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## PROSPECTS FOR THE USE OF DIELECTRIC BARRIER DISCHARGE FOR DIAGNOSTICS AND MONITORING OF METALS IN WATER BODIES IN THE IVANOVO REGION

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**Abstract.** The paper demonstrates the potential applications of optical emission spectroscopy. A dielectric barrier discharge is used as the excitation source. It is implemented on a 'Partsko' experimental device with a mica plate (frequency 1–50 kHz, voltage on electrodes 3–5 kV) for diagnostics and monitoring of pollutants, in particular metals, in natural water bodies. This physico-chemical method is innovative and promising for identifying and analysing sources of metal contamination in wastewater from industrial enterprises, agricultural organisations, domestic facilities, and transport infrastructure.

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### Introduction

Physico-chemical analytical methods, which based on the interaction of electromagnetic radiation with a substance, play a central role in modern science and technology. They enable the identification and quantitative analysis different substances composition. These methods provide valuable information about the structure and the physical and chemical properties of the materials under investigation without causing any damage. It makes them the reliable tools for a wide range of scientific research and industrial applications [1].

In particular, spectral methods are now used for much more than simply identifying substances. They play a vital role in fundamental research, enabling the assessment of atomic



and molecular structures, the study of the nature of intermolecular interactions, the kinetics of chemical reactions, and energy transfer processes [2, 3]. In materials science, spectroscopy ensures the analysis of alloy compositions and the quality of applied and cured coatings, as well as the monitoring of crystal growth processes. However, it is not an entire list of its practical applications [4, 5].

The widespread use of various spectral methods in solving a wide range of industrial, physicochemical, and everyday problems demonstrates their high effectiveness. In particular, during the 2020s, cost-effective X-ray fluorescence spectrometers became widely used for identifying jewellery at customs and verifying its quality and purity [6]. Among the current practical tasks, there is the analysis of residues of ancient artifacts by X-ray fluorescence spectrometry (*XRF*). Raman spectroscopy (spectroscopy of Raman scattering) is currently used to detect cancer biomarkers in tissues, for early diagnosis of diseases, and for improving treatment efficacy. Near-infrared spectroscopy (*NIR spectroscopy*) is used for the analysis of food composition and provides proper quality control and safety. Spectroscopic remote sensing techniques, such as *DOAS* (*Differential Optical Absorption Spectroscopy*), allow the concentration of pollutants in the atmosphere to be determined with high accuracy and sensitivity.

The purpose of this research is to study the feasibility of using optical emission spectroscopy (*OES*), technically implemented by exciting electrons with a dielectric barrier discharge (*DBD*), for monitoring metal content to assess the quality of aqueous solutions and media, with the additional prospect of identifying potential sources of pollution. Since the liquid phase is directly subjected to analysis, this approach can also be successfully applied in the study of biological tissues, addressing scientific and practical problems related to the determination of the elemental composition of blood, urine, saliva, early diagnosis of health issues, and monitoring infection and/or poisoning.

### **Description of the experimental setup and procedures**

*OES* used in conjunction with *DBD* is a powerful physicochemical tool for analysing the composition of samples [7]. However, to obtain reliable results, certain conditions should be observed and factors affecting the type and nature of the emission spectrum should be taken into account [8]. First of all, this concerns sample preparation. In our case, the experiments were conducted with liquid samples collected directly as environmental materials.

Natural samples are often contaminated (with fine particles of silt or sand, especially if the samples are taken from the bottom part of a water body). Therefore, before analysis, the liquids were filtered through paper filters and then allowed to stand at room temperature for 24 hours. The criterion for the acceptable degree of sample purification was the absence of visible contaminants on the filter paper when the analysed sample was passed through it.

Fig. 1 shows a diagram of the experimental apparatus.

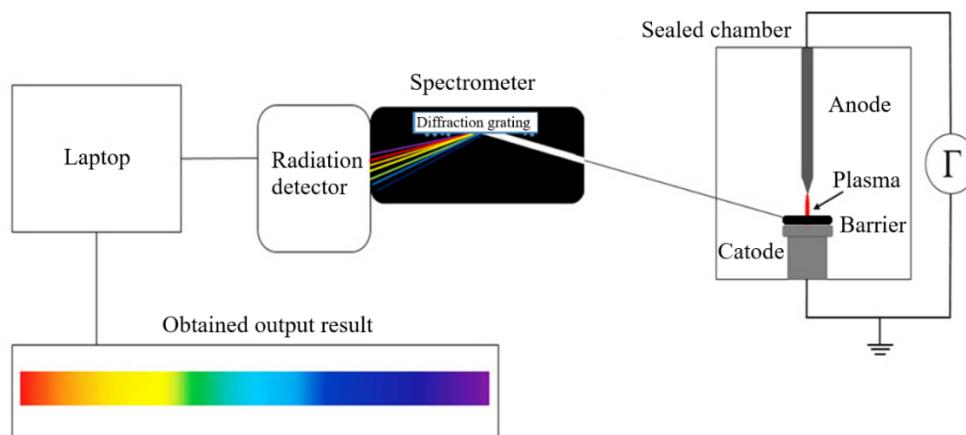


Fig. 1. Experimental setup for conducting research

A sealed transparent chamber with radiation extracted from it through a side opening was used for the experiment. A dielectric barrier was placed on the grounded stage of the cell; a fabric wetted with the liquid was placed on top of it; the liquid composition was being analysed for the presence of metals. The stage (cathode) and the second cylindrical electrode of the discharge system (anode) were made of stainless steel. Experiments were performed with anodes of various configurations, ranging from a sharpened tip to a flat-ended design with a base of varying surface area. The most effective option was an anode with a flat round base 4.0 mm in diameter. Under this condition, the discharge burned stably, and the fabric (the physical carrier of the object of study – the liquid) was not subjected to plasma destruction.

To implement a *DBD*, it is necessary to select a suitable dielectric barrier. Various materials were analysed for this purpose – glass, mica, and a silicone substrate. The best performance was demonstrated by a "Partsko" mica plate. For this plate, an optimal compromise was found between the electric strength and thickness, since an increase in barrier thickness reduces the intensity of the discharge emission. According to our estimates, the average operating time of one mica plate is 75 hours (sufficient for express analysis). In factory condition, such sheets withstood voltages of up to 20 kV across the discharge system electrodes while varying the frequency over a wide range. High frequencies were not used, which was due to the absence of a suitable high-voltage generator at this stage of the experiment. The generator  $G$  for the experiments (see Fig. 6) was constructed using a classic push-pull *ZVS* driver circuit with a fixed frequency; the frequency can be varied within the range of 1-50 kHz (by means of minor design modifications). In this work, a stable discharge without signs of spark breakdown was obtained at a frequency of 15 kHz using a cylindrical anode. The voltage across the electrodes was 3-5 kV, depending on the acquisition of a reproducible spectrum with sufficient emission intensity of characteristic metal lines. A mandatory requirement is the prevention of plasma (thermal) destruction of the fabric substrate. Due to its good liquid absorption, it serves as a carrier for the objects of study. The absence of fabric etching was monitored visually. The decision to use fabric as a carrier for the analysed liquid was intuitive. When the discharge was ignited, the liquid efficiently evaporated from the fabric, forming a vapor cloud of the analysed liquid in the interelectrode space. The strong electric fields in the interelectrode space facilitated the transfer of metal ions into the gas phase.



The proposed innovative physicochemical method for monitoring metal compounds in samples is fairly simple to implement. However, it has a drawback associated with the need to prevent etching of the fabric during the liquid analysis process. The short duration of the experiment (no more than 20-30 s) makes this drawback not critical ones in this case.

A portable small diffraction 55-mm spectrometer, model *CLMG-7206*, manufactured by *Gain Express* (People's Republic of China), was used for the experiments. The spectrometer does not require a separate power supply; it has small dimensions, and can be used in field conditions; its spectral operating range is 350-950 nm; the spectral resolution is 1.2 nm. A *Logitech HD Webcam C270* (1.3 MP) was used as a detector, providing automatic illumination correction and natural color reproduction. The resolving power of this camera allowed the authors to successfully record the resonance lines of metals in the emission spectra.

### Main body

Within the framework of this study, two tasks of both purely scientific and applied nature have been set. The first of these involves the selection of objects for the development of an express method for monitoring metals in aqueous media and water bodies. The second task concerns with the acquisition of systematic spectral data that could be used as a basis for quantitative analysis. It also seems appropriate to determine the main list of elements that we shall consider potentially hazardous.

If the selection of trace elements necessary for the functioning of living beings is straightforward and clear (these are, primarily, *Fe*, *K*, *Na*, and *Mn*) [9], then for metals that have harmful effects, it is necessary to analyse the pathways of their entry into the human body or into plant stems and bark, as well as the corresponding sources of their elevated concentration.

Thus, the sources of lead (*Pb*) accumulation are industrial emissions (primarily from metallurgical enterprises involved in the processing of lead ores), transportation (vehicle emissions, especially in areas with heavy traffic), and agricultural chemicals (pesticides and fertilizers may contain lead as an impurity). Lead accumulates in the soil and can be absorbed by plant roots, particularly in acidic environments.

Cadmium (*Cd*) is often present in phosphorites used for fertilizer production, and when those are applied, it can be accumulated in the soil. Enterprises involved in the production (processing) of batteries, electroplating shops, and metallurgical plants can release cadmium into the environment. Cadmium is readily absorbed by plant roots from the soil and can accumulate in edible parts of plants, such as leaves and fruits.

Ore processing plants and coal-fired power plants are the main sources of mercury (*Hg*) emissions. Mercury compounds are also used as fungicides, which leads to soil contamination. Mercury, like any heavy metal, accumulates in plant leaves and can enter living organisms through the food chain. The uptake of mercury by roots from the soil is less significant but is entirely possible in heavily polluted industrial areas.

Enterprises producing stainless steel and nickel alloys, tanneries, and the textile industry release nickel (*Ni*) and chromium (*Cr*) into the environment. Coal and oil combustion leads to the release of nickel into the atmosphere. Nickel is absorbed by plant roots from the soil and can accumulate in leaves and seeds. The concentration of nickel in plants increases in acidic soils.



The main sources of copper (*Cu*) accumulation in soil include agricultural chemicals. Copper compounds are used as fungicides and pesticides. Frequent application of such products can lead to copper accumulation in the soil. It results in impaired root system development and reduced crop yields.

Metallurgical enterprises, electroplating industries, and the paint and coatings industry contaminate the environment with zinc (*Zn*). Vehicle exhaust, especially near roads with heavy traffic, can serve as a source of zinc, which settles onto soil and plants in the form of salts. The degree of zinc contamination strongly depends on the clay mineral content and the soil pH level (as pH increases, the degree of contamination decreases). Both excess and deficiency of zinc can negatively affect plant growth and development, as well as influence photosynthesis and chlorophyll synthesis.

The sources of strontium (*Sr*) contamination are also quite extensive. Those are accidental releases from nuclear industry facilities. Secondly, non-ferrous metallurgy enterprises processing strontium-containing ores, as well as some chemical industry enterprises and manufacturers of ceramic products and glass. Indeed, strontium can be used as an additive to improve the properties of porcelain and refractory materials. This type of contamination should also be taken into account in the production of phosphate fertilizers, since some types of phosphorites are enriched in strontium [10].

Thus, conducting periodic monitoring of heavy metals in the environment proves to be a highly relevant task. Such studies are being conducted [11], but they are generally of a particular nature. Samples are preliminarily collected, prepared, and only then subjected to analysis. Moreover, these purposes often require expensive reagents of high purity. The use of unconventional physicochemical research methods – OES using mobile discharge excitation setups at atmospheric pressure (e.g., DBD) will allow data on the presence of metals in samples to be obtained directly in the field.

To determine the concentration of metals in water bodies, the internal standard method was used [12]. Its implementation requires the appropriate calibration curves. To obtain these curves, we studied the emission spectra of dilute solutions of metal salts potentially presented in the collected samples.

To construct the calibration curves, spectral data were used for solutions of metal chlorides with concentrations of: 0.005; 0.010; 0.050; 0.100; 0.500; and 1.000 mg/L for each metal. The emission spectra obtained for the metals are presented in Table 1.

**Table 1.** Characteristic emission wavelengths of the studied elements

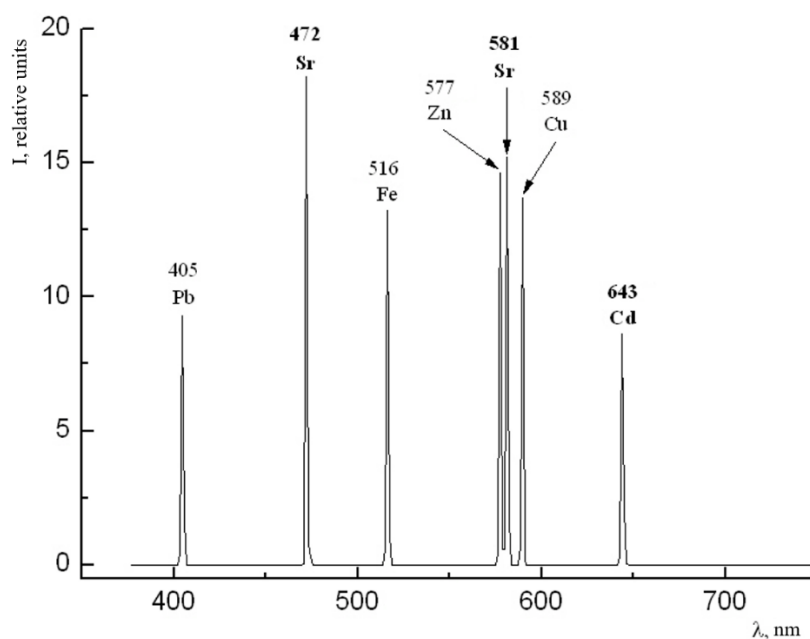
Item n/a	Analyzed metal	Selected emission wavelengths, $\lambda$ , nm
1	Na	589
2	K	583
3	Fe	516; 432
4	Mn	403
5	Cu	589; 406
6	Zn	577
7	Pb	405
8	Cd	643
9	Ni	471
10	Sr	581; 472
11	Hg	546; 436



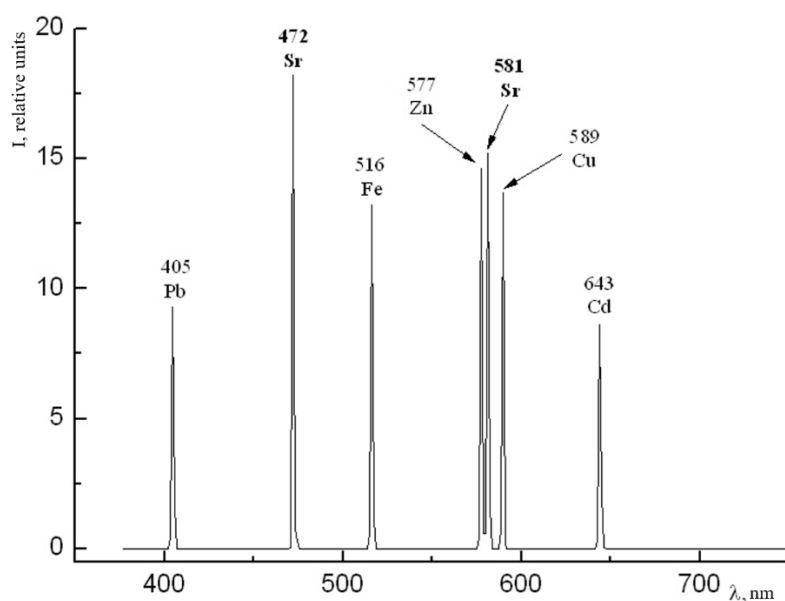
There is a list of selected emission wavelengths that unambiguously correspond to the excitation of a particular chemical element appearing in the spectrum.

Below there are the results of the analysis of samples collected from natural water bodies near the Uvod Reservoir in the vicinity of the localities of Yegoriy, Khudynino, and Mikshino, the Ivanovo region (Russia). Analysis of the emission spectra from samples collected near these localities showed that the main elements spectrally detected are lead, iron, zinc, and copper. The elements strontium and cadmium were not detected, even in trace amounts; therefore, chlorides of these metals were used as the internal standard.

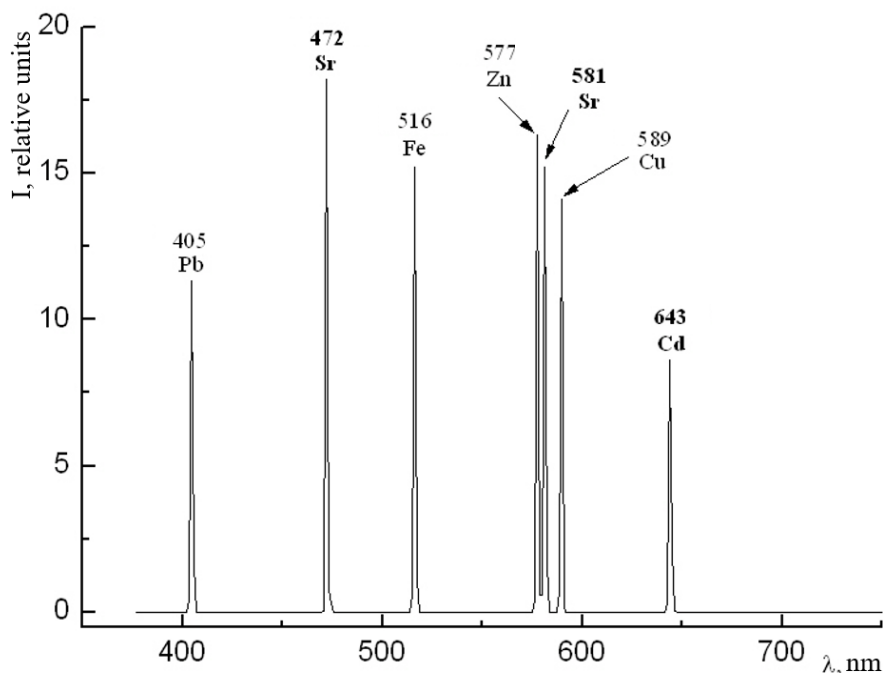
Figs. 2-4 show the emission spectra for the test samples collected near the aforementioned localities.



**Fig. 2.** Emission spectrum from a sample collected by the authors near the settlement of Yegoriy (the Ivanovo region, Russia).



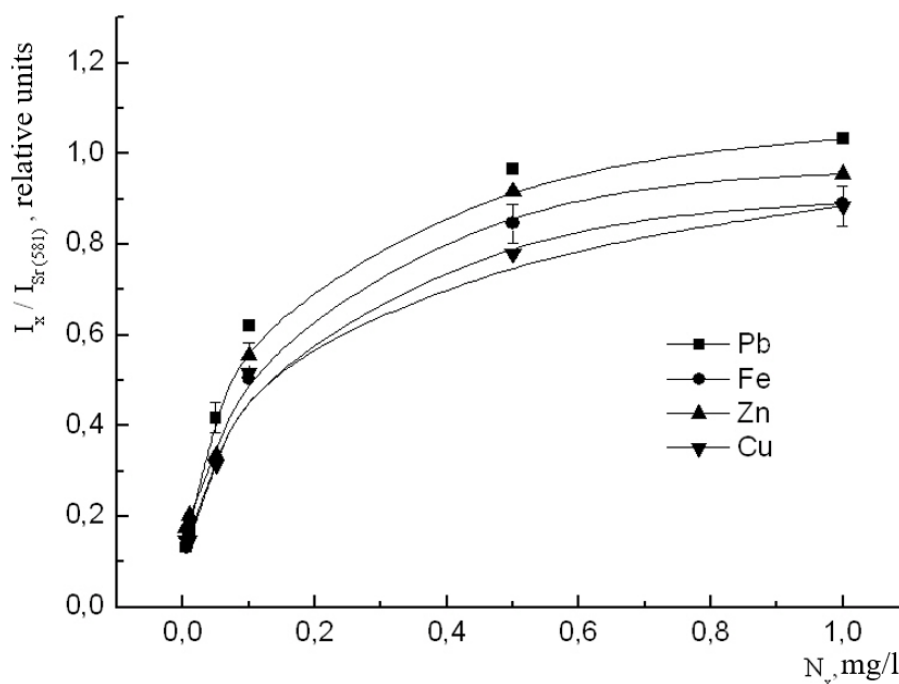
**Fig. 3.** Emission spectrum from a sample collected by the authors near the settlement of Khudynino (the Ivanovo region, Russia).



**Fig. 4.** Emission spectrum from a sample collected by the authors near the settlement of Mikshino (the Ivanovo region, Russia).

Figs. 2-4 show the lines of strontium (*Sr*) and cadmium (*Cd*) correspond to the emission of the internal standard – an additive specially introduced into the sample in the form of metal chlorides at a concentration of 1 mg/L.

The calibration curves (see Figs. 5, 6) were obtained for concentration values of the target metals in the range of 0.005...1.000 mg/L and, accordingly, for concentrations of the internal standard (*Sr* and *Cd*) equal to 1.000 mg/L. Matching the internal standard concentration for the experimental samples to that used for the calibration curves is the main condition for obtaining reliable quantitative results.



**Fig. 5.** Calibration curve; internal standard – strontium (*Sr*)

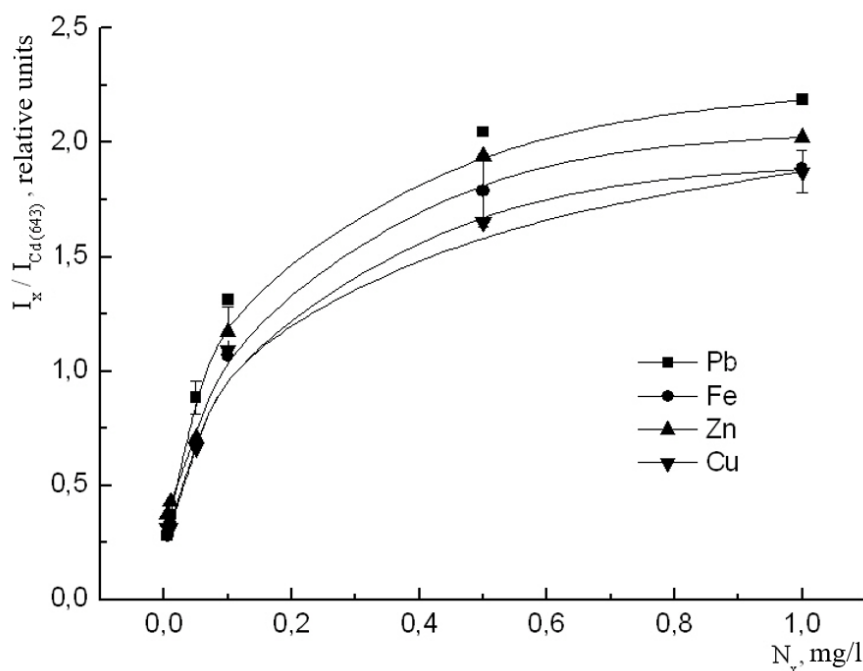


Fig. 6. Calibration curve; internal standard – cadmium (*Cd*)

In the calibration curves,  $N_x$  is the concentration of the chlorides of the corresponding salts. Therefore, a subsequent conversion to the metal concentration was performed using the chemical compound formula to obtain  $N_m$ . The use of salts as the internal standard is necessary to check the influence of the factor of different excitation probabilities of states on the emission intensities. Indeed, if processing data obtained using two different standards yields comparable results for metal concentrations in identical samples, it can be assumed that this factor will be automatically accounted for when implementing the internal standard method. However, it requires separate discussion.

Indeed, the emission intensity in emission spectroscopy depends on concentration and the excitation rate coefficient of the emitting state, which may be different for metals. This is an important aspect in the context of the proposed method; the following reasoning explains why the excitation rate coefficient (*ERC*) was not explicitly taken into account. The implementation of the internal standard method (actinometry method) involves constructing a dependence of the intensity ratio of the target component ( $I_{target}$ ) on the concentration of the target metal ( $C_{target}$ ). The *ERC* for each metal (*Pb*, *Fe*, *Zn*, *Cu*), for strontium (*Sr*) in one variant, and for cadmium (*Cd*) in the second, is implicitly accounted for in the calibration curve. Indeed, the *ERC* (the probability of excitation followed by emission) determines how efficiently an atom of a given metal transitions to an excited state under the action of plasma energy (in this case, *DBD*) and then emits a photon (here we assume that excited states decay via photon emission). This coefficient is closely related:

- to the probability that, upon collision with an electron, an atom will transition to a specific upper energy level;
- to the energy distribution of electrons in the plasma (factors of experimental temperature and electron concentration), i.e., the number of electrons responsible for exciting the atoms.



OES measures the emission, which is the result of all processes, including evaporation, atomization, ionization, excitation, and subsequent emission. Thus, the line intensity is a complex indicator.

Now we define the role of the internal standard. Within a single experiment (whether calibration or for an unknown sample), all analytes are subjected to the same conditions during DBD. This means that they experience the same energy input from the plasma; i.e., fluctuations in the DBD parameters (temperature, electron concentration, plasma geometry) for the elements under consideration will be comparable. On the other hand, in the ratio  $I_{target}/I_{Sr}$ , we are essentially dividing the intensity of the target metal included its ERC and plasma effects, by the intensity of strontium included its ERC and the same plasma effects. If the plasma parameters are not constant (e.g., temperature), this can lead to changes in the intensity of both the target metal and strontium. As a result, their ratio remains relatively constant despite changes in absolute intensities. Thus, the calibration curve  $I_{target}/I_{Sr} = f(C_{target})$  will empirically reflect the combined effect of concentration, the ERC of the target metal, the ERC of strontium (the standard), and the influence of the DBD. It shows how the ratio of measured signals depends on the analytical quantity (concentration). The other factors affecting intensity are averaged out and compensated for through the use of the internal standard.

Based on the above, the concentrations of metals in various samples from water bodies of the Ivanovo region (Russia) were determined (see Table 2). The experimental data shown in Figs. 1-5 were used as the initial ones.

**Table 2.** The results of determining the concentration of metals in water samples collected from water bodies of the Ivanovo region (Russia) recalculated as the concentration of the pure metal ( $N_m$ )

<b>The village of Yegoriy</b>				
Metal in the sample	$N_m$ , mg/l (Sr Standard)	$N_m$ , mg/l (Cd Standard)	Average $N_m$ value obtained using the two standards, mg/L	Maximum permissible concentration (MPC), mg/L
Lead	0.08±0.01	0.05±0.01	0.07±0.01	0.03
Iron	0.23±0.01	0.12±0.01	0.18±0.01	0.30
Copper	0.56±0.04	0.28±0.02	0.42±0.03	1.00
Zinc	0.46±0.03	0.15±0.01	0.31±0.02	1.00
<b>The village of Khudynino</b>				
Lead	0.07±0.01	0.05±0.01	0.06±0.01	0.03
Iron	0.33±0.02	0.12±0.01	0.23±0.02	0.30
Copper	0.64±0.05	0.20±0.02	0.42±0.03	1.00
Zinc	0.62±0.04	0.19±0.01	0.41±0.03	1.00
<b>The village of Mikshino</b>				
Lead	0.18±0.01	0.08±0.01	0.13±0.01	0.03
Iron	0.62±0.04	0.17±0.01	0.40±0.01	0.40
Copper	0.60±0.05	0.23±0.02	0.42±0.03	1.00
Zinc	0.63±0.05	0.23±0.01	0.43±0.03	1.00

Note: in all cases, metal chlorides were used to obtain calibration curves -  $PbCl_2$ ,  $FeCl_3$ ,  $CuCl_2$ ,  $ZnCl_2$ .



The qualitative set of metals detected in samples taken from different localities turned out to be identical. It is explained by the fact that the water bodies are connected by a single system (the Uvod Reservoir, the Ivanovo region, Russia). However, the quantitative composition of metals in the analyzed samples proved to be different. In particular, the results obtained at Mikshino (the Ivanovo region, Russia) indicate a significant exceedance of the maximum permissible concentration (MPC) for lead. It is characteristic that the MPC values applied were those for drinking water as well as for water bodies used for domestic and economic purposes [11].

### Conclusions

The use of OES in practical combination with DBD implemented at atmospheric pressure will allow a range of pollutants, such as heavy metals in natural water samples from the Ivanovo region, Russia, to be detected with a sufficient degree of accuracy. The sensitivity of the proposed physicochemical analytical method is at the level of the maximum permissible concentration of the pollutant. The implementation of this approach proves to be highly promising from a practical standpoint. Moreover, it serves as a basis for designing mobile portable devices necessary for environmental monitoring.

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