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S.R.Hajiyeva,
E.M.Gadirova,
A.A.Musayeva
Baku State University

DETERMINATION OF PAH IN WASTE WATER

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SUMMARY

PAH are one of the most common pollutants entering surface water with effluent from oil refineries, shale refineries, wood chemical, coking, aniline dye industries, as a result of timber rafting, as well as from hydrolysis industry drains. For the analysis of such waters, 3 samples were taken from the points of the Oil Refinery and using analytical methods, we investigated the PAH in polluted waters.

Key words: PAH, gas chromatography-mass spectroscopy, waste water, samples, etc.

С.Р.Гаджиева,
Э.М.Кадырова,
А.А.Мусаева
Бакинский государственный университет

ОПРЕДЕЛЕНИЕ ПАУ В СТОЧНЫХ ВОДАХ

РЕЗЮМЕ

Поли ароматические углеводороды (ПАУ) являются одними из наиболее распространенных загрязнителей, поступающих в поверхностные воды со стоками предприятий нефтеперерабатывающей, сланцеперерабатывающей, лесохимической, коксохимической, анилинокрасочной промышленности, в процессе лесосплава, а также со стоками гидролизной промышленности. Для анализа таких вод нами были взяты 3 образца из точек Нефтеперерабатывающего завода (НПЗ) и с помощью аналитических методов было исследовано содержание ПАУ .

Ключевые слова: ПАУ, хромато-масс спектроскопия, сточные воды, образцы, т.д.

Introduction:

Aromatic compounds enter the biosphere in various ways and their sources are industrial enterprises, transport, and household waste. The particular attention given to aromatic compounds is largely due to their carcinogenic properties. Polycyclic aromatic hydrocarbons (PAHs) enter the atmosphere as a result of emissions and wastes from coke plants, some chemical plants, exhaust from internal combustion engines, and combustion products of various fuels. Groundwater is often contaminated with PAHs due to various wastewater sludge.

Aromatic hydrocarbons in petroleum range from 5 to 55%. These are the most toxic components of oil, and at a concentration of only 1% in water, they kill aquatic lower plants. Oil, containing 38% of aromatic hydrocarbons, significantly inhibits the growth of higher plants, and with an increase in the aromaticity of oil, their herbicidal activity increases. The content of all groups of polycyclic aromatic hydrocarbons gradually decreases with the gradual transformation of oil in the soil. Polycyclic aromatic hydrocarbons (PAHs) formed during fuel combustion are a multicomponent mixture, the individual analysis of each component of which is difficult without preliminary preparation of the sample for analysis. After preliminary chromatography of the sample by column chromatography in a thin layer or by differential sublimation, it is possible to analyze individual PAHs using UV spectroscopy. The method allows to determine the concentration of various PAHs in air up to 10-5%, or 0.1 μg / ml in solution.

Organic compounds such as polycyclic aromatic hydrocarbons (PAHs) are a global environmental concern as they cause inflammation and skin cancer. As you know, there are two types of anthropogenic sources of hydrocarbons: petrogenic and pyrogenic sources. Petrogenic sources include crude oil and petroleum-derived hydrocarbon compounds. Pyrogenic sources of hydrocarbon compounds are formed as a result of incomplete combustion of organic substances such as oil, wood, coal, etc. [one]. About 6.1 million tons of oil products are thrown into the ocean annually, most of which are of anthropogenic origin. Before entering the open sea, these oil products pass through the entire coastal zone.

The Caspian Sea is a very sensitive ecosystem. Over the past decades, under the influence of anthropogenic and biochemical factors, the state of ecosystems in general has deteriorated sharply, and especially in the northeastern part of the sea. Observations of recent years show that the waters of the Caspian Sea, especially along the coast of the National Park, are also polluted with oil and sewage [2]. Industrialization and urbanization in the Caspian region has developed rapidly over the past several decades and the associated increase in hydrocarbons is a concern in the region.Offshore production and accidental oil spills, industrial waste, wastewater, discharges flowing down from river water are considered the main source of anthropogenic hydrocarbons in the marine environment [3]. It is assumed that the main source of oil pollution in the Caspian Sea is industry. The total amount of industrial waste discharged into the Caspian Sea averages 2342.0 million m3 per year. Such waters contain 122.5 thousand tons of oil, 1.1 thousand tons of phenols, 9.9 thousand tons of organic chemistry products. The total content of hydrocarbons in the North-Western part of the South Caspian was small - 32-54.2 μg / g. In this area, in the vicinity of oil fields, the concentration of phenols was 0.002-0.003 μg / g [4,5]. Pollution of water and bottom sediments is noted throughout the Absheron Peninsula and in the Baku Bay. The main volume of pollution (90% of the total) enters the Caspian Sea with river runoff [6,7].

In the article reviewed, we analyzed wastewater taken from an oil refinery. It was important for us to calculate the amount of PAHs in these polluted waters. After purification, these waters are discharged into the Caspian Sea, and even in small quantities these harmful substances are dangerous for the flora and fauna of the sea and the environment. As is already known, PAHs are very dangerous for the environment, for living organisms in aquatic ecosystems, and therefore, the identification of hazardous substances and the application of methods for their destruction is very important [5,8].

Working process:

We used a chromatography-mass spectroscopic method for the analytical determination of PAHs in wastewater taken from an oil refinery. Dichloroethane for water extraction (GC-MS) was used to determine PAHs in aqueous samples. Then PAHs were determined in samples 1,2,3.

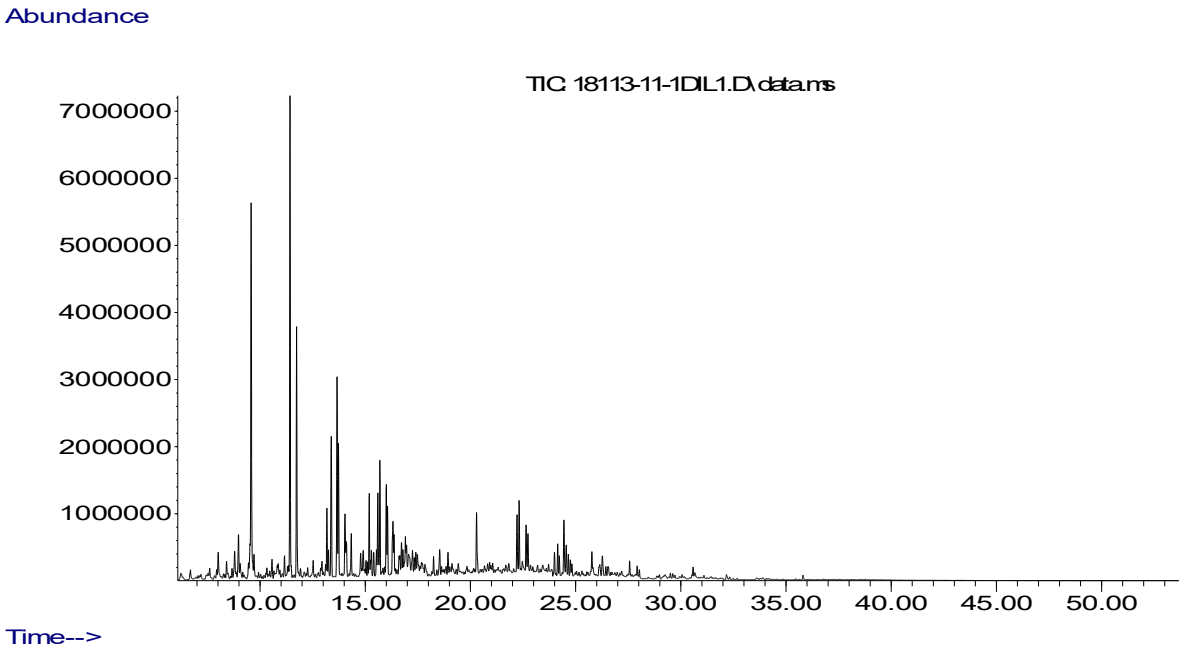
Extraction was carried out in separating funnels previously cleaned with methylene chloride. A mixture of compounds used as an internal standard was added to the seawater samples prior to extraction. Next, 50 ml of methylene chloride was added to a separatory funnel, and the contents of the funnel were vigorously shaken for several minutes. After peeling in a separating funnel, the organic part was separated from the aqueous. The procedure was repeated two more times. All three extracts were combined in a round-bottom flask and concentrated using a rotary evaporator at a

water bath temperature of 30 ± 5 ° C to a volume of 2 ml, then dried under a thin stream of nitrogen and transferred to samplers in a volume of 1 ml. During the analysis of the samples, the solvents used were dichloromethane (Rathburn, Great Britain, Scotland) and n-pentane (HiPerSolv for HPLC, BDH, Great Britain, England) with chromatographic purity. For this purpose, the solvents were previously checked for purity using the gas chromatography method. Special measures have been taken to prevent contamination from glassware, teflons, steel materials. Deionized water and methylene chloride were used to clean the dishes.

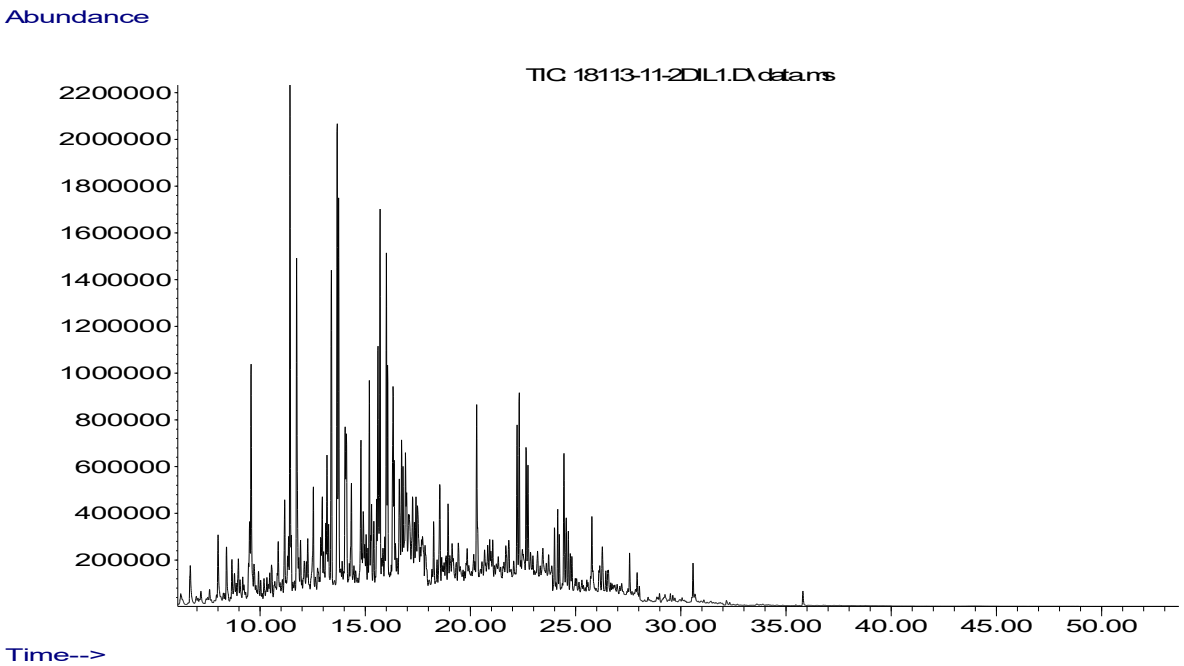
Qualitative analysis was carried out on an HP6890 GC instrument with an HP5975 mass selective detector, GC-MS (Agilent, USA) equipped with a ZB-5 column (Phenomenex, USA).

The discussion of the results:

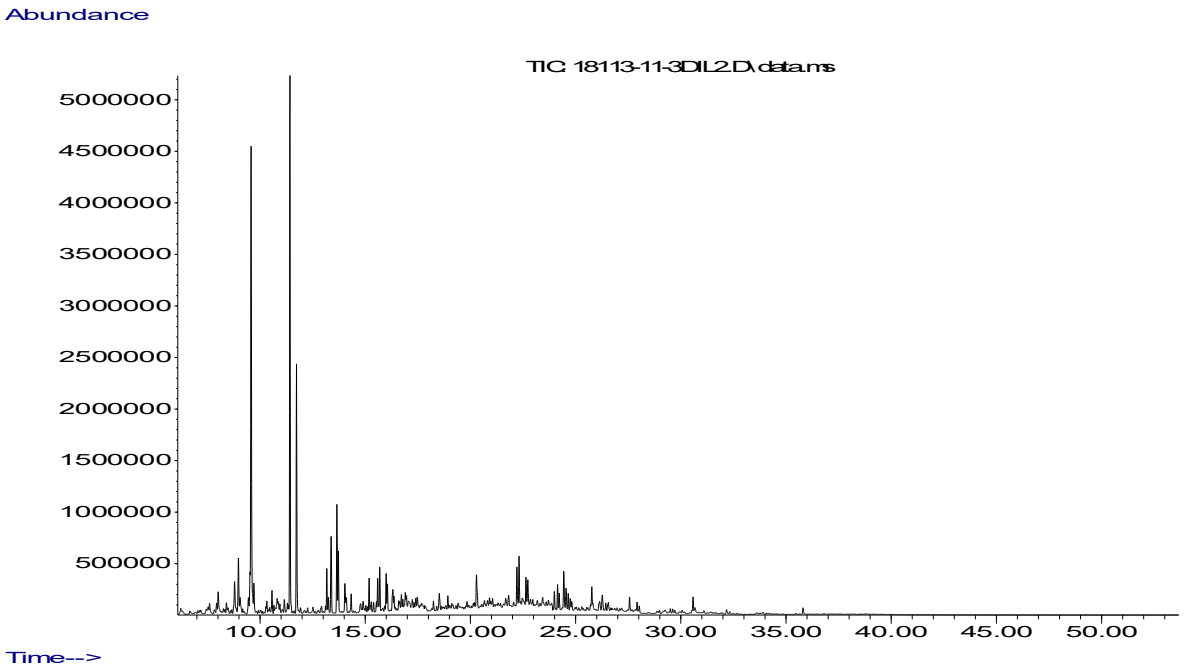
Below are chromatographic-mass spectra of these wastewater samples: 1; 2; 3 analyzes.



Mass spectrum of sample 1



Mass spectrum of sample 2



Mass spectrum of sample 3

For different water courses, the MPC for polyaromatic hydrocarbons ranges from 4246 to 709168 µg /l. It was found that in the analyzed samples the amount of PAHs (mainly the amount of naphthalene) is very high and exceeds the MPC for wastewater.

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Hajiyeva S.R.,
Gadirova E.M.,
Jafarova X.F.
Baku State University

DETERMINATION OF PHENOL AND PHENOL DERIVATIVES IN WATER SAMPLES TAKEN FROM THE CASPIAN SEA

http://dx.doi.org/10.26739/2181-0818-2021-1-2

ANNOTATION

Phenol and its derivatives are currently the main pollutants in the aquatic environment. In the reviewed article, water samples were taken from Shikh and Bulvar areas and phenol and its derivatives were analyzed. Analysis of water samples was performed on a mass detector with Agilent 5975C GC / MS gas chromatography.

Keywords: phenol, water analysis, oil industry, analysis methods.

Гаджиева С.Р.,
Кадырова Э.М.,
Джафарова Х.Ф.
Бакинский Государственный Университет

ОПРЕДЕЛЕНИЕ ФЕНОЛА И ПРОИЗВОДНЫХ ФЕНОЛА В ОБРАЗЦАХ ВОДЫ КАСПИЙСКОГО МОРЯ

АБСТРАКТ

Фенол и его производные в настоящее время являются наиболее распространенными загрязнителями. В статье анализируются пробы воды, взятые в районах Шихова и Бульвара. Количество фенола и его производных в пробах воды анализировали методом хромато-масс-спектроскопии.

Ключевые слова: фенол, анализ воды, нефтяная промышленность, методы анализа.

Hacıyeva.S.R.,
Qədirova E.M.,
Cəfərova X. F.
Bakı Dövlət Universiteti

XƏZƏR DƏNİZİNDƏN GÖTÜRÜLMÜŞ SU NÜMUNƏLƏRİNDƏ FENOL VƏ FENOL TÖRƏMƏLƏRİNİN TƏYİNİ

Fenol və onun törəmələri hazırda su mühitini çirkləndirən əsas çirkləndiricilərdəndir. Baxılan məqalədə Şıx və Bulvar ərazilərindən su nümunələri götürülərək tərkibində fenol və onun

törəmələri analiz edilmişdir. Su nümunələrinin analizi Agilent 5975C GC/MS qaz xromotoqrafiyalı kütlə detektorunda aparılmışdır.

Açar sözlər: fenol, su analizi, neft sənayesi,analiz metodları.

As the Republic of Azerbaijan is an oil country, toxic compounds are common as a result of the discharge of industrial waters into the Caspian Sea. As we distinguish, oil and oil products enter water bodies in different conducts: during oil transportation, during drilling accidents, contamination with produced water, during oil extraction and other processes [1].

At the present moment the principal scientific and technical delinquent is the environmental fortification. It should be distinguished that the environment is sufficiently polluted with both industrial surplus and household waste. The mixing of organic and mineral wastes into the water and soil environment is due to the increase in municipal and industrial wastewater [2].

The Caspian Sea is notable for its sensitive ecosystem. In the last decade, anthropogenic and biochemical impacts have almost devastated the ecosystem. In particular, it should be noted that the situation in the north-eastern part of the Caspian Sea has reached crisis level. Even when walking in the National Park, we come across these pollutants. Anthropogenic pollution - accidental spills of oil into the Caspian Sea during oil transportation, runoff, industrial waste, etc. It is believed that the main source of pollution in the Caspian Sea is the development of industry [3].

As we know, phenol and its derivatives are the most common substances in industrial effluents. Phenols and their compounds are found not only in wastewater, but also in natural waters. It is well known that phenol contains a benzene ring and hydroxyl groups. The permissible amount of volatile phenols in water bodies should not exceed 0.1 g / l. Today, the most global environmental problem is the presence of organic substances in water with a polycyclic structure. Phenol-containing industrial water flows into the municipal sewage system only after it has been neutralized [4].

Experimental part:

As we know, oil and oil products enter the soil and water basins in different ways [5]. Currently, the biggest scientific and technical problem is environmental protection. It should be noted that the environment is sufficiently polluted with both industrial waste and household waste. The mixing of organic and mineral wastes into the water and soil environment is due to the increase in municipal and industrial effluents [6].

Phenol and its compounds have an awful effect on the environment and living organisms. For this reason, methods for the determination of indirect phenol derivatives have not lost their relevance. Even small amounts of phenol and its compounds pose a threat to the flora and fauna of the Caspian Sea [7].

In the presented work, a water trials were taken from Shikh and Boulevard areas and submitted to the laboratory for analysis. Mass spectroscopy equipped with gas chromatography was used to obtain accurate results. The names and quantities of the substances obtained in the analyzes are also reflected in Table 1.

Table 1.

Results of analysis of water trials

Phenol and its derivatives of compounds,μq/ l	water trial taken from Shikh	water trial taken from boulevard
Fenol	0.10	0.14
o-kresol	0.02	0.03
2-nitrophenol	<0.04	0.04
2,4-dimetilfenol	0.02	0.04
2,4-dichlorophenol	<0.02	<0.02
2,6-dichlorphenol	0.02	0.04
4-chloro-3-metilphenol	<0.04	0.05

2,4,5-trichlorphenol	<0.04	<0.04
2,4,6-trichlorphenol	<0.04	<0.04
2,3,4,6-tetrachlorophenol	<0.04	<0.04
Pentachlorphenol	<0.04	<0.04

The most widely used analytical method is mass chromatography. It is used to separate mixtures into components, to distinguish and determine gaseous mixtures, and to control changes in the concentration of reagents during chemical reactions. Its working principle is based on the fact that the flow of the studied gas mixture is transferred to the separating column. The column is filled with special solid neutral balls, non-volatile separating liquid impregnated on the ceramic. The components of the test mixture are sorbed by separating them along the length of the column. In modern devices, it consists of a metal tube with a thin twisted spiral inside the columns. The gas is continuously released into the column, the separated components are removed from it in turn [8].

First, the worst sorbed component of the mixture is removed. The chemist-analyst can distinguish between a mixture of synthetic and natural substances that cannot be transferred to the gas phase by selecting the length of the column, filler (pellets, ceramics) and separator fluid, as well as gas flow rate and temperature.

It is not enough to separate the mixture into its components, but also to determine the concentration and composition of the substances. Therefore, each chromatograph is equipped with a mass detector with a measuring device. The detector records the components that are squeezed out of the column. First of all, the device is equipped with two types of detectors. The catharometer records changes in the thermal conductivity of a gas stream with a change in current in a thin metal wire. The device does not react to the chemical nature of the substance entering the detector. With the amplifier, it is connected to a self-recording pen, and the detector peaks on a paper tape that moves as each part of the substance enters; the corresponding area is directly proportional to the concentration of the substance [9].

Phenol is formed in small industrial enterprises together with technological condensate. Purification of phenols from industrial enterprises is a very difficult process. Agilent 5975C GC / MS gas chromatographic mass spectroscopy was used to analyze water samples taken from the Shikh and Boulevard areas [10].

İn figures 1 and 2 are presented a gas chromatographic mass spectroscopy of a water sample taken from Shikh and Boulevard.

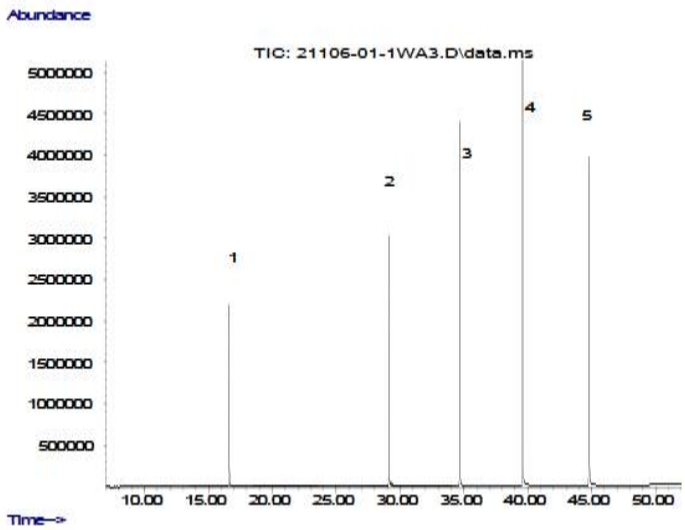

Figure 1. The chromatography of water trial taken from Shikh

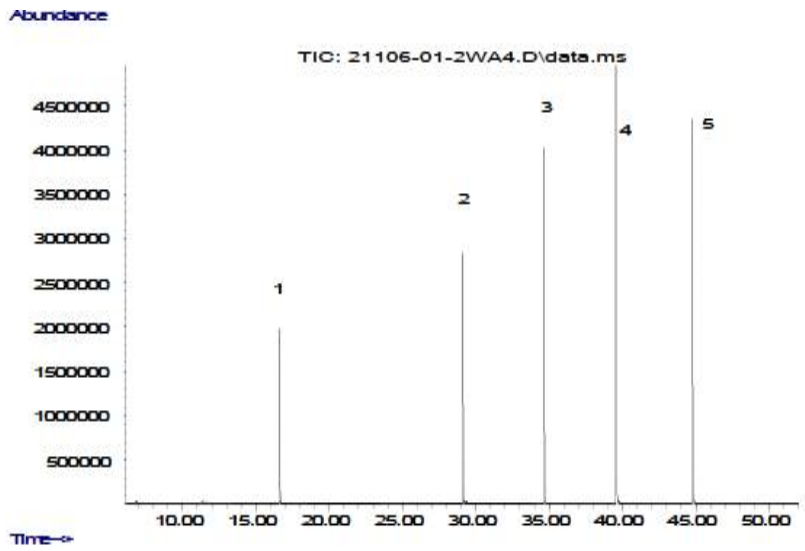

Figure 1. The chromatography of water trial taken from Boulevard

To get acquainted with the problem of water sources, it is necessary to look at the statistics. About 6 million tons of petroleum hydrocarbons are discharged into the sea every year. About 2 million barrels of oil are discharged into the world's oceans by rivers. A thin layer of oil covers huge areas of water[11].

Ast i is known, work is underway to find and improve new methods and technologies. These methods combine their effectiveness and the ability to protect our planet. The main advantage of the methods studied and proposed is that it does not require much funding [12].

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