IS 1447 (Part 3) : 1992
(Reaffirmed 1997)

भारतीय मानक

पेट्रोलियम और इसके उत्पादों के नमूने लेने की पद्धतियां

भाग 3 उप-ठोस और ठोस पेट्रोलियम उत्पादों के नमूने लेने की पद्धतियां

(पहला पुनरीक्षण)

Indian Standard

PETROLEUM AND ITS PRODUCTS — METHODS OF SAMPLING

PART 3 METHOD OF SAMPLING OF SEMI-SOLID AND SOLID PETROLEUM PRODUCTS

(First Revision)

UDC 665·6/·7 : 620·113

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 3

FOREWORD

This Indian Standard (Part 3) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Test for Petroleum, Petrolum Products and Lubricants Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Sampling of petroleum and its products are taken for one or more of the following purposes:

a) For securing representative quantities from a part or from the whole of a quantity of material required for visual or laboratory examination or for preservation for records. Examination may be required to be made for determining physical and chemical characteristics in order to ensure:

   i) The average quality of the whole material, and

   ii) The extent of variation of qualities in different portions of the whole materials.

b) For determining the density and temperature of the material for the purpose of calculating the mass of a known volume of liquid or the volume of known mass.

It need not be emphasized that the most careful work in the laboratory or any quantity measurement may be rendered useless if the samples, upon which such work is based are not truly representative.

In order to implement the detailed provisions given in this sampling should have the necessary experience and skill. At all times scrupulous attention shall be given to detail. The methods covered in this standard shall be supplemented by judgement, skill and experience.

In reporting the results of a test or analysis made in accordance with standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

PETROLEUM AND ITS PRODUCTS — METHODSOF SAMPLING

PART 3 METHOD OF SAMPLING OF SEMI-SOLID AND SOLID PETROLEUM PRODUCTS

(First Revision)

1 SCOPE

This standard prescribes methods for obtaining representative samples of semi-solid and solid petroleum and its products for purpose of test or examination.

2 REFERENCES

The Indian Standard IS 436 (Part 2) : 1965 'Methods of sampling of coal and coke Part 2 : Sampling of coke' forms the necessary adjunct to this standard.

3 TERMINOLOGY

For the purpose of this standard, the following definitions shall apply.

3.1 **Boring Sample**

A sample obtained by collecting the chips made by boring holes with a ship auger from top to bottom of the material contained in a barrel, case, bag or cake.

3.2 Garb Sample

A sample obtained by collecting loose solids in equal quantities from each part or package or a shipment and in sufficient amount to be characteristic of all sizes and components.

3.3 Grease **Sample**

A sample obtained by scooping or dipping a quantity of soft or semi-liquid material, such as grease from a package in such a manner that the material on the scoop or dipper is representative of the material in the package.

4 APPARATUS

4.1 Sample Containers

Sample containers shall be of glass, plastic bottles or metal covered bottles or cans depending on the material to be handled. The container shall be wide mouthed with low necks for easy filling and decanting. The size of such containers will vary depending on the purpose for which sampling is being done.

4.2 **Cleaning Procedures**

All sample containers shall be absolutely clean and free of water, dirt, lint, washing compounds, naphtha or other solvents rust or oil. Before using the container, rinse it with petroleum hydrocarbon solvent or naphtha of similar volatility. Dry either by passing a amount of clean warm air through the container or by placing it in a hot dust-free cabinet at 40°C or higher. When dry, stopper or cap the container immediately.

4.3 **Sampling Apparatus**

Sampling apparatus is described in detail in each of the specific sampling procedure. Sampling apparatus shall be clean, dry and free of all substances that might contaminate the materials.

5 PRECAUTIONS AND INSTRUCTIONS

5.1 It is not possible to give directions for sampling, explicit enough to cover all cases. They shall be supplemented by judgement, skill and sampling experience particularly in regard to the type of samples to be taken. Extreme care and good judgement are necessary to ensure samples which represent general character and average condition of the material.

5.2 In addition to these general precuations, there are special precautions which shall be observed in sampling for certain tests. Some of these are covered in this standard, while the others are given in the relevant methods of test.

5.3 A sample shall not include material other than that to be sampled and shall not be altered, for example, by evaporation of volatile constituents or by oxidation in the process of sampling, storing or transporation.

5.4 Samples shall be taken by, or under the immediate supervision of a person of judgement, skill and experience in sampling.

5.5 The sampling apparatus and sample containers shall be dry and free from any contaminating substance.

5.6 During sampling operations, the material being sampled shall be protected as far as possible from the effects of wind and weather and the sample containers shall be closed immediately after the sample has been taken.

5.7 The operator engaged in sampling shall have clean hands, free from any material (unless it be the material being sampled). Clean gloves may be worn, but only when essential to protect the operator from health or other hazards.

5.8 Labelling Sample Container

Label the container immediately after a sample is obtained. Include the following information:

a) Date and time;
b) Name of the sample;
c) Name or number and owner of the vessel or container;
d) Brand and grade of material; and
e) Reference or identification number and other information necessary to be given.

6 SAMPLING PROCEDURE

6.1 Boring Sampling

6.1.1 *Application*

The boring sampling procedure is applicable for sampling waxes and soft solids in barrels, cases, bags or cakes when they cannot be melted and sampled as liquids.

6.1.2 *Apparatus*

6.1.2.1 *Ship auger*

Use a ship auger 19 mm in diameter similar to that shown in Fig. 1 and of sufficient length to pass through the material to be sampled.

6.1.2.2 *Sample container*

Use clean, wide mouth metal containers, glass jars with covers.

6.1.3 *Procedure*

Remove the heads or covers of barrels and cases. Open bags and wrappings of cakes. Remove any dirt, sticks, string or other foreign substances from the surfaces of the material. Bore three test holes through the body of the material — one at the centre, the other two half-way between the centre and edge of package on right and left sides respectively. If any foreign matter is removed from the interior of the material during boring operation include it as part of the borings. Put the three sets of borings in individual sample containers, label and deliver them to the laboratory.

6.1.4 *Laboratory Inspection*

If there are any visible differences in the samples, examine and test each set of boring at the laboratory otherwise combine the three sets of borings into one sample. If sub-division of the borings is desired, chill pulverize (if necessary), mix, and quarter the borings until reduced to desired amount.

6.2 Garb Sampling

6.2.1 *Application*

The garb sampling procedure is applicable for sampling all lumpy solids in bins, bunkers, freight cars, barrels, bags, boxes and conveyors. It is particularly applicable for the collection of green petroleum coke samples from railroad cars and for the preparation of such samples for laboratory analysis.

6.2.2 *Place of Sampling*

Petroleum coke may be sampled while being loaded into railroad cars from piles or after being loaded into railroad cars from coking drums.

6.2.3 *Apparatus*

6.2.3.1 *Sample container*, a polyethylene pail of approximately 9·5 l capacity.

6.2.3.2 *Scoop*, stainless steel or aluminium.

6.2.4 *Procedure*

6.2.4.1 *Sampling*

Lumpy solids are usually heterogenous and therefore are difficult to sample accurately. It is preferable to take samples during the unloading of trucks or during transit of the material by conveyors. From material in transit, obtain number of portions at frequent and regular intervals and combine them.

6.2.4.2 When sampling from wagons, use one of the following procedures:

a) *Being loaded from a pile* — Take a full scoope of sample at each of the five sampling points shown in Fig. 2 and deposit in the polyethylene pail. Cover the sample and deliver to the laboratory. Each sampling point shall be located equidistant from the sides of the railroad car.

b) *Wagons after direct loading from coking drums* — At any five of the sampling points shown in Fig. 3 take a full scoop of coke from about 0·3 m below the surface and deposit it in the polyethylene pail. Cover the sample and deliver to the laboratory.

FIG. 1 SHIP AUGER

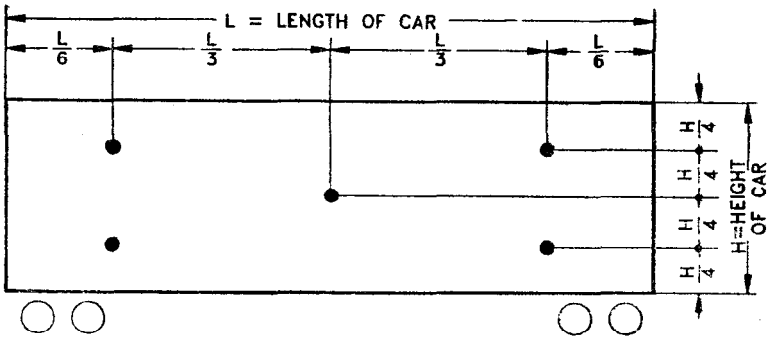

FIG. 2 LOCATION OF SAMPLING POINTS AT DIFFERENT LEVELS OF CAR

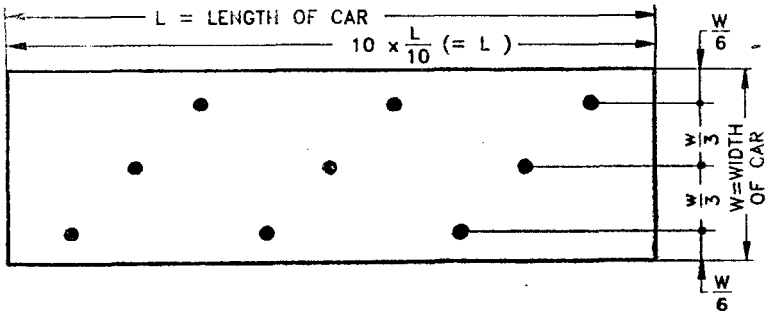

FIG. 3 LOCATION OF SAMPLING POINTS FROM EXPOSED SURFACE OF CAR

6.2.4.3 When sampling from conveyors, take one scoop for each 8 to 10 tons of coke transported. These samples may be handled separately, or composited after all samples representing the lot have been taken.

6.2.4.4 When sampling from bags, barrels, or boxes, obtain portions from a number of packages selected at random as shown in Table 1 or in accordance with the agreement between the purchaser and the supplier.

6.2.4.5 *Quartering*

Carefully mix the sample and reduce it in size to a convenient laboratory sample by the quartering procedure as given in IS 436 (Part 2) : 1965. Perform the quartering operation on a hard, clean surface, free from cracks, and protected from rain, snow, wind and sun. Avoid contamination with cinders, sand, chips from the floor, or any other material. Protect the sample from loss or gain of moisture or dust. Mix and spread the sample in a circular layer, and divide it into quadrants to form a representative reduced sample. If this sample is still too large for laboratory purposes, repeat the quartering operation. In this manner, the sample will finally be reduced to a representative, suitable size for laboratory purposes. Label and deliver the sample to the laboratory in suitable container.

6.3 Grease Sampling

6.3.1 *Application*

This method covers practices for obtaining samples representative of production lots or shipments of lubricating greases, or of soft waxes or soft bitumens similar to grease in consistency. The procedure is necessarily quite general to cover the wide variety of conditions encountered and may require modification to meet individual specification.

6.3.2 *Inspection*

If the material is a lubricating grease and inspection is made at the manufacturing plant, take samples from the finished shipping containers of each production batch or lot. Never take grease samples directly from grease kettle, cooling pans, tanks, or processing

equipment. Do not sample the grease until it has cooled to temperature not more than 8·3°C above that of the air surrounding the containers and until it has been in the finished containers for at least 12 h. When the containers for a production batch of grease are of different sizes, treat the grease in each size of container as a separate lot. When inspection is made at the place of delivery, obtain a sample from each shipment if a shipment consists of containers from more than one production batch (lot numbers), sample each such batch separately.

Table 1 Minimum Number of Packages to be Selected for Sampling

(*Clause* 6.2.4.4)

Number of Packages in a Lot	Number of Packages to be Sampled
(1)	(2)
1 to 3	all
4 to 64	4
65 to 125	5
126 to 216	6
217 to 343	7
344 to 512	8
513 to 729	9
730 to 1 000	10
1 001 to 1 331	11
1 332 to 1 728	12
1 729 to 2 197	13
2 198 to 2 744	14
2 745 to 3 375	15
3 376 to 4 096	16
4 097 to 4 913	17
4 914 to 5 832	18
5 833 to 6 859	19
6 860 to over	20

6.3.2.1 If the material being inspected is of grease-like consistency, but is not actually a lubricating grease, but some mixture of heavy hydrocarbons such as micro-crystalline waxes or soft bitumens, it will be permissible to take samples from pans, tanks, or other processing equipment, as well as from containers of the finished product. The grease sampling method shall be applicable to such stocks only if for some reason it is not possible to apply heat and convert the material into a true liquid.

6.3.3 *Sample Size*

Select containers at random from each lot or shipment to give the desired quantity specified in Table 2.

6.3.4 *Procedure*

6.3.4.1 *Inspection*

Examine the opened containers to determine whether the grease nearest the outer surfaces of the container with that in the centre, at least 152 mm below the top surface, for texture and consistency. When more than one container of a lot or shipment is opened, also compare the grease in the respective containers.

6.3.4.2 *Sampling*

If no marked difference in the grease is found, take one portion from the approximately centre and at sufficient quantity to provide a composite sample of the desired quantity (*see* Table 2). Withdraw portions with a clean scoop, large spoon or spatula and place them in a clean container. Very soft, semi-fluid greases may be sampled by dipping with a 0·4 kg can or suitable dipper. If any marked difference in the grease, two seperate samples of about 0·45 kg each one from the top surface adjacent to the wall, and the other from the centre of marked variation are noted between different containers of a lot or shipment, take separate samples of about 0·45 kg from each container. When more than one sample of a batch or shipment is taken because of lack of uniformity, send them to the laboratory as separte samples.

6.3.4.3 *Handling Samples*

If more than one portion is required to represent a lot or shipment of grease softer than 175 penetration, prepare a composite sample by mixing equal portions thoroughly. Use a large spoon or spatula in a clean container. Avoid vigorous mixing or working of air into the grease. As grease samples become partially 'worked' in being removed from containers, the procedure is not suitable for obtaining samples of grease softer than 175 penetration on which 'unworked' penetration is to be determined. For grease having a penetration less than 175, cut samples from the container with a knife in the form of blocks about 152 × 152 × 51 mm. If required, make unworked penetration tests on blocks as procured, and other inspection tests on grease cut from the blocks.

Table 2 Size of Grease Samples
(*Clause* 6.3.3)

Container	Lot or Shipment	Minimum Sample
(1)	(2)	(3)
Tubes or packages, less than 1 kg	All	Enough units for a 1 kg sample
1 kg cans	All	Three cans
5 or 10 kg cans	All	One can
Larger than 10 kg	Less than 10 000 kg	1 to 2 kg from one or more containers
Larger than 10 kg	10 000 to 50 000 kg	1 to 3 kg from two or more containers
Larger than 10 kg	More than 50 000 kg	1 to 3 kg from three or more containers

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 331 01 31, 331 13 75

Telegrams : Manaksanstha
(Common to all offices)

Regional Offices :	Telephone
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002	331 01 31 331 13 75
Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola, CALCUTTA 700054	37 84 99, 37 85 61 37 86 26, 37 86 62
Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036	53 38 43, 53 16 40 53 23 84
Southern : C. I. T. Campus, IV Cross Road, MADRAS 600113	235 02 16, 235 04 42 235 15 19, 235 23 15
Western : Manakalaya, E9 MIDC, Marol, Andheri (East), BOMBAY 400093	632 92 95, 632 78 58 632 78 91, 632 78 92

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