2.3 The number of bottles to be selected from a lot shall depend on the size of the lot and shall be according to <u>Table 1</u>. The bottles selected for net volume according to co1 (4) of <u>Table 1</u> shall be in addition those selected according to co1 (3) of <u>Table 1</u>.

These bottles shall be chosen at random from the lot. In order to ensure the randomness of selection, procedures given in IS 4905 may be followed.

6.2.4 Initially the number of cartons equal to the number of bottles to be taken from the lot in one set (*see* <u>6.3.3</u>), shall be chosen at random. These cartons thus selected shall be opened and the bottles in these cartons examined visually for the condition of packing, the external appearance and the fill. The lot shall be considered satisfactory for inspection of other characteristics given in the specification, if all the bottles in the cartons opened are found meeting the requirements for these characteristics.

Sl No.	No. of Bottles in the Lot	Sample Size			
		Requirements Other Than Net Volume	Net Volume		
(1)	(2)	(3)	(4)		
i)	Up to 5 000	9	18		
ii)	5 001 to 10 000	12	36		
iii)	10 001 to 15 000	15	72		
iv)	15 001 and 20 000	21	108		
v)	20 001 and 25 000	24	144		

Table 1 Scale of Sampling

(Clauses	6.2.3.	6.3.1	and	6.4.1
۰.	CIGINGUS	0.2.0	0.5.1	unu	0.1.1

6.2.5 In case any defective bottle is found according to 6.2.4, twice the number of cartons shall be opened and the bottles examined for similar characteristics. If no defective bottle is found, the lot shall be considered satisfactory for inspection of other characteristics given in the specification.

6.3 Preparation of Test Samples

6.3.1 From each of the cartons opened according to **6.2.4**, three bottles shall be taken from its different layers so as to obtain the required number of bottles in the sample [*see* co1 (3) and (4) of Table 1].

6.3.2 In case the number of cartons to be opened is according to <u>6.2.5</u>, the number of cartons equal to the number of bottles in a set (*see* <u>6.3.3</u>) shall be taken at random from these cartons and then the required number of bottles picked up according to <u>6.3.1</u>.

6.3.3 The sample bottles selected as in 6.3.1 or 6.3.2 shall be divided at random into three equal sets and labelled with all the particulars of sampling. One of these sets of sample bottles shall be for the purchaser, another for the vendor and the third for referee. The sample bottles to be tested for net volume shall be kept separately.

6.3.4 Referee Sample

Referee sample shall consist of a set of sample bottles marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier so as to be used in case of a dispute between the two.

6.4 Number of Tests

6.4.1 Beer and Wines

Tests for carbon microbiological dioxide, requirements pH and organoleptic requirements in this sequence shall be carried out on the individual sample bottles selected according to co1 (3) of Table 1. The net volume shall be tested on each of the individual bottles selected in each set (see 6.3.3). Tests for the remaining requirements of this specification shall be conducted on the composite sample prepared by thoroughly mixing approximately equal quantity of product from the individual sample bottles selected a in each set (see 6.3.3).

6.4.2 Whisky, Gin, Brandy, Rum

Tests for ethyl alcohol content by hydrometer method shall be carried out on individual sample bottles selected in each set (see 6.3.3) and net

volume shall be tested on each of the individual bottles selected in each set (see 6.3.3). Organoleptic requirements shall also be tested on individual sample bottles. Tests for remaining requirements of the relevant specification shall be carried out on the composite sample prepared by thoroughly mixing approximately equal quantity of product from the individual sample bottles selected in each set (see 6.3.3).

6.5 Criteria for Conformity

The lot shall be declared as conforming to the requirements of the relevant specification if 6.5.1, 6.5.2, 6.5.3 and 6.5.4 are satisfied.

6.5.1 For those characteristics tested on the composite sample, all the test results shall satisfy the corresponding specification requirements.

6.5.2 For each characteristic other than organoleptic and microbiological tested on the individual samples, the average (\bar{x}) and range (R) shall be computed as follows:

Average
$$(\overline{X}) = \frac{\text{The sum of test results}}{\text{Number of test results}}$$

Range (R) = Difference between the maximum and the minimum values of the test results

6.5.2.1 If the specification limit for the characteristic is given as minimum, the value of expression $(\bar{x} - 0.6R)$ shall be calculated from the relevant test results. If the value so obtained is greater than or equal to the minimum limit, the lot shall be deemed to have conformed to the requirements of that characteristic.

6.5.2.2 If the specification limit for the characteristic is given as a maximum, the value of expression ($\overline{x} - 0.6R$) shall be calculated from the relevant test results. If the value so obtained is less than or equal to the maximum limit, the lot shall be considered as satisfying the requirements of that characteristic.

6.5.2.3 If the characteristic has two sided specification limits, the values of the expression $(\bar{x} - 0.6R)$ and $(\bar{x} + 0.6R)$ shall be calculated from the relevant test results. If the values so obtained lie between the two specification limits, the lot shall be considered as meeting the requirements of that characteristic.

6.5.3 For organoleptic and microbiological requirements, if all the test results on each of the individual samples satisfy the relevant specification requirements, the lot shall be considered as satisfying the requirements of that characteristic.

6.5.4 For net volume the bottles selected from each set (*see* **6.4**) meant for testing net volume shall be randomly divided into groups of 12 bottles each. The net volume shall be the volume indicated on the bottle subject to the tolerance for individual bottle given in the relevant specification. The total volume of 12 bottles shall also be examined according to relevant tolerances. The lot shall be deemed to have satisfied the requirement of net volume if <u>6.5.4.1</u> and <u>6.5.4.2</u> are satisfied.

6.5.4.1 Each individual sample bottle shall satisfy the requirement of net volume indicated on the bottle subject to the corresponding tolerances given in the individual product specification.

6.5.4.2 Each group of 12 bottles shall satisfy the requirement of net volume subject to the corresponding tolerances given in the individual product specification.

ANNEX A

(*Clause* 5.3.1.1)

RECOMMENDED FREQUENCY OF TESTING AT DIFFERENT STAGES OF
MANUFACTURE OF BEER

SI No.	Stage	Characteristic	Frequency	
(1)	(2)	(3)	(4)	
i)	Wort boiling	Specific gravity	One sample from each wort kettle	
ii)	Meshing	Specific gravity	One sample from each mesh tank	
iii)	Fermentation	Specific gravity and temperature	One sample from each ferment or	
iv)	Bottling tank	Colour, clarity, ethyl alcohol content, pH and carbon dioxide	A composite sample from each batch after the product is filtered and carbonized. For this purpose, small portions of product drawn periodically at every two hours shall be mixed thoroughly	
v)	Storage	Specific gravity, colour and <i>p</i> H	One sample from each storage tank at regular intervals	
vi)	Bottling machine	a) Organoleptic and ethyl alcohol content	Three samples at regular intervals per shift from each machine	
		b) Microbiological requirements	One sample per shift from each machine	
		c) Net volume	12 bottles per hour in each shift	
vii)	Pasteurization	Temperature control for effective pasteurization	Three samples per shift from each machine	

ANNEX B

(<u>Clause 5.3.2.2</u>)

Sl No.	Stage	Characteristic	Frequency
(1)	(2)	(3)	(4)
i)	Meshing	Dilution and specific gravity of molasses solution	One sample from each mesh tank
ii)	Fermentation	Temperature and specific gravity	One sample from each fermentation tank
iii)	Distillation	All requirements given in the relevant product specification except maturation and net volume	A composite sample prepared by mixing portions of product taken at different intervals from last distillation column in a shift
iv)	Maturation	Quality of maturation	One sample every three months from each wooden cask or barrel or vat
v)	Bottling	 a) Colour, clarity and ethyl alcohol content (by pycnometric or hydrometer method as per the requirements) 	One representative sample from each batch
		b) Net volume	One sample every half-an hour

RECOMMENDED FREQUENCY OF TESTING AT DIFFERENT STAGES OF MANUFACTURE OF WHISKY, RUM, GIN AND BRANDY

ANNEX C

(Foreword)

COMMITTEE COMPOSITION

Alcoholic Drinks Sectional Committee, FAD 29

Organization

CSIR - Central Food Technological Research Institute, Mysuru

All India Distillers Association, New Delhi

Central Revenue Control Laboratory, New Delhi

Confederation of Indian Alcoholic Beverage Companies, New Delhi

Craft Brewers Association of India, Bengaluru

CSIR - Central Food Technological Research Institute, Mysuru

Defence Food Research Laboratory, Mysuru

Directorate General of Supplies and Transport, New Delhi

Food Safety and Standards Authority of India, New Delhi

Mohan Meakin Limited, Ghaziabad

National Research Centre for Grapes, Pune

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Member Secretary Ms Lavika Singh Scientist 'B'/Assistant Director (Food and Agriculture), BIS this Page has been intertionally left blank

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected	

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bl <i>Telephone</i>	navan, 9 Bahadur Shah Zafar Marg, New Delhi 110002 es: 2323 0131, 2323 3375, 2323 9402	Website: www.bis.gov.in		
Regional	Offices:		1	Telephones
Central	: 601/A, Konnectus Tower -1, 6 th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002		{	2323 7617
Eastern	: 8 th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091			2367 0012 2320 9474
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg, Chandigarh 160019		{	265 9930
Southern	: C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113			2254 1442 2254 1216
Western	: Plot No. E-9, Road No8, MIDC, Andheri (East), Mumbai 400093		{	2821 8093

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