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RESEARCH AND IDENTIFICATION AND CLASSIFICATION METHODS OF VEGETABLE OILS

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Abstract

The article presents methods and results for determining the quality of olive, sunflower and corn oil. Modern organoleptic and physicochemical methods for determining the quality of fatty acids have been developed.

Keywords: *olive, sunflower and corn oil, methods of analysis, commodity nomenclature of foreign economic activity, methods of examination, identification*

Introduction

One of the most pressing issues in international economic relations and customs practice is the correct determination of the code numbers of the Commodity Nomenclature of Foreign Economic Activity (CN FEA) of all export-import goods. As a result of the analysis of goods by HS code numbers through customs inspection, they are divided into important information about their chemical composition, structure, organoleptic, physico-chemical and other parameters, as well as the processes of production of goods. This, in turn, makes it possible to correctly name goods, prevent a number of crimes that may occur in economic relations, and, finally, protect the interests of consumers. Therefore, the classification of food products based on the Commodity Nomenclature of Foreign Economic Activity is one of the most pressing issues for participants in foreign economic activity. This depends on a number of factors: under what code the goods are classified on the basis of the Commodity Nomenclature of Foreign Economic Activity, it depends on the customs rate subject to payment of customs duties; – classification of goods according to a particular HS code requires special knowledge in the field of classification and additional knowledge related to determining the description of goods and their application according to the Harmonized System (Andreeva, E.I., 2016).

Selection and preparation of samples for analysis (obtaining methyl esters of fatty acids) – according to Interstate standard (GOST) 31665, section 5 or 6 (except 6.1.3 and 6.2.4), with the following addition:

A sample of the spread weighing from 40 to 50 g is melted in a beaker in a water bath or in an oven at (60 ± 5) °C, and kept at this temperature until complete separation. The fat layer is filtered through a pleated filter. If the filtered fat is transparent, then proceed to measurement. If there is turbidity in the fat, it is filtered again. A sample is taken from the separated fat for measurements.

Considering that the triglycerides of the fat phase of the spread, baked mixture, milk and dairy products contain low molecular weight acids (starting with butyric acid -C4: 0), when obtaining methyl esters, filtration is replaced by centrifugation. The resulting solution of fatty acid methyl esters should be used for analysis immediately after preparation.

Determination (analysis of methyl esters of fatty acids) – according to GOST 31663.

Note – Please note that methyl esters of fatty acids contain methyl esters of low molecular weight acids (starting with butyric acid – C4:0), therefore gas chromatographic analysis should be carried out with mandatory temperature programming, starting from a temperature of 60 to 100 °C (depending on from the stationary phase used), ensuring satisfactory separation of butyric acid methyl ester from the solvent.

Processing the results

B.6.1 Processing of the results of the analysis of methyl esters of fatty acids is carried out by the method of internal normalization based on the peak areas of the components, using chromatograph software or according to GOST 31663 (clause 7.2.2). When calculating the mass fraction of milk fat, the calculation of the mass fraction of butyric acid methyl ester is carried out accurate to the third decimal place, followed by rounding to the second decimal place. Calculation of the mass fraction of milk fat in the fat phase of the product under study. If the obtained value of the mass fraction of butyric acid methyl ester is equal to or more than 3.10%, it is considered that the mass fraction of milk fat in the product exceeds 85%, that is, it exceeds the upper limit of the measurement range using this method.

The mass fraction of milk fat in the analyzed sample X_1,%, is calculated using the formula

$$X_1 = \frac{X_i}{X_{mi}} * 100$$

where X_i is the mass fraction of butyric acid methyl ester,%;

X_{mi} is the average value of the mass fraction of butyric acid methyl ester in milk fat,%, equal to 3.1.

Agilent Technologies 7820A GC Gas Chromatograph

Flame ionization detector

Column: SP-2560100 m × 0.25mm × 0.2 µm

The methods used in the examination of the quality of identification of vegetable oils have been studied. Expertise of the quality of vegetable oil is determined by studying the content of free fatty acids in its composition. The examination is carried out using a GC–MS instrument,

There are different ways to assign a sample to it:

1) Sulfuric acid-methanol method

Place 1 g sample in an Erlenmeyer flask, add 60 ml sulfuric acid-toluene-methanol solution (Add 2 ml concentrated sulfuric acid to 230 ml toluene: methanol = 1:3 (v/v) mixed solution). Heat in a refrigerator and boil for 2.5 hours, then cool, transfer to a separating funnel and add 100 ml of water. Extract twice, using 50 ml of petroleum ether each time. The extracts were combined and washed each time with 20 ml of water. Washing was repeated until it's over. After dehydrating the petroleum ether solution with anhydrous sodium sulfate, the solvent is removed using a rotary evaporator."

2) BF3-methanol method

Place 0.5 g of sample in an Erlenmeyer flask with a regular stopper, add 50 ml of 1 N. KOH-ethanol solution to dissolve, add zeolite, attach a reflux condenser and heat in a boiling water bath for about 1 hour.

After heating, add about 50 ml of water and pour into a separatory funnel. After cooling the contents to room temperature, add 30 ml of petroleum ether each time and shake twice to separate the petroleum ether layer.

Add 1 N. hydrochloric acid to the aqueous layer to make it acidic, and then extract twice, using approximately 30 ml of petroleum ether each time. The extracts are combined, a small amount of anhydrous sodium sulfate is added to dehydrate, and about 20 ml of the mixture is transferred to an Erlenmeyer flask. To this add 10 ml of BF3-methanol solution (boron trifluoride diethyl ether complex: methanol = 1:2 (v/v)), add a reflux condenser and heat in a boiling water bath for about 10 minutes, then add about 50 ml. water, ml, transfer to a separatory funnel, add 20 ml of petroleum ether, shake vigorously and extract the organic layer. 10 ml of petroleum ether is again added to the aqueous layer, extracted in the same way, combined with the previous organic layer and the solvent is removed using a rotary evaporator.

Olive oil quality analysis using GC-MS

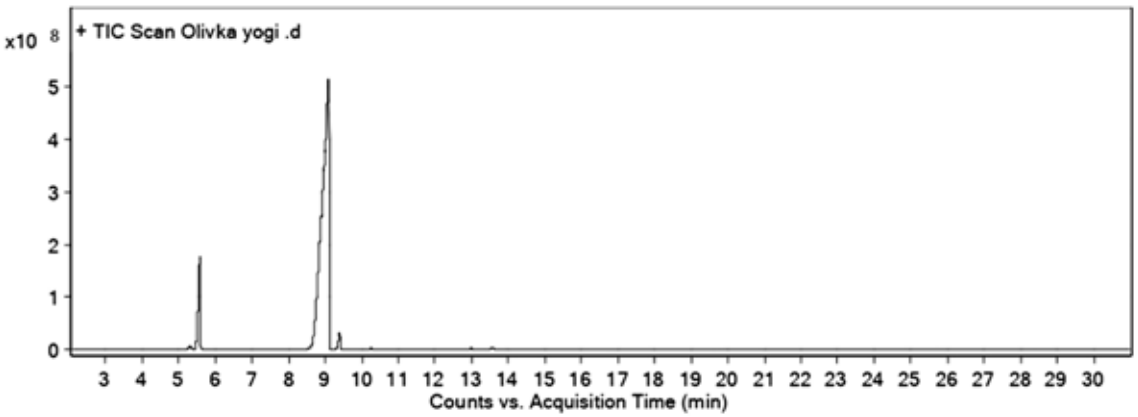
Figure 1. *Olive oil quality analysis*

Qualitative Analysis Report

Data File	Olivka yogi .D	**Sample Name**	
Sample Type		**Position**	1
Instrument Name	GC-MS	**User Name**	Nodir
Acq Method	Нодир ёгпар.m	**Acquired Time**	7/7/2023 5:38:35 PM
IRM Calibration Status	Not Applicable	**DA Method**	default.m
Comment			
Требуется штрихкод		**Количество пробы:**	
Объем двойного ввода		**TuneName**	ATUNE.U
TunePath	C:\MassHunter\GCMS\1\5977\	**MSFirmwareVersion**	6.00.25
OperatorName	Nodir	**RunCompletedFlag**	True
Acquisition SW Version	MassHunter GC/MS Acquisition B.07.03.2129 18-May-2015 Copyright © 1989-2014 Agilent Technologies, Inc.		

User Chromatograms

Fragmentor Voltage	Collision Energy	0	Ionization Mode	Unspecifie d



User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
	0	Unspecified

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Agilent Technologies

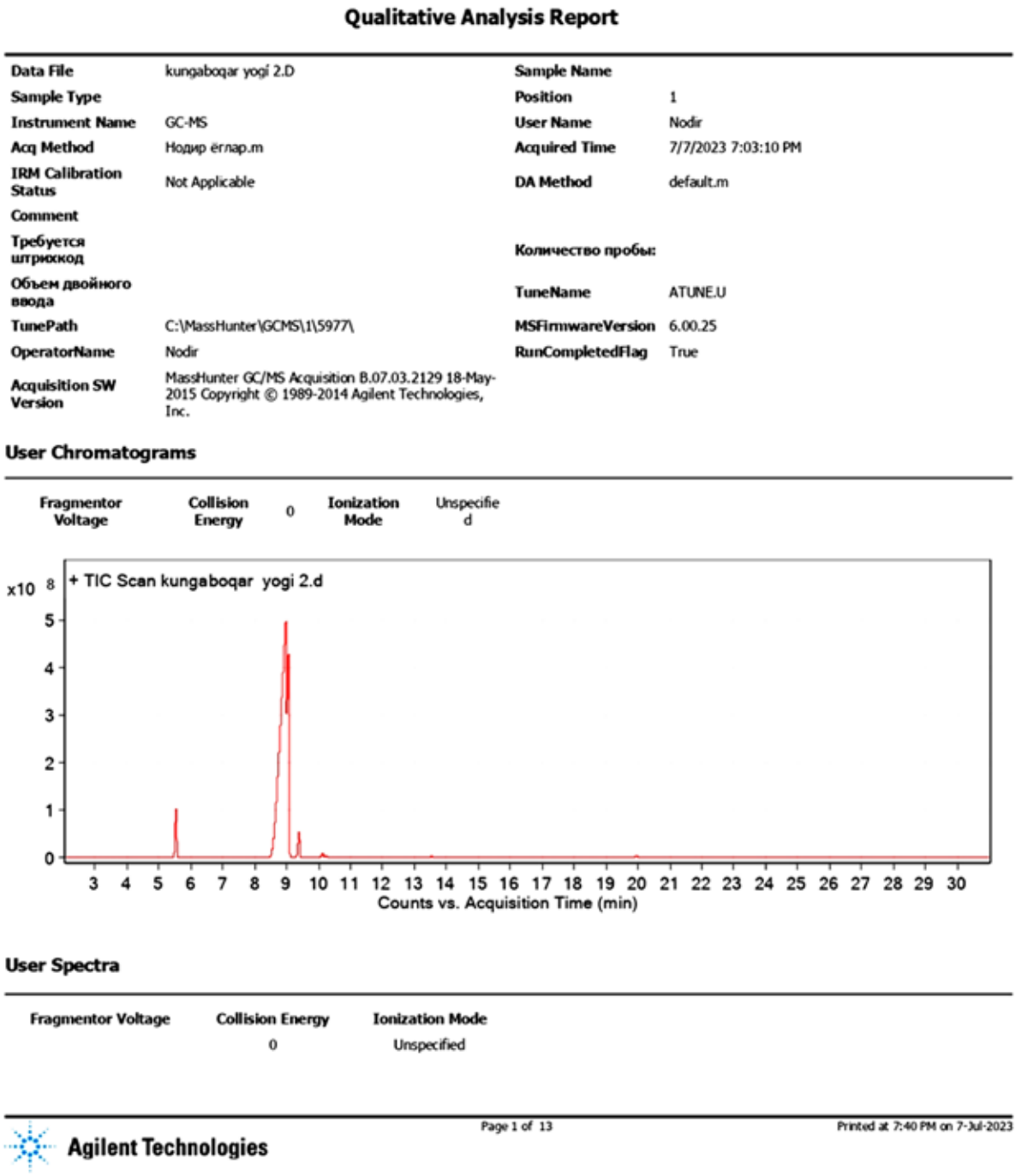
3) BF3-methanol method (standard oil analysis method)

Take about 50 mg of sample into an Erlenmeyer flask, add 1 ml of 0.5 N. CON. -methanol solution Add. Turn on the cooler and heat in a water bath for 5–10 minutes. Add 1 ml of BF3-methanol solution from the top of the cooler. After boiling for 2 minutes, add 5 ml of

hexane from the top of the refrigerator and boil for another 1 minute. Remove the flask from the refrigerator and add saturated aqueous sodium chloride until the hexane layer reaches the neck of the flask. Transfer the hexane layer to a beaker and add anhydrous sodium sulfate."

Analysis of sunflower oil quality using a GC-MS device

Figure 2. *Analysis of sunflower oil quality*



Qualitative Analysis Report

Data File	kungaboqar yogi 2.D	**Sample Name**	
Sample Type		**Position**	1
Instrument Name	GC-MS	**User Name**	Nodir
Acq Method	Нодир ёғлар.m	**Acquired Time**	7/7/2023 7:03:10 PM
IRM Calibration Status	Not Applicable	**DA Method**	default.m
Comment			
Требуется штрихкод		**Количество пробы:**	
Объем двойного ввода		**TuneName**	ATUNE.U
TunePath	C:\MassHunter\GCMS\1\5977\	**MSFirmwareVersion**	6.00.25
OperatorName	Nodir	**RunCompletedFlag**	True
Acquisition SW Version	MassHunter GC/MS Acquisition B.07.03.2129 18-May-2015 Copyright © 1989-2014 Agilent Technologies, Inc.		

User Chromatograms

Fragmentor Voltage	Collision Energy	0	Ionization Mode	Unspecified

User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
	0	Unspecified

Agilent Technologies

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4) m-TFPTAH method. "Melt the sample under heat and stir until homogenized. Transfer about 10 mg of sample to a vial, add 0.5 ml of toluene and stir. Add 0.2 ml m-TFPTA, cap tightly, mix well and leave at room temperature for 15 minutes."

5) KOH-methanol method.

Place approximately 100 mg of sample in an Erlenmeyer flask, add 2 ml of petroleum ether and toluene (1:1) and dissolve completely. Add 2 ml 0.4 N. KOH-methanol solution, mix well at room temperature and leave

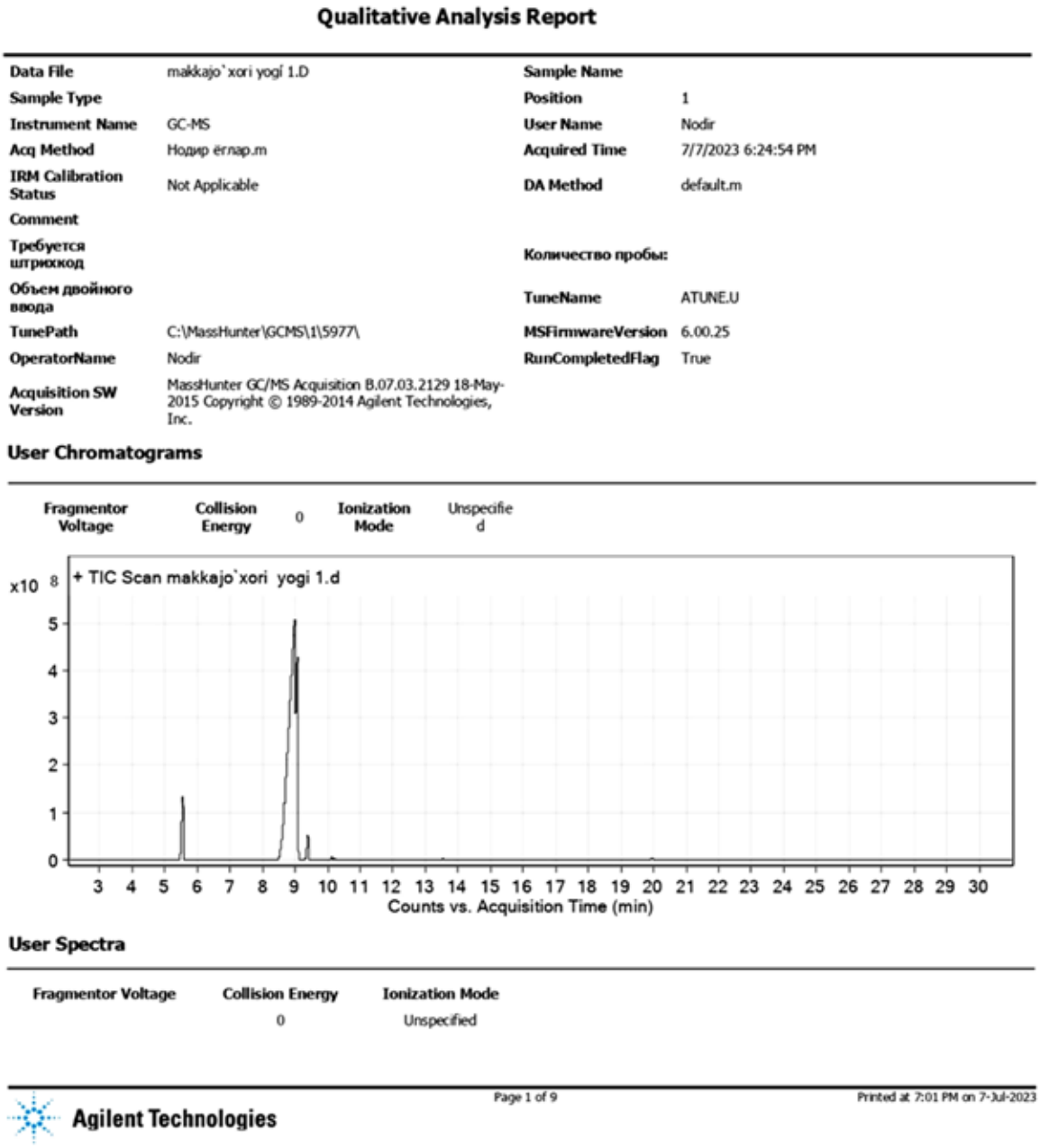
for 5–10 minutes. After settling, distilled water is added until the organic layer reaches the neck of the flask. Add a few drops of ethanol if the organic layer is cloudy. Take the organic layer in a beaker and add anhydrous sodium sulfate."

In this work, sunflower, olive and corn oils were tested using the 5th method, the KOH-methanol method. The results of the analyzes are shown in Fig. 1–3.

Methods and results for determining the quality of corn, olive and sunflower oil were presented.

Corn oil quality analysis using GC–MS

Figure 3. *Corn oil quality analysis*



When the Customs Committee analyzed the database of customs cargo declarations, cases of oil-oil mixtures were found not in commodity heading 1517, but in other commodity headings (1512, 1515). In this case, in addition to inconsistencies in foreign trade statistics, as a result of wrong designation of the CN FEA codes of the goods, there is a situation of shifting the duty rates set for the goods to lower rate codes. If we look at

the tariff rates set according to the Decision No. 3818: vegetable oils – 5 percent; (Commodity position 1517) – 15, not less than 0.15 USD per kg.

An analysis of these rates shows that there are cases of illegal labeling of edible vegetable oil blends as disguised goods to pass customs clearance as single ingredient vegetable oil. At the same time, the prices of vegetable oils are different and are formed based on the method of extraction, time, quantity, demand and supply. In this case, taking into account that the mixtures are composed of different oils, their price (customs value) is determined based on the composition of oil-oil mixtures.

Recommended code for the product commodity nomenclature foreign economic activity of the Republic of Uzbekistan

1517 90910 1 – – – – mixture of olive oil

1517 90910 9 – – – – others

On the basis of the above scientific analysis, the need to correctly determine the CN FEA code of the fat-oil mixture suitable for consumption brings economic benefits in terms of statistics, tariff and customs value. The price of this oil is high due to the complexity of olive oil extraction and production technology. On this basis, it is natural that the price of mixtures of this oil and other vegetable oils is high. Based on the above situation, customs expertise methods were developed in order to introduce a separate CN FEA code to the mixtures of olive and other vegetable oils and to identify them.

Conclusion

Thus, methods and results for determining the quality of corn, olive and sunflower oil were presented. Modern organoleptic and physico-chemical methods for determining the quality of these goods have been developed and introduced into customs practice.

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