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Automated IR determination of petroleum products in water based on sequential injection analysis



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ARTICLE INFO

Article history:

Received 20 February 2015

Received in revised form

14 May 2015

Accepted 18 May 2015

Available online 21 May 2015

Keywords:

Petroleum products

Water

Extraction

Sequential injection analysis

Infrared detection

ABSTRACT

The simple and easy performed automated method for the IR determination of petroleum products (PP) in water using extraction-chromatographic cartridges has been developed. The method assumes two stages: on-site extraction of PP during a sampling by using extraction-chromatographic cartridges and subsequent determination of the extracted PP using sequential injection analysis (SIA) with IR detection. The appropriate experimental conditions for extraction of the dissolved in water PP and for automated SIA procedure were investigated. The calibration plot constructed using the developed procedure was linear in the range of 3–200 $\mu\text{g L}^{-1}$. The limit of detection (LOD), calculated from a blank test based on 3σ was 1 $\mu\text{g L}^{-1}$. The sample volume was 1 L. The system throughput was found to be 12 h^{-1} .

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1. Introduction

Petroleum products inevitably are released into the environment and contaminate surface water bodies particularly near production or storage sites, but also escape accidentally during handling, transport or processing [1]. As it is well known [2,3], the petroleum products are very complex mixtures that contain primarily aliphatic and aromatic hydrocarbons and heterocycles. Currently, World Health Organization regulates the PP content (dissolved/emulsions) in water, which cannot exceed levels higher than 300 $\mu\text{g L}^{-1}$ to provide a conservative level of protection [4]. Thus, one of the most important analytical tasks of environmental monitoring is the PP determination in the natural waters to assess levels of environmental pollution.

The four most commonly used PP testing methods include gas chromatography (GC) [5–10], infrared absorption (IR) [11], spectrofluorimetry (SFL) [12] and gravimetric analysis (GA) [13,14] (Table 1). GC-based methods detect a broad range of hydrocarbons, provide both sensitivity and selectivity, and can be used for petroleum hydrocarbon identification as well as quantification. However, there are difficulties in the automation of whole procedures and determination of total PP content in water. The main advantage of the IR method is the insignificant dependence of absorption on the type of petroleum hydrocarbons, mainly

containing in water samples. However, the IR method assumes the delivery of large volume of water sample in laboratory (up to 1 L per determination). The SFL is the most sensitive method for the determination of aromatic hydrocarbons and heterocycles, but the emission of hydrocarbons depends on their structure [15]. GA methods may be useful for water samples with a high PP concentration.

Generally GC, IR, SFL, and GA methods include conventional liquid-liquid extraction (LLE) of PP from the water samples to another water-immiscible solvent. Commonly used solvents for extraction are carbon tetrachloride, tetrachlorethylene, trifluorotrichloroethane and hexane. Although LLE is relatively simple and inexpensive, it has many drawbacks, among them the need to use large quantities of solvents. In order to achieve the desired enrichment factor, the excess solvent requires removal by evaporation, and extract cleanup may also be necessary. Also the solid-phase (SPME) [7], headspace solid-phase microextraction (HS-SPME) [8] and flow solid-phase (FSPME) [9] microextraction techniques are proposed to the hydrocarbons determination in water by GC. The SPME is based on the extraction of hydrocarbons from the water using a microsyringe equipped by poly(dimethylsiloxane) fiber in a needle. In the case of HS-SPME fiber with a poly(dimethylsiloxane) coating is placed in a headspace of a water sample. For the FSPME water is passed through a syringe steel needle filled with the Tenax GR sorbent at the rate of 2 mL min^{-1} . The double solid-phase extraction (SPE) is suggested for the determination of aliphatic and aromatic hydrocarbons in

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Table 1
Comparison of the methods for determination of PP.

Detection technique	Analyte	Sample	Sample volume, mL	Sample preparation	LOD	Reference
GC-FID	C ₁₀ –C ₄₀ Hydrocarbons	Surface and wastewater	900	Extraction (50 mL hexane), solvent evaporation (to 1 mL)	0.1 mg L ⁻¹	[5]
GC-FID	Hydrocarbons	Water and wastewater	2	Solid-phase microextraction (poly(dimethylsiloxane) fiber)	0.03–1 µg L ⁻¹	[7]
GC-FID	C ₆ –C ₂₀ Hydrocarbons	Water	2	Headspace solid-phase microextraction (poly(dimethylsiloxane) fiber)	2.0–13 µg L ⁻¹	[8]
GC-FID	Hydrocarbons	Water	5	Flow solid-phase microextraction (Tenax GR sorbent)	1–5 µg L ⁻¹	[9]
GC-FID	Hydrocarbons	Groundwater	100	Double solid-phase extraction (Sep-Pak C18 cartridges)	5 µg L ⁻¹	[10]
IR	Aliphatic and aromatic hydrocarbons and heterocycles	Surface and saline waters, industrial and domestic wastes	1000	Extraction (30 × 2 mL trifluorotrichloroethane), interferences adsorption (silica gel)	1 mg L ⁻¹	[11]
SFL	Aromatic hydrocarbons and heterocycles	Surface and wastewater	100	Extraction (10 mL hexane), interferences adsorption (aluminum oxide)	5 µg L ⁻¹	[12]
GA	Oil	Surface and saline waters, industrial and domestic wastes	1000	Extraction (30 mL hexane), interferences adsorption (silica gel)	1.4 mg L ⁻¹	[13]
FIA-SFL	Aromatic hydrocarbons and heterocycles	Natural water	20	On-line extraction-chromatographic preconcentration (PTFE column), elution (0.5 mL hexane) and chromatomembrane phase separation	1 µg L ⁻¹	[15]
FIA-IR	Aliphatic and aromatic hydrocarbons and heterocycles	Water	6.5	On-line extraction (4 mL 1,2,3,4-tetrachloro-1,1,2,3,4,4-hexafluorobutane) and membrane phase separation	0.1 mg L ⁻¹	[16]
SIA IR	Aliphatic and aromatic hydrocarbons and heterocycles	Water	1000	On-site extraction (PTFE cartridge), elution (1 mL trifluorotrichloroethane) and interferences adsorption (silica gel)	1 µg L ⁻¹	This work

IR – infrared absorption; GC-FID – gas chromatography-flame-ionization detector; SFL – spectrofluorimetry; GA – gravimetric analysis; FIA-SFL – flow injection analysis with SFL detection; FIA-IR – flow injection analysis with IR detection; SIA IR – sequential injection analysis with IR detection.

groundwater [10]. By using the first SPE (reverse phase), the hydrocarbons are extracted from groundwater sample, meanwhile the second SPE is accomplished for fractionating hydrocarbons into aliphatic and aromatic hydrocarbons. Finally SPME and SPE devices are introduced into the injection port of gas chromatograph to thermal desorption of hydrocarbons.

The important and rapidly growing trend in modern analytical chemistry is the automation of analysis. Currently, the automation of analytical procedures based on flow analysis is intensively developing. To the best of our knowledge, only two articles have been devoted to the PP determination in water based on flow system [15,16].

The developed flow-injection method with IR detection assumes the mixing carbon tetrachloride with an aqueous carrier containing the sample into an extraction coil. Finally the two phases are separated into a membrane separator and the organic phase is transferred to the detection cell for absorbance measurement [16]. The main disadvantage of this flow method is low sensitivity (0.1 mg L⁻¹) because it is impossible to increase water and organic segments volume ratio to PP preconcentration. In the case of FIA fluorimetric determination of aromatic hydrocarbons and heterocycles in water [15], the on-line preconcentration of analytes is carried out into the extraction-chromatographic column. The extract is eluted by hexane with the following separation of extract from aqueous phase in chromatomembrane cell and aromatic hydrocarbons and heterocycles are detected, wherein all saturated hydrocarbons are ignored. This fact leads to the essential underestimation of the results in the determination of PP by this detection method when the sample contents, for example, petrol, kerosene and other same fractions.

The aim of this work was to develop an automated method for IR determination of total PP content (aliphatic and aromatic hydrocarbons and heterocycles) in water. To increase the sensitivity and exclude the mentioned disadvantages of LLE the special extraction-chromatographic cartridges have been developed and used for on-site extraction of PP during a sampling.

2. Experimental

2.1. Chemicals

Analytical grade chemicals and distilled water were used throughout the experiments. Carbon tetrachloride, tetrachlorethylene, trifluorotrichloroethane, hexane, isooctane, hexadecane, benzene, isopropyl alcohol, aluminum oxide and silica gel for chromatography (01–0.25 mm) were purchased from Sigma-Aldrich. Different types of PP (gasoline, diesel, transformer, industrial and vaseline oils) were obtained from the co-operation partner (Gasprom, Russia).

The 0.5 g L⁻¹ stock solutions of PP in a water-soluble matrix were prepared by dissolving PP in isopropyl alcohol. The working water solutions/emulsions of PP were prepared by dissolving the corresponding aliquots of 0.5 g L⁻¹ stock solutions in water and adjusting the volume up to 1 L by adding distilled water, and then flask contents were carefully mixed and immediately analyzed.

The calibration solutions were prepared by mixing isooctane, hexadecane and benzene at the volume ratio of 1.5:1.5:1 (OCB standard solution) and dissolution of this mixture in trifluorotrichloroethane, and stored in a sealed container to avoid evaporative loss.

2.2. Apparatus

2.2.1. Sampling and sample preparation setup

Extraction-chromatographic cartridges (polytetrafluoroethylene (PTFE)) (height – 20 mm, 5 mm i.d.) using for PP extraction were filled

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