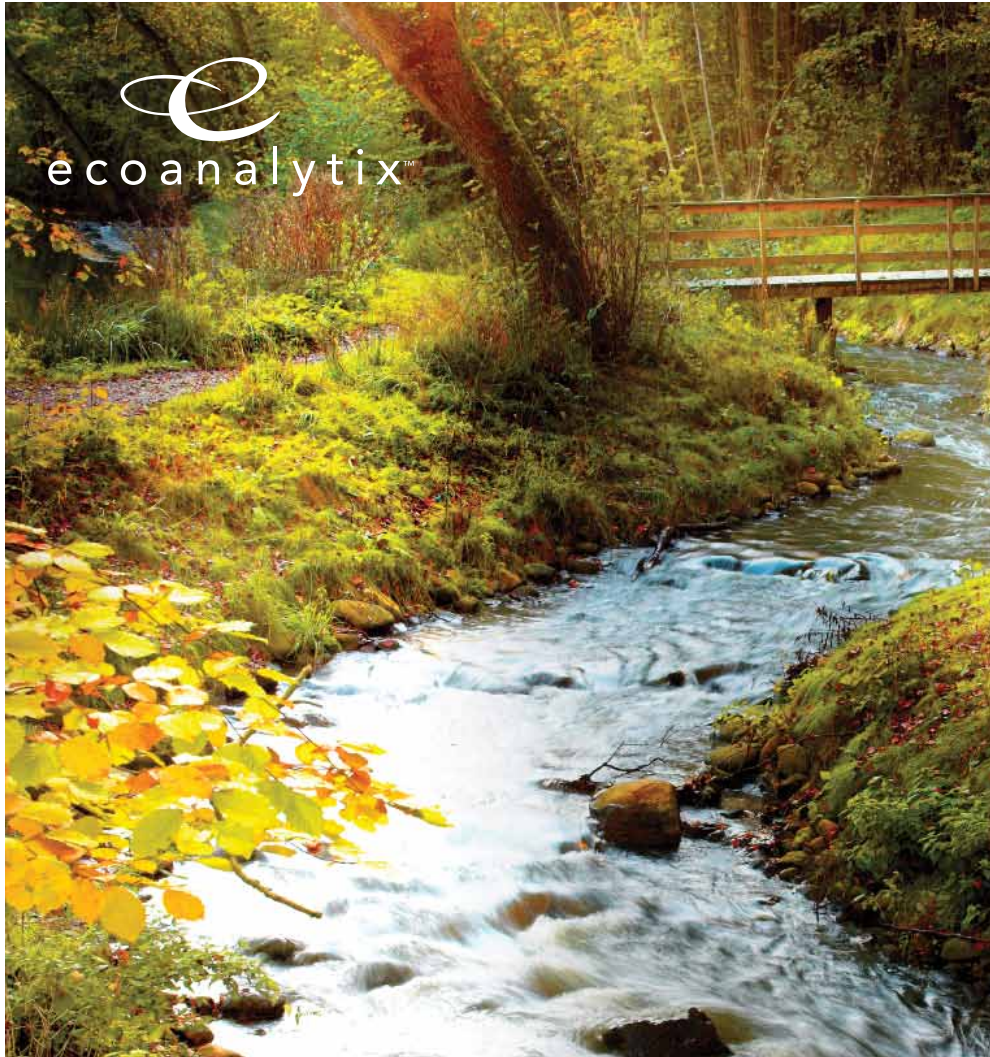


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Indian Standard 3025 Part 39 Determination of Oil and Grease in Water with a Mid Infrared Spectrophotometer

Introduction

The concentration of dispersed oil and grease (OG) is an important parameter for water quality and safety. Regulatory bodies worldwide set limits in order to control the amount of OG entering natural bodies of water or reservoirs through industrial discharges, and also to limit the amount present in drinking water. The Indian standards (IS 10500 specification) for discharge of OG into inland surface water and public sewers are 10 mg/L and 20 mg/L, respectively. However, the established method is based on hexane as an extraction solvent, which as a hydrocarbon-containing solvent, can interfere with the infrared analysis determination of OG.

This paper presents the method development for the analysis of OG in water using Mid-IR in accordance with standard method 'IS 3025 part 39' using carbon tetrachloride as an extraction solvent. Carbon tetrachloride is proved to be a suitable alternative to hexane and the method is shown to be sufficiently sensitive for monitoring OG discharges in surface waters or in the drinking water network as per the operative range specified in the method IS 3025 part 39.

Experimental

Instrumentation and operating conditions

The absorbance measurements were performed using the PerkinElmer® Spectrum™ 400 FT-IR/FT-NIR spectrophotometer in mid-IR mode and equipped with a DTGS detector (PerkinElmer, Inc. Shelton, CT USA), shown in Figure 1. Other instrument models with similar configurations¹ such as the Spectrum One or Spectrum 100 can also be used. The software used to acquire the spectra was Spectrum version 6.3. Spectra were collected in transmission mode using a glass cell with 10 mm pathlength. Spectra were acquired over the range 3200 - 2700 cm^{-1} at 4 cm^{-1} resolution with ~1 minute acquisition time, and ratioed against a spectrum of pure solvent. The peak maximum between 2930 and 2926 cm^{-1} was determined and used in the linear regression described below. A linear baseline fit through the points at 3100 and 2800 cm^{-1} was subtracted before measuring the peak height.

Apparatus and reagents

The reagents, chemicals, standards were prepared as described in the standard method BIS 3025 part 39. Reagents and standards used were of ACS grade.

Apparatus:

- Separating funnel – 1 liter capacity with stopcock.
- Analytical balance – Sartorius® five-decimal analytical balance.
- Pipette – Eppendorf® micropipettes.

Reagents:

- Hydrochloric acid – 1:1
- Carbon tetrachloride (spectroscopy grade): used as a solvent
- Sodium sulphate
- Calibration reference oil: mixture of 37.5% iso-octane, 37.5% hexadecane and 25% benzene.

Procedure

The calibration reference oil was prepared by mixing iso-octane, hexadecane and benzene in the ratio 3:3:2, and stored in a sealed container to avoid evaporative loss. The calibration stock solution was prepared by weighing about 1 gm of calibration reference oil into a clean and dry 100 mL volumetric flask and diluting up to the mark with solvent, i.e., carbon tetrachloride. From the calibration stock solution, a series of standard solutions were prepared using the volumetric techniques in the range 1-40 mg/L with 9 calibration points.

Sample Preparation: Samples were prepared as follows:

1. Acidification of 1 liter of sample using hydrochloric acid to pH 2.0.
2. Extraction of above sample with 30 mL of carbon tetrachloride three times (i.e., 1 x 3 times)
3. Filtration of extract through 10 g of sodium sulfate and dilution of combined collected extract up to 100 mL with solvent.
4. Measurement of the solution at the absorbance maximum near 2930 cm^{-1} .

Results and Discussion

Calibration – Linearity

Over the calibration range excellent linearity was observed; with a correlation coefficient (R^2) of 0.9997 (see Figure 2). A standard error of prediction of 4 mg/L was obtained.

Spike recovery studies

A recovery study has been performed at 6 mg/L concentration in three replicates. The results are summarized in Table 1. As seen in the table, the recoveries are excellent, ranging from 90 to 95 percent. This indicates that the solvent extraction recovers nearly all of the OG and introduces only a small negative bias to the reported result.

Table 1: Replicate spike recoveries.

Sample	% Recovery
Sample 1 (6.51 mg/L)	92.3
Sample 2 (6.23 mg/L)	94.3
Sample 3 (5.54 mg/L)	92.9

Precision

A precision study has been carried out at 7.0 mg/L concentration in six replicates. Standard deviation is found to be 0.062. This is well within the expected precision for this technique.



Figure 1. The Frontier FT-IR Spectrometer.

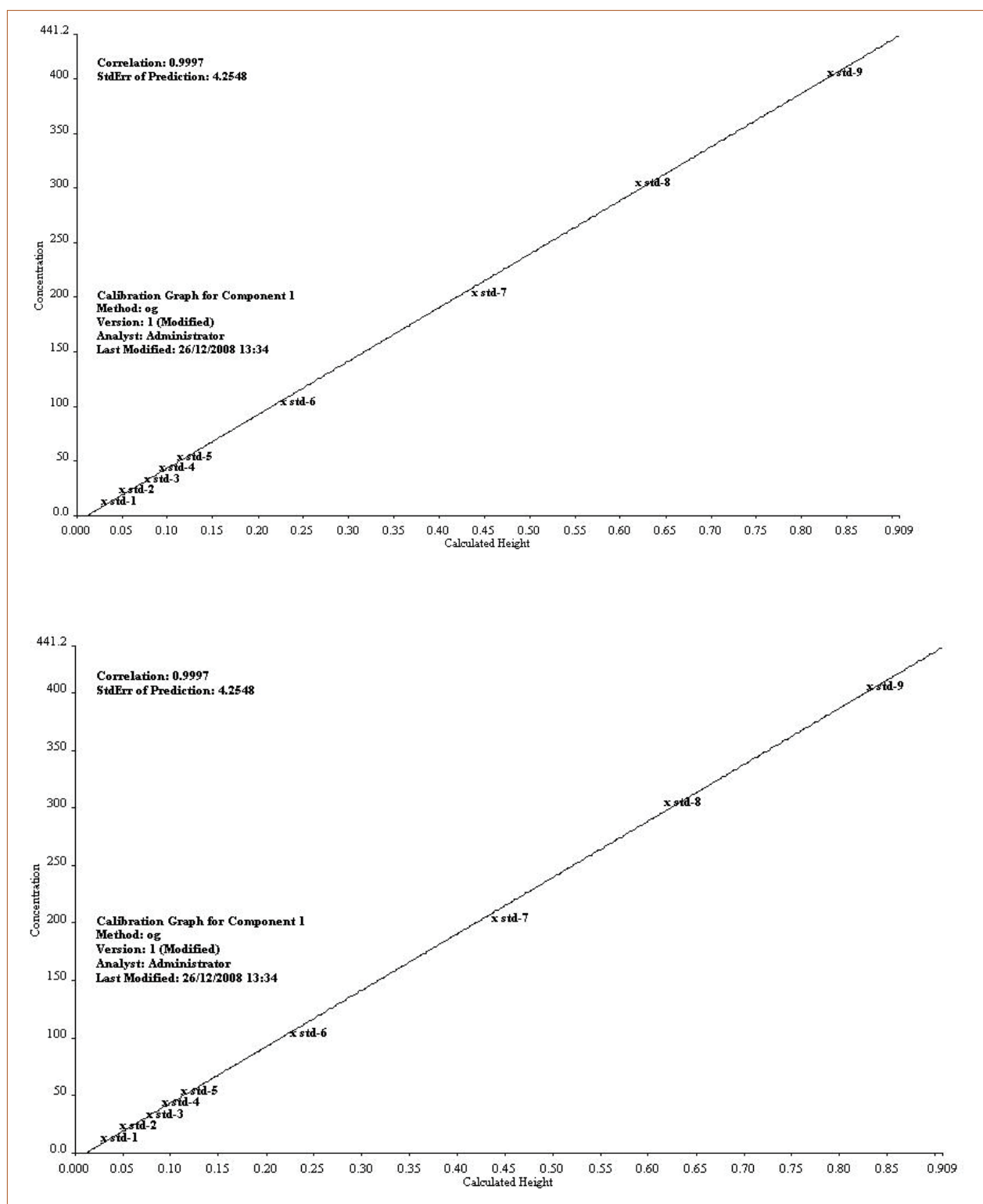


Figure 2. Spectra of standards and calibration graph.

Table 2: Replicate precision data.

Sample	Concentration (mg/L)
Sample 1	6.92
Sample 2	6.95
Sample 3	6.85
Sample 4	6.81
Sample 5	6.81
Sample 6	6.91
Standard Deviation	0.06

Conclusion

The Spectrum 400 Mid FT-IR/FT-NIR, Spectrum 100 or Spectrum One series of spectrophotometers can be utilized successfully for determination of OG in water samples as they show linearity with good correlation coefficient up to concentration of 1 mg/L, good percent recovery and good precision.

The standard method IS 3025 part 39 works well with carbon tetrachloride as an extraction solvent. The working range specified in the method is 4 mg/L to 40 mg/L but concentrations between 1 mg/L to 40 mg/L may be satisfactorily determined using this method.

References

1. IS 3025 (part 39): 1991, Reaffirmed 2003. (Indian standard Methods of sampling and test for water and waste water – Oil and Grease)
2. IS 10500: Indian standard specification for drinking water.