Infrared Spectroscopy Applications for Determining Oil Content in Water

Abstract:

The content of petroleum pollutants in environmental water is one of the indicators reflecting water quality. This article employs a three-wavelength quantitative method to test the oil content in water, which offers convenience in sample testing and ensures accurate data.

The petroleum substances in environmental water originate from industrial wastewater and domestic sewage pollution. These oil substances form an oil film on the water surface, affecting the gas exchange between air and water. Additionally, when oil is dispersed in water, adsorbed onto particles, or exists in an emulsified state, its decomposition by microorganisms consumes dissolved oxygen in the water, leading to potential deterioration in water.

Mineral oil is a mixture composed of alkanes, cycloalkanes, and aromatic hydrocarbons. This article refers to the "Infrared Spectrophotometric Method for Determination of Oil Content in Water Quality" and employs a three-wavelength infrared spectroscopy method to measure surface water. The obtained results are accurate, avoiding the use of "standard oil."

Keywords:

Infrared spectroscopy, Oil in water, Quantitative detection

1. Principle:

The oil substances in water are a mixture composed of alkanes, cycloalkanes, and aromatic hydrocarbons. They can be extracted using carbon tetrachloride to determine the total extract. Subsequently, the extracted liquid is adsorbed with magnesium silicate to remove polar substances like animal and plant oils. The petroleum and animal/plant oil infrared spectra exhibit absorption at 2930 cm-1, 2960 cm-1, or 3030 cm-1. The content can be calculated based on the absorbance values at these three wavenumbers.

2. Experimental Conditions:

Instrument and Accessories:

FTIR-7600S Fourier Transform Infrared Spectrometer

1cm Quartz Cuvette

Reagents:

Carbon tetrachloride (CCl4)

n-Hexadecane [CH3(CH2)14CH3] Analytical Grade

Squalane (2,6,10,14-Tetramethylpentadecane) Analytical Grade

Toluene (C6H5CH3) Analytical Grade

Anhydrous sodium sulfate (Na2SO4) Analytical Grade

Sodium chloride (NaCl) Analytical Grade

Hydrochloric acid (HCl) Analytical Grade

3. Sample Pretreatment:

Transfer the entire water sample into a separatory funnel, use a 20ml carbon tetrachloride-rinsed sampling bottle, combine the rinsing liquid into the separatory funnel, adjust the pH to ≤2, add 20g sodium chloride, vigorously shake for 2 minutes, then allow to settle. Pass the extract through a glass sand core funnel with a 10mm anhydrous sodium sulfate bed, collect the filtrate in a volumetric flask. Take another 20ml carbon tetrachloride for re-extraction, wash the glass sand core funnel adequately with carbon tetrachloride, and combine the extract and washing liquid into the volumetric flask. Fill up to the mark with carbon tetrachloride and shake well.

4. Measurement Results:

1) Determination of Calibration Coefficients:

Using carbon tetrachloride as the solvent, prepare solutions with concentrations of 100mg/L n-hexadecane, 100mg/L squalane, and 400mg/L toluene. Use carbon tetrachloride as a reference solution. Employ 10mm × 10mm quartz cuvettes to measure the absorbance (A2930, A2960, A3030) of these three solutions at 2930cm-1, 2960cm-1, and 3030cm-1, respectively. The absorbance of these three solutions at the mentioned wavenumbers should satisfy the following formula:

$$C=X \cdot A2930+Y \cdot A2960+Z \ (A3030- A2930/F)$$

In the formula:

C - Content of compounds in the extraction solvent, mg/L;

A2930, A2960, A3030 - Absorbance values obtained at the corresponding wavenumbers;

X, Y, Z - Calibration coefficients corresponding to the absorbance of each C-H bond;

F - Correction factor for hydrocarbons to aromatics, i.e., the ratio of the absorbance of n-hexadecane at 2930cm-1 to 3030cm-1;

For n-hexadecane (H) and squalane (P), since their aromatic hydrocarbon content is zero, i.e., A3030 - A2930/F = 0, then

F = A2930(H)/A3030(H);

C(H)=X×A2930(H)+Y×A2960(H)；

C(P)=X×A2930(P)+Y×A2960(P)；

From this, the values of X, Y, and F can be obtained.

For toluene (T), the equation is: C(T) = X•A2930(T) + Y•A2960(T) + Z [A3030(T) - A2930/F], from which the value of Z can be derived. After calculation, the correction coefficients X, Y, Z, and F are 126.6, 242.5, 1575, and 63, respectively.

2) Verification of Calibration Coefficients:

Prepare a hydrocarbon mixture by accurately measuring n-hexadecane, squalane, and toluene in a volumetric ratio of 5:3:1. Prepare a series of solutions with different concentrations by accurately measuring the appropriate amount of the hydrocarbon mixture. Measure their absorbance at A2930, A2960, and A3030 (refer to Figure 1) and calculate the concentration of the hydrocarbon mixture and the recovery rate (see Table 1 for results).

(1) Test Spectra
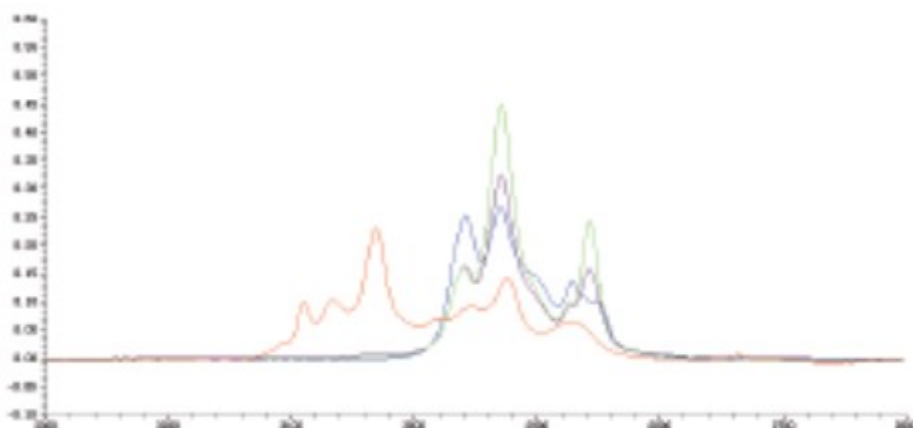
Note:

———— represents the spectrum of the toluene solution.

———— represents the spectrum of the n-hexadecane solution.

———— represents the spectrum of the squalane solution.

———— represents the spectrum of the hydrocarbon mixture solution.

(2) Recovery Rate

Table 1: Recovery Rate of Hydrocarbon Mixture

No.	Actual Concentration of the Hydrocarbon Mixture（mg/L）	Measured Concentration of the Hydrocarbon Mixture（mg/L）	Recovery Rate (%)	Average Recovery Rate（%）
1	94.5	91.3	96.6	98.65
		95.3	100.8	
2	105.0	103.6	98.6	
		103.5	98.6	

5. Summary:

When using the "three-wavelength" infrared spectroscopy method to measure oil content in water, it effectively avoids measurement errors caused by abrupt changes in the relative content of characteristic functional groups in the samples. Comparing the concentration of the hydrocarbon mixture calculated according to national standards with the actual values, the recovery rate meets environmental detection requirements, making it an ideal method for determining oil substances.