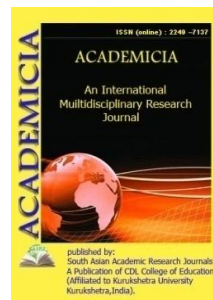


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SPECIAL METHODS FOR STUDYING THE CHEMICAL COMPOSITION OF ENGINE OILS

Imomova M.Yo*; Abduganiev B.Yo; Turdiboev A. Kh*****

*Fergana State University,
UZBEKISTAN

**Central Customs Laboratory of the Republic of UZBEKISTAN

***Fergana State University,
UZBEKISTAN

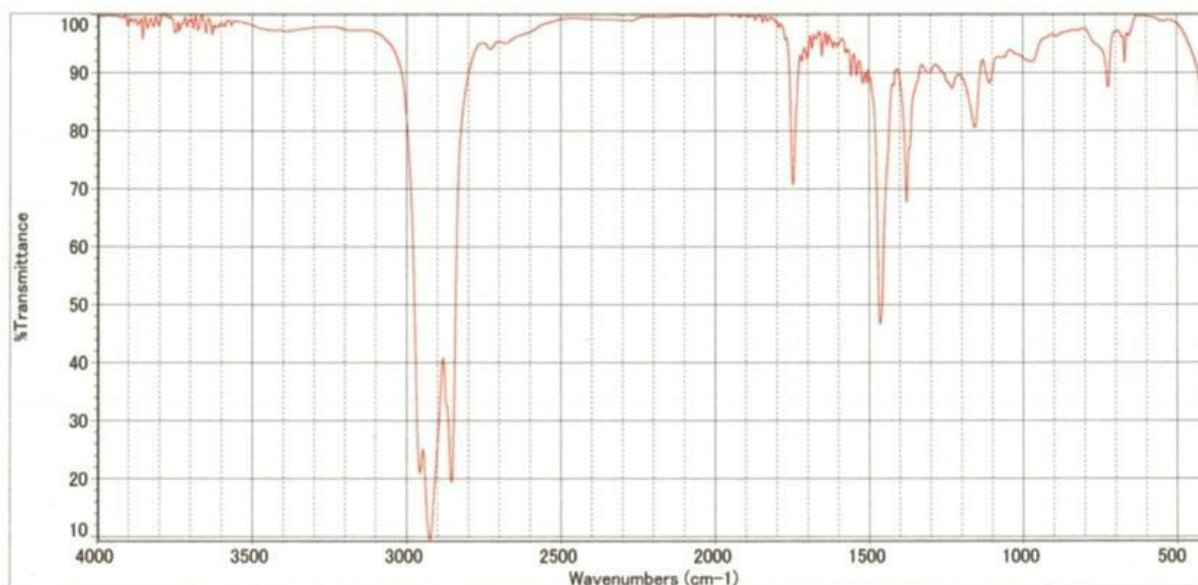
ABSTRACT

Scientific research in the direction of differentiating the belonging of motor oils and lubricants to synthetic, poly- α -olefin or mineral containing from 20 to 70 carbon atoms, or semisynthetic consisting of their mixtures, are the main ones, since the prevention of incorrect classification by the code of one type of engine oil and lubricants to others, it is conditioned by the introduction of customs control methods based on world-class achievements in the field of science and technology, as a result of which it becomes clear that only research in this area will help to solve the above-mentioned problems.

KEYWORDS: *Base Oil, Lubricating Oil, Mineral Oil, Synthetic Oil.*

INTRODUCTION

Our research paper presents data obtained by using an effective method for determining the amount of base oil in an engine and lubricating oils. This method consists of several steps:

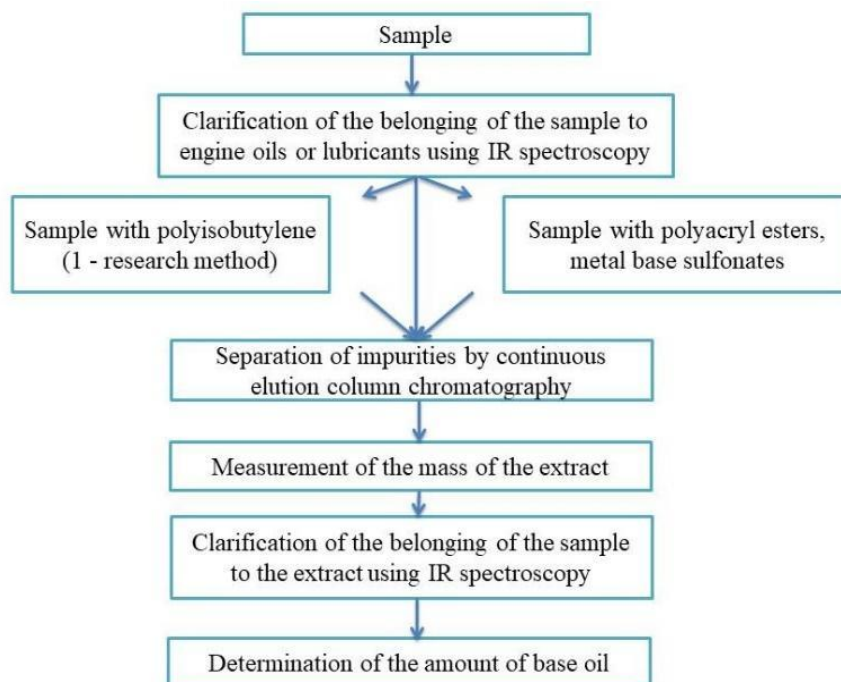


Scheme for determining the amount of base oil in engine and lubricating oils

THE MAIN FINDINGS AND RESULTS

Due to the fact that this study is based on IR spectroscopy, in order to achieve the accuracy and transparency of the results, a program for metrological certification of equipment was developed, as well as an integrated approach to determining the chemical composition of engine oil in the described sequence.

For the analysis of engine oils, two types of samples with a mass of 100 g were taken. In order to make sure that the samples were engine oil, the IR spectra of the samples were taken. In some cases, knowing the frequency of absorption of the bands in the IR spectrum, one can draw conclusions about the functional groups of a substance, thus finding which classes they belong to. The IR spectrum of saturated and unsaturated hydrocarbons is observed only in absorption bands caused by stretching and deformation vibrations of the CH bond.



Synthetic oil sample

Pic. – 6. IR spectrum of engine oil.

However, it is impossible to obtain information on the number of methyl and methylene groups in the hydrocarbon chain from IR spectra. Bands formed by asymmetric and symmetric stretching vibrations of methyl (-CH₃) and methylene (-CH₂) groups in the range 2850-2950 cm⁻¹. Based on this case, it was concluded that the analyzed sample consists of hydrocarbons. Compared to the spectra in the Image Sample Library, the exact match of the spectra of the unknown substance indicates that the fluid being tested is engine oil.

According to the IR spectra, if it was assumed that the sample contains polyisobutylene, then it is necessary to carry out the pretreatment procedure as described in the first way.

The first method of preliminary sample preparation.

Took the first sample with an accuracy of 5 g and placed in a 200 ml Erlenmeyer flask, mixing, added 20 ml of petroleum ether. While constantly mixing, 20 ml of acetone was slowly added to the mixture. The flask was connected to a refrigerator, inserted into a condenser and heated for 10 minutes in a hot water bath. We waited until the liquid above the sediment reaches a transparent state. The decanted liquid content in the flask was collected in a 500 ml glass beaker. The precipitate in the flask was washed three times with a mixture of 20 ml of petroleum ether and acetone (1: 1), and each time the solution from the flask was poured into a 500 ml beaker. The precipitate was washed twice more with 20 ml of petroleum ether, filtered and added to the mixture in a 500 ml beaker. The newly obtained 200 ml Erlenmeyer flask was pre-weighed. The collected extract in a 500 ml glass beaker was poured into a pre-weighed 200 ml Erlenmeyer flask and the solvent was evaporated using a rotary evaporator. The remaining residue was dried

at 100 ± 2 ° C in an oven and weighed on an analytical balance. The percentage of residual oil other than polyisobutylene is calculated using the following formula:

$$X \% = \frac{B_o}{A} * 100 \quad (1)$$

X is the residual part of the oil except for polyisobutylene (%);

A is the weight of the sample (g);

B_o is the residual part of the oil except for polyisobutylene (g).

All processes were performed due to the fact that polyisobutylene does not dissolve in acetone and petroleum ether, and the rest of the oil sample is highly soluble in these solvents. If the IR spectra suggest that the sample contains polyacrylic esters and / or metal bases of sulfonates, preliminary preparation was carried out in accordance with the method described in method two.

Second way of preliminary sample preparation.

2 g of the sample was placed in a rubber membrane. The top of the rubber membrane was attached to the aeration clamp. The attached membrane was placed in the Soxhlet extractor column in such a way that the membrane was above the level of the extractor liquid.

120 ml of petroleum ether was poured into a flask and combined with an extractor. To ensure circulation, the immersed flask was connected to a reflux condenser, then placed in a water bath and left to boil for 10 hours. The dialysis rate of the water bath was adjusted from 6 to 8 cycles per hour. The purified ether in the flask was used as a test material.

At the same time, metal-based polyacrylic esters and sulfonates are easily adsorbed.

After dialysis, the flask was separated from the Soxhlet extractor and the petroleum ether solution was removed as a test material for further processes. If all of the aforementioned additives are assumed to be present in the sample based on IR spectra, then pretreatment should be performed first with the first method and then with the procedure shown in method two.

The procedures shown in the first and second methods were not implemented due to the absence of polyacrylic ethers, sulfonates based on metals and polyisobutylene functional groups in the sample according to IR spectra.

Method for quantitative analysis of motor oils using continuous elution chromatography.

Preparing a Soxhlet column.

50 g of weighed dry silica gel was loaded onto a Soxhlet extractor column.

The upper and lower parts of the Soxhlet column were covered with cotton wool, and the adsorbent was washed with a large amount of petroleum ether. The washed petroleum ether was removed. In a pre-weighed flask was poured 100 ml of petroleum ether and connected to the above Soxhlet column. The sample was slightly dissolved in petroleum ether. The flask was connected to a refrigerator and placed in a water bath. The temperature was set such that the rate of petroleum ether outflowing from the bottom of the column was 100 to 120 drops per minute. Thus, the solvent was circulated for 16 hours. The residue in the flask was dried in a water bath at 100 ± 2 °C until the residual mass became constant.

Mineral oil

Pic. – 7. IR spectrum of motor oil extract.

Determination of volatile substances.

This method was carried out due to the presence of volatiles in base oils in the determination of the quantitative analysis of engine oils using the first method of preliminary sample preparation and the method of continuous elution chromatography.

In parallel with the process of quantitative analysis of engine oil using the method of continuous elution chromatography, 2 g of the sample placed in a Soxhlet column was weighed and dried in an oven at a temperature of 100 ± 2 °C until the mass became constant.

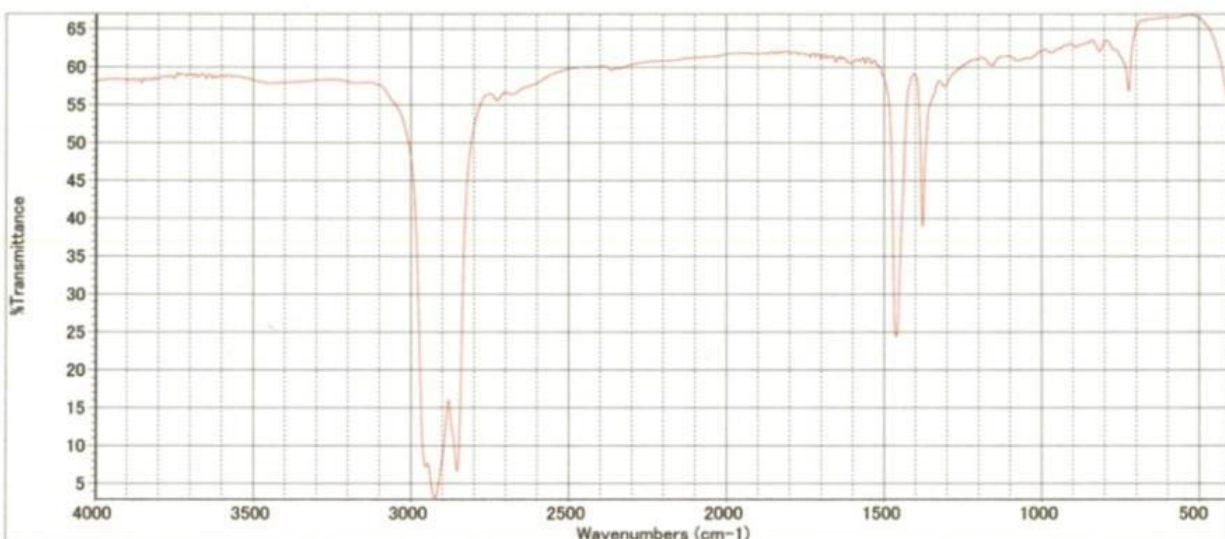
The losses were calculated using the following formula:

$$U \% = \frac{B}{G_o} * 100 \quad (2)$$

U is the amount of volatile substances%;

B is the amount of loss (g);

G_o is the residual part of the test sample (g).



The degree of dissolution of base oils with continuous elution with petroleum ether for 16 hours (%). Calculation of base oil content in engine oil.

The mineral content in engine oil samples is determined using the following formula.

On the occasion, obtain T by the first method of preliminary sample preparation

$$N \% = P \% \times x + \frac{T}{S_o} \text{ volatile substance \% (3)}$$

N is the base component of mineral oil%;

P is the amount after removal of poly-isobutylene (%);

T is the amount of a substance according to the analysis of engine oils using a continuous elution chromatography method (g);

S_o is the collected volume of the test sample (g).

As a result, it was found that the first sample of engine oil = 95.51% - 2710, and the second sample of engine oil = 45.1% - 3403.