IS 3025-39 (1991): Methods of sampling and test (physical and chemical) for water and waste water, Part 39: Oil and grease [CHD 32: Environmental Protection and Waste Management]

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IS 3025 (Part 39) : 1991
(Reaffirmed 2003)

भारतीय मानक
जल और अपशिष्ट जल के लिए नमूने लेने और परीक्षण (भौतिक एवं रासायनिक) की पद्धतियाँ
भाग 39 तेल एवं ग्रीज़

(पहला पुनरीक्षण)

Indian Standard
METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER
PART 39 OIL AND GREASE

(First Revision)

Fifth Reprint SEPTEMBER 2008
(Including Amendment No. 1)

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

July 1991

Price Group 2

Environmental Protection Sectional Committee, CHD 12

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Environmental Protection Sectional Committee had been approved by the Chemical Division Council.

Oil and grease are primarily composed of fatty matter from animal or vegetable origin and petroleum hydrocarbons. Hence in the determination of oil and grease instead of quantifying a particular substance, a group of compounds with similar properties are determined quantitatively on the basis of their common solubility in trichlorotrifluoroethane. The methods covered in this standard are suitable for assessment of biological liquids, mineral hydrocarbons, industrial wastewater or treated effluents containing these materials. The methods shall not be applicable to low boiling fractions that volatalize at temperatures below 70°C. This standard supersedes **13** of IS 2488 (Part 1) : 1966 'Methods of sampling and test for industrial effluents, Part 1' and **59** of IS 3025 : 1964 'Methods of sampling and test (physical and chemical) for water used in industry'.

In the preparation of this standard, considerable assistance has been derived from the Standard 'Methods for the Examination of Water and Wastewater', published by the American Public Health Association, Washington, USA. 16th Edition, 1985.

In reporting the result of a test or analysis made in accordance with this 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*)'.

AMENDMENT NO. 1 FEBRUARY 2004
TO
IS 3025 (PART 39) : 1991 METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER

PART 39 OIL AND GREASE

(First Revision)

(*Foreword, para* 2, *line* 4) — Substitute 'Hexane' *for* 'trichlorotrifluoroethane' and also wherever it occurs in the standard.

(*Page* 1, *clause* **1.1**) — Insert the following at the end:

'Partition gravimetric method is applicable to the estimation of oil and grease from 4-100 mg/l, partition infra-red method from 0.5-100 mg/l, and Soxhlet extraction method from 20-200 mg/l in water and waste water.'

(*Page* 1, *clause* **5.4.2**) —Substitute '*Hexane*' *for* '*Trichlorotrifluoroethane*' and delete the text.

(CHD 32)

Reprography Unit, BIS, New Delhi, India

Indian Standard

METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER

PART 39 OIL AND GREASE

(First Revision)

1 SCOPE

1.1 This standand prescribes three methods for determination of oil and grease as follows:

a) Partition gravimetric method,

b) Partition infra-red method, and

c) Soxhlet extraction method.

1.2 This standard also prescribes the method for estimation of petroleum hydrocarbon content in the extracted oil and grease.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard :

IS No.	*Title*
3025 (Part 1) : 1987	Methods of sampling and test (physical and chemical) for water and wastewater : Part 1 Sampling (*first revision*)
7022 (Part 1) : 1973	Glossary of terms relating to water, sewage and industrial effluents, Part 1
7022 (Part 2) : 1979	Glossary of terms relating to water, sewage and industrial effluents, Part 2

3 TERMINOLOGY

3.1 For the purpose of this standard, definitions given in IS 7022 (Part 1) : 1973 and IS 7022 (Part 2) : 1979 shall apply, in addition to the following.

3.2 Oil and Grease

Any material recovered as a substance soluble in trichlorotrifluoroethane.

4 SAMPLING AND STORAGE

Sampling and storage shall be done as prescribed in IS 3025 (Part 1) : 1987. Collects about 1 litre of a representative sample in a wide mouth glass bottle that has been rinsed with the solvent (trichlorotrifluoroethane). Mark the sample level in the bottle. Acidify to *p*H 2 by adding concentrated hydrochloric acid. Collect a separate sample for oil and grease determination and do not subdivide in the laboratory. Loss of grease will occur on sampling equipment hence collection of a composite sample is not practical. Individual sample collected at prescribed time intervals should be analysed separately to obtain average concentration over an extended period.

5 PARTITION GRAVIMETRIC METHOD

5.1 Principle

Dissolved or emulsified oil and grease is extracted from water by trichlorotrifluoroethane and estimation is made gravimetrically.

5.2 Interferences

The solvent extracts are not only oil and grease but other organic substances also.

5.3 Apparatus

5.3.1 *Separating Funnel* — 1 litre capacity with teflon or equivalent stopcock.

5.3.2 *Distillation Flask* — 250-ml capacity.

5.3.3 *Water Bath*

5.3.4 *Filter Paper* — Whatman No. 40 or equivalent, 11 cm diameter.

5.4 Reagents

5.4.1 *Hydrochloric Acid* — 1 : 1.

5.4.2 *Trichlorotrifluoroethane*

1,1,2 trichloro-1,2,2 trifluoroethane; boiling point 47°C; there should not be any residue on evaporation of the solvent. Distil, if necessary.

5.4.3 *Sodium Sulphate* — anhydrous.

5.5 Procedure

Transfer the acidified sample to a separating funnel. Carefully rinse the sample bottle with

30 ml of trichlorotrifluoroethane and add the solvent washings to the separating funnel.

Shake vigorously for about 2 minutes. However, if it is suspected that a stable emulsion will form, shake gently for 5 to 10 minutes. Let the layers separate. Drain the solvent layer through a funnel containing solvent moistened filter paper into a clean, tared distillation flask. If a clear solvent layer cannot be obtained, add 1 g of sodium sulphate (Na_2SO_4) crystals to the filter paper cone and slowly drain emulsified solvent on to the crystals. Add more sodium sulphate, if necessary. Extract two more times with 30 ml of solvent each time, but first rinse the sample container with the solvent. Collect the extracts in a tared distillation flask and wash filter paper with an additional 10 to 20 ml of the solvent. Distil solvent from distillation flask over a water bath at 70°C. Quantitatively transfer the residue using a minimum quantity of solvent into a clean, tared, dried beaker. Place the beaker on water bath for 15 minutes at 70°C and evaporate off all the solvents. Cool the beaker in a desiccator for 30 minutes and weigh.

5.6 Calculation

$$\text{Oil and grease, mg/l} = \frac{M}{V} \times 1\,000$$

where

M = mass, in mg, of the residue; and

V = volume, in ml, of the sample taken.

6 PARTITION INFRA-RED METHOD

6.1 Principle

The extraction is identical as that for the gravimetric method but detection is made by infra-red spectrometry.

6.2 Apparatus

6.2.1 *Separating Funnel* — 1 litre capacity with teflon or equivalent stopcock.

6.2.2 *Infra-red Spectrophotometer* — Double beam, recording type.

6.2.3 *Cells* — Infra-red, silica.

6.2.4 *Filter Paper* — Whatman No. 40 or equivalent, 11 cm diameter.

6.3 Reagents

6.3.1 *Hydrochloric Acid* — See **5.4.1**.

6.3.2 *Trichlorotrifluoroethane* — See **5.4.2**.

6.3.3 *Sodium Sulphate* — See **5.4.3**.

6.3.4 *Reference Oil*

Prepare a mixture by volume, of 37·5 percent iso-octane, 37·5 percent hexadecane and 25 percent benzene. Store in a sealed container to prevent loss on evaporation.

6.4 Procedure

Follow the steps for acidification, extraction, etc, as given in **3.1** and **5.5**. Collect the combined extracts in a 100-ml volumetric flask and adjust the final volume to 100 ml with solvent. Prepare a stock solution of known oil by rapidly transferring 1 ml of oil or 0·5 to 1 g of grease to a clean tared 100-ml volumetric flask. Stopper the flask and weigh to the nearest milligram. Add solvent to dissolve and dilute to the mark. If the oil identity is unknown, use reference oil as standard. Using volumetric techniques, prepare a series of standards. Select a pair of matched near infra-red silica cells. A 1 cm path length cell is appropriate for a range of about 4 to 40 mg/l of oil and grease. Scan standards and samples from 3 200 cm^{-1} to 2 700 cm^{-1} with solvent, in reference beam and record results on absorbance paper. Measure absorbances of standards and samples by constructing a straight base line over the scan range and measuring absorbance of the peak maximum at 2 980 cm^{-1} and subtracting base line absorbance at that point. If the absorbance exceeds 0·8 for a sample, select a shorter path length or dilute as required. Use scans of standards to prepare a calibration curve.

6.5 Calculation

$$\text{Oil and grease, mg/l} = \frac{A \times 1\,000}{V}$$

where

A = mg of oil or grease in the extract as determined from calibration curve; and

V = volume, in ml, of the sample.

7 SOXHLET EXTRACTION METHOD

7.1 Principle

The material is subjected to extraction in a Sox-hlet apparatus with trichlorotrifluoroethane. The residue left after the evaporation of solvent is weighed. Compounds volatilized at or below 103°C will be lost when filter is dried. The method is empirical and reproducible results can be obtained only by adherence to all the details.

7.2 Apparatus

7.2.1 *Soxhlet Apparatus*

7.2.2 *Vacuum Pump*

7.2.3 *Buchner Funnel* — 12 cm diameter.

7.2.4 *Electric Heating Device*

7.2.5 *Paper Extraction Thimble*

7.2.6 *Filter Paper* — Whatman No. 40 or equivalent, 11 cm diameter.

7.2.7 ***Muslin Cloth Discs*** — 11 cm diameter.

7.3 Reagents

7.3.1 *Hydrochloric Acid* — 1 : 1.

7.3.2 *Trichlorotrifluoroethane* — See **5.4.2**.

7.3.3 ***Diatomaceous Silica Filter Aid Suspension*** — 10 g/l, distilled water.

7.4 Procedure

Prepare a filter consisting of a muslin cloth disc overlaid with filter paper. Wet the cloth and paper. Pass 100 ml of filter aid suspension through the prepared filter using vacuum and wash with 1 litre of distilled water. Filter the acidified sample ($pH \leqslant 2$). Apply vacuum until no more liquid sample passes through filter paper. Using forceps, transfer the filter paper to a watch glass. Add material adhering to edges of muslin cloth disc. Wipe sides and bottom of collecting vessel and Buchner funnel with filter paper soaked in solvent, taking care to remove all films caused by grease and collect all the solid material. Add pieces of filter paper to filter paper on watch glass. Roll all filter papers containing sample and put into a paper extraction thimble. Add any pieces of material remaining on watch glass. Wipe the watch glass with a filter paper soaked in solvent and place it in the thimble. Dry the filled thimble at 103°C for 30 minutes in an oven. Fill the thimble with glass wool or small glass beads. Weigh the extraction flask and extract oil and grease in a Soxhlet apparatus, using trichlorotrifluoroethane at a rate of 20 cycles per hour for four hours counting from first cycle. Distil solvent from extraction flask over a water bath maintained at 70°C. Place the flask on a water bath at 70°C for 15 minutes and draw air through it by vacuum for the final 1 minute. Cool in a desiccator for 30 minutes and weigh.

7.5 Calculation

$$\text{Oil andg rease, mg/l} = \frac{M \times 1\,000}{V}$$

where

M = mass, in mg, of the residue; and

V = volume, in ml, of the sample.

8 MODIFIED METHOD FOR HYDROCARBON

8.1 Principle

To estimate the petroleum hydrocarbon content of oil and grease this modified method is used. Silica gel has the ability to adsorb all constituents of oil and grease except hydrocarbons. The material not adsorbed by silica gel is designated as hydrocarbons by this method.

8.2 Interference

The method is not accurate for the reason that any compound other than hydrocarbon and fatty matter recovered by the solvent interfere by coming either in hydrocarbon part or in fatty matter.

8.3 Reagents

All reagents required for partition gravimetric method shall be applicable for this method also. In addition, silica gel of 75 to 150 micron size, dried at 110°C for 24 hours and stored in tightly sealed container is required.

8.4 Procedure

Follow the extraction procedure as given in **5.5**. To the extracted solution, add 3 to 4 g of silica gel. Stopper the container and stir gently for 10 minutes. Filter the solution through filter paper and wash silica gel and filter paper with 10 ml of solvent. Collect the filtrate. Distil off the solvent from the filtrate and weigh the residue as hydrocarbons.

8.5 Calculation

$$\text{Hydrocarbons, mg/l} = \frac{M \times 1\,000}{V}$$

where

M = mass, in mg, of residue; and

V = volume, in ml, of sample.

Bureau of Indian Standards

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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