HJ

ENVIRONMENTAL PROTECTION STANDARD

OF THE PEOPLE’S REPUBLIC OF CHINA

HJ 637-2018

Replacing HJ 637-2012

Water quality - Determination of petroleum, animal fats and vegetable oils - Infrared spectrophotometry

水质 石油类和动植物油类的测定

红外分光光度法

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Water quality - Determination of petroleum, animal fats and vegetable oils - Infrared spectrophotometry

Warning: The tetrachloroethylene used in the experiment is harmful to human health. The preparation of standard solution, the preparation of sample, the determination process shall be carried out in a fume hood. During operation, it shall wear protective equipment as required to avoid contact with skin and clothing.

1 Scope

This standard specifies the infrared spectrophotometric method for the determination of petroleum, animal fats and vegetable oils in water.

This standard applies to the determination of petroleum, animal fats and vegetable oils in industrial and domestic sewage.

When the volume of sample is 500 ml, the volume of the extract is 50 ml, the 4 cm quartz cuvette is used, the detection limit of method is 0.06 mg/L, the lower limit of determination is 0.24 mg/L.

2 Normative references

The contents of this standard refer to the following documents or their terms. For undated references, the valid version applies to this standard.

HJ/T 91 Technical specifications requirements for monitoring of surface water and waste water

3 Terms and definitions

The following terms and definitions apply to this standard.

3.1

Oil and grease

It refers to, under the conditions of pH ≤ 2, the substances that can be extracted by tetrachloroethylene and have characteristic absorption at wavenumbers of 2930 cm^{-1}, 2960 cm^{-1}, 3030 cm^{-1}, mainly including petroleum, animal fats and vegetable oils.

5.4 n-hexadecane ($C_{16}H_{34}$): Chromatographical pure.

5.5 Isooctane (C_8H_{18}): Chromatographical pure.

5.6 Benzene (C_6H_6): Chromatographical pure.

5.7 Anhydrous sodium sulfate (Na_2SO_4).

PLACE it in a muffle furnace to heat it at 550 °C for 4 h. After it is slightly cooled, CONTAIN it into a ground glass bottle. PLACE it in a desiccator for storage.

5.8 Magnesium silicate ($MgSiO_3$): 150 μm ~ 250 μm (100 mesh ~ 60 mesh).

TAKE magnesium silicate and PLACE it in a porcelain evaporating dish. PLACE it in a muffle furnace at 550 °C to heat it for 4 h. After it is slightly cooled, TRANSFER it into a desiccator to cool it to room temperature. WEIGH an appropriate amount of magnesium silicate in a ground glass bottle. According to the mass of magnesium silicate, ADD an appropriate amount of distilled water in a ratio of 6% (m/m). CAP it tightly and SHAKE it thoroughly. LET it be standing for 12 h before use. PRESERVE it in a glass bottle.

5.9 Glass wool

Before use, USE the tetrachloroethylene (5.3) to soak and rinse the glass wool. DRY it naturally to prepare for use.

5.10 Standard stock solution of n-hexadecane: $\rho \approx 10000$ mg/L.

WEIGH 1.0 g (accurate to 0.1 mg) of n-hexadecane (5.4) in a 100 ml volumetric flask. USE tetrachloroethylene (5.3) to make its volume reach to the mark. SHAKE it uniformly. STORE it at 0 °C ~ 4 °C, which can be preserved in the dark for 1 year.

5.11 Standard use solution of n-hexadecane: $\rho = 1000$ mg/L.

USE tetrachloroethylene (5.3) to dilute the standard stock solution of n-hexadecane (5.10) to the volume in a 100 ml volumetric flask.

5.12 Standard stock solution of isooctane: $\rho \approx 10000$ mg/L.

WEIGH 1.0 g (accurate to 0.1 mg) of isooctane (5.5) in a 100 ml volumetric flask. USE tetrachloroethylene (5.3) to make its volume reach to the mark. SHAKE it uniformly. STORE it at 0 °C ~ 4 °C, which can be preserved in the dark for 1 year.

5.13 Standard use solution of isooctane: $\rho = 1000$ mg/L.

USE tetrachloroethylene (5.3) to dilute the standard stock solution of isooctane (5.12) to the volume in a 100 ml volumetric flask.

6.6 Colorimetric tube: 25 ml, 50 ml, ground, with a stopper.

6.7 Separating funnel: 1000 ml with Teflon cock.

6.8 Measuring cylinder: 1000 ml.

6.9 Commonly used laboratory utensils and equipment.

7 Samples

7.1 Collection of sample

With reference to the relevant provisions of HJ/T 91, USE the sampling bottle (6.3) to collect about 500 ml of water sample. ADD hydrochloric acid solution (5.2) to acid it to pH ≤ 2.

7.2 Preservation of samples

If the sample cannot be determined within 24 h, it shall be stored at 0 °C ~ 4 °C and determined within 3 days.

7.3 Preparation of samples

7.3.1 Preparation of oil and grease samples

TRANSFER the sample into a 1000 ml separatory funnel (6.7). MEASURE 50 ml of tetrachloroethylene (5.3) to rinse the sample bottle. TRANSFER all the rinsing solution into the separatory funnel (6.7). SHAKE it completely for 2 min. Frequently OPEN the cock for exhaust. LET it be standing for stratification. USE tweezers to take glass wool (5.9) and place it in a glass funnel (6.4). TAKE an appropriate amount of anhydrous sodium sulfate (5.7) to pave above the glass wool. OPEN the cock of the separatory funnel. MAKE the lower layer of organic phase extract pass the glass funnel (6.4) which contains anhydrous sodium sulfate (5.7) into a 50 ml colorimetric tube (6.6). USE an appropriate amount of tetrachloroethylene (5.3) to wet and rinse the glass funnel (6.4). COLLECT the rinsing solution into the extract. USE tetrachloroethylene (5.3) to make its volume reach to the mark. TRANSFER all the upper layer of aqueous phase into the measuring cylinder. MEASURE the volume of sample. TAKE record.

Note: It may use automatic extraction instead of manual extraction. It may use aluminum silicate filter cotton instead of glass wool. Before use, the aluminum silicate filter cotton shall be placed in a muffle furnace at 550 °C to heat it for 4 h, cooled before use.

7.3.2 Preparation of petroleum specimens

7.3.2.1 Oscillation adsorption method

solution which is measured under the corresponding wavenumber;

$A_{2930}(I)$, $A_{2960}(I)$, $A_{3030}(I)$ - The absorbance of the isooctane standard solution which is measured under the corresponding wavenumber;

$A_{2930}(B)$, $A_{2960}(B)$, $A_{3030}(B)$ - The absorbance of the benzene standard solution which is measured under the corresponding wavenumber.

Note 1: It may use pristane instead of isooctane, use toluene instead of benzene to determine the correction factor by the use of the same method.

Note 2: At the time of exit-factory of infrared spectrophotometer or infrared oil detector, if the correction factor is set, it can directly check the correction factor according to 11.3.1.

8.2 Determination

8.2.1 Determination of oil and grease

TRANSFER the extract (7.3.1) into a 4 cm quartz cuvette. USE the tetrachloroethylene as a reference. MEASURE the absorbance A_{2930}, A_{2960}, A_{3030} at 2930 cm^{-1}, 2960 cm^{-1}, 3030 cm^{-1}, respectively.

8.2.2 Determination of petroleum

TRANSFER the extract (7.3.2) which was adsorbed by magnesium silicate into a 4 cm quartz cuvette. USE the tetrachloroethylene as a reference. MEASURE the absorbance A_{2930}, A_{2960}, A_{3030} at 2930 cm^{-1}, 2960 cm^{-1}, 3030 cm^{-1}, respectively.

8.3 Determination of blank specimen

According to the procedures same as the determination of specimen (8.2), DETERMINE the blank specimen (7.4).

9 Calculation and representation of results

9.1 Calculation

9.1.1 Calculation of concentration of oil and grease or petroleum

The concentration of oil and grease or petroleum in the sample is calculated according to formula (6):

$$\rho=\left[X\cdot A_{2930}+Y\cdot A_{2960}+Z\cdot\left(A_{3030}-\frac{A_{2930}}{F}\right)\right]\cdot\frac{V_0\cdot D}{V_w}-\rho_0 \qquad (6)$$

retaining up to 3 significant digits.

10 Precision and accuracy

10.1 Precision

Six laboratories perform six repeated determinations on petroleum samples which have a prepared concentration of 0.20 mg/L, 1.00 mg/L, 4.00 mg/L, respectively. The inner-laboratory relative standard deviations are 2.4% ~ 13%, 0.8% ~ 4.7%, 0.8% ~ 3.6%. The inter-laboratory relative standard deviations are: 20%, 9.7%, and 5.9%, respectively. The repeatability limit r is 0.05 mg/L, 0.08 mg/L, 0.26 mg/L, respectively. The reproducibility limit R is 0.13 mg/L, 0.26 mg/L, 0.65 mg/L, respectively.

Six laboratories perform six repeated determinations on two different types of actual samples which contain industrial waste water that has a concentration of 0.94 mg/L and 1.84 mg/L and domestic sewage. The inner-laboratory relative standard deviation is 1.1% ~ 4.7% and 1.0% ~ 5.2%, respectively. The inter-laboratory relative standard deviation is 6.2% and 9.1%, respectively. The repeatability limit r is 0.09 mg/L and 0.17 mg/L, respectively. The reproducibility limits R is 0.18 mg/L and 0.50 mg/L, respectively.

10.2 Accuracy

Six laboratories perform the spiked analytical determination for the blank samples, respectively, the spiked amount is 0.10 mg, 0.50 mg, 2.00 mg, respectively. MAKE six repeated determinations. The spiked recovery rate ranges 75% ~ 138%, 78% ~ 104%, 81% ~ 95%, respectively. The final value of the spiked recovery rate is 111% ± 44%, 94% ± 18%, 91% ± 11%, respectively.

Six laboratories determine the spiked recovery rate of two different types of actual water samples of industrial wastewater and domestic sewage, respectively. The spiked amount is 0.50 mg and 1.00 mg, respectively. MAKE six repeated determinations. The spiked recovery rate ranges 84% ~ 98%, 81% ~ 100%, respectively. The final values of the spiked recovery rate are: 92% ± 11%, 91% ± 15%, respectively.

11 Quality assurance and quality control

11.1 Quality inspection of tetrachloroethylene

Tetrachloroethylene shall be stored in the dark. Before use, it shall follow (5.3) to inspect and determine the quality of tetrachloroethylene, to confirm that is complies with requirements before use.

------ The End ------