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ARTICLES

Dedicated to the 300th Anniversary of St. Petersburg State University

Microextraction-Chromatographic Determination of Furan Derivatives in Transformer Oil

P. A. Godunov[a], *, **A. Yu. Shishov**[a], **and A. V. Bulatov**[a]
[a] *Institute of Chemistry, St. Petersburg State University, St. Petersburg, 199034 Russia*
**e-mail: paulgodunov@yandex.ru*

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Abstract—A rapid and an environmentally friendly method is developed for the microextraction of furan derivatives from transformer oil for their determination by high-performance liquid chromatography with ultraviolet spectrophotometric detection. Various hydrophilic eutectic solvents were studied as extractants to implement vortex-assisted dispersive liquid–liquid microextraction. The results indicate that a three-component eutectic solvent composed of choline chloride, acetic acid, and water achieves the highest recoveries, ranging from 85 to 96%. The rapid spontaneous phase separation eliminated the need in centrifugation. Limits of detection (3σ) were achieved between 1 and 5 μg/L.

Keywords: dispersive liquid–liquid microextraction, eutectic solvents, liquid chromatography, furan derivatives, transformer oil

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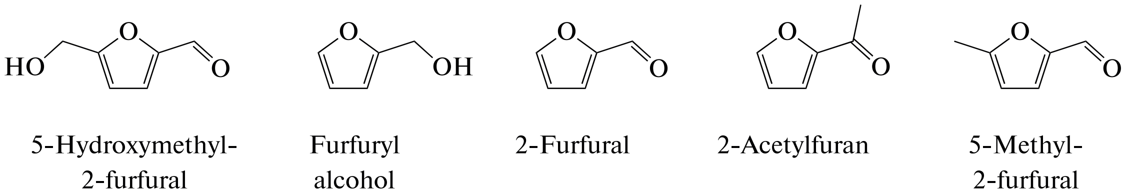
Electrical transformers play a crucial role in the distribution of electrical energy. The condition of these devices during operation at power plants undergoes regular monitoring through the chemical analysis of the transformer oil used for electrical insulation and cooling within the transformer system. In addition to transformer oil, the insulation system also includes paper insulation, which performs the following functions: separation of conductors, prevention of short circuits, and provision of mechanical strength to the windings [1]. During operation, paper loses its mechanical properties, leading to degradation and the formation of furan derivatives, such as 5-hydroxymethyl-2-furfural, furfuryl alcohol, 2-furfural, 2-acetylfuran, and 5-methyl-2-furfural (Scheme 1) [2]. Diffusion facilitates the mass transfer of furan derivatives to transformer oil. In practice, the results of determining the concentration of furan derivatives in transformer oil are used to assess the remaining lifespan of the paper insulation [3].



Scheme 1. Chemical structures of furan derivatives formed during the degradation of paper insulation.

Currently, chromatographic [4–8] and electrochemical [9, 10] methods are used to determine furan derivatives in transformer oil. Sample preparation must include the extraction of furan derivatives through liquid–liquid or solid-phase extraction, because the matrix components of the sample interfere with the determination of analytes. Conventional liquid–liquid and solid-phase extraction methods require large amounts of toxic, volatile, and flammable organic solvents, such as acetonitrile [4, 5], *n*-hexane [4, 6], and *n*-pentane [5]. In some cases, sample preparation can be time-consuming and labor-intensive [7, 8]. A general trend in modern analytical chemistry is the miniaturization of analysis procedures and

Table 1. Composition of eutectic solvents

Hydrogen bond donor	Choline chloride-to-hydrogen donor bonds molar ratio, mol/mol	Weight fraction of water in the ES, %
Ascorbic acid	1.0 : 1.0	10.0
Citric acid	1.0 : 1.0	10.0
Malic acid	1.0 : 1.0	10.0
Malonic acid	1.0 : 1.0	10.0
Oxalic acid	1.0 : 1.0	10.0
Tartaric acid	1.0 : 1.0	10.0
Lactic acid	1.0 : 1.0	10.0
Formic acid	1.0 : 1.0	10.0
Acetic acid	1.0 : 1.0	10.0
	1.0 : 2.0	10.0
	2.0 : 3.0	10.0
	3.0 : 2.0	10.0
	1.0 : 1.0	15.0
	1.0 : 1.0	20.0

ensuring their environmental safety [11]. In this context, liquid–liquid microextraction utilizing extractants of the next generation offers new possibilities.

The method of dispersive liquid–liquid microextraction (DLLME) [12] has found widespread use for the extraction of analytes from organic liquids. DLLME is characterized by its speed, simplicity of technical implementation, and the ability of varying widely the properties and composition of the extractants used. Based on the approach to the extraction process, DLLME can be classified as follows [13]: conventional DLLME (with the use of a dispersive solvent), ultrasound-assisted, vortex-assisted, and in-syringe version using an air stream. For the preparation of viscous petroleum product samples, vortex-assisted DLLME is predominantly used, as this method ensures a rapid equilibrium in extraction systems and the high reproducibility of the results [14].

Next-generation extractants, eutectic solvents (ES), have been proposed [15]. From a thermodynamic viewpoint, eutectic solvents are mixtures of Lewis and Brønsted acids and bases, possessing a eutectic point, in which the melting temperature of the mixture is lower than that of an ideal solution formed by the components [16]. Depending on their stability upon contact with water, eutectic solvents are classified as hydrophilic, quasi-hydrophobic, and hydrophobic [17]. By varying the chemical nature and the ratio of ES precursors, it is possible to design effective extractants capable of isolating analytes from matrices of varying polarity. An additional advantage of eutectic solvents is that their components are inexpensive, nontoxic, and often biodegradable [18, 19]. Furthermore, eutectic solvents possess properties such as high thermal stability, low vapor pressure, and nonflammability, which offer a broad potential for their applications to analytical chemistry [20]. Despite the growing demand for performing “furan tests,” a method for isolating furan derivatives from transformer oil using eutectic solvents based on the principles of vortex-assisted DLLME has not yet been implemented.

This study aims to develop a method for extracting furan derivatives from transformer oil into a hydrophilic eutectic solvent using vortex-assisted DLLME, followed by their determination via high-performance liquid chromatography with spectrophotometric detection in the ultraviolet region (HPLC–UV).

EXPERIMENTAL

Working solutions of furan derivatives (5-hydroxymethyl-2-furfural, furfuryl alcohol, 2-furfural, 2-acetylfuran, and 5-methyl-2-furfural) were prepared immediately before an experiment by sequential dilutions in unused transformer oil (GK oil, Rosneft, Russia) from a standard solution (10.0 g/L), which was obtained by dissolving appropriate amounts of analytes in *o*-xylene. The standard furan derivative solution remains stable when stored in a closed dark glass container in a refrigerator at 5°C for up to 3 months. Calibration solutions of furan derivatives were prepared by diluting the standard solutions (10.0 g/L), made by dissolving appropriate amounts of analytes in methanol, with deionized water.

To prepare the eutectic solvents, precursor portions were weighed based on their molar ratios (Table 1). The reagents were placed in a penicillin vial and heated to 75°C under constant stirring with a magnetic stirrer until a homogeneous, a transparent liquid was formed.

All reagents were of analytical or better grade.

Samples of used transformer oil were collected at a power plant operated by Rosseti (St. Petersburg). The samples were collected in 1-L dark glass bottles and stored in a refrigerator at 5 ± 1°C for up to 1 month. Before an analysis, the transformer oil samples were thoroughly stirred.

For performing vortex-assisted DLLME, an IKA vortex disperser (Germany) was used. The kinematic viscosity of the prepared hydrophilic eutectic solvents was measured using a Stabinger SVM 3001 viscometer (Anton Paar, Austria). Chromatographic analysis was carried out using an LC-20 liquid chromatograph equipped with a spectrophotometric detector (Shimadzu, Japan). The separation was performed in a Luna C18 column (150 mm × 4.6 mm × 5 μm; 100 Å). The mobile phase consisted of deionized water (solvent A) and methanol (solvent B). The following gradient elution mode was employed: 40% B (0–15 min), linear increase to 99% B (15–16 min), 99% B (17–25 min), linear decrease to 40% B (25–26 min), and 40% B (26–30 min). The flow rate was set at 0.6 mL/min, and the injection volume of the extract was 20 μL. The column was thermostatted at 35°C. The detection wavelengths were 220 nm for furfuryl alcohol and 280 nm for 5-hydroxymethyl-2-furfural, 2-furfural, 2-acetylfuran, and 5-methyl-2-furfural. The retention times for 5-hydroxymethyl-2-furfural, furfuryl alcohol, 2-furfural, 2-acetylfuran, and 5-methyl-2-furfural were 5.06, 6.84, 8.59, 10.64, and 13.77 min, respectively.

RESULTS AND DISCUSSION

For the microextraction of furan derivatives from transformer oil, we explored the potential use of hydrophilic eutectic solvents as extractants. Choline chloride, a cost-effective, environmentally friendly, and polar compound ($\log K_{\text{ow}} = -5.16$ [21]), was selected as a hydrogen bond acceptor (HBA) for eutectic solvent formation. Various hydrophilic carboxylic acids, including ascorbic, citric, malic, malonic, oxalic, tartaric, lactic, formic, and acetic ($\log K_{\text{ow}}$ ranging from −1.85 to −0.17 [21]), were selected as hydrogen bond donors (HBDs). At the preliminary stage, two-component eutectic solvents were prepared according to the recommendations [22]. Most of the prepared two-component eutectic solvents were liquids viscous at room temperature, which limited their use in microextraction without heating the extraction system. Certain furan derivatives, such as 5-hydroxymethyl-2-furfural and furfuryl alcohol, may decompose upon heating or exposure to atmospheric oxygen [23]. Therefore, to decrease viscosity, a third precursor—water (10%)—was added to the two-component eutectic solvents [24]. All of the three-component eutectic solvents formed emulsions when mixed with transformer oil using a vortex disperser at room temperature.

To select an effective extractant, the effect of the ES precursor nature (HBD) on the recovery of analytes was studied at a phase ratio of 1 : 1. In a 2.0-mL test tube, 500 μL of transformer oil with an analyte concentration of 500 μg/L was mixed with 500 μL of a three-component eutectic solvent. The systems were stirred for 30 min using a vortex disperser to achieve an extraction equilibrium. Subsequently, the systems were centrifuged at 5000 rpm for 3 min. The extract was collected for the further analysis by HPLC–UV. Based on the obtained data (Fig. 1), it can be concluded that, for most of polar analytes, such as 5-hydroxymethyl-2-furfural and furfuryl alcohol ($\log K_{\text{ow}} = -0.09$ and 0.28 [21], respectively), recovery is virtually independent of the HBD nature. This phenomenon is attributed to the presence of hydroxyl groups in the structures of these analytes, which can form hydrogen bonds with components of the eutectic solvent. For the other analytes, the most satisfactory recovery was achieved using an eutectic solvent based on choline chloride and acetic acid (1 : 1, mol/mol) with 10% water, which was selected for the further studies (the recoveries for all analytes ranged from 85 to 96%). This effectiveness is due to the polarity of the ES precursor—acetic acid ($\log K_{\text{ow}} = -0.17$ [21])—being closest to the polarity of 2-furfural, 2-acetylfuran, and 5-methyl-2-furfural ($\log K_{\text{ow}} = 0.42$, 0.52, and 0.67 [21], respectively), thereby ensuring the maximum affinity of the analytes to the extractant.

The molar ratio of the ES precursors can affect the mass transfer efficiency of the analytes from the transformer oil phase. To investigate this factor, the recoveries of the analytes were determined under equilibrium conditions in the extraction system using eutectic solvents with various molar ratios of choline chloride to acetic acid (ranging from 1 : 2 to 3 : 2), while maintaining a constant water content of 10%. An increase in the amount of acetic acid in the eutectic solvent consistently led to higher recoveries for the less polar analytes (2-furfural, 2-acetylfuran, and 5-methyl-2-furfural) (Fig. 2). In the subsequent studies, an eutectic solvent with a molar ratio of acetic acid to choline chloride of 1 : 1 was used, as at this ratio the maximum recovery for all the five analytes was achieved.

The water content of the eutectic solvent can also affect the recovery of the analytes from the transformer oil phase. For this reason, the mass transfer efficiency of the analytes was studied under equilibrium conditions in the extraction system using an eutectic solvent based on choline chloride and acetic acid (1 : 1, mol/mol) with varying water contents (10, 15, and 20%). An increase in water content beyond 20% may lead to the breakdown of the hydrogen bond structure within the eutectic solvent [25, 26]. It was demonstrated (Fig. 2b) that the water content of the extractant has little effect on the recovery of all analytes. On the other hand, an increase in the water content of the eutectic solvent decreases its viscosity, which affects favorably the rate of reaching an equilibrium in the eutectic solvent–transformer oil

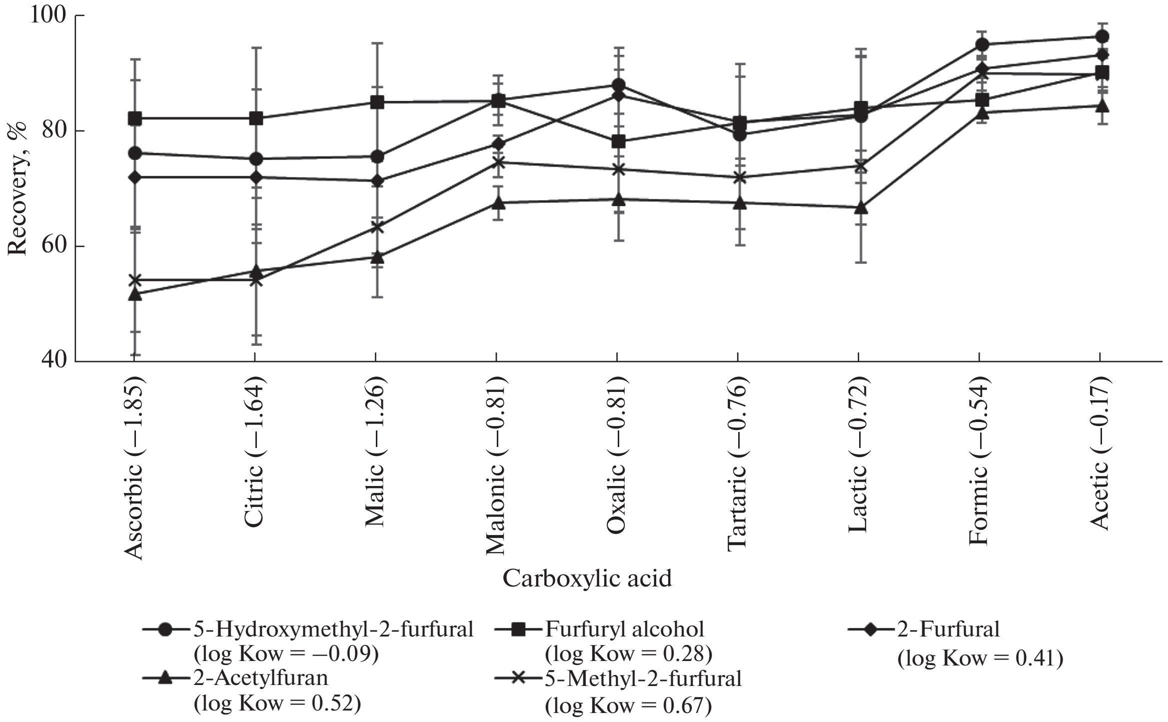


Fig. 1. Effect of the nature of the carboxylic acid—hydrogen bond donor of the eutectic solvent—on the recoveries of furan derivatives from transformer oil ($n = 3$, volumes of the eutectic solvent and transformer oil 500 µL, analyte concentration 500 µg/L, water content in eutectic solvent 10%, mixing time 10 min). Lipophilicity coefficients ($\log K_{ow}$) are indicated in parentheses.

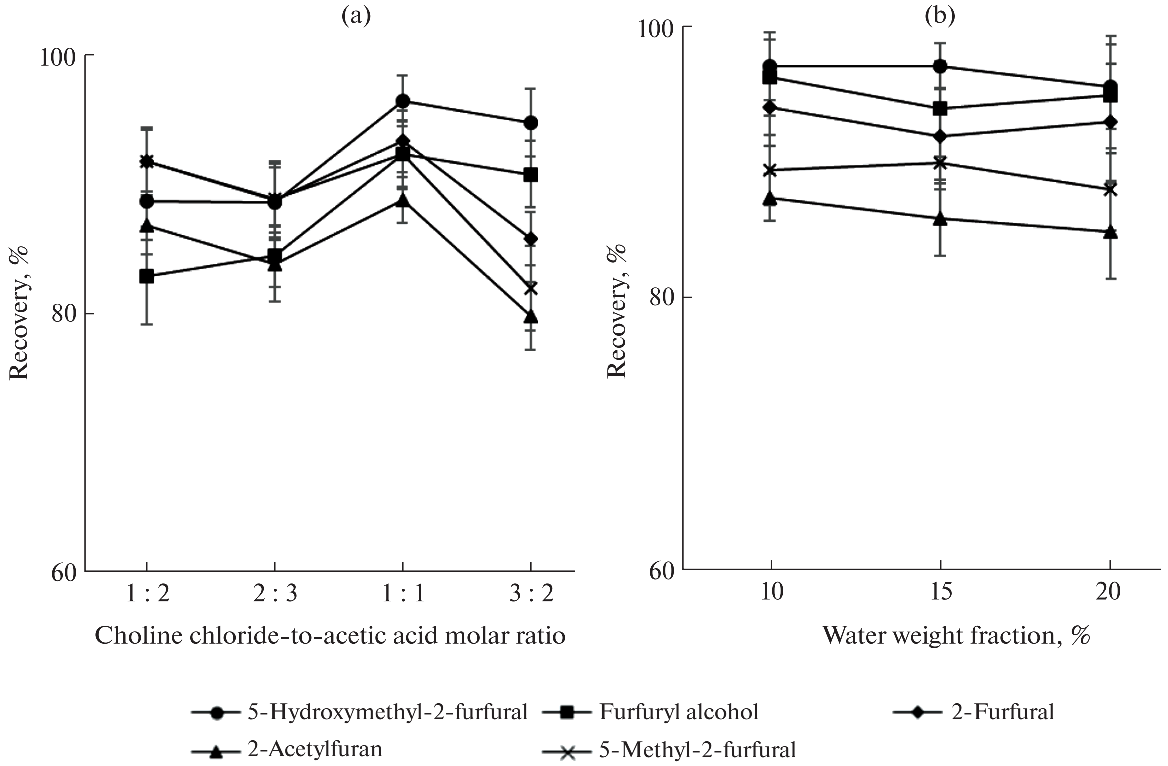


Fig. 2. (a) Effect of (a) the molar ratio of ES precursors and (b) water content in the eutectic solvent on the recoveries of furan derivatives ($n = 3$, volumes of the eutectic solvent and transformer oil 500 µL, analyte concentration 500 µg/L, mixing time 10 min).

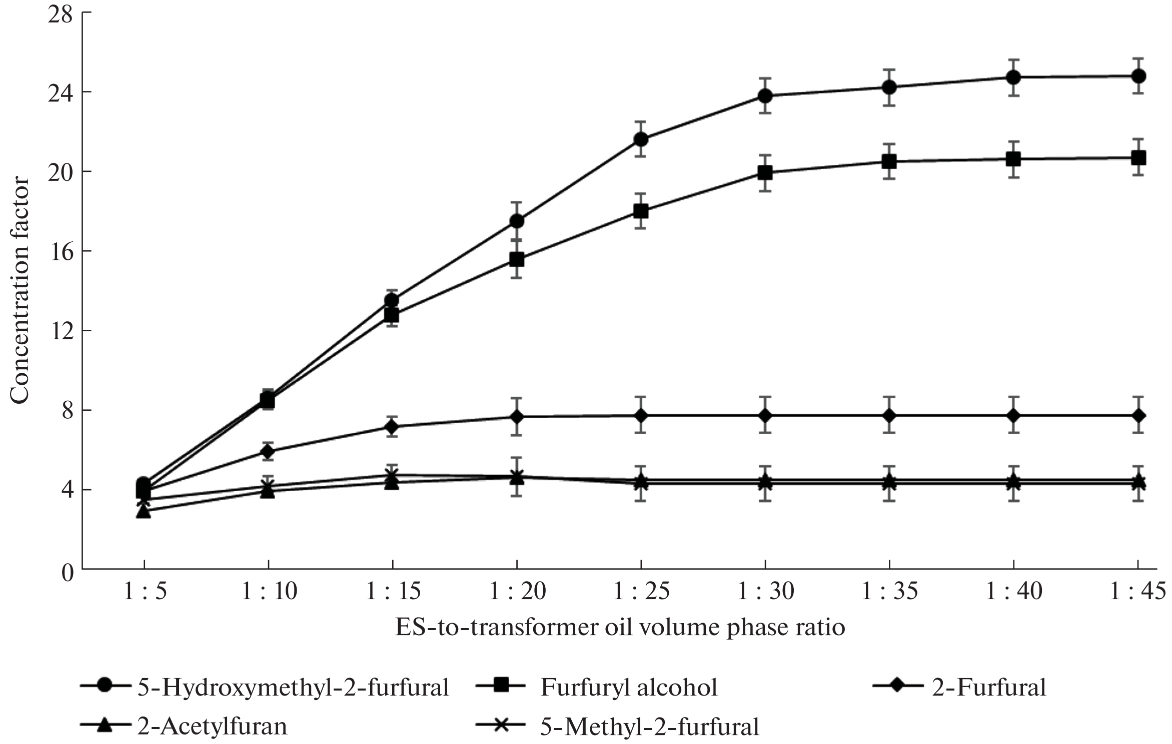


Fig. 3. Effect of phase volume ratios on the concentration factor of furan derivatives ($n = 3$, volume of the eutectic solvent 100 μL, analyte concentration 500 μg/L, ES composition: choline chloride and acetic acid (1 : 1, mol/mol) with 20% water, mixing time 10 min).

extraction system and prevents significant pressure buildup upon introducing the system into HPLC–UV. The further studies were conducted using an eutectic solvent based on choline chloride and acetic acid (1 : 1, mol/mol) with a water content of 20% (dynamic viscosity of 13.3 mPa·s at 25°C).

To lower the limits of detection, a preconcentration stage for the analytes was implemented. The phase ratios of the extractant-to-transformer oil were varied from 1 : 5 to 1 : 45. The volume of the eutectic solvent was set at 100 μL, which aligns with the concept of microextraction [13]. As the volumetric phase ratio was increased, the concentration factors for the less polar analytes decreased significantly more than for the more polar ones (Fig. 3). An optimal volumetric ratio of 1 : 40 was selected for the further studies due to the achievement of equilibrium saturation for all five analytes.

To shorten the sample preparation time, the effect of mixing time on the analytical signal was studied. The resulting extraction systems were stirred with a vortex disperser, with mixing times varied from 1 to 4 min. Equilibrium was reached within 1 min. During this process, choline chloride facilitated spontaneous phase separation (without centrifugation).

Thus, a method for determining furan derivatives in transformer oil has been developed (Fig. 4). A 4.0-mL portion of a transformer oil sample is placed in a polymer test tube, followed by the addition of 100 μL of an extractant based on acetic acid and choline chloride (1 : 1, mol/mol) with a water content of 20%. The mixture is stirred using a vortex disperser for 1 min. After spontaneous phase separation (1–2 min, depending on the viscosity of the transformer oil), 50 μL of the extract phase is collected with a chromatographic syringe for the subsequent determination of the furan derivatives by HPLC–UV.

During the validation of the developed method, the concentration ranges, limits of detection, limits quantification, determination coefficients (R^2), and precision characteristics—represented by repeatability and within-laboratory reproducibility—were experimentally established, along with accuracy according to the recommendations of Eurachem/CITAC [27]. The performance characteristics of the developed method are presented in Table 2.

To assess the accuracy of the results, furan derivatives were determined in samples of used transformer oil by the standard addition method. Recovery was calculated by the equation

$$\text{Recovery} = \frac{c_{\text{with\_addition}} - c_{\text{without\_addition}}}{c_{\text{addition}}} \times 100\%,$$

where $c_{\text{with\_addition}}$ is concentration of furan derivatives in the sample with an addition, mg/L; $c_{\text{without\_addition}}$ is

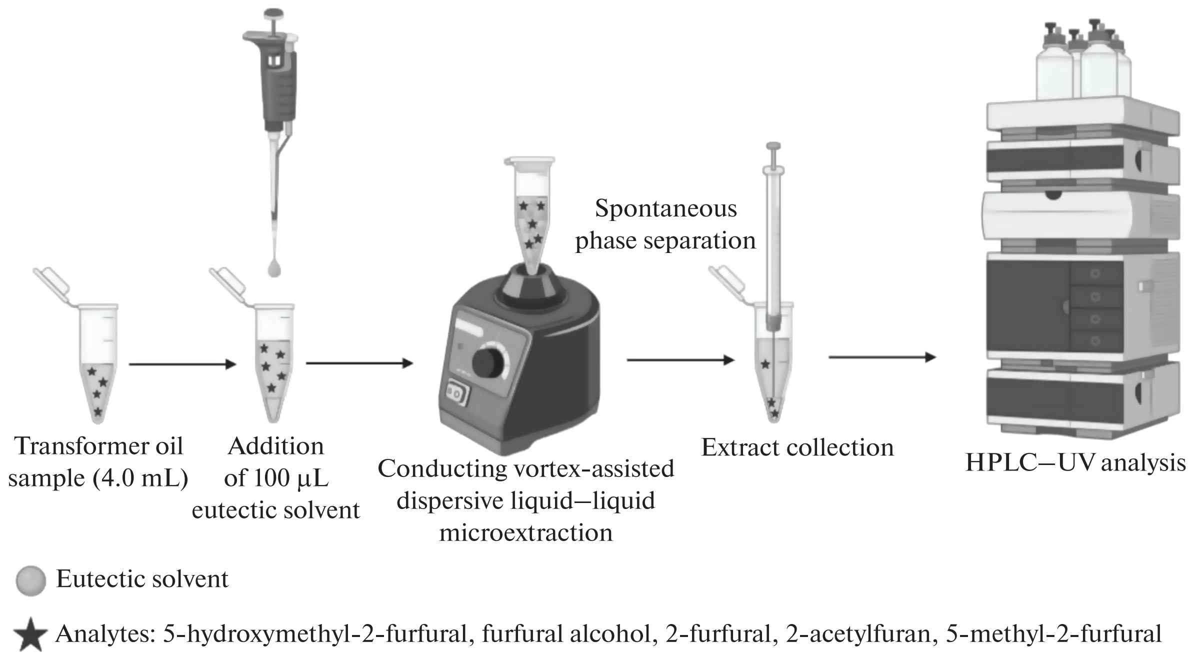


Fig. 4. Schematic diagram of the microextraction-chromatographic determination of furan derivatives in transformer oil.

concentration of furan derivatives in the sample, mg/L; and c_{addition} is concentration of an addition added to the sample, mg/L.

Based on the data in Table 3, it can be concluded that the condition of the paper insulation in the transformer is satisfactory and does not require replacement (the content of 2-furfural is below 100 μg/L) [3]. The developed method achieves recoveries for the analytes ranging from 90 to 101%. According to the recommendations [28], for samples with analyte concentrations at or below 10 mg/L, recoveries between 80 and 110% can be considered acceptable.

CONCLUSIONS

We proved the feasibility of extracting furan derivatives (5-hydroxymethyl-2-furfural, furfuryl alcohol, 2-furfural, 2-acetylfuran, and 5-methyl-2-furfural) from transformer oil using eutectic solvents based on choline chloride, carboxylic acids, and water through vortex-assisted dispersive liquid–liquid microextraction. The effect of the precursors of the eutectic solvents on the mass-transfer efficiency of the target analytes was studied. The eutectic solvent composed of choline chloride, acetic acid, and water ensures the extraction and preconcentration of furan derivatives from a 4-mL sample into 100 μL of extractant. An

Table 2. Performance characteristics of the method for determining furan derivatives in transformer oil

Parameter	5-Hydroxymethyl-2-furfural	Furfuryl alcohol	2-Furfural	2-Acetylfuran	5-Methyl-2-furfural
Analytical range, μg/L	3–10000	7–10000	7–10000	17–10000	13–10000
Coefficient of determination (R^2)	0.9995	0.9997	0.9982	0.9996	0.9993
Limit of detection (3σ), μg/L	1	2	2	5	4
Limit of quantification (10σ), μg/L	3	7	7	17	13
Repeatability (RSD, $n = 8$), % (at concentrations $c_{\min}$/10 000 μg/L)	4/1.8	5/2.0	3/1.7	4/1.2	5/2.3
Intralaboratory reproducibility (RSD, $n = 8$), % (at concentrations $c_{\min}$/10 000 μg/L)	5/3	7/4	4/2.5	5/2.8	5/2.3

RSD, the relative standard deviation; $c_{\min}$, the lower limit of the analytical range.

Table 3. Determination of furan derivatives in samples of used transformer oil ($n = 3$, $P = 0.95$)

Analyte	Added, μg/L	Found, μg/L	Recovery, %	Added, μg/L	Found, μg/L	Recovery, %
Sample 1						
5-Hydroxymethyl-2-furfural	0	<LOD*	–	2.47	2.49 ± 0.17	101
Furfuryl alcohol	0	<LOD*	–	2.52	2.42 ± 0.19	96
2-Furfural	0	251 ± 12	–	2.55	2.73 ± 0.07	97
2-Acetylfuran	0	<LOD*	–	2.53	2.43 ± 0.10	96
5-Methyl-2-furfural	0	29 ± 3	–	2.51	2.57 ± 0.16	101
5-Hydroxymethyl-2-furfural	49	44 ± 5	90	9.54	9.3 ± 0.3	92
Furfuryl alcohol	50	46 ± 5	92	9.63	9.3 ± 0.4	97
2-Furfural	51	300 ± 7	96	9.65	9.7 ± 0.3	98
2-Acetylfuran	51	48 ± 3	94	9.66	9.5 ± 0.3	99
5-Methyl-2-furfural	50	77 ± 7	95	9.61	9.3 ± 0.4	96
Sample 2						
5-Hydroxymethyl-2-furfural	0	<LOD*	–	2.47	2.43 ± 0.0 8	95
Furfuryl alcohol	0	<LOD*	–	2.52	2.43 ± 0.18	96
2-Furfural	0	140 ± 7	–	2.55	2.62 ± 0.1 3	97
2-Acetylfuran	0	<PO*	–	2.53	2.47 ± 0.1 8	98
5-Methyl-2-furfural	0	21.6 ± 2.5	–	2.51	2.47 ± 0.11	97
5-Hydroxymethyl-2-furfural	49	46 ± 7	93	9.54	9.2 ± 0.5	96
Furfuryl alcohol	50	47 ± 10	93	9.63	9.4 ± 0.4	98
2-Furfural	51	187 ± 22	92	9.65	9.66 ± 0.2 6	99
2-Acetylfuran	51	48 ± 4	95	9.66	9.4 ± 0.4	97
5-Methyl-2-furfural	50	68 ± 7	92	9.61	9.4 ± 0.3	98

* LOD is limit of detection.

effective and accessible method for the chromatographic determination of trace concentrations of furan derivatives in transformer oil has been developed.

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CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

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